IEA
SOLAR R&D

INTERNATIONAL ENERGY AGENCY

solar heating and
cooling programme

task X
research on materials
for solar heating and cooling systems

WORKING DOCUMENT OF
SURVEY RESEARCH ON
TEST PROCEDURES AND
MEASUREMENT TECHNIQUES
subtask B

october 1987

WORKING DOCUMENT

OF

SURVEY RESEARCH

ON

TEST PROCEDURES AND MEASUREMENT TECHNIQUES

IEA TASK .X Subtask B

TEST PROCEDURES AND MEASUREMENT TECHNIQUES

K. HAYAKAWA, T. NOGUCHI* AND S. TANEMURA

Government Industrial Research Institute, Nagoya, AIST, MITI
* Japan Machinery & Metals Inspection Institute

This working document is a part of the work of

IEA Solar Heating and Cooling Programme.

Task X   : Research on Materials for Solar Heating and
             Cooling Systems

Subtask B: Test Procedures and Measurement Techniques

Distribution: Participating countries

Additional copies can be ordered from:

Solar System Development Association of Japan;

1-4-10, Nishishinbashi, Minato-ku,
Tokyo   105,   Japan

Table of Contents

International Energy Agency

In order to strengthen cooperation in the vital area of energy policy,
an agreement on an International Energy Programme was formulated among a
number of industrialized countries in November, 1974. The International
Energy Agency (IEA) was established as an autonomous body within the Or-
ganization for Economic Cooperation and Development (OECD) to administer
that agreement. Twenty countries are currently members of the IEA with
the Commission of the European Communities participating under a special
arrangement.

As one element of the International Energy Programme, the participants
undertake cooperative activities in energy research, development and
demonstration. A number of new and improved energy technologies which
have the potential of making significant contributions to our energy
needs were identified for collaborative efforts. The IEA committee for
energy research and development (CRD), assisted by a small Secretariat,
coordinated the energy research development and demonstration programme.

Solar Heating and Cooling Programme

In July, 1975 Solar Heating and Cooling was selected as one of the six-
teen technology fields for multilateral cooperation. the objective was
to undertake cooperative research, development, demonstrations and ex-
changes of information in order to advance the activities of all par-
ticipants in the field of solar heating and cooling systems. Several
tasks were developed in key areas of solar heating and cooling. A for-
mal implementing agreement was prepared, covering the contributions,
obligations and rights of the participants, as well as the scope of each
task. The agreement has been signed by the seventeen countries and the
Commission of the European Communities. The overall programme is
managed by an Executive Committee, while the management of each task is
the responsibility of an Operating Agent who acts on behalf of the other
participants. The tasks of the IEA Solar Heating and Cooling Programme
and their respective Operating Agent (lead organization responsible for
the task) are:

  I   Investigation of the Performance of Solar Heating and Cooling
        Systems - Technical University of Denmark,

  II   Coordination of Research and Development on Solar Heating and
        Cooling Components - Agency of Industrial Science and
        Technology, MITI, Japan,

  III   Performance Testing of Solar Collectors - Kernforschungsanlage
        Jülich, Federal Republic of Germany / University College,
        Cardiff, United Kingdom,

IV   Development of an Insolation Handbook and Instrumentation Package – United States Department of Energy,

V   Use of Existing Meteorological Information for Solar Energy Applications – Swedish Meteorological and Hydrological Institute,

VI   Performance of Solar Heating, Cooling and Hot Water Systems Using Evacuated Collectors – United States Department of Energy,

VII   Central Solar Heating with Seasonal Storage – Swedish Council for Building Research,

VIII   Passive and Hybrid Solar Low Energy Buildings – United States Department of Energy,

IX   Solar Radiation and Pyranometer Studies – Canadian Atmospheric Environment Service,

X   Research on Materials for Solar Heating and Cooling Systems – Agency of Industrial Science and Technology, MITI, Japan,

XI   Passive and Hybrid Solar Commercial Buildings – Swiss Federal Office of Energy.

Collaboration in additional areas may be considered as projects are completed or fruitful topics for cooperation are identified.

Abstract

The test procedures and measurement techniques for optical, thermal, mechanical, physical and chemical properties to evaluate material performance, material reliability and for material durability among the four designated solar materials categories such as window and collector glazing, collector absorber, heat transfer media with or without additives and heat storage material were surveyed.

Evaluation and analysis of the state-of-the-art of materials testing and measurement methods were prepared on the basis of the compiled one hundred and thirty eight reports contributed by the participants.

Recommendations for interlaboratory testing, where required, to determine the durability of materials and to develop the data needed for the development of new and/or modified testing and measurement methods are emphasized.

1. INTRODUCTION

The objectives of this subtask are to determine the most appropriate testing and measurement methods for evaluating the performance of designated materials used in solar applications and to hasten development and acceptance of testing and measurement methods that are mutually acceptable to the participating countries.

Specific objectives are:

to identify existing test procedures and measurement techniques for determining materials performance, durability, reliability and safety,

to determine the use and limitations of currently available test procedures and measurement methods,

to recommend needed new and/or modified testing procedures and measurement techniques.

The Test Procedures and Measurement Techniques Subtask consists of the following activities:

(1) <u>Establish Reporting Formats and Procedures</u>

Based on previous IEA performance data tasks, develop, as agreed, standard formats for reporting test procedures and measurement techniques used by participating countries to determine the performance, durability, reliability and safety of designated materials used in various solar heating and cooling applications.

(2) <u>Identification of the State-of-the-Art of Test Procedures and Measurement Techniques</u>

Prepare a compilation of testing procedures and measurement techniques used by participating countries to determine the performance, durability, reliability and safety of materials used in various solar heating and cooling applications. Performance measurements include the thermal, optical and mechanical properties of materials, as applicable. Both accelerated and abbreviated (short term "real-time" exposure tests will be considered for determining the durability/reliability of materials.

(3) <u>Evaluation and Analysis of the State-of-the-Art of Materials Testing and Measurement Methods</u>

The testing and measurement techniques will be analyzed and evaluated to establish their use and limitations. A report will be prepared identifying the use and limita-

tions of existing methods. This interim report will also make recommendations for interlaboratory testing, where required, to determine the reproducibility of measurements and to resolve differences in methods, and to indicate research required to develop the data needed for the development of new and/or modified testing and measurement methods.

(4) Technical Studies

Interlaboratory studies, identified in (3) above, will be conducted, where required, both to determine the reproducibility of testing and measurement methods and to resolve differences and issues arising from comparisons of existing procedures and techniques. Additional research will be conducted, as needed, to develop the technical basis for new and/or modified testing and measurement methods. Reports will be prepared as required. It is anticipated that each participating country will conduct specific activities in this area.

(5) Recommendations for New and/or Modified Testing and Measurement Methods

The technical studies conducted in (4) above, will be used as the basis for recommendations for new and/or modified testing procedures and measurement techniques.

(6) Participation in Subtask B Working Group Meeting

Participants are expected to send one or more representatives, as necessary, to each working group meeting. Representatives, when possible, should be those individuals directly performing the work to be discussed or reviewed at that particular meeting. It is anticipated that two subtask meetings will be held per year, in conjunction with the main Task X meeting.

Thus far, the Subtask B activity of the first phase is to survey and review the test procedures and measurement techniques on solar energy materials such as absorbers, glazings, heat transfer media with or without additives and storage materials.

According to the agreement at the Workshop on Solar Energy Materials Research held in Washington, D.C. in June, 1985, the lead country has prepared the draft on research items on testing procedures and measurement techniques for key solar energy materials of four kinds. The draft was distributed to the Participants for revision and approval by ballot in November, 1985. The target of this draft is to review the present state-of-the-art of testing

procedures and measurement techniques of solar energy materials and to prepare the matrix coding by which the participants might identify the problem areas and to encourage the discussions on future cooperative works in this Subtask B.

Throughout the past two Expert Meetings both in Vienna in April, 1986, and in Madrid in October, 1986, the solar materials among wide varieties were narrowed down as follows:

(1) Glazing

Glass:

Float, low iron and tempered
Insulating glass, role of sealants*

Plastics:

Polycarbonate
Polyethylene terephthalate (Polyester)
Polymethyl methacrylate (Acrylic)
Glass fiber reinforced polyester and acrylic
Polyvinyl fluoride*

Heat mirror (dielectric-metal-dielectric)

Optical switching materials:

Electrochromics

(2) Absorber:

Cermets including black chrome
 (metal-metal oxide, metal-metal carbides)
Anodized nickel pigmented aluminum
Black Zinc-ZnO
Selective paints*

(3) Heat Transfer Media with or without Additives

Ethylene glycol-water including inhibitors
Propylene glycol-water

(4) Thermal Storage (PCM)

Salt hydrates
Sodium sulfate decahydrate
Calcium chloride hexahydrate
Pentaerythritol
High density polyethylene
Paraffins (alkanes)

The materials indicated with asterisk were recognized as being of secondary importance. Some materials such as silica aerogels for glazing, holographic films for glazing, reflector materials, and chemical heat storage materials may be included in the specific case studies in later projects. The criteria applied to the materials selection were as followings:

(1) Interest for many of the participating countries

(2) Existence of related on-going research

(3) Existence of technical information related with in-use performance, testing and measurement techniques and aging and degradation modes.

The list of items for the survey of the present state-of-the-art of test procedures and measurement techniques are indicated in the Table 1.

The contribution of formatted reporting on this survey research is shown in the tables prepared in such respective chapter as the material groups together with the list of the contributors and the titles of reports.

Evaluation and analysis of the state-of-the-art of testing procedures and measurement techniques is followed by the introduction for respective designated group of materials. As the result of this evaluation and analysis, the areas required for the further cooperative laboratory works are identified and those are shown in the final part of the respective group of materials.

The 138 formatted reports from participants are compiled in the Appendix I for four designated material groups and the survey research items, respectively. The designation shown in the respective report has the meaning listed in Table 2. In some cases, the combination of the symbol can be used, if the described test procedures and/or measurement techniques can be applicable for different materials categories, properties to be tested or measured, or category of standardization.

Table 1.   The List of Research Items

I. Absorber

    (a) Optical Properties

        (i)   Solar absorptance

        (ii)   Emittance at a designated temperature

        (iii)   Reflectance

    (b) Thermal Properties

        (i)   Heat conducting properties (Heat conductivity, etc.)

    (c) Physical Properties

        (i)   Coating (mechanical strength: resistant to crazing,
                checking, cracking, flaking, peeling (delamination))

        (ii)   Substrate (mechanical strength: resistant to bending,
                compression, etc.)

    (d) Durability

        (i)   Durability to a stagnation temperature

        (ii)   Durability to a designated constant temperature
                (thermal degradation)

        (iii)   Durability to heat cycles

        (iv)   Durability to thermal shock

        (v)   Durability to the exposure to solar radiation under
                or without cover plate

        (vi)   Durability to moisture and/or dew

        (vii)   Durability to the specific atmospheric conditions
                (including vacuum condition and polluted air condition)

        (viii) Durability to salt water, acids or bases

        (ix)   Durability to mixed environmental conditions such as
                elevated temperature, solar radiation, moisture, etc.

- continued -

II. Glazing

(a) Optical Properties

    (i)   Solar absorptance

    (ii)   Solar transmittance

    (iii) Reflectance

    (iv)   Emittance at a designated temperature

(b) Thermal Properties

    (i)   Heat conductivity

    (ii)   Heat transfer coefficient

(c) Physical Properties

    (i)   Mechanical strength: tensile properties, dimensional stability, etc.

    (ii)   Flammability of plastic glazing

    (iii) Physical properties on optical switchable materials

        a) Drive voltage and power consumption of electro chromic materials

        b) Switching temperature of thermo chromic materials

        c) Switching light intensity of photo chromic materials

        d) Response speed and optical change ratio

(d) Durability

    (i)   Durability to a stagnation temperature

    (ii)   Durability to a designated constant temperature (thermal degradation)

    (iii) Durability to the exposure to solar radiation

    (iv)   Durability to thermal shock

    (v)   Durability to heat cycles

(vi)   Durability to moisture and/or dew

(vii)   Durability to the specific atmospheric conditions
        (including vacuum condition and polluted air condition)

(viii) Durability to salt water, acids or bases

(ix)   Durability to mixed environmental conditions such as
        elevated temperature, solar radiation, moisture, etc.

III. Heat Transfer Medium with or without Additives

   (a) Thermal Properties

      (i)   Heat conductivity

      (ii)   Specific heat

      (iii)   Latent heat (in the case of phase change heat transfer
              medium)

   (b) Transporting Properties

      (i)   Specific volume

      (ii)   Kinematic viscosity or coefficient of viscosity

   (c) Reliability

      (i)   Solidifying point

      (ii)   Boiling point

      (iii)   Flashing point

      (iv)   Toxicity and human hazards

   (d) Durability

      (i)   Stability of chemical composition and structure during
              the applied temperature elevation and heat cycles

      (ii)   Corrosive effect on the materials (metal, plastic, and
              rubber as sealant, etc.) used in the transfer loop

      (iii)   Durability to the exposure to solar radiation (if the
              heat transfer medium having the function of the direct
              solar-thermal conversion material in such a case of
              solar pond and black liquid tubular collector)
                                                    - continued -

IV. Storage Material

(1) Latent heat storage materials

(a) Thermal Properties

(i)   Phase change temperature (solidifying point, melting
                                  point)

(ii)   Latent heat

(iii) Specific heat

(iv)   Heat conductivity

(v)   Heat diffusivity

(vi)   Density of solid phase

(vii) Density of liquid phase

(b) Reliability

(i)   Vapor pressure at a designated temperature

(ii)   Ignition point

(iii) Flash point

(iv)   Content of toxic material

(v)   Toxicity and human hazards (toxic)

(c) Durability

(i)   Durability to heat cycles

(ii)   Durability at a designated temperature
       (thermal degradation, chemical stability)

(iii) Corrosive effect on the container vessel

Table 2. <u>Meaning of Designation</u>

(I) Material category

Symbol	Meaning
AC	Absoprber Coating
AS	Absorber Substrate
A	Absorber not identified both in AC and AS category
GC	Glazing Coating
GS	Glazing Substrate
G	Glazing not identified both in GC and GS category
HWA	Heat transfer medium with additives
HWNA	Heat transfer medium without additives
SS	Sensible heat storage material
SL	Latent heat storage material
SD	Desiccant material

(II) Properties to be tested or measured

Symbol	Meaning
O	Optical properties listed in the attached list
T	Thermal properties listed in the attached list
R	Reliability/service requirement properties listed in the attached list
D	Durability listed in the attached list

(III) Standardized testing/measurement or laboratory method

Symbol	Meaning
ST	Standardized method
LA	Laboratory method

I. GLAZING MATERIALS (Glass and Plastic)

1. Introduction

For glazing materials used in solar applications, glass and plastics are popular and optical and physical properties of these materials have to be known.

Especially, in plastics and some kinds of glass, it is very important to estimate durability of these materials.

In this document following materials are taken into account.

(1) Raw glass and plastics

   transparent glazing, heat absorbing glazing

(2) Coated glazing

   heat reflective glazing, low emissive glazing

(3) Optical switchable glazing

   electro chromic glazing, thermo chromic glazing, photo chromic glazing

(4) Other glazing

   thermal insulation glazing, (double glazing), photo chromic glazing

Testing and measurement methods for these properties were proposed from the Task X Participants. This document provides summary of these methods and standardized methods due to JIS (Japan Industrial Standard) and ASTM (American Standards of Testing Materials).

The titles of the reports and grouping of the reports are shown in the supplemental tables. Regarding optical properties, 9 reports were proposed. Besides, more than 10 reports were contributed on absorber materials. Basically these measurement methods of optical properties are quite similar and seem to be classified to two types. One is spectral method and the other is thermal method. Former one is well established and standardized.

In addition to the recent state-of-the-art of development of the standard methods for measuring the solar optical properties of materials such as spectral transmittance, reflectance and absorptance between 320 and 3,000 nanometers in wavelength, and total radiant and luminous transmittance and reflectance, it is informative to give the short description of ASHRAE standard which is now drafting and being not reported in this working document. This standard includes the method to measure spectral absorptance,

reflectance and transmittance of materials using spectrophotometers equipped with integrating spheres and the other method to measure solar transmittance (terrestrial) and solar radiant/luminous reflectance of materials using either a pyranometer or photometer, an enclosure, and the sun and sky as the source of radiation. The first method can be applicable to materials having both specular and diffuse optical properties. For transmitting sheet materials that are inhomogeneous, patterned, or corrugated, the second method in which both normal incidence and angles other than normal incidence are applied can be preferable.

Regarding thermal properties, 1 report was proposed, and it is a standard test method in ASTM.

Regarding physical properties, 7 reports were proposed on only mechanical strength. Generally, mechanical strength and flammability test are standardized in ASTM and/or established as a well known laboratory test.

Regarding durability, 12 reports were proposed on glazing materials, and more than 10 reports were proposed on absorber materials and some of them seem to be useful for glazing materials. They are mostly based on standardized test methods in ASTM and/or JIS.

But the problem is that there isn't any evaluation procedure for an accelerated durability test and it is important to cooperate works with other subtasks.

2. Respective Test Procedures

To evaluate the performance of glazing materials used in solar
applications, following properties and durability have to be known.

(a) Optical properties

    (i)   Solar radiation transmittance, absorptance and reflec-
tance

    (ii)   Emittance at a designated temperature

(b) Thermal properties

    (i)   Heat conductance

    (ii)   Heat transfer coefficient

    (iii) Thermal expansion

(c) Physical properties

  (1) Mechanical strength

    (i)   Wind load

    (ii)   Snow deposition load

    (iii) Hail impact

    (iv)   Tensile strength

    (v)   Bending strength

  (2) Flammability of plastic glazing

  (3) Physical properties on optical switchable materials

    (i)   Drive voltage and power consumption of electro chromic
materials

    (ii)   Switching temperature of thermo chromic materials

    (iii) Switching light intensity of photo chromic materials

    (iv)   Response speed and optical change ratio

(d) Durability

    (i)   Stagnation temperature

    (ii)   Designated constant temperature

    (iii) Exposure to solar radiation

    (iv)   Thermal shock

    (v)   Heat cycle

    (vi)   Moisture and/or dew

    (vii) Specific atmospheric conditions

    (viii) Salt water, acids or bases

    (ix)   Mixed environmental conditions
           (elevated temperature, solar radiation, moisture, etc.)

    (x)   High voltage for electro chromic materials

    (xi)   Electrical, thermal or light ON-OFF cycle

(a) Optical properties

    (i)   Solar radiation transmittance, absorptance and
          reflectance

        1) Method using a spectrophotometer (Report No. 1, 2, 15,
           22, 23, 24, 29 and 30)

           Spectral transmittance and reflectance are measured by
           using a scanning spectrophotometer with an integrating
           sphere to take account of the contribution due to dif-
           fused ray.

           Solar radiation transmittance and reflectance are
           obtained by calculation weighting spectral transmit-
           tance and reflectance with intensity distribution of
           solar energy. Solar radiation absorptance is calcu-
           lated from transmittance and reflectance.

           This method is standardized in JIS (R-3106) and ASTM
           (E-424 and E-903).

           Precision of this method is within 2% of full range.

           It is possible to determine the spectral transmittance
           and reflectance of multiple glazing units by calcula-
           tion using above values.

Calculation method is standardized in DIN 67507 and ISO/TC 160/GT2.

2) Method using a pyranometer

This method is applicable only for measurement of transmittance. Actual solar radiation is measured by a pyranometer settled in a box which can support large specimen and can face every directions and is painted flat black.

This method is standardized in ASTM (E-424).

Precision of this method is within ± 1%.

(ii) Emittance

1) Reflection method (Report No. 3, 26 and 31)

Spectral reflectance and transmittance of specimen at a designated temperature are measured by a spectro-photometer. From these data reflectance and normal emittance against heat flux are calculated.

This method is standardized in JIS (R-3106).

Precision of this method is within 2% of full range.

2) Radiometric method (Report No. 4, 25 and 32)

Samples and high-emitting reference are mounted on a turnable, electrically heating device. The radiation emitted by them is directed to the spectrometer system, that means the samples act as radiation source.

Precision of this method is within ± 3% of full range.

(b) Thermal properties

(i) Heat conductance

No report was proposed.

Testing method is standardized in JIS (A-1412 and 1413)

(ii) Heat transfer coefficient

No report was proposed.

(iii)   Thermal expansion

No report was proposed. But test method for absorber
is available for glazing.

This method uses a thermomechanical analyzer or similar
device to determine the linear thermal expansion of
solid materials.

Changes in specimen length are electronically recorded
as a function of temperature.

Precision is from 2% to 15%.

This method is standardized in ASTM (E-831).

(c) Physical properties

(1) Mechanical strength

(i)   Wind load

No report was proposed.

(ii)   Snow deposition load

No report was proposed.

(iii)   Impact strength (Report No. 9)

1) Impact strength of sheet glass using steel ball

This test is applied to tempered sheet glass.

A designated (weight and shape) steel ball is dropped
on the center of sample from various height in order
to know breakage height. this test method is stan-
dardized in JIS (R-3206) and also impact test with
propelled ice balls is standardized in ASTM (E-822).

(iv)   Tensile strength (Report No. 16, 17, 19 and 21)

Using tensile testing machine, uniform and constant rate
increase of stress is added on a sample to determine the
stress/strain characteristics and the point of failure.
Also test conditions are defined.

Testing method is standardized in JIS (K-7113) and ASTM
(D-618, D-638, D-794 and D-882).

(v)   Bending strength

1) Four point bending test (Report No. 7)

This test is used for measuring static surface strength
and edge strength of sheet glass. A narrow piece of
glass is set on a pair of knife edge supporting block,
and loaded on the center with a pair of knife edge
loading block.

Using the load at breakage point, breakage stress can
be calculated by means of bending stress formula.

This test is standardized in ASTM (C-158)

2) Ring on ring bending test (Report No. 8)

This test is applied to measure static surface strength
of sheet glass. Samples are set on a supporting ring
(outer ring) and concentrically loaded with a loading
ring (inner ring). Breakage strength can be calculated
by means of bending stress formula.

(2) Flammability of plastic glazing

No report was proposed.

Testing method is standardized in JIS (A-1321 and A-1322)

(3) Physical properties on optical switchable materials

(i)   Drive voltage and power consumption of electro chromic
materials

No report was proposed, but it is easy to know them by
using electric apparatuses.

(ii)   Switching temperature of thermo chromic materials

No report was proposed but it is easy to know it by
using temperature controlled box.

(iii) Switching light intensity of photo chromic materials

No report was proposed but it is easy to know it by
using light source and light intensity meter.

(iv)   Response speed and optical change ratio

        No report was proposed.

        As to photo chromic materials it is quite difficult to
        know optical change ratio.

(d) Durability

    (i)   Stagnation temperature (Report No. 14 and 27)
            Flat plate collector is exposed to the natural weather
            environment at temperature of stagnation conditions and
            is kept less than 1.5 W/m^2 heat loss.

            This method is standardized in ASTM (E-881 and E-781).

            Proposed method for absorber materials is also
            available.

    (ii)   Designated constant temperature (Report No. 13)

            Flat plate collector is exposed to the natural weather
            environment in the operational condition. This method
            is standardized in ASTM (E-782).

    (iii)   Exposure to solar radiation (report No. 5, 6 and 35)

            This test is defined as examining the change in the
            performance by exposing the test piece outdoor under
            natural conditions. Azimuth of exposure surface is
            0 degree and tilt angle is 30 to 35 degrees.

            This test method is standardized in JIS (A-1410 and
            A-1411).

            Same test using weathering box is standardized in ASTM
            (E-782) and accelerated test using concentrated sunlight
            is standardized in ASTM (E-838).

    (iv)   Thermal shock test (Report No. 10)

            Test machine has three independent chambers which keep
            high, intermediate and low temperature respectively.

            Samples are moved to these chambers in designated
            schedule. Temperature setting depends on material.

            (High temperature : 60 - 250 °C, intermediate tem-
            perature : room temperature, low temperature :
            -10 - -70 °C)

(v)   Heat cycles test (Report No. 11)

        Scheduled heat cycles of ambient condition which simu-
        late extreme day and night temperature difference are
        given to samples.

        Temperature range is -40 to 80 °C.

(vi)   Moisture and/or dew

        No report was proposed.

        Proposed methods for absorber materials are available.

(vii)   Specific atmospheric conditions

        No report was proposed.

        Proposed method for absorber materials is available.

(viii) Salt water, acids or bases

        No report was proposed.

        Proposed methods for absorber materials are available.

(ix)   Mixed environmental conditions (Report No. 12, 18, 20,
                                        28, 33 and 34)

        Samples are exposed to measured quantities of ultra-
        violet radiation within a climatic cabinet. The test
        environment maybe altered to allow different conditions
        of temperature and/or humidity to be maintained through-
        out the exposure period.

        This method is standardized in ASTM (G-53).

        In the case of report No. 12, evaluation of degradation
        by tensile test machine after and before exposure to
        high temperature and xenon arc light is shown. This
        method is standardized in ASTM (E-765).

        In the case of report No. 18 and 20, evaluation of
        dimensional changes at elevated temperature and
        accelerated service conditions are shown.

        These methods are standardized in ASTM (D-1204 and
        D-1042).

(x)   High voltage for electro chromic materials

No report was proposed.

(xi)   Electrical, thermal or light ON–OFF cycle

No report was proposed.

3. Proposal on Future Cooperative Works

Regarding optical switchable material properties, there isn't any report and standard. Especially durability of these materials is quite important and it is necessary to research the test and evaluation methods for these new materials in solar applications in future.

We propose the followings as future cooperative works.

For example.

(1) Accelerated durability test methods for electro, thermo and photo chromic materials.

(2) Measurement methods and discrimination methods of degradation of electro, thermo and photo chromic materials.

Reports Contributed by the Subtask B Participants
- GLAZING -

List of Items	Coding	AUSTRIA	CANADA	DENMARK	FRG	JAPAN	NETHERLANDS	SWEDEN	SWITZERLAND	USA	UK
a-(i)	G-O					●	●		○	●	○
a-(ii)	G-O					●	○	○			○
b-(i)	G-T										
b-(ii)	G-T										
b-(iii)	G-T										
c-(1)-(i)	G-R										
c-(1)-(ii)	G-R										
c-(1)-(iii)	G-R										
c-(1)-(iv)	GP-R					●				●	
c-(1)-(v)	G-R					●				●	
c-(2)	GP-R					○				●	
c-(3)-(i)	G-R									●	
c-(3)-(ii)	G-R										
c-(3)-(iii)	G-R										
c-(3)-(iv)	G-R										
d-(i)	G-D					●				●	●
d-(ii)	G-D					●					
d-(iii)	G-D					●				●	
d-(iv)	G-D					○					
d-(v)	G-D					○					
d-(vi)	G-D										
d-(vii)	G-D						○				
d-(viii)	G-D						○			●	●
d-(ix)	G-D									●	
d-(x)	G-D									●	
d-(xi)	G-D										

Table 1.   Grouping of Reports

	Laboratory Method	Standard Method
Optical properties	3, 22, 23, 24, 25, 26, 29, 31, 32	1, 2, 4, 15, 30
Thermal properties		
Physical properties — Mechanical strength	8	7, 9, 16, 17, 19, 21
Physical properties — Flammability		
Physical properties — Physical properties on optical switchable materials		
Durability	10, 11, 33, 34	5, 6, 12, 13, 14, 18, 20, 27, 28, 35

Table 2-1.   LIst of Reports

No.	Title	Reporter	Country
1.	Testing Method on Solar Transmittance of Glazing	K. Matsuoka	Japan
2.	Testing Method for Yellowness Index and Change of Yellowness of Plastics	K. Hojo	"
3.	Measurement of Normal Spectral Emittance of Absorber and Glazing Material	S. Tanemura	"
4.	Testing Method on Emittance at the Designated Temperature	K. Matsuoka	"
5.	Standard Method of Test for Change in Properties of Plastics Building Materials Resulting from Outdoor Exposure	K. Hojo	"
6.	Recommended Practice for Outdoor Exposure of Plastics Building Materials	"	"
7.	Four Point Bending Test of Sheet Glass	S. Tsuboi	"
8.	Ring on Ring Bending Test	"	"
9.	Test Method of Impact Strength of Sheet Glass Using Steel Ball	"	"
10.	Testing Method of Durability to Thermal Shock for Sheet Glass	"	"
11.	Testing Method of Durability to Heat Cycles for Sheet Glass	"	"
12.	Evaluation of Degradation of Cover Materials for Flat Plate Solar Collectors	C. Hauer	USA
13.	Exposure of Cover Materials for Solar Collectors to Natural Weathering under Conditions Simulating the Operational Mode	"	"
14.	Exposure of Solar Collector Cover Materials to Natural Weathering under Conditions Simulating Stagnation Mode	"	"

Table 2-2. List of Reports

No.	Title	Reporter	Country
15.	Solar Absorptance, Reflectance and Transmittance of Materials Using Integrating Spheres	C. Hauer	USA
16.	Conditioning Plastics for Testing	=	=
17.	Tensile Properties of Plastics	=	=
18.	Linear Dimensional Changes of Nonrigid Termoplastic Sheeting or Film at Elevated Temperature	=	=
19.	Tensile Properties of Thin Plastic Sheeting	=	=
20.	Linear Dimensional Changes of Plastics under Accelerated Service Conditions	=	=
21.	Determining the Permanent Effect of Heat on Plastics	=	=
22.	Solar Absorptance of Glazing	=	Sweden
23.	Measurement of Solar Transmittance Normal/Diffuse, Spectral	=	=
24.	Determination of the Solar Absorptance/Reflectance/Transmittance from Spectral Measurements at Near Normal Incidence	M. G. Hutchins	UK
25.	Measurement of Near Normal Total Emittance Using a Pyroelectric Radiometer	=	=
26.	Measurement of Thermal Emittance from Specular Reflectance Using an Infrared Spectrophotometer	=	=
27.	Outdoor Exposure to Stagnation Conditions for Solar Absorbers and Solar Glazings	=	=
28.	Exposure to Ultraviolet Radiation and Moisture	=	=

Table 2-3.   List of Reports

No.	Title	Reporter	Country
29.	Spectral Transmittance (Normal) of Glazings	J. Keller	Switzerland
30.	Determination of the Spectral Transmittance and Reflectance of Multiple Glazing Units	P. A. van Nij-natten	Netherlands
31.	Measurement of Reflectance for Determination of Hemispherical Total Emittance at 10°C Temperature		Sweden
32.	Determination of Directional and Hemispherical Total Emittance at Temperature of 60°C to 200°C	P. A. van Nij-natten	Netherlands
33.	Durability Aspects of Organic Sealed Insulating Glass	N. Van Santen	"
34.	Weatherometer Aging Test	J. G. M. Mange	"
35.	Aging Test on Materials for Transparent Covers of Collectors	K. Hayakawa	Japan

II. ABSORBER MATERIAL

1. Introduction

Test methods for absorbers on optical properties, thermal properties, service requirement and durability are reported from many countries.

The number of the reports from each country is shown in "Reports contributed by the Subtask B Participants".

In this table, the reports on absorber materials are counted up to 42.

Classification of collected reports is shown in Table 1, classified as laboratory born method or standard method according to the respective terms (optical properties, thermal properties, service requirement properties and durability).

Figures inserted in Tables 1, 2 and 3 coincide with the numbering on the Title and Reporter's name in Table 4.

1) Laboratory method and standard method

"Report Contributed by the Subtask B Participants" in Table 1 shows that the number of laboratory method is much more than that of the standard method.

However, by the referred document in several reports, each test is closely related to systems with reference to standard method (ASTM, DIN, etc.). (Report No. 1, 3, 9, 10, 14, 18, 19, 20, 22, 23, 24, 31, 32 and 33)

ASTM is referred as the standard method of optical property (No. 41, 42, 44, 45, 46 and 47), some of laboratory methods refer to these reports for example, (i) solar range (spectral) refers to ASTM E 903 (No. 3 and 10) or DIN 5033 (No. 1), (ii) thermal range (total) refers to ASTM E 408 (No. 9).

About durability, JIS and ASTM are reported from Japan, Sweden and U.S.A. But, for the most part, a laboratory method is taken up at this moment.

2) Tendency of reports

It is shown in Table 1 that much more descriptions on optical property and durability are reported, while those on thermal property and reliability/service are scarcely found.

Reliability/service signifies the test method about service
requirement properties for coating (mechanical strength;
resistant to crazing, checking, cracking, flaking and peeling
(delamination)), and for substrate (mechanical strength;
resistant to bending, compression, etc.) there is only one
report, JIS K 5400 (Testing Method of Strike Transformation for
Coating Films)(No. 14).

On thermal property four reports are found. Three reports
(No. 55, 56 and 57) are common tests about all materials used
in solar applications. Another report is the heat-resistance
test of solar absorber surfaces.

2. Respective Test Procedures

Reports requested to the participants are listed as the following
optical, thermal and reliability/service properties and durability.

(a) Optical properties

   (i)   Solar range, spectral
   (ii)   Solar range, total
   (iii) Thermal range, spectral
   (iv)   Thermal range, total

(b) Thermal properties

   (i)   Thermal conducting properties (thermal conductivity,
          etc.)

(c) Reliability / service

   (i)   Coating (mechanical strength: resistant to crazing,
          checking, cracking, flaking and peeling (delamination))

   (ii)   Substrate (mechanical strength: resistant to bending,
          compression, etc.)

(d) Durability

   (i)   Durability to stagnation temperature
   (ii)   Durability to a designated operating constant tem-
          perature (thermal degradation)
   (iii) Durability to heat cycles
   (iv)   Durability to thermal shock
   (v)   Durability to the exposure to solar radiation under
          or/and without cover plate
   (vi)   Durability to moisture and/or dew

(vii) Durability to the specific atomospheric conditions
       (including vacuum condition and polluted air condition)
(viii) Durability to salt water, acids or bases
(ix) Durability to mixed environmental conditions such as
       elevated temperature, solar radiation, moisture, etc.

(a) Optical properties

   (i) Solar range − 15 reports

      The measuring principle described in 13 reports is based on
      spectroscopic method. The measuring principles in other two
      reports are different from each other. One is the method to
      measure the total (wavelength integrated) solar reflectance
      by use of optical integrating sphere coupled with tungsten-
      halogen lamp as a solar simulator lamp. The solar reflec-
      tance is evaluated from the ratio between the total amount
      of reflected rays from the specimen for the incident rays
      from the lamp and those from the standard sample such as
      metal coated mirror, $BaSO_4$ plate and black mat painted
      specimen. The other is based on calorimetric measurement
      (ASTM E 434-71). This describes measurement of the radia-
      tive heat gains and losses from the specimen in vacuum
      coupled with cold shroud under the illumination of the
      sample by a equipped solar simulator.

   (ii) Thermal range − 13 reports

      There are four methods in the principle of measurement.

         1. Emittance determined by measuring spectral reflectance
         2. Emittance determined by radiative measurement of the
            spectra of heated samples
         3. Emittance determined by measuring the radiation from
            the samples as the electromotive force
         4. Emittance determined by measuring temperature of
            heated samples in vacuum solar simulator (calorimetric
            method)

      In these reports relations between the emittance and the
      radiation angle are described on following items.

         1. Normal emittance
         2. Angular dependence at the angular range from $0°$ to $70°$

               Using integrated sphere
               Integrating emittance measured each $5°$ interval
               over all emitting angles from $15°$ to $75°$

   In all reports, each accuracy is not exceeding 5 %.

This result means that these methods are practical and relevant.

(b) Thermal properties - 4 reports

1. Determination of heat of fusion and crystallization by differential scanning calorimetry.
2. Determination of melting and crystallization temperatures by thermal analysis.
3. Determination of the linear thermal expansion of solid materials by thermodilatometry.

(c) Reliability / service - 2 reports

One is a testing method of strike transformation for coated films. This report is referred to JIS (Japan Industrial Standard) K 5400, "Testing Methods for Organic Coating". The other is a climatic cycle test.

(d) Durability - 24 reports

Evaluation of the test results in all reports is made by measuring the change of the optical property (the absorptance and the emittance) prior to and after the applied testing.

As far as concerned with the respective items listed, we have several reports. It is difficult to extract the common conditions applied among those reports.

We have 10 reports entitled as "the durability to moisture and/or dew" which are the highest number among the reports. For such testing items ambient temperature, relative humidity, and test period are very important factors, and the following conditions are found in the contributed reports.

No.	Ambient temp.	Relative humidity	Test period
1	90 °C	95 %	10 weeks
2	25 °C	100 %	4 weeks
3	23 °C	95 %	6 weeks
4	50 °C/95 °C	100 %	4 weeks
5	49±1 °C	95 %	?
6	50 °C	98 %	1,000 hours
7	90 °C	95 %	30 days

All of the test methods are classified into 6 items, which are thermal property, moisture and/or dew, specific atomospheric conditions, salt water spray, outdoor test, and mixed environmental conditions (Table 3).

Some reports are referred as the industrial standards.

3. Proposals on Future Cooperative Works

As for the cooperating works, we propose the accelerated aging test at constant temperature for solar absorber coatings.

Reports Contributed by the Subtask B Participants
- ABSORBER -

List of Items	Coding	FRG	Japan	Netherlands	Sweden	Switzerland	UK	USA
a-(i)	AC-O	○1	○3 ○10 ○11 ○12 ○13	○25	○37 ○36	○27	○48	●42 ●44
a-(ii)	AC-O	○4	○2					
a-(iii)	AC-O	○5	○6				○50	●46
a-(iv)	AC-O	○7	○9	○26	○35	○28	○49	●41 (●46) ●55 ●56 ●57
b-(i)	AC-T	○8					○53	
c-(i)	AC-R		◎14	○29				
c-(ii)	AS-R							
d-(i)	AC-D	○16	○15		○32	○40	○52	●45
d-(ii)	AC-D		○17					
d-(iii)	AC-D		○18					
d-(iv)	AC-D		●19					
d-(v)	AC-D		●20					(○45)
d-(vi)	AC/AS-D		○21		◎33 ●31 ○34 ○30	○39 ○38	(○52)	
d-(vii)	AC-D		●22				○54	●43
d-(viii)	AC/AS-D		◎24					
d-(ix)	AC-D		○23	(○29)			○51	(●45)

Note: ● ST (Standardized method   ( ) referred from testings on other materials)
       ○ LA (Laboratory method)   ◎ Solar materials ST

Table 1.   Laboratory and Standard Test Methods.

Material category Properties to be tested or measured	L A			S T		
	A. C	A. S	A	A. C	A. S	A
Optical properties	2,3,6,9,10, 11,12,13, 25,26,27, 28,48,49, 50	6,11,12,13, 25,26,48, 49,50	1,4,5,7,8, 35,36,37	(1), 41,42, 44,46,47	44,46,47	
Thermal properties	53	53				55,56,57
Reliability/service	29	29		14		
Durability	15,17,18, 21,23,29, 30,32,34, 51,52,54	29,51,52, 54	16,38,39, 40	19,20,22, 24,31,33, 45	20,24,31, 43,45	

LA ; Laboratory method   ST ; Standardized method
A.C; Absorber coating   A.S; Absorber substrate
A ; Absorber not identified both in A.C and A.S categories

Table 2. Precision about the Test Method of Optical Properties.

Properties		Apparatus	Precision	Concerned report
Solar range	Spectral	Spectrophotometer	0.01 ~ 0.5%	1, 3,10,11,12,13,25, 27,36,37,42
			2%	44,48
	Total	Alpha meter	0.5%	2
		Calorimetric	1 ~ 2%	46
Thermal range	Spectral	Spectrophotometer	0.02 ~ 0.5%	4, 5, 6
			3%	50
	Total	Emmisiometer	0.03%	7, 8, 9,26,28,35,41
			5%	49
		Calorimetric	-----	46

Table 3. Test Method of Durability

	Apparatus	Report No.
Thermal degradation Heat cycle Thermal shock	Oven Water vessel	16,17,18,19,32,38
Moisture and/or dew	Humidity cabinet	20,21,30,31,33,34 38,39,45,52
Specific atomoshperic conditions	Weather meter	22,33
	Xenon arc	45
	Ultraviolet	54
	Ferric chloride solution	43
Salt water spray	Salt water spray test cabinet	23,24
Outdoor test	Test stand	15,40,45,51
Mixed environmental conditions	Cycle test chamber	29

*34 Method for testing resistance to sulfur dioxide.

Table 4.　　List of Title & Reporter's Name

No.	Title	Reporter's Name
1.	Spectral absorptance for directional incidence (solar range)	K. Gindele
2.	Testing method for absorptance of selective surface coating (by the α meter)	K. Taga
3.	Testing method for absorptance of selective surface coating (by spectrum photo meter)	K. Taga
4.	Spectral directional emittance (infrared range) I (reflection method)	K. Gindele
5.	Spectral directional emittance (infrared range) II (radiometric method)	K. Gindele
6.	Measurement of normal spectral emittance of absorber & glazing material	T. Tanemura
7.	Total hemispherical emittance	K. Gindele
8.	Total directional emittance	K. Gindele
9.	Testing method of emissivity of selective coating surface	K. Taga
10.	Testing method for reflectance of selective surface coating (by spectrum photo meter)	K. Taga
11.	Normal spectral reflectance (NSR) of absorber materials/coating elevated at designated temperature	T. Tanemura
12.	Absolute normal spectral reflectivity (AB-NSR) of absorber material with mirror like smooth surface	T. Tanemura

No.	Title	Reporter's name
13.	Hemispherical directional spectral reflectance (HDSR) of absorber materials at nearly room temperature	T. Tanemura
14.	Testing method of strike transformation for coating films	T. Odaira
15.	Testing method for stagnation of absorber plate	K. Taga
16.	Accelerated aging test at constant temperature for solar absorber coatings	M. Kohl
17.	Testing method of heat resistance of absorber plate	K. Taga
18.	Testing method for heat cycles of absorber plate	K. Taga
19.	Methods of thermal shock test for the selective coating absorber	T. Odaira
20.	Damp heat (constant) testing method for the selective coating absorber	T. Odaira
21.	Testing method for humidity resistance of absorber plate	K. Taga
22.	Testing method of weatherability for selective coating films	T. Odaira
23.	Salt water spray testing method of absorber plate	K. Taga
24.	Method of salt water spray test for the selective coating absorber	T. Odaira
25.	Determination of the solar absorptance of a sample at near normal irradiance	P. A. Nijnatten
26.	Determination of directional and hemispherical total emittance at temperatures of 60°C to 200°C	P. A. Nijnatten
27.	Spectral reflectance/absorptance (hemispherical irradiation, normal detection) of absorber coatings	J. Keller

No.	Title	Reporter's name
28.	Thermal emittance of absorber coatings (hemispherical)	J. Keller
29.	Climatic cycle test	A. J. Faber
30.	Method for testing absorbers for flat plate solar collectors at 90°C and 95% relative humidity	H. Wennerholm
31.	Method for testing absorbers for flat plate solar collectors at 25°C and 100% relative humidity	H. Wennerholm
32.	Method for testing thermal stability of absorber and coating for flat plate solar collectors	H. Wennerholm
33.	Method of testing resistance to UV-radiation and moisture of absorbers for flat plate solar collectors	H. Wennerholm
34.	Method for testing resistance to sulphur dioxide at high relative humidity of absorber and coating for flat plate solar collectors	H. Wennerholm
35.	Measurement of emittance at 100°C temperature, normal	
36.	Measurement of reflectance 15°/diffuse, spectral	
37.	Measurement of solar absorptance normal/diffuse, spectral	
38.	Degradation of absorber coatings by exposition under constant temperature	U. Frei
39.	Degradation of absorber coatings by exposition under constant temperature and high humidity	U. Frei
40.	Degradation of absorber coatings by exposition under solar irradiation	U. Frei
41.	ASTM E 408	
42.	ASTM E 903	

No.	Title	Reporter's name
43.	Pitting and crevice corrosion resistance of stainless steels and related alloys by the use of ferric chloride solution	D. Waksman
44.	Solar absorptance, reflectance, and transmittance of materials using integrating spheres	C. Hauer
45.	Evaluating solar absorptive materials for thermal applications	C. Hauer
46.	Calorimetric determination of hemispherical emittance and the ratio of solar absorptance to hemispherical emittance using solar simulation	C. Hauer
47.	Total normal emittance of surfaces using inspection meter techniques	C. Hauer
48.	Determination of the solar absorptance/reflectance/transmittance from spectral measurements at near normal incidence	M. G. Hutchins
49.	Measurement of near-normal total thermal emittance using a pyro-electric radiometer	M. G. Hutchins
50.	Measurement of thermal emittance from specular reflectance using an infrared spectrophotometer	M. G. Hutchins
51.	Outdoor exposure to stagnation conditions for solar absorbers and solar glazings	M. G. Hutchins
52.	Durability testing of solar absorbers by exposure to controlled conditions of temperature and moisture	M. G. Hutchins
53.	Furnace heating in air of solar absorber surfaces	M. G. Hutchins
54.	Exposure to ultraviolet radiation and moisture	M. G. Hutchins

No.	Title	Reporter's name
55.	Heats of fusion and crystallization by differential scanning calorimetry	D. Waksman
56.	Melting and crystallization temperatures by thermal analysis	D. Waksman
57.	Linear thermal expansion of solid materials by thermodilatometry	D. Waksman

III. HEAT TRANSFER MEDIA WITH OR WITHOUT ADDITIVES

1. Introduction

Many solar systems employ a closed loop circulation with some kind of heat transfer fluid for transferring heat from the collectors to the storage tank. Low freezing point fluids such as propylene glycol or ethylene glycol solutions, silicone oils, fluorocarbons, etc. have been used as heat transfer fluids to avoid the freezing problem.

In the Subtask B activity of the first year, test procedures and measurement techniques were surveyed to identify the present state-of-the-art and reviewed, but evaluation of additives to avoid corrosion was excluded.

We have 31 reports of testing method on heat transfer medium collected from F.R.G., U.S.A. and Japan (F.R.G.: 8 reports, U.S.A.: 22 reports, Japan: 8 reports). These reports are classified to three categories. The first refers to thermal and transport properties, the second to physical properties and the third to durability.

Each of them is concerned with followings:

(1) Thermal and transport properties:

    a) Heat conductivity
    b) Specific heat
    c) Specific volume
    d) Kinematic viscosity or coefficient of viscosity

(2) Physical properties

    a) Solidifying point (freezing point)
    b) Boiling point
    c) Flashing point
    d) pH and alkalinity

(3) Durability

    a) Stability
    b) Corrosive effect on the materials

As widely used evaluation methods have already been established to some extent in regard to thermal properties of heat transfer medium, these methods can be applicable to those of solar system.

Three reports collected are concerned with the evaluation method of a solar system, but the kind of heat transfer medium raised here is rather limited.

2. Respective Test Procedures

The performance and reliability of closed systems are considerably affected by degradation of heat transfer fluid and corrosion of materials exposed to it.

Important properties of heat transfer fluid to determine the performance and reliability are:

(a) Thermal properties

    (i)   Heat conductivity
    (ii) Specific heat
    (iii) Latent heat (in the case of phase change heat transfer
                medium)

(b) Transporting properties

    (i)   Specific volume
    (ii) Kinematic viscosity or coefficient of viscosity

(c) Reliability

    (i)   Solidifying point
    (ii) Boiling point
    (iii) Flashing point
    (iv) Toxicity and human hazards

Moreover, some accelerated and simplified tests are required to determine durability.

We introduce three reports of test procedures on life prediction.

(1) Aging of aqueous fluids and internal corrosion of solar systems:

Electrochemical check is applied for the detection of undesirable changes of the properties of the heat transfer media after a period of time. This is Tafel's extrapolation method on heated rotating-cylinder-electrode through electrical polarization by computerized potentiostat-galvanostat at 30 C through 130 °C.

Weight loss or pitting depth, and surface state of the samples must be taken into consideration.

(2) Testing method for thermal stability of chemical structure of propylene glycol:

50 Vol % propylene glycol aqueous solution 700 ml is continuously heated at 105±1 °C during 16 weeks in deaerated condition, and then the change of both pH and infrared spectrum are checked. Both the degree of pH decrease and of the in-

frared spectrum change indicate the degradation of propylene glycol solution.

(3) Corrosion test for heat transfer medium in glassware:

Tested metal specimens are placed in the test solution 750 ml consisted of city water and heat transfer medium by equivalent volume. They are continuously heated at 88 ± 2 °C during 336 hrs. in constant aerated condition of 100 ± 10 ml per min. correct corrosion weight changes of individual specimens to nearest 0.1 mg for each test must be measured to know the corrosion inhibitive properties of test solution.

3. Proposals to Future Cooperative Works

According to the reports No. 1 – No. 6, present testing and measurement methods for evaluating the properties of heat transfer media are appropriate and practical in solar application.

Therefore, the performance, reliability and safety of heat transfer media can be determined with the present testing procedures.

But the durability is not determined with sufficient accuracy because the available information is scarce.

In general, more than 10-year life is required for a solar system, so that there is need for some kinds of accelerated test to determine the durability of it.

The reports No. 7 – No. 9 suggest the methods of accelerated and simplified tests to determine the durability.

When these methods are applied to the heat transfer media used in solar systems, it is important that the coefficient of correlation between these tests and actual use is obtained.

Therefore, the following future cooperative works associated with heat transfer media are suggested:

(a) Identification of the coefficient of correlation between actual use and accelerated tests.

(b) Establish available accelerated testing methods for determining durability.

Reports Contributed by the Subtask B Participants
-HEAT TRANSFER MEDIUM WITH OR WITHOUT ADDITIVES-

List of Items	Coding	AUSTRIA	CANADA	DENMARK	FRG	JAPAN	NETHERLANDS	SWEDEN	SWITZERLAND	USA
a-(i)	HWA/HWNA-T					○				◎
a-(ii)	HWA/HWNA-T					○				◎
a-(iii)	HWA/HWNA-T									◎
b-(i)	HWA/HWNA-TR									◎
b-(ii)	HWA/HWNA-TR					◎				◎
c-(i)	HWA/HWNA-R					◎				◎
c-(ii)	HWA/HWNA-R					◎				◎
c-(iii)	HWA/HWNA-R					◎				◎
c-(iv)	HWA/HWNA-R									◎
c-(others)	HWA/HWNA-R									◎
d-(i)	HWA/HWNA-D					○				◎
d-(ii)	HWA/HWNA-D				○	◎				◎
d-(iii)	HWA/HWNA-D									
e-(i)	HWA/HWNA-O									
e-(ii)	HWA/HWNA-O									
e-(iii)	HWA/HWNA-O									

○ Laboratory method
◎ Standardized method

Grouping of Reports

	L A	S T
Thermal Properties	1, 2	
Transporting Properties		3, 21, 22, 23, 24, 31
Reliability		4, 5, 6, 20, 25, 26, 27, 28, 29, 30
Durability	7, 9	8, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19
Optical Properties		

LA : Laboratory method

ST : Standardized method

List of Reports

No.	Title	Reporter
1.	Experimental Determination of the Thermal Conductivity of Heat Transfer Medium with Low Electrical Conductivity	T. Asahina
2.	Measuring Method for Specific Heat of Heat Transfer Medium	M. Koga
3.	Testing Method of Kinematic Viscosity of Heat Transfer Medium	T. Sawada
4.	Testing Method for Solidifying Point of Heat Transfer Medium	T. Sawada
5.	Testing Method for Boiling point of Heat Transfer Medium	T. Sawada
6.	Testing Method for Flash Point of Heat Transfer Medium	T. Sawada
7.	Testing Method for Thermal Stability of Chemical Structure of Propylene Glycol	M. Koga
8.	Corrosion Test for Heat Transfer Medium in Glassware	M. Koga
9.	Aging of Aqueous Fluids and Internal Corrosion of Solar Systems	K. H. Tostmann
10.	Laboratory Screening of Metallic Containment Materials for Use with Liquids in Solar Heating and Cooling Systems Practice A: Basic Immersion Test at Atmospheric Pressure	D. Waksman
11.	Laboratory Screening of Metallic Containment Materials for Use with Liquids in Solar Heating and Cooling Systems Practice B: Heat-Rejecting Surface Test at Atmospheric Pressure	D. Waksman
12.	Laboratory Screening of Metallic Containment Materials for Use with Liquids in Solar Heating and Cooling Systems Practice C: High-Pressure Test	D. Waksman

No.	Title	Reporter
13.	Laboratory Screening of Metallic Containment Materials for Use with Liquids in Solar Heating and Cooling Systems Practice D: Repeated Dip Dry Test at Atmospheric Pressure	D. Waksman
14.	Laboratory Screening of Metallic Containment Materials for Use with Liquids in Solar Heating and Cooling Systems Practice E: Crevice Test at Atmospheric Pressure	D. Waksman
15.	Laboratory Screening of Metallic Containment Materials for Use with Liquids in Solar Heating and Cooling Systems Practice F: Test Loop at Atmospheric Pressure	D. Waksman
16.	Simulated Service Testing for Corrosion of Metallic Containment Materials for Use with Heat-Transfer Fluids in Solar Heating and Cooling Systems Practice A: Laboratory Exposure Test for Coupon Specimens	D. Waksman
17.	Simulated Service Testing for Corrosion of Metallic Containment Materials for Use with Heat-Transfer Fluids in Solar Heating and Cooling Systems Practice B: Laboratory Exposure Test of Components or Subcomponents	D. Waksman
18.	Simulated Service Testing for Corrosion of Metallic Containment Materials for Use with Heat-Transfer Fluids in Solar Heating and Cooling Systems Practice C: Field Exposure Test of Components or Subcomponents	D. Waksman
19.	Heat of Fusion and Crystallization by Differential Scanning Calorimetry	D. Waksman
20.	Melting and Crystallization Temperatures by Thermal Analysis	D. Waksman
21.	Kinematic Viscosity of Transparent and Opaque Liquids	D. Waksman
22.	Rheological Properties of Materials by Rotational (Brookfield) Viscometer Method A: Apparent Viscosity	D. Waksman
23.	Viscosity of Transparent Liquids by Bubble Time Method	D. Waksman

No.	Title	Reporter
24.	Flash Point by Tag Closed Tester	D. Waksman
25.	Flash Point by Pensky-Martens Closed Tester	D. Waksman
26.	Freezing Point of Aqueous Coolant Solutions	D. Waksman
27.	Boiling Point of Coolants	D. Waksman
28.	Autoignition Temperature of Liquid Chemicals	D. Waksman
29.	PH of Antifreezes, Antirusts, and Coolants	D. Waksman
30.	Reserve Alkalinity of Antifreese, Antirusts, and Coolants	D. Waksman
31.	Apparent Specific Gravity of Liquid Industrial Chemicals	D. Waksman

IV. THERMAL ENERGY STORAGE MATERIALS

1. Introduction

At the last expert meeting in Vienna it was determined to take up PCM and chemical storage materials for thermal energy storage materials and desiccant materials.

We have collected twenty reports on testing and measurement methods for thermal properties, reliability and durability of latent heat thermal storage materials.

The titles of the reports and grouping of the reports are shown in the supplemental tables respectively.

There are many reports regarding thermal properties (one or more reports for almost every item), but few concerning reliability and durability. The greater part of the reports is from Japan (10 reports) and U.S.A. (7 reports). From other countries three reports are contributed. There are twelve standardized methods based on JIS (Japan Industrial Standard) and ASTM about thermal properties and reliability.

There are not many reports which mention the precision of the test method, but some reports on thermal properties or reliability refer to their precision. As most of these test methods are standardized in JIS or ASTM, the precisions indicated in these reports would also be enough for industrial use.

2. Respective Test Procedures

Important properties to evaluate the performance of latent heat thermal storage materials are shown in the following list:

(a) Thermal Property

(i)   Phase change temperature
(ii) Latent heat
(iii) Specific heat
(iv) Heat conductivity
(v)   Heat diffusivity
(vi) Density of solid phase
(vii) Density of liquid phase

(b) Reliability

(i)   Vapor pressure at a designated temperature

(ii)   Ignition point
(iii) Flash point
(iv)   Content of toxic materials
(v)   Toxicity and human hazards (toxic)

(c) Durability

(i)   Durability for heat cycles
(ii)   Durability at a designated temperature (thermal degradation, chemical stability)
(iii) Corrosive effect on the container vessel

(a) Thermal Property

(i)   Phase change temperature

(Melting point: Report No. 1 and 18)

Melting point is defined as the temperature or the range of temperature where the solid sample fully melts. The sample in a capillary glass tube is heated in a bath and the melting point is determined from the temperature at which the solid sample disappears. This method is standardized in JIS (JIS K 0064).

Method of determining the melting point of waxes is reported in Report No. 18. This method is characterized by using a microscope to observe melting, and is standardized by ASTM (ASTM F 766). Precision of repeatability is less than 2 °C.

(Freezing point: Report No. 2)

Freezing point is measured by cooling the material in the bath of water, ice, or mixture of water and sodium chloride. By measuring the temperature change of the material, freezing point is determined from the temperature where the falling rate of temperature changes. This method is standardized in JIS (JIS K 0065). Precision of repeatability is less than 0.2 °C.

(Thermal analysis: Report No. 15)

Transition temperatures such as the melting and crystallization temperatures of materials can be determined by differential scanning calorimetry (DSC) and differential thermal analysis (DTA).

The method consists of heating or cooling the test material at a controlled rate in a controlled atmosphere through the region of a transition, such as fusion or crystallization. The difference in heat-flow or temperature between the test material and a reference material due to energy changes in the material is continuously monitored and recorded. Absorption or release of energy results in endothermic or exothermic peaks. From the resultant curve, melting and crystallization temperatures are measured.

(ii) Latent heat

(Differential scanning calorimetry: Report No. 3 and 14)

By heating a sample and the standard material separately, the difference of heating energy required to equalize the temperature of both the sample and the standard material is measured. From the measured energy, the phase change energy of the sample can be calculated.

Precision is typically 5 %.

This method is standardized in ASTM (ASTM E 793 and D 3417).

(Quantitative DTA: Report No. 11)

A heat flow sensor is mounted on a metal block. This metal block can be warmed up or cooled down with a constant heating/cooling rate. On the heat flow sensor a platform is mounted on which a sample holder can be placed. The heat necessary for the filled or unfilled sample-holder to follow the temperature of the block is measured directly by the heat flow sensors. The metal block is in principle provided with two heat flow sensors with platforms. On one of them an empty sample holder is placed, on the other a sample holder filled with sample-material. The difference between thus measured heat flows corresponds to the net heat flow to the sample.

The heat effect like melting, solidification, hydrating, etc. can be quantified by determining the energy which corresponds with the area size between the heat effect-curve and the interpolated specific heat-curve between beginning and end of the effect (the base-line).

(iii) Specific heat

(Adiabatic scanning calorimetry: Report No. 4)

A sample cell is thermally insulated from the environment. In order to achieve this condition a vessel which contained the cell is heated to the temperature same to that of the sample. The specific heat of the sample is determined from the ratio of the heating energy and the temperature rise multiplied by the weight of the sample.

Precision of the method is less than 3 %.

(Quantitative DTA: Report No. 11)

Principle of the instrument is described above ((a)-(ii)). From the recorded curve, the known calibration-value of the heat flow-sensors, and the weights of the samples, the specific heat can be calculated.

(iv) Heat conductivity

(Solid storage materials: Report No. 5)

A sample is heated stepwise by light emitted from a Xenon arc lamp. the heat diffusivity of the sample is determined from the temperature variation of the sample.

Since the measurement is carried out in vacuum, the heat loss can be neglected under temperature of 400 °C. Therefore the high precision is expected. The time of measurement is usually a few seconds.

(Liquid storage materials: Report No. 8)

A sample in a metal container is heated stepwise by light emitted from a halogen lamp. The heat diffusivity of the sample is determined from the temperature variation of the container. since the measurement is carried out in vacuum, the heat loss can be neglected under temperature of 400 °C. Therefore the high precision is expected. The time of measurement is usually a few seconds.

(v)   Heat diffusivity: No reports

(vi) Density of solid phase

(Report No. 7)

The specific gravity of the storage material is measured with the use of a specific gravity bottle. The density of water at 4 °C and 1.013250 bar.

Precision of repeatability is less than 1 with the least significant digit. This method is standardized in JIS (JIS Z 8807).

(vii) Density of liquid phase

(Report No. 8 and 20)

The specific gravity of the storage material is measured with the use of a specific gravity bottle. The density of the material is equal to the specific gravity multiplied by the density of water at 4 °C and 1.013250 bar.

Precision of repeatability is less than 0.0002.

This method is standardized in JIS (JIS Z 8804).

Similar methods using a hydrometer or a pycnometer are reported in Report No. 20. These methods are standardized by ASTM (ASTM D 891).

(b) Reliability

(i)   Vapor pressure at a designated temperature: No reports.

(ii) Ignition point

(Report No. 19)

The autoignition temperature is the lowest temperature at which a flame and a sharp rise in temperature occur when a chemical is inserted into a heated glass flask containing air at a predetermined temperature. Hot-flame temperatures are evidenced by the sudden appearance of a flame and a sharp rise in temperature. Cool-flame ignitions are evidenced by small sharp rises in temperature and a faint, pale blue luminescence or flame.

This method is standardized by ASTM (ASTM E659). Precision of repeatability is less than 2 %. Precision of reproducibility is less than 5 %.

(iii) Flash point

(Cleveland open method: Report No. 9)

This method is applied for materials flash points of which are higher than 80 °C.

Sample contained in an open cup is heated at a defined rate of temperature rise. In the vicinity of the flash point, a defined size of small fire is passed over the cup ever 2 °C temperature rise. Then the lowest temperature where the sample flashes is measured.

This method can be also applied for measurement of burning point where a sample burns continuously.

Precision of repeatability is less than 8 °C.

Precision of reproducibility is less than 16 °C.

This method is standardized in JIS (JIS K 2265).

(Tag closed tester: Report No. 16)

This test method covers the determination of the flash point, by Tag closed tester, of liquids with a viscosity of below 5.5 cSt at 40 °C, or below 9.5 cSt at 25 °C, and a flash point below 93 °C with the exception of cutback asphalts, those liquids which tend to form a surface film under test conditions, and materials which contain suspended solids.

The principle of the method is the same as above method.

Precision of repeatability is less than 1.1 °C for   60 °C or 1.7 °C for 60 ~ 93 C.

Precision of reproducibility is less than 3.3 °C for 13 °C and 60 ~ 93 °C, or 2.2 °C for 134 ~ 59 °C.

(Pensky-Martens closed tester: Report No. 17)

These test methods are primarily used to determine the flash point of liquids that either have viscosities more than 5.5 cSt at 40 °C, or 9.5 cSt or more at 25 °C, or that have a flash point greater than 93 °C, or that contain suspended solids or tend to form a surface skin under test conditions.

The principle of the methods is the same as above two methods.

These methods are standardized by ASTM (ASTM D 93).

Precision of repeatability is less than 5.5 °C for any case.

Precision of reproducibility is less than 10 °C for any case.

(iv) Content of toxic materials: No reports.

(v) Toxicity and human hazards (toxic): No reports.

(c) Durability

(i) Durability for heat cycles

(Report No. 10)

A storage material contained in a container is cyclical-
ly heated and cooled in a water bath. During the test
the temperature of a material is measured to assure the
durability in a desired testing condition.

The upper temperature of the heat cycle must be higher
than the phase change temperature of the materials and
it is the same for the lower temperature of the heat
cycle to some extent.

The durability of a material is evaluated in terms of
thermal properties, such as specific heat, latent heat,
phase change temperature, chemical composition, and so
on.

(Report No. 13)

As for the salt hydrates, detailed procedure is
reported. Possible phenomena that influence on the long
time behavior of a particular PCM (salt hydrate) are:

Chemical degradation
Interaction with container materials
Segregation
Undercooling

To investigate the influence of these phenomena on the
PCM, the aging test is carried out on several samples of
1 - 4 kg PCM, packed in steel tube containers that are
sealed by means of welding.

To examine the influence of gravity on segregation one
sample is placed vertically, one horizontally, and one
vertically with turning it upside down every 14 days.

The temperature of the samples is cycled, as such 4
hours high, 4 hours low, etc. and the heat input and
output are continuously measured.

The results are latent heat values as a function of time and number of cycles. One year aging test = 1,000 cycles gives a good prediction of the long time behavior if the cycling is the dominant aging factor (this is expected for salt hydrate PCM's). However, if the total time on high temperature is most important, tests have to be even longer to predict the behavior of the PCM during the storage life time. Shorter testing by using higher temperatures seem to be not feasible because then new physical and chemical problems are introduced.

(Report No. 12)

A rapid experimental procedure for investigating the suitability of salt hydrates to be used in chemical heat pumping is proposed. Thermogravimetry under controlled water vapor pressure is utilized as the key diagnostic method. The test procedure relied largely on two critical tests, namely a cycling and a inhibition test. The former gives information on the stoichiometic reversibility and hysteresis between the dehydration and rehydration branches, while the inhibition test yields more quantitative information about the extent of inhibition. The latter represents a source of irreversibility inherent to the salt hydrate system.

(ii) Durability at a designated temperature (thermal degradation, chemical stability): No reports.

(iii) Corrosive effect on the container vessel: No reports

3. Proposal on Future Cooperative Works

As to the thermal properties, test procedures are almost established and some of them are standardized. Therefore, these test methods would be adopted.
As for the reliability, only four reports regarding ignition point and flash point are presented. No reports are found for the other three items (vapor pressure at a designated temperature; content of toxic material; and toxicity and hazards (toxic)). But, for these items, existing test methods for other materials would be found. More research for the suitable methods on these items might be necessary.

As to the durability, three reports concerning durability for heat cycles are presented. For the other two items (durability at a designated temperature; and corrosive effect on the container vessel), no reports are found. As no suitable methods seem to be found for these two items, they had better be investigated taking

them up as the cooperative works. Durability for heat cycles is thought to be the most important property for latent heat thermal storage materials, so it would be desirable to investigate it for setting up the standard test method.

Therefore, we propose the following three items concerning durability as future cooperative works:

(1) Test methods of durability for heat cycles

(2) Test methods of durability at a designated temperature (thermal degradation, chemical stability)

(3) Test methods of corrosive effects on the container vessel.

Reports Contributed by the Subtask B Participants
- STORAGE MATERIALS, LATENT -

List of Items	Coding	AUSTRIA	CANADA	DENMARK	FRG	JAPAN	NETHERLANDS	SWEDEN	SWITZERLAND	USA
2-a-(i)	SL-T					◎				◎◎
2-a-(ii)	SL-T					○				◎
2-a-(iii)	SL/SS-T					○	○			
2-a-(iv)	SL/SS-T					○				
2-a-(v)	SL-T									
2-a-(vi)	SL/SS-T					◎				
2-a-(vii)	SL/SS-T					◎				◎
2-b-(i)	SL-R									
2-b-(ii)	SL-R									◎
2-b-(iii)	SL/SS-T					◎				◎◎
2-b-(iv)	SL-R									
2-b-(v)	SL-R									
2-c-(i)	SL/SS-D					○	○	○		
2-c-(ii)	SL-D									
2-c-(iii)	SL-D									

- 57 -

Table 1. Grouping of Reports

	L A	S T
Thermal Properties	3, 4, 5, 6, 11	1, 2, 7, 8, 14, 15, 18, 20
Reliability		9, 16, 17, 19
Durability	10, 12, 13	

Table 2.   List of Reports

No.	Title	Reporter	Country
1.	Testing Method for Melting Point of Storage Materials	K. Matsuki	Japan
2.	Testing Method for Freezing Point of Storage Materials	=	=
3.	Measurement Method of Latent Heat of Storage Materials (Differential Scanning Calorimetry)	=	=
4.	Measurement Method of Specific Heat of Storage Materials (Adiabatic Scanning Calorimetry)	=	=
5.	Measurement Method of Thermal Conductivity of Solid Storage Materials	=	=
6.	Measurement Method of Thermal Conductivity of Liquid Storage Materials	=	=
7.	Measurement Method of Specific Gravity of Storage Materials in Solid Phase	=	=
8.	Measurement Method of Specific Gravity of Storage Materials in liquid Phase	=	=
9.	Testing Method for Flash Point of Storage Materials (Cleveland Open Method)	=	=
10.	Testing Method of Durability of Storage Materials for Heat Cycles	=	=
11.	The Quantitative Test of Thermal Qualities as Specific Heat, Melting Heat, Hydratation Heat, of Phase Change Materials (PCM)	F. van der Graaf	Netherlands
12.	Chemical Heat Pumping - A Rapid Experimental Procedure for Investigating the Suitability of Salt Hydrates under Dynamic Conditions	Bo. Carlsson	Sweden

No.	Title	Reporter	Country
13.	Aging Tests of Representative Samples by Means of Thermal Cycling under Realistic Conditions	P. Kratz	Netherlands
14.	Heat of Fusion and Crystallization by Differential Scanning Calorimetry	D. Waksman	U.S.A.
15.	Melting and Crystallization Temperatures by Thermal Analysis	"	"
16.	Flash Point by Tag Closed Tester	"	"
17.	Flash Point by Pensky-Martens Closed Tester	"	"
18.	Melting Point of Waxes	"	"
19.	Autoignition Temperature of Liquid Chemicals	"	"
20.	Apparent Specific Gravity of Liquid Industrial Chemicals	"	"

APPENDIX   I

REPORTS BY THE PARTICIPANTS

REPORTS ON SOLAR ABSORBERS

a. OPTICAL PROPERTIES

a (i) SOLAR ABSORPTANCE

REPORTING FORMAT

Date: 2/86   Reporter's Name: K. Gindele

Country: F.R.G.

Testing Title: Spectral absorptance for directional incidence
               (solar range)

Abstracted Scope (200 words):

Measurement of the spectral absorptance for near-normal
directional incidence using the reflexion technique
($\alpha(\lambda) = 1 - \varsigma(\lambda)$). The spectral reflectance is measured
in the spectral range from 0.36 μm to 2.5 μm. The
photometric integration is done with a $BaSO_4$-coated
integrating sphere.

Sample temperature: room temperature.

Referred Document:

The measuring device is near to the German standard
DIN 5033.

Principle of Measurement:

Spectral measurement of the hemispherically reflected flux with an integrating sphere.

Apparatus:

Measuring geometry: near-normal directional irradiation at an angle of 8°, detection of the hemispherically reflected radiation with a $BaSO_4$-coated integrating sphere. Use of "substitution method", that means sample and reference are successively attached to the sphere ports.

Spectrometer system: Prism double monochrometer.

Calibration: Pressed $BaSO_4$-powder is used as reference.

Sampling: Samples are put on the sphere port.

Conditioning: None

Procedures:

The energy distributions of the sample A_s and of the reference A_r are measured successively. The sample spectrum is related to that of the reference with relation $*$.

Afterwards we calculate the solar absorptance.

Calculation Method (if necessary, please include correction method):

$$\varrho_s (\lambda) = \frac{A_s (\lambda)}{A_r (\lambda)} \varrho_r (\lambda) \qquad *$$

Interpretation/Precision & Accuracy:

+/- 0.01   (with restrictions for   2 um)

For samples of which the reflected radiation is strongly polarized, both directions of polarization have to be measured separately.

Date: February 19, 1986   Reporter's Name: J. Keller, EIR,
                                            CH-5303 Würenlingen

Country: Switzerland

Testing Title:

Spectral reflectance/absorptance (hemispherical irradiation, normal detection) of absorber coatings

Abstracted Scope (200 words):

Measurement of the spectral reflectance of absorber coatings in the range of wavelengths from 0.3 μm to 2.5 μm using a Zeiss PMQII/RA3 spectrometer. The irradiation is hemispherical and the detection of the reflected radiation is normal. An average value of the reflectance (or absorptance) over the above range of wavelengths is obtained by weighing the output by an appropriate solar spectrum.

Referred Document:

- Instruction Manual. Zeiss PMQII
- J. Keller, V. Kyburz, Th. Fisch, "Wärmeerträge und Kenngrössen von Sonnenkollektoren. Bericht über die Prüfkampagne vom Sommer 1983". Eidg. Institut für Reaktorforschung (EIR), CH-5303 Würenlingen. Mai 1984

Principle of Measurement:

See "procedures"

Apparatus: Zeiss PMQII/RA3 Spectrometer

1: integrating sphere, 2: absorber sample, 3: shielding, 4: light source,
5: tipping mirror, 6: lenses, 7: variable inlet slit, 8: prism, 9: detectors
(photomultiplier, photoresistor)

Calibration: white reference sample with known spectral reflectance

Sampling:

sample size about 7 cm x 7 cm

Conditioning:

Procedures:

The integrating sphere of the spectrometer is illuminated by a light source. The sample is put upon the circular opening at the top of the sphere. Opposite to the sample there is a tipping mirror which transmits the beam originating either from the sample or from a section of the sphere to a variable inlet slit. This beam is spectrally split by a prism and detected by a photomultiplier ($\lambda < 0.6$ µm) or a photoresistor ($\lambda > 0.6$ µm). For every wavelength, in the first step the mirror is pointed at the section of the wall. The inlet slit is varied until the amplifier output of the detector reaches a given value of 100 %. In the second step the mirror is tipped towards the sample and (at fixed position of the slit), the amplifier output U is read.

Calculation Method (if necessary, please include correction method):

reflectance ρ: $\rho(\lambda) = U(\lambda) \cdot f(\lambda)$

                        (f: calibration function)

absorptance α: $\alpha(\lambda) = 1 - \rho(\lambda)$

average absorptance $\bar{\alpha}$
over the solar spectrum I_λ:

$$\bar{\alpha} = \frac{\displaystyle\int_{0.3\,\mu m}^{2.5\,\mu m} I_\lambda \alpha(\lambda)\,d\lambda}{\displaystyle\int_{0.3\,\mu m}^{2.5\,\mu m} I_\lambda\,d\lambda}$$

(values of I_λ: see annex)

Interpretation/Precision & Accuracy:

accuracy: $\pm\ 2$ %

Designation :
AC–O–LA

Date : 03/03/85

Reporter's Name : Kazuo Taga

Country : Japan

Testing Title :

Testing Method for Absorptance of Selective Surface Coating
(by the α meter)

Abstracted Scope (200 words) :

Testing method for solar absorptance is considered to use the
instrument which is called α -meter   manufactured by International
Technology Corp..
The data obtained by this method is necessary to be corrected by
the data gained by the spectrum photo meter.

Referred Document :

Y.Shimajiri,ALUTOPIA, Vol.12, No.8, 9(1982)

Principle of Measurement:

The solar absosorptance is determined by the amount of reflection rays from the specimen which is subjected to the rays of halogen-tungsten lamp.

Apparatus: α meter

International Technology Corporation Model 2150

1. silicon detector
2. specimen
3. reflection rays
4. integrated sphere
5. attachment
   to the halogen
   lamp projector
6. incideut rays
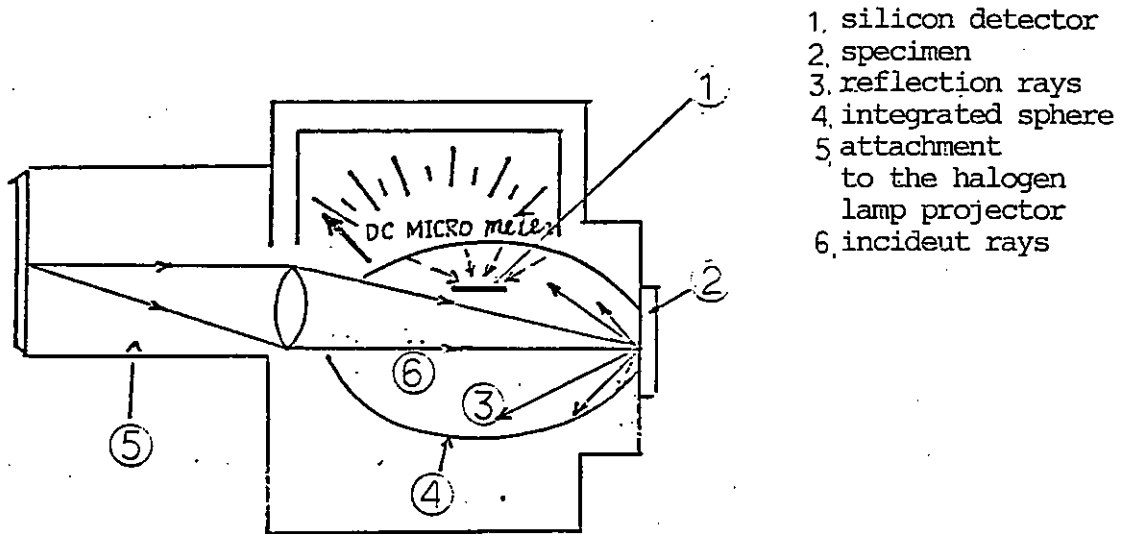
Calibration:

Sampling:

The specimen is necessary to be flat plate and its diameter above 12mm.

Conditioning:

Procedures:

1. The specimen is subjected to the rays of halogen-tungsten lamp.

2. The amount of the reflection rays are gathered by the integrated sphere and are detected by the silicon detector.

3. The absorptance of the sample is determined from the result of this test compared to the result of using MgCO3 plate (98% reflectance) and the result of nothing sample (0% reflectance).

Calculation Method (if necessary, please include correction method):

The absorptance is the reading value of the X -meter.
Its value is necessary to be corrected by the value of the spectrum photo meter.

Interpretation/Precision & Accuracy:

Accuracy : ± 2.9% of full scale
Precision : ± 0.5% of full scale

REPORTING FORMAT

Date: 03/03/86   **Reporter's Name:** Kazuo Taga

Country: Japan

Testing Title:

Testing Method for Absorptance of Selective Surface Coating
(by spectrum photo meter)

Abstracted Scope (200 words):

1. This spectrum absorptance is measured by the spectrum photo meter with integrating sphere.

2. The solar absorptance is determined by product of the spectrum absorptance and the solar spectrum intensity.

Referred Document:

ASTM E903

Standard Test Method of Solar Absorptance Reflectance, and Transmittance of Materials using Integrating Spheres.

Principle of Measurement:

The solar absorptance is determined by product of the spectrum reflectance measured by the spectrum photo meter and solar spectrum intensity in AM2.

Apparatus: spectrum photo meter

Hitachi 323 Spectrophoto meter (with Integrating Sphere)
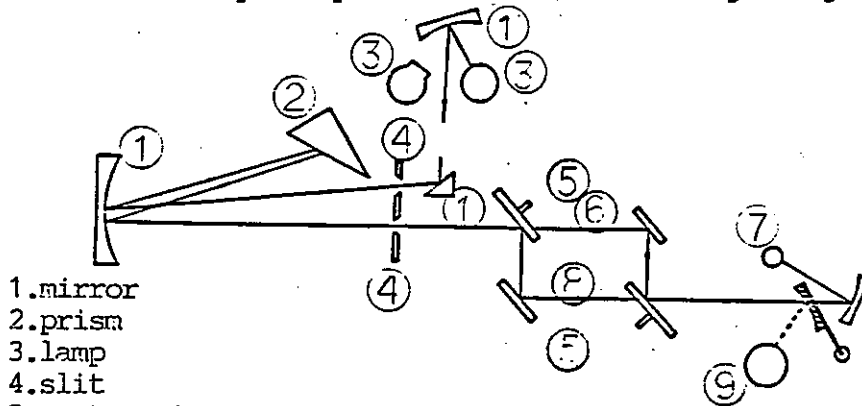
1. mirror
2. prism
3. lamp
4. slit
5. sector mirror
6. specimen side
7. pbs cell
8. standard side
9. detector
10. sample cell
11. window
12. sub white plate
13. integrating sphere   14. standard cell

Calibration:
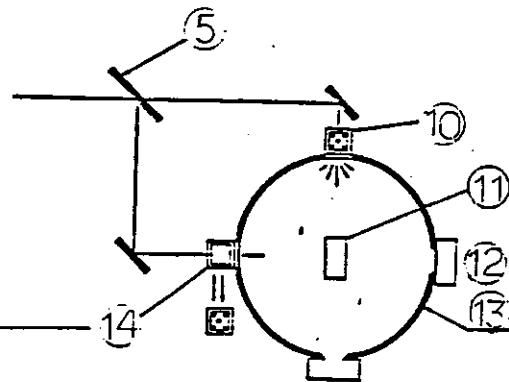
Integrating Sphere (optical flow chart)

Sampling:

20mm x 30mm (flat plate)

Conditioning:

Procedures:

1. Adjust indication of the reflectance of spectrum photo meter by using MgSO4 plate between the wave length from 340nm to 2200nm.

2. Set the specimen on the spectrum photo meter and measure it for the reflectance at 100nm wave length intervals.

3. The spectrum absorptance is calculated from
$$\alpha_\lambda = 1 - R_\lambda$$
   α_λ : spectrum absorptance
   R_λ : spectrum reflectance

4. The solar absorptance is determined by products of the spectrum absorptance and the solar spectrum intensity in air mass 2.
$$\alpha s = \frac{\Sigma \alpha_\lambda \cdot \beta_\lambda}{\Sigma \beta_\lambda}$$
   α_s : absorptance for air mass 2
   β_λ : solar spectrum intensity for air mass 2

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

Accuracy : ± 0.5%

REPORTING FORMAT   Designation:
                          A-O-LA

<u>Date</u>: 1986-03-11   <u>Reporter's Name</u>:

<u>Country</u>: Sweden

<u>Testing title</u>:

Measurement of solar absorptance
Normal/diffuse, spectral

<u>Abstracted Scope (200 words)</u>:

To determine the absorptance of solar collector absorbers by
measurement of 0/d reflectance in the wavelength region 380-2500 nm
and weighting the reflectance values with AM2 solar spectral
irradiance. This gives ρ_{AM2} $\alpha_{AM2} = 1-\rho_{AM2}$

<u>Referred Document</u>:

Principle of Measurement:

Spectral with integrating sphere on monochromatic side of
monochromator. Normal irradiation of the sample and diffuse
detection. Single beam.

Apparatus:

Monochromator:	Zeiss MM12 double prisms
Geometry:	Zeiss RA3
Source:	Zeiss with halogen lamp
Detectors:	380-800 nm wavelength: photomultiplier 800-2500 nm PbS-detector
Standard:	Pressed barium-sulfate tablet
Amplifier:	Keithley 427
Voltmeter:	HP 3455 A
Computer:	HP 216

Calibration:

Absolute scale with $BaSO_4$ standard.
Wavelength scale with spectral lamps.

Sampling:

Samples must be flat, opaque and larger than 3 x 3 cm^2.

Conditioning:

Procedures:

Measure detector output each 10 nm in region 380-800 nm for the following cases:

1. Sample port irradiated, light trap on sample port (stray radiation measurement).

2. Sphere wall irradiated, the $BaSO_4$ standard on sample port.

3. Sample port irradiated, "

4. Sphere wall irradiated, the sample on the sample port.

5. Sample port irradiated, "

At each wavelength the 1) detector output is subtracted from the 2)-5) outputs.

The sample procedure is repeated each 50 nm in the region 800-2500 nm and reflectance values interpolated to each 10 nm.

Calculation method:

At each wavelength the reflectance is calculated from the detector outputs according to:

$$\rho = \rho_s((O_5-O_1)/(O_4-O_1))/((O_3-O_1)/(O_2-O_1))$$

where ρ_s is the reflectance of the standard at the specific wavelength. Absorptance is calculated as $\alpha = 1-\int \rho s \, d\lambda / \int sd\lambda$

where s is spectral irradiance of solar radiation AM2.

Interpretation:

Precision: <±0.005   95 % confidence level

Accuracy: <±0.01   for diffuse samples
            <±0.02   for glossy samples

bs/1726L

REPORTING FORMAT

Date:	April 7, 1986 Reporter's name: P.A. van Nijnatten

Country: Technisch Physische Dienst TNO-TH
(TNO Institute of Applied Physics)
P.O. Box 155, 2600 AD DELFT
The Netherlands

Testing title: DETERMINATION OF THE SOLAR ABSORPTANCE OF A SAMPLE
AT NEAR NORMAL IRRADIANCE

Abstracted Scope (200 words):

The spectral reflectance is measured in the spectral range from
200 nm - 2400 nm with a spectro-photometer using an integrating sphere.
From there measurements, the spectral- and solar absorptance is calculated, the
latter using the spectral energy distribution of the solar irradiation
theoretically calculated by Moon [1].

Referred document:

[1] Moon, P., J. Franklin Inst., 230 (1940) 583.

Principle of measurement:

Spectral measurement of hemispherically reflected radiation.

Apparatus:

Perkin Elmer Lambda 9 double-beam, double monochromator UV/Visible/Near Infrared spectrometer with 60 mm integrating sphere attachment (B013-9941).

Calibration:

White ceramic tile (NBS standard reference material 2020).

Sampling:

Minimum sample size 10 x 20 mm

Conditioning:

Near normal incidence (8 °)

Procedures:

1. Sample is cleaned
2. Spectral reflectance $\rho_\perp (\lambda)$ is measured (10 nm intervals)
3. Spectral absorptance $\alpha_\perp (\lambda)$ is calculated
4. Solar absorptance $\alpha_\perp$ is calculated using the spectral energy distribution of the sun taken from Moon [1] with air mass 2.

Calculation Method (if necessary, please include correction method):

$$\alpha_\perp (\lambda) = 1 - \rho_\perp (\lambda)$$

$$\alpha_\perp = \frac{\int_0^\infty \alpha_\perp (\lambda) E(\lambda) \, d\lambda}{\int_\rho^\infty E(\lambda) \, d\lambda} \approx \frac{\sum_{\lambda=200 \text{ nm}}^{\lambda=2400 \text{ nm}} \alpha_\perp (\lambda) \cdot E(\lambda) \, \Delta\lambda}{\sum_{\lambda=200 \text{ nm}}^{\lambda=2400 \text{ nm}} E(\lambda) \, \Delta\lambda}$$

Interpretation/Precision & Accuracy

0,005 to 0,015 depending on the sample characteristics.

REPORTING FORMAT

Designation:
$A/G-O-ST$

Date: 9/3/86   Reporter's Name: C. Hauer
Country: USA
Testing Title: Solar Absorptance, Reflectance, and Transmittance
of Materials using Integrating Spheres

Abstracted Scope

This test method covers the measurement of spectral absorptance,
reflectance and transmittance of materials using
spectrophotometers equipped with integrating spheres. The methods
of computing solar-weighted properties from the measured spectral
values are specified. The method is applicable to materials having
both specular and diffuse optical properties.

Referred Document: ASTM E 903

The following ASTM documents are referenced:
E 424 Test Method for Solar Energy Transmittance and Reflectance
(Terrestrial) of Sheet Materials
E 490 Standard for Solar Constant and Air Mass Zero Solar Spectral
Irradiance Tables
E 772 Definition of Terms Relating to Solar Energy Conversion
E 891 Standard for Terrestrial Direct Normal Solar Spectral
Irradiance Tables for Airmass 1.5

Principle of Measurement:
Spectrophotometers equipped with integrating spheres are used to make hemispherical transmittance or reflectance measurements over the spectral range of approximately 300 to 2500 nm. The solar transmittance, reflectance or absorptance is obtained for a sample by calculating a weighted average with a standard solar spectral irradiance as the weighting function.

Apparatus:
The primary instrument required is a spectrophotometer capable of measuring in the spectral range of 300 to 2500 nm. A single beam instrument which corrects for background or a double beam, ratio recording instrument is needed. This instrument must be equipped with an integrating sphere attachment. In addition, it is recommended that reference standards having high specular reflectance, high diffuse reflectance, and low diffuse reflectance be available. These standards are available from the National Bureau of Standards as Standard Reference Materials (SRM). In addition, working standards for diffuse reflectance specimens (like barium sulfate or Halon, available from Allied Chemical Co.) and specular reflectance specimens (front surface mirrors) are required.

Calibration:

Calibration of the spectrophotometers should follow the manufacturer's procedures. Working standards should be calibrated against the NBS Standard Reference Materials.

Sampling:

While ASTM E 903 does not specify sampling procedures, three to five samples of the cover material should be used.

Conditioning:

Plastic Solar Collector cover materials should be conditioned in accordance with ASTM D 618.

Procedures:

Calibrate the linearity and wavelength scales of the spectrophotometer as recommended by the manufacturer. It is also necessary to correct for the 100% and zero line errors. For spheres with two ports, record the 100% line curves using identical high reflectance specimens in both ports. For reflectance measurements, record the zero line with a perfect absorber in the sample port. For transmittance measurements, record the zero line with the sample beam blocked. For reflectance of transparent or translucent materials having transmittance greater than .001, back the specimen with a black material having a low reflectance (<0.02) over the 300 to 2500 nm spectral range. Record the spectral reflectance of the specimen relative to the working standard by placing each in the respective port of the sphere.

Calculation Methods:
Compute the spectral reflectance using:

$$\varrho = (S - Z)/(100 - Z)(\varrho') \text{ where}$$

S = recorded specimen reading
Z = the zero line reading
100 = the 100% line reading
ϱ' = the calibrated spectral reflectance of the standard

For transmittance measurements, cover the rear ports of the sphere with the same material as the sphere. Record spectral curves without specimen in place then with specimen in place. Calculate transmittance as:
$$t = (S - Z)/(100 - Z).$$
For absorptance of non-opaque samples, calculate the absorptance using the Kirchoff relation, $\alpha + t + \varrho = 1$, based upon the measured reflectance and transmittance.

For the calculation of the solar spectral irradiance distribution, use ASTM standard E 891, calculating the optical properties using the 50 point selected ordinate method.

Interpretation/Precision and Accuracy:

Uncertainties in the results from this test method arise from random errors in measurement and sytem errors resulting from the geometry of the sphere, errors in standards values, and scattered light. The total uncertainty in the values of solar weighted optical properties is dominated by uncertainties in the spectral properties. The rms of these errors results in about a +-2% expected uncertainty.

REPORTING FORMAT

Date: 9/30/86   Reporter's Name: C. Hauer
Country: USA
Testing Title: Calorimetric Determination of Hemispherical
Emittance and the ratio of Solar Absorptance to Hemispherical
Emittance Using Solar Simulation

Abstracted Scope:

ASTM E 434-71 describes measurements of the radiative properties
of absorber materials or coatings using radiative heat gains and
losses from the specimen in vacuum. The measurements derive
absorptivity and emissivity using the radiation laws of heat
transport. They are carried out in a vacuum environment to
eliminate conductive and convective energy transport.

Referred Document: ASTM E 434-71

Principle of Measurement:

The principle on which the measurement is based is the radiant
transport of energy as defined by the law governing radiation
absorbed by a body and energy radiated by that body to an
environmeent following the rule of the fourth power of the
temperature of the body and its environment. In these measurements
a defined amount of simulated solar radiant energy is absorbed by
the test body. The rate at which this energy is reradiated is
estimated.

Apparatus:

The following apparatus is required:
* a vacuum system equipped with a cold shroud capable of attaining
a vacuum of at least .1 mPa (1 micro torr.) and maintaining a
temperature environment well below ambient around the sample.
* a shutterable aperture in the vacuum apparatus large enough to
illuminate the entire sample
* a minimum of two thermocouples capable of resolving at least .1
C and having rapid time response
* a solar simulator capable of an irradiance of at least 7000 W
per square meter
* a radiometric device for the measurement of the simulated solar
irradiance
* an interval timer capable of .1 second resolution

Calibration:

ASTM E 434 does not specify any sampling methodology. It is
suggested that thermocouples and the radiometric measurement
device should be calibrated in accordance with the manufacturer's
specifications.

Sampling:

ASTM E 434 does not specify any sampling methodology, but it is
suggested that at least five coated samples be used.

Conditioning:

No sample conditioning is specified in ASTM E 434-71

Procedures:

The test method involves suspending a sample, an absorptive coating on a substrate, mostly surrounded by a cooled constant temperature thermal shroud all in a vacuum chamber. The sample area is predicated on the area of the shroud and should be small in relation to this area. The sample substrate should be well charactized as to thermal conductivity and heat capacity. The front surface of the substrate is coated with the with the sample coating while the edges and back surface have a low emittance coating. The substrate back is fitted with a thermocouple and the thermocouple is coated with a low emittance material. The specimen is suspended in the shroud and the vacuum chaber is pumped down. Measured amounts of simulated solar irradiance are made to impinge on the sample. The thermoucouple then serves to register the specimen temperature and the rate of temperature change. From these temperature measurements, the mesured temperature of the thermal shroud and the knowm incident radiant energy from the solar simulator, the absorptivity and emissivity of the coating may be calculated using the relationships of radiatiative heat transfer.

Calculation Methods:

The calculations are made to solve the radiative heat transfer relations, knowing the transient heat content properties of the substrate and coating as well as the temperature of the shroud. From these calculations, the absorptivity and the emissivity of the coating may be determined.

Interpretation/Precision and Accuracy:

Many potential error sources are inherent in this measurement. These relate primarily to the measurement of the temperatures and the rates of change of the temperatures, the reflectivity of the thermal shroud, the "low emittance" of the rear substrate surface, and the precision of the radiometric determination of the incident energy on the sample.

Date: December 1986   Reporter's Name: Michael G. Hutchins

Country: United Kingdom

Testing Title: DETERMINATION OF THE SOLAR ABSORPTANCE/REFLECTANCE/
TRANSMITTANCE FROM SPECTRAL MEASUREMENTS AT NEAR-
NORMAL INCIDENCE

Abstracted Scope (200 words):

Spectral reflectance and/or spectral transmittance measurements are made using a Beckman 5240 integrating sphere spectrophotometer in the spectral range 0.3 - 2.5 µm. In reflectance mode radiation is incident on the surface at 5° to the surface normal. In transmittance mode the radiation is incident normally on a surface placed over a sphere entry port.

Solar optical properties are calculated by convoluting the spectral reflectance curve with a standard solar spectral distribution curve for air mass 2 using a method of 20 selected ordinates. One of two solar spectral distributions may be used (1,2).

The measurement procedure is automated using an Apple IIe microcomputer.

Referred Document:

1.   Wiebelt J.A. and Henderson J.B., J. Heat Transfer 101, 101, 1979.

2.   Moon P., J. Franklin Inst., 230, 583, 1940.

Principle of Measurement:

Near-normal hemispherical spectral reflectance or transmittance of incident radiation.

Apparatus:

Beckman 5240 uv/vis/nir integrating sphere spectrophotometer using photomultiplier for uv/vis detection and PbS for nir detection.
6" diameter integrating sphere coated with barium sulphate white reflectance standard. Double beam ration recording principle of operation.
Apple IIe microcomputer for calculation of optical properties.

Calibration:
For diffuse surfaces : barium sulphate
For highly specular surfaces : aluminium mirror

Sampling:

Minimum sample size : 30mm x 30mm
Maximum sample size : 100mm x 100mm

Conditioning:

None

Procedures:

1. Sample cut to size and cleaned by air blowing.
2. Instrument is calibrated using two barium sulphate reference plates.
3. Sample mounted on sphere wall (rear for reflectance ; front for transmittance) and reflectance or transmittance measured for scan from 2500nm − 350nm.
4. Spectral data at 1nm intervals are sent to an Apple IIe microcomputer.
5. Microcomputer executes calculation to determine surface optical properties, e.g. α_s, τ_s or ρ_s.

Calculation Method (if necessary, please include correction method):

20 selected ordinates for air mass 2 solar spectral distribution giving

$$\alpha_s = \frac{1}{20} \sum_{i=1}^{20} \alpha_i \; , \quad \text{where } \alpha_i \text{ is the absorptance at each of the twenty}$$

selected ordinates and is found from $\alpha_i = 1 - \rho_i$.

$$\tau_s = \frac{1}{20} \sum_{i=1}^{20} \tau_i \; , \quad \text{where } \tau_i \text{ is the transmittance at each of the twenty}$$
$$\text{selected ordinates.}$$

Interpretation/Precision & Accuracy:

Precision : reproducible to one part in 10^3.
Accuracy : 1-2%.

REPORTS ON SOLAR ABSORBERS

a (ii) EMITTANCE AT A DESIGNATED TEMPERATURE

Date: 2/86   Reporter's Name: K. Gindele

Country: F.R.G.

Testing Title: Spectral directional emittance (infrared range) I
(Reflexion method)

Abstracted Scope (200 words):

Measurement of the spectral near-normal directional
emittance using the reflexion technique ($\varepsilon(\lambda) = 1 - \varrho(\lambda)$).
The spectral reflectance is measured in the spectral
range from 1 µm to 15 µm. The photometric integration
is done with a gold-coated integrating sphere.
Sample temperature: room temperature.

Referred Document:

K. Gindele, M. Köhl and M. Mast:
Applied Optics Vol.24, Nr.12, pp.1757-1760, 1985,
Spectral reflectance measurements using an integrating
sphere in the infrared".

K. Gindele, M. Köhl and M. Mast:
"Spectral directional emittance measurements in the
wavelength range from 1 µm to 15 µm",

Proc. of 1985 Int. Conf. on Fourier and Computerized
Infrared Spectroscopy, Ottawa, Canada, June 24-28, 1985.

Principle of Measurement:

   Spectral measurement of the hemispherically reflected
   flux with an integrating sphere.

Apparatus:

   Measuring geometry: near-normal directional irradiation
   at an angle of $10°$, detection of the hemispherically
   reflected radiation with a gold-coated integrating sphere.
   Use of "comparison method", that means sample and reference
   are simultaneously attached to the sphere ports.

   Spectrometer system: rapid-scan Fourier-spectrometer.

Calibration: A specular gold mirror is used as reference.

Sampling: Samples are attached to the respective sphere port.

Conditioning: The spectrometer system has to be evacuated
              and in an equilibrium state.

Procedures:

The energy distributions of the sample and the high-reflecting reference are measured successively. The sample spectrum A_s is related to that of the reference A_r with relation $*$.

For correct evaluation the specular reflectance has also to be measured and with this the specular reflectance factor SRF (compare ASTM 429) has to be calculated.

The reflectance from $*$ then can be corrected for the indicatrix induced effects with relation $**$. (This correction method will be presented by the reporter at 3rd Int. Symp. on Optical and Optoelectronic Applied Sciences and Engineering, Conf. 653, Innsbruck, Austria, 14-18 April 1986 (SPIE Proc. Vol. 653).

Calculation Method (if necessary, please include correction method):

$$\rho_s(\lambda) = \frac{A_s(\lambda)}{A_r(\lambda)} \rho_r(\lambda). \qquad *$$

$$\varrho_{s_{cor}} = \varrho_s \frac{S_w}{1 - \zeta + \zeta\, S_w} \qquad **$$

ϱ_s: reflectance of the sample

ϱ_r: reflectance of the reference

ϱ_w: reflectance of the wall

ζ : SRF of the sample

Interpretation/Precision & Accuracy:

Without correction of the effects caused by the indicatrix in the worst case an error of 0.06 occur.

With correction: $\approx$ +/- 0.02 (still under investigation).

REPORTING FORMAT

Date: 2/86   Reporter's Name: K. Gindele

Country: F.R.G.

Testing Title: Spectral directional emittance (infrared range) II
              (Radiometric method)

Abstracted Scope (200 words):

> Measurement of the spectral normal directional emittance
> of absorber coatings (on heat conducting substrates).
> This radiometric method yields results in the spectral
> range form 4 μm to 25 μm.

> Sample temperature: 373K.

Referred Document:

> K. Gindele, M. Köhl and M. Mast:
> "Spectral directional emittance measurements in the
> wavelength range from 1 μm to 15 μm",
> Proc. of 1985 Int. Conf. on Fourier and Computerized
> Infrared Spectroscopy, Ottawa, Canada, June 24-28, 1985.

Principle of Measurement:

Radiometric measurement of the spectral radiance of heated samples.

Apparatus:

Samples and high-emitting reference are mounted on a turnable, electrically heating device. The radiation emitted by them is directed to the spectrometer system, that means the samples act as radiation source.

Spectrometer system: rapid-scan Fourier-spectrometer.

Calibration: 3M, Velvet Coating 2010 is used as reference.

Sampling:

Samples are mounted on the heater.

Conditioning: The spectrometer system has to be evacuated and in an equilibrium state.
The sample substrates have to be very plane in order to ensure a good heat transition thus resulting in surface temperatures of the samples equal to those of the references. The substrates of the samples and the references have to be of a material with the same conductivity.

Procedures:

The energy distribution of the sample A_s, of the reference A_r, and of an unheated diffusely reflecting gold mirror A_g are measured successively.

Evaluation is done with relation ✳.

Calculation Method (if necessary, please include correction method):

$$\varepsilon_s(\lambda) = \frac{A_s(\lambda) - A_g(\lambda)}{A_r(\lambda) - A_g(\lambda)} \; \varepsilon_r(\lambda) \qquad\qquad ✳$$

Interpretation/Precision & Accuracy:

under investigation

Date: 2/86   Reporter's Name: K. Gindele

Country: F.R.G.

Testing Title: Total hemispherical emittance

Abstracted Scope (200 words):

Measurement of the total hemispherical emittance of
absorber coatings (on heat conducting substrates).
Time-saving measurement of the emittance at a temperature
of 373K.

Referred Document:

K. Gindele, M. Köhl and M. Mast:
Infrared Physics Vol. 24, No. 5, pp.455-461, 1984,
"Properties of the ellipsoidal reflector of the
McDonald emissiometer".

Principle of Measurement:

Radiometric measurement of the flux emitted by heated samples. Hemispherical collection with the aid of an ellipsoidal reflector.

Apparatus: Modified McDonald emissiometer Model 2145.
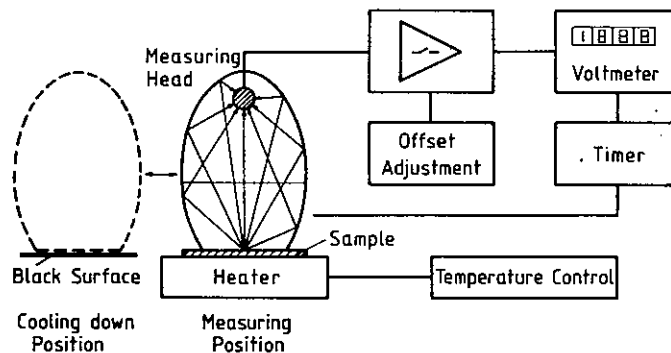
Calibration: The measured values of the samples are related to the measured values of a high-emitting reference (3M, Velvet Coating 2010) as well as a low-emitting reference (electroplated Au).

Sampling: Samples and references, respectively, are put on a heater and fixed there by vacuum.

Conditioning: The sample substrates have to be very plane in order to ensure a good heat transition thus resulting in surface temperatures of the samples equal to those of the references. The substrates of the samples and the references have to be of a material with the same heat conductivity.

Procedures:

The measuring head is placed over the thermostatted sample and the emitted radiation heats the thermopile-detector. The detector signal is read after 10 sec. After the cooling down of the detector the references are measured in the same manner. From the readings R_S (sample), R_L (low-emitting reference), R_H (high-emitting reference) and the emittances $\mathcal{E}_H$ and $\mathcal{E}_L$ of the references we calculate the emittance of the sample $\mathcal{E}_S$ with relation ✱. Afterwards we correct the non-linearities of the measuring head (explicitly elaborated in the referred document) with relation ✱✱, where $\mathcal{E}_m$ means measured emittance (= result $\mathcal{E}_S$ of relation ✱) and $\mathcal{E}$ is the corrected value.

Calculation Method (if necessary, please include correction method):

$$\epsilon_S = \frac{R_S - R_L}{R_H - R_L}(\epsilon_H - \epsilon_L) + \epsilon_L \qquad \text{✱}$$

$$\epsilon = \frac{0.747\epsilon_m}{1 - 0.253\epsilon_m} \qquad \text{✱ ✱}$$

Interpretation/Precision & Accuracy:

$\Delta \mathcal{E}_\Delta = +/- 0.03$

Date: 2/86   Reporter's Name: K. Gindele

Country: F.R.G.

Testing Title: Total directional emittance

Abstracted Scope (200 words):

Measurement of the angular dependance of the total
directional emittance of absorber coatings (on heat
conducting substrates). The measuring temperature is
373K. The angular range is $0 \leq \vartheta \leq 70°$.
(ϑ: angle to the surface normal).

Referred Document:

Principle of Measurement:

Radiometric measurement of the radiance of heated samples.

Apparatus: Measuring geometry:
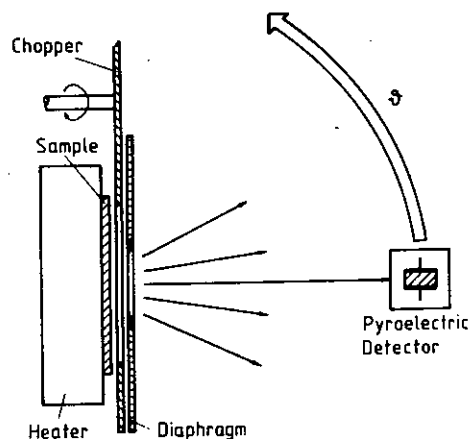
Angle of aperture: 10°.

The detector signal is amplified phase-sensitively.

Calibration: The measured values of the samples are related to the measured values of a high-emitting reference (3M, Velvet Coating 2010) as well as a low-emitting reference (electroplated Au).

Sampling: Samples and references, respectively, are put on a heater and fixed there by vacuum.

Conditioning: The sample substrates have to be very plane in order to ensure a good heat transition thus resulting in surface temperatures of the samples equal to those of the references. The substrates of the samples and the references have to be of a material with the same heat conductivity.

Procedures:

The radiances of the sample and the references, respectively, are measured. From the voltage-readings of the lock-in-amplifier R_S (sample), R_L (low-emitting reference) and R_H (high-emitting reference) as well as the emittances ε_H and ε_L of the references we calculate the emittance of the sample ε_S with relation *.

Calculation Method (if necessary, please include correction method):

$$\epsilon_S = \frac{R_S - R_L}{R_H - R_L}(\epsilon_H - \epsilon_L) + \epsilon_L \qquad \text{*}$$

Interpretation/Precision & Accuracy:

$\Delta \varepsilon = +/- 0.03$

<u>Date</u>: February 19, 1986   <u>Reporter's Name</u>: J. Keller, EIR,
CH-5303 Würenlingen

<u>Country</u>: Switzerland

<u>Testing Title</u>: Thermal emittance of absorber coatings (hemispherical)

Abstracted Scope (200 words):

Measurement of the total emittance of absorber coatings in the range of
wave lengths from 3 µm to about 50 µm using an Emissometer, model AE
(Devices and Services, Co, Texas)

Referred Document:

- Instruction Manual. Emissometer, Model AE.

Principle of Measurement:

See "procedures"

Apparatus:
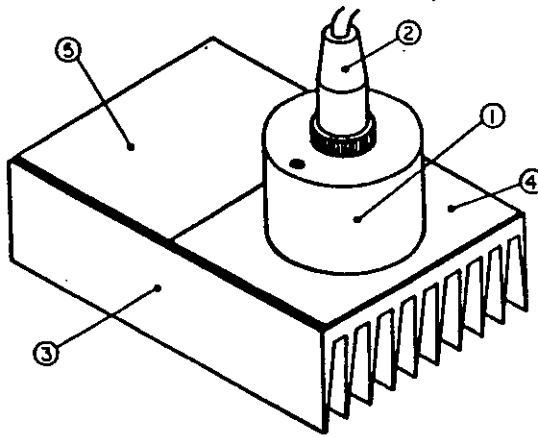
1: Emissometer, 2: cable, 3: heat sink, 4: absorber sample, 5: reference sample

Calibration: Nextel Black paint reference surface (ε = 0.93)

Sampling:

Sample size about 7 cm x 7 cm.
Only flat samples

Conditioning:

Procedures:

The radiation detector is a differential thermopile with sensing surfaces of aluminium foil and Nextel Black paint, and responds to radiative heat transfer between the sensing and the neighbouring flat sample surface. The sample is maintained near ambient temperature by means of a heat sink, whilst the detector assembly is heated internally to 82 °C. The detector is placed on the surface whose thermal emittance is to be determined, and takes about a minute to attain thermal equilibrium with the sample. The detector output voltage is measured using a high input impedance digital voltmeter having .001 mV resolution, and is linearly correlated to the sample emittance within ± .01.

The detector output voltage is calibrated against a Nextel Black reference surface (ε_{ref} = .93) maintained at the same temperature as the sample by means of the heat sink.

Calculation Method (if necessary, please include correction method):

$$\varepsilon = \varepsilon_{ref} \frac{U}{U_{ref}} , \qquad \varepsilon_{ref} = 0.93$$

Interpretation/Precision & Accuracy:

Accuracy: ± 0.03

REPORTING FORMAT

Designation :
AC-O-LA

Date : 02/19/86   Reporter's Name : Kazuo Taga

Country : Japan

Testing Title :

Testing Method of Emissivity of Selective Coating Surface

Abstracted Scope (200 words):

The D & S Emissometer is a special purpose instrument for making
emissivity measurements. The detector portion of the device is heated
to 130F so that the sample does not have to be heated. The detector
responds only to radiation heat transfer, and is designed to have
an output linear with emissivity. The emissivity of an unknown surface
is meaured after calibration of the detector with a sample of known
emissivity, which is maintained at the same temperature as the unknown.

Referred Document :

(1) ASTM E408 "Standard Test Methods for Total Emittance of Surfaces --- "
(2) "-DEVICES & SERVICES COMPANY
     "Emissometer Model AE Operating Instructions "

Principle of Measurement :

The emissivity is determined by measuring the radiation from the specimen by the electromotive force.

Apparatus :

Description	Reference Numbers
Emissometer detector	1
4 pin connector and cable	2
Heat sink	3
Two high emissivity standards-Flat black paint	4
Two low emissivity standards-bare aluminum	5
Screwdriver	
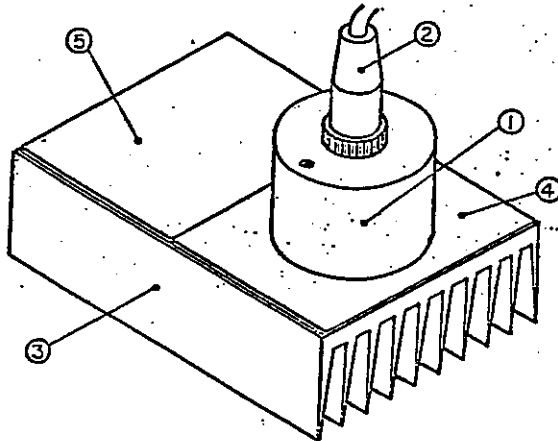
Calibration :

Sampling :

60 x 60mm (flat plate)

Conditioning :

Spectral response : thermal wave length (3 to 30 microns)
Warming up time : 10 munutes
Response time : 10 seconds, Nominal
ditector temperature : about 80 degrees Centigrade

<u>Procedures</u>:

1. Turn on the detector heater and the readout device and allow to warm up for approximately ten minutes.

2. Put a flat black standard on one end of the heat sink and the unknown on the other end. Samples must be relatively flat and the material must be a good thermal conductor.
($k/x > 1100$ watts/m^2 ^{o} C or 200 Btu/hr ft^2 ^{o} F, k-thermal conductivity, x-thickness).
To insure that the standard and the unknown remain at the same temperature, good heat transfer must be maintained between the sample and the heat sink. This can be accomplished by putting several drops of water on the heat sink before putting down the sample. Make sure that there is enough water to fill the gap between the sample and the heat sink, but not so much that it flows up onto the samples or the detector. A heat transfer grease can also be used for this purpose.

3. Place the detector on the flat black standard and wait for the detector voltage reading to become steady. Record this voltage, $V_{standard}$.

4. Move the detector to the sample to be measured. Wait about 45 seconds for the voltage, V_{sample}, to become steady.

5. Emissivity of the sample is calculated as follows:

$$\varepsilon_{sample} = \frac{V_{sample}}{V_{standard}} \times 0.93$$

<u>Calculation Method</u> (if necessary, please include correction method) :

Its value is corrected by the result of calorimetric method.

<u>Interpretation/Precision & Accuracy</u> :

Accuracy : 0.3% + 1 digit for indication
Temperature dependense : 0.03%/ $^\circ$C

REPORTING FORMAT

Date: March 1, 1986   Reporter's Name: S. TANEMURA

Country: JAPAN

Testing Title:

Measurement of Normal Spectral Emittance of Absorber & Glazing Material

Abstracted Scope (200 words):

To evaluate the selective absorbing property of the absorber materials for solar collector and the selective transparent property of the glazing materials for both solar collector and the radiation control window, normal spectral emittance (NSE) from 2.5 µm to 25 µm in wavelength and from 50°C to 250°C in the sample temperature is an important physical property to be measured. To minimumize the measurement errors due to the uncertainity of the equivalence of both sample elevated temperature and the blackbody temperature and the drift of those temperatures during the emperiment, FT-IR spectrophotometry is used. The surface temperature of the sample and that of blackbody were determined by the relationship beween the monitored TC-reading and the indicated temperatures by thermo-visualizer equipment which is experimentally obtained for various total emittance value assumed tentatively to the sample.

Referred Document:

S. TANEMURA et al, Abstract of 39th Semiannual Meeting on Research Achievements in G.I.R.I. Nagoya, p.91 (1985) (in Japanese)

<u>Principle of Measurement:</u> The notation $V_S(\lambda)$, $V_b(\lambda)$ and $V_r(\lambda)$ are defined as the detected spectral signal of the sample heated at T_S K, that of the blackbody controlled at T_S K and that of the perfect reflector at room temperature respectively. Then, NSE $\varepsilon(\lambda, T_S)$ of the sample at T_S K is derived from the equation.

$$\varepsilon(\lambda, T_S) = [V_S(\lambda) - V_r(\lambda)] / [V_b(\lambda) - V_r(\lambda)].$$

Hence, the measurements of $V_S(\lambda)$, $V_b(\lambda)$ and $V_r(\lambda)$ are required.

<u>Apparatus:</u>

Low Temp. Range: (60~85°C) Sample heating furnace (SF) and blackbody (B) have the hot water reservoirs to which the hot water controlled at a designated temperature is circulated simultaneously. The auxiliary electric heater is provided at the inlet tube to the sample furnace.
Medium Temp. Range: (100~250°C) Apparatures SF and B are controlled independently by proportional-differential electronic control device.

S :SAMPLE   B:BLACK BODY   D:DETECTOR   M1:EXCHANGE MIRROR
M2:SAMPLE-BLACK BODY EXCHANGE MIRROR

Fig.1, Schematics of present normal spectral emmisiometer.

<u>Sampling:</u>

The size of the sample; 25 (mm) X 25 (mm) X 5 (mm).

<u>Conditioning:</u>

Preconditioning
1. To avoid the measurement of the transient surface properties under the applied temperature in the normal room environmental conditions, the sample is kept at a designated temperature for 12 hrs at least before measurement.
2. Thick Au coating is required to the rear surface of transparent glazing materials.

Procedures:

After preconditioning, the followings are processed:

(1) Obtaining the relationship between the TC reading of the blackbody and the indicated temperature by thermovisualizer under viewing the blackbody center.

(2) Obtaining the relationships between the readings of TC provided to be touched on the surface of the sample and the indicated temperature by thermovisualizer under viewing the heated sample surface as the function of the varius total emittance values assumed tentatively for the sample.

(3) $V_r(\lambda)$ measurement.

(4) Control the blackbody at a designated temperature (T_b).

(5) Control the sample at the temperature (T_s) approximately to be regarded as the equivalent to T_b value.

(6) $V_s(\lambda)$ and $V_b(\lambda)$ measurement.

(7) ε calculation and the output of ε as the function of wavelength.

(8) Estimation of the total emittance considering with the response function of the detector of the thermovisualizer equipment.

(9) Confirmation of the sample surface temperature according to the relationship described in (2). If necessary, the adjustment of the controlled sample temperature and procedures (5) - (9) are required to be repeated.

Calculation Method (if necessary, please include correction method):

Opaque Material: ε is calculated by the equation described in the Principle.

Transparent Material: Both Normal Spectral Reflectance (NSR) and Normal Spectral Transmittance data of the sample without rear surface Au coating as well as the NSR of Au coating are also required to obtain NSE. The optical model with four phases such as ambient, surface coating, glazing and Au coating is required to calculate the NSE.

Interpretation/Precision & Accuracy:

Typical example:

(1) Precision of NSE: less than ±0.02 under Temperature drift ±0.3°C, Difference of $T_b - T_s = ±0.5°C$ in emittance units.

(2) Reproducibility: less than ±0.01 in emittance units.

REPORTING FORMAT   Designation:
                          A-O-LA

Date: 1986-03-11   Reporter's Name:

Country: Sweden

Testing title:

Measurement of emittance at 100 °C temperature
Normal

Abstracted Scope (200 words):

To determine the spectrally total emissivity of solar collector
absorbers at 100 °C temperatue by comparing its radiance with the
radiance of a blackbody radiator also at 100 °C.

Referred Document:

SP RAPP

/

Principle of Measurement:

The radiance normal to the surface kept at 100 °C temperature is compared with the radiance from a blackbody radiator at 100 °C using a detector with a approximately wavelength independent responsivity in the 1-30 µm wavelength range.

Apparatus:

Aluminium cylinder ø 150x100 mm for sample heating to 100 °C.
Blackbody radiator ø 10x80 mm cavity.
Temperature controller to both sources.
Radiation detector of Golay type with KRS5 window and 500 mm long aperture tube with 5 mm front aperture.
Radiation chopper.
Thermocouples to sample front surface, to the blackbody, to the tube and to surroundings.
Lock-in amplifier.

Calibration:

Sampling:

Samples must be flat and metallic.

Conditioning:

Two 1 mm holes are drifted at each side of the measurement area for the thermocouple leads.

Procedures:

The aluminium cylinder and the blackbody are stabilized to 100 °C.
The sample is mounted on the flat surface of the cylinder tightly to
get good thermal contact using a flat ring and screws.

Thermocouple leads are placed in the drilled holes on the sample and
small plugs forces the leads to the front surface of the sample.

The surface temperature, blackbody temperature, tube temperature,
ambient temperature, detector output with blackbody and detector
output with sample is measured.

Calculation method:

See SP-RAPP

Interpretation:

Precision: <±0.01   95 % confidence level

Accuracy: <±0.04

Designation:

AS/AC/GS/GC-0-LA

REPORTING FORMAT

Date:	April 7, 1986	Reporter's name: P.A. van Nijnatten

Country: Technisch Physische Dienst TNO-TH
(TNO Institute of Applied Physics)
P.O. Box 155, 2600 AD DELFT
The Netherlands

Testing title: DETERMINATION OF DIRECTIONAL AND HEMISPHERICAL TOTAL EMITTANCE
AT TEMPERATURES OF 60 °C TO 200 °C.

Abstracted Scope (200 words):

The hemispherical total emittance is used in calculating radiative heat transfer
in collector and window glazings, collector absorber materials and coatings etc.
An apparatus designed after Irving et al [1] is used for quick measurements of
the total emittance at different emitting angles and at different surface
temperatures.
The hemispherical total emittance is obtained by integrating over all emitting
angles.

Referred document:

[1] Irving, T.F., Hartnett, J.P. and Eckert, E.R.G., Solar Energy, 2 (1958) 13.
[2] Spectral-selective surfaces for the thermal conversion of Solar Energy,
M. van der Leij, Delft University Press, 1979.

Principle of measurement:

Radiometric measurement of the radiance of heated samples.

Apparatus:

θ_e. Emitting angle

1. Sample

2. Heating unit (canthal wire 14 Ω) and Cromel-Alumel thermocouples

3. Water cooled jacket, 5°C.

4. Water cooled diaphragm, $\emptyset$ 0.010 m

5. Bolometer + electronics (Heimann KT 14)

6. Detector head with Ge-lens (Heimann, type L)

7. Voltage supply (24 VAC)

8. Galvanometer Kipp

9. Rotating disc with knob and graduating disc, outside the jacket

10. Outer housing filled with dry air

11. Temperature controller and recorder

The angle θ_c is adjustable from 15° - 75°.
The heating unit is surrounded by a black-pointed cooled jacket in order to reduce reflections from the wall to the sample and to keep the ambient temperature constant. The spectral sensitivity of the detection is from 2-20 μm (mainly determined by the Ge-lens.

Calibration:

Heimann calibration radiator (type SW13) or black body radiator at sample position.

Sampling:

The sample is attached to the heater with a heat transfer compound or screws
Only a spot of $\emptyset$ 4.10^{-3} m is seen by the detector.

Conditioning:

- The samples and references have to have approximately the same thermal
  conductivity.
- To avoid condensation on the coated wall, the apparatus is purged with dry air.

Procedures:

1. The sample is thoroughly cleaned and attached to the heater.
2. The temperature of the heating unit is set.
3. After sufficient warm-up time the recorder reading gets stable and measurement
   begins.
4. Measurements are made at view-angle intervals of 5°.

Calculation Method (if necessary, please include correction method):

If it is assumed that the inner wall of the cooling jacket with temperature T_o (K) is black, the total energy flux density G coming from the direction of the sample surface of temperature T_s (K) received by the detector surface is given by

$$G = F\sigma \left[\varepsilon(\theta_e, T_s)\, T_s^4 + \rho(\theta_e, T_s)\, T_w^4 \right],$$

in which F is the view factor between the sample area and the detector area, σ is the Stephan-Boltzmann constant, $\varepsilon(\theta_e, T_s)$ is the directional total emittance of the sample with θ_e as the angle between the normal on the centre of the sample and the connecting line from the centre of the detector area to the centre of the sample, $\rho(\theta_e, T_s)$ is the hemispherical directional total reflectance of the sample with temperature T_s, and T_w is the temperature of the cooled jacket. If the detector is calibrated with a black body radiator, then the energy flux density from the sample falling on the detector, as seen by the detector as coming from a black surface with temperature T_x, the emitted energy flux density is

$$G' = F\,\sigma\, T_x^4.$$

On the basis of this idea, G = G', so that

$$T_x^4 = \varepsilon(\theta_e, T_s)T_s^4 + \rho(\theta_e, T_s)T_w^4.$$

By taking
$$\varepsilon(\theta_e, T_s) = 1 - \rho(\theta_e, T_s),$$

we can write $\varepsilon(\theta_e, T_s)$ as

$$\varepsilon(\theta_e, T_s) = \frac{T_x^4 - T_w^4}{T_s^4 - T_w^4}.$$

This will be the basic equation for our measurements if we assume that the detector response is (linearly) proportional to the intensity of the radiation received.

Interpretation/Precision & Accuracy:

$\varepsilon < 0,2: \Delta\varepsilon = \pm\, 0,02$

$\varepsilon > 0,2: \Delta\varepsilon = \pm\, 0,01$

REPORTING FORMAT

Date: 9/26/86   Reporter's Name: C. Hauer
Testing Title: Total Normal Emittance of Surfaces Using
Inspection Meter Techniques

Abstracted Scope:

This test method covers determination of the total
normal emittance of surfaces by means of portable
inspection meter instruments. The tests are intended for
large surfaces where rapid and non-destructive methods
are required.

Referred Document:

ASTM Standard E 408-71.
The following references are also applicable:
* Nelson,K.E.,Leudke,E.E., and Bevans,J.T., Journal of
Spacecraft and Rockets, JSCRA, Vol.3, No.5, 1966, p.758
* Gaumer,R.E.,Hohnstreiter,G.F.,and Vanderschmidt,G.F.,
"Measurement of   Thermal   Radiation   Properties   of
Materials", NASA SP-31, 1963,p. 117

Principles of Measurement:

Two measurement methods are described: one which measures radiant energy reflected from a specimen, Mehtod A; the other which measures radiant energy emitted from a specimen, Method B. The first method uses an infrared reflectometer which alternately exposes the specimen to two radiant sources, one near ambient temperature and the other at a somewhat elevated temperature. The radiation reflected from the specimen from each source is detected by a vacuum thermocouple which has been calibrated with known reflectance standards. These data are reduced to obtain the normal emittance using Kirchoff's law. The second method relies upon cooling the specimen and the sample and chamber, viewing the specimen surface, and measuring the the emitted energy with a calibrated thermopile.

Apparatus:

The following instruments, or instruments with similar capabilities are required:
* For Method A:
   Infrared Reflectometer Model DB 100
   Gier-Dunkle Instruments, Inc.
   Santa Monica, Ca.

* For Method B:
   Emissometer, Model 25A
   Lion Research Corp.
   Cambridge, Mass.

Calibration

Calibration is required for both methods, using using reflectance and emittance standards. These calibrations are carried out on the instruments after an appropriate warmup period, calibrating the meter at both the high and low ends of the scale using appropriate reflectance or emittance standards.

Sampling:

ASTM E 408-71 does not specify sampling procedures.

Conditioning:

No special sample conditioning is required.

Procedures:

After the meter has been calibrated, place the specimen
over the aperture of the instrument. For the reflectance
measurement instrument used for Method A, the result is
the reflectance of infrared radiation near room
temperature. In Method A obtain the emittance by
subtracting the reflectance from unity. In Method B, the
emittance is obtained from the instrument maker's
emittance/meter conversion data.

Calculation Methods:

For Method A, the application of Kirchoff's law involved
in subtracting the the reflectance from unity is the
only calculation required. Method B requires no
calculations since the emittance is obtained from the
instrument's calibrated emittance vs. meter reading
curve.

Interpretation/Precision and Accuracy

Both methods are limited in accuracy by the degree to
which the emittance or reflectance properties of the
calibrabing standards are known. Method A is subject to
a small error resulting from the difference in
wavelength distributions between the radiation emitted
by the two cavities at different temperatures and that
emitted by a black body at the specimen temperature.
Method B also exhibits errors since the detector is not
at absolute zero. Both methods are suited for production
control or quality testing.

REPORTING FORMAT

Date: 9/30/86   Reporter's Name: C. Hauer
Country: USA
Testing Title: Calorimetric Determination of Hemispherical
Emittance and the ratio of Solar Absorptance to Hemispherical
Emittance Using Solar Simulation

Abstracted Scope:

ASTM E 434-71 describes measurements of the radiative properties
of absorber materials or coatings using radiative heat gains and
losses from the specimen in vacuum. The measurements derive
absorptivity and emissivity using the radiation laws of heat
transport. They are carried out in a vacuum environment to
eliminate conductive and convective energy transport.

Referred Document: ASTM E 434-71

Principle of Measurement:

The principle on which the measurement is based is the radiant
transport of energy as defined by the law governing radiation
absorbed by a body and energy radiated by that body to an
environmeent following the rule of the fourth power of the
temperature of the body and its environment. In these measurements
a defined amount of simulated solar radiant energy is absorbed by
the test body. The rate at which this energy is reradiated is
estimated.

Apparatus:

The following apparatus is required:
* a vacuum system equipped with a cold shroud capable of attaining
a vacuum of at least .1 mPa (1 micro torr.) and maintaining a
temperature environment well below ambient around the sample.
* a shutterable aperture in the vacuum apparatus large enough to
illuminate the entire sample
* a minimum of two thermocouples capable of resolving at least .1
C and having rapid time response
* a solar simulator capable of an irradiance of at least 7000 W
per square meter
* a radiometric device for the measurement of the simulated solar
irradiance
* an interval timer capable of .1 second resolution

Calibration:

ASTM E 434 does not specify any sampling methodology. It is
suggested that thermocouples and the radiometric measurement
device should be calibrated in accordance with the manufacturer's
specifications.

Sampling:

ASTM E 434 does not specify any sampling methodology, but it is
suggested that at least five coated samples be used.

Conditioning:

No sample conditioning is specified in ASTM E 434-71

Procedures:

The test method involves suspending a sample, an absorptive coating on a substrate, mostly surrounded by a cooled constant temperature thermal shroud all in a vacuum chamber. The sample area is predicated on the area of the shroud and should be small in relation to this area. The sample substrate should be well charactized as to thermal conductivity and heat capacity. The front surface of the substrate is coated with the with the sample coating while the edges and back surface have a low emittance coating. The substrate back is fitted with a thermocouple and the thermocouple is coated with a low emittance material. The specimen is suspended in the shroud and the vacuum chaber is pumped down. Measured amounts of simulated solar irradiance are made to impinge on the sample. The thermoucouple then serves to register the specimen temperature and the rate of temperature change. From these temperature measurements, the mesured temperature of the thermal shroud and the knowm incident radiant energy from the solar simulator, the absorptivity and emissivity of the coating may be calculated using the relationships of radiatiative heat transfer.

Calculation Methods:

The calculations are made to solve the radiative heat transfer relations, knowing the transient heat content properties of the substrate and coating as well as the temperature of the shroud. From these calculations, the absorptivity and the emissivity of the coating may be determined.

Interpretation/Precision and Accuracy:

Many potential error sources are inherent in this measurement. These relate primarily to the measurement of the temperatures and the rates of change of the temperatures, the reflectivity of the thermal shroud, the "low emittance" of the rear substrate surface, and the precision of the radiometric determination of the incident energy on the sample.

Date: December 1986   Reporter's Name: Michael G Hutchins

Country: United Kingdom

Testing Title: MEASUREMENT OF THERMAL EMITTANCE FROM SPECULAR REFLECTANCE USING AN INFRARED SPECTROPHOTOMETER

Abstracted Scope (200 words):

Test samples are mounted in a dual-beam ratio-recording spectrophotometer in a holder that allows incident radiation to strike the surface at 37° to the surface normal. Specularly reflected radiation is detected by a thermocouple and an output of infrared specular reflectance as a function of incident radiation wavelength in the rane 2.5 - 50.0 µm is recorded.

The test sample temperature is known (307 K) and the specular reflectance curve is convoluted with a blackbody spectral emissive curve for the same temperature using an equal energy interval mathod of 20 selected ordinates.

The accuracy of the method is poor if the test surface has surface roughness features which are seen by the incident infrared radiation. Since the diffuse component of the reflectance is lost and

$$\alpha_\lambda = \varepsilon_\lambda = 1 - \rho_\lambda$$

all measured values of thermal emittance, ε, will be equal to or greater than the true value.

Referred Document:

None known

Principle of Measurement:

The incident and reflected angles are fixed at 37° so that values of thermal emittance are spectral or total values but only for the fixed geometry of the system.

Apparatus:

Perkin Elmer 683 dual-beam ratio-recording infrared spectrophotometer. Reference calibration is made using an aluminium mirror deposited onto a glass substrate.

Calibration: Aluminium mirror

Sampling:

Sample size in the range 25 mm x 25 mm - 100 mm x 100 mm

Conditioning:

Sample must be left to reach the working temperature of the spectrophotometer measurement chamber.

Procedures:

1. The reflectance of the spectrophotmeter is calibrated using an aluminium mirror.

2. The test sample is mounted and allowed to come into thermal equilibrium.

3. Spectral reflectance data are recorded for a scan between 2.5 μm and 50.0 μm.

4. 20 selected ordinates for the reference blackbody spectral distribution at 307 K are used to read off reflectance data for the test sample from the chart recording.

5. The thermal emittance is calculated using the standard method.

Calculation Method (if necessary, please include correction method):

For each of the twenty selected ordinates a value of reflectance ρ_i is taken.

The monochromatic thermal emittance, ε_i, is then found from

$$\varepsilon_i = 1 - \rho_i$$

The integrated thermal emittance is then found from $\varepsilon = \dfrac{1}{20} \sum_{i=1}^{20} \varepsilon_i$

Interpretation/Precision & Accuracy:

Reproducibility : 3%

Accuracy : For smooth specular surfaces – 3%
           For rough surfaces – impossible to quantify in a simple way

Date: December 1986

Country: United Kingdom

Reporter's Name: Michael G. Hutchins

Testing Title: Measurement of near-normal total thermal emittance using a pyroelectric radiometer

Abstracted Scope (200 words):

Thermal radiation from a heated test sample is chopped and focussed, using a KBr convex lens, onto the element of a pyroelectric detector equipped with a KRS-5 window. A saw-tooth waveform with peak-to-peak height dependent on the temperature difference between the sample and a black-body background and the test sample thermal emittance is produced. This signal and a reference signal from the chopper are fed to a lock-in amplifier and the resultant dc level is measured. Readings are taken of this dc voltage as a function of the temperature difference between the test sample and the background. A plot of V against ΔT produces a straight line for $\Delta T < 10^{\circ}C$. The ratio of the gradient of this line to that produced by a blackbody reference yields the thermal emittance of the test sample.

Referred Document:

Hutchins M.G. , Optica Pura y Aplicada 12, 15, 1979.

Principle of Measurement:

Direct measurement of thermal radiation (total radiation emitted normal to surface). Pyroelectric detection ensures flat spectral response for all wavelengths of interest. Emittance values are derived by comparison with radiation measurements from blackbody reference standards.

Apparatus:

In-house development. Test sample is resistively heated. Test sample surface temperature and temperature of ambient sensed with K-type thermocouples. Rofin-Sinar chopper, operating at 16 Hz modulates radiation from heated sample. 2" focal length KBr convex lens focusses thermal radiation onto triglycine sulphate pyroelectric detector which is protected by a KRS-5 ir transparent window. Pyroelectric detector is Plessey Model PSC222. Signal conditioning is performed using an Ortec 9501 lock-in amplifier. Optical system is mounted on a 0.25 m optical bench and housed within a light proof, blackened wooden box. The interior of the box provides the blackbody ambient background radiation level.

Calibration: Using a blackbody reference and samples of known emittance

Sampling:

Minimum sample size 5mm x 5mm
Maximum sample size 75mm x 75mm

Conditioning:

None

- 2 -

Procedures:

1. Sample is mounted in holder, thermocouple is attached to front surface, and is then heated to a known temperature T above the ambient temperature T_a.

2. The total thermal radiation emitted normally from the surface is focussed onto the detector element and the peak to peak height of the waveform is measured using a lock-in amplifier.

3. The temperature of the sample is changed and a new value of amplifier output, V (volts), is recorded. This is performed for 6-10 different values of T.

4. A graph of V v. $T-T_a$ is plotted and a straight line is fitted to the data. The gradient of the line is calculated.

5. The ratio of the gradient of the line produced for the test sample and the gradient of the line produced from the reference sample yields the total normal thermal emittance of the test surface.

Calculation Method (if necessary, please include correction method):

The ratio of output voltages from the test sample and the reference yields the thermal emittance on the assumptions that the blackbody and sample have equal temperatures and that for $T - T_a < 10°C$ the radiation terms may be approximated by

$$T^4 - T_a^4 = \overline{T}^3 (T - T_a)$$

where

$$\overline{T} = (T + T_a) / 2$$

Interpretation/Precision & Accuracy:

Reproducibility : 1-2 %

Accuracy : 5%

REPORTS ON SOLAR ABSORBERS

a (iii) REFLECTANCE

Date: February 19, 1986   Reporter's Name: J. Keller, EIR,
                                            CH-5303 Würenlingen

Country: Switzerland

Testing Title:

Spectral reflectance/absorptance (hemispherical irradiation, normal detection)
of absorber coatings

Abstracted Scope (200 words):

Measurement of the spectral reflectance of absorber coatings in the range of
wavelengths from 0.3 µm to 2.5 µm using a Zeiss PMQII/RA3 spectrometer.
The irradiation is hemispherical and the detection of the reflected radiation
is normal. An average value of the reflectance (or absorptance) over the
above range of wavelengths is obtained by weighing the output by an
appropriate solar spectrum.

Referred Document:

- Instruction Manual. Zeiss PMQII
- J. Keller, V. Kyburz, Th. Fisch, "Wärmeerträge und Kenngrössen von Sonnen-
  kollektoren. Bericht über die Prüfkampagne vom Sommer 1983". Eidg.
  Institut für Reaktorforschung (EIR), CH-5303 Würenlingen. Mai 1984

Principle of Measurement:

See "procedures"

Apparatus: Zeiss PMQII/RA3 Spectrometer

1: integrating sphere, 2: absorber sample, 3: shielding, 4: light source,
5: tipping mirror, 6: lenses, 7: variable inlet slit, 8: prism, 9: detectors
(photomultiplier, photoresistor)

Calibration: white reference sample with known spectral reflectance

Sampling:

sample size about 7 cm x 7 cm

Conditioning:

Procedures:

The integrating sphere of the spectrometer is illuminated by a light source.
The sample is put upon the circular opening at the top of the sphere.
Opposite to the sample there is a tipping mirror which transmits the beam
originating either from the sample or from a section of the sphere to a
variable inlet slit. This beam is spectrally split by a prism and detected
by a photomultiplier ($\lambda < 0.6$ µm) or a photoresistor ($\lambda > 0.6$ µm).
For every wavelength, in the first step the mirror is pointed at the section
of the wall. The inlet slit is varied until the amplifier output of the
detector reaches a given value of 100 %. In the second step the mirror is
tipped towards the sample and (at fixed position of the slit), the amplifier
output U is read.

Calculation Method (if necessary, please include correction method):

reflectance ρ: $\rho(\lambda) = U(\lambda) \cdot f(\lambda)$

(f: calibration function)

absorptance α: $\alpha(\lambda) = 1 - \rho(\lambda)$

average absorptance $\bar{\alpha}$
over the solar spectrum I_λ:

$$\bar{\alpha} = \frac{\int_{0.3 \, \mu m}^{2.5 \, \mu m} I_\lambda \alpha(\lambda) d\lambda}{\int_{0.3 \, \mu m}^{2.5 \, \mu m} I_\lambda d\lambda}$$

(values of I_λ: see annex)

Interpretation/Precision & Accuracy:

accuracy: $\pm$ 2 %

Date: 1 March '86   **Reporter's Name:** Sakae TANEMURA

Country: Japan

Testing Title:

Normal Spectral Reflectance(NSR) of Absorber Materials/Coating Elevated at Designated Temperature

Abstracted Scope (200 words):

NSR of opaque materials and/or coatings with mirror like smooth surface from 0.45 to 15.0 μm in wavelength is measured under the condition that the sample being heated at the temperature ranging from room temperature up to about 800 K.

To eliminate the affect of the thermal radiation emitted from the heated sample on the mesured NSR data, the unique optical configuration of the mesuring apparatus, which is exemplified in the "Apparatus" box in this report, is required.

To cover the above described wavelength, the optical elements such as light sorce, grating, width of slit, filter, and detector are properly provided.

Referred Document:

S.Tanemura et al, Journal of High Temperature Soc. of Japan, Vol 6, No.6, 210 (1980) (in Japanese).

Principle of Measurement:

Double beamed optical null method in the scanning spectrophotometry
(refer to the optical scheme described below).

Apparatus:
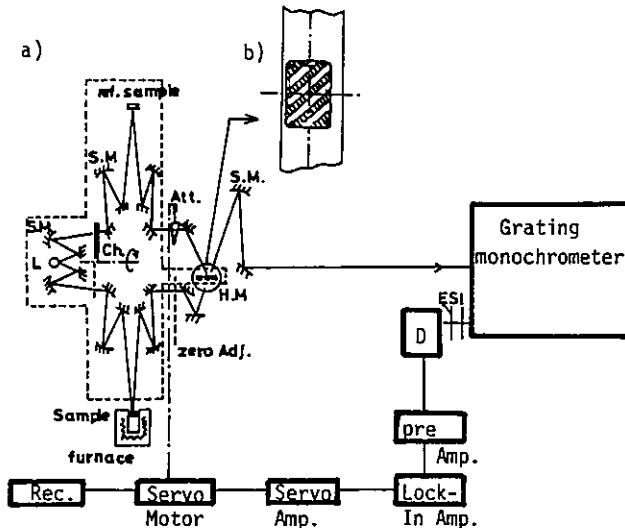
Fig.1,(a) Optical Scheme of the Reflectometer,
and (b) Schematics of the Used Half Mirror.

The shadowed stripes in Fig.1 (b) are mirrors coated with Al on the plate glasses. Half of the beam impinged to this half-mirror is eithe transmitted or reflected .

The beam from the light source is divided to the direction to either sample or reference standard by the copper (Ch) with 8Hz in frequency.

Tungsten lamp and Nichrome heater are provided for the light source for visible-NIR and that for IR wavelength range respectively.

The optical elements such as glating, filter(F1), width of the slit(S), and detector(D), are properly selected as the function of wavelength.

The recorded transmittance of the attenuator(Att) corresponds to the NSR of the heated sample.

The incidence angle both on the sample and reference is 5°.

Calibration:

Sampling:

size; 25(mm) x 25(mm) x 5 (mm, maximum thickness)

Conditioning:

(1) Checking of the flatness of the sample surface comparing with the concerned wavelength

(2) The sample should be pre-heated at a designated temperature at least 12 hrs. for the aging.

(3) Either Al or Au mirror freshly coated on glass substrate should be provided as the reference standard.

- 2 -

Procedures:

(1) Preheating of the sample described in the "Conditioning".
(2) Checking of the operational conditions of the spectrophotometer including of the signal and noise checking.
(3) Operation of the scanning of the photometer.
(4) Selection of the proper optical elements described in the "Apparatus".
(5) Readings of the recorded transmittance of the attenuator .
(6) Correction to the above readings due to the deviation from the perfect mirror for the used reference mirror. Hence, the absolute NSR values of the used reference mirror should be known in the prior to these procedures.

Calculation Method (if necessary, please include correction method):

$$r (o,o,\lambda,T) = R(0,0,\lambda)\tau(\lambda)$$

r: NSR of the sample at T K , R: Absolute NSR of the reference mirror at room temperature

τ: recorded transmittance of the attenuator.

Interpretation/Precision & Accuracy:

Precision: better than ± 0.02 after the confirmation of the dimensional precision of the used halh-mirror.

Reproducibility: better than ± 0.02 in reflectance units.

Date: 1 March '86   **Reporter's Name:** Sakae TANEMURA

Country: Japan

Testing Title:

Absolute Normal Spectral Reflectivity (AB-NSR) of Absorber Material
with Mirror Like Smooth Surface

Abstracted Scope (200 words):

In usual reflectance/reflectivity measurements, a mirror with metal (Al, Au,
Pt, Rh and etc.) coatin on glass or a metal plate polished like a mirror is
used as the reflectance standard. Hence it is necessary to know AB-NSR of the
standard mirror to correct the obtained NSR of the sample. The AB-NSR of the
absorber coating or substrate with mirror like smooth surface from 0.30 to
15.0 μm in wavelength might be relevant datum by itself to characterize the
material and interfaces of which complex dielectric function can be derived
under the ad-hoc assumption of the Kramers-Kronig relationship, because of
the extreamely high precision and accuracy in the normal AB-NSR measurements.
To cover the above described wavelength , the optical elements such as light
source, width of the slit provided to the entrance of the monochrometer, hight
of that, glating, filter, width of the slit provided to the entrance of the
reflectometer, hight of that and detector are properly provided.

Referred Document:

S.Tanemura et al, Abstract of 34th Semi-Annual Meeting of Research Achieve-
ments in G.I.R.I.Nagoya(22 June '83), 88(1983) (in Japanese).

H.E.Bennet et al, J.Opt. Soc. America, Vol 50, 1 (1960).

Bennet- Koehler type reflectometer with scanning spectrophotometer.

(The optical configuration of the reflectometer being slightly modified
from their original configuration. Refer the schematics of the optical
configuration described below.)

Apparatus:

Fig.1. The Schematics of the Right Hand Optical Configuration
with Sample in the Optical Path of the Present Reflectometer.
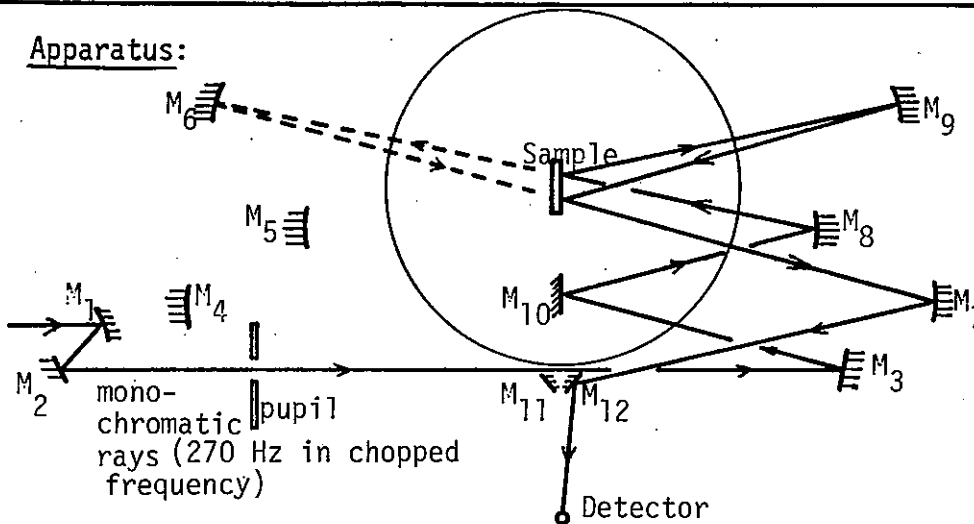
Used optical elements:
(1) Glatings:
1200, 600, 300,
120, & 60 lines/mm.

(2) Light Source:
Tungsten(400W,36
V) & Ni-Cr(60W,
10V).

(3) Filter:
6(0.42- 15.0μm)

(4)Detector:
Photomultiplier
& In-Sb, HCT(2
colours)

(5) Monochro.
ent. slit: width
& hight

(6) Refletometer
ent. slit: width
& hight.

If the sample being pulled up from the optical path, the sphe-
rical mirror M6 is introduced . Inthe case of the left hand
optical configuration, the sample is precisely rotated by 180°
in arc to face to mirror M4, M5 & M6 and mirror M10 being
removed from the optical path.
The incident angle of the beam impinged on the sample is 3° in
the 1st incidence and 6° in the 2nd incidence respectively.

Calibration:

Sampling:

size: 25(mm)x 25(mm)x 5(mm, maximum thickness)

Conditioning:
(1) Confirmation of the opacity of the sample from 0.30 to 15.0 μm in wave-
length. .
(2) Confirmation of the root mean square of the roughness (σ) of the sample
surface being less than 300 nm which is nearly equal to σ of flitglass.

Procedures:

(1) The reduced image of the pupil is made on the sample surface. Hence, the radius of the pupil shoul be adjusted in the manner that the image being enough small to the size of the sample or that of the target part to be measured.

(2) The adjustment of the sensitivity of the gain of the Lock-In amplifier for the optical configuration without sample.

(3) The adjustment of the high voltage applied to the photomultiplier detector for the optical configuration without sample in the uv and visible wavelength range.

(4) Measurement of the spectral signal Vrs at a designated wavelength λ for the right hand optical configuration under the inserting of the sample in the optical path.

(5) Measurement of the spectral signal Vro at the wavelength λ for the above described optical configuration without the sample

(6) Measurement of the spectral signal Vls at the wavelength λ for the left hand optical configuration under the inserting of the sample in the optical path.

(7) Measurement of the spectral signal Vlo at the wavelength λ for the above described optical configuration without the sample.

Calculation Method (if necessary, please include correction method):

$$r(\lambda)=[(Vrs/Vro)(Vls/Vlo)]^{1/4}$$

Interpretation/Precision & Accuracy:

Typycal example,
  Precision: $\Delta r/r$ being less than 0.2 % (best case: 0.05 %)

  Reproducibility :being less than 0.07 %

Date: 1 March '86   Reporter's Name: Sakae TANEMURA

Country: Japan

Testing Title:

Hemispherical Directional Spectral Reflectance(HDSR) of Absorber Materials at nearly room temperature.

Abstracted Scope (200 words):

If the homogeneous hemispherical ligt sourcce illuminate the sample, measured HDSR (r(π ,θ, λ), π : homogeneous hemispherical light source with π str in solid angle on the centere of the sample surface, θ : the polar angle of the reflected beam, and λ : wavelength) is equal to DHSR r(θ, π, λ) due to the reciprocity theorem of the optics. For the opaque material, Kirchoff's radiation theory derives the equation ε (θ, λ)= 1- r(θ, π, λ), where ε(θ,λ) means directional spectral emittance(DSE). Hence, it is worthwhile to obtain HDSR from 0.45 to 15.0 µm in wavelength and from 12 ° to 80 ° in polar angle for the absorber materials and/or coatings with and without optically smooth surface under the isothermal conditinns of the cavity wall of the Cavity type reflectometer with scanning spectrophotometer. To cover the above described wavelength range, the temperature of the cavity wall is required to be elevated at about 1000 K and the optical elements required to the scanning spectroscopy are properly provided .

Referred Document:

(1) R.V.Dunkle et al, Progress in Int. Res. on Thermodynamics and Transport Prop.(ed. by Amer. Soc. of Mech. Engr.), Academic Press, N.Y. pp541 (1962).
(2) S.Tanemura et al, Jornal of High Temp. Soc. of Japan, Vol 6, No.6, 210 (1980).
(3) M.Sando et al , Abstract of 28th Semi-Annual Meeting on Research Achievements in G.I.R.I. nagoya(26th June '80), 88(1980).

Principle of Measurement:

Heated cavity reflectometer with double beamed optical null method in scanning spectrophotometry

The Pt plate with polished smooth surface is used as the reference standard to monitor the spectral irradiance iluuminated hemisherically from the heated cavity wall on the sample. Pt plate should be heated up to the temperature equal to the that of cavity wall.

Apparatus:
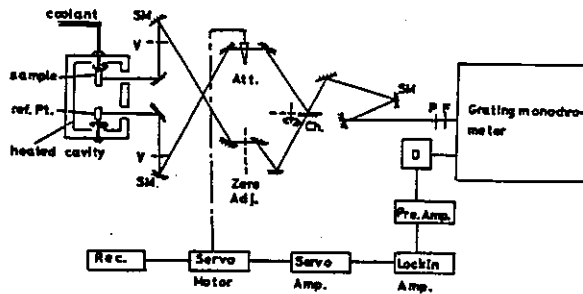
Fig.1. Optical Scheme of the Present Cavity Reflectometer with Double Beams Configuration.

Fig. 2 Schematic cross section of the cavity. The centere of tɛe sample at the vertical position views the front window in solid angle of 0.014 str.

The six segment heaters around the cavity wall and cavity apertures are controlled independentlly by the proportional-differential-integration controll device to realize the isothermality of the cavity.
Fifteen Cr-Al thermocouples are provided around the cavity wall, close to the cavity apertures and on the Pt reference.
The sample is cooled down about 15 °C by the water jet hitted on the rear surface of the sample.
The optical elements such as glatings, filters, slit width, and detectors are properly provided for the corresponding wavelength.

Calibration:

Sampling:

size: 25(MM) X 25(mm) X 5(mm, maximum thickness)

Conditioning:

(1) The confirmation of the isothermality of the heated cavity is essential. The allowance of the maximum temperatuture difference between the monitored temperatures at 15 different positions being less than ± 3.5 °C around the averaged cavity wall temperature of about 800 °C under the usual operational conditions.
(2) Diagnostic check of no water dew/ humidity on the sample surface is required.

Procedures:

(1) Preheating of the cavity wall at about 800 °C.
(2) Fixing tha sample to the holder induced in the cavity and the adjustment of the coolant temperature.
(3) Adjustment of the controll temperatures of the six segmented heaters to obtain the isothermality of the temperature of the cavity wall.
(4) Confirmation of the conditions described in the "Coditioning" in this report.
(5) Checking of the spectral signal and the adjustment of the sensitivity gain of the Lock-In amplifier and the applied high voltage to the photo-multiplier.
(6) Recording of the transmittance of the attenuator driven by the servo motor as the function of both wavelength and polar angle.
(7) Experimental confirmation of the precision of the reflectometer by using the mirror with Au coating on glass substrate and Pt plate cut from the same block of the platinum plate provided for the reference Pt plate under the same conditions as realized in the procedure (6).

Calculation Method (if necessary, please include correction method):

$r(\pi, \theta , \lambda) = \tau(\lambda)$

$\tau (\lambda)$: recorded transmittance of the attenuator.

If the isothermality of the cavity wall is not assured satisfactorily , the correction would be required under the sssumption of the model describing the fraction between the specular component of the reflected spectral irradiance and diffuse component of that due to the surface roughness and the model to calculate the geometrical configuration factors between the elements of the cavity wall having respective different temperatures and the elements of the sample surfaces.

Interpretation/Precision & Accuracy:

Typycal example under the isothermality of the heated cavity being confined into the condition described in the " Conditioning " in this report.

Precision : ± 0.03 in reflectace units.

Reproducibility : ±0.02 in the reflectance units.

REPORTING FORMAT

Date : 03/03/86   Reporter's Name :   Kazuo Taga

Country : Japan

Testing Title :

Testing Method for Reflectance of Selective Surface Coating
(by spectrum photo meter)

Abstracted Scope (200 words) :

1. This spectrum reflectance is measured by the spectrum photo meter with integrating sphere.

2. The solar reflectance is determined by product of the spectrum reflectance and the solar spectrum intensity.

Referred Document :

ASTM E903

Standard Test Method of Solar Absorptance Reflectance and Transmittance of Materials using Integrating Speres.

Principle of Measurement:

The solar reflectance is determined by product of the spectrum reflectance measured by the spectrum photo meter and spectrum internsity in AM2.

Apparatus: spectrum photo meter

Hitachi 323 Spectrophoto meter   (with Integrating Sphere)

1.mirror
2.prism
3.lamp
4.slit
5.sector mirror
6.specimen side
7.pbs cell
8.standard side
9.detector
10.sample cell
11.window
12.sub white plate
13.integrating sphere   14.standard cell
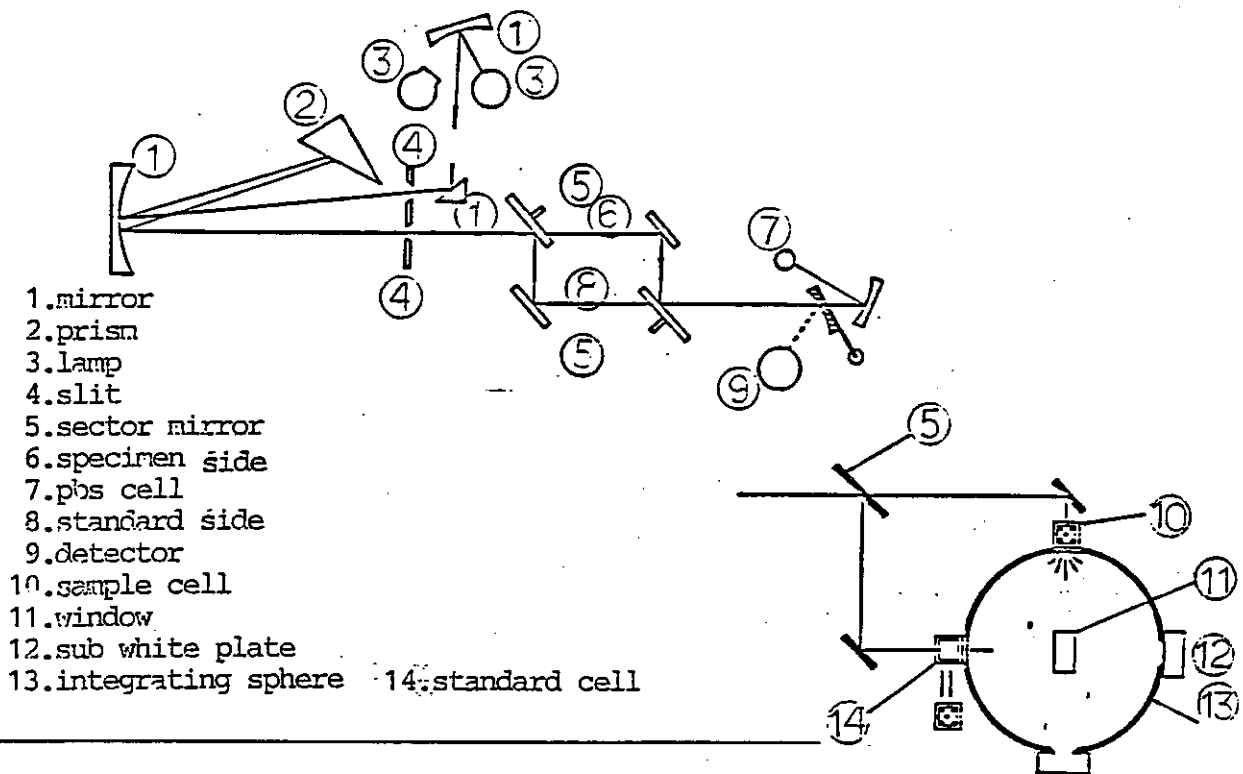
Integrating Sphere (optical flow chart)

Sampling:

20mm x 30mm   (flat plate specimen)

Conditioning:

Procedures :

1. Adjust indication of the reflectance of spectrum photo meter by using MgSO4 plate between the wave length from 340nm to 2200nm.

2. Set the specimen on the spectrum photo meter and measure it, for the reflectance at 100nm wave length intervrls.

3. The solar reflectance is determined by products of the spectrum reflectance and the solar spectrum intensity in air mass2.

$$Rs = \frac{\Sigma R_\lambda \cdot q_\lambda}{\Sigma q_\lambda}$$

Rs : solar reflectance for air mass2
R_λ : spectrum reflectance
q_λ : solar spectrum intensity for air mass2.

Calculation Method (if necessary, please include correction method) :

Interpretation/Precision & Accuracy :

Accuracy : ± 0.5%

REPORTING FORMAT   Designation:
                       A-O-LA

Date: 1986-03-16   Reporter's Name:

Country: Sweden

Testing title:

Measurement of reflectance
15°/diffuse, spectral

Abstracted Scope (200 words):

To measure the reflectance of solar collector absorbers in the
wavelength region 2,5-14 µm at descrete wavelengths with 15/d
geometry of the measurement.

Referred Document:

Principle of Measurement:

The 15/d reflectance is compared with the reflectance of a gold
reflectance standard using a diffusely gold coated integrating
sphere. Diffuse gold standard is used for diffusely reflecting
samples and specular standard for specular samples.
Single beam.

Apparatus:

Monochromator Jobin Yvon HR 1000.
Stabilized glowing rod source with chopper pyrometer.
Cut-on filters.
100 mm diameter integrating sphere (diffuse gold).
InAs- and MCT-detector.
Lock-in amplifier.
Standards: Specular gold mirror, diffuse gold reflector.
Computer: HP 9825 A
Voltmeter: HP 3455 A

Calibration:

Gold reflector is calibrated against $BaSO_4$ in 1-2 μm region.
Wavelength scale is calibrated with a IR He-Ne laser and with a
conventioned 633 nm laser.

Sampling:

Samples must be flat, opaque and larger than 3x3 cm^2.

Conditioning:

Procedure:

The glowing rod source is stabilized at 1100 °C using a pyrometer and feedback.

The integrating sphere is placed on the monochromatic side of the monochromator and the sample is irradiated with 15° angle of incidence.

The detector output is measured with the sample port of the sphere open (do), with the sample at the port (dx), with the gold mirror at the port (dm) and with the diffuse gold reflector (dd) at the port.

The degree of diffuseness D of the sample is estimated by measuring the reduction of the detector signal when placing the sample and the gold mirror at a distance from the sample port.

Calculation method:

$$\rho_1 = \rho_m \cdot (dx-do)/(dm-do) \qquad \rho_m = 0.99$$

$$\rho_2 = \rho_d \cdot (dx-do)/(dd-do) \qquad \rho_d = 0.95$$

$$\rho_3 = D\rho_2 + (1-D)\rho_1$$

$$\rho = -6 + \sqrt{36+13 \cdot \rho_3} \quad \text{(empirical correction formula for the sphere transmission characteristics)}$$

Interpretation:

Precision: <±0.01   2.5–5 µm   95 % confidence level
            <±0.03   5–14 µm   95 % confidence level

Accuracy: <±0.05   (not verified estimation)

Date: December 1986   Reporter's Name: Michael G. Hutchins

Country: United Kingdom

Testing Title: DETERMINATION OF THE SOLAR ABSORPTANCE/REFLECTANCE/
TRANSMITTANCE FROM SPECTRAL MEASUREMENTS AT NEAR-
NORMAL INCIDENCE

Abstracted Scope (200 words):

Spectral reflectance and/or spectral transmittance measurements are made using a Beckman 5240 integrating sphere spectrophotometer in the spectral range 0.3 - 2.5 µm. In reflectance mode radiation is incident on the surface at 5° to the surface normal. In transmittance mode the radiation is incident normally on a surface placed over a sphere entry port.

Solar optical properties are calculated by convoluting the spectral reflectance curve with a standard solar spectral distribution curve for air mass 2 using a method of 20 selected ordinates. One of two solar spectral distributions may be used (1,2).

The measurement procedure is automated using an Apple IIe microcomputer.

Referred Document:

1.   Wiebelt J.A. and Henderson J.B., J. Heat Transfer 101, 101, 1979.

2.   Moon P., J. Franklin Inst., 230, 583, 1940.

Principle of Measurement:

Near-normal hemispherical spectral reflectance or transmittance of incident radiation.

Apparatus:

Beckman 5240 uv/vis/nir integrating sphere spectrophotometer using photomultiplier for uv/vis detection and PbS for nir detection.
6" diameter integrating sphere coated with barium sulphate white reflectance standard. Double beam ration recording principle of operation.
Apple IIe microcomputer for calculation of optical properties.

Calibration:
For diffuse surfaces : barium sulphate
For highly specular surfaces : aluminium mirror

Sampling:

Minimum sample size : 30mm x 30mm
Maximum sample size : 100mm x 100mm

Conditioning:

None

Procedures:

1. Sample cut to size and cleaned by air blowing.
2. Instrument is calibrated using two barium sulphate reference plates.
3. Sample mounted on sphere wall (rear for reflectance ; front for transmittance) and reflectance or transmittance measured for scan from 2500nm - 350nm.
4. Spectral data at 1nm intervals are sent to an Apple IIe microcomputer.
5. Microcomputer executes calculation to determine surface optical properties, e.g. α_s, τ_s or ρ_s.

Calculation Method (if necessary, please include correction method):

20 selected ordinates for air mass 2 solar spectral distribution giving

$$\alpha_s = \frac{1}{20} \sum_{i=1}^{20} \alpha_i \text{ , where } \alpha_i \text{ is the absorptance at each of the twenty}$$

selected ordinates and is found from $\alpha_i = 1 - \rho_i$.

$$\tau_s = \frac{1}{20} \sum_{i=1}^{20} \tau_i \text{ , where } \tau_i \text{ is the transmittance at each of the twenty}$$
selected ordinates.

Interpretation/Precision & Accuracy:

Precision : reproducible to one part in 10^3.
Accuracy : 1-2%.

REPORTS ON SOLAR ABSORBERS

b. THERMAL PROPERTIES

REPORTING FORMAT

Date:	February 25, 1987

Reporter's name: P.A. van Nijnatten

Country: Technisch Physische Dienst TNO-TH
(TNO Institute of Applied Physics)
P.O. Box 155, 2600 AD DELFT
The Netherlands

Testing title: DETERMINATION OF THE TOTAL NORMAL EMITTANCE OF A SAMPLE
USING SPECTRAL REFLECTANCE MEASUREMENTS

Abstracted Scope (200 words):

The spectral reflectance is measured in the spectral range from
4000 cm^{-1} - 200 cm^{-1} ($2,5$ μm - 50 μm).
From these measurements the spectral- and total emittance is calculated, the
latter using the spectral energy distribution of planck's black-body radiator
at a designated temperature.

Referred document:

none.

Principle of measurement:

Spectral measurement of specular reflected radiation.

Apparatus:

Perkin Elmer 883 IR spectro-photometer with specular reflectance accessory.

Calibration:

Gold coated glass substrate (R = 0,975).

Sampling:

Minimum sample size: Ø 20 mm
Sample must be flat!

Conditioning:

Near normal incidence (6°)

Procedures:

1. Sample is cleaned
2. Spectral reflectance is measured (1 cm^{-1} wavenumber intervals)
3. Spectral emittance is calculated
4. Total emittance $\varepsilon_\perp$ is calculated using the spectral energy distribution of the black body radiator.

Calculation Method (if necessary, please include correction method):

$$\varepsilon_\perp (\lambda^{-1}) = 1 - \rho_\perp (\lambda^{-1})$$

λ^{-1} : wave number

$\rho_\perp(\lambda^{-1})$: spectral reflectance

$\varepsilon_\perp(\lambda^{-1})$: spectral emittance

$$\varepsilon_\perp = \frac{\int_0^\infty \varepsilon_\perp(\lambda^{-1}).E(\lambda^{-1},T).d(\lambda^{-1})}{\int_\rho^\infty E(\lambda^{-1},T).d(\lambda^{-1})} \approx \frac{\sum_{\lambda^{-1}=200\ cm^{-1}}^{\lambda^{-1}=4000\ cm^{-1}} \varepsilon_\perp(\lambda^{-1})E(\lambda^{-1},T).\Delta(\lambda^{-1})}{\sum_{\lambda^{-1}=200\ cm^{-1}}^{\lambda^{-1}=4000\ cm^{-1}} E(\lambda^{-1},T).\Delta(\lambda^{-1})}$$

$\varepsilon_\perp$: total normal emittance (at temp. T)

$E(\lambda^{-1},T)$: spectral energy distribution of planck's black body radiator at temperature T.

$\Delta(\lambda^{-1})$: wavenumber interval of integration, usual 1 cm^{-1}.

Interpretation/Precision & Accuracy

estimated accuracy: 0,5% ($\varepsilon_\perp < 0,5$) - 1% ($\varepsilon_\perp > 0,5$)
precision   : better than 0,2%

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Melting and Crystallization Temperatures by Thermal Analysis

Abstracted Scope (200 words):

This method covers the determination of transition temperatures such as the melting and crystallization temperatures of materials by differential scanning calorimetry (DSC) and differential thermal analysis (DTA). The normal operating temperature range is from -120 to 600°C. With special equipment, the temperature range can be extended.

Computer or electronic based instruments, techniques, or data treatment equivalent to this test method may be used.

Referred Document:

ASTM E 794   Standard Test Method for Melting and Crystallization
             Temperatures by Thermal Analysis

ASTM D 3418 Standard Test Method for Transition Temperatures of Polymers
             by Thermal Analysis

Principle of Measurement:

The method consists of heating or cooling the test material at a controlled rate in a controlled atmosphere through the region of a transition, such as fusion or crystallization. The difference in heat flow or temperature between the test material and a reference material due to energy changes in the material is continuously monitored and recorded. Absorption or release of energy results in endothermic or exothermic peaks.

Apparatus:

A differential scanning calorimeter, capable of heating or cooling at rates up to at least $10^{\circ}C/min$ and of automatically recording the differential energy input between the sample and reference material both to the required sensitivity and precision. Typically, the differential sensitivity should be sufficient to provide specimen temperature readability to within $1^{\circ}C$.

A differential thermal analyzer, capable of heating or cooling at rates up to at least $10^{\circ}C/min$ and of automatically recording the differential temperature between sample and reference material both to the required precision; typically within $1^{\circ}C$.

Specimen pans, for DSC, composed of aluminum or other metal of high thermal conductivity. For DTA, sample cups or tubes composed of borosilicate glass, alumina, or quartz may be used. The specimen pans or tubes must not react with the sample.

Nitrogen, or other inert purge gas.

An analytical balance, with a capacity greater than 15 mg, capable of weighing to the nearest 0.01 mg.

Sampling:

Powdered or liquid samples should be thoroughly mixed and sampled from by removing portions from different parts of the container which are then combined and mixed. Film, sheet, molded or pelleted samples can be cut, punched, or bored to obtain pieces that fit the sample containers.

Conditioning:

Materials that have a residual thermal history may require a preliminary thermal cycle to above the melting point to avoid interference with the measurement of the transitions of interest. Care should be taken to avoid thermal decomposition of the test specimen.

Procedures:

1. Use a sample weight appropriate for the material to be tested; about 5 mg. for first order transitions and 10 to 20 mg for glass transitions. An appropriate sample weight results in 25 to 95% scale deflection of the recording system when acceptable sensitivity is used.

2. Ensure intimate thermal contact between the sample, the sample pan or tube and thermocouple, where used. Good thermal contact is also required between the sample container and the test apparatus.

3. Load the specimen into the instrument chamber, purge the chamber with nitrogen or other inert gas at a flow rate of 10 to 50 cm^3/min during the experiment.

4. Heat the specimen rapidly to 50°C below the transition temperature of interest and equilibrate.

5. Heat the specimen at 10°C/min through the transition until the baseline is reestablished above the transition. Hold at this temperature for about two min and then cool the specimen, at the same rate, down through the transition until the baseline is reestablished below the transition.

6. Reweigh the specimen after completion of scanning and discard. Report any mass loss observed.

Calculation Method (if necessary, please include correction method):

From the resultant curve, measure the following points:

T_s = temperature of the first deviation from the baseline, °C
T_e = extrapolated onset temperature for fusion, °C
T_p = melting peak temperature, °C
T_f = temperature of return to baseline, °C
T_n = extrapolated temperature of crystallization onset (nucleation), °C
T_c = temperature of crystallization peak, °C

Report similar temperatures for other transitions.

Interpretation/Precision & Accuracy:

Repeatability (Single Analyst): Two averages for T_s (95% confidence level) should be considered suspect if they differ by more than 7.9°C.
Two averages for T_c, T_p, T_f, T_n, and T_e should be considered suspect (95% confidence level) if they differ by more than 3.1°C.

Reproducibility (Multilaboratory): Two averages for the following should be considered suspect (95% confidence level) if they differ by:
40°C for T_s for broadly melting material, 7.1°C for narrowly melting material, 5.9°C for T_e, T_p, T_n and T_c, and 19°C for T_f.

REPORTING FORMAT   A/G/HWA/HWNA/SS/SL/SD-T-ST

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Heats of Fusion and Crystallization by Differential Scanning Calorimetry

---

Abstracted Scope (200 words):

This test method covers the determination of the heat of fusion and crystallization by differrential scanning calorimetry. It is applicable to solid samples in granular form or in any fabricated shape from which an appropriate specimen can be cut and to liquid samples that crystallize within the range of the instrument.

The normal operating temperature range is from -120 to 600°C. This temperature range can be extended, depending on the instrumentation used. This test method is generally applicable to thermally stable materials with well defined endothermic or exothermic behavior.

Computer or electronic based instruments, techniques, or data treatment equivalent to this test method may be used.

Referred Document:

ASTM E 793   Standard Test Method for Heats of Fusion and Crystallization by Differential Scanning Calorimetry

ASTM D 3417 Standard Test Method for Heats of Fusion and Crystallization of Polymers by Thermal Analysis

Principle of Measurement:

Differential scanning calorimetry (DSC) is a technique in which the difference in energy inputs into a substance and a reference material is measured as a function of temperature, while the substance and reference material are subjected to a controlled temperature program. Differences in the amount of energy required to maintain the test specimen and reference at the same temperature are used to determine heats of transformation.

Apparatus:

A differential scanning calorimeter capable of heating and cooling rates up to at least 10°C/min and of automatically recording the differential heat flow between the sample and reference material to the required sensitivity and precision. The instrument should have sufficient heat flow sensitivity to provide a precision within 1% and a time base precision of within 1%.

A planimeter or other method of area measurement with an accuracy and precision within 1%.

Specimen holders, inert to the sample, composed of aluminum or other material of high thermal conductivity.

Nitrogen, or other inert gas supply, for purging purposes.

A balance, with a capacity greater than 15 mg, capable of weighing to the nearest 0.01 mg.

Sampling:

Powdered or granular specimens should be mixed thoroughly prior to sampling and should be sampled by removing and then blending portions taken from various parts of the container. Liquid samples may be sampled directly after stirring.

Conditioning:

A preliminary test cycle to remove previous thermal history, e.g., residual thermal stresses, or annealing above the glass transition temperature of polymers such as crystallizable polystyrene, may be required before valid test data are taken. Comparison of initial and subsequent test runs can be used to determine whether or not conditioning is required.

Procedures:

1. Into a clean specimen holder, weigh 1 to 15 mg of specimen.

2. Seal the specimen holder with a lid under ambient conditions, minimizing the free space between sample and lid. Hermetic sealing in an inert atmosphere may be desirable for specimens sensitive to oxidation.

3. Load the specimen into the instrument chamber and purge with nitrogen or other inert gas throughout the experiment at 10 to 50 cm^3/min.

4. Select the appropriate heat flow axis and time axis sensitivities to yield an area of 30 to 60 cm^2 under the fusion endotherm, or crystallization exotherm.

5. The specimen may be heated rapidly to 50°C below the melting (or other transition) temperature and allowed to equilibrate.

6. Heat the specimen at 10°C/min through the transition, until a baseline is reestablished above the endotherm or exotherm.

7. Hold the specimen at this temperature for 2 min.

8. Cool the specimen at 10°C/min through the transition until baseline is reestablished below the endotherm or exotherm. Reweigh the specimen after completion of scanning. Discard the data if there is mass loss greater than 1% or evidence of reaction with the sample holder.

Calculation Method (if necessary, please include correction method):

$$H = (ABT/W) (H_S W_S / A_S T_S B_S) \quad , \text{ where}$$

H   = heat of transition of the sample, J/g
H_S = heat of transition of the standard, J/g
A   = peak area of the sample, cm^2
A_S = peak area of the standard, cm^2
B   = time scale used for the sample, min/cm
B_S = time scale used for the standard, min/cm
W   = mass of the sample, mg, W_S = mass of the standard, mg
T   = Y-axis sensitivity used for the sample, mW/cm
T_S = Y-axis sensitivity used for the standard, mW/cm.

Interpretation/Precision & Accuracy:

Repeatability (single analyst): 2.8% coefficient of variation with 88 degrees of freedom. Two such averages should be considered suspect (95% confidence level) if they differ by more than 7.8%.

Reproducibility of pure materials (Multilaboratory): 3.0% coefficient of variation at 58 degrees of freedom for materials melting or crystallizing over a narrow temperature range. Two such results should be considered suspect (95% confidence level) if they differ by more than 8.6%. Results for broad melting range polymers should be considered suspect (95% confidence level) if they differ by more than 23%.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

   Linear Thermal Expansion of Solid Materials by Thermodilatometry

Abstracted Scope (200 words):

This method covers determination of the linear thermal expansion of solid
materials using thermomechanical analysis techniques. It is applicable to
solid materials that exhibit sufficient rigidity over the test temperature
range such that the sensing probe does not produce indentation of the
specimen.

The lower limit of coefficient of linear thermal expansion normally
measured with this method is 5 um/(m.°C). The method may be used at lower
or negative expansion levels with decreased accuracy and precision.

The method is applicable over the temperature range from -120 to 600 °C.
This temperature range may be extended depending upon the instrumentation
and calibration materials used.

Referred Document:

ASTM E 831   Standard Test Method for Linear Thermal Expansion of Solid
             Materials by Thermodilatometry

Principle of Measurement:

This method uses a thermomechanical analyzer or similar device to determine the linear thermal expansion of solid materials when subjected to a constant heating rate. Changes in specimen length are electronically recorded as a function of temperature. The coefficient of linear thermal expansion can be calculated from these recorded data.

Apparatus:

A thermomechanical analyzer, or similar device consisting of:

1. A specimen holder and probe, constructed of low thermal expansion materials, that transmits changes in specimen length to a transducer. The probe shape and size shall not cause indentation of the specimen over the temperature range of interest with the load applied. Flat, circular probes of 2 to 5-mm diameter are normally used for this method.

2. A transducer, with a sensitivity of at least 1 mV/um of probe movement, for sensing movement of the probe resulting from changes in specimen length and translating these changes into electrical signals suitable for input into a recording device.

3. A furnace with provision for uniformly heating and cooling the test specimen over the temperature range of interest.

4. A temperature sensing element for measuring the specimen temperature.

5. A means of purging the specimen environment with a dry inert gas such as nitrogen or helium.

6. A means of recording changes in specimen length versus temperature.

7. Calipers, capable of measuring linear dimensions to ± 25 um.

Sampling:

Specimens shall be between 2 and 10 mm in length and have flat and parallel ends to within 25 um. Lateral dimensions shall not exceed 10 mm.

Specimens are normally measured as received. Heat or mechanical treatment to relieve stresses, distortion, etc., should be noted in the report.

Conditioning:

Procedures:

1. Calibrate the apparatus, both for length change and temperature, using materials of known melting point and thermal expansion.

2. Measure the specimen length in the direction of the thermal expansion test at room temperature, install the specimen in the apparatus, and bring the specimen temperature to at least 20°C below the lowest temperature of interest after purging with inert gas.

3. Place weights, typically 1 to 3 g, on the probe to ensure contact between the probe and specimen.

4. Heat the specimen at a constant heating rate, normally 5°C/min, over the desired temperature range and record changes in specimen length.

5. Make a run using the same test conditions, without a test specimen installed, to establish a baseline for the expansion of the apparatus.

6. Test at least three different specimens of the same material.

Calculation Method (if necessary, please include correction method):

$$a_m = (L_{sp} \times k)/(L \times T) \qquad \text{where}$$

a_m = mean coefficient of linear thermal expansion, um/(m.°C)
L = specimen length at room temperature, m
L_{sp} = change of specimen length, um
T = temperature difference over which the change in specimen length is measured, °C
k = calibration coefficient which is determined by measuring a reference sample having a known expansion coefficient and solving the equation for k

Interpretation/Precision & Accuracy:

Repeatability: Two results obtained by the same laboratory and instrument should be considered suspect (95 % confidence level) if they differ by more than 2.7 um/(m.°C).

Reproducibility: Two averages (each the average of duplicates) obtained by different instruments or laboratories should be considered suspect (95 % confidence level) if they differ by more than 3.1 um/(m.°C).

Accuracy is anticipated, from calculations, to vary with specimen size and magnitude of the coefficient of thermal expansion from <2 to >15 %.

REPORTS ON SOLAR ABSORBERS

C. SERVICE REQUIREMENT PROPERTIES

C (i) COATING

Date: 4/2/86   Reporter's Name: A.J. Faber

Country: The Netherlands

Testing Title: climatic cycle test

Abstracted Scope (200 words):

The climate cycle test is a newly developed test. The absorber plates are placed in a small climatic chamber, in which temperature and moisture contents can be controlled; in addition a solar irradiance of 1000 W/m^2 can be created on the absorber plates with a small solar simulator.
One test cycle (duration 4 hours) simulates one day.
The test cycle can be divided in three periods:

- period 1: temperature 40^0C
            humidity 100%
            duration 1 hour

- period 2: temperature 80^0C
            humidity < 50%
            solar irradiance 1000 W/m^2
            duration 1 hour

- period 3: cooling down of the absorber plates from 80^0C to
            ambient temperature;
            duration 2 hours.

The total duration of the test is 120 cycles; this is not a fixed number.

Referred Document:

   "DEVELOPMENT OF TESTPROCEDURES
   FOR RELIABILITY AND DURABILITY
   OF SOLAR COLLECTORS:
   ACCELERATED AGING/CORROSION TESTS
   ON ABSORBER PLATE MATERIALS AND   Ing. R.J.A. Blankers - Metaalinstituut
   COATINGS"   Ir. F. Simonis   - TPD-TNO
   - final report - March 1, 1984   Ir. A.J.T.M. Wijsman - TPD-TNO

Principle of Measurement:

One test cycle (duration 4 hours) simulates one day.

Apparatus:

1. Climatic chamber; 300 l, Köhler
2. Solar simulator, max. 1000 W/m^2, 25 lamps (Philux 40 W/220 V), lamps are air cooled
3. By water heated plate
4. Test sample
5. Automatically served water drain
6. Automatically served door (open/closed)
7. Thermocouple
8. Controlled heating element (100 W), 40^{0}C
9. Water dosing pump
10. Centrifugal water pump (7,5 l/min)
11. Thermostat bath – 9 l
12. Controlled heating element (3000 W), 80^{0}C
13. Controlled cooling element
14. Mixer

Calibration:

Sampling:

The dimensions of the absorber samples are 300 x 200 mm.

During the test the temperature of one test sample (4) is measured and recorded with a Chromel-Alumel thermocouple (7) and a Kipp recorder.

Conditioning:

For the testing of absorber materials/coatings the following climatic test cycle is ran through:

1 hour, 40^{0}C, 100% relative humidity (RH)

1 hour, 80^{0}C, < 50% RH, solar irradiance 1000 W/m^2

2 hours of cooling down from 80^{0}C to ambient temperature.

The duration of one cycle is 4 hours.

Procedures:

All actions, to run through a test circle are accomplished automatically by a double watch and two roller switches.

One test cycle has the following automatic actions:

a. Start of the test:
   - the door of the chamber (6) is closed
   - the water drain (5) closes
   - the water dosing pump (9) brings 2 1 demi-water in the chamber
   - the heating element (8) is switched on.

   The humidity in the air of the chamber increases because of the heating of water at the bottom of the chamber. Upon colder parts in the chamber, such as the test samples, water is condensing.

b. After one hour phase 2 starts:
   - the heating element (8) at the bottom is switched off
   - the door of the chamber (6) is opened
   - the water drain (5) opens
   - the heating element (12), the pump (10), the mixer (14) and the solar simulator are switched on.

c. After an other one hour starts the cooling down period (2 hours)
   - the solar simulator is switched off
   - the heating element (12) is switched off, the cooling element is switched on
   - after ± 1.5 hour the pump (10) stops.

   The test cycle is ran through and a new test cycle starts.

Interpretation

The changes in the absorber materials/coatings caused by a test are inspected in two ways:

1. visually

2. by measurement of the optical properties of the material before and after the test.

The optical properties measured are the solar absorptance α and thermal emittance ε.

REPORTING FORMAT   Designation:

                                    AC - R - ST

Date: 1986.2.12   Reporter's Name: Takasi   Odaira

Country: Japan

Testing Title:

      Testing Method of Strike Transformation for Coating Films

---

Abstracted Scope(200 words):

   When the strike part is dropped to the surface of coatings,
the resistance for the shock are checked by cracking,peeling,
etc.
   The strength of strike is controled by the weight of strike part
and distance from the test piece surface.

Referred Document:

   JIS K 5400 Testing Methods for organic Coating

Principle of Measurement:

Observe the defect;peeling,cracking,etc;on films of test piece that tranceformed by the strike.

Apparatus:
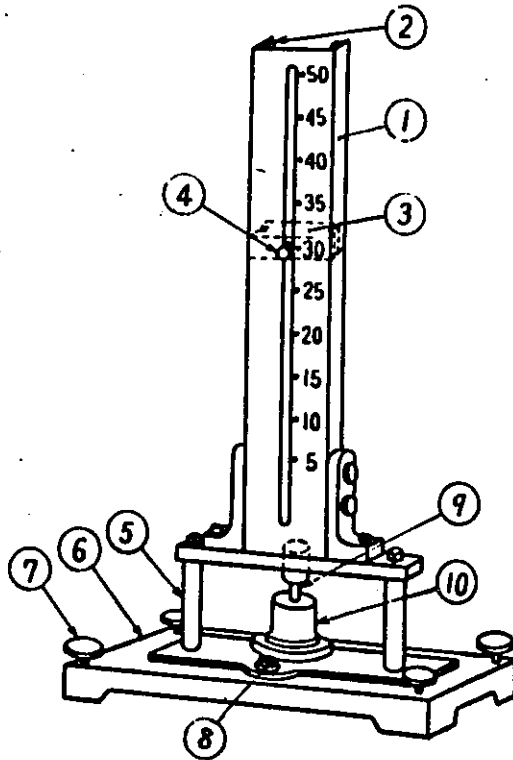
① frame
② guide rail
③ weight
④ pin
⑤ support
⑥ bed
⑦ levering screw
⑧ water level
⑨ strike part
⑩ stand

Calibration:

Sampling:

200mm × 100mm × 0.6mm

Conditioning:

room temperature

Procedures :

1. Let the test piece alone the room tempreture for one hour.
2. Put the testing machine on the level plane.
3. Set the strike part and the stand on the testing machine.
4. Hold the test piece between the strike part and the stand.
5. Drop the weight (500±1g) on the strike part from the estab-
   lished height.
6. Take out the test piece from the testing machine without
   the damage.
7. Observe the cracking and peeling,etc,of the corting films.

Caluclation Method (if necessary,please include correction method):

Interpretation/Precision & Accuracy :

The results of tests is decided on an agreement connected with
each properties.

REPORTS ON SOLAR ABSORBERS

d. DURABILITY

d (i) DURABILITY TO STAGNATION TEMPERATURE

Designation :
AC-D-LA

Date : 03/04/86   Reporter's Name :   Kazuo Taga

Country : Japan

Testing Title :

Testing Method for Stagnation of Absorber Plate

Abstracted Scope (200 words.) :

The durability of the absorber plate under the condition of stagnation is evaluated by this method.
The absorber plate is in the casing with glass cover and is exposed in the sunshine for a year .

The appearance, solar absorptance and solar emissivity of the absober plate is observed.

Referred Document :

   Y.Asano et al, Abstract of 54th Annual Symposium of Metal Finishing Soc. of Japan(25 Nov. 1976), 156(1976) (in Japanese).

Principle of Measurement:

1. Observation of the appearance
2. Measuring the solar absoptance and emissivity of the absorber plate after the stagnation condition

Apparatus:

    mountiong tilt angle : 30 degree
              azimuth   : 0 degree

    casing : The casing of the solar collector with the insulation and the glazing is used as a substitute for the casing of this test.

Calibration:

Sampling:

    The specimens are necessary preparing three pieces above the size of 60 mm x 60 mm.
    The average value is adopted.

Conditioning:

1. The appearance of the absober plate is directly observed by a looker.
2. The solar absoption of the plate is measured by α - meter.
3. The solar emissivity of the plate is measured by the emissometer.

Procedures:

1. Preparing three pieces of specimens above the size of 60mm x 60mm.
2. Observing the appearance and measuring the solar absorptance and emissivity of the specimens before the stagnation test.
3. Keeping the specimens in the casing with the insulation and setting the glass cover, and then mounting the case in the stationalry position with the fixed azimuth and tilt angle (azimuth : 0 degree, tiltangle : 30degree).
4. Observing the appearance and meausuring the solar absoptance and emissivity of the specimens after one year.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

Date: December 1986   **Reporter's Name:**   Michael G Hutchins

Country: United Kingdom

Testing Title: Outdoor exposure to stagnation conditions for
solar absorbers and solar glazings

Abstracted Scope (200 words):

A purpose built outdoor durability trials station located on the roof of
the Science building has been commissioned. The station is instrumented
to log solar irradiance (total), humidity, ambient temperature and
sample temperatures and these data are subsequently stored on tape and then
processed through a standard statistical package to yield daily average
values and bands of exposure conditions.

Solar absorber surfaces are mounted in purpose built boxes, which are single
glazed. Samples of 100mm x 100mm are normal and each box may take up to
40 samples. The boxes face due south and are inclined at 45° to the
horizontal. At periodic intervals samples are removed to the laboratory
where routine tests to check surface optical properties and composition
are undertaken prior to returning them for further exposure.

Referred Document:

ASTM E781 Evaluating absorptive solar receiver materials when exposed to
conditions simulating in solar collectors with cover plates

Principle of Measurement:

The changes in surface optical properties, surface microstructure, and chemical composition are measured and attempts are made to correlate such changes with surface temperatures and solar irradiance experienced during the period of outdoor exposure.

Apparatus:

Purpose built Unistrut support frame:
0.75m x 0.75m purpose built wooden framed, single glazed durability boxes with vent holes and 75mm glass fibre rear insulation,
Kipp-Zonen CM5 pyranometer;
K-type thermocouples;
Christie 248 24 channel datalogger with integral read-write tape facility;
BBC Model B microcomputer with link to PRIME mainframe for data processing.

Calibration: Various

Sampling:

Samples are monitored at one month after initial exposure and thereafter at six monthly periods

Conditioning:

None

Procedures:

1. Samples are cut to size
2. An initial characterisation which involves measurement of solar absorptance and thermal emittance, examination of surface microstructure and chemical composition using electron microscopy, x-ray diffraction, x-ray microanalysis and ion beam analysis is carried out. Standard size of a set of samples is 12.
3. Samples are mounted in durability boxes.
4. On return to laboratory surface optical properties are monitored. If no deleterious changes are observed samples are returned to the trials station. If significant changes are observed more detailed compositional and microstructural examinations are performed.
5. Records of solar irradiance and surface temperatures are compiled on a daily basis from tapes which are read once per week. Frequency of sampling is at 15 minute intervals.

Calculation Method (if necessary, please include correction method):

None

Interpretation/Precision & Accuracy:

Difficult to quantify at this stage

REPORTS ON SOLAR ABSORBER

d (ii) DURABILITY TO A DESIGNATED OPERATING CONSTANT TEMPERATURE (THERMAL DEGRADATION)

REPORTING FORMAT

Designation:

A – D – LA

Testing Title: Accelerated Ageing Test at Constant Temperature
for Solar Absorber Coatings

Abstracted Scope (200 words):

Samples are exposed in ovens with constant temperature
loads. The optical properties as a function of the
exposition-time give the degradation curve for one
temperature. Different temperatures effect only a change
in the time-scale of the degradation curves (if there is
always the same chemical degradation process). The scaling
factors with respect to a reference degradation curve obey
an Arrhenius' law, which allows the extrapolation to lower
temperatures and so an estimation of the life-time.

Referred Document:

M. Köhl, K. Gindele and M. Mast:
"Temperature Resistance of Copper Oxide Solar Absorber
Coatings"
Proc. of the 5th Int. Sonnenforum, Berlin, 1984 S.167

Principle of Measurement:

Optical properties as a function of ageing time.

Apparatus:

Ovens for temperatures up to 1000K.
Spectral and radiometric measuring devices for the
optical properties.

Calibration:

Sampling:

Plane samples from solar absorbers.

Conditioning:

Ageing processes must be caused by chemical processes or
diffusion. There may be no change of the process and no
phase-transition in the tested temperature range.

Procedures:

A sample of the absorber is periodically exposed to the temperature load after measuring the optical properties. Other samples of the same absorber are treated in the same way at other temperatures (at least three).

The time scale of the degradation curves gives the accelerating factor a (T) by imaging the curves on the reference curve at T_o.

Calculation Method (if necessary, please include correction method):

The activation energy $\emptyset$ of the degradation process is given in the slope of the straight line

$$\log a \ (T) = -\frac{\emptyset}{R} \cdot \frac{1}{T} + C \ (T_o)$$

Extrapolation of the straight line yields the acceleration factor for any other temperature for the imaging of the reference degradation curve to the time scale of this temp..

Interpretation/Precision & Accuracy:

This method allows an estimation of the degradation of solar absorbers for working temperature load.
The relevance and the accuracy is under investigation.

Date: 24.02.86   **Reporter's Name:** Ulrich FREI
Country: Switzerland
Testing Titel:

Degradation of absorber coatings by <u>exposition</u>
<u>under constant temperature.</u>

Abstracted Scope (200 words):

The long-therm behavior of a solar collector is
essentially influenced by the durability of the absorber
coating. The following conditions can happen on an absorber
coating in a flat plate collector:

A: temperatures from the minimal ambient temperature
up to the maximum stagnation-temperature

B: Temperature/moister influence caused by condensation
and or rain penetration. Load range: Ambient condi-
tions up to 100°C/100% rel.humidity

C: Solar irradiation with or partly without uv-radiation,
depending on the cover

For the qualification, an absorber coating has to be checked
under controlled conditons (A,B,C). Before and after the
exposition the following analysis should be done:

- solar absorptance (α)
- emittance at a designated temperature (ϵ)
- visual check
- electron microscopic pictures

the content of the present document describes the test
procedures for constant temperature load (A).

Referred Document:

"Beständigkeit von Absorberbeschichtungen"; U.Frei, J.Keller:
Interkantonales Technikum Rapperswil; 1983

Principle of Measurement:

Exposition of the absorber coating samples in the
circulating air oven by constant temperature (100°C/200°C).
The exposition continues 4 weeks.
Analysing of the samples befor and after the test.

Apparatus:

Circulating air oven with a maximum tolerance of ± 0.5°C
to the nominal temperature (100°C/200°C).

Calibration:

Sampling:

The samples are either directly removed from an existing
flat plate collector or delivered by the manufacturer but the
substrate must be the same as in the effective use.

Conditioning:

The samples must have a size of 100 * 100 mm. Before and after
the test the samples must be stored by room conditions.

Procedures:

- From each sample the absorptance and the emittance will be measured (AC-O-LA).
- Exposition of the samples in the oven by constant temperature
- Immediatly after the expositon the absorptance and the emittance must be measured.
- Visuall comparisons between the samples and the references respective analysis with the electron microscop give further informations about changes of the absorber coating.

Calculation Method (if necessary, include correction method):

No calculation methods are necessary. Only the differences of the absorptance and the emittance between the exposed and the unexposed samples are determined.

Interpretation/Precision & Accuracy:

Changes of $\Delta\alpha \leq 2,5$ % and
$\Delta\epsilon \leq 5,0$ % give the conclusion, that the absorber coating is stable under temperatur influence as it occurs in flat plate collector.

Accuracy see AC-O-LA, measurement of absorptance and emittance.

Date: 03/04/86   Reporter's Name: Kazuo Taga

Country: Japan

Testing Title:

Testing Method of heat Resistance of Absorber Plate

Abstracted Scope (200 words):

The durability of the absorber plate under the condition of high
temperature is evaluated by this method.
The appearance, solar absorptance and solar emissivity of the absorber
plate is observed.

Referred Document:

Y.Asano et al, 5 7 th Annual Symposium of
    The Metal Finishing Society of Japan(25th Nov. 1976),

    " 54th Symposium Abstruet " 156(1976).

Principle of Measurement:

1. Observation of the appearance.
2. Measuring the solar absorptance and emissivity of the absorber plate after the high temperature condition.

Apparatus:

gravity oven (0 ~ 400 degrees Centigrade)

Calibration:

Sampling:

The specimens are necessary to prepared three pieces above the size of 60mm x 60mm.
The average value is adopted.

Conditioning:

300 degrees Centigrade x 1000hr in the gravity oven.

Procedures:

1. Preparing three pieces of specimens above 60mm x 60mm size.
2. Observing the appearance and measuring the solar absorptance and emissivity of the spcimens before the heat resistance test.
3. Running the heat resistance test under the following condition in the gravity oven.
   300 degrees Centigrade x 1000 hr
4. Observing the appearance and measuring the solar absorptance and emissivity of the specimens after the test.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

REPORTING FORMAT

Date: 86-05-14   **Reporter's Name:** Hans Wennerholm

Country: Sweden

Testing Title: Method for testing thermal stability of absorber and coating for flat plate solar collectors

Abstracted Scope (200 words):

Expose specimens in a controlled-temperature oven set at a temperature determined by adding 30 °C to the stagnation temperature of the actual collector as defined through a thermal performance test procedure. Exposure period: 4 weeks. Evaluation of the test results can be made by measuring the integrated solar absorptance and the total normal emittance prior and after test. In addition, the absorbers after test shall exhibit to the naked eye no crazing, checking, cracking, flaking or delamination

Referred Document:

Canadian Standards Association,
Preliminary Standard F 378-M 1979

Principle of Measurement:

1. Observation of the appearance prior and after test
2. Measurement of absorptance and emittance prior and after test

Apparatus:

Controlled - temperature oven

Calibration:

Sampling:

The dimension of the specimens should be at least 60 x 60 mm^2

Conditioning:

Temperature: add 30 °C to the stagnation temperature

Procedures:

1. Preparation of test·specimens
2. Observation of appearance, measurement of absorptance·and emittance
3. Running the test for 4 weeks
4. Repeat moment 2.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

Date: 9/12/86   Reporter's Name: C. Hauer
Testing Title: Evaluating Solar Absorptive Materials for Thermal
Applications

Abstracted Scope:

ASTM Standard E 744-80 provides a test methodology for absorptive
materials used in flat plate and concentrating collectors (up to a
concentration ratio of 5). It is not a procedure intended for use
with absorbers in front surface contact with a heat transfer fluid
as in trickle collectors, nor does it evaluate the influence of
heat tranfer fluids on absorbers. The test methods included are
property measurements and aging tests.
The test methods in this practice are intended to aid in the
assessment of long term performance by comparative testing of
absorptive materials. The results, however, have not been shown to
correlate with actual service performance.

Referred Document: ASTM E 744-80

The following additional ASTM standards are referenced:
B 537 Recommended Practice for Rating of Electroplated Panels
Subjected to Atmospheric Exposure
E 408 Test Method for Total Normal Emittance of Surfaces Using
Inspection Meter Techniques
E 424 Test Method for Solar Energy Transmittance and Reflectance
(Terrestrial) of Sheet Materials
E 434 Test Method for Calorimetric Determination of Hemispherical
Emittance and the Ratio of Solar Absorptance to Hemisperical
Emittance Using Solar Simulation
E 781 Practice for Evaluating Absorptive Solar Receiver Materials
When Exposed to Conditions Simulating Stagnation in Solar
Collectors with Cover Plates
G 26 Recommended Practice for Operating Light Exposure Apparatus
(Xenon-Arc) With and Without Water for Exposure of Nonmetallic
Materials

Principle of Measurement:

This test methodology is intended to aid in the assessment of long term comparative performance of absorptive materials.Two test methods are provided, namely a series of tests to simulate various exposure conditions in the laboratory, and a single test of outdoor exposure under conditions simulating thermal stagnation.

Apparatus:

The equipment required includes:
* Spectrophotometer, 300-2500 nm
* Pyranometer, Eppley, 180 degree viewing area
* Thermometers, 100 C, in 1 degree divisions
* Xenon Arc Source, per ASTM G 26
* Outdoor collector test module, meeting the requirements of ASTM E 881-82
* Thermal and humidity cycling equipment

Calibration:

ASTM E 744 does not specify calibration methods. However, calibrations specified in the referenced ASTM test procedures should be carried out.

Sampling:

ASTM E 744 itself does not specify a sampling methodology. However, sampling methodologies outlined in the referenced ASTM test methods for the various measurements should be followed.

Conditioning:

No sample conditioning is required

Procedures:

The property test procedures include the following measurements:
* Absorptance, before and after any aging exposure per ASTM E 903
Emittance, before and after any aging tests per ASTM E 434 or E 408
* Appearance noted before and after any aging tests
The aging test procedures include the following:
* Exposure of the absorber surface to solar radiation, with a cover material surface over the sample to test lightfastness.
* Outdoor exposure under simulated stagnation temperature conditions for at least 12 months per ASTM E 781
* Xenon arc exposure at 340 nanometers, 2970 kJ/square meter, for 1500 hours per ASTM G 26
* Exposure to elevated temperature (expected stagnation temperature) for 500 hours
* Effect of moisture, 90 C, 95% relative humidity for 30 days
* Thermal cycling, 30 cycles between stagnation temperature and -10 C, with temperature held for 1 hour at each level

Calculation Methods:

Since this set of test procedures is a comparative evaluation and involves significant visual inspection data, no mathematical analysis is required. Absorptance is an experimental determination made using practice ASTM E 903, and emittance is determined experimentally using ASTM E 434 or E 408.

Interpretation/Precision and Accuracy:

The correlation between these comparative tests and actual performance has not been defined so that no comments about precision can be made

REPORTING FORMAT

Date: December 1986   **Reporter's Name:**   Michael G Hutchins

Country: United Kingdom

Testing Title: Furnace heating in air of solar absorber surfaces

---

Abstracted Scope (200 words):

Samples to be tested are exposed in a pre-heated furnace in air at atmospheric pressure for pre-determined times. A range of temperatures indicative of operating temperature, stagnation temperature and above are chosen. Surface optical properties, chemical composition and microstructures are examined before and after testing.

Referred Document:

ASTM E744 Evaluating Solar Absorptive Materials for Thermal Applications

Principle of Measurement:

Compositional changes, e.g. oxidation, can be accelerated by prolonged exposure of absorber surfaces to elevated temperatures in air. Such changes may result in deleterious effects on surface optical properties which are used as the primary diagnostic variables.

Apparatus:

Electrserv and Gallenkamp muffle furnaces are used (maximum operating temperature $1200^{o}C$).

A vacuum furnace (maximum operating temperature $300^{o}C$) is also available for testing of surfaces in controlled gaseous environments. No use of this furnace has been made to date.

Calibration:
Temperature gradients, often up to $30^{o}C$, are checked together with oven temperatures using calibrated K-type thermocouples

Sampling:

Maximum sample size : 100mm x 100 mm
Number of samples per run less than or equal to 8.

Conditioning:

Furnace is pre-heated to set temperature prior to entry of samples

Procedures:

1. Oven is pre-heated to set temperature
2. Samples are exposed for pre-determined times

   Temperatures of $75^\circ C$, $150^\circ C$, $200^\circ C$, $300^\circ C$ and $400^\circ C$ are most commonly chosen.
   Exposure times of 1h, 10h, 24h, and intermediate times up to 500h are normally selected.

3. Surface optical properties are measured before and after exposure. If significant changes are observed compositional and microstructural examinations are undertaken.

Calculation Method (if necessary, please include correction method):

None

Interpretation/Precision & Accuracy:

Difficult to quantify at this stage. Principal error in procedure is lack of a uniform temperature in the furnace.

REPORTS ON SOLAR ABSORBERS

d (iii) DURABILITY TO HEAT CYCLES

Date : 03/04/86

Reporter's Name : Kazuo Taga

Country : Japan

Testing Title :

Testing Method for Heat Cycles of Absorber Plate

Abstracted Scope (200 words) :

The durability of the absorber plate under the condition of heat cycles is evaluated by this method.
The appearance, solar absorptance and solar emissivity of the absorber plate is observed.

Referred Document :

ASTM E744, " Standard Practice for Evaluating Solar Absorptive Materials for Thermal Applications ".

Principle of Measurement:

1. Observation of the appearance.
2. Measuring the solar absorptance and emissivity of the absorber plate after the heat cycle test.

Apparatus:

Temperature cabinet (-10 degrees Centigrade)

gravity oven (80 degrees Centigrade)

Calibration:

Sampling:

The specimens are necessary to prepare three pieces above the size of 60mm x 60mm.
The average value is adoplet.

Conditioning:

temperatire : -10 degrees Centigrade and 80 degrees Centigrade

Procedures:

1. Preparing three pieces of specimens above 60mm x 60mm size.
2. Observing the appearance and measuring the solar absorptance and emissivity of the specimens before the heat cycles test.
3. Running the heat cycles test under the following condition.

   $-10°c \times 8\ hr \longrightarrow 80°c \times 16\ hr$
   $\longleftarrow$ (40 cycles)

4. Observing the appearance and measure the solar absorptance and emissivity of the specimens after the test.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

REPORTS ON SOLAR ABSORBERS

d (iv) DURABILITY TO THERMAL SHOCK

REPORTING FORMAT   Designation:

                                    AC - D - ST

Date: 1986, 2, 26   Reporter's Name: Takasi   Odaira

Country: Japan

Testing Title:

    Methods of Thermal shock Test for the Selective Coating Absorber

---

Abstracted Scope(200 words):

    Later the test piece is kept on high temperature,
    it is cooled rapidly to chilled water.
    The defect(cracking, peeling, etc.) by the thermal shock
    is observed about selective coating surface.

Referred Document:

    JIS K 3502 Testing Methods of Glass ware for Chemical Analysis

Principle of Measurement:

Test piece, heated in the oven, is cooling rapidly to the chilled water.
If the ability of coating film is bad, the defect(cracking, peeling, etc.)
produces by the thermal shock.

Apparatus:
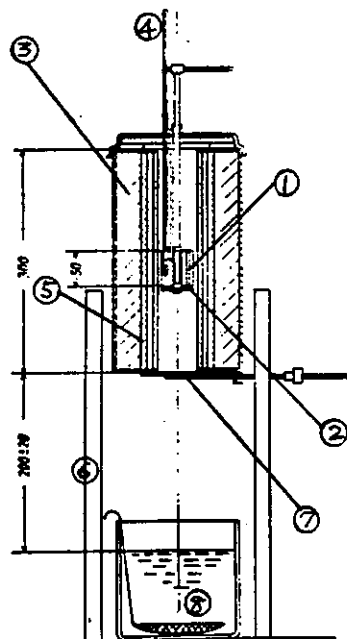
① test piece
② holder
③ electric oven
④ thermomater
⑤ heater
⑥ stand
⑦ under cover
⑧ water tank

Calibration:

Sampling:

40mm × 50mm × 1mm

Conditioning:

temperature(in oven)   125± 1℃
temperature(chilled water)   5℃ ≧

Procedures :

1. Put the test piece in the oven. Let it alone until setting
   temperature.

2. When the test piece becomes setting temperature, it is dropped
   into the chilled water.

3. Put out the test piece.

4. Observe the defect(cracking, peeling, etc)of coating film.

5. Repeat the test, if it is necessary.

Caluclation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy :

  The results of tests is decided on an agreement connected with
each properties.

REPORTS ON SOLAR ABSORBERS

d (v) DURABILITY TO THE EXPOSURE TO SOLAR

RADIATION UNDER OR/AND WITHOUT COVER PLATE

Date: 24.02.86   Reporter's Name: Ulrich FREI
Country: Switzerland
Testing Titel:

Degradation of absorber coatings by exposition
under solar irradiation.

Abstracted Scope (200 words):

The long-therm behavior of a solar collector is
essentially influenced by the durability of the absorber
coating. The following conditions can happen on an absorber
coating in a flat plate collector:

A: temperatures from the minimal ambient temperature
up to the maximum stagnation-temperatur

B: Temperature/moister influence caused by condensation
and or rain penetration. Load range: Ambient condi-
tions up to 100°C/100% rel.humidity

C: Solar irradiation with or partly without uv-radiation,
depending on the cover

For the qualifikation, an absorber coating has to be checked
under controlled conditons (A,B,C). Before and after the
exposition the following analysis should be done:

- solar absorptance (α)
- emittance at a designated temperature (ϵ)
- visual check
- electron microscopic pictures

the content of the present document describes the test proce-
dures for the outdoor exposition under solar irradiation (B).

Referred Document:

"Beständigkeit von Absorberbeschichtungen"; U.Frei, J.Keller;
Interkantonales Technikum Rapperswil; 1983

Principle of Measurement:

Exposition of the absorber coating samples on the outdoor
test-stand.
The exposition period continues 16 weeks.
Analysing of the samples before and after the exposition.

Apparatus:

Outdoor test-stand with mounting device for the samples.
Measurement device for the continuos registration of:

       o ambient temperature
       o relative humidity
       o solar irradiation
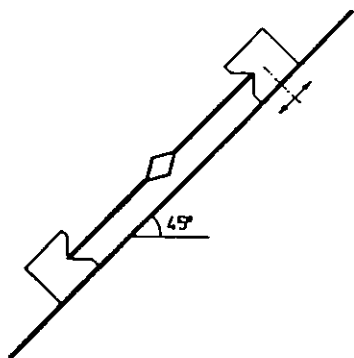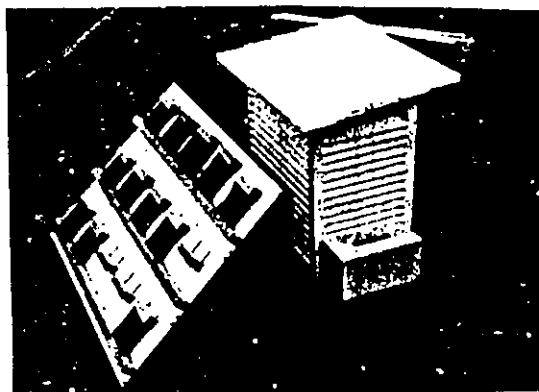
sample rack   test stand

Calibration: -

Sampling:

The samples are either directly removed from an existing
flat plate collector or delivered by the manufacturer but the
substrate must be the same as in the effective use.

Conditioning:

The samples must have a size of 100 * 100 mm. Before and after
the test the samples must be stored by room conditions.

Procedures:

- From each sample the absorptance and the emittance will
  be measured (AC-O-LA).
- Exposition of the samples on the test-stand
- Immediatly after the expositon the absorptance and the
  emittance must be measured.
- Visual comparisons between the samples and the references
  respective analysis with the electron microscop give further
  informations about changes of the absorber coating.

Calculation Method (if necessary, include correction method):

No calculation methods are necessary. Only the differences
of the absorptance and the emittance between the exposed and
the unexposed samples are determined.

Interpretation/Precision & Accuracy:

Changes of $\Delta \alpha \leq 2,5$ % and
$\qquad \Delta \epsilon \leq 5,0$ % give the conclusion, that the
absorber coating is stable under solar irradiation influence
as it occurs in flat plate collectors.

Accuracy see AC-O-LA, measurement of absorptance and
emittance.

REPORTS ON SOLAR ABSORBERS

d (vi) DURABILITY TO MOISTURE AND/OR DEW

REPORTING FORMAT   Designation:

<u>AC/AS - D - ST</u>

<u>Date:</u> 1986.2.26   <u>Reporter's Name:</u>   Takasi   Odaira

<u>Country:</u>   Japan

<u>Testing Title:</u>

      Dump Heat(constant) Testing Method

                              for the Selective Coating Absorber

<u>Abstracted Scope(200 words):</u>

    Put the test piece in the test room that controled a fixed
temperature and moisture.(imagine the glazing)
    The rust or blister,etc,is made on the test piece by that
conditions,whether it is not.

<u>Referred Document:</u>

    JIS K 0236 Testing Methods for Rust priventive Oil

Principle of Measurement:

Observe the defect;peeling,blistering,rust,etc;on the test piece
that put it alone in the test room for a fixet peripd of time.
The test room is maintained an even temperature and moisture humidity.

Apparatus:
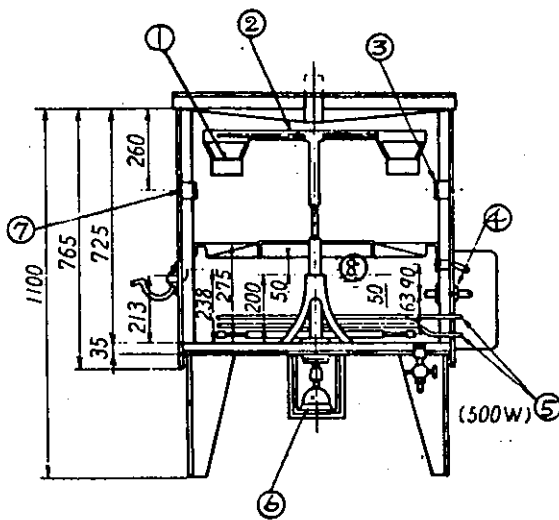
① test piece
② revolving ring
③ thermometer
④ thermostat
⑤ heater
⑥ motor
⑦ thermometer
⑧ water level

Calibration:

Sampling:

150mm×70mm×1mm   or   60mm×80mm×1mm

Conditioning:

room temperature   49± 1℃
humidity   95% ≦
revolution per minute   1/3 R.P.M

Procedures :

1. Hang the test piece from a revolving ring in the test room.

2. The revolving ring is rounding at 1/3 r.p.m.

3. Take out the test piece after a fixed period of time, and wash with water and dry.

4. Both sides(within 3mm)of two diagonals and circumference of test piece within 10mm leave out of the subject of this test.

5. Observe the face of test piece in the direction of revolution.

Caluclation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy :

The results of tests is decided on an agreement connected with each properties.

REPORTING FORMAT

Date: 03/04/86   Reporter's Name: Kazuo Taga

Country: Japan

Testing Title:

Testing Method for Humidity Resistance of Absorber Plate

Abstracted Scope (200 words):

The durability of the absorber plate under the condition of high
temperature and high percentage of humidity is evaluated by this
method.
The appearance, solar absorptance and solar emissivity of the
absorber plate is observed.

Referred Document:

Y.Asamo et al, Abstract of 57th Annual Symposium of Metal Finishing Soc. of
Jaoah(30th May 1978), 156 (1978).

Principle of Measurement:

1. Observation of the appearance.
2. Measuring the solar absoptance and emissivity of the absorber plate after the high temperature and high percentage of humidity condition.

Apparatus:

Humidity Cabinet
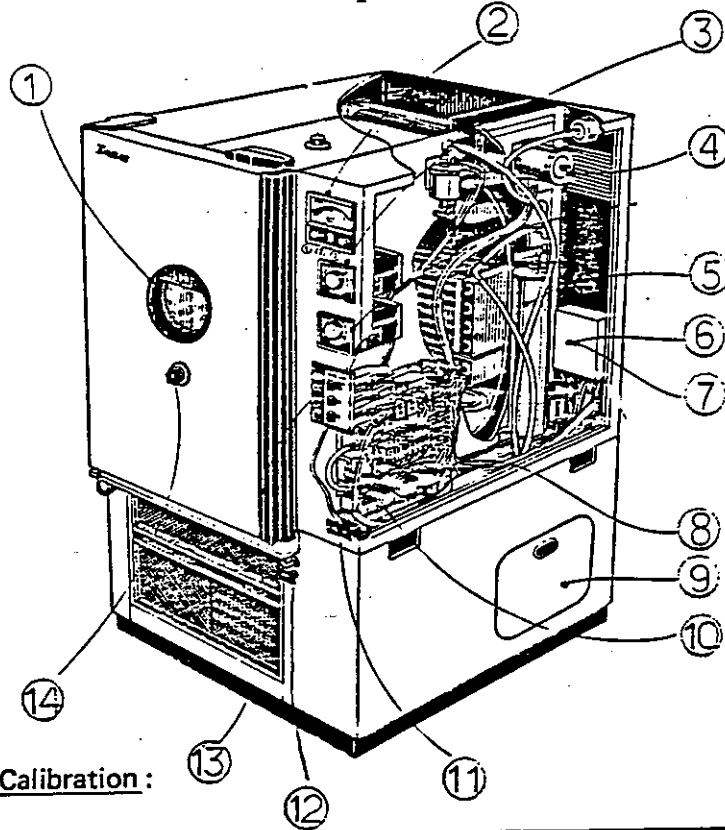
1 observation window
2 temperature indicator
3 temperature controller
4 fan
5 pure water equipment
6 earth leakage breaker
7 alarm connector
8 monitor cord connector
9 inspection door
10 automotive electronic
                    chassis
11 time counter
12 drain
13 operation panel
14 wiper handle

Calibration:

Sampling:

The specimen are necessary to prepare three pieces above the size of 60mm x 60mm. The average value is adopted.

Conditioning:

temperature : 50 degrees Centigrede
relative humidity : 98 %

Procedures:

1. Preparing three pieces of specimens above the size of 60mm x 60mm.
2. Observing the appearance and measuring the solar absorptance and emissivity of the specimens before the humidity test.
3. Running the humidity test under the following condition in the humidity cabinet.
   temperature : 50°c
   relative humidity : 98%) x 1000 hr
4. Observing the appearance and meausuring the solar absoptance and emissivity of the specimens after the test.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

REPORTING FORMAT

Date: 86-05-15   **Reporter's Name:** Hans Wennerholm

Country: Sweden

Testing Title: Method for testing absorbers for flat plate solar collectors at 25 °C and 100 % relative humidity

---

Abstracted Scope (200 words):

This method covers the requirements for testing absorbers at 25 °C and 100 % relative humidity with condensation on the test specimens at all times. Exposure period: 4 weeks. Evaluation of the test can be made by measuring the integrated solar absorptance and the total normal emittance prior and after each test.

Referred Document:

1) ASTM D2247, Standard method for testing coated metal specimens at 100 % relative humidity

2) Wennerholm, H., 10th Scandinavian Corrosion Congress, Stockholm 1986

Principle of Measurement:

1. Observation of the appearance prior and after each test

2. Measurement of absorptance and emittance prior and after each test

Apparatus:
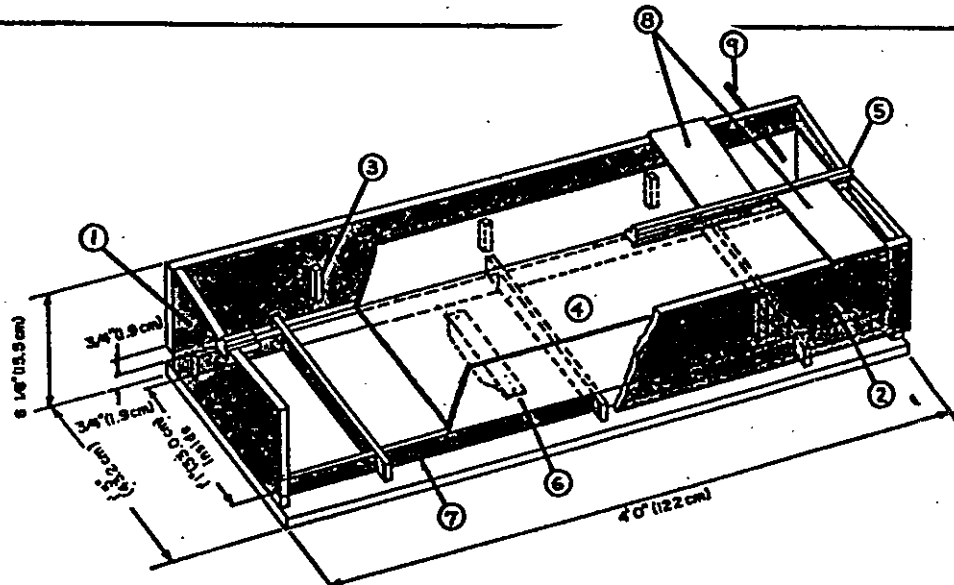
1—End supports, ³₄ hy 6¹₄ in. by 1 ft 3¹₂ in. (1.9 by 15.5 by 39.4 cm) (slanted ¹₄ in./8 in. (0.64 cm/20.3 cm) toward center).
2—Raised side supports, ³₄ by 4⁷₈ in. by 4.0 ft. (1.9 by 12.4 by 122 cm).
3—Tank spacers, ¹₂ by ³₈ by 2¹₂ in. (1.3 by 0.95 by 6.4 cm).
4—Water tank, 14¹₂ by 46¹₂ by 3 in. (36.8 by 118 by 7.6 cm) (distilled water and stainless steel preferred).
5—Depressed central panel support (rigid insulating type, for example, methylmethacrylate resin).
6—Six strip heaters (attached to bottom of tank) spaced equally for uniform heat, rheostat-controlled (115-V, 150-W steel sheath strip heaters and 7½-A capacity rheostat).
7—Tank support and heat barrier, ¾ by 1⅜-in. (1.9 by 3.5-cm) stock.
8—Typical 8-in. (20.3-cm) long test panels or blanks (all positions on cabinet must be occupied) (blanks may be glass).
9—Thermometer (bulb 1 in. (2.5 cm) below test surface).

NOTE 1—Arrangement of 2 and 3 permits access of fresh air.

Calibration:
NOTE 2—A satisfactory construction material is ¾-in. (1.9-cm) exterior grade plywood coated with a polyamide cross-linked epoxy.

FIG. A3. Cleveland Condensing Humidity Cabinet.

Sampling:

Typical dimension is 75 x 203 mm^2
All positions on the Cleveland cabinet must be occupied. Blanks may be glass or aluminium panels. Cut edges and those areas containing identification marks or in contact with the racks or supports, shall be protected with a suitable coating that is stable under the conditions of the test

Conditioning:

Temperature: 25 $^{\circ}$C

Relative humidity: 100 %

Procedures:

1. Preparation of test specimens
2. Observation of appearance, measurement of absorptance and emittance
3. Protection of cut edges and so on (see Sampling)
4. Running the test for 4 weeks
5. Repeat moment 2

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

REPORTS ON SOLAR ABSORBERS

d (vii) DURABILITY TO THE SPECIFIC ATMOSPHERIC CONDITIONS

REPORTING FORMAT   Designation:

                            AC - D - ST

Date: 1986, 2, 12   Reporter's Name: Takasi   Odaira

Country: Japan

Testing Title:

    Testing Method of Weatherability for Selective Coating Films

Abstracted Scope(200 words):

    This test is relative to wetherability for selective coating
surface.
    The coating surface is irradiated with light of carbon arc,
while is sprayed the fogs of water at regular intervals.
    The test valuates weatherability, selectivity, change in
quality of coating films(peeling, fade, stain, change of color,
etc).

Referred Document:

    JIS D 0205 General Rules of Weatherability for Automobile Parts.

    JIS D 0202 General Rules of Coating Films for Automobiles Parts.

2

Principle of Measurement:

The carbon arc(nearly sunlight)is irradiated to coating surface, therefore we can value the weatherability in short time.

Apparatus:
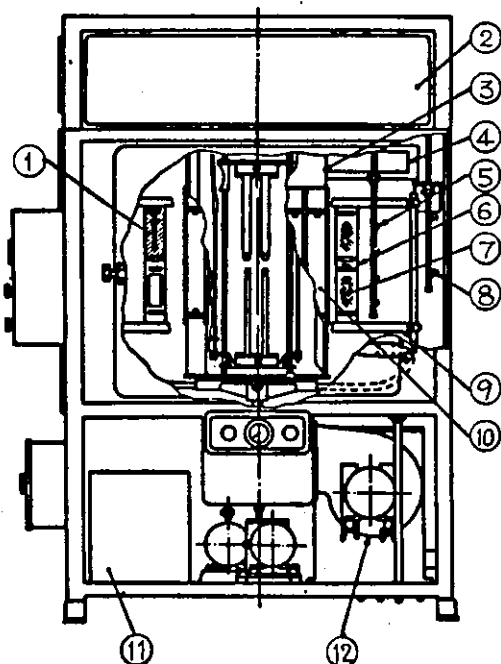
① black panel thermometer
② control box
③ sunshane carbon arc lamp
④ air controler
⑤ spplay for sample
⑥ holder
⑦ sample
⑧ cooling nozzle
⑨ rounding frame
⑩ glass filter
⑪ transformer
⑫ blower

Calibration:

Sampling:

70mm×140mm×0.8mm

Conditioning:

discharge volt   50V±2% ,   discharge ampere   60±2%

transmittance of filter   0% (≦255nm) , ≧90% (400nm)

temp.(black panel)   63±3℃

revolutions per minute   about 1 r.p.m

Procedures :

1. Set up the test piece at the inside of drum in accelarate weatherability test machine.

2. The drum moves around, the light of carbon arc is applied to coating surface, while spply water.

3. Put out the test piece after set times.

4. Compare the test piece with the model.

5. Measure optical properties of selective coating surface.

Caluclation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy :

The results of tests is decided on an agreement connected with each properties.

Date: 1986-05-15   **Reporter's Name:** Hans Wennerholm

Country: Sweden

Testing Title:

Method for testing resistance to sulphur dioxide at high relative humidity of absorber and coating for flat plate solar collectors

Abstracted Scope (200 words):

Expose absorber samples to 0.5 or 1.0 ppm sulphur dioxide gas at 23 ^{o}C and 95 % relative humidity for 6 weeks. Evaluation of test results can be made by measuring the integrated solar absorptance and the total normal emittance prior and after test. In addition, the absorbers after test shall exhibit to the naked eye no cracking, blistering, flaking, pitting, corrosion products or filiform corrosion

Referred Document:

1. Ericsson, R., "Influence of different factors on the atmospheric corrosion of steel", Dep. of Inorganic Chemistry, Chalmers, S-41296 Gothenburg, Sweden (1979)

2. Wennerholm, H., 10th Scandinavian Corrosion Congress, Stockholm 1986

Principle of Measurement:

1. Observation of the appearance prior and after test
2. Measurement of absorptance and emittance prior and after test

Apparatus:
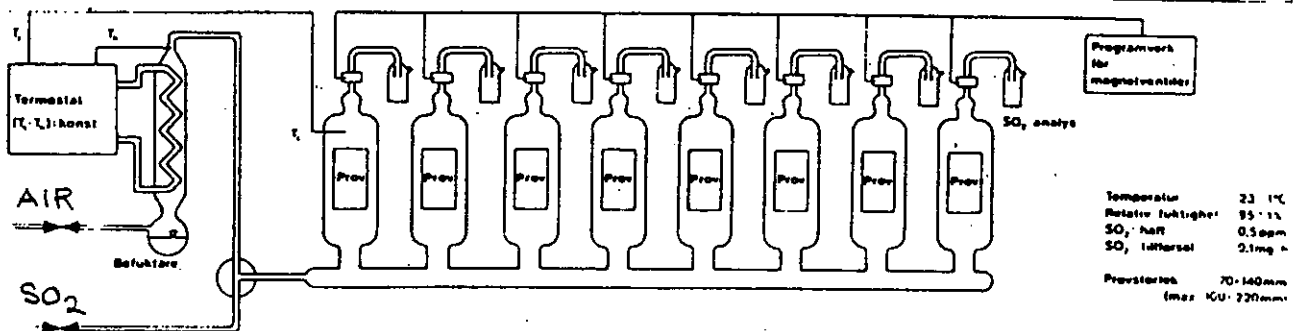
Calibration:

Sampling:

The dimension of the specimens should be 70 x 140 mm^2. Cut edges and those areas containing identification marks shall be protected with a suitable coating that is stable under the conditions of the test

Conditioning:

    Temperature: 23.0 $^{\circ}$C

    Relative humidity: 95 %

    SO_2-concentration: 0.5 or 1.0 ppm

Procedures:

1. Preparation of test specimens
2. Observation of appearance, measurement of absorptance and emittance
3. Protection of cut edges and so on (see Sampling)
4. Running the test for 6 weeks
5. Repeat moment 2

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

27

REPORTS ON SOLAR ABSORBERS

<u>d (viii) DURABILITY TO SALT WATER, ACID OR BASES</u>

Designation :
AC - D - LA

Date : 03/04/86   Reporter's Name : Kazuo Taga

Country : Japan

Testing Title :

Salt Water Spray Testing Method of Absorber Plate

Abstracted Scope (200 words) :

The durability of the absorber plate for salt water spray is evaluated by this method.
The appearance, solar absorptamce and solar emissivity of the absorber plate is observed.

Referred Document :

JIS Z 2371

" Method of Salt Spray Testing "

Principle of Measurement:

1. Observation of the appearance
2. Measuring the solar absorptance and emisivitty of the absorber plate after the salt water spray test.

Apparatus:

Fig.1 Equipment for Testing
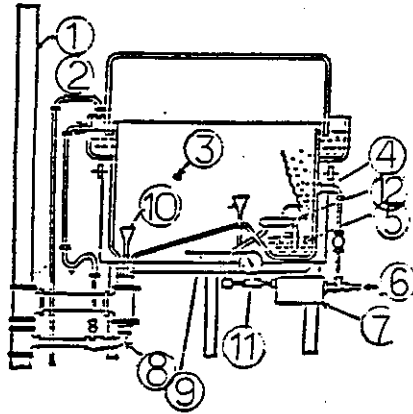
Fig.2 glass nozzle
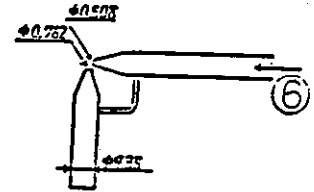
Fig.3 monel metal nozzle
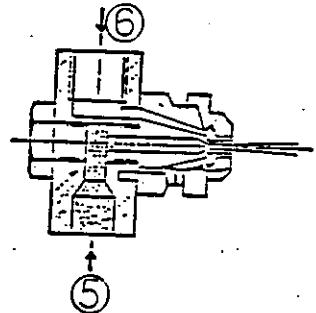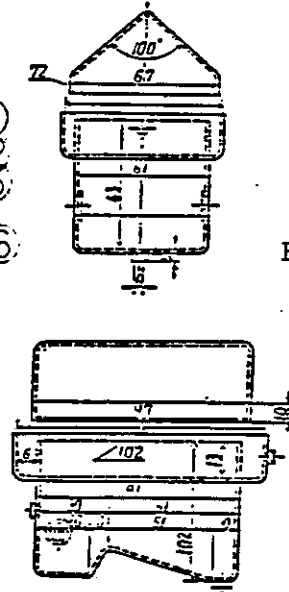
1.exhaust air 2.water 3.thermometer 4.window 5.salt water
6.air 7.pre heater 8.drain 9.heater 10.collecting container
11.pressure gauge 12.nozzle

Calibration:

Sampling:

The specimen are necessary to prepare three pieces above the size of 60mm x 60mm.
The average value is adopted.

Conditioning:

temperature : 35 ± 2 degrees centigrade
quantity of sprays : 0.5 ~ 3.0 ml/hr./80 cm^2
PH : 6.5 ~ 7.2

Procedures:

1. Preparing three pieces of specimens above 60mm x 60mm size.
2. Observing the appearance and measuring the solar absorptance and emissivity of the specimens before the salt water spray test.
3. Running the salt water spray test for 500 hours.
4. Observing the appearance and measuring the solar absorptance and emissivity of the specimens after the test.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

REPORTING FORMAT   Designation:

AC/AS - D - ST

Date: 1986.2.18   Reporter's Name: Takasi Odaira

Country: Japan

Testing Title:

Methods of Salt Splay Test for the Selective Coating Absorber

---

Abstracted Scope(200 words):

Put the test piece in the test room that sprayed salt-water.
The rust or blister is made on the test piece by fogs of
salt-water, whether it is not.

Referred Document:

JIS K5400 Testing Methods for Organic Coating

JIS Z2371 Methods of Salt splay Testing

Principle of Measurement:

Observe the defect;peeling,blistering,rust,etc;on the test piece
that put it alone in the test room for a fixet peripd of time.
The test room is maintained an even temperature and moisture humidity.

Apparatus:

① test room
② test piece
③ supporter
④ nozzle
⑤ filter
⑥ salt-water-tank
⑦ collecting tray
⑧ thermometer
⑨ thermodetector
⑩ hygrometer
⑪ heater
⑫ heater
⑬ exhaust pipe
⑭ drain pipe
⑮ water seal

compressed air

salt-water
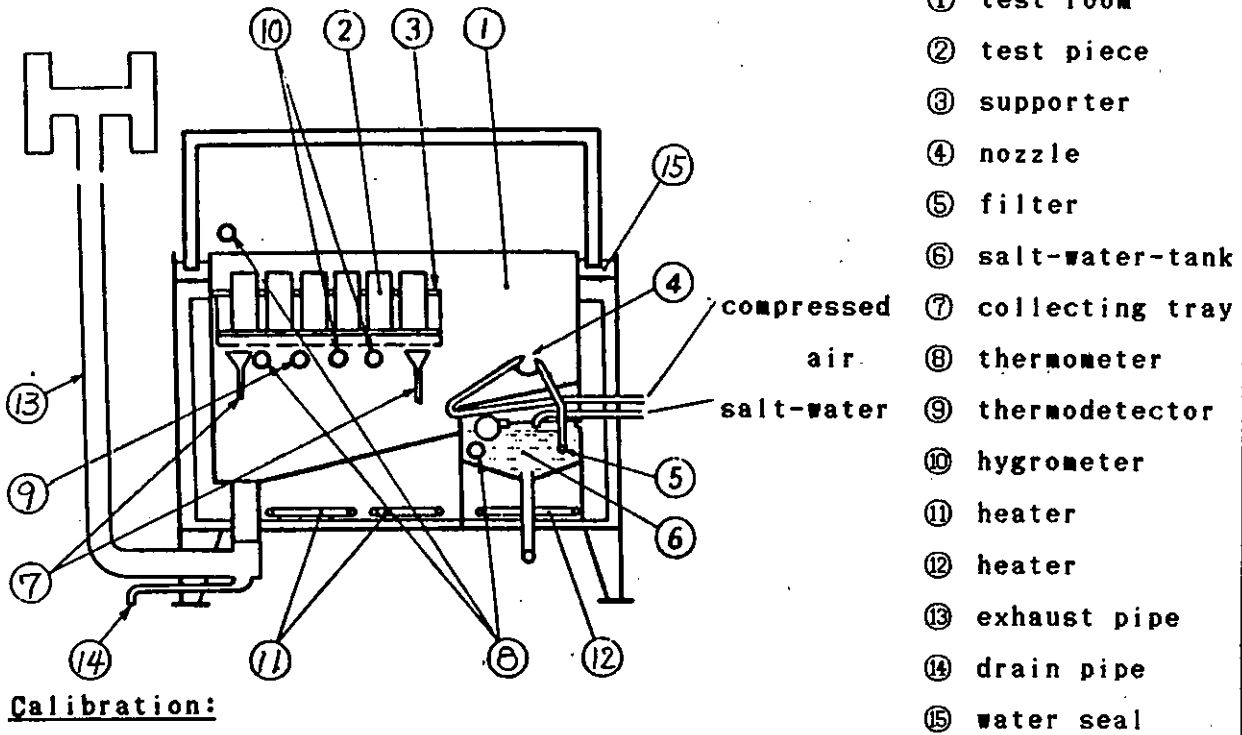
Calibration:

Sampling:

150mm×70mm×1mm   or   60mm×80mm×1mm

Conditioning:

room temperature	35±1℃	pressure of air	1.0±0.025kg /cm²
humidity	95~98%	vol. of collected fog-sol.	1.0~2.0ml/h/80cm²
temp.of humidifier	47±1℃	pH of collected fog-sol.	6.5~7.2(33~35℃)
conc.of salt-water	5w/v%	S.P.collected fog-sol.	1.0263~1.0410(20℃)

Procedures :

1. Draw two diagonals on coating film with the knife, which reach the base metal.

2. Put the test piece in test room, and it is parallel to the flow of fog, at anangle of 60 degrees against the level upwards coating surface.

3. Take out the test piece after a fixed period of time, and wash with water and dry.

4. Both sides(within 3mm)of two diagonals and circumference of test piece within 10mm, leave out of the subject of this test.

Caluclation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy :

The results of tests is decided on an agreement connected with each properties.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Pitting and Crevice Corrosion Resistance of Stainless Steels and Related
Alloys by the Use of Ferric Chloride Solution

Method A: Total Immersion Ferric Chloride Test

Method B: Ferric Chloride Crevice Test

Abstracted Scope (200 words):

These methods cover procedures for the determination of pitting and crevice
corrosion resistance of stainless steels and related alloys when exposed
to oxidizing chloride environments.

Method A is designed to determine the relative pitting resistance of
stainless steels and nickel-base, chromium-bearing alloys, whereas Method
B can be used for determining both the pitting and crevice corrosion
resistance of these alloys.

These tests may be used to determine the effects of alloying additives,
heat treatment, and surface finishes on pitting and crevice corrosion
resistance.

Referred Document:

ASTM G 48   Standard Test Methods for Pitting and Crevice Corrosion
            Resistance of Stainless Steels and Related Alloys by the
            Use of Ferric Chloride Solution

Principle of Measurement:

Weight loss, appearance changes, and pitting and crevice corrrosion resistance are determined for test specimens following their immersion in a ferric chloride solution.

Apparatus:

Method A:
1. 1000-ml, tall-form, glass beakers or wide-neck Erlenmeyer flasks.
2. Glass cradles that will pass through the test container openings.
3. A constant temperature water or oil bath.

Method B:
1. Two cylindrical TFE-fluorocarbon blocks for each test specimen. Each block shall be 12.7 mm in diameter and 12.7 mm high, with perpendicular grooves 1.6 mm wide and 1.6 mm deep cut in the top of each cylinder for the retention of O-rings or rubber bands.
2. Fluorinated elastomer O-rings or rubber bands (low sulfur), two per specimen. Boil in water to remove water soluble ingredients before use.
3. Test tube,, 38 mm in diameter by 300 mm long.
4. Rubber stopper, No. 8, with center hole for insertion of a 6.4-mm diameter, 102-mm long glass tube to serve as a vent and condenser.
5. A constant temperature water or oil bath.

Ferric Chloride Test Solution (for Methods A and B):
Dissolve 100 g of reagent grade ferric chloride, $FeCl_3.6H_2O$, in 900 ml of distilled water. Filter through glass wool or filter paper to remove insoluble particles.

Sampling:

A test specimen 25 by 50 mm is recommended as a standard size, although other sizes and shapes can be tested. Unless end-grain pitting is an integral part of the evaluation, the proportion of end grain surface to specimen surface should be kept small because of the susceptibility of end grains to pitting.

Conditioning:

Remove edge damage caused by cutting or shearing. Polish all surfaces of the specimen to a uniform finish. Wet polishing using 120-grit abrasive paper is recommended. Sharp edges of the specimen should be rounded to avoid cutting the O-ring or rubber band. Clean surfaces with magnesium oxide paste, water rinse, dry in acetone or methyl alcohol and desiccate.

Procedures:

Method A:
1. Pour 600 ml of the ferric chloride test solution into the test
   container, place the container in the constant-temperature bath and
   allow it to come to the equilibrium temperature of interest.
2. Place the specimen in the cradle and immerse in the test solution after
   it has reached the desired temperature. Maintain this temperature
   throughout the test.
3. Cover the container with a watch glass and expose for a reasonable
   test period (for example 72 h).

Method B:
1. Add 150 ml of ferric chloride test solution to each test tube, insert
   a rubber stopper, place the tube in the constant-temperature bath and
   allow to come to equilibrium at the temperature of interest.
2. Fasten two TFE blocks to the test specimen with O-rings or rubber bands
3. After the test solution has reached the desired temperature, place
   the specimen in the tube, replace the stoppeer and return to the bath.
4. Expose for a reasonable period of time such as 72 h.

Both Methods A and B:
1. Following exposure, remove the test specimens, rinse with water, scrub
   with a nylon bristle brush under running water to remove corrosion
   products, dip in actetone or methyl alcohol, and dry.
2. Weigh, and examine visually and with low power microscopy (~20x).
   Characterize pit density and depth, and depth of crevice attack.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

The precision and accuracy of these methods have not been established.

REPORTS ON SOLAR ABSORBERS

d (ix) DURABILITY TO MIXED ENVIRONMENTAL
CONDITIONS SUCH AS ELEVATED TEMPERATURE, SOLAR
RADIATION, MOISTURE AND ETC.

Date: 4/2/86   Reporter's Name: A.J.Faber

Country: The Netherlands

Testing Title: climatic cycle test

Abstracted Scope (200 words):

The climate cycle test is a newly developed test. The absorber plates are
placed in a small climatic chamber, in which temperature and moisture
contents can be controlled; in addition a solar irradiance of 1000 W/m^2
can be created on the absorber plates with a small solar simulator.
One test cycle (duration 4 hours) simulates one day.
The test cycle can be divided in three periods:
- period 1:   temperature 40^0C
              humidity 100%
              duration 1 hour
- period 2:   temperature 80^0C
              humidity < 50%
              solar irradiance 1000 W/m^2
              duration 1 hour

- period 3:   cooling down of the absorber plates from 80^0C to
              ambient temperature;
              duration 2 hours.
The total duration of the test is 120 cycles; this is not a fixed number.

Referred Document:

    "DEVELOPMENT OF TESTPROCEDURES
    FOR RELIABILITY AND DURABILITY
    OF SOLAR COLLECTORS:
    ACCELERATED AGING/CORROSION TESTS
    ON ABSORBER PLATE MATERIALS AND   Ing. R.J.A. Blankers - Metaalinstituut
    COATINGS"   Ir. F. Simonis   - TPD-TNO
    - final report - March 1, 1984   Ir. A.J.T.M. Wijsman - TPD-TNO

Principle of Measurement:

One test cycle (duration 4 hours) simulates one day.

Apparatus:

1. Climatic chamber; 300 1, Köhler
2. Solar simulator, max. 1000 W/m², 25 lamps (Philux 40 W/220 V), lamps are air cooled
3. By water heated plate
4. Test sample
5. Automatically served water drain
6. Automatically served door (open/closed)
7. Thermocouple
8. Controlled heating element (100 W), 40°C
9. Water dosing pump
10. Centrifugal water pump (7,5 l/min)
11. Thermostat bath - 9 l
12. Controlled heating element (3000 W), 80°C
13. Controlled cooling element
14. Mixer

Calibration:

Sampling:

The dimensions of the absorber samples are 300 x 200 mm.

During the test the temperature of one test sample (4) is measured and recorded with a Chromel-Alumel thermocouple (7) and a Kipp recorder.

Conditioning: For the testing of absorber materials/coatings the following climatic test cycle is ran through:

1 hour, 40°C, 100% relative humidity (RH)

1 hour, 80°C, < 50% RH, solar irradiance 1000 W/m²

2 hours of cooling down from 80°C to ambient temperature.

The duration of one cycle is 4 hours.

Procedures:

All actions, to run through a test circle are accomplished automatically by a double watch and two roller switches.
One test cycle has the following automatic actions:

a. Start of the test:
   - the door of the chamber (6) is closed
   - the water drain (5) closes
   - the water dosing pump (9) brings 2 1 demi-water in the chamber
   - the heating element (8) is switched on.

   The humidity in the air of the chamber increases because of the heating of water at the bottom of the chamber. Upon colder parts in the chamber, such as the test samples, water is condensing.

b. After one hour phase 2 starts:
   - the heating element (8) at the bottom is switched off
   - the door of the chamber (6) is opened
   - the water drain (5) opens
   - the heating element (12), the pump (10), the mixer (14) and the solar simulator are switched on.

c. After an other one hour starts the cooling down period (2 hours)
   - the solar simulator is switched off
   - the heating element (12) is switched off, the cooling element is switched on
   - after ± 1.5 hour the pump (10) stops.

   The test cycle is ran through and a new test cycle starts.

Interpretation

The changes in the absorber materials/coatings caused by a test are inspected in two ways:
1. visually
2. by measurement of the optical properties of the material before and after the test.

The optical properties measured are the solar absorptance α and thermal emittance ε.

<u>Date:</u> 24.02.86   <u>Reporter's Name:</u> Ulrich FREI
<u>Country:</u> Switzerland
<u>Testing Titel:</u>

      Degradation of absorber coatings by <u>exposition</u>
      <u>under constant temperature and high humidity.</u>

<u>Abstracted Scope (200 words):</u>

The long-therm behavior of a solar collector is
essentially influenced by the durability of the absorber
coating. The following conditions can happen on an absorber
coating in a flat plate collector:

    A: temperatures from the minimal ambient temperature
       up to the maximum stagnation-temperature

    B: Temperature-humidity influence caused by condensation
       and or rain penetration. Load range: Ambient condi-
       tions up to 100°C/100% rel.humidity

    C: Solar irradiation with or partly without uv-radiation,
       depending on the cover

For the qualification, an absorber coating has to be checked
under controlled conditons (A,B,C). Before and after the
exposition the following analysis should be done:

    - solar absorptance (α)
    - emittance at a designated temperature (ϵ)
    - visual check
    - electron microscopic pictures

the content of the present document describes the test
procedures for constant temperature-humidity load (B).

<u>Referred Document:</u>

"Beständigkeit von Absorberbeschichtungen"; U.Frei, J.Keller;
Interkantonales Technikum Rapperswil; 1983

Principle of Measurement:

Exposition of the absorber coating samples in the climat-
chamber (50°C/100% rel.hum.; 95°C/100% rel.hum.)
The exposition period continues 4 weeks.
Analysing of the samples before and after the exposition

Apparatus:

Climat-chamber with a minimum tolerance of ±0.5°C and
±3% rel.hum.

Calibration: -

Sampling:

The samples are either directly removed from an existing
flat plate collector or delivered by the manufacturer but the
substrate must be the same as in the effective use.

Conditioning:

The samples must have a size of 100 * 100 mm. Before and after
the test the samples must be stored by room conditions.

Procedures:

- From each sample the absorptance and the emittance will be measured (AC-O-LA).
- Exposition of the samples in the climat-chamber by constant temperature and humidity.
- Immediatly after the expositon the absorptance and the emittance must be measured.
- Visual comparisons between the samples and the references respective analysis with the electron microscop give further informations about changes of the absorber coating.

Calculation Method (if necessary, include correction method):

No calculation methods are necessary. Only the differences of the absorptance and the emittance between the exposed and the unexposed samples are determined.

Interpretation/Precision & Accuracy:

Changes of $\Delta\alpha \leq 2,5$ % and
$\Delta\epsilon \leq 5,0$ % give the conclusion, that the absorber coating is stable under temperature and high humidity as it occurs in flat plate collectors.

Accuracy see AC-O-LA and AC-T-LA: measurement of absorptance and emittance.

REPORTING FORMAT

Date: 86-05-15   **Reporter's Name**:   Hans Wennerholm

Country: Sweden

Testing Title: Method for testing absorbers for flat plate solar collectors
at 90 °C and 95 % relative humidity

Abstracted Scope (200 words):

This method covers the requirements for testing absorbers at 90 °C and
95 % relative humidity. Exposure period: 10 weeks. Evaluation of the
test results can be made by measuring the integrated solar absorptance
and the total normal emittance prior and after each test

Referred Document:

1) Wennerholm, H., 10th Scandinavian Corrosion Congress, Stockholm 1986

Principle of Measurement:

1. Observation of the appearance prior and after test

2. Measurement of absorptance and emittance prior and after test

Apparatus:

The humidity chamber shall be so constructed that:

1. The humidity and the temperature of the chamber are monitored by sensing devices located in the working space
2. The temperature and relative humidity in the working space can be maintained at 90 ±2 °C and 95 ±3 % respectively
3. Condensed water is continously drained from the chamber and not used again until it has been repurified
4. When the humidity conditions are obtained by using an injection-type chamber, the water shall have a resistivity not less than 500 Ωm
5. No condensed water from the walls and roof of the test chamber can fall on the specimens

Calibration:

Sampling:

The dimension of the specimens should be at least 60 x 60 mm^2. Cut edges and those areas containing identification marks, shall be protected with a suitable coating that is stable under the conditions of the test

Conditioning:

Temperature: 90 ±2 °C

Relative humidity: 95 ±3 %

Procedures:

1. Preparation of test specimens
2. Observation of appearance, measurement of absorptance and emittance
3. Protection of cut edges and so on (see Sampling)
4. Running the test for 10 weeks
5. Repeat moment 2

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

REPORTING FORMAT

Date: 1986-05-15   **Reporter's Name:** Hans Wennerholm

Country: Sweden

Testing Title:

Method of testing resistance to UV-radiation and moisture of absorbers
for flat plate solar collectors

Abstracted Scope (200 words):

The absorber samples were exposed for 4 weeks in UV-CON apparatus with
fluorescent UV-lamps as per ASTM G53. The following test cycles were
used:

1. 4 h UV-light, temperature 60 $^{\circ}$C on a black panel

2. 4 h condensation, air temperature 40 $^{\circ}$C

Evaluation of the test results can be made by measuring the integrated
solar absorptance and the total normal emittance prior and after test

---

Referred Document:

1. ASTM G53, Operating light- and waterexposure apparatus (fluorescent
    UV-condensation type) for exposure of nonmetallic materials
2. Wennerholm, Hans., 10th Scandinavian Corrosion Congress, Stockholm 1986

Principle of Measurement:

1. Observation of appearance prior and after test
2. Measurement of absorptance and emittance prior and after test
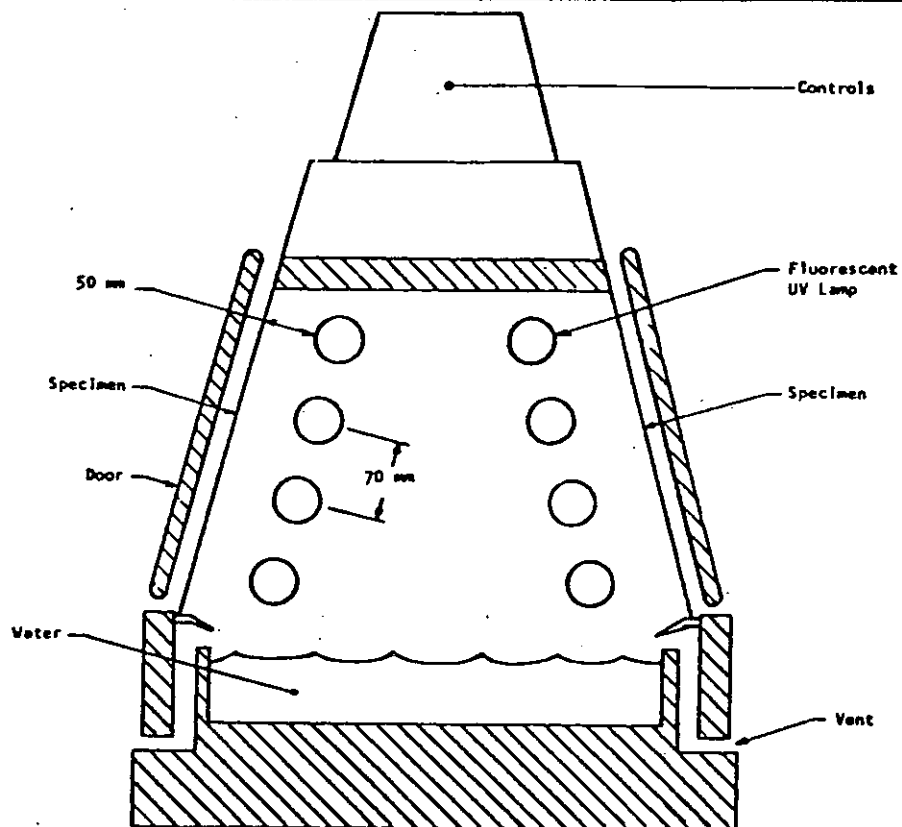
Apparatus:

FIG. 2 Apparatus, Schematic Cross Section.

Calibration:

Sampling:

The dimension of the specimens could be e.g. 75 x 230 mm^2. Cut edges and those areas containing identification marks shall be protected with a suitable coating that is stable under the conditions of the test

Conditioning:

Test cycle:

1. 4 h UV-light, temperature 60 $^{\circ}$C on a black panel
2. 4 h condensation, air temperature 40 $^{\circ}$C

Procedures:

1. Preparation of test specimens
2. Observation of appearance, measurement of absorptance and emittance
3. Protection of cut edges and so on (see Sampling)
4. Running the test for 4 weeks.
5. Repeat moment 2

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

Date: December 1986 **Reporter's Name:** Michael G Hutchins

Country: United Kingdom

Testing Title: Durability testing of solar absorbers by exposure
to controlled conditions of temperature and moisture.

Abstracted Scope (200 words):

A Fisons 300-H climatic cabinet has been installed which enables samples to
be held at or cycled through controlled conditions of both temperature and
relative humidity. The temperature range of the cabinet is $-20^{\circ}C - 180^{\circ}C$.
The active testing volume is 700mm x 700mm x 700mm. The cabinet is
controlled by a BBC Model B microcomputer, in which a number of different
test cycles are stored.

Surfaces are suspended in the cabinet from purpose built jigs. Comparisons
are made between surface optical properties, composition and microstructure
before abd after temperature-humidity testing.

We should like to report that at this stage we are not sure of the precise
cycles to be followed during testing. We have investigated several standards
or derivatives of them (these are listed below). One of the principal
difficulties is the length of time that some of these tests require. We
should like a test that occupies the cabinet for about one week only per test.

Referred Document:

ASTM E744
NBS TN 1196
ASTM D2247
MIL STD 810-D
Wennerholm H, Accelerated testing of absorbers for flat plate solar collectors,
                10th Scandinavian Corrosion Congress, Stockholm, 1986.

Principle of Measurement:

Test samples are exposed to the combined effects of temperature and moisture for pre-determined periods of time. Tests may be carried out under one set of conditions only or the conditions may be varied in a cyclic manner.

Apparatus:

Fisons 300-H IND climatic cabinet with chart recorder for temperature and humidity measurements, BBC Model B microcomputer for control of test cycles.

Calibration: Standard calibration performed annually to check temperature gradients and humidity levels in the chamber.

Sampling:

Standard sample size 100mm x 100mm
Overall cabinet working chamber dimensions 700mm x 700mm x700mm

Conditioning:

As per cycle, no pre-test conditioning

Procedures:

Procedures vary depending on the precise test chosen.

Calculation Method (if necessary, please include correction method):

None

Interpretation/Precision & Accuracy:

Difficult to quantify at this stage.

Date: December 1986   Reporter's Name: Michael G Hutchins

Country: United Kingdom

Testing Title: Exposure to ultraviolet radiation and moisture

Abstracted Scope (200 words):

The Fisons 300-H climatic cabinet is fitted with an ultraviolet lamp.
This enables a range of tests combining exposure of test samples to
ultraviolet radiation with or without humidity or temperature conditions
superposed. Samples are exposed to ultraviolet radiation with or without
the combined effects of temperature or moisture for pre-determined periods.
Surafec optical properties, composition and microstructure may be examined
before and after testing.

Referred Document:

ASTM G53 Operating Light and Water Exposure Apparatus for Exposure of
Non-metallic Materials

25

Principle of Measurement:

Samples are exposed to measured quantities of ultraviolet radiation within a climatic cabinet. The test environment may be altered to allow different conditions of temperature and/or humidity to be maintained throughout the exposure period.

Apparatus:

Fisons 300-H climatic cabinet equipped with ultraviolet irradiation source.

Calibration:

The irradiance from the uv source is calibrated using solid state detectors to determine the uniformity of beam distribution across exposed samples.

Sampling:

Sample size is normally 100mm x 100mm

Conditioning:

255

Procedures:

1. Surface optical properties of test samples are measured.
2. The climatic cabinet is set to the required conditions of temperature and moisture.
3. Surfaces are placed in the cabinet and exposed to ultraviolet radiation of known intensity for set periods of time.
4. The conditions of temperature and humidity within the cabinet may be changed if necessary and the uv lamp may be extinguished on a subsequent part of the testing procedure.
5. The samples are removed from the cabinet and surface optical properties remeasured. If significant differences are observed compositional and microstructural examinations are performed.

Calculation Method (if necessary, please include correction method):

Total ultraviolet irradiance is computed.

Interpretation/Precision & Accuracy:

Difficult to quantify at present

REPORTS ON GLAZING

a. OPTICAL PROPERTIES

a (i) SOLAR ABSORPTANCE

REPORTING FORMAT

Designation:
G-O-LA

Date: 1986-03-16   Reporter's Name:

Country: Sweden

Testing title:

Solar absorptance of glazing

Abstracted Scope (200 words):

Transmittance τ and reflectance ρ is measured spectrally.
Absorptance is calculated:

$\alpha = 1-\tau-\rho$

See IIaii and Iai.

Referred Document:

bs/1733L

REPORTING FORMAT   Designation:
                          G-O-LA

Date: 1986-03-16   Reporter's Name:

Country: Sweden

Testing title:

Measurement of solar transmittance
Normal/diffuse, spectral

Abstracted Scope (200 words):

To determine the transmittance of solar collector glazing by
measurement of 0/d transmittance in the wavelength region
380-2500 nm and weighting the reflectance values with AM2 solar
spectral irradiance.

Referred Document:

Principle of Measurement:

Spectral with integrating sphere on monochromatic side of monochromator. Normal irradiation of the sample and diffuse detection. Single beam.

Apparatus:

Monochromator: Zeiss MM12 double prisms
Integrating sphere
Halogen lamp source
Detectors: 380–800 nm wavelength: photomultiplier
            800–2500 nm wavelength: PbS
Amplifier: Keithley 427
Voltmeter: HP 3455 A
Computer: HP 216

Calibration:

Wavelength scale with spectral lamps.

Sampling:

Samples must be flat and reasonably uniform and larger than 3×3 cm^2.

Conditioning:

Procedures:

Measure detector output each 10 nm in wavelength region 380-800 nm and each 50 nm in the 800-2500 nm. This with (dx) and without (do) the sample at the sphere sample port.

Correct for the reflection of the sphere radiation output at the sample port back into the sphere. This at each wavelength.

Calculation method:

$\tau = k \cdot dx/do$

where k is the sphere correction factor (measured at each wavelength for each sample).

Interpretation:

Precision: <±0.005   95 % confidence level

Accuracy: <±0.01   for good samples

REPORTING FORMAT

Designation:

$A/G - 0 - ST$

Date: 9/3/86   Reporter's Name: C. Hauer
Country: USA
Testing Title: Solar Absorptance, Reflectance, and Transmittance
of Materials using Integrating Spheres

Abstracted Scope

This test method covers the measurement of spectral absorptance, reflectance and transmittance of materials using spectrophotometers equipped with integrating spheres. The methods of computing solar-weighted properties from the measured spectral values are specified. The method is applicable to materials having both specular and diffuse optical properties.

Referred Document: ASTM E 903

The following ASTM documents are referenced:
E 424 Test Method for Solar Energy Transmittance and Reflectance (Terrestrial) of Sheet Materials
E 490 Standard for Solar Constant and Air Mass Zero Solar Spectral Irradiance Tables
E 772 Definition of Terms Relating to Solar Energy Conversion
E 891 Standard for Terrestrial Direct Normal Solar Spectral Irradiance Tables for Airmass 1.5

262

Principle of Measurement:

Spectrophotometers equipped with integrating spheres are used to make hemispherical transmittance or reflectance measurements over the spectral range of approximately 300 to 2500 nm. The solar transmittance, reflectance or absorptance is obtained for a sample by calculating a weighted average with a standard solar spectral irradiance as the weighting function.

Apparatus:

The primary instrument required is a spectrophotometer capable of measuring in the spectral range of 300 to 2500 nm. A single beam instrument which corrects for background or a double beam, ratio recording instrument is needed. This instrument must be equipped with an integrating sphere attachment. In addition, it is recommended that reference standards having high specular reflectance, high diffuse reflectance, and low diffuse reflectance be available. These standards are available from the National Bureau of Standards as Standard Reference Materials (SRM). In addition, working standards for diffuse reflectance specimens (like barium sulfate or Halon, available from Allied Chemical Co.) and specular reflectance specimens (front surface mirrors) are required.

Calibration:

Calibration of the spectrophotometers should follow the manufacturer's procedures. Working standards should be calibrated against the NBS Standard Reference Materials.

Sampling:

While ASTM E 903 does not specify sampling procedures, three to five samples of the cover material should be used.

Conditioning:

Plastic Solar Collector cover materials should be conditioned in accordance with ASTM D 618.

Procedures:

Calibrate the linearity and wavelength scales of the spectrophotometer as recommended by the manufacturer. It is also necessary to correct for the 100% and zero line errors. For spheres with two ports, record the 100% line curves using identical high reflectance specimens in both ports. For reflectance measurements, record the zero line with a perfect absorber in the sample port. For transmittance measurements, record the zero line with the sample beam blocked. For reflectance of transparent or translucent materials having transmittance greater than .001, back the specimen with a black material having a low reflectance (<0.02) over the 300 to 2500 nm spectral range. Record the spectral reflectance of the specimen relative to the working standard by placing each in the respective port of the sphere.

Calculation Methods:
Compute the spectral reflectance using:

$$\rho = (S - Z)/(100 - Z)(\rho') \text{ where}$$

S = recorded specimen reading
Z = the zero line reading
100 = the 100% line reading
ρ' = the calibrated spectral reflectance of the standard

For transmittance measurements, cover the rear ports of the sphere with the same material as the sphere. Record spectral curves without specimen in place then with specimen in place. Calculate transmittance as:

$$t = (S - Z)/(100 - Z).$$

For absorptance of non-opaque samples, calculate the absorptance using the Kirchoff relation, $\alpha + t + \rho = 1$, based upon the measured reflectance and transmittance.

For the calculation of the solar spectral irradiance distribution, use ASTM standard E 891, calculating the optical properties using the 50 point selected ordinate method.

Interpretation/Precision and Accuracy:

Uncertainties in the results from this test method arise from random errors in measurement and sytem errors resulting from the geometry of the sphere, errors in standards values, and scattered light. The total uncertainty in the values of solar weighted optical properties is dominated by uncertainties in the spectral properties. The rms of these errors results in about a +-2% expected uncertainty.

Date: December 1986   Reporter's Name: Michael G. Hutchins

Country: United Kingdom

Testing Title: DETERMINATION OF THE SOLAR ABSORPTANCE/REFLECTANCE/
TRANSMITTANCE FROM SPECTRAL MEASUREMENTS AT NEAR-
NORMAL INCIDENCE

Abstracted Scope (200 words):

Spectral reflectance and/or spectral transmittance measurements are made using a Beckman 5240 integrating sphere spectrophotometer in the spectral range 0.3 - 2.5 um. In reflectance mode radiation is incident on the surface at 5° to the surface normal. In transmittance mode the radiation is incident normally on a surface placed over a sphere entry port.

Solar optical properties are calculated by convoluting the spectral reflectance curve with a standard solar spectral distribution curve for air mass 2 using a method of 20 selected ordinates. One of two solar spectral distributions may be used (1,2).

The measurement procedure is automated using an Apple IIe microcomputer.

Referred Document:

1. Wiebelt J.A. and Henderson J.B., J. Heat Transfer 101, 101, 1979.

2. Moon P., J. Franklin Inst., 230, 583, 1940.

265

Principle of Measurement:

Near-normal hemispherical spectral reflectance or transmittance of incident radiation.

Apparatus:

Beckman 5240 uv/vis/nir integrating sphere spectrophotometer using photomultiplier for uv/vis detection and PbS for nir detection.
6" diameter integrating sphere coated with barium sulphate white reflectance standard. Double beam ration recording principle of operation.
Apple IIe microcomputer for calculation of optical properties.

Calibration:
For diffuse surfaces : barium sulphate
For highly specular surfaces : aluminium mirror

Sampling:

Minimum sample size : 30mm x 30mm
Maximum sample size : 100mm x 100mm

Conditioning:

None

Procedures:

1. Sample cut to size and cleaned by air blowing.
2. Instrument is calibrated using two barium sulphate reference plates.
3. Sample mounted on sphere wall (rear for reflectance : front for transmittance) and reflectance or transmittance measured for scan from 2500nm - 350nm.
4. Spectral data at 1nm intervals are sent to an Apple IIe microcomputer.
5. Microcomputer executes calculation to determine surface optical properties, e.g. α_s, τ_s or ρ_s.

Calculation Method (if necessary, please include correction method):

20 selected ordinates for air mass 2 solar spectral distribution giving

$$\alpha_s = \frac{1}{20} \sum_{i=1}^{20} \alpha_i \, ,$$ where α_i is the absorptance at each of the twenty

selected ordinates and is found from $\alpha_i = 1 - \rho_i$.

$$\tau_s = \frac{1}{20} \sum_{i=1}^{20} \tau_i \, ,$$ where τ_i is the transmittance at each of the twenty selected ordinates.

Interpretation/Precision & Accuracy:

Precision : reproducible to one part in 10^3.
Accuracy : 1-2%.

REPORTS ON GLAZING

a. OPTICAL PROPERTIES

a (ii) SOLAR TRANSMITTANCE

Designation:

REPORTING FORMAT

GS - O- ST

<u>Date</u>: 15 Jan. '86   <u>Reporter's Name</u>: K.MATSUOKA

<u>Country</u>: Japan

<u>Testing Title</u>:

  Testing Method on Solar Transmittance of Glazings

<u>Abstracted Scope (200 words)</u>:

  Testing method of the solar transmittance of such glazings as plate glass[1],
  processed plate glass [2], and layered glass used as building materials
  is described . The optical method is given.

  Note:
   1) normal float sheet glass, glass with low iron contents, glass with
       surface coatings, and glass absorbing thermal radiation are objects
       of this standard.

   2) doubled glass is included in this category.

  Firstly, spectral transmittane (ST) of the sample is measured by using of
  the scanning spetrophotometer with an integrating sphere to take accout of
  the contribution due to the diffusely transmitted rays. Then solar trans-
  mittance is calculated from the tabulated solar irradiance multiplied by
  the obtained ST values.

<u>Referred Document</u>:

1)Japanese Industrial Standard (JIS) R 3106 " Testing Method of Solar
   Transmittance og Glazings ", revised version in 1985.
2) Indian Journal of Pure & Applied Physics, Vol. 15, 104(1977).

<u>Principle of Measurement:</u> Double beamed optical null method .

The notations are defined as followings respectively: Vst, spectral signal with the sample, Vat, spectral signal without the sample, and Vot, back ground spectral signal under the inserting of the optically opaque material in the optical path related with the sample beam. Then spectral transmittance is derived from the equation $\tau(\lambda) = [\,Vst - Vot\,]/[\,Vat - Vot\,]$ where spectral transmittance being defined as $\tau(\lambda)$. An integrating sphere is used to take accout of the diffusely transmitted beam.

<u>Apparatus:</u>
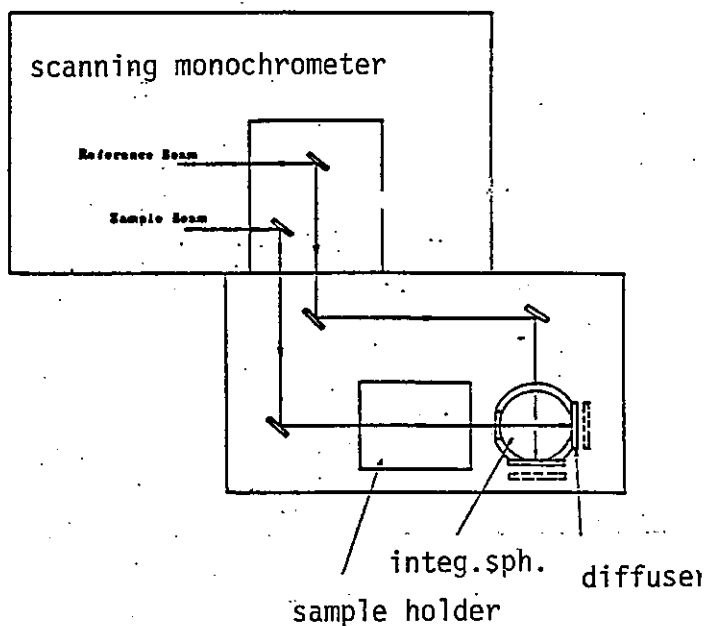
Fig.1. Optical Scheme

<u>Calibration:</u>

<u>Sampling:</u>

    size: 50(mm) x 50(mm)

<u>Conditioning:</u>
(1) Confirmation of the optical flatness of the sample surface.

Procedures:

(1) Measurementsof Vat and Vot.

(2) Measurement of Vst from 340 to 780 nm
in wavelength with the increment of
10 nm in wavelength step.

(3) Measurement of Vst from 800 to 1800 nm
in wavelength with the increment of
50 nm in wavelength step.

(4) Calculation of ST(τ (λ)) by the equation
described in the "Principle" in this
report.

(5) Calculation of the solar transmittance
of the sample by the equation described
in the following box.

Tabulation of the solar spectral irradiance required to the calculation of solar transmittance.

λ (nm)	$E\lambda \cdot \Delta\lambda$	λ (nm)	$E\lambda \cdot \Delta\lambda$
340	0.00291	670	0.01459
350	0.00346	680	0.01447
360	0.00395	690	0.01436
370	0.00474	700	0.01418
380	0.00494	710	0.01403
390	0.00527	720	0.01085
400	0.00739	730	0.01165
410	0.00967	740	0.01309
420	0.01023	750	0.01327
430	0.01012	760	0.01314
440	0.01171	770	0.01292
450	0.01354	780	0.01271
460	0.01447	800	0.04704
470	0.01478	850	0.05583
480	0.01546	900	0.04255
490	0.01497	950	0.02365
500	0.01525	1000	0.04014
510	0.01508	1050	0.03801
520	0.01496	1100	0.02933
530	0.01521	1150	0.01202
540	0.01534	1200	0.02358
550	0.01543	1250	0.02761
560	0.01543	1300	0.01929
570	0.01551	1350	0.00978
580	0.01566	1400	0.00192
590	0.01567	1450	0.04522
600	0.01541	1500	0.01077
610	0.01525	1550	0.01453
620	0.01516	1600	0.01328
630	0.01516	1650	0.01189
640	0.01506	1700	0.01070
650	0.01402	1750	0.00945
660	0.01481	1800	0.00609

■ 注 λ = 340~780nm で $\Delta\lambda$ = 10nm
λ = 800nm で $\Delta\lambda$ = 40nm (785~825nm)
λ = 850~1800nm で $\Delta\lambda$ = 50nm

Calculation Method (if necessary, please include correction method):

$$\tau_e = [\ \Sigma \ (E_\lambda \ \Delta\lambda) \ \tau(\lambda) \ ]/ \ \Sigma \ (E_\lambda \Delta\lambda)$$

where, $E_\lambda \Delta\lambda$ means the solar spectral irradiance tabulated above, and τ_e being solar transmittance.

Interpretation/Precision & Accuracy:

Presision : $\pm$ 0.3 %

REPORTING FORMAT

Designation:

G-O-LA

<u>Date</u>: February 19, 1986   <u>Reporter's Name</u>: J.Keller, EIR, CH-5303 Würenlingen

<u>Country</u>: Switzerland

<u>Testing Title</u>: Spectral transmittance (normal) of glazings

---

<u>Abstracted Scope (200 words)</u>:

Measurement of the spectral transmittance of collector glazings in the range
of wavelengths from 0.3 µm to 2.5 µm using a Zeiss PMQII/RA3 spectrometer.
The irradiation is normal. An average value of the transmittance over the
above range of wave length is obtained by weighing the output by an
appropriate solar spectrum.

<u>Referred Document</u>:

- Instruction Manual. Zeiss PMQII
- J. Keller, V. Kyburz, Th. Fisch, "Wärmeerträge und Kenngrössen von Sonnen-
  kollektoren. Bericht über die Prüfkampagne vom Sommer 1983". Eidg.
  Institut für Reaktorforschung (EIR), CH-5303 Würenlingen. Mai 1984

Principle of Measurement:

See "procedures"

Apparatus: Zeiss PMQII/RA3 Spectrometer
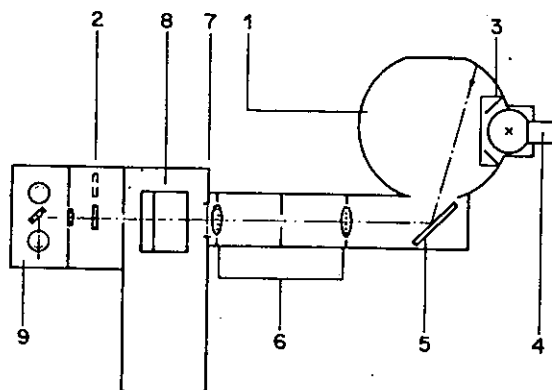
1: integrating sphere, 2: glazing sample, 3: shielding, 4: light source,
5: tipping mirror, 6: lenses, 7: variable inlet slit, 8: prism, 9: detectors
(photomultiplier, photoresistor)

Calibration: none

Sampling:

Sample size 5 cm x 5 cm.
Only samples without scattering effect (i.e. clear glazings with flat
confining surfaces)

Conditioning:

Procedures:

The integrating sphere of the spectrometer is illuminated by a light source. A tipping mirror at the bottom is pointed at a section of the sphere above it and transmits the beam originating from this section to a variable inlet slit. This beam is spectrally split by a prism and detected by a photo-multiplier (λ <0.6 µm) or a photoresistor (λ>0.6 µm).
For every wavelength in the first step the inlet slit is varied until the amplifier output of the detector reaches a given value of 100 %. In the second step the sample is inserted just in front of the detector and (at fixed position of the slit) the amplifier output U is read.

Calculation Method (if necessary, please include correction method):

transmittance τ: $\tau(\lambda) = U(\lambda)$

average transmittance $\bar{\tau}$
over the solar spectrum I_λ:

$$\bar{\tau} = \frac{\int_{0.3 \ \mu m}^{2.5 \ \mu m} I_\lambda \ \tau(\lambda) d\lambda}{\int_{0.3 \ \mu m}^{2.5 \ \mu m} I_\lambda d\lambda}$$

values of I_λ: see annex

Interpretation/Precision & Accuracy:

accuracy: $\pm$ 2 %

- 3 -

Annex

λ $[\mu m]$	$I_\lambda [\%]$
0.3 – 0.4	8.0
0.4 – 0.5	14.5
0.5 – 0.6	14.0
0.6 – 0.7	11.8
0.7 – 0.8	9.4
0.8 – 0.9	6.9
0.9 – 1.0	6.3
1.0 – 1.1	4.9
1.1 – 1.2	4.0
1.2 – 1.3	3.3
1.3 – 1.4	2.6
1.4 – 1.5	2.1
1.5 – 1.6	1.9
1.6 – 1.7	1.4
1.7 – 1.8	1.2
1.8 – 1.9	0.9
1.9 – 2.0	0.8
> 2.0	6.0
> 0.3	100

Date: 3/3/86

Reporter's Name: Katsuhiko Hojo

Country: Japan

Testing Title: Testing Methods for Yellowness Index and Change of Yellowness Index of Plastics.

Abstracted Scope (200 words):

(1) This standard specifies the testing method for the yellowness and the change in yellowness index of colorless or nealy white plastics.
(However, these do not include flouorescent plastics.)
(2) This standard specifies many measuring conditions.
  (a) For opaque on translucent filmy or platy test pieces.
  (b) For transparent filmy or platy test pieces.
  (c) For powdery samples.
  (d) For pellet form samples.
  (e) For thread form samples.
  (f) For liquid samples.

Referred Document:

JIS K 7103
"Testing Methods for Yellowness Index and Change of Yellowness Index of Plastics"

Principle of Measurement:

(1)Expressed by the difference between the initial yellowness index and the yellowness index after exposure.

(2)Reflection method:Transmission Method.(P.4)

Apparatus:

(1)The test apparance used shall be a colorimetric color difference meter having geometric conditions of illuminating from the optical direction of 45 also be used on agreement between the parties concerned.

> Remarks: The measurement shall be performed by the some measuring method with shown in page 4.(ANNEX)

Calibration:

Sampling:

(1)Sample shall be taken by a reasonable method from each lot.

(2)Measure ment shall be made on three test samples or test pieces.

Conditioning:

(1)temperature : 23 ± 5 °C (room temperature)

(2)relative humidity : $50\pm20\%$

Procedures:

(1) Preparation of samples and test pieces.

(2) Preparation of testing pieces.

(3) Perparation of test apparatus and measuring method.

(4) Calculation of the yellowness index and the chage in yellowness index.

Calculation Method (if necessary, please include correction method):

$$\Delta Y1 = Y1 - Y10$$

$$Y1 = \frac{100(1.28X - 1.06Z)}{Y}$$

$\Delta Y1$: change in yellowness index

Y1 : yellowness index after exposure

Y10: initial yellowness index of test sample or test piece

Y1 : yellowness index

X,Y,Z: tristimulus values of test sample or test piece based on standard source C

Interpretation/Precision & Accuracy:

ANNEX

1.Conditions for Colorimetric Measurement

(1)Reflection Method The measurement shall be performed by the
reflection measuring method by using a measuring area
(⌀50 mm diameter) without a backing plate on the backside of
the test piece and with a cover on the measuring apparatus to
shield it from outside light, as shown in Fig.1.
However, a measuring area (⌀30 mm diameter) may also be used
on agreement between the parties concerned. Where for unavoidable
reasons a backing plate is placed on the backside, the kind of
backing plate used or the tristimulus values shall be appended
in the test report.

(2)Transmission Method
The measurment shall be performed by
the transmission measuring method
with a measuring area (12 mm dimenter),
as shown in Fig.2.

Test piece

Measureing surface

Parallel light
rays
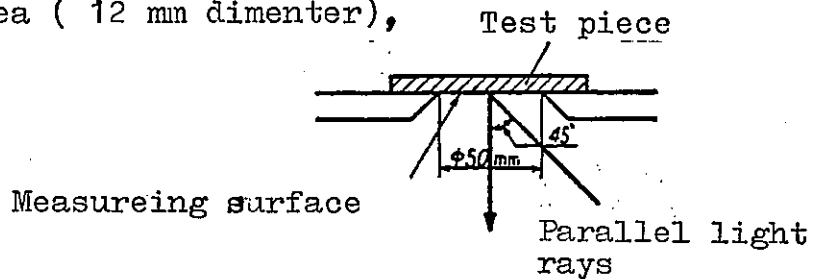
Fig.1. Reflection Measuring
Method

2.Measuring Method

(1)For Opaque or translucent filmy
or platy test pieces.(Fig.1.)
(2)For transparent filmy or platy
test pieces.(Fig.2.)
(3)For powdery samples.(Fig.3.)
For pellet from samples.(Fig.3.)
(4)For thread from samples,
For liquid samples.(Fig.4.)

light rays

Test piece

Measuring surface

Fig.2. Transmission Measuring Method

⌀60mm

Test sample

30mm

2mm

⌀50 mm

Light
rays

Measuring
surface

Fig.3.

⌀50mm

Light
rays

Aluminium measuring
frame

Sample
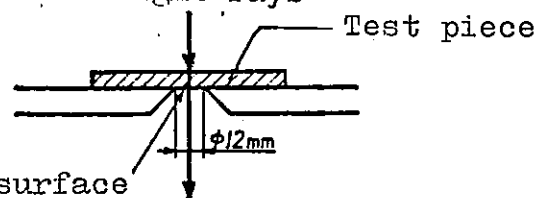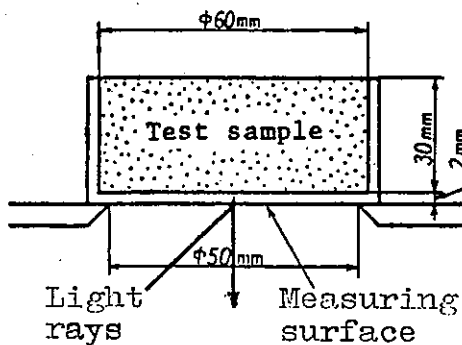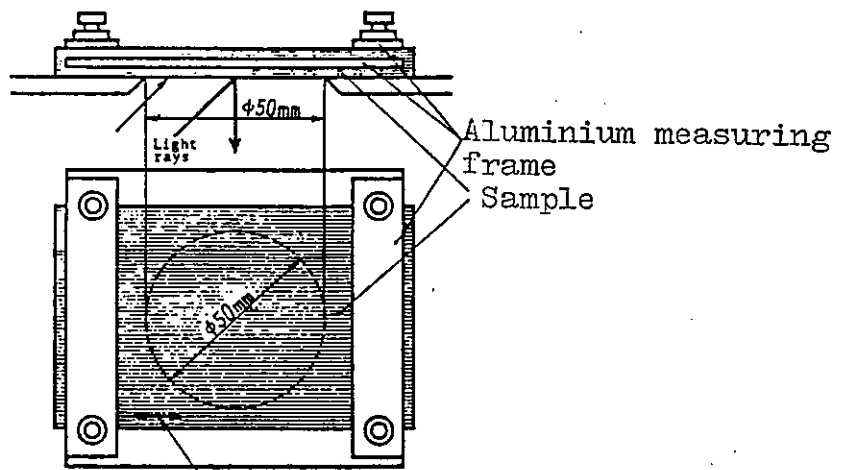
Winding direction of thread

Fig.4.

REPORTING FORMAT

Date:	April 7, 1986 Reporter's name: P.A. van Nijnatten
Country:	Technisch Physische Dienst TNO-TH (TNO Institute of Applied Physics) P.O. Box 155, 2600 AD DELFT The Netherlands
Testing title:	DETERMINATION OF THE SPECTRAL TRANSMITTANCE AND REFLECTANCE OF MULTIPLE GLAZING UNITS

Abstracted Scope (200 words):

Glazing units, consisting of two or more monolitic or composit panes of glass separated by an air or gas space, are usual to thick for making optical measurements with an ordinary spectro-photometer.
Instead, the optical properties of the single components are measured and used for calculations of the optical properties of the complete unit.

Referred document:

DIN 67507, ISO/TC 160/GT2

28

Principle of measurement:

Spectral measurement of the hemispherically transmitted and reflected flux using a spectro-photometer with an integrating sphere.

Apparatus:

Perkin Elmer Lambda 9 double-beam, double monochromator UV/Visible/Near Infrared spectrometer with 60 mm integrating sphere attachment (B013-9941).
For transmittance measurements, the sample is placed directly in front of the radiation inlet port of the sphere. All forward-scattered radiation is gathered in the sphere and included in the measurement.

Calibration:

White ceramic tile (NBS standard reference material 2020).

Sampling:

Approximately 30 x 30 mm

Conditioning:

Spectral transmittance: normal incidence
Spectral reflectance : near normal incidence (8 °)

Procedures:

1. Disassemble the glazing unit and make samples of the appropriate size.
2. Measure the necessary properties (see calculation method)
3. Calculate the spectral properties of the complete unit.
4. Calculate the normal total transmittance, the normal total reflectance and the general color rendering index as for a single pane of glass.

Calculation Method (if necessary, please include correction method):

Light reflectance of a double glazing unit

The spectral reflectances of the unit are equivalent to:

$$\rho_1(\lambda) = \rho_{I1}(\lambda) + \frac{\rho_{II1}(\lambda)\ \tau_I^2(\lambda)}{1 - \rho_{I2}(\lambda)\rho_{II1}(\lambda)} \quad \text{on entry surface}$$

$$\rho_2(\lambda) = \rho_{II2}(\lambda) + \frac{\rho_{I2}(\lambda)\ \tau_{II}^2(\lambda)}{1 - \rho_{I2}(\lambda) \cdot \rho_{II1}(\lambda)} \quad \text{on exit surface}$$

Light transmittance of a double glazing unit

To indicate the direction of the given incident light, the pane through which the light enters is numbered I and the pane through which it leaves is numbered II.

Pane I		Pane II
$\tau_I(\lambda)$	transmittance	$\tau_{II}(\lambda)$
$\rho_{I1}(\lambda)$	reflectance for the given direction	$\rho_{II1}(\lambda)$
$\rho_{I2}(\lambda)$	reflectance for the opposite direction	$\rho_{II2}(\lambda)$

The spectral transmittance of the unit is expressed by:

$$\tau(\lambda) = \frac{\tau_I(\lambda)\ \tau_{II}(\lambda)}{1 - \rho_{I2}(\lambda) \times \rho_{II1}(\lambda)}$$

for units consisting of more than two panes, the calculations are made by associating the components two by two successively.

Interpretation/Precision & Accuracy:

0,5 - 1%

REPORTING FORMAT

Designation:
AS/AC/GS/GC-O-LA

Date: December 1986   Reporter's Name: Michael G. Hutchins

Country: United Kingdom

Testing Title: DETERMINATION OF THE SOLAR ABSORPTANCE/REFLECTANCE/
TRANSMITTANCE FROM SPECTRAL MEASUREMENTS AT NEAR-
NORMAL INCIDENCE.

Abstracted Scope (200 words):

Spectral reflectance and/or spectral transmittance measurements are made
using a Beckman 5240 integrating sphere spectrophotometer in the spectral
range 0.3 - 2.5 μm. In reflectance mode radiation is incident on the
surface at 5° to the surface normal. In transmittance mode the radiation is
incident normally on a surface placed over a sphere entry port.

Solar optical properties are calculated by convoluting the spectral
reflectance curve with a standard solar spectral distribution curve for
air mass 2 using a method of 20 selected ordinates. One of two solar
spectral distributions may be used (1,2).

The measurement procedure is automated using an Apple IIe microcomputer.

Referred Document:

1. Wiebelt J.A. and Henderson J.B., J. Heat Transfer 101, 101, 1979.

2. Moon P., J. Franklin Inst., 230, 583, 1940.

83

Principle of Measurement:

Near-normal hemispherical spectral reflectance or transmittance of incident radiation.

Apparatus:

Beckman 5240 uv/vis/nir integrating sphere spectrophotometer using photomultiplier for uv/vis detection and PbS for nir detection.
6" diameter integrating sphere coated with barium sulphate white reflectance standard. Double beam ration recording principle of operation.
Apple IIe microcomputer for calculation of optical properties.

Calibration:
For diffuse surfaces : barium sulphate
For highly specular surfaces : aluminium mirror

Sampling:

Minimum sample size : 30mm x 30mm
Maximum sample size : 100mm x 100mm

Conditioning:

None

Procedures:

1. Sample cut to size and cleaned by air blowing.
2. Instrument is calibrated using two barium sulphate reference plates.
3. Sample mounted on sphere wall (rear for reflectance : front for transmittance) and reflectance or transmittance measured for scan from 2500nm – 350nm.
4. Spectral data at 1nm intervals are sent to an Apple IIe microcomputer.
5. Microcomputer executes calculation to determine surface optical properties, e.g. α_s, τ_s or ρ_s.

Calculation Method (if necessary, please include correction method):

20 selected ordinates for air mass 2 solar spectral distribution giving

$$\alpha_s = \frac{1}{20} \sum_{i=1}^{20} \alpha_i \ , \quad \text{where } \alpha_i \text{ is the absorptance at each of the twenty}$$

selected ordinates and is found from $\alpha_i = 1 - \rho_i$

$$\tau_s = \frac{1}{20} \sum_{i=1}^{20} \tau_i \ , \quad \text{where } \tau_i \text{ is the transmittance at each of the twenty}$$
selected ordinates.

Interpretation/Precision & Accuracy:

Precision : reproducible to one part in 10^3.
Accuracy : 1-2%.

REPORTS ON GLAZING

a (iii) REFLECTANCE

REPORTING FORMAT

Date:	April 7, 1986	Reporter's name: P.A. van Nijnatten

Country: Technisch Physische Dienst TNO-TH
           (TNO Institute of Applied Physics)
           P.O. Box 155, 2600 AD DELFT
           The Netherlands

Testing title: DETERMINATION OF THE SPECTRAL TRANSMITTANCE AND REFLECTANCE
               OF MULTIPLE GLAZING UNITS

Abstracted Scope (200 words):

Glazing units, consisting of two or more monolitic or composit panes of glass
separated by an air or gas space, are usual to thick for making optical
measurements with an ordinary spectro-photometer.
Instead, the optical properties of the single components are measured and used
for calculations of the optical properties of the complete unit.

Referred document:

DIN 67507, ISO/TC 160/GT2

37

Principle of measurement:

Spectral measurement of the hemispherically transmitted and reflected flux using a spectro-photometer with an integrating sphere.

Apparatus:

Perkin Elmer Lambda 9 double-beam, double monochromator UV/Visible/Near Infrared spectrometer with 60 mm integrating sphere attachment (B013-9941).
For transmittance measurements, the sample is placed directly in front of the radiation inlet port of the sphere. All forward-scattered radiation is gathered in the sphere and included in the measurement.

Calibration:

White ceramic tile (NBS standard reference material 2020).

Sampling:

Approximately 30 x 30 mm

Conditioning:

Spectral transmittance: normal incidence
Spectral reflectance : near normal incidence (8 °)

Procedures:

1. Disassemble the glazing unit and make samples of the appropriate size.
2. Measure the necessary properties (see calculation method)
3. Calculate the spectral properties of the complete unit.
4. Calculate the normal total transmittance, the normal total reflectance and the general color rendering index as for a single pane of glass.

Calculation Method (if necessary, please include correction method):

Light reflectance of a double glazing unit

The spectral reflectances of the unit are equivalent to:

$$\rho_1(\lambda) = \rho_{I1}(\lambda) + \frac{\rho_{II1}(\lambda)\,\tau_I^2(\lambda)}{1 - \rho_{I2}(\lambda)\rho_{II1}(\lambda)} \quad \text{on entry surface}$$

$$\rho_2(\lambda) = \rho_{II2}(\lambda) + \frac{\rho_{I2}(\lambda)\,\tau_{II}^2(\lambda)}{1 - \rho_{I2}(\lambda)\rho_{II1}(\lambda)} \quad \text{on exit surface}$$

Light transmittance of a double glazing unit

To indicate the direction of the given incident light, the
pane through which the light enters is numbered I and the
pane through which it leaves is numbered II.

Pane I		Pane II
$\tau_I(\lambda)$	transmittance	$\tau_{II}(\lambda)$
$\rho_{I1}(\lambda)$	reflectance for the given direction	$\rho_{II1}(\lambda)$
$\rho_{I2}(\lambda)$	reflectance for the opposite direction	$\rho_{II2}(\lambda)$

The spectral transmittance of the unit is expressed by:

$$\tau(\lambda) = \frac{\tau_I(\lambda)\,\tau_{II}(\lambda)}{1 - \rho_{I2}(\lambda) \times \rho_{II1}(\lambda)}$$

for units consisting of more than two panes, the calculations are made by
associating the components two by two successively.

Interpretation/Precision & Accuracy:

0,5 - 1%

Date: December 1986   Reporter's Name: Michael G. Hutchins

Country: United Kingdom

Testing Title: DETERMINATION OF THE SOLAR ABSORPTANCE/REFLECTANCE/
TRANSMITTANCE FROM SPECTRAL MEASUREMENTS AT NEAR-
NORMAL INCIDENCE

Abstracted Scope (200 words):

Spectral reflectance and/or spectral transmittance measurements are made
using a Beckman 5240 integrating sphere spectrophotometer in the spectral
range 0.3 - 2.5 μm. In reflectance mode radiation is incident on the
surface at 5° to the surface normal. In transmittance mode the radiation is
incident normally on a surface placed over a sphere entry port.

Solar optical properties are calculated by convoluting the spectral
reflectance curve with a standard solar spectral distribution curve for
air mass 2 using a method of 20 selected ordinates. One of two solar
spectral distributions may be used (1,2).

The measurement procedure is automated using an Apple IIe microcomputer.

Referred Document:

1.   Wiebelt J.A. and Henderson J.B., J. Heat Transfer 101, 101, 1979.

2.   Moon P., J. Franklin Inst., 230, 583, 1940.

Principle of Measurement:

Near-normal hemispherical spectral reflectance or transmittance of incident radiation.

Apparatus:

Beckman 5240 uv/vis/nir integrating sphere spectrophotometer using photomultiplier for uv/vis detection and PbS for nir detection.
6" diameter integrating sphere coated with barium sulphate white reflectance standard. Double beam ration recording principle of operation.
Apple IIe microcomputer for calculation of optical properties.

Calibration:
For diffuse surfaces : barium sulphate
For highly specular surfaces : aluminium mirror

Sampling:

Minimum sample size : 30mm x 30mm
Maximum sample size : 100mm x 100mm

Conditioning:

None

Procedures:

1. Sample cut to size and cleaned by air blowing.
2. Instrument is calibrated using two barium sulphate reference plates.
3. Sample mounted on sphere wall (rear for reflectance : front for transmittance) and reflectance or transmittance measured for scan from 2500nm – 350nm.
4. Spectral data at 1nm intervals are sent to an Apple IIe microcomputer.
5. Microcomputer executes calculation to determine surface optical properties, e.g. α_s, τ_s or ρ_s.

Calculation Method (if necessary, please include correction method):

20 selected ordinates for air mass 2 solar spectral distribution giving

$$\alpha_s = \frac{1}{20} \sum_{i=1}^{20} \alpha_i \ , \quad \text{where } \alpha_i \text{ is the absorptance at each of the twenty}$$

selected ordinates and is found from $\alpha_i = 1 - \rho_i$.

$$\tau_s = \frac{1}{20} \sum_{i=1}^{20} \tau_i \ , \quad \text{where } \tau_i \text{ is the transmittance at each of the twenty}$$
$$\text{selected ordinates.}$$

Interpretation/Precision & Accuracy:

Precision : reproducible to one part in 10^3.
Accuracy : 1-2%.

REPORTS ON GLAZING

a (iv) EMITTANCE AT A DESIGNATED TEMPERATURE

__Date__: Feb.5,]986   __Reporter's Name__: K. Matsuoka

__Country__: Japan

__Testing Title__: Testing Method On Emittance At The Designated
Temperature.

Abstracted Scope (200 words):

This testing method is applied to measure.
Emittance at the designated temperature of
the transparent flat glass in an optical
manner.

The transparent flat glass includes Float glass,
Heat absorbing glass, Thin film coated reflective
glass, Low iron glass and etc. The both sides
of the glass must be flat.

Referred Document:

JIS   R 3]06-]985
Testing Method on Transmittance and Reflectance for

Daylight and Solar Radiation and Solar Heat Gain

Coefficient of Flat Glass.

Principle of Measurement:

Using the reference reflective material, Spectral
Reflectance is measured. From these data Reflectance
and Absorptance against Heat flux are calculated by
the Equation. Then Hemispherical Emittance is obtained
by modification of Coefficiency.

Apparatus:

H "HITACHI" 270/30 Model Infrared Spectro-photometer

Aluminum vacuum deposited plate

Spectro-photometer

Reference Beam

Sample Beam

Sample

Detector
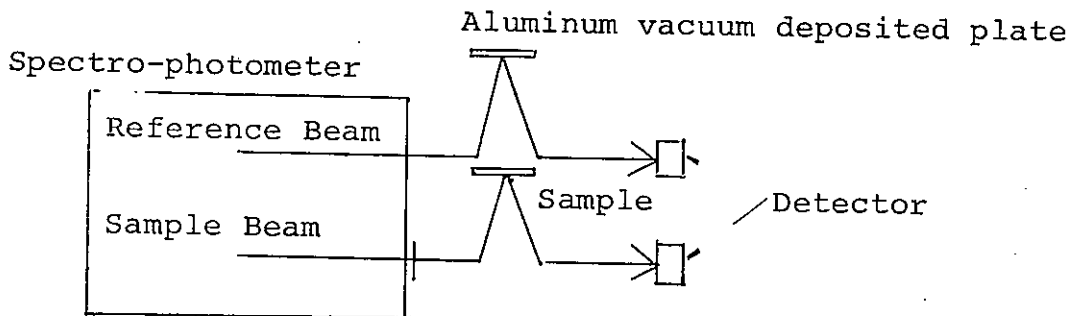
Calibration:

Sampling:

Size of sample : 50mm x 50mm

Conditioning:

Keep the both sides of the sample smooth and parallel.

Procedures:

(1) Use the designated Spectral Photometer.
(2) Measure Spectral Reflectance $\rho(\lambda)$ of the sample
at the intervals of 0.5μm wavelength over the wavelength
range of 4.5 - 25μm, projecting Heat flux in infrared
wave range with the incidence angle less than 15° to
normal.

As the Reference reflective material, use the surface
mirror which is a thick aluminum vacuum deposited
float glass, and it's $\rho(\lambda)=0.98$

(3) Calculate Heat Reflectance ρ_h, using $\rho(\lambda)$ and the
equation (1).

(4) Calculate Normal Heat Absorptance (= Normal Emittance).

(5) Obtain Hemispherical Emittance .

Calculation Method (if necessary, please include correction method):

Heat Reflectance;
$$\rho_h = \sum_{4.5}^{25} G\lambda \cdot \rho(\lambda) \quad \cdots \cdots \cdots (1)$$
here, see Gλ in Annex(2).
Normal Heat Absorptance (=Normal Emittance);
$$\alpha_h = 1 - \rho_h$$
Hemispherical Emittance;
$$\varepsilon_h = C \cdot \alpha_h \qquad \begin{array}{l} c=0.94 \text{ for uncoated flat glass} \\ c=0.94 \text{ for metal oxyde coated surface} \end{array}$$

Interpretation/Precision & Accuracy:

Annex 2. Factor To Calculate Reflectance For Heat Radiation At 293K.

wave length λ (μm)	wave number $\bar{\nu}$ (cm^{-1})	$G\lambda$
4. 5	2 222	0. 005 3
5. 0	2 000	0. 009 4
5. 5	1 818	0. 014 3
6. 0	1 667	0. 019 4
6. 5	1 538	0. 024 4
7. 0	1 429	0. 029 0
7. 5	1 333	0. 032 8
8. 0	1 250	0. 035 8
8. 5	1 176	0. 037 9
9. 0	1 111	0. 039 3
9. 5	1 053	0. 040 1
10. 0	1 000	0. 040 2
10. 5	952	0. 039 9
11. 0	909	0. 039 2
11. 5	870	0. 038 2
12. 0	833	0. 037 0
12. 5	800	0. 035 6
13. 0	769	0. 034 2
13. 5	741	0. 032 7
14. 0	714	0. 031 1
14. 5	690	0. 029 6
15. 0	667	0. 028 1
15. 5	645	0. 026 6
16. 0	625	0. 025 2
16. 5	606	0. 023 8
17. 0	588	0. 022 5
17. 5	571	0. 021 2
18. 0	556	0. 020 0
18. 5	541	0. 018 9
19. 0	526	0. 017 9
19. 5	513	0. 016 8
20. 0	500	0. 015 9
20. 5	488	0. 015 0
21. 0	476	0. 014 2
21. 5	465	0. 013 4
22. 0	455	0. 012 6
22. 5	444	0. 011 9
23. 0	435	0. 011 3
23. 5	426	0. 010 7
24. 0	417	0. 010 1
24. 5	408	0. 009 6
25. 0	400	0. 009 1

注 ＊ 波数目盛の分光測定器を使用するときの便宜のために付
記したものである。

Date: March 1, 1986   Reporter's Name: S. TANEMURA

Country: JAPAN

Testing Title:

    Measurement of Normal Spectral Emittance of Absorber & Glazing Material

Abstracted Scope (200 words):

    To evaluate the selective absorbing property of the absorber materials for solar collector and the selective transparent property of the glazing materials for both solar collector and the radiation control window, normal spectral emittance (NSE) from 2.5 μm to 25 μm in wavelength and from 50°C to 250°C in the sample temperature is an important physical property to be measured. To minimumize the measurement errors due to the uncertainity of the equivalence of both sample elevated temperature and the blackbody temperature and the drift of those temperatures during the emperiment FT-IR spectrophotometry is used. The surface temperature of the sample and that of blackbody were determined by the relationship beween the monitored TC-reading and the indicated temperatures by thermo-visualizer equipment which is experimentally obtained for various total emittance value assumed tentatively to the sample.

Referred Document:

    S. TANEMURA et al, Abstract of 39th Semiannual Meeting on Research Achievements in G.I.R.I. Nagoya, p.91 (1985) (in Japanese)

<u>Principle of Measurement:</u> The notation $V_s(\lambda)$, $V_b(\lambda)$ and $V_r(\lambda)$ are defined as the detected spectral signal of the sample heated at T_s K, that of the blackbody controlled at T_s K and that of the perfect reflector at room temperature respectively. Then, NSE $\varepsilon(\lambda, T_s)$ of the sample at T_s K is derived from the equation.

$$\varepsilon(\lambda, T_s) = [V_s(\lambda) - V_r(\lambda)] / [V_b(\lambda) - V_r(\lambda)].$$

Hence, the measurements of $V_s(\lambda)$, $V_b(\lambda)$ and $V_r(\lambda)$ are required.

<u>Apparatus:</u>

Low Temp. Range: (60~85°C) Sample heating furnace (SF) and blackbody (B) have the hot water reservoirs to which the hot water controlled at a designated temperature is circulated simultaneously. The auxiliary electric heater is provided at the inlet tube to the sample furnace.
Medium Temp. Range: (100~250°C) Apparatures SF and B are controlled independently by proportional-differential electronic control device.

Fig.1, Schematics of present normal spectral emmisiometer.

<u>Sampling:</u>

The size of the sample; 25 (mm) X 25 (mm) X 5 (mm).

<u>Conditioning:</u>

Preconditioning
  1. To avoid the measurement of the transient surface properties under the applied temperature in the normal room environmental conditions, the sample is kept at a designated temperature for 12 hrs at least before measurement.
  2. Thick Au coating is required to the rear surface of transparent glazing materials.

299

Procedures:

After preconditioning, the followings are processed:

(1) Obtaining the relationship between the TC reading of the blackbody and the indicated temperature by thermovisualizer under viewing the blackbody center.

(2) Obtaining the relationships between the readings of TC provided to be touched on the surface of the sample and the indicated temperature by thermovisualizer under viewing the heated sample surface as the function of the varius total emittance values assumed tentatively for the sample.

(3) $V_r(\lambda)$ measurement

(4) Control the blackbody at a designated temperature (T_b)

(5) Control the sample at the temperature (T_s) approximately to be regarded as the equivalent to T_b value.

(6) $V_s(\lambda)$ and $V_b(\lambda)$ measurement.

(7) ε calculation and the output of ε as the function of wavelength.

(8) Estimation of the total emittance considering with the response function of the detector of the thermovisualizer equipment.

(9) Confirmation of the sample surface temperature according to the relationship described in (2). If necessary, the adjustment of the controlled sample temperature and procedures (5) - (9) are required to be repeated.

Calculation Method (if necessary, please include correction method):

Opaque Material: ε is calculated by the equation described in the Principle.

Transparent Material: Both Normal Spectral Reflectance (NSR) and Normal Spectral Transmittance data of the sample without rear surface Au coating as well as the NSR of Au coating are also required to obtain NSE. The optical model with four phases such as ambient, surface coating, glazing and Au coating is required to calculate the NSE.

Interpretation/Precision & Accuracy:

Typical example:

(1) Precision of NSE less than ±0.02 under Temperature drift ±0.3°C, Difference of T_b - T_s = ±0.5°C in emittance units.

(2) Reproducibility less than ±0.01 in emittance units.

REPORTING FORMAT

Designation:
G-O-LA

Date: 1986-03-16   Reporter's Name:

Country: Sweden

Testing title:

Measurement of reflectance for determination of hemispherical total
emittance at 10 °C temperature.

Abstracted Scope (200 words):

To determine hemispherical total emittance from measurements of 15/d
reflectance measurements in four wavelength bands:

3.6-5 μm; 7-13 μm; 13-30 μm and 24-30 μm

Referred Document:

Principle of Measurement:

The 15/d reflectance is compared with the reflectance of a gold
mirror using a diffusely gold coated integrating sphere in four
wavelength bands.

Apparatus:

100 mm diameter integrating sphere (diffuse gold)
InAs - and MCT-detector
Golay detector
Pyrometer
Cut-on filters
Stabilized glowing rod source with chopper
Lock-in amplifier
Standard: Specular gold mirror
Voltmeter: HP 3455 A

Calibration:

Sampling:

Samples must be flat and larger than 3 x 3 cm^2.

Conditioning:

Procedures:

The glowing rod source is stabilized at 1100 °C using a pyrometer
and feedback.

The radiation is collimated into the sphere and the sample
irradiated with 15° angle of incidence.

The wavelength region is limited by the type of detector used and by
a cut-on filter between the sphere and the source.

At each wavelength range the detector output is measured with the
sample port open (do), with the sample attached (dx) and with the
standard attached (ds).

Calculation method:

$\rho_1 = 0.99\ (dx{-}do)/(dm{-}do)$
$\rho = -6 + \sqrt{36+13\cdot\rho_1}$ (empirical correction formula for the
sphere transmission characteristics)

The wavelength ranges gives the reflectance values at the following
effective wavelengths: 4.3; 8.9; 17.7 och 26.3 µm

An interpolating curve is calculated for intermediate wavelengths
using quadratic interpolation. Above 26 µm constant reflectance is
assumed.

This reflectance curve is weighted with the Planckian radiation law
at 10 °C temperature giving a mean reflectance ρ from which the
emissivity ε_{15} is calculated as $\varepsilon_{15} = 1-\rho$.

An empirical curve for calculation of total hemispherical emissivity
from the 15° emissivity is used.

Interpretation:

Precision: <±0.01   95 % confidence level

Accuracy: <±0.02   (estimated value)

bs/1735L

REPORTING FORMAT

Date:	April 7, 1986	Reporter's name: P.A. van Nijnatten

Country: Technisch Physische Dienst TNO-TH
(TNO Institute of Applied Physics)
P.O. Box 155, 2600 AD DELFT
The Netherlands

Testing title: DETERMINATION OF DIRECTIONAL AND HEMISPHERICAL TOTAL EMITTANCE
AT TEMPERATURES OF 60 °C TO 200 °C.

Abstracted Scope (200 words):

The hemispherical total emittance is used in calculating radiative heat transfer
in collector and window glazings, collector absorber materials and coatings etc.
An apparatus designed after Irving et al [1] is used for quick measurements of
the total emittance at different emitting angles and at different surface
temperatures.
The hemispherical total emittance is obtained by integrating over all emitting
angles.

Referred document:

[1] Irving, T.F., Hartnett, J.P. and Eckert, E.R.G., Solar Energy, 2 (1958) 13.
[2] Spectral-selective surfaces for the thermal conversion of Solar Energy,
M. van der Leij, Delft University Press, 1979.

3a

Principle of measurement:

Radiometric measurement of the radiance of heated samples.

Apparatus:

θ_e. Emitting angle
1. Sample
2. Heating unit (canthal wire 14 Ω) and Cromel-Alumel thermocouples
3. Water cooled jacket, 5°C.
4. Water cooled diaphragm, $\emptyset$ 0.010 m
5. Bolometer + electronics (Heimann KT 14)
6. Detector head with Ge-lens (Heimann, type L)
7. Voltage supply (24 VAC)
8. Galvanometer Kipp
9. Rotating disc with knob and graduating disc, outside the jacket
10. Outer housing filled with dry air
11. Temperature controller and recorder

The angle θ_c is adjustable from 15° – 75°.
The heating unit is surrounded by a black-pointed cooled jacket in order to reduce reflections from the wall to the sample and to keep the ambient temperature constant. The spectral sensitivity of the detection is from 2-20 μm (mainly determined by the Ge-lens.

Calibration:

Heimann calibration radiator (type SW13) or black body radiator at sample position.

Sampling:

The sample is attached to the heater with a heat transfer compound or screws
Only a spot of $\emptyset$ 4.10^{-3} m is seen by the detector.

Conditioning:

- The samples and references have to have approximately the same thermal
  conductivity.
- To avoid condensation on the coated wall, the apparatus is purged with dry air.

Procedures:

1. The sample is thoroughly cleaned and attached to the heater.
2. The temperature of the heating unit is set.
3. After sufficient warm-up time the recorder reading gets stable and measurement
   begins.
4. Measurements are made at view-angle intervals of 5°.

Calculation Method (if necessary, please include correction method):

If it is assumed that the inner wall of the cooling jacket with temperature T_o(K) is black, the total energy flux density G coming from the direction of the sample surface of temperature T_s(K) received by the detector surface is given by

$$G = F\sigma \ [\ \epsilon(\theta_e,T_s) \ T_s^4 + \rho(\theta_e,T_s) \ T_w^4],$$

in which F is the view factor between the sample area and the detector area, σ is the Stephan-Boltzmann constant, $\epsilon(\theta_e,T_s)$ is the directional total emittance of the sample with θ_e as the angle between the normal on the centre of the sample and the connecting line from the centre of the detector area to the centre of the sample, $\rho(\theta_e,T_s)$ is the hemispherical directional total reflectance of the sample with temperature T_s, and T_w is the temperature of the cooled jacket. If the detector is calibrated with a black body radiator, then the energy flux density from the sample falling on the detector, as seen by the detector as coming from a black surface with temperature T_x, the emitted energy flux density is

$$G' = F \ \sigma \ T_x^4.$$

On the basis of this idea, G = G', so that

$$T_x^4 = \epsilon(\theta_e,T_s)T_s^4 + \rho(\theta_e,T_s)T_w^4.$$

By taking
$$\epsilon(\theta_e,T_s) = 1 - \rho(\theta_e,T_s),$$

we can write $\epsilon(\theta_e,T_s)$ as

$$\epsilon(\theta_e,T_s) = \frac{T_x^4 - T_w^4}{T_s^4 - T_w^4}.$$

This will be the basic equation for our measurements if we assume that the detector response is (linearly) proportional to the intensity of the radiation received.

Interpretation/Precision & Accuracy:

$\epsilon < 0,2$: $\Delta\epsilon = \pm \ 0,02$

$\epsilon > 0,2$: $\Delta\epsilon = \pm \ 0,01$

ate: December 1986

Reporter's Name: Michael G. Hutchins

Country: United Kingdom

Testing Title: Measurement of near-normal total thermal emittance
using a pyroelectric radiometer

Abstracted Scope (200 words):

Thermal radiation from a heated test sample is chopped and focussed, using
a KBr convex lens, onto the element of a pyroelectric detector equipped
with a KRS-5 window. A saw-tooth waveform with peak-to-peak height dependent
on the temperature difference between the sample and a black-body background
and the test sample thermal emittance is produced. This signal and a reference
signal from the chopper are fed to a lock-in amplifier and the resultant dc
level is measured. Readings are taken of this dc voltage as a function of
the temperature difference between the test sample and the background.
A plot of V against ΔT produces a straight line for $\Delta T < 10^{\circ}C$. The ratio
of the gradient of this line to that produced by a blackbody reference
yields the thermal emittance of the test sample.

Referred Document:

Hutchins M.G. , Optica Pura y Aplicada 12, 15, 1979.

Principle of Measurement:

Direct measurement of thermal radiation (total radiation emitted normal to surface). Pyroelectric detection ensures flat spectral response for all wavelengths of interest. Emittance values are derived by comparison with radiation measurements from blackbody reference standards.

Apparatus:

In-house development. Test sample is resistively heated. Test sample surface temperature and temperature of ambient sensed with K-type thermocouples. Rofin-Sinar chopper, operating at 16 Hz modulates radiation from heated sample. 2" focal length KBr convex lens focusses thermal radiation onto triglycine sulphate pyroelectric detector which is protected by a KRS-5 ir transparent window. Pyroelectric detector is Plessey Model PSC222. Signal conditioning is performed using an Ortec 9501 lock-in amplifier. Optical system is mounted on a 0.25 m optical bench and housed within a light proof, blackened wooden box. The interior of the box provides the blackbody ambient background radiation level.

Calibration: Using a blackbody reference and samples of known emittance

Sampling:

Minimum sample size   5mm x 5mm
Maximum sample size 75mm x 75mm

Conditioning:

None

Procedures:

1. Sample is mounted in holder, thermocouple is attached to front surface, and is then heated to a known temperature T above the ambient temperature T_a.

2. The total thermal radiation emitted normally from the surface is focussed onto the detector element and the peak to peak height of the waveform is measured using a lock-in amplifier.

3. The temperature of the sample is changed and a new value of amplifier output, V (volts), is recorded. This is performed for 6-10 different values of T.

4. A graph of V v. $T-T_a$ is plotted and a straight line is fitted to the data. The gradient of the line is calculated.

5. The ratio of the gradient of the line produced for the test sample and the gradient of the line produced from the reference sample yields the total normal thermal emittance of the test surface.

Calculation Method (if necessary, please include correction method):

The ratio of output voltages from the test sample and the reference yields the thermal emittance on the assumptions that the blackbody and sample have equal temperatures and that for $T - T_a < 10°C$ the radiation terms may be approximated by

$$T^4 - T_a^4 = \bar{T}^3 (T - T_a)$$

where

$$\bar{T} = (T + T_a) / 2$$

Interpretation/Precision & Accuracy:

Reproducibility : 1-2 %

Accuracy : 5%

- 3 -

Date: December 1986   Reporter's Name: Michael G Hutchins

Country: United Kingdom

Testing Title: MEASUREMENT OF THERMAL EMITTANCE FROM SPECULAR REFLECTANCE
USING AN INFRARED SPECTROPHOTOMETER

Abstracted Scope (200 words):

Test samples are mounted in a dual-beam ratio-recording spectrophotometer
in a holder that allows incident radiation to strike the surface at 37° to
the surface normal. Specularly reflected radiation is detected by a
thermocouple and an output of infrared specular reflectance as a function
of incident radiation wavelength in the rane 2.5 - 50.0 µm is recorded.

The test sample temperature is known (307 K) and the specular reflectance
curve is convoluted with a blackbody spectral emissive curve for the same
temperature using an equal energy interval mathod of 20 selected ordinates.

The accuracy of the method is poor if the test surface has surface roughness
features which are seen by the incident infrared radiation. Since the
diffuse component of the reflectance is lost and

$$\alpha_\lambda \;=\; \varepsilon_\lambda \;=\; 1 \;-\; \rho_\lambda$$

all measured values of thermal emittance, ε, will be equal to or greater than
the true value.

Referred Document:

None known

Principle of Measurement:

The incident and reflected angles are fixed at 37° so that values of thermal emittance are spectral or total values but only for the fixed geometry of the system.

Apparatus:

Perkin Elmer 683 dual-beam ratio-recording infrared spectrophotometer. Reference calibration is made using an aluminium mirror deposited onto a glass substrate.

Calibration: Aluminium mirror

Sampling:

Sample size in the range 25 mm x 25 mm - 100 mm x 100 mm

Conditioning:

Sample must be left to reach the working temperature of the spectrophotometer measurement chamber.

Procedures:

1. The reflectance of the spectrophotmeter is calibrated using an aluminium mirror.

2. The test sample is mounted and allowed to come into thermal equilibrium.

3. Spectral reflectance data are recorded for a scan between 2.5 μm and 50.0 μm.

4. 20 selected ordinates for the reference blackbody spectral distribution at 307 K are used to read off reflectance data for the test sample from the chart recording.

5. The thermal emittance is calculated using the standard method.

Calculation Method (if necessary, please include correction method):

For each of the twenty selected ordinates a value of reflectance ρ_i is taken.

The monochromatic thermal emittance, ε_i, is then found from

$$\varepsilon_i = 1 - \rho_i$$

The integrated thermal emittance is then found from $\varepsilon = \dfrac{1}{20} \sum_{i=1}^{20} \varepsilon_i$

Interpretation/Precision & Accuracy:

Reproducibility : 3%

Accuracy : For smooth specular surfaces - 3%
For rough surfaces - impossible to quantify in a simple way

REPORTS ON GLAZING

b.   SERVICE REQUIREMENT PROPERTIES

b (i)   MECHANICAL STRENGTH: TENSILE PROPERTIES,
DIMENSIONAL STABILITY, ETC.

REPORTING FORMAT

Designation :

GS—R—ST

Date : Feb. 27, 1986   Reporter's Name : Sadamitsu Tsuboi

Country : Japan

Testing Title : Four point Bending Test of Sheet glass.

Abstracted Scope (200 words) :

This test is used for measuring static surface strength and edge strength of sheet glass.

A narrow piece of glass is set on a pair of knife edge supporting block, and loaded on the center with a pair of knife edge loading block.

Using the load at breakage point, breakage stress can be calculated by means of bending stress formula.

Referred Document :

ASTM Standard C 158—43

<u>Principle of Measurement</u>:

      Bending stress in glass is nearly constant between a pair of knife edge loading block. Breakage stress can be easily calculated by means of bending stress formula.

<u>Apparatus</u>:
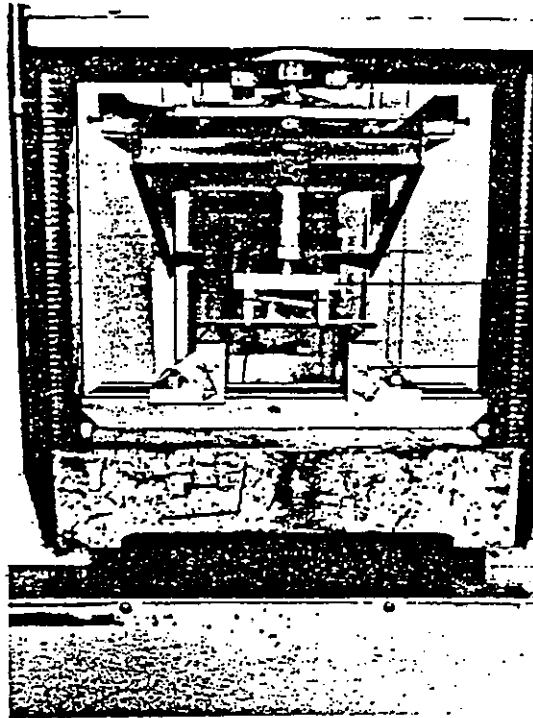
Conventional Testing Machine

Knife edge Loading Block

Knife edge Supporting Block

Four Point Bending Test Machine

<u>Calibration</u>:

<u>Sampling</u>:

<u>Conditioning</u>:

    At room temperature

    Stress speed 10kg/cm^2 sec

    Maximum deflection glass thickness X $\frac{1}{2}$

Procedures:

    1. Set the knife edge supporting blocks and loading blocks on a
       conventional testing machine.

    2. Set sample on a pair of knife edge supporting block.

    3. Put load on a sample with a pair of loading block and record
       the electrical output of load cell.

    4. Read the load at breakage point and caluculate breakage stress.

Calculation Method (if necessary, please include correction method):

$$\sigma = \frac{3p(L-1)}{2bd^2}$$

σ = maximum bending stress

L = distance between supporting blocks

l = distance between loading blocks

b = width of sample

d = thickness of sample

p = load

Interpretation/Precision & Accuracy:

REPORTING FORMAT

Date: Feb. 28, 1986   Reporter's Name: Sadamitsu Tsuboi

Country: Japan

Testing Title: Ring on Ring Bending Test

Abstracted Scope (200 words):

This test is applied to measure static surface strength of sheet glass.

Sampels are set on the supporting ring (outer ring) and concentrically loaded with the loading ring (inner ring).

Breakage strength can be caluculated by means of bending stress formula.

Referred Document:

Test method of measuring glass strength
(NIPPON SHEET GLASS CO., LTD.)

Principle of Measurement:

Bending stress in glass is nearly constant in the area of inner ring. Breakage stress can be easily calculated by means of bending stress formula.

Apparatus:
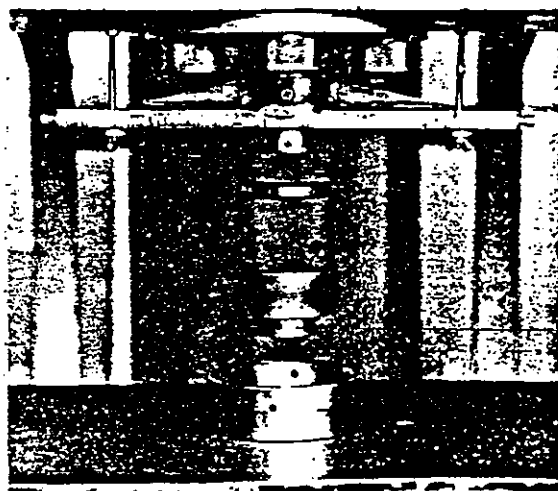
Conventional Testing Machine

Upper Ring

Lower Ring

Ring on Ring Bending Test Machine

Calibration:

Sampling:

Conditioning:

At room temperature

Stress speed ≤ 10kg/cm^2 sec

$a \geq 2.5d$

$r_o \geq 1.7d$

$b'-a > b$ d

maximum deflection $\leq 0.5d$

$b' =$ sample length X $\frac{1}{2}$

Procedures:

1. Set the outer ring and the inner ring which should be selected by sample and several bending conditions.

2. Put sample on the outer ring.

3. Put load on sample with inner ring and record the electrical output of loadcell.

4. Read the load at breakage point and calculate breakage stress, if breakage point starts from area of inner ring.

Calculation Method (if necessary, please include correction method):

$$\sigma_{max} = \frac{3p}{4\pi d^2} \left[2(1+\nu) \ln \frac{a}{r_o} + \frac{(1-\nu)(a^2-r_o^2)}{a^2} \cdot \frac{a^2}{b^2} \right]$$

a = outer ring radius   r = poison's ratio

r_o = inner ring radius   p = load

b = effective radius of sample

d = sample thickness

Interpretation/Precision & Accuracy:

Date: Feb. 27, 1986   Reporter's Name: Sadamitsu Tsuboi

Country: Japan

Testing Title: Test Method of Impact Strength of Sheet Glass using Steel Ball

Abstracted Scope (200 words):

This test is applied to Tempered sheet glass.

A designated (weight and shape) steel ball is dropped on the center of sample from various height in order to know breakage height.

Referred Document:

JIS R 3206 Tempered Glass

Principle of Measurement:

This test provides us relative impact strength of Tempered glass using same steel ball, same glass size and same supporting frame.

Apparatus:

Unite mm

Steel Ball Test Frame
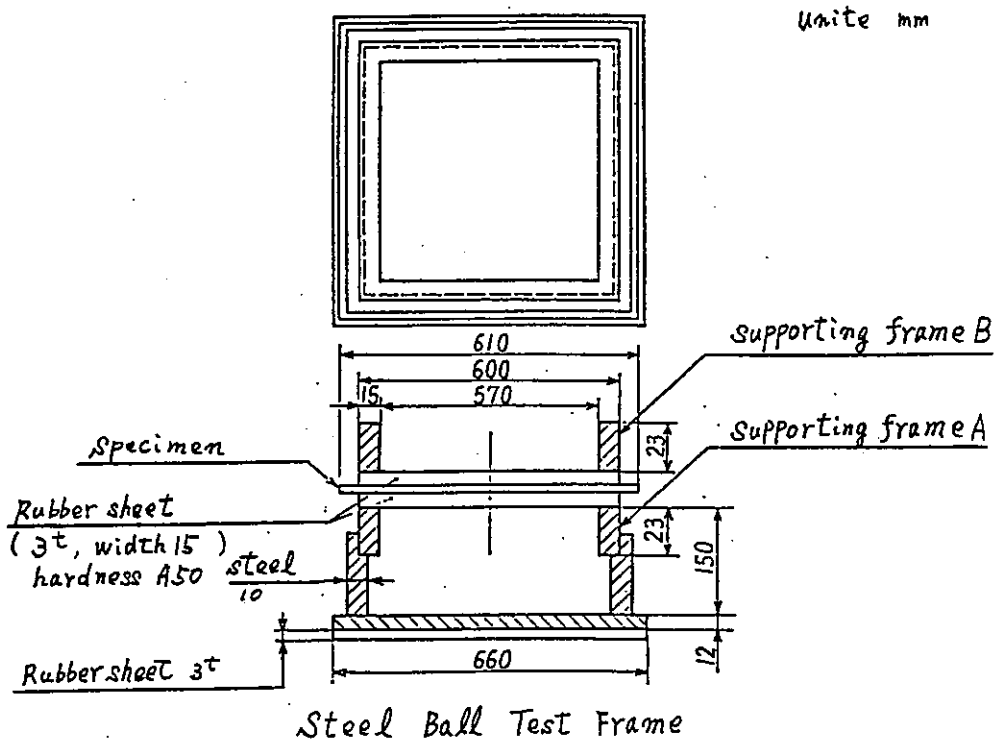
Calibration:

Sampling:

sample size   610mm X 610mm

Conditioning:

At room temperature

Procedures:

    1. Set sample on the supporting frame A and set supporting frame

       B on it to fix the sample.

    2. Drop a spherical steel ball (63.5mm diameter, 1040g) freely on

       the center of sample from certain height.

    3. When a sample is not broken change the height up words and

       repeat 2. until sample is broken.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

Date: 8/20/86   Reporter's Name: C. Hauer

Testing Title: Tensile properties of Plastics

Abstracted Scope:

The test method described covers the determination of the tensile properties of plastics in when tested under defined conditions of pretreatment, temperature, humidity, and tensile test strain rate. The samples shall be conditioned and selected as prescribed in the approppriate ASTM Standard. This method is applicable for samples of any thickness up to 14 mm. However, methods described in D 882 are preferred for films.

Referred Document: ASTM D 638-84

The following ASTM Standards are referenced:
 D 618 Methods for Conditioning Plastics and Electrical Insulating Materials for Testing
 D 882 Test Methods for Tensile Properties of Thin Plastic Sheeting
 E 4 Method of Load Verification of Testing Machines

Principle of Measurement:

The principle of measurement of tensile properties is is the uniform and constant rate increase of stress on a sample to determine the stress/strain characteristics, the yield point, and the point of failure. From these data one may obtain the modulus of elasticity and the yield strength.

Apparatus:

The apparatus required is a tensile testing machine capable of applying increased stress to a sample at a constant rate and equipped with a fixed and a movable gripper to hold the sample without slippage. In addition, it is necessary to use an extension indicator which measures the change of of distance between two fixed points (strain measurement). The tensile tester must meet the requirements of ASTM E 4 and the extensometer must meet the requirements of ASTM E 83.
Conditioning:

Calibration:

Calibration is to be performed per ASTM E 4 for the tensile tester and ASTM E 83 for the extensometer.

Sampling:

At least five test specimens are to be tested for isotropic samples, while at least ten samples, 5 normal to and 5 parallel with the axis of anisotropy, are to be tested for the anisotropic case.

Conditioning:

Samples shall be conditioned in accordance with ASTM Standard D 618.

Procedures:

Test specimens should be prepared in accordance with a "dumbbell" shape or other appropriate shape as required by the design of the tensile test apparatus, using condtioned material. A minimum of five samples should be used per test. The samples should be mounted in the tensile test apparatus. The extensometer is mounted on the sample. Measure the sample thickness and the width of the sample. Establish a stress rate to set a strain rate at 5mm per minute. Proceed with the testing of the samples.

Calculation Methods:

Calculate the tensile strength by dividing the maximum load by the original minimum cross-sectional area of the specimen to three significant figures. When the nominal break point load is less than the maximum load, calculate also the strength at yield and the percent elongation at yield. The modulus of elasticity is calculated from the load extension curve and is obtained by by dividing any load increment along this curve by the corresponding strain increment. It may be useful to estimate the standard deviation of the data points obtained from the samples.

Interpretation/Precision and Accuracy:

The sample sets, five or ten samples, in the isotropic or anistropic cases should yield consistent data and that data should be considered as representative of the material. The objective of this test is neither to duplicate the manufacturer's data or to make service life predictions. Rather, the objective of these tests is to gather information as to whether the materials chosen are adequate for the task.

REPORTING FORMAT

Date: 8/26/86   Reporter's Name: C. Hauer
Country: USA
Testing Title: Determining the Permanent Effect of Heat on
Plastics

Abstracted Scope:

This test procedure is intended to define the conditions of
temperature and time for which plastic sheet or laminated or
molded plastic changes physical properties.

Referred Document: ASTM D 794-82

The following ASTM Standards are referenced
 D 618 Methods for Conditioning Plastics and Electrical Insulating
Materials for Testing
 D 756 Practice for Determination of Weight and Shape Changes of
Plastics under Accelerated Service Conditions
 D 1870 Test Method for Elevated Temperature Aging Using a Tubular
Oven

Principle of Measurement:

Plastic materials exposed to heat may be subject to many types of physical and chemical changes. The severity of exposure, both in terms of temperature and time determines the extent of the changes and damage. While elevated temperatures may not necessarily degrade a plastic material, extended periods of exposure at elevated temperatures usually result in changed pysical properties. The principle of measurement in this test is the prolonged exposure of a sample to elevated temperatures and the qualitative assessment of its appearance in relation to its use as a cover material for solar collectors.

Apparatus:

The apparatus required is a temperature controlled oven employing forced draft air flow with provision for substantial fresh air intake. The sample rack should provide for good air circulation among samples. In addition, equipment for testing the specific physical property in question, say optical transmission, should be available. The oven should meet the requirements of ASTM E 145 and be capable of reaching temperatures of of 200 C.

Calibration:

The oven should meet the requirements of ASTM E 145 which is referenced.

Sampling:

While ASTM 794-82 does not specify sampling, the number of samples should consistent with those contained in other ASTM Standards relating to the physical property of the plastic under consideration. Generally, five samples are required.

Conditioning:

Samples should be conditioned in accordance with ASTM Standard D 618.

Procedures:

Two types of heat tests are of interest for solar collector covers, namely continous and cyclic. Thermal exposure tests should be conducted around the anticipated stagnation temperature for the collector and near the maximum recommended by the manufacturer. Continuous heat testing should start within 25 C of the maximum recommended by the manufacturer or 25 C above the anticipated stagnation temperature, if the stagnation temperature is lower. Should failure over a suitable time occur at the temperature first chosen, then repeat the test at a 25 C lower temperature. Conversely, if failure does not occur, increase the temperature by 25 C. Cycling tests should be run at the anticipated stagnation conditions, both temperature and cycle time.

Calculation Methods:

There are no explicit calculations to be made since the failure of the sample is measured either by visual inspection or specific tests for the physical property in question.

Interpretation/Precision and Accuracy:

No measures of precision or accuracy are ascribable to this test since failure is measured by visual inspection or the accuracy of a measurement, such as transmission, is dependent upon another test procedure.

Date: 8/29/86   Reporter's Name: C Hauer
Country: USA
Testing Title: Linear Dimensional Changes of Plastics Under
Accelerated Service Conditions

Abstracted Scope:

The test method is designed to provide a means for assessing
dimensional changes in plastics resulting from exposure to service
conditions, usually shrinkage or elongation.

Referred Document: ASTM D 1042-83

The following ASTM Standards are referenced:
 D 756 Practice for Determination of Weight and Shape Changes of
Plastics Under Accelerated Service Conditions

Principle of Measurement:

The principle of making the measurement consists of scribing the sample using a scribe 100 +/-.2 mm in length constructed from a stainless steel rod 125 mm in length with two needles mounted in holes drilled in the rod 100+/-.2 mm apart. The material is scribed at the outset of the test using one of the needles to make a center for the arc to be scribed and the other needle to scribe the arc. Upon exposure to accelerated exposure, a second arc is scribed from the same center and a microscope measurement to measure the differences in the arcs.

Apparatus:

The apparatus required is the 100 mm scribe described above and a measuring microscope such as the microscope used in a Brinell hardness tester. The microscope should resolve .01mm. Test specimens should measure 150 mm by 25 mm. A laboratory balance capable of resolving .5 mg should be available to measure water content changes.

Calibration:

ASTM D 1042-83 does not specify any calibration requirements.

Sampling:

Conditioning:

Samples should be conditioned in accordance with ASTM Standard D 618

Procedures:

The procedure for this test involves scribing a sample (150mm x 25mm) which has been conditioned with the scriber described under apparatus. The sample is then exposed to the accelerated service conditions, taking care that it is not confined linearly. Hanging the sample by a wire hook through a drilled hole is satisfactory. At each phase of the test where it is desired to measure linear changes, reinsert the scribe in the center and scribe another arc. Measure the distance between scribes with a microscope. The conditions for the accelerated service are those specified in ASTM D.756.

Calculation Methods:

No calculations are involved for the case where the initial and final arcs scribed are were made at the same temperature. Corrections for thermal are required if the arcs were scribed at different temperatiures. For the isothermal case the difference between arcs represents the linear dimensional change resulting from the test.

Interpretation/Precision and Accuracy

Measurements made at constant temperature (+/-1 C) and relative humidity (+/- 2%) are reproducible to +/-.02mm or .02%. The confidence level of this test is about 95%.

REPORTING FORMAT

Date: 8/30/86   Reporter's Name: C.Hauer
Country: USA
Testing Title: Tensile Properties of Thin Plastic Sheeting

Abstracted Scope:

The test procedures cover the determination of tensile properties
of thin plastic sheeting for film thickness less than 1.0 mm (0.04
in). Thin films are often used as covers for solar collectors.
While ASTM D 882-83 desciribes two test methods, this summary will
focus on the static weighting method which uses a constant rate of
grip separation. This method also results in the determination of
the modulus of elasticity.

Referred Document: ASTM D 882-83

The following ASTM Standards are referenced:
 D 618 Methods for Conditioning Plastics and Electrical Insulating
Materials for Testing
 D 638 Test Method for Tensile Properties of Plastics

Principle of Measurement:

The principle of measurement is the recording of strain in repsonse to the constant rate application of strain to samples prepared to take into account edge effects resuling from the "thinness" of the samples.

Apparatus:

A tensile test machine capable of operation in the constant strain rate mode is required. This tester shall be equipped with a weighing system that moves a maximum distance of 2% of the specimen extension within the range being measured. The motion of the grips shall be variable from 1.3 to 500 mm (.05 to 20 in.) per minute to attain the required strain rates. Thickness gage reading .0025 mm or less, (.0001 inch) and width measuring device capable of measuring .25 mm, (.01 inch) or less are also required. An extensometer is also needed.

Calibration:

ASTM 882-83 does not specify any calibration procedures. However, the tensile test machine should be calibrated in accordance with the manufacturer's procedures.

Sampling:

For thin plastic materials such as those used for covers, five conditioned samples will be required at minimum for isotropic materials and ten samples, five parallel and five normal to the axis of anisotropy, for anisotropic materials.

Conditioning:

Conditioning in accordance with ASTM Standard D 618 is required for the samples.

Procedures:
The test specimens will consist of strips of uniform width at least 50 mm longer than the grip separation used. Sample width shall be not less than 5 mm (.20 inches) or more than 25.4 mm (1 inch). A width to thickness ratio of at least 8 shall be used to reduce edge effects. For modulus of elasticity determinations the sample length shall be 250 mm.
Speed of Testing: The speed of testing shall be calculated from the required strain rates of .1 to .5 mm/mm (in./in.). The rate of grip separation may by obtained by multiplying the initial grip separation by the strain rate.
Measure the width and thickness of the sample carefully and at several points to the best accuracies which can be obtained by the micrometer and the width gage. Take care in mounting specimens to eliminate slip in grippers. Grip separation should be 50 mm for materials having an elongation of 100% or more at break and at 100 mm for materials with less than 100% elongation. Operate tester at selected load range and strain rate. Record load versus extension as measured by the extensometer.

Calculation Methods:

The nominal breaking factor shall be calculated by dividing the maximum load by the original minimum width of the sample. The nominal tensile strength shall be calculated by dividing the maximum load by the original minimum cross-sectional area of the sample. The standard deviation for the five or more samples per test shall be calculated by the usual method.

Interpretation/Precision and Accuracy:

Deviations of a sample set of 8% to 16% (standard deviation as a percent of the average value) can be expected.

35

REPORTS ON GLAZING

c (i) DURABILITY TO A STAGNATION TEMPERATURE

Date: December 1986   **Reporter's Name:** Michael G Hutchins

Country: United Kingdom

Testing Title: Outdoor exposure to stagnation conditions for
solar absorbers and solar glazings

Abstracted Scope (200 words):

A purpose built outdoor durability trials station located on the roof of
the Science building has been commissioned. The station is instrumented
to log solar irradiance (total), humidity, ambient temperature and
sample temperatures and these data are subsequently stored on tape and then
processed through a standard statistical package to yield daily average
values and bands of exposure conditions.

Solar absorber surfaces are mounted in purpose built boxes, which are single
glazed. Samples of 100mm x 100mm are normal and each box may take up to
40 samples. The boxes face due south and are inclined at 45° to the
horizontal. At periodic intervals samples are removed to the laboratory
where routine tests to check surface optical properties and composition
are undertaken prior to returning them for further exposure.

Referred Document:

ASTM E781 Evaluating absorptive solar receiver materials when exposed to
conditions simulating in solar collectors with cover plates

Principle of Measurement:

The changes in surface optical properties, surface microstructure, and chemical composition are measured and attempts are made to correlate such changes with surface temperatures and solar irradiance experienced during the period of outdoor exposure.

Apparatus:

Purpose built Unistrut support frame:
0.75m x 0.75m purpose built wooden framed, single glazed durability boxes with vent holes and 75mm glass fibre rear insulation,
Kipp-Zonen CM5 pyranometer;
K-type thermocouples;
Christie 248 24 channel datalogger with integral read-write tape facility;
BBC Model B microcomputer with link to PRIME mainframe for data processing.

Calibration: Various

Sampling:

Samples are monitored at one month after initial exposure and thereafter at six monthly periods

Conditioning:

None

Procedures:

1. Samples are cut to size
2. An initial characterisation which involves measurement of solar absorptance and thermal emittance, examination of surface microstructure and chemical composition using electron microscopy, x-ray diffraction, x-ray microanalysis and ion beam analysis is carried out. Standard size of a set of samples is 12.
3. Samples are mounted in durability boxes.
4. On return to laboratory surface optical properties are monitored. If no deleterious changes are observed samples are returned to the trials station. If significant changes are observed more detailed compositional and microstructural examinations are performed.
5. Records of solar irradiance and surface temperatures are compiled on a daily basis from tapes which are read once per week. Frequency of sampling is at 15 minute intervals.

Calculation Method (if necessary, please include correction method):

None

Interpretation/Precision & Accuracy:

Difficult to quantify at this stage

39

REPORTS· ON GLAZING

c (ii)   DURABILITY TO A DESIGNATED

CONSTANT TEMPERATURE

Date: 9/3/86   Reporter's Name: C. Hauer
Country: USA
Testing   Title: Linear   Dimensional   Changes   of   Nonrigid
Thermoplastic Sheeting or film at Elevated Temperature

Abstracted Scope:

Test Method ASTM D 1204-84 covers the measurement of changes in
linear dimensions for thermoplastic sheeting or film resulting
from exposure to specified conditions of temperature and time.

Referred Document: ASTM D 1204-84
The following additional ASTM Standards are referenced:
 D 618 Methods for Conditioning Plastics and Electrical Insulating
Materials for Testing
 D 2732 Test Method for Unrestrained Linear Thermal Shrinkage of
Plastic Film and Sheeting

Principle of Measurement:

The test is applicable to plastic sheeting or film made by the calender or extrusion process and indicates lot to lot unformity with regard to internal strains. The principle of measurement relies on the determination of a dimension before and after annealing.

Apparatus:

The following equipment is required:
* Convection oven capable of maintaininga temperature of 100 C +/- 1 C
* Scale graduated in .25 mm (.01 in.) divisions and more than 30 mm (12 in) long.
* Thermometer graduated in 1 C divisions with 100 C range.
* Timer graduated in minutes
* Template 25x25 cm (10x10 in.) for cutting test specimens
* Heavy paper sheets. about 40x40 cm.
* Talcum powder

Calibration:

No calibration requirements are specified in ASTM D 1204-84.

Sampling:

Test specimens shall be two pieces of sheeting or film cut using the stencil from a sample, one from either of two transverse edges and one from the center of the sample sheet. Each 25x25 cm specimen shall be marked to show the direction of calendering or extrusion.

Conditioning:

Samples are to be conditioned in accordance with ASTM Standard D 618.

Procedures:

The midpoint of each edge of the specimen shall be marked as a reference for final measurements. Test measurements shall be made at 23 +/-2 C and 50+/- 5% relative humidity. Each specimen shall be placed between two sheets of heavy paper each of which have been well dusted with talcum powder and held together with clips. The paper/plastic sandwich is to be placed in the oven at the temperature and for the time applicable to the material. At the end of the oven exposure period, the specimens are to be reconditioned for at least one hour at the ambient conditions cited above. Finally, measure the specimens at the reference marks to the nearest .2 mm.

Calculation Method:

Calculate the percentage of linear change as follows:
% Change = $[(D_0-D_1)/D_1]\times100$
where D_0 and D_1 are the initial and final distances at the reference marks.

Interpretation/Precision and Accuracy:

No precision or accuracy interpretation for this test has been established by ASTM.

REPORTS ON GLAZING

c (iii) DURABILITY TO THE EXPOSURE TO SOLAR
RADIATION

Designation :

__GS-D -ST__

Date: 3/3/86

Country: Japan

Reporter's Name: Katsuhiko Hojo

Testing Title:

Standard Method of Test for Change in Properities of Plastics Building Materials Resulting from Outdoor Exposure.

Abstracted Scope (200 words):

(1) This standard specifies the mothod of assessment for the performance of test piece that has been exposed outdoor to conjecture the degree of weathring of plastics building materials.

(2) This standard can apply, as appropiate, to the method of assessment for the performance of plastics building materials that have been subjected to artificially accelerated exposure.

Referred Document:

JIS A 1411
"Standard Method of Test for Change in Properties of Plastics Building Materials Resulting from Outdoor Exposure"

15

Principle of Measurement:

The change in the test piece before and after the exposure test shall be expressed with the test piece defference.-Ex. Shape Change percentage, Dimesional Change percentage, Change in Tensile strength and Elongation Percentage.

Apparatus:

(Caluculation Method is shown in Annex Page, P.4)

Calibration:

Sampling:
(1) The shape and dimensions of test piece shall have sufficient dimensions as a test sample for the assessment of performance.

(2) When the test piece for the assessment test is taken out of an exposed big test piece, the end part of it shall be avoided.

Conditioning:

Wash the test piece with water, as requied, before the performance test.
In this case, cares shall be taken to prevent the test piece from damage by using clean water.

Procedures :

(1) Wash the test piece with the water.

(2) Ajust the test piece under the specified condition, as requred.

(3) Measuring and Recording.

(4) Assessment for the performance.

Calculation Method (if necessary, please include correction method) :

Interpretation/Precision & Accuracy :

ANNEX

Calculation Method

(1) The dimensional change percentage shall be obtained from the following formula:

$$\text{Dimensional (length, width, thickness) change percent (\%)} = \frac{\text{Dimension after exposure} - \text{Dimension before exposure}}{\text{Dimension before exposure}} \times 100$$

Remark: $(+)$ dimension change percentage indicates expansion,
$(-)$ dimension change percentage indicates contraction,

(2) The measurement of glossiness shall be carried out as specified in the method 3 of 5.2 in JIS Z 8741. The change of glossiness after the exposure test shall be expressed by the survival rate of glossiness and obtained from the following formula:

$$\text{Survival rate of glossiness (\%)} = \frac{\text{Value of Gs(45) after exposure test}}{\text{Value of Gs(45) before exposure test}} \times 100$$

(3) Change in Tensile Strength and Elongation Percentage
The tensile test shall be carried out as specified in 5.18.3 of JIS K 6911 and the tensile strength and elongation percentage shall be obtained. However, the elongation percentage shall be calculated from the following formula:

$$\text{Elongation percent (\%)} = \frac{\text{Gauge length at rupture} - \text{Original gauge length}}{(\text{Original gauge length})} \times 100$$

Date: 15th July, 1987   Reporter's Name: K. Hayakawa

Country: Government Industrial Research Institute, Nagoya
          Hirate-cho, Kita-ku, Nagoya, Japan
Testing Title:

Aging Test on Materials for Transparent Covers of Collectors

Abstracted Scope (200 words):

This standard provides the conditions for the aging test of materials used as solar collector covers. It defines the aging procedure and qualification tests to be performed before and after aging. The test applies to the transparent covers for flat plate solar collectors made of plastics or of especially coated glass.

Referred Document:

ISO Standard Draft Proposal (ISO/DP, ISO/IC, 180/WG 2 N 1 E)

Principle of Measurement:

The material is submitted to a natural aging. An additional aging test including artificial aging procedure may be performed jointly as a comparison.

Apparatus:
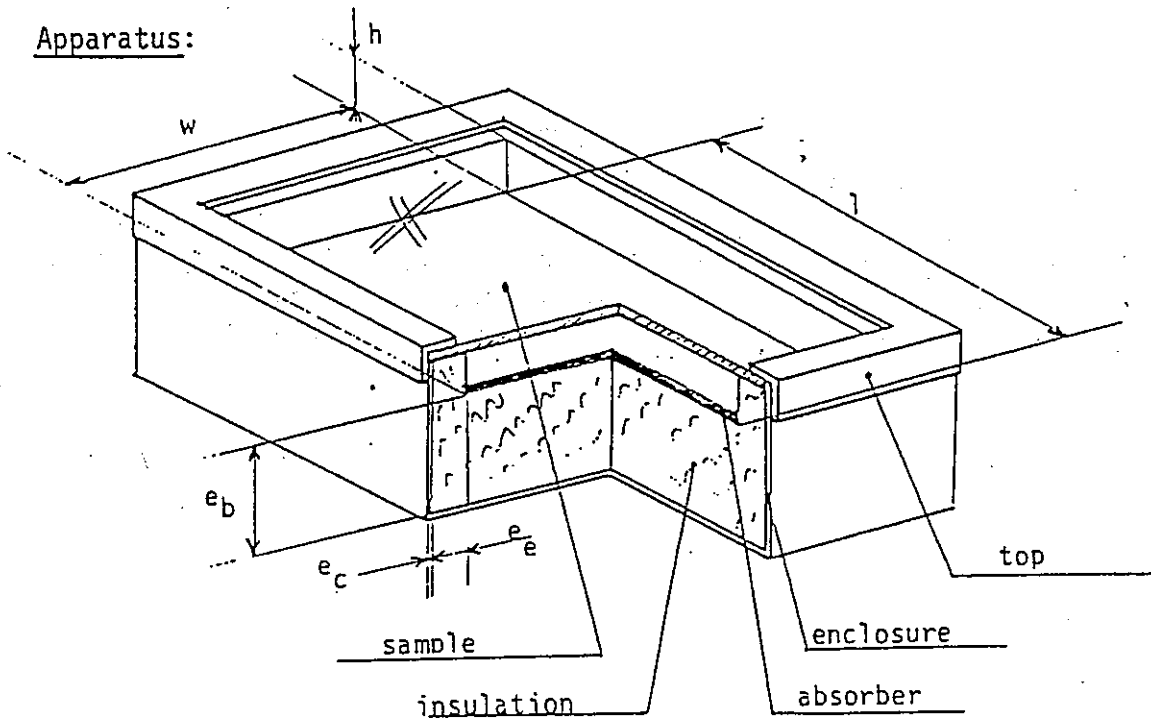
Schema of a natural weathering box for solar collector transparent cover sample.

Calibration:

Sampling:

Rigid plastic sheets, with apparent thickness of 1 mm or more.
Thin plastic films with a thickness under 1 mm.
Glasses with special coating.

Conditioning:

They must withstand a temperature of 150 °C and they will be heated in oven 24 hours at this temperature before use, to minimize outgassing risks.

Procedures:

The samples are fixed in boxes simulating small solar collectors in stagnation (see Figure). These boxes, with insulated enclosure and absorbing plate receive the sample in place of transparent cover, and are exposed to natural environment during one year.

The boxes will be exposed by trying to get the maximum solar irradiance during the year, and by using variable tilt angles according to the season, with not less than three different positions: (latitude-16) ±2.5; latitude ±2.5; (latitude+16) ±2.5.

Calculation Method (if necessary, please include correction method):

The visual inspection will be made at least monthly. The change will be checked and reported as a part of the test-results.

1) Measurement of solar normal hemispherical transmission factor.

2) Mechanical characteristics according to ISO R 527-1966 and ISO 1184-1983.

Interpretation/Precision & Accuracy:

REPORTS ON GLAZING

c (iv) DURABILITY TO THERMAL SHOCK

Designation :

REPORTING FORMAT

GS-D-LA

Date : Feb. 27 1986   Reporter's Name : Sadamitsu Tsuboi

Country : Japan

Testing Title : Testing Method of Durability to Thermal Shock for Sheet Glass.

Abstracted Scope (200 words) :

This test is applied to transparent sheet glass and it's application products.

The Transparent sheet glass includes Float glass and etc.

Application products include Laminated glass and Thermal insulation (double glazed) glass.

Referred Document :

J Nucl Mater (NLD) 111/112 852 855 ('82)

53

Principle of Measurement:

Quick temperature changes give samples critical thermal stress.

Apparatus:

Thermal shock testing machine DTS-40 (Futaba Kagaku Co., LTD.)
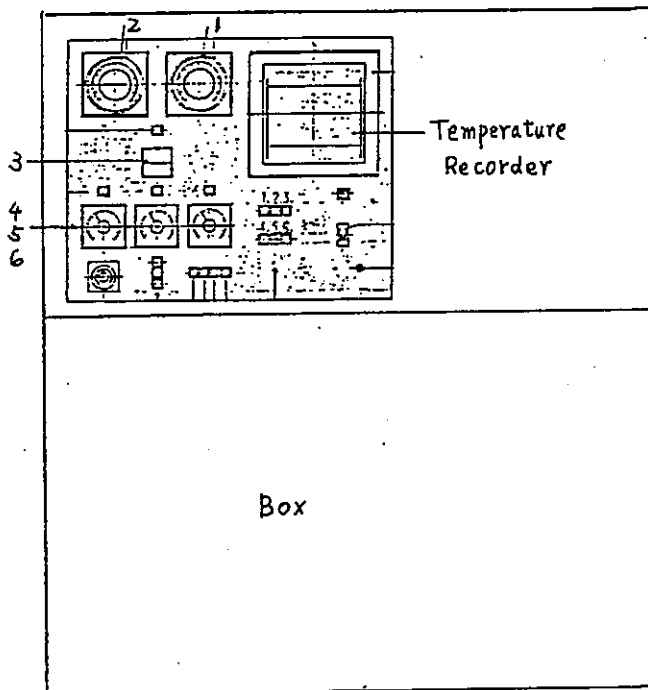
1 Temperature controller (High)

2 Temperature controller (Low)

3 Digital counter

4 Timer (High)

5 Timer (Intermediate)

6 Timer (Low)

Sampling:

Maximum sample size   400mm X 300mm X 300mm

Conditioning:

High temperature $60 \sim 250°C$

Intermediate temperature   room temperature

Low temperature $-10 \sim -70°C$

35

Procedures:

1. Set samples on the holder

2. Set high temperature

3. Set low temperature

4. Push high and low chamber's operation buttons

5. Set timers (three chambers) after temperature reached setting values

6. Set cycle number

7. Start the test

8. Samples are moved into High temperature chamber

9. After setting time elapsed samples are removed to Intermediate temperature chamber and after that samples are moved to next chamber as scheduled

10. After completed setting cycles the test ends

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy :

REPORTS ON GLAZING

c (ix) DURABILITY TO MIXED ENVIRONMENTAL CONDITIONS
SUCH AS ELEVATED TEMPERATURE, SOLAR RADIATION
MOISTURE AND ETC.

Designation :

REPORTING FORMAT <u>GS-D-ST</u>

Date: 2/28/86   Reporter's Name: Katsuhiko Hojo

Country: Japan

Testing Title: Recommended Practice for Outdoor Exposure of
Plastics Building Materials

Abstracted Scope (200 words):

(1) This Standard specifies the recommended practice for
outdoor exposure of the test piece to conjecture the
degree of weathering of the plastics materials.

(2) This test is defined as the test to examine the change in
the performance b exposing the test piece outdoors under
natural conditions.

Referred Document:

JIS A 1410
"Recommended Practice for Outdoor Exposure of Prastics
Building Materials"

Principle of Measurement:

The Test for outdoor exposure is defined as the test to examine the change in the performance by exposing the test piece outdoor under natural conditions.

Apparatus:

(1) The exposure apparatus to expose the test piece shall be one that is constructed strongly and durably and placed firmly on the ground and others.

(2) Where the antiseptic treatment or the anticorrosive treatment is conducted on the exposure apparatus and its accessory appliances, take care not to exert an harmful effect on the test piece.

(noto)
The exposure surface of test piece shall face the correct south (the directional error is within two degrees) and be inclined at 30 to 35 to the holizontal in principle.
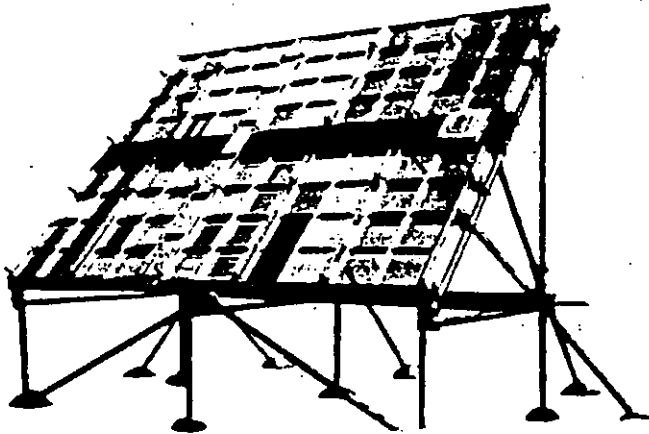
Calibration: Photograph shows an example of the exposure apparatus

Sampling:

Those stages shall be decided as agreed between the parties concerned. (n: the year elapsed)
For the first year, at 1, 2, 3, 4, 6, 9, 12, months.
For the second year and thereafter, at n year and 3, 6, 9, 12, months.

Conditioning:

(1) The weathering is cause as the time elapes on by the influence of the natural conditions such as sunlight, wind, and rain outdoor.

(2) The test piece shall be carried out in the dark room or lightproof container having at least the ordinary temperature and ordinary humidity.

355

Procedures :

(1) The test piece shall be fixed without backing, in principle.
(2) The duration of exposure of the test piece shall be as specified in the Japanese Industrial Standard of the material concerned.-shall be not less than one year.
(3) Not be careless of the state of test in the duration of exposure of the test piece.
(4) The test items of performance test shall be as specified in the Japanese Industrial Standard of the material concerned. -but not specified in JIS, the following shall be applied as the standard。
  (a) Change in shape and dimensions.
  (b) Change in appearance.
  (c) Tensile strength, and elongation percentage.

Calculation Method (if necessary, please include correction method) :

The performance test shall be as specified in JIS A 1411.

(1) Shape Change Percentage.(specified in JIS K 6911)
(2) Dimensional Change Percentage.(specified in JIS K 6911)
(3) Change in Appearance.(Change in Color, Light Transmittance, Survival Rate of Glossiness, etc)
(4) Change in Tensile Strength and Elongation Percentage.

Interpretation/Precision & Accuracy :

REPORTING FORMAT.

Date:	Reporter's name: Nico van Santen

Country: Technisch Physische Dienst TNO-TH
(TNO Institute of Applied Physics)
P.O. Box 155, 2600 AD DELFT
The Netherlands

Testing title: DURABILITY ASPECTS OF ORGANIC SEALED INSULATING GLASS

Abstracted Scope (200 words):

The durability of dual organic sealed insulating glass is discussed: the agencies affecting durability, the restrictions of weathering tests in a climate chamber and the necessity of supplementary accelerated ageing tests in a special weatherometer with a xenon arc lamp to simulate real UV radiation by the sun. Ageing characteristics of the outer seal prove to be decisive for durabilitiy.

KEYWORDS
Durability, insulating glass, weathering tests, dual seal.

Referred document:

N. van Santen, 'Durability aspects of organic sealed insulating glass', Proc. Windows in building design and maintenance, Göteborg, Sweden, 13-15 June 1984, blz. 329 ... 334.

Principle of measurement:

Variable climate tests are the common weathering tests on a laboratory scale to establish the quality and durability of insulating glass. There are different types of climate tests which have in common that a number of test specimens are exposed to fluctuating air temperatures and high humidity. The fluctuating air temperatures affect mechanical stresses on the glass edges and mechanical degradation of the edge seal due to the thermal expansion of the enclosed gas. The high humidity affect water vapour transmission.
Variable climate tests do not affect real ageing of materials except for fatigue effects and interactions with water. Real ageing can only be affected by natural or simulated sun radiation. Some use fluorescent UV lamps or Hg sunlamps, however, best results are achieved by outdoor exposure or at laboratory scale by exposure to the radiation of a xenon arc lamp with rain simulation like ASTM G-53. The spectrum of the xenon arc lamp is very similar to that of the sun.

Apparatus:

The climate chamber need no further explanation. The special weatherometer for insulating glass is described in another paper.

Calibration:

Sampling:

Standard size samples of 360 x 510 mm, for which temperature variation has maximum mechanical impact on the edge seal are chosen.
The same sample size is used for the weatherometer, where sun radiation and mechanical stresses due to temperature variation act at the same time as they do in reality.

Conditioning:

Climate chamber:
Temperature cycling from -10 ° to +55 °C, RH > 95% (55 °C), 3 cycles a day, exposure period over 300 days.

Weatherometer:
Irradiance 1000 W/m^2, temperatures 20 - 60 °C, periodic water spraying and drying, exposure time 2000 hours.

Procedures:

The water vapour penetration through the edges of insulating glass during the variable climate tests is measured with the aid of exangeable capsules with desiccant with high accuracy.
Main criterium of the weatherometer tests is the degradation of the sealants and the adherance of the sealants to glass.
Different double barreer organic sealed insulating glass systems has been tested, with traditional and new outer sealants.

Calculation method (if necessary, please include correction method):

Life time can be calculated by comparing the water vapour transmission during the tests with the maximum water adsorbtion capacity within the unit, taking into account the real climate conditions (temperature and humidity).

In formule:
Water vapour penetration Q
Water adsorbtion capacity $Q_A = M (W_c - W_A)$
(M = mass desiccant in the unit
W_c = specific water adsorbtion capacity desiccant
W_a = initial water capacity desiccant).

Life time:

$$L_t = \frac{Q_A}{Q} \cdot A_T \cdot A_p$$

where:
A_T = acceleration water vapour diffusion due to higher testing temperatures as compared to practice.
A_p = acceleration water vapour diffusion due to higher water vapour pressures during testing as compared to practice.

As to the weatherometer test, the real time acquivalent is determined by comparing the amount of radiation reaching the sealants in practice under glazing conditions and the same during tests.

Interpretation/Precision & Accuracy:

The main conclusion is that the durability of insulating glass, if minimum
requirements as to water vapour resistance of the edge seal are fulfilled,
mainly depends on the resistance of the outer seal, especially of the adherence
to glass, to detioration by sunradiation.

63

REPORTING FORMAT

Date:	Reporter's name: J.G.M. Mangé

Country: Technisch Physische Dienst TNO-TH
(TNO Institute of Applied Physics)
P.O. Box 155, 2600 AD DELFT
The Netherlands

Testing title: WEATHEROMETER AGEING TEST

Abstracted Scope (200 words):

A special weatherometer is built for testing insulating glass. The test gives information on the resistance of the edge seal to ageing when exposed to sunlight, high temperature and water, especially concerning the adhesion to glass. The amount of irradiation equals about 1 year direct outdoor exposure or some years under glazing conditions.

Referred document:

NEN 3567, 1e druk, augustus 1985. Nederlands Normalisatie Instituut.
'Glass in building -insulating glass- requirements and methods of test'.

364

Principle of measurement:

Complete double glazing units are exposed to artificial sunlight, high temperature and water.
Before and after ageing the bonding strength of the edge seal is tested by elongating samples taken from the edge.

Apparatus:

The weatherometer uses a xenon-arc lamp as its light source.
The bonding strength of the edge seal is tested with a Chatillon UTSE Bench Test Stand.
The irradiance of the xenon-arc lamp is controlled at a level of 1000 W/m^2 at a distance of 500 mm. The elongation rate of the bench tester is controlled at 10 mm/min.

Calibration:

The irradiance of the xenon-arc lamp is controlled at a level of 1000 W/m^2 at a distance of 500 mm. The elongation rate of the bench tester is controlled at 10 mm/min.

Sampling:

The test units used for ageing are 510 x 360 mm. Samples of the edge seal of the test units for measuring the bonding strength are taken by using a diamant saw.
The samples have to be 50 mm of length and 15 mm of width.

Conditioning:

Before ageing the test units are conditioned during 1 week at room conditions.
The test units are radially positioned around the Xe-lamp in the weatherometer with one side at a distance of 500 mm.
The following cycle schedule is used:
- light exposure only 1/2 h.
- light exposure and water spray 7 1/2 h.
- light exposure only 1 h.
- rest 3 h.
  Total cycle time: 12 h.
The air temperature in the test chamber is controlled at a level of 55 °C ± 3 °C. The integrated exposure time is 1000 hours, equivalent to 3,6 GJ/m^2 irradiation.

Procedures:

- After reception of the test units, these units are conditioned at room temperature during 1 week.
- The units ment for ageing are placed in the weatherometer after this conditioning, the units ment for comparison stay conditioned at room temperature.
- After 1000 lamp burning hours (approximately 8 weeks) the aged units are taken out of the weatherometer.
- From both aged and not-aged units samples are taken from the edge seal.
- The samples are elongated in the bench tester while registering force and elongation.
- The force-elongation diagrams from aged and not-aged samples are compared and the edge seal is visually checked for discolouration.

Calculation method (if necessary, please include correction method):

The 1000 hours integrated exposure time equals 3,6 GJ/m^2 irradiation.
This amount of irradiation equals about 1 year outdoor exposure in Holland.

Interpretation/Precision & Accuracy:

The force-elongation diagrams from aged and not-aged samples are compared and the edge seal is visually checked for discolouration.
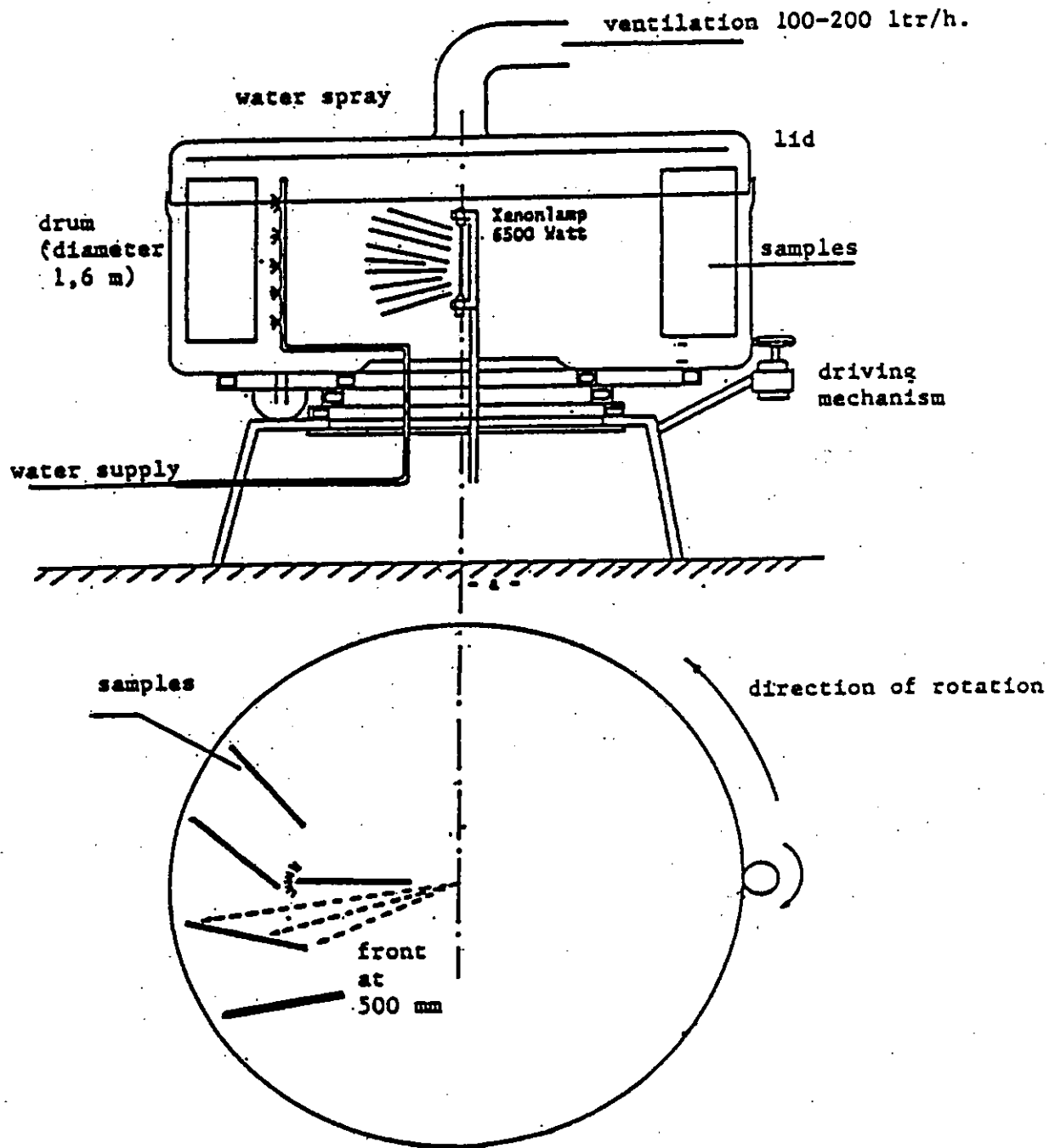
Figure   Weather-o-meter for ageing tests on insulating glass units.

REPORTING FORMAT

Designation:
_GS-D-LA_

Date: 8/8/86   Reporter's Name: C. Hauer
Country: USA
Testing Title: Evaluation the Degradation of Cover Materials for
Flat Plate Solar Collectors

Abstracted Scope:
This test procedure for solar collector cover materials includes
primary property measurement tests both before and after exposure
to aging. Both the primary property measurements and the aging
procedures are described. Property test methods include
measurement of solar transmittance, dimensional stability, impact
resistance, tensile strength, and the effect of dirt retention on
solar transmittance. Aging procedures include the exposure to
heat, natural weathering, and accelerated weathering.

Referred Document: ASTM E 765-80

In addition the following ASTM documents are applicable:
D 618 Conditioning of Plastics and Electrical Materials for
Testing
D 638 Test for Tensile Properties of Plastics
D 794 Recommended Practice for Determining the Permanent Effect
of Heat on Plastics
D 882 Tests for Tensile Properties of Thin Plastic Sheeting
D 1042 Tests for Linear Dimensional Changes of Plastics Under
Acccelerated Service Conditions
D 1204 Test for Linear Dimensional Changes of Nonrigid
Thermoplastic Sheeting or Film at Elevated Temperature
E 903-82 Test Method for Solar Absorptance, Reflectance, and
Transmittance of Materials Using Integrating Spheres
E 782 Standard Practice for Exposure of Cover Materials for Solar
Collectors to Natural Weathering under Conditions Simulating
Operational Mode
ANSI Standard Z97.1-1975 Safety Performance Specifications and
Methods of Test for Safety Glazing Materials Used in Buildings

Principle of Measurement:
 Solar collector covers materials are exposed to tensile, solar
transmission, impact, thermal aging, and dimensional stability
tests both prior to and subsequent to accelerated exposure
processes. The accelerated aging procedures involve exposure to
both concentrated outdoor solar and indoor laboratory exposure
tests.

Apparatus:
The apparatus required includes an oven capable of maintaining
specimens at 125 C , tensile test apparatus, xenon arc light
sources, and outdoor solar collector test fixtures with solar
energy enhanced through the use of Fresnel reflectors. The
equipment is detailed in the referenced ASTM specifications, for
example xenon arc source exposure per ASTM G 26.

Calibration: Calibration of equipment is in accordance with the
ASTM Standard used.

Sampling:
For indoor laboratory tests five sets of test specimens will be
used. For the outdoor exposure two sets of specimens will be
used.

Conditioning:
Conditioning of materials is to be carried out in accordance with
ASTM standard D 618, Condtioning of Plastics and Electrical
Materials for Testing.

Procedures:
The test procedures include solar transmittance measurements, the determination of physical properties such as tensile strength, dimensional stability, and warpege for the plastic cover material, as well as impact resistance to hail and other objects. These tests are carried out per the appropriate referenced ASTM standard procedure before and after sample exposure to aging procedures.
Transmittance tests are per ASTM E 903 where Method A uses a spectrophotometer with an integrating sphere and Method B uses a pyranometer. Temperature aging procedures specify the exposure in an oven at 75 C for exterior covers and 125 C for interior covers for a period of 240 hours. For natural weathering, the cover materials are to be exposed for 12 months in accordance with ASTM Practice E782. After weathering exposure, the effect of dirt retention may be assessed by repeating the transmittance test both before and after cleaning. Accelerated weathering is a screening procedure, but no sustitute for the tests in ASTM E782. This procedure includes light exposure using xenon arc illumination and a prescribed routine for moisture exposure (700, 1400 and 2400 hours of exposure). The moisture cycle shall be 90 minutes of light followed by 30 minutes of light and water spray.

Outdoor accelerated exposure shall use concentrated natural sunlight using Fresnel reflectors, per ASTM E838. The test specimens shall be exposed to a total incident radiant solar energy of 4GJ, 8GJ. and 16GJ, see ASTM E903

Calculation Methods:
This test procedure in the main does not rely on calaculations for assessment. Rather it is designed to obtain qualitative performance data of solar collector cover materials under actual and simulated operating conditions.

Intepretation/Precision and Accuracy:
The interpretation of the test data are largely qualitative in the sense "is the cover material suitable for the service intended". Thus, lack of dimensional stability, appearance of crazing or cloudiness which reduces transmittance are some of the qualitative measures of whether the sample passes or fails the tests. It should be emphasized that these tests are screening methods and that no explicit service life predictions can be made or are intended.

Designation:
G$S-D-LA

Date: 8/18/86   Reporters Name: C. Hauer
Country: USA
Testing Title: Conditioning Plastics for Testing

Abstracted Scope:

In general, the physical properties of plastics are influenced by
temperature and relative humidity in a manner that materiallly
affects test results. So that reliable comparisons can be made
between materials and test laboratory results, it is necessary to
standardize humidity and temperature conditions to which specimens
are subjected prior to and during test.

Referred Document: ASTM D 618

The following ASTM standards are referenced:
    D 709 Specification for Laminated Thermosetting materials
    D 756 Practice for Determination of Weight and Shape   Changes
of Plastics Under Acccelerated Service Conditions,

Principle of Measurement:

The principles involved in the conditioning of plastics prior to testing are:
* to bring the material into equilibrium with normal room conditions
* to obtain reproducible results regardless of previous history of exposure
* to subject the material to normal or abnormal conditions of temperature and humidity in order to predict service behaviour.

Apparatus:

In specifying the conditioning process, three parameters are defined, namely time, temperature, and relative humidity. The time for specimens under 7mm (.25") thickness will be 40 hours; the time forspecimens over 7mm will be 88 hours.The apparatus used will be an air circulation oven equipped with temperature and humidity controls which maintains the requisite temperature and relative humidity conditions, say room temperature, 23 C, and 50% relative humidity.

Calibration:

ASTM 618 does not specify calibration of the oven. Oven manufacturer's procedures should be followed.

Sampling:

ASTM 618 does specify sampling, but sample selection should conform to the requirements of the ASTM test procedure for which the samples are being conditioned.

Conditioning:

The conditions for the "conditioning" as applicable to solar cover materials should reflect the service operating conditions.

Procedures:

Place samples to be conditioned in a temperature and relative
humidity controlled oven, insuring that surfaces are exposed to
adequate air circulation. Set appropriate temperature and relative
humidity conditions which reflect sercice conditions. Expose
samples for 40 or 88 hours as appropriate. Use samples in other
test procedures preferably within 1/2 hour of removal from
conditioning.

Calculation Methods:

No calculation procedures are involved in the conditioning
process.

Interpretation/Precision and Accuracy:

This test procedure is related to sample preparation. The results
are a qualitative simulation of the operating environment intended
to bring the sample to a condtion similar to that resulting from
installation on a system.

REPORTING FORMAT

Date: 9/4/86   Reporter's Name: C. Hauer
Country: USA
Testing Title: Exposure of Cover Materials for Solar Collectors to
Natural Weathering under Conditions Simulating the Operational
Mode

Abstracted Scope:

ASTM E 782-81 provides a test procedure for the exposure of
collector covers to the natural weather environment at
temperatures that approximate operational conditions in a test
fixture. This practice is suitable for glass and plastics as well
as double glaze configurations, but does not apply to evacuated
collectors or photovoltaics.

Referred Document: ASTM E 782-81
The following ASTM Standards apply:
D 1435 Recommended Practice for Outdoor Weathering of Plastics
E 765 Practice for Evaluation of Cover Materials for Flat Plate
Solar Collectors

Principle of Measurement:

The principle of testing involved in this procedure is the construction of a prototype flat plate collector module into which cover materials can be inserted. This module is oriented toward the sun on an east-west axis and is capable of heating glazings to temperatures representative of the operational mode in a solar collector.

Apparatus:

The apparatus required is a test fixture which duplicates a flat plate collector but is not insulated. The absorber surface should have an absorptivity of at least .9. Spacing between the absorber surface and the first cover is about 25mm (1 inch). This spacing also applies between covers. Test specimens shall be of a size adequate to cover the aperture of the module. This test module may be used for films as well as sheet cover materials.

Calibration:

ASTM E 782-81 does not specify a calibration methodology.

Sampling:

ASTM E 782-81 does not specify a sampling methodology.

Conditioning:

No prior conditioning of the test materials is required.

Procedures:

The test materials are mounted on the weathering box test module in either the single or double glazing configuration, with each test sample identified as to its position. The test module is mounted on a variable tilt rack with the tilt axis in an east-west direction. Because the tests are long term, a viewing angle adjustment schedule must be developed to insure that the module receives maximum insolation. A quarterly adjustment is sufficient, ranging from latitude to +/- 20 degrees of latitude. Monthly adjustments are preferred. Because the test results are only visual observations, an inspection schedule must also be developed. It is noted that the glazing surfaces are not to be cleaned during the test period. The data to be noted are:
    * test location, latitude, longitude, and climate description
    * calendar dates of exposure and angle adjustment schedule
    * Climatological data
    * Radiant energy incident on the module and maximum monthly temperaure.
    * Description of samples and periodically observed changes
As an option, climatological data, wetness in the collector module, and incident energy may be collected on a daily basis. Transmittance measurements should be made on the samples before and after testing.

Calculation Methods:
Since the test results are visual observations, no calculational procedures are required for the sample. It is useful to estimate the total insolation for the module over the test period.

Interpretation/ Precision and Accuracy:
Because this test is based upon visual observations, no comments on accuracy or precision are appropriate.

REPORTING FORMAT

Date: 9/4/86   Reporter's Name: C. Hauer
Country: USA
Testing Title: Exposure of Solar Collector Cover Materials to
Natural Weathering under Conditions Simulating Stagnation Mode

Abstracted Scope:

ASTM E 881-82 provides a test procedure for the exposure of
collector covers to the natural weather environment at
temperatures that approximate flow stagnation conditions in a test
fixture having a combined back and edge loss coefficient of less
than 1.5W/(square meter:C). This practice is suitable for glass
and plastics as well as double glaze configurations.

Referred Document: ASTM 881-82

The following additional ASTM Standards are referenced:
D 1435 Recommended Practice for Outdoor Weathering of Plastics
E 765 Practice for Evaluation of Cover Materials for Flat Plate
Solar Collectors
E 782 Practice for Exposure of Cover Materials for Solar
Collectors to Natural Weathering Under Conditions Simulating
Operational Mode
G 7 Recommended Practice for Atmospheric Environmental Exposure
Testing of Non-metallic Materials

Principle of Measurement:

The principle of testing involved in this procedure is the
construction of a prototype flat plate collector which meets the
heat loss standard cited above and the operation of this collector
for a long period of time in the stagnation mode. The collector
module is oriented towards the sun and exposes the glazings
mounted on it to temperatures representative of stagnation
conditions in a flat plate solar collector.

Apparatus:

The apparatus required is a test fixture which duplicates a well
designed flat plate collector. Any good design is acceptable
providing that the heat loss constraint of 1.5W/(square meter C)
is met. The absorber surface should have an absorptivity of at
least .9. Spacing between the absorber surface and the first cover
should be between 13 and 38 mm (.5 to 1.5 inches). This range of
spacing also applies between covers. Test specimens shall be of a
size adequate to cover the aperture of the module. This test
module may be used for films as well as sheet cover materials.

Calibration:

ASTM E 881 does not specify any calibration methodology.

Sampling:

ASTM E 881 does not specify a sampling methodology.

Conditioning:

No prior conditioning of the test materials is required.

Procedures:

The test materials are mounted on the weathering box test module (collector module) in either the single or double glazing configuration, with each test sample identified as to its position. The test module is mounted on a variable tilt rack with the tilt axis east-west. Because the tests are long term, a viewing angle adjustment schedule must be developed to insure that the module receives maximum insolation. A quarterly adjustment is suffficient although monthly is better. Adjustment is from latitude to latitude +/- 16 degrees. Because the test results are only visual observations, an inspection schedule must also be developed. It is noted that the glazing surfaces are not to be cleaned during the test period. The data to be noted are:
* test location, latitude, longitude, and climate description
* calendar dates of exposure and angle adjustment schedule
* Climatological data
* Radiant energy incident on the module and maximum monthly temperature.
* Description of samples and periodically observed changes
As an option, climatological data, wetness in the collector module, and incident energy may be collected on a daily basis.

Calculation Methods:
Since the test results are visual observations, no calculational procedures are required for the sample. At the outset, a heat loss calculation should be made to assess the heat losses of the test fixture to insure compliance with the module design requirement. In addition, it is useful to estimate the total insolation for the module over the test period.

Interpretation/ Precision and Accuracy:
Because this test is based upon visual observations, no comments on accuracy or precision are appropriate.

REPORTING FORMAT

Date: December 1986   Reporter's Name: Michael G Hutchins

Country: United Kingdom

Testing Title: Exposure to ultraviolet radiation and moisture

---

Abstracted Scope (200 words):

The Fisons 300-H climatic cabinet is fitted with an ultraviolet lamp.
This enables a range of tests combining exposure of test samples to
ultraviolet radiation with or without humidity or temperature conditions
superposed. Samples are exposed to ultraviolet radiation with or without
the combined effects of temperature or moisture for pre-determined periods.
Surafec optical properties, composition and microstructure may be examined
before and after testing.

Referred Document:

ASTM G53 Operating Light and Water Exposure Apparatus for Exposure of
Non-metallic Materials

Principle of Measurement:

Samples are exposed to measured quantities of ultraviolet radiation within a climatic cabinet. The test environment may be altered to allow different conditions of temperature and/or humidity to be maintained throughout the exposure period.

Apparatus:

Fisons 300-H climatic cabinet equipped with ultraviolet irradiation source.

Calibration:

The irradiance from the uv source is calibrated using solid state detectors to determine the uniformity of beam distribution across exposed samples.

Sampling:

Sample size is normally 100mm x 100mm

Conditioning:

388/

Procedures:

1. Surface optical properties of test samples are measured.
2. The climatic cabinet is set to the required conditions of temperature and moisture.
3. Surfaces are placed in the cabinet and exposed to ultraviolet radiation of known intensity for set periods of time.
4. The conditions of temperature and humidity within the cabinet may be changed if necessary and the uv lamp may be extinguished on a subsequent part of the testing procedure.
5. The samples are removed from the cabinet and surface optical properties remeasured. If significant differences are observed compositional and microstructural examinations are performed.

Calculation Method (if necessary, please include correction method):.

Total ultraviolet irradiance is computed.

Interpretation/Precision & Accuracy:

Difficult to quantify at present

Date: December 1986   Reporter's Name: Michael G Hutchins

Country: United Kingdom

Testing Title: Durability testing of solar absorbers by exposure
to controlled conditions of temperature and moisture.

Abstracted Scope (200 words):

A Fisons 300-H climatic cabinet has been installed which enables samples to
be held at or cycled through controlled conditions of both temperature and
relative humidity. The temperature range of the cabinet is $-20^{\circ}C - 180^{\circ}C$.
The active testing volume is 700mm x 700mm x 700mm. The cabinet is
controlled by a BBC Model B microcomputer, in which a number of different
test cycles are stored.

Surfaces are suspended in the cabinet from purpose built jigs. Comparisons
are made between surface optical properties, composition and microstructure
before abd after temperature-humidity testing.

We should like to report that at this stage we are not sure of the precise
cycles to be followed during testing. We have investigated several standards
or derivatives of them (these are listed below). One of the principal
difficulties is the length of time that some of these tests require. We
should like a test that occupies the cabinet for about one week only per test.

Referred Document:

ASTM E744
NBS TN 1196
ASTM D2247
MIL STD 810-D
Wennerholm H, Accelerated testing of absorbers for flat plate solar collectors,
10th Scandinavian Corrosion Congress, Stockholm, 1986.

Principle of Measurement:

Test samples are exposed to the combined effects of temperature and moisture for pre-determined periods of time. Tests may be carried out under one set of conditions only or the conditions may be varied in a cyclic manner.

Apparatus:

Fisons 300-H IND climatic cabinet with chart recorder for temperature and humidity measurements, BBC Model B microcomputer for control of test cycles.

Calibration: Standard calibration performed annually to check temperature gradients and humidity levels in the chamber.

Sampling:

Standard sample size 100mm x 100mm
Overall cabinet working chamber dimensions 700mm x 700mm x700mm

Conditioning:

As per cycle, no pre-test conditioning

Procedures:

Procedures vary depending on the precise test chosen.

Calculation Method (if necessary, please include correction method):

None

Interpretation/Precision & Accuracy:

Difficult to quantify at this stage.

85

REPORTS' ON GLAZING

c (v)   DURABILITY TO HEAT CYCLES

Date: Feb. 27, 1986   Reporter's Name: Sadamitsu Tsuboi

Country: Japan

Testing Title: Testing Method of Durability to Heat cycles for Sheet Glass

Abstracted Scope (200 words):

This test is applied to transparent sheet glass and it's application products.

The transparent sheet glass includes Float glass, Heat absorbing glass, Thin film coated reflective glass, Low iron glass and etc.

Application products include Laminated glass and Thermal insulation (double glazed)glass.

Referred Document:

Principle of Measurement:

Scheduled heat cycles of ambiance give samples reciprocal stress and simulate extreme day and night temperature difference.

Apparatus:

Heat cycle testing machine PL-3 (Tabai Sesakusho Co., LTD.)

Door — Dry bulb temperature indicator
Wet bulb temperature indicator
window — Dry bulb temperature controller
wiper. — Wet bulb temperature Controller
Room light switch — Operation switch panel
wiper handle
grill — Cycle indicator
Drain valve
Dew catcher
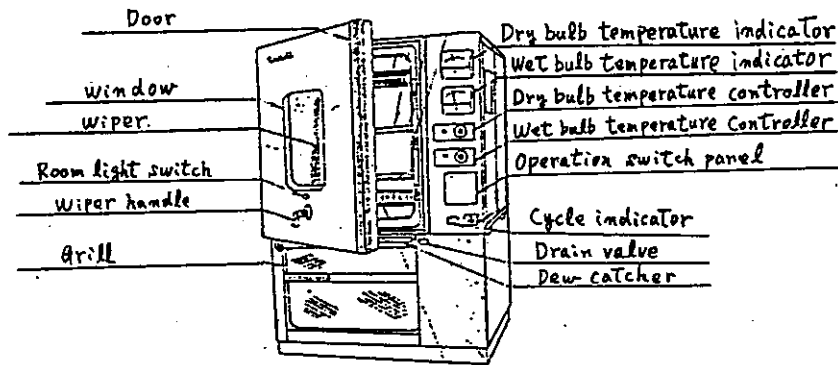
Calibration:

Sampling:

Maximum sample size   200mm X 300mm X 300mm

Conditioning:

Temperature range   −40 ∼ +80 °C

38

Procedures:

    1. Set samples on the holders

    2. Set temperature program

    3. Set humidity program

    4. Put operation switch on

    5. Check quality of the samples after setting time elapsed

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

REPORTS ON HEAT TRANSFER MEDIUM

a(i) THERMAL CONDUCTIVITY

Date: March 12, 1986   Reporter's Name: Tadashi ASAHINA

Country: Japan

Testing Title:

Experimental determination of the thermal conductivity
of heat transfer medium with low electrical conductivity.

Abstracted Scope (200 words):

The thermal conductivity of heat transfer medium with poor
electrical conductivity can be measured rather easily by non
steady-state methods with immersed metallic heaters which are
heated directly by electric current.

Common heat transfer fluid can be devided into two groups.
One is the organic fluid like oils,ethylene glycol and so on,
which has low electrical conductivity and the other is the
inorganic liquid like water,fused salts,fused metals and so on,
which has rather high conductivity.

Non steady-state methods have the advantage of short measur-
ing time and are recommended for the use where the dependence
on temperature is important. Though hot wire methods and stepwise
heated or flash heated flat plate methods are the example of
non steady-state methods, hot wire method is the most prevailing
and widely adaptable method.

Referred Document:

T.Asahina and M.Kosaka:"Thermal conductivity of molten salts;
measuring methods and theory.", Molten Salts, 19,279 (1976).(in
Japanese)

T.Asahina and M.Kosaka:"Thermal conductivity of molten salts;
recent development.", Molten Salts, 25,91 (1982).(in Japanese)

Principle of Measurement:

A thin metallic wire immersed vertically in sample liquid with uniform and stational temperature is heated electrically with constant power and the temperature rise of the wire can be used to calculate the thermal conductivity of the liquid.

Apparatus:

A very thin Pt or Pt alloy wire (typically 50 μm or less in diameter) with a pair of terminals to measure the potential drop along the wire is stretched vertically in the sample liquid. The vertical stretching is superior to the horizontal one because of the slow generation of the convection.

The diameter of the sample vessel must be 160 times greater than that of the wire and the length of the wire should be so long that the obtained values are not affected by the end effect.

Usually, constant current is applyed to the wire after the beginning of the measurement and the change of the potential drop along the wire is recorded to know the temperature rise of the wire.

Calibration:
Usually, distilled water is used as the calibration liquid though the method can get absolute values.

Sampling:

The presence of air bubble has a marked effect on the measurement. Thus dissolved gas must be extracted before the start of the measurement at the temperature.

Conditioning:

The temperature in the sample liquid must be uniform and stational before the start. Thus the furnace or heating equipment must have fine reguration to keep the sample liquid at the intended temperature.

Procedures:

Before the measurement, the dependence of the resistivity of the wire on the temperature must be decided experimentally. After the temperature of the sample liquid becomes uniform and stational and the measuring condition has established, the measuring current begins to be applyed to the wire.

If the suitable current density to the material and the diameter of the wire is chosen, pretty good linear dependence of the temperature rise on the logarithm of time can be obtained within the maximum temperature rise about a few degree.

Calculation Method (if necessary, please include correction method):

The value of the thermal conductivity can be obtained by the following equation.

$$\lambda = (\frac{Q}{4\pi}) / (\frac{d \, \Delta\theta}{d\ln t})$$

where Q is the generated heat from the wire and $\Delta\theta$ is the temperature change. The temperature rise can be calculated from the change of the potential drop through the dependence of the resistivity on the temperature.

Interpretation/Precision & Accuracy:

The generation of the convection can be detected from the bend of the measuring trace. In this case the obtained value of the thermal conductivity is incorrect. The error of the measurement can be within a few percent incase of fine initial temperature field and good measuring instruments.

REPORTS ON HEAT TRANSFER MEDIUM

a-(ii) SPECIFIC HEAT

Designation :

<u>HWA/HWNA-T-LA</u>

<u>Date</u>: 03/07/86   <u>Reporter's Name</u>: Mitsuyuki Koga

<u>Country</u>: Japan

<u>Testing Title</u>:

Measuring Method for Specific Heat of Heat Transfer
Medium.

<u>Abstracted Scope (200 words)</u>:

This test is relative to isopiestic specific heat
measurement of heat transfer medium.

For the first time, the heat capacity of the apparatus
in which the test solution is placed must be measured
by the use of the distilled water with already known
value of specific heat. Then the specific heat of the
test solution is measured.

<u>Referred Document</u>:

Kiyoshi Ogawa : "Thermodynamic Properties of Aqueous
              Solution of Lithium Bromide (1st Report)"
              REFRIGERATION Vol.55 No.630 P.65
              APR · 1980

Principle of Measurement:

Specific heat can be obtained by measuring the increased temperature change of a given weighed test solution which is induced by supplying a certain given heat to it.

Apparatus:
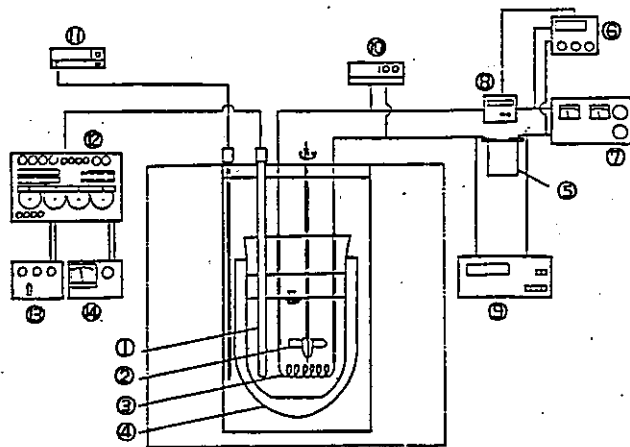
1. Platinum Resistance Thermometer
2. Stirrer
3. Platinum Wire Heater
4. Thermos
5. Standard Resistor
6. Universal Counter
7. Regulated DC Current Power Supply
8. Comparator
9. Accurate Digital Voltmeter
10. Digital Voltmeter
11. Digital Thermometer
12. Accurate Double Bridge
13. Regulated DC Current Equipment
14. Current Indicator

Calibration:

Sampling:

Conditioning:

Measuring Temperature : room temperature ~ 100℃

Solution Volume : 100 ml

34

Procedures:

1. Accurately weigh about 100ml of the distilled water with a known value of the specific heat, then pour it into the thermos.
2. After confirming that the temperature of the distilled water is same as that of the isothermo bath adjusted beforehand, give a heat to it for 360 seconds through the platinum wire heater which is connected to the requlated DC current power supply. At the same time, measure the temperature rises of the distilled water with the accurate double bridge at intervals of 10 seconds.
3. Also, at the same time, measure the supplied heat to the platinum wire heater with two digital voltmeters and universal counter. And then, calculate the heat capacity of the apparatus using the formula (1)
4. Measurement for the specific heat of heat transfer medium is carried out by aforesaid procedures 1~3 with heat transfer medium instead of the distilled water.
5. Then calculate the specific heat of the test solution using the formula (2) from the data of temperature rises, supplied heat, weight of the test solution, and the heat capacity of the apparatus.

Calculation Method (if necessary, please include correction method):

$$\Sigma GC = \frac{i \cdot v \cdot T}{4.18605 \cdot \Delta t} - G \cdot C_p \quad \text{......} \quad (1)$$

$$C_p \cdot e = \frac{1}{GH} \left(\frac{I \cdot V \cdot T}{4.18605 \cdot \Delta t} - \Sigma GC \right) \quad \text{......} \quad (2)$$

where

$C_p \cdot e$: isopiestic specific heat of the test solution.
ΣGC : heat capacity of the apparatus.
C_p : isopiestic specific heat of the distilled water at atomosperic pressure.
GH : weight of the test solution.
G : weight of the distilled water.
I, i : supplied current to the heater.
V, v : supplied voltage to the heater.
T : supplied time of heat to the heater.
Δt : temperature rises of the test solution.

REPORTS ON HEAT TRANSFER MEDIUM

a (iii) LATENT HEAT

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Melting and Crystallization Temperatures by Thermal Analysis

Abstracted Scope (200 words):

This method covers the determination of transition temperatures such as the melting and crystallization temperatures of materials by differential scanning calorimetry (DSC) and differential thermal analysis (DTA). The normal operating temperature range is from -120 to 600°C. With special equipment, the temperature range can be extended.

Computer or electronic based instruments, techniques, or data treatment equivalent to this test method may be used.

Referred Document:

ASTM E 794 Standard Test Method for Melting and Crystallization
            Temperatures by Thermal Analysis

ASTM D 3418 Standard Test Method for Transition Temperatures of Polymers
            by Thermal Analysis

Principle of Measurement:

The method consists of heating or cooling the test material at a controlled rate in a controlled atmosphere through the region of a transition, such as fusion or crystallization. The difference in heat flow or temperature between the test material and a reference material due to energy changes in the material is continuously monitored and recorded. Absorption or release of energy results in endothermic or exothermic peaks.

Apparatus:

A differential scanning calorimeter, capable of heating or cooling at rates up to at least 10°C/min and of automatically recording the differential energy input between the sample and reference material both to the required sensitivity and precision. Typically, the differential sensitivity should be sufficient to provide specimen temperature readability to within 1°C.

A differential thermal analyzer, capable of heating or cooling at rates up to at least 10°C/min and of automatically recording the differential temperature between sample and reference material both to the required precision; typically within 1°C.

Specimen pans, for DSC, composed of aluminum or other metal of high thermal conductivity. For DTA, sample cups or tubes composed of borosilicate glass, alumina, or quartz may be used. The specimen pans or tubes must not react with the sample.

Nitrogen, or other inert purge gas.

An analytical balance, with a capacity greater than 15 mg, capable of weighing to the nearest 0.01 mg.

Sampling:

Powdered or liquid samples should be thoroughly mixed and sampled from by removing portions from different parts of the container which are then combined and mixed. Film, sheet, molded or pelleted samples can be cut, punched, or bored to obtain pieces that fit the sample containers.

Conditioning:

Materials that have a residual thermal history may require a preliminary thermal cycle to above the melting point to avoid interference with the measurement of the transitions of interest. Care should be taken to avoid thermal decomposition of the test specimen.

Procedures:

1. Use a sample weight appropriate for the material to be tested; about 5 mg. for first order transitions and 10 to 20 mg for glass transitions. An appropriate sample weight results in 25 to 95% scale deflection of the recording system when acceptable sensitivity is used.

2. Ensure intimate thermal contact between the sample, the sample pan or tube and thermocouple, where used. Good thermal contact is also required between the sample container and the test apparatus.

3. Load the specimen into the instrument chamber, purge the chamber with nitrogen or other inert gas at a flow rate of 10 to 50 cm^3/min during the experiment.

4. Heat the specimen rapidly to 50°C below the transition temperature of interest and equilibrate.

5. Heat the specimen at 10°C/min through the transition until the baseline is reestablished above the transition. Hold at this temperature for about two min and then cool the specimen, at the same rate, down through the transition until the baseline is reestablished below the transition.

6. Reweigh the specimen after completion of scanning and discard. Report any mass loss observed.

Calculation Method (if necessary, please include correction method):

From the resultant curve, measure the following points:

T_s = temperature of the first deviation from the baseline, °C
T_e = extrapolated onset temperature for fusion, °C
T_p = melting peak temperature, °C
T_f = temperature of return to baseline, °C
T_n = extrapolated temperature of crystallization onset (nucleation), °C
T_c = temperature of crystallization peak, °C

Report similar temperatures for other transitions.

Interpretation/Precision & Accuracy:

Repeatability (Single Analyst): Two averages for T_s (95% confidence level) should be considered suspect if they differ by more than 7.9°C.
Two averages for T_c, T_p, T_f, T_n, and T_e should be considered suspect (95% confidence level) if they differ by more than 3.1°C.

Reproducibility (Multilaboratory): Two averages for the following should be considered suspect (95% confidence level) if they differ by:
40°C for T_s for broadly melting material, 7.1°C for narrowly melting material, 5.9°C for T_e, T_p, T_n and T_c, and 19°C for T_f.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Heats of Fusion and Crystallization by Differential Scanning Calorimetry

Abstracted Scope (200 words):

This test method covers the determination of the heat of fusion and
crystallization by differrential scanning calorimetry. It is applicable
to solid samples in granular form or in any fabricated shape from which an
appropriate specimen can be cut and to liquid samples that crystallize
within the range of the instrument.

The normal operating temperature range is from -120 to 600°C. This
temperature range can be extended, depending on the instrumentation used.
This test method is generally applicable to thermally stable materials
with well defined endothermic or exothermic behavior.

Computer or electronic based instruments, techniques, or data treatment
equivalent to this test method may be used.

Referred Document:

ASTM E 793 Standard Test Method for Heats of Fusion and Crystallization
            by Differential Scanning Calorimetry

ASTM D 3417 Standard Test Method for Heats of Fusion and Crystallization
            of Polymers by Thermal Analysis

Principle of Measurement:

Differential scanning calorimetry (DSC) is a technique in which the difference in energy inputs into a substance and a reference material is measured as a function of temperature, while the substance and reference material are subjected to a controlled temperature program. Differences in the amount of energy required to maintain the test specimen and reference at the same temperature are used to determine heats of transformation.

Apparatus:

A differential scanning calorimeter capable of heating and cooling rates up to at least $10^{\circ}C/min$ and of automatically recording the differential heat flow between the sample and reference material to the required sensitivity and precision. The instrument should have sufficient heat flow sensitivity to provide a precision within 1% and a time base precision of within 1%.

A planimeter or other method of area measurement with an accuracy and precision within 1%.

Specimen holders, inert to the sample, composed of aluminum or other material of high thermal conductivity.

Nitrogen, or other inert gas supply, for purging purposes.

A balance, with a capacity greater than 15 mg, capable of weighing to the nearest 0.01 mg.

Sampling:

Powdered or granular specimens should be mixed thoroughly prior to sampling and should be sampled by removing and then blending portions taken from various parts of the container. Liquid samples may be sampled directly after stirring.

Conditioning:

A preliminary test cycle to remove previous thermal history, e.g., residual thermal stresses, or annealing above the glass transition temperature of polymers such as crystallizable polystyrene, may be required before valid test data are taken. Comparison of initial and subsequent test runs can be used to determine whether or not conditioning is required.

Procedures:

1. Into a clean specimen holder, weigh 1 to 15 mg of specimen.

2. Seal the specimen holder with a lid under ambient conditions, minimizing the free space between sample and lid. Hermetic sealing in an inert atmosphere may be desirable for specimens sensitive to oxidation.

3. Load the specimen into the instrument chamber and purge with nitrogen or other inert gas throughout the experiment at 10 to 50 cm^3/min.

4. Select the appropriate heat flow axis and time axis sensitivities to yield an area of 30 to 60 cm^2 under the fusion endotherm, or crystallization exotherm.

5. The specimen may be heated rapidly to 50°C below the melting (or other transition) temperature and allowed to equilibrate.

6. Heat the specimen at 10°C/min through the transition, until a baseline is reestablished above the endotherm or exotherm.

7. Hold the specimen at this temperature for 2 min.

8. Cool the specimen at 10°C/min through the transition until baseline is reestablished below the endotherm or exotherm. Reweigh the specimen after completion of scanning. Discard the data if there is mass loss greater than 1% or evidence of reaction with the sample holder.

Calculation Method (if necessary, please include correction method):

$$H = (ABT/W) (H_S W_S / A_S T_S B_S) \quad , \text{ where}$$

H = heat of transition of the sample, J/g
H_S = heat of transition of the standard, J/g
A = peak area of the sample, cm^2
A_S = peak area of the standard, cm^2
B = time scale used for the sample, min/cm
B_S = time scale used for the standard, min/cm
W = mass of the sample, mg, W_S = mass of the standard, mg
T = Y-axis sensitivity used for the sample, mW/cm
T_S = Y-axis sensitivity used for the standard, mW/cm.

Interpretation/Precision & Accuracy:

Repeatability (single analyst): 2.8% coefficient of variation with 88 degrees of freedom. Two such averages should be considered suspect (95% confidence level) if they differ by more than 7.8%.

Reproducibility of pure materials (Multilaboratory): 3.0% coefficient of variation at 58 degrees of freedom for materials melting or crystallizing over a narrow temperature range. Two such results should be considered suspect (95% confidence level) if they differ by more than 8.6%. Results for broad melting range polymers should be considered suspect (95% confidence level) if they differ by more than 23%.

REPORTS ON HEAT TRANSFER MEDIUM

b (i) SPECIFIC VOLUME

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

     Apparent Specific Gravity of Liquid Industrial Chemicals

---

Abstracted Scope (200 words):

These test methods cover the apparent specific gravity of liquid industrial
chemicals. Method A determines apparent specific gravity by means of a
hydrometer. Method B determines apparent specific gravity by means of a
pycnometer.

Apparent specific gravity is the ratio of the weight in air of a unit
volume of a material at a stated temperature to the weight, in air of equal
density, of an equal volume of gas-free distilled water at a stated
temperature. It shall be stated as follows:

  When the temperatures of the material and of the water are the same:

$$\text{Specific gravity, } x/x\,^{\circ}C$$

  where x is the temperature of the material and the water.

  When the temperature of the material and the water are not the same:

$$\text{Specific gravity, apparent, } x/y\,^{\circ}C$$

  where x is the temperature of the material and y is the temperature of
  the water.

Referred Document:

ASTM D 891   Standard Test Methods for Specific Gravity, Apparent, of
            Liquid Industrial Chemicals

Principle of Measurement:

Method A determines the specific gravity, apparent, by determining the displacement of a calibrated hydrometer immersed in the sample at the test temperature. Method B uses a tared pycnometer to determine the weights of fixed volumes of freshly boiled water and the sample. The ratio of the weight of sample to the weight of water that fills the pycnometer is the specific gravity, apparent.

Apparatus:

Thermometers meeting the requirements of ASTM Specification E 1 and and covering the required temperature range.

A water bath capable of maintaining the selected test temperature $\pm$ 0.05°C.

Hydrometers meeting the requirements of ASTM Specification E 100 and covering the required range (Method A only).

A hydrometer cylinder, having an inside diameter at least 25.4 mm greater than the outside diameter of the hydrometer, and a height such that after equilibrium has been reached, the lowest point on the hydrometer will be at least 25.4 mm off the bottom of the cylinder (Method A only)

A pycnometer of 25-mL capacity with a ground-glass stopper having a capillary opening, a chamber to provide for expansion up to room temperature, and a cap to prevent evaporation (Method B only).

An analytical balance capable of weighing 150 g with a precision of 0.1 mg and a set of class S analytical weights, if required by the balance (Method B only).

Sampling:

Conditioning:

Procedures:

Method A: Cool the sample to about 2°C below the test temperature, pour it into a clean hydrometer cylinder taking care to avoid splashing and bubble formation, and remove any air bubbles adhering to the surface by touching them with a piece of clean paper. Place the hydrometer cylinder in the water bath, allow it to equilibrate at the bath temperature, and carefully lower the hydrometer into the sample to a level two small scale divisions below that at which it will float and then release the hydrometer. After the hydrometer has come to rest and floats freely away from the walls of the cylinder, read the gravity as the point at which the surface of the sample apparently cuts the hydrometer scale.

Method B: Fill the cleaned pycnometer with freshly boiled water that has been cooled to 2°C below the test temperature. Place it in the water bath maintained at the test temperature and allow it to equilibrate for 30 min. Adjust the level of liquid to the proper point on the pycnometer, put the stopper in place, remove from the bath, wipe dry, and weigh. Empty the pycnometer, rinse successively with alcohol and ether, remove the ether vapor, immerse in the bath, and bring to the test temperature as was done before. After at least 30 min, put the stopper in place, remove from the bath, wipe dry, and weigh. Subtract the weight of the empty pycnometer from the weight when filled with water to get the weight of contained water, W, at the test temperature. Repeat this procedure with the sample instead of water to get the weight of the sample contained in the pycnometer, S, at the test temperature.

Calculation Method (if necessary, please include correction method):

$$\text{Specific gravity, apparent, at } x/y°C = S/W$$

where:

x = temperature of the sample, °C, and
y = temperature of the water, °C.

Interpretation/Precision & Accuracy:

Repeatability: Two averages (each the average of duplicates) obtained by the same analyst on different days should be considered suspect (95 % confidence level) if they differ by more than 0.0005 unit for Method A and 0.0002 unit (ethylene and propylene glycols) to 0.00008 unit (methanol) with Method B.

Reproducibility: Two averages (each the average of duplicates) obtained by analysts in different laboratories would be considered suspect (95 % confidence level) if they differ by more than 0.0021 unit for Method A and 0.0008 unit (glycols) to 0.00055 unit (methanol) for Method B.

REPORTS ON HEAT TRANSFER MEDIUM

b (ii) KINEMATIC VISCOSITY OR COEFFICIENT OF VISCOSITY

Designation :
HWA − T − ST

Date : March 6, 1986   Reporter's Name : T. Sawada

Country : Japan

Testing Title :

Testing Method of Kinematic Viscosity of Heat
Transfer Medium

Abstracted Scope (200 words) :

This method covers the determination of kinematic
viscosity of heat transfer medium, and can be
used for Newtonian fluids.

The time required for a certain quantity of sample
to flow in the glass capillary viscometer is
measured.

Kinematic viscosity is obtained by correcting
measured time using the constant of viscometer.

Referred Document :

JIS K 2283   Testing Method for Kinematic Viscosity
              and Calculating Method for Viscosity
              Index of Crude Oil and Petroleum
              Products

JIS Z 8803   Methods of Viscosity Measurement

Principle of Measurement:

The Hagen-Poiseulle equation of flow through a pipe
is applied to this testing method.

Apparatus:
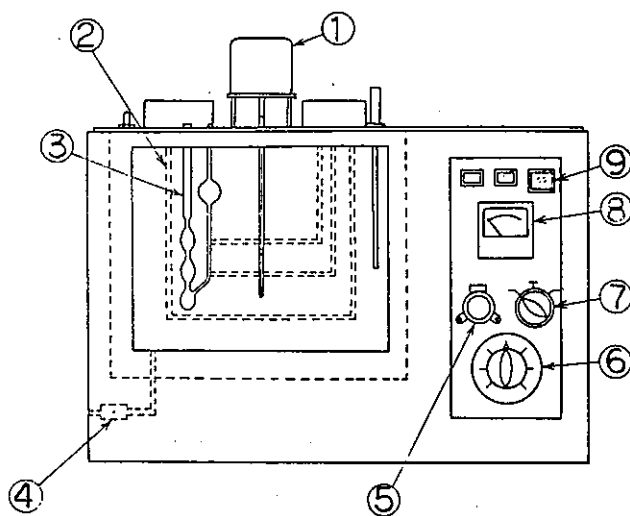
1. Stirring rod
2. Heater
3. Viscometer
4. Drain pipe
5. Heat regulator
6. Voltage-regulator
7. Transfer switch
8. Ammeter
9. Pilot lamp

Calibration:

Sampling:

Conditioning:

Temperature in the thermostat

scattering of distribution   < o.o1 ℃
fluctuation   < 0.01 ℃

Procedures:

1. Contain sample in the capillary viscometer and attach it vertically in the thermostat.

2. Open the tap of viscometer and let the sample flow when the temperature reaches the prescribed value.

3. Measure the time for the meniscus of sample to descend from mark to mark to the nearest 0.1 second.

Calculation Method (if necessary, please include correction method):

$$v = c_1\, t - c_2\, /\, t$$

    v : kinematic viscosity   (St)

    c_1: constant of viscometer (St/s)

    c_2: constant of viscometer (St/s)

    t : measured time   (s)

Interpretation/Precision & Accuracy:

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Viscosity of Transparent Liquids by Bubble Time Method

Abstracted Scope (200 words):

This method covers the determination of the viscosity in bubble seconds by timing or comparison. The bubble seconds are approximately equal to stokes for most liquids.

The method is applicable to transparent liquids that are free from crystalline or gel particles.

Referred Document:

ASTM D 1545   Standard Test Method for Viscosity of Transparent Liquids by Bubble Time Method

<u>Principle of Measurement</u>:

The time required for a bubble to rise a fixed distance in a vertical tube
is measured or compared to that required for liquid standards of known
viscosity.

These bubble seconds are approximately equal to stokes for most liquids.

<u>Apparatus</u>:

A constant temperature bath capable of maintaining temperature to within
0.1°C at 25°C with water as the bath medium.

Standard viscosity tubes of clear glass and with flat bottoms, 10.65 mm
inside diameter and 114 mm in outside length. Plainly legible lines shall
be located at 27, 100, and 108 mm measured from the outside bottom of the
tube.

Reference standards consisting of a series of standard viscosity tubes
filled with transparent liquids having predetermined viscosities in stokes
and bubble seconds and spaced in logarithmically even increments of about
26%.

A timing device such as a stop watch or electric stop clock capable of
being read to a precision of 0.1 s.

Tube racks capable of inverting one or more viscosity tubes 180 deg to
within 1 deg of a vertical position while rack and tubes are immersed in
the constant temperature bath.

Viscosity tubes, No. 2 short.

<u>Sampling</u>:

<u>Conditioning</u>:

Procedures:

1. Fill a standard viscosity tube with the material to be tested to approximately level with the 108-mm line.

2. Transfer the tube to a constant 25°C temperature bath with the cork loosely inserted. Hold at this temperature for 10 min.

3. At the end of the 10 min adjust the level of the liquid so that the bottom meniscus will be level with the 100-mm line. Insert the cork so that the bottom of the cork is at the 108-mm line. This will ensure a bubble of suitable and uniform size.

4. Insert the tube in the rack and immerse in the 25°C water bath, with the proper range standards inserted in the rack alongside the liquid to be tested, if the comparison method is used. Allow to equilibrate for a minimum of 20 min before reading the viscosity.

5. To read, invert the tube quickly and determine the time required for the top of the bubble to travel between the tangent with the 27-mm line and the tangent with the 100-mm line.

6. Report the viscosity obtained by either timing or comparison with standards as "bubble seconds" or "approximate stokes."

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

On the basis of an interlaboratory study of this method, the following criteria should be used for judging the acceptability of results at the 95% confidence level:

Two results obtained by the same operator should be considered suspect if they differ by more than 4.9% relative.

Two results, each the mean of two duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 9.0% relative.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Rheological Properties of Materials by Rotational (Brookfield) Viscometer

Method A: Apparent Viscosity

Abstracted Scope (200 words):

Method A consists of determining the apparent viscosity of coatings and
related materials by measuring the torque on a spindle rotating at a
constant speed in the material. The method is applicable to both Newtonian
and Non-Newtonian liquids.

The method provides for determining the apparent viscosity at a given
rotational speed, although two or more speeds better characterize a
non-Newtonian liquid than does a single viscosity measurement.

Methods B and C of ASTM D 2196, which consist of determining the shear
thinning and thixotropic rheological properties of materials, are not
discussed in these summaries because of their limited applicability to
the types of heat transfer fluids currently used in solar heating and
cooling applications.

These methods cover the shear rate range from 0.1 to 50 s^{-1}.

The shear rate of Newtonian liquids is proportional to the shear stress.

Referred Document:

ASTM D 2196   Standard Test Methods for Rheological Properties of Non-
              Newtonian Materials by Rotational (Brookfield) Viscometer

Principle of Measurement:

The apparent viscosity is determined by measuring the torque on a spindle rotating at a constant speed in a material.

Apparatus:

Rotational-type viscometers having at least four speeds and a set of spindles appropriate for the viscosity range being measured such as: Brookfield viscometer models LVF, LVT, RVF, or RVT.

A thermometer having a range from 20 to 70°C and conforming to the requirements for Thermometer 49C as prescribed in ASTM Specification E 1.

Round containers, 0.5 L and 85 mm in diameter or 1 L and 100 mm in diameter.

A shaker or equivalent machine capable of vigorously shaking the test specimen.

Standard oils, calibrated in absolute viscosity, millipascal seconds.

Sampling:

Conditioning:

Fill the container to within 25 mm of the top with the sample and bring it as close as possible to 25°C, or other agreed upon test temperature. Shake the specimen vigorously for 10 min, and then allow it to stand undisturbed for 60 min at the test temperature (usually 25°C). Start the test no later than 65 min after removing the can from the shaker.

Procedures:

1. Calibrate the apparatus using at least two standard oils having viscosities differing by at least 5 P (0.5 Pa.s) within the viscosity range of the material being measured and of the viscometer. Condition the oils at 25°C or other agreed upon test temperature before testing.

2. Make all measurements as close as possible to the desired temperature.

3. Place the viscometer on an adjustable stand and lower the viscometer to a level that will immerse the spindle to the proper depth, which is indicated by a mark on the spindle.

4. Select the spindle/speed combination that will give a minimum scale reading of 10. A reading in the middle or upper portion of the scale is preferable.

5. Turn on the viscometer. Adjust the viscometer to the rpm selected for the material under test. Allow the viscometer to run until the pointer has stabilized, depress the clutch and switch off the motor so that when it stops, the pointer will be in view.

6. Report the following: the viscometer model and spindle; the viscosity at the spindle/speed utilized; the specimen temperature; and the shake time and rest period used, if other than specified.

Calculation Method (if necessary, please include correction method):

Calculate the apparent viscosity at each speed as follows:

$$V = fs \qquad where:$$

V = viscosity of the sample in centipoises, mPa.s
f = scale factor furnished with the instrument
s = scale reading of the viscometer.

Interpretation/Precision & Accuracy:

A precision statement is being prepared.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Kinematic Viscosity of Transparent and Opaque Liquids

Abstracted Scope (200 words):

This method covers the determination of the kinematic viscosity of both
transparent and opaque liquids by measuring the time for a volume of
liquid to flow under gravity through a calibrated glass capillary
viscometer. It is intended for application to liquids for which the shear
stress and shear rates are proportional (Newtonian flow). By proper
selection of viscometer type and range, it is possible to measure the
viscosity of liquids having values between 0.2 and 300,000 cSt.

The dynamic viscosity can be obtained by multiplying the measured
kinematic viscosity by the density of the liquid.

Referred Document:

ASTM D 445   Standard Test Method for Kinematic Viscosity of Transparent
             and Opaque Liquids (and the Calculation of Dynamic Viscosity)

Principle of Measurement:

Kinematic viscosity is a measure of the resistive flow of a fluid under gravity, the pressure head being proportional to the density. For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, which has the dimension L^2/T, where L is a length and T is a time.

Apparatus:

Viscometers of the glass capillary type, calibrated and capable of measuring kinematic viscosity within the desired limits of precision.

Viscometer holders that enable the viscometer to be suspended in a position similar to the one it is calibrated in.

A thermostated bath capable of maintaining the temperature of the bath medium to within $0.01^{\circ}C$ over the length of the viscometers. The medium may be either a vapor or a transparent liquid.

Standardized liquid-in-glass thermometers of an accuracy after correction of $0.02^{\circ}C$.

A timing device capable of providing readings with a descrimination of 0.2 s or better and an accuracy within 0.07% when tested over intervals of 15 min.

Viscosity standards having the appropriate kinematic viscosity for the liquids being tested.

Drying tubes to be fitted onto the ends of the viscometer to prevent water condensation when the test temperature is below the dew point.

Sampling:

Conditioning:

Heat the sample in the original container to about $50^{\circ}C$ with stirring and shaking. Probe the container bottom to be certain all materials are in solution. Pour 100 ml into a 125 ml flask and immerse in boiling water for 30 min. Mix well, strain through a screen directly into the viscometer and complete the viscosity test within 1 h after preheating.

Procedures:

1. Charge the viscometer and allow it to remain in the bath long enough to equilibrate at the test temperature.

2. Use suction or pressure to adjust the head level of the test sample to a position in the capillary arm about 5 min ahead of the first timing mark.

3. With the sample flowing freely, measure the time required for the meniscus to pass from the first timing mark to the second mark. If this flow time is less than 200 s, select a viscometer with a capillary of smaller diameter and repeat the operation.

4. For modified Ostwald and suspended-level type viscometers, repeat the procedure to make a second measurement of flow time starting at step 2. For reverse flow viscometers, use the same or another viscometer and recharge with sample.

5. If measurements agree to within 0.2% use the average for calculating the kinematic viscosity, with the exception of reverse flow types where flow times should agree to within 0.35%. If these agreements are not obtained, reject the test results.

6. Clean the viscometer carefully between successive determinations. Viscometers used for silicone fluids, fluorocarbons, and other difficult to remove liquids should be reserved for those liquids.

Calculation Method (if necessary, please include correction method):

$$v = Ct \quad \text{where:}$$

v = kinematic viscosity, cSt,
C = calibration constant of the viscometer, cSt/s, and
t = flow time, s .

$$n = pv \quad \text{where:}$$

n = dynamic viscosity, cP,
p = density, g/cm^3, and   v = kinematic viscosity, cSt.

Interpretation/Precision & Accuracy:

Repeatability (single analyst): The difference between successive test results, obtained by the same apparatus under constant operating conditions on identical test material would be suspect if it exceeds 0.35% of their mean (95% confidence level).

Reproducibility (multilaboratory): The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would be suspect if it exceeds 0.70% of their mean (95% confidence level).

REPORTS ON HEAT TRANSFER MEDIUM

C. RELIABILITY

c (i) SOLIDIFYING POINT

Date: March 6, 1986   Reporter's Name: T. Sawada

Country: Japan

Testing Title:

Testing Method for Solidifying Point of Heat
Transfer Medium

Abstracted Scope (200 words):

This method covers the determination of solidifying
point of heat transfer medium.

The solidifying point is defined the temperature
at which cryohydrate forms when the liquid isn't
subcooled.

Referred Document:

JIS K 0065   Testing Method for Freezing Point of
Chemical Products

JIS K 2234   Engine Antifreeze Coolant

<u>Principle of Measurement</u>:

Obtain solidifying point by making temperature-time curve of cooling sample.
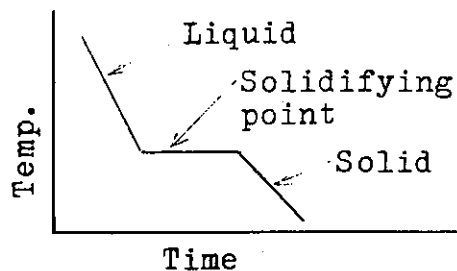
<u>Apparatus</u>:
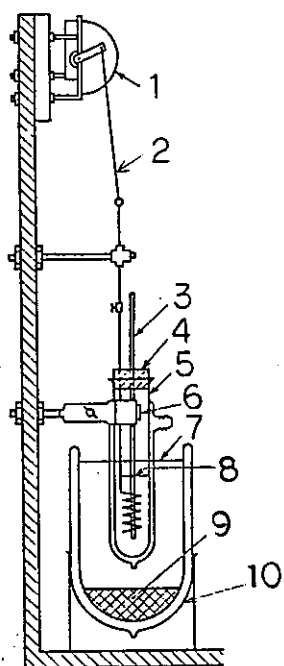
1. Motor
2. Stirring rod
3. Thermometer
4. Cork
5. Cooling tube
6. Clamp
7. Coolant-level
8. Sample-level
9. Glass wool
10. Dewar bottle

<u>Calibration</u>:

<u>Sampling</u>:

<u>Conditioning</u>:

Ambient temperature	20 ± 15 ℃
Ambient humidity	65 ± 20 % RH

Procedures:

1. Make the coolant from dry ice with methanol or
   aceton in Dewar bottle.
2. Put 75-100 ml of sample in the cooling tube.
3. Keep the sample level 10 mm below coolant level
4. Record the temperature of sample every 1 minute.
5. Adjust the cooling rate to 1 ℃/min. when the
   temperature become 5 ℃ higher than expected
   solidifying point.
6. Record the temperature every 15 second to the
   nearest 0.1 ℃.
7. Make the solidifying curve.

Calculation Method (if necessary, please include correction method):

   Round the average of two test results to the tenth's.

Interpretation/Precision & Accuracy :

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

          Freezing Point of Aqueous Coolant Solutions

Abstracted Scope (200 words):

This method covers the determination of the freezing point of an aqueous
engine coolant solution in the laboratory. Freezing point is defined as
the temperature at which crystallization begins in the absence of under-
cooling, or the maximum temperature reached immediately after initial
crystal formation in the case of undercooling.

The method is applicable to heat transfer fluids used in solar heating and
cooling applications.

Referred Document:

ASTM D 1177   Standard Test Method for Freezing Point of Aqueous Engine
              Coolant Solution

Principle of Measurement:

This method involves the determination of the time-temperature curve prior to freezing and the determination of the horizontal or flattened portion of the freezing curve. The freezing point is taken as the intersection of projections of the cooling curve and the freezing curve. If the solution undercools, the freezing point is the maximum temperature reached after undercooling.

Apparatus:

FIG. 2 Time-Temperature Cooling Curves for Determining the Freezing Point of an Engine Coolant

- Driving Arm
- Windshield Wiper Motor
- Driving Rod
- Reciprocating Rod
- Sleeve Bearing
- Holder for Bearing
- Temperature-Measuring Device
- Stirrer
- Stoppered Hole for "Seeding" (6.4 mm or 1/4 in.)
- Stopper with Holes as Shown
- Freezing Tube, 200-ml capacity
- Clamp for Freezing Tube
- Cooling Bath Level
- Sample Level
- Cooling Bath (1.9-liter or 2-qt Dewar)
- Glass Wool Pad

FIG. 1 Assembly of Freezing Point Apparatus

FIG. 3 Bottom of Freezing Tube Showing Seeding Tip

Sampling:

Conditioning:

Procedures:

1. Assemble the apparatus, with no refrigerant or antifreeze installed, and check the stirring mechanism for freedom of operation.

2. Fill the Dewar flask surrounding the freezing tube with refrigerant consisting of either dry ice, in alcohol or other suitable liquids, or liquid nitrogen (for freezing points of -46°C or less).

3. Temporarily remove the stopper from the freezing tube and introduce 75 to 100 mL of the sample. The sample may be precooled to 8°C above the expected freezing point before introducing it into the freezing tube.

4. Start the stirrer and adjust it to operate at 60 to 80 strokes per min. As soon as stirring is begun, observe and record the temperature at regular intervals of time; 15 s, if possible, as the expected freezing point is approached. At the expected freezing point, seeding should be started to prevent undercooling. This can be accomplished by introducing a wire which has a small portion of the solution being tested frozen on its tip.

5. Continue temperature readings for at least 10 min after the apparent freezing point.

Calculation Method (if necessary, please include correction method):

Plot the observed temperature against time on rectangular coordinate paper. Where the curve shows a definite flat or plateau during freezing, the freezing point is taken as the intersection of projections of the cooling curve and the flat or horizontal plateau portion of the freezing curve. If the solution undercools, the freezing point is the maximum temperature reached immediately after undercooling.

Interpretation/Precision & Accuracy:

Results should not differ from the mean by more than the following amounts:

Repeatability: 0.3°C for one operator and apparatus.

Reproducibility: 0.6°C for different operators and apparatus.

REPORTS ON HEAT TRANSFER MEDIUM

c (ii) BOILING POINT

Date :   March 6, 1986   Reporter's Name :   T. Sawada

Country : Japan

Testing Title :

Testing Method for Boiling Point of Heat
Transfer Medium

Abstracted Scope (200 words) :

This method covers the determination of boiling
point of heat transfer medium.

The temprrature is measured as boiling point when
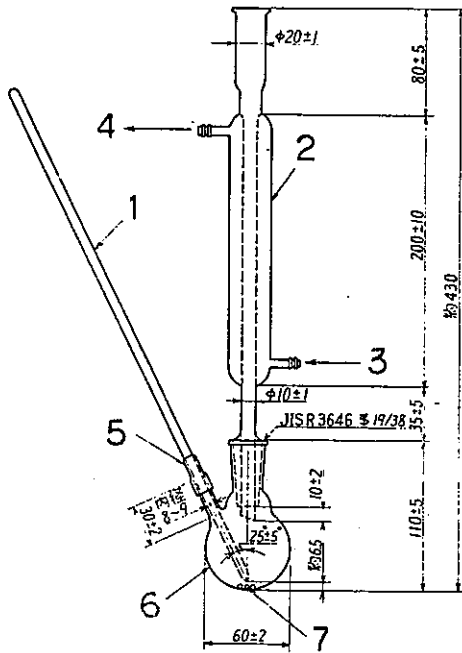the condensation is observed in the cooling tube.

Referred Document :

JIS K 2233   Non-Petroleum Base Motor Vehicle
Brake Fluids

Boiling point is the temperature of heated sample
when the condensation is observed.
The condensation is jduged visually as 1-2 drops per
second in the cooling tube.

Apparatus :

1. Thermometer
2. Cooling tube
3. Coolant-inlet
4. Coolant-outlet
5. Rubber tube
6. Flask
7. Boiling tips

Calibration :

Sampling :

Conditioning :

Ambient temperature   20 $\pm$ 15 ℃
Ambient humidity   65 $\pm$ 20 % RH

Procedures:

1. Measure 60 ml of sample into the flask.

2. Cooling condition

   water temperature   28 ℃
   temperature increase   2 ℃

3. Heating condition

   1-5 drops per sec.   for 10+2 min
   1-2 drops per sec.   for 5+2 min.

4. Record the temperature every 30 sec. to the nearest 0.5 ℃ after keeping above condition for 2 min.

5. Report the average of four values as a test result.

6. If the values obtained in the two test results differ by more than 1 ℃, repeat the procedure descrived above.

Calculation Method (if necessary, please include correction method):

Temperature correction for barometic pressure

$$C = 0.00012 (760 - P) (273 + t)$$

   C : Correction value added to the measured temperature t

   P : Barometic pressure at the test (mmHg)

   t : Mesured temperature (℃)

Interpretation/Precision & Accuracy:

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Boiling Point of Coolants

Abstracted Scope (200 words):

This test method covers the determination of the equiibrium boiling point
of engine coolants that are miscible with water. The equilibrium boiling
point indicates the temperature at which the sample will start to boil in
a cooling system under equilibrium conditions at atmospheric pressure.

The products may be marketed in either a ready-to-use form (prediluted)
or as a concentrate that requires dilution with water. This test procedure
is applicable to diluted solutions as well as to concentrates.

The method is applicable to heat transfer fluids used in solar heating and
cooling applications.

Referred Document:

ASTM D 1120 Standard Test Method for Boiling Point of Engine Coolants

83

Principle of Measurement:

Sixty milliliters of the sample are boiled under equilibrium conditions at atmospheric pressure in a 100-mL flask. The temperature of the liquid corrected for barometric pressure is the boiling point.

Apparatus:
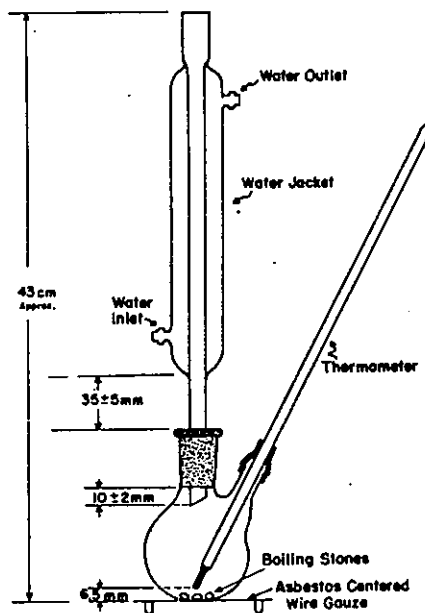
FIG. 1   Boiling Point Test Apparatus

Sampling:

Allow material as received in the original container to come to room temperature (not below 20°C). Shake the container to mix any material that may have separated. Immediately remove the sample required for testing.

To prepare a dilute solution, thoroughly mix the sample, pipet the required volume into a calibrated volumetric flask, and add distilled water.

Conditioning:

Procedures:

When everything is in readiness, turn on the condenser water and apply heat by means of a bunsen burner or electric heater at a rate such that the sample is brought to its boiling point within 10 min. Heat strongly until boiling is reached and then reduce heat input slowly until the required reflux rate is obtained. Adjust the rate of reflux over the next 10-min period to 1 to 2 drops of reflux per second.

Maintain this reflux rate for 2 min and then record the observed temperature and barometric pressure.

Calculation Method (if necessary, please include correction method):

Thermometer Inaccuracy: Correct the observed temperature by applying a factor obtained by calibrating the thermometer.

Variation from Standard Barometric Pressure: Apply the following correction factors for each mm Hg difference in pressure from 760 mm following correction for thermometer inaccuracy:

Under 100°C	0.03°C/mm Hg
100 to 190°C	0.039°C/mm Hg
Over 190°C	0.04°C/mm Hg

Interpretation/Precision & Accuracy:

Reproducibility:

For diluted samples boiling below 100°C, results should not differ from each other by more than 1.4°C and, for samples boiling above 100°C, results should not differ from each other by more than 2.5°C.

For unused concentrated samples boiling below 100°C, results should not differ from each other by more than 1.4°C and, for samples boiling above 100°C, results should not differ by more than 5.6°C.

REPORTS ON HEAT TRANSFER MEDIUM

c (iii) FLASHING POINT

Designation :

HWA – T – ST

Date : March 6, 1986	Reporter's Name : T. Sawada
Country : Japan	

Testing Title :

Testing Method for Flash Point of Heat Transfer
Medium

Abstracted Scope (200 words) :

This method covers the determination of the flash
point of heat transfer medium and can be used for
the sample of which flash point is over 80 ℃ .

Referred Document :

JIS K 2265 Testing Methods for Flash Point of
Crude Oil and Petroleum Products

Principle of Measurement:

Measure minimum temperature when the mixed gas of
vapor of sample and air takes fire from the flame
close to the surface of sample.

Apparatus:
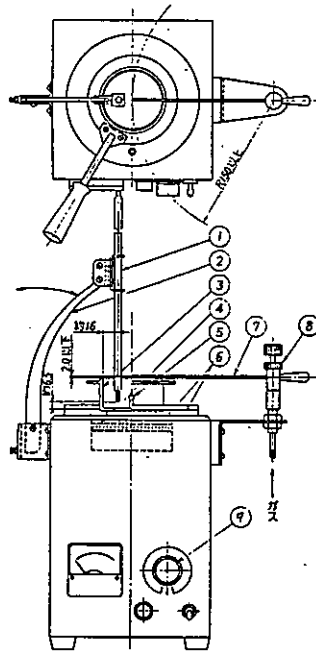
1. Thermometer
2. Holder
3. Sample cup
4. Electric heater
5. Standard glove
6. Heating plate
7. Test flame nozzle
8. Gas supply valve
9. Control of heating

Calibration:

Sampling:

Conditioning:

Procedures:

1. Adjust the size of test flame to standard glove
   or 4 ± 0.8 mm diameter.

2. Heating rate   14-17 ℃/min.

3. Adjust the heating rate 5.5 ± 0.5 ℃/min. when
   the temperature become 28 ℃ lower than flash point
   expected。

4. Bring the test flame close to the samole, and move
   the flame in the same plane 2 mm higher than that
   of sample cup every 2 ℃ of temperature rises.

5. Observe if the sample takes the fire from test flame.

Calculation Method (if necessary, please include correction method):

If the barometic pressure of the test is lower than
715 mmHg, add the following correction value to the
measured temperature.

    715 – 635 mmHg --- 2.8 ℃
    634 – 550 mmHg --- 5.5 ℃

Interpretation/Precision & Accuracy :

repeatability   < 8 ℃
reproducibility   < 16 ℃

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Flash Point by Pensky-Martens Closed Tester

Abstracted Scope (200 words):

These test methods are primarily used to determine the flash point of
liquids that either have viscosities more than 5.5 cSt at 40°C, or 9.5 cSt
or more at 25°C, or that have a flash point greater than 93°C, or that
contain suspended solids or tend to·form a surface skin under test
conditions.

The flash point of liquids having viscosities and flash points below these
values that do not contain suspended solids or have a tendency to form a
skin should be determined using ASTM Method D 56.

Referred Document:

ASTM D 93   Standard Test Methods for Flash Point by Pensky-Martens
            Closed Tester

Principle of Measurement:

The sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite.

Apparatus:
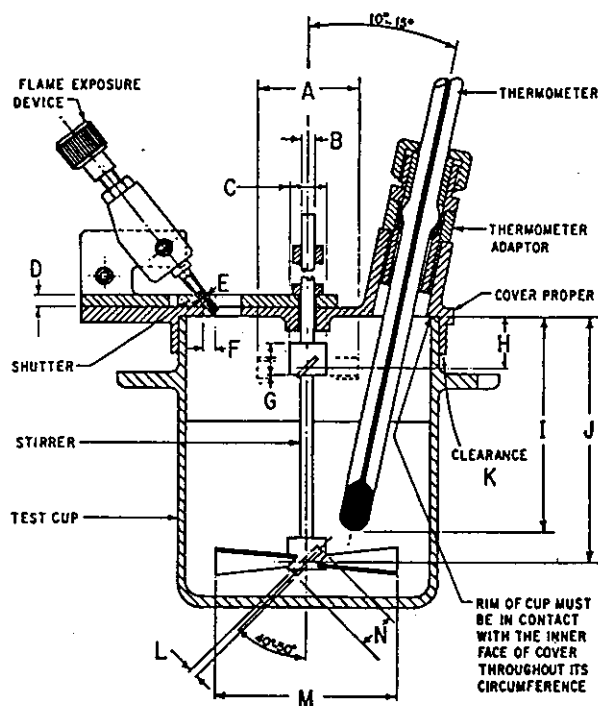
Sampling:

Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily or make a transfer unless the sample temperature is at least 8°C below the expected flash point. Do not use samples from leaky containers and do not store samples in plastic (polyethylene, polypropylene, etc.) containers since volatile materials may diffuse through their walls.

Conditioning:

Samples containing dissolved or free water may be dehydrated with calcium chloride and then filtering through filter paper or dry absorbent cotton.

Warming the sample is permitted, but it shall not be heated for prolonged periods or above a temperature 17°C below its expected flash point.

Procedures:

Method A: Basic Procedure

1. Clean and dry the cup and its accessaries, fill the cup to the filling
   mark, place the lid on the cup, and set the cup in the stove. Insert
   the thermometer, light and adjust the test flame, and heat the cup at
   5 to 6°C/min with the stirrer in the cup stirring at 90 to 120 rpm.
   Bring to 15°C, or 11°C below the flash point, whichever is lower.

2. If the sample is known to have a flash point of 110°C or below, apply
   the test flame by opening the shutter of the apparatus when the sample
   temperature is 17 to 28°C below the expected flash point and at every
   1°C increment until flashing occurs. Do not stir when applying the flame

3. For samples with flash points above 110°C, apply the test flame at 17 to
   28°C below the expected flash point and at each 2°C increment.

Method B: Flash Point of Suspensions of Solids and Highly Viscous Liquids

Bring the material to be tested to a temperature of 15°C, or 11°C lower than
the estimated flash point, whichever is lower as in Method A before heating
Turn the stirrer at 250 rpm and raise the temperature throughout the
duration of the test at a rate of 1 to 1.5°C/min. With the exception of
these requirements for rates of stirring and heating, proceed as with
Method A.

Calculation Method (if necessary, please include correction method):

When the pressure differs from 760 mm Hg, correct the flash point as
follows:

Corrected flash point = C + 0.25 (101.3 - p)   or

Corrected flash point = C + 0.033 (760 - P)   where

C = observed flash point, °C,
P = ambient barometric pressure, mm Hg, and
p = ambient barometric pressure, kPa.

Interpretation/Precision & Accuracy: Method A

Repeatability (95% probability): Duplicate results by the same operator
should be considered suspect if they differ by more than the following:
 2°C for suspensions of solids from 35 to 43°C; 2°C for all others at
 104°C and under; 5.5°C for all others above 104°C. 5°C for Method B.

Reproducibility (95% probability): The results submitted by each of two
laboratories should be considered suspect if the two results differ by
more than the following amounts:
 3.5°C for suspensions of solids from 35 to 43°C; 3.5°C for all others at
 104°C and under; 8.5°C for all others above 104°C. 10°C for Method B.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Flash Point by Tag Closed Tester

Abstracted Scope (200 words):

This test method covers the determination of the flash point, by Tag closed tester, of liquids with a viscosity of below 5.5 cSt at 40°C, or below 9.5 cSt at 25°C, and a flash point below 93°C with the exception of cutback asphalts, those liquids which tend to form a surface film under test conditions, and materials which contain suspended solids.

Liquids having viscosities more than 5.5 cSt at 40°C, or 9.5 cSt at 25°C, that contain suspended solids, or have a tendency to form a surface film while under test, should be tested in accordance with ASTM Method D 93.

Referred Document:

ASTM D 56 Standard Test Method for Flash Point by Tag Closed Tester

Principle of Measurement:

The sample is placed in the cup of the tester and, with the lid closed, heated at a slow constant rate. A small flame of specified size is directed into the cup at regular intervals. The flash point is taken as the lowest temperature at which application of the test flame causes the vapor above the sample to ignite.

Apparatus:
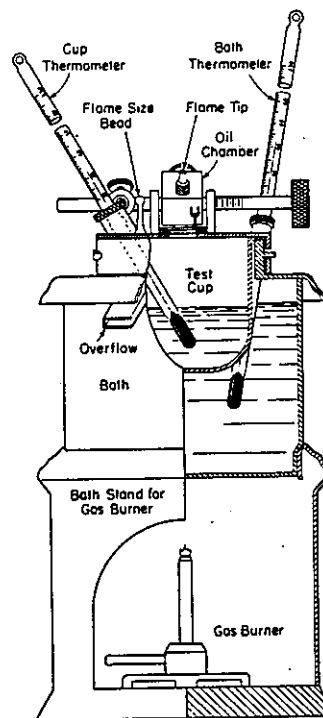
FIG. 1 Tag Closed Flash Tester

Sampling:

Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Containers shall not be opened unnecessarily and transfers shall not be made unless the sample temperature is at least 11°C below the expected flash point. Since volatile material may diffuse through the walls of the bottle, samples should not be stored in plastic (polyethylene, polypropylene, etc.) bottles.

Conditioning:

Procedures:

1. Maintain the temperature of the liquid in the bath at least 11°C below the expected flash point at the time of introduction of the sample into the test cup.

2. Using a graduate and taking care to avoid wetting the sample cup above the final liquid level, measure 50 ml of the sample into the cup. The sample and graduate shall be precooled, if necessary, so that the sample temperature at time of measurement is either 27°C or 11°C below the expected flash point, whichever is lower. Destroy bubbles on the surface of the sample, clean and attach the cover, and place the cup into the temperature bath.

3. If the flash point is known to be below 60°C, apply and adjust the heat so that the temperature of the sample will rise at 1°C/min. When the temperature of the sample in the test cup is 5°C below its expected flash point, apply the test flame, and repeat the application of the test flame after each 0.5°C rise in the temperature of the sample.

4. If the flash point is known to be 60°C or higher, heat the sample at a rate of 3°C/min. Apply the test flame when the sample temperature is 5°C below the expected flash point and repeat application of the test flame after each 1°C rise in temperature.

5. When the test flame application causes a distinct flash in the interior of the cup, record the sample temperature as the flash point.

Calculation Method (if necessary, please include correction method):

Correction for Barometric Pressure:

Corrected flash point = C + 0.25 (101.3 - p)   or

Corrected flash point = C + 0.033 (760 - P)   where

C = observerd flash point, °C
P = ambient barometric pressure, mm Hg
p = ambient barometric pressure, kPa.

Interpretation/Precision & Accuracy:

Repeatability: The difference between successive test results obained by by the same operator with the same apparatus under constant operating conditions on identical test material would exceed the following values only 1 case in 20 (95% confidence level):
    1.1°C for flash points < 60°C and 1.7°C for flash points from 60 to 93°C.

Reproducibility: The difference between two single and independent results, obtained by different operators working in different laboratories on identical material would exceed the following values only 1 case in 20:
    3.3°C for < 13°C, 2.2°C for 13 to 59°C, and 3.3°C for 60 to 93°C.

REPORTS ON HEAT TRANSFER MEDIUM

c (v)   OTHERS

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

   pH of Antifreezes, Antirusts, and Coolants

Abstracted Scope (200 words):

This method covers the determination of the pH of unused engine antirusts,
antifreezes, and coolants, in the concentrations provided before dilution,
and the determination of the pH of used or unused aqueous dilutions of the
concentrated products.

The readings of the pH meter in the case of the concentrated products are
not pH values according to the accepted definition, but are apparent pH
values that are useful in interpreting the condition of the product.

The method is not applicable to products in solid form.

The method is applicable to heat transfer fluids used in solar heating and
cooling applications.

Referred Document:

ASTM D 1287   Standard Test Method for pH of Engine Antifreezes, Antirusts,
              and Coolants

Principle of Measurement:

A sample, as received or after dilution with a specified volume of distilled water, is placed in a beaker or sample cup and the pH is measured with a pH meter and associated glass-saturated calomel electrode pair.

Apparatus:

A voltmeter or potentiometer that will operate with an accuracy of ± 0.005 V and a sensitivity of ± 0.002 V, over a range of at least ± 0.5 V, when the meter is used with calomel and glass electrodes and when the resistance between the electrodes falls within the range from 0.2 to 20 MΩ.

A pencil-type glass electrode 125 to 180 mm in length and 8 to 14 mm in diameter.

A pencil-type calomel electrode 125 to 180 mm in length and 8 to 14 mm in diameter. The electrode shall contain the necessary mercury, calomel, and electrical connection to the mercury, all arranged in a permanent manner. The electrode shall be filled almost to capacity with saturated KCl electrolyte and shall be equipped with a stoppered port through which the electrolyte may be replenished.

A variable-speed mechanical stirrer equipped with a glass propeller-type stirring paddle or a suitable magnetic stirrer.

Standard solutions of the following for use in calibration:
   Potassium Hydrogen Phthalate (0.05 M, pH of 4.01 at 25°C)
   Neutral Phosphate (0.025 M KH_2PO_4 and Na_2HPO_4, pH of 6.86 at 25°C)
   Potassium Chloride Electrolyte - prepare a saturated solution of KCl
   Borax (0.01 M, pH of 9.18 at 25°C)
   Alkaline Phosphate (0.01 M trisodium phosphate, pH of 11.72 at 25°C)

Sampling:

Sample material in accordance with ASTM Method D 1176.

Conditioning:

Procedures:

1. Clean, prepare, and maintain the electrodes as specified in the test
   method. Calibrate using buffer solutions in the appropriate range.

2. Use either concentrated samples or samples diluted to the desired
   concentration and thoroughly mixed.

3. Immerse the calomel and glass electrode tips in the test solution and
   agitate with the stirrer.

4. Allow the system to come to equilibrium and record the pH value.

5. Remove and wash the electrodes with distilled water and immerse them in
   distilled water until the next determination is made. Remove any
   deposits that form on the glass electrodes with an appropriate solvent.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

Repeatability: Duplicate results by the same operator should be considered
suspect if they differ by more than 0.1 pH units

Reproducibility: The results submitted by two or more laboratories should
be considered suspect if they differ by more than 0.2 pH units.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

      Reserve Alkalinity of Antifreeze, Antirusts, and Coolants

Abstracted Scope (200 words):

This test method provides for the determination of the reserve alkalinity
of unused antifreezes, coolants and liquid antirusts in their concentrated
form, as received without dilution, and the reserve alkalinity of both used
and unused aqueous dilutions of the concentrated materials and solid
antirusts.

Reserve alkalinity is a term applied to antifreeze, coolants, and antirusts
to indicate the amount of alkaline inhibitors present in the product.

The method is applicable to heat transfer fluids used in solar heating and
cooling applications.

Referred Document:

ASTM D 1121   Standard Test Method for Reserve Alkalinity of Engine
              Antifreeze, Antirusts, and Coolants

Principle of Measurement:

Reserve alkalinity is the number of milliliters, to the nearest 0.1 mL, of
0.1 N hydrochloric acid required for the titration to a pH of 5.5 of a
10-mL sample of an undiluted, unused antifreeze, antirust, or coolant
additive, and unused or used solutions thereof.

Apparatus:

A voltmeter or potentiometer that will operate with an accuracy of ± 0.005 V
and a sensitivity of ± 0.002 V, over a range of at least ± 0.5 V, when the
meter is used with calomel and glass electrodes and when the resistance
between the electrodes falls within the range from 0.2 to 20 $M\Omega$.

A pencil-type glass electrode 125 to 180 mm in length and 8 to 14 mm in
diameter.

A pencil-type calomel electrode 125 to 180 mm in length and 8 to 14 mm in
diameter. The electrode shall contain the necessary mercury, calomel, and
electrical connection to the mercury, all arranged in a permanent manner.
The electrode shall be filled almost to capacity with saturated KCl
electrolyte and shall be equipped with a stoppered port through which the
electrolyte may be replenished.

A variable-speed mechanical stirrer equipped with a glass propeller-type
stirring paddle or a suitable magnetic stirrer, and a 50-mL buret.

Standard solutions for use in calibration and titration as follows:
  Potassium Hydrogen Phthalate Buffer (0.05 M, pH of 4.01 at 25°C)
  Phosphate Buffer (0.025 M KH_2PO_4 and Na_2HPO_4, pH of 6.86 at 25°C)
  Potassium Chloride Electrolyte - prepare a saturated solution of KCl
  Hydrochloric Acid (0.100 N)

Sampling:

Sample material in accordance with ASTM Method D 1176.

Dilute samples to the desired concentration and thoroughly mix.

Conditioning:

Procedures:

1. Maintain, prepare, and clean the calomel and glass electrodes as specified in the test method. Calibrate the electrodes using the buffer solutions.

2. Place the beaker containing the test solution on the titration stand, adjust so that the lower half of each electrode is immersed, and adjust the stirrer so that there is vigorous stirring, without spattering.

3. Fill the buret with 0.1 N HCl, record the initial pH and titrate to a pH of about 7. Then adjust the buret so that the end point of pH 5.5 is approached using a slow, dropwise titration rate.

4. Record the volume of acid used, acid normality, and sample size.

5. Switch the pH meter to standby and rinse the electrodes with water and dry with clean material before the next determination. When the pH meter is not in use, immerse the electrodes in distilled water.

Calculation Method (if necessary, please include correction method):

Percent Depletion = $100 [(RA_1/C_1 - RA_2/C_2)/(RA_1/C_1)]$,   where:

RA_1 = initial reserve alkalinity,

RA_2 = final reserve alkalinity,

C_1 = initial antifreeze or additive concentration in percent, and

C_2 = final antifreeze or additive concentration in percent.

Interpretation/Precision & Accuracy:

Repeatability: Duplicate results by the same operator should be considered suspect if they differ by more than 0.2 mL.

Reproducibility: The results submitted by two or more laboratories should be considered suspect if they differ by more than 0.2 mL.

REPORTS ON HEAT TRANSFER MEDIUM

d-(i) STABILITY OF CHEMICAL COMPOSITION AND STRUCTURE
      DURING THE APPLIED TEMPERATURE ELEVATION AND HEAT
      CYCLES

Date : 02/25/86   Reporter's Name : Mitsuyuki Koga

Country : Japan

Testing Title :

Testing Method for Thermal Stability of Chemical
Structure of Propylene Glycol.

Abstracted Scope (200 words) :

This method is relative to stability of chemical
structure of propylene glycol under controlled
laboratory conditions.

The test is carried out under condition of
deaeration in order to separate the effect of oxidative
decomposition of propylene glycol solution.

Referred Document :

Simulated Service Testing for Corrosion in Solar
Heating and Cooling Systems, NBSIR 81-2339,
June 1981.

Principle of Measurement:

Thermal decomposition of propylene glycol is evaluated on the basis of the solution PH or infrared spectrum changes.

Apparatus:
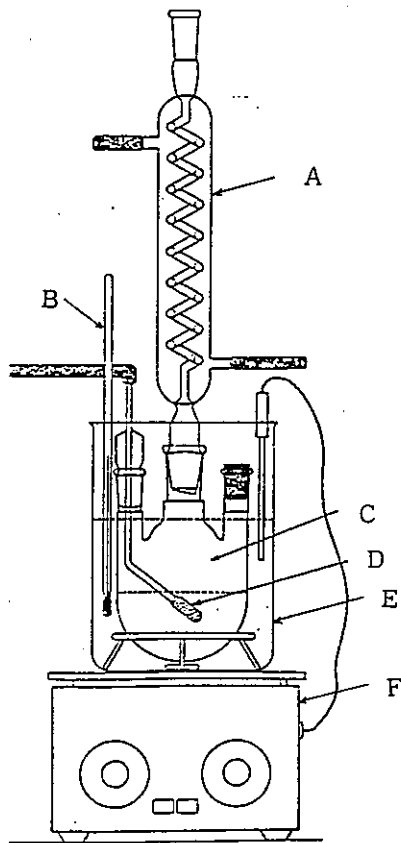
A: Condenser

B: Thermometer

C: Reaction Flask

D: Gas Dispersion Tube

E: Silicone Oil Bath

F: Hot plate

Calibration:

Sampling:

Propylene glycol is diluted to 50 volume percent with city or soft water.

Conditioning:

Temperature: 105 ± 1 ℃
Solution Volume : 700 mℓ
Deaeration Condition : purified N₂ gas
Test Duration : 16 weeks (continuously)

Procedures:

1. Dilute the propylene glycol to 50 volume percent with city water or soft water and measure both initial PH and infrared spectrum.

2. Pour 700 mℓ of the test solution into the round bottomed flask and place this flask in the silicone oil bath.

3. Fit the condenser and gas dispersion tube to the flask, make deaeration with high purity N_2, and pass water through the condenser.

4. Maintain the test solution on the hot plate at a given temperature within ±1 ℃.

5. Remove aliquots of liquid biweekly for analysis.

6. At the end of the test, measure both final PH and infrared spectrum.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

The degree of PH decrease and of the infrared spectrum change of the test solution indicate that of the degradation of propylene glycol solution.

REPORTS ON HEAT TRANSFER MEDIUM

d-(ii) CORROSION EFFECT ON THE MATERIALS USED IN THE
       TRANSFER LOOP

Date: 02/26/86   **Reporter's Name:** Mitsuyuki Koga

Country: Japan

Testing Title:

   Corrosion Test for Heat Transfer Medium in
   Glassware.

Abstracted Scope (200 words):

   This method covers a simple beaker-type procedure
   for evaluating corrosive properties of heat transfer
   medium on metal specimens under controlled laboratory
   conditions.

   The test is carried out in accordance with JIS K 2234
   Engine Antifreeze Coolant.

Referred Document:

   JIS K 2234 Engine Antifreeze Coolant.

Principle of Measurement:

The corrosion inhibitive properties
of the test solution are evaluated on the basis of
the weight changes incurred by the specimens.

Apparatus:

UNIT : mm
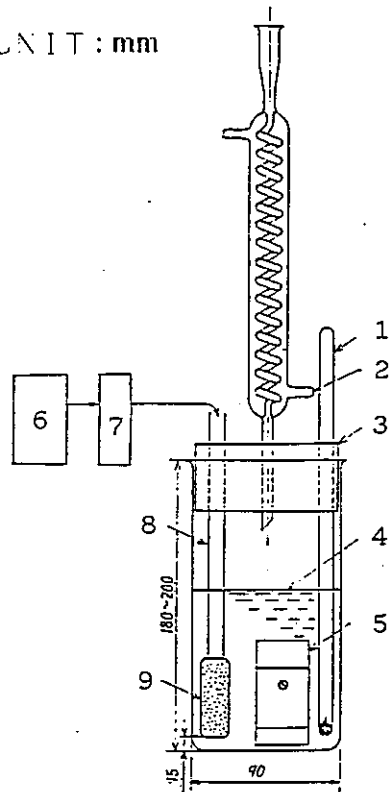
1 : Thermometer

2 : Cooling Water

3 : Rubber Stopper

4 : Liquid Level

5 : Metal Specimens

6 : Dry Air

7 : Flowmeter

8 : Aerator Tube

9 : Gas Dispersion pore

Calibration:

Sampling:
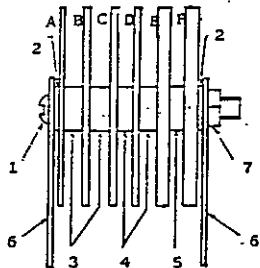
See Table 1

Conditioning:

Test Solution Preparation	50 V/V% city water and heat transfer medium
Test Temperature	88 ± 2 ℃
Test Duration	336h (continuously)
Aeration Rate	100 ± 10ml per min

Procedures :

1. Clean the metal specimens by hand sanding with 320 paper, then rinse with ethyl alcohol, dry, weigh, and store in a desiccator until use.
2. Pour 750ml of the prepared test solution into the 1000ml beaker and place the specimens in it.
3. Fit the condenser and aeration tube to the beaker, and set the aeration rate at 100ml/min, using a flowmeter.
4. Raise the temperature of the test solution to 88°C, pass water through the condenser.
5. At the end of the test, immediately disassemble specimens and brush with a soft bristle brush and water to remove loosely held corrosion products, and rinse with water.
6. Stainless Steel, Cast Iron, Steel and Zinc —— Remove adherent materials by means of a brass scraper or a brass bristle brush, rinse with water, then rinse with ethyl alcohl, dry, and weigh.
7. Copper and Brass — Dip in a 1+1 mixture of concentrated HCL (sp gr 1.19) with water for 15s to remove tarnish films, rinse with tap water to remove acid, then rinse with ethyl alcohl, dry, and weigh.

Calculation Method (if necessary, please include correction method) :

Correct corrosion weight changes of individual specimens to nearest 0.1mg for each test.

$$C = \frac{W' - W}{S}$$

where
C = corrosion weight change (mg/cm^2),
W = weight of specimen befor test (mg)
W'= weight of specimen after test (mg),
S = area of specimen (cm^2)

Interpretation/Precision & Accuracy :

Table 1

	Materials	Size, Dimensions
1	Brass bolt covered with insulating sleeve	$\phi 5 \times 60$ mm
2	Insulating spacer	$\phi 12 \times \phi 6.5 \times 1.5$ mm
3	Steel spacer	$\phi 12 \times \phi 6.5 \times 4.5$ mm
4	Insulating spacer	$\phi 12 \times \phi 6.5 \times 4.5$ mm
5	Brass spacer	$\phi 12 \times \phi 6.5 \times 4.5$
6	Brass leg	$50 \times 25 \times 1.6$ mm
7	Brass nut	$\phi 5$
A	Stainless steel	$50 \times 25 \times 0.3$ mm
B	Steel	$50 \times 25 \times 1.6$ mm
C	Cast iron	$50 \times 25 \times 3.0$ mm
D	Zinc	$50 \times 25 \times 1.0$ mm
E	Brass	$50 \times 25 \times 1.6$ mm
F	Copper	$50 \times 25 \times 1.6$ mm

Note : The metal test specimens shall be drilled through
the center with 6.5mm drill to accommodate 60mm
brass bolt covered with a thin ----- walled
insulating sleeve.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Laboratory Screening of Metallic Containment Materials for Use with
Liquids in Solar Heating and Cooling Systems

Practice B: Heat-Rejecting Surface Test at Atmospheric Pressure

Abstracted Scope (200 words):

Practice B is intended to simulate deterioration of metal containment
materials resulting from heat transfer through the containment material
into the heat transfer fluid, e.g., in solar collectors.

The practice is applicable to flat surfaces. If curvature of the heat-
transfer surface is considered to be significant, Practice F of ASTM E 712
should be considered.

The apparatus, as typically constructed, is open to the atmosphere.
Therefore, results of this test may not be applicable to closed systems.

Practice B is one of six practices described in ASTM E 712 for evaluating
the corrosion performance of metallic containment materials under
conditions similar to those that may occur in solar heating and cooling
systems. These conditions include (1) operating full flow, (2) stagnant
full, (3) stagnant partial full, and (4) stagnant empty. The test results
relate to the performance of the material only as part of a metal/fluid
pair.

Referred Document:

ASTM E 712   Standard Practice for Laboratory Screening of Metallic
              Containment Materials for Use with Liquids in Solar Heating
              and Cooling Systems

Principle of Measurement:

Mass loss, appearance changes, and localized corrosion resulting from the exposure of a heated test specimen surface to heat transfer fluid are determined. Stirring may be used to simulate operating flow conditions.
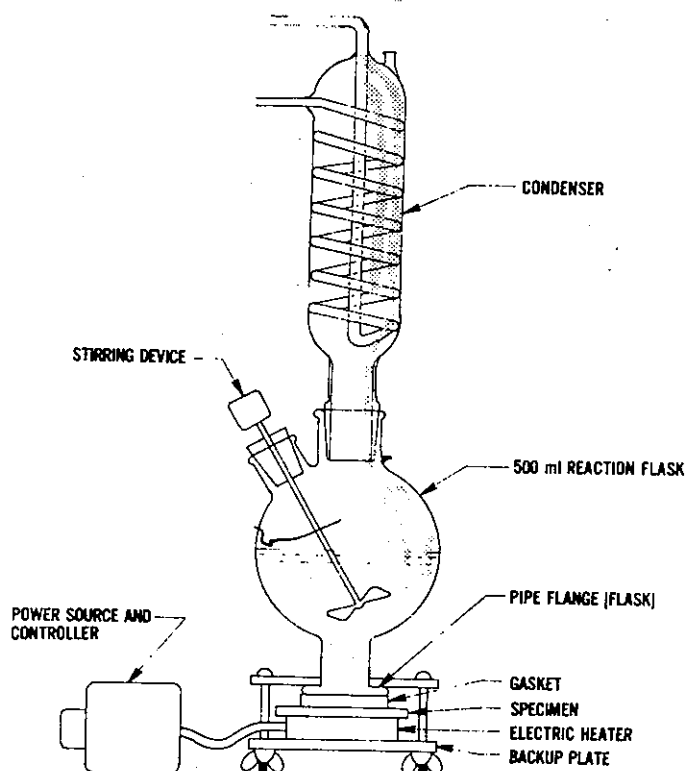
Apparatus:

FIG. 1   Schematic of Apparatus for Heat-Rejecting Surface Test

Sampling:

Test specimen materials shall be representative of those used in actual solar heating and cooling systems. For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, such as a mill finish, closely resembling the one that would be used in service, will yield the most significant results. Standard surface finishes may be preferred for more detailed studies of the metal or the environment.

Conditioning:

Surface cleaning with solvent or water-based cleaners, or abrasives and removal of edge damage, prior to corrosion testing. Scrubbing and electrolytic or chemical cleaning after corrosion testing.

Procedures:

Clean the specimens and weigh to an accuracy of 0.1 mg immediately prior to testing.

Mount the specimen between the gasket and heater, and secure to the reaction flask.

Fill the flask with 250 mL of the test fluid. Mount the condenser, temperature-measuring device, and stirring device, as necessary.

It is permissible to select any schedule of times and temperatures. It is recommended that consideration be given to anticipated field operation in the selection of a schedule of heating, holding, and cooling cycles. It is recommended that a test period of no less than 30 days be used, and that studies be conducted to determine any time dependence of corrosion attack.

It is recommended that stirring be used for simulation of operating full-flow conditions.

Following exposure, dry and weigh the specimens after cleaning and rinsing.

Determine mass loss per unit area, examine at 10x magnification for localized attack, and document changes in the specimen surface and the heat transfer fluid.

Calculation Method (if necessary, please include correction method):

$$Rmdd = 100,000 \ [(Wo - Wt)/AT] \qquad or \qquad Rmpy = 393.7 \ [(Wo - Wt)/ATD]$$

Rmdd = corrosion rate, $mg/dm^2 \cdot day$	Rmpy = corrosion rate, mils/year
Wo = original mass, g	Wo = original mass, g
Wt = final mass, g	Wt = final mass, g
A = area, cm^2, and	A = area, cm^2
T = duration, days.	T = duration, years, and
	D = density, g/cm^3.

Interpretation/Precision & Accuracy:

Available data are not adequate for the development of precision and accuracy statements.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Laboratory Screening of Metallic Containment Materials for Use with
Liquids in Solar Heating and Cooling Systems

Practice A: Basic Immersion Test at Atmospheric Pressure

Abstracted Scope (200 words):

Practice A is concerned with the interaction of metal and fluid when both
are at the same temperature with no heat transfer from one to the other.
It is regarded as useful for plumbing, pumps, tanking, etc., but of less
significance, taken by itself, for screening collector absorber materials.

Practice A is intended to provide a simple, rapid exposure test for
evaluation of metal and fluid interaction. The apparatus, as typically
constructed, is open to the atmosphere. Therefore, the results of this
test may not be applicable to closed systems.

The practice provides for suspending a test specimen in a heated fluid
which may be stirred to simulate flow conditions. Aeration or deaeration
of the fluid by gas sparging is provided as an option.

Practice A is one of six practices described in ASTM E 712 for evaluating
the corrosion performance of metallic containment materials under
conditions similar to those that may occur in solar heating and cooling
systems. These conditions include (1) operating full flow, (2) stagnant
full, (3) stagnant partial full, and (4) stagnant empty. The test results
relate to the performance of the material only as part of a metal/fluid
pair.

Referred Document:

ASTM E 712   Standard Practice for Laboratory Screening of Metallic
             Containment Materials for Use with Liquids in Solar Heating
             and Cooling Systems

Principle of Measurement:

Mass loss, appearance changes, and localized corrosion resulting from the immersion of test specimens in a heated fluid are determined. The fluid may be stirred and aerated to simulate fluid flow and aeration.

Apparatus:

The vessel is typically a 1000-mL beaker or reaction flask of heat-resistant glass. Provision is made for closing the top of the reaction vessel while providing openings for temperature-measuring devices, reflux condenser, and stirring device, as necessary. The specimen may be suspended in a cradle of nonmetallic material or supported by a rack either constructed of a nonmetallic material or insulated so as to prevent galvanic interaction of specimen and rack.

The vessel may be heated by mantle, hot plate, or bath. Localized heating may result from the use of a hot plate.

The fluid may be stirred to simulate flow conditions. For those fluids in which aeration or deaeration can be simulated by gas sparging, the use of such gas sparging is optional. For low boiling fluids, the use of a cold trap is recommended.

Sampling:

Test specimen materials shall be representative of those used in actual solar heating and cooling systems. For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, such as a mill finish, closely resembling the one that would be used in service, will yield the most significant results. Standard surface finishes may be preferred for more detailed studies of the metal or the environment.

Conditioning:

Surface cleaning with solvent or water-based cleaners, or abrasives and removal of edge damage, prior to corrosion testing. Scrubbing and electrolytic or chemical cleaning after corrosion testing.

Procedures:

Clean the specimens and weigh to an accuracy of 0.1 mg immediately prior to testing.

Suspend the specimen in a cradle or attach to a specimen rack and place in the test vessel.

Fill the vessel with sufficient heat transfer fluid to cover the specimen. A volume of about 500 mL is recommended.

Mount the condenser, temperature measuring device, stirring device, and heating device, as necessary.

Typically, the vessel is then heated to the desired test temperature and held at that temperature for the duration of the test. However, it is permissible to select any schedule of times and temperatures. It is recommended that a test period of no less than 30 days be used, and that studies be conducted to determine any significant time dependence of corrosion attack.

Then dry and weigh the samples after cleaning and thorough rinsing.

Determine mass loss per unit area, examine at 10x magnification for localized attack, and document changes in the specimen surface and the heat transfer fluid.

Calculation Method (if necessary, please include correction method):

$$Rmdd = 100,000 \ [(Wo - Wt)/AT] \qquad or \qquad Rmpy = 393.7 \ [(Wo - Wt)/ATD]$$

Rmdd = corrosion rate, $mg/dm^2.day$
Wo = original mass, g
Wt = final mass, g
A = area, cm^2, and
T = duration, days.

Rmpy = corrosion rate, mils/year
Wo = original mass, g
Wt = final mass, g
A = area, cm^2
T = duration, years, and
D = density, g/cm^3.

Interpretation/Precision & Accuracy:

Available data are not adequate for the development of precision and accuracy statements.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Laboratory Screening of Metallic Containment Materials for Use with
Liquids in Solar Heating and Cooling Systems

Practice D: Repeated Dip Dry Test at Atmospheric Pressure

Abstracted Scope (200 words):

Practice D is intended to simulate alternating wetting and drying
conditions. This procedure, as customarily performed, is open to the
atmosphere. Therefore, the results of this procedure may not be applicable
to closed systems.

Practice D is one of six practices described in ASTM E 712 for evaluating
the corrosion performance of metallic containment materials under
conditions similar to those that may occur in solar heating and cooling
systems. These conditions include (1) operating full flow, (2) stagnant
full, (3) stagnant partial full, and (4) stagnant empty. The test results
relate to the performance of the material only as part of a metal/fluid
pair.

Referred Document:

ASTM E 712   Standard Practice for Laboratory Screening of Metallic
              Containment Materials for Use with Liquids in Solar Heating
              and Cooling Systems

Principle of Measurement:

Mass loss, appearance changes, and localized corrosion resulting from the exposure of test specimens to alternating wetting and drying conditions are determined.

Apparatus:

The apparatus shall consist of a hydraulically or mechanically operated arm from which the specimen is suspended in a glass cradle.

The arm lowers the specimen into a beaker containing the test fluid and then raises the specimen into a device that will provide for drying of the specimen.

Selection of the drying device shall take into consideration the possible degradation effects related to the method of drying. For example, degradation of the test fluid by infrared radiation from heat lamps, or effects of atmospheric exposure.

A system of automatic control shall provide repeated cycles of selected periods of immersion and selected periods of drying.

Sampling:

Test specimen materials shall be representative of those used in actual solar heating and cooling systems. For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, such as a mill finish, closely resembling the one that would be used in service, will yield the most significant results. Standard surface finishes may be preferred for more detailed studies of the metal or the environment.

Conditioning:

Surface cleaning with solvent or water-based cleaners, or abrasives and removal of edge damage, prior to corrosion testing. Scrubbing and electrolytic or chemical cleaning after corrosion testing.

Procedures:

Clean the specimens and weigh to an accuracy of 0.1 mg immediately prior to testing.

Suspend the specimen from the movable arm in the cradle.

Align a beaker of the test fluid under the specimen and activate the drying device and cycling device.

Any cycle of dip and dry times may be selected. It is recommended that studies be conducted to determine any time dependence of corrosion attack and that a test period of no less than 30 days be used.

Following exposure, dry and weigh the specimens after cleaning and rinsing.

Determine mass loss per unit area, examine at 10x magnification for localized attack, and document changes in the specimen surface and the heat transfer fluid.

Calculation Method (if necessary, please include correction method):

$$Rmdd = 100,000 \ [(Wo - Wt)/AT] \quad \text{or} \quad Rmpy = 393.7 \ [(Wo - Wt)/ATD]$$

Rmdd = corrosion rate, $mg/dm^2.day$	Rmpy = corrosion rate, mils/year
Wo = original mass, g	Wo = original mass, g
Wt = final mass, g	Wt = final mass, g
A = area, cm^2, and	A = area, cm^2
T = duration, days.	T = duration, years, and
	D = density, g/cm^3.

Interpretation/Precision & Accuracy:

Available data are not adequate for the development of precision and accuracy statements.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Laboratory Screening of Metallic Containment Materials for Use with
Liquids in Solar Heating and Cooling Systems

Practice C: High-Pressure Test.

Abstracted Scope (200 words):

Practice C is intended to simulate the conditions of high temperature and
pressure in a pressurized system under stagnant full, stagnant partial
full, and stagnant empty conditions.

This practice can provide for test conditions simulating operation of
solar energy systems closed to the atmosphere, e.g., closed loop collector
systems.

Practice C is one of six practices described in ASTM E 712 for evaluating
the corrosion performance of metallic containment materials under
conditions similar to those that may occur in solar heating and cooling
systems. These conditions include (1) operating full flow, (2) stagnant
full, (3) stagnant partial full, and (4) stagnant empty. The test results
relate to the performance of the material only as part of a metal/fluid
pair.

Referred Document:

ASTM E 712   Standard Practice for Laboratory Screening of Metallic
             Containment Materials for Use with Liquids in Solar Heating
             and Cooling Systems

Principle of Measurement:

Mass loss, appearance changes, and localized corrosion resulting from the exposure of test specimens in a heated pressure vessel containing heat transfer fluid. The specimens may be immersed, partially immersed, or suspended in the vapor phase.

Apparatus:

This test is performed using an autoclave or comparable device, capable of producing the required conditions of temperature and pressure.

Specimens shall be supported independently from one another by a rack and may be suspended in cradles or mounted directly on the rack.

Supporting materials shall be selected so as to avoid interaction with the specimens or test fluid and mounting shall be accomplished so as to avoid any galvanic interaction.

Sampling:

Test specimen materials shall be representative of those used in actual solar heating and cooling systems. For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, such as a mill finish, closely resembling the one that would be used in service, will yield the most significant results. Standard surface finishes may be preferred for more detailed studies of the metal or the environment.

Conditioning:

Surface cleaning with solvent or water-based cleaners, or abrasives and removal of edge damage, prior to corrosion testing. Scrubbing and electrolytic or chemical cleaning after corrosion testing.

Procedures:

Clean the specimens and weigh to an accuracy of 0.1 mg immediately prior to testing.

Mount the specimens in a cradle or on the rack.

Place the rack in the test chamber and fill the chamber with the test fluid so that the specimen is totally immersed, partially immersed, or suspended in the vapor phase, as desired.

Seal the chamber.

Run the test at the desired schedule of time, temperature, and pressure conditions.

It is recommended that a test period of no less than 30 days be used, and that studies be conducted to determine any significant time dependence of corrosion attack.

Dry and weigh the samples after cleaning and thorough rinsing.

Determine mass loss per unit area, examine at 10x magnification for localized attack, and document changes in the specimen surface and the heat transfer fluid.

Calculation Method (if necessary, please include correction method):

$$Rmdd = 100,000 \ [(Wo - Wt)/AT] \qquad \text{or} \qquad Rmpy = 393.7 \ [(Wo - Wt)/ATD]$$

Rmdd	= corrosion rate, $mg/dm^2.day$	Rmpy	= corrosion rate, mils/year
Wo	= original mass, g	Wo	= original mass, g
Wt	= final mass, g	Wt	= final mass, g
A	= area, cm^2, and	A	= area, cm^2
T	= duration, days.	T	= duration, years, and
		D	= density, g/cm^3.

Interpretation/Precision & Accuracy:

Available data are not adequate for the development of precision and accuracy statements.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Simulated Service Testing for Corrosion of Metallic Containment Materials
for Use with Heat-Transfer Fluids in Solar Heating and Cooling Systems

Practice C: Field Exposure Test of Components or Subcomponents

Abstracted Scope (200 words):

Practice C provides for the use of a field test to evaluate the fluid
corrosion resistance of materials used in components and simulated
subcomponents to contain heat transfer fluids. The field test simulates
various conditions of full operation and stagnation.

Practice C is one of three practices described in ASTM E 745 for evaluating
the corrosion performance of metallic containment materials under
conditions simulating field service.

Referred Document:

ASTM E 745 Standard Practices for Simulated Service Testing for Corrosion
            of Metallic Containment Materials for Use with Heat Transfer
            Fluids in Solar Heating and Cooling Systems

Principle of Measurement:

Deterioration, including general corrosion, localized corrosion, erosion, and other evidence of physical changes resulting from the field testing of components and subcomponents containing heat transfer fluid is determined. The field testing simulates both full operation and stagnation conditions.

Apparatus:

The apparatus shall consist of a storage tank, a pump, suitable plumbing, a process controller, monitoring equipment, and the solar collector test specimen.

The tank shall be of sufficient size to receive the thermal output of the collector without reaching temperatures in excess of those expected in service. Typical system tank materials should be used.

The pump shall be constructed of typical or inert materials and shall be sized to provide a flow rate typical of anticipated service conditions.

The system shall be plumbed with the same materials as would be typically employed in service conditions. Materials used and their attachment to the absorber shall be reported.

The test shall be operated by a controller, typically a timer, that will activate the pump on a daily cycle of operation.

Provision shall be made for monitoring the temperature of the absorber panel, other parts of the collector, and the fluid at various points in the system.

Several test collectors may be operated at the same time on a single system if the collectors are plumbed in parallel and there is no likelihood of interaction through alteration of fluid chemistry.

Sampling:

The test specimen shall consist of a full-size solar collector, or a subsize unit constructed to simulate a full-size unit, containing a metallic absorber.

The panel and the collector shall be of materials and construction typical of actual use.

Conditioning:

None

Procedures:

Assemble the apparatus and charge with heat-transfer fluid.

Mount the collector with the aperture aligned with true south and tilted at a angle equal to the latitude of the test site.

Any schedule of operation is permitted; however, it is recommended that a schedule of full operation and stagnation cycles representative of anticipated field operation be used. For example:
1. Pump the system overnight to cool the fluid, except under conditions that would lead to freezing of the fluid.
2. Continue pumping through the morning until solar noon.
3. Interrupt pumping at solar noon. The fluid may be allowed to remain in the panel or to draindown, depending on the condition being evaluated.
4. Resume pumping 1-1/2 h after solar noon.
5. Continue pumping thereafter until the next cycle.

It is recommended that the test be operated on the specified daily cycle for at least 1 year. Panel and fluid temperatures should be measured periodically to characterize conditions during the cycle of operation.

Seal leakage when it occurs and document the occurrence.

At the end of the test period, open the panel and document evidence of general or localized corrosion, erosion or other changes.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

Available data are not adequate for the development of precision and accuracy statements.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Simulated Service Testing for Corrosion of Metallic Containment Materials
for Use with Heat-Transfer Fluids in Solar Heating and Cooling Systems

Practice B: Laboratory Exposure Test of Components or Subcomponents

Abstracted Scope (200 words):

Practice B provides a laboratory simulation of various operating
conditions of a solar heating and cooling system utilizing a component or
simulated subcomponent construction, and provides for heating of the fluid
by the containment material.

By proper selection of test conditions, a wide range of operating
conditions may be evaluated. These conditions include: operating full
flow; stagnant empty, vented; stagnant, closed to atmosphere, non-
draindown; and stagnant, closed to atmosphere, draindown.

The practice does not restrict the selection of either the containment
material or the fluid for testing. Selection of materials and test
conditions should be representative of actual use.

Practice B is one of three practices described in ASTM E 745 for evaluating
the corrosion performance of metallic containment materials under
conditions simulating field service.

Referred Document:

ASTM E 745   Standard Practices for Simulated Service Testing for Corrosion
             of Metallic Containment Materials for Use with Heat Transfer
             Fluids in Solar Heating and Cooling Systems

Principle of Measurement:

Mass loss, appearance changes, and localized corrosion resulting from the testing of components or simulated subcomponents under heat transfer fluid conditions representative of those occurring in actual service are determined.

Apparatus:

The apparatus shall consist of a storage tank, a pump, suitable plumbing, a process controller, monitoring equipment, a refrigeration unit, and a suitable means of heating the metallic containment loop.

The tank shall be of sufficient size to receive the thermal output of the heating unit without reaching temperatures in excess of those expected in service. Typical system tank materials should be used.

The pump shall be constructed of typical or inert materials and shall be sized to provide a flow rate typical of anticipated service conditions.

The system shall be plumbed with the same materials as would be typically employed in service conditions. Materials used and their attachment to the test specimen shall be reported.

The test shall be operated by a controller, typically a timer, that will activate the pump on a daily cycle of operation.

Provision shall be made for monitoring the temperature of the exterior surface of the test specimens, other parts of the containment system, and the liquid in the tank at various points in the system.

Provision shall be made for aeration or deaeration of the heat transfer fluid.

Sampling:

Test specimen materials shall be representative of those used in actual solar heating and cooling systems.

The test specimen may be a tube or pipe, a section of flow channel cut from a panel or a simulated subcomponent. It is recommended that the specimen be constructed to permit weight loss measurements.

Conditioning:

Surface cleaning with solvent or water-based cleaners, or abrasives and removal of edge damage, prior to corrosion testing. Scrubbing and electrolytic or chemical cleaning after corrosion testing.

Procedures:

Specimens suitable for weight loss measurements shall be cleaned using solvent or water-based cleaners, or abrasives and weighed immediately prior to testing.

Assemble the apparatus and charge with heat-transfer fluid.

It is recommended that the test schedule selected reflect the range of conditions encountered in field service, including a combination of full operation and stagnation cycles.

It is recommended that the test be conducted on a specified cycle for at least 90 days. For example:
1. Initiate the cycle at the minimum expected service temperature.
2. Heat the containment system to a maximum service temperature over a 1-h period.
3. Maintain the containment system at the maximum service temperature temperature for 2 h under designated stagnation conditions.
4. Permit flow to resume and cool the containment system to the minimum service temperature over a period of 1 h.
5. Provide for periodic withdrawal of samples of the heat transfer fluid for chemical analysis.
6. At the end of the test period, disassemble the apparatus and visually inspect for indications of corrosion or accumulation of deposits. Determine mass loss where appropriate.

Calculation Method (if necessary, please include correction method):

$$Rmdd = 100,000 \ [(Wo - Wt)/AT] \quad \text{or} \quad Rmpy = 393.7 \ [(Wo - Wt)/ATD]$$

$Rmdd$ = corrosion rate, $mg/dm^2 \cdot day$	$Rmpy$ = corrosion rate, mils/year
Wo = original mass, g	Wo = original mass, g
Wt = final mass, g	Wt = final mass, g
A = area, cm^2, and	A = area, cm^2
T = duration, days.	T = duration, years, and
	D = density, g/cm^3.

Interpretation/Precision & Accuracy:

Available data are not adequate for the development of precision and accuracy statements.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Simulated Service Testing for Corrosion of Metallic Containment Materials for Use with Heat-Transfer Fluids in Solar Heating and Cooling Systems

Practice A: Laboratory Exposure Test for Coupon Specimens

Abstracted Scope (200 words):

Practice A provides a laboratory simulation of various operating conditions of solar heating and cooling systems. It utilizes coupon test specimens and does not provide for heating of the fluid by the containment material.

By proper selection of test conditions, a wide range of operating conditions may be evaluated. These conditions include: operating full flow; stagnant empty, vented; stagnant, closed to atmosphere, non-draindown; and stagnant, closed to atmosphere, draindown.

The practice does not restrict the selection of either the containment material or the fluid for testing. Selection of materials and test conditions should be representative of actual use.

Practice A is one of three practices described in ASTM E 745 for evaluating the corrosion performance of metallic containment materials under conditions simulating field service.

Referred Document:

ASTM E 745   Standard Practices for Simulated Service Testing for Corrosion of Metallic Containment Materials for Use with Heat Transfer Fluids in Solar Heating and Cooling Systems

Principle of Measurement:

Mass loss, appearance changes, and localized corrosion resulting from the testing of metal/fluid pairs under conditions representative of those occurring in actual service are determined.

Apparatus:

For those portions of the practice performed at atmospheric pressure, the test cell is typically a 4-L container of heat-resistant glass. Provision is made for closing the top of the test cell with a nonmetallic cover with openings provided for temperature-measuring devices, tubes for simulating draindown, and tubes for gas sparging. With the possible exception of a mechanically imposed crevice, materials selected for use in the apparatus should not interact with either the test cell or the heat-transfer fluid.

Specimens shall be secured to nonmetallic holders utilizing a nonmetallic fastener or a fastener of the same material as the specimen. The specimen holders are in turn secured to a mixing shaft capable of rotating such that the outer edge of the specimen encounters a fluid to metal speed equivalent to the fluid flow rates encountered in service.

The test cell may be heated by mantle, hot plate, or bath. The more localized heating, typical of a hot plate, may produce changes in the heat transfer fluid.

Control of temperature at the upper and lower limits should be within $3^{o}C$. A refrigeration unit may be used for cooling below ambient temperature.

Gas sparging may be used to produce conditions of aeration and deaeration.

The mixing shaft and attached specimens may be moved to an autoclave for test conditions which cannot be achieved in an open atmosphere.

Sampling:

Test specimen materials shall be representative of those used in actual solar heating and cooling systems. For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, such as a mill finish, closely resembling the one that would be used in service, will yield the most significant results. Standard surface finishes may be preferred for more detailed studies of the metal or the environment.

Conditioning:

Surface cleaning with solvent or water-based cleaners, or abrasives and removal of edge damage, prior to corrosion testing. Scrubbing and electrolytic or chemical cleaning after corrosion testing.

Procedures:

Cut test specimens from sheet having a gage similar to the wall of a solar absorber channel. Typical dimensions are 41 mm long by 25 mm wide.

Clean specimens and weigh to within 0.1 mg immediately prior to testing.

Assemble the apparatus and fill the test cell with sufficient heat transfer fluid to cover all specimens (for example, 15 cm^3/cm^2). Add solvent or full strength fluid as required to maintain the fluid concentration and fluid volume-to-specimen surface area ratio.

It is recommended that the test schedule selected reflect the range of conditions encountered in field service. For example:
1. Test cells are at 10°C. Pump fluids from reservoirs to test cells and heat cells to 93°C in 1 h.
2. Maintain cells at 93°C for 6 h.
3. Allow test cells to cool for 1 h.
4. Pump fluid from test cell to reservoir. Cool test cells to 10°C in 1-1/2 h and maintain until cycle is repeated the following day.
5. Once every two weeks, replace the daily cycle with an autoclave cycle consisting of heating to 204°C, holding for 4 h, and then cooling.

Specimens may be withdrawn at interim periods, e.g. 1 and 3 months, and at the conclusion of the test, typically 6 months.

Determine mass loss per unit area, examine at 10x magnification for localized attack, and document specimen surface and fluid changes.

Calculation Method (if necessary, please include correction method):

$$Rmdd = 100,000 \ [(Wo - Wt)/AT] \quad \text{or} \quad Rmpy = 393.7 \ [(Wo - Wt)/ATD]$$

Rmdd = corrosion rate, mg/dm².day	Rmpy = corrosion rate, mils/year
Wo = original mass, g	Wo = original mass, g
Wt = final mass, g	Wt = final mass, g
A = area, cm², and	A = area, cm²
T = duration, days.	T = duration, years, and
	D = density, g/cm³.

Interpretation/Precision & Accuracy:

Available data are not adequate for the development of precision and accuracy statements.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Laboratory Screening of Metallic Containment Materials for Use with
Liquids in Solar Heating and Cooling Systems

Practice F: Test Loop at Atmospheric Pressure

Abstracted Scope (200 words):

Practice F is intended to simulate deterioration of metal containment
materials when heat is transferred through the containment material tubing
into heat transfer fluid flowing inside the tubing.

The practice is applicable to tubular test specimens similar to those that
would be used in the absorbers of solar collectors

Practice F is one of six practices described in ASTM E 712 for evaluating
the corrosion performance of metallic containment materials under
conditions similar to those that may occur in solar heating and cooling
systems. These conditions include (1) operating full flow, (2) stagnant
full, (3) stagnant partial full, and (4) stagnant empty. The test results
relate to the performance of the material only as part of a metal/fluid
pair.

Referred Document:

ASTM E 712   Standard Practice for Laboratory Screening of Metallic
             Containment Materials for Use with Liquids in Solar Heating
             and Cooling Systems

Principle of Measurement:

Mass loss, appearance changes, and localized corrosion resulting from the flow of heat transfer fluid through a heated tubular test specimen is determined.

Apparatus:
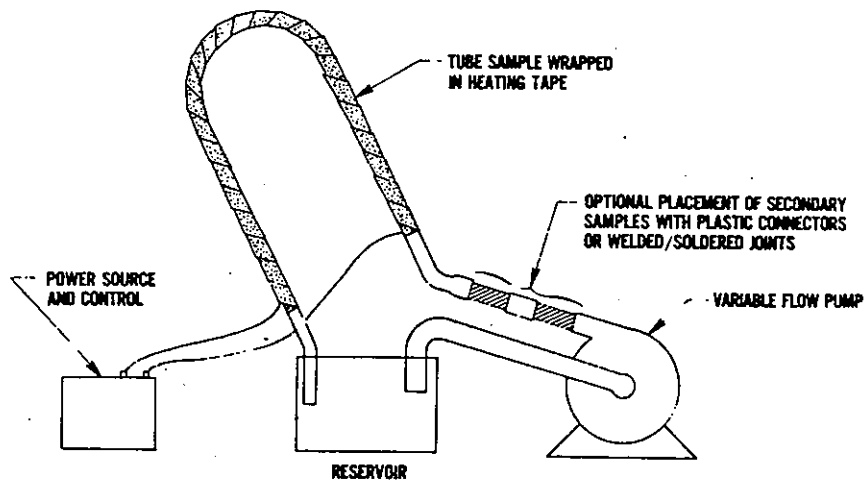
FIG. 2   Schematic of Apparatus for Tube Loop Test

Sampling:

Test specimen materials shall be representative of those used in actual solar heating and cooling systems. The primary sample tubing consists of a 914-mm length of tubing bent around a 203-mm diameter mandrel so that each leg of the U-bend is approximately equal in length. Secondary samples of similar or dissimilar metal tubing may be introduced in the loop as indicated in the figure above.

Conditioning:

Surface cleaning with solvent or water-based cleaners, or abrasives and removal of edge damage, prior to corrosion testing. Scrubbing and electrolytic or chemical cleaning after corrosion testing.

Procedures:

Clean the U-bend specimen and any secondary specimens and install as shown in the figure.

Wrap the U-bend with a heating tape or mount an alternative heating device.

Fill the reservoir and activate the cycling mechanism for control of temperature and fluid flow.

Any cycle of fluid flow and heating may be used for this test. It is recommended that a test period of no less than 30 days be used and that studies be conducted to determine any time dependence of corrosion attack.

Following exposure, dry and weigh the specimens after cleaning and rinsing.

Determine mass loss per unit area, examine at 10x magnification for localized attack, and document changes in the specimen surface and the heat transfer fluid.

Calculation Method (if necessary, please include correction method):

$$Rmdd = 100,000 \ [(Wo - Wt)/AT] \quad \text{or} \quad Rmpy = 393.7 \ [(Wo - Wt)/ATD]$$

Rmdd = corrosion rate, $mg/dm^2 \cdot day$	Rmpy = corrosion rate, mils/year
Wo = original mass, g	Wo = original mass, g
Wt = final mass, g	Wt = final mass, g
A = area, cm^2, and	A = area, cm^2
T = duration, days.	T = duration, years, and
	D = density, g/cm^3.

Interpretation/Precision & Accuracy:

Available data are not adequate for the development of precision and accuracy statements.

Designation:

REPORTING FORMAT

HWA/HWNA-D-ST

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Laboratory Screening of Metallic Containment Materials for Use with
Liquids in Solar Heating and Cooling Systems

Practice E: Crevice Test at Atmospheric Pressure

Abstracted Scope (200 words):

Practice E is intended for use in evaluating the general corrosion
resistance and susceptibility to crevice corrosion attack of metallic
containment materials. This test procedure is generally similar to ASTM
Method G 48, a test for stainless steels.

Practice E is one of six practices described in ASTM E 712 for evaluating
the corrosion performance of metallic containment materials under
conditions similar to those that may occur in solar heating and cooling
systems. These conditions include (1) operating full flow, (2) stagnant
full, (3) stagnant partial full, and (4) stagnant empty. The test results
relate to the performance of the material only as part of a metal/fluid
pair.

Referred Document:

ASTM E 712   Standard Practice for Laboratory Screening of Metallic
             Containment Materials for Use with Liquids in Solar Heating
             and Cooling Systems

Principle of Measurement:

Mass loss, appearance changes, and localized corrosion resulting from the exposure of test specimens immersed in a heated test fluid are determined. A simulated crevice is created by the use of a rubber band or a polymeric washer.

Apparatus:

The apparatus for this test is described in ASTM Method G 48, summarized elsewhere in this report.

Because the rubber band typically employed in ASTM Method G 48 loses elasticity at temperatures above 50°C, an alternative specimen design may be employed at higher temperatures.

In this design, washers of an inert material such as acetal copolymer are secured to the faces of the coupon. Grooves may be cut in the face of the washer that bears on the metal coupon specimen so that each washer forms several separate crevice contacts.

The ratio of the exposed area to the crevice area is significant, a ratio of 15 to 1 being typical.

The plastic nut and bolt, holding the washers in contact with the metal test specimen, are torqued to 5 lbf.in (0.6 N.m).

Sampling:

Test specimen materials shall be representative of those used in actual solar heating and cooling systems. For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, such as a mill finish, closely resembling the one that would be used in service, will yield the most significant results. Standard surface finishes may be preferred for more detailed studies of the metal or the environment.

Conditioning:

Surface cleaning with solvent or water-based cleaners, or abrasives and removal of edge damage, prior to corrosion testing. Scrubbing and electrolytic or chemical cleaning after corrosion testing.

Procedures:

Clean the specimens and weigh to an accuracy of 0.1 mg immediately prior to testing.

Apply the crevice either by rubber band as described in Method G 48, or by application of washers.

Place the specimen in the test flask, add the fluid, and install the condenser.

Heat the flask to the desired test temperature using a mantle, hot plate, or bath. Localized heating typical of a hot plate may produce changes in the test fluid.

Any length of test time may be selected. It is recommended that a test period of no less than 30 days be used and that studies be conducted to determine the time dependence of corrosion.

Following exposure, dry and weigh the samples after cleaning and thorough rinsing.

Determine mass loss per unit area, examine at 10x magnification for localized attack, and document changes in the specimen surface and the heat transfer fluid.

Calculation Method (if necessary, please include correction method):

$$Rmdd = 100,000 \ [(Wo - Wt)/AT] \qquad \text{or} \qquad Rmpy = 393.7 \ [(Wo - Wt)/ATD]$$

$Rmdd$ = corrosion rate, $mg/dm^2 \cdot day$
Wo = original mass, g
Wt = final mass, g
A = area, cm^2, and
T = duration, days.

$Rmpy$ = corrosion rate, mils/year
Wo = original mass, g
Wt = final mass, g
A = area, cm^2
T = duration, years, and
D = density, g/cm^3.

Interpretation/Precision & Accuracy:

Available data are not adequate for the development of precision and accuracy statements.

REPORTING FORMAT

Designation:

HWA-D-LA

Date: 01.03.1986   **Reporter's Name:** Karl. H. Tostmann

Country: Fed. Rep. Germany

Testing Title:

  Aging of Aqueus Fluids and Internal Corrosion of Solar Systems.

  Polytechnique Aalen, Department of Surface Technology and Material Science

---

Abstracted Scope (200 words):

  Heat transfer media for open or closed solar heating systems include water
  or water with addition of antifreezes. The principal problems associated
  with the aqueus fluids are corrosion of construction metals and decomposi-
  tion of orqanic compounds: antifreezes (i.e. ethylene glycol), added corro-
  sion inhibitors ect. Some decomposition products can even promote corrosion.

  Maintenance miaht be restricted to check of pH, inhibitor strenght of the
  solution and reserve alkalinity. Inhibitor effectiveness may be changed in
  service, a possibility of control is an electrochemical check of the fluid
  after a period of time to detect undisirable chanqes of the properties of
  the heat transfer media.

  To predict longtime stability of the heat transfer fluids and corrosion-free
  metallic containement materials and to relate the results to possible check-
  intervals, modified electrochemical methods are set up.

Referred Document:

Principle of Measurement:

Tafel extrapolation on heated rotating cylinder probes. This procedure include electrical polarisation (bimetallic- or redox-influence) fluid-velocity and heat-transfer condition.

Apparatus:

Calibration:

Rotating-cylinder-electrode with internal heating system in an electro-chemical cell, computerized potentiostat-galvanostat. Separate pilot circuit with heat-transfer and flow condition and electrochemical potential-control.

Sampling:

Rotating Zylinder
Flat plate disks

Conditioning:

Flow control, heat control, potential control in situ.
Temperature range 30 °C to 130 °C

Procedures:

    (1) Surface finish of samples (electropolishing)
    (2) Weight loss determination or pitting depth
    (3) Documentation of surface state
    (4) Chemical and electrochemical control of fluid after each over-turn

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

REPORTS ON HEAT STORAGE MATERIALS

2-a LATENT HEAT STORAGE MATERIALS, THERMAL PROPERTIES

(i) PHASE CHANGE TEMPERATURE

REPORTING FORMAT

Date : Feb. 19, 1986   **Reporter's Name :** Kenji Matsuki

Country : Japan

Testing Title :

Testing Method for Melting Point of Storage Materials

Abstracted Scope (200 words) :

1. In this method melting point is defined as the temperature or the range of temperature where the solid sample fully melts.

2. The sample in a capillary glass tube is heated in a bath and the melting point is determined from the temperature at which the solid sample disappears.

Referred Document :

JIS K 0064–1966
"Testing Methods for Melting Point of Chemical Products"

Principle of Measurement:

The melting point is determined from the temperature at which the solid sample disappears.

Apparatus:
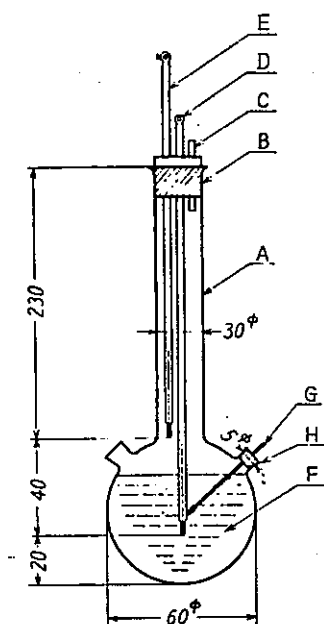
Unit mm

A: Measuring Bottle

B: Cork Cap

C: Air Vent Pipe

D: Thermometer

E: Supplementary Thermometer

F: Bath

G: Capillary Glass Tube
   (Length 70 - 100 mm,
    Inner Radius 0.8 - 1.2 mm,
    Wall Thickness 0.2 - 0.3 mm)

H: Branch Pipe

Fluid in bath: Sodium sulfate or liquid parafin
               (For melting point being lower than 250 deg C)
               Mixture of sodium sulfate and potasium sulfate (3:2)

Calibration:
               (For melting point being higher than 250 degree C)

Sampling:

Powder the sample and dry it in a desiccater of sodium sulfate for 24 hours. Pack the powder tightly with the thickness of 2 to 3 mm in a dry capillary tube by dropping the capillary tube, which is loosely placed in a supporting tube, to a glass dish.

Conditioning:

Procedures:

1. Heat up the measuring bottle A to approximately 30 degree C below the expected melting point. Insert the capillary tube G through the branch pipe H to set the sample close by the tip of the thermometer.

2. Continue heating at the rate of approximately 3 degree C per minute to the temperature approximately 5 degree C below the expected melting point. Then reduce the heating rate to 1 degree C per minute.

3. The beginning of melting is defined as the time when the sample begins to change, and the end is defined as the time when no solid sample is recognized. Record the readings of the thermometer D and the supplementary thermometer E at the beginning and in the end of melting.

Calculation Method (if necessary, please include correction method):

Melting point T (degree C) is calculated from

$$T = t + 0.00016 \; (t - t') \; n$$

where t is the reading of the thermometer D (degree C), t' is the reading of the supplementary thermometer E (degree C), and n is the number of scales of the thermometer outside the sample.

Interpretation/Precision & Accuracy:

REPORTING FORMAT

Date :   Feb. 19, 1986   Reporter's Name : Kenji Matsuki

Country :   Japan

Testing Title :

Testing Method for Freezing Point of Storage Materials

Abstracted Scope (200 words) :

1. In this method freezing point is measured by cooling the material in the bath of water, ice, or mixture of water and sodium chloride.

2. Recommended temperature range is from room temperature to approximately –10 degree C.

Referred Document :

JIS K 0065–1966
"Testing Method for Freezing Point of Chemical Products"

Principle of Measurement:

By measuring the temperature change of the material, freezing point is determined from the temperature where the falling rate of temperature changes (Fig. 1).

Apparatus:

unit mm
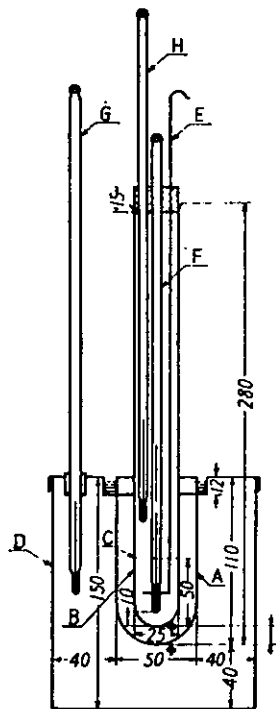
A: Air Jacket

B: Sample Container

C: Standard Level Line

D: Bath

E: Stick for Mixing

F: Thermometer

G: Thermometer

H: Supplementary Thermometer

Calibration:

Sampling:

Sample cup must be filled with the sample up to a standard level of the cup. Solid sample may be heated up to fill the cup at least 20 degree C below the expected freezing point.

Conditioning:

Procedures:

1. Fill the bath with water(1) whose temperature is 5 degree C lower than the expected freezing point.

2. Set the container in the air jacket. When the sample temperature is cooled to 5 degree C higher than the expected freezing point, move the mixing stick up and down gently 20 times per minute and read the thermometer every 30 seconds.

3. When the sample begins to freeze, stop mixing and read the thermometer F and the supplementary thermometer H.

4. This measurement need to be carried at least 4 times continually, and the freezing point is determined from the average value where the difference of measured temperature must be within 0.2 degree C.

   (1) At room temperature to 0 dgree C use water or ice. At 0 to −10 degree C, use mixture of ice and sodium chloride (3:1).

Calculation Method (if necessary, please include correction method):

Freezing point T (degree C) is calculated from

$$T = t + 0.00016 \ (t - t') \ n$$

where t is the reading of the thermometer F (degree C), t' is the reading of the thermometer H (degree C), and n is the number of scales of the thermometer outside the sample.

Interpretation/Precision & Accuracy:

Precision of repeatability: less than 0.2 degree C.
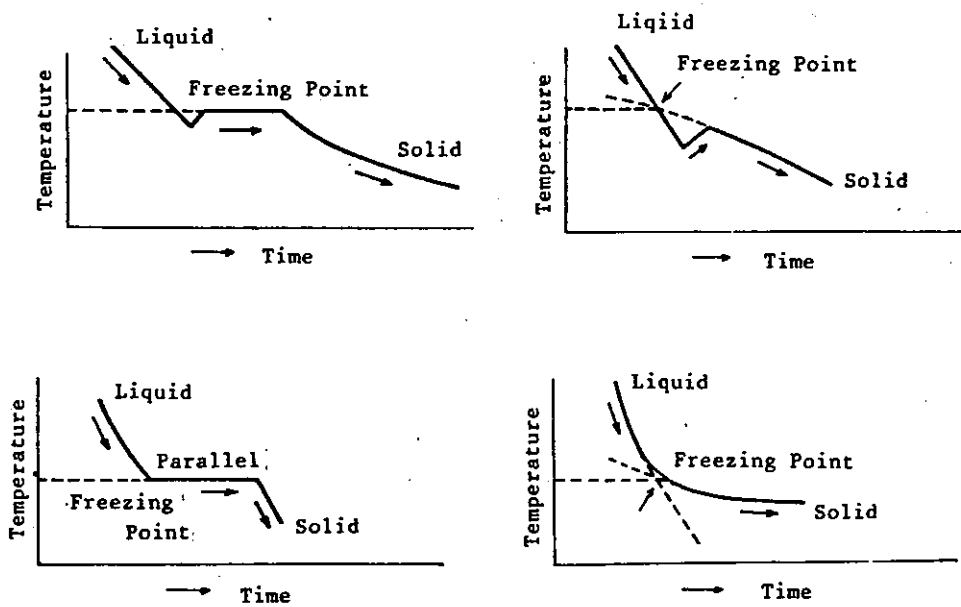
Fig. 1   Examples of temperature variations

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Melting Point of Waxes

Abstracted Scope (200 words):

This method covers the determination of the initial and final melting
points and recovery point of waxes using a hot stage and microscope. Wax
shavings or granules are observed through the microscope, and the
temperature at which first movement of wax is noted and recorded as the
initial melting point. The temperature at the time that the wax is observed
to be completely melted is recorded as the final melting point. The
temperature at which the first cystal appears in the melted wax, when the
hot stage is slowly cooled, is recorded as the recovery point.

Referred Document:

ASTM F 766 Standard Test Method for Melting Point of Waxes

Principle of Measurement:

Wax shavings or granules are heated on a hot stage installed on a microscope. The temperatures of initial movement and of complete melting are recorded as the initial and final melting points, respectively. The temperature at which the first crystal appears on cooling is recorded as the recovery point.

Apparatus:

A controlled micro hot stage and microscope; 40 to 50x power, or a suitable alternative.

Microscope slides; 25 by 37 mm.

No. 2 cover slides; 18 mm square.

A test reagent set, containing substances with known melting points for checking the accuracy of the hot-stage thermometer over the temperature range used.

Sampling:

The test sample should be selected to be representative of the whole sample

Prepare sufficient wax to give 10 $\pm$ 2 mg of sample for each melting point determination. Brittle wax is prepared by grinding the sample in a mortar and pestle and passing the wax through a 200 mesh screen. Non-brittle wax is prepared by shaving off a few small particles with a sharp instrument.

Conditioning:

Procedures:

1. Transfer the 10 mg test specimen onto a microscope slide, install a cover glass and place the sample assembly on the hot stage. Adjust the microscope light source and focus the microscope using 40 to 50x power to bring the wax sample into sharp focus.

2. Turn on the hot-stage heat control and set the control so that the stage will heat to within $10^{\circ}C$ of the expected melting point of the sample at a rate of approximately $3^{\circ}C/min$. When this temperature is reached, reset the heat control so that the temperature of the stage increases approximately $2^{\circ}C/min$.

3. Observe the wax through the microscope and record the temperature of the initial (first movement of wax) and final (wax completely melted) melting points.

4. Rotate the ocular for maximum definition and continue observing the melted wax. Reduce the heat control setting and record the temperature at which the first crystal appears as the recovery point.

5. Correct the observed temperatures by calibrating the hot-stage thermometer, over the temperature range used, using test reagents having known melting points.

Calculation Method (if necessary, please include correction method):

Correct the observed temperatures using the thermometer calibration values and report to the nearest $^{\circ}C$.

Interpretation/Precision & Accuracy:

Repeatability: Duplicate determinations (one operator and apparatus) on uniform samples should not differ by more than $2^{\circ}C$.

Reproducibility: Melting point differences between separate testing facilities using different manufacturers' test instruments would be expected to be greater than those obtained with one apparatus and operator because of instrument and operator variability. Values are not given.

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Melting and Crystallization Temperatures by Thermal Analysis

Abstracted Scope (200 words):

This method covers the determination of transition temperatures such as the melting and crystallization temperatures of materials by differential scanning calorimetry (DSC) and differential thermal analysis (DTA). The normal operating temperature range is from -120 to 600°C. With special equipment, the temperature range can be extended.

Computer or electronic based instruments, techniques, or data treatment equivalent to this test method may be used.

Referred Document:

ASTM E 794   Standard Test Method for Melting and Crystallization
             Temperatures by Thermal Analysis

ASTM D 3418 Standard Test Method for Transition Temperatures of Polymers
             by Thermal Analysis

Principle of Measurement:

The method consists of heating or cooling the test material at a controlled rate in a controlled atmosphere through the region of a transition, such as fusion or crystallization. The difference in heat flow or temperature between the test material and a reference material due to energy changes in the material is continuously monitored and recorded. Absorption or release of energy results in endothermic or exothermic peaks.

Apparatus:

A differential scanning calorimeter, capable of heating or cooling at rates up to at least $10^\circ C$/min and of automatically recording the differential energy input between the sample and reference material both to the required sensitivity and precision. Typically, the differential sensitivity should be sufficient to provide specimen temperature readability to within $1^\circ C$.

A differential thermal analyzer, capable of heating or cooling at rates up to at least $10^\circ C$/min and of automatically recording the differential temperature between sample and reference material both to the required precision; typically within $1^\circ C$.

Specimen pans, for DSC, composed of aluminum or other metal of high thermal conductivity. For DTA, sample cups or tubes composed of borosilicate glass, alumina, or quartz may be used. The specimen pans or tubes must not react with the sample.

Nitrogen, or other inert purge gas.

An analytical balance, with a capacity greater than 15 mg, capable of weighing to the nearest 0.01 mg.

Sampling:

Powdered or liquid samples should be thoroughly mixed and sampled from by removing portions from different parts of the container which are then combined and mixed. Film, sheet, molded or pelleted samples can be cut, punched, or bored to obtain pieces that fit the sample containers.

Conditioning:

Materials that have a residual thermal history may require a preliminary thermal cycle to above the melting point to avoid interference with the measurement of the transitions of interest. Care should be taken to avoid thermal decomposition of the test specimen.

Procedures:

1. Use a sample weight appropriate for the material to be tested; about 5 mg. for first order transitions and 10 to 20 mg for glass transitions. An appropriate sample weight results in 25 to 95% scale deflection of the recording system when acceptable sensitivity is used.

2. Ensure intimate thermal contact between the sample, the sample pan or tube and thermocouple, where used. Good thermal contact is also required between the sample container and the test apparatus.

3. Load the specimen into the instrument chamber, purge the chamber with nitrogen or other inert gas at a flow rate of 10 to 50 cm^3/min during the experiment.

4. Heat the specimen rapidly to 50°C below the transition temperature of interest and equilibrate.

5. Heat the specimen at 10°C/min through the transition until the baseline is reestablished above the transition. Hold at this temperature for about two min and then cool the specimen, at the same rate, down through the transition until the baseline is reestablished below the transition.

6. Reweigh the specimen after completion of scanning and discard. Report any mass loss observed.

Calculation Method (if necessary, please include correction method):

From the resultant curve, measure the following points:

T_s = temperature of the first deviation from the baseline, °C
T_e = extrapolated onset temperature for fusion, °C
T_p = melting peak temperature, °C
T_f = temperature of return to baseline, °C
T_n = extrapolated temperature of crystallization onset (nucleation), °C
T_c = temperature of crystallization peak, °C

Report similar temperatures for other transitions.

Interpretation/Precision & Accuracy:

Repeatability (Single Analyst): Two averages for T_s (95% confidence level) should be considered suspect if they differ by more than 7.9°C.
Two averages for T_c, T_p, T_f, T_n, and T_e should be considered suspect (95% confidence level) if they differ by more than 3.1°C.

Reproducibility (Multilaboratory): Two averages for the following should be considered suspect (95% confidence level) if they differ by:
40°C for T_s for broadly melting material, 7.1°C for narrowly melting material, 5.9°C for T_e, T_p, T_n and T_c, and 19°C for T_f.

REPORTS ON HEAT STORAGE MATERIALS

2-a-(ii) LATENT HEAT

Date :   Feb. 28, 1986   Reporter's Name : Kenji Matsuki

Country :   Japan

Testing Title :

    Measurement Method of Latent Heat of Storage Materials
        (Differential Scanning Calorimetry)

Abstracted Scope (200 words) :

1. In this method a sample and the standard material are heated in an electric furnace at a constant rate of temperature variation. While heating, the electric energy of the two internal heaters required to equalize the temperature is recorded. When phase change of the sample occurs, a peak value of the energy is observed in the time variation. The latent heat of the sapmle is calculated in the area of the peak.

2. The phase change temperature of the sample is determined from the onset of the peak.

3. Measurable weight of a sample is typically on the order of 10 mg.

4. Since in this method the thermally insulated condition required for a precision calorimeter is not satisfied, an appropriate calibration must be conducted.

Referred Document :

    H. Kanbe: Thermal Analysis, Kodansha, Tokyo, 1975.

Principle of Measurement:

Heating a sample and the standard material separately, the difference of heating energy required to equalize the temperature of the two is measured. From the measured energy, the phase change energy of the sample can be calculated.

Apparatus:

Differential Temperature Thermocouple

Slope Adjusting Circuit

Standard Material

Electric Furnace

Sample

Cold Junction Temperature Compensater

Internal Heater

Temp

DSC Circuit

DSC

Recorder

DTA Circuit
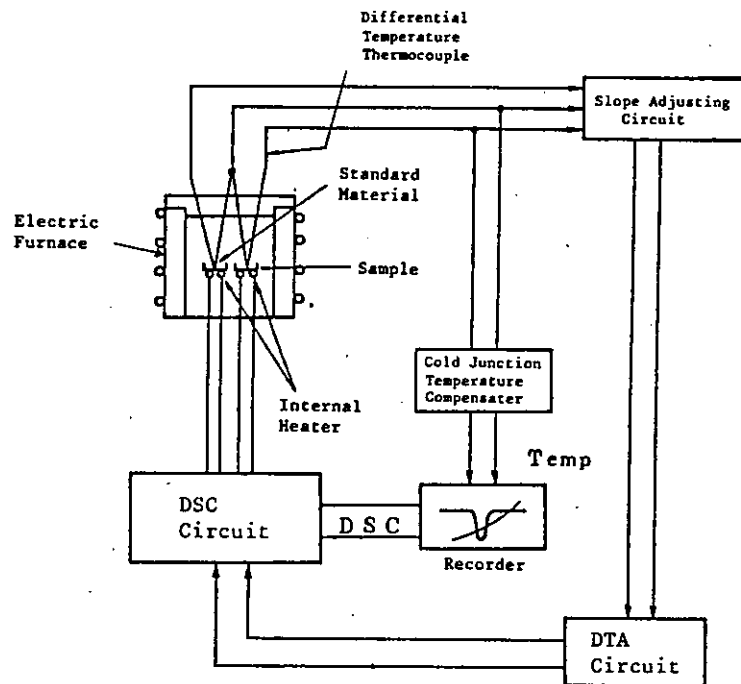
Calibration

For the choise of the standard material, the transition temperature of the standard material must be close to the phase change temperature of the sample (Table 1).

Sampling:

1. Powder the sample to be 100 to 300 mesh in size.

2. Pack the sample loosely into a cell as thin and uniform as possible.

Conditioning:

Procedures:

1. Set the standard material (usually -Alumina) in an electric furnace.

2. Weigh the material for calibration (W_c), pack it into a cell, and set the material in the furnace.

3. Raise the temperature of the furnace at a constant rate (e.g. 10 degree C per minute). Then record the heating energy difference.

4. Measure the area of the peak of the energy difference (A_c).

5. Weigh the sample (W_S), pack it into a cell, and set it in the furnace.

6. Raise the temperature of the furnace at the same rate as the calibration. Then record the heating energy difference.

7. Measure the area of the peak of the energy difference (A_S).

Calculation Method (if necessary, please include correction method):

The latent heat of the sample ΔH is calculated from

$$\Delta H = (\Delta H_c \ W_c \ \frac{A_s}{A_c}) \ / \ W_S$$

where ΔH_c and W_c are the heat of phase change and the weight of the material for calibration, respectively, W_S is the weight of the sample, and A_c and A_S are the peak of the material and the sample, respectively.

Interpretation/Precision & Accuracy:

Precision: typically 5%

Table 1 Examples of Standard Materials for Calorimetry

Temperature (deg. C)	Material	Heat of Fusion (cal/g)	Heat of Transition (cal/g)
47	CBr_4		4.81
48.2	Benzophenon	23.5	
69	Stearic Acid	47.5	
69.8	Biphenyl	28.7	
99.3	Phenanthrene	25.0	
114	o-Dinitrobenzene	32.3	
121.8	Benzoic Acid	33.9	
128	KNO_3		12.86
156.9	In	6.79	
160	$AgNO_3$		3.5
187.8	Pentaerythritol	77.1	
212	$AgNO_3$	17.7	
231.9	Sn	14.4	
252	$LiNO_3$	88.5	
299.8	$KClO_4$		23.7
306.2	$NaNO_3$	44.2	
327.4	Pb	5.50	
337	KNO_3	28.1	
419.5	Zn	24.4	
498	$PbCl_2$	20.9	
553	LiBr	36	
575	$LiSO_4$	62	
588.8	Na_2WO_4		28.57
659	Al	95.8	

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Heats of Fusion and Crystallization by Differential Scanning Calorimetry

Abstracted Scope (200 words):

This test method covers the determination of the heat of fusion and
crystallization by differrential scanning calorimetry. It is applicable
to solid samples in granular form or in any fabricated shape from which an
appropriate specimen can be cut and to liquid samples that crystallize
within the range of the instrument.

The normal operating temperature range is from -120 to 600°C. This
temperature range can be extended, depending on the instrumentation used.
This test method is generally applicable to thermally stable materials
with well defined endothermic or exothermic behavior.

Computer or electronic based instruments, techniques, or data treatment
equivalent to this test method may be used.

Referred Document:

ASTM E 793 Standard Test Method for Heats of Fusion and Crystallization
            by Differential Scanning Calorimetry

ASTM D 3417 Standard Test Method for Heats of Fusion and Crystallization
            of Polymers by Thermal Analysis

Principle of Measurement:

Differential scanning calorimetry (DSC) is a technique in which the difference in energy inputs into a substance and a reference material is measured as a function of temperature, while the substance and reference material are subjected to a controlled temperature program. Differences in the amount of energy required to maintain the test specimen and reference at the same temperature are used to determine heats of transformation.

Apparatus:

A differential scanning calorimeter capable of heating and cooling rates up to at least 10°C/min and of automatically recording the differential heat flow between the sample and reference material to the required sensitivity and precision. The instrument should have sufficient heat flow sensitivity to provide a precision within 1% and a time base precision of within 1%.

A planimeter or other method of area measurement with an accuracy and precision within 1%.

Specimen holders, inert to the sample, composed of aluminum or other material of high thermal conductivity.

Nitrogen, or other inert gas supply, for purging purposes.

A balance, with a capacity greater than 15 mg, capable of weighing to the nearest 0.01 mg.

Sampling:

Powdered or granular specimens should be mixed thoroughly prior to sampling and should be sampled by removing and then blending portions taken from various parts of the container. Liquid samples may be sampled directly after stirring.

Conditioning:

A preliminary test cycle to remove previous thermal history, e.g., residual thermal stresses, or annealing above the glass transition temperature of polymers such as crystallizable polystyrene, may be required before valid test data are taken. Comparison of initial and subsequent test runs can be used to determine whether or not conditioning is required.

Procedures:

1. Into a clean specimen holder, weigh 1 to 15 mg of specimen.

2. Seal the specimen holder with a lid under ambient conditions, minimizing the free space between sample and lid. Hermetic sealing in an inert atmosphere may be desirable for specimens sensitive to oxidation.

3. Load the specimen into the instrument chamber and purge with nitrogen or other inert gas throughout the experiment at 10 to 50 cm^3/min.

4. Select the appropriate heat flow axis and time axis sensitivities to yield an area of 30 to 60 cm^2 under the fusion endotherm, or crystallization exotherm.

5. The specimen may be heated rapidly to 50°C below the melting (or other transition) temperature and allowed to equilibrate.

6. Heat the specimen at 10°C/min through the transition, until a baseline is reestablished above the endotherm or exotherm.

7. Hold the specimen at this temperature for 2 min.

8. Cool the specimen at 10°C/min through the transition until baseline is reestablished below the endotherm or exotherm. Reweigh the specimen after completion of scanning. Discard the data if there is mass loss greater than 1% or evidence of reaction with the sample holder.

Calculation Method (if necessary, please include correction method):

$$H = (ABT/W) (H_SW_S/A_ST_SB_S) \quad , \text{ where}$$

H = heat of transition of the sample, J/g
H_S = heat of transition of the standard, J/g
A = peak area of the sample, cm^2
A_S = peak area of the standard, cm^2
B = time scale used for the sample, min/cm
B_S = time scale used for the standard, min/cm
W = mass of the sample, mg, W_S = mass of the standard, mg
T = Y-axis sensitivity used for the sample, mW/cm
T_S = Y-axis sensitivity used for the standard, mW/cm.

Interpretation/Precision & Accuracy:

Repeatability (single analyst): 2.8% coefficient of variation with 88 degrees of freedom. Two such averages should be considered suspect (95% confidence level) if they differ by more than 7.8%.

Reproducibility of pure materials (Multilaboratory): 3.0% coefficient of variation at 58 degrees of freedom for materials melting or crystallizing over a narrow temperature range. Two such results should be considered suspect (95% confidence level) if they differ by more than 8.6%. Results for broad melting range polymers should be considered suspect (95% confidence level) if they differ by more than 23%.

REPORTS ON HEAT STORAGE MATERIALS

2-a-(iii) SPECIFIC HEAT

Date: Feb. 28, 1986   **Reporter's Name:** Kenji Matsuki

Country: Japan

Testing Title:

    Measurement Method of Specific Heat of Storage Materials
        (Adiabatic Scanning Calorimetry)

Abstracted Scope (200 words):

1. In this method a sample cell is thermally insulated from the environment. In order to achieve this condition a vessel contained the cell is heated to be the same temperature as that of the sample. Thus the thermally insulated condition required for a precise calorimeter is attained. The specific heat of the sample is calculated from the change of measured temperature and the heating energy required.

2. Since sensitivity of this method is not depend on the heating rate of the sample, data in quasi-static state can be obtained in the case of a low heating rate such as less than 1 degree C per minute.

3. In the case of materials which exhibit the transition, the heat of the transition can be calculated from the integration of the specific heat.

Referred Document:

    R. Kato et. al: Proceedings ICTA 5th Thermal Analysis (1977), p64.

Principle of Measurement:

When a sample is thermally insulated from the environment, the specific heat of the sample is determined from the ratio of the heating energy and the temperature raise multiplied by the weight of the sample.

Apparatus:

Calorimeter and Control System
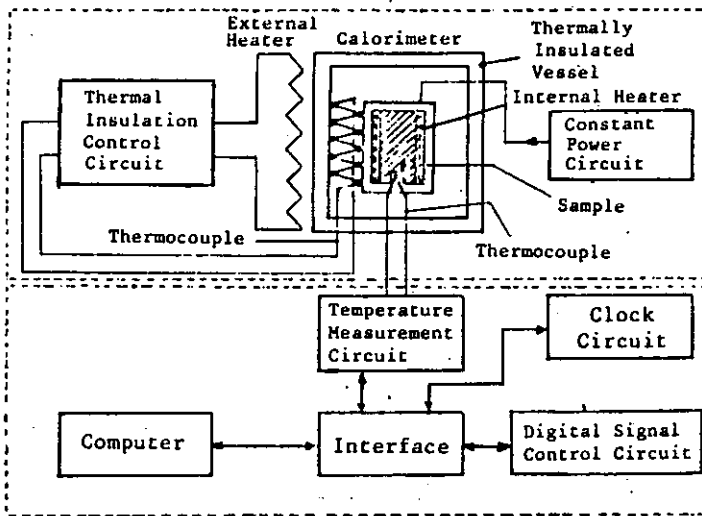
Calorimeter

Measurement and Data Processing System
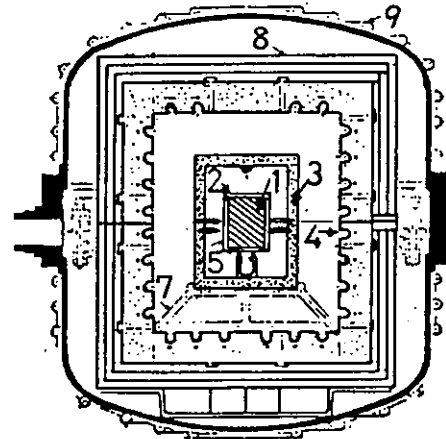
1. Sample
2. Sample Holder
3. Thermally Insulated Vessel
4. External Heater
5. Internal Heater
6. Thermocouple for Thermal Insulation Control
7. Spacer
8. Heat Shield
9. Belljar

Calibration:

Sampling:

Conditioning:

Procedures:

1. Place a sample cell without the sample in the calorimeter. Heat the cell by the internal heater with a constant power input and measure the temperature raise of the cell.

2. Pack the sample into the cell and place it in the calorimeter. Then heat the sample with the same power input and measure the temperature raise of the cell.

Calculation Method (if necessary, please include correction method):

The specific heat of the sample C_P is calculated from

$$C_P = (W \Delta t) / (M \Delta\theta) - M'C_P' / M$$

where W is the constant power input to the internal heater, M is the weight of the sample, $M'C_P'$ is the heat capacity of the cell and the sample holder, $\Delta\theta$ is the temperature raise of the sample, and Δt is the time required for the temperature raise.

Interpretation/Precision & Accuracy:

Precision: less than 3%

Designation:

Date: 7 april 1986   Reporter's Name: F. van der Graaf

Contry: the Netherlands

Testing Title:. The quantitative test of thermal qualities as specific
              heat, melting heat, hydratation heat,of phase change
              materials (PCM). ·

Abstracted Scope (200 words):

In order to compare various PCM's the measurement of their relevant thermal
characteristics is required.

The applied instrument for these tests is covered by the term Quantitative
differential thermal analysis (quantitative DTA). In this instrument, the
material is subjected to a controlled temperature programme (DTA-run)
while the energy input is measured directly with a heat flow sensor.
·The measuring results are presented as curve where a specific-heat-
equivalent is plotted against the temperature.

There are commercial available DTA-instruments for sample .sizes of
milligrams, temperature ranges of o to 1000°C and heating/cooling ranges
up to 30°C/min.

For PCM-tests these instrument-parameters-values are in general not
optimized. The instrument used for the test is designed for PCM's and has
next qualities:

- sample size: 1 to 5 grammes (this is representative sufficiently fore
  the -oftenly very unhomogeneous- PCM's)
- temperature range circa .0-100°C
- heating/cooling circa 0,02 to 1°C/min.

Referred documents:

1- Manual "Calorimeter for quantitative differential thermal Analysis"
   TPD 23-1-1978 F. v.d. Graaf.
2- CEC Storage testing programme part II, .edited .by E. v. Galen, nov. '84.
   page 173 to 189 "Problems in the interpretation of measurements on
   heat storage materials with thermal analysing instruments" by
   F. v.d. Graaf.
3- "For better thermal Analysis" by G. Lombardi .ICTA 1980, distributed
   by TAWN.
4- "Analysis of thermally stimulated processes" .R. Chen, Y. Kirch.

Principle of the instrument:

A heat flow sensor is mounted on a metal block. This metal block can be warmed up or cooled down with a constant heating-/cooling rate.
On the heat flow sensor a platform is mounted on which a sample holder can be placed.
The heat, necessary for the filled or unfilled sample-holder to follow the temperature of the block is measured directly by the heat flow sensors.
The metal block is in principle provided with two heat flow sensors with platforms.
On one of them an empty sample holder is placed, on the other a sample holder filled with sample-material. The difference between the thus measured heat flows corresponds with the net heat flow to the sample.
The applied instrument is extended with 3 "sample units" and one "compensating" unit.

Apparatus:
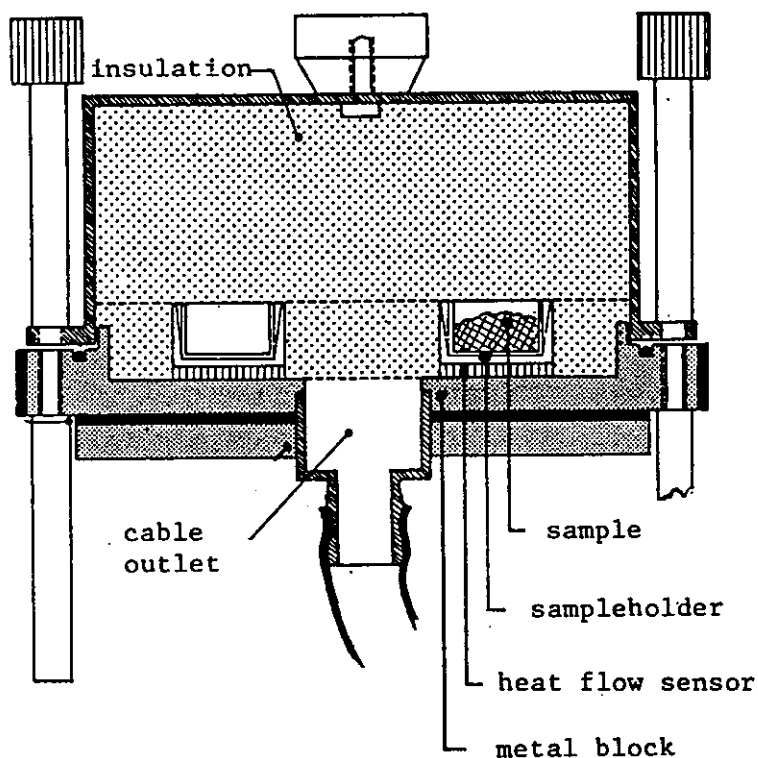
cross-section of the DTA-calorimeter
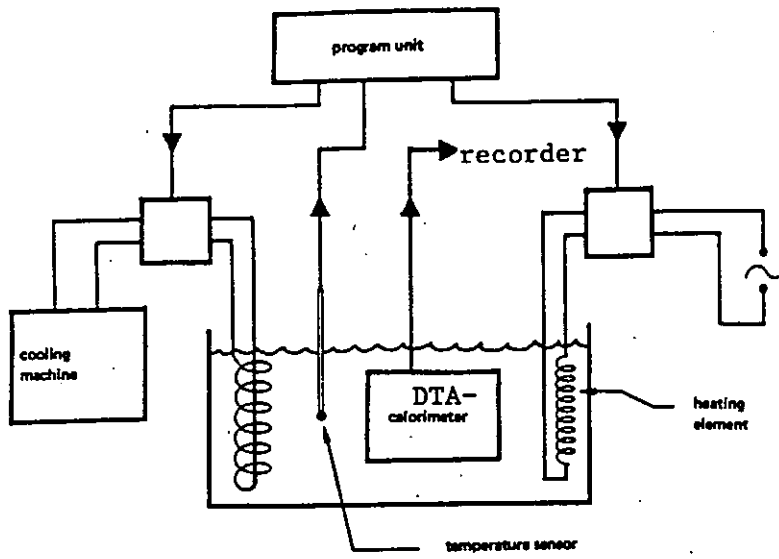
block-diagram of the temperature-controlsystem
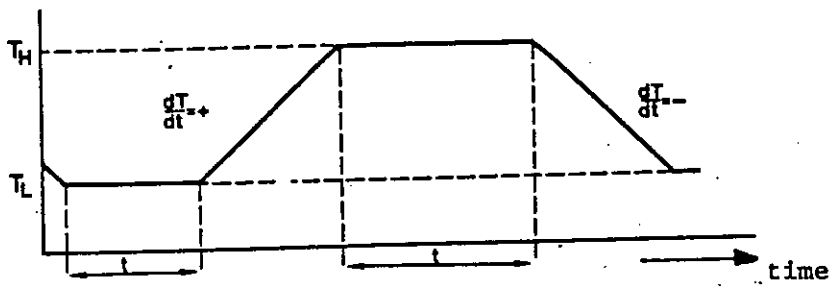
DTA - temperature-programme

Calibration

method 1: A piece of pure copper with well-known weight and specific heat is subjected to a "DTA"-run. From this the relation between heat flow rate and signal can be derived.

method 2: with an electrical heater, well-fitted on the heat flow meter a well-known electrical power is dissipated while the temperature is constant. This also leads to a calibration value of the heat flow sensors.

remark: The applied instrument is provided with 4 heat flow sensors. With 4 equal copper-pieces or heating elements it can be calibrated. During the calibration the 4 heat flow sensors are controlled to the same calibration value by shunting the heat flow sensors electrically.

Sampling:

Separate removable sample holders allow to fill them with sample material with an accurate weight-measurement. To avoid losses in weight by evaporation, the sample holders can be closed by a rubber cover.

Condition:

Procedure:

- weight the 3 samples and eventually close with covers
- place the calorimeter in the bath and control the constant temperature (low level)
- check all heat flow signals (they have to be zero)
- start programme with a constant heating rate
- registrate 3 differential signals and the temperature with a recorder
- determine the "Full Ranges" to be sure all effects are within range
- go back to start-temperature
- repeat this part of the programme.
- do the same with a constant cooling rate
- for finding the reproducability the programme can be repeated several times.

Calculation Method:

- From the recorder curve, the known calibration-value of the heat flow
  sensors and the weights of the samples the specific heat can be
  calculated [kJ / gr. K.].
- The heat effects like melting, solidification, hydratating, etc. can
  be quantified by determining the energy, corresponding with the
  area size between the heat effect-curve and the interpolated specific
  heat-curve between beginning and end of the effect (the base-line).
  [kJ / gr] (see ref. 4)
- Finally the calculated values have to be plotted as function of the
  temperature.
- For a large number of samples a datalogging / computer-system is
  applied for economical reasons.
  All calculations are done automatically, inclusive the plotting.

Interpretation / Precision & Accuracy:

- The interpretation of the registrated curve becomes complicated if
  the testing materials have heat-effect-trajects and specially if the
  heat-effects are influenced by the heating and/ore cooling rate
  (e.g. undercooling see ref. 2)
  By repeating the measurements with different values for the heating
  and cooling rate, the interpretation becomes more reliable and the
  final result more accurate.
- Another important aspect for the interpretation and the accuracy
  is the determination of the base-line. Several theoretical views
  are existing.
  (see ref. 4)

REPORTS ON HEAT STORAGE MA TERIALS

2-a-(iv) THERMAL CONDUCTIVITY

REPORTING FORMAT

Date :   Feb. 28, 1986   Reporter's Name : Kenji Matsuki

Country :   Japan

Testing Title :

Measurement Method of Thermal Conductivity of Solid Storage Materials

---

Abstracted Scope (200 words) :

1. In this method a sample is heated stepwise by light emitted from a xenon
   arc lamp. The thermal diffusivity of the sample is determined from the
   temperature variation of the sample.

2. Since the measurement is carried out in vacuum, the heat loss can be
   neglected under temperature 400 degree C. Therefore the high precision
   is expected.

3. The time of measurement is usually a few seconds.

Referred Document :

    Y. Katto et al.: Special Heat Transfer Theory, Yokendo, Tokyo, 1984.

Principle of Measurement :

Since heat loss can be neglected in measurements carried out in vacuum, the temperature raise of the sample heated up stepwise by light can be calculated theoretically considering the thermal diffusivity of the sample. Therefore the thermal diffusivity of the sample can be determined from measured data.

Apparatus :
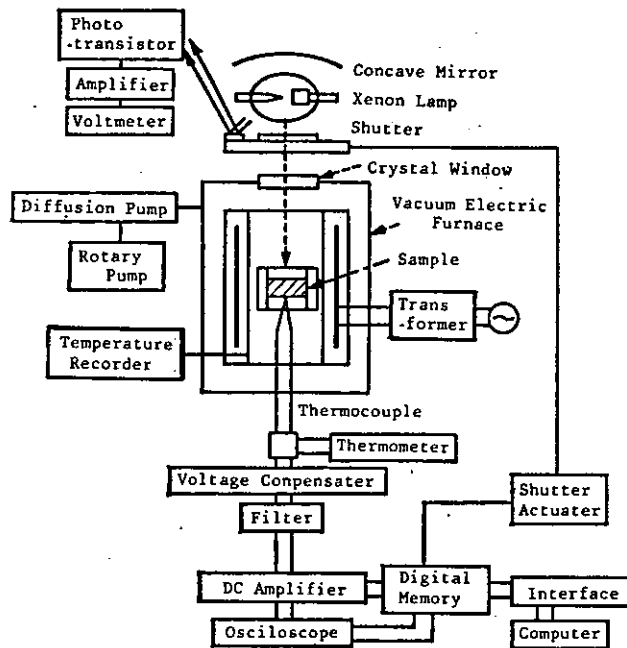
Calibration :

Sampling :

The sample is shaped into a disk whose diameter is 10 to 15 mm.

Conditioning :

Procedures:

1. Heat up the sample to the measurement temperature in a vacuum furnace.

2. When the temperature of the sample becomes constant, open the shutter to illuminate the sample stepwise with the light of a xenon lamp for heating.

3. Record the temperature raise of the back side of the sample.

Calculation Method (if necessary, please include correction method):

The temperature variation of the sample is calculated theoretically with thermal properties of the sample. Therefore the thermal diffusivity a of the sample is determined from the ratio of the temperature raise of the sample after t_1 seconds and $t_2 (=2t_1)$ seconds (Fig. 1). The thermal conductivity λ is calculated from

$$\lambda = a c \rho$$

where c is the mass of the sample and ρ is the density of the sample.

Interpretation/Precision & Accuracy:

$$\Phi = 8\,\mathcal{E}_o\,\sigma\,T_o^3\,1\,/\,\lambda = 8\,\mathcal{E}_o\,\sigma\,T_o^3\,1\,/\,(a\,c\,\varphi)$$

$\mathcal{E}_o$: Emissivity of Sample σ : Stephan Boltzman Constant

T_o : Initial Temperature   1 : Thickness of Sample

λ : Thermal Conductivity   a : Thermal Diffusivity

$c\varphi$: Heat Capacity
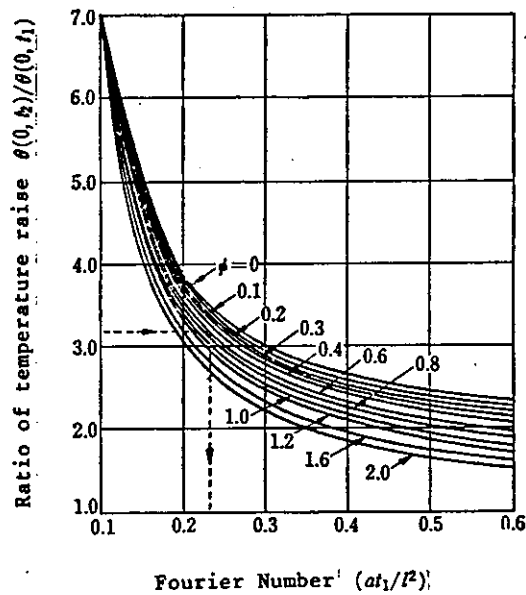
Fig. 1 Relationship between the ratio of temperature
raise and Fourier Number ($t_2=2t_1$)

REPORTING FORMAT

Date : Feb. 28, 1986   Reporter's Name : Kenji Matsuki

Country : Japan

Testing Title :

Measurement Method of Thermal Conductivity of Liquid Storage Materials

---

Abstracted Scope (200 words) :

1. In this method a sample in a metal container is heated stepwise by light emitted from a halogen lamp. The thermal diffusivity of the sample is determined from the temperature variation of the container.

2. Since the measurement is carried out in vacuum, the heat loss can be neglected under temperature 400 degree C. Therefore the high precision is expected.

3. The time of measurement is usually a few seconds.

Referred Document :

   Y. Katto et.al.: Special Heat Transfer Theory, Yokendo, Tokyo, 1984.

Principle of Measurement:

Since heat loss can be neglected in measurements carried out in vacuum, the temperature raise of the sample heated up stepwise by light can be calculated theoretically considering the thermal diffusivity. Therefore the thermal diffusivity of the sample can be determined from measured data.

Apparatus:
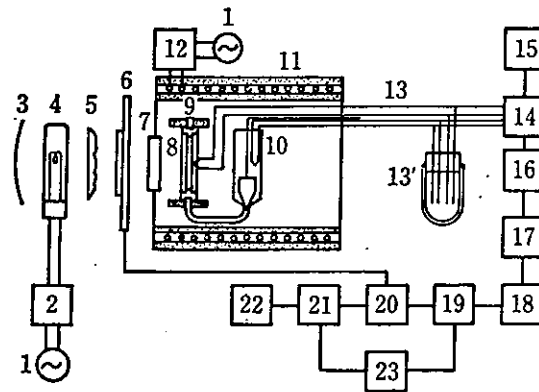
1. AC Power	11. Electric Furnace
2. Power Supply for Lamp	12. Power Supply for Furnace
3. Concave Mirror	13. Thermocouple
4. Halogen Lamp	14. Scanner
5. Fresnel Lens	16. Voltage Compensater
6. Shutter	17. DC Amplifier
7. Crystal Window	20. Digital Memory
8. Sample Container	22. Recorder
9. Sample Holder	
10. Melting Tank	

Calibration:

Sampling:

Conditioning:

Procedures:

1. Pour a liquid sample into a container between two thin matal plates. In the case of a sample which is solid at room temperature, melt the sample in a melting tank and feed the sample into the container by opening a valve at the bottom.

2. Heat up the sample to the measurement temperature in a vacuum furnace.

3. When the temperature of the sample becomes constant, open the shutter to illuminate the sample stepwise with the light of a halogen lamp for heating.

4. Record the temperature raise of the back side of the container.

Calculation Method (if necessary, please include correction method):

The temperature variation of the container is calculated theoretically with thermal properties of the container and the sample. Therefore the thermal diffusivity a of the sample is determined from the ratio of the temperature raise of the sample after t seconds and t ($=2t$) seconds.
The thermal conductivity is calculated from

$$= a\ c$$

where c is the mass of the sample and is the density of the sample.

Interpretation/Precision & Accuracy:

REPORTS ON HEAT STORAGE MATERIALS

2-a-(vi) DENSITY OF SOLID PHASE

REPORTING FORMAT

Date: Feb. 28, 1986   Reporter's Name: Kenji Matsuki

Country: Japan

Testing Title:

Measurement Method of Specific Gravity of Storage Materials in Solid Phase

---

Abstracted Scope (200 words):

1. In this method the specific gravity of the storage material is measured with the use of a specific gravity bottle.

2. The density of the material is equal to the specific gravity multiplied by the density of water at 4 degree C and 1.013250 bar.

Referred Document:

     JIS Z 8807-1976
     "Measuring Methods for Specific Gravity of Solid"

Principle of Measurement:

The weight of the specific gravity bottle contained a sample is M_1. Total weight of the sample and the bottle which is fill with the liquid is M_2. Then $M_2 - M_1 = m$ is the weight of the liquid. If m_2 is assigned to the weight of the bottle filled only with the liquid, $m_2 - m_1 = m_0$ is the weight of the same volume of the liquid. The specific gravity is determined from the ratio m/m_0. where m is the weight of the sample.

Apparatus:

1. Specific Gravity Bottle: Glass bottle with ground glass plug to minimize the loss of the liquid by expansion and evaporation.

Cap

Standard Level Line

Plug

Capillary Tube

Ground Glass Fitting

25ml
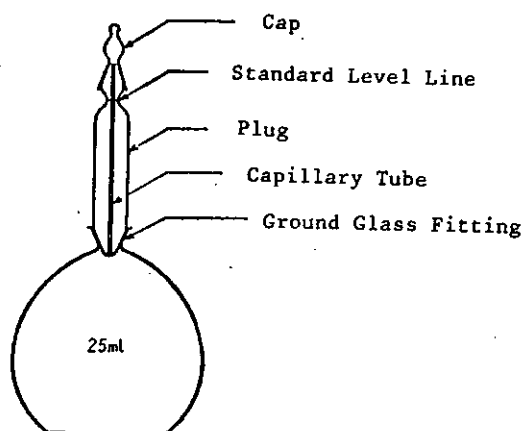
2. Balance: Balance with precision less than 0.01 %.
3. Thermometer: Thermometer with less than 0.2 degree C scale interval.
4. Thermostatic Water Bath: Water bath whose temperature is controlled to be constant within 0.2 degree C.

Calibration:

Sampling:

Conditioning:

Procedures:

1. Fill the specific gravity bottle with a mixture of the chromic acids, and wash it thoroughly with water after one or two days(1). Then wash it with appropriate volatile solvent and dry it.
2. Keep the bottle in the desiccater until its temperature reaches room temperature, and weigh it (W_1).
3. Put the suitable size of the cracked sample into the bottle, and weigh it (W_2).
4. Pour the liquid into the bottle until the sample is sunk in the liquid, and remove bubbles completely. Use a vacuum pump or a vacuum desiccater, if necessary.
5. Add the liquid until the bottle is filled up, and put it in a thermostatic water bath. Then set its temperature to t degree C.
6. After the temperature of the bottle being to t degree C, adjust the meniscus to the standard level, and take out the bottle.
7. After the bottle is wiped thoroughly, leave it until its temperature becomes room temperature and weigh it (W_3).
8. Fill the bottle only with the liquid, setting its temperature to t degree C, and adjust the meniscus to the standard level. Weigh it at room temperature after thorough wiped (W_4).

(1) If not necessary, ignore this procedure.
(2) Distilled water is generally used as the liquid. In the case of samples which are water soluble or form hydrates, other liquid whose specific gravity is known must be used.

Calculation Method (if necessary, please include correction method):

Specific gravity s t/4 degree C is calculated from

$$s\ t/4 = \frac{(W_2 - W_1)(s - sa)}{(W_4 - W_1) - (W_3 - W_2)} + sa$$

where W_1 : weight of the specific gravity bottle in the atmosphere,
W_2 : weight of the specific gravity bottle contained the sample in the atmosphere,
W_3 : weight of the specific gravity bottle contained the sample and the liquid at t degree C in the atmosphere,
W_4 : weight of the specific gravity bottle contained the liquid at t degree C in the atmosphere,
s : specific gravity of the liquid at t degree C,
sa : specific gravity of air equal to 0.0012

Interpretation/Precision & Accuracy:

Precision of repeatability: less than 1 with the least sigificant degit

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

   Linear Thermal Expansion of Solid Materials by Thermodilatometry

Abstracted Scope (200 words):

This method covers determination of the linear thermal expansion of solid
materials using thermomechanical analysis techniques. It is applicable to
solid materials that exhibit sufficient rigidity over the test temperature
range such that the sensing probe does not produce indentation of the
specimen.

The lower limit of coefficient of linear thermal expansion normally
measured with this method is 5 um/(m.°C). The method may be used at lower
or negative expansion levels with decreased accuracy and precision.

The method is applicable over the temperature range from -120 to 600 °C.
This temperature range may be extended depending upon the instrumentation
and calibration materials used.

Referred Document:

ASTM E 831   Standard Test Method for Linear Thermal Expansion of Solid
             Materials by Thermodilatometry

Principle of Measurement:

This method uses a thermomechanical analyzer or similar device to determine the linear thermal expansion of solid materials when subjected to a constant heating rate. Changes in specimen length are electronically recorded as a function of temperature. The coefficient of linear thermal expansion can be calculated from these recorded data.

Apparatus:

A thermomechanical analyzer, or similar device consisting of:

1. A specimen holder and probe, constructed of low thermal expansion materials, that transmits changes in specimen length to a transducer. The probe shape and size shall not cause indentation of the specimen over the temperature range of interest with the load applied. Flat, circular probes of 2 to 5-mm diameter are normally used for this method.

2. A transducer, with a sensitivity of at least 1 mV/um of probe movement, for sensing movement of the probe resulting from changes in specimen length and translating these changes into electrical signals suitable for input into a recording device.

3. A furnace with provision for uniformly heating and cooling the test specimen over the temperature range of interest.

4. A temperature sensing element for measuring the specimen temperature.

5. A means of purging the specimen environment with a dry inert gas such as nitrogen or helium.

6. A means of recording changes in specimen length versus temperature.

7. Calipers, capable of measuring linear dimensions to $\pm$ 25 um.

Sampling:

Specimens shall be between 2 and 10 mm in length and have flat and parallel ends to within 25 um. Lateral dimensions shall not exceed 10 mm.

Specimens are normally measured as received. Heat or mechanical treatment to relieve stresses, distortion, etc., should be noted in the report.

Conditioning:

Procedures:

1. Calibrate the apparatus, both for length change and temperature, using materials of known melting point and thermal expansion.

2. Measure the specimen length in the direction of the thermal expansion test at room temperature, install the specimen in the apparatus, and bring the specimen temperature to at least 20°C below the lowest temperature of interest after purging with inert gas.

3. Place weights, typically 1 to 3 g, on the probe to ensure contact between the probe and specimen.

4. Heat the specimen at a constant heating rate, normally 5°C/min, over the desired temperature range and record changes in specimen length.

5. Make a run using the same test conditions, without a test specimen installed, to establish a baseline for the expansion of the apparatus.

6. Test at least three different specimens of the same material.

Calculation Method (if necessary, please include correction method):

$$a_m = (L_{sp} \times k)/(L \times T) \qquad \text{where}$$

a_m = mean coefficient of linear thermal expansion, um/(m.°C)
L = specimen length at room temperature, m
L_{sp} = change of specimen length, um
T = temperature difference over which the change in specimen length is measured, °C
k = calibration coefficient which is determined by measuring a reference sample having a known expansion coefficient and solving the equation for k

Interpretation/Precision & Accuracy:

Repeatability: Two results obtained by the same laboratory and instrument should be considered suspect (95 % confidence level) if they differ by more than 2.7 um/(m.°C).

Reproducibility: Two averages (each the average of duplicates) obtained by different instruments or laboratories should be considered suspect (95 % confidence level) if they differ by more than 3.1 um/(m.°C).

Accuracy is anticipated, from calculations, to vary with specimen size and magnitude of the coefficient of thermal expansion from <2 to >15 %.

REPORTS ON HEAT STORAGE MATERIALS

2-a-(vii) DENSITY OF LIQUID PHASE

REPORTING FORMAT

Date: Feb. 28. 1986   **Reporter's Name:** Kenji Matsuki

Country: Japan

Testing Title:

Measurement Method of Specific Gravity of Storage Materials in Liquid Phase

Abstracted Scope (200 words):

1. In this method the specific gravity of the storage material is measured with the use of a specific gravity bottle.

2. The density of the material is equal to the specific gravity multiplied by the density of water at 4 degree C and 1.013250 bar.

Referred Document:

JIS Z 8804-1976
"Methods of Measuring Specific Gravity of Liquid"

Principle of Measurement:

The specific gravity of the sample is determined from the ratio of the weight of the sample to the weight of the same volume of pure water in the pressure of 1.013250 bar.

Apparatus:

1. Specific Gravity Bottle: Glass bottle with ground glass plug to minimize the loss of the liquid by expansion and evaporation.

- Cap
- Standard Level Line
- Plug
- Capillary Tube
- Ground Glass Fitting

25ml
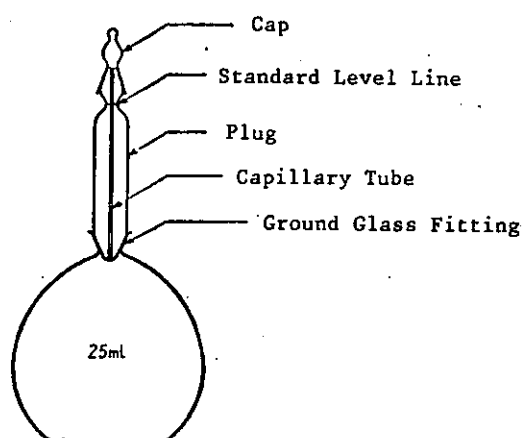
2. Thermometer: Thermometer with less than 0.1 degree C scale interval.

3. Thermostatic Water Bath: Water bath with a stirrer whose temperature is controlled to be constant within 0.1 degree C.

Calibration:

Sampling:

Conditioning:

Procedures:

1. Wash and dry the specific gravity bottle, or wash it using the sample liquid two or three times. Fill the bottle with the sample whose temperature is adjusted to near the temperature of measurement (e.g. 15±2 degree C), avoiding bubbles mixed in the sample. Then plug it, and keep it in a thermostatic water bath for approximately an hour at the temperature of measurement (e.g. 15±0.1 degree C).

2. Adjust the level of the meniscus to the standard level.

3. Set the cap and wipe the water outside the bottle with clean and dry cloth.

4. After the temperature of the bottle becomes room temperature and the absense of bubbles in the bottle is confirmed, weigh the bottle accurately.

Calculation Method (if necessary, please include correction method):

Specific gravity s t/t is calculated from

$$s \, t/t_c = \left\{ \frac{W_3 - W_1}{W_2 - W_1}(St - \rho) + \rho \right\} \frac{1}{St_0}$$

where $s \, t/t_0$: specific gravity t/t_0 degree C,

W_1 : weight of the specific gravity bottle in the atmosphere,

W_2 : weight of the specific gravity bottle contained pure water at t degree C in the atmosphere,

W_3 : weight of the specific gravity bottle contained the sample at t degree C in the atmosphere,

St : specific gravity of water at t degree C (Table 1),

ρ : specific gravity of air during measurement (Table 2),

St_0 : specific gravity of water at t_c degree C.

Interpretation/Precision & Accuracy:

Precision of repeatability: less than 0.0002.

REPORTS ON HEAT STORAGE MATERIALS

2-b(ii)   IGNITION POINT

5

Date: 8/86   Reporter's Name: David Waksman

Country: U.S.A.

Testing Title:

Autoignition Temperature of Liquid Chemicals

Abstracted Scope (200 words):

This method covers the determination of hot- and cool-flame autoignition
temperatures of a liquid chemical in air at atmospheric pressure in a
uniformly heated vessel.

Autoignition is evidenced by the sudden appearance of a flame and by a
sharp rise in temperature of a gas mixture inside a glass flask heated
to a predetermined temperature. The lowest temperature at which this occurs
is taken as the autoignition temperature.

Within certain limitations, this method can also be used to determine the
autoignition temperature of solid chemicals which readily melt and vaporize
at temperatures below the test temperature.

Referred Document:

ASTM E 659 Standard Test Method for Autoignition Temperature of
            Liquid Chemicals

<u>Principle of Measurement</u>:

The autoignition temperature is the lowest temperature at which a flame and
a sharp rise in temperature occur when a chemical is inserted into a heated
glass flask containing air at a predetermined temperature. Hot-flame
temperatures are evidenced by the sudden appearance of a flame and a sharp
rise in temperature. Cool-flame ignitions are evidenced by small sharp
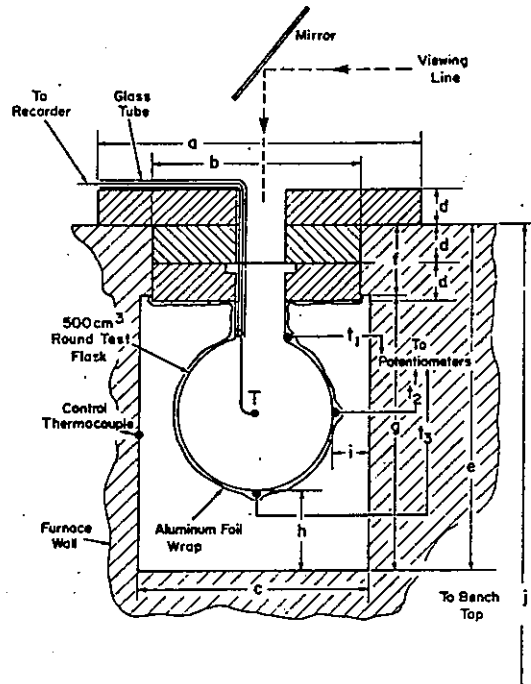rises in temperature and a faint, pale blue luminescence or flame.

<u>Apparatus</u>:

FIG. 1 Autoignition Temperature Apparatus.

<u>Sampling</u>:

<u>Conditioning</u>:

Procedures:

1. Allow the flask to reach the desired internal flask temperature and maintain to within 1°C for temperatures up to 350°C and to within 2°C above 350°C.

2. Extinguish the lights as the sample is inserted to allow observation of the flame. Hot flames can be observed in subdued lighting; cold-flame tests are generally conducted in total darkness.

3. Inject 100 uL of liquid samples or insert a 100-mg powder sample into the flask, start the timer, and place a mark on the recorder trace of the flask interior thermocouple temperature.

4. Observe the inside of the test flask in total darkness using the mirror. If ignition is not observed in 10 min, the sample is nonflammable at the concentration and flask temperature tested. Completely purge the flask with a hot-air gun and repeat the procedure at a 30°C higher temperature

5. If ignition occurs, record the time interval between sample insertion and ignition as the ignition delay time. Lower the test temperature by 30°C and repeat the procedure until autoignition is no longer observed. Narrow the test interval to 3°C and determine the break point between ignition and nonignition.

6. Repeat with larger, and then smaller samples, until the minimum autoignition temperature is obtained.

Calculation Method (if necessary, please include correction method):

A plot of the logarithm of the delay time versus the reciprocal of the absolute temperature normally results in a straight line passing through both the autoignition and cool-flame test results.

Interpretation/Precision & Accuracy:

The following criteria should be used for judging the acceptability of results (95% confidence):

Repeatability: Duplicate results by the same operator should be considered suspect if they differ by more than 2 % of the temperature in degrees Celsius.

Reproducibility: The results submitted by each of two laboratories should be considered suspect if the two results differ by more than 5 % of the temperature in degrees Celsius.

REPORTS ON HEAT STORAGE MATERIALS

2-b (iii) FLASH POINT

REPORTING FORMAT

Date: Feb. 19, 1986   Reporter's Name: Kenji Matsuki

Country: Japan

Testing Title:

> Testing Method for Flash Point of Storage Materials
> (Cleveland Open Method)

Abstracted Scope (200 words):

1. This method is applied for materials whose flash points are higher than 80 degree C.

2. Sample contained in an open cup is heated at a defined rate of temperature raise. In the vicinity of the flash point, a defined size of small fire is passed over the cup every 2 degree C temperature raise. Then the lowest temperature where the sample flashes is measured.

3. This method can be also applied for measurement of burning point where a sample burns continuously.

Referred Document:

> JIS K 2265
> "Testing Methods for Flash Point of Crude Oil and Petroleum Products"

Principle of Measurement :

The sample is heated up gradually and a small fire is passed over the open cup of the sample. The lowest temperature where the sample flashes is the flash point.

Apparatus :

Sample Cup
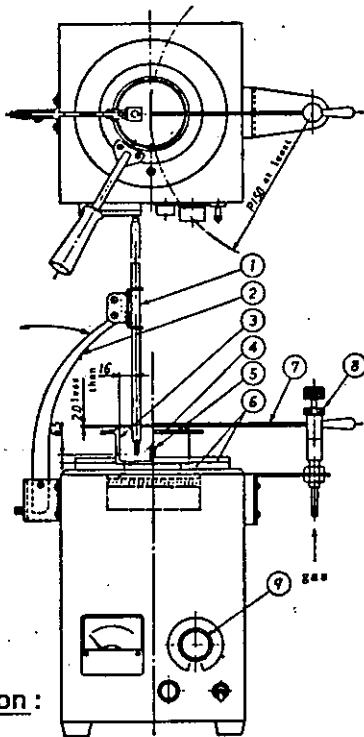
① Thermometer

② Holder of Thermometer

③ Cup of Sample

④ Electric heater

⑤ Standard Lamp

⑥ Heating Panel

⑦ Nozzle of Fire

⑧ Gas Control Valve

⑨ Heating Controller

Handle
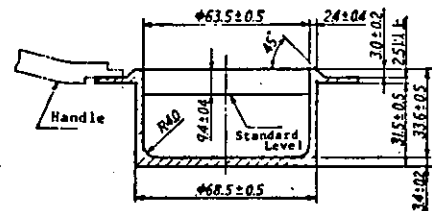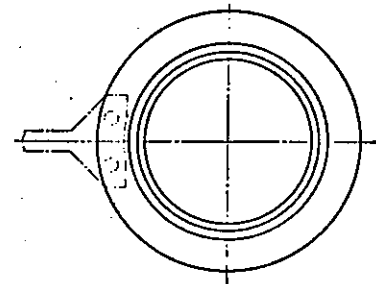
unit mm

Calibration :

Sampling :

Sample cup must be filled with the sample up to a standard level of the cup and air bubbles must be removed. Samples of high viscosity may be heated up to fill the cup at least 55 degree C below the expected flash point.

Conditioning :

Procedures :

1. Light up a testing fire and adjust its size to be the same as the standard ball or 4±0.8 mm in diameter.

2. Begin heating at the rate of 14 to 17 degree C/min. When the sample temperature reaches 55 degree C below the expected flash point, adjust the heating rate to 5.5±0.5 degree C/min.

3. When the sample temperature reaches 28 degree C below the expected flash point, move the testing fire as described below at every 2 degree C temperature raise. Move the fire across the sample cup through its center along by a straight line or an arc whose radius is larger than 150 mm in approximately one second. During this, the center of fire must be kept within 2 mm above the edge of the sample cup and moved horizontally.

4. When the clear flash is recognized on the surface of the sample, record the reading of the thermometer.

Calculation Method (if necessary, please include correction method) :

Correction for Atmospheric Pressure

In the case when atmospheric pressure during measurement is less than 715 mmHg (95.32 kPa), correction value must be added to the measured temperature.

Atmospheric pressure mmHg{kPa}	Correction value degree C
715 – 635 {95.32 – 84.66}	2.8
634 – 550 {84.53 – 73.33}	5.5

Interpretation/Precision & Accuracy :

Precision of repeatability: less than 8 degree C.
Precision of reproducibility: less than 16 degree C.

REPORTS ON HEAT STORAGE MATERIALS

c-(iii) DURABILITY FOR HEAT CYCLES

REPORTING FORMAT

Date :   Feb. 28, 1986   Reporter's Name : Kenji Matsuki

Country :   Japan

Testing Title :

Testing Method of Durability of Storage Materials for Heat Cycles

Abstracted Scope (200 words):

1. In this method a storage material contained in a container is cyclically heated and cooled in a water bath. During the test the temperature of a material is to be measured to assure the durability in a desired testing condition.

2. In the case of latent heat storage materials, the upper temperature of the heat cycle must be higher than the phase change temperature of the materials and it is the same for the lower temperature of the heat cycle to some extent.

3. The durability of a material is evaluated in terms of thermal properties, such as the specific heat, the latent heat, the phase change temperature, the chemical composition, and so on.

Referred Document :

H. Kimura, J. Kai: Improvement of Phase Change Stability of $CaCl\ 6H_2O$ as a Latent Heat Storage Material, Journal of Energy and Resources, Vol. 5(1984), p 586.

53

Principle of Measurement:

Since storage materials undergo cyclic heating and cooling in practice, the durability of materials for heat cycles must be tested under a realistic condition.

Apparatus:
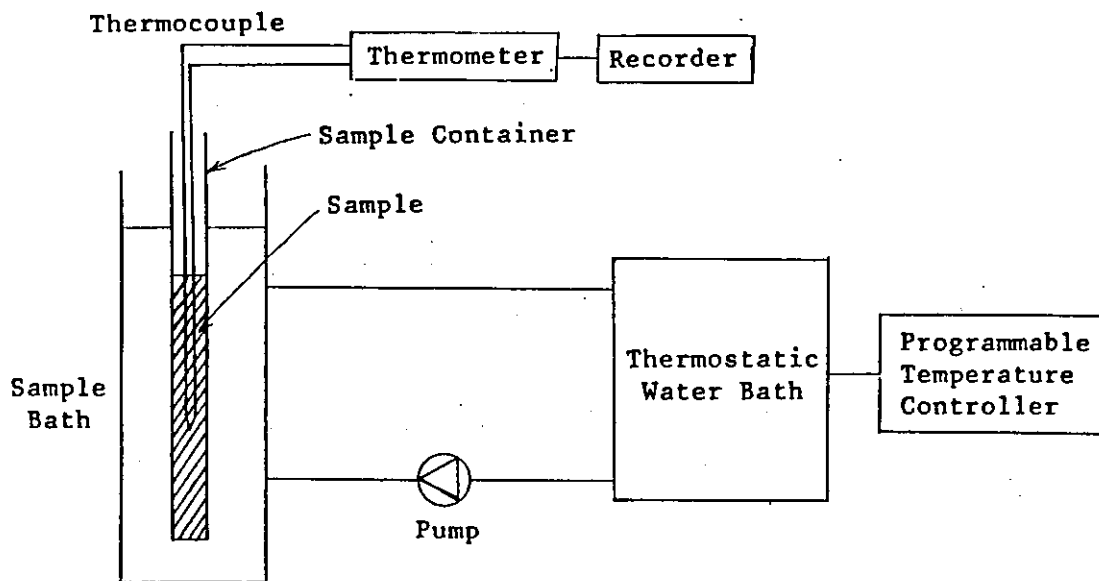
Calibration:

Sampling:

Put a material in a container which does not react with the material in the testing temperature range.

Conditioning:

554

Procedures:

1. Set a sample container in a sample bath.

2. Circulate water between the thermostatic water bath and the sample bath.

3. Change the temperature of the water bath cyclically with a programmable temperature controller.

4. After some desired cycles, measure the properties of the sample.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

Durability of the material must be evaluated in terms of critical properties for utilization.

REPORTING FORMAT

Date: 1986-04-25   **Reporter's Name**: Bo Carlsson

Country: Sweden

Testing Title:

Chemical Heat Pumping - A rapid experimental procedure for investigating the suitability of salt hydrates under dynamic conditions.

Abstracted Scope (200 words):

A rapid experimental procedure of interest in determining the suitability of salt hydrates to be used in chemical heat pumping is described. Thermogravimetry under controlled water vapour pressure is utilized as the key diagnostic method. The test procedure relies largely on two critical tests, _viz._ a cycling and an inhibition test. The former gives information on the stoichiometric reversibility and hysteresis between the dehydration and rehydration branches, while the inhibition test yields more quantitative information about the extent of inhibition. The latter represents a source of irreversibility inherent to the salt hydrate system.
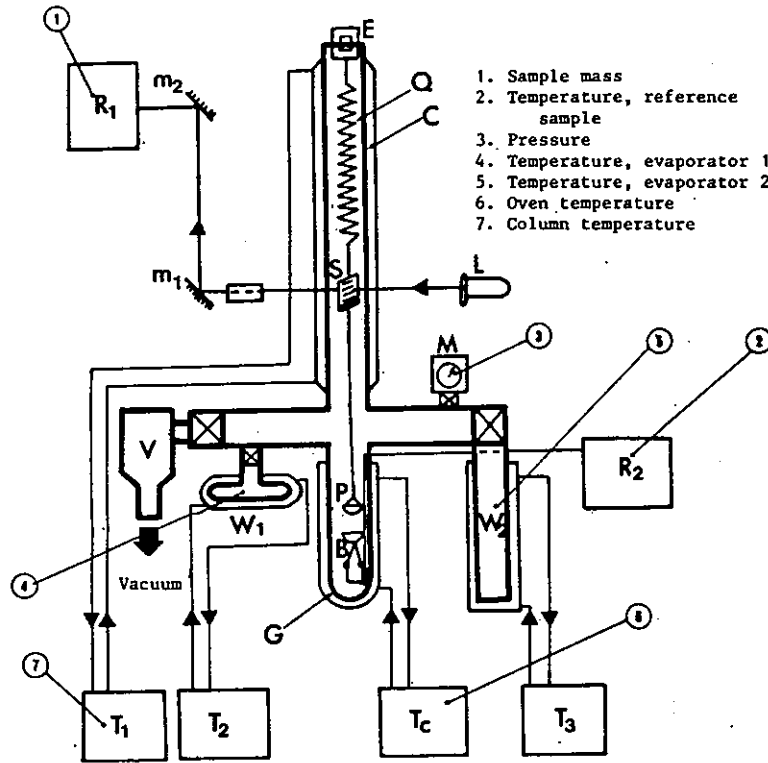
Referred Document:

"Chemical Heat Pumping - A rapid experimental procedure for investigating the suitability of salt hydrates under dynamic conditions "Jan Y Andersson, Michel Azoulay and Joan De Pablo in thesis "Kinetic and Mechanistic Studies of Reactions between Water Vapour and some Solid Sorbents" by Jan Y Andersson at The Royal Institute of Technology Stockholm 1984 (TRITA-FYK-8404)

Principle of Measurement:

A Mc Bain quartz spring balance, Maintained at a constant temperature, is used to measure the weight change through an optical system of a sample (P). An identical sample B is used for measurements of sample temperature. The sample compartment is maintained at a temperature T_c. The experimental data are collected and treated by a microcomputerized acquistion system.

Apparatus:

1. Sample mass
2. Temperature, reference sample
3. Pressure
4. Temperature, evaporator 1
5. Temperature, evaporator 2
6. Oven temperature
7. Column temperature

Calibration: see referred document

Sampling: see referred document

Conditioning: se referred document

REPORTING FORMAT

Date:	Reporter's name: P. Kratz
Country:	Technisch Physische Dienst TNO-TH (TNO Institute of Applied Physics) P.O. Box 155, 2600 AD DELFT The Netherlands

Testing title: AGEING TESTS OF REPRESENTATIVE SAMPLES BY MEANS OF THERMAL CYCLING UNDER REALISTIC CONDITIONS.

Abstracted Scope (200 words):

This test was developed for the determination of the long time performance and stability of especially salt hydrates.

Possible phenomena that can influence the long time behaviour of a particular PCM are:

- chemical degradation
- interaction with container materials
- segregation (salt crystals tend to sink, driving water to the top if the thickener deteriorates, S gets worse with time)
- undercooling (solidification at temperatures lower than melting temperatures, if the nucleing agent deteriorates, U gets worse with time).

To investigate the influence of these phenomena on the PCM the ageing test is carried out on several samples of 1-4 kg PCM, packed in steel tube containers that are sealed by means of welding. To distinguish the influence of gravity on segregation, one sample is placed vertically, one horizontally, and one vertically but turned every 14 days.

The temperature of the samples is cycled, 4 hours high, 4 hours low etc. the heat input and output is continuously measured.

Referred document:

None.

- 1 -

555

Principle of measurement:

While the ageing is performed, flow and temperature difference over the samples are measured. From these the heat input and output are calculated. Measurements during each stabilisation before the temperature change allow for compensation of heat losses and measurement offsets.

Apparatus:

The samples are placed in perspex containers (A). The samples are heated and cooled to the requested temperature by water from a thermostat bath that is circulated through the perspex containers. The temperature is cycled by means of a timer (B) that switches the high/low temperature setting of the thermostat bath (C). 4 hours high and 4 hours low enable to do three cycles per day. The water flow through each container is regulated with flow control valves (D) and measured with gapmeters (E). Temperatures are measured on each container with absolute and differential thermocouples (F) that are connected to a datalogger that records the temperatures on magnetic tapes.

Calibration:

The flow meters are checked against positive displacement meters of known accuracy.
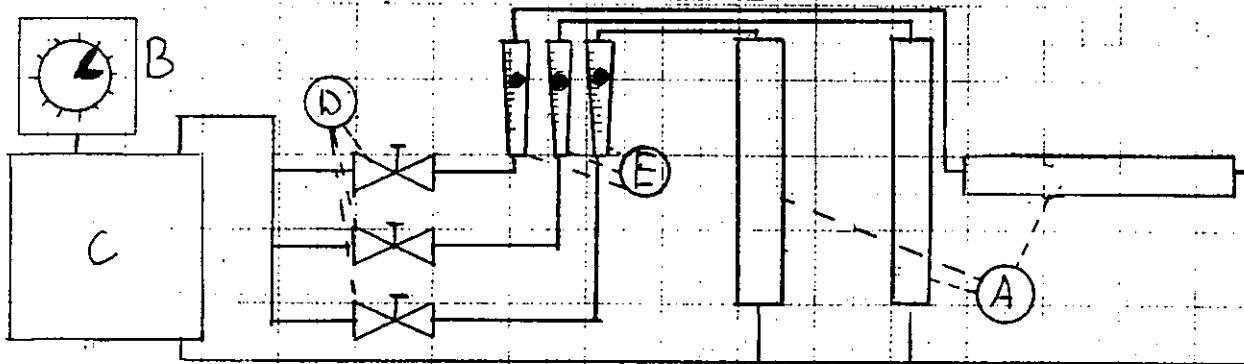
Sampling:

Samples are taken from the producers batch, and are sealed in steel containers (normally the pcm is used in the same steel containers).

Conditioning:

None.

Procedures:

Before the tests are carried out all components in between the differential thermocouples have to be weighed, this is necessary to calculate the latent heat from the measurements.
After the testing the steel containment of the samples can be opened for visual inspection of the PCM and additonal DTA measurements can be done.

Calculation method (if necessary, please include correction method):

The data tapes are processed with a computer-programme, in which the heat losses and temperature measurement errors are compensated using the measurements during the stabilisation time before and after each temperature step.
The heat inputs and outputs are computed from the flows and different temperatures and are converted to latent heat values, using the specific heat and weight of the perspex containers, water contents and the steel containment etc.

Interpretation/Precision & Accuracy:

The results are latent heat values as a function of time and number of cycles.
A one year ageing test = 1000 cycles gives a good prediction of the long time behaviour if the cycling is the dominant ageing factor (this is expected for salt hydrate PCM's). However, if the total time on high temperature is most important, tests have to be even longer to predict the behaviour of the PCM during the storage lifetime. Shorter testing by using higher temperatures seem not feasable because than new physical and chemical problems are introduced.

REPORTS ON HEAT STORAGE MATERIALS

3-a DESICCANT MATERIALS, THERMAL PROPERTIES

(i) EQUILIBRIUM VAPOR PRESSURE AS A FUNCTION OF TEMPERATURE

Date : Feb. 28, 1986   Reporter's Name : K. Matsuki

Country : Japan

Testing Title :

Measuring Method for Equilibrium Vapor Pressure as Function of Temperature

Abstracted Scope (200 words) :

1. This method deals with the measurement of equilibrium vapor pressure for a desiccant material.

2. Temperature and equilibrium pressure of a sample are measured with a constant temperature bath which contains the sample vessel.
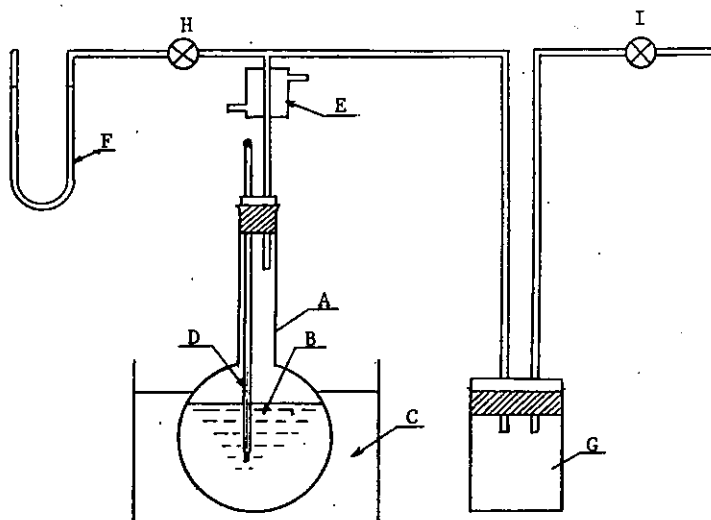
Referred Document :

Laboratory method

562

Principle of Measurement:

The pressure of the vessel is measured when the temperature of the sample becomes constant.

Apparatus:

A: Measuring Bottle
B: Sample
C: Bath
D: Thermometer
E: Condenser
F: Manometer
G: Constant Pressure Vessel
H: Valve 1
I: Valve 2

Calibration:

Sampling:

Conditioning:

Room temperature

Procedures :

1. Put the sample B, whose equilibrium vapor pressure is to be measured, into the measuring container A.

2. Steep the measuring container A into the bath C whose temperature must be constant in order to keep the temperature of the sample B at a set value.

3. In the case when the sample B is water solution, increase the degree of vacuum after the temperature of the water solution reaching a set value.

4. In the case when the sample B is solid, increase the degree of vacuum as high as possible after the temperature of the sample reaching the set value. Then leave for a certain time by closing valve I, and measure the pressure when the reading of a manometer F becomes constant. This pressure gives the equilibrium pressure.

Calculation Method (if necessary, please include correction method) :

Interpretation/Precision & Accuracy :

56

Date: Mar.14,1986   Reporter's Name: Koji TAJIRI

Country: JAPAN

Testing Title: (3)(a)-(1)

Equilibrium vapor pressure as a function of temperature
of desiccant matereals.

Abstracted Scope (200 words):

It is indispensable to get the equilibrium water vapor
pressure on the adsorbent materials,(e.g. zeolite,silica
gel) used for desiccant air conditioning,for designing and
estimating the running performance of it.

Thus, for the candidate materials selected by other
thermodynamic properties as heat of adsorption, we determined
the relation between equilibrium water vapor pressure and
temperature by the method of maximun adsorption.

While,on inorganic salt hydrate,received much attention
for heat storage, chemical heat pump etc., we determine
the equilibrium water vapor pressure.

Referred Document:

T.Asahina et al., Thermophysical Properties 3, 3-49(1982)

Principle of Measurement:

The determination of maximum adsorption quantity of water on the adsorbent in constant water vapor pressure and constant temperature.
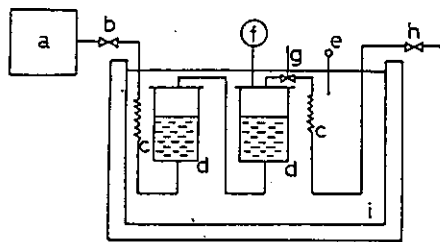
Apparatus:

Fig. Schematic diagram of the humidity generator.

a:compressor,b:pressure control valve,
c:heat exchangers,d:saturation tanks,
e:thermometer,f:pressure gauge,
g:flow control valve,h:shut valve,
i:water bath.

Calibration:

Sampling:

some 10 g of dried adsorbent are weighted.

Conditioning:

Procedures:

Adsorbent dried in vacuum oven(for zeolite, 0.1torr, 300°C) were weighted. Then it was put into the thermobath in which there were constant temperature and humidity by the air through humidity generator. Adsorbed quantities of water in that temperature and water vapor pressure were gained from maximum weight increase, and then, the equilibrium vapor pressure were estimated.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

REPORTS ON HEAT STORAGE MATERIALS

3-a (ii) REACTION RATE OF DESICCANT MATERIAL WITH WATER VAPOR

Date :   Feb. 28, 1986   **Reporter's Name :** K. Matsuki

Country :   Japan

Testing Title :

Measuring Method for Reaction Rate of Desiccant Material with Water Vapor

Abstracted Scope (200 words) :

1. This measuring method concerns with the reaction rate of a desiccant material with water vapor.

2. Set a sample wheel, into which a desiccant material is permeated, in the constant temperature and humidity chamber (30 deg. C, 80 %RH). Then measure the the weight of the sample wheel.

3. Obtain the water vapor absorption rate which is determined from the weight change of the wheel.

4. Wheel is made by rolling up a sheet of corrugated material together with a flat sheet material into a cylindrical drum.
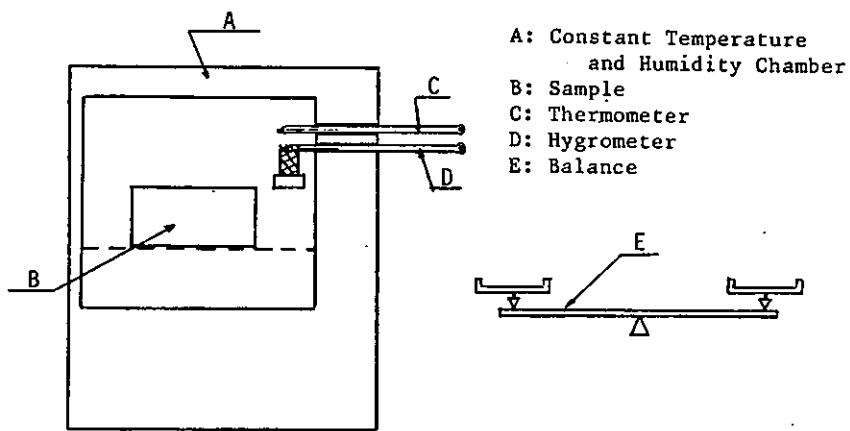
Referred Document :

Laboratory method

Principle of Measurement:

Measure the water vapour absorption weight in the time variation at constant condition of surroundings of 30 deg. C (80% RH).

Apparatus:

A: Constant Temperature and Humidity Chamber
B: Sample
C: Thermometer
D: Hygrometer
E: Balance

Calibration:

Sampling:

The dimension of the wheel is 150-200 mm in diameter and 50-70 mm in thickness. The weight of the desiccant material to be permeated is 5 % to the weight of the wheel.

Conditioning:

Measurment is to be done in the surroundings of 30 deg. C (80% RH).

Procedures :

1. Perfectly dry a sample B, in which a certain weight (W) of a desiccant material is permeated, and weigh (Wo) the sample with the balance G.

2. Place the sample B into the constant temperature and humidity chamber A of which the condition is kept at 30 deg. C and 80 %RH.

3. Take out the sample B at a certain period, and weigh (Wt) the sample. The absorption ratio is calculated from

$$\frac{Wt - Wo}{W}$$

4. Obtain the relationship between the absorption ratio and time.

Calculation Method (if necessary, please include correction method) :

Interpretation/Precision & Accuracy :

Temperature: $\pm$ 0.5 degree
Humidity   : $\pm$ 3 %RH
Weight   : 0.5 %

REPORTS ON HEAT STORAGE MATERIALS

3-c (i) DURABILITY FOR DESICCANT CYCLES

Designation :

SD–D–LA

Date: Feb. 28, 1986   **Reporter's Name:** K. Matsuki

Country: Japan

Testing Title:

Testing Method for Durability of Desiccant Materials

Abstracted Scope (200 words):

1. This testing method concerns with the durability of desiccant materials which are subject to charge/discharge of moisture.

2. Observe the time variation of a sample, into which a desiccant material is permeated, when air of 30 deg. C (80% RH) and air of which temperature is raised up to the regenerative temperature of the material is passed to the sample alternatively.

3. Wheel is made by rolling up a sheet of corrugated material together with a flat sheet material into a cylindrical drum.
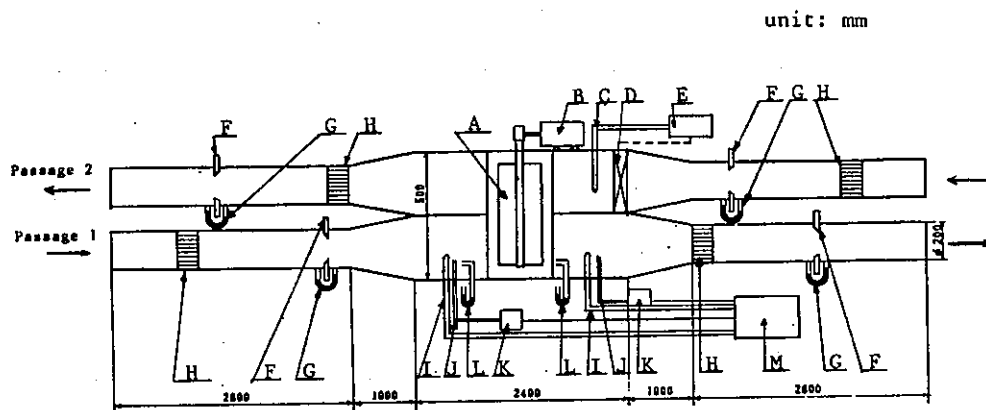
Referred Document:

Labolatory· method

Principle of Measurement:

Observe the dehumidifying capacity and the pressure drop of the wheel with the lapse of the cycle of humidification and dehumidification.

Apparatus:

unit: mm

A: Sample
B: Motor
C: Thermometer
D: Heater
E: Temperature Controller
F: Orifice Meter
G: Pressure Gauge
H: Lectifying Lattice
I: Thermometer
J: Hygrometer
K: Interface
L: Pressure Gauge
M: Recorder

Calibration:

Sampling:

The dimension of the wheel is 150-200 mm in diameter and 50-70 mm in thickness. The weight of the desiccant material to be permeated is 5 % of the weight of the wheel.

Conditioning:

Ambient testing conditions are 30 deg. C and 80% RH.

Procedures:

1. Set the wheel A, into which a desiccant material is permeated, to the test rig. Then rotate the wheel A by a motor B at the rate of 15-20 rph.

2. In the test rig, air passage is divided into two passages, i.e. passage 1 and passage 2. In the passage 1 air of 30 deg. C (80% RH) is passed. On the other hand, air which has been heated up to 100 deg. C by a electric heater D, is passed in the passage 2.

3. Determine the air flow rate of two passages in order that the air speed in front of the wheel may become 1-2 m/s.

4. The temperature, the humidity and the static pressure at the inlet and the outlet of the wheel in the passage 1 are measured with thermometer I the hygrometer J and the static pressure gauge L.

5. Define the time variation of characteristics of the wheel with the measurment of the humidity difference and the static pressure difference between the inlet and the outlet of the wheel in the passage 1. The measurment is to be taken in every 1000 hours.
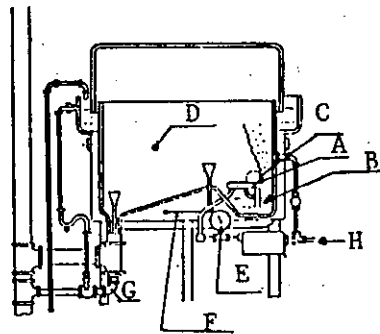
Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

Principle of Measurement:

Observe the surface condition (rust, cracks, peeling etc.) of a sample tested with the salt spray.

Apparatus:

A: Nozzle
B: Salt Water
C: Window
D: Thermometer
E: Pressure Gauge
F: Heater
G: Drain
H: Saturated Air

Calibration:

Sampling:

Specimen: 50 mm x 150 mm

Conditioning:

Room temperature

Procedures:

1. Wash the specimen.

2. Decline the principal surface of the specimen to 15-30 deg. from the plumb line.

3. Prepare salt water of which concentration is 5 ± 1 %.

4. Keep the temperature of the salt spraying chamber at $35\pm2°C$. The PH range of the salt water is 6.5-7.2 when spraying at $35°C$.

5. Keep the pressure of supplied air at 0.0687-0.1765 MPa.

6. The rate of the spray is 0.5-3.0 ml/h, which is measured with the collecting vessel of which horizontal opening is 80 cm².

7. Test cycle is 8 hours' salt spraying and 16 hours' pause at the temperature of 15-35°C. Repeat this cycle 20 times and check rust, crack and peeling on the specimen surface.

Calculation Method (if necessary, please include correction method):

Interpretation/Precision & Accuracy:

APPENDIX   II

LIST OF THE SUBTASK B PARTICIPANTS

The reports compiled here were communicated from the
below described participants.

FRG
   Prof. Dr. Ing. K. Tostmann   Fachhochschule, Aalen
   Dr. J. P. Baselt   DECHEMA
   Dr. K. Gindele   University of Stuttgart

Japan
   Dr. T. Noguchi   JMI
   Dr. K. Hayakawa·   GIRI, Nagoya/AIST/MITI
   Dr. S. Tanemura   GIRI, Nagoya/AIST/MITI

The Netherlands
   Dr. A. J. Faber   TNO-TH

Switzerland
   Mr.·U. Frei   Technical Highschool
                             ITR

Sweden
   Dr. B. O. Carlsson   National Testing Institute

UK
   Dr. M. Huchins   Oxford Polytechnic

USA
   Dr. D. Waxman   NBS
   Dr. C. M. Lampert   Lawrence Berkeley Labo-
                             ratory

The followings are the list of participants who wish
to share the result of the survey research.

Austria
  Prof. G. Faninger   Austrian Solar and
                                       Space Agency

Denmark
  Dr. T. Vest Hansen   Danish Solar Energy
                                       Testing Laboratory

Spain
  Dr. Ing. E. Mezquida   INTA