Working Group 9 report to CCT, May 2003 (7 pages)

Members

John Redgrove (Chairman)	NPL	UK
Giancarlo Bussolino	IMGC	Italy
Jean-Rémy Filtz	BNM-LNE	France
Gerald T. Fraser	NIST	USA
Sang-Hyun Lee	KRISS	Korea
Juntaro Ishii	NMIJ	Japan
Carlos Nieto de Castro	IPQ	Portugal
Ping Qiu	NIM	China
Stefan Sarge	PTB	Germany

1. Terms of Reference

Extract from the 21st session of the CCT, 12 – 14 September 2001:

4.6 New Key comparisons

It has been brought to the attention of the CCT that members of the thermophysical properties community believe that there is a need for comparisons in the fields of thermophysical quantities. It was decided that the CCT delegates possess insufficient knowledge of the field to address the concern. A Working Group is proposed to identify the needs.

... and later in the minutes:

6.9 Working Group 9 is established as a new Working Group on thermophysical properties. The CCT accepted John Redgrove (NPL) as chairman, and NIST (Gerald Fraser - new entry 3 Oct 01), IMGC (...), IPQ (Carlos Nieto de Castro), KRISS (Sang Hyun Lee), PTB (Stefan Sarge), NIM (Ping Qiu) NMIJ (Juntaro Ishii) and BNM-INM (Jean-Rémy Filtz) as members.

The Terms of Reference are:

To advise the CCT on matters related to thermophysical properties, and to assess the need in this subject field for a key comparison.

2. Key Comparisons

The first meeting of WG9, held in September 2002, considered the possibility of undertaking a key comparison, carried out over 2- 4 years. It was agreed that **partners** for a key comparison should satisfy the following conditions:

- should be a signatory of the Mutual Recognition Arrangement
- must have evidence of suitability such as traceability to a national standard and an uncertainty budget compliant with the *Guide to the Expression of Uncertainty in Measurement* (GUM) published by ISO in 1995

Candidate properties identified were:

thermal conductivity, thermal diffusivity, heat capacity and **emissivity**.

Some candidate **materials** for a comparison are listed below. Materials should be stable, reproducible, isotropic and homogeneous. It's best if they are already reference materials, and even better if their thermal properties are unknown.

Candidate materials for key comparisons	
Solids	**Properties**
Pyroceram 9606	λ, a, Cp
Glass fibre (available from IRMM, Belgium)	λ
Pure metals: In, Sn, Pt	λ, ε
Pt	Cp
W, Mo	λ, Cp
Referceram (alumina)	a
Glassy carbon	a
Liquids	**Properties**
Water	λ, Cp
Toluene	λ, Cp
Molten tin	λ, Cp

Key:
λ – thermal conductivity
a – thermal diffusivity
Cp – specific heat capacity
ε – emissivity

3. Definition of thermophysical properties

WG9 needs to determine the scope of its interest area– see the attached informal list of properties.

The following two definitions of **thermophysical properties** were proposed following the meeting.

"All material properties that vary with temperature without altering the material's chemical identity are thermophysical properties ... however, it has become customary to limit the scope of the term to properties having a bearing on the transfer and storage of heat."

or:

"All material properties affecting the transfer and storage of heat, that vary with the state variables temperature, pressure and composition (in mixtures), and of other relevant variables, without altering the material's chemical identity. These properties will include thermal conductivity and diffusivity, heat capacity, thermal expansion and thermal radiative properties, as well as viscosity and mass and thermal diffusion coefficients, speed of sound, surface and interfacial tension in fluids. "

4. WG9 member comments arising since Sept 02 meeting

Sarge

We should first identify all thermophysical properties which we believe are the scope of thermophysics. We should discuss whether we should include, e. g. emissivity, density, electric resistance, heat capacity ...

Measuring techniques applied are in my opinion of minor importance, because as metrologists we should first concentrate on high-level techniques before we deal with industrial applications.

Developing standards in the sense of reference materials as proposed in your issues 1 and 3 should not be the primary goal of our work. The goal is to establish comparability by any suitable means.

Ishii

The WG9 should consider issues related to total and spectral emissivity.

Lee

International comparisons are required for thermal conductivity & thermal diffusivity.

Filtz

High temperature thermal conductivity measurements on insulating materials are inconsistent and a widening of the consortium of NMIs to increase consistency of the results between metrology areas is necessary. Consistency is also lacking in composite materials, of low thickness, multilayer mediums, at higher temperatures than 1000°C and in radiative properties of materials.

For this field, just local or regional comparisons have been performed but it is important to improve significantly the consistency of comparisons. Needs for low and medium temperatures should be considered as a first approach. In conclusion, I see at least four subjects for investigation by the working group:

1) to identify in priority the intercomparisons to set up for the 3 following topics: emissivity (or reflectivity), thermal diffusivity and thermal conductivity in areas where a maximum of national laboratories are ready

2) to assist regional organisations of metrology in the development of the thermophysical lists of thermal properties which concern the CMC. The work is tedious but makes it possible to clarify discussion and give the subject visibility to CCT

3) to work out recommendations for new metrological fields (new materials, and temperature range for example)

4) to initiate or update technical and scientific recommendations on current subjects (good practice guides …)

5. Letter to NMIs

It was proposed at the Sept 02 meeting that a letter should be issued to NMIs, ideally by BIPM on behalf of WG9, to identify national thermophysical property **measurement capabilities**, and possible **reference materials**; and also to obtain any **recommendations for intercomparisons**.

6. Actions

	Action	By whom	By when
1	Approach Michael Stock/Martin de Groot with the idea of forming a list of NMI thermophysical property measurement capabilities (before issuing letter – see 2. below)	John Redgrove	Oct 02
2	If agreed from 1. above draft a **letter** for BIPM to issue to NMIs for reply to BIPM by Dec 02. (The replies to be entered in a spreadsheet adopting the BIPM's CMC format.)	John Redgrove	Oct 02
3	Name a deputy to cover for your absence, providing full contact details. (This will help to ensure that there is always a response to decisions that need to be made by WG9 via email.)	WG9 members	Oct 02
4	Summarise replies from NMIs	John Redgrove	Jan 03
5	John R to draft WG9 recommendations and obtain WG9 agreement before presentation to BIPM by Mar/Apr 03	John Redgrove	Mar 03
6	Comment on what should be the scope of WG9, including a working definition of "thermophysical properties"	WG9 members	Oct 02

Next meeting of WG9

Next meeting to be held during the15th Symposium on Thermophysical Properties in Boulder, Colorado, USA, June 22 - 27, 2003.

Appendices (see below)

1. Possible thermophysical properties within the scope of WG9
2. Thermophysical properties issues and possible need for key comparisons

John Redgrove, May 2003

Appendix 1. Possible thermophysical properties within the scope of WG9

THERMOPHYSICAL QUANTITIES

1 Thermal Conductivity
- 1.1 Absolute Measurements
  - 1.1.1 Longitudinal heat flow
  - 1.1.2 Radial heat flow
  - 1.1.3 Hot wire
  - 1.1.4 Transient hot strip
  - 1.1.5 Others
- 1.2 Relative Measurements
  - 1.2.1 Longitudinal heat flow
  - 1.2.2 Radial heat flow
  - 1.2.3 Others
- 1.3 Reference Materials

2 Thermal Diffusivity
- 2.1 Measurements
  - 2.1.1 Laser flash
  - 2.1.2 Transient hot strip
  - 2.1.3 Others
- 2.2 Reference Materials

3 Thermal Expansion Coefficient
- 3.1 Absolute Measurements
- 3.2 Relative Measurements
  - 3.2.1 Push rod
  - 3.2.2 Others
- 3.3 Reference Materials

4 Emissivity
- 4.1 Total Emissivity Measurements
  - 4.1.1 Calorimetric
  - 4.1.2 Radiometric
- 4.2 Spectral Emissivity Measurements
  - 4.2.1 Blackbody method
  - 4.2.2 Ellipsometry
- 4.3 Reference Materials

5 Reflectivity
- 5.1 Measurements
- 5.2 Reference materials

6 Specific Heat Capacity
- 6.1 Measurements
  - 6.1.1 Adiabatic calorimetry
  - 6.1.2 Drop calorimetry
  - 6.1.3 Differential scanning calorimetry
  - 6.1.4 Relaxation method
  - 6.1.5 Laser flash

6.1.6 Electrical pulse heating
6.1.7 Optical modulation heating
6.1.8 Others
6.2 Reference Materials

7 Heat of Transition (fusion, vaporisation, sublimation, phase transition)

7.1 Measurements
7.1.1 Adiabatic calorimetry
7.1.2 Drop calorimetry
7.1.3 Differential scanning calorimetry
2.1.4 Others
7.2 Reference Materials
7.2.1 Fusion
7.2.2 Vaporisation
7.2.3 Sublimation
7.2.4 Phase/glass transition

8 Heat of Combustion

8.1 Measurements
8.1.1 Combustion in oxygen
8.1.2 Others
8.2 Reference Materials
8.2.1 Combustion in oxygen
8.2.2 Others

9 Heat of Reaction and Solution

9.1 Measurements
9.1.1 Flow calorimeter
9.1.2 Heat conduction calorimetry
9.1.3 Isoperibolic calorimetry
9.1.4 Titration
9.1.5 Differential scanning calorimetry
9.2 Reference Materials

10 Heat Flux

2. Thermophysical properties issues and possible need for key comparisons

Below are some thermophysical property issues or needs that the Thermophysical Properties Group at NPL are aware of and wish to bring to the attention of WG9.

1. Thermal conductivity reference materials and the need for key comparisons

Ambient temperatures

NPL (UK), NIST (USA), NRC (Canada), LNE (France) and JTCCM (Japan) have made thermal conductivity measurements at ambient temperatures on three international reference materials, two different densities of fibre glass and expanded polystyrene and a proposed reference material consisting of a mixed oxide fibre. The results are in good agreement, with a spread of 1% -2 %, and the partners believe that this work could form the basis of a Key Comparison, involving a wider range of NMIs and leading to an MRA for thermal conductivity of a standard reference material.

Elevated temperatures

NPL and other partners, as part of a European project to develop a high temperature reference material, have measured the thermal conductivity and thermal diffusivity of a glassy ceramic, Pyroceram 9606, from room temperature to 1000 °C. The final project report and Certification Report have just been submitted to the EC for their consideration. The aim is to produce Certified Reference Material (CRM) values for this batch of material. There is already a large database of published data on Pyroceram 9606, obtained from a number of different batches, compiled by Flynn and colleagues at NIST. Their aim is to produce a NIST reference material. Ideally, the values obtained by the European project and NIST should agree. However, there is the potential for the assigned values to be different although within the claimed uncertainties of each group and we would have a situation where two different values are assigned to the same reference material, which is unacceptable.

3. Validated techniques and reference materials for measurement of thin films

Measurement of the thermal properties (e.g. thermal diffusivity) of thin films (Angstroms to micrometres thick) is difficult and lacks a standard. Is the lack of standards a problem?

Many methods have been proposed and used in several informal intercomparisons, e.g. on CVD diamond, silicon carbide and aluminium nitride thin films. There was a recent VAMAS study on very thin silicon oxide films and significant discrepancies occurred between the various measurement methods. The 3-omega method was the most consistent but it is not suitable for all materials.

END OF DOCUMENT