

INTRODUCTION TO THE WORK OF THE ICTAC COMMITTEE ON STANDARDISATION

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The work of the ICTAC Committee on Standardisation is reviewed and current Task Group activities are summarised.

Keyword: standardisation

Introduction

The Committee on Standardisation of the International Confederation for Thermal Analysis and Calorimetry (ICTAC) was formed in 1965. The objectives of the Committee were defined as [1]:

- a) to provide a common basis for relating independently acquired data,
- b) to provide the means for comparing and calibrating all available instrumentation,
- c) to provide the means for relating thermoanalytical data to physical and chemical properties defined by conventional isothermal means, and together with the ICTAC Nomenclature Committee:
- d) to define good practice, both in experimentation and in reporting, so that the information obtained and communicated is of maximum value,
- e) to promote ease of communication through uniform nomenclature and data presentation.

ICTAC Certified Reference Materials

The Committee on Standardisation realised its first three objectives by the development of a wide range of Certified Reference Materials (CRMs) for the temperature calibration of thermal analysis equipment. The CRMs which were primarily developed for use as inter-laboratory comparison materials are listed in Table 1, together with the transition type and temperature. The certified values were developed from extensive international round-robin programmes.

The CRMs consist of materials for the calibration of DTA and DSC equipment in the low-temperature range [2] and the range 130° to 930°C [3], based on solid–solid or fusion transitions. In addition, polystyrene was included to provide a material with a well characterised glass transition [4].

Table 1 ICTAC Certified Reference Materials

Ref. No.	Material	Transition	Temperature/°C
RM754	Polystyrene	Glass	105
RM757	1,2-Dichloroethane	Fusion	–32
	Cyclohexane	Solid–solid	–83
		Fusion	7
	Phenyl ether	Fusion	30
	<i>o</i> -Terphenyl	Fusion	58
RM758	Potassium nitrate	Solid–solid	128
	Indium	Fusion	157
	Tin	Fusion	232
	Potassium perchlorate	Solid–solid	300
	Silver sulphate	Solid–solid	430
RM759	Potassium perchlorate	Solid–solid	300
	Silver sulphate	Solid–solid	430
	Quartz	Solid–solid	573
	Potassium sulphate	Solid–solid	583
	Potassium chromate	Solid–solid	665
RM760	Quartz	Solid–solid	573
	Potassium sulphate	Solid–solid	583
	Potassium chromate	Solid–solid	665
	Barium carbonate	Solid–solid	810
	Strontium carbonate	Solid–solid	925
RM761	Permanorm 3	Magnetic	259
	Nickel	Magnetic	353
	Mumetal	Magnetic	381
	Permanorm 5	Magnetic	454
	Trafoperm	Magnetic	750

Table 2 Task Groups of the ICTAC Committee on Standardisation

Task Group	Task Group Leader
Materials for DSC and DTA, sub-ambient to 450°C	Dr. M. J. Richardson, National Physical Laboratory, UK
Materials for DSC and DTA, 450°–1100°C	Prof. E. L. Charsley, Leeds Metropolitan University, UK
Magnetic Materials for Thermogravimetry	Prof. P. K. Gallagher, Ohio State University, USA
Recommendation for Reporting Thermal Analysis Data	Prof. J. G. Dunn, Curtin University of Technology, Australia
Literature Review Group – Standard Materials	Prof. V. Balek, Nuclear Research Institute, Czechoslovakia

A set of magnetic reference materials was also developed for the temperature calibration of thermobalances [5].

The materials are distributed by the U.S. National Institute for Standards and Technology (NIST) and may be ordered from the Office of Standard Reference Materials, National Institute for Standards and Technology, Gaithersburg, MD 20899, U.S.A. Fax: 301-948-3730.

Recommendations for reporting thermal analysis data

Recommendations for reporting thermal analysis data were published for differential thermal analysis and thermogravimetry [6], evolved gas analysis [7] and thermomechanical analysis [8]. These recommendations have been widely adopted and have been published in a number of languages.

Current work programmes

It became increasingly clear from discussions within the Standardisation Committee and with a range of users of the CRMs that there was a major requirement for CRMs for direct temperature calibration of thermal analysis instrumentation. At present this is not possible due to the magnitude of the error ranges on the specified temperatures for the materials. These errors derive from the large round-robin programmes used to determine values for the material, where rigorous calibration procedures could not be carried out by a number of the participating laboratories.

It was therefore decided in 1991 to establish a series of Task Groups to work on the CRMs with the following objectives:

- a) to investigate the suitability of the present CRMs for accurate temperature calibration purposes using small round-robin programmes and to evaluate their potential for enthalpy calibration,
- b) to evaluate additional materials to supplement the current CRMs and to replace individual materials where necessary,
- c) to arrange for accurate values to be measured for the selected materials by calorimetric techniques.

In addition to the Task Groups on reference materials, two additional Task Groups were formed. The first is concerned with a revision of the 'Recommendations' for reporting thermal analysis data and with the implications of modern data processing methods on the presentation of thermal analysis data. The second Group is undertaking a review of the thermal data available in the literature for the ICTAC materials.

The Task Groups and the Task Group Leaders are given in Table 2. The results of the initial work programmes of these Groups are reported in these Proceedings.

A Task Group for Calorimetric Certification of Standard Materials has been established in conjunction with the ICTAC Committee on Calorimetry. The purpose of this Group will be to determine accurate values for temperatures and enthalpies of transition of selected materials, using calorimetric techniques. In addition a new Task Group for DTA/DSC materials with transitions above 1100°C has just been established under the leadership of Prof. C. M. Earnest (USA).

Other areas under consideration include: thermomechanical analysis and dynamic thermomechanical analysis; dilatometry; and thermomicroscopy.

National Group Activities

The Committee recognises the very valuable standardisation work that is going on at national level in a number of countries. It is anxious to encourage and assist this work and would not wish to duplicate any programmes that are being carried out nationally.

Active liaison is being maintained with a number of national groups and the work of the following organisations is presented in these Proceedings: Committee E-37 on Thermal Measurements of the ASTM; German Thermal Methods Group (GEFTA); U.K. Laboratory of the Government Chemist.

Committee membership

The Past-Chairmen of the Committee on Standardisation are:

- 1965–1974 Dr. H. G. McAdie (Canada)
- 1974–1985 Prof. P. D. Garn (U.S.A.)
- 1985–1988 Prof. P. K. Gallagher (U.S.A.)
- 1988–1992 Prof. E. L. Charsley (U.K.)

The current Committee membership is given below.

Chairman: Dr. M. J. Richardson (UK), Treasurer: Prof. P. K. Gallagher (USA)
Secretary: Prof. J. G. Dunn (Australia)

Members: Prof. V. Balek (Czechoslovakia), Prof. E. L. Charsley (UK), Dr. J. E. Callanan (USA), Mrs J. S. Colbert (USA), Prof. G. Della Gatta (Italy), Prof. C. M. Earnest (USA), Prof. E. Gmelin (Germany), Dr. W. Hemminger (Germany), Dr. R. G. Ferrillo (USA), Dr. H. G. McAdie (Canada), Dr. O. Toft Sorensen (Denmark), Dr. M. Todoki (Japan)

Ex-officio: Dr. J. Rouquerol (France), Chairman ICTAC Scientific Commission

The Chairman of the Committee on Standardisation would be very pleased to hear from colleagues who would like to become involved in the work of the Committee. His address is:

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References

- 1 H. G. McAdie, *Thermochim. Acta*, 1 (1970) 325.
- 2 P. D. Garn and O. Menis, *Thermochim. Acta*, 42 (1980) 125.
- 3 H. G. McAdie, in 'Thermal Analysis', Vol. 1, Ed. H. G. Wiedemann, Birkhauser Verlag, Basel 1972, p. 591.
- 4 P. D. Garn, O. Menis, *J. Macromol. Sci., Phys.*, B13(4) (1977) 611.
- 5 P. D. Garn, O. Menis and H. G. Wiedemann, *J. Thermal Anal.*, 20 (1981) 185.
- 6 H. G. McAdie, *Anal. Chem.*, 39 (1967) 543.
- 7 H. G. McAdie, *Anal. Chem.*, 44 (1972) 640.
- 8 H. G. McAdie, *Anal. Chem.*, 46 (1974) 1146

Zusammenfassung — Es wird ein Überblick über die Tätigkeit des ICTAC Committee on Standardisation gegeben und die Aktivitäten der gegenwärtigen Arbeitsgruppen zusammengefaßt.