

## **THERMAL STANDARDS FROM THE LABORATORY OF THE GOVERNMENT CHEMIST**

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The Laboratory of the Government Chemist (LGC) is a focal point for the production, analysis and certification of reference materials. Within the field of thermal analysis the LGC is concerned with the development of purity standards and materials certified for enthalpy of fusion and melting point. For some time the LGC has been concerned with the significant differences in purity data which can be produced by the different manufacturers' differential scanning calorimeters. This paper will highlight the initiatives the LGC is undertaking in overcoming this uncertainty in purity measurements through the use of certified thermal standards.

**Keywords:** purity standards, reference materials, thermal standards

### **Introduction**

The development of reference materials and standards has been carried out for over twenty years at the Teddington site, first within the National Physical Laboratory (NPL) and, since 1988, at the Laboratory of the Government Chemist (LGC).

In response to new industrial, commercial and social needs there has been a significant increase in the use of Certified Reference Materials (CRMs). There are now more than one hundred producers in the western world alone. This large increase in the number of reference materials being produced has led to a growing need for coordination of all CRM activities. To this end, ISO-REMCO, the Council Committee on Reference Materials of the International Organisation for Standardisation, was set up in 1976 and has since published several guidelines [1-4] including the formal definition of Certified Reference Materials.

It has been estimated that up to 10% of the Gross National Product of industrialised countries may be associated with costs of measurement. However a study in the USA estimated that one test in every ten has to be repeated. As such the costs involved with obtaining the wrong answers are enormous. Achieving the necessary accuracy when undertaking analytical measurements is therefore not only important in terms of the quality of industrial goods, R&D, the health, safety

and welfare of the community, and the protection of the environment, but also in terms of the economic well being of the nation.

### Thermal standards

There is increasing evidence that analytical results obtained in one laboratory may not always be comparable with those obtained in another. This is exemplified by the significant differences in purity data, which can be produced on the same material by different manufacturers' differential scanning calorimeters (Fig. 1). This is a major source of concern since both the pharmaceutical and agrochemical industries use the technique extensively for routine assessment of raw and finished products.

The LGC intends to address this problem in purity measurements by developing a set of purity, enthalpy of fusion and melting point reference materials for the calibration of differential scanning calorimeters. This will be carried out in collaboration with manufacturers and users of thermal analysis equipment. It will form part of the LGC's Valid Analytical Measurement initiative (VAM) – which is a long term commitment to improving the quality of analytical measurements.

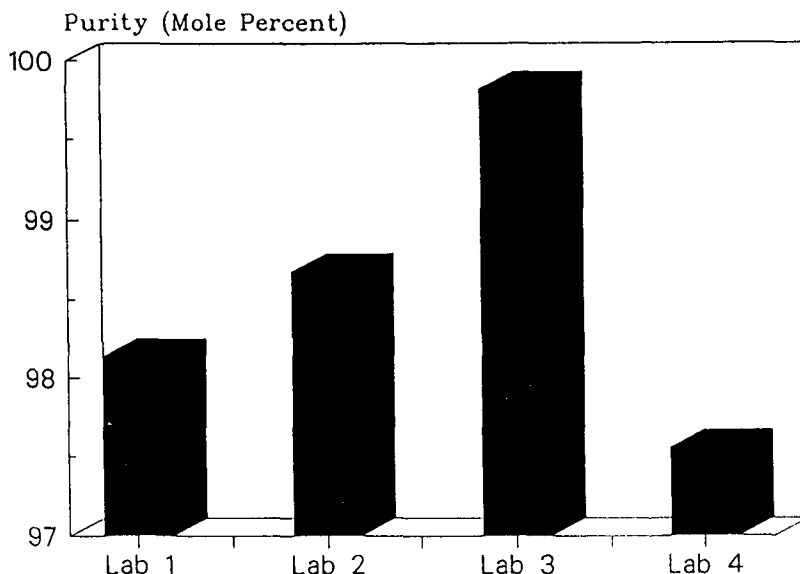


Fig. 1 Purity survey results

Table 1 shows reference materials certified for enthalpy of fusion and melting point respectively, which are available from LGC's Office of Reference Materials.

**Table 1** Data on certified reference materials available from the Laboratory of the Government Chemist

Material	Melting point /°C	Enthalpy of fusion / Jg <sup>-1</sup>
4-Nitrotoluene	52	-
Naphthalene	81	149
Benzil	95	112
Acetanilide	115	160
Benzoic acid	123	148
Diphenylacetic acid	147	147
Indium	157	29
Anisic acid	184	-
2-Chloroanthraquinone	210	-
Carbazole	246	-
Anthraquinone	285	-
Alumina	2052	-

Samples of organic compounds to be used for reference purposes must be of high purity. Commercial materials are rarely adequate for these purposes and a purification programme is therefore required. Several methods including re-crystallisation, vacuum sublimation, fractional-freezing, and zone-refining are used. The method employed depends on the physical and chemical properties of the individual substance. For example, zone-refining may be used for purifying thermally stable solid CRM's. Successful application of this technique pre-supposes that the sample is thermally-stable at temperatures a few kelvins above the melting point. If this is not so, vacuum-sublimation or preparative liquid chromatography may be applicable.

Purity determinations are usually made by gas-liquid chromatography, high-performance liquid chromatography or differential scanning calorimetry. If possible, each purified sample is assayed by two different methods, at least one of which is different in character from the purification method. Adiabatic calorimetry [5, 6] is the preferred technique for characterisation of enthalpy of fusion values because the sample can be melted very slowly, thus ensuring the establishment of thermodynamic equilibrium phases.

## Conclusion

Compatibility of analytical measurements used by the many laboratories engaged in determination of chemical, physical and technological properties on a wide range of materials is often best achieved by the use of reference materials. When the property values assigned to the reference materials are certified by a

national standards laboratory such as the LGC, the Certified Reference Materials ensure not only compatibility between the measurements made in the different laboratories, but also their traceability to a national standard.

## References

- 1 ISO Guide 30 1981 (E)
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- 3 ISO Guide 33 1989 (E)
- 4 ISO Guide 35 1989 (E)
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**Zusammenfassung** — Das Labor von Government Chemist (LGC) ist ein Mittelpunkt der Herstellung, Analyse und Zertifizierung von Referenzsubstanzen. Innerhalb des Bereiches Thermoanalyse beschäftigt sich LGC mit der Entwicklung von Reinheits-Standards und anderen, für Schmelzenthalpie und Schmelzpunkt zertifizierte Substanzen. Seit einiger Zeit befaßt sich LGC mit signifikanten Abweichungen bei Reinheitsangaben, die sich zwischen den DSC-Geräten verschiedener Hersteller ergeben. Vorliegende Arbeit zeigt die Aktivitäten von LGC zur Überwindung dieser Unsicherheiten von Reinheitsmessungen unter Zuhilfenahme zertifizierter thermischer Standards.