CERTIFICATE OF ANALYSIS

ERM®- FC024a

Diphenylacetic acid		
Measurand	Certified value [1,2] (°C)	Uncertainty [3] (°C)
Liquefaction Point	147.26	0.31

1) Value traceable to the International Temperature Scale of 1990 (ITS-90).

2) The liquefaction point is the lowest temperature at which the sample is completely liquid.

3) The uncertainty quoted is the half-width of the expanded uncertainty interval, calculated using a coverage factor (*k*) of 2, which provides a level of confidence of approximately 95 %.

This certificate is valid for 12 months from the date of shipment provided the sample is stored under the recommended conditions.

The minimum amount of sample to be used is 5 mg.

NOTE

European Reference Material ERM®-FC024a was produced and certified under the responsibility of LGC according to the principles laid down in the Technical Guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM.

Accepted as an ERM®, Teddington, March 2012.
Certificate amended July 2013.

Signed: _____

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DESCRIPTION OF THE MATERIAL

A batch of diphenylacetic acid was obtained from a commercial source, ground to pass through a 710 μm sieve, mixed and dried. 0.25 g units were packaged in 4 mL amber glass vials and sealed with screw-top caps fitted with PTFE/silicone septa. The purity of the material was checked using Differential Scanning Calorimetry (DSC) and found to be 99.96 mol %.

INTENDED USE

This material is intended for use in calibrating and checking apparatus used for determining melting points of samples in glass capillary tubes.

ANALYTICAL METHOD USED FOR CHARACTERISATION

This material has been characterised using measurements made by LGC [1]. Temperatures were measured using a platinum resistance thermometer (PRT) (100 Ω, Isotech) and a thermocouple (Type N, ceramic coated, Isotech) connected to a precision multimeter (TTI-7, Isotech). Two PRTs were calibrated by the National Physical Laboratory, Teddington, UK in terms of ITS-90 and using the International Electrotechnical Commission standard for PRTs (IEC 751) [2] for the range 0 °C to 250 °C.

Approximately 1.5 g of dried sample was packed in a clean dry glass tube. A thermocouple was inserted into the sample tube ensuring that it was in good contact with the sample. The tube was placed in an aluminium sample block in an oil bath (model 798 EHT, Isotech) containing silicone oil. The oil bath temperature was held approximately 7 °C below the sample melting point before applying a temperature ramp of 0.2 °C/min to approximately 7 °C above the sample melting point where it was held for a further period. The sample temperature, block temperature, and oil bath temperatures were monitored. The liquefaction point of the sample was determined using a differential approach as the temperature of the sample (measured using the thermocouple calibrated within each run against the PRT) at the end of the plateau region in the melting curve; specifically, the first point in the melting curve after the onset of liquefaction at which the sample temperature gradient exceeded 3.2 mK/s.

In July 2013 the certificate was amended to improve the clarity of table note 2 on page 1 and the instructions for use. The homogeneity paragraph was changed to correct the description from thermodynamic mode to pharmacopoeia mode. A third reference was added.

HOMOGENEITY

The homogeneity of the material was confirmed by analysis of twelve randomly selected units from across a fill run of 100 units, using a Büchi melting point apparatus B-545 (Flawil, CH-9230) in the pharmacopoeia mode at 0.2 °C/min [3]. The capillary tubes were filled to a depth of 6 mm of material. An uncertainty was calculated to allow for possible inhomogeneity and included in the uncertainty of the certified value.

STABILITY

A contribution to the combined uncertainty was calculated using data obtained over an 11 year period to represent any possible long-term instability of the material. This was included in the calculation of the uncertainty of the certified value.

SAFETY INFORMATION

Refer to the Safety Data Sheet.

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INSTRUCTIONS FOR USE

The minimum quantity of sample to be used is 5 mg, which for a capillary instrument corresponds to the amount required to provide a depth of approximately 6 mm in a glass capillary tube.

A sub sample should be taken from the screw-cap vial supplied, lightly crushed in an agate mortar to produce a fine powder and dried, for example by storage for 48 hours under reduced pressure in a desiccator containing a suitable desiccant. After opening the vial, precautions should be taken to guard against contamination of the material.

The certified value for the liquefaction point given on page 1 of this certificate was obtained using a heating rate of ~0.2 °C/min with the temperature probe in direct contact with the sample. This provides an accurate thermodynamic melting point within the stated uncertainty. However, this configuration is rare in commercial melting point instruments. Considerable caution should therefore be exercised in comparing the certified value with values obtained from commercial instruments, because the time of response of the temperature sensor to the changing sample temperature may differ in different systems depending on the mode selected. It is recommended to calibrate and check the instrument at the same heating rate as used for test materials. If another heating rate or mode is selected the observed values are likely to differ as follows:

- The pharmacopoeia mode for a capillary instrument measures the temperature of the oven, whilst in the thermodynamic mode the temperature reading indicates the temperature of the sample itself, resulting in an apparent temperature difference owing to temperature lag between the sample and the oven. We have found that in pharmacopoeia mode corrections of approximately 0.5 °C are necessary, even at a heating rate of 0.2 °C/min. The correction differs from one instrument to another. It is therefore essential to calibrate the instrument and include any necessary correction according to the manufacturer's instructions when comparing the certified value with the temperature in pharmacopoeia mode.
- In addition if a heating rate other than 0.2 °C/min is used in a capillary instrument set in pharmacopoeia mode and calibrated at 0.2 °C/min, the user should observe a melting temperature that differs from the certified value. The melting temperature can be expected to increase by 1 to 1.5 °C per °C/min increase in the heating rate selected, in the heating rate range 0.2 to 1.0 °C/min. For example a heating rate of 0.5 °C/min would generate apparent melting temperatures of 0.5 - 0.75 °C higher than the certified value.

STORAGE

This certified reference material should be stored at (20 ± 5) °C, in the original container, under dark, dry conditions.

REFERENCES

1. T. Le Goff, B. Joseph and S. Wood, *Journal of Thermal Analysis and Calorimetry*, **96(2)**, 653-662, 2009
2. N. P. Moiseeva, *Measurement Techniques*, **44(5)**, 502-507, 2001
3. S.L.R. Ellison, N. Grimes, F. Fardus, M.P.L. Ellison, Accreditation and Quality Assurance, **17(3),** 283-2900, 2012

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Unit Number:	Shipment Date:

LEGAL NOTICE

The values quoted in this certificate are the best estimate of the true values within the stated uncertainties and based on the techniques described herein. No warranty or representation, express or implied, is made that the use of the product or any information, material, apparatus, method or process which is the subject of or referred to in this certificate does not infringe any third party rights. Further, save to the extent: (a) prohibited by law; or (b) caused by a party's negligence; no party shall be liable for the use made of the product, any information, material, apparatus, method or process which is the subject of or referred to in this certificate. In no event shall the liability of any party exceed whichever is the lower of: (i) the value of the product; or (ii) £500,000; and any liability for loss of profit, loss of business or revenue, loss of anticipated savings, depletion of goodwill, any third-party claims or any indirect or consequential loss or damage in connection herewith is expressly excluded.

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