

# Certificate of measurement

ISO 9001 Quality  
Management certified by BSI  
under certificate number  
FS27613

## Phenyl Salicylate

Certified Reference Material LGC2613

### Certified values

Property	Certified value	Uncertainty
Enthalpy of fusion (kJ/mol) <sup>1</sup>	19.18	0.08 <sup>2</sup>
Melting temperature (°C) <sup>3</sup>	41.79 <sup>4</sup>	0.03 <sup>5</sup>

#### Notes:

1. The molar mass of phenyl salicylate has been taken as 214.21 g mol<sup>-1</sup>.
2. The uncertainty quoted is the half-width of the expanded uncertainty interval, calculated using a coverage factor of 2.776, which gives a level of confidence of approximately 95 %.
3. The certified melting temperature corresponds to the final melting temperature i.e. complete liquefaction.
4. The melting temperature was obtained by making a correction of + 0.02 °C to allow for the difference between the triple point pressure and atmospheric pressure.
5. The uncertainty quoted is the half-width of the expanded uncertainty interval calculated using a coverage factor of 2, which gives a level of confidence of approximately 95 %.

Date of Issue: March 1998  
Latest revision: March 2009

Signed: \_\_\_\_\_  
Gill Holcombe (Mrs)  
for the Government Chemist



## Material Preparation

A commercial supply of phenyl salicylate was obtained and purified by fractional crystallisation. The purified material was ground to pass a 710  $\mu\text{m}$  sieve, dried under vacuum and then dispensed as 0.5 g portions into amber glass vials. Using adiabatic calorimetry, the measured mole fraction was 99.994 per cent.

## Homogeneity Assessment

As the material is a single substance of high purity, it is considered to be sufficiently homogeneous for its intended use. A formal experimental demonstration of homogeneity was considered to be unnecessary.

## Stability Assessment

The nature of the material is such that deterioration is not anticipated. However, this material will be subjected to routine stability testing under LGC's 'reference material stability testing' programme. Purchasers will be informed of any changes that affect the certified values.

## Certification

This material has been certified by LGC in collaboration with the University of Oslo, Norway using adiabatic calorimetry to determine the enthalpy of fusion and melting temperature. The general methodology is described in references 2 and 3.

## Analytical Methods Used

Approximately 60 g of sample was placed in a sealed fused silica container which was evacuated to about  $1 \times 10^{-2}$  Pa. The container was fitted into the calorimeter and a well in the silica container accommodated a heater and platinum resistance thermometer. Zero temperature difference was maintained between the electrically heated adiabatic shield surrounding the calorimeter and the calorimeter during the measurement.

For each measurement, electrical energy was introduced into the calorimeter in a stepwise fashion. Each step was typically of 100 minutes duration. Sufficient steps were applied to ensure complete melting of the entire sample. Each measuring cycle started with the establishment of 'zero' temperature drift of the calorimeter, usually over a 40 minute period. The cycle started at a temperature a few degrees below the melting (fusion) temperature and continued for a few degrees above the melting temperature.

The energy required to bring about fusion of the sample was derived from measurements of electrical potential across the calorimeter heater, the applied current and time. The electrical potential and applied current was measured with a multimeter calibration on the SI 1990 volt scale. Calibration of the platinum resistance thermometer was carried out according to recommendation for the International Temperature Scale 1990 (ITS-90)<sup>1</sup>.

In March 2009, a minor change was made to the wording of the Stability Assessment section.

## Intended Use

The material is intended for the calibration of differential scanning calorimeters and similar instruments.

## Instructions for Use

After opening the vial, precautions should be taken to guard against contamination of the material.

## Storage Conditions and Shelf Life

The reference material should be stored in the unopened vial at ambient temperature ( $20\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ ) in a desiccator, until it is required for use. If stored under the recommended conditions, the certified values of the unopened material will remain valid for 36 months from the date of shipment.

## Guidance on the Interpretation of Results

When comparing an analytical result obtained on the certified reference material to the appropriate certified value, users should not necessarily expect the result always to fall within the uncertainty limits of the certified value quoted on page 1. The uncertainty of the analytical result ( $U_{\text{result}}$ ) must also be taken into account and if the uncertainty of the certified value is  $U_{\text{RM}}$ , then, as a simple test, a result should normally lie within the following limits of the certified value:

$$\pm \sqrt{U_{\text{RM}}^2 + U_{\text{result}}^2} \dots\dots\dots(1)$$

Both  $U_{\text{RM}}$  and  $U_{\text{result}}$  are expanded uncertainties providing a level of confidence of approximately 95 %. If a result lies outside these limits a problem with the measurement procedure and/or the uncertainty estimate of the result is indicated, which should be investigated.

If an estimate of the uncertainty of the result is not available, an approach recommended<sup>4</sup> by ISO Guide 33 is to obtain a value for the expected precision of the method ( $\sigma$ ). The precision has two components, the within-laboratory standard deviation ( $s_w$ ) and the between-laboratory standard deviation ( $\sigma_{\text{Lm}}$ ). The precision ( $\sigma$ ) is given by:

$$\sigma = \sqrt{\sigma_{\text{Lm}}^2 + \frac{s_w^2}{n}} \dots\dots\dots(2)$$

The within-laboratory standard deviation ( $s_w$ ) is that observed when  $n$  replicate measurements are carried out under repeatability conditions (i.e. same analyst, same equipment, same reagents, and same calibration, over the shortest practical time period). The between-laboratory standard deviation ( $\sigma_{\text{Lm}}$ ) cannot be estimated by a single laboratory, but in many cases it is sufficient<sup>4</sup> to substitute the standard deviation observed in a single laboratory under intermediate precision conditions. The latter may be taken to refer to the situation where a series of measurements (10 or more) have been carried out over an extended period of time (1 month or more), preferably involving different analysts, equipment, reagents, calibrations, etc.

Once a value for  $\sigma$  has been obtained, a simple test for an analytical result is that it should normally lie within the following limits of the certified value:

$$\pm \sqrt{U_{\text{RM}}^2 + (2\sigma)^2} \dots\dots\dots(3)$$

If a result lies outside these limits a problem with the measurement procedure and/or the estimate of  $\sigma$  is indicated, which should be investigated.

It will be realised that the value  $2\sigma$  is a substitute estimate of the expanded measurement uncertainty,  $U_{\text{result}}$ . In either case, a larger value for  $2\sigma$  or  $U_{\text{result}}$  will make it more likely that an analytical result will lie within the limits calculated according to equations 1 or 3. The analyst must decide whether the adopted value for  $2\sigma$  or  $U_{\text{result}}$  is consistent with their measurement results being fit for the particular purpose for which they are intended.

## References

1. The International Temperature Scale of 1990, HMSO, 18 (1991)
2. Grønvold, F., *Acta Chem.Scand.* 21,1695 (1967)
3. Grønvold, F.J., *Journal Chem. Thermodynamics* 25, 1133 (1993)
4. ISO Guide 33:2000. Uses of Certified Reference Materials.

Unit Number:

Date of Shipment:

## Legal Notice

(revised January 2009)

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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