

Certificate

NBS-ICTA Standard Reference Material 758

DTA Temperature Standards (125 – 435 °C)

These standards include: high-purity potassium nitrate, indium, tin, commercial grade potassium perchlorate, and analyzed reagent silver sulfate. They are for use in calibrating the temperature scale on differential thermal analysis and related thermoanalytical equipment under the operating conditions. They are to be used only in the heating mode.

Transition Temperature Data (°C)

Material	Equilibrium Value [1]	DTA Mean Values	
		Extrapolated Onset	Peak
KNO ₃	127.7	128	135
In (metal)	157	154	159
Sn (metal)	231.9	230	237
KClO ₄	299.5	299	309
Ag ₂ SO ₄	-(a)	424	433

(a) The value for the transition temperature reported for this material [1] is currently under review. A more recent value of 430 °C has been reported [2].

For potassium nitrate only, the first heating of the material is not a reliable measure of the transition temperature. This sample must be cycled through the transition temperature in situ before recording data.

The extrapolated onset temperature is defined (Point A, Figure 1) as that temperature found by extrapolating the base line, prior to the peak, and the leading side of the peak to their intersection. The peak temperature (Point B, Figure 1) is defined as the temperature corresponding to the point of maximum deflection of the differential temperature curve.

Based on the mean value from 34 cooperating laboratories, the standard deviation was 5.8 °C, which includes the bias among laboratories as well as measurement errors. In the heating mode, onset and peak temperatures have about equal precision. However, the mean extrapolated onset temperatures are closer to the equilibrium transition temperatures.

The materials for these standards were furnished by the Committee on Standardization of the ICTA.

The International Test Program leading to the data on which certification is based was coordinated by H. G. McAdie, Chairman, ICTA Committee on Standardization.

Coordination of efforts leading to certification by NBS was under the chairmanship of O. Menis.

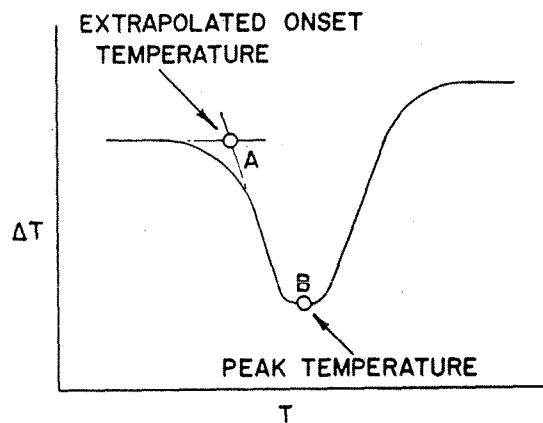
The technical and support aspects involved in the preparation, certification, and issuance of these Standard Reference Materials were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
June 11, 1971

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The transition temperatures given are the means of values obtained on 28 different types of commercial or custom-made instruments, and do not necessarily agree with the equilibrium values [1, 2]. Generally, values from DTA are somewhat higher than the equilibrium values and vary in a complex manner for different instruments and heating rates. Details of the instruments used, the test conditions and complete data will be given in an NBS Special Publication in the 260 series (in publication).



- [1] Nat. Bur. Stand. (U.S.), Circ. 500 (1952).
- [2] Hedvall, J. A., Lindner, R., and Hartler, N., Acta. Chem. Scand. 4, 1099 (1950).

Cooperating Laboratories

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