U. S. Department of Commerce Maurice H. Stans Secretary National Bureau of Standards L. M. Branscond, Director

Certificate

STANDARD REFERENCE MATERIAL 756 Potassium Nitrate

This standard is specially prepared high-purity potassium nitrate. It is intended for use as a material for checking the performance of differential thermal analysis equipment.

Effective Phase Transition Temperature

| Intersection | 130°C | 1 |
|--------------|--------|---|
| Peak | 131 °C | , |

Because the first heating of the material is not a reliable measure of the transition temperature, the sample must be cycled through the transition temperature in situ before recording data. Tests of the homogeneity of the material under nearly adiabatic conditions (1 to 2 °C per minute) provided values which are within a range of \pm 0.5 °C of the effective transition temperature.

The intersection temperature is defined as that temperature corresponding to the intercept of the extrapolated base line with the extrapolated approximate straight line of the starting side of the peak on the differential temperature curve. The peak temperature is defined as the temperature corresponding to the point of maximum inflection of the differential temperature curve peak. The thermocouples used to the measurements were prepared from platinum and platinum- 10 percent rhodium wire and calibrated by the NBS Heat Division to the International Practical Temperature Scale.

The transition temperatures given are the means of the values obtained on several differential thermal analysis instruments, and do not necessarily agree with the thermodynamic value of 127.7 °C given in NBS Circular 500, "Selected Values of Chemical Thermodynamic Properties" (1952). Generally, the differential temperature curve value is somewhat higher than the adiabatic value and will vary in a complex manner for different equipment and heating rates. Details of the equipment used and homogeneity testing of this material are given in NBS Technical Note 504 (Superintendent of Documents, U. S. Government Printing Office, Washington, D. C. 20402, Price \$1).

The following conditions were used to determine the transition temperatures of this reference standard, although the conditions may vary somewhat within the limits with a particular instrument.

Sample condition Because the first heating of the sample is not a reliable measure of the transition temperatures, the

sample must be cycled once through the transition

recording data (135 °C).

Sample size 3 to 300 mg.

Sample container Graphite or platinum cylindrical crucible, or alumina

cylindrical sample block.

Heating rates 1 °C per min to 10 °C per min.

Reference material Alumina (Al_2O_3) .

Atmosphere Static air or dry nitrogen.

Thermocouple Located on the axis of the material with the

measuring point at, or near, the midpoint of the axis.

The Committee of Standardization, H. G. McAdie, Chairman, of the International Conference on Thermal Analysis, recommended the use of potassium nitrate as a differential thermal analysis standard reference material. Cooperative work and values were furnished by P. D. Garn, Department of Chemistry, University of Akron, Akron, Ohio; and by H. G. McAdie, Ontario Research Foundation, Sheridan Park, Ontario, Canada.

Determinations were made at the National Bureau of Standards by J. T. Sterling, E. R. Deardorff, D. S. Bright, and T. A. Rush of the Analytical Chemistry Division.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of O. Menis.

The technical and support aspects involved in the preparation certification, and issuance of this standard were coordinated through the Office of Standard Reference Materials by J. L. Hague.