U. S. Department of Commerce Malcolm Baldrige Secretary National Bureau of Standards Ernest Ambler, Director

National Bureau of Standards Certificate

Standard Reference Material 2151

Nicotinic Acid

Combustion Calorimetric Standard

This Standard Reference Material (SRM) consists of twenty-five grams of commercial high-purity nicotinic acid. SRM 2151 is intended for checking the suitability of apparatus, analytical procedures, and calculations involved in oxygen-bomb calorimetry of substances containing up to 15 weight-percent of nitrogen.

The certified value for the quantity of heat evolved by combustion of a sample of this material in an oxygen-bomb calorimeter is:

 $22.184 \pm 0.005 \text{ MJ/kg}.$

The certified value is for the following reaction at 25 °C:

 $C_6H_5O_2N(c) + 6.25 O_2(g) \Rightarrow 6CO_2(g) + 0.5N_2(g) + 2.5 H_2O(liq).$

Bomb Conditions:

- (1) The SRM must be crushed to a powder and dried for 24 hours at 85 °C. It may be stored at room temperature in a closed container. The dried material must be pressed into a pellet before placing in the bomb.
- (2) The mass of sample, in grams, is equal to 3.5 times the volume of the bomb, in liters. When the mass of sample exceeds 4 times the volume of the bomb, in liters, the combustion reaction is usually not complete.
- (3) The mass of water in grams, initially placed in the bomb, is equal to 30 times the volume of the bomb in liters.
- (4) The sample, in pellet form, is burned in a bomb of constant volume, filled with pure oxygen at a pressure of 30 atmospheres measured at 25 °C. No auxiliary material shall be used.

Recommended Procedures:

- (1) The ignition energy must be determined in a separate experiment.
- (2) The quantity of nitric acid produced in the combustion is determined by titration with standard alkali. The energy of decomposition of nitric acid into nitrogen (gas) and water (liquid) was taken as 59 kJ/mol.
- (3) The energy evolved by the actual bomb process, ΔU_B, must be corrected to that for the ideal process, ΔU_C, by the method of Prosen [1], Hubbard, Scott, and Waddington [2], or Mansson and Hubbard [3]. For further information regarding procedures in the calibration of combustion bomb calorimeters, the discussions by Coops, Jessup, and Van Nes [4] and Mosselman and Churney [5] should be consulted.

The measurements were performed and analyzed in the Thermochemical Measurements and Standards Section by W.H. Johnson, under the technical direction of G.T. Armstrong.

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

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Stanley D. Rasberry, Chief Office of Standard Reference Materials

References

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- [2] Hubbard, W.N., Scott, D.W., and Waddington, G., J. Phys. Chem. 58, 152 (1954).
- [3] Mansson, M., and Hubbard, W.N., Chapter 5, Combustion Calorimetry, S. Sunner and M. Mansson, Editors, (Pergamon Press, New York, 1979).
- [4] Coops, J., Jessup, R.S., and Van Nes, K., Chapter 3, Experimental Thermochemistry, F.D. Rossini, Editor, (Interscience Publishers, Inc., New York, 1956).
- [5] Mosselman, C., and Churney, K.L., Chapter 3, Combustion Calorimetry, S. Sunner and M. Mansson, Editors, (Pergamon Press, New York, 1979).