



Certificate of Analysis

Standard Reference Material 141b

Acetanilide

This standard was purified and prepared at the National Bureau of Standards for use as a standard reference material in the microdetermination of carbon, hydrogen, and nitrogen. Evaluations based on measurements of the freezing temperature of acetanilide indicate a purity of 99.99 mole percent. Tests show that this material is not hygroscopic under ordinary conditions of storage and can be used without preliminary drying. It is recommended that the theoretical percentages be used, namely:

Carbon	-----	71.09
Hydrogen	-----	6.71
Nitrogen	-----	10.36

WASHINGTON, D. C. 20234
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W. WAYNE MEINKE, Chief,
Office of Standard Reference Materials.

(over)

Purification of Standard Reference Material 141b, Acetanilide

The starting material for this purification was approximately 12 kg of acetanilide acquired from the Fisher Scientific Company and the North-Strong Corporation. Purification was effected by fractional crystallization of the acetanilide from its melt. The purity was evaluated by means of freezing-temperature studies.

The purification was carried out in two stages. In the first stage, two batches of purchased acetanilide were crystallized from the melt. In one, 66 percent of the melt crystallized over a 24-hr period, and in the other, 60 percent crystallized in an equivalent period. The material used in the second-stage crystallization was a portion of the product prepared in the first-stage crystallization. In this stage, 80 percent of the acetanilide crystallized over a period of 40 hr. These crystals were carefully melted, poured into porcelain dishes and allowed to solidify. Samples were taken from each dish and combined for purity evaluation. The solid acetanilide was crushed, sieved to size through a 40-mesh onto a 100-mesh sieve, blended, and stored. The yield of purified acetanilide was 3700 g.

The freezing-temperature measurements of the composite samples were made in cells of the type described by Schwab and Wichers [Temperature, Its Measurement and Control in Science and Industry, Vol. 1, p. 265 (Reinhold Publishing Corporation, New York, N. Y. 1941)]. The mean value of several determinations was 114.289°C . This compares with the value of $114.29 \pm 0.01^{\circ}\text{C}$ reported Schwab and Wichers [J. Res. NBS 32, 253 (1944)] as the freezing point extrapolated to zero impurity. The purity of the acetanilide evaluated from this data was 99.99 mole percent. Several microanalyses were made in which the present material was compared with acetanilide previously issued as a standard reference material. The results of these analyses indicate no significant difference between the two samples within the precision of the measurements.

The purification and measurements of purity of the acetanilide were performed by Delmo Enagonio. The microanalyses were performed by Rolf A. Paulson and Robert J. Hall.