Certificate of Analysis

Standard Reference Material 2142

o-Bromobenzoic Acid

R. A. Paulson and W. P. Schmidt

This standard reference material is certified for use in the calibration and standardization of microchemical procedures for the determination of bromine in organic material.

Bromine . . . . . 39.80 ± 0.05 wt. percent

The uncertainty shown represents the 95 percent confidence interval of the mean based on 16 determinations and allows for the effects of known sources of possible error. Bromine was determined by both the micro-Carius method and the macro-sodium peroxide bomb method.

The o-bromobenzoic acid was prepared by C. L. Stanley of the Office of Standard Reference Materials. Analytical measurements to further characterize this material were performed by R. Schaffer, R. F. Brady, Jr., P. Douglas, A. Fatiadi, and E. E. Hughes.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J. K. Taylor.

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 T. W. Mears.

Washington, D. C. 20234
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J. Paul Cali, Acting Chief
Office of Standard Reference Materials

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Bromine was determined both by the micro-Carius method and by the macro-sodium peroxide fusion method. Six samples were received, and at least one determination by each method was made on each bottle. The material represented by these samples is considered to be homogeneous within the limit of experimental error.

Using the Carius method, accurately weighed 10 mg samples were digested in 0.5 ml fuming nitric acid and 15 mg of silver nitrate for 12 hrs at 250 °C. After cooling the tubes were opened and the contents diluted with water. Following a digestion period the silver bromide was filtered on a weighed micro-filter tube. The average value for 9 determinations was 39.81 ± 0.09 weight percent.

Using the sodium peroxide bomb method, 275 mg samples were mixed with 15 g of sodium peroxide-sugar mixture (14:1) in a Parr peroxide bomb and ignited. The resulting melt was washed from the bomb and the solution boiled to destroy peroxides. Hydrazine sulfate was added to reduce any bromate present. The solution was made acid with nitric acid and sufficient 0.1 N silver nitrate solution added to precipitate all of the bromide. The precipitate was filtered off and redissolved in concentrated ammonium hydroxide solution and reprecipitated by the addition of silver nitrate and nitric acid. This precipitate of silver bromide was collected on a weighed fritted-glass filter. The average value for 7 determinations was 39.80 ± 0.07 weight percent.

Carbon and hydrogen were determined on each sample using a commercial carbon-hydrogen-nitrogen analyzer. Carbon found was 41.84 wt. percent, hydrogen 2.57 wt. percent. The neutralization equivalent was found to be 99.6_3 percent of that calculated for o-bromobenzoic acid. Gas-liquid chromatography showed about 0.5 percent impurity having a shorter retention time than the major component. This may be p-bromobenzoic acid or o-bromobenzyl alcohol. Differential scanning calorimetry indicates a purity of about 99.6 mole percent. Mass spectrometric examination showed no detectable impurities.