U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards Certificate

Standard Reference Material 2144

m-Chlorobenzoic Acid

W. P. Schmidt

This Standard Reference Material is certified for use in the calibration and standardization of microchemical procedures for the determination of chlorine in organic material.

Chlorine ..... 22.62 ± 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. Chlorine was determined by both the micro-Carius method and the macro-sodium peroxide bomb method.

The m-chlorobenzoic acid is a highly purified commercial material. Analytical measurements to further characterize the material were performed by J. E. Fearn 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
April 5, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

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Chlorine 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 20 mg samples were digested in 0.5 ml fuming nitric acid and 15 mg of silver nitrate for 12 hours at 250 °C. After cooling the tubes were opened and the contents diluted with water. Following a digestion period the silver chloride was filtered on a weighed micro-filter tube. The average value for 8 determinations was 22.65 ± 0.03 weight percent.

Using the sodium peroxide bomb method, 300 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. The solution was made acid with nitric acid and sufficient 0.1 N silver nitrate solution added to precipitate all of the chloride. The precipitate of silver chloride was collected on a weighed fritted-glass filter. The average value for 8 determinations was 22.59 ± 0.01 weight percent.

Carbon and hydrogen were determined on each sample using a commercial carbon-hydrogen-nitrogen analyzer. Carbon found was 53.71 wt. percent, hydrogen 3.25 wt. percent. The neutralization equivalent was found to be 99.8 percent of that calculated for m-chlorobenzoic acid. Differential scanning calorimetry indicates a purity of about 99.7 mole percent. Mass spectrometric examination showed no detectable impurities.