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 2143

p-Fluorobenzoic Acid

R. A. Paulson

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

Fluorine ..... 13.54 ± 0.01 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. Fluorine was determined by titration of the hydrofluoric acid resulting from combustion of the material.

Fluorine was determined by titration of the hydrofluoric acid resulting from combustion of the material. A one-gram sample was combusted in a platinum-lined Parr oxygen bomb containing 20 ml of water. A weight aliquot of the solution containing the combustion products was titrated with 0.1N sodium hydroxide. In the titration, which was performed in a platinum crucible, approximately one-half of the required sodium hydroxide was added. The solution was then heated to boiling to remove carbon dioxide and cooled. The titration was continued under an atmosphere of air free of carbon dioxide to a pH of 8.6. In most cases duplicate aliquots were titrated. Corrections were applied for nitric acid formed and for any acid carried out when the oxygen pressure was released from the bomb.

The p-fluorobenzoic 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
January 23, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

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Homogeneity of the material was determined by duplicate acidimetric titrations of each of eight samples randomly selected from the bulk of the material. No inhomogeneity was found within the analytical uncertainty. These titrations indicated an overall purity of 99 .95 ± 0.02 weight percent.

Confirmatory analysis for fluorine was made by the specific but relatively imprecise lead chlorofluoride gravimetric method. Five determinations by this procedure indicated a purity of 100.1 ± 0.2 weight percent.

Differential scanning calorimetry indicated a purity of 99.9 percent. Mass spectrometric examination showed no detectable impurities.