U. S. Department of Commerce
John T. Connor, Secretary
National Bureau of Standards
A. V. Astin, Director

Replaced by final July 9, 1969

Certificate of Analysis

Standard Reference Material 143b

Cystine

This standard was purified at the National Bureau of Standards for use in checking microdeterminations of carbon, hydrogen, sulfur, and nitrogen. The absolute purity of the standard has not been established, but microanalytical determinations yielded values that check the theoretical composition within the limits of experimental error. It is recommended that the theoretical percentages be used, namely:

Carbon	29.99
Hydrogen	5.03
Sulfur	26.69
Nitrogen	11.66

PROVISIONAL

WASHINGTON, D. C. 20234
May 3, 1965.

W. Wayne Meinke, Chief,
Office of Standard Reference Materials.

(over)

Purification of Microchemical Standard Reference Material 143b

Cystine

The starting material for this purification was approximately 3 kg of cystine acquired by the Organic Chemistry Section from Distillation Products Industries, a division of Eastman Kodak Co. Purification was effected by solution of the cystine in acidified water followed by precipitation of the product as the acid strength of the solution was diminished.

To a well-stirred slurry of 100 g of cystine in 2000 cm^3 of water was added slowly a 6 percent solution of hydrochloric acid until a clear solution resulted. This solution was filtered through fine paper. The cystine was regenerated by raising the *p*H of the solution to about 4.8 by the addition of a concentrated solution of sodium acetate. The precipitate and liquor were placed on a steam bath and maintained at 60° C overnight. The cystine was then filtered off using suction and washed 6 times with hot water. For effective washing, the cystine was removed from the filter to a beaker, stirred with hot water, and refiltered. The products from several runs were combined and dried at 90° C with periodic stirring for several hours. The dried product was of excellent particle size, and free-flowing with very few small, matted pills. The final material was screened through a 40-mesh sieve onto a 100-mesh sieve. An attempt was made to operate using batches of 200 g. However, this larger run did not yield a satisfactory product. The total yield of purified cystine was about 2800 g.

Studies were made to attempt to use infrared or nuclear magnetic resonance spectroscopy for evaluating the purity of the final cystine. However, these methods did not appear to be satisfactory for the purpose. Therefore, the purity of the cystine was evaluated by several microanalyses comparing this material with a previously issued standard reference material. The results on these analyses indicate no significant difference between the two samples within the precision of the methods.

The purification of the cystine was accomplished by Delmo Enagonio. The microanalyses was performed by Rolf A. Paulson and Robert J. Hall.