U. S. Department of Commerce
Maurice H. Stans
Secretary

National Bureau of Standards
L. M. Branscomb, Director

Certificate of Analysis

Standard Reference Material 2141

Urea

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 nitrogen in organic matter. It is also useful for the standardization of techniques for the determination of nitrogen in which carbon and hydrogen are determined simultaneously.

Nitrogen . . . . . 46.63 ± 0.02 percent

The uncertainty shown represents the 95 percent confidence interval of the mean based on 24 determinations and allows for the effects of known sources of possible error. Nitrogen was determined by a modification of the AOAC micro-Kjeldahl method in which the boric acid solution was replaced by 0.05 N hydrochloric acid solution. Analysis of small samples (0.6 mg) indicate the material to be homogeneous within the limits of the technique used.

Analytical measurements to further characterize this material were performed by R. Schaffer, R. F. Brady, Jr., B. Coxon, and J. Thomas of the Analytical Chemistry Division.

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

The technical and support aspects concerning 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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Nitrogen was determined by a modification of the AOAC micro-Kjeldahl method in which the boric acid absorbing solution was replaced by 0.05 N hydrochloric acid solution. Urea samples of approximately 50 mg were accurately weighed and transferred to 30-ml Kjeldahl flasks. Mercuric oxide, potassium sulfate, and sulfuric acid were then added and the samples digested for at least 2 hours. The ammonia was distilled off and absorbed in 50 ml of standardized 0.05 N hydrochloric acid solution. The excess acid was titrated with standardized sodium hydroxide solution to a pH of 5.5. To be certain that all of the ammonia had distilled, the first absorber was replaced by a second containing 5 ml of boric acid solution and the ammonia absorbed titrated directly with standard acid using methyl purple indicator. It was found that when the first distillation ran for 10 minutes, the titration of the second distillation did not exceed 0.10 ml of standard acid.

For homogeneity, 24 samples were selected from the bulk material and analyzed according to a statistical plan. There was no evidence of variability of nitrogen content between samples beyond that observed within samples. There was no evidence of systematic differences in analytical values due to day-to-day variability nor to the order in which the samples were taken.

An estimate of the homogeneity of small (0.6 mg) samples was made using a commercial carbon-hydrogen-nitrogen analyzer. The urea was found to be homogeneous in respect to these elements within the limit of precision of the analytical method.

Phase-solubility analysis using isopropyl alcohol indicated a minimum purity of 99.8 percent. Insoluble matter and ash were both determined to be 0.002 percent. The biuret content was determined to be 0.07 percent. Mass spectrometric examination indicated no detectable impurities. The moisture content as determined by the Karl Fischer titration method is 0.05 percent.

A satisfactory method for further drying this standard reference material was not found. Either incomplete drying, or excessive (and progressive) weight loss occurred, depending on the degree and intensity of heating and evacuation. However, it was found that this material showed little tendency to gain or lose weight (± 0.003 percent) when exposed to laboratory air for a two-week period. Hence, the analyses were performed on this standard reference urea without attempting to lower its moisture content. It is recommended that, after the withdrawal of portions of the standard reference material, the container should be tightly closed. Thus protected, it may be assumed that the moisture content of the standard reference material will remain unchanged.

This standard reference material is useful for the calibration of the various procedures for the microchemical determination of nitrogen including those in which the determination of carbon and hydrogen is performed simultaneously. While highly precise determinations of carbon and hydrogen were not made, the value obtained for the nitrogen content and other supporting evidence indicate the urea to be of high purity. Therefore, it is recommended that the stoichiometric values, namely carbon = 20.00 percent and hydrogen = 6.71 percent, be used for the latter application.