

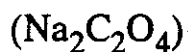


National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 40h

Sodium Oxalate



Reductometric Standard

This Standard Reference Material (SRM) was prepared to provide material of uniform, high purity for use as a working standard for oxidation-reduction reactions. The material conforms to the American Chemical Society specification for reagent-grade material, but should not be considered as entirely free from impurities such as moisture, sodium hydrogen oxalate (0.03%), and sodium hydrogen carbonate (0.03%). The material is not appreciably hygroscopic.

Reductometric Assay 99.972 ± 0.016 weight percent

The above assay is a weighted mean, based on the comparison of this lot of sodium oxalate to SRM 136b Potassium Dichromate, to SRM 136c Potassium Dichromate, and also to the previous Sodium Oxalate, SRM 40g. The uncertainty figure represents the 95 percent confidence interval for the mean of four sets of intercomparison data.

The reductometric assay of SRM 40h, Sodium Oxalate, is based on a molecular weight of 133.999. Corrections for the effect of the buoyancy of air were applied using the value $2.34 \text{ g}\cdot\text{cm}^{-3}$ for the density of sodium oxalate.

The reductometric assay of the sodium oxalate can be used for standardizing KMnO_4 solutions by following the procedure in this Certificate.

Homogeneity testing, reductometric assay, sodium hydrogen oxalate, and sodium hydrogen carbonate determinations were made by K.M. Sappenfield (ret) of the NBS Inorganic Analytical Research Division. American Chemical Society specification tests were made by W.P. Schmidt of the NBS Inorganic Analytical Research Division.

The critical evaluation of data leading to the final certification of this material was performed by George Marinenko and W.F. Koch of the Inorganic Analytical Research Division.

The technical and support aspects involved in the original preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by J.L. Hague. Revision of the Certificate was coordinated through the Standard Reference Materials Program by J.C. Colbert.

This Certificate has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate value or any technical data presented in this Certificate.

Gaithersburg, MD 20899
May 19, 1992
(Revision of Certificate dated 5-4-82)

William P. Reed, Chief
Standard Reference Materials Program

(over)

DRYING INSTRUCTIONS

To remove the small amount of surface moisture (about 0.009 weight percent), dry the samples for two hours at 105 °C.

DIRECTIONS FOR USE IN OXIDIMETRY

Standardization of 0.1N permanganate. Transfer 0.3 g, weighed to the nearest 0.1 mg, of sodium oxalate to a 600-mL beaker. Add 250 mL of dilute sulfuric acid (1 part concentrated to 20 parts final volume with deionized water) previously boiled for 10 to 15 minutes and cooled to room temperature. Stir until the oxalate has dissolved. Add 39 to 40 mL, measured to the nearest 0.02 mL, of 0.1N potassium permanganate at a rate of 25 to 35 mL per minute while stirring slowly. Let stand until the pink color disappears (about 45 seconds; if the pink color should persist because the endpoint has been passed, discard, and begin again, adding less of the KMnO_4 solution: [(0.3 g of sodium oxalate requires approximately 44.8 mL of 0.1N KMnO_4)]. Heat to 55 to 60 °C and complete the titration by adding permanganate until a faint pink color persists for 30 seconds. Add the last 0.5 to 1 mL dropwise, with particular care to allow the solution to become decolorized before the next drop is introduced.

Determine the excess of permanganate required to impart the permanent pink color to the solution. This can be done by matching the color by adding permanganate to the same volume of the specially treated dilute sulfuric acid at 55 to 60 °C. This correction usually amounts to 0.03 to 0.05 mL.

In potentiometric titrations, the correction is negligible if the end point is reached slowly.

For the standardization of more dilute solutions, the same conditions are recommended except that the initial volume and size of sample are proportionally reduced.

In very accurate work, the correction is best obtained iodometrically [1] as follows: Cool the titrated solution to 25 °C, add 0.5 g of KI, 2 mL of starch solution, and titrate the liberated iodine with 0.02N thiosulfate. To obtain the ratio of the thiosulfate to the permanganate solution, add 1 mL of the 0.1N permanganate to 350 mL of the diluted sulfuric acid (1 part concentrated to 20 parts final volume with deionized water), stir, add 0.5 g KI, and titrate with the thiosulfate solution, adding starch just before the end point is reached.

Reference

[1] W.C. Bray, J. Am. Chem. Soc. 32, 1205 (1910)