

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
WASHINGTON

PROVISIONAL CERTIFICATE

OF ANALYSIS OF
STANDARD SAMPLE 40e
SODIUM OXALATE

Purity.- This material meets the specification of the American Chemical Society for Analytical Reagents. A small amount of NaHCO_3 (0.025 percent) is indicated. No fundamental assay of the salt has been made thus far, but the data at hand indicate that the effective purity can be taken as 99.96 if standardization is made as directed.

Directions for use in oxidimetry.

Drying.- Sample No. 40e is not appreciably hygroscopic. However, for an accuracy of better than 1 part in 1000 the sample should be dried for 1 hour at 105°C .

Standardization of 0.1 N permanganate.- Transfer 0.3 g of sodium oxalate (dried at 105°C) to a 600-ml beaker. Add 250 ml of diluted sulfuric acid (5+95) previously boiled for 10 to 15 minutes and then cooled to $27^\circ \text{C} \pm 3^\circ$. Stir until the oxalate has dissolved. Add 39 to 40 ml¹ of 0.1 N potassium

¹ 0.3 g of sodium oxalate should require approximately 44.8 ml of 0.1 N KMnO_4 .

permanganate at a rate of 25 to 35 ml per minute while stirring slowly. Let stand until the pink color disappears² (about 45 seconds). Heat to 55° to 60°C and complete the titration by

² If the pink color should persist because the permanganate is too strong, discard, and begin again, adding less of the KMnO_4 solution.

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 ml³.

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In very accurate work the correction is best obtained iodometrically (cf. W. C. Bray, J. Am. Chem. Soc., 32, 1205 (1910)) 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 0.02 N thiosulfate. To obtain the ratio of the thiosulfate to the permanganate solution, add 1 ml of the 0.1 N permanganate to 350 ml of the diluted sulfuric acid (5+95), stir, add 0.5 g KI, and titrate with the thiosulfate solution, adding starch just before the end point is reached.

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.

It will be noted that the directions for use in oxidimetry differ from those recommended prior to 1935. This change has been made as a result of intercomparison with other primary standards, which showed that the older method gave results that were slightly high. For further details consult National Bureau of Standards Journal of Research 15, 493 (1935) or Research Paper 843.

Use in acidimetry.- Sodium oxalate is issued by this Bureau primarily as an oxidimetric standard, since no thorough investigation has been made here of the effect of conditions upon the results obtained in its use as an acidimetric standard. For further details regarding the testing and use of sodium oxalate, including its use as an acidimetric standard, consult Circular of the Bureau of Standards No. 381 on "The Use of Sodium Oxalate as a Standard in Volumetric Analysis."

(Signed) LYMAN J. BRIGGS, Director.

Washington, D. C.

April 26, 1945.