

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

PROVISIONAL CERTIFICATE OF ANALYSIS

OF

STANDARD SAMPLE 40d

The effective purity of the sodium oxalate can be taken as 99.95 if standardizations are made as directed.

Drying.- Sample No. 40d 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.

Directions for use in oxidimetry.

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° C ± 3°. Stir until the oxalate has dissolved. Add 39 to 40 ml¹ of 0.1 N

1. 0.3 g of sodium oxalate required 44.78 ml of 0.1 N KMnO₄.

potassium 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

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If the pink color should persist because the permanganate is too strong, discard, and begin again, adding a few ml less of the KMnO₄ solution.

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 each drop to become decolorized before the next is introduced.

Determine the excess of permanganate required to impart a 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.

Certificate of Analysis of Standard Sample 40d.

³ 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 ration 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 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 No. 381 on "The Use of Sodium Oxalate as a Standard in Volumetric Analysis."

(Signed) LYMAN J. BRIGGS, Director

Washington, D. C.

June 27, 1940.