

DEPARTMENT OF COMMERCE

Bureau of Standards

Certificate of Analysis

OF

STANDARD SAMPLE No. 40a

SODIUM OXALATE

[The preparation of this sodium oxalate of the requisite purity for use as an oxidimetric standard was undertaken as a laboratory investigation and successfully accomplished by the Mallinckrodt Chemical Works at St. Louis, Mo.]

ANALYSIS ¹

H ₂ O		NaHCO ₃	S	K	Fe	Cl	Organic impurity
105°	240°						
0.005	0.022	0.015	none found	none found	none found	< 0.002	none found

¹ By J. I. Hoffman, Bureau of Standards.

EFFECTIVE PURITY.

If dried at 105° immediately before use, it is believed that the total impurity in this material will not exceed one part in two thousand. Without such drying, the total impurity will probably not exceed one part in one thousand, since this material is not appreciably hygroscopic, except when exposed to atmosphere of very high humidity. The *absolute accuracy* attainable in its use for standardizing can not, however, be assumed to be greater than one part in one thousand, until an exhaustive investigation of the whole subject of volumetric standards has been made.

CONDITIONS FOR USE IN OXIDIMETRY.

"In a 400 cc beaker dissolve 0.25 to 0.30 g of sodium oxalate in 200 to 250 cc of hot water (80°-90°) and add 10 cc of (1 : 1) sulphuric acid. Titrate at once with 0.1 N KMnO₄ solution, *stirring the liquid vigorously and continuously*. The permanganate must not be added more rapidly than 10 to 15 cc per minute, and the last 0.5 to 1 cc must be added dropwise, with particular care to allow each drop to be fully decolorized before the next is introduced. The excess of permanganate used to cause an end point color must be estimated by matching the color in another beaker containing the same bulk of acid and hot water. The temperature of the solution should not be below 60° by the time the end point is reached." (R. S. McBride—J. Am. Chem. Soc. *34*, pp. 393-416; 1912.) For standardization of more dilute permanganate solutions the same conditions are recommended except that the initial volume is proportionately reduced. E. g., with 0.03 N permanganate, such as is frequently used in the bismuthate method for manganese, the initial volume should be about 75 cc.

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. 40 on "The Use of Sodium Oxalate as a Standard in Volumetric Analysis."

S. W. STRATTON,
Director.

Washington, D. C., June 1, 1920

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