



Report of Investigation

Reference Material 8040a

Sodium Oxalate (Reductometric Standard)

This Reference Material (RM) 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 and sodium hydrogen carbonate. A unit of RM 8040a consists of one bottle containing 60 g of crystalline sodium oxalate.

Non-Certified Values and Uncertainties: A non-certified value is the best estimate of the true value; however, the value does not meet NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision and may not include all sources of uncertainty. Non-certified values should not be used to establish metrological traceability to the International System of Units (SI) or other higher-order reference system [1].

Non-certified values for the reductometric assay are expressed as the mass fraction of sodium oxalate, $w_{\text{Na}_2\text{C}_2\text{O}_4}$. The combined standard uncertainty, u , is calculated according to the ISO/JCGM Guide [2]. The value of u is intended to represent the combined effect of the uncertainty components associated with the measurement method and material homogeneity. The value of u is multiplied by the coverage factor, k , which is obtained from the Student's t -distribution with effective degrees of freedom, ν_{eff} , to obtain an expanded uncertainty, U , with an approximate 95 % confidence level.

Table 1. Non-Certified Values for RM 8040a Sodium Oxalate Reductometric Standard

Mass Fraction ($w_{\text{Na}_2\text{C}_2\text{O}_4}$)	Combined Standard Uncertainty (u)	Effective Degrees of Freedom (ν_{eff})	Coverage Factor (k)	Expanded Uncertainty (U)
99.975 %	0.016 %	11	2.2	0.036 %

Traceability: The non-certified value is metrologically traceable to materials and methods used in its determination.

Expiration of Value Assignments: RM 8040a is valid, within the measurement uncertainty specified, until **30 April 2028**, provided the RM is handled and stored in accordance with the instructions given in the Report of Investigation (see "Instructions for Storage and Use"). The report is nullified if the RM is damaged, contaminated, or otherwise modified.

Maintenance of RM Value Assignment: NIST will monitor this RM over the period of its certification. If substantive technical changes occur that affect the value assignment before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the production of RM 8040a was provided by K.W. Pratt and J.F. Waters of the NIST Chemical Sciences Division.

Analytical measurements leading to the production of RM 8040a were made by B.E. Lang and T.W. Vetter of the NIST Chemical Sciences Division.

Statistical analyses were performed by W.F. Guthrie of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this RM were coordinated through the NIST Office of Reference Materials.

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INSTRUCTIONS FOR STORAGE AND USE

Storage: This RM is stable when stored in its original container, with the cap tightly closed, in a dry environment, and under normal laboratory temperatures.

Use: Before it is sampled, the unit should be thoroughly mixed by carefully inverting and rotating the tightly sealed bottle. A minimum test portion mass of 200 mg should be used for analytical determinations.

Drying Instructions: To remove the small amount of surface moisture, dry samples for 2 h at 105 °C. After the material has been dried, store it in a desiccator over anhydrous magnesium perchlorate until use.

SOURCE, HOMOGENEITY, AND ANALYSIS

Source of Material: The $\text{Na}_2\text{C}_2\text{O}_4$ used for this RM was obtained from a commercial supplier. The material was examined for compliance with the specification for reagent grade $\text{Na}_2\text{C}_2\text{O}_4$ as specified by the American Chemical Society [3]. The material was found to meet or exceed the minimum requirements in every respect.

Homogeneity: This RM is homogeneous within the uncertainty limits for the nominal sample mass, 200 mg, used for the reductometric assays. To avoid possible heterogeneity with smaller sample sizes, samples less than 200 mg are not recommended.

Assay Techniques: The non-certified value is based on the titration of sodium oxalate against ceric ammonium nitrate, $(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$, which had been standardized with SRM 40h, *Sodium Oxalate, $\text{Na}_2\text{C}_2\text{O}_4$* , using a modification of the method described by Smith and Fly [4]. The reductometric assay is based on a molecular weight of 133.9983 g/mol [5]. Corrections for the effect of buoyancy in air were applied using the values of 2.34 g/cm³ and 2.61 g/cm³ for the densities of sodium oxalate [6] and ceric ammonium nitrate [4], respectively.

REFERENCES

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