National Institute of Standards and Technology
U.S. Department of Commerce

Date of Issue:
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Standard Reference Material® 136f

Potassium Dichromate

(Oxidimetric Standard)

CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is certified as a chemical of known assay and is intended for use as a primary oxidimetric standard.

Description: A unit of SRM 136f consists of 60 g of highly purified potassium dichromate ($K_2Cr_2O_7$) in a clear glass bottle.

Certified Value: The certified mass fraction of total oxidizing capacity expressed as $K_2Cr_2O_7$, $w_{K_2Cr_2O_7}$, is listed in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [1].

Table 1. Certified Value[(a)] for SRM 136f Potassium Dichromate

$w_{K_2Cr_2O_7}$	99.9954 %	±	0.0044 %

[(a)] The certified value is expressed as the value ± its expanded uncertainty, U. The expanded uncertainty is calculated as $U = ku_c$, where k is the coverage factor and u_c is the combined standard uncertainty calculated according to the ISO/JCGM "Guide to the Expression of Uncertainty in Measurement" [2] and NIST Technical Note 1297 [3]. The value of u_c represents the combined uncertainty in the certified value, at the level of one standard deviation, arising from material homogeneity and from all sources of uncertainty inherent to the coulometric and titrimetric assay techniques. The value of k controls the approximate level of confidence associated with U. For this SRM, $k = 2.00$. This value corresponds to a level of confidence of approximately 95 %. The value of k is obtained from the Student's t-distribution with effective degrees of freedom, $\nu_{eff} > 300$.

Period of Validity: The certified value delivered by **SRM 136f** is valid within the measurement uncertainty specified until **01 February 2028**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Additional Information: Additional information is provided in Appendix A.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Storage: This SRM should be stored in its original bottle at room temperature. It must be tightly re-capped after use and protected from moisture and organic fumes.

Use: Dry for 2 hours at 110 °C. Store the dried material over anhydrous magnesium perchlorate.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

Certificate Revision History on Page 2

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory; NIST Special Publication 260 136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Jan 2023).

[2] JCGM 100:2008; Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jan 2023).

[3] Taylor, B.N.; Kuyatt, C.E., *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at https://www.nist.gov/pml/nist-technical-note-1297 (accessed Jan 2023).

[4] *Reagent Chemicals*, 9th ed., American Chemical Society: Washington, DC (1999).

[5] Pratt, K.W.; *Automated, High-Precision Coulometric Titrimetry Part I. Engineering and Implementation*; *Anal. Chim. Acta*, Vol. 289, pp. 125–134 (1994).

[6] Marinenko, G.; Taylor, J.K.; *Precise Coulometric Titrations of Potassium Dichromate*; J. Res. Natl. Bur. Stand., Vol. 67A, No. 5, pp. 453–459 (1963).

[7] Willard, H.H.; Young, P.; *Standardization of Potassium Dichromate*; Ind. Eng. Chem. Anal. Ed., Vol. 7, No. 1, pp. 57–58 (1935).

[8] Mohr, P.J.; Taylor, B.N.; *CODATA Recommended Values of the Fundamental Physical Constants: 2002*; Rev. Mod. Phys., Vol. 77, pp. 1–107 (2005).

[9] IUPAC, Commission on Isotopic Abundances and Atomic Weights; *Atomic Weights of the Elements 2005*; Pure Appl. Chem., Vol. 78, No. 11, pp. 2051–2066 (2006).

Certificate Revision History: 23 January 2023 (Change of period of validity; updated format; editorial changes); **10 April 2008** (Original certificate date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Homogeneity: Tests indicate that this SRM is homogeneous within the uncertainty limits for sample sizes greater than or equal to 250 mg. The use of samples of mass less than 250 mg is not recommended.

Density: The density of SRM 136f was taken as 2.686 g/cm^3 in the correction for air buoyancy associated with weighing the material.

Source of Material: The potassium dichromate used for this SRM was obtained from a commercial company. The material was examined for compliance with the specification for reagent grade $K_2Cr_2O_7$ as specified by the American Chemical Society [4]. The material was found to meet or exceed the minimum requirements in every respect.

Assay Techniques: Coulometric assays were performed by an automated procedure [5] using electrogenerated iron (II) [6]. Titrimetric assays were performed by reaction with excess arsenious oxide (SRM 83d), with the excess arsenic (III) back-titrated with a standard cerium (IV) solution [7]. The certified value was obtained as a weighted mean of the results from the two techniques. Results were calculated based on a value of 96 485.336 C/mol for the Faraday constant [8] and 294.1846 g/mol for the molar mass of $K_2Cr_2O_7$ [9].

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