



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 136f

Potassium Dichromate (Oxidimetric Standard)

This Standard Reference Material (SRM) is certified as a chemical of known assay and is intended for use as a primary oxidimetric standard. A unit of SRM 136f consists of 60 g of highly purified potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in a clear glass bottle.

Certified Value: The certified mass fraction, $w_{\text{K}_2\text{Cr}_2\text{O}_7}$, of total oxidizing capacity expressed as $\text{K}_2\text{Cr}_2\text{O}_7$ 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

$w_{\text{K}_2\text{Cr}_2\text{O}_7}$	99.9954 %	±	0.0044 %
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^(a) The certified value is expressed as the value \pm 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 "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, $\nu_{\text{eff}} > 300$.

Expiration of Certificate: The certification of SRM 136f is valid, within the measurement uncertainties specified, until **01 February 2023**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Notice and Warnings to Users" and "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements leading to the certification of SRM 136f was provided by K.W. Pratt of the NIST Analytical Chemistry Division.

Coulometric and titrimetric analyses were performed in the NIST Analytical Chemistry Division by K.W. Pratt.

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Robert L. Watters, Jr., Chief
Measurement Services Division

Gaithersburg, MD 20899
Certificate Issue Date: 10 April 2008

NOTICE AND WARNINGS TO USERS

Stability and 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.

Homogeneity: Tests indicate that this SRM is homogeneous within the uncertainty limits for sample sizes greater than 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/cm³ in the correction for air buoyancy associated with weighing the material.

INSTRUCTIONS FOR USE

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

OTHER INFORMATION

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 K₂Cr₂O₇ 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 coul/mol for the Faraday constant [8] and 294.1846 g/mol for the molar mass of K₂Cr₂O₇ [9].

REFERENCES

- [1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000); available at <http://ts.nist.gov/MeasurementServices/ReferenceMaterials/upload/sp260-136.pdf>.
- [2] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993).
- [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 <http://physics.nist.gov/Pubs/pdf.html>.
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- [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).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at telephone (301) 975-6776; fax (301) 926-4751; email srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.