



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 2701

#### Hexavalent Chromium in Contaminated Soil (High Level)

This Standard Reference Material (SRM) is intended primarily for use in the analysis of waste, soils, sediments, or other materials of a similar matrix. SRM 2701 is a soil, heavily contaminated with chromite ore processing residue (COPR), which has been air-dried, sieved, radiation-sterilized, and blended to achieve a high degree of homogeneity. All constituents in SRM 2701 for which certified, reference, and information values are provided were naturally present in the material before processing. A unit of SRM 2701 consists of approximately 75 g of the dried, radiation-sterilized material in an amber screw-capped glass bottle.

**Certified Values:** The certified concentrations of hexavalent chromium, total chromium, iron, and manganese in SRM 2701 are given 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 taken into account [1]. The certified values are based on the agreement of results from two or more independent analytical methods. The certified value for hexavalent chromium is the unweighted mean of analytical measurements by NIST and three collaborating laboratories. The value is based on the extraction of hexavalent chromium from the material using an approved U.S. Environmental Protection Agency (EPA) SW-846 analytical method (Method 3060A), which was used by all participating laboratories. The certified values for total chromium and iron are the unweighted means of results from three independent analytical methods at NIST. The certified value for manganese is the unweighted mean of results from two independent analytical methods at NIST.

**Reference Values:** Reference concentration values for aluminum, calcium, magnesium, potassium, sodium, silicon, titanium, and vanadium are provided in Table 2. Reference values are non-certified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1]. The reference values are based on the results of measurements from a single method performed at NIST.

**Information Values:** Information values for sulfide, total organic carbon (TOC), chemical oxygen demand (COD), pH, and oxidation/reduction potential are provided in Table 3 as additional information on the composition and properties of the material. An information value is considered to be a value that will be of interest and use to the SRM user, but for which insufficient information is available to assess adequately the uncertainty associated with the value, or only a limited number of analyses were performed [1]. The information values are based on measurements from a single collaborating laboratory.

**Expiration of Certification:** The certification of **SRM 2701** is valid, within the measurement uncertainty specified, until **31 December 2018**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "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 2701 was provided by S.E. Long of the NIST Chemical Sciences Division.

Carlos A. Gonzalez, Chief  
Chemical Sciences Division

Gaithersburg, MD 20899  
Certificate Issue Date: 13 September 2013  
*Certificate Revision History on Page 4*

Robert L. Watters, Jr., Director  
Office of Reference Materials

Analytical measurements at NIST were performed by W.C Davis, W.R. Kelly, S.E. Long, J.L. Mann, A.F. Marlow, K.E. Murphy, R.L. Paul, J.R. Sieber, R.O. Spatz, and R.L. Zeisler of the NIST Chemical Sciences Division.

Measurements for the certification of hexavalent chromium were provided by the following collaborating laboratories: H.M. Kingston of Duquesne University (Pittsburgh, PA), D. Dunlop of TestAmerica (Pittsburgh, PA), and R. Gerads of Applied Speciation and Consulting LLC (Tukwilla, WA). Information values are based on data supplied by C. Armbruster of TestAmerica (Edison, NJ).

A multi-laboratory test on the usage of EPA SW-846 methods for hexavalent chromium, leachable chromium, and total chromium in SRM 2701 was conducted by NIST and NJDEP. The results of the study are summarized in Appendix A.

The material was collected by S.E. Long of the NIST Analytical Chemistry Division and B.S. MacDonald of the Office of Reference Materials in collaboration with S.J. Nagourney of the New Jersey Department of Environmental Protection (NJDEP), Office of Quality Assurance (OQA).

Statistical consultation was provided by Z.Q.J. Lu of the NIST Statistical Engineering Division.

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

## NOTICE AND WARNINGS TO USERS<sup>(1)</sup>

**Storage:** SRM 2701 must be stored in its original bottle, tightly capped, at temperatures less than 30 °C and away from light.

**Handling:** This material is a contaminated soil that contains a significant amount of hexavalent chromium. HEXAVALENT CHROMIUM IS A KNOWN CARCINOGEN. Due caution and care should be exercised during its handling and use. This material has been processed to maintain homogeneity and is composed of a particle size distribution that is readily respirable. Stringent precautions should be taken to avoid inhalation of dust during material transfer and in the event of bottle breakage or compromise of the lid seal.

## INSTRUCTIONS FOR USE

**Use:** A minimum sample mass of 200 mg (dry mass – see “Instructions for Drying”) should be used for analytical determinations to be related to the certified values on this Certificate of Analysis. Prior to use, the contents of the bottle should be thoroughly mixed by gently rotating the bottle by hand and inverting several times. As this process may generate electrostatic charges, caution should be exercised when opening the bottle to avoid accidental exposure to the contents.

**Instructions for Drying:** Samples should be dried for 2 h at 110 °C in a laboratory convection oven to obtain a correction factor for moisture. Correction for moisture is to be made to the data before comparison with the certified values. The approximate mass loss on drying has been found to be in the range of 1.9 % to 2.3 % (n = 6), with a mean of 1.93 %.

**Source and Preparation of Material:** The material was processed by S.A. Wilson of the United States Geological Survey (USGS) under contract to NIST. The source material was chromite ore-contaminated soil from a location in Hudson County, New Jersey. Material was acquired from a three-acre site behind the Interpretative Center at the Liberty Science Center in Liberty State Park, Jersey City, NJ. By means of a mechanical excavator, samples of soil were collected in metal drums fitted with polyethylene liners to prevent contact between the material and the surface of the drums. The drums were then sealed and shipped to USGS (Lakewood, CO) for processing. At USGS, the soil was removed from the drums, transferred onto plastic-lined cardboard trays, and dried in a forced-air oven operating at room temperature for three days. Once dry, the material was transferred into a series of plastic-lined, five-gallon buckets. The material was then mechanically disaggregated using a ceramic auger and grinding bowl. The oversized material was separated from the soil fraction by sieving through a vibrating 2 mm screen. The fraction that was less than 2 mm was then ground in 25 kg batches using a ceramic-lined ball mill and corundum grinding media for a period of 8 h. The ground material was transferred to a series of five-gallon containers and

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<sup>(1)</sup> Certain commercial equipment, instruments or materials are identified in this certificate 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.

sealed prior to gamma radiation sterilization at an average dosage rate of 39.1 kGy per container. Upon return to USGS, the material was transferred to a 10 ft<sup>3</sup> cross-flow V-blender and allowed to mix for 24 h. The blended material was then finally split into 4 oz. brown glass screw-capped bottles using a custom-designed spinning riffler.

**Analytical Methods:** The analytical methods used for determination of certified and reference values in SRM 2701 are listed in Table 4.

**Homogeneity:** The homogeneity of SRM 2701 was assessed by analyzing duplicate samples from 20 bottles selected by stratified random sampling. The material was pressed into briquettes and analyzed by wavelength-dispersive X-ray fluorescence spectrometry using 20 selected elements as indicators. The data were subjected to analysis of variance (ANOVA) testing. The findings for between-bottle heterogeneity indicated that, at the 95 % confidence level, none of the bottles' contents exhibited statistically significant differences from the rest of the population of the sampled bottles with the exception of the copper content. For chromium, the relative standard deviation (RSD) of the measurements was less than 1 % for an estimated sample size of 170 mg. Results for all elements listed in Table 2 also had RSDs of less than 1 %, with the exception of sodium, which had an RSD of 1.9 % for an estimated sample size of 3 mg.

Table 1. Certified Values for Concentrations of Hexavalent Chromium and Selected Elements

Hexavalent Cr <sup>(b)</sup>	Mass Fraction <sup>(a)</sup>			
	551.2	mg/kg ±	34.5	mg/kg
Total Cr	4.26	% ±	0.12	%
Fe	23.73	% ±	0.19	%
Mn	0.2137	% ±	0.0014	%

<sup>(a)</sup> Each certified concentration value, expressed as a mass fraction on a dry-mass basis, is an equally weighted mean of the individual sets of results. The uncertainty in the certified value, calculated according to the method described in the ISO/JCGM Guide [2], is expressed as an expanded uncertainty,  $U$ . The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  is intended to represent, at the level of one standard deviation, the combined effects of inherent sources of uncertainty of the measurements and corrections for interferences and species interconversions (hexavalent chromium). The value of the coverage factor,  $k = 2$ , corresponds to a level of confidence of approximately 95 %.

<sup>(b)</sup> As determined after extraction using EPA Method 3060A.

Table 2. Reference Values for Concentrations of Selected Elements

Element	Mass Fraction, % <sup>(a)</sup>			Element	Mass Fraction, % <sup>(a)</sup>		
Al	5.05	±	0.15	Na	0.255	±	0.005
Ca	7.47	±	0.09	Si	4.17	±	0.04
K	0.174	±	0.007	Ti	0.547	±	0.008
Mg	7.47	±	0.17	V	0.236	±	0.011

<sup>(a)</sup> Each reference concentration value, expressed as a mass fraction on a dry-mass basis, is an equally weighted mean of the individual sets of results. The uncertainty in the reference value, calculated according to the method described in the ISO/JCGM Guide [2], is expressed as an expanded uncertainty,  $U$ . The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  is intended to represent, at the level of one standard deviation, the combined effects of inherent sources of uncertainty of the measurements and corrections for interferences. The value of the coverage factor,  $k = 2$ , corresponds to a level of confidence of approximately 95 %.

Table 3. Information Values for Selected Species and Properties

Species/Property	Value	Unit
Chemical oxygen demand (COD)	<10	mg/kg
Redox potential	526	mV
Sulfide	<10	mg/kg
Total organic carbon (TOC)	36900	mg/kg
pH	9.6	

Table 4. Analytical Methods Used for Determination of  
Certified and Reference Values in SRM 2701

Analyte	Methods
Hexavalent Cr	EPA Method 3060A [3] and EPA Method 6800 [3,4] performed at collaborating laboratories  EPA Method 3060A [3] and speciated isotope dilution inductively coupled plasma mass spectrometry (SID-ICP-MS) [4] performed at NIST
Total Cr	Isotope dilution (ID) ICP-MS [5] performed at NIST  X-ray fluorescence spectrometry (XRF) following borate fusion preparation performed at NIST  Instrumental neutron activation analysis (INAA) performed at NIST
Fe	XRF following borate fusion performed at NIST  INAA performed at NIST  Prompt gamma activation analysis (PGAA) performed at NIST
Mn	XRF following borate fusion performed at NIST  INAA performed at NIST
Al, Ca, K, Mg, Na, Si, Ti, V	XRF following borate fusion performed at NIST

#### REFERENCES

- [1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at <http://www.nist.gov/srm/upload/SP260-136.PDF> (accessed Sep 2013).
- [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 [http://www.bipm.org/utls/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed Sep 2013); see also 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); <http://www.nist.gov/pml/pubs/index.cfm> (accessed Sep 2013).
- [3] *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*; United States Environmental Protection Agency, Office of Solid Waste, Washington D.C.; available at <http://www.epa.gov/epaoswer/hazwaste/test/main.htm> (accessed Sep 2013).
- [4] Huo, D.; Kingston, H.M.; *Correction of Species Transformations in the Analysis of Cr(VI) in Solid Environmental Samples Using Speciated Isotope Dilution Mass Spectrometry*; Anal. Chem., Vol. 72, pp. 5047–5054 (2000).
- [5] Kelly, W.; Murphy, K.; Becker D.; Mann, J.; *Determination of Cr in Certified Reference Material HISS-1, Marine Sediment, by Cold Plasma Isotope Dilution ICP-MS and INAA: Comparison of Microwave Versus Closed (Carius) Tube Digestion*; J. Anal. At. Spectrom., Vol. 18, pp. 166–169 (2003).

<p><b>Certificate Revision History:</b> <b>13 September 2013</b> (Extension of certification period; editorial changes); <b>23 September 2009</b> (Editorial changes and additional information added to Table A2 in Appendix A); <b>23 January 2009</b> (editorial changes); <b>13 January 2009</b> (Original certificate date).</p>
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Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.

## APPENDIX A

Multi-Laboratory Testing Using Approved U.S. Environmental Protection Agency (EPA) Analytical Methods: Method 3060A, Method 7196A, Method 7199, and Method 6800 (Hexavalent Chromium), Method 3050 (Leachable Chromium), and Method 3052 (Total Chromium)

The certified values of inorganic constituents in most National Institute of Standards and Technology (NIST) chemical composition Standard Reference Materials (SRMs) reflect the total absolute concentration of each measurand in the given SRM matrix. The certified concentrations are based on rigorous measurements obtained by well-characterized primary methods, or two or more independent methods or techniques. If dissolution of the matrix is necessary, the measurement approach generally requires complete sample decomposition, which can usually be achieved in a high-pressure digestion system such as a Carius tube. Alternatively and where applicable, the sample may also be analyzed nondestructively.

For environmental monitoring purposes, the concentrations of labile or extractable fractions of elements are often more useful than total concentrations. Concentrations of labile or extractable fractions are generally determined using relatively mild leach conditions, which are unlikely to totally decompose the sample. Reported concentrations of labile or extractable fractions of elements are generally lower than total absolute concentrations; recovery can be total only if an element in a given sample is completely labile. The recovery of an element as a percentage of the total concentration is a function of several factors that are defined by the sample matrix and the measurement method conditions.

In its monitoring programs, the U.S. EPA has promulgated a number of analytical methods to determine toxic elements, or element species, in soils, sediments, and sludges. The EPA publication SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* [3], is the official compendium of analytical and sampling methods compiled by the EPA Office of Solid Waste (OSW). The methods have been evaluated and approved for use in accordance with the 1976 Resource Conservation and Recovery Act (RCRA). SW-846 Methods 3060A, 7196A, 7199, and 6800 are applicable to the measurement of hexavalent chromium, while Methods 3050 and 3052 are applicable to measurements of leachable chromium and total chromium respectively. Method 3060A is currently the best available analytical method for the extraction of hexavalent chromium from solid matrices and preservation of the oxidation state prior to analytical determinations using other finishing methods such as 7196A (colorimetric), 7199 (ion chromatography with post-column derivatization), and 6800 (elemental and speciated isotope dilution mass spectrometry). Method 3060A was used as the common basis for the certified value of hexavalent chromium in SRM 2701. Method 3050 is a strong acid leachable digestion method intended for the dissolution of elements that are environmentally available, while Method 3052 employs microwave-assisted digestion of solid samples, which is intended to provide almost complete decomposition of the sample, including silica, primarily using concentrated nitric acid and hydrofluoric acid combined with microwave heating.

To assess the performance of these SW-846 methods for the determination of hexavalent chromium, leachable chromium, and total chromium in SRM 2701 and to provide data that are relevant to users of these SW-846 methods, a multi-laboratory test was organized jointly by NIST and the New Jersey Department of Environmental Protection (NJDEP), Office of Quality Assurance (OQA) involving 22 participating laboratories listed in Table A1. Stratified random samples of SRM 2701 were distributed to a number of collaborating laboratories who were asked to measure the hexavalent chromium, leachable chromium, and/or total chromium concentrations in the material. Several laboratories provided replicate analyses for each data set from which the mean was calculated. The means of all the data sets forwarded by each laboratory are compiled by method in Table A2 and include summary statistics together with the percent recovery relative to the certified value.

Please note that none of the values in Table A2 are certified; these values are provided as information on the typical relative performance of the various EPA SW-846 methods for the determination of hexavalent chromium and total chromium in SRM 2701. The data should not be used for any other purpose. **The certified values, provided in Table 1 of the Certificate of Analysis, are the best estimates of the true concentrations.**

Table A1. Laboratories Participating in the Multi-laboratory Study of SRM 2701 Usage with SW-846 Methods  
(Appendix A)

S. Nagourney New Jersey Department of Environmental Protection Office of Quality Assurance Trenton, NJ, USA	J. Blouin Direction du Laboratoire des Pollutions Industrielles Center D-Expertise en Analyse Environnementale du Quebec Laval, Quebec, Canada
S. Wilson U.S. Geological Survey Lakewood, CO, USA	M. Goss Kentucky Department of Environmental Protection Frankfort, KY, USA
D. Speis Accutest Laboratories Dayton, NJ, USA	A. Doupe Lancaster Laboratories Lancaster, PA, USA
L. Call ALS Laboratory Group Environmental Division Smithfield, NSW, Australia	J. Ghandi Metrohm-Peak Houston, TX, USA
R. Gerads Applied Speciation and Consulting, LLC Tukwila, WA, USA	V. Shah City of New York Department of Environmental Protection Wards Island, NY, USA
L. Reyes Columbia Analytical Services Rochester, NY, USA	C. Nowlan Pace Analytical Services, Inc. Minneapolis, MN, USA
D. Thomas Dionex Corporation Sunnyvale, CA, USA	B. Buckley Rutgers University Environmental and Occupational Health Sciences Institute Piscataway, NJ, USA
H.M. Kingston Duquesne University Department of Chemistry and Biochemistry Pittsburgh, PA, USA	C. Armbruster TestAmerica, Edison Edison, NJ, USA
J. DuPont DHL Analytical, Inc. Round Rock, TX, USA	D. Dunlap TestAmerica, Pittsburgh Pittsburgh, PA, USA
B. Goyette EMT Morton Grove, IL, USA	M. Nash Thermo Electron Corporation Winsford, Cheshire, UK
C. Huff Environmental Resource Associates Arvada, CO, USA	O.X.N. Donard University of Pau HELIOPARC Pau, France

Table A2. Multi-Laboratory Concentration Data (Dry-Mass Basis) for SRM 2701 by SW-846 Methods

Data Set	Method 7196A Hexavalent Cr mg/kg	Method 7199 Hexavalent Cr mg/kg	Method 6800 Hexavalent Cr mg/kg	Method 3050 Leachable Cr % mass fraction	Method 3052 Total Cr % mass fraction
1	503.8	389.1	553.0	0.294	1.025
2	314.3	354.4	568.7	0.384	
3	415.7	461.6	529.8	0.465	
4	408.7	391.1	549.4	0.514	
5	399.9	405.7	575.2	0.317	
6	326.2	363.3	544.3	0.255	
7	266.0	358.6		0.360	
8	500.5	557.2		0.597	
9	351.7	414.2		0.441	
10	364.9	274.2		0.833	
11	336.7	393.8		0.236	
12	469.2	439.3		0.235	
13	338.4	270.4			
14		368.3			
Mean	<b>384.3</b>	<b>388.7</b>	<b>553.4</b>	<b>0.411</b>	<b>1.025</b>
SD <sup>(a)</sup>	73.5	72.1	16.5	0.175	
RSD <sup>(b)</sup>	19.1	18.5	3.0	42.7	
Median	364.9	390.1	551.2	0.372	
Minimum	266.0	270.4	529.8	0.235	1.025
Maximum	503.8	557.2	575.2	0.833	1.025
# Labs <sup>(c)</sup>	12	10	3	10	1
# Measurements <sup>(d)</sup>	13	14	6	12	1
Recovery (%) <sup>(e)</sup>	69.7	70.5	100.4	9.6	24.1

<sup>(a)</sup> Standard deviation<sup>(b)</sup> Relative standard deviation<sup>(c)</sup> Number of laboratories reporting data sets<sup>(d)</sup> Number of data sets reported by all laboratories<sup>(e)</sup> Mean recovery for the method relative to the certified value expressed as a percentage