

Role of SIT in the accreditation of ISPRA Laboratories for Reference Materials production

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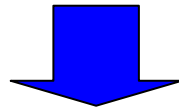
INRIM – Istituto Nazionale di Ricerca Metrologica
Torino (Italia)

SIT accreditation

- **Evaluation of the technical procedures**
- **Technical assessment**
 - assessment visit
 - selection of a proper comparison
 - data evaluation
- **Reporting the results: calibration certificate**

Scope of ISPRA's accreditation

Measurands: mass fraction [$\mu\text{g/g}$] of metals (As, Cd, Ni, Pb, Cu, Co, Mn) in matrices of sediment and soil



**accreditation for the
production of **CRMs****

Accreditation of a CRM producer



EN ISO/IEC 17025

**General
requirements
for the competence
of testing and
calibration
laboratories**

ISO Guide 34

**General
requirements
for the competence
of reference
material producers**

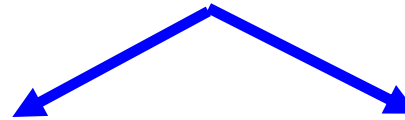
Evaluation of the whole production process

- **Selection** of a candidate CRM
- **Material preparation** (incl. particle size distribution)
- **Assessment of homogeneity** (within bottle and between bottles) and **stability**
- **Traceability**
- **Characterization** (*ad hoc* interlaboratory study)
- **Assignment of property values** and their uncertainties

Experimental verification

- **Selection of suitable CRMs**
- **Analysis by the laboratory following the procedures subjected to accreditation**
- **Data evaluation**
- **Calculation of a compatibility index (E_n , EA-2/03)**

CRMs for the experimental verification



NIST SRM n. 2709
San Joaquin soil

NIST RM 8704
Buffalo river sediment



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2709

San Joaquin Soil

Baseline Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2709 is an agricultural soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2709 consists of 50 g of the dried material.

The certified elements for SRM 2709 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should NOT be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification of SRM 2709 is valid, within the measurement uncertainties specified, until 31 December 2011, provided the SRM is handled in accordance with instructions given in this certificate (see *Instructions for Use*). This 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. Return of the attached registration card will facilitate notification.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Waters, Jr. of the NIST Inorganic Analytical Research Division.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by T.E. Gills and J.S. Kane. Revision of this certificate was coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Willie E. May, Chief
Analytical Chemistry Division

John Rumble, Jr., Chief
Measurement Services Division

Gaithersburg, MD 20899
Certificate Issue Date: 18 July 2003
See *Certificate Revision History* on Page 6

Analyte	Mass fraction $\mu\text{g/g}$	$U (k=2)$ $\mu\text{g/g}$
Mn	538	17
Co	13.4	0.7
Ni	88	5
Cu	34.6	0.7
As	17.7	0.8
Cd	0.38	0.01
Pb	18.9	0.5

Results

NIST SRM n. 2709
San Joaquin soil

Analyte	E_n
Mn	-0.7
Co	0.6
Ni	0.4
Cu	0.9
As	0.5
Cd	0.03
Pb	-0.1



Report of Investigation

Reference Material 8704

Buffalo River Sediment

This Reference Material (RM) is intended primarily for use in the analysis of sediments, soils, or materials of a similar matrix. A unit of RM 8704 consists of 50 g of freeze-dried, radiation-sterilized, homogenized, river sediment that is very similar in composition to SRM 2704, Buffalo River Sediment, having been collected at the same time and location (see "Source and Preparation of Material").

Reference Concentration Values: Reference values for concentrations of 25 elements are given in Table 1. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty.

Information Concentration Value: An information value for arsenic is provided in Table 2. An information value is a noncertified value for which there is insufficient information to assign an uncertainty.

Expiration of Reference Values: The reference values of RM 8704 are valid, within the measurement uncertainties specified, until 01 December 2017, provided the RM is handled in accordance with instructions given in this report (see "Instructions for Use"). The reference values are nullified if the RM is contaminated or otherwise modified.

Maintenance of Reference Values: NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect the reference values before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

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

The overall direction and coordination of the analyses were provided by G.C. Turk of the NIST Analytical Chemistry Division.

Spectrometric measurements were performed by A.P. Lindstrom, G.C. Turk, L.J. Wood, and L.L. Yu of the NIST Analytical Chemistry Division. Instrumental Neutron Activation Analysis was performed by R.R. Greenberg of the NIST Analytical Chemistry 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
Report Issue Date: 08 January 2008
See Report Revision History on Last Page

Analyte	Mass fraction μg/g	U (k=2) μg/g
Mn	544	21
Co	13.57	0.43
Ni	42.9	3.7
Cu	-	-
As	17	-
Cd	2.94	0.29
Pb	150	17

Results

NIST RM 8704
Buffalo river sediment

Analyte	E_n
Mn	-0.8
Co	0.6
Ni	0.04
Cu	-
As	-
Cd	-0.5
Pb	-0.2

Conclusions

- **Competence of personnel**
- **Technical procedures**
- **Accommodation and environmental conditions**
- **Calibrations, traceability, method validation, uncertainty evaluation, equipment**
- **Calibration certificates (according to SIT requirements and ISO Guide 31)**
- **Outcomes from the assessment visit and experimental verification**

ISPRA laboratory fulfilled the technical requirements for the accreditation according to EN ISO/IEC 17025 and ISO Guide 34

**Thank you
for your attention**