

# NBS SPECIAL PUBLICATION 260-105

U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Standard Reference Materials:

Summary of the Environmental Research, Analysis, and Control Standards Issued by the National Bureau of Standards

**R. Mavrodineanu and S. D. Rasberry** 

-QC 100 .U57 260-105 1986 C. 2 he National Bureau of Standards<sup>1</sup> was established by an act of Congress on March 3, 1901. The Bureau's overall goal is to strengthen and advance the nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, the Institute for Computer Sciences and Technology, and the Institute for Materials Science and Engineering.

## The National Measurement Laboratory

Provides the national system of physical and chemical measurement; coordinates the system with measurement systems of other nations and furnishes essential services leading to accurate and uniform physical and chemical measurement throughout the Nation's scientific community, industry, and commerce; provides advisory and research services to other Government agencies; conducts physical and chemical research; develops, produces, and distributes Standard Reference Materials; and provides calibration services. The Laboratory consists of the following centers:

- Basic Standards<sup>2</sup>
  - Radiation Research
  - Chemical Physics
  - Analytical Chemistry

## The National Engineering Laboratory

Provides technology and technical services to the public and private sectors to address national needs and to solve national problems; conducts research in engineering and applied science in support of these efforts; builds and maintains competence in the necessary disciplines required to carry out this research and technical service; develops engineering data and measurement capabilities; provides engineering measurement traceability services; develops test methods and proposes engineering standards and code changes; develops and proposes new engineering practices; and develops and improves mechanisms to transfer results of its research to the ultimate user. The Laboratory consists of the following centers:

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Conducts research and provides scientific and technical services to aid Federal agencies in the selection, acquisition, application, and use of computer technology to improve effectiveness and economy in Government operations in accordance with Public Law 89-306 (40 U.S.C. 759), relevant Executive Orders, and other directives; carries out this mission by managing the Federal Information Processing Standards Program, developing Federal ADP standards guidelines, and managing Federal participation in ADP voluntary standardization activities; provides scientific and technological advisory services and assistance to Federal agencies; and provides the technical foundation for computer-related policies of the Federal Government. The Institute consists of the following centers:

## The Institute for Materials Science and Engineering

Conducts research and provides measurements, data, standards, reference materials, quantitative understanding and other technical information fundamental to the processing, structure, properties and performance of materials; addresses the scientific basis for new advanced materials technologies; plans research around cross-country scientific themes such as nondestructive evaluation and phase diagram development; oversees Bureau-wide technical programs in nuclear reactor radiation research and nondestructive evaluation; and broadly disseminates generic technical information resulting from its programs. The Institute consists of the following Divisions:

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- Metallurgy
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<sup>1</sup>Headquarters and Laboratories at Gaithersburg, MD, unless otherwise noted; mailing address Gaithersburg, MD 20899.

<sup>&</sup>lt;sup>2</sup>Some divisions within the center are located at Boulder, CO 80303.

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#### Preface

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards (NBS) are well-characterized materials, produced in quantity and certified for one or more physical or chemical properties. They are used to assure the accuracy and compatibility of measurements throughout the Nation. SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. They are also used extensively in the fields of environmental and clinical analysis. In many applications, traceability of quality control and measurement processes to the national measurement system is carried out through the mechanism and use of SRM's. For many of the Nation's scientists and technologists it is therefore of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values of the SRM's produced. An NBS series of papers, of which this publication is a member, called the <u>NBS Special Publication - 260 Series</u>, is reserved for this purpose.

The 260 Series is dedicated to the dissemination of information on different phases of the preparation, measurement, certification and use of NBS SRM's. In general, much more detail will be found in these papers than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity and accuracy of the measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing the greatest care and accuracy. These papers also should provide sufficient additional information not found on the certificate so that new applications in diverse fields not foreseen at the time the SRM was originally issued will be sought and found.

Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth, will receive prompt attention from:

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> Stanley D. Rasberry, Chief Office of Standard Reference Materials

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- Mavrodineanu, R. and Rasberry, S.D., Standard Reference Materials: Summary of the Environmental Research, Analysis, and Control Standards Issued by the National Bureau of Standards, NBS Spec. Publ. 260-105.
  - \* Send order with remittance to Superintendent of Documents, US Government Printing Office Washington, DC 20402. Remittance from foreign countries should include an additional one-fourth of the purchase price for postage.
- \*\* May be ordered from: National Technical Information Services (NTIS). Springfield Virginia 22161.

#### TABLE OF CONTENTS

Pa	ge
Preface	ii
Abstract	х
Introduction	1
Table 1. Categories of Standard Reference Materials available from the           National Bureau of Standards         • • • • • • • • • • • • • • • • • • •	2
Environmental Research, Analysis, and Control Standards	4
Table 2.       Summary of the Environmental Research, Analysis, and Control         Standards	6
Table 3.       Comparative List of Chemical Elements for the SRM's Described         in Table 2	12
Appendix I. Alphabetical Index by Standard Reference Material Name	15
Appendix II. Certificates for the Environmental Research, Analysis, and Control Standards (listed in numerical order)	28
Appendix III. Guide for Requesting Development of Standard Reference Materials	81
Appendix IV. Guide to Ordering Standard Reference Materials	82

#### Abstract

This publication is a summary of the environmental research, analysis, and control standards issued by NBS as Standard Reference Materials (SRM's). The material, composition, certification, use, and remarks concerning each of the SRM's described are presented in tabular form. Copies of the certificates of these SRM's are contained in the appendix for more detailed information.

Key Words: chemical composition; environmental standards; Standard Reference Materials.

#### Introduction

Since its inauguration in 1901, the National Bureau of Standards (NBS) has issued nearly 2000 different Standard Reference Materials (SRM's). Many of these have been renewed several times, many have been replaced or discontinued as technology changed. Today, over 900 SRM's are available, together with a large number of scientific publications related to the fundamental and applied characteristics of these materials. Each material is certified for chemical composition, chemical properties, or its physical or mechanical characteristics. Each SRM is provided with a Certificate or Certificate of Analysis that contains the essential data concerning its properties or characteristics. The SRM's currently available cover a wide range of chemical, physical, and mechanical properties, and a corresponding wide range of measurement interests in practically all aspects of fundamental and applied science. These SRM's constitute a unique and invaluable means of transferring to the user accurate data obtained at NBS, and provide essential tools that can be used to improve accuracy in practically all areas where measurements are performed.

In addition to SRM's, the National Bureau of Standards issues a variety of Research Materials (RM's) having various properties described in individual "Reports of Investigation." They are intended primarily to further the scientific or technical research on that particular material. Other materials, called Special Reference Materials (GM's), are also available from NBS. These are materials produced and certified by other Government agencies, standard organizations, or other nonprofit organizations, that are considered useful to the public and for which no alternate method of national distribution exists.

The various categories of materials available from NBS are given in table 1. This table lists these materials according to their chemical composition, physical properties, or engineering characteristics. A more detailed alphabetic enumeration of these materials is given in appendix I. Table 1 and appendix I were taken from NBS Special Publication 260, NBS Standard Reference Materials Catalog, 1984-85 Edition<sup>1</sup>. This publication lists every material available from the NBS Office of Standard Reference Materials.

Further information on the reference materials available from NBS may be obtained from the Office of Standard Reference Materials, National Bureau of Standards, Gaithersburg, MD 20899. Information on other NBS services may be obtained from the Technical Information and Publications Division, National Bureau of Standards, Gaithersburg, MD 20899.

In addition to reference materials, NBS provides many additional services. These include: Measurement Assurance Programs, Calibration and Related Measurement Services, Proficiency Sample Programs, a National Voluntary Laboratory Accreditation Program, Standards Information Services, Standard Reference Data, and Technical Information and Publications.

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, under Stock No. 003-003-02558-5 (Price \$5.50, add 25 percent for foreign orders.)

## Table 1. Categories of Standard Reference Materials available from the National Bureau of Standards.

#### CERTIFIED CHEMICAL COMPOSITION STANDARDS

Steels (chip form) Plain carbon Low alloy High alloy Stainless Tool Steels (granular form) Steels (solid form) Ingot iron and low alloy Special ingot irons and low alloy Stainless Specialty High-temperature alloys Tool Steelmaking Alloys Cast Irons (chip form) Cast Steels, White Cast Irons, Ductile Irons, and Blast Furnace Irons (solid form) Nonferrous Alloys (chip form) Aluminum "Benchmarks" Cobalt Copper Copper "Benchmarks" Lead Magnesium Nickel Nickel Superalloy, Trace Elements Nickel oxide Selenium Tin Titanium Zinc Zirconium Nonferrous Alloys (solid form) Aluminum "Benchmarks" Copper Copper "Benchmarks" Lead Nickel Titanium Zinc Zirconium

Gases in Metals High-Purity Metals Electron Probe Microanalytical Standards Primary, Working, and Secondary Standard Chemicals Microchemical Standards Clinical Laboratory Standards Biological Standards Environmental Standards Analyzed gases Analyzed liquids and solids Permeation tubes Industrial Hygiene Standards Spectrochemical Standards Hydrocarbon Blends Metallo-Organic Compounds Fertilizers Ores Minerals, Refractories, Glasses, and Carbides Cement Trace Element Standards Nuclear Materials Special nuclear materials Plutonium assay Plutonium isotopic Uranium assay Uranium isotopic Neutron density standards Fission track glass standards Isotopic Reference Standards

Table 1. continued.

#### CERTIFIED PHYSICAL PROPERTY STANDARDS

Ion Activity Standards Optical Standards pH standards Spectrophotometric pD standards Thermal emittance Ion selective electrodes Refractive index Mechanical and Metrology Standards Radioactivity Standards Magnification Alpha-particle standards Coating thickness Beta-particle and gamma-ray gas Glass standards Elasticity Alpha-particle, beta-particle, Density gamma-ray, and electron-capture Polymer solution standards Contemporary standard for carbon-14 Rheology Rheology dating laboratories Heat Standards Environmental standards Low energy photon sources Gamma-ray "point-source" standards Radium gamma-ray solution standards Superconductive thermometric fixed point devices Freezing Points Radium solution standards for random analysis Defining fixed points Radioactivity standard reference Determined reference points materials currently not in stock Melting points Metallurgical Calorimetric Mossbauer Combustion X-ray Diffraction Solution Heat source Enthalpy and heat capacity Gas Transmission Vapor pressure Permittivity Thermal expansion Thermocouple materials Reference Fuels Thermal resistance Resistivity Magnetic Standards Magnetic susceptibility

ENGINEERING TYPE STANDARDS

Standard Rubber and Rubber- Compounding Materials	X-ray and Photographic Standards
Reference Magnetic Tapes	Surface Flammability Standards
Reference Magnetic Tapes	Semiconductor Production Standard
Lubricant Standards	Water Vapor Permeance
Sizing Standards	Tape Adhesion Testing Standards
Glass spheres for particle size Turbidimetric and fineness (cement)	Color Standards
RESEARCH MATERIALS	SPECIAL REFERENCE MATERIALS

Magnetic moment

Paramagnetic resonance

This work is the fourth in a series of NBS Special Publications dedicated to the description of the Standard Reference Materials (SRM's) issued by the National Bureau of Standards (NBS).

The first volume, NBS SP 260-71, "Summary of the Clinical Laboratory Standards Issued by the National Bureau of Standards" (176 pp., November 1981), describes in a tabular form 41 SRM's available in that field and includes copies of the corresponding Certificates of Analysis for further information.

The second volume, NBS SP 260-97, "Summary of the Coal, Ore, Mineral, Rock, and Refractory Standards Issued by the National Bureau of Standards" (134 pp., September 1985), describes in the same manner 39 SRM's available in that field.

The third volume, NBS SP 260-104, "Summary of the Biological and Botanical Standards Issued by the National Bureau of Standards" (68 pp., October 1985), presents in a similar manner the essential data concerning 9 SRM's issued in that field.

The present volume, NBS SP 260-105, "Summary of the Environmental Research, Analysis, and Control Standards Issued by the National Bureau of Standards" (97 pp., March 1986), describes in a tabular form the characteristic properties of 22 SRM's issued in the field of environmental analytical instrumentation and methodology. Copies of the Certificates of Analysis are reproduced in this work also, for further information.

Tables 2 and 3 contain the essential information concerning the material composition, the certification parameters, and use. Under "Remarks," additional data such as storage conditions and stability is provided. All the data and information contained in these tables were extracted from the Certificates or Certificates of Analysis issued for the SRM's included in the table. An examination of these tables gives the reader a general view of these SRM's. For more detailed information, the individual Certificates reproduced in appendix II should be consulted as well as the references cited in each Certificate. The SRM's listed in the tables include all of the environmental standards that were issued or were in preparation by the end of 1984. These SRM's are the result of the concerted efforts of a number of scientists from the NBS National Measurement Laboratory. Each Certificate lists the individuals who contributed to development of the SRM.

Appendix III provides a guide to the reader to assist in requesting NBS to develop new SRM's. A final appendix, appendix IV, is a guide to ordering SRM's.

In addition to the SRM's and their Certificates, NBS issues a series of Special Publications, called the "260 Series," that relate directly to Standard Reference Materials as stated in the preface. The list of available publications in the "260 Series" is given at the beginning of this publication.<sup>2</sup>

<sup>&</sup>lt;sup>1</sup>NOTE: The use of proprietary designations in table 2 is for information only, and should not be construed as an endorsement of the product by either the Department of Commerce or the National Bureau of Standards.

<sup>&</sup>lt;sup>2</sup> For complete bibliographic reference and ordering information, see "Other NBS Publications in This Series," pp. iv.

 TABLE 2.
 SUMMARY OF THE ENVIRONMENTAL

 RESEARCH, ANALYSIS, AND
 CONTROL STANDARDS.

SRM	Material	COMPOSITION		
1579 Powdered Lead Based Paint	Collected by the Philadelphia Dept. Public Health, and sieved to a powder passing through a 325 mesh sieve.	Pb: 11.87 <u>+</u> 0.04 % by wt., based on samples <u>&gt;</u> 0.1 g of as-received material.		
1580 Organics in Shale Oil	The shale oil came from the Laramie Energy Technology Center, Laramie, Wyoming, and was collected from the Mahogany Zone of the Colorado Green River Formation.	Fluoranthene: 54; pyrene: 104; benzo[a]pyrene: 21; benzo[e]pyrene: 18; perylene: 3.4; phenol: 407; o-cresol: 385; 2,6-dimethylphenol: 175; benzol[f]quinoline: 16, µg/g.		
1620a Sulfur in Residual Fuel Oil	The material is a commercial "No. 5 Heavy" residual fuel oil as defined by the American Society for Testing and Materials.	S: 4.504 <u>+</u> 0.010 wt. %.		
1621b Sulfur in Residual Fuel Oil	The material is a commercial "No. 6" residual fuel oil as defined by the American Society for Testing and Materials.	S: 0.950 <u>+</u> 0.005 wt. %.		
1622b Sulfur in Residual Fuel Oil	Same as for SRM 1621b.	S: 1.982 <u>+</u> 0.018 wt. %.		
1623a Sulfur in Residual Fuel Oil	Same as for SRM 1620a.	S: 0.240 <u>+</u> 0.003 wt. %.		
1624a Sulfur in Distillate (Diesel) Fuel Oil	The material is a commercial "No. 2-D" distillate fuel oil as defined by the American Society for Testing and Materials.	S: 0.141 <u>+</u> 0.002 wt. %.		
1630 Trace Mercury in Coal	Commercial coal crushed to 210-500 micrometer particle size.	Hg: 0.13 μg/g <u>+</u> l in the last significant figure.		

Certification	Use	Remarks		
By x-ray fluorescence, atomic absorption spectrometry, polarography.	In the calibration of apparatus and methods for determining Pb in paint removed from the interior surfaces of old housing.			
By gas chromatography, gas chromatography/mass spec- trometry, and high performance liquid chromatography.	For evaluating the reliability of analytical methods used for trace organic compounds in oil materials.	Seven additional organic compounds are included for information only and are not certified.		
By gravimetry, ion chroma- tography, and x-ray fluor- escence.	As analytical standard in the determination of total S in fuel oils and similar materials.	Four additional physical properties are indicated but are not certified; similarly, l6 trace elements are mentioned semi-quantitatively only.		
As for SRM 1620a. Is valid for 3 years from date of purchase.	As for SRM 1620a.			
As for SRM 1621b.	As for SRM 1621b.			
As for SRM 1620a.	As for SRM 1620a.			
By gravimetry and ion chromatography. Is valid for 3 years from date of purchase.	As for SRM 1620a.			
By neutron activation and flameless atomic absorption spectrometry (0.14 µg/g). The neutron activiation procedure is described in the certificate.	As analytical standard for the determination of trace mercury in coal.	Selenium is also given for information, but is not certified (2.1 µg/g).		

#### Table 2. Continued.

SRM	Material	Composition		
1632b Trace Elements in Coal (Bituminous)	Obtained from an underground mine that recovers coal from the Pittsburgh seam, crushed and sieved through a ~60 mesh at the Humphrey No. 7 mine and coal preparation plant of the Consolidation Coal Co., Christopher Coal Co. Div., Osage, W. Va.	Material should be vacuum dried at ambient temperature for 24 hours prior to use. Values based on a minimum sample size of 250 mg. C (total) 78.11; H 5.07; N 1.56; S 1.89; Volatile matter 35.4; Al 0.855; Ca .204; Fe .759; Mg .0383; K .0748; Na .0515; Ti .0454; values % by wt. As 3.72; Ba 67.5; Cd 0.0573; Co 2.29; Cu 6.28; Pb 3.67; Mn 12.4; Ni 6.10; Rb 5.05; Se 1.29; Th 1.342; U 0.436; Zn 11.89; values µg/g. Not certified: 17 additional constituents (see certificate).		
l633a Trace Elements in Coal Fly Ash	Obtained from a coal fired power plant using Pennsylvania and West Virginia Coal. The ash was sieved through a No. 170 sieve.	Determined on 250 mg or more sample dried to constant weight. Al 14.3; Ca 1.11; Fe 9.40; K 1.88; Mg 0.455; Na 0.17; Si 22.8; wt. %. Sb 6.8; As 145; Cd 1.0; Cr 196; Cu 118; Hg 0.16; Ni 127; Pb 72.4; Rb 131; Se 10.3; Sr 830; Th 24.7; Tl 5.7; U 10.2; V 2.97; Zn 220; µg/g. Addi- tional 15 elements determined but not certified.		
l634a Trace Elements in Fuel Oil	Commerical No. 6 residual fuel oil as defined by ASTM.	Determined on at least 1 g sample. Pb 2.80; Mn 0.19; Ni 29; Se 0.15; Na 87; V 56; Zn 2.7; $\mu$ g/g. S 2.85 wt. %. Values for additional 11 ele- ments and 4 physical proper- ties are given but not certified.		
1635 Trace Elements in Coal (Subbituminous)	Subbituminous coal from Eagle Mine of the Imperial Coal Co. of Erie, Colorado; sieved through a No. 65 sieve.	Determined on at least 250 mg of dried sample. As 0.42; Cd 0.03; Cr 2.5; Cu 3.6; Pb 1.9; Mn 21.4; Ni 1.74; Se 0.9; Th 0.62; U 0.24; V 5.2; Zn 4.7; µg/g; and Fe 0.239; S 0.33 wt %. Additional 10 elements determined but not certified.		
1636a, 1637a, 1638a Lead in Reference Fuel	Supplied by Phillips Petroleum Co., Bartlesville, Okla. Lead was added as tetraethyl lead motor mix.	Determined on at least l g sample. Vial I-ll.2; Vial II-l8.8; Vial III-25.1; Vial IV-764 µg/g.		

Certification	Use	Pemark		
Analyses performed in the NBS Center for Analytical Chemistry. Estimated uncertainty 0.006 depending on constituent.	For the calibration of appa- ratus and the evaluation of techniques employed in the analysis of coal or similar materials.	Should be kept in its original bottle. Should not be exposed to intense sources of radia- tion, including ultraviolet lamps or sunlight.		
Using two to four different analytical procedures for each element as shown in the Certifi- cate, on the dried sample.	For calibration of apparatus and methods used in the analysis of coal ash and similar materials.	The materials should be dried as indicated in the Certifi- cate. Stability (>3 years) not yet established.		
Using two to three independent analytical procedures for each element.	For calibration of apparatus and methods used in the analysis of fuel oils and similar materials for trace elements.	Store in tightly sealed bottle. Certificate valid for 3 years from date of purchase.		
Using two to three independent	For calibration of apparatus and	Store in tightly sealed		

element. For sample drying, see the Certificate.

analytical procedures for each techniques used in trace element bottle in a cool, dark place. analysis of coal and similar materials.

Long term (>1 year) stability not yet established.

By isotope dilution mass spectrometry. SRM 1636a is made from Vials I, II, III, and IV. SRM 1637a is made from Vials I, II, and III. SRM 1638a is made from Vial IV.

For calibration of instruments and techniques used for the analysis of Pb in gasoline.

The vials should be stored at 10-30 °C in darkness, and not reused after first opening.

#### Table 2. - Continued.

SRM	Material	Composition		
l64lb Mercury in Water - μg/mL	This SRM was prepared at NBS and is delivered in 6 ampoules of 20 mL each.	Hg: 1.52 <u>+</u> 0.04 µ/mL.		
1642b Mercury in Water — ng/mL	Same as SRM 1641b.	Hg: 1.49 <u>+</u> 0.06 ng/mL.		
l643b Trace Elements in Water	Prepared at the U.S. Geological Survey, National Water Quality Laboratory, Arvada, Colo., by using high-purity reagents and sterilization.	Ba 44; Be 19; Cd 20; Cr 18.6; Co 26; Cu 21.9; Fe 99; Pb 23.7; Mn 28; Mo 85; Ni 49; Se 9.7; Ag 9.8; Sr 227; Tl 8; V 45.2; Zn 66 ng/g. Additional 3 elements determined but not certified.		
1644 Generator Columns for Polynuclear Aromatic Hydrocarbons	Three 50 cm x 0.6 cm coiled stainless steel tubes each packed with fine quintus quartz coated with 0.5 wt. % of polynuclear aromatic hydrocarbon (PAH).	At 20 °C: anthracene 30.7; benzo(a)anthracene 6.45; benzo(a)pyrene 1.13, all in µg/Kg.		
1645 River Sediment	Deposit dredged from the bottom of Indiana Harbor Canal, Gary, Ind., freeze-dried, sieved (No. 80 screen) and radiation- sterilized.	Al 2.26; Cr 2.96; Fe 11.3; K 1.26; Mg 0.74; Na 0.54; Zn 0.172, wt %. Cd 10.2; Cu 109; Co 10.1; Pb 714; Mn 785; Hg 1.1; Ni 45.8; Tl 1.44; Th 1.62; U 1.11; V 23.5; µg/g. Additional 13 values deter- mined but not certified.		
1646 Estuarine Sediment	Dredged from the Chesapeake Bay, freeze-dried, radiation- sterilized and sieved (No. 100 sieve).	Al 6.25; Ca 0.83; Fe 3.35; Mg 1.09; P 0.054, wt. %. As 11.6; Cd 0.36; Cr 76; Co 10.5; Cu 18; Pb 28.2; Mn 375; Hg 0.063; Ni 32; V 94; Zn 138, µg/g.		
1648 Urban Particulate Matter	Urban particulate matter was collected in St. Louis, Mo., over a period of 12 months.	Al 3.42; Fe 3.91; K 1.05; Pb 0.655; Na 0.425; Zn 0.476, wt. %. As 115; Cd 75; Cr 403; Cu 609; Ni 82; Se 27; U 5.5; V 140 μg/g. Non-certified values are given for 26 elements.		

Certification	Use	Remarks		
By atomic absorption spec- trometry and neutron activation. Use of blank samples is necessary.	For primary calibration of instruments and techniques and as a spike sample in method-of- addition procedures for the determination of Hg in natural waters.	Stability limited to l year from date of purchase.		
Same as SRM 1641b.	Same as SRM 1641b.	Should be used without dilution. For precautions in use see Certificate.		
At least two from the nine analytical procedures employed were used for the determination of each element.	For evaluating the accuracy of trace element determination in fresh water and for instrument calibration.	The certification is valid for two years from the shipping date. For pre- cautions in use see Certificate.		
Performed by two independent analytical procedures. Con- centrations for the three PAH at other temperatures (10- 30 °C) are given in the Certificate.	This SRM is intended to provide accurate concentrations of the three PAH.	For further properties, use, and stability, see Certifi- cate.		
Six indepedent analytical pro- cedures and 100 mg to 1 g of sample were used for certifica- tion.	For calibration of apparatus and methods used in the analysis of river sediments or similar materials.	For details on stability, use, and homogeneity, see Certifi- cate. Data valid for 5 years from date of purchase.		
By 6 indepedent analytical procedures on 500 mg or more dried sample, using two to four different procedures for each element.	For calibration of instrumen- tation and evaluatin of analytical methods in sediments and similar matrices.	Certified data valid for 5 years after date of shipping.		
On 100 mg or more of dried sample using 9 independent analytical procedures.	Calibration of apparatus and evaluation of methods used in the analysis of atmospheric particulate matter and similar matrices.	Certification valid for 5 years from date of purchase, for samples kept in the original bottle at 10-30 °C in a desiccator and in dark.		

#### Analyzed Liquids and Solids

#### MULTI-ELEMENT

Concentrations:

Values expressed as microgram per gram, except: Weight Percent -- % Nanogram per gram -- *italics* 

Parenthesis indicates elements not certified and given for information only.

SRM	Туре	Unit Size	A1	Sb	As	Ва
1632b	Trace Elements in Coal					
	(Bituminous)	75 g	0.855%	(0.24)	3.72	67.5
1633a	Trace Elements in Coal	0				
	Fly Ash	75 g	14.3%	6.8	145	(0.15%)
1634a	Trace Elements in Fuel Oil	100 mL			(0.12)	
1635	Trace Elements in Coal					
	(Subbituminous)	75 g	(0.32%)	(0.14)	0.42	
1643Ъ	Trace Elements in Water					
	(ng/g)	950 mL			(49)	44
1645	River Sediment	70 g	2.26%	(51)	(66)	
1646	Estuarine Sediment	75 g	6.25%	(0.4)	11.6	
1648	Urban Particulate	2 g	3.42%	(45)	115	(737)

SRM	Ве	Bi	В	Br	Cd	Ca	С	Се
1632b				(0, 17)	0 0573	0.204%	78 117	(9)
1633a	(12)				1.00	1.11%		(180)
1634a	(0.006)			(<1)	(0.002)	(16)		
1635					0.03			(3.6)
1643b	19	(11)	(94)		20			
1645					10.2	(2.9%)		
1646	(1.5)				0.36	0.83%		(80)
1648				(500)	75			(55)

SRM	Cs	C1	Cr	Со	Cu	Eu	F	
1632Ъ	(0.44)	(1260)	(11)	2.29	6.28	(0.17)		
1633a	(11)		196	(46)	118	(4)		
1634a		(31)	(0.7)	(0.3)				
1635			2.5	(0.65)	3.6	(0.06)		
1643b			18.6	26	21.9			
1645			2.96%	10.1	109		(0.09%)	
1646	(3.7)		76	10.5	18	(1.5)		
1648	(3)	(0.45%)	403	(18)	609	(0.8)		

SRM	Ga	Ge	Hf	Н	In	I	Fe	La	
1632Ъ			(0.43)	5.07%			0.759%	(5.1)	
1633a	(58)		(8)				9.4%		
1634a							(31)		
1635	(1.05)		(0.29)				0.239%		
1643Ъ							99		
1645							11.3%	(9)	
1646		(1.4)					3.35%		
1648			(4.4)		(1.0)	(20)	3.91%	(42)	

SRM	Pb	Li	Mg	Mn	Hg	Мо	Ni	N
1632b	3.67	(10)	0 0383%	12 4		(0,9)	6 10	1 56
1633a	72.4	(10)	0.455%	179	0.16	(29)	127	1.50
1634a	2 80			0 19	(< 0, 002)	(2)	29	
1635	1 9			21 4	(<0.002)	(0.12)	1 74	
16/3b	23 7			21.4		05	1.74	
1645	71/		0.74%	785	1 1	00	49 45 Q	
1646	28 2	(49)	1 00%	275	1.1	(2,0)	40.0	
1649	0.655%	(49)	(0.9%)	(960)	0.003	(2.0)	24	
	0.055%		(0.0%)	(800)			02	
SRM	P	K	Rb	Sm	Sc	Se	Si	Ag
16325		0 07/8%	5 05	(0, 87)	(1, 0)	1 29	(1, 1, 7)	
16332		1 999	121	(0.07)	(1, y)	10 3	(1.4%)	
163/2		1.00%	131		(40)	0.15	22.0%	
1625					(0 (2))	0.15		
16/21					(0.03)	0.9		
16450		1 0 ( 9/			(2)	9./ (1 E)		
1045		1.20%			(2)	(1, 5)	(019)	
1640	0.054%	(1.4%)	(87)		(10.8)	(0.0)	(31%)	
		1.05%	(32)	(4.4)	(7)	27		(6)
SRM	Na	Sr	S	Те	Tl	Th	Ti	W
1632Ъ	0.0515%	(102)	1.89%			1.342	0.0454%	(0.48)
1633a	0.17%	830	(0.18%)		5.7	24.7	(0.8%)	
16 <b>3</b> 4a	87		2.85%					
1635	(0.24%)		0.33%			0.62	(0.02%)	
1643b	/	227			8.0			
1645	0.54%		(1.1%)		1.44	1.62		
1646	(2.0%)		(0.96%)	(0.5)	(0.5)	(10)	(0.51%)	
1648	0.425%		(5.0%)	/		(7.4)	(0.40%)	(4.8)

SRM	U	V	Zn
1632Ъ	0.436	(14)	11.89
1633a	10.2	297	220
1634a		56	2.7
1635	0.24	5.2	4.7
1643b		45.2	66
1645	1.11	23.5	0.172%
1646		94	138
1648	5.5	140	0.476%

## Analyzed Liquids and Solids

#### SINGLE ELEMENT

SRM	Туре	Unit Size	Lead	Sulfur	Mercury
1579	Powdered Lead Base Paint	35 g	11.87%		
1620a	Sulfur in Residual Fuel Oil	100 mL		4.504%	
1621b	Sulfur in Residual Fuel Oil	100 mL		0.950%	
1622Ъ	Sulfur in Residual Fuel Oil	100 mL		1.982%	
1623a	Sulfur in Residual Fuel Oil	100 mL		0.240%	
1624a	Sulfur in Residual Fuel Oil	100 mL		0.141%	
1630	Trace Mercury in Coal	50 mg			0.13 µg/g
1641b	Mercury in Water - µg/mL	120 mL			1.52 µg/mL
1642b	Mercury in Water - ng/mL	950 mL			1.49 ng/mL

		•		
		Element	Nominal	
SRM	Туре	Certified	Concentration	No. Units
1636a	Lead in Reference Fuel	РЬ	0.03, 0.05, 0.07, and	3 vials each
			2.0 g/gal	
1637a	Lead in Reference Fuel	Pb	0.03, 0.05, 0.07 g/gal	4 vials each
1638a	Lead in Reference Eucl	РЪ	2.0 g/gal	12 vials
10000	Bedd in Reference ruci	10	2:0 6/641	it vidio

## Organic Constituents

SRM	Туре	Unit of Issue
1580 1644	Shale Oil Polyaromatic Hydrocarbon Generator Columns	Set of 5-2 mL ampoules Set of 3 columns

	SRM 1580	SRM 1644
Constituents	(µg/g)	(µg/kg)
Anthracene		30.7
Benzo[a]anthracene		6.45
Benzo[a]pyrene	21	1.13
Benzo[e]pyrene	18	
Fluoranthene	54	
o-Cresol	385	
Phenol	407	
Perylene	3.4	
Pyrene	104	
2,6-Dimethylphenol	175	
Benzo $[f]$ quinoline	16	

### Appendix I.

## Alphabetical Index by Standard Reference Material Name

Name	SRM
Acetanilide	141c
Acid Open-Hearth Steel, 0.2% Carbon	19G
Acid Potassium Phthalate	84i
AISI 1045 Steel	20g
AISI 4340 Steel	361
AISI 4340 Steel	1261a
AISI 94B17 Steel (Modified)	362
AISI 94B17 Steel (Modified)	1262a
Albacore Tuna	RM 50
Alkali Lead Silicate Glass	712
Alpha Quartz	1878
Alumina (Reduction Grade)	699
Alumina Silicate Glass	714
Aluminosilicate Glass	715
Aluminum Alloy	85B
Aluminum Alioy 6011 (Modified)	858
Aluminum Alloy 6011 (Modified)	1258
Aluminum Alloy 7075	859
Aluminum Alloy 7075	1259
Aluminum Block, Eddy Current	1860
Conductivity	
Aluminum Block, Eddy Current	1861
Conductivity	
Aluminum Block, Eddy Current	1862
Conductivity	
Aluminum Block, Eddy Current	1863
Conductivity	
Aluminum Brass Standard for	1118
Optical Emission and X-ray	
Spectroscopic Analysis	<b>G</b> 1110
Aluminum Brass Standard for	CIII8
Optical Emission and X-ray	
Spectroscopic Analysis	1110
Aluminum Brass Standard for	1119
Optical Emission and X-ray	
Spectroscopic Analysis	C1110
Aluminum Brass Standard for	CIII9
Spectroscopic A polycic	
Aluminum Casting Alloy 356	855
Aluminum Casting Alloy 380	856
Aluminum Cube Illtra Purity	RM 10
Aluminum 2. Ethylbexanoate	1075a
Anuminum 2-Emymeranoate	10/54

Name	SKW
Aluminum, Freezing Point Standard Aluminum, Magnetic Gram Suscentibility	44f 763
Aluminum Oxide Melting Point	742
Aluminum Rod Ultra Purity	RM 1R
Aluminum 76 Radioactivity Standard	4229
Americium-241 Alpha-Particle	4904F
Standard	
Americium-241 Gamma-ray Standard	4213
Ammonium Dihydrogen Phosphate	194
Angiotensin I (Human)	998
Anisic Acid	142
Anticonvulsant Drug Level Assay	1599
Standard	
Antiepilepsy Drug Level Assay	900
Standard	
Antimony-125-Tellurium-125m,	4275B
Europium-154, Europium-155 Mixed-	
Radionuclide Point-Source Standard	
Antimony-125-Tellurium-125m,	4276B
Europium-154, Europium-155 Mixed-	
Radionuclide Solution Standard	
A.O.H., 0.4C Spectrographic Steel	413
Standard	
Argillaceous Limestone	1C
Arsenic Trioxide Reductometric	83d
Standard	0.05
Assay-Isotopic Standard for Potassium	985
Assay-Isotopic Standard for Knenium	989
Assay-Isotopic Standard for Stillcon	990
Assay-isotopic Standard for Strontium	987
2% Austenite in Ferrite	488
15% Austenite in Ferrite	485a 194
30% Austenite in Ferrite	400
JU70 Austennie in Felffie	40/

CDN

Name	SRM
Austenitic Stainless Steel, Thermal Conductivity and Electrical Resistivity	1460
Austenitic Stainless Steel, Thermal Conductivity and Electrical Resistivity	1461
Austenitic Stainless Steel, Thermal Conductivity and Electrical Resistivity	1462
Barium Crown Glass	713
Barium Cyclohexanebutyrate	1051b
Barrium-133 Radioactivity Point-Source Standard	4241B
Barium-133 Radioactivity Standard	4251B
Basalt Rock	688
Base Oil	1083
Basic Electric Spectrographic Steel Standard	404a
Basic Open-Hearth Steel, 0.1% Carbon	15g
Basic Open-Hearth Steel, 0.1% Carbon	335
Basic Open-Hearth Steel, 0.1% Carbon	1228
Basic Open-Hearth Steel, 0.2% Carbon	11h
Basic Open-Hearth Steel, 0.4% Carbon	12H
Basic Open-Hearth Steel, 0.5% Carbon	152A
Basic Open-Hearth Steel, 0.8% Carbon	14f
Basic Open-Hearth Steel, 1% Carbon (Disk)	1227
Basic Open-Hearth Steel, 1.1% Carbon	16f
Basic Open-Hearth Steel, 1.1% Carbon	337
0.4C Basic Oxygen Furnace Steel	178
Bauxite (Arkansas)	69b
Bauxite (Dominican)	697
Bauxite (Jamaican)	698
Bauxite (Surinam)	696
Benzene in Nitrogen	1805
Benzene in Nitrogen	1806
Benzene Permeation Device	1911
Benzoic Acid	140b
Benzoic Acid	350a
Benzoic Acid Calorimetric Standard	39i
Benzothiazyl Disulfide Rubber Compound	373f
Beryllium-Copper Standard	1122
Beryllium-Copper Standard	C1122
Beryllium-Copper Standard	C1123

Name	SRM
Parullium on Filter Madia	2675
Berymuni on Finer Media Bessemer Steel (Simulated)	2075
0.1% Carbon	0]
Biligubin	916
Bis(1 phenyl 1 3 butanediono)	1080a
copper (II)	10004
Bis(1, phenyl-1, 3, butanediono)	1052h
oxovanadium (IV)	10520
Black Porcelain Enamel for Directional	2021
Hemispherical Reflectance	2021
Black Porcelain Enamel for Directional	2022
Hemispherical Reflectance	2022
Blast Eurnace Iron Standard	1143a
(Chill Cast White)	
Blast Eurnace Iron Standard	1144a
(Chill Cast White)	
B O H., 0 4C Spectrographic Steel	417a
Standard	
Boric Acid	951
Boron-Doped Silicon Slices for	1521
Resistivity Measurements	
Borosilicate Glass	93a
Borosilicate Glass	623
Borosilicate Glass	717
Borosilicate Glass	1825
Borosilicate Glass, Thermal Expansion	731
Bovine Liver	1577a
Bovine Serum Albumin	926
Bovine Serum Albumin (7% Solution)	927
Branched Polyethylene	1476
Brewers Yeast	1569
Bright Copper Microhardness	1894
Standard	
Bright Nickel Microhardness Standard	1895
Bromobenzoic Acid	2142
Burnt Refractory	76a
Burnt Refractory	77a
Burnt Refractory	78a
Cadmium Cyclohexanebutyrate	1053a
Cadmium, Vapor Pressure	746
Calcium Carbonate	915
Calcium 2-Ethylhexanoate	1074a
Calcium in Low-Alloy (Silicon) Steel	1254
Calcium Molybdate	71
Calibrated Glass Beads	1004
Calibrated Glass Beads	101/a
Calibrated Glass Beads	1018a
Calibrated Glass Spheres	1670
Carbon Dioxide in Air	1671
Carbon Dioxide in Air	1672
Carbon Dioxide in Nitrogen	1674h
Carbon Dioxide in Nitrogen	1675b
Carbon Dioxide in Nitrogen	2619a
(Combustion Efficiency Gas Standard)	20174
Carbon Dioxide in Nitrogen	2620a
(Combustion Efficiency Gas Standard)	

Name	SRM
Carbon Dioxide in Nitrogen	2621a
(Combustion Efficiency Gas Standard)	
Carbon Dioxide in Nitrogen	2622a
(Combustion Efficiency Gas Standard)	
Carbon Dioxide in Nitrogen	2623a
(Combustion Efficiency Gas Standard)	26240
(Combustion Efficiency Gas Standard)	2024a
Carbon Dioxide in Nitrogen	2625a
(Combustion Efficiency Gas Standard)	Louid
Carbon Dioxide in Nitrogen	2626a
(Combustion Efficiency Gas Standard)	
Carbon Dioxide in Nitrogen (Mobile	2632
Source Emission Gas Standard)	2(22
Carbon Dioxide in Nitrogen (Mobile	2633
Source Emission Gas Standard)	26120
Air Quality Gas Standard)	2012a
Carbon Monoxide in Air (Ambient	2613a
Air Quality Gas Standard)	
Carbon Monoxide in Air (Ambient	2614a
Air Quality Gas Standard)	
Carbon Monoxide in Nitrogen	1677c
Carbon Monoxide in Nitrogen	1678c
Carbon Monoxide in Nitrogen	16/90
Carbon Monoxide in Nitrogen	1680b
Carbon Monoxide in Nitrogen (Mobile	1681b
Source Emission Gas Standard)	2035
Carbon Monoxide in Nitrogen (Mobile	2636
Source Emission Gas Standard)	2050
Carbon Monoxide in Nitrogen (Mobile	2637
Source Emission Gas Standard)	
Carbon Monoxide in Nitrogen (Mobile	2638
Source Emission Gas Standard)	
Carbon Monoxide in Nitrogen (Mobile	2639
Carbon Monovide in Nitrogen (Mobile	2640
Source Emission Gas Standard)	2640
Carbon Monoxide in Nitrogen (Mobile	2641
Source Emission Gas Standard)	2011
Carbon Monoxide in Nitrogen (Mobile	2642
Source Emission Gas Standard)	
Carbon-14 Radioactivity Standard	4245
Carbon-14 Radioactivity Standard	4246
Carbon Steel	1224
Cast Iron	13g
Cast Iron	4K 51
Cast Iron	6g
Cast Iron	7G
Cast Iron Car Wheel	122h
Cast Steel 3	C1173
Cast Steel Standard	1138a
Cast Steel Standard	1139a

Name	SRM
Catalyst Package for Lubricant	1817
Oxidation	
Centerline Drawings for Optical	1901
Character Recognition, B	
Characters	
Centroid Color Chart	2106
Centroid Color Kit	2107
Cesium-137, Barium-137m Point-Source	4200B
Radioactivity Standard	4207
Cesium-137, Barium-137m Point-Source	4207
Coolum 127 Pure Un Stondard	42220
Cesium 134 Padioactivity Standard	4255D 4250B
Channel Black Rubber Compound	3750
Chlorine 36 Beta-ray Standard	4943
Chlorine-36 Radioactivity Standard	44221
Chlorobenzoic Acid	2144
Chrome Refractory	103a
Chromium-Molybdenum-Aluminum	106B
Steel	
Chromium-Molybdenum Steel	36b
Chromium-Molybdenum Steel	133B
Chromium-Nickel-Molybdenum Steel	139ь
Chromium-Nickel-Molybdenum Steel	1222
17Chromium-9 Nickel-0.2 Selenium Steel	339
Chromium-Nickel Spectrographic Steel Standard	408a
15Chromium-7 Nickel Steel	344
16 Chromium-4 Nickel Steel	345
Chromium-51 Radioactivity Standard	4400L-F
Chromium Steel	163
Chromium-Tungsten Steel	155
Chromium-Vanadium Spectrographic	407a
Steel Standard	
Cholesterol	911a
Chrysotile Asbestos Fibers	1876
Citrus Leaves	1572
Clinical Laboratory Thermometer	934
Cobalt Cyclohexanebutyrate	1055b
Cobalt-Molybdenum-Tungsten Steel	153A
Cobalt-57 Radioactivity Standard	4408L-C
Cobalt-60 Radioactivity Standard	4915D
Commerical Bronze Standard for	1115
Optical Emission and X-ray	
Spectroscopic Analysis	C1115
Commercial Bronze Standard for	CIIIS
Spectroscopic A palusis	
Commercial Bronze Standard for	1116
Ontical Emission and X-ray	1110
Spectroscopic Analysis	
opeen oscopie / marysis	

Name	SRM	Name	SRM
Commercial Bronze Standard for Optical Emission and X-ray	C1116	Cupro-Nickel, 1 <mark>0%</mark> (CDA 706) High Purity	874
Spectroscopic Analysis		Cystine	143c
Commercial Bronze Standard for	1117	Dextrose	41b
Optical Emission and X-ray		D-Glucose	917
Spectroscopic Analysis		Dibutyltin Bis(2-ethylhexanoate)	1057b
Commercial Bronze Standard for	C1117	Didymium Glass Filter for Checking	2009
Optical Emission and X-ray		the Wavelength Scale of	
Spectroscopic Analysis		Spectrophotometers	
Common Lead Isotopic Standard	981	Didymium Glass Fitler for Checking	2010
Copper Concentrate	332	the Wavelength Scale of	
Copper Heat Capacity Test Specimen	RM5	Spectrophotometers	
Copper-Nickel-Chromium Cast Iron	115A	Disodium Hydrogen Phosphate	186IIc
Copper Ore, Mill Heads	330	Disodium Hydrogen Phosphate	2186II
Copper Ore, Mill Tails	331	D-Mannitol	920
Copper-Thermal Expansion	736a	Dolomitic Limestone	88a
Copper. Secondary Freezing Point	45d	Doped Platinum	681L1
Standard	154	Doped Platinum	6811.2
Cortisol (Hydrocortisone)	921	Ductile Cast Iron	341
Creatinine	914	Electrical Residual Resistivity Ratio	769
Cr-Mo Low Alloy Steel	1270	Standard	105
Cr-Mo Steel (ASTM A-213)	291	Electrolytic Iron	365
Cr-Mo (SAE 4140) Spectrographic	414	Electrolytic Iron	1265a
Steel Standard	717	Electrolytic Iron Thermal	1463
Cr-Mo (SAE 4150) Spectrographic	427	Conductivity and Electrical	1405
Steel Standard	721	Resistivity	
Cr-Mo (SAE X4130) Spectrographic	4182	Electrolytic Iron Thermal	1464
Steel Standard	410a	Conductivity and Electrical	1404
Cr.Ni-Mo Steel (AISI 8620)	203	Resistivity	
18Cr-10Ni Steel (AISI 304L)	101f	Electronic and Magnetic Alloy	1150
Cr.V Steel (Modified)	363	Standard	1159
Cr.V Steel (Modified)	12630	Electronic and Magnetic Alloy	1160
$Cr_V$ Steel (SAE 6150)	206	Standard	1100
Crystalline Potassium Dichromate	025	Enriched Boric Acid	052
Crystalline Potassium Iodide	2022	Equal Atom Lead Isotopic Standard	932
Heterochromatic Stray Radiant	2032	Equal-Atom Lead Isotopic Standard	1646
Energy Standard		Europium 152 Point Source Stendard	42195
Crystalline (Ruby) Electron	2601	Europium 152 Padioactivity Standard	4210E
Paramagnetic Resonance	2001	Europhini-152 Radioactivity Standard	4370B
Absorption Intensity Standard		Extra Dense Leau Olass Ea Cr Ni Alloy Microproba Standard	/09
Cupro Nickel (CDA 706)	1275	Fe 3Si Allou Microprobe Standard	4/98
Cupro Nickel (CDA 700)	1275	Foldspor	403
Cupro Nickel 10% (CDA 706) Doped	1270	Feldspar	70a
Cupio-ruckei, 1070 (CDA 700) Doped	610	Forrochromium (Low Corbor)	99a
		Ferrochromium Silicon	190
		Ferroniobium	089
		Ferrenhoenhorus	340
		Ferrecilicon	90
		Ferrosincon	58a

Ferrosilicon

Ferrosilicon (75% Si) First Surface Aluminum Mirror for

Specular Reflectance First Surface Mirror, Gold on Glass

59a

195 2003a

2008a

Name	SRM
Fission Track Glass Standard	961
Fission Track Glass Standard	962a
Fission Track Glass Standard	963a
Fission Track Glass Standard	964
Flint Clay	97a
Fluorobenzoic Acid	2143
Fluorspar	79a
Free-Cutting Brass	1103
Free-Cutting Brass	C1104
Freeze-Dried Urine	2670
Freeze-Dried Urine Certified	2671a
for Fluoride	
Freeze-Dried Urine Certified	2672a
for Mercury	
Fused-Silica Thermal Expansion	739
Gadolinium-148 Alpha-Particle	4907
Standard Gallium Melting Point Standard	1068
Gallium 67 Padioactivity Standard	4416L D
Gas Europas Black Pubbar Compound	2820
Gasometrie Set (1095-1099)	1080
Gasometric Standard for Unalloyed	357
Zirconium	557
Gasometric Standard for Unalloyed	358
Zirconium	550
Generator Columns for Polynuclear	1644
Aromatic Hydrocarbons	
Gilding Metal	1112
Gilding Metal	C1112
Gilding Metal	1113
Gilding Metal	C1113
Gilding Metal	1114
Gilding Metal	C1114
Glasses for Microchemical Analysis	1871
Glasses for Microchemical Analysis	1872
Glasses for Microchemical Analysis	1873
Glasses for Microchemical Analysis	1874
Glasses for Microchemical Analysis	1875
Glass Fibers for Microanalysis	RM 31
Glass Filter for Transmittance	2030
Measurement	
Glass Filters for Spectrophotometry	930D
Glass Fluorescence Source	477
Glass Sand	81a
Glass Sand	165a
Glass Spheres	1019a
Gold Coating on Glass Sealing Alloy	1398a
Gold Coating on Nickel	1379
Gold Coating on Nickel	1380
Gold Coating on Nickel	13996
A polycic	482
Gold-195 Radioactivity Standard	44211
Our 175 Radioactivity Standard	11211

Name	SRM
Gold-198 Radioactivity Standard	4405L-B
A nalysis	481
Gold Vapor Pressure	745
Grav Cast Iron	334
Halocarbons (in methanol) for Water	1639
Analysis	1057
High-Allov Steel (A-743)	C1288
High-Allov Steel (AISI 310 Mod.)	C1287
High-Allov Steel, (AISI 414 Mod.)	C1289
High-Alloy White Cast	892
High-Alloy White Cast Iron	890
High-Alloy White Cast Iron	891
High-Carbon Ferrochromium	64c
High-Carbon Ferromanganese	68c
High-Carbon Steel (Modified)	364
High-Carbon Steel (Modified)	1264a
High-Grade Fluorspar	180
High-Nickel Steel	126c
High-Nickel Steel	1158
High-Purity Gold	685
High-Purity Platinum	680L1A
High-Purity Platinum	680L2A
High-Purity Platinum Thermoelement	1967
High-Purity Zinc	682
High-Silicon Steel	179
High-Silicon Steel	1134
High-Silicon Steel	1135
High-Silicon Steel (Calcium Bearing)	1250
High-Sulfur Steel	105
High-Sulfur Steel	129C
High Temperature Allow A 286	249
High Temperature Alloy M308	240 1107
High Temperature Alloy I 605 and	S1100
S816	31177
High-Temperature Allov	1206-2
High-Temperature Alloy	1207-1
High-Temperature Alloy	1207-2
High-Temperature Alloy	1208-1
High-Temperature Alloy	1208-2
Homogeneous River Sediment for	RM 45B
Radioactivity Measurements	
Human Liver, Environmental	4352
Radioactivity	
Human Lung, Environmental	4351
Radioactivity	
Human Serum	909

Name	SRM
Hydrogen in Unalloyed Titanium	352b
Hydrogen in Unalloved Titanium	1086
Hydrogen in Unalloyed Titanium	1087
Hydrogen in Unalloved Titanium	1088
Hydrogen-3 Radioactivity Standard	4361
Hydrogen-3 Radioactivity Standard	4926C
Hydrogen-3 Toluene Radioactivity	4947
4-Hydroxy-3 methoxy-DL-mandelic	925
ICTA High Temperature Set	GM 760
ICTA Low Temperature Set Differen-	GM 757
ICTA Mod Temperature Set Differen-	GM 759
tial Thermal Analysis ICTA Mid Temperature Set Differen-	GM 758
tial Thermal Analysis ICTA Polystyrene Differential	GM 754
Thermal Analysis	GM 761
Incolov 901 and Hastellov X	\$1198
Inconels Alloy 600 (Chips)	864
Incouels, Alloy 600 (Solid)	1244
Inconels, Alloy 625 (Chips)	865
Inconels, Alloy 625 (Solid)	1245
Incolov Alloy 800 (Chips)	866
Incoloy, Alloy 800 (Solid)	1246
Incoloy, Alloy 825 (Chips)	867
Incoloy, Alloy 825 (Solid)	1247
Indium-111 Radioactivity Standard	4417L-C
Ingot Iron Spectrographic Steel	420a
Standard	
Intermediate Purity Selenium	726
Intermediate-Purity Zinc	728
Iodine-123 Radioactivity Standard	4414L-C
Iodine-125 Radiactivity Standard	4407L-H
Iodine-129 Radioactivity Standard	4949B
Iodine-131 Radioactivity Standard	4401L-I
Iron Foil Mössbauer Standard	1541
Iron-55 Low-Energy Photon Standard	4260C
Iron Metal (Clinical Standard)	937
Iron Ore (Labrador)	692
Iron Ore (Nimba)	693

Name	SRM
Iron Ore (Sibley)	27f
Iron Ore Concentrate (Canada)	690
Iron-59 Radioactivity Standard	4411L-B
Isobutylene-Isoprene (Butyl) Rubber	1495
Isobutylene-Isoprene (Butyl) Rubber	388L
Isotopic Standard for Bromine	977
Isotopic Standard for Chlorine	975
Isotopic Standard for Chromium	979
Isotopic Standard for Copper	976
Isotopic Standard for Magnesium	980
Isotopic Standard for Silver	978
Standard Standard	4308C
Krypton-85 Radioactivity Standard	4235
Krypton-85 Radioactivity Standard	4935C
Lead-Barium Glass	89
Lead-Base Bearing Metal	53e
Lead-Base Bearing Metal	1132
Lead Cyclohexanebutyrate	1059c
Lezd in Reference Fuel	1636a
Lead in Reference Fuel	1637a
Lead in Reference Fuel	1638a
Lead Nitrate	928
Lead on Filler Media	2074
Lead Secondary Freezing Point	4420L
Standard	490
Lead Silica Glass	1827
Lead-Silica Glass (Viscosity)	711
Lead-Silica Glass for dc Volume	624
Resistivity	
Lead-Silica Glass for Dielectric	774
Constant	
Lead 206 Spike Assay and Isotopic	991
Solution Standard	
Leaded-Tin Bronze Alloy	1035
Light-Sensitive Paper	700d
Light-Sensitive Paper	/01d
Light-Sensitive Plastic Chip	103
Linear Polyethylene	14/5
Linear Polyethylene	1402
Linear Polyethylene	1405
Linear Polyethylene	1810
Adhesion Testing	1010
Liquid Absorbance Standard for	931c
Ultraviolet and Visible	
Spectrophotometry	
Lithium Carbonate	924
Lithium Ore	181
Lithium Ore	182
Lithium Ore	183
Low-Alloy Steel, (AISI 4130)	1225
Low Alloy Steel	1226
Low Alloy Steel (A242 Mod.)	C1285
Low-Alloy Steel, AISI 4130	72g
Low Alloy Steel (AISI 1526, Modified)	1269
Low-Alloy Steel (Hy 80)	1286

Name	SRM
Low Allow Steel Set (((1 ((5)	5(()
Low-Alloy Steel Set (001-005)	3008 1210
Low-Carbon Silicon Steel	131C 1036
Low Carbon Stainless Steel (AISI	1660
216L)	1000
Magnasium base Alleu	171
Magnesium Cycloberenebuturate	10610
Magnesium Cluconeta Dibudrete	020
Magnetic Costing on Magnetic	929 1365a
Substrate (Nickel on Steel)	1505a
Magnetic Coating on Magnetic	13662
Substrate (Nickel on Steel)	1500a
Magnetic Coating on Non-Magnetic	1367a
Substrate (Nickel and Chromium	15074
on brass	
Magnetic Tane High Density	6250
Magnetic Tupe, High Density Manganese Eluoride Magnetic Gram	766
Suscentibility	,00
Manganese Ore	25d
Manganese-54 Point-Source	4997E
Radioactivity Standard	
Manganese-54 Radioactivity Standard	4257
Manganese Steel	100B
Manganous Cyclohexanebutyrate	1062b
Maraging Steel	1156
Metal on Quartz Filters for	2031
Spectrophotometry	2001
Metals on Filter Media	2676b
Methane in Air	1658a
Methane in Air	1659a
Methane in Air	1660a
Medium Manganese Spectrographic	405a
Steel Standard	
Mercaptobenzothiazole	383a
Mercury, Freezing Point	743
Mercury-203 Radioactivity Standard	4418L
Mercury in Water, µg/mL	164lb
Mercury in Water, ng/mL	1642b
Microcopy Resolution Test Chart	1010a
Microprobe Standard - Cartridge Brass	478
Mineral Glasses for Microanalysis	470
Molybdenum Concentrate	333
Molybdenum, Heat Capacity	781
Molybdenum-99 Radioactivity	4412L-H
Standard	
Molybdenum-Tungsten-Chromium-	134A
Vanadium Steel	
Naval Brass Standards for Optical	1106
Emission and Spectroscopic	
Analysis	
Naval Brass Standards for Optical	C1106
Emission and Spectroscopic	
Analysis	1107
Naval Brass Standards for Optical	1107
Emission and Spectroscopic	
Analysis	C1107
Naval Brass Standards for Optical	CH0/
A polygio	
Analysis	

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Name	SRM
Naval Brass Standards for Optical Emission and Spectroscopic Analysis	1108
Naval Brass Standards for Optical Emission and Spectroscopic	C1108
Neutral Glass	716
Neutron Density Monitor Wire	953
Nickel-Chromium Cast Iron	82h
Nickel-Chromium-Molybdenum Cast Iron	107C
Nickel-Chromium Steel	32E
Nickel-Copper Alloy	882
Nickel Cyclohexanebutyrate	1065b
Nickel Oxide, No. 1	671
Nickel Oxide, No. 2	672
Nickel Oxide, No. 3	673
Nickel-63 Radioactivity Standard	4226
Nickel Silver (CDA 762)	879
Nickel Siver (CDA 770)	880
Nickel Spectrographic Steel Standard	409b
Nickel Sphere, Magnetic Moment	772
Nickel Steel	33d
Ni-Cr-Mo-V Steel	1173
Nicotinic Acid	148
Niobium-94 Gamma-ray Standard	4201B
Nitric Oxide in Nitrogen	1683b
Nitric Oxide in Nitrogen	1684b
Nitric Oxide in Nitrogen	1685b
Nitric Oxide in Nitrogen	1686b
Nitric Oxide in Nitrogen	1687b
Nitric Oxide in Nitrogen (Mobile	2627
Source Emission Gas Standard)	
Nitric Oxide in Nitrogen (Mobile	2628
Source Emission Gas Standard)	
Nitric Oxide in Nitrogen (Mobile	2629
Source Emission Gas Standard)	2620
Source Emission Gas Standard)	2030
Nitrie Ovide in Nitrogen (Mobile	2621
Source Emission Gas Standard)	2031
Nitrogen Diovide in Air (Stationary	2653
Source Emission Gas Standard)	2055
Nitrogen Dioxide in Air (Stationary	2654
Source Emission Gas Standard)	2000
Nitrogen Dioxide in Air (Stationary	2655
Source Emission Gas Standard)	
Nitrogen Dioxide in Air (Stationary Source Emission Gas Standard)	2656
Nitrogen Dioxide Permeation Device	1629a
4-Nitrophenol	938
A Contraction of the second se	

Name	SRM
Nodular Cast Iron	342a
Nominal One Micrometer Polystyrene Spheres	1690
Non-Fat Powdered Milk	1549
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1359
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1360
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	13611
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1362a
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1363a
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1364;
NPL GM Alpha Alumina	8005
NPL GM Alpha Alumina	8006
NPL GM Alpha Alumina	8007
NPL GM Alpha Alumina	8008
NPL GM Graphitized Carbon Black	8001
NPL GM Graphitized Carbon Black	8002
NPL GM Melting Point Set	8000
NPL GM Non-porous Silica	8003
NPL GM Non-porous Silica	8004
N-tertiary-Butyl-2-benzothiazolesulfen- amide Rubber Compound	384d
Obsidian Rock	278
Octaphenylcyclotetrasiloxane	1066
Oil Furnace Black Rubber Compound	3786
Opal Glass Powder	91
Spectroscopic Analysis	1102
Optical Microscope Linewidth Measurement Standard	474
Optical Microscope Linewidth Measurement Standard	475
Optical Microscope Linewidth Measurement Standard	476

Name	SRM
Organics in Shale Oil	1580
Oxalic Acid	4990C
Oxygen in Ferrous Materials	1090
Ingot Iron	
Oxygen in Ferrous Materials	1091
(Stainless Steel AISI 431)	
Oxygen in Ferrous Materials Vacuum	1092
Melted Steel	
Oxygen in Maraging Steel	1094
Oxygen in Nitrogen (Gas Standard)	2657
Oxygen in Nitrogen (Gas Standard)	2658
Oxygen in Titopium Pase Materials	2039
Oxygen in Value Steel	1003
Ovster Tissue	1566
Palladium, Magnetic Gram	765
Susceptibility	
Penetrant Test Block	1850
Peruvian Soil, Environmental	4355
Radioactivity	
Petroleum Crude Oil	1582
Phosphate Rock (Florida)	120b
Phosphor Bronze (CDA 521)	871
Phosphor Bronze (CDA 544)	872
Phosphorized Copper, Cu VIII	C1251 C1252
Phosphorized Copper, Cu X	C1252
Phosphorus 32 Radioactivity Standard	4406L G
Photographic Step Tablet	1008
Pine Needles	1575
Plastic Clav	98a
Platinum, Magnetic Gram	764
Susceptibility	
Plutonium-238 Alpha-Particle Standard	4906B
Plutonium-240 Alpha-Particle Emission-	4338
Rate Solution Standard	
Plutonium-239 Alpha-Particle Solution	4331
Standard	4324D
Plutonium-242 Alpha-Particle Solution	4334B
Standard Plutonium Isotonic Standard	046
Plutonium Isotopic Standard	940
Plutonium Isotopic Standard	948
Plutonium Metal	949f
Plutonium Metal (Standard Matrix	945
Material)	
Plutonium-244 Spike Assay and	996
Isotopic Standard	
Polychlorinated Biphenyls in Oil	1581
Polycrystalline Alumina Elasticity	718
Standard	1470
Polyester Plastic Film for Oxygen	1470
Gas Transmission	1400
Polystyrene	1490
Polystyrene	1479
Polystyrene (Broad Molecular Weight)	706
Polystyrene (Narrow Molecular	705
Weight)	
Polystyrene Spheres	1691
Portland Cement (Black)	1880

Name	SRM
Portland Coment (Blue)	635
Portland Coment (Clear)	620
Portland Coment (Cield)	634
Portland Coment (Green)	629
Portland Coment (Bink)	637
Portland Coment (Pad)	622
Portland Coment (White)	1001
Portland Coment (Valleur)	626
Portland Coment Finances Standard	114.
Potossium Chlorida	2202
Potassium Chlorida (Clinical Standard)	018
Potassium Chlorida (Primary	000
Chemical)	,,,,
Potassium Chloride for Solution	1655
Calorimetry	1055
Potassium Dichromata	136d
Potassium Dibydrogen Phoenbate	200
Potassium Dihydrogen Phosphate	186Ic
Potassium Dihydrogen Phosphate	21861
Potassium Erucate	1076
Potassium Feldspar	607
Potassium Fluoride	2203
Potassium Hydrogen Phthalate	1850
Potassium Hydrogen Tertrete	1850
Potassium Indide with Attenuator	2022
Potassium Nitrato	103
Potassium Tetrovalate	193
Powdered Lead Based Point	1570
Priority Pollutant Polypuoloor	1647
Aromatic Hudrocarbons (in	1047
Acetonitrile)	
Propaga in Air	1665b
Propane in Air	1666b
Propane in Air	1667b
Propane in Air	1668b
Propane in Air	16695
Propane in Nitrogen (Mobile Source	2643
Emission Gas Standard)	2045
Propage in Nitrogen (Mobile Source	2644
Emission Gas Standard)	2011
Propage in Nitrogen (Mobile Source	2645
Emission Gas Standard)	2015
Propage in Nitrogen (Mobile Source	2646
Emission Gas Standard)	2010
Propane in Nitrogen (Mobile Source	2647
Emission Gas Standard)	2017
Propage in Nitrogen (Mobile Source	2648
Emission Gas Standard)	2010
Propane in Nitrogen (Mobile Source	2649
Emission Gas Standard)	2017
Propane in Nitrogen (Mobile Source	2650
Emission Gas Standard)	2020
Propane in Nitrogen and Oxygen	2651
(Mobile Source Emission Gas	2001
Standard)	
Propane in Nitrogen and Oxygen	2652
(Mobile Source Emission Gas	
Standard)	
Quartz Cuvette for Spectrophotometry	932
Quartz for Hydrofluoric Acid	1654
Solution Calorimetry	

Name	SRM
Quartz on Filter Media	2679a
Ouinine Sulfate Dihydrate	936
Radiogenic Lead Isotopic Standard	983
Radium-226 Gamma-ray Standard	4956
Radium 226 Gamma ray Standard	4957
Radium 226 Gamma ray Standard	4958
Radium 226 Gamma ray Standard	4050
Radium 226 Gamma ray Standard	4960
Radium 226 Common row Standard	4900
Radium-226 Gamma-ray Standard	4901
Radium-220 Gamma-ray Standard	4902
Radium-226 Gamma-ray Standard	4903
Radium-226 Gamma-ray Standard	4904B
Radium Standard (Blank Solution)	4952B
Radon-226 for Radon Analysis	4953C
Red Brass	1109
Red Brass	C1109
Red Brass	1110
Red Brass	C1110
Red Brass	1111
Red Brass	C1111
Reduced Iron Oxide	691
Reference Fuel Isooctane	1816a
Reference Fuel n-Heptane	1815a
Reflection Step Tablet	2061
Refractive Index Glass	1820
Refractive Index Silicone Liquids	1823
Refractive Index, Soda-Lime Glass	1822
Relative Stress-Optical Coefficient	708
Glass	
Resulfurized-Rephosphorized Steel	C1221
Rice Flour	1568
River Sediment	1645
River Sediment, Environmental	4350B
Radioactivity	
Rocky Flats Soil Number 1,	4353
Environmental Radioactivity	
Rubidium Melting Point	1969
Rutile Ore	670
Scanning Electron Microscope	484c
Magnification Standard	
Scanning Electron Microscope	2069
Performance Standard	
Secondary Standard Flexible Disk	3210
Cartridge (Computer Amplitude	
Reference)	
Secondary Standard Magnetic Tape	3200
Secondary Standard Magnetic Tape	1600
Cassette	
Secondary Standard Magnetic Tape	3216
Cartridge (Computer Amplitude	
Reference)	
Second Surface Aluminum Mirror for	2023
Specular Reflectance	

Name	SRM
Second Surface Aluminum Mirror for Specular Reflectance	2024
Second Surface Aluminum Mirror with Wedge for Specular Reflectance	2025
Selenium-Bearing Steel	1170ь
Selenium-75 Radioactivity Standard	4409L-D
Sheet Brass	37E
Silica Brick	198
Silica Brick	199
Silicon-Aluminum Alloy	87a
Silicon Bronze	158A
Silicon Density Standard	1840
Silicon Density Standard	1841
Silicon Metal	57a
Silicon Powder, Spacing Standard	640a
for X-ray Diffraction	
Silicon Power Device Level	1522
Resistivity Standard	
Silicon Resistivity Standard for Eddy	1523
Current Testers	
Silver 2-Ethylhexanoate	1077a
Silver-Gold Thermocouple Wire	733
Silver, Vapor Pressure	748
Sintered and Arc-Cast Tungsten,	1465
Thermal Conductivity and	
Electrical Resistivity	
Sintered and Arc-Cast Tungsten,	1466
Thermal Conductivity and	
Electrical Resistivity	
Sintered and Arc-Cast Tungsten,	1467
Thermal Conductivity and	
Electrical Resistivity	
Sintered and Arc-Cast Tungsten,	1468
Thermal Conductivity and	
Electrical Resistivity	
Smoke Density Chamber Standard	1007a
(Flaming Exposure Condition)	
Smoke Density Chamber Standard	1006ь
(Non-flaming Exposure Condition)	
Soda-Lime Container Glass	621
Soda-Lime Flat Glass	620
Soda-Lime Float Glass	1830
Soda-Lime Glass	1826
Soda-Lime Glass Powder	92

T

Name	SRM
Soda-Lime Sheet Glass	1831
Soda-Lime Silica Glass	622
Soda-Lime Silica Glass	710
Soda-Lime Silica Glass for Liquidus	773
Temperature	
Sodium Bicarbonate	191a
Sodium Bicarbonate	2191
Sodium Carbonate	192a
Sodium Carbonate	2192
Sodium Chloride	2201
Sodium Chloride (Clinical Standard)	919
Sodium Cyclonexanebutyrate	10696
Sodium Oxalate Reductometric	40n
Sodium Puruvata	010
Sodium Tetrabarata Decebudrata	910 1876
(Boray)	1670
Solder	127h
Solder	1131
Special Nuclear Container DOT 6M	9940
15 gal.	<i>,,,,</i> ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,
Special Nuclear Container, 55 gal	9941
Special Nuclear Container Type A	9942
10 gal.	
Special Nuclear Container, Type A.	9943
55 gal.	
Special Nuclear Material Package	9910
Spectrographic Ingot Iron and	461
Low-Alloy Steel Standard (Rod)	
Spectrographic Ingot Iron and	462
Low-Alloy Steel Standard (Rod)	
Spectrographic Ingot Iron and	463
Low-Alloy Steel Standard (Rod)	
Spectrographic Ingot Iron and	464
Low-Alloy Steel Standard (Rod)	
Spectrographic Ingot Iron and	465
Low-Alloy Steel Standard (Rod)	ACC
Spectrographic Ingot Iron and	400
Low-Alloy Steel Standard (Rod)	167
Low Allow Steel Stendard (Red)	407
Spectrographic Ingot Iron and	468
Low-Alloy Steel Standard (Rod)	400
Spectrographic Ingot Iron and	1166
Low-Allov Steel Standard	
Spectrographic Stainless Steel	442
Standard Spectrographic Staipless Steel	443
Standard	
Spectrographic Stainless Steel	444
Standard Spectrographic Staipless Steel	D849
Standard (Disc)	<b>D</b> 079
Spectrographic Stainless Steel	D850
Standard (Disc)	
Spectrographic Stainless Steel	445
Standard (Group II)	
Name	SRM
---	-------------------
Spectrographic Stainless Steel Standard (Group II)	446
Spectrographic Stainless Steel Standard (Group II)	447
Spectrographic Stainless Steel Standard (Group II)	448
Spectrographic Stainless Steel Standard (Group II)	449
Spectrographic Stainless Steel Standard (Group II)	450
Spectrographic Stainless Steel Standard (Rod)	849
Spectrographic Stainless Steel Standard (Rod)	850
Spectrographic Steel Standard (Disc)	D803a
Spectrographic Steel Standard (Disc)	D807a
Spectrographic Steel Standard (Bod)	8032
Spectrographic Steel Standard (Rod)	804a
Spectrographic Steel Standard (Rod)	00 <del>4</del> a
Spectrographic Steel Standard (Rod)	805a
Spectrographic Steel Standard (Rod)	807a
Spectrographic Steel Standard (Rod)	808a
Spectrographic Steel Standard (Rod)	809a
Spectrographic Steel Standard (Rod)	817Ь
Spectrographic Steel Standard (Rod)	820a
Spectrographic Steel Standard (Rod)	821
Spectrographic Steel Standard (Rod)	827
Spectrographic Tool Steel Standard	136
Spectrographic Tool Steel Standard	427
Spectrographic Tool Steel Standard	420
Spectrographic Tool Steel Standard	438
Spectrographic Tool Steel Standard	439
Spectrographic Tool Steel Standard	440
Spectrographic Tool Steel Standard	441
Spectrographic Tool Steel Standard	837
Spectrographic Tool Steel Standard	840
Spectrographic Tool Steel Standard	D837
	D940
(Disc)	D840
(Disc)	D841
Spectrographic Zinc-Base Die-Casting Alloy A	625
Spectrographic Zinc-Base Die-Casting Alloy B	626
Spectrographic Zinc-Base Die-Casting Alloy C	627
Spectrographic Zinc-Base Die-Casting Alloy D	628
Alloy E	629
Alloy F	630
Spectrographic Zinc Spetter Standard	031
Spectroscopic Litanium-Base Standard	041
Spectroscopic Titanium-Base Standard	642
Spectroscopic Titanium-Base Standard	643

Name	SRM
Spectroscopic Titanium-Base Standard	644
Spectroscopic Titanium-Base Standard	645
Spectroscopic Titanium-Base Standard	646
Spheroidized Iron Carbide in Ferrite	493
Spreading Resistance Calibration	2529
(100) n Type Silicon	
Spreading Resistance Calibration	2528
(100) p.Type Silicon	
Spreading Resistance Calibration	2527
(111) n-Type Silicon	
Spreading Resistance Calibration	2526
(111) p-Type Silicon	
Stabilized Wine	1590
Stainless Steel	121d
Stainless Steel	123c
Stainless Steel	160b
Stainless Steel (AISI 446)	367
Stainless Steel (AISI 446)	1267
Stainless Steel, 13% Chromium	/3c
Stainless Steel, Cr-Ni	
Stainless Steel, Cr-Ni	C1152
Stainless Steel, Cr-Ni	1152
Stainless Steel, Cr-Ni	C1153
Stainless Steel, Cr-Ni	11539
Stainless Steel, Cr-Ni	C1154
Stainless Steel, Cr-Ni	1154a
Stainless Steel, Cr-Ni	1155
Stainless Steel, Cr-Ni-Mo	1172
Stainless Steel, Cr-Mi-IND	1171
Stainless Steel for Ditting or Crewice	1890
Corrosion	
Stainless Steel Thermal Expansion	738
Stearic Acid Rubber Compound	372h
Steel (AISL 1211)	368
Steel (Lead-Bearing)	1169b
Strontium Cyclohexanebutyrate	1070a
Strontium-85 Radioactivity Standard	4403L-B
Strontium-89 Radioactivity Standard	4945D
Styrene-butadiene Rubber (Type 1500)	386h
Succinonitrile Freezing Point	1970
Sucrose	1/c
Sulfate and Nitrate on Filter Media	20/3
Sulfur Dioxide in Nitrogen	1001a
Sulfur Dioxide in Nitrogen	1662a
Sulfur Dioxide in Nitrogen	1663a
Sultur Dioxide in Nitrogen	1664a
Sultur Dioxide in Nitrogen	1693
Sulfur Dioxide in Nitrogen	1694

Name	SRM
Sulfur Dioxide in Nitrogen	1696
Sulfur Dioxide Permeation Tube	1627
(2 cm tube)	1027
Sulfur Dioxide Permeation Tube	1626
(5 cm tube)	
Sulfur Dioxide Permeation Tube	1625
(10 cm tube)	
Sulfur in Coal	2682
Sulfur in Coal	2683
Sulfur in Coal	2684
Sulfur in Coal	2685
Sulfur in Residual Fuel Oil	1619
Sulfur in Residual Fuel Oil	1620a
Sulfur in Residual Fuel Oil	1621b
Sulfur in Residual Fuel Oil	1622b
Sulfur in Residual Fuel Oil	1623a
Sulfur in Residual Fuel Oil	1624a
Sulfur Rubber Compound	371g
Superconductive Thermometric Fixed	767a
Point Device	
Superconductive Thermometric Fixed	768
Point Device	
Surface Flammability Standard	1002c
Synthetic Sapphire	720
Technetium-99 Radioactivity Standard	4288
Technetium-99m Radioactivity	4410H-I
Standard	1000
The life of the li	1808
Thannah Basistan as Eihness Class	4404L-F
Pott	1451
Dall Thormal Resistance, Ethrous Class	14501
Roard	14500
Thorium 228 Thallium 208 Gamma ray	4206C
Point Source Standard	4200C
Tin-Base Bearing Metal	54D
Tin Freezing Point	741
Tin-113-Indium-113m Radioactivity	141 14021 C
Standard	4402L-C
Tin-121m Point-Source Gamma-ray	4264B
Emission-Rate Standard	7207D
Tin. Secondary Freezing Point	42ø
Standard	
Titanium Allov	654a
Titanium-Base Allov	173b
Titanium-Base Allov	176

Name	SRM
Titanium Pase Alloy (Unalloyed)	650
Titanium Base Alloy (Unalloyed)	651
Titanium-Base Alloy (Unalloyed)	652
Titanium Dioxide	154b
Toluene	2110
Tomato Leaves	1573
Tool Steel (AISI M2)	1375
Tool Steel (AISI M2)	1157
Tool Steel Abrasive Wear Standard	1857
Tracealloy (Nickel-Base	897
High-Temperature Alloy)	
Tracealloy (Nickel-Base	898
High-Temperature Alloy)	
Tracealloy (Nickel-Base	899
High-Temperature Alloy)	
Trace Elements in a Glass Matrix	61 <mark>0</mark>
Trace Elements in a Glass Matrix	611
Trace Elements in a Glass Matrix	612
Trace Elements in a Glass Matrix	613
Trace Elements in a Glass Matrix	614
Trace Elements in a Glass Matrix	615
Trace Elements in a Glass Matrix	616
Trace Elements in a Glass Matrix	617
Trace Elements in Coal (Bituminous)	1632a
Trace Elements in Coal (Sub-	1635
bituminous	1(22
Trace Elements in Coal Fly Ash	1633a
Trace Elements in Fuel Oil	1634a
Trace Elements in Water	16438
2.2.4 Trimethylpontone	1030
Z, 2, 4-1 Thilethylpentane	217C
Tris Basimetric	7720
Tris, for Solution Calorimetry	723a
Tris(hydroxymethyl)aminomethane	927a
Tris(hydroxymethyl)aminomethane	923
hydrochloride	125
Tris(1-phenyl-1, 3-butanediono)	1078b
Chromium (III)	10/00
Tris(1-phenyl-1, 3-butanediono)	1079Ь
Iron (III)	
Triphenyl Phosphate	1071b
Tungsten Carbide	276a
Tungsten-Chromium-Vanadium Steel	50c
Tungsten Concentrate	277
Tungsten, Heat Capacity	782
Tungsten-20% Molybdenum Alloy	480
Electron Microprobe Standard	
Tungsten Thermal Expansion	737
Unalloyed Copper	1034
Unalloyed Copper, Cu "O"	393
Unalloyed Copper, Cu IV	457
Unalloyed Copper, Cu XI	454
Unalloyed Copper, Cu I (Chip)	394
Unalloyed Copper, Cu II (Chip)	395
Unalloyed Copper, Cu III (Chip)	396
Unalloyed Copper, Cu V (Chip)	398
Unalloyed Copper, Cu VI (Chip)	399
Unalloyed Copper, Cu V II (Unip)	400
onanoyeu Copper, Cu I (Kou)	+74

Name	SRM
Unalloved Copper, Cu II (Rod)	495
Unalloyed Copper, Cu III (Rod)	496
Unalloyed Copper, Cu V (Rod)	498
Unalloyed Copper, Cu VI (Rod)	499
Unalloyed Copper, Cu VII (Rod)	500
Unalloyed Titanium	354
Uranium Isotopic Standard (Nominally	U-0002
depleted to 0.02%)	
Uranium Isotopic Standard	U-005a
Uranium Isotopic Standard	U-010
(Nominally 1% Enriched)	
Uranium Isotopic Standard	U-015
(Nominally 1.5% Enriched)	
Uranium Isotopic Standard	U-020
Uranium Isotopic Standard	U-030a
Uranium Isotopic Standard	U-050
(Nominally 5% Enriched)	11100
Uranium Isotopic Standard	U-100
(Nominally 10% Enriched)	11150
(Naminally, 15%, Enriched)	0-150
(Nominally 15% Enriched)	11.200
(Nominally 20% Enriched)	0-200
(Nominany 20% Enriched)	U.350
(Nominally 35% Enriched)	0.000
Uranium Isotopic Standard	U-500
(Nominally 50% Enriched)	0.000
Uranium Isotopic Standard	U-750
(Nominally 75% Enriched)	
Uranium Isotopic Standard	U-800
(Nominally 80% Enriched)	
Uranium Isotopic Standard	U-850
(Nominally 85% Enriched)	
Uranium Isotopic Standard	U-900
(Nominally 90% Enriched)	11.020
Uranium Isotopic Standard	U-930
(Nominally 93% Enriched)	11070
(Neminally 07% Engineed)	0-970
(Nominally 97% Enriched)	960
Uranium Oxide	950b
Uranium Oxide	969
Uranium 233 Spike Assay and	995
Isotopic Solution Standard	
Uranium-235 Spike Assav and	993
Isotopic Solution Standard	
Urban Dust/Organics	1649
Urban Particulate Matter	1648
Urea	912a
Urea	2141
Urea	2152
Uric Acid	913
Vanadium and Nickel in Residual	1618
Fuel Oll Vanadium in Curda Oil	8505
Vanadium 49 Low Energy Photon	4266
Standard	1200
Waspaloy	349
Wear-Metals in Lubricating Oil	1084
(100 ppm)	

Name	SRM
Wear-Metals in Lubricating Oil	1085
(300 ppm)	
Wheat Flour	1567
White Cast Iron	338
White Cast Iron (Disc)	1145
White Cast Iron (Disc)	1146
White Cast Iron (Disc)	1150
White Cast from (Disc)	20105
Hamicharical Reflectance	20190
White Commis Tile for Directional	2020
white Ceramic The for Directional	2020
Hemispherical Reflectance	2.1
White Iron	3015
White Opan Glass Diffuse Spectral	2015
Reflectance Standard for the	
Visible Spectrum	
Xenon-127 Gaseous Radioactivity	4309G
Standard	
Xenon-133 Gaseous Radioactivity	4307I
Standard	
Xenon-133 Gaseous Radioactivity	4415L-I
Standard	
Xenon-133, Xenon-137, Krypton-85	4310B
Mixed Gaseous Radioactivity	
Standard	
X-ray Film Step Tablet	1001
X-ray Powder Diffraction Intensity	674
Standard	
X-ray Powder Diffraction (Mica)	675
Low 2 Theta	0,2
Vtterbium, 169 Radioactivity Standard	44191 .B
Zinc Base Alloy (Die Casting)	940
Zine Concentrates	1130
Zine Concentrates	370
Zine Concentrates	329 1077h
	10730
Zinc, Freezing Point	740
Zinc, Freezing Point Standard	43h
Zinc Metal	683
Zinc Oxide Rubber Compound	370e
Zircaloy-2	360a
Zircaloy-4 Metal	1237
Zircaloy-4 Metal	1238
Zircaloy-4 Metal	1239
Zirconium-Barium Chromate	1651
Formulation for Heat-Source	
Powder Calorimetry	
Zirconium-Barium Chromate	1652
Formulation for Heat-Source	
Powder Calorimetry	
Zirconium-Barium Chromate	1653
Formulation for Heat-Source	
Powder Calorimetry	
Zirconium Metal	1234
Zirconium Metal	1234
Zirconium Metal	1235
Zirconium wietai	1230

U. S. Department of Commerce Frederick B. Dent Secretary

National Bureau of Standards Richard W. Roberts, Director Appendix II.

Certificates for the Environmental Research, Analysis, and Control Standards (listed in numerical order).

## National Bureau of Standards Certificate of Analysis Standard Reference Material 1579 Powdered Lead Based Paint

This Standard Reference Material is intended for use in the calibration of apparatus and methods used in the determination of lead in paint removed from the interior surfaces of old housing. The certified value is based on at least a 100 milligram sample of the as-received, total material.

The certified value of 11.87 percent lead is the weighted average value determined by a statistical analysis of the results of 32 determinations by atomic absorption spectrometry (average 11.84 percent lead, s = 0.13 percent lead), and 16 determinations by polarography (average 11.93 percent lead, s = 0.13 percent lead). The standard error of the weighted average is 0.02 percent lead, and the half-width of the 95 percent confidence interval is taken to include  $\pm 0.04$  percent lead by weight.

X-ray fluorescence spectrometry showed the bottle-to-bottle inhomogeneity of the material with respect to lead content to be no greater than 0.02 percent lead; no within-bottle inhomogeneity was detected.

Analyses for lead and determinations of homogeneity were carried out in the NBS Analytical Chemistry Division by the following persons:

X-ray Fluorescence: S. D. Rasberry Atomic Absorption Spectrometry: T. C. Rains and T. A. Rush Polarography: E. J. Maienthal

Statistical calculations were carried out by J. Mandel of the NBS Institute for Materials Research.

The overall direction and coordination of the technical measurements leading to this certificate were performed under the chairmanship of B. Greifer.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234 January 23, 1973 J. Paul Cali, Chief Office of Standard Reference Materials

#### Preparation, Testing, and Analysis

#### Collection

The paint for this Standard Reference Material was collected by the staff of the Philadelphia Department of Public Health from the interior surfaces of dwellings undergoing renovation. The paint was softened with a hand torch, scraped from the plaster and wood substrates, and collected in plastic bags as a heterogeneous mixture of many different kinds of paints. In the laboratory, non-paint matter such as bits of metal, plastic, glass, and wood were removed and the paint mixture was ground in a disk mill to produce a material suitable for feeding into a jet mill. The paint was comminuted in a jet mill operating at 100 psig air pressure, then sieved through a 100-mesh vibrating screen to remove the coarse, non-grindable fraction. Two additional passes through the jet mill at 97 to 107 psig gave a fine powder with 99.31 weight percent passing through a 325 mesh sieve.

#### Homogeneity

Sample homogeneity was ascertained by x-ray fluorescence analysis for lead content on 17 samples chosen at random from the total lot. A statistical analysis of the data from 136 observations showed the bottle-to-bottle variability among the samples to be no greater than 0.02 percent lead. No within-bottle variation with respect to lead was detected.

#### Dissolution

A procedure used to dissolve the sample is summarized briefly: dry ash the weighed paint for 2 hours at  $450^{\circ}$  C, digest with 2:5 HCl - HNO<sub>3</sub> containing HF, evaporate to dryness; treat with HNO<sub>3</sub>, evaporate to dryness; treat twice with HCl and evaporate to dryness each time. Extract the solids twice with portions of acetic acid - ammonium acetate solution, heating for several hours just below boiling. Combine the extracts and heat the mixture (including solids) for one hour, just below boiling. Cool the mixture and determine lead in solution. (The solids need not be removed for polarographic analysis.)

An alternate procedure for sample dissolution is: dry ash the weighed paint for 6 hours at 500 ° C, cool, then digest for 2 hours in 1:1 HCl -  $HNO_3$ . Separate the insoluble solids from the solution by centrifuging, and wash 3 times with 1:10  $HNO_3$  combining the rinsings with the principal solution. Determine lead in solution.

Details of the dissolution procedures, the analytical procedures, and results will be published in the 260 series of NBS Special Publications.

U. S. Department of Commerce Philip M. Klutznick Secretary National Bureaus of Standards Ernest (Ambler, Director

## National Bureau of Standards Certificate of Analysis Standard Reference Material 1580 Organics in Shale Oil

This SRM is intended primarily for evaluating the reliability of analytical methods for the determination of trace level organic compounds in an oil matrix, i.e., shale oil, petroleum crude oil, or coal-derived liquids.

Certified Values of Constituent Organic Compounds: The certified values for selected organic constituents are shown in Table 1. These values are based on results obtained by two independent, analytical methods (see Table 2). Non-certified values, which are given for information only, are listed in Table 3.

#### NOTICE AND WARNINGS TO USER

Expiration of Certification: This certification is valid, within the limits certified, for 3 years from the date of purchase. In the event that the certification should become invalid before then, purchasers will be notified by NBS.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10-30 °C.

Use: Samples for analysis should be withdrawn from ampoules immediately after opening and processed without delay for any certified value in Table 1 to be valid within the stated uncertainty. Certified values are not applicable to ampoules stored after opening, even if resealed.

#### PREPARATION AND ANALYSIS

The shale oil for this SRM came from a 150-ton retort for *in-situ* simulated combustion of oil shale, operated by the Laramie Energy Technology Center, Laramie, Wyoming. The shale was from the Mahogany Zone of the Colorado Green River Formation. The shale oil had been supplied in November 1975 to the Oak Ridge National Laboratory (ORNL) where it underwent centrifugation to separate the oil from water and sludge. The shale oil was provided to NBS by Bruce R. Clark, ORNL, Oak Ridge, Tennessee.

At NBS, the centrifuged sample was filtered through fine filter paper and mixed in a 20-liter, Teflonstoppered, glass bottle by rolling for 40 hours. Samples were aliquoted into 2-mL amber glass ampoules. Although not intended to be representative of all shale oils, SRM 1580 provides a typical specimen of this matrix for use in developing analytical methods.

Randomly selected ampoules were analyzed. Each analyst examined at least six ampoules, sometimes measuring replicates from one ampoule. No trend was found in measured values with the ampouling sequence.

Two independent techniques were employed for the determination of the certified values for the organic constituents. Three different methods of sample preparation were used prior to analysis: simple dilution of the shale oil with methylene chloride (or other suitable solvent); acid/base extraction to isolate acidic, basic, and neutral components; and a high performance liquid chromatographic fractionation. The following techniques were employed for the final quantitative analysis: gas chromatography (GC), gas chromatography/mass-spectrometry (GC/MS) with single ion monitoring for selective detection, and high performance liquid chromatography (HPLC) with selective fluorescence detection. All GC/MS analyses used the standard addition method for quantitation. The GC and HPLC analyses employed either internal standard, external standard, or standard addition methods. The analytical methods and the corresponding values are summarized in Table 2.

Consultation on the statistical design of the experimental work was provided by K. R. Eberhardt of the Statistical Engineering Division.

The coordination of the technical measurements leading to certification were performed under the direction of H. S. Hertz, S. N. Chesler, L. R. Hilpert, W. E. May, and S. A. Wise.

The technical and support aspects involved in preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234 November 24, 1980 (Revision of Certificate dated 3-10-80)

(over)

George A. Uriano, Chief Office of Standard Reference Materials The following members of the staff of the Center for Analytical Chemistry, Organic Analytical Research Division, performed the analytical determinations.

1. J. M. Brown-Thomas	5. P. L. Konash
2. S. N. Chesler	6. W. E. May
3. F. R. Guenther	7. R. M. Parris
4. L. R. Hilpert	8. K. L. Richie

Compound	Concentration $(\mu g/g^a)$
Fluoranthene	$54 \pm 10$
Pyrene	$104 \pm 18$
Benzo[a]pyrene	$21 \pm 6$
Benzo[e]pyrene	$18 \pm 8$
Perylene	$3.4 \pm 2.2$
Phenol	$407 \pm 50$
o-Cresol	$385 \pm 50$
2,6-Dimethylphenol	$175 \pm 30$
Benzo[f]quinoline	$16 \pm 4$
(5,6-Benzoquinoline)	

TABLE 1. Certified Values of Organic Constituents

<sup>a</sup>The estimated uncertainty listed for a constituent is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material inhomogeneity. The estimated uncertainty is intended to correspond to approximately 95% confidence limits.

TABLE 2. Summar	y of Results b	y the Analytical	Methods Used in	Certification
			-	

Compound	Concentration $(\mu g/g)^a$	Number of Ampoules Analyzed	Sample Preparation Technique	Analytical Technique
Fluoranthene	$55 \pm 5$	6	Direct Injection	GC/MS
	53 ± 2	9	HPLC	HPLC
Ругепе	$101 \pm 5$	6	Direct Injection	GC/MS
	$107 \pm 8$	10	HPLC	HPLC
Benzo[a]pyrene	$20 \pm 1$	6	Direct Injection	GC/MS
	$23 \pm 1$	8	HPLC	HPLC
Benzo[e]pyrene	17 ± 1	6	Direct Injection	GC/MS
	$20 \pm 3$	8	HPLC	HPLC
Perylene	$2.8 \pm 0.6$	5	Direct Injection	GC/MS
	$3.9 \pm 0.6$	11	HPLC	HPLC
Phenol	412 ± 35	8	HPLC	GC/MS
	$402 \pm 4$	8	Acid/Base Extraction	GC
o-Cresol	$386 \pm 42$	8	HPLC	GC/MS
	$384 \pm 9$	8	Acid/Base Extraction	GC
2,6-Dimethylphenol	183 ± 23	9	HPLC	GC/MS
	$168 \pm 8$	8	Acid/Base Extraction	GC
Benzo[/]quinoline	$16 \pm 1$	7	HPLC	HPLC
(5,6-Benzoquinoline)	15 ± 1	8	Acid/Base Extraction	Multi- dimen-

<sup>a</sup>Uncertainty is the standard deviation of a single measurement.

GC

#### TABLE 3. Non-Certified Values of Organic Compounds in Shale Oil

NOTE: The values shown in this table are not certified because they are not based on the results of two independent methods. These values are included for information only.

Compound	Concentration $(\mu g/g)$
p-Cresol	(270) <sup>a</sup>
<i>m</i> -Cresol	(330) <sup>a</sup>
2,5-Dimethylphenol	(320) <sup>a</sup>
2,4-Dimethylphenol	(380) <sup>a</sup>
2,5,6-Trimethylphenol	(360) <sup>a</sup>
2,4,6-Trimethylphenol	(120) <sup>a</sup>
Phenanthridine	(45) <sup>b</sup>
<sup>a</sup> Acid/base extraction - GC analysis	
<sup>b</sup> HPLC extraction - HPLC analysis	

# National Bureau of Standards

# Certificate of Analysis

### Standard Reference Material 1620a

### Sulfur in Residual Fuel Oil

This Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1620a is a commercial "No. 5 Heavy" residual fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certified using three independent methods of analysis: gravimetry, ion chromatography, and x-ray fluorescence.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE AND RECOMMENDED USE: Due to the high sulfur content of SRM 1620a, it is recommended that the bottle be shaken vigorously before sampling. Homogeneity and stability testing at NBS indicates that the best results are achieved when the material is shaken before use.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1620a was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

Physical	Properties	for SRM	1620a
T nysteur i	ropenties	TOT SIGM	10204

Table I

Flash Point <sup>a</sup> °C	Kinematic Viscosity <sup>b</sup> 50 °C (cSt)	Pour Point <sup>°</sup> °C	Density @ 20 °C <sup>d</sup> g <sub>/</sub> cm
70	47.75	2	1.096

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

#### Methods Used for Physical Tests

a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester

b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids

c. ASTM D97-66 (1978) Pour Point of Petroleum Oils

d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

#### Table 2

#### Semi-Quantitative Emission Spectrometry

Element	$\mu g/mL$	Element	µg, mL	
AI	20	· Mo	<1	
В	<1	Na	31	
Ca	9	Ni	<1	
Cr	<1	Si	13	
Cu	<I	Sn	<1	
Fe	<5	Ti	<1	
Mg	<I	V	<1	
Mn	<1	Zn	23	

#### Analysis for SRM 1620a

Note: SRM 1620a was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed by J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry.

# National Bureau of Standards

# Certificate of Analysis

## Standard Reference Material 1621b

### Sulfur in Residual Fuel Oil

Sulfur Concentration . . . . .0.950 ± 0.005 weight percent

This Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1621b is a commercial "No. 6" residual fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certified using three independent methods of analysis: gravimetry, ion chromatography, and x-ray fluorescence.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE: The certification of SRM 1621b is valid for 3 years from date of purchase.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1621b was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

Physical Properties for SRM 1621b					
Flash Point <sup>a</sup> °C	Kinematic Viscosity <sup>b</sup> 50 °C (cSt)	Pour Point <sup>c</sup> °C	Density @ 20 °C <sup>d</sup> g/cm <sup>3</sup>		
111	89.2	11	0.929		

Table 1 Physical Properties for SRM 1621b

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

#### Methods Used for Physical Tests

a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester

b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids

c. ASTM D97-66 (1978) Pour Point of Petroleum Oils

d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

#### Table 2

#### Semi-Quantitative Emission Spectrometry

Elemen	t μg/mL	Element	μg/mL
Al	6	Mo	<1
В	<1	Na	8
Ca	9	Ni	6
Cr	3	Si	6
Cu	<1	Sn	<1
Fe	<5	Ti	<1
Mg	<1	V	15
Mn	1	Zn	15

#### Analysis for SRM 1621b

Note: SRM 1621b was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed by J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry.

# National Bureau of Standards

# Certificate of Analysis

## Standard Reference Material 1622b

### Sulfur in Residual Fuel Oil

Sulfur Concentration . . . . . 1.982 ± 0.018 weight percent

This Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1622b is a commercial "No. 6" residual fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certified using three independent methods of analysis: gravimetry, ion chromatography, and x-ray fluorescence.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE: The certification of SRM 1622b is valid for 3 years from date of purchase.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1622b was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

	Physical	2	
Flash Point <sup>a</sup> °C	Kinematic Viscosity <sup>b</sup> 50 °C (cSt)	Pour Point <sup>°</sup> °C	Density @ 20 °C' g/cm <sup>3</sup>

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

-7

0.984

#### Methods Used for Physical Tests

a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester

b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids

c. ASTM D97-66 (1978) Pour Point of Petroleum Oils

377.34

65

d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

#### Table 2

Semi-Quantitative Emission Spectrometry

#### Analysis for SRM 1622b

Element µg/mL		Element	µg/ mL	
Al	8	Мо	<1	
В	<1	Na	25	
Ca	24	Ni	15	
Сг	1	Si	13	
Cu	<1	Sn	<1	
Fe	<5	Ti	<1	
Mg	2	v	50	
Mn	1	Zn	11	

Note: SRM 1622b was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry

# National Bureau of Standards

# Certificate of Analysis

## Standard Reference Material 1623a

### Sulfur in Residual Fuel Oil

#### Sullur Concentration. . . . $0.240 \pm 0.003$ weight percent

I his Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1623a is a commercial "No. 5 Heavy" residual fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certilied using three independent methods of analysis: gravimetry, ion chromatography, and x-ray fluorescence.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE: The certification of SRM 1623a is valid for 3 years from date of purchase.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1623a was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

	Table	1		
Physical	Properties	for	SRM	1623a

Flash Point " ° C	Kinematic Viscosity <sup>b</sup> 50 °C (cSt)	Pour Point <sup>e</sup> °C	Density @ 20 °C <sup>d</sup> g/cm <sup>3</sup>
140	53.82	17	0.918

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

#### Methods Used for Physical Tests

a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester

b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids

c. ASTM D97-66 (1978) Pour Point of Petroleum Oils

d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

#### Table 2

#### Semi-Quantitative Emission Spectrometry Analysis for SRM 1623a

 Element	μg/mL	Element	µg/mL
Al	5	Мо	<1
В	<1	Na	9
Ca	9	Ni	1
Cr	1	Si	<1
Cu	<1	Sn	<1
Fe	<5	Ti	<1
Mg	<1	V	3
Mn	<1	Zn	15

Note: SRM 1623a was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed by J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry.

# National Bureau of Standards Certificate of Analysis Standard Reference Material 1624a

### Sulfur in Distillate (Diesel) Fuel Oil

Sulfur Concentration . . . . .  $0.141 \pm 0.002$  weight percent

This Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1624a is a commercial "No. 2-D" distillate fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certified using two independent methods of analysis: gravimetry and ion chromatography.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE: The certification of SRM 1624a is valid for 3 years from date of purchase.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1624a was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

<u>Table 1</u> Physical Properties for SRM 1624a				
Flash Point <sup>a</sup> °C	Kinematic Viscosity <sup>b</sup> 40 °C (cSt)	Cloud Point <sup>c</sup> °C	Density @ 20 °C <sup>d</sup> g/cm <sup>3</sup>	
53	2.57	-14	0.848	

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

#### Methods Used for Physical Tests

a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester

b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids

c. ASTM D2500-66 (1976) Cloud Point of Petroleum Oils

d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

#### Table 2

#### Semi-Quantitative Emission Spectrometry

#### Analysis for SRM 1624a

Element	μg/mL	Element	μg/mL
Al	1	Мо	<1
В	<1	Na	<1
Ca	7	Ni	<1
Cr	<1	Si	<1
Cu	<1	Sn	<1
Fe	<5	Ti	<1
Mg	<1	V	<1
Mn	<1	Zn	<1

Note: SRM 1624a was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed by J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry.

U.S. Department of Commerce Juanita M, Kreps Secretary National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis Standard Reference Material 1630

#### Trace Mercury in Coal

This Standard Reference Material is intended as an analytical standard for the determination of trace mercury in coal. The material is a commercially available coal that was crushed to a size of 210 to 500 micrometers with a roll crusher. From a total of 500 packaged bottles, 30 were randomly selected for analysis. Duplicate determinations were made on 0.5 g portions of 25 of these bottles, and single determinations were made on the other five. The mercury content of this material was obtained by destructive neutron activation analysis.

The recommended value is the average of these 55 determinations on 30 bottles, which was found to be:

#### Mercury content = 0.13 $\mu g/g$

The recommended value is not expected to change by more than  $\pm 1$  in the last significant figure.

A study of homogeneity showed no variability among bottles that could not be accounted for by analytical error. Duplicate samples from the same bottle indicated a homogeneity for mercury of  $\pm$  5% (relative).

The mercury content was also determined by flameless atomic absorption spectrometry, yielding an average value of 0.14  $\mu$ g/g.

Selenium was also determined using destructive neutron activation analysis. The value obtained, which is not certified but included for information only, was found to be 2.1  $\mu g/g$ .

The homogeneity testing and analyses for certification were performed in the NBS Analytical Chemistry Division by T. E. Gills and H. Rook under the direction of P. D. LaFleur.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by C. L. Stanley.

Washington, D.C. 20234 August 1, 1979 (Revision of Certificate dated 11-2-71 Editorial Revision only.) George A. Uriano, Chief Office of Standard Reference Materials

#### ANALYTICAL PROCEDURE

The bottles containing the samples were allowed to remain open at room temperature (about 25 °C) for twenty-four hours.

The coal samples, along with solution standards of mercury and NBS Standard Reference Material 1571 (Orchard Leaves) used as a control, were encapsulated in cleaned quartz vials. The geometry of both the samples and the standards were optimized so that flux monitors were not needed. The samples were irradiated for four hours at a thermal flux of  $6 \times 10^{13}$  n·cm<sup>-2</sup> sec<sup>-1</sup>. The samples were allowed to decay for three days to minimize the personnel dose rate. The samples were postweighed into porcelain boats and burned in a combustion tube. The volatile mercury compounds and other volatile products liberated during burning were trapped in a liquid nitrogen cold trap. The cold trap was allowed to warm to room temperature. The mercury compounds were then transferred to polyethylene bottles by washing the cold trap with concentrated nitric acid and water. For this analysis, <sup>197</sup>Hg produced by <sup>196</sup>Hg(n,  $\gamma$ ) <sup>197</sup>Hg was used as the measuring activity.

Bromine-82, an interfering isotope, was separated from the sample by using the classical silver bromide precipitation.

The samples were counted on a  $22 \text{ cm}^3$  Ge(Li) detector connected to a 2048-multichannel analyzer. The accumulated data was processed by computer for peak identification and integration. The concentrations were determined by using a Standard Comparator Method.

#### NOTE TO USER

It is suggested that persons using SRM 1630 to check their analytical technique should adopt the following criteria. If the average,  $\overline{X}$ , of N replicate measurements on this SRM is found to lie in the interval-

$$0.127 - \frac{0.013}{\sqrt{N}} < \vec{X} < 0.127 + \frac{0.013}{\sqrt{N}}$$

then the analytical technique used gives a result compatible with that found at NBS. However, if the value  $\overline{X}$  lies outside this interval, then the technique should be examined for possible bias or miscalibration.

NOTE: The above expression is not rigorously correct. It does not include a possible component for between laboratory variability nor sources of systematic error.

SRM 1630 Page 2

# National Bureau of Standards

# Certificate of Analysis

## Standard Reference Material 1632b

Trace Elements in Coal

### (Bituminous)

This Standard Reference Material (SRM) is intended for use in the calibration of apparatus and the evaluation of techniques employed in the analysis of coal or similar materials. SRM 1632b is a bituminous coal with a nominal sulfur content of 1.9%. It is in the form of a fine powder (-60 mesh).

<u>Certified Values of Constituent Elements:</u> The certified values for the constituent elements are given in Table 1. The certified values are based on measurements using proven techniques and methods. Noncertified values are given in Table 2 and are provided for information only. These values are based on measurements made using a single technique or method. While no reason exists to suspect systematic bias in the information values, no attempt was made to determine if such a bias exists that is attributable to the technique and/or method used. A list of analytical techniques and methods used for the different analyses is given in Table 3. As part of its update certification program, NBS will periodically update many of these values to certification status.

Expiration of Certification: The certification of SRM 1632b will be valid up to 5 years from the purchase date. Should any of the certified constituents become invalid prior to that date, purchasers will be notified by NBS.

<u>Use:</u> This material should be vacuum dried at ambient temperature for 24 hours prior to use. The certified concentrations are reported on a "dry-weight" basis, thus the concentration determined on undried samples should be adjusted for the moisture content of the sample. Typical moisture loss using the drying procedure stated above is 1.3%.

A minimum sample size of 250 mg of the dried material is required for the certified values to be valid.

This SRM should be kept in its original bottle. It should not be exposed to intense source of radiation, including ultraviolet lamps or sunlight.

The statistical analysis of the certification data was performed by R.C. Paule of the National Measurement Laboratory.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Gaithersburg, MD 20899 June 20, 1985 Stanley D. Rasberry, Chief Office of Standard Reference Materials

Source and Preparation of Material: The coal for this SRM was obtained from the Humphrey No. 7 mine and coal preparation plant of the Consolidation Coal Company, Christopher Coal Company Division, Osage, West Virginia. This mine produces bituminous coal with a sulfur content of 1.8-1.9 percent (dry basis). This coal was obtained from an underground mine that recovers coal from the Pittsburgh seam, which is considered the single most valuable and extensive coal seam in the United States.

Approximately 900 kg of the coal for SRM 1632b was oven dried prior to processing, in accordance with procedures outlined in ASTM D2013. The coal was reduced in size to -60 mesh and sieved prior to blending. The coal was then blended in a stainless steel cone blender (approximate capacity 0.85 cubic meter). After blending the coal was packaged in polyethylene-lined aluminum cans and was subsequently repackaged in fifty gram units.

#### Analysis

Major, Minor, and Trace Constituents: In general, the major, minor, and trace constituents were certified using two or more independent methods of analysis or two or more different laboratories. For those constituents that were determined using a single method, technique, or laboratory, the values are given for information only. (See Table 3).

<u>Calorific Value</u>: The calorific value was determined using measurements made in an isoperibol calorimeter, an isothermal calorimeter, and an adiabatic calorimeter at two different laboratories.

Moisture, Ash, and Volatile Matter: The moisture, ash, and volatile matter values were determined on measurements made using the standard ASTM methods, D3173, D3174, and D3175, respectively. In addition, commercial instruments commonly used for the determination of the parameters provided additional values.

Page 2 SRM 1632b

Major Con	stituents	Minor (	Constituents
	Content		Content
Elements	Wt. Percent	Elements	Wt. Percent
Carbon (Total)	$78.11 \pm 0.37^{a}$	Aluminum	$0.855 \pm 0.019$
Hydrogen	$5.07 \pm 0.06$	Calcium	$0.204 \pm 0.006$
Nitrogen	$1.56 \pm 0.07$	Iron	$0.759 \pm 0.045$
Sulfur	1.89 ± 0.06	Magnesium	$0.0383 \pm 0.0008$
Volatile matter	35.4 ±1.1	Potassium	$0.0748 \pm 0.0028$
		Sodium	$0.0515 \pm 0.0011$
		Titanium	$0.0454 \pm 0.0017$

Table 1. Certified Values of Constituent Elements

#### Trace Constituents

	Co	ntent	Content		
Element	/	g/g	Element	μg/g	
Arsenic	3.72	±0.09	Manganese	12.4 ±1.0	
Barium	67.5	± 2.1	Nickel	6.10 ± 0.27	
Cadmium	0.057	$3 \pm 0.0027$	Rubidium	5.05 ± 0.11	
Cobalt	2.29	±0.17	Selenium	1.29 ± 0.11	
Copper	6.28	$\pm 0.30$	Thorium	$1.342 \pm 0.036$	
Lead	3.67	$\pm 0.26$	Uranium	$0.436 \pm 0.012$	
			Zinc	11.89 ±0.78	
Calorif	ic Value <sup>b</sup>	, c		Ash, wt.%	

	14	10	0	5	±		35	; ]	Bt	u	/1	b	(	32.57	±	0.08	MJ	kg <sup>-1</sup> )	6.79 ± 0.16	
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-						

<sup>8</sup> The listed  $\pm$  uncertainties for carbon, hydrogen, volatile matter, and calorific value are two standard deviations of the certified value. The listed  $\pm$  uncertainties for all other constituents are two standard deviations for the certified values and include an allowance for minor sample heterogeneity. The observed sample variability was generally less than two percent of the constituent value.

<sup>b</sup>The calorific value (MJ kg<sup>-1</sup>) may decrease upon aging or normal oxidation of the coals. NBS will continue to monitor this value and report any substantive change in the certified calorific value to the purchaser. The reference date for the calorific value is May 1985. <sup>c</sup> The calorific value is determined as HHV2 (Higher Heating Value-Moisture Free).

#### Table 2. Noncertified Values for Constituent Elements

	Trace Constituents								
Element	Content µg/g	Element	Content µg/g						
Antimony	(0.24)	Lithium	(10)						
Bromine	(17)	Molybdenum	(0.9)						
Cerium	(9)	Samarium	(0.87)						
Cesium	(0.44)	Scandium	(1.9)						
Chlorine	(1260)	Silicon, wt %	(1.4)						
Chromium	(11)	Strontium	(102)						
Europium	(0.17)	Tungsten	(0.48)						
Hafnium	(0.43)	Vanadium	(14)						
Lanthanum	(5.1)								

Page 3 SRM 1632b

- - -

Method/ Element	A	В	с	D	E	F	G	н	1	J	к	L	м
Al			•	•								٠	
As			•		•								
Ash Content							• 2		•				
Ва			•										
Br			•										
C (Total)							• 5	٠	٠		٠		
Ca		•	•	•								•	
Cal Val									•	•			
Cd	•	•											
Ce			•										
Cl			٠										
Co			٠	•									
Cr			•										•
Cs			٠										
Cu					•								•
Eu			٠										
Fe	٠		•									٠	
н							• 5	٠	٠				
Hſ			•										
К		•	•	•								•	
La			•										
Li				•									
Mg	•	•	•										
Mn			•	•									
Мо			•										
N							• 6						
Na			•	•									
Ni	•												•
РЪ	•	•											
Rb		٠	٠	•									
s						•	• 4		٠			٠	
Sb			•										
Sc			٠										
Se			•		•								
Si			•									•	
Sm			•										
Sr			•										
Th		•	•										
Ti			•	•								•	•
		•	•										
v			•										•
Volatile Matta-							• 1						
W			•				- 3						
7-													
Zn				1									

## Table 3. Analytical Techniques and Methods Used for the Characterization of SRM 1632b

Page 4 SRM 1632b

#### Analytical Methods

- A. Atomic absorption spectrometry
- B. Isotope dilution mass spectrometry
- C. Instrumental neutron activation analysis
- D. Flame emission spectrometry
- E. Flameless atomic absorption spectrometry
- F. Ion chromatography
- G. ASTM Methods: (1)D3173, (2)D3174, (3)D3175, (4)D3177, (5)D3178, (6)D3179
- H. Combustion coulometry
- 1. Commercial coal analyzers: moisture, ash, sulfur, Btu, volatile matter, carbon, hydrogen, nitrogen
- J. Commercial calorimeter
- K. Gas chromatography
- L. X-ray fluorescence
- M. Inductively coupled plasma emission spectrometry

#### Analysts

NBS

E.S. Beary W.A. Bowman K.A. Brletic T.A. Butler J.D. Fassett J.W. Gramlich R.R. Greenberg W.F. Koch L.A. Machlan A. Marlow D.M. Mo T.J. Murphy P.J. Paulsen P. Pella T.C. Rains T.A. Rush T.A. Sleater S.F. Stone

### R. Zeisler

Laboratories

E. Huffman Huffman Laboratories Wheat Ridge, Colorado 80034

J.B. Bodkin College Earth & Mineral Science The Pennsylvania State University University Park, Pa. 16802

Page 5 SRM 1632b R. Peck Dickerson Laboratory El Paso, Texas 79912 National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis Standard Reference Material 1633a

### Trace Elements in Coal Fly Ash

This Standard Reference Material (SRM) is intended for use in the evaluation of analytical methods for the determination of constituent elements in coal fly ash or materials with a similar matrix.

SRM 1633a is a fly ash that was sieved through a No. 170 sieve with a nominal sieve opening of 90  $\mu$ m.

<u>Certified Values of Constituent Elements</u>: The certified values for the constituent elements are shown in Table 1. The analytical techniques used and the analysts are given in Table 3. The certified values are based on results obtained by reference methods of known accuracy or from two or more independent, reliable analytical methods. Noncertified values are given for information only in Table 2.

Notice and Warnings to Users: This certification is invalid 5 years from date of purchase of the SRM. The constituents certified or analyzed are reviewed periodically and may be updated to reflect improved measurement. Updated certificates will be made available upon request.

Use: This material should be dried to a constant weight before using. Recommended procedures for drying are: (1) Vacuum drying for 24 hours at ambient temperature using a cold trap at or below -50 °C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying for 2 hours in an oven at 105 °C; (3) drying in a dessicator over P<sub>2</sub>O<sub>5</sub> or Mg<sub>2</sub>ClO<sub>4</sub>. Samples of the dried material weighing at least 250-mg should be used for analysis. When not in use the material should be kept in a tightly sealed bottle.

Source and Preparation of Material: The fly ash material was supplied by a coal fired power plant and is a product of Pennsylvania and West Virginia coals. It was selected as a typical fly ash and is not intended as a fly ash from a specific coal or combustion process. The material was sieved and blended for 2 hours in a Vee blender. The material was then removed and placed in a series of bulk containers from which specific samples were taken for homogeneity testing and certification analysis. Twelve bottles were selected for the homogeneity test. Samples from each bottle were analyzed for cobalt, chromium, europium, iron, scandium, and thorium using nondestructive neutron activation analysis. The observed standard deviations for both 50 and 250 mg sample sizes were consistent with counting statistics, indicating that the fly ash is homogeneous within  $\pm$  5% (relative) based on these elements. The homogeneity testing and certification analyses were performed in the NBS Center for Analytical Chemistry.

The overall direction and coordination of the analytical measurements leading to the initial certification were performed in the Center for Analytical Chemistry under the chairmanship of L.A. Machlan.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W.P. Reed and T.E. Gills.

Gaithersburg, MD 20899 January 5, 1985 (Revision of certificate dated April 18, 1979) Stanley D. Rasberry, Chief Office of Standard Reference Materials

#### Table 1. Certified Values of Constituent Elements

Major	Content	Minor	Content
Constituents	Wt. Percent	Constituents	Wt. Percent
Aluminum Iron Potassium Silicon Calcium	$\begin{array}{rrr} 14.3 & \pm 1.0^{a} \\ 9.4 & \pm 0.1 \\ 1.88 \pm 0.06 \\ 22.8 & \pm 0.8 \\ 1.11 \pm 0.01 \end{array}$	Magnesium Sodium	$\begin{array}{r} 0.455 \pm 0.010 \\ 0.17 \pm 0.01 \end{array}$

# $\frac{\text{Trace Constituents}}{\text{Content } \mu g/g}$

Element	Content $\mu g/g$	Element	Content $\mu g/g$
Antimony	$6.8 \pm 0.4$	Rubidium	131 ± 2
Arsenic	145 ± 15	Selenium	$10.3 \pm 0.6$
Cadmium	$1.00 \pm 0.15$	Strontium	830 ± 30
Chromium	$196 \pm 6$	Thorium	$24.7 \pm 0.3$
Copper	118 ± 3	Thallium	$5.7 \pm 0.2$
Manganese	179 ± 8	Uranium	$10.2 \pm 0.1$
Mercury	$0.16 \pm 0.01$	Vanadium	<b>297</b> ± 6
Nickel	127 ± 4	Zinc	220 ± 10
Lead	724 + 04		

<sup>a</sup>The uncertainties of the certified values are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 250-mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents).

#### Supplemental Information

Note: The following values are not certified because they are not based on the results of either a reference method or of two or more independent methods. These values are included for information only.

#### Table 2. Noncertified Values for Constituent Elements

	Content		Content
Element	Wt. Percent	Element	<u> </u>
Barium	0.15	Beryllium	12
Titanium	0.8	Cerium	180
Sulfur	0.18	Cobalt	46
		Cesium	11
		Europium	4
		Gallium	58
		Hafnium	8
		Molybdenum	29
		Scandium	40

SRM 1633a Page 2

Method/ Element	А	В	С	D	E	F	G	н	I
Aluminum	•		•		[				•
Antimony			•				•		
Arsenic	•		•						
Cadmium		•	•	•			•		
Calcium	•	•			•				
Chromium	•	•	•						
Copper	9	•	•						
lron	~	•	•						
Lead		•		•	•				
Magnesium	•	•							
Manganese	•		•						•
Mercury	•		•						
Nickel	•	•		۰	•				
Potassium	•	•			•				
Rubidium	•	•	•		•				
Selenium	•		•				•		
Silicon					•			•	
Sodium	•		•						
Strontium	•				•	•			
Thallium		•					•		
Thorium		•	•						
Uranium		•							
Vanadium	o	•	•						
Zinc	•	•		•	•	•			

#### Table 3. Analytical Methods Used for Certified Constituent Elements

Analytical Methods

- A. Atomic Absorption Spectrometry or Flame Emission Spectrometry
- B. Isotope Dilution Mass Spectrometry
- C. Neutron Activation Analysis
- D. Polarography
- E. X-ray Fluorescence Spectrometry
- F. Inductively-Coupled Plasma Emission Spectrometry
- G. Isotope Dilution Spark Source Mass Spectrometry
- H. Gravimetry
- 1. Direct Coupled Plasma Emission Spectrometry

SRM 1633a Page 3

#### Analysts

NBS Center for Analytical Chemistry

J. B. Baldwin
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 M. G. Dias
 L. J. Powell
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- 17. H. M. Kingston
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- 20. L. A. Machlan
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- 24. L. J. Moore
- 25. P. J. Paulsen
- 26. P. A. Pella
- 27. T. C. Rains
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- 28. K. J. R. Rosman
- 29. T. A. Rush
- 30. P. A. Sleeth
- 31. R. L. Watters, Jr.

SRM 1633a Page 4

# National Bureau of Standards Certificate of Analysis

## Standard Reference Material 1634a

### **Trace Elements in Fuel Oil**

This Standard Reference Material is intended for use in the evaluation of methods and the calibration of apparatus used in the analysis of fuel oils and other materials with similar matrices for trace elements. SRM 1634a is a commercial "No. 6" residual fuel oil as defined by the American Society for Testing and Materials (ASTM). This SRM was certified using two or more independent methods of analysis and a single method that has been carefully evaluated with respect to its accuracy and precision. Methods were selected to include those that are commonly used in the field and in laboratories.

The certified values are given in table 1 and are based on at least a 1.0 g sample of the material which is the minimum amount that should be used for analysis.

Table 1									
Element <sup>1</sup>	Content <sup>2</sup> $\mu g/g$	Element <sup>1</sup>	Content <sup>2</sup> , Wt %						
Lead	$2.80 \pm 0.08$								
Manganese <sup>b, c</sup>	$0.19\pm0.02$	Sulfur <sup>f,g,h</sup>	$2.85 \pm 0.05$						
Nickel <sup>d</sup> , <sup>e</sup>	29 ± 1								
Selenium <sup>b, c</sup>	$0.15 \pm 0.02$								
Sodium <sup>b,e</sup>	87 ± 4								
Vanadium <sup>a,b,d</sup>	56 ± 2								
Zinc <sup>b,d</sup>	$2.7 \pm 0.2$								

- 1. Method of Analysis
  - a. Isotope Dilution Mass Spectrometry
- e. Inductive Coupled Plasma Spectrometry
- b. Neutron Activation Analysis
- f. Gravimetry
- c. Atomic Absorption Spectrometry
- g. Ion Chromatography
  h. X-ray Fluorescence
- d. Spark Source Mass Spectrometry
  h. X-ray Fluorescence
  2. The uncertainties shown are expressed at the 95% confidence level and include any observed
- The uncertainties shown are expressed at the 95% confidence level and include any observed material heterogeneity, possible method differences, and errors of measurement.

NOTICE: The certification of SRM 1634a is valid for 3 years from date of purchase.

The statistical analysis of the certification data was performed by K.R. Eberhardt of the Statistical Engineering Division.

The overall direction and coordination of the analytical measurements leading to certification were performed in the Inorganic Analytical Research Division, E.L. Garner, Chief.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Washington, D.C. 20234 February 19, 1982 George A. Uriano, Chief Office of Standard Reference Materials

#### PREPARATION, TESTING, AND ANALYSIS

A random scheme for sample selection was used in assessing the homogeneity of this material. The elements calcium and vanadium were measured by x-ray fluorescence as indicators of homogeneity. Based on these elements, the material variability for this lot of 1634a is within  $\pm 2\%$  relative.

Long-term stability of this SRM has not been rigorously established. When not in use, the material should be stored in the tightly sealed bottle. NBS will continue to monitor this material and any substantive change in its certification will be reported to the purchasers.

Analyses for the various elements were performed in the Center for Analytical Chemistry, Inorganic Analytical Research Division, by I. L. Barnes, T.A. Butler, E.R. Deardorff, J.W. Gramlich, S. Hanamura, H.M. Kingston, W.F. Koch, G.M. Lambert, R.M. Lindstrom, L.A. Machlan, J.R. Moody, P.J. Paulson, T.C. Rains, T.A. Rush, and R. Zeisler.

The homogeneity studies were performed in the Gas and Particulate Science Division by P.A. Pella and M. Watson.

The physical properties were measured by S. Weeks, Materials Chemistry Division, Center for Materials Science.

The values and physical properties data in table 2 are *not certified* because they are based on a non-reference method or were not determined by two or more independent methods. The values are included for information only.

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Supp	lementa	llni	ormation
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Element	Content, $\mu g/g$	Physical Properties	
Arsenic	( 0.12 )		
Beryllium	( 0.006 )	Flash Point <sup>a</sup>	64 °C
Bromine	(<1)		
Cadmium	( 0.002 )	Kinematic Viscosity <sup>b</sup>	321.66
Calcium	(16)	at 50 °C	
Chlorine	(31)	Pour Point <sup>e</sup>	-10 °C
Chromium	( 0.7 )		
Cobalt	( 0.3 )	Density at 20 °C <sup>d</sup>	$0.995 \text{ g/cm}^3$
lron	(31)		
Mercury	(<0.002)		
Molybdenum	( 0.12 )		

Methods Used for Physical Tests

a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester

b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids

c. ASTM D97-66 (1978) Pour Point of Petroleum Oils

d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

U.S Department of Commerce Juanita M, Kreps Secretary

National Bureau of Standards Ernest Ambler, Director

## National Bureau of Standards Certificate of Analysis Standard Reference Material 1635 Trace Elements in Coal (Subbituminous)

This Standard Reference Material is intended for use in the calibration of apparatus and the evaluation of techniques employed in the trace element analysis of coal and similar materials. The material should be dried without heat to constant weight before use.

The recommended procedures for drying are either vacuum drying at ambient temperature for 24 hours, or freeze drying in which the drying chamber is kept at room temperature. The moisture content of this material is approximately 20%. Because of this moisture level, it is recommended that small individual samples be dried immediately before use. Drying of large samples may result in a violent discharge of water vapor and resultant loss of sample. When not in use, the material should be kept in a tightly sealed bottle and stored in a cool, dark place. Long-term (>1 year) stability of this SRM has not been rigorously established. NBS will continue to monitor this material and any substantive change will be reported to purchasers.

The certified values given below are based on at least a 250-mg sample of the dried material, the minimum amount that should be used for analysis.

Element <sup>1</sup>	Content, $\mu g/g^2$	Element <sup>1</sup> Content, $\mu g/g$	
Arsenic <sup>a, b</sup>	$0.42 \pm 0.15$	Thorium <sup>c, e</sup>	$0.62 \pm 0.04$
Cadmium <sup>c, d, e</sup>	$0.03\pm0.01$	Uranium <sup>c</sup>	$0.24 \pm 0.02$
Chromium <sup>c, e</sup>	$2.5 \pm 0.3$	Vana <mark>diu</mark> m <sup>e, g</sup>	$5.2 \pm 0.5$
Copper <sup>a, c, e</sup>	$3.6 \pm 0.3$	Zinc <sup>c, d</sup>	$4.7 \pm 0.5$
Lead <sup>c,d</sup>	1.9 ± 0.2		
Manganese <sup>a, e</sup>	21.4 ± 1.5	Element <sup>1</sup>	Wt. $\%$ <sup>2</sup>
Nickel <sup>c,d</sup>	$1.74 \pm 0.10$	Iron <sup>c, d, e, f</sup>	$0.239 \pm 0.005$
Selenium <sup>a, e</sup>	$0.9 \pm 0.3$	Sulfur <sup>f,h</sup>	$0.33 \pm 0.03$

I. Methods of Analysis:

a. Atomic Absorption Spectrometry

e. Neutron Activation

b. Photon Activation

c. Isotope Dilution Mass Spectrometry

f. Spectrophotometry

g. Flame Emission Spectrometry

d. Polarography

h. Gravimetry

2. The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 250-mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

The overall direction and coordination of the analytical measurements leading to this certificate were performed in the Analytical Chemistry Division under the chairmanship of L. J. Moore.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234 August 22, 1979 (Revision of Certificate dated 1-23-78)

(over)

George A. Uriano Office of Standard Reference Materials

#### PREPARATION, TESTING, and ANALYSIS

This material was prepared from one lot of subbituminous coal from the Eagle Mine of The Imperial Coal Company, Erie, Colorado. The material was ground and sieved thru a No. 65 (230  $\mu$ m) sieve by the Colorado School of Mines Research Institute. The material was then blended in a V-type blender.

Samples for homogeneity testing were taken from the top, middle, and bottom of three bulk containers of coal, and analyzed by neutron activation analysis for sodium, scandium, chromium, iron, cobalt, lanthanum, cerium, and thorium. Replicate analyses of 250-mg samples indicated a homogeneity for these elements of  $\pm 2.5\%$  (relative) except for chromium, which was homogeneous within counting statistics of  $\pm 6\%$ . The homogeneity measurements were performed in the NBS Analytical Chemistry Division by R. R. Greenberg. Certification analyses for the various elements were made in the NBS Analytical Chemistry Division by T. J. Brady, B. I. Diamondstone, L. P. Dunstan, M. S. Epstein, M. Gallorini, E. L. Garner, T. E. Gills, J. W. Gramlich, R. R. Greenberg, S. H. Harrison, G. M. Hyde, G. J. Lutz, L. A. Machlan, E. J. Maienthal, J. D. Messman, T. J. Murphy, and T. C. Rains.

The following values are *not certified* because they were based on a non-reference method, o were not determined by two or more independent methods. They are included for information only.

(µg/g)
(0.14)
(3.6)
(0.65)
(0.06)
(1.05)
(0.29)
(0.63)
(wt. %)
(0.32)
(0.24)
(0.02)

U. S. Department of Commerce Philip M. Klutznick Secretary National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis Standard Reference Materials 1636a, 1637a, 1638a

### Lead in Reference Fuel

This Standard Reference Material is intended for use in the calibration of instruments and the evaluation of techniques used for the analysis of lead in gasoline. Samples of the reference fuel are supplied at four lead concentrations, nominally 0.03, 0.05, 0.07, and 2.0 g/gal. These Standard Reference Materials are made up of various combinations of the four concentrations, see Table 1 on the back of this certificate.

Certified Values: The certified values for the lead content, expressed in units of  $\mu g/g$ , are shown below. These certified values are based on results obtained by isotope dilution mass spectrometry, a definitive method of known accuracy.

	Nominal	Certified
Vial	Lead Concentration	Lead Concentration
<b>Identification</b>	g/gal	<u>μg/g</u>
I	0.03	$11.2 \pm 0.2^{a}$
II	0.05	$18.8 \pm 0.1$
III	0.07	$25.1 \pm 0.2$
1V	2.0	764 ± 4

<sup>a</sup>The uncertainties shown are the 95 percent confidence intervals for a single determination plus allowance for known sources of possible error.

Use: The certification of these materials is based on a minimum sample size of 1.0 gram and only samples equal to or greater than 1 gram should be used for any analytical determination to be related to the certified values of this certificate.

Stability: The ampoules should be stored at temperatures between 10-30 °C. They should not be exposed to intense sources of radiation, including ultraviolet lamps or sunlight. The ampoules should be opened only at time of use. No attempt should be made to keep the material in opened ampoules for future use.

Source and Preparation of Material: The reference fuel containing lead at the four concentration levels were supplied by Phillips Petroleum Company of Bartlesville, Oklahoma. The 91-octane number (Research Octane Number) reference fuel is a mixture of 91 percent by volume (0.899 mole-fraction) 2,2,4,-trimethylpetane and 9 percent by volume (0.101 mole-fraction) n-heptane. Lead was added in the form of tetraethyl lead motor mix.

Analyses leading to certification were performed in the Inorganic Analytical Research Division by T. J. Murphy and I. L. Barnes.

The overall direction and coordination of the technical measurements leading to this certificate were performed by E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of these Standard Reference Materials were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 February 5, 1980

(over)

George A. Uriano, Chief

Office of Standard Reference Material:

58

	Table 1	
	Composition of SRM's 1636a, 1637a	, 1638a
<u>SRM</u>	Nominal Concentration	<u>No. Units.</u>
1636a	0.03,0.05,0.07, 2.0 g/gal	3 vials each
1637a	0.03,0.05,0.07 g/gal	4 vials each
1638a	2.0 g/gal	12 vials each

Additional Information: Because the volume of the reference material varies with temperature, the various concentrations of lead are certified by weight, i.e., micrograms of lead per gram of fuel. For convenience to the user, information is given for the concentration in the customary units, grams per gallon and grams per liter, at 23 °C. These data are shown in Table 2.

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Vial	Nominal Concentration	Density <sup>a</sup> at 23 °C	Lead Conc at 23	Lead Concentration <sup>b</sup> at 23 °C	
<b>Identification</b>	g/gal	_g/mL	g/gal	g/L	
I	0.03	0.6888	0.0292	0.0077	
11	0.05	0.6888	0.0490	0.0129	
III	0.07	0.6888	0.0654	0.0173	
IV	2.0	0.6895	1.994	0.527	

<sup>a</sup> The density ( $\rho$ ) of each concentration was measured at 23 °C using a modification of ASTM Method D1217. The temperature coefficient of these materials is 0.0008 g.(mL)<sup>-T</sup>.(°C)<sup>-1</sup>

<sup>b</sup>The conversion of the certified values  $(\mu g/g)$  to C(g/gal) and C(g/L) was done using equations I and 2 respectively.

Eq. 1 
$$C_{g/gal} = \frac{3.785 \ \rho C \ \mu E/g}{10^3}$$
  
Eq. 2  $C_{g/L} = \frac{\rho C \ \mu E/g}{10^3}$ 



# National Bureau of Standards Certificate of Analysis Standard Reference Material 1641b Mercury in Water – µg/mL

This Standard Reference Material is intended for use in the primary calibration of instruments and techniques used for the determination of mercury in natural waters. It is designed for the preparation of calibration solutions and for use as a "spike" sample in a "method-of-additions" type analytical procedure.

Mercury concentration

 $1.52 \pm 0.04 \ \mu g/mL$ 

The estimated uncertainty,  $0.04 \,\mu g/mL$ , includes the effects of the observed random variability and an upper bound estimate of possible systematic errors. The random variability expressed as two standard deviations of the certified value is  $\pm 0.02 \,\mu g/mL$  and reflects both internal and between-method variabilities for the NBS atomic absorption and neutron activation measurements. The upper bound estimate of possible systematic errors is  $\pm 0.02 \,\mu g/mL$ .

Stability: The long-term stability of trace mercury solutions has been a constant problem. At or below the  $\mu g/mL$  level, mineral acid stabilization is not sufficient. However, the addition of trace gold to a nitric acid solution of mercury was found to stabilize the concentration of mercury in the two previous issues of this Mercury in Water SRM. Although the mercury concentration of SRM 1641b has not changed significantly in eight months, the stability will continue to be monitored. However, SRM 1641b should *not* be used after ONE YEAR FROM date of purchase.

<u>Precautions</u>: Traces of mercury vapor are present in most laboratory situations. Therefore, contamination of reagents, equipment, and common laboratory materials may cause a severe problem. Apparatus for analysis at this level must be scrupulously cleaned immediately before use, and only the purest-grade reagents, with respect to mercury, should be used.

SRM 1641b was prepared by J.R. Moody. Atomic absorption analyses were performed by T.C. Rains and T.A. Butler; and neutron activation analyses by R. Zeisler, Inorganic Analytical Research Division.

The overall direction and coordination of technical measurements leading to certification were performed under the chairmanship of E.L. Garner, Inorganic Analytical Research Division. The statistical evaluation was done by R.C. Paule.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Analytical: Two independent techniques were used in the certification of this Standard Reference Material: atomic absorption spectrometry and instrumental neutron activation analysis.

<u>Use:</u> This SRM consists of six ampoules, each containing approximately 20 mL of solution. Dilutions may be made by the addition of accurately measured aliquots, withdrawn from an ampoule, to known volumes of pure or natural water (spiking mode) using conventional techniques. Blank determinations should be made of the water and other reagents used.

The reliability of the dilution process will depend on the care exercised and the reliability of the calibration of the volumetric apparatus, which should have an uncertainty no greater than one percent. The volumetric apparatus should be scrupulosuly cleaned. Diluted solutions should be used without delay, as their stability cannot be guaranteed. SRM 1642b, which is certified for mercury at the ng/mL level, should be used to validate methodology for these concentrations. The long-term retention of unused portions of this Standard Reference Material in opened ampoules is not recommended.

Washington, D.C. 20234 April 13, 1903 George A. Uriano, Chief Office of Standard Reference Materials
U. S. Department of Commerce Malcolm Baldrige Secretary National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate Standard Reference Material 1642b

### Mercury in Water - ng/mL

This Standard Reference Material is intended for use in the primary standardization of instruments and techniques used for the determination of mercury in water. It is intended for use as received, without dilution or other alteration. The concentration of mercury in this Standard Reference Material is at, or near, the detection limit of most commercial instruments used for the determination of mercury in water. It is to be used for the primary standardization of these instruments near these detection limits where many analytical problems occur.

Mercury Concentration 1.49  $\pm$  0.06 ng/mL

The estimated uncertainty, 0.06, includes the effects of the observed random variability and an upper bound estimate of possible systematic errors. The random variability expressed as two standard deviations of the certified value is  $\pm 0.04$  and reflects both internal and between-method variabilities for the NBS atomic absorption and neutron activation measurements. The upper bound estimate of possible systematic errors is  $\pm 0.02$ .

<u>Stability:</u> Trace mercury solutions have been a constant problem when long-term storage is required. Below the  $\mu g_{c}$  mL level, mineral acid stabilization is not sufficient. A stabilizing technique has been applied to this Standard Reference Material that allows for prolonged storage. Gold, as the tetrachloride, has been added in a concentration 10 times that of the mercury. The gold ion, in conjuction with the normal mineral acid, has proven to be an effective stabilizer. It is recommended that this Standard Reference Material not be used after ONE YEAR FROM DATE OF PURCHASE.

<u>Precautions:</u> Traces of mercury vapor are present in most laboratory situations. Therefore, contamination of reagents, equipment, and common laboratory materials is a severe problem. Apparatus for analyses at this level must be scrupulously cleaned immediately before use, and only the purest-grade reagents should be employed. After use, the bottle should be capped tightly and placed inside the aluminized bag, which should be folded and sealed with a sealing tape. This safeguard will assist in maintaining the integrity of the sample.

<u>Analytical:</u> Two independent techniques were used in the certification of this Standard Reference Material: atomic absorption spectroscopy and neutron activation analysis.

Use: This Standard Reference Material should be used, as received, without dilution. It may be carried through the chemical manipulations required for the analytical procedure normally used for the analysis of natural waters.

This Standard Reference Material was prepared by J.R. Moody. Atomic absorption analyses were performed by 1.C. Rains and T.A. Butler and neutron-activation analyses were performed by R. Zeisler.

The overall direction and coordination of the technical measurements leading to the certification were performed under the chairmanship of E.L. Garner. The statistical evaluation was done by R.C. Paule.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234 July 15, 1982 George A. Uriano, Chief Office of Standard Reference Materials

## National Bureau of Standards

## Certificate

## Standard Reference Material 1643b

## Trace Elements in Water

This Standard Reference Material (SRM) is intended primarily for use in evaluating the accuracy of trace element determinations in filtered and acidified fresh water and for calibrating instrumentation used in these determinations. SRM 1643b consists of approximately 950 mL of water in a polyethylene bottle, which is sealed in an aluminized bag to maintain stability. SRM 1643b simulates the elemental composition of fresh water. Nitric acid is present at a concentration of 0.5 moles per liter to stabilize the trace elements.

<u>Concentrations of Constituent Elements</u>: The concentrations of the trace elements that were determined are shown in Table 1. The certified values are based on results obtained either by reference methods of known accuracy or by two or more independent, reliable analytical methods. Noncertified values, which are given for information only, appear in parentheses.

Notice and Warnings to Users:

Expiration of Certification: This certification is invalid two years after the shipping date.

<u>Precautions:</u> The bottle should be shaken before use because of possible water vapor condensation. To prevent possible contamination of the SRM, do not insert pipets into the bottle. After use, the bottle should be capped tightly and placed inside the aluminized bag, which should be folded and sealed with sealing tape. This safeguard will protect the SRM from possible environmental contamination and long-term loss of water.

Elemental determinations of ng/g levels are limited by contamination. Apparatus should be scrupulously cleaned and only the purest grade reagents employed. Sampling and manipulations, such as evaporations, should be done in a clean environment, for example, a Class 100 clean hood.

The overall direction and coordination of the technical measurements leading to this certification were performed under the direction of E. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, DC 20234 May 18, 1984 Stanley D. Rasberry, Chief Office of Standard Reference Materials

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(Table 1)		
Concentrations	of Constituent	Elements

Element	Concentration,* ng/g	Element	Concentration,* ng/g
Arsenic <sup>1,5</sup>	(49)**	Lead <sup>3,4b</sup>	$23.7 \pm 0.7$
Barium <sup>2a,2b,5</sup>	44 ± 2	Manganese <sup>1, 2a, 3</sup>	28 ± 2
Beryllium <sup>1,2a</sup>	19 ± 2	Molybdenum <sup>2a, 5</sup>	85 ± 3
Bismuth <sup>1</sup>	(11)	Nickel <sup>2a,3</sup>	49 ± 3
Boron <sup>2a</sup>	(94)	Selenium <sup>1,5</sup>	$9.7 \pm 0.5$
Cadmium <sup>2b,3,5</sup>	$20 \pm 1$	Silver <sup>1,5</sup>	$9.8 \pm 0.8$
Chromium <sup>4b</sup>	$18.6 \pm 0.4$	Strontium <sup>2a, 5</sup>	227 ± 6
Cobalt <sup>1,5</sup>	26 ± 1	Thallium <sup>4b</sup>	$8.0 \pm 0.2$
Copper <sup>3,4b</sup>	$21.9 \pm 0.4$	Vanadium <sup>4b</sup>	$45.2 \pm 0.4$
$\operatorname{Iron}^{2a, 4a, 5}$	99 ± 8	Zinc <sup>2a,5</sup>	66 ± 2

<sup>\*</sup> The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision and possible systematic errors among methods. To convert to nanograms per milliliter, multiply by the density of the SRM. The density at 23 °C is 1.017 grams per milliliter.

\*\* Values in parentheses are not certified.

1. Atomic absorption spectrometry, electrothermal	4. Isotopic dilution mass spectrometry,
2. Atomic emission spectrometry,	a. resonance ionization
a. dc plasma	b. thermal ionization
b. flame	5. Neutron activation,
3. Laser enhanced ionization flame spectrometry	instrumental

Source and Preparation of Material: SRM 1643b was prepared at the U.S. Geological Survey, National Water Quality Laboratory, Arvada, Colorado, under the direction of V.J. Janzer of that laboratory and J.R. Moody of the NBS Center for Analytical Chemistry. Only high-purity reagents were used and the containers were acid-cleaned and sterilized before use. In the preparation, a polyethylene cylindrical tank was filled with distilled water and sufficient nitric acid to make the solution approximately 0.5 moles HNO<sub>3</sub> per liter. Solutions containing known amounts of calcium, sodium, magnesium, potassium, and the elements to be determined were added to the acidified water solution with constant stirring. After thoroughly mixing, the solution was filtered, sterilized, and then transferred to one-liter polyethylene bottles. The approximate concentrations, in  $\mu g/mL$ , of Ca, Na, Mg, and K are respectively 35, 8, 15, and 3.

#### Analysts:

Center for Analytical Chemistry, National Bureau of Standards

1.	K. A. Brletic	10. J. R. Moody
2.	T. A. Butler	11. L. J. Powell
3.	E. C. Deal	12. T. C. Rains
4.	M. S. Epstein	13. T. A. Rush
5.	J. D. Fassett	14. S. F. Stone
6.	K. Fitzpatrick	15. G. C. Turk
7.	H. M. Kingston	16. R. L. Watters, Jr.
8.	R. M. Lindstrom	17. R. Zeisler
9.	L. A. Machlan	

U. S. Department of Commerce Malcolms Baldrige Secretary National Burnu of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate

## Standard Reference Material 1644 Generator Columns for Polynuclear Aromatic Hydrocarbons

SRM 1644 is intended to provide accurate concentrations of anthracene, benzo(a) anthracene (1,2-benzanthracene), and benzo(a)pyrene (3,4-benzpyrene) in water. The SRM consists of three 50 cm x 0.6 cm (coiled) stainless steel tubes, each packed with fine quintus quartz (sea sand) coated with approximately 0.5 percent by weight of the polynuclear aromatic hydrocarbon (PAH) of interest.

Principle of Operation: A saturated aqueous solution of the PAH of interest is generated by flowing high-purity water slowly through the column. Because the aqueous solubility of a compound is a well-defined thermodynamic quantity, a saturated solution has a fixed concentration (1, 2, 3).

Equilibration and Use of Generator Columns: To equilibrate a new column before initial use, purge with high-purity water, such as commercial HPLC grade water. The volume required for equilibration of each column is: 500 mL for anthracene, 1000 mL for benzo(a)anthracene, and 500 mL for benzo(a)pyrene. After equilibration, pump the high-purity water at a constant temperature ( $\pm 0.1$  °C) through the column at a flow rate between <u>0.1 and 5 mL/min</u> to produce a saturated solution. Record the temperature. The solution should be used immediately after generation to avoid sorption losses.

If either the temperature is changed by as much as  $1 \,^{\circ}$ C or the flow is interrupted for <u>less than one hour</u>, pump 25 mL water through the column under the new conditions to restore equilibrium prior to sample collection. However, if the flow is interrupted for <u>more than one hour</u>, pump 50 mL water prior to sample collection. During periods of frequent use, a column can be kept equilibrated by maintaining a steady, but low, flow rate of approximately 0.1 mL/min through the column. The flow rate can be increased to collect a large volume of sample and then decreased again. Columns should be purged with 10 liters of oil-free nitrogen prior to storage periods of more than one month.

<u>Certified Concentrations</u>: When used as directed, these columns generate saturated solutions. The concentrations of the compounds in these solutions at temperatures between 10 and 30 °C were determined by two independent analytical methods. The data obtained were combined by fitting an empirical expression of the form  $\ln[\text{Conc}] = A + B(1/T) + C(1/T^2)$  by least squares. In this equation,  $\ln[\text{Conc}]$  is the natural logarithm of the concentration, T is the absolute temperature, and A, B, and C are constants for each compound. The derived equations for anthracene, benzo(a)anthracene, and benzo(a)pyrene were used to calculate the certified concentrations at one degree intervals between 10 and 30 °C. These certified concentrations are given in Tables I-III.

<u>Service Life of Columns</u>: Generator columns for anthracene, benzo(a)anthracene, and benzo(a)pyrene are certified for either two years or for total aqueous purge volumes of  $7.5 \times 10^2$ ,  $3 \times 10^3$ , and  $1.5 \times 10^4$  liters, respectively, whichever comes first.

Consultation on the statistical design of the experimental work and statistical analysis of the data was provided by K. R. Eberhardt of the Statistical Engineering Division. Coordination of the technical measurements leading to certification was performed by W. E. May, R. A. Velapoldi, and H. S. Hertz. Technical measurements leading to the development and certification of SRM 1644 were performed by the following members of the Center for Analytical Chemistry: W. E. May, J. M. Brown-Thomas, W. J. Sonnefeld, R. A. Velapoldi, and P. A. White.

The technical and support aspects concerning the certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234 April 27, 1981 George A. Uriano, Chief Office of Standard Reference Materials

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The anthracene generator column, after equilibration, produces aqueous solutions that contain very small amounts of phenanthrene. Because of its relatively high solubility (1000  $\mu$ g/kg), residual amounts of phenanthrene are depleted from the column at a rapid rate.

The benzo(a) anthracene generator column, after equilibration, produces aqueous solutions that contain as much as  $0.3 \mu g/kg$  anthracene in addition to the certified concentrations of benzo(a) anthracene. The concentration of anthracene in the generator column effluent varies with time (volume) and, therefore, is not certified.

The benzo(a)pyrene generator column, after equilibration, produces aqueous solutions that contain as much as 0.1  $\mu$ g/kg benzo(a)anthracene and 1  $\mu$ g/kg chrysene in addition to the certified concentrations of benzo(a)pyrene. The concentration of these non-analyte components vary with time and, therefore, are not certified.

Analyses of the Saturated Aqueous Solutions: The concentrations of the saturated aqueous solutions of anthracene, benzo(a)anthracene, and benzo(a)pyrene in the effluent from the respective generator columns were determined by two independent analytical techniques. The first technique was high-performance liquid chromatography (HPLC). It involved quantitative extraction of the PAH of interest from the aqueous effluent by an "extractor column" packed with an octadecylsilane (C<sub>18</sub>) bonded phase; use of an acetonitrile-water eluant to transfer components from the extractor column to an analytical C<sub>18</sub> column for separation of the analyte from non-analyte components; and detection of the analyte by measuring its absorbance at 254 nm. The second technique involved the use of a "standard addition" spectrofluorimetric technique for "on stream" analysis. The aqueous effluent from the generator column was mixed with PAH standards dissolved in acetonitrile and the PAH concentration of the resultant mixture was determined by fluorescence at the following excitation ( $\lambda_{ex}$ ) and emission ( $\lambda_{em}$ ) wavelengths: anthracene,  $\lambda_{ex} = 254$  nm,  $\lambda_{em} = 384$  and 404 nm; benzo(a)anthracene,  $\lambda_{ex} = 290$  nm,  $\lambda_{em} = 395$  nm; benzo(a)pyrene,  $\lambda_{ex} = 296$  nm,  $\lambda_{em} = 414$  nm. The PAH effluent concentration was determined mathematically. Where necessary, corrections for inner filter effects or the emission-absorbance contributions by impurities were made to obtain the values for determining the certified PAH concentrations listed in Tables I-III.

#### References:

- 1. May, W. E., The Solubility Behavior of Polycyclic Armomatic Hydrocarbons in Aqueous Systems, American Chemical Society Advances in Chemistry Series <u>185</u> (7), 143-192 (1980).
- 2. May, W. E., Wasik, S. P., and Freeman, D. H., Determination of the Aqueous Solubility of Polynuclear Aromatic Hydrocarbons by a Coupled-Column Liquid Chromatographic Technique, Anal. Chem. <u>50</u>, 1 (1978).
- 3. Schwarz, F. P. and Miller, J. M., Determination of the Aqueous Solubilities of Organic Liquids at 10°C, 20°C, and 30 °C by Elution Chromatography, Anal. Chem. <u>52</u>, 2162-2164 (1980).

°C	Concentration and I µg/kg	ts Uncerta	ainty', ol/L
10	$166 \pm 0.7$	03.1	+ 40
11	$17.6 \pm 0.7$	09.7	+ 26
12	$17.0 \pm 0.0$	105	⊥ J.0 ⊥ J.1
12	18.7 ± 0.0	105 :	± 3.3
13	$19.8 \pm 0.5$	111 :	± 3.1
14	$21.1 \pm 0.5$	118 :	± 2.9
15	$22.4 \pm 0.5$	126 :	± 2.9
16	$23.8 \pm 0.5$	134 :	± 2.9
17	$25.4 \pm 0.5$	142 :	± 2.9
18	$27.0 \pm 0.5$	151 :	± 3.0
19	$28.8 \pm 0.5$	161 :	± 3.1
20	$30.7 \pm 0.6$	172 :	± 3.2
21	$32.8 \pm 0.6$	184 :	± 3.2
22	$35.0 \pm 0.6$	196 :	± 3.2
23	$37.4 \pm 0.6$	210 :	± 3.2
24	$39.9 \pm 0.6$	224 :	± 3.3
25	$42.7 \pm 0.6$	239 :	± 3.7
26	$45.7 \pm 0.8$	256 :	± 4.4
27	$48.9 \pm 1.0$	273	± 5.5
28	$52.4 \pm 1.3$	293 :	± 7.2
29	$56.1 \pm 1.7$	313 :	± 9.5
30	$60.1 \pm 2.2$	336 :	± 12

Table I. Certified Aqueous Concentrations of Anthracene and Their Uncertainties, in Micrograms/Kilogram and Nanomoles/Liter, as a Function of Temperature

<sup>1</sup>The uncertainties are 99 percent (Working-Hotelling) confidence bands for the entire regression curve. The difference between the true and certified concentrations should be less than the stated uncertainty at the 99 percent confidence level.

Temperature	Concentration and It	s Uncertainty',
<u>°C</u>	μg/kg	nmol/L
10	$3.38 \pm 1.2$	14.8 ± 5.3
11	$3.60 \pm 1.1$	$15.8 \pm 4.6$
12	$3.83 \pm 0.91$	$16.8 \pm 4.0$
13	$4.09 \pm 0.79$	17.9 ± 3.4
14	$4.36 \pm 0.68$	$19.1 \pm 3.0$
15	$4.65 \pm 0.59$	20.4 ± 2.6
16	$4.96 \pm 0.54$	21.7 ± 2.4
17	$5.29 \pm 0.55$	$23.2 \pm 2.4$
18	$5.65 \pm 0.60$	24.8 ± 2.6
19	$6.04 \pm 0.68$	$26.4 \pm 3.0$
20	$6.45 \pm 0.77$	$28.2 \pm 3.4$
21	$6.90 \pm 0.87$	$30.2 \pm 3.8$
22	$7.38 \pm 0.94$	$32.3 \pm 4.1$
23	$7.90 \pm 1.0$	34.5 ± 4.4
24	$8.45 \pm 1.0$	36.9 ± 4.5
25	$9.05 \pm 1.0$	39.5 ± 4.6
26	$9.69 \pm 1.0$	42.3 ± 4.6
27	$10.4 \pm 1.1$	45.4 ± 4.7
28	$11.1 \pm 1.2$	$48.4 \pm 5.0$
29	$11.9 \pm 1.3$	51.9 ± 5.9
30	12.8 ± 1.7	55.8 ± 7.3

 Table II.
 Certified Aqueous Concentrations of Benzo(a)anthracene and Their Uncertainties, in Micrograms/Kilogram and Nanomoles/Liter, as a Function of Temperature

<sup>1</sup>The uncertainties are 99 percent (Working-Hotelling) confidence bands for the entire regression curve. The difference between the true and certified concentrations should be less than the stated uncertainty at the 99 percent confidence level.

Temperature	Concentration and Its	S Uncertainty',
°C	μg/kg	nmol/L
10	$0.59 \pm 0.06$	$2.34 \pm 0.23$
11	$0.63 \pm 0.05$	$2.50 \pm 0.20$
12	$0.67 \pm 0.04$	$2.65 \pm 0.17$
13	$0.71 \pm 0.04$	$2.81 \pm 0.15$
14	$0.76 \pm 0.03$	$3.01 \pm 0.12$
15	$0.81 \pm 0.03$	$3.21 \pm 0.11$
16	$0.87 \pm 0.03$	$3.44 \pm 0.10$
17	$0.93 \pm 0.03$	$3.68 \pm 0.10$
18	$0.99 \pm 0.03$	$3.92 \pm 0.11$
19	$1.06 \pm 0.03$	$4.19 \pm 0.12$
20	$1.13 \pm 0.03$	$4.47 \pm 0.13$
21	$1.21 \pm 0.04$	$4.79 \pm 0.15$
22	$1.30 \pm 0.04$	$5.14 \pm 0.16$
23	$1.39 \pm 0.04$	$5.50 \pm 0.16$
24	$1.49 \pm 0.04$	5.89 ± 0.17
25	$1.59 \pm 0.04$	$6.28 \pm 0.17$
26	$1.71 \pm 0.04$	$6.76 \pm 0.18$
27	$1.83 \pm 0.05$	$7.23 \pm 0.19$
28	$1.96 \pm 0.05$	$7.74 \pm 0.22$
29	$2.11 \pm 0.07$	$8.33 \pm 0.27$
30	$2.26 \pm 0.09$	8.92 ± 0.35

Table III. Certified Aqueous Concentrations of Benzo(a)pyrene and Their Uncertainties, in Micrograms/Kilogram and Nanomoles/Liter, as a Function of Temperature

<sup>1</sup>The uncertainties are 99 percent (Working-Hotelling) confidence bands for the entire regression curve. The difference between the true and certified concentrations should be less than the stated uncertainty at the 99 percent confidence level.

U. S. Department of Commerce Malcolm Baldrige Secretary

National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis Standard Reference Material 1645

### **River Sediment**

This Standard Reference Material (SRM) is intended for use for the calibration of apparatus and the vermeation of methods used in the analysis of river sediments and material with a similar matrix.

<u>Certified Values of Constituent Elements:</u> The certified values for the constituent elements are shown in Table 1. The analytical techniques used and the names and affiliations of the analysts are shown in Table 3. Certified values are based on results obtained by reference methods of known accuracy and analyses performed by two or more analysts; or alternatively, from results obtained by two or more independent, reliable analytical methods. Noncertified values are given for information only in Table 2. All values are based on measurements made on a dried sample of at least 100 mg for all constituents except iron and chromium for which a 1-g sample was used.

#### Notice and Warnings to Users:

Expiration of Certification: This certification is invalid 5 years from the date of purchase.

Stability: This material has been freeze-dried and is essentially free of moisture. However, its stability has not been rigorously assessed. NBS will continue to monitor this material and if substantive changes in certification occur the purchasers will be notified. The material should be kept in its original bottle and stored at temperatures between 10-30 °C. The material should be dried without heat to a constant weight before using. Recommended procedures for drying are: (1) drying for 24 hours using a cold trap at or below -50 °C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying for 24 hours in a desiccator over P<sub>2</sub>O<sub>5</sub> or Mg (ClO<sub>4</sub>)<sub>2</sub>.

Use: Material of this kind is intrinsically heterogeneous. Consequently, the analyst should endeavor to minimize any segregation by thoroughly mixing the contents of the bottle by shaking and/or rolling before each use. In addition, when taking a portion for analysis, the analyst should strive to remove as representative a sample as possible.

Source and Preparation of Material: The material for this SRM was prepared from material dredged from the bottom of the Indiana Harbor Canal near Gary, Indiana. The material was screened to remove foreign objects, freeze-dried, and sieved to pass a No. 80 (180  $\mu$ m) screen. The material was thorougly mixed in a V-blender and bottled. The bulk material was radiation-sterilized to minimize alteration due to biological activity.

The collection, freeze-drying and homogenization of this SRM were performed under the supervision and direction of H.L. Rook, Gas and Particulate Science Division.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J.K. Taylor, Center for Analytical Chemistry.

The technical and support aspects involved in the preparation, current and previous certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills and W.P. Reed.

Washington, D.C. 20234 May 5, 1982 (Revision of Certificate dated 11-16-78) George A. Uriano, Chief Office of Standard Reference Materials

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Homogeneity Assessment and Certification: The homogeneity of this material was established using a minimum sample size of 100 milligrams for all constituents except iron and chromium for which the sample size was 1.0 gram.

Randomly selected bottles were used for the analytical measurements. Each analyst examined at least 6 different bottles, some of them measuring replicate samples from each bottle. Accordingly, it is believed that all bottles of this SRM have substantially the same composition. Measurements and calibrations were made to reduce random and systematic errors to no more than one percent, relative.

Major Co	onstituents	Minor Co	onstituents
Element	Content wt. percent <sup>a</sup>	Element	Content wt. percent <sup>a</sup>
Aluminum <sup>b</sup>	$2.26 \pm 0.04$	Magnesium <sup>b</sup>	$0.74 \pm 0.02$
Chromium	$2.96 \pm 0.28$	Sodium	$0.54 \pm 0.01$
lron	$11.3 \pm 1.2$	Zinc	$0.172 \pm 0.017$
Potassium <sup>b</sup>	$1.26 \pm 0.05$		
	Trace Co	nstituents	
	Content		Content
Element	$\mu g/g^a$	Element	$\mu g/g^a$
Cadmium	$10.2 \pm 1.5$	Nickel	45.8 ± 2.9
Copper	109 ±19	Thallium	$1.44 \pm 0.07$
Cobalt <sup>b</sup>	$10.1 \pm 0.6$	Thorium	$1.62 \pm 0.22$
Lead	$714 \pm 28$	Uranium	$1.11 \pm 0.05$
Manganese	785 ±97	Vanadium	23.5 ± 6.9

Table 1. Certified Values of Constituent Elements

"The uncertainties of the certified values for the elements, except those noted by superscript "b", include those errors associated with both measurement and material variability. They represent the 95 percent tolerance limits for an individual sub-sample, i.e., 95 percent of the sub-samples from a unit of this SRM would be expected to have a composition within the indicated range of values 95 percent of the time.

 $1.1 \pm 0.5$ 

Mercury

<sup>b</sup>These elements are certified as a part of the NBS update certification program. For each element a "best value" is given based on all methods of measurement that were used as well as a *standard error* of this value. Both are based on considerations of variability both within and between analytical methods.

#### Supplemental Information

Note: The following values are not certified because they are not based on the results of either a reference method or of two or more independent methods. These values are included for information only.

	Table 2. Noncertified Valu	es for Constituent Elements	
Element	Content wt. Percent	Element	Content µg/g
Calcium	(2.9)	Antimony	(51)
Fluorine	(0.09)	Arsenic	(66)
Sulfur	(1.1)	Lanthanum	(9)
		Scandium	(2)
		Selenium	(1.5)

Additional Information: The values listed below are based on measurements made in one laboratory and while no reason exists to suspect systematic bias in these numbers, no attempt was made to evaluate such bias attributable to either the method or the laboratory. The method used for each set of measurements is also listed. The indicated uncertainties are two times the standard deviation of the mean. These values are included for information only.

#### Table 3

	Content
Constituent	wt. percent
Kjeldahl Nitrogen	$(0.0797\% \pm 0.0048)$
Total Phosphorus	$(0.051\% \pm 0.001)$ [1]
Loss on Ignition (800 °C)	$(10.72\% \pm 0.28)$
Oil and Grease (Freon)	( 1.71% ± 0.26) [3]
Chemical Oxygen Demand (Dichromate)	$(149,400 \text{ mg/kg} \pm 9,000)$ [2]

#### References

I. ASTM Method E-350

2. Standards Methods for the Examination of Water and Waste Water, 14th Edition (1975), Section 508, pp 550.

3. Ibid., Section 502, pp 518.

#### lable 3A Methods and Analysts

Method / Element	А	В	С	D	Е	F
Aluminum	٠		•		•	
Arsenic			•			
Antimony	•	1				
Cadmium			•	•		
Calcium			•			
Chromium		•	٠			
Cobalt			•		•	
Copper		•	•			
Fluorine						•
Iron	•		٠			
Lanthanum			•			
Lead		•		•		
Magnesium	•				•	
Manganese		9	•			
Manganese Mercury	6	9	•			
Manganese Mercury Nickel	0	•	•	•		
Manganese Mercury Nickel Potassium	•	•	•	•		
Manganese Mercury Nickel Potassium Scandium	•	•	•	•		
Manganese Mercury Nickel Potassium Scandium Selenium	•	•	•	•		
Manganese Mercury Nickel Potassium Scandium Selenium Sodium	•	•	•	•		
Manganese Mercury Nickel Potassium Scandium Selenium Sodium Sultur	•	•	•	•		•
Manganese Mercury Nickel Potassium Scandium Selenium Sodium Sultur Thallium	•	•	•	•		•
Manganese Mercury Nickel Potassium Scandium Selenium Sodium Sultur Thallium Thallium	•	•	•	•		•
Manganese Mercury Nickel Potassium Scandium Selenium Sodium Sultur Thallium Thorium Uranium	•	•	•	•		
Manganese Mercury Nickel Potassium Scandium Selenium Sodium Sultur Thallium Thallium Thorium Uranium Vanadium	•	•		•		•

Analytical Methods

- A. Atomic Absorption Spectrometry
- B. Isotope Dilution Mass Spectrometry
- C. Neutron Activation Analysis
- D. Polarography
- E. D. C. Plasmas Atomic Emission Spectrometry
- F. Ion Chromatography

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6. M. Gallorini	15. T. C. Rains
7. E. L. Garner	16. H. L. Rook
8. T. E. Gills	17. T. A. Rush
9. J. W. Gramlich	18. W. P. Schmidt

#### Cooperators

19. L. Kosta (Nuclear Chemistry Section, Josef Stefen Institute, Ljubljana, Yugoslavia

# National Bureau of Standards

## Certificate of Analysis

## Standard Reference Material 1646

## **Estuarine Sediment**

This Standard Reference Material is intended primarily for calibrating instrumentation and evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in sediments, and similar matrices.

<u>Values of Constituent Elements:</u> The certified values for the constituent elements are shown in Table 1. They are based on results obtained either by definitive methods or by two or more independent, reliable analytical methods. *Non*certified values, which are given for information only, appear in Table 2. All values are based on a minimum sample size of 500 mg of the material dried as indicated under "Instructions for Drying".

#### Notice to Users:

Expiration of Certification: The certification of this SRM will be invalid 5 years after date of shipping.

<u>Use</u>: The material should be kept in its original bottle and shaken well before each use. A minimum sample of 500 mg of the dried material (see Instructions for Drying) should be used for any analytical determination to be related to a certified value of this certificate.

Statistical consultation was provided by K. R. Eberhardt of the Statistical Engineering Division.

The overall direction and coordination of the technical measurements leading to certification were performed in the Inorganic Analytical Research Division, E. L. Garner, Chief.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234 June 7, 1982 (Revision of Certificate dated 1-6-82)

(over)

George A. Uriano, Chief Office of Standard Reference Materials

Floment	Concentration,	Element	Concentration,		
Element	weight %	le 2	weight %		
Aluminum <sup>20, c; 6</sup>	$6.25 \pm 0.20$	Magnesium	$1.09 \pm 0.08$		
Calcium <sup>26,c;6</sup>	$0.83 \pm 0.03$	Phosphorus <sup>2a;6</sup>	$0.054 \pm 0.005$		
lron <sup>2c;4a;6</sup>	$3.35 \pm 0.10$				
	Concentration,		Concentration,		
Element	μg/g	Element	<u>μg/g</u>		
Arsenic <sup>1d;4b</sup>	11.6 ± 1.3	Manganese <sup>1c;2c</sup>	375 ± 20		
Cadmium <sup>1b,3a,b;4b</sup>	$0.36 \pm 0.07$	Mercury <sup>1a;4b</sup>	$0.063 \pm 0.012$		
Chromium <sup>1c;3b;4a</sup>	$76 \pm 3$	Nickel <sup>1b;2c;5</sup>	32 ± 3		
Cobalt <sup>1b;4a</sup>	$10.5 \pm 1.3$	Vanadium <sup>2a, 3a</sup>	94 ± 1		
Copper <sup>1c;2c;4b</sup>	18 ± 3	Zinc <sup>1b,c;2c;3b;5</sup>	138 ± 6		
Lead <sup>1b;3a;5</sup>	$28.2 \pm 1.8$				
1. Atomic absorption spectro	ometry	3. Isotope dilution	mass spectrometry		
a. cold vapor		a. thermal ionizatio	n		
b. graphite furnace		b. spark source			
c. flame		4. Neutron activation			
d. hydride generation		a. instrumental			
2. Atomic emission spectrometry		b. radiochemical			
a. dc plasma		5. Polarography			
b. flame		6. X-ray fluorescence spectrometry			
c. inductively coupled plas	sma				

#### Table 1. Certified Concentration of Constituent Elements

Notes: (1.) Analytical values are based on the "dry-weight" of material (see Instructions for Drying). Mercury should be determined on samples without drying and the results adjusted to a "dry-weight" basis by determining the moisture content of separate samples.

(2.) The estimated uncertainty for an element is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples 500 mg or more.

#### Table 2. Non-certified Concentrations of Constituent Elements

Note: The values shown in this table are not certified because they are not based on the results of either a definitive method or two or more independent analytical methods. These values are included, for information only, to provide additional information on the composition.

Element	Concentration, Weight %	Element	Concentration, Weight %
Potassi <b>um</b>	(1.4)	Sulfur	(0.96)
Silicon	(31)	Titanium	(0.51)
Sodium	(2.0)		
	Concentration,		Concentration,
Element	<u>μ</u> g/g	Element	<u> </u>
Antimony	(0.4)	Molybdenum	(2.0)
Beryllium	(1.5)	Rubidium	(87)
Cerium	(80)	Scandium	(10.8)
Cesium	(3.7)	Selenium	(0.6)
Europium	(1.5)	Tellurium	(0.5)
Germanium	(1.4)	Thallium	(0.5)
Lithium	(49)	Thorium	(10)

#### Analysts:

Inorganic Analytical Research Division, National Bureau of Standards. I. L. Barnes, M. B. Blackburn, C. G. Blundell, 1. A. Butler, M. S. Epstein, T. E. Gills, J. W. Gramlich, R. R. Greenberg, S. Hanamura, W. R. Kelly, H. M. Kingston, L. Machlan, E. J. Maienthal, J. D. Messman, T. J. Murphy, T. C. Rains, T. A. Rush, R. Sedivy, and R. L. Watters, Jr.

Cooperating Analysts:

University of Tokyo, Tokyo, Japan; present address: Meteorological Research Institute; Tsukuba, Ibaraki, Japan; Y. Dokiya (NBS Guest Worker).

Division of Chemistry, National Research Council of Canada, Ottawa, Canada; S. Berman, A. Desaulniers, R. Sturgeon, A. Mykytuik, J. McLaren, V. Boyko, and P. Semeniuk.

Instructions for Drying: Except for mercury, elements should be determined on samples that have been dried at 110°C for 2 hours.

Mercury should be determined on undried samples. However, because the certified concentration is reported on a "dryweight" basis, the concentration determined on undried samples should be adjusted for the moisture content of the samples.

Source and Preparation of Material: The material for this SRM was supplied by R. Huggett, Virginia Institute of Marine Sciences, Gloucester Point, Va. It had been dredged from the Chesapeake Bay at a location:  $37^{\circ}$  11.1'N,  $76^{\circ}$  17.1'W. The material was freeze-dried at Eastern Freeze-Dry Corporation, Lancaster, Pa., and radiation sterilized at Neutron Products Inc., Dickerson, Md. At NBS, the sediment was sieved through a screen with openings of 1.00 mm (No. 18) to remove coarse contaminants; ball-milled to pass a sieve with openings of 150  $\mu$ m (No. 100); thoroughly mixed in a V-blender; placed in polyethylene bags; and bottled.

<u>Homogeneity Assessment:</u> A preliminary evaluation of homogeneity was made by instrumental neutron activation using samples of approximately 250 mg taken from various locations of the bulk materials. The samples were irradiated and the activities from radionuclides of Ce, Co, Cr, Cs, Eu, Fe, Rb, Sc and Th were counted. Except for Ce and Th, the observed sample-to-sample variations for the elements were approximately the same as the counting statistics indicating satisfactory homogeneity for these elements within approximately 2%. The homogeneity of the material for As, Cd, Hg, N, and Zn was evaluated by various analytical techniques using samples weighting 250 to 300 mg and found to be satisfactory. The homogeneity of the remaining certified elements was determined using sample weights not exceeding one gram.

The uncertainties of the elemental concentrations in Table 1 take into account possible material inhomogeneity for samples weighing 500 mg.

U. S. Department of Commerce Malcolm Baldrige Secretary National Bureau of Standards Ernest Ambler, Director

## National Bureau of Standards Certificate of Analysis Standard Reference Material 1648 Urban Particulate Matter

This Standard Reference Material (SRM) is intended for use in the calibration of apparatus and evaluation of methods used in the analysis of atmospheric particulate matter and materials with a similar matrix.

<u>Certified Values of Constituent Elements:</u> The certified values for the constituent elements are shown in Table 1. The analytical techniques used in the certification are shown in Table 3. The certified values are based on measurements of 6 to 30 samples by each of the analytical techniques indicated. Noncertified values are given for information only in Table 2.

Notice and Warnings to Users: This material is a naturally occuring urban dust to be used for analytical purposes only. It may contain a number of chemicals of unknown toxicities, therefore, the utmost caution and care must be exercised in its use.

Expiration of Certification: This certification is invalid after 5 years from date of purchase. Should it be shown to be invalid prior to that time, users will be notified by NBS.

Stability: This material should be kept in its original bottle and stored at temperatures between 10-30 °C. It should not be exposed to intense source of radiation, including ultraviolet lamps or sunlight. Ideally, the bottle should be kept in a desiccator at the recommended temperature.

Use: A minimum of 100 mg of the dried material (See Drying Instructions) should be used for any analytical determination to be related to the certified values of this certificate.

Source and Preparation of Material: This SRM was prepared from urban particulate matter collected in the St. Louis, Missouri, area in a baghouse specially designed for this purpose. The material was removed from the filter bags and combined in a single lot. This product was screened through a fine-mesh sieve to remove extraneous materials and thoroughly blended in a V-blender. The material was then bottled and sequentially numbered. The material was collected over a period in excess of 12 months and, therefore, is a time-integrated sample. While not represented to be typical of the area in which it was collected, its use should typify the analytical problems of atmospheric samples obtained from industrialized urban areas.

<u>Homogeneity Assessment</u>: Randomly selected bottles were used for the analytical measurements. Each analyst examined at least 6 bottles, some of them measuring replicates from each bottle. No correlation was found between measured values and the bottling sequence. Also, the results of measurements of samples from different bottles were not significantly different than the measurements of replicate samples from single bottles. Accordingly, all bottles of this SRM have been assigned the same certified values of constituent elements.

Instructions for Drying: This material should be dried at  $105 \,^{\circ}$ C for 8 hours before use. Because the certified concentrations are reported on a "dry-weight" basis, the concentrations determined on undried samples should be adjusted for the moisture content of the samples.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J.K. Taylor.

The technical and support aspects involved in the preparation, current and previous certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills and W.P. Reed.

Washington, D.C. 20234 May 11, 1982 (Revision of Certificate dated 11-16-78)

(over)

George A. Uriano, Chiel Office of Standard Reference Materials

Major Constituent	<u>ts</u>	Minor Constituents	
	Content <sup>a</sup>		Content <sup>a</sup>
Element	Wt. Percent	Element	Wt. Percent
Aluminum <sup>b</sup>	$3.42 \pm 0.11$	Lead	$0.655 \pm 0.008$
lron	$3.91 \pm 0.10$	Sodium <sup>b</sup>	$0.425 \pm 0.002$
Potassium <sup>b</sup>	$1.05 \pm 0.01$	Zinc	$0.476 \pm 0.014$

#### Table 1. Certified Values of Constituent Elements

#### Trace Constituents

	Co	ontent <sup>a</sup>		Co	ntent <sup>a</sup>
Element		ug/g	Element	<u> </u>	<u>g/g</u>
Arsenic	115	± 10	Nickel	82	±3
Cadmium	75	± 7	Selenium <sup>b</sup>	27	±1
Chromium	403	± 12	Uranium	5.5	$\pm 0.1$
Copper	609	± 27	Vanadium <sup>b</sup>	140	±3

<sup>d</sup> The uncertainties shown for the elements except those noted by superscripts include errors associated with both measurement and material variability. They represent the 95 percent tolerance limits for individual subsamples, i.e., 95 percent of the subsamples from a single unit of this SRM would be expected to have a composition within the indicated range of values 95 percent of the time.

<sup>b</sup>These elements were recently certified as a part of the NBS update certification program. The values for the indicated constituent are the "best value" based on all measurement methods used and the associated uncertainty is expressed as the standard error considering variability within and between analytical methods.

#### Table 2. Noncertified Values for Constituent Elements

Note: The following values are not certified because they are not based on the results of either a reference method or two or more independent methods. These values are included for information only.

Major C	onstituents	Minor C	onstituents
Element	Content Wt. Percent	Element	Content Wt. Percent
Sulfur	(5.0)	Chlorine	(0.45)
Magnesium	(0.8)	Titanium	(0.40)
	Trace C	<u>Constituents</u>	
	Content		Content
Element	$\mu g/g$	Element	<u>µg/g</u>
Antimony	(45)	Lanthanum	(42)
Barium	(737)	Rubidium	(52)
Bromine	(500)	Manganese	(860)
Cerium	(55)	Samarium	( 4.4)
Cesium	(3)	Scandium	(7)
Cobalt	(18)	Silver	(6)
Europium	( 0.8)	Thorium	(7.4)
Hafnium	( 4.4)	Tungsten	( 4.8)
Indium	( 1.0)		
lodine	(20)		

#### Supplemental Information

The values listed below are based on measurements made in a single laboratory and are given for information only. While no reason exists to suspect systematic bias in these numbers, no attempt was made to evaluate such bias attributable to either the method or the laboratory. The method used for each set of measurements is also listed. The uncertainties indicated are two times the standard deviation of the means.

Constituent	Content Wt Percent
constituent	wt. refeelit
Nitrogen (NO3)	( 1.07 ± 0.06)
Nitrogen (NH4)	( 2.01 ± 0.08)
Sulfate	(15.42 ± 0.14)
SiO <sub>2</sub>	(26.8 ± 0.38)
Ficon Soluble	( 1.19 ± 0.47)

#### Methods Used:

Nitrate - Extraction with water and measurement by ASTM Method D992

Ammonia - NaOH addition followed by steam distillation and titration

Sulfate - Extraction with water and measurement by ASTM D516

SiO<sub>2</sub> - Solution and measurement by ASTM Method E350

Freon Soluble - Extraction with Freon 113, using the Method described in "Standard Methods in Examination of Water and Waste Water," 14th Edition, p. 518, American Public Health Association, Washington, D.C.

#### Analysts

Inorganic Analytical Chemistry Division

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5.	B. I. Diamondstone	13.	T. J. Murphy
6.	M. S. Epstein	14.	L. P. Powell
7.	M. Gallorini	15.	T. C. Rains
8.	E. L. Garner	16.	T. A. Rush

#### Table 3 Methods and Analysis

Method/ Element	А	В	С	D	E	F	G	н	i
Ag			•						
Al			•						
As			٠		٠				
Ba			٠						
Br			•						
Cd	•	•	•	•					
Ce			•						
CI							٠		
Со			•						
Cr		•	•						
Cs			•						
Cu	•	•			•				
Eu			•						
Fe	•	•	•		•				
Hf			•						
I			•			•			
ln			•						
K	•		•						
La			•						
Mg			•						
Mn	•		•						•
Na	•		•						•
Ni	•	•		•					
Pb	٠	•		•					
Rb			•						
S							•		
Sb			٠						
Sc			•						
Se	•		•						٠
Sm			•						
Th			•						
Ti			•						
U		•							
V	•		•						
W			•						
Zn	•	•	•	•					

Analytical Methods

- A. Atomic Absorption Spectrometry
- B. Isotope Dilution Mass Spectrometry
- C. Neutron Activation Analysis
- D. Polarography
- E. Spectrophotometry
- F. Photon Activation Analysis
- G. Ion Chromatography
- H. D.C. Plasma Atomic Emission Spectrometry
- 1. Flame Emission Spectrometry

# Guide for Requesting Development of Standard Reference Materials

The National Bureau of Standards develops Standard Reference Materials (SRM's) to provide a basis for comparison of measurements on materials and to aid in the control of production processes. The Office of Standard Reference Materials evaluates the requirements of science, industry, and government for carefully characterized reference materials, then directs the production and distribution of these materials.

NBS currently has over 1000 SRM's available, about 100 new ones in preparation, and requests for the development of many more. The demand for new SRM's greatly exceeds the Bureau's capacity to produce and certify these materials. Consequently, requests for new SRM's of limited use are deferred in favor of those that serve a substantial area of interest. In determining which requests receive top priority, NBS relies heavily upon information supplied by industry and interested organizations.

The Bureau welcomes all requests for SRM's. Both the Bureau and potential users would be helped if these requests included as much of the information below as possible.

**1.** Short title of the proposed Standard Reference Material.

2. Purpose for which this SRM is intended.

3. Reason why the SRM would be useful.

**4.** Special characteristics and/or requirements of the material. Include necessary additional information, if more than one SRM is needed for standardization in an area.

**5.** An estimate of the possible present and future (6-10 years) demand for such an SRM in your operations and elsewhere. (National and international estimates are very useful).

**6.** Facts about whether such an SRM (or a similar one) could be produced by, or obtained from a source other than NBS. If so, justify preparation by NBS.

7. Other pertinent information to justify the SRM, such as: (a) an estimate of the range of application, economic significance of the measurement affected, and scientific and/or technological significance, including estimates of the impact upon industrial productivity or growth, and (b) supporting letters from industry leaders, trade organizations, interested committees, and others.

In developing an NBS-SRM, the candidate material must r set one or more of the criteria listed below:

**1.** The SRM must permit users to attain more accurate measurements.

2. The production of the SRM must not be economically or technically feasible elsewhere,

3. The SRM must serve as an industry-wide standard for commerce, provided by a unique neutral source,

**4.** NBS production of the SRM would provide readily available, highly characterized material useful to science, industry, or government.

NBS has recognized the need to enlarge the scope of the SRM program to include all types of wellcharacterized materials that can be used to calibrate a measurement system, or to produce scientific data that can be widely used. Input from science, industry, and government assists NBS in continuing to provide Standard Reference Materials that will be valuable in many areas.

#### GUIDE TO ORDERING STANDARD REFERENCE MATERIALS

#### ORDERING

Orders should be addressed to: Office of Standard Reference Materials Room B311, Chemistry Building National Bureau of Standards Gaithersburg, MD 20899 Telephone: (301) 921-2045

Orders should give number of units, catalog number, and name of the material requested. For example, l each, No. 11h, Basic-Open-Hearth Steel, 0.2 percent C. The materials described in this Catalog are distributed only in the units listed or in multiples thereof.

Acceptance of an order does not imply acceptance of any provision set forth in the order contrary to the policy, practice, or regulations of the National Bureau of Standards or the U.S. Government.

Orders received for "out-of-stock" materials are cancelled if only out-of-stock items are ordered. On other orders, shipment is made of available materials and out-of-stock items are cancelled. Back-orders are not accepted for out-of-stock materials; if a renewal lot of material is available, it will be furnished automatically.

#### TERMS

Prices quoted are in U.S. dollars, and are published in the SRM Price List. When SRM Price Lists are issued they are sent to persons or organizations who have requested them. These prices are subject to revision without notice and orders will be billed for the prices in effect at the time of shipment. No discounts are given on purchases of SRM's, RM's, or GM's.

Remittances of the purchase price need not accompany purchase orders. Payment of invoices is expected within 30 days of receipt of an invoice. Payment on foreign orders may be made by any of the following:

- a. banker's draft against U.S.A. bank
- b. bank to bank transfer to U.S.A. bank
- c. cash against documents
- d. sight draft
- e. International Money Order
- f. UNESCO coupons

Letters of Credit cannot be accepted. If a Letter of Credit or any method of payment other than those listed above is to be used, you must secure the services of an agent in the United States to act in your behalf. Your agent would purchase the material and our invoice would indicate that he is the purchaser. The material would be shipped to your agent, who would transship in accordance with your instructions.

NBS cannot "prepay and add" shipping charges to the invoice. Restricted categories such as hydrocarbons, organic sulfur compounds, compressed gasses, rubber compounding materials, radioactive standards, and similar materials are shipped FOB Gaithersburg, MD.

#### LATE CHARGES

Unless otherwise notified, payment for SRM's is due within 30 days of shipment of the order to the customer. For non-Federal customers, the U.S. Treasury regulations require late charges, based on the current value of funds to Treasury, be assessed for each 30-day period or portion thereof that the payment is overdue.

#### PROFORMA INVOICE (PRICE QUOTATION)

Proforma invoice service will frequently require three to four weeks to process, and will be furnished only to those requiring such service.

#### DOMESTIC SHIPMENTS

Shipments of material (except for certain restricted categories) intended for the United States and Canada are normally shipped prepaid (providing that the prcel does not exceed the weight limitations as prescribed by postal laws and regulations).

#### FOREIGN SHIPMENTS

The regulations of various nations covering the importation of SRM's, GM's, and RM's differ widely; any attempt to list all possible variations would be impractical. Therefore, where the shipping practices outlined below do not apply, purchasers will be informed of the best method of shipment for their countries.

Most orders will be shipped by prepaid International Air Parcel Post. Exceptions are items in restricted categories and those shipments that exceed parcel post weight limitations. These exceptions will be shipped FOB Gaithersburg, MD, unless an agent (shipping or brokerage firm) located in the United States is required. Where an agent is required, the purchaser will be so notified and will be requested to designate an agent of his/her choice. In this case, the material will be packaged for overseas shipment and will be forwarded to the agent FOB Gaithersburg, MD.

#### DOCUMENTATION

Listed below are the only documents that we will furnish. All documents are printed in English.

- a. six commercial invoices
- b. two sight drafts
- c. two packing slips
- d. customs invoces for Canada, New Zealand, Australia, and South Africa
- e. Certificate of Origin
- f. parcel post receipts for parcel post shipments
- g. air waybill for air shipments

If documents other than those listed above are required, the services of an agent in the United States will be needed to purchase and ship the materials.

Note: Orders and inquiries submitted in English will be processed more rapidly than those requiring translations.

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NB3-114A (REV. 2-80)						
BIRLINGRAPHIC DATA	1. PUBLICATION OR REPORT NO.	2. Performing Organ. Report No. 3.	Publication Date			
SHEET (See instructions)	NBS/SP-260/105		March 1986			
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This publication is a summary of the environmental research, analysis, and control standards issued by NBS as Standard Reference Materials (SRM's). The material, composition, certification, use, and remarks concerning each of the SRM's described are presented in tabular form. Copies of the certificates of these SRM's are contained in the appendix for more detailed information.						
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