

NBS SPECIAL PUBLICATION 260-97

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

Standard Reference Materials:

Summary of the Coal, Ore, Mineral, Rock, and Refractory Standards Issued by the National Bureau of Standards

Radu Mavrodineanu and Thomas E. Gills

Summary of the Coal, Ore, Mineral, Rock, and Refractory Standards Issued by the National Bureau of Standards

R. Mavrodineanu¹

Center for Analytical Chemistry National Measurement Laboratory National Bureau of Standards Gaithersburg, MD 20899

and

T. E. Gills

Office of Standard Reference Materials National Measurement Laboratory National Bureau of Standards Gaithersburg, MD 20899

¹Present Address: 227 Almeria Road West Palm Beach, FL 33405



U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, Secretary NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Director

Library of Congress Catalog Card Number: 85-600577

National Bureau of Standards Special Publication 260-97 Natl. Bur. Stand. (U.S.), Spec. Publ. 260-97, 134 pages (Sept. 1985) CODEN: XNBSAV

U.S. GOVERNMENT PRINTING OFFICE WASHINGTON: 1985

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 technologicts 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 NBS Special Publication - 260 Series, is reserved for this purpose.

This 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 measurment 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 concered with the availability, delivery, price, and so forth, will receive prompt attention from:

Office of Standard Reference Materials National Bureau of Standards Gaithersburg, MD 20899

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

OTHER NBS PUBLICATIONS IN THIS SERIES

- Catalog of NBS Standard Reference Materials (1984-85 edition), Catherine H. Hudson, ed., NBS Spec. Publ. 260 (February 1984). \$5.50* SN003-003-02558-5.
- Michaelis, R. E., and Wyman, L. L. Standard Reference Materials: Preparation of White Cast Iron Spectrochemical Standards. NBS Misc. Publ. 260-1 (June 1964). COM74-11061**
- Michaelis, R. E., Wyman, L. L., and Flitsch, R., Standard Reference Materials: Preparation of NBS Copper-Base Spectrochemical Standards. NBS Misc. Publ. 260-2 (October 1964). COM74-11063**
- Michaelis, R. E., Yakowitz, H., and Moore, G. A., Standard Reference Materials: Metallographic Characterization of an NBS Spectrometric Low-Alloy Steel Standard. NBS Misc. Publ. 260-3 (October 1964). COM74-11060**
- Hague, J. L. Mears, T. W., and Michaelis, R. E., Standard Reference Materials: Sources of Information, NBS Misc. Publ. 260-4 (February 1965). COM74-11059
- Alvarez, R., and Flitsch R., Standard Reference Materials: Accuracy of Solution X-Ray Spectrometric Analysis of Copper-Base Alloys. NBS Misc. Publ. 260-5 (March 1965). PB168068**
- Shultz, J. I., Standard Reference Materials: Methods for the Chemical Analysis of White Cast Iron Standards, NBS Misc. Publ. 260-6 (July 1965). COM74-11068**
- Bell, R. K., Standard Reference Materials: Methods for the Chemical Analysis of NBS Copper-Base Spectrochemical Standards. NBS Misc. Publ. 260-7 (October 1965). COM74-11067**
- Richmond, M.S., Standard Reference Materials: Analysis of Uranium Concentrates at the National Bureau of Standards. NBS Misc. Publ. 260-8 (December 1965). COM74-11066**
- Anspach, S. C., Cavallo, L. M. Garfinkel, S. B. Hutchinson, J. M. R., and Smith, C. N., Standard Reference Materials: Half Lives of Materials Used in the Preparation of Standard Reference Materials of Nineteen Radioactive Nuclides Issued by the National Bureau of Standards NBS Misc. Publ. 260-9 (November 1965). COM74-11065**
- Yakowitz, H., Vieth, D. L., Heinrich, K. F. J., and Michaelis, R. E., Standard Reference Materials: Homogeneity Characterization of NBS Spectrometric Standards II: Cartridge Brass and Low-Alloy Steel, NBS MIsc. Publ. 260-10 (December (1965). COM74-11064**
- Napolitano, A., and Hawkins, E. G., Standard Reference Materials: Viscosity of Standard Lead-Silica Glass, NBS Misc. Publ. 260-11 (November 1966). NBS Misc. Publ. 260-11**
- Yakowitz, H., Vieth, D. L., and Michaelis, R. E., Standard Reference Materials: Homogeneity Characterization of NBS Spectrometric Standards III. White Cast Iron and Stainless Steel Powder Compact, NBS Misc. Publ. 260-12 (September 1966). NBS Misc. Publ. 260-12**

- Spijkerman, J. L., Snediker, D. K., Ruegg, F. C., and DeVoe, J. R., Standard Reference Materials: Mossbauer Spectroscopy Standard for the Chemical Shift of Iron Compounds, NBS Misc. Publ. 260-13 (July 1967). NBS Misc. Publ. 260-13**
- Menis, O., and Sterling, J. T., Standard Reference Materials: Determination of Oxygen in Ferrous Materials - SRM 1090, 1091, and 1092, NBS Misc. Publ. 260-14 (September 1966). NBS Misc. Publ. 260-14**
- Passaglia, E., and Shouse, P. J., Standard Reference Materials: Recommended Method of Use of Standard Light-Sensitive Paper for Calibrating Carbon Arcs Used in Testing Textiles for Colorfastness to Light, NBS Misc. Publ. 260-15 (June 1967). (Replaced by NBS Spec. Publ. 260-41.)
- Yakowitz, H., Michaelis, R. E., and Vieth, D. L., Standard Reference Materials: Homogeneity Characterization of NBS Spectrometric Standards IV: Preparation and Microprobe Characterization of W-20% MO Alloy Fabricated by Powder Metallurgical Methods, NBS Spec. Publ. 260-16 (January 1969). COM74-11062**
- Catanzaro, E. J., Champion, C. E., Garner, E. L., Marinenko, G., Sappenfield, K. M., and Shields, W. R., Standard Reference Materials: Boric Acid: Isotopic and Assay Standard Reference Materials, NBS Spec. Publ. 260-17 (February 1970). Out of Print.
- Geller, S. B., Mantek, P.A., and Cleveland, N. G., Standard Reference Materials: Calibration of NBS Secondary Standard Magnetic Tape (Computer Amplitude Reference) Using the Reference Tape Amplitude Measurement "Process A," NBS Spec. Publ. 260-18 (November 1969). (See NBS Spec. Publ. 260-29.)
- Paule, R. C., and Mandel, J., Standard Reference Materials: Analysis of Interlaboratory Measurements on the Vapor Pressure of Gold (Certification of Standard Reference Material 745). NBS Spec. Publ. 260-19 (January 1970). PB190071**
- Paule, R. C., and Mandel, J., Standard Reference Materials: Analysis of Interlaboratory Measurements on the Vapor Pressures of Cadmium and Silver. NBS Spec. Publ. 260-21 (January 1971). COM74-11359**
- Yakowitz, H., Fiori, C. E., and Michaelis, R. E., Standard Reference Materials: Homogeneity Characterization of Fe-3 Si Alloy, NBS Spec. Publ. 260-22 (February 1971). COM74-11357**
- Napolitano, A., and Hawkins, E. G., Standard Reference Materials: Viscosity of a Standard Borosilicate Glass, NBS Spec. Publ. 260-23 (December 1970). COM71-00157**
- Sappenfield, K. M., Marineko, G., and Hague, J. L., Standard Reference Materials: Comparison of Redox Standards, NBS Spec. Publ. 260-24 (January 1972). COM72-50058**

- Hicho, G. E., Yakowitz, H., Rasberry, S. D., and Michaelis, R. E., Standard Reference Materials: A Standard Reference Material Containing Nominally Four Percent Austenite, NBS Spec. Publ. 260-25 (February 1971). COM74-11356**
- Martin, J. F., Standard Reference Materials: National Bureau of Standards-US Steel Corporation Joint Program for Determining Oxygen and Nitrogen in Steel, NBS Spec. Publ. 260-26 (February 1971). 85 cents* PB 81176620
- Garner, E. L., Machlan, L. A., and Shields, W. R., Standard Reference Materials: Uranium Isotopic Standard Reference Materials, NBS Spec. Publ. 260-27 (April 1971). COM74-11358**
- Heinrich, K. F. J., Myklebust, R. L., Rasberry, S. D., and Michaelis, R. E., Standard Reference Materials: Preparation and Evaluation of SRM's 481 and 482 Gold-Silver and Gold-Copper Alloys for Microanalysis, NBS Spec. Publ. 260-28 (August 1971). COM71-50365**
- Geller, S. B., Standard Reference Materials: Calibration of NBS Secondary Standard Magnetic Tape (Computer Amplitude Reference) Using the Reference Tape Amplitude Measurement "Process A-Model 2," NBS Spec. Publ. 260-29 (June 1971). COM71-50282
- Gorozhanina, R. S., Freedman, A. Y., and Shaievitch, A. B. (translated by M. C. Selby), Standard Reference Materials: Standard Samples Issued in the USSR (A Translation from the Russian). NBS Spec. Publ. 260-30 (June 1971). COM71-50283**
- Hust, J. G., and Sparks, L. L., Standard Reference Materials: Thermal Conductivity of Electrolytic Iron SRM 734 from 4 to 300 K, NBS Spec. Publ. 260-31 (November 1971). COM71-50563**
- Mavrodineanu, R., and Lazar, J. W., Standard Reference Materials: Standard Quartz Cuvettes, for High Accuracy Spectrophotometry, NBS Spec. Publ. 260-32 (December 1973). 55 cents* SN003-003-01213-1
- Wagner, H. L., Standard Reference Materials: Comparison of Original and Supplemental SRM 705, Narrow Molecular Weight Distribution Polystyrene, NBS Spec. Publ. 260-33 (May 1972). COM72-50526**
- Sparks, L. L., and Hust, J. G., Standard Reference Materials: Thermoelectric Voltage, NBS Spec. Publ. 260-34, (April 1972). COM72-50371**
- Sparks, L. L., and Hust, J. G., Standard Reference Materials: Thermal Conductivity of Austenitic Stainless Steel, SRM 735 from 5 to 280 K, NBS Spec. Publ. 260-35 (April 1972.) 35 cents* COM72-50368**
- Cali, J. P., Mandel, J., Moore, L. J., and Young, D. S., Standard Reference Materials: A Referee Method for the Determination of Calcium in Serum, NBS SRM 915, NBS Spec. Publ. 260-36 (May 1972). COM72-50527**
- Shultz, J. I. Bell., R. K. Rains, T. C., and Menis, O., Standard Reference Materials: Methods of Analysis of NBS Clay Standards, NBS Spec. Publ. 260-37 (June 1972). COM72-50692**

- Richmond, J. C., and Hsia, J. J., Standard Reference Materials: Preparation and Calibration of Standards of Spectral Specular Reflectance, NBS Spec. Publ. 260-38 (May 1972). COM72-50528**
- Clark, A. F., Denson, V.A., Hust, J. G., and Powell, R. L., Standard Reference Materials: The Eddy Current Decay Method for Resistivity Characterization of High-Purity Metals, NBS Spec. Publ. 260-39 (May 1972). COM72-50529**
- McAdie, H. G., Garn, P.D., and Menis, O., Standard Reference Materials: Selection of Thermal Analysis Temperature Standards Through a Cooperative Study (SRM 758, 759, 760), NBS Spec. Publ. 260-40 (August 1972.) COM72-50776**
- Wood, L. A., and Shouse, P. J., Standard Reference Materials: Use of Standard Light-Sensitive Paper for Calibrating Carbon Arcs Used in Testing Textiles for Colorfastness to Light, NBS Spec. Publ. 260-41 (August 1972) COM72-50775**
- Wagner, H. L. and Verdier, P. H., eds., Standard Reference Materials: The Characterization of Linear Polyethylene, SRM 1475, NBS Spec. Publ. 260-42 (September 1972). COM72-50944**
- Yakowitz, H., Ruff, A. W., and Michaelis, R. E., Standard Reference Materials: Preparation, and Homogeneity Characterization of an Austenitic Iron-Chromium-Nickel Alloy, NBS Spec. Publ. 260-43 (November 1972). COM73-50760**
- Schooley, J. F., Soulen, R. J., Jr., and Evans, G. A., Jr., Standard Reference Materials: Preparation and Use of Superconductive Fixed Point Devices, SRM 767, NBS Spec. Publ. 260-44 (December 1972). COM73-50037**
- Greifer, B., Maienthal, E. J., Rains, T. C., and Rasberry, S. D., Standard Reference Materials: Powdered Lead-Based Paint, SRM 1579, NBS Spec. Publ. 260-45 (March 1973). COM73-50226**
- Hust, J. G., and Giarratano, P. J., Standard Reference Materials: Thermal Conductivity and Electrical Resistivity Standard Reference Materials: Austenitic Stainless Steel, SRM's 735 and 798, from 4 to 1200 K, NBS Spec. Publ. 260-46 (March 1975). SN003-003-01278-5*
- Hust, J. G., Standard Reference Materials: Electrical Resistivity of Electrolytic Iron, SRM 797, and Austenitic Stainless Steel, SRM 798, from 5 to 280 K, NBS Spec. Publ. 260-47 (February 1974). COM74-50176**
- Mangum, B. W., and Wise, J. A., Standard Reference Materials: Description and Use of Precision Thermometers for the Clinical Laboratory, SRM 933 and SRM 934, NBS Spec. Publ. 260-48 (May 1974). 60 cents* SN003-003-01278-5
- Carpenter, B. S., and Reimer, G. M., Standard Reference Materials: Calibrated Glass Standards for Fission Track Use, NBS Spec. Publ. 260-49 (November 1974). COM74-51185

- Hust, J. G., and Giarratano, P. J., Standard Reference Materials: Thermal Conductivity and Electrical Resistivity Standard Reference Materials: Electrolytic Iron, SRM's 734 and 797 from 4 to 1000 K, NBS Spec. Publ. 260-50 (June 1975). \$1.00* \$N003-003-01425-7
- Mavrodineanu, R., and Baldwin, J. R., Standard Reference Materials: Glass Filters As a Standard Reference Material for Spectrophotometry; Selection; Preparation; Certification; Use-SRM 930, NBS Spec. Publ. 260-51 (November 1975). \$1.90\cdot SN003-003-01481-8
- Hust, J. G., and Giarratano, P. J., Standard Reference Materials: Thermal Conductivity and Electrical Resistivity Standard Reference Materials 730 and 799, from 4 to 3000 K, NBS Spec. Publ. 260-52 (September 1975). \$1.05* SN003-003-01464-8
- Durst, R. A., Standard Reference Materials: Standardization of pH Measurements, NBS Spec. Publ. 260-53 (December 1975, Revised). \$1.05 SN003-003-01551-2
- Burke, R. W., and Mavrodineanu, R., Standard Reference Materials: Certification and Use of Acidic Potassium Dichromate Solutions as an Ultraviolet Absorbance Standard, NBS Spec. Publ. 260-54 (August 1977). \$3.00* SN003-003-01828-7
- Ditmars, D. A., Cezairliyan, A., Ishihara, S., and Douglas, T. B., Standard Reference Materials: Enthalpy and Heat Capacity; Molybdenum SRM 781, from 273 to 2800 K, NBS Spec. Publ. 260-55 (September 1977). \$2.20* SN003-003-01836-8
- Powell, R. L., Sparks, L. L., and Hust, J. G., Standard Reference Materials: Standard Thermocouple Materials, Pt.67: SRM 1967, NBS Spec. Publ. 260-56 (February 1978). \$2.20* SN003-003-018864
- Cali, J. P. and Plebanski, T., Guide to United States Reference Materials, NBS Spec. Publ. 260-57 (February 1978). \$2.20* PB 277173
- Barnes, J. D., and Martin, O. M., Standard Reference Materials: Polyester Film for Oxygen Gas Transmission Measurements SRM 1470, NBS Spec. Publ. 260-58 (June 1979) \$2.00* SN003-003-02077
- Chang, T., and Kahn, A. H. Standard Reference Materials: Electron Paramagnetic Resonance Intensity Standard; SRM 2601, NBS Spec. Publ. 260-59 (August 1978) \$2.30* SN003-003-01975-5
- Velapoldi, R. A., Paule, R. C., Schaffer, R., Mandel, J., and Moody, J. R., Standard Reference Materials: A Reference Method for the Determination of Sodium in Serum, NBS Spec. Publ. 260-60 (August 1978). \$3.00* SN003-003
- Verdier, P. H., and Wagner. H. L., Standard Reference Materials: The Characterization of Linear Polyethylene (SRM 1482, 1483, 1484), NBS Spec. Publ. 260-61 (December 1978). \$1.70* SN003-003-02006-1

- Soulen, R. J., and Dove, R. B., Standard Reference Materials: Temperature Reference Standard for Use Below 0.5 K (SRM 768). NBS Spec. Publ. 260-62 (April 1979). \$2.30* SN003-003-02047-8
- Velapoldi, R. A., Paule, R. C., Schaffer, R. Mandel, J., Machlan, L. A., and Gramlich, J. W., Standard Reference Materials: A Reference Method for the Determination of Potassium in Serum. NBS Spec. Publ. 260-63 (May 1979). \$3.75* SN003-003-02068
- Velapoldi, R. A., and Mielenz, K. D., Standard Reference Materials: A Fluorescence Standard Reference. Material Quinine Sulfate Dihydrate (SRM 936), NBS Spec. Publ. 260-64 (January 1980). \$4.25* SN003-003-02148-2
- Marinenko, R. B., Heinrich, K. F. J., and Ruegg, F. C., Standard Reference Materials: Micro-Homogeneity Studies of NBS Standard Reference Materials, NBS Research Materials, and Other Related Samples. NBS Spec. Publ. 260-65 (September 1979). \$3.50° SN003-003-02114-1
- Venable, W. H., Jr., and Eckerle, K. L., Standard Reference Materials: Didymium Glass Filters for Calibrating the Wavelength Scale of Spectrophotometers (SRM 2009, 2010, 2013). NBS Spec. Publ. 260-66 (October 1979). \$3.50* SN003-003-02127-0
- Velapoldi, R. A., Paule, R. C., Schaffer, R., Mandel, J., Murphy, T. J., and Gramlich, J. W., Standard Reference Materials: A Reference Method for the Determination of Chloride in Serum, NBS Spec. Publ. 260-67 (November 1979). \$3.75* SN003-003-02136-9
- Mavrodineanu, R. and Baldwin, J. R., Standard Reference Materials: Metal-On-Quartz Filters as a Standard Reference Material for Spectrophotometry-SRM 2031, NBS Spec. Publ. 260-68 (April 1980). \$4.25* SN003-003-02167-9
- Velapoldi, R. A., Paule, R. C., Schaffer, R., Mandel, J., Machlan, L. A., Garner, E. L., and Rains, T. C., Standard Reference Materials: A Reference Method for the Determination of Lithium in Serum, NBS Spec. Publ. 260-69 (July) 1980). \$4.25* SN003-003-02214-4
- Marinenko, R. B., Biancaniello, F., Boyer, P. A., Ruff, A. W., DeRobertis, L., Standard Reference Materials: Preparation and Characterization of an Iron-Chromium-Nickel Alloy for Microanalysis, NBS Spec. Publ. 260-70 (May 1981). \$2.50* \$N003-003-02328-1
- Seward, R. W., and Mavrodineanu, R., Standard Reference Materials: Summary of the Clinical Laboratory Standards Issued by the National Bureau of Standards, NBS Spec. Publ. 260-71 (November 1981). \$6.50* SN003-003-02381-7
- Reeder, D.J., Coxon, B., Enagonio, D., Christensen, R. G., Schaffer, R., Howell, B. F., Paule, R. C., Mandel, J., Standard Reference Materials: SRM 900, Antiepilepsy Drug Level Assay Standard, NBS Spec. Publ. 260-72 (June 1981). \$4.25* SN003-003-02329-9

- Interrante, C. G., and Hicho, G. E., Standard Reference Materials: A Standard Reference Material Containing Nominally Fifteen Percent Austenite (SRM 486), NBS Spec. Publ. 260-73 (January 1982). \$2.75* SN003-003-02386-8
- Marinenko, R. B., Standard Reference Materials: Preparation and Characterization of K-411 and K-414 Mineral Glasses for Microanalysis: SRM 470. NBS Spec. Publ. 260-74 (April 1982). \$3.50 SN003-003-023-95-7
- Weidner, V. R., and Hsia, J. J., Standard Reference Materials: Preparation and Calibration of First Surface Aluminum Mirror Specular Reflectance Standards (SRM 2003a), NBS Spec. Publ. 260-75 (May 1982). \$3.75 SN003-003-023-99-0
- Hicho, G. E. and Eaton, E. E., Standard Reference Materials: A Standard Reference Material Containing Nominally Five Percent Austenite (SRM 485a), NBS Spec. Publ. 260-76 (August 1982). \$3.50 SN003-003-024-33-3
- Furukawa, G. T., Riddle, J. L., Bigge, W. G., and Pfieffer, E. R., Standard Reference Materials: Application of Some Metal SRM's as Thermometric Fixed Points, NBS Spec. Publ. 260-77 (August 1982). \$6.00 SN003-003-024-34-1
- Hicho, G. E. and Eaton, E. E., Standard Reference Materials: Standard Reference Material Containing Nominally Thirty Percent Austenite (SRM 487), NBS Spec. Publ. 260-78 (September 1982).
 \$3.75* SN003-003-024-35-0
- Richmond, J. C., Hsia, J. J. Weidner, V. R., and Wilmering, D. B., Standard Reference Materials: Second Surface Mirror Standards of Specular Spectral Reflectance (SRM's 2023, 2024, 2025), NBS Spec. Publ. 260-79 (October 1982). \$4.50* SN003-003-024-47-3
- Schaffer, R., Mandel, J., Sun, T., Cohen, A., and Hertz, H. S., Standard Reference Materials: Evaluation by an ID/MS Method of the AACC Reference Method for Serum Glucose, NBS Spec. Publ. 260-80 (October 1982). \$4.75* SN003-003-024-43-1
- Burke, R. W., and Mavrodineanu, R. (NBS retired),
 Standard Reference Materials: Accuracy in
 Analytical Spectrophotometry, NBS Spec. Publ.
 260-81 (April 1983). \$6.00* SN003-003-024-8
- Weidner, V. R., Standard Reference Materials: White Opal Glass Diffuse Spectral Reflectance Standards for the Visible Spectrum (SRM's 2015 and 2016). NBS Spec. Publ. 260-82 (April 1983). \$3.75* SN003-003-024-89-9**
- Bowers, G. N., Jr., Alvarez, R., Cali, J. P. (NBS retired), Eberhardt, K. R., Reeder, D. J., Schaffer, R., Uriano, G. A., Standard Reference Materials: The Measurement of the Catalytic (Activity) Concentration of Seven Enzymes in NBS Human Serum SRM 909, NBS Spec. Publ. 260-83 (June 1983). \$4.50* SN003-003-024-99-6
- Gills, T. E., Seward, R. W., Collins, R. J., and Webster, W. C., Standard Reference Materials: Sampling, Materials Handling, Processing, and Packaging of NBS Sulfur in Coal Standard Reference Materials, 2682, 2683, 2684, and 2685, NBS Spec. Publ. 260-84 (August 1983). \$4.50* SN003-003-025-20-8

- Swyt, D. A., Standard Reference Materials: A Look at Techniques for the Dimensional Calibration of Standard Microscopic Particles, NBS Spec. Publ. 260-85 (September 1983). \$5.50* SN003-003-025-21-6
- Hicho, G. E. and Eaton, E. E., Standard Reference Materials: A Standard Reference Material Containing Two and One-Half Percent Austenite, SRM 488, NBS Spec. Publ. 260-86 (December 1983). \$1.75° SN003-003-025-41-1
- Mangum, B. W., Standard Reference Materials: SRM 1969: Rubidium Triple-Point - A Temperature Reference Standard Near 39.30 °C, NBS Spec. Publ. 260-87 (December 1983). \$2.25* SN003-003-025-44-5
- Gladney, E. S., Burns, C. E., Perrin, D. R., Roelandts, I., and Gills, T. E., Standard Reference Materials: 1982 Compilation of Elemental Concentration Data for NBS Biological, Geological, and Environmental Standard Reference Materials. Spec. Publ. 260-88 (March. 1984). \$7.00* SN003-003-02565-8
- Hust, J. G., Standard Reference Materials: A Fine-Grained, Isotropic Graphite for Use as NBS Thermophysical Property RM's from 5 to 2500 K, NBS Spec. Publ. 260-89 (September 1984). \$4.50* SN003-003-02608-5
- Hust, J. G., and Lankford, A. B., Standard Reference Materials: Update of Thermal Conductivity and Electrical Resistivity of Electrolytic Iron, Tungsten, and Stainless Steel, NBS Spec. Publ. 260-90 (September 1984). \$3.00* SN003-003-02609-3
- Goodrich, L. F., Vecchia, D. F., Pittman, E. S., Ekin, J. W., and Clark, A. F., Standard Reference Materials: Critical Current Measurements on an NbTi Superconducting Wire Standard Reference Material, NBS Spec. Publ. 260-91 (September 1984). \$2.75° SN003-003-07614-0
- Carpenter, B. S., Standard Reference Materials: Calibrated Glass Standards for Fission Track Use (Supplement to NBS Spec. 260-49). NBS Spec. Publ.. 260-92 (September 1984). \$1.50* SN003-003-003-02610-7
- Ehrstein, J., Preparation and Certification of Standard Reference Materials for Calibration of Spreading Resistance Probes, NBS Spec. Publ. 260-93 (January 1985).
- Gills, T. E., Koch, W. F. Stolz, J. W., Kelly, W. R., Paulsen, P. J., Colbert, J. C. Kirklin, D. R., Pei, P.T.S., Weeks, S., Lindstrom, R. M. Fleming, R. F., Greenberg, R. R., and Paule, R. C., Methods and Procedures Used at the National Bureau of Standards to Certify Sulfur in Coal SRM's for Sulfur Content, Calorific Value, Ash Content, NBS Spec. Publ. 260-94 (December 1984).
- Mulholland, G. W., Hartman, A. W., Hembree, G. G., Marx, E., and Lettieri, T. R., Standard Reference Materials: Development of a 1 m Diameter Particle Size Standard, SRM 1690, NBS Spec. Publ. 260-95 (May 1985).

- Carpenter, B. S., Gramlich, J. W., Greenberg, R. R., and Machlan, L. A., Standard Reference Materials: Uranium-235 Isotopic Abundance Standard Reference Materials for Gamma Spectrometry Measurements, NBS Spec. Publ. 260-96 (In Press).
- Mavrodineanu, R. and Gills, T. E., Standard Reference Materials: Summary of the Coal, Ore, Mineral, Rock, and Refractory Standards Issued by the National Bureau of Standards, NBS Spec. Publ. 260-97 (In Press).
- Hust. J. G., Standard Reference Materials: Glass Fiberboard SRM for Thermal Resistance, NBS Spec. Publ. 260-98 (In Press).
- Callanan, J. E., Sullivan, S. A., and Vecchia, D. F., Standard Reference Materials: Feasibility Study for the Development of Standards Using Differential Scanning Calorimetry, NBS Spec. Publ. 260-99 (In Press).
- Taylor, J. K., Standard Reference Materials: Handbook for SRM Users, NBS Spec. Publ. 260-100 (In Press).
- * 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

											Page
Preface .								•		•	iii
Abstract		,	• • • •	• • • •	• • • •	• • • •		•	• •	•	x
Introduct	ion					• • • •	• • •	•		• (1
Table 1.	Category In	ndex of Star ls	ndards ava	ilable fr	om the N	National	Burea	u •		•	2
Coal, Ore	e, Mineral, I	Rock, and Ro	efractory	Standards			• • •	•		•	4
Table 2.	Summary of	Coal, Ore,	Mineral,	Rock, and	Refract	tory Sta	ındards	•		•	6
	A. Coals				• • • •		· • •			•	6
	B. Rocks,	Minerals,	and Refrac	tories .			··· • •	•	• •	•	8
	C. Ores				• • • •	• • •		•		•	14
Table 3.	Composition Refractorio		Prepared f	rom Ores,					• •	•	22
Table 4.		ations in tock, and Re						•		•	26
Acknowled	igment			• • • •,		• • •		•		•	26
Appendix	I. Alphabe	tical Index	by Standa	rd Refere	nce Mat	erial Na	ame	•		•	27
Appendix	II. Certif Standa	icates for rds (listed	Coal, Ore, in numeri	Mineral, cal order	Rock,	and Ref	ractory	•		•	40

Abstract

This publication is a summary of the coal, ore, mineral, rock, and refractory 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; coals; ores; refractories; rocks; 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. 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 these types of 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.

¹For 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

Forensic Standards

Hydrocarbon Blends

Metallo-Organic Compounds

Fertilizers

0res

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

pH standards pD standards

Ion selective electrodes

Mechanical and Metrology Standards

Magnification Coating thickness Glass Elasticity Density Polymer

Heat Standards

Rheology

Superconductive thermometric fixed point devices Freezing Points

Defining fixed points
Determined reference points

Melting points Calorimetric

> Combustion Solution Heat source Enthalpy and heat capacity

Vapor pressure Thermal expansion Thermocouple materials Thermal resistance

Magnetic Standards

Magnetic susceptibility Magnetic moment Paramagnetic resonance Optical Standards

Spectrophotometric Thermal emittance Refractive index

Radioactivity Standards

Alpha-particle standards
Beta-particle and gamma-ray gas
standards
Alpha-particle, beta-particle,
gamma-ray, and electron-capture
solution standards
Contemporary standard for carbon-14
dating laboratories
Environmental standards
Low energy photon sources
Gamma-ray "point-source" standards
Radium gamma-ray solution standards
Radium solution standards for
random analysis
Radioactivity standard reference
materials currently not in stock

Metallurgical

Mössbauer

X-ray Diffraction

Gas Transmission

Permittivity

Reference Fuels

Resistivity

ENGINEERING TYPE STANDARDS

Standard Rubber and Rubber-Compounding Materials

Reference Magnetic Tapes

Centerline Drawings, OCR-B

Sizing Standards

Glass spheres for particle size Turbidimetric and fineness (cement)

RESEARCH MATERIALS

X-ray and Photographic Standards

Surface Flammability Standards

Smoke Density Chamber Standards

Water Vapor Permeance

Tape Adhesion Testing Standards

Color Standards

SPECIAL REFERENCE MATERIALS

The accurate determination of major and minor constituents in coals, ores, minerals, rocks, and refractories is important to our nation's scientific and industrial community as well as to commerce and trade. In many industries, traceability of quality control to the national measurement system is achieved through the use of Standard Reference Materials (SRM's). This publication is an attempt to describe, in general terms, the composition, certification, and use of such SRM's.

Essential information concerning the material composition, the certification parameters, and use is given in Table 2. Under "Remarks," additional data are provided. All the data and information contained in this table were extracted from the Certificates or Certificates of Analysis issued for the included SRM's. An examination of the table 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 any references cited in each certificate. The composition of the same SRM's, excepting the coals, by chemical elements are listed in Table 3. In the tables, similar types of SRM's are grouped together to facilitate comparisons of their properties. The Certificates in Appendix II, however, are arranged in numerical order. The SRM's listed in the tables include all of the coal, ore, mineral, rock, and refractory standards that were in stock as of January 1, 1984. These SRM's are the result of the concerted efforts of a number of scientists from the NBS National Measurement Laboratory, Center for Analytical Chemistry, and from industry. Each Certificate lists the individuals and laboratories who contributed to the preparation and certification of the SRM.

In addition to the SRM's and their Certificates, NBS issues a series of Special Publications (SP), call 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.

Table 4 lists several NBS SP 260 publications directly related to the SRM's described in this work. They should be of value to the user of these particular materials. Also, NBS SP 260-54 contains in its appendices three reprints on: "Standard Quartz Cuvettes for High-Accuracy Spectrophotometry" (Appendix III); "Testing of Glass Volumetric Apparatus, 1959" (Appendix V); and "The Calibration of Small Volumetric Laboratory Glassware, 1974" (Appendix VI). The last two publications have been out of print for several years.

Other NBS publications, not in the "260 Series," and a number of NBS staff authored papers have been published that deal with specific SRM's or measurement techniques used in analytical chemistry. Some of these are: SP 148, The Role of Standard Reference Materials in Measurement Systems; SP 378, Accuracy in Spectrophotometry and Luminescence Measurements (255 pp., 1973); and SP 466, Standardization in Spectrophotometry and Luminescence Measurements (150 pp., 1977). NBS Monograph 100, Trace Characterization, Chemical and Physical (580 pp., 1967); SP 422, Accuracy in Trace Analysis, Sampling, Sample Handling, Analysis, Vol. 1 and 2 (1304 pp., 1976); and SP 464, Proceedings of the Eighth Materials Research Symposium, Methods and Standards for Environmental Measurements (691 pp., 1977), provide further information on methodology for the analysis of a variety of materials and the determination of numerous constituents. They all should be of particular interest to the analytical chemist.

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. Table 3 was taken from an article by S. D. Rasberry published in Amer.
Lab., 15 (5), 96 (1983).

² For complete bibliographic reference and ordering information, see "Other NBS Publications in This Series," pp. iv.

Table 2. Summary of Coal, Ore, Mineral, Rock, and Refractory Standards

A. Coals

SRM Material Composition 1632ъ Obtained from Humphrey No. 7 Material should be vacuum Trace Elements in Coal mine and coal preparation plant dried at ambient temperature of the Consolidation Coal Co., (Bituminous) for 24 hours prior to use. Christopher Coal Co. Div., Values based on a minimum Osage, W.V. The coal was sample size of 250 mg. reduced in size to -60 mesh and C (total) 78.11; H 5.07; sieved prior to blending. The N 1.56; S 1.89; Volatile coal was then blended in a matter 35.4; A1 0.855; stainless steel coal blender. 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). 1633a Supplied by a coal fired power Material should be dried to a Trace Elements in plant and is a product of constant weight before use. Coal Fly Ash Pennsylvania and West Virginia Values based on a 250-mg or coals. Material was sieved more sample size. Ca 1.11; through a #170 sieve and blended Fe 9.40; K 1.88; Mg 0.455; in a Vee blender. Na 0.17; Si 22.8, values % by wt. 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; T1 5.7; U 10.2; Zn 220, values μg/g. Not certified: 15 additional constituents (see certificate) 1635 Provided by the Eagle Mine of Material should be dried with-Trace Elements in Coal the Imperial Coal Company, out heat to constant weight (Sub-bituminous) Erie, Colorado. Ground and before use. Values based on sieved through a 230 m sieve 250-mg or more sample size. by the Colorado School of Mines As 0.42; Cd 0.03; Cr 2.5; Research Institute. Cu 3.6; Pb 1.9; Mg 21.4; Ni 1.74; Se 0.9; Th 0.62; U 0.24; V 5.2; Zn 4.7, values $\mu g/g$. Fe 0.239; S 0.33, values % by wt. Not certified 10 additional constitutents (see certificate).

2682 Sulfur in Coal (Sub-bituminous)

Obtained from Bell Ayr Mine, Gillette, Wyoming The coal was reduced in size to -60 mesh and screened prior to blending. The -60 mesh coal was then blended in a stainless steel cone blender.

Sample vacuum dried at ambient temperature for 24 hours or oven drying for 24 hours at 105 °C. Values based on 250-mg or more sample size.

Sulfur (wt. %) 0.47; Furnace Ash (wt. %) 6.37; Calorific Content 27.45 MJ.Kg 1. Not certified: 32 inorganic constituents (see certificate).

^{*}National Bureau of Standards, Center for Analytical Chemistry. $^{\circ}$ American Society for Testing and Materials.

Analyses performed in the NBS CAC.* Estimated uncertainty 0.0008 TO 2.1 depending on constituent.

For the calibration of apparatus 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 radiation, including ultraviolet lamps or sunlight.

Analyses performed in the NBS CAC. Estimated uncertainty 0.010 to 30 depending on constituent.

For the calibration of apparatus and methods used in analyses of coal fly ash and other materials with similar matrices for trace elements.

Should be kept in a tightly sealed bottle.

Analyses performed in the NBS CAC. Estimated uncertainty 0.15 to 1.5 depending on constituent.

Same as SRM 1632a.

Same as SRM 1632a.

Analyses performed in the Inorganic Analytical Research Division and Chemical Thermodynaics Division of NBS. Calorific values were determined by procedures recommended in standard ASTMO methods.

For the determination of sulfur in coal.

The Sulfur in Coal SRM's are sold individually rather than in sets; however, only one Certificate of Analysis is provided. Therefore, the user must be careful to use the data specific to the SRM being used. Material should be stored in a tightly sealed bottle away from sunlight and intense sources of radiation.

SRM

Material

Composition

	· · · · · · · · · · · · · · · · · · ·	
2683 Sulfur in Coal (Bituminous)	Obtained from Humphrey Mine, Osage, West Virginia. Processing same as SRM 2682.	Same as SRM 2682 except: Sulfur (wt. %) 1.85; Furnace Ash (wt. %) 6.85; Calorific Content 32.70 MJ.Kg .
2684 Sulfur in Coal (Bituminous)	Obtained from Delta Mine, Marion, Illinois. Processing same as SRM 2682.	Same as SRM 2682 except: Sulfur (wt. %) 3.00; Furnace Ash (wt. %) 11.09; Calorific Content 29.68 MJ.Kg ⁻¹ .
2685 Gulfur in Coal (Bituminous)	Obtained from McElroy Mine, Captina, West Virgnia. Processing same as SRM 2682.	Same as SRM 2682 except: Sulfur (wt. %) 4.62; Furnace Ash (wt. %) 16.53; Calorific Content 28.15 MJ.Kg
B. Rocks, Minerals, and Refracto	ries	
lc Argillaceous Limestone	Provided by Lone Star Ind., Inc., Cement and Construction Materials Group, Houston, Texas.	Limestone, ground, sieved, blended. Analyses performed on 0.5-g or more sample. Dried 2 hours at 110 °C. SiO, 6.84; Fe ₂ O ₃ 0.55; Al ₂ O ₃ 1.30; TiO ₂ 0.07; P ₂ O ₅ 0.04; Mno 0.025; CaO 50.3; SrO 0.030; MgO 0.42; Na ₂ O 0.02; k ₂ O 0.28; loss on
	· · · · · · · · · · · · · · · · · · ·	ignition 39.9; all values % by wt.
70a Feldspar	Feldspar, sample dried 2 hours at 105 °C.	SiO ₂ 67.1; Al ₂ O ₃ 17.9; Fe ₂ O ₃ 0.07; TiO ₂ 0.01; CaO 0.11; BaO 0.02; Na ₂ O 2.5; K ₂ O 11.8; Rb ₂ O 0.06; loss on ignition 0.40; all values % by wt.
76a Burnt Refractory	Provided by Harbison-Walker Refractories Co., Garber Research Center, Pittsburgh, Pennsylvania. Dobies were air dried at 120 °C, then fired at 1427 °C for 10 hours. The ground material was converted to a fine powder (95%, 325 mesh) and was thoroughly mixed.	SiO ₂ 54.9; Al ₂ O ₃ 38.7; Fe ₂ O ₃ 1.6; TiO ₂ 2.0; ZrO ₂ 0.15; MgO 0.52; CaO 0.22; K ₂ O 1.33; Na ₂ O 0.07; P ₂ O ₅ 0.12; Li ₂ O 0.042; SrO 0.037; loss on ignition 0.34; all values % by wt.
77a Burnt Refractory	Same as SRM 76a.	Si0 ₂ 35.0; Al ₂ 0 ₃ 60.2; Fe ₂ 0 ₃ 1.0; Ti0 ₂ 2.6; Zro ₂ 0.21; MgO 0.38; CaO 0.05; K ₂ O 0.09; Na ₂ O 0.037; P ₂ O ₅ 0.092; Li ₂ O 0.02; SrO 0.009; loss on ignition 0.22; all values % by wt.

Table 2. B. Rocks, Minerals, and Refractories - continued.

SRM	Material	Composition
78a Burnt Refractory	Same as SRM 76a.	SiO ₂ 19.4; Al ₂ O ₃ 71.7; Fe ₂ O ₃ 1.2; TiO ₂ 3.2; ZrO ₂ 0.31; MgO 0.70; CaO 0.11; K ₂ O 1.22; Na ₂ O 0.078; P ₂ O ₅ 1.3; Li ₂ O 0.12; SrO 0.25; Ioss on ignition 0.42; all vaues %
		by wt.
81a Glass Sand	Ground glass sand powder 95% less than 106 μm_{\bullet} Blended and dried at 105 $^{\circ} \text{C}$ for 2 hours before use.	Al ₂ O ₃ O.66; Fe ₂ O ₃ O.082; TiO ₂ O.12; ZrO ₂ O.034; all values % by wt. Cr ₂ O ₃ 46 μg/g.
<u> </u>		
88a Dolomitic Limestone	Limestone	Sample dried 2 hours at 110 °C. SiO ₂ 1.20; Al ₂ O ₃ 0.19; Fe ₂ O ₃ 0.28; TiO ₂ 0.02; MnO 0.03; CaO 30.1; SrO 0.01;
		MgO 21.3; Na,O 0.01; K ₂ O 0.12; P ₂ O ₅ 0.01; CO ₂ 46.6; loss on ignition 46.7; all values expressed as percent.
97a Flint Clay	Provided by A. P. Green Fire Brick Co., Mexico, Missouri.	Sample dried 2 hours at 140 °C. SiO ₂ 43.67; Al ₂ O ₃ 38.79; Fe ₂ O ₃ 0.45; TiO ₂ 1.90; P ₂ O ₅ 0.36; K ₂ O 0.50; Na ₂ O 0.037; Li ₂ O 0.11; BaO 0.07;
		MgO 0.15; CaO 0.11; SrO 0.18; Cr ₂ O ₃ 0.03; loss on ignition 13.32; all values are % by wt.
98a Plastic Clay	Same as SRM 97a.	Sample dried for 2 hours at 140 °C. SiO ₂ 48.94; Al ₂ O ₃ 33.19; Fe ₂ O ₃ 1.34; TiO ₂ 1.61; P ₂ O ₅ 0.11; K ₂ O 1.04; Na ₂ O ₃
		MgO 0.42; ² CaO 0.31; SrO 0.039; Cr ₂ 0 ₃ 0.03; loss on ignition 12.44; all values %
		by wt.
99a Feldspar		Sample dried at 105 °C for 2 hours. SiO ₂ 65.2; Al ₂ O ₃ 20.5; Fe ₂ O ₃ 0.06; TiO ₂ 0.007; CaO 2.14; BaO 0.26; MgO 0.02; Na ₂ O 6.2; K ₂ O 5.2; P ₂ O ₅
		0.02; loss on ignition 0.26; all values are % by wt.

Chemical analyses performed by Same as SRM 76a. The recommended data is the best estimate of the true value based on data from the cooperators and NBS. Issued in cooperation with ASTM. Revision of Certificate date 1-31-67. Chemical analyses performed by three cooperating laboratories including NBS-CAC. Same as SRM 76a. Zro_ was determined by only one of the cooperating laboratories and found to be 0.063 % by wt.				
Chemical analyses performed by hight cooperating laboratories and NBS-CAC. Chemical analyses performed by three cooperating laboratories and NBS-CAC. Chemical analyses performed by three cooperating laboratories and found to be 0.063 % by wt. Chemical analyses performed by three laboratories including NBS-CAC. Chemical analyses performed by three laboratories including laboratories and found to be 0.063 % by wt. Chemical analyses performed by three laboratories including laboratories and found to be 0.042 % by wt. Revision of Certificate date.	Certification	Use		Remarks
best estimate of the true value based on data from the cooperators and NBS. Issued in cooperation with ASTM. Revision of Certificate date 1-31-67. Chemical analyses performed by three cooperating laboratories including NBS-CAC. Chemical analyses performed by three laboratories including NBS-CAC. Same as SRM 76: ZrO ₂ was determined by only one of the cooperating laboratories and found to be 0.063 % by wt. ZrO ₂ was determined by only one of the cooperating laboratories and found to be 0.063 % by wt. ZrO ₂ was determined by only one of the cooperating laboratories including the cooperating laboratories and found to be 0.042 % by wt.	Same as SRM 76a.	Same as SRM 76a.		Same as SRM 76a.
best estimate of the true value based on data from the cooperators and NBS. Issued in cooperation with ASTM. Revision of Certificate date 1-31-67. Chemical analyses performed by three cooperating laboratories including NBS-CAC. Chemical analyses performed by three laboratories including NBS-CAC. Same as SRM 76: ZrO ₂ was determined by only one of the cooperating laboratories and found to be 0.063 % by wt. ZrO ₂ was determined by only one of the cooperating laboratories and found to be 0.063 % by wt. ZrO ₂ was determined by only one of the cooperating laboratories including the cooperating laboratories and found to be 0.042 % by wt.				
best estimate of the true value based on data from the cooperators and NBS. Issued in cooperation with ASTM. Revision of Certificate date 1-31-67. Chemical analyses performed by three cooperating laboratories including NBS-CAC. Chemical analyses performed by three laboratories including NBS-CAC. Same as SRM 76: ZrO ₂ was determined by only one of the cooperating laboratories and found to be 0.063 % by wt. ZrO ₂ was determined by only one of the cooperating laboratories and found to be 0.063 % by wt. ZrO ₂ was determined by only one of the cooperating laboratories including the cooperating laboratories and found to be 0.042 % by wt.				
best estimate of the true value based on data from the cooperators and NBS. Issued in cooperation with ASTM. Revision of Certificate date 1-31-67. Chemical analyses performed by three cooperating laboratories including NBS-CAC. Chemical analyses performed by three laboratories including NBS-CAC. Same as SRM 76: ZrO_ was determined by only one of the cooperating laboratories and found to be 0.063 % by wt. ZrO_ was determined by only one of the cooperating laboratories and found to be 0.063 % by wt. ZrO_ was determined by only one of the cooperating laboratories including the cooperating laboratories and found to be 0.042 % by wt. Revision of Certificate date				
Chemical analyses performed by hree cooperating laboratories and found to be 0.063 % by wt. Chemical analyses performed by hree laboratories including NBS-CAC. Chemical analyses performed by hree laboratories including laboratories including laboratories including laboratories including laboratories and found to be 0.042 % by wt. Revision of Certificate date	Chemical analyses performed by eight cooperating laboratories including NBS-CAC.	Same as SRM 76a		best estimate of the true value based on data from the cooperators and NBS. Issued
chemical analyses performed by Same as SRM 76a. Chemical analyses including TBS-CAC. Chemical analyses including TBS-CAC. Chemical analyses formed by Same as SRM 76a. Chemical analyses performed by Same as SRM 76a. Chemical analyses performed by Only one of the cooperating labor tories and found to be 0.042 % by wt. Chemical analyses performed by Same as SRM 76a. Chemical analyses performed by Only one of the cooperating labor tories and found to be 0.042 % by wt.				
chemical analyses performed by Same as SRM 76a. Chemical analyses including SBS-CAC. Chemical analyses including SBS-CAC. Chemical analyses formed by Same as SRM 76a. Chemical analyses formed by Same as SRM 76a. Chemical analyses performed by Only one of the cooperating labor tories and found to be 0.042 % by wt. Chemical analyses performed by Same as SRM 76a. Chemical analyses performed by Only one of the cooperating labor tories and found to be 0.042 % by wt.				
chemical analyses performed by Same as SRM 76a. Chemical analyses including SBS-CAC. Chemical analyses including SBS-CAC. Chemical analyses formed by Same as SRM 76a. Chemical analyses formed by Same as SRM 76a. Chemical analyses performed by Only one of the cooperating labor tories and found to be 0.042 % by wt. Chemical analyses performed by Same as SRM 76a. Chemical analyses performed by Only one of the cooperating labor tories and found to be 0.042 % by wt.				
TBS-CAC. tories and found to be 0.042 % by wt.	Chemical analyses performed by three cooperating laboratories including NBS-CAC.	Same as SRM 76a		one of the cooperating labor tories and found to be 0.063
TBS-CAC. tories and found to be 0.042 % by wt.				
Tories and found to be 0.042 % by wt. Revision of Certificate date				
Tories and found to be 0.042 % by wt. Revision of Certificate date			: 144 :	
	Chemical analyses performed by three laboratories including NBS-CAC.	Same as SRM 76a.		tories and found to be 0.042
				· ·

Table 2. B. Rocks, Minerals, and Refractories - continued.

SRM	Material	Composition
103a Chrome Refractory		Sample dried at 110 °C for 2 hours. Cr ₂ O ₃ 32.06; Al ₂ O ₃ 29.96; FeO ² 12.43; MnO 0.11; MgO 18.54; CaO 0.69; SiO ₂ 4.63; TiO ₂ 0.22; ZrO ₂ 0.01; P ₂ O ₅ 0.01; all values are % by wt.
154b Titanium Dioxide	Prepared by the National Lead Industries, Research and Development Dept., St. Louis, Missouri.	TiO 99.74% by wt. on samples dried 2 hours at 110 °C.
165a Glass Sand	Glass sand blended to insure homogeneity. Should be dried for 2 hours at 105 °C.	Al ₂ 0 ₃ 0.059; Fe ₂ 0 ₃ 0.012; Ti0 ₂ 0.011; Zro ₂ 0.006; all values are % by ² wt.
198 Silica Brick		Al ₂ O ₃ 0.16; Fe ₂ O ₃ 0.66; TiO ₂ 0.02; P ₂ O ₅ 0.022; MnO 0.008; CaO 2.71; MgO 0.07; Na ₂ O 0.012; K ₂ O 0.017; Li ₂ O 0.001; loss on ignition 0.21; all values % by wt.
199 Silica Brick		Al ₂ O ₃ 0.48; Fe ₂ O ₃ 0.74; TiO ₂ 0.06; P ₂ O ₅ 0.015; MnO 0.007; CaO 2.41; MnO 0.13; Na ₂ O 0.015, K ₂ O 0.094; Li ₂ O 0.002; loss on ignition 0.17; all values % by wt.
278 Obsidian Rock	Natural glass obtained from Clear lake, Newberry Crater, Oregon, and processed by the Colorado School of Mines, Golden, Colorado, to a powder of <200 mesh.	The material should be dried to constant weight at 350-600 °C. Al ₂ O ₃ 14.15; CaO 0.983; FeO 1.36; Fe ₂ O ₃ 2.04; K ₂ O 4.16; MnO 0.052; Na ₂ O 4.84; P ₂ O ₅ 0.036; SiO ₂ 73.05; TiO ₂ 0.245; all data wt %. Cu 5.9; Ni 3.6; Rb 127.5; Sr 63.5; Th 12.4; Tl 0.54; U 4.58; Pb 16.4; data wt (µg/g).

Chemical analyses performed by eight cooperating laboratories including NBS-CAC.

Certified primarily for use in the analysis of ores and geological materials containing chromium. For additional information on methods used for analysis and certification of constituent elements see certificate.

Analyses performed by three cooperating laboratories including NBS-CAC. TiO₂ was determined by controlled-potential coulometry and by volumetry. The uncertainty is 0.05 and represented the 95% confidence interval of the mean of all accepted values.

Certified primarily for application in the paint and ceramic industries.

For additional information on the material and its composition see Certificate.

Chemical analyses performed by eight cooperating laboratories including NBS-CAC.

The certified data is the best estimate of the true value based on the measurements from the cooperators and NBS. SRM 165a was issued in cooperation with ASTM.

Chemical analyses performed by twelve cooperating laboratories including NBS-CAC. All results are based on samples dried at 105-110 $^{\circ}\text{C}_{\bullet}$

One cooperating laboratory has reported a content of less than 0.01 $\rm ZrO_2$ % by wt.

Same as SRM 198.

One cooperating laboratory has reported a content of 0.01 $0.01~{\rm Zro}_2$ % by wt.

Chemical analyses performed by four cooperating laboratories including NBS-CAC. Uncertainty of measurements varies from ±0.3 to 0.002 depending on element.

For evaluation of the accuracy of analytical methods and instrumentation used in the analysis of geological type materials. Additional 21 constituents given but not certified (see certificate).

Table 2. B. Rocks, Minerals, and Refractories - continued.

SRM	Material	Composition
688 Basalt Rock	Obtained from a Cenozoic basalt flow near Jackpot, Nevada, and processed by the Colorado School of Mines, Golden, Colorado.	Sample dried at 105 °C for 24 hours. Al ₂ O ₃ 17.36; FeO 7.64; Fe ₂ O ₃ 10.35, K ₂ O 0.187; MnO 0.167; Na ₂ O 2.15, P ₂ O ₅ 0.134; SiO ₂ 48.4; TiO ₂ 1.17, all % by wt. Cr 332; Rb 1.91; Sr 169.2
		Th 0.33; Pb 3.3, all μg/g.
C. Ores		
27f Iron Ore (Sibley)	The iron ore was provided by the U.S. Steel Corp., Pittsburgh, Pennsylvania. The ore was crushed, dry ground, and sieved	Total Fe 65.97; SiO ₂ 4.17; Al ₂ O ₃ 0.82; P 0.041; S 0.005; TiO ₂ 0.019; MnO 0.011; CaO 0.039; MgO 0.019;
	at the Colorado School of Mine Research Institute, Golden, Colorado, to pass a 150 mesh sieve with about 50% passing a 200 mesh sieve. Analytical	Na ₂ 0 0.012; K ₂ 0 0.008; all values % by wt.
	samples were dried 1 hour at 105 °C.	
69b Bauxite (Arkansas)	Provided by the Aluminum Company of America, Bauxite, Arkansas. The material was processed at	The bauxite powder (<0.08 mm) was dried at 140 °C for 2 hours. Analytical sample of
	the Colorado School of Mine Research Institute. Mineral- ogical composition: 30% kaolinite, 60% gibbsite, 10%	1-g or more. Al ₂ O ₃ 48.8; Fe ₂ O ₃ 7.14; SiO ₂ 13.43; TiO ₂ 1.90; ZrO ₂ 0.29; P ₂ O ₅ 0.118; V ₂ O ₅ 0.028; Cr ₂ O ₃ 0.011; CaO 0.13; MgO 0.085; MnO 0.110;
	siderite (semiquantitative).	0.13; Mg0 0.085; Mn0 0.110; Zn0 0.0035; K ₂ 0 0.068; S0 ₃ 0.63; loss of ignition 27.2 at 1050 °C; all values % by wt.
		Estimated uncertainty 0.0005 to 0.2 depending on element.
79a Fluorspar	Provided by American Smelting and Refining Co., El Paso, Texas, and ground to pass an 80 mesh screen.	CaF ₂ 97.39 ± 0.06 % by wt.

Analyses performed by three cooperating laboratories including NBS-CAC. Uncertainties for % by wt. constituents: from 0.1 to 0.002; for µg/g constituents: 9 to 0.01.

For verifying the accuracy of chemical and instrumental methods of analysis used in the analysis of geological type materials.

Chemical analyses performed by seven cooperating laboratories including NBS-CAC using the methods described in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards. For use in verifying chemical methods of analysis and in calibrating instrumental methods of analysis.

Issued in cooperation with ASTM.

Chemical analyses performed by eleven cooperating laboratories including NBS-CAC.

For verification of chemicals and instrumental methods of analysis.

Additional composition (not certified): BaO (0.008); Na₂O (0.025); Ce (0.024); Co (0.0001); Hf (0.0063); Sc (0.0008). Issued in cooperation with ASTM.

Chemical analyses performed in cooperation with the U.S. Customs Laboratories, Division of Technical Services, following a standard procedure described in detail in the Certificate. To assay imported fluorspar for industrial applications.

Additional percent composition (not certified): Fe (0.05-0.1) Al and Sr (0.01-0.1); Mg (0.01-0.05); Na (0.001-0.01); Ba (0.001-0.005); K (<0.005); Cu, Li, Mn, Pb, and Ti (<0.001); Sio (0.67); all are % values. Revision of Certificate dated 12-6-71.

Table 2. C. Ores - continued.

SRM	Material	Composition		
113a Zinc Concentrate	Provided by Cominco American Inc., Spokane, Washington, and prepared at the Magmont Mines, Bixby, Missouri, in the form of powder (<0.15 mm).	Sample dried 1 hour at 105 °C. Zn 57.3; Co (0.11); Ni (0.07); S 30.6; Cd 0.78; Cu 0.31; Pb 2.80; Fe 2.08; CaO 1.1; MgO 0.75; SiO ₂ (1.54); Ag 0.046, all values are % by wt.		
120b Phosphate Rock (Florida)	Provided by American Cyanamid Co. as a powder to pass 200 mesh sieve.	Sample dried 1 hour at 105 °C. P ₂ O ₅ 34.57; CaO 49.40; SiO ₂ 4.68; F 3.84; Fe ₂ O ₃ 1.10; A1 ₂ O ₃ 1.06; MgO 0.28; Na ₂ O 0.35; MnO 0.032; K ₂ O 0.12-0.090; TiO ₂ 0.15; Co ₂ 2.79; CdO 0.002; all values are % by wt. Uranium 128.4 µg/g.		
180 High-Grade Fluorspar	Provided by Minera Frisco of San Francisco Del Oro, Chih., Mexico.	CaF ₂ 98.80 ± 0.03 % by wt.		
181, 182, 183 Lithium Ores	Fine powder of spodumene (SRM 181), petalite (SRM 182), and lepidolite (SRM 183).	Li ₂ 0 wt. % in SRM 181 - 6.3 ₉ ; SRM 182 - 4.3 ₄ ; and SRM 183 - 4.1 ₂ .		
277 Tungsten Concentrate	Provided by GTE Sylvania, Towanda, Pennsylvania, as a powder sieved (<0.15 mm) and blended. It is a mixture of con- centrates from China, Thailand, and USA.	WO_3 - 67.4 wt. %, determined on 31 -g or more sample.		
329 Zinc Concentrate	Provided by Cominco American Inc., Spokane, Washington, and prepared at the Sullivan Mine, Trail, B.C., Canada, in the form of powder (<0.15 mm).	Sample dried 1 hour at 105 °C. Zn 45.5; Pb 6.06; Fe 12.94; CaO 0.08; MgO 0.16; Cd 0.14; Cu 0.13; Co (0.009); Ni (0.006); S (31.7); SiO ₂ (0.61) In 0.019; Ag 0.0089; aI1 values are % by wt.		

Chemical analyses performed by Intended for the verification Figures in parentheses are not four cooperating laboratories of chemical and instrumental certified. Should be kept including NBS-CAC. The data methods of analysis. tightly closed except when in are not expected to deviate direct use. Store in a from the true value by more desiccator over desiccant. than +1 in the last significant figures reported. For the verification of chemical For potassium values see Chemical analyses performed by six cooperating laboratories optical emission and x-ray spec-Certificate. including NBS-CAC. Uranium was trometric methods of analysis. determined at NBS by thermal ionization mass spectrometry. Detailed information on the Additional trace elements Issued primarily for geochemical detected but not certified: method used for the determination of ${\tt CaF}_2$ is given in the Certificate. Fe 0.1-1.0; Al, Ba, Mg, Pb, Si, and Sr 0.01-0.1; Cu, K, Mn, Na, Ti, and V 0.001-0.01; Ag and Li less than 0.001; all values expressed in percent. The deviation from given values For verifying the accuracy of Revision of Certificate dated is not expected to be more than assay methods. 2-24-58. +5 subscript number. To verify the accuracy of Chemical analyses performed in Issued in cooperation with cooperation with 13 laborachemical and instrumental ASTM. tories including NBS-CAC. The methods of analysis. estimated uncertainty 0.3 wt. %. Additional elements indicated but not certified are: Ca, Fe, Pb, Mn, Nb, P, Si, S, Sn, Ti, As, Mo, Bi, Ce, Cr, Cu, Gd, La, Nd, O, Sc, Ta, Th, U, Y, and Zr. Chemical analyses performed by Intended for the verification Figures in parentheses are not four cooperating laboratories of chemical and instrumental certified. including NBS-CAC. The data methods of analysis. are not expected to deviate from the true value by more than +1 in the last significant figure reported.

Table 2. C. Ores - continued.

SRM	Material	Composition
330 Copper Ore, Mill Heads	Provided by Magma Copper Co., San Manuel, Arizona. It was sieved and blended at NBS.	Sample dried at 105 °C for 2 hours. Cu 0.84; Mo 0.018; both values % by wt. Re 0.30 ppm by wt. Not certified: Au (0.093); Ag (1.51) ppm by wt.
331 Copper Ore, Mill Tails	Provided by Magma Copper Co., San Manuel, Arizona.	Cu 0.091; Mo 0.0022; both % by wt. Re 0.04 ppm by wt. on samples dried at 105 °C for 2 hours. Not certified: Au (0.034); Ag (0.243) ppm by wt.
332 Copper Concentrate	Provided by Magma Copper Co., San Manuel, Arizona.	Cu 28.4; Mo 0.64 % by wt. Re 10.2 ppm by wt. on samples dried at 105 °C for 2 hours. Not certified: Au (2.14); Ag (38.7) ppm by wt.
333 Molybdenum Concentrate	Provided by Magma Copper Co., San Manuel, Arizona. Homo- geneity obtained by special blending and mixing procedures.	Cu 1.038; Mo 55.3; Re 0.087; all % by wt. on samples "as received." Not certified: Au (8.9); Ag (25.0) ppm by wt.
690 Iron Ore Concentrate (Canada)	Provided by the Iron Ore Company of Canada, Labrador City, Newfoundland, Canada, as a powder (<0.1 mm).	Fe 66.85; SiO ₂ 3.71; Al ₂ O ₃ 0.18; P 0.011; S 0.003; TiO ₂ 0.022; MnO 0.23; CaO 0.20; MgO 0.18; Na ₂ O 0.003; K ₂ O 0.0030, on samples dried at 105 °C for 1 hours. All values are % by wt.
691 Reduced Iron Oxide	Provided by Allis-Chalmers, Reduction Systems Div., Milwaukee, Wisconsin, and processed as a powder (-200 mesh) at the Colorado School of Mines Research Institute, Golden, Colorado.	Values based on 0.5-g or more sample size. Fe, total 90.8; Fe, metallic 84.6; SiO ₂ 3.7; Al ₂ O ₃ 1.22; TiO ₂ 0.27; CaO 0.53; MnO 0.043; MgO 0.52; Na ₂ O 0.186; Cu 0.032; Co 0.030 P 0.006; S 0.008; C 0.12; all values % by wt. Not certified 11 additional constituents (see certificate).
692 Iron Ore (Labrador)	Prepared by the Bethlehem Steel Corp., Bethlehem, Pennsylvania, as a fine powder (200 mesh).	Sample dried 1 hour at 105 °C, values based on 0.5-g or more sample. Fe 59.58; SiO ₂ 10.14; A1 ₂ O ₃ 1.41; P 0.039; S ² 0.005; TiO ₂ 0.045; MnO 0.46; CaO 0.023; MgO 0.035; Na ₂ O 0.008; K ₂ O 0.039, all values % by wt.

Analyses performed by two coop-For verifying the accuracy of chemical and instrumental Keep the material tightly erating laboratories including closed in a desiccator over NBS-CAC. Estimated uncertainty methods of analysis, and for desiccant. Revision of 0.01 and 0.001 for Cu and Mo, evaluation of the material Certificate of 2-20-73. and 0.06 for Re. balance in copper mining and metallurgical industries. Estimated uncertainty 0.001 for Same as SRM 330. Same as SRM 330. Cu; 0.0002 for Mo; and 0.02 for Re. Estimated uncertainty 0.1 for Same as SRM 330. Same as SRM 330, except Cu; 0.01 for Mo; and 0.2 for Re. revision of Certificate dated 2-20-73 and 1-20-77. Analyses performed by two Same as SRM 330. Same as SRM 330. cooperating laboratories in-cluding NBS-CAC. Estimated uncertainty 0.010 for Cu; 0.1 for Mo; 0.001 for Re. Analyses performed by six For verifying the chemical and Issued in cooperation with cooperating laboratories ininstrumental methods of ASTM. cluding NBS-CAC. Estimated analysis. uncertainty from 0.0005 to 0.01 depending on constituent. Analyses performed by six For the evaluation of chemical Material packaged in dry cooperating laboratories inand spectroscopic methods of nitrogen to prevent oxidation. cluding NBS-CAC. Estimated analysis. Should be stored in desiccator uncertainty 0.001 to 0.6 over desiccant. Issued in cooperation with ASTM. depending on constituent. Analyses performed by six For verifying the chemical and Issued in cooperation with cooperating laboratories ininstrumental methods of cluding NBS-CAC. Estimated analysis. uncertainty 0.001 to 0.06depending on constituent.

Table 2. C. Ores - continued.

SRM	Material	Composition
693 Iron Ore (Nimba)	Prepared by the Bethlehem Steel Corp., Bethlehem, Pennsylvania, as a fine powder (200 mesh).	Sample dried 1 hour at 105 °C, values based on 0.5-g or more sample. Fe 65.11; SiO ₂ 3.87; AI ₂ O ₃ 1.02; P 0.056; S 0.005; TiO ₂ 0.035; MnO 0.091; CaO 0.016; MgO 0.013; Na ₂ O 0.0028; K ₂ O 0.0028; all values % by wt
696 Bauxite (Surinam)	Same as SRM 69b. The material was mined in Surina, South America. Mineralogical composition: 5% kaolinite, 80% gibbsite, 10% pyrite, 5% anatase (semiquantitative).	The bauxite powder (<0.08 mm) was dried at 140 °C for 2 hours. Analytical sample of 1-g or more. Al 203 54.5; Fe 203 8.70; SiO 3.79; TiO 2.64; ZrO 2.0.14; P 20 0.050; V 20 0.072; Cr 20 0.047; CaO 0.018; MgO 0.012; MnO 0.004; ZnO 0.0014; K 20 0.009; SO 0.21; loss on ignition 29.9 at 1050 °C; all values % by wt. Estimated uncertainty 0.0007 to 0.3 depending on element.
697 Bauxite (Dominican)	Same as SRM 69b. The material was mined in the Dominican Republic. Mineralogical composition: 15% kaolinite, 50% gibbsite, 10% boehmite, 5% anatase; 20% hematite (semiquantitative).	The bauxite powder (< 0.08 mm) was dried at 140 °C for 2 hours. Analytical sample of 1-g or more. A1 ₂ 0 ₃ 45.8; Fe ₂ 0 ₃ 20.0; SiO ₂ 6.81; TiO ₂ 2.52; ZrO ₂ 0.065; P ₂ O ₃ 0.97; V ₂ O ₅ 0.063; Cr ₂ O ₃ 0.100; CaO 0.77; MgO 0.18; MnO 0.41; ZnO 0.037; K ₂ O 0.062; SO ₃ 0.13; loss on ignition 22.1 at 1050 °C. All values % by wt. Estimated uncertainty 0.2 to 0.003 depending on element.
698 Bauxite (Jamaican)	Mined in Jamaica, provided by Reynolds Metals Co., Bauxite, Arkansas, processed at the Colorado School of Mines Research Institute. Mineralogical composition: 75% gibbsite, 20% hematite, 5% anatase (semiquantitative).	The bauxite powder (<0.08 mm) was dried at 140 °C for 2 hours. Analytical sample of I-g or more. Al 20 48.2; Fe20 19.6; Si0 0.69; Ti0 2.38; Zr0 0.061; P20 0.37; V20 0.064; Cr20 0.080; Ca0 0.62; Mg0 0.058; Mn0 0.38; Zn0 0.029; K20 0.010; S0 0.22; loss on ignition 27.3 at 1050 °C. All values % by wt. Estimated uncertainty 0.002 to 0.4 depending on element.

Use

Remarks

Analyses performed in cooperation with five laboratories including NBS-CAC. Estimated uncertainty 0.0005 to 0.07 depending on constituent.

Same as SRM 692.

Same as SRM 692.

Same as SRM 69b.

Same as SRM 69b.

Additional composition (not certified): BaO (0.004);
Na₂O (0.007); Ce (0.0041);
Co (0.00009); Hf (0.0032);
Sc (0.0008). Issued in cooperation with ASTM.

Same as SRM 69b.

Same as SRM 69b.

Additional composition (not certified): BaO (0.015);
Na₂O (0.036); Ce (0.069);
Co (0.0013); Hf (0.0014);
Sc (0.0058). Issued in cooperation with ASTM.

Same as SRM 69b.

Same as SRM 69b.

Additional composition (not certified): BaO (0.008);
Na₂O (0.015); Ce (0.030);
Co²(0.0045); Hf (0.0015);
Sc (0.0051). Issued in cooperation with ASTM.

Table 3. Composition of SRM's Prepared from Ores, Minerals, Rocks, and Refractories (listed by chemical constituents and category).

Composition of ore SRMs a

	SRM type, wt/unit						
	271	690	691	692	693	69b	
	Iron ore, Sibley,	iron ore, Canada,	Iron oxide, reduced,	iron ore, Labrador,	Iron ore, Nimba,	Bauxite, Ark.	
Constituents	100 g	150 g	100 g	150 g	150 g	60 g	
Al ₂ O ₃	0.82	0.18	1.22	1.41	1.02	48.8	
BaO	_	_	_	-	_	d(800.0)	
C (Total)			.12	-	· -		
Cd	_	-	· -		_	_	
CdO			_		_		
CaO	.039	.20	.63	.023	.016	.13	
Co	-		.030	- '		(.0001)	
CO2	<u>-</u>	_	-		· _	·_ ′	
Cu	_		.032		_	_	
Cr ₂ O ₃	_	_	_		, , 	.011	
F	_			_			
In	_	_		-	_	_	
Hf .					_	(.0063)	
Fe (Total)	65.97	66.85	90.8	59.58	65.11	`- `	
Fe ₂ O ₃		_		_	_	7.14	
Pb			_				
Ce	_	-	_		_	(0.024)	
MgO	0.019	0.18	0.52	0.035	0.013	.085	
MnO	.011	.23	.043	.46	.091	.110	
Р	.041	.011	.006	.039	.056		
Ni			_		_	_	
P ₂ O ₅	·	_		· · ·	_	.118	
K₂O	.008	.0030	· _	.039	.0028	.068	
SiO ₂	4.17	3.71	3.7	10.14	3.87	13.43	
Ag	· _	_	-	_		_	
Na ₂ O	0.012	0.003	0.186	0.008	0.0028	(0.025)	
S	.005	.003	.008	.005	.005	(0.023)	
Sc					.005	(8000.)	
SO ₃		_	_			.63	
TiO ₂	.019	.022	.27	.045	.035	1.90	
U	.013	.022		.043	.033	1.50	
V ₂ O ₅	_			<u> </u>		0.028	
WO ₃					-	0.028	
Zn			<u> </u>		· -	-	
ZnO	. —	- -				.0035	
ZrO ₂	_	· <u>-</u>	_	_	_	.0035	
Loss on		_			_	.29 27.2	
ignition		_	_	-		21.2	
Moisture	_	· -	_	_	_	_	

	696 Bauxite, Surinam,	697 Bauxite, Dominican,	698 Bauxite,	RM type, wt/unit 120b Phosphate rock, Fla.,	277 Tungsten	113a Zinc ore	329 Zinc
Constituents	60 g	60 g	Jamaican, 60 g	90 g	conc., 100 g	conc., 100 g	conc., 100 g
Al ₂ O ₃	54.5	45.8	48.2	1.06		_	
BaO	(0.004)	(0.015)	(800.0)	_	Ta (0.20)	_	
C (Total)	-	_	_				
Cd	_	_		_	-	0.78	0.14
CdO		· -		0.002	. -	_	
CaO	0.18	.71	.62	49.40	Ca (.37)	1.19	.08
Co	(.00009)	(.0013)	(.0045)		_ `	(0.11)	(.009)
CO2	_	-		2.79	_		-
Cu	_	_			_	0.31	132
Cr ₂ O ₃	.047	.100	.080	_	Nb (1.00)	_	
F		· - .		3.84	<u></u>	_	_
In	_	-		_	_		.019
Hf	(.0032)	(.0014)	(.0015)	- -	_	· -	_
Fe (Total)	_				(7.4)	2.08	12.9₄
Fe ₂ O ₃	8.70	20.0	19.6	1.10		· —	_
Pb			_	-	(0.07)	2.80	6.06
Ce	(0.0041)	(0.069)	(0.030)	. —			_
MgO	.012	.18	.058	0.28	_	0.75	0.165
MnO	.004	.41	.38	.032	Mn (10.0)	_	
Р		_		_	(0.03)	_	_
Ni		_	_	_		(.07)	(.006)
P2Os	.050	.97	.37	34.57		`	`- `
K₂O -	.009	.062	.010	0.12	_		
SiO ₂	3.79	6.81	.69	4.68	Si (.85)	(1.54)	(.61)
Ag				_	_ ` `	0.0467	.0089
Na ₂ O	(0.007)	(0.036)	(.015)	0.35		_ '	
s	`-'	` _ ′	`_ `	_	(.25)	30.6	(31.7)
Sc	(8000.)	(.0058)	(.0051)	_	_ ` `	· <u></u>	
SO ₃	.21	.13	.22	_	O ₂ (21.4)		-
TiO ₂	2.64	2.52	2.38	.15	Ti (2.2)		_
U	_	- <u>-</u>	_	128.4 µg/g	_` `	_	_
V ₂ O ₅	0.072	0.063	0.064		Mo (0.06)	_	_
WO ₃					67.4	-	
Zn	_	_	_	_	_	57. ₃	45. ₅
ZnO	.0014	.037	.029		Sn (0.54)	-	_
ZrOz	.14	.065	.061	_		_	_
Loss on ignition	29.9	22.1	27.3	_	_	-	·
Moisture	_		-	· 	-	0.08	0.45

^aConcentrations expressed in wt% unless noted otherwise. ^bValues in parentheses are not certified, but are given for information only.

Composition of rock, mineral, and refractory SRMs $^{\it a}$

				SRM type	, wt/unit			
Constituents	1c Limestone, argiliaceous, 50 g	88a Limestone, dolomitic, 50 g	70a Feldspar, potash, 40 g	99a Feldspar, soda, 40 g	97a Clay, flint 60 g	98a Clay, plastic 60 g	81a Glass sand, 75 g	165a Glass sand, (low iron), 75 g
Al ₂ O ₃	1.30	0.19	17.9	20.5	38.79	33.19	0.66	0.059
BaO	. —	. —	0.02	0.26	0.075	0.03	_	_
CaO	50.3	30.1 ₅	.11	2.14	.11	.31	_	· -
Cr ₂ O ₃	_	_			.03	.03	46 μg/g	(1.1) ^b μg/g
CO ₂	_	46.6	_	_		-	_	
FeO	_	-	_		_		_	_
Fe ₂ O ₃	0.55	0.28	0.075	0.065	0.45	1.34	0.082	0.012
Li₂O		·	_ `	_ `	.11	0.070		-
MgO	0.42	21.3	_	.02	.15	.42	_	_
MnO	.025	0.03	_		_		-	_
P ₂ O ₅	.04	.01	_	.02	.36	.11		_
K₂O	.28	.12	11.8	5.2	.50	1.04		<u>-</u>
Rb ₂ O	_	_	0.06	, —	· —		-	_
SiO₂	6.84	1.20	67.1	65.2	43.67	48.94	_	
Na₂O	0.02	0.01	2.5 ₅	6.2	0.037	0.082		'
SrO	.030	.010		_	.18	.039		
TiO ₂	.07	.02	0.10	0.007	1.90	1.61	.12	.011
ZrO₂	_	_		_	_		.034	.006
Loss on ignition	39.9	46.7	.40	.26	13.32	12.44	- -	-

	SRM type, wt/unit								
	154b	278	688	76a Burnt	77a Burnt	78a Bumt	103a	198	199
Constituents	Titanium dioxide, 90 g	Obsidian rock, 35 g	Basalt rock, 60 g	refractory, (Al ₂ O ₃ —40%) 75 g	refractory, (Al ₂ O ₂ —60%) 75 g	refractory, (Al ₂ O ₃ —70%) 75 g	Chrome refractory, 60 g	Silica refractory, 45 g	Silica refractory, 45 g
Al ₂ O ₃		14.15	17.36	38.7	60.2	71.7	29.96	0.16	0.48
BaO		-	-	-	_	_	-		
CaO		0.983		0.22	0.05	0.11	0.69	2.71	2.41
Cr ₂ O ₃	_	_	_	_	· · -	_	32.06		
CO2	_	_	_		_	_	:	_	_
FeO		_	. —		_		12.43		_
Fe ₂ O ₃	-	2.04	10.35	1.6 ₀	1.00	1.2	_	0.66	0.74
Li₂O	_	_	_	0.042	0.25	0.12	-	.001	.002
MgO		_	_	.52	.38	.70	18.54	.07	.13
MnO	_	0.052	0.167	_			0.11	.008	.007
P ₂ O ₅	_	.036	.134	0.120	0.092	1.3	.01	.022	.015
K₂O		4.16	.187	1.33	.09 ₀	1.22	_	.017	.094
Rb₂O	_	_	_	_	_ ~	-	_	_	_
SiO ₂	-	73.05	48.4	54.9	35.0	19.4	4.63	_	_
Na₂O	_	4.84	2.15	0.07	0.037	0.078	-	.012	.015
SrO	_		_	.037	.009	.25	_	_	. —
TiOz	99.74	0.245	1.17	2.03	. 2.6 ₆	3.22	0.22	.02	.06
ZrO ₂	-		_	0.15	0.21	0.31	0.01		
Loss on ignition	-	_	- .	(0.34)	(0.22)	(0.42)	_	.21	.17

[&]quot;Concentrations expressed in wt% unless noted otherwise.
"Numbers in parentheses are not certified, but are given for information only.

Composition of ore standard reference materials a

	SRM, Type, wt/unit								
	79a Fluorspar,	180 Fluorspar,	181 Lithium	182 Lithium	183 Lithium	330 Copper	331 Copper	332	333 Molyb-
Constitu- ents	customs grade, 120 g	high grade, 120 g	ore (spodumene), 45 g	ore (petalite), 45 g	ore (lepidolite), 45 g	ore mill heads, 100 g	ore mill tails, 100 g	Copper conc., 50 g	denum conc., 35 g
CaF ₂	97.39	98.80	_	_	_	-	. —		
Li₂O			6.3 ₉	4.3	4.1	-	_	_	_
Cu	_	_		_	_	0.84	0.091	28.4	1.038
Re	_		. - .	_	· —	.30 ppm	.04 ppm	10.2 ppm	.087
Mo	_	_	_	_	_	.018	.0022	.64	55.3
Au	_			-	-	(0.93) ppm ^b	(.034) ppm	(2.14) ppm	(8.9) ppm
Ag	_	-			-	(1.51) ppm	(.243) ppm	(38.7) ppm	(25) ppm

^aConcentrations expressed in wt percent unless noted otherwise. ^bValues in parentheses are not certified, but are given for information only.

Table 4. NBS Publications in the "260 Series" Related to Coal, Ore, Mineral, Rock, and Refractory Standards

Publication	<u>Title</u>	SRM No.
260-8	Analysis of Uranium Concentrates at the National Bureau of Standards	
260-37	Methods of Analysis of NBS Clay Standards	97a, 98a
260-94	Methods and Procedures Used at the National Bureau of Standards to Certify Sulfur in Coal SRM's for Sulfur Content, Calorific Value, Ash Content	2682, 2683, 2684, 2685

ACKNOWLEDGMENT

The authors gratefully acknowledge the contributions of Ms. Julie Frum who prepared this manuscript for publication.

Appendix I.

Alphabetical Index by Standard Reference Material Name

Name	SRM	Name	SRM
Acetanilide	141c	Aluminum, Freezing Point Standard	44f
Acid Open-Hearth Steel, 0.2% Carbon	19G	Aluminum, Magnetic Gram	763
Acid Potassium Phthalate	8 4 j	Susceptibility	
AISI 1045 Steel	20g	Aluminum Oxide, Melting Point	742
AISI 4340 Steel	361	Aluminum Rod Ultra Purity	RM 1R
AISI 4340 Steel	1261a	Aluminum-26 Radioactivity Standard	4229
AISI 94B17 Steel (Modified)	362	Americium-241 Alpha-Particle	4904F
AISI 94B17 Steel (Modified)	1262a	Standard	
Albacore Tuna	RM 50	Americium-241 Gamma ray Standard	4213
Alkali Lead Silicate Glass	712	Ammonium Dihydrogen Phosphate	194
Alpha Quartz	1878	Angiotensin I (Human)	998
Alumina (Reduction Grade)	699	Anisic Acid	142
Alumina Silicate Glass	714	Anticonvulsant Drug Level Assay	1599
Aluminosilicate Glass	715	Standard	
Aluminum Allov	85B	Antiepilepsy Drug Level Assay	900
Aluminum Alloy 6011 (Modified)	858	Standard	
Aluminum Alloy 6011 (Modified)	1258	Antimony-125-Tellurium-125m,	4275B
Aluminum Alloy 7075	859	Europium-154, Europium-155 Mixed-	
Aluminum Alloy 7075	1259	Radionuclide Point-Source Standard	
Aluminum Block, Eddy Current	1860	Antimony-125-Tellurium-125m,	4276B
Conductivity		Europium-154, Europium-155 Mixed-	
Aluminum Block, Eddy Current	1861	Radionuclide Solution Standard	
Conductivity		A.O.H., 0.4C Spectrographic Steel	413
Aluminum Block, Eddy Current	1862	Standard	
Conductivity		Argillaceous Limestone	1C
Aluminum Block, Eddy Current	1863	Arsenic Trioxide Reductometric	83d
Conductivity	1000	Standard	
Aluminum Brass Standard for	1118	Assay-Isotopic Standard for Potassium	985
Optical Emission and X-ray	1110	Assay-Isotopic Standard for Rhenium	989
Spectroscopic Analysis		Assay-Isotopic Standard for Silicon	990
Aluminum Brass Standard for	C1118	Assay-Isotopic Standard for Strontium	987
Optical Emission and X-ray	0	2% Austenite in Ferrite	488
Spectroscopic Analysis		5% Austenite in Ferrite	485a
Aluminum Brass Standard for	1119	15% Austenite in Ferrite	486
Optical Emission and X-ray	1117	30% Austenite in Ferrite	487
Spectroscopic Analysis			
Aluminum Brass Standard for	C1119		
Optical Emission and X-ray	CIII)		
Spectroscopic Analysis			
Aluminum Casting Alloy 356	855		
Aluminum Casting Alloy 380	856		
Aluminum Cube Ultra Purity	RM 1C		
Aluminum Cube Ottra Purity Aluminum 2-Ethylhexanoate	1075a		
Adminum 2-Etnymexanoate	10/3a		

Name	SRM	Name	SRM
Austenitic Stainless Steel, Thermal	1460	Beryllium on Filter Media	2675
Conductivity and Electrical		Bessemer Steel (Simulated)	8j
Resistivity		0.1% Carbon	
Austenitic Stainless Steel, Thermal	1461	Bilirubin	916
Conductivity and Electrical		Bis(1-phenyl-1, 3-butanediono)	1080a
Resistivity		copper (II)	
Austenitic Stainless Steel, Thermal	1462	Bis(1-phenyl-1, 3-butanediono)	1052ь
Conductivity and Electrical		oxovanadium (IV)	
Resistivity Barium Crown Glass	712	Black Porcelain Enamel for Directional	2021
	713	Hemispherical Reflectance	2022
Barium Cyclohexanebutyrate	1051b	Black Porcelain Enamel for Directional	2022
Barrium-133 Radioactivity Point-Source Standard	4241B	Hemispherical Reflectance	1143-
Barium-133 Radioactivity Standard	4251B	Blast Furnace Iron Standard (Chill Cast White)	1143a
Basalt Rock	688 ·		1144.
Base Oil	1083	Blast Furnace Iron Standard	1144a
Basic Electric Spectrographic Steel	1063 404a	(Chill Cast White)	417.
Standard	404a	B.O.H., 0.4C Spectrographic Steel	417a
Basic Open-Hearth Steel, 0.1% Carbon	15g	Boric Acid	951
Basic Open-Hearth Steel, 0.1% Carbon	335	Boron-Doped Silicon Slices for	1521
Basic Open-Hearth Steel, 0.1% Carbon	1228	Resistivity Measurements	1321
Basic Open-Hearth Steel, 0.2% Carbon	11h	Borosilicate Glass	93a
Basic Open-Hearth Steel, 0.4% Carbon	12H	Borosilicate Glass	623
Basic Open-Hearth Steel, 0.5% Carbon	152A	Borosilicate Glass	717
Basic Open-Hearth Steel, 0.8% Carbon	14f	Borosilicate Glass	1825
Basic Open-Hearth Steel, 1% Carbon	1227	Borosilicate Glass, Thermal Expansion	731
(Disk)		Bovine Liver	1577a
Basic Open-Hearth Steel, 1.1% Carbon	16f	Bovine Serum Albumin	926
Basic Open-Hearth Steel, 1.1% Carbon	337	Bovine Serum Albumin (7% Solution)	927
0.4C Basic Oxygen Furnace Steel	178	Branched Polyethylene	1476
Bauxite (Arkansas)	69Ь	Brewers Yeast	1569
Bauxite (Dominican)	697	Bright Copper Microhardness	1894
Bauxite (Jamaican)	698	Standard	
Bauxite (Surinam)	696	Bright Nickel Microhardness Standard	1895
Benzene in Nitrogen	1805	Bromobenzoic Acid	2142
Benzene in Nitrogen	1806	Burnt Refractory	76a
Benzene Permeation Device	1911	Burnt Refractory	77a
Benzoic Acid	140ь	Burnt Refractory	78a
Benzoic Acid	350a	Cadmium Cyclohexanebutyrate	1053a
Benzoic Acid Calorimetric Standard	39i	Cadmium, Vapor Pressure	746
Benzothiazyl Disulfide Rubber	373f	Calcium Carbonate	915
Compound	1100	Calcium 2-Ethylhexanoate	1074a
Beryllium-Copper Standard	1122	Calcium in Low-Alloy (Silicon) Steel	1254
Beryllium-Copper Standard	C1122	Calcium Molybdate	71
Beryllium-Copper Standard	C1123	Calibrated Glass Beads	1004
		Calibrated Glass Beads	1017a
		Calibrated Glass Beads	1018a
		Calibrated Glass Spheres	1003a
		Carbon Dioxide in Air	1670
		Carbon Dioxide in Air	1671
		Carbon Dioxide in Air	1672
		Carbon Dioxide in Nitrogen	16746
		Carbon Dioxide in Nitrogen	1675ь
		Carbon Dioxide in Nitrogen	2619a
		(Combustion Efficiency Gas Standard)	2620
		Carbon Dioxide in Nitrogen	2620a
		(Combustion Efficiency Gas Standard)	

Name	SRM	Name	SRM
Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2621a	Catalyst Package for Lubricant Oxidation	1817
Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2622a	Centerline Drawings for Optical Character Recognition, B	1901
Carbon Dioxide in Nitrogen	2623a	Characters	
(Combustion Efficiency Gas Standard)		Centroid Color Chart	2106
Carbon Dioxide in Nitrogen	2624a	Centroid Color Kit	2107
(Combustion Efficiency Gas Standard)		Cesium-137, Barium-137m Point-Source	4200B
Carbon Dioxide in Nitrogen	2625a	Radioactivity Standard	
(Combustion Efficiency Gas Standard)	2626	Cesium-137, Barium-137m Point-Source	4207
Carbon Dioxide in Nitrogen	2626a	Radioactivity Standard	4032D
(Combustion Efficiency Gas Standard) Carbon Dioxide in Nitrogen (Mobile	2632	Cesium-137 Burn-Up Standard Cesium-134 Radioactivity Standard	4233B 4250B
Source Emission Gas Standard)	2032	Channel Black Rubber Compound	4250В 375g
Carbon Dioxide in Nitrogen (Mobile	2633	Chlorine-36 Beta-ray Standard	4943
Source Emission Gas Standard)		Chlorine-36 Radioactivity Standard	4422L
Carbon Monoxide in Air (Ambient	2612a	Chlorobenzoic Acid	2144
Air Quality Gas Standard)		Chrome Refractory	103a
Carbon Monoxide in Air (Ambient Air Quality Gas Standard)	2613a	Chromium-Molybdenum-Aluminum Steel	106B
Carbon Monoxide in Air (Ambient	2614a	Chromium-Molybdenum Steel	36b
Air Quality Gas Standard)		Chromium-Molybdenum Steel	133B
Carbon Monoxide in Nitrogen	1677c	Chromium-Nickel-Molybdenum Steel	139ь
Carbon Monoxide in Nitrogen	1678c	Chromium-Nickel-Molybdenum Steel	1222
Carbon Monoxide in Nitrogen	1679c	17Chromium-9 Nickel-0.2 Selenium Steel	
Carbon Monoxide in Nitrogen	1680b	Chromium-Nickel Spectrographic Steel Standard	408a
Carbon Monoxide in Nitrogen Carbon Monoxide in Nitrogen (Mobile	1681b	15 Chromium-7 Nickel Steel	344
Source Emission Gas Standard)	2635	16 Chromium-4 Nickel Steel	345
Carbon Monoxide in Nitrogen (Mobile	2636	Chromium-51 Radioactivity Standard	4400L-F
Source Emission Gas Standard)	2030	Chromium Steel	163
Carbon Monoxide in Nitrogen (Mobile	2637	Chromium-Tungsten Steel	155
Source Emission Gas Standard)	2007	Chromium-Vanadium Spectrographic	407a
Carbon Monoxide in Nitrogen (Mobile	2638	Steel Standard	
Source Emission Gas Standard)		Cholesterol	911a
Carbon Monoxide in Nitrogen (Mobile	2639	Chrysotile Asbestos Fibers	1876
Source Emission Gas Standard)		Citrus Leaves	1572
Carbon Monoxide in Nitrogen (Mobile	2640	Clinical Laboratory Thermometer	934 1055b
Source Emission Gas Standard)		Cobalt Cyclohexanebutyrate Cobalt-Molybdenum-Tungsten Steel	10336 153A
Carbon Monoxide in Nitrogen (Mobile	2641	Cobalt-Molybdenum-Tungsten Steel Cobalt-57 Radioactivity Standard	133A 4408L-C
Source Emission Gas Standard) Carbon Monoxide in Nitrogen (Mobile	2642	Cobalt-60 Radioactivity Standard	4915D
Source Emission Gas Standard)	2642	Commerical Bronze Standard for	1115
Carbon-14 Radioactivity Standard	4245	Optical Emission and X-ray	
Carbon-14 Radioactivity Standard	4245 4246	Spectroscopic Analysis	
Carbon Steel	1224	Commercial Bronze Standard for	C1115
Carbon Steel, 0.6%	13g	Optical Emission and X-ray	
Cast Iron	4k	Spectroscopic Analysis	
Cast Iron	5L	Commercial Bronze Standard for	1116
Cast Iron	6g	Optical Emission and X-ray	
Cast Iron	7Ğ	Spectroscopic Analysis	
Cast Iron Car Wheel	122h		
Cast Steel 3	C1173		
Cast Steel Standard	1138a		
Cast Steel Standard	1139a		

Name	SRM	Name	SRM
Commercial Bronze Standard for	C1116	Cupro-Nickel, 10% (CDA 706) High	874
Optical Emission and X-ray		Purity	
Spectroscopic Analysis		Cystine	143c
Commercial Bronze Standard for	1117	Dextrose	41b
Optical Emission and X-ray		D-Glucose	917
Spectroscopic Analysis	C1117	Dibutyltin Bis(2-ethylhexanoate)	1057Ь
Commercial Bronze Standard for	CHI	Didymium Glass Filter for Checking	2009
Optical Emission and X-ray		the Wavelength Scale of	
Spectroscopic Analysis		Spectrophotometers	2010
Common Lead Isotopic Standard	981	Didymium Glass Fitler for Checking	2010
Copper Concentrate Copper Heat Capacity Test Specimen	332	the Wavelength Scale of Spectrophotometers	
Copper-Nickel-Chromium Cast Iron	RM5	Disodium Hydrogen Phosphate	186IIc
Copper Ore, Mill Heads	115A 330	Disodium Hydrogen Phosphate	2186II
		D-Mannitol	920
Copper Ore, Mill Tails Copper-Thermal Expansion	331	Dolomitic Limestone	88a
Copper, Secondary Freezing Point	736a 45d	Doped Platinum	681L1
Standard	430	Doped Platinum	681L2
Cortisol (Hydrocortisone)	921	Ductile Cast Iron	341
Creatinine	914	Electrical Residual Resistivity Ratio	769
Cr-Mo Low Alloy Steel	1270	Standard	705
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	1403
Steel Standard	127	Resistivity	
Cr-Mo (SAE X4130) Spectrographic	418a	Electrolytic Iron, Thermal	1464
Steel Standard		Conductivity and Electrical	1101
Cr-Ni-Mo Steel (AISI 8620)	293	Resistivity	
18Cr-10Ni Steel (AISI 304L)	101f	Electronic and Magnetic Alloy	1159
Cr-V Steel (Modified)	363	Standard	,
Cr-V Steel (Modified)	1263a	Electronic and Magnetic Alloy	1160
Cr-V Steel (SAE 6150)	306	Standard	
Crystalline Potassium Dichromate	935	Enriched Boric Acid	952
Crystalline Potassium Iodide,	2032	Equal-Atom Lead Isotopic Standard	982
Heterochromatic Stray Radiant		Estuarine Sediment	1646
Energy Standard		Europium-152 Point-Source Standard	4218E
Crystalline (Ruby) Electron	2601	Europium-152 Radioactivity Standard	4370B
Paramagnetic Resonance		Extra Dense Lead Glass	709
Absorption Intensity Standard		Fe-Cr-Ni Alloy Microprobe Standard	479a
Cupro-Nickel (CDA 706)	1275	Fe-3Si Alloy Microprobe Standard	483
Cupro-Nickel (CDA 715)	1276	Feldspar	70a
Cupro-Nickel, 10% (CDA 706) Doped	875	Feldspar	99a
		Ferrochromium (Low Carbon)	196
		Ferrochromium Silicon	689
		Ferroniobium	340
		Ferrophosphorus	90
		Ferrosilicon	58a
		Ferrosilicon	39a
		Ferrosilicon (75% Si)	195
		First Surface Aluminum Mirror for	2003a
		Specular Reflectance	
		First Surface Mirror, Gold on Glass	2008a

Name	SRM	Name	SRM
Fission Track Glass Standard	961	Gold-198 Radioactivity Standard	4405L-B
Fission Track Glass Standard	962a	Gold-Silver Wires for Microprobe	481
Fission Track Glass Standard	963a	Analysis	
Fission Track Glass Standard	964	Gold, Vapor Pressure	745
Flint Clay	97a	Gray Cast Iron	334
Fluorobenzoic Acid	2143	Halocarbons (in methanol) for Water	1639
Fluorspar	79a	Analysis	
Free-Cutting Brass	1103	High-Alloy Steel (A-743)	C1288
Free-Cutting Brass	C1104	High-Alloy Steel (AISI 310 Mod.)	C1287
Freeze-Dried Urine	2670	High Alloy Steel, (AISI 414 Mod.)	C1289
Freeze-Dried Urine Certified	2671a	High-Alloy White Cast	892
for Fluoride		High-Alloy White Cast Iron	890
Freeze-Dried Urine Certified	2672a	High-Alloy White Cast Iron	891
for Mercury		High-Carbon Ferrochromium	64c
Fused-Silica Thermal Expansion	739	High-Carbon Ferromanganese	68c
Gadolinium-148 Alpha-Particle	4907	High-Carbon Steel (Modified)	364
Standard		High-Carbon Steel (Modified)	1264a
Gallium Melting-Point Standard	1968	High-Grade Fluorspar	180
Gallium-67 Radioactivity Standard	4416L-D	High-Nickel Steel	126c
Gas Furnace Black Rubber Compound	382a	High-Nickel Steel	1158
Gasometric Set (1095-1099)	1089	High-Purity Gold	685
Gasometric Standard for Unalloyed	357	High-Purity Platinum	680L1A
Zirconium		High-Purity Platinum	680L2A
Gasometric Standard for Unalloyed	358	High-Purity Platinum Thermoelement	1967
Zirconium		High-Purity Zinc	682
Generator Columns for Polynuclear	1644	High-Silicon Steel	179
Aromatic Hydrocarbons		High-Silicon Steel	1134
Gilding Metal	1112	High-Silicon Steel	1135
Gilding Metal	C1112	High-Silicon Steel (Calcium Bearing)	125b
Gilding Metal	1113	High-Sulfur Steel	105
Gilding Metal	C1113	High-Sulfur Steel	129c
Gilding Metal	1114	High-Sulfur Steel	1136
Gilding Metal	C1114	High Temperature Alloy A286	348
Glasses for Microchemical Analysis	1871	High Temperature Alloy M308	1197
Glasses for Microchemical Analysis	1872	High Temperature Alloy L605 and	S1199
Glasses for Microchemical Analysis	1873	S816	
Glasses for Microchemical Analysis	1874	High-Temperature Alloy	1206-2
Glasses for Microchemical Analysis	1875	High-Temperature Alloy	1207-1
Glass Fibers for Microanalysis	RM 31	High-Temperature Alloy	1207-2
Glass Filter for Transmittance	2030	High-Temperature Alloy	1208-1
Measurement		High-Temperature Alloy	1208-2
Glass Filters for Spectrophotometry	930 D	Homogeneous River Sediment for	RM 45B
Glass Fluorescence Source	477	Radioactivity Measurements	
Glass Sand	81a	Human Liver, Environmental	4352
Glass Sand	165a	Radioactivity	
Glass Spheres	1019a	Human Lung, Environmental	4351
Gold Coating on Glass Sealing Alloy	1398a	Radioactivity	
Gold Coating on Nickel	1379	Human Serum	909
Gold Coating on Nickel	1380		
Gold Coating on Nickel	1399ь		
Gold-Copper Wires for Microprobe Analysis	482		
Gold-195 Radioactivity Standard	4421L		

Name	SRM	Name	SRM
Hydrogen in Unalloyed Titanium	352b	Iron Ore (Sibley)	27f
Hydrogen in Unalloyed Titanium	1086	Iron Ore Concentrate (Canada)	690
Hydrogen in Unalloyed Titanium	1087	Iron-59 Radioactivity Standard	4411L-B
Hydrogen in Unalloyed Titanium	1088	Isobutylene-Isoprene (Butyl) Rubber	1495
Hydrogen-3 Radioactivity Standard	4361	Isobutylene-Isoprene (Butyl) Rubber	388L
Hydrogen-3 Radioactivity Standard	4926C	Isotopic Standard for Bromine	977
Hydrogen-3 Toluene Radioactivity	4947	Isotopic Standard for Chlorine	975
Standard		Isotopic Standard for Chromium	979
4-Hydroxy-3 methoxy-DL-mandelic	925	Isotopic Standard for Copper	976
Acid (VMA)		Isotopic Standard for Magnesium	980
ICTA High Temperature Set	GM 760	Isotopic Standard for Silver	978
Differential Thermal Analysis		Krypton-85 Gaseous Radioactivity	4308C
ICTA Low Temperature Set Differen-	GM 757	Standard	
tial Thermal Analysis		Krypton-85 Radioactivity Standard	4235
ICTA Mod Temperature Set Differen-	GM 759	Krypton-85 Radioactivity Standard	4935C
tial Thermal Analysis		Lead-Barium Glass	89
ICTA Mid Temperature Set Differen-	GM 758	Lead-Base Bearing Metal	53e
tial Thermal Analysis		Lead-Base Bearing Metal	1132
ICTA Polystyrene Differential	GM 754	Lead Cyclohexanebutyrate	1059c
Thermal Analysis		Lead in Reference Fuel	1636a
ICTA Thermogravimetry Set	GM 761	Lead in Reference Fuel	1637a
Incoloy, 901 and Hastelloy X	S1198	Lead in Reference Fuel	1638a
Inconels, Alloy 600 (Chips)	864	Lead Nitrate	928
Inconels, Alloy 600 (Solid)	1244	Lead on Filter Media	2674
Inconels, Alloy 625 (Chips)	865	Lead-203 Radioactivity Standard	4420L
Inconels, Alloy 625 (Solid)	1245	Lead, Secondary Freezing Point Standard	49e
Incoloy, Alloy 800 (Chips)	866	Lead-Silica Glass	1027
Incoloy, Alloy 800 (Solid)	1246		1827 711
Incoloy, Alloy 825 (Chips)	867	Lead-Silica Glass (Viscosity) Lead-Silica Glass for dc Volume	624
Incoloy, Alloy 825 (Solid)	1247		024
Indium-111 Radioactivity Standard	4417L-C	Resistivity	774
Ingot Iron Spectrographic Steel Standard	420a	Lead-Silica Glass for Dielectric Constant	
Intermediate Purity Selenium	726	Lead 206 Spike Assay and Isotopic	991
Intermediate-Purity Zinc	728	Solution Standard	1025
Iodine-123 Radioactivity Standard	4414L-C	Leaded-Tin Bronze Alloy	1035
Iodine-125 Radiactivity Standard	4407L-H	Light-Sensitive Paper	700d
Iodine-129 Radioactivity Standard	4949B	Light-Sensitive Paper	701d
Iodine-131 Radioactivity Standard	4401L-I	Light-Sensitive Plastic Chip	703
Iron Foil Mössbauer Standard	1541	Linear Polyethylene	1475
Iron-55 Low-Energy Photon Standard	4260C	Linear Polyethylene	1482
Iron Metal (Clinical Standard)	937	Linear Polyethylene	1483
Iron Ore (Labrador)	692	Linear Polyethylene	1484
Iron Ore (Nimba)	693	Linerboard, Standard for Tape Adhesion Testing	1810
		Liquid Absorbance Standard for Ultraviolet and Visible	931c
		Spectrophotometry Lithium Carbonate	924
		Lithium Ore	181
		Lithium Ore	182
		Lithium Ore	183
		Low-Alloy Steel, (AISI 4130)	1225
		Low Alloy Steel	1225
		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
		Zon-Miloy Sicol (119 60)	.200

Name	SRM	Name	SRM
Low-Alloy Steel Set (661-665)	S668	Naval Brass Standards for Optical	1108
Low-Carbon Silicon Steel	131c	Emission and Spectroscopic	
Low-Carbon Silicon Steel	1036	Analysis	
Low-Carbon Stainless Steel (AISI 316L)	166c	Naval Brass Standards for Optical Emission and Spectroscopic	C1108
Magnesium-base Alloy	171	Analysis	
Magnesium Cyclohexanebutyrate	1061c	Neutral Glass	716
Magnesium Gluconate Dihydrate	929	Neutron Density Monitor Wire	953
Magnetic Coating on Magnetic Substrate (Nickel on Steel)	1365a	Nickel-Chromium Cast Iron Nickel-Chromium-Molybdenum Cast Iron	
Magnetic Coating on Magnetic	1366a	Nickel-Chromium Steel	32E
Substrate (Nickel on Steel)		Nickel-Copper Alloy	882
Magnetic Coating on Non-Magnetic Substrate (Nickel and Chromium	1367a	Nickel Cyclohexanebutyrate Nickel Oxide, No. 1	1065b 671
on brass	(250	Nickel Oxide, No. 2	672
Magnetic Tape, High Density	6250	Nickel Oxide, No. 3	673
Manganese Fluoride, Magnetic Gram	766	Nickel-63 Radioactivity Standard	4226
Susceptibility	251	Nickel Silver (CDA 762)	879
Manganese Ore	25d	Nickel Siver (CDA 770)	880
Manganese-54 Point-Source	4997E	Nickel Spectrographic Steel Standard	409Ь
Radioactivity Standard	10.55	Nickel Sphere, Magnetic Moment	772
Manganese-54 Radioactivity Standard	4257	Nickel Steel	33d
Manganese Steel	100B	Ni-Cr-Mo-V Steel	1173
Manganous Cyclohexanebutyrate	1062Ь	Nicotinic Acid	148
Maraging Steel	1156	Niobium-94 Gamma-ray Standard	4201B
Metal on Quartz Filters for	2031	Nitric Oxide in Nitrogen	1683b
Spectrophotometry Metals on Filter Media	2676b	Nitric Oxide in Nitrogen	1684b 1685b
Methane in Air	1658a	Nitric Oxide in Nitrogen Nitric Oxide in Nitrogen	1686b
Methane in Air	1659a	Nitric Oxide in Nitrogen	1687b
Methane in Air	1660a	Nitric Oxide in Nitrogen (Mobile	2627
Medium Manganese Spectrographic	405a	Source Emission Gas Standard)	2027
Steel Standard	383a	Nitric Oxide in Nitrogen (Mobile	2628
Mercaptobenzothiazole	383a 743	Source Emission Gas Standard)	2/20
Mercury, Freezing Point Mercury-203 Radioactivity Standard	4418L	Nitric Oxide in Nitrogen (Mobile	2629
Mercury in Water, µg/mL	164lb	Source Emission Gas Standard) Nitric Oxide in Nitrogen (Mobile	2630
Mercury in Water, ng/mL	1642b	Source Emission Gas Standard)	2030
Microcopy Resolution Test Chart	1010a	Nitric Oxide in Nitrogen (Mobile	2631
Microprobe Standard - Cartridge Brass	478	Source Emission Gas Standard)	2031
Mineral Glasses for Microanalysis	470	Nitrogen Dioxide in Air (Stationary	2653
Molybdenum Concentrate	333	Source Emission Gas Standard)	2055
Molybdenum, Heat Capacity	781	Nitrogen Dioxide in Air (Stationary	2654
Molybdenum-99 Radioactivity	4412L-H	Source Emission Gas Standard)	2034
Standard	4412L-11	Nitrogen Dioxide in Air (Stationary	2655
Molybdenum-Tungsten-Chromium-	134A	Source Emission Gas Standard)	2033
Vanadium Steel	15474	Nitrogen Dioxide in Air (Stationary	2656
Naval Brass Standards for Optical	1106	Source Emission Gas Standard)	
Emission and Spectroscopic		Nitrogen Dioxide Permeation Device	1629a
Analysis		4-Nitrophenol	938
Naval Brass Standards for Optical Emission and Spectroscopic	C1106		
Analysis Naval Brass Standards for Optical Emission and Spectroscopic	1107		
Analysis Naval Brass Standards for Optical Emission and Spectroscopic	C1107		
Analysis			

Name	SRM	Name	SRM
Nodular Cast Iron	342a	Organics in Shale Oil	1580
Nominal One Micrometer Polystyrene	1690	Oxalic Acid	4990C
Spheres		Oxygen in Ferrous Materials	1090
Non-Fat Powdered Milk	1549	Ingot Iron	
Nonmagnetic Coating on Magnetic	1359	Oxygen in Ferrous Materials	1091
Substrate (Copper and Chromium		(Stainless Steel AISI 431) Oxygen in Ferrous Materials Vacuum	1002
on Steel)	1360	Melted Steel	1092
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium	1300	Oxygen in Maraging Steel	1094
on Steel)		Oxygen in Nitrogen (Gas Standard)	2657
Nonmagnetic Coating on Magnetic	1361b	Oxygen in Nitrogen (Gas Standard)	2658
Substrate (Copper and Chromium	13010	Oxygen in Nitrogen (Gas Standard)	2659
on Steel)		Oxygen in Titanium-Base Materials	355
Nonmagnetic Coating on Magnetic	1362a	Oxygen in Valve Steel	1093
Substrate (Copper and Chromium		Oyster Tissue	1566
on Steel)		Palladium, Magnetic Gram	765
Nonmagnetic Coating on Magnetic	1363a	Susceptibility	
Substrate (Copper and Chromium		Penetrant Test Block	1850
on Steel)		Peruvian Soil, Environmental	4355
Nonmagnetic Coating on Magnetic	1364a	Radioactivity	
Substrate (Copper and Chromium		Petroleum Crude Oil	1582
on Steel)		Phosphate Rock (Florida)	120b
NPL GM Alpha Alumina	8005	Phosphor Bronze (CDA 521) Phosphor Bronze (CDA 544)	871
NPL GM Alpha Alumina	8006	Phosphorized Copper, Cu VIII	872 C1251
NPL GM Alpha Alumina NPL GM Alpha Alumina	8007 8008	Phosphorized Copper, Cu IX	C1251 C1252
NPL GM Graphitized Carbon Black	8001	Phosphorized Copper, Cu X	C1252
NPL GM Graphitized Carbon Black	8002	Phosphorus-32 Radioactivity Standard	4406L-G
NPL GM Melting Point Set	8000	Photographic Step Tablet	1008
NPL GM Non-porous Silica	8003	Pine Needles	1575
NPL GM Non-porous Silica	8004	Plastic Clay	98a
N-tertiary-Butyl-2-benzothiazolesulfen-	384d	Platinum, Magnetic Gram	764
amide Rubber Compound		Susceptibility	
Obsidian Rock	278	Plutonium-238 Alpha-Particle Standard	4906B
Octaphenylcyclotetrasiloxane	1066a	Plutonium-240 Alpha-Particle Emission-	4338
Oil Furnace Black Rubber Compound	378ь	Rate Solution Standard	
Opal Glass Powder	91	Plutonium-239 Alpha-Particle Solution	4331
Optical Emission and X-ray	1102	Standard	
Spectroscopic Analysis		Plutonium-242 Alpha-Particle Solution	4334B
Optical Microscope Linewidth	474	Standard	
Measurement Standard Optical Microscope Linewidth	475	Plutonium Isotopic Standard	946
Measurement Standard	475	Plutonium Isotopic Standard Plutonium Isotopic Standard	947
Optical Microscope Linewidth	476	Plutonium Metal	948 949f
Measurement Standard	470	Plutonium Metal (Standard Matrix	945
The day of the standard		Material)	773
		Plutonium-244 Spike Assay and	996
		Isotopic Standard	,,,
		Polychlorinated Biphenyls in Oil	1581
		Polycrystalline Alumina Elasticity	718
		Standard	
		Polyester Plastic Film for Oxygen	1470
		Gas Transmission	
		Polyisobutylene Solution in Cetane	1490
		Polystyrene	1478
		Polystyrene	1479
		Polystyrene (Broad Molecular Weight)	706
		Polystyrene (Narrow Molecular	705
		Weight)	1.01
		Polystyrene Spheres Portland Cement (Black)	1691 1880
		- 5. mild Comont (Diack)	1000

Name	SRM	Name	SRM
Portland Cement (Blue)	635	Quartz on Filter Media	2679a
Portland Cement (Clear)	639	Quinine Sulfate Dihydrate	936
Portland Cement (Gold)	634	Radiogenic Lead Isotopic Standard	983
Portland Cement (Green)	638	Radium-226 Gamma-ray Standard	4956
Portland Cement (Pink)	637	Radium-226 Gamma-ray Standard	4957
Portland Cement (Red)	633	Radium-226 Gamma-ray Standard	4958
Portland Cement (White)	1881	Radium-226 Gamma-ray Standard	4959
Portland Cement (Yellow)	636	Radium-226 Gamma-ray Standard	4960
Portland Cement Fineness Standard	114n	Radium-226 Gamma-ray Standard	4961
Potassium Chloride	2202 918	Radium-226 Gamma-ray Standard	4962 4963
Potassium Chloride (Clinical Standard)	999	Radium-226 Gamma-ray Standard	4964B
Potassium Chloride (Primary Chemical)	999	Radium-226 Gamma-ray Standard Radium Standard (Blank Solution)	4952B
Potassium Chloride for Solution	1655	Radon-226 for Radon Analysis	4953C
Calorimetry	1055	Red Brass	1109
Potassium Dichromate	136d	Red Brass	C1109
Potassium Dihydrogen Phosphate	200	Red Brass	1110
Potassium Dihydrogen Phosphate	186Ic	Red Brass	C1110
Potassium Dihydrogen Phosphate	2186I	Red Brass	1111
Potassium Erucate	1076	Red Brass	C1111
Potassium Feldspar	607	Reduced Iron Oxide	691
Potassium Fluoride	2203	Reference Fuel Isooctane	1816a
Potassium Hydrogen Phthalate	185e	Reference Fuel n-Heptane	1815a
Potassium Hydrogen Tartrate	188	Reflection Step Tablet	2061
Potassium Iodide with Attenuator	2033	Refractive Index Glass	1820
Potassium Nitrate	193	Refractive Index Silicone Liquids	1823
Potassium Tetroxalate	189	Refractive Index, Soda-Lime Glass	1822
Powdered Lead Based Paint	1579 1647	Relative Stress-Optical Coefficient	708
Priority Pollutant Polynuclear Aromatic Hydrocarbons (in	1047	Glass Resulfurized-Rephosphorized Steel	C1221
Acetonitrile)		Rice Flour	1568
Propane in Air	1665b	River Sediment	1645
Propane in Air	1666b	River Sediment, Environmental	4350B
Propane in Air	1667b	Radioactivity	
Propane in Air	1668b	Rocky Flats Soil Number 1,	4353
Propane in Air	1669b	Environmental Radioactivity	
Propane in Nitrogen (Mobile Source	2643	Rubidium Melting Point	1969
Emission Gas Standard)		Rutile Ore	670
Propane in Nitrogen (Mobile Source	2644	Scanning Electron Microscope	484c
Emission Gas Standard)		Magnification Standard	
Propane in Nitrogen (Mobile Source	2645	Scanning Electron Microscope	2069
Emission Gas Standard)		Performance Standard	2210
Propane in Nitrogen (Mobile Source	2646	Secondary Standard Flexible Disk	3210
Emission Gas Standard)		Cartridge (Computer Amplitude	
Propane in Nitrogen (Mobile Source	2647	Reference)	3200
Emission Gas Standard)	2640	Secondary Standard Magnetic Tape Secondary Standard Magnetic Tape	1600
Propane in Nitrogen (Mobile Source Emission Gas Standard)	2648	Cassette Cassette	1000
Propane in Nitrogen (Mobile Source	2649	Secondary Standard Magnetic Tape	3216
Emission Gas Standard)	2047	Cartridge (Computer Amplitude	3210
Propane in Nitrogen (Mobile Source	2650	Reference)	
Emission Gas Standard)	2030	Second Surface Aluminum Mirror for	2023
Propane in Nitrogen and Oxygen	2651	Specular Reflectance	
(Mobile Source Emission Gas		•	
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	Name	SRM
Second Surface Aluminum Mirror for	2024	Soda-Lime Sheet Glass	1831
Specular Reflectance	2024	Soda-Lime Silica Glass	622
Second Surface Aluminum Mirror with	2025	Soda-Lime Silica Glass	710
Wedge for Specular Reflectance	2020	Soda-Lime Silica Glass for Liquidus	773
Selenium-Bearing Steel	1170b	Temperature	
Selenium-75 Radioactivity Standard	4409L-D	Sodium Bicarbonate	191a
Sheet Brass	37E	Sodium Bicarbonate	2191
Silica Brick	198	Sodium Carbonate	192a
Silica Brick	199	Sodium Carbonate	2192
Silicon-Aluminum Alloy	87a	Sodium Chloride	2201
Silicon Bronze	158A	Sodium Chloride (Clinical Standard)	919
Silicon Density Standard	1840	Sodium Cyclohexanebutyrate	1069ь
Silicon Density Standard	1841	Sodium Oxalate Reductometric	40h
Silicon Metal	57a	Standard	
Silicon Powder, Spacing Standard	640a	Sodium Pyruvate	910
for X-ray Diffraction		Sodium Tetraborate Decahydrate	187ь
Silicon Power Device Level	1522	(Borax)	
Resistivity Standard		Solder	127b
Silicon Resistivity Standard for Eddy	1523	Solder	1131
Current Testers		Special Nuclear Container DOT 6M,	9940
Silver 2-Ethylhexanoate	1077a	15 gal.	
Silver-Gold Thermocouple Wire	733	Special Nuclear Container, 55 gal.	9941
Silver, Vapor Pressure	748	Special Nuclear Container Type A.	9942
Sintered and Arc-Cast Tungsten,	1465	10 gal.	
Thermal Conductivity and		Special Nuclear Container, Type A, 55 gal.	9943
Electrical Resistivity	1466		9910
Sintered and Arc-Cast Tungsten, Thermal Conductivity and	1466	Special Nuclear Material Package Spectrographic Ingot Iron and	461
			401
Electrical Resistivity	1475	Low-Alloy Steel Standard (Rod)	462
Sintered and Arc-Cast Tungsten, Thermal Conductivity and	1467	Spectrographic Ingot Iron and	402
Electrical Resistivity		Low-Alloy Steel Standard (Rod)	463
Sintered and Arc-Cast Tungsten.	1468	Spectrographic Ingot Iron and	403
Thermal Conductivity and	1468	Low-Alloy Steel Standard (Rod)	464
Electrical Resistivity		Spectrographic Ingot Iron and	464
Smoke Density Chamber Standard	1007a	Low-Alloy Steel Standard (Rod) Spectrographic Ingot Iron and	465
(Flaming Exposure Condition)	1007a	Low-Alloy Steel Standard (Rod)	403
Smoke Density Chamber Standard	1006ь	Spectrographic Ingot Iron and	166
(Non-flaming Exposure Condition)	10000		466
Soda-Lime Container Glass	621	Low-Alloy Steel Standard (Rod)	467
Soda-Lime Flat Glass	620	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	407
Soda-Lime Float Glass	1830		468
Soda-Lime Glass	1826	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	408
Soda-Lime Glass Powder	92	Spectrographic Ingot Iron and	1166
Soda Dine Glass I Owder	72	Low-Alloy Steel Standard	1100
		Spectrographic Stainless Steel	442
		Standard Stainless Steel	442
		Spectrographic Stainless Steel Standard	443
			444
		Spectrographic Stainless Steel Standard	444
		Spectrographic Stainless Steel Standard (Disc)	D849
		Spectrographic Stainless Steel	D850
		Standard (Disc)	
		Spectrographic Stainless Steel Standard (Group II)	445

Name	SRM	Name	SRM
Spectrographic Stainless Steel	446	Spectroscopic Titanium-Base Standard	644
Standard (Group II)		Spectroscopic Titanium-Base Standard	645
Spectrographic Stainless Steel	447	Spectroscopic Titanium-Base Standard	646
Standard (Group II)		Spheroidized Iron Carbide in Ferrite	493
Spectrographic Stainless Steel	448	Spreading Resistance Calibration	2529
Standard (Group II)		(100) n-Type Silicon	
Spectrographic Stainless Steel	449	Spreading Resistance Calibration	2528
Standard (Group II)		(100) p-Type Silicon	
Spectrographic Stainless Steel	450	Spreading Resistance Calibration	2527
Standard (Group II)	.50	(111) n-Type Silicon	
Spectrographic Stainless Steel	849	Spreading Resistance Calibration	2526
Standard (Rod)	017	(111) p-Type Silicon	
Spectrographic Stainless Steel	850	Stabilized Wine	1590
Standard (Rod)	000	Stainless Steel	121d
Spectrographic Steel Standard (Disc)	D803a	Stainless Steel	123c
Spectrographic Steel Standard (Disc)	D807a	Stainless Steel	160b
Spectrographic Steel Standard (Rod)	803a		367
Spectrographic Steel Standard (Rod)	804a	Stainless Steel (AISI 446)	1267
	805a	Stainless Steel (AISI 446)	73c
Spectrographic Steel Standard (Rod)	807a	Stainless Steel, 13% Chromium	C1151
Spectrographic Steel Standard (Rod)	808a	Stainless Steel, Cr-Ni	1151a
Spectrographic Steel Standard (Rod)		Stainless Steel, Cr-Ni	C1152
Spectrographic Steel Standard (Rod)	809a 817b	Stainless Steel, Cr-Ni	1152a
Spectrographic Steel Standard (Rod)		Stainless Steel, Cr-Ni	C1153
Spectrographic Steel Standard (Rod)	820a	Stainless Steel, Cr-Ni	1153a
Spectrographic Steel Standard (Rod)	821	Stainless Steel, Cr-Ni	C1154
Spectrographic Steel Standard (Rod)	827	Stainless Steel, Cr-Ni	1154a
Spectrographic Tool Steel Standard	436	Stainless Steel, Cr-Ni	1155
Spectrographic Tool Steel Standard	437	Stainless Steel, Cr-Ni-Mo	1172
Spectrographic Tool Steel Standard	438	Stainless Steel, Cr-Ni-Nb	1171
Spectrographic Tool Steel Standard	439	Stainless Steel, Cr-Ni-Ti	1890
Spectrographic Tool Steel Standard	440	Stainless Steel for Pitting or Crevice	1090
Spectrographic Tool Steel Standard	441	Corrosion	738
Spectrographic Tool Steel Standard	837	Stainless Steel Thermal Expansion	736 372h
Spectrographic Tool Steel Standard	840	Stearic Acid Rubber Compound	368
Spectrographic Tool Steel Standard	D837	Steel (AISI 1211)	1169b
(Disc)		Steel (Lead-Bearing)	1070a
Spectrographic Tool Steel Standard	D840	Strontium Cyclohexanebutyrate	4403L-B
(Disc)		Strontium-85 Radioactivity Standard	4945D
Spectrographic Tool Steel Standard	D841	Strontium-89 Radioactivity Standard	386h
(Disc)		Styrene-butadiene Rubber (Type 1500)	
Spectrographic Zinc-Base Die-Casting	625	Succinonitrile Freezing Point	1970 17c
Alloy A		Sucrose	
Spectrographic Zinc-Base Die-Casting	626	Sulfate and Nitrate on Filter Media	2673
Alloy B		Sulfur Dioxide in Nitrogen	1661a
Spectrographic Zinc-Base Die-Casting	627	Sulfur Dioxide in Nitrogen	1662a
Alloy C		Sulfur Dioxide in Nitrogen	1663a
Spectrographic Zinc-Base Die-Casting	628	Sulfur Dioxide in Nitrogen	1664a
Alloy D		Sulfur Dioxide in Nitrogen	1693
Spectrographic Zinc-Base Die-Casting	629	Sulfur Dioxide in Nitrogen	1694
Alloy E			
Spectrographic Zinc-Base Die-Casting	630		
Alloy F	000		
Spectrographic Zinc Spelter Standard	631		
	641		
Spectroscopic Titanium-Base Standard	642		
Spectroscopic Titanium-Base Standard	643		
Spectroscopic Titanium-Base Standard	043		

			. 19. 11. 19.
Name	SRM	Name	SRM
Sulfur Dioxide in Nitrogen	1696	Titanium-Base Alloy (Unalloyed)	650
Sulfur Dioxide Permeation Tube	1627	Titanium-Base Alloy (Unalloyed)	651
(2 cm tube)		Titanium-Base Alloy (Unalloyed)	652
Sulfur Dioxide Permeation Tube	1626	Titanium Dioxide	154b
(5 cm tube)		Toluene	211c
Sulfur Dioxide Permeation Tube	1625	Tomato Leaves	1573 132b
(10 cm tube)	2602	Tool Steel (AISI M2) Tool Steel (AISI M2)	1157
Sulfur in Coal Sulfur in Coal	2682 2683	Tool Steel Abrasive Wear Standard	1857
Sulfur in Coal Sulfur in Coal	2684	Tracealloy (Nickel-Base	897
Sulfur in Coal	2685	High-Temperature Alloy)	
Sulfur in Residual Fuel Oil	1619	Tracealloy (Nickel-Base	898
Sulfur in Residual Fuel Oil	1620a	High-Temperature Alloy)	
Sulfur in Residual Fuel Oil	1621b	Tracealloy (Nickel-Base	899
Sulfur in Residual Fuel Oil	1622b	High-Temperature Alloy)	
Sulfur in Residual Fuel Oil	1623a	Trace Elements in a Glass Matrix	610
Sulfur in Residual Fuel Oil	1624a	Trace Elements in a Glass Matrix	611
Sulfur Rubber Compound	371g	Trace Elements in a Glass Matrix	612
Superconductive Thermometric Fixed	767a	Trace Elements in a Glass Matrix	613 614
Point Device	7.00	Trace Elements in a Glass Matrix Trace Elements in a Glass Matrix	615
Superconductive Thermometric Fixed	768	Trace Elements in a Glass Matrix	616
Point Device Surface Flammability Standard	1002c	Trace Elements in a Glass Matrix	617
Synthetic Sapphire	720	Trace Elements in Coal (Bituminous)	1632a
Technetium-99 Radioactivity Standard	4288	Trace Elements in Coal (Sub-	1635
Technetium-99m Radioactivity	4410H-I	bituminous	
Standard	111011-1	Trace Elements in Coal Fly Ash	1633a
Tetrachloroethylene in Nitrogen	1808	Trace Elements in Fuel Oil	1634a
Thallium-201 Radioactivity Standard	4404L-F	Trace Elements in Water	1643a
Thermal Resistance, Fibrous Glass	1451	Trace Mercury in Coal	1630
Batt		2,2,4-Trimethylpentane	217c
Thermal Resistance, Fibrous Glass	1450ь	Tripalmitin	1595
Board		Tris, Basimetric	723a
Thorium-228, Thallium-208 Gamma-ray	4206C	Tris, for Solution Calorimetry	724a
Point-Source Standard	# 4 PD	Tris(hydroxymethyl)aminomethane	922 923.
Tin-Base Bearing Metal Tin, Freezing Point	54D 741	Tris(hydroxymethyl)aminomethane hydrochloride	923.
Tin-113-Indium-113m Radioactivity	4402L-C	Tris(1-phenyl-1, 3-butanediono)	1078ь
Standard	7102L-C	Chromium (III)	10,00
Tin-121m Point-Source Gamma-ray	4264B	Tris(1-phenyl-1, 3-butanediono)	1079ь
Emission-Rate Standard	12012	Iron (III)	
Tin, Secondary Freezing Point	42g	Triphenyl Phosphate	1071ь
Standard		Tungsten Carbide	276a
Titanium Alloy	654a	Tungsten-Chromium-Vanadium Steel	50c
Titanium-Base Alloy	173b	Tungsten Concentrate	277
Titanium-Base Alloy	176	Tungsten, Heat Capacity	782
		Tungsten-20% Molyhdenum Alloy	480
		Electron Microprobe Standard	
		Tungsten Thermal Expansion	737
		Unalloyed Copper	1034
		Unalloyed Copper, Cu "O"	393 457
		Unalloyed Copper, Cu IV Unalloyed Copper, Cu XI	457 454
		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 VII (Chip)	400
		Unalloyed Copper, Cu I (Rod)	494

Name	SRM	Name	SRM
Unalloyed Copper, Cu II (Rod)	495	Wear-Metals in Lubricating Oil	1085
Unalloyed Copper, Cu III (Rod)	496	(300 ppm)	1005
Unalloyed Copper, Cu V (Rod)	498	Wheat Flour	1567
Unalloyed Copper, Cu VI (Rod)	499	White Cast Iron	338
Unalloyed Copper, Cu VII (Rod)	500	White Cast Iron (Disc)	1145
Unalloyed Titanium	354	White Cast Iron (Disc)	1146
Uranium Isotopic Standard (Nominally	U-0002	White Cast Iron (Disc)	1150
depleted to 0.02%)		White Ceramic Tile for Directional	2019b
Uranium Isotopic Standard	U-005a	Hemispherical Reflectance	20170
Uranium Isotopic Standard	U-010	White Ceramic Tile for Directional	2020
(Nominally 1% Enriched)		Hemispherical Reflectance	2020
Uranium Isotopic Standard	U-015	White Iron	3d
(Nominally 1.5% Enriched)		White Opan Glass Diffuse Spectral	2015
Uranium Isotopic Standard	U 020	Reflectance Standard for the	2013
Uranium Isotopic Standard	U-030a	Visible Spectrum	
Uranium Isotopic Standard	U-050	Xenon-127 Gaseous Radioactivity	4309G
(Nominally 5% Enriched)		Standard	4309G
Uranium Isotopic Standard	U-100		42057
(Nominally 10% Enriched)	0-100	Xenon-133 Gaseous Radioactivity Standard	4307I
Uranium Isotopic Standard	U-150		
(Nominally 15% Enriched)	0-130	Xenon-133 Gaseous Radioactivity	4415L-I
Uranium Isotopic Standard	U-200	Standard	
(Nominally 20% Enriched)	0-200	Xenon-133, Xenon-137, Krypton-85	4310B
Uranium Isotopic Standard	11 250	Mixed Gaseous Radioactivity	
	U-350	Standard	
(Nominally 35% Enriched) Uranium Isotopic Standard	11.500	X-ray Film Step Tablet	1001
	U-500	X-ray Powder Diffraction Intensity	674
(Nominally 50% Enriched)	*** 750	Standard	
Uranium Isotopic Standard	U-750	X-ray Powder Diffraction (Mica)	675
(Nominally 75% Enriched)	*** 000	Low 2 Theta	
Uranium Isotopic Standard	U-800	Ytterbium-169 Radioactivity Standard	4419L-B
(Nominally 80% Enriched)	** **	Zinc Base Alloy (Die Casting)	94c
Uranium Isotopic Standard	U-850	Zinc Concentrates	113a
(Nominally 85% Enriched)		Zinc Concentrates	329
Uranium Isotopie Standard	U-900	Zinc Cyclohexanebutyrate	1073ь
(Nominally 90% Enriched)		Zinc, Freezing Point	740
Uranium Isotopic Standard	U-930	Zinc, Freezing Point Standard	43h
(Nominally 93% Enriched)		Zinc Metal	683
Uranium Isotopic Standard	U-970	Zinc Oxide Rubber Compound	370e
(Nominally 97% Enriched)		Zircaloy-2	360a
Uranium Metal	960	Zircaloy-4 Metal	1237
Uranium Oxide	950ь	Zircaloy-4 Metal	1238
Uranium Oxide	969	Zircaloy-4 Metal	1239
Uranium-233 Spike Assay and	995	Zirconium-Barium Chromate	1651
Isotopic Solution Standard		Formulation for Heat-Source	
Uranium-235 Spike Assay and	993	Powder Calorimetry	
Isotopic Solution Standard		Zirconium-Barium Chromate	1652
Urban Dust/Organics	1649	Formulation for Heat-Source	1002
Urban Particulate Matter	1648	Powder Calorimetry	
Urea	912a	Zirconium-Barium Chromate	1653
Urea	2141	Formulation for Heat-Source	1033
Urea	2152	Powder Calorimetry	
Uric Acid	913	Zirconium Metal	1234
Vanadium and Nickel in Residual	1618	Zirconium Metal	1234
Fuel Oil	1010	Zirconium Metal Zirconium Metal	
Vanadium in Curde Oil	8505	Zirconium Metai	1236
Vanadium-49 Low-Energy Photon	4266		
Standard	4200		
Waspalov	349		
Wear-Metals in Lubricating Oil	349 1084		
	1084		
(100 ppm)			

Appendix II. Certificates for Coal, Ore, Mineral, Rock, and Refractory Standards (listed in numerical order).

U.S. Department of Commerce Juanita M. Kreps Secretary

National Bureau of Standards Certificate of Analysis

Standard Reference Material 1c Argillaceous Limestone

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 110°C)

		(All	analyse	s are ba	sea on s	ampies	aried 2 i	nours at	110°C)			
Constituent	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	P ₂ O ₅	MnO	CaO	SrO	MgO	Na ₂ O	K ₂ O	Loss on Ignition
Certified Value, % by wt.	6.84	0.55	1.30	0.07	0.04	0.025	50.3	0.030	0.42	0.02	0.28	39.9
Estimated ² Uncertainty	0.08	0.03	0.03	0.01	0.01	0.005	0.3	0.005	0.04	0.01	0.01	0.1
Method ³	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Photometric	Atomic Absorption		Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	
Α Α	6.82	ь _{0.53}	b 1.33	° 0.07	d 0.04	e 0.03	f 50.40	LLL	f 0.45	g 0.03	g 0.28	39.93
В	h _{6.77}	i .61	J _{1.31}	07.	d .03	e .02	k 50.19		1 .54		g .29	39.80
с	6.82 ^m 6.77 h _{6.80}	.55	1.27	c .06	d .05	.02 m .03	50.18 50.56 f 50.20	0.03	.38 m _{.43}	.02	m .28 m .29	39.82 39.85
D	h 6.92	i .55 .	1.30	.066	d .038	.022	50.18	.030	.45	g .02	9 .30	39.90
E	6.92	.57	1.29	m.066	d .039	.021	k 50.57	.031	.41	.028	.27	39.87
F	6.76	.57		.08	.04	.027	50.52	.03	.42	.02		39.97
G	P 6.91	.54	1.35	.07	d .04	.027	k 50.20	.03	.42	.02	.28	39.89
н						.022	9 _{50.58}	.034	.38	g .025	g .30	

- 1. The certified value listed for a constituent is the present best estimate of the "true" value based on results of the cooperative analytical program
- 2. The estimated uncertainty of the "true" value 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 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination.)
- 3. Detailed descriptions of many of the methods of analysis employed in the certification program for this SRM may be found in Part 13, Annual Book of ASTM Standards. They are also available as separate reprints, C25 and C114, from ASTM headquarters. ASTM Standard Technical Publication No. 395 also describes methods of analysis used in this certification work.

Washington, D.C. 20234 December 14, 1978

P Dehydration with HC1O4.

J. Paul Cali, Chief Office of Standard Reference Materials

(over)

aSilicomolybdate photometric method

^bFerron (8 hydroxy7-iodo-5-quinolinesulfonic acid) photometric method.

Tiron (disodium-1, 2 dihydroxybenzene-3, 5-disulfonate)

photometric method.

Molybdenum blue photometric method.

Peroxydisulfate photometric method.

EDTA titration

Flame emission spectrometry.

Dehydration with HC1.

SnCl₂ reduction-K₂Cr₂O₇ titration

By difference between total NH4OH group and oxides of

iron, phosphorus, and titanium.

k Calcium precipitated as oxalate and titrated with standard KMnO₄.

Magnesium determined gravimetrically as Mg₂P₂O₇.

"X-ray fluorescence spectrometry.

Atomic absorption spectrometry.

H₂O₂ photometric.

PLANNING, PREPARATION, TESTING, AND ANALYSIS:

The material for this SRM was provided by Lone Star Industries, Inc., Cement and Construction Materials Group, Houston, Texas, through the courtesy of C. W. Moore.

At NBS, the material was ground, sieved and thoroughly blended.

Chemical analyses for certification were performed in the following laboratories:

Atlantic Cement Co., Inc., Ravena, N.Y., F. J. Hogan and W. Twiss.

California Portland Cement Co., Colton, Calif., P. Hawkins and N. Norton.

Ideal Basic Industries, Cement Division, Ft. Collins, Colo., J. W. Yule.

Lone Star Industries, Inc., Cement and Construction Materials Group, Houston, Texas, C. W. Moore, L. S. Scheline, and I. Z. Somcio.

Martin Marietta Laboratories, Baltimore, Md., E. H. Scott.

National Bureau of Standards, Center for Analytical Chemistry, Washington, D.C., T. C. Rains, M. B. Blackburn, T. J. Brady, J. D. Messman, and T. A. Rush, and by R. K. Bell, Assistant Research Associate, ASTM-NBS Research Associate Program.

Portland Cement Association, Skokie, Ill., W. F. Mivelaz, R. F. Crow, E. LaBonde, A. G. Mateos, C. P. Palmiano, and H. Seiler.

Universal Atlas Cement, Division of United States Steel Corp., Gary, Ind., Z. T. Jugovic.

The overall direction and coordination of the technical measurements leading to certification were performed by J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

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.

National Bureau of Standards Certificate of Analysis

Standard Reference Material 27f

IRON ORE (Sibley)

(In Cooperation with the American Society for Testing and Materials)

This material is in the form of fine powder, for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

(Results based o	sample dried for	one hour at 105 °C.)
------------------	------------------	----------------------

	(Results based on sample direct for one hour at 105 °C.)										
Consti- tuent	Total Fe	SiO ₂	Al ₂ O ₃	P	S	TiO ₂	MnO	CaO	MgO	Na ₂ O	K ₂ O
Certified 1 Value% by wt.	65.97	4.17	0.82	0.041	0.005	0.019	0.011	0.039	0.019	0.012	• 0.008
Estimated ² Uncertainty	0.05	0.03	0.03	0.001	≤0.001	0.002	0.002	0.003	0.004	0.003	0.002
Method ³	SnCl ₂ K ₂ Cr ₂ O ₇	HClO ₄ Dehydration	Atomic Absorption	Photometric	Combustion- Titration	Photometric	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption
A	66.01	a4.12	0.78	b _{0.042}	0.005	c _{0.020}	0.009	0.042	0.02	d _{0.012}	d0.006
В	e 65.98	4.18	.83	.042	.006	f .018	9.014 9.012	9.041 9.042	g.013 g.015	d _{.012} 9.010	d .011 g .009
С	65.93	^h 4.18	.87 i.84	.039	i.005	k.019	010. ¹ .011	d _{.039}	.018	d.009	d.008
D	65.92	4.14	m.82	.041	.006	.02	.006	.036	.023	d.016	d.008
Е	ⁿ 65.96	4.17	.79	b .040	j.005	°.021	P .011	.035	.021	.010	.009
F	ⁿ 66.04	-	-	b .042	-	q.017	.011	-	.027	.015	.007
G	a _{65.96}	4.20	r 82 .81	.040	005. ز	f .018	.011 1.013	.035	.018	.009	.007

- 1. The certified value listed for a constituent is the present best estimate of the "true" value based on results of the cooperative analytical program for certification.
- 2. Estimated uncertainty includes method imprecision, bias among methods, and material variability for samples 0.5 g or
- 3. A detailed description of many of the methods of analysis employed in the certification program for this SRM may be found in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards.

NOTE: Laboratory C reported a value of 0.002 percent ZrO2 by the pyrocatechol photometric method.

- Sample fused in Na₂O₂.
- Alkali-molybdate method.
- X-ray spectrometric method.

 d Flame emission spectrometry
- H2S reduction-K2Cr2O7 titration.
- Chromotropic acid photometric.
- Spectrographic method.
- h Sample dissolved in HCl, silica removed by double dehydration with HCl. Ignited silica treated with H2SO4 and HF.

Aluminum separated by anion-exchange and determined by chelometric titration using 1,2 diaminocyclohexanetetraacetic acid and back titration with standard zinc

Washington, D.C. 20234 May 31, 1977

- Combustion-chromatographic
- k Titanium separated by anion-exchange and determined
- photometrically with diantipyrylmethane.
- Photometric
- ^mAluminum separated by anion-exchange and determined gravimetrically with phenylhydrazine.
- SnCl₂ reduction KMnO₄ titration.
- ^o H₂O₂ photometric. ^p Peroxydisulfate-arsenite.
- Atomic absorption spectrometry.
- Mercury cathode-NH₄OH-Cupferron-AlPO₄.

J. Paul Cali, Chief Office of Standard Reference Materials

(Over)

PLANNING, PREPARATION, TESTING, ANALYSIS: The iron ore material for this SRM was provided to NBS by the United States Steel Corporation, Pittsburgh, Pa., through the courtesy of R. H. Colin.

The "as received" material was crushed, dry ground, and sieved under contract with the Colorado School of Mines Research Institute, Golden, Colorado, under direction of M. G. Pattengill and H. O. VanMale. The final product passed a 150 mesh (105 µm) sieve, with about 50% passing a 200 mesh (74 µm) sieve.

At NBS the material was sieved and thoroughly blended. Homogeneity testing of selected samples representative of the final lot was performed at NBS by R. K. Bell, Assistant Research Associate, ASTM/NBS Research Associate Program. The observed range of values is as follows:

Constituent	Range, %	No. of determinations	Sample size, grams
Fe	± 0.05	16	0.5
SiO ₂	<± 0.02	4	1.0
P	<± 0.001	4	0.5

It is concluded that the material variability is within the method imprecision.

Chemical analyses for certification were performed in the following laboratories:

Alan Wood Steel Company, Conshohocken, Pa., V. J. Mercaldo.

Andrew S. McCreath and Son, Inc., Harrisburg, Pa., R. F. Lippi.

Booth, Garrett and Blair, Inc., Ambler, Pa., J. H. Ormsbee.

Ledoux and Company, Teaneck, N. J., S. Kallman.

National Bureau of Standards, Analytical Chemistry Division, Washington, D.C. by T. C. Rains and S. A. Wicks, and by R. K. Bell. ASTM Assistant Research Associate.

United States Steel Corporation, Research Laboratory, Monroeville, Pa., by J. D. Selvaggio, D. G. Cunningham, J. DiNardi, J. B. Ferons, A. V. Fioravanti, J. E. Friedline, J. R. Lucas, II, K. G. Mikos, C. W. Ponsonby, D. Shafferman, and R. J. Wargo.

Weirton Steel Division, Weirton, W. Va., R. L. Zickefoose.

The overall direction and coordination of the technical measurements leading to certification were performed jointly by R. E. Michaelis, Office of Standard Reference Materials, and by J. I. Shultz, Research Associate, ASTM/NBS Research Associate Program.

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.

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 69b

Bauxite (Arkansas)

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 140 °C) This material is in the form of fine powder ($<0.08\,\mathrm{mm}$) for use in checking chemical and instrumental methods of analyses.

Constituent	Certified Value ¹ Percent, by weight	Estimated Uncertainty ²
Al ₂ O ₃	48.8	0.2
Fe ₂ O ₃	7.14	.12
SiO ₂	13.43	.10
TiO ₂	1.90	.05
ZrO ₂	0.29	.07
P ₂ O ₅	.118	.004
V_2O_5	.028	.003
Cr ₂ O ₃	.011	.002
CaO	.13	.02
MgO	.085	.008
MnO	.110	.005
ZnO	.0035	.0005
K ₂ O	.068	.009
SO ₃	.63	.02
Loss on Ignition ³	27.2	.2

¹The certified value listed for a constituent is the present best estimate of the "true" value.

Washington, D.C. 20234 August 24, 1979 George A. Uriano, Chief Office of Standard Reference Materials

(over)

²The 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 variability for samples 1.0 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

³Determined by igniting to constant weight at 1050 °C.

ADDITIONAL INFORMATION ON THE COMPOSITION

Elements other than those certified may be present in this material as indicated below. These are not certified but are given as additional information on the composition.

Constituent	Concentration, Percent by weight	Constituent	Concentration, Percent by weight
BaO	(0.008)	Со	(0.0001)
Na ₂ O	(0.025)	Hf	(0.0063)
Ce	(0.024)	Sc	(0.0008)

The mineralogical composition of SRM 69b was determined by x-ray diffraction studies at the Geological Survey, U.S. Department of the Interior, Reston, Va., (J.W. Hosterman) to be 30% kaolinite, 60% gibbsite, and 10% siderite. These results are semiquantitative (to the nearest 5%).

PLANNING, PREPARATION, TESTING, ANALYSIS:

The mine run material for this SRM was provided by the Aluminum Company of America, Bauxite, Arkansas, through the courtesy of T.J. Forbes and by the Alcoa Technical Center, Pittsburgh, Pa., courtesy of H.B. Hartman. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute under a contract with the National Bureau of Standards.

Homogeneity testing was performed at NBS by J.S. Maples and T.E. Gills.

Cooperative analyses for certification were performed in the following laboratories:

Aluminum Company of America, Alcoa Center, Pa., R. C. Obbink.

Aluminum Company of Canada, Ltd., Arvida Research Center, Arvida, Quebec, Canada, L. Girolami.

Andrew S. McCreath & Son, Inc., Harrisburg, Pa., F. A. Pennington, Jr., R. F. Eakin, and S. L. Miller.

General Refractories Co., U.S. Refractories Division, Research Center, Baltimore, Md., S. Banerjee.

Geological Survey, U.S. Department of the Interior, Reston, Va., H. J. Rose, Jr., and J. W. Hosterman.

Kaiser Aluminum and Chemical Corp., Center for Technology, Pleasanton, Calif., H. J. Seim, A. E. McLaughlin, D. F. G. Marten, A. Kermaninejad, R. C. Kinne, J. R. Skarset, J. Boruk, and U. Vogel.

National Bureau of Standards, Washington, D.C., R. K. Bell, ASTM-NBS Assistant Research Associate.

National-Southwire Aluminum Co., Hawesville, Ky., N. Robinson and E. Gotzy.

Ormet Corp., Burnside, La., W. L. Brown and A. D. Lafleur.

Reynolds Aluminum Co., Alumina Research Division, Bauxite, Ark., J. B. Ezell, Jr.

University of Kentucky, Institute for Mining and Minerals Research, Center for Energy Research Laboratory, Lexington, Ky., T. V. Rebagay.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

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

NBS Standard Reference Materials BAUXITE SERIES September 4, 1979

R. E. Michaelis and R. Alvarez, NBS Office of Standard Reference Materials and

J. I. Shultz, ASTM Research Associate

The following table gives the values for four bauxite SRM's that are available in the form of fine powder (<0.08 mm) for use in chemical and instrumental methods of analysis. They are being issued as a culmination of a major Industry-ASTM-NBS cooperative program.

SRM No. Designation	69b Arkansas	696 Surinam	697 Dominican	698 Jamaican
Constituent		Percent b	y Weight	
Al ₂ O ₃	48.8	54.5	45.8	48.2
Fe ₂ O ₃	7.14	8.70	20.0	19.6
SiO ₂	13.43	3.79	6.81	0.69
TiO ₂	1.90	2.64	2.52	2.38
ZrO ₂	0.29	0.14	0.065	0.061
P ₂ O ₅	0.118	0.050	0.97	0.37
V_2O_5	0.028	0.072	0.063	0.064
Cr ₂ O ₃	0.011	0.047	0.100	0.080
CaO	0.13	0.018	0.71	0.62
MgO	0.085	0.012	0.18	0.058
MnO	0.110	0.004	0.41	0.38
ZnO	0.0035	0.0014	0.037	0.029
BaO	$(0.008)^{a}$	(0.004)	(0.015)	(0.008)
Na ₂ O	(0.025)	(0.007)	(0.036)	(0.015)
K ₂ O	0.068	0.009	0.062	0.010
SO ₃	0.63	0.21	0.13	0.22
Loss on Ign.	27.2	29.9	22.1	27.3
Се	(0.024)	(0.0041)	(0.069)	(0.030)
Co	(0.0001)	(0.00009)	(0.0013)	(0.0045)
Hf	(0.0063)	(0.0032)	(0.0014)	(0.0015)
Sc	(0.0008)	(0.0008)	(0.0058)	(0.0051)
Total	(100.0)	(100.1)	(100.1)	(100.1)

^aValues in parenthesis are not certified.

The value listed for a certified constituent is the present best estimate of the "true" value based on the results of the analytical program for certification (10-12 laboratories). The individual certificates of analysis list the "estimated uncertainties" associated with the certified values (also listed is a semiquantitative mineralogical composition (±5%) as determined by x-ray diffraction studies at the U.S. Geological Survey).

Inquiries regarding the Bauxite SRM's 69b, 696, 697, and 698, should be directed to the Office of Standard Reference Materials, Chemistry Building, B311. National Bureau of Standards, Washington, D.C. 20234. (301) 921-2045.

George A. Uriano, Chief Office of Standard Reference Materials U. S. Department of Commerce
Malcolm-Baldrige
Secretary
National Bursup of Standards
Ernest Ambler, Director

Certificate of Analysis

Standard Reference Material 70a

Feldspar

(All Analyses are Based on Samples Dried 2 hours at 105 °C)

	Percent
Silica (SiO:)	67.1
Alumina (A1:Oz)	17.9
Iron (as Fe ₂ O ₃)	0.07
Titania (TiO:)	.01
Calcium (as CaO)	.11
Barium (as BaO)	.02
Sodium (as Na ₂ O)	2.5
Potassium (as K ₂ O)	11.8
Rubidium (as Rb ₂ O)	0.06
Loss on Ignition	.40

Washington, D.C. 20234 August 10, 1981 (Revision of Certificate dated 3-26-65)

George A. Uriano, Chief Office of Standard Reference Materials

National Bureau of Standards Ernest Ambler, Acting Director

National Bureau of Standards Certificate of Analysis

Standard Reference Material 76a, 77a, and 78a **Burnt Refractories**

(In Cooperation With the American Society for Testing & Materials)

These materials are in the form of fine powder (<0.15 mm) and are intended for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

SRM No.	76a	77a	78a		
Constituent	Percent by Weight ^a				
SiO ₂	54.9	35.0	19.4		
Al ₂ O ₃	38.7	60.2	71.7		
Fe ₂ O ₃	1.60	1.00	1.2		
TiO ₂	2.03	2.66	3.22		
ZrO ₂	0.15	0.21	0.31		
MgO	.52	.38	.70		
CaO	.22	.05	.11		
K ₂ O	1.33	.090	1.22		
Na ₂ O	0.07	.037	.078		
P_2O_5	.120	.092	1.3		
Li ₂ O	.042	.025	0.12		
SrO	.037	.009	.25		
Loss on ignition	(.34) ^b	(.22)	(.42)		
Total	(100.0)	(100.0)	(100.0)		

CERTIFICATION: The value listed for a certified constituent is the present best estimate of the "true" value based on the results of the analytical program. The value is not expected to deviate from the "true" value by more than ± 1 in the last significant figure reported. For a subscript figure, the deviation is not expected to be more than \pm 5. Based on the results of the homogeneity testing, maximum variations within and among samples are estimated to be less than the uncertainty figures given above.

Washington, D.C. 20234

April 5, 1977

(Revision of certificate dated 6-23-76.

Only change: SrO values from uncertified to certified.)

J. Paul Cali, Chief Office of Standard Reference Materials

(Over)

Based on samples dried at 105 °C for one hour.
 A figure in parenthesis is not certified but is given for additional information on the composition.

PLANNING, PREPARATION, TESTING, AND ANALYSIS: These replacements for the original Burnt Refractory SRM's were especially prepared and provided to NBS by Harbison-Walker Refractories Company, Garber Research Center, Pittsburgh, Pa., through the efforts of Dr. R. K. Scott.

Carefully selected raw materials were batched to form extruded dobies. The dobies were air dried, oven dried at 120 °C, and then fired at 1427 °C for ten hours in kilns. The fired dobies were crushed and ground and converted to the fine powder product by air classification (about 95 %-325 mesh). The final products were mixed in a blender. At NBS, each of the materials was reblended and resieved.

Homogeneity testing by chemical analyses was performed at NBS by K. M. Sappenfield on selected samples representative of each lot of material for the key constituents, SiO₂ and Fe₂O₃.

Standard Deviation of a Single Determination (in wt. %), n = 7

Constituent	<u>76a</u>	<u>77a</u>	_78a
SiO ₂	0.09	0.06	0.08
Fe ₂ O ₃	.037	.034	03.

Cooperative analyses for certification were performed in the analytical laboratories at Harbison-Walker Refractories Company, Pittsburgh, Pa., R. K. Scott and J. Ryan; and at Pennsylvania State University, College of Earth and Mineral Sciences, University Park, Pa., N. H. Suhr, J. C. Devine, and J. B. Bodkin.

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell, O. Menis, T. C. Rains, T. A. Rush, K. M. Sappenfield, M. A. Waguespack, and S. A. Wicks.

The overall responsibility for the technical measurements at NBS was under the direction of W. R. Shields, I. L. Barnes, and O. Menis. The final coordination of the technical measurements was under the direction of J. 1. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of these SRM's were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

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 79a

Fluorspar

This Standard Reference Material is an acid-grade fluorspar concentrate, to be used primarily for the assay of imported fluorspar for industrial applications. The assay value certified is dependent on the U.S. Customs Laboratory Method given in this Certificate; however, two options exist for the determination of soluble fluoride calculated as calcium fluoride in the acetic acid leach solution. Either the spectrophotometric procedure (Part B), which was used for the original certification of this material, or the ion electrode procedure (Part C), which is included on this Certificate, may be used. The results of the two procedures show excellent agreement.

 $\begin{array}{c} \underline{\text{Constituent}} & \underline{\text{Percent by weight}} \\ \underline{\text{CaF}_2} & 97.39^{\text{a}} \pm 0.06^{\text{b}} \end{array}$

The following values, obtained by quantitative spectrochemical analysis, are given for information only and are not certified: Fe, 0.05-0.1%; Al and Sr, 0.01-0.1%; Mg, 0.01-0.05%; Na, 0.001-0.01%; Ba, 0.001-0.005%; K, <0.005%; and Cu, Li, Mn, Pb, and Ti, <0.001%.

The chemically determined value for SiO₂ is 0.67%.

The analytical work leading to certification was performed in the Division of Technical Services, U.S. Customs Laboratories, and by J. R. Moody and K. M. Sappenfield of the NBS Center for Analytical Chemistry. The spectrochemical determinations were made by M. Darr of the NBS Center for Analytical Chemistry.

A comparison of the ion electrode procedure with the spectrophotometric procedure was performed by R. L. Zimmerman, Jr., and H. G. Bertrand of the U.S. Customs Laboratory, New Orleans, La., and by J. R. Moody of the NBS Center for Analytical Chemistry.

This material was supplied by the American Smelting and Refining Company of El Paso, Texas; it was ground to pass a 177 μ m (80 mesh) screen.

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 J. L. Hague, R. E. Michaelis, and C. L. Stanley.

Washington, D.C. 20234 January 8, 1980 (Revision of Certificate dated 12-6-71)

George A. Uriano, Chief Office of Standard Reference Materials

(over)

^a Mean value based on 32 determinations using the U.S. Customs Laboratory Method of analysis (attached). The determinations were made by two analysts at each of three Customs laboratories and one NBS laboratory.

^bStandard deviation of a single determination

Method for the Determination of CaF2 in Fluorspar

A. Calcium Fluoride Determination: Transfer a 0.5 ± 0.01 g sample, previously dried at 100 to 105 °C, to a 60 mL Pt dish. Add 15 mL acetic acid (1:9) and approximately 50 mg of dry, ashless filter pulp. Digest on a steam bath for 30 minutes and stir with a small glass rod at 5-minute intervals. Stir and filter through an 11-cm close-texture filter paper (S&S No. 589 Red Ribbon, or equal) to which has been added approximately 4 mL filter pulp slurry (1 g ash-free pulp in 100 mL of water). Thoroughly wash crucible, stir rod, and paper with small portions of hot water (approximately 35 mL). The filtrate and washings should be collected in a polyethylene bottle for the determination of dissolved fluoride by either the spectrophotometric method described in Part B or the ion-selective electrode method described in Part C. Transfer paper and residue to original crucible, wipe the funnel with a small piece of filter paper, and add the paper to the crucible to assure recovery of fine particles. Dry crucible and contents, ignite to a dull red heat (600 °C), and cool to room temperature.

Transfer the residue, as completely as possible, from the crucible to a 400-mL beaker by gently tapping the crucible. Add 5 to 10 mL HC1 to the crucible and warm on a steam bath or a temperature-regulated sand bath. Transfer the HC1 to the beaker containing the residue. Police and wash the crucible with a minimum amount of not water. Repeat adding HC1 and washing 2 or 3 times using a total of 25 mL HC1. Add approximately 0.5 g of crystalline boric acid to beaker, cover with a watch glass, and digest on the steam bath for 15 minutes. Remove the beaker and cool to room temperature. Add 0.5 mL HNO3 and slowly evaporate on the steam bath to approximately 10 mL. During evaporation, the cover glass should be gradually removed from the beaker. Wash down the sides of the beaker with a fine stream of water and adjust the volume to approximately 75 mL. Heat to 70 to 80 °C, remove from heat, and add 100 mL of precipitating solution [3.8 g ammonium oxalate, 1.1 g oxalic acid, and 0.05 g disodium ethylenediaminetetracetate (EDTA) in 100 mL of water]. If a white precipitate forms at this point, add HC1 dropwise until it dissolves.

Heat to hoiling and slowly add NH₄OH (1:1) until a heavy white precipitate forms. Add 1 mL of bromocresol green solution (0.1 g soluble salt in 100 mL of water) and continue the addition until the color of the solution changes from yellowish-orange to grayish-green. Digest for 30 minutes on the steam bath, let stand for 60 minutes at room temperature, and filter by decantation through a prepared Gooch crucible (size 3, prepared with 2.1 cm glass-fiber filter paper, H. Reeve Angel No. X-934AH, or equivalent). Wash 3 or 4 times with a cold wash solution (0.2 g ammonium oxalate and 0.1 g oxalic add in 100 mL of water) and finally with 3 or 4 10-mL portions of cold water. (The washed precipitate should be free of chloride, ammonium oxalate, or any other contaminant that might reduce the K MnO₄ solution.) Transfer the contents of the crucible to a 400-mL beaker, add 250 mL $\rm H_2SO_4$ (1:19), and adjust the solution temperature to $\rm 27\pm3$ °C. While stirring, add approximately 90 percent of the 0.15 N K MnO₄ solution to be used for titration. This should be added at a rate of approximately 30 mL per minute from a buret. Allow to stand until the pink color disappears, e.g., approximately 45 seconds. (If pink color persists, too much K MnO₄ solution has been added and the determination must be repeated.) Heat to $\rm 57\pm3$ °C and complete the titration by slowly adding the last 0.5 to 1 mL K MnO₄ solution dropwise and allowing each drop to decolorize completely before adding the next drop. The end point is a pink color persisting for 30 seconds or more. See calculations.

- B. <u>Determination of Fluoride in Acetic Acid Leach Solution</u>: Remove all ions from the solution that might cause interference in the spectrophotometric determination of dissolved fluoride, by using either the ion-exchange method, or the fluorine-distillation method described in 2 and 3. Prepare a reagent blank, by substituting 15 mL of acetic acid (1:9), diluted to approximately 55 mL with distilled water and analyze simultaneously with sample.
- 1. Preparation of calibration curve: Prepare a dilute fluoride standard (1 mL = 0.1 mg CaF₂) from a standard solution (1 mL = 1 mg CaF₂) of 1.0755 g NaF diluted to one liter in a polypropylene volumetric flask with distilled water. Take a series of aliquots of 1, 2, 4, 6, 8, 11, 16 mL, etc., from the diluted fluoride standard and transfer them to 60-mL polyethylene bottles. Add 15 mL of acetic acid (1:9) and sufficient distilled water to bring the final volume to approximately 55 mL. Proceed to the ion-exchange or fluorine-distillation method.

2. <u>Ion-exchange method:</u> Prepare an anion-exchange column by pouring a slurry of distilled water and Rexyn 201 (OH) resin, or equivalent, sufficient to give a total exchange capacity of 27 to 30 meq, into a 25 mL polystyrene buret plugged with washed absorbent cotton (not glass wool) and drain off the excess water without allowing the water level to fall below the top of the resin column at any time. Continue this procedure until the resin is within about 5 mL of the top of the buret. Wash the resin with 20mL N NaOH, and finally with distilled water (approximately 100 mL), until the effluent is no longer alkaline. Adjust the stopcock to give a flow rate of approximately 100 drops per minute. The flow rate is an indicator of the state of the column. Backwash with distilled water whenever the flow rate falls below 100 drops per minute. Resin must be covered with liquid at all times.

Prepare a cation-exchange column in the same manner as the anion exchange column using a slurry of distilled water and Rexyn 101(H) resin, or equivalent, sufficient to give a total exchange capacity of 40 to 45 meq. Wash the resin with 50 mL of 1N HCl, and finally with distilled water (approximately 200 mL) until the effluent is free of chloride ion. Flow rate should be approximately 100 drops per minute.

Each of the diluted aliquots, as well as the blank, is run separately as follows: After completing both columns, pass the entire volume from the polyethylene bottle through the anion-exchange column, always maintaining the liquid level slightly above the top of resin. Wash the polyethylene bottle with distilled water and pass washings through the column using a total of approximately 60 mL for each sample. Discard the effluent and washings. Recover the fluoride, acetate, and other anions from the column by passing 20 mL of 1N NaOH through the column at a flow rate of approximately 100 drops per minute. Collect the effluent in the original polyethylene bottle. Wash the column with distilled water and collect washings in the same bottle to capacity (60 mL). Pour the contents of this bottle, and water used to rinse it, into the cation column and collect the effluent in a 100-mL volumetric flask. Wash the column with distilled water and collect just short of the mark in the same flask. Bring the liquid to the mark with distilled water. Proceed to the spectrophotometric determination.

- 3. <u>Distillation method</u>: Have water in a steam generator actively boiling, but do not connect to the fluoride distillation apparatus (Willard and Winter, or equivalent) at this time. Distill each of the diluted aliquots as well as the blank, separately as follows: Transfer the entire volume from the polyethylene bottle to the 500-ml. Claisen flask of the distillation apparatus using a minimum quantity of water for rinsing. Add 50 mL H₂SO₄ (1:1) and a few glass beads. Insert a stopper carrying the steam tube and thermometer, and heat the flask with a Bunsen burner or an electric heater. Connect the condenser at once and place a 200-ml. polypropylene volumetric flask at the receiving end. Water soon begins to distill over and the temperature of the liquid rises as the H₂SO₄ concentration increases. When the temperature reaches 120 °C connect the flask to the steam generator by means of the rubber tube. The rate of steam generation and the rate at which the Claisen flask is being heated should be regulated so that the temperature of the liquid in the distilling flask is maintained at 135 to 145 °C. Distill until the total volume in the receiver is approximately 185 mL (Note 1). Disconnect the condenser and rinse with a small portion of water, catching the washings in the flask containing the distillate, and fill to mark with distilled water. Proceed to the spectrophtometric determination.
- 4. Spectrophotometric determination: Pipette about one-tenth of the volume of the solution from steps B-2 or B-3 containing approximately 0.1 to 0.2 mg CaF_2 (not more than 50 mL) into a 100-mL polypropylene volumetric flask and add 15 mL 0.1 N sodium acetate solution and 25 mL methyl cellosolve. Bring the solution level in the flask almost to mark with distilled water and allow to cool to room temperature (mixing reaction is slightly exothermic) before making a final volume adjustment. (Resulting solution should have a pH of 3 to 5; optimum color development occurs in this range.)

Pour the entire contents of the volumetric flask into a 125-mL stoppered erlenmeyer flask containing 0.1 g of thorium chloranilate (Note 2). Stopper the flask and place on a shaking apparatus. Shake for 55 minutes, remove, and allow the contents of the flask to settle for 5 minutes. Filter through a close-texture filter paper, discarding the first 5 to 10 mL of filtrate.

Read the absorbance of sample against the reagent blank using 1 cm absorption cells in a Beckman DU spectrophotometer, or its equivalent, at a wavelength of 330 nm and a slit width of about 0.2 mm. Plot absorbance vs mg of CaF₂. This should yield a straight line passing through the origin.

- 5. Analysis of acetic acid leach solution: Carry the entire filtrate from the acetic acid digestion through steps B-2 or B-3 and finally B-4. Determine mg of CaF₂ by using the calibration curve and proceed with calculation.
- C. <u>Determination of Fluoride in Acetic Acid Leach Solution with an Ion-Selective Electrode.</u>

1. Apparatus, Reagents, and Solutions

- a. Special Apparatus
 - 1) Fluoride specific ion electrode
 - 2) Single junction reference electrode
 - 3) Expanded scale pH meter with millivolt capability

b. Reagents

ACS Reagent grades of sodium fluoride, glacial acetic acid, sodium chloride, and sodium hydroxide, must be used. For (1,2 Cyclohexylenedinitrilo)- tetraacetic acid (CDTA), a practical grade available from a commercial source may be used.

c. Solutions

1) TISAB II - (Total Ionic Strength Adjustment Buffer)

To 500 mL of distilled water in an 800 mL beaker, add 57 mL of glacial acetic acid, 58 g of sodium chloride, and 4 g of CDTA. Place the beaker in a cold water bath, on a magnetic stirrer. Stir the mixture, while adding concentrated sodium hydroxide, to provide a pH between 5.0 and 5.5. Transfer the contents of the beaker to a 1-liter flask, and dilute to the mark with distilled water. (TISAB II is used to provide a constant ionic strength background, decomplex the fluoride ions, and adjust and buffer the pH of the solution.)

2) Standard:

- a) Stock Solution (1900 ppmF) Dissolve 4.199 g of sodium fluoride in a 1-liter polypropylene volumetric flask and dilute to volume with distilled water.
- b) Working stock solution (19.00 ppmF.) Transfer 10 mL of stock solution (a) to a 1-liter polyproplene volumetric flask and dilute to the mark with distilled water.
- c) Working standards Prepare standards according to Table 1.

TABLE I

F Conc.	Working Stock	TISAB II	Distilled Water
(μg/mL)	(mL)	(mL)	(mL)
9.50	50.0	50	
4.75	25.0	50	25
2.85	15.0	50	35
1.90	10.0	50	40
0.95	5.00	50	45
0.38	2.00	50	48

If prepared in a 100 mL flask, dilute to mark with distilled water.

Store working standards in polyethylene bottles and refrigerate until use.

2. Procedure

Transfer the contents of the polyethylene bottles containing the acetic acid wash (Section A) to 200 mL polyethylene volumetric flasks and dilute to volume with distilled water. Transfer a 25 mL aliquot to a 50 mL polyethylene volumetric flask and dilute to volume with TISAB II. Place in refrigerator until ready for use. In a polyethylene bottle, prepare a reagent blank consisting of 15 mL of acetic acid (1:9) and approximately 55 mL of distilled water.

Transfer the working standards, test solutions, and the reagent blank to 150 mL polyethylene beakers and allow to come to room temperature. Stir all solutions with magnetic stirrers. Adjust the pH meter to –150 mV by use of the 19.00 ppm F working stock solution. Record the values of the solutions beginning with the least concentrated working standard. NOTE: For solutions containing low concentrations of fluoride, the pH meter may require approximately two minutes to stablize (cessation of drift). Rinse and dry the electrodes with tissue paper between readings. Record the values of the test solutions and the reagent blank immediately following the working standards.

Plot a curve from the values obtained for the working standards, and determine the concentration of the test solutions from the curve. Proceed with calculations.

D. Calculations:

First, calculate the % CaF_2 found in Part A. Then calculate the % CaF_2 found in either Part B (spectrophotometric procedure) or Part C (electrode procedure). Add the value calculated for either Part B or C to that calculated for Part A, to determine the total % CaF_2 in the sample.

For Part A:
$$\% \text{ CaF}_2 = \frac{3.904 \text{ V}_1 \text{N}}{\text{W}_5}$$

where: V₁ = Volume of KMnO₄ solution used (corrected for blank)

N = Normality of KMnO₄ solution

W_s = gram weight of sample

For Part B:
$$\% \text{ CaF}_2 = 0.1 \text{ W}_c \text{F}$$
W.

where: $W_c = milligram$ weight of CaF_2 in aliquot

F = aliquot factor; Total volume from B-2 or B-3

volume used

We = gram weight of sample

$$\frac{\text{For Part } C}{\text{W}_s} \approx \frac{\text{CV}_2 \text{AG}}{\text{W}_s} \times 10^{-4}$$

where: C = observed concentration in ppm $F^-(\mu g F^-/mL)$

 $V_2 =$ volume of test solution

A = aliquot factor;

total volume volume used

G = gravimetric factor;

CaF₂ 2F

W_s = gram weight of sample

For the conditions described in Part C, this formula may be simplified as follows:

- 1) The sample is diluted to 200 mL and a 25 mL aliquot is taken; thus the aliquot factor, A, is: $A = \frac{200}{25} = 8.00$
- 2) The volume of test solution V_2 is always 50 mL; $V_2 = 50.0$ mL
- 3) The gravimetric factor, G, is: $G = \frac{CaF_2}{2F} = \frac{78.0768}{37.9968} = 2.055$

by substitution:

$$\% \text{ CaF}_2 = \frac{\text{C x 50.0 x 8.00 x 2.055}}{\text{W}_{\circ}} \text{ x } 10^{-4} \text{ or}$$

$$\% CaF_2 = \frac{0.0822C}{W_s}$$

Note I. The volume of distillate necessary for complete recovery of fluoride will vary with the capacity of the distillation apparatus. Calibrate the apparatus by distilling a known quantity of fluoride to various final volumes to determine the volume necessary for complete recovery.

Note 2. Chloranilates are supplied as dry powders of high stability. However, long storage may reduce activity. Drying at 105 °C for at least 4 hours restores activity. Keep bottles tightly capped to prevent moisture absorption, which reduces activity and hinders the color development.

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

National Bureau of Standards Certificate of Analysis Standard Reference Material 81a

Glass Sand

(In Cooperation with the American Society for Testing and Materials)

This SRM is issued in the form of a ground powder (95% less than 106 μ m) blended to ensure homogeneity. It should be dried for 2 hours at 105 °C before use.

	Recommended Value		
Constituent	Percent by Weight	Range	<u>.s</u> .
Al ₂ 0 ₃	0.66	0.62 - 0.69	0.011
Fe ₂ 0 ₃	.082	.075089	.0024
TiO ₂	.12	.1014	.0064
Zr0 ₂	.034	.025042	.0026
Cr203	46 µg/g	33 - 58	3.9

Certification - The recommended value listed for each oxide is the best estimate of the true value based on the analytical data from both cooperators and NBS. The range of values listed is the tolerance interval, constructed such that it will cover at least 95% of the population with a probability of 0.99. It is computed as $X \pm Ks$: where is the standard deviation, K is a factor that depends on n (the number of samples measured), p, the proportion of the total sample covered (95%), and γ , the probability level (99%). In all cases none of the n values used exceeded the range specified. Thus, it includes variability between laboratories and between samples.

The overall direction and coordination of the round-robin analysis leading to certification were performed by Paul Close, Chairman of ASTM Subcommittee C-14.02 on Chemical Analysis of Glass and Glass Products.

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 January, 1978 J. Paul Cali, Chief Office of Standard Reference Materials

(over)

Chemical analyses for certification were performed in the following laboratories:

Anchor Hocking Corp., Lancaster, Ohio, R. E. Carr Brockway Glass Co., Inc., Brockway, Pa., E. I. McKinley. Corning Glass Works, Corning, N.Y., Y. S. Su. Ford Motor Co., Lincoln Park, Mich., T. O. LaFramboise. National Bureau of Standards, Analytical Chemistry Division, E. J. Maienthal, J. D. Messman and T. C. Rains.

Kimble Div. Owens-Illinois, Vineland, N. J., H. S. Moser. Owens-Illinois, Inc., Toledo, Ohio, P. Close. Penn State Univ., University Park, Pa., J. B. Bodkin.

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

Certificate of Analysis

Standard Reference Material 88a

Dolomitic Limestone

(All analyses are based on samples dried two hours at 110 $^{\circ}\text{C})$

	Percent
Silica (SiO ₂)	1.20
Alumina (Al ₂ O ₃)	0.19
Total Iron (as Fe ₂ O ₃)	.28
Titania (TiO ₂)	.02
Manganese (as MnO)	.03
Calcium (as CaO)	30.1 «
Strontium (as SrO)	0.01
Magnesium (as MgO)	21.3
Sodium (as Na ₂ O)	0.01
Potassium (as K ₂ O)	.12
Phosphorus (as P ₂ O ₅)	.01
Carbonate (as CO ₂)	46.6
Loss on ignition	46.7

Washington, D.C. 20234 July 22, 1982 (Revision of Certificate dated 1-31-67) George A. Uriano, Chief Office of Standard Reference Materials U. S. Department of Commerce Maurice H. Stans Secretary National Bureau of Standards L. M. Branscomb, Director

Certificate of Analysis

STANDARD REFERENCE MATERIAL 97a

Flint Clay

(Results based on sample dried for two hours at 140 °C)

Analyst	SiO ₂	Al ₂ O ₃	Fe₂O₃	TiO ₂	P2O5	K₂O	Na ₂ O	Li ₂ O	ZrO2	BaO	MgO	CaO	SrO	Cr ₂ O ₃	Loss on Igni- tion
1111	43.74	38.65		${1.88^{c} \choose 1.89^{d}}$	0.34	0.53	0.033*	0.12°	0.0631	0.078	0.16	0.118	0.17	0.028h	13.32
2[2]	43.68	38.95	.45	1.95	.35	.51°	.041°	.10€		.07	.148	.11s	.18s	.03	13.31
3	43.60	38.79	.432	1.87 ^d	.38i	.46e									
Average	43.67	38.79	0.45	1.90	0.36	0.50	0.037	0.11		0.075	0.15	0.11	0.18	0.03	13.32

References: [1] G.E.F. Lundell and J.I. Hoffman, NBS J. Res. 1, 91 (1928) RP5. [2] L. C. Peck, Geological Survey Bulletin 1170, (1964).

List of Analysts

- 1. R. K. Bell, B. B. Bendigo, T. C. Rains, T. A. Rush, E. R. Deardorff, J. R. Baldwin, R. A. Paulson, W. P. Schmidt, and S. D. Rasberry, Analytical Chemistry Division, Institute for Materials Research, National Bureau of Standards.
- 2. L. C. Peck, United States Geological Survey, Denver, Colorado.
- 3. L. M. Melnick, J. D. Selvaggio, and D. G. Cunningham, Applied Research Laboratory, United States Steel Corporation, Pittsburgh, Pennsylvania.

The material for the preparation of this standard was provided by the A. P. Green Fire Brick Company,

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanships of O. Menis and J. I. Shultz.

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 J. L. Hague.

Washington, D. C. 20234 October 8, 1969

J. Paul Cali, Acting Chief Office of Standard Reference Materials

^{*}o-phenanthroline photometric method.
*Iron reduced with SnCl₂ and titrated with standard potassium dichromate solution.
*Cupferron gravimetric method.
4H₂O₂ photometric method.

eFlame emission spectrometric method. Pyrocatechol violet photometric method. Atomic absorption method. *Atomic absorption method. *Diphenylearbazide photometric method. *Molybdenum-blue photometric method.

U. S. Department of Commerce Maurice H. Stans Secretary

Certificate of Analysis

STANDARD REFERENCE MATERIAL 98a

Plastic Clay

(Results based on sample dried for two hours at 140 °C)

Analyst	SiO ₂	Al ₂ O ₂	Fe ₂ O ₃	TiO ₂	P2O5	K₂O	Na ₂ O	Li ₂ O	ZrO2	BaO	MgO	CaO	SrO	Cr ₂ O ₃	Loss on Igni- tion
101	48.98	33.13	${1.34^{\circ} \choose 1.37^{\circ}}$	${1.56^{\circ} \choose 1.63^{d}}$	0.11	1.07°	0.080€	0.075	0.0421	0.031	0.42€	0.31*	0.041*	0.030h	12.40
2[2]	48.91	33.31	1.35	1.64	.10	1.08°	.083°	.064€		.03	.43€	.31*	.037	.04	12.49
3		33.12	1.28*	1.61 ^d	.111	0.98*									
Average	48.94	33.19	1.34	1.61	0.11	1.04	0.082	0.070		0.03	0.42	0.31	0.039	0.03	12.44

References: [1] G.E.F. Lundell and J.I. Hoffman, NBS J. Res. 1, 91 (1928) RP5. [2] L.C. Peck, Geological Survey Bulletin 1170, (1964).

*o-phenanthroline photometric method.

'Iron reduced with SnCl: and titrated with standard potassium dichromate solution.

"Cupferron gravimetric method.

4H₂O₂ photometric method.

«Flame emission spectrometric method. 'Pyrocatechol violet photometric method. «Atomic absorption method. «Diphenylcarbazide photometric method. 'Molybdenum-blue photometric method.

List of Analysts

- 1. R. K. Bell, B. B. Bendigo, T. C. Rains, T. A. Rush, E. R. Deardorff, J. R. Baldwin, R. A. Paulson, W. P. Schmidt, and S. D. Rasberry, Analytical Chemistry Division, Institute for Materials Research, National Bureau of Standards.
- 2. L. C. Peck, United States Geological Survey, Denver, Colorado.
- 3. L. M. Melnick, J. D. Selvaggio, and D. G. Cunningham, Applied Research Laboratory, United States Steel Corporation, Pittsburgh, Pennsylvania.

The material for the preparation of this standard was provided by the A. P. Green Fire Brick Company, Mexico, Missouri.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmenship of O. Menis and J. I. Shultz.

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 J. L. Hague.

Washington, D. C. 20234 October 8, 1969

J. Paul Cali, Acting Chief Office of Standard Reference Materials



Certificate of Analysis

Standard Reference Material 99a

Feldspar

(All Analyses are Based on Samples Dried 2 hours at 105 °C)

	Percent
Silica (SiO ₂)	65.2
Alumina (A12O2)	20.5
Iron (as Fe ₂ O ₂)	0.06
Titania (TiO2)	.007
Calcium (as CaO)	2.14
Barium (as BaO)	0.26
Magnesium (as MgO)	.02
Sodium (as Na ₂ O)	6.2
Potassium (as K ₂ O)	5.2
Phosphorus (as P ₂ O ₆)	0.02
Loss on Ignition	

Washington, D.C. 20234 August 10, 1981 (Revision of Certificate dated 3-26-65)

George A. Uriano, Chief Office of Standard Reference Materials

UNITED STATES DEPARTMENT OF COMMERCE WASHINGTON 25, D.C.

National Bureau of Standards Certificate of Analyses

Standard Sample No. 103 a Chrome Refractory

(All results are based on a sample dried for 2 hours at 105 to 110° C)

Analyst	Cr ₂ O ₃	Al ₂ O ₃	Total iron as FeO	MnO	MgO	CaO	SiO ₂	TiO ₂	ZrO ₂	P ₂ O ₅
1	a 32. 05	ь 29. 95	o 12. 43	d 0. 12	• 18. 57	10.70	4. 68	ь 0. 22	0.01	i 0. 007
2	± 31.95	1 30. 10 m 30. 01	n 12.43	. 12	18.50	. 69	• 4.68	. 21		.01
3	{ ° 32.07 } ° 32.09 }	30.02 29.95	t 12. 45	u. 11	18. 51	{ 7.68 }	¥ 4. 59	=. 22		y<.004
4	₽ 32.08	• 30.05		·. 10	18.62	ъ′. 60	* 4. 58	e'. 20		ď. 017
5	≈ 31.98		• 12. 43				4. 63	****		
6	₽ 32. 10	1' 29. 91	e' 12.40	h'. 09	• 18. 45	f. 72	o 4. 64	i'. 21	i. 01	i'. 01
7	32. 12	29.85		. 10	18.49	. 70		. 25		
8	p. r' 31. 78	k' 29. 80	1' 12.43	∞′. 10	a' 18. 63	ь. 74	· 4.60	o'. 23		{ °.009 a'.009 a'.009
Average	32.06	29.96	12. 43	0.11	18. 54	0. 69	4.63	0. 22	0.01	0.01

- l. solved in perchloric acid. Gravimetric phosphate. Corrected for Ca and Mo. cipitated with magnesium, separated as sulfate in precipitated with oxalate and weighed as CaO, chloric acid dehydration.

- reson, TiOs, and Psos, arated with cupferron, corrected for CrsOs and PsOs, the sodium peroxide fusion, filtered and dissolved ate. SnCl-KMnOs, liboric acid-sulfuric acid dehydration. used with excess ferrous iron and titrated with
- permanganate.

 9 Reduced with excess ferrous iron and titrated with dichromate. Corrected for vanadium.

 1 Separated with ammonia and mercury cathode and precipitated with ammonia. Corrected for TiO₃.

- ranadium.

 "SiO; removed, R₂O; precipitated with NH₂OH. Acid
 upferron separation, aluminum precipitated in filtrate
 with cupferron after neutralization with NH₂OH. Cor-
- with cuplers on after neutralization with NH₂OH. Corrected for Cr₂O₂.

 "Dissolved in perchloric and sulfuric acids, precipitated as sulfide, ignited, fused with potassium bisulfate, oxidized with permanganate and titrated with titanous chloride.

- construct solution as in footnote "m." Peroxide colorietric method.

 **Dissolved in nitrie, hydrochloric and perchloric acide,
 tered, volatilized arsenic as bromide, and precipitated
 hosphorus as phosphomolybdate. Determined the equiva"Third of the precipitate of the properties of the properties

List of Analysts

- Keith M. Sappenfield, National Bureau of Standards.
 Paul J. Byler, Booth, Garrett & Blair, Philadelphia, Pa.
 Andrew S. McCreath & Sons, Inc., Harrisburg, Pa.
 John H. Montague, E. G. Lavino and Co., Norristown,
- George Oplinger, K. A. Lane, and M. S. Budd, Solvay Process Division, Allied Chemical Corp., Syracuse, N.Y.
- B. C. Ruprecht and R. P. Lucas, Harbison-Walker Refractories Co., Pittsburgh, Pa.
- 7. L. J. Trostel, General Refractories Co., Baltimore, Md.
- 8. C. E. A. Shanahan, Richard Thomas & Baldwins, Ltd., Whitchurch, Aylesbury, Bucks, England.

A. V. ASTIN, Director.

Washington, D.C., September 28, 1962.

U.S. Department of Commerce Rogers C.B. Morton, Secretary

National Bureau of Standards Certificate of Analysis Standard Reference Materials 113a and 329

Zinc Concentrates

These Standard Reference Materials are in the form of fine powder (<.15 mm) and are intended for use both in checking chemical methods of analysis and in calibration with instrumental methods

CAUTION: The bottle should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.

SRM No. Element	113a 329 Percent by Weight ^a				
Zinc	57 _{.3}	45.5			
Lead	2.80	6.06			
Iron	2.08	12.94			
Calcium Oxide	1.19	80.0			
Magnesium Oxide	0.75	.165			
Cadmium	.78	.14			
Copper	.31	.132			
Cobalt	(.11)b	(.009)			
Nickel	(.07)	(.006)			
Sulfur (Total)	30.6	(31.7)			
Silicon Dioxide	(1.54)	(0.61)			
Indium	c	.019			
Silver	0.0467 d	.0089 ^d			

Based on samples dried at $105\,^{\circ}\mathrm{C}$ for one hour. Moisture loss: for SRM 113a 0.08%; for SRM 329 0.4 $_{5}$ %. Figures in parenthesis are not certified but are given for information only. Not determined. Troy ounces per ton is 13.6_{3} for SRM 13a and 2.6_{0} for SRM 329.

NOTE: The total of constituents reported for SRM 113a is 97.6% and that for SRM 329 is 98.0%. The remainder is expected to consist mainly of oxygen, carbon, and water of crystallization.

CERTIFICATION: The value listed for a certified element is the present best estimate of the "true" value based on the results of the analytical program. The value listed is not expected to deviate from the "true" value by more than ± 1 in the last significant figure reported. For a subscript figure, the deviation is not expected to be more than ± 5. Based on the results of homogeneity testing, maximum variations within and among samples are estimated to be less than the uncertainty figures given above.

Washington, D.C. 20234 December 29, 1975 (Revision of Certificate of May 6, 1974 for change in silver results)

J. Paul Cali, Chief Office of Standard Reference Materials

The material for these standards was supplied by Cominco American Inc., Spokane, Wash. The material for SRM 113a was prepared at the Magmont Mines, Bixby, Mo., and that for SRM 329 at the Sullivan Mine, Trail, B.C., Canada.

Following sieving and blending operations at NBS, homogeneity testing was performed by S. D. Rasberry and J. McKay, (x-ray fluorescence analyses); by E. J. Maienthal, (polarographic analyses); and by R. K. Bell, (chemical analyses).

Selected samples representative of the lot were analyzed and no significant variability was observed when using subsamples of 0.5 g or larger. (Moisture determinations usually were made on larger samples--up to 10 g.)

Cooperative analyses for certification were performed in the analytical laboratories of Cominco, Sullivan Mine, Trail, B.C., Canada, C. J. Mitchell; Cominco American Inc., Magmont Mines, Bixby, Mo., R. J. Gibson; and St. Joe Minerals Corp., Zinc Smelting Division, Monaca, Pa., J. J. Aldrich.

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell and E. J. Maienthal.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of O. Menis and J. I. Shultz.

The technical and support aspects involved in the preparation, certification, and issuance of these SRM's were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

U. S. Departing of Commerce Peter Greenson

National Bureau of Standards Certificate of Analysis

Standard Reference Material 120b

Phosphate Rock (Florida)

This standard is a finely powdered material intended for use in checking chemical methods of analysis and in calibration with optical emission and x-ray spectrometric methods of analysis.

See ADDENDUM* (Over) for Uranium (Radium and Thorium) (All results are based on samples dried for 1 hour at 105 °C.)

Percent by Weight

ANALYST*	P ₂ O ₅	CaO ·	SiO ₂	F	Soluble Fe ₂ O ₃	Soluble Al ₂ O ₃	Mg()	Na ₂ O	MnO	ı	(₂0	TiO ₂	CO2	CdO
1	34.51 ^a	49.42 ^b	4.70 ^c	3.82 ^d	1.10 ^e	1.09 ^f .g	0.29h	0.33 ^f	0.032 ⁱ	0.12 ^{f,j}		0.15 ^k		0.002l
2	34.51 ^m	49.35 ^m	4.73 ¹¹	3.79 ^m	1.10 ^h	1.07 ^h	.28 ^h	.36 ^h	.031 ^h	.12 ^j	0.09°		2.76P	.002 ^h
3	34.66 ⁿ	49.38 ^m	4.67 ^q	3.83	1.09 ^h	1.07 ^h	.30	.36 ^h	.032h	.12 ^j	.098º	.15	2.79	.002h
4	34.67 ^r	49.47 ^m	4.699	3.81 ^s	1.13 ^h	1.04 ^h	.28h	.35h	.032 ^h		.087°	.15 ^k	2.78P	.003 ^h
5	34.57	49.32 ^m	4.63 ^q	3.86	1.06 ^h	1.05 ^h	.25h	.34 ^h	-		.085°	-	2.83	-
6	34.48 ^m	49.45 ^m	-	3.92 ⁸	1.14 ^m	1.07 ^t	-	-	-			-	-	-
Average	34.57	49.40	4.68	3.84	1.10	1.06	0.28	0.35	0.032	0.12	0.090	0.15	2.79	0.002

^a Phosphorus precipitated with magnesia mixture, ignited an weighed as $Mg_2P_2O_7$.

Washington, D.C. 20234 July 31, 1972 ADDENDUM* (Over) July 31, 1979 J. Paul Cali, Chief Office of Standard Reference Materials

b Calcium precipitated as oxalate, ignited and weighed as CaO.

Sample fused with Na, CO₃, silica precipitated with ZnO and dehydrated with HCl. Traces of SiO₂ recovered by H₂SO₄ dehydration.

d Fluorine distilled into NaOH solution and precipitated as lead chlorofluoride. Chloride is precipitated with excess AgNO₃ and excess AgNO₃ is titrated with standard KCNS solution.

e SnCl₂ reduction - K₂Cr₂O₇ titration.

f Flame emission spectrometry with repetitive optical scanning.

g A value of 1.13 percent was obtained for total Al₂O₃ by graving

h Atomic absorption spectrometry.

i KIO, spectrophotometric method.

j Sample digested with mixed acids for I hour. Determination completed by atomic absorption spectrometry.

k H₂O₂ spectrophotometric method.

l Polarographic method.

^mVolumetric method.

n Gravimetric method.

O Sample digested with dilute HCl or aqua regia for 15 minutes. Determination completed by atomic absorption spectrometry.

^pCO₂ absorbed and weighed.

q Dehydration with HClO4 in presence of bonic acid.

r Molybdovanadophosphate spectrophotometric method.

⁸ Distillation – titration with standard thorium nitrate solution.

^t Aluminum precipitated with 8 hydroxyquinoline and weighed.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of O. Menis and J. I. Shultz.

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. E. Michaelis and C. L. Stanley.

PREPARATION, TESTING, AND ANALYSIS: The material for this standard was prepared by the American Cyanamid Company. Eighty five percent of the lot was made to pass 200 mesh sieve and some blending was done at the plant. Final sieving and blending operations were accomplished at NBS.

Homogeneity testing was performed by S. D. Rasberry, C. E. Fiori, and J. McKay with x-ray fluor-escence analysis. Calcium and phosphorus determinations were made on 14 samples representative of the top and the bottom of seven containers. The size of the samples taken for analysis was approximately 35 mg. The maximum variations in concentration among samples were within 0.09 percent for CaO and 0.12 percent for P_2O_5 .

The laboratories and analysts cooperating in the analytical program for certification were:

- R. K. Bell, E. R. Deardorff, E. J. Maienthal, T. C. Rains, T. A. Rush, and S. A. Wicks, Analytical Chemistry Division, Institute for Materials Research, National Bureau of Standards.
- 2. J. Padar, Agrico Chemical Co., Division of Continental Oil Company, Pierce, Florida.
- 3. D. B. Underhill, Borden Chemical Co., Plant City, Florida.
- 4. C. C. Thornton, Thornton Laboratories, Inc., Tampa, Florida.
- 5. W. W. Harwood, R. M. Lynch and H. N. Gomez, International Minerals and Chemical Corp., Bartow, Florida.
- 6. J. A. Sielski, American Cyanamid Co., Brewster Plant, Bradley, Florida.

*ADDENDUM

Uranium has been determined at NBS by thermal ionization mass spectrometry, E. L. Garner and L. A. Machlan, and the following certification is made:

Value, μg/g

Estimated Uncertainty^a

Uranium

128.4

± 0.5

^aThe estimated uncertainty is based on judgment and represents an evaluation of method imprecision and material variability.

(NOTE: On similar phosphate rock materials, a value of 127 μ g/g for uranium was reported in Ref. 1; additionally, values of 17 μ g/g for thorium and 43 pCi²²⁵Ra/g for radium also were reported.)

Ref. 1 Agr. Food Chem., 16, No. 2, 1968 (p232)

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

National Bureau of Standards Richard W. Roberts, Director

National Bureau of Standards Certificate of Analysis Standard Reference Material 154b Titanium Dioxide

This standard is in the form of fine powder, certified primarily for application in the paint and ceramic industries.

Constituent	Percent by Weight ^a	<u>Uncertainty</u> ^c
Titanium Dioxide (Ti02)	99.74 b	0.05

a Based on material dried at 110 °C for two hours.

 $^{^{\}mathrm{c}}$ The uncertainty figure represents the 95% confidence interval of the mean of all accepted values.

	NBS		
Method	Average	Standard Deviation ¹	Number of Determinations
Controlled-potential coulometric (0.2g samples)	99.73	0.05	9
Volumetric (0.35g samples)	99.71	0.03	10 ²
	Cooperators ³		
Volumetric (Analyst A) ⁴ Volumetric (ASTM D1394)	99.75 99.78	0.04 0.05	3

¹ Of single determinations.

Washington, D. C. 20234 May 16, 1973 J. Paul Cali, Chief Office of Standard Reference Materials

 $^{{\}bf b}$ The value given in this certificate is based on the following pertinent analytical data:

 $^{^2}$ Two discrepant results were omitted.

Results from one cooperating laboratory were deemed significantly high and have been omitted.

Results from Analyst B at the same laboratory were deemed significantly low and have been omitted.

PLANNING, PREPARATION, TESTING, and ANALYSIS: The material for this SRM has been carefully selected and prepared not only to reflect the present composition needs but also the anticipated future requirements.

A particular ilmenite ore was chosen so that, after beneficiation, the material would provide the desired high rutile to anatase ratio (rutile 97+%, anatase about 2%). Bleaching agents were not added; thus the material has the characteristically yellowish color of rutile. The lot was thoroughly blended in the laboratory and then screened through a $44\mu m$ (325 mesh) sieve. Preliminary testing on 6 samples, representative of the lot, showed no evidence of inhomogeneity. The planning, preparation and preliminary testing were under the supervision of John J. Libera, Research and Development Department, National Lead Industries, St. Louis, Missouri.

Cooperative analyses for certification were performed in the analytical laboratories of E. I. Du Pont De Nemours & Co., Pigments Department, Wilmington, Delaware, T. D. McKinley; National Lead Industries, Titanium Pigment Division, South Amboy, New Jersey, Benjamin S. Sanderson; and Sherwin-Williams Research Center, Chicago, Illinois, R. W. Scott.

Analyses were performed in the Analytical Chemistry Division of the National Bureau of Standards by J. R. Baldwin and G. Marinenko.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of O. Menis and J. I. Shultz.

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. E. Michaelis and C. L. Stanley.

ADDITIONAL INFORMATION ON THE COMPOSITION:

Certification is made only for the TiO₂ content. Investigations of this material at NBS and at cooperating laboratories provided some additional information that may be useful, but is not certified:

Constituent	Weight Percent
$P_{2}O_{5}$	(0.04)
SiO ₂	(0.01)
Fe_2O_3	(0.006)
Pb	(0.003)
Ca0	(~0.01)
. v	(~0.001
Cr	(~0.0005)
Cu	(~0.0005) (~0.0005)
MgO	(~0.01)
Moisture (110 °C-2 hours)	(0.02 to 0.05)
Loss on Ignition (900 °C for one hour under helium)	(0.06 to 0.07)

If in the use of this SRM, determinations are made for any of the uncertified minor and trace constituents, it would be appreciated if the results were forwarded to the Office of Standard Reference Materials. When sufficient information has been received the Certificate will be revised.

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 165a

Glass Sand

(In Cooperation with the American Society for Testing and Materials)

This SRM has been blended to ensure homogeneity. It should be dried for 2 hours at 105 °C before use.

Constituent	Percent by Weight	Range	<u>s</u>
Al_2O_3	0.059	0.051 - 0.066	0.0024
Fe ₂ O ₃	.012	.007017	.0018
TiO ₂	.011	.0065015	.0016
ZrO ₂	.006	.0005012	.002

Certification - The value listed for each oxide is the best estimate of the true value based on the analytical data from both cooperators and NBS. The range of values listed is the tolerance interval, constructed such that it will cover at least 95% of the population with a probability of 0.99. It is computed as X±Ks: where s is the standard deviation, K is a factor that depends on n (the number of samples measured), p, the proportion of the total samples covered (95%), and γ , the probability level (99%). In all cases none of the n values used exceeded the range specified. Thus, it includes variability between laboratories and samples.

The overall direction and coordination of the round-robin analysis leading to certification were performed by Paul Close, Chairman of ASTM Subcommittee C-14.02 on Chemical Analysis of Glass and Glass Products.

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 October 16, 1978 J. Paul Cali, Chief Office of Standard Reference Materials

Additional Information

A content of 1 μ g/g for Cr₂O₃ is not certified but rather is provided for information only.

Chemical analyses for certification were performed in the following laboratories:

Anchor Hocking Corp., Lancaster, Ohio, R. E. Carr.

Brockway Glass Co., Inc., Brockway, Pa., E. L. McKinley.
Corning Glass Works, Corning, N.Y., Y. S. Su.

Ford Motor Co., Lincoln Park, Mich., T. L. LaFramboise.

National Bureau of Standards, Analytical Chemistry Division, E. J. Maienthal, J. D. Messman and T. C. Rains.

Kimble Div. Owens-Illinois, Vineland, N.J., H. S. Moser. Owens-Illinois, Inc., Toledo, Ohio, P. Close.

Penn State Univ., University Park, Pa., J. B. Bodkin.

Certificate of Analysis

Standard Reference Material 180

High-Grade Fluorspar

This Standard Reference Material has a high calcium fluoride content and is issued primarily for the geological and geochemical scientific community. [NOTE: This material is not a substitute for the fluorspar standard, SRM 79, used primarily for the assay of fluorspar imported for industrial use 1

Constituent	Percent, by weight
CaF ₂	$98.80^{a} \pm 0.03^{b}$

^aThe value certified is based entirely on the method given in this certificate and is the mean of eight determinations. ^bThe standard deviation of a single determination.

Trace elements: These were determined by a general qualitative spectrochemical method and are given for information only. Fe, 0.1 – 1.0%; Al, Ba, Mg, Pb, Si, and Sr, 0.01 – 0.1%; Cu, K, Mn, Na, Ti, and V, 0.001 – 0.01%; and Ag and Li, less than 0.001%. Analyst E. K. Hubbard.

The analytical work leading to certification was performed by K. M. Sappenfield, Analytical Chemistry Division, National Bureau of Standards.

This material was supplied by Minera Frisco of San Francisco Del Oro, Chih., Mexico.

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 J. L. Hague, R. E. Michaelis, and C. L. Stanley.

Washington, D. C. 20234 March 31, 1971

J. Paul Cali, Chief Office of Standard Reference Materials

METHOD FOR THE DETERMINATION OF ${\rm CaF_2}$ IN FLUORSPAR

- *Transfer 0.50 g of fluorspar, previously dried at 100 to 105 °C, to a tared, ignited platinum crucible. Add 15 ml of acetic acid (1:9) containing 0.667 g of CaCO₃ per liter. Digest on a steam bath for 30 minutes and stir at five minute intervals. Add about 0.04 g of ashless filter pulp and stir for an additional minute. Filter through a double 12.5 cm extra dense filter paper (S & S No. 589 Red Ribbon) to which has been added about 0.04 g ashless filter pulp. Thoroughly wash the crucible, paper, and stirring rod with 5 ml portions of hot water (40 to 60 °C), using a total of about 35 ml. After washing, wipe the stirring rod with a small piece of wet filter paper and add the paper to the filter. Transfer the paper and residue to the crucible, dry in an oven at 80 °C, char slowly under an infrared lamp, and ignite in a muffle furnace at 600 °C.
- **Add 3 ml of HF to the residue in the crucible and evaporate to dryness. Add 1 to 2 ml of HClO₄ and evaporate to dryness under the hood. Cool, wash the inside of the crucible with 1 ml more of HClO₄ (to catch any undecomposed particles of calcium fluoride), and again evaporate to dryness. Cool the crucible and immerse in a beaker containing 150 ml of dilute HCl (5:95). Warm the beaker gently and remove the platinum crucible, being sure to remove adhering particles of Ca(ClO₄)₂.

Boil the contents of the beaker for 10 minutes. If any insoluble matter remains; filter, wash, and ignite it in platinum. Treat the ignited residue with a few drops of HF and HClO₄ and heat to expel the acids. Add 1 to 2 ml of HCl and digest on a steam bath. Transfer the contents to the main filtrate

Pass H₂S into the clear filtrate for several minutes and then make the solution ammoniacal. Continue with H₂S for 10 minutes and allow the precipitate to settle for 20 to 30 minutes. Filter and wash with NH₄Cl-(NH₄)₂S solution. (Prepare by passing a moderate stream of H₂S for 5 minutes through a solution containing 5 ml of NH₄OH and 10 g of NH₄Cl per liter.)

Neutralize the filtrate and washings with HCl, and add an additional 20 ml of HCL. Boil for 2 to 3 minutes to expel most of the $\rm H_2S$. Add KBr-Br₂ solution (20% solution of KBr saturated with Br₂) until the solution remains yellow. Boil until the finely divided sulfur has been oxidized and the bromine has been expelled. If the sulfur or sulfides are not removed by the bromine treatment, filter, wash the paper well and discard the residue. Dilute the filtrate to about 200 ml. Precipitate the calcium by adding 2 g of (NH₄)₂C₂O₄· $\rm H_2O$ and, while stirring, slowly add NH₄OH until the solution is slightly ammoniacal. Heat on a steam bath for one-half to one hour, stirring occasionally. Cool to room temperature. Filter on a close textured paper. Wash with a cold 0.1 percent solution of (NH₄)₂C₂O₄· $\rm H_2O$. Ignite to constant weight. Calculate the percent CaF₂ from the weight of the CaO.

References:

- *Removal of soluble calcium in acetic acid. Interlaboratory study of soluble calcium.
- **Method: Calcium in fluorspar
 The Analysis of Fluorspar by G. E. F. Lundell and J. I. Hoffman, J. Res. Nat. Bur. Stand. (U.S.), 2
 (1929) R.P. 51.

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

Certificate of Analysis

STANDARD REFERENCE MATERIALS

181, 182, and 183 Lithium Ores

These SRM's are intended for use in checking the accuracy of assay methods. They are certified for their constituent of economic interest. Additional data for information only appears on page 2. These SRM's are supplied in the form of fine powder.

	SRM 181	SRM 182	SRM 183
	(Spodumene)	(Petalite)	(Lepidolite)
	%		%
Li ₂ O	6.3_{9}	4.34	4.1_{2}

The value listed for Li_2O in the three SRM's is the best estimate of the "true" value. The deviation is not expected to be more than \pm 5 in the subscript number.

Washington, D.C. 20234 October 1, 1981 (Revision of Certificate dated 2/24/58 and Reprinted 8/20/70)

George A. Uriano, Chief Office of Standard Reference Materials

The following values are approximate, and are listed only for information.

Constituent	Wt. %	Wt. %	<u>Wt. %</u>
Na ₂ O	(0.8)	(0.4)	(0.2)
K ₂ O	(.3)	(1.)	(8.)
Rb ₂ O		(.03)	(3.5)
Cs ₂ O			(.3)

UNITED STATES DEPARTMENT OF COMMERCE WASHINGTON 25, D.C.

National Bureau of Standards Certificate of Analyses

Standard Sample 198 Silica Brick

(All results are based on samples dried at 105° to 110° C.)

Analyst	Al_2O_3	Total iron as Fe ₂ O ₃	TiO ₂	ZrO ₂	P2O5	MnO	CaO	MgO	NagO	K ₂ O	Li ₂ O	*Loss on ignition
1	{ b0.17 e.d,15	*0, 65 (.e. 67)	0.02	< 0, 01	b 0. 022	i 0. 006	2,72	0.07	i 0, 007	10.016	i 0. 001	0, 21
2	¥. 19	k. 64	¥.01		⊧. 012		¥2.74	k. 06	k. 009	k. 010	¥. 001	. 32
3	{	м. 67 ч. 65}	.01		•.011		2.75	.06	», 02	». 03		. 22
4	a. 17	1.69	.02		°. 026		2,73	.08	r, 008	k. 016	k <.007	. 15
5	۲. 17	- 66	. 01		٠. 025		2.72	. 07	ء, 005	. 012	i. 002	. 23
6		. 68	. 03		.026		2.72	. 07	i, 005		ì. 001	. 23
7	ь. 16	1, 68	. 03		. 025	<.005	2.69	09	i, 02	3.02	i. 001	. 22
8	v. 16	1.66	. 02		հ. 019		2.70	.08				.20
9	*. 15	r. 66	. 02		{ *.024} *.02}	i. 008	2.74	.07			k<.01	. 14
10	ь. 16	٠.66	. 01	nii			2.67	.06				. 20
11	u.w. 16	1.68	.01		t. 024	¥.01	2.67	.09				
12	u. 16	f. 67	.02		. 024	.008	2.71	.08	1,02	i. 018		
Average	0.16	0, 66	0.02		0. 022	0.008	2.71	0.07	0.012	0.017	0.001	0, 21

- p heated at 900° on 1,000° C. in a covered platinum is to constant weight. giphed ignited NH₂OH precipitate corrected for TrO₂, and P₂O₃. uninum separated from iron, titanium, etc., with hydroxide, precipitated, and weighed as AIPO₄ me value obtained by the Aluminon photometric

- yanate photometric method. KrCrO7 method.

- IOs. ic. Weighed as MgsPsO7. nich AcNOs, following ion exchange separa-and potassium chlorides.

- 4 E-Hvdrgvruginoling prycipitation. Bromate-shioantfer-tratesion. See Trata. British Ceramic Society SI, No. 9, 45 Sodium usayl zinc acetate gravimetric method. * Titrated with Tit (SO). * Phosphomolyhdate-alkalimetric method. * Weighed as APCu. * Weighed as APCu. * Same value obtained by eriochromocyanius-R photo-metric method.

List of Analysts

- K. M. Sappenfield and R. A. Paulson, National Bureau of Standards.
 G. R. Eusner, U. S. Steel Corp., Monroeville, Pa.
 B. E. Gummo, W. O. Osborn, and R. N. Smith, Crescent Division, North American Refractories Co., Curwensville, Pa.
 M. P. Bennett and Falba Whitney, Gladding, McBean & Co., Los Angeles, Calif.
 B. C. Ruprecht, Harbison-Walker Refractories Co., Pittsburgh, Pa.

- A. R. Lesar, A. P. Green Fire Brick Co., Mexico, Mo.
 L. J. Trostel, General Refractories Co., Baltimore, Md.
 D. J. Hallisey, Jones & Laughlin Steel Corp., Pittsburgh, Pa.
 J. J. Hazel, Republic Steel Corp., Cleveland, Ohio.
 Paul Smith, The Refractories Institute, Mellon Institute, Pittsburgh, Pa.
 J. B. Armstrong, Sparrows Point Plant, Bethlehem Steel Co., Sparrows Point, Md.
 W. F. Zollinger, Bethlehem Steel Co., Bethlehem, Pa.

A. V. ASTIN, Director.

Washington 25, D.C., January 6, 1960.

UNITED STATES DEPARTMENT OF COMMERCE WASHINGTON 25, D.C.

National Bureau of Standards Certificate of Analyses

Standard Sample 199 Silica Brick

(All results are based on samples dried at 105° to 110° C.)

Analyst	Al ₂ O ₃	Total iron as Fe ₂ O ₃	TiO ₂	ZrO ₂	P ₂ O ₅	MnO	CaO	MgO	Na ₂ O	K ₂ O	Li ₂ O	* Loss on ignition
1	{ ⁶ 0. 47 • 48	4 0. 76 •. 74}		0.01	10.010	* 0, 005	2.41	0.13	h 0. 015	₽ 0. 09	h 0. 001	0.14
2	i. 4 9	i. 70	i. 05		1.016ء		i 2.45	i. 14	i. 010	i. 07	1.002	. 23
3	b. i. 48	k. 75	. 05				2,42	.12	1.02	1.11		. 16
4	m. 48	d. 73	.06		n. 026		2, 38	. 14	°. 014	١.11	i<.007	. 12
5	ь. 51	P. 74	.06		٩. 012		2, 39	. 13	h. 010	հ. 088	h. 002	. 16
6			. 07		.015	.008	2.46	. 14	1.004	h. 10	h. 001	. 12
7	ь. 50	a. 75	. 07		. 025	<.005	2, 43	. 10	h. 02	h. 09	h. 002	. 16
8	r. 48	d. 76	.07		1,005		2.38	. 14				. 13
9	·	. 72	.06		٩. 013	e. 007		i. 14			i<.01	.30
10	b. 49	a. 75	.06	. nil	.03		2,37	. 13				. 17
11	r. t. 48	d. 74	.06		a. 008	u. 01	2.38	. 14		v. 097		
12	r. 48	d. 74	.06		۹. 010	≈. 007	2, 39	. 14	h. 03	h. 087		
Average	0.48	0.74	0.06		0.015	0.007	2.41	0, 13	0.015	0.094	0, 002	0. 17

- 1 g heated at 900° C. to 1,000° C. in a covered platinum rucible to constant weight of Widghed ignited NH₂OH precipitate corrected for ZrO₂, Weighted ignited NH₂OH precipitate corrected for ZrO₃, 4 Annianum separated from iron, titanium, and zirconium, youtperron, precipitated with ammonium hydroxide, eighed as Al₂O₃ and corrected for PrO₂.
 8 SuCL-K-CrO₃ method.

- quinoline precipitation. Bromate-thiosul-See Trans. British Ceramic Society 51, No.
- Gravimetric. Weighed as MgsPsOn.
 Sodium uranyl zinc acetate-gravimetric method.
 Titrated with Ti(SOA):
 Phosphomolybate-silizalmetric method.
 Weighed as AIPO.
 Orthophenanthroline photometric method.
 Same value obtained by eriochromecyanine-R photomentic method.
- Persulfate-arsenite method.
 Gravimetric. Decomposition with NH4Cl-CaCO1.

List of Analysts

- 1. K. M. Sappenfield and R. A. Paulson, National Bureau of Standards.
 2. G. R. Eusner, U.S. Steel Corp., Monroeville, Pa.
 3. B. E. Gummo, W. O. Osborn, and R. N. Smith, Crescent Division, North American Refractories Co., Curwensville, Pa.
 4. M. P. Bennett and Falba Whitney, Gladding, McBean & Co., Los Angeles, Calif.
 5. B. C. Ruprecht, Harbison-Walker Refractories Co., Pittsburgh, Pa.

- A. R. Lesar, A. P. Green Fire Brick Co., Mexico, Mo.
 L. J. Trostel, General Refractories Co., Baltimore, Md.
 D. J. Hallisey, Jones & Laughlin Steel Corp., Pittsburgh, Pa.
 J. J. Hazel, Republic Steel Corp., Cleveland, Ohio.
 Paul Smith, The Refractories Institute, Mellon Institute. Pirtsburgh Pa
- 10. Fall Smith, The Refractories Institute, Mellon Institute, Pittsburgh, Pa.
 11. J. B. Armstrong, Sparrows Point Plant, Bethlehem Steel Co., Sparrows Point, Md.
 12. W. F. Zollinger, Bethlehem Steel Co., Bethlehem, Pa.

WASHINGTON 25, D.C., January 6, 1960.

A. V. ASTIN, Director.

U.S. GOVERNMENT PRINTING OFFICE 537582

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 277

Tungsten Concentrate

(In cooperation with the American Society for Testing and Materials)

This material is in the form of powder (<0.15 mm) intended for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

 Constituent
 Certified Value¹ (wt%)
 Estimated Uncertainty² (wt%)

 WO3
 67.4
 0.3

(Results are based on samples dried at 110 °C for one hour)

CAUTION: The material for this SRM primarily was derived from wolframite ores. It is, however, a mixture of concentrates from China, Thailand, and USA that contains titanium, niobium, tantalum, and tin in amounts not normally encountered in most wolframite concentrates. These constituents may interfere in the "classic" chemical procedures and may necessitate appropriate changes in methodology. Also, x-ray fluorescence methods of analysis that use SRM 277 to make relative measurements of "pure" wolframite (or scheelite) concentrates may exhibit systematic errors because of the unusual constituents contained in this concentrate.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, NBS-ASTM Research Associate Program.

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. E. Michaelis.

Washington, D.C. 20234 October 24, 1978 J. Paul Cali, Chief Office of Standard Reference Materials

The certified value is the present best estimate of the "true" value based on the results of the cooperative program for certification.

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 I g or more (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination.)

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for SRM 277 was carefully selected and provided to NBS by GTE Sylvania, Towanda, Pennsylvania, courtesy of J. Demangone. At Sylvania, the material was crushed and ground to a fine powder. At NBS the material was sieved (<0.15 mm) and thoroughly blended

Homogeneity testing of selected samples representative of the lot of SRM 277 was performed by an x-ray fluorescence technique by R. E. Michaelis. The material variability was determined to be within ± 0.1 (wt%) of WO₃.

Cooperative analyses for certification were performed in the following analytical laboratories:

Alfred H. Knight International, Ltd., Cheshire, U.K. - J. F. L. Knight.

Benedict Kitto and Sons, London, U.K. - R. Peck.

General Electric Company, Cleveland, Ohio - J. Rynasiewicz and J. W. Fulton.

GTE Sylvania, Towanda, Pa. - R. Dyck; and Waltham, Mass. - J. F. Cosgrove.

Hermann C. Starck, Berlin, Germany - O. Hilmer.

Ledoux & Company, Teancek, N. J. - S. Kallmann.

National Bureau of Standards, Washington, D.C. - E. R. Deardorff.

Sandvik, A. B., Stockholm, Sweden - K. Käärik; and Sandvik Asia Ltd. - N. R. Sanjana.

Spectro Chem Labs, Inc., Franklin Lakes, N. J. - E. W. Hobart.

Treibacher Chemische Werke, Treibach, Austria - Z. Otto.

Westinghouse Electric Corporation, Bloomfield, N. J. - P. J. Walitsky.

Union Carbide Corporation, Bishop, Calif. - E. C. Gibbs and K. M. Wilder; and Niagara Falls, N. Y. - P. Greenberg.

VEW Ternitz, Austria - A. J. Leeb.

NOTE: Details regarding the methodology employed in the analysis of this SRM, along with other pertinent information, will appear in a separate publication.

ADDITIONAL INFORMATION OF THE COMPOSITION: Certification is made only for the WO₃ content; however, SRM 277 contains additional constituents of interest as indicated below. These are not certified and are provided for information only. Table 1 lists those constitutents for which data were received from two or more laboratories that were in good agreement. (Most of these are expected to be proposed for certification at a later date and, therefore, an indication of the uncertainty also is given.) Table 2 lists those constituents for which data were received from a single laboratory (or discrepant data from two or more laboratories).

NOT CERTIFIED

Т	2	h	۱e	1

4400 1						
Constituent	Approximate Value (wt%)	Approximate Uncertainty (wt%)				
Calcium	(0.37)	(0.02)				
Iron	(7.4)	(.1)				
Lead	(0.07)	(.01)				
Manganese	(10.0)	(.2)				
Molybdenum	(0.06)	(.01)				
Niobium	(1.00)	(.03)				
Phosphorus	(0.03)	(.01)				
Silicon	(.85)	(.05)				
Sulfur	(.25)	(.03)				
Tin	(.54)	(.07)				
Titanium	(2.2)	(.2)				

NOT CERTIFIED

Table 2

Constituent	Information Value (wt%)	Constituent	Information Value (wt%)
Arsenic	(0.015)	Oxygen	(21.4)
Bismuth	(.07)	Scandium	(0.05)
Cerium	(.03)	Tantalum	(.20)
Chromium	(.04)	Thorium	(80.)
Copper	(.02)	Uranium	(.13)
Gadolinium	(.01)	Yttrium	(.03)
Lanthanum	(.02)	Zirconium	(.01)
Neodymium	(.03)		, ,

In addition to the above, trace concentrations were reported (ppm by wt.) for the following as an indication of the relative amounts observed: Antimony (3), Barium (20), Boron (20), Dysprosium (30), Erbium (20), Europium (10), Holmium (5), Hafnium (20), Indium (20), Lutetium (5), Praseodymium (50), Samarium (40), Tellurium (2), Terbium (10), Thallium (2), Thulium (10), Vanadium (70), Ytterbium (50) and Zinc (20).

NOTE: Although SRM 277, Tungsten Concentrate, is expected to be stable under normal storage conditions, prudent cautions in the laboratory should be observed. The bottle should be kept tightly capped except when in direct use. Store in a desiccator over desiccant.

National Bureau of Standards

Certificate

Standard Reference Material 278 **Obsidian Rock**

This Standard Reference Material (SRM) is intended for use in evaluating the accuracy of analytical methods and instruments used in the analysis of geological type materials. SRM 278 is a finely powdered obsidian rock, which was obtained from Clear Lake, Newberry Crater, Oregon.

Certified Values of Constituents

The concentrations of the constituents were determined by methods that are widely used in the field of geological analysis and have a demonstrated accuracy. The values given are "certified" values, i.e., those values that were determined by either a definitive method, reference method, or by two or more independent methods, and "information" values that were determined by single or non-reference methods. The certified values are given in Table 1.

Table 1. Certified Values of Constituents

	Content ²		Content ²
Constituent 1	(wt %)	Constituent 1	wt (μg/g)
Al ₂ O ₃ d	14.15 ± 0.15	Cu ^e	5.9 ± 0.2
CaO ^c	0.983 ± 0.002	Ni ^e	3.6 ± 0.3
FeO i	1.36 ± 0.02	Rb a,e	127.5 ± 0.3
Fe ₂ O ₃ i, f (Total Fe as Fe ₂ O ₃)	2.04 ± 0.02	Sr °	63.5 ± 0.1
K ₂ O a,d,e	4.16 ± 0.02	Th e,f	12.4 ± 0.3
MnO b,f	0.052 ± 0.002	TI °	0.54 ± 0.04
Na ₂ O a,d,f	4.84 ± 0.05	U e	4.58 ± 0.04
P ₂ O ₅ b,d	0.036 ± 0.003	Pb °	16.4 ± 0.2
SiO ₂ d	73.05 ± 0.13		
TiO ₂ b,g	0.245 ± 0.007		

Methods of Analysis:

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 T. E. Gills.

Washington, D.C. 20234 August 19, 1981

George A. Uriano, Chief Office of Standard Reference Materials

Atomic Absorption ^bColorimetry

Emission Spectrometry

^{&#}x27;Isotope Dilution Mass Spectrometry

Neutron Activation Analysis

Prompt-gamma Activation Analysis
Specific Ion Electrode Potentiometry

Volumetry

The estimated 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 of 250 mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the

Supplemental Information

Preparation

The material was processed by the Colorado School of Mines, Golden, Colorado. Approximately 350 lbs of obsidian rock were crushed, ground, and sieved to <200 mesh. The material was mixed in a cone blender to ensure homogeneity. For homogeneity testing and certification samples were randomly chosen and analyzed for both major and minor constituents. The inhomogeneity of this material is considered to be $\leq 2\%$ relative.

Analysis

SRM 278, a natural glass, is hygroscopic and contains water that cannot be driven off by drying at low temperatures. This material will pick up additional water on exposure to the atmosphere. Thus, exposure time should be kept to a minimum. Furthermore, the sample should be ignited to a constant weight in a muffle furnace or over a small flame at a temperature between 350-600 °C. This procedure will ensure the accurate and precise determination of SiO₂, K_2O , Na_2O , Al_2O_3 and possibly other major constituents.

The analysts and laboratories cooperating in the analytical program for certification were:

- M. J. Blackman, E. L. Garner, J. W. Gramlich, L. A. Machlan, L. J. Moore, and R. Zeisler of the Inorganic Analytical Research Division, National Bureau of Standards.
- J. B. Bodkin, J. C. DeVine, and N. H. Suhr of the Mineral Constitution Laboratories, The Pennsylvania State University, University Park, Pa.
- S. S. Goldich of the Department of Geology, Northern Illinois University, DcKalb, Ill.
- M. D. Glascock, C. C. Graham, J. R. Vogt, University of Missouri, Columbia, Mo.

The constituents given in Table 2 are not certified but are included for information only.

	Table 2. Information	n Values	
Constituent ¹	Content wt %	Constituent 1	Content wt (µg/g)
C (Total Carbon) ^j	(0.05)	Ba ^f	(1140)
CO ₂ d	(0.01)	В	(25)
F h	(0.05)	Ce ^f	(62.2)
MgO ^d	(0.23)	Co f	(1.5)
		Cr f	(6.1)
		Cs ^f	(5.5)
		Eu f.	(0.84)
		Gd ^g	(5.3)
		Hf ^f	(8.4)
		Lu ((0.73)
		Sb f	(1.5)
		Sc ^f	(5.1)
		Sm ^{f,g}	(5.7)
		Ta ⁽	(1.2)
		Tb 1	(1.0)
		Yb ((4.5)
		Zn f	(55)

U.S. Department of Commerce Rogers C. Morton,

National Bureau of Standards Certificate of Analysis Standard Reference Materials 113a and 329 Zinc Concentrates

These Standard Reference Materials are in the form of fine powder (<.15 mm) and are intended for use both in checking chemical methods of analysis and in calibration with instrumental methods

CAUTION: The bottle should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.

SRM No. Element	113a 329 Percent by Weight ^a		
Zinc	57.3	45.5	
Lead	2.80	6.06	
Iron	2.08	12.94	
Calcium Oxide	1.10	0.08	
Magnesium Oxide	0.75	.165	
Cadmium	.78	.14	
Copper	.31	.132	
Cobalt	$(.11)^{b}$	(.009)	
Nickel	(.07)	(.006)	
Sulfur (Total)	30.6	(31.7)	
Silicon Dioxide	(1.54)	(0.61)	
Indium	c	.019	
Silver	0.046 z ^d	.0089 ^d	

odried at 105 °C for oue hour, Moisture loss: for SRM 113a 0.08%, for SRM 329 0.4₅%, hesis are not certified but are given for information only.

Figures in parent Not determined.

NOTE: The total of constituents reported for SRM 113a is 97.6% and that for SRM 329 is 98.0%. The remainder is expected to consist mainly of oxygen, carbon, and water of crystallization.

CERTIFICATION: The value listed for a certified element is the present best estimate of the "true" value based on the results of the analytical program. The value listed is not expected to deviate from the "true" value by more than ± 1 in the last significant figure reported. For a subscript figure, the deviation is not expected to be more than ± 5 . Based on the results of homogeneity testing, maximum variations within and among samples are estimated to be less than the uncertainty figures given above.

Washington, D.C. 20234 December 29, 1975 (Revision of Certificate of May 6, 1974 for change in silver results)

J. Paul Cali, Chief Office of Standard Reference Materials

Not determined.

Troy ounces per ton is 13.63 for SRM 113a and 2.60 for SRM 329.

The material for these standards was supplied by Comineo American Inc., Spokane, Wash. The material for SRM 113a was prepared at the Magmont Mines, Bixby, Mo., and that for SRM 329 at the Sullivan Mine, Trail, B.C., Canada.

Following sieving and blending operations at NRS, homogeneity testing was performed by S. D. Rasberry and J. McKay, (x-ray fluorescence analyses); by E. J. Maienthal, (polarographic analyses); and by R. K. Bell, (chemical analyses).

Selected samples representative of the lot were analyzed and no significant variability was observed when using subsamples of $0.5~\rm g$ or larger. (Moisture determinations usually were made on larger samples-up to $10~\rm g$.)

Cooperative analyses for certification were performed in the analytical laboratories of Cominco, Sullivan Mine, Trail, B.C., Canada, C. J. Mitchell; Cominco American Inc., Magmont Mines, Bixby, Mo., R. J. Gibson; and St. Joe Minerals Corp., Zine Smelting Division, Monaca, Pa., J. J. Aldrich.

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell and E. J. Maienthal.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of O. Menis and J. I. Shultz.

The technical and support aspects involved in the preparation, certification, and issuance of these SRM's were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

U.S. Department of Commerce Elliot L. Richardson, National Bureau of Standards Ernest Ambler, Acting Director

National Bureau of Standards Certificate of Analysis

Standard Reference Material 330 Copper Ore, Mill Heads

This material is in the form of fine powder intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

This SRM is one of a series of four SRM's issued primarily for use in evaluation of the critically important "material balance" in the copper mining and metallurgical industries. The other three are: SRM 331, Copper Ore, Mill Tails; SRM 332, Copper Concentrate: and SRM 333, Molybdenum Concentrate.

CAUTION: The bottle should be kept tightly closed except when in direct use. Store in a desiccator over

Constituent	Certified Value ^{a, b}	Estimated Uncertainty ^c	
	Percen	t by Weight	
Total Copper	0.84	0.01	
Molybdenum	.018	.001	
	PPM by Weight		
Rhenium	0.30	0.06	

The following values indicate the results of the analytical tests made at NBS and the Magma Copper Company.

Constituent / Method ¹	Average	Standard Deviation ²	Number of Determinations
	Percent by	/ y Weight	
Total Copper			
Isotopic dilution mass spectrometry	0.837	0.007	8
(0.5 g samples)			
Polarographic (0.5 g samples)	.85	.01	3
Iodometric (2.5 g samples) ⁴	.85	.01	21
Molybdenum		Range	
Isotopic dilution mass spectrometry	.0180	0.0001	,
(1 g samples)			-
	PPM by	Weight	
Rhenium		Range	
Isotopic dilution mass spectrometry	0.303	0.054	6
(2.5 g samples)	5.555	0.054	U

Details of the methods used, including drying and dissolution procedures, are given in a separate publication [1].

Of single determinations for total Cu; range given for Mo and Re.

This method has been studied extensively and the data are considered free from systematic errors [2].

Results from Magma Copper Company

Washington, D. C. 20234 January 20, 1977 (Revision of Provisional Certificate of 2-20-73)

J. Paul Cali, Chief Office of Standard Reference Materials

(Over)

Based on samples dried at 105 °C for two hours. Separate samples are used for rhenium and calculated to a dry-weight basis.
The certified value is the best estimate of the "true" value.
Estimated uncertainty includes both method imprecision and material variability with samples 0.5 g (or more) for total copper, 1.0 g (or more) for molybdenum, and 2.5 g (or more) for rhenium.

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for this SRM (330) was carefully selected and provided to NBS by Magma Copper Company, San Manuel, Arizona, through the courtesy of T. L. Young.

At NBS this material was sieved and thoroughly blended, which involved several independent procedures [1].

Homogeneity testing of selected samples representative of the lot of SRM 330 was performed simultaneously with the analytical program for certification. At NBS, 0.5 g samples showed the maximum variability for total copper to be ± 0.010 percent. At Magma, 2.5 g samples showed the maximum variability to be ± 0.01 percent.

Cooperative analyses were performed at the Magma Copper Company, San Manuel, Arizona, by B. Cripe, R. L. Culder, A. B. Hall, D. A. Shah, J. T. Tadano, and M. Toelkes.

Analyses were performed in the NBS Analytical Chemistry Division by E. L. Garner, J. W. Gramlich, L. A. Machlan, E. J. Maienthal, J. R. Moody, and T. J. Murphy.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of W. R. Shields and I. L. Barnes.

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. E. Michaelis.

ADDITIONAL INFORMATION ON THE COMPOSITION: Certification is made *only* for total Cu, Mo, and Re. Although <u>NOT CERTIFIED</u>, the following additional information on the composition may be of interest.

Gold and Silver

Fire assay determinations for Au and Ag were made at Magma Copper Company:

	Gold	Silver	
	PPM b	PPM by Weight	
Fire assay	(0.093)	*(1.51)	

^{*}Revised 02/28/79

"Acid-Soluble" Copper

The total copper content includes "recoverable" sulfide copper and "nonrecoverable" oxide copper. Industrial practice is to determine "acid-soluble" copper and to relate this result to the oxide copper content. Investigation at NBS provided additional information on "acid-soluble" copper that may be useful, but is not certified:

Constituent/Method	Average	Range	Number of Determinations
"Acid-Soluble" Copper	Percent	by Weight	
Isotopic dilution mass spectrometry (2.5 g samples)	(0.069)	(0.063-0.081)	9

REFERENCES

- J. R. Moody, I. L. Barnes, and R. E. Michaelis, Standard Reference Materials: Copper Ore, Mill Heads-SRM 330: Copper Ore, Mill Tails - SRM 331; Copper Concentrate - SRM 332, and Molybdenum Concentrate - SRM 333; Nat. Bur. Stand. Spec. Publ. 260-xx (in press).
- [2] W. R. Shields, Editor, Nat. Bur. Stand. Tech. Note 546, (1970).

U.S. Department of Commerce Elliot L. Richardson,

National Bureau of Standards Mational Bureau of Standards Ernest Ambler, Acting Director Certificate of Analysis Standard Reference Material 331

Copper Ore, Mill Tails

This material is in the form of fine powder intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

This SRM is one of a series of four SRM's issued primarily for use in evaluation of the critically important "material balance" in the copper mining and metallurgical industries. The other three are: SRM 330, Copper Ore, Mill Heads; SRM 332, Copper Concentrate; and SRM 333, Molybdenum Concentrate.

CAUTION: The bottle should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.

Constituent	Certified Value ^{a,b}	Estimated Uncertainty ^c	
	Percent by Weight		
Total Copper	0.091	0.001	
Molybdenum	.0022	.0002	
	PPM by Weight		
Rhenium	.04	.02	

- Based on samples dried at 105 °C for two hours. Separate samples are used for rhenium and calculated to a dry-weight basis.
- The certified value is the best estimate of the "true" value.
- Estimated uncertainty includes both method imprecision and material variability with samples 1.0 g (or more) for total copper and molybdenum, and 2.5 g (or more) for rhenium.

The following values indicate the results of the analytical tests:

Constituent/Method ¹	Average	Standard Deviation ²	Number of Determinations
Total Copper	Percent	by Weight	
Isotopic dilution mass spectrometry ³	0.0915	0.0005	15
(1.0 g samples) Polarographic (2.5 g samples)	.091	.001	4
Molybdenum		Range	
Isotopic dilution mass spectrometry ³ (1.0 g samples)	.0022 PPM b	0.0001 y Weight	2
Rhenium Isotopic dilution mass spectrometry (2.5 g samples)	0.043	<u>Range</u> 0.016	6

- Details of the methods used, including drying and dissolution procedures, are given in a separate publication [1].
 Of single determinations for total Cu; range given for Mo and Re.
 This method has been studied extensively and the data are considered free from systematic errors [2].

Washington, D. C. 20234 January 20, 1977 (Revision of Provisional Certificate of 2-20-73)

J. Paul Cali, Chief Office of Standard Reference Materials

(Over)

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for this SRM (331) was carefully selected and provided to NBS by Magma Copper Company, San Manuel, Arizona, through the courtesy of T. L. Young.

At NBS, this material was sieved and thoroughly blended, which involved several independent procedures [1].

Homogeneity testing of selected samples representative of the lot of SRM 331 was performed simultaneously with the analytical program for certification. At NBS, the maximum variability for total copper was determined to be ±0.0008 percent (15 determinations with 1 g samples).

Analyses were performed in the NBS Analytical Chemistry Division by E. L. Garner, J. W. Gramlich, L. A. Machlan, E. J. Maienthal, J. R. Moody, L. J. Moore, and T. J. Murphy.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of W. R. Shields and I. L. Barnes.

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. E. Michaelis.

ADDITIONAL INFORMATION ON THE COMPOSITION: Certification is made *only* for total Cu, Mo, and Re. Although NOT CERTIFIED, the following additional information on the composition may be of interest.

Gold and Silver

Fire assay determinations for Au and Ag were made at Magma Copper Company:

		Gold	Silver
		PPM by Weight	
Fire assa	ıy	(0.034)	*(0.243)

*Revised 02/28/79

"Acid-Soluble" Copper

The total copper content includes "recoverable" sulfide copper and "nonrecoverable" oxide copper. Industrial practice is to determine "acid-soluble" copper and to relate this result to the oxide copper content. Investigation at NBS provided additional information on "acid-soluble" copper that may be useful, but is not certified:

Constituent/Method	Average	Range	Number of Determinations
"Acid-Soluble" Copper	Percent by Weight		
Isotopic dilution mass spectrometry (2.5 g samples)	(0.051)	(0.051-0.052)	3

REFERENCES

- J. R. Moody, I. L. Barnes, and R. E. Michaelis, Standard Reference Materials: Copper Ore, Mill Heads-SRM 330; Copper Ore, Mill Tails - SRM 331; Copper Concentrate - SRM 332, and Molybdenum Concentrate - SRM 333, Nat. Bur. Stand. Spec. publ. 260-xx (in press).
- [2] W. R. Shields, Editor, Nat. Bur. Stand. Tech. Note 546, (1970).

National Bureau of Standards Certificate of Analysis

Standard Reference Material 332 Copper Concentrate

This material is in the form of fine powder intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

This SRM is one of a series of four SRM's issued primarily for use in evaluation of the critically important "material balance" in the copper mining and metallurgical industries. The other three are: SRM 330, Copper Ore, Mill Heads; SRM 331, Copper Ore, Mill Tails; and SRM 333, Molybdenum Concentrate.

CAUTION: The bottle should be kept tightly closed except when in direct use. Store in a desiccator over

Constituent	Certified Value ^{a b}	Estimated Uncertainty ^c
	Percent	by Weight
Total Copper	28.4	0.1
Molybdenum	0.64	.01
	PPM by	Weight
Rhenium	10.2	0.2
		1.

a Based on samples dried at 105 °C for two hours. Separate samples are used for rhenium and calculated to a dry-weight basis.

The following values indicate the results of the analytical tests:

Constituent/Method ¹	Average	Standard Deviation ²	Number of Determinations
	Percent by	y Weight	
Total Copper Isotopic dilution mass spectrometry ³ (0.5 g samples)	28.40	0.04	12
Electrogravimetry (2 g samples)	28.39	.02	6
Molybdenum Isotopic dilution mass spectrometry ³	0.639	Range 0.008	3
(0.2 g samples) Rhenium	PPM I	by Weight	
Isotopic dilution mass spectrometry ³ (2.5 g samples)	10.20	<u>Range</u> 0.20	3

Details of the methods used, including drying and dissolution procedures, are given in a separate publication [1].

Washington, D.C. 20234

(Revision of Provisional Certificate of 2-20-73

and draft certificates of 7-4-76 and 1-20-77)

(Over)

J. Paul Cali, Chief Office of Standard Reference Materials

b The certified value is the best estimate of the "true" value.

c Estimated uncertainty includes both method imprecision and material variability with samples 0.5 g (or more) for total copper, 0.2 g (or more) for molybdenum and 2.5 g (or more) for rhenium.

Of single determinations for total Cu; range given for Mo and Re.

This method has been studied extensively and the data are considered free from systematic errors. [2].

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for this SRM (332) was carefully selected and provided to NBS by Magma Copper Company, San Manuel, Arizona, through the courtesy of

At NBS, the material was sieved and thoroughly blended, which involved several independent procedures [1].

Homogeneity testing of selected samples representative of the lot of SRM 332 was performed simultaneously with the analytical program for certification. The maximum variability for total copper was determined to be ±0.06 percent (0.5 g samples).

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell, E. L. Garner, J. W. Gramlich, L. A. Machlan, J. R. Moody, L. J. Moore, and T. J. Murphy.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of W. R. Shields and I. L. Barnes.

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. E. Michaelis.

ADDITIONAL INFORMATION ON THE COMPOSITION: Certification is made only for total Cu, Mo, and Re. Although NOT CERTIFIED, the following additional information on the composition may be of interest.

Gold and Silver

Fire assay determinations for Au and Ag were made at Magma Copper Company:

	Gold	Silver		
	PPM by Weight			
Fire assay	*(2.14)	*(38.7)		

*Revised 02/28/79

REFERENCES

- [1] J. R. Moody, I. L. Barnes, and R. E. Michaelis, Standard Reference Materials: Copper Ore, Mill Heads-SRM 330; Copper Ore, Mill Tails - SRM 331; Copper Concentrate - SRM 332; and Molybdenum Concentrate - SRM 333, Nat. Bur. Stand. Spec. Publ. 260-xx (in press).

 [2] W. R. Shlelds, Editor, Nat. Bur. Stand. Tech. Note 546, (1970).

U.S. Department of Commerce Elliot L. Richardson,

National Bureau of Standards

National Bureau of Standards Certificate of Analysis

Standard Reference Material 333 Molybdenum Concentrate

This material is in the form of fine powder intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

This SRM is one of a series of four SRM's issued primarily for use in evaluation of the critically important "material balance" in the copper mining and metallurgical industries. The other three are: SRM 330, Copper Ore, Mill Heads; SRM 331, Copper Ore, Mill Tails; and SRM 332, Copper Concentrate.

CAUTION: The bottle should be kept tightly closed except when in direct use. Store in a desiccator over

Constituent	Certified Value * b	Estimated Uncertainty ^c	
	Percent b	y Weight	
Total Copper	1.038	0.010	
Molybdenum	55.3	.1	
Rhenium	0.087	.001	

Based on samples run "as received." CAUTION: The bottle should be kept tightly closed except when in direct use.

b The certified value is the best estimate of the "true" value.

The following values indicate the results of the analytical tests made at NBS and the Magma Copper Company.

Constituent/Method	Average	Standard Deviation ²	Number of Determinations
	Percent	by Weight	
Total Copper			·
Isotopic dilution mass spectrometry ³	1.038	0.007	11
(0.25 g samples)			
Polarographic	1.034	.011	. 6
(1.0 g samples)			
Atomic absorption spectrometry	1.033	.002	3
(1.0 g samples)			
Molybdenum			
Isotopic dilution mass spectrometry ³	55.31	.02	10
(0.25 g samples)			
α-Benzoinoxime gravimetric ⁵	55.43	.04	10
(0.25 g samples)			
Rhenium		1	
Isotopic dilution mass spectrometry ³	0.0869	0.0003	17
(0.25 g samples)			**
Thermal neutron activation analysis ⁶	.085	.002	9
(0.15 g samples)			

Details of the methods used are given in a separate publication [1].

Of single determinations.

Washington, D. C. 20234 January 20, 1977

J. Paul Cali. Chief Office of Standard Reference Materials

(Revision of Provisional

Certificate of 2-20-73)

(Over)

Estimated uncertainty includes both method imprecision and material variability with samples 0.25 g (or more) for total copper, molybde-

This method has been studied extensively and the data are considered free from systematic errors [2].

Results from Magma Copper Company.

Average of two independent analysts at NBS.

Determinations made relative to an industrial molybdenum concentrate reference material containing 0.1140 wt. % of Re. (as determined by a number of industrial laboratories and by isotopic dilution mass spectrometry at NBS.)

NOTE: Recommendation is made that this material be analyzed in the "as received" condition. SRM 333 contains a small quantity (about 1%) of fuel oil introduced in the flotation process. Investigations at NBS indicate that the analytical results made on the "as received" samples may be calculated to the dry-weight basis by use of an acetone stripping procedure (includes fuel oil and moisture)[1]. (For information only, a calculated MoS₂ content thus would be 93.2±0.2%.)

ADDITIONAL INFORMATION ON THE COMPOSITION: Although certification is made only for total Cu, Mo, and Re, recommended values for Au and Ag are given below:

	Au	Ag
	ppn	n by weight
Fire Assay, Magma (14.58 g samples)	8.9	25.0

Neutron activation values at NBS on 0.2 g portions (final blended material) gave values of 12.5 ppm Au and 22.6 ppm Ag. Definite evidence of inhomogeneity was observed for Au on these 0.2 g portions. Recommendation is made that sample sizes of 2.5 g (or more) be used to ensure homogeneity (<5% relative), regardless of the method used.

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for this SRM was carefully selected and provided to NBS by Magma Copper Company, San Manuel, Arizona, through the courtesy of T. L. Young.

At NBS, highly specialized blending and mixing procedures were employed to obtain satisfactory homogeneity. Extensive chemical analyses performed by Magma both for total copper and for molybdenum sulfide demonstrated that homogeneity was not achieved until the total blending and mixing procedures were performed three separate times [1].

Final homogeneity testing was performed at NBS simultaneously with the analytical program for certification. The maximum variability of the accepted lot was determined to be ± 0.010 percent for total copper, ± 0.0010 percent for rhenium; and ± 0.06 percent for molybdenum (all using 0.25 g samples).

Cooperative analyses were performed at the Magma Copper Company, San Manuel, Arizona, by Y. Arias, B. Cripe, R. L. Culver, A. B. Hall, D. A. Shah, J. T. Tadano, and M. Toelkes.

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell, E. L. Garner, T. E. Gills, J. W. Gramlich, P. D. LaFleur, L. A. Machlan, E. J. Maienthal, J. R. Moody, L. J. Moore, and T. J. Murphy.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of W. R. Shields and I. L. Barnes.

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. E. Michaelis.

REFERENCES

- J. R. Moody, I. L. Barnes, and R. E. Michaelis, Standard Reference Materials: Copper Ore, Mill Heads-SRM 330; Copper Ore, Mill Tails - SRM 331 Copper Concentrate - SRM 332, and Molybdenum Concentrate - SRM 333, Nat. Bur. Stand. Spec. Publ. 260-xx (in press).
- [2] W. R. Shields, Editor, Nat. Bur. Stand. Tech. Note 546, (1970).

U. S. Department of Commerce
Malcolar Baldrige
Secretary
National Bussess of Standards
Ernest Ambler, Director

National Bureau of Standards Certificate

Standard Reference Material 688

Basalt Rock

This Standard Reference Material (SRM) is intended for use in evaluating the accuracy of analytical methods and instruments used in the analysis of geological type materials. SRM 688 is a finely powdered basalt rock that was obtained from a Cenozoic basalt flow near Jackpot, Nevada.

Certified Values of Constituents

The concentrations of the constituents were determined by methods that are widely used in the field of geological analysis and have a demonstrated accuracy. The values given are "certified" values, i.e., those values that were determined by either a definitive method, reference method, or by two or more independent methods, and "information" values that were determined by single or non-reference methods. The certified values are given in Table 1.

Table 1 Certified Values of Constituents

Constituent i	Content ² wt (%)	Constituent 1	Content ² wt (μg/g)		
Al ₂ O ₃ c,g	17.36 ± 0.09	Cr b,e	332 ± 9		
FeO ^g	7.64 ± 0.03	Rb ^d	1.91 ± 0.01		
Fe ₂ O ₃ e,g	10.35 ± 0.04	Sr d	169.2 ± 0.7		
(Total Fe as Fe ₂ O ₃)		Th d	0.33 ± 0.02		
K ₂ O b,d	0.187 ± 0.008	Pb ^d	3.3 ± 0.2		
MnO a,b,e	0.167 ± 0.002				
Na ₂ O b,c,e	2.15 ± 0.03				
P ₂ O ₅ a,c	0.134 ± 0.003				
SiO ₂ c	48.4 ± 0.1				
TiO ₂ a,b	1.17 ± 0.01				

^{1.} Methods of Analysis

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 T. E. Gills.

Washington, D.C. 20234 August 18, 1981 George A. Uriano, Chief Office of Standard Reference Materials

^aColorimetry Emission spectrometry

Gravimetry

disotope dilution mass spectrometry

Neutron activation analysis

Specific ion electrode potentiometry Titrimetry

^{2.} The estimated 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 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

Preparation

The material was processed by the Colorado School of Mines, Golden, Colorado. Approximately 600 pounds of basalt rock were crushed, ground, and sieved to <200 mesh. The material was mixed in a cone blender to ensure homogeneity. The inhomogeneity was tested by taking random samples and analyzing for both major and minor constituents and was found to be $\leq 2\%$ relative.

Analysis

SRM 688 may pick up additional water on exposure to the atmosphere. Thus, exposure time should be kept to a minimum. Before analysis, it is recommended that the material be dried at 105 °C for 24 hours. Typical weight loss upon drying is approximately 0.2 percent.

The analysts and laboratories cooperating in the analytical program for certification were:

- I. L. Barnes, M. J. Blackman, E. L. Garner, J. W. Gramlich, L. A. Machlan, L. J. Moore, and R. Zeisler of the Inorganic Analytical Research Division, National Bureau of Standards.
- J. B. Bodkin, J. C DeVine, and N. H. Suhr of the Mineral Constitution Laboratories, The Pennsylvania State University, University Park, Pa.
- S. S. Goldich of the Department of Geology, Northern Illinois University, Dekalb, Ill.

The constituents given in Table 2 are not certified, but are included for information only.

Table			

Constituent 1	Content wt %	Constituent 1	Content wt (µg/g)
CaO c	(12.17)	Ce e	(13.3)
CO ₂ f	(0.05)	Co °	(49.7)
F ^f	(0.02)	Eu e	(1.07)
MgO ^c	(8.4)	Hf c	(1.6)
		Lu ^e	(0.34)
		Sc e	(38.1)
		Ва	(200)
		V b	(250)
		Cu b	(96)
		Ni ^b	(150)
		Sm °	(2.79)
		Tb °	(0.448)
		υď	(0.37)
		Yb °	(2.09)
		Zn ^e	(58.0)

U.S. Department of Commerce Juanita M. Kreps Secretary

ational Bureau of Standards Ernest Ambler, Director

National Bureau of Standards Certificate of Analysis

Standard Reference Material 690

Iron Ore Concentrate (Canada)

(In Cooperation with the American Society for Testing and Materials)

This material is in the form of powder (<0.1mm) for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

(Results based on samples dried for one hour at 105 °C.)

	,	(1703	uits oas	Cu On sa	mpies are	7	TIC HOUL	41 103	····		
Constituent	Total Fe	SiO ₂	Al ₂ O ₃	P	S	TiO ₂	MnO	CaO	MgO	Na ₂ O	K ₂ O
Certified 1 Value (wt. %)	66.85	3.71	0.18	0.011	0.003	0.022	0.23	0.20	0.18	0.003	0.0030
Estimated ² Uncertainty	0.07	0.02	0.01	0.002	0.001	0.002	0.01	0.01	0.01	0.001	0.0005
Method ³	SnCl ₂ K ₂ Cr ₂ O ₇	HClO ₄ Dehydration	Atomic Atsorption	Photometric	Combustion- Titration	Photometric	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption
A	66.91	3.70	0.17	° 0.009	<0.005	d 0.021	0.24	0.20	0.17	0.0026	0.0030
В	66.88	3.76	.17	c .013	.003	e .026	.23	.20	.19	.004	.003
С	f 66.82	3.70	.19	.011	.006	9.024	h .23	i .19	i .19	.0023	.0034
D	66.85	3.73	ر 18.	_	-	J .021	.23	.21	.18	.0028 J.0030	.0029
E	66.83	^h 3.69 3.73	k .20	.009	.002	.022	.24	.21	.18	.002	.003

- 1. The certified value listed for a constituent is the present best estimate of the "true" value based on results of the cooperative analytical program for certification.
- 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 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determinations.)
- A detailed description of many of the methods of analysis employed in the certification program for this SRM may be found in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards.
- a H₂S reduction
- b Sample fused in Na₂CO₃ c Alkali-molybdate method
- d H₂O₂ photometric e Atomic absorption f Silver reductor

- g Chromotropic acid photometric h Photometric method
- i Spectroscopic method
- j Flame emission
- k Chromazurol S photometric

Washington, D.C. 20234 October 24, 1978

J. Paul Cali, Chief Office of Standard Reference Materials

PLANNING, PREPARATION, TESTING, ANALYSIS:

The iron ore powder concentrate material for this SRM was prepared in final powder form, minus 74 μm (200 mesh), by the Iron Ore Company of Canada, Labrador City, Newfoundland, Canada, through the courtesy of L. Rompré.

At NBS, the material was resieved and thoroughly blended.

Homogeneity testing of selected samples representative of the final lot was performed at NBS by R. K. Bell, Assistant Research Associate, ASTM-NBS Research Associate Program. The results for iron indicate that the material variability (0.5 g samples) is $\overline{<}$ the method imprecision.

Chemical analyses for certification were performed in the following laboratories:

Bethlehem Steel Corporation, Homer Research Laboratories, Bethlehem, Pa., D. A. Flinchbaugh.

Inland Steel Company, Indiana Harbor Works, East Chicago, Indiana, J. E. Joyce. Ledoux and Company, Teaneck, New Jersey, S. Kallman and C. L. Maul.

National Bureau of Standards, Center for Analytical Chemistry, Washington, D.C., T. C. Rains, T. J. Brady,

J. D. Messman, and T. A. Rush and by R. K. Bell, ASTM Assistant Research Associate.

STELCO, The Steel Company of Canada, Ltd., Hilton Works, Hamilton, Ontario, Canada, O. P. Bhargava.

The overall direction and coordination of the technical measurements leading to certification were performed jointly by R. E. Michaelis, Office of Standard Reference Materials and by J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

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.

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 691 Reduced Iron Oxide

(In cooperation with the American Society for Testing and Materials)

This Standard Reference Material (SRM) is intended for use in evaluating chemical methods and in calibrations associated with optical emission and x-ray spectrometric methods of analysis. SRM 691 is a finely powdered material (~200 mesh) and must be kept in a tightly sealed bottle when not in use. It is recommended that the material be stored in a desiccator over a suitable desiccant when not in use. Long term (>3 years) stability of this SRM has not been rigorously established. NBS will continue to monitor this material and any substantive changes will be reported to users.

The certified values given below in Table 1 are based on samples of at least 0.5 g, the minimum amount to be used for analysis. Non-certified values which are given for information only, are listed in Table 2.

Table

Constituent	Certified, Value, by by wt.	Estimated Uncertainty ²	Constituent	Certified Value, 1 % by wt.	Estimated Uncertainty
Iron (Total)	90.8	± 0.5	Copper	0.032	± 0.003
Iron (Metallic)3	84.6	.6	Cobalt	.030	.007
SiO ₂	3.7	.2	Phosphorus	.006	.001
Al ₂ O ₃	1.22	.07	Sulfur	.008	.001
TiO ₂	0.27	.04	Carbon (Total)	.12	.03
CaO	.63	.03		1	
MnO	.043	.002			
MgO	.52	.02			
Na ₂ O	.186	.602			

- 1. The certified value listed for a constituent is the present best estimate of the "true" value based on the results of the cooperative program for
- 2. 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 variability for samples 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)
- 3. The metallic iron was determined by the ISO (Bromine-methanol) method.

The overall coordination of the technical measurements leading to certification was performed under the direction of J.I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

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 April 12, 1982 George A. Uriano, Chief Office of Standard Reference Materials

PLANNING, PREPARATION, TESTING, ANALYSIS:

The material for this SRM was provided by Allis-Chalmers, Reduction Systems Division, Milwaukee, Wis., courtesy of L.J. Wrangell. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute, Golden, Colorado, under a contract to the National Bureau of Standards. The final product (-200 mesh) was blended at NBS.

Homogeneity testing of selected samples representative of the final lot was performed at Allis-Chalmers, L.J. Wrangell; at Ledoux & Co., Teaneck, New Jersey, by S. Kalimann; and at NBS by E.R. Deardorff.

Stability tests conducted over a seven-month period at NBS, during which samples were exposed to relative humidities of 75 and 90 percent at room temperature, indicated sufficient stability of the material for use as an SRM.

This material was packaged in a dry nitrogen atmosphere to prevent oxidation. If signs of oxidation are detected, please transmit this information to NBS for documentation into the monitoring program.

Cooperative analyses for certification were performed in the following laboratories:

Andrew S. McCreath & Son, Inc., Harrisburg, Pa.; F.A. Pennington, Jr., R.F. Eakin, G.L. Dobbs, J.C. Forney, and L.W. Richards.

Inland Steel Company, Indiana Harbor Works, East Chicago, Indiana; J.E. Joyce.

Institut de Recherches de la Siderurgie, Maizieres-les-Metz, France, G. Jecko.

Ledoux and Company, Teaneck, New Jersey; S. Kallmann.

National Bureau of Standards, Inorganic Analytical Research Division, C.G. Blundell, T.A. Butler, E.R. Deardorff, M.S. Epstein, R.M. Lindstrom, T.C. Rains, M. Sadjadi, and R.M. Stone.

United States Steel Corp., Research Laboratory, Monroeville, Pa.; J.D. Selvaggio, D.S. Shafferman, A.W. Fioravanti, D.G. Cunningham, K.G. Mikos, R.C. Cline, and H.S. Karp.

The values shown below are not certified since they are not based on the results of at least two independent laboratories or methods. These values are included for information only.

Table 2

Element	Content Wt. Percent	Element	Content µg/g
Cr	(0.03)	As	(14)
Ni	(.3)	Zn	(40)
ĸ	(.06)	Pb	(<20)
		Cd	(< 5)
		Мо	(<20)
		Sn	(<10)
		N	(50)
	And the second second	V	(135)

National Bureau of Standards Ernest Ambler, Director

National Bureau of Standards Certificate of Analysis

Standard Reference Material 692

Iron Ore (Labrador)

(In Cooperation with the American Society for Testing and Materials)

This material is in the form of powder (<0.1 mm) for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

(Results based on samples dried for one hour at 105 °C)

		(XC	suits base	ZU OII SE	impies ai	lea for e	one nou	r at 105	°C.)		
Constituent	Total Fe	SiO ₂	Al ₂ O ₃	P	S	TiO ₂	MnO	CaO	MgO	Na ₂ O	K ₂ O
Certified ¹ Value (wt. %)	59.58	10.14	1.41	0.039	0.005	0.045	0.46	0.023	0.035	0.008	0.039
Estimated ² Uncertainty	0.06	0.05	0.04	0.002	0.001	0.005	0.01	0.003	0.004	0.002	0.003
Method ³	SnC ₂ – K ₂ C ₂ O ₇	HClO ₄ Dehydration	Atomic Abscrption	Photometric	Combustion- Titration	Photometric	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption
A	^a 59.55	b 10.09	1.40	0.039	<0.005	d 0.041	0.47	0.022	0.034	0.008	0.040
., В	59.58	10.26	1.48	.035	.004	e .05	.45	.02	.04	.005	.036
c	f 59.63 f 59.62	10.10	1.42	.040	.007	g .048	h .47	i .025	.028	i .008	1 .040 1 .039
D	59.60	10.16	ر 1.40	_	-	.043	.47	ر 021.	.033	.010. t .009	J .041
E	59.50	10.12 10.05	k 1.41	.039	.004	9 .050	.46	.01	.038	.008	.041
F	59.58	10.18	1.37 1.46	.040	.005	g .043	h .46	.026	.035	.008	.035

- I. The certified value listed for a constituent is the present best estimate of the "true" value based on results of the cooperative analytical program for certification.
- 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 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determinations.)
- A detailed description of many of the methods of analysis employed in the certification program for this SRM may be found in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards.
- b Sample fused in Na₂CO₃ c Alkali-molybdate method d H₂O₂ photometric
- e Atomic absorption
- f Silver reductor
- g Chromotropic acid photometric h Photometric method
- i Spectroscopic method
- i Flame emission
- k Chromazurol S photometric
- I Mercury cathode-NH4OH-Cupferron-AlPO4

Washington, D.C. 20234

October 24, 1978

J. Paul Cali, Chief Office of Standard Reference Materials

PLANNING, PREPARATION, TESTING, ANALYSIS:

The iron ore material for this SRM was prepared in final powder form, minus 74 μ m (200 mesh), by the Bethlehem Steel Corporation, Bethlehem, Pa. through the courtesy of J. M. Karpinski.

At NBS, the material was resieved and thoroughly blended.

Homogeneity testing of selected samples representative of the final lot was performed at NBS by R. K. Bell, Assistant Research Associate, ASTM-NBS Research Associate Program. The results for iron indicate that the material variability (0.5 g samples) is $\overline{<}$ the method imprecision.

Chemical analyses for certification were performed in the following laboratories:

Bethlehem Steel Corporation, Homer Research Laboratories, Bethlehem, Pa., D. A. Flinchbaugh.

Inland Steel Company, Indiana Harbor Works, East Chicago, Indiana, J. E. Joyce.

Ledoux and Company, Teaneck, New Jersey, S. Kallman and C. L. Maul.

National Bureau of Standards, Center for Analytical Chemistry, Washington, D.C., T. C. Rains, T. J. Brady, J. D. Messman, and T. A. Rush, and by R. K. Bell, ASTM Assistant Research Associate.

STELCO, The Steel Company of Canada, Ltd., Hilton Works, Hamilton, Ontario, Canada, O. P. Bhargava.

United States Steel Corporation, Research Laboratory, Monroeville, Pa., L. M. Melnick, J. D. Selvaggio, R. W. Cline, D. G. Cunningham, A. V. Fioravanti, J. R. Lucas II, C. W. Ponsonby, L. E. Povirk, D. Shafferman and R. J. Wargo.

The overall direction and coordination of the technical measurements leading to certification were performed jointly by R. E. Michaelis, Office of Standard Reference Materials, and by J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

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.

U.S. Department of Commerce Juanita M. Kreps Secretary

National Bureau of Standards Certificate of Analysis Standard Reference Material 693

Iron Ore (Nimba)

(In Cooperation with the American Society for Testing and Materials)

This material is in the form of powder (<0.1 mm) for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

	(Results based on samples dried for one hour at 105 °C.)										
Constituent	Total Fe	SiO ₂	Al ₂ U3	Р	s	TiO ₂	MnO	CaO	MgO	Na ₂ O	K ₂ O
Certified ¹ Value (wt. %)	65.11	3.87	1.02	0.056	0.005	0.035	0.091	0.016	0.013	0.0028	0.0028
Estimated ² Uncertainty	0.07	0.02	0.04	0.001	0.001	0.003	0.004	0.004	0.002	0.0005	0.0006
Method ³	SnCl ₂ – K ₂ Cr ₂ O ₇	HClO ₄ Dehydration	Atomic Absorption	Photometric	Combustion- Titration	Photometric	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption
A	65.11	b 3.87	1.04	0.056	<0.005	^d 0.034	0.094	0.016	0.013	0.0029	0.003
В	65.13 65.14	3.83	1.11	.055	.005	f .036	9 .095	h .020	h .011	h .0023	h .003
С	65.10	3.89	1.06	-	-	i .032	.087	i .015	.012	.0030 .0033	.003
D	65.11	3.86 3.88	J 0.98	.056	.005	f .038	.090	.01	.015	.003	.003
Е	65.09	3.87	k 0.98 1.06	.056	.007	f .033	9 .091	.018	.015	.002	.002

- 1. The certified value listed for a constituent is the present best estimate of the "true" value based on results of the cooperative analytical program for certification.
- 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 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determinations.)
- 3. A detailed description of many of the methods of analysis employed in the certification program for this SRM may be found in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards.
- a H₂S reduction
- b Sample fused in Na₂CO₃ c Alkali-molybdate method d H₂O₂ photometric
- Silver reductor
- f Chromotropic acid photometric
- g Photometric method
- h Spectroscopic method i Flame emission
- j Chromazurol S photometric k Mercury cathode-NH₄OH-Cupferron-AlPO₄

Washington, D.C. 20234 October 24, 1978

J. Paul Cali, Chief Office of Standard Reference Materials

PLANNING, PREPARATION, TESTING, ANALYSIS:

The iron ore material for this SRM was prepared in final powder form, minus 74 µm (200 mesh), by the Bethlehem Steel Corporation, Bethlehem, Pa. through the courtesy of J. M. Karpinski.

At NBS, the material was resieved and thoroughly blended.

Homogeneity testing of selected samples representative of the final lot was performed at NBS by R. K. Bell, Assistant Research Associate, ASTM-NBS Research Associate Program. The results for iron indicate that the material variability (0.5 g samples) is \leq the method imprecision.

Chemical analyses for certification were performed in the following laboratories:

Bethlehem Steel Corporation, Homer Research Laboratories, Bethlehem, Pa., D. A. Flinchbaugh. Ledoux and Company, Teaneck, New Jersey, S. Kallman and C. L. Maul. National Bureau of Standards, Center for Analytical Chemistry, Washington, D.C., T. C. Rains, T. J. Brady, J. D. Messman, and T. A. Rush, and by R. K. Bell, ASTM Assistant Research Associate. STELCO, The Steel Company of Canada, Ltd., Hilton Works, Hamilton, Ontario, Canada, O. P. Bhargava. United States Steel Corporation, Research Laboratory, Monroeville, Pa., L. M. Melnick, J. D. Selvaggio, R. W. Cline, D. G. Cunningham, A. V. Fioravanti, J. R. Lucas II, C. W. Ponsonby, L. E. Povirk, D. Shafferman and R. J. Wargo.

The overall direction and coordination of the technical measurements leading to certification were performed jointly by R. E. Michaelis, Office of Standard Reference Materials and by J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

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.

U.S. Department of Commerce Juanita M. Kreps Secretary

National Bureau of Standard

analyses.

National Bureau of Standards Certificate of Analysis

Standard Reference Material 696

Bauxite (Surinam)

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 140 °C) This material is in the form of fine powder (<0.08 mm) for use in checking chemical and instrumental methods of

Constituent	Certified Value ¹ Percent, by weight	Estimated Uncertainty ²
Al ₂ O ₃	54.5	0.3
Fe ₂ O ₃	8.70	.10
SiO ₂	3.79	.10
TiO ₂	2.64	.05
ZrO ₂	0.14	.02
P ₂ O ₅	.050	.006
V_2O_5	.072	.006
Cr ₂ O ₃	.047	.003
CaO	.018	.002
MgO	.012	.003
MnO	.004	.001
ZnO	.0014	.0007
K ₂ O	.009	.003
SO ₃	.21	.03
Loss on Ignition ³	29.9	.2

¹The certified value listed for a constituent is the present best estimate of the "true" value.

³Determined by igniting to constant weight at 1050 °C.

Washington, D.C. 20234 August 24, 1979

George A. Uriano, Chief Office of Standard Reference Materials

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 variability for samples 1.0 gor more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

ADDITIONAL INFORMATION ON THE COMPOSITION

Elements other than those certified may be present in this material as indicated below. These are not certified but are given as additional information on the composition.

Constituent	Concentration, Percent by weight	Constituent	Concentration, Percent by weight
BaO	(0.004)	Co	(0.00009)
Na ₂ O	(0.007)	Hf	(0.0032)
Ce	(0.0041)	Sc	(0.0008)

The mineralogical composition of SRM 696 was determined by x-ray diffraction studies at the Geological Survey, U.S. Department of the Interior, Reston, Va., (J.W. Hosterman) to be 5% kaolinite, 80% gibbsite, 10% pyrite, and 5% anatase. These results are semiquantitative (to the nearest 5%).

PLANNING, PREPARATION, TESTING, ANALYSIS:

The material for this SRM was mined in Surinam, South America, and was provided by the Aluminum Company of America, Alcoa Technical Center, Pittsburgh, Pa., through the courtesy of H. B. Hartman. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute under a contract with the National Bureau of Standards.

Homogeneity testing was performed at NBS by J.S. Maples and T.E. Gills.

Cooperative analyses for certification were performed in the following laboratories:

Aluminum Company of America, Alcoa Center, Pa., R. C. Obbink.

Aluminum Company of Canada, Ltd., Arvida Research Center, Arvida, Quebec, Canada, L. Girolami.

Andrew S. McCreath & Son, Inc., Harrisburg, Pa., F. A. Pennington, Jr., R. F. Eakin, and S. L. Miller.

General Refractories Co., U.S. Refractories Division, Research Center, Baltimore, Md., S. Banerjee. Geological Survey, U.S. Department of the Interior, Reston, Va., H. J. Rose, Jr., and J. W. Hosterman.

Kalser Aluminum and Chemical Corp., Center for Technology, Pleasanton, Calif., H. J. Seim, A. E. McLaughlin, D. F. G. Marten, A. Kermaninejad, R. C. Kinne, J. R. Skarset, J. Boruk, and U. Vogel.

National Bureau of Standards, Washington, D.C., R. K. Bell, ASTM-NBS Assistant Research Associate.

National-Southwire Aluminum Co., Hawesville, Ky., N. Robinson and E. Gotzy.

Ormet Corp., Burnside, La., W. L. Brown and A. D. Lafleur.

Reynolds Aluminum Co., Alumina Research Division, Bauxite, Ark., J. B. Ezell, Jr.

University of Kentucky, Institute for Mining and Minerals Research, Center for Energy Research Laboratory, Lexington, Ky., T. V. Rebagay.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

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. E. Michaelis and R. Alvarez.

NBS Standard Reference Materials BAUXITE SERIES September 4, 1979

R. E. Michaelis and R. Alvarez, NBS Office of Standard Reference Materials and

J. I. Shultz, ASTM Research Associate

The following table gives the values for four bauxite SRM's that are available in the form of fine powder (<0.08 mm) for use in chemical and instrumental methods of analysis. They are being issued as a culmination of a major Industry-ASTM-NBS cooperative program.

SRM No. Designation	69b Arkansas	696 Surinam	697 Dominican	698 Jamaican
Constituent		Percent b	y Weight	
Al ₂ O ₃	48.8	54.5	45.8	48.2
Fe ₂ O ₃	7.14	8.70	20.0	19.6
SiO ₂	13.43	3.79	6.81	0.69
TiO ₂	1.90	2.64	2.52	2.38
ZrO ₂	0.29	0.14	0.065	0.061
P ₂ O ₅	0.118	0.050	0.97	0.37
V ₂ O ₅	0.028	0.072	0.063	0.064
Cr ₂ O ₃	0.011	0.047	0.100	0.080
CaO	0.13	0.018	0.71	0.62
MgO	0.085	0.012	0.18	0.058
MnO	0.110	0.004	0.41	0.38
ZnO	0.0035	0.0014	0.037	0.029
BaO	$(0.008)^a$	(0.004)	(0.015)	(0.008)
Na ₂ O	(0.025)	(0.007)	(0.036)	(0.015)
K ₂ O	0.068	0.009	0.062	0.010
SO ₃	0.63	0.21	0.13	0.22
Loss on Ign.	27.2	29.9	22.1	27.3
Ce	(0.024)	(0.0041)	(0.069)	(0.030)
Co	(0.0001)	(0.00009)	(0.0013)	(0.0045)
Hf	(0.0063)	(0.0032)	(0.0014)	(0.0015)
Sc	(0.0008)	(0.0008)	(0.0058)	(0.0051)
Total	(100.0)	(100.1)	(100.1)	(100.1)

Values in parenthesis are not certified.

The value listed for a certified constituent is the present best estimate of the "true" value based on the results of the analytical program for certification (10-12 laboratories). The individual certificates of analysis list the "estimated uncertainties" associated with the certified values (also listed is a semiquantitative mineralogical composition (±5%) as determined by x-ray diffraction studies at the U.S. Geological Survey).

Inquiries regarding the Bauxite SRM's 69b, 696, 697, and 698, should be directed to the Office of Standard Reference Materials, Chemistry Building, B311, National Bureau of Standards, Washington, D.C. 20234. (301) 921-2045.

George A. Uriano, Chief Office of Standard Reference Materials U.S. Department of Commerce Juanita M. Kreps Secretary National Bureaur of Standards Ernest Ambler, Director

National Bureau of Standards Certificate of Analysis

Standard Reference Material 697

Bauxite (Dominican)

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 140 °C) This material is in the form of fine powder ($<0.08\,\mathrm{mm}$) for use in checking chemical and instrumental methods of analyses.

Constituent	Certified Value ¹ Percent, by weight	Estimated Uncertainty ²
Al ₂ O ₃	45.8	0.2
Fe ₂ O ₃	20.0	.2
SiO ₂	6.81	.07
TiO ₂	2.52	.05
ZrO ₂	0.065	.007
P_2O_5	.97	.06
V_2O_5	.063	.005
Cr ₂ O ₃	.100	.005
CaO	.71	.03
MgO	.18	.02
MnO	.41	.03
ZnO	.037	.003
K ₂ O	.062	.007
SO ₃	.13	.03
Loss on Ignition ³	22.1	.2

¹The certified value listed for a constituent is the present best estimate of the "true" value.

Washington, D.C. 20234

August 24, 1979

George A. Uriano, Chief Office of Standard Reference Materials

²The 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 variability for samples 1.0 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

³Determined by igniting to constant weight at 1050 °C.

ADDITIONAL INFORMATION ON THE COMPOSITION

Elements other than those certified may be present in this material as indicated below. These are not certified but are given as additional information on the composition.

Constituent	Concentration, Percent by weight	Constituent	Concentration, Percent by weight
BaO	(0.015)	Co	(0.0013)
Na ₂ O	(0.036)	Hf	(0.0014)
Ce	(0.069)	Sc	(0.0058)

The mineralogical composition of SRM 697 was determined by x-ray diffraction studies at the Geological Survey, U.S. Department of the Interior, Reston, Va., (J.W. Hosterman) to be 15% kaolinite, 50% gibbsite, 10% boehmite, 20% hematite, and 5% anatase. These results are semiquantitative (to the nearest 5%).

PLANNING, PREPARATION, TESTING, ANALYSIS:

The material for this SRM was mined in the Dominican Republic and was provided by the Aluminum Company of America, Alcoa Technical Center, Pittsburgh, Pa., through the courtesy of H. B. Hartman. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute under a contract with the National Bureau of Standards.

Homogeneity testing was performed at NBS by J.S. Maples and T.E. Gills.

Cooperative analyses for certification were performed in the following laboratories:

Aluminum Company of America, Alcoa Center, Pa., R. C. Obbink.

Aluminum Company of Canada, Ltd., Arvida Research Center, Arvida, Quebec, Canada, L. Girolami. Andrew S. McCreath & Son, Inc., Harrisburg, Pa., F. A. Pennington, Jr., R. F. Eakin, and S. L. Miller.

General Refractories Co., U.S. Refractories Division, Research Center, Baltimore, Md., S. Banerjee.

Geological Survey, U.S. Department of the Interior, Reston, Va., H. J. Rose, Jr., and J. W. Hosterman. Kaiser Aluminum and Chemical Corp., Center for Technology, Pleasanton, Calif., H. J. Seim, A. E. McLaughlin, D. F. G. Marten, A. Kermaninejad, R. C. Kinne, J. R. Skarset, J. Boruk, and U. Vogel.

National Bureau of Standards, Washington, D.C., R. K. Bell, ASTM-NBS Assistant Research Associate.

National-Southwire Aluminum Co., Hawesville, Ky., N. Robinson and E. Gotzy.

Ormet Corp., Burnside, La., W. L. Brown and A. D. Lafleur.

Reynolds Aluminum Co., Alumina Research Division, Bauxite, Ark., J. B. Ezell, Jr.

University of Kentucky, Institute for Mining and Minerals Research, Center for Energy Research Laboratory, Lexington, Ky., T. V. Rebagay.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

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. E. Michaelis and R. Alvarez.

NBS Standard Reference Materials BAUXITE SERIES September 4, 1979

R. E. Michaelis and R. Alvarez, NBS Office of Standard Reference Materials

and J. I. Shultz, ASTM Research Associate

The following table gives the values for four bauxite SRM's that are available in the form of fine powder (<0.08 mm) for use in chemical and instrumental methods of analysis. They are being issued as a culmination of a major Industry-ASTM-NBS cooperative program.

SRM No.	69b	696	697	698
Designation	Arkansas	Surinam	Dominican	Jamaican
Constituent		Percent b	y Weight	
Al ₂ O ₃	48.8	54.5	45.8	48.2
Fe ₂ O ₃	7.14	8.70	20.0	19.6
SiO ₂	13.43	3.79	6.81	0.69
TiO ₂	1.90	2.64	2.52	2.38
ZrO ₂	0.29	0.14	0.065	0.061
P_2O_5	0.118	0.050	0.97	0.37
V_2O_5	0.028	0.072	0.063	0.064
Cr ₂ O ₃	0.011	0.047	0.100	0.080
CaO	0.13	0.018	0.71	0.62
MgO	0.085	0.012	0.18	0.058
MnO	0.110	0.004	0.41	0.38
ZnO	0.0035	0.0014	0.037	0.029
BaO	$(0.008)^{a}$	(0.004)	(0.015)	(0.008)
Na ₂ O	(0.025)	(0.007)	(0.036)	(0.015)
K ₂ O	0.068	0.009	0.062	0.010
SO ₃	0.63	0.21	0.13	0.22
Loss on Ign.	27.2	29.9	22.1	27.3
Ce a	(0.024)	(0.0041)	(0.069)	(0.030)
Co	(0.0001)	(0.00009)	(0.0013)	(0.0045)
Hf	(0.0063)	(0.0032)	(0.0014)	(0.0015)
Sc	(0.0008)	(0.0008)	(0.0058)	(0.0051)
Total	(100.0)	(100.1)	(100.1)	(100.1)

^aValues in parenthesis are not certified.

The value listed for a certified constituent is the present best estimate of the "true" value based on the results of the analytical program for certification (10-12 laboratories). The individual certificates of analysis list the "estimated uncertainties" associated with the certified values (also listed is a semiquantitative mineralogical composition (±5%) as determined by x-ray diffraction studies at the U.S. Geological Survey).

Inquiries regarding the Bauxite SRM's 69b, 696, 697, and 698, should be directed to the Office of Standard Reference Materials, Chemistry Building, B311, National Bureau of Standards, Washington, D.C. 20234. (301) 921-2045.

George A. Uriano, Chief Office of Standard Reference Materials U.S. Desartment of Commerce Juanita M. Kreps Secretary National Surem of Standards

National Bureau of Standards Certificate of Analysis

Standard Reference Material 698

Bauxite (Jamaican)

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 140 °C) This material is in the form of fine powder (<0.08 mm) for use in checking chemical and instrumental methods of analyses.

Constituent	Certified Value ¹ Percent, by weight	Estimated Uncertainty ²
Al ₂ O ₃	48.2	0.4
Fe ₂ O ₃	19.6	.2
SiO ₂	0.69	.03
TiO ₂	2.38	.07
ZrO ₂	0.061	.009
P_2O_5	.37	.01
V_2O_5	.064	.005
Cr ₂ O ₃	.080	.006
CaO	.62	.02
MgO	.058	.008
MnO	.38	.03
ZnO	.029	.002
K₂O	.010	.002
SO ₃	.22	.03
Loss on Ignition ³	27.3	.2

¹The certified value listed for a constituent is the present best estimate of the "true" value.

Washington, D.C. 20234

August 24, 1979

George A. Uriano, Chief Office of Standard Reference Materials

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 variability for samples 1.0 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

3 Determined by igniting to constant weight at 1050 °C.

ADDITIONAL INFORMATION ON THE COMPOSITION

Elements other than those certified may be present in this material as indicated below. These are not certified but are given as additional information on the composition.

Constituent	Concentration, Percent by weight	Constituent	Concentration, Percent by weight
BaO	(0.008)	Co	(0.0045)
Na ₂ O	(0.015)	Hf	(0.0015)
Ce	(0.030)	Sc	(0.0051)

The mineralogical composition of SRM 698 was determined by x-ray diffraction studies at the Geological Survey, U.S. Department of the Interior, Reston, Va., (J.W. Hosterman) to be 75% gibbsite, 20% hematite, and 5% anatase. These results are semiquantitative (to the nearest 5%).

PLANNING, PREPARATION, TESTING, ANALYSIS:

The material for this SRM was mined in Jamaica, and was provided by the Reynolds Metals Company, Bauxite, Arkansas, through the courtesy of J. B. Ezell, Jr. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute under a contract with the National Bureau of Standards.

Homogeneity testing was performed at NBS by J.S. Maples and T.E. Gills.

Cooperative analyses for certification were performed in the following laboratories:

Aluminum Company of America, Alcoa Center, Pa., R. C. Obbink.

Aluminum Company of Canada, Ltd., Arvida Research Center, Arvida, Quebec, Canada, L. Girolami.

Andrew S. McCreath & Son, Inc., Harrisburg, Pa., F. A. Pennington, Jr., R. F. Fakin, and S. L. Miller.

General Refractories Co., U.S. Refractories Division, Research Center, Baltimore, Md., S. Banerjee. Geological Survey, U.S. Department of the Interior, Reston, Va., H. J. Rose, Jr., and J. W. Hosterman.

Kaiser Aluminum and Chemical Corp., Center for Technology, Pleasanton, Calif., H. J. Seim, A. E. McLaughlin, D. F. G. Marten, A. Kermaninejad, R. C. Kinne, J. R. Skarset, J. Boruk, and U. Vogel. National Bureau of Standards, Washington, D.C., R. K. Bell, ASTM-NBS Assistant Research Associate.

National-Southwire Aluminum Co., Hawesville, Ky., N. Robinson and E. Gotzy.

Ormet Corp., Burnside, La., W. L. Brown and A. D. Lafleur.

Reynolds Aluminum Co., Alumina Research Division, Bauxite, Ark., J. B. Ezell, Jr.

University of Kentucky, Institute for Mining and Minerals Research, Center for Energy Research Laboratory, Lexington, Ky., T. V. Rebagay.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

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. E. Michaelis and R. Alvarez.

NBS Standard Reference Materials BAUXITE SERIES September 4, 1979

R. E. Michaelis and R. Alvarez, NBS Office of Standard Reference Materials and

J. I. Shultz, ASTM Research Associate

The following table gives the values for four bauxite SRM's that are available in the form of fine powder (<0.08 mm) for use in chemical and instrumental methods of analysis. They are being issued as a culmination of a major Industry-ASTM-NBS cooperative program.

SRM No. Designation	69b Arkansas	696 Surinam	697 Dominican	698 Jamaican
Constituent		Percent b	y Weight	
Al ₂ O ₃	48.8	54.5	45.8	48.2
Fe ₂ O ₃	7.14	8.70	20.0	19.6
SiO ₂	13.43	3.79	6.81	0.69
TiO ₂	1.90	2.64	2.52	2.38
ZrO ₂	0.29	0.14	0.065	0.061
P ₂ O ₅	0.118	0.050	0.97	0.37
V ₂ O ₅	0.028	0.072	0.063	0.064
Cr ₂ O ₃	0.011	0.047	0.100	0.080
CaO	0.13	0.018	0.71	0.62
MgO	0.085	0.012	0.18	0.058
MnO	0.110	0.004	0.41	0.38
ZnO	0.0035	0.0014	0.037	0.029
BaO	(0.008) ^a	(0.004)	(0.015)	(0.008)
Na ₂ O	(0.025)	(0.007)	(0.036)	(0.015)
K ₂ O	0.068	0.009	0.062	0.010
SO ₃	0.63	0.21	0.13	0.22
Loss on Ign.	27.2	29.9	22.1	27.3
Се	(0.024)	(0.0041)	(0.069)	(0.030)
Co	(0.0001)	(0.00009)	(0.0013)	(0.0045)
Hf	(0.0063)	(0.0032)	(0.0014)	(0.0015)
Sc	(0.0008)	(0.0008)	(0.0058)	(0.0051)
Total	(100.0)	(100.1)	(100.1)	(100.1)

^aValues in parenthesis are not certified.

The value listed for a certified constituent is the present best estimate of the "true" value based on the results of the analytical program for certification (10-12 laboratories). The individual certificates of analysis list the "estimated uncertainties" associated with the certified values (also listed is a semiquantitative mineralogical composition (±5%) as determined by x-ray diffraction studies at the U.S. Geological Survey).

Inquiries regarding the Bauxite SRM's 69b, 696, 697, and 698, should be directed to the Office of Standard Reference Materials, Chemistry Building, B311, National Bureau of Standards, Washington, D.C. 20234. (301) 921-2045.

George A. Uriano, Chief Office of Standard Reference Materials



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).

Certified Values of Constituent Elements: 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).

Calorific Value: 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

Table 1. Certified Values of Constituent Elements

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

Trace Constituents

Element	Content μg/g	Element	Content µg/g
Arsenic	3.72 ±0.09	Manganese	12.4 ± 1.0
Barium	67.5 ± 2.1	Nickel	6.10 ±0.27
Cadmium	0.0573 ± 0.0027	Rubidium	5.05 ± 0.11
Cobalt	2.29 ± 0.17	Selenium	1.29 ±0.11
Copper	6.28 ±0.30	Thorium	1.342 ± 0.036
Lead	3.67 ± 0.26	Uranium	0.436 ± 0.012
		Zinc	11.89 ±0.78

Calorific Valueb, c	Ash, wt.%
$14005 \pm 35 \text{ Btu/lb} (32.57 \pm 0.08 \text{ MJ kg}^{-1})$	6.79 ± 0.16

a The listed ± uncertainties for carbon, hydrogen, volatile matter, and calorific value are two standard deviations of the certified value. The listed ± 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.

b The calorific value (M lkg⁻¹) 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.

The calorific value is determined as HHV2 (Hieber Heating Value-Moisture Free)

Table 2. Noncertified Values for Constituent Elements

	Trace Co	nstituents	
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

^cThe calorific value is determined as HHV2 (Higher Heating Value-Moisture Free).

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

						1					Γ		
Method/ Element	A	В	С	D	E	F	G	н	1	J	ĸ	L	М
Al			•	•								•	
As			•		•								
Ash Content							• 2		•				
Ba			•										
Br			•										
C (Total)							• 5	•	•		•		
Ca		•	•	•								•	
Cal Val									•	•			
Cd	•	•				1							
Ce			•										
Cl			•					T					
Со			•	•							1		
Cr			. •			t	l	 					•
Cs			•										
Cu				<u> </u>	•							1	•
Eu			•										
Fe	•		•	·	 	1						•	
Н					l	1	• 5	•	•			1	
Hf			•		†	1-		<u> </u>			<u> </u>		
K		•	•	•	 	 		†	\vdash			•	
La		<u> </u>	•	H	1	 		 			 	<u> </u>	!
Li				•	 	 		<u> </u>			\vdash	<u> </u>	
Mg	•	•	•		1	+	 	 		\vdash		 	<u> </u>
Mn						+	 	 	 	l	 	 	\vdash
Mo			•	+-	 	+	\vdash	1	+-		+	 	\vdash
		-	<u> </u>	 -	 	┼─	• 6	 	 	 		+	
N		-		•	┼	┼	100	 	├	├	+-	 	
Na			<u> </u>	+-	 	+		 	├	-	 	┼	+-
Ni	•	-		┼	┼	┼		 	 	-	 		•
Pb	•	•	 	1.	-	┼	ļ	 	-	ļ			
Rb		•	•	•	 		 	 	<u> </u>		 		<u> </u>
S	<u> </u>		 	 	┼	 •	• 1		<u> • </u>	 	┼	•	
Sb			•	 	ऻ	-	 	 	<u> </u>	ļ	 		ļ
Sc		_	•	ļ	ļ	-	ļ	-	├	ļ	ļ		
Se			•	ļ	ļ:		<u> </u>	<u> </u>	<u> </u>	ļ	<u> </u>	<u> </u>	ļ
Si	<u> </u>	ļ	•	ļ	1	↓	<u> </u>	-	 	-	ļ.,	·	ļ
Sm			•	↓	ļ	↓	<u> </u>	 		<u> </u>	ļ	1	
Sr			•	1		1	ļ	<u> </u>	1	<u> </u>			<u> </u>
Th	İ	•	•	1	ļ	1	<u> </u>	1	1	ļ	I	<u> </u>	L_
Ti			•	•			<u> </u>	L	<u> </u>	<u> </u>		•	•
U		•	•										
v			•										•
Volatile Matter							• 3		•				
w	i		•						1				1

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
- Commercial coal analyzers: moisture, ash, sulfur, Btu, volatile matter, carbon, hydrogen, nitrogen
- Commercial calorimeter
- K. Gas chromatography
- 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

R. Peck

Dickerson Laboratory El Paso, Texas 79912

J.B. Bodkin

College Earth & Mineral Science The Pennsylvania State University

University Park, Pa. 16802

Page 5 SRM 1632b

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

Trace Elements in Coal Fly Ash

This Standard Reference Material is intended for use in the calibration of apparatus and methods used in analyses of coal fly ash and other materials with similar matrices for trace elements. This material should be dried to a constant weight before using. Recommended procedures for drying are: (1) 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 in a desiccator over P2O5 or Mg(C104)2. When not in use, the material should be kept in a tightly sealed bottle. Long term (>3 years) 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 ¹	Content ²	Element	Content
Calcium ^{a, b, c} Iron ^{a, b, c} Potassium ^{a, b, c} Magnesium ^{a, b} Sodium ^{a, c}	$ \frac{\%}{1.11 \pm 0.01} 9.40 \pm 0.10 1.88 \pm 0.06 0.455 \pm 0.010 0.17 \pm 0.01 $	Mercury ^{a, c} Nickel ^{a, b, d, e} Lead ^{b,d, e} Rubidium ^{a, b, c, e} Selenium ^a	$ \begin{array}{cccc} $
Silicon ^{c,h} Arsenic ^{a,c} Cadmium ^{b,c,d,g} Chromium ^{a,b,c} Copper ^{a,b,c}	22.8 \pm 0.8 $\mu g/g$ 145 \pm 15 1.0 \pm 0.15 196 \pm 6 118 \pm 3	Strontium ^{b, c} Thorium ^{b, c} Thallium ^{b, g} Uranium ^b Zinc ^{a, b, d, e, f}	830 ± 30 24.7 ± 0.3 5.7 ± 0.2 10.2 ± 0.1 220 ± 10

1. Methods of Analysis:

^aAtomic Absorption Spectrophotometry or Flame Emission

Spectrometry

Bisotope Dilution Mass Spectrometry

Neutron Activation

^dPolarography

X-ray Fluorescence Spectrometry

Inductively Coupled Plasma Emission Spectrometry Isotope Dilution Spark Source Mass Spectrometry

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.)

Washington, D.C. 20234 April 18, 1979

George A. Uriano, Chief Office of Standard Reference Materials

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

PREPARATION, TESTING, AND ANALYSIS

This 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 through a # 170 sieve 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.

Twelve bottles were selected for homogeneity tests. These samples were analyzed for cobalt, chromium, europium, iron, scandium, and thorium by nondestructive neutron activation analysis. The observed standard deviations for both 50 and 250 mg samples were consistent with counting statistics indicating that the fly ash is homogeneous within ± 5% (relative) based on these elements. The homogeneity analyses were performed in the NBS Center for Analytical Chemistry by R. R. Greenburg and J. S. Maples. Analyses for the various elements were made in the NBS Center for Analytical Chemistry by the following analysts: J. R. Baldwin, T. J. Brady, E. R. Deardorff, M. G. Dias, L. P. Dunstan, M. S. Epstein, E. L. Garner, T. E. Gills, C. A. Grabnegger, J. W. Gramlich, R. R. Greenberg, S. Hanamura, S. H. Harrison, E. F. Heald, H. M. Kingston, E. C. Kuehner, L. A. Machlan, E. J. Maienthal, J. S. Maples, J. D. Messman, L. J. Moore, P. J. Paulsen, P. A. Pella, T. C. Rains, K. J. R. Rosman, T. A. Rush, P. A. Sleeth, and R. L. Waters, Jr.

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

Element	Content	Element	Content
	<u>%</u>		μg/g
Aluminum	14	Europium	4
Barium	0.15	Gallium	58
Titanium	0.8	Hafnium	7.6
	$\mu g/g$	Manganese	190
Beryllium	12	Molybdenum	29
Cerium	180	Antimony	7
Cobalt	46	Scandium	40
Cesium	11	Vanadium	300

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 1	Content, μg/g ²	Element 1	Content, $\mu g/g^2$
Arsenic ^{a,b}	0.42 ± 0.15	Thorium ^{c, e}	0.62 ± 0.04
Cadmium ^{c, d, e}	0.03 ± 0.01	Uranium ^c	0.24 ± 0.02
Chromium ^{c, e}	2.5 ± 0.3	Vanadium ^{e, g}	5.2 ± 0.5
Copper a, c, e	3.6 ± 0.3	Zinc ^{c, d}	4.7 ± 0.5
Lead ^{c,d}	1.9 ± 0.2		
Manganese*,*	21.4 ± 1.5	Element ¹	Wt. % 2
Nickel ^{c, d}	1.74 ± 0.10	Iron ^{c, d, e, f}	0.239 ± 0.005
Selenium ^{a, e}	0.9 ± 0.3	Sulfur ^{f, h}	0.33 ± 0.03

- 1. Methods of Analysis:
 - a. Atomic Absorption Spectrometry
- b. Photon Activation
- c. Isotope Dilution Mass Spectrometry d. Polarography
- ·v
- e. Neutron Activation f. Spectrophotometry
- g. Flame Emission Spectrometry 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 μ 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, or were not determined by two or more independent methods. They are included for information only.

	Content
Element	(μg/g)
Antimony	(0.14)
Cerium	(3.6)
Cobalt	(0.65)
Europium	(0.06)
Gallium	(1.05)
Hafnium	(0.29)
Scandium	(0.63)
	(wt. %)
Aluminum	(0.32)
Sodium	(0.24)
Titanium	(0.02)

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

National Bureau of Standards

Certificate of Analysis

Standard Reference Materials 2682, 2683, 2684 and 2685

Sulfur in Coal

These Standard Reference Materials (SRM's) are intended primarily for use as analytical standards for the determination of sulfur in coal. In addition to sulfur they are certified for their calorific value (MJ·Kg⁻¹) and ash content. SRM's 2682-2685 each consists of a 50-g bottle of a different coal composition. Each material was ground to pass a 60-mesh sieve and homogenized. The certification of the materials for sulfur is based on at least a 250-mg sample of the dried material, the minimum amount that should be used for analysis (see drying instructions). The calorific values were determined by procedures recommended in standard ASTM methods (see references in Table 1). The certification data for the four different coals are given in Table 1 along with methods used for certification. Noncertified values for major and minor elements are given in Table 2. These values are provided for information only.

Notice to Users: These SRM's are sold individually rather than in sets; however, only one Certificate of Analysis is provided. Therefore, the user must be careful to use the data specific to the SRM being used.

The uncertainty of a certified value is expressed as two times the standard error and includes observed variability within and between measurement methods and any observed material heterogeneity. For the certified heating values the uncertainty also includes additional allowances for possible limited sample degradation due to aging or normal oxidation.

Certification analyses were performed by W.R. Kelly, W.F. Koch, P.J. Paulsen, and J.W. Stolz of the Inorganic Analytical Research Division and J.C. Colbert and D.R. Kirklin of the Chemical Thermodynamics Division.

Analyses for supplemental information were performed in the Inorganic Analytical Research Division by R. Fleming, R. Greenberg, and R.M.Lindstrom.

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 these Standard Reference Materials were coordinated through the Office of Standard Reference Materials by T.E. Gills.

February 7, 1983 (Revision of Certificate dated 12-14-82)

(over)

George A. Uriano, Chief Office of Standard Reference Materials

Table 1 Certified Values for SRM's 2682, 2683, 2684, and 2685

SRM No.	Coal Type	Sulfur ^{1,2,3} Wt. %	Furnace ⁴ Ash Wt. %	HHV2 ^{5, 6} MJ·Kg ⁻¹ (BTU·lb ⁻¹)
2682	Sub-bituminous	0.47 ± 0.03	6.37 ± 0.18	27.45 ± 0.56 (11800 ± 240)
2683	Bituminous	1.85 ± 0.06	6.85 ± 0.02	32.70 ± 0.14 (14060 ± 60)
2684	Bituminous	3.00 ± 0.13	11.09 ± 0.18	29.68 ± 0.47 (12760 ± 200)
2685	Bituminous	4.62 ± 0.18	16.53 ± 0.15	28.15±0.42(12100±180)

ASTM D3177 Standard Test Method for Total Sulfur in the Analysis Sample of Coal and Coke

HHV2-Higher Heating Value - Moisture Free)

Note: The cultorific values (MJ-Kg⁻¹) may decrease upon the aging or normal exidation of the coals. NBS will continue to monitor these calcrific values and report any substantive change to the purchaser. The uncertainty of the heating value includes an additional allowance of 0.12 MJ·Kg⁻¹ for possible sample degradation. The reference date for the calorific data is October 1982.

PREPARATION AND TESTING

Approximately one ton of coal was obtained from each of four different coal mine locations. All coals were oven dried prior to processing in accordance with procedures outlined in ASTM D2013. At least 1000 pounds of each of the four coals were reduced in size to -60 mesh and screened prior to blending. Each of the -60 mesh coals was blended in a stainless steel cone blender (approximate capacity 0.85 cubic meter). The coals were then packaged into individual 50-g bottled units. Homogeneity testing was done on the bulk materials and 50-g bottled units. The homogeneity analyses were performed using x-ray fluorescence analysis. Replicate analyses indicated the material variability for sulfur to be within \pm 2% (relative) for all four SRM's.

The homogeneity studies were performed by T.E. Gills and M. Watson of the Office of Standard Reference Materials and P.A. Pella of the Gas and Particulate Science Division.

²Ion Chromatography with Bomb Combustion

³Thermal Ionization Mass Spectrometry, Sealed Glass Tube Digestion

ASTM D3174, Standard Test Method for Ash in the Analysis Sample of Coal and Coke

SASTM D2015 Standard Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Calorimeter

ASTM D3180 Standard Test Method for Calculating Coal and Coke Analyses from As-Determined to Different Bases

ANALYSIS

Sulfur: The certified sulfur content is based upon the results of 3 independent methods of analysis: ion chromatography, gravimetry, and thermal ionization mass spectrometry. Agreement with the certified values was found using 2 additional independent techniques, prompt-gamma activation analysis and a combustion IR technique.

Calorific Value (MJ·Kg⁻¹) and Ash Content: The certified values for the calorific values and ash contents were determined using measurements made in an adiabatic bomb calorimeter of the type used in commercial laboratories. This calorimeter is capable of reproducing determinations on benzoic acid to a precision of 0.07% (relative). This statement of precision was arrived at by averaging 5 calibrations made on the calorimeter using a benzoic acid standard that is traceable to the NBS SRM 39i, Benzoic Acid.

Major and Minor Elements: Analyses for major and minor elements were performed by thermal neutron activation analysis and neutron capture gamma-ray activation analysis. These values are not certified but are to be used for information only.

STABILITY

The long-term physical and chemical stability of these SRM's has not been rigorously established. However, NBS recommends that the material be stored in the tightly sealed bottle away from sunlight and intense sources of radiation. NBS will continue to monitor these materials and any substantive change in their certification will be reported to the purchaser.

INSTRUCTIONS FOR DRYING

The certification of sulfur in these SRM's is based upon a properly dried sample. The recommended procedures for drying are vacuum drying at ambient temperature for 24 hours or oven drying for 24 hours at 105° C. Typical moisture loss using the recommended methods for drying are the following: SRM 2682, 18%; SRM 2683, 1.4%; SRM 2684, 3.6%; and SRM 2685, 1.8%.

SUPPLEMENTAL INFORMATION

The values listed below are based on measurements made using a single method or technique and are given for information only. While no reason exists to suspect systematic bias in these numbers, no attempt was made to determine if such bias attributable to the methods exists.

The analyses of SRM's 2682-2685 for major and minor elements were made using NBS SRM's 1632a and 1635. Trace Elements in Coal, as controls.

Table 2 Inorganic Constituents in SRM's 2682, 2683, 2684, and 2685 Mean Concentrations ($\mu g/g$) Unless Noted

Element/SRM	2682	2683	2684	2685
Al %	0.46	0.86	1.1	1.7
As	1.0	3.6	3.9	12
В	39	67	114	109
Ba	382	71	41	105
Br	3.7	17	11	5.6
С%	75	79	68	66
Ca %	1.1	0.20	0.44	0.52
Ce	10	9	12	18
Co	1.7	2.2	3.9	4.6
Cr	15	11	17	22
Cs	<0.1	0.4	1.2	1.3
Fu	0.17	0.18	0.23	0.36
Fe %	0.24	0.76	1.5	2.9
Н %	4.7	5.0	4.8	4.6
Hſ	0.60	0.42	0.57	0.91
К %	0.01	0.08	0.20	0.26
La	5.2	5.1	6.7	10
Mg %	0.2	0.05	0.08	0.1
Mn	26	13	36	41
N %	0.8	1.6	1.6	1.1
Na %	0.10	0.05	0.03	0.08
Rb	<2	5.3	15	17
Sb	0.19	0.28	0.35	0.36
Sc	1.5	1.9	2.7	3.7
Se	0.91	1.2	1.9	1.9
Sm	0.78	0.86	1.1	1.7
Th	1.5	1.4	2.0	2.7
Ti %	0.05	0.04	0.06	0.09
U	0.52	0.42	0.90	0.95
v	15	14	22	31
W	1.8	0.48	0.56	1.2
Zn	8.6	9.5	110	17