NBS SPECIAL PUBLICATION 260-75

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

Standard Reference Materials:

Preparation and Calibration of First-Surface Aluminum Mirror Specular Reflectance Standards

NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards[1] was established by an act of Congress on March 3, 1901. The Bureau's overall goal is to strengthen and advance the Nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the Nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, and the Institute for Computer Sciences and Technology.

THE NATIONAL MEASUREMENT LABORATORY provides the national system of physical and chemical and materials measurement; coordinates the system with measurement systems of other nations and furnishes essential services leading to accurate and uniform physical and chemical measurement throughout the Nation's scientific community, industry, and commerce; conducts materials research leading to improved methods of measurement, standards, and data on the properties of materials needed by industry, commerce, educational institutions, and Government; provides advisory and research services to other Government agencies; develops, produces, and distributes Standard Reference Materials; and provides calibration services. The Laboratory consists of the following centers:

Absolute Physical Quantities[2] — Radiation Research — Chemical Physics — Analytical Chemistry — Materials Science

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

Applied Mathematics — Electronics and Electrical Engineering[2] — Manufacturing Engineering — Building Technology — Fire Research — Chemical Engineering[2]

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

Programming Science and Technology — Computer Systems Engineering.

[1]Headquarters and Laboratories at Gaithersburg, MD, unless otherwise noted;
mailing address Washington, DC 20234.
[2]Some divisions within the center are located at Boulder, CO 80303.

Standard Reference Materials:

Preparation and Calibration of First-Surface Aluminum Mirror Specular Reflectance Standards

Victor R. Weidner
Jack J. Hsia

Radiometric Physics Division
Center for Radiation Research
National Bureau of Standards
Washington, DC 20234

U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, Secretary

NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Director

Issued May 1982

Library of Congress Catalog Card Number: 82-600542

National Bureau of Standards Special Publication 260-75
Nat. Bur. Stand. (U.S.), Spec. Publ. 260-75, 26 pages (May 1982)
CODEN: XNBSAV

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 1982

For Sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.
Price
(Add 25 percent for other than U.S. mailing.)

PREFACE

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards 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 are carried out through the mechanism and use of SRM's. For many of the Nation's scientists and technologists it is therefore of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values of the SRM's produced. An NBS series of papers, of which this publication is a member, called the 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 measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing the greatest care and accuracy. These papers also should provide sufficient additional information not found on the certificate so that new applications in diverse fields not foreseen at the time the SRM was originally issued will be sought and found.

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

Office of Standard Reference Materials
National Bureau of Standards
Washington, D.C. 20234

George A. Uriano, Chief
Office of Standard Reference Materials

OTHER NBS PUBLICATIONS IN THIS SERIES

Catalog of NBS Standard Reference Materials (1981-83 edition), R. W. Seward, ed., NBS Spec. Publ. 260 (November 1981).

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 1975). 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 on 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 Corportion 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**

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* SN003-003-01425-7

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* 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, G. 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 01978-0

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*

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*

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*

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).

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 (in press).

Weidner, V. R., Hsia, J. J., Standard Reference Materials: Preparation and Calibration of First Surface Aluminum Mirror Specular Reflectance Standards (SRM 2003a), NBS Spec. Publ. 260-75.

* 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 22151.

TABLE OF CONTENTS

Preparation and Calibration of First-Surface Aluminum Mirror Specular Reflectance Standards (Standard Reference Material 2003a)

Victor R. Weidner and Jack J. Hsia

Radiometric Physics Division
Center for Radiation Research
National Bureau of Standards
Washington, D. C. 20234

A number of first-surface aluminum mirrors of high optical quality have been prepared and calibrated for use as specular reflectance standards over the wavelength range 250 to 2500 nm. The specular reflectance calibrations are provided at 25 selected wavelengths, including the laser wavelengths of 632.8 nm and 1060 nm. These mirrors are approximately 50 mm in diameter. The aluminum coating is vacuum deposited on a 9.5 mm thick glass substrate. The mirrors were aged for two years before calibrating. The absolute reflectances of these mirrors were determined by direct comparison to the master first-surface aluminum mirror. The calibration of the master mirror was accomplished by extensive measurements, using the NBS Reference Specular Reflectometer-Spectrophotometer. The absolute techniques for measuring specular reflectance by means of this instrument include analysis of the reflectance of the mirror as a function of wavelength, polarization, and angle of incidence. The measurements obtained through these techniques are uncertain by ±0.2%. The calibration of the Standard Reference Material mirrors was accomplished by direct comparison with the master mirror, using a commercial spectrophotometer. The uncertainty in the values of reflectance obtained by this comparative method of calibration is ±0.5%.

Key Words: Absolute reflectance; aluminum mirrors; first-surface mirrors; specular reflectance; specular standards; standard mirrors; standard reference material.

1. INTRODUCTION

Specular reflectance standards are required by laboratories involved in optical research and by manufacturers of mirrors and related components. Absolute specular reflectance measurements can be made without referring to a calibrated reflectance standard. However, such measurements require great care if highly accurate results are to be achieved. The use of a specular reflectance standard of known absolute reflectance can reduce the problems of standardizing such measurements.

There are several ways of preparing specular reflectors such as evaporating metal onto glass substrates, polishing or diamond turning metal, or electroplating. The most popular technique is evaporating metal onto glass substrates. The substrate can be polished to high optical tolerances and the coatings can be tailored to meet special requirements. Unfortunately, there are no coatings that ideally meet the basic requirements of a specular reflectance standard for durability and long term stability. Nevertheless, because there is a need for such standards, NBS has prepared specular reflectance standards.

The specular reflectance standards available as Standard Reference Materials are first-surface aluminum mirrors that are not very durable but are reasonably stable if properly cared for, and second-surface aluminum mirrors that are durable but not satisfactory for some applications because of the first-surface reflectance from the protective quartz window. Both are calibrated over the spectral range 250 to 2500 nm.

This paper deals with the preparation and calibration of the first-surface aluminum mirrors only. These are available as Standard Reference Material 2003a (50.8 mm diameter). The second-surface mirrors are available as Standard Reference Materials 2023 (51 x 51 mm) and 2024 (25 x 100 mm)[1].

Two types of first-surface mirrors, aluminum mirrors and rhodium mirrors, were originally planned. The rhodium mirrors were selected because of the durability of this metal. However, because of problems with nonuniformity of the coatings, they had to be rejected as being unsuitable as specular reflectance standards.

2. ALUMINUM MIRROR TOLERANCES

The aluminum mirrors were prepared by a fast evaporation in high vacuum. The rate of evaporation was such that the entire coating was deposited in less than 3 seconds and the vacuum was 7×10^{-5} Pa (5×10^{-7} torr.). The aluminum element is pre-fired to coat the tungsten conductor. The evaporation of the aluminum is raised to a level at which a coating of approximately 300 Angstroms thickness per second is deposited. When this level of evaporation is reached, a shutter is opened and the mirrors are coated in approximately 3 seconds. The mirrors are arranged in a hemispherical configuration above the evaporating element so that all mirrors are approximately the same distance from this element. This procedure results in mirrors having a relatively high ultraviolet reflectance and good uniformity. The coating is specified to be 99.999% pure aluminum. The substrate is Cervit[2] C-101, 50.8 mm diameter and 9.5 mm thickness. The substrate surface is polished flat to within 1/10 wavelength of 500 nm, and smooth to within 2.5 nm.

The mirrors prepared by this technique have reflectance greater than 85% at 250 nm and greater than 95% at 2500 nm. The reflectance of the mirrors is uniform over their surface to ±0.1%. The same uniformity in reflectance was observed in a mirror-to-mirror comparison of all the mirrors. The mirrors show some small pinholes in the coating when examined in front of an intense light source. However, these pinholes do not affect the uniformity of the measured reflectance. The mirrors were aged by storing in glass containers for approximately two years before the specular reflectance calibrations were made. Ageing changes are thought to be due to oxidation. More will be said on the subject of mirror ageing in a discussion of uncertainties.

3. SPECULAR REFLECTANCE CALIBRATIONS

A. Survey Measurements

The purpose of the survey measurements was to determine the nonuniformity of the aluminum mirrors at several wavelengths. A survey was made of the reflectance of the 30 mirrors at 250, 550, and 2000 nm, using a commercial spectrophotometer equipped with an integrating sphere reflectance attachment. Although this reflectance attachment is not designed to measure specular reflectance it is suitable for a comparative survey. Mirror No. 1 was used as a control to monitor the instrument drift, while the other mirrors were compared to it on the same photometric scale. From these measurements it was determined that the mirrors had the same reflectance at each of these three wavelengths to within the measurement uncertainty of ±0.1%.

B. Calibration of the Master Standard

The master standard was selected from the lot of 30 mirrors. Calibration of the master standard was accomplished by measurements on the NBS Reference Specular Reflectometer-Spectrophotometer[3]*. This instrument measures specular reflectance by absolute techniques. The measurements are made as a function of wavelength, angle of incidence, and polarization.

In calibrating the master first surface mirror, the instrument was operated with a spectral pass band of 10 nm. The collimated incident beam had a cross section of 18 x 12 mm at the sample. The incident beam was polarized either parallel or normal to the plane of incidence.

* Reference 3 is part of the appendix.

The sample or test mirror was mounted on a turntable. The surface of the mirror and the axis of rotation of the turntable occupied a common vertical plane, thus making it possible to vary the angle of incidence. A complete description of these measurement procedures and a more detailed explanation of the mechanics of the specular reflectometer is given in the accompanying reprint documenting that instrument.

The calibration of the master mirror was made at 50 nm intervals from 250 to 900 nm, at 100 nm intervals from 900 to 1300 nm, at 250 nm intervals from 1500 to 2500 nm, and at the laser wavelengths 632.8 nm and 1060 nm. The total time required to complete these calibrations was approximately 25 hours of instrument running time. The measurements were made at each wavelength for both vertically (S) and horizonally (p) polarized incident beams and at three angles of incidence. The measurements were repeated six times for each of these conditions. Three of the six measurements were made with the angles of incidence set by rotating the mirror clockwise from the normal, and the other three measurements were made with the angles of incidence set by rotating the mirror counterclockwise from the normal. The final reflectance value is an average of six measured values for a given polarization, angle of incidence, and wavelength setting.

The overall uncertainties in the calibration of the master are believed to be on the order of ±0.2%. This uncertainty is based on an analysis of the known uncertainties in the performance of the Reference Spectrophotometer[4] and the specular reflectometer.

Measurements of specular reflectance made at NBS were compared with similar measurements on the same mirror by other laboratories[5]. These comparisons agreed to within ±0.001 indicating that the high accuracy techniques used at NBS and these other laboratories are valid even though the geometry and procedural approach may be different for each laboratory.

C. Calibration of the Standard Reference Material First-Surface Mirrors

Because of the time required to calibrate a mirror on the Reference Specular Reflectometer-Spectrophotometer and the cost of such calibrations it was necessary to resort to less time consuming techniques in order to transfer the absolute reflectance scale from the master mirror to the remainder of the first-surface mirrors that would eventually be issued as Standard Reference Materials. The calibration of these mirrors was accomplished by direct comparison of each mirror with the master mirror at each of the 25 wavelengths for which the master mirror was previously calibrated. This comparison was made on a commercial spectrophotometer equipped with an integrating sphere reflectometer. The comparison was made for 6° incidence only because of the fixed geometry of the commercial reflectometer. The absolute reflectance for 6° incidence of each of the Standard Reference Material mirrors was obtained directly by setting the photometric scale of the spectrophotometer with the master mirror so that the recorder reading matched the corresponding absolute reflectance value of the master mirror at a given wavelength setting. The master mirror was then replaced by a test mirror and the recorded value of its reflectance was read directly from the photometric scale. Since the difference in reflectance between the master mirror and the other Standard Reference Materials mirrors was always within ±0.1%, the direct reading obtained by this procedure required no further corrections. The master mirror reflectance was checked before and after each test mirror in order to detect any drifting of the photometric scale.

4. UNCERTAINTIES

At some wavelengths, the instrument noise of the commercial spectrophotometer was slightly greater than ±0.2%. Therefore the final uncertainty for the Standard Reference Material mirrors (the sum of the uncertainty for the master standard and that for the measured mirror) was increased to ±0.5%. This uncertainty is larger than the ±0.2% assigned to the master through the more accurate determinations made on the Reference Specular Reflectometer. However, the uncertainty of ±0.5% is probably realistic for the Standard Reference Material mirrors. Uncertainties less than ±0.5% cannot be guaranteed without careful absolute techniques, and the assigned values of reflectance may not remain valid with smaller uncertainties for mirrors that are used regularly or have aged several years.

The reflectance of aluminum mirrors is influenced by many factors. The highest reflecting aluminum mirrors are obtained by reducing the evaporation to only a few seconds in ultra-high vacuum. Oxidation of the aluminum upon exposure to air results in a lowering of the reflectance particularly in the ultraviolet. Although most of the oxidation and resulting reduction in reflectance takes place within the first year of ageing, the process continues at a slower rate for a longer time. Extensive investigations into the influence of various parameters such as purity, temperature, and oxidation have been reported by other authors[6,7,8,9,10,11].

The best alternative to making specular reflectance measurements by absolute techniques is to obtain a new calibration of the user's standard mirror after two years if the mirror is in reasonably good condition. In this manner, the user will have an aged mirror that is less likely to change rapidly after recalibration. An every-day working standard can be calibrated relative to the Standard Reference Material mirror by the user for situations in which the standard must be handled often. This will help to preserve the Standard Reference Material mirror for periodic control of the measurement process.

5. CLEANING AND RECALIBRATION

It is not recommended that the Standard Reference Material mirror be cleaned by any technique. Any cleaning attempted, no matter how careful, is likely to damage the mirror or result in some change that will render the calibration invalid. If the mirror is damaged through accident or careless handling and cannot be recalibrated it may be possible to salvage the glass substrate for future use.

6. THE CERTIFICATES OF CALIBRATION

The first-surface aluminum mirrors have been designated Standard Reference Material 2003a. A copy of the certificate is included here for general information. The spectral reflectance of the mirrors in the wavelength range 250 to 2500 nm is shown in the graph on page 2. The wavelength scale is greatly compressed and the photometric scale expanded in this graph. Therefore, the absorption feature at approximately 800 nm is emphasized. The data in Table 1 of the certificate are valid for 6° incidence only. However, they are useful for other angles of incidence near normal for essentially unpolarized sources. The data given in Table 2 of the certificate are uncertified but represent a typical first-surface aluminum mirror. They show that the reflectance of the mirrors for the unpolarized incident beam does not vary significantly for angles of incidence up to 45°. The variation in reflectance with angle of incidence may be several percent for polarized incident beams depending on the wavelength.

7. REFERENCES

1. Inquires about Standard Reference Materials 2023 and 2024 (second-surface aluminum mirrors) should be addressed to the National Bureau of Standards, Office of Standard Reference Materials, Washington, D. C. 20234.

2. Reference to products by commercial name are given in this paper for identification only and in no way imply endorsement by the National Bureau of Standards.

3. Weidner, Victor R., and Hsia, Jack J., NBS Specular Reflectometer-Spectrophotometer, Applied Optics, Vol. 19, page 1268, April, 1980.

4. Venable, William H. Jr., Hsia, Jack J., and Weidner, Victor R., Development of an NBS Reference Spectrophotometer for Diffuse Reflectance and Transmittance, Nat. Bur. Stand. (U.S.), Tech. Note 594-11, 47 pages, (Oct. 1976).

5. Linford, R. M. F., Strouse, E. A., Optical Coatings for Space Laser Communications System, SPIE, Vol. 150, Laser & Fiber Optics Communications, Page 55-61, 1978.

6. Hass, G., Filmed Surfaces for Optics, JOSA, Vol. 45, No. 11, Page 945-952, 1955.

7. Hass, G., Hunter, W. R., Tousey, R., Influence of Purity, Substrate Temperature, and Ageing Conditions on the Extreme Ultraviolet Reflectance of Evaporated Aluminum, JOSA, Vol. 47, No. 12, Page 1070-1073, 1957.

8. Hass, G., Tousey, R., Reflecting Coatings for the Extreme Ultraviolet, JOSA, Vol. 49, No. 6, Page 593-602, 1959.

9. Hass, G., Waylonis, J. E., Optical Constants and Reflectance and Transmittance of Evaporated Aluminum in the Visible and Ultraviolet, JOSA, Vol. 51, No. 7, Page 719-722, 1961.

10. Bennett, H. E., Silver, M., Ashley, E. J., Infrared Reflectance of Aluminum Evaporated in Ultra-High Vacuum, JOSA Vol. 53, No. 9, Page 1089-1095, 1963.

11. Hass, G., Reflectance and preparation of front-surface mirrors for use at various angles of incidence from the ultraviolet to the far infrared, JOSA, Vol. 72, No. 1, Page 27-39, 1982.

8. APPENDICES

These appendices contain reproductions of a publication "NBS Specular Reflectometer-Spectrophotometer" and a certificate issued by NBS for the SRM 2003a.

Reprinted from **Applied Optics**, Vol. 19, page 1268, April, 1980

NBS specular reflectometer–spectrophotometer

Victor R. Weidner and Jack J. Hsia

A specular reflectometer has been constructed and tested for calibrating the reflectance of mirror standards over the 250–2500-nm spectral range. This instrument is a measurement accessory to a reference spectrophotometer, which is also used for diffuse hemispherical spectral reflectance and 45°/0° spectral reflectance. The specular reflectometer is designed to measure mirror reflectances at angles of incidence between 5 and 80° using both vertically and horizontally polarized radiation. Absolute reflectance measurements are obtained by an optical system, which provides for direct measurement of the incident beam and for the sample mirror reflectance using the same beam. This is accomplished by means of a beam tracking system through which the beam is directed into a signal averaging sphere. The sphere rotates with the beam tracking optics, and the stationary detector views the interior of the sphere. Control of the beam tracking optical system is accomplished by a computer-controlled stepping-motor-driven precision turntable. Uncertainties of the reflectance measurements obtained with this system are estimated to be ±0.2% of the measured value.

I. Introduction

The increasing requirements for standard specular reflectors, such as mirrors for reference measurements in activities associated with the solar energy utilization program, have also increased the need for instrumentation development to provide the required standard mirrors. The spectrophotometry group of the Radiometric Physics Division at NBS has developed a specular reflectometer for this purpose. It is designed to measure the specular reflectance of mirrors over the 250–2500-nm wavelength range. The instrument can be used to measure the specular reflectance of mirrors at angles of incidence between ~5 and 80°. The spectrophotometer provides a collimated beam, which can be polarized in either the vertical or horizontal plane. The spectral bandpass is usually set at 5 or 10 nm.

The specular reflectometer will be used primarily to calibrate mirrors for the NBS Standard Reference Materials program. Several types of mirrors will be available through the NBS Office of Standard Reference Materials when work is completed on the calibration of several master standards, and this reflectance scale can be transferred to the SRM mirrors. The mirrors being prepared for calibration include (a) glass substrates with either aluminum or rhodium front-surface coatings and (b) second-surface aluminum coatings sealed between quartz. A portion of this mirror preparation and calibration work is funded by the Department of Energy.

Calibrating a mirror for specular reflectance as a function of wavelength, polarization, and angle of incidence requires many individual measurements. These calibrations are impractical without automation. As an example, measurement of the specular reflectance of a single mirror at fifty wavelengths, for five angles of incidence and two polarizations requires 500 determinations. Moreover, this number is doubled if the angle of incidence is measured to both the left and right of the normal and then averaged. Since at each angle of incidence three measurements are made and other calibration data also have to be recorded, the total number of data may reach 13,000 individual numbers to be accumulated and processed. Even with automation it would be time-consuming and expensive to calibrate many mirrors at this many wavelengths and angles of incidence. For this reason most mirrors are calibrated at near normal incidence for 50- or 100-nm intervals and at only a few wavelengths for other angles of incidence.

II. NBS Reference Spectrophotometer

The specular reflectometer is an accessory to the NBS reference spectrophotometer for reflectance.[1] The monochromator, light source, and associated equipment are located in a system control room. The exit-slit housing of the monochromator is attached to a light-tight diaphragm in a wall, which allows the exit beam

The authors are with U.S. National Bureau of Standards, Washington, D.C. 20234.

Received 29 October 1979.

or sample beam to enter a second room where the various reflectance-measuring devices, such as integrating spheres or the specular reflectometer, are installed. The advantages of this arrangement are that the second room can be used as an experimental dark chamber, while the electronics, light sources, and control systems are isolated from the experimental area. The sample beam emerging from the exit slit of the monochromator can be controlled to provide a spectral bandpass of 2, 5, 10, or 20 nm. The beamwidth is determined by the selected bandpass. For a 10-nm bandpass, the beam is $\sim 12 \times 18$ mm at the sample plane.

To measure spectral reflectance over the full wavelength range of the monochromator, the system employs a xenon source for measurements in the UV spectrum, a tungsten strip lamp for the visible spectrum, and a tungsten IR source for the IR spectrum. A photomultiplier detector is used for the UV and visible spectrum and a lead sulfide detector for the IR spectrum. A complete description of the spectrophotometer is given in an earlier publication,[1] with the exception of the IR detection and signal-processing system, which was completed at a later date.

III. Construction of the Specular Reflectometer

The concept around which the specular reflectometer is designed allows for detecting the incident beam either directly or after it is reflected from a mirror sample at some selected wavelength and angle of incidence. To accomplish this it is necessary to employ two turntables—one for positioning the sample mirror at a desired angle of incidence with respect to the sample beam and another for directing the reflected sample beam to reach the detector. The instrument is illustrated in Figs. 1–3. It is undesirable to irradiate the detector directly with the beam to be measured because the detector area lacks the necessary uniformity of sensitivity required for a system involving the movement of optical elements that direct the beam to the detector. Therefore, reproducibility of the measurements is not adequate for high-accuracy determinations. Since photomultiplier detectors tend to be affected by magnetic fields, which vary from one location to another, the instrument was designed with the detector located in a fixed position.

To overcome the problems of detector nonuniformity, the specular reflectometer is designed so that the sample beam enters a 15-cm diam averaging sphere; output variations caused by nonuniformity of the beam or by nonuniformity of the detector sensitivity to this beam are effectively reduced when the beam is first diffusely mixed by multiple reflections within the averaging sphere. The sphere coating is a fluorocarbon powder, which is a nearly perfect diffuser of very high reflectivity throughout the 250–2500-nm spectral range. The detector views the interior of the averaging sphere and is irradiated by the diffused flux. The incoming sample beam is incident on the sphere wall at a location outside the field of view of the detector to avoid direct viewing of this bright spot on first reflection.

The problem of magnetic fields affecting the detector performance required a design for the reflectometer that would keep the detector in a fixed position for all measurements regardless of the angle of incidence selected for the specular-reflectance measurements. To do this a mirror was attached to an arm suspended from a turntable located above the sample turntable. This mirror, referred to as the tracking mirror, can orbit the sample, intercepting the reflected sample beam at any angle of reflectance (= angle of incidence) between ~ 5 and 80°. The tracking mirror also intercepts the direct beam when the sample mirror is moved out of the beam by the slider on which it is mounted.

The tracking mirror is tilted upward at an angle of 20° with respect to the incident beam and reflects the beam up through a baffle tube and into the averaging sphere, which rotates with the tracking mirror and baffle tube. The axis of rotation of the averaging sphere is collinear with the axis of rotation of the sample turntable. The detector is mounted on a stationary platform above the averaging sphere and does not rotate with the sphere. A diaphragm allows the top of the sphere to rotate just below the detector aperture and maintains a radiation seal between the stationary and rotating parts. A 51-mm diam port on the top of the sphere allows the signal to pass into the detector. A shutter between the sphere and the detector can be opened or closed automatically or as required.

The baffle tube is attached to the averaging sphere and extends from the 32-mm diam entrance aperture of the sphere to near the 51-mm diam tracking mirror. The baffle tube limits the field of view of the averaging-sphere entrance aperture to the direction of the tracking mirror. The entrance aperture to the sphere is the limiting aperture in the optical system.

The tracking mirror orbits the sample at a radius of 25 cm. The axis of rotation of the sample and the axis of rotation of the tracking mirror must be collinear if the tracking mirror is to intercept accurately the reflected beam at all angles of incidence and reflection and redirect the beam into the averaging sphere.

The sample-mirror turntable and tracking-mirror turntable are driven by identical 240-tooth precision worm gears. Two hundred steps are required to turn the stepping-motor drive shaft through 360°. This translates through the worm gear into 48,000 steps to rotate the turntables 360°. The angular resolution for the turntable rotation is ~ 27 sec of arc/step of the stepping motor. The problem of backlash in the gear drive is avoided by always approaching the desired position from the same direction. This is done through the computer routines that control the direction of movement in approaching the desired angular settings for specular-reflectance measurements.

The IR measurements required the addition of a chopper at the light source, a preamplifier for the lead sulfide detector, and a lock-in amplifier that locks in the chopped signal and provides a suitable input to the data processing electronics. Figure 4 is a schematic diagram of the IR-system electronics and the signal-generation and detection system.

The signal processing of the output from the detector utilizes a current-to-frequency converter that can in-

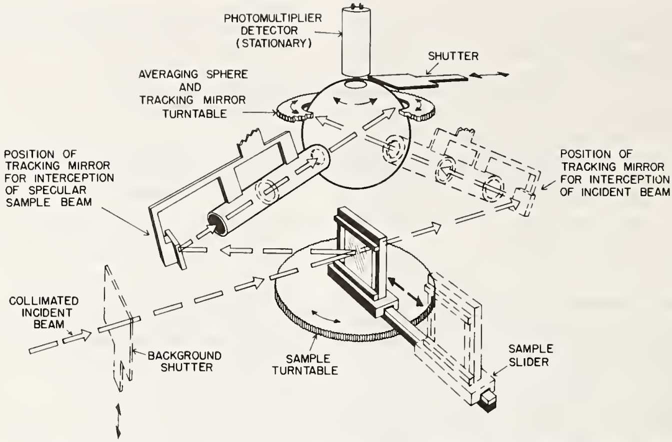

Fig. 1. General scheme of the NBS specular reflectometer showing the relationship of the sample turntable and beam-tracking turntable and detector systems.

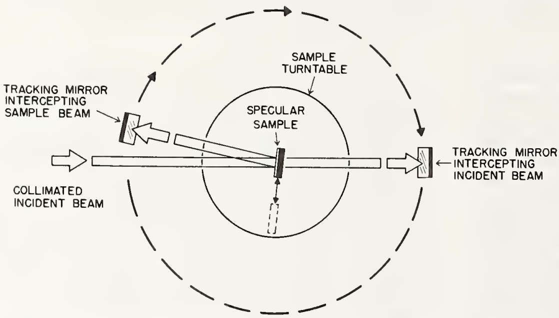

Fig. 2. Horizontal x section through the plane of the sample beam, specular sample, and the tracking mirror that orbits the sample.

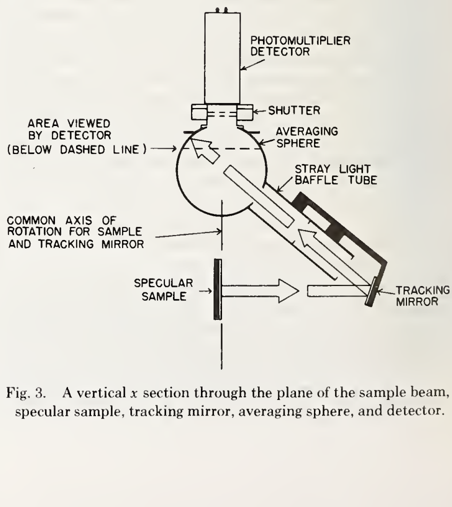

Fig. 3. A vertical x section through the plane of the sample beam, specular sample, tracking mirror, averaging sphere, and detector.

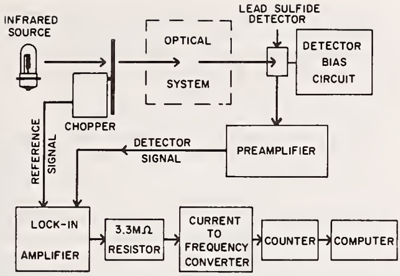

Fig. 4. Schematic of the IR signal processing system for the NBS specular reflectometer–spectrophotometer system.

tegrate the detector output over a selected time interval. The integration time may be as short as 1 sec or as long as 9 sec. This integrated signal is accumulated on a digital counter. The digital results in the counter are transferred to a computer for analysis. The results of the computer analysis are stored for future reference and printed out on a teletype printer.

IV. Alignment of the Reflectometer

Alignment of the specular reflectometer involves the spatial relationship of three basic elements in the system. These are the sample beam, the sample, and the tracking mirror. The sample beam is initially adjusted so that it is in a horizontal plane and passes over the sample turntable with the axis of rotation bisecting the beam. The reflectometer is mounted on a lift table for convenience in selecting the desired vertical position for the instrument. This lift table rides on bearings so that the instrument can be moved into or out of the sample beam at right angles to the beam. When these vertical and horizontal adjustments are correct for the relationship of the sample turntable with respect to the sample beam the system is locked to prevent further movement.

The upper portion of the specular reflectometer, consisting of the tracking-mirror turntable and the stationary detector platform, is independent of the sample turntable. This assembly is supported by three columns located at 120° intervals with respect to the axis of rotation of the turntables. Each of these columns is designed to provide a kinematic alignment of the detector and tracking-mirror assembly. The three legs of the tracking-mirror turntable assembly rest on the kinematic supports at the top of the three columns. The legs are fitted with steel-ball-tipped feet that fit into the kinematic supports consisting of a flat for one ball tip, a V-groove for the second ball tip, and a conical cavity for the third. The ball tips of the legs are on finely threaded bolts that screw into the legs and provide a means for adjusting the height and leveling the tracking mirror to the plane of the incident beam and sample mirror.

The alignment procedure involves a number of adjustments of the sample mirror and tracking mirror. These procedures involve a number of mechanical fixtures and optical techniques that will not be described in detail. As a final check of the overall alignment of the reflectometer system, the baffle tube is temporarily removed from the tracking mirror arm, and the sample turntable and tracking mirror turntable are programmed to move to several angles of incidence and interception, respectively. At these locations the sample beam must pass through the center of the entrance aperture to the averaging sphere. If this condition is not satisfactorily arrived at, the alignment procedures are rechecked and refined until they meet the required conditions.

A laser is used to monitor the system alignment for possible trouble during continued lengthy measurements in which the system is being driven by computer-controlled stepping motors. The laser is fixed to a stationary pier. The laser beam reflects off the sample mirror and strikes a target scale, which is used to initiate a measurement cycle. At the end of each measurement cycle the turntables return to this position if the stepping motors and computer have not malfunctioned and no mechanical misalignment has occurred. This laser alignment-checking procedure is especially useful when the measurements of specular reflectance are being made at wavelengths where the sample beam cannot be visually observed. However, the initial alignment is usually made with a sample-beam wavelength of 550 nm. Should a computer malfunction occur while working at IR or UV wavelengths, the laser is turned on, and the turntables are manually reset to the starting angles. Any large malfunction involving the positioning of the turntables is usually readily detected because the sample beam under these conditions ordinarily misses the detector system completely, a condition easily recognized in the data output. The laser alignment check alerts the operator to small misalignment problems that might not be apparent in the data output. The laser is used primarily to check the sample-turntable alignment. The tracking-mirror starting point can be accurately set by a scale mark on the 60-cm diam track and a similar scale mark on the tracking-mirror holder, which moves around this track. This initial sample-turntable position must be established by optical techniques to achieve the required accuracy in programming the angles of incidence for specular-reflectance measurements.

V. Measuring Specular Reflectance

Measurements of specular reflectance on the NBS specular reflectometer involve the following series of measurements repeated for each selected wavelength, polarization, and angle of incidence: B, S_1, S_2, S_1, B, S_1, S_2, S_1, B, S_1, S_2, S_1, B, where B is the background signal, S_1 is the measured signal of the incident beam, and S_2 is the measured signal of the beam after reflection from the sample mirror. The average B, average S_1, and average S_2 are used in the data reduction. The background is subtracted from S_1 and S_2. A correction is also made for those effects associated with the rotation of the averaging sphere as determined in the β-light mapping procedure, which will be described in a later section.

The sample mirror reflectance R is calculated as $R = S_2/S_1$. The average value of specular reflectance for the vertically and horizontally polarized incident beam is usually reported. The usual practice is to determine the specular reflectance of a mirror at a specified angle of incidence by measuring the reflectance with the plane of the mirror rotated to the left with respect to the incident beam and again for the same angle of incidence with the mirror plane rotated to the right. These two determinations are then averaged. The specular reflectometer is programmed to perform these measurements, and the data are processed at the completion of each measurement cycle. A complete measurement cycle includes the specified wavelength and angles of incidence.

VI. Performance

It is beyond the intent of this paper to describe in detail the various checks that were made on the performance of the monochromator and other components of the spectrophotometer, except to mention briefly the magnitudes of errors associated with this portion of the system, since they do influence the accuracy of measurements of specular reflectance. Information regarding some of these investigations is presented in Ref. 1.

Involved in checking the performance of the instrument are wavelength scale uncertainties, stray light levels, detector linearity, and variations in detector response associated with movement of the specular reflectometer optical components.

The wavelength scale of the monochromator is periodically checked by measuring the emission lines of several line-source lamps, the transmission of a didymium glass wavelength standard, polystyrene absorption bands, and the absorption bands of 1-2-4 trichlorobenzene.[2] Uncertainty in the wavelength scale is 1 nm or less. Corrections for these errors are made by adjustment of the wavelength-scale setting to compensate for differences between the wavelength counter and the true wavelength.

The recent extension of the instrument to cover the IR spectral range to 2.5 μm involved checking for stray light in this spectral range. The amount of information on stray radiation in the IR range is limited somewhat by the lack of suitable filters for this purpose in the 2–2.5-μm wavelength range. Several IR cutoff selenium glass filters, silicon filters, and chloroform were used to check for stray radiation in the 0.8–1.7-μm wavelength range.[3] The selenium glass filters have sharp cutoffs between 0.7 and 0.9 μm, and the silicon filter cuts off at $\sim$1.0 μm. The filters block the UV and visible wavelengths and transmit the IR. The chloroform absorbs strongly[3] at 1.69 μm. The stray radiation levels for the wavelengths checked with these filters were found to be less than 0.1%. This is below the signal level for an optical density of 3, which is about as low a level as can be measured with a lead sulfide detector of the type used for these measurements.

The linearity of the lead sulfide detector was measured by the light addition method with a double aperture apparatus.[4] The results of these linearity measurements indicate that the detector is linear to better than $\pm$0.1% at all photometric levels between 10 and 100%, which is the photometric range of interest for most of the reflectance measurements of mirrors.

Radiation scattered off the optical components such as mirrors between the exit slit of the monochromator and the limiting aperture of the detector system is undesirable in a specular reflectometer. The room containing the reflectometer is lined with a black felt material to absorb scattered radiation. The level of scattered radiation in this room is low enough to be undetectable above the dark current signal of the detector with the sample beam trapped by the background shutter at the exit slit of the monochromator. Since the baffle tube limits the field of view of the limiting aperture to the area of the tracking mirror, very little scattered radiation can enter the averaging sphere. Most of the scattered radiation is associated with the scattering in the optical path of the sample beam. It is important that all the mirrors in this system be kept reasonably clean and free of dust.

One problem associated with a measurement of reflectance in which optical components are moved between the source and the detector to change the incidence and viewing angles is that of determining the magnitude of errors introduced by these movements. In this system the number of components that require movement has been reduced to the beam-tracking mirror and the averaging sphere. The detector remains stationary and views the interior of the sphere through a 51-mm diam port, which rotates with minimum translation in the field of view of the detector. The flux received by the detector is diffuse in nature since the interior of the sphere is a nearly perfect diffuser. To test the sensitivity of the detector response to changes in the angular positioning of the beam-tracking mirror, a β light was attached to the baffle tube entrance. The β light is radioactive tritium gas, which emits visible light when the β radiation excites a phosphorous target. This stable light source is also enclosed in a magnetic shield. Light from this source illuminates the averaging sphere at the other end of the baffle tube. Variations in the intensity of the signal from the β light as the tracking system turntable rotates may then be attributed to some change in the signal intensity at the detector, which is related to the turning of the averaging sphere and not to the instability of the light source. The maximum variation in signal intensity observed by this technique was $\sim\pm$0.2%. It was thought that this variation might be caused by the fact that the entrance port of the averaging sphere, which appears as a black spot in the detector field of view, is changing position as the averaging sphere rotates. However, when a white baffle was installed in the sphere to prevent the detector from seeing this black spot, the variation in signal intensity for the β light test still existed and was actually larger than that observed without the baffle. The variation in signal intensity can be mapped as a function of the turntable position with respect to the incident beam, and corrections can be derived for this error. The β light mapping check is a regular part of the calibration procedure.

An analysis of the various sources of error that may be expected to affect the accuracy of specular reflectance measurements on the NBS specular reflectometer indicates that the final uncertainty in these measurements will be on the order of ±0.2% of the measured value.

VII. Summary

A specular reflectometer spectrophotometer was constructed for calibrating the reflectance of mirrors over the 250–2500-nm spectral range. The instrument is automated and computer controlled. The measurements are made as a function of wavelength and angle of incidence with a polarized light beam. The system performance was examined for such parameters as stray radiation levels, detector linearity, wavelength scale uncertainties, and spatial sensitivity of the detector system. Uncertainty in the measurements of specular reflectance will be on the order of ±0.2% of the measured value. The instrument will be used to provide calibrated mirrors, which will become available through the NBS Office of Standard Reference Materials.

The authors are indebted to William H. Venable, Jr., formerly of NBS and presently employed by Hunter Associates, Inc., for his contributions to the design and development of the specular reflectometer and reference spectrophotometer.

References

1. W. H. Venable, Jr., J. J. Hsia, and V. R. Weidner, Development of an NBS Reference Spectrophotometer for Diffuse Transmittance and Reflectance, Nat. Bur. Stand. Tech. Note U.S. 594-11 (October 1976).
2. E. K. Plyer and C. W. Peters, J. Res. Nat. Bur. Stand. **45**, 462 (December 1950).
3. W. Kaye, Appl. Opt. 14, 1977 (1975).
4. K. D. Mielenz and K. L. Eckerle, Appl. Opt. 11, 2294 (1972).

U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 2003a

First Surface Aluminum Mirror for Specular Reflectance from 250 to 2500 nm

V. R. Weidner and J. J. Hsia

This Standard Reference Material (SRM) is intended for use in calibrating the photometric scale of specular reflectometers. SRM 2003a is 5.1 cm in diameter. The aluminum is vacuum deposited on a glass substrate and aged two years before calibration. No other protective coatings are applied to the mirror.

The specular reflectance of the mirror was measured at 50-nm intervals from 250 nm to 900 nm, 100-nm intervals from 900 nm to 1300 nm, and 250-nm intervals from 1500 nm to 2500 nm. In addition to these wavelengths, the reflectance was measured at the laser wavelengths 632.8 nm and 1060 nm. The certified values were determined in the following way. The reflectance of a master mirror was measured at the above specified wavelengths with a highly accurate specular reflectometer-spectrophotometer at angles of incidence of 6°, 30°, and 45°. These measurements were made for both vertically and horizontally polarized incident beams. The overall uncertainty in these measurements is ±0.2 percent. The specular reflectance of the SRM first surface mirror was measured relative to the master mirror on a high-precision reflectometer for 6° incidence only. The certified values of specular reflectance for the SRM mirror are based on the average value of the vertical and horizontal polarizations for the master mirror at 6° incidence. The certified values listed in Table 1 are assigned an uncertainty of ± 0.005. The uncertified data listed in Table 2 indicate the variation in the specular reflectance of a typical first surface SRM mirror as a function of angle of incidence and plane of polarization.

Figure 1 shows the spectral distribution of a typical first surface aluminum mirror. The wavelength scale of this plot is greatly compressed and the reflectance scale expanded to emphasize the absorption features. Note that the absorption band at 800 nm is an inherent characteristic of aluminum mirrors.

SRM 2003a cannot be cleaned without adversely affecting the aluminum coating. It is suggested that the mirror be handled carefully so as not to touch the aluminum surface and that the mirror be stored in a covered glass enclosure when not being used.

The calibration of this SRM was done in the Radiometric Physics Division of the Center for Radiation Research. The technical and support aspects involved in the certification and issuance of this SRM were coordinated through the Office of Standard Reference Materials by R. K. Kirby.

Washington, D.C. 20234
March 30, 1981

George A. Uriano, Chief
Office of Standard Reference Materials

Table 1

First Surface Mirror

(6° Incidence)

Wavelength (nm)	Reflectance
250	0.895
300	.910
350	.914
400	.915
450	.913
500	.912
550	.909
600	.904
632.8	.901
650	.898
700	.890
750	.877
800	.857
850	.856
900	.890
1000	.935
1060	.947
1100	.951
1200	.958
1300	.962
1500	.965
1750	.967
2000	.968
2250	.968
2500	.968

Table 2

The spectral reflectance of a typical first surface mirror as a function of wavelength, angle of incidence, and polarization.

(These values are not certified)

Wavelength and Angle of Incidence	Parallel(p) Polarized	Perpendicular(s) Polarized	Unpolarized (ordinary)
250 nm			
6°	0.894	0.891	0.8925
30°	.882	.904	.893
45°	.867	.920	.8935
300 nm			
6°	.907	.904	.9055
30°	.898	.915	.9065
45°	.881	.929	.905
400 nm			
6°	.914	.913	.9135
30°	.903	.924	.9135
45°	.885	.937	.911
600 nm			
6°	.904	.905	.9045
30°	.892	.917	.9045
45°	.871	.932	.9015
800 nm			
6°	.856	.858	.857
30°	.837	.875	.856
45°	.806	.896	.851
1000 nm			
6°	.935	.936	.9355
30°	.929	.946	.9375
45°	.915	.956	.9355
1500 nm			
6°	.965	.966	.9655
30°	.964	.973	.9685
45°	.959	.978	.9685
2000 nm			
6°	.967	.968	9675
30°	.968	.976	.972
45°	.963	.981	.972
2500 nm			
6°	.966	.970	.968
30°	.970	.973	.9715
45°	.966	.980	.973

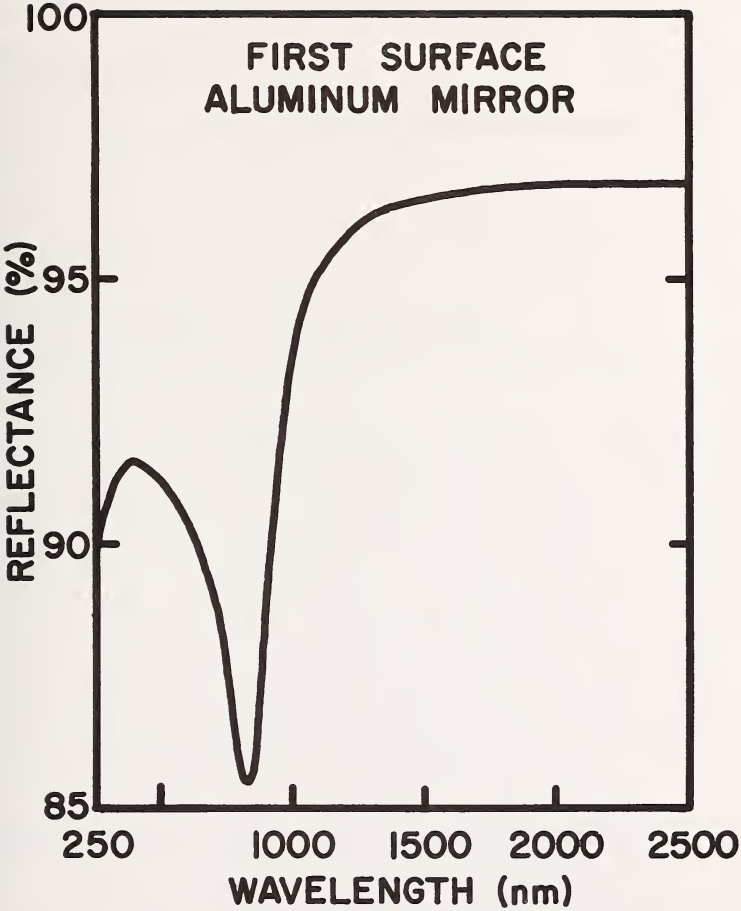

Figure 1. Typical spectral reflectance curve of the SRM 2003a first surface alumninum mirrors.

NBS-114A (REV. 2-80)

U.S. DEPT. OF COMM. **BIBLIOGRAPHIC DATA SHEET** *(See instructions)*	1. PUBLICATION OR REPORT NO. NBS SP 260-75	2. Performing Organ. Report No.	3. Publication Date May 1982

4. TITLE AND SUBTITLE

Standard Reference Materials:
Preparation and Calibration of First-Surface Aluminum Mirror Specular Reflectance Standards

5. AUTHOR(S)

Victor R. Weidner and Jack J. Hsia

6. PERFORMING ORGANIZATION *(If joint or other than NBS, see instructions)*

NATIONAL BUREAU OF STANDARDS
DEPARTMENT OF COMMERCE
WASHINGTON, D.C. 20234

7. Contract/Grant No.

8. Type of Report & Period Covered

Final

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS *(Street, City, State, ZIP)*

same as above

10. SUPPLEMENTARY NOTES

This Special Publication is intended as supplemental information to accompany Standard Reference Material 2003a.

Library of Congress Catalog Card Number: 82-600542

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

A number of first-surface aluminum mirrors of high optical quality have been prepared and calibrated for use as specular reflectance standards over the wavelength range 250 to 2500 nm. The specular reflectance calibrations are provided at 25 selected wavelengths, including the laser wavelengths of 632.8 nm and 1060 nm. These mirrors are approximately 50 mm in diameter. The aluminum coating is vacuum deposited on a 9.5 mm thick glass substrate. The mirrors were aged for two years before calibrating. The absolute reflectances of these mirrors were determined by direct comparison to the master first-surface aluminum mirror. The calibration of the master mirror was accomplished by extensive measurements, using the NBS Reference Specular Reflectometer-Spectrophotometer. The absolute techniques for measuring specular reflectance by means of this instrument include analysis of the reflectance of the mirror as a function of wavelength, polarization, and angle of incidence. The measurements obtained through these techniques are uncertain by ±0.2%. The calibration of the Standard Reference Material mirrors was accomplished by direct comparison with the master mirror, using a commercial spectrophotometer. The uncertainty in the values of reflectance obtained by this comparative method of calibration is ±0.5%.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

absolute reflectance; aluminum mirrors; first-surface mirrors; specular reflectance; specular standards; standard mirrors; standard reference material

13. AVAILABILITY

[x] Unlimited
[ ] For Official Distribution. Do Not Release to NTIS
[X] Order From Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.
[ ] Order From National Technical Information Service (NTIS), Springfield, VA. 22161

14. NO. OF PRINTED PAGES

26

15. Price

USCOMM-DC 6043-P80

☆U.S. GOVERNMENT PRINTING OFFICE: 1982-360-997/2093

NBS TECHNICAL PUBLICATIONS

PERIODICALS

JOURNAL OF RESEARCH—The Journal of Research of the National Bureau of Standards reports NBS research and development in those disciplines of the physical and engineering sciences in which the Bureau is active. These include physics, chemistry, engineering, mathematics, and computer sciences. Papers cover a broad range of subjects, with major emphasis on measurement methodology and the basic technology underlying standardization. Also included from time to time are survey articles on topics closely related to the Bureau's technical and scientific programs. As a special service to subscribers each issue contains complete citations to all recent Bureau publications in both NBS and non-NBS media. Issued six times a year. Annual subscription: domestic $18; foreign $22.50. Single copy, $4.25 domestic; $5.35 foreign.

NONPERIODICALS

Monographs—Major contributions to the technical literature on various subjects related to the Bureau's scientific and technical activities.

Handbooks—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications—Include proceedings of conferences sponsored by NBS, NBS annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

Applied Mathematics Series—Mathematical tables, manuals, and studies of special interest to physicists, engineers, chemists, biologists, mathematicians, computer programmers, and others engaged in scientific and technical work.

National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a worldwide program coordinated by NBS under the authority of the National Standard Data Act (Public Law 90-396).

NOTE: The principal publication outlet for the foregoing data is the Journal of Physical and Chemical Reference Data (JPCRD) published quarterly for NBS by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements available from ACS, 1155 Sixteenth St., NW, Washington, DC 20056.

Building Science Series—Disseminates technical information developed at the Bureau on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

Technical Notes—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NBS under the sponsorship of other government agencies.

Voluntary Product Standards—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The standards establish nationally recognized requirements for products, and provide all concerned interests with a basis for common understanding of the characteristics of the products. NBS administers this program as a supplement to the activities of the private sector standardizing organizations.

Consumer Information Series—Practical information, based on NBS research and experience, covering areas of interest to the consumer. Easily understandable language and illustrations provide useful background knowledge for shopping in today's technological marketplace.

Order the **above** *NBS publications from: Superintendent of Documents, Government Printing Office, Washington, DC 20402.*

Order the **following** *NBS publications—FIPS and NBSIR's—from the National Technical Information Services, Springfield, VA 22161.*

Federal Information Processing Standards Publications (FIPS PUB)—Publications in this series collectively constitute the Federal Information Processing Standards Register. The Register serves as the official source of information in the Federal Government regarding standards issued by NBS pursuant to the Federal Property and Administrative Services Act of 1949 as amended, Public Law 89-306 (79 Stat. 1127), and as implemented by Executive Order 11717 (38 FR 12315, dated May 11, 1973) and Part 6 of Title 15 CFR (Code of Federal Regulations).

NBS Interagency Reports (NBSIR)—A special series of interim or final reports on work performed by NBS for outside sponsors (both government and non-government). In general, initial distribution is handled by the sponsor; public distribution is by the National Technical Information Services, Springfield, VA 22161, in paper copy or microfiche form.

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Washington, DC 20234

OFFICIAL BUSINESS

Penalty for Private Use, $300

POSTAGE AND FEES PAID
U.S. DEPARTMENT OF COMMERCE
COM—215

U.S. MAIL

FIRST CLASS