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NBS Response to the Fourth CORM Report on Pressing Problems and Projected National Needs in Optical Radiation Measurements

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Radiometric Physics Division
Gaithersburg, MD 20899

May 1984

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**NBS RESPONSE TO THE FOURTH
CORM REPORT ON PRESSING
PROBLEMS AND PROJECTED NATIONAL
NEEDS IN OPTICAL RADIATION
MEASUREMENTS**

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

The Council for Optical Radiation Measurements (CORM) was founded in 1972 as a non-profit organization of professionals and institutions engaged in optical radiometry and spectrophotometry. The aims and purposes of CORM include:

- o Assessment of national requirements for standards, calibrations, and interlaboratory collaborations.
- o Establishment of national consensus on priorities for these requirements.
- o Liason with the National Bureau of Standards in order to advise NBS of requirements and priorities.
- o Dissemination of information on standards activities in optical radiometry and spectrophotometry.

In fulfilment of its statutory obligations, CORM has periodically issued technical reports on measurement problems and needed standards. In its past reports, CORM listed many requests for specific services from NBS over such a wide range that it was difficult for the Bureau to make a significant response. To allow more expeditious solutions, the CORM Directors chose a new approach in preparing their Fourth Report and have now classified the perceived standards needs into three categories: those that must be addressed by NBS in its role as national laboratory, those that can be solved by collaborative efforts of industry and NBS, and those that can be solved by the users themselves with guidance from the Bureau. NBS agrees that such a classification of needs is realistic.

This publication constitutes the NBS Response to the Fourth CORM Report. It describes NBS policies for radiometry and spectrophotometry, the current status of projects suggested by CORM, and future plans. It also contains specific proposals for collaborative CORM/NBS efforts to provide needed standards and measurement services. With permission by CORM, the Fourth CORM Report itself is included as an appendix.

Klaus D. Mielenz, Chief
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NBS RESPONSE TO THE FOURTH CORM REPORT ON PRESSING PROBLEMS AND PROJECTED NATIONAL NEEDS IN OPTICAL RADIATION MEASUREMENTS

I. GENERAL COMMENTS

Like its predecessors, the Fourth Report on "Pressing Problems and Projected National Needs" of the Council for Optical Radiation Measurements (CORM) offers sound technical advice to the management and staff of the National Bureau of Standards. The information contained in it is important for evaluating the relevance and effectiveness of our ongoing programs in optical radiometry and for planning our future activities in these areas. Without such input from its constituents, NBS could not be responsive to user requirements for physical standards and measurement services.

The importance of the Fourth CORM Report has been enhanced by the fact that major portions of it were incorporated into the 1982 National Conference of Standards Laboratories (NCSL) National Measurement Requirements Survey.^{1,2)} The NCSL survey augments the findings of CORM, inasmuch as it addresses some of the radiometric measurement needs of user groups, notably the defense and aerospace communities, which are not as strongly represented in CORM. However, these needs were not ranked by priority as in the CORM Report.

NBS is indebted to CORM for the considerable degree of thought and effort that went into the preparation of the Report. In particular the Bureau appreciated the new approach taken by CORM in classifying the measurement needs in radiometry and spectrophotometry, not only by their technical significance and industrial impact, but also in terms of ways of achieving them in the most efficient manner. This approach has led CORM to conclude that it is impossible for the National Bureau of Standards to supply every standard, but that the voluntary cooperation of industry is needed if all requirements are to be met. The Bureau endorses this view as a realistic basis for progress in a climate of growing industrial needs and limited public resources, and gladly accepts CORM's proposal of a collaborative industry/NBS program to improve and extend existing radiometric standards. We believe that the Fourth CORM Report constitutes an excellent basis for such a program, and will cooperate to the fullest extent possible in the achievement of mutually established goals.

NBS management assigns a high priority to providing measurement services in optical radiometry and has allocated a fair share of Bureau resources to this area. Even so, the resources available to us are too small to achieve the CORM goals in a reasonable time unless the burden is appropriately shared. In our opinion, there are three areas in which active participation by CORM is needed:

- o Delegation of Industrial Research Associate and Guest Scientists. During the past several years, the programs of the Radiometric Physics Division of NBS have attracted a substantial number of foreign guest scientists who have joined our staff for extended periods of time. In our opinion, the same mechanism should be used to effect a better leverage of NBS resources in the transfer of federally developed technology to the radiometric community of the U.S. The Bureau has several programs under which our facilities can be made available to guest scientists. For the purposes addressed by CORM, the most appropriate of these is the NBS Industrial Research Associate Program, as described in available brochures.³⁾ We invite the industries that comprise CORM to sponsor research associates to work at NBS on the priority items identified in the Fourth CORM Report as suitable for industry/NBS cooperation. Likewise, we invite CORM members affiliated with academic institutions to work with our staff on radiometric problems of mutual interest, or to assign graduate students to the Bureau for thesis work on research required for radiometric standardization.

- o Certification of Intermediate Calibration Laboratories. As suggested in the Fourth CORM Report, intermediate calibration laboratories linked to NBS through appropriate measurement assurance programs could supplement the range of calibrations and standards provided by the Bureau. The formal accreditation of such laboratories is possible through the NBS National Voluntary Laboratory Accreditation Program (NAVLAP).⁴⁾ However, formal accreditation may not be necessary in the instances cited in the Fourth CORM Report where documentation of traceability to standards maintained at NBS is not a legal requirement. We believe that it would be more cost-effective in such cases if CORM, rather than NBS, would accredit laboratories. Suggested procedures for accreditations by CORM are given in Section III.3, below.

- o Technical Task Forces, Workshops, and Intercomparisons.
In a few cases, the requirements stated in the Fourth CORM Report are more stringent than those identified by other sources. When questions of this nature arise, a CORM/NBS task force should be set up to make a reassessment of needs. We believe that this approach would quickly produce positive results, as is demonstrated by the Fourth CORM Report itself and by the recent establishment of a CORM Array Radiometer Users' Group. One of the jobs of a users' group or task force should be to sponsor workshops in which experts discuss specific measurement problems, assess needs and priorities, and suggest solutions. In cases where the state of the art is in doubt, intercomparisons should be conducted to diagnose the problem. Specific needs for task forces will be identified wherever appropriate in this Response.

II. RADIOMETRY

Section II of the Fourth CORM Report has been especially useful to NBS because it focuses on a few, carefully chosen priority proposals for R&D projects with a good probability of succeeding within the framework of available resources. The ranking of these projects confirms information received from other segments of the user community, and is generally consistent with NBS priorities and long-range plans in radiometry. As may be seen from the following progress reports, the Radiometric Physics Division of NBS has already begun to implement several of the CORM radiometry proposals.

AK-1: Spectral Irradiance Standard to 2500nm

In 1973, a new NBS scale of spectral irradiance was realized with a three-fold increase in accuracy over the previous scale. The detection system that was chosen comprises a cooled photomultiplier tube for the 250-800nm spectral region and a cooled lead-sulfide detector for the near infrared. Because of poor signal-to-noise ratios at longer wavelengths, the infrared limit of the scale was set at 1600nm.

We have just implemented an improved method of spectral-irradiance scale realization. The new method is simpler and potentially more accurate than that used before, because the measurement chain from blackbody standards to spectral-irradiance lamps has been shortened through increased reliance on the verified linearity of photomultiplier tubes rather than on numerous temperature settings of transfer lamps. A 1984 re-realization of the NBS spectral-irradiance scale between 250 and 800nm showed no departures from the 1973 scale outside of experimental uncertainties.

Work is under way to characterize the linearity of the lead-sulfide detector, so that the new method can also be applied in the near infrared. However, this in itself will not allow an extension of the scale to longer wavelengths. We have tested the spectral response of our equipment in the infrared and have found that the signal levels obtained in straight lamp-to-lamp comparisons drop so rapidly that the signal-to-noise ratios beyond 1600nm are excessively low. This indicates that a new approach may be needed for extending the spectral-irradiance scale to 2500nm, requiring a larger effort than previously anticipated.

ES-2: Develop a Sustained Source of Radiometric Standards with Rapid Accessibility

We agree that this proposal is timely and should be given a high priority. To explore the problems associated with the establishment of an intermediate laboratory program, a measurement area with a well-defined user community was chosen.

For industrial measurements of geometrically total flux, the only NBS standards needed thus far were incandescent luminous-flux lamp standards. Secondary standard lamps of other types were developed independently by the U.S. lighting industry, and industry-wide measurement quality assurance was provided through intercomparisons conducted by the Lamp Testing Engineers' Conference (LTEC). However, the development of new, energy-efficient lamp types that have numerous emission lines superimposed on a continuous spectrum now requires improved standards for spectral measurements of total flux.

The U.S. lamp companies and NBS, working through LTEC, have formed a task force to study these needs and to formulate a plan to meet them. The task force met several times during 1983 and 1984, and agreed that there is a need for NBS to provide incandescent spectral flux standards and at least one spectrally rich type. A 400W clear high-pressure sodium lamp was deemed most appropriate for the latter. A detailed list of control parameters and measurement conditions for this lamp was formulated.

This additional effort on the part of NBS is considered sufficient to build redundancy into the measurement system. It will allow the establishment of measurement assurance procedures for an intermediate calibration laboratory to issue additional incandescent, metal-halide, and fluorescent lamp standards as required. The measurement capability of the intermediate laboratory is to be verified by LTEC intercomparisons with NBS participation.

The project is now in an equipment procurement phase. The major items are: A spectroradiometer with associated microprocessor, a 3kVA ac line conditioner, and a high-accuracy, true-rms wattmeter. It is presently hoped that laboratory work on the incandescent-lamp phase of the project can begin in 1985.

WS-1: Develop Low-Light-Level Calibration Standards

At present, NBS has no plans to produce low-level standard sources. In the past we have performed special tests on LED's and tritium-activated self-luminous sources, but have not found enough demand for a regular issue of low-level calibration sources by NBS.

Because of the demonstrated linearity of silicon photodiodes, it is now possible to extend the use of absolute detector standards to lower levels. It seems, therefore, that a detector/amplifier package with verified linearity over several decades may have greater versatility than specific low-level sources. Would such a package be useful to a broad community of users? What spectral region is needed most? What is the most useful range of radiant power or irradiance?

We suggest that a CORM/NBS task force be established to determine the standards needs in this area.

ES-3: Evaluate Silicon Detectors as Reliable Standards for Radiometry

The application of solid-state physics as a new theoretical and practical basis for absolute radiometry has been the most important research activity of the Radiometric Physics Division for the past several years. As a result, the feasibility of using silicon photodiodes as reliable radiometric standards for the visible spectrum is now firmly established.

For example, the Division participated in several intercomparisons that have demonstrated full agreement of self-calibrated silicon diodes with other radiometric standards:

- o An electrically calibrated, cryogenic cavity radiometer of the National Physical Laboratory in Teddington, England, was compared at 568nm with an NBS self-calibrated silicon photodiode. Agreement was achieved to within +/- 0.2%.
- o The spectral irradiance of a tungsten-halogen standard lamp was measured with a spectroradiometer that was calibrated at 633nm with a HeNe laser and a self-calibrated silicon photodiode. The ratio of the blackbody-based irradiance to detector-based irradiance was found to be 1.011 with a combined uncertainty of 1.1%.

- o The three-way intercomparison of a silicon-photodiode filter radiometer, a tungsten-halogen spectral-irradiance standard lamp, and synchrotron radiation at 600nm demonstrated the mutual consistency of these three scales within 1%.

The Radiometric Physics Division has also devoted substantial efforts to facilitate the practical application of silicon photodiodes:

- o Collaborating with a domestic detector manufacturer, staff members of the Radiometric Physics Division have developed 100% quantum-efficient detector packages for measurements of incoherent sources in the visible spectral region. This was achieved by forming several large-area silicon photodiodes into a tightly-packed, multiple-reflection light trap in which all of the incident radiation is absorbed within an f/7 cone, thus producing one electron in the external circuit for each incident photon. These detector packages are now commercially available.
- o We have developed a self-contained electronics package for the self-calibration of silicon photodiodes in user laboratories. The package contains channels for high-accuracy dc current-to-voltage conversion and conditioning of diodes, divider circuits, and power sources for forward and reverse biasing. It was developed for the Army Calibration Laboratory at Redstone Arsenal in Huntsville, Alabama, where it is now used for primary radiometric calibrations below 10mW.
- o A collaborative project with the National Standards Institute (NIS) of Egypt is under way to generate a spectral-irradiance scale in the visible spectral region by means of interference-filtered, self-calibrated silicon photodiodes. This scale will in turn be used to generate and maintain absolute photometric scales at NIS. The required optical and electronic equipment has been specified, procured, characterized, and shipped to Cairo with the help of two Egyptian guest scientists. At the present time, the actual realization of absolute photometric scales at NIS is dependent on the resolution of customs delays.

Silicon-diode radiometry is rapidly finding new applications in foreign and domestic standards laboratories and academic institutions. We believe that this technology also offers unique opportunities for industrial applications and, therefore, urge the appointment of research associates to gain hands-on experience by participating in our R&D activities in this field.

WS-2: Near-Infrared Detectors

For the past two years, NBS has provided research grants to the Photovoltaic Laboratory of Purdue University to develop germanium detectors for high-accuracy radiometry in the 600-1600nm spectral region. The program consists of iterative computer modeling, construction, and testing of germanium diodes in an attempt to develop a viable procedure for the fabrication of near-infrared detectors that meet the needs of accurate radiometry. We have received two prototype germanium photodiodes from Purdue University and will evaluate them when the construction of a new, monochromator-based detector characterization facility has been completed.

The quantum-efficiency model used to interpolate the spectral response of silicon photodiodes was modified to improve its accuracy at long wavelengths. Reflectance measurements at infrared laser lines and our model of the reflectance of an air-SiO₂-Si interface were used to determine the loss at the back of the diode for infrared radiation that passes through the diode without being fully absorbed. The term in the collection-efficiency model that describes recombination in the region between the depletion region and the rear of the diode was replaced by a more accurate expression. The net result is that the interpolation procedure is now accurate to +/-1% out to 960nm.

AK-2: Spectral Radiance Standard to 50 μ m

Long-wave infrared (LWIR) radiometry is highly relevant to needs of the Department of Defense (DoD), and consequently most of the LWIR work at NBS is in response to these needs. As may be seen from the NCSL requirements study^{1,2)} the importance of this area is becoming more widely recognized. Low-level ir sources and attenuator performance are of prime interest. We are seeking DoD funding for a major LWIR program at NBS, but have had only limited success so far.

During the past fiscal year, we have built and characterized a highly precise room-temperature blackbody for the Air Force. Its design was based on a blackbody that had been designed about ten years ago at NBS. The blackbody will be used to calibrate a reference room-temperature blackbody for FLIR calibrations. Another calibration involved a blackbody in support of the DARPA Teale-Ruby project.

In the past, we have also performed special calibrations of variable-temperature (200-400K) blackbodies in a cryogenic, high-vacuum environment. Because the cryogenic chamber used for this work was not originally designed for routine testing, a decision was made to build a new chamber, making various improvements such as enlarging the working volume and increasing the detectivity. A new capacitance thermometer and high-frequency signal generator have been ordered with DoD funds, and will be installed in the new chamber.

As a component of our pyrometry program, we have developed a new gold-point blackbody, using a heat pipe to achieve a very uniform temperature distribution. This technology appears to be easily adaptable to variable, lower-temperature (down to about 300°C) blackbodies, and DoD has expressed interest in supporting their further development. One of the intended applications of these blackbodies is their use as spectral-radiance standards for the infrared.

WP-2: Computerized Array Radiometry

In response to the CORM proposal that we evaluate the array-detector approach to radiometry, NBS management authorized the procurement of an array radiometer. We examined two commercial instruments and purchased one during Fiscal Year 1983.

We have studied the linearity of our array radiometer and found that the instrument is capable of radiometric measurements to an accuracy of 1% without applying corrections. This is a significant improvement over an earlier model that we had examined. Below the 1% level, we observed two distinctly different response functions for the odd and even numbered diodes of the array. This can be attributed to the fact that two amplifiers, having different characteristics, are used in this design in order to increase the measurement speed of the array. One amplifier services the odd diodes, while the other is simultaneously servicing the even ones.

As an alternative to the spatial-dispersion devices, we are currently evaluating the feasibility of Fourier-transform array radiometers. The optical design employed in our study is a lateral-shear Michelson interferometer, producing a Fizeau fringe pattern that is detected by a 1024-pixel, linear diode array and optical multichannel analyzer interfaced with a microprocessor for fast Fourier transforms. Initial results are encouraging, and we expect to complete the feasibility study next year. The most promising final design is a compact interferometer consisting of two right-angle prisms joined into a cube, so that the common hypotenuse forms the beam splitter. The throughput of this cube interferometer should be fully characterizable by simple measurements and calculations.

III. SPECTROPHOTOMETRY

NBS welcomes the CORM proposal of a joint effort to establish a measurement support system for intermediate calibration laboratories in spectrophotometry. Because the Fourth CORM Report does not recommend specific steps to achieve this goal, we urge the formation of a CORM/NBS task force to define such action steps and to implement them expeditiously. In our opinion, the task force should:

1. Define the respective roles of NBS and intermediate calibration laboratories;
2. Identify candidate laboratories;
3. Evaluate their measurement capabilities;
4. Specify the standards or calibrations that are needed.

As a basis for discussion, our views on these topics are summarized as follows. Pertinent comments may also be found in a 1977 NBS study of the National Measurement System for Spectrophotometry.⁵⁾

1. NBS and Private Calibration Laboratories

Spectrophotometric standards can be classified into four broad categories, as listed below.

Absolute Standards. Absolute accuracy in spectrophotometry requires direct measurements of radiant flux in relative units, as well as calibrations of wavelength and bandwidth. The standards for such measurements are procedural and documentary in form, and are provided by NBS and other national laboratories. They include:

- o Critically evaluated data of intrinsic properties of physical standards (e.g., wavelengths of emission and absorption lines).
- o Published methodologies for making absolute measurements (e.g., of diffuse reflectance for specified geometries).

- o Recommended procedures for instrument calibrations based on first principles (e.g., linearity testing by beam-addition methods.)

In principle anyone can use these "absolute standards" for spectrophotometry, but in fact few laboratories have the facilities to do so on a large scale. During the last decade, a major effort was made at NBS to develop the high-accuracy instruments and techniques that are required for absolute measurements of a broad range of spectrophotometric parameters. This capability now constitutes the absolute basis for spectrophotometry standardization in the U.S.

Master Standards. Because of the complexities of absolute spectrophotometric measurements, a need exists for standards that have been compared to absolute standards but are themselves similar to test objects. The calibration of a spectrophotometer then becomes a straightforward matter of comparing like objects under like conditions. NBS supplies a large number of "master standards" for this purpose:

- o Standard Reference Materials to ensure the accuracy of spectrophotometric measurements.
- o Reference Data for other materials that are suitable for instrument calibrations.
- o Measurement Assurance Programs for establishing quality control procedures.
- o Special Calibrations of materials or objects for high-accuracy measurements.

The common denominator for this class of standards is that they serve the spectrophotometric user community as a whole. Most of them allow the calibration of highly precise and versatile commercial spectrophotometers that are used in different fields such as analytical chemistry and measurements of color and appearance.

Transfer Standards. In routine applications of spectrophotometry, it is desirable that standards meet certain convenience criteria. For example, solution standards are best for use in analytical chemistry while glass filters are preferred for color measurements. Many of the specific standards needs

cited in the Fourth and earlier CORM Reports fall into this category of "transfer standards." Although we recognize the need for these standards, we believe that their development at NBS would lead to duplication of efforts and would overextend the resources available to us. It is more appropriate that these standards be issued by intermediate calibration laboratories which have a broad measurement capability in spectrophotometry and receive measurement assistance from NBS.

Working Standards. There are dozens of specialized applications in which spectrophotometry is simply one of several test methods. In these applications, the operator cannot pay attention to details and, therefore, needs "working standards" which are specific to his purpose. Examples of these are crossed filters to assess the wavelength accuracy of abridged spectrophotometers, colored ceramic tiles used as standards of chromaticity, or polished glass used as a gauge for gloss measurements. In general, these working standards are so specialized that only a small subset of users is served by their issuance. Their development should be left to private laboratories that specialize in the respective subdisciplines of spectrophotometry.

The main conclusion that can be drawn from this classification of spectrophotometric standards is that the priorities stated in Section III.C of the Fourth CORM Report need to be reconsidered. We agree that NBS leadership is required in the development of absolute standards and master standards, but suggest a stronger industry involvement in the areas of transfer and working standards.

2. Requirements for Calibration Laboratories

At present, the main obstacle to a workable NBS/industry program in spectrophotometry standardization is the lack of private calibration laboratories that can supply transfer and working standards to end users. We believe that the requirements for such laboratories are as follows.

The greatest need exists for laboratories capable of developing transfer standards with a broad applicability in spectrophotometry. The internal quality control of these laboratories should be based on absolute standards to the greatest extent possible. A few private laboratories in the U.S.

have already developed their own high-accuracy spectrophotometers for absolute measurements of selected parameters, such as regular transmittance or diffuse reflectance for one of the standard CIE geometries. Calibration laboratories that wish to issue spectrophotometric transfer standards should be encouraged to do so also. At a minimum, they should employ master standards from NBS to ensure consistency of their calibration equipment and procedures with national standards. Where master standards are not provided by NBS, it may be possible to obtain them from other national laboratories under the auspices of reciprocity agreements that assure the compatibility of the national standards of different countries. A committee of CIE Division 2 is currently compiling a list of spectrophotometric standards that are available from different standardizing laboratories.

Working standards for spectrophotometry could be supplied by the same private laboratories that issue transfer standards. An alternative source would be through specialized laboratories, such as instrument manufacturers and others who adapt transfer standards for applications within a given field. Because the accuracy requirements for working standards are less, the quality control for their development can be based on transfer standards and, therefore, does not require direct measurement assistance from NBS. However, standards organizations should establish control procedures to ensure that the working standards used in different fields are properly correlated with each other and are consistent with national standards. In many cases, NBS staff members are engaged in the work of these standards organizations and will therefore be able to assist in this endeavor.

3. Evaluation of Measurement Capabilities

The Radiometric Physics Division of NBS has developed several Measurement Assurance Programs (MAPs) that can be used to evaluate the measurement capabilities of private calibration laboratories for spectrophotometry. These MAP packages contain artifact standards which are fully characterized by NBS before being sent to a customer's laboratory. The customer measures the artifacts also, and returns them to NBS along with his data. NBS then repeats the measurements, performs a diagnostic analysis of all data, and issues a test report which contains estimates of the random and systematic uncertainties of the customer's measurements. NBS charges a set fee for these services.

The spectrophotometric MAPs that are or will soon be available from NBS are:

- o Spectral Transmittance MAP. A package containing one didymium glass filter for checking wavelength scales from 400 to 750nm, and seven neutral-density filters (0.001 to 0.92 T) for checking transmittance accuracy.
- o Diffuse Reflectance MAPs. One package contains a white ceramic tile (0.85 RF) and a black porcelain tile (0.07 RF) for 6°/hemispherical measurements. The other contains five gray porcelain enamel tiles (0.02 - 0.87 RF) and one black glass (0.001 RF) for 45°/0° measurements.
- o Retroreflectance MAP for Luminous-Intensity Coefficients. A package containing one high-intensity specimen and one engineering-grade specimen of bead sheeting, and one cube-corner retroreflector. Also included are seven filters for spectral tests.

The first two of these are suitable for measurement assurance in the calibration of broadly applicable transfer standards for transmission and reflection spectrophotometry. The last should be useful in the development of additional standards for retroreflection.

Where MAP services are not provided by NBS, blind intercomparisons among calibration laboratories can be used as an alternative method of verifying the performance of laboratories. In our opinion, the following steps are necessary for conducting such intercomparisons:

- o The CORM/NBS task force determines the objects to be intercompared and establishes control parameters. This can be done in cooperation with voluntary standards organizations, such as ASTM or ANSI.
- o CORM convenes the intercomparison. NBS participates as a reference laboratory on a fee basis, thus providing a tie to national standards.
- o CORM performs the data analysis. If the intercomparison is done for accreditation purposes (see below), CORM documents the results by publication in the archival literature or in the CORM Newsletter.

The issue of a MAP report by NBS or its participation in an intercomparison cannot be construed as Bureau endorsement or accreditation of laboratories. As already mentioned, a formal accreditation of private laboratories is possible through the NBS NAVLAP program but might not be necessary if the demonstrated competence of a laboratory is sufficient for user acceptance of standards. In these cases the accreditation of laboratories by CORM, based on the results of MAPs or intercomparisons, may be possible. We suggest that criteria and guidelines for such accreditations be established by the CORM/NBS task force. The requirements for accreditation should include:

- o Definitions of quantities measured, physical principles of measurements, instruments used, etc.
- o Requirements for measurement assurance (MAPs, intercomparisons, statistical controls, operator requirements, etc.)
- o Procedures for accreditation, renewals, and terminations.

4. Establishment of Needs

The extensive list of "Specific Needs" in Section III.D of the Fourth CORM Report emphasizes the urgency of establishing private calibration laboratories for spectrophotometry. However, the list is apparently not prioritized and, in a few cases, states requirements that seem beyond the state of the art in spectrophotometry. For example, the required absolute accuracy for spectral reflectance scale standards is given as 0.1% from 200 to 2500nm whereas the current NBS capability is only 0.15% in the visible and increases to 1% at the stated uv and ir limits.

These and other questions regarding the establishment of a national standardization system for spectrophotometry should be resolved by the CORM/NBS task force. Specifically, the task force should:

- o Prioritize Needs. The need for spectrophotometric transfer and working standards should be ranked in the same way as outlined in the Introduction to the Fourth CORM Report; i.e., in terms of technical impact as well as availability of resources. The availability of services from NBS should be considered together with the capabilities of private calibration laboratories. Those areas should be identified where urgently needed standards can be developed most expeditiously.

- o Conduct Intercomparisons. The list of "Specific Needs" in the Fourth CORM Report is likely to overwhelm even the combined efforts of NBS and CORM. It appears that the list can still be narrowed. In order to decide which standards and SRMs are most needed, carefully organized intercomparisons could be conducted. NBS can serve as a reference laboratory in these intercomparisons.
- o Identify Measurement Assistance Needs. NBS needs to be informed if presently offered master standards are insufficient to support the development of transfer standards by private laboratories. We offer the use of our facilities to industrial research associates for the development of needed standards or measurement assurance procedures, if these cannot be provided by NBS staff.
- o Assist in Transfer of NBS Technology. NBS currently offers services for specialized applications of spectrophotometry, such as measurements of haze, gloss, and optical density. Our long-term goal is to gradually reduce the number of these services, allowing them to be done by private laboratories. Proper procedures for effecting this transfer of technology must be established.

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May 1983
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Survey of the NCSL, NBSIR 84-2847, March 1984
- 3) Inquiries regarding the NBS Industrial Research Program may be
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- 4) Inquiries regarding the NBS National Voluntary Laboratory
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Mr. J. W. Locke, Manager
Laboratory Accreditation
National Bureau of Standards
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(301) 921-3431
- 5) The National Measurement System for Spectrophotometry,
NBSIR 75-940, November 1977

COUNCIL FOR OPTICAL RADIATION MEASUREMENTS

FOURTH REPORT TO THE NATIONAL BUREAU OF STANDARDS

PRESSING PROBLEMS AND PROJECTED
NATIONAL NEEDS IN OPTICAL
RADIATION MEASUREMENTS

AUGUST, 1982

I. INTRODUCTION

In preparing its Fourth Report on Pressing Problems and Projected National Needs in Optical Radiation Measurements, the Council for Optical Radiation Measurements (CORM) has revised its previous approach to the subject in an effort to produce a more realistic assessment, not of the problems themselves, but of the means for their practical and expeditious solutions.

In its past reports, CORM listed at length the needs it perceived in the optical radiation community, identifying many specific needs for standards and procedures, all of which were presented to the Bureau. The objective, of course, was to define those major needs so that the NBS in its planning would have the opportunity to develop new programs or incorporate the requested needs into their existing project scopes. Subsequently the Bureau advised CORM that the very wide range of requests diluted the planning effort and created uncertainty when those requests from CORM were applied to the NBS programs in the planning process. Even so, the NBS response to those requests was highly significant.

To avoid this difficulty, and with the realization that no one organization can possibly respond to all needs at all levels, the CORM Directors undertook for the Fourth Report two steps not incorporated in previous reports. The first dealt with the assignment of priorities. Here, areas of technology were first studied to identify the most significant fields in terms of standards needs and industrial implications. Second, once those areas were identified the most significant needs were defined, and finally those needs were ranked in order of priority.

Second, the CORM Directors studied the identified national needs in terms of ways of achieving them in the most effective and efficient manner. The Directors, after many meetings with the Bureau program managers and scientists, suggested that some of the requests for standards could only be acted on effectively by the Bureau in its role as the national laboratory. Other needs could be achieved most effectively through the coordinated efforts of the NBS and segments of industry, and there were still others which the users themselves could solve through programmed efforts, asking the Bureau only for guidance or consultation.

The Directors prepared a listing of standards needs and found that the most essential projects were in either of two technical categories, spectroradiometry or spectrophotometry. Having made this division, CORM established two Task Forces, one in each of the technical areas, and charged them with the responsibility of identifying only the most essential standards needs within their respective areas. These standards were then studied and statements of the specifications and technical implications were prepared for each one. These standards needs were ranked in priority and recommendations were prepared as to which of the three paths would be the most effective and realistic in achieving the goal.

Details of the specifications and technical implications of standards needs in each of these areas form the body of this report. The report reflects, to the best of our understanding, the consensus of those who use optical standards in the measurement of radiant energy. Before preparing the report, CORM established criteria to assure that the users' needs stated in the report, needs involving specific standards and their specifications, were the most essential to industry in the conduct of their business.

CORM has as its purpose establishing and defining a consensus of the industrial and academic requirements for physical standards and calibration sources in the field of optical radiation measurements. The Directors urge that this report be seriously considered by the Bureau as well as by industrial and academic standards users in order to improve the adequacy of the required standards. CORM and NBS should work together to improve the present state of the art of optical radiation measurements by providing needed standards and by increasing national expertise through technological advances.

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II. CURRENT AND FUTURE NEEDS IN RADIOMETRY

A. Selection of Needs and Assignment of Priorities

We report the findings of the CORM Radiometry Task Force, which was appointed to determine and assign priorities to the most critical needs of the nation's radiometry-photometry community. The task force gathered its data and assembled proposals for programs which, if carried out, would each satisfy a particular area of need. Each proposal had a specific technical objective, and approaches were suggested for meeting it. Documentation was provided on which user groups would benefit from the programs and on their technical, economic, and strategic impact. We also stated whether CORM feels that NBS should carry out the program, whether it is a responsibility of the user community represented by CORM, or whether it should be a joint effort.

A formal scoring procedure was followed to evaluate the proposals. The Task Force then met to review the scores and debate the merits of the proposals in order to arrive at a group consensus of priorities of the proposals. The decisions were made based on the following factors.

1. Degree of need; breadth and depth of need;
2. Appropriateness; compatibility of the proposed program with CORM-NBS objectives;
3. Probability of succeeding in the proposal's objective within the framework of reasonably possible CORM-NBS resources;
4. Immediacy of urgency of need.

Seven of the proposals submitted were placed on the priority list. They were grouped in three classifications:

- Priority 1: CORM feels that immediate action is necessary;
- Priority 2: These are also important but not as immediate as Priority 1 items;
- Priority 3: These are back-burner items; they will be important in the future.

We present a synopsis of the results of the study in Table I. The priority proposals are summarized in section B.

B. Priority Proposals

AK-1: Spectral Irradiance Standard to 2.5 Micrometers (Priority 1)

The objective of this proposal is to provide a lamp standard of spectral irradiance to at least 2.5 micrometers. The 1973 NBS scale improved the accuracy of the 1963 NBS scale over the range 0.25 to 1.6 micrometers, but did not cover the region from 1.6 to 2.5 micrometers. The two scales differ by several percent at the 1.6 micrometer point, so an attempt to provide a calibration that covers both regions results in a discontinuity at 1.6 micrometers which has no real basis. Alternatively, the 1963 scale may be used entirely, but it is known to be in error below 1.6 micrometers.

TABLE I. CORM Radiometry Proposal and Priorities

Proposal	Priority	Proposal title
AK-1	1	Spectral irradiance standard to 2.5 micrometers
ES-2	1	Develop a sustained source of radiometric standards with rapid accessibility
WS-1	2	Develop low-light-level calibration standards
ES-3	2	Evaluate silicon detectors as reliable standards for radiometry
WS-2	2	Near-infrared detectors
AK-2	3	Spectral radiance standard to 50 micrometers
WP-2	3	Computerized-array radiometry

Users of irradiance standards in this region of the spectrum include almost every industry, agency, and university deeply involved in radiometric measurements. A customer list from a major calibration laboratory covering the last two years includes 27 industries, 11 universities, 8 government laboratories, and 6 foreign countries. 80% of customers purchased the 1.6 to 2.5 micrometer calibration range which is an extra cost option.

The economic impact of the confusion resulting from the scale inconsistency is difficult to determine, but it is not unreasonable to estimate that it bears on the quality of products representing several millions of dollars in sales. Research which relies on near-infrared calibrations can impact on large markets. There has recently been increased activity in this region of the spectrum, for example, with the push toward the infrared in fiber-optic communications.

We feel that this work would not require a large effort and should be performed at the NBS as part of their continuing improvements and extensions of measurement scales.

ES-2: Develop a Sustained Source of Radiometric Standards with Rapid Accessibility (Priority 1)

The proposed technical objective is to develop an organization or system from which the standards users can quickly and easily obtain radiometric standards of total flux, ultraviolet and visible spectral irradiance, and geometric total flux for a number of lamps.

Beneficiaries would include all those users of radiometric standards who often urgently need lamp standards but who now must wait long periods of time before calibrated sources are available.

All areas of source-based radiometry would be impacted by the proposed development. The lamp industry, for example, would benefit substantially. This industry is beginning to be a factor in international trade in the 1980's as several foreign lamp manufacturers are moving into the United States markets and local companies are entering foreign markets. Our ability to market abroad depends on the quality of our standards.

One approach to this problem that has received favorable acclaim is to adopt an intermediate standards laboratory program, monitored by the NBS, which has proven itself in other technical disciplines to be capable of providing standards with the authenticity of the national laboratory. Users of lamp standards who have been contacted are almost unanimously in favor of such a system.

Such a program should be carried out as a cooperative effort between the NBS, CORM, the standard users, and the participating intermediate calibration laboratories. The initial costs of the system to these intermediate labs must be carried by them on a venture basis, with the standard users ultimately paying for these costs and profits by the purchase of standards and calibrations. The NBS would have the responsibility of accreditation of the intermediate laboratories by providing adequate guidance and conducting intercomparisons. The

role of CORM in this process would be to determine which standards and services are most needed and to communicate this to NBS and the intermediate labs.

WS-1: Develop Low-Light-Level Calibration Standards (Priority 2)

Present irradiance standards have irradiance levels of $10^{-5} \text{W cm}^{-2} \text{ nm}^{-1}$ in the visible region. It is impossible to use these standards to calibrate instrumentation measuring level 5 to 10 decades lower. Accordingly, a line of low-light-level calibration sources which can be used as standards of spectral irradiance, spectral radiance, luminance, and illuminance is needed to satisfy these requirements.

Beneficiaries would include about 40% of the users of calibrated radiometric standards, representing industries, agencies, and academic institutions. Areas which routinely involve low-light-level measurements include fluorescence, phosphorescence, atomic and molecular spectroscopy, flame and plasma diagnostics, star tracking, LEDs and miniature lamps, display screens and arrays, astronomy, optical countermeasures, photograph, television, bioluminescence, and more.

This development would require a medium sized effort at the NBS carried out over a several year period.

ES-3: Evaluate Silicon Detectors as Reliable Standards for Radiometry (Priority 2)

The objective of this program is to evaluate the feasibility of using silicon photodiodes with enhanced ultraviolet response as standards in spectroradiometric measurements. CORM realized that there will always be a need for source standards. In many cases, however, detector-based radiometry can be used situations where source-based radiometry was traditional, offering many advantages in cost, stability, portability, and ease of intercomparison and measurement assurance program (MAP) services. The program for the development of self-calibrated silicon-detector radiometry at the NBS has made great strides in advancing detector-based radiometry. Its usefulness for measuring well-controlled monochromatic sources such as lasers is a reality. It remains to adapt this technique to the broadband, wide-angle applications most often encountered in applications of spectroradiometry.

The impact of the achievement of broadly applicable detector radiometry would be felt throughout the entire field of radiometry. The achievement of this goal can be accomplished by a long-term cooperative effort between the NBS and the radiation measurement community. CORM gives its full support to the continuing commitment that NBS has made to detector-based radiometry, and points out that several CORM members have begun using self-calibrated silicon detectors in practical applications of spectroradiometry. This foothold of acceptance is an opportunity for NBS to broaden the impact of its accomplishments by structuring the detector programs so as to support and complement these early users. We feel that it is the role of CORM to encourage the interaction of the NBS and users by providing a forum for communication and interchange at our CORM conferences.

WS-2: Near-Infrared Detectors (Priority 2)

The objective of this proposal is to improve detector performance in the 0.9 to 1.7 micrometer range, evaluating germanium and other detectors for this purpose. As mentioned earlier, the near-infrared is the focus of much recent activity, but stable, efficient detectors are not available beyond the 1 micrometer limit of silicon photodiodes. We feel that it is important to evaluate the alternative detectors that have been used or suggested for this region of the spectrum, to identify the major technical obstacles to their use in detector radiometry, and to determine approaches to overcoming these obstacles.

The fiber-optic industry would benefit from this development, and its competitive position against Japan and Europe could be improved by our technical leadership in this area.

Most of this work should be carried out at the NBS, but detector manufacturers can provide a valuable contribution to this investigation by supplying the NBS with detectors, especially large-area devices made to custom specifications.

AK-2: Spectral Radiance Standard to 50 Micrometers (Priority 3)

The objective is to provide a blackbody-type source for calibrating infrared detection systems in the 2.5 to 50 micrometer range, possibly approached in steps of 15, 25, then 50 micrometers.

This would impact on earth-resources data collection, military surveillance, meteorology, and remote sensing of extraterrestrial bodies, providing benefits to the world's food supply, long-range weather forecasting, and defence and astronomical capability.

A joint effort is suggested between the NBS and other agency users of far-infrared radiation, such as the Department of Defense.

WP-2: Computerized Array Radiometry (Priority 3)

The objective of this proposal is to bring the NBS into array-radiometry technology. This technology has developed over a period of years mainly as a research tool, but it is beginning to be used in calibrations and commercial radiometric measurements. In the future the quality and credibility of measurements made with these devices will have increasing impact on business and trade. Involvement of the Bureau in this field will bring to the surface various difficult and unanticipated problems that may accompany the introduction of these techniques to standard-quality measurements. We feel that this objective need not be an end in itself, as if for example the NBS acquired instruments from several manufacturers and evaluated them. Rather, we think that the Bureau should use the array-detector approach in the instrumentation needed to accomplish its other objectives in the radiometric area.

The resources for accomplishing this objective must come from the capital budget of the NBS, utilizing array technology in the new instruments rather than conventional approaches. Although initial costs might be higher, they would be offset to some extent later by the benefits of efficiency, speed, convenience, and flexibility that can be obtained with array devices.

III. A NATIONAL MEASUREMENT STANDARDIZATION SYSTEM FOR SPECTROPHOTOMETRY

A. Introduction

By spectrophotometry we mean the quantitative application of measurements of spectral transmittance, reflectance, and luminescence, and related quantities, in the near-ultraviolet, visible, and near-infrared wavelength regions. This type of metrology is of vital importance to many materials-producing and using industries for research and development and for quality control; to many operations involving clinical (health) analysis; and to the analysis of many processes and materials related to energy conservation, to name only a few applications.

Aspects of standardization related to spectrophotometry have long been neglected for various reasons. Foremost among these may be a misunderstanding of the complexities associated with this type of metrology; the opinion that spectrophotometry is no more than a simple ratio measurement is far from realistic. Two examples may be given of the support in depth of the standardization needs in spectrophotometry:

1. The United States Army wishes to have spectrophotometric methods of setting color tolerances for uniforms. The Army has been accepting shipments of textiles to be made into uniforms on a visual basis. The inspector is provided with a standard sample and eight limit samples representing maximum allowable deviations from the standard in each of eight directions in color space. Armed with these nine samples, the inspector examines the submitted sample and issues a pass-or-fail verdict. This visual method has proved troublesome, as evidenced by frequent disagreements between the Army and the textile mills submitting the samples. The Army wishes to change its acceptance procedure to one using instrumental measurement and valid tolerance limits for acceptance.

This example is typical and represents several unsolved problems that need attention. All three aspects of standardization (calibration, systems tests, and diagnosis) must be carried out with great care for this application. As documented in the Third CORM Report (items* 1 and 5), adequate standard reference materials (SRM's) for the calibration step, needed by both the instrument manufacturers and the users (the Army laboratories in this example) do not exist. In this case the systems check standards must be colorimetric in nature since it is necessary to correlate instrumental results to visual judgments. Such material standards were called for in the Third CORM Report (item 6). In their absence, reliance has been placed on equivalent standard materials obtained from foreign national standardizing laboratories. Delays result from United States Customs restrictions, costs are high, continuing supplies cannot be assured, and the available standards do not meet the specific need for checking color differences. Finally there are no adequate diagnostic standards for spectrophotometry available in the United States, the last ones having disappeared when the supply of SRM 2101-2105 was exhausted and the NBS decided not to replace them.

* Numbers refer to items in Section IIID of this report, repeated with abridgement and revision from Section B of the Third CORM Report.

2. A large United States industrial firm wishes to replace material standards for an important product line with stored numerical standards in computer-controlled spectrophotometers. Because of the difficulty in maintaining for long periods of time relatively fragile product standards, subject to day-to-day use in a production environment, it is felt that product uniformity, critical in this application, can be improved by replacing product material standards with the stored numerical equivalent. The investment in the necessary equipment has approached \$500,000. Tests of the system over several years have pinpointed inadequacies in the same three types of material standards mentioned in the first example: calibration, systems check, and diagnostic. Lacking these, the goal of the undertaking has not been satisfactorily achieved.

While the above examples refer to material goods, there is another area of use of spectrophotometers that gives rise to even greater concern: its increasing use as a diagnostic tool in many medical and health-hazard applications, where the precision and accuracy of these radiation-measuring devices is, often literally, of vital importance.

A survey estimates that there are over 32,000 reflectance and fluorescence spectrophotometers of various types in use in various installations. One can easily estimate that there are many more transmission (analytical) spectrophotometers in use. That only a small fraction of these instruments could meet government specifications for accuracy and traceability is clear from existing inconsistencies when results are compared from one spectrophotometer to another. Usually, instrument calibrations and measurement quality-assurance tests are employed to achieve consistency. In the case of spectrophotometry and spectroradiometry, the proper use of reference materials can serve this purpose. The current system of measurement standardization lacks the availability of key calibration and measurement quality assurance tests that are needed for effective, equitable control of spectrophotometric metrology without unnecessary economic burden.

B. Objectives

CORM believes that a collaborative industry-NBS program should be set up to improve the deficiencies that exist in spectrophotometer standardization. The goal of such a program should be to improve services from NBS to the private, state, and federal sectors related to spectrophotometric measurement in a manner to assure safety, compatibility, and traceability, consistent with the economic health of the nation.

The overall objective is to remove at least major deficiencies in the key areas of metrology and to develop and implement a national measurement support system that will enable periodic demonstration of the adequacy of spectrophotometric measurements made routinely at the field level. When implemented, such a system will assure adequate measurement quality by providing the mechanisms needed to achieve consistency with national and international standards.

Secondary or intermediate standards calibration laboratories, certified by the NBS, are badly needed to provide the day-to-day workings of measurement consistency.

CORM feels that the proposed program should be carried out in cooperation between intermediate laboratories and NBS. By doing so the burden will be appropriately shared between industry and NBS. If properly executed such a program will:

1. Establish intermediate calibration laboratories;
2. Design and test SRM's that can be used as transfer standards;
3. Provide guidelines on methodologies for measurements;
4. Provide training programs.

C. Implementation.

First of all, the most urgent areas of deficiency should be specified and the role of NBS and that of the industry and academia in the program should be established in each of the areas. The procedure to achieve the goals in a reasonable time must be established. The four essential components of the suggested program are:

1. A MAP service in spectrophotometry
2. SRM's for instrument standardization for routine spectrophotometry
3. SRM's for special measurements
4. New methodology for spectrophotometry

In the first two items NBS leadership is required; item three would be a joint venture (NBS-CORM), and in the fourth one the NBS participation is sought. The plans for implementation of this proposed program should be worked out jointly with NBS representatives and the CORM Board of Directors.

1. A MAP Service in Spectrophotometry (Priority 1)

NBS should provide and maintain on a continuing basis a MAP service in the area of spectrophotometry. It is anticipated that establishing such a service will provide the basis for the national standardization program in spectrophotometry previously referred to. It should adhere to the concept of measurement traceability promulgated by the NBS's Ionizing Radiation Section.

Establishment of this service will require the identification of laboratories providing accurate calibration services to users of spectrophotometry. These laboratories will serve as intermediate standards laboratories in the measurement traceability chain. Their own measurements will be monitored by the NBS through the MAP procedure, and they in turn will monitor the measurements performed in user laboratories by similar mechanisms.

It is anticipated that CORM can play a major role in the establishment of these quality assurance programs by seeking out and identifying suitable candidate intermediate standards laboratories. These may come from several sectors: government laboratories, for example the United States Army laboratory responsible for development of the program for color tolerances for uniforms cited above; industrial laboratories, for example those responsible for coordinating coloring efforts in a series of manufacturing plants; academic laboratories; producers of spectrophotometric instruments; and private standardization laboratories.

What is required from NBS is the provision or at least primary calibration of the necessary SRM's, the final selection and certification of the intermediate standards laboratories, the implementation of the necessary performance tests, and oversight of the development of the relations between the intermediate standards laboratories and the user laboratories. Once the system is operating, it is anticipated that the NBS's effort would be minimal in comparison to the overall benefits of the program.

It is anticipated that CORM could play a continuing role in monitoring and promoting the national standardization system for spectrophotometry, relieving the NBS of a considerable burden in this respect.

2. SRM's for Instrument Standardization for Routine Spectrophotometry (Priority 1)

The purpose of this objective is to insure the provision to both the intermediate standards laboratories and the user laboratories of the calibration, systems check, and diagnostic standards required for satisfactory operation of the measurement traceability scheme. Unfortunately virtually none of these standards now exist, either as SRM's or as identified materials. In achieving this objective more than any other, NBS leadership is required. CORM will assist as much as possible in identifying needs and possible sources of supply. A partial listing of needs in this area follows, with references to the Third CORM Report where appropriate.

a. Calibration Standards

(1) Wavelength. As described in the Third CORM Report (item 5), standards based on absorptance maxima of the didymium and holmium oxide filters, including SRM's 2009, 2010, 2013, and 2014, fulfill only a small part of the need. Additional standards based on the linear-filter concept are also required, and possibly others not yet identified. The goal of the national standardization system should be to provide SRM's suitable for calibrating the wavelength scales of all spectrophotometers -- analytical as well as color-measuring -- regardless of spectral bandwidth, including those utilizing both conventional prism and grating monochromators and those utilizing interference filters or interference wedges as monochromators, array detectors, or other technologies.

(2) Photometric. Transmittance scale standards suitable for the calibration of the photometric scales of spectrophotometers were called for in the Third CORM Report (item 2). The deficiencies of existing SRM's (e.g. 930) and recommendations (e.g. dichromate solutions) were pointed out. Other SRM's that might be suitable for this purpose have either been discontinued (2101-2105) or are under consideration for discontinuation (items 7.8B-E in NBS SP 250). A few suitable reflectance-scale standards exist (three of twelve ceramic tiles supplied by the National Physical Laboratory in England) but the supply is expected to be exhausted in 1982 and it is not clear when suitable replacements will be available. In any case, the applicability of reflectance-scale standards is limited rather than general. (This series of standards is discussed further under item b.)

(3) Reflectance. For reflectance measurement additional calibration standards are required to set the level of the photometric scale. Here the need is not so much for photometric linearity standards, since this can usually be accomplished by transmittance-scale standards. Rather, what are required are suitable transfer standards for the calibration of user working standards in terms of the international primary standard of reflectance, the perfect reflecting diffuser. These are required for the four internationally recommended measuring geometries. While some progress has been made in establishing international agreement for some of these geometries, this work needs to be brought to completion and the necessary SRM's developed.

b. Systems Check Standards

Since all the types of systems check standards that would be required in the national standardization system have not been identified (e.g. clinical, analytical, densitometric, colorimetric, etc.), one class only will be discussed: colorimetric systems-check standards. The need for these was also pointed out in the Third CORM Report (item 6). As noted there, it is extremely regrettable that SRM 2101-2105, the only set of such standards available from NBS, was discontinued, particularly so shortly after the excellent recalibration study that significantly enhanced their utility. At the very least a replacement set of SRM's, free from the defects known to exist in the previous set, should be provided.

Because of the differences in geometry, an additional set of systems-check SRM's for reflectance is required. In the past, leadership in producing such standards has been taken by the NPL in England, but as mentioned in item a(2) the supply of these standards is near exhaustion. A replacement set is being developed which should ultimately be even more useful, but full details are not yet available. Procurement will no doubt continue to be hampered by customs restrictions that, we are informed, effectively prevent these standards being imported for some important uses. The possibility of NBS working in cooperation with NPL to make the new standards available as SRM's should be explored.

Systems check standards for color densitometry were also called for in the Third CORM Report (item 4).

c. Diagnostic Standards

Diagnostic standards are the least developed of the Priority 1 items required for a national measurement system for spectrophotometry. In some cases systems check standards can also serve as diagnostic standards. Set 2101-2105 was originally designed with this use in mind, for example. Nevertheless, it is not certain that all of the needs for diagnostics have been identified, and the required standards and methodology developed. CORM should play an active role in supplying this information to the NBS.

Among the defects in spectrophotometers for which diagnostic standards are needed are: stray light, spectral bandpass and slit-function shape, and (for integrating-sphere reflectance geometry) condition of specular-inclusion ports.

3. SRM's for Special Measurements (Priority 2)

The usefulness of a national standardization system for spectrophotometry should not be limited to the routine determination of transmittance, reflectance, and related quantities of specimens that pose no special measurement problems. It should be obvious that the greatest need for the system will be in the solution of difficult rather than simple measurement problems. The reason that the development of SRM's for these special measurements is assigned Priority 2 is merely that the establishment of the system and its implementation for the simpler cases must of necessity come first. Among the measurements that require special consideration and special SRM's are: fluorescence, retroreflection, translucency, specular reflectance including gloss, and extremely low transmittance.

a. Fluorescence

There are two aspects of the measurement of fluorescent specimens to be considered: the spectrofluorimetry of fluorescent liquids and the spectrophotometry and colorimetry by reflectance of fluorescent solids. The latter is discussed here by way of example. The topic was the subject of a symposium at the CORM 81 International Conference on Optical Radiation Measurement of Fluorescent and Retroreflective Materials, at which one paper dealt specifically with fluorescent standards. International activity in the development of methods for the spectrophotometry and colorimetry of reflecting fluorescent materials continues and is closely monitored by CORM. Specific recommendations for SRM's, supplementing the request in the Third CORM Report (items 7 and 8), will be developed in the near future.

b. Retroreflection

The situation for the measurement of retroreflection is not unlike that for fluorescence in that rapid progress in the establishment of the necessary techniques is being made at both the national and international levels. This work was also discussed at a symposium at CORM 81, and is being closely monitored by CORM. More specific needs for SRM's, probably in the form of systems check and diagnostic standards, supplementing the request in the Third CORM Report (items 9-11), will be developed in the near future.

c. Translucency

One of the most difficult series of measurements by spectrophotometry is that of diffuse transmittance, required together with the determination of diffuse reflectance for the characterization of the translucency, opacity, and related light-scattering properties of translucent specimens. In part new standard methodology is required, and to this extent the problem falls under objective 4. The need for SRM's when the necessary methodology is developed remains to be addressed in the future, supplementing item B6 in the Third CORM Report.

d. Specular Reflection and Gloss

The recent development of SRM's for specular reflection (in response to item B11 in the Third CORM Report) has addressed part of the need in this area, but more needs to be done, particularly in the transfer of these results

to the calibration of scales of gloss. Although it may be argued that this is not directly an application of spectrophotometry, nevertheless experience dictates that measurement and control of gloss is essential for the accurate spectrophotometry of reflecting materials, so the two cannot be fully separated. Additional requests for SRM's can be anticipated in the future.

e. Extremely Low Transmittance (High Optical Density)

The need for SRM's of high optical density was addressed in the Third CORM Report (item 3). The need remains, and should be addressed consistent with the Priority 2 level assigned.

4. Methodology for Spectrophotometry (Priority 2)

It should be clear from the above discussion that the development of new methodology for spectrophotometry and in related areas is required for the solution of several difficult measurement problems. In addition to those already mentioned, three already identified are mentioned here.

a. Evaluation of Metameric Pairs

Evidence from the field clearly shows that there is a highly significant difference in the performance of spectrophotometers for the colorimetric evaluation (in terms of color differences) of nonmetameric and metameric pairs of specimens. The uncertainty of measurement of the color difference between the members of a metameric pair is approximately an order of magnitude greater than for a nonmetameric pair. Although it now appears that this reflects poor practice in the calibration of the instruments, further study, with the possible development of new methodology, is required. CORM is prepared to take the initiative in this work.

b. Definition of Geometry of Measurements

The precise definitions of the geometric conditions for reflectance and transmittance measurements have never been made. The advantages of doing so can be inferred from the significant improvements achieved in the field of densitometry when the corresponding definitions were made. International standards for these geometries are inadequate, but there is no indication that the problem will be addressed in the near future at the international level. CORM is prepared to take the initiative in this work. The related problem of the inclusion or exclusion of the specular component of reflectance will also be addressed.

c. Advancement of the State of the Art by Work on Objectives Not Yet Identified

CORM wishes to be informed of additional objectives in all aspects of the field of spectrophotometry for which new methodology or new standards are required.

D. Specific Needs

Specific needs and problems in spectrophotometry were clearly identified in the Third CORM Report (June 1979). Seventeen items were listed in that report but only two of them have so far been made available. The NBS has provided the SRM of absolute white spectral reflectance (item No. 2 in 1979 CORM Report) and also the SRM for regular (specular) spectral reflectance (item No. 11 in 1979 Report). The needs for the remaining items have neither changed nor diminished in the interim, as has been indicated in the foregoing discussion. For the convenience of those wishing to refer to these items, they are summarized below with emphasis on the intended use and technical specifications of the proposed material standards.

1. Spectral Reflectance Scale Standards

a. Description

Physical standards of diffuse spectral reflectance at various reflectance levels are needed to establish a scale. These standards are required for checking the linearity of the photometric scales of reflectance spectrophotometers.

b. Specifications

Reflectance Levels	Initially, 0.10, 0.50, and 0.90 nominal ($\pm 5\%$). Other levels to be provided later
Spectral Character	As near neutral as possible
Geometry of Calibration	0/45 and d/0
Wavelength Range	360-740 nm; extended to 0.2-2.5 μm later
Uncertainty	0.1% absolute
Physical Size	50 \times 50 mm
Diffusing Characteristics	Either approximately Lambertian from 0° to 60°, with no specular peak, or highly glossy (polished glass) with approximately Lambertian diffuse reflectance and narrow specular peak

2. Regular Spectral Transmittance Scale Standards

a. Description

Physical standards of regular spectral transmittance at various transmittance levels are needed to establish a scale. These standards are required for checking the linearity of the photometric scales of transmittance spectrophotometers, including analytical instruments. The dichromate solutions recommended as transmission standards (NBS SP378) are neither convenient nor stable. Permanent solid standards are required. Both the manufacturers of spectrophotometers as well as the users need these regular spectral transmittance standards. The majority are not happy with presently available SRM 930 standards. Glass must be more carefully selected, and uncertainties must be reduced.

b. Specifications

Transmittance Levels	Initially, 0.1, 1, 10, 50, and 90% nominal. Others to be provided later.
Spectral Character	Nominally flat for at least 2 nm on either side of the calibration wavelengths
Geometry of Calibration	0/0
Wavelength Range	350-1100 nm; 0.2-2.5 μm later
Uncertainty	0.1% of value
Physical Size	Both 50 \times 50 mm and 8 \times 29 mm
Diffusing Characteristics	Fully transparent and haze free

3. Visual Diffuse Transmission and Reflection Density Standards

a. Description

Visual diffuse transmission and reflection density standards are needed with the following specifications. These standards are used by the photographic industry to calibrate densitometers which are in turn used to evaluate and control the production of photographic film. They are also used to calibrate other modulators or attenuators which are used in the exposure of film prior to densitometry. At present NBS provides a transmission density standard of limited uncertainty as a standard reference material. More accurate special calibrations are also available. However, NBS now has essentially no capability to provide reliable visual diffuse reflection density standards or calibrations. The need to develop some capability in the reflection density area is especially critical.

In practical transmission densitometry an opal glass is often used in the optical system in place of an integrating sphere. Densitometry measurements made with an opal glass do not agree with those made with an integrating sphere, principally at low densities. To resolve the differences between the two methods, it is recommended that

(1) The practice of densitometry be reviewed regarding usage of integrating spheres and opal glasses, the significance of the measurements made by these two methods to the user, the reproducibility which may be achieved by these two methods, and any recommendations being made by ANSI committees.

(2) Using a variety of samples, agreement be explored between the currently used photoelectric densitometer method and the inverse-square-law procedures; and, where necessary, the measurements from these two techniques be brought into agreement.

Measurement laboratories using NBS calibrated samples measured by different methods now experience difficulty in making measurements that agree. Since most densitometers routinely employ an opal glass and since opal glass density measurement correlates well with the density seen by many photographic printing operations, a standard calibrated by an opal glass method would serve a real need.

b. Specifications

Reflection Density:

Range	0 to 2.5 density
Accuracy	0.5% of density
Spectral	V_{λ} response detector
Geometry	45/0
Source	3000 K \pm 100 K
Materials	Selected ceramic tiles (spectrally neutral, nonfluorescent)

Transmission Density:

Range	0 to 6 density
Accuracy	0.5% of density
Spectral	V_{λ} response detector
Geometry	0/integrating sphere and 0/opal glass
Source	3000 K \pm 100 K
Materials	Silver photographic step tablet and/or carbon step tablet

4. Standards for Calibration of Transmission and Reflection Density for Color Materials

a. Description

Standards should be provided for the calibration and check procedures for densitometers used in three-color densitometry. These standards are used in photographic printing and graphic arts industries to calibrate densitometers that are used in quality control of these products. There are many such densitometers in the field yet there is no SRM available for validation of these instruments. This results in large variations in measured values from laboratory to laboratory and densitometer to densitometer. Leadership is required in the realization of ANSI standards and to suggest possible alternatives to the documentary standard if this standard is not readily realizable.

Samples of neutral areas of three-layer color reversal and color negative film should be calibrated for red, green and blue densities as specified in ANSI PH 2.1-1952, and also using specified color filters, such as Kodak certified AA and MM filters for the measurement of Status A and Status M density.

b. Specifications

Reflection Density:

Range	0-2.5 density
Accuracy	0.5% of density
Spectral	Status A, Status M, PH 2:1-1952
Geometry	45/0, 0/45
Source	3000 K \pm 100 K
Materials	Suitable color step tablets

Transmission Density:

Range	0 to 6 density
Accuracy	0.5% of density
Spectral	Status A, Status M, PH 2:1-1952
Geometry	0/integrating sphere and 0/opal glass
Source	3000 K \pm 100 K
Materials	Suitable color step tablets

5. Wavelength Scale Standards

a. Description

Standards are required for the calibration of the wavelength scales of spectrophotometers. These standards are needed for checking and calibrating the wavelength scales of all spectrophotometers operating in the wavelength region of interest. Present SRM's using absorption filters should be supplemented by other types of standards. In particular, standards are required with sufficient calibration wavelengths to provide point-by-point error correction curves for instruments utilizing interference wedges; and standards are required to provide information on the centroid wavelength, spectral bandpass, and shape of the slit function for all broad-bandpass spectrophotometers. The problem is particularly acute for abridged spectrophotometers, for which no standards are readily available. The availability of SRM 2009, 2010, 2013, and 2014, based on the didymium absorption filter, answers only a small part of this need. New methodology suitable for the calibration of array detectors, as well as new types of standards, may be required.

b. Specifications

Spectral Range	200-760 nm; extend to 2500 nm later
Spectral Bandwidth	1-10 nm
Type	(a) Absorption Filter (b) Other
Uncertainty	± 0.5 nm or better
Applicability	Dispersive spectrophotometers, instruments using interference wedges as monochromators, and abridged spectrophotometers
Information Required	(a) Effective or centroid wavelength; (b) Spectral bandwidth and some indication of slit function shape

6. Colorimetric Reflectance and Transmittance Standards

a. Description

Physical standards of reflectance and transmittance, calibrated in colorimetric terms, are required as systems check standards. These standards would meet the needs for systems testing and diagnostic standards in this application. NBS SRM 2101-2105, now no longer available, provided some examples of the type of standard required, but had some defects. Better selected transmittance standards and a variety of reflectance standards are both required. They should be selected to provide diagnostic information on sources of error, as was done in past with SRM 2101-2105.

b. Specifications

Chromaticity	High purity, spaced around the CIE locus
Spectral Range	380-760 nm
Sources	CIE standard illuminants A, C, D ₆₅ , D ₅₀
Observers	CIE 1931 2° and 1964 10° standard observers
Uncertainty	0.0005 in x and y ± 0.2% in Y
Physical Size	50 × 50 mm
Geometry:	
Reflectance	45/0 and /0
Transmittance	d/0

7. Standards of Total Radiance Factor of Luminescent Materials

a. Description

Physical standards of total radiance factor are required. Total radiance factor is the sum of the reflected and fluoresced flux from a luminescent material. It is measured by irradiating the sample with heterochromatic light, with controlled spectral distribution, and detecting the radiance monochromatically.

b. Specifications

Spectral Excitation Range	300-400 nm
Spectral Emission Range	400-700 nm
Source Spectral Distribution	3000 K, D ₆₅
Geometry	d/0, 45/0
Physical Size	50 × 50 mm
Stability	Fully stable to conditions of use
Uncertainty	± 1% of value

8. Spectrofluorimeter Calibration Standards

a. Description

At least three standard materials are required for checking and calibrating spectrofluorimeters. These standards should luminesce in the blue (~ 450 nm), green (~ 550 nm), and red (~ 620 nm) spectral regions. Having such standards, properly calibrated, it will be possible to compute from the apparent luminescence spectra the relative efficiencies of luminescent materials and intercompare the results. Nearly all analytical laboratories are now equipped with some fluorimetric equipment to measure apparent luminescence as a part of their material characterization tests. Apparent spectra greatly depend on instrumental parameters, hence are not comparable because of the differences in instrumental parameters. The absolute fluorimeters capable of providing corrected (absolute) luminescence spectra are not readily available. There are now many clinical laboratories that are using fluorescence measurements as a tool in clinical diagnostics. These measurements are often made in turbid media (dispersions, solid plates and webs). It is imperative for these laboratories to have reliable SRM's through which their measurement can be kept under control. They also need procedural directives for making correct measurements and evaluations.

b. Specifications

1. Samples should preferably be solid non-diffusing and fit a standard cuvette holder. Also useful would be a set of liquid standards.
2. Should have moderate to high quantum efficiency.
3. Should be calibrated for 0/45 and 0/90 geometry.
4. Should exhibit low absorbance ($< 10\%$) in the region of luminescence emission (and also at the wavelength of excitation if intended for 0/90 geometry).
5. Samples should be stable to light and heat, easy to handle.
6. The following calibrations are required:

Excitation λ_{\max} ,

Emission λ_{\max} ,

Absorbance (α) at λ_{\max} of excitation,

Quantum Yield,

Refractive Index.

7. Suitable samples are glass such as Corning 3750 or B&L uranium glass and similar samples such as Rhodamine and other stable fluors imbedded in plastic chips.

9. Retroreflective Standards of Nighttime Brightness

a. Description

Physical standards of brightness (in units of candelas per lux per square meter for nighttime observation conditions) are required for retroreflective materials. These standards, in retroreflective materials, are for use in substitutional testing against government specifications of the nighttime brightness of retroreflective sheeting.

b. Specifications

Brightness Levels	Midpoints of ranges of commonly used Federal and State government specifications
Colors	Traffic colors as called for in above specifications, including white, yellow, red, blue, green, and orange
Geometry of Calibration	Observation and entrance angles commonly used in above specifications
Source	CIE Source A
Uncertainty	± 1% of value
Physical Size	300 × 300 mm
Material	Retroreflective

10. Retroreflective Standards of Daytime Color

a. Description

Physical colorimetric standards are required for the daytime observation conditions for retroreflective materials. These reference standards, in retroreflective materials, are for use in substitutional testing against government specifications of the daytime color of retroreflective materials. Since these materials must be tested using 45/0 geometry, and 45/0 spectrophotometers are not widely available, most testing is carried out using 45/0 colorimeters. It is particularly urgent that suitable calibrated standards, spectrally and geometrically similar to the material being tested, are available for local calibration of these instruments.

b. Specifications

Calibration Method	Spectrophotometric
Wavelength Range	380-760 nm
Illuminants	CIE standard illuminants C and D ₆₅
Observers	CIE 1931 2° and 1964 10° standard observers
Colors	Traffic colors as called for in commonly used Federal and State government specifications, including white, yellow, red, blue, green, brown, and orange
Geometry of Calibration	45/0
Uncertainty	± 0.001 in x,y; ± 0.2 in Y
Physical Size	100 × 100 mm
Material	Retroreflective

11. Retroreflective Standards of Nighttime Chromaticity

a. Description

Physical chromaticity standards are needed for the nighttime observation conditions for retroreflective materials. These reference standards, in retroreflective materials, are for use in substitutional testing of the nighttime chromaticity of retroreflective materials.

b. Specifications

Calibration Method	Spectroradiometric
Wavelength Range	380-760 nm
Illuminant	CIE standard illuminant A
Observer	CIE 1931 2° standard observer
Colors	Traffic colors as called for in commonly used Federal and State government specifications, including white, yellow, red, blue, green, and orange
Geometry of Calibration	Initially, 0.2° observation angle and 1° entrance angle; later, other sets of angles

b. Specifications (continued)

Uncertainty	± 0.002 in x,y
Physical Size	300 × 300 mm
Material	Retroreflective

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