

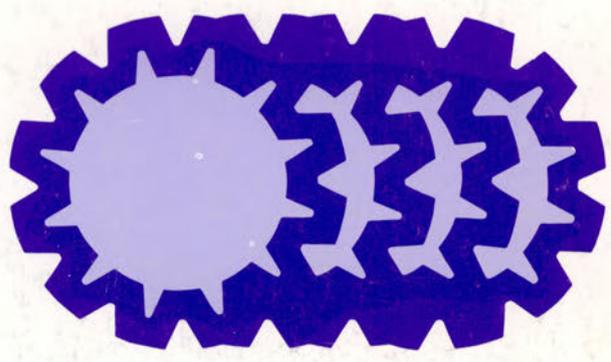
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# **NIOSH**

**TECHNICAL INFORMATION**

## **Ultraviolet Transfer Standard Detectors and Evaluation and Calibration of NIOSH UV Hazard Monitor**





ULTRAVIOLET TRANSFER STANDARD DETECTORS  
AND  
EVALUATION AND CALIBRATION OF NIOSH UV HAZARD MONITOR

Robert P. Madden  
Chief, Far Ultraviolet Physics Section  
Optical Physics Division  
Institute for Basic Standards  
National Bureau of Standards  
Washington, D.C. 20234

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U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE  
Public Health Service  
Center for Disease Control  
National Institute for Occupational Safety and Health  
Division of Laboratories and Criteria Development  
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## PREFACE

In 1972 the National Institute for Occupational Safety and Health (NIOSH) published "Criteria for a Recommended Standard. . . Occupational Exposure to Ultraviolet Radiation." This document indicated a need for a portable ultraviolet measurement instrument with weighted response based on the recommended standard for actinic (200 to 320 nm) ultraviolet radiation. Development of an ultraviolet radiation monitor was initiated by the Physical Agents Branch of the Division of Laboratories and Criteria Development, NIOSH, through a contract awarded to Columbia Broadcasting System Laboratories.

During development of the prototype instrument, a search was undertaken for a method to calibrate and evaluate the spectral sensitivity of the instrument. For this purpose, the National Bureau of Standards (NBS) and commercial optical laboratories were contacted. No ultraviolet source of spectral irradiance or detector could be found that was accurately calibrated over the 200 to 320 nm wavelength range.

Consequently, NIOSH entered into an Interagency Agreement with the Far Ultraviolet Physics Section, Optical Physics Division, NBS, to develop and calibrate transfer standard detectors for ultraviolet radiation in the 200 to 320 nm wavelength range. The technical aspects of this agreement are shown in Appendix I.

This document contains the report received from NBS indicating the methods used and results obtained. Additional efforts are underway at NBS and NIOSH to determine the stability of these transfer standards.

Edwin L. Sensintaffar  
Acting Chief, Radiation Section  
Physical Agents Branch  
National Institute for Occupational  
Safety and Health

This report was written at the National Bureau of Standards as a final calibration project report for internal use at NIOSH. However, as it contains considerable information which may be of general interest, it is being made available for limited distribution.

Robert P. Madden  
Chief  
Far UV Physics Section  
National Bureau of Standards

## ABSTRACT

A recommended standard for occupational exposure to ultraviolet radiation has created a need for accurate ultraviolet radiation measurements in industry. As instruments are developed to perform these measurements, it becomes necessary to provide a means for determining the accuracy of the measurements. An important aspect in assuring accuracy is the calibration of instruments with a reliable standard. The National Bureau of Standards has developed for NIOSH a primary standard for laboratory calibration of transfer standards. This report describes the development of these calibration standards and the calibration procedure for an ultraviolet radiation monitor.



## I PURPOSE OF PROJECT

This project was undertaken in order to develop the capability, at the National Bureau of Standards (NBS), to calibrate transfer standard irradiance detectors to be used for the calibration of the National Institute for Occupational Safety and Health (NIOSH) ultraviolet hazard monitors (UVHM) in the 200 - 320 nm region, and to supply two detectors to NIOSH. In addition, the prototype UVHM, a CBS Laboratories development, was to be calibrated by NBS both absolutely and relatively in order to establish, prior to the initiation of a NIOSH calibration facility, the degree of conformity to specifications.

## II METHODS

### A. Calibration of Transfer Standard Detectors

The reference detector used in these measurements was an EMR Photoelectric 541N-05M-00 vacuum photodiode with a tetraphenyl-butadiene (TPB)-coated silica disc 2.5cm in diameter located 1.7cm in front of the face of the photodiode. TPB has been investigated as a fluorescent wavelength converter<sup>1</sup> and was found to be very suitable for this application. It has the advantage of having relatively uniform quantum efficiency in the 200 - 320 nm region of interest and of being evaporable, implying that spatial uniformity might be expected in such coatings. This was of particular interest, since the EMR detector employed did not have a high degree of uniformity over the photocathode. One would expect a

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<sup>1</sup>W. M. Burton and B. A. Powell, Appl. Opt. 12, 87 (1973).

TPB coating placed somewhat in front of the photocathode to have a beneficial effect similar to a diffusing screen in effectively smoothing non-uniformities. (The direct calibration of an EMR photodiode using a calibrated thermopile was possible, but was rejected in favor of the TPB/photodiode approach. The non-uniformities of the photocathodes would have tended to degrade the accuracy of thermopile-based measurements, which utilize a monochromator other than that used for interdiode comparisons.) In addition, the stability of TPB was investigated over the period of time necessary to conduct these measurements.

The absolute quantum efficiency of the TPB-photodiode package was determined in two steps. Secondary standard photodiodes with absolute calibrations from 200-254 nm were on hand and were used in this region to transfer an absolute calibration. At the longer wavelengths, since no calibrated detectors were available, it was necessary to compare the response at each wavelength with the response of a special vacuum thermopile, utilizing a vacuum monochromator with a diffraction grating obtained for this purpose and blazed for these longer wavelengths. These latter calibrations were overlapped in wavelength with the measurements using standard photodiodes for the absolute normalization.

The TPB-photodiode package which had received an absolute calibration (described above) was available following its calibration to transfer

this calibration to a photodiode which would then become a working standard.

For this phase of the project, a vacuum monochromator with a plasma source and an attachment for intercomparing detectors was employed. This apparatus allows the alternate placement of either of two detectors (sample and standard) in a fixed exit beam, which is stopped to give an illuminated area approximately 6 x 6 mm on the photocathodes of the detectors. The ratio of the photocurrents from sample and standard at each wavelength leads to the direct calculation of sample quantum efficiency, given the quantum efficiency of the standard.

Spatial uniformity of the photocathode of each transfer standard was measured by stopping the monochromator exit beam to about 0.5 mm and scanning the detector behind this stop. A complete sensitivity map of the photocathode may be obtained with this apparatus. Representative vertical and horizontal scan results for one diode are shown in Figures 1A and 1B.

The stability of the quantum efficiency of the photodiodes was investigated by repeated measurements against the standard over a period of several weeks. With several sample detectors in the measurement rotation, it was possible to detect any changes in the quantum efficiency of the standard, as well as of a given sample.

## B. Calibration of the UVHM

The UVHM calibration was separated into two phases: relative and absolute. Once the relative spectral response of the UVHM had been determined in the region of interest, a single-wavelength absolute measurement would serve to normalize the entire response in absolute units.

### 1. Relative Response of the UVHM

Relative response measurements were carried out using the general experimental format shown in Fig. 2. A deuterium light source was arranged to fill the grating of an f/10 Fastie-Ebert monochromator via a single silica lens. The exit beam was similarly directed through a silica lens arranged so as to slightly underfill the UVHM detector. Space was left to allow the insertion of a reference detector without disturbing the UVHM or the lens. Subsequently, "A-B" type comparisons could be made at any wavelength, with the analog voltages from either detector being converted to pulses (with the pulse rate directly proportional to the voltage) and the number of pulses in a timed period counted electronically.

Even though the TPB-photodiode had been calibrated absolutely, it was, in fact, used only as a relative spectral detector in the UVHM calibration. The reason for this is that while this detector was underfilled and had good spatial uniformity, the detector in the UVHM did not have

good uniformity and it was essentially impossible to determine the exact area illuminated due to inaccessibility. Although a few techniques were available to interpret the data in absolute units, these would have tended to introduce additional errors, so a more direct approach was employed.

## 2. Absolute Calibration of the UVHM

The transformation of a relative spectral calibration to an absolute one requires but a single wavelength for normalization. To achieve this in a manner equivalent to the ordinary conditions of use of the UVHM, i.e., with uniform flux illuminating the UVHM detector, a calibrated ultra-stable 253.7 nm mercury source was employed. This source was calibrated by members of the NBS Heat Division using three different radiance lamps. With the source 2.00m distant from the UVHM detector (which was taken from the manufacturer's literature to be 10 cm from the outer portion of the UVHM mounting flange) the analog voltage was extracted as per Fig. 2 and measured. The statistical average of the net counter readings was equated to an absolute voltage reading by measuring, with a digital voltmeter, the voltage necessary to give an equal net count rate.

## III RESULTS

### A. Transfer Standards

The relative quantum efficiency of the TPB samples used was found to be constant  $\pm 3\%$  from 220 - 290 nm, and gradually decreased at longer

wavelengths to -20% at 320 nm. These empirical corrections to flatness were applied to data taken using TPB.

Discussion of the results of studies on the transfer standard detectors will be restricted to generalized comments, since the specific information pertaining to a transferred photodiode is included in the Report of Calibration accompanying each photodiode. However, a plot of the quantum efficiency of a typical photodiode is given in Fig. 3.

The uniformity across the photocathodes was better than expected, considering that the manufacturer lacked the confidence to accept any specification of this parameter. Generally, the variation of the quantum efficiency across the 6 x 6 mm area used for calibrations was on the order of less than 10% peak-to-peak. It is probable that this would degrade the accuracy of a transfer by only a fraction of this percentage.

The stability of the measured quantum efficiency of the transfer standards has been excellent. With non-desiccated storage, no apparent changes in excess of 2% of the measured quantum efficiency have been observed. Typically, over a one month period no changes as great as 1% of the value have been detected. It appears likely that, with careful handling and use, three months could be set as the minimum recalibration interval. However, it should be noted that photoelectric detectors have been observed to change in efficiency suddenly without obvious cause,

so periodic recalibration is mandatory if confidence in the detectors is to be maintained.

#### B. UVHM Calibration

The relative spectral response of the UVHM is listed in Table I, and shown graphically in Fig. 4. The combination of insufficient intensity and very weak UVHM response from 200 - 220 nm made it impossible to extract meaningful information in this region.

The curve of Fig. 4 is an almost exact duplicate of the relative response curve originally measured by CBS laboratories and shown in the UVHM manual. This indicates the probability that the interference filters and MOS detector employed in the UVHM have not changed significantly in relative response since the original CBS calibration.

The calibrated mercury source used for the absolute measurement has, according to the manufacturer, about 90% of its intensity in the 253.7 nm line. A previous investigation<sup>2</sup> of this type of low pressure Hg source has shown that the contribution to the total radiated intensity from 200 - 320 nm (the spectral region over which the UVHM has significant sensitivity) other than the 253.7 nm line should be negligible. To confirm this, the source and UVHM were set up for an intensity measurement, but two 253.7 nm interference filters were employed -- the first to monochromatize the source and thus enable the measurement of the

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<sup>2</sup>H. M. Crosswhite and G. H. Dieke, "American Institute of Physics Handbook," 2nd Edition p. 7-125, McGraw-Hill Book Co., Inc., New York

transmittance of the second filter by insertion. The UVHM was used as the detector (at 2.00 m) for this measurement. Following the transmittance determination, the second (T-measured) filter was left in place and the first was removed. The analog UVHM voltage thus obtained was corrected for the filter loss and compared to the analog voltage obtained with no filter. Several trials of this nature indicated that there was no statistically significant difference in the results whether the interference filter was used or not. This confirmed that the spectral emission of the low pressure mercury discharge was almost entirely in the 253.7 nm region as seen by the UVHM detector, and the balance of the absolute calibrations were carried out without the use of a filter.

The result of this calibration was:

<u>irradiance (w/cm<sup>2</sup>)</u>	<u>V(volts, net)</u>
3.51 ± 0.18 x 10 <sup>-7</sup>	-1.259 ± 0.035

which corresponded, roughly, to a net meter reading of 1.60 on the 3 x 10<sup>-7</sup>w/cm<sup>2</sup> scale. All measurements were taken with the ambient temperature 21-22°C.

The relative calibration of the UVHM indicated that the 253.7 nm response is 54.8% of the maximum, therefore the meter would be expected to indicate 54.8% of the incident 3.51 x 10<sup>-7</sup>w/cm<sup>2</sup>, or 1.92 x 10<sup>-7</sup>w/cm<sup>2</sup>. (This assumes that the designer intended the meter to read the true value of the incident flux density where the UVHM relative response

is at its maximum.) Since, in fact, it indicated  $1.60 \times 10^{-7} \text{ w/cm}^2$ , the indicated value must be increased by a factor of 1.20 to obtain the correct value. Neither the linearity of the UVHM nor the accuracy on other scales were investigated.

At the request of E. Sensintaffar of NIOSH an attempt was made to bring the meter readings into agreement with the incident flux density via a gain adjustment in the UVHM electronics. It was found that the available adjustment was already at the maximum gain setting, so no change was made. The electrical zero of the instrument was observed to drift as much as 3% of full scale, so it is recommended that the exact "zero" meter reading be checked before any serious measurements are attempted, and rechecked before each subsequent reading.

#### Summary

A study has been conducted of the uniformity and stability of four transfer standard photodiodes. Two of these standards have been calibrated from 200 - 320 nm and supplied to NIOSH for the establishment of their calibration facility.

The CBS-NIOSH UVHM has been calibrated both relatively and absolutely from 220 - 320 nm and has been returned to NIOSH. The relative spectral efficiency of the UVHM on the most sensitive scale is essentially the same as determined by CBS Laboratories, but the absolute readings should be multiplied by 1.20 to give the true values.

TABLE I

## RELATIVE SPECTRAL RESPONSE OF UVHM

<u>Wavelength (nm)</u>	<u>Measured Response (% of Maximum)</u>	<u><math>\frac{\sigma}{\sqrt{n}}</math> (%)</u>
220	2.61	22
225	2.39	19
230	4.77	15
235	6.56	10
240	11.1	7.8
245	23.8	4.4
250	37.9	1.8
255	60.8	0.7
260	84.0	0.4
265	99.3	0.2
270	99.9	0.3
275	89.2	0.6
280	79.0	0.8
285	64.6	1.1
290	49.6	1.4
295	31.2	1.6
300	17.0	1.8
305	8.30	2.2
310	3.40	2.4
315	1.13	9.0
320	0.66	29

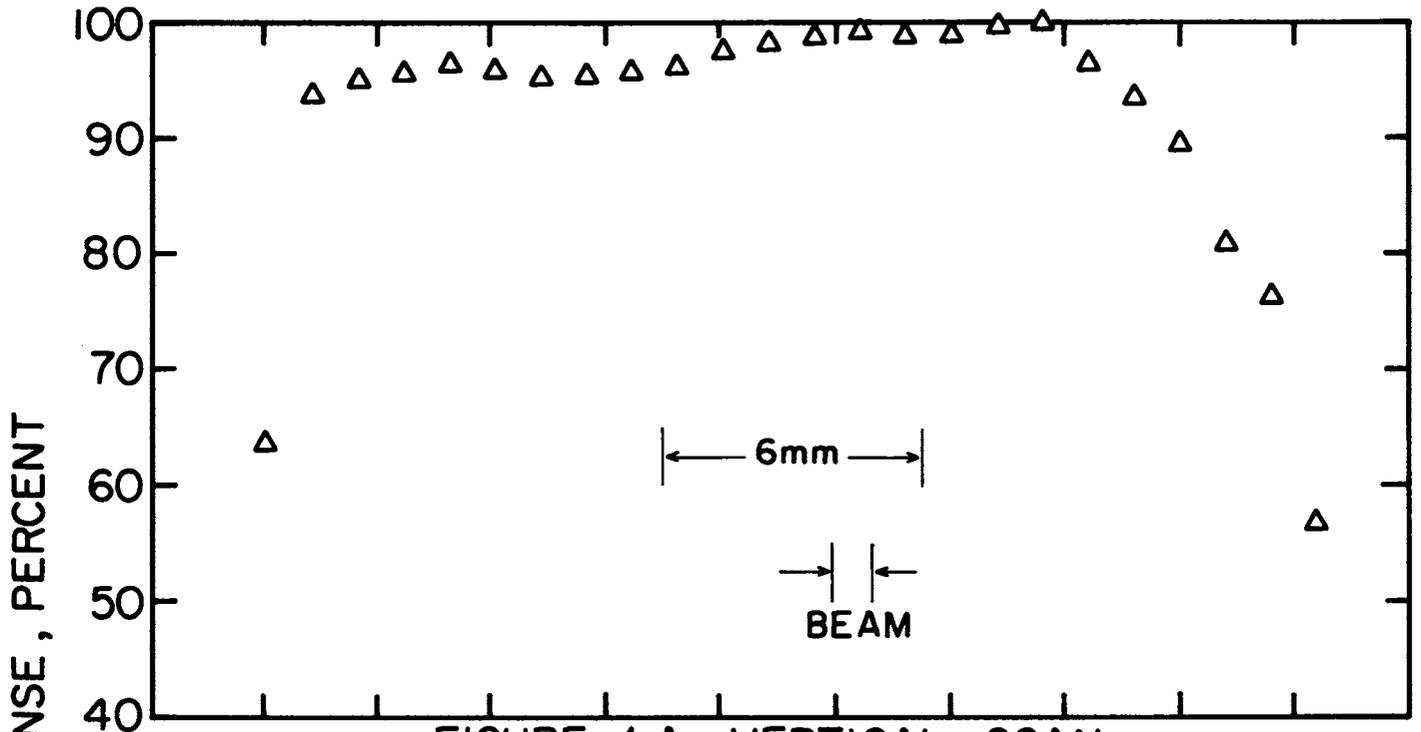


FIGURE 1A . VERTICAL SCAN

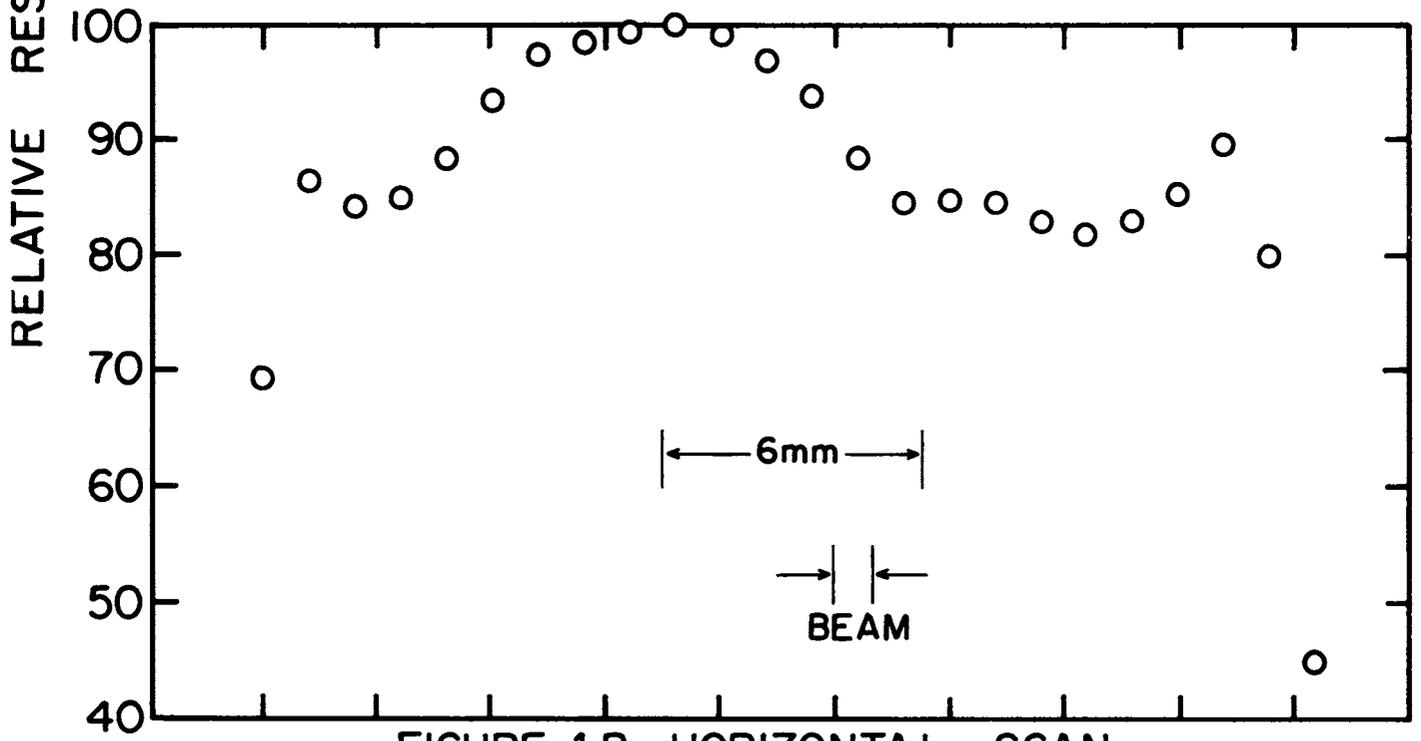
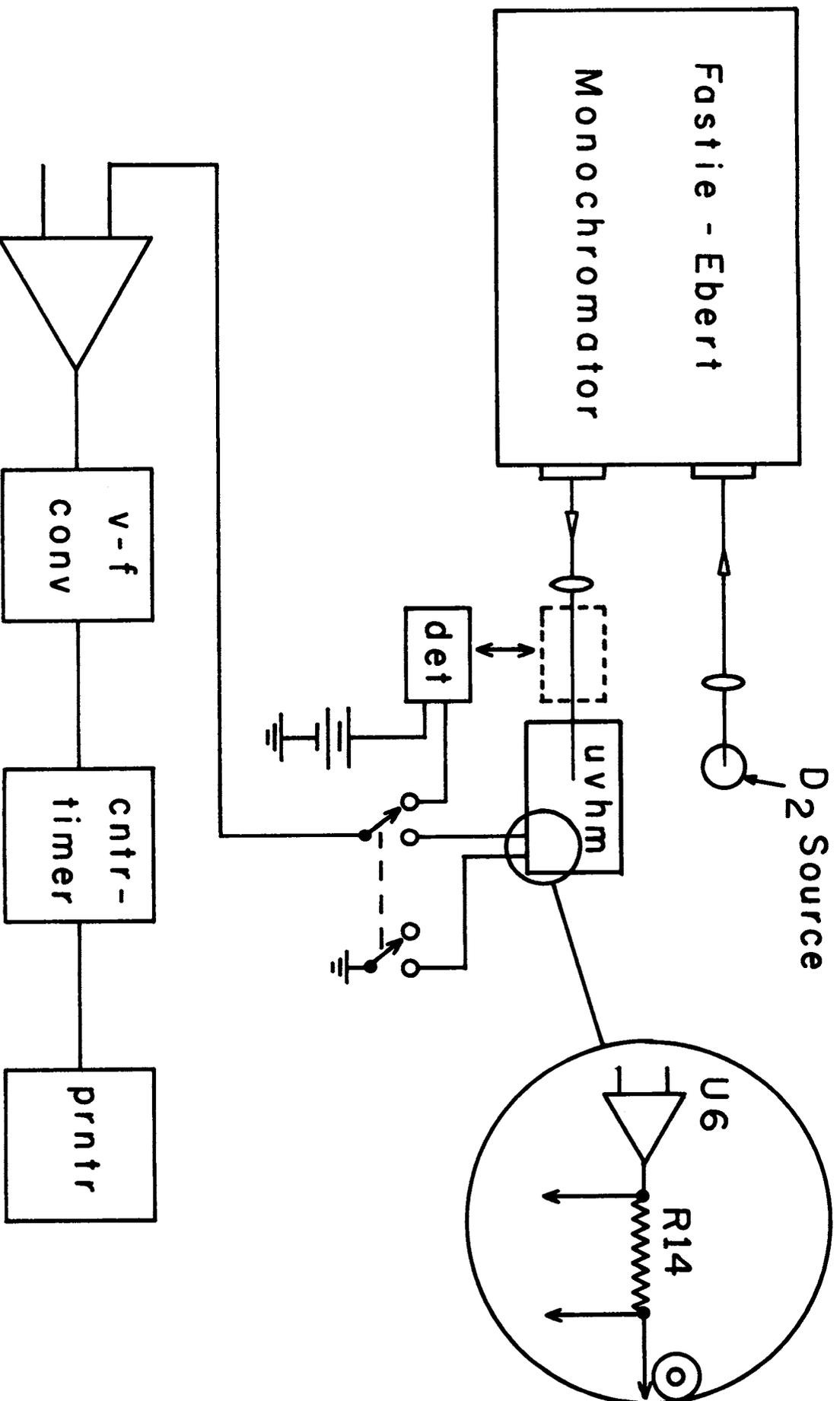
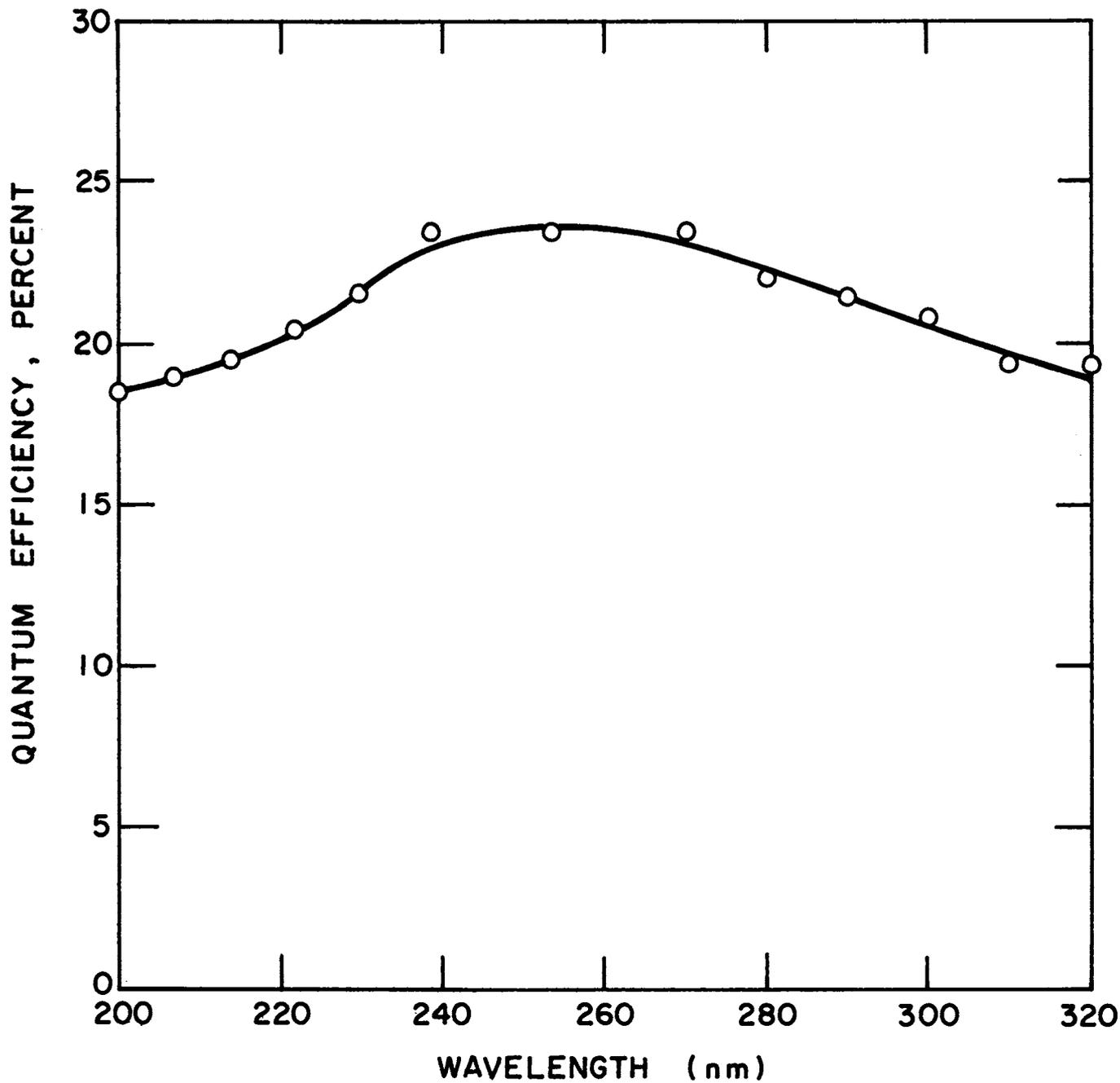


FIGURE 1B . HORIZONTAL SCAN

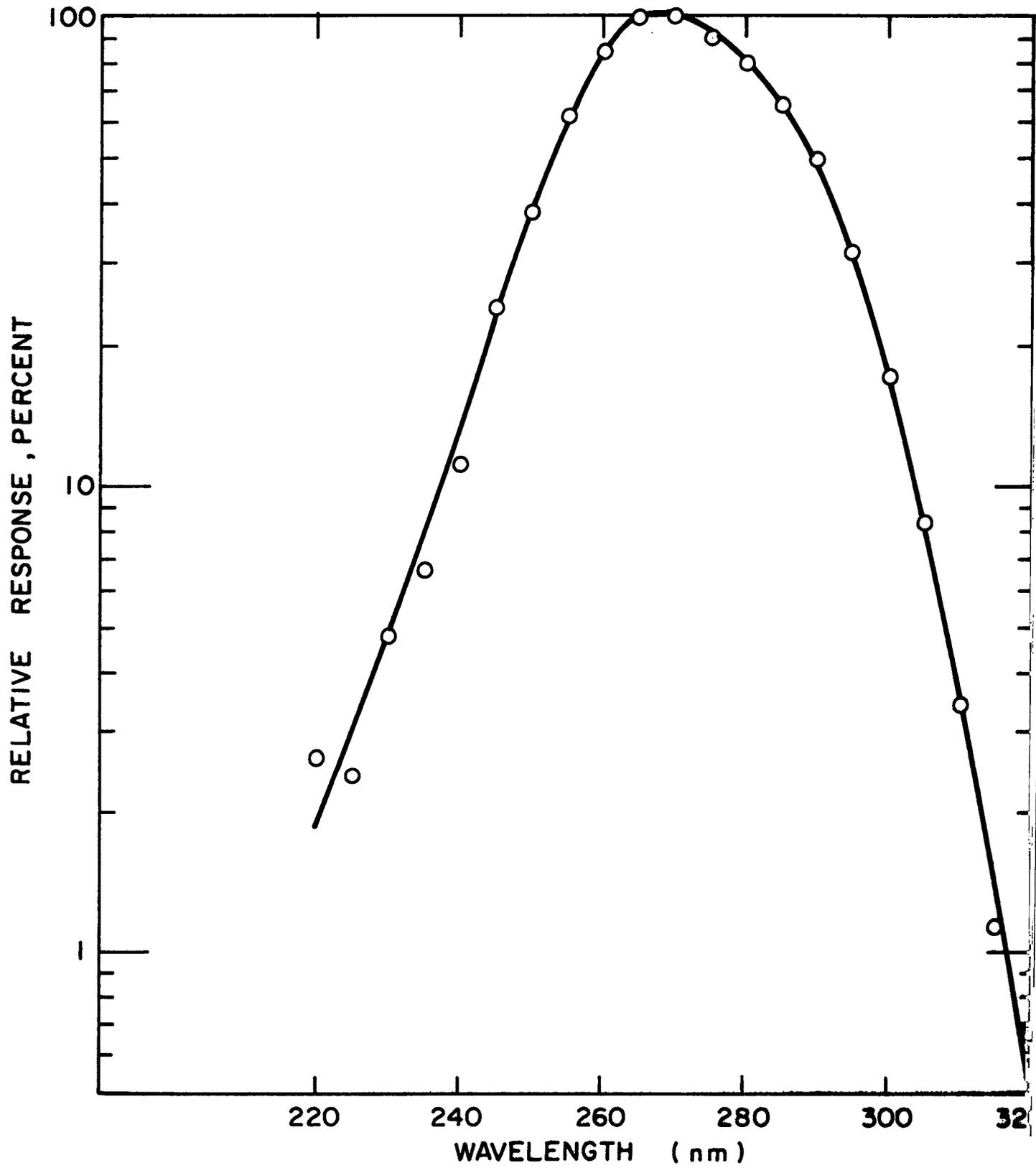
**FIGURE 1. Spatial uniformity measured across vertical (Fig. 1A) and horizontal (Fig. 1B) axis of detector**



**FIGURE 2. Experimental arrangement for ultraviolet hazard monitor relative response testing**



**FIGURE 3. Quantum efficiency of a typical photodiode for actinic ultraviolet radiation wavelengths**



**FIGURE 4. Response of CBS-NIOSH UV hazard monitor**

APPENDIX I

MEMORANDUM OF AGREEMENT

BETWEEN

THE NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH

AND

THE NATIONAL BUREAU OF STANDARDS



MEMORANDUM OF AGREEMENT

BETWEEN

PHYSICAL AGENTS BRANCH  
DIVISION OF LABORATORIES AND CRITERIA DEVELOPMENT  
NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH  
HEALTH SERVICES AND MENTAL HEALTH ADMINISTRATION  
DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

AND

NATIONAL BUREAU OF STANDARDS  
WASHINGTON, D. C.

The National Bureau of Standards (NBS) shall develop ultraviolet calibration capabilities and related transfer standard diodes for calibration of National Institute for Occupational Safety and Health (NIOSH) UV Hazard Monitors.

OBJECTIVES OF THE AGREEMENT

1. To evaluate quantitatively the performance of a prototype UV Hazard Monitor that has been built to NIOSH specifications by a commercial contractor. This evaluation will involve both absolute and relative spectral calibrations of this prototype instrument in the wavelength region 200 - 320 nm to assess conformity to contract specifications. Such an evaluation is of paramount importance, since it is envisioned that the prototype is the forerunner of a quantity production of UV Hazard Monitors. Because Ultraviolet Hazard Monitors have potential use in compliance measurements, an accurate ultraviolet radiation calibration capability is required. Consequently, the ability of the Monitor to perform in the specified fashion must be confirmed.

2. To provide standards for a proposed NIOSH facility at which post-prototype UV Hazard Monitors could be accurately calibrated by NIOSH personnel. This requires establishing the capability at NBS of providing absolute and relative spectral calibrations of transfer standard photodiodes in the wavelength region 200 - 320 nm. These calibrated photodiodes will be provided to NIOSH.

3. NBS is to maintain this calibration capability for the indefinite future to allow subsequent absolute calibrations on a regular basis. Transfer standard diodes will be provided to NIOSH, on a rotating basis, to serve as in-house standards which will form the continuing foundation of the NIOSH calibration facility. At this time, a total of at least three such calibrated photodiodes are needed. One of these will be permanently retained at NBS, where one will always be available for calibration of NIOSH photodiodes with the second undergoing primary calibration. The third diode will thus always be available for use at NIOSH.

#### APPROACH

A phosphor will be used to extend the single line calibration provided by a Hg lamp over the desired wavelength range. A photodiode (e.g., EMR) will be used to monitor the fluorescence of an optically coupled phosphor screen which will be illuminated by a calibrated Hg line. This photodiode, with phosphor screen, will become the NBS in-house transfer standard by means of which outgoing (NIOSH) photodiodes will be calibrated.

Because the phosphor most appropriate for the application, TPB, has not been studied at NBS, a preliminary study is necessary. Periodic absolute calibrations of the detectors will be required. Probable error in the calibration of a photodiode by this approach shall not exceed 15 - 20 percent.

NIOSH - Project Officer  
Mr. Alfred Mendenhall  
Physical Agents Branch, NIOSH  
1014 Broadway  
Cincinnati, Ohio 45202

NBS - Project Officer  
Mr. Robert P. Madden, Chief  
232.03  
National Bureau of Standards  
Washington, D.C.

7 June 73  
Date

s/J. H. Tyson (acting)  
Chief, 232.00

7 June 73  
Date

s/Robert P. Madden  
Chief, 232.03

Jun 28, 1973  
Date

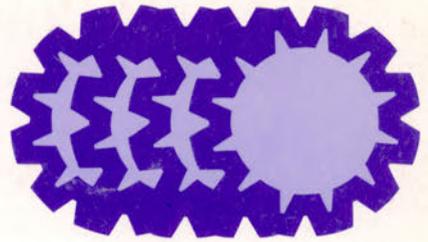
s/Marcus M. Key  
Director, National Institute for  
Occupational Safety and Health

Accepted for the National  
Bureau of Standards:

s/Thomas C. Austin (Actg.)  
J. E. Skillington, Jr.  
Chief, Budget Division



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U.S. DEPARTMENT OF  
HEALTH, EDUCATION, AND WELFARE

Public Health Service

Center for Disease Control

National Institute for Occupational  
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