NBS SPECIAL PUBLICATION 529

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

Interagency Comparison of Ultraviolet Photometric Standards for Measuring Ozone Concentrations

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Interagency Comparison of Ultraviolet Photometric Standards for Measuring Ozone Concentrations

J. Wendt, J. Kowalski, A. M. Bass, C. Ellis, and M. Patapoff

ABSTRACT

In August 1977, an interlaboratory comparison study was initiated after the California Air Resources Board and the Environmental Protection Agency, Research Triangle Park independently designed and constructed ozone absolute ultraviolet photometers. These units augmented the photometers already designed and in use by the National Bureau of Standards and the Jet Propulsion Laboratory. Previous ozone comparisons involving indometry, ultraviolet photometry and gas phase titration have reported results differing by as much as $\pm$ 30%. The protocol of this study, performed at the California Air Resources Board, Haagen-Smit Laboratory, El Monte, was a multipoint comparison using the NBS photometer as the standard. The results of the data analysis indicate excellent agreement with a total variation of 2.8%. Compared to the NBS photometer the EPA-RTP photometer read 1.3% low, the JPL photometer read 1.5% high and the ARB photometer read 0.4% low. The correlation coefficients of the same data sets were 1.0000, 1.0000 and 0.9999 respectively.

Key words: Air pollution; atmospheric monitoring; calibration; ozone; ultraviolet; photometry.

1. INTRODUCTION

For more than 100 years, ozone concentrations have been determined by utilizing the chemical reaction between ozone and potassium iodide to liberate iodine which is then measured by chemical or physical means. The neutral buffered, potassium iodide (NBKI) method has gained wide acceptance and is the calibration technique of the Federal Reference Method for measuring photochemical oxidant concentrations.[6] Despite variations in the chemical composition of the reagents, all of the potassium iodide methods in common use assume a one to one stoichiometry between the iodine released and the ozone reacted.[2]

Studies conducted by the California Air Resources Board and the U. S. Environmental Protection Agency have shown significant departures from the assumed stoichiometry. The stoichiometry departures were found to depend on the buffering agent and potassium iodide concentrations. These departures also depend on the nature of the buffering agent and the absolute humidity of the sample air stream.[2,3]

As a result of the various studies, the California Air Resources Board in May of 1975 adopted ultraviolet photometry as its reference method for measuring ozone concentrations.[7] Since California's adoption, interest in ultraviolet photometry as a standard for measuring ozone concentrations has steadily escalated. Ultraviolet photometry is now generally considered a prime candidate for replacing the existing

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[1] Figures in brackets indicate literature references at the end of this paper.

NBKI calibration procedure and ASTM Methods for measuring photochemical ozone concentrations.

By June of 1977, four agencies, the California Air Resources Board, National Bureau of Standards, U. S. Environmental Protection Agency, and the Jet Propulsion Laboratory independently designed and fabricated long path ultraviolet ozone photometers. The four photometers are all of different design with two located on each coast of the United States.

In August of 1977 the photometers or suitable transfer standards were assembled at the California Air Resources Board's Laboratory in El Monte, California. The four photometric standards were allowed to sample a common ozone supply and their respective readings compared. The results of the comparison are reported.

2. PHOTOMETER DESIGNS

2.1 National Bureau of Standards (NBS)

The NBS photometer is designed to measure ozone concentrations ranging from 0.05 to 10.0 ppm with an accuracy of approximately 0.005 ppm by volume. The design of the photometer, shown in figure 1, incorporates a double-beam arrangement to provide greater precision in the measurement by elimination of variability in the UV source. The cells of the photometer are approximately 3 meters long and are made of 1-1/2" diameter pyrex pipe; teflon gaskets are used to make vacuum-tight seals for the fused silica windows. The light from a low pressure mercury discharge lamp is passed through a narrow-band interference filter in order to isolate the 253.7 nm emission line. The light is collimated by a fused silica lens and passed through a partially-transmitting neutral density filter which serves as a beam splitter. The two beams then pass through

the two absorption cells. Adjustable aperture stops limit the diameter of the beams to ensure that there are no reflections from inner walls of the cells. The light beams emerge from the cells and are recombined on the face of a photomultiplier tube by another partially reflecting filter.

Light passing through each sample cell is alternately allowed to fall on the photomultiplier by means of a light chopper. A chopper blade with a single hole is driven by a hysteresis synchronous motor. Light emitting diode-phototransistor pairs are used to sense the positioning of the hole in the chopper blade; the signal from the phototransistor triggers a discriminator to start the timing and counting cycle for each sample tube.

A logic system, triggered by the discriminators, controls pulse counters associated with each sample tube. In order to ensure precise counting times as the photomultiplier is exposed to each tube, an electronic gate is used to ensure that the photomultiplier is fully (and not merely partly) exposed to the light beam passing through the sample.

The pulse output from the photomultiplier is amplified, and pulse counting is performed by conventional 100 MHz pulse counters and a rough guide of overall pulse rate is provided by a rate meter. Counting time is determined by a preset counter. At the end of a counting interval, the results are printed out.

House air, dried and filtered, flows through one cell ("reference cell"), then into an ozone generator from which ozonized air flows through the second cell ("sample cell"). The measurement is made by comparing the ratio of the signals transmitted by the two cells in the

presence and in the absence of ozone. This provides the transmittance ($Tr = \frac{I}{I_o}$), and the ozone concentration is determined by application of the Beer-Lambert Law. Since the mercury lamp is viewed, nearly simultaneously, through both cells, fluctuations in lamp intensity do not affect the measurement. Any impurity present in the air stream will be observed in both cells and will not interfere with the O_3 determination,[8] unless it too is photolysed by the O_3 generator and it or the products absorb at 253.7 nm.

2.2 Jet Propulsion Laboratory (JPL)

There are three components to the JPL photometer, the light source, the absorption cell, and the light detector as shown in figure 2. A low-pressure mercury lamp is used as a source of the 253.7 nm light. A 1/4-m monochromator was used to spectrally isolate the 253.7 nm line. (This component is not strictly necessary and can be replaced by an interference filter to isolate the 253.7 nm line). Collimation of the light beam was aided by use of a 25-cm focal length quartz lens. Stability of the light source was enhanced by operation from a constant voltage transformer and by enclosing the lamp in an aluminum block for increased thermal inertia. The absorption cell is constructed of 2-in o.d. Pyrex tubing, and has a path length of 5.00 m. Quartz windows are attached at each end by means of epoxy cement, with care being taken to minimize internal exposure of cement. The 253.7 nm light emerging from the cell is monitored with a solar-blind, side-on photomultiplier tube. The photomultiplier is operated at 340 V. The photomultiplier output voltage is measured across a 10-MΩ resistor and is read with a four-

digit voltmeter. A 0.4-μfd condensor is placed in parallel with the resistor to obtain a suitable time constant.

Ozone is generated in a stream of cylinder air by means of a photochemical generator of conventional design, employing a low-pressure mercury lamp with a sliding shield to vary the O_3 concentration. The gas flow through the cell, usually 5-7 l/min, enters near one end of the cell and exits at the opposite end. It has been established experimentally that there is no appreciable gradient of ozone concentration in the cell.

With carrier gas flowing, photomultiplier output voltages are read periodically during the calibration procedure and are plotted as a function of time. At intervals, transmittance readings are taken at ozone concentrations in the range 0.00 to 0.85 ppm of O_3. Transmittance ratios (I_o/I_t) are then calculated, and ozone concentrations determined by application of the Beer-Lambert Law.[9]

2.3 Environmental Protection Agency (EPA-RTP)

The EPA-RTP photometer, as shown in figure 3, is a two meter instrument designed to demonstrate how off-the-shelf electronics could be used to produce a high precision ($\pm$ 1 ppb) photometer. The absorption volume consists of two one-meter cells in a folded path configuration which reduces the overall instrument length to 1.27 meters and greatly improves the system's portability. The cells are made of 12 mm inside diameter pyrex tubing and are fitted with quartz windows via teflon gaskets. The optical path is folded by means of small plane mirrors which are located outside the absorption cell.

The photometer is essentially monochromatic due to use of a Vycor nvelope on the low pressure mercury source and solar blind vacuum hoto-diode detectors. A reference detector is used to monitor the ight source in a quasi-double beam configuration. A sample detector is sed to monitor the absorption of the light in the cell.

An ozone concentration is determined by alternately filling the ell with ozone-free reference air and the ozone calibration atmosphere. he transmittance of the ozone sample is equal to the ratio of the ormalized sample channel frequency obtained with ozone in the absorption ell to that obtained with reference air in the cell. The ozone concentration s calculated from the transmittance via the Beer-Lambert absorption aw.

2.4 California Air Resources Board (ARB)

The ARB photometer is designed to measure ozone concentrations anging from 0.02 to 15.0 ppm with an accuracy of 0.01 ppm over the ntire range. To achieve the design accuracy, the photometer incorporates double beam principle which eliminates errors resulting from light ource or air supply variations. The cells of the photometer are onstructed from 1/2 inch diameter Pyrex pipe with a path length of 260 entimeters. Quartz windows, sealed with epoxy cement, are affixed to ach end of the cells as shown in figure 4.

A low pressure mercury discharge lamp of sufficient length to span oth cells of the photometer is used as a source of 253.7 nm light. The ycor sheath of the lamp and the detector spectral response characteristics imit the photometer sensitivity to the 253.7 nm emission line. Lamp utput stability is achieved by operating the mercury discharge lamp

from a crystal controlled, chopped d.c. power supply, and housing the lamp in a thermally controlled aluminum block.

Light passing through the sample and reference cells is converted to analog current signals by separate detectors mounted in a thermally controlled aluminum block. The analog current signals are converted to frequency signals utilizing digital electrometers. The electrometer outputs are connected to a dual channel frequency counter capable of counting the reference channel to a preset value. Each time the preset reference count is reached, the ratio of the sample detector count to reference detector count is displayed.

An ozone concentration measurement is accomplished by first admitting ozone free air to each cell of the photometer. The ratio I_0/REFERENCE INTENSITY (ratio of zero air to reference detector count) is recorded. Ozonized air from the ozone generator is then admitted to the sample cell with ozone free air flowing through the reference cell. The ratio I/REFERENCE INTENSITY (ratio of sample to reference detector count) is recorded and the transmittance ratio I/I_0 calculated. The ozone concentration is calculated from the Beer-Lambert Law.

3. EXPERIMENTAL

The experimental phase of the photometer comparison was conducted at the California Air Resources Board's El Monte Laboratory utilizing the experimental setup of figure 5. Clean, dry, ozone free air was supplied to the experimental setup by an Aadco clean air supply designed to ARB specifications. The clean air was metered to a Monitor Labs Inc. Model 8500 calibration system and to a multiported glass manifold by mass flow controllers. The ozone output of the calibration system was

ducted to a multiported glass manifold. All interconnections were made using Teflon tubing.

A direct comparison of the NBS and JPL photometers with those of the ARB and EPA-RTP could not be accomplished due to the large size, complexity and construction characteristics of the photometers. NBS and JPL participation in the comparison studies was accomplished through the use of Dasibi Corporation Model 1003 AH ozone monitors[10] as transfer standards. Each transfer standard was calibrated against the respective agencies' photometer before and after the El Monte comparison. Both the ARB and EPA-RTP photometer, described herein, were used directly in the comparison studies. It should be noted that the EPA-RTP photometer was disassembled, shipped, and reassembled at the ARB laboratories. With minimal checkout prior to the comparison, the precision and stability the readings were less than in previous laboratory measurements.

A total of eight experimental runs, each consisting of pre and post run instrument zero, and an ozone concentration ranging from 0.05 to 0.70 ppm, were completed. Following each experimental run, the respective agencies calculated the ozone concentration recorded by their photometer by making the necessary temperature and pressure adjustments using the following equations:

ARB, EPA-RTP*

$$C\ (ppm) = \frac{(10^6)\ (760)\ (T)}{(K)\ (L)\ (P)\ (273)} \ln \frac{I_o}{I}$$

where:

T = absolute temperature, °K

P = absolute pressure in photometer, torr

L = photometer path length, cm

k = extinction coefficient, base e, cm^{-1} atm^{-1}

I_o = intensity of light with zero ozone

I = intensity of light with ozone at concentration

* The EPA-RTP calculations also include a 1% correction for ozone losses which occur in the photometer.[11]

JPL

$$C\ (ppm) = \frac{(127.098)\ (\beta)\ (T)}{(P)}\ \ln\ \frac{100 - \frac{B}{0.539}}{100 - \frac{R}{0.539}} \quad [12]$$

where:

β = correction constant for reference Dasibi S/N 1386; (1.0685)

B = Dasibi background reading

R = Dasibi ozone reading

P = absolute pressure, torr

T = absolute temperature, °K

NBS

$$C\ (ppm) = \frac{(127.098)\ (\beta)\ (T)}{(P)}\ \ln\ \frac{54.6}{54.6 - \Delta I} \quad [13]$$

where:

ΔI = change in light intensity between zero and ozonized air; Dasibi reading)

T = absolute temperature, °K

P = absolute pressure, torr

β = correction constant for reference Dasibi S/N 1707; (1.0134

54.6 = span factor of Dasibi transfer standard

The extinction coefficient used by all participants in the comparison study was 308.5 cm^{-1} atm^{-1} base e.[14]

4. RESULTS

The final ozone concentrations for each participant are tabulated as follows:

TABLE I

Ozone Concentrations

(Part per million)

Data Pt.	NBS	EPA-RTP	JPL	ARB
1	0.689	0.683	0.700	0.681
2	0.109	0.110	0.112	0.109
3	0.605	0.597	0.612	0.599
4	0.185	0.184	0.189	0.183
5	0.494	0.490	0.503	0.494
6	0.301	0.299	0.307	0.296
7	0.389	0.385	0.396	0.389
8	0.047	0.049	0.047	0.042

The NBS photometer was selected as the reference standard in the data analysis. As indicated in Table I the data show excellent agreement. A linear regression and correlation coefficient (r) analysis of the data confirms the agreement. From the analysis, the following relationships were obtained:

$$[O_3]_{NBS} = (1.013)\ [O_3]_{EPA\text{-}RTP} - (0.002) \qquad r = 1.0000$$

$$[O_3]_{NBS} = (0.985)\ [O_3]_{JPL} - (0.001) \qquad r = 1.0000$$

$$[O_3]_{NBS} = (1.004)\ [O_3]_{ARB} - (0.002) \qquad r = 0.9999$$

Figures 6, 7, and 8 are linear regression plots of the NBS ozone concentrations versus the EPA-RTP, JPL, and ARB ozone concentrations.

It should be emphasized that for the comparison, both NBS and JPL utilized transfer standards, rather than the actual absolute photometers. Additionally the EPA-RTP photometer was shipped from North Carolina and assembled at the ARB laboratory with a minimum of checkout. The ARB photometer was the only unit compared in its stationary position. With this realization, the agreement to within 2.8% of all four units is highly significant.

The analysis indicates an excellent agreement utilizing the ultraviolet photometric techniques. The linear regression confirmed the accuracy of each method, with the correlation coefficients indicating a high degree of precision.

5. ACKNOWLEDGMENT

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14. The value of k used in this work (308.5 cm^{-1} atm^{-1}) is based on an evaluation by R. Hampson and D. Garvin of measurements reported in the published literature:

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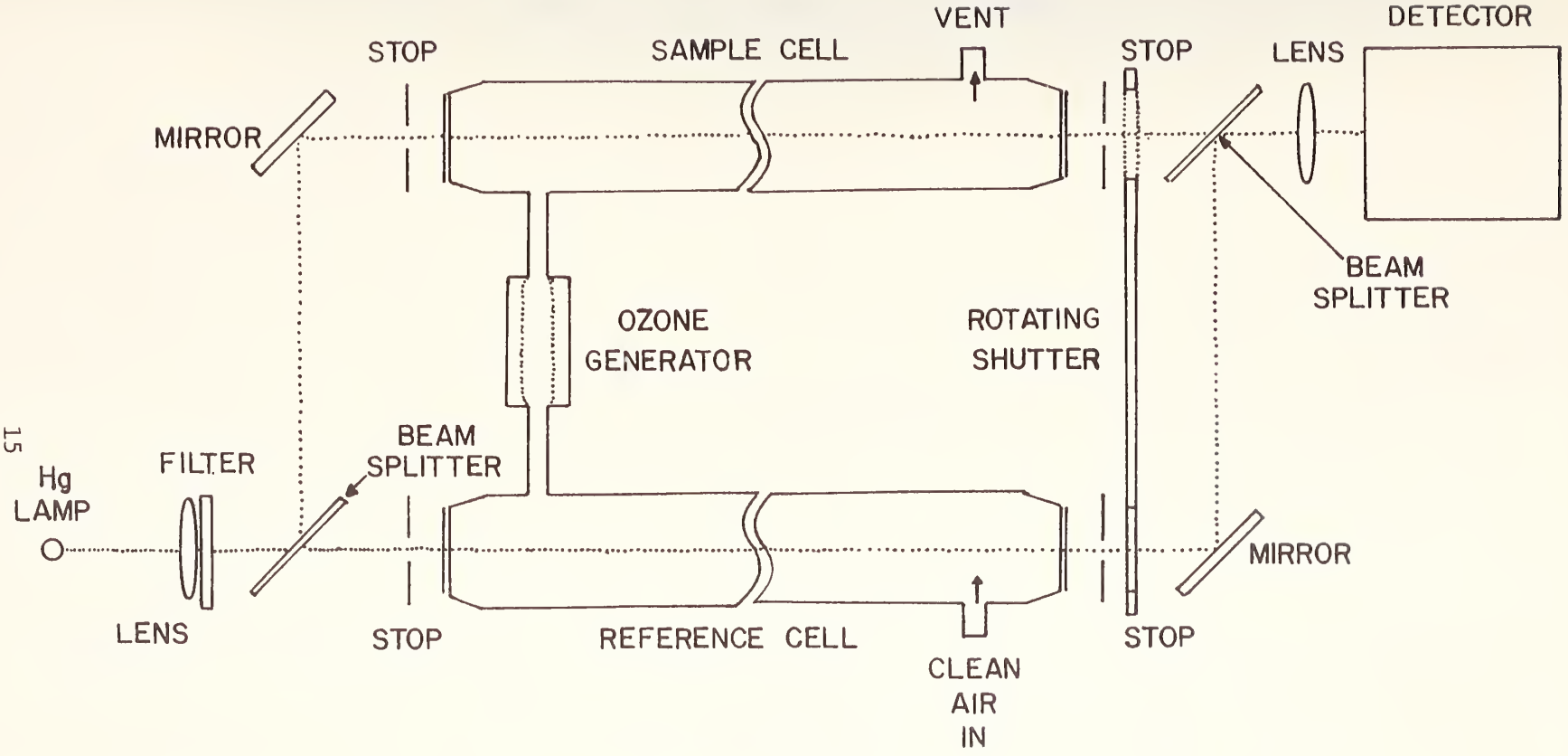

FIGURE 1 NBS ABSOLUTE PHOTOMETER

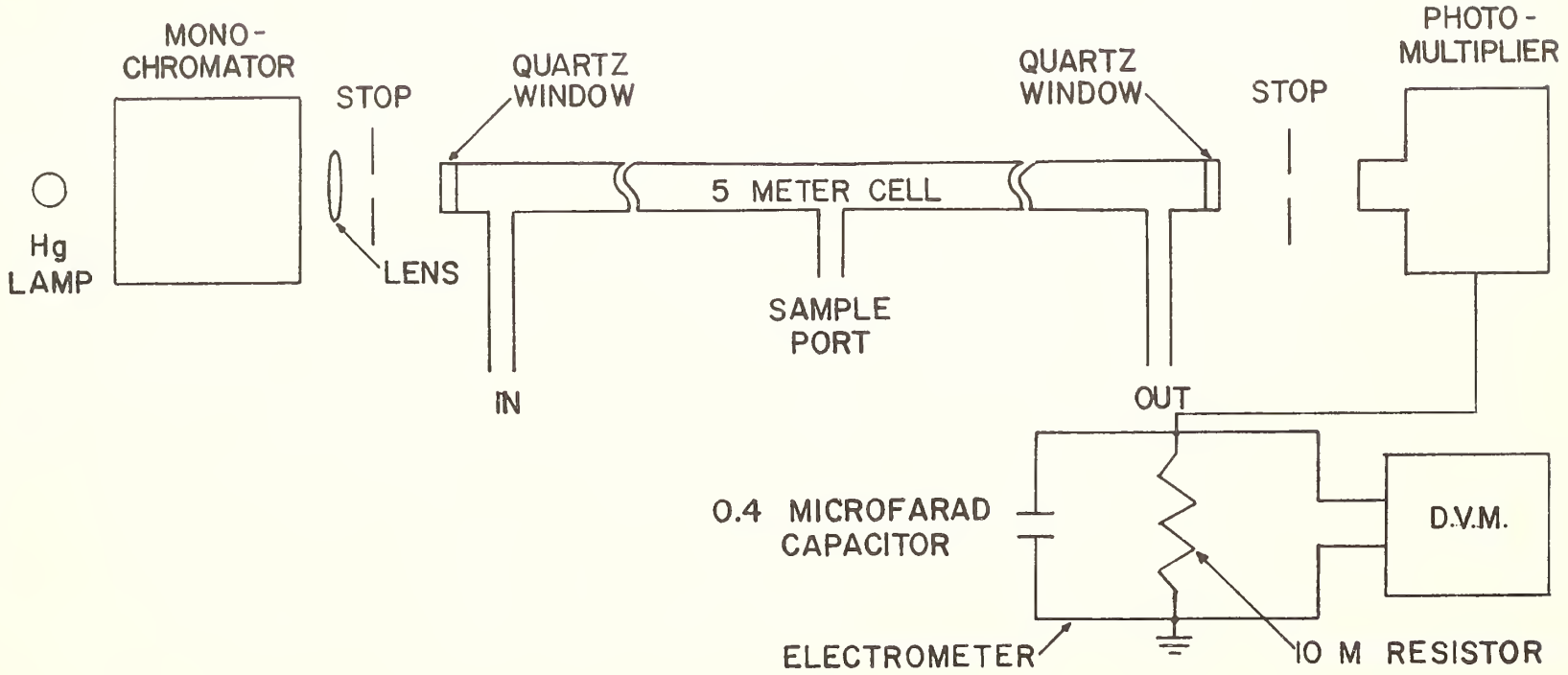

FIGURE 2 JPL ABSOLUTE PHOTOMETER

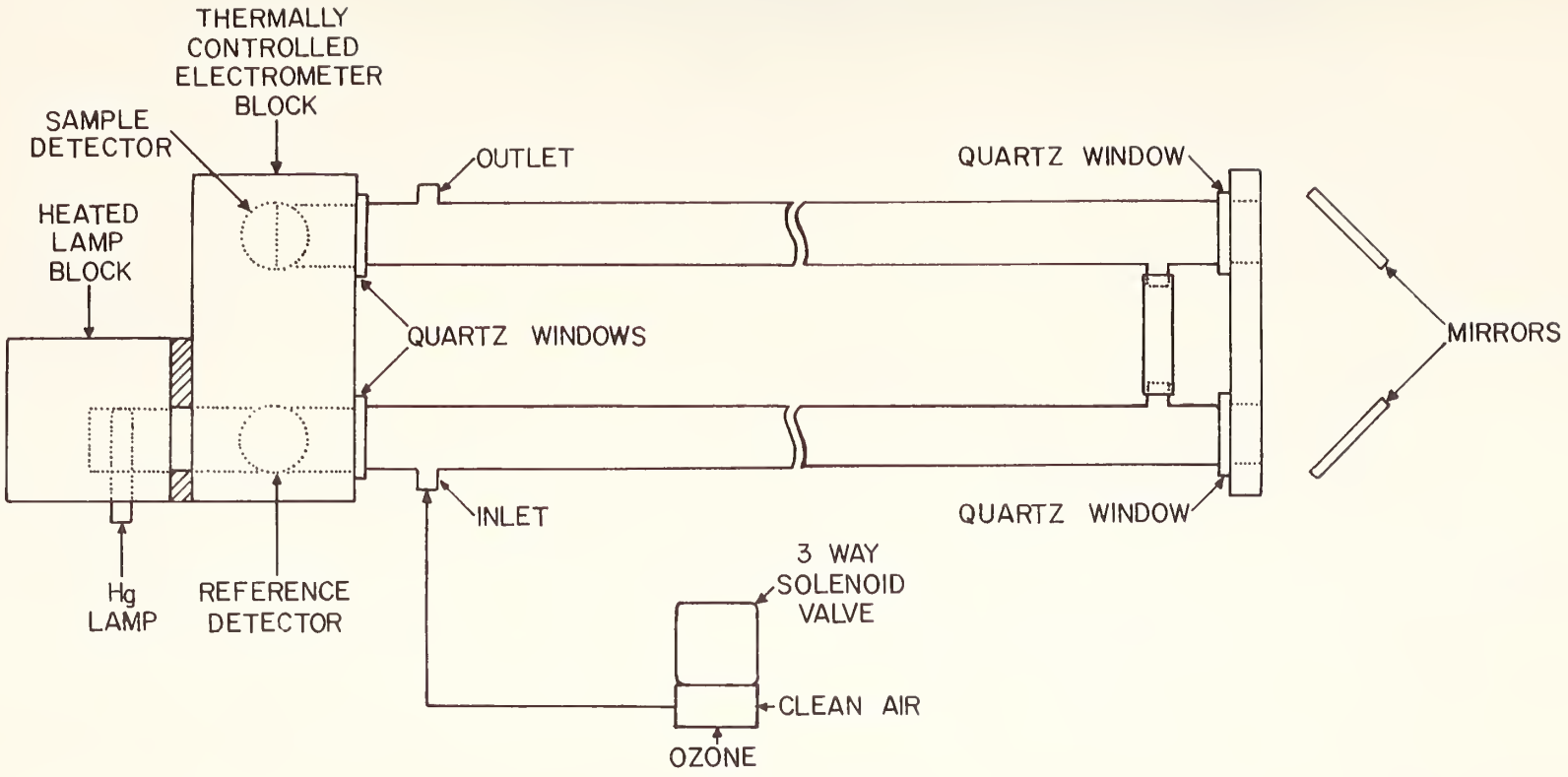

FIGURE 3 EPA ABSOLUTE PHOTOMETER

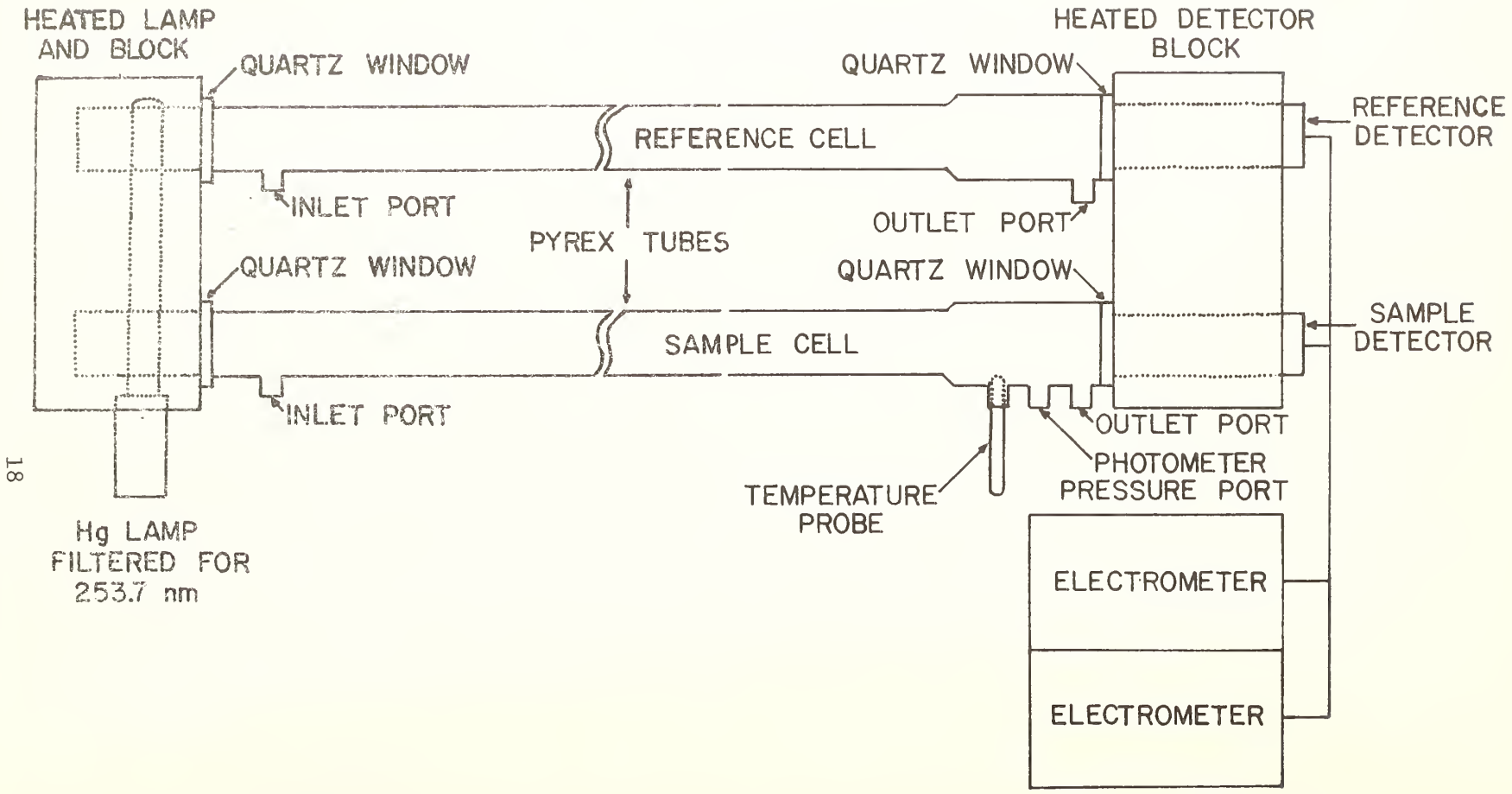

FIGURE 4 ARB ABSOLUTE PHOTOMETER

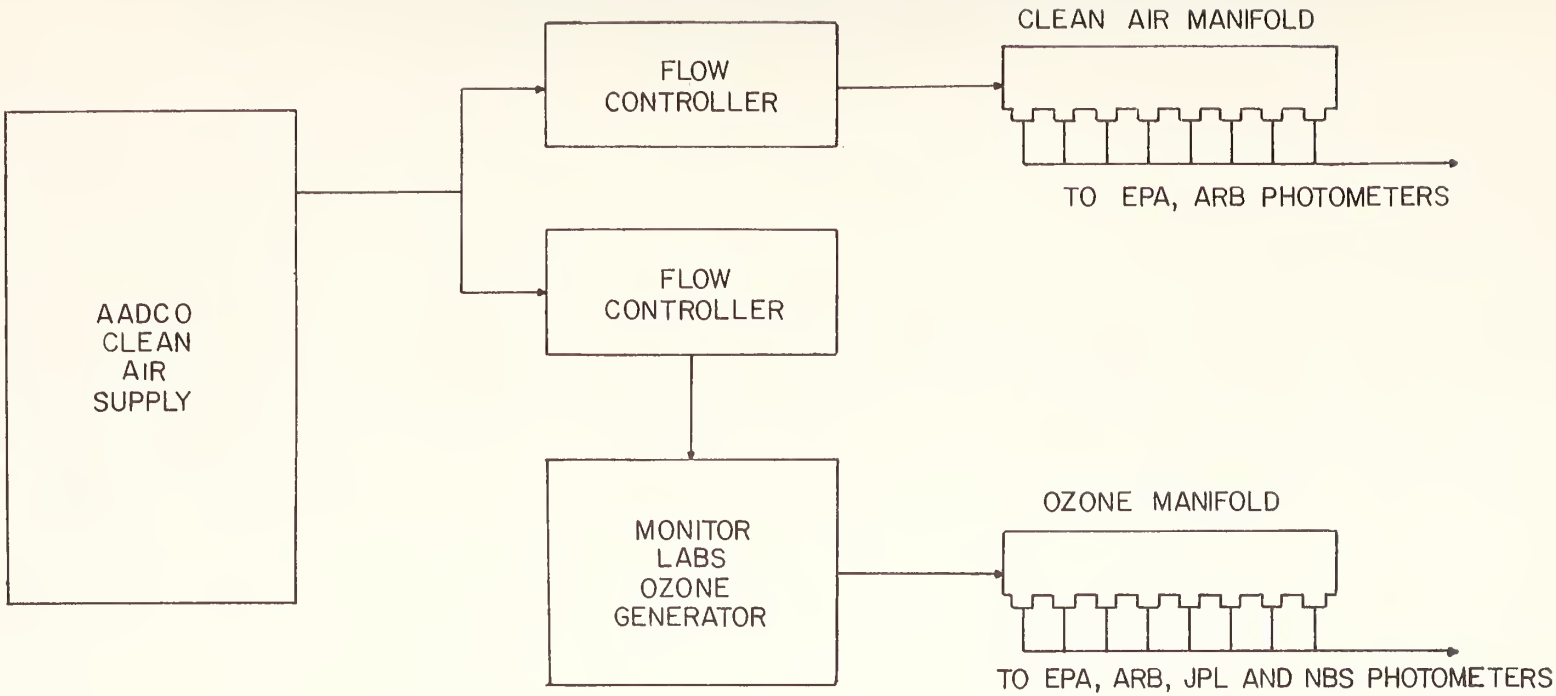

FIGURE 5 ZERO AIR AND OZONE SOURCES

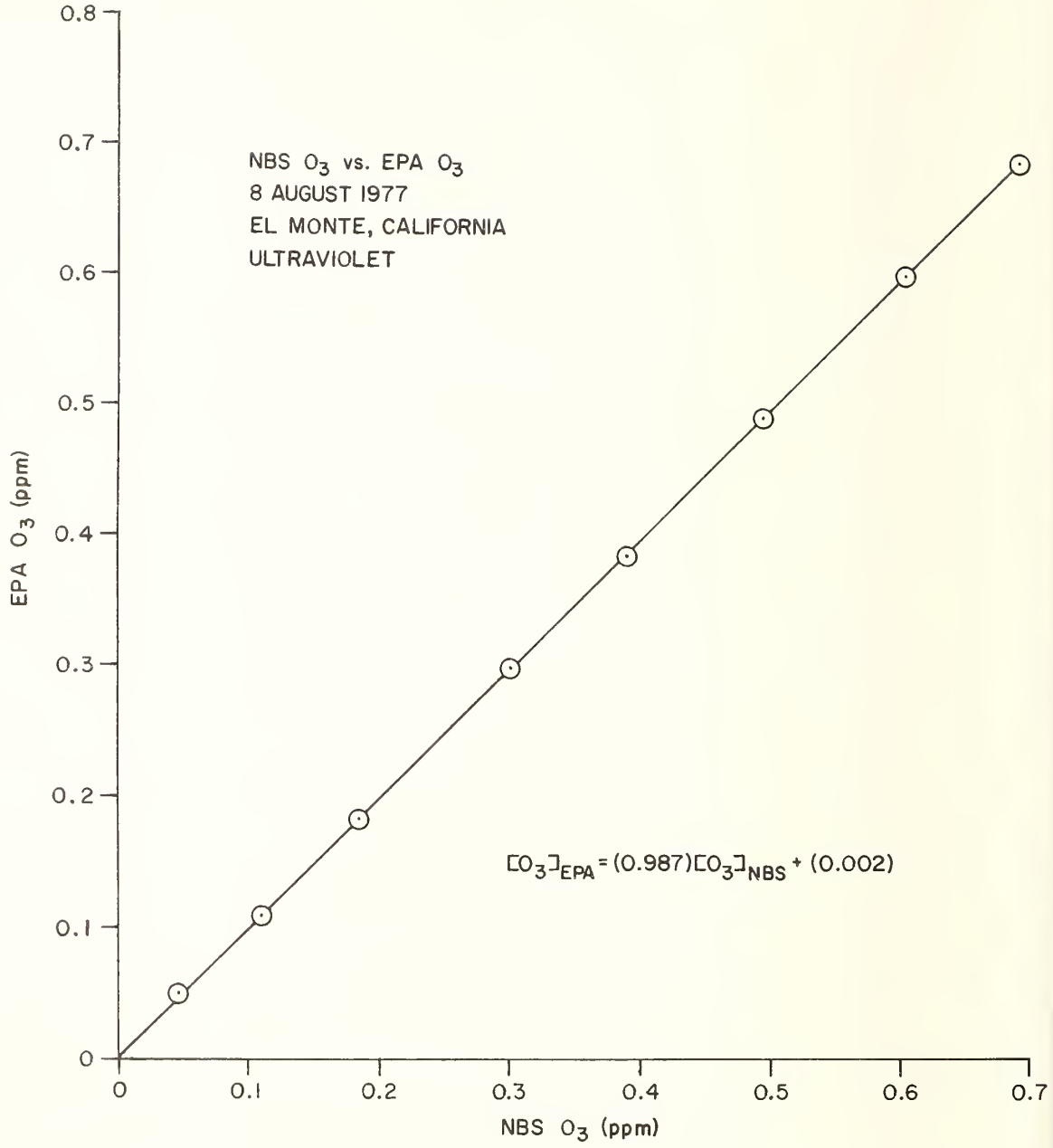
NBS O_3 vs. EPA O_3
8 AUGUST 1977
EL MONTE, CALIFORNIA
ULTRAVIOLET
$[O_3]_{EPA} = (0.987)[O_3]_{NBS} + (0.002)$
EPA O_3 (ppm)
NBS O_3 (ppm)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8

FIGURE 6

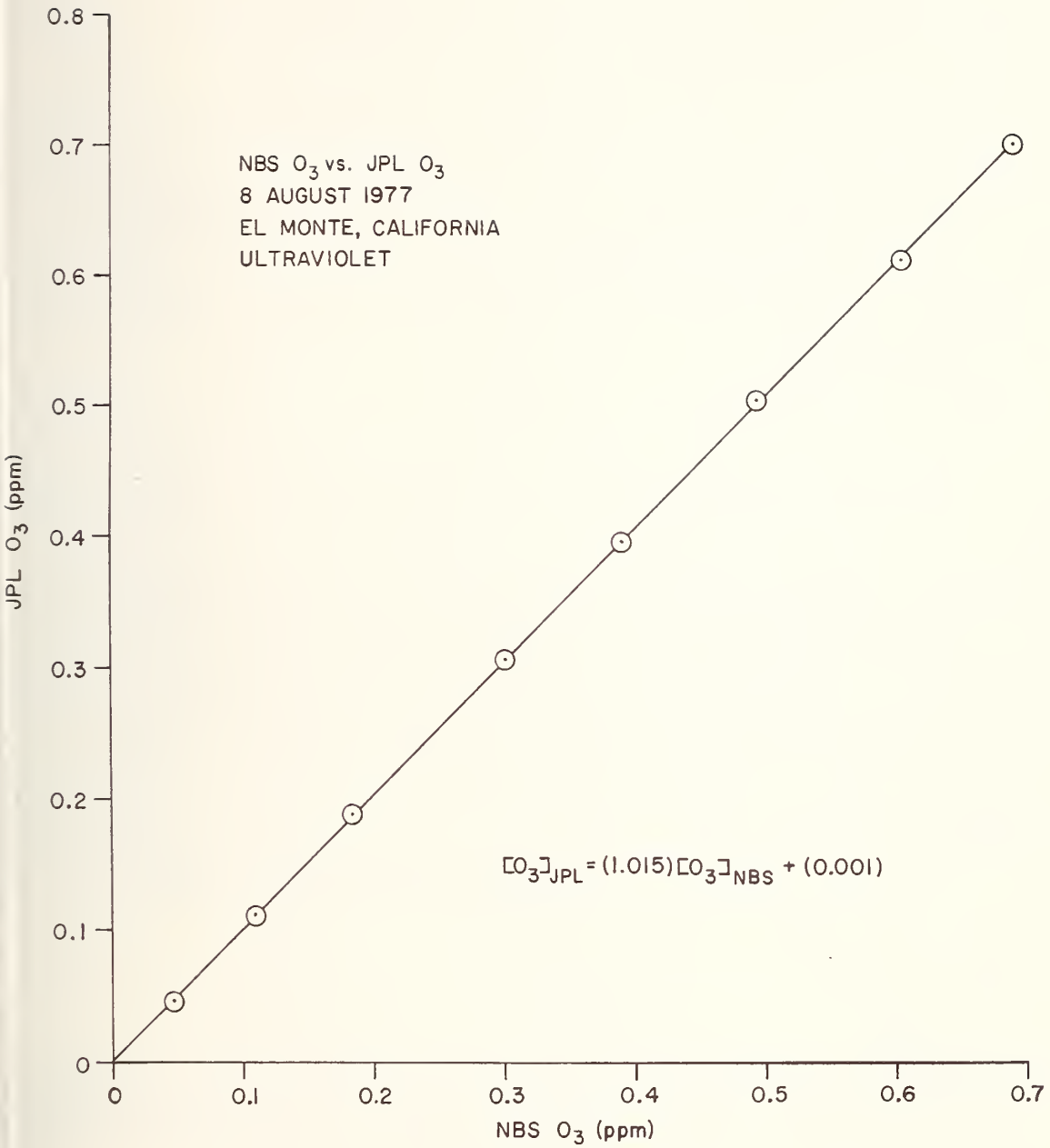
NBS O_3 vs. JPL O_3
8 AUGUST 1977
EL MONTE, CALIFORNIA
ULTRAVIOLET
$[O_3]_{JPL} = (1.015)[O_3]_{NBS} + (0.001)$
JPL O_3 (ppm)
NBS O_3 (ppm)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8

FIGURE 7

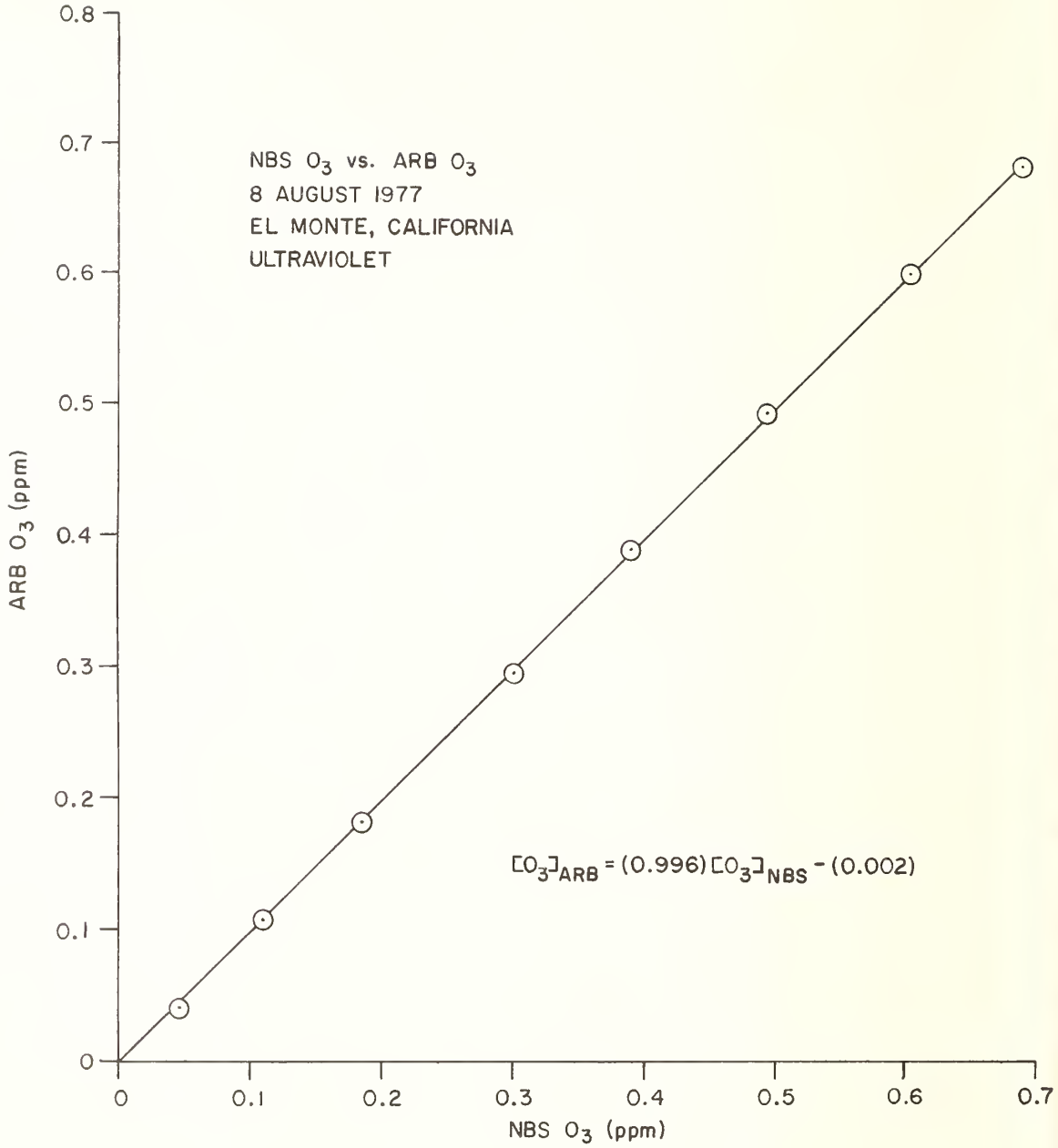
NBS O_3 vs. ARB O_3
8 AUGUST 1977
EL MONTE, CALIFORNIA
ULTRAVIOLET
$[O_3]_{ARB} = (0.996)[O_3]_{NBS} - (0.002)$
ARB O_3 (ppm)
NBS O_3 (ppm)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8

FIGURE 8

BS-114A (REV. 7-73)

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

In August 1977, an interlaboratory comparison study was initiated after the California Air Resources Board and the Environmental Protection Agency, Research Triangle Park independently designed and constructed ozone absolute ultraviolet photometers. These units augmented the photometers already designed and in use by the National Bureau of Standards and the Jet Propulsion Laboratory. Previous ozone comparisons involving indometry, ultraviolet photometry and gas phase titration have reported results differing by as much as ±30%. The protocol of this study, performed at the California Air Resources Board, Haagen-Smit Laboratory, El Monte, was a multi-point comparison using the NBS photometer as the standard. The results of the data analysis indicate excellent agreement with a total variation of 2.8%. Compared to the NBS photometer the EPA-RTP photometer read 1.3% low, the JPL photometer read 1.5% high and the ARB photometer read 0.4% low. The correlation coefficients of the same data sets were 1.0000, 1.0000 and 0.9999 respectively.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

Air pollution; atmospheric monitoring; calibration; ozone; ultraviolet photometry

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