

MIL-STD-1564A
11 NOV 1977

Superseding
MIL-STD-1564(USAF)
8 April 1975

MILITARY STANDARD
PROCEDURE FOR CALIBRATION AND ANALYSIS
OF TRACE CONTAMINANTS
IN AVIATOR'S BREATHING OXYGEN
BY INFRARED SPECTROSCOPY



FSC 6830

MIL-STD-1564A

DEPARTMENT OF DEFENSE
Washington DC 20301

PROCEDURE FOR CALIBRATION AND ANALYSIS OF TRACE CONTAMINANTS IN AVIATOR'S
BREATHING OXYGEN BY INFRARED SPECTROSCOPY

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2. Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: San Antonio ALC/SFRM, Kelly AFB TX 78241, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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1. SCOPE

1.1 Principal Statement. This Military Standard describes a procedure for determining trace gaseous contaminants in oxygen, nitrogen, and air by use of infrared spectroscopy.

2. REFERENCED DOCUMENTS (Not applicable)

3. DEFINITIONS

3.1 Absorbance. Absorbance (A) is defined as the negative logarithm to the base 10 of the transmittance, or

$$A = - \log_{10} T$$

3.2 Absorption. Absorption is the property of an absorbing material to transform radiation into other forms of energy.

3.3 Absorptivity. Absorptivity is defined as the absorbance divided by the product of the sample path length (b) and the concentration of the absorbing substance (c).

$$a = \frac{A}{bc}$$

3.4 Band, Absorption. An absorption band is a region of the absorption spectrum in which the absorbance passes through a maximum.

3.5 Baseline. A baseline is a straight line drawn tangent to the absorption spectrum which represents a function of the radiant power incident on the sample.

3.6 Beer's Law. The absorbance is directly proportional to the concentration of the absorbing material.

3.7 Blank. A blank is an analysis for the purpose of determining absorbance corrections necessary due to the presence of contaminants in the dilution gas and inherent characteristics of the instrument.

3.8 Concentration. Quantity of a given substance contained in a unit quantity of sample.

3.9 Gas, Dilution. Dilution gas is a pure gas used for diluting contaminant gases and contaminant gas standards.

3.10 Infrared. Infrared is that part of the electromagnetic spectrum which includes the range of wavelength (wavenumbers) from 0.78 μm (13000 cm^{-1}) to 300 μm (33 cm^{-1}).

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3.11 Limits, Procurement. Procurement limits are those limits in specifications which define the quality of product required from the supplier.

3.12 Limits, Use. Use limits define the minimum quality of product normally considered suitable for an intended purpose. They are a second level of quality.

3.13 $\mu\text{m Hg}$. This symbol represents the expression "micrometers of mercury." 1000 $\mu\text{m Hg}$ are equal to 1mm Hg.

$$(1 \mu\text{m} = 10^{-6} \text{ m})$$

3.14 mm Hg. This symbol represents the expression "millimeters of mercury." It is a unit of the absolute pressure in terms of the height of a column of mercury which a given pressure will support. For example, the average atmospheric pressure at sea level is 760 mm Hg.

3.15 Parameters, Energy. Energy parameters are instrument settings which alter absorption spectra and, as a result, affect precision and accuracy. Optimum parameters are selected to provide the best results for the specific material to be analyzed. These parameters include slit width, scan speed, and period control (or damping).

3.16 ppm. This symbol represents the expression "parts per million."

3.17 psig. This symbol represents the expression "pounds per square inch gauge." It is the unit of pressure normally read from a pressure gauge. The sum of the gauge pressure and the barometric pressure is the absolute pressure or true total pressure.

3.18 Scan. For purposes of this standard, scan means to operate the infrared analyzer and record the absorbance through a given range of wavelength. An infrared or IR scan refers to the absorption spectrum through the range 2.5 to 15.5 μm (4000. to 645 cm^{-1}).

3.19 Spectrum, Absorption. An absorption spectrum is a plot of a function of absorbance against wavelength.

3.20 Transmittance. Ratio of the radiant power transmitted by a sample to the radiant power transmitted by some reference material which does not exhibit any significant absorption.

3.21 Wavelength. Wavelength is the distance measured along the line of propagation, between two points which are in phase on adjacent waves. It may be represented by the symbol λ . The unit used to express wavelength is the "micrometer (μm)."

3.22 Wavelength, Analytical. An analytical wavelength is a wavelength selected for absorbance measurement from which contaminant concentration is to be determined.

3.23 Wavenumber. The wavenumber is the number of waves per unit length. It may be represented by the symbol ω . The unit used in this standard is "reciprocal centimeters" or " cm^{-1} ." It describes the number of waves per centimeter. Wavenumber is related to the wavelength.

$$\omega \text{ (in } \text{cm}^{-1}\text{)} = \frac{10\,000}{\lambda \text{ (in } \mu\text{m)}}$$

4. GENERAL REQUIREMENTS

4.1 Instrument Parameters. Instrument parameters shall be set in accordance with procedures established by the instrument manufacturer. The analyst shall check parameters prior to calibration and reset if necessary. Parameters shall be checked at a minimum frequency of once every 30 days to ensure that they have not been changed since the previous calibration. A record of settings and check shall be kept in a log book. Instrument parameters shall be the same for calibration and sample analysis. If conditions arise which necessitate changing these parameters, the instrument shall be recalibrated.

4.2 Pressure of Analysis. Calibration curves are prepared for contaminants at 50 and 135 psig.

4.2.1 Field Samples. Samples should be analyzed at 135 psig but may be analyzed at pressures between 50 and 135 psig provided the absorbance values are normalized to the appropriate pressure of the calibration curve. Samples of insufficient quantity to pressurize the gas cell to 50 psig shall not be analyzed.

4.2.2 Contractor Samples. Samples must be analyzed at 135 psig to ensure detection of all contaminants in accordance with the specification. Government contract requires submittal of sufficient sample to analyze at 135 psig. Samples of insufficient quantity to pressurize the gas cell to 135 psig shall not be analyzed.

4.3 Pressure of System. Prior to each pressurization step of Section 5, check the valves to ensure that only those parts of the system will be pressurized which can withstand the intended pressure.

4.4 Carbon Dioxide Interference. In some IR gas cell systems such as that manufactured by Perkin Elmer Corp, a significant length of the light path passes through the atmosphere. In such systems, atmospheric carbon dioxide and water vapor will always be observed in the IR spectrum unless the light path is enclosed and purged with an inert gas. When it is impractical to purge the light path, the absorbance due to atmospheric carbon dioxide shall be determined daily with cell evacuated. Each sample should be then corrected for this blank value.

4.5 Maintenance. After a maintenance operation has been performed on the instrument, its calibration shall be checked. This may be accomplished by preparing a standard of known concentration and comparing its absorbance with the appropriate calibration curve. A significant difference from the expected absorbance shall be cause for recalibration.

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4.6 Manifold and Tubing. Manifold and tubing shall be constructed of stainless steel or other oxygen compatible metal. Internal volume of this system shall be kept to minimum.

4.7 Alternate Methods. Section 5 contains approved methods for mixing standards and measuring absorbance. Other methods may be acceptable. Alternatives to the approved methods shall be submitted to San Antonio ALC/SFQT for review and for possible inclusion in this standard. Final acceptance of the method and technique employed at each laboratory designated to perform analysis of Aviator's Breathing Oxygen (ABO) shall depend on acceptable performance in the ABO Correlation Program.

4.8 Rounding-Off. For purposes of determining conformance to a procurement or use limit, an observed or calculated value shall be rounded-off so that the last place retained has the same position in relation to the decimal point as the last right-hand place of figures used in expressing the limiting value. The figure in the last place retained shall remain the same or shall have one added to it as described below. See Table I for examples.

4.8.1 When the next figure to the right of the last place to be retained is less than 5, do not change the figure in the last place retained.

4.8.2 When the next figure to the right of the last place to be retained is greater than 5, add 1 to the figure in the last place retained.

4.8.3 When the figures to the right of the last place to be retained are 5 followed by one or more nonzero digits, add 1 to the figure in the last place retained.

4.8.4 When the figures to the right of the last place to be retained are 5 alone or 5 followed by any number of zeros, add 1 to the figure in the last place retained if it is odd; do not change the figure in the last place retained if it is even.

5. DETAILED REQUIREMENTS

5.1 Typical Apparatus.

5.1.1 Infrared Spectrophotometer - A double beam, optical null, automatic recording spectrophotometer capable of making absorbance measurements through the range 2.5 to 15.5 μm utilizing a gas cell which has an optical pathlength of no less than 10 meters.

5.1.2 The infrared chart paper should have an abscissa scale in wavenumbers or wavelengths, and an ordinate scale in absorbance units.

5.1.3 Pump high vacuum, Kinney KC-8 or equivalent. Use no oil; fill pump with tricresylphosphate. Other sources: Welch, Precision.

5.1.4 Manifold and Tubing - A manifold with suitable outlets to permit assemblage of the gas system.

TABLE I. Examples of Rounding-Off

Procurement/ Use Limit	Observed/ Calculated Value	Rounded- Off Value	Conforms to Limit	Reference Paragraph
2 max	2.15	2	Yes	4.7.1
99.5 min	99.43	99.4	No	4.7.1
5 max	5.75	6	No	4.7.2
25 max	25.9	26	No	4.7.2
1 max	1.58	2	No	4.7.3
2 max	2.51	3	No	4.7.3
10 max	10.5	10	Yes	4.7.4
5 max	5.50	6	No	4.7.4

5.1.4.1 Valves, stainless steel, diaphragm packless, Matheson 939 series or equal. Other sources: Whitey, Robbins, Nu-Pro, Hoke.

5.1.4.2 Hose, flexible, stainless steel, teflon lines, braided, as needed.

5.1.4.3 Tubing, stainless steel, as needed.

5.1.4.4 Tube fittings, stainless steel, as needed.

5.1.5 Gauge, test, with minimum range of 0 to 150 psig in increments of one psi or less. Heise and Ashcroft are sources of satisfactory gauges.

5.1.6 Manometer, Mercury, Meriam Model 310EF10 or equivalent. Wallace and Tiernan is an alternate source.

5.1.7 Gauge, McLeod, Sargent-Walch P/N S-39798-10 or equivalent.

5.1.8 Regulators for compressed gases, as required. Air Products, Airco, and Matheson are sources.

5.1.9 Gas chromatography gas sampling valve (Perkin Elmer No. 154-0068 or equivalent) with 0.25, 1.0, and 5.0 ml sample loops. Carle and Barber Coleman are other sources of valves.

5.1.10 Syringe and needle 1.0 ml, 2.5 ml, 5.0 ml. Disposable types may be used. Hamilton and Precision are sources.

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- 5.1.11 Wet test meter. Available from Precision.
- 5.1.12 Gas cell with optical pathlength of no less than 10 meters; suitable for pressurization to not less than 135 psig.
- 5.1.13 Water vapor saturation - Gas Washing Bottle, Sargent P/N S-39590 or equivalent.
- 5.1.14 Barometer, Mercury, Fisher Catalog No. 2-383 or equivalent.
- 5.1.15 Septum Device.
 - 5.1.15.1 Tube fitting, Male Connector, stainless steel, 3/16 inch, Swagelok P/N SS-300-1-2 or equivalent.
 - 5.1.15.2 Septum, Gas Chromatography, Cylindrical Type, Half-Hole, Analabs P/N HGC-138 or equivalent.

5.2 Preparation and Assembly of Apparatus. All valves, fittings, hose, manifold blocks, mixing cylinders, and gauges must be cleaned with trichloroethane prior to assembling the gas system. After washing with this solvent and drying, all parts shall be assembled using teflon tape.

5.3 Gas Requirements.

5.3.1 Contaminant Gases. Following gases and chemicals are required for instrument calibration and must be at least 98.5% pure.

- 5.3.1.1 Methane.
- 5.3.1.2 Carbon Dioxide.
- 5.3.1.3 Nitrous Oxide.
- 5.3.1.4 Ethylene.
- 5.3.1.5 Various Halogenated Refrigerants.
- 5.3.1.6 Ethane.
- 5.3.1.7 Acetylene.
- 5.3.1.8 Various Halogenated Solvents.

5.3.2 Dilution Gases. Following gases are required for instrument calibration and must meet the purity requirements of a zero grade gas.

- 5.3.2.1 Oxygen.
- 5.3.2.2 Nitrogen.

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5.4 Preparation and Scanning of Gas with Known Standard Concentration. This shall be accomplished by the volume measurement method (5.5) or the partial pressure method (5.6).

5.5 Volume Measurement Method.

5.5.1 Gas System. An acceptable configuration of the gas system is diagramed in figures 1 and 2.

5.5.2 Determination of Volume of Mixing Cylinder and Manifold.

- a. Assemble the gas system such that gas from the vent valve of the manifold passes through a water vapor saturator into a wet test meter.
- b. Affix a thermometer to the mixing cylinder so that the bulb makes contact with the surface of the cylinder.
- c. Pressurize the mixing cylinder and manifold to near the rated pressure of the mixing cylinder.
- d. Allow cylinder to equilibrate to within 1 C⁰ of room temperature.
- e. Record pressure (P₁) to the nearest psi.
- f. Vent the cylinder and manifold slowly through the saturator and the wet test meter to near atmospheric pressure.
- g. Allow cylinder to equilibrate to within 1 C⁰ of room temperature.
- h. Record pressure (P₂) to the nearest psi.
- i. Record volume (V₁) registered by the wet test meter to the nearest 0.1 liter.
- j. Record room temperature to the nearest 0.1 C⁰.
- k. Record barometric pressure to the nearest 0.1 mm Hg.
- l. Calculate the container and manifold volume (V) as follows (see Appendix A, 10.1 for example).

$$V = \frac{K (P_b - P_w) V_1}{P_1 - P_2}$$

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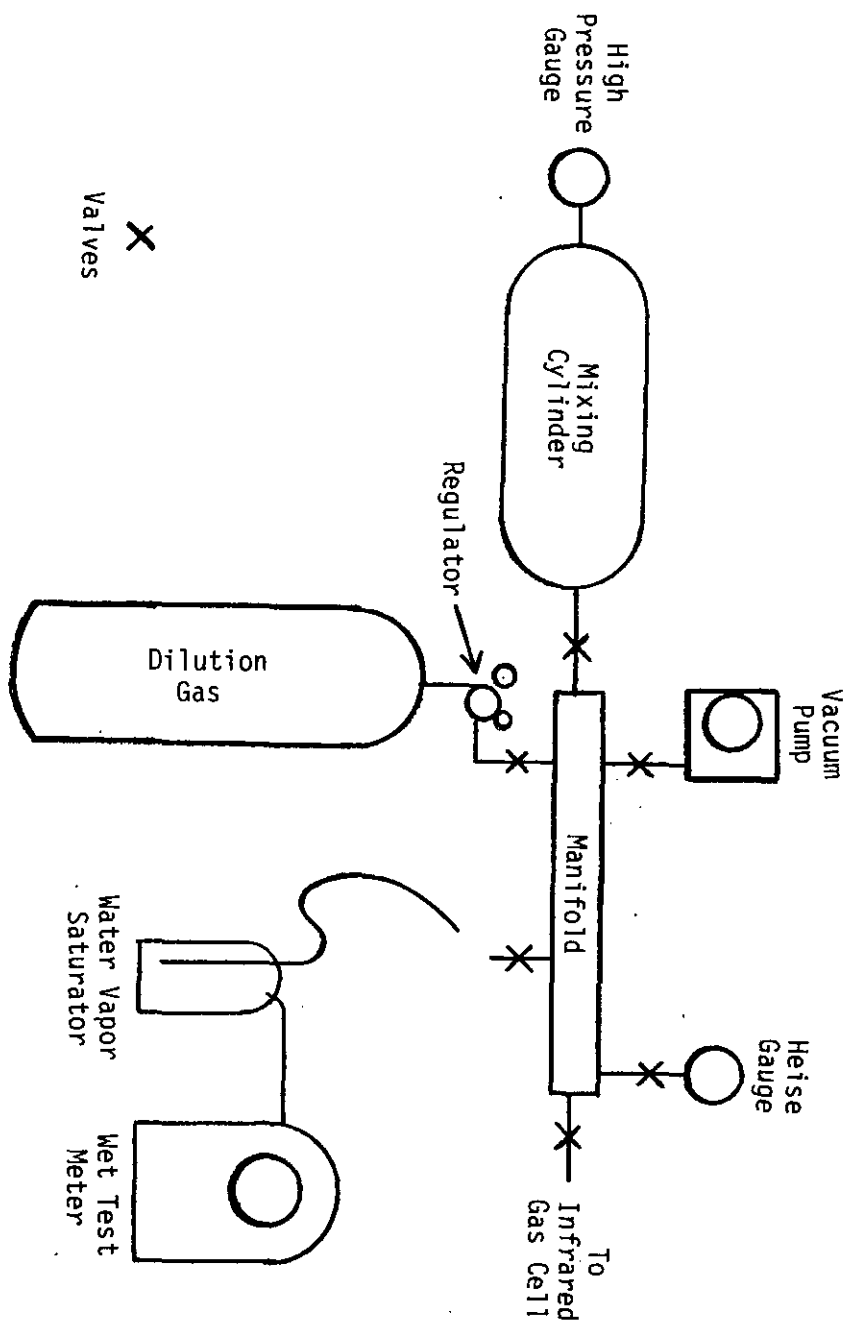


FIGURE 1. Gas System for Determination of Volume of Mixing Cylinder and Manifold.

Where V_1 = Volume recorded by wet test meter in liters

P_b = Barometric pressure in mm Hg corrected for temperature and altitude

P_w = Vapor pressure of water in mm Hg

P_1 = Initial Pressure

P_2 = Final Pressure

K = 0.01934 psi/mm Hg

5.5.3 Purging of Gas System.

- a. Evacuate entire gas system. 1/
- b. Pressurize mixing cylinder to approximately 400 psig with dilution gas. Pressurize gas cell to 135 psig with gas from mixing cylinder. Let stand for 5 to 10 minutes. 2/
- c. Vent and evacuate entire system.
- d. Pressurize mixing cylinder to approximately 400 psig with dilution gas. Pressurize gas cell to 135 psig with gas from mixing cylinder.
- e. Run IR scan. If there is no observable absorbance, blank values are zero. Proceed to 5.5.5.
- f. If there is observable absorbance repeat procedure "c" and "d" above.
- g. Run IR scan. If absorbance is observed again repeat procedure "c" and "d" until IR scans show no absorbance or the absorbance remains constant. Retain final scan for blank determination (5.5.4).

1/ The effectiveness of purging is enhanced by evacuating the system to low pressures. Evacuating to higher pressures may increase the number of purge cycles required. No maximum pressure of evacuation is specified because the final criteria of no observed or constant absorbance determines when purging is complete.

2/ Pressurization of system may cause high boiling impurities to condense on interior surfaces. Five to ten minutes should be sufficient time to allow these impurities to vaporize and thereby be detectable. This stand time should be followed whenever the gas cell is pressurized.

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h. Vent gas cell to 50 psig. Run IR scan and save for blank determination (5.5.4).

5.5.4 Determination of Blank Values. Reproducible absorbance indicates either the presence of contaminants in the dilution gas or inherent characteristics of the instrument (see also 4.3). These measurable absorbances must be included as blank values when determining absorbance values for the calibration curves. Using the wavelengths or wavenumbers of typical contaminants listed in Table II, evaluate the spectra saved from 5.5.3. Record these absorbance values for use in preparing the calibration curves.

5.5.5 Addition of Contaminant Gas; Dilution; Scanning. Known quantities of contaminant gas shall be transferred to the mixing cylinder by use of a gas chromatograph (GC) gas sampling valve or by use of a syringe and septum device.

5.5.5.1 GC Gas Sampling Valve Method. Assemble GC valve and lecture bottle of contaminant gas into gas system as shown in figure 2. Evacuate gas cell, mixing cylinder, manifold, and hoses back to dilution gas cylinder valve. Cut off pump, close all valves and start contaminant gas flowing through gas sample valve loop and out to the atmosphere. Start dilution gas flowing and immediately turn handle on gas sampling valve so that the standard sample loop is coupled to the dilution gas stream. Turn off the flow from the lecture bottle and pressurize the mixing cylinder and manifold to a predetermined pressure with dilution gas. This pressure shall be as described in 5.5.5.2.m. Pressurize the cell with gas from the mixing cylinder. Check instrument zero and scan through the absorption band of interest for the contaminant gas in the cell. Vent gas cell to 50 psig and rescan. Dilutions are made by repressurizing with dilution gas after venting operation. Calculate concentration of standard in accordance with paragraph 5.5.5.3.

5.5.5.2 Syringe and Septum Method. Assemble two septum devices; each shall consist of a 1/8 or 3/16 inch male connector tube fitting and a half-hole septum. One septum device shall be placed in the gas system as shown in figure 2. The other shall be fitted to the lecture bottle of contaminant gas via a regulator.

- a. Evacuate mixing cylinder, manifold, and septum device.
- b. Insert syringe of appropriate volume into septum connected to lecture bottle.
- c. Adjust pressure regulator of lecture bottle so that pressure is sufficient to move the plunger out of the syringe barrel.
- d. Remove plunger to allow purging of the septum device and regulator. Purging will be aided if the lecture bottle valve is turned off, and the pressure to the regulator and septum device is allowed to

decrease to near zero. Turn the lecture bottle valve on and off to repeat the process. Repeat this process a third time.

e. Remove the syringe from the septum device and replace the plunger discharging the contents.

f. Turn on the lecture bottle valve.

g. Insert the syringe into the septum device, and allow it to fill. Do not let the plunger come out of the syringe.

h. Remove syringe from the septum device and discharge its contents.

i. Repeat step g and proceed to step j.

j. Withdraw syringe from septum.

k. Immediately set plunger to the required volume and inject contaminant gas into the septum device of the mixing cylinder.

l. Close valve to the septum device of the mixing cylinder.

m. Pressurize mixing cylinder and manifold to a pressure sufficient to provide an amount of gas to pressurize the gas cell to 135 psig and yet allow for the remaining pressure in the mixing cylinder to be no less than 1/2 of its initial value.

n. Allow container to equilibrate until consecutive mixing cylinder surface temperature readings taken at one minute intervals are within 0.1°C. Record temperature and gauge pressure.

o. Record barometric pressure and room temperature.

p. Pressurize the evacuated gas cell to 135 psig using the material in the mixing cylinder and manifold.

q. Run IR scan.

r. Vent cell to 50 psig and run IR scan.

s. Calculate concentration in accordance with 5.5.5.3. If concentration is below lower limit of Table II, terminate procedure. If not, proceed to t.

t. Vent mixing cylinder to approximately 1/2 the pressure of m.

u. Allow cylinder to equilibrate until consecutive temperature readings at 1 minute intervals are within 0.1°C of each other.

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TABLE II. Analytical Data

Contaminant Gas	Location of Major <u>3/</u> Absorption Band		Range of Contaminant Gas Standard Concentration <u>4/</u>
	Analytical Wavelength (μm)	Analytical Wavenumber (cm^{-1})	Lower Limit
Ethane and Higher Hydrocarbons	3.40	2941	0.5 <u>5/</u>
Carbon Dioxide	4.24 13.91*	2360 719*	0.5
Nitrous Oxide	4.47	2235	0.1
Methane	3.40 <u>6/</u> 7.68	2941 1302	1.0
Ethylene	10.54	949	0.02
Acetylene	13.74	730	0.025
Freon 11	9.21* 11.83	1085* 845	0.1
Freon 12	8.62* 9.09* 10.87	1160* 1100* 920	0.1
Freon 13	8.24 9.05*	1213 1105*	0.1
(Freon 113 Freon TF)	8.93* 11.01* 12.22	1120* 908* 818	0.1
Freon 22	8.96 12.35*	1116 810*	0.1
Trichloroethylene	10.55* 11.75	948* 851	0.01

3/ When two or more locations are provided, alternate locations are marked with an asterisk (*). Alternate locations are provided for use when there is interference from another contaminant at the primary location, that is, when two contaminants absorb at the same wavelength.

TABLE II. Analytical Data - Continued

- 4/ The upper limit of the range of the contaminant gas standard concentration shall be twice the applicable use limit. For example, if the use limit for methane is 50 ppm, the concentrations of methane prepared shall include the range 1 to 100 ppm.
- 5/ As ethane equivalent.
- 6/ Calibration curves at both wavelengths are required.

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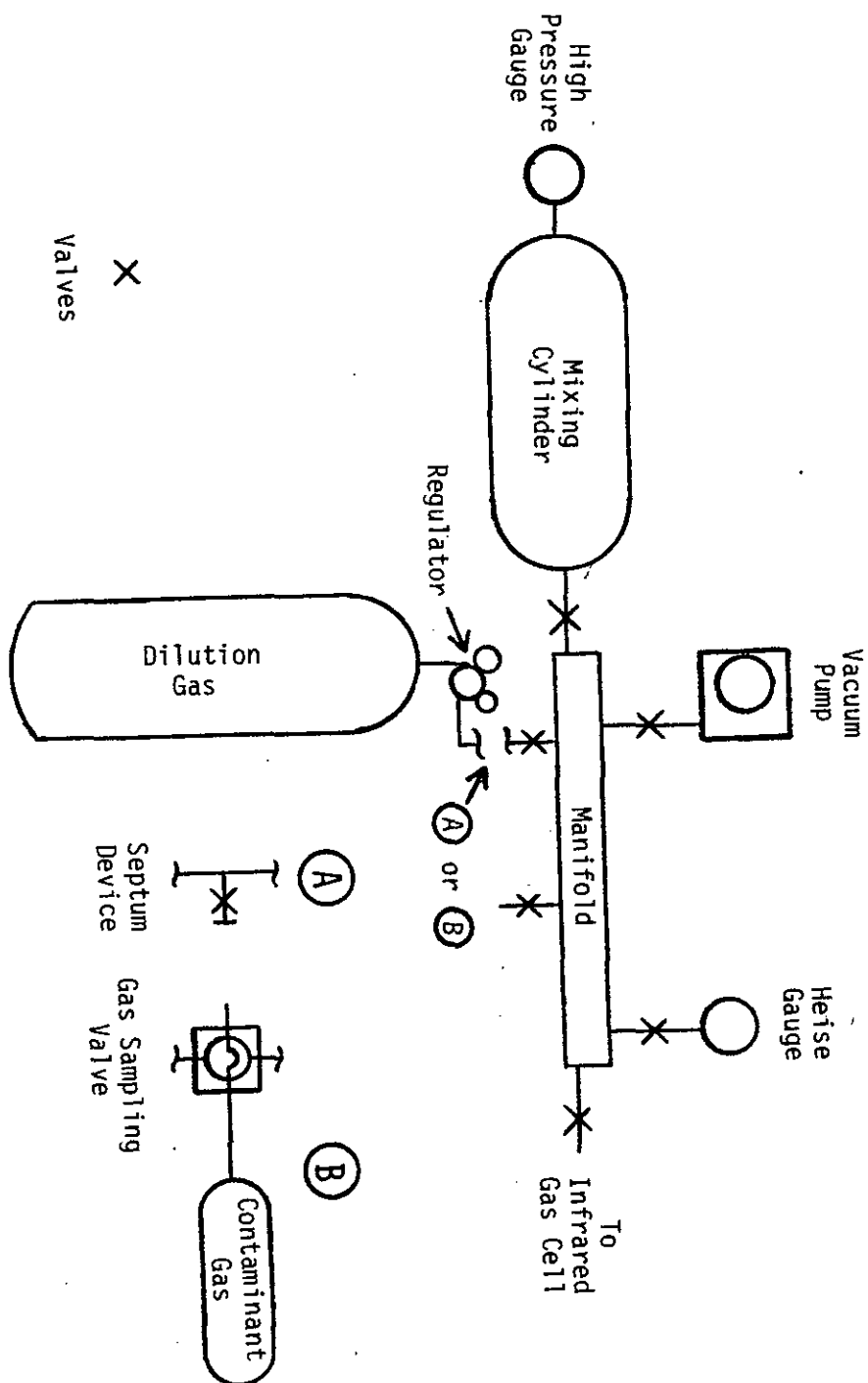


FIGURE 2. Gas System for Volume Measurement Method.

v. Record temperature and pressure.

w. Proceed to m.

5.5.5.3 Calculation of Concentration. See Appendix A for example.

a. Calculate initial concentration as follows:

$$C_o = \frac{10^3 V_s P_b}{V_c (P_g + P_b)}$$

where

C_o = Initial concentration in ppm

V_s = Volume of contaminant gas in ml. (This is volume injected with syringe or volume of gas sampling valve loop.)

V_c = Volume of mixing cylinder and manifold in liters

P_b = Barometric pressure in psi corrected for temperature and altitude

P_g = Gauge pressure of mixing cylinder

b. Calculate concentrations following dilutions as follows:

$$C_f = \frac{C_i T_f (P_i + P_b)}{T_i (P_f + P_b)}$$

where

C_f = Concentration following dilution in ppm

C_i = Concentration before dilution in ppm

P_i = Pressure before dilution in psi

P_f = Pressure following dilution in psi

T_f = Temperature following dilution in °K

T_i = Temperature before dilution in °K

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5.6 Partial Pressure Method.

5.6.1 Gas System. An acceptable configuration of the gas system is diagramed in figure 3.

5.6.2 Purging of Gas System and Blank Determination. See paragraphs 5.5.3 and 5.5.4.

5.6.3 Addition of Contaminant Gas; Dilution; Scanning.

5.6.3.1 Low Pressure Dilution. Evacuate the gas system. Record the barometric pressure (P_b), the pressure indicated by the McLeod Gauge (P_{mc}) and the manometer reading (M_1). Pressurize the system with contaminant gas to approximately 70 mm Hg. Record the manometer reading (M_2). Pressurize the cell to approximately 700 mm Hg with dilution gas. Record the manometer reading (M_3). At this point, the concentration of the contaminant gas (Z in ppm) is approximately 100 000 ppm. To adjust this concentration to the approximate final concentration for absorbance measurement, first calculate E and d as shown below.

$$\frac{100\ 000}{Z} = E \times 10^d$$

$$1 \ll E < 10$$

where

 d = whole number

Evacuate the system to approximately $\frac{700}{E}$ mm Hg. Record the manometer reading (M_4). Pressurize the cell to approximately 700 mm Hg (M_5). From this point $d-2$ successive evacuations (P_6, P_8, \dots) to approximately 70 mm Hg and pressurizations (P_5, P_7, \dots) to approximately 700 mm Hg with associated recording of data are performed.

5.6.3.2 High Pressure Dilution. Evacuate the system to approximately 76 mm Hg. Record the manometer reading (M_{2+2d}) and the barometric pressure. Close the valve to the manometer and McLeod Gauge and pressurize the system to 135 psig with dilution gas. Record the system pressure taken from the test gauge (P_g) and the barometric pressure (P_b). The concentration of the contaminant gas standard is determined with the following expression:

$$C_o = 10^6 \left(\frac{P_2 - P_1}{P_3} \right) \left(\frac{P_4}{P_5} \right) \left(\frac{P_6}{P_7} \right) \dots$$

where $P_1 = M_1 + P_{mc} + \Delta P_b - M_1$

$P_2 = M_1 + P_{mc} + \Delta P_b - M_2$

etc.,

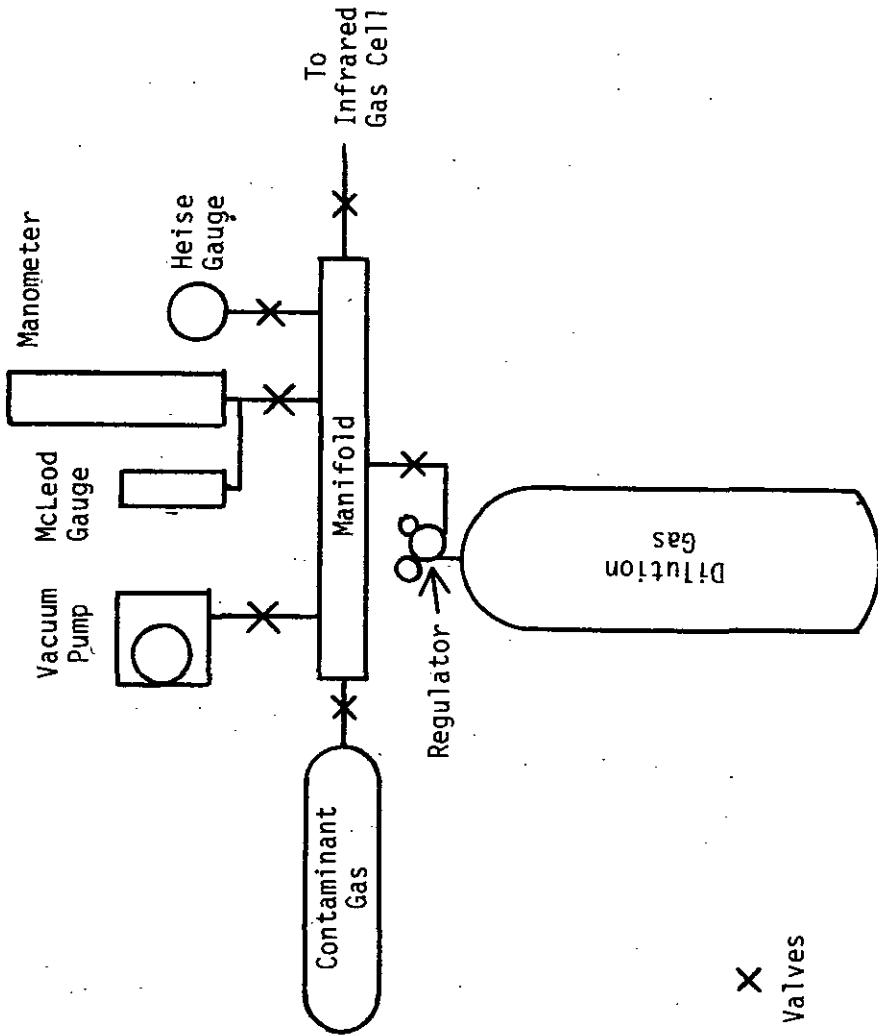


FIGURE 3. Gas System for Partial Pressure Method.

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and the final evacuation and pressurization are described by

$$P_{2+2d} = M_1 + P_{mc} + \Delta P_b - M_{2+2d}$$

converted to psi

$$P_{3+2d} = P_g + P_b$$

See paragraph 10.3 for an example.

5.6.3.3 Scanning. Run IR scan. Vent cell to 50 psig. Run IR scan. Repeat pressurization to 135 psig, venting to 50 psig, and running of IR scans until concentration is below lower limit of Table II. Concentrations are calculated as in paragraph 5.5.5.3.b.

5.7 Preparation of Calibration Curves. Select wavelengths λ_1 and λ_3 (see Figure 4), one on each side of the band to be measured. The absorption spectrum at these wavelengths should be as near as possible to the blank curve (B). Draw baseline (ab) tangent to the absorption spectrum at the selected wavelengths. Locate points (c and d) where the analytical wavelength (λ_2) intersects the absorption spectrum and the baseline. Read and record the values in absorbance units (A_1 and A_2) at these points. The absorbance due to the contaminant gas (A_c) is the difference between A_1 and A_2 , or

$$A_c = A_2 - A_1$$

If the blank determination has produced a measurable absorbance (A_b) at this analytical wavelength it shall be determined in a similar manner and subtracted from the contaminant gas absorbance (A_c),

$$A_s = A_c - A_b$$

where A_s is the contaminant gas standard absorbance. Values of A_s are plotted versus concentration on loglog graph paper and the calibration curve is drawn through these points.

5.7.1 If the blank determination has not produced a measurable absorbance then $A_b = 0$ and $A_s = A_c$.

5.7.2 Calibration curves shall be prepared at two analytical wavelengths for methane contaminant (see Table II). The reason for this is that methane, ethane, and higher hydrocarbons absorb at 3.40 μm . A correction for methane absorbance must be made when determining ethane and higher hydrocarbons in samples from the field.

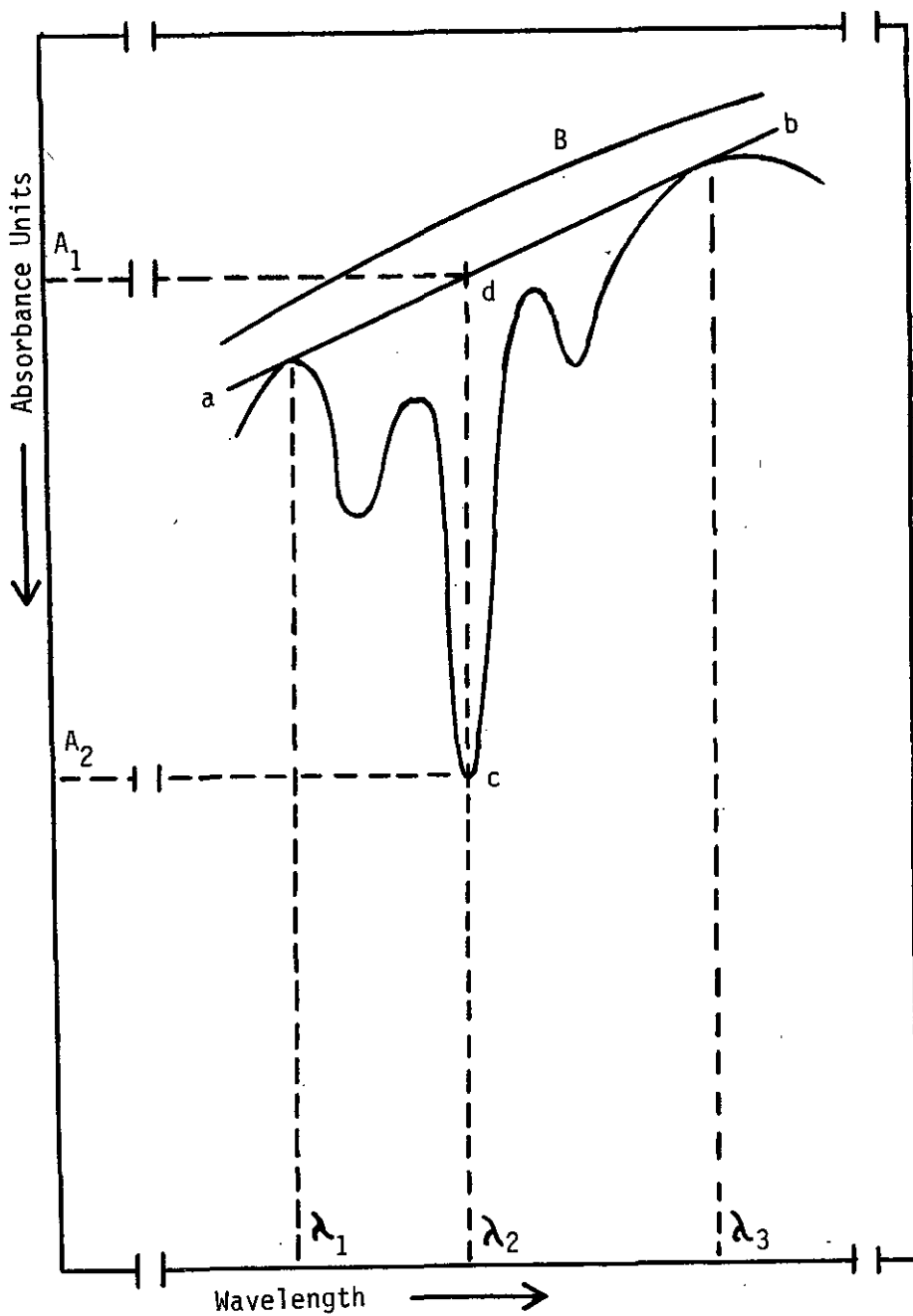


FIGURE 4. Determination of Contaminant Absorbance.

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5.8 Analytical Requirements.

5.8.1 Purging of Gas System. Purging is not required prior to routine analysis except when the system has been contaminated by a previous sample or other means. A contaminated system shall be purged in accordance with 5.5.3.

5.8.2 Transfer of Sample to Gas System. Connect the sampler to the manifold of the gas system. Evacuate the gas system to less than 50 $\mu\text{m Hg}$. Pressurize gas system to 135 psig with sample. If insufficient sample is available to pressurize the system to 135 psig, pressurize to the maximum pressure attainable. If the system cannot be pressurized to a minimum of 50 psig, the sample shall be considered insufficient for analysis.

5.8.3 Sample Absorbance Measurement and Contaminant Concentration Determination. Scan sample through the applicable range and determine the contaminant absorbance (A_C) in accordance with 5.7 for each contaminant present. Correct for atmospheric carbon dioxide absorbance, if applicable (see 4.3). If the cell was pressurized to a value other than 50 or 135 psig, the absorbance values shall be normalized to 135 psig in accordance with the expression

$$A_N = \frac{150 A_C}{P}$$

where

A_N = normalized absorbance

P = pressure at which sample was analyzed in psia ($P_g + P_b$)

The absorbance value at 50 or 135 psig or the normalized absorbance shall be used for determination of contaminant concentration. Except for ethane and higher hydrocarbons, contaminant concentrations can be read directly from the calibration curves.

5.8.4 To determine the concentration of ethane and higher hydrocarbons, first, determine the concentration of methane in the sample using the calibration curve prepared at the analytical wavelength 7.68 μm . Read the absorbance due to this methane concentration from the calibration curve for methane prepared at 3.40 μm . Subtract this absorbance from the sample absorbance at 3.40 μm . The difference is used to read the concentration of ethane and higher hydrocarbons (as ethane equivalents) directly from the ethane calibration curve. See paragraph 10.4.

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Appendix A Examples of Calculations

10.1 Calculation of Mixing Container Volume. A compressed gas cylinder rated at a maximum pressure of 1000 psig was pressurized to 950 psig. After cylinder equilibrated to room temperature, a pressure of 933 psig was recorded. After venting cylinder to 5 psig through the wet test meter and allowing the temperature to equilibrate, a pressure of 5 psig was recorded. A volume of 535.4 liters was registered on the wet test meter. Room temperature was 25.7°C. Height of mercury column was 753.6 mm.

a. Barometer reading was corrected to 0 °C and sea level gravity using Tables III and IV. The temperature of the barometer was 25.7 °C. The elevation at the site of the laboratory was 400 m.

$$P_b = (753.6 - 3.1 - 0.1) \text{ mm Hg}$$

$$P_b = 750.4 \text{ mm Hg}$$

b. The vapor pressure of water from Table V at 25.7 °C is 24.5 mm Hg.

c. Values were substituted in formula of paragraph 5.5.2.

$$V = \frac{0.01934 (750.4 - 24.5)}{933 - 5} (535.4) \text{ liters}$$

$$V = 8.10 \text{ liters}$$

10.2 Preparation of Calibration Curve by Volume Method. A calibration curve for nitrous oxide was to be prepared. The range of concentration for calibration was determined to be 3 to 0.5 ppm from Table II. A pressure of 400 psig was selected as sufficient for the system being calibrated. Similarly, a volume of 0.75 ml of contaminant gas was selected to provide a concentration of slightly greater than 3 ppm.

10.2.1 Nitrous oxide was drawn into a syringe calibrated at the 0.75 ml mark. This quantity was injected into a mixing container which had an internal volume of 8.10 liters. The mixing container was pressurized to approximately 400 psia (385 psig). The container was allowed to equilibrate to room temperature and the gauge pressure, barometric pressure, and temperature were recorded. IR scans at 135 and 50 psig were run. The mixing container was vented to approximately 200 psia (185 psig). After equilibration, the gauge pressure and temperature were recorded. This procedure

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TABLE III. Correction To Be Subtracted From Barometer Readings

t, °C	720 mm	730 mm	740 mm	750 mm	760 mm	770 mm	780 mm
15	1.76	1.78	1.81	1.83	1.86	1.88	1.91
16	1.88	1.90	1.93	1.96	1.98	2.01	2.03
17	1.99	2.02	2.05	2.08	2.10	2.13	2.16
18	2.11	2.14	2.17	2.20	2.23	2.26	2.29
19	2.23	2.26	2.29	2.32	2.35	2.38	2.41
20	2.34	2.38	2.41	2.44	2.47	2.51	2.54
21	2.46	2.50	2.53	2.56	2.60	2.63	2.67
22	2.58	2.61	2.65	2.69	2.72	2.76	2.79
23	2.69	2.73	2.77	2.81	2.84	2.88	2.92
24	2.81	2.85	2.89	2.93	2.97	3.01	3.05
25	2.93	2.97	3.01	3.05	3.09	3.13	3.17
26	3.04	3.09	3.13	3.17	3.21	3.26	3.30
27	3.16	3.20	3.25	3.29	3.34	3.38	3.42
28	3.28	3.32	3.37	3.41	3.46	3.51	3.55
29	3.39	3.44	3.49	3.54	3.58	3.63	3.68
30	3.51	3.56	3.61	3.66	3.71	3.75	3.80

TABLE IV. Reduction of Barometer to Gravity at Sea Level

Height Above Sea Level in Meters	Observed Height Of Barometer In Millimeters						
	500	550	600	650	700	750	800
10002	.02	.02
20004	.05	.05
30007	.07	.07
40009	.10	.10
50011	.12	.13
60012	.13	.14	
70014	.15	.16	
80016	.18	.19	
90018	.20	.22	
100018	.19	.20	.22	.24	
110019	.21	.22	.24		
120021	.23	.24	.26		
130022	.24	.26	.29		
140024	.26	.28	.31		
1500	.24	.26	.28	.30	.33		
1600	.25	.28	.30	.32			
1700	.27	.30	.32	.34			
1800	.28	.31	.34	.36			
1900	.30	.33	.36	.39			
2000	.31	.34	.38	.41			
2100	.33	.36	.40				
2200	.35	.38	.41				
2300	.36	.40	.43				
2400	.38	.42	.45				
2500	.39	.43	.47				

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TABLE V. Water Vapor Pressure

mm Hg

Temp °C	0.0	0.2	0.4	0.6	0.8
15	12.788	12.953	13.121	13.290	13.461
16	13.634	13.809	13.987	14.166	14.347
17	14.530	14.715	14.903	15.092	15.284
18	15.477	15.673	15.871	16.071	16.272
19	16.477	16.685	16.894	17.105	17.319
20	17.535	17.753	17.974	18.197	18.422
21	18.650	18.880	19.113	19.349	19.587
22	19.827	20.070	20.316	20.565	20.815
23	21.068	21.324	21.583	21.845	22.110
24	22.377	22.648	22.922	23.198	23.476
25	23.756	24.039	24.326	24.617	24.912
26	25.209	25.509	25.812	26.117	26.426
27	26.739	27.055	27.374	27.696	28.021
28	28.349	28.680	29.015	29.354	29.697
29	30.043	30.392	30.745	31.102	31.461
30	31.824	32.191	32.561	32.934	33.312
31	33.695	34.082	34.471	34.864	35.261
32	35.663	36.068	36.477	36.891	37.308
33	37.729	38.155	38.584	39.018	39.457
34	39.898	40.344	40.796	41.251	41.710

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TABLE VI. Data and Calculations for Volume Measurement Method

Calibrated Volume

Mixing Container: 8.10 liters

Syringe: 0.802 ml at 0.75 ml mark

Barometric Pressure

Height of mercury column: 747.9 mm

Temperature: 24.5 °C

Altitude: 400 m

$$P_b = (748.1 - 3.0 - 0.1) \text{ mm Hg} = 745.0 \text{ mm Hg}$$

$$P_b = 0.01934 (745.0) \text{ psia} = 14.41 \text{ psia}$$

Data and Calculated Results

P_g (psig)		C_o (ppm)			
388		3.546			
P_i (psig)	P_f (psig)	T_i (°C)	T_f (°C)	C_i (ppm)	C_f (ppm)
186.5	379.4	26.4	26.9	3.546	1.812
174.5	377.0	26.8	27.2	1.812	0.8757
187.5	378.0	26.6	27.2	0.8757	0.4515

Calculation of Initial Concentration

$$C_o = 10^3 \left(\frac{0.802}{8.10} \right) \frac{14.41}{388 + 14.41} = 3.546 \text{ ppm}$$

Calculation of Concentration after First Dilution

$$C_f = 3.546 \left(\frac{186.5 + 14.41}{379.5 + 14.41} \right) \frac{300.0}{299.5} = 1.812 \text{ ppm}$$

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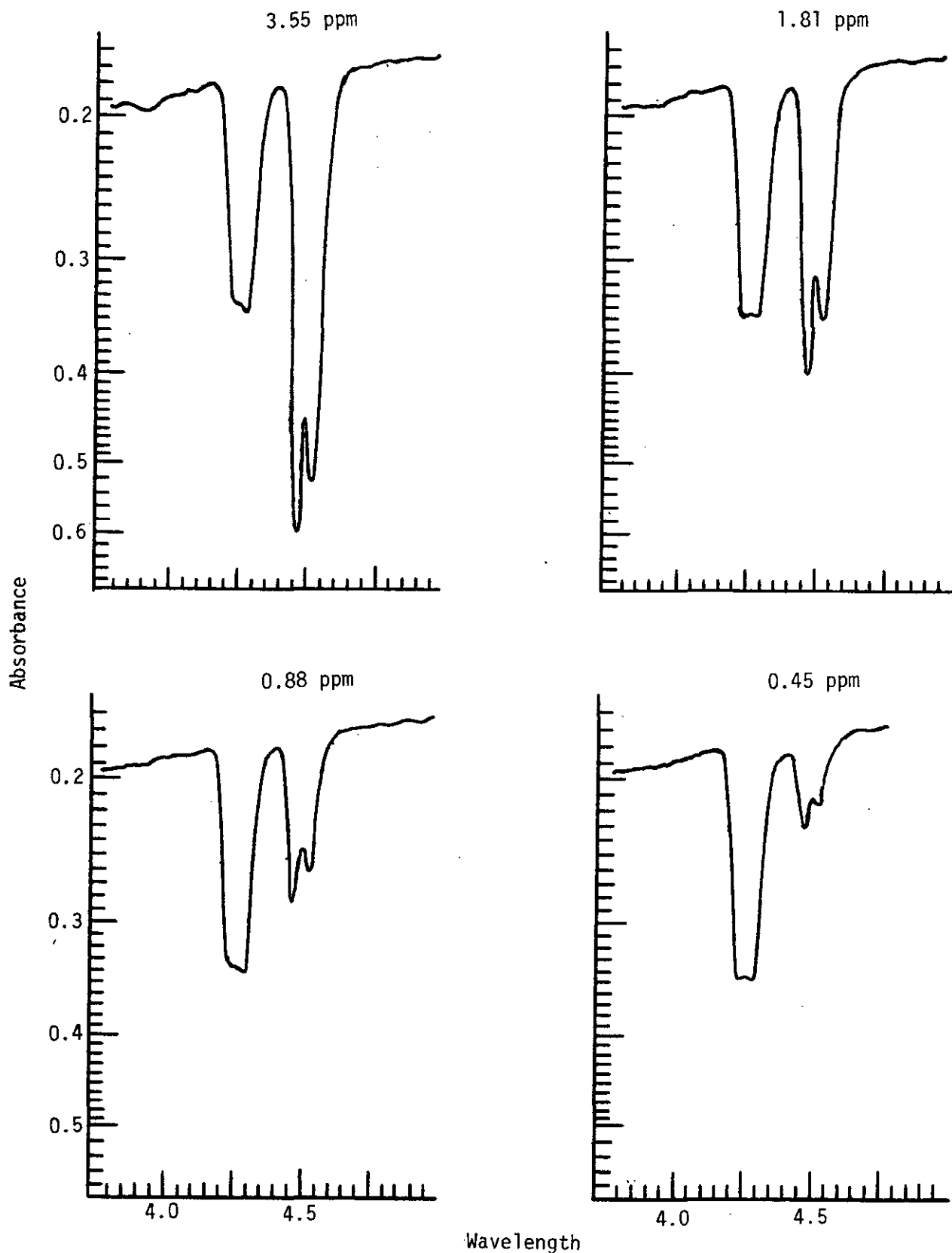
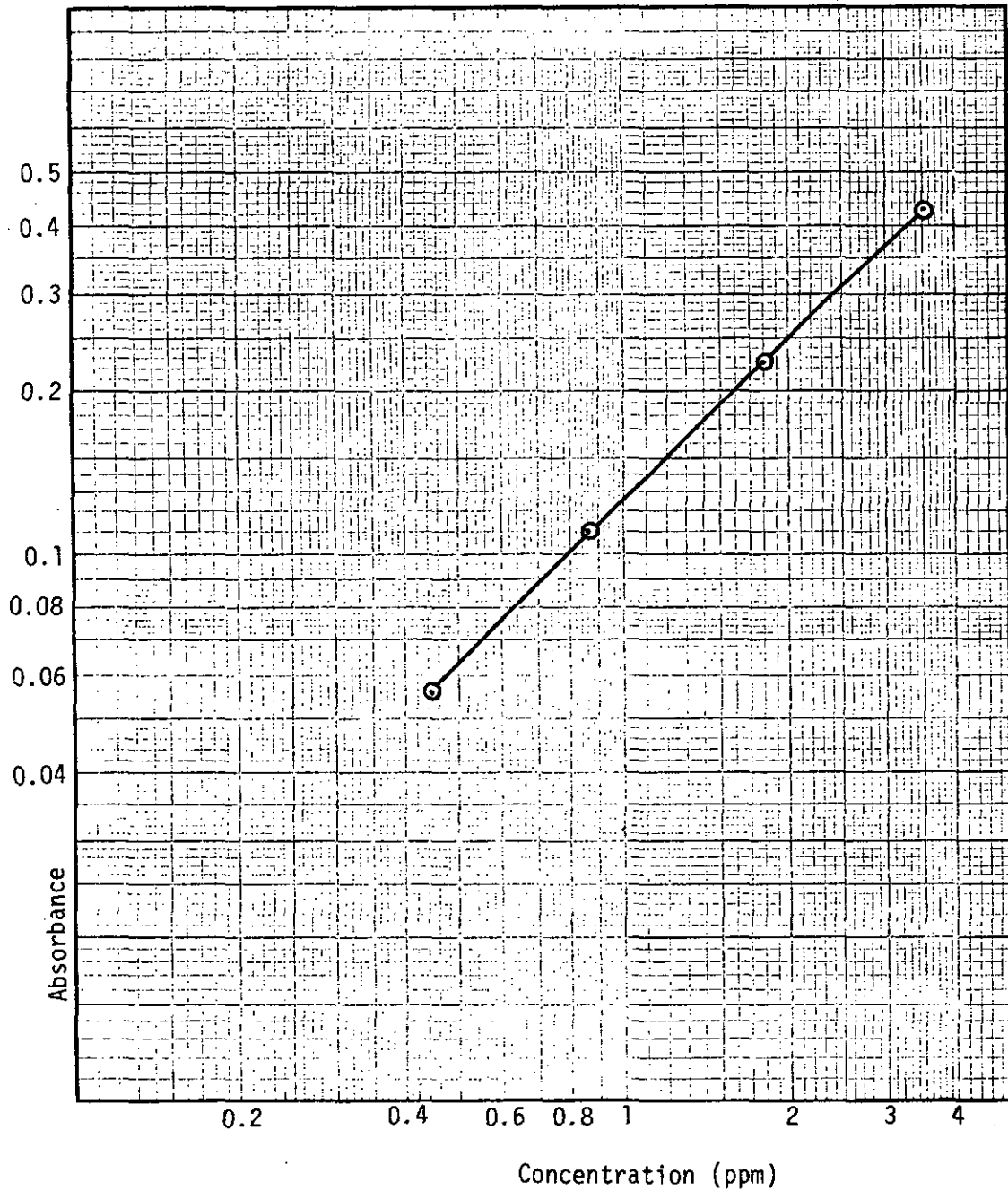


FIGURE 5. Absorbance of Nitrous Oxide.

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FIGURE 6. Calibration Curve for Nitrous Oxide.

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of pressurization, running IR scans, and venting with appropriate data recording was repeated until the final concentration was less than 0.5 ppm. Recorded data and calculations are presented in Table VI. IR scans at 135 psig are presented in Figure 5. 7/ The calibration curve at 135 psig is presented in Figure 6.

10.3 Contaminant Concentrations Prepared by the Partial Pressure Method. A calibration curve for methane was to be prepared. The range of concentration was determined to be 100 to 1 ppm from Table I. An approximate value of Z was chosen to be 125 ppm. Therefore

$$\frac{100\ 000}{125} = E \times 10^d$$

$$800 = E \times 10^d$$

$$E = 8$$

$$d = 2$$

The pressure of the second evacuation was calculated.

$$\frac{700}{E} \text{ mm Hg} = \frac{700}{8} \text{ mm Hg} = 85 \text{ mm Hg}$$

The number of additional low pressure dilutions required were also calculated.

$$d - 2 = 2 - 2 = 0$$

No additional low pressure dilutions were required. During the low pressure dilution, the following data was recorded. Barometric pressures have been corrected for temperature and altitude and are reported in inches of mercury (in Hg).

n	P _b	ΔP _b	P _{mc}	M	P _n
1	29.23	0	0.004	29.182	0.004
2	29.23	0	-	26.442	2.744
3	29.23	0	-	1.858	27.328
4	29.22	-0.01	-	26.208	2.968
5	29.22	-0.01	-	1.747	27.429

7/ The doublet peak of varying height is nitrous oxide. The other peak of constant height is an example of atmospheric carbon dioxide interference described in paragraph 4.3.

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The gas cell was evacuated to an approximate manometer reading of 26.2 in Hg and pressurized to 135 psig. The following data was recorded:

n	P _b	P _b	M	P _g	P _n (in Hg)	P _n (psi)
6	29.22	-0.1	26.053	-	3.123	1.534
7	29.21	-	-	135	-	149

The concentration of the contaminant gas standard was then calculated.

$$C_o = 10^6 \left(\frac{2.744 - 0.004}{27.328} \right) \left(\frac{2.968}{27.429} \right) \frac{1.534}{149}$$

$$C_o = 111.7 \text{ ppm}$$

IR scans and dilutions were made. Subsequent calculations are similar to those of paragraph 10.2 and will not be presented here.

10.4 Determination of the Concentration of Ethane and Higher Hydrocarbons.

An IR scan was run on an oxygen sample from the field. Portions of the spectrum are presented in Figure 7.

a. The absorbance due to methane at 7.68 μm was determined from Figure 7 and as follows:

$$A_1 = 0.087$$

$$A_2 = 0.462$$

$$A_{7.68} = 0.462 - 0.087 = 0.375$$

b. The methane concentration which exhibits an absorbance of 0.375 was read from Figure 8. This concentration was 16.0 ppm.

c. The absorbance due to methane at 3.40 μm was determined from Figure 9. The absorbance was 0.048.

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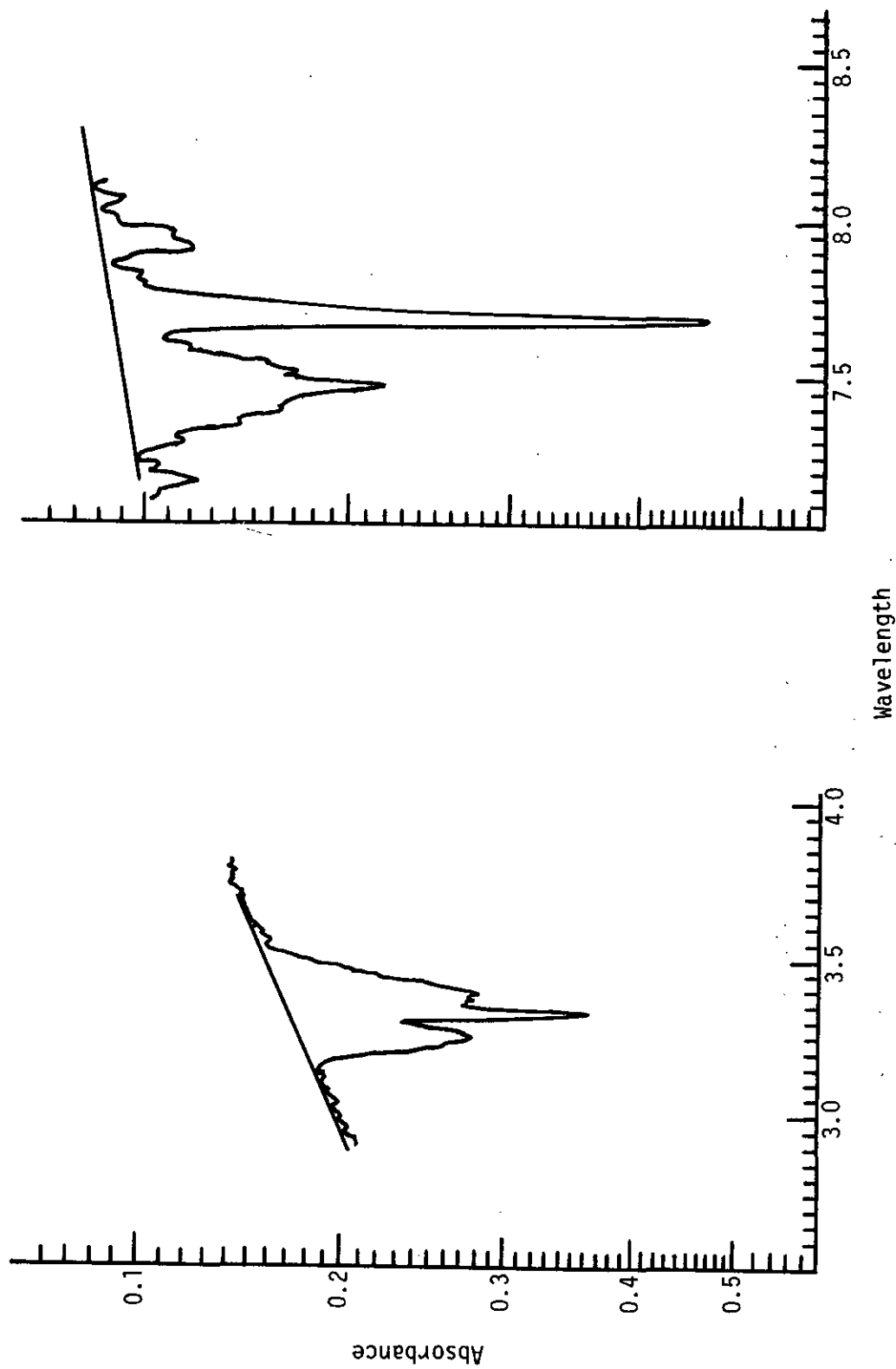


FIGURE 7. Absorbance of Hydrocarbons.

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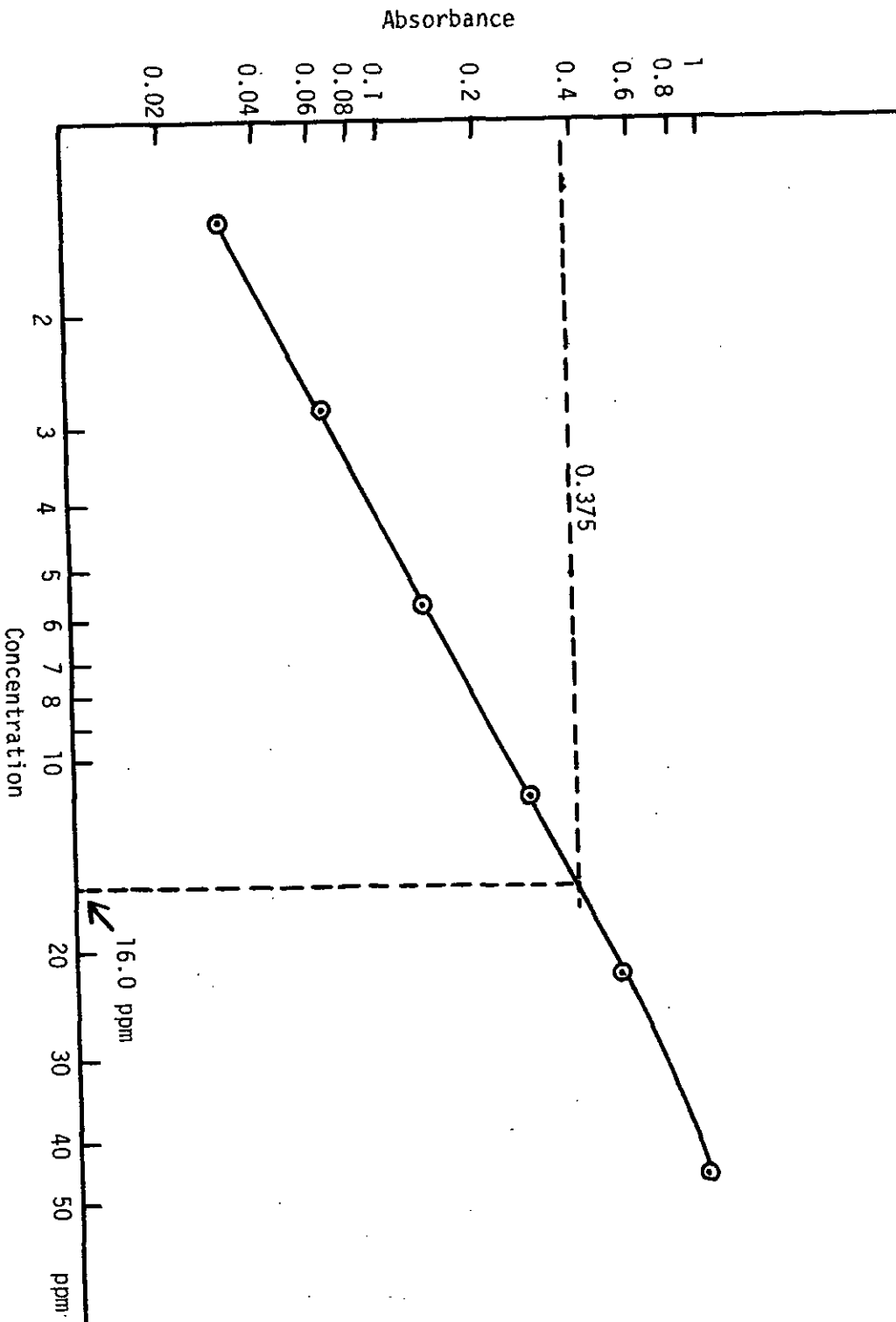


FIGURE 8. Calibration Curve, Methane at 7.68 μm.

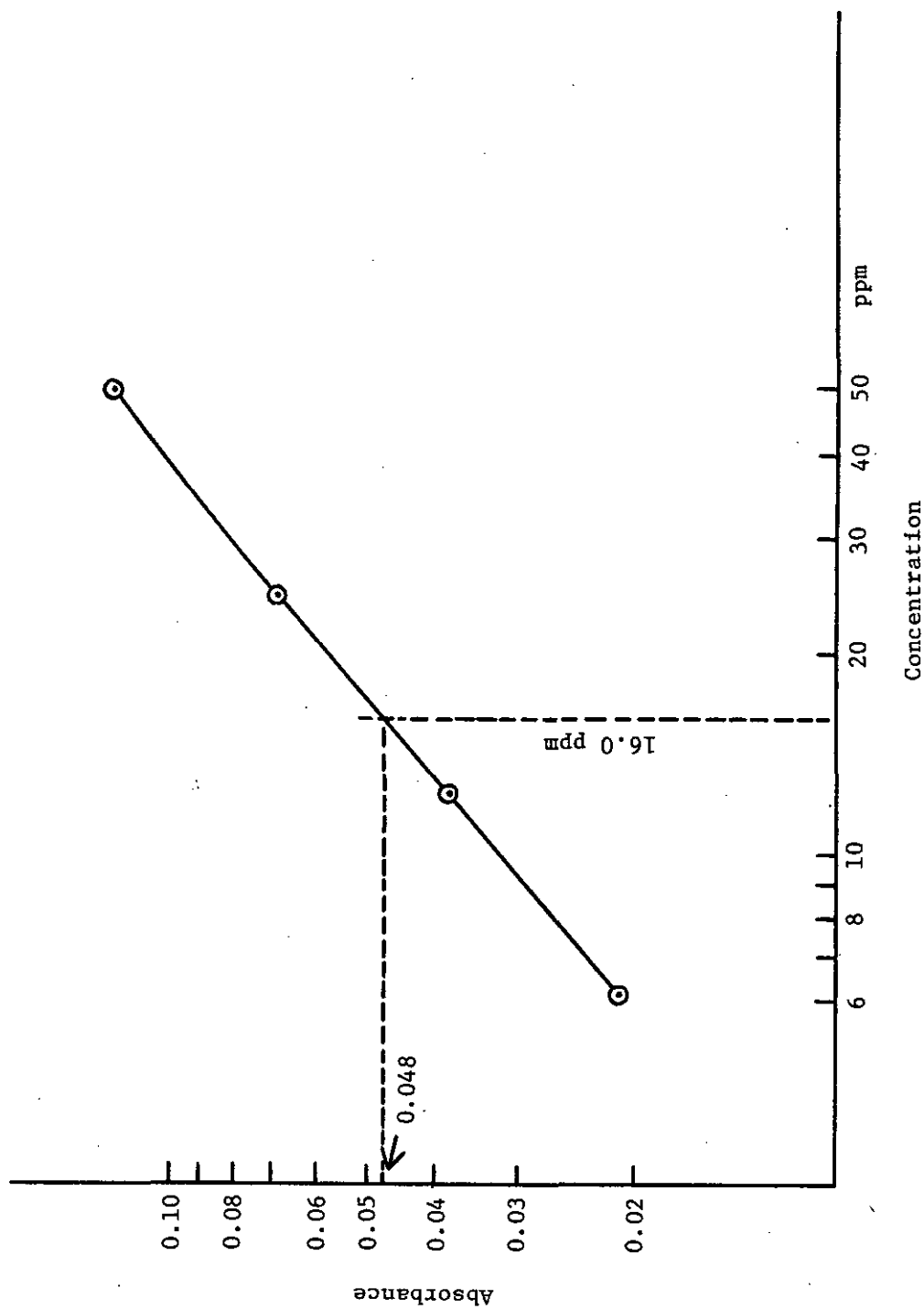


FIGURE 9. Calibration Curve, Methane at 3.40 μm .

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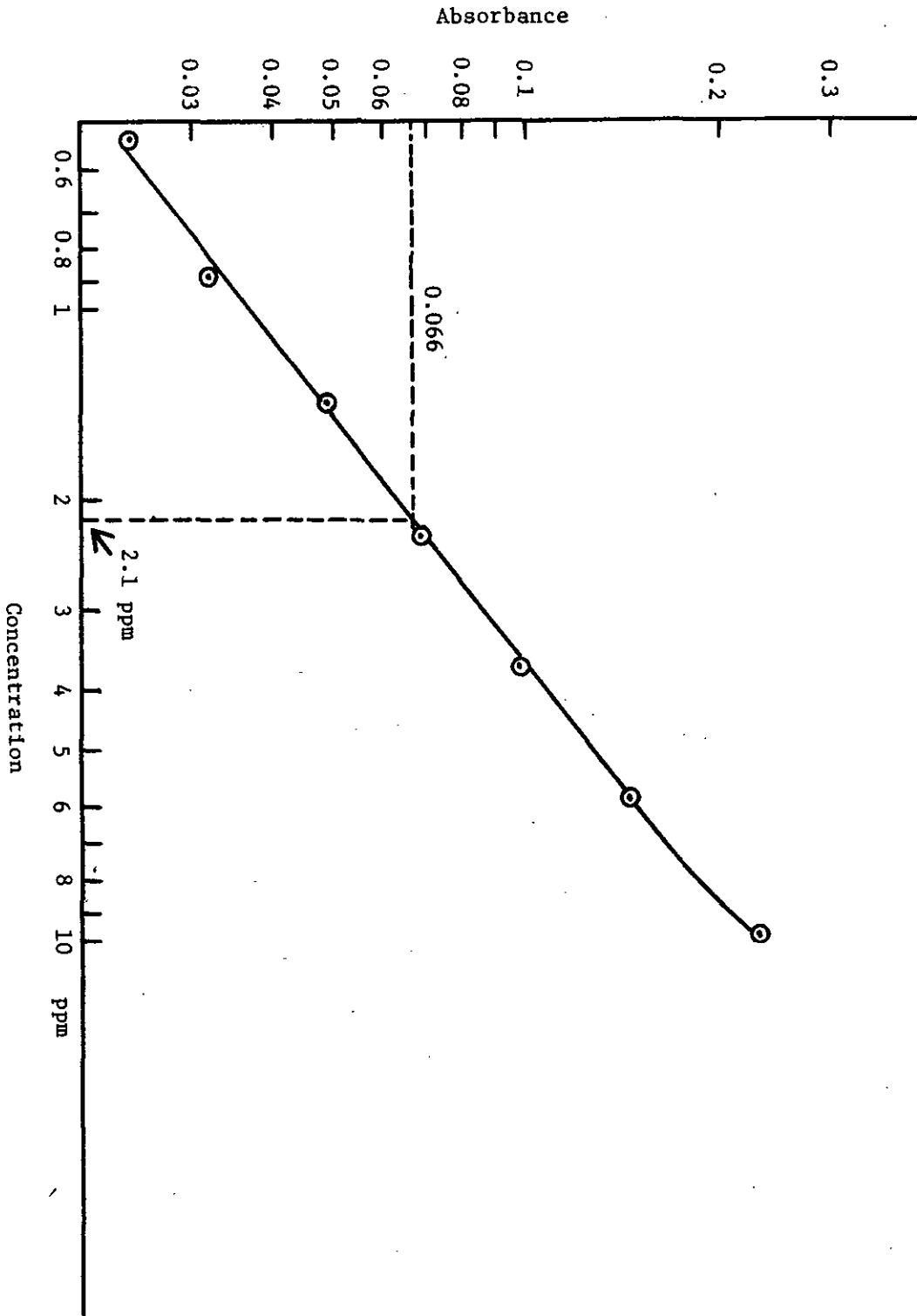


FIGURE 10. Calibration Curve, Ethane at 3.40 μ m.

d. The sample absorbance at 3.40 μm was determined from the spectrum (Figure 7) as follows:

$$A_1 = 0.167$$

$$A_2 = 0.281$$

$$A_{3.40} = 0.281 - 0.167 = 0.114$$

This absorbance was due to methane, ethane, and higher hydrocarbons. The desired absorbance, however, would be due to ethane and higher hydrocarbons alone.

e. To obtain the desired absorbance, the absorbance due to methane was subtracted from the sample absorbance at 3.40 μm .

$$A_{3.40}(\text{C}_{2+}) = A_{3.40}(\text{Sample}) - A_{3.40}(\text{Methane})$$

$$A_{3.40}(\text{C}_{2+}) = 0.114 - 0.048 = 0.066$$

f. Finally, the ethane and higher hydrocarbon concentration which exhibits an absorbance of 0.066 was read from the ethane calibration curve (Figure 10). This concentration was 2.1.

Appendix B

Verification of Formulas

20.1 Volume of Mixing Container System. For simplicity all pressures are considered to be in the same units of absolute pressure.

Let Δn = number of moles exhausted through wet test meter

n_1 = initial moles in mixing container

n_2 = final moles in mixing container

$\Delta n = n_1 - n_2$

P_b = barometric pressure

P_w = vapor pressure of water

$P_b - P_w$ = pressure of vented gas

P_1 = initial pressure of mixing container

P_2 = final pressure of mixing container

V_1 = volume of gas through wet test meter

Using the form of the general equation

$$P V = n R T$$

we may write $(P_b - P_w) V_1 = \Delta n R T$

$$P_1 V = n_1 R T$$

$$P_2 V = n_2 R T$$

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and

$$n_1 = \frac{P_1 V}{R T}$$

$$n_2 = \frac{P_2 V}{R T}$$

then

$$(P_b - P_w) V_1 = (n_1 - n_2) R T$$

$$(P_b - P_w) V_1 = \left(\frac{P_1 V}{R T} - \frac{P_2 V}{R T} \right) R T$$

$$(P_b - P_w) V_1 = (P_1 - P_2) V$$

$$V = \frac{(P_b - P_w) V_1}{P_1 - P_2}$$

20.2 Concentration by Volume Method (Initial). For simplicity, pressures are considered to be in the same units of absolute pressure, volumes are in the same unit and concentrations are in mole fractions.

Let n_s = number of moles in syringe or gas sampling valve loop

n_c = number of moles in mixing container system

T = temperature

P_b = barometric pressure

P_g = mixing container system pressure

V_s = volume of syringe or gas sampling valve loop

V_c = volume of mixing container system

$C_o = n_s/n_c$

Using the form of the general equation

$$n = \frac{P V}{R T}$$

we may write

$$n_s = \frac{P_b V_s}{R T}$$

$$n_c = \frac{(P_g + P_b) V_c}{R T}$$

therefore

$$C_o = \frac{n_s}{n_c} = \left(\frac{P_b V_s}{R T} \right) \left[\frac{R T}{(P_g + P_b) V_c} \right]$$

$$C_o = \left(\frac{V_s}{V_c} \right) \left(\frac{P_b}{P_g + P_b} \right)$$

20.3 Concentration by Volume Method (Following Dilution). For simplicity, all pressures are in the same units of absolute pressure and concentrations are in mole fractions.

Let n_i = Total moles in mixing container before dilution

n_f = Total moles in mixing container after dilution

Then $C_i n_i$ = Number of moles of contaminant in mixing container

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Using the form of the general equation

$$n = \frac{P V}{R T}$$

we may write

$$n_i = \frac{P_i V}{R T_i}$$

$$n_f = \frac{P_f V}{R T_f}$$

then

$$C_f = \frac{C_i n_i}{n_f} = C_i \left(\frac{P_i V}{R T_i} \right) \left(\frac{R T_f}{P_f V} \right)$$

$$C_f = C_i \left(\frac{P_i}{P_f} \right) \left(\frac{T_f}{T_i} \right)$$

20.4 Concentration by Partial Pressure Method. Concentration in this verification are in mole fractions.

- Let n_1 = Moles of residual gas after evacuation
 n_2 = Moles after addition of contaminant
 n_3 = Total moles after addition of dilution gas
 n_c = Moles of contaminant

then

$$n_c = n_2 - n_1$$

$$C_A = \frac{n_c}{n_3} = \frac{n_2 - n_1}{n_3}$$

Using the form of the general equation

$$n = \frac{P V}{R T}$$

we may write

$$C_A = \frac{\frac{P_2 V}{R T} - \frac{P_1 V}{R T}}{\frac{P_3 V}{R T}}$$

Assuming volume and temperature are constant

$$C_A = \frac{P_2 - P_1}{P_3}$$

For successive evacuation and pressurization it can be shown from paragraph 20.3

$$C_B = C_A \left(\frac{P_4}{P_5} \right)$$

$$C_B = \left(\frac{P_2 - P_1}{P_3} \right) \left(\frac{P_4}{P_5} \right)$$

and so on.

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Finally,

$$C = \left(\frac{P_2 - P_1}{P_3} \right) \left(\frac{P_4}{P_5} \right) \left(\frac{P_6}{P_7} \right) \dots$$

20.5 Absolute Pressure from Manometric Readings. Ideally, the absolute pressure (P) is defined for open-end manometers

$$P = P_b - M$$

At very low pressures inaccuracies in the barometric and manometric readings make this approach impractical. One can arrive at the barometric pressure through use of the manometer and a McLeod gauge.

$$P_b = M + P_{mc}$$

During the course of dilutions the barometric pressure may change. If the barometric pressure changes between the first and second pressure reading we would have

$$P_{b2} - P_{b1} = P_{b2} - (M_1 + P_{mc})$$

$$\Delta P_b = P_{b2} - M_1 - P_{mc}$$

$$P_{b2} = M_1 + P_{mc} + \Delta P_b$$

the absolute pressure at the second reading would then be

$$P_2 = P_{b2} - M_2$$

$$P_2 = M_1 + P_{mc} + \Delta P_b - M_2$$

It can be shown that this is true for all pressures and that generally

$$P_n = M_1 + P_{mc} + \Delta P_b - M_n$$

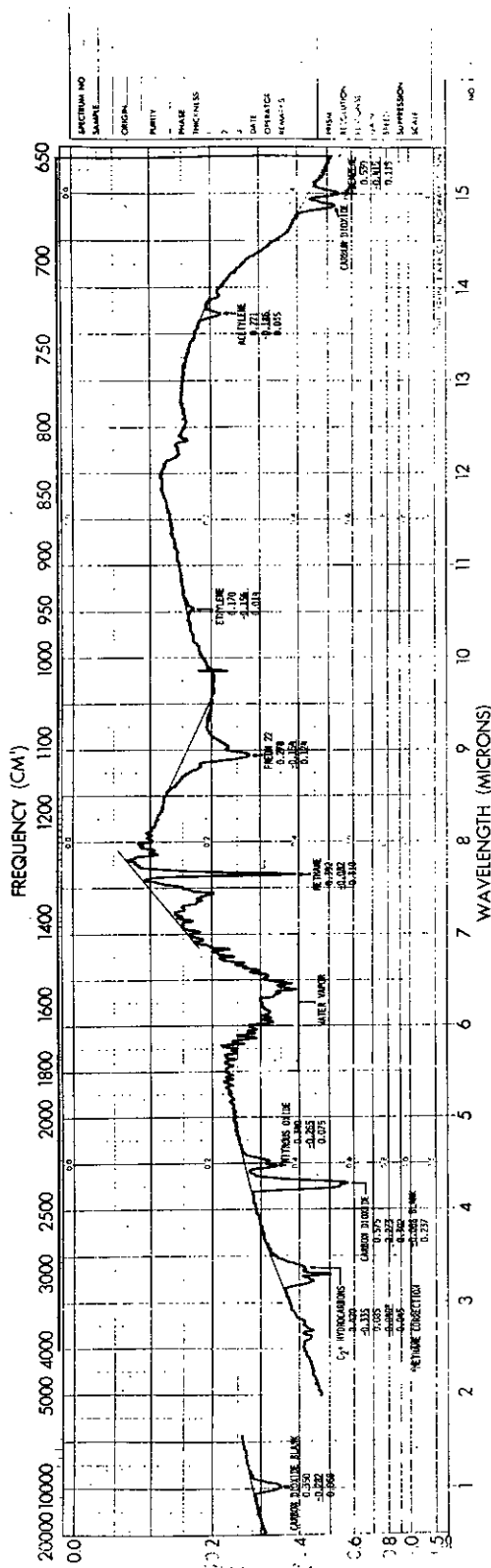


FIGURE 11. Typical Absorption Spectrum.

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