

NAVY EXPERIMENTAL DIVING UNIT

TECHNICAL REPORT NO. 1-00

FIELD-BASED PROCEDURES FOR SCREENING DIVER'S AIR

R.S. LILLO, Ph.D., W.R. PORTER, B.S., D.M. FOTHERGILL, Ph.D.,
J.M. CALDWELL, B.S., A. RUBY, B.S.

MARCH 2000

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INTRODUCTION

The U.S. Navy Diver's Air Sampling Program coordinates the mandatory semi-annual air purity testing of compressors used to supply diver's air in the Fleet¹. Gas sampling kits supplied by a contract laboratory are sent to the field where gas samples are taken and then returned to the laboratory for analysis. Analytical results are reported back to the field indicating pass or fail based on specifications for diving air in Volume 1 of the U.S. Navy Diving Manual¹. This approach is expensive, cumbersome, and potentially unreliable. Accuracy of results will depend partly on gas collection procedures in the field, which can be difficult to perform correctly even under the best conditions. Relying on a contract laboratory also introduces concerns including the insurance of accurate data and the long time delay between sampling and reporting of results.

Advancements in portable instruments for gas analysis during the past 15 years have made reliable field testing of diver's air a practical alternative. Accurate test procedures can be developed for on-site use based on current testing requirements and expected contaminant profiles of compressor air. Such testing would:

1. Eliminate current logistical requirements associated with ensuring sampling kit delivery,
2. Eliminate potential sampling problems associated with collection of gas for later analysis,
3. Provide credible data immediately to the person in the field,
4. Allow immediate re-testing in case of questions and for troubleshooting problems.

In addition, field-testing may cost substantially less than the current program, particularly if the cost of the portable analyzers is spread across their expected lifetimes.

This report describes the set of procedures that the Naval Medical Research Institute (NMRI), now the Naval Medical Research Center (NMRC), developed for screening diver's air on-site using three portable analyzers. These procedures could replace or supplement present methods that encompass the U.S. Navy Diver's Air Sampling Program. However, these procedures will need to be field tested under actual sampling conditions to determine what modifications should be made prior to possible transition to the Fleet.

RATIONALE OF SCREENING PROCEDURES

SCREENING LIMITS

Air purity standards defined in the U.S. Navy Diving Manual¹ were used as the screening limits for diver's air:

O ₂ :	20-22%
CO ₂ :	1,000 ppm (max)
CO:	20 ppm (max)
Total hydrocarbons: (expressed in CH ₄ equivalents, but excluding CH ₄)	25 ppm (max)
Oil, mist, particulates:	5 mg/m ³ (max)
Odor:	not objectionable

ppm: parts per million
mg/m³: milligrams per cubic meter

SCREENING INSTRUMENTS AND HARDWARE

Procedures were developed that allowed screening of compressor air on-site using three analyzers: 1) O₂/CO₂/CO analyzer, 2) total hydrocarbon analyzer, and 3) aerosol monitor, and a sampling apparatus. A brief description of each analyzer and sampling apparatus follows:

1. O₂/CO₂/CO Analyzer. A portable analyzer (Anagas Dive Air, model DV 1.1, Geotechnical Instruments, Inc., Leamington Spa, England) was developed in collaboration with the manufacturer and allows simultaneous measurement of O₂, CO₂, and CO. This unit was adapted from the Geotechnical CO₂ analyzers that NMRI has been testing the past several years for submarine use²⁻³. A small internal pump draws a gas sample into the unit where CO₂ is measured with a non-dispersive infrared detector. Oxygen and CO are measured with separate electrochemical detectors. Measurements are based on factory-generated calibration curves stored in memory of the analyzer. On-site calibration involves zeroing of the O₂ and CO detectors with 100% N₂ followed by a one-point span of all three detectors with a calibration gas containing ~21% O₂, ~1,000 ppm CO₂, and ~20 ppm CO, balance N₂. These steps adjust the position of the calibration curve to improve accuracy of measurements. Calibration concentrations were chosen to approximate the screening limits in order to minimize measurement errors about these concentrations. Based on laboratory testing described in **Appendix A**, accuracy of the analyzer at the limits is estimated to be:

O ₂ :	21.0% ± 0.2%
CO ₂ :	1,000 ± 100 ppm
CO:	20 ± 2 ppm

2. Total Hydrocarbon Analyzer. A portable gas analyzer (Toxic Vapor Analyzer – TVA-1000B, Foxboro Co., East Bridgewater, MA), that has been incorporated in the new NMRI Revised Air Purity Guidelines for Dry Deck Shelter (DDS) Operations⁴, was adopted for use with diver's air. This analyzer can be configured as it is for the DDS Guidelines, with dual detectors, a flame ionization detector (FID) that detects most volatile organic compounds (i.e., those containing carbon), and a photoionization detector (PID) which provides additional diagnostic information. However, for screening diver's air, the TVA is fitted with only an FID to provide the required "total hydrocarbon" reading.

The FID uses the principle of hydrogen flame ionization for detection and measurement of volatile organic compounds down to the ppm level. Electrically-charged species are formed when organic compounds are introduced into a small hydrogen-in-air flame and are detected by a collecting electrode. Common gases (e.g., CO₂, CO) give no response. The air that is being sampled provides the O₂ for the flame combustion.

The total hydrocarbon limit of 25 ppm is in terms of methane. However, the TVA is calibrated in these procedures with a ~10 ppm isobutylene/balance air, rather than a methane standard. The isobutylene standard is non-toxic and produces an intermediate FID response compared to expected contaminants such as alkanes and aromatic hydrocarbons. Isobutylene is also currently used for Trace Gas Analyzer (TGA) calibration on submarines and will be used for TVA calibration during DDS operations⁴. Thus, using ~10 ppm isobutylene for diver's air testing, keeps the calibration gas requirement uniform for the three procedures and reduces the number of calibration gases needed in the field.

During calibration, the TVA is first zeroed using a charcoal filter adaptor to remove the isobutylene from the calibration gas, effectively delivering hydrocarbon-free air to the instrument. The filter is then removed, the normal TVA probe installed, and the calibration gas is sampled for span adjustment. The TVA is then used to screen diver's air with and without the charcoal filter. Because the charcoal removes trace organic vapors heavier than methane, ethane, and some related compounds, filtering allows determination of the methane concentration. The methane value is then subtracted from the unfiltered reading to calculate a total hydrocarbon concentration, which excludes methane. Because the FID responds to different compounds with differing sensitivities, it is necessary to adjust the analyzer reading in isobutylene equivalents to the desired methane equivalents. To do this, the TVA reading is multiplied by 1.5, which is the manufacturer's reported value of isobutylene/methane sensitivity, which has also been confirmed by NMRI. However, it is emphasized here that different types of analyzers will respond differently to specific contaminants. Consequently, total hydrocarbon measurements of diver's air will depend not only on the nature of the contaminants but also on the specific analyzer used. Thus, for this report, testing of the TVA focused on measurement linearity across a range of

concentrations of specific contaminants (**Appendix B**) rather than on the accuracy in determining total hydrocarbons.

3. Aerosol Monitor. A portable off-the-shelf aerosol monitor (Personal/DataRAM, model pDR-1000, MIE, Inc., Bedford, MA) is used to measure the aerosol concentration (oil, mist, and particulates) of diver's air. This analyzer is a photometer that uses the principle of light scattering to measure the concentration of an aerosol. The response of the monitor has been calibrated at the factory with a fine test dust. However, the angle and intensity of the scattered light depend on the refractive index and size of the particle. Therefore, it is important that the monitor reading be corrected for the oil used in the compressor being tested. Details of the theory and principle of operation of these types of aerosol monitors are given in Reference (5).

The Personal/DataRAM was evaluated in the laboratory to determine calibration factors for different oils commonly used in air compressors (described in **Appendix C**). The calibration factors ranged from 0.4 to 0.6 for six oils including several synthetic lubricants. Based on these results, a calibration factor of 0.5 was incorporated into the operating procedures of the Personal/DataRAM for screening diver's air. Experience will dictate if this correction factor needs to be adjusted for oils that have not yet been tested.

The Personal/DataRAM is generally designed for inside use at locations away from strong breezes or winds. Air passively (i.e., without a pump) moves by convection, diffusion, and adventitious motion into the optical sensing chamber of the device via the slots under the top cover of the monitor. For sampling outside and under high flow conditions (e.g., ducts and stacks), active sampling instruments are required that are configured with special inlets designed to promote representative sampling. For compressor air screening, the top cover of the Personal/DataRAM is replaced with a plastic "Flow-Adaptor," designed by NMRI, that can be fabricated in-house or commercially, and is described in **Appendix D**. The Flow-Adaptor allows a very low flow of the compressor air to be directed through the sensing chamber of the DataRAM using a small vane flowmeter to monitor the flow.

Overall accuracy associated with Personal/DataRAM measurements of compressor air is conservatively estimated, as discussed in **Appendix C**, at $\pm 40\%$ relative.

4. Sampling apparatus. A sampling apparatus, defined here as the "Air-Sampler" and described in **Appendix D**, was designed to allow sampling from the high pressure compressor output with the preceding three instruments. The primary concern with any sampling device is its potential effect on the constituents of the gas that will be measured. Here, the concern would be with 1) volatile hydrocarbons due to loss and gain of contaminants and 2) aerosols

which can be lost through dropout of particles as they impact on the inside surfaces of the hardware. The fixed gases (O₂, CO₂, and CO) are much less likely to be affected by the sampling process.

The Air-Sampler, with its all metal, stainless steel construction should be acceptable for most hydrocarbon sampling if the apparatus is kept clean and free of oil, grease, and solvents. The device, with its wide bore tubing, large valves, and straight-through flow path was designed to avoid problems with aerosol loss and the resulting underestimation of the actual aerosol concentration. Reliability of the Air-Sampler for oil mist sampling was confirmed by testing results in **Appendix C.**

SCREENING ACCURACY

The acceptable level of measurement accuracy for screening diver's air will depend on the air purity standards and the health and operational consequences of making either of two types of errors: 1) false positive responses (i.e., falsely concluding that the air fails to meet the standards) or 2) false negative responses (i.e., falsely concluding that the air meets the standards). False positive outcomes are more of an operational, rather than a health concern, in terms of readiness and the ability to perform a mission in a timely manner. False negative readings are a diver health issue, which depends on the rationale of the standards and the implications of exceeding them. Unfortunately, the justification is unclear for many of the current limits for gas contaminants contained in various U.S. Navy documents including those in the U.S. Diving Manual¹. For hyperbaric exposures, there is the additional problem of there being little information on contaminant effects on humans (or animals) at pressure.

Reassuringly, past experience with the Diver's Air Sampling Program has not revealed any major problems in terms of adverse acute symptoms with use of air from compressors that had recently passed the sampling standards. However, because the actual sampling is routinely done only once semiannually, past "success" of the program may have little meaning. Furthermore, because high CO₂ has been the main reason for air to fail U.S. Navy testing (J. Ruth, personal communication, NAVSEA 00C), there may be minimal experience with use of diving air that approaches or exceeds the limits for the other contaminants. Thus, lack of past problems should not be used as an endorsement of either the safety or suitability of the limits in terms of avoiding at least acute symptoms while diving. Certainly, the combined limit for total hydrocarbons remains an issue, in view of the relatively high toxicity attributed to some of the individual species, such as aromatics, aldehydes, and ketones, that might be expected to occur as a result of compressor malfunction or failure.

The O₂/CO₂/CO monitor, with its high accuracy, would not be expected to increase the measurement error of these three constituents. The TVA analysis, like the current hydrocarbon screening method using gas chromatography in the laboratory, will depend on the response factors of the instrument for specific species and the nature of the organic contaminants. However, there is no reason to presume that the TVA reading

would be less reliable than the laboratory value. In fact, direct analysis of hydrocarbons in the field by the TVA would be expected to produce a more accurate index of hydrocarbon contamination as no gas collection and storage is involved which could potentially compromise the sample. The $\pm 40\%$ error estimated for DataRAM measurement of aerosols is probably at least as accurate as the current system which requires direct collection of aerosols on filter paper in the field, subsequent weighing of the filter in the laboratory, and calculation based on estimated gas sample flow rate and time.

Testing here was done at laboratory temperatures generally ranging between 19 and 24 °C. Rather than evaluate temperature extremes, we assumed minimal error introduced by temperature changes when analyzers are 1) operated within the recommended temperature ranges and 2) calibrated and used at similar temperatures. Instrument operating temperatures given by the manufacturers are 1) 0 to 40 °C for the O₂/CO₂/CO analyzer, 2) 0 to 40 °C for the TVA, and 3) -10 to 50 °C for the DataRAM. These screening procedures should not be used at the extreme limits of these temperatures without further testing.

Although the pass/fail decision is based on meeting the air purity standards, close attention should be paid to any measurement that is abnormal but may still meet the limit. These suspect readings may alert the user to conditions such as a failing compressor or contaminated intake air that may soon deteriorate resulting in unacceptable diver's air. Thus, caution should be used to avoid merely declaring that the compressor unconditionally passes or fails. Certainly, the ability to easily re-test the air using these field-based procedures allows for the kind of follow-up that may be required in these types of situations.

SCREENING PROCEDURES

1. Air is sampled at locations given in Volume 1 of Reference (1) using the Air-Sampler apparatus and the three portable analyzers: a) Geotechnical O₂/CO₂/CO analyzer, b) Foxboro Toxic Vapor Analyzer (TVA), and c) MIE Personal/DataRAM aerosol monitor.
2. Necessary components and instruments required for sampling are specified in **Appendix D**. Record all data and sampling information on a data sheet comparable to that given in **Appendix E**.

BATTERY CHECKS AND H₂ CHARGING

Done pre-deployment if air screening is off-shore.

1. Ensure that the batteries of the two O₂/CO₂/CO analyzers and the two TVAs are fully charged according to **Appendix F**. Recharge if necessary.
2. Check the non-rechargeable batteries of the two DataRAMs according to **Appendix G** and replace if necessary.
3. Refill two to three TVA H₂ cylinders following procedures in the manufacturer's TVA manual.
4. For off-shore screening: at least one day prior to air screening, check the batteries again of all three analyzers.

ON-SITE AIR SCREENING

1. Complete the top portion of the first page of the data sheet. (**Appendix E**.)
2. Prepare the three analyzers for use, according to **Appendices F-G**. Ideally, analyzers should be calibrated at the site where screening is done. Extreme changes in ambient temperature should be avoided: do not calibrate at one temperature (e.g., a warm room) and then screen compressors at a very different temperature (e.g., outside in the cold). Be sure to allow analyzers to equilibrate to the ambient temperature before calibrating, especially after storage in the cold or hot. For example, do not bring analyzers inside after being stored in the trunk of a car in the winter without first waiting (several hours) for the instruments to return to room temperature.
3. Prior to sampling, each compressor should be operated for at least 10 minutes to warm up. Following this, blow out the compressor line through the sample site at a highly audible rate to remove any water and to equilibrate the line with the gas. After two minutes, ensure that no water (or any liquid) is being blown out by holding

a cloth or tissue in the gas stream and checking for wetness with your hand. If a wet spot is noted, continue to vent gas until dry to touch. Close the valve at the sample site to shut off gas flow.

4. After purging the line, connect the Air-Sampler (pressure rated to at least 5,000 psi, as described in **Appendix D**) to the sample site using the fittings provided. Prior to connection, ensure that all three valves on the Air-Sampler are fully closed.
5. Attach a new plastic stopcock and new 5 ml syringe barrel (or equivalent) to the Air-Sampler (**Fig. 1a**).
6. Slowly open the valve(s) on the compressor line to deliver pressure to the Air-Sampler. Once this valve is fully open, slowly open fully the upstream Air-Sampler valve. After line pressure has equalized, slowly open (partially) the downstream side Air-Sampler valve so gas is directed out through the open port away from the analyzers at a clearly audible, but not excessively loud, level.
7. Allow gas to purge the Air-Sampler for two minutes to equilibrate with the gas and then close the side valve. Then open the downstream valve to produce a clearly audible, but not excessively loud, level to allow sampling with analyzers.
8. Sample the gas with the O₂/CO₂/CO analyzer.
 - a. Turn on the O₂/CO₂/CO analyzer (if not already on) by pressing the red key. Then press "0", "2" (read gas levels), "2" (no), "5" (Pump On); allow to warm up five minutes before using. All three gas concentrations are presented simultaneously on the display.
 - b. Remove the sampling tubing from the O₂/CO₂/CO analyzer and attach the tubing (only) to the free port of the plastic stopcock on the Air-Sampler (**Figs. 1a-b**). Adjust the downstream Air-Sampler valve and the plastic stopcock so that the gas flow is just audible as it exits the tubing so that the analyzer will not be overpressurized when connected.
 - c. Attach the analyzer to one branch of the tubing as done during calibration, check gas flow again, and wait at least one minute until readings are stable.
 - d. Record measurements on data sheet and determine if values pass or fail the limits.
 - e. Unless additional compressor sampling will be done right now, turn the analyzer off to conserve batteries by pressing the red key. Remove its sampling tubing from the Air-Sampler.
9. Sample the gas with the TVA.

- a. Adjust the downstream Air-Sampler valve to direct a clearly audible gas flow to the syringe barrel using the plastic stopcock to fine tune the flow (**Fig. 1a**). Wait one minute.
- b. The TVA should be operating and ready to use if step #2 was done.
- c. Position the normal sampler (no charcoal) halfway into syringe barrel and wait at least one minute until the reading stabilizes.
- d. The FID value is read off the display on either the instrument or the sampling probe.
- e. Record measurement on data sheet.
- f. Remove the normal sampler and install the charcoal filter.
- g. Again wait at least one minute until the reading stabilizes and record.
- h. Calculate the total hydrocarbon concentration (excluding methane) using the data sheet and determine if value passes or fails the limit.
- i. Leave the TVA turned on.

10. Sample the gas with the DataRAM.

- a. The DataRAM should be operating and ready to use if step #2 was done.
- b. Turn the plastic stopcock to shut off all flow to the free port where the DataRAM will be attached (see **Figs. 1a-b**).
- c. Connect the DataRAM with its flow monitor to the stopcock using its attached tubing.
- d. Carefully adjust the plastic stopcock so the flow monitor on the outlet port of the DataRAM just begins to rotate. Avoid higher flows that could overpressurize the head of the DataRAM.
- e. The DataRAM is now measuring the aerosol concentration in the gas flowing over its sensing chamber. The CONC value is the instantaneous concentration. The TWA value is the Time Weighted Average concentration.
- f. Monitor the changes in the CONC reading for two minutes and record the maximum value observed under flow conditions on the data sheet.

- g. Multiply the value on the data sheet by the correction factor (0.5) to convert the DataRAM reading to oil mist concentration. Determine if this concentration passes or fails the limit.
 - h. If the calculated oil mist concentration exceeds the limit of 5 mg/m^3 , the Air-Sampler should be cleaned to U.S. Navy O_2 -use specifications before air is sampled again. The Tygon tubing on the DataRAM and three-way stopcock and syringe barrel on the Air-Sampler should be replaced too.
 - i. Leave the DataRAM turned on and remove its sampling tubing from the Air-Sampler.
11. Close the valve(s) on the compressor line. Bleed down and remove the Air-Sampler.
 12. Record the presence of any objectionable odor in the sample gas on the data sheet.
 13. If the air did not meet the purity requirements for any of constituents tested (including odor), blow out the compressor line through the sample site (without the Air-Sampler attached) at a highly audible rate for another five minutes and re-test for all components again according to steps #4-12.
 14. If the air still fails the limit requirements, refer to the next section, "Interpretation of Screening Results."
 15. Abnormally high, but acceptable, measurements may suggest a developing problem that can be monitored by repeated screening and referring again to the section, "Interpretation of Screening Results."
 16. If another compressor will be sampled, go to that site and repeat steps #3-13.
 17. When finished for the day, close the red H_2 supply valve on the TVA and press the "OFF" key. Press the "ON/OFF" button on the DataRAM and then "ENTER" to turn off. The $\text{O}_2/\text{CO}_2/\text{CO}$ analyzer may already have been turned off.
 18. Return all three analyzers to their storage locations, ensure again that the analyzers are turned off, and, if possible, plug the TVA and $\text{O}_2/\text{CO}_2/\text{O}_2$ analyzer into their battery chargers.

INTERPRETATION OF SCREENING RESULTS

1. High CO₂, CO, or TVA (Hydrocarbon) readings

or

Low O₂ reading

or

Objectionable Odor

- a. Compressor intake air may be contaminated due to engine exhaust or shipboard activities such as painting, cleaning, and equipment repair.
- b. Compressor intake air may be contaminated due to failure to ventilate closed spaces (e.g., submarine), where air intake is located, prior to compressor operation.
- c. Compressor malfunction or failure,
- d. Filters on compressor need servicing.

2. High DataRAM (oil, mist, particulates)

- a. Compressor malfunction or failure,
- b. Filters on compressor need servicing.

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FIGURE LEGENDS

Figure 1.

1a. Air-Sampler. Used to sample air from the compressor line with the O₂/CO₂/CO analyzer, Toxic Vapor Analyzer (TVA), and DataRAM aerosol monitor.

1b. Attachment to Air-Sampler. Shows how the DataRAM and O₂/CO₂/CO analyzer connect to the Air-Sampler.

Figure 2.

Test system for O₂/CO₂/CO analyzer. Delivers test gases to multiple analyzers in the laboratory at ambient pressure via the STEC gas divider.

Figure 3.

O₂/CO₂/CO analyzer accuracy. Measurement error for two analyzers vs. test gas concentration for four tests done in the laboratory over a period of several months. Analyzers calibrated with 21.0% O₂, 1,000 ppm CO₂, and 20 ppm CO prior to testing.

Figure 4.

CO₂/CO interference accuracy. Interference response curves generated using the STEC gas divider connected simultaneously to both the CO₂ standard and CO standard (one gas as the STEC calibration gas and the other gas as the diluent gas). In this manner, mixtures of CO₂ and CO were produced for testing in the laboratory. Top graphs in all four figures (a-d) show the measurement error vs. test gas concentration for four tests done over a period of several months. Bottom graphs show the difference between a normal test (diluent gas is zero nitrogen) done the prior day and the interference test. Analyzers calibrated with 21.0% O₂, 1,000 ppm CO₂, and 20 ppm CO prior to testing.

Figure 5.

TVA accuracy. Measurement error vs. toluene gas concentration during one representative test in the laboratory. Analyzer first calibrated with 10 ppm toluene, balance air, which was then diluted using the STEC gas divider and analyzed with the TVA.

Figure 6.

DataRAM setup with Flow-Adaptor. Used to sample air from the compressor line.

Figure 7.

Calibration setup: O₂/CO₂/CO analyzer and TVA. Allows calibration of the O₂/CO₂/CO analyzer and the Toxic Vapor Analyzer (TVA).

Figure 1a. Air-Sampler.

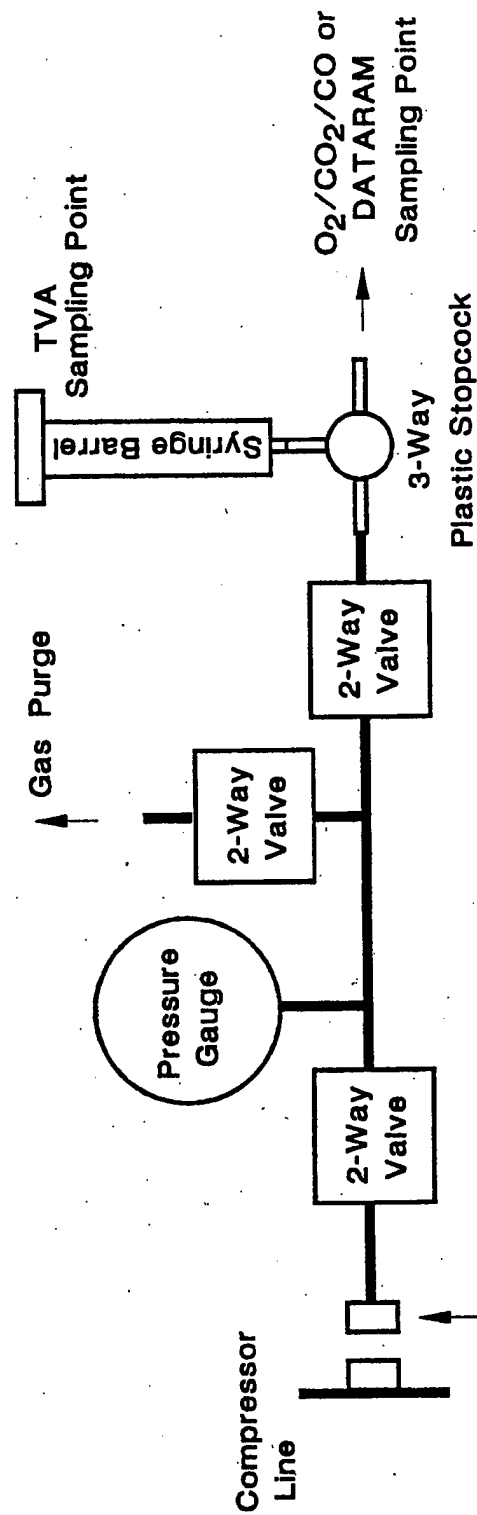


Figure 1b. Attachment to Air-Sampler.

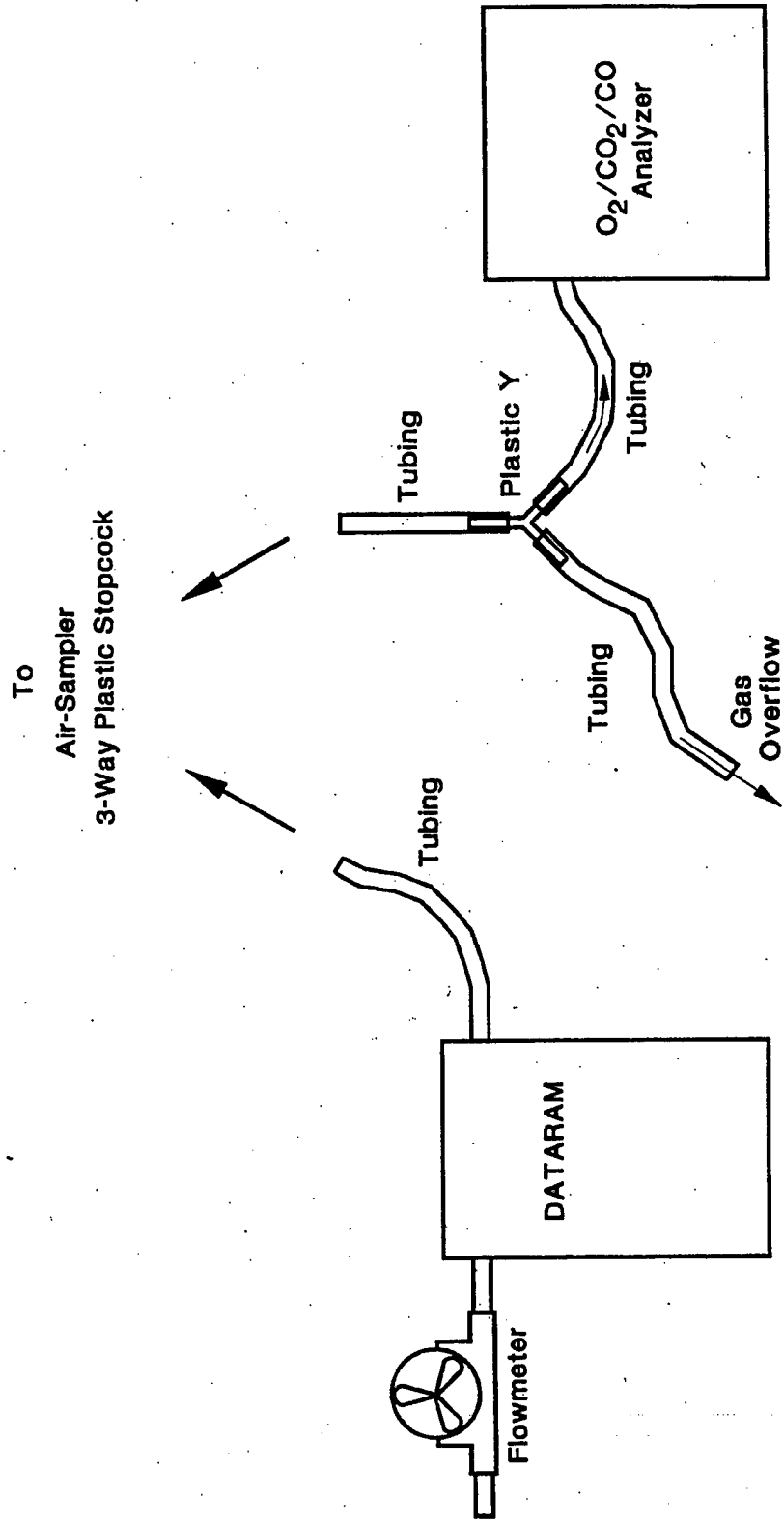


Figure 2. Test system for $O_2/CO_2/CO$ analyzer.

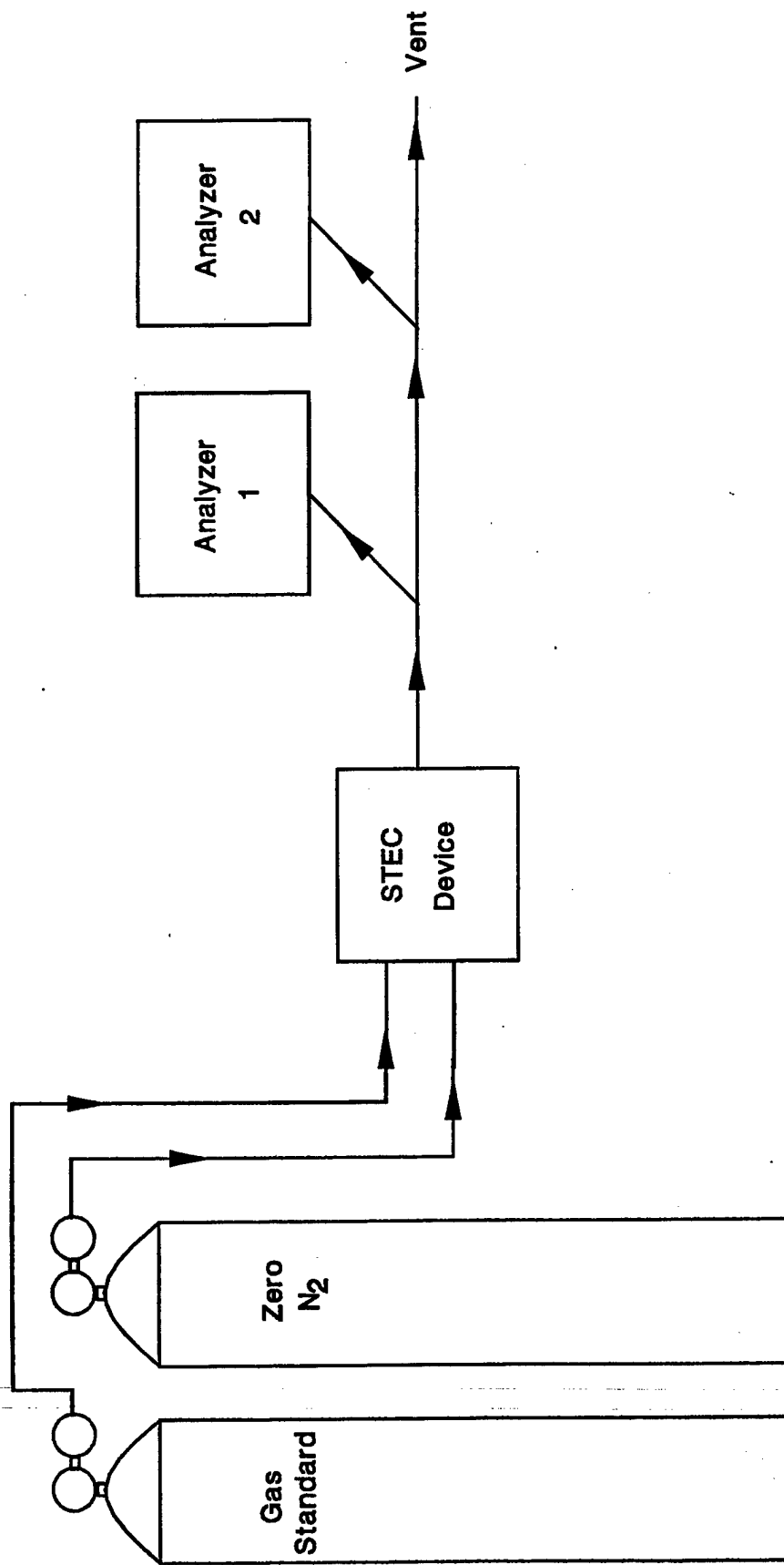


Figure 3a. O₂ accuracy.

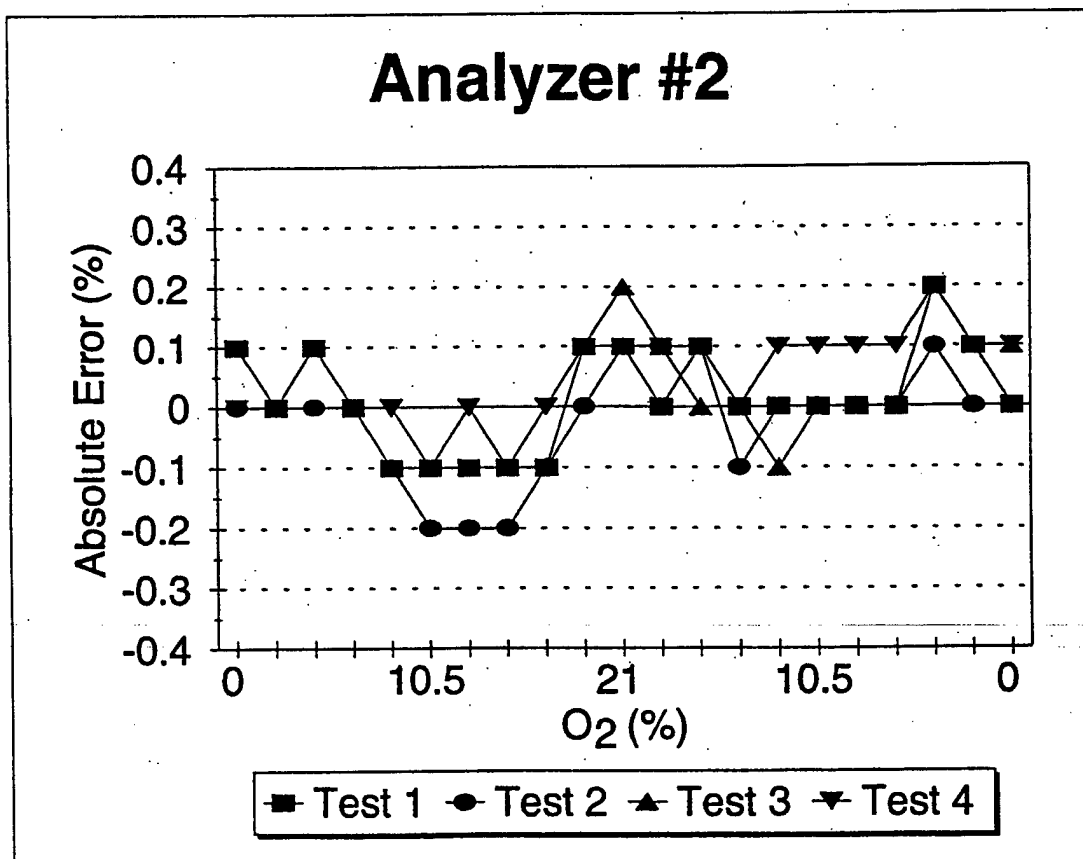
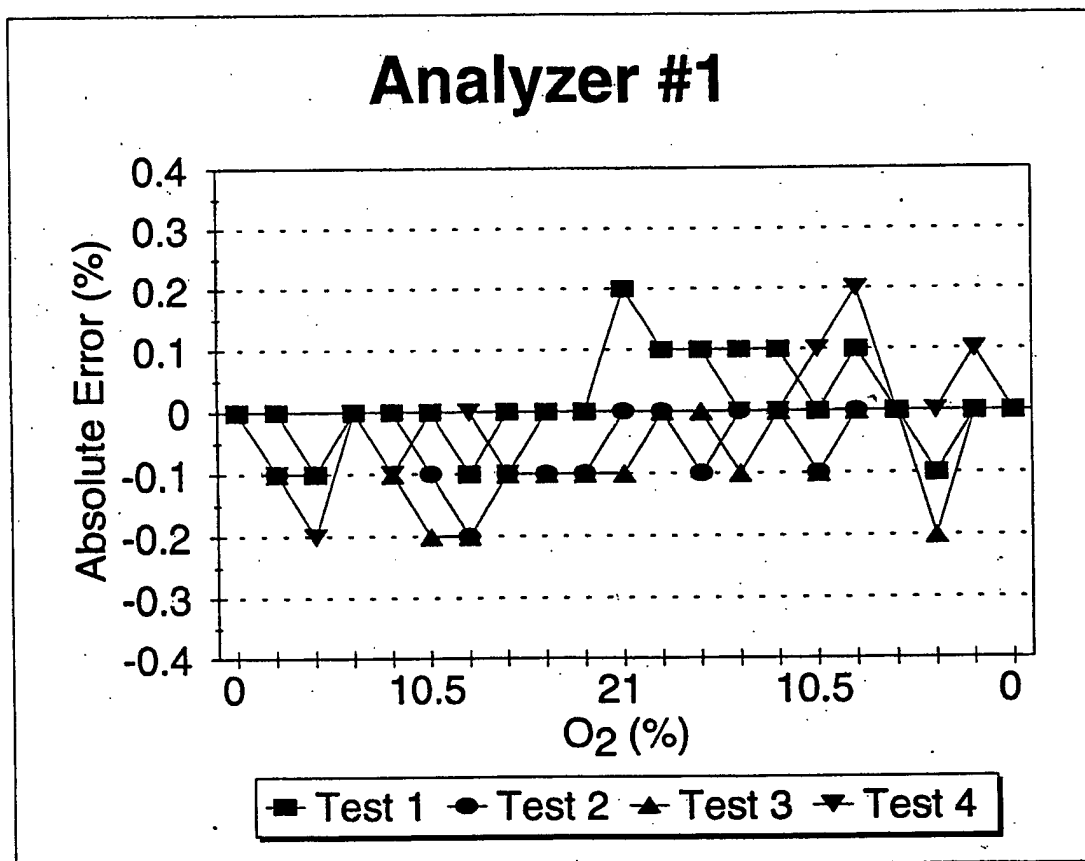


Figure 3b. CO₂ accuracy.

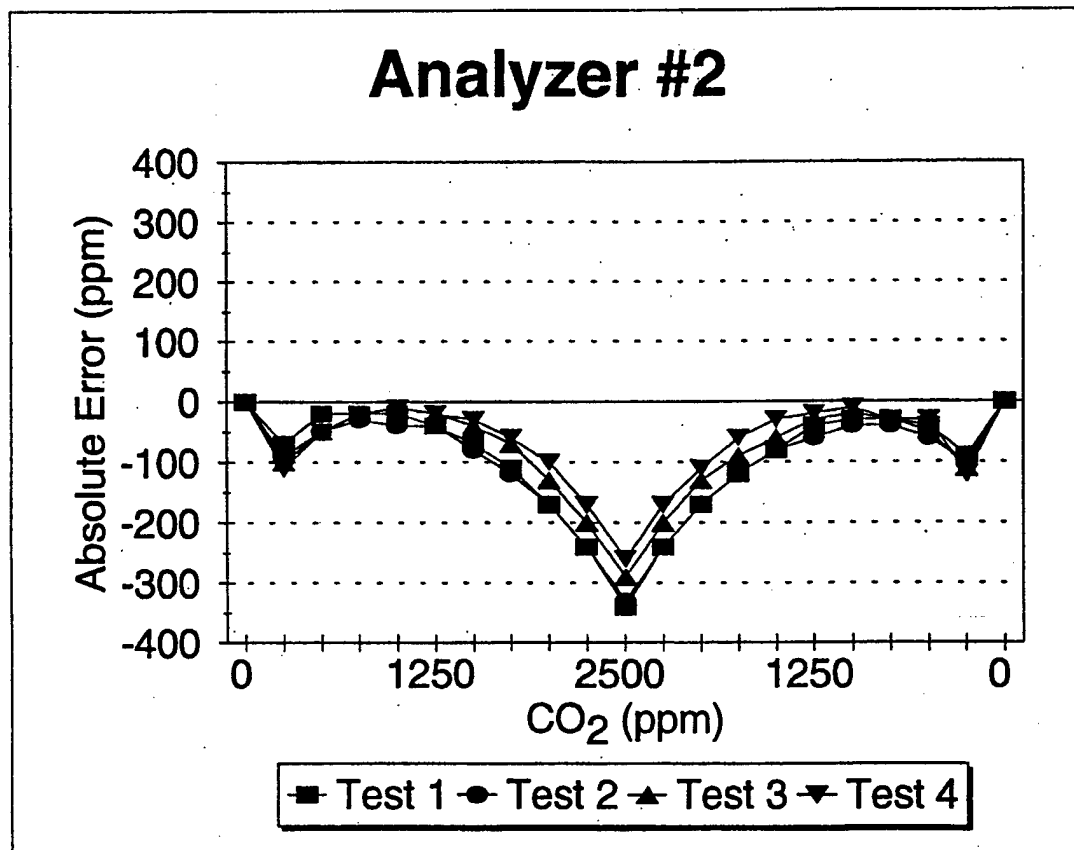
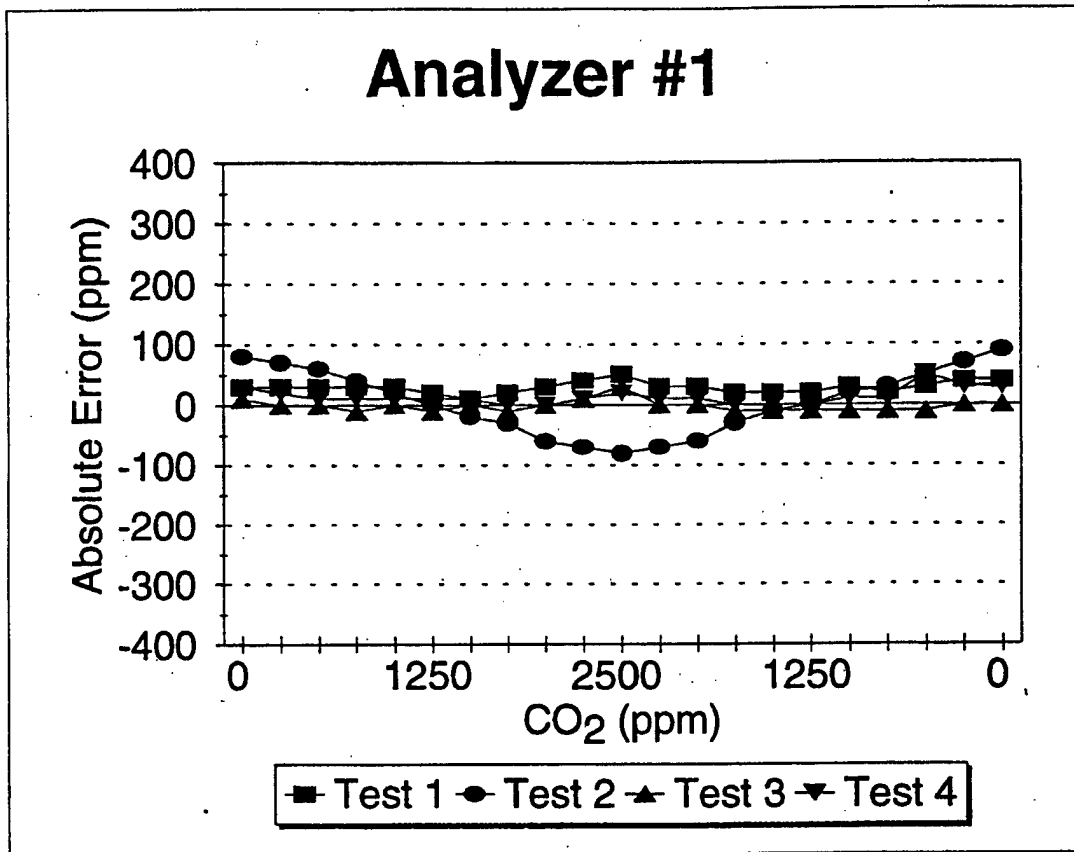


Figure 3c. CO accuracy.

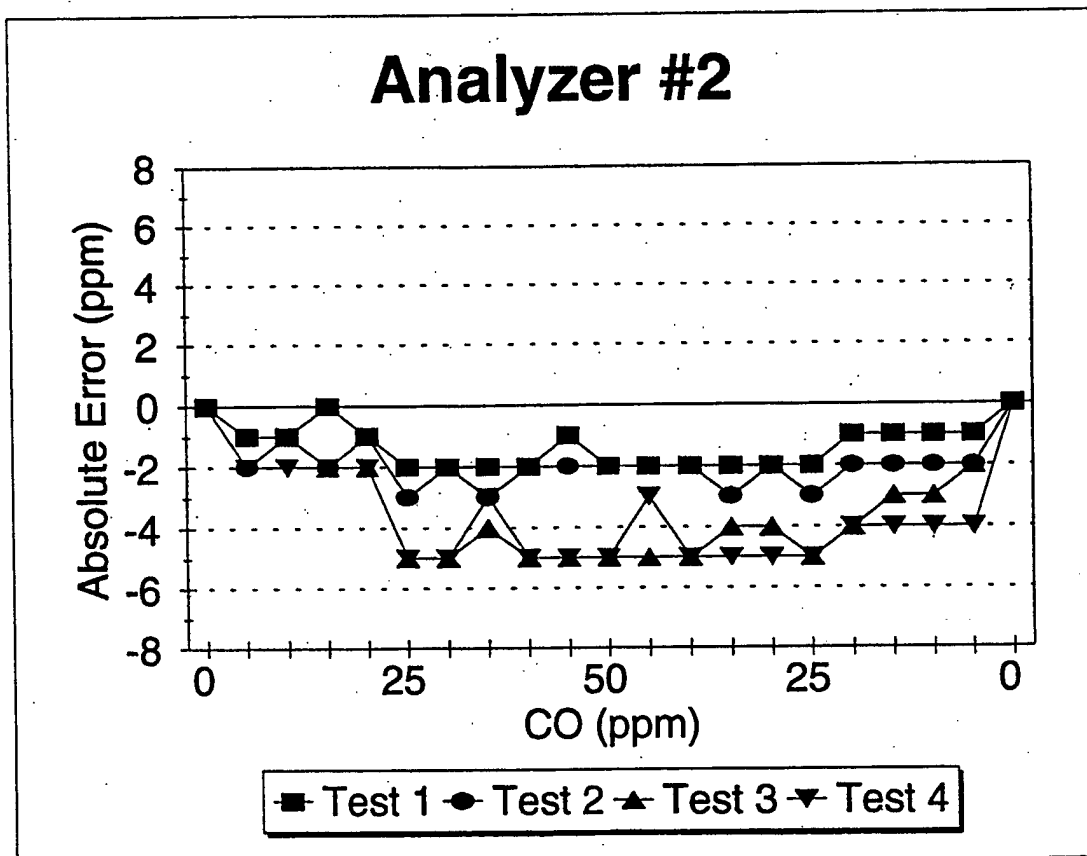
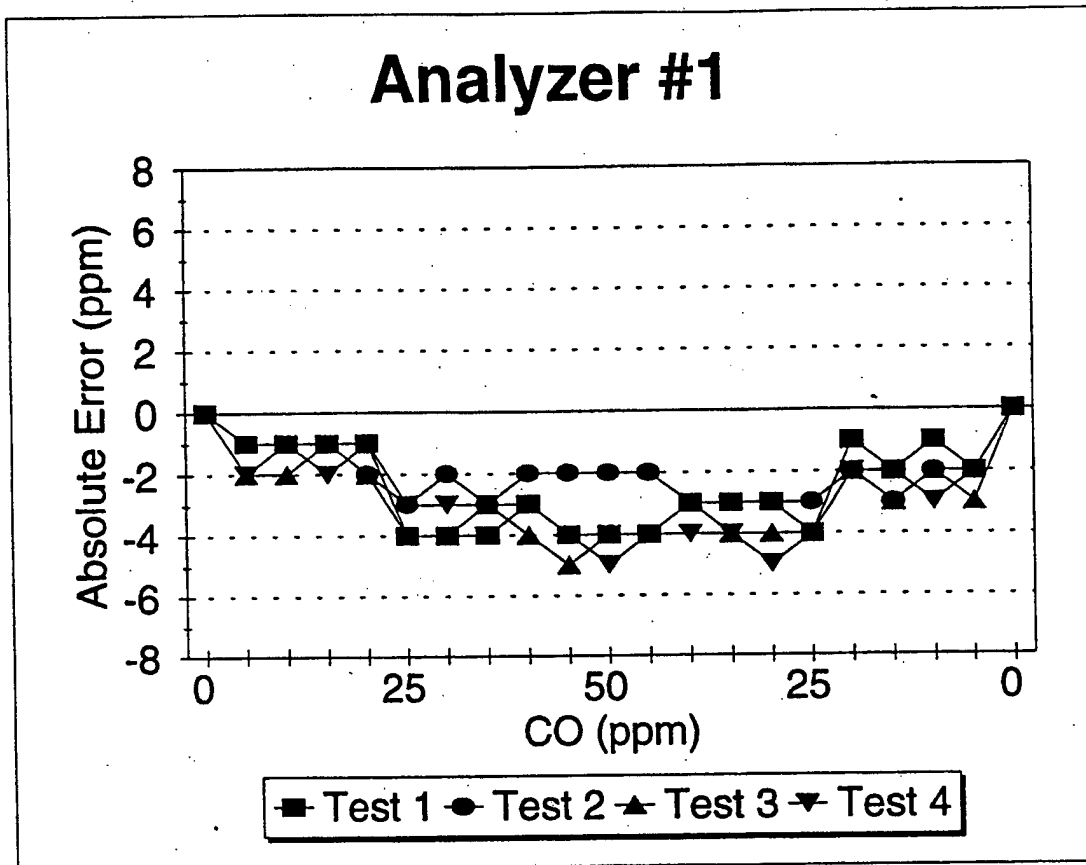


Figure 4a. CO₂ interference testing - analyzer #1.

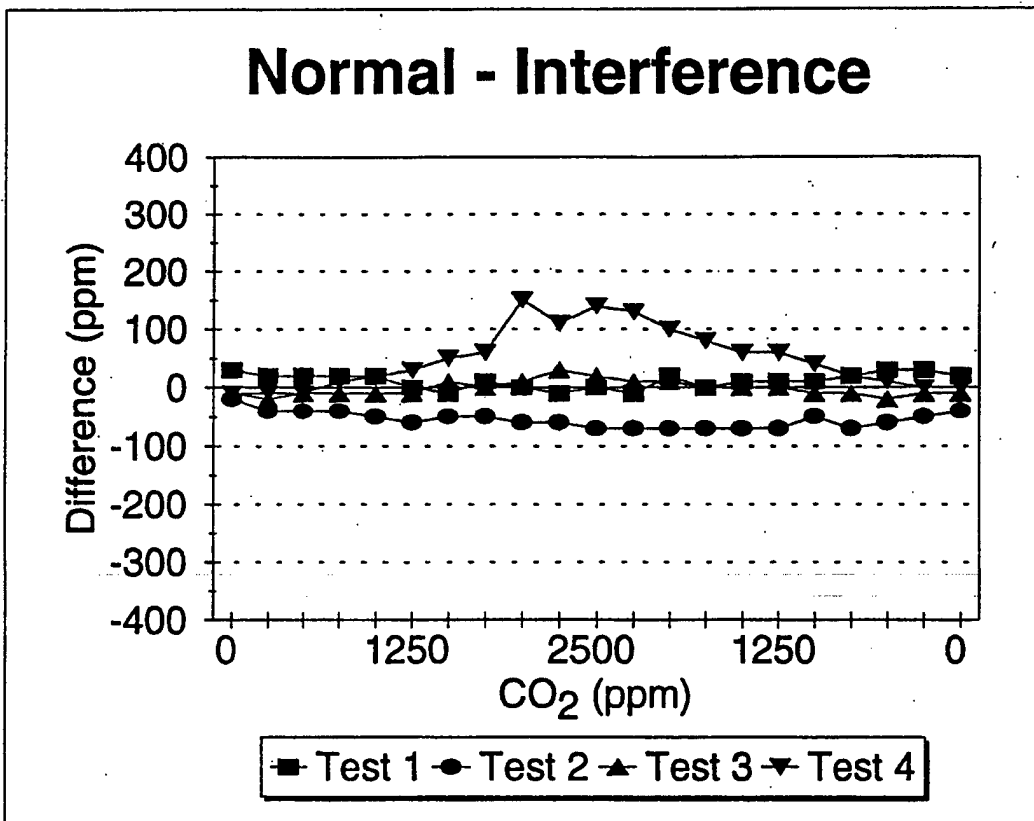
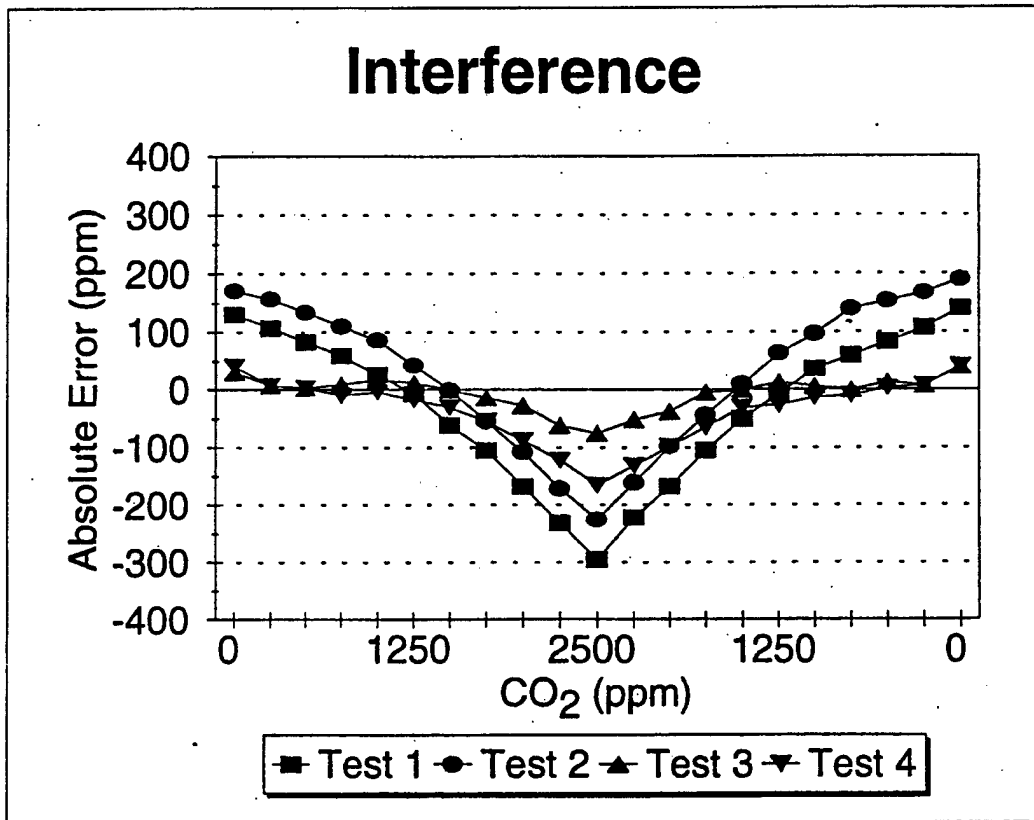


Figure 4b. CO interference testing - analyzer #1.

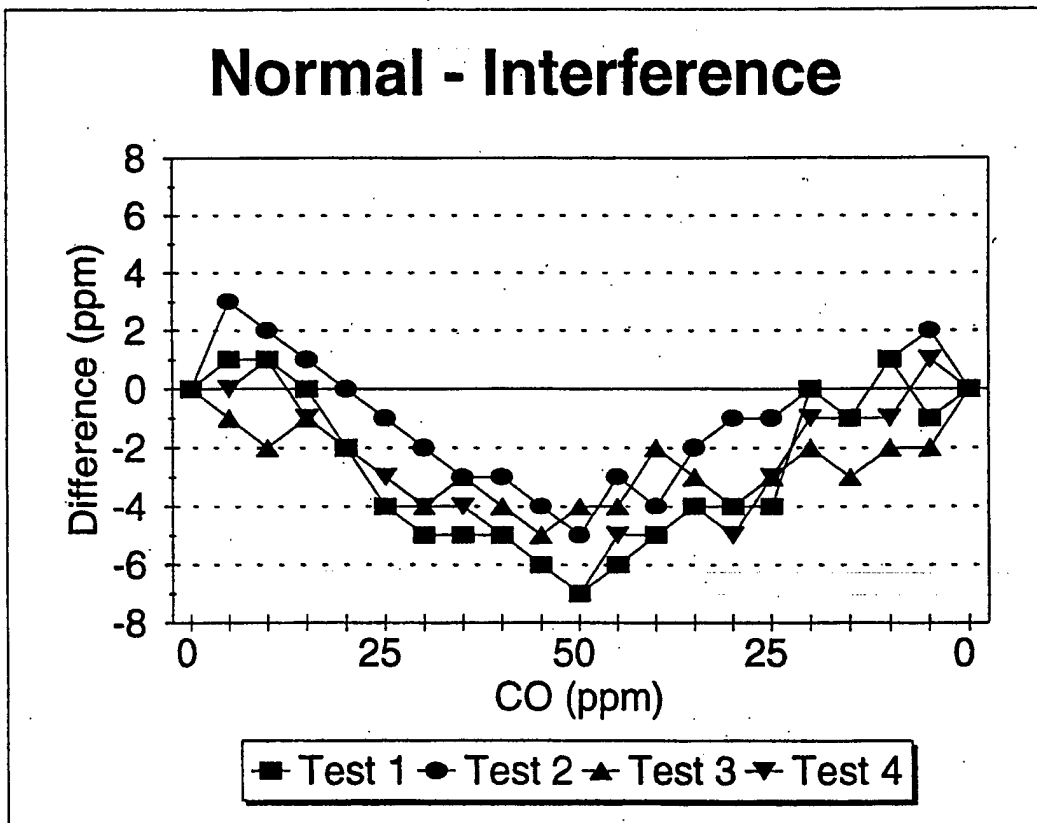
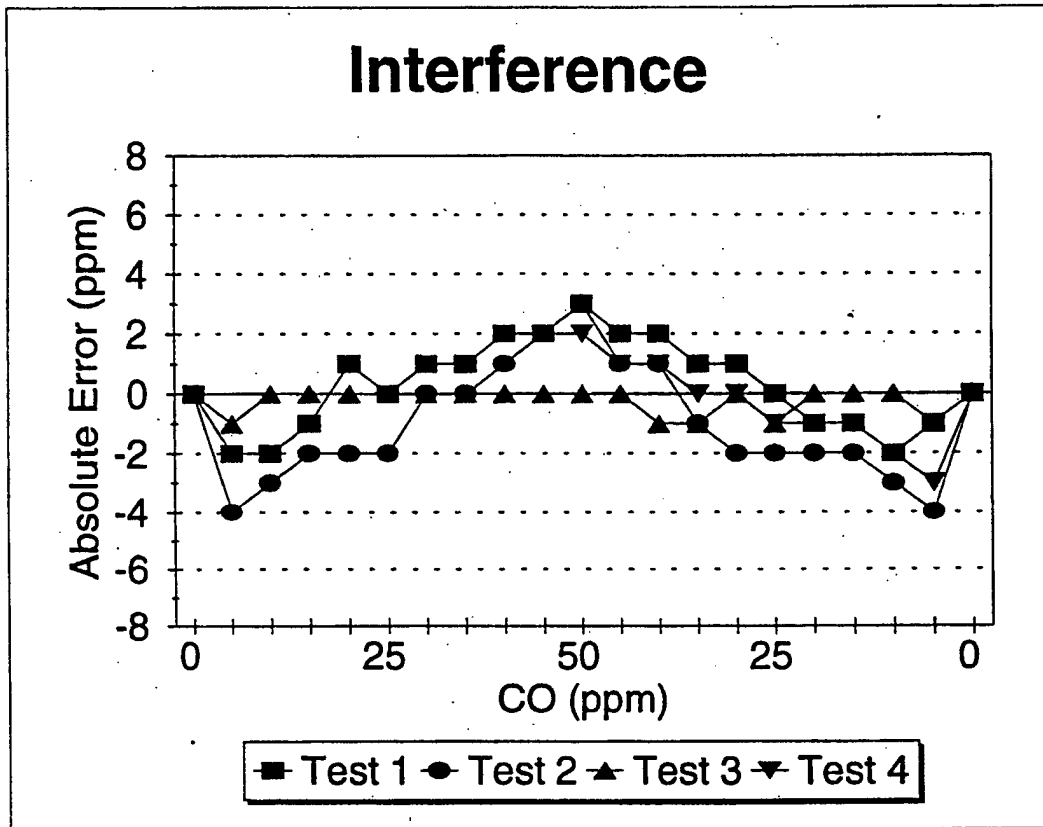


Figure 4c. CO₂ interference testing - analyzer #2.

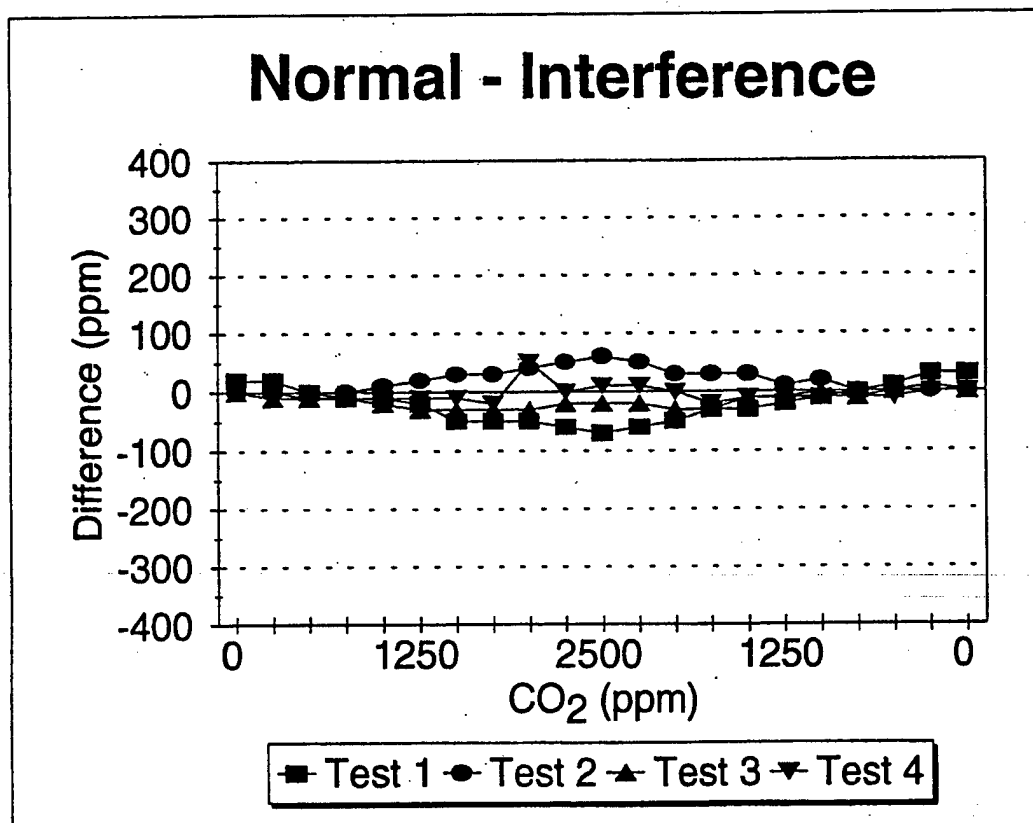
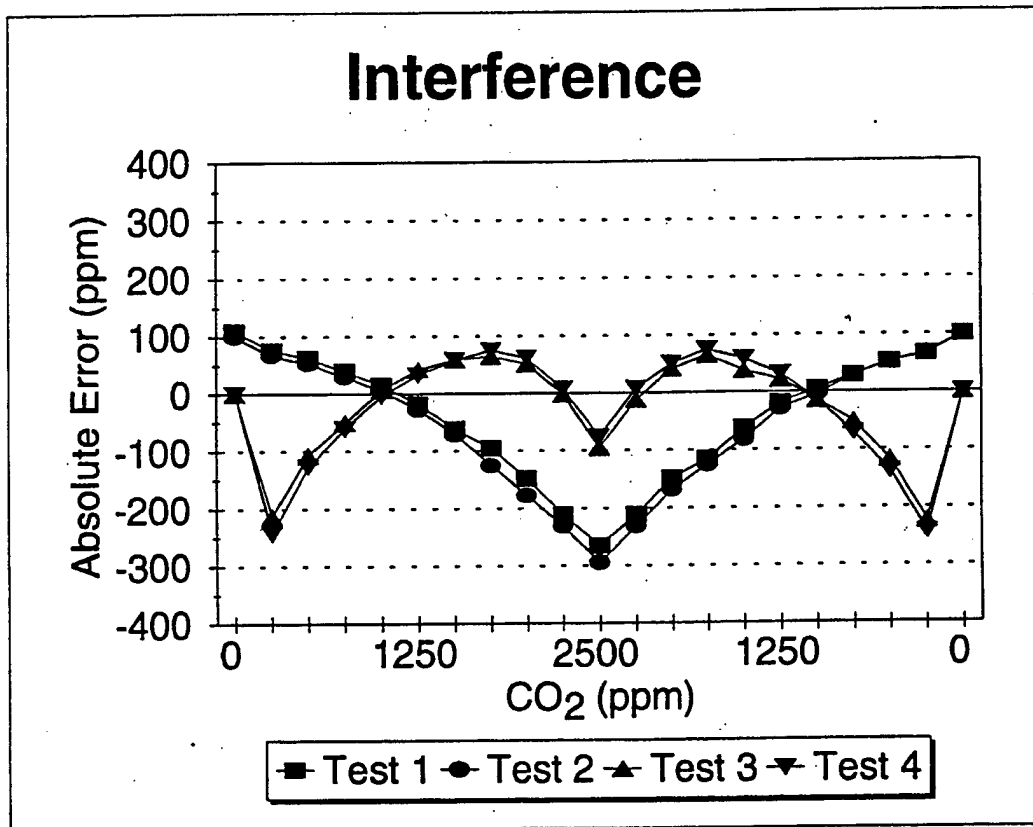


Figure 4d. CO interference testing - analyzer #2.

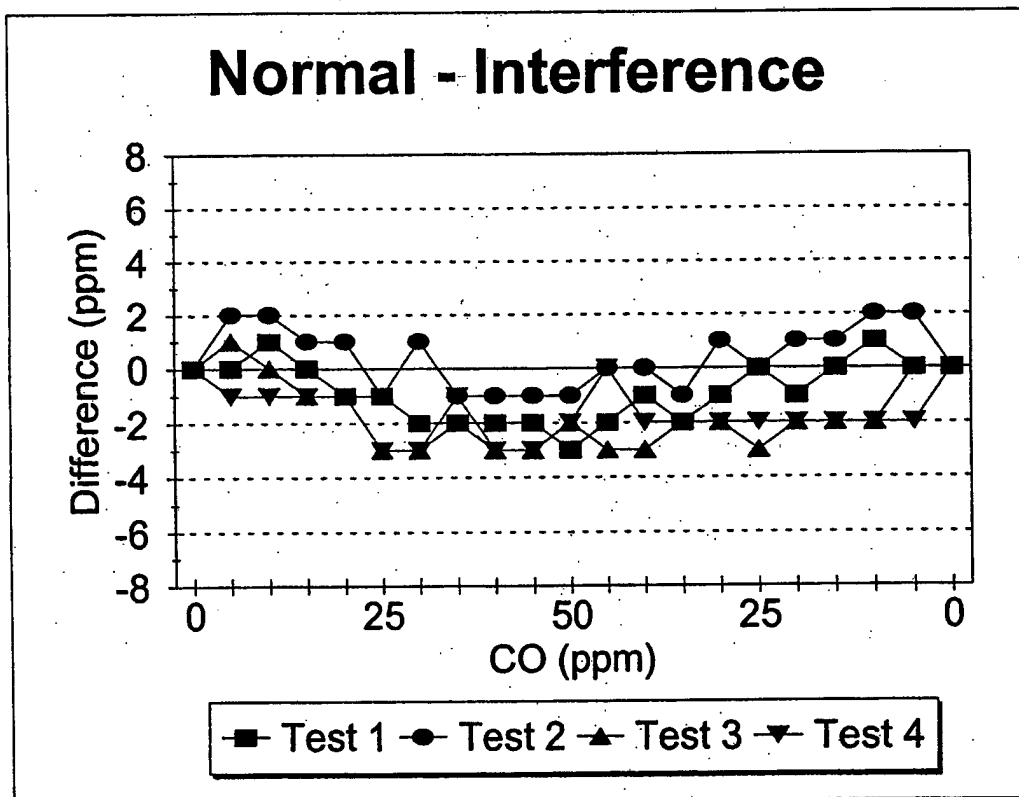
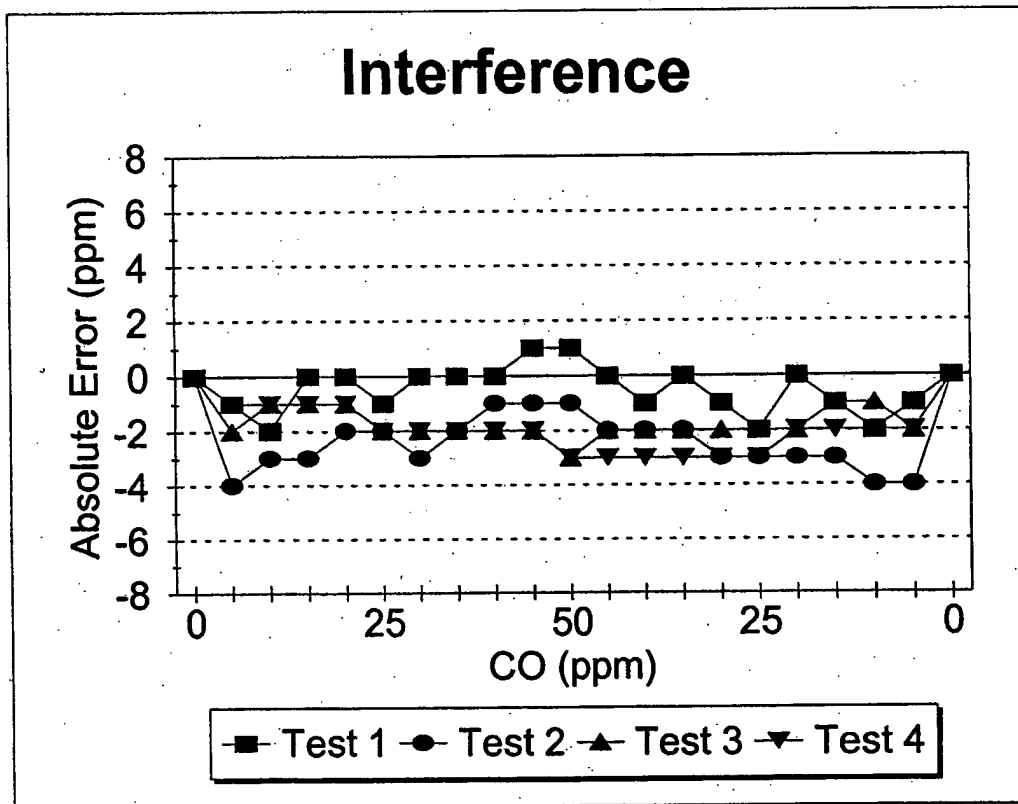
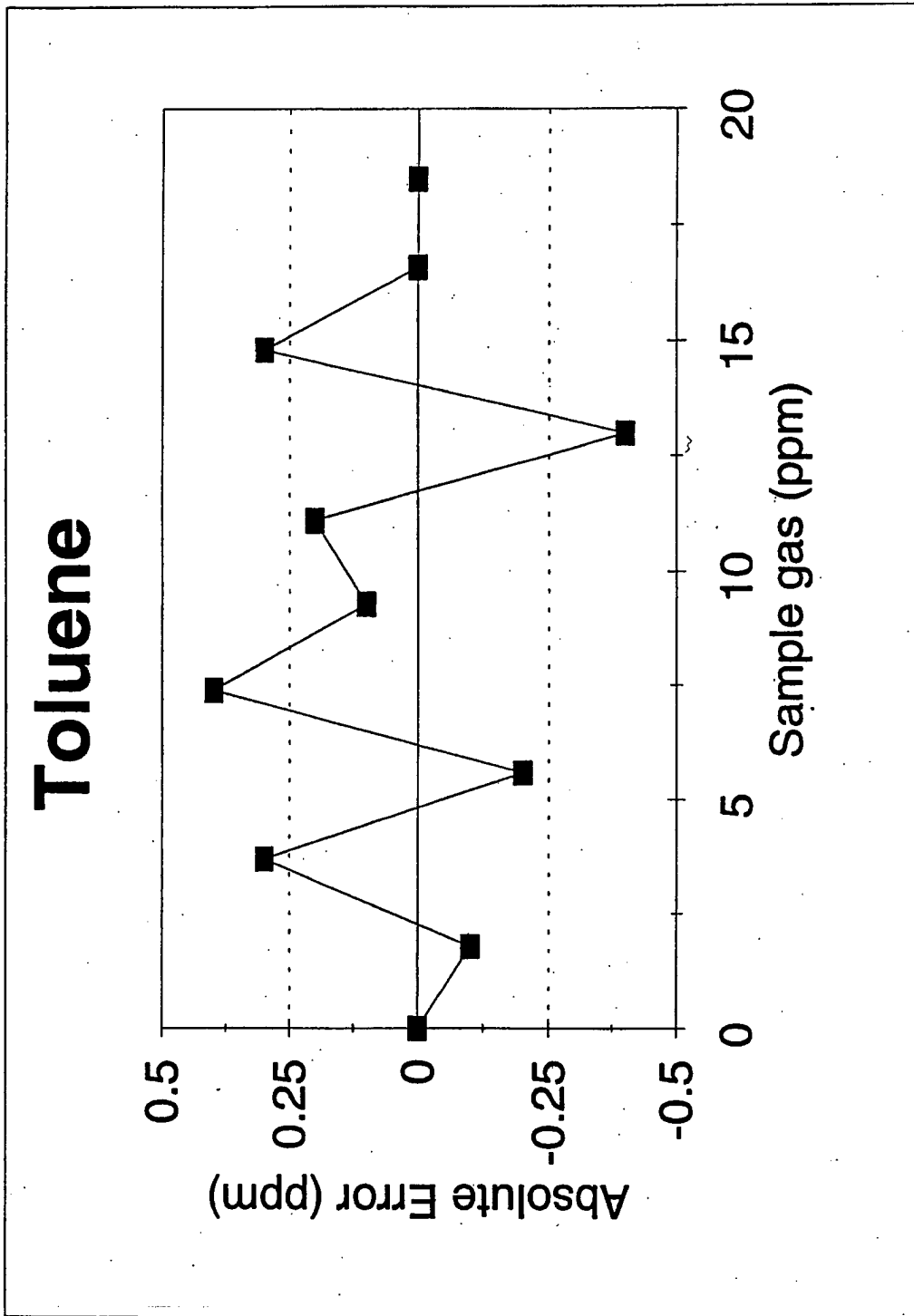
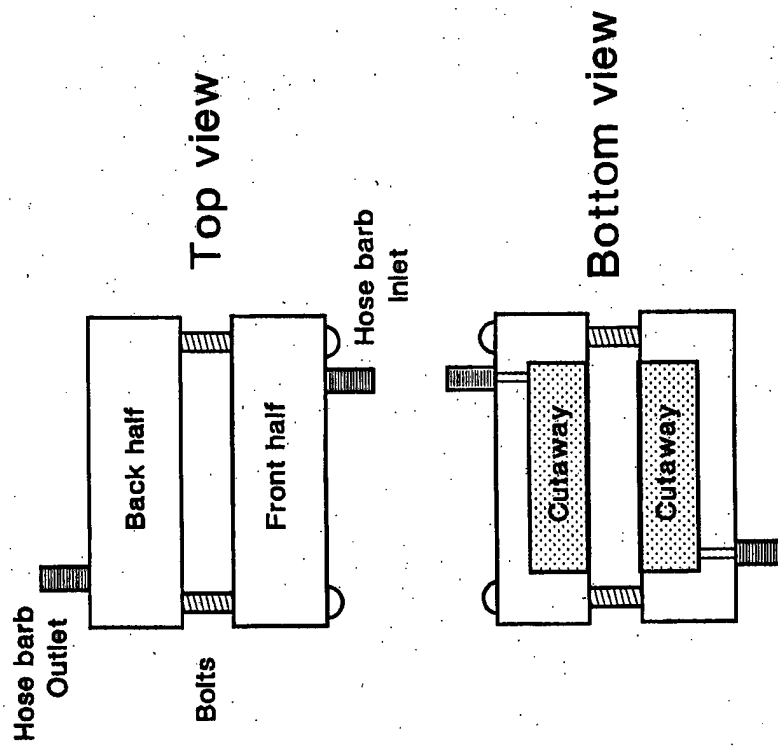
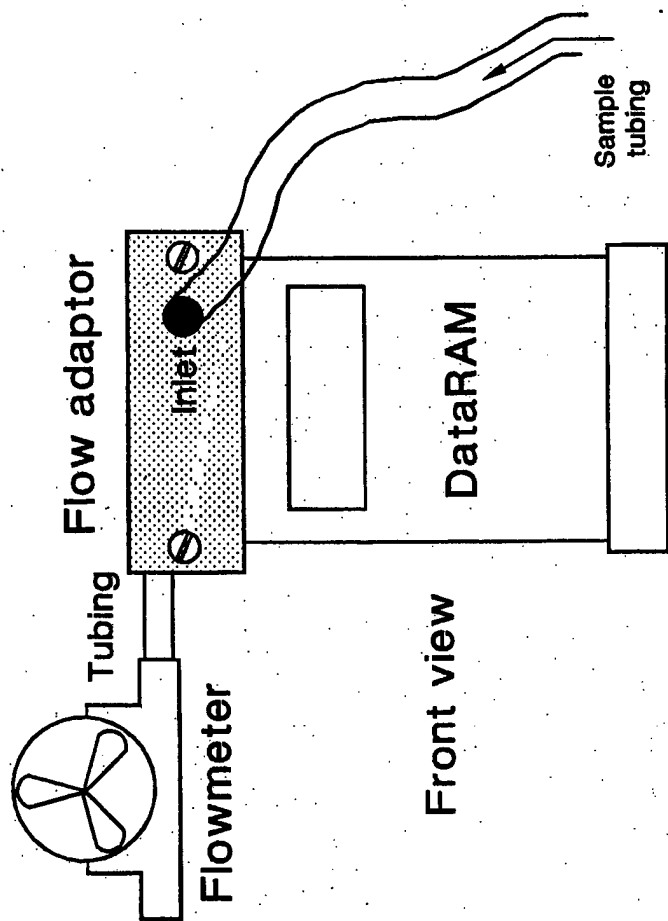


Figure 5. TVA accuracy.



**Figure 6. DataRam setup
Flow adaptor**



Flow adaptor

Setup

Figure 7. Calibration setup: O₂/CO₂/CO analyzer and Toxic Vapor Analyzer (TVA).

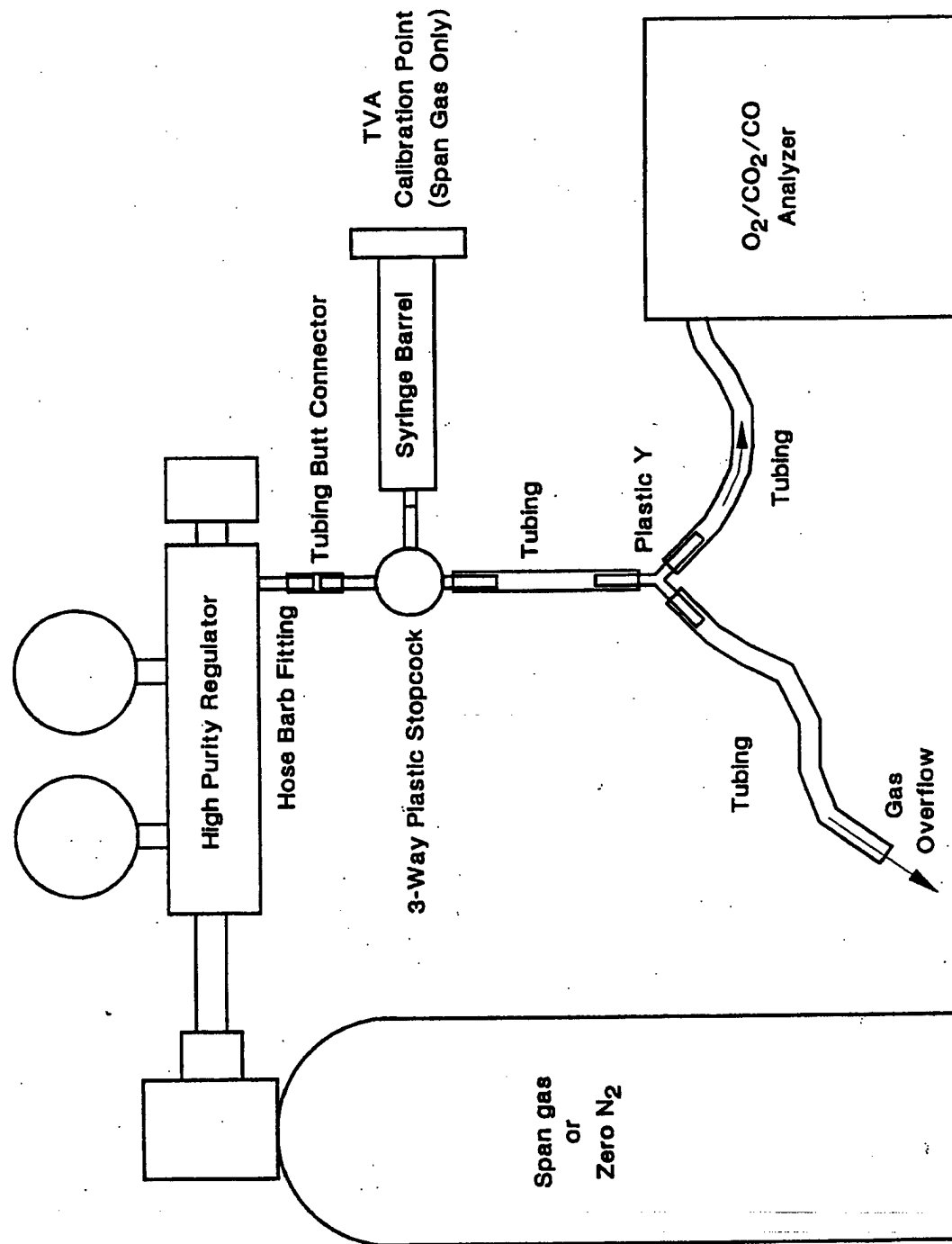


Table 1. Oil response factors for 2 Personal/DataRAM Aerosol Monitors

<u>Oil Type</u>	<u>Mean (SD)</u>	
	DataRAM 1	DataRAM 2
<u>Hydrocarbon-based</u>		
2190 TEP (Imperial Oil)	.450 (.086)	.472 (.084)
2190 TEP (Duro)	.370 (.013)	.385 (.006)
<u>Synthetic</u>		
Mobil Rarus 827	0.623 (.039)	0.639 (.036)
Mobil Rarus 829	0.463 (.053)	0.461 (.063)
Anderol 750	0.431 (.051)	0.451 (.020)
QuinSyn	0.429 (.048)	0.464 (.091)
ALL 6 OILS	0.466 (0.087) n = 19	0.480 (0.088) n = 21

Values for individual oils based on 2-5 tests.

$$\text{Response factor} = \frac{\text{Gravimetric concentration (mg/m}^3\text{)}}{\text{DataRAM measurement (mg/m}^3\text{)}}$$

APPENDIX A

LABORATORY TESTING – O₂/CO₂/CO ANALYZER

Two Anagas Dive Air analyzers, model DV1.1, were evaluated in the laboratory for accuracy in screening diver's air for O₂, CO₂, and CO.

METHODS

Instruments were stored, calibrated, and tested at laboratory temperatures ranging between 19 and 24 °C. Occasionally, overnight storage temperatures were several degrees centigrade outside of this range due to problems with the building temperature control. Analyzers were recharged if necessary the night before each day's testing by connecting the battery charger, which was plugged into line power. The charger was disconnected the next day prior to calibration and testing so that all procedures were done with analyzers operating on battery power.

The following gases were used during testing:

1. Zero N₂: hydrocarbon-free
2. Two gravimetric standards with the following nominal concentrations:
 - a. 2,500 ppm CO₂
 - b. 50 ppm CO

Both standards were in balance hydrocarbon-free air, obtained commercially, and certified to ± 1% relative.

Analyzers were tested on the laboratory counter at ambient pressure. Gas was delivered to both analyzers simultaneously via a branching circuit (**Fig. 2**) made of wide-bore (3/16 inch inner diameter) Tygon tubing and plastic Y-shaped connectors so that a slight excess of gas exited the overflow. This ensured that the analyzers sampled only test gas, and not ambient air, and that only minimal back pressure (< 1 psi) was placed on the flow to the analyzer. Overflow was adjusted to several liters/min, although it was confirmed numerous times that instrument reading was unaffected when gas flow to the instruments was varied over a wide range.

Gas was supplied to the analyzers via the branching circuit using a precision gas divider (STEC model SGD-710, Horiba Instruments, Inc. Ann Arbor, MI; **Fig. 2**). The STEC device allowed blending of the standards with a diluent gas (zero N₂) in 10 equal steps from 0 to 100% of the standard concentrations of O₂, CO₂, and CO. Teflon tubing was used to connect the standards and diluent gases to the STEC. This gas divider has been previously shown to be linear to within the manufacturer's specification of ± 0.5% of full scale, using low ppm levels of volatile organic compounds and up to 25%

of fixed gases (e.g., O₂, CO₂; Reference (3)). Thus, with the STEC, an entire response curve could be generated from a single gas standard.

Analyzers were turned on at the beginning of each test day, disconnected from the battery charger, and batteries verified as fully charged. Analyzers were then allowed to warm up for at least five minutes and calibrated together as described in **Appendix F** at the screening limits. Span calibration for O₂ and CO₂ was done first using the 2,500 ppm CO₂ standard (nominal 21.0% O₂). In this case, the STEC was first set to 40 resulting in a nominal 1,000 ppm CO₂ concentration. The STEC was then reset to 100 producing a nominal 21.0% O₂ concentration. The STEC standard was then switched to the 50 ppm CO standard and the STEC set back to 40 resulting in a nominal 20 ppm CO span concentration. No correction for barometric pressure was made.

Calibration involved first zeroing the O₂ and CO channels using 100% N₂ delivered via the STEC; CO₂ does not require zeroing. Span gas was then delivered to the analyzers and the digital readouts were checked. If the readings were not within 20 ppm CO₂, 0.1% O₂, and 1 ppm CO of the delivered concentrations, the analyzer was recalibrated by entering the correct concentrations using the keyboard. At least one minute was allowed for readings to stabilize.

Analyzers were tested up to several times per week over a period of approximately six months for the following:

1. Precision/accuracy. Calibrated instruments were presented with the undiluted CO₂ standard followed by zero N₂ and time allowed for readings to stabilize. This was done for a total of five cycles over a ten-minute period in order to define short-term repeatability (precision). Calibration was then checked and the analyzer(s) recalibrated if the readings were not within the ranges defined above. STEC response curves were then generated by varying the STEC setting from 0 to 100% and back to 0% in order to define the accuracy across the range of concentrations. These procedures were repeated using the CO standard.

2. CO₂/CO interference testing. STEC response curves were generated using one standard as the calibration gas and the other standard as the diluent. In this way, mixtures of CO₂ and CO were produced testing whether these two gases interfered with each other during analyzer operation.

ANALYSIS

1. Precision

Means and standard deviations were calculated from the precision data.

2. Accuracy

Accuracy data were used to calculate absolute error:

$$\text{Absolute error} = \text{Observed reading} - \text{Expected reading}$$

Where

$$\text{Expected reading} = \text{Gas standard concentration} * (\text{STEC setting}/100)$$

3. CO₂/CO interference testing

Differences were calculated between response curves produced by normal STEC tests (using N₂ as the diluent) on one day and response curves produced during CO₂/CO interference tests (using one of the gas standards as the diluent) the following day.

RESULTS AND DISCUSSION

The following findings are based on extensive testing performed over a period of approximately six months.

PRECISION

Standard deviations based on five repeated measurements of ~21.0% O₂, 2,500 ppm CO₂, and 50 ppm CO were routinely < 0.1% for O₂, < 20 ppm for CO₂, and < 2 ppm for CO. No problem with zero drift was observed with either instrument; when presented with zero N₂, analyzers read less than 0.1% O₂, 80 ppm CO₂, and 2 ppm CO.

ACCURACY

After calibration with 21.0% O₂, 1,000 ppm CO₂, and 20 ppm CO, measurement error for the two instruments (**Figs. 3a-c**) were with rare exceptions:

1. Within 0.2% O₂ for concentrations from 0 to 21% O₂,
2. Within 100 ppm CO₂ for the 1,000 ppm limit concentration, although error increased up to ± 500 ppm over the entire concentration range of 0 to 2,500 ppm CO₂,
3. Within 2 ppm CO for the 20 ppm limit concentration, although the error increased up to ± 6 ppm over the concentration range of 0 to 50 ppm CO.

Testing indicated that there are differences between the two analyzers in accuracy over the concentration ranges tested, particularly with regard to CO₂.

CO₂/CO INTERFERENCE

Any differences between normal and interference curves were within the observed measurement error reported in the preceding section (Figs. 4a-d). The relatively large difference in CO responses, between normal and interference tests, seen in the bottom graph of Fig. 4b, are undoubtedly day-to-day differences as no CO₂ was present in the test mixture when 50 ppm CO was delivered with the STEC set at 100. As earlier described, the interference tests were conducted one day after each of the normal tests.

CONCLUSIONS

Based on these results, accuracy of measurements at the screening limits, after calibration at the limits, is estimated to be:

O ₂ :	21.0% ± 0.2%
CO ₂ :	1,000 ± 100 ppm
CO:	20 ± 2 ppm

However, measurement of concentrations much higher than the limit, after calibrating at the limit, may have considerable error associated with them. There was no evidence of CO₂/CO interference during use of the analyzer.

APPENDIX B

LABORATORY TESTING – TOXIC VAPOR ANALYZER (TVA)

Six Toxic Vapor Analyzers, model TVA-1000B, were tested extensively in the laboratory during the development of the new NMRI Revised Air Purity Guidelines for Dry Deck Shelter (DDS) Operations⁴. Instrument performance was evaluated, and response factors for a number of chemicals were determined and compared to those reported by the manufacturer. In this report, only testing of instrument linearity across a range of chemical concentrations will be described.

METHODS

Instruments were stored, calibrated, and tested at laboratory temperatures ranging between 19 and 24 °C. Occasionally, overnight storage temperatures were several degrees centigrade outside of this range due to problems with the building temperature control. Testing was done with the TVA either 1) connected to the battery charger, which was plugged into line power or 2) disconnected from the charger and, thus, operating on battery power.

The following gases were used during testing:

1. Zero air: CO₂-free, hydrocarbon-free air.
2. Gravimetric standards of individual volatile organic compounds including: isobutylene, methane, octane, Freon 113, toluene, and benzene; all at nominal concentrations from 10-20 ppm. All standards were in balance hydrocarbon-free air, obtained commercially, and certified to at least $\pm 2\%$ relative.

TVAs were turned on at the beginning of each test day and allowed to warm up for at least 30 minutes. Instruments were then calibrated as described in **Appendix F** using one of the gas standards listed above. TVA response curves were then generated in the laboratory over a range of 0 to 100% of the same standard used for calibration. This was done using the STEC gas divider in similar fashion as for the O₂/CO₂/CO analyzer (**Appendix A**). The only exception was that zero air, instead of zero N₂, was used for the STEC diluent gas to maintain ~21% O₂ in all gases delivered to the TVA. This was necessary to support the TVA flame which, as previously indicated, obtains its O₂ from the gas being sampled. Response curves were produced for the other standards following recalibration with those standards. The STEC response curves were used to define the accuracy of the TVA across the range of concentrations.

ANALYSIS

Accuracy data were used to calculate absolute error:

$$\text{Absolute error} = \text{Observed reading} - \text{Expected reading}$$

Where

$$\text{Expected reading} = \text{Gas standard concentration} * (\text{STEC setting}/100)$$

RESULTS AND CONCLUSION

After calibration with any of the 10-20 ppm volatile organic standards, TVA measurement error was less than 0.5 ppm over the concentration range from tested (see the representative response curve in **Fig. 5**). Although linearity is important, total hydrocarbon measurement will depend significantly on the nature of the contaminants and the type of analyzer used.

APPENDIX C

LABORATORY TESTING – AEROSOL MONITOR

Two Personal/DataRAMs, model pDR-1000, were tested in the laboratory to determine their response factors for different oils commonly used in air compressors. The DataRAMs were evaluated using a precision aerosol generator (model 3076, TSI Inc., St. Paul, MN) and two GRIMM Portable Dust Monitors, (series 1,100; Pioneer Emissions Detection & Control, Inc., Wilmington, DE). The GRIMM devices, like the DataRAMs, are photometers that use light scattering to measure aerosols. Unlike the DataRAMs, the GRIMM monitors actively sample the air using a pump and are designed to allow gravimetric verification of their optical readings by simultaneous aerosol collection on an internal filter. Operating both the DataRAMs and the GRIMM monitors together in close proximity, thus, permitted DataRAM response factors for the various oils to be determined by comparison to the GRIMM gravimetric results. Although response factors were also determined for the GRIMM monitors, these will not be reported here. The DataRAMs were also tested after being configured with the Flow-Adaptor (Fig. 6), a device developed by NMRI to allow aerosol sampling of a gas stream. In addition, the Air-Sampler, that allows gas to be sampled from the high pressure air line, was evaluated for its effect on aerosols. All oil mist experiments were done inside a fume adsorber hood (Model 69000, Labconco Corp., Kansas City, MO) to avoid exposure to the aerosol.

METHODS

Selection of oils for the aerosol tests

Six different oils, commonly used for lubrication of compressors for diver's breathing air, were obtained from several sources within the Navy. These consisted of two hydrocarbon-based oils and four synthetic oils. All oils tested were unused samples taken directly from the original manufacturer's container and are listed below.

1. Hydrocarbon-based oils

- a. 2190 TEP:
Imperial Oil Co. Inc., Toronto, Canada
- b. Duro 2190 TEP:
Lyondell Petrochemical Co., Houston, TX

2. Synthetic Oils

- a. Mobil Rarus 827:
Mobil Oil Corp., Fairfax, VA
- b. Mobil Rarus 829:
Mobil Oil Corp., Fairfax, VA

- c. Anderol 750:
Huls America, Inc., Piscataway, NJ
- d. Quin-Syn-Recip (111479):
Quincy Compressor Division, Colt Industries, Quincy, IL

Based on information from within the Navy, the oil most commonly used in air compressors aboard submarines and ships is believed to be the hydrocarbon-based 2190 TEP. However, for shore-based compressors, the lubricating oil of choice is usually that recommended by the manufacturer of the particular compressor. Compressor manufacturers tend to favor the use of ester-based synthetic lubricants such as Rarus or Anderol. Some manufacturers of air compressors also recommend their own brand of oil.

Oil mist generation

Oil mists were generated using a constant output atomizer (model 3076, TSI Inc., St. Paul, MN) set up to operate in the recirculating mode. The atomizer works by forcing compressed air through a small orifice to form a high velocity jet which atomizes the oil introduced through a vertical passage in the atomizer head. Large droplets are removed by impaction on the wall opposite the jet and excess oil from the atomizer drains back into the originating container.

Clean, dry compressed air from a high pressure cylinder was used as the air source. The line pressure to the atomizer assembly was maintained at 30 psi via the cylinder regulator. The oil mist was emitted from the atomizer at atmospheric pressure and directed to the fume hood via a 90 cm length of 1/2 inch Poly-Flo tubing supplied by the manufacturer of the atomizer. The dimensions of the fume hood were 0.3 m (height) x 0.7 m (width) x 0.3 m (depth) thus giving it an internal volume of approximately 0.06 m³. Twelve 1/2 inch diameter holes were drilled at various intervals through the top surface of the hood to permit the aerosol delivery tube to be inserted at various locations. Those holes not used during an aerosol test were sealed using rubber stoppers.

It was found during preliminary work that it was impossible to achieve the desired aerosol concentration (5-10 mg/m³, discussed below) inside the fume hood without modification of the setup. The aerosol concentration either quickly built up to very high levels when the hood fan was off or was maintained at low levels when the one-speed fan was on. Consequently, the aerosol concentration was brought into the desired range by 1) adjusting the air flow into the hood by sealing with tape all or a portion of the small gap between the front cover of the hood and the laboratory bench, and 2) restricting air flow leaving the hood by taping a cardboard cover in front of the vents inside the hood leading to the charcoal filter and fan. The atomizer was cleaned with methanol and dried between tests involving different types of oil.

Testing of oil response factors

Each type of oil was tested a minimum of two times to determine its response factor. In order to keep weighing errors associated with gravimetric analysis to < 10%, it was necessary to run aerosol trials until approximately 1 mg of aerosol was collected on the internal filter of the GRIMM dust monitor. In addition, it was desired to keep the test aerosol concentrations in the range of 5-10 mg/m³ to be close to the 5 mg/m³ limit. Due to the flow rate of the GRIMM, this necessitated a total testing time of up to seven hours.

Prior to testing, the two DataRAMs were zeroed, following the manufacturer's instructions, by placing the instrument into a ziplocked plastic bag (noted as "Z-pouch" by the manufacturer) and pumping air into the bag using the hand pump supplied with the analyzer. The hand pump with its air filter ensured that the air entering the Z-pouch was free of any significant particulates or aerosols. During the two-minute zeroing sequence, the DataRAM measures its own optical background and stores that level in memory. Subsequently, the DataRAM then automatically subtracts that background from all measured concentration values until the user updates the zero.

During each test, one or two each of the DataRAMs and the GRIMM monitors were placed in the fume hood and allowed to simultaneously sample the oil mist produced by the atomizer. In order to minimize errors due to variations in the concentration of aerosol within the fume hood, the analyzers were placed at the opposite end from where the aerosol delivery tube entered the hood. In addition, the sampling heads of the four analyzers were carefully adjusted so as to be located at the same height above the floor of the hood and as close to each other as possible. The positions of the two DataRAMs were switched midway through each test to minimize any biasing effects that might have been caused by local differences in aerosol concentration. To allow the GRIMMs' gravimetric readings to be used to determine response factors for the DataRAMs, all monitors' internal clocks were synchronized and data collection was started and stopped at the same time. During tests, the DataRAM logging feature was activated to store the aerosol data at one-minute intervals and the display averaging time was set at six seconds. DataRAM configuration settings were adjusted using the computer communications software supplied with the instrument.

Testing of the DataRAM Flow-Adaptor

Comparisons were made between simultaneous aerosol measurements taken inside the fume hood with 1) one DataRAM, configured normally, where air passively moved into the optical sensing chamber of the instrument and 2) the other DataRAM, equipped with the NMRI-designed Flow-Adaptor (Fig. 6), where air was actively pumped into the optical chamber. Prior to testing, the DataRAM equipped with the Flow-Adaptor was zeroed by pumping air over the optical chamber during the two-minute procedure, after attaching the hand pump/filter to the inlet port of the Flow-Adaptor. After also zeroing the normal DataRAM, both were placed inside the hood. A circulating gas pump (Model MB-41, Metal Bellows Corp., Sharon, MA), positioned on

the outside of the hood, was then attached to the inlet side of the Flow-Adaptor using a tube inserted through one of the holes in the top of the hood. This pump was used to deliver oil mist at approximately 0.4 scfm to the DataRAM drawn from within the hood close to where the other DataRAM was passively sampling.

Testing of the Air-Sampler

The effect of the Air-Sampler on the aerosol concentration of the sample gas was also evaluated. A method to test the Air-Sampler at pressures above ambient, where it would normally be used during sampling diver's air, was not developed. However, ambient pressure testing was done by circulating oil mist, generated by the atomizer in the fume hood, using the circulating gas pump (described just above), through a circuit consisting of first one DataRAM, then the Air-Sampler, followed by the second DataRAM, before being returned back into the hood space. Flow-Adaptors installed on both DataRAMs allowed the gas to be circulated with a slight positive pressure. Aerosol concentrations were stored in DataRAM memory at six-second intervals. Midway through the approximately six-hour testing, the DataRAMs were switched in position, to avoid potential instrument bias.

GRIMM Dust Monitor gravimetric measurements

The GRIMM Dust Monitor actively samples the environment via its internal pump. After flowing through the optical chamber, the air sample passes through a 0.5 μm PTFE filter at a constant flow rate of 1.2 liters/min where the particulates are collected. The internal filter is weighed before and after an aerosol test to determine the weight of aerosols deposited over the course of the test. This allows the instrument's optical readings, or in this case, the DataRAM's measurement, to be compared with a direct gravimetric measurement.

Prior to each aerosol test, a new pre-weighed filter was inserted into the back of the instrument. The weight of each filter was determined to the nearest 10 μg by taking the average of three readings using an electronic balance (Model ER-182A, A&D Company Ltd., Tokyo, Japan). Immediately following an aerosol test, the filter was weighed again to determine the increase in weight due to the accumulation of aerosol on the filter. The weight of the aerosol was determined by subtracting the average pre-test from the average post-test weight.

Response factor calculation

The mean concentration of aerosol over the course of a test was determined by dividing the weight of aerosol on the filter by the volume of gas that passed through the filter. The gas volume was calculated as the product of the GRIMM flow rate (defined as 0.0012 m^3/min), and the duration of test in minutes. When two GRIMM monitors were used in a test, the average of the two aerosol weights was used for calculation. The DataRAM response factor for a given oil was then determined by dividing the

concentration based on the gravimetric measurement (GRIMM) by the average concentration recorded by the DataRAM:

$$\text{Response factor} = \frac{\text{Gravimetric concentration (mg/m}^3\text{)}}{\text{DataRAM measurement (mg/ m}^3\text{)}}$$

ANALYSIS

Data were compared using t-tests and Analysis of Variance (ANOVA).

RESULTS AND DISCUSSION

Gravimetric measurements

There was no significant difference ($P > 0.05$) in the weight of particulates collected on the filters of the two GRIMM monitors operated side by side. From 15 tests, the average particulate weight (SD) of the two instruments was 1.68 (0.95) mg vs. 1.89 (1.06) mg.

Different oils

Table 1 shows the response factors calculated for each DataRAM for each of the oils tested. Because the two DataRAMs were calibrated at the factory with the same type of dust, they were expected to give similar response factors for a given oil. This was confirmed by ANOVA which showed the difference between the mean response factors (of all measurements) of 0.466 and 0.480 for the two DataRAMs to be non-significant ($P > 0.05$). Since the oils tested had similar refractive indices, it was also expected that there would be very little difference in their response factors. However, this was not the case due to the significantly larger ($P < 0.01$) response factor for the Rarus 827 compared to the other oils tested.

Response factors must be used to convert the DataRAM concentration to an estimated concentration for the type of oil being measured. Based on the limited amount of testing, the value of 0.5 was chosen to correct DataRAM measurements to oil concentration in air. This value may require modification as suggested by additional testing of these or other oils, especially those with markedly different viscosity ratings, as this may affect the particle size distribution. Overall accuracy associated with DataRAM measurements is conservatively estimated, through propagation of error analysis, at $\pm 40\%$ based on: 1) the manufacturer's reported accuracy of $\pm 5\%$ for the test dust, 2) $\pm 20\%$ error assuming a correction factor of 0.5 for oils when the true factor ranges from 0.4 to 0.6, and 3) an additional estimated error of $\pm 10\%$ associated with the proposed air sampling procedures.

Flow-Adaptor

There was no apparent change (< 5% relative difference) in DataRAM readings during several tests comparing the DataRAM configured with the Flow-Adaptor vs. configured normally. This supports the reliability of the Flow-Adaptor for use in sampling aerosols.

Air-Sampler

No apparent change (< 5% relative difference) was observed, during the one test conducted, between oil mist concentrations measured upstream and downstream of the Air-Sampler. This suggests that under low flow and low pressure conditions there is insignificant impaction or drop out of oil particles as the aerosol passes through the sampling apparatus.

CONCLUSIONS

Accuracy of measurement of compressor oil aerosols using the Personal/DataRAM is conservatively estimated to be $\pm 40\%$. This degree of error, as discussed earlier, is probably at least as accurate as the current system.

APPENDIX D

REQUIRED SAMPLING EQUIPMENT

1. The sampling apparatus is defined here as the "Air-Sampler."
 - a. This adaptor or equivalent will be used to sample diver's air and allow analysis using the three portable analyzers. The Air-Sampler should be kept clean and free of oil, grease, and solvents.
 - b. Configuration. The Air-Sampler consists of two 1/4 inch ball valves in series with a 5,000 psi pressure gauge (**Fig. 1a**), with a third 1/4 inch ball valve off to the side. The two-way upstream valve allows the Air-Sampler to be either connected to or isolated from compressor gas. The downstream two-way valve can be turned to direct gas to the analyzers at a flow rate that can be coarsely controlled. The two-way valve on the side allows the Air-Sampler to be purged with sample gas prior to sampling with the analyzers. A plastic stopcock on the downstream valve allows the gas stream to be directed to a specific analyzer as well as the flow rate to be finely controlled.

The Air-Sampler is fabricated using two 1/4 inch stainless steel tees which are connected to the ball valves using NPT to 1/4 inch Swagelock (or alternative) fittings. The pressure gauge is connected to the side branch of the metal tee. All components should be pressure rated to at least 5,000 psi which is the maximum output pressure of any U.S. Navy air compressor. A three-way hard plastic stopcock is attached to one port of the three-way ball valve. This connection can be made using a metal hose barb fitting (1/8 inch) to which the stopcock can be connected using a very short (e.g., 1/2 inch) piece of flexible tubing such as Tygon or rubber as a butt connector, taking care to minimize the amount of direct contact the gas will have with the tubing. Alternatively, a metal luer lock connector (commercially available) can be used to connect the stopcock to the Air-Sampler, thus avoiding the need for a butt connector. To avoid potential aerosol loss, the Air-Sampler should be constructed to provide a straight-through path (no angles) when sampling with the DataRAM.

- c. Sampling. See **Figs. 1a-b**. A syringe barrel (or equivalent; see item #2) is attached to the stopcock to allow sampling with the TVA. The O₂/CO₂/CO analyzer is attached to the stopcock using a short (< 1 foot) length of Tygon or rubber tubing that contains a Y-shaped connector with a side branch of approximately six inches; this allows sampling without pressurizing the analyzer. The DataRAM is attached to the stopcock using its attached length (< 1 foot) of Tygon or rubber tubing.

2. Clean open-mouth gas sampling fittings of 1-1.5 cm diameter and 5-10 cm length, made of hard plastic or metal (not rubber), will be used for sampling air and calibration gas with the TVA. A 5 ml plastic syringe barrel or equivalent is recommended.

3. Tygon or rubber tubing (~1/18-3/16 inch inner diameter) is used for butt connectors, to construct the sampling tubing for the O₂/CO₂/CO analyzer and DataRAM, and to connect a flow monitor to the particulate monitor. Hard plastic Y-shaped connectors are used to construct branching tubing (CO₂ analyzer). See **Fig. 1b**.

4. Two (one as a spare) portable O₂/CO₂/CO analyzers and annual service contract.

a. The manufacturer of the O₂/CO₂/CO analyzer is:

Geotechnical Instruments
Sovereign House, Queensway
Leamington Spa, Warwickshire, CV31 3JR
England
Tel: +44(0)1926 338111

b. The Geotechnical product name and model number for their O₂/CO₂/CO analyzer is the Anagas Dive Air, model DV 1.1. This unit includes an analyzer with the following ranges: 0-25% O₂, 0-2,500 ppm CO₂, 0-50 ppm CO, blue analyzer case, battery charger, blue carry case, and operating manual.

c. Geotechnical Instruments offer an annual service contract for the above analyzer, which is described in **Appendix F**. This contract should be renewed annually at the start of every fiscal year.

5. Two (one as a spare) Toxic Vapor Analyzers (model TVA-1000B; Foxboro Co., 600 North Bedford St., East Bridgewater, MA 02333), with a single flame ionization detector. No special product numbers or warranties have been defined for the U.S. Navy. Additional parts that do not come standard but should be ordered with the TVA include a charcoal filter sampler, charcoal, and at least one spare H₂ cylinder. Recommended spare parts that might need replacement in the field as a result of normal use include: battery pack, filters, replacement charcoal, and detector parts. Extended service plans can be purchased from manufacturer as described in **Appendix F**.

6. Two (one as a spare) portable Personal/DataRAM aerosol monitors (model pDR-1000, MIE, Inc., Bedford, MA 01730). No special product numbers or warranties have been defined for the U.S. Navy. Standard accessories include the carrying case, digital communications cable, zeroing kit, and AC power supply.

7. Sampling head for DataRAM, including inlet and outlet fittings, to allow aerosol sampling of a gas stream (**Fig. 6**). Equivalent to the NMRI-designed Flow-Adaptor. Not available from the manufacturer; must be specially constructed in-house or contracted out commercially.

8. Flow monitor, Thomas Scientific #5084-B10 (Thomas Scientific, Swedesboro, NJ) or equivalent, for indicating low flows (< 1 l/min) on the outlet of the DataRAM. See Fig. 6.

9. Calibration gases and regulators.

- a. Span gas. Two (one as a spare) charged high-pressure cylinders of calibration gas containing four components in balance N_2 : $\sim 21\%$ O_2 , $\sim 1,000$ ppm CO_2 , ~ 20 ppm CO , ~ 10 ppm isobutylene. This is a primary gravimetric standard with accuracy guaranteed to at least $\pm 1\%$ relative for O_2 , CO_2 , and CO and at least $\pm 2\%$ for isobutylene. Cylinders should preferably contain at least 30 ft^3 of gas to ensure an adequate volume of gas for repeated calibrations and should be aluminum and/or treated by the supplier to promote stability of contents.
- b. Zero N_2 . Two (one as a spare) charged high-pressure gas cylinders of hydrocarbon-free N_2 . Again, cylinders should contain at least 30 ft^3 of gas.
- c. Two high-purity regulators (with stainless steel diaphragm, 0-50 psi delivery pressure) with appropriate CGA connections to install onto the span and zero N_2 cylinders. In similar fashion as for the Air-Sampler described above, a metal hose barb fitting (1/8 inch) on the delivery side of the regulator will allow a three-way hard plastic stopcock to be attached using a very short (e.g., 1/2 inch) piece of flexible tubing such as Tygon or rubber as a butt connector (see Fig. 7). Care should be taken to minimize the amount of direct contact the gas will have with the tubing. Alternatively, the regulator can be configured with a metal luer lock connector (commercially available) to connect the stopcock to the regulator, thus avoiding the need for a butt connector.
- d. Gas mixtures and regulators can be obtained from: 1) Scott Specialty Gases, Plumsteadville, PA; 2) Air Products and Chemicals, Inc., Allentown, PA; or 3) MG Industries, Specialty Gas Division, Malvern, PA; as well as from other suppliers.

10. High-pressure gas collection cylinders. The cylinders have been evacuated and have an internal volume of at least 500 ml to ensure an adequate volume of gas for analysis. These cylinders must be suitable for storage of ppm levels of volatile organic compounds for up to one month. Such cylinders should have a working pressure rating of at least 1,000 psig and will require appropriate connection hardware (including a high-purity, high-pressure regulator) to allow cylinders to be filled at ~ 200 psig with compressor air. The laboratory analyzing the gas should be able to supply the cylinders and sampling hardware. Use of the Emergency Air Sampling Kit specified in the Nuclear Powered Submarine Atmosphere Control Manual⁶ is an acceptable alternative.

APPENDIX E

AIR SAMPLING DATA SHEET

SAMPLE DATE _____
FACILITY _____
FACILITY POINT OF CONTACT _____
COMPRESSOR MAKE/SERIAL # SAMPLED _____
SAMPLE SITE (VALVE OR FITTING LOCATION OR #) _____
PERSON(S) DOING SAMPLING _____
COMPRESSOR HISTORY AND/OR COMMENTS:

CALIBRATION

Calibration Gas

ZERO N₂ CYLINDER# _____
CALIBRATION GAS CYLINDER# _____
(BALANCE N₂)
O₂ _____ % CO₂ _____ PPM CO _____ PPM
ISOBUTYLENE _____ PPM

O₂/CO₂/CO Analyzer

ANALYZER SERIAL# _____
TIME CALIBRATED _____

TVA

ANALYZER SERIAL # _____
SPAN READING ENTERED _____ PPM
TIME CALIBRATED _____

DataRAM

ANALYZER SERIAL # _____
TIME ZEROED _____

AIR SAMPLING DATA SHEET (cont.)

DATE _____

COMPRESSOR READINGS

O₂/CO₂/CO Analyzer

O₂ _____ % RANGE = 20-22% PASS _____ FAIL _____
 CO₂ _____ PPM MAXIMUM = 1,000 ppm PASS _____ FAIL _____
 CO _____ PPM MAXIMUM = 20 PPM PASS _____ FAIL _____

SAMPLE TIME _____

TVA - Total Hydrocarbons

FID _____ PPM (without charcoal) FID _____ PPM (with charcoal)
 SAMPLE TIME _____ SAMPLE TIME _____

TOTAL HYDROCARBONS (excluding methane) IN METHANE EQUIVALENTS =

[FID READING (without charcoal) - FID READING (with charcoal)] * 1.5

= (_____ - _____) * 1.5

= _____ PPM (METHANE) MAXIMUM = 25 PPM

Total Hydrocarbons: PASS _____ FAIL _____

Note: The TVA reading in isobutylene equivalents is converted to methane equivalents by multiplying by 1.5, which is the manufacturer's reported value of isobutylene/methane sensitivity.

DataRAM - Oil, Mist, Particulates

_____ mg/m³ SAMPLE TIME _____

OIL MIST CONCENTRATION = DataRAM CONC reading * 0.5

= _____ mg/m³ * 0.5 = _____ mg/m³ MAXIMUM = 5 mg/m³

Oil, Mist, Particulates: PASS _____ FAIL _____

Note: The DataRAM reading is converted to a value for compressor oil aerosols by multiplying by 0.5, as determined by NMRI testing.

Odor

Objectionable Odor: NO (PASS) _____ YES (FAIL) _____

AIR SAMPLING DATA SHEET (cont.)

DATE _____

HIGH PRESSURE GAS COLLECTION (OPTIONAL - FOR TROUBLESHOOTING)

SAMPLE SITE (e.g., VALVE#) _____

CYLINDER # 1) _____ 2) _____

SAMPLE TIME 1) _____ 2) _____

SAMPLED BY (NAME) _____

SAMPLE HISTORY (COMPRESSOR HISTORY AND SAMPLING PROCEDURES):

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APPENDIX F

OPERATING PROCEDURES: O₂/CO₂/CO ANALYZER AND TOXIC VAPOR ANALYZER (TVA)

BATTERIES/CHARGING

Both the O₂/CO₂/CO analyzer and the TVA are normally operated from their self-contained NiCad batteries. These batteries are recharged using chargers that plug into 110 volt line current. However, both analyzers can be operated while attached to their battery chargers when batteries are low. Ideally, NiCad batteries should be fully discharged and recharged during use rather than continually topped off without a full discharge. However, this probably will not be possible and the best approach is to check the battery (described below) well before analyzers will be needed to allow time for 24-hour charging if the battery is low. Complete recharging should provide 12 hours of usage for the O₂/CO₂/CO analyzer and at least eight hours of operation for the TVA. Note: The instruments must have batteries in place to operate.

BATTERY CHECK/CALIBRATION

1. A battery check and calibration need to be done once prior to each day's use of both the O₂/CO₂/CO analyzer and the TVA.
2. Locate the zero N₂ and span gas cylinders. The span mixture is: ~21% O₂, ~1,000 ppm CO₂, ~20 ppm CO, ~10 ppm isobutylene, in balance N₂. NOTE: The isobutylene calibration gas is completely non-toxic and has a long history of safe use. Ensure that both regulators are in place and pressure cycle each regulator three times to remove all ambient air. Dial in a delivery pressure of several psig. Leave gas cylinder valves open but close regulator delivery valves so gas is not flowing.
3. Attach a new plastic stopcock on the end of both regulators and a new 5 ml syringe barrel (or equivalent) on the stopcock of the span gas (see Fig. 7).
4. O₂/CO₂/CO analyzer (fill out calibration information on the data sheet).
 - a. Disconnect analyzer from charger.
 - b. Turn on analyzer by pressing the red key. The LCD will show the company name, model, and information on key functions. Press the "0" key to exit that screen.
 - c. Press "1" for General Utilities.

- d. Battery check. Batteries should be checked without the charger connected. Press "4" to read the available battery capacity. If there is insufficient charge (< 50%), the analyzer should be charged. The instrument may be used with line power via the battery charger if necessary.
- e. Install on the zero N₂ cylinder a short (< 1 foot) length of Tygon tubing that contains a Y-shaped connector with a side branch of approximately six inches. Connect the inlet port of the O₂/CO₂/CO analyzer to the side branch of tubing so that N₂ can be sampled without pressuring the analyzer (see Fig. 7).
- f. Press "0" and then "2" (calibrate), "1" (O₂), "1" (zero). Press "5" to turn on pump. Analyzer is now measuring O₂. Allow to warm up for five minutes.
- g. Open the N₂ regulator valve and adjust gas flow so it is just audible as it exits the overflow.
- h. Wait at least one minute for reading to stabilize. Press "1" (zero level), "0" (exit), "0" (previous menu). O₂ is now zeroed.
- i. Press "3" (CO), "1" (zero). Press "5" to turn on pump. Analyzer is now measuring CO.
- j. Wait at least one minute for reading to stabilize. Press "1" (zero level), "0" (exit), "0" (previous menu). CO is now zeroed.
- k. CO₂ does not require zeroing.
- l. Shut off N₂ gas flow but leave cylinder valve open in case it is needed below for re-calibration. Remove sampling tubing and attach it to the span gas in similar fashion; adjust gas flow so it is just audible as it exits the overflow.
- m. Press "1" (O₂), "2" (span). Press "5" to turn on pump. Analyzer is now measuring O₂.
- n. Wait at least one minute for reading to stabilize. Press "1" (enter gas con) and enter the calibration value. Then press "0" (exit), "1" (yes), "0" (exit), "0" (previous menu). O₂ is now calibrated.
- o. Press "2" (CO₂) and then "5" to turn on pump. Analyzer is now measuring CO₂.
- p. Wait at least one minute for reading to stabilize. Press "1" (enter gas con) and enter the calibration concentration. Then press "0" (exit), "1" (yes), "0" (exit). CO₂ is now calibrated.

- q. Press "3" (CO), "2" (span). Press "5" to turn on pump. Analyzer is now measuring CO.
- r. Wait at least one minute for reading to stabilize. Press "1" (enter gas con) and enter the calibration concentration. Then press "0" (exit), "1" (yes), "0" (exit), "0" (previous menu). CO is now calibrated.
- s. Observe readouts. Meter readings should be within 0.1% O₂, 20 ppm CO₂, and 1 ppm CO of calibration values. Calibration of one or more of the gases can be repeated if needed.
- t. Close valve on the N₂ cylinder, bleed down regulator, and remove sampling tubing. Shut off span gas flow but leave cylinder valve open, as the TVA will next be calibrated using this gas. Turn off O₂/CO₂/CO analyzer by pressing the red key to conserve batteries. Remove tubing from regulator but leave attached to analyzer for sampling of compressor air.

5. TVA (fill out calibration information on the data sheet).

- a. Disconnect TVA from charger.
- b. Attach the TVA probe and electrical cable to the TVA. Ensure that the normal sampler (the smaller of the two types of samplers) is installed on the end of the probe.
- c. Check the pressure of the H₂ cylinder located on the left-hand side of the analyzer. Maximum pressure is 2,300 psig. The TVA uses ~150 psi of H₂ per hour. If pressure is low, replace H₂ cylinder with a fully charged cylinder.
- d. Turn the red H₂ supply valve located on the back side of the analyzer to "ON." Press the "ON" key and wait for the beep and "Main Menu" to appear on the display.
- e. Battery check. Batteries should be checked without the charger connected. At the Main Menu, select "Info." Press the down arrow to cycle through "Info" and check the battery. Fully charged batteries should read approximately eight volts. If the battery is less than seven volts, batteries should be charged. The instrument may be used with line power via the battery charger if necessary.
- f. Ensure that two to three minutes have elapsed since turning on the H₂ before proceeding to the next step, to allow the H₂ to fill the FID chamber.
- g. Press the "EXIT" key and then select "Run." The pump will then start and after approximately 10 seconds, autoignition will occur. If the pump does not

come on, it will need to be fixed. If the FID lights, the TVA will automatically go into the run mode with the FID reading displayed. The FID value should be varying and other than zero.

- h. If the FID did not light, a Flameout warning will display. Wait two to three minutes more and try again to ignite by pressing the "EXIT" key twice and then selecting "Run" again.
- i. If the FID ignites but then goes out anytime afterward, a Flameout warning will appear.
- j. Wait 30 minutes for instrument equilibration.
- k. Ensure that the calibration mode is set to automatic so that the TVA will automatically accept the calibration (rather than require a prompt to accept). To do this, first press the "EXIT" key to reach the Main Menu. Then select "Setup," then "Calib," then "Cfg" (configuration). Press the up arrow twice. Then select "Accept mode" and "Auto." Press "EXIT" to return to the Calibration Menu.
- l. Check the calibration gas concentration currently in TVA memory by first selecting "Span Conc." If the concentration of the isobutylene calibration gas (e.g., 10.50 ppm) is correctly displayed, press "EXIT."
- m. If the calibration concentration is not correct, press "ENTER," enter the concentration of the isobutylene calibration gas (e.g., 10.5 ppm), and again press the "ENTER" key.
- n. To zero the FID detector, select "Zero" and then "ENTER."
- o. Install the charcoal filter sampler (the larger of the two types of samplers) loaded with fresh charcoal on the end of the probe to filter the calibration gas and provide zero gas to the TVA. The zero N₂ used with the O₂/CO₂/CO analyzer above cannot be used to zero the TVA as the FID requires O₂ in the sample gas to support the flame.
- p. Open the delivery valve on the regulator and adjust gas flow so it is just audible as it exits the syringe barrel.
- q. Insert the TVA probe halfway into the syringe barrel. Wait 30 seconds and then press "ENTER." Wait approximately 15 seconds until the menu returns. Shut off calibration gas flow. The FID detector has now been zeroed.
- r. Remove the charcoal filter and replace normal sampler to prepare to span the TVA.

- s. To span the FID, select "Span" and then "ENTER."
- t. Adjust calibration gas flow again so it is just audible and insert probe into syringe barrel. Wait 30 seconds and press "ENTER." Wait approximately 15 seconds until menu returns. The FID has now been spanned.
- u. Press "EXIT" key twice and then select "Run."
- v. Close valve on the calibration gas cylinder, bleed down regulator, and remove regulator.
- w. Leave TVA in run mode and with normal sampler in probe. FID values are now read off the TVA display or the sampling probe.

O₂/CO₂/CO ANALYZER "COLD START" OPTION (WHEN THERE IS A SUSPECTED PROBLEM)

1. When there is a suspected problem with the O₂/CO₂/CO analyzer, the cold start option reboots the software and often restores the analyzer to normal operation. Problems often cured with a cold start include: 1) analyzer not responsive to key strokes and 2) analyzer display indicates that it will not accept the calibration value that is entered.

2. Cold start procedure:

- a. Hold the "3" key and turn the analyzer on by pressing the red key.
- b. The display should show "Cold Start." Press "1" to continue.

FACTORY REPAIR/MAINTENANCE SERVICE - O₂/CO₂/CO ANALYZER

1. O₂/CO₂/CO analyzers are kept under service contract with the manufacturer (Geotechnical Instruments). This contract should be renewed annually at the start of every fiscal year.

2. This service contract provides for the following:

- a. One planned yearly service by the manufacturer that includes re-calibration, battery replacement if needed, any necessary repairs, and general checkout of instrument function.
- b. Free software upgrades.

- c. Hardware modifications due to product improvement providing the modification is compatible with the current unit: no extra charge.
- d. Repair of any malfunction in the field: no extra charge. This excludes excessive physical damage.
- e. Shipping charges to and from Geotechnical Instruments, U.K. and the Fleet sampling sites. Geotechnical Instruments will provide shipping labels.

3. In the event of a problem with a CO₂ analyzer that cannot be corrected by the above suggested steps, contact Geotechnical Instruments via telephone. In the U.S.A., dial **011-44-1926-338111**. Outside of the U.S.A. dial: commercial +44(0)1926 338111.

FACTORY REPAIR/MAINTENANCE SERVICE – TVA

1. TVAs can be purchased with an extended service plan of up to four years which, when added to the standard one-year warranty, should cover the expected lifetime of the instrument. Recommended spare parts that are desirable to have on hand include as described earlier: battery pack, filters, charcoal, and detector parts. The service plan includes one yearly service and routine inspection by the manufacturer.

2. In the event of a problem with a TVA that cannot be corrected using the spare parts on hand and the guidance of the manual, contact the manufacturer via telephone at toll free: **1-888-FOXBORO**.

APPENDIX G

OPERATING PROCEDURES: AEROSOL MONITOR

BATTERIES

The Personal/DataRam aerosol monitor is normally operated from its self-contained non-rechargeable 9V alkaline battery which is replaced when necessary. A fresh battery should provide a minimum of 12 hours of continuous operation. However, the DataRAM can also be operated while attached to its line voltage power supply.

BATTERY CHECK/ZEROING

1. Prior to each day's use, the battery should be checked and the DataRAM zeroed.
2. Battery check. Turn the DataRAM on by pressing the "ON/OFF" button. Press the "NEXT" button five times until the battery status is displayed. If the remaining battery charge is < 60%, replace battery after turning DataRAM off.
3. Ensure that the Flow-Adaptor is installed on the DataRAM (Fig. 6).
4. Press the "ON/OFF" button and then "ENTER" to turn off.
5. In a reasonably clean area, connect the end of the hand pump to the inlet port (front) of the DataRAM. Start pumping the hand pump.
6. Turn on the DataRAM by pressing the "ON/OFF" button. Press "ENTER" to start zeroing; continue pumping the hand pump. The screen should display the following: "ZEROING V1.41."
7. After approximately one minute, the display should read: "CALIBRATION: OK." Fill out the calibration information on the data sheet.
8. If the display reads: "BACKGROUND HIGH," accurate readings can still be taken. However, the sensor head should be cleaned at the earliest opportunity as described in the DataRAM manual. If this fails to correct the problem, the manufacturer should be contacted to determine whether the DataRAM needs to be sent back to the factory for cleaning.
9. Remove the hand pump, install a short (<1 foot) length of Tygon tubing to the inlet port of the DataRAM, and attach the flow monitor to the outlet (see Fig. 6).
10. Press the "NEXT" key and then "ENTER" to start measuring.

11. The DataRAM is now measuring the aerosol concentration in the gas in its sensing chamber. The CONC value is the instantaneous concentration which will be used for diver's air. The TWA value is the Time Weighted Average concentration.

12. Leave DataRAM on.

FACTORY REPAIR/MAINTENANCE SERVICE

1. No special service contract for the DataRAM has been defined for the U.S. Navy. In the event of a problem with the DataRAM that cannot be corrected by guidance of the manual, contact the manufacturer, MIE, Inc. at **1-781-275-1919**.

2. In the absence of any problems with the DataRAM, the manufacturer recommends that the unit be returned to the factory once every two years for routine checkout, cleaning, and calibration.

APPENDIX H

HIGH PRESSURE AIR COLLECTION PROCEDURE (For troubleshooting air purity problems)

1. Gas samples should be obtained in high pressure cylinders to diagnose suspected gas purity problems including unexplained high readings by any of the three portable analyzers. These samples will be analyzed in detail, as described below, by an on-shore analytical laboratory.
2. High-pressure gas cylinders (previously evacuated) and connection hardware including a high-purity, high-pressure regulator should be available from the laboratory that will perform the analysis. These items are described in **Appendix D**.
3. Duplicate compressor air samples should be taken at the same site(s) where the gas was sampled with the three portable analyzers. Extreme care must be taken not to expose sample cylinders to more than their pressure rating.
4. Record sampling information on the data sheet.
5. Before proceeding, ensure that adequate purging of the sampling lines has been performed as described in the main section on screening procedures.
6. After purging, attach the high-pressure regulator (configured to attach to the sample cylinders) to the sample site to allow filling cylinders with ~200 psig of bank air.
7. Slowly open the valve(s) on the compressor line to pressurize the regulator. Then pressure cycle the regulator three times to remove all ambient air.
8. Dial in sufficient delivery pressure with the regulator to purge at a highly audible rate for five minutes.
9. Check cylinder valves just prior to use to ensure they have not become opened during transit. Do not use any cylinders that have loose valves and are suspected of having lost vacuum.

Extreme care must be taken not to expose cylinders to more than their working pressure rating.

10. The laboratory that will perform the analysis may provide instructions for gas sampling using their cylinders. However, in the event that instructions are not provided, the following procedures may work, or may be adapted to work, with their cylinders:
 - a. Attach the first cylinder to the regulator as gas is flowing. The cylinder connection is made wrench-tight and the downstream valve on the regulator is

opened fully. The cylinder valve closest to the regulator is then fully opened slowly (e.g., it should take approximately 10 seconds for each full turn (360 degree rotation) of the valve handle).

- b. After the cylinder valve has been opened, dial in 200 psig, and allow two minutes for cylinder to equilibrate with the upstream pressure. The cylinder valve is then closed tightly. Leave gas flow on as the first cylinder is disconnected from the regulator and the second cylinder is attached and filled in similar fashion. Flow can be throttled back slightly during cylinder removal and attachment.

11. After the second cylinder has been disconnected from the regulator, shut off flow at the sample site and disconnect hardware.

12. Record on a label or tag attached to each cylinder:

- a. cylinder #
- b. sample site and sample conditions
- c. person doing sampling
- d. date
- e. time

and complete the data sheet.

13. Cylinders must be declared as hazardous cargo prior to air transport (e.g., Federal Express) to the laboratory.

14. The procedures for analysis of the cylinder gas are defined below. These procedures, or equivalent procedures that meet or exceed the specifications listed here, must be followed.

- a. Gas samples are screened initially using Gas Chromatography (GC) with flame ionization detection (FID) configured to allow detection of a wide range of volatile organic compounds. In order to ensure the detection of the most probable contaminants, GC should be configured for detection of the following:
 - ◆ Highly volatile light compounds such as Freon 11, Freon 12, Freon 114, and Freon 113,
 - ◆ Less volatile heavier compounds such as Benzene, Toluene, Xylenes, and Trimethyl Benzenes,
 - ◆ Polar compounds such as Methyl Alcohol and Isopropyl Alcohol.

More than one GC configuration (i.e., column type, temperature profile, carrier gas flow) may be necessary to analyze for this range of compounds. The specific chemicals listed above are representative of the three classes of volatile

organic compounds that may be present in compressor air; they are not a "list" of all the individual compounds that may be expected. The ability to detect the range of organic species defined by the above chemicals using GC will provide effective organic screening of diving air.

- b. GC must be able to detect 0.5 ppm Freon 113 in gas. Any GC peak exceeding the equivalent of 1 ppm Freon 113 will be positively identified by direct injection of chemical species into the GC and/or by Gas Chromatography/Mass Spectrometry (GC/MS).
- c. GC equipped with methanization/FID is used to measure CO₂ and CO due to the limited amount of sample gas provided. Such a system must be able to detect 5 ppm CO.
- d. GC with thermal conductivity detection is used for analysis of O₂.
- e. Results will be reported as follows for each sample:
 - ♦ Volatile organic compounds (> 1 ppm): identification and quantitation to the nearest 0.1 ppm (relative to Freon 113, or relative to a standard of the actual chemical if available),
 - ♦ CO₂ reported to the nearest 5 ppm, CO (> 5 ppm) to the nearest ppm, and O₂ to the nearest 0.1%.