United States Environmental Protection Agency

Standard Test Procedures for Evaluating Leak Detection Methods: Vapor-phase Out-of-tank Product Detectors

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KWA

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United States
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Solid Waste And
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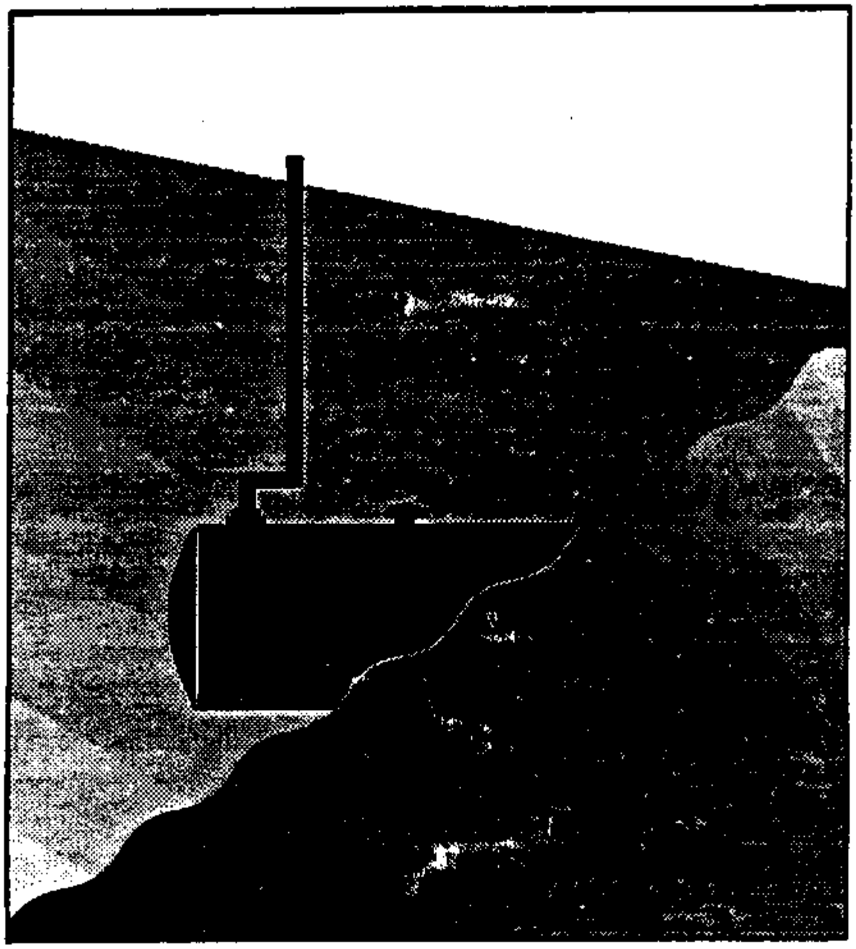

Standard Test Procedures for Evaluating Leak Detection Methods: Vapor-Phase Out-of-Tank Product Detectors

Final Report

U.S. Environmental Protection Agency
Office of Research and Development

March 1990

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FOREWORD

HOW TO DEMONSTRATE THAT LEAK DETECTION METHODS MEET EPA'S PERFORMANCE STANDARDS

The Environmental Protection Agency's (EPA's) regulations for underground storage tanks require owners and operators to check for leaks on a routine basis using one of a number of detection methods (40 CFR Part 280, Subpart D). In order to ensure the effectiveness of these methods, EPA set minimum performance standards for equipment used to comply with the regulations. For example, after December 22, 1990, all tank tightness testing methods must be capable of detecting a 0.10 gallon per hour leak rate with a probability of detection of at least 95% and a probability of false alarm of no more than 5%. It is up to tank owners and operators to select a method of leak detection that has been shown to meet the relevant performance standard.

Deciding whether a method meets the standards has not been easy, however. Until recently, manufacturers of leak detection methods have tested their equipment using a wide variety of approaches, some more rigorous than others. Tank owners and operators have been generally unable to sort through the conflicting sales claims that are made based on the results of these evaluations. To help protect consumers, some state agencies have developed mechanisms for approving leak detection methods. These approval procedures vary from state to state, making it their method nationwide. The purpose of this policy is to describe the ways that owners and operators can check that the leak detection equipment or service they purchase meets the federal regulatory requirements. States may have additional requirements for approving the use of leak detection methods.

EPA will not test, certify, or approve specific brands of commercial leak detection equipment. The large number of commercially available leak detection methods makes it impossible for the Agency to test all the equipment or to review all the performance claims. Instead, the Agency is describing how equipment should be tested to prove that it meets the standards. Conducting this testing is left up to equipment manufacturers in conjunction with third-party testing organizations. The manufacturers will then provide a copy of the report showing that the method meets EPA's performance standards. This information should be provided to customers or regulators as requested. Tank owners and operators should keep the evaluation results on file to satisfy EPA's record keeping requirements.

EPA recognizes three distinct ways to prove that a particular brand of leak detection equipment meets the federal performance standards:

1. Evaluate the method using EPA's standard test procedures for leak detection equipment;

2. Evaluate the method using a national voluntary consensus code or standard developed by an nationally recognized association or independent third-party testing laboratory; or,

3. Evaluate the method using a procedure deemed equivalent to an EPA procedure by a nationally recognized association or independent third-party testing laboratory.

The manufacturer of the leak detection method should prove that the method meets the regulatory performance standards using one of these three approaches. For regulatory enforcement purposes, each of the approaches is equally satisfactory. The following sections describe the ways to prove performance in more detail.

EPA Standard Test Procedures

EPA has developed a series of standard test procedures that cover most of the methods commonly used for underground storage tank leak detection. These include:

1. "Standard Test Procedures for Evaluating Leak Detection Methods: Volumetric Tank Tightness Testing Methods"

2. "Standard Test Procedures for Evaluating Leak Detection Methods: Nonvolumetric Tank Tightness Testing Methods"

3. "Standard Test Procedures for Evaluating Leak Detection Methods: Automatic Tank Gauging Systems"

4. "Standard Test Procedures for Evaluating Leak Detection Methods" Statistical Inventory Reconciliation Methods"

5. "Standard Test Procedures for Evaluating Leak Detection Methods: Vapor-Phase Out-of-Tank Product Detectors"

6. "Standard Test Procedures for Evaluating Leak Detection Methods: Liquid-Phase Out-of-Tank Product Detectors"

7. "Standard Test Procedures for Evaluating Leak Detection Methods: Pipeline Leak Detection Systems"

Each test procedure provides an explanation of how to conduct the test, how to perform the required calculations, and how to report the results. The results from each standard test procedure provide the information needed by tank owners and operators to determine if the method meets the regulatory requirements.

The EPA standard test procedures may be conducted directly by equipment manufacturers or may be conducted by an independent third party under contract to the manufacturer. However, both state agencies and tank owners typically prefer that the evaluation be carried out by an independent third-party in order to prove compliance with the regulations. Independent third-parties may include consulting firms, test laboratories, not-for-profit research organizations, or educational institutions with no organizational conflict of interest. In general, EPA believes that evaluations are more likely to be fair and objective the greater the independence of the evaluating organization.

National Consensus Code or Standard

A second way for a manufacturer to prove the performance of leak detection equipment is to evaluate the system following a national voluntary consensus code or standard developed by a nationally recognized association (e.g., ASTM, ASME, ANSI, etc.). Throughout the technical regulations for underground storage tanks, EPA has relied on national voluntary consensus codes to help tank owners decide which brands of equipment are acceptable. Although no such code presently exists for evaluating leak detection equipment, one is under consideration by the ASTM D-34 subcommittee. The Agency will accept the results of evaluations conducted following this or similar codes as soon as they have been adopted. Guidelines for developing these standards may be found in the U.S. Department of Commerce "Procedures for the Development of Voluntary Product Standards" (FR, Vol. 51, No. 118, June 20, 1986) and OMB Circular No. A-119.

Alternative Test Procedures Deemed Equivalent to EPA's

In some cases, a specific leak detection method may not be adequately covered by EPA standard test procedures or a national voluntary consensus code, or the manufacturer may have access to data that makes it easier to evaluate the system another way. Manufacturers who wish to have their equipment tested according to a different plan (or who have already done so) must have that plan developed or reviewed by a nationally recognized association or independent third-party testing laboratory (e.g. Factory Mutual, National Sanitation Foundation, Underwriters Laboratory, etc.). The results should include an accreditation by the association or laboratory that the conditions under which the test was conducted were at least as rigorous as the EPA standard test procedure. In general this will require the following:

1. The evaluation tests the system both under the no-leak condition and an induced-leak condition with an induced leak rate as close as possible to (or smaller than) the performance standard. In the case of tank testing, for example, this will mean testing under both 0.0 gallon per hour and 0.10 gallon per hour leak rates. In the case of

ground-water monitoring, this will mean testing with 0.0 and 0.125 inch of free product.

2. The evaluation should test the system under at least as many different environmental conditions as the corresponding EPA test procedure.

3. The conditions under which the system is evaluated should be at least as rigorous as the conditions specified in the corresponding EPA test procedure. For example, in the case of volumetric tank tightness testing, the test should include a temperature difference between the delivered product and that already present in the tank, as well as the deformation caused by filling the tank prior to testing.

4. The evaluation results must contain the same information and should be reported following the same general format as the EPA standard results sheet.

5. The evaluation of the leak detection method must include physical testing of a full-sized version of the leak detection equipment, and a full disclosure must be made of the experimental conditions under which (1) the evaluation was performed, and (2) the method was recommended for use. An evaluation based solely on theory or calculation is not sufficient.

ACKNOWLEDGMENTS

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CONTENTS

Designation: X 0001

Standard Test Method for
ACCURACY AND RESPONSE TIME FOR VAPOR-PHASE OUT-OF-TANK PETROLEUM DETECTORS

1. Scope

1.1 This test method covers determination of accuracy and response time of vapor-phase out-of-tank petroleum hydrocarbon leak detectors.

1.2 This method is applicable to only the components associated with detection of vapor-phase petroleum releases for detection systems utilizing multiple operating principles.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards*:

E 1 Standard Specification for ASTM Thermometers

E 456 Standard Terminology Relating to Statistics

3. Terminology

3.1 *Definitions* -- For formal definitions of statistical terms, see Terminology E 456.

3.2. Descriptions of Terms Specific to This Method

3.2.1 *activated*—refers to the state of a qualitative detector's response when indicating the presence of hydrocarbon vapors.

3.2.2 *detection time*—elapsed time from a detector's first contact with test gas to an output that is 95% of full scale or activated.

3.2.3 *fall time*—elapsed time after a detector is removed from test hydrocarbon vapor until its output returns to within 5% of its original baseline level or there is no detectable signal output.

3.2.4 *non-activated*—refers to the state of a qualitative detector's response when indicating that no hydrocarbon vapors are detected.

3.2.5 *probe*—component of a detection system that must come into contact with petroleum gas before the gas can be detected.

3.2.6 *qualitative responses*—type of detector response that indicates only the presence or absence of hydrocarbon vapors without determining the specific hydrocarbon concentration.

3.2.7 *quantitative responses*—type of detector response that quantitates the concentration of the hydrocarbon vapor present.

3.2.8 *relative accuracy*—absolute mean difference between a group of measured values and the true value, plus the 2.5% error confidence coefficient, divided by the true value. Relative accuracy is a measure of the maximum expected bias (without regard to sign) for a series of measurements.

3.2.9 *responses*—detector's indication of the presence of petroleum hydrocarbon vapors. Responses can be qualitative or quantitative.

3.2.10 *response time*—general term that refers to the more specific terms detection time and fall time.

4. Summary of Test Method

4.1 Detector probes are subjected to test atmospheres of either benzene or 2-methylbutane inside a sealed test chamber. Detectors are tested five times at each concentration of both test gases. Test gas concentrations are nominally 50 parts per million by volume (ppmv), 250 ppmv, 500 ppmv, and 1000 ppmv. Detector response is monitored for up to 24 hours.

5. Significance and Use

5.1 For vapor-phase petroleum hydrocarbon detectors, accuracy is a measure of how well the detector's output compares to a known concentration of hydrocarbon vapors. Accuracy measurements provide a means for estimating the reliability of a detector.

5.2 Precision is the degree of agreement of repeated measurements of the same parameter. Precision estimates reflect random error and are not affected by bias. In this method, precision is expressed in terms of the percent coefficient of variation.

5.3 In addition to these primary performance characteristics, procedures for estimating the magnitude and direction of measurement bias are presented. Bias is the systematic error inherent in a method, which may be positive or negative. In this method, bias is expressed as the signed percent difference between the average measured value for a series of tests and the true value.

5.4 Response time is the general term that refers to a combination of the more specific terms detection time and fall time. Detection time is the elapsed time from a detector's first contact with a given concentration of petroleum vapors to 95% of its full-scale signal output or to an activated response. Fall time is the elapsed time after the detector is removed from contact with petroleum hydrocarbon vapors until the detector output returns to a stable baseline response.

5.5 Results obtained using this method will permit the most advantageous use of a detector. Weaknesses as well as strengths of the instrument should become apparent. It is not the interest of this method to compare similar detectors of different manufacture, but to enable the user to choose a suitable detector.

6. Interferences

6.1 Conditions that can cause interferences with this method include temperature changes, high temperatures, excessive test apparatus volumes, and leaks in the test apparatus. To avoid these conditions, tests should be conducted at constant (±3°C) normal laboratory temperatures with a leak-tested test apparatus.

6.2 Cross contamination (e.g., memory effects from residual test atmospheres) may be a major cause of inaccurate data. To minimize this potential problem, avoid using rubber or plastic parts for components of the test apparatus that contact test gases and purge the testing system as described in Section 10.

6.3 Fluctuations of the test chamber internal pressure may significantly affect the detector's response. Maintain a constant internal chamber pressure (±0.2 inches of water relative to ambient pressure) while the probe is exposed to the test atmosphere by maintaining a constant inlet and exit test atmosphere flow rate. If the pressure varies outside the acceptance limits, repeat the tests within the accepted pressure fluctuation limits.

7. Apparatus

7.1 *Test apparatus*—The test apparatus, as depicted in Figure 1, shall be constructed from materials that are inert with respect to test gases. The test apparatus consists of compressed gas cylinders, pressure regulators, tubing, valves, tubing connectors, rotameters, test chamber, thermocouple, and manometer.

7.1.1 *Compressed gas cylinders*—Test gases are supplied in standard compressed gas cylinders having Compressed Gas Association (CGA) fittings compatible with regulator fittings.

Note 1—Dilute hydrocarbon test gases and ultrahigh-purity air are commonly supplied in compressed gas cylinders having CGA 590 fittings.

7.1.2 *Pressure regulators*—A dual stage regulator with a fitting compatible with the test gas cylinders is needed. The regulator(s) shall have a range of at least 0 psi to 15 psi and have a diaphragm made of stainless steel.

7.1.3 *Tubing*—Sufficient tubing to link all test apparatus components is needed. The tubing shall be free from contaminants and have an internal diameter of at least 1/8 inch. The tubing shall be made of a material that is inert with respect to test gases.

7.1.4 *Tubing connectors*—Various compression-type tubing fittings are needed to make test apparatus connections. These fittings shall be free from contaminants, inert with respect to test gases, and of a material that is compatible with the tubing. These fittings can include, but are not limited to, tees, cross tees, reducers, and elbows. A thermocouple compression fitting is also needed.

7.1.5 *Rotameters*—Two rotameters, each having a flow range bracketing the required flow rates for the detector, are needed to measure test chamber inlet and outlet vapor rates. A third rotameter is required for detectors that aspirate gas samples.

7.1.6 *Valves*—A shut-off valve and a flow controlling needle valve are needed.

Note 2—Gas shut off can be accomplished using a regulator and the integral shut-off valve commonly included with compressed gas cylinders. Many rotameters include an integral flow controlling needle valve.

7.1.7 *Thermocouple*—A thermocouple and temperature readout, or equivalent, that responds from 0°C to 40°C and is accurate to within 1°C over this range is needed.

7.1.8 *Manometer*—A relative pressure manometer is required to monitor the test chamber's internal pressure. The manometer must have a working range of at least 0-10 inches of water (0 to 20 mm Hg) with an accuracy of ±5%. The manometer should be scaled using at least 0.2 inches of water (0.4 mm Hg) subdivisions.

7.1.9 *Test chamber*—The test chamber should be gas-tight and made from materials that are inert with respect to test gases. Figure 2 contains a test chamber schematic representation. The test chamber must have fittings to allow connection to the detector probe, a manometer, and a thermocouple. Test chamber volumes should be kept as small as possible without interfering with detector operation. The chamber must also have an inlet and outlet for flow of test atmospheres. Diagrams of suitable test chambers are presented in Appendix A.

7.2 *Timer*—A timer that is accurate and precise to at least one second per 10 minutes is required. Alternatively, a chart recorder or other data acquisition system may be used. If a chart recorder or other data acquisition system is to be used, the timer is not required. If used, the recorder or data acquisition system timing must be accurate and precise to at least one second per 10 minutes.

7.3 *Electronic recorder*—A chart recorder or other electronic data acquisition system may be used if it is compatible with the specific detector that is being evaluated. The output of the data recorder should be accurate and precise to ±2% over the range of output from a quantitative detector. A data recorder used with a qualitative detector must unambiguously identify activated and inactivated states.

7.4 *Thermometer*—ASTM Solvents Distillation Thermometer having a range from -2°C to 52°C and conforming to the requirements for Thermometer 37C as prescribed in Specification E 1.

7.5 *Bubble meter*—Use NBS-traceable soap bubble flow meters to calibrate rotameters. The bubble meters must have a working range that brackets the range of all rotameters.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents for the American Chemical Society where such specifications are available.[1] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of determination.

8.2 *Test Gases*—Use factory-mixed benzene and 2-methylbutane gas standards in conventional gas cylinders as test chamber atmosphere sources.

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[1] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemicals Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., In., New York, NY, and the "United States Pharmacopeia."

Make-up gas for all standards shall be air. All gas standards must be certified accurate (±2%). The gas standards required are listed in Table 1. (Danger—*Gases under high pressure. May be harmful if inhaled. See Annex A1.1 and Annex A1.2.)*

8.3 *Ultrahigh-Purity Air*—The ultrahigh-purity air standard must have less than 0.5 ppmv total hydrocarbon content. (Caution—*Gas under high pressure. See Annex A1.3.*)

9. Calibration and Standardization

9.1 *Chart Recorder or Other Data Recording System*—If used, a chart recorder or other data recording system should be calibrated along with the detector. The data recording system should be calibrated according to instructions from its manufacturer and the detector manufacturer. Also, any recording device should be compatible with the detector being investigated. Consult specifications from the manufacturers of the recording device and the detector.

9.2 *Detector*—Because of wide design variability among different petroleum detectors, it is impossible to give complete calibration instructions for all possible detector designs. Calibrate all detectors according to manufacturer instructions.

9.3 *Rotameters*—Calibrate each rotameter prior to initiating test procedures, once a year thereafter, and after any internal contamination (e.g., dirt, moisture) during testing. Instructions accompanying the NBS-traceable bubble meters should be followed. The rotameters are to be calibrated at a minimum of five points ("multipoints") within the working range. Flow readings should be made from the middle of the ball float. All readings should be made from the upper float on dual-float rotameters until it is off scale. Once the upper float is off scale, readings should be made from the lower float.

9.3.1 *Assembly*--Remove the rotameter from the test system, if assembled, and connect the rotameter in series between a controllable compressed air source and an NBS-traceable bubble meter. Use a bubble meter cylinder that will allow a flow measurement over a period of 15 to 45 seconds.

9.3.2 *First calibration point*--Bring the gas flow rate to the lowest calibration flow for testing. Let the system run at this setting until the rotameter is steady. If the flow rate is not within the first level range, adjust the flow rate until it is within that range and wait for a steady-state value. Record the steady-state flow meter value that is within the first flow rate level. Measure the flow rate with the bubble meter and stopwatch according to the NBS bubble meter instructions. Record each reference flow rate.

9.3.3 *Remaining calibration points*--Record steady-state settings for triplicate runs as described in Section 9.3.2 for at least four more flow rates throughout the rotameter range.

9.4 *Thermocouple*--Perform side-by-side multipoint calibrations for each thermocouple used in the test procedure in a 1-L glass beaker filled with water. The reference thermometer should be an ASTM Solvents Distillation thermometer having a range from -2°C to 52°C and conforming to the requirements for Thermometer 37C as prescribed in Specification E 1. The levels tested are low (room temperature - 10°C), room temperature, and high (room temperature + 10°C).

9.4.1 Insert both the thermocouple and reference thermometer into the beaker of water and add small quantities of ice. Allow the ice to melt and the temperature to stabilize. Continue adding ice until a steady-state reading (±0.5°C over two minutes) of room temperature - 10°C (±2°C) occurs.

9.4.2 Repeat this procedure using room temperature water (15°C to 30°C), and room temperature + 10°C (±2°C) water. If the temperature difference is more than 1°C, either repeat the test with the same thermocouple or replace the thermocouple and repeat the test until it is acceptable.

9.4.3 Perform thermocouple calibration at the onset of testing and at least once a year.

10. Conditioning

10.1 *Before each test*—Purge the test chamber for at least three minutes with ultrahigh-purity air at 0.2 L/min.

11. Procedure

11.1 *Test Series*—The detector should be tested a minimum of five times for each combination of test gas and concentration listed in Table 1.

11.1.1 Perform tests in a random order such that variables of test gas and hydrocarbon layer concentration are isolated.

11.2 *Assembly*—Assemble the detection system as described by the manufacturer. Insert the detector probe into the test chamber. The seal between the probe and the test chamber should be gas-tight.

11.2.1 Connect the detector output to a chart recorder or other data acquisition system if one is being used. All connections should be in compliance with specifications from the manufacturers of the detector and the data recording system.

11.3 Calibration—Calibrate the detector if necessary. Many detectors do not require any calibration. Perform calibrations, if necessary, according to manufacturer recommendations. Calibration may need to occur before mounting in the test container. If a data recording system is being used, it should be calibrated with the detector. Calibrate the data acquisition system according to manufacturer instructions.

11.4 *Background*—Supply ultrahigh-purity air to the test chamber at a rate that is 0.2 L/min. greater than the detector's aspiration rate.

Note 3—Many detectors are passive and do not aspirate gas samples. Use total test gas flow rates of 0.2 L/min for these detectors.

Monitor the detector's response every 15 seconds until a steady-state reading (±2% change of full scale over one minute) is achieved or four minutes has elapsed, whichever is longer. Monitor the temperature inside the test chamber during background testing.

11.5 *Test Atmosphere Response*—Introduce the appropriate test gas to the test chamber at a sufficient rate to produce a 0.2 L/min test system vent flow rate. Monitor the temperature inside the test chamber during testing.

11.5.1 Monitor quantitative detectors until a steady-state (±2% of full scale over 1 minute) response occurs or for 24 hours, whichever is shorter. Monitor qualitative detectors for a positive response ("activated") for up to 24 hours.

11.6 *Detection Time*—If the detector gives a positive response within 24 hours, the elapsed time between when the test atmosphere was introduced into the chamber and when the detector responded is the detection time. The nature of a response is dependent on whether a detector gives quantitative output or qualitative output.

11.6.1 The period for detection time of quantitative detectors is from introduction of the test atmosphere into the test chamber to the time the detector reaches 95% of its final stable output. Calculate the 95% of final stable output level from the following equation:

$$\text{High Level output, ppmv} = BL + (HL - BL) \times 0.95 \qquad (7)$$

where:

BL = stable baseline output, ppmv; and

HL = stable high level output,ppmv.

11.6.2 A positive response for qualitative detectors occurs when the detector output goes from an inactivated state to an activated state.

11.6.3 If the detector gives a response within 24 hours, report the elapsed time between when the detector probe was added to the container with test gas and when the detector responded as the detection time.

11.6.4 Detectors with lower detection limits above a particular test gas concentration do not need to be tested for the full 24-hour period. They should be tested for at least five times their maximum expected detection time.

11.7 *Fall Time*—Close the valves for the test gas and introduce ultrahigh-purity air at a sufficient rate to produce a 0.2 L/min test system vent flow rate. Allow the probe to remain in the ultrahigh-purity air for at least 4 minutes or until a steady-state background response is achieved (±2% of full scale over 1 minute).

Note 3—Fall time is not applicable to some detectors such as qualitative detectors that did not activate in response to addition of gas to the test container.

11.7.1 Start the timer or mark the beginning of the fall time test on the recording system when ultrahigh-purity air is introduced into the test chamber.

11.7.2 Monitor the detector output for fall time response. The nature of a fall time response is dependent on whether a detector gives quantitative or qualitative output. Fall time response for a quantitative detector is when the detector output returns to within 5% of its original stable baseline level. Calculate the 5% stable baseline level according to the following equation:

$$\text{5\% Stable baseline output, ppmv} = BL + (HL - BL) \times 0.05 \qquad (2)$$

where:

BL = stable baseline output, ppmv; and

HL = stable high level output, ppmv.

Fall time response for a qualitative output detector is when the detector output goes from an activated state to an inactivated state.

11.7.3 Continue fall time monitoring for up to 24 hours.

12. Calculation

12.1 *Relative Percent Difference*—Calculate relative percent difference as follows:

$$\text{Relative percent difference, \%} = 200 \times [(V_1 - V_2)/(V_1 + V_2)] \qquad (3)$$

where:

V_1 = larger value; and

V_2 = smaller value.

12.2 *Coefficient of Variation*—Calculate the coefficient of variation as follows:

$$\text{Coefficient of variation, \%} = (s/\bar{X}) \times 100 \qquad (4)$$

where:

s = standard deviation of n values (n-1 degrees of freedom),ppmv; and

$\bar{X}$ = mean of n values, ppmv.

12.3 *Accuracy*—Calculation for accuracy is dependent on the type of output a detector produces.

12.3.1 *Quantitative detectors*—Accuracy for quantitative detectors is a function of systematic error (bias) and random error (precision). Bias and precision calculations are given in Equations 9 and 3, respectively. Calculate relative accuracy of a set of data as follows:

$$\text{Relative accuracy, \%} = (|\bar{d}| + |cc|) / V_r \times 100 \qquad (5)$$

where:

V_r = reference (theoretical) value;

$\bar{d}$ = arithmetic mean of the difference of a data set, Equation 6; and

cc = 2.5% error confidence coefficient (one tailed, Equation 7).

12.3.2 *Mean difference*—Calculate the arithmetic mean of the difference of a data set as follows:

$$\text{Mean difference, ppmv} = 1/n \sum_{i=1}^{n} d_i \qquad (6)$$

where:

d_i = measured response - theoretical response.

12.3.3 *Confidence coefficient*—Calculate the one-tailed 2.5% confidence coefficient (cc) as follows:

$$\text{Confidence coefficient, ppmv} = t_{0.975} \times s/\sqrt{n} \qquad (7)$$

where:

s = the standard deviation (n-1) of the data set; and

$t_{0.975}$ = 2.5% t value = from Table 2; and

n = number of tests for a test gas at a particular concentration.

12.3.4 *Qualitative detectors*—Use the following formula to calculate the accuracy of qualitative detectors:

$$\text{Accuracy, \%} = 100 \times (r_p/n) \qquad (8)$$

where:

r_p = number of positive responses; and

n = number of tests for a particular test gas at a particular concentration.

12.4 *Bias*—Bias for quantitative detectors is calculated as follows:

$$\text{Bias, \%} = 100 \times [(\bar{V}_o - V_r)/V_r] \qquad (9)$$

where:

V_r = the reference (theoretical) value, ppmv; and

$\bar{V}_o$ = the average observed value, $1/n \sum_{i=1}^{n} V_i$;

where:

n = the number of tests with a particular test gas at a particular concentration; and

V_i = the individual response to test gas, ppmv;

12.5 *Detection Time*—Calculate detection time according to the following formula:

$$\text{Detection time} = T_2 - T_1 \qquad (10)$$

where:

T_1 = clock time when test gas was first added to test chamber; and

T_2 = clock time when detector output went from an inactivated state to an activated state for a qualitative-output detector or from a baseline reading to 95% of stable high level output for a quantitative detector.

12.6 *Fall Time*—Calculate fall time according to the following formula:

$$\text{Fall time} = T_2 - T_1 \tag{11}$$

where:

T_1 = clock time when ultrahigh-purity air was added to the test chamber; and

T_2 = clock time when detector output went from an activated state to an inactivated state for a qualitative detector or from a high level reading to within 5% of stable baseline level output for a quantitative-output detector.

13. Report

13.1 Use the form displayed in Figure 3 to report results. Report the following information:

13.1.1 *Detector type*—Report whether the detector was a quantitative or qualitative type.

13.1.2 *Accuracy*—For quantitative detectors, report relative accuracy for both test gases at every concentration (nominally 50, 250, 500, and 1000 ppmv) according to Equation 5. Report accuracy for qualitative detectors according to Equation 8.

13.1.3 *Precision*—Precision for quantitative detectors is defined as the percent coefficient of variation. Use Equation 4 to calculate coefficient of variation. For quantitative detectors, report precision as the percent coefficient of variation for both test gases at every concentration (nominally 50, 250, 500, and 1000 ppmv). Precision is not applicable to qualitative detectors, and shall be reported as "NA", not applicable, for these detectors.

13.1.4 *Bias*—Bias at a particular concentration for a test gas is the percent difference between the average detector output for a series of tests and the actual concentration of test gas, Equation 9. For quantitative detectors, report bias for both test gases at every concentration (nominally 50, 250, 500, and 1000 ppmv). Bias is not applicable to qualitative detectors, and shall be reported as "NA," not applicable, for these detectors.

13.1.5 *Detection Time*--Report detection time in the largest convenient units (seconds, minutes, or hours) for both test gases at every concentration (nominally 50, 250, 500, and 1000 ppmv). If detector response is immediate, report detection time as "<1 second." If the detector does not respond within 24 hours, report detection time as "No response in 24 hours."

13.1.6 *Fall Time*—Report fall time in the largest convenient units (seconds, minutes, or hours) for both test gases at every concentration (nominally 50, 250, 500, and 1000 ppmv). Fall time is not applicable to some detectors such as gas-soluble detectors and qualitative detectors that did not activate in response to addition of test gas to the chamber. For these detectors, report fall time as "NA," not applicable. Also, record the reason(s) why fall time determination was not applicable. If the detector does not respond within 24 hours, report fall time as "No response in 24 hours."

14. Precision and Bias

14.1 *Precision*—The precision of the procedure in Test Method X 0001 for measuring accuracy and response time for vapor-phase out-of-tank petroleum detectors is being determined.

14.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in Test Method X 0001 for measuring accuracy and response time for vapor-phase out-of-tank petroleum detectors, no statement on bias is being made.

TABLE 1. Test Atmospheres

Gas Compound	Concentrations (ppmv)
benzene	50 ±5, 250 ±10, 500 ±10, 1000 ±25
2-methylbutane	50 ±5, 250 ±10, 500 ±10, 1000 ±25

TABLE 2. 2.5% T Values[A]

n-1	$t_{0.975}$
0	12.706
1	4.303
2	3.182
3	2.776
4	2.571
5	2.447
6	2.365
7	2.306
8	2.262
9	2.228
10	2.201
11	2.179
12	2.160
13	2.145
14	2.131
15	2.120
16	2.110
17	2.101
18	2.093
19	2.086
20	2.080
21	2.074
22	2.069
23	2.064
24	2.060
25	2.056
26	2.052
27	2.048
28	2.045
29	2.042
30	2.021
40	2.000
60	1.980
120	1.960

[A] Taken from *CRC Standard Mathematical Tables*, 26th ed. CRC Press, Inc. Boca Raton, FL, 1981.

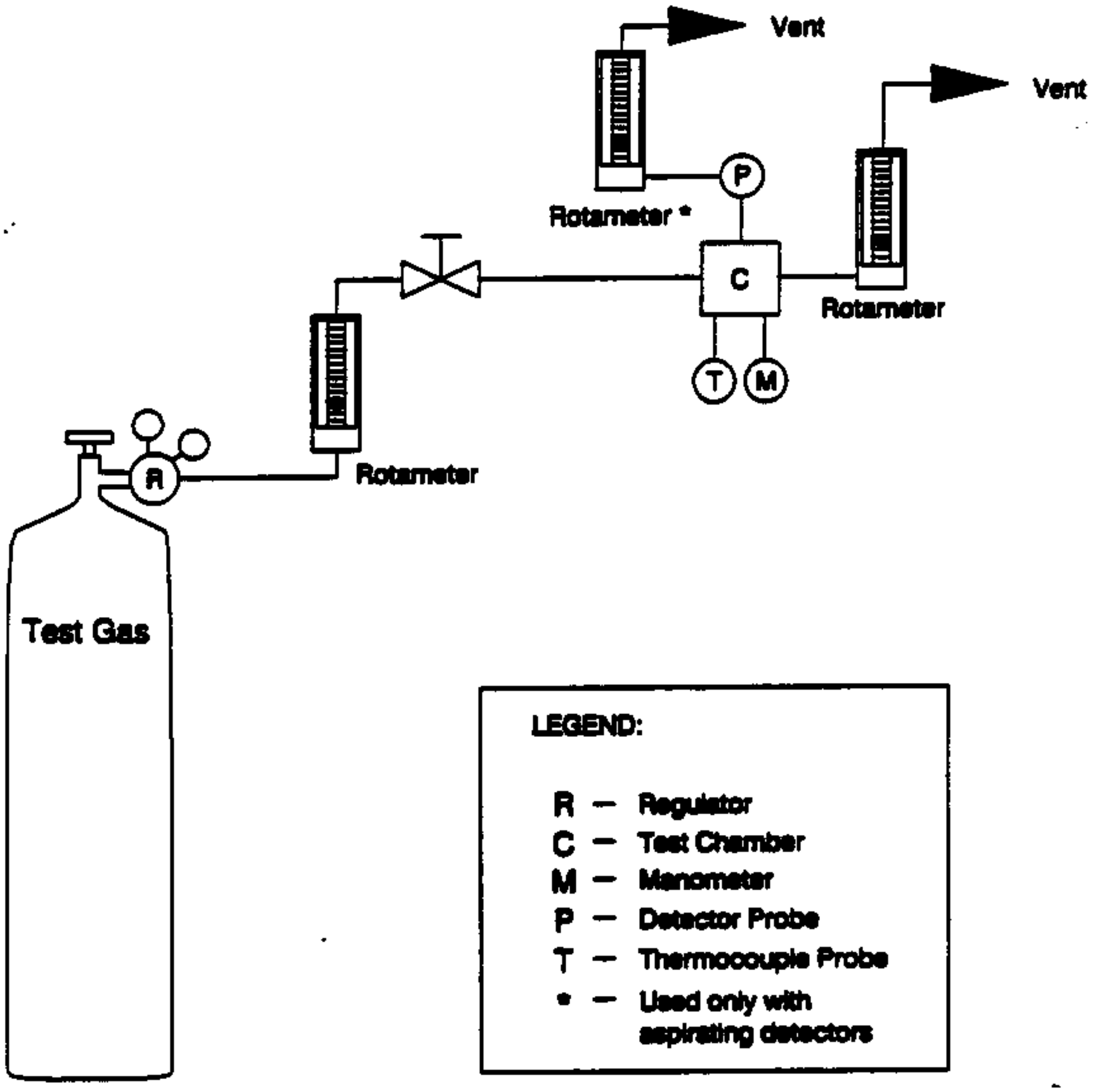

FIG. 1. Test Manifold Schematic Diagram

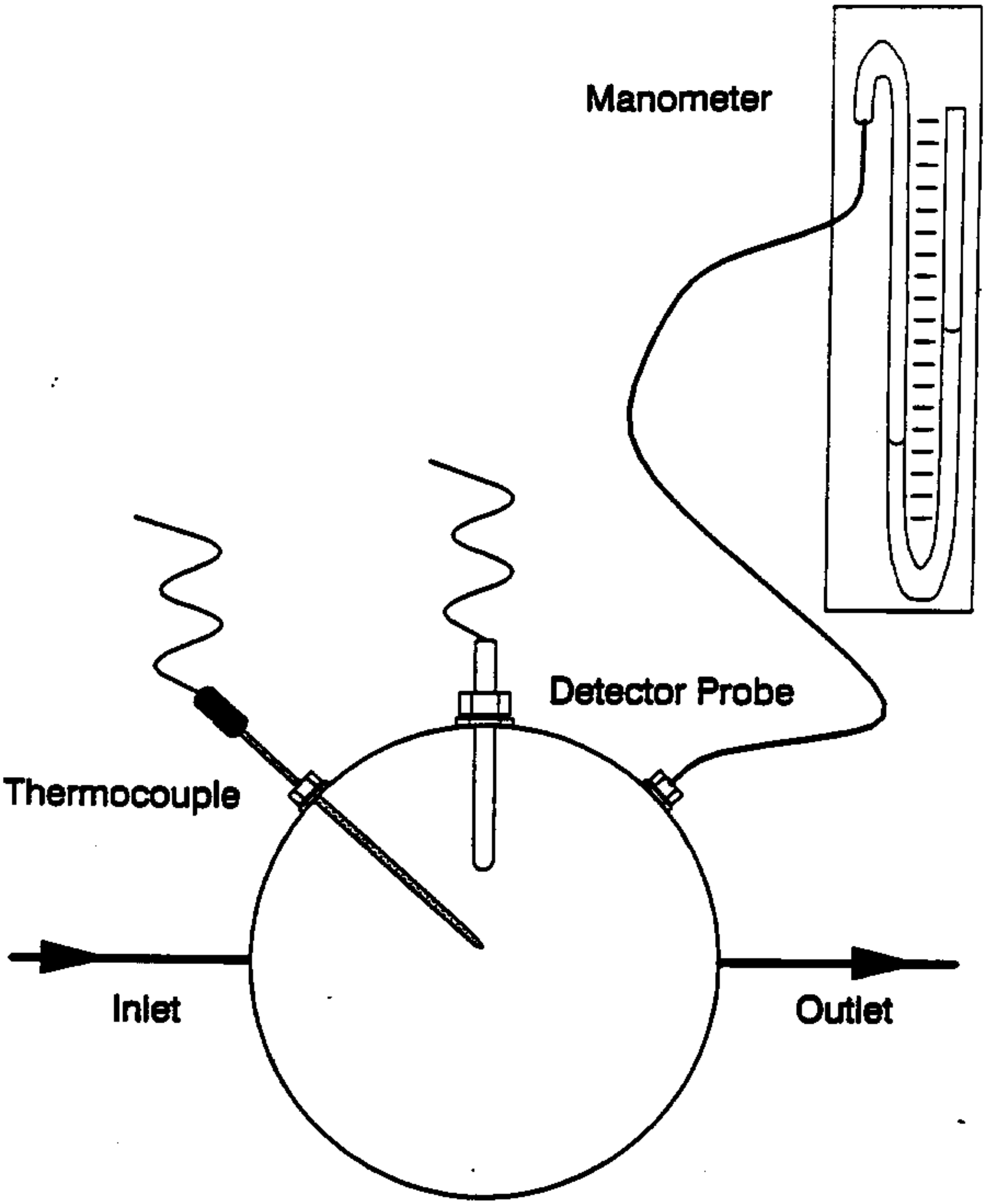

FIG. 2. Test Chamber Schematic Diagram

Detector Name: ____

Detector Type: ___ Quantitative ___ Qualitative

Test Gas	Concentration, ppmv	Accuracy, %	Precision, %	Bias, %	Detection Time	Fall Time
benzene	50					
benzene	250					
benzene	500					
benzene	1000					
2-methylbutane	50					
2-methylbutane	250					
2-methylbutane	500					
2-methylbutane	1000					

FIG. 3. Data Recording Form

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENTS

A1.1 Compressed Gas. Benzene in Air.

Danger—Poison Carcinogen.

Keep container closed.

Use fume hood whenever possible.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do no mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder out of sun and away from heat.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.2 Compressed Gas. 2-Methylbutane in Air.

Warning—May be harmful if inhaled.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.3 Compressed Gas. Air.

Caution—Compressed gas under high pressure.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder out of sun and away from heat.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

APPENDIX

X1. EXAMPLE TEST CHAMBERS

X1.1 It is not reasonable to try to design a single test chamber that accommodates all configurations of detector probes. Instead, it is better to define the general requirements for the chamber and allow specific chambers to be designed to meet requirements for particular probes. Included in this appendix are diagrams of three test chambers that, combined, should be suited to the vast majority of vapor-phase detectors. The included chamber designs may also serve as starting points for alternate chamber designs.

X1.2 *Test Chamber for Tubing Probes*—Figure X1 contains a diagram detailing a test chamber design that should be suitable for almost all vapor-phase detectors that have probes consisting of tubing. These detectors primarily aspirate gas samples through tubing to a sensor located in the control box.

X1.3 *Test Chamber for Probes Up to 3/8-Inch Diameter*—Figure X2 is a diagram of a test chamber design that should be adequate for detector probes with diameters up to 3/8 inch.

X1.4 *Test Chamber for Probes Up to 1-1/4-Inch Diameter*—Figure X3 is a diagram of a test chamber design that should be adequate for detector probes with diameters up to 1-1/4 inches.

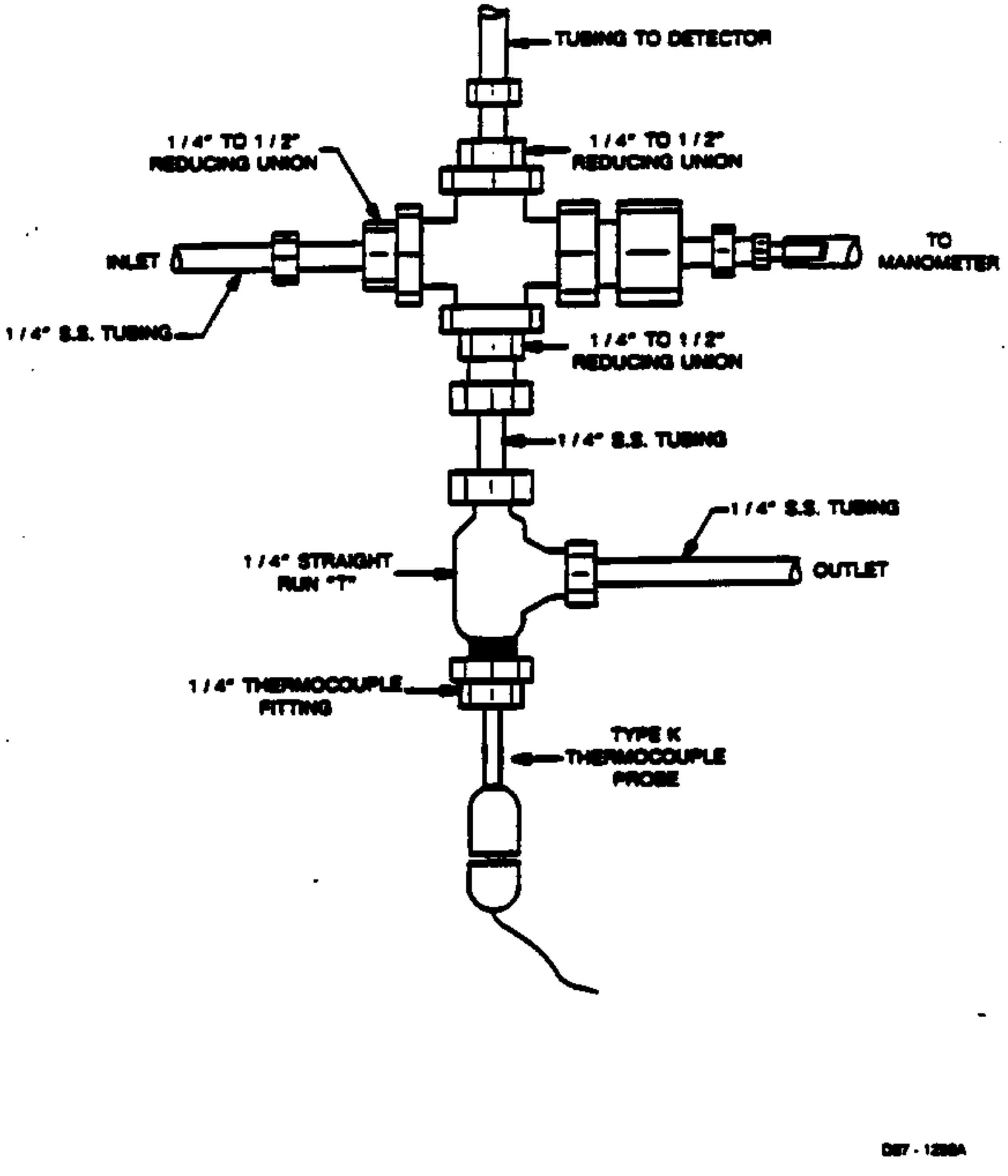

FIG. X1. Gas Test Chamber for Tubing Probes

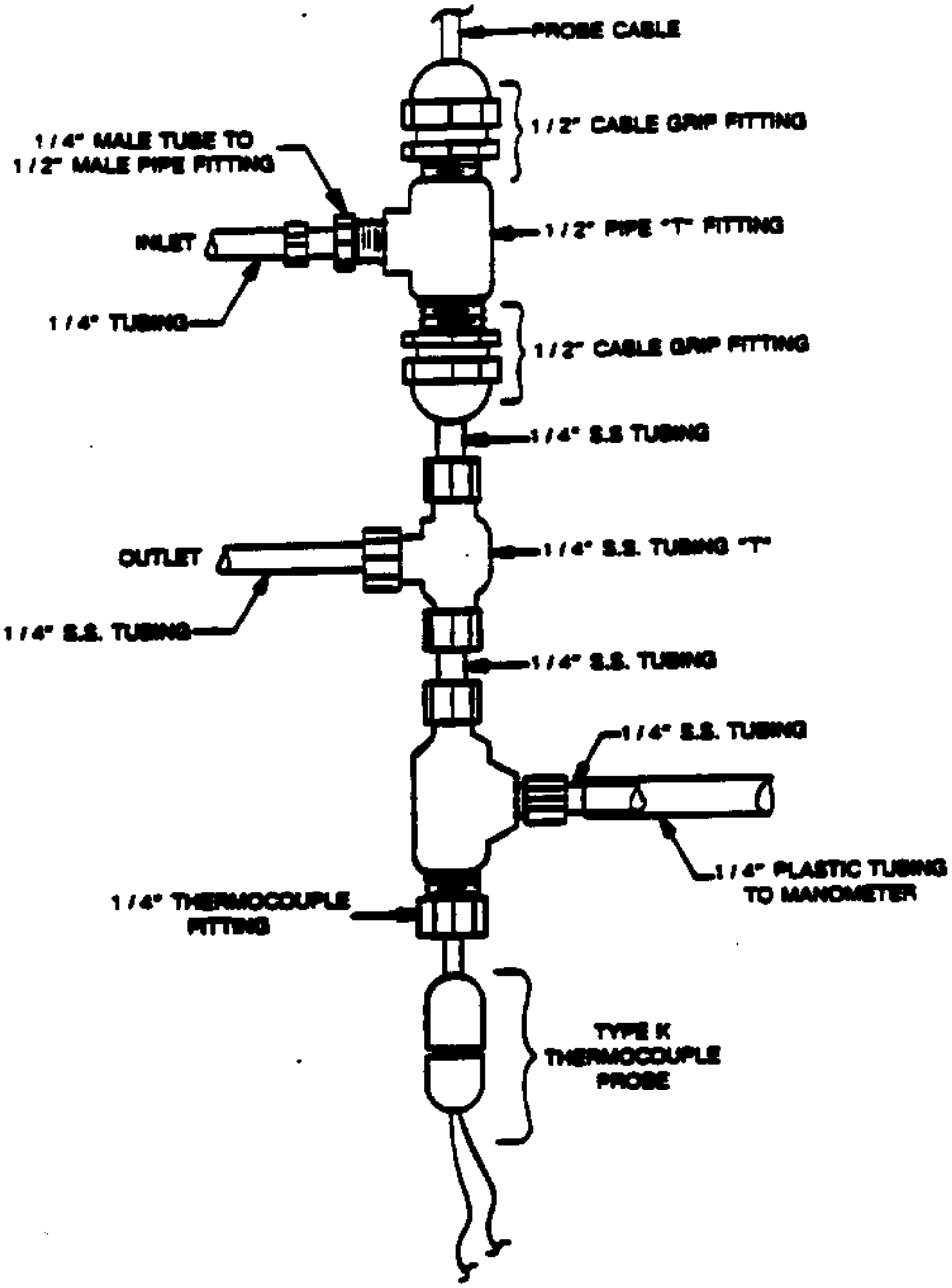

FIG. X2. Gas Test Chamber for Probes with Diameters Up To 3/8 Inch

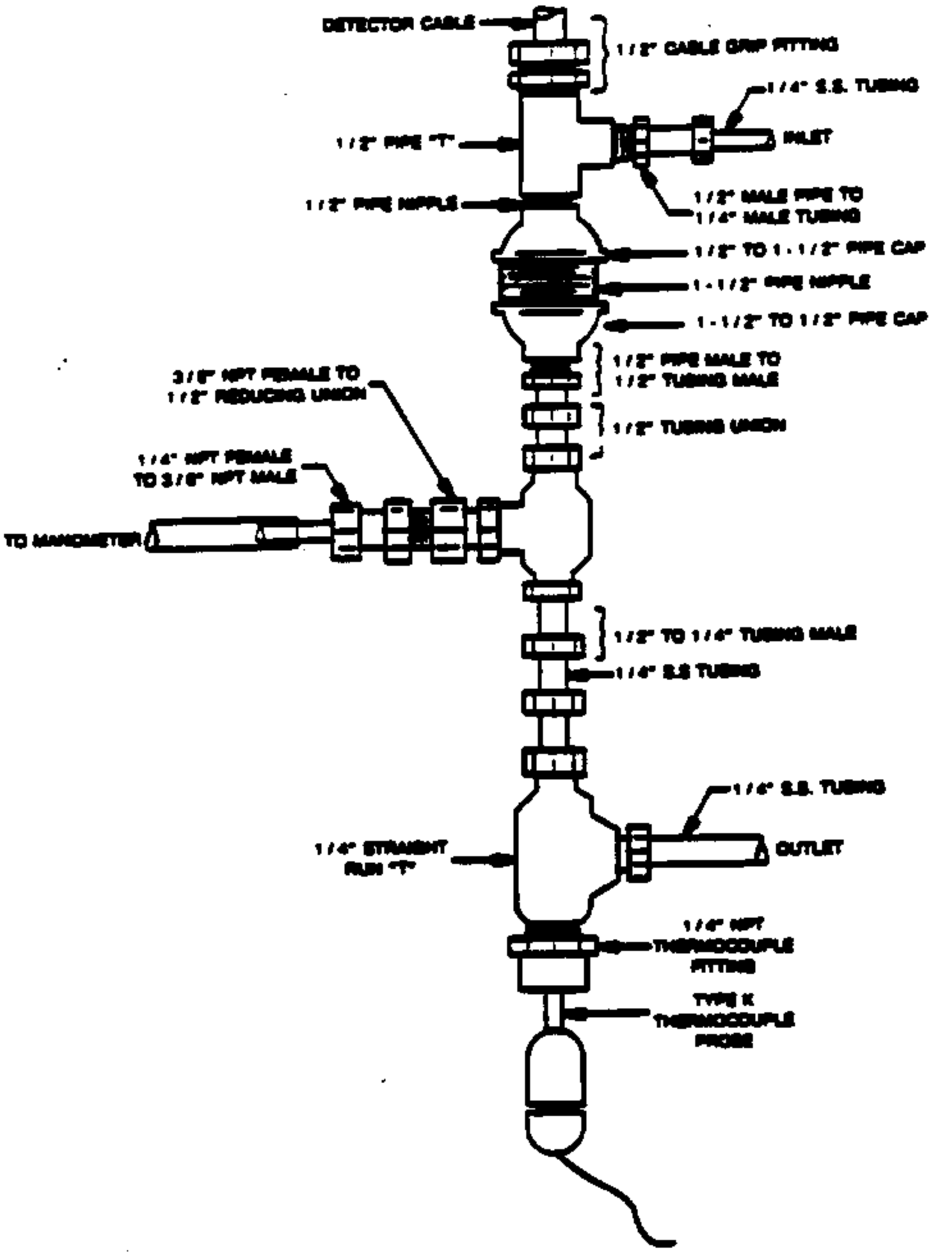

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FIG. X3. Gas Test Chamber for Probes with Diameters Up To 1-1/4 Inches

Designation: X 0003

Standard Test Method for
SPECIFICITY FOR VAPOR-PHASE OUT-OF-TANK PETROLEUM DETECTORS

1. Scope

1.1 This test method covers determination of specificity of vapor-phase out-of-tank petroleum hydrocarbon leak detectors.

1.2 This method is applicable to only the components associated with detection of vapor-phase petroleum releases for detection systems utilizing multiple operating principles.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards*:

E 1 Standard Specification for ASTM Thermometers

E 456 Standard Terminology Relating to Statistics

3. Terminology

3.1 *Definitions* — For formal definitions of statistical terms, see Terminology E 456.

3.2. Descriptions of Terms Specific to This Method

3.2.1 *activated*—refers to the state of a qualitative detector's response when indicating the presence of hydrocarbon vapors.

3.2.2 *non-activated*—refers to the state of a qualitative detector's response when indicating that no hydrocarbon vapors are detected.

3.2.3 *probe*—component of a detection system that must come into contact with petroleum gas before the gas can be detected.

3.2.4 *qualitative responses*—type of detector response that indicates only the presence or absence of hydrocarbon vapors without determining the specific hydrocarbon concentration.

3.2.5 *quantitative responses*—type of detector response that quantitates the concentration of the hydrocarbon vapor present.

3.2.6 *responses*—detector's indication of the presence of petroleum hydrocarbon vapors. Responses can be qualitative or quantitative.

3.2.7 *specificity*—ability of a detector to respond to various substances.

4. Summary of Test Method

4.1 Detector probes are subjected to each of six different test atmospheres inside a sealed test chamber. Test gas concentrations are nominally 500 parts per million by volume (ppmv). Detector response is monitored for up to 24 hours.

5. Significance and Use

5.1 For vapor-phase petroleum hydrocarbon detectors, specificity is a measure of how sensitive a detector is to different test gases.

5.2. Results obtained using this method will permit the most advantageous use of a detector. Weaknesses as well as strengths of the instrument should become apparent. It is not the interest of this method to compare similar detectors of different manufacture, but to enable the user to choose a suitable detector.

6. Interferences

6.1 Conditions that can cause interferences with this method include temperature changes, high temperatures, excessive apparatus volumes, and leaks in the test apparatus. To avoid these conditions, tests should be conducted at relatively constant normal laboratory temperatures with a leak-tested test apparatus.

6.2 Cross contamination (i.e., memory effects from residual test atmospheres) may be a major cause of inaccurate data. To minimize this potential problem, avoid using rubber or plastic parts for components of the test apparatus that contact test gases and purge the testing system as described in Section 11.

6.3 Fluctuations of the test chamber internal pressure may significantly affect the detector's response. Maintain a constant internal chamber pressure (±0.2 inches of water relative to ambient pressure) while the probe is exposed to the test atmosphere by maintaining a constant inlet and exit test atmosphere flow rate. If the pressure varies outside the acceptance limits, repeat the tests within the accepted pressure fluctuation limits.

7. Apparatus

7.1 *Test apparatus*—The test apparatus, as depicted in Figure 1, shall be constructed from materials that are inert with respect to test gases. The test apparatus consists of compressed gas cylinders, pressure regulators, tubing, valves, tubing connectors, rotameters, test chamber, thermocouple, and manometer.

7.1.1 *Compressed gas cylinders*—Test gases are supplied in standard compressed gas cylinders having Compressed Gas Association (CGA) fittings compatible with regulator fittings.

Note 1—Dilute hydrocarbon test gases and ultrahigh-purity air are commonly supplied in compressed gas cylinders having CGA 590 fittings.

7.1.2 *Pressure regulators*—A dual stage regulator with a fitting compatible with the test gas cylinders is needed. The regulator(s) shall have a range of at least 0 psi to 15 psi and have a diaphragm made of stainless steel.

7.1.3 *Tubing*—Sufficient tubing to link all test apparatus components is needed. The tubing shall be free from contaminants and have an internal diameter of at least 1/8 inch. The tubing shall be made of a material that is inert with respect to test gases.

7.1.4 *Tubing connectors*—Various compression-type tubing fittings are needed to make test apparatus connections. These fittings shall be free from contaminants, inert with respect to test gases, and of a material that is compatible with the tubing. These fittings can include, but are not limited to, tees, cross tees, reducers, and elbows. A thermocouple compression fitting is also needed.

7.1.5 *Rotameters*—Two rotameters, each having a flow range bracketing the required flow rates for the detector, are needed to measure test chamber inlet and outlet vapor rates. A third rotameter is required for detectors that aspirate gas samples.

7.1.6 *Valves*—A shut-off valve and a flow controlling needle valve are needed.

Note 2—Gas shut off can be accomplished using a regulator and the integral shut-off valve commonly included with compressed gas cylinders. Many rotameters include an integral flow controlling needle valve.

7.1.7 *Thermocouple*—A thermocouple and temperature readout, or equivalent, that responds from 0°C to 40°C and is accurate and precise to within 1°C over this range is needed.

7.1.8 *Manometer*—A relative pressure manometer is required to monitor the test chamber's internal pressure. The manometer must have a working range of at least 0-10 inches of water (0 to 20 mm Hg) with an accuracy of ±5%. The manometer should be scaled using at least 0.2 inches of water (0.4 mm Hg) subdivisions.

7.1.9 *Test chamber*—The test chamber should be gas-tight and made from materials that are inert with respect to test gases. Figure 2 contains a test chamber schematic representation. The test chamber must have fittings to allow connection to the detector probe, a manometer, and a thermocouple. The chamber must also have an inlet and outlet for flow of test atmospheres. Test chamber volumes should be kept as small as possible without interfering with detector operation. Diagrams of suitable test chambers are presented in Appendix A.

7.2 *Timer*—A timer that is accurate and precise to at least one second per 10 minutes is required. Alternatively, a chart recorder or other data acquisition system may be used. If a chart recorder or other data acquisition system is to be used, a timer is not required. If used, the recorder or data acquisition system timing must be accurate and precise to at least one second per 10 minutes.

7.3 *Electronic recorder*—A chart recorder or other electronic data acquisition system may be used if it is compatible with the specific detector that is being evaluated. The output of the data recorder should be accurate to ±2% over the range of output from a quantitative detector. A data recorder used with a qualitative detector must unambiguously identify activated and inactivated states.

7.4 *Thermometer*—ASTM Solvents Distillation Thermometer having a range from -2°C to 52°C and conforming to the requirements for Thermometer 37C as prescribed in Specification E 1.

7.5 *Bubble meter*—Use NBS-traceable soap bubble flow meters to calibrate rotameters. The bubble meters must have a working range that brackets the range of all rotameters.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents for the American Chemical Society where such specifications are available.[1] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of determination.

8.2 *Test Gases*—Use factory-mixed gas standards in conventional gas cylinders as test chamber atmosphere sources. Make-up gas for all standards shall be air. The concentration of each gas shall be 500±10 ppmv and must be certified accurate (±2%). The gas standards required are as follows:

8.2.1 *Benzene*—500 ppmv in air. (<u>Danger</u>—*Gas under high pressure. May be harmful if inhaled. See Annex A1.1.)*

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[1] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemicals Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., In., New York, NY, and the "United States Pharmacopeia."

8.2.2 *n-Butane*—500 ppmv in air. (Caution—*Gas under high pressure. See Annex A1.2.)*

8.2.3 *n-Hexane*—500 ppmv in air. (Caution—*Gas under high pressure. See Annex A1.3.)*

8.2.4 *Isobutane*—500 ppmv in air. (Caution—*Gas under high pressure. See Annex A1.4.)*

8.2.5 *2-Methylpentane*—500 ppmv in air. (Caution—*Gas under high pressure. See Annex A1.5.)*

8.2.6 *3-Methylpentane*—500 ppmv in air. (Caution—*Gas under high pressure. See Annex A1.6.)*

8.2.7 *Toluene*—500 ppmv in air. (Caution—*Gas under high pressure. See Annex A1.7.)*

8.3 *Ultrahigh-purity air*—The ultrahigh-purity air standard must have less than 0.5 ppmv total hydrocarbon content. (Caution—*Gas under high pressure. See Annex A1.8.*)

9. Calibration and Standardization

9.1 *Chart Recorder or Other Data Recording System*—If used, a chart recorder or other data recording system should be calibrated along with the detector. The data recording system should be calibrated according to instructions from its manufacturer and the detector manufacturer. Also, any recording device should be compatible with the detector being investigated. Consult specifications from the manufacturers of the recording device and the detector.

9.2 *Detector*—Because of wide design variability among different petroleum detectors, it is impossible to give complete calibration instructions for all possible detector designs. Calibrate all detectors according to manufacturer instructions.

9.3 *Rotameters*—Calibrate each rotameter prior to initiating test procedures, once a year thereafter, and after any internal contamination (e.g., dirt, moisture, etc.) during testing. Instructions accompanying the NBS-traceable bubble meters should be followed. The rotameters are to be calibrated at a minimum of five points ("multipoints") within the working range. Flow readings should be made from the middle of the ball float. All readings should be made from the upper float on dual-float rotameters until it is off scale. Once the upper float is off scale, readings should be made from the lower float.

9.3.1 *Assembly*—Remove the rotameter from the test system, if assembled, and connect the rotameter in series between a controllable compressed air source and an NBS-traceable bubble meter. Use a bubble meter cylinder that will allow a flow measurement over a period of 15 to 45 seconds.

9.3.2 *First calibration point*—Bring the gas flow rate to the lowest calibration flow for testing. Let the system run at this setting until the rotameter is steady. If the flow rate is not within the first level range, adjust the flow rate until it is within that range and wait for a steady-state value. Record the steady-state flow meter value that is within the first flow rate level. Measure the flow rate with the bubble meter and stopwatch according to the NBS bubble meter instructions. Record each reference flow rate.

9.3.3 *Remaining calibration points*—Record steady-state settings for triplicate runs as described in Section 10.3.2 for at least four more flow rates throughout the rotameter range.

9.4 *Thermocouple*—Perform side-by-side multipoint calibrations for each thermocouple used in the test procedure in a 1-L glass beaker filled with water. The reference thermometer should be an ASTM Solvents Distillation thermometer having a range from -2°C to 52°C and conforming to the requirements for Thermometer 37C as prescribed in Specification E 1. The levels tested are low (room temperature - 10°C), room temperature, and high (room temperature + 10°C).

9.4.1 Insert both the thermocouple and reference thermometer into the beaker of water and add small quantities of ice. Allow the ice to melt and the temperature to stabilize. Continue adding ice until a steady-state reading (±0.5°C over two minutes) of room temperature - 10°C (±2°C) occurs.

9.4.2 Repeat this procedure using room temperature water (15°C to 30°C), and room temperature + 10°C (±2°C) water. If the temperature difference is more than 1°C, either repeat the test with the same thermocouple or replace the thermocouple and repeat the test until it is acceptable.

9.4.3 Perform thermocouple calibration at the onset of testing and at least once a year.

10. Conditioning

10.1 *Before Each Test*—Purge the test chamber for at least three minutes with ultrahigh-purity air at 0.2 L/min.

11. Procedure

11.1 *Test Series*—The detector should be tested with each test gas. Test gases are benzene, *n*-butane, *n*-hexane, isobutane, 2-methylpentane, 3-methylpentane, and toluene.

11.1.1 Perform tests in a random order.

11.2 *Assembly*—Assemble the detection system as described by the manufacturer. Insert the detector probe into the test chamber. The seal between the probe and the test chamber should be gas-tight.

11.2.1 Connect the detector output to a chart recorder or other data acquisition system if one is being used. All connections should be in compliance with specifications from the manufacturers of the detector and the data recording system.

11.3 Calibration—Calibrate the detector if necessary. Many detectors do not require calibration. Perform calibrations, if necessary, according to manufacturer recommendations. Calibration may need to occur before mounting in the test container. If a data recording system is being used, it should be calibrated with the detector. Calibrate the data acquisition system according to manufacturer instructions.

11.4 *Background*—Supply ultrahigh-purity air to the test chamber at a rate that is 0.2 L/min. greater than the detector's aspiration rate.

Note 3: Many detectors are passive and do not aspirate gas samples. Use total test gas flow rates of 0.2 L/min for these detectors.

Monitor the detector's response every 15 seconds until a steady-state reading (±2% change of full scale over one minute) is achieved or four minutes has elapsed, whichever is longer. Monitor the temperature inside the test chamber during background testing.

11.5 *Test atmosphere response*—Introduce the appropriate test gas to the test chamber at a sufficient rate to produce a 0.2 L/min test system vent flow rate. Monitor the temperature inside the test chamber during testing.

11.5.1 Monitor quantitative detectors until a steady-state (±2% of full scale over 1 minute) response occurs or for 24 hours, whichever is shorter. Monitor qualitative detectors for a positive response ("activated") for up to 24 hours.

12. Calculations

12.1 *Specificity for Quantitative Detectors*—Specificity for quantitative detectors is the ratio of detector output, or measured concentration, to the actual concentration of hydrocarbon test gas expressed as a percentage. The following equation should be used to calculate specificity for quantitative detectors:

$$\text{Specificity, \%} = 100 \times m_d/c$$

where:

m_d = detector's output reading, ppmv; and

c = hydrocarbon concentration, ppmv (c = 500 ppmv).

13. Interpretation of Results

13.1 *Specificity for Qualitative Detectors*—Specificity for qualitative devices should be reported as "activated" if the detector responds within 24 hours. Otherwise, specificity should be reported as "inactivated."

14. Report

14.1 Report detector type (quantitative or qualitative) and specificity results for each test product.

15. Precision and Bias

15.1 *Precision*—The precision of the procedure in Test Method X 0003 for measuring specificity for vapor-phase out-of-tank petroleum detectors is being determined.

15.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in Test Method X 0003 for measuring specificity for vapor-phase out-of-tank petroleum detectors, no statement on bias is being made.

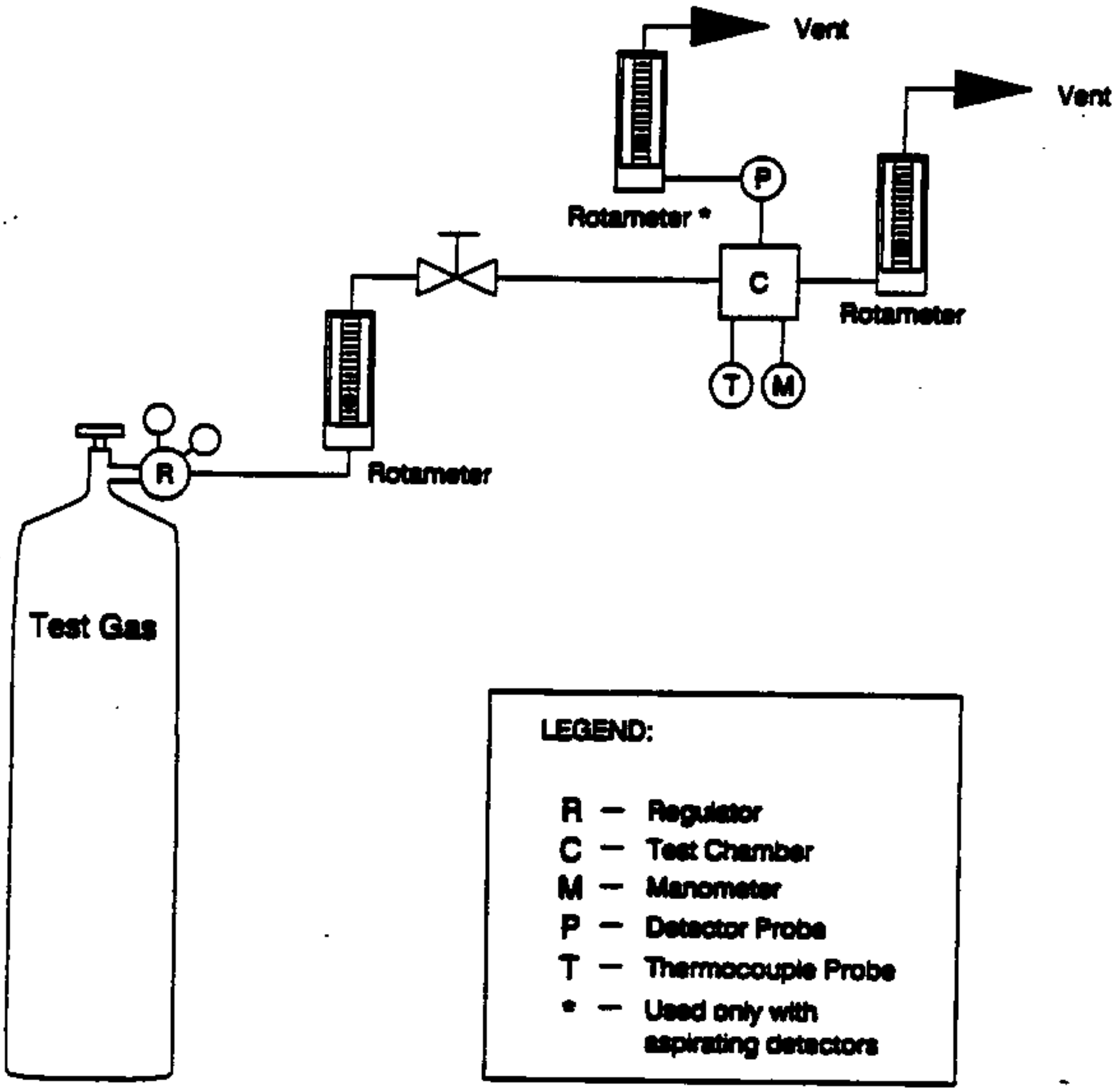

FIG. 1. Test Manifold Schematic Diagram

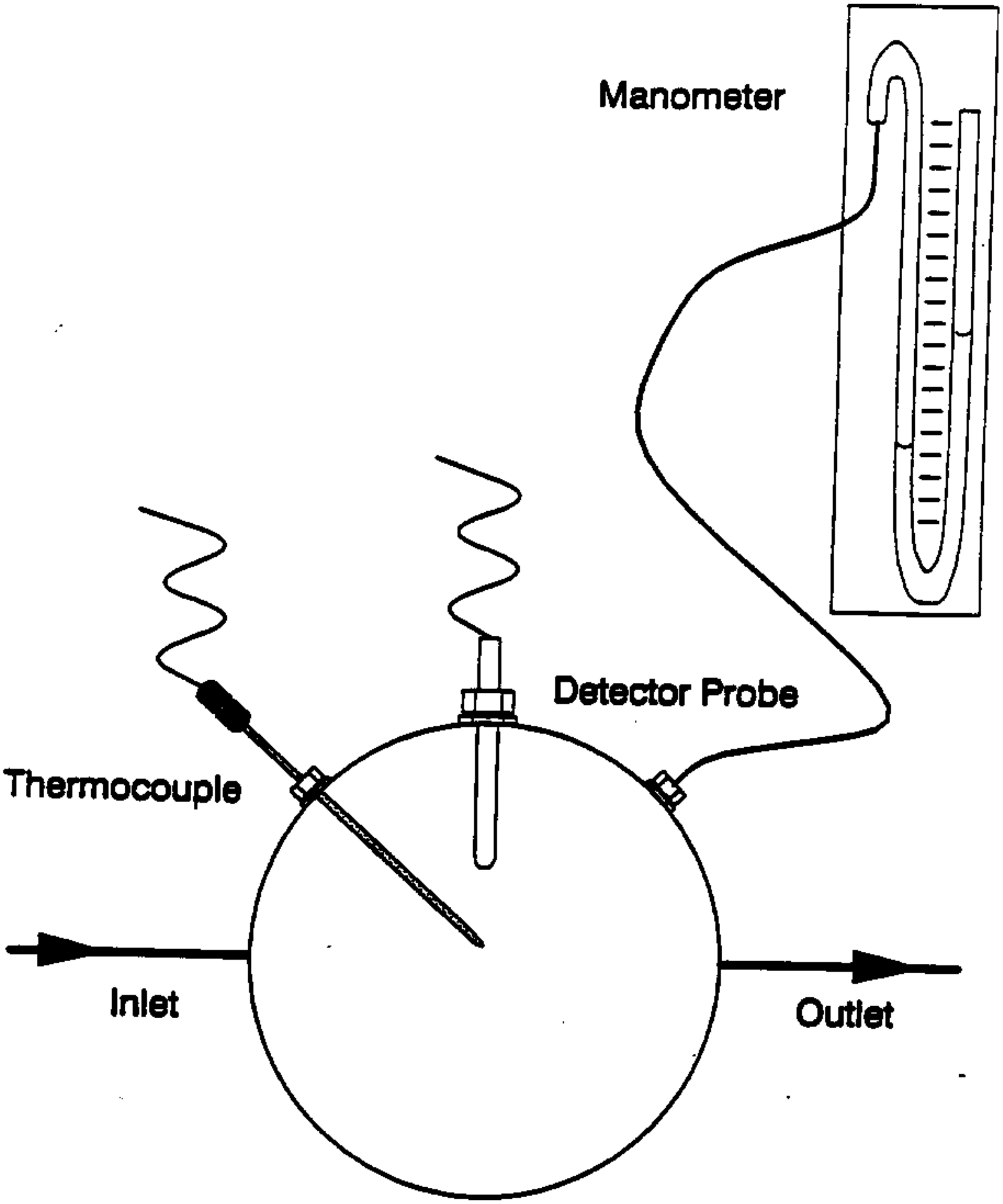

FIG. 2. Test Chamber Schematic Diagram

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENTS

A1.1 Compressed Gas. Benzene in Air.

Danger—Poison, Carcinogen.

Keep container closed.

Use fume hood whenever possible.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do no mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder out of sun and away from heat.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.2 Compressed Gas. _n_-Butane in Air.

Warning—May be harmful if inhaled.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.3 Compressed Gas. n-Hexane in Air.

Warning--May be harmful if inhaled.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.4 Compressed Gas. Isobutane in Air.

Warning--May be harmful if inhaled.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.5 **Compressed Gas. 2-Methylpentane in Air.**

Warning—May be harmful if inhaled.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.6 Compressed Gas. 3-Methylpentane in Air.

Warning—May be harmful if inhaled.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.7 Compressed Gas. Toluene in Air.

Warning—May be harmful if inhaled.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.8 **Compressed Gas. Air.**

Caution—Compressed gas under high pressure.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder out of sun and away from heat.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

APPENDIX

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X1.1 It is not reasonable to try to design a single test chamber that accommodates all configurations of detector probes. Instead, it is better to define the general requirements for the chamber and allow specific chambers to be designed to meet requirements for particular probes. Included in this appendix are diagrams of three test chambers that, combined, should be suited to the vast majority of vapor-phase detectors. The included chamber designs may also serve as starting points for alternate chamber designs.

X1.2 *Test Chamber for Tubing Probes*—Figure X1 contains a diagram detailing a test chamber design that should be suitable for almost all vapor-phase detectors that have probes consisting of tubing. These detectors primarily aspirate gas samples through tubing to a sensor located in the control box.

X1.3 *Test Chamber for Probes Up to 3/8-Inch Diameter*—Figure X2 is a diagram of a test chamber design that should be adequate for detector probes with diameters up to 3/8 inch.

X1.4 *Test Chamber for Probes Up to 1-1/4-Inch Diameter*—Figure X3 is a diagram of a test chamber design that should be adequate for detector probes with diameters up to 1-1/4 inches.

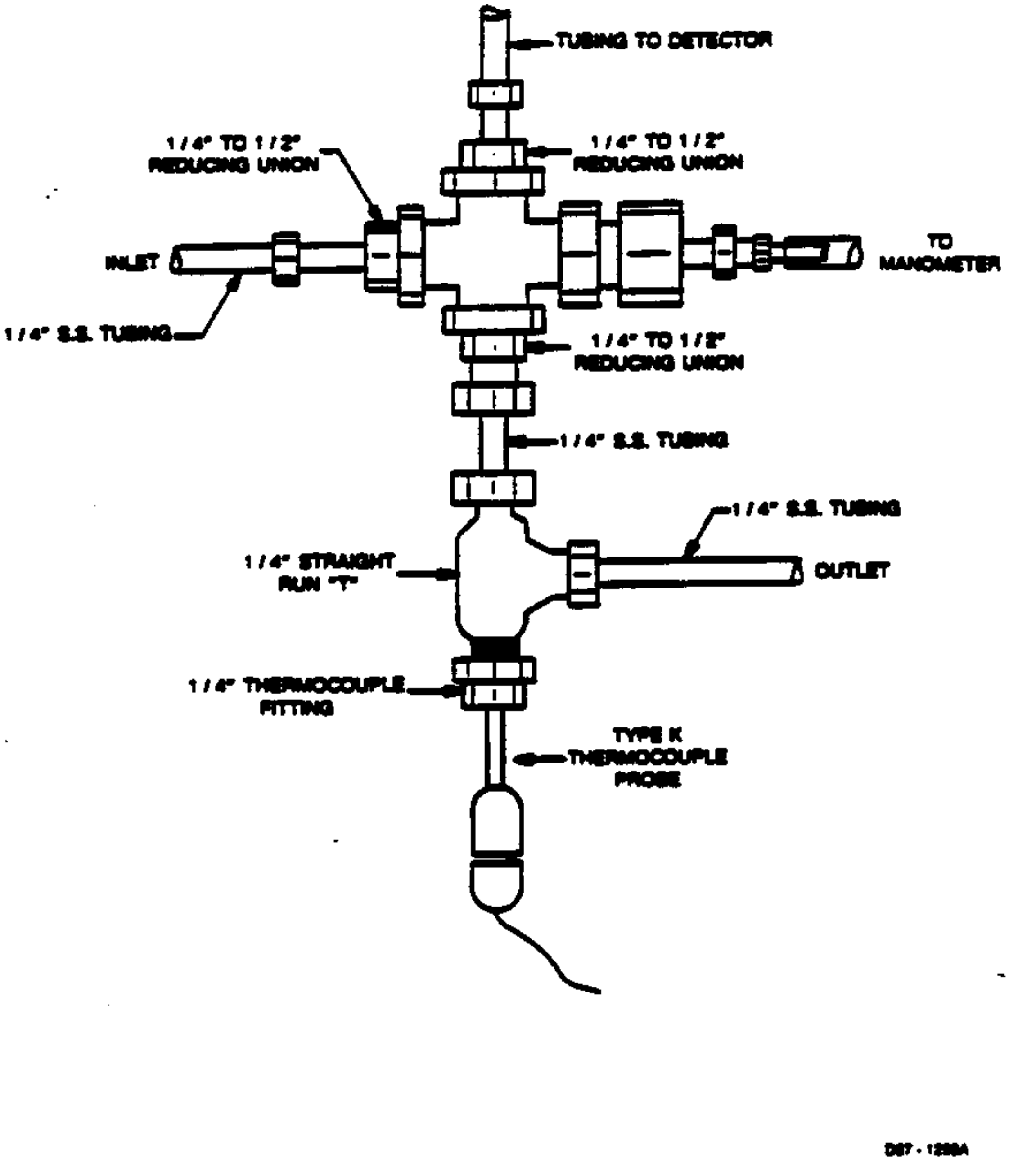

FIG. XI. Gas Test Chamber for Tubing Probes

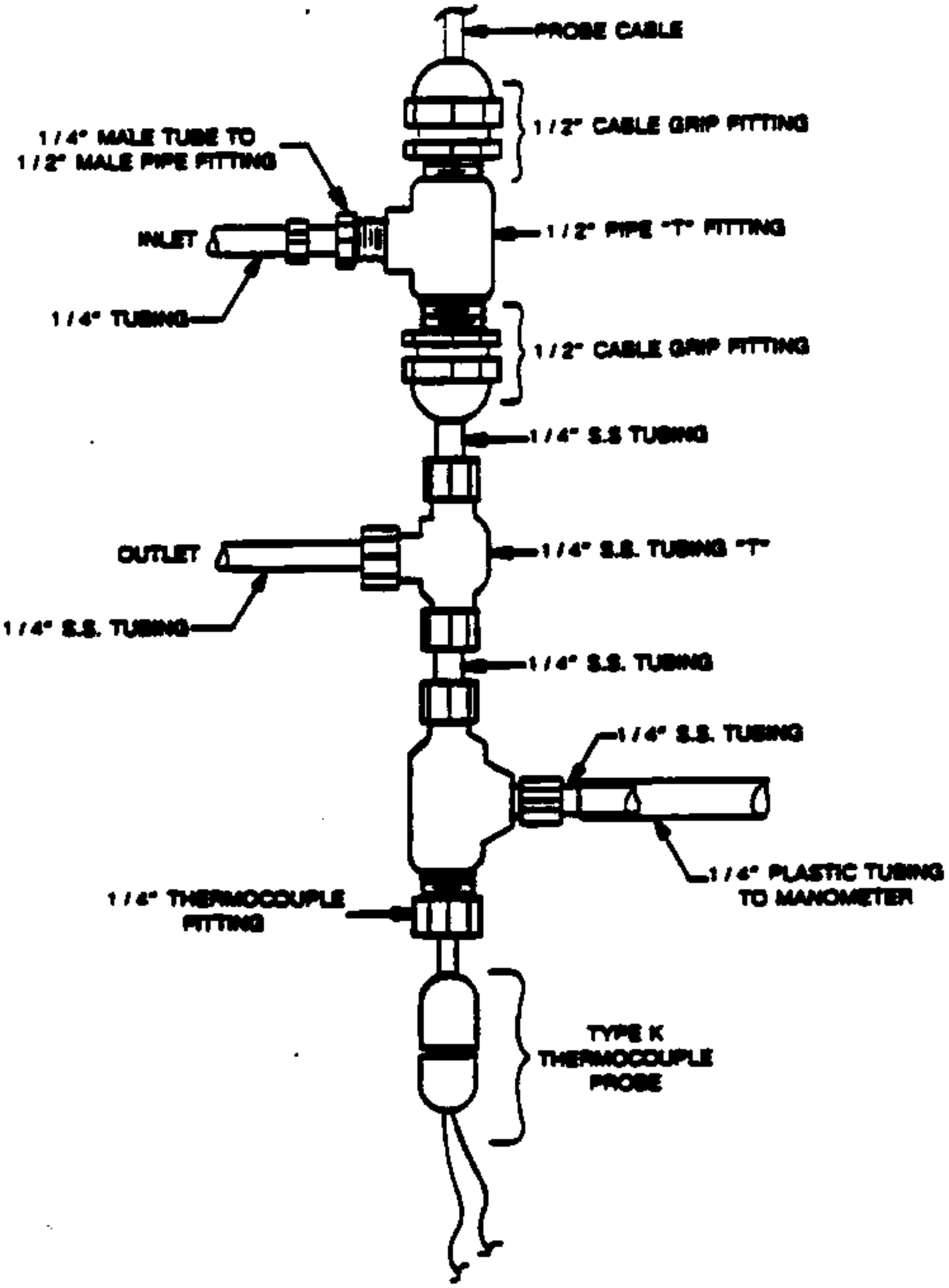

FIG. X2. Gas Test Chamber for Probes with Diameters Up To 3/8 Inch

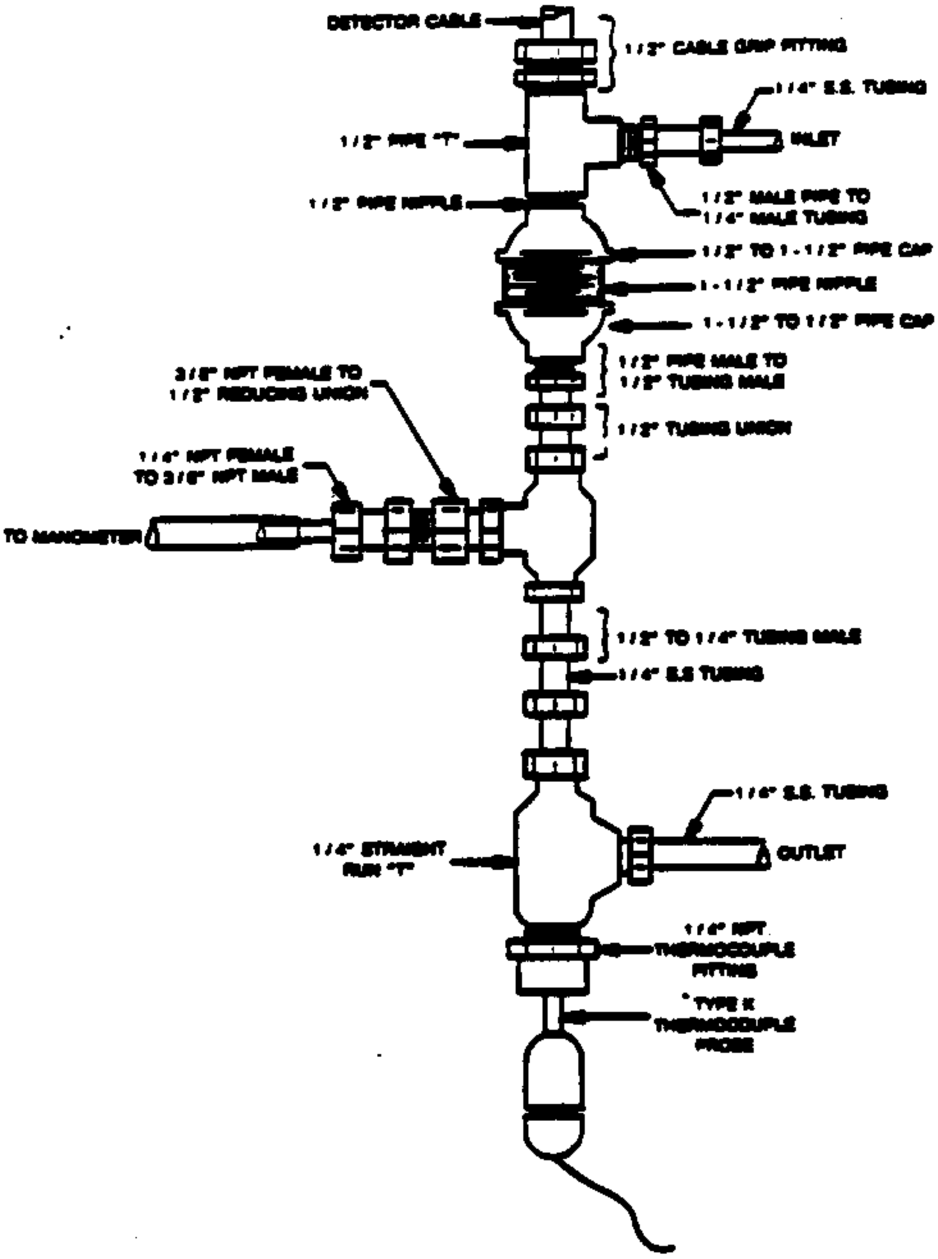

FIG. X3. Gas Test Chamber for Probes with Diameters Up To 1-1/4 Inches

Designation: X 0006

Standard Test Method for
LOWER DETECTION LIMIT FOR VAPOR-PHASE OUT-OF-TANK PETROLEUM DETECTORS

1. Scope

1.1 This test method covers determination of lower detection limit of vapor-phase out-of-tank petroleum hydrocarbon leak detectors.

1.2 This method is applicable to only the components associated with detection of vapor-phase petroleum releases for detection systems utilizing multiple operating principles.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards*:

E 1 Standard Specification for ASTM Thermometers

E 456 Standard Terminology Relating to Statistics

3. Terminology

3.1 *Definitions*—For formal definitions of statistical terms, see Terminology E 456.

3.2. Descriptions of Terms Specific to This Method

3.2.1 *activated*—refers to the state of a qualitative detector's response when indicating the presence of hydrocarbon vapors.

3.2.2 *critical level*—point at which a detector's response becomes significantly different from a blank response.

3.2.3 *lower detection limit*—minimum concentration of hydrocarbon test vapor that has only a 5 percent chance of not being detected at a 95 percent confidence level.

3.2.4 *non-activated*—refers to the state of a qualitative detector's response when indicating that no hydrocarbon vapors are detected.

3.2.5 *probe*—component of a detection system that must come into contact with petroleum gas before the gas can be detected.

3.2.6 *qualitative responses*—type of detector response that indicates only the presence or absence of hydrocarbon vapors without determining the specific hydrocarbon concentration.

3.2.7 *quantitative responses*—type of detector response that quantitates the concentration of the hydrocarbon vapor present.

3.2.8 *responses*—detector's indication of the presence of petroleum hydrocarbon vapors. Responses can be qualitative or quantitative.

4. Summary of Test Method

4.1 Detector probes are subjected to test atmospheres of either benzene or 2-methylbutane inside a sealed test chamber. Detectors are screened at several different test product concentrations to determine the lowest concentration to which the detectors will respond reliably. Test product concentrations are 1.25 parts per million by volume (ppmv), 2.5 ppmv, 5 ppmv, 12.5 ppmv, 25 ppmv, 50 ppmv, 125 ppmv, 250 ppmv, 500 ppmv, and 1000 ppmv.

4.2 The concentration at which a detector is tested is referred to as the lower detection limit, or "LDL." For qualitative detectors, the next lowest concentration is referred to as "LDL-." Quantitative detectors require seven tests for each test gas at the LDL concentration. Qualitative detectors require a total of twelve tests for each gas, six tests at both the LDL and LDL- concentrations. If a qualitative detector fails to respond positively to one or more of the six LDL tests, then the LDL concentration is increased and six more tests are performed at the new LDL concentration. Similarly, if a detector responds positively six times to the LDL concentration and one or more times to the LDL- concentration, the LDL- concentration is decreased and six more tests are performed at the new LDL- concentration.

5. Significance and Use

5.1 For vapor-phase petroleum hydrocarbon detectors, the lower detection limit, or "LDL," is the minimum hydrocarbon concentration to which a detector will respond positively. This method uses hydrocarbon gas concentrations from 1.25 ppmv to 1000 ppmv. Benzene and 2-methylbutane are the hydrocarbon gases used in this method.

5.2 The lower limits of a detector's ability to detect hydrocarbon vapors may be described in terms of two types of detection errors that are possible. One type of error involves concluding that hydrocarbon product is present when there actually is not any product. This is the classic "false alarm," which is referred to as a false positive or Type I error. The risk of making a Type I error is usually denoted as α (alpha). The second type of error that can be made is referred to as a false negative or Type II error.

This is the error of not detecting the presence of hydrocarbon product, and the risk associated with making this type of error is denoted as β (beta). Limits of detectors described below are based on the risks associated with these two types of error to prescribed hydrocarbon vapor concentrations.

5.3 Data obtained from repeated testing of quantitative detectors will be used to define two characteristics associated with the detector capabilities. These two characteristics, corresponding to the two possible types of detection errors described above, will be referred to as the critical level and the detection limit. The critical level is the decision-point test-vapor concentration, above which hydrocarbons are "detected" and below which they are "not detected." For quantitative detectors, the critical point will be established at a concentration corresponding to a 5 percent risk of false positive error. The detection limit will be defined as a second, higher test-vapor concentration, at which the risk of false negative error is no more than 5 percent. The estimated magnitude of α and β risk assume a normal error distribution.

5.3.1 Figure 1 shows the critical level, the lower detection limit, and the α and β risks applicable to quantitative detectors. The critical level is the point at which detector response becomes significantly different from a blank response. The two bell-shaped curves represent probability density functions (PDF). The curve on the left represents the PDF for possible detector responses for a test vapor having a true hydrocarbon concentration of zero. The critical level (L_c) is located on the right tail of the curve. The critical level will be established using a one-sided tolerance limit for normal distribution. Using the tolerance limit approach, it may be stated with 95 percent confidence that a false positive error will be made no more than five percent of the time.

5.3.2 The curve to the right in Figure 1 represents the PDF for possible detector responses for a test vapor having a true hydrocarbon concentration equal to the detection limit (L_D). The detection limit will be established by letting the false negative error coincide with the false positive error at the critical level and then applying the tolerance limit equation. Using the tolerance limit, it can be stated with 95 percent confidence that the lower detection limit concentration is the lowest concentration that has only a five percent chance of not being detected.

5.4 Qualitative detectors require a different test strategy because the data collected are attribute measurements. The critical level and the detection limit may still be used to characterize detector performance, and the risk of detection error may still be expressed in terms of α and β. However, different levels of α and β will be used for these detectors. Figure 2 illustrates the probability of detection versus concentration for qualitative detectors. The false positive risk α is a dependent on the detector and represents the probability that the detector will go to an activated state at zero concentration. For these detectors, the critical level is defined as the test vapor concentration at which the detector has a fifty percent probability of detection. The lower detection limit is the test vapor concentration at which the detector has a probability of not activating that is equal to the false negative risk β.

5.4.1 The critical level and lower detection limit will be established with 95 percent confidence to be within a range bounded by a lower test vapor concentration (LDL-) and an upper test vapor concentration (LDL). LDL and LDL- will be established by measuring the detector response six times at the LDL concentration and six times at the LDL- concentration. If the detector responds six times at the higher concentration and does not respond to the lower concentration, it will be concluded with 95 percent confidence that the critical level and lower detection limit are between the LDL- and LDL concentrations. If, however, the detector fails to respond to one or more of the six tests, then the LDL concentration will be increased and six more tests will be performed at the new LDL concentration. Similarly, if the detector responds six times to the LDL concentration and one or more times to the LDL- concentration, then the LDL- concentration will be decreased and six more tests will be performed at the new LDL- concentration.

5.4.2 A risk of 50 percent will be set for false negative error (β) and false positive error (α). This risk level requires six tests at the LDL and LDL- test vapor concentrations. By establishing these risk levels, it can be stated with 95 percent confidence that the detector will respond to a minimum of 50 percent to the LDL concentration and a maximum of 50 percent to the LDL- concentration. The number of experiments is based on a binomial distribution and is directly related to false positive risk and false negative risk.

5.5 Results obtained using this method will permit the most advantageous use of a detector. Weaknesses as well as strengths of the instrument should become apparent. It is not the interest of this method to compare similar detectors of different manufacture, but to enable the user to choose a suitable detector.

6. Interferences

6.1 Conditions that can cause interferences with this method include temperature changes, high temperatures, excessive test apparatus volumes, and leaks in the test apparatus. To avoid these conditions, tests should be conducted at constant normal laboratory temperatures with a leak-tested test apparatus.

6.2 Cross contamination (e.g., memory effects from residual test atmospheres) may be a major cause of inaccurate data. To minimize this potential problem, avoid using rubber or plastic parts for components of the test apparatus that contact test gases and purge the testing system as described in Section 10.

6.3 Fluctuations of the test chamber internal pressure may significantly affect the detector's response. Maintain a constant internal chamber pressure (±0.2 inches of water relative to ambient pressure) while the probe is exposed to the test atmosphere by maintaining a constant inlet and exit test atmosphere flow rate. If the pressure varies outside the acceptance limits, repeat the tests within the accepted pressure fluctuation limits.

7. Apparatus

7.1 *Test apparatus*—The test apparatus, as depicted in Figure 3, shall be constructed from materials that are inert with respect to test gases. The test apparatus consists of compressed gas cylinders, pressure regulators, tubing, valves, tubing connectors, rotameters, test chamber, thermocouple, and manometer.

7.1.1 *Compressed gas cylinders*—Test gases are supplied in standard compressed gas cylinders having Compressed Gas Association (CGA) fittings compatible with regulator fittings.

Note 1—Dilute hydrocarbon test gases and ultrahigh-purity air are commonly supplied in compressed gas cylinders having CGA 590 fittings.

7.1.2 *Pressure regulators*—At least one dual stage regulator with a fitting compatible with the test gas cylinders is needed. The regulator(s) shall have a range of at least 0 psi to 15 psi and have a diaphragm made of stainless steel.

7.1.3 *Tubing*—Sufficient tubing to link all test apparatus components is needed. The tubing shall be free from contaminants and have an internal diameter of at least 1/8 inch. The tubing shall be made of a material that is inert with respect to test gases.

7.1.4 *Tubing connectors*—Various compression-type tubing fittings are needed to make test apparatus connections. These fittings shall be free from contaminants, inert with respect to test gases, and of a material that is compatible with the tubing. These fittings can include, but are not limited to, tees, cross tees, reducers, and elbows. A thermocouple compression fitting is also needed.

7.1.5 *Rotameters*—Four rotameters, each having a flow range bracketing the required flow rates for the detector, are needed to measure and dilute test chamber inlet vapor rates. Another rotameter is required for measuring outlet flow rates. A sixth rotameter is required for detectors that aspirate gas samples.

7.1.6 *Valves*—A shut-off valve and a flow controlling needle valve are needed.

Note 2—Gas shut off can be accomplished using a regulator and the integral shut-off valve commonly included with compressed gas cylinders. Many rotameters include an integral flow controlling needle valve.

7.1.7 *Thermocouple*—A thermocouple and temperature readout, or equivalent, that responds from 0°C to 40°C and is accurate to within 1°C over this range is needed.

7.1.8 *Manometer*—A relative pressure manometer is required to monitor the test chamber's internal pressure. The manometer must have a working range of at least 0-10 inches of water (0 to 20 mm Hg) with an accuracy of ±5%. The manometer should be scaled using at least 0.2 inches of water (0.4 mm Hg) subdivisions.

7.1.9 *Test chamber*--The test chamber should be gas-tight and made from materials that are inert with respect to test gases. Figure 2 contains a test chamber schematic representation. The test chamber must have fittings to allow connection to the detector probe, a manometer, and a thermocouple. The chamber must also have an inlet and outlet for flow of test atmospheres. Test chamber volumes should be kept as small as possible without interfering with detector operation. Diagrams of suitable test chambers are presented in Appendix A.

7.2 *Timer*--A timer that is accurate and precise to at least one second per 10 minutes is required. Alternatively, a chart recorder or other data acquisition system may be used. If a chart recorder or other data acquisition system is to be used, the timer is not required. If used, the recorder or data acquisition system timing must be accurate and precise to at least one second per 10 minutes.

7.3 *Electronic recorder*--A chart recorder or other electronic data acquisition system may be used if it is compatible with the specific detector that is being evaluated. The output of the data recorder should be accurate and precise to ±2% over the range of output from a quantitative detector. A data recorder used with a qualitative detector must unambiguously identify activated and inactivated states.

7.4 *Thermometer*--ASTM Solvents Distillation Thermometer having a range from -2°C to 52°C and conforming to the requirements for Thermometer 37C as prescribed in Specification E 1.

7.5 *Bubble meter*—Use NBS-traceable soap bubble flow meters to calibrate rotameters. The bubble meters must have a working range that brackets the range of all rotameters.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents for the American Chemical Society where such specifications are available.[1] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of determination.

8.2 *Test Gases*—Use factory-mixed benzene and 2-methylbutane gas standards in conventional gas cylinders as test chamber atmosphere sources. Make-up gas for all standards shall be air. All gas standards must be certified accurate (±2%). The gas standards required are listed in Table 1. Only the gas standards necessary to bracket the lower detection limit of the detector to be tested need to be acquired. (Danger—*Gases under high pressure. May be harmful if inhaled. See Annex A1.1 and Annex A1.2.)*

8.3 *Ultrahigh-Purity Air*—The ultrahigh-purity air standard must have less than 0.5 ppmv total hydrocarbon content. (Caution—*Gas under high pressure. See Annex A1.3.)*

9. Calibration and Standardization

9.1 *Chart Recorder or Other Data Recording System*—If used, a chart recorder or other data recording system should be calibrated along with the

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[1] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemicals Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., In., New York, NY, and the "United States Pharmacopeia."

detector. The data recording system should be calibrated according to instructions from its manufacturer and the detector manufacturer. Also, any recording device should be compatible with the detector being investigated. Consult specifications from the manufacturers of the recording device and the detector.

9.2 *Detector*—Because of wide design variability among different petroleum detectors, it is impossible to give complete calibration instructions for all possible detector designs. Calibrate all detectors according to manufacturer instructions.

9.3 *Rotameters*—Calibrate each rotameter prior to initiating test procedures, once a year thereafter, and after any internal contamination (e.g., dirt or moisture) during testing. Instructions accompanying the NBS-traceable bubble meters should be followed. The rotameters are to be calibrated at a minimum of five points (multipoints) within the working range. Flow readings should be made from the middle of the ball float. All readings should be made from the upper float on dual-float rotameters until it is off scale. Once the upper float is off scale, readings should be made from the lower float.

9.3.1 *Assembly*—Remove the rotameter from the test system, if assembled, and connect the rotameter in series between a controllable compressed air source and an NBS-traceable bubble meter. Use a bubble meter cylinder that will allow a flow measurement over a period of 15 to 45 seconds.

9.3.2 *First calibration point*—Bring the gas flow rate to the lowest calibration flow for testing. Let the system run at this setting until the rotameter is steady. If the flow rate is not within the first level range, adjust the flow rate until it is within that range and wait for a steady-state value. Record the steady-state flow meter value that is within the first flow rate level. Measure the flow rate with the bubble meter and stopwatch according to the NBS bubble meter instructions. Record each reference flow rate.

9.3.3 *Remaining calibration points*—Record steady-state settings for triplicate runs as described in Section 9.3.2 for at least four more flow rates throughout the rotameter range.

9.4 *Thermocouple*—Perform side-by-side multipoint calibrations for each thermocouple used in the test procedure in a 1-L glass beaker filled with water. The reference thermometer should be an ASTM Solvents Distillation thermometer having a range from -2°C to 52°C and conforming to the requirements for Thermometer 37C as prescribed in Specification E 1. The levels tested are low (room temperature - 10°C), room temperature, and high (room temperature + 10°C).

9.4.1 Insert both the thermocouple and reference thermometer into the beaker of water and add small quantities of ice. Allow the ice to melt and the temperature to stabilize. Continue adding ice until a steady-state reading (±0.5°C over two minutes) of room temperature - 10°C (±2°C) occurs.

9.4.2 Repeat this procedure using room temperature water (15°C to 30°C), and room temperature + 10°C (±2°C) water. If the temperature difference is more than 1°C, either repeat the test with the same thermocouple or replace the thermocouple and repeat the test until it is acceptable.

9.4.3 Perform thermocouple calibration at the onset of testing and at least once a year.

10. Conditioning

10.1 *Before Each Test*—Purge the test chamber for at least three minutes with ultrahigh-purity air at 0.2 L/min.

11. Procedure

11.1 *Test Series*--Quantitative detectors shall be tested seven times for each test product. Qualitative detectors shall be tested a total of twelve tests for each test gas, six tests at both the LDL and LDL- concentrations. If a qualitative detector fails to respond positively to one or more of the six LDL tests, then the LDL concentration shall be increased and six more tests shall be performed at the new LDL concentration. Similarly, if a detector responds positively six times to the LDL concentration and one or more times to the LDL- concentration, the LDL- concentration shall be decreased and six more tests shall be performed at the new LDL- concentration.

11.1.1 Perform tests in a random order such that variables of test gas and hydrocarbon concentration are isolated.

11.2 *Assembly*--Assemble the detection system as described by the manufacturer. Insert the detector probe into the test chamber. The seal between the probe and the test chamber should be gas-tight.

11.2.1 Connect the detector output to a chart recorder or other data acquisition system if one is being used. All connections should be in compliance with specifications from the manufacturers of the detector and the data recording system.

11.3 *Calibration*--Calibrate the detector if necessary. Many detectors do not require calibration. Perform calibrations, if necessary, according to manufacturer recommendations. Calibration may need to occur before mounting in the test container. If a data recording system is being used, it should be calibrated with the detector. Calibrate the data acquisition system according to manufacturer instructions.

11.4 *Background*—Supply ultrahigh-purity air to the test chamber at a rate that is 0.2 L/min. greater than the detector's aspiration rate.

Note 3—Many detectors are passive and do not aspirate gas samples. Use total test gas flow rates of 0.2 L/min for these detectors.

Monitor the detector's response every 15 seconds until a steady-state reading (±2% change of full scale over one minute) is achieved or four minutes has elapsed, whichever is longer. Monitor the temperature inside the test chamber during background testing.

11.5 *Identify LDL Concentration*—Based on previous testing or vendor information, identify the lowest hydrocarbon concentration to which the detector is known to respond. This test gas concentration is referred to as the lower detection limit, or "LDL." The LDL may be different for benzene and 2-methylbutane.

11.5.1 If the LDL test gas concentration is unknown, begin with the 1000 parts per million by volume (ppmv) concentration and then test at successively lower standard concentrations until the detector does not respond. The standard test gas concentrations are 1.25 ppmv, 2.5 ppmv, 5 ppmv, 12.5 ppmv, 25 ppmv, 50 ppmv, 125 ppmv, 250 ppmv, 500 ppmv, and 1000 ppmv.

11.6 *Test Atmosphere Response*—Introduce the appropriate test gas to the test chamber at a sufficient rate to produce a 0.2 L/min test system vent flow rate. Monitor the temperature inside the test chamber during testing.

11.6.1 Appropriate test gas concentrations and corresponding dilution factors are listed in Table 2. Consult Figure 3 for making test gas dilutions. Ultrahigh-purity air should not flow through either rotameter 2 or 4 (Figure 3) when a test gas dilution is not necessary. The flow of ultrahigh-purity air through rotameter 2 should equal the flow of test gas through rotameter 1 when a 50% test gas dilution is desired. Ultrahigh-purity air should not flow through rotameter 4 for 50% dilution. For 75% dilution of test gas, ultrahigh-purity air flow through rotameters 2 and 4 should equal test gas flow through rotameters 1 and 3, respectively.

11.6.2 Monitor quantitative detectors until a steady-state (±2% of full scale over 1 minute) response occurs or for 24 hours, whichever is shorter. Monitor qualitative detectors for a positive response ("activated") for up to 24 hours.

12. Calculation

12.1 *Critical Level for Quantitative Detectors*—Calculate the critical level for quantitative detectors as follows:

$$\text{Critical level, ppmv} = K \times s + B \tag{7}$$

where:

K = tolerance limit statistic from Table 3;

s = standard deviation (n-1 degrees of freedom) of the data set, ppmv; and

B = absolute bias = $|V_o - V_r|$, ppmv;

where:

V_r = the reference (theoretical) value, ppmv; and

V_o = average observed value = $1/n \sum_{i=1}^{n} V_i$, ppmv;

where:

V_i = individual response to a test gas or concentration, ppmv;

12.2 *Lower Detection Limit for Quantitative Detectors*—Calculate the lower detection limit for quantitative detectors as follows:

$$\text{Lower detection limit, ppmv} = B + 2 \times (K \times s) \quad (8)$$

where:

B = absolute bias, ppmv; and

K = tolerance limit statistic (from Table 3); and

s = standard deviation (n-1 degrees of freedom) of the data set, ppmv.

13. Interpretation of Results

13.1 *Lower Detection Limit for Qualitative Detectors*—The lower detection limit for qualitative detectors is between the LDL and LDL- concentrations. For detectors that respond at least one time to a test concentration of 1.25 ppmv, the LDL- concentration is less than 1.25 ppmv (< 1.25 ppmv). For detectors that fail to respond at 1000 ppmv, the LDL concentration is greater than 1000 ppmv (> 1000 ppmv).

14. Report

14.1 Report the following information:

14.1.1 *Detector type*—Report whether the detector was a quantitative or qualitative type.

14.1.2 *Lower Detection Limit*—Report the lower detection limit. For qualitative detectors, report the LDL and LDL- concentrations.

15. Precision and Bias

15.1 *Precision*—The precision of the procedure in Test Method X 0006 for measuring lower detection limit for vapor-phase out-of-tank petroleum detectors is being determined.

15.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in Test Method X 0006 for measuring lower detection limit for vapor-phase out-of-tank petroleum detectors, no statement on bias is being made.

Table 2. Vapor-Phase Lower Detection Limit Test Gas Concentrations

Test Gas Number	Test Gas Concentration, ppmv	Standard Test Gas Concentration, ppmv	Dilution With Air, %
1	1.25	5	75
2	2.5	5	50
3	5	5	0
4	12.5	50	75
5	25	50	50
6	50	50	0
7	125	500	75
8	250	500	50
9	500	500	0
10	1000	1000	0

Table 3. One-Sided Tolerance Limit Factors for a Five-Percent Beta Error at a Ninety-Five-Percent Confidence Level[A]

Number of Tests (n)	Tolerance Limit Factor (K)
3	7.655
4	5.145
5	4.202
6	3.707
7	3.399
8	3.188
9	3.031
10	2.911
11	2.815
12	2.736
13	2.670
14	2.614
15	2.566
16	2.523
17	2.486
18	2.453
19	2.423
20	2.396
21	2.371
22	2.350
23	2.329
24	2.309
25	2.292
30	2.220
35	2.166
40	2.126
45	2.092
50	2.065

[A] Taken from Natrella, M.G., Experimental Statistics. National Bureau of Standards Handbook 91. United States Department of Standards. Stock Number 003-003-00135-0. August, 1963. Reprinted October, 1966.

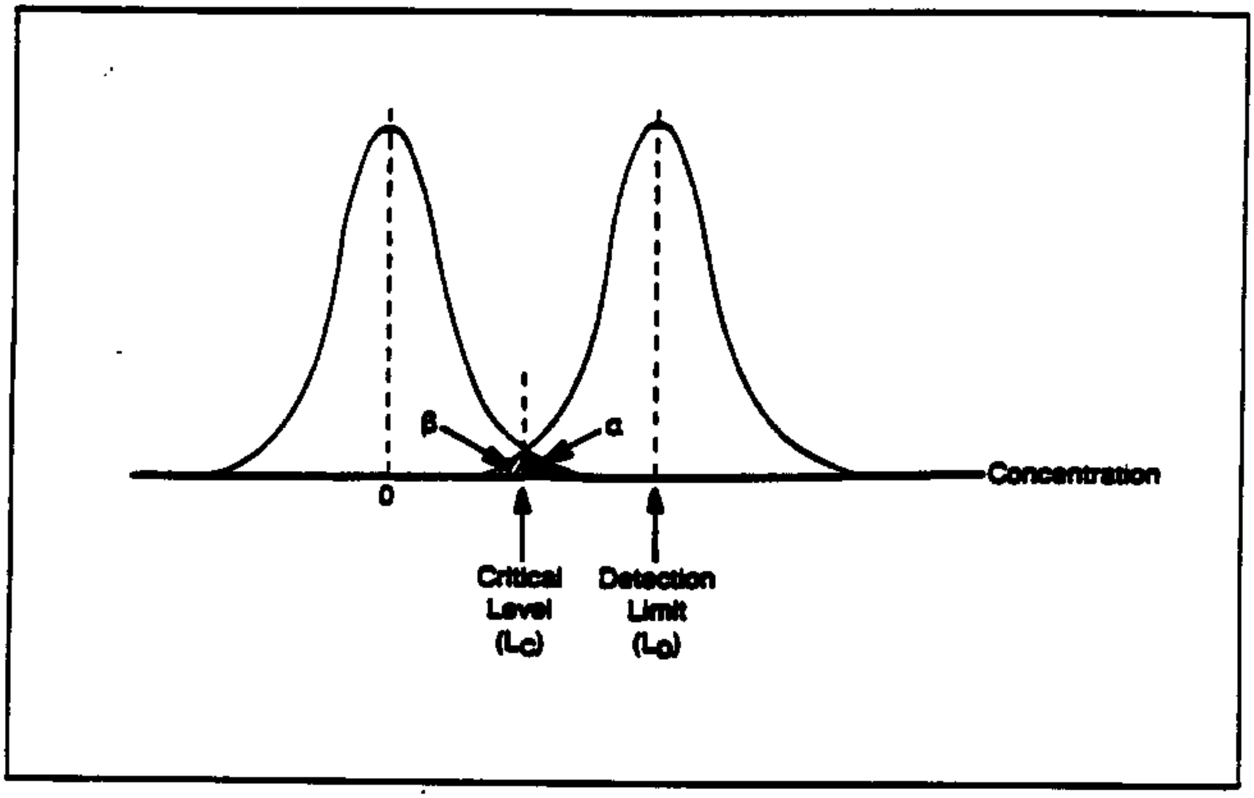

FIG. 1. Graphical Representation of Detection Limit and Critical Level

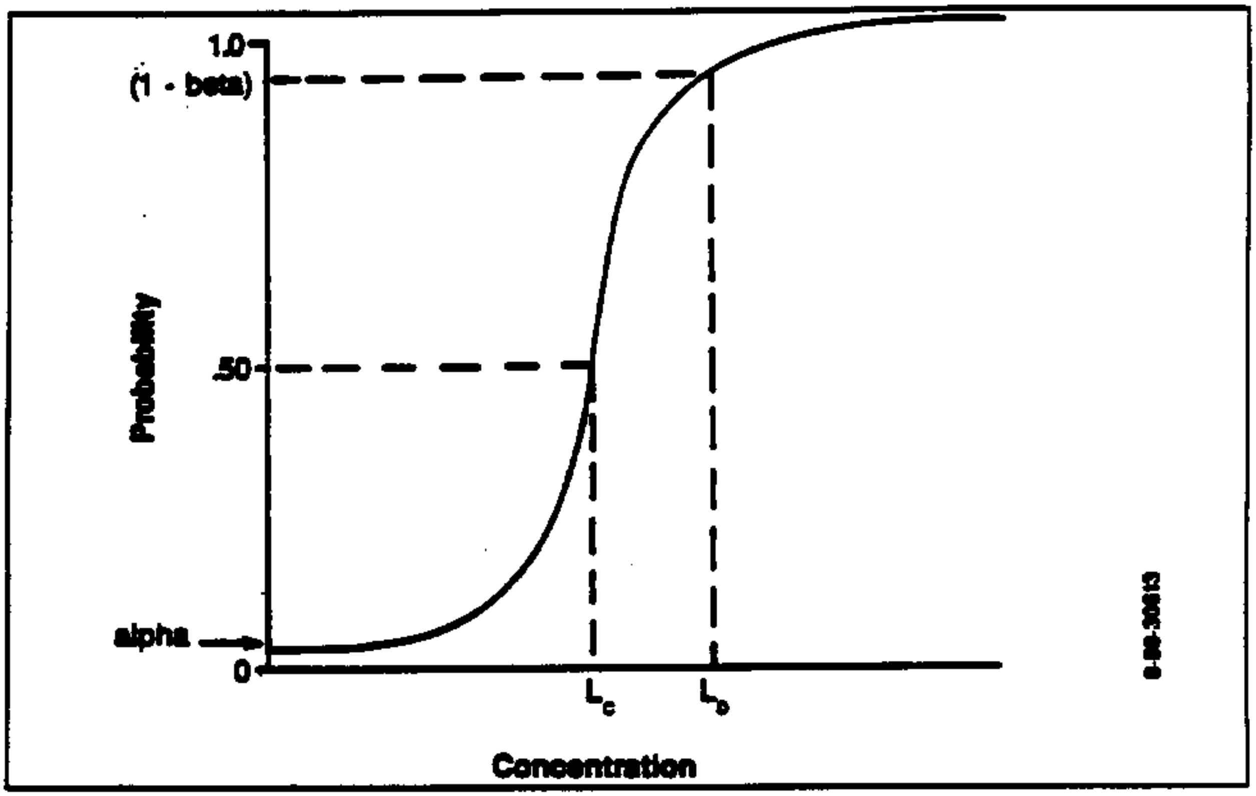

FIG. 2. Probability of Detection Versus Concentration

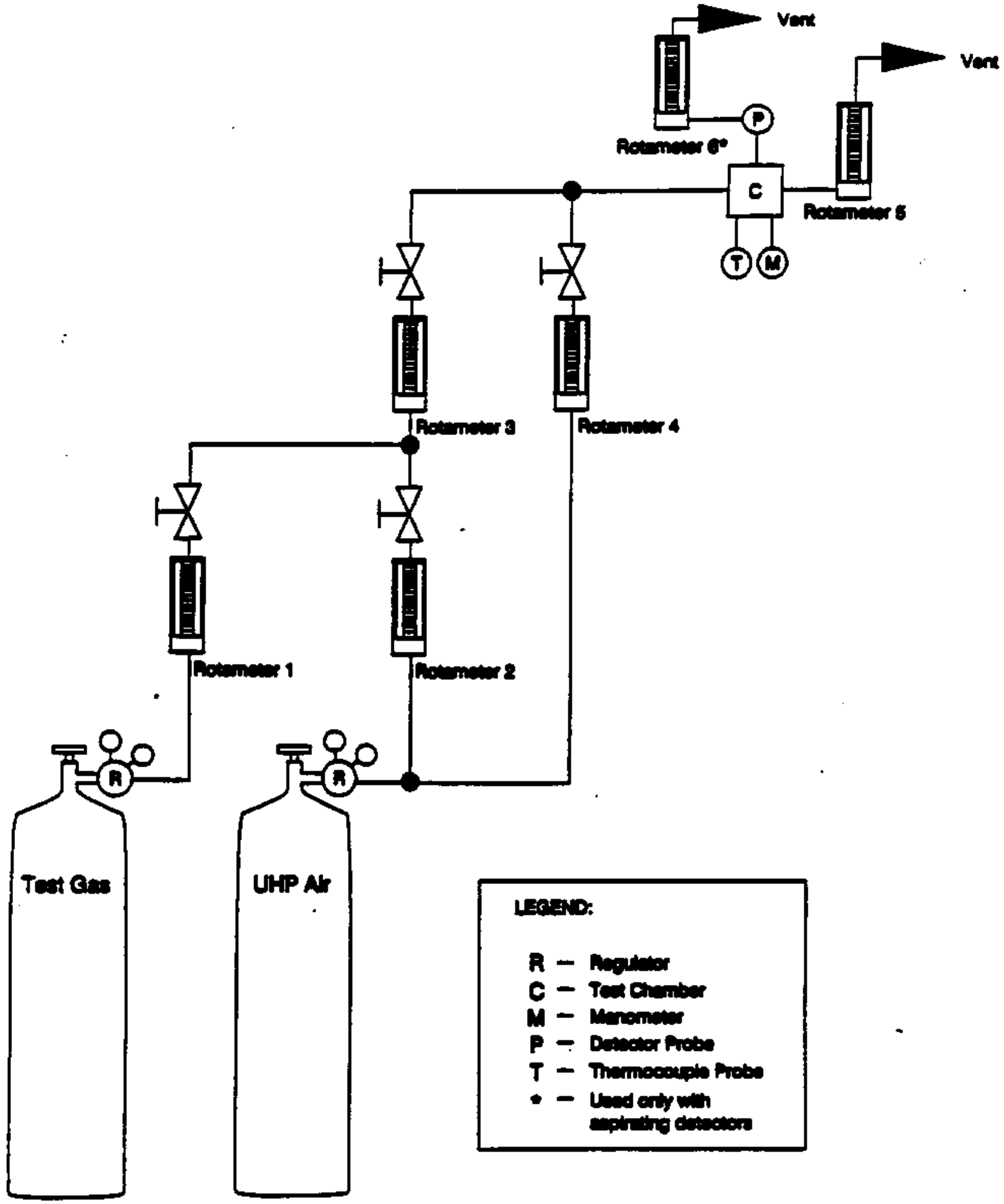

FIG. 3. Test Manifold Schematic Diagram

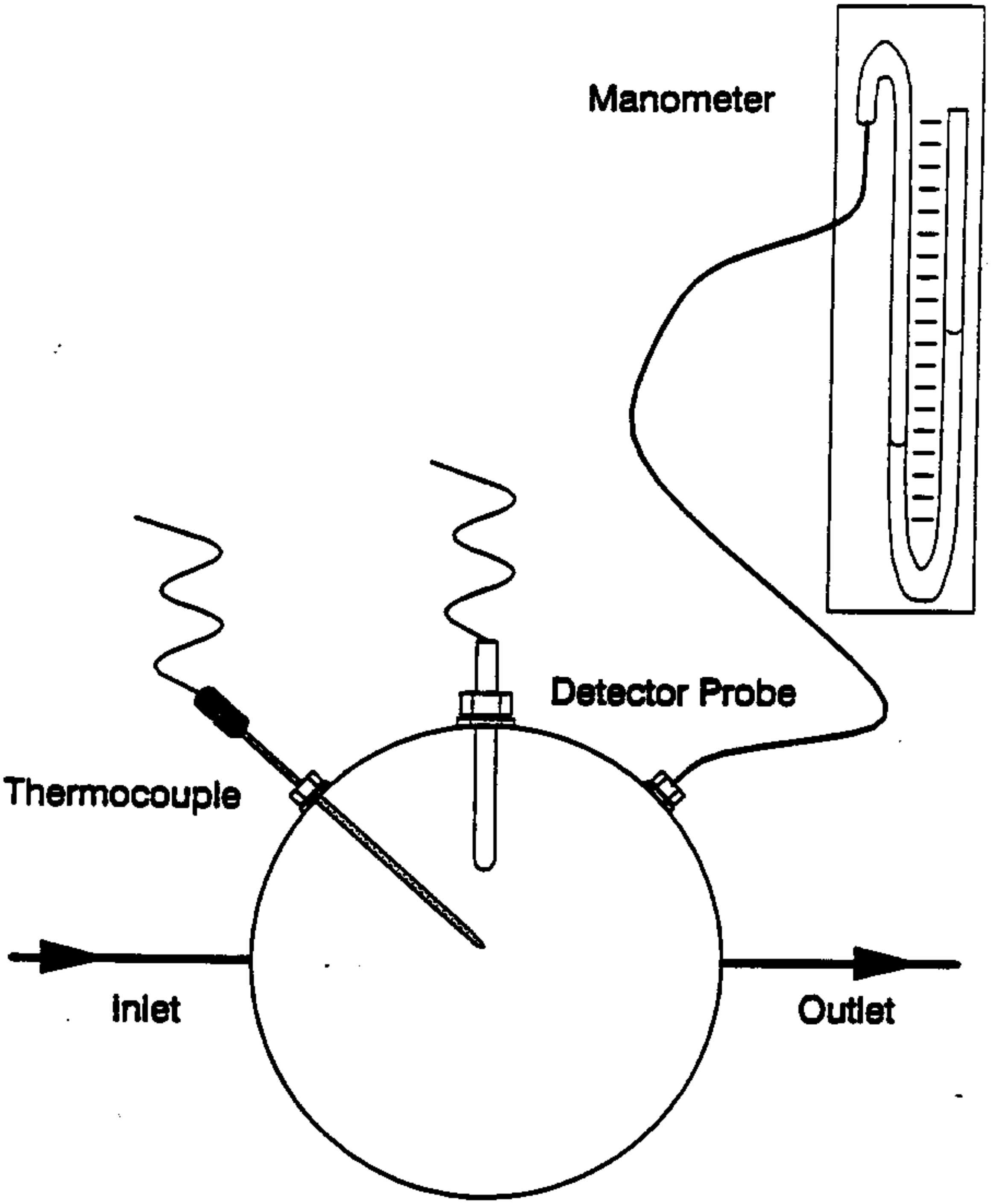

FIG. 4. Test Chamber Schematic Diagram

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENTS

A1.1 Compressed Gas. Benzene in Air.

Danger—Poison, Carcinogen.

Keep container closed.

Use fume hood whenever possible.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do no mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder out of sun and away from heat.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.2 Compressed Gas. 2-Methylbutane (Isopentane) in Air.

Warning—May be harmful if inhaled.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

A1.3 **Compressed Gas. Air.**

Caution—Compressed gas under high pressure.

Keep container closed.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder out of sun and away from heat.

Do not use cylinder without label.

Do not use dented or damaged cylinder.

For technical use only. Do not use for inhalation purposes.

APPENDIX

X1. EXAMPLE TEST CHAMBERS

X1.1 It is not reasonable to try to design a single test chamber that accommodates all configurations of detector probes. Instead, it is better to define the general requirements for the chamber and allow specific chambers to be designed to meet requirements for particular probes. Included in this appendix are diagrams of three test chambers that, combined, should be suited to the vast majority of vapor-phase detectors. The included chamber designs may also serve as starting points for alternate chamber designs.

X1.2 *Test Chamber for Tubing Probes*—Figure X1 contains a diagram detailing a test chamber design that should be suitable for almost all vapor-phase detectors that have probes consisting of tubing. These detectors primarily aspirate gas samples through tubing to a sensor located in the control box.

X1.3 *Test Chamber for Probes Up to 3/8-Inch Diameter*—Figure X2 is a diagram of a test chamber design that should be adequate for detector probes with diameters up to 3/8 inch.

X1.4 *Test Chamber for Probes Up to 1-1/4-Inch Diameter*—Figure X3 is a diagram of a test chamber design that should be adequate for detector probes with diameters up to 1-1/4 inches.

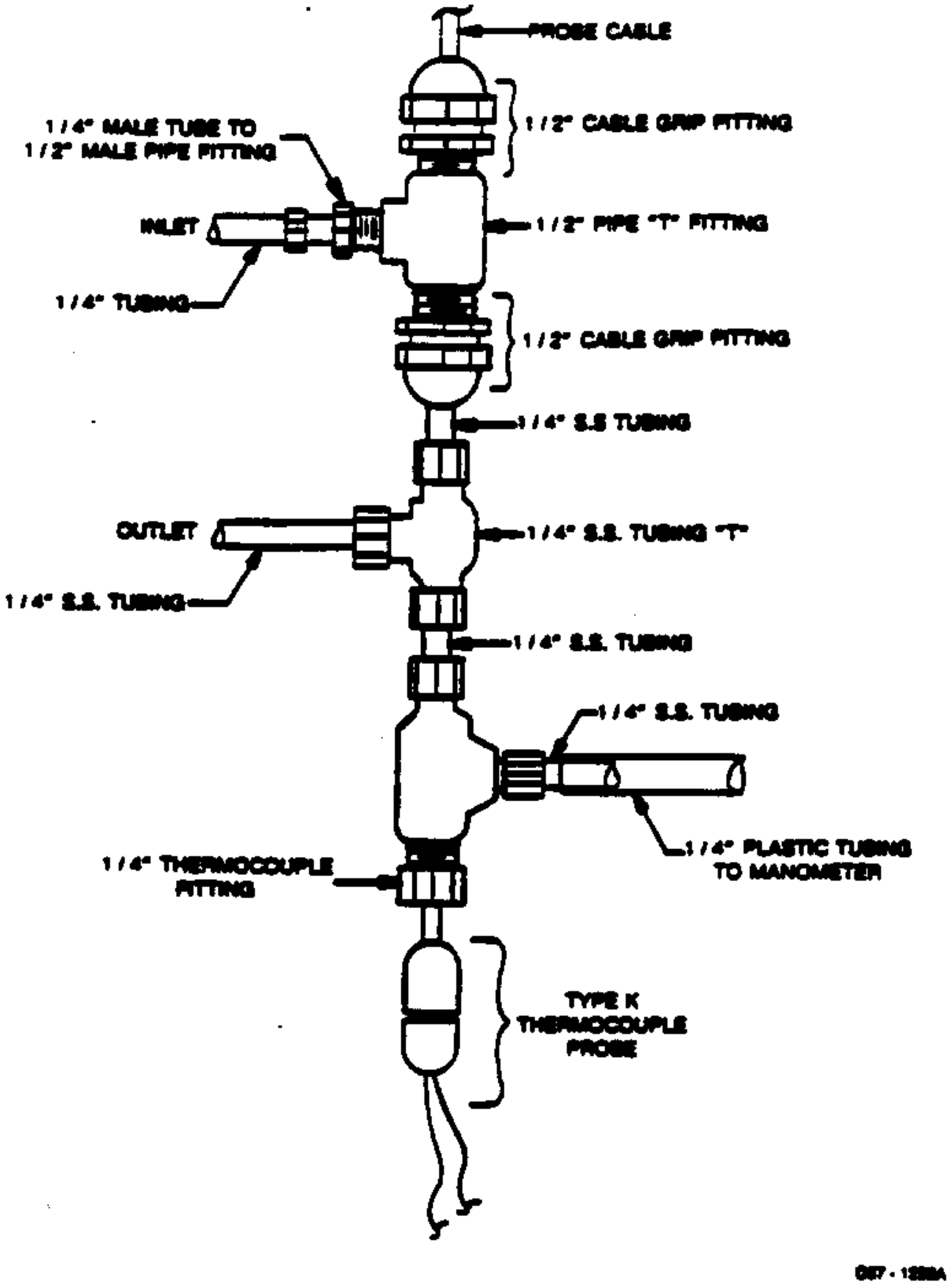

FIG. X2. Gas Test Chamber for Probes with Diameters Up To 3/8 Inch

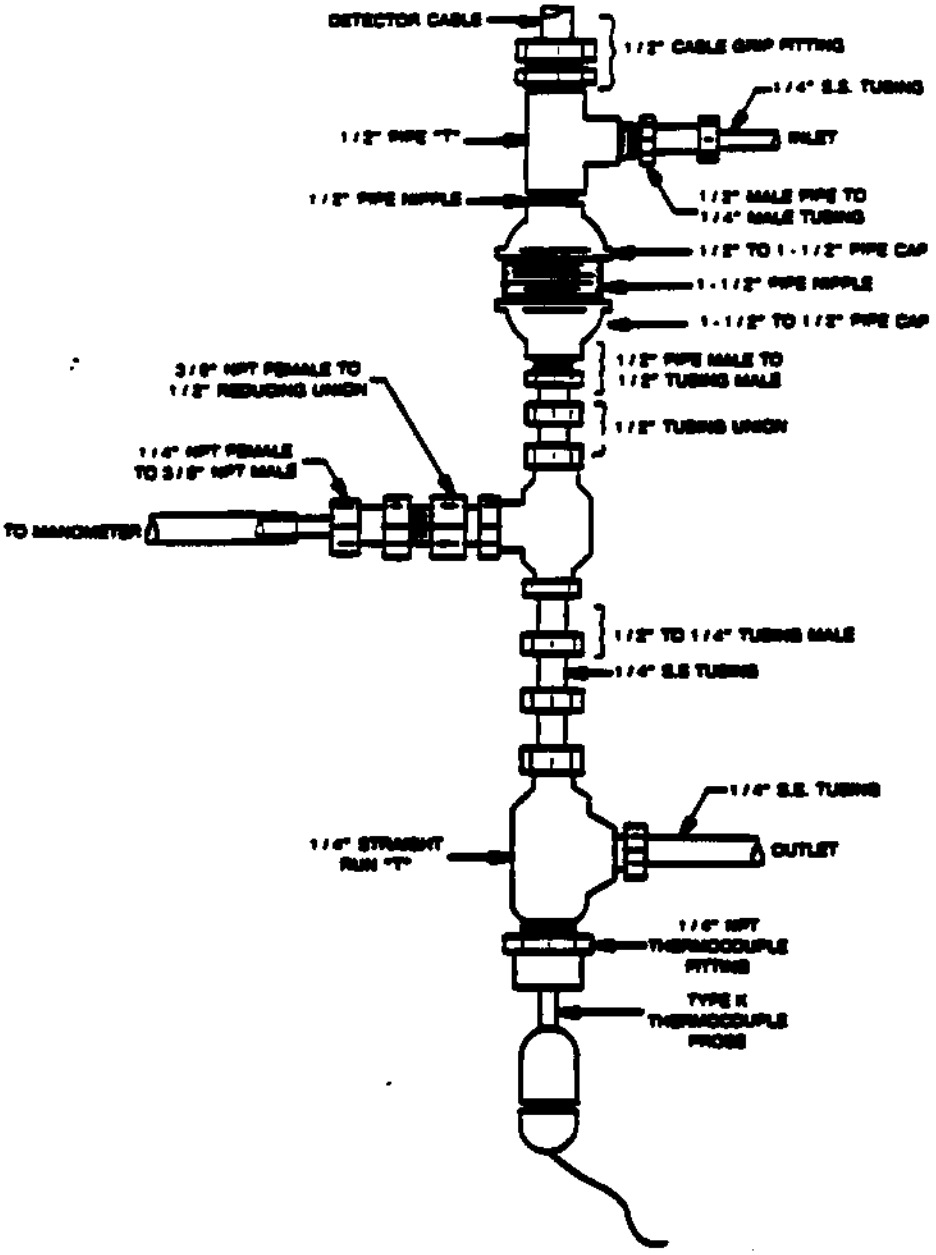

FIG. X3.. Gas Test Chamber for Probes with Diameters Up To 1-1/4 Inches

Results of U.S. EPA Standard Evaluation

Vapor-Phase Out-of-Tank Product Detectors

This form documents the performance of the vapor-phase product detector described below. The evaluation was conducted by the equipment manufacturer or a consultant to the manufacturer according to the U.S. EPA's "Standard Test Procedure for Evaluating Leak Detection Methods: Vapor-Phase Out-of-Tank Product Detectors."

Tank owners using this leak detection system should keep this form on file to prove compliance with the federal regulations. Tank owners should check with state and local agencies to verify that this form satisfies their requirements.

Method Description

Name ____________________

Version ____________________

Vendor ____________________

____________________ (street address)

____________________ (city) (state) (zip) (phone)

Detector output type: ☐ Quantitative ☐ Qualitative

Detector operating principle: ☐ Metal Oxide Semiconductor ☐ Adsistor ☐ Detector Tube ☐ Catalytic Gas Sensor ☐ Combustible Gas Detector ☐ Photoionization Detector ☐ Product-Permeable Detector ☐ IR Detector ☐ Other ____________________

Detector sampling frequency: ☐ Intermittent ☐ Continuous

Evaluation Results

The detector described above was tested for its ability to detect known concentrations of test gas. The following parameters were determined:

Accuracy - How closely test gas concentration, as measured by the detector, agrees with the actual gas concentration.

Bias - Whether the method consistently over-estimates or under-estimates gas concentration. Not applicable to qualitative detectors.

Precision - Agreement between multiple measurements of the same gas concentration. Not applicable to qualitative detectors.

Detection Time - Amount of time the detector must be exposed to test gas before it responds.

Fall Time - Amount of time that passes before the detector returns to its baseline reading after test gas is removed.

Lower Detection Limit - The smallest gas concentration that the detector can reliably detect.

Specificity - Indicates the ability of the detector to detect several different test gases.

Vapor-Phase Product Detector ______________________________
Version ____

Evaluation Results (continued)

> **Compiled Test Results** (for tests conducted with 1000 ppm of test gas)

Test	Benzene	2-Methylbutane
Accuracy (%)		
Bias* (%)		
Precision* (%)		
Detection Time (hh:mm:ss)		
Fall Time (hh:mm:ss)		
Lower Detection Limit (ppm)		

* Not applicable to qualitative detectors.

> **Specificity Results (%)**

Benzene	
n-Butane	
n-Hexane	
Isobutane	
2-Methylpentane	
Toluene	

> **Safety disclaimer: This test procedure only addresses the issue of the method's ability to detect leaks. It does not test the equipment for safety hazards.**

Certification of Results

I certify that the vapor-phase product detector was operated according to the vendor's instructions and that the evaluation was performed according to the standard EPA test procedure for vapor-phase out-of-tank product detectors except as noted on any attached sheets. I also certify that the results presented above are those obtained during the evaluation.

(printed name)	(organization performing evaluation)
(signature)	(city, state, zip)
(date)	(phone number)