RESOLUTION NO. 540

RESOLUTION OF THE BOARD OF DIRECTORS OF THE PUGET SOUND AIR POLLUTION CONTROL AGENCY ADOPTING MODIFIED PARTICULATE SOURCE TEST PROCEDURES

WHEREAS, Regulation I Section 9.09(f) requires procedures for source sampling performed in connection with standards of Regulation I and II for particulate and gases to be done using current Environmental Protection Agency requirements or procedures and definitions adopted by the Board; and

WHEREAS, to conform to current safe and less toxic chemical storage, the particulate measurement procedures currently used by the Agency have been proposed for modification; and

WHEREAS, the Expanded Advisory Council reviewed and approved said source test laboratory procedure modifications; and

WHEREAS, a public hearing was held by the Puget Sound Air Pollution Control Agency Board of Directors on August 11, 1983, to allow public input and critique on the proposal; and

WHEREAS, the Board deems it necessary to adopt said modification to source test procedures; now therefore,

BE IT RESOLVED BY THE BOARD OF PUGET SOUND AIR POLLUTION CONTROL AGENCY:

The Board of Directors does hereby adopt the modifications to the source test procedures, a copy of which is attached hereto and made a part hereof.

PASSED AND APPROVED by the Board of Directors of the Puget Sound Air Pollution Control Agency held this 11th day of August, 1983.

PUGET SOUND AIR POLLUTION CONTROL AGENCY

By ______________________
Chairman

Attest:

____
Air Pollution Control Officer

Approved as to form:

____
Agency Attorney

Proposed Revised PSAPCA
Particulate Source Test Procedures

Engineering Division

Puget Sound Air Pollution Control Agency
200 West Mercer Street, Room 205
P.O. Box 9863
Seattle, Washington 98109

June 9, 1983

I. Procedures for Particulate Source Sampling

Unless otherwise authorized by the Control Officer, all particulate source sampling performed to demonstrate compliance with the emission standards of Regulation I shall be done using current Environmental Protection Agency Methods 1-5 contained in 40 CFR Part 60, Appendix A, as modified in Section II of this document.

II. Procedure for Determining Particulate Matter in the Impinger Catch (Back Half)

The analysis and calculations for Method 5 shall conform to that described by EPA in the current 40 CFR Part 60, Appendix A, except that the back half catch shall be included as particulate matter. The back half weight is the sum of the impinger catch (organic and inorganic) and the back half acetone rinse weights.

A. Sample Recovery of the Back Half

1. Purging

Whenever SO_2 interference is suspected, purge the impingers immediately after the test run is complete with N_2 or clean air for a minimum of one-half the sample volume.

2. Impinger Liquid

Measure the volume of water collected in all impingers and place the water from the first three impingers in a container. Thoroughly rinse all sample-exposed surfaces between the filter and fourth impinger with water and place in above container.

3. Acetone Rinse

Thoroughly rinse all sample-exposed surfaces between the filter and the fourth impinger with acetone and place the washings in a tared beaker to dry.

B. Analysis of the Back Half

1. Impinger Liquid Extraction

a. Add 50-100 mℓ of dichloromethane to the impinger liquid.

b. Spin for at least ten minutes.

c. Pour the liquid into a separatory funnel and drain the organic phase into a tared beaker (organic fraction).

d. Drain the remaining liquid into a beaker and repeat Steps a, b, and c. Perform the extraction several times with fresh dichloromethane until the organic fraction is clear. Keep each organic extraction in a separate beaker.

e. Following the last extraction, drain the remaining liquid from the separatory funnel into a tared beaker (inorganic fraction).

f. Allow the organic fraction beakers to dry under a hood at room temperature.

g. Evaporate the inorganic fraction in such a manner that the beaker contents do not become exposed to temperatures greater then 212°F.

h. Dry weighed beakers containing a sample of the acetone, dichloromethane and a sample of distilled deionized water to check for blank weight.

i. Desiccate organic, inorganic and blank beakers for at least 24 hours at room temperature in a disiccator containing silica gel. Weigh to a constant weight and report the results to the nearest 0.1 mg. Constant weight is defined in Section 4.3 of Method 5.

2. Back Half Acetone Rinse

a. Dry the acetone rinse in a hood at room temperature.

b. Desiccate and weigh the beaker to constant weight and record.

C. Reagents

1. Water

Use distilled deionized water in the impingers and to rinse all glassware.

2. Acetone

Use reagent grade, $\leq$ 0.001 percent residue in glass bottles.

3. Dichloromethane

Use reagent grade, $\leq$ 0.001 percent residue in glass bottles.

Revised July 12, 1990

STATE OF WASHINGTON DEPARTMENT OF ECOLOGY

SOURCE TEST METHOD 9A

VISUAL DETERMINATION OF OPACITY FOR A THREE MINUTE STANDARD

1. Principle

The opacity of emissions from stationary sources is determined visually by a qualified observer.

2. Procedure

The observer must be certified in accordance with the provisions of Section 3 of 40 CFR Part 60, Appendix A, Method 9, as in effect on July 1, 1990, which are hereby adopted by reference.

The qualified observer shall stand at a distance sufficient to provide a clear view of the emissions with the sun oriented in the 140° sector to his back. Consistent with maintaining the above requirement, the observer shall, as much as possible, make his observations from a position such that his line of vision is approximately perpendicular to the plume direction, and when observing opacity of emissions from rectangular outlets (e.g., roof monitors, open baghouses, noncircular stacks), approximately perpendicular to the longer axis of the outlet. The observer's line of sight should not include more than one plume at a time when multiple stacks are involved, and in any case, the observer should make his observations with his line of sight perpendicular to the longer axis of such a set of multiple stacks (e.g., stub stacks on baghouses).

The observer shall record the name of the plant, emission location, type of facility, observer's name and affiliation, and the date on a field data sheet. The time, estimated distance to the emission location, approximate wind direction, estimated wind speed, description of the sky condition (presence and color of clouds), and plume background are recorded on a field data sheet at the time opacity readings are initiated and completed.

The observer should make note of the ambient relative humidity, ambient temperature, the point in the plume that the observations were made, the estimated depth of the plume at the point of observation, and the color and condition of the plume. It is also helpful if pictures of the plume are taken.

Opacity observations shall be made at the point of greatest opacity in the portion of the plume where condensed water vapor is not present. The observer shall not look continuously at the plume, but instead shall observe the plume momentarily at 15-second intervals.

When condensed water vapor is present within the plume as it emerges from the emission outlet, opacity observations shall be made beyond the point in the plume at which condensed water vapor is no longer visible.

When water vapor in the plume condenses and becomes visible at a distinct distance from the emission outlet, the opacity of emissions should be evaluated at the emission outlet prior to the condensation of water vapor and the formation of the steam plume.

Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on an observational record sheet. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period.

3. Analysis

The opacity of the plume is determined by individual visual observations. Opacity shall be reported as the range of values observed during a specified time period, not to exceed 60 consecutive minutes. The opacity standard is exceeded if there are more than 12 observations, during any consecutive 60-minute period, for which an opacity greater than the standard is recorded.

4. References

Federal Register, Vol. 36, No. 247, page 24895, Dec. 23, 1971.

"Criteria for Smoke and Opacity Training School 1970-1971" Oregon-Washington Air Quality Committee.

"Guidelines for Evaluation of Visible Emissions" EPA 340/1-75-007.

ETHYLENE OXIDE

1614

$CH_2(O)CH_2$ MW: 44.05 CAS: 75-21-8 RTECS: KX2450000

METHOD: 1614, Issue 2

EVALUATION: FULL

Issue 1: 15 August 1987
Issue 2: 15 August 1994

OSHA : 1 ppm
NIOSH: 0.1 ppm; C 5 ppm/10 min; carcinogen; Group I Pesticide
ACGIH: 1 ppm; suspect carcinogen

PROPERTIES: gas; d (liquid) 0.8694 g/mL @ 20 °C; BP 10.7 °C; MP -111 °C; explosive limits 3 to 100% (v/v) in air

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SYNONYMS: 1,2-epoxyethane; oxirane

SAMPLING

SAMPLER: SOLID SORBENT TUBE (HBr-coated petroleum charcoal, 100 mg/50 mg)

FLOW RATE: 0.05 to 0.15 L/min

VOL-MIN: 1 L @ 5 ppm
-MAX: 24 L

SHIPMENT: routine

SAMPLE STABILITY: 90% recovery after 17 days @ 25 °C in the dark [3]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.04 to 0.98 ppm (24-L samples) [1]

BIAS: -6.9% [1]

OVERALL PRECISION ($\hat{S}_{rT}$): 0.062 [1]

ACCURACY: ±19%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, ECD

ANALYTE: 2-bromoethylheptafluorobutyrate

DESORPTION: 1 mL dimethylformamide; stand 5 min

INJECTION VOLUME: 1 µL

TEMPERATURE-INJECTION: 200 °C
-DETECTOR: 300 °C
-COLUMN: 100 °C

CARRIER GAS: 5% CH_4 in Ar, 25 mL/min

COLUMN: 3 m x 4 mm glass; 10% SP-1000 on 80/100 Chromosorb WHP

CALIBRATION: standard solutions of 2-bromoethanol in dimethylformamide

RANGE: 2 to 42 µg ethylene oxide per sample

ESTIMATED LOD: 1 µg EtO per sample [2]

PRECISION ($\hat{S}_r$): 0.020 @ 18 to 71 µg EtO per sample [1]

APPLICABILITY: The working range is 0.05 to 4.6 ppm (0.08 to 8.3 mg/m^3) for a 24-L air sample. The method is applicable to short-term (10-min) samples.

INTERFERENCES: 2-Bromoethanol, if present in the sample interferes. No other significant interferences have been found [1].

OTHER METHODS: This is a modification of OSHA Method 50 [1] and replaces NIOSH method 1607, which has smaller sample capacity, and S286 [3], which is useful for higher levels. Method 3702 (Ethylene Oxide by Portable GC) describes field-readable gas chromatography for the determination of ethylene oxide.

REAGENTS:

1. *N,N*-Dimethylformamide (DMF), high purity.
2. Isooctane (2,2,4-trimethylpentane), reagent grade.
3. Benzene, reagent grade.*
4. *N*-Heptafluorobutyrylimidazole (HFBI;*N*-heptafluorobutanoylimidazole; Alfa Chemical #31594, Alfa Products, Ward Hill, MA 01835).
5. Water, high purity (e.g., deionized distilled).
6. 2-Bromoethanol (2-FrEt), 95 to 98%.
7. Sodium hydroxide (NaOH), reagent grade.
8. Potassium hydrogen phthalate (KHP), reagent grade, dried at 90 °C.
9. HCl, conc., reagent grade.*
10. Pyridine, reagent grade.
11. Phenolphthalein, 1% (w/v) in ethanol or methanol.
12. Hydrobromic acid (HBr), 48%, reagent grade.*
13. Charcoal, petroleum-based (SKC Lot 208 or equivalent, SKC, Eighty-Four, PA).
14. Ethylene oxide (EtO), liquid or gas, 99.7% purity.*
15. Sodium hydroxide, 0.5 N. Dissolve 20 g NaOH in distilled water to make 1 L of solution. Standardize with KHP to a phenolphthalein endpoint (APPENDIX B).
16. HCl in pyridine, 0.2 N. Dissolve 8.5 mL conc. HCl in pyridine to make 500 mL solution. Standardize (APPENDIX C).
17. Ethylene oxide stock solution,* ca. 40 µg/µL. Bubble EtO gas (or pipet 4 mL EtO liquid) into 85 mL benzene in a graduated cylinder until solution volume increases ca. 4 mL. Seal tightly and store in freezer. Standardize weekly (APPENDIX D).
18. HFBI, 2% in isooctane. Dissolve 2 mL HFBI in isooctane to make 100 mL solution. Store in refrigerator.
19. 5% Methane in argon.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 6-mm OD x 4-mm ID x 45 mm glass tube, flame-sealed, with plastic caps, contaiing HBr-coated charcoal (see APPENDIX A), two sections, 100-mg front, 50-mg back, separated and contained with silanized glass wool plugs. Tubes are commercially available (Supelco Orbo-78, SKC 226-38-03, or equivalent.
2. Gas chromatograph, electron capture detector, integrator, and column (p. 1614-1).
3. Burette, 50-mL, 0.1-mL graduations.
4. Flask, round-bottom, 100-mL, with ground-glass joint.
5. Condenser, reflux, with ground-glass joint to fit round-bottom flask.
6. Heating mantle, with heat control, to fit round bottom flask.
7. Graduated cylinder, glass, 100-mL, with stopper that seals tightly.
8. Vials, glass, 5-mL, with PTFE-lined screw caps.
9. Pipets, volumetric, 2- to 500-µL and 1-, 2-, 4-, and 40-mL, with pipet bulb.
10. Syringes.
11. Vials, glass, 2-mL, with PTFE-lined septum screw caps.
12. Flasks, Erlenmeyer, 125-mL.
13. Gloves.
15. Fume hood.
16. File, triangular.
17. Evaporator, rotary.

SPECIAL PRECAUTIONS: Ethylene oxide and benzene are toxic and serious fire and explosion hazards; they are also suspect carcinogens [4-6]. Hydrobromic and hydrochloric acids are eye, skin, and inhalation hazards [6]. Work with these substances only in a hood. Use protective gloves.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break ends of sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.05 and 0.15 L/min for a total sample size of 1

to 24 L.

4. Cap each tube. Pack securely for shipment.

SAMPLE PREPARATION:

5. Score each sampler with a file. Break sampler at score line.
6. Transfer front sorbent section and first glass wool plug to a 2-mL vial. Transfer middle glass wool plug and back sorbent section to a separate vial.
7. Add 1.0 mL DMF to each vial. Cap each vial.
8. Shake at least 10 sec. Allow to stand at least 5 min.
9. Pipet a 20-µL aliquot of the DMF solution to another 5-mL vial containing 2.0 mL 2% HFBI (v/v) in isooctane.
   NOTE: Pipet 20 µL of each working standard (step 13) into vials containing 2% HFBI at this step.
10. Cap and shake 1 min: Allow to stand at room temperature at least 5 min.
11. Add 2.0 mL high purity water. Mix 1 min to ensure complete hydrolysis of excess HFBI.
12. Transfer at least 1.0 mL of isooctane (top) layer to 2-mL vial.
    NOTE: If the sample concentration is higher than the standards, dilute an aliquot of sample with DMF, reanalyze starting at step 9, and apply the appropriate dilution factor in calculations. The desorbed sample (in DMF) keeps well in a freezer [1].

CALIBRATION AND QUALITY CONTROL:

13. Calibrate daily with at least six working standards over the range 1 to 42 µg EtO per sample. For example:
    a. Pipet 1.0 mL 2-BrEt (density 1.763 g/mL @ 20°C) into a 10-mL volumetric flask. Dilute to volume with DMF. This is stock solution "A." Dilute 3 mL of "A" to 25 mL with DMF to give standard "B." Dilute 1 mL of "B" to 10 mL with DMF to give standard "C."
       NOTE: A sample calculation with 95% pure 2-BrEt (MW = 124.98) expressed as its equivalent weight in EtO (MW = 44.05) is:

       Standard Solution "A":

$$\frac{1.763\ \text{g}\ \ 2\text{-BrEt}}{10\ \text{mL}} \cdot 0.95 \cdot \frac{44.05}{124.97} \cdot 10^6\ \mu\text{g} = 59{,}036\ \mu\text{g EtO/mL.}$$

       Standard Solution "B":

$$3\ \text{mL} \cdot 59{,}030/25\ \text{mL} = 7084\ \mu\text{g EtO/mL.}$$

       Standard Solution "C":

$$1\ \text{mL} \cdot 7084/10\ \text{mL} - 708.4\ \mu\text{g EtO mL.}$$

    b. Prepare working standards by injecting microliter volumes of "B" and "C" into vials containing 1.0 mL DMF. The following serial dilution scheme is suggested:

Working Standard	Aliquot added to 1.0 mL DMF	Final µg EtO/mL
D	2.5 µL "C"	1.77
E	5.0 µL "C"	3.54
F	10.0 µL "C"	7.08
G	2.5 µL "B"	17.7
H	3.5 µL "B"	24.8
I	5.0 µL "B"	35.4
J	6.0 µL "B"	42.5

       Higher standards may be used if detector output remains acceptable. (Some detectors may become saturated at higher levels.)
    c. Analyze working standards together with samples and blanks.
    d. Prepare calibration graph (µg EtO per sample vs. peak area). Use a nonlinear (e.g.,

parabolic) least squares fit if necessary to obtain the best fit of the data.

14. Determine recovery (R) at least once for each lot of charcoal used for sampling in the concentration range of interest. Prepare three tubes at each concentration of interest plus three media blanks.
    a. Remove and discard back sorbent section of a media blank sampler.
    b. Inject a known amount (2 to 15 µL) of EtO stock solution or a serial dilution thereof directly onto the charcoal with a microliter syringe.
    c. Cap the tube. Allow to stand overnight.
    d. Desorb (steps 5 through 8) and analyze together with working standards (steps 9 through 12 and 16 and 17).
15. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

16. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1614-1. Inject sample aliquot manually using solvent-flush technique or with autosampler.
17. Measure peak area.

CALCULATIONS:

18. Determine the mass, µg (corrected for R), of EtO found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.
    NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
19. Calculate concentration, C, of EtO in the air volume sampled, V(L):

$$C = \frac{(W_f + W_b - B_f - B_b)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

This method uses HBr-coated charcoal to collect EtO and rapidly convert it to 2-bromoethanol. This method was evaluated at the OSHA Analytical Laboratory, Salt Lake City, UT, as OSHA Method 50 [1]. Fifteen-min and 30-min air samples were collected from a constant 5-ppm test atmosphere (80% relative humidity, ambient temperature) at 0.1 L/min with observed recoveries in the range 88 to 100%.

Fifteen pairs of side-by-side area samples were collected and analyzed by OSHA using this method and the Qazi-Ketcham method. The sampling rate was ca. 50 mL/min for 4 to 7.5 H. Results showed no statistical difference in the two methods, with no bias over the range 0.3 to 7 ppm EtO. test atmospheres of 0.1, 0.5, 1.0, and 16 ppm at 70 to 80% relative humidity and ambient temperature were sampled for 4 h at 0.1 L/min with no breakthrough. The 5% breakthrough volume for sampling a 16-ppm atmosphere of EtO at 0.15 L/min was 39 L. No significant storage effects were observed for samples in the 0.1 to 16 ppm range at high humidity and stored at ambient temperature for a minimum of two weeks.

The precision, S_r, of chromatographic response of working standards in the range 18 to 71 µg EtO per sample was 0.028 [1]. Recovery of EtO spikes in NIOSH laboratories averaged 75% at 11, 22, 33, and 44 µg EtO per sample.

REFERENCES:

[1] Cummins, K.J. OSHA Method No. 50, "Ethylene Oxide," OSHA Analytical Laboratory, Salt Lake City, UT (unpublished, January, 1985).

[2] User check, DataChem, Inc., NIOSH Seq. #5860-J (unpublished, May 15, 1987).

[3] NOSH Manual of Analytical Methods, 2nd. ed., V. 3, S286, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157C (1977); also cited in NIOSH Special Occupational Hazard

Review with Control Recommendations - Use of Ethylene Oxide as a Sterilant in Medical Facilities, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-200 (1977).
[4] Threshold Limit Values and Biological Exposure Indices for 1993-94, ACGIH, Cincinnati, OH (1993).
[5] NIOSH Testimony to USDOL at OSHA rulemaking hearing for ethylene oxide (July 20, 1983); also see Current Intelligence Bulletin 35, "Ethylene Oxide," U.S. Department of Health and Human Services, Publ. (NIOSH) 81-130 (May 22, 1981).
[6] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services Publ. (NIOSH) 81-123 (1981), available as GPO stock #17-033-00337-8 from Superintendent of Documents, Washington, D.C. 20402.
[7] Siggia, S. and J.G. Hanna. Quantitative Organic Analysis via Functional Groups, 4th ed., Chapter 5, John Wiley & Sons, Inc., New York, NY (1979).

METHOD REVISED BY:

George Williamson, NIOSH/DPSE.

APPENDIX A: PREPARATION OF SOLID SORBENT

Slowly add a mixture of 25 mL 48% hydrobromic acid and 125 mL acetonitrile to 75 g petroleum-based charcoal (SKC Inc., Lot 208) contained in a 500-mL round-bottom flask. Allow the slurry to cool to room temperature. Dry the coated charcoal by rotary evaporation using gentle heat and keep it overnight under vacuum at ambient temperature. The product is stable for four months when stored in a tightly sealed amber glass jar at room temperature [1]. Recovery may be lower as the sample medium ages.

APPENDIX B: STANDARDIZATION OF NaOH

1. Transfer duplicate, accurately weighed, ca. 1.5-gram portions of KHP (W, mg) to separate 125-mL Erlenmeyer flasks. Dissolve in 20 to 30 mL distilled water, warming if necessary.
2. Add one drop phenolphthalein solution to each flask.
3. Fill the burette with 0.5 N NaOH. Titrate, with constant mixing, to a pink endpoint in each flask. Record the volume, V (mL), of 0.5 N NaOH used.
4. Calculate the normality, N_b, of the NaOH solution.

$$N_b = \frac{W}{(204.22 \cdot V)}.$$

APPENDIX C: STANDARDIZATION OF HCl IN PYRIDINE

1. Pipet 40.0 mL 0.2 N HCl in pyridine into a 100-mL round-bottom flask.
2. Add 4.0 mL benzene.
3. Attach the flask to a water-cooled condenser and heat flask with a heating mantle to boiling. Reflux for 20 min.
4. Cool to near room temperature. Add 5 mL distilled H_2O through condenser and collect washings in the flask.
5. Remove reflux condenser and add a drop of phenolphthalein solution to the flask.
6. Titrate, with mixing, 0.5 N NaOH from the burette to the flask to a pink endpoint. Record volume, V_b (mL), of NaOH used.
7. Repeat steps 1 through 7 and average the results. Calculate the normality, N_a of the HCl-pyridine solution using N_b from APPENDIX B:

$$N_a = \frac{N_b V_b}{40}.$$

APPENDIX D: STANDARDIZATION OF ETHYLENE OXIDE STOCK SOLUTION [7]

1. Pipet 20.0 mL standardized HCl-pyridine solution into a 100-mL round bottom flask.
2. Add 2.0 mL EtO stock solution.
3. Attach the flask to the condenser. Reflux for 20 min.
   NOTE: The reaction is: EtO + HCl(xs) → chloroethanol. The excess HCl is back-titrated in step D-6.
4. Cool to near room temperature. Add 5 mL distilled H_2O through the condenser, collecting washings in the flask.
5. Remove the condenser and add a drop of phenolphthalein solution to the flask.
6. Titrate, with mixing, 0.5 N NaOH from the burette to a pink endpoint. Record volume, V_c (mL), of NaOH used.
7. Calculate the normality, N_s, of the excess HCl using N_b from APPENDIX B:

$$N_s = \frac{N_b V_c}{20\ \text{mL}}.$$

8. Repeat steps 1 through 7 to obtain an average of two titrations. Results should agree to within 1%. Rinse flask thoroughly before reuse.
9. Calculate the concentration of the EtO stock solution:

$$\frac{\text{mg EtO}}{\text{mL EtO stock solution}} = (N_o - N_s) \cdot \frac{20\ \text{mL}}{10^3\ \text{mL}} \cdot \frac{44.05\ \text{g}}{1\ \text{equivalent}} \cdot \frac{1}{2\ \text{mL}} \cdot \frac{10^3\ \text{mg}}{1\ \text{g}} = (N_o - N_s) \cdot 440.5$$

ETHYLENE OXIDE by Portable GC

3702

C_2H_4O MW: 44.05 CAS: 75-21-8 RTECS: KX2450000

METHOD: 3702, Issue 2 **EVALUATION: FULL** **Issue 1: 15 August 1987**
Issue 2: 15 August 1994

OSHA : 1 ppm
NIOSH: 0.1 ppm; C 5 ppm/10 min; carcinogen; Group I Pesticide
ACGIH: 1 ppm; suspect carcinogen
1 ppm = 1.801 mg/m^3 @ NTP

PROPERTIES: gas; d (liq.) 0.8694 g/mL @ 20 °C; BP 10.7 °C; MP -111 °C; explosive limits 3 to 100% (v/v) in air

SYNONYMS: 1,2-epoxyethane; oxirane

SAMPLING

SAMPLER: AMBIENT AIR OR BAG SAMPLE

FLOW RATE: ≥ 0.02 L/min; spot samples possible (See Step 1)

SHIPMENT: calibration and carrier gas shipment must comply with hazardous materials shipment regulations

SAMPLE STABILITY: bag samples stable 24 h @ 25 °C [1]

FIELD BLANKS: clean air, either in bag or from non-work area

ACCURACY

RANGE STUDIED: 0.1 to 700 ppm [1]

BIAS: not significant

OVERALL PRECISION ($\hat{S}_{rT}$): <0.09 [1]

ACCURACY: ± 17%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY (PORTABLE), PHOTOIONIZATION DETECTOR

ANALYTE: ethylene oxide

COLUMN: 1.2 m x 3 mm OD PTFE, packed with Carbopak BHT 40/100 mesh

CARRIER GAS: ultrapure air, 15 mL/min

CALIBRATION: bag standards or calibrated gas mixtures

RANGE: 0.001 to 1000 ppm

ESTIMATED LOD: 2.5 pg per injection @ .001 ppm (1-mL injection)

PRECISION ($\hat{S}_r$): <0.07 @ 0.05 to 0.2 ppm [1]

APPLICABILITY: The working range is 0.001 to 1000 ppm in relatively non-complex atmospheres (e.g., sterilization facilities).

INTERFERENCES: Freon 12, carbon dioxide, and alcohols do not interfere. Other compounds with similar retention times under the same chromatographic conditions are potential interferences.

OTHER METHODS: This method complements Method 1614 [2] which utilizes solid sorbent collection, derivatization, and gas chromatographic measurement for ethylene oxide.

REAGENTS:

1. Uncontaminated air for preparation of standards and purging samplers.
2. Carrier gas, air, ultra-pure.*
3. Ethylene oxide (EtO) for standards, pure or in known concentration (i.e., 88/12 Freon 12/EtO mixture).*

   NOTE: Commercial sterilant mixtures are sold on a weight/weight ratio. An 88/12 mixture is 27% EtO by volume.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Portable gas chromatograph (GC) with photoionization detector, column (page 3702-1), and (if appropriate) portable strip chart recorder or integrator, and battery chargers, regulators, and other peripherals necessary for individual instruments.
2. Personal sampling pump, 0.02 to 4 L/min or other rate suitable for filling bag, with flexible connecting tubing.
3. Syringes, gas-tight (0.01 to 1-mL, 1-L), for standards preparation, sample collection and sample injection.
4. Bags, inert plastic (e.g., aluminized polyester or Tedlar), 2- to 20-L, for standard preparation, and (if needed) sample collection.

   NOTE: Care must be taken to assure that the pump, bag, and tubing are all inert and impermeable to the analyte (step 3).

SPECIAL PRECAUTIONS: Ethylene oxide is flammable. Shipment of compressed calibration gas and carrier gas must comply with 49 CFR regulations on shipment of hazardous materials.

SAMPLING:

1. Collect samples by one of the following methods:

   NOTE: Other techniques such as liquid displacement or use of an evacuated vessel may be adaptable to this method but have not been evaluated.

   a. **Draw air directly into a syringe.** Collect syringe samples by first purging a gas-tight syringe several times with clean air to remove any residual ethylene oxide from previous samples, then draw air into the syringe at the time and location of interest.

      NOTE: This technique requires less equipment and also has the advantage of allowing for a grab sample. It is not easily applied to TWA measurements. Since this is a one-time analysis, the concentration of the sample must be estimated in advance so that the proper size sample can be collected. An injection volume as small as ten microliters can be used if the concentration is expected to be several hundred ppm, while a 1.0- mL syringe is more appropriate if a concentration on the order of 0.01 ppm is expected.

   b. **Bag samples for TWA.**

      (1) Evacuate the sampling bag.

      (2) Allow the pump to be purged with sample air before opening the valve on the sampling bag. Collect bag sample by using pump to pull air from a point of interest and push it into the bag.

      NOTE: This technique can obtain a sample in a relatively short time (e.g., at 4 L/min, a few seconds sampling time will yield the fraction of a liter necessary for several replicate analyses). By selecting a low flow rate and larger bag (i.e., 10 mL/min flow and 5 L bag), an 8-h TWA sample can be obtained. A few mL will allow for replicate injections of 1 mL or less, although in practice several hundred mL to 1 L is a more reasonable minimum. The maximum volume is limited only by the size of bags available and space in which to store them.

CALIBRATION AND QUALITY CONTROL:

2. Calibrate the GC daily in the field.
   a. Prepare bag standards by adding a known volume of EtO to a known volume of clean air in a bag. This creates a standard of known concentration in ppm (µL EtO/L air).
      (1) Evacuate a 5- to 10-L bag completely by drawing the air out with a large (1- to 2-L) syringe.
      (2) Draw clean air (or oxygen or nitrogen) from a supply cylinder into the syringe for measured transfer into the bag. Alternately, if a clean air supply is not available, draw room air through charcoal sorbent into the syringe. Repeat until the bag contains 5 L of air.
      (3) Add a known amount of pure EtO or standard EtO mixture to the bag by means of gas-tight syringe.
      Example:
      Using a gas-tight syringe, take 50 µL from a cylinder of pure EtO and inject it into 5 L of air to create a 10 ppm standard. Alternately, 200 µL of a 27% v/v EtO mixture (i.e., 88/12 w/w Freon 12 and EtO) can be added to the 5 L of air to obtain a 10.8 ppm standard.
      (4) Allow the bag to equilibrate, with occasional kneading, for at least 5 min.
   b. Analyze aliquots of various sizes to establish a calibration graph (steps 4 and 5). Analyze three or more replicates at each point.
      NOTE: On a high instrument attenuation (low sensitivity), injections of 0.2, 0.4, 0.6, 0.8 and 1.0 mL might be possible. This would correspond to injections of 2, 4, 6, 8, and 10 nL. On a more sensitive attenuation, injections of 0.02, 0.04, 0.06, 0.08 and 0.10 mL would be typical. Results will vary from instrument to instrument, and from time to time on the same instrument.
   c. Plot nL EtO vs. peak height or area. This plot should be a straight line.
   d. Periodically throughout the day, check calibration by repeating some of these injections of standards. Ideally, each sample would be bracketed, before and after, with injections of standards, although this is seldom practical.
3. Check the sampling equipment to prevent contamination.
   a. Use different syringes for sampling and for standard preparation. Identify each syringe with a unique number.
      NOTE 1:
   So called gas-tight syringes appear to be so only when new. Leakage results in injections less than indicated, resulting in inaccurate reporting of the actual concentration.
      NOTE 2:
   Even after more than a dozen purges, a syringe used to transfer pure ethylene oxide continues to elute small amounts of EtO.
   b. Bag sampling.
      (1) Check inertness and impermeability of the entire sampling train. This is best accomplished in the laboratory before going to the field. Purge a bag of the type to be used for sampling with clean air, and then fill it with clean air and an amount of EtO to create a concentration in the range of interest. Take replicate samples from that bag and inject them into the GC. Measure peak heights. Pump the contents of this bag through a sampling train of the type to be used in the field, into a second bag. Analyze replicate samples from the second bag immediately after transfer and hourly thereafter. If the concentrations found immediately after transfer are equal to those prior to transfer, the sampling train is not altering the EtO concentration (i.e., is inert). If the concentrations do not change over time, the bags are inert.
      (2) Consider any residual analyte which may remain in a bag after use, since practicality dictates reuse of bags. One or two purges with clean air are usually sufficient to remove residual EtO unless the concentration of the previous sample was extremely high. Analyze a sample of "clean" air taken from the bag during its final purge. If no measurable EtO is found, the bag is ready for reuse.
      NOTE: Some bags continue to show traces of analyte, even after several purges, and

these bags should be discarded.

(3) Segregate bags used for sample collection from those used for calibration standards.

MEASUREMENT:

4. Fill a gas-tight syringe, purged several times with sample, from the sample bag. Then empty it to the desired volume, and inject that volume into the chromatograph with a quick firm motion. Record identity of syringe. Use replicate analyses to determine the repeatability of the analysis.

   NOTE 1: If no estimate of concentration is available, use an injection volume of 10 to 25 µL at a high attenuation to reduce the possibility of column and detector overload. Depending on the results of this injection, larger volumes and/or more sensitive attenuations may be selected.

   NOTE 2: The procedure for analysis of samples collected directly in a gas tight syringe is the same as for bag samples, with the obvious elimination of the step where the sample is withdrawn from the bag.

5. Along with the sample identification, record the injection volume (mL), the instrument attenuation, and the resultant peak height or area.

CALCULATIONS:

6. Divide the EtO volume (nL) from the calibration graph, by the injection volume (mL) to calculate sample EtO concentration (ppm):

$$\mathrm{ppm} = \frac{\mu\mathrm{L\ (gas)}}{\mathrm{L\ (gas)}} = \frac{\mathrm{nL\ (gas)}}{\mathrm{mL\ (gas)}}.$$

EVALUATION OF METHOD:

This method was evaluated in the laboratory where the accuracy and precision were determined by a comparison of measured values with accepted concentrations from a dynamic (permeation tube) generation system with confirmation by lab G.C. with flame ionization detector [1]. Field evaluations were conducted in hospital and manufacturing sterilization facilities which used commercial mixtures of EtO with Freon 12 and carbon dioxide, respectively.

REFERENCES:

[1] Burroughs G. E., and Busch, K. A., "Field Validation of Direct-Reading Instrumental Methods for the Sampling and Analysis of Environmental Contaminants," unpublished NIOSH report (1985).

[2] NIOSH Manual of Analytical Methods, 3rd ed., Vol. 1, Method 1614, Department of Health and Human Services (NIOSH) Publ. No. 84-100 (1984).

METHOD WRITTEN BY:

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California Environmental Protection Agency

Air Resources Board

Vapor Recovery Test Procedure

TP-201.3

Determination of 2 Inch WC Static Pressure Performance of Vapor Recovery Systems of Dispensing Facilities

Adopted: April 12, 1996
Amended: March 17, 1999

California Environmental Protection Agency
Air Resources Board
Vapor Recovery Test Procedure

TP-201.3

Determination of 2 Inch WC Static Pressure Performance of Vapor Recovery Systems of Dispensing Facilities

1 APPLICABILITY

Definitions common to all certification and test procedures are in:

D-200 **Definitions for Certification Procedures and Test Procedures for Vapor Recovery Systems**

For the purpose of this procedure, the term "ARB" refers to the State of California Air Resources Board, and the term "ARB Executive Officer" refers to the Executive Officer of the ARB or his or her authorized representative or designate.

1.1 This test procedure is used to quantify the vapor tightness of vapor recovery systems installed at gasoline dispensing facilities (GDF) equipped with pressure/vacuum (P/V) valves, provided that the designed pressure setting of the P/V valves is a minimum of 2.5 inches of water column (inches H_2O).

1.2 Systems equipped with a P/V valve(s) allowed to have a designed cracking pressure less than 2.5 inches H_2O shall be bagged to eliminate any flow contribution through the valve assembly from the test results. The valve/vent pipe connection, however, shall remain unobstructed during this test.

1.3 At facilities not required to be equipped with a P/V valve(s), the vent pipe(s) shall be capped. For those installations, the test may be conducted at the vent pipe(s).

2 PRINCIPLE AND SUMMARY OF TEST PROCEDURE

2.1 The entire vapor recovery system is pressurized with nitrogen to two (2.0) inches H_2O. The system pressure is then allowed to decay and the pressure after five (5) minutes is compared with an allowable value. The minimum allowable five-minute final pressure is based on the system ullage and pressure decay equations. For the purpose of compliance determination, this test shall be conducted after all back-filling, paving, and installation of all Phase I and Phase II components, including P/V valves, has been completed.

2.2 For GDF equipped with a coaxial Phase I system, this test shall be conducted at a Phase II vapor riser. For GDF which utilize a two-point Phase I system, this test may be conducted at either a Phase II riser or a Phase I vapor coupler provided that the criteria set forth in Section 6.7 have been met. If the integrity criteria for two-point systems specified in Section 6.7 are met, it is recommended that this test be conducted at the Phase I vapor coupler.

3 RANGE

3.1 If mechanical pressure gauges are employed, the full-scale range of pressure gauges shall be 0-2.0, 0-1.0, and 0-0.50 inches H_2O column. Maximum incremental graduations of the pressure gauge shall be 0.05 inches H_2O and the minimum accuracy of the gauge shall be three percent of full scale. The minimum diameter of the pressure gauge face shall be 4 inches.

3.2 If an electronic pressure measuring device is used, the full-scale range of the device shall not exceed 0-10 inches H_2O with a minimum accuracy of 0.5 percent of full-scale. A 0-20 inches H_2O device may be used, provided the equivalent accuracy is not less than 0.25 percent of full-scale.

3.3 The minimum total ullage, for each individual tank, shall be 1,000 gallons or 25% of the tank capacity, whichever is less. The maximum total ullage, for all manifolded tanks, shall not exceed 25,000 gallons. These values are exclusive of all vapor piping volumes.

3.4 The minimum and maximum nitrogen feed-rates, into the system, shall be one (1) and five (5) CFM, respectively.

4 INTERFERENCES

4.1 Introduction of nitrogen into the system at flowrates exceeding five (5) CFM may bias the results of the test toward non-compliance. Only gaseous nitrogen shall be used to conduct this test. Air, liquefied nitrogen, helium, or any gas other than nitrogen shall not be used for this test procedure.

4.2 For vacuum-assist Phase II systems which utilize an incinerator, power to the collection unit and the processor shall be turned off during testing.

4.3 For vacuum-assist systems, with positive displacement vacuum pumps, which locate the vacuum producing device in-line between the Phase II vapor riser and the storage tank, the following requirements shall apply:

4.3.1 A valve shall be installed at the vacuum producing device. When closed, this valve shall isolate the vapor passage downstream of the vacuum producing device.

4.3.2 The storage tank side of the vacuum producing device shall be tested in accordance with the procedures outlined in Section 7 of this method. Compliance shall be determined by comparing the final five-minute pressure with the allowable minimum five-minute final pressure from the first column (1-6 affected nozzles) in Table IB or use the corresponding equation in Section 9.2.

4.3.3 The upstream vapor passage (nozzle to vacuum producing device) shall also be tested. Methodology for this test shall be submitted to the California Air Resources Board (CARB) for approval prior to submission of test results or shall be conducted in accordance with the procedures set forth in the applicable CARB Executive Order.

4.4 The results of this static pressure integrity test shall not be used to verify compliance if an Air to Liquid Volumetric Ratio Test (TP-201.5 or equivalent) was conducted within 24 hours prior to this test.

4.5 Thermal Bias for Electronic Manometers

Electronic manometers shall have a warm-up period of at least 15 minutes followed by a five minute drift check. If the drift exceeds 0.01 inches water column, the instrument should not be used.

5 APPARATUS

5.1 Nitrogen

Use commercial grade nitrogen in a high pressure cylinder, equipped with a two-stage pressure regulator and a one psig pressure relief valve.

5.2 Pressure Measuring Device

Use 0-2.0, 0-1.0, and 0-0.50 inches H_2O pressure gauges connected in parallel, a 0-2 inches H_2O manometer, or an electronic pressure measuring device to monitor the pressure decay in the vapor recovery system. The pressure measuring device shall, at a minimum, be readable to the nearest 0.05 inches H_2O.

5.3 "T" Connector Assembly

See Figure 1 for example.

5.4 Vapor Coupler Integrity Assembly

Assemble OPW 633-A, 633-B, and 634-A adapters, or equivalent, as shown in Figure 2. If the test is to be conducted at the storage tank Phase I vapor coupler, this assembly shall be used prior to conducting the static leak test in order to verify the pressure integrity of the vapor poppet. The internal volume of this assembly shall not exceed 0.1 cubic feet.

5.5 Vapor Coupler Test Assembly

Use a compatible OPW 634-B cap, or equivalent, equipped with a center probe to open the poppet, a pressure measuring device to monitor the pressure decay, and a connection for the introduction of nitrogen into the system. See Figure 3 for an example.

5.6 Stopwatch

Use a stopwatch accurate to within 0.2 seconds.

5.7 Flow Meter

Use a Dwyer flowmeter, Model RMC-104, or equivalent, to determine the required pressure setting of the delivery pressure gauge on the nitrogen supply pressure regulator. This pressure shall be set such that the nitrogen flowrate is between 1.0 and 5.0 CFM.

5.8 Combustible Gas Detector

A Bacharach Instrument Company, Model 0023-7356, or equivalent, may be used to verify the pressure integrity of system components during this test.

5.9 Leak Detection Solution

Any liquid solution designed to detect vapor leaks may be used to verify the pressure integrity of system components during this test.

6 PRE-TEST PROCEDURES

6.1 The following safety precautions shall be followed:

6.1.1 Only nitrogen shall be used to pressurize the system.

6.1.2 A one psig relief valve shall be installed to prevent the possible over-pressurizing of the storage tank.

6.1.3 A ground strap should be employed during the introduction of nitrogen into the system.

6.2 Failure to adhere to any or all of the following time and activity restrictions shall invalidate the test results:

6.2.1 There shall be no Phase I bulk product deliveries into or out of the storage tank(s) within the three (3) hours prior to the test or during performance of this test procedure.

6.2.2 There shall be no product dispensing within thirty (30) minutes prior to the test or during performance of this test procedure.

6.2.3 Upon commencement of the thirty minute "no dispensing" portion of this procedure, the headspace pressure in the tank shall be measured. If the pressure exceeds 0.50 inches H_2O, the pressure shall be carefully relieved in accordance with all applicable safety requirements. After the thirty minute "no dispensing" portion of this procedure, and prior to introduction of nitrogen, the headspace pressure shall again be lowered, if necessary, to less than 0.50 inches H_2O.

6.2.4 There shall be no Air to Liquid Volumetric Ratio Test (TP-201.5 or equivalent) conducted within the twenty-four (24) hour period immediately prior to this test.

6.2.5 The test shall be conducted with the station in normal operating mode. This includes all nozzles properly hung up in the dispenser boots and all dispenser cabinet covers in place. The exception to normal operating mode is that dispensing is disallowed as specified.

6.3 Measure the gallons of gasoline present in each underground storage tank and determine the actual capacity of each storage tank from facility records. Calculate the ullage space for each tank by subtracting the gasoline gallonage present from the actual tank capacity. The minimum ullage during the test, for all manifolded tanks, shall be 1,000 gallons or 25 percent of the tank capacity, whichever is less. The total ullage, for all manifolded tanks, shall not exceed 25,000 gallons.

6.4 For two-point Phase I systems, this test shall be conducted with the dust cap removed from both the product and the vapor coupler. This is necessary to determine the vapor tightness of the Phase I vapor poppet. See Section 6.7 if this test is to be conducted at the Phase I vapor coupler.

6.4.1 For coaxial Phase I systems, this test shall be conducted with the dust cap removed from the Phase I coupler. This is necessary to insure the vapor tightness of the Phase I vapor poppet.

6.4.2 Verify that the liquid level in the storage tank is at least four (4) inches above the highest opening at the bottom of the submerged drop tube.

6.5 If the Phase I containment box is equipped with a drain valve, this test shall be conducted with the drain valve installed and the manhole cover removed. If the drain valve is cover-actuated, the test shall be done once with the cover removed and repeated with the cover installed.

6.6 If the test is to conducted at a Phase II vapor riser, disconnect the dispenser end of one vapor recovery hose and install the "T" connector assembly (see Figure 1). Connect the nitrogen gas supply (do not use air) and the pressure measuring device to the "T" connector.

6.6.1 For those Phase II vapor systems utilizing a dispenser mounted remote vapor check valve, the "T" connector assembly shall be installed on the vapor riser side of the check valve.

6.7 If this test is to be conducted at the Phase I vapor coupler on a two-point Phase I system, the procedures set forth in subsections 6.7.1 and 6.7.2 shall be successfully completed prior to testing. The static pressure integrity test shall not be conducted at the Phase I coupler at facilities equipped with coaxial Phase I systems.

6.7.1 Connect the Vapor Coupler Integrity Assembly to the Phase I vapor coupler. Connect the Vapor Coupler Test Assembly. Connect the nitrogen supply to the assembly and carefully pressurize the internal volume of the assembly to two (2.0) inches H_2O. Start the stopwatch. Record the final pressure after one minute.

6.7.2 If the pressure after one minute is less than 0.25 inches H_2O, the leak rate through the Phase I vapor poppet precludes conducting the static leak test at this location. If the pressure after one minute is greater than or equal to 0.25 inches H_2O, the static leak test may be conducted at this location. This criteria assures a maximum leak rate through the Phase I vapor poppet of less than 0.0004 cubic feet per minute.

6.7.3 Disconnect the Vapor Coupler Integrity Assembly to the Phase I vapor coupler. If the requirements of subsection 6.7.2 were met, connect the Vapor Coupler Test Assembly to the Phase I vapor coupler.

6.7.4 Product may be poured onto the Phase I vapor coupler to check for leaks. This diagnostic procedure shall not be substituted for the procedures set forth in subsections 6.7.1 and 6.7.2.

6.8 All pressure measuring device(s) shall be bench calibrated using either a reference gauge or incline manometer. Calibration shall be performed at 20, 50, and 80 percent of full scale. Accuracy shall be within two percent at each of these calibration points. Calibrations shall be conducted on a frequency not to exceed 90 days.

6.9 Use the flowmeter to determine the nitrogen regulator delivery pressures which correspond to nitrogen flowrates of 1.0 and 5.0 CFM. These pressures define the allowable range of delivery pressures acceptable for this test procedure. Also record the regulator delivery pressure setting, and the corresponding nitrogen flowrate that will be used during the test. As an alternative, the flowmeter may be connected, in-line between the nitrogen supply regulator and Vapor Coupler Test Assembly, during the test.

6.10 Use Equation 9.3 to calculate the approximate time required to pressurize the system ullage to the initial starting pressure of two (2.0) inches H_2O. This will allow the tester to minimize the quantity of nitrogen introduced into those systems which cannot comply with the static leak standards.

6.11 Attach the Vapor Coupler Test assembly to the Phase I poppet or the "T" connector assembly to the Phase II vapor riser. Read the initial pressure of the storage tank and underground piping. If the initial pressure is greater than 0.5 inches H_2O, carefully bleed off the pressure, in accordance with all applicable safety procedures, in the storage tank and underground piping to less than 0.5 inches H_2O column.

6.12 Any electronic manometers shall be subject to warm-up and drift check before use; see Section 4.5.

7 TESTING

7.1 Open the nitrogen gas supply valve and set the regulator delivery pressure within the allowable range determined in Section 6.9, and start the stopwatch. Pressurize the vapor system (or subsystem for individual vapor return line systems) to **at least 2.2 inches H_2O** initial pressure. It is critical to maintain the nitrogen flow until the pressure stabilizes, indicating temperature and vapor pressure stabilization in the tanks. Check the test equipment using leak detecting solution or a combustible gas detector to verify that all test equipment is leak tight. Note: if a combustible gas detector is used to search for leaks, components which were certified with an allowable leak rate, such as 0.38 CFH at a pressure of two (2) inches, cannot be determined to be faulty solely on the basis of the concentration registered on the instrument.

7.1.1 If the time required to achieve the initial pressure of two (2.0) inches H_2O exceeds twice the time derived from Equation 9.3, stop the test and use liquid leak detector, or a combustible gas detector, to find leak(s) in the system. Failure to achieve the initial starting pressure within twice the time derived from Equation 9.3 demonstrates the inability of the system to meet the performance criteria. Repair or replace the faulty component(s) and restart the test pursuant to Section 7.1.

7.2 Close and disconnect the nitrogen supply. Start the stopwatch when the pressure has decreased to the initial starting pressure of two (2.0) inches H_2O.

7.3 At one-minute intervals during the test, record the system pressure. After five minutes, record the final system pressure. See the applicable of Tables 1A (or Equation 9.1) or 1B (or equation 9.2) to determine the acceptability of the final system static pressure results. For intermediate values of ullage in Tables 1A and 1B, linear interpolation may be employed.

7.4 If the system failed to meet the criteria set forth in Table 1A or 1B (or the appropriate equation in Section 9), repressurize the system and check all accessible vapor connections using leak detector solution or a combustible gas detector. If vapor leaks in the system are encountered, repair or replace the defective component and repeat the test. Potential sources of leaks include nozzle check valves, nozzle vapor paths, pressure/vacuum relief valves, containment box drain valve assemblies, and plumbing connections at the risers.

7.4.1 If the facility fails to comply with the static leak test standards and the two point Phase I system utilizes overfill prevention devices in the drop tubes which were installed before July 1, 1993, and which are unable to pass the test with the dust caps removed from the product and vapor couplers (see Sec. 6.4), the test may be conducted with the caps on the couplers, as an exception.

This exception is not intended to allow bleed holes in drop tubes.

This exception expires on January 1, 2002, after which date all testing shall be conducted with the fill and vapor caps removed from two point systems. Under no circumstances may the test be conducted with the caps on coaxial Phase I couplers.

7.5 After the remaining system pressure has been relieved, remove the "T" connector assembly and reconnect the vapor recovery hose, if applicable.

7.6 If the vapor recovery system utilizes individual vapor return lines, repeat the leak test for each gasoline grade. Avoid leaving any vapor return line open longer than is necessary to install or remove the "T" connector assembly.

7.7 If the applicable CARB Executive Order requires the test to be conducted with and without the containment box cover in place, repeat the test with the cover in place. In these cases clearly specify, on Form 1, which results represent the pressure integrity with and without the cover in place.

8 POST-TEST PROCEDURES

8.1 Use the applicable of Table 1A or 1B, or the applicable of Equations 9.1 or 9.2, to determine the compliance status of the facility by comparing the final five-minute pressure with the minimum allowable final pressure.

8.1.1 For balance Phase II systems use Table 1A or the applicable of Equation 9.1 to determine compliance.

8.1.2 For vacuum-assist Phase II systems use Table 1B or the applicable of Equation 9.2 to determine compliance.

9 CALCULATIONS

9.1 For Phase II Balance Systems, the minimum allowable five-minute final pressure, with an initial pressure of two (2.0) inches H_2O, shall be calculated as follows:

[Equation 9-1]

$$P_f = 2e^{\left(\frac{-760.490}{V}\right)} \quad \text{if } N = 1-6$$

$$P_f = 2e^{\left(\frac{-792.196}{V}\right)} \quad \text{if } N = 7-12$$

$$P_f = 2e^{\left(\frac{-824.023}{V}\right)} \quad \text{if } N = 13-18$$

$$P_f = 2e^{\left(\frac{-855.974}{V}\right)} \quad \text{if } N = 19-24$$

$$P_f = 2e^{\left(\frac{-888.047}{V}\right)} \quad \text{if } N > 24$$

where:

N = The number of affected nozzles. For manifolded systems, N equals the total number of nozzles. For dedicated plumbing configurations, N equals the number of nozzles serviced by the tank being tested.

P_f = The minimum allowable five-minute pressure, inches H_2O

V = The total ullage affected by the test, gallons

e = A dimensionless constant approximately equal to 2.718

2 = The initial starting pressure, inches H_2O

9.2 For Phase II Vacuum Assist Systems, the minimum allowable five-minute final pressure, with an initial pressure of two (2.0) inches H_2O, shall be calculated as follows:

$$P_f = 2e^{\left(\frac{-500.887}{V}\right)} \quad \text{if } N = 1-6 \qquad \text{[Equation 9-2]}$$

$$P_f = 2e^{\left(\frac{-531.614}{V}\right)} \quad \text{if } N = 7-12$$

$$P_f = 2e^{\left(\frac{-562.455}{V}\right)} \quad \text{if } N = 13-18$$

$$P_f = 2e^{\left(\frac{-593.412}{V}\right)} \quad \text{if } N = 19-24$$

$$P_f = 2e^{\left(\frac{-624.483}{V}\right)} \quad \text{if } N > 24$$

where:

N = The number of affected nozzles. For manifolded systems, N equals the number of nozzles. For dedicated plumbing configurations, N equals the number of nozzles serviced by the tank being tested.

P_f = The minimum allowable five-minute final pressure, inches H_2O

V = The total ullage affected by the test, gallons

e = A dimensionless constant approximately equal to 2.718

2 = The initial starting pressure, inches H_2O

9.3 The minimum time required to pressurize the system ullage from zero (0) to two (2.0) inches H_2O gauge pressure shall be calculated as follows:

$$t_2 = \frac{V}{[1980]\,F} \qquad \text{[Equation 9-3]}$$

where:

t_2 = The minimum time to pressurize the ullage to two inches H_2O, minutes

V = The total ullage affected by the test, gallons

F = The nitrogen flowrate into the system, CFM

1980 = The conversion factor for pressure and gallons

9.4 If the policy of the local District requires an allowable tolerance for testing error, the minimum allowable five-minute final pressure, including testing error, shall be calculated as follows:

$$P_{f-E} = 2 - \left[1+\left(\frac{E}{100}\right)\right](408.9-(P_f+406.9)) \qquad \text{[Equation 9-4]}$$

where:

P_{f-E} = The minimum allowable five-minute final pressure including allowable testing error, inches H_2O

E = The allowable testing error, percent

P_f = The minimum allowable five-minute final pressure calculated in Equations 9-1 or 9-2, inches H_2O

2 = The initial starting pressure, inches H_2O

408.9 = Atmospheric pressure plus the initial starting pressure, inches H_2O

406.9 = Atmospheric pressure, inches H_2O

10 REPORTING

10.1 The calculated ullage and system pressures for each five-minute vapor recovery system test shall be reported as shown in Form 1. Be sure to include the Phase I system type (two-point or coaxial), the Phase II system type, whether the system is manifolded, and the one-minute pressures during the test.

TABLE 1A

PHASE II BALANCE SYSTEMS

PRESSURE DECAY CRITERIA

INITIAL PRESSURE OF 2 INCHES WATER COLUMN (WC)

MINIMUM PRESSURE AFTER 5 MINUTES, INCHES WC

	NUMBER OF AFFECTED NOZZLES				
ULLAGE, GALLONS	**01-06**	**07-12**	**13-18**	**19-24**	**>24**
500	0.44	0.41	0.38	0.36	0.34
550	0.50	0.47	0.45	0.42	0.40
600	0.56	0.53	0.51	0.48	0.46
650	0.62	0.59	0.56	0.54	0.51
700	0.67	0.64	0.62	0.59	0.56
750	0.73	0.70	0.67	0.64	0.61
800	0.77	0.74	0.71	0.69	0.66
850	0.82	0.79	0.76	0.73	0.70
900	0.86	0.83	0.80	0.77	0.75
950	0.90	0.87	0.84	0.81	0.79
1,000	0.93	0.91	0.88	0.85	0.82
1,200	1.06	1.03	1.01	0.98	0.95
1,400	1.16	1.14	1.11	1.09	1.06
1,600	1.24	1.22	1.19	1.17	1.15
1,800	1.31	1.29	1.27	1.24	1.22
2,000	1.37	1.35	1.32	1.30	1.28
2,200	1.42	1.40	1.38	1.36	1.34
2,400	1.46	1.44	1.42	1.40	1.38
2,600	1.49	1.47	1.46	1.44	1.42
2,800	1.52	1.51	1.49	1.47	1.46
3,000	1.55	1.54	1.52	1.50	1.49
3,500	1.61	1.59	1.58	1.57	1.55
4,000	1.65	1.64	1.63	1.61	1.60
4,500	1.69	1.68	1.67	1.65	1.64
5,000	1.72	1.71	1.70	1.69	1.67
6,000	1.76	1.75	1.74	1.73	1.72
7,000	1.79	1.79	1.78	1.77	1.76
8,000	1.82	1.81	1.80	1.80	1.79
9,000	1.84	1.83	1.83	1.82	1.81
10,000	1.85	1.85	1.84	1.84	1.83
15,000	1.90	1.90	1.89	1.89	1.89
20,000	1.93	1.91	1.92	1.92	1.91
25,000	1.94	1.94	1.94	1.93	1.93

Note: For manifolded Phase II Balance Systems, the "**Number of Affected Nozzles**" shall be the total of all gasoline nozzles. For dedicated return configurations, the "**Number of Affected Nozzles**" shall be the total of those nozzles served by the tank being tested.

TABLE 1B

PHASE II ASSIST SYSTEMS

PRESSURE DECAY CRITERIA

INITIAL PRESSURE OF 2 INCHES WATER COLUMN (WC)

MINIMUM PRESSURE AFTER 5 MINUTES, INCHES WC

ULLAGE, GALLONS	NUMBER OF AFFECTED NOZZLES				
	01-06	07-12	13-18	19-24	>24
500	0.73	0.69	0.65	0.61	0.57
550	0.80	0.76	0.72	0.68	0.64
600	0.87	0.82	0.78	0.74	0.71
650	0.93	0.88	0.84	0.80	0.77
700	0.98	0.94	0.90	0.86	0.82
750	1.03	0.98	0.94	0.91	0.87
800	1.07	1.03	0.99	0.95	0.92
850	1.11	1.07	1.03	1.00	0.96
900	1.15	1.11	1.07	1.03	1.00
950	1.18	1.14	1.11	1.07	1.04
1,000	1.21	1.18	1.14	1.10	1.07
1,200	1.32	1.28	1.25	1.22	1.19
1,400	1.40	1.37	1.34	1.31	1.28
1,600	1.46	1.43	1.41	1.38	1.35
1,800	1.51	1.49	1.46	1.44	1.41
2,000	1.56	1.53	1.51	1.49	1.46
2,200	1.59	1.57	1.55	1.53	1.51
2,400	1.62	1.60	1.58	1.56	1.54
2,600	1.65	1.63	1.61	1.59	1.57
2,800	1.67	1.65	1.64	1.62	1.60
3,000	1.69	1.68	1.66	1.64	1.62
3,500	1.73	1.72	1.70	1.69	1.67
4,000	1.76	1.75	1.74	1.72	1.71
4,500	1.79	1.78	1.77	1.75	1.74
5,000	1.81	1.80	1.79	1.78	1.77
6,000	1.84	1.83	1.82	1.81	1.80
7,000	1.86	1.85	1.85	1.84	1.83
8,000	1.88	1.87	1.86	1.86	1.85
9,000	1.89	1.89	1.88	1.87	1.87
10,000	1.90	1.90	1.89	1.88	1.88
15,000	1.93	1.93	1.93	1.92	1.92
20,000	1.95	1.95	1.94	1.94	1.94
25,000	1.96	1.96	1.96	1.95	1.95

Note: For manifolded Phase II Assist Systems, the "**Number of Affected Nozzles**" shall be the total of all gasoline nozzles. For dedicated return configurations, the "**Number of Affected Nozzles**" shall be t he total of those nozzles served by the tank being tested.

California Environmental Protection Agency

Air Resources Board

Vapor Recovery Test Procedures

TP - 201.4

DETERMINATION OF DYNAMIC PRESSURE PERFORMANCE OF VAPOR RECOVERY SYSTEMS OF DISPENSING FACILITIES

Adopted: April 12, 1996
Amended: April 28, 2000

California Environmental Protection Agency
Air Resources Board

Vapor Recovery Test Procedure

TP-201.4

Determination of Dynamic Pressure Performance of Vapor Recovery Systems of Dispensing Facilities

1 APPLICABILITY

Definitions common to all certification and test procedures are in:

D-200 Definitions for Certification Procedures and Test Procedures for Vapor Recovery Systems

For the purpose of this procedure, the term "ARB" refers to the State of California Air Resources Board, and the term "ARB Executive Officer" refers to the Executive Officer of the ARB or his or her authorized representative or designate.

This test procedure can be used to quantify the dynamic pressure (back-pressure) in the vapor path leading from the dispensing nozzle to the storage tank. The dynamic pressure associated with vehicle fueling is determined by various alternative procedures, one of which is applied as appropriate for the operational characteristics of the subject vapor recovery system.

This test procedure is used to determine the pressure performance standard of a vapor recovery system during the certification process and subsequently to determine compliance with that performance standard for any installations of such a system.

This test procedure is applicable only to balance type vapor recovery systems and is explicitly not applicable to vapor assist type systems.

2 PRINCIPLE AND SUMMARY OF TEST PROCEDURE

The principle of this test procedure is to determine the dynamic pressure of a vapor recovery system at known dispensing flow rates. Some alternative procedures are provided and one procedure shall be chosen for application appropriate to the operational characteristics of the subject vapor recovery system. A novel test procedure may be developed and used which incorporates some aspects of the procedures provided.

3 BIASES AND INTERFERENCES

3.1 Any leaks in the nozzle vapor path, vapor hose, or underground vapor return piping will result in erroneously low dynamic back pressure measurements.

3.2 The same procedure must be used to determine a dynamic pressure performance standard and determine compliance with that standard.

4 SENSITIVITY, RANGE, AND PRECISION

4.1 Sensitivity

Sensitivity of measurements of pressure and volumetric flow rate is approximately equal to the graduation interval specified for each instrument in Section 5.

4.2 Range

The range of practical measurements of pressure and volumetric flow rate consistent with this test procedure is limited by the instrument range specified for each instrument in Section 5.

4.3 Precision

Non-compliance with an applicable pressure limit shall be determined only when the measured pressure exceeds the applicable limit by more than 5% of the limit value or 0.02 inches of H_2O, whichever is greater.

5 EQUIPMENT

5.1 Nitrogen Pressure Drop Test Unit

The unit shall consist of a suitable frame or cabinet to which the pressure measurement device, the rotameter, and the fill pipe adaptor are rigidly attached and shall be equipped with suitable leveling bubble(s) and leveling screws or other provisions for leveling the pressure measurement device and the rotameter while in use. The fill pipe shall be mounted so that nozzles will hang in the normal semi-horizontal position when inserted, and gauges shall be mounted at a height suitable for proper observation. See Figure 1.

Use a fill pipe known to be compatible with all vapor recovery nozzles and equipped with a pressure tap and a separate feeder line consisting of 1/4" or larger copper or stainless steel tubing (or teflon tubing of similar diameter and wall thickness) not longer than 18" delivering nitrogen from the rotameter.

Use a high pressure nitrogen cylinder capable of maintaining a pressure of 2000 psig and equipped with a compatible two-stage pressure regulator. Use commercial grade nitrogen.

5.1.1 A fillpipe without a leaded gasoline restrictor plate, or from which the leaded gasoline restrictor plate has been removed, shall be used to avoid the pressure drop which would otherwise be caused by the restrictor plate. The pressure tap shall be located near the end of the fillpipe to which the nozzle connects, and the nitrogen feeder line shall be well separated from the pressure tap to ensure that nitrogen impinging on the pressure tap does not cause a significant pressure reading.

5.2 Rotameter(s)

Use a calibrated rotameter having a range of 10-100 SCFH Air and a graduation interval no greater than 2 SCFH Air, equipped with a flow control valve. A rotameter designed for measurement of air flow rates, or calibrated against such a rotameter, shall be used and no correction for gas density shall be applied to readings when measuring nitrogen flow rates.

5.3 Pressure gauge(s)

Use a pressure measuring device (either a transducer with electronic readout, an inclined manometer, or a Magnahelic gauge with a range of 0 to 1.00 inches of H_2O and a graduation interval no greater than 0.02 inches of H_2O. Additional gauges with a lesser range may be used for low-range measurements if desired.

The low pressure vent of the pressure measuring device shall be effectively shielded from the wind.

5.4 Hand Pump

Use a gasoline compatible hand pump to drain condensate pots.

6 CALIBRATION PROCEDURE

6.1 Rotameters

Rotameters' calibration shall be checked annually at 20%, 40%, 60%, 80% and 100% of full scale against a dry gas meter, passing air (not nitrogen) through a toggle valve, thence through the rotameter at a constant rate, and thence through the dry gas meter for a measured time interval of at least one minute. If volume measured by the dry gas meter divided by the measured time interval (converted to hours) does not agree with the rotameter's indicated flow rate within 3% of that indicated flow rate or 2 CFH (whichever is greater) at each flow rate, the rotameter shall be replaced or repaired.

6.2 Pressure Measurement Devices

Pressure measurement devices' calibration shall be checked annually at 20%, 40%, 60%, 80% and 100% of full scale against an inclined manometer. If pressure measured by the inclined manometer does not agree with the pressure measurement device's indicated pressure within 3% of that indicated pressure or 0.02 inches H_2O (whichever is greater) at each pressure level, the pressure measurement device shall be replaced or repaired.

6.3 Assembled Nitrogen Pressure Drop Test Unit

Before first use of the nitrogen pressure drop test unit, verify that no significant pressure is indicated when a dismounted nozzle spout, or a nozzle with no boot, is inserted as in normal use and 100 CFH of nitrogen is passed through the apparatus. Passage of nitrogen through passages of the spout or nozzle shall be prevented during this test. Pressure indicated at 100 CFH flow shall be less than 0.02 inches of water.

7 PRE-TEST PROTOCOL

7.1 Test, Challenge, and Failure Modes for Certification Testing

The specification of test, challenge, and failure modes such as the number of liquid transfer episodes, volume and volumetric rate of liquid transfer, storage tank volumes, etc. shall be done according to the principles of CP-201 § 5 for the testing and evaluation of vapor recovery equipment. The facility and system shall be prepared to operate according to any specified test, challenge, and failure modes.

7.2 System and Facility Preparation

System equipment and components shall be completely operational and, at newly constructed facilities, any storage tanks involved in the test shall have been initially filled for the first time to the appropriate volume a minimum of 24 hours prior to the scheduled test.

7.3 Check Facility Operating Mode

7.3.1 (1) If performing a test during the certification process, examine the subject facility to determine the most appropriate application of the alternative test procedures provided, giving preference to Procedure 1 except where it's use is demonstrated to be impractical. If none of these are appropriate, document those features necessary for incorporation into a novel test procedure. If reasonable and practical, make field revisions to the most appropriate procedure and proceed. Otherwise report the need for novel test procedure development.

(2) If performing a test to determine the compliance status of a subject facility, use the test procedure which was specified during the certification process.

7.3.2 For those Phase II systems which do not utilize a remote vapor check valve, use apparatus as shown in Figure 1 unless otherwise required by an ARB Executive Order applicable to the particular type of vapor recovery system. If the vapor recovery system is equipped with a device acting to reduce internal system pressure to a level below atmospheric pressure, the vacuum producing device shall be turned off during this test.

NOTE: The vapor check valve, which acts to block the vapor passage when the nozzle is not in use, is commonly located in the nozzle and actuated by compressing the bellows, but in some rare instances may be located "remotely" in or near the dispenser.

7.3.3 Disconnect the vapor return riser for all dispensers to be tested. Pour two to five gallons of gasoline into each vapor return riser. Allow fifteen (15) minutes for liquid in the vapor return piping to drain, then reconnect the vapor return risers.

7.3.3.1 If all dispensers to be tested have previously passed this test and no changes have been made to underground piping, addition of 2 gallons of gasoline to each vapor return riser may be omitted unless required by the regulatory authority having jurisdiction.

NOTE: The intention of adding liquid gasoline to the vapor return risers is to verify proper drainage of underground piping and ensure that newly constructed or modified stations which may not have had time for condensate to accumulate in any low spots in underground piping are appropriately tested.

7.3.4 Completely drain all gasoline from the spout and bellows.

7.3.5 For those vapor piping configurations which utilize a condensate pot, drain the pot prior to testing.

7.3.6 All Phase I vapor poppets shall be propped open in such a manner that the valve is not damaged.

7.4 Check Equipment and Supplies

The test equipment must be leak-checked each day prior to use.

For the nitrogen pressure drop test unit, plug the nozzle end of the auto fill pipe with a suitable gas cap or other device and disconnect nitrogen supply line at the nitrogen cylinder. Open any toggle valves isolating the rotameter and pressure measuring device(s). With a hand pump or by blowing into the nitrogen supply line, introduce air until a pressure of approximately 1 inch of H_2O is indicated. Close the rotameter valve and observe any progressive loss of pressure. A pressure decay of up to 0.10 inches H_2O, in one minute is considered acceptable.

8 TEST PROCEDURE

Each test procedure is based on direct measurements only; no sampling, recovery, or analysis is involved.

8.1 Procedure 1 - Nitrogen Pressure Test

(Systems *without* a Remote Vapor Check Valve)

Phase II systems which do not utilize a remote vapor check valve may be tested using the following procedure.

8.1.1 Perform an initial visual examination for vapor leaks at the nozzle and hose of the Phase II system to be tested.

NOTE: If obvious vapor leaks are present, report them and do not proceed further. This test assumes the vapor passages, including the bellows and hose, are intact.

Drain all gasoline from the spout, bellows and hose, compressing the bellows and extending the hose to ensure proper drainage.

Insert the nozzle in the fillpipe of the test apparatus, ensuring that a tight seal at the fillpipe/nozzle interface is achieved.

Open the nitrogen supply, set the delivery pressure to 10 psig, and use the rotameter control valve to adjust the flow rate to 20 CFH.

8.1.2 Observe the pressure measurement device. A pulsating pressure, if observed, indicates nitrogen passing through a liquid obstruction in the vapor return system. If this occurs, verify that liquid from recent "topping off" is not present in the hose as follows: close the rotameter control valve, disengage the nozzle and redrain the nozzle and hose assembly. Re-engage the nozzle, open the rotameter control valve and repeat the test. Record the measured pressure, or the midpoint of the range of measured pressures if pulsation continues.

NOTE: All mechanical gauges including rotameters, Magnahelic gauges, and inclined manometers must be read with the eye on a line normal to the scale face where the indicator rests and never from an oblique angle!

8.1.3 Increase the nitrogen flow rate in steps and measure the pressure drop for nitrogen flowrates of 40, 60 and 80, and 100 CFH. In certification testing or when a dispenser nozzle does not comply with an applicable dynamic back pressure limit, repeat testing (at the entire sequence of nitrogen flowrates) until dynamic pressure has been measured at each flowrate three times.

8.1.4 Close and replace the dust cover on the Phase I poppet after all dispenser nozzles have been tested.

8.1.5 Record data as instructed in the section, "RECORDING DATA".

8.2 Procedure 2 - Torus Pressure Test

For some systems, the dynamic pressure can be measured directly during dispensing into vehicles using apparatus assembled according to the design in Figure 2; the range on the pressure gauge is for example only.

Warning: This procedure shall only be used as a screening procedure for the other procedures provided. If this is the only procedure with which a system is compatible, then such system shall be considered to be incompatible with the application of TP-201.4 unless an alternative procedure is developed per § 13.

8.2.1 Measure the dispensing rate and dynamic pressure for any fueling episode during which four or more gallons is dispensed.

8.2.2 Collect data at high, mid-range, and low dispensing rates for five dispensing episodes at each rate.

8.2.3 Record the actual dispensing rate and dynamic pressure for each dispensing episode.

8.3 Procedure 3 - Fixed Volume Pressure Test

IMPORTANT: Use this procedure for compliance determinations only if specified by Executive Order applicable to the specific type of vapor recovery system being tested.

For some systems, the dynamic pressure can be measured directly during dispensing into a surrogate for a vehicle tank using apparatus assembled according to the design in Figure 3; the range on the pressure gauge is for example only.

8.3.1 Measure the dispensing rate (using a stopwatch and the dispenser's metered gallonage) and dynamic pressure for any fueling episode during which half of the fixed volume is dispensed.

8.3.2 Collect data at high, mid-range, and low dispensing rates for five dispensing episodes at each rate. Set constant dispensing rates using the nozzle's hold-open clip or a wooden wedge.

8.3.3 Record the dispensing time, gallons dispensed, calculated dispensing rate and dynamic pressure for each dispensing episode.

9 QUALITY ASSURANCE / QUALITY CONTROL (QA/QC)

This section is reserved for future specification.

10 RECORDING DATA

Figure 4, for example, is the field data sheet for the procedures provided.

Data sheets for other procedures shall be composed in a similar manner, based on field operating parameters.

The following information shall be recorded on the field data sheet:

Facility Identification and Address
Pump Number and Product Grade

Nozzle Make and Model
Nitrogen Flowrate, CFH
Dynamic Back Pressure, inches H_2O

11 CALCULATING RESULTS

Calculate the average dynamic pressure for each dispensing rate tested at each nozzle.

12 REPORTING RESULTS

In compliance testing, the maximum allowable dynamic back pressure for individual dispenser nozzles, with the dry breaks open, is as specified in the CARB Executive Order applicable to the specific vapor recovery system or in any applicable regulation. In certification testing, appropriate allowances for performance variations between individual dispenser nozzles and associated system components shall be made in establishing dynamic pressure limits. Dynamic pressure limits shall be applicable to individual dispenser nozzles.

12.1 Procedure 1

The dynamic pressure performance of each dispenser nozzle shall be reported as the average dynamic pressure at each flow rate.

The dynamic pressure performance measured during certification shall be used as a basis for the performance standard for any installation of the subject vapor recovery system tested. The dynamic back pressure limits specified at each flow rate shall be indicative of the upper limit of the normal range of dynamic back pressures for individual dispensing nozzles at the facility during certification.

12.2 Procedure 3

The dynamic pressure performance shall be reported as the average dynamic pressure at each flow rate.

The dynamic pressure performance measured during certification shall be used as a basis for the performance standard for any installation of the subject vapor recovery system tested. The dynamic back pressure limits specified at each flow rate shall be indicative of the upper limit of the normal range of dynamic back pressures for individual dispensing nozzles at the facility during certification.

13 ALTERNATIVE TEST PROCEDURES

Test procedures, other than specified above, shall only be used if prior written approval is obtained from the ARB Executive Officer. In order to secure the ARB Executive Officer's approval of an alternative test procedure, the applicant is responsible for demonstrating to the ARB Executive Officer's satisfaction that the alternative test procedure is equivalent to this test procedure.

(1) Such approval shall be granted on a case-by-case basis only. Because of the evolving nature of technology and procedures for vapor recovery systems, such approval shall not be granted in subsequent cases without a new request for approval and a new demonstration of equivalency.

(2) Documentation of any such approvals, demonstrations, and approvals shall be maintained in the ARB Executive Officer's files and shall be made available upon request.

14 REFERENCES

This section is reserved for future specification.

15 EXAMPLE FIGURES AND FORMS

Each figure or form provides an illustration of an implementation which conforms to the requirements of this test procedure; other implementations which so conform are acceptable, too. Any specifications or dimensions provided in the figures or forms are for example only, unless such specifications or dimensions are provided as requirements in the text of this or some other required test procedure.

Figure 1
Typical Apparatus for Procedure 1

Figure 2
Torus Pressure Test Assembly

Figure 3
Fixed-Volume Pressure Test Assembly

Figure 4
Field Data Form

Figure 1 - Typical Apparatus for Procedure 1

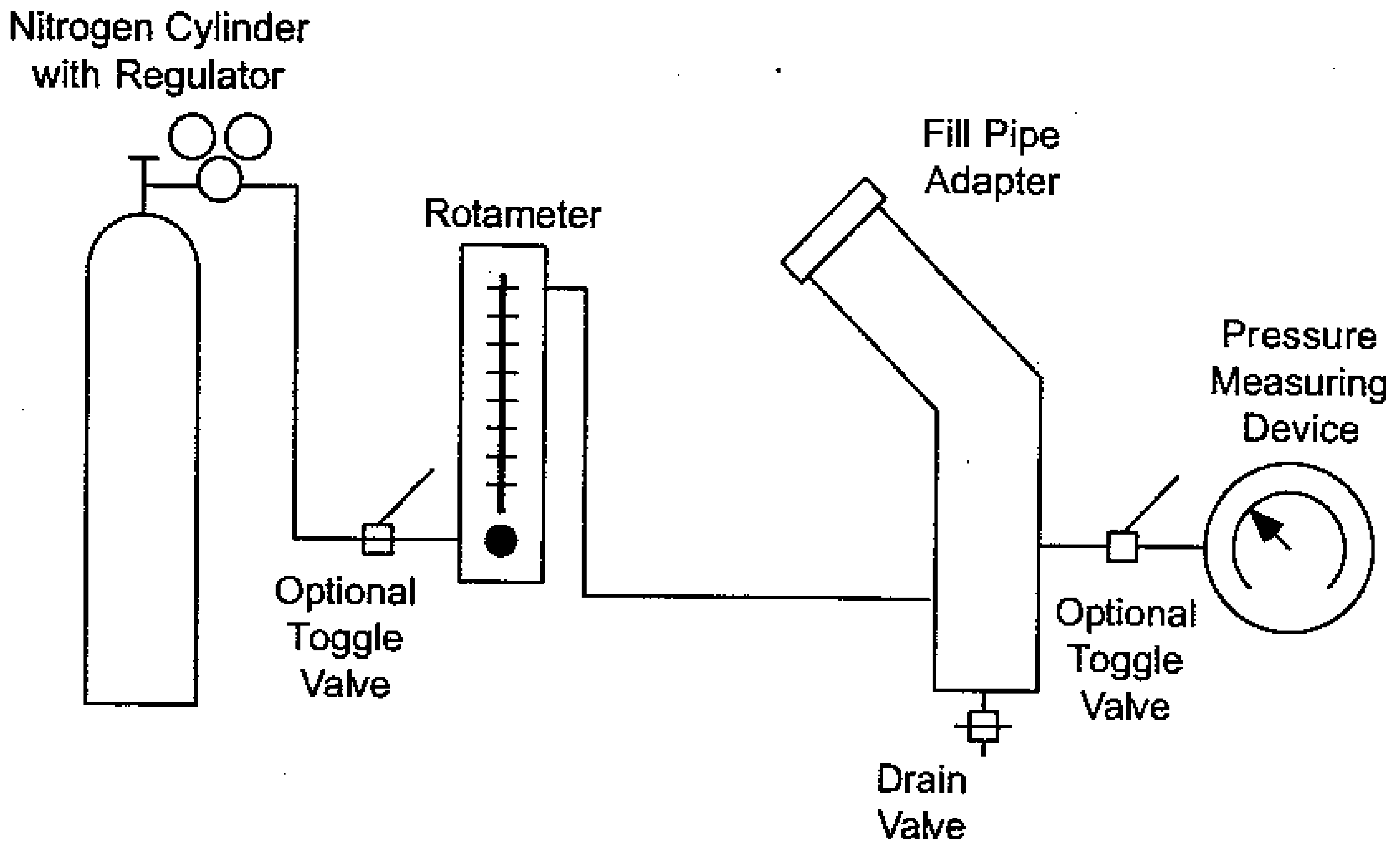
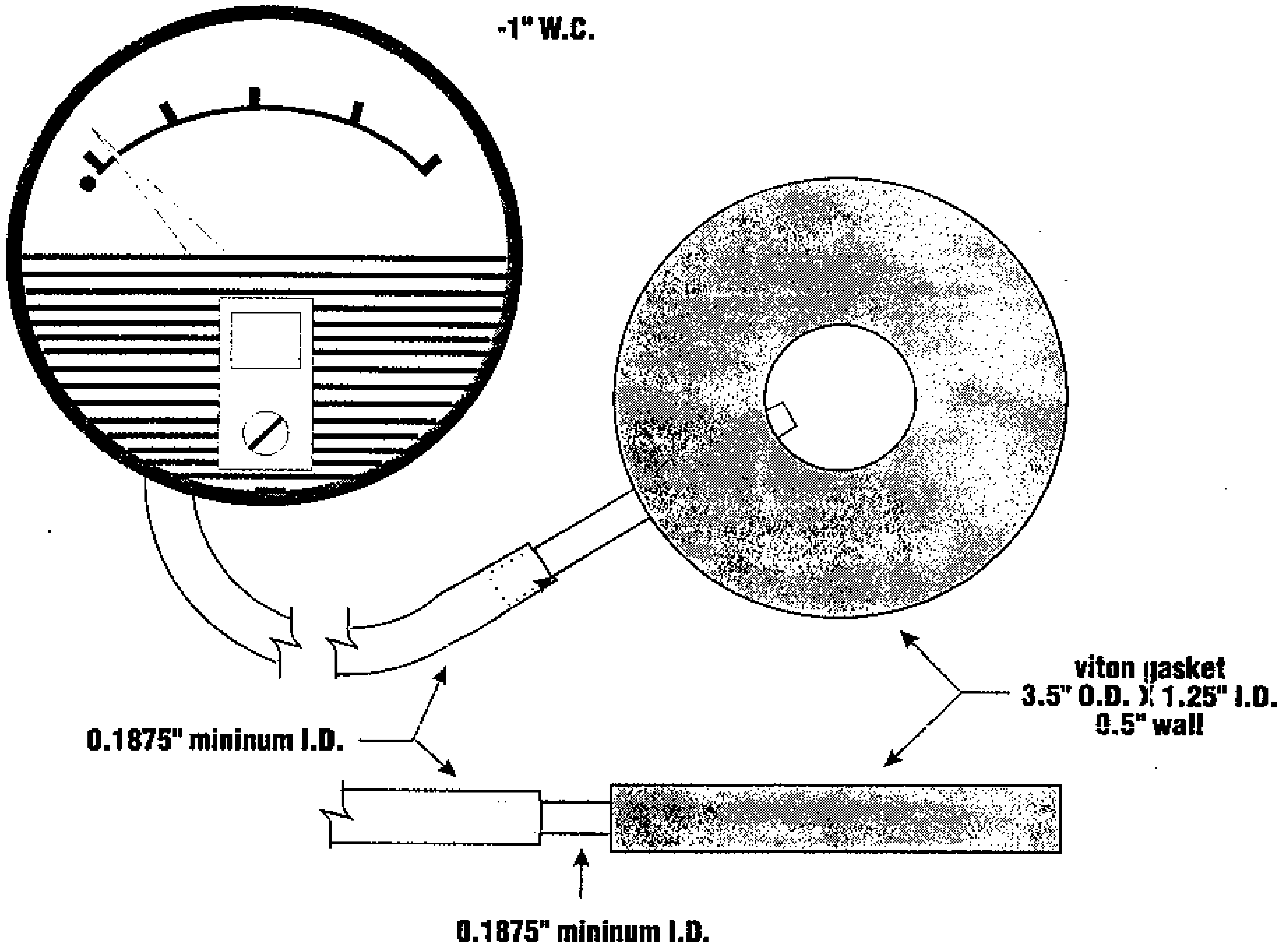


FIGURE 2

Torus Pressure Test Assembly



TP 201.4 F.2/ D. CORDOVA '95

Figure 3 – Fixed Volume Pressure Test Assembly

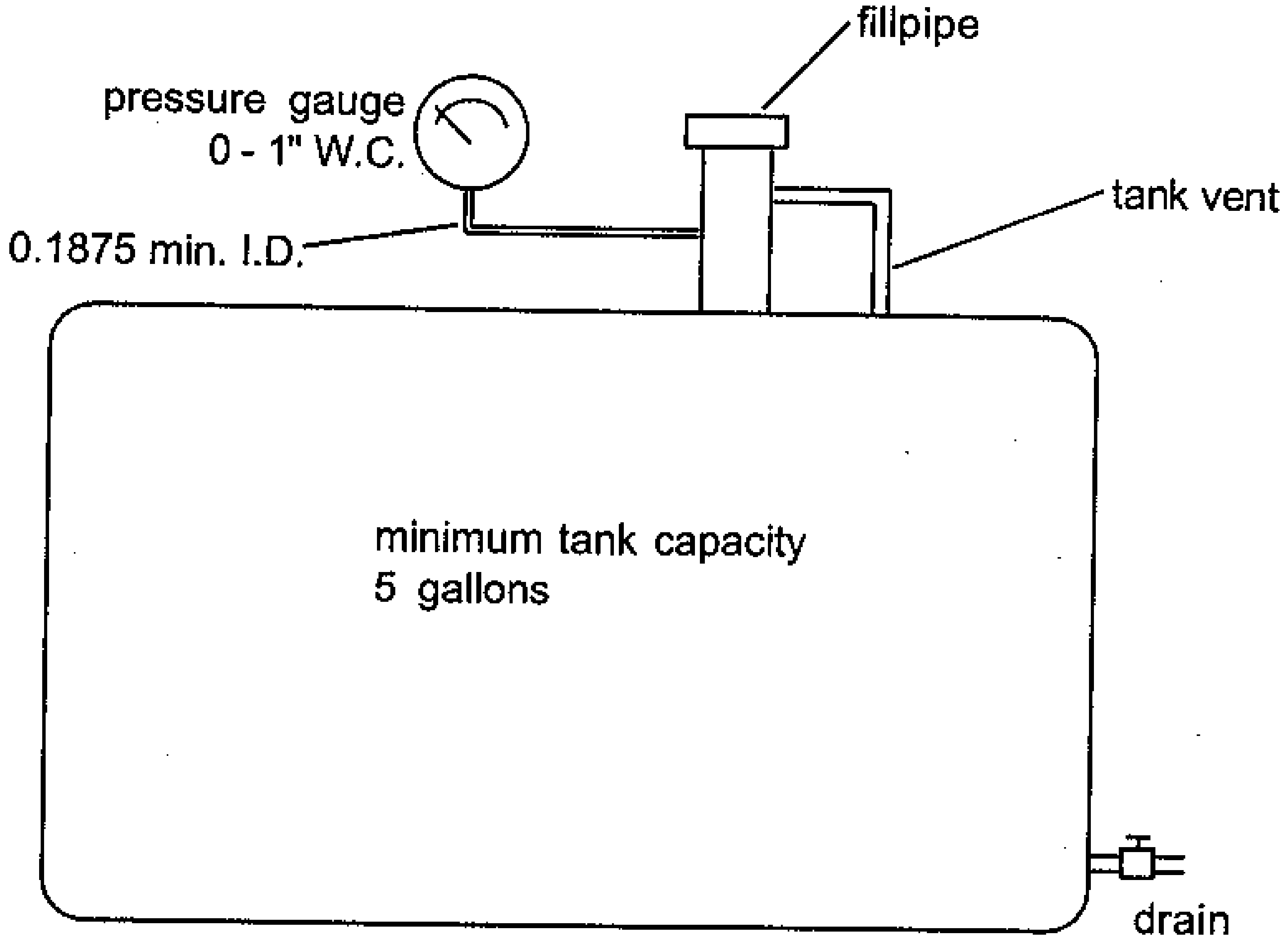


Figure 4 - Field Data Form

Facility Name & Address____
Inspector________________________ Date__________________
Vapor Recovery System Type____
Applicable Air Resources Board Executive Order # ___________________________
Dynamic Pressure Limits from Executive Order: Inches of H_2O CFH Nitrogen
__________ @ ____________
__________ @ ____________
__________ @ ____________

Pump Number	Gasoline Grade	Pressure, Inches of H_2O		Proc. 1 Nitrogen Flow, CFH		Proc. 3 Gallons Dispensed	Proc. 3 Time to Dispense	Proc. 3 Dispensing Rate, CFH

California Environmental Protection Agency

Air Resources Board

Vapor Recovery Test Procedure

TP - 201.5

DETERMINATION (BY VOLUME METER) OF AIR TO LIQUID VOLUME RATIO OF VAPOR RECOVERY SYSTEMS OF DISPENSING FACILITIES

Adopted: April 12, 1996

California Environmental Protection Agency
Air Resources Board

Vapor Recovery Test Procedure

TP-201.5

Determination (by Volume Meter) of Air to Liquid Volume Ratio of Vapor Recovery Systems of Dispensing Facilities

1 APPLICABILITY

Definitions common to all certification and test procedures are in:

D-200 Definitions for
Certification Procedures and
Test Procedures for
Vapor Recovery Systems

For the purpose of this procedure, the term "ARB" refers to the State of California Air Resources Board, and the term "ARB Executive Officer" refers to the Executive Officer of the ARB or his or her authorized representative or designate.

This test procedure can be used to quantify the air to liquid volume ratio (A/L) of a vapor recovery system. This test procedure is particularly well suited to dispensing facility vapor recovery systems which use bootless nozzles with circumferential holes near the front of their spouts; but it may be adapted for other systems.

This test procedure can be used to determine the performance specification for air to liquid volume ratio of a vapor recovery system during the certification process and subsequently to determine compliance with that performance specification for any installations of such a system.

When this test procedure is used to set a performance specification for a system, any deviations from the use of the equipment and procedures specified below shall be written into the certification report for such system if it is certified. Any compliance testing of a system shall be done according to this procedure, with appropriate adjustments for such deviations.

2 PRINCIPLE AND SUMMARY OF TEST PROCEDURE

The air to liquid volume ratio (A/L) of a vapor recovery system is, for a given dispensing episode, the quotient of the volume of air collected by a nozzle and the volume of liquid dispensed by that nozzle. In principle, any equipment and procedure which provides for the simultaneous measurement of air volume collected and liquid volume dispensed, from the same system, is a basis for determination of A/L for that system.

TP-201.5 measures A/L rather than the volume ratio of vapor (mixed with air) to liquid (V/L), because doing so is much more consistent, repeatable, and less expensive. A/L testing can be coordinated with efficiency testing to yield A/L performance specifications for compliance testing.

3 BIASES AND INTERFERENCES

There are no known biases or interferences inherent to the equipment and procedures specified; however several system parameters must be monitored and controlled so that this procedure can serve its intened purpose.

3.1 Non-Repeatable or Non-Representative Test Conditions

It is possible that system components could operate during testing in such a way that results are non-repeatable or are non-representative of subsequent installations of the system. To minimize such effects, the ARB test monitor shall note any relevant operating parameters for inclusion in the certification process as conditions on certification at a particular A/L ratio.

3.1.1 Non-Repeatable Test Conditions

For example, the liquid dispensing rate can introduce bias if it is non-repeatable; for many systems, the A/L performance varies with liquid flow rate.

In the procedures below, a maximum repeatable flow rate of liquid is required. If A/L performance varies with liquid flow rate for some system, it is necessary to place an upper limit on liquid flow rate in the ARB Executive Order.

(1) **(Liquid) Fuel Pumps**

To achieve repeatability, it is necessary to control the number of simultaneous dispensing episodes from a common liquid pump during certification testing. Such number shall be a performance specification in the ARB Executive Order so that subsequent installations of the system can be consistently tested.

(2) **(Air and Vapor) Assist Pumps**

To achieve repeatability, it is necessary to control the number of simultaneous dispensing episodes served by a common assist pump during certification testing. Such number shall be a performance specification in the ARB Executive Order so that subsequent installations of the system can be consistently tested.

3.1.2 Non-Representative Test Conditions

For example, nozzle quantities, qualities, and interactions can introduce bias if they are non-representative; for many systems, the A/L performance varies with such parameters.

In the procedures below, if more than one nozzle is served by the same assist pump, precautions are required to eliminate nozzle interactions which yield non-representative A/L performance. Within a system subject to certification testing, nozzle qualities must be representative of the nozzle qualities within subsequent installations.

To achieve representativeness, it may be necessary to control the nozzle quantities, qualities, and interactions during certification testing and subsequently by inclusion of specific requirements in the ARB Executive Order.

3.2 Condensation, Evaporation, and Other Factors

Different systems have different tendencies to condense and evaporate liquid in vapor lines. This and other factors can cause different A/L values in different modes of system operation. In consideration of such factors, the ARB Executive Officer may determine a different data collection protocol and a different data reduction protocol than the examples given in §§ 8 and 11.

4 SENSITIVITY, RANGE, AND PRECISION

The values of the determinations required by this test procedure are well within the limits of sensitivity, range, and precision of the specified equipment.

5 EQUIPMENT

Some of the equipment for testing a bootless nozzle is shown in:

Figure 1
A/L Volumetric Test Meter and

Figure 2
A/L Test Tank.

5.1 Air Volume Meter and Plumbing Hardware

The plumbing hardware shall connect the nozzle spout to a positive displacement air volume meter (e.g. Roots® meter) so that the air volume pulled into the collection holes in the spout can be measured with minimal pressure drop.

Use a calibrated positive displacement gas volume meter (e.g. a Roots meter) for measurement of volumetric flow rate through the sleeve.

Use rotary type positive displacement meter(s) with a back pressure limit (BPL) less than:

1.10 inches water column at a flowrate of 3,000 CFH down to
0.05 inches water column at a flowrate of 30 CFH for a meter with a rating over 1000 CFH and
0.70 inches water column at a flowrate of 800 CFH down to
0.04 inches water column at a flowrate of 16 CFH for a meter with a rating of or under 1000 CFH.

Meter(s) shall be equipped with taps accomodating the following equipment:

(1) taps on the inlet side for

  (a) a thermocouple with a range of 0 to 150 °F and

  (b) a pressure gauge with a range providing absolute pressure readings within 10 to 90% of the range (more than one gauge shall be used, if necessary) and

(2) taps on the inlet and outlet sides for a differential pressure gauge with a range of 0 to < 2x BPL (i.e. full scale shall be less than twice the back pressure limit) or any other range appropriate to allow detection of a pressure drop greater than the BPL.

5.2 Liquid Volume Meter

Use the meter on the liquid dispenser.

5.3 Portable Liquid Tank

A portable tank shall be used to receive dispensed liquid. The tank shall have sufficient volume so that 7.5 gallons can be received without triggering a premature shutoff. In the development of this procedure, a 25 gallon tank was adequate for two dispensing episodes between emptyings. The tank shall be on a wheeled cart and plumbed so that liquid received by the tank can be returned to the appropriate storage tank.

Figure 2, for example, shows an optional carbon scrubber arrangement which provides personnel protection from hazardous vapors and reduces emissions due to the performance of this test procedure.

5.4 Stop Watch

Use a stop watch accurate and precise to within 0.2 seconds.

6 CALIBRATION PROCEDURE

Follow the appropriate calibration procedures from TP-201.2.

7 PRE-TEST PROTOCOL

7.1 Location of Test Site

Prototype systems will be located within 100 miles of Sacramento for testing. Other locations may be accepted at the discretion of the ARB Executive Officer.

7.2 Specification of Test, Challenge, and Failure Modes

The specification of test, challenge, and failure modes such as the number of liquid transfer episodes, volume and volumetric rate of liquid transfer, storage tank volumes, etc. shall be done according to the principles of CP-201 § 5 for the testing and evaluation of vapor recovery equipment.

8 TEST PROCEDURE

The facility and system shall be prepared to operate according to any specified test, challenge, and failure modes.

The procedures below are for testing a bootless nozzle; with appropriate changes, these procedures can be used on other equipment. The procedure below shall be performed by at least two people familiar with the safety and mechanical principles of liquid dispensing equipment, especially for dispensing gasoline and other hazardous liquids.

8.1 General A/L Test Instructions

(1) Assemble the equipment shown in Figures 1 and 2, for example,. If more than one nozzle is served by the same assist pump, all nozzles other than the test nozzle shall be sealed vapor tight with, e.g., plastic bags and tape or rubber bands.

(2) Read and record the initial value on the air volume meter. Do not depend on using the terminal reading from a prior dispensing episode. The pressure drop across an appropriate volume meter is so low that a light breeze can change this value.

(3) Set the liquid meter and stopwatch to zero.

(4) Fully engage the dispensing lever and hold for the maximum repeatable flow rate of liquid. For most systems, there will be a brief pause before the liquid flows and is registered by the liquid meter.

(5) Start the stop watch when the liquid meter indicates liquid flow.

(6) Attempt to dispense 7.48 gallons (one cubic foot) of liquid and simultaneously:

(a) shut off liquid flow and

(b) stop the stop watch.

Read and record the liquid volume dispensed and the elapsed time.

(7) Read and record the final value on the air volume meter.

8.2 Certification Test Instructions

Different systems have different tendencies to condense and evaporate liquid in vapor lines. This and other factors can cause different A/L values in different modes of system operation. In consideration of such factors, the ARB Executive Officer may determine a different data collection protocol and a different data reduction protocol than the examples below. However, instructions must be determined before collection of final certification test data.

(1) Collect three sets of A/L test data per nozzle:

(2) from any nozzle (or nozzles) on any dispenser (or dispensers) used by the applicant for certification efficiency testing and

  (a) at three flow rates (e.g. repeatable minimum, average of repeatable minimum and repeatable maximum, and repeatable maximum.

  (b) Calculate the performance specification as an allowed range of A/L values according to one of the alternatives provided in § 11.

8.3 Compliance Test Instructions

Different systems have different tendencies to condense and evaporate liquid in vapor lines. This and other factors can cause different A/L values in different modes of system operation. In consideration of such factors, the ARB Executive Officer may determine a different data collection protocol and a different data reduction protocol than the examples below. However, instructions may not be changed after certification.

(1) Collect one set of A/L test data per nozzle:

(2) Compare the resulting A/L value with the allowed range of A/L values given as a performance specification in the ARB EXecutive Order for the tested system.

  (a) If the resulting value is in the allowed range of A/L values, the system complies.

  (b) If the resulting value is not in the allowed range of A/L values, collect two more sets of A/L test data and calculate the average A/L for all three sets.

    (i) If the resulting value is in the allowed range of A/L values, the system complies.

    (ii) If the resulting value is not in the allowed range of A/L values, the system does not comply.

9 QUALITY ASSURANCE / QUALITY CONTROL (QA/QC)

This section is reserved for future specification.

10 RECORDING DATA

This section is reserved for future specification.

11 CALCULATING RESULTS

Different systems have different tendencies to condense and evaporate liquid in vapor lines. This and other factors can cause different A/L values in different modes of system operation. In consideration of such factors, the ARB Executive Officer may determine a different data reduction protocol than the examples below. However, all calculation protocols must be determined before collection of final certification test data.

11.1 A/L Values

Calculate A/L for each test of a dispensing episode:

$$A/L = \frac{\text{(volume of air collected)}}{\text{(volume of liquid dispensed)}}$$

11.2 Performance Specification

The performance specification shall be expressed as an allowed range of A/L values. The performance specification range shall be the mean value of A/L ±10% of the mean.

11.3 Alternative Performance Specification

This performance specification may be used after an engineering evaluation by the ARB Executive Officer has determined that it is necessary to statistically account for the variance of A/L values for a system.

The performance specification shall be expressed as an allowed range of A/L values. The performance specification shall be the same as the 95% confidence interval for the expectation value of a single observation of A/L.

For example, assume that a nozzle was tested with the following results for A/L:

observation number	A/L
1	1.02
2	0.99
3	1.02

(1) Find the mean value of A/L.

$$\bar{x} = \frac{1.02 + 0.99 + 1.02}{3} = 1.01$$

(2) Find the sample standard deviation of the mean value of A/L.

$$s = \sqrt{\frac{(1.02 - \bar{x})^2 + (0.99 - \bar{x})^2 + (1.02 - \bar{x})^2}{(3 - 1)}} = 0.0173$$

(3) Find the 95% confidence interval for the expectation value of a single observation of A/L using Student's t Statistic and assuming a normal distribution of A/L values for all system nozzles.

Note that for three observations, there are two degrees of freedom and the Student's t Statistic is 4.303 for a 95% confidence interval.

$$95\%\ \text{c.i.} = \bar{x} \pm (t\ s) = 1.01 \pm 0.075$$

Other values of t are provided below for convenience:

number of observations	t
4	3.182
5	2.776
6	2.571
7	2.447
8	2.365
9	2.306
10	2.262
15	2.145
30	2.045

12 REPORTING RESULTS

12.1 Certification Report

12.1.1 Performance Specification

Report:

(1) the mean value of A/L,

(2) 10% of the mean value of A/L, and

(3) the mean value of A/L± 10% of the mean.

Report (3) as the performance specification which is the allowed range of A/L values for subsequent installations of the system.

12.1.2 Alternative Performance Specification

Report:

(1) the mean value of A/L,

(2) the variance of the mean value of A/L, and

(3) the 95% confidence interval for the expectation value of a single observation of A/L using Student's t Statistic and assuming a normal distribution of A/L values for all system nozzles.

Report (3) as the performance specification which is the allowed range of A/L values for subsequent installations of the system.

12.2 Compliance Test Report

Report:

(1) the number of nozzles at the dispensing facility which do not meet the performance specification and

(2) the total number of nozzles at the dispensing facility.

Report any other system operating parameters technically pertinent to the A/L performance specification as required by the certification procedure.

13 ALTERNATIVE TEST PROCEDURES

Test procedures, other than specified above, shall only be used if prior written approval is obtained from the ARB Executive Officer. In order to secure the ARB Executive Officer's approval of an alternative test procedure, the applicant is responsible for demonstrating to the ARB Executive Officer's satisfaction that the alternative test procedure is equivalent to this test procedure.

(1) Such approval shall be granted on a case-by-case basis only. Because of the evolving nature of technology and procedures for vapor recovery systems, such approval shall not be granted in subsequent cases without a new request for approval and a new demonstration of equivalency.

(2) Documentation of any such approvals, demonstrations, and approvals shall be maintained in the ARB Executive Officer's files and shall be made available upon request.

14 REFERENCES

This section is reserved for future specification.

15 EXAMPLE FIGURES

Each figure provides an illustration of an implementation which conforms to the requirements of this test procedure; other implementations which so conform are acceptable, too. Any specifications or dimensions provided in the figures are for example only, unless such specifications or dimensions are provided as requirements in the text of this or some other required test procedure.

Figure 1
A/L Volumetric Test Equipment

Figure 2
A/L Test Tank

FIGURE 1

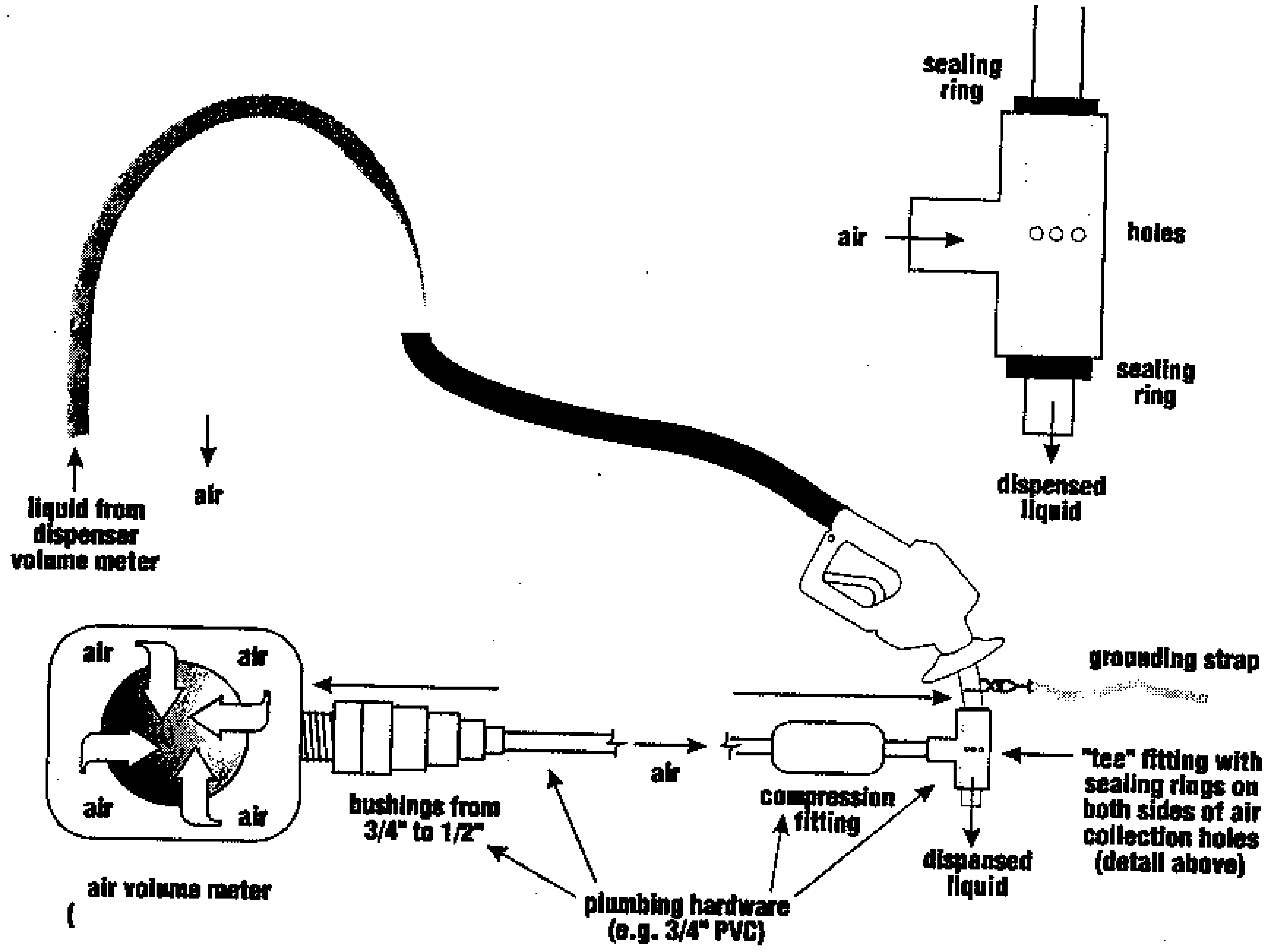


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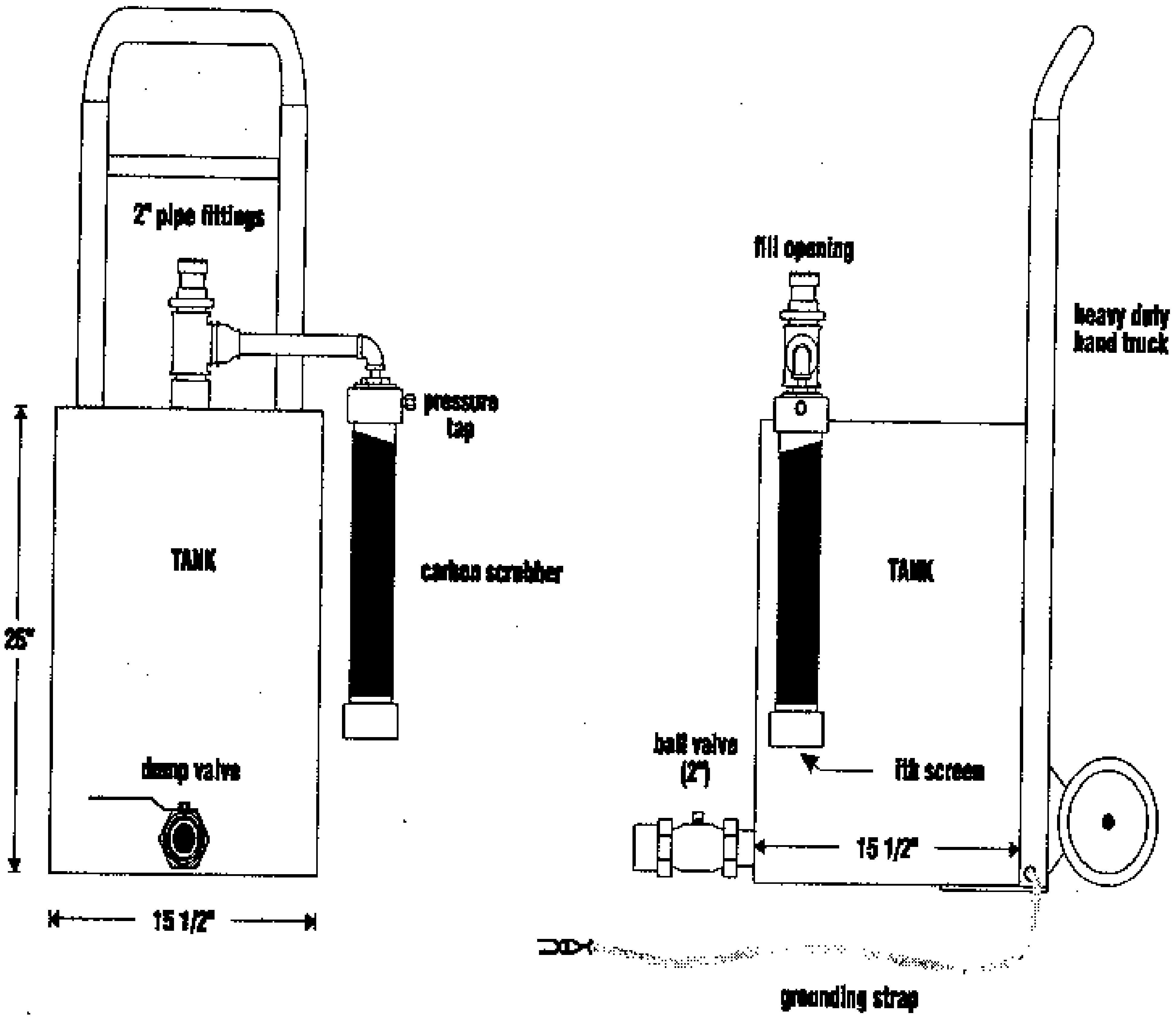
FIGURE 2

A/L Testing Tank



This design can meet the performance specifications of this procedure, any other design which meets such specifications is acceptable.

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