APPENDIX C

DILUTION PROBE VERIFICATION
State of California
California Environmental Protection Agency
AIR RESOURCES BOARD

EVALUATION TEST REPORT

Organic Vapor Analyzer
Dilution Probe Validation Test

Project No. C97-014

Engineering and Laboratory Branch
Monitoring and Laboratory Division

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This Evaluation Test Report has been reviewed and approved by the Air Resources Board (ARB) staff. Approval does not necessarily signify that the contents reflect the views and policies of the ARB, nor does the mention of trade names or commercial products constitute endorsement or recommendation for use.
Organic Vapor Analyzer
Dilution Probe Validation Testing

Introduction

The following sections discuss results of tests performed according to the July 15, 1997 Protocol which was distributed by fax to members of the CAPCOA Fugitive Hydrocarbon Emissions Subcommittee. The goal of the Protocol is to evaluate and compare the performance of the Foxboro Organic Vapor Analyzer (OVA) with and without its dilution probe (dilutor) on a variety of leaks simulated in the laboratory. Although the dilutor is designed to extend the range of the OVA beyond the one percent hydrocarbon concentration limit of the normal probe, the dilutor should work below one percent as well. The ARB had no convenient means of generating validated concentrations beyond one percent hydrocarbon. Therefore the performance of the dilutor was compared against the normal probe in the concentration range at and below one percent hydrocarbon, using the normal probe concentration as the reference concentration. Data in this report was obtained from tests conducted from August 14 to 27, 1997.

Summary of Results

In all of the cases investigated, the dilutor tip did not consistently perform with a 10 to 1 dilution ratio even though the dilutor is designed to have a 10 to 1 dilution ratio. Testing was done using a single OVA (Model OVA 108 S/N 2503) and may not be representative of all OVA instruments.

Conclusions

The dilutor tip can not be depended upon to yield quantitative readings for leaks beyond one percent based on its performance on leaks at and below one percent hydrocarbon.

Experimental Details

A. Calibrations

Calibration of Rotameters

Chart A shows the correlation between rotometer readings at 0 psig with bubble meter readings at 0 psig. The valve downstream from the rotometer is fully open for these readings.

Chart B shows the correlation between rotometer readings at 10 psig with bubble meter readings at 0 psig. The valve downstream from the rotometer is partially closed for these readings. The correlation in Chart B was used in further work discussed below because having positive gauge pressure on the rotameters is a better model of the mechanics of leaking components. It is just good luck that it also yields a more precise correlation.

Calibration of OVA

The OVA was calibrated using certified bottles of 10,000 ppmv and 40 ppmv methane-in-air gas mixtures and Tedlar sample bags. First a Tedlar bag was filled with 10,000
ppmv calibration gas mixture and then the OVA pump drew the mixture through tubing, a rotameter, and the OVA flame ionization detector (FID). As shown in Figure 2 of the Protocol, the flow through the normal tip of the OVA was 1400 cc/min; the flow through the dilutor tip of the OVA was 45 cc/min for a flow ratio of 31 to 1 even though the dilutor is designed to have a 10 to 1 dilution ratio.

**Calibration of Dilutor**

Using the setup shown in Figure 3 of the Protocol, the OVA with the dilutor tip was forced to read 1,000 ppmv when drawing from the 10,000 ppmv sample bag by adjusting the restricting valve to the side arm tube. First a Tedlar bag was filled with 10,000 ppmv calibration gas mixture and then the OVA pump drew the mixture through tubing, a rotameter, and the OVA flame ionization detector (FID). Then a 40 ppmv bag was attached and the stable reading was 15 ppmv for a dilution ratio of 2.7 even though the dilutor is designed to have a 10 to 1 dilution ratio.

Using the setup shown in Figure 4 of the Protocol, a total flow of 525 cc/min was recorded for the dilutor tip and side arm tube together. Using this setup, the stable reading was 20 ppmv for a dilution ratio of 2.0 even though the dilutor is designed to have a 10 to 1 dilution ratio.

During the execution of the Protocol, two more methods were tried to test the performance of the dilutor: the methods of serial dilutions performed with pure air and serial concentrations performed with a sample bag and syringes of pure methane. The results, shown in Chart C, have the dilution ratio clustered between 5.5 and 7.5 even though the dilutor is designed to have a 10 to 1 dilution ratio.

**B. Generating Leaks**

**Setup of a Leaking Component**

The bottled gas used for this setup shown in Figure 5 of the Protocol is pure methane gas. The results plotted in Chart D are for the normal probe and dilutor probe stuck into the opening of a quarter-inch valve. Thus, there was zero distance between the leak and the probe tips. This may account for the fact that the dilutor probe value was systematically higher than the normal probe value even though the dilutor is designed to have a 10 to 1 dilution ratio.

**Setup of a Leak Mixed with Air**

The setup shown in Figure 6 of the Protocol was used to test the performance of the dilutor tip on diluted leaks. A two inch diameter, twelve inch long tube was placed around the leak outlet tube and the normal tip and dilutor tip were used to sample the diluted leaks six inches from the leak opening. In Chart E, the dilution ratio for the dilutor tip ranged from 0.04 to 100 even though the dilutor is designed to have a 10 to 1 dilution ratio.

In Chart F, the diluted leak values for the normal tip and dilutor tip are compared versus leak flow rate. In 5 out of seven cases, the dilutor tip measured higher values than the normal tip, even though the dilutor is designed to have a 10 to 1 dilution ratio.

**C. Measurement and Recording**

Three ways to introduce gas mixtures to the probes were used, as described in each subsection: from a tube connected to a Tedlar sample bag, from direct contact of a probe to the
leak source, and six inches away from a diluted source. The hydrocarbon concentration of each mixture was then measured by the FID of the OVA. After fifteen seconds of constant reading from the dial face of the OVA, the concentration was recorded in ppmv or percent, as appropriate.
RM | BM
---|---
3.0 | 9.0
3.5 | 8.0
4.0 | 9.5
6.0 | 12.0
6.0 | 15.0
8.0 | 16.0
8.0 | 18.0
10.0 | 22.0
11.0 | 25.0
11.5 | 19.0
14.0 | 29.0
15.0 | 26.5
17.0 | 31.0
20.0 | 30.0
21.0 | 41.0
21.0 | 42.0
25.0 | 46.0
27.0 | 39.0
28.0 | 36.5
33.0 | 55.0
35.0 | 45.5
38.0 | 64.5
39.0 | 54.0
43.0 | 72.0
45.0 | 75.0
62.0 | 89.0
66.0 | 88.0

SLOPE: 1.34
INTCPT: 6.81

Chart A - Rotameter Calibration

y = bubble meter @ 0 psig
x = rotameter @ 0 psig
RM  BM
7.5  20.0
11.0  33.0
14.5  41.0
19.0  47.0
26.0  59.5
35.0  85.5
30.5  69.0
38.0  90.0

SLOPE: 2.17
INTCPT: 6.33

Chart B - Rotameter Calibration

y = bubble meter @ 0 psig
x = rotameter @ 10 psig
Chart C - Sample Bag

y = dilution ratio: (w/o)/(w)

x = normal probe: (w/o); ppmv

empty circles = serial dilutions
filled circles = serial concentrations
flow rate (w/o)
7.2  500
8.5  1900
10.2 4200
10.9 5300
8.9  2800

flow rate (w)
7.2  850
8.5  2500
10.2 6200
10.9 9500
8.9  4000

Chart D - Leaking Valve

y = concentration, ppmv
x = leak flow rate; cc/min
diamonds = normal probe: (w/o)
circles = dilutor probe: (w)
<table>
<thead>
<tr>
<th>w/o</th>
<th>(w/o)/(w)</th>
</tr>
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<tbody>
<tr>
<td>150</td>
<td>0.30</td>
</tr>
<tr>
<td>4000</td>
<td>100</td>
</tr>
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<td>30</td>
<td>0.04</td>
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<tr>
<td>10000</td>
<td>1.67</td>
</tr>
<tr>
<td>10000</td>
<td>5.0</td>
</tr>
</tbody>
</table>

**Chart E - Dilution Tube**

y = dilution ratio: (w/o)/(w)

x = normal probe: (w/o); ppmv
Chart F - Dilution Tube

y = concentration; ppmv

x = leak flow rate; cc/min

diamonds = normal probe: (w/o)
circles = dilutor probe: (w)
OVA Test Protocol

Monitoring and Laboratory Division
California Air Resources Board
California Environmental Protection Agency

Test Objectives: The goal of this protocol is to evaluate and compare the performance of the Foxboro Organic Vapor Analyzer (OVA) with and without its dilution probe (dilutor) on a variety of leaks simulated in the laboratory.

1 Equipment List

1.1 Cylinder of 100% Methane with Two Stage Regulator
1.2 Rotameters (0 to 80 cc/min)
1.3 Soap Film Volume Meter(s) (0 to 100 cc/min)
1.4 Pressure Gauge (0 to 30 psi)
1.5 Leaking Components
1.6 Foxboro OVA Model 108 with Dilution Probe

2 Pre-Test Preparation

2.1 Calibration of Rotameters

Calibrate the rotameters at the elevated pressure (e.g., 10 psig) for each type of hydrocarbon that will be used in the test. If more than one elevated pressure will be used (e.g., 20 psig), then calibrate the rotameters at each pressure.

2.1.1 Assemble equipment as shown in Figure 1.
2.1.2 Close Valve A and establish target pressure (e.g., 10 psig) at inlet to rotameters by opening hydrocarbon supply cylinder and adjusting pressure regulator. CAUTION: DO NOT OVER-PRESSURIZE ROTAMETERS.
2.1.3 Slowly open Valve A to start flow of hydrocarbon through the system.
2.1.4 Make any necessary adjustments so that pressure at inlet to rotameters remains constant.
2.1.5 Measure the actual flow rate of hydrocarbon at outlet of Valve A using a soap film volume meter.

February 22, 1999
2.1.6 Repeat procedure throughout the flow range of the rotameters.

2.1.7 Construct a chart comparing the rotameters' readings with the actual flow rates.

2.2 Calibration of OVA

2.2.1 Calibrate OVA according to manufacturer's instructions using both a high range calibration gas (approximately 10,000 ppmv methane-in-air) and a low range calibration gas (approximately 25 to 100 ppmv methane-in-air).

2.2.2 Measure the flow into the OVA probe tip using setup shown in Figure 2. Verify that the OVA reading of the calibration gas has remained constant.

2.3 Calibration of Dilutor

2.3.1 Connect dilutor to the OVA using the setup shown in Figure 3. Connect dilutor tip to 10,000 ppmv calibration gas.

2.3.2 Adjust restricting valve to side arm tube until OVA reads 1,000 ppmv.

2.3.3 Connect dilutor tip and side arm tube to rotamer using "Y" connector using the setup shown in Figure 4.

2.3.4 Measure and record total flow into the OVA (combined flow of dilutor tip and side arm tube) and the concentration on the OVA dial.

2.4 Setup of a Leaking Component

STOP: Calibrate the OVA and the dilution probe if this has not been done before.

2.4.1 Assemble equipment as shown in Figure 5.

2.4.2 Establish target pressure (e.g., 10psig) at inlet to rotameters and inside of the component to be tested by opening hydrocarbon supply cylinder and adjusting pressure regulator. CAUTION: DO NOT OVER-PRESSURIZE ROTAMETERS.

2.4.3 Initiate leak by opening valve, loosening packing, loosening threads, or whatever procedure is appropriate.

2.4.4 Make any necessary adjustments so that pressure at inlet of rotameters and inside of component remains constant.

2.4.5 Calculate the volume (in cubic centimeters) inside the tubing and component downstream from the rotameters.

2.4.6 Flush the tubing and component with pure hydrocarbon until at least 5 times the volume of the system has escaped through the leak. (This only needs to
be done once if pressure is maintained inside of the leaking component throughout the test procedure.

3 Standard Operating Procedures

3.1 Measuring Leak without Dilution Probe

3.1.1 The OVA probe tip will be placed at the point of highest reading.

3.1.2 The OVA probe tip will be left at the point of highest reading for at least 15 seconds.

3.1.3 The OVA reading will be recorded along with the hydrocarbon flow rate as indicated by the rotameter.

3.2 Measuring Leak with Dilution Probe

3.2.1 The OVA dilution probe tip will be placed at the point of highest reading.

3.2.2 The OVA dilution probe tip will be left at the point of highest reading for at least 15 seconds.

3.2.3 The OVA reading will be recorded along with the hydrocarbon flow rate as indicated by the rotameter.

3.3 Range of Hydrocarbon Leak Rates

Hydrocarbon will be caused to leak at rates throughout the range of 1 cc/minute to 100 cc/minute.

3.4 Reporting the Data

A graph will be prepared showing the known leak rate on the "x" axis, and both of the OVA readings (without and with the dilution probe in place) on the "y" axis.

4 Optional Test of Monitoring Distance

The effect of distance on the OVA probe tip and the dilutor tip can be determined by moving the tip away from the leaking component. Suggested distances are 0.25 cm, 0.5 cm, and 1.0 cm.
Optional Test of the Effect of Mixing Air with a Leak before Collection by the Probe Tip

Hydrocarbon leaking from Valve A will be conveyed via a small tube to the approximate center of a large tube where air can mix with the leak; see Figure 6. Measurement of the mixture will be done using both the regular OVA tip and the dilutor tip.
Figure 1
Calibration of Rotameters

Figure 2
Setting Probe to 10,000 ppmv

10,000 ppmv Calibration Gas
Figure 3
Setting Dilutor to 1,000 ppmv

Figure 4
Using "Y" Connector
Figure 5
Setup of a Leaking Component

Figure 6
Setup of a Leak Mixed with Air