THREE STAGE GAS DILUTION SYSTEM
FOR CALIBRATION OF AIR QUALITY
INSTRUMENTATION

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PRICES SUBJECT TO CHANGE
Tests were conducted on a three stage gas dilution system to evaluate the performance of the system for preparation of gas mixtures in the range of concentrations from 1 to 200 ppm. The test gas used in the evaluation was carbon monoxide. Mixtures of carbon monoxide and air were prepared in the gas dilution system and analyzed on stream with a commercially available infrared CO detector. Results showed the gas dilution system to be accurate to within 5, 3, and 2 percent in the respective ranges of 1-20 ppm, 20-50 ppm, and 50-200 ppm.
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FOR CALIBRATION OF AIR QUALITY INSTRUMENTATION

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SUMMARY

Tests were conducted on a three-stage gas dilution system to evaluate the performance of the system for preparation of gas mixtures in the range of concentrations from 1 to 200 ppm. The test gas used in the evaluation was carbon monoxide. Mixtures of carbon monoxide and air were prepared in the gas dilution system and analyzed on-stream with a commercially available infrared CO detector. Results showed the gas dilution system to be accurate to within 5, 3, and 2 percent in the respective ranges of 1-20 ppm, 20-50 ppm, and 50-200 ppm.

INTRODUCTION

In the field of air pollution measurements, there is a continual need for calibration of various monitoring instruments. For gas monitoring instrumentation, these calibrations can range from concentrations of a few ppm, as for carbon monoxide, to hundreds of ppm, as for carbon dioxide. In many cases, it is desirable to have a universal gas calibration system in which various gases may be mixed over a wide range of concentrations. This paper describes a three-stage, gas-dilution system theoretically capable of mixing gases from the pure state to concentrations below 0.1 ppm. Experimental results are shown validating the ability of the gas dilution system to accurately mix carbon monoxide over the range of 1 to 200 ppm.
SYMBOLS

$D_1$ - first stage dilution factor
$D_2$ - second stage dilution factor
$D_3$ - third stage dilution factor
$F_1$ - flow rate of first stage diluent, cc/min
$F'_2$ - flow rate of first stage test gas, cc/min
$F_3$ - flow rate of second stage diluent, cc/min
$F'_4$ - flow rate of second stage test gas, cc/min
$F_5$ - flow rate of third stage diluent, cc/min
$F'_6$ - flow rate of third stage test gas, cc/min
$F'_7$ - output flow rate of test gas from dilution system, cc/min
$X_f$ - concentration of final gas stream, ppm
$X_i$ - concentration of initial test gas supply, ppm

SYSTEM DESCRIPTION

The flow schematic of the gas dilution system is shown in figure 1. The gas supply consists of a cylinder of diluent gas and a cylinder of test gas, each equipped with appropriate dual stage regulators capable of supplying gas at two to three atmospheres gage pressure. The diluent gas is normally an inert and relatively inexpensive gas that contains little, preferably none, of the test gas. For low ppm mixtures, using pure gas as the test gas supply, the diluent gas can be consumed at a rate of 20 to 40 times the flow rate ($F'_7$) of the final gas stream. Typically, air or nitrogen is used for the diluent gas. To reduce the quantity of diluent gas consumed, the pure test gas may be replaced with a pre-mixed concentration of test gas; for example, 1000 ppm. With the current system it has been calculated that 2
mixtures as low as 0.04 ppm can be prepared using pure gas as the test gas supply. Lower concentrations are theoretically possible with the 1000 ppm test gas supply. As shown in figure 1, a flowmeter (F'7) and metering valve (V 7) are provided at the exit from the mixing system to control the flow rate of test gas into the instrument being calibrated. Under normal operating conditions, V 7 has little influence on the concentration of the test gas stream.

Each stage of the mixing system (figure 1) consists of six main elements: two metering valves, two flowmeters, a flow mixer, and a vent valve. The metering valves must provide precise flow regulation for the range of flow rates anticipated in each stage. Mass flowmeters with flow ranges selected based on the desired dilution factors are used for each stage. Table I lists the flowmeter ranges used in the current system. The flow mixers must provide an adequate chamber for uniform mixing of the test gas. The current system uses glass bulbs filled with glass wool. The vent valves function as a means of disposing of surplus gas not needed in the latter stages of dilution. Construction materials for tubulation, valves, flowmeters, etc., are selected according to the reactivity of the gas species of interest. In the present system tygon, brass, glass, and stainless steel are used in the dilution side of the mixing system and teflon, monel, and glass are used on the test gas side.

The first two stages of the system are designed for a maximum dilution factor of 500 to 1; the third stage, 100 to 1. Therefore, the overall maximum dilution factor is $2.5 \times 10^7$ to 1 which will yield a 0.04 ppm mixture when a pure test gas supply is used. The dilution factors for stages one through three are given by equations 1 through 3. The concentration of the
final gas mixture is given by equation 4.

\[
D_1 = \frac{F_1 + F_1'}{F_1'} \quad (1)
\]

\[
D_2 = \frac{F_3 + F_4'}{F_4'} \quad (2)
\]

\[
D_3 = \frac{F_5 + F_6'}{F_6'} \quad (3)
\]

\[
X_F = X_i / (D_1 \times D_2 \times D_3) \quad (4)
\]

In equations 1 through 3, it is assumed that the diluent gas contains none of the test gas. In addition, the flow rates in equation 1 through 3 are true flow rates already corrected by the manufacturer's calibration for gas composition. Figure 2 shows the gas dilution system consoles. Console 1 houses the flowmeter readout systems. Console 2 contains the metering valves, flowmeters, flow mixers, vent valves, and associated tubulation. Figure 3 shows the construction and layout of console 2. The cabling to interface the two consoles is shown in figure 4.

CALIBRATION STANDARD

For calibration of the gas mixing system, carbon monoxide (CO) was selected as the test gas. Research grade CO (99.98%) was used as the test gas supply and dry air was used as the diluent gas. The air was commercially obtained, and contained 0.5 ppm CO. For concentrations greater than 20 ppm,
no correction was made for the 0.5 ppm CO impurity in the diluent gas supply. The output from the mixing system was analyzed with a commercially available CO detector described in reference 1. Basically it is a non-dispersive infrared analyzer using dual-isotope fluorescence detection principles. The detector was calibrated prior to the test using commercially prepared and certified carbon monoxide in nitrogen standards. The standards used in the pretest calibration were: 0, 0.5, 5.5, 19, 47, 99, and 190 ppm CO. Table II summarizes the results of this pretest calibration. The flow rate of gas into the CO detector was 1000 cc/min for all tests.

RESULTS

The results of the two test runs used to compare the mixing system with the CO detector are shown in Table III. These runs were taken on successive days. Column 1 is the calculated mixture concentrations obtained from equations 1 through 4. The following points should be noted:

(1) The largest discrepancy between the mixing system and the CO detector for a single observation occurred at the 174 ppm mixture and was 6 ppm. The largest percentage discrepancy occurred at 5 ppm and was 10 percent.

(2) Averaging the two runs at each ppm, the largest discrepancy was 4 ppm, again at the 174 ppm mixture; the largest percentage discrepancy was 4.5 percent at the 26.4 ppm mixture.

(3) Statistically, the mixing system and CO detector agreed to within 5, 3, and 2 percent, respectively, in the CO ranges of 1-20, 20-50, and 50-200 ppm.

(4) The response time of the mixing system to concentration changes
was good, if not better, than the stated response time for the CO detector (Table II), provided concentration changes were made with adjustments to third stage only. Concentration changes requiring first and second stage adjustments took several minutes as a first stage adjustment typically required numerous second and third stage adjustments and several readjustment in the first stage in order to obtain the desired dilution ratio.

(5) Varying the flow rate at exit from the mixing system (F'7) had little influence on the concentration of the mixture provided F'7 did not approach 60 to 70 percent of F5 plus F'6.

(6) Once a mixture was set on the gas mixing system, it would remain stable for several hours.

Points 4 through 6 are not supported by specific data in the report, but summarize additional experience obtained from several months of operation of the system.

CONCLUDING REMARKS

In conclusion, the gas dilution system has been shown by these results to be accurate to at least 5 percent in the range of 1 to 200 ppm. Evaluation of the dilution system below 1 ppm requires a more accurate CO detector than that used in the present investigation. In the authors' opinion, the 5 percent accuracy is a upper limit influenced partly by the capabilities of the CO detector. In addition, it is speculated by the authors that further studies below the 1 ppm level would result in accuracies similar to those documented in this report.
REFERENCE


<table>
<thead>
<tr>
<th>Flow Stage</th>
<th>Diluent Flow, cc/min</th>
<th>Test Gas Flow, cc/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stage 1</td>
<td>0-10,000 (F1)**</td>
<td>0-100 (F-2)</td>
</tr>
<tr>
<td>Stage 2</td>
<td>0-10,000 (F3)</td>
<td>0-100 (F-4)</td>
</tr>
<tr>
<td>Stage 3</td>
<td>0-5,000 (F5)</td>
<td>0-100 (F-6)</td>
</tr>
<tr>
<td>Output *</td>
<td>----</td>
<td>0-5000 (F-7A)</td>
</tr>
<tr>
<td>Output</td>
<td>----</td>
<td>0-500 (F-7B)</td>
</tr>
</tbody>
</table>

* Select flowmeter range to match flow requirements of monitoring instrument

** See figure 1 for flowmeter location
TABLE II - SUMMARY OF PRETEST CALIBRATION OF CO DETECTOR

<table>
<thead>
<tr>
<th>CO ppm Range</th>
<th>Accuracy</th>
<th>Response Time to 90% of Reading</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 - 20 ppm</td>
<td>± 0.2 ppm</td>
<td>40 seconds</td>
</tr>
<tr>
<td>0 - 50 ppm</td>
<td>± 0.5 ppm</td>
<td>30 seconds</td>
</tr>
<tr>
<td>0 - 100 ppm</td>
<td>± 1.0 ppm</td>
<td>25 seconds</td>
</tr>
<tr>
<td>0 - 200 ppm</td>
<td>± 2.0 ppm</td>
<td>20 seconds</td>
</tr>
</tbody>
</table>

* Range is selected to correspond with range settings on the CO detector

** Response time is dependent on the range selected for instrument use
<table>
<thead>
<tr>
<th>Theoretical Mixture (ppm)</th>
<th>Run 1</th>
<th>Run 2</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CO Detector reading (ppm)</td>
<td>Difference (ppm)</td>
</tr>
<tr>
<td>1.20</td>
<td>1.2</td>
<td>0</td>
</tr>
<tr>
<td>2.15</td>
<td>2.3</td>
<td>+0.15</td>
</tr>
<tr>
<td>2.33</td>
<td>2.5</td>
<td>+0.17</td>
</tr>
<tr>
<td>2.73</td>
<td>2.9</td>
<td>+0.17</td>
</tr>
<tr>
<td>5.0</td>
<td>5.5</td>
<td>+0.50</td>
</tr>
<tr>
<td>7.6</td>
<td>8.0</td>
<td>+0.40</td>
</tr>
<tr>
<td>13.2</td>
<td>13.5</td>
<td>+0.30</td>
</tr>
<tr>
<td>17.2</td>
<td>17.5</td>
<td>+0.30</td>
</tr>
<tr>
<td>26.4</td>
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<td>-0.40</td>
</tr>
<tr>
<td>48.0</td>
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<td>0</td>
</tr>
<tr>
<td>65.0</td>
<td>65.0</td>
<td>0</td>
</tr>
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<td>-1.0</td>
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<td>120</td>
<td>-2.0</td>
</tr>
<tr>
<td>147.0</td>
<td>143</td>
<td>-4.0</td>
</tr>
<tr>
<td>174.0</td>
<td>168</td>
<td>-6.0</td>
</tr>
<tr>
<td>194.0</td>
<td>193</td>
<td>-1.0</td>
</tr>
</tbody>
</table>

*Difference = CO detector reading - theoretical mixture*
Figure 1. - Schematic of Gas Dilution System
Figure 2. - Gas Dilution System
Figure 3. - Console 2

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Figure 4. - Console 2 Interfaced With Console 1

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