

The Performance of a Dynamic Atmosphere Generation System

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A controlled test atmosphere system for gaseous pollutants was designed and constructed. For a reliable characterization of indoor air pollution, a suitable set of sampling and analysis procedures has to be devised and accomplished. The precision and accuracy of the measurements must be determined exactly for a correct interpretation of the results.⁽¹⁻³⁾ The two main difficulties appear to be the actual generation of the individual standard and the preparation of physico-chemically thoroughly characterized mixtures. This system utilized two methods for generation of dynamic standards: permeation tubes⁽⁴⁻⁹⁾ and gas saturators. Special care also was devoted to the achievement of both a good time stability of the concentrations of the standard mixtures and a satisfactory agreement between expected and measured concentration values.

System Design

The system is comprised of four sections: 1) standard generation; 2) mixture homogenization and humidification; 3) sampling; and 4) analytical control (see Figure 1).

The generation section includes a permeation tube chamber, thermostated at $35 \pm 0.1^\circ\text{C}$, and two saturation chambers (Figure 2) with temperature regulation adjustable within $\pm 0.25^\circ\text{C}$ ranging from ambient temperature to 99°C . Sample gas concentration in the generated mixture may be varied, adjusting the relative flow rates of carrier air (low flow) and diluent air (higher flow) into a mixing device.

The homogenization section was developed by modifying a 300-L AISI 304 Mazzali Climatest chamber in which temperature may be regulated within $\pm 1^\circ\text{C}$ over 0° to 100°C and relative humidity within $+5/-2\%$ over 18% to 92% range. The chamber is provided with a manipulator allowing exposure of passive samplers and testing of portable instruments.

The sampling section is composed of three lines with the flow rate adjustable within $\pm 1\%$ of the full scale value over 0 to 100 mL/min range.

The analytical control section includes an automatic sampling valve (eight port rotary valve Danj RSU 108), a gas chromatograph (GC) and a data processing system (Shimatzu GC MINI2, F.I.D. with Shimatzu Chromatopac CR 3A).

Performance Data

The standard generation and the homogenization sections of the controlled atmosphere system were tested extensively and modified repeatedly to achieve satisfactory performance. The goals were to minimize the differences between expected and measured concentration values and optimize the time stability of concentrations in the generated gas mixtures.

TFE Teflon® tubes with an external diameter of 6.35 mm, a 0.76 mm wall thickness and a 2 cm effective length were used for benzene and toluene. FEP Teflon tubes were chosen having the same diameter and wall thickness, and a 6 cm effective length for m-xylene.

The emission rates for the different samples were determined weighing the tubes about every 50 hr over a period of approximately 800 hr. The final value of emission rate is equal to 96.5% of the one calculated according to the permeability coefficient supplied by the tube manufacturer.

The air saturators emission rates were obtained from the gas chromatographically determined output concentrations and are compared with the calculated emission rates at three different temperatures as a function of the carrier air flow rate. The rates were found to be satisfactory.

Inside the homogenization/exposure chamber, the desired pollutant concentration is attained by evaporating a weighed quantity of the pollutant itself before letting the standard gas mixture in. Several tests demonstrated that a pollutant

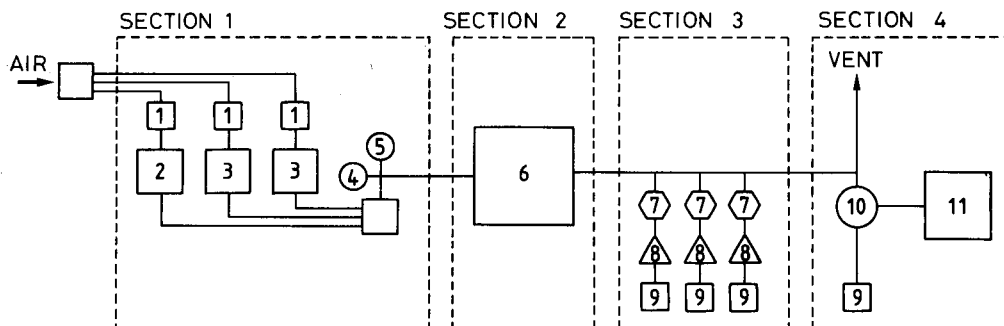


Figure 1 — System design: 1. pressure regulator; 2. permeation tube chamber; 3. air saturator; 4. thermometer; 5. manometer; 6. homogenization/exposure chamber; 7. charcoal tube; 8. mass flow controller; 9. suction pump; 10. sampling valve; 11. gas chromatograph.

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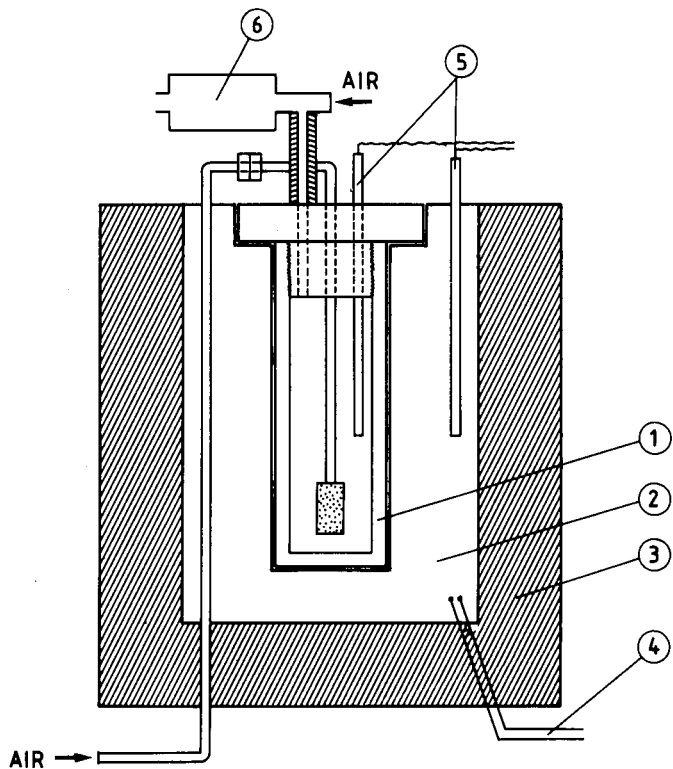


Figure 2 — Air saturator: 1. saturation chamber; 2. aluminum cylinder; 3. thermal insulation; 4. electric heater; 5. resistance thermometer; 6. mixing device.

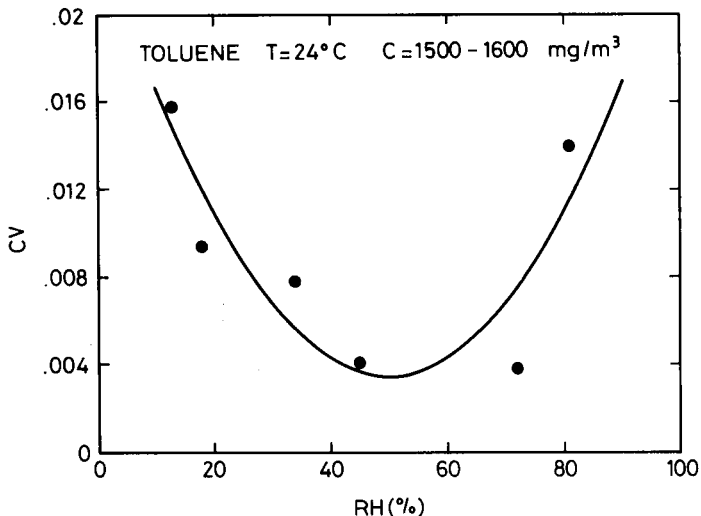


Figure 3 — Effect of the humidity control system operating in the homogenization/exposure chamber on the reproducibility of the gas mixtures generated using an air saturator: coefficient of variation vs. relative humidity. (CV refers to 15 consecutive sample concentration measurements by gas chromatography at the outlet of the homogenization chamber, with input concentration values ranging between 1500 and 1600 mg/m³).

quantity about 10% greater than the expected amount was needed in order to balance the various losses occurring during evaporation (wall effects). This procedure leads to output pollutant concentrations that initially decrease quickly and then, about 40 min after the standard mixture flow has been started, become stable.

Finally, the effects of the humidity control system, operating in the homogenization/exposure chamber, were studied with respect to the reproducibility of the generated gas mixtures. For this purpose, tests were performed maintaining constant temperature and pollutant concentration (as measured at the chamber inlet), while varying relative humidity. The results are plotted in Figure 3, each point referring to 15 consecutive output concentration measurements carried out by gas chromatography. As can be seen, the variation coefficient, as affected by the analytical procedure and by the performance of both the air saturator and the homogenization chamber, ranges between 0.004 and 0.016.

Conclusions

The controlled atmosphere system that the authors have developed is capable of giving good results in the generation of dynamic standard gas mixtures. Its performance currently is being improved further through additional modifications.

The differences between the calculated and the measured concentration values are mainly due to the difficulties in controlling temperatures and flows. In the case of sample generation with permeation tubes, differences between calculated and measured values principally result from inaccuracy inherent in the determination of the emission parameters.

Good reproducibility of the gas mixtures at the outlet of the homogenization chamber was achieved either by disconnecting the humidity control system or by maintaining the relative humidity within the intermediate regulation range. In the extreme ranges, however, the humidity control system still represents a source of disturbance though not to the point of making the generated standards unacceptable.

References

1. Schirmer, R.E., T.R. Pahl and D.W. Phelps: Application of Internal Standards in Routine Vapor Measurements by Gas Chromatography. *Am. Ind. Hyg. Assoc. J.* 45:95-98 (1984).
2. Dixon, S.W., J.F. Vasta, L.T. Freeland, D.J. Calvo and R.E. Hemingway: A Multiconcentration Controlled Test Atmosphere System for Calibration Studies. *Am. Ind. Hyg. Assoc. J.* 45:99-104 (1984).
3. Borders, R.A., R.G. Melcher and S.J. Gluck: Improved Methodology for Field Validation of Industrial Hygiene Monitoring Methods. *Am. Ind. Hyg. Assoc. J.* 45:299-305 (1984).
4. O'Keefe, A.E. and G.C. Ortman: Primary Standards for Trace Gas Analysis. *Anal. Chem.* 38:760-763 (1966).
5. Scaringelli, F.P., A.E. O'Keefe, E. Rosenberg and J.P. Bell: Preparation of Known Concentrations of Gases and Vapors with Permeation Devices Calibrated Gravimetrically. *Anal. Chem.* 42:871-876 (1970).
6. Lucero, D.P.: Performance Characteristics of Permeation Tubes. *Anal. Chem.* 43:1744-1749 (1971).
7. Williams, D.L.: Permeation Tube Equilibration Times and Long-Term Stability. In *Calibration in Air Monitoring* (ASTM-STP 598). Philadelphia: American Society for Testing and Materials, 1976. pp. 183-197.
8. Vanwrevelen, D.W.: *Properties of Polymers*. Amsterdam: Elsevier Scientific Publishing Company, 1976. pp. 403-425.
9. Crank, J.: *The Mathematics of Diffusion*. London: Oxford University Press, 1956. pp. 62-83.

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