Evaluation of evacuated glass tubes for sampling of SF6/ air mixture for air exchange measurement

Tamura, G. T.; Evans, R. G.

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EVALUATION OF EVACUATED GLASS TUBES FOR SAMPLING OF SF_6/AIR MIXTURE FOR AIR EXCHANGE MEASUREMENT

by G.T. Tamura and R.G. Evans

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ABSTRACT

Grab sampling of tracer gas/air mixture is a convenient method for conducting a survey of air infiltration rates in homes. Gas samples may be collected with a hypodermic needle and stored in Vacutainers. This technique is particularly suited when sulphur hexafluoride (SF_6) is used as a tracer gas, as only a small quantity of gas sample is required to measure its concentration with an ion capture detector/chromatograph.

Because an extended delay is probable between collection and analysis of gas samples, storage stability tests of SF_6/air mixture in 20 mL Vacutainers were conducted. Samples can be stored for at least 21 days without significant changes in their concentrations.

The gas sampling and analysis procedures are described in detail.

RÉSUMÉ

Le prélèvement au hasard d'échantillons de mélange gaz traçant/air est une méthode pratique pour étudier les taux d'infiltration de l'air dans les habitations. Les échantillons de gaz peuvent être prélevés avec une seringue hypodermique et conservés dans des t
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été effectués.
au moins 21 jour
de concentration

Les méthodes de
gaz sont décrite

Evaluation Of Evacuated Glass Tubes For Sampling Of SF_6/Air Mixture For Air Exchange Measurement

G.T. TAMURA
Member ASHRAE

R.G. EVANS

HEAT loss caused by air that leaks in and out through cracks in the house envelope can be a significant component of the total heat loss. Extensive measurements of air exchange rate, therefore, are being conducted to assess and develop means of reducing this heat loss and to study the influence of air exchange rate and house air tightness on indoor air quality.

Air exchange rate in houses is usually measured using the tracer gas technique. It involves releasing a small quantity of tracer gas inside the house to mix with the room air, and measuring its rate of decay which is related to the rate of air infiltration by:

$$I = \frac{1n(C_i/C_o)}{t_o - t_i} \quad (1)$$

where:
I = air exchange rate, air change per unit time
C_i = concentration of tracer gas at time, t_i
C_o = concentration of tracer gas at time, t_o

At least two readings of tracer gas concentration are required during the decay period, but usually several readings are taken. The air infiltration rate is determined from the slope of the 1n C versus time curve using the least square method.

G.T. Tamura and R.G. Evans are with the Energy and Services Section, Division of Building Research, National Research Council Canada in Ottawa.

The tracer gas concentration is measured with an instrument either in the house or in the laboratory. In the latter case, gas samples are collected at the site and transported to the laboratory for analysis. This method is suited for a survey of houses because gas samples can be collected following a simple procedure, also, the gas analysis instrument and its associated equipment need not be transported and set up for operation at the test site.

One method of collecting gas samples investigated by Harrje, Gadsby and Linteris[1] is the use of a 500 mL flexible polyethylene bottle with a 6 mm hole through the cap, covered with a 2 mm thick natural rubber gasket. The gasket seals the bottle and acts as a septum into which a hypodermic syringe can be inserted to draw a collected gas sample for analysis. A sample is collected by removing the gasket and hand squeezing the bottle ten times to ensure that the air in the bottle represents the sampled air.

Another method of collecting gas samples used by Grot[2] is the use of a 10L, five layer air sample bag and a hand pump or a small battery-powered pump. The hand pump requires about 50 strokes to fill the bag, and the battery-powered pump, about five minutes of operation.

For collecting mine atmospheric samples, Freedman et al.[3,4] investigated the use of 10 and 30 mL septum-stoppered evacuated glass tubes ordinarily used for routine blood sampling in hospitals. They are light and compact and

Table 1 Storage Stability Test Of SF_6 In Evacuated Glass Tubes
Test No.1

Time (min)	SF_6 Direct Sampling, ppb	SF_6 Concentration in Tubes, ppb Storage Period, Days						Mean	Standard Deviation
		0	1	3	5	7	14		
0	16.6	16.1	16.5	16.5	16.3	16.6	16.5	16.3	0.15
0		16.1	16.3	16.3	16.2	16.3	16.4		
15	14.8	14.3	14.5	14.4	14.4	14.5	14.6	14.4	0.10
15		14.3	14.4	14.4	14.3	14.4	14.4		
30	13.0	12.7	12.8	12.8	12.7	12.8	12.8	12.7	0.10
30		12.5	12.7	12.7	—	12.7	12.8		
45	11.5	11.1	11.4	11.3	11.3	11.4	11.4	11.3	0.11
45		11.0	11.2	11.3	11.2	11.3	11.3		
60	10.1	9.8	9.9	10.0	9.9	10.0	10.0	9.9	0.08
60		9.8	9.8	9.9	—	9.9	9.9		
Calculated Air Change/Hr Using Decay Data Above									
	0.50	0.50	0.50	0.51	0.50	0.50	0.49	0.50	0.006
		0.50	0.51	0.50	0.49	0.50	0.50		

Grab sampling of a tracer gas/air mixture in conjunction with the tracer gas decay technique is a convenient method for conducting a survey of air infiltration rates in homes. Such a method, using sulphur hexafluoride (SF_6) as the tracer gas and storing the concentration in evacuated glass tubes, is examined.

reduce the weight and bulk of equipment that is carried underground. The storage stability of the air/gas mixtures of CO_2, CH_4, O_2, N_2, and hydrocarbons during storage in the evacuated glass tubes was reported.

The evacuated glass tubes are suited for taking grab samples when sulphur hexafluoride (SF_6) is used as a tracer gas, because only a small quantity of air/gas mixture is required to measure its concentration with an ion capture detector/chromatograph. Because the glass tube has been evacuated, a gas sample can be collected in less than a minute. The evacuated glass tubes are light and compact, can be transported easily and cheaply, and because of low cost, can be discarded after use.

A series of tests was conducted to develop and verify a procedure for grab sampling with evacuated glass tubes as applied to air exchange measurement, and also to determine the storage stability of the gas samples. Additional tests were conducted to assess the effect of possible exposure of the stored gas samples to extreme temperature during transport and the hazard caused by accidental glass tube breakages.

Test procedure

The sampling tubes used for the tests were 20 mL evacuated glass tubes, silicone coated and stopped by 6.35 mm thick butyl rubber septa. Ten randomly selected tubes tested for vacuum gave an average pressure of 0.036 atmosphere (3.65 kPa). The SF_6 gas samples were taken from the indoor air with a 60 mL plastic syringe and transferred to the evacuated glass tubes by inserting the needle of the syringe into the septum (Figure 1). Twice the tube volume of gas was injected by pushing the plunger of the syringe, raising the tube pressure to about one atmosphere (101.3 kPa) above ambient pressure.

The instrument used for measuring the SF_6 concentration was the ion capture detector/chromatograph, sensitive to SF_6 in the few-parts-per-billion range. A syringe needle was attached to the inlet port of the instrument to permit transfer of the sample gas under pressure in the glass tube to the sampling loop of the instrument (sampling pump was removed).

The instrument has a 2 mL sampling loop with a 6 port sampling valve, a 6.35 mm O.D. 380 mm column of 100/120 mesh aluminum oxide and an ion capture detector, both of which are continuously flushed with carrier gas of nitrogen. The detector has a radiation source of Ni 63 (10 mc), which ionizes the carrier gas to produce a standing current. When a measurement is required, the sample gas trapped in the sampling loop is directed to the column, where it is absorbed. The oxygen is eluted first, followed by SF_6. These gases capture the ions produced by the radiation source, causing the standing current to decrease in proportion to the gas concentration. The concentration of SF_6 can be determined either by the SF_6 peak or the area under the peak; in these tests the former method was used.

To collect samples of SF_6 for air exchange measurement, tests were conducted in a two-story house with the furnace fan of the central warm air heating system operating continuously. A sufficient quantity (about 20 mL) of SF_6 was injected with a syringe into the return air duct to reach a tracer gas/air concentration of 25 ppb. As a check, nitrous oxide, another commonly used tracer gas, was also injected at the same time into the return air duct. The SF_6/air mixture was sampled 30 minutes after injection (when the tracer gas concentration had started to decline), and thereafter at 15-minute intervals during the next hour.

The set-up for gas sampling is shown in Figure 3. Gas samples were pumped through two plastic tubes from the return air duct; one to the SF_6 detector/chromatograph, and the other to an N_2O non-dispersive infrared gas analyzer. Two additional sample lines were installed to permit six people, at the same time, to draw two samples from the lines by

Table 2 Storage Stability Test Of SF_6 In Evacuated Glass Tubes
Test No. 2

Time (min)	SF_6 Direct Sampling, ppb	SF_6 Concentration in Tubes, ppb Storage Period, Days						Mean	Standard Deviation
		0	1	3	5	7	21		
0	20.2	19.3	19.6	19.7	19.6	19.6	19.7	19.5	0.15
0		19.2	19.5	19.5	19.6	19.6	19.5		
15	18.8	17.9	17.9	18.0	18.0	18.1	18.1	18.0	0.09
15		17.8	17.9	17.9	18.0	17.9	18.0		
30	17.3	16.3	16.4	16.6	16.5	16.4	16.6	16.4	0.16
30		16.1	16.3	16.5	16.4	16.2	16.5		
45	15.8	15.1	15.2	15.4	15.3	15.2	15.4	15.2	0.11
45		15.1	15.1	15.3	15.3	15.2	15.3		
60	14.5	14.0	14.0	14.1	14.1	14.0	14.1	14.0	0.05
60		14.0	13.9	14.1	14.0	14.0	14.0		
Calculated Air Change/Air Using Decay Data Above									
	0.34	0.32	0.34	0.33	0.33	0.34	0.33	0.33	0.006
		0.32	0.33	0.32	0.33	0.33	0.33		

Figure 1 Transfer of gas sample from plastic syringe into evacuated glass tube.

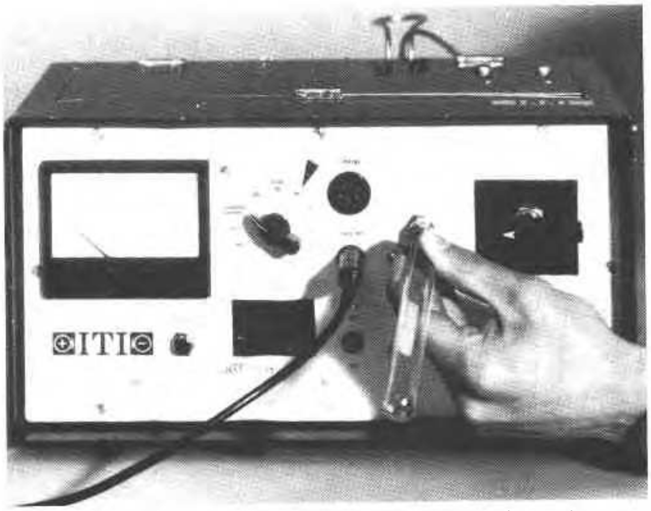

Figure 2 Transfer of gas sample from evacuated glass tube to inlet port of ion capture detector/chromatograph.

inserting the syringe into the plastic tubing and transferring its contents into the evacuated glass tubes. At each sampling time, therefore, twelve samples were collected in the evacuated glass tubes for storage stability tests. The gases in the evacuated glass tubes were analyzed after storage at room temperature for 0, 1, 3, 5, 7, 14 and 21 days. The SF_6 detector/chromatograph was calibrated with a prepared standard gas mixture prior to measuring the concentration of each set of samples. The first series of tests was conducted in the test house with air exchange induced by an exhaust fan; the second series with the house under natural air exchange caused by weather.

Details of the gas sampling and gas analysis procedures used for the tests are given in Appendix A.

The sample gas in the evacuated glass tube may be subjected to low or high temperature during transport from the test site to the laboratory. To check the stored gas samples under these conditions, which may cause leaks, and reaction of SF_6 with the surfaces of glass and rubber septum, five tubes with SF_6 of known concentration were subjected to −16°C and another five to 36°C for 24 hours. Five tubes were stored at room temperature to serve as a control set.

Because the gas samples were stored in the evacuated glass tubes with a pressure of one atmosphere (101.3 kPa) above ambient, glass breakage tests were conducted by striking the tube against a metal edge and noting the spread of glass after breakage. The evacuated glass tubes were pressurized by 0 to 1 atmosphere above ambient pressure in increments of 1/4 atmosphere (25 kPa).

Results and discussion

The results of the two series of tests (Tables 1 and 2) show that the calculated air exchange rates for the first test were 0.51 air changes per hour with direct measurement of N_2O concentration, 0.50 with direct measurement of SF_6 concentration and 0.50 with laboratory measurement of SF_6 using the evacuated glass tubes; for the second test they were 0.34, 0.34 and 0.32, respectively. The N_2O tracer gas was used as a comparative check on the air exchange rates as determined with the SF_6 tracer gas. The results validated the procedures used for taking a sample of SF_6/air mixture with the evacuated glass tubes and transferring it to the detector/chromatograph for analysis. The results also confirmed that there was so little air in the evacuated glass tubes that there was no significant effect on the reading of the SF_6 concentration in the sample.

There were no significant changes in the concentration of SF_6 samples after storage in the evacuated glass tubes for up to 21 days. The standard deviations were less than 0.1 percent of the mean concentrations and less than 2 percent of the mean air change rates. Table 3 gives the results of measurements of SF_6/air mixture stored in 15 evacuated glass tubes for 24 hours, with five subjected to an air

APPENDIX A

Procedures for Gas Sampling with Evacuated Glass Tubes and Measurement with Ion Capture Detector/Chromatograph

Gas Sample Collection

1. Check syringe for leaks.
2. Flush the syringe once by drawing the plunger from 0 to 60 mL, and then push the plunger back to 0. The plunger will have to be moved slowly to allow the air to be drawn into or forced out of the syringe.
3. To collect a gas sample, slowly pull the plunger of the syringe to the 60 mL mark. Wait until the plunger is no longer drawn back and then push the plunger to the 50 mL mark.
4. Insert the needle of the syringe into the septum of the evacuated glass tube. The plunger will be drawn by the vacuum to about the 35 mL mark; failure to do so indicates a faulty evacuated glass tube. After the plunger stops, push it slowly to the 5 mL mark and hold it there for approximately 15 seconds.

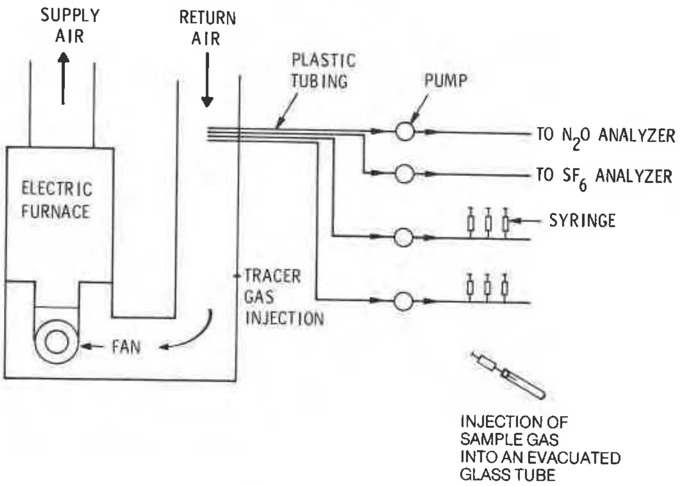

Figure 3 Test arrangement for collecting gas samples from the return air duct.

Table 3 Storage Stability Test Of SF_6 In Evacuated Glass Tubes Exposed For 24 Hours At −16°C And 36°C

	SF_6 Concentration in Tube, ppb		
	Room Temp.	−16°C	36°C
	14.2	14.1	14.2
	14.3	14.3	14.3
	14.3	14.4	14.4
	14.4	14.4	14.4
	14.3	14.3	14.3
	14.3	14.3	14.3
Mean Standard Deviation	0.09	0.12	0.08

temperature of −16°C, five to 36°C and the remaining five at room temperature. The results indicated that there were no significant changes in the SF_6 concentration when the evacuated glass tubes were exposed to these extreme conditions.

There were no breakages with normal handling of evacuated glass tubes. The breakage tests with pressures of 1/4, 1/2, 3/4, and 1 atmosphere (25, 50, 75, 101.3 kPa) above ambient, all resulted in a 2 m spread of glass shards. These tests repeated with evacuated glass tubes covered with a layer of 50 mm wide masking tape resulted in no spread of shards or sharp edges on the surface of the masking tape. This method of protection can be used where special precaution against accidental glass breakage is required.

Grab sampling with plastic syringes and evacuated glass tubes can also be applied in houses without a central warm air system. Initially, the house needs to be seeded with tracer gas from room to room, and gas samples collected during the decay period at a central location while operating floor fans to obtain a homogeneous mixture of tracer gas and air.[1,2]

Conclusions

- The sampling and measurement procedures with evacuated glass tubes for air exchange measurement with SF_6, described in Appendix A, were validated.
- Sample gases of SF_6 can be stored in the evacuated glass tubes for at least 21 days without significant changes in their concentrations.
- The results of the tests given in Tables 1, 2 and 3 indicated conclusively that the evacuated glass tubes are suitable for storage of SF_6 in concentrations normally encountered in air exchange measurements.

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5. Remove the needle from the septum.
   With this procedure the sample of gas in the evacuated glass tube is under pressure of about one atmosphere (101.3 kPa) above ambient pressure.

Gas Sample Measurement

1. Ensure that the sampling valve of the detector/chromatograph is in the off position.
2. Push the septum of the evacuated glass tube against the sampling valve needle of the detector/chromatograph to allow the sample gas to enter the sampling loop.
3. Wait 25 seconds for the sample to flush through the sample loop.
4. Remove the evacuated glass tube and immediately turn the sampling valve to the on position, hold it for 6 to 8 seconds, and then turn it back to the off position.
   This step allows the carrier gas to transfer the sample gas in the sample loop to the column and the detector.