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by [Kalliokoski P](#), [Niemelä R](#), [Salmivaara J](#)

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The tracer gas technique — A useful tool for industrial hygiene

by Pentti Kalliokoski, PhD,¹ Raimo Niemelä, MSc,² Juha Salmivaara, MSc (Eng)³

KALLIOKOSKI P, NIEMELÄ R, SALMIVAARA J. The tracer gas technique — A useful tool for industrial hygiene. *Scand j work environ health* 6 (1980) 123-130. The tracer gas technique was applied in industrial hygiene surveys. Carbon dioxide, nitrous oxide, and sulfur hexafluoride were used to elicit ventilation rates and determine spreading routes of dilution air in the work environment. The continuous monitoring of nitrous oxide with an infrared spectrophotometer was found to be the most practicable choice for ventilation testing, whereas plastic bag sampling of nitrous oxide or sulfur hexafluoride with subsequent analysis by gas chromatography appeared to be a good method for emission spreading studies. The local flow rates of dilution air and the percentages of make-up air present were the most important information gained in the ventilation studies.

Key terms: tracers, ventilation testing.

The tracer technique has been used in ventilation and contaminant spreading investigations in buildings. The vast majority of the studies have been conducted in dwellings [comprehensive reviews have been made, eg, by Hitchin & Wilson (8) and Gilath (7)] and in hospitals (2, 16, 17, 22). Industrial hygiene applications have been rare (12, 13).

Tracers can specifically be utilized to determine local effective ventilation rates with the rate of decay method (8, 13, 23) and volumetric flow rates of dilution air with the equilibrium concentration method (8, 13), to simulate the spreading of contaminants, as well as make-up air, with the determination of the transfer index

(8, 16, 17), to evaluate the significance of various in-plant pollution sources with the determination of the emission index (11), to conduct air flow measurements in ducts (20), and to study the reentry of pollutants (6, 12).

Desirable properties of a tracer include the following (8): density and diffusion properties that are similar to those of air, nonexplosiveness, unlikelihood to condense, unreactive with constituents of the air or test area, odorless, nontoxic, detectable quantitatively at low concentrations, and not produced in the test area. Many different radioactive and chemical tracers have been used in the past. The most widely used radioactive tracers are Ar⁴¹, Kr⁸⁵, and Xe¹³³ (7). Even though measurements can be performed relatively safely with these radioactive tracers, developments in analytical chemistry, which allow measurements even below the parts-per-million range, make chemical tracers the more practicable alternative. Visible tracers, such as fluorescent dyes, have been replaced with gaseous tracers. Not all of them fulfill the aforementioned requirements however. Thus, acetone (14),

¹ Department of Environmental Hygiene, University of Kuopio, Kuopio, Finland.

² Department of Industrial Hygiene and Toxicology, Institute of Occupational Health, Helsinki, Finland.

³ Laboratory of Heating and Ventilating, Technical Research Center of Finland, Espoo, Finland.

Reprint requests to: Dr Pentti Kalliokoski, Department of Environmental Hygiene, University of Kuopio, PO Box 138, SF-70101 Kuopio 10, Finland.

ammonia (18), carbon monoxide (19), helium (1, 3, 10), hydrogen (4), and water vapor (9) are obviously not good tracers.

Carbon dioxide (CO_2) and nitrous oxide (N_2O) are satisfactory for studies inside buildings. Both are relatively stable and safe compounds. Nitrous oxide can also be measured in quite low concentrations (less than 1 ppm) with either an infrared analyzer or a gas chromatograph. The possible use of carbon dioxide is somewhat more limited due to its natural and varying background concentration.

Sulfur hexafluoride (SF_6) is an almost ideal tracer. It can be measured in extremely small concentrations with an electron capture gas chromatograph. The detection limit is about 10^{-7} ppm without preconcentration (5). In addition it is not a normal background constituent of air and is odorless and nontoxic. Rats have been exposed for 24 h to an atmosphere containing 80 % sulfur hexafluoride without any ill effects (16).

We have gained much experience in applying gaseous tracers in industrial hygiene surveys. We have applied the tracer gas technique when the information obtainable merely with conventional duct and opening measurements would have been insufficient because of a considerable recirculation rate, open doors and windows, or an intensive centralization of ventilation to some part of the room. The purpose of this article is to describe the

suitability of carbon dioxide, nitrous oxide, and sulfur hexafluoride as tracers under field conditions. Only the methodological aspects have been treated in the present communication. The underlying theories and the method of application in exposure evaluation have been discussed in more detail elsewhere (13).

Methods

Ventilation studies

The equilibrium concentration method and the rate of decay method were generally applied simultaneously. The tracer was released into the make-up duct in order to obtain effective mixing; in addition local ventilation rates can be measured reliably only when the injection of the tracer takes place at the position of the highest air exchange rate (23).

Carbon dioxide and nitrous oxide were used as tracers in three rotogravure pressrooms. Sulfur hexafluoride and nitrous oxide were compared in a fourth. The (six) sampling sites of the carbon dioxide experiments are depicted in fig 1. When other tracers were used, the sampling site was located in front of the press.

Tracer gases were released directly from compressed gas cylinders (Oy AGA Ab, Kilo, Finland). Two gas cylinders were needed simultaneously for carbon dioxide in order to raise the airborne concentration in the workroom air clearly above the background level (300–600 ppm). A heating device (AGA UF-1165) was placed between the carbon dioxide cylinder and the pressure valve in order to prevent the freezing of the slight amount of moisture present in the gas. The release rate was measured with a rotameter (AGA TMO-950/sp, Oy AGA Ab; Flowmaster Fisher & Porter; Sho-Rate 150 Purzometer, Brooks; the probable total measuring error ± 10 %). The liberation of the tracer was continued until there was no increase in the airborne tracer level during a 10-min period. The average concentration during that period was taken as the equilibrium concentration, and the gas release was

Fig 1. Location of sampling points in three rotogravure pressrooms.

stopped. The tracer concentration was monitored until the detection limit or background level was achieved. The decay graph was plotted on semilogarithmic paper, and the effective ventilation rate determined by the rate of decay method.

The carbon dioxide concentration in the air was determined with a six-channel nondispersive two-beam infrared spectrophotometer (Uras 2, Hartmann & Braun, Frankfurt am Main, Federal Republic of Germany). The device was calibrated with pure nitrogen and with a 500-ppm standard gas (AGA) twice a day. Infrared spectrophotometers are sensitive to temperature changes, and, although the apparatus was provided with a thermostat, marked variation (about $\pm 5^{\circ}\text{C}$) was observed in temperature. According to the manufacturer of the Uras 2, a deviation of $+1^{\circ}\text{C}$ from 20°C will cause an error of 0.3 %.

The airborne concentration of nitrous oxide was continuously monitored with a single-channel infrared spectrophotometer (Miran IA General Purpose Gas Analyzer, Wilks Scientific Co., S. Norwalk, CT). The original pump of the analyzer was replaced with a more efficient one (Millipore Miniature; flow rate 20 l/min), and a recorder was provided. The analyzing conditions were as follows: wavelength $4.45\ \mu\text{m}$, path length 20.25 m, and slit width 1.0 mm. Since water vapor and carbon dioxide interfere with the analysis, a self-made filter unit (calcium chloride, Na-asbestos, and calcium sulfate) was installed between the pump and the analyzer.

A detection limit of 0.02 ppm for nitrous oxide was given by the manufacturer of the analyzer. Such sensitivity was not achieved under field conditions. Voltage variation in the electric current, temperature changes due to air movements, and vibration of machines were found to influence the readings. After casual strong disturbance peaks were excluded, the variation in the readings was, however, less than 5 % of the whole scale. Base-line variation made measurements below the concentration of 1 ppm rather inaccurate. This inaccuracy also appeared as nonexponentiality in the lowest part of the decay graph. The phenomenon was also partly due to the reentry of the tracer gas from

adjoining rooms and the outdoors. The zero point was reset between each measurement. Otherwise, the calibration was found to remain unchanged for long periods. The calibration was, however, checked monthly with standards prepared in laminated plastic bags (the coefficient of variation 1.5 %).

Gas chromatography was employed to analyze sulfur hexafluoride. One-minute samples were collected into laminated plastic bags with Sipin pumps (Sipin Personal Sampler Pumps Model SP-1P, Anatole J Sipin Co, New York, NY). The concentrations of the samples were analyzed with a Hewlett-Packard 5750G (Hewlett-Packard, Avondale, PA) laboratory gas chromatograph with an electron capture detector (Ni-63). A glass column (length 2 m, internal diameter 3 mm) was used. The column was packed with a 13 X Molecular Sieve (Mesh 30/60). The carrier gas was argon/methane (5 % methane: 30 ml/min). The operating temperatures were as follows: oven 190°C , injector 150°C , and detector 230°C . The detection limit was $3 \cdot 10^{-5}$ ppm.

Air flow measurements were carried out with a standard Pitot tube and micromanometer (Mark 5, Airflow Developments Ltd, High Wycombe, United Kingdom) and a rotating vane anemometer (Flügelradanemometer 1443, With Lambrecht KG, Göttingen, Federal Republic of Germany) according to the Finnish standard method (22). The directions of the air currents in the rooms were observed in smoke tube experiments. The air velocities of these currents were measured with a Fues thermomanometer (Wolters & Möhring, Federal Republic of Germany).

Make-up air distribution studies

The distribution of the make-up air was investigated by transfer index determinations in two pressrooms. Carbon dioxide was used as the tracer. The experimental arrangement was otherwise similar to that of the former part of the study, but the gas was released over a period of 20–30 min. The concentration monitoring was continued until the background level was achieved.

Table 1. Results of ventilation duct and opening measurements in rotogravure pressrooms.

Room	Volume (m ³)	Make-up air supply				Exhaust rate				Ventilation rate (h ⁻¹)
		Towards the press		Total		Dryers		Total		
		m ³ /s	m ³ /h	m ³ /s	m ³ /h	m ³ /s	m ³ /h	m ³ /s	m ³ /h	
A	1,800	5.00	18,000	5.00	18,000	1.51	16,000	4.45	16,000	10.0
B	3,500	10.40	37,500	10.40	37,500	4.70	17,000	10.80	39,000	11.1
C	4,600	3.75	13,500	12.35	44,500	1.95	7,000	11.10	40,000	9.7
D	2,800	3.85	14,000	7.60	27,500	5.00	18,000	13.00	47,000	16.7

Table 2. Volumetric flow rates of dilution air in the rotogravure pressrooms by the equilibrium concentration method with carbon dioxide (CO₂), released at a rate of 65 l/min in rooms A and C and 70 l/min in room B. (The measuring sites refer to fig 1)

Room	Increase in CO ₂ concentration (ppm)					Volumetric flow rate (m ³ /s)						Coefficient of variation (%)					
	Site 1	Site 2	Site 3	Site 4	Site 5	Site 1	Site 2	Site 3	Site 4	Site 5	Mean (+SD)	Site 1	Site 2	Site 3	Site 4	Site 5	Mean (+SD)
A	323	308	163	220	203	3.61	3.75	6.83	5.17	5.72	5.02 (1.36)	12.4	19.6	13.2	19.3	26.1	18.1 (5.6)
B	108	100	148	163	135	10.92	11.81	7.94	7.19	8.75	9.32 (1.97)	9.9	14.2	7.2	2.2	15.7	9.8 (5.4)
C	200	138	213	148	165	5.50	8.33	5.11	7.39	7.19	6.70 (1.35)	10.4	28.3	4.6	5.8	17.2	13.3 (9.8)

Table 3. Effective ventilation rates in the rotogravure pressrooms by the rate of decay method with carbon dioxide. (The measuring sites refer to fig 1)

Room	Effective ventilation rate (h ⁻¹)						Coefficient of variation (%)					
	Site 1	Site 2	Site 3	Site 4	Site 5	Mean (±SD)	Site 1	Site 2	Site 3	Site 4	Site 5	Mean (±SD)
A	11.9	12.3	16.0	10.4	10.6	12.2 (2.3)	21.8	21.1	16.9	34.6	20.8	23.0 (16.7)
B	12.3	9.7	12.6	13.4	8.5	11.3 (2.1)	11.4	20.6	33.3	22.4	8.2	19.2 (9.9)
C	9.5	11.7	7.7	7.2	11.4	9.5 (2.1)	22.1	12.8	27.3	20.8	9.6	21.5 (9.8)

Table 4. Pressroom measurements of the nitrous oxide (N₂O) tracer, released at a rate of 2 l/min in room A and 10 l/min in the other rooms. (Measurements were performed in front of the presses)

Press-room	Equilibrium N ₂ O concentration (ppm)	Volumetric flow rate (m ³ /s)	Effective ventilation rate	
			h ⁻¹	Coefficient of variation (%)
A	7.7	4.33	11.1	18.9 (N = 3)
B	17.7	9.41	11.5	13.9 (N = 5)
C	31.3	5.31	10.9	19.3 (N = 7)
D	27.0	6.17	11.7	17.1 (N = 6)

Results

Ventilation studies

The distribution of air was basically arranged similarly in every pressroom. Most of the make-up air was supplied towards the press, which lay parallel with the back wall, and the air polluted with toluene (ink solvent) was removed by the press dryer exhausts located behind or above the press. The ventilation data obtained with ventilation duct and opening measurements are given in table 1.

The results of the experiments made with carbon dioxide are presented in tables 2 and 3. The location of the sampling points are presented in fig 1. The mean coefficients of variation for the test sets ($N = 4$) performed at each individual measuring site were 13.7 % (volumetric flow rates) and 21.2 % (effective ventilation rates).

Table 4 provides data concerning the measurements with the nitrous oxide tracer. The agreement of these values with the corresponding results (measuring site 1) of tables 2 and 3 was within 15 % in most cases. The mean coefficient of variation for the effective ventilation rates was 17.3 %. (Volumetric flow rates were determined only once.)

Fig 2 exemplifies the difficulties met in determining the effective ventilation rate with carbon dioxide in pressrooms. Arising from the intensive ventilation, a higher equilibrium concentration than 100–300 ppm above the natural background level was impossible to achieve in spite of the high carbon dioxide release rate (up to $70 \text{ l} \cdot \text{min}^{-1}$). When the release was stopped, the carbon dioxide concentration fell to the background level in 10–15 min, and only three to six readings per channel were obtained.

The problems were much smaller when nitrous oxide was used as a tracer because of its very low natural background concentration and the continuous concentration monitoring. As shown in fig 3, the data points above a level of about 1 ppm fit the straight line well. The reasons for the nonexponentiality of the lowest part of the decay graph are the reentry of the tracer from adjoining rooms (low air ex-

change compartments) and the unstable zero level of the analyzer.

A decay graph of the test employing sulfur hexafluoride as the tracer gas is also depicted in fig 3. The mean effective ventilation rate in the experiments with sulfur hexafluoride was 13.0 h^{-1} , which is within one standard deviation of the corresponding mean with nitrous oxide. The coefficient of variation was 13.9 % ($N = 3$).

Fig 2. Determination of effective ventilation rates in the rotogravure pressrooms. Decay graphs for carbon dioxide (CO_2) (measuring site 4 in pressroom A).

Fig 3. Determination of effective ventilation rates in a rotogravure pressroom. Decay graphs for nitrous oxide (N_2O) and sulfur hexafluoride (SF_6) (pressroom C).

Table 5. Transfer indices for the spreading of make-up air from the inlet openings to various parts of two pressrooms. (Sampling sites refer to fig 1; standard deviations are given in parentheses; $N = 3$)

Room	Transfer index (ppm · min/l)					Mean
	1	2	3	4	5	
Pressroom A	3.9 (0.8)	3.1 (1.3)	2.2 (0.5)	2.9 (0.6)	2.5 (0.5)	2.9 (0.6)
Pressroom C	2.6 (0.5)	1.8 (0.1)	3.1 (0.5)	2.0 (0.8)	2.1 (0.5)	2.3 (0.5)

Table 6. Comparison of normalized equilibrium concentrations (C'_{eq}) and transfer indices (T') and percentages of tracer-labeled make-up air in the local dilution air (C_{eq}/C_0).

Measuring site	Pressroom A			Pressroom C		
	C'_{eq}	T'	$(C_{eq}/C_0) \%$	C'_{eq}	T'	$(C_{eq}/C_0) \%$
1	1.00	1.00	100	0.94	0.84	69
2	0.95	0.80	95	0.67	0.59	48
3	0.50	0.55	50	1.00	1.00	74
4	0.68	0.75	68	0.70	0.65	59
5	0.63	0.63	63	0.78	0.69	57

Make-up air distribution

The transfer indices obtained for the spreading of the make-up air labeled with tracer from the inlet openings to the sampling sites (same as in the previous part of the study) are listed in table 5. Clear differences appeared among the indices, indicating an uneven make-up air distribution in the pressrooms.

Discussion

With the exception of the disadvantage due to the natural (and possibly varying) background concentration associated with the use of carbon dioxide as a tracer, the advantages and disadvantages of the three investigated tracer gases mainly originated in the analytical methods used. Infrared spectrophotometry was employed to analyze carbon dioxide and nitrous oxide. There are both single-channel (Miran I) and multichannel infrared analyzers (eg, the Miran Multi-Point Ambient Air Analyzer with 12 channels and the Uras 2 with 6 channels) available. Information is gained faster with a multichannel appa-

ratus. In addition, they, unlike the single-channel models, are provided with thermostats. The drawbacks of multichannel monitors are the slow channel change and short integration time per sample. A complete channel change run took about 17 min with the multichannel Miran analyzer available to us. This disadvantage outweighed the benefits, and the apparatus was not used. Nitrous oxide was, therefore, analyzed only with the single-channel model (the Uras 2 has no wavelength adjustment). Even though the channel change was substantially faster with the Uras 2 (a run about 3 min), only three to six readings were obtained per channel in rooms with high ventilation rates. When the results for determining the effective ventilation rates were plotted on semi-logarithmic paper, the data points were scattered around the theoretical decay lines; therefore it was necessary to repeat each measurement series several times. When the equilibrium concentrations were measured, a long monitoring period was needed before it could be ascertained that a steady state had been reached. Thus, the continuous concentration registration and the very low background concentration make the single-channel monitoring of

nitrous oxide with the Miran I generally a more advantageous choice for tracer applications in industrial spaces than the multichannel determination of carbon dioxide with the Uras 2.

Nitrous oxide was also analyzed by gas chromatography, which was the only analytical method used for sulfur hexafluoride. All samples were collected in laminated plastic bags. Even though a direct determination is possible with a portable gas chromatograph, the direct analysis was given up after a short trial (with the AID portable gas chromatograph model 511 equipped with a tritium electron capture detector, Analytical Instrument Development Inc, Avondale, PA) because of the long stabilization period and non-linear operation. The bag sampling method is quite laborious and is, therefore, suited mainly for the determination of the transfer index where this kind of accumulative detector is advantageous because the concentration integral can be measured directly. However, if the ventilation rate is very high, the especially low detection limit of sulfur hexafluoride outweighs the difficulties.

The coefficient of variation was relatively high, on the order of 15 %, even when nitrous oxide and sulfur hexafluoride were used as tracers for the determination of effective ventilation rates. This high coefficient was partly due to fluctuations in the ventilation itself with time and partly due to the experimental errors dealt with earlier.

Knowledge of the local volumetric flow rates is by far more important for an industrial hygienist than his being aware of the effective ventilation rates, since the concentrations of noxious substances in the workroom air are determined mainly on the basis of the former. There are examples where the results of the experiments with the equilibrium concentration method have been used successfully to predict the concentration of airborne impurities (19). It should be noted, however, that when the tracer is released into the room through injection into the make-up air duct, as in the present study (being often the only practicable method in large industrial halls), the results obtained in this way equal the local total volumetric flow of dilution air only in spaces that are

single-compartment systems in respect to the make-up air supply. Even if this condition is fulfilled, but the compartment is at a negative pressure relative to adjoining spaces, the result is useful only when the air entering the compartment due to the pressure difference is pure. In industrial halls, a multicompartment system is usually formed; therefore, caution must be applied when the results obtained with the equilibrium concentration method are being interpreted.

The actual meaning of the equilibrium concentration is illustrated in table 6 with a comparison of the equilibrium concentrations with transfer indices obtained for make-up spreading. To provide a better base of comparison, we normalized both kinds of values with respect to the highest value. Both the order and mutual magnitude of the normalized equilibrium concentrations and transfer indices appear to be similar. This is hardly an unexpected result, because actually the same phenomenon, the spreading and distribution of make-up air, was studied with both methods. (The basic experimental designs were also similar.)

The concentration of the tracer in the room air cannot exceed the initial concentration in the make-up air anywhere. If the equilibrium concentrations (C_{eq}) are normalized with respect to the concentration in the make-up air (C_0) instead of the highest concentration observed in the room being used, the new normalized values show the fraction of local dilution air flow rate originating from the labeled make-up air supply. These C_{eq}/C_0 ratios are presented as percentages in table 6.

When the C_{eq}/C_0 ratios of pressroom A and C are compared, it appears that the ratios were close to unity in the press area of room A, whereas only a maximum of 74 % of the dilution air in the press area of room C originated from the make-up air supply directed towards the press. This result agrees well with the fact that all make-up air was discharged to the press area in room A. In addition the press area of room A was maintained at a positive pressure relative to the surroundings. Only 40 % of the total make-up air of room C was delivered towards the press area. In this case also the prevailing pressure gradient contributed to the air flow

from the bulk of the room towards the press.

The values obtained for the dilution air flow rate with the equilibrium concentration method correspond to the real flow rates only in the "press compartment." Thus, if the solvent emission from the press is known, the average concentration in the space behind the press, but usually not in the main part of the room, can be predicted.

The transfer indices cannot be directly interpreted in the same way as the equilibrium concentrations. They cannot be used for estimating pollutant concentrations either. The application of the simple transfer index method is, however, not only feasible, but advisable when the spreading of pollutants is being simulated.

Engineering control of workroom air impurities is an important part of industrial hygiene. When corrective measures are being planned in existing plants, rates and spreading routes of in-plant emission, as well as the distribution of the ventilation air, should be elicited. The tracer technique appears to be a valuable tool for this purpose.

In summary, it was found that the tracer gas technique is well suited also for an industrial hygienist, especially, since industrial hygiene laboratories usually already have the essential equipment required.

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