

# Static headspace gas chromatographic determination of fault gases dissolved in transformer insulating oils

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## ABSTRACT

The analysis of dissolved gases in power transformer oils is an efficient diagnostic tool for routine performance monitoring of power transformers. A static headspace gas chromatographic method has been developed to automate the analysis. The parametric study showed that initial equilibration time is about 200 min at 70°C and set the sample volume to 15 ml of oil. Distribution coefficient values were determined under these optimized headspace conditions, avoiding the need of subsequent calibration from oil standards. The precision of the method was better than 5% and detection limits for hydrogen, carbon monoxide and methane, carbon dioxide, and hydrocarbons were 7, 10, 5 and 1 ppm (v/v), respectively. The response is linear over 3 decades up to 1000 ppm. A comparison study between the headspace and the currently used ASTM D3612 method using on-line transformer oil samples showed a good agreement. The headspace method permits an increased number of analysis per day, as much as four times, compared to the ASTM method.

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## INTRODUCTION

Analysis of dissolved gases in power transformer oils is widely performed by public utilities worldwide for routine performance monitoring. The presence of faults such as arcing, local overheating and partial discharges in the equipment always results in the chemical decomposition of the insulating materials, which are composed of mineral oil and cellulose [1]. The main degradation products are gases ( $H_2$ , CO,  $CO_2$ ,  $CH_4$ ,  $C_2H_2$ ,  $C_2H_4$ ,  $C_2H_6$  and  $C_3H_8$ ) which partially or totally dissolve in the insulating oil. Other gases, such as  $O_2$  and  $N_2$  along

with  $CO_2$ , are also dissolved by contact of ambient air with a limited surface of oil inside the oil expansion chamber of conservator-type transformers. A normally operating transformer may contain as much as 10% (v/v) dissolved gas, consisting mostly of air as well as fault gases at concentration up to 100 ppm (v/v)<sup>☆</sup>. Since correlations between the nature and proportion of the gases and the types of faults have been established, it is possible to diagnose the presence of faults at an early stage and take corrective actions before they lead to unexpected failure of the unit [1,2]. Dissolved gas analysis (DGA) is thus an efficient diagnostic tool that cuts costs and improves service reliability.

Various approaches exist for DGA in transformer oils and differ mainly through the gas extrac-

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☆ All the concentrations are given in ppm (v/v).

tion technique rather than the component gas chromatographic separation step. Vacuum gas extraction, widely used in North America [3], is the basis of a method approved by the American Society for Testing and Materials (ASTM) [4]. The oil sample is introduced into a degassing flask attached to a vacuum glassware maintained at less than  $1 \cdot 10^{-3}$  Torr (1 Torr = 133.322 Pa). The extracted gases are then compressed by a mercury column into a burette and a sample is injected into a gas chromatograph. This method is tedious, requiring the operation of many valves, and has limited sensitivity when the total gas content is very low (<2%). A second method, described in International Electrotechnical Commission Publication 567 and mainly used in Europe, involves gas stripping [5]. The system consists of a carrier gas sweeping through a U-shaped glass tube containing the oil sample and connected to the chromatographic system. However, due to peak broadening resulting from a loss of the pin-point feature of the injection, the detection limits are around the same as with the preceding method. A variant of the stripping method involves a straight tube filled with stainless steel beads providing a greater surface area which accelerates the extraction process [6]. However, the detection limit for hydrogen is too high for early fault warning [7]. All these methods have a common drawback: the possibilities for automation are limited, which increases manpower costs in DGA laboratories, reduces their ability to fully optimize the use of the chromato-

graphic equipment, and limits the number of DGA that could be achieved in a regular day shift.

This paper describes an automated method for the analysis of dissolved gases in power transformer oils. The static headspace gas chromatographic method (HSP-GC) allows more samples to be analyzed with minimal supervision, and offers analytical performances comparable to those of the vacuum extraction method. A parametric study on equilibration time, temperature and sample volume will be presented. The performances of the method will be estimated using the optimized headspace parameters. The results obtained with this method and the ASTM method on in-service transformer oil samples will be compared to assess the validity and performances of the HSP-GC approach.

## EXPERIMENTAL

### Instrumentation

A schematic of the chromatographic system is presented in Fig. 1. An HP-5890 gas chromatograph equipped with a thermal conductivity detection (TCD) system, a nickel catalyst unit to convert CO and CO<sub>2</sub> into CH<sub>4</sub> and a flame ionization detection (FID) system, was used for all analyses (Hewlett-Packard, Palo Alto, CA, USA). The methanizer was maintained at 350°C and fed with pure hydrogen serving both as reactant for the catalytic reactor and fuel for the FID system. A pneumatic 6-port valve (Valco, Houston, TX, USA) with 1/16-in. (1

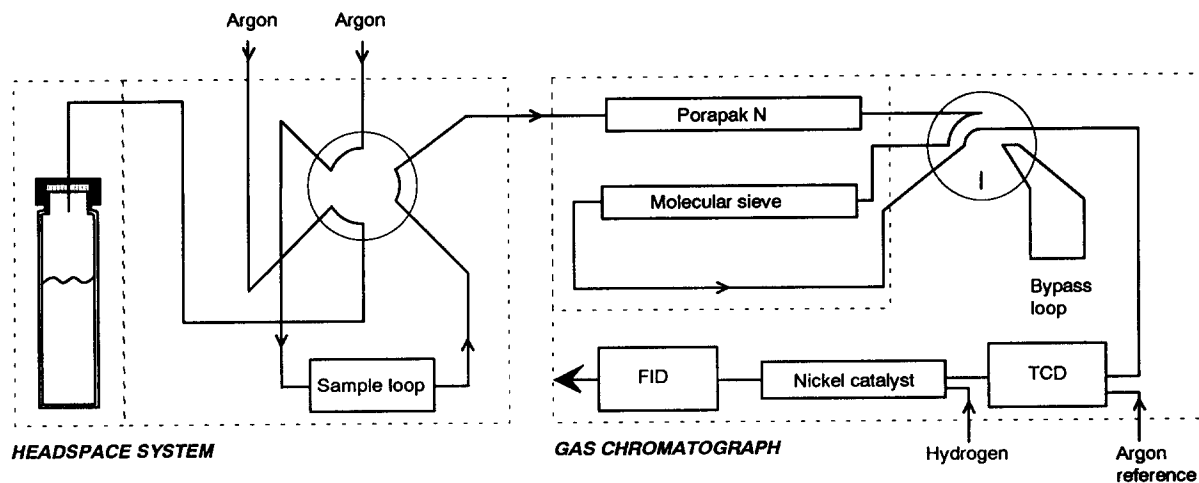


Fig. 1. Schematic diagram of HSP-GC instrumentation.

TABLE I  
INSTRUMENTAL CONDITIONS

<i>Headspace system</i>	
Auxiliary pressure	0.4 bar
Valve timing	0 s, probe needle down 3–13 s, argon pressurization 43–53 s, venting 54–64 s, injection 65 s, probe needle up
Sample loop temperature	3°C above bath temperature
<i>Gas chromatograph</i>	
Carrier gas	Ar, 40 ml/min
Catalytic gas	H <sub>2</sub> , 70 ml/min
Valve operation	0–7 min, column in series 7–18 min, molecular sieve bypassed
Oven program	40°C for 4 min 40 to 180°C at 20°C/min 180°C for 7 min
Detector temperature	TCD at 250°C FID at 350°C

in. = 2.54 cm) fittings installed on the gas chromatograph and maintained at 150°C was used for column selection. An HP molecular sieve column 13X (0.8 m × 1/8 in., 45–60 mesh) was connected at the outlet of an HP Porapak N column (4 m × 1/8 in., 80–100 mesh) through the GC valve to separate the lighter gases (H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub> and CO) that were poorly resolved on the Porapak. Once the lighter gases have been eluted, the valve is switched automatically so that the CO<sub>2</sub> and C<sub>2</sub> and C<sub>3</sub> hydrocarbons separated by the porous polymer column bypass the molecular sieve to end up in the detectors. TCD and FID signals were recorded simultaneously using the HP-3365 Chemstation software. The valve was activated by an HP-19405A event controller. The headspace sampling unit was an HP-19395A equipped with a 1-ml injection loop. This unit consists of a rotating temperature-controlled sample carousel able to load up to 24 vials and a control unit for programming the time and temperature of the sampling. The 20-ml glass vials purchased from Wheaton (Millville, NJ, USA) were hermetically sealed by PTFE-lined septa. The 10-, 30- and 100-ml glass syringes used for oil manipulations were from Perfectum (Thomas Scientific, Swedesboro, NJ, USA). Table I gives the instrumental conditions.

### Chemicals and standards

The absolute response of the detectors was calibrated with standard gas mixtures of H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>6</sub> and C<sub>3</sub>H<sub>8</sub> at concentration levels of 100, 1000, and 5000 ppm (v/v) in argon (Scott Specialty Gases, Plumsteadville, PA, USA). Dissolved-gas oil standards were prepared using Voltesso 35 (Esso Imperial Oil Company, Sarnia, Canada) transformer insulating oil and gases at least 99.9% pure (Matheson Gas Products, Secaucus, NJ, USA). Argon used as carrier gas and hydrogen were 99.999% pure (Union Carbide, Toronto, Canada).

### Procedure

The preparation of the dissolved-gas oil standard used for calibration was done according to Duval and Giguère [8]. Sub-standards were obtained by diluting the dissolved-gas oil standard with degassed oil in the headspace vials. The dilution factor was determined by weighting. Preparation of headspace vials for analysis was done as follows: after being sealed, the vials were purged with argon; oil sample was then introduced into the vial, releasing pressure buildup through a 0.5 mm O.D. needle. The vials were weighed before and after filling to control the volume injected. A 30-ml syringe with a 1.2 mm O.D. stainless-steel needle was used for this transfer.

## RESULTS AND DISCUSSION

### Parametric study

*Equilibration time.* Fig. 2 shows typical variations in the gas phase concentration as a function of time at 50°C for ethane and ethylene. For each curve, a set of vials filled with 5 ml of dissolved-gas standard containing 354 ppm of both ethane and ethylene was loaded into the thermostated bath at time zero and analyzed sequentially. The time elapsed between two data point measurements corresponds to the time required to perform a chromatographic run. The signals measured at time zero are the result of the partial phase equilibration that occurs during the few min elapsed between the end of the filling and the gas-phase injection of the first vial. In order to obtain consistent results from one vial to another, the sample must be thermostated for a minimum amount of time to bring the oil and gas phases into

equilibrium. The time required to reach such an equilibrium is about 500 min at 50°C. Similar curves were obtained with all the other fault gases studied and gave approximately the same equilibration time, although the volatility and solubility levels were quite different. This suggests that the equilibration time is governed by the matrix viscosity rather than the nature of the gases. The gas concentrations in the phases at equilibrium depend on the solubility of the analyte in oil at a given temperature. As can be seen in Fig. 2, for an oil standard of equimolar concentration of gases, ethane has a higher solubility which gives a lower maximum headspace concentration at 50°C.

**Temperature.** The same set of analyses was repeated for temperatures at 35, 60, 70, 80 and 90°C. As shown in Fig. 3, for ethylene a similar trend is observed for the variation of gas phase concentrations as a function of time. However, the first segment of the curves is steeper with increased temperature, which can be explained by an increase of the analyte liquid–gas exchange rates associated with the decrease in matrix viscosity. Fig. 4 shows the effect of the temperature on the equilibration time averaged over the ten gases studied; the time required to reach 90% of the asymptotic value is used for that comparison. Based on these results, it seems obvious that in order to minimize thermostating time, the highest temperature allowed by the matrix will be required. However, the solubility changes with temperature are different from one gas to another: H<sub>2</sub>, N<sub>2</sub>, O<sub>2</sub> and CO have a lower solubility

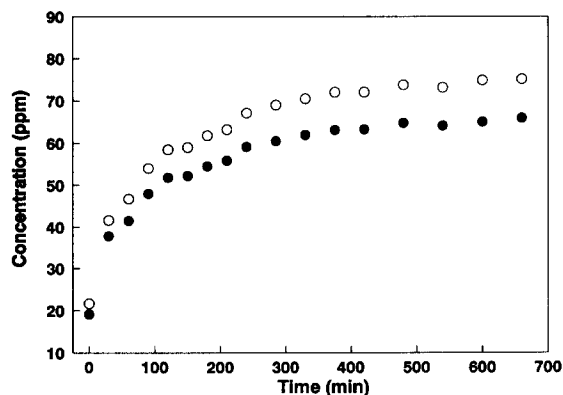


Fig. 2. Gas phase concentration in headspace as time allowed to equilibrate (50°C). ○ = C<sub>2</sub>H<sub>4</sub>; ● = C<sub>2</sub>H<sub>6</sub>.

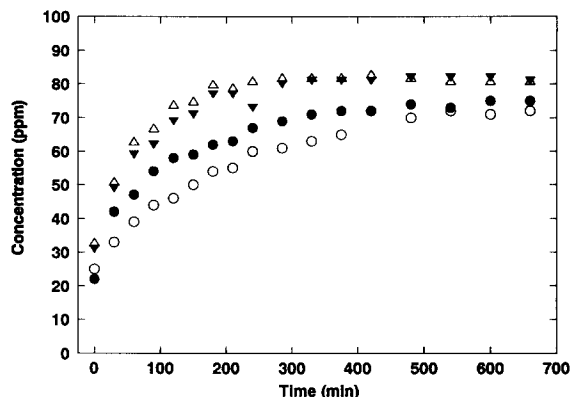


Fig. 3. Influence of the temperature on the gas phase concentration and equilibration time for ethylene. ○ = 35°C; ● = 50°C; ▼ = 70°C; △ = 80°C.

when the temperature is raised, whereas CH<sub>4</sub>, CO<sub>2</sub> and C<sub>2</sub> and C<sub>3</sub> hydrocarbons have higher solubility [9]. For O<sub>2</sub> and N<sub>2</sub> present in relatively high concentrations, the temperature is not a limitation since their determination can tolerate a loss in analytical sensitivity. Also, carbon monoxide is not specific enough to be solely associated to cellulose degradation. It can also appear as a result of long term oxidation of the oil [10]. Therefore, in DGA, CO is not a key compound in determining a transformer's internal state from correlation rules. On the other hand, hydrogen is important for fault diagnosis and a low detection limit is highly desirable. This would normally be achieved by lowering the temperature so that hydrogen is less soluble in oil. A compro-

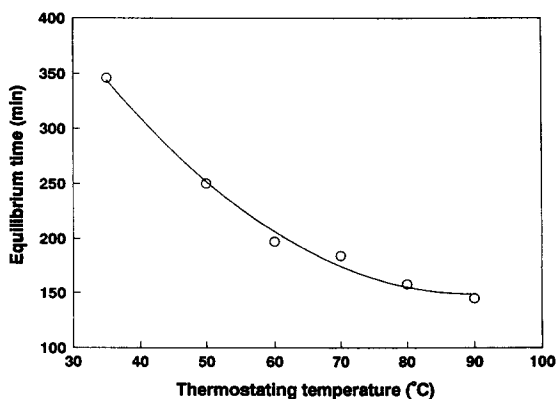


Fig. 4. Temperature effect on equilibration time considering 90% maximum value (5 ml of oil).

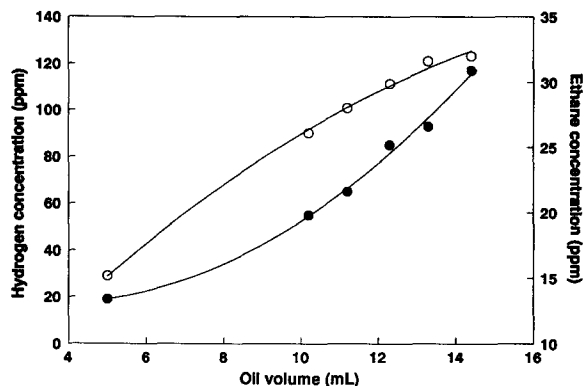


Fig. 5. Effect of volume  $V_L$  on equilibrium gas concentration in volume  $V_G$ . ● =  $H_2$ ; ○ =  $C_2H_6$ .

mise between sensitivity and equilibration time is achieved by setting the temperature at  $70^\circ C$ . At this temperature, partial vaporization of certain oil components which could produce subsequent chromatographic ghost peaks will be minimized.

**Sample volume.** The sensitivity of the headspace method is also strongly affected by the ratio  $V_G/V_L$  as indicated by the relation:

$$C_G = \frac{C_L^0}{K + \left(\frac{V_G}{V_L}\right)} \quad (1)$$

where  $C_G$  is the concentration of the analyte in the gas phase at equilibrium,  $C_L^0$  the initial concentration of the analyte in the liquid phase,  $K$  the distribution coefficient of the analyte between the two phases in equilibrium, and  $V_G$ ,  $V_L$  the volumes of the gas and liquid phases in the headspace vials, respectively [11]. Since the total volume is fixed ( $V_{\text{total}} = V_G + V_L$ ), only the liquid volume  $V_L$  can be varied and optimized. Fig. 5 shows the variation of the gas phase concentration  $C_G$  with the volume of oil for  $H_2$  and  $C_2H_6$ . For gases with a  $K$  lower than unity,  $C_G$  increases with a positive curvature, whereas gases with a  $K$  greater than unity have a negative curvature. The greater the difference between the  $K$  value and unity, the more pronounced the curvature. This observation agrees with eqn. 1 and suggests that  $V_L$  should be set to the highest volume allowed by the headspace system. However, the expression obtained after differentiation of eqn.

1 indicates that the error in the determination of  $C_L^0$  increases as  $V_G/V_L$  decreases:

$$\frac{\Delta C_L^0}{C_L^0} = \frac{\Delta C_G}{C_G} + \frac{\Delta K}{K + \frac{V_G}{V_L}} \quad (2)$$

Under our conditions,  $\Delta K/K$  usually exceeds  $\Delta C_G/C_G$ , so that  $V_L$  should be increased while maintaining precision within acceptable values. Table II gives the relative standard deviations obtained from ten replicates of the same standard for each of the three sample volumes studied. The variation in the sample volume from one vial to another (0.1 ml for a 15-ml injection) had a negligible contribution to these values. As expected from eqn. 2, the precision is generally improved by lowering  $V_L$  so that a good compromise is obtained by setting the sample volume at 15 ml. The precision for  $H_2$  is less than expected at  $V_L = 10$  ml; in this case, the analytical conditions are close to the detection limit and the contribution of  $\Delta C_G/C_G$  becomes more important.

The final set of conditions retained for the headspace unit is  $70^\circ C$  and 15 ml sample volume with an equilibration time of 200 min.

#### Analytical performances

**Calibration.** The calibration curves were established over a concentration range of 5 to 400 ppm generally encountered in transformer oils, and their linearity verified to a concentration of up to 1000 ppm. An example of the chromatograms is shown

TABLE II  
INFLUENCE OF  $V_L$  ON ANALYTICAL PRECISION

Gas	Precision (R.S.D.) (%) <sup>a</sup>		
	$V_L = 10$ ml	$V_L = 15$ ml	$V_L = 18$ ml
$H_2$	4	3	4
CO	2	2	4
$CH_4$	1	2	4
$CO_2$	14	14	10
$C_2H_4$	1	2	3
$C_2H_6$	1	3	3
$C_2H_2$	5	4	7

<sup>a</sup> Measured with a standard of 70 ppm  $H_2$  and  $C_2$  hydrocarbons, 141 ppm CO and  $CH_4$ , and 220 ppm  $CO_2$ .

in Fig. 6. Table III lists the calibration curve parameters of the first-order regressions used. The detection limits for a signal-to-background noise ratio of 3 are listed in Table IV together with the ASTM D3612 values determined under identical chromatographic conditions. The detection limits using the HSP-GC technique are slightly higher than those using the ASTM method but still sufficiently low to allow the use of the method for the diagnosis on in-service transformers. As expected, both techniques have better detection limits for the FID-de-

tected hydrocarbons even though these analytes are far more soluble than permanent gases.

Generally it is not necessary to use dissolved-gas oil standards to calibrate the chromatographic response provided that the analyte distribution coefficients ( $K$  values) are known and a linear relation exists between  $C_L^0$  and  $C_G$  in the range of concentrations of interest. As expressed by eqn. 1, if  $K$  and the ratio  $V_G/V_L$  are known, then  $C_L^0$  can be calculated from the chromatographic determination of  $C_G$ . Subsequently, the only response necessary for cali-

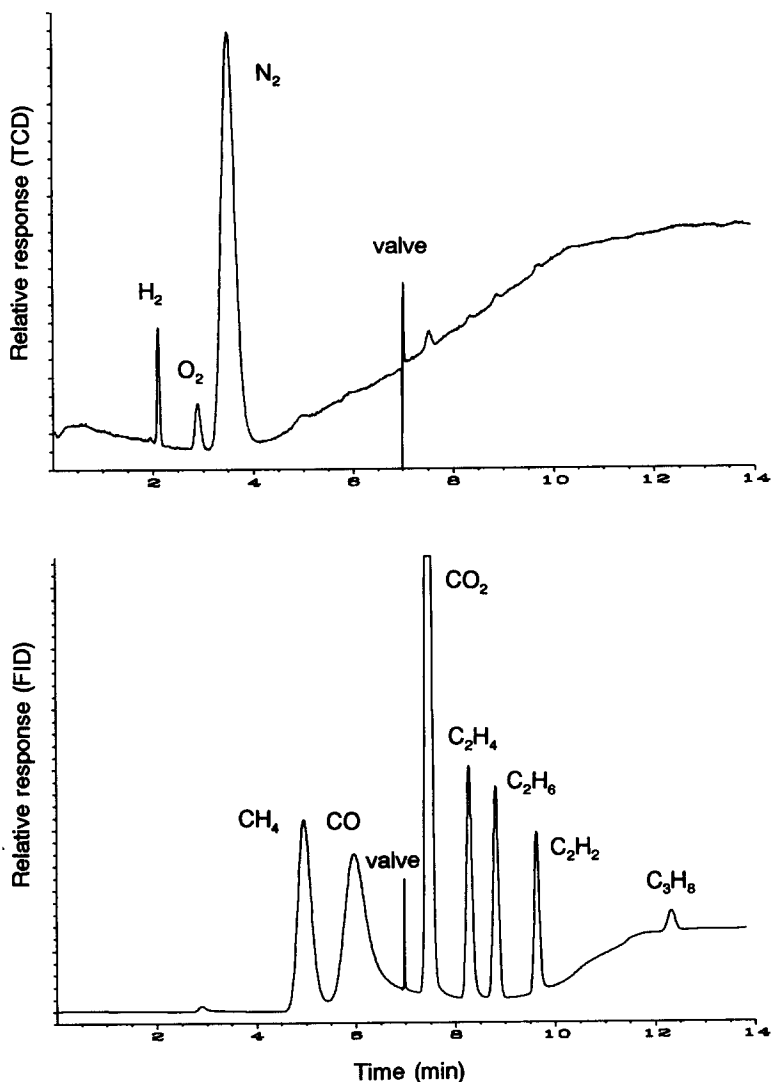


Fig. 6. Typical chromatograms of the headspace of a dissolved-gas-in-oil sample.

TABLE III  
CALIBRATION CURVE PARAMETERS

Gas	Regression coefficients <sup>a</sup>		Correlation coefficient	Number of datapoints
	b	m		
H <sub>2</sub>	10.7	1.7693	0.9991	9
CH <sub>4</sub>	1.0	1.1450	0.9940	10
CO	42.7	1.4418	0.9691	10
CO <sub>2</sub>	0.0	0.7219	0.9856	10
C <sub>2</sub> H <sub>4</sub>	0.0	0.5315	0.9986	10
C <sub>2</sub> H <sub>6</sub>	1.1	0.4370	0.9992	10
C <sub>2</sub> H <sub>2</sub>	-4.9	0.6659	0.9981	10
C <sub>3</sub> H <sub>8</sub>	0.0	0.1799	0.9988	10

$$^a y = mx + b.$$

bration would be  $C_G$  through direct introduction of gas mixtures in headspace vials. The experimental distribution coefficients  $K$  shown in Table V were obtained from the determination of an equilibrium gas phase concentration of a dissolved-gas oil standard. These values evaluated for Voltesso 35 were used for the calculations of  $C_L^0$  under the final headspace conditions. Negligible changes are expected when using a different type of oil, especially as modern transformer oils do not vary widely in chemical composition. However, if an oil significantly different in composition is used (aromatics, aliphatics, synthetics, etc.) or if high precision analysis is required, recalculating  $K$  values with an oil standard might be advisable.

**Precision and accuracy.** Table VI gives the accuracy and precision of the HSP-GC and ASTM D3612 techniques for the determination of fault gases. These parameters were estimated by analyz-

TABLE IV  
DETECTION LIMITS OF THE HEADSPACE AND ASTM EXTRACTION METHODS (ppm)

Signal-to-background noise ratio = 3

Gas	Headspace <sup>a</sup>	ASTM D3612
H <sub>2</sub>	7	1
CO, CH <sub>4</sub>	10	5
CO <sub>2</sub>	5	2
Hydrocarbons	1	0.5

<sup>a</sup> 70°C,  $V_L = 15$  ml of oil.

ing an oil sample of known gas composition prepared by the technique of Duval and Giguère [8]. A slight improvement of the precision is noted with the headspace. The difference in R.S.D. between the headspace and the ASTM method can be explained by a contamination of the sample by the injection of the oil into the vial. Five gases out of the seven listed in Table VI were measured with greater accuracy with the headspace method compared to the ASTM method.

**In-service oil samples analysis.** Since the HSP-GC method was developed as an alternative to the ASTM method, a series of comparisons were made between these two methods on real transformer oil samples. A total of 20 syringes were collected from on-line units. Fig. 7 show the correlation obtained for two of the gases studied. The slopes and correlation factors for all gases studied given in Table VII show that good correlation exists between the two methods. Most of the points are well within the 95% confidence range. The deviation noted for the total gas content is due to the difference in its determination: with the ASTM method, the volume compressed in the burette at atmospheric pressure is used whereas the HSP-GC total gas content is the numerical summation of the individual gases measured by chromatography. Thus, headspace extraction allows quantitative analysis of dissolved gases to be performed in transformer insulating oil with analytical performances similar to the currently used ASTM D3612 vacuum method.

TABLE V  
DISTRIBUTION COEFFICIENTS OF FAULT GASES IN VOLTESSO 35 OIL-ARGON SYSTEM AT 70°C

Gas	Distribution coefficient $K$ (Ostwald) <sup>a</sup>
H <sub>2</sub>	0.074 ± 0.003
O <sub>2</sub>	0.20 ± 0.04
N <sub>2</sub>	0.11 ± 0.04
CO	0.20 ± 0.02
CH <sub>4</sub>	0.40 ± 0.01
CO <sub>2</sub>	0.90 ± 0.03
C <sub>2</sub> H <sub>4</sub>	1.40 ± 0.04
C <sub>2</sub> H <sub>6</sub>	1.80 ± 0.04
C <sub>2</sub> H <sub>2</sub>	1.01 ± 0.02
C <sub>3</sub> H <sub>8</sub>	5.1 ± 0.5

<sup>a</sup> ( $V_G/V_L = 0.4913$ ).

TABLE VI  
ACCURACY AND PRECISION OF HEADSPACE AND ASTM EXTRACTION METHODS

Gas	Reference value (ppm)	Measured value (ppm)	Precision (%)	Accuracy (%)	Extraction method
H <sub>2</sub>	71	66	3	-6	HSP
		82	4	+16	ASTM
CH <sub>4</sub>	141	129	3	-9	HSP
		170	3	+20	ASTM
CO	142	119	2	-16	HSP
		150	3	+6	ASTM
CO <sub>2</sub>	220	234	14	+6	HSP
		266	7	+21	ASTM
C <sub>2</sub> H <sub>4</sub>	71	76	3	+8	HSP
C <sub>2</sub> H <sub>6</sub>	72	79	5	+12	ASTM
		66	3	-8	HSP
C <sub>2</sub> H <sub>2</sub>	67	83	5	+16	ASTM
		57	4	-14	HSP
		69	4	+3	ASTM

<sup>a</sup> ± 1% [8].

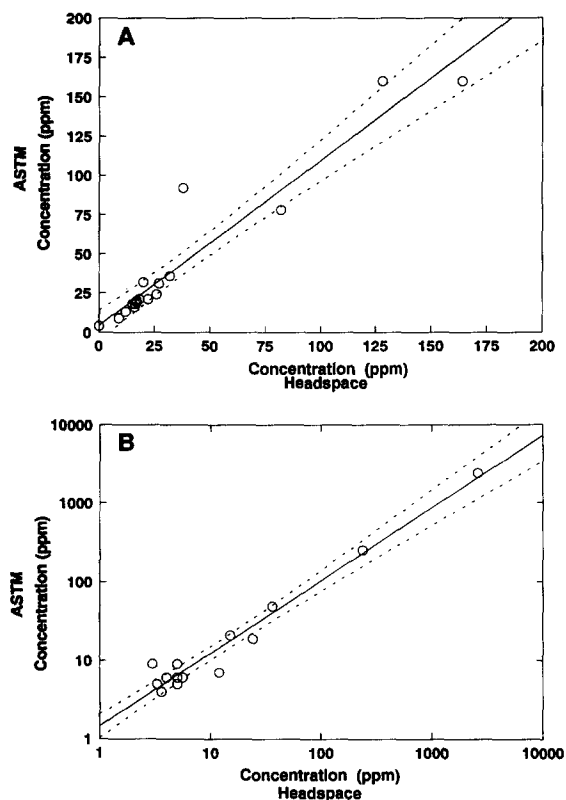


Fig. 7. Correlation between analysis results from ASTM D3612 and headspace extraction method for hydrogen and ethylene. (A) H<sub>2</sub>; (B) C<sub>2</sub>H<sub>4</sub>.

## CONCLUSIONS

The fact that the HSP-GC sampling can be automated allows for the possibility of achieving a higher number of analyses daily with minimal supervision. As much as 41 samples can be analyzed daily as compared to nine using the ASTM method over

TABLE VII  
CORRELATION BETWEEN DISSOLVED GAS ANALYSIS RESULTS FROM HEADSPACE AND ASTM D3612 EXTRACTION METHODS FOR IN-SERVICE TRANSFORMER OILS

Gas	Slope	Correlation factor
H <sub>2</sub>	1.05	0.9556
O <sub>2</sub>	1.05 <sup>a</sup>	0.9862 <sup>a</sup>
N <sub>2</sub>	0.85	0.9561
CH <sub>4</sub>	0.88	0.9714
C <sub>1</sub> H <sub>4</sub>	0.99	0.9997
CC	0.94	0.9857
C <sub>2</sub> H <sub>2</sub>	0.96	0.9887
C <sub>2</sub> H <sub>4</sub>	0.92	0.9999
C <sub>2</sub> H <sub>6</sub>	1.00	0.9990
C <sub>2</sub> H <sub>2</sub>	0.64 <sup>b</sup>	0.9992 <sup>b</sup>
C <sub>3</sub> H <sub>8</sub>	0.81	0.9661
Total gas content	1.30	0.9572

<sup>a</sup> Without erroneous point on Fig. 7.

<sup>b</sup> Detected in five samples only.



a regular day shift. The major drawback of this method is the long initial equilibration time (200 min) necessary before the chromatographic analysis. However, once the batch of vials is at equilibrium, the time elapsed between two successive analyses will depend only on the length of the chromatographic run which is approximately 35 min.

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