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A Comparison Study of Sampling and Analyzing Volatile Organic Compounds in Air in Kuwait by Using Tedlar Bags/Canisters and GC-MS with a Cryogenic Trap

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Kuwait experiences desert climatic weather. Due to the extreme hot and dry conditions in this country, some analytical phenomena have been discovered. Therefore, a systematic study of sampling and analyzing volatile organic compounds in air by using GC-MS with a cryogenic trap is reported in this paper. This study included comparisons of using different sample containers such as Tedlar bags and SUMMA canisters, and different cryogenic freezing-out air volumes in the trap. Calibration curves for different compounds and improvement of replicated analysis results were also reported here. The study found that using different sample containers produced different results. Analysis of ambient air samples collected in Tedlar bags obtained several volatile organic compounds with large concentrations compared to using SUMMA canisters. Therefore, to choose a sample container properly is a key element for successfully completing a project. Because GC-MS with a cryogenic trap often generates replicated results with poor agreement, an internal standard added to gas standards and air samples by using a gas syringe was tested. The study results proved that it helped to improve the replicated results.

KEYWORDS: volatile organic compounds, analysis, Tedlar bag, SUMMA canister

INTRODUCTION

Ambient and indoor airs contain a large number of volatile organic compounds (VOCs) at levels that exhibit ecological and health effects. The role and importance in atmospheric chemistry of organic compounds produced by human activity was recognized by Haagen-Smith about 50 years ago[1]. The VOCs with 1–11 carbons in the molecules are precursors required for the photochemical production of ozone in the atmosphere. The very important thing to human beings is that many VOCs are individually carcinogenic and mutagenic in their own right. VOCs are also greenhouse gases. Under the UN Framework Convention on Climate Change on December 4, 1997, they should be controlled. Therefore, monitoring VOC concentrations in air has become a very important issue in environmental protection.

Health effects of VOCs have focused on benzene, which is a carcinogenic compound even if at very low concentrations (sub-ppb level). It is thought that benzene and 1,3-butadiene exposure may account for around 10% of leukemia incidence across the U.K.[2]. It has been recognized that the benzene added to unleaded petrol to maintain vehicle performance may be a worse threat to human health than the lead that it displaced[2].

Concern about public health and the correct assessment of the factors associated with visibility impairment by organic compounds have produced a concerted effort throughout the world to develop sampling and analytical methods in air.

The demand for accurate, reliable, and sensitive techniques for monitoring trace levels of VOCs has been an issue for further research and improvement due to their volatility and small concentration in ambient air for a long time. The selection of an optimal sampling method for target compounds depends greatly on the physicochemical nature of these compounds and their expected concentrations in the air.

The most important physical properties governing the design of a sampling method for VOCs from air are vapor pressure and polarity. VOCs are compounds whose vapor pressures are ranged less than 10^{-5} KPa[3]. Over the past few decades, many methods for monitoring VOCs in air have been developed and subsequently improved. Those methods include active integrative method, passive integrative method, and active real-time analytical method or semi-real-time analytical method. Semi-real-time methods employ various types of bags, canisters, and adsorbent cartridges to collect samples that may then be analyzed in the field by a variety of techniques such as gas chromatograph (GC) or GC with mass spectrometry (MS). The majority of methods that have been developed for ambient VOC monitoring have been either container based (such as a canister) or sorbent based[4]. Canister sampling involves the collection of the whole air matrix in a precleaned evacuated collection device. This method is very useful for the most volatile species like C2 – C3 hydrocarbons. The sorbent method normally uses a pump to sample air samples. The common adsorbents include activated carbon, porous polymers, molecular sieve, silica gel, etc.

The canister includes SUMMA canisters and specially prepared canisters[5]. They can be used for collecting air samples over the atmospheric pressure (pressurized) or below the atmospheric pressure (subatmospheric pressure). Many of the VOCs in the USEPA Method TO-15 have been tested for stability in concentration when stored in canisters under conditions typical of those encountered in routine ambient air. Actually, the stability of these compounds under all possible conditions is not known[6]. A model to predict compound losses due to physical adsorption of VOCs on canister walls and to dissolution of VOCs in water condensed in the canisters was developed by Coutant[7]. The losses of VOCs in the canister can be generally classified as physical adsorption, chemical reaction, aqueous hydrolysis, and biological degradation. Unfortunately, there is no model that can be used to estimate all these losses.

Canister-based samplers have been evaluated by Jayanty[8]. The study found that the canister was an efficient means for the collection of some selected volatile and toxic compounds. The selected organic compounds could be stable over a period of 2 weeks. Numerous compounds, many of which are chlorinated VOCs, have been successfully tested for storage stability in pressurized canisters[9,10]. However, minimal documentation is currently available demonstrating stability of VOCs in subatmospheric pressure canisters.

Tedlar bags have been routinely used for collection of air samplers for many years and the Tedlar bag sampling system is one of the recommended sampling techniques cited in USEPA Reference Method 18[11]. The applicability of Tedlar bag sampling systems for collection and analysis of VOCs was studied by McGaughey et al.[12]. Pau et al. introduced a new, smaller (5–7 l) Tedlar bag in 1991[13], and showed that smaller Tedlar bags can be used effectively for sampling many toxic organic compounds and that the small bags have similar relative standard deviations in comparison to the large ones. The big drawback for the Tedlar bags is the loss of smaller molecular VOCs such as C_2 during storage.

Gas chromatography equipped with a mass spectrometer as detector (GC-MS), because of its high sensitivity and selectivity for VOCs, has been incorporated into cryogenic traps for VOC analysis. A cryogenic trap normally is packed with small particles such as glass beads in order to increase contact

surfaces. When gaseous samples pass the trap, the sample flow cannot keep the same pathway from run to run. If the sample flow is closer to the trap wall, the trapping efficiency will be higher than in the trap center since the trap is surrounded by liquid nitrogen and the walls directly contact the liquid nitrogen. Thus, trapping efficiencies from run to run will be different. Therefore, due to the operation nature in the system, the replicate analytical results always have some large deviations. Average results for the replicated analysis are normally used in the EPA TO14 and TO15 methods.

Kuwait lies at the northwest corner of the Arabian Gulf, between latitudes of 28 and 30 N and between longitudes 46 and 48 E. Most of the Kuwait mainland is a flat sandy desert, gradually sloping down from the extreme west of Shigaya and Salmi (300 m high) towards sea level in the east. Therefore, the weather in summer in Kuwait is extremely hot and has fewer rains. The photochemical reactions in this area are expected to be really active. We used SUMMA canisters and Tedlar bags to sample VOCs in the same outdoor location, and found different compounds from air samples in canisters and Tedlar bags. In order to find the reasons, a systematic study of VOC analysis including sampling methods, cryogenic trapping volume, replicate analysis with and without internal standard, etc. was conducted. The findings are reported in this paper.

EXPERIMENTAL PROCEDURE

Sampling Method

Three different sampling methods were employed in this study, which include SUMMA canister, Tedlar bag, and activated carbon tube.

- **SUMMA Canister** — 6-l SUMMA stainless steel canisters (Entech, USA) were used. Before sampling, the canister was cleaned and evacuated by a canister clean system (Entech, USA). Subatmospheric pressure procedure was employed in this study.
- **Tedlar Bag** — 1- and 5-l Tedlar bags were used. The Tedlar bags were cleaned as follows. Each Tedlar bag was filled with zero air and heated above an oven at about 50°C for 5 min. Then, the Tedlar bag was evacuated. This procedure was repeated 10 times.
- **Activated Carbon Tube** — The activated carbon tubes were supplied by SKC, Canada. The carbon was packed in two sections in the tube separated by glass wool in the middle.

VOC and Internal Standards

In this study, 39 different VOC mixtures (Scott-Marrin, Inc. USA) kept in a cylinder were used as standards. The compound names, formulas, and molecular weights are listed in Table 1. With pure air (zero air), each VOC was diluted to three concentration levels in Tedlar bags: 5, 25, and 50 ppb.

2-Methoxy-2-methyl propane (Fisher, USA) was used as internal standard. The compound was first diluted to 42 ppm in a Tedlar bag as a stock standard. Then 1 ml of the stock standard was injected into a Tedlar bag filled with air samples or calibration standards by using a gas syringe.

Analytical Device

A GC-MS system (Hewlett Packard, USA, Model G1800A) equipped with a preconcentrator (Entech,USA, Model 7000) was used for analysis of air samples in canisters and Tedlar bags. The GC-MS was operated in scan mode. A fused silica capillary column (60 m × 0.2 mm I.D., 1.1-µm film thicknesses) was used in the GC. A temperature program was set up as follows: initial temperature 35°C; initial time 3 min; temperature from 35 to 95°C at 15°C/min, then to 105°C at 2.5^{o}C/min; finally to 205°C at 5°C/min, and at 205°C for 5 min.

TABLE 1
List of VOC Concentrations (Volume Fraction) in the Standard Mixture

SR No.	Name of Compounds	Concentration	Mol. Wt.	Formula
1	Chlorodifluoromethane	50 ppb	86.5	$CHClF_2$
2	n-Butane	50 ppb	58.08	C_4H_{10}
3	Butane, 2-methyl	50 ppb	72.15	C_5H_{12}
4	Furan	50 ppb	68.07	C_4H_4O
5	n-Pentane	50 ppb	72.14	C_5H_{12}
6	Dichloromethane	50 ppb	84.93	CH_2Cl_2
7	Ethane, 1,1,2-trichlotrifluoro	50 ppb	187.38	$C_2Cl_3F_3$
8	Cyclopentane	50 ppb	70.134	C_5H_{10}
9	Acetone	50 ppb	58.07	C_3H_6O
10	Pentane, 2-methy	50 ppb	86.17	C_6H_{14}
11	2-Methyl-1-pentene	50 ppb	84.16	C_6H_{12}
12	Trichloromethane	50 ppb	119.38	$CHCl_3$
13	n-Hexane	50 ppb	86.176	C_6H_{14}
14	Methylcyclopentane	50 ppb	84.16	C_6H_{12}
15	Tetrachloromethane	50 ppb	153.82	CCl_4
16	Ethanol	50 ppb	46.06	C_2H_6O
17	2-Butanone	50 ppb	72.106	C_4H_8O
18	Benzene	50 ppb	78.11	C_6H_6
19	Cyclohexane	50 ppb	84.16	C_6H_{12}
20	3-Methylhexane	50 ppb	100.203	C_7H_{16}
21	n-Heptane	50 ppb	100.2	C_7H_{16}
22	Methylcyclohexane	50 ppb	98.18	C_7H_{14}
23	1,4-Dioxane	50 ppb	88.106	$C_4H_8O_2$
24	2-Pentanone	50 ppb	86.13	$C_5H_{10}O$
25	2,4-Dimethylhexane	50 ppb	114.23	C_8H_{18}
26	Toluene	50 ppb	92.14	C_7H_8
27	3-Methylheptane	50 ppb	114.23	C_8H_{18}
28	Ethylbenzene	50 ppb	106.167	C_8H_{10}
29	p-Xylene	50 ppb	106.17	C_8H_{10}
30	m-Xylene	50 ppb	106.17	C_8H_{10}
31	o-Xylene	50 ppb	106.17	C_8H_{10}

TABLE 1 (CONTINUED)

32	1-Ethyl-3-methylbenzene	50 ppb	120.2	C_9H_{12}
33	1-Ethyl-4-methylbenzene	50 ppb	120.2	C_9H_{12}
34	1,3,5-Trimethylbenzene	50 ppb	120.2	C_9H_{12}
35	1-Ethyl-2-methylbenzene	50 ppb	120.2	C_9H_{12}
36	1,2,4-Trimethylbenzene	50 ppb	120.2	C_9H_{12}
37	n-Decane	50 ppb	142.28	$C_{10}H2_2$
38	1,2,3-Trimethylbenzene	50 ppb	120.2	C_9H_{12}
39	n-Dodecane	50 ppb	170.337	$C_{12}H_{26}$

The VOCs collected in the canisters and Tedlar bags were enriched by passing a known volume of air through a trap in the preconcentrator using cryogenic preconcentration and cryofocusing. The sample was finally transferred to the GC.

A GC-MS system (Agilent Technologies, USA, Model 5973) was used for analysis of VOCs collected in the activated carbon tube. After collection, the carbon in the tube was extracted with dichloromethane. The extract was injected into the GC-MS system by using a syringe.

Both GC-MS systems had a Wiley 138 library for identifying VOCs.

RESULTS AND DISCUSSION

VOCs in Canisters and Tedlar Bags

Basically, the ambient air VOCs found in the canisters and Tedlar bags were almost the same at the same location in Kuwait. But, it was a surprising finding that several high-concentration VOCs (such as VOCs at retention time 18 and 23 min, hereby named as UVOC) found in the Tedlar bags had very small peaks in the canister samples (see Figs. 1 and 2). In order to further confirm the findings, air samples were also collected outdoors and indoors by Tedlar bags, and outdoors by an activated carbon tube. The analytical results from indoor and outdoor air samples in the Tedlar bags clearly showed that UVOC did indeed exist in the ambient air (see Fig. 3).
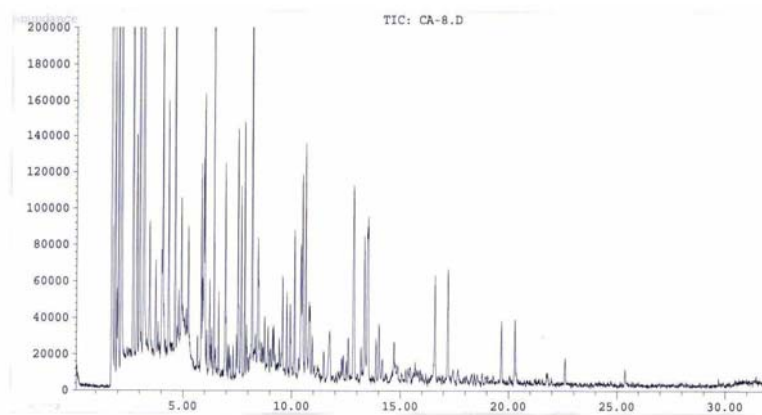

FIGURE 1. Total ion chromatogram of an air sample collected in a canister, preconcentration volume 300 ml.
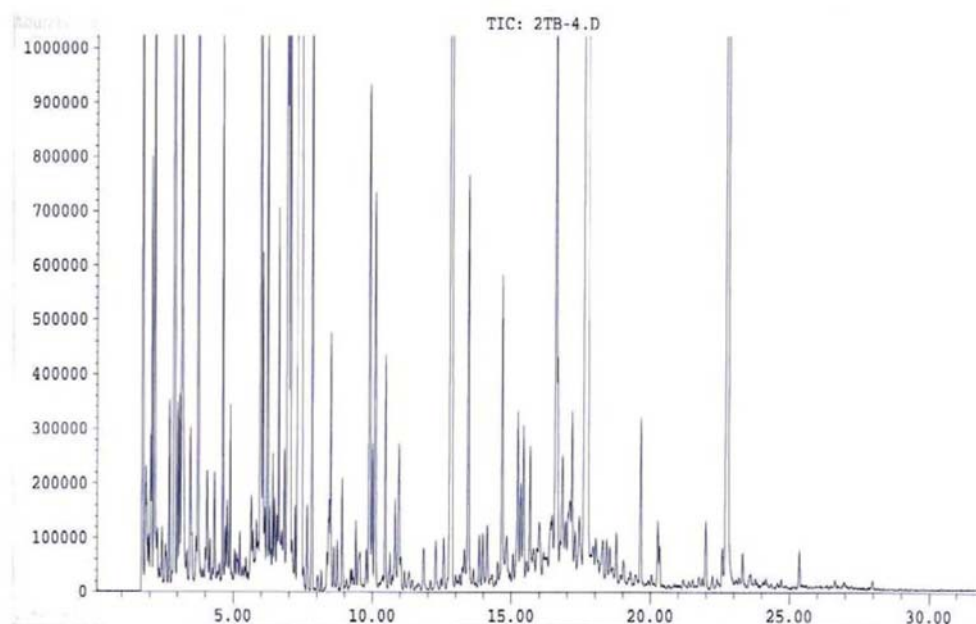
FIGURE 2. Total ion chromatogram of an air sample collected in a Tedlar bag, preconcentration volume 300 ml.
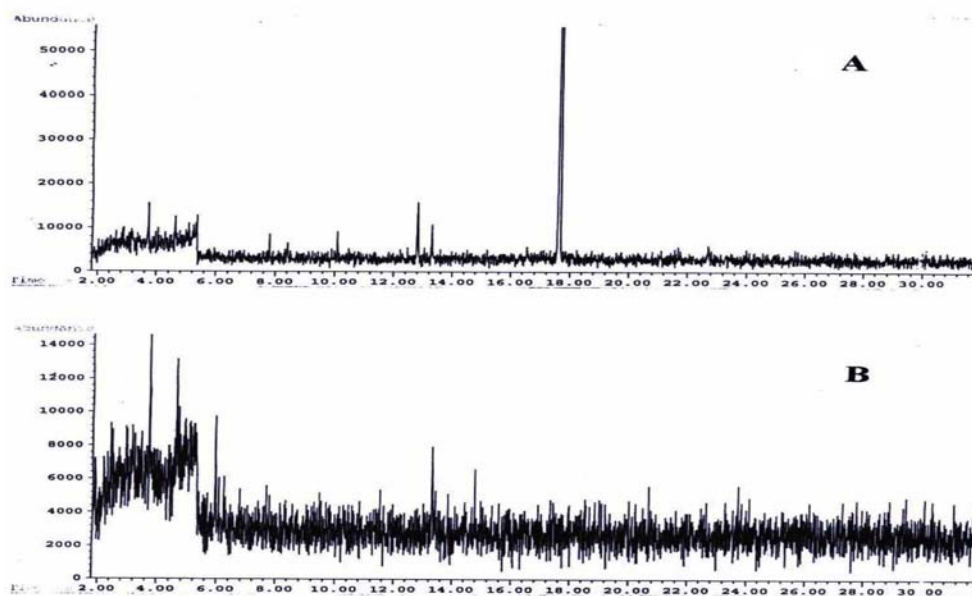

FIGURE 3. Comparison of total ion chromatograms of air samples collected outdoors (A) and indoors (B) in Tedlar bags, preconcentration volume 75 ml.

The GC-MS full-scan total ion chromatograms of air samples collected in an activated carbon tube and analyzed by a GC-MS without a cryogenic preconcentrator are shown in Fig. 4. It is not surprising to find that a very big peak, which has a relative longer retention time (about 26 min), appears in the chromatogram. Unfortunately, library search results from GC-MS cannot give positive identifications of the UVOC, but they do give a strong indication of the existence of the UVOC in Kuwait's ambient air.
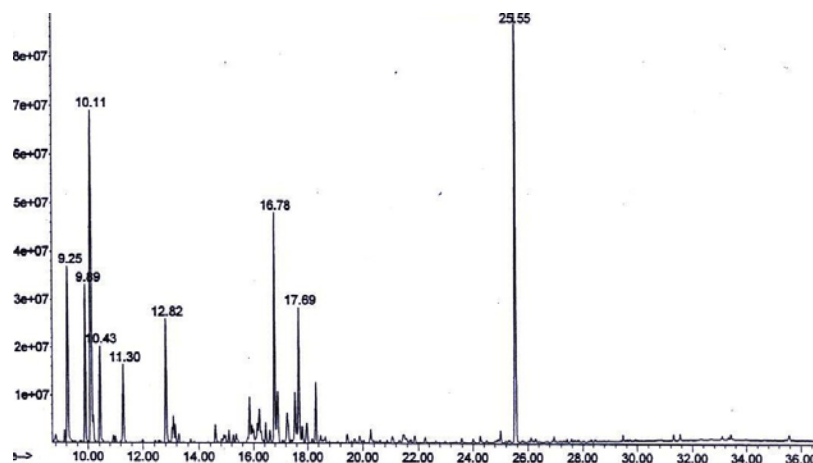
FIGURE 4. Total ion chromatogram of air sample collected by an activated carbon tube.

Tedlar bags utilize a lightweight, patented single fitting of inert polypropylene that combines the hose/valve and the septum holder into one compact fitting. A SUMMA canister is a stainless steel vessel that has had the internal surfaces specially passivated using a special process. This process combines an electropolishing step with chemical deactivation in order to produce a surface that is chemically inert. Actually, it is very difficult to deactivate all active points inside the canister's internal surface. The remaining active points will chemically or physically react with some compounds, which might be the reason to explain what we found in samples stored in canisters.

As described above, Kuwait belongs to a desert climate. Some photochemical reactions may produce special VOCs. It is necessary to further study this issue in Kuwait. This study also indicates that in order to obtain a more accurate picture of VOCs in a selected location, using different sampling technologies can avoid unnecessary mistakes.

The Use of Internal Standard

It has been noticed that the analytical deviations among replicated results were large when using GC-MS equipped with a preconcentrator. In order to improve the analytical data quality, an add-in internal standard to Tedlar bags was studied. The procedure included simply using a syringe to add the internal standard to Tedlar bags containing air samples and VOC standards. The operation is easy and flexible. Our experiments found that adding an internal standard into air samples in Tedlar bags can greatly help to improve the data quality. As described above, using a syringe can easily introduce the internal standard into a Tedlar bag. However, there are some difficulties to adding an internal standard to a SUMMA canister by syringe at this moment. Table 2 lists the comparisons of relative deviations of analytical results for several VOCs. It can be seen that after correction of the peak areas based on a correction factor (CF) derived from the internal standard peak areas at different analyses, the quality of the duplicate results has been greatly improved. The average relative deviations change from 19% (no correction) to 4% (with correction). The CF is obtained as follows.

$$CF = PA1/PA2 \quad (1)$$

where CF – correction factor, PA1 – internal standard peak area in the first analysis, and PA2 – internal standard peak area in the second analysis. The corrected area (CPA2) for the second analysis is obtained by Equation 2.

$$CPA2 = CF \; x \; PA2 \quad (2)$$

TABLE 2
Comparison of Relative Divisions in Duplicate Analysis of Several VOCs
With and Without Internal Standard

VOC ID	Peak Area in 1st Run	Peak Area in 2nd Run	Corrected Peak Area in 2nd Run	Relative Deviation Without Correction (%)	Relative Deviation After Correction (%)
Internal STD	8648348	6171565	(8648348)		
1	701020	401024	561955	43	20
2	235183	186654	261558	21	11
3	197965	132067	185065	33	7
4	209875	152944	214320	27	2
5	832409	607262	850956	27	2
6	197055	132067	185065	33	6
7	690236	530503	743394	23	8
			Average Relative Deviation:	30	8

The relative deviation (RDEV) is obtained using Equation 3.

$$RDEV = 100\frac{/(PA1 - PA2)/}{PA1} \quad (3)$$

Actually, using internal standard in calibration curve preparation can also improve the quality of the curve. For example, Table 3 lists three-level (5, 25, and 50 ppb) VOC standard peaks in three analyses. The peak areas of the internal standard in the three analyses were very different. Therefore, it is not surprising to find that each VOC's peak area had no reasonable relations compared to its standard values (see Fig. 5).

We chose 36.E+6 as the internal standard peak area for all three-level concentration analyses and the corrected peak areas are listed in Table 4. It can be observed that after correction, the relation between the peak area value and the concentration level looks much more reasonable (see Fig. 6). Table 4 and Fig. 6 demonstrate the importance of using internal standard for accurately analyzing VOCs in air.

Effects of Preconcentrated Air Sample Volumes to Analytical Results

From an analytical point of view, preconcentrating larger air sample volumes can absolutely decrease VOC detection limits. Unfortunately, there are some risks from the instrument itself. Large preconcentration volumes might exceed the capacities of GC-MS or the preconcentrtor. For example, the Model 7000 preconcentrator declares that it can handle up to a 2000-ml air sample. Our study found that the proper volume must be obtained from tests. We used our three-level standards to study different preconcentration volumes at 150, 200, 300, and 450 ml. Then, each VOC peak's areas obtained at 25 and 50 ppb were divided by the peak area obtained at 5 ppb, respectively. The average peak area ratios for all VOCs at different standard levels are listed in Table 5. It can be found that 300 ml was the highest preconcentration volume for most of the VOCs in the three standard levels. But when using 450 ml as the preconcentration volume, the ratios already dropped to about 85% of the theoretical ratio. The above findings indicate that the VOC concentrations in an air sample are a key factor for selecting a proper preconcentration volume. For accurate results, conducting several analyses using different preconcentration volumes will help to improve analytical data quality. It is also noticed that some VOCs (such as 1,2,3-trimethylbenzene) showed almost the same peak areas at 5-, 25-, and 50-ppb levels in the studied preconcentration volumes, which indicates that even at 150 ml, the system was already saturated for 1,2,3-trimethylbenzene.

TABLE 3
Comparison of VOCs and Internal Standard Peak Areas at Three Concentration Levels

| Number | VOC ID | Original Peak Area | | |
		5 ppb	25 ppb	50 ppb
	Internal standard	36350888	8604547	16874926
1	Methanechlorodifluoro	3241465	1440118	11394498
2	Butane	1657076	922832	5759330
3	Butane, 2-methyl	2642256	2042028	12092306
4	Pentane	2183484	1861501	10502774
5	2,3-Pentadiene	3455701	2039580	9410610
6	Ethane, 1,1,2-trichlo	8596797	7430762	48840642
7	Methanedichloro	2315715	2010836	10651796
8	Pentane, 2-methyl	2575466	2852925	14781786
9	Cyclopentane	1620627	1814409	8842649
10	1-Pentene, 2-methyl	2392801	2987193	14938405
11	Hexane	2457312	3111803	15863266
12	2-Butanone	1063975	1552708	5981736
13	Chloroform	5693086	9049861	46501868
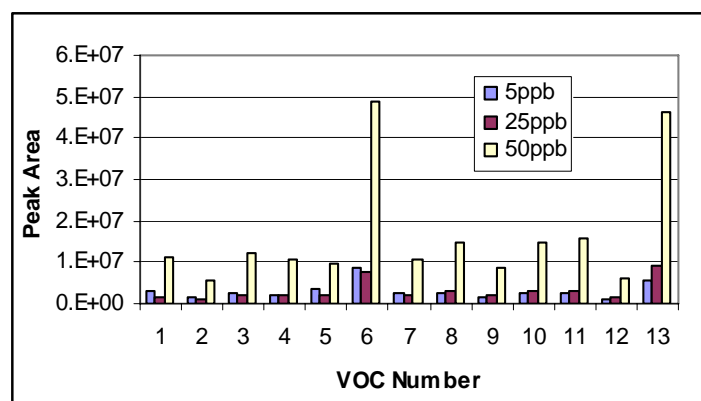
FIGURE 5. Uncorrected VOC peak areas comparison at three-level concentrations.

Our study also showed that there was a risk to use different preconcentration volumes for standards and air samples. Table 6 lists some VOC peak area comparisons at different preconcentration volumes. All the areas have been corrected based on the internal standard. It can be found that the peak areas are not proportional to the preconcentration volumes. For example, the area ratio between volume 150 to 50 ml should be 3, but the actual ratios are from 3 to 5. Thus, if using 50 ml as preconcentration volume for standard and using 150 ml as preconcentration volume for air samples, relative standard deviations for some VOCs such as VOCs with area rations 5 will be very large. Therefore, using same volumes for calibration standards and air samples can improve analytical data quality.

TABLE 4
Comparison of Corrected VOCs Peak Areas and Internal Standard
Peak Areas at Three Concentration Levels

Number	VOC ID	Corrected Peak Area		
		5 ppb	25 ppb	50 ppb
	Internal standard	36000000	36000000	36000000
1	Chlorodifluoromethane	3209050	6019693	24270281
2	Butane	1640505	3857438	12267373
3	Butane, 2-methyl	2615833	8535677	25756612
4	Pentane	2161649	7781074	22370909
5	2,3-Pentadiene	3421144	8525444	20044599
6	Ethane, 1,1,2-trichlo	8510829	31060585	104030567
7	Dichloromethane	2292558	8405294	22688325
8	Pentane, 2-methyl	2549711	11925227	31485204
9	Cyclopentane	1604421	7584230	18834842
10	1-Pentene, 2-methyl	2368873	12486467	31818803
11	Hexane	2432739	13007337	33788757
12	2-Butanone	1053335	6490319	12741098
13	Chloroform	5636155	37828419	99048979

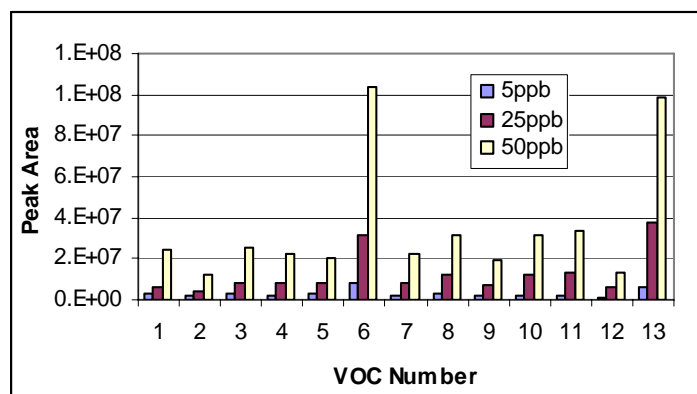**FIGURE 6**. Corrected VOC peak areas comparison at three-level concentrations.

TABLE 5
Comparisons of Average Peak Ratios at Different Preconcentration Volumes

Preconcentration Volume (ml)	Average Peak Area Ratio of 25 ppb/5 ppb	Average Peak Area Ratio of 50 ppb/5 ppb
150	4.55	9.20
300	4.29	9.12
450	4.20	8.54

TABLE 6
Comparison of Several VOC Peak Areas at Different Preconcentration Volumes

VOC Number	50 ml (Cor. Area)	150 ml (Cor. Area)	300 ml (Cor. Area)	Area Ratio (150 ml/50 ml)	Area Ratio (300 ml/50 ml)
1	264867	892845	1375196	3	5
2	115618	434844	742237	4	6
3	146620	739170	1362560	5	9
4	126760	619799	1109848	5	9
5	224276	652942	1152989	3	5
6	575311	2817176	5370401	5	9
7	181048	618710	1145457	3	6
8	201831	808736	1485582	4	7
9	112331	523807	992741	5	9
10	178888	803416	1594996	4	9
11	194841	779392	1564041	4	8
12	81697	332093	703246	4	9
13	444177	2199468	4855595	5	11
14	250570	1078752	2215903	4	9
15	240958	1145566	2672100	5	11
16	578807	2510966	5003997	4	9

CONCLUSIONS

Kuwait experiences desert climatic weather. Due to the extreme hot and dry conditions in this country, our systematic study of analysis of VOCs in air by using GC-MS with a cryogenic trap has found useful information. Using Tedlar bags and SUMMA canisters to collect VOCs in the ambient air had different results; some VOCs had substantially decreased their concentrations in canisters. Different cryogenic preconcentration volumes in the trap could affect the analytical data quality, calibration curves for different compounds, and replicated results. The study concludes that to choose a sample container properly is a key element for successfully completing a project. Because GC-MS with a cryogenic trap is a dynamic system, replicated results normally showed poor agreement. The add-in internal standard to Tedlar bags containing standards and air samples can help to improve the replicated results.
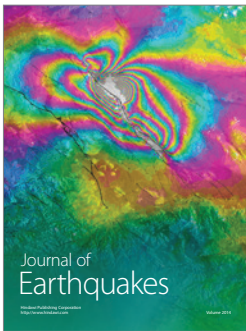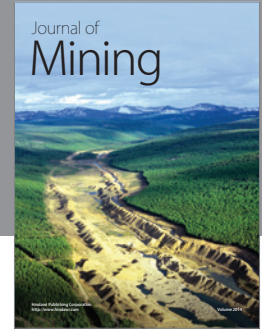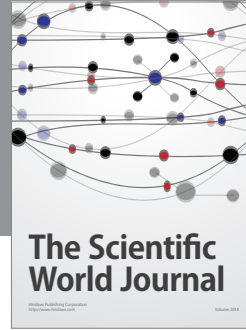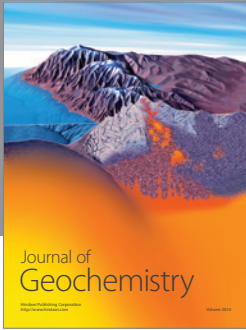
ACKNOWLEDGMENTS

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