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Contamination of Air Samples by Alkanes from Parafilm™

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The presence of organic contaminants of various kinds in samples has been a problem in sampling and analysis for environmental pollutants. These contaminants have been found in solvents, filters, sorbents, glasswares, and various materials used in the process of sample preparation (Bowers et al. 1981, Karasek et al. 1981). Some contaminants may be formed during sampling and during storage due to the reaction between pollutants and sampling media (Hanson et al. 1981, Hunt et al. 1982). Other contaminants may be introduced accidentally to the sample. Contaminants may interfere with the analysis for intended compounds. Sometimes they can even lead to wrong assignments of compounds; that is the situation of false positive or false negative.

In this paper, we report the study of contaminants in an air sample caused by accidental introduction. A series of alkanes dissolved from Parafilm by solvent was the cause of contamination.

MATERIALS AND METHODS

High-purity cyclohexane from Merck, Inc. was used for extraction. Glass-fiber filters used in sampling were purchased from SKC, Inc. Parafilm was a commercial product manufactured by American National Can. These materials were used without further cleaning up. Field blank was always used along with samples for analysis. Air samples were taken using the personal pump made by SKC, Inc. Sampling rate was set at 2 L/min and the duration was for 8 hr. Filters were returned to the laboratory for ultrasonic extraction with cyclohexane for one hour. Extracts were concentrated by purging with nitrogen before subjecting to GC analysis.

Samples were analyzed by a Hewlett-Packard 5988A GC/MS system. The HP 5890 GC was fitted with a 30 m x 0.25 mm i.d. DB-5 fused silica capillary column. The oven was

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at first maintained at 75°C for 3 min, and then followed by an increase of 12°C per min to 174°C, 4°C per min to 192°C, 8°C per min to 252°C and finally 3°C per min to 290°C. This temperature was maintained for 5 min. The MS system was operated in the electron impact (EI) mode at 70 eV with scanning from 50 to 350 amu. Compounds were identified using a HP 59970 MS Chem-Station to conduct PBM searching in the NIST library database NBS49K.L.

RESULTS AND DISCUSSION

Samples were analyzed by GC/MS system in the EI mode. They were a Parafilm sample and contaminated and non-contaminated air samples. Total-ion chromatogram (TIC) of these samples are shown in Figure 1 where the similarity of profile can be seen readily in the longer-retention-time region of the chromatogram. Comparison of air sample with Parafilm sample is made further and is shown in Figure 2. Four parameters were used to determine whether there was any external contaminants introduced. These four parameters were retention time, peak area, peak height and mass spectrum of each compound for the samples; i.e., Parafilm and contaminated and non-contaminated air samples. A comparison of these parameters for identified peaks is given in Table 1. Retention time of each peak agrees within acceptable limit for these three samples. The normalized area to peak at retention time 33.2 min is given in the same table. All three samples have a largest peak at 33.2

Table 1. Peak area comparison of Parafilm and contaminated and non-contaminated air samples.

Retention time(min)	Parafilm sample	Contaminated air sample	Non-contaminated sample
1. 21.9	0.03	0.01	0.08
2. 23.1	0.12	0.46	3.82
3. 24.3	0.27	0.38	5.24
4. 25.7	0.48	0.56	0.52
5. 27.4	0.73	0.76	0.55
6. 29.2	0.83	0.93	0.99
7. 31.2	0.87	0.73	0.92
8. 33.2	1.00	1.00	1.00
9. 35.2	0.91	0.48	0.79
10. 37.2	0.82	0.88	0.66
11. 39.3	0.50	0.71	0.44
12. 41.3	0.36	0.58	0.27
13. 43.7	0.24	0.35	0.15
14. 46.5	0.13	0.22	----
15. 49.8	0.05	0.07	----

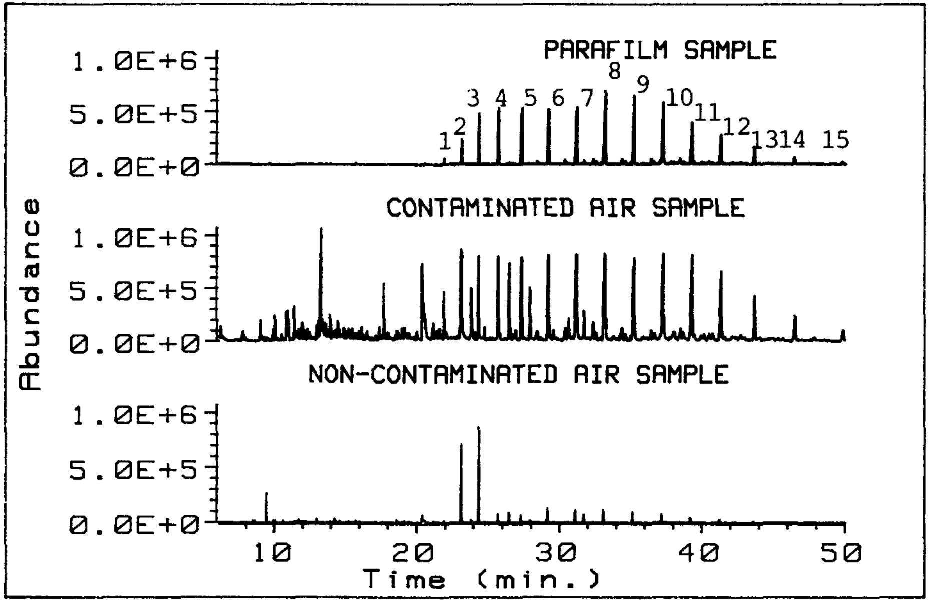

Figure 1. Total-ion chromatogram of Parafilm and contaminated and non-contaminated air samples.

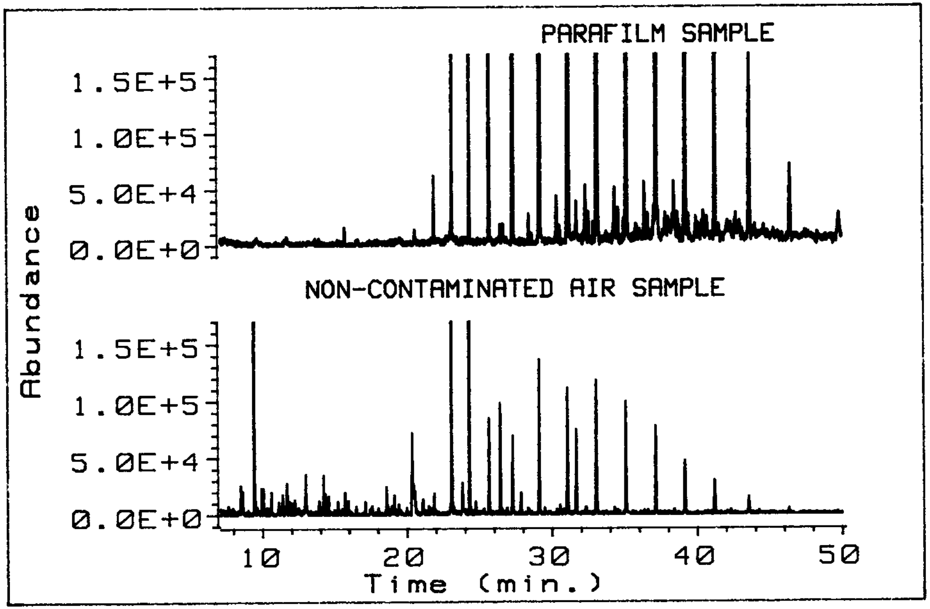

Figure 2. Total-ion chromatogram of Parafilm and non-contaminated air samples.

min. These results are further shown in Figure 3 where one can see a close similarity of relative area for all peaks except for #1 and #2 peak of the air sample. They are the result of contamination from the glass fiber filter.

In Table 2, peak height comparison for these three samples is given and is further shown in Figure 3. There is close similarity for all except for #1 and #2 peak. There is no striking difference when comparison is made for both peak area and peak height for three samples.

Table 2. Peak height comparison of Parafilm and contaminated and non-contaminated air samples.

Retention time(min)		Parafilm sample	Contaminated air sample	Non-contaminated sample
1.	21.9	0.09	0.56	0.15
2.	23.1	0.35	1.03	5.95
3.	24.3	0.68	0.97	7.48
4.	25.7	0.79	0.96	0.69
5.	27.4	0.79	0.96	0.60
6.	29.2	0.78	0.99	1.17
7.	31.2	0.82	0.99	0.96
8.	33.2	1.00	1.00	1.00
9.	35.2	0.96	0.84	0.85
10.	37.2	0.85	0.99	0.64
11.	39.3	0.56	0.97	0.40
12.	41.3	0.40	0.78	0.26
13.	43.7	0.24	0.50	0.13
14.	46.5	0.10	0.29	----
15.	49.8	0.03	0.10	----

Finally, the mass spectra of three samples are analyzed. Comparison of a peak at 29.2 min of the non-contaminated air sample with the equivalent peaks of the other two samples were made and shown in Figures 4, 5 and 6. All mass spectra have a certain degree of similarity among them. However, peaks of the non-contaminated air sample show a less complicated spectrum while the other two have similar complication in their spectra. Further analysis of them reveals a mixture of many alkanes in each peak. Parafilm and contaminated air sample have more resemblance with each other.

This is the evidence that one air sample was most likely contaminated by Parafilm while the other one was not contaminated. Further confirmation with student revealed that one of the air samples was accidentally contaminated by Parafilm dissolving in sample solution during the period of solvent extraction. The series of

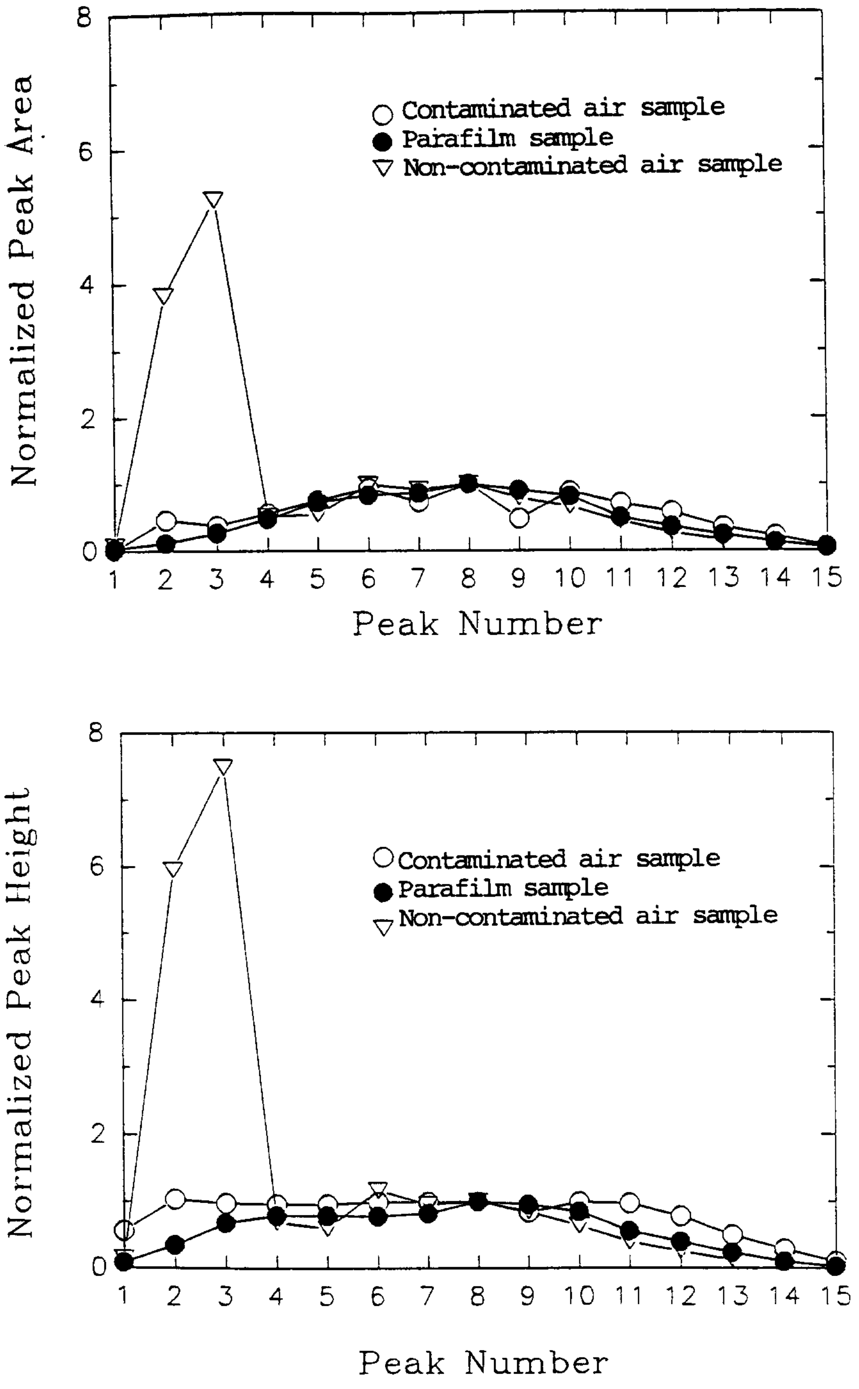

Figure 3. Comparison of peak area and peak height for three samples.

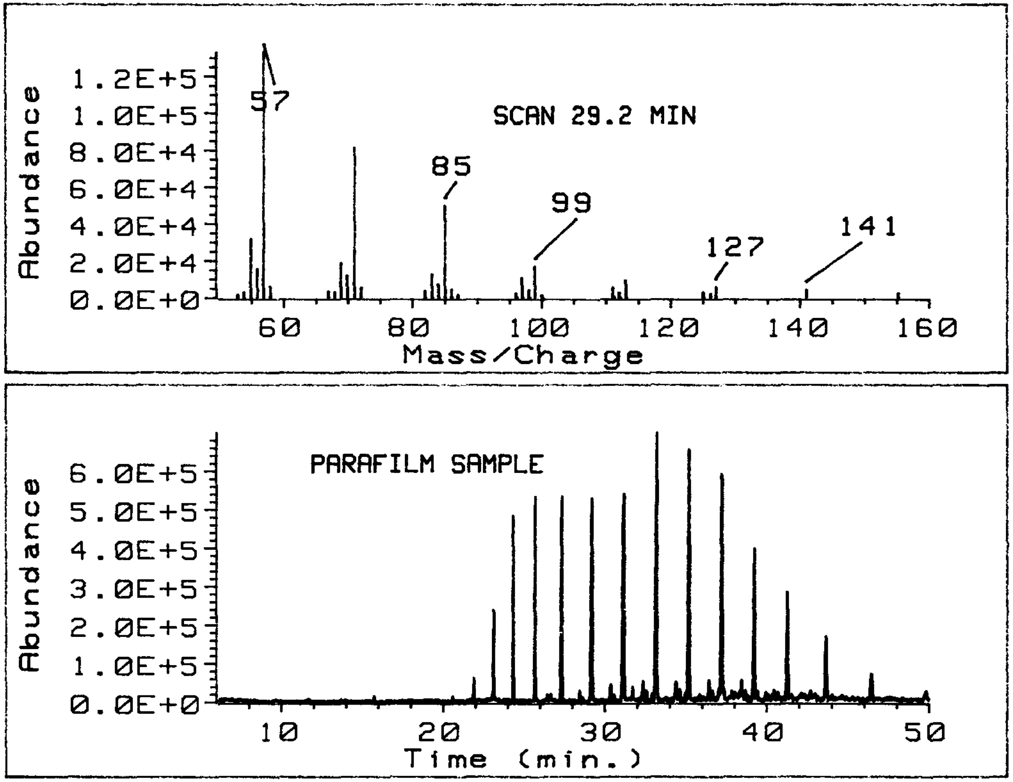

Figure 4. Mass spectrum and total-ion chromatogram of Parafilm sample.

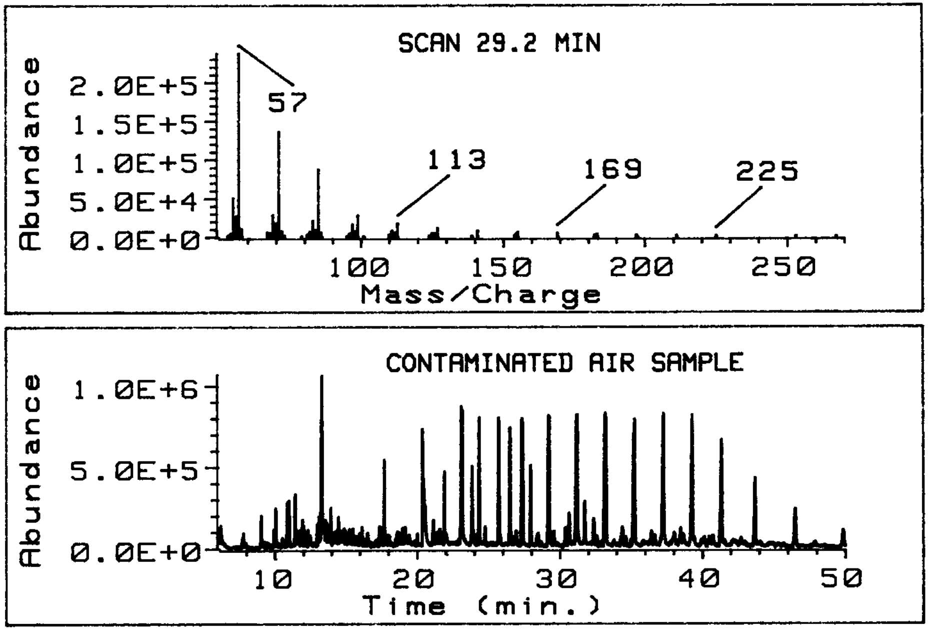

Figure 5. Mass spectrum and total-ion chromatogram of contaminated air sample.

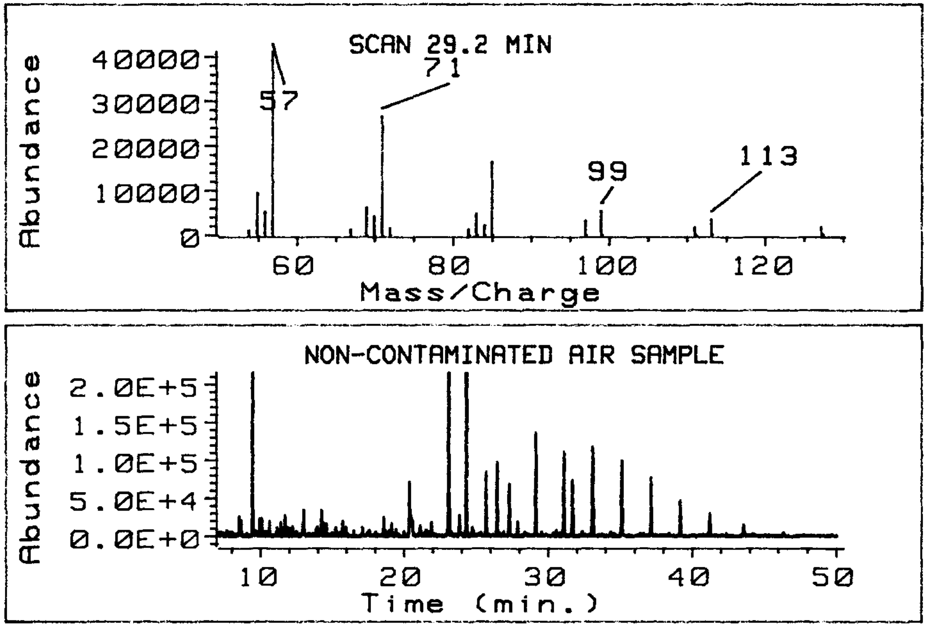

Figure 6. Mass spectrum and total-ion chromatogram of non-contaminated air sample.

peaks in the non-contaminated sample is comparable to those found and reported previously in polluted ambient air by automobile exhaust (Hampton et al. 1982, 1983).

Although each parameter alone is not conclusive enough to prove one way or the other, a combination of four parameters with the aid of reported results in the literature appears to support the conclusion that the external accidental contamination by Parafilm is the cause of the presence of a series of alkanes in the air sample. One could have reported wrong alkane compounds and much higher concentrations of the contaminated air sample if these analyses were not carefully examined. It is therefore important for analyst to take extra care to avoid contamination of any kind in sampling and analysis of air samples to safeguard the integrity of his results.

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