

This article was downloaded by:

On: 18 January 2011

Access details: Access Details: Free Access

Publisher Taylor & Francis

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## International Journal of Environmental Analytical Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713640455>

### GC-MS Analysis of Organic Vapours Emitted from Polyurethane Foam Insulation

M. E. Krzymien<sup>a</sup>

<sup>a</sup> National Research Council, Ottawa, Canada

**To cite this Article** Krzymien, M. E.(1989) 'GC-MS Analysis of Organic Vapours Emitted from Polyurethane Foam Insulation', International Journal of Environmental Analytical Chemistry, 36: 4, 193 — 207

**To link to this Article:** DOI: 10.1080/03067318908026873

**URL:** <http://dx.doi.org/10.1080/03067318908026873>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

## GC-MS ANALYSIS OF ORGANIC VAPOURS EMITTED FROM POLYURETHANE FOAM INSULATION\*

M. E. KRZYMIEŃ

*National Research Council, Ottawa, Canada K1A 0R6*

*(Received 14 November 1988)*

The vapours emitted by rigid polyurethane foam at 40° and 80° in dry and in humid (90% RH) air were trapped with a Tenax TA sampling tube and, after thermal desorption, analyzed by high resolution gas chromatography – mass spectrometry. The chromatograms obtained demonstrate a certain characteristic pattern. The qualitative composition of the effluent mixture is basically independent of both temperature and humidity of the foam. Over seventy compounds were identified as polyurethane foam off-gases. Among them the most numerous are hydrocarbons. The most abundant is the blowing agent, trichlorofluoromethane. The most interesting are cyclic acetals, aldehydes, cyclic ethers, alcohols, chloroform and chlorobenzene. The headspace concentration of the majority of them is below 10 mg/m<sup>3</sup>, there are, however, several compounds with the concentration exceeding 100 mg/m<sup>3</sup>.

**KEY WORDS:** Polyurethane foam, off-gassing, dynamic headspace sampling, GC-MS analysis.

### INTRODUCTION

The structural integrity, very good insulating properties and ease of application make rigid polyurethane foam particularly attractive as thermal and acoustic insulation. It has been extensively used in both board and spray-in-place forms in ships, aircraft, recreational vehicles and commercial buildings. Recently, on a limited scale, it has found application in residential construction. The caution with which it is used in residential buildings is primarily caused by a concern with the possible emission of harmful vapours. It has been long recognized that, when exposed to fire the foams generate toxic decomposition products.<sup>1-8</sup> The problem of the off-gassing of the foam during normal use at temperatures below 100°C and its possible effect on indoor air quality has found interest only recently.<sup>9</sup> The off-gassing is of particular interest to the construction industry and its regulating authorities as one aspect of the evaluation of polyurethane foam as an insulation in residential construction. A programme to identify and quantify the emissions has been initiated. As part of this programme, a method for sampling and analysis

---

\*Presented at the 18th International Symposium on Environmental and Analytical Chemistry, Barcelona 5-8 September, 1988.

of the emitted vapours was developed and a number of foams of various origin were analyzed.

This report describes the collection, separation and identification of the volatile emission of sprayed-in-place rigid polyurethane foam. Although complete quantitative analysis of the emissions was beyond the scope of the project, a number of off-gases was quantitated and the results are presented herein.

The off-gases emitted at 40°C and 80°C in dry air and in 90% relative humidity were identified.

## EXPERIMENTAL

### *Sampling*

A small quantity of foam to be tested (4–6 g) was placed in a dynamic gas sampling apparatus in which a stream of air swept the headspace vapours to a sampling port. Shown in Figure 1, the apparatus was a glass tube 43 mm I.D.  $\times$  30 cm closed at both ends with two teflon lined caps. The lower cap was fitted with a carrier gas inlet and the upper cap with a sampling port where an adsorber tube could be attached. The glass tube was contained in a temperature controlled aluminium heating jacket. The foam was conditioned in the sealed vessel (without flow) for a minimum of 24 hours before sampling.

The carrier gas used was compressed laboratory air dried with silica gel and purified by passage through a bed of charcoal. For most of the tests the carrier flow rate was 50 cm<sup>3</sup>/min, as was the sampling flow rate. The sample volume was varied by changing sampling time.

For humid air testing a beaker containing a saturated solution of lead nitrate was placed at the bottom of the vessel below the foam sample. This solution maintains a fairly constant relative humidity of about 90%, independent of temperature. The actual relative humidity of the air in the vessel was measured with a Humicap humidity probe (Vaisala OY, Finland). At 40° and 80°C, the relative humidity was 84% and 90% respectively.

The samples of the off-gases were collected and preconcentrated using glass adsorber tubes. An adsorber was a 6 mm I.D.  $\times$  76 mm length of Pyrex tube packed with a 25 mm column of Tenax TA 30/60 mesh. During sampling the adsorber was cooled with an ice jacket to increase the breakthrough volume.

Before introduction of the foam into the head space vessel a number of blank samples were collected and analysed to ensure that the system was contaminant free.

For analysis the adsorber was detached from the head space vessel and was inserted into a specially modified injection port of a gas chromatograph.<sup>10</sup> The injection system allowed for thermal desorption of the collected gases and vapours; the thermally desorbed analytes were then chromatographed.

### *Analysis*

The analyses were run on two gas chromatographs: a Varian Vista 4600 and a

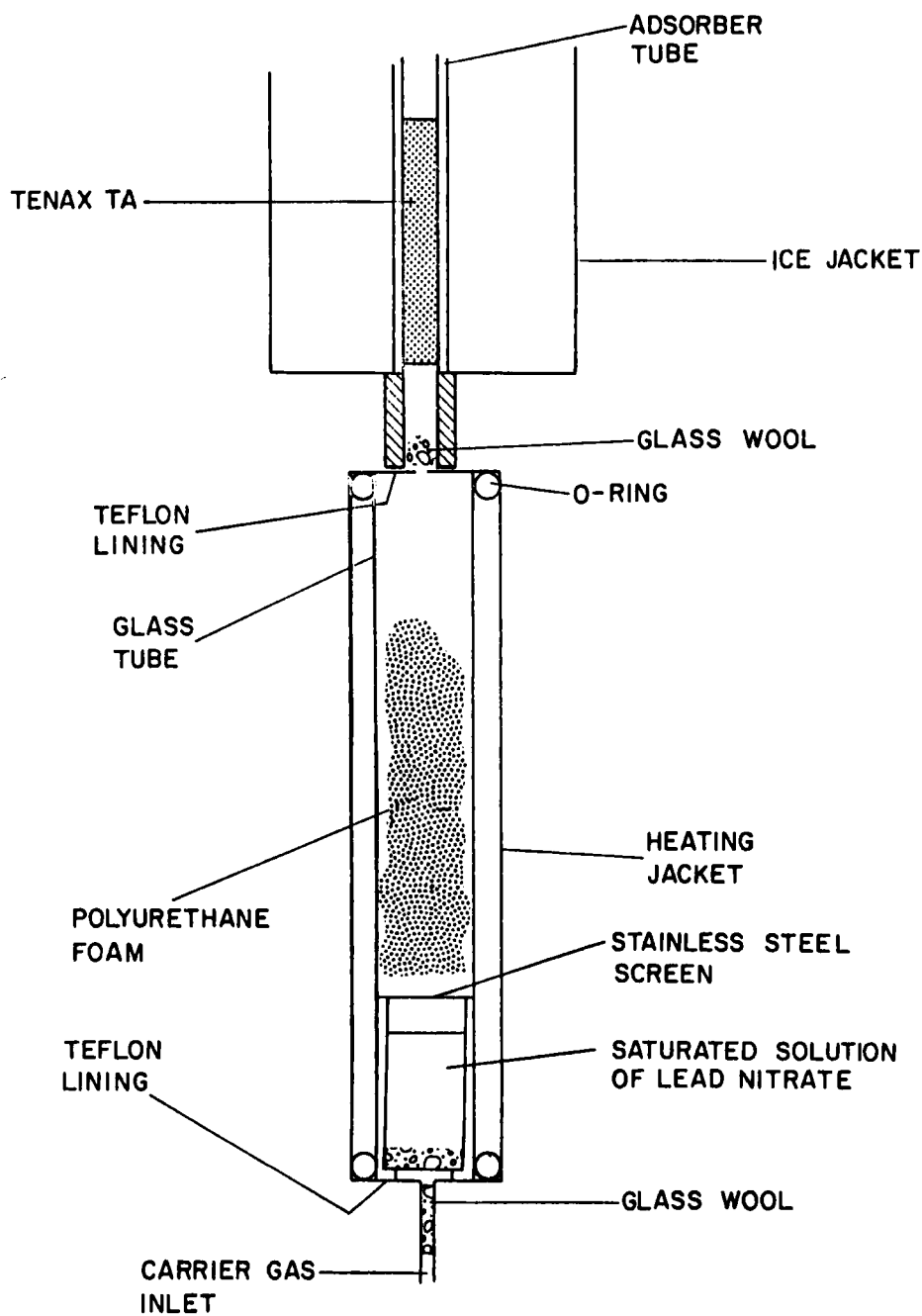


Figure 1 Dynamic headspace sampling apparatus.

Hewlett Packard 5790A. Both chromatographs were fitted with the dual adsorber desorption system described in ref. 10.

If the most volatile components of the off-gas mixture (e.g. freons, acetaldehyde) were not to be measured the Varian Vista GC was fitted with an SPB-5 30 m  $\times$  0.32 mm, 0.25  $\mu$ m film thickness column while the HP 5790A GC was fitted with DB-5 column of similar size and film thickness. Operating parameters for both GCs were as follows: Carrier gas: Helium (44 cm/sec at 30°) First adsorber: flow rate, 60 cm<sup>3</sup>/min; desorption temperature, 260°. Second adsorber: retrapping temperature,  $\leq -20^\circ$ ; desorption temperature,  $\sim 300^\circ$ . Oven temperature program:  $-20^\circ$  for 6 min,  $-20^\circ$  to  $0^\circ$  at  $20^\circ$ /min,  $0^\circ$  to  $80^\circ$  at  $2^\circ$ /min, and  $80^\circ$  held for 2 min.

Freons and compounds of similar volatility as well as other off-gases were separated on DB-1 30 m  $\times$  0.32 mm, 5  $\mu$ m film column with the following oven temperature program:  $-20^\circ$  for 6 min,  $-20^\circ$  to  $0^\circ$  to  $20^\circ$ /min,  $0^\circ$  to  $60^\circ$  at  $10^\circ$ /min,  $60^\circ$  to  $220^\circ$  at  $5^\circ$ /min, and  $220^\circ$  held for 5 min. The Varian GC operated with a flame ionization detector (FID) under optimal parametric conditions recommended by the manufacturer and at electrometer range setting of  $10^{-11}$  A/mV.

The HP 5790A GC was coupled to a HP 5970B Mass Selective Detector (MSD). The operating parameters of the MSD were set automatically in the Auto-Tune mode. Scan speed was 2.09 scans/sec from 15-220 m/z.

Effluents from each foam were first analysed on the Varian GC to determine the size of sample most suitable for the analysis. The FID also provided the supplementary information for peak identification. The effluents were later analysed on the HP 5790 GC-5970 MSD to obtain mass spectra of the eluted peaks. The unknown spectra were compared with the NBS spectral library. In some cases the Cornell University's spectral library was consulted. Standards of (tentatively) identified compounds were then analyzed on the HP 5790 GC-HP 5970 MSD and their spectra and retention times were recorded in a user generated library, which served as a basis for final confirmation of identity.

### *Foam Samples*

Four commercial sprayed-in-place foam insulation samples were collected at construction sites and delivered to the laboratory in 1 liter metal cans. The interiors of the cans had been sand blasted to remove the varnish coating, rinsed with distilled-in-glass hexane and acetone and heated for 24 hours in an oven at  $250^\circ\text{C}$ . A blank air sample from each can collected and analyzed in a similar manner as a foam effluent sample, did not indicate the presence of any organics. The cans were sealed with tight fitting metal lids.

The foam samples were labelled F-1, F-2, F-3 and F-4. Samples F-3 and F-4 were prepared by the same manufacturer from the same starting materials. The F-4 sample was foamed directly in the can whereas the other samples were cut from sprayed-in-place bulk insulation.

## RESULTS AND DISCUSSION

### *Sampling*

The emissions were determined at two temperatures, 40°C and 80°C, in dry and in humid (90% RH) air to demonstrate the effect of temperature and humidity on the off-gassing. The 80°C temperature was somewhat higher than the maximum value observed for a wall surface at the IRC (Institute for Research in Construction) Outdoor Test Building, located in Saskatoon.<sup>11</sup> A relative humidity of 90% has been observed in wall cavities of new buildings.<sup>12</sup>

The Tenax TA adsorber tube employed here trapped a sufficient quantity of off-gases from 250 ml (40°C) and 50 ml (80°C) samples to allow the separation and identification of about 70 individual compounds. Trapping efficiency for the lighter gases was enhanced by cooling the adsorber with an ice jacket. The cooling, however, caused excessive trapping of water in the humid air tests. The water droplets condensed on the wall of the adsorber just ahead of the Tenax packing were removed before analysis with a small piece of lint free paper tissue. The polar off-gases which dissolve in water were partly removed with this procedure; however, enough of them were trapped on the Tenax to allow their detection and identification. The water condensed on Tenax packing could not be removed and, when transferred to the GC column with the sample, affected retention times of acetaldehyde, dichlorofluoromethane, propanal and freon. The retention time of the less volatile components were only slightly shifted.

### *Chromatography and Peak Identification*

The off-gas mixture was separated initially on DB-5 and SPB-5 0.25 µm film columns. These columns afforded excellent separation of the components with boiling points above 60°C.

The chromatograms of the effluents emitted by F-3 foam sample at 40°C and 80°C separated on SPB-5 column are presented in Figure 2 and Figure 3. The peak indexes refer to compound numbers listed in the first column of Table 1.

For separation of the more volatile off-gases (boiling point below 60°C) a thick film DB-1 column was employed. This column not only separated the volatile components but at higher oven temperature it was suitable for separation of all components of the off-gas mixture. Moreover, the analysis time was the same (about 40 minutes) as with SPB-5 thin film column. The thick film DB-1 column is thus recommended for routine analysis of the off-gases emitted by polyurethane foam insulation.

Figures 4–6 present the examples of the separation of the effluents on the DB-1 column. The chromatograms obtained on the Varian GC equipped with FID as well as the total ion chromatograms obtained on HP GC with MSD demonstrate a pattern which might be regarded as characteristic of polyurethane foam off-gases. There is an early, moderate size peak of acetaldehyde followed by a cluster of four large peaks representing chlorofluorocarbons and propyl aldehyde. Then there are several small peaks with retention times between 12 and 20 min. At

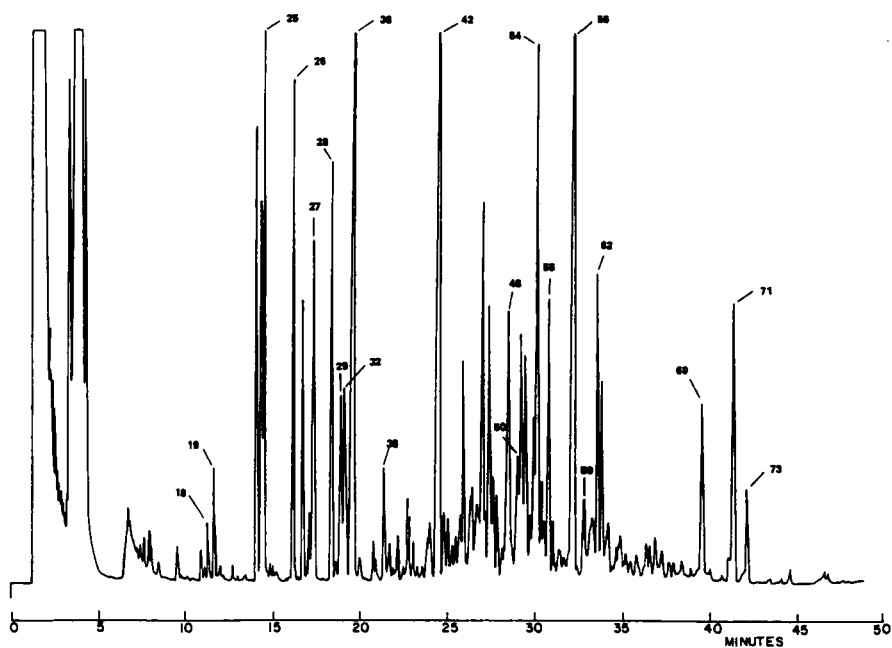


Figure 2 Effluents from foam 3. 40°C, 250 ml sample. SPB-5 column. FID.

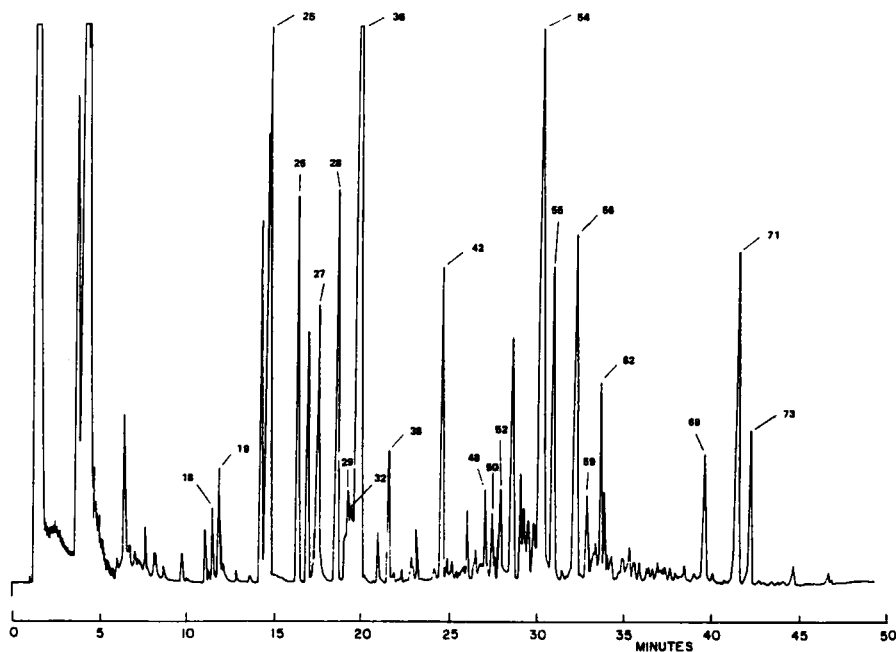


Figure 3 Effluents from foam 3. 80°C, 50 ml sample. SPB-5 column. FID.

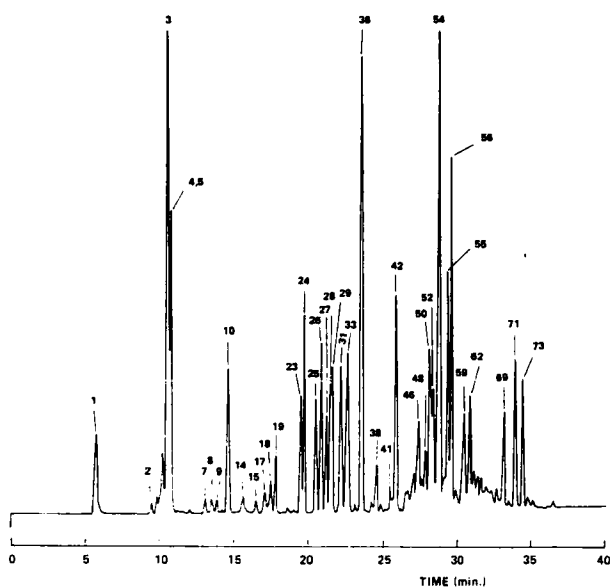


Figure 4 Effluents from foam F-3. 40 °C, 250 ml sample. DB-1 column. FID.

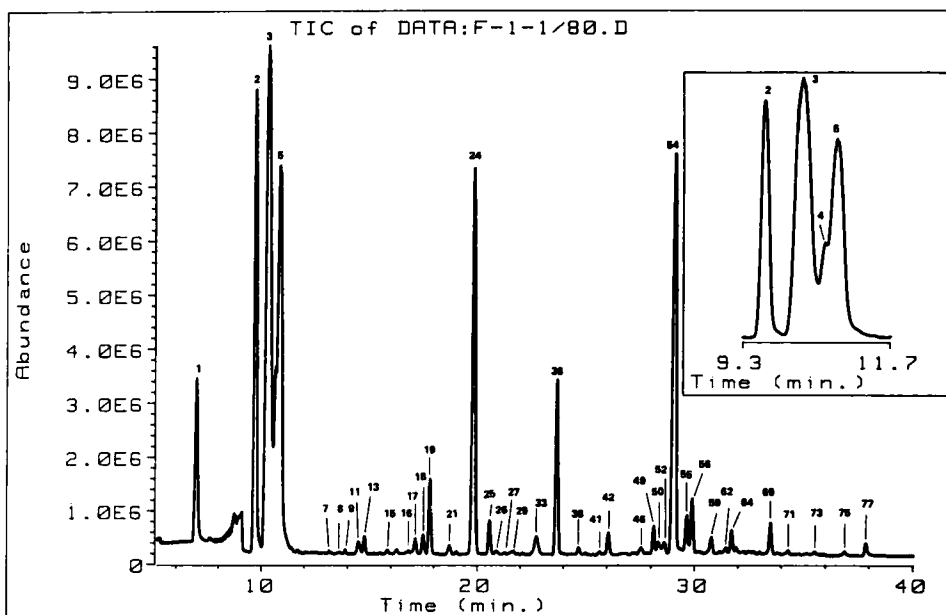


Figure 5 Effluents from foam F-1. 80 °C, 50 ml sample. DB-1 column. MSD.

about 20 minutes there is a moderate size peak of methyl-1,3-dioxane followed by a smaller toluene peak. At about 24 minutes there is a large peak of chlorobenzene and at 29 minutes a larger peak of  $\alpha$ -methyl styrene. There is a peak of 2-phenoxypropanol-1 at 38 minutes which is large in sample F-2, small in sample F-1 and absent in samples F-3 and F-4.

The pattern can be easily observed in Figure 7 where chromatograms of the effluents from the four foam samples are presented on the same time and abundance scale. The early large peaks on the bottom chromatogram (F-4-1-80. D) are somewhat delayed, presumably due to the effect of the residual moisture left in the foam from humid air testing. These peaks have similar retention times to those in humid air tests (cf. Figure 8).

The effect of humidity on off-gassing is demonstrated in Figure 8 where chromatograms of the effluents from foam sample emitted at 80°C in dry and humid air and presented side by side on the same time and abundance scales. The chromatograms of effluents emitted in dry air and in 90% RH seem to be different. Although there are essentially the same peaks present on both chromatograms (with few exceptions), many of them are larger in the 90% RH case. However, it may not follow that the emission rates in 90% RH are significantly higher.

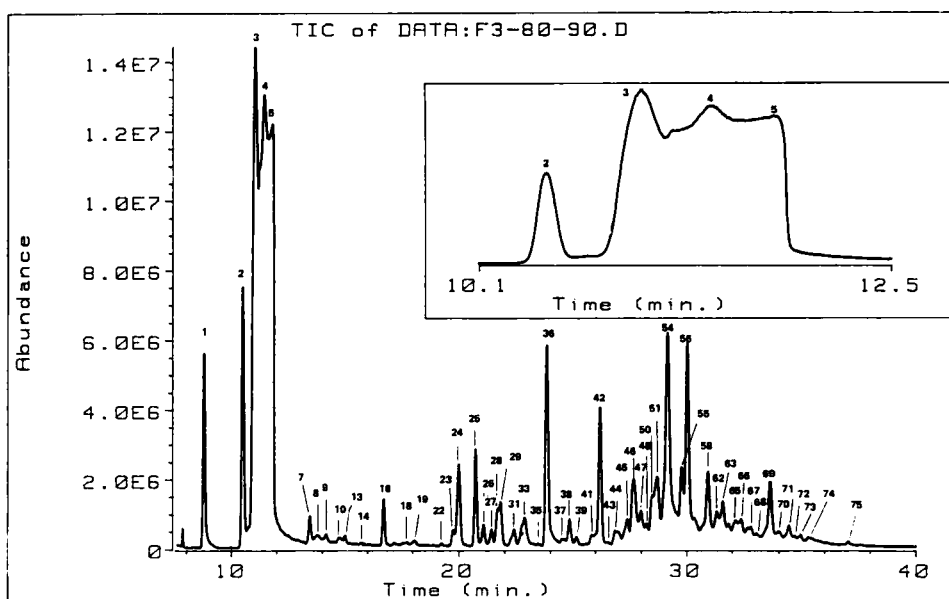
The smaller peaks of emissions observed in dry air may be the result of the sampling sequence rather than the effect of humidity. In conducting the experiments the foams were first sampled in 90% RH, then the vessel was purged with dry air at 80° to dry the foams. The drying was followed by a 24 hour "recharging" period during which the off-gases were allowed to accumulate in the head space of the sealed vessel. It is possible that the purging might have partially depleted the source, hence the smaller peaks.

Table 1 list compounds identified as the off-gases. Only few of them (the major peaks) were detected in all four samples at 40° and 80° as well as in dry and humid air. Many of them were not detected at 40° because their headspace concentration at this temperature was below detection limit. There are, however, several peaks which are characteristic of a particular sample. 2,2'-Bi-1,3-dioxolane and 2-methyl furan are present only in samples F-3 and F-4, which as mentioned before are chemically identical, but F-4 was foamed directly in a tin. N,N,N',N'-Tetramethyl-1,3-propanediamine, 1,2-dichloro-1-fluoroethane and 2,4-dimethyloxethane were detected only in samples F-1 and F-2 at 80° in dry air and N-methyloxymethanamine was found in the same foams in humid air at 80° only.

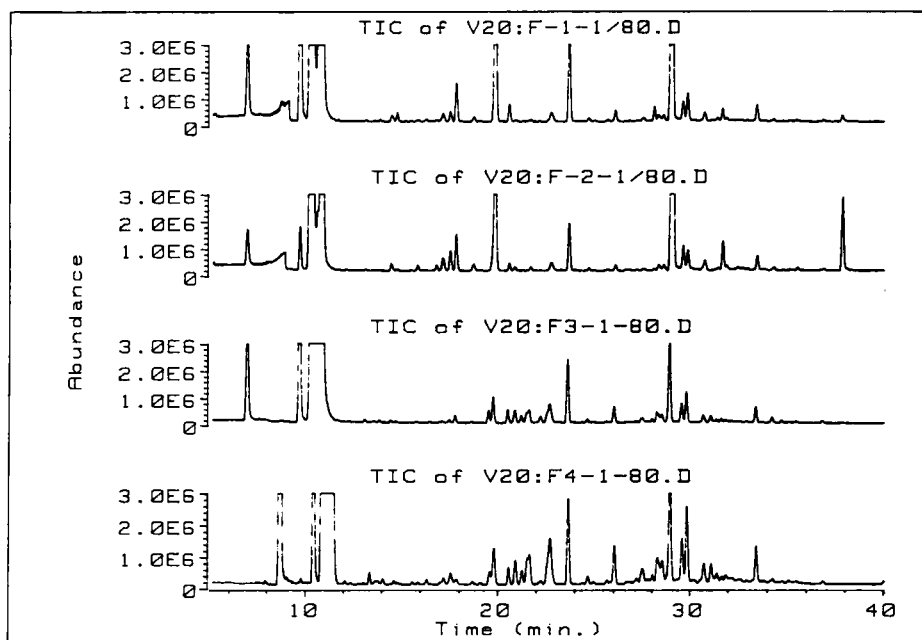
#### *Estimate of the Head-space Concentration of Selected Off-gases*

Although the quantitative analysis was not an objective of this project, a sufficient amount of data was acquired to attempt at least a semi-quantitative estimate of the headspace concentration of selected off-gases which might be of particular interest to a toxicologist. The amounts of the compounds were determined by an external standard method and recalculated to concentrations in the headspace. The results of analysis are presented in Tables 2, 3 and 4.

The results are estimates only primarily because the breakthrough volume of the Tenax TA adsorber was not determined. It could be expected that more volatile



**Figure 6** Effluents from foam F-3. 80°C, 90% RH. 50ml samples. DB-1 column. MSD.



**Figure 7** Chromatograms of the effluents from foams F-1, F-2, F-3, F-4 emitted at 80°C in dry air. 50ml samples.

**Table 1** Compounds identified as off-gases of rigid polyurethane foam

| No. | Off-gas                                     | F-1 |    |    |                   | F-2 |    |    |      | F-3 |    |    |    | F-4 |    |    |    |
|-----|---|-----|----|----|-------------------|-----|----|----|------|-----|----|----|----|-----|----|----|----|
|     |   | 40  | 80 | 40 | 80                | 40  | 80 | 40 | 80   | 40  | 80 | 40 | 80 | 40  | 80 | 40 | 80 |
| 1   | Acetaldehyde <sup>a</sup>                   | +   | +  | +  | n.a. <sup>b</sup> | +   | +  | +  | n.a. | +   | +  | +  | +  | +   | +  | +  | +  |
| 2   | Dichlorofluoromethane <sup>a</sup>          | +   | +  | +  | n.a.              | +   | +  | +  | n.a. | +   | +  | +  | +  | +   | +  | +  | +  |
| 3   | Trichlorofluoromethane <sup>a</sup>         | +   | +  | +  | n.a.              | +   | +  | +  | n.a. | +   | +  | +  | +  | +   | +  | +  | +  |
| 4   | Propanal                                    |     |    | +  | n.a.              |     | +  | +  | n.a. |     | +  |    |    |     | +  |    | +  |
| 5   | Bromodichlorofluoromethane                  | +   | +  | +  | n.a.              | +   | +  | +  | n.a. | +   | +  | +  |    | +   | +  | +  | +  |
| 6   | N-Methoxymethanamine                        |     |    |    | +                 |     |    |    | +    |     |    |    |    |     |    |    |    |
| 7   | 2-Methyl-2-propenal                         |     | +  | +  | +                 |     | +  | +  | +    | +   | +  |    | +  | +   | +  | +  | +  |
| 8   | Trimethyl silanol                           |     | +  | +  | +                 |     | +  | +  | +    |     | +  |    | +  |     | +  |    | +  |
| 9   | 2-Butanone <sup>a</sup>                     |     | +  |    | +                 |     | +  |    | +    |     | +  |    | +  |     | +  |    | +  |
| 10  | 2-Methyl furan                              |     |    |    |                   |     |    |    |      |     | +  |    | +  |     | +  |    | +  |
| 11  | N,N,N',N'-Tetramethyl-1,3-propanediamine    |     | +  |    |                   |     | +  |    |      |     |    |    |    |     |    |    |    |
| 12  | 1,2-Dichloro-1-fluoroethane                 |     | +  |    |                   |     |    | +  |      |     |    |    |    |     |    |    |    |
| 13  | Chloroform <sup>a,c</sup>                   |     | +  | +  | +                 |     | +  |    | +    | +   | +  |    | +  | +   | +  |    | +  |
| 14  | 2-Butenal <sup>c</sup>                      |     |    |    | +                 |     |    |    | +    |     |    |    | +  |     | +  | +  | +  |
| 15  | 2-Methyl-1,3-dioxolane <sup>a,c</sup>       |     | +  |    |                   |     | +  |    |      |     | +  |    |    |     | +  |    |    |
| 16  | Dimethoxypropane                            |     | +  |    | +                 |     | +  |    | +    |     |    | +  | +  |     |    | +  | +  |
| 17  | Dimethyl-1,3-dioxolane <sup>a,c</sup>       |     | +  | +  | +                 |     | +  | +  | +    | +   | +  |    |    | +   | +  | +  |    |
| 18  | 2,2-Dimethyl-1,3-dioxolane <sup>c</sup>     |     | +  |    | +                 |     | +  |    | +    | +   | +  | +  |    | +   | +  |    | +  |
| 19  | 1,4-Dioxane <sup>a,c</sup>                  |     | +  |    | +                 |     | +  |    | +    | +   | +  | +  | +  | +   | +  |    | +  |
| 20  | 2,2,4-Trimethyl-1,3-dioxolane               |     |    |    | +                 |     |    |    | +    |     |    |    |    |     | +  |    |    |
| 21  | 2,4-Dimethyl oxethane                       |     | +  |    |                   |     | +  |    |      |     |    |    |    |     |    |    |    |
| 22  | 2-Methyl-2-butenal                          |     |    |    | +                 |     |    |    | +    |     |    |    | +  |     | +  |    | +  |
| 23  | 2,2'-Bi-1,3-dioxolane                       |     |    |    |                   |     |    |    |      | +   | +  | +  | +  | +   | +  | +  | +  |
| 24  | Methyl-1,3-dioxane                          | +   | +  | +  | +                 | +   | +  | +  | +    | +   | +  | +  | +  | +   | +  | +  | +  |
| 25  | Toluene <sup>a,c</sup>                      |     | +  |    | +                 | +   | +  |    | +    | +   | +  | +  | +  | +   | +  | +  | +  |
| 26  | 2-Ethyl-4-methyl-1,3-dioxolane <sup>c</sup> |     | +  |    |                   |     | +  |    |      | +   | +  |    | +  | +   | +  | +  | +  |

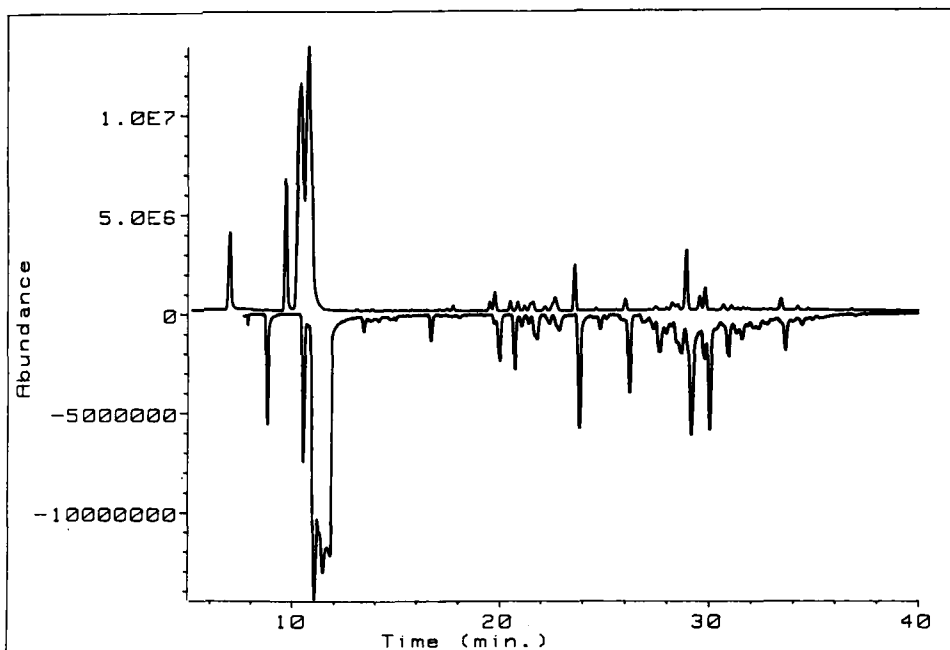
**Table 1** (continued)

| No. | Off-gas                                     | F-1 |    |    |    | F-2 |    |    |    | F-3 |    |    |    | F-4 |    |    |    |
|-----|---|-----|----|----|----|-----|----|----|----|-----|----|----|----|-----|----|----|----|
|     |   | 40  |    | 80 |    | 40  |    | 80 |    | 40  |    | 80 |    | 40  |    | 80 |    |
|     |   | 40  | 80 | 90 | 90 | 40  | 80 | 90 | 90 | 40  | 80 | 90 | 90 | 40  | 80 | 90 | 90 |
| 27  | 2-Ethyl-2-methyl-1,3-dioxolane <sup>c</sup> |     | +  |    | +  |     | +  |    |    | +   | +  |    | +  | +   | +  | +  | +  |
| 28  | Dimethyl-1,4-dioxane <sup>c</sup>           |     |    |    |    |     |    |    |    | +   | +  |    | +  | +   |    |    | +  |
| 29  | Dimethyl-1,4-dioxane <sup>c</sup>           |     | +  |    |    |     | +  |    |    | +   | +  |    | +  | +   |    |    | +  |
| 30  | O-Hexyl-hydroxylamine                       |     |    |    | +  |     |    |    | +  |     |    |    |    |     |    |    | +  |
| 31  | 4-Methyl-2,3-pentanedione                   |     |    |    |    |     |    |    |    | +   | +  |    | +  | +   | +  | +  | +  |
| 32  | 2,5-Dimethyl-1,4-dioxane <sup>c</sup>       | +   |    |    |    | +   |    |    |    | +   | +  |    |    | +   | +  |    |    |
| 33  | Hexamethyl-cyclotrisiloxane <sup>c</sup>    |     | +  | +  | +  |     | +  | +  | +  |     | +  | +  | +  |     | +  | +  | +  |
| 35  | 2,6-Dimethylheptane <sup>c</sup>            |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 36  | Chlorobenzene <sup>a,c</sup>                | +   | +  | +  | +  | +   | +  | +  | +  | +   | +  | +  | +  | +   | +  | +  | +  |
| 37  | 7-Methyl-1-undecene                         |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 38  | <i>p</i> -Xylene <sup>a,c</sup>             |     | +  |    | +  |     | +  |    | +  | +   | +  |    | +  | +   |    |    | +  |
| 39  | 3-Methyl octane                             |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 40  | Propyl cyclopentane                         |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 41  | <i>m</i> -Xylene <sup>a,c</sup>             |     | +  |    |    |     | +  |    |    | +   | +  |    |    | +   | +  |    |    |
| 42  | <i>n</i> -Nonane <sup>a,c</sup>             |     | +  |    | +  |     | +  | +  |    | +   | +  |    | +  | +   | +  | +  |    |
| 43  | Butyl cyclopentane                          |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 44  | 4-Ethyl-1-methyl-cyclohexane                |     |    |    | +  |     |    |    | +  | +   |    |    | +  | +   |    |    | +  |
| 45  | 1-Propenyl-cyclohexane                      |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 46  | Propyl cyclohexane                          |     | +  |    | +  |     | +  |    | +  | +   | +  |    | +  | +   | +  | +  | +  |
| 47  | 4-(1-Methylethyl)-heptane                   |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    | +  | +  |
| 48  | Propyl benzene <sup>a,c</sup>               |     |    |    | +  |     |    |    | +  | +   | +  |    | +  | +   |    |    |    |
| 49  | Phenol                                      |     | +  |    |    |     |    | +  |    |     |    |    |    |     |    |    |    |
| 50  | Ethyl Toluene <sup>c</sup>                  |     | +  |    | +  |     | +  |    | +  | +   | +  |    | +  | +   | +  | +  | +  |
| 51  | 2,5-Dimethyl-octane                         |     |    |    | +  |     |    |    | +  |     |    |    |    | +   |    |    | +  |
| 52  | Mesitylene                                  |     | +  |    |    |     | +  |    |    | +   | +  |    |    |     | +  | +  |    |

**Table 1** (continued)

| No. | Off-gas                                 | F-1 |    |    |    | F-2 |    |    |    | F-3 |    |    |    | F-4 |    |    |    |
|-----|---|-----|----|----|----|-----|----|----|----|-----|----|----|----|-----|----|----|----|
|     |   | 40  | 80 | 40 | 80 | 40  | 80 | 40 | 80 | 40  | 80 | 40 | 80 | 40  | 80 | 40 | 80 |
| 53  | 4-Methyl-2-propylpentanol-1             |     |    |    |    |     |    |    |    |     |    |    |    |     |    |    | +  |
| 54  | $\alpha$ -Methyl styrene <sup>a,c</sup> | +   | +  | +  | +  | +   | +  | +  | +  | +   | +  | +  | +  | +   | +  | +  | +  |
| 55  | Pseudocumene <sup>a,c</sup>             |     | +  |    | +  |     | +  |    | +  |     | +  |    | +  |     | +  |    | +  |
| 56  | <i>n</i> -Decane <sup>a,c</sup>         |     | +  |    | +  |     | +  |    | +  |     | +  |    | +  |     | +  |    | +  |
| 58  | 2,6-Dimethyl-nonane                     |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 59  | Hemimellitene <sup>a</sup>              |     | +  |    |    |     | +  |    |    |     | +  | +  | +  |     | +  | +  | +  |
| 62  | Limonene <sup>c</sup>                   |     | +  |    |    |     |    |    |    |     | +  | +  |    |     | +  |    | +  |
| 63  | 2-Methylpropyl-cyclohexene              |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 64  | Acetophenone <sup>c</sup>               |     | +  |    |    |     | +  |    |    |     | +  |    |    |     | +  |    |    |
| 65  | 2,5-Dimethyl-nonane                     |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 66  | Pentyl heptane                          |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 67  | Decaline                                |     |    |    | +  |     |    |    | +  |     | +  |    | +  |     |    |    | +  |
| 68  | 1,2-Dibutyl-cyclopentane                |     |    |    | +  |     |    |    | +  |     | +  |    | +  |     |    |    | +  |
| 69  | <i>n</i> -Undecane <sup>a,c</sup>       |     | +  |    | +  |     | +  |    | +  |     | +  | +  | +  |     | +  | +  | +  |
| 70  | 2,2-Dimethylcyclopentyl-cyclohexane     |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 71  | Alloocimene-2 <sup>c</sup>              |     | +  |    | +  |     | +  |    |    |     | +  | +  |    |     | +  |    | +  |
| 72  | Dimethyl decane                         |     |    |    | +  |     |    |    | +  |     |    |    | +  |     |    |    | +  |
| 73  | Alloocimene <sup>c</sup>                |     |    |    |    |     | +  |    |    |     | +  | +  |    |     | +  |    | +  |
| 74  | Hexyl cyclohexane                       |     |    |    | +  |     |    |    |    |     | +  |    | +  |     | +  |    | +  |
| 75  | <i>n</i> -Dodecane                      |     | +  |    | +  |     | +  |    | +  |     | +  |    | +  |     | +  |    | +  |
| 76  | 2-Phenoxy-propanol-1                    |     | +  |    |    |     | +  |    |    |     |    |    |    |     |    |    |    |

<sup>a</sup>Identity of the compounds confirmed by comparison of their retention times and mass spectra with those of the standards.<sup>b</sup>n.a. - not analyzed.<sup>c</sup>Compounds identified by both Cornell's and NBS spectral libraries.



**Figure 8** Chromatograms of the emissions from foam at F-3 at 80°C in dry air (top) and 90% RH (bottom). 50 ml samples.

**Table 2** Head-space concentrations of the selected off-gases emitted at 80° in dry air

| No. | Off-gas                    | F-1               | F-2               | F-3               | F-4               |
|-----|----------------------------|-------------------|-------------------|-------------------|-------------------|
|     |                            | mg/m <sup>3</sup> | mg/m <sup>3</sup> | mg/m <sup>3</sup> | mg/m <sup>3</sup> |
| 1   | Acetaldehyde               | 105               | 51                | 123               | 367               |
| 3   | Trichlorofluoromethane     | 256               | 235               | 322               | 326               |
| 9   | 2-Butanone                 | 1                 | 0.3               | 1                 | 2                 |
| 13  | Chloroform                 | 4                 | 1                 | 1                 | 1                 |
| 17  | Dimethyl-1,3-dioxolane     | 3                 | 4                 | 1                 | 3                 |
| 18  | 2,2-Dimethyl-1,3-dioxolane | 3                 | 6                 | 1                 | 4                 |
| 19  | 1,4-Dioxane                | 16                | 15                | 3                 | 3                 |
| 24  | Methyl-1,3-dioxane         | 61                | 50                | 7                 | 11                |
| 25  | Toluene                    | 4                 | 2                 | 3                 | 4                 |
| 36  | Chlorobenzene              | 35                | 18                | 25                | 29                |
| 38  | <i>p</i> -Xylene           | 0.5               | 0.5               | 0.5               | 1                 |
| 41  | <i>m</i> -Xylene           | 0.3               | 0.5               | 0.2               | 1                 |
| 42  | <i>n</i> -Nonane           | 3                 | 2                 | 4                 | 8                 |
| 48  | Propyl benzene             | n.d. <sup>a</sup> | n.d. <sup>a</sup> | 1                 | 2                 |
| 50  | Ethyl toluene <sup>b</sup> | 2                 | 2                 | 4                 | 9                 |
| 52  | Mesitylene                 | 2                 | 2                 | 2                 | 6                 |
| 54  | $\alpha$ -Methyl styrene   | 93                | 114               | 27                | 35                |
| 55  | Pseudocumene               | 6                 | 7                 | 5                 | 11                |
| 56  | <i>n</i> -Decane           | 4                 | 3                 | 4                 | 8                 |

<sup>a</sup>n.d., not detected.

<sup>b</sup>Quantitation based on the response factor of propyl benzene.

**Table 3** Head-space concentrations of the selected off-gases emitted at 40° in 90% RH

| No. | Off-gas                    | F-1               | F-2               | F-3               | F-4               |
|-----|----------------------------|-------------------|-------------------|-------------------|-------------------|
|     |                            | mg/m <sup>3</sup> | mg/m <sup>3</sup> | mg/m <sup>3</sup> | mg/m <sup>3</sup> |
| 1   | Acetaldehyde               | 4                 | 1                 | 9                 | 56                |
| 3   | Trichlorofluoromethane     | 12                | 3                 | n.a. <sup>a</sup> | 56                |
| 13  | Chloroform                 | 0.2               | <0.1              | n.d. <sup>a</sup> | n.d.              |
| 17  | Dimethyl-1,3-dioxolane     | 0.2               | 0.2               | n.d.              | 0.1               |
| 18  | 2,2-Dimethyl-1,3-dioxolane | n.d.              | n.d.              | 0.3               | n.d.              |
| 19  | 1,4-Dioxane                | n.d.              | n.d.              | 1                 | n.d.              |
| 24  | Methyl-1,3-dioxane         | 0.2               | 0.3               | 3                 | 1                 |
| 25  | Toluene                    | n.d.              | n.d.              | 0.04              | 0.5               |
| 36  | Chlorobenzene              | 0.1               | 0.2               | 0.3               | 2                 |
| 42  | <i>n</i> -Nonane           | n.d.              | 0.04              | n.d.              | 2                 |
| 50  | Ethyl toluene <sup>b</sup> | n.d.              | n.d.              | n.d.              | 1                 |
| 52  | Mesitylene                 | n.d.              | n.d.              | n.d.              | 1                 |
| 54  | $\alpha$ -Methyl styrene   | 1                 | 1                 | 1                 | 3                 |
| 56  | <i>n</i> -Decane           | n.d.              | n.d.              | 0.06              | 1                 |

<sup>a</sup>n.a.—not analysed, n.d.—not detected.<sup>b</sup>Quantitation based the response factor of propyl benzene.**Table 4** Head-space concentrations of the selected off-gases emitted at 80° in 90% RH

| No. | Off-gas                    | F-1               | F-2               | F-3               | F-4               |
|-----|----------------------------|-------------------|-------------------|-------------------|-------------------|
|     |                            | mg/m <sup>3</sup> | mg/m <sup>3</sup> | mg/m <sup>3</sup> | mg/m <sup>3</sup> |
| 1   | Acetaldehyde               | n.a. <sup>a</sup> | n.a.              | 144               | 225               |
| 3   | Trichlorofluoromethane     | n.a.              | n.a.              | 352               | 392               |
| 9   | 2-Butanone                 | 2                 | 2                 | 4                 | 2                 |
| 13  | Chloroform                 | 17                | 19                | 3                 | 4                 |
| 17  | Dimethyl-1,3-dioxolane     | 1                 | 2                 | n.d. <sup>a</sup> | n.d.              |
| 18  | 2,2-Dimethyl-1,3-dioxolane | 2                 | 4                 | 1                 | 3                 |
| 19  | 1,4-Dioxane                | 10                | 12                | 3                 | 6                 |
| 24  | Methyl-1,3-dioxane         | 120               | 123               | 24                | 29                |
| 25  | Toluene                    | 24                | 28                | 19                | 26                |
| 36  | Chlorobenzene              | 113               | 57                | 79                | 87                |
| 38  | <i>p</i> -Xylene           | 5                 | 9                 | 3                 | 7                 |
| 41  | <i>m</i> -Xylene           | 2                 | n.d.              | 1                 | n.d.              |
| 42  | <i>n</i> -Nonane           | 34                | 75                | 29                | 110               |
| 48  | Propyl benzene             | 7                 | 13                | 6                 | n.d.              |
| 50  | Ethyl toluene <sup>b</sup> | 13                | 29                | 11                | 46                |
| 54  | $\alpha$ -Methyl styrene   | 181               | 242               | 109               | 125               |
| 55  | Pseudocumene               | 27                | 44                | 21                | 51                |
| 56  | <i>n</i> -Decane           | 30                | 65                | 27                | 67                |

<sup>a</sup>n.a.—not analysed, n.d.—not detected.<sup>b</sup>Quantitation based on the response factor of propyl benzene.

components of the off-gas mixture such as acetaldehyde, trichlorofluoromethane, butanone, chloroform and perhaps even toluene were not quantitatively trapped. Moreover, polar compounds were partly removed from the adsorber with the water condensed during humid air sampling. The concentrations of these compounds, listed in Tables 2, 3 and 4, may therefore be low.

### *Acknowledgement*

The project was in part financially supported by Canada Mortgage and Housing Corporation.

### *References*

1. J. R. Ward and L. J. Decker, *Ind. Eng. Chem. Prod. Res. Dev.* **21**, 460 (1982).
2. D. H. Napier and T. W. Wong, *Br. Polym. J.* **4**, 45 (1972).
3. W. D. Woolley, *Br. Polym. J.* **4**, 27-43 (1972).
4. W. D. Woolley, *Fire Flam. J./Comb. Toxicol.* **1**, 259 (1974).
5. J. Chambers and C. B. Reese, *Br. Polym. J.* **8**, 48 (1976).
6. J. Trzeczczynski and D. Wlodarczak, *Polimery*. **28**, 17 (1983).
7. J. Hetper., C. Lotacha and K. Weber, *Polimery*. **25**, 49 (1980).
8. J. Simon, F. Barla, A. Kelemen-Haller, F. Farkas and M. Kraxner, *Chromatographia*. **25**, 99 (1988).
9. M. E. Krzymien, *Am. Ind. Hyg. Assoc. J.* **48** (1), 67 (1987).
10. M. E. Krzymien, *Intern. J. Environ. Anal. Chem.* **21**, 43 (1985).
11. C. P. Hedlin, Institute for Research in Construction, Prairie Regional Station; Private communication.
12. D. M. Onysko, Forintek Canada Corp.; Private communication.