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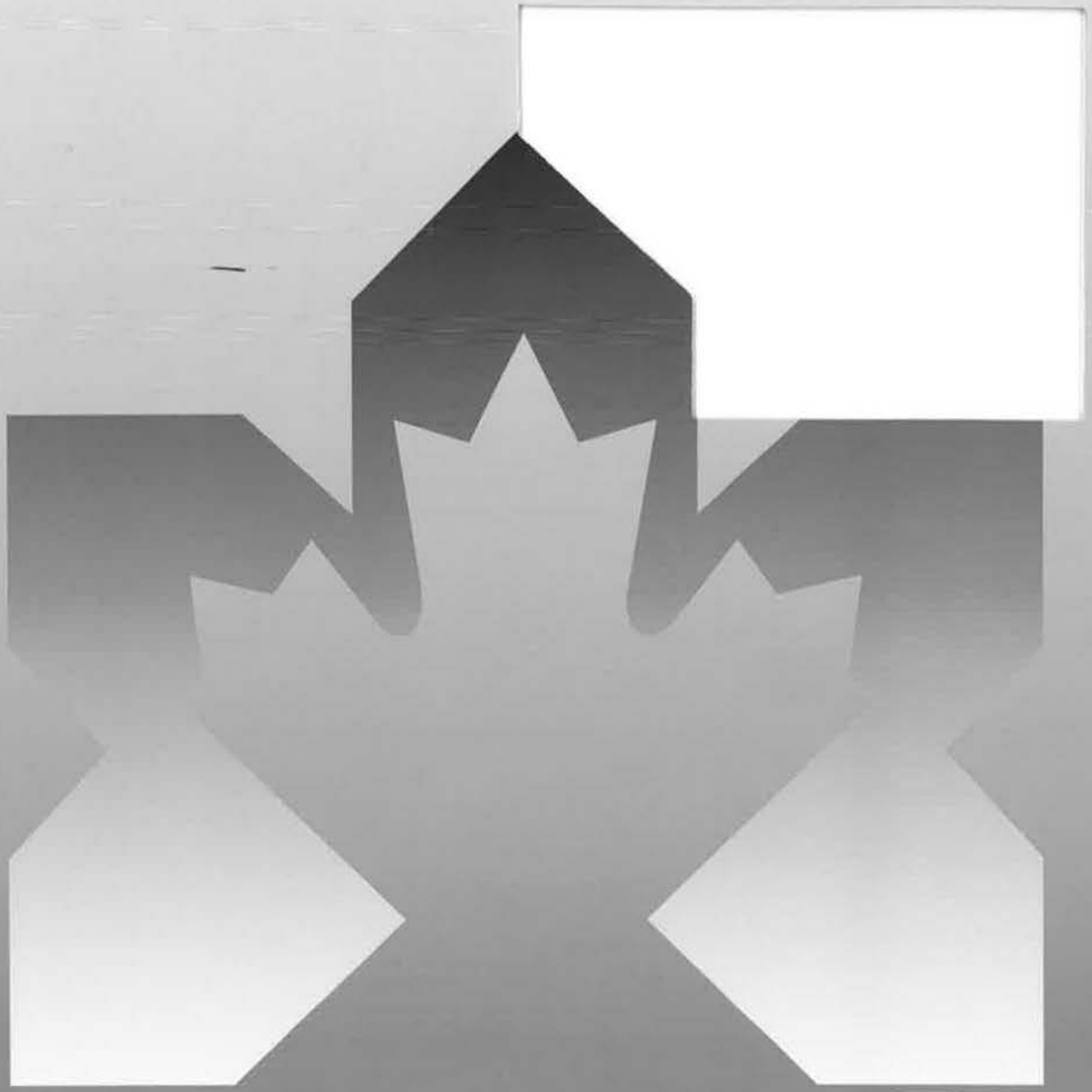

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PASSIVE MONITORING OF VOC IN AIR USING ACC

By H.D. Gesser

August 1997

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FINAL REPORT

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Passive Monitoring of VOC in Air Using ACC

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EXECUTIVE SUMMARY

Passive Monitoring of VOC in Air Using Activated Carbon Cloth

This project dealt with developing the method of using activated carbon cloth as a sampler for measuring volatile organic compounds (VOC's) in air. Strips of carbon cloth mounted in slide holders were tested as diffusive samplers. These were exposed to known concentrations of standard chemicals in test chambers. The adsorbed chemicals were extracted with solvents and analyzed. The tests showed that relative humidity has some effect on adsorption, and carbon cloths from different manufacturers showed some variation in their performance.

The method of storage of the samplers can lead to some losses in concentration. Aluminium foil was found to be superior to polyethylene bags in preventing diffusion of gases from the carbon cloth.

Two methods of extraction - using a syringe or an ultrasonic technique were compared. Ultrasonic extraction was found to be superior to using a syringe for extraction.

It appeared that the cloths had a different response rate to chemicals of varying molecular weight. Molecules with higher molecular weight are preferentially adsorbed compared to those with lower molecular weight. A correction factor may be necessary.

Comparison between the activated carbon cloths and 3M passive samplers gave smaller sampling rates for the carbon cloth. The difference may be due to the effect of humidity. It would appear that a hydrophobic carbon cloth would be preferable.

Field testing using these activated carbon cloths in houses remains to be done. The potential for using these samplers for semi-quantitative analysis also remains.

RÉSUMÉ

Contrôle passif de la teneur de l'air en COV à l'aide d'un tissu à charbon actif

La présente recherche visait à mettre au point une méthode permettant d'utiliser un tissu à charbon actif comme échantillonneur en vue de mesurer la teneur de l'air en composés organiques volatils (COV). Des bandes de tissu carboné montées sur des supports coulissants ont été testés à titre d'échantillonneurs de diffusion. Elles ont été exposées à des concentrations connues de substances chimiques courantes dans des chambres d'essais. Les substances chimiques adsorbées ont été extraites avec des solvants, puis analysées. Les tests ont révélé que l'humidité relative exerçait un certain effet sur l'adsorption, et que les tissus carbonés en provenance de différents fabricants variaient quelque peu par leur performance.

La méthode d'entreposage des échantillonneurs peut entraîner certaines pertes de concentration. La feuille d'aluminium, a-t-on constaté, prévenait mieux la diffusion des gaz émanant du tissu carboné que le sac de polyéthylène.

Deux méthodes d'extraction, soit par seringue, soit par technique ultrasonique, ont fait l'objet d'une comparaison. Le mode d'extraction ultrasonique s'est révélé supérieur à l'autre moyen.

Il semble que les tissus enregistraient un taux de réaction différent aux substances chimiques de poids moléculaire différent. Les molécules affichant un poids moléculaire plus élevé sont l'objet d'une adsorption privilégiée comparativement à celles qui affichent un poids moléculaire plus faible. Un facteur de correction pourrait s'imposer.

La comparaison entre les tissus à charbon actif et les échantillonneurs passifs 3M ont donné des taux d'échantillonnage moindres pour le tissu carboné. La différence peut être attribuable à l'effet de l'humidité. Il semble que le tissu à charbon hydrophobe serait préférable.

Il reste à effectuer des essais avec ces tissus de charbon actif dans des maisons, tout comme à établir la possibilité d'utiliser ces échantillons pour fins d'analyse semi-quantitative.

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ABSTRACT

Passive Monitoring of VOC in Air Using Activated Carbon Cloth

This project dealt with developing the method of using activated carbon cloth mounted in slide holders as a sampler for measuring volatile organic compounds (VOC's) in air. Preliminary tests were carried out to determine the effects of relative humidity, methods of extraction, materials for storing the cloth strips and the type of carbon cloth. Compared to the 3M passive samplers, the activated carbon cloth samplers gave lower concentrations, presumably due to the effect of humidity. Further work will require finding a hydrophobic type of carbon cloth, determining the response of the sampler to various chemicals and field testing in houses.

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1 INTRODUCTION:

Activated carbon cloth (ACC) can be used as a diffusive sampler for the quantitative and qualitative analysis of volatile organic compounds (VOC). To investigate the ability of the cloth to passively sample VOC, a controlled environment was needed. This was accomplished by converting an aquarium into a test chamber with a steady concentration of VOC. As test compounds we used benzene, toluene, ethyl benzene, p-xylene, m-xylene, o-xylene, or as more commonly known, BTEX. Once the retention times of BTEX on the gas chromatograph column were known, the analysis became one of a quantitative nature only. To determine the unknown amount of BTEX on the cloth by GC, the following formula was used:

$$f = \frac{A_x}{A_{std}} x \frac{D_{std}}{D_x} \qquad \text{Equation 1}$$

where A_{std} is the area of the internal standard (*sec*-butyl benzene), D_{std} is the amount of internal standard added, A_x is the area of the BTEX compound, D_x is the amount of the desired BTEX, f is the response factor of the flame ionization detector on the GC for compound x.

Rearranging to solve for D_x :

$$D_x = \frac{A_x}{A_{std}} x \frac{D_{std}}{f} \qquad \text{Equation 2}$$

It should be noted that the response factor in the above equation was used throughout, however the comparisons made between different samplers in the same experiments did not depend on knowing the exact quantitation, as only the amounts relative to each other are important. The importance of the response factor becomes apparent when inter-experimental results are compared. A typical G.C. trace of BTEX is shown in Figure 1.

1.1 Objectives:

The following objectives are outlined, although not all listed were accomplished. Alternatively some important factors were discovered and investigated that are not in the objectives but were deemed important in development of the passive monitor. The objectives are outlined as follows:

a) -A comparison of 6-8 ACC samples for their adsorptive characteristics for BTEX using internal standards and CS_2 extraction of the exposed ACC. Sampling rates of the ACC will be established for these substances at different relative humidities (RH) to determine which ACC is least affected.

b) -A comparison will be made between the two best ACC and 3M passive samplers and SKC active samplers to establish sampling rates for the ACC and effect of relative humidity.

c) -The desorption or off-gassing of the pollutants from the ACC will be tested to determine the extent of loss if any.

d) -The selection of test homes and buildings will be made on the basis of their presumed contamination and the level of discomfort.

e) -The thermal desorption of the ACC samplers will be tested with samplers of various sizes so as not to overload the column of the gas chromatograph. Samplers using a 35 mm slide holders will be used initially and compared with solvent extracted samplers to which an internal standard has been added. This would make quantitative analysis possible for a thermally desorbed sample. Thermal desorption can be effected by electrical, microwave, RF heating, The concentration of the adsorbed gases on the column may facilitate the analysis process.

f) -The ACC will be used to establish the profile of a building suffering from indoor air problems and which may be designated as a "sick" building. From this profile it is believed that it may be possible to establish the source of the problem and to determine the source of the contamination.

g) -The final testing protocol will be evaluated and compared to active sampling with SKC tubes and with commercially available passive samplers by 3M and SKC with corrections made for temperature and humidity.

h) -Field testing will be conducted throughout the program. It is anticipated that 100 homes and at least one "sick" building will be profiled.

i) -Compare the ability of polyurethane foams (PUF) to ACC for high humidity adsorption of heavy molecules. It is believed that PUF is not humidity-dependent and therefore the insecticide room of plant science will be sampled as a test site along with an ACC for comparison.

2 EXPERIMENTAL:

2.1 Chemicals, Materials and Equipment

2.1.1 Chemicals

The chemicals used were *sec*-butyl benzene, CS_2 and CH_2Cl_2

2.1.2 Materials

Activated carbon cloth

Samples of C-TEX standard woven cloth were used for most of the analyses. The sizes of exposed ACC were designated small (23 mm x 36 mm), medium (38 mm x 38 mm) or large (55 mm x 55 mm) depending on the slide holders used.

2.1.3 Analytical Equipment

Gas Chromatograph

A Hewlett Packard 5710A gas chromatograph with a flame ionization detector was used for

BTEX analysis. It had a 3 m column packed with 5% Bentone 5% Isodecylphthalate on Chromosorb W. Operating Conditions: Injector Port - 150°C, detector - 150°C; the column was held at 50°C for 8 minutes and linearly increased to 130°C at a rate of 8°C/min.

Gas Chromatograph - Mass Spectrometer

A Finnigan 800 ion trap mass spectrometer connected to a Varian 3400 gas chromatograph with a 30 m SPB-20 capillary column was used. Operating conditions: injector port - 150°C, column 80°C for 10 minutes, 8°C/min to 260°C.
A V.G.7070E-HF mass spectrometer directly coupled to an H.P. 5890 gas chromatograph with a SP 2100 capillary column was used for some analysis and extensive use was made of the internal spectral library search routine to identify the compounds.

Environmental Chamber

Air from the laboratory air line was purified by passage through an activated charcoal filter, dried by silica gel and 13X molecular sieve, and introduced into the bottom of the 41 x 31 x 61 cm box through a system of holes. The flow rate through the chamber was 1.45 L/min. which was measured by means of a calibrated rotameter.

Each component of the BTEX (benzene, toluene, ethyl benzene, p-xylene, m-xylene and o-xylene) was introduced to the air from separate small vials with a semipermeable membrane in the cap which had a small hole. The rate of diffusion through the membrane was constant as determined by the weight loss over time. Three small fans were used (two in the bottom, and one on the right side of the platform) to mix the air in the chamber. The fan on the side was added at a later stage to ensure the absence of concentration gradients in the chamber. A diagram of the chamber is shown in Figure 2.

2.2 Chamber Improvements

After much testing of various drying methods for air being passed through the chamber, it was finally possible to achieve a very low relative humidity of ~1%. The chamber lid was sealed with weatherstripping and the air was dried by passage through a silica gel column, a 13x molecular sieve, a Drierite™ ($CaSO_4$) column and finally through a column of granular carbon. For high humidity levels, the air was bubbled through a solution saturated with various salts which have been designated to give the required relative humidity.

The test chamber described in our previous work (1,2) was too small to accommodate several samples at one time. A larger chamber was constructed from an aquarium. The upper chamber fan was added to ensure a uniform concentration of BTEX throughout the sampling chamber. During the course of the work with duplicate and triplicate tests of the cloth in the chamber, we found poor reproducibility. This could be attributed to the lack of uniformity of BTEX in the test chamber and so a small fan was introduced into the testing area to mix

the air and to establish a uniform concentration throughout the testing chamber. The presence of this fan created some turbulence which affected the sampling rate. A diagram of this chamber is given as Figure 2 of this overall report.

The criteria used to evaluate the different ACC were high sampling rates and small effect of humidity on this sampling rate.

3. METHODOLOGY:

3.1 Extraction Process

Two methods, syringe and ultrasonics, were used in the extraction of VOC from the ACC passive samplers. Both methods use internal standards dependent on whether the GC-MS or GC-FID instrument was used. For the GC-FID usually used for BTEX analysis, carbon disulphide was used with *sec*-butyl benzene as the internal standard. Initially this was added to the individual CS_2 aliquots from a dilute standard solution but later was included in the solvent prior to the extraction process. An internal standard of *o*-hydroxyacetophenone in either CS_2 or CH_2Cl_2 was used with the GC-MS analysis.

The syringe method consisted of placing a fixed volume of the solvent in a 50 mL beaker and the ACC in a 30 mL syringe. The solvent is drawn into the syringe twenty times and the ACC squeezed each time as the liquid is expelled. The volume of solvent chosen depended on the type of ACC sampler used as shown in Table 1.

Table 1

ACC	Size	Volume of Solvent used with Syringe	Volume* used with Ultrasonics
Small	35 x 22 mm^2	1 mL	1-2 mL
Medium	36 x 36 mm^2	2 mL	2-3 mL
Large	55 x 55 mm^2	3 mL	3-4 mL

* dependent on the thickness of the cloth.

The solvent in the syringe was then ejected into a preweighed vial and the solvent weighed. After the addition of the internal standard, the vial was re-weighed. The vial was sealed with Parafilm™ and stored in the freezer compartment of a refrigerator until ready for analysis.

The SKC carbon tubes were extracted by the syringe method using only 1 mL of solvent. The solvent was weighed into a vial, internal standard added and weighed, the vial sealed and then stored.

3.2 Analysis

Each ACC strip that was exposed to the air (either in the chamber or out) was removed from its frame and placed in a 30 mL glass syringe and squeezed 20 times with exactly 1mL (for small ACC and SKC tubes), 2 mL (for medium ACC) or 3 mL (for large ACC) of carbon disulfide. The solvent was weighed and spiked with approximately 0.04g - 0.1g of a standard solution of *sec*-butyl benzene depending on the size of cloth. In order modify this procedure and to decrease the amount of CS_2 evaporation, the internal standard was added to the CS_2, with a concentration of 110 µg/g. This modification will be specified as Method A when applicable in the report.

Another extraction technique was tested in which the ACC was placed in a gas chromatography (GC) reaction vial along with the appropriate amount of CS_2 and internal standard. The vial was then shaken for a varying lengths of time (5 min - 1 hour) and a sample of this extract was injected into the gas chromatograph for analysis. This procedure will be called Method B when used.

SKC activated charcoal tubes were extracted in a similar manner by pumping 1 mL of CS_2 through each tube with a syringe. For active sampling, the flow rate through the carbon tube was 0.145 L/min.

A second extraction of the ACC showed that an insignificant trace amount of BTEX was left on the cloth and that it was unnecessary to correct for this. (See Table 2). The order of elution from the GC was as follows: CS_2, benzene, toluene, ethyl benzene, p-xylene, m-xylene, o-xylene, internal standard, naphthalene. This was determined by injecting each individual compound in its pure form and observing its retention time.

4. RESULTS AND DISCUSSION:

4.1 Results

4.1.1 Comparison of Molar Ratios to Area Ratios of BTEX to Internal Standard

This comparison was done to determine if the GC response factor was a 1:1 ratio between weight and area of BTEX, with respect to the internal standard. To determine if this was true a known concentration of BTEX was made, and from this we were able to see if the ratio of the areas: weight was a one to one relation. This was accomplished by using equation 1 and solving for the response factor (f).

Using equation 1, the individual response factors for each compound given by the weight /area ratios as well as the molecular weight/area ratios were calculated and are listed at the bottom of Table 3. From these results it can be concluded that the weight to area ratio is not a 1:1 relationship.

4.1.2 Analysis of Unknown BTEX in Order to Determine the Reliability of the Response Factor

Part A

To ensure that the response factors calculated from a known BTEX concentration (sec.4.1.1) are correct, an "unknown" BTEX solution was used for analysis.

Part B

The quantitative analysis n part A was found to be in error by a constant factor throughout. The reason for this fact is that the response ratio is dependant on the ratio of BTEX to internal standard. Thus, if the ratio of internal standard to BTEX is not what is expected experimentally then the response ratio will be incorrect as well. Therefore since the unknown analysed in Part A was more concentrated than normal, the amount of internal added was insufficient to give the ratio that was expected as found in sec. 4.1.1.

The unknown solution was analysed using the response ratios previously calculated. Using these values the incorrect quantitative value was calculated as seen in the Table 4, this value was consistently in error . The reason for this is in the ratio of internal standard to BTEX. When the response factors are used in the calculation, the ratio of standard to BTEX is what is expected experimentally. When the unknown solution was quantitatively analysed the ratio of internal to BTEX was not what is expected experimentally. Thus the concentrations found were incorrect. Once the ratio was corrected by adding double the amount of internal standard, the correct experimental conditions were again reached and the response factors were calculated by solving for f as before. The results for the response factor are well within error limits (5%) of what was previously calculated.

4.1.3 Active Sampling to Determine the Sampling Rate of Medium-sized ACC

To determine the sampling rate of the medium-sized cloths, two were placed in the chamber along with an SKC active sampler (0.145 L/min). The relative humidity of the chamber(10%) was the lowest that was ever obtained in this research. In line there was a 13X molecular sieve, silica gel and a charcoal filter. In the chamber there was two petri dishes that contained fresh Drierite™ that was changed upon attainment of 'pinkness'. The results are listed in Table 5.

The average sampling rate for the medium cloth was found to be 41.5L /hr which is quite high when compared to previous work [1] which found that the sampling rate for the large cloth to be 45L /hr. The discrepancy between the two results is due to the RH of the chamber at the time of analysis. In the present work the humidity is very low (10%), while in past work the humidity was not a controlled variable and therefore would be straight out of the laboratory air line which has an approximate humidity of 30 to 45%. This leads to the

conclusion that the sampling rate is definitely affected by humidity, and also that the sampling rate found in the present work would be near the maximum sampling rate obtainable.

4.1.4 Calculation of Off-gassing Through Various Times after 5 hr Exposure to BTEX

10 samplers were placed in the chamber for 5 hrs. After 5 hrs samples C, E, I, D were removed and immediately placed into plastic bags. Following this, the BTEX vials were removed from the chamber in order to determine the off-gassing in air of the remaining samplers. Samplers A, L and 36 were left in chamber for 10 hrs and then they too were immediately placed into bags. Samplers G and F were left in the chamber for the longest (22 hrs) and then they were removed as well. All samplers were analysed as they became available in order to circumvent the problem of off-gassing in the bags which would then give erroneous representation of the off-gassing in the chamber. Samplers C and E were not analysed for off-gassing since there was nothing to compare to, however they were looked at for reproducibility between themselves. All of the samplers were compared against C and E. Samplers 5 and 36 were polyurethane foams and were treated the same as the ACC, the humidity of the chamber was 10% and the air was dried as stated in the previous section. The results can be seen in Table 6.

Samplers A, L, G, F were left in the chamber after the BTEX was removed. Due to the residual VOC present, all of the samplers continued to gain BTEX, making it difficult to determine what the off-gassing is since the peak concentration of the chamber is not known. However the off-gassing for the 1 month incubation is determinable. As seen in Table 6, there is a trend in the off-gassing of the BTEX. This is as expected due to decreasing volatility of the compounds; this order has been seen previously in the order of elution from the column due in part to the boiling point differences. No BTEX was found on either of the two polyurethane foam samplers, 5 and 36. This is due to the poor sampling of the lighter compounds by the PUF [1] and thus since no compounds were found, no conclusions concerning off-gassing can be mentioned.

4.1.5 Comparison of Two ACC Samples Off-gassing in Air or in a Plastic Bag.

As seen in the previous section the residual BTEX in the chamber after removal of the vials does not allow for determination of the off-gassing. Thus to circumvent this problem two samplers (B9, C1) were exposed for 5 hrs, one was placed in a bag(C1) and the other was hung by a wire from the ceiling in Rm 340 of the Chemistry Building along with a blank that had not been in the chamber. After 19.5 hrs, the blank, B9, and C1 were analysed, the results are in Table 7.

Alternatively to what is seen in the previous section, there is off-gassing over the 19.5 hr period of hanging in air. Also, as seen previously in a plastic bag, there is a general decrease in the off-gassing as the compounds become less volatile. This trend is not so obvious in the

open air and may be attributed to air flow or perhaps humidity which was approximately 30% at the time of experiment. The average loss of BTEX is 0.51 μg/hr which could be significant if the sampling environment is a situation where contamination is not steady (ie. as in a school environment). There were no BTEX present on the blank, thus proving that there was no adsorption of compounds while hanging in the air for the 19.5 hrs.

4.1.6 Water Adsorption onto ACC at Equilibrium

The purpose of this experiment was to determine the extent of water uptake by ACC at different humidities. To achieve this 5 desiccators were partially filled with a saturated salt solution of various salts that gave humidities of 15%, 35%, 52%,72%, and 95%. These salts were chosen from the CRC Handbook for the humidities they gave, as well as their availability. The analysis was done in triplicate over a three day period. To ensure that equilibrium was reached a second weight was taken two days later which gave the same weights. This is graphed in Figure 3.

From the graph it can be seen that as the humidity becomes higher, more water was found on the cloth, finally reaching a saturation at 95%. This can now be used as a method of calculating the surface area of ACC since the cross sectional area of the H_2O molecule is known to be 10.53 $Å^2$. The saturation level was approximately 100 mg/g.

4.2 GC Vial Extraction Method

4.2.1 Testing the GC Vials for Evaporation of Solvent

This experiment was used to determine whether there was any evaporation or leakage of the CS_2 solvent through the cap or septum of the vials. Since the CS_2 is the most volatile of the components worked with, we wanted to make sure that it was not lost. All of the septa used were new and had no holes in them, but to see the effect of having a hole in the septum, one was used in this test. Two millilitres of CS_2 was placed into each vial and left for a week. The vials were weighed at different time intervals and these results were recorded in Table 8. The % loss was at most 8.5% (after 1 week, with a hole in the septum), which led us to believe that it was possible to use these vials for extraction purposes without losing any appreciable sample from evaporation.

The GC vial extraction method is a technique that was tested to determine if an ACC strip, which was exposed to BTEX (or VOC), could be solvent extracted by placing it in a GC reaction vial along with the appropriate amount of CS_2 and internal standard and shaking it for a length of time (later determined). This technique would be quicker and there would be less evaporation of the solvent, which will be discussed later (see Table 9).

The GC vial method of extraction was very thoroughly investigated as seen in the results. Although in theory the GC vial method seems to be simpler the results are inconsistent throughout. Thus it is not a viable method with the extraction being 20% reproducible at best

for the small cloth shaken for ½ hr. The use of ultrasonics for 15 min. was later found to be more effective in the extraction process of the ACC in vials than shaking. See Section 5.

The vial method involves less manipulation and is a cleaner method which allows the injection of several replicate samples into the GC-MS. We believe that good reproducibility can be achieved by using ultrasonics instead of simply shaking the vials. The vial can also be used to thermally desorb the VOC from ACC and it is our intention to test these methods. We have also developed a modification of the SPME methods which can serve as a passive monitor for VOC's in indoor air. See Appendix A.

4.2.2 Syringe Extraction Method vs. GC Vial Extraction Method

The two methods of extraction, syringe and GC vial, were compared in this experiment. It was observed, in Table 9, that the syringe method extracted more BTEX, but this did not take into account that we lose a fair amount of CS_2 through evaporation. Therefore, another experiment was done to see the approximate amount of solvent evaporation that takes place using the syringe method. It was observed that approximately 20% of the sample was lost using the syringe method. Taking this loss into consideration, the GC vial method works about as well as the syringe method.

4.2.3 Test for Uniform Adsorption onto an ACC strip and Reproducibility within a Sample

Two large ACC strips were exposed to BTEX in the chamber and then randomly hole punched into GC vials upon their removal. As seen in Table 10, samples 1-5 were from one cloth and samples 6-10 were from the second cloth. Each GC vial contained 6 random punches, along with 1 mL of a CS_2/ IS (internal standard) mixture. The vials were shaken for one hour and run on the GC. The first set of results were very widespread and had a high standard deviation. It is believed that there was an error present in one of the analytical steps along the way. However, the second set of results showed that there was a relatively uniform distribution of BTEX onto the cloth. Lastly, the reproducibility within a sample was checked by injecting sample #10, several times. It was observed that reproducible results were achieved within the sample themselves.

4.2.4 Determination of the Appropriate Extraction Time for the GC Vial Method

For this next section, several experiments were conducted to determine if the samples had to be shaken for 1 hour, or if 5,15 or 30 minutes (shaken or unshaken) was enough time for a proper extraction. In Tables 11 and 12, the CS_2 and the internal standard were added separately, but after that (unless specified) the internal standard was included with the CS_2.

4.2.4a *5, 15 & 30 Minute Extraction Times*

Table 10 shows that 5 minutes was not enough time for the extraction method.

4.2.4b *15 Minutes vs. 30 Minutes Pt.1*

The samples were again either shaken or left standing for 15 or 30 minutes. The results (see Table 12) showed inconsistencies and a second trial was attempted, using a CS_2/IS mixture to reduce evaporation losses and error.

4.2.4c *15 Minutes vs. 30 Minutes Pt.2*

In this trial, medium cloths were used. Although the CS_2 and the internal standard were added together, there was still a fair amount of inconsistency in the results (see Table 13). Again, these results made it hard to come to any conclusions about the proper time for extraction, or if the GC vial method could be used. Therefore large amount of samples were taken and extracted by shaking them in the vials for 30 minutes to determine the precision.

4.2.4d *30 Minute Extraction Time*

Twelve samples were used in this trial to see if 30 minutes (unshaken) was enough time to extract BTEX from medium cloths. The results are listed in Table 14. The standard deviation ranged between 12.7 - 20.0%. One final experiment was attempted to determine if this deviation could be reduced by shaking for an hour.

4.2.4e *1 Hour Extraction Time*

Small ACC strips were used for this trial. The samples were shaken for 1 hour and then run on the GC. The results, seen in Table 15 shows a standard deviation ranging from 12.5-18.7%. There was not a significant change from that of the 30 minute trial, in terms of deviation. Therefore, this technique was put on hold for the time being. This was repeated using ultrasonic techniques to improve the extraction.

5. ULTRASONIC EXTRACTION METHOD:

Chemical reactions in an ultrasonic field deal closely with the phenomenon of cavitation[3]. Cavitation is the formation of cavities in the liquid and their collapse, which is accompanied by intense hydraulic shocks[3]. Therefore, the liquid, in our case CS_2, vibrates and these cavitation bubbles expand and compress, eventually collapsing under the action of the next compression. The strength of the hydraulic shocks produced by the collapse of cavitation bubbles can be estimated from the intensity level of the observed hissing sound. The importance of ultrasonics for our extraction is that these intense vibrations of the liquid not only affect the CS_2 in the GC vial, but help move the layer of water that is comfortably placed between the CS_2 and the BTEX compounds adsorbed in the pores of the different activated carbon cloths. This movement of the water molecules allows for the CS_2 to penetrate the water barrier and extract the BTEX compounds from the cloth.

For the following experiments, we used a 20 kHz KONTES Ultrasonic Cleaner.

5.1 Results And Discussion

5.1.1 *Ultrasonics: Part 1*

Eight small ACC samplers were exposed in the BTEX chamber for 5 hours at a relative humidity of ~6%. The samplers were then removed and placed into a GC vial, along with 1 mL of CS_2/IS mixture. The lids were wrapped with Parafilm™ and the vials were placed into the ultrasonic bath for various lengths of time.(See Table 16) Three blanks were also run to make sure that the CS_2/IS mixture was not being degraded during the extraction process.

It was observed that this ultrasonic extraction technique worked very well and there was no apparent degradation of the solvent or internal standard. Also, 15 minutes in the ultrasonic bath seemed to be a sufficient amount of time for an extraction of these small samplers.

5.1.2 *Ultrasonics: Part 2*

The next objective for the ultrasonic technique was to determine if it was better than the syringe technique currently being used. Six small sized samplers were placed in the chamber for 5 hours. Once removed, three of the samplers were extracted using the syringe method and the other three were extracted by being placed in the ultrasonic bath for 15 minutes.(See Table 17).

The results showed that the ultrasonic extraction technique worked better than the syringe method. It extracted more BTEX and it had a lower standard deviation (5-10%). Since we observed from these results that the ultrasonic bath is a good extraction technique, we used it on different cloths, namely the top three cloths identified previously.

5.1.3 *Ultrasonics: 15 Minutes - Part 1* *Top 3 ACC*

The best three ACC cloths were determined to be Porton Downs, ACC-5092-10 and ACC-5092-20 (American Kynol), respectively. Several small sized strips were cut from these fabrics and then silylated. Activated carbon cloths are hydrophilic and adsorb water readily (especially at higher relative humidities). This silylation process was performed to make the cloths more hydrophobic.

The ACC strips were first treated with CS_2. Then the strips were soaked in a 10% dimethyldichlorosilane/CS_2 solution and placed in the ultrasonic bath for 15 minutes. Lastly,

the strips were soaked in a 10% trimethylchlorosilane/CS_2 solution and placed in the ultrasonic bath for another 15 minutes. The ACC strips were dried on a hot plate for a day and a half at 150°C.

Nine samplers (3 of each type of cloth) were placed in the BTEX chamber for 3 hours, with a relative humidity of ~2%. An SKC tube was also placed into the chamber, for 3 hours.

As can be seen from the results (See Table 18), there is considerable inconsistency between the ACC strips of the same brand. But more importantly, the amount of BTEX adsorbed onto the cloths ACC-5092-10 & ACC-5092-20 is extremely low. This may be due to the fact that there were several impurities on the cloths from the silylation process because they had not been on the hot plate long enough. The Porton Downs ACC strips worked a lot better, but still had a few impurities. Therefore, all of the ACC strips were put on another "hotter" hot plate for several days, and exposed again to BTEX.

5.1.4 *Ultrasonics: 15 Minutes – Part 2* *Top 3 ACC*

Nine samplers (3 of each type) were exposed again to BTEX in the chamber and extracted using the ultrasonic bath for 15 minutes. Again there seems to be a variation in the results obtained, even though the previously found impurities were not present.(See Table 19). It appears that these cloths, although they are small in size, need 2 mL of CS_2/IS to be extracted properly. The cloths, from American Kynol, are stiff and thick and do not get completely covered by 1 mL of the extracting solvent. Therefore, the next trial used 2 mL of the CS_2/IS mixture.

5.1.5 *Ultrasonics: 15 Minutes – Part 3* *Top 3 ACC*

In this experiment, six samplers (2 of each) were exposed to BTEX, along with an SKC tube. The humidity, again, was very low (~0.5 %) and the exposure time was 3 hours. For this trial though, 2 mL of CS_2/IS mixture was used to extract these cloths. They were placed in the ultrasonic bath for 15 minutes and then run on the GC. The results (See Table 20) were considerably more consistent than in previous trials. Therefore, the next few trials were done using the same cloths, but at a higher relative humidity.

5.1.6 *Ultrasonics: 15 Minutes – Part 4* *Top 3 ACC*

In this experiment, six samplers (2 of each) were exposed in the BTEX chamber for 5 hours. But, this time, the relative humidity was ~55%. To increase the humidity, most of the drying agents hooked up to the air line into the chamber were bypassed and the air bubbled through a solution of calcium nitrate. There were problems though in keeping the humidity in the chamber stable and it fluctuated throughout the exposure time between 45% and 75%.

Several times, we had to open the door slightly to get the humidity down from 75%. This, in turn, caused the results to fluctuate and there was loss of BTEX from the chamber.(See Table 21).

5.1.7 *Ultrasonics: 15 Minutes – Part 5* *Top 3 ACC*

Six more samplers were exposed to BTEX for 5 hours. In this second trial, the chamber humidity was much more stable than in the first trial. The relative humidity was ~50% and fluctuated only slightly from this value. These small samplers were extracted with 2 mL of CS_2/IS and placed into the ultrasonic bath for 15 minutes.(See Table 22). The results were quite good and a last experiment was done at a low humidity to make comparisons and more importantly, to see if the silylation process had made the cloths hydrophobic and less susceptible to moisture interference.

5.1.8 *Ultrasonics: 15 Minutes – Part 6* *Top 3 ACC*

The relative humidity in the chamber was decreased to ~8.5%. The calcium nitrate solution was disconnected and the drying system was reconnected. Six samplers (2 of each type of cloth) were exposed to BTEX and then extracted using the same ultrasonic technique. Small samplers were exposed in the test chamber for 5h. The contents of the chamber were simultaneously actively tested with an SKC carbon tube (145 mL/min). The passive samplers were extracted with 2 mL of CS_2 (which included the internal standard) for 15 min with ultrasonics. The attenuation of the GC was set at 128 for the 1 L injection of CS_2 extract. It was observed (see Table 23) that the first two cloths from American Kynol (ie ACC-5092-10 and ACC-5092-20) still fluctuate in their results and it is difficult to come to any conclusions. The evaporation of the solvent and its loss during the extraction process using the syringe were probably responsible for the irreproducible results with the ultrasonics extraction process. It is apparent, though, that the Porton Downs cloth, which is a lot lighter in weight and is not as stiff as the other two, works consistently well, and was determined to be the best cloth in other experiments (See sec. 5.4). Future experiments, therefore, should be concentrated on this cloth only.

5.2 Conclusion

The new ultrasonic extraction technique is simple and efficient. The procedure allows for almost no evaporation of solvent, internal standard or BTEX and appears to work better than the syringe method of extraction. Also, the time for extraction (ie 15 minutes) is very reasonable. It should be noted though that there are problems with consistency between results when using ACC that are thick and stiff in form. It is difficult to ensure that the cloths get completely covered and stay covered during the extraction process. Therefore, future experiments should concentrate on

the use of the best cloth, Porton Downs.

5.3 12 Different ACC Fabrics

We have been able to obtain a selection of different ACC materials from several suppliers. The 12 different types of ACC fabrics were tested to determine which were least affected by humidity. The results are shown in Tables 24-27.

5.3.1 *Comparison of 12 Different ACC Fabrics at Different Relative Humidities*

Twelve medium sized ACC were exposed to BTEX for 5 hours at 19.5% relative humidity and extracted using the GC vial method. Table 24 shows the best 5 cloths and the brand and type of ACC fabric. We had been using C-TEX St ndard Woven (sample 10 (A5)).We next used the same 12 cloths, but at the much higher relative humidity of 71%.

At this high relative humidity, we observed that different ACC fabrics had higher sampling rates. Again, the best 5 cloths, along with the rest of the result , are listed in Table 25. The samplers were extracted using the GC vial method. These cloths were extracted via the GC vial method and thus are suspect since it is unclear if the cloth is truly better or if this is simply a function of the systematic error associated with the GC vial method. Concurrently, C-TEX Standard Woven is the only ACC found in the top 5 cloths for both humidities which seems to be rather odd since similar result in the order of sampling efficiency would be expected for both humidities.

The two previous results were obt ined by extract ng the samplers us ng the GC vial method. Since it was previously observed that this method showed inconsistencies, the 12 fabrics were retested at 28% relative humidity and extracted using the syringe method. The result , along with the best 5 cloths, are listed in Table 26.

The syringe method was used to extract the ACC samplers, which were exposed to the BTEX for 5 hours. The results, long with the best 5 cloths, are shown in Table 27. Since the GC vial method was inconsistent (sections 4.2.1, 4.2.2) the experiment were repeated at similar humidities with syringe extraction. However, again only one cloth (Porton Down) was in the top 5 at each RH. This led us to an investigation into the consistency of the BTEX in the chamber (see results 4.2.3). In experiment 4.2.3 two large samplers were placed in the chamber for 5 hrs. Hole punches (6 holes) were randomly taken from each sampler and analysed. Keeping in mind that the extraction was done via the GC vial method the results between the two were still in large disagreement. This then leads to the conclusion that the chamber is not homogeneous with respect to BTEX concentrations throughout. Thus the experiment 5.3.1 needs to be repeated once the chamber is known to be homogeneous i order to reach any meaningful conclusions.

5.4 Results and Discussion

Sections 5.4.1, 5.4.2 and 5.4.3 were run before the final modifications to the chamber.

5.4.1 *12 Different ACC In "New" Chamber*

The 12 different activated carbon cloths (ACC) that we have been working with were tested in the chamber with the additional fan. This was done to see if there was a more consistent flow of air in the chamber and if the BTEX concentration also stayed consistent.

Although there were differences between this trial and a previous trial in which there was NO upper fan, it seems that the cloths that adsorbed the most are still working very well. The air circulation in the chamber has obviously changed and some fluctuations in results between different trials can be expected. (See Table 28). The main objective of this experiment was to determine the best three cloths. After several trials, it was found that ACC-5092-10, ACC-5092-20 (American Kynol) and Porton Downs worked the best and was studied further. Porton Downs is rated as the best cloth that we have worked with.

5.4.2 *Comparison of Different CCL* Fabrics in "New" Chamber*

Several different types of CCL (Charcoal Cloth Limited)* cloths were tested to determine which ones were the best, especially at higher humidities.(See Table 29). The activated carbon cloths were exposed for the same amount of time as the 12 different ACC listed above and at the same relative humidity (~30%). It was observed that certain cloths worked well, but not as well as those chosen above. Also, the CCL cloth that was fluorinated (and apparently hydrophobic) did not work better than the three ACC chosen. Therefore, the following experiments focussed on the performance of the three chosen cloths at different humidities and after undergoing a silylation process to make them more hydrophobic.

5.4.3 *Comparison of the Top 3 ACC, an SKC Tube and a 3M Sampler*

The top three ACC were exposed for 5 hours in the BTEX chamber, with a relative humidity of ~30%. The amount of BTEX was unusually high, but this can be accounted for by the fact that the BTEX vials in the chamber had been filled up that day, and therefore the concentration of BTEX in the chamber was higher than usual.(See Table 30). The results in Table 30 show that the 3M sampler and the SKC carbon tube give values which are different by a factor of 3. This is explained by the humidity effect which does not affect the 3M sampler as much but reduces the sampling rate (ie adsorption efficiency) of the SKC carbon tube. This was confirmed by Vylkov [2]. Thus if the 3M sampler is assumed to be correct, then the passive samplers (1 to 3) indicate uniform but slightly different sampling rates as shown.

Similar results were obtained from our samplers which were sent to ORTEC for comparison with the 3M sampler and active sampling at a relative humidity of 50% (see Appendix B). Under these high humidity conditions the ACC passive samplers showed very low values of

about 33% of the 3M samplers. It is for this reason that we believe that a **hydrophobic** cloth may be a better passive monitor since moisture should have a minimal effect on the sampling rate.

5.4.4 *Comparison Of CCL Cloths At ~8% Relative Humidity*

The chamber had undergone further improvements and the different CCL cloths were run to see the effect of a low relative humidity.(See Table 31). As expected, there was an increase in BTEX adsorption, but this was seen in all of the cloths exposed at such a low R.H. Therefore, these cloths were not used for any further experiments.

5.5 Study of the Polyethylene Bags Used for the Samplers

We originally used a 'Zip-Loc™'- type polyethylene bag to hold the samplers after exposure to the volatile organics. A series of experiments were conducted to see if the VOCs could permeate through the bag and contaminate the sampler. This information was most important when analysing the "blanks" sent along with each sampler.

5.5.1 *Aluminum Foil Wrapped Samples*

In this experiment, samplers were placed in a polyethylene bag and placed into the chamber for 4.5 hours. As a blank, we placed one sampler in a bag only and left it in the chamber also. The second sampler was placed in a bag and then wrapped in the aluminum foil, "shiny side in". The third and final sampler was placed in a bag and wrapped with the "shiny side out". These medium sized samplers were extracted using a syringe. The results are shown in Table 32. The blank, B, placed in only a plastic bag, shows that BTEX can permeate through the bag and adsorb onto the fabric. This can become important since we are looking at such low concentrations of VOC. The aluminum foil drastically reduces this permeation, especially when it was wrapped "shiny side in". Either way, the aluminum foil was a considerable improvement.

The evidence to investigate the permeability of the bags came from a blank that was sent for field testing. The blank was left at the site of contamination while another was hung in location. Upon analysis of the blank and sampler, it was found that both had significant amounts of VOC present. In fact the amounts were close enough to be uncertain which was the blank and which was the sampler. To test this theory of bag permeability experiment 5.5.1 was run. The results found that there were BTEX present on the non-foiled sampler, but none on the one wrapped in foil. Thus to ensure proper analysis of field samplers, they should be wrapped in aluminum foil preferably shiny side in after they are placed in bags.

5.5.2 *Testing of Aluminized Bags (from Ludlow Corp.) for Permeation of BTEX*

A medium-sized sampler was placed into an aluminized bag and then put into the chamber for 18 hours. A second medium-sized sampler was placed into an aluminized bag and left for a week. This BLANK was used to make sure that the aluminized bag was not contaminating the sampler. It was observed that the BTEX did not permeate through the aluminized bag and that it did not contaminate the sampler. The samplers were extracted using the syringe technique and run on the GC; but the attenuation on the GC was much lower (ie. more sensitive) than usual for regular samplers. This was set at 4, as opposed to the regular setting of 128. Therefore, it can be concluded that the aluminized bags helps to prevent any cross-contamination between samplers, or permeation of BTEX.(See Table 33).

5.5.3 *Determination of a New Sampling Rate for Medium ACC at Low R.h.*

In this experiment, four medium-sized samplers and an SKC tube were exposed to BTEX for several hours. Two of these samplers, along with the SKC tube were extracted immediately after their removal from the chamber. The last two samplers were placed in aluminized bags, from the Ludlow Corporation, in order to determine if there was any off-gassing.(See Table 34). The average sampling rate (benzene excluded) was 22.7 L/hr. This is an increase from the previously determined 18 L/hr for a medium-sized sampler [1]. Since humidity plays a major role in the adsorption of BTEX, or any volatile organic compound, onto the cloth, this increase in sampling rate is expected.

The last two samplers were placed in the aluminized bags for a week and then extracted. The off-gassing ranged from 35.2% to 59.2%. These results are very high and will have to be further studied.

5.5.4 *Permeation of BTEX Through Aluminized Bags from Winpak*

Three types of aluminized bags were obtained from Winpak and tested for permeation of BTEX. The first bag was 10cm x 12cm and the second bag was 10cm x 15cm. These bags were made of the same aluminum material, but the third bag was a little bit different. It was 6" x 8.5" in size and aluminized on only one side. The other side of the bag was clear plastic.

Two medium-sized samplers were placed, respectively, into the first two aluminized bags and exposed in the chamber for 24 hours. Several other medium-sized samplers were placed into the chamber, but only for 5 hours. These samplers would then be placed into aluminized bags from Winpak to determine the off-gassing of the bags.The results obtained showed that there was NO permeation of BTEX through the bags. However the chamber had been altered slightly by disconnecting the upper fan and this caused the results to fluctuate greatly. The air in the chamber was not circulating very well without an upper fan at this low humidity. Therefore, no off-gassing results could be obtained from this trial.

Lastly, the third type of Winpak bags was tested for permeation of BTEX. These samplers

were tested with the upper fan working. Three large samplers were placed into three bags, respectively, and placed into the chamber for ~24 hours. Upon their removal, they were extracted and run on the GC at an attenuation of 4. Since we have had problems previously with permeation through plastic bags, it came as no surprise that there was permeation through these bags.(See Table 35). Although only benzene and o-xylene were in high enough concentrations to be detected, it is quite apparent that these bags are unsuitable because we are dealing with BTEX concentrations of this magnitude. A summary of the sampling rates at various relative humidities is given in Table 36 for small size ACC samplers.

6.1 Inside Needle Capillary Adsorption Trap (INCAT)

An INCAT device was prepared by intemally coating a needle with carbon (carbon paint in our case) and sampling BTEX with it. Once the needle was coated, it was heated in the injector port in order to drive off all of the organics already present in the paint. What was left was a needle with an internal coating of carbon. Sampling was performed by passing air through the needle by means of a syringe, or by passive diffusion. The VOC. are concentrated onto the inner surface. The INCAT device was chromatographically analysed by thermally desorbing the sorbcd BTEX. This was done by placing the needle into the hot injection port of the GC and then running it. Although this technique worked in other research, we did not get any significant results.

The conclusions on the INCAT device is that the process is simple and more convenient then the ACC. Testing in the chamber for periods of 3-5 days of passive monitoring showed no peaks on the GC which had the sensitivity increased due to the insensitive nature of the INCAT. Active samplingo f the chamber air was attempted but this too was unsuccessful.

6.2 Painted Slides

Several glass slide were painted with ink (from Pollard Banknote), dried, and then placed on a hot plate to drive off any organics that were present. These slides were then placed in the BTEX chamber for 18 hours, at a relative humidity of ~7%. Also, the upper fan in the chamber was NOT on. Upon their removal, the paint was scraped off into a GC vial and 1ml CS_2/IS mixture was added. This mixture was left for an hour and then run on the GC at an attenuation of 4 (regularly 128). Although the sensitivity on the machine was very high, no BTEX was detected. It should be noted that the slides had been left on the hot plate for a few weeks and the paint layer was thin and chipping off. This may have played a factor in these results. Lastly, more glass slides were painted with a suspension made of a carbon black powder (Raven-15, Columbian Carbon Company) and water. Several coats of this suspension were applied to these slides, but it kept cracking once it had dried. Therefore, these slides could not be used for testing. In order to do any further experiments, a paint or ink must be found that stays intact once dried on a hotplate.

6.3 Conclusion

We saw in the previous experiments that the improvements made to the chamber enabled us to perform experiments at extremely low relative humidities. They also helped us stabilize and seal the chamber, as well as increasing the circulation of air and BTEX. This increased circulation allowed for a more uniform concentration of BTEX in the chamber, as well as a more uniform adsorption of BTEX onto the cloths. Lastly, we were able to determine which activated carbon cloths worked the best, even at higher humidities. These top 3 ACC were Porton Downs, ACC-5092-10 and ACC-5092-20 (American Kynol), respectively.

7. INSIDE NEEDLE CAPILLARY ADSORPTION TRAP (INCAT):

An attempt was made to thermally desorb the carbon cloth instead of using a solvent to extract the cloth. This was done by inserting the exposed cloth into a Reactivial™ which was fitted with a screw cap and septum. The vial was then inserted into a heated metal block (150°C) for 15 min. A 0.5 mL gas tight syringe was used to sample the off-gases from the ACC and for injection into a GC/FID. No consistent results were obtained and in view of our work on INCAT it was decided to abandon the thermal desorption of ACC and to further test the INCAT method.

Various methods were used to obtain the coating on the inner area of the needle but reproducibility was not achieved. Different coatings showed significant variations in sampling rate although the reproducibility with a single needle was good. This is illustrated in Figure 4 where two different needles with different carbon coatings A and B were exposed for 1 hour in a BTEX chamber. The same needle, B, showed similar areas for BTEX when exposed for the same time in the same chamber.

An estimate of the carbon loading of 1 mg/needle leads to a surface area of about 1 m^2 if it is assumed that the carbon has a modest area of 1000 m^2/g. If we assume that the average molecular area is 50 $Å^2$ or less and since 1 $m^2 = 10^{20}$ $Å^2$,then the carbon can accommodate 2 x 10^{18} molecules or about 3 micromoles for a monolayer coverage.

A 1 hour exposure of the INCAT sampler in a BTEX chamber with a benzene concentration of 50 $\mu mol/m^3$ gave about 50 pmol of benzene into the GC on thermal desorption. This corresponds to a sampling rate of about 1 mL/h and leads to a coverage of the carbon of about 0.001% of a monolayer.

We believe that there are several aspects of INCAT that remain to be clarified. These are (1) the need to achieve reproducible coatings (2) to achieve sampling rates which will not be greatly

affected by humidity changes, and (3) to obtain stable coatings which are not changed or destroyed while the needle is handled, mailed or exposed for long periods. Leaving the bottom (Luerlock end) of the needle open during the sampling period may increase the sampling rate by allowing the air to flow through the open ended needle by convection flow; this has to be evaluated. The off-gassing of the INCAT passive sampler in the storage vial has yet to be determined.

Several INCAT devices were prepared by internally coating a needle with carbon (carbon paint or a carbon suspension in our case) and sampling BTEX with it. To coat the needles, the carbon paint (a carbon graphite paste used for SEM mounts) was drawn through a 22 gauge needle several times in order to internally coat it with carbon. Then, air was drawn through the needle to ensure that it was not clogged and to remove excess paint. The needle was left to dry for several hours and this procedure was repeated. When using the carbon suspension to coat needles, the procedure above was repeated several times to ensure that the needles were coated, because it was thinner than the paint. To make the suspension, a small amount of carbon black powder (# Raven-15, Columbian Carbon Company), along with 2 drops of Igepal CO-630, was added to ~10mL of water and stirred thoroughly.

After the needles have been coated and dried, they are heated in the injector port of the GC in order to drive off all of the organics that may be present. What was left were needles with an internal coating of carbon. Sampling was performed by placing the needles, upright in a vial, into the BTEX chamber and passively sampling for various lengths of time. But, more importantly, a piece of septum was placed in the bottom of the needle before exposure. This piece of septum is very important because it prevents the loss of sample when the needle is later placed into the injector port to thermally desorb the BTEX. This minor detail was not done in some of the preliminary trials and may be the reason why no results were obtained.

Once the needles are placed into the chamber, the BTEX becomes concentrated onto the inner surface. As stated before, the INCAT device was analysed chromatographically by thermally desorbing the sorbed BTEX. This was done by placing the needle into the hot injector port of the GC and then running it. The results of several trials are listed below.

7.1 Results And Discussion

Several trials were performed using the INCAT needles produced. The exposure times tested ranged from 1 hour to 48 hours. During these trials, a septum was in place in the needles. One previous trial, which showed results after 1.5 weeks, did not contain a septum. The attenuation was set at 4 (regularly 128) on the GC for most trials, but for the 1 hour trial it was set at 1, the highest sensitivity setting for our GC.

From the first trial of 1.5 weeks, at a relative humidity of 5%, the needles labelled: Raven-15, M, M1 and INCAT showed BTEX adsorption. The next trial was for 48 hours, at a relative humidity of ~55%. In this trial, a piece of septum was used to seal the Luerlock end of the needles before exposure. The results were positive for all four samples, "M1" & "Raven-15" showing the

most BTEX adsorption. Further trials were performed at 20.5 hours, 4 hours and 1 hour. After 20.5 hours exposure, at a relative humidity of ~40%, all of the INCAT needles, except for the one labelled "INCAT" showed BTEX adsorption. The attenuation on the GC was set at 4. Perhaps results would have been observed if it was set at 1. After 4 hours exposure, at ~11% RH, the results were seen with the needle, "M1", only. Again, the attenuation on the GC should have been set to 1 instead of four. The last trial was for 1 hour, at a relative humidity of ~8.5%. The attenuation on the GC was set at 1, the most sensitive. The injector port temperature was also raised from 150 C (in all previous trials) to 200 C. Sample "M" showed benzene and toluene, but no xylene adsorption. Samples "M1" and "Raven -15" showed adsorption of all BTEX compounds.

7.2 Conclusion

The conclusion regarding the INCAT devices (ie. needles) is that the process is simple and more convenient than the ACC. It is a sensitive and solvent-free technique that works for exposure times as short as one hour. Previous trials in which these INCAT devices were used to actively sample BTEX also showed positive results, but passive sampling is a more important application. Further research using these devices will have to establish sampling rate and reproducible coatings or coated inserts.

Recent additional work has indicated that the passive sampling of BTEX by the INCAT sampler is reproducible within 10% for a single sampler as well as for three different samplers.

8. CONCLUSIONS:

We have obtained from one manufacturer a sample of a hydrophobic carbon cloth and another has promised to send a similar material which we will test for RH effects.

It was initially proposed that the ACC samples all vapours with equal efficiency and at a rate proportional to their molar concentration in air. This has been based on previous results and was valid within about 50%.

Using an internal standard, it is believed that the GC-MS chromatograph peak areas which are based on the total ion currents, would give the molar concentration of the compound in the sampled air. When tested with known concentrations of various substances relative to an internal standard, it was shown that in general, the larger molecules gave higher areas per unit molar concentration probably because of the greater number of ions produced in the cracking pattern. For improved accuracy therefore, it is necessary to establish a general correction factor which will depend on the molar mass of the compound relative to that of the internal standard chosen.

We have been using the ACC samplers to test indoor air in homes and various businesses, factories and restaurants. The GC-MS results of some ACC sampling are given in Figure 5. These

typical results include an automobile and a restaurant in Holland. We have also tested the off-gassing of beetles for a colleague, Dr. N. Holliday in Entomology at this university.

We have not been able to locate a "sick" building to test as yet. We have put a request on the Internet received no positive responses.

We are also testing the efficiency of a GAC filter for the Farr Co. (California) who have tested the effect of air velocity on the sampling rate of ACC. Please see Appendix C for their report.

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Table 2

Comparison Between First and Second Extractions

First Extraction

Sample	($x 10^{-3}$ g)	benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene	
14		0.699	1.113	0.439	0.404	0.381	0.252	
15		0.806	0.943	0.395	0.358	0.340	0.256	
16		0.241	0.473	0.199	0.179	0.167	0.110	--excluded
17		0.615	1.453	0.597	0.544	0.517	0.339	
19		0.683	0.967	0.383	0.348	0.343	0.228	
20		0.848	1.551	0.633	0.574	0.556	0.366	
	Avg	0.730	1.205	0.489	0.446	0.428	0.288	
	SD	0.095	0.280	0.117	0.106	0.102	0.060	
	%SD	13	23	24	24	24	21	

Second Extraction

Sample	($x10^{-3}$)							
14		0.004	0.006	0.002	0.002	0.002	0.002	
15		0.004	0.008	0.003	0.004	0.005	0.006	
16		0.009	0.017	0.008	0.010	0.011	0.012	--excluded
17		0.003	0.006	0.003	0.003	0.004	0.005	
19		0.001	0.006	0.004	0.005	0.007	0.008	
20		0.007	0.008	0.008	0.008	0.013	0.011	
	Avg	0.003	0.007	0.004	0.004	0.006	0.006	
	SD	0.002	0.001	0.002	0.002	0.004	0.003	
	%SD	64	17	61	53	68	56	

Percentage of Sample Recovered in Second Extraction

Sample				%				
14		0.53	0.52	0.43	0.52	0.49	0.66	
15		0.45	0.89	0.76	1.06	1.40	2.18	
16		3.55	3.69	4.16	5.45	6.28	10.89	--excluded
17		0.46	0.40	0.42	0.57	0.75	1.35	
19		0.73	0.66	0.98	1.36	2.04	3.36	
20		0.79	0.50	1.22	1.43	2.26	2.99	
	Avg	0.59	0.60	0.76	0.99	1.39	2.11	
	SD	0.16	0.19	0.35	0.43	0.77	1.12	
	%SD	26	32	46	43	56	53	

The above small ACC samplers were exposed to BTEX for 17 hours, at a relative humidity of ~20%.
The GC vial extraction method was used where the samples were shaken for an hour for each extraction.

Table 3

Comparison of Molar Ratios vs Area Ratios of BTEX to Internal Standard

Weight ratio of BTEX to *sec*- Butyl Benzene(IS)

Compound	MW	Wt(g)	mmoles	Ratio(mmol)	Ratio(wt)
benzene	78.12	0.11	1.39	3.28	1.91
toluene	92.15	0.11	1.23	2.90	1.99
ethyl benzene	106.17	0.10	1.08	2.54	1.74
p-xylene	107.18	0.10	0.91	2.16	1.72
m-xylene	107.18	0.11	1.07	2.53	2.01
o-xylene	107.18	0.13	1.21	2.85	2.27
2-butyl benzene	134.22	0.06	0.42	1.00	1.00
naphthalene	128.19	0.07	0.57	1.33	1.27

GC Area ratio of BTEX to *sec*- Butyl Benzene (IS)

Compound	Trial 1		Trial 2		Trial 3		Trial 4	
	Area	Ratio	Area	Ratio	Area	Ratio	Area	Ratio
benzene	14.90	2.17	18.68	2.26	18.93	2.24	18.49	2.21
toluene	15.86	2.31	19.39	2.35	19.69	2.33	19.80	2.37
ethyl benzene	13.65	1.99	15.91	1.93	16.20	1.92	16.52	1.98
p-xylene	12.93	1.88	15.79	1.91	16.13	1.91	16.17	1.94
m-xylene	15.98	2.33	18.47	2.23	18.85	2.23	18.84	2.26
o-xylene	16.65	2.42	19.77	2.39	20.25	2.40	20.12	2.41
2-butyl benzene	6.87	1.00	8.26	1.00	8.45	1.00	8.35	1.00
naphthalene	9.64	1.40	11.69	1.42	11.96	1.42	11.74	1.41

	benzene	toluene	ethylbenzene	p-xylene	m-xylene	o-xylene	naphthalene
Avg Ratio	2.22	2.34	1.96	1.91	2.26	2.41	1.41
SD	0.04	0.03	0.04	0.02	0.05	0.01	0.01

Compound	Weight ratio / area ratio	Mole ratio/area ratio
benzene	0.86	1.48
toluene	0.85	1.24
ethyl benzene	0.89	1.30
p-xylene	0.90	1.13
m-xylene	0.89	1.12
o-xylene	0.94	1.18
2-butyl benzene	---	---
naphthalene	0.90	0.94

Conclusions: The weight to area ratio is not a 1:1 relationship. Thus to correctly quantitate the results a response factor of the detector to BTEX is needed. This response factor for each individual compound is the weight ratio/area ratio listed above.

Table 4

Determination of Reliability of Response Factor

Part A

Trial	(g) benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene	naphthalene
1	0.0209	0.0197	0.0129	0.0219	0.0233	0.0216	0.0042
2	0.0208	0.0197	0.0129	0.0219	0.0233	0.0215	0.0042
3	0.0195	0.0186	0.0127	0.0214	0.0228	0.0209	0.0050
Average	0.0204	0.0193	0.0128	0.0217	0.0231	0.0213	0.0045
SD	0.0008	0.0006	0.0001	0.0003	0.0003	0.0004	0.0005
% Composition	1.08	1.02	0.68	1.15	1.22	1.12	0.24
Actual Composition	1.56	1.69	1.07	1.69	1.82	1.64	0.33

Part B

Amount of Standard = 0.0136

Compound	Response Ratio
Benzene	0.96
Toluene	0.85
Ethyl benzene	0.84
p-xylene	0.91
m-xylene	0.91
o-xylene	0.88
naphthalene	0.89

Table 5

Sampling Rate of Medium-sized ACC

Relative Humidity=10% Rate of active sampling= 0.145L/min

Therefore 33.32 L of air were sampled.

BTEX	µg/L	Bottom Tube	ACC (mg)	BTEX / L in chamber (µg/L)	Rate(L/hr)
benzene	1.10	0.037	0.23	1.10	41.8
toluene	1.30	0.044	0.26	1.30	40.0
ethyl benzene	0.42	0.014	0.085	0.42	40.3
p-xylene	0.78	0.026	0.18	0.78	47.2
m-xylene	0.45	0.015	0.086	0.45	38.0
o-xylene	0.25	0.008	0.052	0.25	41.6
				Avg	41.5
				SD	3.1

rate=41.5L/hr

The medium size ACC sample used for the above calculation of the sampling rate was exposed to the BTEX for 5 hrs. The average sampling rate was found to be 41L/hr with a std dev. of 3L/hr

Table 6

Off-gassing Over Various Times

Samplers		benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
		(mg)	(mg)	(mg)	(mg)	(mg)	(mg)
C	5 h	0.208	0.230	0.076	0.168	0.077	0.046
E	5 h	0.255	0.290	0.093	0.200	0.094	0.058
		within 10% of each other (reproducibility)					
A	10 h	20.00	22.00	25.00	22.00	21.00	22.00
L	10 h	15.00	27.00	27.00	23.00	34.00	56.00
		The above results (A&L) show the % of increased uptake of BTEX with respect to C&E					
G	22 h	20.00	33.00	38.00	33.00	34.00	42.00
F	22 h	12.00	19.00	27.00	18.00	18.00	36.00
		The above results (G&F) show the % of increased uptake of BTEX with respect to C&E					
I	1 mon.	22.00	16.00	9.00	8.00	10.00	1.00
D	1 mon.						
		The above results (I&D)showed % of off-gassing with respect to C&E.					
5 PUF	5 h	nothing was present for either					
36 PUF	10 h						

Table 7

Comparison of ACC Samplers for Off-gassing

Samplers		benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
		(μg)	(μg)	(μg)	(μg)	(μg)	(μg)
B9(in bag)	5 hr exp.	288.30	327.20	113.50	100.50	94.27	98.75
C1(in air)	5 hr exp.	206.60	305.50	111.00	98.90	78.30	76.00
% Off-gassing		28.30	6.63	11.00	2.00	17.00	23.00
% Off-gassing/hr		1.45	0.34	0.56	0.10	0.87	1.20

Average % loss 0.51 μg/hr

Table 8

Loss of CS_2 From GC Vials

Weights (g):

Sample	Empty	0 hr	I hr	2 hr	4 hr	20.5 hr	24 hr	48 hr	1 week
1	27.733	30.256	30.256	30.256	30.256	30.250	30.249	30.250	30.192
2	28.016	30.794	30.790	30.787	30.785	30.781	30.780	30.754	30.648
3	25.979	28.486	28.486	28.487	28.486	28.485	28.485	28.477	28.417
4	26.389	29.232	29.232	29.232	29.232	29.232	29.232	29.233	29.232
5	26.246	28.743	28.742	28.741	28.741	28.740	28.740	28.723	28.605
6	27.237	29.736	29.735	29.734	29.732	29.711	29.706	29.676	29.524

Sample	CS_2 taken	Weight Change (g)						
1	2.5230	0.0001	0.0002	0.0002	0.0061	0.0070	0.0060	0.0638
2	2.7786	0.0046	0.0072	0.0094	0.0132	0.0140	0.0402	0.1466
3	2.5073	0.0000	-0.0002	0.0001	0.0011	0.0012	0.0092	0.0690
4	2.8425	-0.0001	-0.0003	-0.0002	-0.0002	-0.0001	-0.0013	-0.0003
5	2.4974	0.0020	0.0021	0.0025	0.0034	0.0038	0.0202	0.1381
6	2.4994	0.0007	0.0016	0.0038	0.0252	0.0297	0.0601	0.2114

Sample	% weight loss						
1	0.00	0.01	0.01	0.24	0.28	0.24	2.53
2	0.16	0.26	0.34	0.48	0.50	1.45	5.28
3	0.00	-0.01	0.01	0.04	0.05	0.37	2.75
4	0.00	-0.01	-0.01	-0.01	0.00	-0.05	-0.01
5	0.08	0.08	0.10	0.13	0.15	0.81	5.53
6	0.03	0.06	0.15	1.01	1.19	2.41	8.46

Table 9

Comparison of Syringe vs GC Vial Extraction Methods

AMOUNT OF BTEX FOUND

Sample	x10^{-3} (g)	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
new K (syr.)		1.30	2.91	0.84	0.65	0.78	0.57
new L (GC)		1.44	1.95	0.62	0.47	0.57	0.42
% L : K		111.25	67.10	73.00	71.96	72.32	72.81

Amount of CS_2 Evaporation With Syringe Method

TRIAL	CS_2 used (g)	CS_2 on cloth (g)	CS_2 in vial (g)	% sample recovered	% loss
1	2.481	0.544	1.450	80.29	19.71
2	2.467	0.505	1.511	81.65	18.35

CS_2 used = CS_2 used for the extraction
CS_2 on cloth = CS_2 left on the cloth after the extraction
CS_2 in vial = residual CS_2 that was collected from the extraction and placed in the vial
The above medium ACC samplers were exposed to BTEX for 19 hours,
at a relative humidity of 16%.

Table 10

Hole-punched Samples Using GC Vial Method

AMOUNT OF BTEX FOUND

Sample	$x10^{-4}$ (g)	benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
1		1.18	4.26	0.32	0.37	0.38	0.29
2		1.71	4.99	0.37	0.39	0.39	0.29
3		1.24	4.72	0.33	0.35	0.41	0.34
4		1.09	2.84	0.25	0.26	0.25	0.18
5		excl. {0.441}	2.86	0.16	0.20	0.21	0.17
	Avg	1.30	3.93	0.29	0.31	0.33	0.25
	SD	0.27	1.02	0.08	0.08	0.09	0.07
	%SD	21.08	25.98	28.33	26.71	27.89	29.87
6	(excluded)	1.63	1.08	0.24	0.23	0.22	0.20
7		0.86	0.58	0.14	0.14	0.15	0.22
8		0.88	0.58	0.14	0.15	0.16	0.15
9		0.81	0.53	0.13	0.13	0.14	0.14
10(1st inj)		0.88	0.56	0.14	0.15	0.15	0.15
10(2nd inj)		0.89	0.58	0.14	0.15	0.15	0.15
10(3rd inj)		0.88	0.57	0.14	0.14	0.15	0.14
	Avg	0.86	0.56	0.14	0.14	0.15	0.16
	SD	0.03	0.02	0.01	0.01	0.01	0.03
	%SD	3.50	2.26	4.38	3.97	3.80	18.98
Avg for #10 (1-3)		0.882	0.570	0.139	0.145	0.154	0.146
	SD	0.008	0.008	0.004	0.003	0.001	0.002
	% SD	0.89	1.43	2.53	2.10	0.38	1.43

The above 2 large samplers were exposed for 5 hours, at a relative humidity of 26%. The ACC samplers wer taken and randomly hole punched, so that 6 hole punches made up each of the above samples (ie 1-10). The samples were extracted using 1 mL of a CS_2/ Internal Standard mixture and shaken in GC vials for an hour. Samples 1-5 came from the first large ACC and samples 6-10 were taken from the second large ACC. Furthermore, sample 10 was injected 3 times to check for reproducibility within a sample.

Table 11

Comparison of 5, 15, and 30 min. Extraction Times Using the GC Vial Method

AMOUNT OF BTEX FOUND

Sample	x10^{-4} (g)	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
B (5 min)		4.84	5.67	1.66	1.41	1.28	1.15
14 (5 min)*		6.75	7.29	2.12	1.76	1.61	1.43
15 (15 min)		7.93	8.75	2.53	2.13	1.93	1.71
17 (15 min)*		6.56	7.62	2.24	1.86	1.72	1.51
19 (30 min)		7.44	7.80	2.25	1.88	1.71	1.53
20 (30 min)*		8.70	9.16	2.62	2.20	2.00	1.79

* = shaken

The above small ACC samplers were exposed to BTEX for 18 hours, at a relative humidity of 21%.

Table 12

Comparison of 15 and 30 min. Extraction Times Using the GC Vial Method: Pt. 1

AMOUNT OF BTEX FOUND

Sample	x10^{-4}(g) benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
1 (15 min)	2.52	2.42	0.68	0.63	0.66	0.60
2 (15 min)	2.02	2.48	0.93	0.93	0.87	0.85
3 (15 min)*	1.94	4.31	1.72	1.59	1.48	1.47
4 (15 min)*	2.30	5.60	1.80	1.65	1.48	1.45
5 (30 min)	1.69	3.01	1.58	1.52	1.34	1.39
6 (30 min)	2.33	4.17	1.62	1.51	1.32	1.34
7 (30 min)*	1.73	2.32	0.81	0.87	0.87	0.90
8 (30s min*	1.26	1.44	0.52	0.58	0.56	0.60
9 (30 min)*b	2.29	2.53	0.79	0.80	0.76	0.76
10 (30 min)*b	2.19	2.35	0.80	0.87	0.84	0.87
11 (30 min)*e	2.86	3.01	0.83	0.85	0.86	0.88
12 (30 min)*e	2.19	2.57	0.85	0.83	0.77	0.76

* = shaken

b = internal standard added at beginning

e = internal standard added at end

The above small ACC samplers were exposed to BTEX for 5 hours, at a relative humidity of 30%.

Table 13

Comparison of 15 and 30 min. Extraction Times Using the GC Vial Method: Pt. 2

AMOUNTS OF BTEX FOUND

CS_2 contained 0.000102g/g internal standard before extraction

Sample	$x10^{-4}$(g) benzene	toluene	ethylbenzene	p-xylene	m-xylene	o-xylene
1 (15 min)	7.11	7.30	2.02	1.91	1.41	1.55
2 (15 min)	8.92	10.80	2.46	2.30	1.81	2.01
3 (15 min)*	5.06	5.50	1.59	1.67	1.19	1.20
4 (15 min)*	5.52	7.02	2.19	2.23	1.74	1.78
5 (30 min)	6.83	7.73	1.99	1.95	1.69	1.75
6 (30 min)	7.00	7.05	2.58	2.94	2.38	2.75
7 (30 min)*	5.80	6.87	2.01	2.17	1.82	1.82
8 (30 min)*	5.10	4.92	1.46	1.59	1.19	1.27
9 (30 min)*b	5.47	5.23	1.28	1.27	0.74	1.04
10 (30 min)*b	5.05	4.88	1.42	1.53	1.08	1.17
11 (30 min)*#	1.49	1.51	0.47	0.52	0.42	0.46
12 (30 min)*e	1.66	2.03	0.57	0.56	0.46	0.50

* = shaken
b = internal standard was added at beginning
e = internal standard was added at end
# = 3.46mg/g IS added at the beginning

The above medium sized ACC samplers were exposed to BTEX for 5 hours, at a relative humidity of ~30%.

Table 14

30 min. Extraction Using the GC Vial Method

AMOUNT OF BTEX FOUND

Sample	$x10^{-4}$(g)	benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
1		5.74	12.58	5.48	5.10	4.77	3.69
2		3.93	4.58	1.42	1.44	1.12	1.19
3		3.80	4.18	1.29	1.38	1.11	1.31
4		3.45	3.60	1.14	1.21	0.99	1.11
5		5.47	6.17	1.83	1.80	1.38	1.44
6		4.21	4.59	1.57	1.69	1.33	1.52
7		5.14	5.89	1.77	1.81	1.38	1.47
8		4.26	5.43	1.65	1.67	1.29	1.38
9		4.17	4.84	1.51	1.63	1.30	1.45
10		6.18	7.41	2.16	2.17	1.64	1.74
11		4.55	5.44	1.83	1.85	1.38	1.46
12		4.55	5.25	1.84	1.94	1.59	1.65
Average BTEX Values and Standard Deviations							
Avg.		4.52	5.22	1.64	1.69	1.32	1.43
SD		0.80	1.04	0.29	0.27	0.19	0.18
%SD		17.60	20.01	17.63	16.04	14.73	12.71

The above medium-sized ACC samplers were exposed to BTEX for 5 hours at a relative humidity of 32%.

Table 15

One Hour Extraction Using GC Vial Method

AMOUNTS OF BTEX FOUND

Sample	x10^{-4}(g)	Benzene	Toluene	Ethyl-benzene	p-xylene	m-xylene	o-xylene
1		--	--	--	--	--	--
2		2.89	3.60	0.89	0.92	0.85	0.73
3		2.27	2.62	0.79	0.84	0.80	0.80
4		3.63	4.65	1.25	1.35	1.16	0.94
5		2.18	2.82	0.81	1.01	0.84	0.72
6		3.11	4.02	1.18	1.28	1.12	0.97
7		2.62	3.30	0.96	1.03	--	--
8		2.65	3.25	0.90	0.89	0.87	0.79
9		3.08	3.77	0.98	0.94	0.90	0.74
	Avg	2.80	3.50	0.97	1.03	0.93	0.81
	SD	0.48	0.66	0.17	0.18	0.14	0.10
	%SD	17.00	18.71	17.20	17.78	15.41	12.54

-- = excluded

The above small ACC samplers were exposed to BTEX for 5 hours, at a relative humidity of ~30%.

Table 16

Ultrasonics: Part 1

GC vials were used, along with 1 mL of CS_2 /IS, to extract BTEX from small ACC strips. They were put into an ultrasonic bath for various lengths of time, and then run on a GC.
8 small ACC strips with 5h exposure at ~6% R.H.

AMOUNT OF BTEX FOUND

SAMPLE	(mg)	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
1 (15)		1.71	1.42	0.24	0.67	0.46	0.56
5 (15)		1.55	1.45	0.23	0.67	0.45	0.45
2 (30)		1.22	1.00	0.16	0.47	0.33	0.44
6 (30)		1.69	1.41	0.22	0.63	0.41	0.39
3 (45)		1.69	1.37	0.23	0.63	0.41	0.39
7 (45)		1.55	1.29	0.20	0.56	0.38	0.34
4 (60)		1.65	1.33	0.21	0.60	0.39	0.36
8 (60)		1.60	1.32	0.20	0.57	0.38	0.37
	Avg	1.58	1.32	0.21	0.60	0.40	0.41
	SD	0.16	0.14	0.03	0.07	0.04	0.07

9 (30B)
10 (60B)
11 (0B)

There was no apparent degradation of the CS_2 or the IS in the ultrasonic bath.

TIMES:

15 = 15 min. in ultrasonic bath
30 = 30 min. in ultrasonic bath
45 = 45 min. in ultrasonic bath
60 = 60 min. in ultrasonic bath

0B = 1 mL CS_2 /IS in vial; no BTEX & no ultrasonic bath
30B = 1 mL CS_2 /IS in vial; no BTEX & 30 minutes in ultrasonic bath
60B = 1 mL CS_2 /IS in vial; no BTEX & 60 minutes in ultrasonic bath

Table 17

Ultrasonics: Part 2

In this experiment, six small ACC strips were exposed to BTEX for 5 hours, with a relative humidity of ~4.5 %. Three were extracted using the syringe method of extraction and the other three were put into an ultrasonic bath for 15 minutes. All of the samples were run on the GC.

AMOUNT OF BTEX FOUND

SAMPLE x10-4 (g)		benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
ultrasonic*							
1		2.87	2.40	0.50	1.23	0.92	1.18
2		2.78	2.32	0.48	1.21	0.98	1.26
3		3.07	2.63	0.57	1.39	1.12	1.44
	Avg	2.90	2.45	0.52	1.27	1.00	1.29
	SD	0.15	0.16	0.05	0.10	0.10	0.13
Syringe							
B		1.83	1.66	0.35	0.86	0.64	0.84
C	***	0.41	0.36	0.08	0.19	0.16	0.20
D		1.58	1.51	0.32	0.76	0.56	0.70
	Avg	1.70	1.58	0.33	0.81	0.60	0.77
	SD	0.18	0.11	0.02	0.08	0.06	0.11
R (SKC)		0.43	0.37	0.08	0.21	0.19	0.25

*** = excluded from average

* = 15 minutes in an ultrasonic bath.

SD = standard deviation

Table 18

Ultrasonics: 15 Minutes - Part 1 Top 3 ACC

The ACC-5092-10 and ACC-5092-20 cloths were received from American Kynol and cut several small strips. These strips along with the Porton Downs strips were then treated with CS_2. Next, the cloths were soaked in a 10% dimethyldichlorosilane/CS_2 mix and placed in an ultrasonic bath for 15 minutes. Lastly, the cloths were soaked in a 10% trimethylchlorosilane/CS_2 mix and placed in the bath for another 15 minutes. The cloths were dried on a hotplate for a day and a half.

Nine samplers (3 of each) were placed in the BTEX chamber for 5 hours, at a relative humidity of ~2%. An SKC tube was also placed in the chamber. All of the samplers were extracted with 1 mL CS_2 /IS mix.

AMOUNT OF BTEX FOUND

SAMPLE x10-4 (g)	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
ACC-5092-10						
1	0.56	1.02	0.54	1.16	0.74	0.76
2	0.21	0.65	0.63	1.32	0.88	1.10
3	0.32	0.98	0.80	1.70	1.15	1.34
ACC-5092-20						
4	0.99	0.78	0.19	0.50	0.35	0.53
5	0.45	0.37	0.09	0.08	0.21	0.14
6	1.12	0.95	0.21	0.24	0.58	0.38
Porton Downs						
7	3.93	3.33	1.22	2.01	1.53	1.63
8	10.37	9.04	2.10	4.73	3.50	3.37
9	9.92	8.08	2.01	4.17	3.20	3.43
SKC (R)	1.54	1.22	0.08	0.44	0.32	0.24
mg/m^3	5.90	4.70	0.31	1.70	1.20	0.92

As can be seen from the above results, there is inconsistency between the ACC strips of the same brand. However, more importantly, the amount of BTEX adsorbed onto the cloth (in ACC-5092-10 & 20) is extremely low. This may be due to the fact that there were several impurities on the cloths from the silylation process, and the cloths may not have been on the hot plate long enough. The Porton Downs ACC strips worked much better. All of the ACC strips will be put on another "hotter" hot plate for several days, and will be exposed again.

Table 19

Ultrasonics: 15 Minutes - Part 2 Top 3 ACC

The 3 different ACC strips (ACC-5092-10, ACC-5092-20 & Porton Downs) were exposed to BTEX for 5 hours and extracted using the ultrasonics method. The relative humidity was 0.5%.

AMOUNT OF BTEX FOUND

SAMPLE	x10-4 (g)	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
ACC-5092-10							
	1	5.34	4.79	1.11	2.52	1.90	1.68
	2	6.42	5.36	1.11	2.51	1.86	1.46
	3	3.49	2.83	0.44	1.20	0.90	0.68
ACC-5092-20							
	4	6.92	6.47	1.54	3.51	2.59	2.32
	5	7.41	6.45	1.38	3.16	2.37	2.16
	6	5.18	4.39	0.79	1.84	1.40	1.12
Porton Downs							
	7	7.19	6.29	1.31	3.03	2.33	2.20
	8	4.64	3.77	0.81	1.86	1.45	1.30
	9	8.28	6.75	1.39	3.27	2.49	2.10

There seems to be, as was seen in the first trial (Table 18), variation in the results. These cloths, although they are the small size, probably need 2 mL of CS_2 /IS to be extracted properly. The cloths are stiff and thick and do not get completely covered by 1 mL of the extracting solvent.

Table 20

Ultrasonics: 15 Minutes - Part 3 Top 3 ACC

In this experiment, 2 small size strips from each of the three ACC brands were exposed to BTEX, along with an SKC tube, for 3 hours at a relative humidity of ~0.5%. For this trial, we used 2 mL of CS_2 instead of 1 mL because the cloths were not being totally immersed in the 1 ml. They were put into an ultrasonic bath for 15 minutes and then run on the GC.

AMOUNT OF BTEX FOUND:

SAMPLE		benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
Porton Downs	$x10^{-4}g$	7.28	6.18	1.17	2.90	2.51	2.82
	g/h	243	205	39	96	84	93
	M/h	3.10	2.20	0.37	0.91	0.78	0.88
	SR L/h	97	91	90	86	81	80
ACC-5092-10							
1	$x10^{-4}g$	4.31	3.67	0.53	1.53	1.32	1.27
2		4.07	3.49	0.60	1.60	1.49	1.81
Avg		4.19	3.58	0.56	1.56	1.41	1.54
	g/h	140	120	19	52	47	51
	M/h	1.80	1.30	0.18	0.49	0.44	0.48
	SR L/h	55	53	43	46	45	44
ACC-5092-20							
3	$x10^{-4}$ g	4.33	3.68	0.71	1.77	1.59	2.04
4		5.47	4.52	0.85	2.13	1.89	2.71
Avg		4.90	4.10	0.78	1.95	1.74	2.37
	g/h	163	140	26	65	58	79
	M/h	2.00	1.50	0.24	0.61	0.55	0.75
	SR L/h	63	60	60	57	56	68
SKC (R)	$x10^{-4}g$	0.67	0.59	0.11	0.30	0.27	0.31
	mg/m^3	2.50	2.26	0.43	1.10	1.00	1.20
	M/m^3	33.0	25.0	4.1	11.0	9.8	11.0

Table 21

Ultrasonics: 15 Minutes - Part 4 Top 3 ACC

Adsorption of BTEX onto 3 different small ACC strips (ACC-5092-10, ACC-5092-20 & Porton Downs) at ~55%R.H. The three different types of ACC strips were exposed, in duplicate, for 5 hours in the BTEX chamber. The samplers were small in size and the relative humidity was ~ 55%, but we had some difficulties keeping it stable. The chamber humidity fluctuated throughout the exposure time between 75% RH and 45% RH. But, it stayed closer to 45% RH for the most part. To increase the humidity, we bubbled a saturated solution of calcium nitrate through the chamber and bypassed most of the drying systems. The samplers were extracted with 2 mL of CS_2 /IS mixture.

AMOUNT OF BTEX FOUND

SAMPLE	x10-4 (g)	benzene	toluene	ethyl - benzene	p-xylene	m-xylene	o-xylene
ACC-5092-10							
1		4.07	2.07	0.84	1.66	1.59	2.21
2		2.02	1.75	0.64	1.67	1.49	1.84
ACC-5092-20							
3		5.32	2.67	0.64	1.00	1.01	1.32
4	**	12.18	6.50	1.41	2.10	2.09	2.36
Porton Downs							
5	**	17.66	13.95	3.14	4.72	4.53	4.40
6		5.37	3.81	0.89	1.40	1.42	1.92
	g/h	108	76	18	27	28	38
	M/h	1.40	0.83	0.17	0.26	0.27	0.36
SKC Tube (C)	$x10^{-4}g$	0.785	0.406	0.109	0.185	0.241	0.456
	mg/m^3	1.80	0.93	0.25	0.43	0.55	1.05
	M/m^3	23.0	10.0	2.4	4.0	5.2	9.9
	L/h	60	81	70	66	51	37

** = excluded

Table 22

Ultrasonics: 15 Minutes - Part 5 Top 3 ACC

The three different types of activated carbon cloth were exposed to BTEX for 5 hours, at a relative humidity of ~50%. In this second trial, the chamber humidity was more stable than in the first trial. Each type of cloth was done in duplicate and an SKC tube was also exposed. The samplers were small in size and extracted, with 2 mL of CS_2 /IS, in an ultrasonic bath.

AMOUNT OF BTEX FOUND

SAMPLE	x10-4 (g)	benzene	toluene	ethyl - benzene	p-xylene	m-xylene	o-xylene
ACC-5092-10							
1		3.57	1.82	0.52	0.96	1.02	1.50
2		3.37	3.39	0.80	1.97	1.88	1.92
	Avg.	3.47	2.60	0.66	1.47	1.45	1.71
	g/h	69	52	13	29	29	34
	SR* L/h	16	24	26	42	45	53
ACC-5092-20							
3		5.44	2.64	0.71	1.04	1.15	1.57
4		6.66	3.65	1.07	1.36	1.45	1.98
	Avg.	6.05	3.15	0.89	1.20	1.30	1.78
	g/h	120	63	18	24	26	36
	SR* L/h	29	29	35	34	40	55
Porton Downs							
5	$x10^{-4}g$	4.22	2.74	0.64	0.82	0.87	0.88
6		5.58	3.52	0.97	1.28	1.40	1.95
	Avg	4.85	3.13	0.81	1.05	1.14	1.41
	g/h	9.7	62.0	16.0	21.0	23.0	28.0
	L/h	38	45	45	37	37	32
	SR* L/h	23	28	31	30	35	43
SKC (A)							
	$x10^{-4}g$	1.09	0.59	0.16	0.24	0.26	0.39
	mg/m^3	2.50	1.36	0.36	0.56	0.61	0.90
	M/m^3	3.2	15.0	3.4	5.3	5.7	8.3
Using data Table 35	mg/m^3	4.23	2.20	0.51	0.70	0.65	0.65

* Using BTEX concentration from Table 23 with relative humidity 8.5%.

Table 23

Ultrasonics: 15 Minutes - Part 6 Best 3 ACC

The samplers were the small size and were exposed to BTEX for 5 hours, at a relative humidity of ~8.5% They were then extracted with 2 mL of CS_2 /IS mixture and placed into the ultrasonic bath for 15 minutes.

AMOUNT OF BTEX FOUND

SAMPLE	x10-4(g)	benzene	toluene	ethyl - benzene	p-xylene	m-xylene	o-xylene
ACC-5092-10							
1		1.84	1.45	0.52	1.49	1.11	1.57
2		4.71	2.51	0.83	1.44	1.49	2.54
	Avg.	3.30	1.98	1.09	1.47	1.30	2.06
	g/h	66	39	22	29	26	41
	SR L/h	16	18	43	42	40	63
ACC-5092-20							
3		5.34	2.78	0.74	0.86	0.79	0.94
4		8.38	4.43	1.05	1.21	1.06	1.02
	Avg.	6.68	3.61	0.90	1.03	0.93	0.98
	g/h	140	72	18	21	19	20
	SR L/h	33	33	35	29	29	30
Porton Downs							
5		8.78	4.62	1.02	1.41	1.29	1.34
6		8.28	4.53	1.11	1.32	1.25	1.32
	Avg.	8.53	4.57	1.06	1.36	1.27	1.33
	mg/m^3	170	91	21	27	25	27
	SR L/h	40	41	42	39	39	41
SKC							
		1.84	0.97	0.22	0.30	0.28	0.28
	mg/m^3	4.23	2.20	0.51	0.70	0.64	0.64
	M/m^3	54	24	5	7	6	6

Table 24

Comparison of Different Types of ACC Fabrics at 19.5% Relative Humidity

AMOUNT OF BTEX FOUND

Sample	Name of Cloth	$x10^{-4}$ (g) benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
1 (B2)	ACC 507-20	1.50	1.45	2.19	0.39	0.47	1.21
2 (B3)	S&G RH3	1.57	1.48	2.33	0.40	0.49	1.21
3 (B4)	CCC AP1 880(1.0)	0.39	0.45	0.74	0.16	0.16	0.33
4 (B5)	ACC 5092-10	2.12	2.02	3.16	0.54	0.66	1.64
5 (B6)	CCC AP1 880(1.8)	2.68	2.60	3.92	0.68	0.85	2.08
6 (A1)	C-TEX STD. OPEN WEAVE	2.15	2.16	3.35	0.59	0.72	1.83
7 (A2)	* ACC 5092-15	1.46	1.43	2.20	0.38	0.46	1.19
8 (A3)	S & G STD. KNIT.	2.65	2.50	3.91	0.67	0.82	2.11
9 (A4)	ACC 5092-20	1.84	1.82	2.87	0.49	0.62	1.58
10 (A5)	C-TEX STD. WOVEN 2.50	2.38	3.70	0.64	0.77	1.97	
11 (A6)	ACC 507-10	1.51	1.43	2.24	0.40	0.51	1.27
12 (A7)	ACC PORTON DOWN	2.17	2.14	3.34	0.58	0.71	1.82

Best 5 cloths:

5 (B6) CCC AP1 880(1.8)
8 (A3) S & G STD KNIT.
10 (A5) C-TEX STD. WOVEN
12 (A7) ACC PORTON DOWN
6 (A1) C-TEX STD. OPEN WEAVE

* = 5g of CS_2 added instead of 2.5

The above medium sized ACC samplers were exposed to BTEX for 5 hours and then extracted using the GC vial method. The samples were shaken in the vials for 1 hour.

Table 25

Comparison of Different ACC Fabrics at 71% Relative Humidity

AMOUNT OF BTEX FOUND

Sample	Name of Cloth	$x10^{-4}$(g)	benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
1 (B2)	ACC 507-20		0.767	0.988	0.560	0.401	0.369	0.612
2 (B3)	S&G RH3		1.043	1.341	0.772	0.414	0.365	0.655
3 (B4)	CCC AP1 880(1.0)		0.121	0.237	0.114	0.111	0.069	0.069
4 (B5)	ACC 5092-10		0.081	0.102	0.064	0.029	0.026	0.047
5 (B6)	CCC AP1 880(1.8)		0.970	1.446	0.680	0.435	0.368	0.636
6 (A1)	C-TEX STD OPEN WEAVE		0.326	0.840	0.405	0.355	0.324	0.516
7 (A2)	ACC 5092-15		2.137	2.278	0.919	0.685	0.535	0.814
8 (A3)	S&G STD. KNIT.		0.876	1.280	0.684	0.408	0.356	0.640
9 (A4)	ACC 5092-20		2.469	2.848	1.746	1.008	0.859	1.566
10 (A5)	C-TEX STD WOVEN		1.121	1.517	0.613	0.490	0.391	0.647
11 (A6)	ACC 507-10		2.048	2.154	0.912	0.653	0.526	0.877
12 (A7)	ACC PORTON DOWN		0.910	2.016	1.190	0.721	0.584	1.027

TOP 5 CLOTHS

9 (A4) ACC 5092-20
7 (A2) ACC 5092-15
11 (A6) ACC 507-10
10 (A5) C-TEX STD WOVEN
2 (B3) S&G RH3

The above medium sized ACC samplers were exposed to BTEX for 5 hours and were extracted used the GC vial method. The samples were shaken in these vials for 1 hour.

Table 26

Comparison of Different ACC Fabrics at 28% Relative Humidity

AMOUNT OF BTEX FOUND

Sample	Name of Cloth	x10-4(g)benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
1 (B2)	ACC 507-20	3.696	4.053	1.040	0.982	0.887	0.950
2 (B3)	S&G RH3	2.145	2.938	0.829	0.705	0.662	0.822
3 (B4)	CCC AP1 880(1.0)	0.410	0.450	0.089	0.115	0.100	0.087
4 (B5)	ACC 5092-10	3.808	4.593	1.304	1.030	0.945	1.160
5 (B6)	CCC AP1 880(1.8)	2.651	3.257	0.674	0.662	0.582	0.596
6 (A1)	C-TEX STD. OPEN WEAVE	3.098	3.851	0.926	0.908	0.841	0.860
7 (A2)	ACC 5092-15	2.352	2.998	0.792	0.755	0.688	0.727
8 (A3)	S&G STD. KNIT.	4.021	4.359	1.098	1.094	1.048	1.160
9 (A4)	ACC 5092-20	3.524	3.714	1.070	0.984	0.908	1.108
10 (A5)	C-TEX STD. WOVEN	1.559	4.793	1.037	1.024	0.972	0.999
11 (A6)	ACC 507-10	4.233	4.423	1.036	0.988	0.925	0.986
12 (A7)	ACC PORTON DOWN	5.061	5.217	1.314	1.259	1.169	1.334

Best 5 cloths

12 (A7)ACC PORTON DOWN
8 (A3) S&G STD. KNIT.
4 (B5) ACC 5092-10
10 (A5) C-TEX STD. WOVEN
11 (A6) ACC 507-10

The above medium-sized ACC samplers were exposed to BTEX for 6 hours and extracted using the syringe method.

Table 27

Comparison of Different ACC Fabrics at 78% Relative Humidity

AMOUNT OF BTEX FOUND

Sample	Name of Cloth	x10-4 (g)	benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
1 (B2)	ACC 507-20		2.388	3.365	1.079	2.451	1.125	0.902
2 (B3)	S&G RH3		2.878	4.343	1.256	1.246	1.345	1.148
3 (B4)	CCC AP1 880(1.0)		0.239	0.509	0.104	0.141	0.138	0.115
4 (B5)	ACC 5092-10		7.637	8.479	2.365	2.287	2.450	1.980
5 (B6)	CCC AP1 880(1.8)		2.319	3.771	0.988	1.070	1.099	0.852
6 (A1)	C-TEX STD. OPEN WEAVE		0.279	0.652	0.195	0.217	0.238	0.208
7 (A2)	ACC 5092-15		1.929	2.401	0.645	0.686	0.739	0.626
8 (A3)	S&G STD. KNIT.		2.097	3.211	0.686	0.905	0.993	0.812
9 (A4)	ACC 5092-20		4.211	5.070	1.472	1.471	1.590	1.329
10 (A5)	C-TEX STD. WOVEN		1.346	1.994	0.668	0.697	0.783	0.661
11 (A6)	ACC 507-10		1.724	1.794	0.408	0.461	0.542	0.497
12 (A7)	ACC PORTON DOWN		3.276	5.465	1.397	1.562	1.7080	1.397

Best 5 cloths

4 (B5) ACC 5092-10
9 (A4) ACC 5092-20
12 (A7) ACC PORTON DOWN
2 (B3) S&G RH3
1 (B2) ACC 507-20

The above medium sized ACC samplers were exposed to BTEX for 5 hours and extracted using the syringe method.

Table 28

Different ACC In "New" Chamber

The ACC were placed in the new and improved chamber for 5 hours, at a relative humidity of ~30%.They were medium in size and were extracted using the syringe method

AMOUNT OF BTEX FOUND

SAMPLE	X10-4(g)	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
B2		2.09	2.53	0.67	0.70	0.83	0.59
B3		2.51	3.04	0.86	0.93	1.17	0.85
B4		-------	-------	NO RESULTS		--------	--------
B5		2.98	4.53	1.16	1.39	1.71	1.21
B6		0.46	0.52	0.12	0.14	0.20	0.16
R (SKC)		0.31	0.37	0.11	0.10	0.12	0.08
A1		4.46	5.89	1.55	1.56	1.85	1.25
A2		2.21	2.65	0.80	0.92	1.16	0.90
A3		2.68	3.11	0.84	0.93	1.16	0.83
A4		2.95	3.34	0.88	0.90	1.11	0.78
A5		2.22	2.68	0.67	0.64	0.77	0.49
A6		4.61	5.34	1.39	1.42	1.74	1.20
A7		4.31	5.41	1.49	1.55	1.89	1.33

Table 29

Comparison Of CCL* Fabrics In The Improved Chamber

SAMPLE	x10-4 (g)	AMOUNT OF BTEX FOUND benzene	toluene	ethyl - benzene	p-xylene	m-xylene	o-xylene
87 (B2)		2.43	2.59	0.71	0.72	0.82	0.55
87 (B3)		2.99	3.47	0.90	0.93	1.08	0.76
	Avg	2.71	3.03	0.80	0.83	0.95	0.66
	SD	0.40	0.63	0.13	0.15	0.18	0.15
88 (B4)		1.54	1.78	0.67	0.76	0.74	0.53
88 (B5)		5.83	7.20	2.58	2.36	2.49	1.69
89 (A3)		3.58	4.33	1.10	1.08	1.41	1.32
89 (A4)		4.97	5.72	1.53	1.57	1.78	1.19
	Avg	4.27	5.03	1.32	1.32	1.59	1.26
	SD	0.98	0.98	0.30	0.34	0.26	0.09
90 (A5)		1.94	2.23	0.56	0.59	0.68	0.53
90 (A6)		2.45	2.82	0.73	0.75	0.82	0.57
	Avg	2.19	2.52	0.65	0.67	0.75	0.55
	SD	0.37	0.42	0.12	0.12	0.12	0.02
R (SKC)		0.46	0.55	0.14	0.15	0.16	0.10
	mg/m^3	1.10	1.30	0.32	0.34	0.38	0.23
91 (P1)		3.06	3.84	1.02	1.03	1.28	0.80
91 (P2)		3.22	4.10	0.64	1.03	1.38	0.89
	Avg	3.14	3.97	0.83	1.03	1.33	0.85
	SD	0.11	0.19	0.27	0.00	0.07	0.07
92 (P8)		0.82	1.03	0.32	0.34	0.51	0.44
92 (P9)		1.04	1.31	0.42	0.42	0.55	0.43
	Avg	0.93	1.17	0.37	0.38	0.53	0.44
	SD	0.16	0.20	0.07	0.05	0.03	0.01
93 (A1)		2.00	2.73	0.74	0.75	0.95	0.61
93 (A2)		2.02	2.66	0.69	0.72	0.92	0.58
	Avg	2.01	2.70	0.71	0.74	0.94	0.60
	SD	0.01	0.04	0.04	0.02	0.02	0.01
S&G knitted CTex (B8)		2.24	2.98	0.85	0.89	1.16	0.74
S&G knitted CTex (B9)		1.86	2.35	0.64	0.66	0.86	0.56
	Avg	2.05	2.66	0.74	0.77	1.01	0.65
	SD	0.27	0.45	0.15	0.16	0.21	0.13

* CCL = Charcoal Cloth Limited; (xx) = frame number; SD = standard deviation

The CCL strips used were medium sized and were exposed to BTEX for 5 hours, at a relative humidity of ~33%. Strips were extracted using the syringe method.

Table 30

Comparison of the Top 3 ACC Cloths, an SKC Tube and a 3M Sampler*

TOP 3 ACC CLOTHS:

1) ACC -5092-10 2) ACC -5092-20 3) ACC Porton Down

The following medium sized samplers were exposed for 5 hours, at a relative humidity of ~30%. They were extracted using the syringe method.

AMOUNT OF BTEX FOUND

SAMPLE		benzene	toluene	ethyl - benzene	p-xylene	m-xylene	o-xylene
R (SKC)	mg	0.148	0.298	0.302	0.270	0.391	0.239
	mg/m^3	3.4	6.9	6.9	6.2	9.0	5.5
	M/m^3	43.6	74.5	65.5	58.6	84.8	51.8
3M	mg	0.096	0.190	0.020	0.018	0.026	0.017
SR	mL/min	35.5	31.4	27.3	27.3	27.3	27.3
	mg/m^3	9.0	20.0	2.4	2.2	3.2	2.1
	M/m^3	117.0	220.0	23.0	20.0	30.0	19.0
1)	mg	1.140	2.770	0.243	0.237	0.322	0.232
	mg/h	0.228	0.554	0.049	0.047	0.064	0.046
	mg/m^3	9.0	20.0	2.4	2.2	3.2	2.1
	SR L/h	25.0	28.0	20.0	22.0	20.0	22.0
2)	mg	0.692	1.470	0.176	0.167	0.227	0.187
	mg/h	0.138	0.294	0.035	0.033	0.045	0.046
	mg/m^3	9.0	20.0	2.4	2.2	3.2	2.1
	SR L/h	15.4	15.0	15.0	15.0	14.0	22.0
3)	mg	1.170	2.360	0.260	0.236	0.323	0.233
	mg/h	0.234	0.470	0.052	0.047	0.065	0.047
	mg/m^3	9.0	20.0	2.4	2.2	3.2	2.1
	SR L/h	26.0	24.0	22.0	21.0	20.0	22.0

* The results are higher than usual because the BTEX vials had refilled.
SR = sampling rate

Table 31

Comparison Of CCL Cloths At ~8% Relative Humidity

The medium sized CCL samplers were exposed to BTEX for 5 hours, at a relative humidity of ~8%. They were extracted using the syringe method.

AMOUNT OF BTEX FOUND

SAMPLE		x10-4 (g)	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
87*	[1]	++	1.21	0.97	0.14	0.45	0.55	0.61
88	[3]		3.50	2.96	0.26	1.05	1.17	0.77
89	[5]		9.35	7.48	1.08	3.03	3.27	2.08
90	[7]	++	1.74	1.47	0.30	1.72	0.88	0.86
91	[B1]		7.50	6.36	0.89	2.33	2.57	1.80
92	[K]		7.14	6.01	0.78	2.24	2.42	1.68
93	[L]		8.38	7.36	1.10	2.95	3.12	2.18
**	[N5]		7.01	5.92	0.73	2.12	2.43	1.72
	[N6]		5.84	4.83	0.66	1.85	2.04	1.41

* = fluorinated cloth

** = cloth currently used

++ = area of internal standard is unusually high and this is the reason for the low concentration of BTEX

Table 32

Aluminium Foil Wrapped Samples

AMOUNT OF BTEX	$x10^{-5}$ (g)	benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
Sample							
2 (B)		2.255	2.243	0.758	1.406	1.374	0.754
3 (SSI)		0.060	0.070	------	0.063	0.057	0.029
4 (SSO)		0.359	-----	0.265	0.548	0.892	0.749

B = sampler was placed in plastic bag only.
SSI = sampler was placed in plastic bag and then wrapped in aluminium foil with the shiny side in.
SSO = sampler was placed in plastic bag and wrapped in aluminium foil with the shiny side out.
----- = no results

These medium size ACC samplers were exposed to BTEX for 4.5 h at 28% relative humidity.

Table 33

Testing Of Aluminized Bags (From Ludlow Corp.)
For Permeation Of BTEX

SAMPLES

1 (sampler in bag and placed in chamber)

C (blank sampler left in bag for 1 week)

Sample 1 was placed in an aluminized bag and then put into the chamber for 18 hours.
Sample C was a blank sampler left in the aluminized bag for one week to make sure that the bag was not contaminating the ACC.

All of the above samplers were medium sized, and #1 was exposed to BTEX for 18 hours, at a relative humidity of ~12%. They were all extracted using the syringe method.

AMOUNT OF BTEX FOUND

SAMPLE	benzene	toluene	ethyl benzene	p-xylene	m-xylene	o-xylene
1			NO RESULTS			
C			NO RESULTS			

* = the attenuation on the GC was very low (ie. very sensitive) in this trial. Set at attenuation = 4 cf. 128.

Table 34

Determination of a New Sampling Rate for Medium ACC Strips at Very Low R.H. (~8%)

Four medium samplers were placed in the chamber for 23.5 hours, with a relative humidity of ~8%. Two of these samplers, along with an SKC tube, were immediately extracted using the syringe method and run on the GC. The last two samplers were stored in the aluminized bags, from the Ludlow Corp., in order to determine if there is any off-gassing.

AMOUNT OF BTEX FOUND

SAMPLE		(mg)	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene	
S			5.07	4.35	0.89	1.91	1.90	1.34	
T			4.21	4.71	0.97	2.10	2.11	1.47	
		Avg	4.64	4.53	0.93	2.00	2.00	1.41	
R (SKC)			1.31	0.71	0.13	0.30	0.31	0.22	
		mg/m^3	6.40	3.50	0.63	1.50	1.50	1.10	
C	one week in bag		2.17	4.47	1.04	2.26	2.31	1.64	*
J2	one week in bag		1.63	2.46	0.54	1.14	1.16	0.83	
		% off-gas	35.17	54.30	58.42	56.87	57.88	59.18	
SAMPLING RATE:		(L/h)							
S			13.13*	21.00	23.95	21.91	20.67	20.66	
T			10.911*	22.77	26.30	24.17	22.89	22.60	

* = excluded

		21.64
Rate of active sampling = 0.145L/h		23.75
	Average	22.69

Table 35

Permeation Through The New Winpak Bags

We received new samples from Winpak and they were tested to see if they were permeable to BTEX. They were 6" x 8.5" in size and aluminized on one side, while the other side was clear plastic. Since we have had problems in the past with permeation of BTEX through the bags, we wanted to test these new samples to see if the same permeation would occur. Three large samplers were placed into three Winpak bags, respectively, and placed in the environmental chamber for 23.5 hrs, with a relative humidity of ~0.5%. The cloths were extracted using the syringe method.

AMOUNT OF BTEX FOUND

SAMPLE	x10-4 (g)	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
1	att. 4	0.16	----	----	----	----	0.34
2	att. 8	0.27	----	----	----	----	0.20
3	att.16	0.15	----	----	----	----	0.06

att. = attenuation setting on the GC (regularly at 128)

Table 36

Effect Of Relative Humidity On Sampling Rate

	benzene	toluene	ethyl-benzene	p-xylene	m-xylene	o-xylene
Porton Downs						
0.5% Relative Humidity[1]	97	91	90	86	81	80
50% Relative Humidity[2]	38	45	45	37	37	32
50% Relative Humidity [3]	23	28	31	30	35	43
8% Relative Humidity[4]	40	41	42	39	39	41

1 = Taken from Table 20; values are suspect since they are 2x others obtained.
2 = Taken from Table 22; based on SKC at 50% relative humidity.
3 = Based on data from Table 22 for BTEX
using the SKC sampler data in Table 23 at 8.5% relative humidity.
4 = Taken from Table 23.

Figure 1

Typical Gas Chromatogram of BTEX

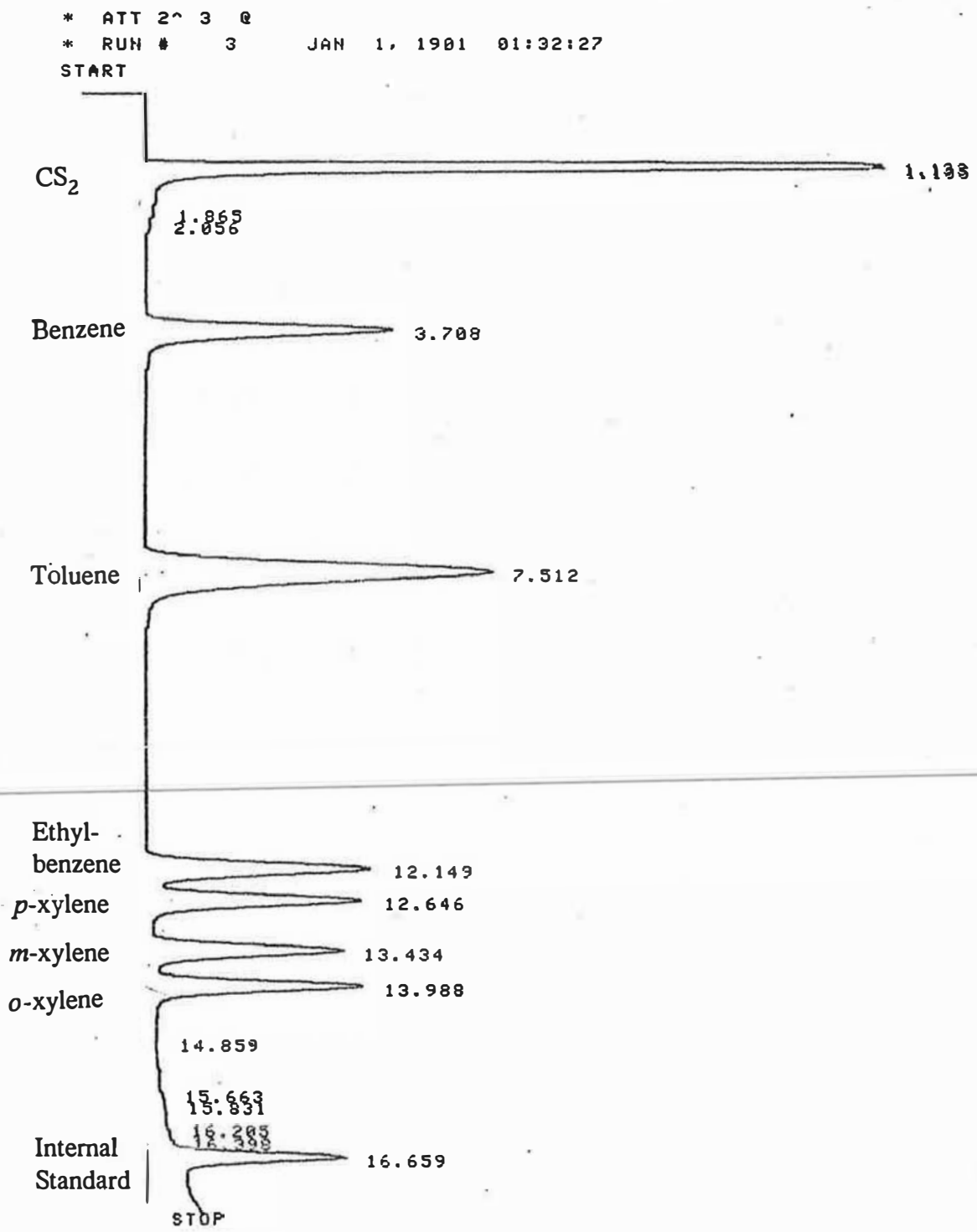


Figure 2

Sampling Chamber

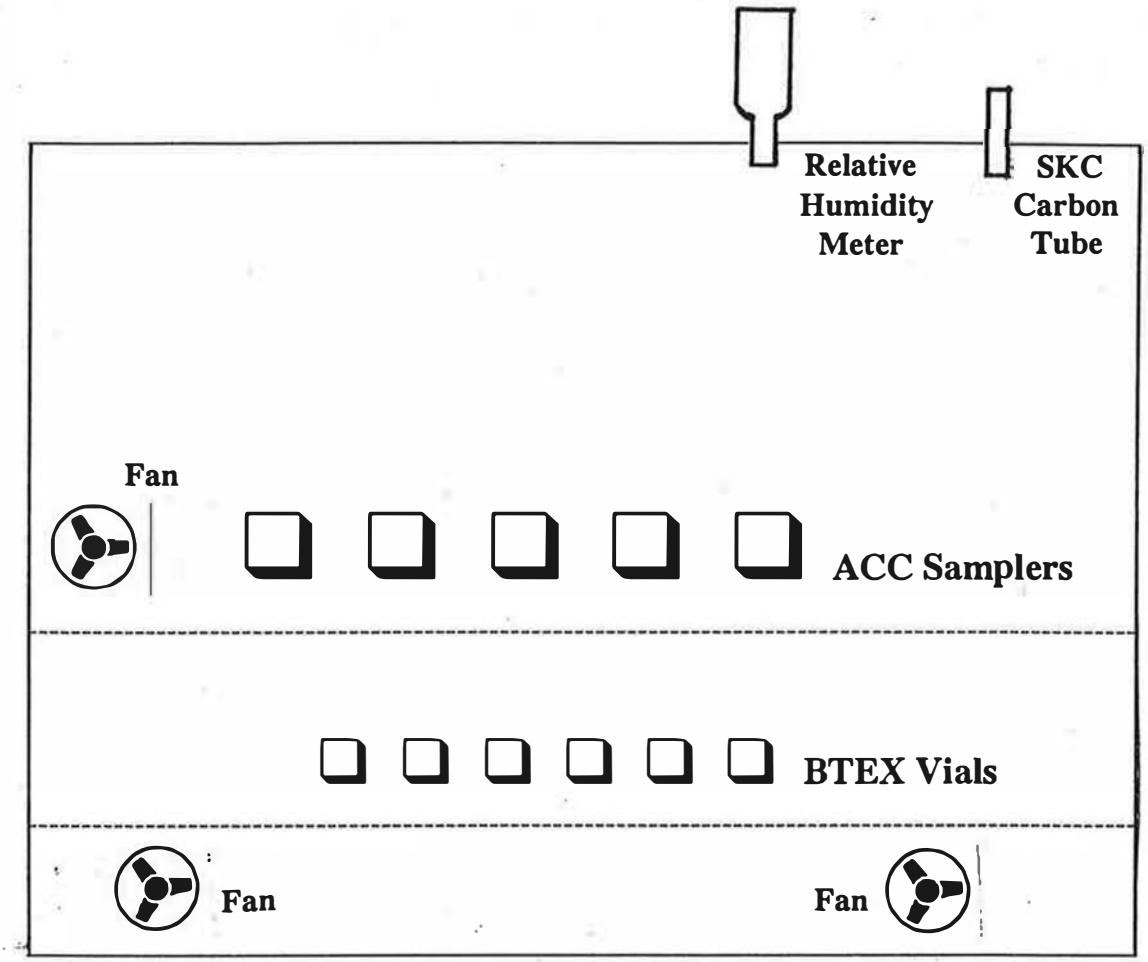


Figure 3

H_2O Adsorption on ACC at Different Relative Humidities

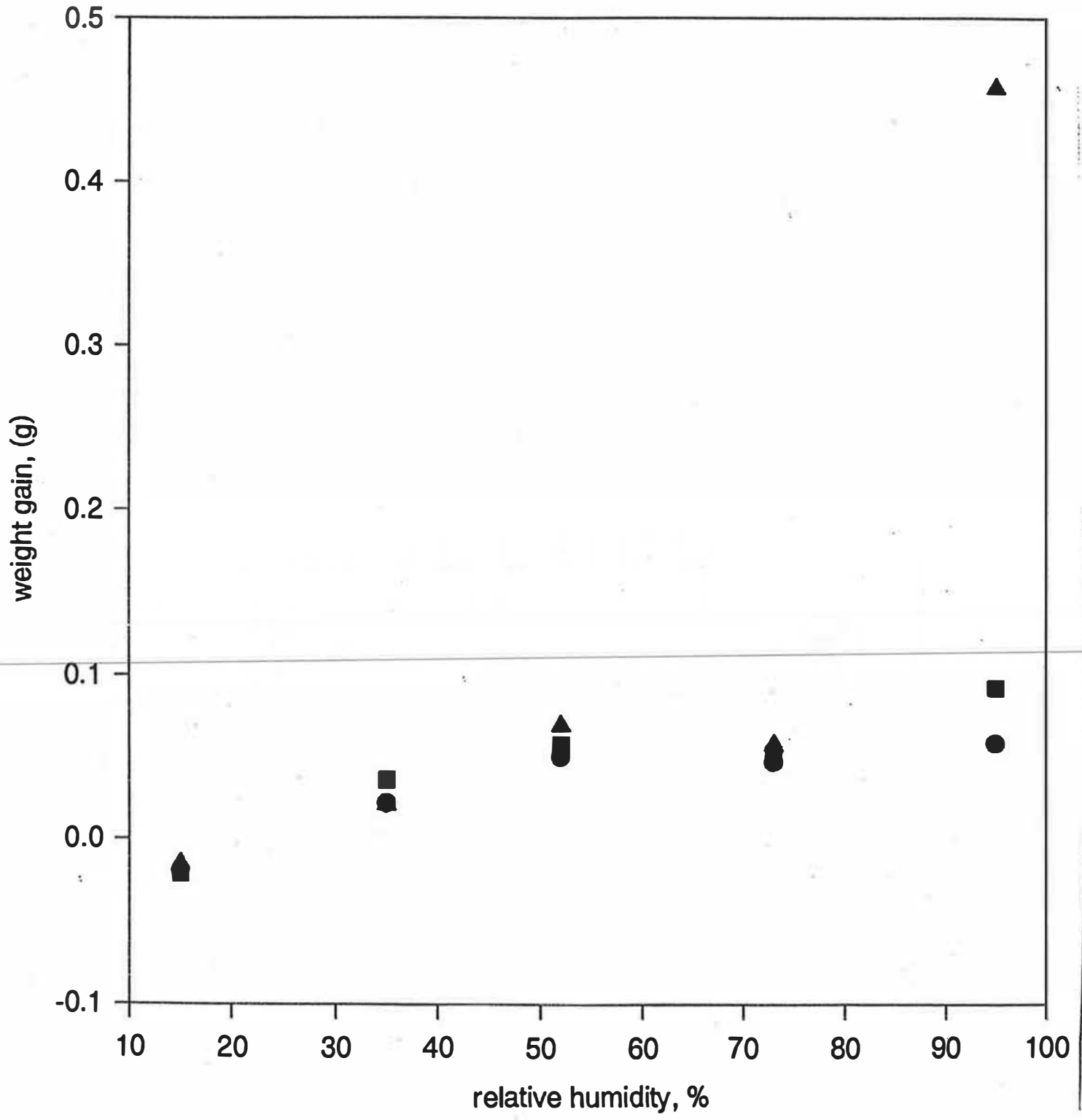


Figure 4

Different INCAT Carbon Types

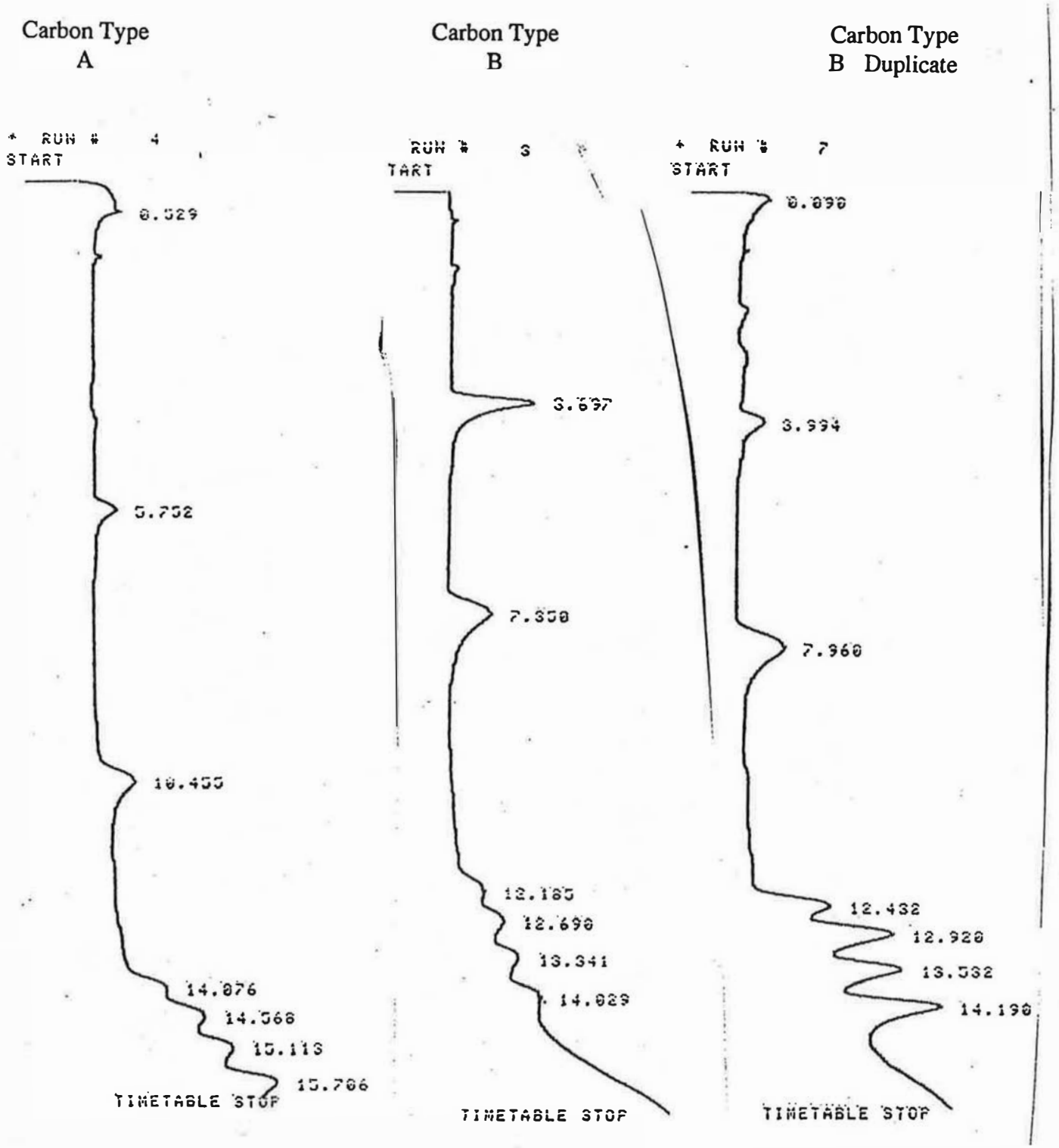


Figure 5a

GC-MS Results: Restaurant

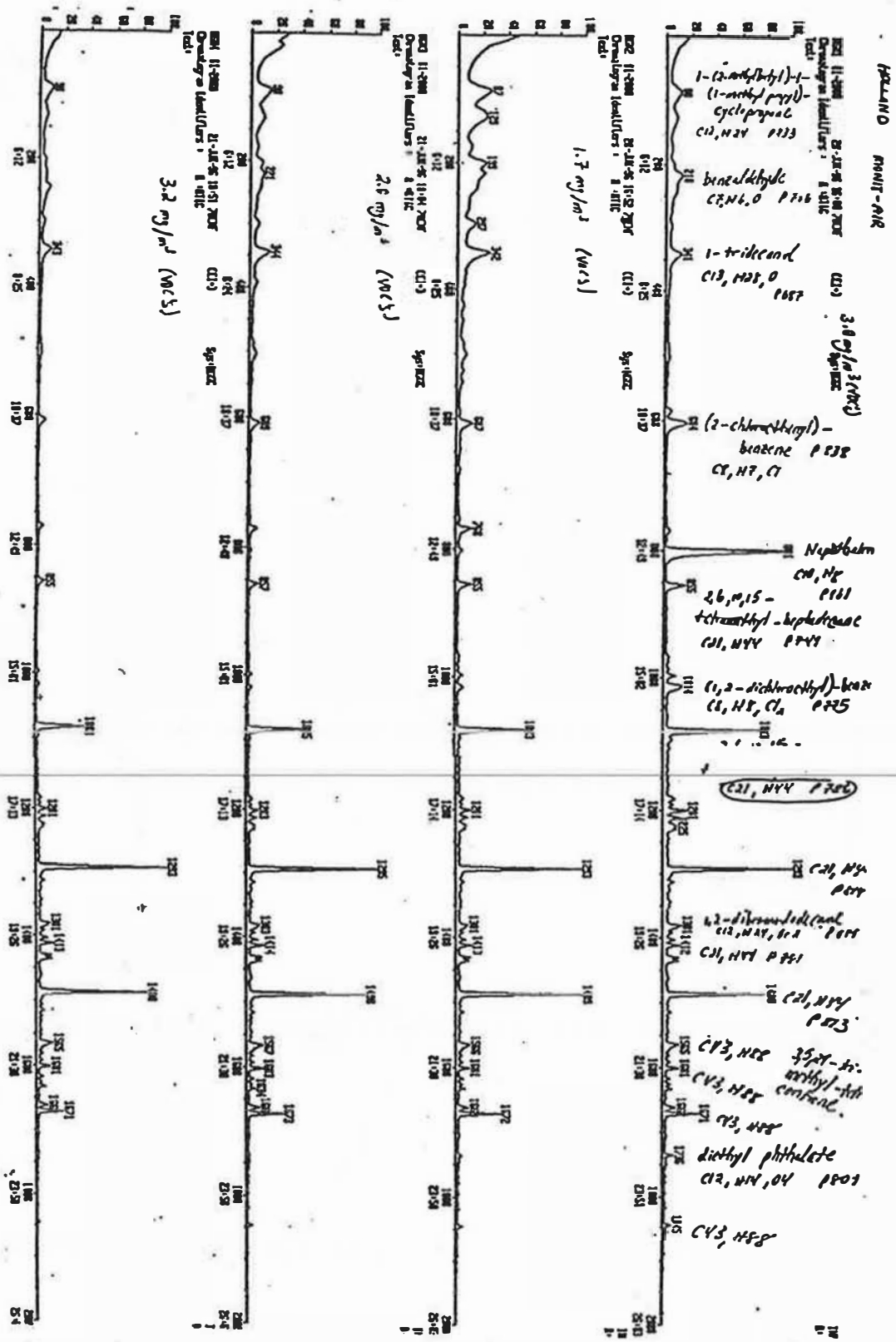


Figure 5b

GC-MS Results: Automobile

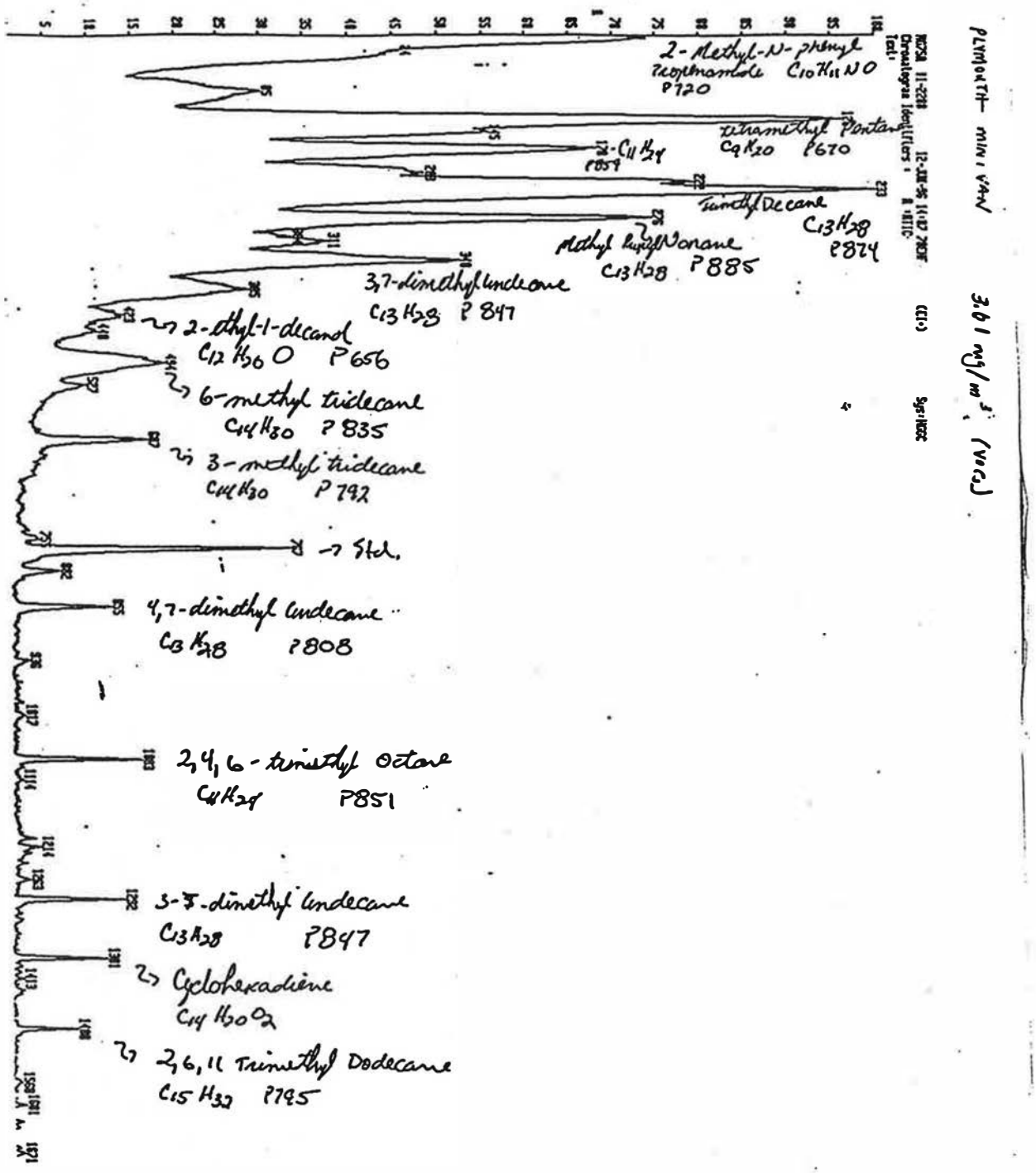
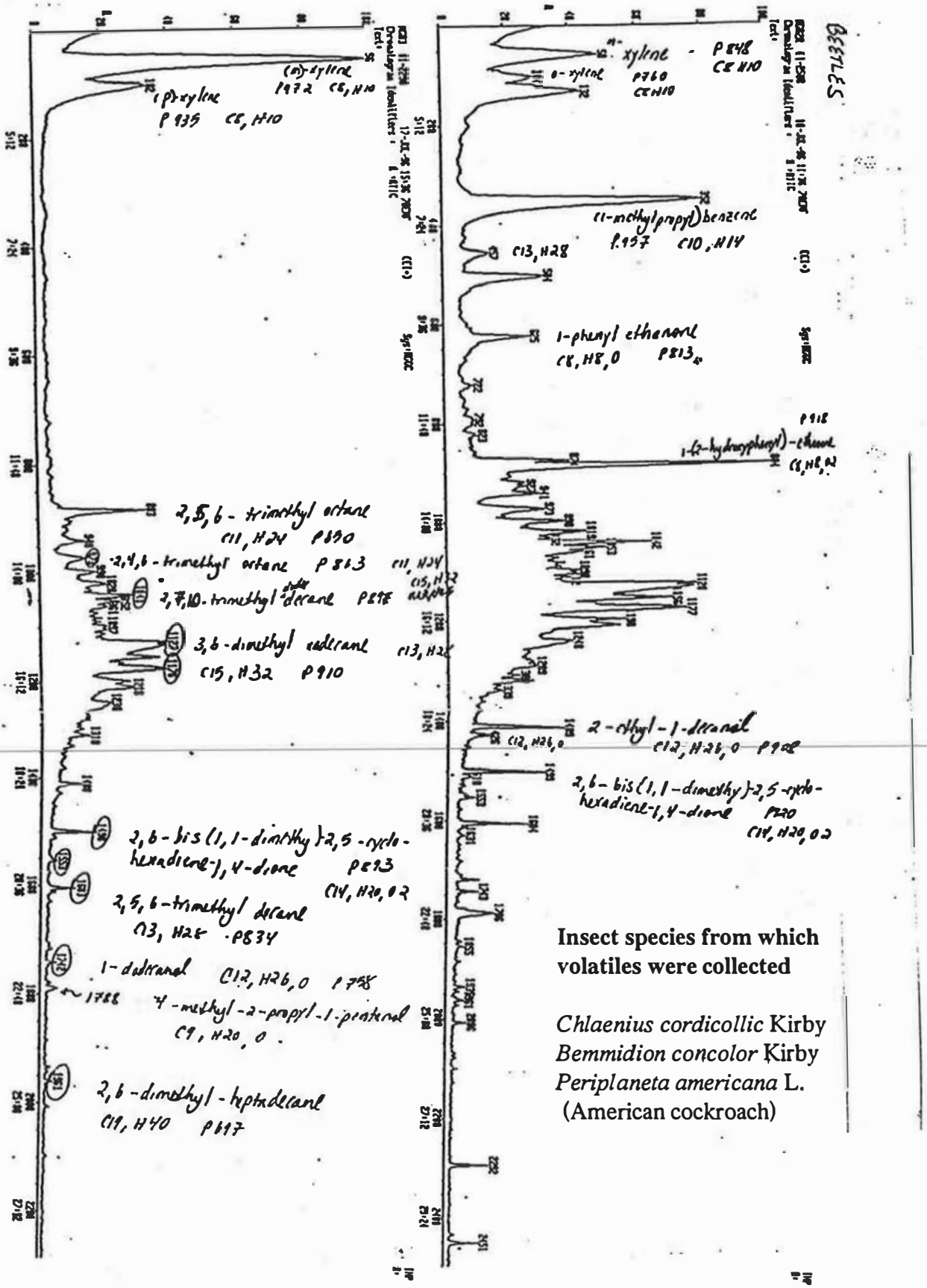


Figure 5c

GC-MS Results: Beetles



Insect species from which volatiles were collected

Chlaenius cordicollic Kirby
Bemmidion concolor Kirby
Periplaneta americana L.
(American cockroach)

Appendix A

INSIDE NEEDLE CAPILLARY ADSORPTION TRAP (INCAT)

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A novel method of solventless extraction has been developed based on a combination of solid phase micro extraction and purge and trap methods. In this technique, a hollow needle with either a short length of GC capillary column placed inside it, or an internal coating of carbon, is used as the preconcentration device. Sampling may be performed on ambient air, on solution, or the solution headspace, by passing the gas or liquid through the device either actively with a syringe, or passively via diffusion. The VOC are sorbed and concentrated onto either the carbon layer, or the liquid stationary phase of the capillary column, within the needle. Placing the needle into a heated GC injection port thermally desorbs the organic compounds directly into the GC without the need for solvent extraction. Results suggest that this procedure provides a rapid and sensitive alternative method to those currently available.

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INSIDE NEEDLE CAPILLARY ADSORPTION TRAP (INCAT)

Introduction

Volatile organic compounds (VOC) may be found as contaminants in both air and water, at concentrations ranging from 1 ppbv-1000 ppmv (part per billion/ million by volume).[1,2] The presence of these compounds in air has been linked to various health problems[3,4] making the analysis and monitoring of these compounds necessary.

Several methods exist for the analysis of VOC.[5] These methods include solid phase micro extraction (SPME)[6], purge and trap, activated carbon cloth (ACC,)[7,8] as well as commercial samplers manufactured by Perkin-Elmer,[9] SKC[10] and 3M.[11] Each of these methods is based on the sorption of the VOC by a solid or liquid sorbent followed by either solvent extraction, or thermal desorption prior to analysis by gas chromatography (GC).

Here we introduce a novel method for the sorption and solventless extraction of VOC followed by GC analysis. Inside needle capillary absorption trap (INCAT) is a technique that uses a hollow needle with either a short length of GC capillary column placed inside it, or an internal coating of carbon, as the preconcentration medium. Sampling may be performed on ambient air, on solution, or on the solution headspace, by passing the gas or liquid through the device actively with a syringe, or passively via diffusion. Organic compounds present in the sample are sorbed onto the deposited carbon sorbent, or the liquid stationary phase of the capillary, within the needle. The INCAT device with the sorbed organic compounds is placed into the injection port of a GC. The rapid heating of the metal needle induces the desorption of the organic compounds. This eliminates

the need for a solvent extraction step prior to analysis.

In this paper we introduce the INCAT technique for the analysis of VOC including BTEX compounds (benzene, toluene, ethylbenzene, and xylenes). The use of the INCAT device in either an active or passive sampling mode is demonstrated.

Experimental Section

Reagents. All reagents used in the preparation of solutions were analytical grade. Water used was purified with a Barnstead NanoPure™ water filtration system using a reverse osmosis treated feedstock.

Instrumentation. Gas chromatography was performed on a Varian Aerograph 2100 GC equipped with a flame ionization detector (FID) and a 30m x 0.25mm Supelcowax capillary column (Supelco). Analysis of the BTEX compounds was performed on a Hewlett Packard 5710A GC equipped with a FID. Separation was performed using a 2m x 3mm packed column; 5% bentone, 5% isodecylphthalate on Chromosorb W. Stainless steel capillary tubing used in the INCAT devices was purchased from Small Parts Inc.[12]

Preparation of the INCAT Device. The INCAT devices were prepared using tubing with inside diameters of 0.250 and 0.406 mm. A 75 mm length of steel capillary tubing was cut from stock material and the ends sanded smooth. The cut length of tubing was then pressed into the end of a common Luer-Lok fitting. The junction was sealed with 2 part epoxy cement. Within the needle, a 2.5 cm length of GC capillary column was inserted, and held in place by crimping the circumference

of the needle. The short length of capillary column with its internal coating of liquid stationary phase provides the media for the preconcentration of sampled VOC. As an alternate method, a coating of carbon was deposited within the needle to provide a media for the preconcentration of VOC. A diagram of INCAT devices with the two types of preconcentration media is given in Figure 1.

Sampling Protocol. Two types of samples were investigated in this work; ambient air and the headspace over an aqueous solution. With the INCAT device, sampling may be carried out in one of two ways, passively or actively. Passive sampling is performed simply by exposing the end of the INCAT device to the sample allowing the sample to diffuse into the needle and onto the sorbent within. Active sampling (Figure 2) is performed by drawing the sample through the device with either a syringe or pump. In both cases, the VOC are sorbed and concentrated onto the inner surface of the device. Chromatographic analysis of sorbed VOC is performed by capping the end of the INCAT device at the Luer-Lok fitting and then placing the needle into the heated injection port of a GC. Thermal desorption of the VOC then occurs directly within the injection port allowing chromatographic analysis. A desorption temperature of 175 °C was used with the time of desorption fixed at 15 seconds. A cleaning step involving heating the INCAT device to temperatures greater than 175 °C between injections eliminated the possibility of sample carryover.

Results and Discussion

The INCAT technique is based upon the premise that if one could expose a short length of a GC capillary column to a sample of interest and re-connect the column to the GC for analysis, then a very easy and sensitive means of analysis may be obtained. INCAT devices were thus constructed

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with 2.5 cm lengths of GC capillary columns and tested. Preliminary results indicated that the devices performed well. A chromatogram obtained from the headspace sampling over a saturated solution of benzene in water is given in Figure 3. The chromatogram shows only the peak for benzene (Rt=5.4 minutes) and no peak due to the presence of water in the sample injected. A large degree of peak tailing was observed with the initial INCAT devices. This indicated that the transfer of heat from the injection port of the GC to the capillary within the INCAT device was a relatively long process. Initial experiments performed indicated that the time and temperature of desorption were optimised at 15 seconds and 175 °C. Desorption temperatures lower than 175 °C and desorption times of less than 15 seconds resulted in peak tailing due to insufficient heating of the INCAT device within the injection port. Desorption temperatures greater than 175 °C or desorption times longer than 15 seconds did not result in any significant improvement. This resulted in less than a 5% carryover during replicate sampling. A cleaning step eliminated any possible sample carryover between injections.

Both passive sampling and active sampling were investigated. Passive diffusive sampling was investigated as it has the same accuracy in analysis as active sampling but does not require the use of a syringe or pump.[13] The analyte is allowed to passively diffuse into the needle and concentrate onto the sorbent within. The limiting step in this case is the diffusion of the analyte from the sample through the end of the needle, a process which may be used to calibrate the passive monitoring of VOC in ambient air.[14] With active sampling the analyte is drawn through the INCAT device at a fixed rate as indicated in Figure 2. This results in the active transfer of the analyte from the sample through the INCAT device and effectively eliminates the time required for the analyte to diffuse from the sample to the preconcentration media. Active sampling increases the speed of analysis considerably

compared to passive diffusion sampling.

The precision of replicate sampling was examined for the passive sampling of the headspace over a saturated solution of benzene in water. The results are presented in Table 1 for sampling times of 3 minutes. The error involved, ~ 7%, was attributed primarily to differences in the manual sampling technique in replicate injections. This could be improved with automation. However, this precision is sufficient to allow the application of the INCAT device for the passive monitoring of VOC in ambient air.[15]

The headspace of a saturated solution of benzene in water was sampled passively for increasing periods of exposure up to 45 minutes. The amount of benzene sorbed was found to be dependent on the time of exposure. The results are presented in Figure 4. Initially, a linear correlation is observed with the concentration of benzene in solution, up to ~ 5 minutes of exposure (R^2=0.998). Overall, the sorption profile indicates that after 45 minutes saturation or equilibrium has not been reached. This indicates that long exposure times may be possible in an environmental setting without the problems associated with saturation.

The dependence of sampling on the concentration of an analyte in solution was investigated. Active sampling of the headspace over solutions containing increasing amounts of 1,1,1-trichloroethane in water was performed. Approximately 50 mL of the headspace was passed through the INCAT device over a 60 second interval using a gas-tight syringe. The 50 mL sample and the 60 second time interval were arbitrarily chosen to ensure that enough sample passed through the INCAT device for analysis. Other sample sizes and times were not investigated at this time. A linear correlation was observed with respect to the initial concentration in solution, R^2=0.998 (Figure 5). These results indicate that quantitative analysis is possible with the INCAT device in an active

sampling mode. The active mode of sampling was found to be superior to the passive sampling mode in the case of headspace sampling. This was due to the shorter time required to take up a sufficient amount of sample for analysis by GC in the active mode. In passive sampling the rate of diffusion of the analyte through the end of the needle is slow relative to the rate of analyte transfer within the needle itself during active sampling.

Use of carbon as the extraction media was investigated because construction of the INCAT devices with the internal piece of capillary column was found to be difficult. The choice of a carbon sorbent was based upon a number of criteria, foremost of which was the experience in our laboratory with carbon-based passive monitors for VOC in air.[7,8] As well, carbon as a sorbent has several advantages over that of a liquid stationary phase. Carbon fibres exhibit a high level of saturation (> 20% weight of fibre)[16,17] which is greater than the liquid stationary phase available with a short length of GC column. Activated carbon monitors have been found to be essentially independent of temperature and pressure fluctuations during sampling.[18-20] Use of the carbon coating also resulted in an improvement in the desorption of VOC from within the INCAT device. This was attributed to an increase in thermal conductivity of the thin carbon film in comparison to the relatively thick fused silica layer and liquid stationary phase of the CG column.

A carbon-coated INCAT device was compared with an uncoated INCAT device for the analysis of the headspace over a saturated solution of BTEX compounds. Sampling was performed actively by passing ~ 5 mL of headspace over a 60 second interval using a gas-tight syringe. A 1.0 µL direct injection of the aqueous sample was also performed for comparison. The results in Figure 6 illustrate that the coated INCAT device performed well in comparison with the uncoated device and the direct injection. The uncoated INCAT device was essentially used as a blank, demonstrating the

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effectiveness of the carbon-coated INCAT device. The peak due to the presence of water in the direct injection (Rt=2.5 minutes) is absent in the chromatograms of both the INCAT injections indicating that water is not taken up by the INCAT device in an observable amount.

The possibility of using the carbon-coated INCAT device as a passive monitor for VOC in air was examined. An INCAT device was left exposed to ambient air at various locations within our laboratory and then analysed by GC. Sampling was performed passively with the duration of exposure fixed at 24 hours. The chromatogram is given in Figure 7.A. Several similar chromatograms were obtained at different sampling locations within the laboratory. A chromatogram derived from exposing one of the INCAT devices to the inside of a solvent storage cabinet for 2 hours is given in Figure 7.B. These results indicate that a number of compounds may be taken up by the device in sufficient quantities for analysis by GC. This also demonstrates the feasibility of using the INCAT device as a method for the passive monitoring of VOC in air.

It is noted that these results are still preliminary. However, some of the possible advantages of such a device are given here. The INCAT device is mechanically simple with no moving parts and inexpensive to produce. The use of solventless extraction ensures maximum sensitivity in analysis. The active mode of sampling with its short diffusion path length may provide for more rapid analysis times in the laboratory as compared with passive diffusive sampling. The passive mode of sampling in conjunction with a high degree of saturation using a carbon coating and a 7% error in replicate measurements would allow for the passive monitoring of VOC in the environment.

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Conclusions

Results suggest that the INCAT device may provide for a rapid and sensitive alternate method for the analysis of VOC in both air and water samples in either an active or passive sampling mode. We are currently investigating the physical characteristics and possible applications of this device.

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Table 1. Reproducibility in replicate measurements using the INCAT device. Headspace sampling of a saturated solution of benzene in water. Conditions: passive sampling, 3 minutes exposure.

Trial #	Net Peak Area
1	254234
2	287986
3	309464
4	266401
Mean Area	276897
SD	19583
RSD	7.1%

Figure 1. Diagram of INCAT devices with A; 2.5 cm length of GC column, and B; carbon.

Figure 2. Diagram showing the active mode of sampling using the INCAT device over the headspace of a solution.

Figure 3. Chromatogram from the headspace sampled over a saturated solution of benzene in water. Conditions: Passive sampling of headspace for 7 minutes.

Figure 4. Sorption characteristics with respect to the time of sampling over a saturated solution of benzene in water. Conditions: passive sampling of headspace.

Figure 5. Concentration dependence on sampling of 1,1,1-trichloroethane. Conditions: active sampling, 50 mL of headspace withdrawn during 80 seconds.

Figure 6. Comparison between A; carbon coated INCAT device, B; blank needle and, C; 1.0 mL direct injection of BTEX compounds. Conditions for A and B: active sampling, 5 mL headspace withdrawn during 60 seconds; saturated solution of BTEX compounds in water; equivalent GC parameters.

Figure 7. Chromatograms from a carbon coated INCAT device exposed to ambient air. Sampling locations were: A; the laboratory and B; solvent storage cabinet. Conditions: passive sampling for 24 hours and 2 hours respectively.

Fig. 1

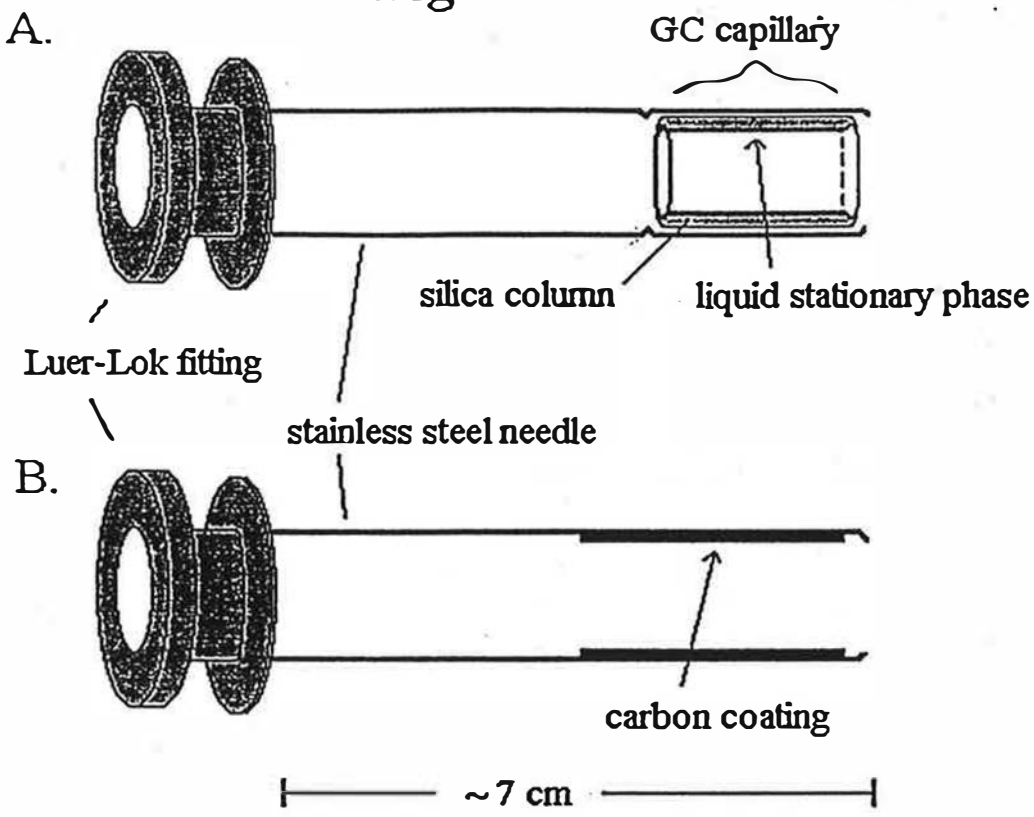

A.
GC capillary
silica column
liquid stationary phase
Luer-Lok fitting
stainless steel needle
B.
carbon coating
~7 cm

Fig. 2

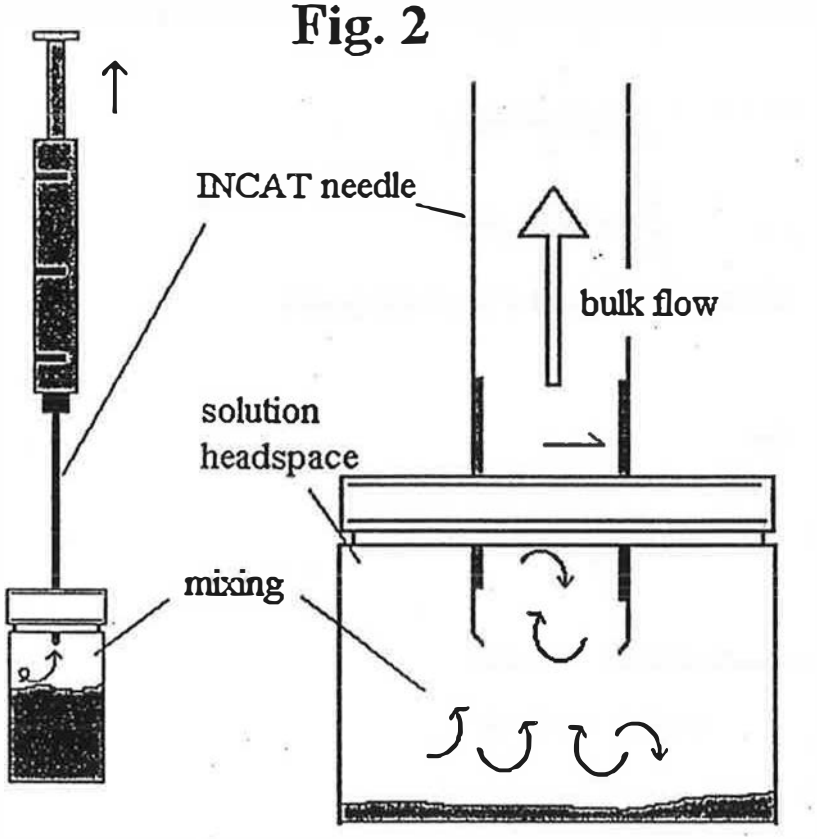


Fig. 3

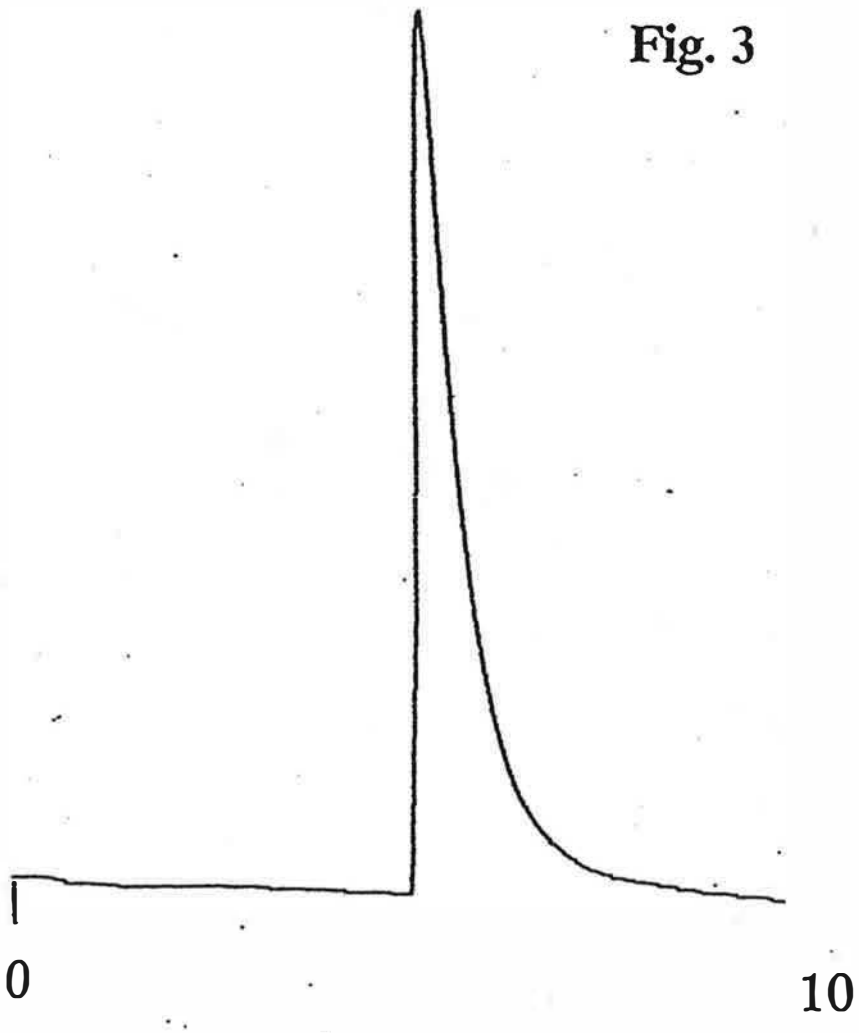


Fig. 4

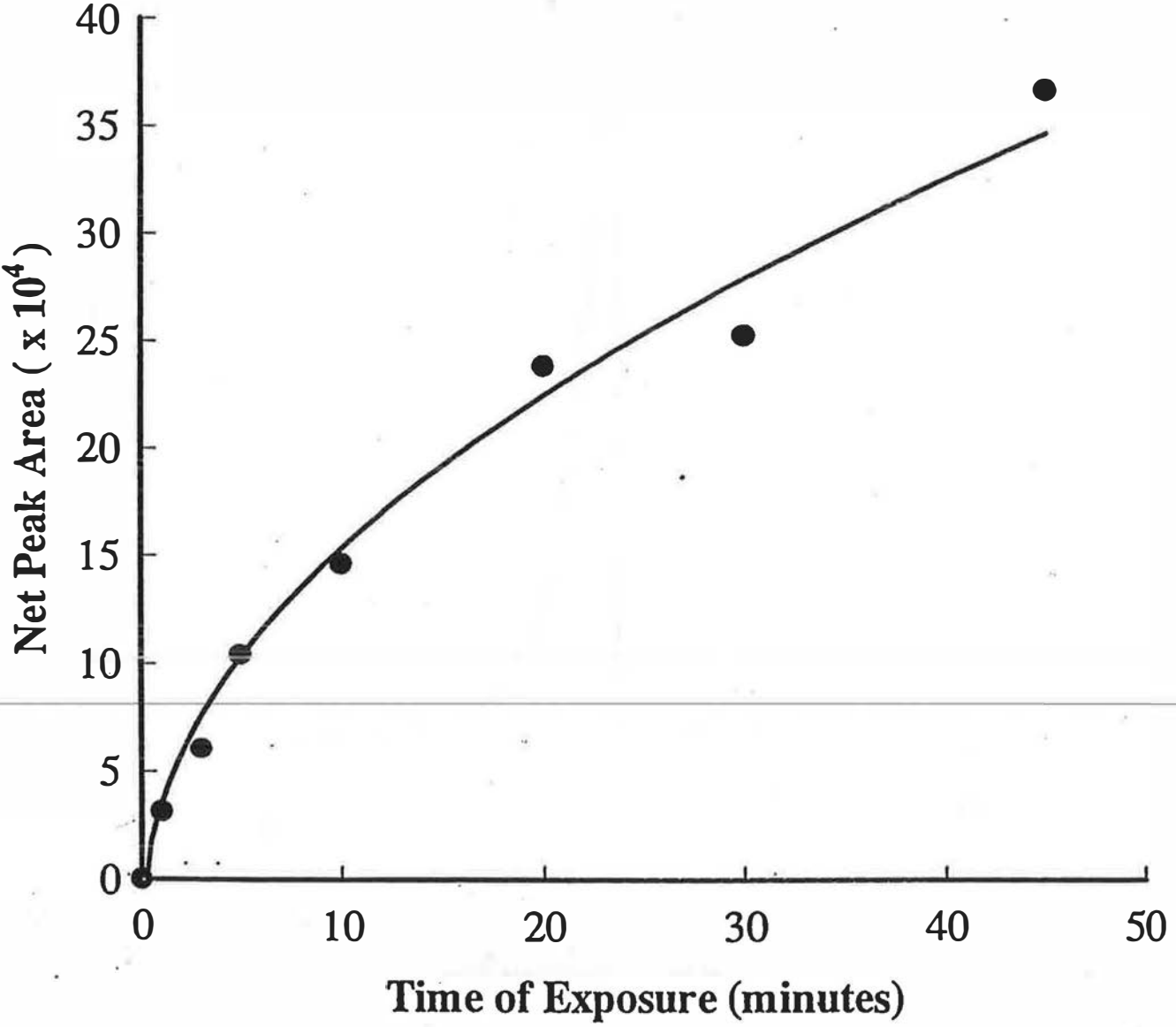
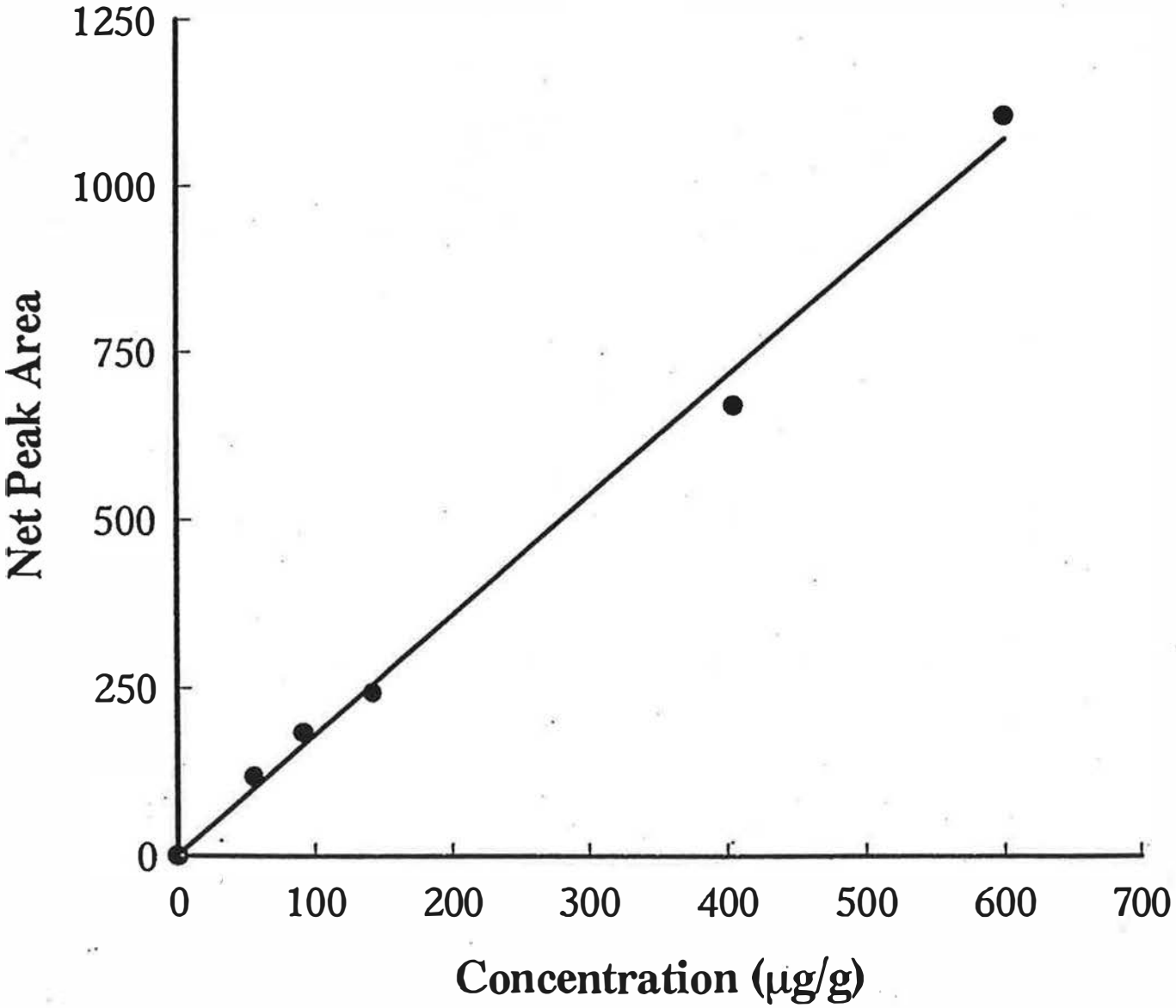

Net Peak Area (x 10⁴)
40
35
30
25
20
15
10
5
0
0
10
20
30
40
50
Time of Exposure (minutes)

Fig. 5


1250
1000
750
500
250
0
Net Peak Area
0
100
200
300
400
500
600
700
Concentration (μg/g)

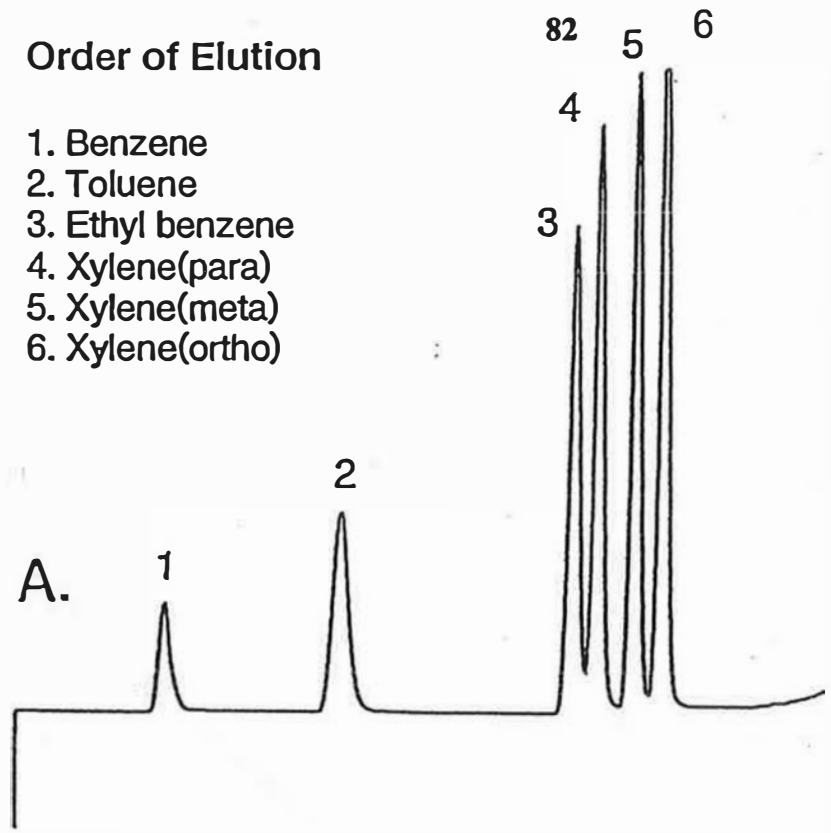
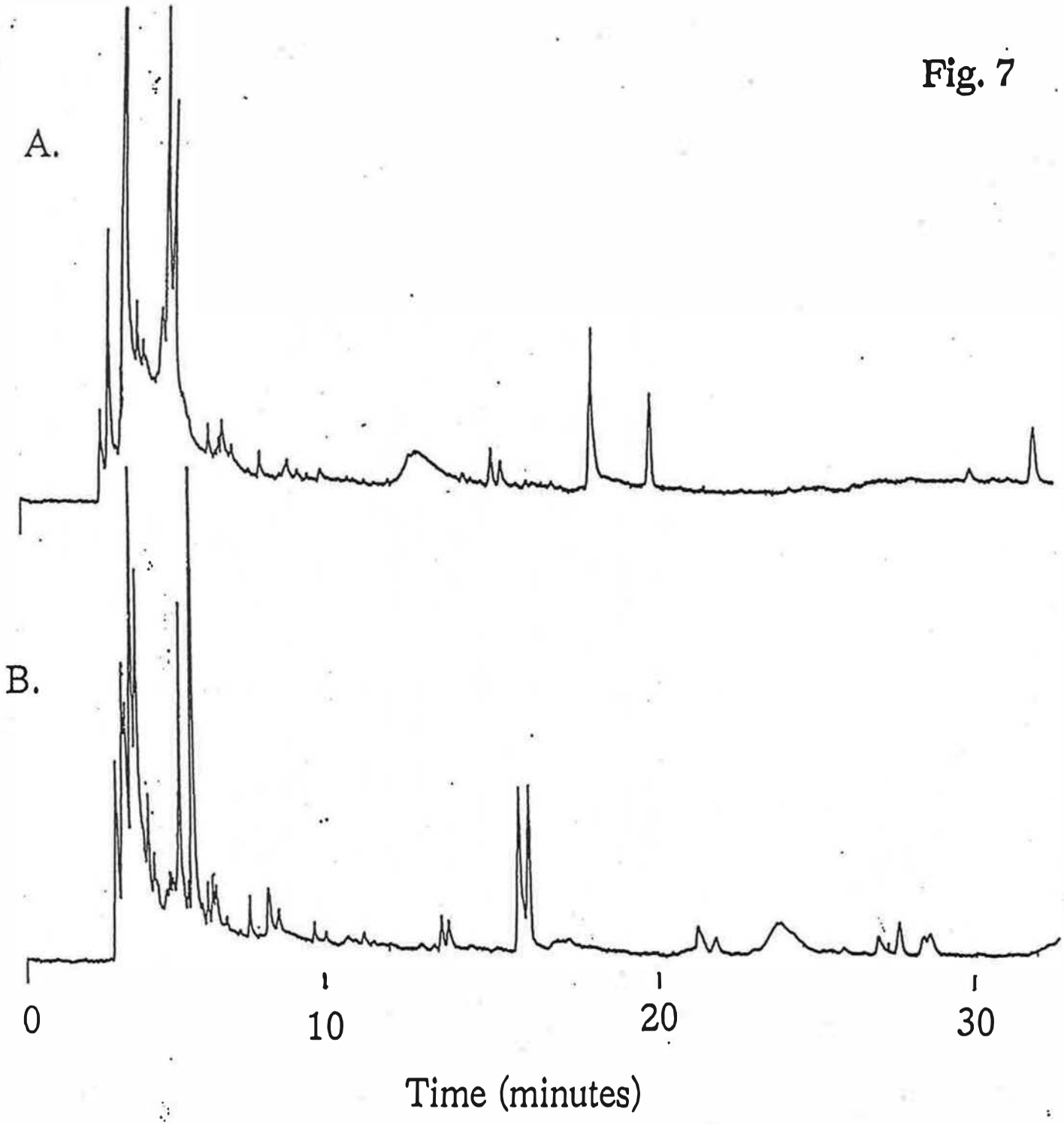

Order of Elution
1. Benzene
2. Toluene
3. Ethyl benzene
4. Xylene(para)
5. Xylene(meta)
6. Xylene(ortho)
A.
1
2
3
4
5
6
B.
C.
5
10
15
Time (minutes)

Fig. 6

Fig. 7



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September 19, 1996

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Dear Duncan,

Re: VOC Comparison Tests

During the house monitoring this summer, ORTECH was approached by the Chemistry Department of the University of Manitoba to deploy a passive VOC monitor developed by Dr. H.D. Gesser. Dr. Gesser had talked to V. Salares who mentioned we were conducting VOC sampling in 6 houses and to contact ORTECH. We agreed to deploy these samplers at the same time as the project VOC sampling.

Samplers were sent to ORTECH by Dr. Gesser and installed during the monitoring of the last house in July 1996. Two other passive monitors, 3M and SKC, were also installed at the same time. This testing allowed the comparison of the Manitoba sampler with other passive methods and the active system used on the project. All methods were deployed for the same 7-day period. The results are as follows:

VOC Test Method	Location	Results, TVOCs (mg/m^3)
Active Sampling/GS/MSD	Furnace Return Air	6.64
University of Manitoba	Kitchen Master Bedroom Basement	1.59, 1.19 1.50, 1.40 1.83,1.43
3M Badge	Kitchen Master Bedroom	4.92 5.40
SKC Badge	Kitchen Master Bedroom	5.69 5.20

The 3M and SKC results are in very good agreement which are in good agreement with the active sampling project method. The U of Manitoba method results are lower that the other three methods. The sampling location and collection and analysis techniques are different between the active project methods and the passive methods. This may explain the difference between the passive 3M and SKC methods and the project method. I do not know enough about the University of Manitoba method to comment on the lower values obtained.

This comparative sampling does confirm that the VOC levels found in the first two test periods are above 1 mg/m^3.

Yours truly,

Peter Piersol
Air Quality Section
Environmental Assessment Technologies

cc: Dr. H.D. Gesser, University of Manitoba
PP:or

Mr. Peter Piersol
Air Quality Section
Ortech Corporation
Mississauga, Ontario
L5K 1B3

September 10, 1996

Dear Mr. Piersol,

The eleven samplers were solvent extracted and analysed for the volatile organic compounds (VOCs) that may cause health problems. Six of these samplers had been exposed and the remaining five were blanks. The analysis was done using a gas chromatography / mass spectrometry system and the chromatograms are enclosed. The results of the analysis are summarized below for you.

The results of the samplers are as follows, based on an average molar mass of 200g/mol :

1,2	Kitchen (D5 & D16)	1.59 mg/m^3 , 1.19 mg/m^3	
3,4	Master Bedroom (D6 & D2)	1.50 mg/m^3 , 1.40 mg/m^3	
5,6	Basement (D8 & D11)	1.83 mg/m^3 , 1.43 mg/m^3	
7	BLANK	0.212 mg/m^3	
8	BLANK	0.122 mg/m^3	
9	BLANK	0.050 mg/m^3	average ~ 0.116 mg/m^3
10	BLANK	0.115 mg/m^3	
ii	BLANK	0.082 mg/m^3	

The total VOCs present for the cloth samplers is above normal and in the range where irritation has been proven to occur. The blanks were below the range, which is what was expected. We have some indication that the adsorbed VOC will migrate from one bag to another and may account for the high blank values. We now cover the plastic bags in aluminum foil to prevent loss and migration. Please let us know your results from the 3M & SKC passive badges and the active collection on the multi-absorbent tube so that we may compare them with ours. We apologize for the delay in getting these results to you. If you have any questions feel free to contact us. We hope that this has been of some help to you.

Sincerely yours,

Dr. H.D. Gesser and P. Mavroudis
University of Manitoba

FARR COMPANY
LABORATORY REPORT

DATE: September 4, 1996
LAB FILE: 1169-990
BY: O.M. delaCruz
LWR: 8759

TITLE: **Toluene Application on Two (2) Passive Samplers Being Developed by the University of Manitoba**

PURPOSE:

To apply toluene vapor on subject samplers for evaluation.

PROCEDURE:

The two (2) University of Manitoba passive samplers 35 mm square were installed in the 5" and 6-7/8" diameter test duct as shown on the attached diagram.

TEST RESULTS:

1. R & D test data:

   Airflow: 68.2 cfm (500 fpm for 5" dia. and 264 fpm for 6-7/8" dia.)
   Sample time: 192 minutes (3.2 hours)
   Toluene conc: 36* ppm (51.6 gms injected)

   * initially reported at 34 ppm based on standard conditions of 0°C and 760 mm Hg. The 36 ppm is based on 20°C and 760 mm Hg.

2. University of Manitoba data (also refer to the attached report):

   Toluene for 500 fpm sample: 0.020 g
   Toluene for 264 fpm sample: 0.021 g
   Average: 0.0205 g

   Calculated conc: 51.6g/192 mm x 68 ft^3/min x 0.0283 m^3/ft^3 = 0.14 g/m^3 = 0.14 mg/l

   Sampling rate: 20.5 mg/(3.2 h x 0.14 mg/l) = 46* l/h
   * compared to the 18 l/m static sampling rate.

DISCUSSION:

There will be more tests to be conducted with lower air flow and lower toluene concentration.

O.M. delaCruz

/lh

c: Dr. H. Gesser, University of Manitoba

L.F. 1169-990

GC TEST DUCT SET-UP