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Ultrasonic Extraction of Total Particulate Aromatic Hydrocarbons (TpAH) from Airborne Particles at Room Temperature

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KEY WORDS: Aromatic hydrocarbons, ultrasonic extraction, airborne particulates.

ABBREVIATIONS: 1. AH = Aromatic Hydrocarbon(s) 2. PAH = Polynuclear Aromatic Hydrocarbon(s) 3. pAH = Particulate Aromatic Hydrocarbon(s) 4. TpAH Total Particulate Aromatic Hydrocarbon(s)

A superior enrichment procedure for the extraction of TpAH from airborne particles collected on glass fiber filter paper is described.† The sample is suspended in a solvent and subjected to ultrasonic waves at room temperature with glass powder to adsorb polar coextractives. The TpAH in the filtered extracts are separated from other compounds by high speed liquid chromatography. Sensitivity is in the nanogram range, and the procedure is highly reproducible. Significantly larger amounts of TpAH are recovered than with Soxhlet extraction for 6 to 8 hours, and the percentage of pAH in the extracts is much higher. The entire procedure requires approximately 40 minutes, most of which is waiting time.

INTRODUCTION

Many of the aromatic hydrocarbons (AH) in airborne particulates are carcinogenic, therefore they are of interest to epidemiologists and to those people concerned with monitoring and attempting to control air pollution. Enrichment procedures for the measurement of TpAH in a particulate sample include sublimation, extraction by shaking with solvents at room temperature and Soxhlet extraction.

[†] Presented at the 165th National meeting of the American Chemical Society in Dallas, Texas, April 9, 1973.

Sublimation followed by fluorescence measurements is faster than Soxhlet extraction followed by column chromatography and UV spectral analysis, but in general the results are lower. Room temperature extraction by shaking requires much work, although reasonably good recovery of added PAH has been achieved with some solvents. The widely used Soxhlet extraction is time-consuming, decomposes some of the pollutants and lacks good precision. The benzene or cyclohexane extracts evaporated to dryness not only do not contain all the pAH, but have lost volatiles; they contain polymerized and/or otherwise modified material and many compounds that are not hydrocarbons. Too often the evaporated extracts will not redissolve in benzene. The non-pAH material interferes with the determination of TpAH, and with class and individual separation of pAH on high speed liquid chromatographic columns, so that the extracts are poorly suited for separations even on chromatographic systems which give separations of mixed AH standards.

Neither refluxing at 80°C nor shaking can be expected to extract all the organic compounds; this is especially true with respect to the AH in air particulate material because these procedures cannot break up the cohesive clumps or aggregates formed during long periods of sampling with glass fiber filters. An extraction procedure is needed which shreds the glass fiber filter on which the particles are imbedded; breaks up the particle aggregates; releases all compounds as fine particles or as solutes in solution, and selectively adsorbs most or all of the non-pAH on solid material, without heat.

Ultrasonic extraction of large amounts of dry atmospheric dust with various organic solvents was reported recently by Chatot et al.^{4,5} Their method involves evaporating to dryness followed by a time-consuming combination of separations by two-dimensional thin-layer chromatography, and chemical tests. It does not attack the problem of shredding glass fiber filter or the removal of polar coextractives in the sonifying vessel.

In the present procedure, ultrasonification of the air particles imbedded on glass fiber filter in a non-polar solvent in the presence of silica powder yields a colorless extract and has a potentiality for further development. Approximately half of the organic material in the extract is pAH, which is separated from the non-pAH compounds on a silica column by high speed liquid chromatography.

The method reported here measures TpAH (not benzo(a)pyrene). However, TpAH should be at least as realistic an indication of environmental cancer hazard as benzo(a)pyrene since, as already noted, several AH are carcinogenic and others are cocarcinogenic. The ultrasonic extracts have the added advantage of being suitable for the determination of aliphatic hydrocarbons⁶ and the analysis of AH by gas chromatography.

EXPERIMENTAL

Instrumentation

A Branson W 185 Sonifier Cell Disruptor† (20 KHz power ultrasonic generator with a 1.27 cm (½ inch) horn disruptor) and Sonabox from Heat Systems Ultrasonics Inc. were used for the sonification. Chromatographic separation of PAH was performed on a Nester-Faust Liquid Chromatograph No. 1200 with stainless steel column 2.6 × 300 mm, UV Detector with 254 nm filter, Hewlett Packard Strip Chart Recorder with Disc Integrator and a device for bypassing the column and pumping the sample directly through the detector (column bypass available from Perkin-Elmer Corporation). A Fisher filtrator and medium sintered glass filter were used for filtering extracts. A Lab-Size Spinning Band Still from B/R Glass, Inc., Stanton, Delaware was used to distill solvents. The glass powder was sieved in a U.S. Standard Sieve Series No. 120, with 125 micron openings, from the Fisher Scientific Company.

Reagents

Cyclohexane (certified A.C.S. spectranalyzed) obtained from the Fisher Scientific Company, was redistilled from glass at least once. Other solvents were A.C.S. reagent grade. Polynuclear aromatic hydrocarbons were from various commercial sources and were not repurified. Silica I was glass powder, "5 micron" non-wettable, obtained from Heat Systems Ultrasonics, Inc. Silica II was Code 7930 200/325 mesh, fragmented, obtained from Corning Biological Products Group. Column packing was Corasil II, obtained from Waters Associates, Inc.

ANALYTICAL PROCEDURES

Extraction

Support the 1.27 cm ($\frac{1}{2}$ inch) horn of the sonifier disruptor in a sonabox to reduce noise. The sonifying vessel is a beaker 3.8 cm I.D.×10 cm tall. Keep the end of the horn about 0.6 cm above the bottom of the beaker to insure adequate "stirring" of the mixture and equal exposure to areas of intense cavitation. Take a glass fiber filter sample approximately 16 square cm in size, and cut into roughly 1 cm squares to facilitate shredding. Surround the sonifying vessel with an icewater bath up to the level of the solvent mixture.

[†] Mention of a specific product or company name does not constitute endorsement by the Environmental Protection Agency.

Place 60 ml cyclohexane, 5 ml Silica I glass powder and the sample in the sonifying vessel. Sonify for 8 minutes at 70 watts. Decant the supernatant into the sintered glass filter supported on a Fisher filtrator. Add cyclohexane to the sonifying vessel to the level of the original mixture (usually about 50 ml). Sonify for an additional 4 minutes. Filter, combining with the first fraction. Rinse with 50 ml cyclohexane. Collect the filtrates and rinsings in an Erlenmeyer flask and evaporate the solvent to a volume of about 5 ml. Transfer quantitatively to a 10 ml volumetric flask and make to the mark. For a blank use 16 square cm of fiber glass filter. Samples are reasonably stable for a few days at room temperature in the dark.

Chromatographic separation

A schematic of the chromatographic system is shown in Figure 1. The stain-

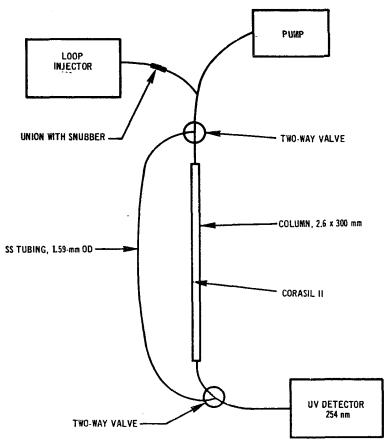


FIGURE 1 Schematic of chromatographic system.

less steel column is 2.6×300 mm; the packing is Corasil II; the eluent is cyclohexane. Two two-way valves are installed in the chromatographic line, one before the column, the other after it. They are connected with stainless steel tubing. This enables the sample to be pumped through either the column or the tubing (column bypass) into the UV detector. A Swagelok union with snubber may be placed in the tubing after the loop injector to prevent extraneous particles from clogging the line.

The column bypass is used in two ways: (1) To determine the percent of the PAH standards which elutes through the column. A satisfactory column elutes close to 100% of the PAH with the solvent front and retains polar compounds. (2) To determine the percent of the organic material in the extracts which elutes through the column.

Inject the sample through a loop injector with capacities varying from 0.2 to 2 ml. A flow rate of 1.6 ml/minute gives a pressure drop of less than 200 PSI. Measure the peak area with a disk integrator, driven by a 0 to 10 mv servo strip chart recorder with a 0.5 cm/minute chart speed. The pAH elute in 3 to 4 minutes and are measured at 254 nm. Most polar compounds are retained on the column. Samples may be chromatographed every 5 to 10 minutes.

The relationship of sample concentration and peak area is linear for both benzo(a)pyrene and other AH tested, and for dilutions of sample extracts over a satisfactory analytical range which varies with the sensitivity of the individual detector.

Calculations

The peak area of the TpAH in a cubic meter of air is given by the equation

$$PA/m^3 = \frac{PA \times A \times B}{V \times a \times b}$$

Where PA is the corrected peak area, V is the volume of air sampled in m^3 , A is the area of the glass fiber filter in cm^2 , B is the volume of extract in ml, a is the area of glass fiber filter sample in cm^2 , and b is the volume of extract injected in ml.

Note that there are only three variables in the equation, the volume of air sampled, the volume of extract injected and the peak area of the sample analyzed. The fiber glass filters do not need to be conditioned and weighed, nor the weight of dry organics determined.

RESULTS AND DISCUSSION

The capability of the chromatographic system to elute AH and retain

non-AH was investigated by screening a number of compounds which might be found in air particles. The percent of the compounds which eluted through the column was calculated from the peak areas through the column and the column-bypass. Typical AH chromatograms from column and tubing are shown in Figure 2. Recovery of benzo(ghi)perylene was 99%. The unit of measurement is peak area per microgram of sample (PA/ug).

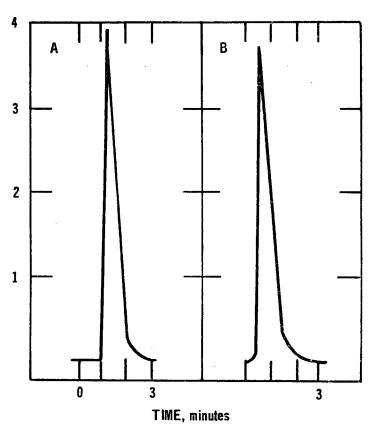


FIGURE 2 Chromatograms of benzo(ghi)perylene, $0.629 \mu g$ in cyclohexane, through the column (A) and through the column bypass (B). Stationary phase, Corasil II; eluent, cyclohexane; flow rate, 1.6 ml/minute. Peak area on column, 1165; on column bypass, 1180; recovery on column, 99%; peak area/ μg , 1850.

AH elute through the column with retention times of approximately 1 to 2 minutes. Elution percentages range from 91 to 105, as shown in Table I Alkyl benzenes, which may be present in vehicle exhaust air particulate material, elute but have very low PA/ug values. Anthracene and phenanthrene

have very high PA/ug values, but are not usually present on glass fiber filters because of their volatility. Fluorene and its derivatives (except 11 H-benzo(b)-

TABLE I Elution of PAH^a

Compound	% Eluted through column	PA/ug×10 ⁻³
Mono-,dicyclics		
Benzene	9 9	0.4
N-Hexylbenzene	100	0.5
N-Heptylbenzene	100	0.7
Naphthalene	101	0.7
Azulene	93	3.0
Tricyclics		
Anthracene	100	36.0
9-Methylanthracene	99	15.0
Xanthene	102	1.3
Phenoxathiin	92	0.2
Phenanthrene	100	10.0
Tetracyclics		
Naphthacene	95	4.7
Chrysene	105	4.5
Pyrene	96	3.6
4-Methylpyrene	100	1.7
1,3-Dimethylpyrene	96	0.9
Triphenylene	100	9.0
Benz(a)anthracene	96	4.3
7,12-Dimethylbenz(a)anthracene	102	3.3
Pentacyclics		
Dibenz(a,h)anthracene	96	0.6
Benzo(a)pyrene	100	5.3
Benzo(e)pyrene	92	2.2
Picene	99	5.0
Perylene	96	5.8
Hexacycclics		
Benzo(ghi)perylene	99	1.8
Anthanthrene	93	2.6
Dibenzo(fg, op)naphthacene	93	0.6
Coronene	91	0.5
Dibenzo(g,p)chrysene	96	1.0
Naphtho(2,1,8-qra)naphthaceneb	100	0.7

Retention time is approximately 2 minutes.

fluorene) and hetero tricyclics with oxygen or sulfur or both in the ring have low PA/ug values as shown in Table II. Polychloro derivatives have retention

b Or naphtho(2,3-a)pyrene.

times of 2 minutes or less, elution percentages from 85 to 104 but quite low PA/ug levels, listed in Table III. However, they would probably be present in the atmosphere in the vapor phase. Hydroxyl and amino groups increase retention time or even cause complete retention. Nitro, ethyl, methyl, dimethyl and chloro substitution tend to lower PA/ug levels. Hexacyclics have a lower average level than tetra- and pentacyclics.

Oxygenated compounds, some phenols, and aza and iminoheterocyclics (except for some members of the indole series) are retained. These include the following: benzoquinone, fluorenone, anthrone, acenaphthenone, 9-

TABLE II
Elution of fluorene, analogues and derivatives

Compound	t min. R	% Eluted through column	PA/ug×10 ⁻³
Fluorene	2.0	100	2.9
Dibenzothiophene	2.0	96	1.8
Dibenzofuran	2.0	98	0.3
Fluoranthene	2.0	95	2.5
Benzo(k)fluoranthene	1.8	97	1.4
Benzo(b)fluoranthene	1.0	99	1.6
2-Ethylfluorene	1.0	95	1.6
11 H-Benzo(b)fluorene	1.0	110	5.6
2-Nitrofluorene	4.8	104	0.2
2,5-Dinitrofluorene	7.0	71	0.3
9-Fluorenol	8.5	14	0.2
3,6-Dinitrodibenzoselenophene	18.2	38	0.2
3-Aminofluorene	18.2	68	0.4
4-Fluorenecarboxylic acid		Retained on column	
2-Hydroxyfluorene	Retained of column		
2-Nitro-7-hydroxyfluorene	Retained on column		
Fluorenone	•	Retained on column	

xanthenone, anthraquinone, perinaphthenone, benzanthrone, 2-hydroxy-carbazole, benzo(c)cinnoline, 5H-dibenzo(a,d)cyclohepten-5-one, p-phenylphenol, o-ethylphenol, anthanthrone, indeno(1,2,3-ij)isoquinoline, benzo (1mn)phenanthridine, acridine, benzo(f)quinoline and quinoline. Indole and carbazoles elute with retention times of 5.3 to 18.3 minutes, in percentages varying from 55 to 92 and somewhat low PA/ug levels, as indicated in Table IV. Alkyl substitution on the ring nitrogen permits elution since the new molecule cannot form a hydrogen bond. Hydroxyl and amino groups cause retention on the column. Elution of the aldehydes examined was delayed. Compounds eluting after five minutes, of course, do not interfere

TABLE III

Elution of polychloro derivatives of di- and tricyclic hydrocarbons^a

Compound	% Eluted through column	PA/ug×10 ⁻³
1,1-Dichloro-2,2-bis(p-chlorophenyl)ethane		
(p,p'DDD)	94	0.02
1,1-Dichloro-2,2-bis-(p-chlorophenyl)ethylene (DDE)	97	0.50
1,1,1-Trichloro-2,2-bis(p-chlorophenyl)ethane		
(p,p'DDT)	85	0.02
Aroclor 1260 (chlorinated biphenyls, 60 % chlorine)	100	0.13
Aroclor 5432 (chlorinated triphenyls, 32% chlorine)	104	0.61
Halowax 1099 (mixture of tri- and tetrachloro		
naphthalenes, 52% chlorine)	101	0.25
1,2,3,4,5,6,7,8-Octachloronaphthalene	97	0.64
2,3,4,5,6,2',3',4',5',6'-Decachlorobiphenyl	95	0.19
1,2,3,4,5,6,7,8-Octachlorodibenzofuran	93	0.33
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin	98	0.85
Tetradecachloro-p-terphenyl	95	0.22

[•] Retention times from 1 to 2 minutes.

TABLE IV

Elution of some indoles, carbazoles and aromatic aldehydes

Compound	t_R min.	% Eluted through column	PA/ug×10 ⁻³	
Indole	5.3	82	1.1	
Carbazole	11.8	67	0.7	
4 H-Benzo(def)carbazole	8.0	98	2.0	
11 H-Benzo(a)carbazole	14.5	55	3.0	
7 H-Dibenzo(c,g)carbazole	18.0	92	2.1	
N-Phenylcarbazole	2.3	74	1.8	
N-Ethylcarbazole	2.5	98	0.5	
5-Methyl-5,10-dihydroindeno				
(1,2-b)indole	2.8	103	1.9	
2,3-Dimethylindole	5.3	90	5.5	
2-Methylcarbazole	6.8	100	0.8	
2-Hydroxycarbazole	Retained on column			
N-Ethyl-3-aminocarbazole	Retained on column			
Benzaldehyde	12.8	56	1.1	
2-Naphthaldehyde	8.2	78	0.3	

with the measurement of AH. In the air samples studied, no elution of material after five minutes was observed, when cyclohexane was used for the extraction.

On the basis of these findings, since Corasil II is an adsorbent of high activity and the eluent is non-polar cyclohexane, it is assumed that organic compounds in the air particulate ultrasonic extracts which elute with the solvent front are, for the most part, hydrocarbons or their nonpolar derivatives. It is possible the results may be weighted in favor of tetra- and pentacyclic hydrocarbons, since their PA/ug levels are higher in general than those of other compounds tested. Aliphatics do not interfere, since they do not absorb in the ultraviolet.

The wide variation between the PA/ug levels of the AH raises the question of the value of absorbance readings at 254 nm or at any other single wavelength. The spectra of AH show much fine structure, usually with several peaks of varying intensity. Maximum intensity peaks of different AH differ widely in wavelength. Perhaps the most meaningful measurement of a mixture of AH such as in these extracts would be the total absorbance between 400 and 230 nm obtained by integrating a spectrogram of this area. Such a measurement would greatly increase the sensitivity of the method. Multiwave detectors with scanning spectro-photometers are now available which could be used for such an analysis.

Choice of solvent and use of glass powder

Non-wettable glass powder is included in the sonification mixture to adsorb non-AH as well as to promote cavitational intensity for shredding the glass fiber filter and finely subdividing the black air particulate aggregates. Also the powder becomes enmeshed with the glass fiber shreds and prevents them from stopping up the sintered glass filter.

When a non-polar solvent such as cyclohexane is used, the AH and some of the less polar organic compounds go into solution to produce a clear, colorless extract. The carbon and the remainder of the organic material adhere to the surface of the powder and/or the glass fibers. When increasingly polar solvents such as acetone are used, increased amounts of the more polar organic compounds are not adsorbed and remain in solution. The carbon is suspended and does not settle out, although it can be filtered off. The filtrate is pale brown. Sonification with methanol also puts the carbon in suspension. Much of the remaining organic material remains in solution and a darker brown filtrate is obtained. Thus, there is the possibility of extracting many of the organic compounds from air particles and separating them into groups on the basis of their desorption from silica powder in solvents of varying polarity. Since it is desirable to have as pure a pAH extract as possible, the solvents of choice are non-polar, such as cyclohexane or hexane. Methylene

chloride and mixtures of methylene chloride and cyclohexane are unsuitable as they form large and unreproducible blanks.

The adsorption of organic compounds on the surface of the silica powder in the sonifying vessel depends also on the volume and condition of the powder. Silica I particles average 25 microns in diameter and are spherical. When amounts of up to 10 ml of these particles were used, extracts were obtained in which 50% or more of the extracted material was AH; with 15 ml the figure was 65%; with 20 ml it was 78%. It was not possible to use more than 20 ml of Silica I, because the ultrasonic waves were unable to "stir" the mixture and shred the glass fiber filter. Silica II particles are somewhat larger in diameter and are fragmented. Five ml of Silica II gave extracts containing close to 100% AH, but only 70% of the AH were recovered. This product is being studied. Maximization of such parameters as volume and condition of the powder, standing time after homogenization and filtering techniques to insure adsorption of all organic compounds except AH should make possible the production of pure AH extracts in the sonification vessel. Also it is very likely to shorten the sonification time, perhaps to as little as 5 minutes.

Other ultrasonic-adsorbent-solvent systems might be applicable to the extraction of insecticides and other organic pollutants in soil, and plant and animal tissues, with the removal of unwanted coextractives. The glass powder is relatively expensive, but can be reclaimed by sieving to remove the glass fiber shreds, washing with distilled water, and firing at 500°C to remove organic material and carbon.

Parameters

The temperature in the sonification vessel must remain constant. Without a cooling bath, the temperature rises to the boiling point of whatever solvent is being used. As the temperature rises, the wattage decreases and the recovery of AH becomes unreproducible. The icewater bath maintains the temperature near 25°C. Maximum recovery of AH was obtained with 70 watts, 60 ml solvent and 12 minutes sonifying time.

Recovery of added PAH

PAH standards were added to glass fiber filter blanks and sonified. These recovery standards were compared with readings of the standard solutions. The results are summarized in Table V. Ninety-five per cent of added anthracene was recovered; 97.5% of added phenanthrene and 98.2% of added benzo(a)pyrene were recovered. This indicates the processes of sonification, filtration and evaporation do not cause appreciable loss of PAH. Three

hundred and fifty-five nanograms of benzo(a)pyrene gave a peak area of 1880. Since the peaks are reproducible within one or two per cent, the detection limit is obviously on the order of three or four nanograms, or less. Corresponding values for anthracene and phenanthrene would be roughly one or two nanograms. However, no reproducibility tests were made at this level.

TABLE V
Recovery of added PAH

Compound		Peak area		
	Sample, ug	Sonified Filter + Std.	Standard solution	% Recovery
Anthracene	0.035	1005	1055	95.0
Phenanthrene	0.147	1155	1185	97.5
Benzo(a)pyrene	0.355	1846	1880	98.2

TABLE VI

Comparison of ultrasonic and soxhlet extractions

PA/ug 0.449	% Eluted
	28
0.500	
0.509	
0.500	
0.545	31
· 	
	
0.509	30
±26.1%	

a Refers to % of TpAH in the extracted material.

Comparison of ultrasonic and soxhlet extraction

Composited homogeneous glass fiber filter samples on which air particulates had been collected were analyzed using the ultrasonic and Soxhlet extraction procedures. The chromatograms of Sample I in Table VI made

with the column and with the column bypass are compared in Figure 3. The peaks are virtually identical, except that the ultrasonic-obtained peaks are larger than those obtained by Soxhlet. The results are tabulated in Table VI.

The average PA/ug for the ultrasonic extracts was 0.569, with a relative standard deviation of $\pm 1.33\%$. Corresponding figures for the Soxhlet extracts were 0.509 and $\pm 26.1\%$. The average AH per cent of the organic material in the ultrasonic extracts was 49; in the Soxhlets it was 30. The ratio of ultrasonic to Soxhlet recovery was 1.14. Thus, ultrasonic extraction

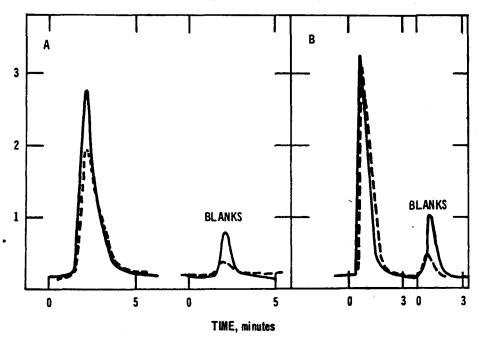


FIGURE 3 Chromatograms of ultrasonic and Soxhlet extracts of composited sample No. 1, Table VI. and blanks, through the column (A) and through the column bypass (B). Stationary phase, Corasil II; eluent, cyclohexane; flow rate, 1.6 ml/minute. Solid lines are ultrasonic extracts; broken lines are Soxhlet extracts. Extracts were diluted ×3.3 for column bypass.

recovers larger amounts of AH, with lower percentages of non-AH, and a much lower relative standard deviation than does Soxhlet extraction.

The enormous difference between the PA/ug values of the AH standards and the homogeneous composited samples used for comparative experimental work is due to the fact that the weight of these samples, which was carefully adjusted to 100 mg, necessarily includes the weight of the fiber glass filter. For example, 2222 ug of the composite Sample No. 1, Table VI, was injected

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TABLE VII Analysis of particulate samples

Description	Corrected peak area	M ³ air sampled	PA/m³ª air
Urban I	1200	1500	1120
Urban II	620	1500	580
Urban III	545	1500	509
Mt. Storm	0	1673	0

a See Calculations.

onto the column to give a peak area of 1278, or a PA/ug of 0.575; 0.355 ug of benzo(a)pyrene gave a peak area of 1880, or a PA/ug of 5300. However, routine air particulate samples are measured by filter area and air volume rather than by weight, and are reported in terms of PA/m³ air.

Analysis of air particulate samples

Four Hi-Vol air particulate samples were analyzed. See Table VII for the results. The samples ranged from very polluted Urban I to Mt. Storm with slight accumulation of air particles on the filter. The peak area/m³ is calculated from the peak area measured by the disk integrator and the total volume of air sampled. The PA/m³ values range from 1120 for Urban I, apparently the most polluted of the samples, to 0 for Mt. Storm. It is beyond the scope of this work to relate these figures for TpAH to carcinogenicity, but they obviously do provide an index of AH pollution. For example, they could be used to compare pollution levels at different sites, and at different times at the same site.

This method is applicable in its present form to routine determinations of total AH in air particules collected on glass fiber filters. Injection by loop injector is more reproducible and more easily handled than on-column injection with a microsyringe. It enables the use of larger samples, so the extracts need not be evaporated to an extremely small volume. It is fast enough to allow the performance of an economically feasible number of determinations per day. Since the filters need not be weighed nor dry organics obtained, a substantial amount of work and calculations are eliminated.

Essentially this ultrasonic extraction procedure represents a superior method for the cleanup of organic extracts of airborne particles prior to analysis for benzo(a)pyrene or AH. In contrast to the Soxhlet extraction procedure gravimetry the ultrasonic method is faster, much more reliable, extracts more AH and gives much cleaner samples for analysis.

Acknowledgements

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