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Sampling of Airborne Pesticides Using Chromosorb 102

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Pesticides are of great concern when used in the workplace or in the home because of possible toxic, chronic or irritating effects, and in some cases, their ability to exist for long periods of time. Because of the complexity in the types of pesticides used, their different physical states and difficulty in collection and analysis, monitoring personnel are usually reluctant to deal with them. In earlier works, collection efficiencies for select pesticides were evaluated on home-made Chromosorb 102 sampling tubes (Thomas 1974, Jackson 1977, Jackson 1978, Thomas 1980, Yeboah 1984). In this study, commercially available Chromosorb 102 sorbent air sampling tubes were used to evaluate the commonly used pesticides: sevin, DDVP, pyrethrum, aldrin, toxaphene and pentachlorophenol. Our work showed that these pesticides were efficiently collected by these new commercial tubes, analytical recovery was acceptable and that these readily available tubes could be easily used by sampling personnel in the workplace or homes to evaluate possible hazardous situations.

MATERIALS AND METHODS

Chromosorb 102 sorbent air sample tubes (also called XAD-2) are now commercially available (SKC or Supelco). They are similar in design to the conventional charcoal sampling tubes. They are designed into two portions, 66/33 milligrams (mg) and separated by either glass wool or a polyurethane plug. Each section may be analyzed separately if breakthrough is a concern. These tubes show acceptable gas chromatographic backgrounds.

To simplify this study, the pesticides were deposited as a solvent concentration on the front portion of the commercial SKC sampling tube (Figure 1). This was done for several reasons. First, by depositing all the pesticides at once, this would favor breakthrough if this was a problem. Secondly, using this technique would keep us from having to modify the commercial tubes to fit conventional pesticide generators.

After depositing the pesticides, known volumes of air were drawn through the tubes to approximate 0.5X, 1.0X and 2.0X workplace threshold limit values of the pesticides studied. This flowrate, drawn through the tubes, varied between 0.6 to 1.0 liters per minute

(Lpm). As the tubes were hooked to the vacuum, they were gently tapped for a few seconds to cause the collecting spheres to compact. This was necessary to avoid potential channeling problems.

After the desired sampling volume had been obtained, the tubes were etched with a file and broken so the contents became easily accessible. The glass wool or polyurethane plug was removed, dropped into a flask and the contents of the tube tapped so that it fell into the flask also. The sampling tube was also quantitatively rinsed with solvent (pesticide-grade hexane). Approximately 25-50 milliliters (mL) of solvent was added to the flask and then the flask placed into an ultrasonic cleaner containing about one inch of water. A lead donut was used to keep the flask from falling over. The ultrasonic unit (480 watts power) was turned on for about 30 minutes. This action assisted in quantitative removal of pesticide from the collector. The solvent was filtered through a glass fiber filter paper containing a one inch layer of anhydrous sodium sulfate. The filter solvent was then flash evaporated down to approximately 0.5 mL. The contents were quantitatively transferred to a five mL volumetric flask and brought up to the five mL mark with solvent.

Toxaphene, aldrin and pyrethrum were analyzed on a Shimatzu Model GC-6AM gas chromatograph equipped with 5 ft x 2 mm glass column of 6% OV-210 + 4% SE-30 on 80/100 Gas Chrom Q. It was equipped with an electron capture detector (^{63}Ni), column temperature 200°C, injector and detector at 250°C.

DDVP was analyzed on a Hewlett-Packard Model 5880 GC equipped with a 5 ft x 2 mm glass column of 20% SP2100 + 0.1% Carbowax 20M on 100/200 Supelocoat. It was equipped with an electron capture detector. Column temperature was 200°C, injector was 210°C and detector temperature was 300°C.

Pentachlorophenol and carbaryl were analyzed on a Waters HPLC equipped with a Waters Radial Pak B LC cartridge. A mobile phase of 90% methanol-10% water was used at 1.0 mL/min. The UV detector used was the 254 millimeter (mm) filter.

RESULTS AND DISCUSSION

For the pesticides studied, recovery and collection efficiency with these commercial sampling tubes was well within quantitative requirements (Tables 1 to 6). In all cases, none of the pesticides penetrated into the backup sections of the tubes. The variation in collection efficiency and recovery was due to analytical errors or nondesorption from the collector. To reach recommended workplace ACGIH TLV values, large amounts of pesticides had to be deposited onto the collector. This made very large dilutions of the extracts necessary in order to be within the linearity ranges of the analytical instruments. Although the analyst took great care in the necessary steps, recovery variations may be due to these problems of large dilutions.

Several advantages can be seen in the use of these sampling tubes: First, like charcoal tubes, they can easily be attached near workers breathing zones to check for pesticide inhalation, or set up in a stationary position. The sampling tubes are easy to prepare for analysis leading to quick turnaround times. Various solvents can be used for sample workup, depending on the analyst's needs. Methylene chloride, acetone, isooctane and ethanol have all been successfully used in addition to hexane. Extracts of the sample tubes show low background levels and do not cause problems in chromatographic analysis. Samples can be analyzed by liquid chromatography in addition to gas chromatography. For compounds like pentachlorophenol and carbaryl, which are gas chromatographed with difficulty, liquid chromatography works nicely.

Table 1. Toxaphene Collection Efficiency and Recovery

Sample	Amount (μg) Deposited	Recovery (μg) Front	Back	% Recovery	Air Volume (Liters)	mg/m³ [b]
#1	2.34	2.35	N.D.[a]	100.4	10.0	0.234
#2	2.34	2.38	N.D.	101.5	10.0	0.234
#3	4.63	4.63	N.D.	100.0	10.0	0.463
#4	4.63	3.62	N.D.	78.3	10.0	0.463
#5	9.26	10.58	N.D.	114.4	10.0	0.926
#6	9.26	10.44	N.D.	112.9	10.0	0.926
			AVG:	101.2		

[a] Nondetected (N.D.) equals less than 0.010 μg per section
[b] TLV = 0.50 mg/m³ Skin, 1.0 mg/m³ Short Term Exposure Limit (STEL)

Table 2. DDVP Collection Efficiency and Recovery

Sample	Amount (μg) Deposited	Recovery (μg) Front	Back	% Recovery	Air Volume (Liters)	mg/m³ [b]
#1	54.5	48.5	N.D.[a]	88.8	120	0.454
#2	53.8	47.5	N.D.	88.4	90	0.598
#3	54.0	49.5	N.D.	91.8	62	0.871
#4	54.0	49.8	N.D.	92.3	56	0.964
#5	54.0	48.5	N.D.	89.8	35	1.543
#6	54.2	53.5	N.D.	98.8	41	1.322
			AVG:	91.7		

[a] N.D. equals less than 0.05 μg per section
[b] TLV = 1.0 mg/m³ Skin, 3.0 mg/m³ STEL

Table 3. Aldrin Collection Efficiency and Recovery

Sample	Amount (μg) Deposited	Recovery (μg)		% Recovery	Air Volume (Liters)	mg/m^3[b]
		Front	Back			
#1	1.25	1.25	N.D.[a]	100	9.90	0.126
#2	1.25	1.45	N.D.	116	9.90	0.126
#3	2.50	2.05	N.D.	82	9.90	0.252
#4	2.50	2.40	N.D.	96	9.90	0.252
#5	5.00	5.60	N.D.	112	9.90	0.505
#6	5.00	5.70	N.D.	114	9.90	0.505
			AVG:	103.3		

[a] N.D. equals less than 0.010 μg per section

[b] TLV = 0.25 mg/m^3 Skin, 0.75 mg/m^3 STEL

Table 4. Sevin Collection Efficiency and Recovery

Sample	Amount (μg) Deposited	Recovery (μg)		% Recovery	Air Volume (Liters)	mg/m^3[b]
		Front	Back			
#1	10.56	10.04	N.D.[a]	95.1	10.66	0.991
#2	10.67	11.2	N.D.	104.2	15.48	0.698
#3	20.57	23.33	N.D.	113.8	11.99	1.72
#4	21.45	23.55	N.D.	109.8	9.99	2.15
#5	21.56	24.17	N.D.	111.9	4.66	4.63
#6	21.56	24.17	N.D.	111.9	5.33	4.04
			AVG:	107.8		

[a] N.D. equals less than 0.050 μg per section

[b] TLV = 5 mg/m^3 Skin, 15 mg/m^3 STEL

Table 5. Pyrethrum Collection Efficiency and Recovery

Sample	Amount (μg) Deposited	Recovery (μg)		% Recovery	Air Volume (Liters)	mg/m^3[b]
		Front	Back			
#1	13.99	9.95	N.D.[a]	71	10.35	1.351
#2	13.80	11.91	N.D.	86	10.67	1.293
#3	28.84	29.68	N.D.	103	10.35	2.79
#4	29.04	35.00	N.D.	120	15.8	1.84
#5	28.94	29.68	N.D.	91	5.3	5.46
#6	28.75	22.73	N.D.	79	5.6	5.13
			AVG:	92		

[a] N.D. equals less than 0.050 μg per section

[b] TLV = 5 mg/m^3 Skin, 10 mg/m^3 STEL

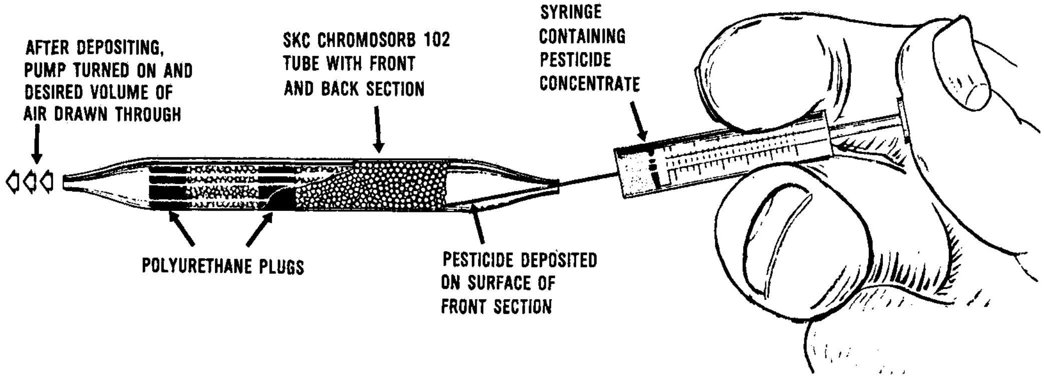

Figure 1. Technique used to evaluate collection efficiency of SKC Chromosorb 102 Tube.

Table 6. Pentachlorophenol Collection Efficiency and Recovery

Sample	Amount (µg) Deposited	Recovery (µg) Front	Recovery (µg) Back	% Recovery	Air Volume (Liters)	mg/m^3[b]
#1	22.8	19.0	N.D.[a]	83.3	68.2	0.334
#2	23.0	23.0	N.D.	100.0	68.2	0.337
#3	23.2	19.0	N.D.	81.9	33.0	0.703
#4	23.2	21.0	N.D.	90.5	34.5	0.672
#5	23.2	21.2	N.D.	91.4	22.8	1.018
#6	23.2	19.2	N.D.	82.8	22.8	1.018
			AVG:	88.3		

[a] N.D. equals less than 0.1 µg per section
[b] TLV = 0.5 mg/m^3 Skin, 1.5 mg/m^3 STEL

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