Bull. Environm. Contam. Toxicol. 20,445-446 (1978)

Charcoal Column Separation of Chlordane and Toxaphene

J. C. Underwood
Total Diet Laboratory, Kansas City District Office, Food & Drug Administration, Kansas City, Mo. 64106

The separation and determination of chlordane and toxaphene has been a difficult task for the pesticide residue chemist. The following method allows the separation and determination of chlordane and toxaphene. The extracted residues must have had prior florisil cleanup (HORWITZ 1975) and be in petroleum ether or hexane.

MATERIALS AND METHODS

Charcoal Preparation: Charcoal is boiled for 10 min with acetone on a steam bath, cooled and filtered by suction. The procedure is repeated. The filter cake in the funnel is washed with cold acetone. The charcoal is air dried and stored at 135°C for 12 h before using. (BERG et al. 1972).

Apparatus:

1. Chromaflex column (10 mm ID) size 213 #K420550 Kontes ⅋ 24/40 inner joint. This column is equipped to handle a 500 mL K-D Concentrator, which acts as a reservoir for the eluting solvent.
2. 500-mL Kuderna Danish (K-D) concentrator Kontes ⅋ 24/40 inner joint and a ⅋ 19/22 outer joint.
3. Snyder Condenser-Kontes ⅋ 24/40 joint.
4. 500-mL K-D Concentrator ⅋ 24/40 inner joint and a ⅋ 24/25 outer joint.
5. 5 mL tip Kontes ⅋ 19/22 inner joint.
6. Tracor 222 gas chromatograph or equivalent with an electron capture detector.

Procedure: Prepare a chromaflex column by placing ca 25 mm of Ottawa sand in the column. Remove the charcoal from the oven, weigh out 1.0 g and transfer it to the column. Pack the column by tapping. Place ca 5 mm of sodium sulfate on top of the charcoal, let cool for 20 min and rinse with 25 mL hexane. Place a 500 mL K-D under the column to receive the eluate. Transfer the petroleum ether or hexane (maximum 5 mL) containing the chlordane-toxaphene to be separated to the column and allow to enter the column.

0007-4861/78/0020-0445 $01.00

Rinse the container with two 5 mL portions of hexane, letting each rinse enter the column before adding next rinse. Finally elute fraction #1 with 25 mL 6% acetone/hexane v/v. Remove the first K-D and replace with a second K-D similarly equipped and elute fraction #2 with 300 mL benzene. When fraction #2 is eluted, evaporate both fractions to appropriate volume for injection on GLC. Fraction #1 contains chlordane and fraction #2 contains toxaphene.

A standard mixture of chlordane and toxaphene was prepared containing 10 ug/mL chlordane and 50 ug/mL toxaphene. A one mL aliquot of the standard mixture was placed on each of two charcoal columns and separated using the above procedure.

TABLE 1

Recovery Data on Chlordane and Toxaphene Separated on Charcoal.

	Pesticide	Recovered	Added	% Recovery
Deter. 1	Chlordane	8.25 ug	10.0 ug	82.5
	Toxaphene	42.4 ug	50.0 ug	84.8
Deter. 2	Chlordane	7.80 ug	10.0 ug	78.0
	Toxaphene	38.9 ug	50.0 ug	77.8

RESULTS AND DISCUSSION

The solvent containing the chlordane and toxaphene to be separated must be free of product extract which could cause overloading of the micro-charcoal column and a change in the elution pattern. A trace of toxaphene will be in fraction #1 and a trace of chlordane will be in fraction #2, but the separation is suitable for quantitative calculation. This method has been used successfully on crops, fish, meats, oils and fats.

ACKNOWLEDGEMENTS

The author thanks chemists W. F. McCullough, K. R. Griffitt, and C. Meloan for their technical assistance.

REFERENCES

BERG, O. W., DIOSADY, P. L, and REES, G.A.V.: Bull. Environ. Contam. Toxicol. 7, 338 (1972)

HORWITZ, W. (Ed.) Offic. Methods of Analysis, J. Assoc. Offic. Anal. Chem., Wash. D.C., 12 (1975), 29.015