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Estimation of Fluoroalkane Propellants

During 1971 four fatal cases occurring in Cook County, Ill., were determined to have been due to the inhalation of trichlorofluoromethane and dichlorofluoromethane, which are common propellants used in various aerosol dispensers.

Early studies have generally indicated that these compounds have a rather low degree of toxicity in humans [*1*]. Probably for this reason few methods have been published for determination of fluoroalkanes in postmortem human tissues.

This report describes a simple and reliable method for the identification and estimation of trichloromonofluoromethane and dichlorodifluoromethane using a gas chromatographic procedure.

Experimental Reagents

Benzene (Specto grade)
Trichloromonofluoromethane (Freon 11)[3]
Dichlorodifluoromethane (Freon 12)[3]
Column Packing—Chromosorb 102 60/80 mesh[4]

Apparatus

A Barber-Coleman Gas-Liquid Chromatograph (GLC) Series 5000, equipped with an Electron Capture (Tritium Cell) and flame ionization detector, was used. The column was 6 ft in height and packed with a 60/80 mesh Chromasorb 102. The temperature of the detector was 210°C, the injection temperature was 240°C, and the column temperature was 180°C. Nitrogen was used as a carrier gas with a flow rate of 45 ml/min with an inlet pressure of 40 lb.

Collection of Postmortem Tissues

Blood should be collected prior to proceeding with autopsy to minimize evaporation of the gases. A known amount of blood is put into a calibrated conical centrifuge tube with a screw cap fitted with Teflon disk[5] containing a known amount of benzene, and immediately frozen. Lung, liver, brain, and any other tissue are put into a preweighted

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[3] Standards were obtained from E. I. du Pont de Nemours and Co.

[4] Obtained from Johns-Manville Co.

[5] Obtained from Kontes Glass Co.

conical centrifuge tube containing a known amount of benzene (1–3 ml) and are immediately frozen and retained in that state until analyzed. The net weight of the tissue can then be determined (usually about 1–3 grams). To assure minimum loss of gases the samples should be collected during the autopsy and immediately secured as stated above.

Procedure

The samples are defrosted at room temperature. Vigorous shaking of the tube with its contents for about 5 min will adequately extract the fluoroalkane into the benzene. The mixture will quickly separate into two layers. Aliquots of the benzene layer can then be injected into the gas chromatograph for analysis.

Standard Curve

About 50 ml of benzene are introduced into a 100-ml volumetric flask and weighed. Trichlorofluoromethane from the pressure can is liquefied in a test tube, while it cools at 15°C. Dichlorodifluoromethane is liquefied at −40°C using acetone and dry ice, while at the same time cooling a micropipet. The fluoroalkanes are introduced into the 100-ml volumetric flask containing the benzene. The amount of fluoroalkane dissolved in the benzene is then determined by weighing. More benzene is added to bring the volume to 100 ml. Thus the weight of fluoroalkane in 100 ml of benzene is known. For the determination of the standard curves properly diluted aliquots of this stock solution can then be used. For the standard curves peak heights are used because of the sharpness of the peaks [*2*] (Figs. 1 and 2). As shown in Fig. 3 trichlorofluoromethane is more sensitive than dichlorofluoromethane [*3*].

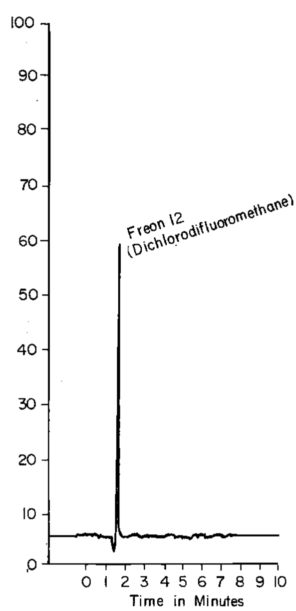

FIG. 1—*Gas liquid chromatography of dichlorodifluoromethane (Freon 12).*

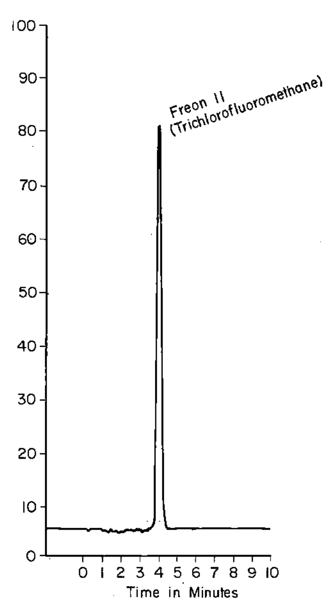

FIG. 2—*Gas liquid chromatography of trichlorofluoromethane (Freon 11).*

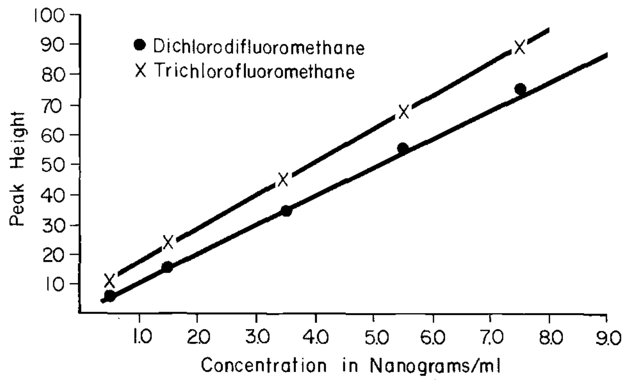

FIG. 3—*Standard curve—plot of peak height versus concentration.*

Discussion

This method for the identification and estimation of fluoroalkane propellants is sufficiently quantitive for use in the forensic laboratory. It has proved to be reliable for the analysis of blood, liver, and lung tissue.

In the Cook County cases lung tissue was found to contain a higher level of the fluoroalkane propellants than the other tissues analyzed (Table 1). The fluoroalkanes are readily soluble in benzene and the solution can be chromatographed directly without further treatment. As shown in Fig. 4 no interference occurs for benzene.

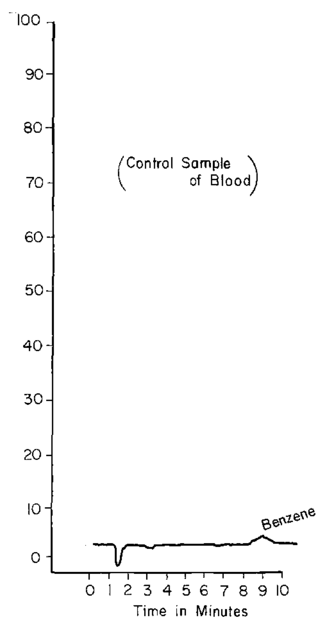

FIG. 4—*Gas liquid chromatography of a control sample of blood, when treated with benzene as recommended in extractive procedure.*

TABLE 1—*Distribution of fluoroalkanes in postmortem tissue.*

	Amount in Tissues (mg/100 g)[a]							
Case No.	71-1653		71-1783		71-1926		71-503	
	A	B	A	B	A	B	A	B
Lungs	1.1	1.7	1.9	...	4.0	5.0	0.6	1.0
Liver	0.2	0.5	0.4	...	1.0	3.0	0.1	0.6
Blood	0.1	0.7	0.5	...	1.2	2.0	0.2	0.3
Brain	0.05	0.04	...	...	...	...	...	...

Column A = Values for Dichlorofluoromethane
Column B = Values for Trichlorofluoromethane
[a] Wet specimen.

The number of cases analyzed in our laboratory is too few in number to permit an estimate as to probable toxic level of fluoroalkanes in human tissues. The mechanism of action has been explored by a number of investigators. Taylor and Harris [*4*] have stated that inhalation of aerosol propellants may cause sudden death. Bass [*5*] found that healthy persons inhaling these compounds may die within moments. Taylor et al [*6*], on the basis of their work on monkeys, suggested that some deaths after propellant inhalation may be caused by ventricular tachycardia or fibrillation. Flowers and Horan [*7*] believe that death due to fluoroalkane propellants is caused by the propellants themself and not due to anoxia. Their work using dogs exposed to high concentrations of the propellants produced disturbances in heart rhythms. These reports, therefore, seem to contradict widely published statements [*8*] that fluoroalkane propellants are safe.

References

[*1*] Wilson, C. O., Giswold, O., and Doerge, R. T., *Textbook of Organic Medicinal and Pharmaceutical Chemistry*, 6th ed, J. B. Lippincott Company, Philadelphia, 1971, pp. 137–138.

[*2*] Gudzinowicz, B. J., *Gas Chromatographic Analysis of Drugs and Pesticides*, Vol. II, Marcel Dekker Inc., New York, 1957, pp. 164–171.

[*3*] Lovelock, J. C., "Affinity of Organic Compounds for Free Electrons with Thermal Energy: Its Possible Significance in Biology," *Nature*, Vol. 189, 1961, pp. 729–732.

[4] Taylor, G. J. and Harris, W. S., "Cardiac Toxicology of Aerosol Propellants," *Journal of theAmerican Medical Association*, Vol. 214, 1970, pp. 81–85.

[*5*] Bess, M., "Sudden Sniffing Death," *Journal of the American Medical Association*, Vol. 212, 1970, pp. 2075–2079.

[*6*] Taylor, G. J., IV, Harris, W. S., and Boddonoff, M. D., "Ventricular Arrhythmias Induced in Monkeys by the Inhalation of Aerosol Propellants," *Journal of Clinical Investigation*, Vol. 50, 1971, pp. 1546–1550.

[*7*] Flowers, N. C. and Horran, L. S., "Nonanoxic Aerosol Arrhythmias," *Journal of the American Medical Association*, Vol. 219, 1972, pp. 33–37.

[*8*] *Physicians Desk Reference of Pharmaceutical Specialities and Biologicals*, 24th ed, Medical Economics, Inc., Oradell, N.J., 1971, pp. 1081, 1492.

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