Environment Protection Engineering

Vol. 12 1986 No. 3

COMMUNICATION

IRENEUSZ STĘPIEŃ*

TOXIC CONTAMINANTS IN THE VISTULA RIVER

1. INTRODUCTION

The modern analytical techniques of water quality measurement must be highly specific and sensitive, because organic compounds can cause serious problems at very low concentrations (for example, some are toxic to aquatic organisms at concentrations below 10 $\mu g/dm^3$, McGUIRE [4]). The method that best meets these requirements for toxic contaminants in water is gas chromatography-mass spectrometry. The mentioned technique since many years has been used extensively in Environmental Contaminants Division, National Water Research Institute, Burlington, and was also applied for the purpose of analysis reported in this paper.

2. ANALYTICAL PROCEDURE

600 cm^3 of DCM (dichlorometane, CH_2Cl_2) was added to 1 dm^3 sample** of the Vistula River water and the mixture was stirred for 10 minutes to ensure thorough mixing of the contents. After this operation the strirrer was removed and the sample was left for 5–10 minutes until DCM settled down on the bottom of the container. DCM extract was transferred next to the 1 dm^3 screw capped glass bottle using 1–2 psi (pounds per square inch) nitrogen pressure. The excess of water from glass bottle was returned to the extraction container. The extraction procedure was repeated twice using 100 cm^3 DCM each time. The combined extract was dried by passing through a 5 cm bed of anhydrous Na_2SO_4 and 5 cm^3 of 2,2,5-trimethylpentane was added as a keeper. The dried sample was evaporated to approximately 2 cm^3 on the rotovapor at 25° C. The concentrated extract was allowed to flow by gravity through pasteur pipet mini-columns containing 2.5 cm 44% H_2SO_4 on silica gel topped with 0.5 cm anhydrous Na_2SO_4. The extract was eluted from the columns with 2×2 cm^3 of hexane. The combined eluant was collected in 10 cm^3 Kuderna–Danish tube and concentrated to a final volume of 500 µl (0.5 cm^3) under a stream of dry nitrogen on an N-Evap concentrator at 25° C. The final extract was analysed by electron capture gas chromatography using capillary glass columns.

* Office for Environment Protection and Water Management, ul. Wspólna 30, 00-930 Warszawa, Poland.

c/o National Water Research Institute, 867 Lakeshore Rd, Burlington, Ontario, Canada L7R 4A6.

** The sample was collected in Warsaw, approximately in the middle of the town, close to the left riverside, 18 November, 85.

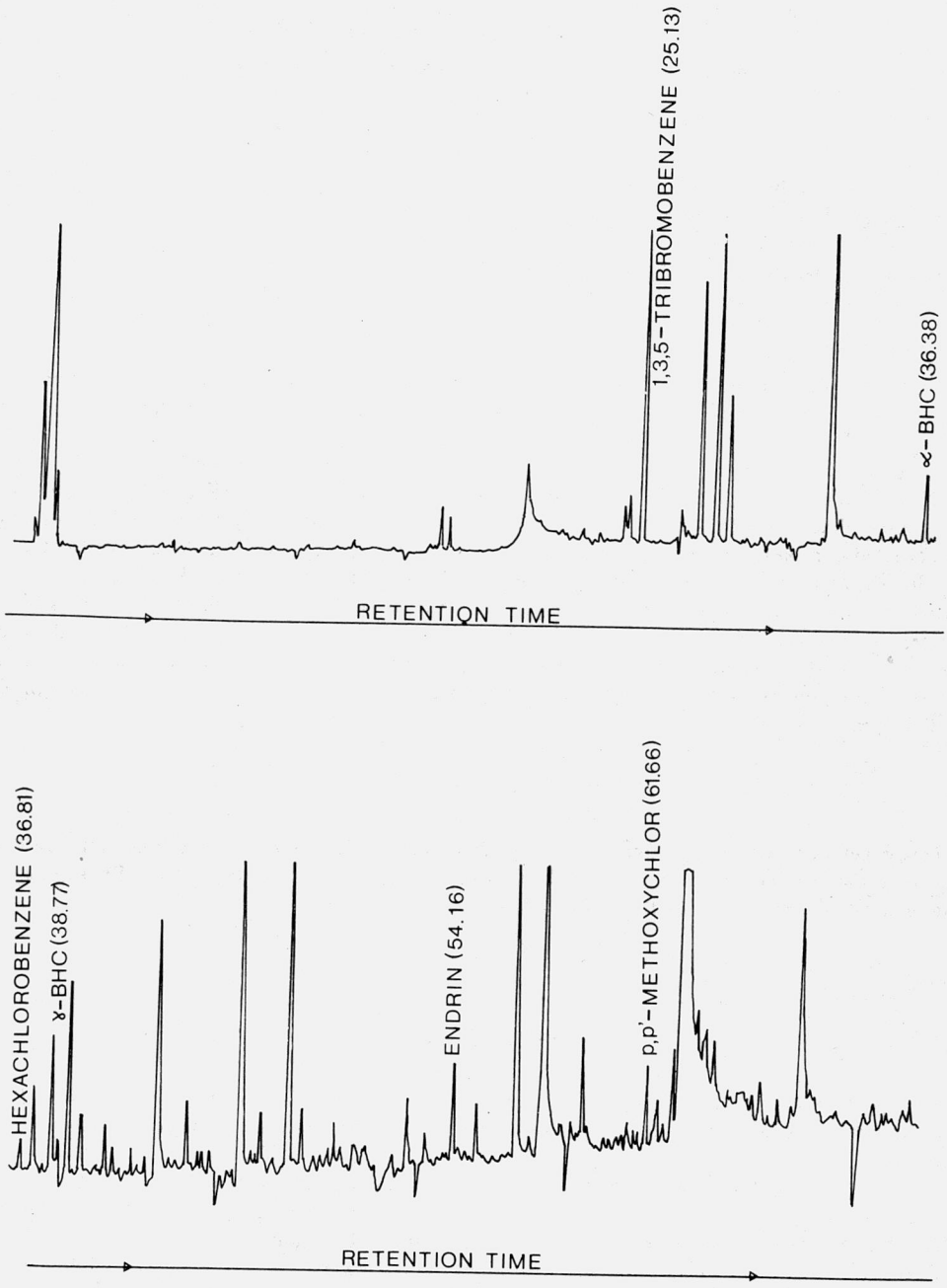
3. RESULTS AND DISCUSSION

Because of lack of information concerning chemical composition of the sample, the Niagara River standard (Fox [2]) was used as a calibrate run. Table 1 shows a set of standard compounds; the chemicals are listed in order of gas chromatographic elution which is also approximately the order of

Table 1

Specification of toxic chemicals in Niagara River calibrate run

Compound	Retention time (minutes)
1,3-dichlorobenzene	7.72
1,4-dichlorobenzene	8.17
1,2-dichlorobenzene	9.09
hexachloroethane	10.15
1,3,5-trichlorobenzene	12.52
1,2,4-trichlorobenzene	13.95
1,2,3-trichlorobenzene	15.99
hexachlorobutadiene	16.23
1,2,4,5-tetrachlorobenzene	21.14
1,2,3,4-tetrachlorobenzene	23.26
1,3,5-tribromobenzene	25.13
pentachlorobenzene	29.29
2,3,5,6-tetrachloronitrobenzene	32.70
2,3,4,5-tetrachloronitrobenzene	35.12
α-BHC	36.38
hexachlorobenzene	36.80
β-BHC	38.41
γ-BHC	38.77
pentachlorobenzene	39.13
heptachlor	43.72
aldrin	46.01
octachlorostyrene	48.61
heptachlor epoxide	49.06
γ-chlordane	50.33
o,p'-DDE	50.87
α-endosulfan	51.09
α-chlordane	51.34
dieldrin	52.79
p,p'-DDE	52.95
o,p-DDD	53.45
endrin	54.14
β-endosulfan	54.78
p,p'-DDD	55.60
o.p-DDT	55.76
p,p'-DDT	57.93
p,p'-methoxychlor	61.66
mirex	63.71



Chromatogram of Vistula River water sample, 18 November, 1985, Warsaw

Table 2

Detected compounds in the Vistula River sample, 18 November, 1985

Compound	Retention time (minutes)	Concentration (ng/dm^3)
α-BHC	36.36	2
hexachlorobenzene	36.81	9
γ-BHC	41.46	11
endrin	54.16	7
p,p'-methoxychlor	61.66	17

their decreasing solubility in water. Figure shows chromatogram of the analysis run (volume of injected sample – 5 μl). The identified compounds and their corresponding retention times are shown in the tab. 2.

The quantitative analysis of concentration was done by the internal standard method (FREEMAN [3]). 20 μl of 1,3,5-tribromobenzene was added to the analysed extract – this compound is usually used as internal standard for detection of toxic contaminants in Niagara River (FOX and CAREY [1]). The computed concentrations (in ng/dm^3, 1 nanogram $= 10^{-9}$ g) are given in column 3 of the tab. 2. Generally, the concentrations of the detected compounds are of the same range like concentrations of chlorinated organic contaminants in the Niagara River (FOX and CAREY [1]). However, it is likely that reported concentrations are underestimated, because the sample was transported via air and stored in the cold room three weeks until the analysis was completed. It is also worth noting that a number of unidentified compounds with much more significant concentration might be easily found in the sample.

ACKNOWLEDGMENTS

This paper was completed during the author's fellowship in the National Water Research Institute, Burlington, 1984/85. The author's grateful thanks are also given to M. E. Fox of the Environmental Contaminants Division, who kindly performed laboratory analysis.

REFERENCES

[1] FOX M. E., CAREY J. H., *Transport of selected organochlorine contaminants in the Niagara River plume*, Environmental Contaminants Division, National Water Research Institute, Burlington 1986, Canada.

[2] FOX M. E., *A practical sampling and extraction system for the quantitative analysis of organochlorine contaminants in filtered water and suspended solids*, Environmental Contaminants Division, National Water Research Institute, Burlington 1985, Canada.

[3] FREEMAN R. R., *High Resolution Gas Chromatography*, Hewlett–Packard Company, 1981, 196 p.

[4] MCGUIRE J. M., WEBB R. G., *Organic water pollutant analysis by gas chromatography-mass spectrometry*, [In:] *Water Quality Measurement*, Mark H. B. and Mattson J. S., (Ed.) Marcel Dekker Inc., New York 1981, 1–33.