

## Note

### Recovery of *s*-triazines from water and their analysis by gas chromatography with photoionization detection

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For the trace analysis of *s*-triazine herbicides in water, the methods of enrichment generally employed are liquid–liquid extraction<sup>1</sup> and sorbent extraction<sup>2,3</sup>. The resulting extracts are then analysed by gas–liquid chromatography (GLC)<sup>1–3</sup>, high-performance liquid chromatography (HPLC)<sup>2</sup> or thin-layer chromatography (TLC)<sup>4,5</sup>. In this study, the possibility of the isolation of *s*-triazines from water using continuous steam distillation–extraction (SDE) prior to GLC analysis was investigated.

#### EXPERIMENTAL

##### *Preconcentration of s-triazines*

An apparatus for continuous steam distillation–extraction was used<sup>6</sup>. The conditions were as follows: volume of aqueous sample, 400 ml; total volume of diethyl ether, 4 ml; 240 g of potassium chloride were added to the sample. As in previous work<sup>6,7</sup>, lower recoveries were obtained in the absence of a salting-out effect.

##### *Analyses of the SDE extracts*

Extracts and reference mixtures, representing 100% recoveries of herbicides from water, were analysed by GLC on a Chrom 5 gas chromatograph (Laboratorní Přístroje, Prague, Czechoslovakia). A glass column (1.2 m × 3 mm I.D.) packed with 2% Carbowax 20M on Gas-Chrom Q was used. *n*-C<sub>22</sub>, C<sub>24</sub> and C<sub>26</sub> hydrocarbons were used as internal standards. For the detection of *s*-triazines a photoionization detector, Model PI 52-02 (HNU Systems, U.S.A.) was used.

##### *Recovery study*

The recoveries of seven triazine herbicides, propazine, terbutylazine, atrazine, prometryn, terbutryn, desmetryn and simazine, from water by SDE were investigated. In Table I are summarized the recoveries obtained after 1 h of SDE, when the pH of the aqueous sample was adjusted to 5, 7 and 9. It is seen that the recoveries of some *s*-triazines are poor. The recoveries also do not depend on the pH. The last column of Table I shows the recoveries, obtained after 3 h of SDE. Only simazine is not recovered sufficiently. The recoveries of *s*-triazines from water are also illustrated in Fig. 1.

TABLE I

RECOVERIES OF *s*-TRIAZINES FROM WATER BY SDE

Concentrations ( $\mu\text{g/l}$ ) of *s*-triazines in aqueous sample: propazine, 18.0, terbutylazine, 44.0; atrazine, 79.5; prometryn, 55.0; terbutryn, 47.5; simazine, 15.5 and desmetryn, 240.0. Amount of KCl added to the sample: 240 g.

<i>s</i> -Triazine	Recovery (%) <sup>*</sup>			
	1 h (5.0)	1 h (7.0)	1 h (9.0)	3 h (7.0)
Propazine	77.0	74.0	72.5	83.9
Terbutylazine	82.0	81.3	74.1	88.9
Atrazine	50.0	51.2	40.1	88.7
Prometryn	84.7	85.0	79.3	84.0
Terbutryn	77.1	83.0	83.5	83.4
Simazine	<5	<5	<5	<10
Desmetryn	29.1	28.0	29.0	69.0

\* After stated periods of SDE. Values in parentheses are of the pH of the sample.

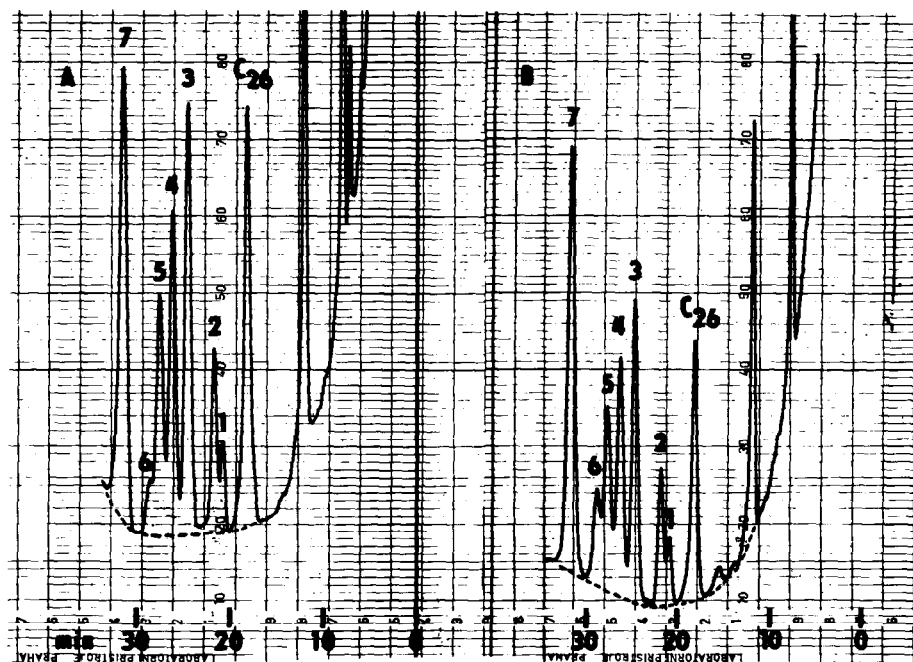


Fig. 1. Analyses of an extract obtained by SDE (A) and of a reference mixture (B). Duration of SDE: 3 h; 240 g of KCl added to a 400-ml aqueous sample. Column: glass (1.2 m  $\times$  3 mm I.D.), packed with 2% Carbowax 20M on Gas-Chrom Q; temperature 120–160°C at 4°/min, then 160–185°C at 1°/min. Carrier gas (nitrogen) flow-rate: 20 ml/min. Injection temperature: 240°C. Amount injected: 5  $\mu\text{l}$ . Detection: photoionization, 10.2-eV lamp, 205°C; attenuation 1  $\times$  40, except for peak 7, 1  $\times$  80. Peaks: 1 = propazine; 2 = terbutylazine; 3 = atrazine; 4 = prometryn; 5 = terbutryn; 6 = simazine and 7 = desmetryn. Concentrations as in Table I. Hydrocarbons *n*-C<sub>22</sub>, C<sub>24</sub> and C<sub>26</sub> were used as internal standards.

## DISCUSSION AND CONCLUSION

SDE can be used for the analysis of propazine, terbutylazine, prometryn, terbutryn, atrazine and desmetryn. It is not suitable for simazine, probably because of the low volatility of this compound under conditions of steam distillation.

Linear calibration graphs were obtained and the detection limit of the method is of the order of micrograms per litre. If the extracts are concentrated by evaporation, the detection limit decreases, no losses of *s*-triazines during evaporation of diethyl ether being observed.

However, the fact that the method is not suitable for the analysis of simazine and that it takes 3 h for atrazine is a serious handicap.

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