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### High-performance liquid chromatographic determination of pyrazon in water

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Pyrazon is the common name for 5-amino-4-chloro-2-phenyl-3-pyridizone which is used as a pre- and post-emergence herbicide, particularly for sugar beet and beet crops. The usual commercial formulation is Pyramin which contains 80% technical pyrazon. Pyrazon has been determined in plants and soils using colorimetric and thin-layer chromatographic methods<sup>1-3</sup>. A semi-quantitative method has been proposed for its determination in water<sup>4</sup> which involves extraction with chloroform-ethyl acetate, separation by thin-layer chromatography and a visual estimation of the level of pyrazon by comparison with reference plates. A method for the analysis of pyrazon in water using high-performance liquid chromatography (HPLC) is presented in this note.

#### EXPERIMENTAL

Pyrazon was isolated from water samples (500 ml) by rotary evaporation to dryness *in vacuo*, extraction of the solid residue with methanol (2 × 25 ml) and further evaporation of the methanol extract (to approx. 2 ml). Final concentration (to 0.5 ml) was achieved by removal of methanol under a stream of nitrogen.

#### HPLC analysis

The equipment used consisted of two Model 6000A solvent delivery systems (Waters Assoc.), a Model 660 gradient former (Waters Assoc.) and a Model CE 212 variable wavelength UV monitor (Cecil Instruments) operated at 270 nm. Syringe injections were made through a stop-flow septumless injection port (H. S. Chromatography Packings). The column (15 cm × 7 mm I.D.) was packed in an upward manner<sup>5</sup> with Spherisorb-ODS (Phase Separations, Queensferry, Great Britain), by a slurry procedure using acetone as slurry medium. A pneumatic amplifier pump (Haskel DST-122 supplied by Olin Energy Systems) was used to pack the column. A linear gradient was established from two solvent mixtures consisting of (A) 10% methanol in 0.1% acetic acid in water and (B) 80% methanol in 0.1% acetic acid in water. The initial concentration was 35% B in A and the final concentration was 100% B with the gradient terminated after 20 min. The flow-rate was maintained at 2.0 ml/min throughout the analysis.

## RESULTS AND DISCUSSION

The recovery efficiencies of pyrazon from water were determined by analysing samples of river water spiked at levels of 10, 50, 100 and 200  $\mu\text{g/l}$ . The recovery efficiency varied from 17% at the 10  $\mu\text{g/l}$  level to over 90% recovered at 200  $\mu\text{g/l}$ . A calibration curve was constructed for quantitation of water samples (Fig. 1).

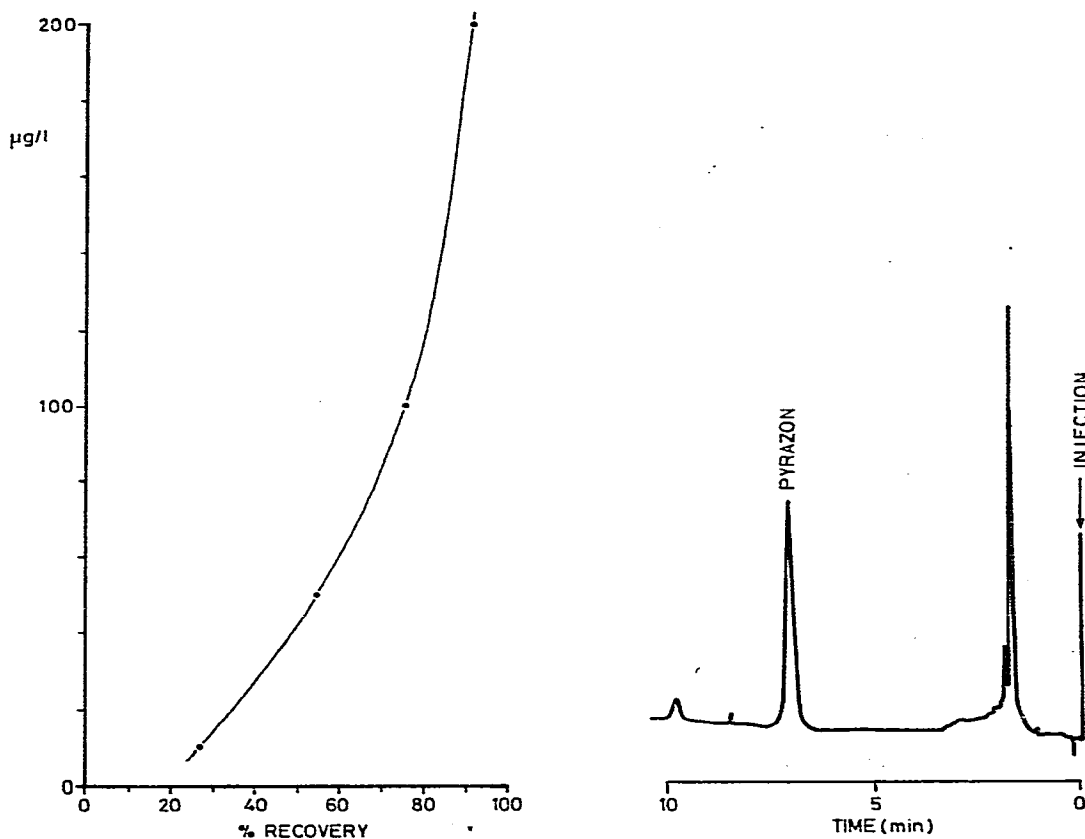


Fig. 1. Calibration curve for extraction efficiency of pyrazon from surface water samples.

Fig. 2. Chromatogram of a standard solution of Pyrazon.

The HPLC chromatogram for a standard solution of Pyrazon in methanol (Fig. 2) shows a major peak due to pyrazon and a minor peak at longer retention time. The minor peak was present in all of the standard solutions analysed, the spiked water extracts and in the polluted surface water extracts.

The chromatogram from the analysis of a spiked water extract (Fig. 3) shows the peak for pyrazon clearly separated from a large, essentially unretained peak. This large peak is usually observed in the analysis of water extracts and appears to arise from excluded components.

The chromatogram of a polluted river water extract (Fig. 4) exhibits the peak

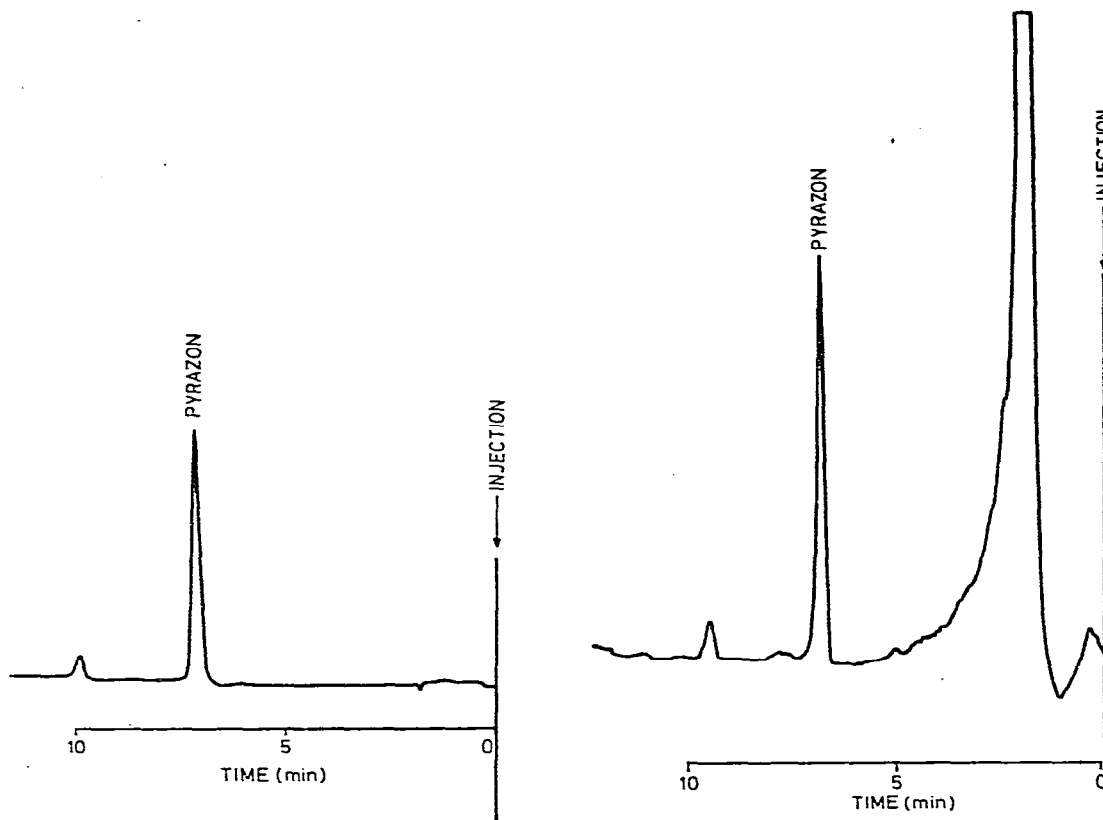


Fig. 3. Chromatogram of a river water extract spiked at 100  $\mu\text{g/l}$  with Pyramin.

Fig. 4. Chromatogram of a polluted river water extract.

due to pyrazon and the minor peak observed previously in the chromatograms of the standard solution of Pyramin and the spiked water extract. This provides confirmatory evidence that the pollution is due to the herbicide formulation Pyramin.

#### ACKNOWLEDGEMENT

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