

EXTRACTION AND HIGH PRESSURE LIQUID  
CHROMATOGRAPHIC DETERMINATION OF ATRAZINE, PYRAZON  
AND SIMAZINE IN WATER AND SOIL

BY

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## ABSTRACT

(a) Pyrazon is the common name for 5-amino-4-chloro-2-phenyl-3-pyridazinone which is used as pre- and post-emergence herbicide. A simple, fast method for extracting and concentrating pyrazon in water using C<sub>18</sub> Sep-Pak<sup>TM</sup> cartridge (Waters Associates Inc.) and consequently determining the herbicide using reversed-phase high pressure liquid chromatography (HPLC) has been developed. The recovery of pyrazon from water was found to improve considerably when sodium chloride was added and dissolved in the water. The recovery of pyrazon was found to depend on the volume of water passing through C<sub>18</sub> Sep-Pak cartridge and also on the amount of the sodium chloride dissolved in the water. A 500 cm<sup>3</sup> sample of lake water with dissolved 25% NaCl was passed through a C<sub>18</sub> Sep-Pak cartridge and the adsorbed pyrazon eluted with about 5 cm<sup>3</sup> of dichloromethane. The dichloromethane solution was evaporated under nitrogen at a water bath (50°C), and the residue dissolved in methanol. Pyrazon was determined using reversed-phase HPLC with 55% sodium phosphate buffer (0.05, M pH 5.0) and 45% methanol as the mobile phase. Ultra violet (UV) detector at fixed wavelength of 280 nm was used for detecting pyrazon. The average recovery of pyrazon was 99%. The detection limit reached was 2 ppb.



(b) A method for extracting pyrazon from soil and consequently determining the herbicide using reversed-phase HPLC has been developed. 50 g soil sample containing pyrazon was shaken with 150 cm<sup>3</sup> of acetone-methanol mixture. The acetone-methanol filtrate was evaporated in a rotary evaporator at a temperature of 80-90°C. The residue was dissolved in 100 cm<sup>3</sup> of distilled water and 25 g of sodium chloride dissolved in the aqueous solution. The aqueous solution was passed through a C<sub>18</sub> sep-pak cartridge and pyrazon eluted with dichloromethane. Pyrazon dissolved in methanol was determined in reversed-phase HPLC with 55% sodium phosphate buffer (0.05 M; pH 5.0) and 45% methanol as the mobile phase. A UV detector at a fixed wavelength of 280 nm was used. The average recovery of pyrazon was 96%. The detection limit reached was 0.04 ppm.

(c) A simple, fast method for extracting and concentrating atrazine and simazine in water using C<sub>18</sub> sep-pak cartridge (Waters Associates Inc.) and consequently determining the herbicides using reversed-phase HPLC has been developed. A 500 cm<sup>3</sup> sample of lake, river and ocean water containing atrazine and simazine was passed through a C<sub>18</sub> sep-pak cartridge and the adsorbed herbicides eluted with about 5 cm<sup>3</sup> of dichloromethane. The CH<sub>2</sub>Cl<sub>2</sub> solution was evaporated under nitrogen and the residue dissolved in methanol. Atrazine and simazine were determined using

reversed-phase HPLC with 45% sodium phosphate buffer (0.05 M, pH 5.0) and 95% methanol as the mobile phase. A UV detector at a fixed wavelength of 230 or 254 nm was used for detecting the two herbicides. A recovery of about 100% was obtained for both herbicides. A detection limit of 4 ppb was reached.

(d) A method has been developed for extracting and determining residues of atrazine and simazine in soil. 50 g of soil containing the two herbicides were shaken with about 160 cm<sup>3</sup> of a mixture of chloroform and ethylacetate. The solvent was evaporated off in a rotary evaporator at a temperature of about 80°C. The residue was dissolved in 20 cm<sup>3</sup> mixture of dichloromethane and methanol (3:7) and evaporated to about 3 cm<sup>3</sup>. 40 cm<sup>3</sup> of distilled water were added to the 3 cm<sup>3</sup> of organic solution and passed through a C<sub>18</sub> sep-pak cartridge. The herbicides were eluted from the C<sub>18</sub> cartridge with about 5 cm<sup>3</sup> of dichloromethane. Dichloromethane was evaporated off by passing nitrogen over it at a water bath (50°C) and the residue dissolved in methanol. The two herbicides were determined using reversed-phase HPLC with 45% sodium phosphate buffer (0.05 M, pH 5.0) and 55% methanol as the mobile phase. A UV detector at a fixed wavelength of 230 or 254 nm was used for detecting atrazine and simazine



respectively. An average recovery of 91% and 93% was obtained for atrazine and simazine respectively. The detection limit reached was 0.1 ppm.