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On-line Coupled Extraction and Separation using Superheated Water for the Analysis of Triazine Herbicides

Ruziyati Tajuddin and Roger M. Smith*

Chemistry Department, Loughborough University, Loughborough, LE11 3TU Leicestershire, UK.

INTRODUCTION

Superheated water extraction (SWE) has been coupled on-line with superheated water chromatography (SWC) via a solid phase trap. The extraction, trapping, release from the trap and chromatography are all controlled by altering the temperature and no organic solvents are required at any stage of the process.

Cold and warm wash aqueous steps were included to remove humic acids and other interfering components of the compost samples.

EQUIPMENT

The equipment is shown below.

![Equipment Diagram](Image)

1. Extraction phase

The sample in the extraction cell was first flushed with ambient water to wash very polar extractable components to waste.

The outlet flow was then switched to the trap column and the sample heated to 170 ºC. As the aqueous solution left the extraction cell it was cooled and passed to the X-Terra trap column to collect and focus the aqueous solution left the extraction cell and was cooled and passed to the X-Terra trap column to collect and focus it.

2. Clean up

The trap was washed with water at 60 ºC to remove medium polar components and these were passed to waste.

3. Separation

The flow of water was then switched to the analytical column and the trap was heated rapidly to 200°C to release the analytes to the PGC analytical column. The analytical column was programmed from 160 to 200°C to chromatograph the extracts.

RESULTS

Extraction from the seed compost spiked at 20 µg/g

Peaks:

1. propazine, 2. atrazine, 3. simazine, 4. ametryn, 5. terbutryn extracted from 500 mg seed compost at 170 ºC.

The recoveries of the triazines were examined at different extraction temperatures from the seed compost.

Recoveries of the chloro-triazines decreased at the higher temperature due to decomposition, however the recoveries of the ethyl-methyl-triazines increased at the higher temperature because they are more thermally stable. In previous work, McGowin et al [2] reported that the recoveries of chloro-triazines (atrazine and propazine) decreased significantly from 110 ºC to 250 ºC, but the recovery of ametryn was less affected.

The apparent recovery of 150% terbutryn at 210 ºC was because the peak was enhanced by a matrix interference peak, which was confirmed when non-spiked compost was examined.

The limits of detection ranged from 1- 2.4 µg/g from the seed compost.

CONCLUSION

# The construction of the on-line SWE-SWC with the inclusion of clean-up steps, enabled quantitative analysis of the triazines from complicated sample matrices. The higher organic matter in the sample matrix (ericaceous compost), the more interference is found, which affected the measurement of the recoveries.

# 170 °C is the optimum SWE temperature for the triazines. The recoveries of chloro-triazines decreased at high temperatures due to the thermal degradation.

# The full sequence of clean-up, extraction and collection further clean-up and chromatographic separation were carried out with the use of no organic solvents in a continuous on-line system. Consequently all the analysis that were in the compost are carried through to the detector and sensitivity can be high.

REFERENCES


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