

DOI: <http://doi.org/10.21698/simi.2020.ab35>

DETERMINATION OF THIABENDAZOLE FROM ENVIRONMENTAL MATRICES BY HPLC WITH FLUORESCENCE DETECTION

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Keywords: *environmental matrices, fluorescence, liquid chromatography, thiabendazole, Strata X*

Introduction

Thiabendazole 2-(4-thiazolyl)-1H-benzimidazole (TBZ), known as E233, is a pesticide of the benzimidazole class with an imidazole ring, used as a systemic fungicide in agriculture, and also as a broad-spectrum antiparasitic agent for various animal species. Thiabendazole is also widely used in the treatment of fruits and vegetables, before and after harvesting to control a variety of diseases, such as mold, rot and stains caused by various fungi. TBZ is applied by fruits dipping, by spraying or as an ingredient in the wax applied to the peel of fruits and vegetables to ensure their freshness. Because of these practices, the occurrence of thiabendazole has been reported in surface waters and sediments, in fruits peels and in effluents from the agriculture and food industry.

Materials and methods

Extraction of thiabendazole from wastewater and surface water was performed by solid phase extraction (SPE) on Strata X (33 µm, 500 mg / 6mL) cartridges, using a Dionex Autotrace 280 (Thermo Scientific) automated extractor. The volume of the sample was 200 mL and the pH was adjusted to 8 value with NH₄OH, 0.24%. The polymeric sorbent was preconditioned with 10 mL methanol, and 10 mL ultrapure water. The cartridge was air-dried and finally the compound was eluted with methanol. The eluate was evaporated to dryness in gentle nitrogen stream at a temperature of 40°C and the analyte was reconstituted with 1 mL of 60/40 methanol/ultrapure water. TBZ was further analyzed with an Agilent 1200 LC system endowed with a fluorescence detector. Separation was achieved on a Zorbax Eclipse Plus C18 column (150 x 4.6 mm, 5 µm dp), at a temperature of 25°C using an isocratic mobile phase consisting of methanol: water (60:40, v/v). The flow rate was 1.0 mL/min and the injected volume was 20 µL. TBZ fluorescence detection was performed using an excitation wavelength of 280 nm and monitoring analyte emission at 315 nm.

Results and conclusions

The performance of the SPE-HPLC-FLD method was evaluated for wastewater and surface water in terms of linearity, limit of quantification, precision (intra-day and inter-day) and method accuracy. Linear detector response was observed after a 6-point

calibration in the range of 50 - 2000 µg/L with a correlation coefficient $R^2=0.9999$. Figures 1 and 2 show the above-mentioned linear regression and a typical HPLC-FLD chromatogram of thiabendazole.

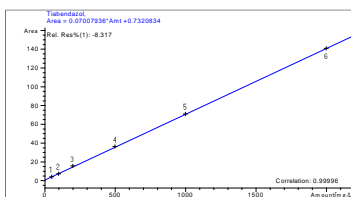


Figure 1. Linear regression of thiabendazole

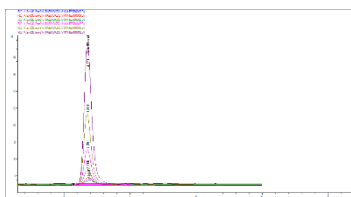


Figure 2. Chromatograms of thiabendazole calibration standards

Table 1. Performance parameters determined for thiabendazole in wastewater samples

Compound	Retention time (min)	LOQ (µg/L)	Recovery rate (%)	Precision	
				Intra-day precision (%)	Inter-day precision (%)
Thiabendazole	3.354	0.06	77.5	5.2%	10.6%

Successful application of the developed SPE-HPLC-FLD method was proven by the analysis of four samples, of influent and effluent, taken from two municipal wastewater treatment plants (Targoviste and Campulung Muscel), but also two surface water samples taken downstream and upstream of the Targoviste wastewater treatment plant.

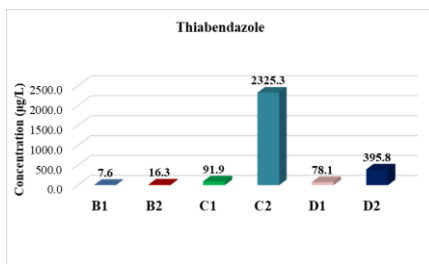


Figure 3. Concentration of thiabendazole found in environmental matrices

B1, B2 – downstream / upstream wastewater treatment plant Targoviste;
C1, C2 – influent / effluent wastewater treatment plant Targoviste;
D1, D2 – influent / effluent wastewater treatment plant Campulung Muscel.

The method was applied for detection of thiabendazole in wastewater and surface water samples. Concentrations in the range 7.6 - 2325.3 µg/L were obtained.

Acknowledgement

This study has been conducted within the framework of the National Research Program Nucleu, contract number 20N/2019, PN 19 04 01 01.