DETERMINATION MONOCROTOPHOS BY HPLC/UV TECHNIQUES FROM VEGETABLES SAMPLES

Cozma Alina*

**University of Oradea, str. Universităţii nr. 3-5, Oradea, Bihor, Roumania*
acozma@uoradea.ro

Abstract
The aim of this study is the presentation a method for the determine monocrotophos from vegetables samples by HPLC/UV techniques.Pesticides residues are extracted from the test portion following the acetone. The mixture is centrifuged,filtered and directly analyzed by the method proposed. The tested samples showed contamination with low ,but measurable amounts of residues.The limit of detection for the method was calculated from regression data, and recovery results were in the range of 85- 109%.

Key words: pesticide residue, vegetables samples, HPLC, UV

INTRODUCTION

Monocrotophos is an organophosphorus pesticides. Pesticides residue analysis plays an important role in food quality for evaluating food safety and possible risks to human health. Organophosphorus pesticides (OPPs) are widely used for agricultural practices because of its high effectivity and relatively low price. Compared with organoclorine pesicides, OPPs demonstrate relatively low environmetal persistence, but a high toxicity [1].
Therefore, the OPPs residue in food has been strictly regulated by government in all countries in order to determine whether the concentrations of the pesticides used exceed their maximum residues limits (MRLs) [2].

MATERIALS AND METHODS

A method is described for the analysis of polar, non – QuEChERS-amenable pesticides (log K_{ow}<- 2) in vegetables and processed products thereof.Five samples of vegetables comprising cauliflower (5) were collected from different farmer s around Oradea.Samples were collected from fields when the samples were ready for sale and were refrigerated and analysed with in a week of collection. All the samples were extracted fresh.

Residues are extracted from the test portion following the acetone.The mixture is centrifuged, filtered and directly analyzed by HPLC with detection UV. Quantification is performed with the help of isotopically labeled internal standards, which are added directly to the test portion at the beginning of the procedure.

Apparatus for extraction:

- ultrasound extraction apparatus, Elmasonic S15H;
- 50 mL centrifuge tubes with screw caps;
- automatic pipettes, suitable for handling volumes of 10 to 100µL, 200 to 1000 µL and 1 to 10 mL;
- 10 mL solvent – dispenser methanol;

- centrifuge, suitable for the centrifuge tubes employed in the procedure and capable of achieving at least 2000g;
- syringes, 2 or 5 mL disposable syringes;
- syringe filters, 0,45 μm pore size;
- rotavapor;

Substances:

- acetone, HPLC quality;
- acetonitrile, HPLC quality;
- concentrated acid formic (> 95%);
- citric acid monohydrate;
- dimethylamine;
- ammonium formate;
- water deionizate;
- monocrotophos standard;
- N – octadecyl (standard intern);

Method:

Of 50 g of cauliflower and was macerated with 5 -10 g anhydrous sodium sulfate in blender to make a fine paste. The macerated sample was extracted with 100mL acetone , then the ultrasound extraction is applied for 30 minutes at a temperature of 55C. The extract is filtered, washed for 2-3 times with acetone and then introduced in a 25 ml balloon.

The sample is placed in a concentration rotary evaporator. The concentrated product is introduced in a balloon of 10ml and brought to sign with acetone 100 μg N – octadecyl is added and it is brought to the sign with acetone. After concentrating the eluate on rotary evaporator, final volume was made to 2 mL for HPLC analysis.

Measurement Conditions for HPLC with detection UV

For the analysis are used:

- HPLC system with UV detector- VIS, model Young – Lin AT 7000;
- gradient pomp930 D, Yuong Lin Acme 9000; flow: 0,3 mL/ min;gradient: 100%A in 8 min to 50 or 10 0% B hold B for about 7 min;
- UV detector –VIS 732 D, Yuong Lin Acme 9000;
- PN Rheodyne injector; injection volume is 10μL
- eluent A, acetone;
- eluent B, 1 mM citric acid in water adjusted to pH 11 with dimethylamine;
- column Dionex 250mm;

RESULTS AND DISCUSSION

All five samples of cauliflower were analysed and were found contaminated with monocrotophos in the range 0,003 – 0,192μg/g.

As a result of chromatographic division, the chromatogram obtained is presented in following figure:

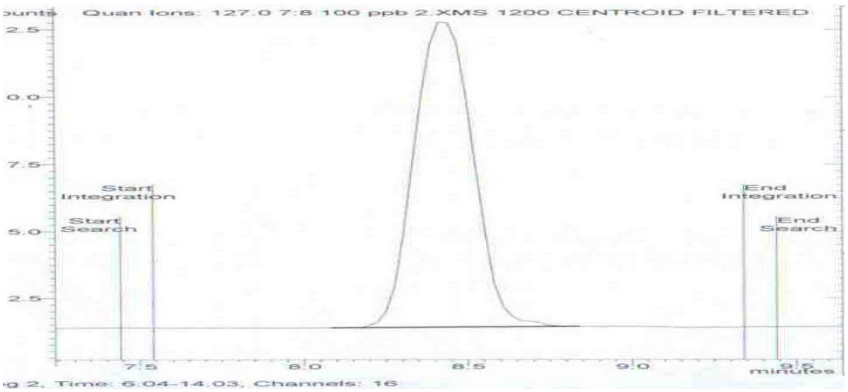

Fig.1 The chromatogram for monocrotophos standard

The chromatogram shows that the chromatographic parameters are the best possible, the degree of asymmetry of the spike being 1,05 . The retention time for the active substance was minimum 6,9. Using the calibration solution, the following calibration curve emerged, as shown in figure 2:

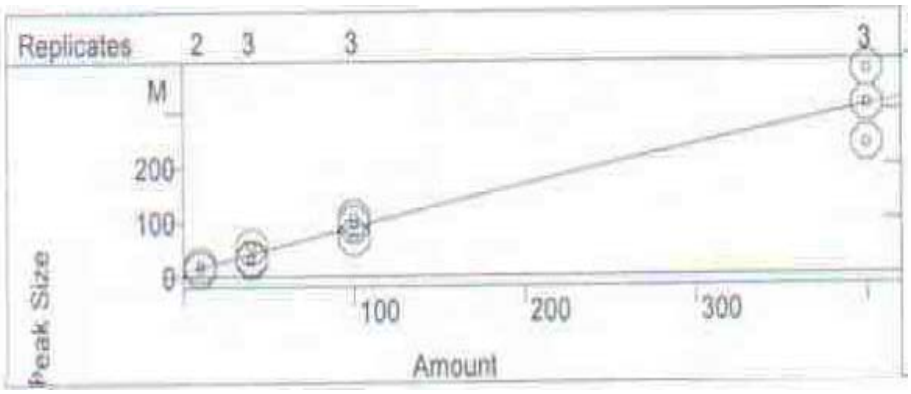

Fig.2 The calibration curve for calibration solution

The calibration curve is represented by an equation line: y = - 246,5330x2+ 8,4514e+5x with a correlation value of 0, 965137.

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CONCLUSIONS

The proposed method is precise, fast and accurate, a standard relative error being obtained. The recovery and reproductibility, based on matrix spiked standards, were acceptable for monocrotophos.The impurities and matrix effects from cauliflower were minimal and did not interfere with the quantitation of any target compound.

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