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Abstract

This study aimed to develop a suitable analytical method for pesticide detection using Gas Chromatography-Mass Spectrometry (GC-MS). The focus was on optimizing conditions for the analysis of 10 pesticides from the carbamate and organophosphate groups. The results showed that Condition B was the most appropriate, as it allowed the detection of 9 out of 10 pesticides, including carbaryl, carbosulfan, fenobucarb, methomyl, propoxur, diazinon, dichlorvos, ethion, and malathion, except for pirimiphos-methyl, which could not be analyzed due to its boiling point being higher than the column temperature. Additionally, Condition B also reduced the analysis time effectively. Regarding the extraction of pesticides from rice samples using solid-phase extraction, the choice of solvent was found to affect extraction efficiency. Acetone was the most suitable solvent for extracting methomyl, while ethyl acetate was the most effective for extracting fenobucarb. These findings highlight the importance of selecting the appropriate conditions and solvents for pesticide analysis. The developed method can be applied in the detection of pesticide residues in forensic samples to enhance the reliability and accuracy of the justice process.

Introduction

The examination of unknown substances is one of the responsibilities of the Chemistry and Physics Subdivision at the Police Forensic Science Center 5. The evidence submitted for analysis is diverse, with pesticides being among the most common types encountered. These chemicals are widely used in agriculture to prevent and control pests. However, such pesticides can pose serious health risks, particularly when contaminating food.

Among the commonly used pesticides, carbamates and organophosphates have received special attention due to their low cost and easy accessibility. However, both groups of chemicals are neurotoxic to humans and animals and carry the risk of misuse in some cases.

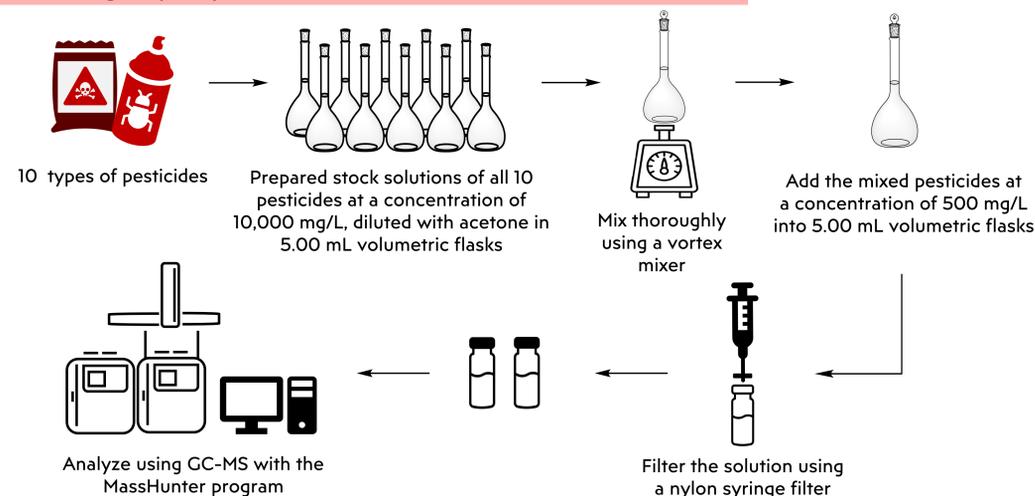
This research aims to develop methods for the analysis of carbamate and organophosphate pesticides in evidence samples, focusing on optimizing the operational conditions of gas chromatography/mass spectrometry (GC-MS) and selecting appropriate solvents for extraction. The goal is to effectively analyze these two groups of pesticides.

Objectives

- To determine optimal conditions for analyzing carbamate and organophosphate pesticides using GC-MS.
- To identify suitable solvents for extracting carbamate pesticides from contaminated rice samples.

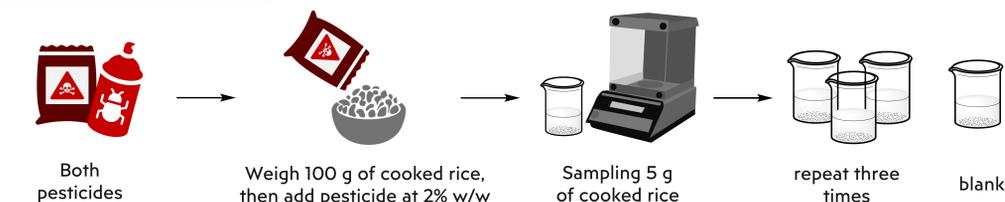
Methodology

1. Optimization of GC-MS Conditions for the Analysis of Carbamate and Organophosphate Pesticides

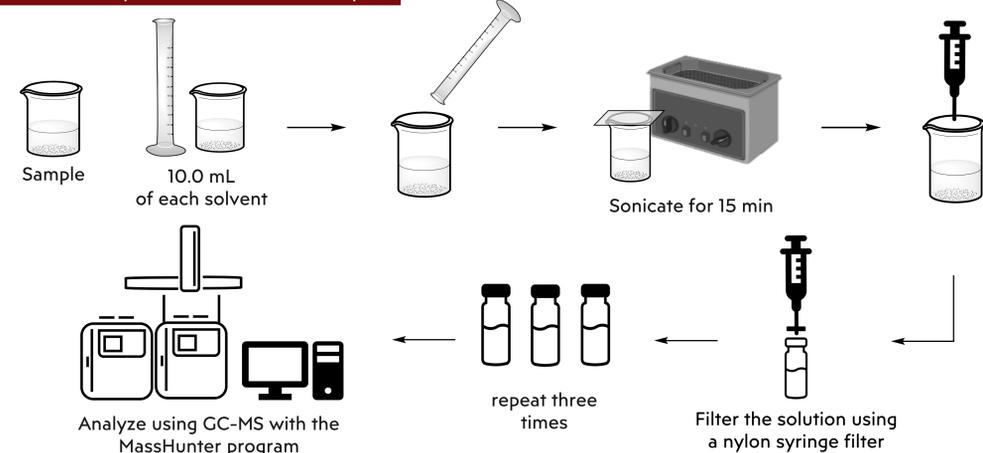


2. Finding the Appropriate Solvent for Extracting Carbamate Pesticides from Contaminated Rice Samples

Preparation of rice samples



Extraction of pesticides from rice samples



Analytical Instrument

Gas Chromatography-Mass Spectrometer (GC-MS)

Gas Chromatography: 7890B Agilent Technologies Gas Chromatograph

Column: Agilent Technologies DB-5MS capillary column of 30 m × 0.320 mm i.d. × 0.25 μm film thickness

Carrier Gas: Helium (99.9995%)

Detector: 5977A MSD Agilent Technologies Mass Spectrometric

Library Search: DEMO.L

Results and Discussion

1. Optimization of GC-MS Conditions for the Analysis of Carbamate and Organophosphate Pesticides

Condition 2 is suitable for the analysis of Carbamate and Organophosphate pesticides.

- More peaks of pesticides were detected.
- The analysis time was shorter.

Table 6: Condition 2 for GC-MS analysis.

Parameters	Condition 2
Injection Volume (μL)	1
Injection Temp. (°C)	250
Split Ratio	80:1
Split Flow	160 mL/min
Initial Temp. Oven	70 °C/hold 1 min
Carrier Gas Flow (He)	2 mL/min
Rate (°C/min)	15 1 15 30
Value (Ramp to) (°C)	175 190 250 300
Hold Time (min)	1 - - 3
Run Time	32.667

Table 7: Analysis of pesticides.

No.	Pesticides	RT
1	Propoxur	4.856
2	Dichlorvos	5.537
3	Diazinon	5.892
4	Methomyl	8.324
5	Fenobucarb	8.753
6	Carbaryl	13.096
7	Malathion	14.704
8	Ethion	25.347
9	Carbosulfan	27.991

2. Finding the Appropriate Solvent for Extracting Carbamate Pesticides from Contaminated Rice Samples

1. Methomyl; RT= 8.324 min.

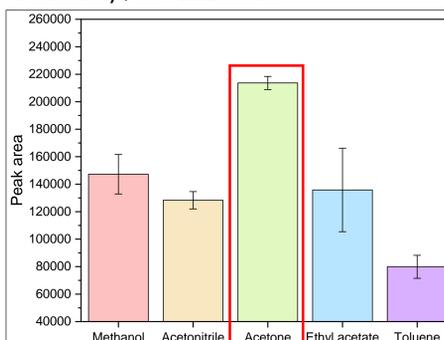


Fig. 4: Solvents used for extracting Methomyl from rice samples, tested in triplicate.

2. Fenobucarb; RT= 8.742 min.

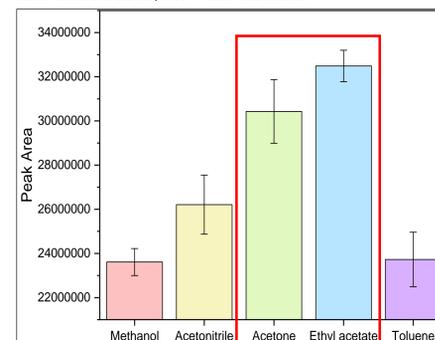


Fig. 5: Solvents used for extracting Fenobucarb from rice samples, tested in triplicate.

The study revealed that Methomyl had a retention time of 8.324 minutes, while Fenobucarb had a retention time of 8.742 minutes. For Methomyl extraction, acetone was found to be the most effective solvent, providing the highest peak area compared to other solvents. For Fenobucarb extraction, ethyl acetate and acetone showed similarly high extraction efficiency, with peak areas higher than those of other solvents. The peak area varied according to the concentrations of Methomyl and Fenobucarb, respectively.

Conclusion

The determination of optimal conditions for carbamate and organophosphate pesticides revealed that Condition 2 is the most suitable, as it allowed for the detection of more peaks and reduced the analysis time. Regarding the selection of appropriate solvents for the extraction of carbamate and organophosphate pesticides contaminated from cooked rice samples, it was found that acetone could effectively extract methomyl and fenobucarb, while ethyl acetate was effective for fenobucarb.

References

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