

Evolution GC-MS/MS

Transforming the best GC-MS system
into something even better:

Application Note Evo801

Triple Quadrupole MS: The Methode of Choice for Complex Samples



INTRODUCTION

Improper and/or illicit use of pesticides on agricultural produce can cause unacceptable high levels of their residues in produce intended for human consumption.

Worldwide there are over 800 compounds currently in use, including insecticides, fungicides, rodenticides, herbicides and growth regulators.

Food safety concerns are increasing among consumers worldwide.

Thus all food produce in or imported into Germany must contain less than the statutory maximum residue limit (MRL) of any given residue, requiring regular monitoring of foodstuff.

Mass spectrometry as a highly selective analytical technique can be used to monitor the masses of specific ions generated by the analytes of interest. This technique is also known as target analysis.

By using the Selected Ion Monitoring technique a greater level of selectivity is achieved compared to other traditional GC detection methods, e.g. using the flame ionization detector (FID) or the electron capture detector (ECD). But due to the low selectivity of the clean-up methods applied to a broad variety of produce samples for multiresidue pesticide analysis SIM does not eliminate the possibility of interfering matrix effects.

The MS/MS technique, also known as Multiple Reaction Monitoring (MRM) allows for monitoring specific collision induced dissociation (CID) reactions. Since the CIS process itself is dependent on molecular structure as well as on mass, significant gain in analytical selectivity can be obtained using MRM.

This application note compares the results of SIM and MRM using the **CHROMTECH EVOLUTION MS/MS** triple quadrupole mass spectrometer for Tebuconazol, a triazole high-effective fungicide in a real sample (green cabbage) extract.

METHODS

Extraction Method

Sample preparation was performed according to both the “DFG Method S19”:

1. The sample was rapidly homogenized using a Ultra-Turrax blender
2. Extraction with acetone/water
3. Liquid/liquid partition with cyclohexane/ethylacetate
4. Gel permeation chromatography
5. Analysis of the extract by GC/MS (SIM) and GC/MS/MS (MRM)

GC Method

Agilent 7890N GC with CTC CombiPAL autosampler

Column: HP-5ms, 30m x 0.25 mm ID x .25 µm

Flow Rate: 1mL/min Constant Flow

Oven Program: 70 °C, Hold 2 min

280°C @ 25°C/min, Hold 5 min

Inlet: 250°C, 1min splitless, 1 µL injected

MS Method

CHROMTECH EVOLUTION GC/MS/MS triple quadrupole mass spectrometer

Ionization Mode: Electron ionization (EI)

Electron energy: 70eV

Ion source temp.: 230 °C

Quadrupole temp.: 150°C

SIM mode: 1 Group with 4 ions: m/z 250, 125, 83, 70

Scan Time: 0.2

Resolution (SIM width): Low (0.9)

MRM mode: 1 Group with 4 transitions:

m/z 125> m/z 89 @ -15V CE

m/z 250> m/z 125 @ -18V CE

m/z 250> m/z 139 @ -18V CE

m/z 250> m/z 70 @ -15V CE

Scan Time: 0.2

Resolution Q1.1.0 / Q3: 1.0

Collision gas pressure (Argon): 1 bar

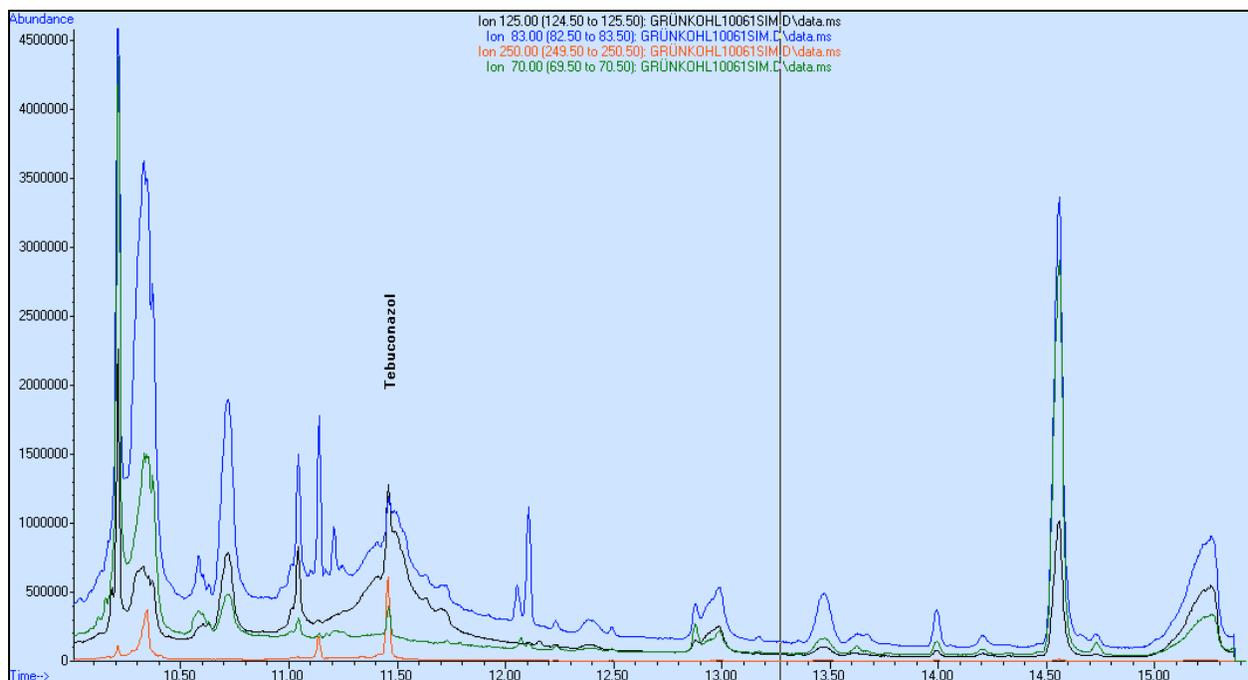


Figure 1: Green cabbage extract, target analyte Tebuconazol, 4 SIM traces (merged):
m/z 250 ,m/z 125, m/z 83 ,m/z 70

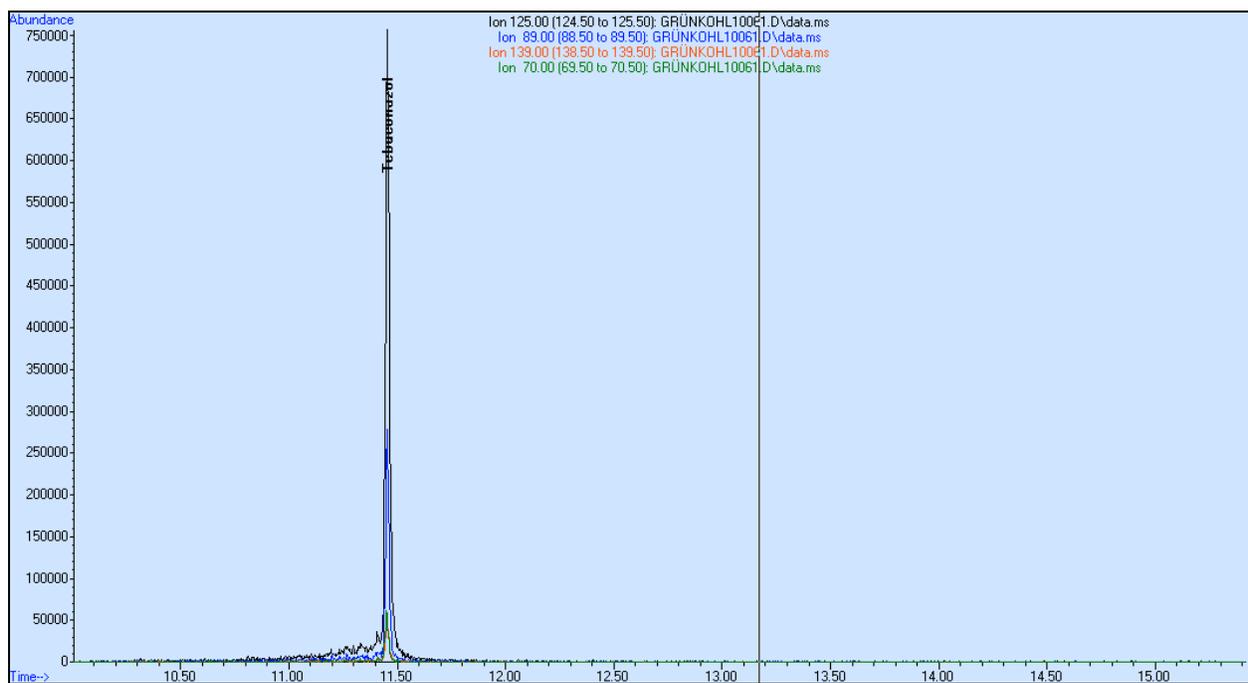


Figure 2: Green cabbage extract, target analyte Tebuconazol, 4 MRM traces (merged):
m/z 250>125@-18V, m/z 125>89@-15V, m/z 250>139@-18V ,m/z 250>70@-15V

CONCLUSIONS

Selected ion monitoring (SIM) is a useful and selective method for the screening and confirmative analysis of pesticide residues in relatively simple matrices.

MRM is the method of choice when it comes to the determination of low limit concentration of pesticide residues in complex (“heavy”) matrix since it offers significant improvements in the signal/noise ratio due to the matrix elimination effect of the MRM.

The described SIM and MRM method was performed on a “real” sample where underlying pesticide levels were confirmed above the reporting level.

