Using RTL and 3-Way Splitter to Identify Unknown in Strawberry Extract

Application Brief

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Fruit and vegetable extracts are usually very complex to analyze. It is common to use the very selective GC detectors, for example NPD, µECD, and FPD, to look for trace pesticide residues in the extracts. Mass spectrometry is most often used to confirm the hits from GC detectors. A previous application note [1] describes a GC/MS system with a three-way splitter added to the end of the column. The column effluent could be split three ways to two GC detectors and the MSD. The splitter system is therefore capable of providing up to four signals (two GC signals, SIM, and full-scan chromatograms) from a single injection. The combination of element selective detectors, SIM/scan, and Deconvolution Reporting Software (DRS) makes a very powerful pesticide analysis system [2]. The trade-off is the decrease of analyte concentration in any detector due to the flow splitting at the end of the column.

The system used for this study consists of an Agilent 7890A GC with split/splitless inlet, a three-way splitter, µECD, dual flame photometric detector (DFPD), and 5975C MSD. Figure 1 shows chromatograms from 2 separate injections (each injection provides two GC signals) of the same strawberry extract without any hardware or filter changes. All of the target compounds were found and confirmed by DRS, GC, and MS signals except the unknown peak at about 41 minutes. The peak shows responses from µECD, DFPD(S) and DFPD(P). However, no peak was observed in the MS full-scan signal. This makes it difficult to confirm the unknown peak using the full-scan TIC.

Since the analysis was retention time locked, it is therefore possible to find potential matches by examining the RTL pesticide database (part number G1672AA). The unknown compound, containing electron-capturing atoms (for example, Cl or O), P, and S atoms, would have a target retention time inside the

Highlights

Splitter—an inert, easy-to-use capillary flow technology that splits column effluent to multiple detectors (for example, MSD, DFPD, and µECD). The splitter configuration provides a comprehensive screening and quantitative system.

By combing RTL, element-selective detector chromatograms, and the RTL pesticide database, a trace level pesticide residue was identified without the full-scan mass spectrum.

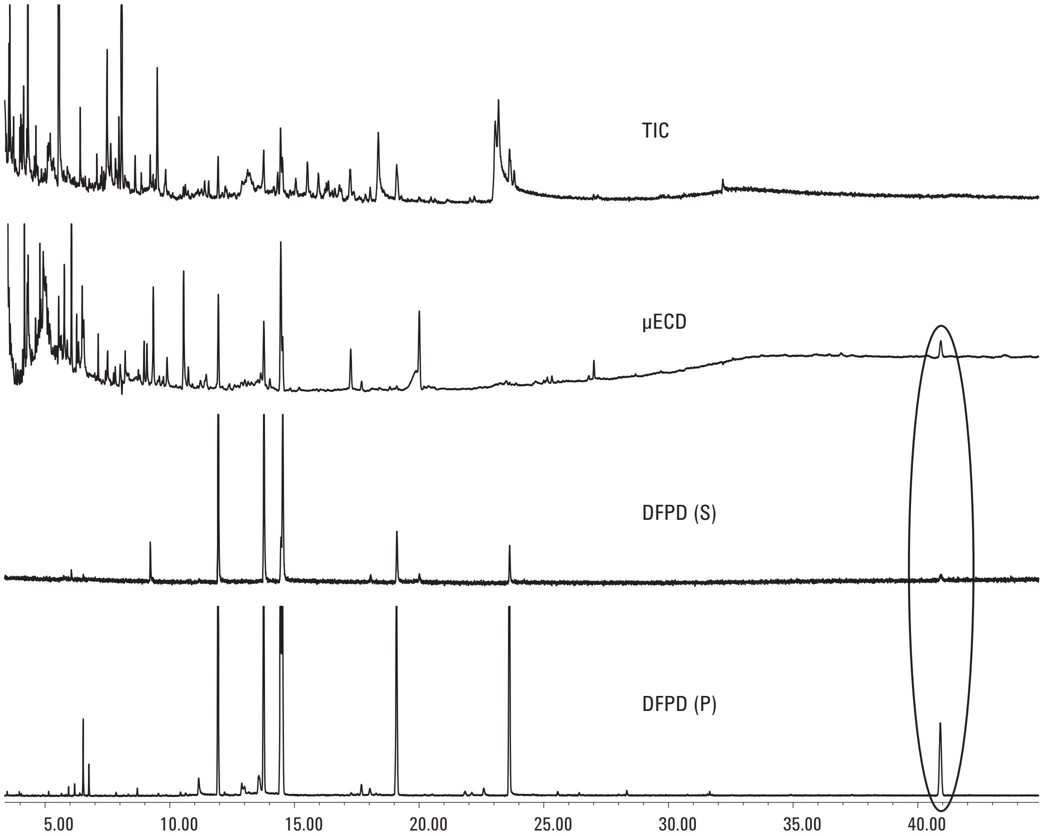

Figure 1. Unknown compound detected by GC signals not found in strawberry extract TIC.

Table 1. Compound List Extracted from the RTLPest3.tab File

Name	CAS	Mol form	Mol wt	R.T.	Target Ion	Q1	Q2	Q3
Fluthiacet-methyl	117337196	$C_{15}H_{15}ClFN_3O_3S_2$	403.9	39.10	403	56	405	232
Benzo[g,h,i]perylene	191242	$C_{22}H_{12}$	276.3	39.13	276	277	138	275
Temephos	3383968	$C_{16}H_{20}O_6P_2S_3$	466.5	40.74	466	125	93	109
PBB 169 hexabrombiphenyl	60044260	$C_{12}H_4Br_6$	627.6	40.93	308	468	148	154
Rotenone	83794	$C_{23}H_{22}O_6$	394.4	41.70	192	191	394	177

41 ± 0.5-minute window (that is, 40.5 to 41.5 min) in the database, if it is included in the database. Table 1 is a portion of the RTLPest3.tab file[1] opened in Microsoft Excel. The compound temephos at locked retention time 40.74 min meets all the criteria for the unknown peak. To further confirm peak identity, extracted ion chromatograms (EICs) of the four major ions of temephos were plotted. Figure 2 shows EICs of target ion and three qualifiers (ions 466, 125, 93, and 109 from Table 1) of temephos. Although the ion intensities were weak, the noticeable presence of all four ions at 40.9 min helped to confirm that the unknown peak was temephos.

[1] The RTLPest3.tab file is created in the C:\Database directory while executing the Tools\List Screen Database... command (in MSD Enhanced Data Analysis software) and selecting the RTLPest3.scd from the C:\Database directory.

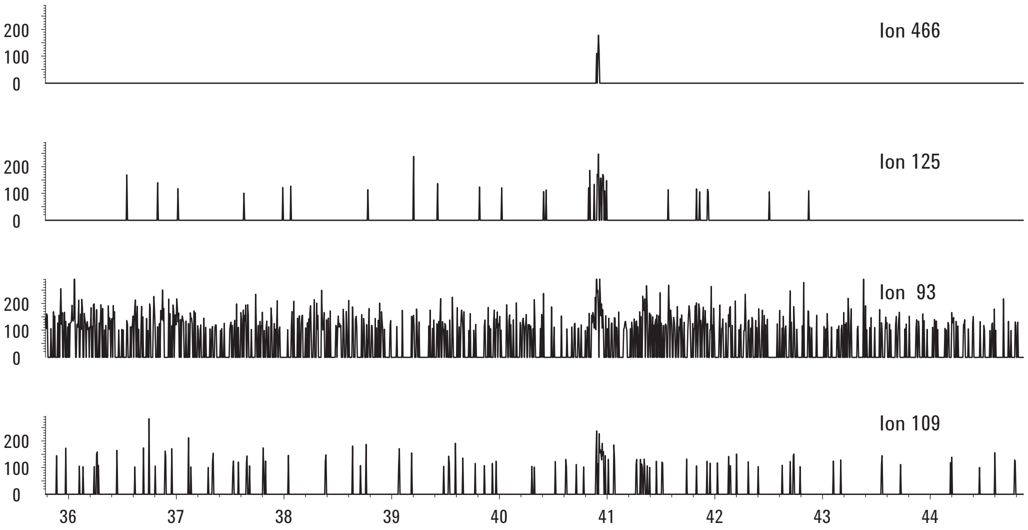

Figure 2. EICs of target ion 466 (temephos) and three qualifier ions.

References

1. Chin-Kai Meng and Bruce Quimby, "Identifying Pesticides with Full Scan, SIM, µECD, and FPD from a Single Injection," Application Note, 5989-3299, July 2005.
2. Mike Szelewski and Bruce Quimby, "New Tools for Rapid Pesticide Analysis in High Matrix Samples," Application Note, 5989-1716, October 2004.

Acknowledgement

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