

Finding Pesticides using an Agilent 7200 GC/Q-TOF and Unknowns Analysis Software

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Introduction

With increased international trade in food and food ingredients, there is even more emphasis on food safety. GC with triple quadrupole mass spectrometry (GC/QQQ) is the gold standard for the target analysis of volatile contaminants. But, if a residue is present that is not on the target list, it will be missed. This presentation describes a new tool, GC with a quadrupole time of flight (GC/Q-TOF) mass spectrometer, for the retrospective analysis of unknowns (Figure 1). The TOF provides high resolution and accurate mass measurements.

Instrumentation and Software

| | |
|------------------------|--|
| GC | Agilent 7890A |
| Mass Spectrometer | Agilent 7200 Q-TOF in TOF mode |
| Autosampler | Agilent 7693A |
| GC Column | Agilent 15m X 0.25mm X 0.25um HP-5MS UI |
| Oven Temp Program | 70°C (1 min), 50°C/min to 150°C (0 min), 6°C/min to 200°C (0 min), 16°C/min to 280°C (5 min) |
| Retention Time Locking | Chlorpyrifos-methyl locked to 8.298 min (constant pressure) |
| Software | MassHunter Acquisition, Qualitative Analysis, Quantitative Analysis, and Unknowns Analysis |

- Accurate mass
- High resolution
- MS/MS capability
- Removable ion source
- High dynamic range
- Internal reference mass
- Fast scanning



Figure 1. Agilent 7200 GC/Q-TOF

Unknowns Analysis

1. Analyze sample by GC/TOF
2. Run quant method first or go directly to Unknowns Anal.
3. Set up method parameters for deconvolution, library searching and hit matching
4. Analyze samples
 1. Deconvolute chromatogram(s)
 2. Library search all components
 1. Use Agilent RT-Locked Pesticide and Endocrine Disruptor Library
 2. Match library retention times to filter out false positives
 3. Report identified compounds
 4. Display results as shown in Figure 2
5. Review Unknowns Analysis results
6. Confirm in MassHunter Qual by extracting out accurate masses of identified compounds.

Identifying Triclosan in an Okra "Blank"

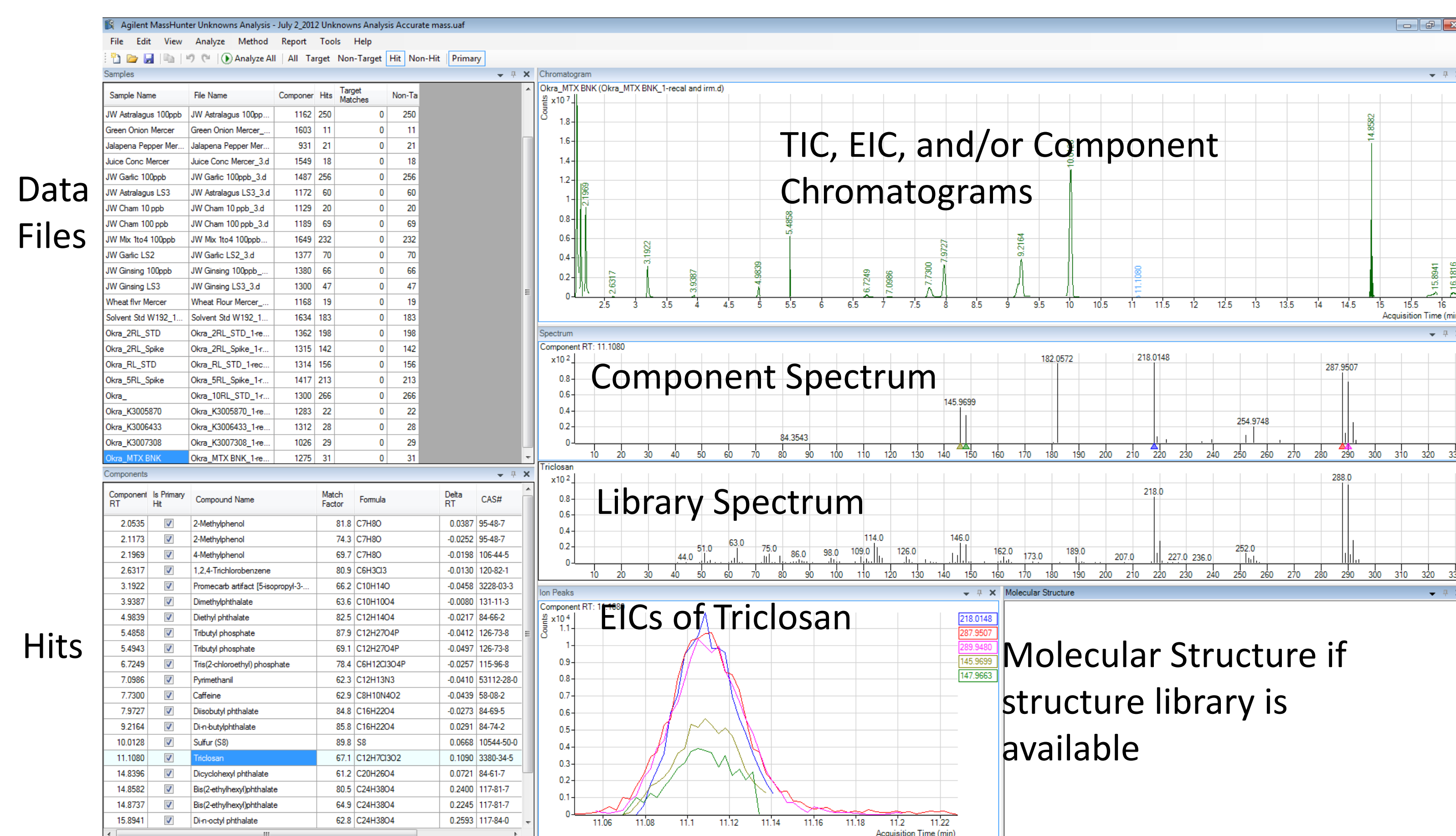


Figure 2. One of the hits is Triclosan, a disinfectant used in soaps, toothpaste, deoderants, and many other consumer products. Residues are widespread in the environment.

Figure 3. Use MassHunter's Accurate Mass Calculator to find actual mono-isotopic accurate mass

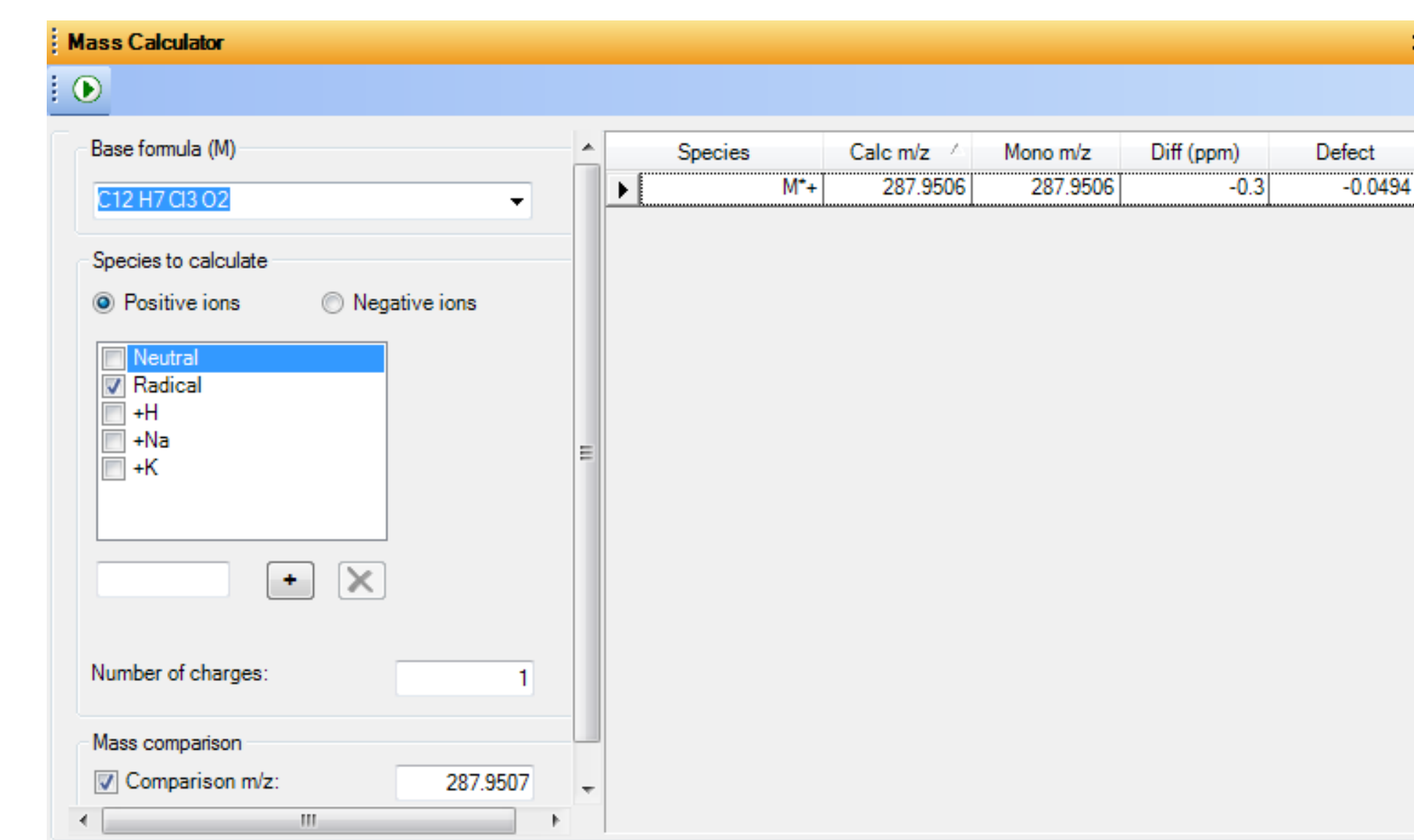
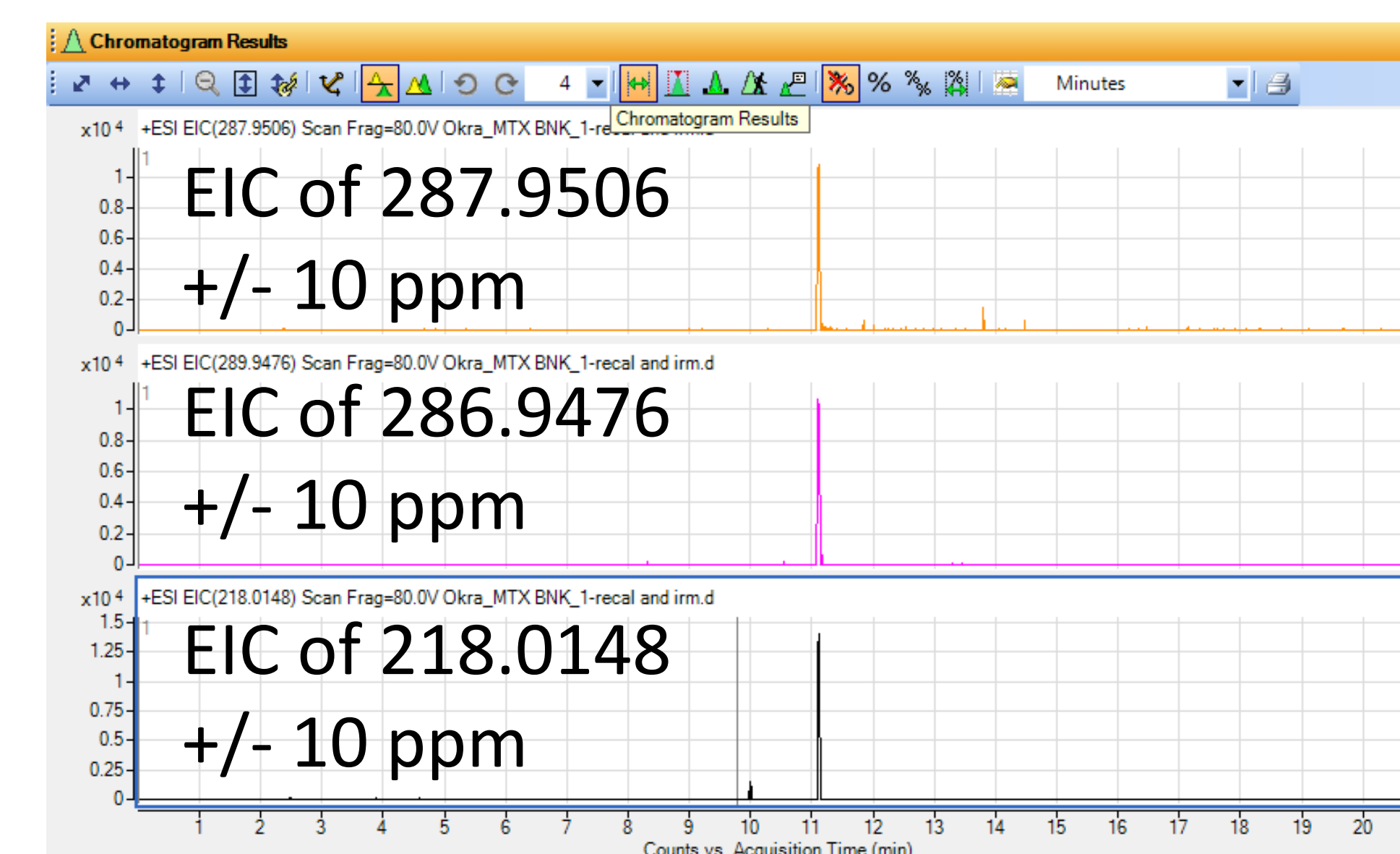


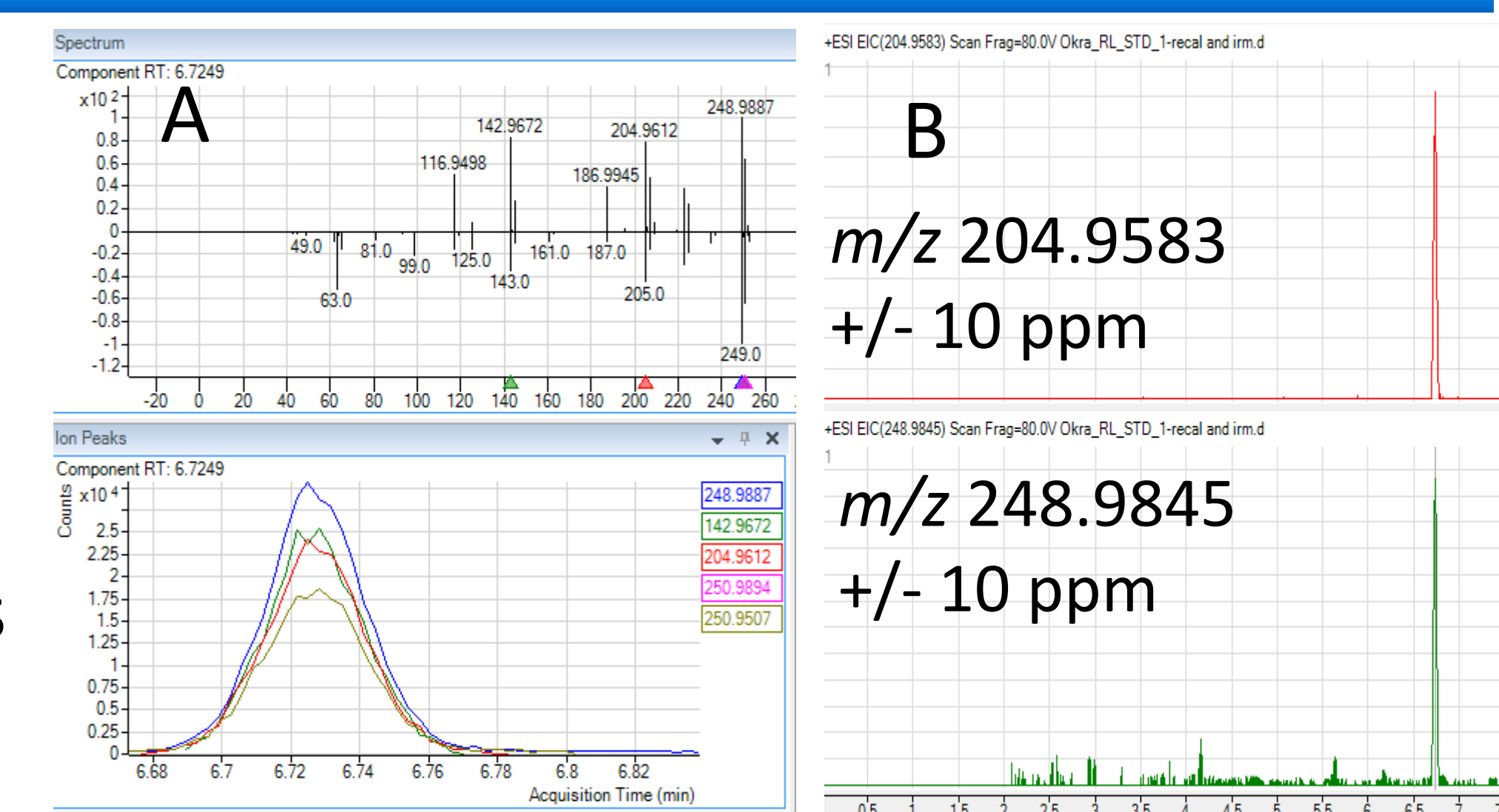
Figure 4. EICs for the mono-isotopic molecular ion and the molecular ion with one ³⁷Cl. m/z 218.0148 was the measured value.



Identifying a Flame Retardant in an Okra Blank

Tris(2-chloroethyl) phosphate (TCEP) was identified by the Unknowns Analysis software in the same Okra "blank." TCEP is carcinogenic and has shown broad toxicity in animal models.

Figure 5. A) Comparison of the spectrum & EICs for the deconvoluted component identified as TCEP. B) Two accurate mass EICs for TCEP



Conclusions

1. MassHunter Unknowns Analysis software is a simple, but powerful tool to identify unknowns, i.e., compounds not on the target list but in a mass spectral library.
2. Accurate mass offers high selectivity and the ability to calculate formulas for the molecular ion and fragments.
3. MS/MS can aid in true unknown characterization.