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Analysis of 446 Pesticides and 1202 Transitions in a Single Run on the Thermo Scientific TSQ Quantum XLS GC-MS/MS

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Key Words

- TSQ Quantum XLS GC-MS/MS
- Food Safety
- MRM
- Pesticide
- Triple Quadrupole

Introduction

As more compounds are being added to governmental monitoring lists, screening methods for large numbers of compounds are becoming important to laboratories analyzing foods for pesticides. An instrument method was developed on the Thermo Scientific TSQ Quantum XLS triple quadrupole mass spectrometer to analyze 446 pesticides using 1202 transitions. A peak width of 0.7 Da was used to provide the selectivity needed to analyze this number of compounds in a 35 minute run. The use of the software's EZ method feature allows the user to import all of the compound transition information from an Excel® spreadsheet.

Experimental Conditions

Injection and separation took place using a Thermo Scientific TriPlus liquid autosampler and a Thermo Scientific TRACE GC Ultra gas chromatograph with a programmable temperature vaporizing (PTV) inlet. Chromatographic separation was achieved with a Thermo Scientific TRACE TR-Pesticide II 30 m × 0.25 mm × 0.25 µm column with a 5 m guard column. Standards were prepared in hexane and injected onto the column utilizing the programmable temperature capabilities of the PTV inlet.

All gas chromatograph (GC) and mass spectrometer (MS) parameters, including selective reaction monitoring (SRM) transitions, were taken from the Thermo Scientific Pesticide Analyzer Reference.[1]

Injection

Injection was performed on the PTV inlet utilizing a inert baffled liner. The initial injection temperature was 75 °C and was ramped to 300 °C at a rate of 2.5 °C/second, held for 3 minutes then increased to 330 °C at 14.5 °C/second and held for 20 minutes. Holding the final temperature of the inlet at this elevated temperature prevents carryover. The inlet parameters are shown in Figure 1.

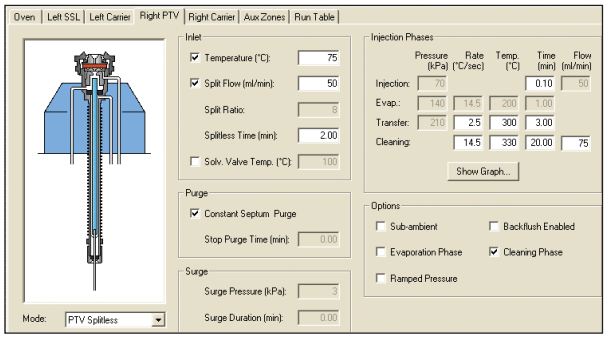


Figure 1: GC PTV inlet parameters

Separation

Chromatographic separation was achieved by using the TR-Pesticide II, a 5 % diphenyl/95 % dimethyl polysiloxane column (0.25 mm × 30 meter, and a film thickness of 0.25 µm with a 5 m guard column). The guard column was inserted through the transfer line and into the source of the MS. Placing the guard column through the transfer line reduces the column bleed caused by the constant high temperature at the end of a normal column. The oven was programmed as follows: Initial Temp: 90 °C, 5.0 min., 25 °C/min. to 180 °C, 0.0 min, 5 °C/min. to 280 °C, 0.0 min., 10 °C/min. to 300 °C with a final hold time of 5 min. and a constant column flow rate of 1.2 mL/min. The entire set of oven parameters is listed in Figure 2.

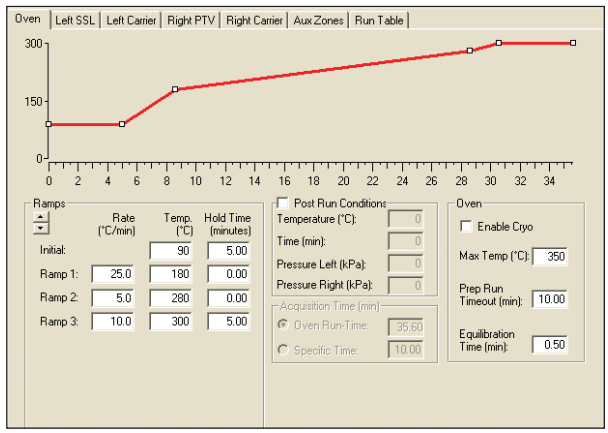


Figure 2: GC oven parameters

Detection

Detection of the pesticides was performed using the TSQ Quantum XLS™ GC-MS/MS. After retention times were determined in full scan, a timed-SRM method was constructed to analyze the compounds in one single mix. An external calibration curve was constructed from 20 to 2000 pg with all compounds in each calibration standard mix. One quantitation ion and one or two confirming ions were used for each compound for a total of 1202 transitions in 35 minutes. The extra confirming ion allows for flexibility when analyzing matrix-matched calibration standards. A partial list of MS parameters can be seen in Figure 3. After the external calibration, standards of 1, 5, and 10 pg were injected ten times to determine at what level each compound could be detected, confirmed, and to calculate the %RSDs at these levels. Table 1 shows a partial list of %RSDs at 10 pg. To achieve these same levels of reproducibility and confirming ions for lower concentrations a larger injection volume would need to be used.

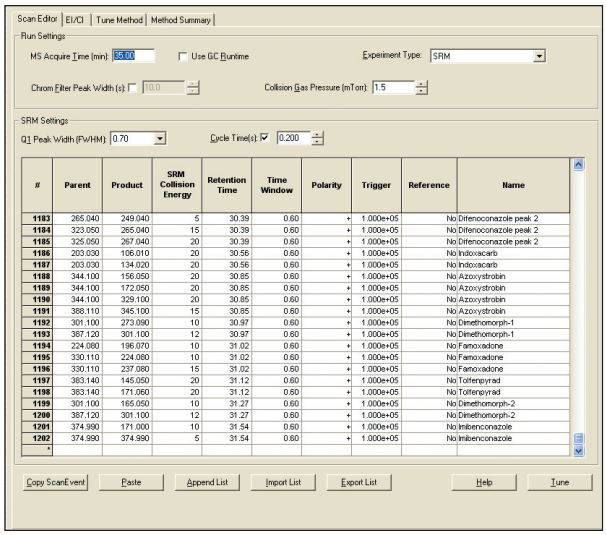


Figure 3: Partial list of timed SRM parameters

1,2,3-trichlorobenzene %RSD at 1 pg was 7.41%. Figure 4 shows the quantitation ion, confirming ion, and ion overlay for 1,2,3-trichlorobenzene at 1 pg.

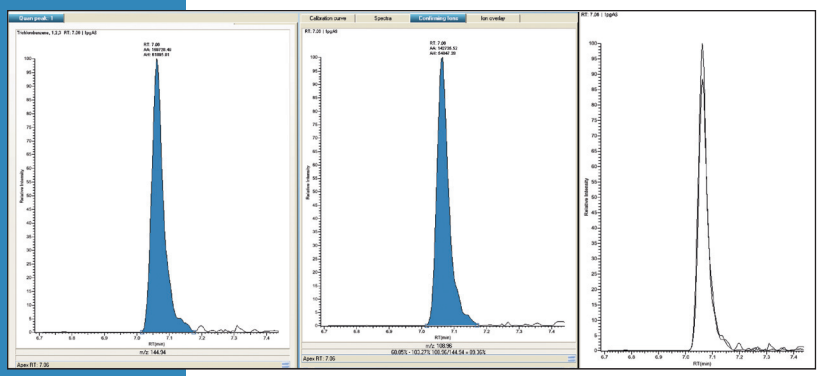


Figure 4: 1,2,3-trichlorobenzene quantitation and confirming ion at 1 pg

Results and Discussion

Linearity

The calibration curve ranged from 20 to 2000 pg for all compounds. The linearity for most of the 446 compounds was $r^2 > 0.995$. Only 42 compounds had r^2 values less than 0.995 and none of these were under 0.990. Curves were generated using Thermo Scientific QuanLab Forms (Figures 5 and 6). Figure 7 shows the number of scans across the peak for a number of compounds at 1 pg.

Compound	%RSDs at 10 pg
1,2,3-trichlorobenzene	3.50
1,2,4-trichlorobenzene	4.68
1,2,5-trichlorobenzene	5.21
Hexachlorobutadiene (HCBD)	5.50
1,2,3,4-tetrachlorobenzene	4.24
Dichlobenil	4.14
1,2,3,5-tetrachlorobenzene	5.74
Biphenyl	6.65
1,3,5-tribromobenzene	4.99
PCB – monochlorbiphenyl	4.97
Molinate	4.71
PCB – dichlorobiphenyl	6.90
Fenclorim	7.77
Hexachlorobenzene (HCB)	7.94
Propyzamide	6.31
Mirex	7.44
PCB – nonachlorobiphenyl	6.47
PCB 209 – decachlorobiphenyl	6.51

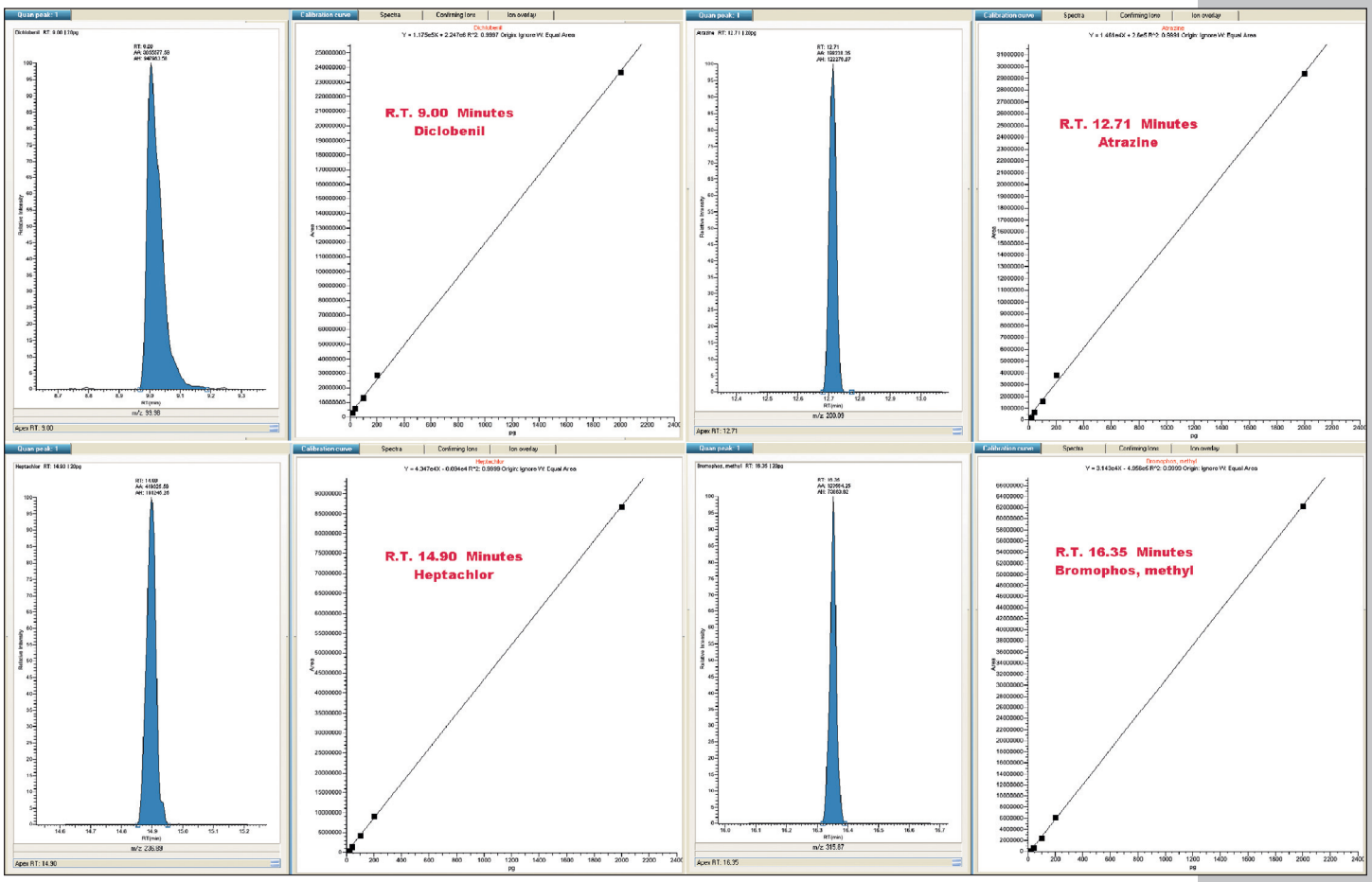
Table 1: The %RSDs for 18 of the 446 compounds that were injected in a single analysis 10 times

Conclusions

The TSQ Quantum XLS GC-MS/MS paired with the TRACE GC Ultra™ gas chromatograph showed excellent results for the 446 pesticides. Calibration curves for most of the pesticides studied met a linear least squares calibration with a correlation coefficient of $r^2 > 0.995$. One or two ion ratios were used to confirm each pesticide. Good repeatability was demonstrated by the single digit %RSDs for a number of compounds at 10 pg. The TSQ Quantum XLS GC-MS/MS is capable of screening for large numbers of pesticides in a single analytical run.

References

1. *Pesticide Analyzer Reference*, Thermo Fisher Scientific, Austin 2009



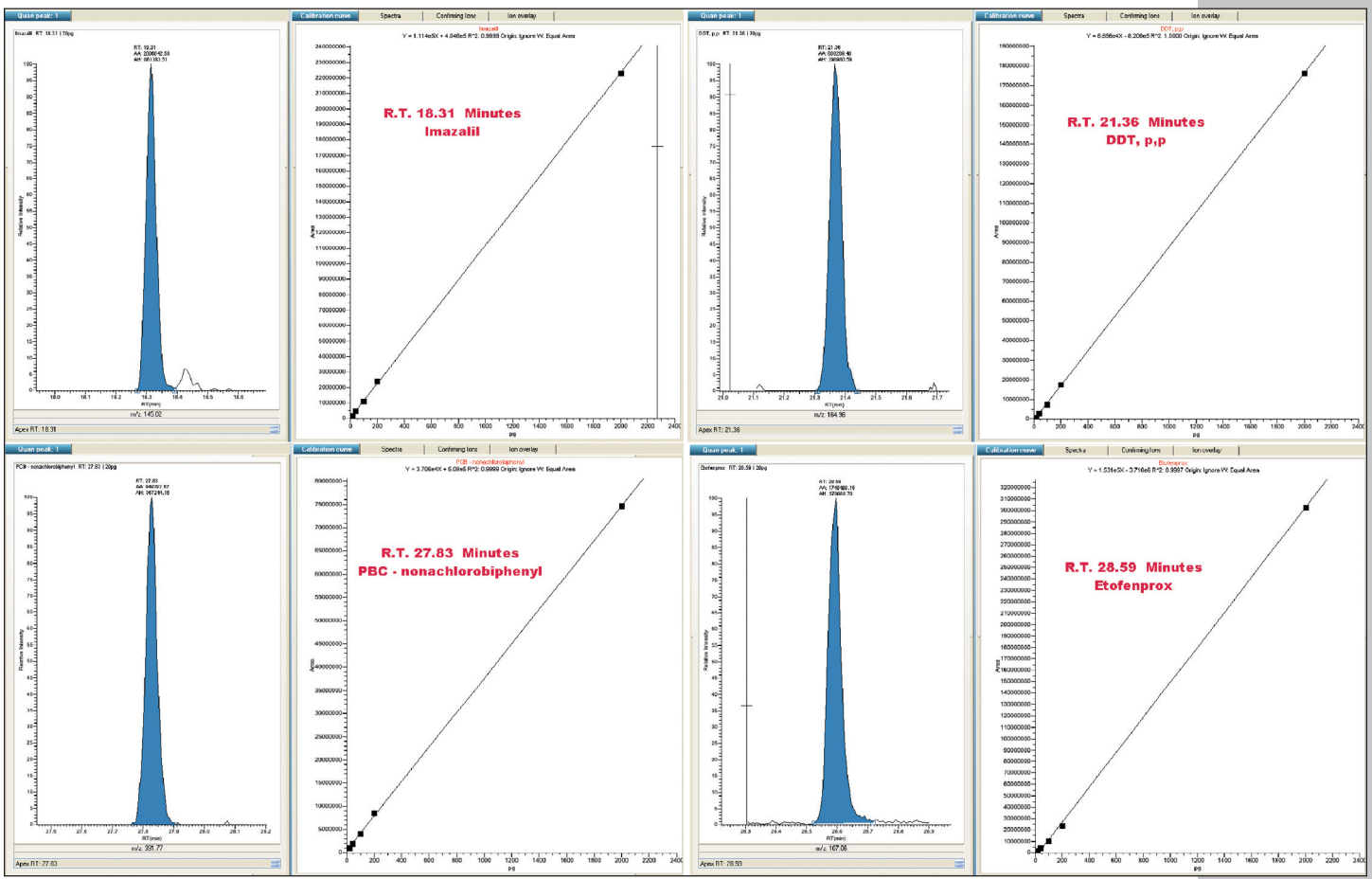


Figure 5 and 6: Calibration curves and peak shape at 20 pg

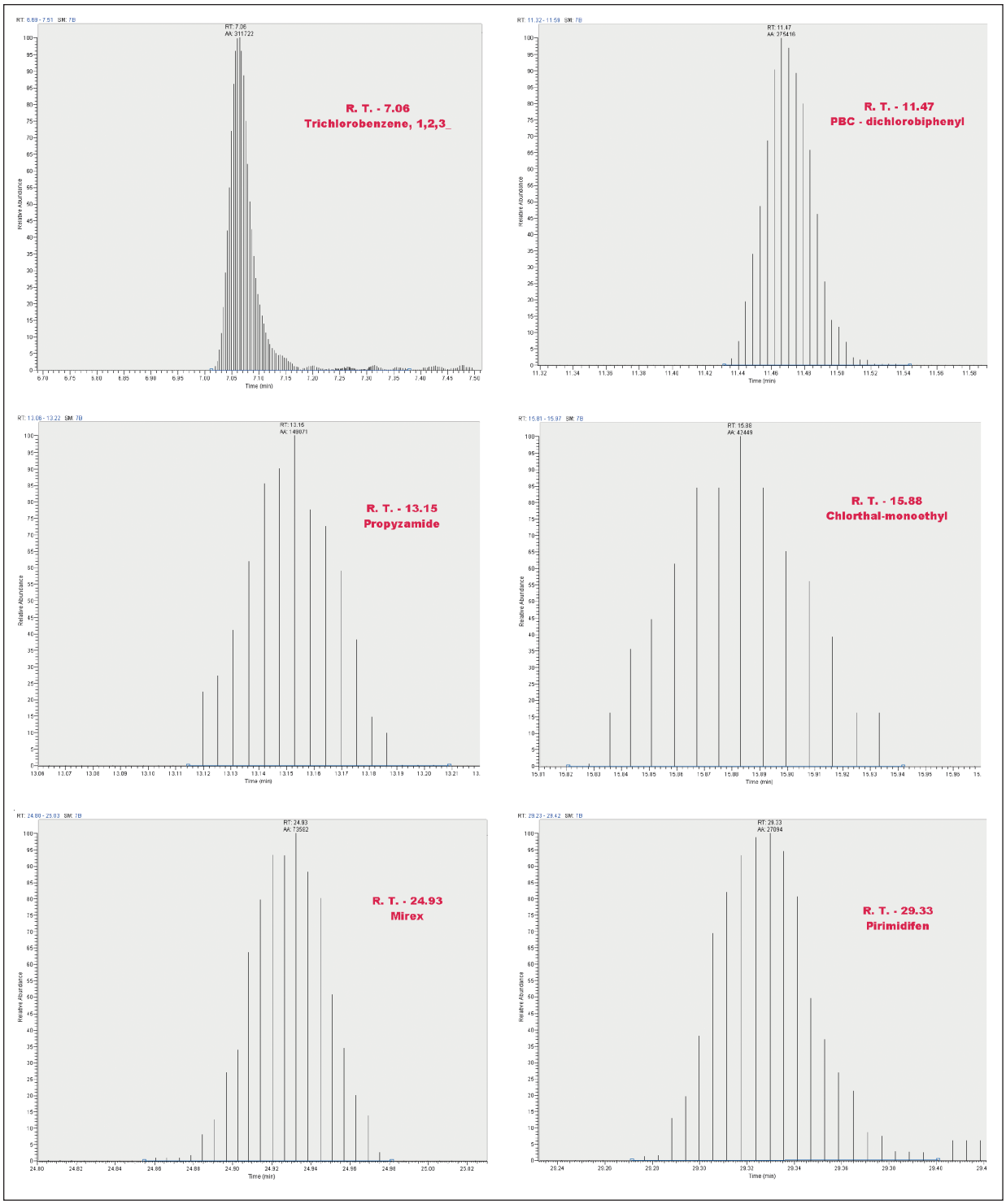


Figure 7: Large number of scans across the peak at 1 pg throughout the chromatographic run

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