

# Analysis of Pesticides by GC-Triple Quad Made Easy with Novel Software and New MS Ion Source Design

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## Overview

Pesticide residue analysis is extremely challenging due to the complexity of both the analyte list and the matrices in which those analytes are monitored. Also, current regulation requires that many of these analytes be monitored at low ppb concentrations, requiring highly optimized methodology. Added to these challenges are the complexities of developing the MS/MS parameters needed when new pesticides require monitoring.

Because GC-triple quadrupole technology is being widely adopted for this analysis, new instruments must be designed to overcome these challenges. Described below are software and hardware design elements of a new GC-triple quadrupole, the Thermo Scientific TSQ 8000 GC-MS/MS, that facilitate the analyst ability to overcome these difficulties, and in doing so, fully optimize their methodology to ensure they achieve the performance necessary for low level detection.

## Software Design

### Software Overview

There are three pieces of software on the TSO™ 8000 that enable the user to create and maintain a high performance multi-pesticide residue analysis. These software components are shown in Figure 1:

- **AutoSRM** – Guides the user through the complete SRM development process, automating many of the tedious steps
- **TSQ 8000 Instrument Method** – Contains many features which facilitate very complex SRM methods, typical of pesticide analysis
- **Thermo Scientific TraceFinder Software**– Batch acquisition, data review, and reporting software integrated with the TSO 8000 instrument method

The next sections will discuss how each of these pieces are integrated together to give a total solution to pesticide method development, optimization, and analysis.

### Starting Point

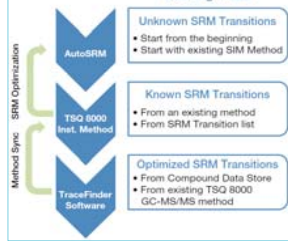


FIGURE 1. Workflow diagram of the three software components of the TSO 8000 GC-MS/MS, indicating their interconnectivity and possible user starting points within that workflow.

### AutoSRM

Figures 2, 3 and 4 show the workflow for developing SRM transitions through AutoSRM. This is done in a three step process, which is guided by the software.

#### Step 1 – Precursor Ion Study

The user begins by naming the compounds whose SRM transitions are to be developed. AutoSRM acquires a full scan for each solution containing these compounds and presents the user with the resulting chromatogram. The user specifies the retention time by clicking on the peak for each compound, aided by the spectra and the ability to display an extracted ion chromatogram for a representative ion for each compound. Once peaks are identified, AutoSRM displays a table of the most intense ions from which the user can choose precursor ions. The top precursor ions can also be automatically selected by AutoSRM.



FIGURE 2. Precursor Ion Study

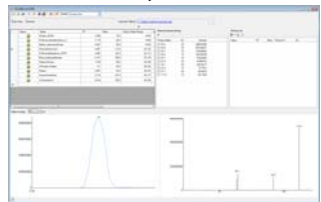


FIGURE 3. Product Ion Study



FIGURE 4. SRM Optimization Study

#### Step 2 – Product Ion Study

In this step, AutoSRM acquires three product ion scans at different collision energies for each of the selected precursor ions. The user then is presented with a table of the top product ions for each precursor ion from which the user can select. If desired, AutoSRM can select the top product ions for the user.

#### Step 3 – SRM Optimization Study

The final step of AutoSRM is to optimize each chosen transition. AutoSRM will run SRMs at 10 different collision energies, and the user is presented with an intensity vs. collision energy curve. The top collision energy for each transition will then be chosen by AutoSRM, completing each transition. The user can either optimize the collision energy over a range from 5-50 eV, in 5 eV steps, or over a range targeted around the best collision energy found in the Product Ion Study, in 2 eV steps. Once AutoSRM optimizes all of the selected transitions, the SRM list can be exported to a TSO 8000 instrument method



FIGURE 5. Schematic representation of how Timed-SRM works. Each compound's acquisition windows is centered around the retention time of the analyte, insuring no transitions elute close to segment breaks.

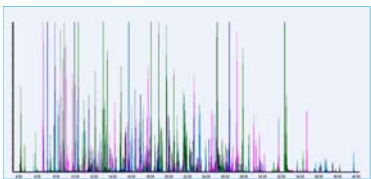


FIGURE 6. Chromatogram of 330 pesticides, each with two transitions each, analyzed in a single run. The method was originally run in multiple injections, but was consolidated to one with Timed-SRM.

### TSQ 8000 Instrument Method

The TSO 8000 instrument method features retention time based SRM (timed-SRM) acquisition. The user needs only to enter the transition parameters and the retention times for each SRM, and the instrument method will set the acquisition windows for each transition so they are centered around when the compound elutes (Figure 5). This ensures that no transitions fall close to a segment break, so there is no need to worry about a compound shifting outside a retention time window. Also, because each acquisition window is centered around the SRM, and the windows are allowed to overlap, the system will waste less time acquiring transitions away from when compounds elute, allowing for higher overall dwell times and increasing instrument sensitivity. This allows the setup of acquisition methods with hundreds of pesticides in a single run, allowing users to combine several methods into one (Figure 6).

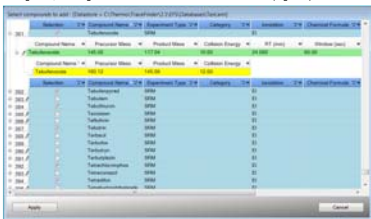


FIGURE 7. Compound selection within the TraceFinder™ software Compound Data Store. To create a method, the user simply selects the compounds to be monitored. If the user then links the TraceFinder method to the instrument method, the compounds selected will be those monitored during acquisition.

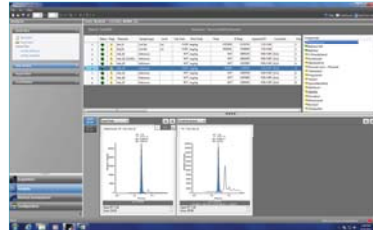


FIGURE 8. The TraceFinder Data Review window.

### TraceFinder Software

From the TSO 8000 instrument method, the acquisition list can be easily exported to the TraceFinder Compound Data Store (CDS) (Figure 7). The CDS is a database of compounds and their acquisition parameters. The user can create a TraceFinder processing method from the CDS by selecting only those compounds they are interested in monitoring.

Processing methods in TraceFinder software can be linked directly to the acquisition method. This means that while in data review, the user can update a retention time of a compound based on when that compound elutes. This will then update this retention time in the timed-SRM instrument method, ensuring that the acquisition window for that compound is centered around the peak.

Another advantage of linking the instrument method with the TraceFinder processing method, is that when the user creates the TraceFinder method from the compound data store, only the compounds the user selects from the database will be acquired and processed. With provided pesticide SRM parameters in the CDS, this greatly simplifies the process of creating a complex pesticide method.

## Source Design

The above sections describe the software features on the TSO 8000 that enable easy pesticide method creation and analysis. Another aspect of the instrument design that facilitates detection of pesticides in complex matrices is its Thermo Scientific ExtractaBrite ion source (Figure 9).

Typical matrices that require pesticide analysis are food, beverage and waste. The complexity of these matrices can all be very taxing on a source if it is not designed to handle the high volume of matrix material being passed into it. To account for this, the ExtractaBrite source on the TSO 8000 has three features designed to overcome the negative effects of matrix:

- **Dual source heaters** – Two heater blocks heat the ion volume and lens stack separately, providing a hot surface throughout the source, reducing matrix condensation on the ion volume and lenses
- **RF Lens** – The first source of RF that the ions see is one place where ion burn will begin to form. The ExtractaBrite ion source features a patented RF lens at the end of the source. This first RF stage is directly heated to keep it clean and protects the ion guide and analytical quadrupoles from contamination.
- **Repeller** – The repeller on the ExtractaBrite ion source is designed to overcome the effects of ion burn when it eventually does form.



FIGURE 9. The ExtractaBrite™ ion source of the TSO 8000

Due to the complexities of the typical matrices laboratories analyze pesticides in, it is likely an instrument that will eventually require source maintenance, however robust it is. To make this process as fast and easy as possible, the ExtractaBrite ion source is designed to be easily removed, without venting.

This is done with the vacuum interlock interfaces (Figures 10 and 11). The user simply presses a button on the front of the instrument to evacuate the inner ball volume, which then gives the ability to open the valve and insert the source removal tool into the manifold. The tool then latches onto the source, facilitating its removal. This process is accomplished in less than a minute, and reinserting the source follows a similar process. Since vacuum is never broken on the system, downtime due to source maintenance can be reduced from a day to an hour.



FIGURE 10. Rendering of Source Exchange Tool inserted into TSO 8000.



FIGURE 11. Cross section of the front of the analyzer tray, showing how the source is inserted into the source block.

Several software design elements of the TSO 8000 GC-MS/MS that overcome many of the analytical challenges of multi-pesticide residue analysis were described. These include: 1.) AutoSRM, which assists the user in creation of SRM transitions for new compounds, automating many of the tedious steps in the process, 2.) Retention time based SRM instrument method, allowing the user to build both processing methods and instrument methods by simply selecting the compounds they are interested in monitoring and allowing the update of acquisition windows based on retention time updating in data review.

Also described was how the TSO 8000 source was designed to enable analysis in complex matrices, typical of pesticide analysis. Through several design elements, including the dual source heaters, the RF lens and the repeller, high resistance to the negative effects of heavy matrix introduction into the source is achieved. Also, if source maintenance is eventually required, all parts which need periodic source maintenance can be removed through the unique vacuum interlock system without venting the system, removing a major source of downtime for pesticide analysis in complex matrices.

## Conclusion

Several software design elements of the TSO 8000 GC-MS/MS that overcome many of the analytical challenges of multi-pesticide residue analysis were described. These include: 1.) AutoSRM, which assists the user in creation of SRM transitions for new compounds, automating many of the tedious steps in the process, 2.) Retention time based SRM instrument method, allowing the user to build both processing methods and instrument methods by simply selecting the compounds they are interested in monitoring and allowing the update of acquisition windows based on retention time updating in data review.