

Using Triple Quadrupole GC-MS in Full Scan, SIM, SRM, and Mixed Scan Modes to Provide the Highest Coverage for Target and Non-target Analysis of Contaminants

David Steiniger¹, Dwain Cardona¹, Cristian Cojocariu², Paul Silcock², Alexander Semyonov¹, Jason Cole¹

¹Thermo Fisher Scientific, Austin, TX, ²Thermo Fisher Scientific, Runcorn, UK

Overview

This work describes how triple quadrupole gas chromatography-mass spectrometry (GC-MS) systems can be used in single quadrupole modes to satisfy current regulations as well as be able to easily switch to powerful SRM methods when needed, especially in preparation for future updates to regulations. Also included are some potential applications of mixed MS modes, such as simultaneous FS/SRM.

Introduction

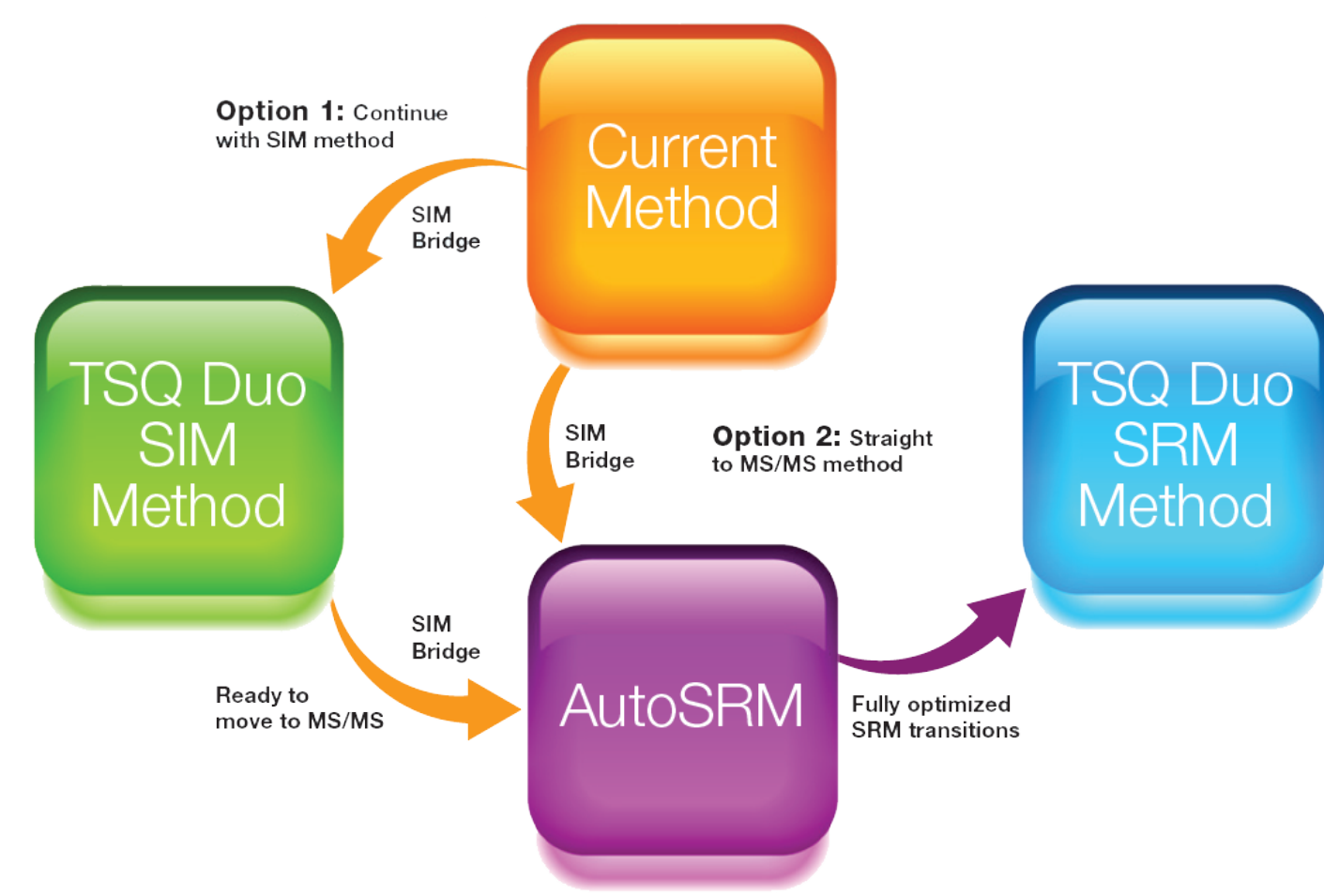
Many laboratories are adopting triple quadrupole GC-MS systems to access the widely accepted advantages brought by high-selectivity detection of target analytes. However, while the advantages of MS/MS scan modes, particularly SRM, are well documented, some regulated methods such as EPA 8270 do not permit use of triple quadrupole GC-MS systems. Laboratories that are accredited for these “prescriptive” regulated methods are then compelled to continue with current technology such as single quadrupole GC-MS instruments. These regulated methods prevent users becoming familiar with the latest technology so they are not able to use triple quadrupole technology unregulated sample analysis or special projects.

The versatility of the Thermo Scientific™ GC-MS/MS systems will be demonstrated in the various operational modes offered. Methods were developed for semi-volatile organic compounds (SVOCs) and pesticides, and data is presented in full scan, SIM, SRM, and SRM/full scan modes. Figure 1 provides an overview of triple quadrupole method development.

The Thermo Scientific™ TSQ™ Duo GC-MS/MS coupled to a Thermo Scientific™ TRACE™ 1310 GC, enables truly simple operation whether you choose to use it in single or triple quadrupole mode. It also bridges the gap between single and triple quadrupole mode use within the same instrument to allow easy adoption of powerful MS/MS workflows when required.

Using the instrument in single quadrupole GC-MS mode allows you to continue running your current instrument configuration and established methods. The integrated software walks you through a step-by-step process to manage the transition to powerful, triple quadrupole methods.

FIGURE 1. Overview of triple quadrupole method development.



Full Scan Data

SVOC calibration curves were generated from 1 to 200 ppm in methylene chloride and analyzed on the TSQ Duo GC-MS/MS and ISQ GC-MS instruments. A comparison of the %RSDs of the curves is shown in Figure 2. A comparison of 10 replicate injections at 1 ppm is displayed in Figure 3.

FIGURE 2. Comparison between the TSQ Duo GC-MS/MS and the ISQ GC-MS %RSDs of the curve for 100+ compounds.

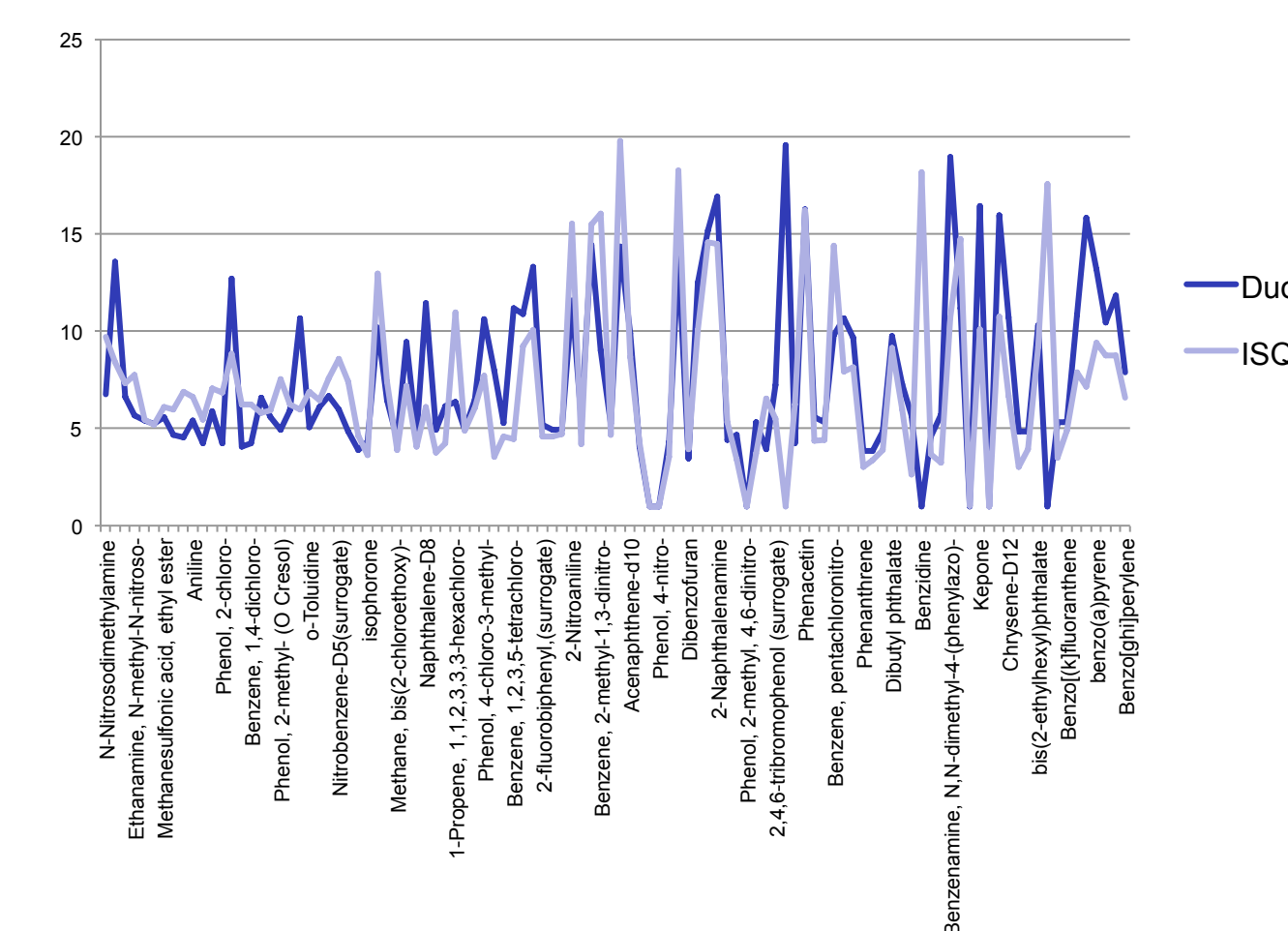
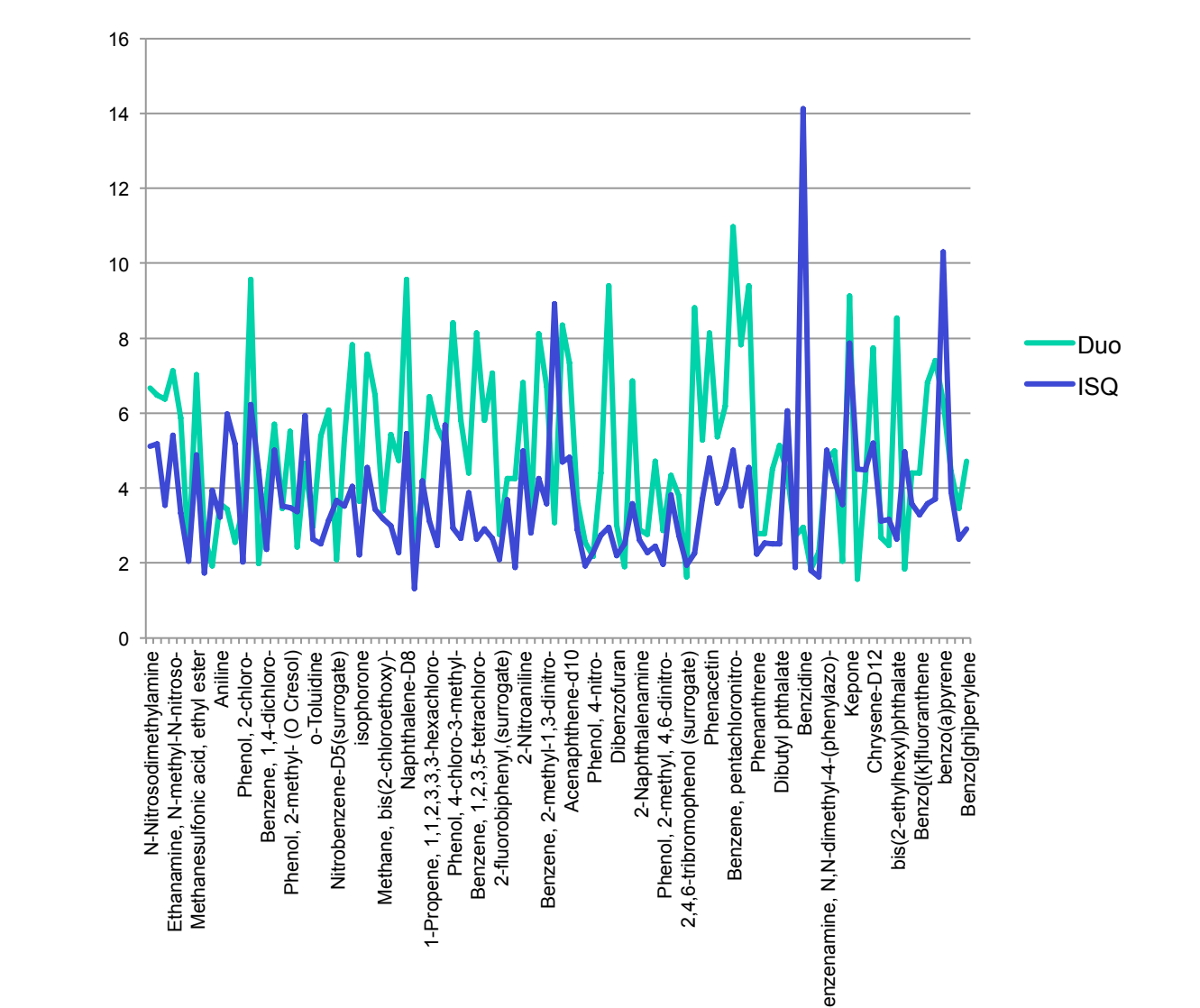


FIGURE 3. Comparison between the TSQ Duo GC-MS/MS and the ISQ GC-MS %RSDs of 10 replicates at 1 ppm.



SIM Data

SIM was used for the analysis of THC in urine samples. THC, THC-OH, and THC-COOH were extracted and derivatized for this study. Concentrations used were the cutoff value (15 ng/mL), 125% of the cutoff value (18.75 ng/mL), and 40% of the cutoff value (6 ng/mL). Table 1 shows the %RSDs of 10 replicate analyses of these three concentrations.

TABLE 1. %RSD of 10 replicates THC samples – TSQ Duo vs ISQ.

| | TSQ Duo Cutoff | ISQ Classic Cutoff | TSQ Duo 125% Cutoff | ISQ Classic 125% Cutoff | TSQ Duo 40% Cutoff | ISQ Classic 40% Cutoff |
|----------|----------------|--------------------|---------------------|-------------------------|--------------------|------------------------|
| THC | 2.8 | 5.8 | 4.6 | 9.4 | 3.9 | 2.4 |
| THC-OH | 2.3 | 2.8 | 4.4 | 4.0 | 8.2 | 1.9 |
| THC-COOH | 2.0 | 3.2 | 2.8 | 3.5 | 9.5 | 1.9 |

SRM Data

SVOC calibration standards were prepared in methylene chloride from 1 to 1000 ppb. Transitions and collision energies for SRM were determined and optimized using the AutoSRM function of the software. Curves using internal standards were generated. Figure 4 displays the linearity of the curves. Figure 5 is a comparison of the curve %RSDs for the TSQ Duo GC-MS/MS and the ISQ GC-MS.

FIGURE 4. r² values for a curve 1 ppb to 1000 ppb.

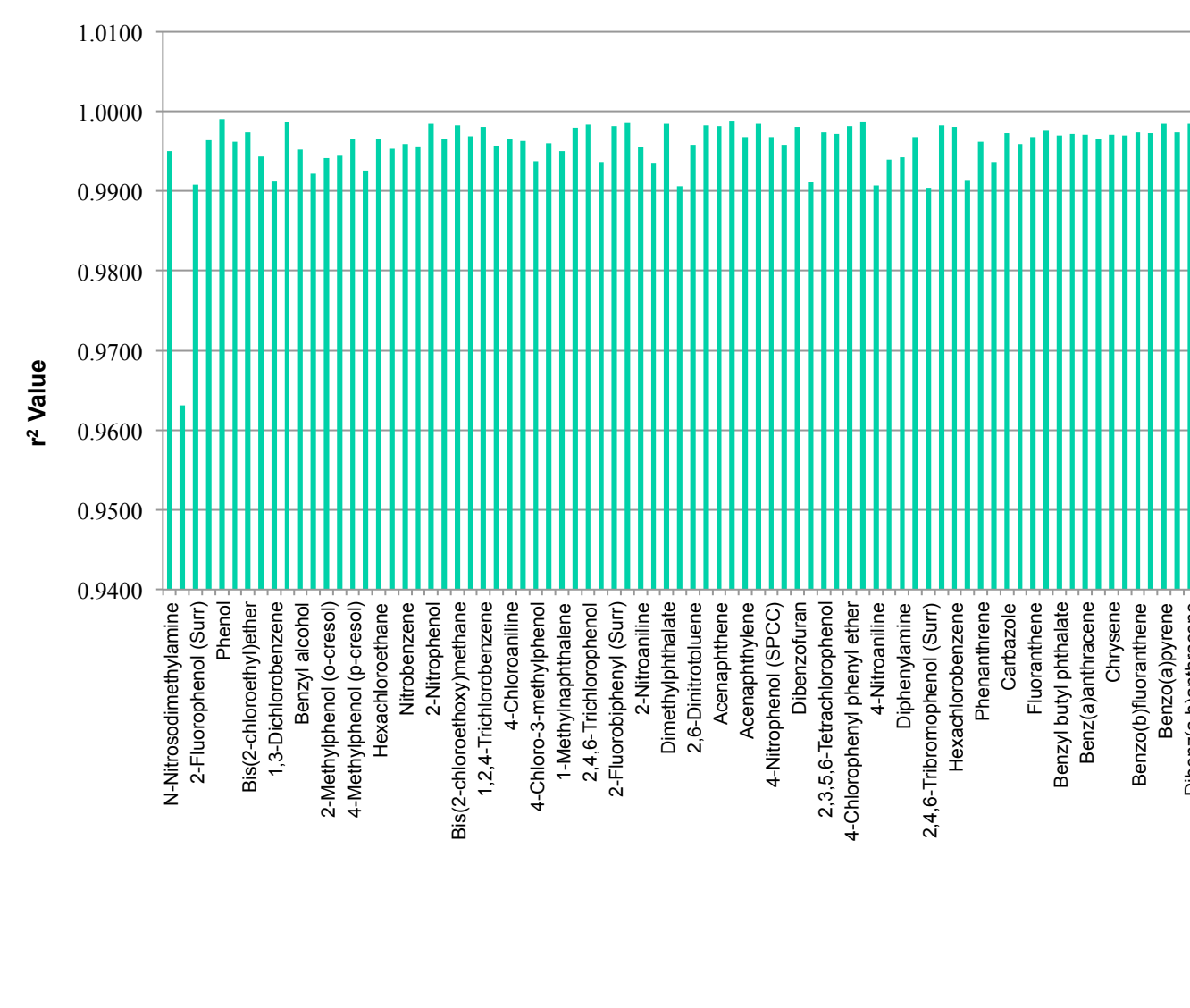
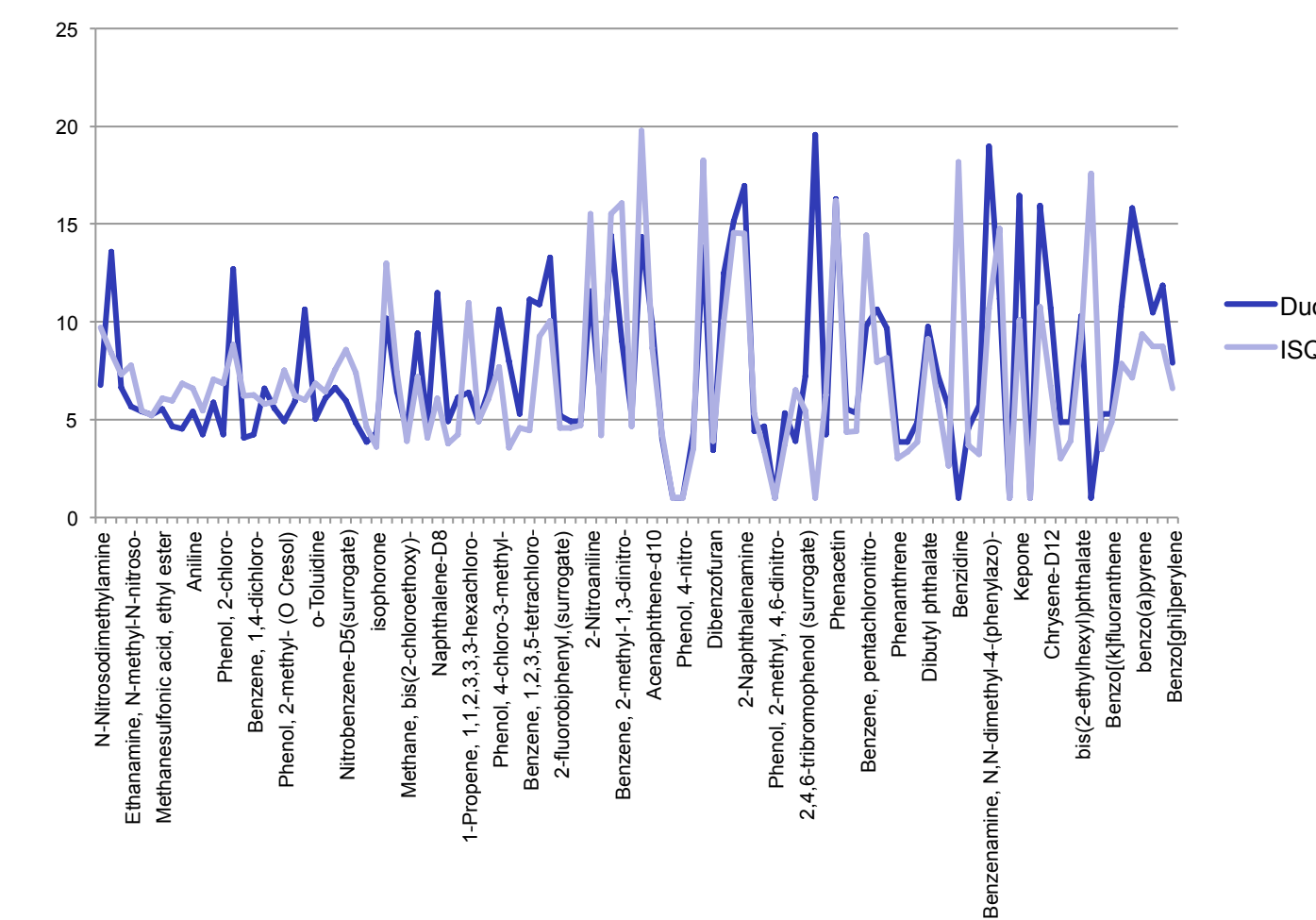


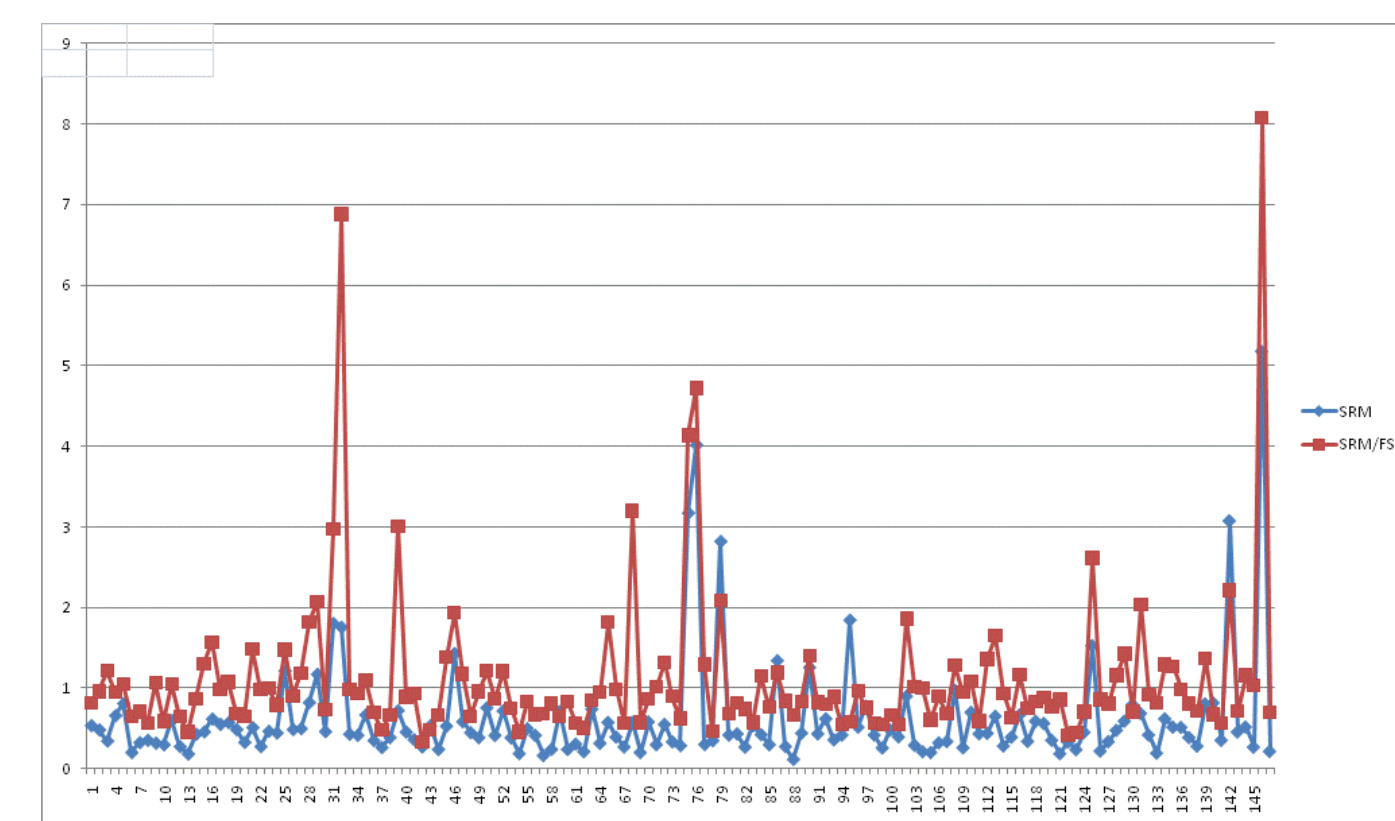
FIGURE 5. %RSDs for the calibration curve – TSQ Duo and ISQ.



SRM/FS Data

A sample of fruit drink was extracted using the QuEChERS method of extraction and cleanup. The extract was concentrated 5x, and then 147 pesticides were spiked into the extract to produce calibration curves from 1 ppb to 200 ppb. The calibration curves were constructed for both methods: SRM and alternating SRM/full scan for 147 pesticides. The linearity for most of the compounds was R² > 0.98 for both methods of analysis. Ten replicates of a 1 ppb and 10 ppb standard in fruit juice extract were analyzed to determine the MDLs for the two instrument methods. A comparison of the MDLs of both methods are shown in Figure 6. MDLs are slightly higher with the full scan added to the instrument method, but very comparable.

FIGURE 6. Comparison of MDLs from SRM vs. SRM/FS analysis (ppb).



Fruit drink was spiked at 100 ppb and analyzed using the SRM/full scan instrument mode. This extract was also spiked with two phthalates at a 1 ppm level. The full scan chromatogram shows several peaks above the 100 ppb pesticide spike. Peaks are at retention times of 9.29, 9.73, 10.39, 10.91, and a very large saturated peak at 31.00 minutes. A close-up view of the first four compounds is shown in Figure 7. Figure 8 displays the NIST library matches for those non-targeted compounds.

FIGURE 7. Close-up view of four unknown peaks in 100 ppb spiked fruit drink.

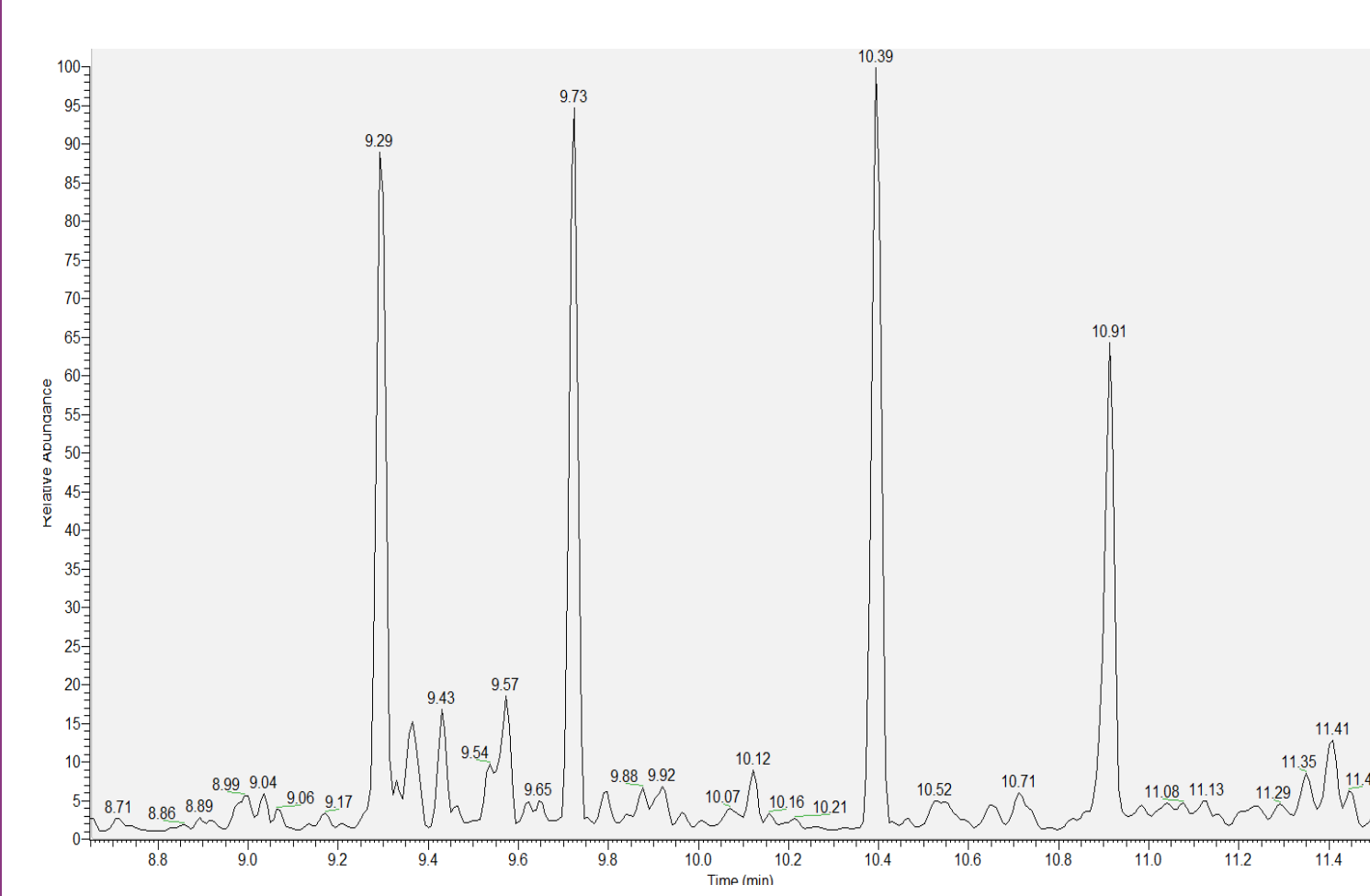
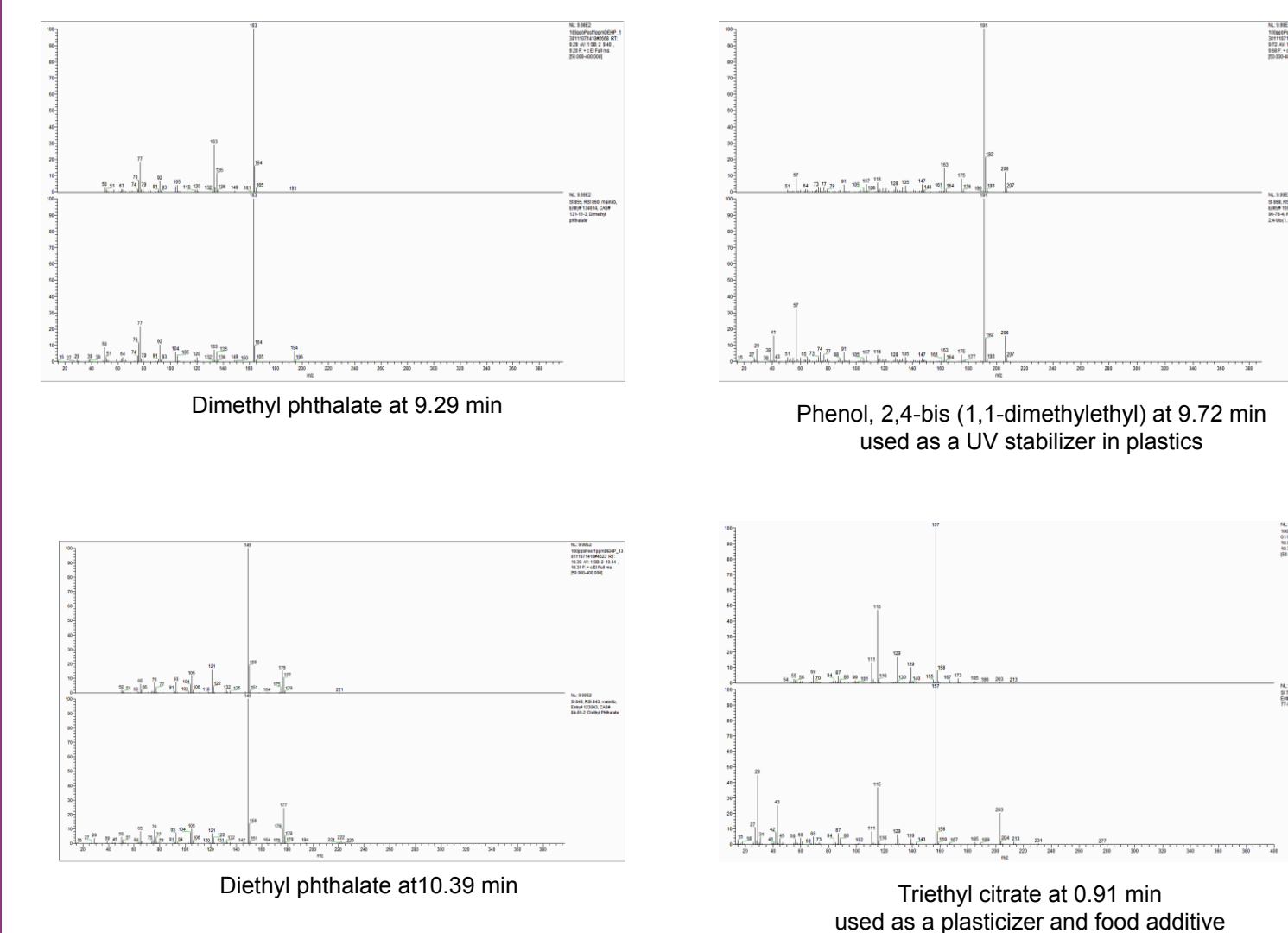


FIGURE 8. NIST Library match for four unknown peaks.



Conclusion

As can be seen from this data, the TSQ Duo GC/MS-MS system can be used as a single quadrupole instrument that provides additional triple quadrupole capabilities to any laboratory. Whether the application is full scan, SIM, SRM, or a combination of SRM and full scan, the TSQ Duo system produces sensitive, accurate results.

All trademarks are the property of Thermo Fisher Scientific and its subsidiaries. This information is not intended to encourage use of these products in any manner that might infringe the intellectual property rights of others.