

## Rapid and Sensitive Detection of 8 Organic Explosives in Soil

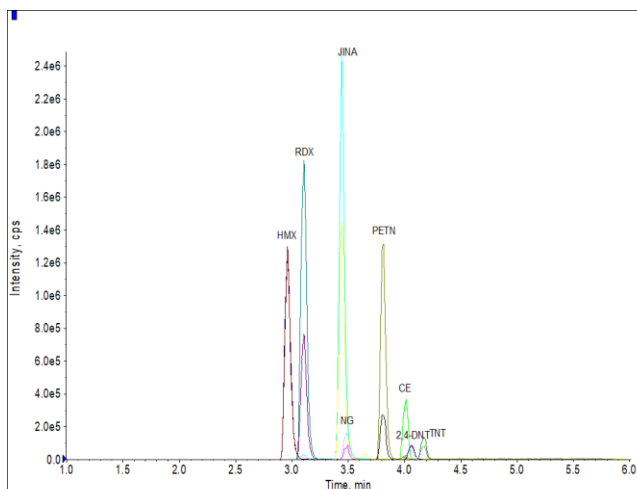
Using the SCIEX Triple Quad™ 3500 LC-MS/MS System with an ExionLC™ AD System

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The widespread use of explosive materials by the military, blasting, mining and other industries has fueled the continuous development of explosive production technology. The fast and simple process of manufacturing explosive devices has also enabled terrorists to clandestinely construct and use explosives to pursue their criminal endeavors. As a result, the presence of explosives in geographical warzones or in crime scenes has important political and legal implications.

Although their detection provides valuable legal evidence of their use as weapons, the daily occurrence of explosive materials is a rising concern for health and legal authorities alike. In addition to their destructive nature, the majority of organic explosive materials are carcinogenic to the human body. Inevitably, their production and use have serious impact on environmental pollution and contamination and can pose serious health issues to the population exposed to it. As a result, rapid and accurate screening techniques are critically needed to detect trace levels of explosives with a high level of sensitivity and selectivity.

Mass spectrometry is a sensitive analytical technique with the throughput and selectivity required for rapid and confident security screening. Because explosive compounds are intrinsically rich in electron-withdrawing nitrogen-containing groups and thermally instable in nature, they are suitable for analysis by LC-MS/MS technology.



**Figure 1: Chromatographic Profile of the 8 Organic Explosives.** Extracted Ion Chromatogram (XIC) shows rapid LC separation and identification of the 8 organic explosives used in this study.



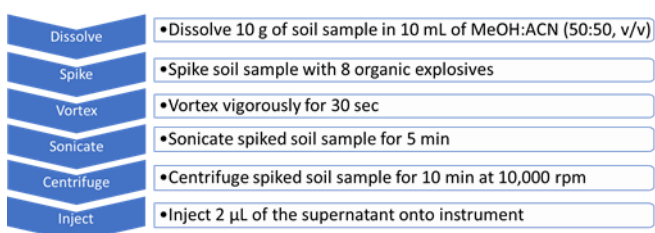
In this technical note, a comprehensive workflow combining the use of the SCIEX Triple Quad™ 3500 System with a simple and fast sample preparation procedure for the sensitive detection of 8 organic explosives is described. The method is shown to provide a simple solution for high-throughput detection of trace explosives in soil samples.

### Key Features of the SCIEX Triple Quad 3500 LC-MS/MS System for High-Throughput Detection of Organic Explosives in Soil

- Sample pre-treatment method is fast and simple, which greatly reduces sample preparation time while negating the need for time-consuming steps such as solid-phase extraction (SPE), evaporation and reconstitution
- Fast, 8 minute LC run provides high throughput and baseline separation of the 8 organic explosives used in this study
- MRM acquisition method on the SCIEX Triple Quad 3500 LC-MS/MS System provides accurate and sensitive quantitation of the organic explosives in soil samples
- Excellent specificity and sensitivity, with LODs in the pg/g levels for the organic explosives in soil samples
- Excellent linearity from 0.05 to 1000 ng/g is observed for all 8 organic explosives accurately identified in the soil samples
- Excellent negative ion mode sensitivity for explosive detection

## Methods

**Sample Preparation:** 10 g of surface soil samples were dissolved in 10 mL of methanol:acetonitrile (50:50, v/v), spiked at various concentration levels with a standard mixture containing the 8 organic explosives, vigorously vortexed for 30 seconds, sonicated for 5 minutes and centrifuged at 10,000 rpm for 10 minutes. 2  $\mu$ L of the supernatant was injected for analysis. Table 1 lists the 8 organic explosives used in this study. The sample preparation procedure is summarized in Figure 2.



**Figure 2. Sample Preparation Procedure for the 8 Organic Explosives in Soil Samples.** A 6-step sample pre-treatment method was used for preparing the soil samples for analysis with the SCIEX Triple Quad™ 3500 LC-MS/MS System.

**Liquid Chromatography:** LC separation was performed using a Phenomenex Kinetex biphenyl column (50  $\times$  3 mm, 2.6 $\mu$ m, 00A-4723-AN) held at 40 °C on a SCIEX ExionLC™ AD System. Mobile phases used were ammonium formate, ammonium chloride, and methanol. The flow rate was 0.4 mL/min. The injection volume was 2  $\mu$ L and the total LC runtime was 8 min.

**Mass Spectrometry:** A SCIEX Triple Quad 3500 LC-MS/MS System with IonDrive™ Turbo V source and Electrospray Ionization (ESI) was used. The 8 organic explosives were detected in negative ion mode using two MRM transitions per compound to allow quantification and confirmation of the explosive compounds.

**Data Analysis:** Data was acquired in Analyst® Software 1.7 and processed in MultiQuant™ Software 3.0.3.

**Table 1. List of the 8 Explosive Compounds Analyzed in This Study Using the SCIEX Triple Quad 3500 System.**

Compound	Abbreviation
2,4,6-trinitrobenzidine	CE
Octahydro-1,3,5,7-tetranitro-1,3,5,7-triazine	HMX
Pentaerythritol tetranitrate	PETN
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX
2,4-Dinitrotoluene	2,4-DNT
Diethanolnitramine dinitrate	DINA
2,4,6-trinitrotoluene	TNT
Nitroglycerine	NG

## Developing a Robust Workflow for the Detection of 8 Explosives in Soil Samples

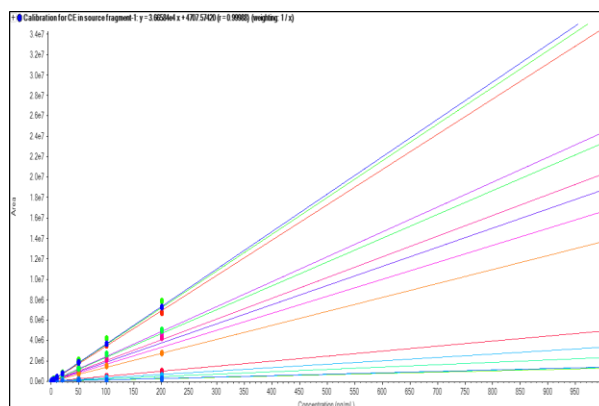
10 g of surface soil samples were dissolved in 10 mL of methanol:acetonitrile (50:50, v/v) and spiked with the 8 organic explosives at various concentrations. The resulting samples were vortexed vigorously, sonicated for 5 minutes, centrifuged at 10,000 rpm for 10 minutes and 2  $\mu$ L of the supernatant was injected to the instrument to build a data processing method.

Figure 1 shows the extracted ion chromatogram (XIC) for the 8 organic explosives. The combination of an appropriate mobile phase composition, column choice and optimized LC conditions resulted in baseline separation of all 8 organic explosives.

## Accurate and Sensitive Quantification of Organic Explosives

The quantitative performance of the assay was investigated by injecting a series of soil samples spiked with the explosive compounds at various concentration levels ranging from 0.05 to 1000 ng/g. Calibration curves were generated to evaluate the sensitivity and linearity of the assay as well as the linear dynamic range of the instrument for each of the 8 explosive compounds.

Figure 3 shows the calibration curves for both the quantifier and qualifier ions of the 8 explosive compounds for concentrations ranging from 0.05 to 1000 ng/g. The calibration curves show excellent linearity, with  $R^2$  values >0.99 for all the explosive compounds. Sensitivity down to sub ng/g was achieved and the linear dynamic range exceeded 4 orders of magnitude for the majority of the explosive compounds without internal standard correction. This demonstrates that the SCIEX Triple Quad 3500 System provides sensitive and accurate quantification of explosive compounds across a wide range of concentration levels. Table 2 summarizes the quantitative results for the assay and includes the concentration range and the  $R^2$  values for the 8 explosive compounds analyzed in this study.



**Figure 3. High Sensitivity, Linearity, and Dynamic Range for the 8 Explosive Compounds.** Calibration curves resulting from the calibration series for both the quantifier and qualifier ions of the 8 explosive compounds from 0.05 to 1000 ng/g.

**Table 2. Summary of the Quantitative Performance of the SCIEX Triple Quad 3500 LC-MS/MS System for the Detection of the 8 Organic Explosive Compounds Analyzed in This Study.**

Compound Name	Chemical Formula	Precursor Mass (Da)	Molecular Species	Concentration Range (ng/g)	R <sup>2</sup> Value
CE	C <sub>7</sub> H <sub>5</sub> N <sub>5</sub> O <sub>8</sub>	288.0	<i>In-source cleavage</i>	0.05-200	0.9998
HMX	C <sub>4</sub> H <sub>8</sub> N <sub>8</sub> O <sub>8</sub>	331.0	[M+Cl] <sup>-1</sup>	0.05-200	0.9987
PETN	C <sub>5</sub> H <sub>8</sub> N <sub>4</sub> O <sub>12</sub>	351.0	[M+Cl] <sup>-1</sup>	0.5-1000	0.9997
RDX	C <sub>3</sub> H <sub>6</sub> N <sub>6</sub> O <sub>6</sub>	267.0	[M+FA] <sup>-1</sup>	0.05-200	0.9995
2,4-DNT	C <sub>7</sub> H <sub>6</sub> N <sub>2</sub> O <sub>4</sub>	181.1	[M-H] <sup>-1</sup>	1-1000	0.9999
DINA	C <sub>4</sub> H <sub>8</sub> N <sub>4</sub> O <sub>8</sub>	285.1	[M-H] <sup>-1</sup>	0.2-1000	0.9993
TNT	C <sub>7</sub> H <sub>5</sub> N <sub>3</sub> O <sub>6</sub>	226.0	[M-H] <sup>-1</sup>	1-1000	0.9997
NG	C <sub>3</sub> H <sub>5</sub> N <sub>3</sub> O <sub>9</sub>	262.1	[M-H] <sup>-1</sup>	1-1000	0.9981

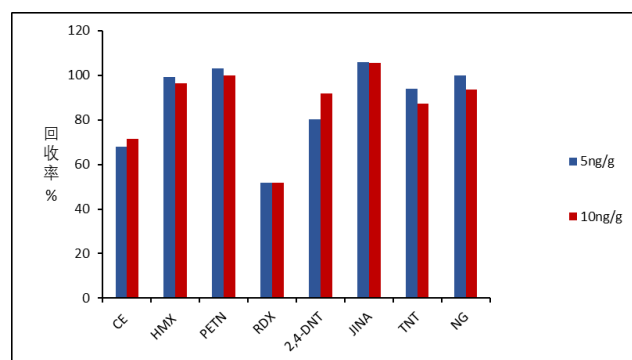
## Fast and Simple Sample Pre-Treatment Leads to High Recovery of Explosive Compounds in Soil Samples

One of the challenges associated with the detection of analytes extracted from soil samples is the presence of many matrix components that can affect the detection performance of the assay. As a result, a reliable sample preparation procedure is critical to achieve the desired reproducibility and analytical performance of the assay.

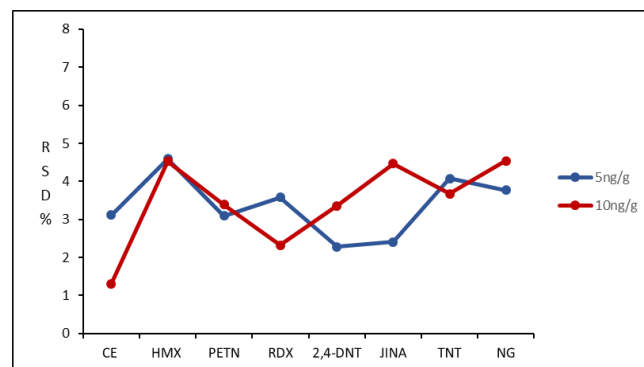
To assess the efficiency of the sample preparation procedure used in this experiment, the recovery was calculated by spiking a soil sample with the standard mixture at two concentration levels (5 and 10 ng/mL) before and after the sample pre-treatment procedures. Three replicate injections were performed for each of the two concentrations. The recovery values (depicted as “RE”) were calculated for each of the 8 organic explosive compounds at both concentration levels by dividing the average peak area of the pre-spiked sample (depicted as “A”) by the average average peak area of the post-spiked sample (depicted as “B”) as shown in the following equation:

$$RE (\%) = A/B \times 100 \quad (1)$$

The sample preparation procedure used in this experiment demonstrated recovery values ranging between 51.7 and 106%, as shown in Figure 4. In addition, the assay showed excellent reproducibility across the three injections for the two concentration levels with RSD values between 1.31 and 4.59% (Figure 5) for all the explosive compounds used in this study. The result shown in Figure 4 and 5 illustrate the overall robustness and reproducibility of the assay.



**Figure 4. Reproducible and Consistent Recovery Values Observed for All 8 Organic Explosive Compounds Across the Two Concentration Levels.** Diagram showing the recovery (RE) values calculated for the 8 organic explosive compounds at 5 ng/g and 10 ng/g across three replicate injections.



**Figure 5. High Reproducibility of Injection for All 8 Organic Explosive Compounds Across the Two Concentration Levels.** Plot showing the RSD values values calculated for the 8 organic explosive compounds at 5 ng/g and 10 ng/g across three replicate injections.

## Conclusions

A complete workflow for the detection of 8 organic explosives in soil samples was successfully developed using the SCIEX Triple Quad 3500 System. The combination of a simple sample preparation procedure and a robust LC-MS method enabled accurate detection of the organic explosives in soil samples.

- A simple sample preparation procedure enabled fast and reproducible extraction of 8 organic explosives from soil samples without the need for long and tedious sample extraction procedures commonly needed for this type of matrix
- Quantification of the 8 organic explosives in a single method
- Explosive compound extraction recovery values were found to be between 51.7 and 106%
- Reproducibility of injection (measured as RSD) across two concentration levels (5 ng/g and 10 ng/g) was found to be between 1.31 and 4.59% for all the explosive compounds
- The optimized LC-MS/MS method on the SCIEX Triple Quad™ 3500 LC-MS/MS System enabled sensitive detection, with LODs in the pg/g levels for the organic explosives in soil samples
- Excellent accuracy and linearity for concentrations ranging between 0.05 and 1000 ng/g, with R<sup>2</sup> values above 0.99 for all 8 explosive compounds used in this study
- Overall, the developed workflow enabled reproducible, sensitive and reproducible detection of 8 organic explosives from soil samples

## References

1. Gilbert-López Bienvenida; Lara-Ortega Felipe J, *et al.* (2019) Detection of multiclass explosives and related compounds in soil and water by liquid chromatography-dielectric barrier discharge ionization-mass spectrometry. *Analytical and Bioanalytical Chemistry*, **411** (2), 11-17.
2. S. Ehlert, J. Hölzer, *et al.* (2013) Rapid on-site detection of explosives on surfaces by ambient pressure laser desorption and direct inlet single photon ionization or chemical ionization mass spectrometry. *Indian Journal of Pharmaceutical Sciences*, **405** (22), 6979-6993.

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