

Analysis of Explosives in Soil Samples:

High-Throughput Screening at 8 Seconds per Sample Using Luxon Ion Source®

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Introduction

In forensic science, explosives analysis of swabbed surfaces is used in crime scene investigation. Jagerdeo et al¹ report a rapid screening method for explosives analysis using LDTD® technology for forensic application. The Environmental Protection Agency (EPA) developed Method 8330B (SW-846) for the analysis of explosives in different matrices (water, soil or sediment) using a HPLC-UV method.

Our goal for this application note is to develop an automated and fast screening tool for explosives analysis in soil samples using the Luxon Ion Source®, based on the LDTD® technology.

LDTD®-MS/MS offers specificity combined with an ultra-fast analysis for an unrivaled screening method. To develop this application, we adapted and automatized the EPA extraction method. Explosives are analyzed, and results are obtained in less than 8 seconds per sample.

Luxon Ionization Source

The Luxon Ion Source® (Figure 1) is the second-generation sample introduction and ionization source based on the LDTD® technology for mass spectrometry. Luxon Ion Source® uses Fiber-Coupled Laser Diode (Figure 2) to obtain unmatched thermal uniformity providing more precision, accuracy, and speed. The process begins with dry samples which are rapidly evaporated using indirect heat. The thermally desorbed neutral molecules are carried into a corona discharge region. High efficiency protonation and strong resistance to ionic saturation characterize this type of ionization and is the result of the absence of solvent and mobile phase. This thermal desorption process yields high-intensity molecular ion signal in less than 1 second sample-to-sample and allows working with very small volumes.



Figure 1 - Luxon Ion Source®



Figure 2 - Schematic of the Luxon Ionization Source

Sample Preparation Method

Sample Collection

1 g of soil samples were transferred in 2 mL barcoded tubes and mixed with 1 mL of internal standard (ETN, 200 ng/mL in acetonitrile). After centrifugation, samples were transferred into the Azeo Liquid Handler (Figure 3).

Automated Sample Extraction

Each barcoded vial was scanned by the Azeo Liquid Handler and an automatic batch file was created. The Azeo extraction system is used to extract the samples using the following conditions:

- 200 µL of Ammonium chloride (5 mM in Methanol:Water / 75:25) were added to a deep-well plate placed on the Lumo Vortexer
- 100 µL of sample were transferred from the vials to the deep-well plate placed on the Lumo Vortexer
 - Mix
- Spot 6 µL of the mixture on a LazWell™-DEC 96 plate
 - Dry 4 minutes at 40 °C

Note: For a cleaner extract, a salt assisted liquid-liquid extraction (SALLE) can be used in this automated extraction process.



Figure 3 - Automated extraction system

LDTD®-MS/MS Parameters

LDTD

Model: Luxon S-960, PhytroniX

Carrier gas: 3.0 L/min (air)

Laser pattern:

- 6-second ramp to 65% power
- Hold 2 seconds at 65% power

MS/MS

MS model: QTrap® System 5500, Sciex

Curtain: 10

CAD: 6

IonSpray Voltage: 6000

Temperature, GS1 and GS2 set to zero.

Scan Time: 20 msec

Analysis Method: Negative MRM mode

Table 1 - MRM transitions for LDTD-MS/MS

Explosive	Transition	CE
RDX	257.0 → 46.0	-30
NG	262.0 → 46.0	-20
Tetryl	322.0 → 199.0	-10
HMX	331.0 → 109.0	-25
ETN (IS)	337.0 → 46.0	-30
PETN	351.0 → 46.0	-45

Results and Discussion

Screening Cut-off Test (ppb)

Explosives list and screening cut-offs reached on the mass spectrometer used can be found in **Table 2**.

Table 2 - Explosive cut-offs

Explosive	Cut-off (ppb)
HMX	4
RDX	10
PETN	20
Tetryl	40
NG	100

Precision

Spiked samples around the decision point (50% cut-off: QC-L, cut-off: CO and 200% cut-off: QC-H) and blank extracts were used to validate the precision of the method. The peak area against the internal standard (IS) ratio was used to normalize the signal. Replicate extractions were deposited onto a LazWell™-DEC plate and dried before analysis.

The following acceptance criteria were used:

- Each concentration must not exceed 20% CV
- The mean concentration ± 2 times the standard deviation must not overlap with other concentrations at the cut-off.

For the inter-run precision experiment, each fortified sample set is analyzed in triplicate on five different days. **Table 3** shows the inter-run precision results. No overlapping at the cut-off is observed for PETN and the %CV was below 20%. Similar results were obtained for the other explosives in the panel.

Table 3 - Inter-Run Precision

PETN	QC-L	CO	QC-H
Conc (ppb)	10	20	40
N	15	15	15
Mean (ppb)	9.3	19.2	39.5
%CV	10.6	9.4	7.2
Mean - 2SD (ppb)	7.4	15.6	33.8
Mean + 2SD (ppb)	11.3	22.8	45.2

For the intra-run precision experiment, each fortified sample is extracted and analyzed in 8 replicates. Area ratio results are plotted using the ± 2 STD error bars. **Figure 4** shows the intra-run results for PETN. No overlapping is observed for each concentration and the %CV was below 20%. Similar results are obtained for the other drugs in the panel.

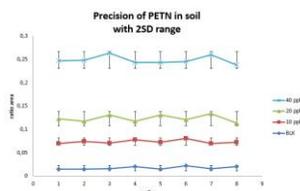


Figure 4 - Intra-Run Precision Curves for PETN

Wet Stability of Sample Extracts

Following the extraction, sample extracts were kept at 4°C in closed containers. After 1 day, sample extracts were spotted on a LazWell™-DEC plate, dried and analyzed. Precision QCs samples are reported in **Table 4**. All the results were within the acceptable criteria range for 1 day at 4°C.

Dry Stability of Samples Spotted in LazWell™

Extracted samples were spotted onto a LazWell™-DEC plate, dried and kept at room temperature for 1 hour before analysis. The precision of QC samples is reported in **Table 4**. All the results were within the acceptable criteria range for 1 hour at room temperature.

Table 4 - Wet and Dry Stability of PETN

Parameters	Dry stability (1 hour / RT)			Wet stability (1 day / 4°C)		
	QC-L	CO	QC-H	QC-L	CO	QC-H
PETN	10	20	40	10	20	40
Conc. (ppb)	10	20	40	10	20	40
N	3	3	3	3	3	3
Mean (ppb)	9.6	19.9	40.4	9.0	18.3	40.1
%CV	5.5	6.7	4.7	4.1	7.4	7.2

Field map evaluation

To simulate a field sampling, deep-well plates were filled with 96 X 1 g of negative soil. A HMX solution was added in the middle of the plate and dried overnight. All 96 samples were extracted and screened to evaluate the contaminated area (concentration higher than cut-off screen). **Figure 5** shows the heat map results of HMX positive area (white means it is below the cut-off value. Yellow to red is a positive sample).

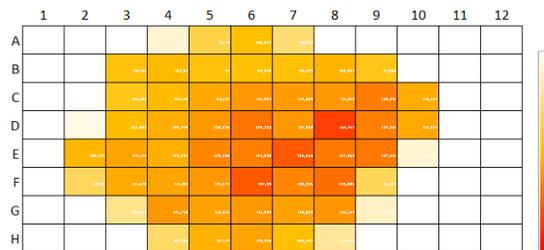


Figure 5 – Heat map of HMX screen

Conclusion

The Luxon Ion Source® combined with Sciex QTrap® 5500 mass spectrometer system allows the ultra-fast (**8 seconds per sample**) analysis of explosives in soil.

References

Jagerdeo, E and Auger, S. Rapid screening procedures for a variety of complex forensic samples using laser diode thermal desorption (LDTD) coupled to different mass spectrometers. Rapid Commun Mass Spectrom. 2022;36:e9244. <https://doi.org/10.1002/rmc.9244>