

A Targeted Method for Explosives Detection on a Prototype Thermal Desorption-QDa instrument.

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INTRODUCTION

Tests for explosive residues at transport locations (airports etc) are predominantly conducted using ion mobility instrumentation which can be limited for some compounds of interest. In this poster we discuss the use of a prototype thermal desorption APCI source attached to a small single quadrupole mass spectrometer to investigate the response of a range of explosives.

In collaboration with Mass Spec Analytical (MSA) and initially with the Defence Science and Technology Laboratory (Dstl), and Surrey University, we have been investigating the possibility of using the Waters QDa single quadrupole mass spectrometer as a base for a specific explosives residue detector. Challenges have included:

- ♦ An appropriate volatilisation process for the current sampling method
- ♦ The most generic ionisation process
- ♦ Sensitivity for all analytes at 1-10ng
- ♦ Increased specificity through an appropriate method
- ♦ A real time acquisition/identification software.

All the Ion Mobility instruments that currently dominate this market use a swabbing technique to sample luggage surfaces (or peoples hands) prior to the swab being heated in a thermal desorption unit to volatilise all compounds on the swab. A number of thermal desorption units have been developed, the following discussion is based on a prototype unit designed and supplied by MSA.



Figure 1. Shows the front of the prototype thermal desorption source with two heated blocks to induce volatilisation and used to show proof of principle.

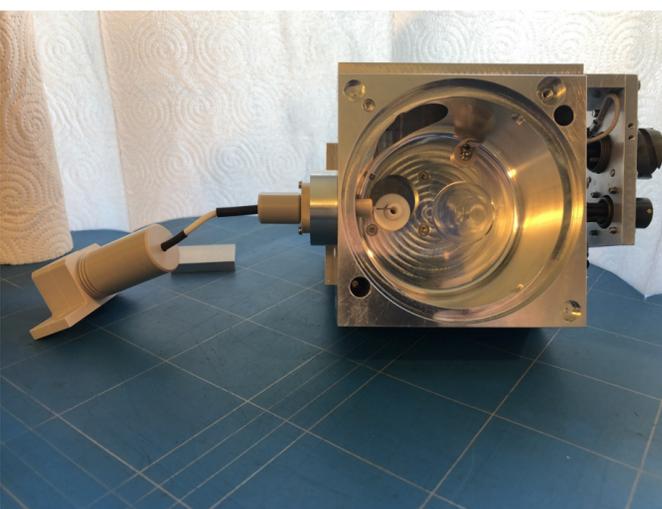


Figure 2. Shows the back of the prototype thermal desorption source. The ceramic inlet tube and the corona pin used to ionise the volatilise analytes can be seen.

METHODS

The MS is used in full scan mode using a two function cone voltage experiment to detect nanogram levels of the explosives. This type of acquisition method increases the specificity of detection and provides both nominal mass and structural confirmation.

Samples.

A range of explosives were investigated including: TNT, DNT, RDX, PETN, HMX, TATP and HMTD.

There are a significant number of potential variables that can affect the spectral information and sensitivity of the instrument. These were optimised for maximum sensitivity however, the following data does not provide an exhaustive investigation of these potential variables.

System setup (Negative mode)

The TD unit was set at 200°C and the corona voltage was -2.4kV, with a flow of 10l/min through the exhaust, produced by a standalone backing pump. 1µl of the sample was injected directly into the injection region between the heated plates. The source temperature was 150°C with the cone voltage set to 5V (single function) or 5V and 20V for the two function experiment to investigate chemical fragmentation.

A full scan method (50-550 m/z) at each cone voltage was collected in continuum at 5Hz.

(Positive Ion) The TD unit was set at 200°C and the corona voltage was 2.5kV, with a flow of 10l/min through the exhaust. A full scan method (50-550 m/z) at each cone voltage was collected in continuum at 5Hz.

RESULTS/DISCUSSION

Below are some of the results obtained for the explosives investigated. Figure 3 shows the reconstructed ion chromatogram (RIC) of the [M-H]⁻ ion (226 m/z) and the M⁻ species (227 m/z) of TNT. A response is clearly observed for each of the three injections of the analyte.

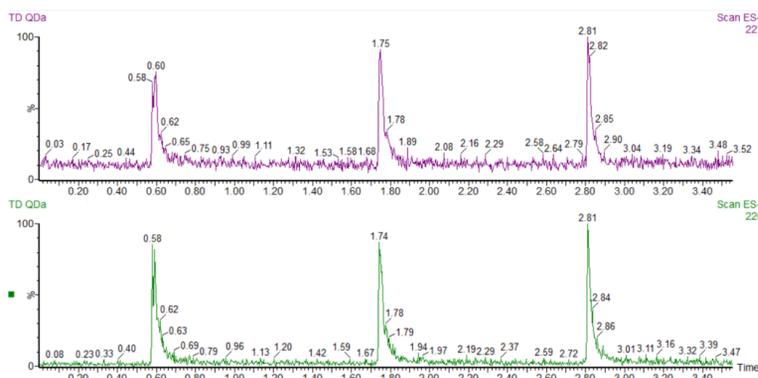


Figure 3. Shows the RIC of the [M-H]⁻ ion (226 m/z) and the M⁻ species (227 m/z) of TNT at 1ng loading.

Figure 4 shows the full mass spectrum of the TNT sample loading at the 1ng (a level relevant for homeland security applications). A two function full mass scan experiment was completed for TNT at this level to improve the specificity of a library match application.

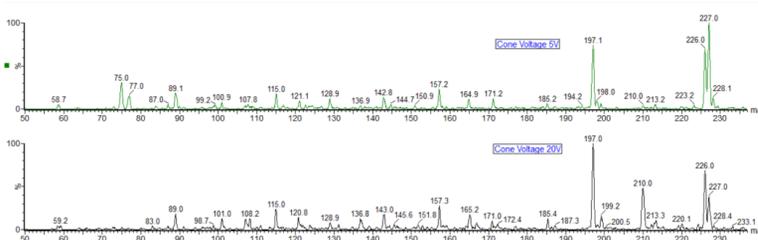


Figure 4. Shows the full mass spectrum at 5 and 20V for TNT at 1ng loading.

The premise of a two function experiment provides the possibility of increased specificity if enough additional fragments can be produced from the higher cone voltage experiment for all of the compounds of interest within the source ionisation environment. The higher energy does increase fragmentation with a reduction in 227 m/z and an increase in 210 m/z. A higher energy may increase specificity further.

Figure 5 shows other explosives acquired using a full scan experiment with the RIC responses of the appropriate adduct ions (acquired with a low cone voltage). 1µl of sample (1ng/µl) was injected into the injector of the heated plates module. Responses for all the less volatile compounds, (RDX, HMX and PETN) were observed. The PETN response was obtained with a TD unit temperature of 200°C while the RDX and HMX TD temperature was set at 250°C.

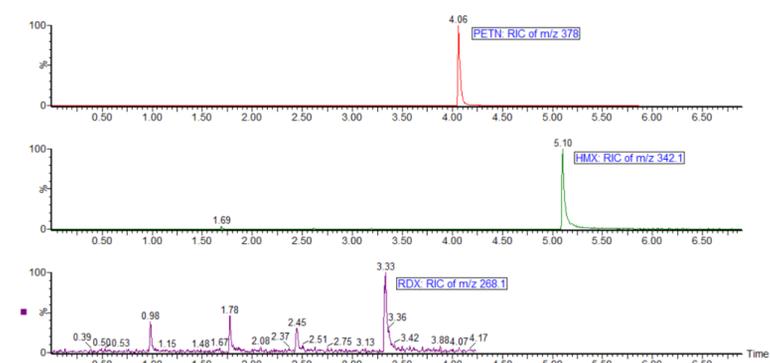


Figure 5. Shows the Responses for RDX, HMX and PETN. The PETN adduct response is based on the [M+NO₃]⁻ while the RDX and HMX are both constructed from the [M+NO₂]⁻ adduct

In addition, the analysis of some of the more difficult improvised explosives were also investigated, these included TATP and HMTD. Both of these compounds are analysed in positive ion mode (See Figure 6). The thermal desorption unit was set at 200°C and 1µl of sample (10ng/µl) was applied to a foil surface and allowed to dry. The backing gas flow was 10l/min, with the corona voltage at 2.5kV and the CE at 5V.

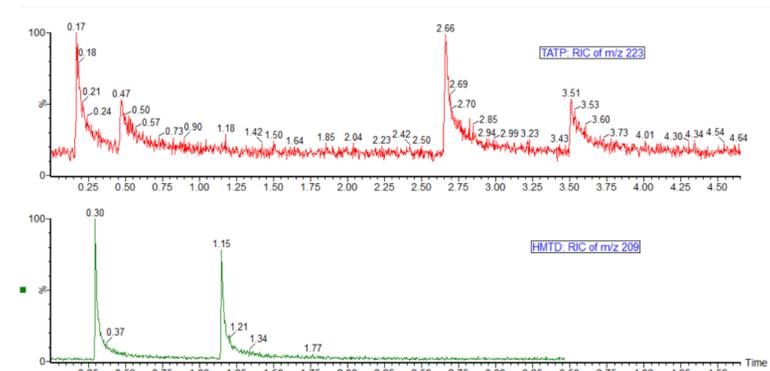


Figure 6. Responses were observed for both the TATP and HMTD analytes, although these were at elevated concentrations of 10ng. The TATP and HMTD adduct responses are both based on the [M+H]⁺.

CONCLUSION

The above data acquired on a prototype/proof of principle system (in collaboration with Mass Spec Analytical) has shown the possibility of a small mass spectrometer capable of the detection of explosive residues in the 1-10ng level. A new version 2 prototype is currently being produced (by Mass Spec Analytical) to increase sensitivity and reduce size. A single TD temperature also needs determining, as currently different analytes volatilised optimally at different temperatures.

FURTHER WORK

The specific identification of explosive adducts/fragments using a two function (or more if required) cone voltage experiment needs to be expanded to all analytes as this could be used to produce increased specificity of the assay and reduce false positive through the use of an appropriate two function library matching software.