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The Use of Advanced Analytical Techniques to Enable Batch and Source Matching of Homemade Explosives

A thesis submitted for fulfilment of the degree of Doctor of Philosophy

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Contents

Summary

This thesis describes the application of advanced analytical techniques: namely isotope ratio mass spectrometry (IRMS) and inductively coupled plasma mass spectrometry (ICP-MS) to the analysis of a variety of ammonium nitrate (AN) based homemade explosives.

AN has been widely used in the preparation of homemade explosives (HME) due its relative stability and ease of acquisition. The aim of this research was to develop methods that enable the identification of batch-to-batch matches between samples and to determine the origin of source materials used in such mixtures.

The work described in Chapter 2 indicated that the IRMS technique has the potential to discriminate samples of AN-based explosives due to the variations in their isotopic composition, e.g. nitrogen and carbon. An investigation on the ICP-MS technique is also described, which allowed for the multi-elemental profiling of trace impurities present in AN and AN-based explosives. These trace impurities may be used to compare samples in order to identify samples that have a similar origin or manufacturing process.

Lab based samples (as analysed in Chapter 2) tend to be considerably simpler to analyse than real samples, so in order to test the validity of the methods developed, DSTO provided realistic HME samples for analysis. These samples were used as they have been prepared in a manner directly analogous to HME samples commonly encountered in a real world scenario. Analysis of these genuine samples is covered in Chapter 3. The analysis of genuine samples highlighted a number of problems with the interpretation of results obtained from a single measurement of the bulk HME sample. These included: contamination, sampling issues, storage issues, dual carbon sources and dual nitrogen sources. The process used to concentrate and purify AN from calcium ammonium nitrate (CAN) also proved to be an important factor for the analysis of AN-based HMEs The results obtained in Chapter 3 highlights the usefulness of IRMS and ICP-MS for batch-to-batch matching of HME, but indicated that analysis of bulk sample is not sufficient for determining sourcing information and has limited intelligence value.

In Chapter 4 a new technique is described which mitigates the problems determined in Chapter 3. This technique is based on the separation and analysis of the nitrogen sources in AN, namely nitrate ion and ammonium ion. The isotopic ratio of the nitrogen in the nitrate ion was shown to be unchanged regardless of the purification process used, thus is an important marker for determining sourcing relationships. This Chapter described the separation technique and uses IRMS to determine the provenance of AN and CAN based explosives to their source/precursor materials.

Chapter 5 discusses the problem associated with dual carbon sources commonly encountered in fuel-oxidiser-based HME. It details a new method for gaining forensic intelligence from the exploitation of HME comprising CAN/sugar compositions.

Chapter 6 details techniques and methodologies for the analysis of non-explosive components occasionally found in HME compositions. The non-explosive component considered in this examination is glitter. Glitter is often found in paint grade aluminium flake to introduce various lustre effects, however, if this paint grade aluminium flake is used in the fabrication of HME, such as AN/aluminium, then those glitter particles become a

useful marker. The different types of analysis are detailed in this Chapter and the usefulness of this extra layer of information for linking HME samples is demonstrated.

The chemical analysis of a number of organic-based HME is detailed in Chapter 7. A series of experiments illustrating that both IRMS and ICP-MS can be utilised to extract information from samples of organic-based HME. This information can be used for batch-to-batch matching of samples but also to determine the origin of source materials.

The conclusions and recommendations from this research task are detailed in Chapter 8 of this thesis. It describes two new analytical methodologies for the analysis of HME samples using IRMS. These methodologies improve the confidence of source matching, which is important for of the provision of chemical intelligence. These techniques highlight a need to change from the bulk analysis of CAN-based HME to the separate analysis of each individual component (carbonate/nitrate) by IRMS. This new methodology has shown potential to be implemented as a way to determine the origin of the CAN used in the preparation of CAN-based HME.

The research described in this thesis has sort to highlight the use of IRMS and ICP-MS for the provision of chemical intelligence in the analysis of HME. By understanding the limitations of bulk analysis and how various processes affect the isotope ratios, or the introduction of trace impurities, it is now possible to link like samples and identify their source materials. The use of the analytical techniques described in this thesis may now be used as an additional layer of information in the general intelligence picture, which when combined with other intelligence collection methods may allow for “attack the network” operations.

Declaration

'I certify that this thesis does not incorporate without acknowledgment any material previously submitted for a degree or a diploma in any university; and that to the best of my knowledge and belief it does not contain any material previously published or written by another person where due reference is made in the text.'

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