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Short Communications

Development of a methodological framework for applying isotope ratio mass spectrometry to explosive components

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ABSTRACT

Isotope ratio mass spectrometry (IRMS) techniques have been used for decades to characterize explosives alongside traditional chemical analyses. We present here a broadly applicable framework for methodologically applying a variety of separation techniques to explosive mixtures and preparing different explosive components for stable isotope analysis. This framework allows an analyst to collect the most characteristic information possible from an explosive sample, by analyzing multiple components with independent isotope signatures to produce multivariate datasets for discrimination. A case illustration demonstrating the application of methods in the framework to plastic explosives (specifically C-4 and Semtex) has been prepared separately.

In developing this framework, we focused on explosives samples that may contain RDX, HMX, PETN, TNT, AN, and/or nitrocellulose along with various binders, plasticizers, oils, and other additives. This paper describes the theory and processes used to develop a component-specific approach to prepare explosives samples for isotope ratio analysis, focusing specifically on optimization of solvent extraction methods. Other methods used in framework development include gravimetric analysis and HPLC.

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1. Introduction

Following an explosive attack or thwarted bombing attempt, investigators are concerned with determining the responsible party (or parties). Characteristics of the explosive may provide clues about those who were responsible, whether they were involved in other events, and the source of materials they used. Chemical analyses play a central role in these investigations, supplying the tools to characterize explosives and their residues [1–3].

Most analyses focus on describing chemical features of the explosive. For example, plastic explosives can be characterized based on moisture content, acidity, explosive content (e.g., RDX, HMX), and non-explosive component identity and quantity (e.g., plasticizer content). Techniques used to measure these characteristics include chromatography, mass spectrometry, and spectroscopy [4,5]; spectroscopic techniques such as FTIR have also proven useful to identify non-explosive components [6,7]. Within the last decade, mass spectrometry has been further used to characterize oils found in the non-explosive portion of an explosive [8].

Isotope ratio mass spectrometry (IRMS) is a powerful analytical tool for characterizing explosives and discriminating samples, even

those having identical chemical formulations. Benson et al. [9] published the first review paper describing forensic applications of IRMS to explosive characterization. That review was followed by a recent publication by Gentile et al. [10], who described the use of IRMS for sourcing, including specific case examples on source attribution for explosives. Using these contributions as foundations, we can broadly summarize the approaches used to date in the application of IRMS techniques to the isotopic characterization of explosives.

Previously published methods using stable isotopes to characterize explosives can be categorized into one of three approaches. First and rudimentarily, the analyst can apply IRMS methods to a "bulk" explosive [11–13], thereby considering all components contained within together in a single measurement. Second, the analyst can focus on measurement of an explosive recovered as a pure material, which requires no preparation or cleanup [14–20]. Finally, the investigation can consider analysis of explosive components [5,21] that have been isolated for isotope ratio measurement – for example, RDX extracted from a plastic explosive. In this approach, any other components, such as binders or plasticizers, are not isolated or considered for isotope ratio analysis. Of these three approaches for applying IRMS techniques to explosives, we contend that the most explicit and reproducible approaches, such as component-specific analytical techniques, are preferable and

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most likely to be allowed as evidence in the judicial system [22]. In their recent review, Gentile and colleagues [10] suggested evaluation of compound-specific isotope analysis (CSIA) for explosive source attribution.

In this short communication, we introduce a methodological framework for preparing different explosive components for stable isotope analysis. The methods included in the framework allow analysts to isolate and then characterize a variety of components using chemical and isotopic analyses. We first describe the overall concept of the process and then describe the steps taken to optimize one method used in the framework, namely solvent extraction of explosive materials. Finally, we present the framework in a graphical format. An example application of the framework to plastic explosives—including component isolation and subsequent stable isotope analysis for discrimination purposes—has been prepared as a separate manuscript [23].

2. Considerations for component-specific separation and analysis

An explosive is a chemical mixture whose purpose is to shatter or destroy surrounding objects by a rapid expansion of gases. Common explosives include RDX (cyclotrimethylene trinitramine), HMX (cyclotetramethylene tetranitramine), PETN (pentaerythritol tetranitrate), TNT (2,4,6-trinitrotoluene), ammonium nitrate (AN), and nitrocellulose. Explosive mixtures often contain these components in various combinations, in addition to small quantities of other components. Binders can be added to bind components together and decrease the sensitivity of the explosives. Two common binders found in plastic explosives include styrene-butadiene rubber (SBR) and polyisobutylene (PIB). The addition of plasticizers and oils such as *bis*(2-ethylhexyl) adipate (BEHA), *bis*(2-ethylhexl) sebacate (BEHS), diisobutyl phthalate (DIBP), and tributyl citrate can make an explosive easier to mold and safer to handle. Oil can act as a release agent while additives such as metal or wood can increase blast effects. In some cases, explosives may also contain very small amounts of antioxidants, dyes, and/or taggants.

A key consideration in explosive characterization using chemical and isotopic analyses is the careful separation of different components contained within the mixture. This is important for accurate quantification of the constituent components. It is also crucially important for component-specific stable isotope analysis as splitting a component into multiple pools or a loss in yield may be associated with isotopic fractionation effects.

A schematic of the components contained within explosive mixtures is presented in Fig. 1. The schematic considers a variety of components: explosive material(s), binders, plasticizers, oils, and other additives such as dyes, metal, wood, etc. In developing a methodological framework for separating these components for isotopic analysis, we considered mixtures that may contain the explosive materials RDX, HMX, PETN, TNT, AN, and/or nitrocellulose.

3. Materials and equipment used in framework development

We used a variety of explosive materials during methods development, procured as follows. A single sample each of RDX (identifier 522), HMX (identifier 291), PETN (identifier 544), and AN (identifier SAN) came from materials previously investigated by us [5,18,21]. A single sample of TNT (identifier 268) was provided by the U.S. Government. Nitrocellulose was made in-house by nitrating a cellulose (laboratory) reference material [24]. Standard concentration solutions of RDX, HMX, PETN, and TNT for HPLC/UV
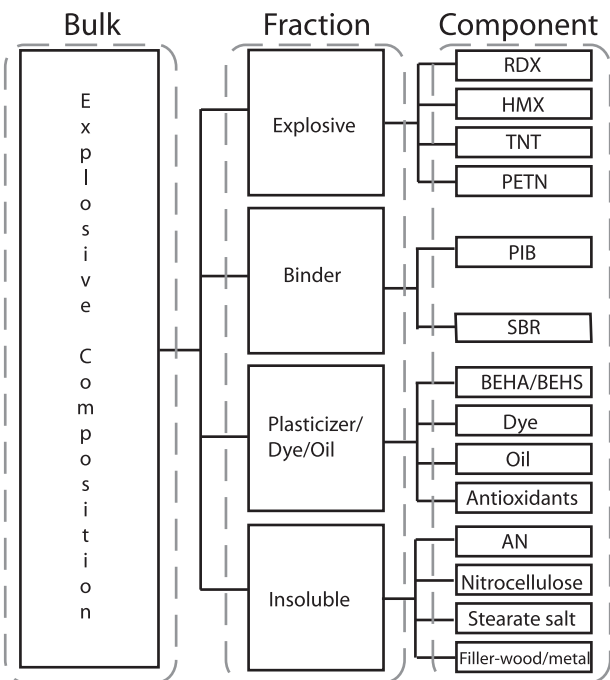

Fig. 1. A schematic representation of the breakdown of the bulk explosive into components contained within explosive mixtures, which include explosive material (s), binders, plasticizers, oils, and other additives.

analysis were purchased from AccuStandard, Inc. (New Haven, CT, USA) and prepared as described by Howa et al. [5].

Non-explosive materials used in method development included the polymeric binders SBR and PIB; plasticizers BEHA, BEHS, and DIBP; mineral oil; an antioxidant (N-phenyl-2-naphthylamine); and Sudan I dye. The mineral oil was purchased from Malinckrodt. All other non-explosive materials were purchased from Sigma–Aldrich.

Solvents were Optima-grade and included acetone, chloroform, cyclohexane, hexanes, and methanol; all solvents were purchased from Fisher Scientific. Samples were dried under a stream of purified air at room temperature using a Pierce evaporator. HPLC/UV analyses were completed using a Waters HPLC system as described by Howa et al. [5]. Gravimetric analysis was conducted in 1-dram vials (mass ∼4.5 g) using a Sartorius MC-5 microbalance (Bradford, Massachusetts, USA).

4. Methodological framework development and results

4.1. Solvent selection for initial extraction

A variety of analytical methods have been published for the chemical characterization of explosive mixtures and most begin with an extraction to separate binders, plasticizers, and oils from the explosive(s), salts, and any fillers. The solvent most commonly used for initial extraction is chloroform, followed by an acetone extraction of both fractions [4,6,7,25]. For subsequent spectroscopic and spectrometric examination, explosive material is typically collected from the chloroform-insoluble, acetone-soluble fraction while binders are collected from the chloroform-soluble, acetone-insoluble fraction; plasticizers and oils are collected from the chloroform-soluble, acetone-soluble fraction. However, previous studies have noted the presence of explosives in the spectra of binders and plasticizers (e.g., [7]), suggesting that initial chloroform extraction partially extracts explosives as well, leading to incomplete separation.

To select the solvent best suited for initial sample extraction in a component-specific isotope analysis framework, we compared the amount of explosives RDX, HMX, and PETN extracted in chloroform to that extracted in cyclohexane. Cyclohexane is both less toxic than chloroform and less likely to dissolve these materials, making it potentially a better choice for initial extraction.

We note that earlier applications of this component separation framework to RDX/HMX and PETN explosives used hexanes for initial sample extraction [5,21]. In the current work we investigated the use of cyclohexane instead of hexanes. C-4 typically contains polyisobutylene (PIB) as a binder, while Semtex contains styrene-butadiene rubber (SBR). Ideally, the same extraction method should effectively separate binders from the explosive materials (i.e., RDX, HMX, and PETN) in both of these plastic explosives; however, in our experience (Supplemental Table) as well as previous publications [26] the binder in Semtex did not dissolve completely in hexanes, leading us to investigate cyclohexanes as an alternative.

To investigate the extraction of different explosives into chloroform and cyclohexane, pure explosive material was mixed with 2 mL of chloroform or cyclohexane and left overnight (8–12 h). Initial explosive mass was approximately 20 mg for RDX, HMX, and PETN (Table 1). AN, TNT, and nitrocellulose were extracted with cyclohexanes only, as TNT is known to be soluble in chloroform [27], whereas AN and nitrocellulose are not. Following overnight extraction, the solvent fraction was collected and insoluble material extracted a second time with 2 mL solvent for 1 h. Solvent fractions were combined and dried. The ratio of the extracted mass and the initial mass was used to calculate the percent of original material extracted (Table 1).

We found that significant amounts of RDX and PETN were extracted into chloroform, with up to 5% of RDX and up to 23% of the original PETN extracted. This suggests CHCl₃ is not the ideal solvent for extracting binders, plasticizers, oils, and other non-explosive materials from these explosive materials as it would concurrently extract some explosive material as well. In contrast, RDX,

HMX, and PETN—as well as AN and nitrocellulose—were not extracted into cyclohexane (i.e., <1% extracted), although TNT was soluble in cyclohexane (Table 1).

4.2. Back-extraction of TNT

As a consequence of TNT's solubility in non-polar solvents, we next investigated a method for recovering TNT from the cyclohexane-soluble fraction. For this, we extracted the dry cyclohexane fraction containing TNT by agitating 5 s with 0.5 mL of methanol. The methanol fraction was then added to the TNT remaining in the cyclohexane-insoluble fraction, dried, and weighed. Results in Table 1 demonstrate that this "quick rinse" back-extraction of TNT with methanol was sufficient to collect TNT that would otherwise remain in the cyclohexane-soluble fraction.

We note that back-extraction of TNT with methanol does have the potential to co-extract some non-explosive components, such as plasticizers, some additives, and short-chain hydrocarbons. In practice, we rarely observe TNT with the plasticizers and additives here described. In addition, long-chain hydrocarbons that remain in the cyclohexane-soluble fraction after the "quick rinse" procedure are often more useful for compound-specific isotope ratio analysis than other components that may be co-extracted by the methanol.

4.3. Extraction of explosive components

Following initial sample extraction with cyclohexane (and methanol back extraction, as needed, for TNT), the cyclohexane-insoluble fraction may include a variety of explosives, and may also include stearate salts or additives such as metal or wood. Acetone extraction [5,21] of the cyclohexane-insoluble fraction will separate most organic explosives (RDX, HMX, PETN, and TNT) from the insoluble material, which are then ready for HPLC preparation,

Table 1
Solubility test of some common components of explosives in chloroform and/or cyclohexane. Recovery rates are calculated from one measure in most tests; where tests were repeated, duplicate results are averaged and presented with a standard deviation (sd). Extraction % was calculated using the total extracted mass (i.e., amount soluble) versus the initial mass.

Component	Alt. name	Purpose	Chloroform			Cyclohexane			
			Initial mass (mg)	Extracted mass (mg)	% Ext.	Initial mass (mg)	Extracted mass (mg)	% Ext.	sd (%)
RDX	–	Explosive	20.92	1.00	4.8	21.31	0.14	0.7	n/a
RDX	–	Explosive	21.38	0.96	4.5	21.24	0.11	0.5	n/a
PETN	–	Explosive	20.50	4.74	23.1	20.63	0.16	0.8	n/a
PETN	–	Explosive	23.27	4.87	20.9	20.85	0.14	0.7	n/a
HMX	–	Explosive	20.79	0.25	1.2	22.26	0.15	0.7	n/a
HMX	–	Explosive	21.75	0.13	0.6	20.84	0.10	0.5	n/a
TNT	–	Explosive	n/a	n/a	n/a	19.16	2.92	15.2	0.2
TNT	–	Explosive	n/a	n/a	n/a	19.70	3.00	15.2	0.2
TNT + methanol extraction	–	Explosive	n/a	n/a	n/a	19.16	0.22	1.2	0.2
TNT + methanol extraction	–	Explosive	n/a	n/a	n/a	19.70	0.22	1.1	0.2
AN	–	Explosive	n/a	n/a	n/a	18.36	−0.02	−0.1	0.2
AN	–	Explosive	n/a	n/a	n/a	19.73	−0.04	−0.2	0.2
Nitrocellulose	–	Explosive	n/a	n/a	n/a	18.88	−0.02	−0.1	0.2
Nitrocellulose	–	Explosive	n/a	n/a	n/a	21.86	0.00	0.0	0.2
bis(2-ethylhexyl) adipate	BEHA	Plasticizer	n/a	n/a	n/a	1.92	1.93	100.8	2.9
bis(2-ethylhexyl) sebacate	BEHS	Plasticizer	n/a	n/a	n/a	1.76	1.73	98.6	3.1
Diisobutyl phthalate	DIBP	Plasticizer	n/a	n/a	n/a	1.75	1.76	100.3	3.2
Tetraethyl citrate	Citroflex 2	Plasticizer	n/a	n/a	n/a	1.84	1.78	96.7	2.9
Mineral oil	MOil	Release agent/ process oil	n/a	n/a	n/a	1.78	1.72	96.2	3.0
Polyisobutylene	PIB	Binder	n/a	n/a	n/a	0.83	0.83	100.5	6.6
Styrene-butadiene rubber	SBR	Binder	n/a	n/a	n/a	4.56	4.15	91.2	1.2
2-Naphthylphenylamine	2NPA	Antioxidant	n/a	n/a	n/a	0.51	0.46	90.2	10.2
Sudan I	–	Dye	n/a	n/a	n/a	0.32	0.25	77.9	15.9
Sudan IV	–	Dye	n/a	n/a	n/a	0.34	0.31	91.3	15.4

analysis, isolation, and subsequent measurement of hydrogen, carbon, nitrogen, and/or oxygen isotope ratios via IRMS.

Before HPLC analysis, however, any nitrocellulose that is present must be removed from the cyclohexane-insoluble, acetone-soluble fraction. This can be done by replacing acetone with dichloromethane (DCM) during extraction of the cyclohexane-insoluble fraction [21]. This replacement is recommended when working with explosives that are known to contain nitrocellulose, such as sheet explosives. For homemade explosives that may contain a variety of explosive materials, it is possible that nitrocellulose will not be identified until attempting to filter the cyclohexane-insoluble, acetone-soluble fraction. In this case, the filter used to protect the HPLC instrument from microscopic particles will clog due to the viscosity of the nitrocellulose in solution. The extraction must be redone from the start, substituting DCM for acetone to extract the RDX, HMX, PETN, and TNT from the cyclohexane-insoluble fraction. The remaining nitrocellulose, which is insoluble in DCM [28], can then be extracted from the cyclohexane-insoluble, DCM-insoluble fraction using acetone.

4.4. Extraction of water-soluble salts

Considering the cyclohexane-insoluble, acetone-insoluble fraction, water can be used to extract any AN and other soluble salts
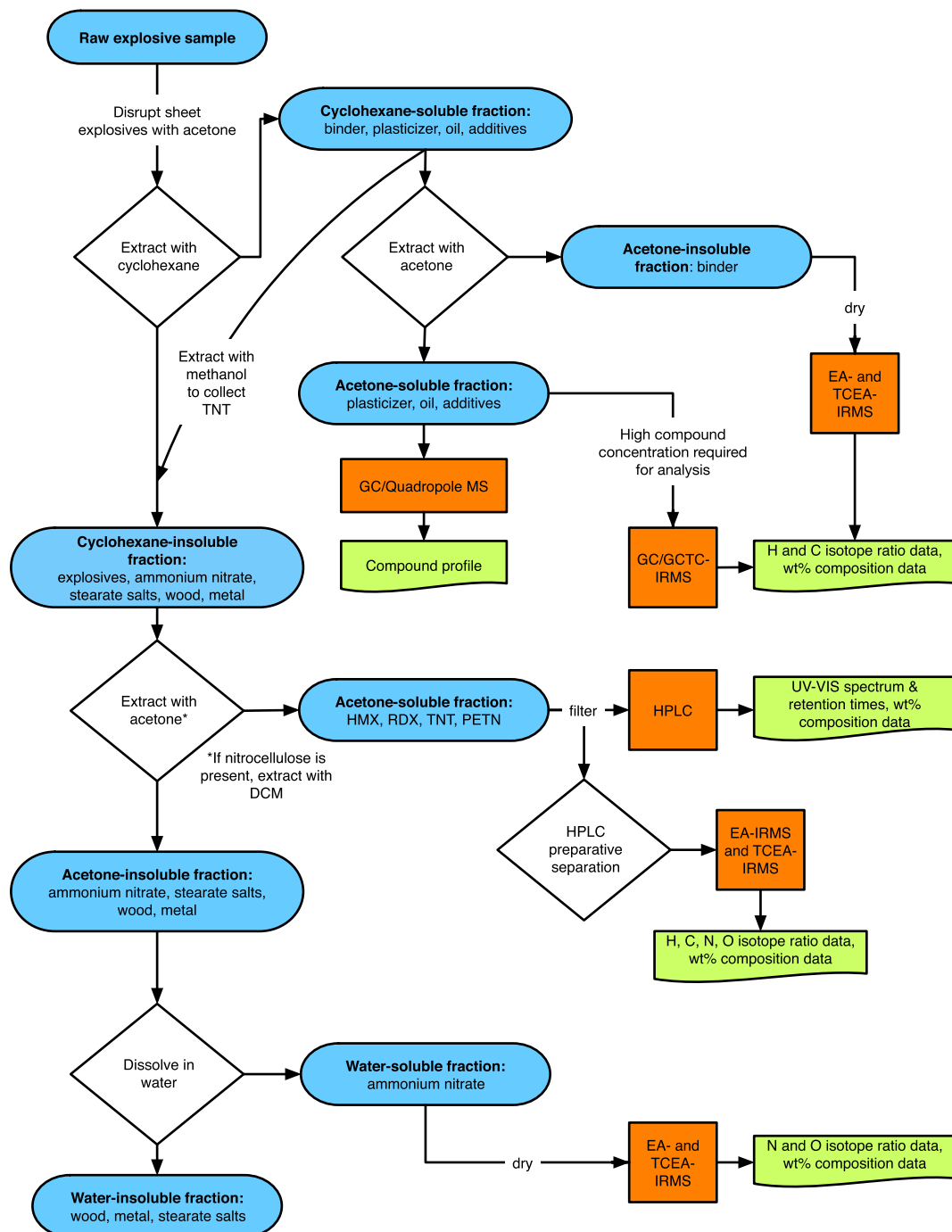

Fig. 2. A framework for separating, isolating, and preparing a variety of components from an explosive mixture. Components may be analyzed using a combination of gravimetric, HPLC, GC–MS, and isotope ratio mass spectrometry techniques.

from water-insoluble additives such as stearate salts, metal or wood. (We note that this water washing step will change the $^2H/^1H$ signal of AN.) Once collected and dried, AN contained in the raw explosive sample would be ready for measurement of nitrogen and oxygen isotope ratios via IRMS. If desired, insoluble material such as stearate salts, metal, or wood can be chemically and/or isotopically analyzed for element composition and/or isotope ratios, though it is difficult to confirm the identity of these insoluble materials without additional analytical techniques.

4.5. Separation of binders, plasticizers, oils, and other additives

We also tested the solubility of several non-explosive components—including binders, plasticizers, oil, an antioxidant, and dye—in cyclohexane (Table 1). We found the extraction efficiency of the technique on plasticizers, oils, PIB, and Sudan IV to be near 100%; only SBR had a significantly lower recovery (93.5%). Sudan I showed the lowest absolute recovery of all the components tested (77.9%), though this result was not significant due to the large relative error for gravimetric measurements at this size. This additionally confirmed cyclohexane as a good alternative to chloroform for initial explosive extraction.

Acetone can be used to isolate binders from the plasticizers, oils, antioxidants, and/or dyes that may be present [7]. Once dried, the polymeric binder component contained in the cyclohexane-soluble, acetone-insoluble fraction can be analyzed for elemental composition and isotope ratios. Most binders such as PIB and SBR are hydrocarbons, and will only contain hydrogen and carbon in quantity enough for isotope ratio analysis. The cyclohexane-soluble, acetone-soluble fraction can be analyzed using gas chromatography-mass spectrometry (GC–MS) to identify and quantify the components present. Separation of oils from plasticizers, dyes, and other more complex components is possible using silica gel column chromatography or sep-packs [8,26] if isolation of plasticizers from complex organic mixtures is required prior to gas chromatography. If compound-specific isotope ratio analysis of individual hydrocarbons is desired over bulk analysis of complex oils, further isolation of n-alkanes is possible with urea adduction followed by dissolution in water and extraction into pentane [29,30].

5. Discussion

In this work we developed a methodological framework for separating a variety of components from an explosive mixture, which is shown in graphical format in Fig. 2. This general approach has the capacity to be applied to multiple explosive formulations and to extract and prepare multiple components for IRMS. Using the framework, it is possible to measure, free of interference, isotope ratios of a variety of explosive materials—including RDX, HMX, PETN, TNT, AN, and/or nitrocellulose—as well as common non-explosive minor components such as binders, plasticizers, oils, and other additives present in a mixture. A detailed case example on the application of this framework to two plastic explosives—namely, Composition C-4 and Semtex—has been prepared as a separate publication [23].

Applying this framework to an explosive sample will generate multivariate datasets, especially when the components are analyzed for multiple isotope ratio parameters [23]. For this discussion, these parameters may include hydrogen, carbon, nitrogen, and/or oxygen isotope ratios as well as the quantification of various components. As an example, consider the analyses possible for each component of a plastic explosive, outlined in Table 2. The list illustrates the maximum number of variables (e.g., wt% compositions, isotope ratios) it is possible to measure in a single sample of Semtex H or Composition C-4.

Chemical analyses of the isolated components—such as MS, UV–Vis, and FTIR—will generate compositional data that complements measured stable isotope ratios. To track and link these data, we recommend the use of relational databases for data storage. The use of databases for data organization as well as data storage follows key recommendations by Steele [4] and Gentile et al. [10] to create searchable repositories of data that facilitate sample comparison and examination of relationships between variables. These collections make it easier for laboratories to share and compare data. Databases can also be a fundamental feature of data quality control management plans, allowing review and identification of long-term data trends [31]. Populating these databases increases the probability of being able to correctly attribute an unknown sample to a known source or discriminate materials from different sources.

Table 2

Multi-component analyses possible for two types of plastic explosives (Semtex H and Composition C-4). Assumed or military specifications for wt% composition are provided alongside ranges observed during previous analyses of samples labeled as "Semtex H" and "C-4". All listed elements are present within a component and could be measured for isotopic composition. These isotope ratio measurements alongside a wt% measurement sum to a total number of variables available per component to characterize and discriminate explosive samples.

Fraction	Component	Assumed %	Observed %	Elements	Variables
Semtex H					
Explosive	RDX	41.55	10–58	H, C, N, O	5
Explosive	PETN	41.55	21–82	H, C, N, O	5
Binder	Styrene-butadiene rubber (SBR)	9.0	4	H, C	3
Oil	"Fuel oil" [32]	6.9	11–12	H, C, O	4
Antioxidant	N-phenyl-2-naphthylamine	0.5	<1	H, C, N	4
Dye	Sudan I	0.5	<1	H, C, N, O	5
		Total independent variables			25
Fraction	Component	MIL-SPEC %	Observed %	Isotope	Variables
Composition C-4					
Explosive	RDX	81.8	60–93	H, C, N, O	5
Explosive	HMX	8.2	0–11	H, C, N, O	5
Binder	Polyisobutylene (PIB)	2.6	2–4	H, C	3
Plasticizer	*bis*(2-ethylhexyl) adipate (BEHA), *bis*(2-ethylhexl) sebacate (BEHS)	5.6	2–8	H, C, O	4
Oil	"Process oil"	1.8	0–4	H, C	3
		Total independent variables			19

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.forc.2016.08.003.

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