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# Evaluation of preliminary isotopic analysis ($^{13}C$ and $^{15}N$) of explosives A likelihood ratio approach to assess the links between semtex samples☆

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**Abstract**

Currently, the use of isotopic ratio as corroborative evidence in criminal trials is explored. Beyond the analytical challenges that have been reported elsewhere, the crucial issue of the interpretation of analytical results in a fair and balanced way remains poorly documented.

The aim of this paper is to propose a likelihood ratio approach for the evaluation of stable isotope data acquired from semtex samples. It will also lead to recommendations in relation to the acquisition of normalised international data.


*Keywords:* Forensic science; Explosive; Semtex; Carbon isotope ratio; Nitrogen isotope ratio; Evidence evaluation

## 1. Introduction

Isotope ratio mass spectrometry is a method in mass spectrometry able to provide measurements of the ratios of abundances of stable isotopes [1]. More recently, on-line or isotope ratio monitoring offered an easier experimental approach in many discipline, particularly in forensic sciences. First of all, carbon is the most popular stable isotope analysed, but as already shown with illicit drugs this parameter is not discriminatory enough. For this reason, usually more that one isotope is analysed (e.g. carbon, nitrogen). The method is rapidly expanding to deuterium and oxygen, so hydrogen and oxygen should be acknowledged as well.

It is worth knowing that in explosives carbon mainly comes from the explosive (60% from explosives, 10% from polymeric matrix and 30% from fatty hydrocarbons). As regards nitrogen, 99% come from the explosive through the manufacturing process, resulting in part from precursor of the explosive and in part from the compound used in the nitration process. One percent is held for dye.

Depending on the manufacturing process, the explosive acquires a specific isotope ratio signature due to fractionation processes.

In the United Kingdom, since 1999, the Forensic Explosives Laboratory (FEL) has investigated the use of stable isotope ratios in the forensic analysis of bulk explosives. In the present study, both carbon and nitrogen analyses have been carried out, generating multivariate data.

We propose the evaluation of this data using a likelihood ratio framework that has received increasing attention in the past 15 years in all areas of forensic science [2]. The main advantage of the approach is that it allows the scientist to assess the respective likelihood of the data under a competing set of propositions. That mechanism allows a balanced assessment of the contribution of the findings in the criminal proceeding.

Recently, Aitken and Lucy [3] have described methods for interpreting sets of multivariate measurements involving glass

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fragments and published software routines written in "R" [4]. The programs are available on a specific website [5].

It is proposed to apply two of their methods to the data from semtex H samples and examine the advantage of a third more intuitive method of our own that will be compared with the two published proposals.

All these methods can be used in evaluative cases.

## 2. Materials and analytical methods

### 2.1. Samples

The measurements have been carried out on 26 random samples from a semtex H collection of 500 samples.

The plastic explosive semtex H is a complex compound broadly composed of a plasticizer (about 20% of polymeric matrix and fatty hydrocarbons), about 80% of PETN and RDX in various ratios and a dye.

### 2.2. Samples preparation

About 300 μg of semtex was removed from the bulk and placed into a tin capsule. Six tin capsules (replicates) were prepared for a dual carbon/nitrogen isotopic analysis of the semtex.

### 2.3. Elemental analyser—isotope-ratio mass spectrometer

Dual isotope-ratio measurements (C, N) were performed using a Delta Plus XP isotope-ratio mass spectrometer (Thermo Finnigan) on line coupled to an elemental analyser (Costech ECS 4010) with a ConFlo III Interface. Helium was used as carrier gas with rate of 100 ml/min. The combustion furnace consisted of a quartz tube filled with chromium oxide catalyst and silvered colbatous/cobaltic oxide. Oxidations are performed at 1030 °C. Nitrogen oxides were then reduced to $N_2$ in a reduction furnace, a quartz tube packed with copper wires reduced, set at 650 °C. Water was removed using a magnesium perchlorate trap.

Before entering the IRMS, gases pass through a short GC-column (3–4 m) with the aim of separating the $CO_2$ and $N_2$ peaks. The $N_2$ working standard (urea, $\delta\ ^{15}N = -1.3‰$) was carefully calibrated using the appropriate reference (IAEA-N1, ammonium sulphate, $\delta\ ^{15}N = 0.4‰$), in addition as the $CO_2$ working standard (Tate & Lyle sugar $\delta\ ^{13}C = -11.7‰$) was calibrated against reference (IAEA-CH-7, polyethylene, $\delta\ ^{13}C = -31.8‰$). Isotope ratios are expressed as $\delta$ values (in per thousand, ‰) versus international standard. Data were processed using ISODAT NT software.

Notice that the presence of 12 oxygen atoms in PETN and six oxygen atoms in RDX could suggest that the determination of $\delta\ ^{18}O$ ratio may also be of interest. This aspect will be the subject of further research and is outside the scope of this paper.

## 3. Analytical results

The stable isotope ratios $\delta\ ^{13}C$ and $\delta\ ^{15}N$ values from the 26 samples measured by EA-IRMS are shown in Fig. 1.

The values obtained varied between −24.76 and −36.53‰ for carbon and between −2.72 and −25.68‰ for nitrogen.

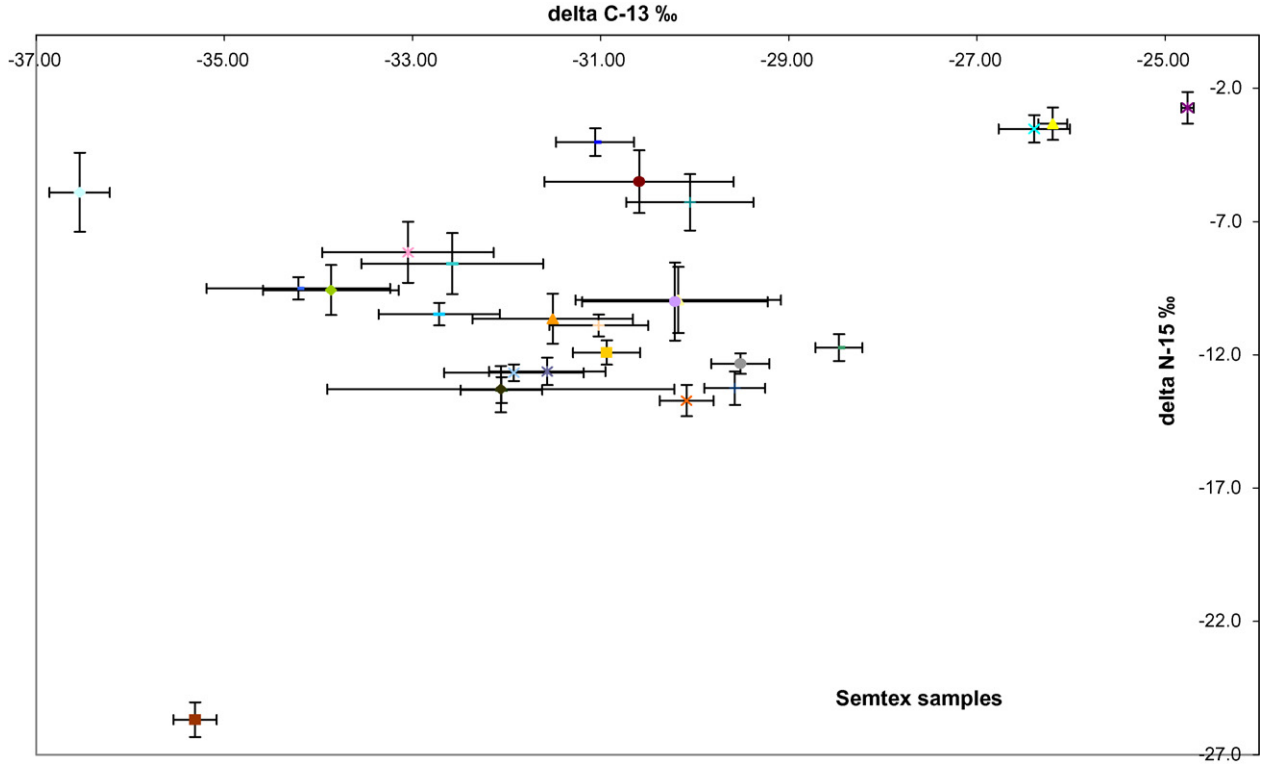


Fig. 1. Carbon and nitrogen isotope ratios of 26 semtex (1 s.d.).