



## Results of four inter-laboratory comparisons provided by the Forensic Isotope Ratio Mass Spectrometry (FIRMS) network

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### ABSTRACT

Between 2004 and 2008 the Forensic Isotope Ratio Mass Spectrometry (FIRMS) network organised four Inter-Laboratory Comparison exercises with the aim of harmonising and validating isotopic measurements to be used for forensic applications.

The samples distributed comprised materials of potential forensic interest such as packaging and pharmaceuticals to be analysed for  $\delta^2\text{H}$ ,  $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$  and  $\delta^{18}\text{O}$  composition. As many as 30 international laboratories participated in these comparisons. Results have shown overall improvements for both within- and inter-laboratory reproducibility with respect to  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  measurements.

Only a small number of laboratories returned results for  $\delta^2\text{H}$  and  $\delta^{18}\text{O}$  analysis and these results highlighted a need for improvement in the reproducibility of these measurements and a need to address the exchange of hydrogen between samples and ambient moisture. Results also highlighted the importance of sample preparation procedures and the need to standardise both these procedures and calibration against Standard Reference Materials. Future Inter-Laboratory Comparison exercises will assess the suitability of laboratories to submit data to national and international databases similar to those currently operated for fingerprints, DNA etc.

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### 1. Introduction

Alfred Nier first developed dual inlet Isotope Ratio Mass Spectrometry (IRMS) in 1947 as a technique for the measurement of the isotopic ratio of gaseous elements [1]. Developments of the technique led to on-line measurement of the isotopic ratios of elements present in solid and liquid samples, using elemental analysers to convert them into gases such as  $\text{CO}_2$ , and  $\text{N}_2$  [2,3]. Early applications of the technique were established in the fields of geochemistry and hydrology, with vast amounts of data being produced in these areas [4]. The unique ability to provide information relating to origin, timescales, climate and behaviour, explains why isotopic analysis is particularly suited to forensic investigations. Forensic science spans many disciplines and the growth of isotopic analysis is mirrored by the number of publications in this field [5]. The ability, for example, to demonstrate that two or more samples are different, or potentially share a common source contributes to the evidence that forms part of a criminal investigation. Aided by background databases, isotopic analysis may contribute to intelligence gathering, search refinement or focused enquiries. One important potential application for a global database is to combat illegal immigration, and people trafficking, through so called human provenancing i.e. the determination of the geographical origin of an individual based on isotopic characterisation of materials such as nail or

hair [6]. A prerequisite for any such database must be to establish adequate inter-laboratory reproducibility of contributing laboratories.

The Forensic Isotope Ratio Mass Spectrometry (FIRMS) network was established as an Environmental and Physical Sciences Research Council (EPSRC) funded network on the 1st January 2002. In September of the same year the network hosted an international meeting at Brands Hatch, Kent, UK to promote forensic applications of the technique. Another objective of the network was to promote and harmonise good practice in the acquisition and use of isotope abundance data. To this end members were encouraged to seek accreditation to appropriate standards, specifically ISO/IEC 17025:2005 for which the United Kingdom Accreditation Service (UKAS) is the national signatory within the UK. This standard requires laboratories to have quality control procedures for monitoring the validity of tests and recommends participation in inter laboratory comparisons (ILC) [7].

Within the UK, the National Measurement System (NMS) is responsible for stimulating good measurement practice by promoting the six principles of Valid Analytical Measurement (VAM). Of these six principles, the fourth states that “there should be a regular independent assessment of the technical performance of a laboratory” and the fifth that “analytical measurements made in one location should be consistent with those elsewhere” [8]. Again, this is taken as recommendation that laboratories participate in ILCs.

The objective of the ILCs described in this paper was to distribute samples of forensic interest to participating laboratories aiming to promote adoption of the VAM principles and to encourage and assist

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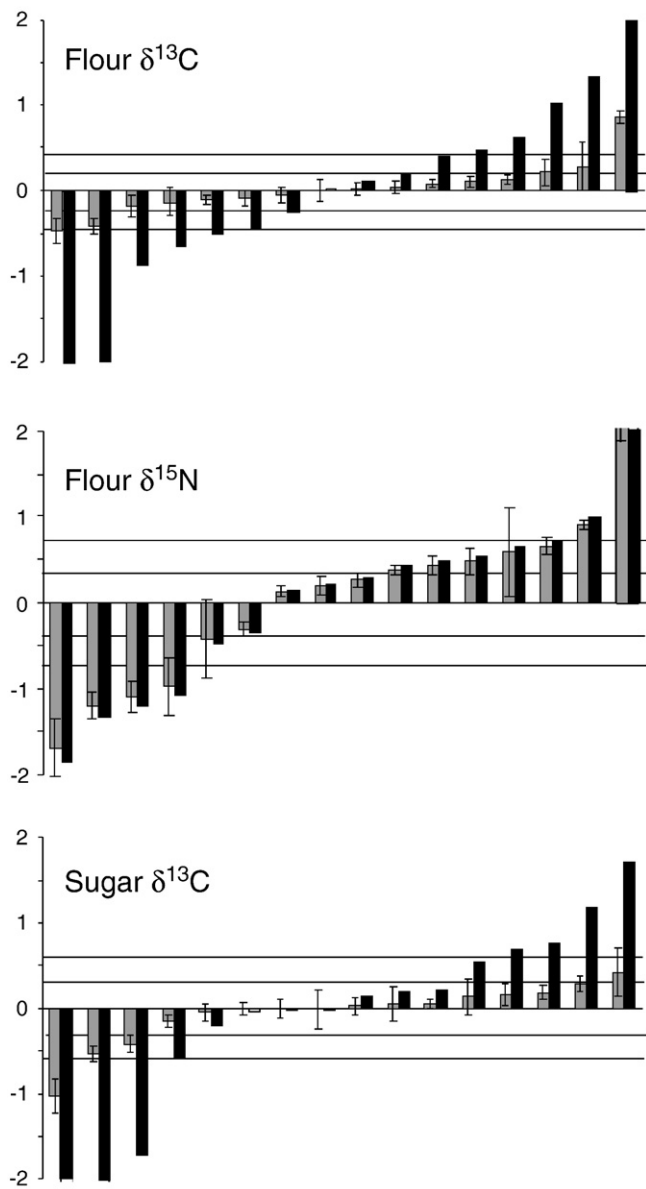


Fig. 1. Results for ILC#1 (2004). Grey bars show the difference from H15 mean with  $\pm 1$  SD error bars (permil), black bars show z-score (dimensionless units).

accreditation to the above standard. Although the samples distributed for ILC#1 comprised food stuffs, flour and sugar, it is ironic that these materials have now attracted forensic interest as components of improvised explosive devices [9]. Other materials were considered to be of potential forensic interest, for example as cutting agents or packaging for illicit drugs.

Unless otherwise acknowledged, the samples distributed were prepared by Mass Spec Analytical Ltd (MSA) (Filton, UK) and distributed by the Defence Science and Technology Laboratory (Dstl) (Sevenoaks, UK). Individual laboratory results were returned to Dstl, compiled and an anonymised version sent to MSA for processing before being returned to Dstl. Overall summary data were then distributed via the FIRMS Network newsletter.

## 2. Materials and methods

### 2.1. ILC#1 (2004)

Tate and Lyle “pure granulated cane sugar” (Batch 10401663) and Morrison’s “economy plain flour” were purchased from a local supplier

(Cheshire, UK) in 2000. Samples were transferred to clean, labelled vials with no further processing.

A total of 20 samples of each type were prepared and distributed.

### 2.2. ILC#2 (2005)

Phenacetin (*N*-(4-ethoxyphenyl)acetamide) was purchased from Sigma-Aldrich (Dorset, UK) (Lot S16829-214) with a certified purity by HPLC of 99.7%. Approximately 10 g of this material was ground to a fine powder using an agate mortar and pestle. Physical homogeneity of this sample was ascertained by examination under a stereo microscope (Brunel Microscopes, Chippenham, UK) at approximately 40 $\times$  magnification. Aliquots of the sample, approximately 200 mg, were weighed into clean, labelled vials. Ten vials were selected at random and analysed for both carbon and nitrogen isotopic composition. The standard deviations for these measurements were less than 0.1 and 0.2‰ (permil) respectively.

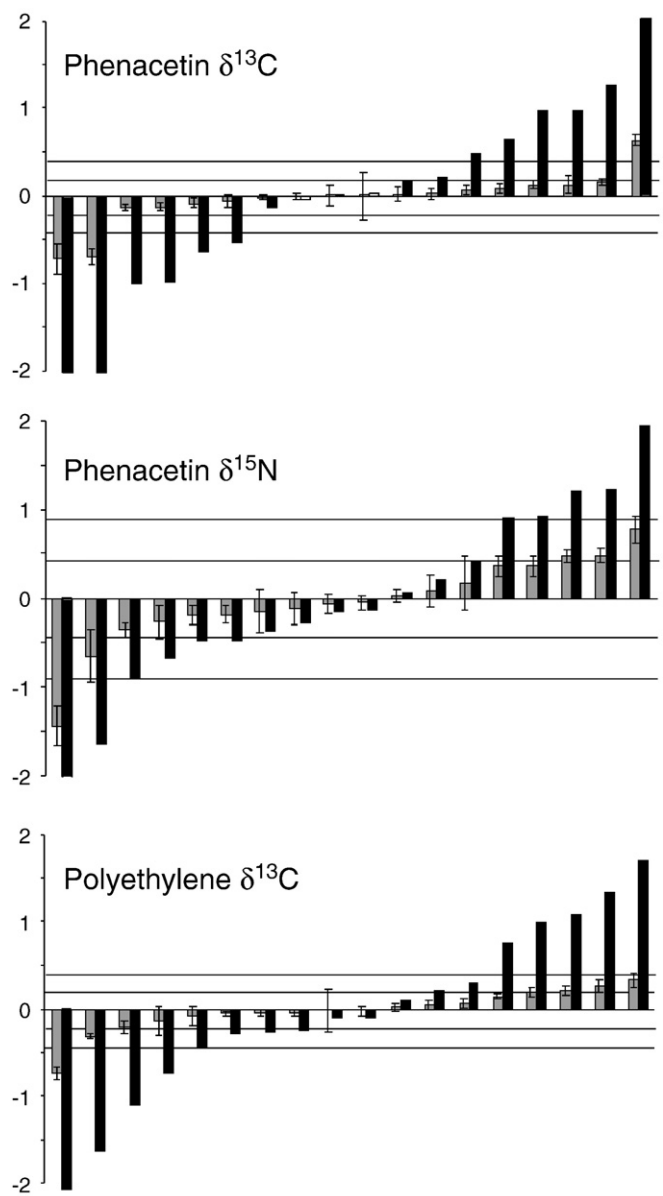


Fig. 2. Results carbon and nitrogen isotopic analyses for ILC#2 (2005). Grey bars show the difference from H15 mean with  $\pm 1$  SD error bars (permil), black bars show z-score (dimensionless units).

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