EA-IRMS: Unattended analysis of low µg to high mg sample sizes with SmartEA Option

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Keywords: CNSOH, Flash IRMS EA, Isotope analysis, SmartEA Option

Goal

Demonstrate how the Thermo Scientific SmartEA Option of the Thermo Scientific EA IsoLink IRMS System allows unattended analysis and ease-of-use delivering high precision results.

Introduction

It is common in Elemental Analysis Isotope Ratio Mass Spectrometry (EA-IRMS) to encounter sample types with a wide range of elemental concentrations of carbon (C), nitrogen (N), sulphur (S), oxygen (O), and hydrogen (H). This creates an analytical challenge for the analysis of samples with a diverse range of elemental concentrations. Laboratories require simultaneous CN isotope ratio measurements or simultaneous CNS isotope ratio measurements due to increasing demand for multi-isotope analysis, a desire to reduce analytical and labor costs and deliver strong return of investment. For example, this is true of samples with very high C/N ratios, such as plant roots, and high C/S ratios, such as bone collagen.

Thermo Scientific™ EA IsoLink™ IRMS System offers a solution allowing for dilution of the signal of the most highly concentrated elements, such that the mass spectrometer detectors do not become saturated, yet the very low elemental concentrations are analyzed with sufficient signal-to-noise ratio to produce reliable results. Consequently, this allows simultaneous CN measurements and simultaneous CNS isotope ratio measurements from the sample. This is achieved through flexible automated dilution provided by the Thermo Scientific™ SmartEA™ Option.

Instrument setup

EA IsoLink IRMS System includes the Thermo Scientific™ Flash IRMS™ Elemental Analyzer (EA), the Thermo Scientific™ ConFlo IV™ Universal Interface and a Thermo Scientific™ Isotope Ratio Mass Spectrometer (IRMS). The SmartEA Option of the EA IsoLink IRMS System builds a signal calibration between the signal produced on the Thermal Conductivity Detector (TCD) of the Flash IRMS EA and the signal produced by the IRMS. The straightforward and automated workflow allows the analysis of very small amounts, such as nitrogen and sulfur, alongside high abundant elements, such as carbon, from the same sample.

This approach is unique to the EA IsoLink IRMS System and protected by United States of America (US8402814B2), European (EP2095868B1), Japanese (JP5153763B2) and Canadian (CA2647686C) patents.

The calibration of the TCD and IRMS signal is achieved by analyzing an appropriate standard material with balanced elemental composition (e.g. urea, sulfanilamide) across several different weights (Figure 1).

This analysis setup produces a linear relationship as shown in Figure 2. The linear relationship is stored in a software database and automatically applied to each subsequent sample analysis proceeding the calibration and is subsequently used to determine the required dilution of each sample gas signal. The dilution is achieved by the software automatically instructing the ConFlo IV Universal Interface to dilute the sample depending on the sample gas signal and its relationship to the linear relationship as exemplified in Figure 2. A re-calibration isn't necessary until/unless analytical conditions change significantly.

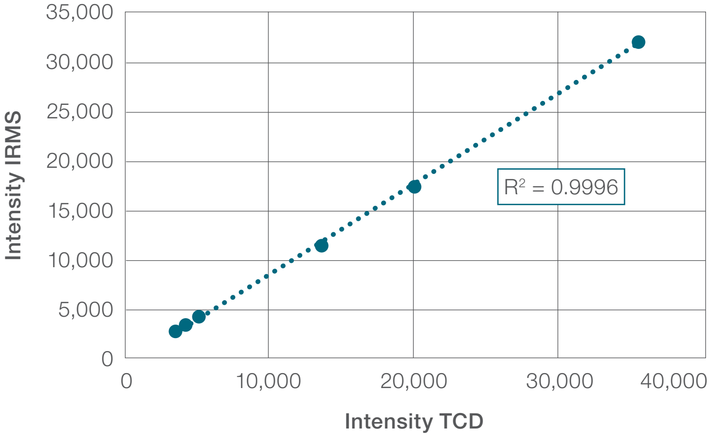
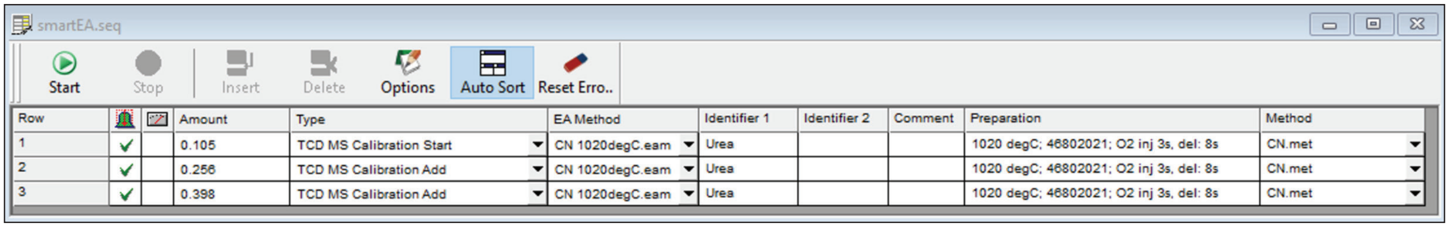


Figure 2. TCD-IRMS calibration of the EA IsoLink IRMS System.

Results

The effect of this automated dilution of the sample peaks, resulting from the linear calibration, is that linearity effects in the IRMS ion source are completely removed. This allows for analysis of samples with a wide range of elemental concentrations, within the same sequence, without having to develop individual methods for each sample or worry about not producing useable data.



Row			Amount	Type	EA Method	Identifier 1	Identifier 2	Comment	Preparation	Method
1	✓		0.105	TCD MS Calibration Start	CN 1020degC.eam	Urea			1020 degC; 46802021; O2 inj 3s, del: 8s	CN.met
2	✓		0.256	TCD MS Calibration Add	CN 1020degC.eam	Urea			1020 degC; 46802021; O2 inj 3s, del: 8s	CN.met
3	✓		0.398	TCD MS Calibration Add	CN 1020degC.eam	Urea			1020 degC; 46802021; O2 inj 3s, del: 8s	CN.met

Figure 1. An example of sequence table for the SmartEA Option analysis. The sequence table allows defining analyses to be used for a calibration of the TCD-IRMS detectors by the software. Choosing the appropriate entries in the column "Type" the software automatically creates a new calibration which will be active and used for subsequent sample analyses. Additional data points can be added to the calibration at any time of sequence. Due to the high linear relationship between the signal intensities of both detectors (see R2 in Figure 2) two-three calibration points are sufficient.

Figure 3 shows a triple isotope analysis of a standard with the SmartEA Option with the mass traces above and the TCD trace in the lower window.

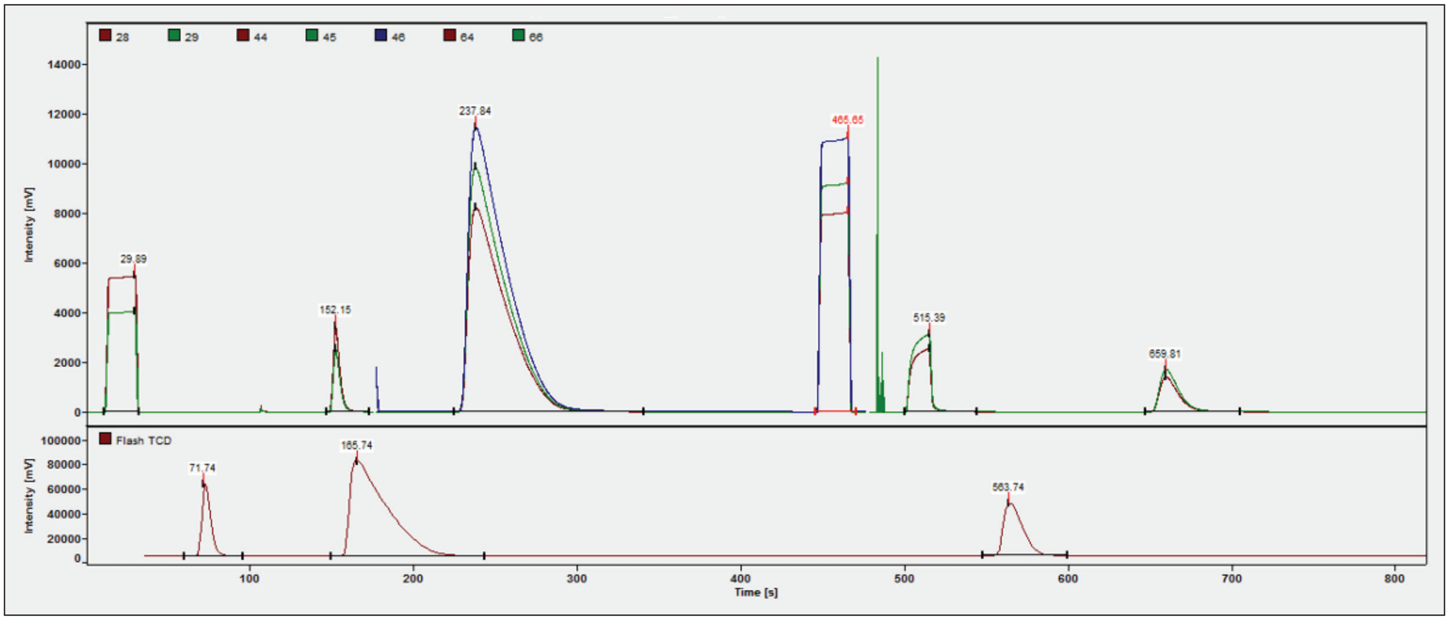


Figure 3. Analysis of 2.38 mg of sulfanilamide using the SmartEA Option. The figure shows the mass traces above and the TCD trace in the lower window. Squared peaks are reference gas. N_2 monitored on *m/z* 28–30, CO_2 on 44–46, SO_2 on 64/66. The requested signal intensity (mV on main mass) and dilution was for N_2: 5,000/89%, CO_2: 8,000/89%, SO_2: 2,000/80%.

As indicated in Figure 3, the IRMS peaks are delayed allowing for adjusted dilution in the ConFlo IV Universal Interface. This extends the analytical time by about 100 s. Once the TCD signal clearly indicates a down-sloping, the signal is identified by the software and labelled. In the following step the required dilution is calculated and applied using the incremental dilution of the ConFlo IV Universal Interface. Although peak areas of mass traces are a viable indicator for the elemental composition of the analyte, any dilution of the area makes it less accurately quantifiable. The TCD trace, however, is uncompromised. The area of the peak in the TCD trace can be used accurately to determine the weight percentage of an element, while the mass trace information is used to determine the isotope ratios.

Conclusions

The SmartEA Option for the EA IsoLink IRMS System extends the ease-of-use for routine analysis of samples with widely differing elemental composition. The user can set up a method that will bring all sample peaks within a narrow range of intensity, minimizing the risk of high sample loads or high elemental concentration in the ion source of the mass spectrometer. The SmartEA Option allows users to focus on their research by combining ease-of-use and unattended analysis to deliver high precision results.

Find out more at **thermofisher.com/EAIsoLink**

TN30809-EN 0121C