TECHNICAL NOTE 30807

EA-IRMS: Added flexibility for elemental analysis by macro reactor

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Goal

Demonstrate the flexibility of the macro reactor to perform elemental analysis in spectra of sample sizes, ranging from several tens of milligrams and carbon amounts of up to 30 mg, down to sub- milligram sample amounts.

Introduction

Peak shapes in elemental analysis are predominantly formed in the reactor. It is thus that volumes are normally reduced for analyses in the microgram range. Especially in systems using gas chromatographic principles, narrow peak shapes are a prerequisite for precise and accurate analysis. As part of the Thermo Scientific™ EA Isolink™ IRMS System, the Thermo Scientific™ Flash IRMS™ Elemental Analyzer (EA) is commonly equipped with an 18 mm outer diameter (OD) reactor tube. However, for large sample volumes and high carbon amounts, the macro reactor can be used. With 25 mm OD, the macro reactor conveniently fits into the furnaces. Quick and easy exchanges of the top feedthrough and the adapter of the Thermo Scientific™ MAS Plus Autosamplers allow the modification from a conventional 18 mm tube to the larger diameter tube within less than 10 minutes. Originally introduced for bulky samples of several tens of milligrams and carbon amounts of up to 30 mg, the macro reactor is also applicable to normal size samples down to the range of sub- milligram sample amount. The maintenance intervals are extended due to higher amount of chemicals used in the macro reactor, allowing for analysis of 400–600 samples. In this technical note we explain how to install macro rectors in the Flash IRMS EA and compare the performance of both reactors.

Instrument setup

The Flash IRMS EA in basic configuration includes all the required parts for the installation of the macro reactor. Please refer to Flash IRMS EA Operating Manual for detailed instructions. The furnace opening is already prepared for the wider diameter of the macro reactor. The upper opening is equipped with a detachable end plate to avoid heat loss when using the 18 mm reactor tubes. For installation of the macro reactor, the end plate needs to be taken off. The feedthrough on the top of the Flash IRMS EA can be removed by loosening two screws on the top of the instruments and replacing the feedthrough with the appropriate one for the 25 mm reactor tubes. As a final step, the adapter of the autosampler is exchanged to fit over wider tubes. Reactors can be packed according to the instructions for the 18 mm reactor type. The only parameter that requires adjustment is the sample drop delay. As O_2 moves slower through a wider volume the delay must be increased by about 5 seconds. A detailed description is given in the maintenance chapter of the Flash IRMS EA Operating Manual.

Results

The chromatograms in Figures 1 and 2 show the TCD trace of 200 ug Sulfanilamide. We compare the peak shape of the macro reactor (25 mm OD) to the conventional reactor (18 mm OD) by using the TCD trace. In contrast to the mass traces where dilution settings are used to keep the signal intensity within the amplifier range, the TCD trace is unambiguous. In both analyses, the carrier gas setting was 180 ml/min. In addition, both analyses use the Thermo Scientific™ Helium Management (HeM) Module of the Flash IRMS EA which reduces the flow to 50 ml/min on the separation column by automatic split control. Therefore, the conditions during combustion and separation were identical.

Figure 1 shows the NCS analysis in an 18 mm OD reactor, Figure 2 the analysis in a 25 mm OD reactor tube. The little ledge between the first peak (N_2) and the second peak (CO_2) indicates the activation of the HeM.

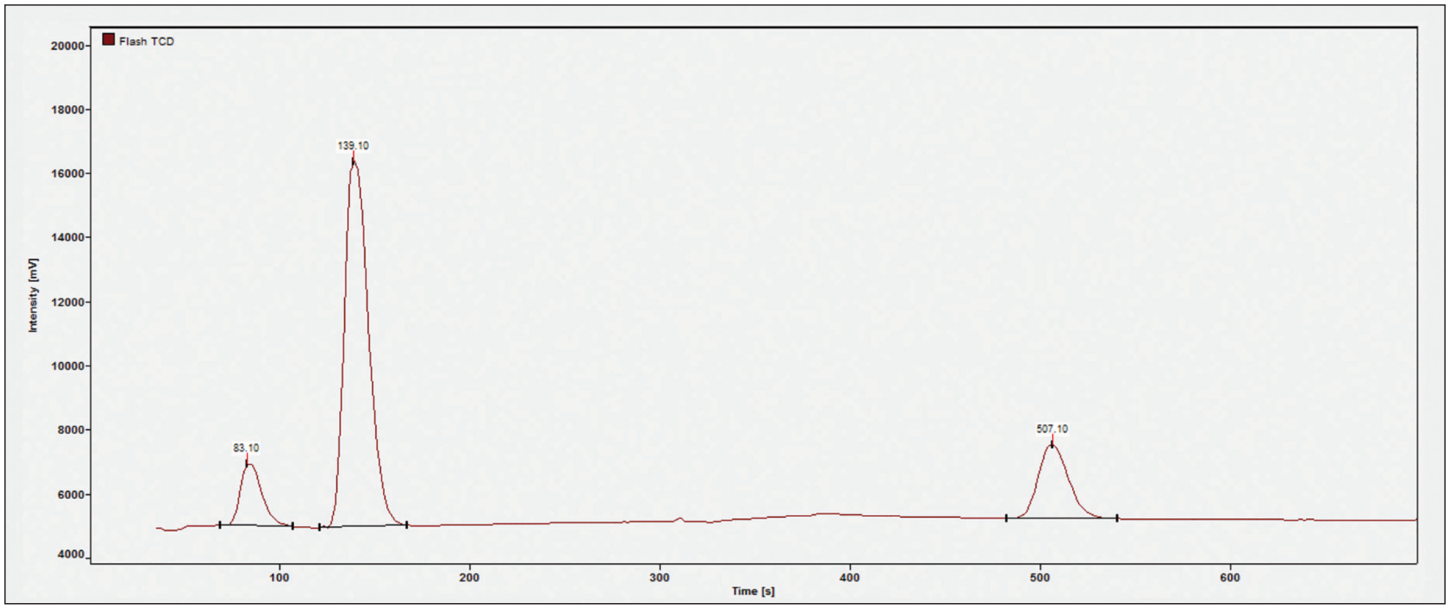

Figure 1. TCD trace of 204 ug of Sulfanilamide in an 18 mm reactor tube; N_2, CO_2 and SO_2 sample gas chromatographically separated.

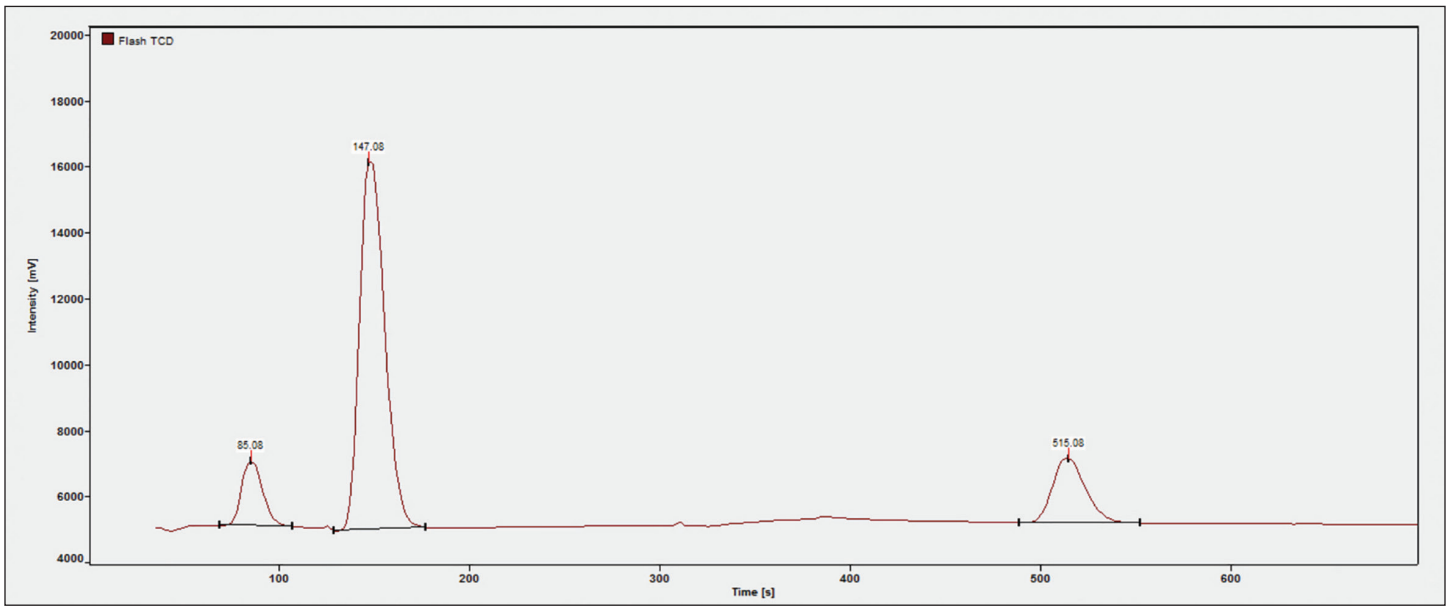

Figure 2. TCD trace of 200 ug of Sulfanilamide in a 25 mm reactor tube; N_2, CO_2 and SO_2 sample gas chromatographically separated.

As demonstrated in Table 1, the peak width in the macro reactor is insignificantly wider however coherent with the bigger volume of the macro reactor. Separation is fully preserved and there is no overlap of individual peaks.

Table 1. Comparison of peak widths between the 18 mm OD and 25 mm OD reactors.

	Sample amount (ug)	Peak width N_2 (s)	Peak width CO_2 (s)	Peak width SO_2 (s)
Sulfanilamide 18 mm OD reactor	204	38	46	58
Sulfanilamide 25 mm OD reactor	200	38	48	64

Conclusions

The installation of the macro reactor in the Flash IRMS EA is quick, easy and at no additional cost. Please refer to Flash IRMS EA Operating Manual for detailed instructions. By using the macro reactor there is no significant peak broadening and users benefit from significantly higher throughput alongside less frequent system maintenance.

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