Introduction

A blank from solid sample introduction limits the performance of any EA system. Particularly for small and isotopically enriched samples any contribution of air affects the isotopic ratio results. The bigger the blank and its isotopic difference from the sample, the greater the effect on the measured isotope value. Particularly in the on-line analysis of oxygen isotope, $^{16}$O/$^{18}$O ratio determination suffers for interference of atmospheric nitrogen ($^{14}$N). In this publication we present the first results obtained with the new NoBlank solid autosampler (Thermo Electron Co.) coupled with FlashEA 1112 elemental analyzer (Thermo Electron Co.) and Delta Plus XP mass spectrometer (Thermo Electron Co.).

Moreover, in order to improve the analytical condition, a new reactor with a -SiC- material tube was used, due to the currently available elemental analyzer’s reactor for the measurement of the oxygen isotope composition using CR-CF-IRMS method (carbon reduction - continuous flow - isotopic ratio mass spectrometry) produces amounts of background CO, with a stable baseline drifting upward proportionally to the temperature and downward to the time.

The only disadvantage is connected to the high thermal conductivity: 30 W*m$^{-1}$K$^{-1}$ at 1400°C for -SiC (Munro 1997), compared with 3.8 W*m$^{-1}$K$^{-1}$ at 1400°C for mullite ($^\circ$C). This problem has been solved by the use of oxygen-free (CMC), possibly choosing between the materials with the best cost/benefit ratio. In this study, a high purity silicon carbide (sintered -SiC) tube was selected as outer tube of the reactor (Fig. 6). Performance tests on the tube were executed on FlashEA 1112 elemental analyzer device (Thermo Electron Co., Milano - Italy) for the XRay-analyses.

References

- We are grateful to Guido Giazzi, Liliana Krotz, Martino Villa (Thermo Electron Co. - Milano) for their help in the X-ray analyses.

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**Strategies of background lowering in high temperature-carbothermic reduction-continuous flow oxygen isotope ratio determinant**

Tiziano Boschetti$^{1}$, Paola Iacumin$^{2}$ and Isabella Bombagi$^{3}$

1 - Earth Sciences Department, Parma University, Parco Area delle Scienze 157/A, 43100 Parma, Italy.

**Fig. 1** - Complete Sampling Unit.

**Fig. 2** - Schematic drawings of the NoBlank sampling phases.

**Fig. 3** - Front view of the NoBlank Sampler Device.

**Fig. 4** - Chromatograms comparison showing the NoBlank efficacy.

**Fig. 5** - Lower Fitting

**Fig. 6** - Schematic diagram of the modified tube on line tube reactor (left) and of the elemental analyzer (bottom) used for continuous flow isotopic measurements (CF-IRMS).

**Fig. 7** - Signal on mass 28 ($^{16}$O) vs. temperature of the reactor. Open diamonds: signal with the Al O- ceramic reactor’s tube; open squares: signal with the -SiC- ceramic reactor’s tube (Fig. 6).

**Fig. 8** - Calibration determined using various solid standard compounds, SiC-reactor and NoBlank autosampler device. For laboratory routine and intercomparison suitable standards, AgI+HCl (Kieselguhr (Wehmeier and lauer, 2003)).

**Fig. 9** - Calibration curve for the NoBlank autosampler device, also compatible with other elemental analyzer brands, introduction: 1) an innovative technique for the solid sample introduction exploiting the time of previous analysis for the purging. It does not require pre-warming time for conditioning or after carousel addition. In the carousel rotor a washing chamber is present. It is a particular shaped cavity to accommodate the sample. According to the movement of the rotating sampling system, the washing chamber has three positions: Loading, Purging and Sampling (Fig. 2).

**Fig. 10** - SEM micrograph showing the graphite (real graphite) surface (a) and the glassy carbon tube after the high temperature treatment (b).

**Fig. 11** - Somewhat higher current for the glassy carbon tube and a lower injection temperature (250°C) vs. CO) (5.5 grade) compared with 2.7 mV (5.5 grade) for the NoBlank device: 0.1-0.3 bar.

**Fig. 12** - Schematic sections of the rotor, front view. (Thermo Electron, 2005).

**Fig. 13** - Calibration curve for the NoBlank autosampler device, also compatible with other elemental analyzer brands, introduction: 1) an innovative technique for the solid sample introduction exploiting the time of previous analysis for the purging. It does not require pre-warming time for conditioning or after carousel addition. In the carousel rotor a washing chamber is present. It is a particular shaped cavity to accommodate the sample. According to the movement of the rotating sampling system, the washing chamber has three positions: Loading, Purging and Sampling (Fig. 2).

**Fig. 14** - SOM micrograph showing the graphite (real graphite) surface (a) and the glassy carbon tube after the high temperature treatment (b).