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

Tiziano Boschetti[1], Paola Iacumin[1], Isabella Bombagi[1]

1 - Dipartimento di Scienze della Terra, Università degli Studi di Parma, Parco Area delle Scienze 157/A, 43100 Parma, Italy.

On-line analyses of $^{18}O/^{16}O$ ratios suffer for interference of atmospheric nitrogen ($^{14}N_2$). Moreover, the reactors of the currently available elemental analyzers for the measurement of the oxygen isotopic composition produce increasing amounts of background CO. The reaction of glassy carbon (inner tube of reactor) with the ceramic tube (outer mantel tube of reactor) at temperatures higher than 1330°C is responsible for the CO production due to self diffusion of aluminium and oxygen from Al_2O_3-ceramic (Oishi and Kingery, 1960; Boschetti and Iacumin, 2005). This process is confirmed by 1) the growth of glassy pockets in the hottest zone of ceramic tube; 2) the surface alteration which may be observed on the outside of used glassy carbon tubes which show metallic aluminium inclusions; and 3) the formation of Al_2O_3 ashes visible on the outer surface of the glassy carbon tube even several days after the analysis and analyzed by a scanning electron microscope (SEM). To improve the conditions of these analyses, a high purity silicon carbide (SiC) tube was selected as outer tube of the reactor. Performance tests of this reactor have been carried out on solid standards and samples using: 1) Thermo Electron Flash HT-1112 elemental analyzer coupled with 2) Thermo Electron MAS 200R autosampler plus a new NoBlank device that prevents the entrance of air and 3) a Thermo Electron Delta Plus XP mass spectrometer. By means of these devices, the CO background at the maximum temperature of use (1450 °C) is lowered by four times when compared with standard reactors, while the nitrogen peak due to air is lower than 3 mV. The new NoBlank sampling device uses an innovative technique for the solid sample introduction exploiting the time of previous analysis for the purging and it does not require pre-analytic time for conditioning or after carousel addition.

References

OHISHI Y and KINGERY WD. 1960. Self-diffusion of oxygen in single crystal and polycristalline alluminum oxide. *The Journal of Chemical Physics*, **3**(2), 480-486.

BOSCHETTI T and IACUMIN P. 2001. Continuous-flow $\delta^{18}O$ measurements: new approach to standardization, high-temperature thermodynamic and sulfate analysis. *Rapid Communications in Mass Spectrometry*, **19**, 3007-3014.