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Mineralogical investigations coupling XRPD, HR-TEM and PIXE analytical method: preliminary results on standard minerals and Aeolian dust trapped in Antarctic ice

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Antarctic ice cores are actively studied to understand and reconstruct past climate (Petit et al., 1999). Aeolian mineral particles trapped in Antarctic ice are mainly transported by winds. Its mineralogical and geochemical composition depends on a number of factors among which the dust provenance and the geochemical characteristics of the dust source regions. After pioneering mineralogical studies (e.g. Briat et al., 1986; Gaudichet et al., 1988, Maggi, 1997), the mineralogical composition of mineral dust in polar ice has not been investigated in detail. Some recent studies on major elemental composition from PIXE analyses (Marino, PhD Thesis, 2006) however, provided some indirect indication of the mineral composition of dust.

In this work we present the preliminary results of a project aimed to set up an analytical protocol to define the mineralogical and crystal chemical composition of dust trapped in Antarctic ice. The main challenge is the very low amount of material in the ice samples, which is generally around the ppm range (1-10 μg * kg^{-1} of melted ice). Sample preparation for the different analytical techniques is therefore critical and prone to the external contamination. Firn and ice samples were decontaminated in a 1000 class clean room under a laminar flow bench. After melting, a first evaluation of dust concentration and size distribution was obtained by means of a particle counter Beckman Counter Multisizer III.

The analytical protocol include X-ray powder diffraction (XRPD), high resolution transmission electron microscopy (HR-TEM) coupled to energy dispersive X-ray fluorescence analysis (ED-XRF) and proton induced X-ray emission (PIXE) analysis on the same samples. Dust was deposited both on polycarbonate filters (for XRPD and PIXE investigation) and on copper-carbon grids (for HR-TEM analysis).

Specific analytical methodologies have been optimized for each of the selected techniques in order to enhance sensitivity and deal with the very small amount of materials. XRPD is performed using a state-of-the-art PANalytical X'Pert diffractometer in a parallel beam mode (Dapiaggi et al., 2006, *submitted*). The procedure allows detection, identification, and quantitative evaluation of 1-2 μg of mineral phases. PIXE has been optimized to detect sub-ppm levels of all major elements (Marino et al., PhD Thesis, 2006). Also, special crystallographic procedures are used to identify mineral phases starting from the HR-TEM diffraction images obtained on single particles and using the ED-XRF data as complementary information.

The results from the different techniques provide robust procedures for mineral phases identification. XRPD results are cross-checked with single-particle TEM analyses, but the former provides the statistics intrinsically lacking in single-particle TEM analyses. The coupling between TEM and ED-XRF analysis yields an approximate crystal chemistry of each mineral phase. Finally, the quantitative phase analyses obtained by XRPD allow a robust assessment of PIXE data.

Up to now, successful identification and analyses have been obtained on illite, kaolinite, montmorillonite, quartz and K-feldspar standards. The first part of this work focuses on different concentrations and size of certified minerals:

(1) bulk fraction (all size included) were investigated in order to define the lower concentration revealed by different techniques;

(2) a selected fraction (size < 5 μm) of the same minerals was checked by a particle counter and deposited on membrane/grid. Such particle size has been chosen as it is comparable to Aeolian dust trapped in Antarctic ice cores;

(3) a standard mineral mix (i.e. kaolinite, montmorillonite, quartz and K-feldspar) was prepared in different concentrations for mineralogical multi-phase analyses in order to simulate the mineral composition of real samples.

Successful identifications were also performed on smectite, kaolinite, illite, quartz, K-feldspar, calcite and albite contained within a few firn and ice samples from different East Antarctic shallow cores. The experimental protocol is now applied systematically to the investigation of all dust components from the ice cores recovered in the East Antarctic Plateau.