



**Experiment title:**

High Resolution Microprobe Analysis of Fluid Inclusion

**Experiment**

**number:**

CH-223

**Beamline:**

ID 10 (ID22)

**Date of experiment:**

from: 22/11/96 (16/07/97) to: 25/11/97 (19/07/97)

**Date of report:**

26/08/97

**Shifts:**

6

**Local contact(s):**

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### **Report:**

The aim of experiment CH-223 performed at ESRF on beamline ID10 from November 22 to 25 1996 was to determine the concentration of trace metals trapped in microscopic (10-50  $\mu\text{m}$ ) individual fluid inclusions and to perform 2-D maps of element distribution with high-sensitivity (below 1 ppm) and high spatial resolution (few microns).

In order to perform the X-ray measurements, the entire experimental set-up (mirror, Bragg-Fresnel lens, sample scanning stage and solid state detector) had to be installed. Unfortunately, the installation took much more time than expected (more than 48 hours) and we were unable to perform any measurements during the allocated beam time. In order to compensate for this lack of experiment, we recently (July 97) performed X-ray microfluorescence analyses on line ID22 still under commissioning. Different from the experimental set-up of line ID10 was the use of a Fresnel plate rather than a Bragg-Fresnel lens. The Fresnel plate ensured focusing of monochromatized X-rays at about 12 keV, with a spatial resolution of  $2 \times 7 \text{ pm}$  and a flux of  $\sim 10^9$  ph/sec. All measurements were

performed in collaboration with Michael Drakopoulos and Anatoly Snigirev.

The fluid inclusions studied were from natural quartz-bearing rocks collected in a variety of gold deposits (France, Portugal, Switzerland, Columbia and Brasil). These samples were chosen for two reasons: 1) they contain a unique fluid inclusion population (no problem of mixed fluids) and 2) the bulk composition of the trapped fluid is known and was used to evaluate the detection limits of the micro-fluorescence X analysis. (Bulk fluid inclusion compositions were determined by “crush-leach”; the method consists in crushing several gramms of quartz (i.e., containing several thousands of fluid inclusions) and analyzing the released fluid through conventional chemical analysis.)

Most of the results obtained during July 97 are still under treatment and cannot be presented here. In particular, it is not possible at this stage to confirm that the  $(K\alpha/K\beta)_i$  ratio of an element  $i$  in solution can be used as a reliable correction term for concentration estimates (Philippot et al., 1997). Two important points can be discussed, however: 1) Within the range of energy used (4-12 keV), we were able to detect in a single fluid inclusion all the trace elements determined by bulk-leach analysis. 2) The limit of detection was of the order of 100 ppm for trace metal such as arsenic (*an element of economic interest*). This value is one order of magnitude lower than the detection limits obtained by Mavrogenes et al. (1995) at NSLS and by ourselves (Ménez et al., 1997 a & b) at LURE. Although we are far from the expected ppm level, which is required for Au or U dosage, these preliminary results indicate that it is necessary to use the highly-intense ESRF source for analyzing trace metal concentrations in individual fluid inclusions.

## References

- Mavrogenes JA, Bodnar RJ, Anderson AJ, Bajt S, Sutton SR, Rivers ML (1995). *Geochim. Cosmochim. Acta* 59 : 3987-3995
- Ménez B, Philippot P, Chevallier P, Legrand F & Populus, P (1997a). *Terra Nova*, Abst. Vol.9, p.446.
- Ménez B, Philippot P, Chevallier P, Gibert F, Legrand F & Populus, P (1997b). *ECROFI* 14, Abst. Vol., p.207
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