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Trace element analysis with the electron microprobe: New data and perspectives

MICHEL FIALIN,[1,*] HUBERT RÉMY,[1] CLAUDINE RICHARD,[1] AND CHRISTIANE WAGNER[2]

[1]Centre de Microanalyse Camparis, Université Pierre et Marie Curie, 4 Place Jussieu, 75252 Paris cedex 05, France
[2]Laboratoire de Pétrologie, ESA 7058-CNRS, Université Pierre et Marie Curie, 4 Place Jussieu, 75252 Paris cedex 05, France

ABSTRACT

This work presents a procedure developed for trace element analysis using the electron microprobe (EMP). The method is demonstrated by analysis of seven glasses prepared from reference rock powders, with compositions of granite, granodiorite, andesite, diabase, and basalt. The melting process was adapted to prevent the loss of volatile elements and to obtain homogeneous samples. A routine procedure for analysis of major and minor (above 1000 ppm) elements yielded results in fairly good agreement with published values. The methods presented in this study produced detection limits as low as: (1) 6–8 ppm when the $K\alpha$ peaks of transition metals of the first row (Cr and Ni) were used; (2) 23 ppm with soft $L\alpha$ peaks (Y, Zr, and Sr); (3) 15 ppm with high-energy $L\alpha$ peaks (rare earth elements); and (4) 35 ppm with the $M\alpha$ peaks of heavy elements (Pb and Th). These limits were achieved after total counting times (peak + two background measurements) of about 15 min with a beam of 35 kV and 500 nA. Precision ($\pm 2\sigma$) on the weight percent concentrations was below 50% for concentrations above 13 ppm for group 1 elements, 35 ppm for group 2, 25 ppm for group 3, and 55 ppm for group 4. In the present work, a commercial software package was adapted for quantitative trace element analysis. One modification addresses beam-sensitive materials for which the long counting times required for measurements need to be divided into subsets (10–20 s count), each acquired from different sites of the sample surface, to minimize damage. This analytical mode is referred to as ''multi-site'' mode.