

Determination of PGE in nonsilicate rocks by fire assay with NiS collector

Jan Pašava*, Ivan Rubeška*, Václav Sixta*

For the study entitled *Sources, transport and fractionation of PGE in classical and unconventional geologic environments of the Bohemian Massif* carried out at the Czech Geological Survey, black shale, basic and ultrabasic rocks as well as samples of Cu-Ni ores from the Ransko gabbro-peridotite massif, Cu-Ni ores bound to diabase dykes intruded into granite rocks of the Lužický (Lusatian) plutonium had to be analysed.

The samples were powdered to analytical grain size and the PGE collected into a NiS button with reduced mass (approx. 2 g). The advantage of reducing the mass button is a lower blank, generally due to PGE impurities in the nickel reagent used in the fusion charge and a less time consuming treatment (crushing, dissolution, filtering) of the buttons. The disadvantage, on the other hand, is that the composition of the sample influences the composition of the fusion charge more strongly and thus may affect the processes and the final sulphide button formed during the fusion.

Any sulphur present in the sample increases the total sulphur content in the charge and phases with a higher sulphur content insoluble in hydrochloric acid may be formed. To avoid this difficulty the sulphur content in the samples was determined first and the amount of sulphur added to the charge was correspondingly reduced.

When analysing black shale another problem is caused by non-carbonate carbon. During fusion part of the sulphur present in the fusion charge is oxidised to sulphur dioxide and escapes into the atmosphere. This is evident from the fact, that the $S/(S+Ni)$ ratio in the fusion charges used by different authors varies between 0.35–0.48, whereas in the resulting button (which should mainly be Ni_3S_2) this ratio is only 0.27. Any carbon present in the sample reduces the amount of sulphur oxidised and thus again may affect the final phases of the nickel sulphide formed. The effect of carbon had to be taken into account above about 3% C.

In the solutions resulting by dissolution of the buttons PGE were measured by graphite furnace AAS as well as by ICP-MS (low contents). The results were checked by analyses of several SRM.

*Chemical Laboratory, Czech Geological Survey, Prague