DETERMINATION OF POTASSIUM, RUBIDIUM, THORIUM AND URANIUM BY ICP-QMS FOR TRAPPED CHARGE DATING OF QUATERNARY SEDIMENTS

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The last decade of Quaternary and palaeoclimatic research revealed an increased interest in the sensitive luminescence dating procedures, used to estimate age and evolution of different quaternary deposits such as fluvial or aeolian sediments (1). The luminescence signal results from radiation damage within minerals (electrons trapped at crystal lattice defects) caused by naturally occurring radioactivity, mainly due to elements from the \textsuperscript{232}Th and \textsuperscript{238}U/\textsuperscript{235}U decay chains and the decay of \textsuperscript{40}K and \textsuperscript{87}Rb. During sediment transport, the light sensitive luminescence signal in quartz and feldspars is zeroed by exposure to sunlight. After sedimentation, the mineral grains are sealed from daylight and the luminescence signal starts rising again. The deposition age of a sediment is calculated as the ratio of radiation damage, as evaluated by comparison of luminescence intensities from natural samples and artificially irradiated subsamples, and the dose rate, defined as the amount of effective ionising radiation within a sediment (2).

Considering improved accuracy, precision and the principally higher speed of determination, inductively coupled plasma quadrupole mass spectrometry (ICP-QMS, ELAN 6000, Perkin Elmer/Sciex) represents an actual alternative choice to gamma spectrometry, alpha and beta counting or solid state dosimetry. These conventional methods are not only time consuming but the need for large samples (up to 1.5 kg) is rather detrimental. However, the lack of adequate certified reference materials is a disadvantage in determination of thorium and uranium in quartz grains by ICP-QMS. A further serious inconvenience of the proposed method is the considerable degree of inhomogeneity of analysed Quaternary sediments. Our task was to prove, whether a large number of determinations on 100-200 mg samples combined with higher sample density may balance material inhomogeneity.

Conventional digestion of samples containing acid resistant detrital minerals like zircon, rutile, anatase or brookite in an open system lasts 4-6 days. Nevertheless the contents of U and Th were systematically too low compared with gamma spectrometric data (Preusser, unpl.). Consequently the quantitative digestion of all samples was performed under pressure
(170°C, 75.10^5 Pa) in the MULTIWAVE microwave digestion system (Perkin Elmer/Anton Paar) with HClO₄ (70%) and HF (40 %) (3). Using an ASPIVAP closed evaporation system connected with the MULTIWAVE, sample solutions were first evaporated to a volume of less than 1 ml and, in a second step after addition of HCl (30 %), nearly to dryness. Finally the residue was dissolved in HCl (20%) and the perfectly transparent solution was transferred to a 50 volumetric flask and stored in PFA bottles. Almost 100% of elements of interest were recovered by this method; moreover digestion time was shortened to one day.

We are encouraged by good accordance of ICP-QMS results with existing data obtained by low-level gamma spectrometry. For a final assessment it is compelling to considerably enlarge the small number of data. Furthermore, we intend to extend determinations of Th and U on biogenic carbonaceous materials, e.g. raised coral reefs, which are dated by Electron Spin Resonance (ESR) focusing on palaeo-sealevel changes and tectonic up-lift rates.