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In Situ on Microprobe U-Th-Pb Dating and REE Abundance of Biogenic Apatite

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In the past two decades, direct dating of fossiliferous sedimentary sequences has shown only limited success based on the Pb isotopic analyses of metamorphic carbonates that are biogenic in origin. This is partly due to complex post-depositional histories which prevent U-Pb chronometers from working correctly in carbonate rocks. If the direct U-Pb dating of a fossil itself is possible, the method could have great impact on stratigraphic studies in establishing the absolute chronology of sedimentary sequences. Tooth of fossil animals and micro fossil "conodont" are candidates for this purpose since they consist of apatite ($Ca_5(PO_4)_3$ (F,Cl,OH)), which would uptake U, Th and Pb after sedimentation no longer than a few million years and is supposed to remain closed to U and Pb under relatively low effective closure temperature. We report here results of direct ion microprobe U-Th-Pb dating of fossil tooth of a cartilaginous fish, "Orthacanthus senckenbergianus" collected in the Rotliegendes (Early Permian) sedimentary sequence in west Germany and a single fragment of conodont "Trichognathus" from Kinderhookian stage of Mississippian sedimentary sequence from Illinois Basin region in North America. Secondary purpose of the study is to indicate in situ analysis of all REEs on the same spots of U-Pb measurements. We have adopted a high mass resolution mode which can separate possible spectroscopic interferences of heavy REEs from oxides of light REEs. The samples were cast into epoxy resin discs with a few grains of standard apatite, "PRAP", derived from an alkaline rock of Prairie Lake circular complex in the Canadian Shield dated at 1156 Ma. The sample and standard were polished until they were exposed through their mid-sections to provide a flat surface for sputtering of secondary ions. About 5-nA O_2^- primary beam was focused to sputter a 30-mm-diameter area of sample and/or standard and the positive secondary ions were extracted using 10 kV. No isobaric interferences were found in the mass range over ^{204}Pb and ^{208}Pb at mass resolution of 5800. The magnet was cyclically peak-stepped from mass 159 ($^{40}Ca_2{}^{31}P^{16}O_4{}^+$) to mass 254 ($^{238}U^{16}O^+$), including the background and all Pb isotopes and 238 and 248 for ^{238}U and $^{232}Th^{16}O$, respectively. Identical primary beam condition to the U-Pb measurement was used in REE analysis of conodont while enhanced mass resolution of 9300 at 1% peak height was adopted. The magnet was cyclically peak-stepped from mass 139 ($^{139}La^+$) to mass 175 ($^{175}Lu^+$), including the background and all significant REE isotopes and the matrix peak ($^{40}Ca_2{}^{31}P^{16}O_3{}^+$). Observed intensities of the isotopes are calibrated against those of PRAP standard whose REE contents were determined by ICP-MS after chemical dissolution and separation. There is a positive correlation between of $^{238}U/^{204}Pb$ and $^{206}Pb/^{204}Pb$ ratios for the fossil tooth of fresh-water shark. A least-square fit using the York method gives the ^{238}U-$^{206}Pb^*$ isochron age of 266±18 Ma (2σ; MSWD=1.4), which agrees well with the depositional age of the fossil in Early Permian (256-290 Ma) sedimentary sequences. Similar correlation is found in the Carboniferous conodont, which yields the $^{206}Pb^*$-^{238}U isochron age of 323±36 Ma (2σ; MSWD=1.03). The age indicates marginal agreement with the depositional age of the fossil in Kinderhookian stage and/or Tournaisian epoch (350-363 Ma) sedimentary sequence. The success of the ion microprobe U-Pb dating of biogenic apatite depends on the chemical fractionation of U from Pb in a hundred mm size in the sample and the consequent variations in Pb isotopic compositions due to radioactive decay of U. The variation in this size can not be easily observed by IDTIMS method after chemical dissolution and separation of the samples. REEs concentrations of two spots on the Carboniferous conodont vary significantly from less than 1 ppm of heavy REE to 60~70 ppm of light REE. Shale-normalized REE’s pattern show a flat from light REE to middle REE and decrease from middle REE to heavy REE except for negative anomalies of Ce and Eu, which is significantly different from Devonian and Ordovician conodonts having concave-shape with the middle REE enrichment and a weak or absent Ce anomaly reported by other workers. Discrepancy of the patterns may be attributable to either redox state during the diffusive exchange of REEs or different formation environments such as river and sea water.