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Supplemental Material

Methods, Figures S1–S7, and Tables S1–S3.

Polymorphic transformations of titanium oxides contribute to economic U mineralization in sandstone

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Supplementary Material

METHODS

Polished thin sections of samples from the grey sandstone of the Lower Yaojia Formation were coated by carbon films for electronic conduction. Back-scattering electron (BSE) images were obtained by a scanning electron microscope (Nova Nano SEM450) at the State Key Laboratory of Nuclear Resources and Environment (NRE), East China University of Technology (ECUT). A TESCAN Integrated Mineral

Analyzer (TIMA) system at the Guangzhou Tuoyan Analytical Technology Co., Ltd. (GTCL) was performed at an accelerating voltage of 25 kV, a probe current of 9 nA, a working distance of 15 mm, a pixel spacing of 3 μm, and a dot spacing of 9 μm, generating a colored mineral distribution map and a corresponding BSE image for the selected thin section. A focused ion beam (FIB) combined with SEM (TESCAN AMBER) was utilized to mill specific sites from the related thin section, producing an electron-transparent foil at the GTCL. The obtained foil was observed by an FEI Talos F200S transmission electron microscope (TEM) at the Guangzhou institute of Geochemistry, Chinese Academy of Sciences. Energy dispersive X-ray analysis, bright-field image, dark-field image, as well as high-resolution lattice fringe image and related diffraction pattern were routinely acquired.

Two groups of titanium oxides were picked up from the upper and lower members of Yaojia Formations respectively, and further embedded into an epoxy. At room temperature, a Raman spectroscopy (Renishaw inVia) equipped with a 532 nm excitation source at the NRE was used to obtain spectra of anatase and rutile (100 to 1000 cm^{-1}) with power levels of 1 mW and 10 mW, respectively. For each analysis, an accumulation of 2 and an acquisition time of 10 s are executed. Wavenumbers of main bands are indicated to identify polymorph type of titanium oxide (e.g., anatase and rutile). Major elements contents in titanium oxides were determined by an electron microprobe analyzer (JXA-8530F Plus) at the NRE. The instrumental settings include a diameter of 1 μm, an accelerating voltage of 20 keV, a beam current of 20 nA and an integration time varying from 15 to 90 s for different elements. The obtained data can be found in Table S1.

Trace elements concentrations of titanium oxides were determined by an Agilent 7900 ICP-MS combined with a GeoLasHD ArF Excimer laser at the NRE. Characterized by 44 μm ablating spot at 6 Hz with energy of ~90 mJ/pulse, each analysis was consisted of 20 s for gas blank and 40 s for sample signal. The calibration was performed by the external standards (NIST610, NIST612, BCR-2G, BHVO-2G, BIR-1G) in conjunction with the internal standard element of ^{49}Ti provided by electron microprobe analysis. The ARM-3 material was used as the monitor standard to ensure analytical accuracy. The data reduction was conducted by ICPMSDataCal (Liu et al., 2008). The detailed results are presented in Tables S2.

The quantitative analysis for Ti isotope of titanium oxide was conducted by a Neptune Plus MC-ICP-MS equipped with a femtosecond laser ablation system. Detailed procedures and instrumental settings can be found in Liu et al. (2023). The standard-sample bracketing technique was adopted to correct for instrumental mass bias during all analytical sessions. Offline data processing including cycle selection, integration of background and analytical signals, and isotopic ratio calibration were performed using the Iso-Compass software (Zhang et al., 2020). Rutile USA75 and BRA10 standards were measured in all sessions as quality control. The yielded results of USA75 (0.25±0.22, 2sd, n=11) and BRA10 (-0.20±0.13, 2sd, n=11) were consistent with the reported values within analytical errors (Liu et al., 2023). The obtained $\delta^{49/47}Ti$ in Table S3 refers to the per mil difference of $^{49}Ti/^{47}Ti$ relative to the international OL-Ti standard in Origins Laboratory, University of Chicago.

Reference

Liu, H., Zhang, W., Deng, Z., Hu, Z., Schiller, M., Bizzarro, M., Liu, Y., Luo, T., Feng, Y., and Feng, L., 2023, Determination of titanium isotopes in rutiles with high spatial resolution by femtosecond laser ablation multi-collector inductively coupled plasma mass spectrometry: Spectrochimica Acta Part B: Atomic Spectroscopy, v. 202, p. 106646.

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Supplementary Material

Figures S1-10

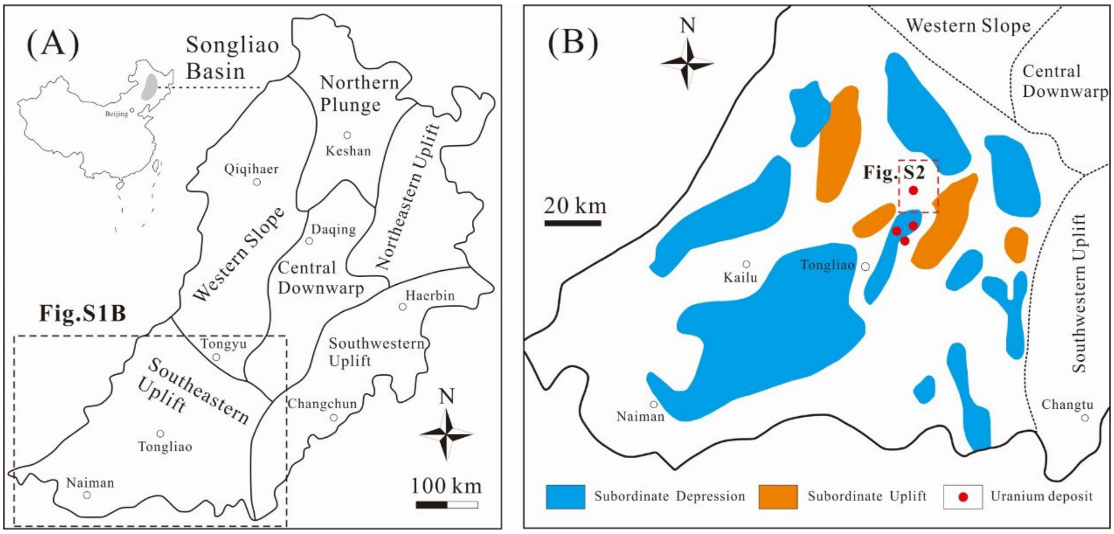

Figure S1. (A) Tectonic units of Songliao Basin, NE China, modified from Jia et al. (2020). (B) Tectonic units of SW Songliao Basin, NE China, modified from Zang et al. (2023).

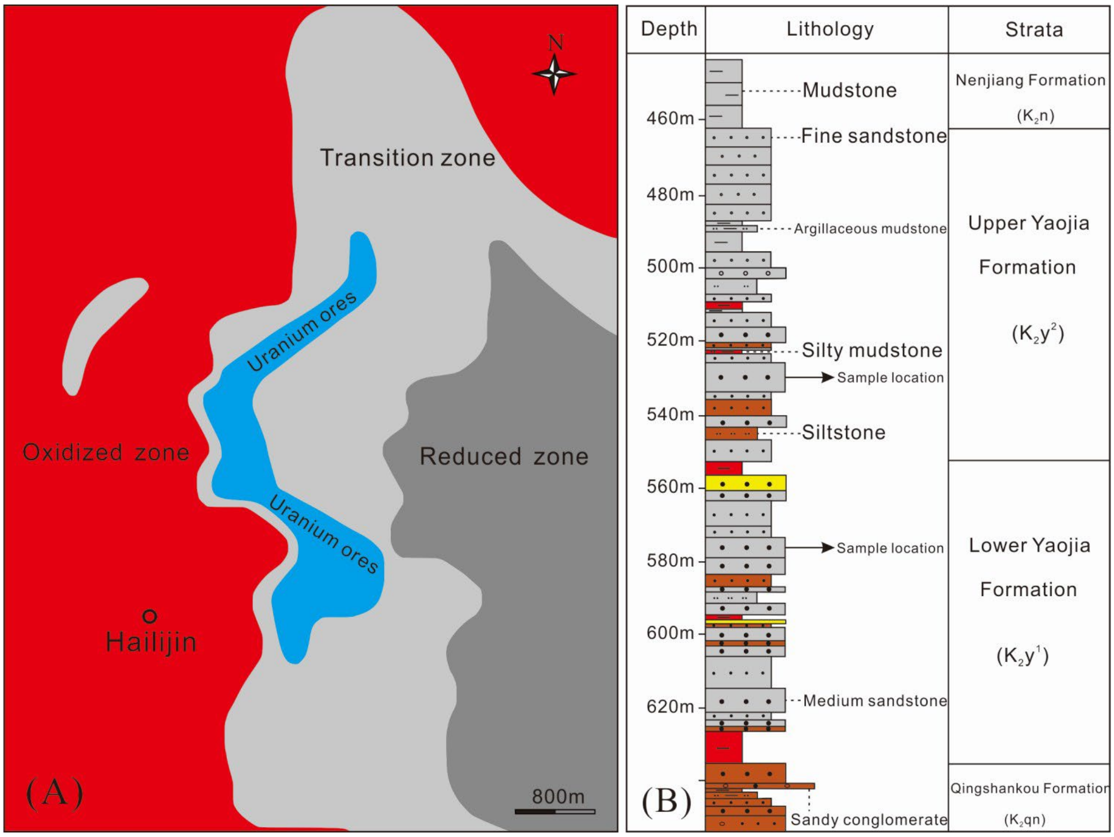

Figure S2. (A-B) Redox state and representative stratigraphic column of the Hailijin ore field, modified from Zang et al. (2023).

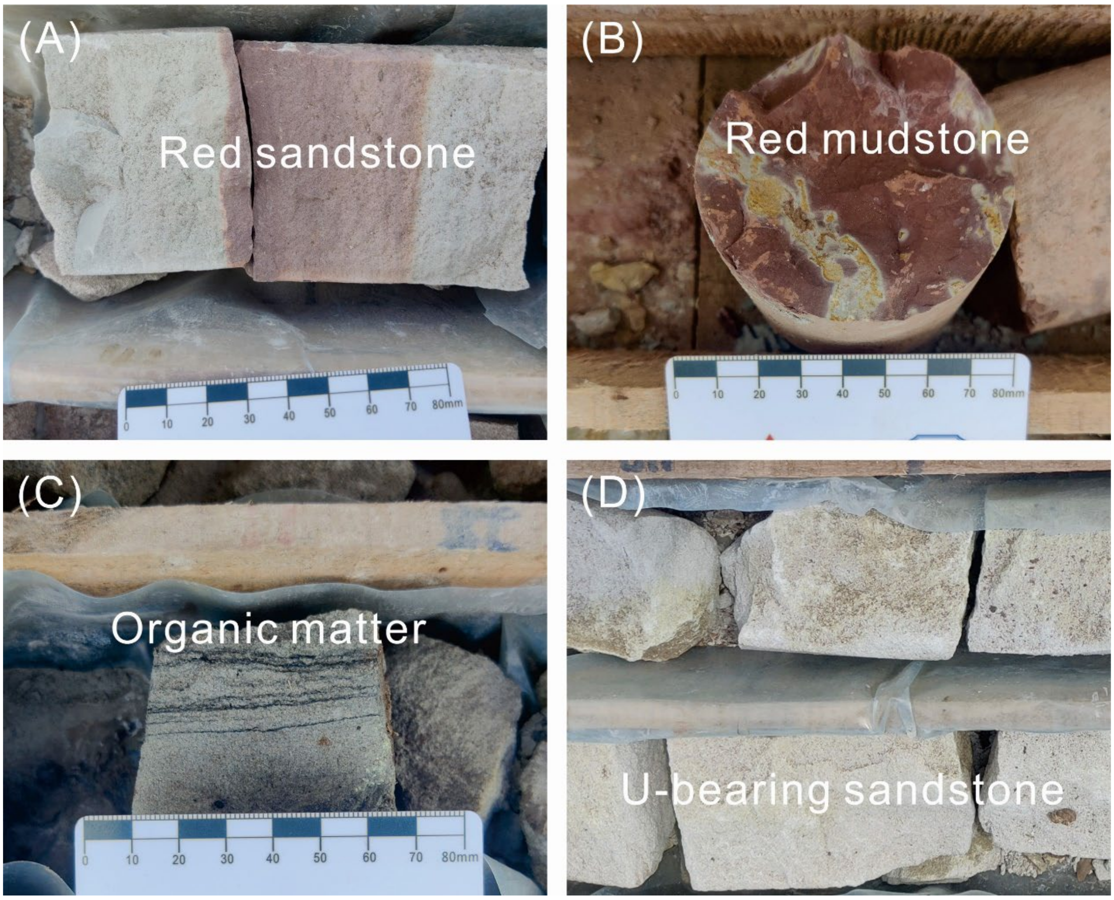

Figure S3. (A-D) Representative samples of sandstone and mudstone in the Hailijin ore field.

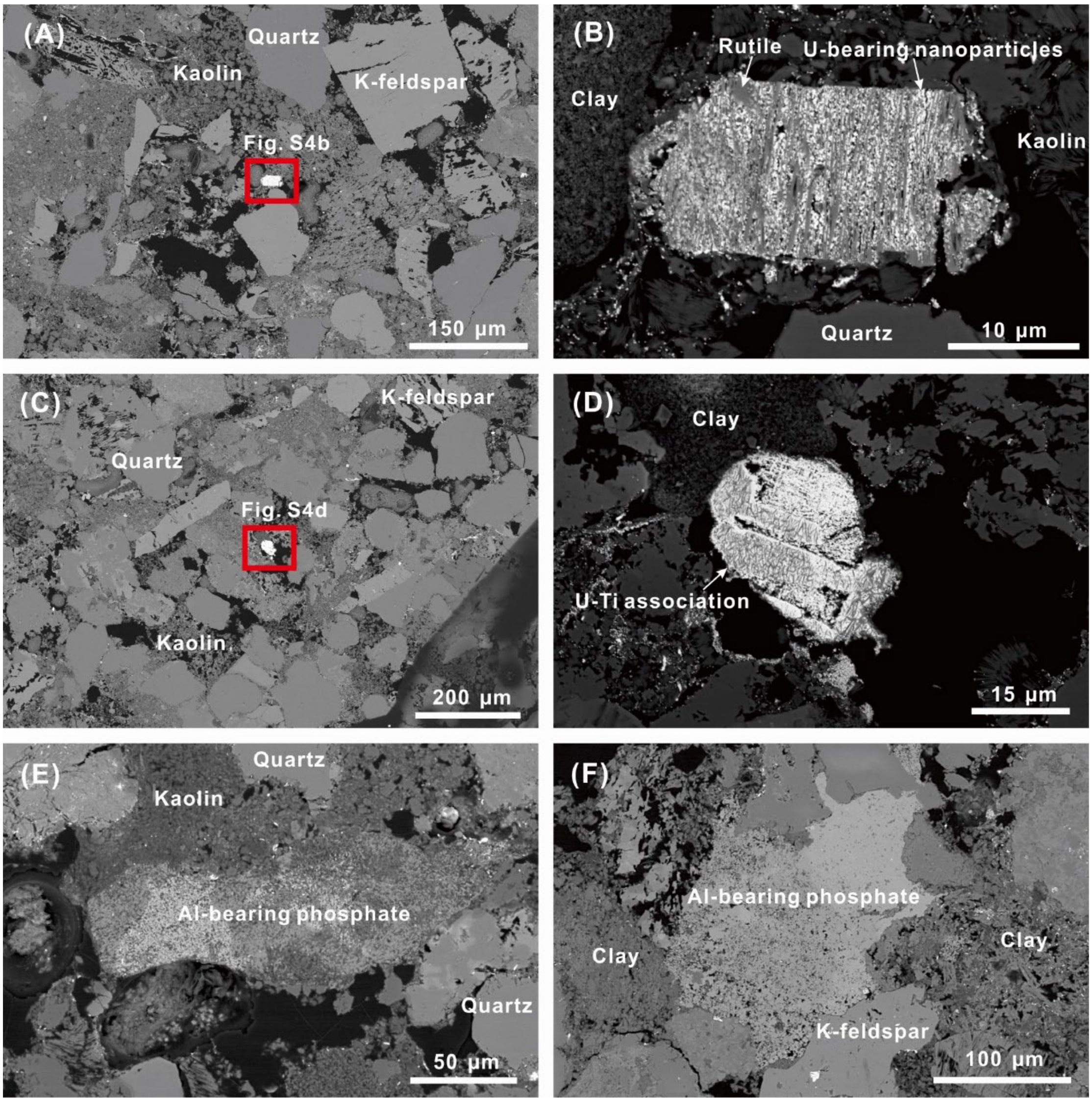

Figure S4. (A-D) Uranium-bearing nanoaggregates coexist with rutile. Note that U is strongly concentrated by rutile, rather than neighbouring clays. (E-F) Aluminum-bearing phosphates can be observed in cements.

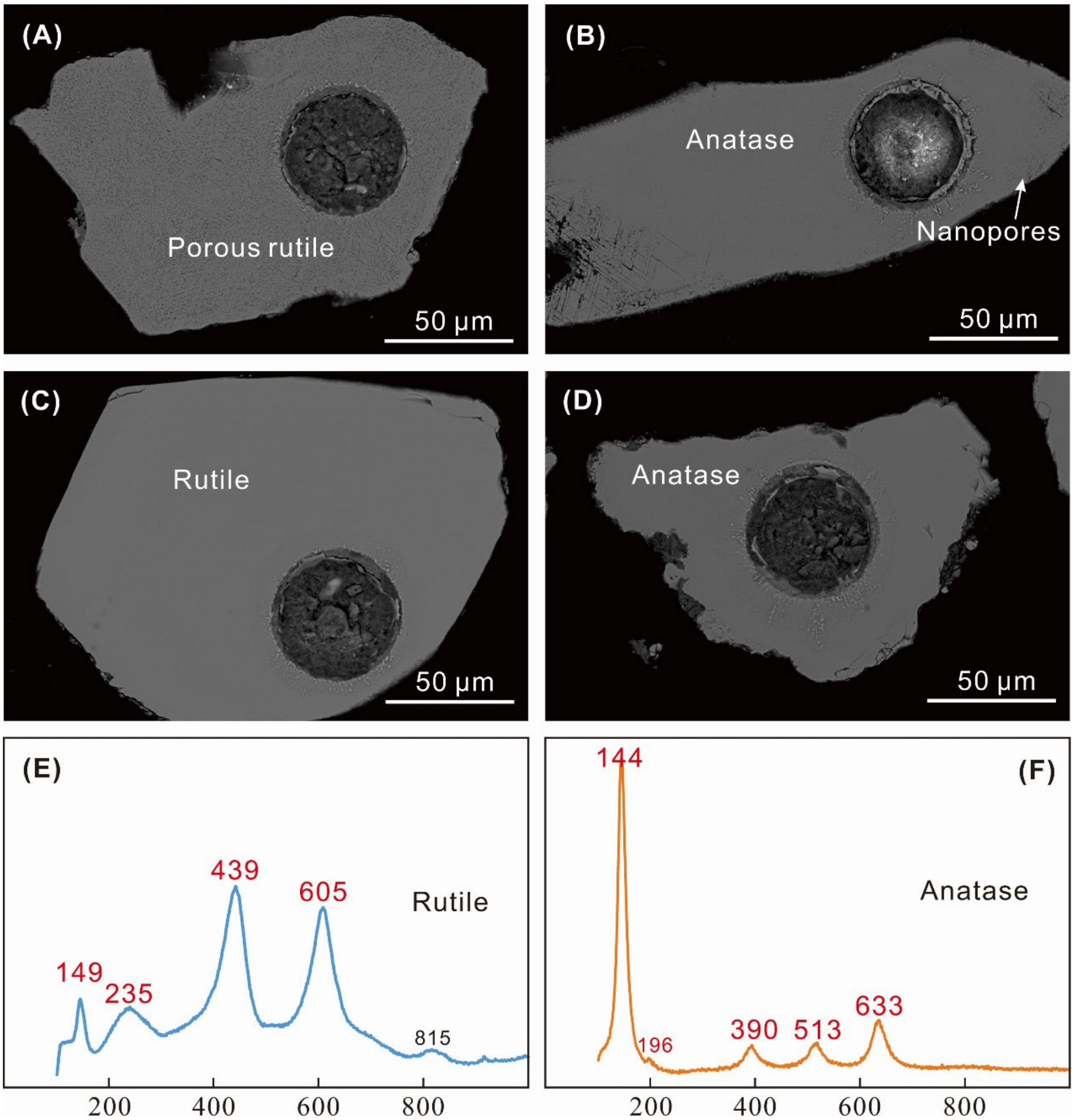

Figure S5. (A-D) Titanium oxides are either smooth or porous. (E-F) Wavenumbers of main bands (red-colored wavenumber) are indicated to identify polymorph type of titanium oxide (i.e., anatase or rutile). Note that the pits were produced by laser ablation system.

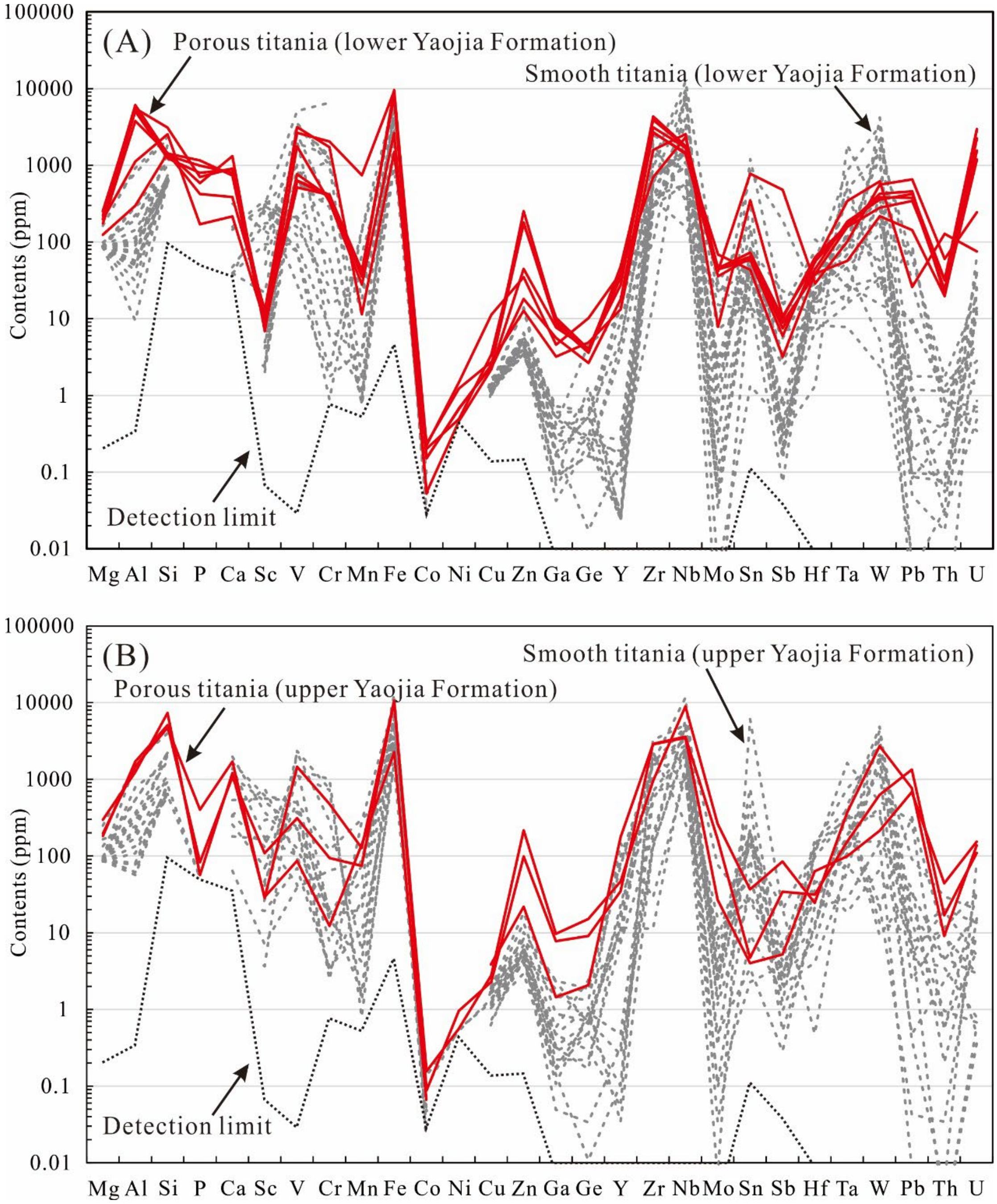

Figure S6. (A-B) Trace elements concentrations of porous and smooth titanium oxides from the upper and lower members of Yaojia Formations.

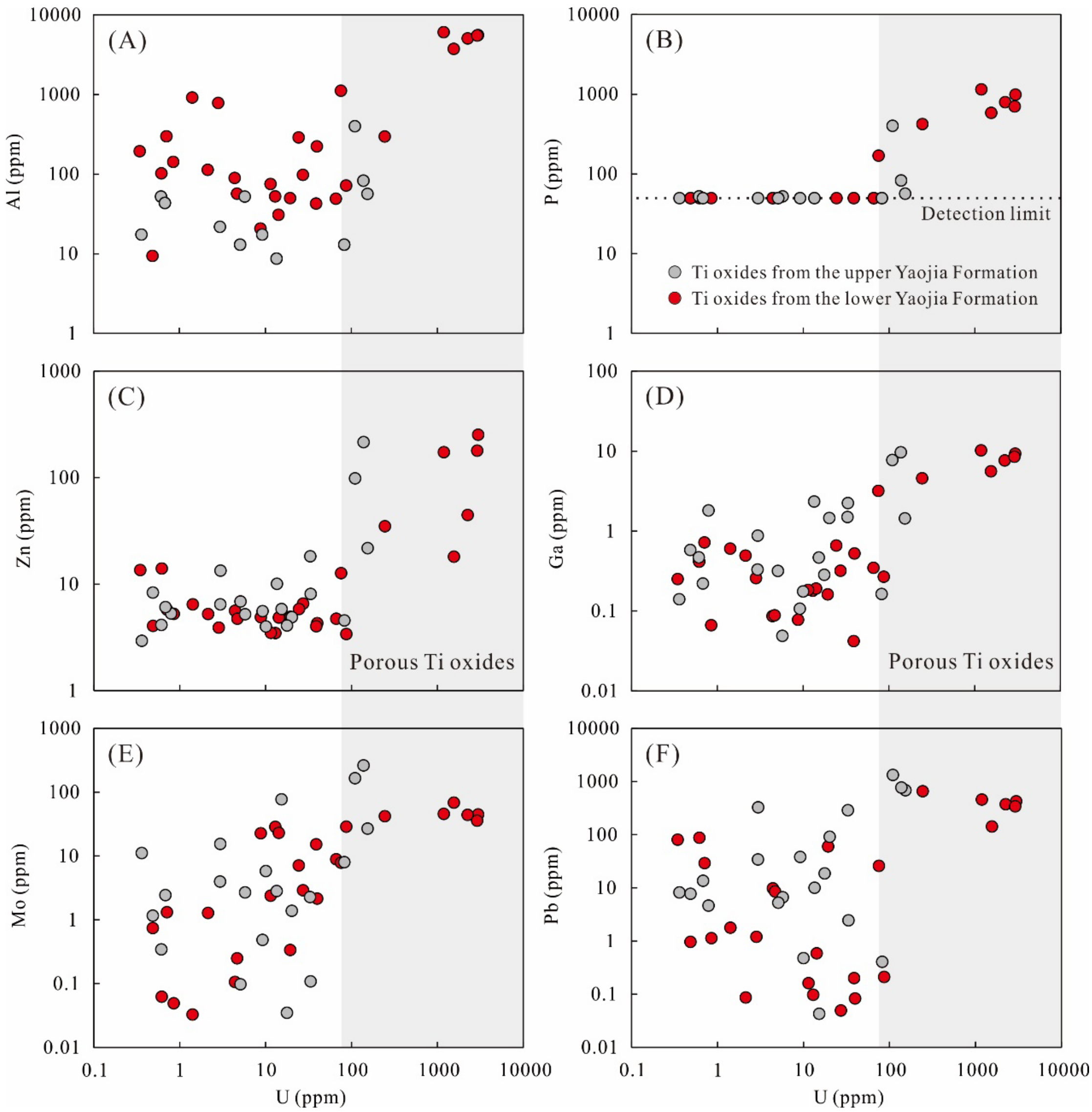

Figure S7. (A-F) Porous titanium oxides have distinctly higher contents of U, Al, P, Zn, Ga, Mo, and Pb than those of smooth grains. Note that the P contents in some titanium oxides are below detection limit, and thus plot along this line for comparison.

Reference

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