

## Vibrational spectroscopic study of Np(V) sorption on mineral oxides

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Mineral oxides play a decisive role in regulating the mobility of contaminants in the environment because of their widespread occurrence in rocks and soils, their tendency to form coatings on mineral surfaces and their wide-ranging technical applications [1].

Due to its long half-life and its toxicity, Np-237 is considered as a major contaminant of the ecosystem in the long-term safety assessment of nuclear waste repositories. The pentavalent state is environmentally most relevant [2].

For the first time, in-situ Np(V) sorption is comparatively studied on the oxyhydroxides of Fe, Mn, Si and Ti by ATR FT-IR spectroscopy under a variety of environmentally relevant sorption conditions. From the results, the formation of binary inner-sphere complexes on oxides of Si, Mn, Fe and Ti can be derived [3]. In case of ferrihydrite, the formation of an additional ternary Np-carbonato surface species is assumed. In addition, time resolved spectra provide kinetic information on the surface reactions.

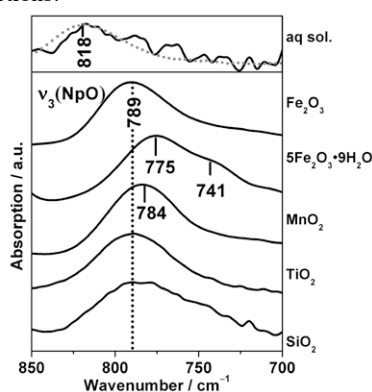


Fig. 1: IR spectra of an aq. Np(V) solution and of the sorption complexes formed onto several mineral oxides (50  $\mu$ M Np(V), 0.1 M NaCl, pH 7, 60 min sorption, 0.1 mg mineral oxide/cm<sup>2</sup>, N<sub>2</sub>).

[1] Dixon J. B. *et al.* (1989) *Minerals in soil environments*. Madison, Wis.: Soil Science Soc. of America. 1244. [2] Kaszuba J. P. *et al.* (1999) *Env. Sci. & Techn.* **33**(24), 4427-4433. [3] Müller K. *et al.* (2009) *Env. Sci. & Techn.* **43**(20): p. 7665-7670

## Nano-particulate pressed powder tablets for LA-ICP-MS

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The accurate determination of ultra-trace elements in geological samples containing refractory minerals - e.g., zircon in plutonic rocks and sediments, spinel in ultramafic rocks and eclogites - is a challenge. While standard table-top or microwave-assisted digestion protocols fail in completely dissolving these minerals, alternative procedures also show shortcomings: (i) pressurized bomb digestion yields accurate results but is time-consuming; (ii) preparation of fused glasses with, and without, addition of a flux for subsequent analysis by either LA-ICP-MS or after re-dissolution with ICP-MS suffers from contamination from heterogeneously distributed impurities in lithiummetaborate, and strong memory for Li, B; (iii) shock melting using a strip-heater produces glasses contaminated with strip material. Ultrabasic or evolved rocks cannot be melted without matrix modification. All fusion techniques bear the risk of volatile losses, and glass beads typically show significant inhomogeneity of trace elements if not thoroughly homogenized during melting.

Pressed powder tablets have repeatedly reported as a means for the direct analysis of solids by laser ablation. Analytical results, however, were not convincing in terms of detection limits, accuracy, and precision if compared to results obtainable with solution analysis or homogeneous glasses. Here we show that undiluted pressed powder tablets can be successfully used for ultra-trace element analysis of granitoid, gabbroic, and ultrabasic rocks from the Oman ophiolite (Wadi Gideah reference profile) after pulverisation to nano-particle grain size. More than 40 trace elements have been analysed in a series of rock CRMs with average RSDs of 1-3% and excellent accuracy for most elements incl. HFSE. Detection limits are in the low ppb range.