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The matrix influence on the determination of low uranium concentrations

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We refer about a development of the measurement technique enabling the determination of low uranium concentrations. The method was inspired by a commercially available KPA method (kinetic phosphorescence analysis). It is fast, sensitive, and non-destructive, allowing the uranium concentrations determination on a mass basis of $\mu\text{g U/l}$.

Contrary to the conventional KPA method it is in our approach not necessary to use an external standard as a reference sample. Moreover the solid state laser is used instead of nitrogen or dye laser and thus no dye solution preparation is required and no laser tube degradation appears. As an excitation source we use the solid state tunable laser, commonly utilized for TRLFS (time resolved laser induced fluorescence spectroscopy) measurement, operating at 416 nm for which the uranium complex fluorescence signal has the highest intensity. Excitation energy was 4.5 mJ. The fluorescence spectra were recorded using the grating monochromator followed by an ICCD camera. To eliminate the influence of possible excitation energy fluctuation on the measured fluorescence intensities, each recorded spectra were normalized with respect to this energy. For every sample ten measurements with 200 acquisitions were realized (the overall acquisition time was ~ 3 min). Besides the intensity measurement the method enables the study of the emission spectral characteristic and the detail measurement of complexes wavelength shift. Because the fluorescence is strongly temperature-dependent, the temperature of the sample was controlled and stabilized.

In our laboratory, the concentration of uranium is determined mainly in samples resulting from the leachates of rock and sediment materials and of surface water. Therefore we have focused on verifying the applicability of our method for samples with such matrix that could significantly influences the determination of uranium concentration. For that reason we studied the influence of the particular matrix, complexing agent, and the sample pH on the recorded fluorescence spectrum shape and integral intensity.

The uranium concentration in samples ranged from 1 to 1000 $\mu\text{g/l}$. To acidify the samples the HNO_3 was used. To limit the quenching effect and to prolong the UO_2^{2+} life-time, a complexing agent URAPLEX or optionally more available H_3PO_4 was added.

We can conclude that for most of the investigated matrixes the described method offered sufficient precision of the uranium concentration determination. On the other side, it was found that the presence of nitrates and chlorides could be problematical. Especially in case of nitrates even the method of standard addition did not offered satisfying results. This behavior is under further investigation.

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