QUANTITATIVE DETERMINATION OF UO₂ AND ThO₂ IN POWDERY MIXTURES BY INFRARED SPECTROMETRY

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Infrared spectra of powdery mixtures of UO₂ and ThO₂ were quantitatively evaluated with a computer. The computer programme was tested using the spectra of sulphates. Preliminary results are given and a possible use of this method for analyzing the mixtures of UO₂ with FuO₂ is suggested.

Introduction

The infrared spectra of UO₂ and ThO₂, which are isomorphous with PuO₂, have been measured by different authors on single crystals, polycrystalline materials, and powders, using reflection or absorption methods. The spectra were evaluated mostly from the theoretical point of view to establish the structures of the oxides.

In spite of the fact that the spectra of both oxides are very similar, we have made an attempt to utilize them for the quantitative analysis of the oxide mixture and thus to develop a non-destructive, relatively simple, rapid and, within certain limits, reliable, method. We supposed that it would be rather an orientation method as the results could be probably affected by the stoichiometry of UO₂, size and shape of powder grains due to the precision of spectrometric methods which lies usually within the limits of ±5% rel.

Experimental

The infrared absorption spectra of powdered samples of the same origin, prepared strictly by the same method in the form of paraffin oil suspensions were measured. The samples were ground in an agate mortar for 10 min and mixed (~15 : ~50 mg) with paraffin oil. The paraffin oil mulls were placed into the demountable cells provided with CsI windows, the path length of the cell being ~0.02 mm, and their infrared spectra were measured with a Perkin-Elmer double-beam grating spectrometer, model 225.
Results and discussion

The spectra of pure stoichiometric UO$_2$ and ThO$_2$ powders are in Fig. 1. The most interesting and useful part of the spectra is in the region of ~250–600 cm$^{-1}$, where a strong, poorly resolved, doublet or triplet appears for both oxides. The spectral patterns of both oxides do not differ much from one another. The frequencies of the maxima have very similar values and the only larger difference appears in the relative multiplet branch intensities, where the branch of ThO$_2$ at 290 cm$^{-1}$ displays a considerably higher intensity. It is clear from this figure that it would be a very unpleasant task to analyze a mixture of both oxides by using the usual spectrophotometric evaluation which needs either a perfect fit to Beer's law or calibration curves for a known cell thickness or the use of an internal standard.

The computer programme (prepared by Dr. LELEK of this Institute) eliminates all these experimental difficulties. To prepare the input data, the spectra of the