Evaluation of the extraction efficiency of low-soluble impurities from aqueous solutions by supercritical fluids in the dynamic flow mode by the chromatography-mass spectrometry method

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Supercritical fluid extraction (SFE) is actively used in the separation of valuable components or especially toxic and radioactive substances from various natural sources, such as water systems. The convenience of using this method is due to the fact that the fluids often used in it (CO2, freons, propane) have a low solubility in water and also dissolve water quite little. Therefore, there are no difficulties with the separation of the extract and raffinate (water purified from the dissolved substances). Despite the active study and application of this method, its possibilities have not yet been fully studied.

In this study, two poorly soluble substances are considered as model objects - tributyl phosphate (TBP) and methanitrobenzotrifluoride (meta-nitro-trifluoromethyl-benzene, F-3), which are used in technologies for the release of radionuclides during the reprocessing of spent nuclear fuel (SNF). The purpose of this study is to evaluate the efficiency of the extraction purification of aqueous solutions containing TBP and F-3 under dynamic conditions based on the estimated calculation and to verify its results in an experiment on a countercurrent extraction column.

The calculation is based on the data on the distribution of impurities between the water and fluid phases, which were obtained in static experiments. The concentrations of substances in the initial solutions (before extraction) were determined by gas chromatography with mass spectrometric detection (GC-MS) using a SHIMADZU GCMS-TQ8040 instrument. For chromatographic separation, a standard ZB-5MS capillary column (60 m × 0.25 mm × 0.25 μm) was used. The same method was applied to determine the residual concentrations of TPB and F-3 in final aqueous solutions (after extraction).

Based on the data on extraction under static conditions, the interfacial distribution coefficients (K) were determined for TBP and F-3 between the aqueous phase and supercritical (SC) fluids - CO_2 and R-23 freon (CHF_3) at 50°C and a fluid density of about 0.4 g/cm^3. It was shown that, under these conditions, SC CO_2 is a more effective extractant for the isolation of TBP and F-3 from aqueous solutions. The obtained K values were used to evaluate the efficiency of the extraction purification of aqueous solutions from TBP and F-3 in various modes and high degrees of purification of aqueous solutions from poorly soluble organic substances were predicted. The performance of such an evaluation has been demonstrated in experiments in a countercurrent extraction column (PEC). The results of the experiments in PEC show a good agreement with the made forecast calculation. It was shown that in all the pairs of "extractable component - extractant" studied, the degree of purification in PEC can reach 0.995 at a moderate consumption of the extractant.

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