

LATE PAPERS

4P-13

RADIATION STABILITY OF CYMe₄-BTBP AND CYMe₄-BTPHEN IN SYSTEM WITH PHENYL TRIFLUOROMETHYL SULFONE DILUENT

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The CyMe₄-BTBP and CyMe₄-BTPhen molecules are currently most promising extractants for the European SANEX and/or GANEX processes for the recovery of minor actinides from a spent nuclear fuel solution. Any extractant used in such processes must show a sufficient selectivity toward the actinides, good solubility in organic phase, must be resistant toward hydrolysis and radiolysis and any degradation products formed must not influence the separation¹.

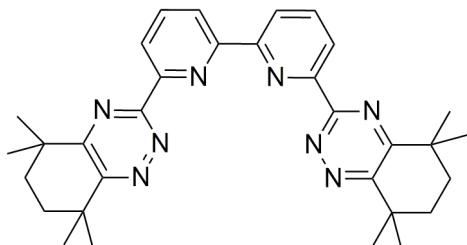


Fig. 1. Structure of CyMe₄-BTBP

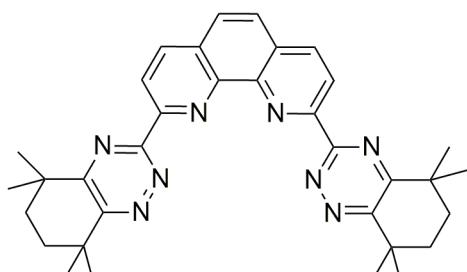


Fig. 2. Structure of CyMe₄-BTPhen

In this study, radiation stability of two ligands, CyMe₄-BTBP and CyMe₄-BTPhen were examined in system with the FS-13 (phenyl trifluoromethyl sulfone) diluent, to study influence of ionizing radiation on the separation process efficiency and safety. The solutions of both ligands were irradiated by accelerated electrons up to the absorbed dose of 200 kGy.

The irradiated samples were analysed by HPLC (High Performance Liquid Chromatography) and residual concentrations of tested ligands were determined. The effect of the presence of HNO₃ during the irradiation was studied.

Moreover, extraction properties of the irradiated solvents were evaluated and compared with the extraction properties of non-irradiated solvents to assess the impact of the degradation products on extractions properties.

HPLC analyses of irradiated samples of CyMe₄-BTBP and CyMe₄-BTPhen indicate that the stabilities of these ligands are higher in FS-13 than in the cyclohexanone-type of solvents².

The results of extraction studies showed that the extraction properties are significantly influenced by degradation products contained in these systems. Surprisingly, the distribution ratios for both americium and europium increase with the absorbed dose for the system with CyMe₄-BTPhen in FS-13. Obviously, the degradation products of this ligand are efficient extractants, too.

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9P-02

THERMAL ANALYSIS OF STEEL

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Structure and properties of steel alloys is object of extensive research^{1,2}. Unfortunately, a lack of experimental data about these systems still persists. Some information can be found in the literature, but these data are usually incomplete³. Due to necessity to use exact experimental data as an input data for many simulation programs, numerical or physical models and requirements of practice (i.e. casting, welding), an investigation of steel alloys is highly topical theme.

Missing data, such as temperatures of phase transformations, heat capacity and others can be obtained using thermal analysis measurements⁴. The DTA (Differential Thermal Analysis) is one of the possible methods. Another

common method is the DSC (Differential Scanning Calorimetry).

Also, missing data can be calculated using simulation programs. As for all calculations, it is necessary to validate its results with exact experimental data, i.e. density, enthalpy, heat capacity and other material data⁵. It is also suitable to support these data by structural and phase analysis.

In this paper, temperatures of phase transformations, latent heat (of melting), heat capacity, linear coefficient of thermal expansion and surface tension of one real steel grade were obtained. Also, theoretical calculation of properties of this steel grade was performed and compared with experiments.

Calculated temperatures, except for temperature of eutectoid transformation, are in good agreement with experiments. Calculated latent heat and heat capacity are in very good agreement with experiments. The agreement between measured and calculated linear coefficient of thermal expansion and surface tension is also good.

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1P-19

MOŽNOSTI VYUŽITIA PRIETOKOVEJ COULOMETRIE AKO METÓDY NA STANOVENIE VYBRANÝCH ANIÓNOV

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Úlohou prezentovanej práce bolo štúdium možností aplikácie prietokovej coulometrie ako coulometrickej titrácie. Metóda sa použila na analýzu fosforečnanov vo vzorkách odpadových vôd. Princíp stanovenia spočíval vo vzniku molybdofosfátu a jeho následnej jednoelektrónovej redukcii. Fosforečnany po reakcii s molybdénanom amónnym vytvárajú komplexnú zlúčeninu. Podľa literatúry¹ je pomer P : Mo v zlúčenine 1:12. Takto viazaný šesťmocný molybdén sa elektrochemicky redukuje na päťmocný pri rozdielnom potenciáli ako molybdén neviazaný do soli heteropolykyseliny.

Podľa množstva takto zredukovaného molybdénu je možné stanoviť obsah fosforečnanov. Vysoký pomer viazania

molybdénu a fosforu zaraďuje danú metódu medzi veľmi citlivé metódy kvantitatívnej analýzy fosforečnanov vo vodách³. Fosfor sa vo vodách vyskytuje prevažne vo forme rôznych fosforečnanov: anorganické fosforečnany, polyfosforečnany a organický viazaný fosfor. Metódami, ktoré sú v bežnom laboratóriu dostupné, stanovenie jednotlivých skupín zlúčenín, nie je možné dosiahnuť celkom exaktné rozdelenie. Všetky skupiny zlúčenín fosforu stanovujeme tak, že sa prevádzajú na rozpustné anorganické orthofosforečnany, ktoré sa potom stanovujú absorpciou spektrofotometriou².

Prezentovaná metóda je pri optimálnych podmienkach aplikovateľná v koncentračnom rozsahu $1,5 \cdot 10^{-6}$ až $5,5 \cdot 10^{-5}$ mol/dm³. Medza detekcie metódy je $3,42 \cdot 10^{-7}$ mol dm⁻³. Problém pri stanovení celkového obsahu fosforečnanov spočíval v mineralizačnom kroku. Ak sa mineralizácia nepreviedla, stanovili sa len anorganické rozpustné fosforečnany. Ide o novú metódu, ktorá je charakteristická svojou prístrojovou a manipulačnou jednoduchosťou. To je predpoklad jej ďalšieho využitia v oblasti stopovej analýzy³. Skutočnosť, že je to absolútна metóda, zaraďuje prietokovú coulometriu medzi spoľahlivé a nezávislé metódy.

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