Rapid Extraction and Simultaneous Spectrophotometric Determination of U(VI) with N-p-Tolyl-o-methoxy Benzohydroxamic Acid

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Rasche Extraktion und spektralphotometrische Bestimmung von Uran(VI) mit N-p-Tolyl-o-methoxy-benzohydroxamsäure

Best. von Uran(VI) mit Tolylmethoxybenzohydroxamsäure; Spektralphotometrie

N-p-Tolyl-o-methoxy benzohydroxamic acid (TMBHA) is proposed as a reagent more sensitive and selective than N-phenyl benzohydroxamic acid [3] or N-phenyl-2-naphthohydroxamic acids [1], the two analogues of TMBHA which have so far been used for the liquid-liquid extraction and spectrophotometric determination of U(VI).

TMBHA can be readily prepared in good yield and is stable towards storing [2]. It forms an orange-yellow complex with U(VI) which can be extracted into chloroform, between pH 3.9–4.6. The extract has $\lambda_{\max}$ at 360 nm ($\epsilon = 2.1 \times 10^3$) and 520 nm ($\epsilon = 290$). Beer's law is obeyed in the range 1.2–12 $\mu\text{g}/\text{ml}$ at 360 nm and 8–90 $\mu\text{g}/\text{ml}$ at 520 nm. The method can thus be used for separation and determination of U(VI) at both microgram and milligram levels. A 100-fold excess of reagent to U(VI) is adequate for complete extraction within 5 min of equilibration. Further excess of reagent is permissible.

The U(VI) is easily back-stripped from the chloroform extract into 0.1 M H_2SO_4 solution and it is possible to cyclise the extraction and stripping processes in such a way that large quantities of U(VI) can be extracted with very little loss of reagent solution.

The presence of following ions in 10-fold the amount of U(VI) has no influence: Hg^{2+} , Co^{2+} , Ba^{2+} , Ca^{2+} , Cd^{2+} , Zn^{2+} , Mn^{2+} , Al^{3+} , As^{3+} , Ga^{3+} , Ge^{4+} , ClO_4^- , NO_3^- , SO_4^{2-} . Following ions are tolerated in a 20-fold excess Ni^{2+} , Mg^{2+} , Pb^{2+} , Cl^- , Br^- . The presence of Ti^{4+} , Ce^{4+} , Mo^{4+} and Zr^{4+} causes interference but it can be removed by prior extraction of these metals with TMBHA from solutions 0.15 N in HCl.

Procedure. Adjust the pH of the uranium solution, containing 15–900 μg of U(VI), between 3.9–4.6 with dil. HNO_3 or NaOH, and extract with two 4 ml portions of the reagent solution, 0.1 M in chloroform, for 5 min each. Transfer the organic layers after drying with Na_2SO_4 , to a volumetric flask, dilute to 10 ml and measure the absorbance at 360 nm or 520 nm. Set up a calibration curve accordingly.

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