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Chemistry

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Abstract

Full Text

Chemistry

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Extraction of Niobium Pyrrolidinedithiocarbamate

Derivatives of dithiocarbamic acid are valuable analytical reagents for rare elements. Among this group of compounds, sodium diethyldithiocarbamate ($\text{Na} = \text{DDTC}$) has been well studied. As for other derivatives, they have been studied insufficiently. Among the little-studied ones is ammonium pyrrolidinedithiocarbamate ($\text{NH}_4 = \text{PDTC}$). This reagent is more stable in aqueous solutions than $\text{Na} = \text{DDTC}$. It has been used ⁽¹⁾ for the gravimetric determination of niobium and for separating it from tantalum by precipitation. We have for the first time studied the conditions for quantitative precipitation of niobium with $\text{NH}_4 = \text{PDTC}$ and the extraction of the compound formed by various organic solvents immiscible with water.

Experimental part. The reagent was synthesized by us from pyrrolidine ⁽²⁾; tetrahydrofuran ⁽³⁾ was used as the starting substance.

$\text{NH}_4 = \text{PDTC}$, recrystallized from alcohol, is a white crystalline substance melting at 128-130°, readily soluble in water and alcohol, and practically insoluble in organic solvents (CHCl_3 , CCl_4 , etc.).

Niobium solutions were prepared by fusing niobium pentoxide containing the radioactive isotope Nb-95 with a tenfold amount of potassium pyrosulfate and dissolving the melt in 2-3% tartaric, citric, and oxalic acids. The concentration of Nb_2O_5 was ~ 1 mg/ml, and the radioactivity was 4500-5500 counts/min/ml.

Fig. 1. Extraction of niobium pyrrolidinedithiocarbamate with chloroform (2-5 mg Nb_2O_5); **1**—tartaric-acid, **2**—oxalic-acid, **3**—citric-acid solutions.

Experiments with different amounts of Nb_2O_5 (2-30 mg) showed that niobium pyrrolidinedithiocarbamate ($\text{Nb} = \text{PDTC}$) is precipitated quantitatively only from tartaric-acid and oxalic-acid solutions in the form of a white amorphous

precipitate by a 20-fold excess of reagent in the presence of an acetate buffer at pH 4-5.

In concentrated hydrochloric acid, NH_4 -PDTK forms an orange-red compound with niobium. We studied the extraction of Nb-PDTK with many organic solvents at various hydrogen-ion concentrations. The best solvent is chloroform. The dependence of extraction with chloroform on the acidity of the solutions is shown in the figure, from which it is evident that Nb-PDTK is quantitatively extracted in a weakly acidic medium (pH 4-5) and in concentrated HCl (8.5-10 M). Citric acid, as well as fluoride and sulfate ions, hinder the extraction of Nb-PDTK.

For quantitative recovery of niobium, the extraction must be repeated 2-3 times at 20-30 min after the addition of a 15-20-fold excess of the dry reagent; the shaking time is 2-3 min. NH_4 -PDTK does not react with tantalum, titanium, zirconium, tungsten, beryllium, rare earths, and other elements, and can be used for their separation from niobium by the extraction method.

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REFERENCES CITED

1. H. Malissa, *Mikrochim. Acta*, No. 6, 726 (1958).
2. Yu. K. Yur' ev, *Scientific Notes of Moscow University*, issue 79, 32, 88 (1945).
3. H. Malissa, E. Schoffmann, *Mikrochim. Acta*, No. 1, 180 (1955).

Note: Figure translations are in progress. See original paper for figures.

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