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Solvent Extraction Separation of Chromium(VI), Iron(III), Cobalt(II), and Nickel(II) with Di-*n*-pentyl Sulfoxide

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NOTE

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Abstract

Di-*n*-pentyl sulfoxide (DPSO) was employed for the solvent extraction separation of some transition metals from hydrochloric acid medium. It was found that chromium(VI) gets extracted at low acid concentrations (3 to 4.5 *M*), and iron(III) and cobalt(II) only above 5 *M* HCl. Nickel was not extracted at any of the test conditions. These transition metals were separated from one another by suitable choice of the extraction conditions.

GENERAL PROCEDURE

The transition metal solutions were prepared in required acidities such that the initial concentration was 0.002 *M* with respect to chromium(VI), iron(III), and nickel(II), and 0.01 *M* with respect to cobalt(II). The aqueous phase was equilibrated with an equal volume of di-*n*-pentyl sulfoxide (DPSO) in carbon tetrachloride at 30 ± 1 °C for 10 min. The extraction equilibrium was attained within 3 min for iron and cobalt. In the extraction of chromium(VI), the metal gets reduced to chromium(III) above 6 *M* HCl and for longer contact periods. The extraction studies on chromium(VI) were made over the range 1 to 5 *M* HCl for a contact period of 10 min. Chromium(III) was not extracted under the present experimental conditions. The phases were allowed to settle and separate. The chromium content was estimated by the absorptiometric method as its diphenyl carbazide complex (*1*), iron by the thiocyanate method (*1*), cobalt

by the spectrophotometric (*2*) or tracer technique (using ^{60}Co), and nickel by the β-activity counting technique.

RESULTS AND DISCUSSIONS

Figure 1 presents the results on the dependence of the extraction of chromium, iron, and cobalt by DPSO in carbon tetrachloride on the acid concentration. It is seen that chromium is well extracted even at low acid concentrations. The distribution of iron and cobalt starts at about 5 *M* HCl, reaches a maximum at about 8.5 *M*, and decreases thereafter. However, the extraction of cobalt is considerable at higher acid and extractant concentrations. Nickel is not extracted under these experimental conditions.

The effect of salting-out agent (LiCl) for the extraction of the metals is in the order chromium > iron > cobalt. For example, at 0.5 *M* HCl in the presence of 4.7 *M* LiCl, chromium alone is extracted (52%) while at higher concentrations of the salt (6.5 *M*) iron gets extracted (60%) by 0.02 *M* DPSO in carbon tetrachloride. Cobalt is extracted (60%) only at very high LiCl (9 *M*) and DPSO (0.5 *M*) concentrations.

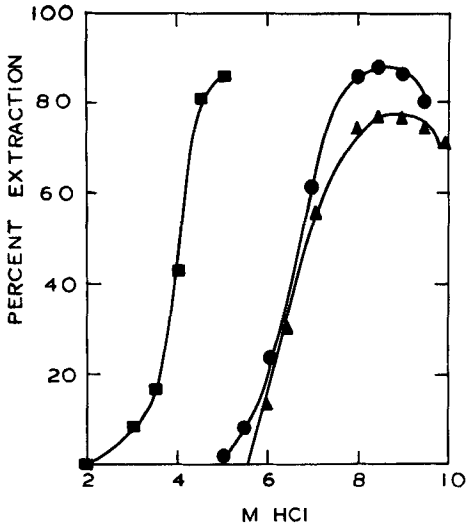

FIG. 1. Extraction as a function of acid concentration: (■) chromium by 0.02 *M* DPSO, (●) iron by 0.02 *M* DPSO, and (▲) cobalt by 0.5 *M* DPSO in carbon tetrachloride.

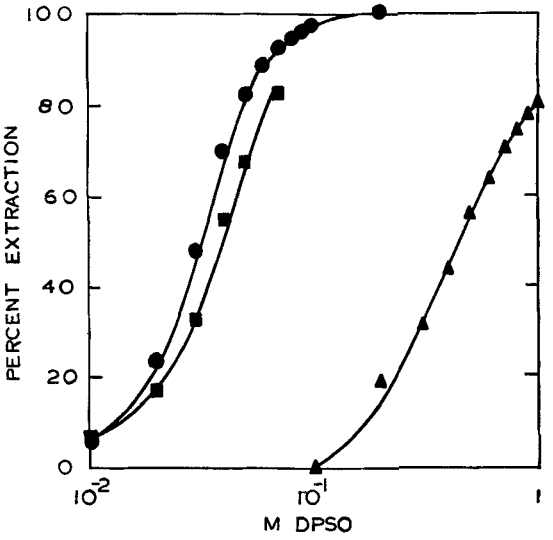

FIG. 2. Extraction as a function of di-*n*-pentyl sulfoxide (DPSO) concentration in carbon tetrachloride: (■) chromium from 3.5 *M* HCl, (●) iron from 6 *M* HCl, and (▲) cobalt from 7 *M* HCl.

The variation of extraction with different concentrations of DPSO in carbon tetrachloride for the extraction of chromium from 3.5 *M* HCl, iron from 6 *M* HCl, and cobalt from 7 *M* HCl is presented in Fig. 2.

Separation of the Metals

An aqueous phase 3.5 *M* in HCl containing a mixture of chromium, iron, cobalt, and nickel (0.002 *M* each) was made. The solution was labeled with ^{51}Cr, ^{59}Fe, ^{60}Co, and ^{63}Ni. The aqueous phase was then extracted repeatedly 6 times with an equal volume of 0.05 *M* DPSO in carbon tetrachloride using a fresh solution of the extractant every time. After each extraction the phases were analyzed for chromium, iron, and cobalt by taking the γ-energy spectrum using a 400 channel analyzer with an 8 cc Ge(Li) detector. The separation of chromium is complete in 5 runs. The absence of the energy maxima in the organic phase corresponding to ^{59}Fe and ^{60}Co in the γ-energy spectrum under these conditions shows the nonextraction of iron and cobalt.

After the removal of chromium, the aqueous acidity was increased to

7 *M* HCl and the extraction procedure repeated 5 times with 0.05 *M* DPSO. Iron is completely removed in 3 runs. Cobalt is then separated from nickel by repeating the solvent extraction procedure with 0.5 *M* DPSO. Cobalt is extracted quantitatively in 3 runs. The β-activity of ^{63}Ni in the aqueous phase remains the same before and after extraction of chromium, iron, and cobalt. This shows that nickel practically remains in the aqueous phase without being extracted.

Application

Cochrome alloy (chromium, iron, and cobalt), austenitic stainless steel (chromium, iron, and nickel), invar, and platinite (iron and nickel) alloys can be analyzed by this procedure.

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