EXTRACTION OF GROUP VIII ELEMENTS WITH 1-PHENYL-3-METHYL-4-TRIFLUOROACETYL-PYRAZOLONE-5

S. M. HASANY,* IMTIAZ HANIF

Nuclear Chemistry Division, Pakistan Institute of Nuclear Science and Technology, P. O. Nilore, Rawalpindi (Pakistan)

(Received December 13, 1977)

The extraction of group VIII elements with 1-phenyl-3-methyl-4-trifluoracetylpyrazolone-5 from aqueous solutions in chloroform has been studied as a function of pH. Fe(III), Ni(II), Pd(II) and Rh(III) show partial extraction whereas Co(II) is extracted quantitatively. The effect of citrate, cyanide, fluoride, iodide, thiosulphate and thiourea on the extraction of metal ions has been investigated. Back-extraction studies were carried out to strip metal ions from organic phase into appropriate aqueous solutions. Based on these findings some useful analytical separations have been proposed and a few of their possible applications have been discussed.

Introduction

In recent years a great deal of interest has been manifested in solvent extraction for the separation and concentration of metals and their quantitative determination. Most versatile and widely used chelating agents are $\beta$-diketones. In their enolic form they have a hydrogen replaceable by a metal and a ketonic oxygen for coordination. In our laboratory, 1-phenyl-3-methyl-4-trifluoracetylpyrazolone-5 (FAP) a fluorinated $\beta$-diketone, has been proved to be a useful extractant for certain metal ions.2,3 These investigations have further been extended and this communication describes the extraction study of group VIII elements with this reagent. The purpose of this study was to test the analytical potentiality of FAP for the metal ions involved and to evolve selective separations based on their extraction behaviour.

*Author to whom correspondence should be addressed.

J. Radioanal. Chem. 45 (1978) 115
Experimental

Reagents

FAP was synthesised and purified as reported earlier. A stock solution of FAP (0.1% w/v) in absolute ethanol was prepared and stored in an amber bottle. Buffer solutions of pH 1 to 5.9 were prepared with either 1M hydrochloric acid and 1M sodium acetate or with 0.2M acetic acid and sodium acetate solutions. For pH 5.8 to 8, 0.2M of potassium dihydrogen phosphate and sodium hydroxide and for pH 7.8 to 10 buffers, boric acid, potassium chloride and sodium hydroxide solutions were used.

All chemicals used in this study were of either C.P. or AnalyR grade. Doubly distilled deionised water was used throughout.

Radiotracers

$^{60}$Co was obtained from the Radiochemical Centre, Amersham, England. All other radiotracers used were prepared by neutron irradiation of their respective spec-pure metals or salts in PARR-I reactor of this institute at a flux of $2 \times 10^{13} \text{n} \cdot \text{cm}^{-2} \cdot \text{sec}^{-1}$. The radiochemical purity of the tracers was checked by gamma-spectrometry.

Apparatus

The solutions were agitated with a wrist-action Griffin Flask Shaker in a 35 ml capacity glass vial equipped with a polythene-lined screw cap. The pH measurements were made with a Pye model 79 pH meter using glass and calomel electrode. Gamma-ray spectrometry was carried out with a Nuclear Data model ND-4410 computerized multichannel analyzer coupled with a 30 cm$^3$ Ge(Li) detector. Gross gamma-ray activities were assayed with the aid of a Nuclear Chicago model 8725 well-type scintillation counter using a 3" X 3" NaI(Tl) crystal. Beta-activities were measured with a Nuclear Chicago model 402 GM-counter.

Extraction procedure

To a vial containing 4 ml buffer solution, 100 µl radiotracer of the test element was added and mixed uniformly. The amount of the carrier in the aliquot of the tracer was always less than 100 µg. One ml FAP solution was added and the solution was allowed to stand for 15 min. to ensure the complete reaction between metal and the reagent. After adding 4 ml of chloroform, the contents were shaken vigorously for 5 min, a sufficient time for equilibration, then centrifuged for phase separation. After the separation of two phases, the activity of one ml aliquot of