Cupric (di) glycinate was prepared from glycine and a small excess of freshly precipitated and washed basic cupric carbonate. It was purified by repeated crystallization from water and obtained as the monohydrate. The solutions were analyzed on a Beckman spectrophotometer. The composition of the crystals was determined by iodometric titration for copper. At 25° the solubility was 0.0364 *M* and the molar extinction coefficient at 6300 Å. was 46. The molar conductivity was 2.8×10^{-6} mho cm.$^{-1}$ indicating very slight dissociation into ions.

Values of *D* obtained at 0.00917 *M* and 0.0274 *M* were not significantly different and the pooled results gave a normal cumulative probability distribution. The mean value of *D* at 25.0 was $0.723 \pm 0.004 \times 10^{-5}$ cm.^{2} sec.$^{-1}$. The indicated error is the 95% confidence range for the mean.[5] The molar volume was found from density measurements to be 100.4 cm.^{3} and independent of concentration. *D* calculated from the Einstein–Sutherland equation assuming a sphere is 0.717×10^{-5}. If approximate corrections for departures from a spherical shape are applied *D* is calculated to be 0.67×10^{-7}.[6]

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The Distribution of the Subgroup VII Elements between Pyridine and 4 *N* NaOH[1]

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In the course of studies directed toward the development of a new method for isolating technetium activities from meson irradiated silver samples, extraction from basic media with pyridine was attempted. It appeared reasonable to try this in view of the fact that permanganate has been found to be extractable under these conditions.[3] A preliminary experiment with carrier-free $NH_4Tc^{99}O_4$ indeed showed that pyridine extracted pertechnetate almost quantitatively from 4 *N* NaOH. The following is a report of the results of the distribution measurements which were made. The distribution of +7 rhenium and of +7 manganese between the same liquids was also studied.

Experimental

Perrhenate.—Measured volumes of 4 *N* NaOH containing known amounts of NH_4ReO_4 were equilibrated with measured volumes of pyridine saturated with 4 *N* NaOH. The layers were analyzed by acidifying to about 4 *N* with HCl, heating almost to boiling, precipitating Re_2S_7 by passing in hydrogen sulfide,[4] dissolving the collected precipitate in a 1:1 mixture of 6 *N* NaOH and 30% H_2O_2, and gravimetrically determining the amount of rhenium by conversion to the tetraphenylarsonium derivative.[5] In each case the sulfide precipitation was repeated on the filtrate from the first sulfide precipitation after first diluting with 4 *N* HCl, until no more sulfide was precipitated.

Technetium.—Carrier-free $NH_4Tc^{99}O_4$ solutions were extracted with pyridine exactly as in the perrhenate extractions. Measured volumes of standardized NH_4ReO_4 solution were added to the separated layers and the sulfide precipitated[6] and determined as the tetraphenylarsonium derivative as before. The tetraphenylarsonium perrhenate precipitates carrying the pertechnetate[7] were measured with a Geiger counter for their Tc^{99} content.

Manganese.—A small measured volume of a freshly prepared dilute solution of $KMnO_4$ was poured into a centrifuge bottle containing some pyridine. After shaking for a few seconds 4 *N* NaOH was poured into the bottle, the mixture again shaken for a few seconds and then immediately centrifuged to hasten the separation of the layers. Speed was essential to minimize the decomposition of the permanganate to form the green manganate. The alkaline layer was siphoned off and a measured portion of it was reextracted with a measured volume of pyridine. A measured volume of the first pyridine extract was diluted with pyridine until a color match was obtained visually between the diluted solution and the second pyridine solution with the two solutions in long test-tubes filled to the same depth.

The results obtained are summarized in Table I.

TABLE I

DISTRIBUTION BETWEEN PYRIDINE AND 4 *N* NaOH

Element, M	Distribution coefficient, $\frac{[M]_{Py}}{[M]_{aq}}$	$[M]_{Py}$, (mg. of M/ml.)
Re	255	3.8
	195	7.4
	130	12.8
	30	13.5
Tc	778	3.8×10^{-5}
Mn	8000	5

It is clear from Table I that all the subgroup VII elements in their heptavalent states are extractable into pyridine from strongly alkaline aqueous solution.

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[1] This research was supported in part by the Office of Scientific Research of the Air Research and Development Command of the U. S. Air Force.

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Polarographic Behavior of Organic Compounds. XIII. Chloroacetic Acids and their Ethyl Esters. *p*H-Independence of Carbon–Halogen Bond Fission. Wave Splitting Due to Buffer Interaction

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In view of the S-shaped relation between *p*H and $E_{1/2}$ for the polarographic reduction of acids of various types[1,2] including those involving carbon–halogen bond fission, the related system of acid–

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