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Solvent Extraction Behavior and Mutual Separation of Some Bivalent Metal Ions Using Thiocarbohydrazide as the Complexing Agent

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NOTE

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Abstract

Thiocarbohydrazide ($NH_2 . NH . CS . NH . NH_2$) was used as a complexing agent for the solvent extraction separation of some bivalent metals. Separation of Cd from Co, Cu, and Pd, and of Pb from Ni and Pd was carried out using the effect of pH on their extractability. The separation of Cu from Zn and Hg, and of Pb from Zn and Cd was also carried out using various masking agents.

In an earlier communication the authors reported the mutual solvent extraction separation of zinc (II), cadmium (II), and mercury (II) using thiocarbohydrazide (TCH)—the precursor of dithizone—as the complexing agent (**1**). The studies were further extended to other bivalent metal ions, and the extraction behavior of cobalt (II), nickel (II), copper (II), palladium (II), and lead (II) complexes has been investigated.

GENERAL PROCEDURE

The distribution ratios in the case of the extraction of cobalt (II) complex were found by using ^{58}Co as tracer in a manner similar to the one described earlier (*1*). The distribution ratios in all other cases were found

by determining the amount of metal ion in the organic layer after destroying the organic material by standard procedures. The metal ion content was determined by using a suitable spectrophotometric method. The amount of metal ion in the aqueous layer was determined by difference from the known initial total metal ion concentrations which varied from 30 to 60 ppm. In cases where extractions were poor, the metal ion concentration was determined in the aqueous layer to minimize the error in determination. The distribution ratios showed a standard deviation of approximately 2% in each case at room temperature (30 ± 2°C).

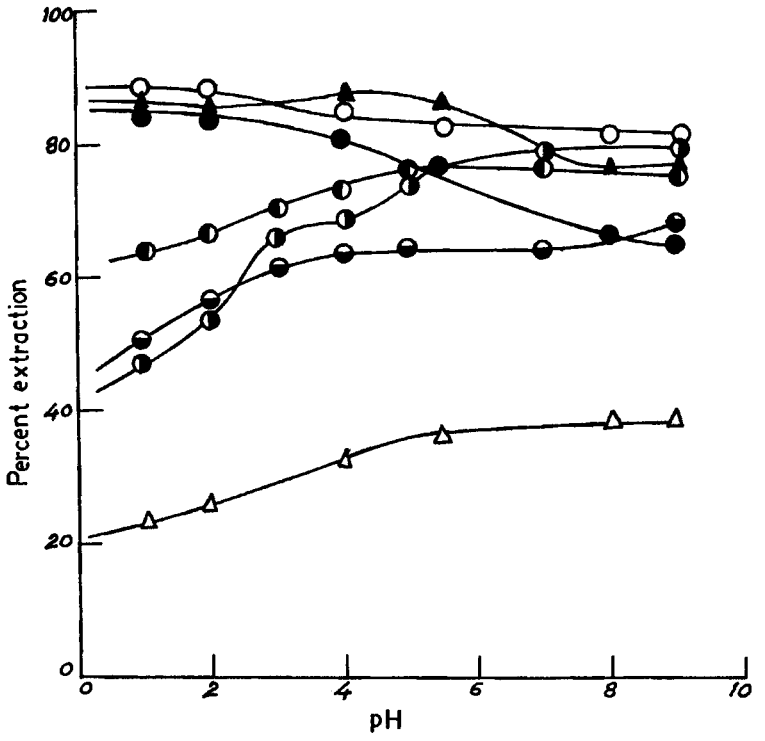

FIG. 1. Extraction behavior of Co(II)–TCZ complex in different solvents at various pH values: (●) benzene, (○) chloroform, (◒) ethyl acetate, (◑) amyl acetate, (◐) amyl alcohol, (▲) carbon tetrachloride, and (△) diisopropyl ether.

RESULTS AND DISCUSSION

Separations Based on pH Effect

The results of pH effect on percent extraction of cobalt (II), nickel (II), copper (II), palladium (II), and lead (II) complexes of TCH in various solvents are given in Figs. 1 through 5. The blanks (which were $\leqslant 5\%$ in each case) are not shown in these figures. None of the complexes shows any preferential extraction in either oxygenated or nonoxygenated solvents. Based upon their extraction behavior in different solvents, cadmium (II) (*I*) can be separated from cobalt (II), copper (II), and palladium (II), and lead (II) can be separated from nickel (II) and palladium (II). The

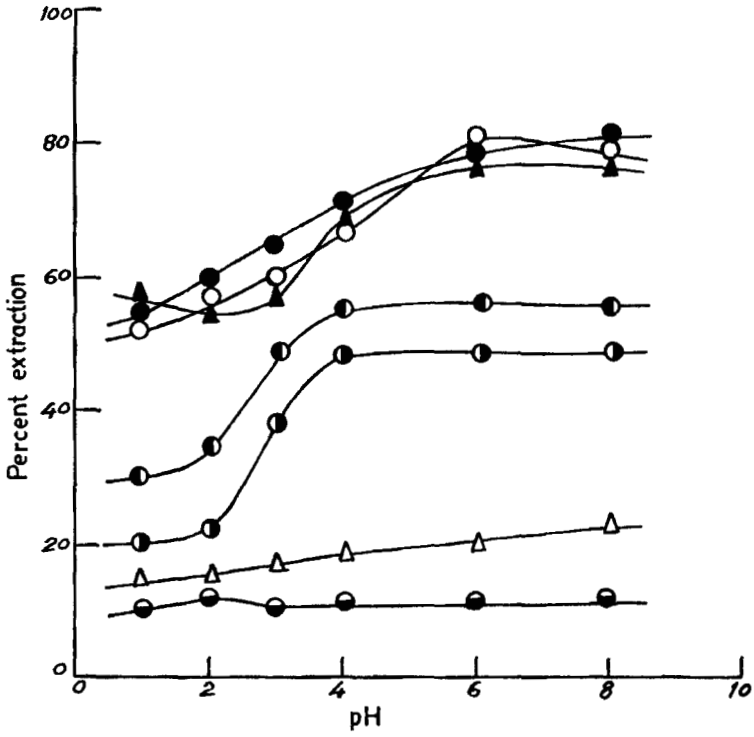

FIG. 2. Extraction behavior of Ni(II)–TCZ complex in different solvents at various pH values. Symbols as in Fig. 1.

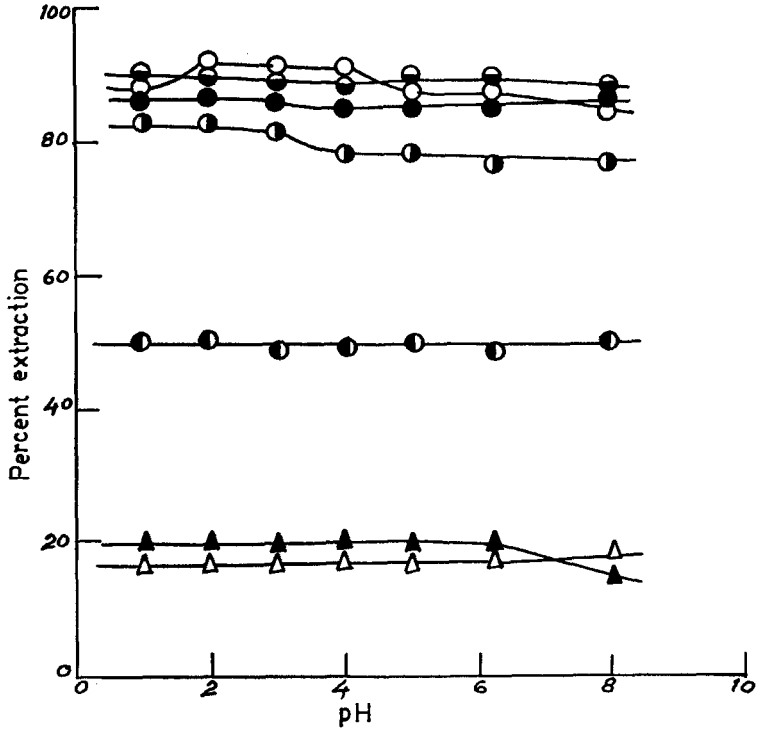

FIG. 3. Extraction behavior of Cu(II)–TCZ complex in different solvents at various pH values: (●) benzene, (◒) chloroform, (○) carbon tetrachloride, (◐) amyl alcohol, (◑) amyl acetate, (▲) diisopropyl ether, and (△) ethyl acetate.

results outlining procedural conditions and the effectiveness of separation in each case are given in Table 1. Though the values for the separation factor S are not very high, they deviate considerably from unity to provide fairly good separations.

Separations Based upon the Use of Masking Agents

The effect of those anions which possibly could suppress the extraction was investigated in each case in benzene. The results are given in Table 2. Values of average percent extraction given under Column 3 of Table 2 are those where none of the masking agents was present. For the sake

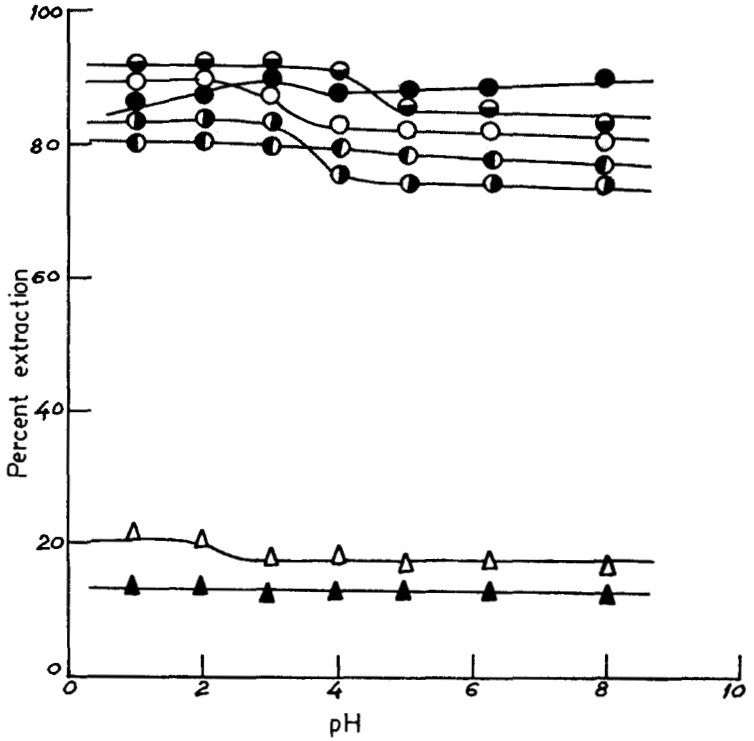

FIG. 4. Extraction behavior of Pd(II)–TCZ complex in different solvents at various pH values: (●) benzene, (◒) chloroform, (○) carbon tetrachloride, (◑) amyl alcohol, (◐) amyl acetate, (▲) diisopropyl ether, and (△) ethyl acetate.

of comparison, data on the extraction of Zn (II), Cd (II), and Hg (II) complexes (*1*) are also given.

From these results it is evident that some separations can be carried out using various masking agents. The results outlining procedural conditions and the effectiveness of each separation are given in Table 3.

Acknowledgments

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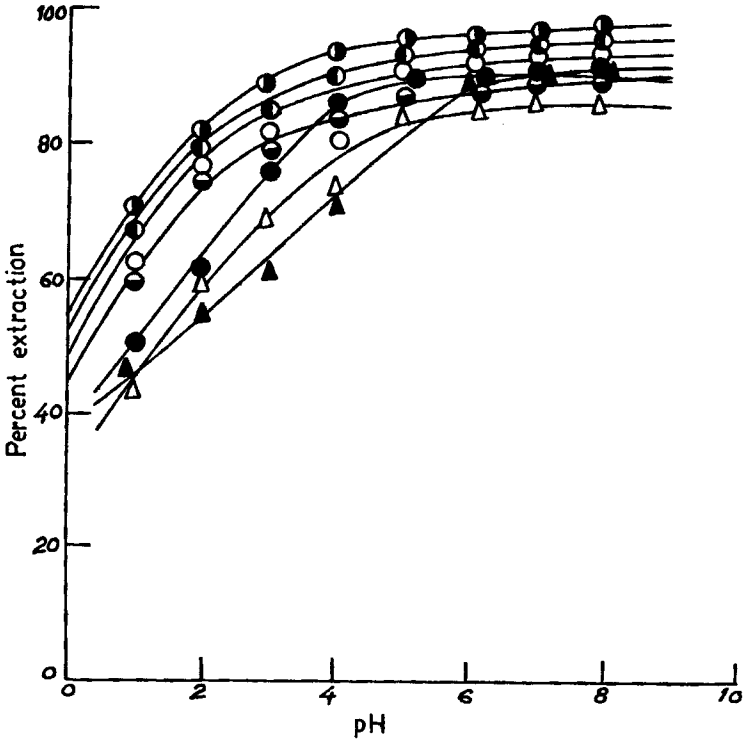

FIG. 5. Extraction behavior of Pb(II)–TCZ complex in different solvents at various pH values. Symbols as in Fig. 4.

TABLE 1

Separations Based upon the Effect of pH on Extraction

Metal ion	Separated from	S	pH	Solvent
Co	Cd	100	1.0	Benzene
	Cd	150	1.0	Chloroform
	Cd	127	1.0	Carbon tetrachloride
Cu	Cd	110	1.0	Benzene
	Cd	175	1.0	Chloroform
	Cd	143	1.0	Carbon tetrachloride
Pd	Cd	131	1.0	Benzene
	Cd	160	1.0	Chloroform
	Cd	185	1.0	Carbon tetrachloride
	Cd	95	8.0	Ethyl acetate
Pb	Ni	95	8.0	Ethyl acetate
	Pd	145	8.0	Ethyl acetate
	Pd	180	8.0	Ethyl acetate

TABLE 2

Effect of Masking Agents on the Extractability of Various Complexes

Metal ion	pH	Average percent extraction	Percent extraction in presence of									
			EDTA	CN^-	SCN^-	F^-	Br^-	I^-	$S_2O_3^{2-}$	Oxalate	Citrate	Tartrate
Co	1.0	84.0	43.0	11.0	17.0	13.5	9.5	8.0	60.5	83.5	86.0	85.5
Ni	8.0	81.5	7.0	6.0	19.0	61.0	43.0	36.0	59.0	83.0	80.0	84.5
Cu	1.0	84.5	17.5	11.5	47.0	81.0	84.5	83.0	3.0	76.5	74.0	79.0
Pd	3.0	89.5	17.0	11.0	35.5	81.5	80.0	43.5	27.5	85.0	87.5	82.5
Pb	8.0	90.5	37.5	59.5	29.0	86.5	39.0	33.5	3.5	13.5	7.5	3.0
Zn	8.0	88.0	23.0	20.5	56.0	26.5	17.0	13.5	89.0	47.0	5.0	4.5
Cd	8.0	90.5	46.5	56.0	80.5	88.0	87.5	80.0	3.0	88.5	89.0	89.5
Hg	8.0	82.5	67.0	40.0	51.5	62.5	9.5	7.0	3.0	83.0	81.5	82.5

TABLE 3

Separation of Some Bivalent Metal Ions Based on the Use of Various Masking Agents and Employing Benzene as the Organic Phase

Metal ion	Separated from	S	pH	Masking agent
Cu	Hg	89	1.0	Potassium iodide
Zn	Cu	158	8.0	Sodium thiosulfate
	Pb	169	8.0	Sodium thiosulfate
Cd	Pb	167	8.0	Sodium citrate
	Pb	187	8.0	Sodium tartrate

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