Studies in the Two Phases System Sodium Formate-Pyridine. Extraction of Nickel and Separation from Chromium

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Chloroform[1-4] and benzene[5-8] have been widely used for the extraction of nickel and its separation from many elements. A new two phase system sodium formate-pyridine[9] has been reported from these laboratories recently. The behavior of nickel in this system, its quantitative extraction by the batch extraction technique and its estimation in presence of chromium have now been studied.

Experimental

Reagents.—Nickel chloride (B. D. H. AnalaR) sample), sodium formate (Reidel de Haen's recrystallized sample), Baker's analyzed pyridine and sodium chromate (E. Merck's AnalaR sample) are used.

Effect of various factors like the volume of pyridine, molarity of sodium formate, pH and the metal ion concentration on the extraction of nickel have been studied in detail.

Effect of Volume of Pyridine.—Five milliliters of sodium formate containing 1 ml. 0.1 N nickel chloride is taken, various volumes of pyridine are added each time and the amount of nickel extracted into pyridine after equilibration is determined. It is found that with 2 ml. pyridine, both the layers tend to separate but it is not possible to effect a clear separation of the two immiscible phases. The percentage extraction (% E) increased with the volume of pyridine upto 5 ml. and remained constant with a further increase in the volume of pyridine (Fig. 1) and therefore in all the cases 5 ml. of pyridine are used.

Effect of Formate Concentration.—Formate concentration has got very little effect on the amount of metal extracted into the pyridine and its strength need not be rigorously adjusted provided its molarity is above 3 M. At and below 2.3 M formate mixes freely with pyridine and no separation of layers occurs.

Effect of pH.—pH has got a profound influence on the amount of nickel extracted into the pyridine layer. The amount of metal extracted into the organic phase remained constant over a pH range of 7.5~9.2, but on further increase of pH, the percentage extraction decreased markedly. These results are given in Fig. 2.

Effect of Nickel Concentration.—Maintaining optimum conditions of pH, concentration of formate

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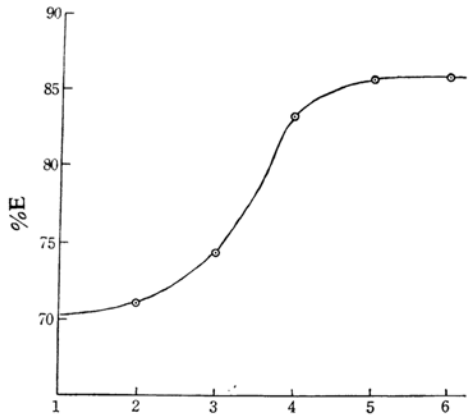

Fig. 1. Effect of volume of pyridine.

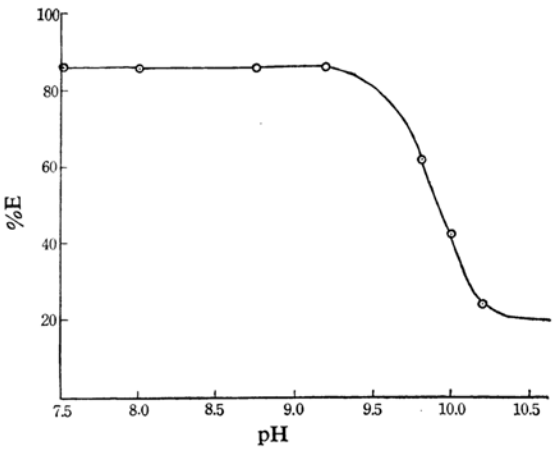

Fig. 2. Effect of pH on nickel extraction.

and pyridine, nickel solution of various concentrations are added each time to the system and the amount extracted into the pyridine is determined in each case. In all the cases the distribution coefficient is found to be constant, thus proving the validity of Nernst's distribution law (Table I).

Table I. Effect of nickel concentration on the extraction

System: 5 ml. of pyridine + 5 ml. 3 M formate containing $NiCl_2$, pH=8

S. No.	Amount of nickel added mg.	Amount of nickel extracted mg.	D
1	0.2	0.172	6.14
2	0.4	0.345	6.27
3	0.6	0.515	6.05
4	0.8	0.690	6.27
5	1.0	0.860	6.14

Procedure for the Extraction of Nickel.—From the study of all the above variables it is concluded that maximum extraction is obtained when the formate concentration is 3 M and 5 ml. of pyridine are used for the extraction, the pH range being 7.5~9.2. To an aliquot of sodium formate (necessary to give 5 ml. of 3 M solution when diluted) nickel chloride solution is added and the volume is made upto 5 ml. in a separating funnel. Five milliliters of pyridine is now added and the whole mixture is equilibrated for 2 min. The formate layer is collected into another separating funnel to which further 5 ml. of pyridine is added to extract some more nickel which remain in the formate during the first extraction. The process is repeated once more so as to get an almost complete extraction of nickel into pyridine. All the pyridine extracts are collected together in 100 ml. conical flask, diluted with a little water and the pyridine is boiled off. The nickel content of this residue is determined polarographically using 1 M ammonium chloride and 0.1 M ammonium hydroxide as the supporting electrolyte[10].

Table II. Analysis of a synthetic mixture of nickel and chromium

Amount of nickel		Amount of chromium	
Taken mg.	Found mg.	Taken mg.	Found mg.
1.45	1.45	1.25	1.243
2.90	2.87	2.50	2.496
4.35	4.34	3.75	3.750
5.80	5.82	5.00	4.926
7.25	7.20	6.25	6.250

Thus a complete extraction of nickel by pyridine from sodium formate is achieved in a three stage batch extraction technique.

Preliminary experiments with chromium under identical experimental conditions showed that it remains completely in the formate layer and is not extracted into pyridine. The observation has been utilized for the separation of nickel and chromium.

Analysis of a Mixture of Nickel and Chromium.—A mixture of nickel and sodium chromate solutions containing various concentrations of nickel and chromium are taken to which the required quantity of sodium formate (necessary to give 5 ml. of 3 M solution) and 5 ml. of pyridine are added and the whole mixture is shaken thoroughly. The formate layer is transferred into another separating funnel and the remaining nickel is re-extracted with a further aliquot of pyridine. For a complete extraction a three stage batch extraction is thus carried out. All the pyridine extracts are mixed together and the nickel content was determined polarographically (vide infra) while the chromium in the formate layer is determined iodometrically[11].

Summary

The distribution of nickel in the two phase system, sodium formate-pyridine is studied in

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detail. The effects of pH, volume of pyridine and molarity of formate are studied and the optimum conditions for the complete extraction are established. The possibility of using this extraction technique for the separation of nickel and chromium has also been worked out and a procedure for the analysis of a synthetic mixture of nickel and chromium is given.

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