

THE ELECTROLYTIC PRECIPITATION OF NICKEL FROM PHOSPHATE SOLUTIONS.¹

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NICKEL has been quantitatively precipitated in the electrolytic way from various electrolytes.¹ The purpose of this particular investigation was to ascertain the exact conditions under which this metal could be deposited from phosphate solutions without carrying down phosphorus with it; also to effect, if possible, separations of nickel from other metals in the same electrolyte. The method pursued consisted in precipitating nickel sulphate solutions with varying amounts of disodium hydrogen phosphate of 1.038 specific gravity, dissolving the precipitate in phosphoric acid of 1.347 specific gravity, and after dilution with water, electrolyzing the solutions with varying current and pressure conditions.

The precipitations were made in platinum dishes—the cathodes, while flat platinum spirals were used as anodes. The deposits were treated as ordinarily directed in electrolytic work. The details of more than forty experiments appear in those recorded in the accompanying tables, with sufficient minuteness to enable any person, so desiring, to repeat them with the confidence of obtaining like satisfactory results.

EXPERIMENTS.

	1.	2.	3.	4.	5.	6.
N. D ₁₀₀	2.0	2.0	4.0	4.0	2.0	3.0
Volts	6	6	6	6	6	6
Dilution, cc.....	225	125	200	250	125	175
Temperature	50°	65°	65°	55°	65°	65°
Time, hours.....	7	7½	8¼	9½	6¾	8½
Na ₂ HPO ₄ , cc.....	90	90	130	180	90	135
H ₃ PO ₄ , cc	4.5	4.5	9.0	9.0	4.5	6.75
Nickel taken, gram .	0.2166	0.2166	0.4332	0.4332	0.2166	0.3249
Nickel found, gram .	0.2163	0.2161	0.4298	0.4321	0.2165	0.3253

¹ From the author's thesis for the Ph.D. degree.

² See Smith's "Electrochemical Analysis," p. 91, third edition, 1902.

	7.	8.	9.	10.	11.
N.D ₁₀₀	4.0	2.0	3.0	3.0	4.0
Volts	6	6	6	6	6
Dilution, cc	250	125	175	175	250
Temperature	55°	65°	70°	62°	55°
Time, hours	9½	7¼	9	9½	9¾
Na ₂ HPO ₄ , cc	180	90	135	135	180
H ₃ PO ₄ , cc	9.0	4.5	6.75	6.75	9.0
Nickel taken, gram	0.4332	0.2166	0.3249	0.3249	0.4332
Nickel found, gram	0.4327	0.2163	0.3247	0.3251	0.4321
	12.	13.	14.	15.	16.
N.D ₁₀₀	1.-1.75	1.4.-1.8	1.0-1.5	1.4-1.8	1.25
Volts	6	6	6	6	6
Dilution, cc	125	125	175	175	175
Na ₂ HPO ₄ , cc	90	90	90	135	90
H ₃ PO ₄ , cc	4.5	4.5	4.5	6.75	4.5
Nickel taken, gram .	0.2176	0.2176	0.2176	0.3264	0.2176
Nickel found, gram .	0.2179	0.2181	0.2176	0.3271	0.2180
Temperature	56°	50°	50°	50°	50°
Time, hours	7	7	7	9¼	7¼
	17.	18.	19.	20.	21.
N.D ₁₀₀	1.4-1.8	1.25-1.6	1.25-1.6	0.25-1.6	1.25-1.6
Volts	6	6	6	6	6
Dilution, cc	225	230	230	230	230
Na ₂ HPO ₄ , cc	135	180	180	180	180
H ₃ PO ₄ , cc	6.75	9.0	9.0	9.0	9.0
Nickel taken, gram .	0.3264	0.4352	0.4352	0.4352	0.4352
Nickel found, gram	0.3271	0.4353	0.4355	0.4353	0.4354
Temperature	50°	50°	50°	50°	50°
Time, hours	9	10	10	10	10

In every case except No. I and No. II the precipitation was complete. The success of the process depends greatly on keeping the dish covered and washing down its sides with water at frequent intervals, to supply the loss by evaporation and to prevent the phosphate of nickel from separating out on the dish.

These results need no further explanation. The deposits were equal in appearance, if not superior, to any deposits of nickel ever obtained in this laboratory. Those of 0.4352 gram of nickel, when dissolved in nitric acid and tested with ammonium molybdate, showed but a trace of phosphorus, which is without influence, for solutions electrolyzed by Fresenius and Bergmann's method¹ gave exactly the same amount of nickel.

The influence of elevation of temperature is shown in the following examples:

¹ *Ztschr. anal. Chem.*, 19, 320.

	22.	23.	24.
N.D ₁₀₀	2.4-2.8	2.25	1.80-2.75
Volts	6	6	6
Dilution, cc.....	125	175	225
Temperature	88°	88°	88°
Time, hours	2½	5	6¼
Na ₂ HPO ₄ , cc.....	90	135	180
H ₃ PO ₄ , cc.....	4.5	6.75	9
Nickel taken, gram.....	0.2176	0.3264	0.4352
Nickel found, gram.....	0.2167	0.3265	0.4331
	25.	26.	27.
N.D ₁₀₀	1.80-2.75	1.80-2.75	1.80-2.75
Volts	6	6	6
Dilution	125	125	175
Temperature	82°	80°	80°
Time, hours.....	3½	4	5½
Na ₂ HPO ₄ , cc.....	90	90	135
H ₃ PO ₄ , cc.....	6.75	5.6	9.1
Nickel taken, gram.....	0.2176	0.2176	0.3264
Nickel found, gram.....	0.2150	0.2161	0.3255

The precipitation was only complete in 23. If the conditions there given be adopted, satisfactory results can be obtained in a considerably shorter time than is required when working at the ordinary temperature.

The separation of nickel from manganese, iron, aluminum and chromium was considered. The results were negative in each instance.

Hence, it may be concluded from what has preceded that nickel free from phosphorus can be completely precipitated from a solution of nickel sulphate containing disodium hydrogen phosphate and free phosphoric acid, a mineral acid; that the deposit is equal to that precipitated by any other known method; that the time factor can be reduced by raising the temperature and that nickel can not be separated from manganese, iron, aluminum or chromium in phosphoric acid solutions. It was found that chromium salts in the presence of disodium hydrogen phosphate and free phosphoric acid are oxidized to chromates by the electric current.