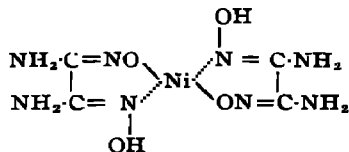




The constitutional formula of the nickel complex is tentatively given as



in analogy with the dimethylglyoxime complex.

#### EXPERIMENTAL.

*Preparation of the Reagent.*—Fischer's method of preparation was followed (*Ber.*, 1889, 22, 3930). A rapid stream of cyanogen gas was passed into a concentrated, ice-cold, aqueous solution of hydroxylamine hydrochloride (1 mol.) and caustic potash (1 mol.). Oxalenediamidoxime separated in colourless crystals, and was purified by recrystallisation from hot water (m.p. lit. 200° with decomp., found, 199° with decomposition). The reagent is readily soluble in hot water; a dilute solution of the reagent was used in the estimations.

*Nickel Oxalenediamidoxime.*—The light orange, crystalline precipitate of the nickel complex, obtained by precipitating with a dilute solution of the reagent from faintly ammoniacal solution of nickel chloride containing ammonium chloride, was washed with cold water, and readily obtained anhydrous by drying at 110–120° for half an hour. [Found: Ni. (by ignition to oxide), 20.02. Calc. for  $\text{Ni}(\text{C}_2\text{H}_3\text{N}_4\text{O}_2)_2$ : Ni, 20.04 per cent].

*Estimation of Nickel.*—A solution of chemically pure nickel chloride containing about 0.2% nickel was prepared and standardised by the dimethylglyoxime method. Certified standard pipettes were used for measuring out known volumes of nickel solution. To the nickel solution at room temperature was added a cold dilute solution of the reagent in slight excess, and then ammonium hydroxide drop by drop with stirring, till the solution smells of ammonia. The light orange precipitate of the nickel complex was allowed to settle, filtered through asbestos filter, washed with cold water, dried at 110–120°, and weighed. Table I shows the results.

TABLE I.

Wt. of Ni oxime ppt.	Weight of nickel found.		Wt. of Ni oxime ppt.	Weight of nickel found.	
	found.	taken.		found.	taken.
0.0297 g.	0.00595 g.	0.00598 g.	0.1116 g.	0.0224 g.	0.0224 g.
0.0596	0.01194	0.01195	0.1120	0.0224	0.0224
0.1193	0.0239	0.0239	0.1111	0.0223	0.0224

*Separation of Nickel from Zinc and Manganese.*—A known volume of the standard nickel solution was mixed respectively with known volumes of zinc and manganous chloride solutions, containing approximately known amounts of Zn and Mn. The mixture was in each case treated with a sufficient quantity of ammonium chloride to prevent precipitation of the hydroxides in ammoniacal medium. Nickel was then precipitated and estimated as described above. The results are shown in Table II.

TABLE II.

Composition of soln.	Wt. of ppt.	Wt. of Ni found.	Error.
0·0224 g. Ni + 0·025 g. Zn	0·1112 g.	0·0223 g.	-0·0001
0·0239      0·025	0·1190	0·0238	-0·0001
0·0230      0·05	0·1193	0·0239	Nil
0·0239      0·125	0·1196	0·0240	+0·0001
0·0239      0·25	0·1192	0·0239	Nil
0·0239      0·375	0·1191	0·0239	Nil
0·0224      0·014 Mn	0·1117	0·0224	Nil
0·0239      0·024	0·1194	0·0239	Nil
0·0239      0·048	0·1196	0·0240	+0·0001
0·0239      0·24	0·1196	0·0240	+0·0001
0·0239      0·36	0·1193	0·0239	Nil

*Separation of Nickel from Cobalt.*—A dark brown, insoluble powder slowly separated from ammoniacal solutions containing cobalt on the addition of the reagent. The precipitation was found to be accelerated by passing a current of air, and temporarily prevented by the addition of hydroxylamine hydrochloride. The estimation of nickel in presence of cobalt was carried out in exactly the same manner as in other cases, with the only difference that nearly 1 g. of hydroxylamine hydrochloride was added for every 100 c.c. of the solution, before rendering the solution ammoniacal. Some of the results are shown in the following table. Larger amounts of cobalt than those shown here led to high results for nickel.

TABLE III.

Composition of soln.	Wt. of ppt.	Wt. of Ni found.	Error.
0·0239 g. Ni + 0·0125 g. Co	0·1199 g.	0·0240 g.	+0·0001
0·0239      0·025	0·1190	0·0238	-0·0001
0·0239      0·05	0·1193	0·0239	Nil

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