

AN ABSTRACT OF THE THESIS OF

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Title The Preparation and Heats of Formation of Some
Nickel and Cobalt Complex Salts with certain Organic Bases

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The preparation of the amine complex salts of nickel oxalate, cobalt oxalate, nickel phthalate and cobalt phthalate with pyridine, β -picoline and γ -picoline has been accomplished. The nickel phthalate and cobalt phthalate complex salts of β -picoline and γ -picoline have been prepared by means of chloroform extraction. The chloroform was added to a water solution containing the complex. The complex was extracted with the chloroform and the solvent evaporated. The other complex salts were made by adding excess amine to the anhydrous simple salt. The compounds were analyzed for per cent metal content. The metal oxalate complex salts were shown to be di-amine compounds. The metal phthalate complex compounds were tetra-amine compounds.

The heats of solution in 2N HCl at 25°C of Nickel oxalate, cobalt oxalate, nickel phthalate, cobalt phthalate, pyridine, β -picoline, γ -picoline and their respective complex salts have been determined. The Calorimeter used was identical in design to that employed by previous workers. From the heat of solution values the heats of formation of the twelve complex salts have been calculated. The heats of formation of the six metal oxalate-amine complex salts lie in the range of -18,800 calories per mole to -20,800 calories per mole. The heats of formation of the metal phthalate-amine complex salts lie in the range of -28,800 calories per mole to -35,300 calories per mole.

THE PREPARATION AND HEATS OF FORMATION OF
SOME NICKEL AND COBALT COMPLEX SALTS
WITH CERTAIN ORGANIC BASES

by

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THE PREPARATION AND HEATS OF FORMATION OF
SOME NICKEL AND COBALT COMPLEX SALTS
WITH CERTAIN ORGANIC BASES

INTRODUCTION

The relative strengths of the coordinate linkage between metals and nitrogen in amine complex salts have been studied by numerous investigators. Most of these studies have been conducted using either a series of metals with the same negative radical and the same amine or by varying the negative radical with the metal and the amine kept constant. This investigation deals with four salts: nickel oxalate, cobalt oxalate, nickel phthalate and cobalt phthalate. Pyridine and the picolines are the amines used. This study should then aid in determining the effect of varying two closely related metals, two dibasic acid radicals and three similar amines on the relative bond strength of the metal-nitrogen coordinate linkage.

Three methods have been employed in investigating the effect of different atoms and groups on the coordinate bond. These methods are the determination of the dissociation pressures of the complex salts, the determination of volume changes during the formation of the complex and the heat of formation of the complex. The method of heats of formation has been applied only to a few compounds. Very little of this work has offered any means of comparing the

effects of varying either the metal, the amine or the negative radical.

This investigation deals with the preparation of the complex salts of the aforementioned compounds and the determination of the heat of formation of the metal-nitrogen coordinate linkage by an indirect means. A stronger coordinate bond should have a higher heat of formation if all other factors are constant.

To determine the heats of formation, the heat of solution of the simple nickel or cobalt salt in 2N hydrochloric acid is measured. The heat of solution of the amine and the heat of solution of the complex salt are also determined in 2N HCl. The complex compound is decomposed in the acid to the amine hydrochloride and the simple salt; thus the heats of solution of the amine and the simple salt will differ from the heat of solution of the complex by an amount equal to the heat of formation of the complex. The heat of formation, ΔH_f , is given by the relation:

$$\Delta H_f = \Delta H_s + \Delta H_A - \Delta H_c$$

ΔH_s = heat of solution of the simple salt

ΔH_A = heat of solution of the amine

ΔH_c = heat of solution of the complex salt

PREPARATION OF THE COMPOUNDS

Preparation of Nickel Oxalate and Cobalt Oxalate

The oxalates of nickel and cobalt were made by adding in equivalent amounts solutions of oxalic acid to a solution of the nitrate salts of these metals. The pink cobalt oxalate and the light green nickel oxalate immediately precipitated and were filtered, washed thoroughly and dried in an oven.

Preparation of Nickel Phthalate and Cobalt Phthalate

The phthalate salts of nickel and cobalt were prepared by refluxing for six hours a mixture of nickel or cobalt carbonate with a solution containing an equivalent amount of phthalic acid. At the end of approximately six hours any remaining residue was removed and the solution from the reaction mixture evaporated to dryness. Nickel phthalate was a yellow-green color while cobalt phthalate was light purple.

Preparation of the Complex Salts of Nickel Oxalate and Cobalt Oxalate with Pyridine, ϵ -Picoline and γ -Picoline

Excess pyridine was added to the oxalate salts. The light green nickel oxalate became a deep blue in color which upon evaporation yielded light blue crystals. Using the same procedure with cobalt oxalate rose-colored crystals

were obtained. The complex salts were immediately stored in a dessicator containing a beaker of pyridine.

With the picolines, crystals of similar color were obtained with both nickel oxalate and cobalt oxalate.

Preparation of the Complex Salts of Nickel Phthalate and Cobalt Phthalate with Pyridine, ϵ -Picoline and γ -Picoline

Attempts were made to isolate the complex salts of the phthalates and pyridine by means of chloroform extraction from a water solution. However, this method was unsuccessful. The same method used in preparing the oxalate complex salts was then employed. Bright blue crystals of the nickel phthalate-pyridine complex were obtained. The crystals of the cobalt phthalate-pyridine complex were rose-colored. These complex salts were soluble in chloroform. Upon evaporating the chloroform solutions the resulting crystals were assayed for nickel or cobalt content and proved to be of the same constitution as the complex salts obtained by simply adding pyridine to the salt.

A chloroform extraction procedure was employed in preparing the phthalate-picoline complex salts. A water solution of nickel phthalate was made to which γ -picoline was added. A blue color was imparted to the previously green solution. Chloroform was then added and the complex salt extracted with the chloroform and evaporated. Deep blue-green crystals were obtained. Chloroform extraction of the nickel phthalate ϵ -picoline complex yielded light blue

crystals. The cobalt phthalate ϵ -picoline complex and the cobalt phthalate γ -picoline complex both yielded deep red crystals by chloroform extraction and evaporation of the solvent.

ANALYSIS OF THE COMPOUNDS

The anhydrous salts and the complex salts were analyzed for per cent metal content in the compound. Nickel was determined by the potassium cyanide-silver nitrate volumetric method using potassium iodide as indicator (10, pp. 660-663). Cobalt was determined gravimetrically as the sulfate (10, p.188). The analytical results may be found in Table I.

The metal analyses revealed that the nickel and cobalt phthalate complex salts with the three amines were tetra-amine complex compounds. The oxalate-amine compounds were di-amine complex salts.

TABLE I
ANALYSIS OF THE COMPOUNDS

Compound	Per cent metal		
	calculated	found	
Nickel oxalate	40.04	39.85	39.82
Nickel oxalate·2 pyridine	19.25	19.50	19.23
Nickel oxalate·2 ϵ -picoline	17.63	17.64	17.76
Nickel oxalate·2 γ -picoline	17.63	17.60	17.40
Cobalt oxalate	40.10	40.38	40.32
Cobalt oxalate·2 pyridine	19.31	19.16	19.27
Cobalt oxalate·2 ϵ -picoline	17.69	17.83	17.76
Cobalt oxalate·2 γ -picoline	17.69	17.54	17.51
Nickel phthalate	26.34	26.25	26.24
Nickel phthalate·4 pyridine	10.88	10.61	10.66
Nickel phthalate·4 ϵ -picoline	9.86	9.63	9.67
Nickel phthalate·4 γ -picoline	9.86	9.80	9.80
Cobalt phthalate	26.42	26.71	26.68
Cobalt phthalate·4 pyridine	10.92	10.88	10.86
Cobalt phthalate·4 ϵ -picoline	9.90	9.98	10.01
Cobalt phthalate·4 γ -picoline	9.90	10.13	10.07

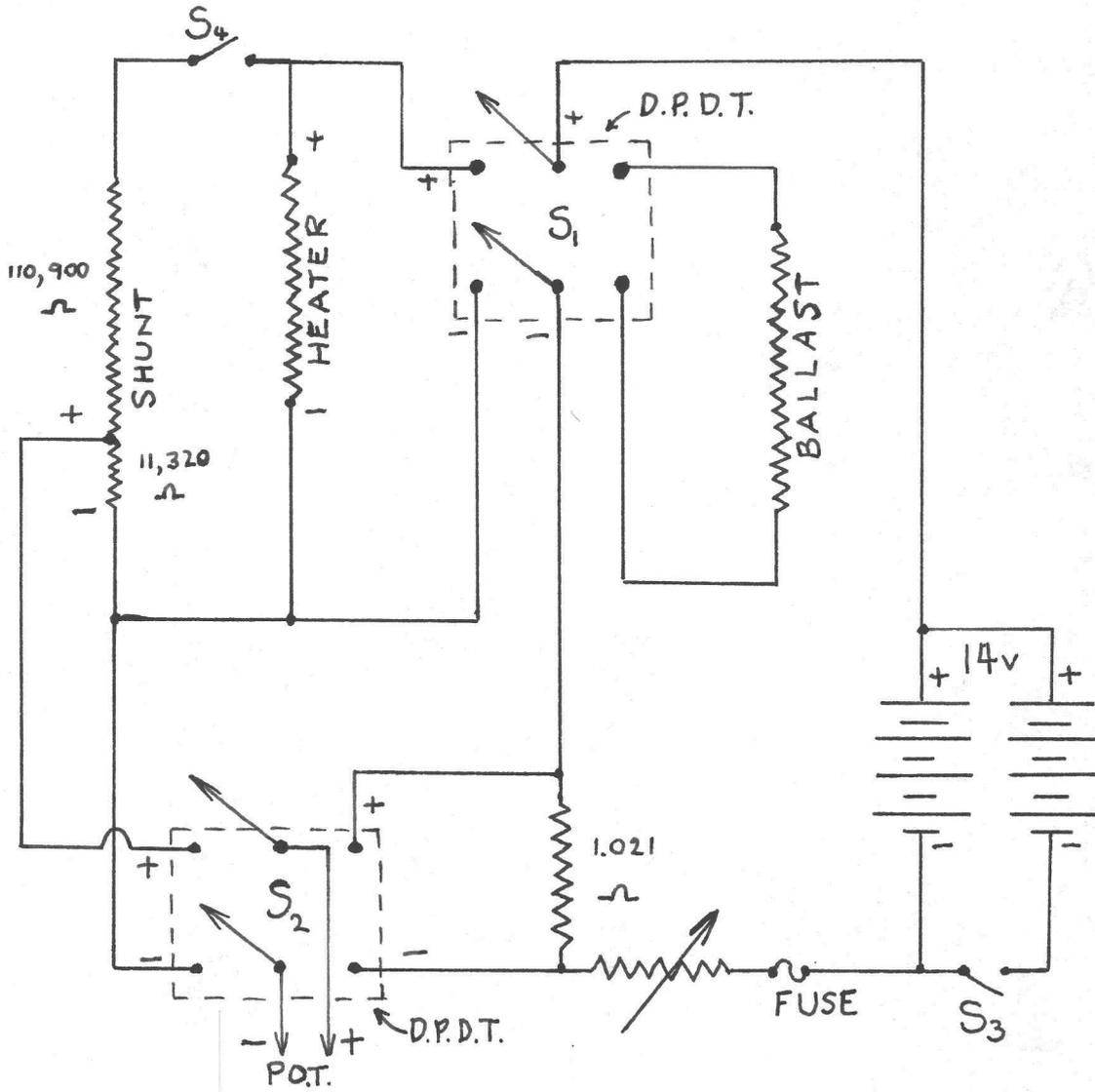
THE CALORIMETER

The calorimeter was the same one used by Bush (1, p.10), Rogers (9, p.11) and Carle (2, pp.20-21). A detailed description of the design and construction of the calorimeter may be obtained by referring to reference (1, pp.9-19) or (9, pp.6-10).

The calorimeter consisted of a two-liter Dewar flask placed in a constant temperature bath which was equipped with an efficient stirring device. The bath was kept at 25° C during all determinations made with the calorimeter. The Dewar flask contained a wooden lid with four openings through which a stirrer, a Beckmann thermometer, a heater and a sample ejector could be introduced into the flask.

The heating circuit was identical in design to that used by Bush and Rogers. The shunt resistance was needed since the potentiometer could not measure voltages greater than 1.6 volts. The electrical source of energy was provided by four Edison batteries which consisted of five cells to a battery. Two batteries were connected in series while two of these sets were connected in parallel to give added stability to the voltage on the power source. The heater circuit wiring diagram is shown in Figure 1.

FIGURE 1
HEATING CIRCUIT WIRING DIAGRAM



DETERMINATION OF THE HEAT CAPACITY

The heat capacity of the calorimeter was calculated by passing into the calorimeter a known quantity of electrical energy. The current is passed through a resistance wire changing electrical energy into heat energy which is then measured by the temperature rise on a Beckmann thermometer.

The electrical energy is measured by determining the voltage and current flowing in a definite time interval and converting these values to heat units by means of the following equation:

$$H = \frac{E I t}{4.1833}$$

H = energy in calories

E = voltage across heater

I = current passing through heater

t = time of heating in seconds

The Dewar flask was filled with 1500 ml. of 2N HCl, the lid fastened into place and the flask put into the constant temperature bath. Stirring was started and current passed through the heater until the contents of the flask were heated to the bath temperature. After allowing the system to come to equilibrium and observing whether there were any heating or cooling effects due to heat of stirring or evaporation, the heating was begun again. Temperature readings on the Beckmann thermometer were then taken every five minutes for a period of twenty minutes. The heater was

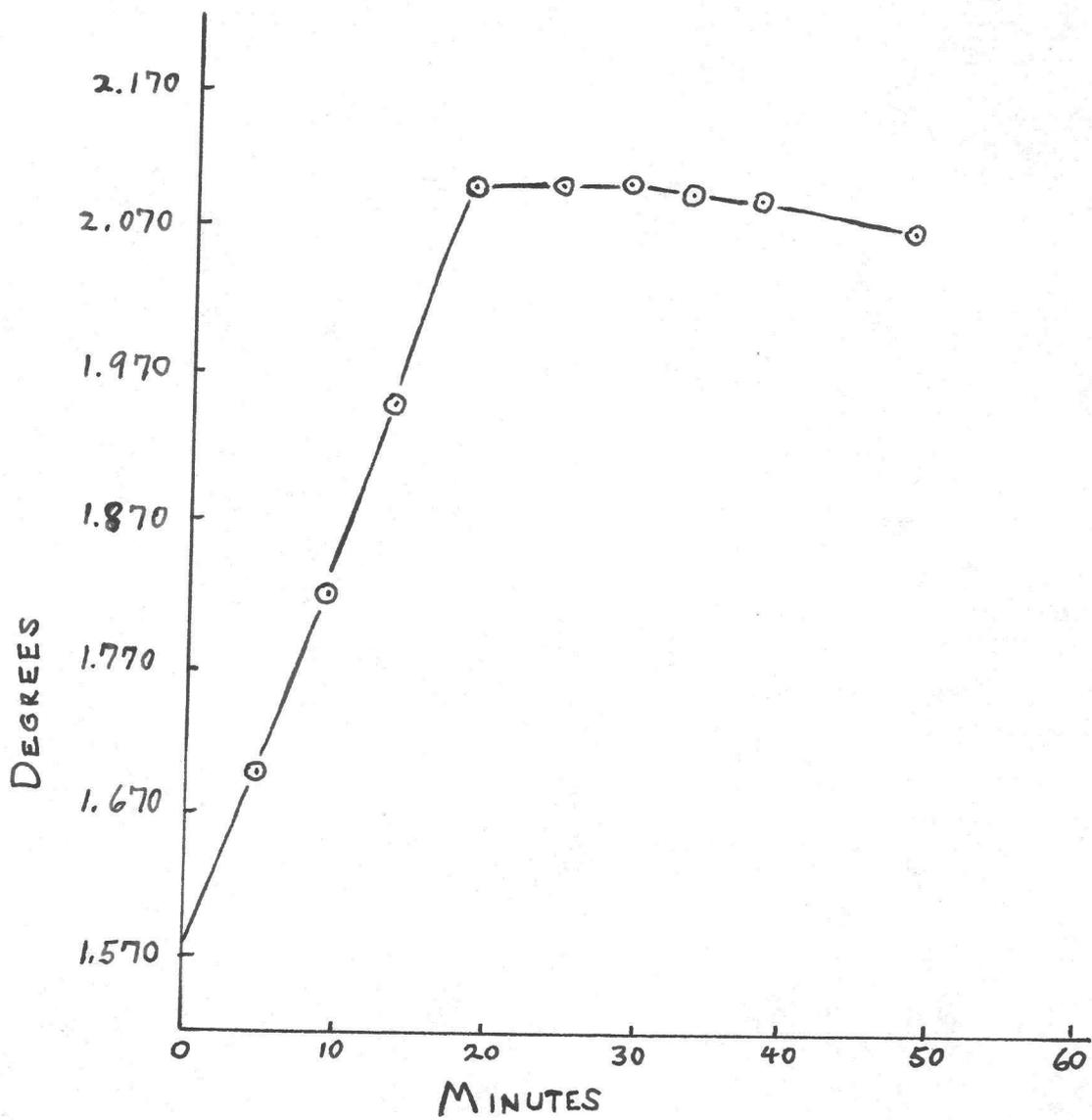
then turned off and temperature readings taken at intervals of one minute for five minutes and then every five minutes for 25 additional minutes. While the heater was on, measurements on the potentiometer were made in order to determine the voltage and current being used. Once the voltage, current, time of heating and temperature rise are known the heat capacity in calories per degree can be calculated. A corrected temperature rise is obtained by plotting a time vs. temperature curve (see Figure 2). The heat capacity data is shown in Table II.

An error has been found in the heat capacity determination of the calorimeter in reference (1, p.19) and (9, p.9). These workers calculated the fraction of the power through the potentiometer by taking the simple ratio of the two resistances used in the shunt instead of taking the ratio of the 10,000 Ohm resistance to the sum of the two resistances. This correction would give a value of 1538 calories for the heat capacity instead of 1403 calories. This value of the heat capacity will make a considerable difference in the heat of solution values previously obtained.

TABLE II
HEAT CAPACITY OF THE CALORIMETER
AT 25° C

Time (seconds)	Volts shunt	Volts 1 OHM	Temperature rise	Heat capacity calories per degree
1200	.8100	.3460	.558	1523
1200	.7047	.3150	.440	1528
1200	.8016	.3438	.547	1526
1200	.7847	.3302	.517	1525
			average	1525

FIGURE 2
HEAT CAPACITY OF THE
CALORIMETER CONTAINING
2N HCl AT 25° C



HEATS OF SOLUTION

The heats of solution were determined using sample ejectors identical in design to those used by Bush (1, pp. 14-15) and Rogers (9, p.12). The calorimeter was filled with 1500 ml. of 2N HCl and the lid fastened into place. The sample ejector, thermometer, heater and stirrer were fitted into the lid and the flask placed in the constant temperature bath. The contents of the flask were heated to 25° C and then allowed to reach equilibrium taking the usual precautions regarding heat of stirring and cooling effects. The solid or liquid sample was then injected into the hydrochloric acid and time-temperature readings taken every 30 seconds until the temperature remains constant. Additional readings were taken every five minutes to obtain a cooling curve. Time-temperature graphs were plotted for each compound. A typical curve for some of the compounds may be found in Figures 3 through 7. The heats of solution of the compounds are shown in Tables III through XXI.

HEATS OF FORMATION

The heats of formation of the various compounds calculated from the heats of solution data are shown in Table XXII.

TABLE III
HEAT OF SOLUTION OF PYRIDINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
3.4588	.245	-8530
3.3142	.235	-8540
3.7696	.268	-8530
	average	-8530

TABLE IV
HEAT OF SOLUTION OF β -PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
3.3666	.219	-9230
3.4693	.225	-9210
3.2958	.213	-9180
	average	-9210

TABLE V
HEAT OF SOLUTION OF γ -PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
3.4695	.230	-9440
3.2975	.218	-9370
3.2937	.218	-9390
	average	-9400

TABLE VI
HEAT OF SOLUTION OF NICKEL OXALATE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.3470	.067	-3450
4.9014	.076	-3480
4.3738	.068	-3460
	average	-3460

TABLE VII

HEAT OF SOLUTION OF NICKEL OXALATE·2 PYRIDINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.5105	.013	-1340
4.8649	.014	-1350
	average	-1340

TABLE VIII

HEAT OF SOLUTION OF NICKEL OXALATE·2 *e*-PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.3915	.018	-2040
4.3250	.017	-2000
	average	-2020

TABLE IX

HEAT OF SOLUTION OF NICKEL OXALATE·2 π -PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.3730	.019	-2210
4.6257	.023	-2230
4.4836	.019	-2190
		average -2210

TABLE X

HEAT OF SOLUTION OF COBALT OXALATE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
3.8760	.087	-5040
3.0582	.069	-5060
3.5920	.082	-5110
		average -5070

TABLE XI

HEAT OF SOLUTION OF COBALT OXALATE·2 PYRIDINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.9014	.013	-1280
4.8633	.013	-1240
4.9013	.014	-1320
	average	-1280

TABLE XII

HEAT OF SOLUTION OF COBALT OXALATE·2 β -PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.8560	.044	-4600
4.8428	.042	-4410
4.7483	.042	-4490
	average	-4500

TABLE XIII

HEAT OF SOLUTION OF COBALT OXALATE·2 γ -PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.8357	.053	-5070
4.8474	.049	-5090
	average	-5080

TABLE XIV

HEAT OF SOLUTION OF NICKEL PHTHALATE
IN 2N NCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.3062	.139	-10,930
4.3426	.139	-10,910
4.5477	.147	-10,970
	average	-10,940

TABLE XV

HEAT OF SOLUTION OF NICKEL PHTHALATE·4 PYRIDINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.5697	.064	-10,290
4.5212	.063	-10,220
	average	-10,260

TABLE XVI

HEAT OF SOLUTION OF NICKEL PHTHALATE·4 β -PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.4652	.061	-12,400
4.5315	.063	-12,610
4.5014	.062	-12,500
	average	-12,500

TABLE XVII

HEAT OF SOLUTION OF NICKEL PHTHALATE·4 γ -PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.6120	.099	-19,420
4.5254	.097	-19,450
	average	-19,440

TABLE XVIII

HEAT OF SOLUTION OF COBALT PHTHALATE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.8640	.100	-6980
4.9402	.102	-7030
4.8018	.099	-7020
	average	-7010

TABLE XIX

HEAT OF SOLUTION OF COBALT PHTHALATE·4 PYRIDINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
4.8240	.081	-12,330
4.8245	.081	-12,330
4.6836	.072	-12,370
	average	-12,340

TABLE XX

HEAT OF SOLUTION OF COBALT PHTHALATE·4 β -PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per mole
3.6274	.051	-12,740
3.7981	.053	-12,680
	average	-12,710

TABLE XXI

HEAT OF SOLUTION OF COBALT PHTHALATE·4 γ -PICOLINE
IN 2N HCl AT 25° C

Weight sample (grams)	Temperature change	Heat of solution calories per gram
4.8852	.071	-13,190
4.8112	.069	-13,010
4.7366	.068	-13,050
	average	-13,080

TABLE XXII

HEATS OF FORMATION AT 25° C

Compound	Heat of formation calories per mole
Nickel oxalate·2 pyridine	-19,200
Nickel oxalate·2 β -picoline	-19,900
Nickel oxalate·2 γ -picoline	-20,000
Cobalt oxalate·2 pyridine	-20,800
Cobalt oxalate·2 β -picoline	-19,000
Cobalt oxalate·2 γ -picoline	-18,800
Nickel phthalate·4 pyridine	-34,800
Nickel phthalate·4 β -picoline	-35,300
Nickel phthalate·4 γ -picoline	-29,100
Cobalt phthalate·4 pyridine	-28,800
Cobalt phthalate·4 β -picoline	-31,100
Cobalt phthalate·4 γ -picoline	-31,500

FIGURE 3
HEAT OF SOLUTION OF γ -PICOLINE
IN 2N HCl AT 25° C

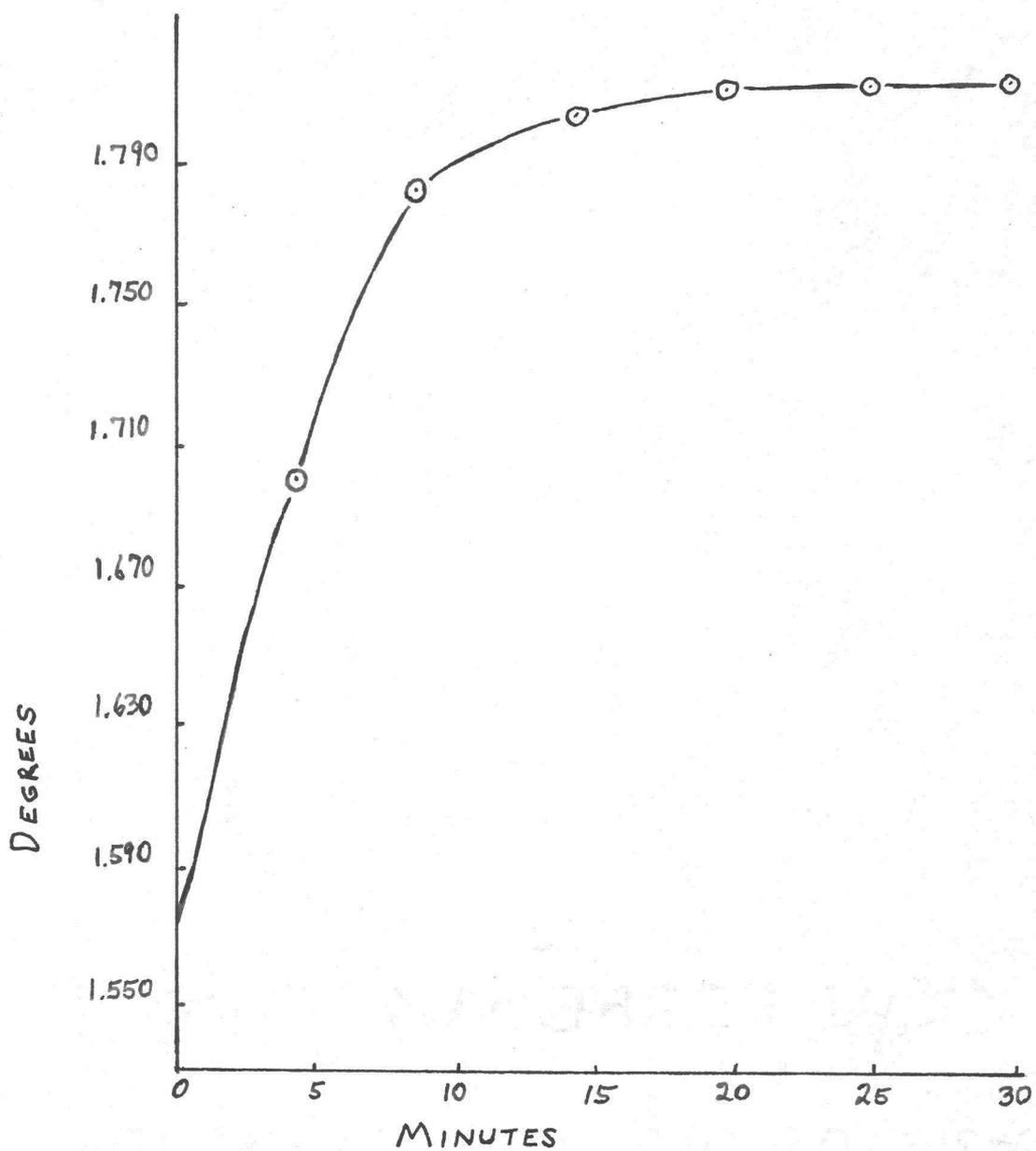


FIGURE 4
HEAT OF SOLUTION OF COBALT OXALATE
IN 2N HCl AT 25° C

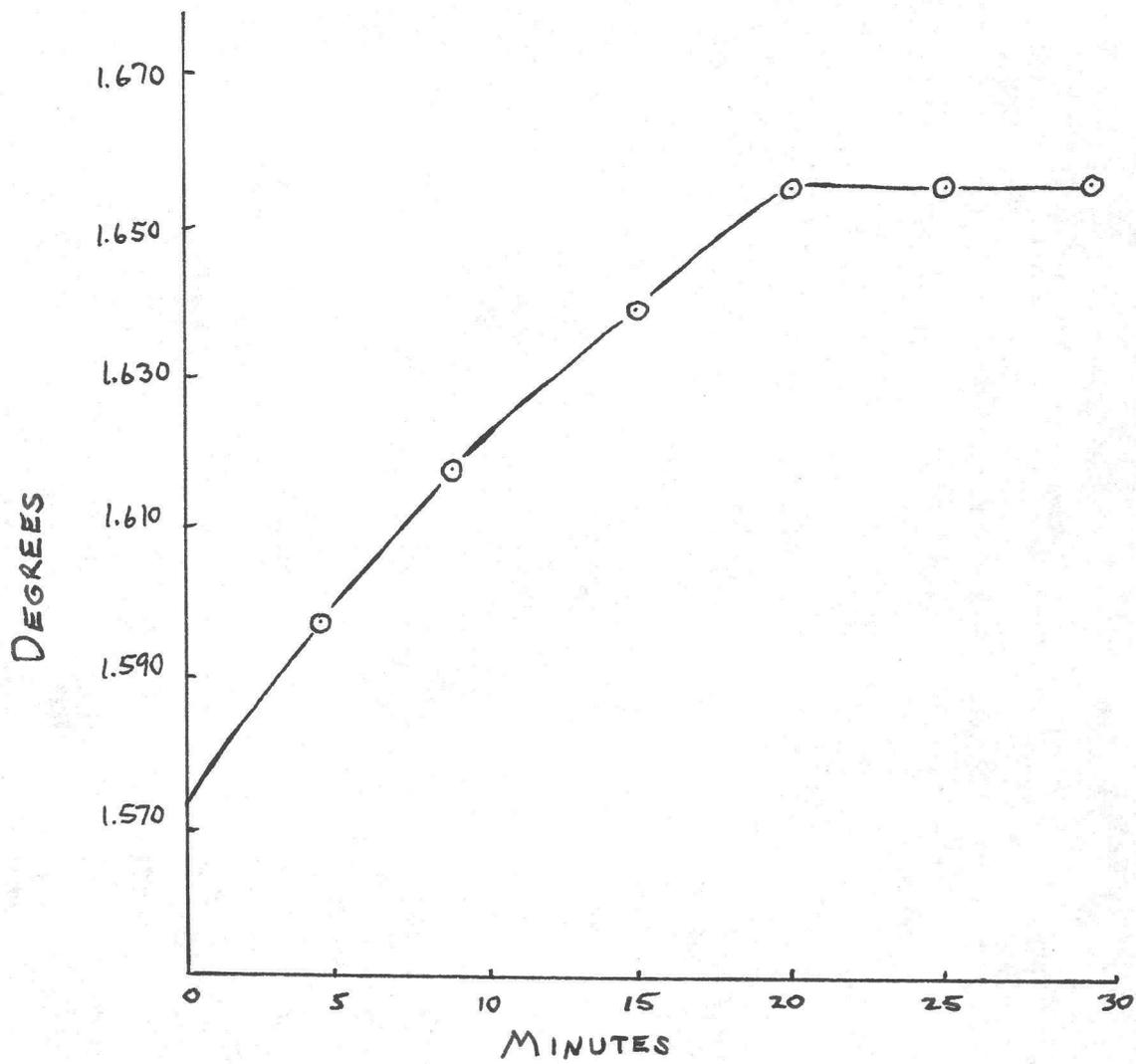


FIGURE 5

HEAT OF SOLUTION OF NICKEL PHTHALATE
IN 2N HCl AT 25°C

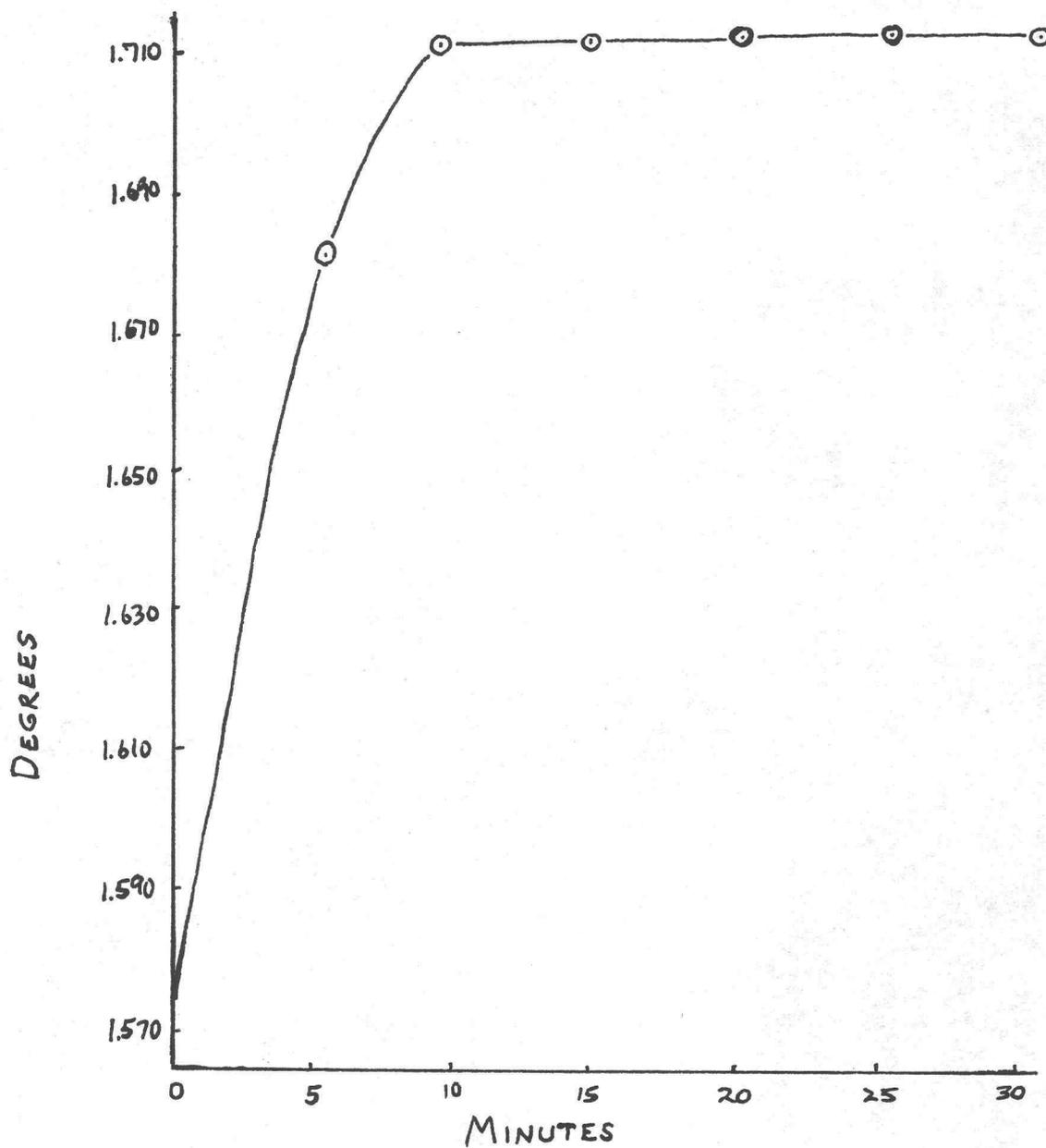


FIGURE 6

HEAT OF SOLUTION OF $\text{NiC}_2\text{O}_4 \cdot 2\text{PICOLINE}$
IN 2N HCl AT 25°C

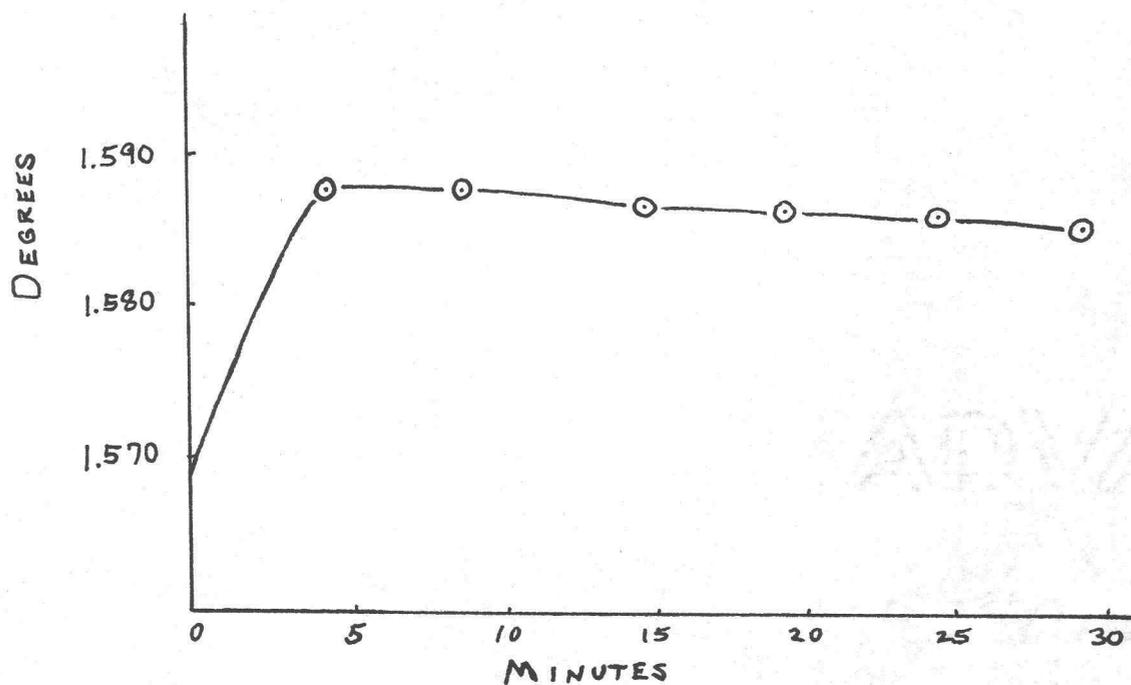
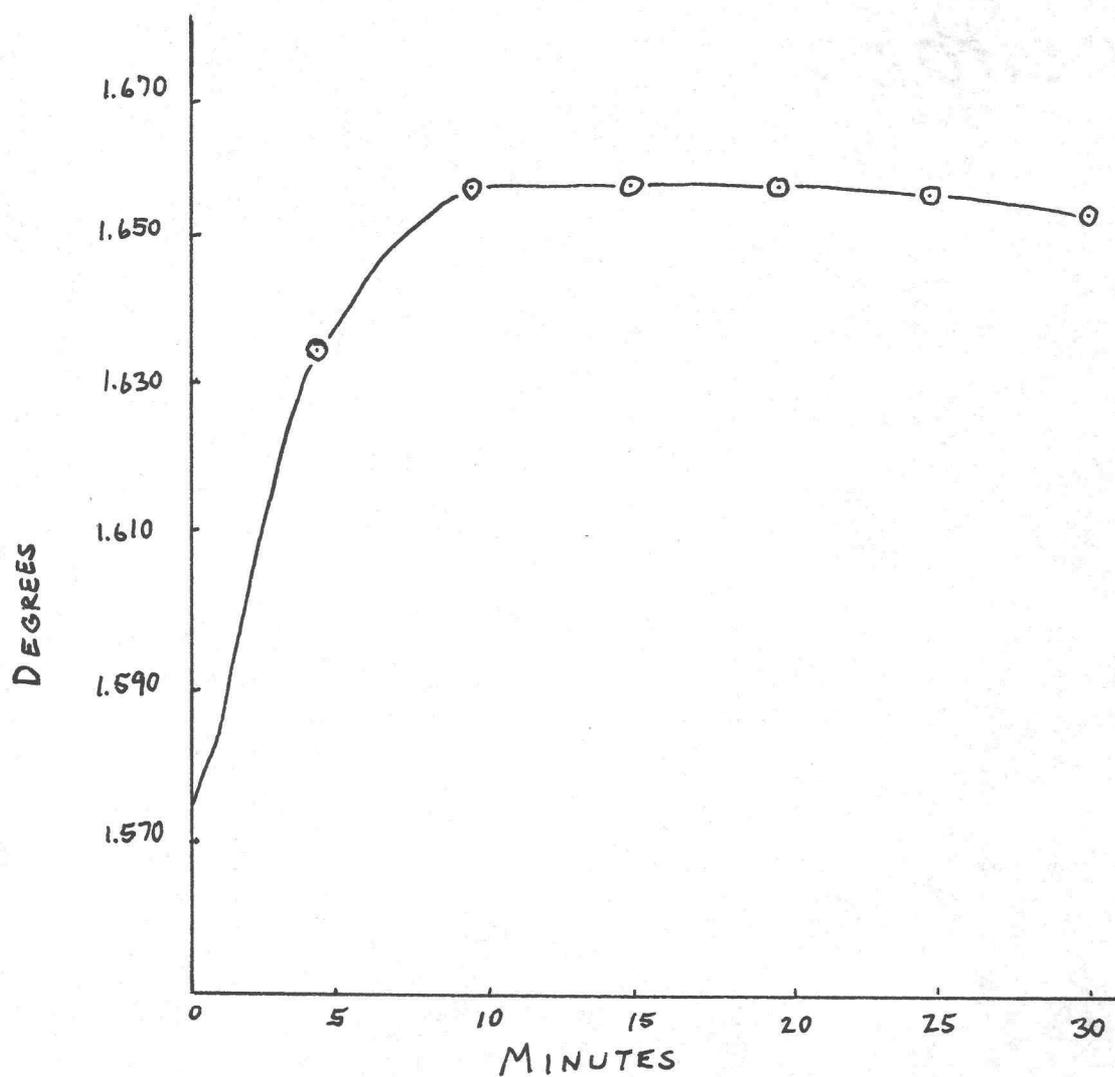


FIGURE 7

HEAT OF SOLUTION OF COBALT PHTHALATE·4 PYRIDINE
IN 2N HCl AT 25°C



DISCUSSION

The values of the heats of formation shown in Table XXII allow some good comparisons to be made. A comparison of the effect of the negative radicals on the metal-nitrogen linkage is limited because of the different number of amine molecules coordinated in forming the complex. However, it would seem that the negative radical exerts a greater effect than the metal or the amine in the formation of the complex compounds since a different number of amine molecules coordinated in forming the complex compounds of the metal oxalate and metal phthalate salts.

A comparison can be made of the effect of the amine. The values of the heat of formation of any one salt with the three amines reveal little variance. The effect of the greater heat of solution of one amine with respect to the others is largely canceled by the heats of solution of the corresponding complex salts. One can conclude that the heat of formation of the complex salts is practically independent of the amine used in its formation.

The effect of the metal seems to be of lesser importance. This observation is in agreement with previous studies on the effect of nickel and cobalt on the metal-nitrogen coordinate linkage.

The Beckmann thermometer was definitely a limiting factor in this work. An error of one-thousandth of a degree would correspond in some cases to a difference of 200 calories in the heats of solution. For this reason the heat of formation values were rounded off to the nearest 100 calories.

This investigation would indicate that more study is needed on the effect of the negative radical as this seems to be the most important factor involved. Work is currently in progress in this laboratory on nickel and cobalt amine complex salts using mono-carboxylic acid radicals as the negative radical.

SUMMARY

The compounds nickel oxalate, cobalt oxalate, nickel phthalate, cobalt phthalate and the complex compounds of these salts with pyridine, ϕ -picoline and γ -picoline have been prepared and analyzed. The heats of solution of these compounds were determined in 2N HCl. From the values of the heats of solution the heats of formation of twelve complex compounds were calculated.

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