

0.0684 gave 0.0152 CuO; Cu, 17.70.

0.1280 gave 0.1357 AgBr; Br, 45.11.

Calc. for  $C_9H_7NHBr.CuBr$ : Cu, 17.86; Br, 45.32.

**Isoquinolinium Cuprous Bromide.**—At first, a yellow, viscous oil separated which, on shaking, soon became crystalline. This was washed and dried as usual.

0.1138 gave 0.0254 CuO; Cu, 17.79.

0.1502 gave 0.1585 AgBr; Br, 44.9.

Calc. for  $C_9H_7NHBr$ : Cu, 17.86; Br, 45.32.

**$\alpha$ -Picolinium Cuprous Bromide.**—This was obtained as glistening brown crystals.

0.1002 gave 0.0246 CuO; Cu, 19.58.

0.1128 gave 0.1330 AgBr; Br, 50.19.

Calc. for  $C_8H_4NCH_3HBr$ : Cu, 19.87; Br, 50.47.

### The Double Mercurous Iodides.

The double mercurous iodides have been prepared by adding a solution of mercurous nitrate to the substituted ammonium iodides when the double salts were obtained as precipitates. The reaction was successful with the heavily substituted ammonium bases.

**Trimethyl-*p*-tolylammonium Iodide.**—To a strong solution of trimethyl-*p*-tolylammonium iodide, a solution of mercurous nitrate was added when a white emulsion was produced which on shaking deposited a gray precipitate. This was washed and dried.

0.4122 gave 0.1059 HgS; Hg, 22.13.

0.1410 gave 0.1124 AgI; I, 43.07.

Calc. for  $2N(CH_3)_3C_7H_7I.HgI$ : Hg, 22.70; I, 43.24.

**Tetrapropylammonium Mercurous Iodide.**—This was obtained as a grayish white precipitate. The salt on drying appeared white with a very faint yellow tinge.

0.3560 gave 0.0865 HgS; Hg, 20.93.

0.1246 gave 0.0918 AgI; I, 39.81.

Calc. for  $2N(C_3H_7)_4I.HgI$ : Hg, 21.03; I, 39.97.

CALCUTTA.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORY, UNIVERSITY OF COLORADO.]

## ON THE SALTS OF PHTHALIC ACID.

BY JOHN B. ECKLEY AND CLIFFORD BANTA.

Received February 12, 1917.

### Introduction.

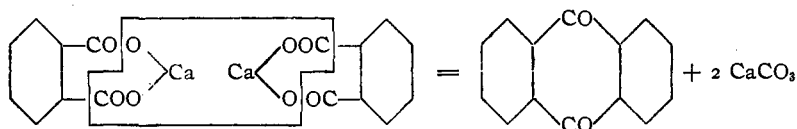
The decomposition of calcium phthalate under dry distillation has been studied by Miller,<sup>1</sup> and by Panaotovits.<sup>2</sup> The former states that the main products obtained are benzophenone and benzol, together with small

<sup>1</sup> *J. Russ. Chem. Soc.*, 11, 256 (1879).

<sup>2</sup> *Ber.*, 17, 312 (1884).

quantities of anthraquinone and certain hydrocarbons. Panaotovits claims to have obtained considerable quantities of anthraquinone, but does not give the details concerning his method of distillation. He promises another paper on the subject, but, as far as we know, has not yet been heard from. In Beilstein's Handbuch<sup>1</sup> the statement is made, without giving the authority, that acid calcium phthalate decomposes at 140°, yielding phthalic anhydride. In the light of these conflicting statements, it seemed desirable to study the effect of heat upon calcium phthalate under varying conditions of temperature and pressure. This has been done with results as indicated under the experimental part of this paper. During the course of the investigation, the desirability of a verification of the composition of certain phthalic acid salts and the preparation of new ones became evident.

From the behavior of calcium salts of saturated aliphatic acids on dry distillation, it would seem that calcium phthalate would follow a similar decomposition according to the equation



with the formation of anthraquinone.

That small quantities of anthraquinone are formed seems to be the case, but we were unable, by changing the conditions of the experiment, to obtain any appreciable yield of the substance. The optimism of Panaotovits seems to have been unwarranted, as is perhaps shown also by his failure to give any further information on the subject.

In studying this question, we found that no systematic study of the metal salts of phthalic acid had been made. We therefore verified the most important preparations of others, and prepared a number of new salts not described in the literature.

Up to the present, the following salts<sup>2</sup> have been prepared:

$\text{NH}_4\text{H}\bar{\text{A}}^3$ , Laurent, Marignac;  $\text{NaH}\bar{\text{A}} \cdot 2\text{H}_2\text{O}$ , Wislicenus;  $\text{Na}_2\bar{\text{A}}$ , Wislicenus;  $\text{KH}\bar{\text{A}}$ , Volhard;  $\text{K}_2\bar{\text{A}}$ , Wislicenus;  $\text{Ca}\bar{\text{A}} \cdot \text{H}_2\text{O}$ , Hermann, Weith;  $\text{BaH}_2\bar{\text{A}}_2$ , Hermann;  $\text{Ba}_3\text{H}_2\bar{\text{A}}_6$ , Carius, Hermann, Weith;  $3\text{Ba}\bar{\text{A}} \cdot \text{BaO}$ , Carius;  $\text{Pb}\bar{\text{A}}$ , Schunck, Carius;  $\text{Cu}\bar{\text{A}} \cdot \text{H}_2\text{O}$ , Hermann;  $\text{Ag}_2\bar{\text{A}}$ , Marignac;  $\text{CaH}_2\bar{\text{A}}_2$ .

### Experimental.

(a) Calcium phthalate was heated in a distilling flask under a pressure of 9 mm., the temperature being increased gradually. At about 45° a lemon yellow colored substance sublimed on to the sides of the flask and a gas

<sup>1</sup> "Ergänzungsband," Vol. II, 1047 (1902).

<sup>2</sup> Beilstein, "Ergänzungsband," Ed. 3, Vol. II, 1047, 1793; Ed. 4, Vol. III, 1047.

<sup>3</sup> The symbol  $\bar{\text{A}}$  is used for the phthalic acid radical  $\text{C}_6\text{H}_4\text{O}_4$ .

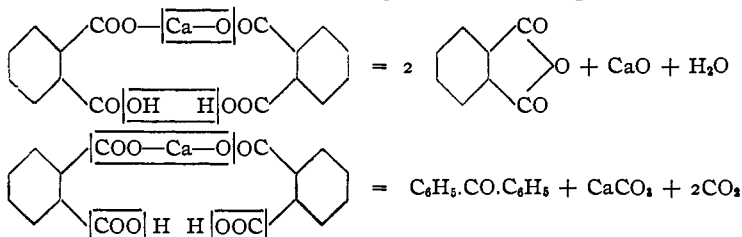
was evolved. The temperature of about  $45^{\circ}$  was maintained for ten minutes without any appreciable increase in the amount of the yellow substance. As the temperature was gradually increased, the color of the sublimate became gradually darker and at  $77^{\circ}$  a reddish brown resinous substance distilled over.

(b) A small amount of the calcium phthalate was placed in a Jena glass distilling flask. A rubber stopper with entrance tube connected to a carbon dioxide generator was wired on to the flask. The exit tube of the flask was connected to a second distilling flask and wired fast. The exit tube of the second distilling flask was connected with a mercury U tube capable of measuring pressure somewhat over 1000 mm. Dry carbon dioxide was run into the apparatus until the pressure was 1000 mm. The flask was then gradually heated. At  $55^{\circ}$  small quantities of a yellow substance appeared in the salt. No sublimate formed. No further change appeared until the temperature reached  $95^{\circ}$ . The pressure then began to increase rapidly and after an increase of about 50 mm. the flask exploded.

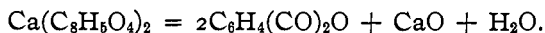
(c) A small amount of calcium phthalate was sealed in a bomb tube and heated in the furnace for 3 hours at  $200^{\circ}$  and at  $300^{\circ}$  without visible change. The tube burst on heating again and the contents were found to be unchanged.

(d) Five g. of acid calcium phthalate were heated in a distillation flask at 22 mm. pressure. At about  $80^{\circ}$ , the evolution of gas began and a very small amount of phthalic anhydride needles and moisture formed in the neck of the flask. On increasing the temperature more gas was given off and a liquid condensed in the neck of the flask and ran down into the salt. At  $170^{\circ}$  this liquid distilled over. Meantime a vigorous evolution of gas was observed, the particles of salt being kept in lively agitation by the escaping gas. The distillate proved to be a mixture of phthalic anhydride and benzophenone.

It seems probable that the following reactions take place:



(e) Molecular proportions of acid calcium phthalate and anhydrous calcium chloride were heated in a distilling flask under a pressure of 20 mm. There was not any reaction until a temperature of  $80^{\circ}$  was reached when phthalic anhydride distilled over completely. The decomposition evidently is



(f) Molecular proportions of acid calcium phthalate and dry calcium carbonate were heated in a distilling flask at 20 mm. pressure. At 110° a resinous substance distilled over. No definite compound could be isolated from the distillate.

(g) A mixture of molecular proportions of acid calcium phthalate, anhydrous calcium chloride and dry calcium carbonate was sealed in a bomb tube and heated in the furnace for 4 hours to 150°, 175°, 200° and 225° each, without visible change. There was no visible change on heating for 3 hours to 250°, 275°, 300°, 335° and above 360°. The tube exploded after heating 1 hour at 450° (estimated), giving strong odor of benzophenone.

(h) A small amount of phthalic anhydride was sealed in a bomb tube and heated in a furnace for 3 hours to 200° and 300° when, in both cases, it melted and resolidified on cooling. On heating 3 hours at about 400°, it charred slightly. The tube was broken and the contents were found to be entirely soluble in water, crystallizing out as phthalic acid.

(i) Sodium phthalate was distilled under a pressure of 13 mm. at 80°. Benzophenone distilled over.

(j) Lead phthalate was heated in a distilling flask under 7 mm. pressure. At 35° water and carbon dioxide were given off and this continued until all of the lead was reduced to finely divided metal.

(k) Lead phthalate was exposed in a small quartz crucible to the action of light from a quartz mercury vapor lamp. The outside layer of the salt decomposed rapidly with the formation of a yellowish brown substance of unknown composition, probably containing lead oxide.

(l) A similar experiment performed with mercuric phthalate showed the salt to be unaffected by light from a quartz mercury vapor lamp.

#### The Salts of the Metals and Phthalic Acid.

(a) **Verification of Results Obtained by Other Investigators. Sodium Phthalate,  $\text{Na}_2\text{C}_8\text{H}_4\text{O}_4$ .**—Phthalic acid solution was neutralized with sodium hydroxide, using phenolphthalein as indicator. The solution was allowed to evaporate to dryness and white crystals formed. The salt is exceedingly soluble in water. It was dried in an air oven for four hours at 130° and but slight loss in weight occurred.

Sodium was estimated by igniting the salt and decomposing the carbonate thus formed by adding hydrochloric acid. This solution was evaporated to dryness. The sodium was weighed as the chloride and the percentage converted to percentage of oxide.

Calc. for  $\text{Na}_2\text{C}_8\text{H}_4\text{O}_4$ :  $\text{Na}_2\text{O}$ , 29.52%. Found:  $\text{Na}_2\text{O}$ , 29.68%.

**Potassium Phthalate,  $\text{K}_2\text{C}_8\text{H}_4\text{O}_4$ .**—Phthalic acid solution was neutralized with potassium hydroxide using phenolphthalein as indicator. The

solution was evaporated to dryness and the salt crystallized in long, silky, white needles. This salt is exceedingly soluble in water. It was dried in an air oven for three hours at  $130^{\circ}$  and but slight loss in weight occurred. Potassium was estimated by igniting the salt, decomposing the carbonate thus formed by adding hydrochloric acid and evaporating the solution to dryness. The potassium was weighed as chloride and the percentage converted to percentage of oxide.

Calc. for  $K_2C_8H_4O_4$ :  $K_2O$ , 38.89%. Found:  $K_2O$ , 38.61%.

**Acid Calcium Phthalate**,  $Ca(C_8H_5O_4)_2 \cdot H_2O$ .—Calcium oxide was boiled with phthalic acid solution for an hour. The solution was filtered and the salt, which was only slightly soluble in water, was allowed to crystallize. It was dried in an air oven at  $120^{\circ}$  for three hours and the loss in weight, due to driving off the water of crystallization, was determined. The anhydrous salt was then ignited in platinum and the calcium weighed as calcium oxide.

Calc. for  $Ca(C_8H_5O_4)_2 \cdot H_2O$ :  $CaO$ , 14.43%;  $H_2O$ , 4.64%.

Found:  $CaO$ , 14.40%;  $H_2O$ , 4.49%.

Normal calcium phthalate can not be prepared by boiling an excess of milk of lime with phthalic acid. Weith<sup>1</sup> claims that, when finely powdered calcite is boiled for an hour with phthalic acid solution, normal calcium phthalate,  $CaC_8H_4O_4$ , is formed. He gives in the analysis of the salt he thus prepared

Calc.:  $Ca$ , 16.6%. Found:  $Ca$ , 16.39%.

As a matter of fact the calculated amount of calcium in calcium phthalate is not 16.6% but 19.23%, and the salt obtained by this method is not normal calcium phthalate but acid calcium phthalate. The normal calcium salt is not produced from calcite, or calcium carbonate, or milk of lime, with phthalic acid, for, when phthalic acid solution is added to calcium carbonate in molecular proportions, a solution of the *acid* salt is formed and half the calcium carbonate remains unchanged. This is true in both hot and cold solutions.

No precipitate occurred when alcoholic solutions of phthalic acid and calcium nitrate were mixed in molecular proportions.

**Normal Calcium Phthalate**,  $CaC_8H_4O_4$ .—Phthalic acid solution was neutralized with *filtered lime water* according to the method of Hermann.<sup>2</sup> The salt was allowed to crystallize and was found to be sparingly soluble in water.

The salt was dried to constant weight in an air oven at  $120^{\circ}$  and the loss in weight represented less than one-fifth of a molecule of water. On ignition the salt decrepitated violently, showing that this was probably enclosed water. It would therefore appear that the salt does not have a

<sup>1</sup> *Ber.*, 7, 1059 (1874).

<sup>2</sup> *Ann.*, 151, 77 (1869).

molecule of water of crystallization, as stated by Hermann. Calcium was estimated by drying the salt to constant weight and then igniting in platinum to calcium oxide.

Calc. for  $\text{CaC}_8\text{H}_4\text{O}_4$ :  $\text{CaO}$ , 27.45%. Found:  $\text{CaO}$ , 27.49%.

**Barium Phthalate**,  $\text{BaC}_8\text{H}_4\text{O}_4$ .—Sodium phthalate solution was added to barium nitrate solution and a white crystalline precipitate was obtained on boiling. The salt was dried in an air oven at  $120^\circ$  for three hours and but slight loss in weight occurred. Barium was estimated as barium carbonate by ignition of the salt in platinum and the percentage found was converted to percentage of barium oxide.

Calc. for  $\text{BaC}_8\text{H}_4\text{O}_4$ :  $\text{BaO}$ , 50.89%. Found:  $\text{BaO}$ , 51.43%.

**Lead Phthalate**,  $\text{PbC}_8\text{H}_4\text{O}_4$ .—A white precipitate was formed on the addition of sodium phthalate solution to lead nitrate solution. The salt was dried in an air oven for four hours at  $120^\circ$  and but slight loss in weight occurred. Lead was estimated as lead sulfate by digesting the salt with concentrated sulfuric acid, diluting the solution with 50% alcohol and filtering through a weighed Gooch crucible. The percentage of  $\text{PbSO}_4$  found was converted to percent age of  $\text{PbO}$ .

Calc. for  $\text{PbC}_8\text{H}_4\text{O}_4$ :  $\text{PbO}$ , 60.12%. Found:  $\text{PbO}$ , 60.36%.

**Copper Phthalate**,  $\text{CuC}_8\text{H}_4\text{O}_4 \cdot \text{H}_2\text{O}$ .—Phthalic acid solution was added to basic copper carbonate with the carbonate in excess. The solution was filtered and evaporated until blue crystals appeared. The salt is sparingly soluble in water.

The salt was dried at  $120^\circ$  for three hours and the loss in weight, due to driving off the water of crystallization, was determined. Copper was estimated by igniting the salt to  $\text{CuO}$ .

Calc. for  $\text{CuC}_8\text{H}_4\text{O}_4 \cdot \text{H}_2\text{O}$ :  $\text{H}_2\text{O}$ , 7.33%;  $\text{CuO}$ , 32.40%.

Found:  $\text{H}_2\text{O}$ , 7.19%;  $\text{CuO}$ , 32.29%.

**Silver Phthalate**,  $\text{Ag}_2\text{C}_8\text{H}_4\text{O}_4$ .—Silver carbonate was decomposed by adding phthalic acid solution. The silver salt is sparingly soluble in water, its solubility being practically the same as that of phthalic acid. The first crystals were a mixture of phthalic acid and silver phthalate. The phthalic acid was removed by washing with alcohol. The pure salt, after being air dried, was then dried in an air oven at  $110^\circ$  for three hours with only slight loss in weight. Silver was estimated by igniting the salt to metallic silver.

Calc. for  $\text{Ag}_2\text{C}_8\text{H}_4\text{O}_4$ :  $\text{Ag}$ , 56.82%. Found:  $\text{Ag}$ , 56.47%.

#### (b) New Salts of Phthalic Acid.

**Lithium Phthalate**,  $\text{Li}_2\text{C}_8\text{H}_4\text{O}_4$ .—Lithium carbonate was decomposed by adding phthalic acid solution and the resulting solution was allowed to crystallize. The salt was found to be only moderately soluble in water. It was dried in an air oven at  $120^\circ$  for three hours with only a slight loss in

weight. Lithium was estimated by igniting the salt to lithium carbonate and converting the percentage found to percentage of  $\text{Li}_2\text{O}$ .

Calc. for  $\text{Li}_2\text{C}_8\text{H}_4\text{O}_4$ :  $\text{Li}_2\text{O}$ , 16.80%. Found:  $\text{Li}_2\text{O}$ , 16.65%.

**Beryllium Phthalate**,  $\text{BeC}_8\text{H}_4\text{O}_4 \cdot \text{H}_2\text{O}$ .—Phthalic acid solution was added to an excess of beryllium carbonate. The solution was filtered and evaporated to dryness. A glassy gum was formed which finally crystallized. The salt was dried in an air oven at  $120^\circ$  for three hours and the loss in weight, due to driving off the water of crystallization, was determined. Beryllium was estimated by igniting the salt to  $\text{BeO}$ .

Calc. for  $\text{BeC}_8\text{H}_4\text{O}_4 \cdot \text{H}_2\text{O}$ :  $\text{H}_2\text{O}$ , 9.42%;  $\text{BeO}$ , 13.14%.  
Found:  $\text{H}_2\text{O}$ , 9.23%;  $\text{BeO}$ , 13.91%.

**Manganese Phthalate**,  $\text{MnC}_8\text{H}_4\text{O}_4$ .—Sodium phthalate solution was added to manganese nitrate solution. A flesh-colored precipitate was obtained on boiling the solution. This was almost white when dry. The salt was dried in an air oven at  $120^\circ$  for three hours with only a slight loss in weight. Manganese was estimated by igniting the salt to  $\text{Mn}_3\text{O}_4$  and converting the percentage found to percentage of  $\text{MnO}$ .

Calc. for  $\text{MnC}_8\text{H}_4\text{O}_4$ :  $\text{MnO}$ , 32.39%. Found:  $\text{MnO}$ , 32.79%.

**Basic Ferric Phthalate**,  $\text{Fe}(\text{C}_8\text{H}_4\text{O}_4)(\text{OH}) \cdot 2\text{H}_2\text{O}$ .—Sodium phthalate solution was added to ferric nitrate solution. A brown precipitate was formed which was very difficult to filter. The salt was dried in an air oven at  $130^\circ$  for three hours and the loss in weight, due to driving off the water, was determined. Iron was estimated by igniting the salt to  $\text{Fe}_2\text{O}_3$ .

Calc. for  $\text{Fe}(\text{C}_8\text{H}_4\text{O}_4)(\text{OH}) \cdot 2\text{H}_2\text{O}$ :  $\text{H}_2\text{O}$ , 13.19%;  $\text{Fe}_2\text{O}_3$ , 29.28%.  
Found:  $\text{H}_2\text{O}$ , 12.78%;  $\text{Fe}_2\text{O}_3$ , 29.17%.

**Cobalt Phthalate**,  $\text{CoC}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ .—Phthalic acid solution was added to an excess of cobalt carbonate. The solution was filtered and evaporated until the salt crystallized. The salt is sparingly soluble in water and is lilac colored when first crystallized but rapidly loses two molecules of water to exposure to air and changes to a beautiful light purple. On account of this fact it is a difficult matter to determine the water of crystallization at the proper time. The determination will show that the salt is not thoroughly air dried or that water of crystallization has been lost. The salt was dried in an air oven at  $105^\circ$  for two hours. Cobalt was estimated by igniting the salt to  $\text{CoO}$ .

Calc. for  $\text{CoC}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ :  $\text{CoO}$ , 28.96%;  $\text{H}_2\text{O}$ , 13.90%.  
Found:  $\text{CoO}$ , 28.99%;  $\text{H}_2\text{O}$ , 14.36%, 12.96%.

**Nickel Phthalate**,  $\text{NiC}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ .—Phthalic acid solution was added to an excess of nickel carbonate. The solution was filtered and evaporated to dryness on a water bath. A green jelly was formed which slowly crystallized. The salt was dried in an air oven at  $125^\circ$  for three hours and the loss in weight, due to driving off the water of crystallization, was determined. Nickel was estimated by igniting the salt to  $\text{NiO}$ .

Calc. for  $\text{NiC}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ :  $\text{H}_2\text{O}$ , 13.92%;  $\text{NiO}$ , 28.87%.  
 Found:  $\text{H}_2\text{O}$ , 13.35%;  $\text{NiO}$ , 28.32%.

**Acid Cerium Phthalate**,  $\text{Ce}(\text{C}_8\text{H}_5\text{O}_4)_3$ .—Phthalic acid solution was added to cerium nitrate solution and the resulting solution was allowed to crystallize. The salt was found to be moderately soluble in water. Only a slight loss in weight occurred when the salt was dried in an air oven at  $120^\circ$  for three hours. Cerium was estimated by igniting the salt to  $\text{CeO}_2$ .

Calc. for  $\text{Ce}(\text{C}_8\text{H}_5\text{O}_4)_3$ :  $\text{CeO}_2$ , 27.12%. Found:  $\text{CeO}_2$ , 26.43%.

**Mercuric Phthalate**,  $\text{HgC}_8\text{H}_4\text{O}_4 \cdot \text{H}_2\text{O}$ .—A white, crystalline precipitate was formed when sodium phthalate solution was added to mercuric nitrate solution. The precipitate was filtered off and air dried. It was then heated in an air oven at  $120^\circ$  for three hours and the loss in weight, due to driving off the water of crystallization, was determined. Mercury was estimated by the method given in Schimpf's Volumetric Analysis, fifth edition, page 415.

Calc. for  $\text{HgC}_8\text{H}_4\text{O}_4 \cdot \text{H}_2\text{O}$ :  $\text{H}_2\text{O}$ , 4.70%;  $\text{Hg}$ , 52.43%.  
 Found:  $\text{H}_2\text{O}$ , 5.03%;  $\text{Hg}$ , 51.97%.

**Mercurous Phthalate**,  $\text{Hg}_2\text{C}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ .—A white, crystalline precipitate was formed when sodium phthalate solution was added to mercurous nitrate solution. The precipitate was filtered off and air dried. It was then dried in an air oven at  $120^\circ$  for three hours and the loss in weight due to driving off the water of crystallization, was determined. Mercury was estimated as in the mercuric salt.

Calc. for  $\text{Hg}_2\text{C}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ :  $\text{H}_2\text{O}$ , 5.99%;  $\text{Hg}$ , 66.73%.  
 Found:  $\text{H}_2\text{O}$ , 5.69%;  $\text{Hg}$ , 66.99%.

**Thorium Phthalate**,  $\text{Th}(\text{C}_8\text{H}_4\text{O}_2)_2 \cdot 5\text{H}_2\text{O}$ .—Thorium carbonate was prepared by adding potassium carbonate solution to thorium nitrate solution. The carbonate was decomposed by adding phthalic acid solution and the resulting solution was allowed to crystallize. The salt was found to be moderately soluble in water. It was dried in an air oven at  $140^\circ$  for three hours and the loss in weight, due to driving off the water of crystallization, was determined. Thorium was estimated by igniting the salt to  $\text{ThO}_2$ .

Calc. for  $\text{Th}(\text{C}_8\text{H}_4\text{O}_2)_2 \cdot 5\text{H}_2\text{O}$ :  $\text{H}_2\text{O}$ , 13.84%;  $\text{ThO}_2$ , 40.65%.  
 Found:  $\text{H}_2\text{O}$ , 14.38%;  $\text{ThO}_2$ , 41.34%.

**Uranyl Phthalate**,  $\text{UO}_2\text{C}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ .—Phthalic acid solution was added to uranyl chloride solution and the resulting solution was allowed to evaporate until yellow crystals were formed. The salt was found to be moderately soluble in water. It was dried in an air oven at  $110^\circ$  for three hours and the loss in weight was determined. Uranium was estimated by igniting the salt to  $\text{U}_3\text{O}_8$ .

Calc. for  $\text{UO}_2\text{C}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ :  $\text{H}_2\text{O}$ , 7.65%;  $\text{U}_3\text{O}_8$ , 59.76%.  
 Found:  $\text{H}_2\text{O}$ , 7.49%;  $\text{U}_3\text{O}_8$ , 59.47%.



**Zinc Salt.**—Sodium phthalate solution was added to zinc nitrate solution and a white precipitate was formed on boiling. The analysis follows:

$\text{H}_2\text{O}$ , 3.72%;  $\text{ZnO}$ , 40.91%.

This does not check for any definite salt. It is probably a basic salt, the composition of which would vary with varying conditions.

**Cadmium Salt.**—Sodium phthalate solution was added to cadmium nitrate solution and a white, crystalline precipitate was formed which gave the following results on analysis:

$\text{H}_2\text{O}$ , 6.62%;  $\text{Cd}$ , 34.43%.

This analysis does not correspond to any definite salt.

**Thallium.**—Neither phthalic acid or sodium phthalate precipitate thallium from solution. Phthalic acid solution was added to thallium sulfate solution and the resulting solution was allowed to evaporate until crystals formed. These crystals did not contain thallium but were phthalic acid.

**Chromium.**—Chromium hydroxide does not dissolve in phthalic acid solution.

**Bismuth.**—No reaction occurred when bismuth nitrate, which had been dissolved in just enough dilute nitric acid to keep it in solution, was added to phthalic acid solution. On evaporation, the phthalic acid crystallized out separately.

**Aluminium.**—Aluminium hydroxide is precipitated from aluminium nitrate solution by the addition of sodium phthalate solution.

### Summary.

The results of this investigation may be briefly summarized as follows:

1. A number of salts of phthalic acid were decomposed under varying conditions of temperature and pressure. None of these yielded more than a trace of anthraquinone. The products obtained were resinous matter, phthalic anhydride and benzophenone, the latter often occurring in such large amounts, that the dry distillation of sodium or copper phthalate is suggested as a method for the preparation of this compound. The direct preparation of anthraquinone from phthalic acid or some of its derivatives still appears theoretically feasible, but the problem remains unsolved.

2. The preparation and composition of a number of phthalates, already in the literature, were verified or corrected.

3. The following salts, concerning which nothing could be found in the literature, were prepared and analyzed:

Normal lithium phthalate, $\text{Li}_2\text{C}_8\text{H}_4\text{O}_4$ .	Acid cerium phthalate, $\text{Ce}(\text{C}_8\text{H}_5\text{O}_4)_3$ .
Normal beryllium phthalate, $\text{BeC}_8\text{H}_4\text{O}_4 \cdot \text{H}_2\text{O}$ .	Normal mercuric phthalate, $\text{HgC}_8\text{H}_4\text{O}_4 \cdot \text{H}_2\text{O}$ .
Normal manganese phthalate, $\text{MnC}_8\text{H}_4\text{O}_4$ .	Normal mercurous phthalate, $\text{Hg}_2\text{C}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ .
Basic ferric phthalate, $\text{Fe}(\text{C}_8\text{H}_4\text{O}_4)(\text{OH}) \cdot 2\text{H}_2\text{O}$ .	Normal thorium phthalate, $\text{Th}(\text{C}_8\text{H}_4\text{O}_4)_2 \cdot 5\text{H}_2\text{O}$ .
Normal cobalt phthalate, $\text{CoC}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ .	Normal uranyl phthalate, $\text{UO}_2\text{C}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ .
Normal nickel phthalate, $\text{NiC}_8\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$ .	

From these results, it may be concluded that phthalic acid forms salts readily with the more basic metals. The solubility of these salts in water varies through a wide range and it apparently bears no relation to the atomic weight of the metallic element present. This is shown by the following examples:

The lithium salt is only moderately soluble in water while the beryllium salt is exceedingly soluble.

The cobalt salt is only moderately soluble while the nickel salt is exceedingly soluble.

The lead salt is insoluble while the thorium salt is moderately soluble.

Probably the best method for preparing these salts is the decomposition of the carbonate of the metal by phthalic acid solution. This method does not introduce any foreign acid radicals into the solution and removes the possibility of a mixture of crystals being formed.

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[CONTRIBUTION FROM THE BIOCHEMICAL LABORATORY OF THE UNIVERSITY OF ILLINOIS.]

## THE NITROGENOUS HYDROLYSIS PRODUCTS OF HEART LECITHIN.

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It has been known for some time<sup>1</sup> that egg lecithin yields on hydrolysis both choline and amino ethyl alcohol. Recently a paper appeared<sup>2</sup> showing the presence of both of these compounds, as well as an unhydrolyzable form of nitrogen in the fatty acid residue, in brain lecithin. The object of the investigation recorded here was to study the hydrolysis products of heart lecithin, to show the presence or absence of each of the above mentioned nitrogenous compounds and to obtain quantitative data on their distribution.

### Preparation and Purification.

Beef hearts, each weighing 1800 to 2500 g. in the fresh state, were used as a source of the material for this experiment. After removing the adherent fat, the valves and the connective tissue as completely as possible, the muscle was minced by passage through a grinder. The ground meat was pressed to remove the blood, weighed approximately and dried. Drying was accomplished by one of three methods: (1) dehydration with alcohol, (2) dehydration with acetone, (3) removal of water by current of air in a drier at a temperature of 35-40° C.

**Dehydration.**—(1) In the dehydration by alcohol the ground material was placed in large bottles and treated with twice its volume of 95%

<sup>1</sup> G. Trier, *Z. physiol. Chem.*, **76**, 496 (1911-12).

<sup>2</sup> J. E. Darrah and C. G. MacArthur, *THIS JOURNAL*, **38**, 922 (1916).