

at room temperature for one hour, in order that the hydrogen chloride should not be given off so rapidly as to prevent complete absorption in the Liebig bulbs, and then the flask was heated on the water bath and kept boiling for two hours. 100 cc. of dilute hydrochloric acid (20 cc. conc. hydrochloric acid to 80 cc. water) were added to the cooled contents of the flask, the benzene distilled off with steam, the residue cooled to 0°, and the orthobenzoyl-benzoic acid filtered off; this was washed with cold water, redissolved in sodium carbonate and precipitated with hydrochloric acid. In this way we obtained a 96-97% yield with no phthalic acid and never more than a trace of diphenylphthalide.

When less aluminium chloride was used both phthalic acid and diphenylphthalide were obtained. The diphenylphthalide was separated by its insolubility in sodium carbonate solution and the remaining acids were separated by extracting the benzoyl-benzoic acid from the phthalic acid by chloroform. Small quantities of the two acids were also recovered from the first filtrate containing the aluminium chloride.

In the following table the phthalic acid recovered has been omitted and the hydrogen chloride has been expressed in formula weights per formula weight of aluminium chloride used ( $Al_2Cl_6$ ):

No.	Benzene. Cc.	Ph. anhy. Grams.	Al. chl. Grams.	Time boiled. Hours.	Benzoyl- benz. ac. Grams.	Diphen- phth. Grams.	Hydr. chl.
1.....	18	5	9	2	7.36	None	1.17
2.....	18	5	9	2	7.29	None	1.18
3.....	18	5	9	2	7.39	None	1.17
4.....	18	5	4.5	2	2.03	0.45	1.02
5.....	18	5	4.5	2	2.01	0.48	0.91
6.....	18	5	4.5	2	2.47	0.86	1.09
7.....	18	5	4.5	0.5	2.87	0.17	0.90
8.....	20	5	4.5	3.5	..	1.17	1.27
9.....	20	5	4.5	8.5	..	1.36	1.25
10.....	36	5 + 5	9	2 + 2	5.14	0.88	1.20
11.....	40	5 + 5	9	2 + 2	6.72	6.96	1.02
12.....	40	5 + 2	9	2 + 2	7.51	0.30	1.24
13.....	30	5	18	2	7.23	None	..
14.....	20	5	4.5 + 4.5	2 + 2	6.50	0.65	1.31
15.....	20	5	2.3	2	0.78	0.6	1.00

From Nos. 1-3 it will be seen that, using the proportions of the reagents indicated, the yield of orthobenzoyl-benzoic acid is practically constant and the hydrogen chloride given off is nearly 1.2 formula weights per formula weight of aluminium chloride used. There is no diphenylphthalide formed. In Nos. 4-6 using half the amount of aluminium chloride, the yield of orthobenzoyl-benzoic acid is less than one-third and not so constant and diphenylphthalide is found. Nos. 7-9 show that longer boiling materially increases the yield of diphenylphthalide. In Nos. 10-12 the usual proportions of the reagents were used and after