

### A THIRD METHYL ESTER OF PHTHALIC ACID.

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These experiments were undertaken under the direction of Professor Allan with the object of finding out whether it was possible to prepare an ester of phthalic acid corresponding to the barium salt  $(C_6H_4C_2O_4Ba)_2C_6H_4C_2O_3$ . This barium salt was obtained by heating the acid salt  $(C_6H_4C_2O_4)_2H_2BaH_2O$  at  $120-140^\circ$  until constant weight was obtained.

*Barium Salt and Ethyl Iodide.*—(a) Five g. of the barium salt and 5 cc. ethyl iodide were shaken for several hours at room temperature.

(b) Five g. of the barium salt and 5 cc. ethyl iodide with 3 cc. ethyl alcohol were shaken for several hours at room temperature.

(c) Five g. of the barium salt and 5 cc. ethyl iodide were heated at  $100^\circ$  for two hours.

The contents of each of these tubes were then treated with water and tested for barium iodide with negative results in each case. This indicated that no ester had been formed.

*Barium Salt and Methyl Sulphate.*—(d) 0.5 g. of the barium salt and 5 cc. methyl sulphate were shaken for four hours at room temperature. When the liquid was removed, the solid part was found to contain barium sulphate, which showed that a reaction had taken place.

(e) Barium salt and methyl sulphate were heated in a sealed tube at  $100^\circ$  for four hours and then filtered. Part of the filtrate was shaken with water to decompose the methyl sulphate but all the oil disappeared, the ester being saponified by the sulphuric acid from the methyl sulphate. Another part of the filtrate, on standing, separated into two layers, one of which was found to contain much more sulphate than the other. The part containing least sulphate was washed with water, dried over sulphuric acid and saponified with potassium hydroxide solution.

0.1500 gram of the oil required, 12.17 cc. potassium hydroxide sol.

Calculated for dimethyl phthalate, 16.13 cc.

Calculated for monomethyl phthalate, 18.59 cc.

This showed that this oil was, at any rate, not the pure ester corresponding to the barium salt, as the potassium hydroxide solution required to saponify it would be intermediate between that required for the monomethyl ester and that necessary for the dimethyl ester. Other experiments showed that when the neutral barium salt or the acid salt was