Also sodium phosphate (5 g.) in 10 per cent. solution and 2 g. ammonium chloride were substituted for the ammonium phosphate in the first procedure above.

Grams zinc taken: 0.1841. Found: 0.1844, 0.1840, 0.1851. Grams zinc taken: 0.2240. Found: 0.2242, 0.2250, 0.2242.

The precipitate in this case did not settle as readily as where ammonium phosphate was used. The large excess of sodium phosphate is necessary, as two grams gave very low results.

It appears that the results are not improved by washing with 1 per cent. ammonium phosphate and alcohol, and that ammonium phosphate is more satisfactory than sodium phosphate. The presence of an excess of ammonium salts does not interfere.

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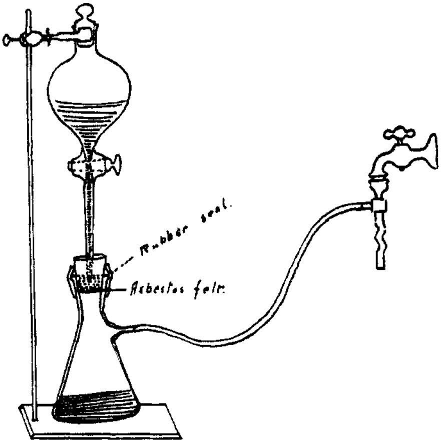
Constant-level Reservoir.—The arrangement shown in the figure has been used by us for some time for purposes similar to the one described by Mr. Fitzgerald.[1] We have found it especially useful during the filtration of solutions of bitumen in carbon tetrachloride containing large amounts of carbenes, when it is essential that the filter be not allowed to become dry.

As seen in the sketch, the Gooch filter is connected with the Erlenmeyer filtering flask in the usual manner, and a globe-shaped, stoppered separatory funnel is adjusted till the lower end of the funnel stem is about a half inch from the felt of the filter. It is then filled with the solution, and the stopper tightly inserted. When the stopcock is opened the filtration commences, and continues until all the solution has left the funnel stem. The stopcock is then closed, and the stopper removed, when the inside of the funnel can be washed with clean solvent. The stopper is inserted and the stopcock opened, allowing the operation to continue, washing the precipitated carbenes.



The parts of this apparatus are all glass, and therefore are not attacked by the vapors from the carbon tetrachloride.

If it is desired to make the operation continuous to the end without the necessity of watching for the emptying of the reservoir, another funnel containing fresh solvent may have its stem inserted in the tubular

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[1] THIS JOURNAL, 31, 839.

of the lower funnel and pushed down through the cork almost to the bottom of the lower globe. If the lower globe is covered with black paper, or painted with black paint, there will be no deposition of carbenes on the sides of the lower globe, and the vapors will drain the sides clean.

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THE ACTION OF ALCOHOLS, ACIDS AND AMINES ON METHYL OXOMALONATE.

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Anschütz and Parlato[1] produced ethyl oxomalonate, $O:C(CO_2C_2H_5)_2$, by the dissociation of ethyl acetylbromtartronate, when distilled under reduced pressure. They were not able to separate the two liquids and obtain the pure keto ester. They also prepared it by the distillation of ethyl dihydroxymalonate under reduced pressure, passing the vapors through a tube containing phosphorus pentoxide. This did not give a pure product inasmuch as the water formed in the process of distillation reassociates with the ethyl oxomalonate to form ethyl dihydroxymalonate.

Owing to the great reactivity of the carbonyl group of the ester, a strong dehydrating agent is required to remove the water as soon as it is dissociated. We tried at first, mixing the methyl dihydroxymalonate with zinc chloride, and with barium oxide, and distilling the mixtures, but with unsatisfactory results. We succeeded only when phosphorus pentoxide was chosen. This gave a yield of 92 per cent. of pure anhydrous methyl oxomalonate.

We have studied the reactions of ethyl, methyl and propyl alcohols on methyl oxomalonate. Ethyl alcohol gives a crystalline body, methyl ethoxytartronate, $(C_2H_5O)(HO)C(CO_2CH_3)_2$. The reaction products of methyl and propyl alcohols with methyl oxomalonate were thick glycerol-like oils, changed by moist air into alcohols and methyl dihydroxymalonate.

In a somewhat similar manner, Kuntz[2] has succeeded in adding several members of the alcohol series on the carbonyl group of chloral,

$$CCl_3.CHO + C_5H_{11}OH \longrightarrow CCl_3CH(OH)(OC_5H_{11}).$$

We have succeeded in adding hydrochloric and hydrobromic acids to methyl oxomalonate forming compounds of the structure

$$(HO)(Cl)C(COOCH_3)_2 \text{ and } (HO)(Br)C(COOCH_3)_2.$$

This class of compounds has few representatives. Vorländer[3] has

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[1] *Ber.*, **25**, 3616.

[2] *Chem. Centr.*, **1**, 79, 1560 (1908).

[3] *Ann.*, **341**, 1 (1905).