

The mixture was then brought to boiling and live steam passed through it until the total volume of distillate was 250 ml.

The steam distillate was extracted with two 75-ml. portions of ether and the ethereal extract dried over sodium sulfate. The ether was removed on the steam-bath and the residue vacuum-distilled: yield of phenylacetaldehyde, b. p. 87–88° (18 mm.), 5.4 g., 0.045 mole, 33%.

The other aldehydes studied were prepared by much the same procedure. The results are summarized in the table.

Aldehyde prepared	Time allowed for pptn. of addn. complex	Yield, %
Benzaldehyde ⁴	5 hours	97
β -Naphthaldehyde ⁵	12 hours	91
<i>p</i> -Tolualdehyde ⁴	12 hours	77
Phenylacetaldehyde ⁴	18 hours	33
Isocaproaldehyde	7 days	31
<i>o</i> -Tolualdehyde ^{7,8}	8 days	9
α -Naphthaldehyde ⁷	7 days	7
β -Hydroxypropionaldehyde ⁶	7 days	0

(4) Reported by Stephen² in almost quantitative yields.

(5) One liter of hot water was added to the residue and live steam passed through the mixture as long as any solid came over in the steam distillate. The solid was recrystallized from 50% aqueous alcohol. Fulton and Robinson, *J. Chem. Soc.*, **152**, 200 (1939), reported the preparation of β -naphthaldehyde by the Stephen technique in 75% yield. Their work was published after the preparation had been completed at the University of Maryland.

(6) Removal of the aldehyde by steam distillation was not attempted; 500 ml. of water was added to the addition complex, the mixture warmed on the steam-bath for one hour, allowed to cool and extracted with ether. The product was a thick, tarry liquid which gave negative tests with Schiff and Tollens reagents.

(7) Reported by Stephen² in poor yield.

(8) There was no precipitate after seven days so one-half of the ether was removed and the reaction mixture allowed to stand another twenty-four hours.

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Crystalline Lead Orthophosphate

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In the preparation of crystalline metathetical reaction products where the solubility of the reaction product is very low, it is often difficult to obtain crystals large enough for microscopic examination, and a number of methods have been employed for maintaining the necessary slight supersaturation. The writer has successfully employed a cellophane membrane for a number of such cases.

By treating a solution of lead acetate with sodium hydrophosphate, Aiders and Stähler¹ succeeded in preparing lead orthophosphate, but without any signs of microscopically recognizable crystals. In fact, it appears from the literature that the only method of preparing this salt is the rather cumbersome one of Zambonini,² who fused

(1) Aiders and Stähler, *Ber.*, **42**, 2263 (1909).

(2) Zambonini, *Z. Kryst.*, **58**, 226 (1923).

and slowly cooled the white powder. In our experiments, a cellophane membrane was tied over the mouth of a test-tube (an open tube could of course be used) containing a 1% solution of Na₂HPO₄ and the tube inverted in a slightly acidified 0.2% solution of Pb(C₂H₃O₂)·3H₂O. At once an almost amorphous precipitate of lead orthophosphate appeared on the under side of the membrane and in about a minute crystals of the white salt started to fall.

The crystals (some of which were 2 mm. in length) were uniaxial hexagonal prisms exhibiting strong birefringence and showing an optically negative character. The values of $\omega = 1.970$ and $\epsilon = 1.936$ check the data in the literature for the refractive indices of lead orthophosphate. Analysis of the crystals gave a value of 17.4% as compared with the calculated value of 17.5% for Pb₃(PO₄)₂.

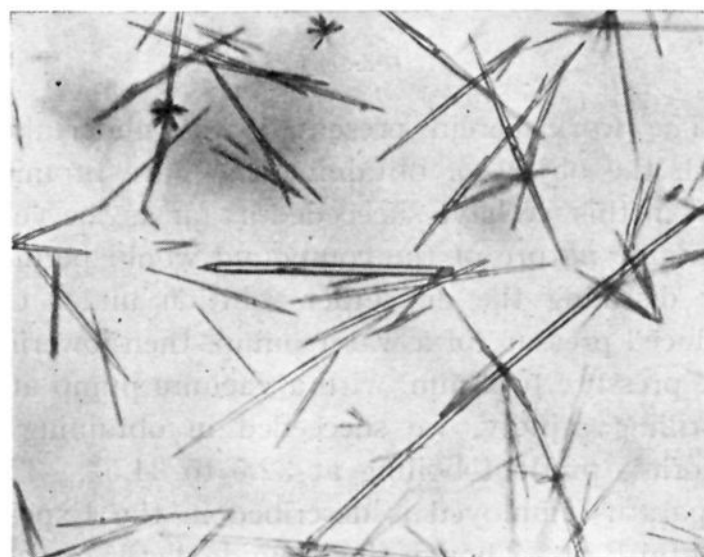


Fig. 1.

The accompanying photomicrograph at (50 X) shows some of the crystals obtained.

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α -Furfuryl Bromide (2-Bromomethylfuran)

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In 1927 one of us¹ prepared an ether solution of α -furfuryl bromide by acting on furfuryl alcohol with phosphorus tribromide. The solution proved satisfactory in the synthesis of various furfuryl ethers but the pure bromide was not separated as its decomposition was rapid once the solvent was evaporated. Von Braun and Kohler² had

(1) Zanetti, *THIS JOURNAL*, **49**, 1065 (1927).

(2) Von Braun and Kohler, *Ber.*, **51B**, 86 (1918).