compound $C_{26}H_{20}ON_2$, melting at 156–7°, and a white compound $(C_{13}H_{10}NO)_4$, melting at 193° with decomposition.

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An Improved Method of Preparing Diethylgermanium Derivatives

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In a previous paper[1] a method of preparing diethylgermanium derivatives, involving bromination of triethylgermanium fluoride, was described. A much simpler and more economical method of preparing these compounds is as follows.

Ethylgermanium triiodide and an excess of 40–60 lead–bismuth alloy are heated together, in the absence of air, to 150° for one or two days, shaking occasionally to subdivide the molten metal into droplets. When reaction appears complete the product is extracted with dry ether or ligroin, preferably in the absence of air. After distilling off the solvent the viscous yellow residue[2] is dissolved in ethyl iodide and the solution, in an air-free sealed tube, heated at 125° for two or three days. The excess ethyl iodide is now distilled off and the impure diethylgermanium diiodide is purified by fractional distillation *in vacuo*. If the oxide is desired, the impure diiodide may be hydrolyzed with aqueous sodium hydroxide, boiled for a few minutes, cooled, and the diethylgermanium oxide filtered off.

The characteristics of these new substances are as follows: diethylgermanium dichloride ($(C_2H_5)_2GeCl_2$); colorless liquid with pungent odor; b. p. 175° (758 mm.); m. p. -39 to -37°; Ge, calcd.: 36.01%; found: 36.03, 35.76%; Cl, calcd.: 35.18%; found: 35.26, 35.17%. Diethylgermanium diiodide ($(C_2H_5)_2GeI_2$); colorless liquid; b. p. 252° (759 mm.); m. p. -2 to -1°; Ge, calcd.: 18.88%; found: 18.90, 18.85%; I, calcd.: 66.02%; found: 66.45, 66.27%.

When the former method of preparation is used considerable difficulty is encountered in separating the diethylgermanium derivatives from the triethylgermanium compounds usually present, whereas in the present method the probable impurities are monoethylgermanium derivatives which, being water-soluble, are easily removed. Where a supply of germanous sulfide is available the simplicity of the present method is particularly marked in that only four steps are involved all of which may be carried out in good yields while some eight steps were involved in the former method.

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(1) Flood, THIS JOURNAL, **54**, 1667 (1932).

(2) Analyses of this residue indicated it to be impure $(C_2H_5GeI)_x$. Calcd.: I, 55.6%. Found: I, 58.5%. This interesting compound will be studied later.