in 50 ml. of ethanol). Trituration of the resultant gummy, red mass with ethanol and with acetone produced a tan solid (18.0 g.) which melted 75–94°. It was crystallized twice from absolute ethanol to give light tan platelets (17.0 g., 51% yield), m.p. 82–89°. The salt retained moisture and solvent of crystallization very firmly, and these could be removed only at 100° *in vacuo*, accompanied with some decomposition.

Anal. Calcd. for $C_{18}H_{27}N_3O.H_3PO_4$: N, 10.52; H_3PO_4, 24.54. Found:[12] N, 10.68; H_3PO_4, 24.58.

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(12) Corrected for 3.50% loss at 100°, *in vacuo*. Of this total, 1.04% was moisture, as determined by the Karl Fischer method.

7-Chloro-4-(4-dibutylaminobutylamino)-3-methylquinoline

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4-Aminoquinolines are well known for a considerable range of used as chemotherapeutic agents. Representatives of the group have antibacterial,[2] antimalarial,[3] and antitrypanosomal[4] activities, and also worth against extra-intestinal forms of amebiasis.[5–7] 7-Chloro-4-(4-dibutylaminobutylamino)-3-methylaminoline triphosphate has now been shown[8] to be effective against both intestinal and extra-intestinal forms of *Endameba muris*, the protozoan responsible for amebiasis in the hamster. The preparation of the drug was achieved by reaction of 4,7-dichloro-3-methylquinoline with 4-dibutylaminobutylamine, followed by conversion of the resultant base to the phosphate.

EXPERIMENTAL[9]

The reaction of 4,7-dichloro-3-methylquinoline[10,11] (21.2 g., 0.1 mole) with 4-dibutylaminobutylamine[12] (44 g., 0.22 mole) was run in phenol (60 g.) at 160–165° in the presence of a trace of potassium iodide. After 13 hr., the viscous mixture was cooled and quenched in an excess of cold aqueous sodium hydroxide, and the bases taken into methylene chloride. The mixture was extracted with a 2*N* hydrochloric acid, the bases then liberated, extracted with methylene chloride, dried, and fractionated. A 70% yield (25.6 g.) of the desired base was obtained as a viscous, golden oil; b.p. 190–193° (0.08 mm.); n_D^{25} 1.5741.

Anal. Calcd. for $C_{22}H_{34}ClN_3$: C, 70.28; H, 9.11; N, 11.18. Found: C, 69.98; H, 8.74; N, 11.18.

The base (25.5 g.) was dissolved in 150 ml. of propanol-2, chilled to 5°, and treated with a cold solution of 85% phosphoric acid (25.4 g.) in ethanol (100 ml.). A creamy-white phosphate resulted in crystalline form after scratching the vessel, and the solid was collected, washed (ether) and dried superficially. It was suspended in 500 ml. of boiling ethanol, boiling water added to effect solution, and then treated with charcoal. The pure 7-chloro-4-(4-dibutylaminobutylamino)-3-methylquinoline triphosphate (20.5 g., m.p. 186–186.6°) was obtained by two further crystallizations of the slightly impure salt (32.0 g.).

Anal. Calcd. for $C_{22}H_{34}ClN_3.3H_3PO_4$: N, 6.27; H_3PO_4, 43.90. Found: N, 6.17; H_3PO_4, 44.20.

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Preparation of Aliphatic Ketones from Lithium Alkyls and Dimethylamides

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The condensation of lithium alkenyls, alkyls, and aryls with dimethylamides to give aldehydes and methyl ketones as a preparative method has been studied by Evans and co-workers.[1,2] In the ketone series, Evans[2] prepared several methyl ketones in practical yields by reacting lithium alkyls with *N,N*-dimethylacetamide. The present paper describes an extended application of this reaction to the synthesis of other aliphatic low molecular weight ketones. This extension includes the use of longer chain *N,N*-dimethylamides and the comparison of yields by interchanging the alkyl chains associated with the carboxamide and lithium groups. For example, ethylisopentyl ketone was formed in 75% yield from the condensation of isopentyllithium and *N,N*-dimethylpropionamide and in 78% yield from ethyllithium and *N,N*,4-trimethylvaleramide. Incidental to this work, this method of ketone synthesis was compared with those involving the well known nitrile-Grignard, and acid halide–

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