Anal. Calcd. for $C_{18}H_{14}O_4N_2$: C, 67.07; H, 4.38; N, 8.70. Found: C, 67.39; H, 4.50; N, 8.86.

1-(7′-Chloro-2′-phenylquinoline-4′)-5-diethylaminohexanedione-1,3 (II) was prepared by acylating 1-diethylaminopentanone-4 (V) with ethyl 2-phenyl-7-chlorocinchoninate (VI) by means of sodium amide according to an adaptation of the method developed in this Laboratory.[11] To a stirred suspension of 0.4 mole of sodium amide[11] in 500 ml. of liquid ammonia was added rapidly 0.4 mole of the ketone in 75 ml. of dry ether. After replacing the ammonia by ether,[11] 0.2 mole of the ester in one liter of dry ether was added and the mixture refluxed five hours and then allowed to stand overnight. The mixture was poured onto 750 ml. of 10% acetic acid and crushed ice; after shaking thoroughly, the ether phase was extracted with 500 ml. of 10% acetic acid and combined with the aqueous phase. To the combined acetic acid solution was added cold 20% sodium hydroxide solution to a *p*H of 11 (oil separating). Carbon dioxide was passed into the mixture to a *p*H of 9, and the oil extracted with ether. The solvent was distilled from the dried ether solution leaving the crude oily β-diketone (79%) which, after drying over phosphorus pentoxide in a vacuum desiccator, was recrystallized from a mixture of benzene and 30–60° petroleum ether, freezing out with dry ice. There was obtained a 15% yield of pure β-diketone, melting at 88°; this melting point was not raised by further recrystallization but was depressed by admixture with the cinchoninic ester (VI).

Anal. Calcd for $C_{25}H_{27}O_2Cl$: C, 70.98; H, 6.44; N, 6.62. Found: C, 70.96, 70.61; H, 6.34, 6.17; N, 6.63, 6.40.

The β-diketone gave a deep red enol test with alcoholic ferric chloride, and formed a 2,4-dinitrophenylhydrazone which soon became oily. Attempts to convert the crude β-diketone to a picrate, pyrazole, hydrochloride or a copper salt failed. The crude β-diketone, which was entirely soluble in dilute hydrochloric acid, appeared to decompose on distillation at 0.1 mm., since the distillate, b. p. 175–190°, was not entirely soluble in the acid.

Hydrogenation of the crude β-diketone in glacial acetic acid at room temperature and low pressure in the presence of Adams catalyst[12] absorbed the calculated amount of hydrogen within two hours and produced an oil, b. p. 240–250° at 0.1 mm., which, as should be expected, failed to give the enol test with alcoholic ferric chloride; however, the pure 1,3-diol or a solid derivative of it has not been isolated.

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(11) Adams and Hauser, THIS JOURNAL, **66**, 1220 (1944); Levine, Adams, Conroy and Hauser, *ibid.*, **67**, 1510 (1945).

(12) Adams, Voorhees and Shriner, "Organic Syntheses," Coll. Vol. I, 463 (1946).

DEPARTMENT OF CHEMISTRY
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Identification of Esters of Dibasic Acids by the Use of Ethanolamine

BY WILLIAM H. RAUSCHER AND WILLIAM H. CLARK

In an attempt to find a rapid and satisfactory method for the identification of esters, their ammonolysis by ethanolamine in the absence of water has been investigated. It was hoped that on refluxing esters with ethanolamine that solid amides which would suitably characterize the acid would be formed, and that the alcohol simultaneously formed could be distilled out of the reaction mixture in such a state of purity that it could be easily identified in the usual manner.

On refluxing a number of esters with ethanolamine it was found that they were rapidly ammonolyzed, and that the alcohol could be distilled from the reaction mixture at a temperature very close to its boiling point. A redistillation of the alcohol from a simple distilling flask gave a product of correct boiling point from which a standard solid derivative of correct melting point was made. The reaction mixture from which the alcohol had been distilled did not yield solid amides on cooling in the case of most esters of monobasic acids which were investigated. However, the series of esters of dibasic acids shown in the table did give solid amides on cooling the reaction mixture after distilling off the alcohol. After a recrystallization the amides proved to have melting points suitable for purposes of identification.

Experimental

The reflux apparatus consisted of a 50-cc. flask and water cooled reflux condenser, both with ground joints. A mixture of 5 g. of the ester and 15 g. of ethanolamine was refluxed for fifteen minutes. After cooling below the boiling point of the alcohol, the condenser was replaced by a short Vigreux column and the alcohol distilled off. The residue was cooled to room temperature to obtain a solid product in the case of the esters of dibasic acids listed in the table. The amides were recrystallized from a 1:1 solution of alcohol and benzene. The alcohol which had been distilled from the reaction mixture was redistilled from a simple distilling flask, its boiling point checked and at least one solid derivative prepared from the distillate by standard procedures.

TABLE I

N,N′-DI-β-HYDROXYAMIDES OF DIBASIC ACIDS

Ester	M. p. (cor.), °C.	Nitrogen, % Calcd.	Nitrogen, % Found
Methyl oxalate[a]	169–169.2	15.95	15.90–16.03
Ethyl oxalate[a]	169–169.2	15.95	
n-Butyl oxalate[a]	169–169.2	15.95	
Ethyl malonate	127.0–127.5	14.71	14.73–14.81
Ethyl succinate	156.2–156.7	13.69	13.55–13.61
Ethyl glutarate[b]	119.6–120.0	12.85	12.86–12.99
Ethyl adipate	130.2–130.7	12.08	12.02–12.05
Ethyl suberate	138.5–138.9	10.77	10.88–10.79
Ethyl azelate	125.5–125.9	10.22	10.33–10.81
Methyl sebacate	144.5–145.0	9.72	9.77– 9.89

[a] Required no refluxing. [b] Dioxane used for recrystallization of amide.

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NEW COMPOUNDS

α-Amino-β-mercapto-*n*-valeric Acid Hydrochloride

α-Amino-β-mercapto-*n*-valeric acid hydrochloride was prepared by following the procedure of Carter, Stevens and Ney[1] for the corresponding butyric acid.

2-Phenyl-4-*n*-propylidene-5-oxazolone was synthesized by the method of Carter, Handler and Melville[2] using

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(1) Carter, Stevens and Ney, *J. Biol. Chem.*, **139**, 247 (1941).

(2) Carter, Handler and Melville, *ibid.*, **129**, 359 (1939).