SYNTHESIS OF NEW PYRROLIDONE DERIVATIVES.

I. THE REACTION OF N-CHLOROMETHYLPYRROLIDONE WITH MALONIC AND MONOSUBSTITUTED MALONIC ESTERS

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Reaction of N-chloromethylpyrrolidone with the sodio-derivatives of malonic and substituted malonic esters in dry ether has given the diethyl esters of the corresponding 2-oxopyrrolidinomethylmalonic acids.

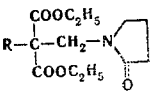
In order to synthesize organic compounds containing the lactam ring, the reaction of the diethyl esters of malonic, γ-chlorocrotylmalonic, and monoalkylmalonic acids with N-chloromethylpyrrolidine has been examined [1].

Reaction of N-chloromethylpyrrolidone with monosubstituted diethyl malonates in the presence of metallic sodium in dry ether affords esters of alkyl-(2-oxopyrrolidinomethyl)malonic acids. Diethyl malonate itself gives derivatives of mono- and bis-(2-oxopyrrolidinomethyl)malonic acid. In absolute alcohol, yields were much lower.

EXPERIMENTAL

Reaction of N-Chloromethylpyrrolidone with Diethyl Malonate. To 10.38 g (0.45 g-atom) of finely-divided sodium in 250 ml of dry ether was added slowly with stirring and cooling 72.3 g (0.45 mole) of malonic ester. The mixture was heated at 45-50° until the sodium had dissolved completely. N-Chloromethylpyrrolidone (60.3 g, 0.45 mole) was then added with stirring and cooling. The mixture was heated for 4 h, and kept for a day at room temperature. The resulting salt was dissolved in water, and the organic layer was separated and dried over anhydrous magnesium sulfate. Unreacted diethyl malonate (24.1 g) was distilled off, followed by 39.2 g of diethyl 2-oxopyrrolidinomethylmalonate (50.7%), bp 161-164° (4 mm);

TABLE 1



R	Bp, °C (press., mm)	n_D^{20}	d_4^{20}	MR_D Found	MR_D Calculated	Molecular formula	N, % Found	N, % Calculated	Yield, %
n-C_3H_7	176 (1,5)	1,4675	1,0867	76,50	76,35	$C_{15}H_{25}O_5N$	4,52	4,68	75
n-C_4H_9	170—176 (1)	1,4660	1,0700	80,99	81,17	$C_{16}H_{27}O_5N$	4,50	4,49	43
i-C_5H_{11}	196—203 (1)	1,4650	1,0585	85,37	85,66	$C_{17}H_{29}O_5N$	4,20	4,28	79
$CH_3CCl{=}CHCH_2$	199—202 (2)	1,4830	1,1473	85,01	85,55	$C_{46}H_{16}ClO_5N$*	4,36	4,05	89

*Found: Cl 10.05%. Calculated: Cl 10.27%.

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n_D^{20} 1.4655: d_4^{20} 1.1354. Found: 55.96; H 7.52; N 5.00%; MR_D 62.62. $C_{12}H_{19}NO_5$. Calculated: C 56.03; H 7.39%; N 5.44%; MR_D 62.69. The residue was an extremely viscous substance which crystallized. It was diethyl bis-(2-oxopyrrolidinomethyl)malonate, mp 57° (from benzene). Yield 27.8 g (26.4%). Found: C 57.3; H 7.12; N 8.36%. $C_{17}H_{26}N_2O_6$. Calculated: C 57.6; H 7.34; N 7.91%.

The diethyl esters of the substituted (2-oxopyrrolidonomethyl)malonic acids were obtained by analogous methods (see Table 1).

LITERATURE CITED

1. F. P. Sidel'kovskaya, M. G. Zelenskaya, and M. F. Shostakovskii, Izv. AN USSR, OKhN, 901 (1959).