SATURATED NITROGEN-CONTAINING HETEROCYCLES

V.* SYNTHESIS OF 5-ALKYL-N-(β-HYDROXYETHYL)-2-PYRROLIDONES

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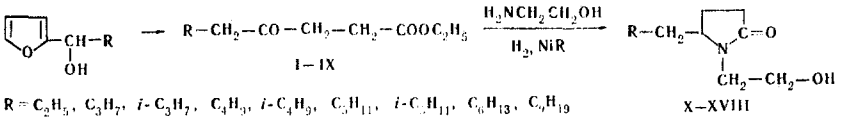
A number of 5-alkyl-N-(β-hydroxyethyl)-2-pyrrolidones were synthesized by the reductive ethanolamination of ethyl esters of γ-keto carboxylic acids in the presence of Raney nickel.

Compounds of the N-(β-hydroxyethyl)-substituted 2-pyrrolidone series have recently found application in the synthesis of practically valuable substances [2]. However, 5-alkyl-N-(β-hydroxyethyl)-2-pyrrolidones may also be of independent interest as possible physiologically active substances. The reaction of γ-lactones with ethanolamine [3] and the hydroxyethylation of pyrrolidones with ethylene oxide [2] are used for the synthesis of compounds of this type.

We have proposed a method for the synthesis of 5-alkyl-N-(β-hydroxyethyl)-2-pyrrolidones that consists in the reductive ethanolamination of ethyl esters of the γ-keto carboxylic acids [4, 5] obtained from furylalkylcarbinols [4-6].



R = C_2H_5, C_3H_7, i-C_3H_7, C_4H_9, i-C_4H_9, C_5H_{11}, i-C_5H_{11}, C_6H_{13}, C_9H_{19} X–XVIII

The reductive ethanolamination of esters of γ-keto carboxylic acids was carried out in a rotating autoclave at 100°C at a hydrogen pressure of 100-120 atm in methanol or ethanol in the presence of Raney nickel. A group of previously undescribed 5-alkyl-N-(β-hydroxyethyl)-2-pyrrolidones (X-XVIII) (Table 1) was thus obtained.

The 5-alkyl-N-(β-hydroxyethyl)-2-pyrrolidones are light-yellow, oily liquids that are stable on storage and quite soluble in water, ether, ethanol, methanol, dichloroethane, and benzene. Broad, intense absorption bands at 3400 cm^{-1}, which correspond to the stretching vibrations of associated OH groups, are observed in the IR spectra of X-XVIII. The stretching vibrations of the C=O bond appear as in intense band at 1690-1700 cm^{-1}.

EXPERIMENTAL

The ethyl esters of γ-ketoheptanoic (I), γ-ketooctanoic (II), γ-ketoisooctanoic (III), γ-ketononanoic (IV), γ-ketoisononanoic (V), γ-ketodecanoic (VI), γ-ketoisodecanoic (VII), γ-ketoundecanoic (VIII), and γ-ketopentadecanoic (IX) acids were obtained via previously described methods [4, 5].

5-Amyl-N-(β-hydroxyethyl)-2-pyrrolidone (XIII). A rotating autoclave was charged wtih 10 g (0.05 mole) of ethyl γ-ketononanoate in 20 ml of absolute ethanol, 3.9 g (0.06 mole) of ethanolamine in 10 ml of

*See [1] for communication IV.

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TABLE 1. 5-Alkyl-N-(β-hydroxyethyl)-2-pyrrolidones (X-XVIII)

Compound	R	Obtained from	Bp, °C (pressure, mm)	d_4^{20}	n_D^{20}	MR_D found	MR_D calc.	Empirical formula	Found, % C	Found, % H	Found, % N	Calc., % C	Calc., % H	Calc., % N	Yield, %
X	C_3H_7	I	140—142 (2)	1,0445	1,4842	46,92	47,088	$C_9H_{17}NO_2$	63,3	10,3	8,5	63,2	10,0	8,2	70
XI	C_4H_9	II	142—143 (2)	1,0271	1,4860	51,79	51,706	$C_{10}H_{19}NO_2$	64,4	10,3	7,6	64,8	10,3	7,6	70
XII	i-C_4H_9	III	170—172 (2)	1,0214	1,4821	51,71	51,706	$C_{10}H_{19}NO_2$	64,5	10,1	7,7	64,8	10,3	7,6	70
XIII	C_5H_{11}	IV	162—164 (2)	1,0088	1,4796	56,11	56,274	$C_{11}H_{21}NO_2$	66,4	10,8	7,2	66,4	10,6	7,0	70
XIV	i-C_5H_{11}	V	192—194 (2)	1,0123	1,4800	55,93	56,274	$C_{11}H_{21}NO_2$	66,4	10,9	6,9	66,4	10,6	7,0	70
XV	C_6H_{13}	VI	183—184 (2)	1,0005	1,4792	60,50	60,942	$C_{12}H_{23}NO_2$	67,9	10,9	6,7	67,6	10,9	6,6	70
XVI	i-C_6H_{13}	VII	196—197 (2)	0,9945	1,4772	60,63	60,942	$C_{12}H_{23}NO_2$	67,9	10,9	6,3	67,6	10,9	6,6	70
XVII	C_7H_{15}	VIII	201—202 (1)	0,9698	1,4709	65,50	65,51	$C_{13}H_{25}NO_2$	69,4	11,2	6,3	68,8	11,1	6,2	71
XVIII	$C_{10}H_{21}$	IX	220—222 (1)	0,8991	1,4640	79,01	79,364	$C_{16}H_{31}NO_2$	71,7	11,5	5,4	71,4	11,5	5,2	72

absolute ethanol, and 3 g of Raney nickel. The reaction was carried out at 100° and an initial hydrogen pressure of 100-120 atm until the calculated amount of hydrogen had been absorbed (8-10 h). The hydrogenation product was freed from the catalyst, the alcohol was removed by distillation, and the residue was vacuum-distilled. The other 5-alkyl-N-(β-hydroxyethyl)-2-pyrrolidones (X-XII, XIV-XVIII) (Table 1) was similarly obtained.

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