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SYNTHESIS AND PROPERTIES OF 3-OXA-7,11-DIAZASPIRO[5.6]DODECANES

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A method for the synthesis of 2,2-dialkyl-3-oxa-7,11-diazaspiro[5.6]dodecanes was developed on the basis of 4-bromo-4-formyltetrahydropyrans. The products were alkylated at the nitrogen atom in the 11 position by means of alkyl halides and acrylic acid derivatives, and the alkoxycarbonyl groups in the side chain were reduced.

In recent years considerable attention has been paid to methods for the synthesis and study of the biological properties of spiro biheterocyclic compounds [1-4]. Having an accessible method of synthesis of 4-bromo-4-formyltetrahydropyrans [5] at our disposal, we have developed a method for the synthesis of a new spiro biheterocyclic system in which the tetrahydropyran and perhydrodiazepine rings are spiro-bonded via the scheme



I-XIII a $R^1 = CH_3$; b $R^1 = C_2H_5$; R^2 see Table 1.

Halo aldehydes Ia,b react smoothly with β -methylaminopropionitrile [6] to give formyl nitriles IIa,b, which undergo hydrogenation with cyclization to give spiro amines IIIa,b in good yields at a hydrogen pressure of 110 atm (technical) at 90°C in the presence of Raney nickel. The N-monosubstituted perhydrodiazepine ring makes it possible to obtain numerous derivatives of this spiro biheterocycle (Table 1), some of which we synthesized with the aid of ethyl bromoacetate (IVa, b), propargyl bromide (Va, b), α -chloroacetamide (VIa,b), 5-phen-yl-l-chloro-2-hexene (VIIa,b), methyl acrylate (VIIIa,b), and acrylamide (IXa,b) as the reagents. We also obtained several alcohols (Xa,b, XIa) by hydrogenation of IVa,b and VIIIa with lithium aluminum hydride.

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TABLE 1. 2,7-Dimethy1-2-R¹-11-R²-3-oxa-7,11-diazaspiro[5.6]dodecanes (IV-XIII)

	ويعارفهم والمستعمل والمستعمل والمستعلق المستقر المستقر والمستعمل والمستعمل والمستعمل والمستعمل والمتنا والمستعم															
Com-	20	bp, deg C	n 20	4, 20	IR spectrum. cm ⁻¹	PMR enectrum. δ	⁷ R, min	Fot	md, %		Empirical	Calcu	llated,	%	% 'PI	bihydro- hloride
punod	ч	(mm)		5		mdd	or Rf	С	H	z	Tormula	υ	н	z	PIX	np, deg C fr. ethanol)
IVa	CH2COOC2H5	160—162 (3)	1,4895	1,0563	1090 (COC), 1760 (C)	3,26 (2H, s, NCH ₂)	4,35 (230)	64,5	10,2	9,4	C ₁₆ H ₃₀ N ₂ O ₃	64,4	10,1	9,4	74	9293
lVb	CH ₂ COOC ₂ H ₅	172173 (3)	1,4890	1,0549	1090 (COC),	3,26 (2H, s, NCH2)	5,5 (220)	65,1	10,1	0,0	C ₁₇ H ₃₂ N ₂ O ₃	65,3	10,3	9,0	70	78—80
Va	CH₂C≡CH	131-132 (3)	1,5050	0,9930	2120 (C = C), $2310 (C = C)$, CH ,	3,33 (2H, d, 1-9 9 Hz NCH.)	2,7 (210)	72,0	10,4	11,0	C ₁₅ H ₂₆ N ₂ O	71,9	10,5	11,2	60	145—146
٨b	CH₂C=CH	150-151 (4)	1,5080	0,9945	2120 (= CII) 2120 (C = C), 3300 (- CII)	3,23 (2H, d, 1,-9,9 Hz NCH)	3,7 (210)	72,4	10,6	10,7	C ₁₆ H ₂₈ N ₂ O	72,7	10,7	10,6	61	103105
VIa	CH2CONH2	211-213 (4)	1,5205		1690 (= 0), $(= 0)$, $3900 - 3450 (NHL)$	6,83,7,46 (2H,s)	0,64 (a)	62,6	6,6	15,5	C ₁₄ H ₂₇ N ₃ O ₂	62,8	10,1	15,6	68	187—189
qIA	CH2CONH2	235-236 (5)	1,5210		1690 (C=0), 1690 (C=0), 3200-3450 (NH3)	6,81, 7,43 (2H, s, NH ₃)	0,60 (a)	63,8	10,1	14,7	C ₁₅ H ₂₉ N ₃ O ₂	63,6	10,2	14,8	09	140-141
	CH ₃					ì										
VIIa	CH2CH=CHCH2CHC6H5	209-210 (2,5)	1,5300	1,0168	1680 (C=C), 1610 (C=Carom)	7,10 (5H, s , C ₆ H ₅); 5,36 (2H, t , <i>J</i> = =5,0 Hz, CH=CH)	0,52	78,0	10,4	7,6	C ₂₄ H ₃₈ N ₂ O	77,8	10,3	7,6	64	95—96
	CH ₃		1.11													
qIIV	CH ₂ CH=CHCH ₂ CHC ₆ H ₅	220-221 (2,5)	1,5310	1,0089	1680 (C=C), 1610 (C=Caron)	7,06 (5H, \mathbf{s} , C ₆ H ₅); 5,30 (2H, \mathbf{t} , $J = \frac{5}{6}$, \mathbf{u} , C_{H} , C_{H} , $J = \frac{5}{6}$, \mathbf{u} , C_{H} , C_{H} , $J = \frac{1}{6}$	0,50	77,8	10,3	7,2	C ₂₆ H ₄₀ N ₂ O	78,0	10,5	7,3	60	73—74
VIIIa	CH2CH2COOCH3	159-160 (2,5)	1,4925	1,0637	1080 (COC), 1750 (C)	3,60 (3H, s, OCH ₃)	4,6 (218)	64,2	10,1	9,3	C ₁₆ H ₃₀ N ₂ O ₃	64,4	10,1	9,4	76	108—110
VIIIb	CH2CH2COOCH3	167-168 (2)	1,4945	1,0607	1090 (COC),	3,60 (3H, s, OCH ₃)	6,2 (220)	65,1	10,4	6,0	C ₁₇ H ₃₂ N ₂ O ₃	65,4	10,3	9,0	70	9092
IXa	CH2CH2CONH2	220-222 (4)	1,5100		1690 (C=O), 1690 (C=O), 3900 3400 (NH)	6,9, 7,2 (2H, s, NH ₂)	0,55 (a)	63,5	10,1	14,6	C ₁₅ H ₂₉ N ₃ O ₂	63,5	10,2	14,8	102	180—181
IXb	CH2CH2CONH2	228-230 (3)	1,5210		1690 (C=O), 3200 3400 (NH	6,9, 7,2 (2H, s, NH ₂)	0,51 (a)	64,4	10,5	14,3	C ₁₆ H ₃₁ N ₃ O ₂	64,6	10,5	14,1	65]	170172
Xa	CH2CH2OH	152 (2)	1,5065	1,0375	2200-3700 (NI12) 1080 (COC), 3900 3500 (OH)	$3,40$ (2H, t, $1-6$ / H^{2} CH OH)	3,2 (220)	65,6	11,2	10,7	C ₁₄ H ₂₈ N ₂ O ₂	65,6	11,1	10,9	78	151—152
Хb	CH2CH2OH	161162 (2)	1,5080	1,0290	1080 (COC), 2900 3500 (OH)	3,43 (2H, t, 1,-60 Hz, CH OH)	4,15 (220)	66,7	11,3	10,4	C15H30N2O2	66,6	11,2	10,4	81	132—134
XIa	CH2CH2CH2OH	188-190 (5)	1,5070	1,0285	2200-3500 (OII) 1080 (COC), 2300 3500 (OII)	3.50 (2H, t)	4,3 (220)	66,7	11,0	10,1	C ₁₅ H ₃₀ N ₂ O ₂	66,6	11,2	10,4	67	195—197
XIIa	CH2COC6H5	216—218 (3)			1500, 1590 (COC), 1500, 1590 (C=Carom),	7,0-8,0 (5H, m, C.12011) 7,0-8,0 (5H, m, C.6H, m	0,69 (b)	72,8	9,3	8,3	C20H30N2O2	72,7	9,1	8,5	53	134
XIIb	CH2COC6H5	229—231 (3)			1000 (C=C) 1080 (COC), 1500, 1600 (C=Caron),	7,0—8,0 (5H, m, C ₆ H ₅)	0,62 (b)	73,5	9,4	8,2	$C_{21}H_{32}N_2O_2$	73,2	9,4	8,1	37	106—108
XIIIa	CH ₂ C(OH) (C ₆ H ₅) ₂	250—253 (3)			1000 (C=U) 1080 (COC), 1500, 1610 (C=Carom),		0,71 (c)	76,5	0,6	6,7	C26H36N2O2	76,4	8,9	6,9	20	135—136
AIIIX	CH ₂ C(OH) (C ₆ H ₅) ₂	268—270 (4)			3300—3500 (OH) 1080 (COC), 1500, 1610 (C=Caroni, 3300—3500 (OH)		0,65 (c)	76,9	6'8	6,4	C ₂₇ H ₃₈ N ₂ O ₂	76,7	9,1	6,6	45	[24—125

Com- pound	m/z values (relative intensities of the ion peaks in percent of the maximum peak)
IIIa	212 (21), 197 (26), 182 (9), 168 (26), 155 (74), 142 (21), 140 (100), 126 (21), 110 (18), 97 (40)
Шь	226 (23), 211 (17), 197 (100), 182 (19), 169 (27), 168 (33), 140 (47), 126 (23), 211 (17), 197 (100), 182 (19), 169 (27), 168 (33), 140 (47), 126 (23), 27 (30) 84 (30)
IVa	(298, (57), 283, (72), 225, (31), 211, (40), 182, (10), 168, (59), 155, (100), 140, (72), 97, (32), 84, (36)
VIIIa	(298) (21), 283 (35), 269 (20), 254 (8), 225 (10), 211 (10), 197 (20), 168 (54), 155 (100), 140 (86)

*The 10 most intense peaks in the mass spectrum of each compound are presented.

The structures of the compounds obtained were confirmed by IR and PMR spectroscopy (Table 1), whereas the structures of IIa, IIIa,b, IVa, and VIIIa were also confirmed by mass spectrometry (Table 2). The mass spectrum of IIa does not contain a molecular-ion peak but does contain an ion peak with m/z 195, the formation of which is associated with splitting out of the formyl group. We established a similar principle for substituted α -amino aldehydes of the tetrahydropyran series in [7]. The molecular masses of IIIa,b, IVa, and VIIIa, determined by mass spectrometry, correspond to the calculated values. Their structures are also confirmed by the presence of a number of characteristic peaks of fragment ions in their mass spectra.

EXPERIMENTAL

Gas-liquid chromatography (GLC) was carried out with a Khrom-4 chromatograph with a flame-ionization detector; the stationary phase was E-301 methylsilicone elastomer (6%) on Chromaton NAW (0.20-0.25 mm) treated with hexamethyldisiloxane, the column dimensions were 120 cm by 0.3 cm, the carrier gas was nitrogen, and the flow rate was 0.9 liter/h. The retention times (τ_R) are presented in Table 1. Thin-layer chromatography (TLC) for VI, IX, XII, and XIII was carried out in Silufol UV-254 plates in water-acetic acid (3:1) (a), water-dioxane-acetic acid (2:2:1) (b), and water-dioxane-acetic acid (3:3:1) systems, whereas TLC for VII was carried out on activity II aluminum oxide in benzene-chloroform (1:1). The chromatograms were developed with iodine vapors. The IR spectra of thin layers of the compounds were recorded with a UR-20 spectrometer. The PMR spectra of solutions in CC14 were recorded with a Varian T-60 spectrometer with tetramethylsilane as the internal standard. The mass spectra were obtained with an MKh-1303 spectrometer with direct introduction of the samples into the ion source.

2,2-Dimethyl-4-formyl-4-(methyl- β -cyanoethylamino)tetrahydropyran (IIa). A mixture of 22.1 g (0.1 mole) of 2,2-dimethyl-4-bromo-4-formyltetrahydropyran (Ia), 21 g (0.25 mole) of β -methylaminopropionitrile, and 100 ml of dry ether was refluxed for 18-20 h, after which it was cooled with ice water, acidified with 20% hydrochloric acid, and washed with ether. The aqueous solution was cooled with ice water and neutralized with concentrated potassium carbonate solution to pH 8 and extracted with ether. The extract was dried over anhydrous magnesium sulfate, the ether was removed, and the residue was distilled *in vacuo* to give 18 g (80%) of a product with bp 162-163°C (3 mm) and $n_D^{2°}$ 1.4870. IR spectrum: 1080 (COC), 1720 (C=0), 2730 (CHO), and 2270 cm⁻¹ (C=N). Found: C 64.4; H 8.9; N 12.7%. C₁₂H₂₀N₂O₂. Calculated: C 64.2; H 9.0; N 12.5%.

 $\frac{2-\text{Methyl-}2-\text{ethyl-}4-\text{formyl-}4-(\text{methyl-}\beta-\text{cyanoethylamino})\text{tetrahydropyran (IIb).} Under similar conditions we obtained 16.1 g (63%) of IIb, with bp 173-174°C (3 mm), and n_D²⁰ 1.4905, from 25.1 g (0.11 mole) of 2-methyl-2-ethyl-4-bromo-4-formyltetrahydropyran (Ib), 16.8 g (0.2 mole) of <math>\beta$ -methylaminopropionitrile, and 100 ml of ether. IR spectrum: 1070 (COC), 1730 (C=0), 2730 (CHO), and 2270 cm⁻¹ (C=N). Found: C 65.7; H 9.0; N 11.6%. C₁₃H₂₂N₂Q₂. Calculated: C 65.5; H 9.3; N 11.7%.

 $\frac{2,2,7-\text{Trimethyl}-3-\text{oxa}-7,11-\text{diazaspiro}[5.6]\text{dodecane}$ (IIIa). An autoclave was charged with 84.3 g (0.37 mole) of nitrile IIa in 300 ml of tert-butyl alcohol and 25 g of Raney nickel [8], and hydrogenation was carried out for 9 h at a hydrogen pressure of 110 atm (technical) and a temperature of 90°C. At the end of the reaction, the solution was filtered, the alcohol was removed by distillation, and the residue was distilled *in vacuo* to give 46.6 g (58%) of a product with bp 114-115°C (3 mm), $n_D^{2°}$ 1.5030, and $d_4^{2°}$ 1.0291. IR spectrum: 1080 (COC) and 3350 cm⁻¹ (NH). PMR spectrum: 1.05 and 1.27 [6H, s, 2,2-(CH₃)₂]; 2.25 (3H, s, 7-CH₃); 2.4-3.0 [4H, m, 8,10-(CH₂)₂] 3.2-3.9 (2H, m, 4-CH₂); 1.2-2.2 ppm [6H, m, 1.5,9-(CH₂)₃]. The retention time was 1.9 min (215°C). Found: C 67.8; H 11.4; N 13.2%; [M]⁺ 212. $C_{12}H_{24}N_{2}O$. Calculated: C 67.9; H 11.4; N 13.2%; M 212. The dihydrochloride of IIIa had mp 138-139°C (from ethanol).

 $\frac{2,7-\text{Dimethyl}-2-\text{ethyl}-3-\text{oxa}-7,11-\text{diazaspiro}[5.6]\text{dodecane (IIIb).} Under similar conditions we obtained 63.2 g (60%) of IIIb, with bp 125-126°C (3 mm), n₀^{2°} 1.5015, and d₄^{2°} 1.0584, from 110 g (0.46 mole) of IIb in 300 ml of tert-butyl alcohol and 30 g of Raney nickel. IR spectrum: 1080 (COC) and 3350 cm⁻¹ (NH). PMR spectrum: 1.0 and 1.25 (3H, s, 2-CH₃); 0.78 (3H, t, J = 5 Hz, 2-CH₂CH₃); 2.3 (3H, s, 7-CH₃); 3.2-3.8 (2H, m, 4-CH₂); 2.5-3.1 [4H, m, 8,10-(CH₂)₂]; 1.2-2.2 ppm [8H, m, 1,2,5,9-(CH₂)₄]. The retention time was 2.7 min (215°C). Found: C 69.1; H 11.7; N 12.4%; [M]⁺ 226. C₁₃H₂₆N₂O. Calculated: C 69.0; H 11.6; N 12.3%; M 226. The dihydrochloride of IIIb had mp 91-92°C (from ethano1).$

 $2-R^1-11-R^2-2$,7-Dimethyl-3-oxa-7,11-diazaspiro[5.6]dodecanes (IV-VII, Table 1). A 0.08mole sample of reagent R^2X (X = Br for the preparation of IV and V, whereas X = Cl for the preparation of VI and VII) was added dropwise with stirring to a mixture of 0.08 mole of IIIa or IIIb, 40 ml of acetonitrile, 20 g (0.14 mole) of potassium carbonate, and 10 ml of water, and the mixture was heated at 70°C for 7-8 h. It was then cooled, acidified with 20% hydrochloric acid, and washed with ether. The aqueous salt was cooled with ice water and made alkaline with concentrated potassium carbonate solution, and the alkaline solution was extracted with ether. The extract was dried with magnesium sulfate, the solvent was removed by distillation, and the residue was distilled *in vacuo*.

<u>11-(β -Methoxycarbonylethyl)</u>- and <u>11-(β -Carbamoylethyl)</u>-3-oxa-7,11-diazaspiro[5.6]dodecanes (VIII and IX, Table 1). A mixture of 0.02 mole of IIIa or IIIb and 0.03 mole of methyl acrylate or acrylamide was heated at 75-80°C for 7-8 h, after which it was distilled *in vacuo*.

<u>11-Hydroxyalkyl-3-oxa-7,ll-diazaspiro[5.6]dodecanes (X and XI, Table 1).</u> A 20-mmole sample of one of the esters (IVa,b, VIIIa) was added dropwise with cooling with a mixture of ice and salt at no higher than 0°C to 1 g (27 mmole) of lithium aluminum hydride in 60 ml of dry ether, after which the mixture was stirred at room temperature for 6-7 h and then cooled with a mixture of ice and salt. Water (4 ml) and 1 ml of 15% of sodium hydroxide was added dropwise with caution, and the mixture was stirred for 2 h and filtered. The ether was removed by distillation, and the residue was distilled *in vacuo*.

<u>11-Phenacyl-3-oxa-7,ll-diazaspiro[5.6]dodecanes (XII, Table 1).</u> A mixture of 40 mmole of IIIa or IIIb, 4 g (20 mmole) of bromoacetophenone, and 40 ml of dry ether was refluxed for 6-7 h, after which it was cooled with water, acidified with 20% hydrochloric acid, and washed with ether. The extract was dried with magnesium sulfate, the ether was removed by distillation, and the residue was distilled *in vacuo*.

<u>11-(β , β -Diphenyl- β -hydroxethyl)-3-oxa-7,11-diazaspiro[5.6]dodecanes (XIII, Table 1).</u> A solution of phenyllithium, prepared from 0.42 g (0.06 mole) of lithium, 4.7 g (0.03 mole) of bromobenzene, and 50 ml of dry ether, was added dropwise to a solution of 0.01 mole of ester IVa or IVb in 50 ml of dry ether in such a way that the ether boiled evenly. The mix-ture was then refluxed for 7 h, after which it was cooled, and 5-6 ml of water was added dropwise. The ether layer was dried with magnesium sulfate, the ether was removed by distillation, and the residue was distilled *in vacuo*.

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SYNTHESIS OF 4-CHLOROMETHYL-SUBSTITUTED PYRYLIUM SALTS

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The diacylation of methallyl chloride gave 2,6-dialkyl-4-chloromethylpyrylium salts, which were isolated in the form of stable perchlorates.

One of the principal methods for the synthesis of pyrylium salts is the diacylation of olefins [1]. In order to synthesize chloromethyl-substituted pyrylium salts and study their properties we carried out the diacylation of methallyl chloride for the first time.*

Monoacylation of methallyl chloride by the Kondakov method [3] gave alkyl γ -chloropropenyl ketones I, which proved to be intermediates in the syntheses of furans and pyrroles [4, 5].

In the present paper we describe the synthesis of 2,6-dialkyl-4-chloromethylpyrylium salts. The latter are of interest in connection with their ability to undergo three types of transformations, viz., recyclization with retention of the number of atoms in the ring, ring contraction [6], and reactions at the chloromethyl group [7], detailed information regarding which will be presented in a separate communication.

The diacylation of methallyl chloride makes it possible to obtain 2,6-dialkyl-4-chloromethylpyrylium salts IV, which were isolated in the form of crystalline perchlorates, in satisfactory yields.

In conformity with the generally accepted mechanism of the diacylation of olefins [8], the formation of the IV cations proceeds through intermediate β , γ -unsaturated ketones III.



IV a $R = CH_3$, b $R = C_2H_5$, c $R = C_3H_7$

Carboxylic acid anhydrides and 70% perchloric acid or acid chlorides and AlCl $_3$ were used as the acylating agents.

The IR and UV spectra of perchlorates IV are similar to the spectra of 2,4,6-trimethylpyrylium perchlorate. The PMR spectra contain signals of 2,6-dialkyl substituents at strong field, as well as singlets of CH₂Cl and ring protons.

In addition to salts IV, we also isolated liquid products that undergo partial decomposition during distillation *in vacuo* and darken on storage. The IR spectra of these products in the region of stretching vibrations contain strong absorption bands at 1720, 1690, 1665, and 1625 cm⁻¹, the first two of which we assigned to the vibrations of unconjugated (1720

*See [2] for our preliminary communication.

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