THE SYNTHESIS OF SOME NEW DERIVATIVES OF 1,7-DIMETHYLXANTHINE

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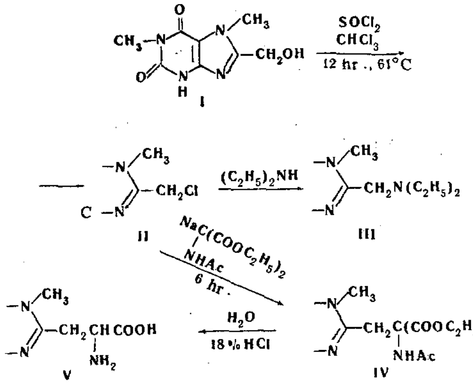
Khimiya Geterotsiklicheskikh Soedinenii, Vol. 3, No. 6, p. 1129, 1967

UDC 547.857.4.07

We have synthesized a group of new 8-substituted 1,7-dimethylxanthines which are of interest for biological investigation as very close structural analogs of some 8-substituted theobromine derivatives obtained previously. In particular, we have carried out the conversion of 8-hydroxymethyl-1,7-dimethlxanthine (I) [1] into 8-chloromethyl-1,7-dimethylxanthine (II) and from this, by heating it for 6 hours with an excess of diethylamine or by condensing it with sodioacetylaminomalonic ester in ethanol we have obtained 8-diethylaminomethyl-1,7-dimethylxanthine (III) and its hydrochloride (compare [2]) or 8-(β-acetylamino-β,β-diethoxycarbonylethyl)-1,7-dimethylxanthine (IV). The hydrolytic cleavage of the latter, accompanied by partial decarboxylation, led to 1,7-dimethyl-8-xanthinylalanine (V) (cf. [3]).



II: mp 250°—251° C (decomp., from a mixture of ethanol and dimethylformamide.) Found, %: Cl 15.47; N 24.63. Calculated for $C_8H_9ClN_4O_2$, %: Cl 15.53; N 24.57.

III: mp 192°—194° C (from 30% ethanol). Found, %: N 25.77. Calculated for $C_{12}H_{19}N_5O_2$, %: N 26.41. Hydrochloride: mp 260°—262° C (decomp.). Found, %: Cl 11.57; N 23.00. Calculated for $C_{12}H_{19}N_5O_2 \cdot HCl$, %: Cl 11.77; N 23.22.

IV: mp 229°—230° C (from ethanol). Found, %: C 50.17; H 5.79; N 17.29. Calculated for $C_{17}H_{23}N_5O_7$, %: C 49.87; H 5.62; N 17.11.

V: mp 265° C (from water). Found, %: C 42.40; H 5.13; N 24.56; H_2O 6.0. Calculated for $C_{10}H_{13}N_5O_4H_2O$, %: C 42.10; H 5.20; N 24.55; H_2O 6.3.

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1 August 1966

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SOME REACTIONS OF PHOSPHORUS-CONTAINING HETEROCYCLES

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Khimiya Geterotsiklicheskikh Soedinenii, Vol. 3, No. 6, p. 1130, 1967

UDC 661.718.1+547.77

It has been reported previously that derivatives of dioxaphospholene [1] and oxaphospholene [2] react readily with water. The reaction, which is a saponification of a cyclic ester, ends with the formation of the corresponding acid, which contains a keto group in the organic part of the molecule. The reaction of the cyclic compounds mentioned with alcohols has not been studied.

We have found [3] that derivatives of oxaphospholene readily react with alcohols on being heated, forming the corresponding esters of

Esters of Ketophosphonic and Ketophosphinic Acids

Compound	R	R′	Bp, °C (pressure, mm)	n_D^{20}	d_4^{20}	Empirical formula	Found				Calculated				Yield, %
							C, %	H, %	P, %	MR_D	C, %	H, %	P, %	MR_D	
I	OCH_3	C_2H_5	73—74 (0,04)	1.4467	1.0896	$C_9H_{19}O_4P$	48.67	8,43	13.81	54.45	48.64	8.62	13,94	54.64	65,8
II	OC_4H_9-*n*	C_2H_5	76—78 (0,02)	1.4429	1.0235	$C_{12}H_{25}O_4P$	54,46	10,03	11,66	68,46	54.53	9.53	11.72	68.49	41.6
III	C_2H_5	CH_3	71—72 (0,1)	1.4640	1.0638	$C_9H_{19}O_3P$	52,62	9.39	15.38	53.48	52,42	9,29	15,02	53.52	71,6
IV	C_2H_5	C_2H_5	78—80 (0,07)	1.4595	1,0362	$C_{10}H_{21}O_3P$	54.48	9,67	13.91	58,15	54,53	9.61	14,06	58.14	30,8
V	C_2H_5	H	Mp., 112—113	—	—	$C_8H_{17}O_3P$	49,52	8.64	15,78	192,10*	50,00	8.92	16,11	192.18*	89,0

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*Equivalent weight, found 192.10, calculated 192.18.