the pyrolysis of 2-(2-chloroethoxy)-ethyl acetate. Since the latter is a new compound, its preparation and pyrolysis are described. Diglycol chlorohydrin was acetylated with acetic anhydride, and the product (94% yield) was purified by distillation: b. p. 80° (1 mm.); d^{20}_4 1.1546; $n^{20}D$ 1.4398. Found: $M^{20}D$ 38.02; C, 43.3; H, 6.8. Calcd.: $M^{20}D$ 38.07; C, 43.3; H, 6.7. Pyrolysis over Pyrex glass at 500 and 550° (contact time, 8 sec.) decomposed 32 and 83%, respectively, of the ester but produced little if any chloroethyl vinyl ether. Most of the products were gases.

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Alkoxy-*s*-triazines. III

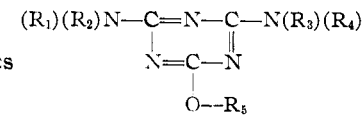
BY WILLIAM M. PEARLMAN, JACQUELINE DOWNS MITULSKI AND C. K. BANKS

In the search for antihistaminic compounds of the triazinyl ether type the alkyl 2,4-diamino-6-*s*-triazinyl ethers were found to have a peak of activity at the *n*-propyl compound.[1] A subsequent investigation of the methyl and butyl ethers of thirty substituted-aminotriazines disclosed no regular progression of activity as was noted in the first series.[2] Subsequently, an examination of the previously determined antihistaminic activities indicated that certain miscellaneous alkyl ethers should be prepared to determine if any products of appreciable activity had been overlooked. The ethers were prepared by previously described methods[1,2] from 2-chlorotriazines already described.[3]

The compounds were tested by Dr. Graham Chen and Mr. Charles Ensor of our laboratories by the histamine-aerosol technique of Dr. E. R. Loew.[2] The physical properties and effective antihistaminic values are recorded in the accompanying table.

TABLE I

ALKOXY-*s*-TRIAZINES



R_1, R_2	R_3, R_4	R_5	M. p., °C.	Yield, %	Recrystallization solvent[a]	Analyses,[b] % Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found	A. H.[c] value (effective dose, mg./kg.)
H_2	H_2	C_5H_{11}[d]	181–183	30	Ch	48.7	48.7	7.7	7.4	25
H_2	H_2	C_5H_{11}[e]	170–172	30	B	48.7	48.7	7.7	7.3	50
H_2	H, CH_3	C_2H_5	170–171	79	H_2O-E	42.6	42.8	6.6	6.7	12.5
H_2	H, CH_3	*n*-C_3H_7	175–177	68	H_2O-P	45.9	46.1	7.2	7.2	12.5
H_2	H, CH_3	*n*-C_6H_{13}	166–168	75	H_2O-E	53.3	53.7	8.5	8.5	>50
H_2	H, CH_3	*c*-C_6H_{11}	232–234	64	H_2O-MC	54.0	54.2	7.7	7.5	12.5
H_2	H, CH_3	Phenyl	211–213	64	H_2O-D	55.3	55.5	5.1	4.9	>25
H, CH_3	H, CH_3	C_2H_5	171–173	61	H_2O-E	45.9	46.1	7.2	7.3	12.5
H_2	$(CH_3)_2$	C_2H_5	156–158	88	B	45.9	46.2	7.2	7.2	25
H, CH_3	$(CH_3)_2$	C_2H_5	173–175	80[f]	B	48.7	48.6	7.7	7.6	25
H, CH_3	$(CH_3)_2$	*c*-C_6H_{11}	154	75[f]	M	57.3	57.6	8.4	8.4	>25
H_2	H, C_5H_{11}	C_2H_5	103–105	55	H_2O-E	53.3	53.4	8.5	8.2	>25
H_2	H, C_5H_{11}	*n*-C_3H_7	92–95	46	H_2O-E	55.2	54.8	8.9	8.7	..
H, C_2H_5	H, C_2H_5	C_2H_5	116–118	44	H_2O-E	51.2	51.2	8.1	8.2	>25
H, C_2H_5	H, C_2H_5	*n*-C_3H_7	82–84	88	H_2O-E	53.3	53.5	8.5	8.4	..
H_2	C_2H_5O,[g] C_6H_5	C_2H_5	194–196 d.	83	H_2O-EC	56.7	56.7	6.2	6.1	50

[a] All compounds were colorless: B = benzene, Ch = chloroform, D = dioxane, E = ethanol, EC = Ethyl Cellosolve, M = methanol, MC = Methyl Cellosolve, P = propanol. [b] Analyses by our Microanalytical Department under the direction of Messrs. A. W. Spang and C. E. Childs. [c] See ref. 2. [d] 3-Methylbutyl. [e] 2-Methylbutyl. [f] Prepared by Mr. John Controulis. [g] Hydroxyethyl.

(1) Controulis and Banks, THIS JOURNAL, **67**, 1946 (1945).

(2) Pearlman and Banks, *ibid.*, **71**, 1128 (1949).

(3) Pearlman and Banks, *ibid.*, **70**, 3726 (1948).

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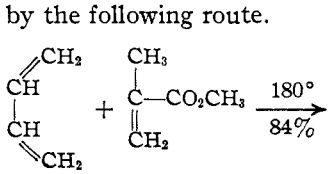
Synthesis of 3-Carbomethoxy-3-methylcyclopentanone

BY JOHN D. ROBERTS, A. K. JEYDEL AND ROSE ARMSTRONG

Ruzicka[1] has reported the preparation of 3-carbethoxy-3-methylcyclopentanone (I) through several steps from ethyl levulinate. In the present investigation, a shorter synthesis of the corresponding methyl ester (II) was achieved from the adduct of butadiene with methyl methacrylate by the following route.



(1) Ruzicka, *Ber.*, **50**, 1362 (1917).