TABLE I

Derivative	M. p., °C. (cor.)	Formula	Analyses, % Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found	Nitrogen Calcd.	Nitrogen Found	Other Calcd.	Other Found
p-Toluide	123	$C_{12}H_{15}ON$	76.15	75.46	7.99	7.87	7.40	7.62		
Hydrazide	156.0–156.5	$C_{11}H_{14}ON_2$	69.44	70.20	7.42	7.32	14.73	14.03		
p-Bromophenacyl ester	81.5–82.0	$C_{13}H_{13}O_3Br$	52.54	52.77	4.41	4.59			(Br) 26.89	27.07
p-Phenyl phenacyl ester	91.5	$C_{19}H_{18}O_3$	77.53	77.45	6.16	6.31				
Benzylisothiouronium salt	160–161	$C_{13}H_{18}O_2N_2S$	58.62	58.65	6.81	6.72	10.52	10.34	(S) 12.04	12.04

in this Laboratory by standard methods[2] and are listed in Table I. The anilide, m.p. 111°, has been reported previously.[3]

[2] Shriner and Fuson, "Identification of Organic Compounds," 3rd ed., John Wiley and Sons, Inc., New York, N.Y., 1944, pp. 154-159.

[3] Freund and Gudeman, *Ber.*, **21**, 2692 (1888).

DEPARTMENT OF CHEMISTRY
ILLINOIS INSTITUTE OF TECHNOLOGY
CHICAGO 16, ILLINOIS
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Non-reduction of the Acetal Group by Lithium Aluminum Hydride

BY C. S. MARVEL AND H. W. HILL, JR.[1]

We have recently had occasion to prepare the glycol acetal (II) and it occurred to us that the reduction of the corresponding malonic ester derivative (I) would produce this substance if the acetal group were not affected by the action of lithium aluminum hydride.

$$\underset{\text{I}}{(C_2H_5O)_2CHCH_2CH_2CH(CO_2C_2H_5)_2} \longrightarrow \underset{\text{II}}{(C_2H_5O)_2CHCH_2CH_2CH(CH_2OH)_2}$$

Preliminary tests of the action of lithium aluminum hydride on acetal, $CH_3CH(OC_2H_5)_2$, indicated no reaction. Hence the ester was prepared by the condensation of the diethylacetal of β-chloropropionaldehyde with sodium malonic ester[2] and the reduction of the acetal ester accomplished with lithium aluminum hydride. The glycol acetal (II) was isolated from the alkaline medium in 33% yield.

Preparation of 5-Hydroxy-4-hydroxymethylpentanal Diethyl Acetal.—When 83.3 g. (0.286 mole) of 3,3-diethoxy-1-propylmalonic acid diethyl ester was added dropwise over a period of 2.5 hours to a solution of 14.6 g. (0.385 mole) of lithium aluminum hydride in 300 ml. of absolute ether and the excess hydride decomposed by the cautious addition of water, a product which boiled at 140–144° at 1.2 mm.; $n^{20}D$ 1.4540; d^{20}_4 1.0178 was obtained upon distillation of the ethereal solution. The yield was 19.5 g. (33.2%).

Anal. Calcd. for $C_{10}H_{22}O_4$: C, 58.22; H, 10.75; *MR*, 54.72. Found: C, 58.33; H, 10.99; *MR*, 54.89.

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[1] Allied Chemical and Dye Corporation Fellow, 1949-1950. Department of Chemistry, Massachusetts Institute of Technology, Cambridge, Massachusetts.

[2] D. T. Warner and O. A. Moe, THIS JOURNAL, **70**, 3470 (1948).

NOYES CHEMICAL LABORATORY
UNIVERSITY OF ILLINOIS
URBANA, ILLINOIS
RECEIVED JULY 31, 1950

Polyalkylene Sulfides. VII. The Polymer from Tetramethylenedithiol and Biallyl

BY C. S. MARVEL AND ALEX KOTCH

Marvel and Chambers[1] added tetramethylenedithiol to biallyl under the influence of ultraviolet light to obtain a low-molecular weight polymer. This addition reaction carried out in emulsion according to the method recently described[2] gives a polymer with an inherent viscosity of 0.52 which melts at 64–67°. The polymer can be cold drawn to give a fiber. Surprisingly enough we have also been able to form this polymer with an inherent viscosity of 0.63 and m.p. 65–68° from tetramethylene bromide and the disodium salt of hexamethylenedithiol in a benzene–alcohol mixture.

This condensation polymerization reaction was carried out by dissolving 0.46 g. of sodium in 15 ml. of absolute alcohol (distilled from magnesium ethoxide[3]) and then adding 1.5 g. of hexamethylenedithiol. The sodium salt of the dithiol precipitated and then redissolved when the mixture was heated. To this boiling solution were added 25 ml. of dry thiophene-free benzene and then 2.15 g. of tetramethylene bromide. Immediately a vigorous reaction set in, and vigorous refluxing of the solvent mixture occurred. When the spontaneous reaction subsided, the mixture was diluted with an additional 25 ml. of benzene and then heated under refluxing conditions overnight. The cold, filtered solution was poured into methanol, and the polymer was collected on a filter. The yield was 1.08 g. of polymer, m.p. 63–65° with an inherent viscosity of 0.36. The benzene insoluble material was treated with water and an additional 0.26 g. of polymer, m.p. 65–68°, with an inherent viscosity of 0.63 was obtained.

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[1] C. S. Marvel and R. R. Chambers, THIS JOURNAL, **70**, 993 (1948).

[2] C. S. Marvel and P. H. Aldrich, *ibid.*, **72**, 1978 (1950).

[3] L. F. Fieser, "Experiments in Organic Chemistry," D. C. Heath and Company, New York, N. Y., 1941, p. 359.

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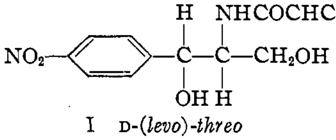
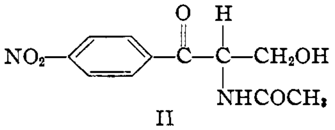
Chloromycetin.[1] Synthesis of α-Dichloroacetamido-β-hydroxy-*p*-nitropropiophenone

BY LOREN M. LONG AND H. D. TROUTMAN

In an earlier paper[2] the authors describe a method for the preparation of D-(*levo*)-*threo*-2-dichloroacetamido-1-*p*-nitrophenyl-1,3-propanediol (Chloromycetin, I) in which a necessary intermediate is *p*-nitroacetophenone. One of the latter steps in the synthesis involves the preparation of α-acetamido-β-hydroxy-*p*-nitropropiophenone (II). A comparison of the structure of II with that of I suggests that the substitution of a dichloroacetyl



I D-(*levo*)-*threo*



II

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[1] Parke, Davis & Co. registered trademark for chloramphenicol.

[2] L. M. Long and H. D. Troutman, THIS JOURNAL, **71**, 2473 (1949).