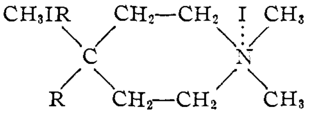
TABLE I



R′	R	Formula	M.p., °C.	Yield, %	Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found	Iodine Calcd.	Iodine Found	Activity,[a] mg./kg. ED50	Activity,[a] mg./kg. LD50
C_6H_5–	$(CH_3)_2NCH_2CH_2OCH_2$–	$C_{19}H_{34}I_2N_2O$	243.8–245.7	73	40.69	40.69	6.12	6.08	45.30	45.20	20	60 ± 2
	$(C_2H_5)_2NCH_2CH_2OCH_2$–	$C_{21}H_{38}I_2N_2O$	208.4–209.5	93	42.87	42.71	6.51	6.57	43.14	42.85	16 ± 1	32 ± 2
	$OC_4H_8NCH_2CH_2OCH_2$–	$C_{21}H_{36}I_2N_2O_2$	249–250 dec.	94	41.87	41.95	6.09	6.18	42.14	41.80	16 ± 1	62 ± 6
	$C_5H_{10}NCH_2CH_2OCH_2$–	$C_{22}H_{38}I_2N_2O$	240.0–241.6	82	44.00	43.88	6.38	6.38	42.28	41.89	8 ± 0.7	30 ± 6
	$C_5H_{10}NCH_2CH_2CH_2OCH_2$–	$C_{23}H_{40}I_2N_2O$	270.4–272.5	54	44.96	45.19	6.56	6.47	41.31	40.78	5.3 ± 0.3	14
	$C_5H_{10}NCH_2CH_2O$–	$C_{21}H_{36}I_2N_2O$	251.5–252.5	90	43.01	43.22	6.19	6.17	43.29	43.05	70 ± 7	280
H–	$C_5H_{10}NCH_2CH_2OCH_2$–	$C_{16}H_{34}I_2N_2O$	280–282 dec.	43	36.66	36.59	6.54	6.54	48.43	48.09	20 ± 2	70 ± 6
H–	$(C_2H_5)_2NCH_2CH_2OCH_2$–[b]	$C_{15}H_{34}I_2N_2O$	234.6–235.8	68	35.16	35.32	6.69	6.69	49.56	49.15	23 ± 1	60 ± 4

[a] C. J. Cavallito, A. Soria and J. Hoppe, THIS JOURNAL, **72**, 2661 (1950). Curarimimetic dose injected subcutaneously in mice. Dr. Hoppe carried out the biological screening. [b] R is on the 3-position.

solution was added 1-butanol (3.5 l.) and the combined solution distilled until the distilling vapors reached 117°. After cooling to 50°, sodium (115 g., 5 moles) was added portionwise until the violent reaction subsided. Then the remainder of the sodium was added and the mixture refluxed for an hour after all the sodium had been consumed. The reaction mixture was cooled slightly and water (100 ml.) was carefully added to the hot solution. Butanol was steam distilled from the mixture and the residual two-phase system poured into a beaker. The oil started to solidify immediately. The product was collected, dissolved in benzene, dried azeotropically and then Skelly B added to the first permanent cloud. On cooling, 185 g. (90% of theory) of product was obtained, m.p. 136.8–137.4°. An additional 10 g. was obtained on concentrating the filtrates.

Anal. Calcd. for $C_{13}H_{19}NO$: N, 6.82. Found: N, 6.73.

Ethers.—The alcohol was added to the sodamide (10% excess) suspended in dry toluene and refluxed until complete solution was effected. After cooling slightly, a second portion of sodamide (equivalent to the first portion) was added, followed by an equimolar quantity of the *t*-amino substituted alkyl halide hydrochloride. The resultant mixture was refluxed two hours, cooled and water carefully added to dissolve any unreacted sodamide and the salts that had formed. The aqueous layer was extracted with benzene which was combined with the organic layer, the solvent removed and the residual oil distilled or else converted directly to the dimethiodide.

Quaternaries.—The basic ether was dissolved in benzene and an excess of methyl iodide was added. If necessary, the solution was warmed to ensure complete reaction. The product was collected and crystallized several times from ethanol.

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Synthesis of β,β-Dicarbethoxytetrahydrofuran

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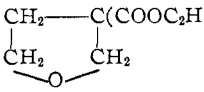
β,β-Dicarbethoxytetrahydrofuran (I) obtained in place of α-oxymethyl-α-carbethoxy-γ-butyrolactone (II) is interesting not only because of the preferential formation of the ether to that of the ester, *i.e.*, lactone linkage but also for its antibacterial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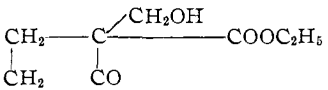
II

activity. The details of this work will be published in due course.

Experimental

Four grams (0.05 mole) of ethylene chlorohydrin was added with constant shaking to the sodio salt obtained from 9.5 g. (0.05 mole) of oxymethylmalonic ester[1] and sodium ethoxide prepared from 1 g. of sodium metal (0.05 mole) and 15 ml. of absolute alcohol. The reaction mixture on keeping at 30° for 48 hr. or on refluxing for 8 hr. at steam temperature yielded a liquid b.p. 152–153° (0.6 mm.) in amounts varying between 3.6 g. (33.3%) and 4.85 g. (44.9%).

Anal. Calcd. for $C_{10}H_{16}O_5$: C, 55.55; H, 7.46. Found: C, 55.68; H, 7.16.

The liquid on hydrolysis with 15% hydrochloric acid for 30 hr. yielded a solid acid, m.p. 115–120°, crystallizations from water raised the m.p. to constant 129–130°.

Anal. Calcd. for $C_5H_8O_3$: C, 51.72; H, 6.90; sapn. equiv., 116. Found: C, 51.95; H, 7.00; sapn. equiv., 114, 117.

Acknowledgments.—Authors' thanks are due to Dr. D. M. Bose, Director, Bose Institute, and to Dr. J. K. Chowdhury, Head of the Department of Chemistry, Bose Institute, for their kind interest in this work.

[1] S. Matsuura, *J. Pharm. Soc. Japan*, **71**, 525 (1951); *cf. C. A.*, **46**, 906 (1952).

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The Production of Tocored upon the Oxidation of *dl*-α-Tocopherol with Ferric Chloride[1]

BY VERNON L. FRAMPTON, WILFRED A. SKINNER, JR., AND PHILIP S. BAILEY

RECEIVED JUNE 30, 1952

All of the chemical methods used for the determination of tocopherols involve their oxidation. The most widely used procedure is that proposed by Emmerie and Engel,[2] which involves the rupture of the chroman ring with ferric chloride and the production of α-tocoquinone. A stoichiometric relationship is assumed, and the ferrous iron produced is determined colorimetrically as the red α,α′-bipyridyl complex.

Upon the oxidation at room temperature of *dl*-α-tocopherol (absorption maximum at 292 mμ with an extinction coefficient, $E^{1\%}_{1\text{cm.}}$ 74.2) with ferric chloride in methyl alcoholic solution, the absorption maximum at 292 mμ disappeared in about 45 seconds, and an absorption maximum which showed an

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[1] Taken from a dissertation presented by Wilfred A. Skinner, Jr., to the Graduate Faculty of The University of Texas in partial fulfillment of the requirements of the Ph.D. Degree, June, 1952.

[2] A. Emmerie and C. Engel, *Rec. trav. chim.*, **57**, 1351 (1938).