

TABLE I
ALKYL HALF ESTER OF *cis*-3,6-ENDOMETHYLENE- Δ^4 -TETRAHYDROPHTHALIC ACID

R	M.p., °C.	Formula	Carbon, %		Hydrogen, %		Acid no.	
			Calcd.	Found	Calcd.	Found	Calcd.	Found
CH ₃ ^a	101-102	C ₁₀ H ₁₂ O ₄	61.21	61.35	6.17	6.08	196	195
C ₂ H ₅	74-75	C ₁₁ H ₁₄ O ₄	62.68	63.12	6.72	6.63	210	212
<i>n</i> -C ₃ H ₇	71-72	C ₁₂ H ₁₆ O ₄	64.31	64.44	7.19	7.10	224	226
<i>n</i> -C ₄ H ₉	65-66	C ₁₃ H ₁₈ O ₄	65.56	65.63	7.62	7.22	238	240
<i>n</i> -C ₆ H ₁₁	51-52	C ₁₄ H ₂₀ O ₄	66.64	66.95	7.99	7.73	252	254
<i>n</i> -C ₈ H ₁₃	52-53	C ₁₆ H ₂₂ O ₄	67.68	68.00	8.33	7.98	266	270
<i>n</i> -C ₇ H ₁₅	43-44	C ₁₆ H ₂₄ O ₄	68.59	68.88	8.63	8.28	280	279
<i>n</i> -C ₈ H ₁₇	32-33	C ₁₇ H ₂₆ O ₄	69.32	69.24	8.99	9.05	294	290
<i>n</i> -C ₉ H ₁₉	45-46	C ₁₈ H ₂₈ O ₄	70.10	70.01	9.15	9.13	308	310
<i>n</i> -C ₁₀ H ₂₁	50-51	C ₁₉ H ₃₀ O ₄	70.77	71.10	9.38	9.38	322	321
<i>n</i> -C ₁₁ H ₂₃	53-54	C ₂₀ H ₃₂ O ₄	71.39	71.60	9.62	9.46	336	333
<i>n</i> -C ₁₂ H ₂₅	52-53	C ₂₁ H ₃₄ O ₄	71.96	72.13	9.78	9.61	350	356
<i>n</i> -C ₁₃ H ₂₇	53-54	C ₂₂ H ₃₆ O ₄	72.46	72.08	9.95	9.98	364	365
<i>n</i> -C ₁₄ H ₂₉	59-60	C ₂₃ H ₃₈ O ₄	73.24	73.17	10.12	10.44	378	373
<i>n</i> -C ₁₅ H ₃₁	59-60	C ₂₄ H ₄₀ O ₄	73.42	73.26	10.27	10.18	392	389
<i>n</i> -C ₁₆ H ₃₃	63-64	C ₂₅ H ₄₂ O ₄	73.84	73.70	10.41	10.44	406	402
<i>n</i> -C ₁₇ H ₃₅	63-64	C ₂₆ H ₄₄ O ₄	74.24	73.98	10.54	10.48	420	414
<i>n</i> -C ₁₈ H ₃₇	69-70	C ₂₇ H ₄₆ O ₄	74.61	74.46	10.67	10.64	435	432

^a Prepared by Morgan, Tipson, Lowy and Baldwin, *THIS JOURNAL*, 66, 404 (1944).

discontinued and the temperature rose spontaneously to 160° and was maintained at this degree by intermittent application of heat for 15 minutes. After cooling the oily half esters were purified by dissolving them in 10% sodium carbonate solution, followed by filtration. The filtrate was extracted twice with ether and acidified with 10% hydrochloric acid. The aqueous solution was decanted and the oily residue dissolved in benzene. After filtering the benzene solution was washed with water and extracted with 10% sodium bicarbonate solution. The extract was made acid with 10% hydrochloric acid and the free acid ester which separated was washed with water and dissolved in benzene. After drying over sodium sulfate the solvent was removed by distillation under vacuum. The half esters were further purified by dissolving them in petroleum ether and freezing them out by immersion in an acetone-Dry Ice mixture. This procedure was repeated until a constant melting compound was obtained after drying for 24 hours in a vacuum.

The Half Esters Tridecyl through Octadecyl.—A mixture of the appropriate alcohol and equivalent molar amount of the anhydride was heated for 12 hr. at 125°. The mixture was stirred occasionally during the heating to give a homogeneous liquid. The cooled mixture was poured into an excess of 10% sodium carbonate and stirred until the solid dissolved. The solution was extracted with benzene and filtered through a wetted filter. An excess of 10% hydrochloric acid was then added and the oily precipitate was allowed to harden, then filtered and dried. The product was recrystallized from petroleum ether until constant melting after drying 24 hr. in vacuum.

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The Si-C Bond Distance in Si(CH₃)₄

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A reinvestigation of tetramethylsilane by electron diffraction¹ has led to the result Si-C = 1.888 ± 0.02 Å. (previous report²: 1.93 ± 0.03 Å.), in agreement with the values found for carborundum³

(1) See K. Hedberg and A. J. Stosick, *THIS JOURNAL*, 74, 954 (1952), for details of the methods used.

(2) L. O. Brockway and H. O. Jenkins, *ibid.*, 58, 2036 (1936).

(3) N. W. Thibault, *Am. Mineral.*, 29, 249, 327 (1944); L. S. Ramsdell, *ibid.*, 29, 431 (1944); 30, 519 (1945).

(1.88-1.90 Å.), the three other methylsilanes⁴ (1.87 Å.), and Si₂(CH₃)₆⁵ (1.90 ± 0.02 Å.).

The observed diffraction pattern, which has eleven rings extending to $s = 32$, is well represented by the simplified intensity curve calculated with appropriate temperature factors for a symmetrical (T_d) model with the methyl groups in the staggered orientation, as held by independent 1.3 kcal./mole threefold potential barriers,⁶ and with Si-C = 1.89, Si-H = 1.10, and \angle Si-C-H = 110°; the average of the deviations $|(s_{\text{calcd}}/s_{\text{obsd}}) - (s_{\text{calcd}}/s_{\text{obsd}})|$ is 0.005 for the eleven well-located and reasonably symmetrical features used for the scale determination. Models with opposed methyl groups are unsatisfactory, both in the position and qualitative aspects of the first five or six rings, and it seems certain that the methyl groups are indeed predominantly staggered.

The final results are: \angle Si-C-H, 110 ± 3°; C-H, 1.10 ± 0.05 Å.⁷; and Si-C, 1.888 ± 0.02 Å.⁷

(4) L. O. Brockway and A. C. Bond, Second Int. Cong. Cryst., Stockholm (1951), Abstr. ED 12.

(5) L. O. Brockway and N. R. Davidson, *THIS JOURNAL*, 63, 3287 (1941).

(6) J. G. Aston, R. M. Kennedy and G. H. Messerly, *ibid.*, 63, 2343 (1941).

(7) Including 0.8% estimated limit of scale error.

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Antitubercular Studies. III. Hydroxylamines and Thiosemicarbazones

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Our interest in antitubercular studies has led us to synthesize a number of compounds with varying structures for testing against tuberculosis. Among these substances there have been O-substituted