C, 76.71; H, 5.07. Treatment of the isoxazole with sodium methoxide[5] followed by methyl iodide gave 2-cyano-1-keto-7-methoxy-2-methyl-1,2,3,4-tetrahydrophenanthrene, IV; m. p. 135–137.5° cor. *Anal.* Calcd. for $C_{17}H_{15}O_2N$: C, 76.96; H, 5.70. Found: C, 77.10; H, 5.90.

The condensation of IV with diethyl succinate in the presence of potassium *t*-butoxide[1] afforded 15-carbethoxy-14,15-dehydroequilenin methyl ether, V; m. p. 183.6–184.2° cor. *Anal.* Calcd. for $C_{22}H_{22}O_4$: C, 75.41; H, 6.33. Found: C, 75.64; H, 6.47. The free acid melted at 196.5–197.5° cor. *Anal.* Calcd. for $C_{20}H_{18}O_4$: C, 74.52; H, 5.63. Found: C, 75.01; H, 5.49. This acid decarboxylated readily on heating under reduced pressure to give a dehydroequilenin methyl ether (probably VI); m. p. 161.5–162.5° cor. *Anal.* Calcd. for $C_{19}H_{18}O_2$: C, 81.99; H, 6.52. Found: C, 82.07; H, 6.36. Hydrogenation over palladium-charcoal catalyst afforded an easily separable mixture of two compounds. The more soluble substance, m. p. 129–130° cor., was probably racemic *iso*-equilenin methyl ether (reported m. p. 127–127.5° and 130–130.5°[3]). The preponderant component of the mixture was racemic equilenin methyl ether; m. p. 189–190.5° cor. (reported[3] 185–186.5°). Demethylation followed by resolution according to the excellent procedures of Bachmann, Cole and Wilds[3] gave *d*-equilenin m. p.[6] 257.4–258.2° cor., undepressed on admixture with a specimen of the natural product, m. p.[6] 258.2–259.2° cor. The *l*-menthoxyacetate melted[6] at 177.2–177.8° cor., and the acetate at 153.5–157° cor. The mixed melting points with the corresponding derivatives prepared from natural equilenin likewise showed no depression.

The above synthesis exemplifies a process which promises to be of general use. We are now engaged in investigating the method in connection with an attempt to synthesize estrone.

(6) Melting point taken in an evacuated tube.

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RECEIVED OCTOBER 23, 1945

THE STRUCTURE OF HEXADECAMETHYLCYCLOOCTASILOXANE

Sir:

Hexadecamethylcyclooöctasiloxane, $[(CH_3)_2SiO]_8$, was isolated from two sources: (1) from the vacuum destructive distillation product of high-polymer dimethylsiloxane fluids and (2) from the low molecular weight fraction of completely hydrolyzed diethoxydimethylsiloxane. The purified material melting at 31.5 ± 0.5° formed transparent water-white crystals of columnar habit. These crystals were rather brittle and showed no definite cleavage. Laue photographs taken with the X-ray beam parallel to the column axis (*c*-axis) and also parallel to the *a*-axis revealed the diffraction symmetry D_{4h}-4mmm. The presence of a piezoelectric effect and absence of optical activity were established, thus restricting the point-group symmetry of the crystal to D_{2d}-$\bar{4}$2m or C_{4v}-4mm. Oscillation and rotation diagrams were obtained with [001] and [110] as axes of rotation. The unit cell (a = 13.95 ± 0.06 kX, c = 8.55 ± 0.04 kX) was found to be primitive and to contain two $[(CH_3)_2SiO]_8$. The systematic absences of (*hhl*) *reflections with l odd and of (h00) with h odd led to* the space group D_{2d}^4-P$\bar{4}$2, *c*. Evaluation of the (x, y) parameters substantiated the ring structure of hexadecamethylcyclooöctasiloxane as illustrated in Fig. 1. The "puckered" nature of the silicon-oxygen ring is expected in all cyclosiloxanes except $[(CH_3)_2SiO]_3$ which has been found to possess an essentially planar configuration similar to that in benitoite.

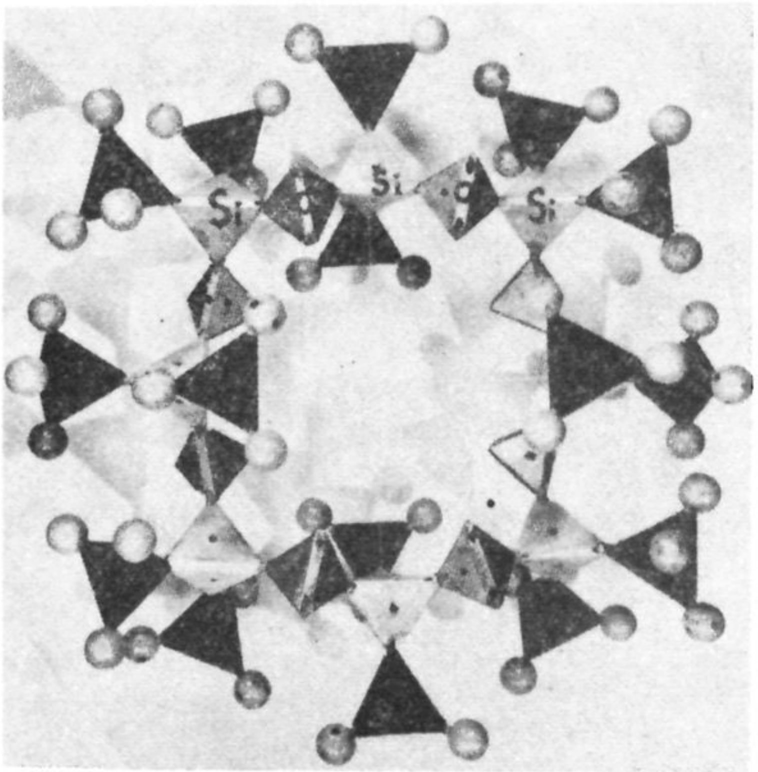

Fig. 1.—Configuration of hexadecamethylcyclooöctasiloxane molecule.

A detailed crystal structure analysis of $[(CH_3)_2SiO]_8$ will be published later.

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RECEIVED SEPTEMBER 26, 1945

SOLID SOLUTIONS IN HYDROCARBON SYSTEMS

Sir:

As a result of the evaluation of the purity of hydrocarbon samples by the freezing point depression method at the Cryogenic Laboratory, Pennsylvania State College, and at the Research Department, Phillips Petroleum Company, the existence of six binary hydrocarbon systems which