

cated the methyl group was sweeping out a large umbrella-like surface, as though oscillating with a large amplitude at a fixed radius. This concept was introduced into the calculations by assuming the contribution of each methyl group to result from a precession about its equilibrium position. Important discrepancies between calculated and observed  $F^2$  values immediately were resolved, and parameters now are being successfully refined. Because of steric reasons, two methyl groups attached to the same silicon atom could not oscillate independently over such a large area, so this is tantamount to assuming the Si atom is free to move in the ring, as in a ball and socket joint.

We consider this evidence for the extreme ionic nature of bonds in the silicone compounds. Bonds may form in the average covalent directions, but there is little resistance to angular deformation. The effect was observed only for methyl groups, probably because movement of the . . . Si-O-Si . . . atoms is constrained by the ring structure. The "softness" of the bond angles, plus the favorable geometry reducing steric interactions of attached groups, should result in a negligible barrier to free rotation about the Si-O bonds in the linear polymers. Consequently, the low boiling points and temperature coefficients of viscosity may be attributed to free rotation preventing chains from packing sufficiently closely for the short range intermolecular forces to be strongly operative.

The details of the structure analysis of the spiro-silicone will be published in the near future.

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RECEIVED JANUARY 20, 1947

#### HYDROGENATION OF SPIROPENTANE

Sir:

The assignment of the spiro-pentane structure to a  $C_5H_8$  hydrocarbon prepared by Murray and Stevenson<sup>1</sup> was shown to be justified by Raman spectrographic<sup>1</sup> and electron diffraction<sup>2</sup> studies. The same hydrocarbon was prepared at this Laboratory<sup>3</sup> by a modification of the procedure reported by Murray and Stevenson, and an identification of the structure by analysis of hydrogenation products has now been attempted.

The hydrocarbon was found to hydrogenate readily in hydrogen at 2 to 3 atmospheres in the presence of  $PtO_2$  at room temperature. The products of the hydrogenation were separated by fractionation through a 14-mm. glass column, 7-ft. in length, packed with  $1/8$ -inch single-turn glass

(1) Murray and Stevenson, *THIS JOURNAL*, **66**, 812-816 (1944).

(2) Donohue, Humphrey and Schomaker, *ibid.*, **67**, 332-335 (1945).

(3) Slabey *ibid.*, **68**, 1335 (1946).

helices, and identified by their physical constants as indicated in the following table:

Compound	B. p., °C.		$n_D^{20}$		Moles of product
	Lit. <sup>4</sup>	Found	Lit. <sup>4</sup>	Found	
Neopentane	9.45	8-10	1.3472 <sup>a</sup>	1.3483 <sup>a</sup>	0.082
1,1-Dimethylcyclopropane	19.9	18-20	1.3659	1.3677 <sup>b</sup>	.188
Isopentane	27.89	26-28	1.3539	1.3548	.043

<sup>a</sup> 5°. <sup>b</sup> 17.5°.

Infrared spectroscopic examination supported the identification proposed from the physical constants (thanks are due Dr. A. P. Cleaves of this Laboratory).

An examination of the structures of the products indicates that the strained rings of spiro-pentane are cleaved by hydrogen preferentially between the methylene groups. Spiro[2,5]octane has been shown to cleave in an analogous manner to yield 1,1-dimethylcyclohexane.<sup>5</sup>

Ethylcyclopropane and *n*-pentane, which might be expected to be among the hydrogenation products, were not found.

A detailed examination of the hydrogenation products of spiro-pentane by precise fractional distillation and infrared techniques will be reported later.

(4) Doss, "Physical Constants of the Principal Hydrocarbons," 4th ed., The Texas Company, New York, N. Y., 1943.

(5) Boord and Craig, Division of Organic Chemistry, Atlantic City Meeting, American Chemical Society, April, 1946.

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RECEIVED SEPTEMBER 12, 1946

#### PROPERTIES OF *m*-NITROCINNAMYL ALCOHOL

Sir:

Recently it was of some interest in this Laboratory to prepare some *m*-nitrocinnamyl alcohol. The physical properties observed for this preparation were not in accord with those previously reported in the literature, and it seems desirable to report the discrepancy.

Meerwein, *et al.* (*J. prakt. Chem.*, **147**, 211 (1936)) prepared *m*-nitrocinnamyl alcohol by the aluminum ethoxide reduction of *m*-nitrocinnamaldehyde, the product being described as light yellow needles, m. p. 51-51.5°. Effecting the reduction with aluminum isopropoxide, we obtained the material, after several recrystallizations from a 20:80 mixture of ethyl acetate and 68° ligroin, in the form of practically white needles having a pale greenish tint, m. p. 54.5°. In view of the discrepancy in melting point, the *n*-phenyl carbonate was prepared, m. p. 130°. *Anal.* Calcd.: N, 9.40. Found: N, 9.54.

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