[Contribution from the Department of Chemistry, Massachusetts Institute of Technology]

Cyclic Polyolefins. XXXVI. *trans*-Cyclononene and *trans*-Cyclodecene from Cyclononyldimethylamine Oxide and Cyclodecyldimethylamine Oxide

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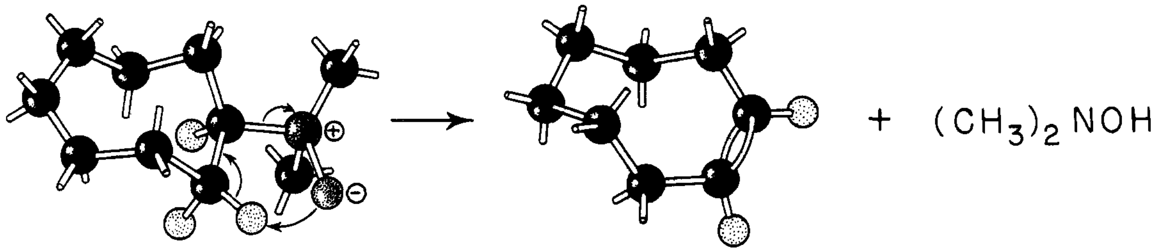
The thermal decomposition of cyclononyldimethylamine oxide has been shown to form *trans*-cyclononene (90%); cyclodecyldimethylamine oxide yields *trans*-cyclodecene (94%). The same *trans* olefins were obtained by the Hofmann exhaustive methylation procedure from the corresponding quaternary bases in yields of 83 and 90%, respectively. These results may be compared with those obtained in the preparation of cyclooöctene by the two methods; the amine oxide-elimination procedure yields *cis*-cyclooöctene, while the Hofmann route forms a mixture of *cis*- and *trans*-cyclooöctene in an approximate ratio of 2:3. With smaller ring sizes, both methods lead to *cis* olefins. The acid-catalyzed isomerization of *trans*-cyclodecene to *cis*-cyclodecene has been shown to form small amounts of *cis*- and *trans*-decalins as by-products, in an intramolecular alkylation or bridging reaction explained by the spatial proximity of the 1- and 6-carbon atoms of cyclodecene.

Thermal decomposition of several N,N-dimethylalkylamine oxides has been shown to proceed with elimination of a β-hydrogen atom and the amine oxide group, forming an olefin and N,N-dimethylhydroxylamine.[1,2] The formation of pure *cis*-cyclooöctene in the thermal decomposition of cyclooctyldimethylamine oxide, as contrasted with the formation of a mixture of *cis*- and *trans*-cyclooöctene in the decomposition of cyclooöctyltrimethylammonium hydroxide,[2] was interpreted as evidence for an intramolecular cyclic mechanism, in which the amine oxide group and a *cis*-β-hydrogen are eliminated.



I

● CARBON ● NITROGEN ● OXYGEN ○ HYDROGEN

It is evident that if the alicyclic ring is small (eight or fewer carbon atoms), only a *cis*-β-hydrogen can assume the favored planar transition state[3] for the intramolecular elimination reaction. The thermal decompositions of cyclononyltrimethylammonium hydroxide[4] and cyclodecyltrimethylammonium hydroxide[5] have been shown to form *trans*-cyclononene and *trans*-cyclodecene, respectively. We now have studied the thermal decomposition of cyclononyldimethylamine oxide and cyclodecyldimethylamine oxide, to obtain further information concerning the effect of ring size upon the stereochemistry of this elimination reaction.

Cyclononyldimethylamine and cyclodecyldimethylamine were prepared by methods similar to those described previously for the preparation of these and other alicyclic amines. Azeloin and suberoin were prepared by the acyloin condensation from dimethyl azelate and dimethyl sebacate, respectively.[4-7] The cyclic acyloins were reduced to cyclononanone and cyclodecanone with zinc and hydrochloric acid,[6,8] and the ketones were converted to their oximes. Reduction of the oximes with sodium and ethanol[9] yielded cyclononylamine and cyclodecylamine, and methylation of the primary amines with formic acid and formaldehyde formed the N,N-dimethylalicyclic amines.

Cyclononyldimethylamine oxide was prepared from cyclononyldimethylamine and hydrogen peroxide in aqueous methanol. The oxidation was shown to be essentially quantitative by preparation of the amine oxide picrate from an aliquot in 96% yield. The amine oxide decomposed readily when heated at 12 mm. and 80–95°, forming N,N-dimethylhydroxylamine (isolated as the hydrochloride in 96% yield) and *trans*-cyclononene in 80–90% yield. A small amount of a by-product of unknown structure was separated as a lower boiling fraction, but there was no positive evidence for the presence of *cis*-cyclononene in the product. Thermal decomposition of cyclononyltrimethylammonium hydroxide yielded 83% of *trans*-cyclononene, in agreement with previous results.[4]

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[1] A. C. Cope, T. T. Foster and P. H. Towle, This Journal, **71**, 3929 (1949).

[2] A. C. Cope, R. A. Pike and C. F. Spencer, *ibid.*, **75**, 3212 (1953).

[3] D. H. R. Barton and E. Miller, *ibid.*, **72**, 1066 (1950); D. H. R. Barton and W. J. Rosenfelder, *J. Chem. Soc.*, 1048 (1951).

[4] A. T. Blomquist, L. H. Liu and J. C. Bohrer, This Journal, **74**, 3643 (1952).

[5] A. T. Blomquist, R. E. Burge, Jr., and A. C. Sucsy, *ibid.*, **74**, 3636 (1952).

[6] V. Prelog, L. Frenkiel, M. Kobelt and P. Barman, *Helv. Chim. Acta*, **30**, 1741 (1947).

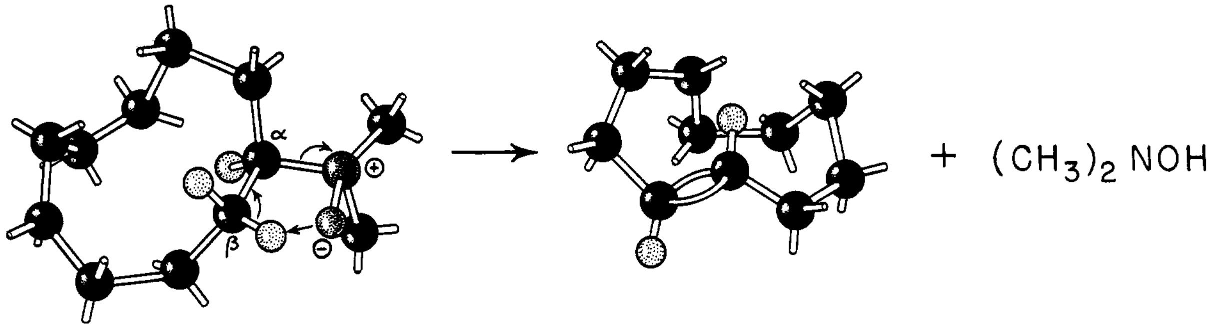
[7] M. Stoll and J. Hulstkamp, *ibid.*, **30**, 1815 (1947); M. Stoll and A. Rouve, *ibid.*, **30**, 1822 (1947).

[8] H. C. Brown and M. Borkowski, This Journal, **74**, 1894 (1952).

[9] V. Prelog, M. F. El-Neweihy and O. Häfliger, *Helv. Chim. Acta*, **33**, 365 (1950).

Cyclodecyldimethylamine was oxidized quantitatively to cyclodecyldimethylamine oxide with hydrogen peroxide in aqueous methanol, as shown by isolation of the amine oxide picrate from an aliquot in 96% yield. Thermal decomposition of the amine oxide at 7 mm. and 75–100° yielded 91% of N,N-dimethylhydroxylamine, isolated as the hydrochloride, and 90–94% of pure *trans*-cyclodecene. The infrared spectrum of *trans*-cyclodecene from the amine oxide decomposition was identical with the spectrum of an authentic sample prepared in 87–90% yield by thermal decomposition of cyclodecyltrimethylammonium hydroxide.[5]

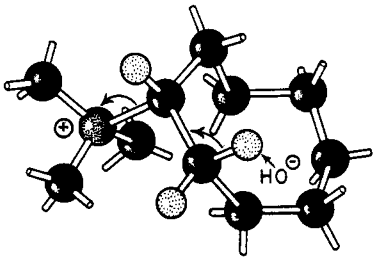
Since *trans* cyclic olefins were obtained from both amine oxides, it is evident that nine- and ten-membered carbocyclic rings can assume conformations in the transition states (represented in formula II) in which a *trans*-β-hydrogen is in the same plane as the amine oxide group.



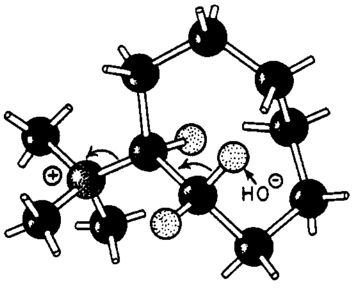
II

If the assumption that the elimination of dimethylhydroxylamine occurs by an intramolecular process is correct, it follows that the transition state with lowest energy is one in which the bonds from the α- and β-carbon atoms to the adjacent carbons are in the *trans* configuration, as shown in formula II, when the ring is nine- or ten-membered. The fact that both the Hofmann exhaustive methylation and amine oxide-elimination reactions yield *cis*-cyclic olefins with rings containing seven or fewer carbon atoms can be explained by the strain that would be produced if the bonds from the α- and β-carbon atoms to adjacent ring carbons assumed the *trans* configuration; only the *cis* olefins are known with seven-membered and smaller rings. With cyclooctyltrimethylammonium hydroxide, the two conformations of the transition state (A and B) which place a β-hydrogen in the same plane as the nitrogen evidently have similar energies, and the elimination reaction occurs both through A leading to *cis*-cyclooöctene and through B forming *trans*-cyclooöctene. Evidently the amine oxide conformation in the transition state (formula I) leading to *cis*-cyclooöctene is a lower energy form than a conformation corresponding to II which would form *trans*-cyclooöctene, since the only product obtained from the amine oxide is the *cis* olefin.

trans-Cyclononene and *trans*-cyclodecene were rearranged to the *cis*-isomers by heating with β-naphthalenesulfonic acid in the presence of hydroquinone,[4,5,10] in order to obtain samples of the *cis* olefins for comparison. The principal product obtained from *trans*-cyclononene was the *cis* isomer, as previously reported.[4] The product obtained from *trans*-cyclodecene contained, in addition to *cis*-cyclodecene, a hydrocarbon with a slightly lower boiling point which had infrared absorption bands at 7.3, 7.65, 8.8, 10.8 and 11.7 μ that are not present in the spectra of either *cis*- or *trans*-cyclodecene. This hydrocarbon was isolated by chromatography on silica gel, from which it was eluted by pentane more rapidly than cyclodecene, followed by distillation. It was identified by its infrared spectrum as a mixture of *cis*- and *trans*-decalins. The intramolecular alkylation or bridging reaction forming decalin represented in the following formulas is believed to be facilitated by the spatial proximity of the 1- and 6-carbon atoms of cyclodecene. The mixture of *cis*- and *trans*-decalins obtained from *trans*-cyclodecene did not appear to contain cyclopentanocycloheptane (bicyclo[5.3.0]decane), which could be formed by a similar 1,5-bridging process, for the infrared spectrum of the decalins showed no appreciable absorption at 10.5 μ, at which wave length there is an absorption band of medium intensity in the spec-



A



B

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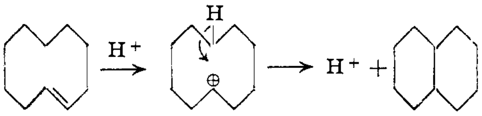
(10) K. Ziegler and H. Wilms, *Ann.*, **567**, 1 (1950).

trum of *cis*-bicyclo[5.3.0]decane.[11] The cyclodecene obtained by acid-catalyzed isomerization, after separation of the decalins, had the infrared spectrum of *cis*-cyclodecene, and showed no evidence that any of the *trans* isomer was present.



Silver nitrate complexes were prepared from both the *cis* and *trans* isomers of cyclonene and cyclodecene. These complexes contained the hydrocarbons and silver nitrate in a molecular ratio of 2:1, and were unstable, dissociating readily into the cyclic olefins and silver nitrate.

Experimental[12]

Cyclononyldimethylamine.—Dimethyl azelate was converted to azeloin in 38% yield by methods previously described.[4,6,7] The acyloin was reduced with zinc and hydrochloric acid[6,8] in 69% yield to cyclononanone, b.p. 90–96° (10–13 mm.), $n^{25}\text{D}$ 1.4760. A sample of cyclononanone that was regenerated in 89% yield from its semicarbazone (recrystallized from methanol, m.p. 179–180.5°) by steam distillation in the presence of oxalic acid was unchanged in physical constants including the infrared spectrum. Cyclononanone oxime was prepared in 78% yield, m.p. 73–76°, and after two recrystallizations from methanol melted at 78.5–79.5° (lit.[13] m.p. 79°). Reduction of the oxime with sodium and ethanol[9] yielded 82% of cyclononylamine, b.p. 85–87° (10 mm.), $n^{25}\text{D}$ 1.4837, which was characterized as the picrate, m.p. 222.5–225° dec. (lit.[9] m.p. 219–221° dec.). Methylation of 5.74 g. of cyclononylamine with formic acid and formaldehyde[14] yielded 5.29 g. (77%) of cyclononyldimethylamine, b.p. 87–88° (4.3 mm.), $n^{25}\text{D}$ 1.4763.

Anal. Calcd. for $C_{11}H_{23}N$: C, 78.02; H, 13.69; N, 8.27. Found: C, 77.69; H, 13.37; N, 8.47.

Cyclononyldimethylamine picrate was prepared from the amine and picric acid in ethanol and recrystallized from 95% ethanol; m.p. 180–182° dec.

Anal. Calcd. for $C_{17}H_{26}N_4O_7$: C, 51.24; H, 6.58; N, 14.07. Found: C, 51.49; H, 6.53; N, 14.13.

Cyclononyldimethylamine Oxide.—A solution of 12.6 g. of cyclononyldimethylamine in 30 ml. of methanol was cooled to 0° and 25.5 g. of 30% hydrogen peroxide was added in small portions with swirling in a period of 10 minutes. The mixture was allowed to come to room temperature and stand for 25 hours, at which time it gave a negative test for the amine with phenolphthalein.[2] The solution was stirred overnight with approximately 0.05 g. of platinum black,[15] added as an aqueous suspension, and then gave a negative test for hydrogen peroxide with lead sulfide paper.[2] An aliquot of the solution corresponding to 0.63 g. of the original amine was added to a solution of 1.0 g. of picric acid in 60 ml. of warm water, and the picrate that separated was collected on a filter and washed with cold water. The yield of cyclononyldimethylamine oxide picrate was 1.47 g. (96%), m.p. 152–156°. An analytical sample was recrystallized from absolute ethanol to a constant melting point of 153–155.4°.

Anal. Calcd. for $C_{17}H_{26}N_4O_8$: C, 49.37; H, 6.33; N, 13.52. Found: C, 49.54; H, 6.33; N, 13.77.

trans-**Cyclononene.** (a) **From Cyclononyldimethylamine Oxide.**—The remainder of the solution of the amine oxide described above was concentrated under reduced pressure, and the viscous, oily residue was decomposed by heating at 12 mm. in a 200-ml. round-bottomed flask attached to a short Vigreux column connected to two traps that served as receivers, the first cooled with Dry Ice and the second with liquid nitrogen. Decomposition of the amine oxide occurred during a period of 30 minutes at a bath temperature of 80–95°. The distillate was acidified with dilute hydrochloric acid, and the organic layer was removed with a capillary pipet and dried by cooling with Dry Ice and decanting from the ice that formed. Distillation through a semimicro column separated 1.1 g. of a forerun, most of which had b.p. 72–73° (31 mm.), $n^{25}\text{D}$ 1.4755, and contained an impurity of unknown structure with a strong infrared band at 13.3 μ and weaker bands at 11.15 and 14.25 μ. After the forerun 7.02 g. (80%) of *trans*-cyclononene was collected, b.p. 73–75° (31 mm.), $n^{25}\text{D}$ 1.4770–1.4777. In another preparation of *trans*-cyclononene by this procedure the yield was 90%.

The structure of *trans*-cyclononene prepared from the amine oxide was verified by comparison of its infrared spectrum with the published spectrum[4] and with the spectrum of a sample described below, prepared by Hofmann exhaustive methylation. A sample was reduced quantitatively in acetic acid in the presence of platinum with the absorption of 103.7% of one molar equivalent of hydrogen to cyclononane, b.p. 80–81° (41 mm.), $n^{25}\text{D}$ 1.4643, which also had an infrared spectrum identical with the published spectrum.[4]

The N,N-dimethylhydroxylamine hydrochloride present in the aqueous layer from which the *trans*-cyclononene had been separated was isolated by a procedure previously described (ref. 1, p. 3932) in a yield of 6.60 g. (96%), m.p. 107–109°.

(b) **From Cyclononyltrimethylammonium Hydroxide.**—Cyclononyltrimethylammonium iodide was prepared in a yield of 12.55 g. (95%) by adding 14.9 g. of methyl iodide to a solution of 7.4 g. of cyclononyldimethylamine in 150 ml. of dry ether and allowing the mixture to stand for 2 hours; m.p. 272–273° (dec., uncor.), (lit.[4] m.p. 273–273.5° dec., uncor.). A solution of 14.2 g. of the methiodide in 125 ml. of water was stirred overnight with the moist, neutral silver oxide freshly prepared from 15.0 g. of silver nitrate. The mixture was filtered, the solids were washed with water, and the filtrate was concentrated under reduced pressure. The residual quaternary base was decomposed by heating at 8 mm. with a bath temperature of 90–125° in a period of 30 minutes by the procedure described above for the amine oxide decomposition. The yield of *trans*-cyclononene was 4.70 g. (83%), b.p. 74.5–76.5° (30–33 mm.), $n^{25}\text{D}$ 1.4773, and the infrared spectrum of the product was identical with the spectrum previously reported.[4]

Cyclodecyldimethylamine.—The acyloin condensation of dimethyl sebacate yielded sebacoin (42%), which was reduced with zinc and hydrochloric acid in 82% yield to cyclodecanone, b.p. 102–104° (9 mm.), $n^{25}\text{D}$ 1.4798–1.4802. A sample of the ketone regenerated from its semicarbazone, m.p. 202–203° (lit.[5] m.p. 203.5–205.5°), by steam distillation in the presence of oxalic acid had the same infrared spectrum within experimental error as the ketone before such purification, and the spectra were identical with the published spectrum.[5] Cyclodecanone oxime was prepared in 80–90% yield and after recrystallization from aqueous methanol melted at 79.5–81° (lit.[13] m.p. 80°). Reduction of the oxime with sodium and ethanol yielded cyclodecylamine (75%), b.p. after redistillation 84–86° (4 mm.), $n^{25}\text{D}$ 1.4867, m.p. of the picrate 241–243° dec. (lit.[9] m.p. 239° dec.). Methylation of 16.95 g. of cyclodecylamine with formic acid and formaldehyde[14] yielded 17.12 g. (86%) of cyclodecyldimethylamine, which after redistillation had b.p. 101–103° (3–4 mm.), $n^{25}\text{D}$ 1.4800.

Anal. Calcd. for $C_{12}H_{25}N$: C, 78.62; H, 13.75; N, 7.64. Found: C, 78.60; H, 13.70; N, 7.78.

Cyclodecyldimethylamine picrate was prepared from the amine and picric acid in ethanol and recrystallized from absolute ethanol to a constant melting point of 145–146.5°.

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[11] E. Kováts, A. Fürst and H. H. Gunthard, *Helv. Chim. Acta*, **37**, 534 (1954). The infrared spectrum of a sample of bicyclo[5.3.0]-decane of unknown stereochemistry (*cis*, *trans* or a mixture) prepared in this Laboratory by Robert J. Cotter by Wolff–Kishner reduction of bicyclo[5.3.0]-2-decanone also contained a band of medium intensity at 10.5 μ.

[12] Melting points are corrected and boiling points are uncorrected. Infrared spectra were determined with a Baird Double Beam Infrared Recording Spectrometer, Model B, fitted with a sodium chloride prism. We are indebted to Dr. S. M. Nagy and his associates for analyses.

[13] L. Ruzicka, M. Kobelt, O. Häfliger and V. Prelog, *Helv. Chim. Acta*, **32**, 544 (1949).

[14] By a procedure based on one described by R. N. Icke and B. B. Wisegarver in *Org. Syntheses*, **25**, 89 (1945).

[15] R. Feulgen, *Ber.*, **54**, 360 (1921).

Anal. Calcd. for $C_{18}H_{28}N_4O_7$: C, 52.44; H, 6.83; N, 13.59. Found: C, 52.59; H, 7.01; N, 13.48.

Cyclodecyldimethylamine Oxide.—Cyclodecyldimethylamine (8.0 g.) was oxidized with hydrogen peroxide by the procedure described above for preparation of cyclononyldimethylamine oxide. An aliquot of the solution corresponding to 0.64 g. of the amine was treated with picric acid in water and yielded 1.442 g. (96%) of cyclodecyldimethylamine oxide picrate, m.p. 129.5–130.5°. An analytical sample was recrystallized from absolute ethanol, m.p. 131–132°.

Anal. Calcd. for $C_{18}H_{28}N_4O_8$: C, 50.48; H, 6.59; N, 13.08. Found: C, 50.57; H, 6.66; N, 13.11.

***trans*-Cyclodecene. (a) From Cyclodecyldimethylamine Oxide.**—The remainder of the solution of the amine oxide described above (corresponding to 7.36 g. of the amine) was concentrated under reduced pressure and the residue was decomposed by heating in a nitrogen atmosphere at 7 mm. with a bath temperature of 75–100° in the manner described for the next lower homolog. The yield of *trans*-cyclodecene was 5.05 g. (90%). b. p. 70.5–71.5° (11 mm.). $n^{25}D$ 1.4795. The *trans*-cyclodecene had an infrared spectrum identical with the spectrum of the sample prepared by the Hofmann exhaustive methylation described below and with the published spectrum.[5]

N,N-Dimethylhydroxylamine hydrochloride was isolated from the aqueous hydrochloric acid solution from which the *trans*-cyclodecene was separated in a yield of 3.57 g. (91%), m.p. 103–107° (sealed capillary).

(b) From Cyclodecyltrimethylammonium Hydroxide.—Cyclodecyltrimethylammonium iodide was prepared in a yield of 16.26 g. (99%) from 9.22 g. of cyclodecyldimethylamine and 15.6 g. of methyl iodide in 200 ml. of dry ether, m.p. 266.5–267° dec. (lit.[5] m.p. 265–265.5° dec.). The quaternary base obtained from 15.4 g. of the methiodide and silver oxide was concentrated and decomposed by heating in a nitrogen atmosphere at 7 mm. with a bath temperature of 100–110°, according to the procedure described above for preparing *trans*-cyclononene. The yield of *trans*-cyclodecene was 5.89 g. (90%), b.p. 71° (11 mm.). $n^{25}D$ 1.4789–1.4793. The yield in another similar preparation was 87%. The infrared spectrum of *trans*-cyclodecene prepared in this way was identical with the published spectrum.[5]

***cis*-Cyclononene and *cis*-Cyclodecene.**—*trans*-Cyclononene (2.94 g.), β-naphthalenesulfonic acid (50 mg.) and hydroquinone (50 mg.) were heated in a sealed tube under nitrogen at 150° for 2 hours. Distillation of the product separated a low boiling fraction from 1.93 g. (66%) of *cis*-cyclononene, b.p. 71–72° (31 mm.), $n^{25}D$ 1.4763–1.4780. The infrared spectrum of the *cis*-cyclononene was essentially identical with the published spectrum.[4]

trans-Cyclodecene (5.34 g.) was heated under nitrogen for 2 hours at 170–180° with 90 mg. of β-naphthalenesulfonic acid and 90 mg. of hydroquinone. Distillation of the product separated 0.40 g. of a forerun, b.p. 79–83° (20 mm.), $n^{25}D$ 1.4730–1.4752, from 3.81 g. of crude *cis*-cyclodecene, b.p. 83–85° (20 mm.), $n^{25}D$ 1.4776–1.4818. The infrared spectra of the first fractions contained bands at 7.3, 7.65, 8.8, 10.8 and 11.7μ which are not present in the spectra of *cis*- or *trans*-cyclodecene. These bands can be accounted for by the presence of *cis*- and *trans*-decalins, which do absorb at these wave lengths.[16] The infrared spectra of the (higher boiling) cyclodecene fractions showed no evidence for the presence of *trans*-cyclodecene.

In order to obtain more conclusive evidence for the presence of decalin in the product obtained by heating *trans*-cyclodecene with β-naphthalenesulfonic acid, the experiment described in the preceding paragraph was repeated with a 2.0-g. sample of *trans*-cyclodecene, and yielded 1.66 g. of crude *cis*-cyclodecene, b.p. 78–84° (19 mm.), $n^{25}D$ 1.4810. The infrared spectrum of this product contained very weak bands at 7.65 and 10.8 μ and otherwise was essentially identical to the spectrum of *cis*-cyclodecene.[5] A 1.40-g. sample of the product was chromatographed on 50 g. of silica gel and eluted with pentane. Examination of the infrared spectra of the eluted fractions in the 9.5–13.2 μ region showed that the first 45 ml. of eluate contained little if any cyclodecene. The material in these fractions was rechromatographed on 30 g. of silica gel, eluted with pentane, and distilled. This procedure yielded 0.135 g. of a mixture of *cis*- and *trans*-decalins, $n^{25}D$ 1.4728. The infrared spectrum of this mixture was identical (24 comparisons of absorption maxima) with the spectrum of an authentic mixture of *cis*- and *trans*-decalins.

Silver Nitrate Adducts. (a) *cis*-Cyclononene.—Aqueous silver nitrate (7.0 g. of a 30% solution by weight) was added to a mixture of 0.50 g. of *cis*-cyclononene and 2.0 g. of methanol at room temperature. Crystals began to separate almost immediately, and after cooling they were collected on a filter. In this and other preparations of silver nitrate adducts the product was dried in a small desiccator over Drierite and paraffin chips in an atmosphere saturated with the vapor of the hydrocarbon[17] (in this case *cis*-cyclononene). The yield of the adduct was 0.55 g., m.p. 64–70°. An analytical sample that was recrystallized twice from methanol melted at 67–69° with softening at 66°.

Anal. Calcd. for $(C_9H_{16})_2 \cdot AgNO_3$: C, 51.69; H, 7.71. Found: C, 50.79; H, 7.84.

(b) *trans*-Cyclononene.—A mixture of 10 ml. of 30% aqueous silver nitrate and 0.50 g. of *trans*-cyclononene was shaken vigorously and then cooled to 0°. The product was collected on a filter, washed rapidly with a small volume of cold water, and dried by the procedure described above for 15 hours at room temperature. The adduct (0.43 g.) melted at 90–94°. Attempted recrystallizations from methanol and mixed solvents were unsuccessful.

Anal. Calcd. for $(C_9H_{16})_2 \cdot AgNO_3$: C, 51.69; H, 7.71. Found: C, 52.01; H, 7.49.

***cis*-Cyclodecene.**—Aqueous silver nitrate (5 ml. of a 30% solution) was added slowly with swirling to a mixture of 0.50 g. of *cis*-cyclodecene and 5 ml. of methanol. After cooling at 0° for 1 hour, the adduct was collected on a filter and dried by the procedure described above. The product (0.54 g.) softened at 85° and decomposed slowly on heating to 160°. An analytical sample that was recrystallized from methanol softened at 97–98° and decomposed with gas evolution at 167–187°.

Anal. Calcd. for $(C_{10}H_{18})_2 \cdot AgNO_3$: C, 53.81; H, 8.13; Ag, 24.17. Found: C, 53.60; H, 8.04; Ag, 24.54.

***trans*-Cyclodecene.**—Aqueous silver nitrate (14 g. of a 25% solution) was added slowly with swirling to a mixture of 0.51 g. of *trans*-cyclodecene and 6 g. of methanol. The crystals that separated rapidly were collected on a filter after cooling. The yield of the adduct was 0.60 g., m.p. 75–80°. An analytical sample that was recrystallized from methanol and dried by the procedure described above melted at 81–83.5°.

Anal. Calcd. for $(C_{10}H_{18})_2 \cdot AgNO_3$: C, 53.81; H, 8.13. Found: C, 53.58; H, 8.05.

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[16] J. Seidman, *Anal. Chem.*, **23**, 559 (1951).

[17] See A. C. Cope and F. A. Hochstein, THIS JOURNAL, **72**, 2519 (1950).