Cyclohexane-ethanol.—This compound, 0.45 mole, with 3 g. of catalyst at 250° absorbed no hydrogen, but at 300° showed 24.2% hydrogenolysis based on pressure drop and 28% based on refractive index of the product, n^{25}D −1.4540, *vs.* that of the starting material, n^{25}D −1.4630, and that of ethylcyclohexane, n^{25}D −1.4308.

Phenethyl Alcohol.—At 250° with 4 g. of catalyst, phenethyl alcohol, 0.66 mole, absorbed 27.6% of the hydrogen required for four double bonds. The product was separated from the several ml. of water, dried over "Drierite" and fractionated through a 50-plate column to give 95% ethylbenzene, 3% ethylcyclohexane and 2% cyclohexaneëthanol.

Acknowledgment.—We wish to express our appreciation to Dr. Robert B. Carlin of the Department of Chemistry of Carnegie Institute of Technology and to Dr. H. H. Lowry for their suggestions, to Mrs. Matilda Fine and Mr. Joseph B. Simsic for assistance in the analytical work and Mr. Daniel Muth for help in several of the autoclave experiments. The ultimate analysis was performed by the Huffman Microanalytical Laboratories, Denver, Colorado, and the infrared spectra were determined on a Baird Recording Spectrophotometer at the Mellon Institute.

PITTSBURGH 13, PENNA. RECEIVED FEBRUARY 1, 1951

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, MASSACHUSETTS INSTITUTE OF TECHNOLOGY]

Cyclic Polyolefins. XVIII. Rearrangement of Cycloöctatetraene Oxide to 1,3,5-Cycloöctatrien-7-one[1]

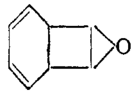
BY ARTHUR C. COPE AND BURRIS D. TIFFANY[2]

Cycloöctatetraene oxide (I) has been prepared by the oxidation of cycloöctatetraene with peracetic acid, a more convenient preparation than the oxidation with perbenzoic acid previously employed. The reaction of I with phenyllithium formed as one product 8% of a yellow liquid with the empirical formula C_8H_8O. This compound is formed by a base-catalyzed rearrangement of cycloöctatetraene oxide, and was obtained in a yield of 39% with mesityllithium as the base and 71% or more with lithium diethylamide. The isomer was proved to be 1,3,5-cycloöctatriene-7-one (II) by the preparation of ketonic derivatives and by quantitative hydrogenation to cycloöctanone and cycloöctanol.

This paper reports the base-catalyzed isomerization of cycloöctatetraene oxide (I) to 1,3,5-cyclooctatrien-7-one (II), which was discovered during an investigation of I as an intermediate in the preparation of substituted cycloöctatetraenes from the parent hydrocarbon.

I II

Cycloöctatraene oxide (I) originally was prepared from cycloöctatetraene and perbenzoic acid, by the method described by Reppe, Schlichting, Klager and Toepel.[3] It was found that I could be prepared more conveniently and in slightly better yield (55%) by oxidation with the commercially available 40% solution of peracetic acid in acetic acid by careful control of the temperature of the exothermic reaction. Friess and Boekelheide[4] have interpreted the ultraviolet absorption spectrum of the epoxide I, its behavior on hydrogenation, and the fact that it forms a maleic anhydride adduct containing one double bond as indicating that the compound has the fused-ring structure Ia. The evidence concerning the structure of I appears to us to be inconclusive. Bridging as well as addition could occur in the reaction of I with maleic anhydride, causing the disappearance of two double bonds, as is the case in the reaction of cycloöctatetraene with maleic anhydride.[3] A choice between structures I and Ia cannot be made with certainty at present on the basis of the ultraviolet absorption spectrum of the compound, for the spectra of models with structures related to I and Ia are too similar; 1,3,5-cycloöctatriene has $\lambda_{\max.}$ 265 mμ (ϵ 3600)[5] and 1,3-cyclohexadiene has $\lambda_{\max.}$ 258 mμ (ϵ 3980)[6] (both in cyclohexane). The fact that cyclooctanol is formed by the hydrogenation of cyclooctatetraene oxide[3,4] provides evidence supporting structure I rather than Ia, for cycloöctatetraene dichloride (which has a bridged ring structure analogous to Ia) yields bicyclo[4.2.0]octane rather than cycloöctane on hydrogenation.[3]



Ia

The reaction of cycloöctatetraene oxide with phenyllithium was investigated in order to determine whether cleavage of the epoxide ring would result in the formation of a phenylcycloöctatrienol that could be dehydrated to obtain phenylcyclooctatetraene. The reaction took an entirely different course. The principal product was an unsaturated ketone $C_{14}H_{14}O$, which was characterized as the 2,4-dinitrophenylhydrazone but not investigated further. Of greater interest was a small amount (8%) of a yellow liquid product, C_8H_8O, formed by the isomerization of cycloöctatetraene oxide. This liquid reduced permanganate, gave a negative fuchsin aldehyde test, and formed a crystalline 2,4-dinitrophenylhydrazone, semicarbazone and oxime. Evidence establishing the structure of this product as 1,3,5-cycloöctatrien-7-one (II) was obtained by its quantitative hydrogenation, which in the presence of palladium-on-calcium carbonate in methanol resulted in the absorption of 95% of

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[1] Supported in part by the Office of Naval Research under Contract N5ori-07822, Project Designation NR-055-96.

[2] du Pont Postdoctorate Fellow, 1949–1950.

[3] W. Reppe, O. Schlichting, K. Klager and T. Toepel, *Ann.*, **560**, 1 (1948).

[4] S. L. Friess and V. Boekelheide, THIS JOURNAL, **71**, 4145 (1949).

[5] A. C. Cope and F. A. Hochstein, *ibid.*, **72**, 2515 (1950).

[6] We are indebted to Dr. Floyd L. Ramp for these data.

three molar equivalents of hydrogen and formation of cycloöctanone (identified as the semicarbazone). In the presence of platinum in acetic acid II absorbed 101% of four molar equivalents of hydrogen and formed cycloöctanol (characterized as the phenylurethan). The ultraviolet absorption spectrum of II (Fig. 1) and the infrared spectrum (Fig. 2) are consistent with its structure. The infrared spectrum shows strong absorption at 6.0 μ, consistent with the presence of an α,β-unsaturated carbonyl group, and weak absorption at 3.05 μ in the region of absorption characteristic of compounds containing hydrogen-bonded hydroxyl groups, suggesting that a small amount of the enol form may be present in the liquid at room temperature.

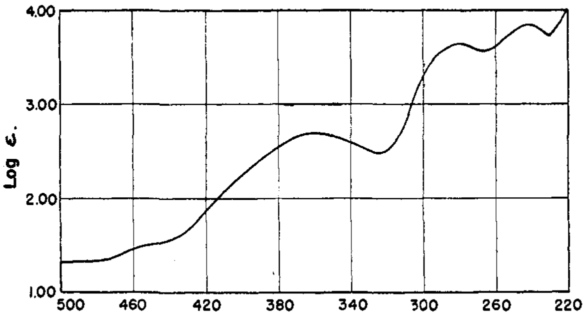


Fig. 1.—Ultraviolet absorption spectrum of 1,3,5-cycloöctatrien-7-one (II) in cyclohexane.

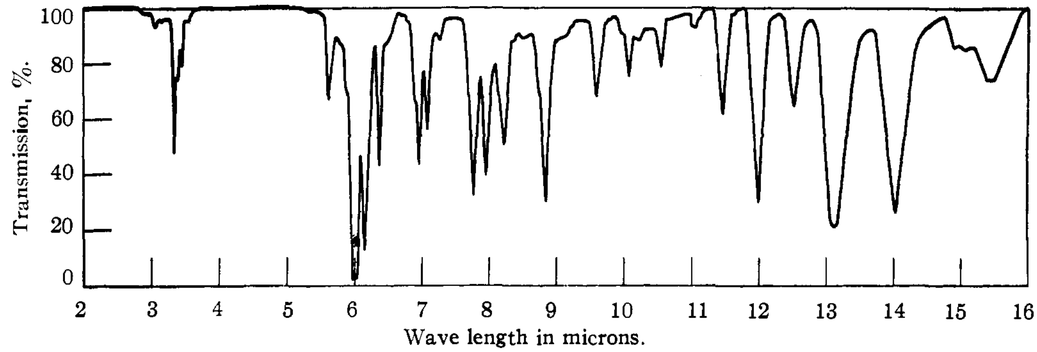
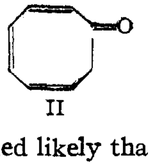
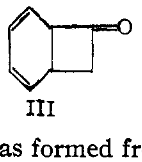


Fig. 2.—Infrared absorption spectrum of 1,3,5-cycloöctatrien-7-one (II) as a solution (100 mg./ml.) in carbon tetrachloride in the region 2–7.1 μ and in carbon disulfide in the region 7.1–16 μ.

The spectrum of II also contains a band at 5.6 μ (much weaker than the 6.0 μ band), in the region where the carbonyl group of cyclobutanone causes absorption.[7] This band cannot be interpreted with certainty, but an explanation consistent with the facts now known would be that II contains a small amount (of the order of 5%) of a fused ring isomer such as III, present either as an impurity or in equilibrium with II.[8] These possibilities are being investigated at present.

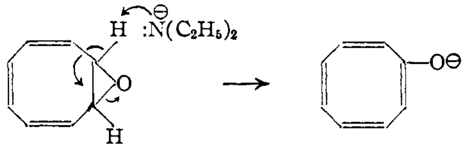


II



III

It seemed likely that II was formed from I by a base-catalyzed removal of a proton from I, forming an anion which rearranged to another anion from which II was formed by acidification and ketonization of the enol (hydroxycycloöctatetraene).



Accordingly a base more suitable for effecting the isomerization was sought, which would not reduce the yield of II by competing reactions of addition either to the epoxide ring of I or the carbonyl group of II, forming carbon–carbon bonds.

The sterically hindered organolithium compound mesityllithium gave an improved yield (39%) of II, presumably because the competing side reactions of addition were slower than corresponding addition reactions of phenyllithium. The weaker base lithium diethylamide proved to be a still more satisfactory reagent for causing the isomerization of I to II, and by its use II was obtained in a crude yield of 95%. The ketone II was purified by conversion to the stable, crystalline semicarbazone. The ketone could be regenerated from its semicarbazone by acid hydrolysis and steam distillation in the presence of aqueous sulfuric acid or oxalic acid, but an exchange reaction with pyruvic acid afforded a better method for the regeneration of II (in 88% yield). The over-all yield from I to the crystalline semicarbazone of II was 71%. The ketone II was moderately stable if it was kept under nitrogen (to prevent a very rapid reaction with atmospheric oxygen) at a low temperature, but it was convenient to store the semicarbazone and regenerate samples of the ketone as they were needed.

In a test of the thermal stability of the ketone II it was observed that decomposition occurred when a sample was heated at 180 to 215° in a nitrogen atmosphere, resulting in partial resinification and the formation of benzene. In the formation of benzene from II the elements of ketene are eliminated, but a test with aniline showed that no appreciable amount of ketene was evolved as a gas during the reaction.

Investigation of the reactions of the epoxide I and the ketone II is being continued.

[7] J. D. Roberts and C. W. Sauer, This Journal, 71, 3925 (1949).

[8] We are indebted to Dr. R. C. Lord for discussions of the infrared spectra.

Experimental[9]

Cycloöctatetraene Oxide (I).—The epoxide I was prepared from cycloöctatetraene and perbenzoic acid[3] or more conveniently by the following procedure from commercial peracetic acid. Cycloöctatetraene[10] (208 g., 2.00 moles) was placed in a 3-l. three-necked flask equipped with a mechanical stirrer, a dropping funnel and a thermometer which extended into the liquid. The 1% of sulfuric acid present in commercial 40% peracetic acid[11] (containing 45 g. of peracetic acid per 100 ml. according to titration) was neutralized by addition of 5 g. of anhydrous sodium acetate to 100-ml. quantities immediately before they were used. The stirrer was started and 10 ml. of the peracetic acid solution was added. When the temperature of the reaction mixture reached 29° the flask was cooled with a bath containing water at 15°, and the addition of peracetic acid was continued at a nearly constant rate of 10 ml. per minute while the temperature of the reaction mixture was kept in the range of 29–31° by raising or lowering the cooling bath. Constant attention was required to control the temperature of the exothermic reaction. After 400 ml. of the peracetic acid solution (containing 180 g. or 2.4 moles of peracetic acid) had been added during 45 minutes stirring was continued at 29–31° and 0.2-ml. aliquots of the reaction mixture were analyzed periodically. The aliquots were diluted with 100 ml. of water, treated with 10 ml. of 10% potassium iodide solution, allowed to stand for 20 minutes, and the iodine was titrated with 0.1 *N* sodium thiosulfate solution. One and one-quarter hours after the addition was completed, when approximately 2.3 moles of peracetic acid had been consumed, the reaction mixture was diluted with 2 l. of cold water. The yellow oil which separated was extracted with three 500-ml. portions of pentane and the combined extracts were washed with two 250-ml. portions of water and five 100-ml. portions of 10% sodium hydroxide solution. The first alkaline extract was dark orange and the last pale orange in color. The pentane solution then was washed with three 250-ml. portions of water, and dried over sodium sulfate at 5°. The dried solution was concentrated by distillation of the pentane through a 20-cm. Vigreux column by heating on a steam-bath, and the concentrate was distilled through a simple still head under reduced pressure. Crude I was obtained as a very pale yellow liquid in a yield of 75 g., b.p. 74–75° (12 mm.), $n^{25}\mathrm{D}$ 1.5383. Fractionation of the fore-run through a 14 × 1.4 -cm. column packed with glass helices yielded 53 g. (25%) of recovered cycloöctatetraene, b.p. 72–75° (90 mm.), and 23 g. of I, b.p. 75–76° (12 mm.), $n^{25}\mathrm{D}$ 1.5383, making the total amount of I 98 g. (55% yield or 41% conversion).

All samples of I prepared by this procedure or by oxidation with perbenzoic acid[3] showed weak absorption at 5.81 μ in the infrared spectrum, indicating the presence of a small amount of a carbonyl compound as an impurity. This impurity could be partially removed by selective adsorption on alumina, but the procedure was wasteful and accordingly I was purified only by distillation. Its ultraviolet absorption spectrum in 95% ethanol, determined with a Beckman model DU quartz spectrophotometer, showed a maximum at 240 mμ ($\log \epsilon$ 3.57) (lit.[4] $\lambda_{\max}$ 241 mμ, $\log \epsilon$ 3.60).

The residue from the first distillation of I was extracted with ether, the extract was concentrated, and the residue was sublimed at 2 mm. with a heating block temperature of 60–70°. The white hexagonal plates which sublimed were purified by resublimation and recrystallization from the minimum amount of hot benzene required for solution, diluted with an equal volume of methylcyclohexane. The white, crystalline solid, which was not identified, weighed 20.7 mg. and melted at 166.6–168.2°.

Anal. Calcd. for $C_8H_8O_2$: C, 70.58; H, 5.92. Found: C, 70.96; H, 6.12.

Reaction of I with Phenyllithium.—Phenyllithium was prepared in the manner described in the first part of the procedure for preparing II (below) from 8.6 g. of bromobenzene and 0.76 g. of lithium in 50 ml. of dry ether. The solution was cooled to −13° in an atmosphere of nitrogen with an ice-salt-bath and a solution of 6.0 g. of I in 50 ml. of dry ether was added dropwise during a period of 50 minutes with stirring and cooling to maintain a reaction temperature of −7 to −10°. The mixture became deep yellow in color. It was stirred for three hours at −15° and one hour at room temperature, and then 50 ml. of water was added, slowly at first. The yellow ether layer was separated from the dark water layer, washed with three 25-ml. portions of water, and dried over sodium sulfate. The solution was concentrated and then fractionated several times through a semimicro column.[12] The following fractions were isolated, in addition to intermediate fractions: (1) 0.3 g. of recovered I, b.p. 87° (27 mm.), $n^{25}\mathrm{D}$ 1.5375; (2) 0.5 g. of II as a yellow liquid, b.p. 113° (27 mm.), $n^{25}\mathrm{D}$ 1.5695–1.5705 (low because of exposure to air, see below); (3) 2.0 g. of a viscous, yellow liquid ketone, b.p. 120–123° (0.4 mm.), $n^{25}\mathrm{D}$ 1.5796–1.5800; (4) a few milligrams of biphenyl, m.p. 68.5–69°; (5) 11 mg. of a white solid, m.p. 159.5–161.5°, which was not characterized.

The II isolated as fraction (2) was characterized as the yellow semicarbazone, which was identical (m.p. and mixed m.p.) with a sample obtained by the preparative method described below.

The ketone which was separated as fraction (3) gave a positive unsaturation test with potassium permanganate, a very weak (negative) fuchsin aldehyde test, and no color with aqueous ferric chloride. A sample with b.p. 117–118° (0.3 mm.) and $n^{25}\mathrm{D}$ 1.5788 obtained from another preparation was analyzed. The structure of this compound was not determined.

Anal. Calcd. for $C_{14}H_{14}O$: C, 84.81; H, 7.11. Found: C, 84.76; H, 7.24.

The 2,4-dinitrophenylhydrazone of the ketone (fraction 3) was prepared in the usual way and recrystallized from a mixture of ethanol and nitromethane as orange blades, m.p. 181.8–183.2°.

Anal. Calcd. for $C_{20}H_{18}N_4O_4$: C, 63.49; H, 4.80. Found: C, 63.33; H, 5.09.

1,3,5-Cycloöctatrien-7-one (II).—A dry 2-l. three-necked flask was fitted with a mechanical stirrer and a reflux condenser and dropping funnel which were protected with drying tubes attached to a source of nitrogen under a pressure of 1–2 cm. The air in the flask was displaced with nitrogen, and 120 ml. of dry ether and 8.4 g. (1.20 gram atoms) of lithium wire (in small pieces) were added, followed by a solution of 104 g. (0.66 mole) of bromobenzene in 240 ml. of dry ether, which was added (with stirring) at a rate that maintained reflux. The mixture was heated under reflux for 30 minutes after the addition was completed and then cooled to −15° in a bath containing trichloroethylene and Dry Ice. The phenyllithium was converted to lithium diethylamide by addition of a solution of 43.8 g. (0.60 mole) of diethylamine in 120 ml. of dry ether during a period of 15 minutes. The conversion was shown to be complete by a negative color test for phenyllithium with Michler ketone.[13]

A solution of 36 g. (0.3 mole) of cycloöctatetraene oxide (I) in 120 ml. of dry ether was added to the lithium diethylamide solution with stirring during a period of ten minutes, with cooling to maintain a reaction temperature of −8 to −12°. A deep orange color formed and there was an appreciable heat of reaction. The mixture was stirred at −10° for five minutes after the addition was completed, and then 400 ml. of 3 *N* sulfuric acid was added, slowly at first and then at a rate which kept the internal temperature at 0 to −5°, with continued cooling and stirring (15 minutes). The two pale orange layers were separated and the aqueous layer was extracted with two 300-ml. portions of ether. The combined ether solutions were washed with 25 ml. of saturated sodium bicarbonate solution and 50 ml. of water, and then were dried over sodium sulfate. The ether was distilled through a 20-cm. Vigreux column and the residue was distilled through a simple still head under reduced pressure. Crude II was collected as a deep yellow liquid, b.p. 75–105° (13 mm.), $n^{25}\mathrm{D}$ 1.5750, in a yield of 34 g. (95%). This crude product was converted to the semicarbazone for purification.

Semicarbazide hydrochloride (50 g.) and 75 g. of anhy-

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

[10] A. C. Cope and L. L. Estes, Jr., THIS JOURNAL, **72**, 1129 (1950).

[11] Obtained from the Becco Sales Corporation, Buffalo, New York.

[12] C. W. Gould, Jr., G. Holzman and C. Niemann, *Anal. Chem.*, **20**, 361 (1948).

[13] H. Gilman and F. Schulze, THIS JOURNAL, **47**, 2002 (1925).

drous sodium acetate were added to a solution of the 34 g. of crude II described above in 120 ml. of 50% ethanol. A yellow solid separated as the mixture was shaken, forming a thick paste. The mixture was allowed to stand overnight at room temperature and then the solid was collected on a suction filter, washed with water, and dried at 80–90°. The crude semicarbazone was pulverized and digested with 300 ml. of water at 70–80° for 20 minutes. The mixture was cooled, filtered and the yellow powder was dried at 80–90°. The yield of the semicarbazone was 37.8 g. (71% from I), m.p. 192–194° (dec.), sample introduced at 188°). Two crystallizations from aqueous ethanol furnished an analytical sample of 1,3,5-cycloöctatrien-7-one semicarbazone as bright yellow crystals, m.p. 192.8–193.8° (turning orange at the melting point, sample introduced at 188°).

Anal. Calcd. for $C_9H_{11}N_3O$: C, 61.00; H, 6.26; N, 23.71. Found: C, 60.87; H, 6.11; N, 23.53.

1,3,5-Cycloöctatrien-7-one (II) was regenerated from the semicarbazone by hydrolysis in the presence of sulfuric acid or oxalic acid, or by the following preferred procedure through reaction with pyruvic acid. The semicarbazone (4.0 g.) was dissolved in 13.2 g. of pyruvic acid by swirling for a few minutes. The deep yellow solution was allowed to stand at room temperature for 2.5 hours in a nitrogen atmosphere. A fine granular precipitate began to form after 40 minutes and increased in amount until the reaction mixture became a thick paste. The mixture was diluted with 50 ml. of water and treated with a suspension of 15 g. of sodium bicarbonate in 50 ml. of water. The product was extracted with four 50-ml. portions of ether, and the combined extracts were washed with 25 ml. of water and dried over sodium sulfate at 5°. The solution was concentrated and the residue was fractionated through a semimicro column[12] in a nitrogen atmosphere under reduced pressure. A total of 2.37 g. (88%) of II was collected as a deep yellow liquid in five fractions, b.p. 107° (23 mm.), n^{25}_D 1.5759 to 1.5764. Reproducible values for the refractive index were obtained only if the determinations were completed rapidly with a minimum of exposure of the liquid to the air. Absorption of atmospheric oxygen caused the refractive index to drop rapidly (by 0.0045 in one minute).

Anal. Calcd. for C_8H_8O: C, 79.93; H, 6.71. Found: C, 79.77; H, 6.74.

A larger-scale regeneration of II from the semicarbazone furnished a sample of II boiling at 96–96.5° (12 mm.), n^{25}_D 1.5764, d^{25}_4 1.0494. The ultraviolet absorption spectrum of II in purified cyclohexane (Fig. 1), determined with a Beckman model DU quartz spectrophotometer, showed three maxima; $\lambda_{max.1}$ 245 mμ (ε 7030), $\lambda_{max.2}$ 282 mμ (ε 4340), $\lambda_{max.3}$ 360 mμ (ε 490).

Hydrogenation of a solution of 120 mg. of II in 10 ml. of dry methanol in the presence of 0.5 g. of 1% palladium-on-calcium carbonate[14] at room temperature and atmospheric pressure proceeded rapidly to completion in 14 minutes with the absorption of 95% of three molar equivalents of hydrogen. The reduction product was identified as cycloöctanone by treatment with semicarbazide hydrochloride and sodium acetate. The cycloöctanone semicarbazone which was formed was recrystallized from 70% ethanol as white plates, m.p. and mixed m.p. with a known sample 169.4–170°.

Hydrogenation of a solution of 120 mg. of II in 10 ml. of glacial acetic acid in the presence of 0.5 g. of pre-reduced Adams platinum oxide catalyst at room temperature and pressure was complete in two hours and 101% of four molar equivalents of hydrogen was absorbed. The reduction product was isolated by distillation, which yielded 74 mg. of cycloöctanol, identified by conversion to the phenylurethan, m.p. 55.2–57.4° (not depressed on mixture with an authentic sample).

Prior to development of the preferred procedure for preparing II from I by treatment with lithium diethylamide, mesityllithium was used as the base to effect the isomerization. Mesityllithium was prepared from 20 g. of bromomesitylene and 1.4 g. of lithium ribbon in 75 ml. of ether in the same manner as phenyllithium (described above), except that the mixture was heated under reflux for 18 hours because the reaction was slow. The mesityllithium separated as a paste, and was characterized by carbonation of a small portion, which yielded mesitoic acid, m.p. 152–154°. A solution of cycloöctatetraene oxide (6.0 g.) in 50 ml. of dry ether was added to the suspension of mesityllithium at 5–10°, and the mixture was stirred for 40 hours at room temperature. The crude II which was isolated in the manner described above contained some bromomesitylene. The crude product (4.81 g., b.p. 108–125° at 27 mm.) was converted to the semicarbazone of II, which was obtained in a yield of 3.44 g. (39% from I), m.p. 191–193° dec.

1,3,5-Cycloöctatrien-7-one 2,4-dinitrophenylhydrazone was prepared from II and recrystallized from 95% ethanol containing 15% of nitromethane as deep scarlet needles, m.p. 158.2–159.4°.

Anal. Calcd. for $C_{14}H_{12}N_4O_4$: C, 55.99; H, 4.03; N, 18.66. Found: C, 55.66; H, 3.83; N, 18.69.

1,3,5-Cycloöctatrien-7-one oxime was prepared by heating a solution of 0.6 g. of II and 0.7 g. of hydroxylamine hydrochloride in 8 ml. of 50% ethanol with 4 ml. of 10% sodium hydroxide under reflux for 90 minutes. An oil separated from the solution on cooling and crystallized slowly on scratching. The crude oxime separated as yellow needles (0.5 g.), which were purified by sublimation at 0.4 mm. with a heating block temperature of 80°, followed by crystallization from pentane and resublimation, m.p. 55–57°.

Anal. Calcd. for C_8H_9NO: C, 71.08; H, 6.71; N, 10.36. Found: C, 70.88; H, 6.68; N, 10.07.

Pyrolysis of 1,3,5-Cycloöctatrien-7-one.—A 1.2-g. sample of II was placed in a 25-ml. distilling flask attached to a receiver that was connected to a drying tube containing glass wool moistened with aniline. The outlet of the drying tube led to a gas buret. The air in the system was displaced with nitrogen, and the flask was immersed in an oil-bath at 180°. The bath was heated to 215° during a period of two hours, in which 0.43 g. of a pale yellow distillate collected (apparent b.p. 75–95°) and less than 10 ml. (5% of one molar equivalent) of gas was formed. The distillate was fractionated through a semimicro column[12] and treated with sodium, and yielded 0.27 g. of benzene, b.p. 79–80°, m.p. and mixed m.p. with a known sample 4.3–5.3°. The glass wool in the tube connected to the receiver was washed with ether to separate the aniline, and the ether solution was concentrated and dissolved in dilute hydrochloric acid. The solution appeared to contain no acetanilide, indicating that no appreciable amount of ketene was evolved as a gas during the pyrolysis.

CAMBRIDGE, MASSACHUSETTS

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[14] M. Busch and H. Stöve, *Ber.*, **49**, 1063 (1916).