Attempted ketalization of the ester with a 10-mol excess of triethyl orthoformate in refluxing absolute ethanol was completely unsuccessful.

B. From β,γ-Diphenyl-γ-chloro-$\Delta^{\alpha,\beta}$-butenolide [Pseudo-(*Z*)-β-benzoylcinnamoyl Chloride].—Hydrolysis of (*Z*)-β-benzoylcinnamic acid ethyl ester with 5% methanolic KOH afforded a 71% yield of β,γ-diphenyl-γ-hydroxy-$\Delta^{\alpha,\beta}$-butenolide [pseudo-(*Z*)-β-benzoylcinnamic acid].[19] Reaction of the hydroxybutenolide with a 2-mol excess of thionyl chloride–dimethylformamide[6] in methylene chloride gave a 92% yield of β,γ-diphenyl-γ-chloro-$\Delta^{\alpha,\beta}$-butenolide. An 82% yield of the same chlorobutenolide was obtained from treatment of the hydroxybutenolide with 1 equiv of sodium methoxide followed by 3 equiv of oxalyl chloride.[23]

Attempted reduction of the pseudo acid chloride with lithium tri-*tert*-butoxyaluminohydride[24] or sodium borohydride[25] afforded only unreacted starting chloride.

C. From α-Phenyl-$\Delta^{\alpha,\beta}$-butenolide.—Analogous to the preparation of $\Delta^{\alpha,\beta}$-butenolide from vinylacetic acid,[26] treatment of phenylvinylacetic acid with 1 equiv of performic acid followed by hydrolysis with 1.5 *M* aqueous HCl afforded a 69% yield of α-phenyl-$\Delta^{\alpha,\beta}$-butenolide.[27] Reaction of the butenolide with a 10% excess of phenyllithium analogous to the work of Pablova and coworkers[28] afforded a 75% yield of a mixture containing (nmr) 74% 2,3-diphenylfuran, 10% unreacted butenolide, and 15% of unidentified material(s).

D. From 1,2-Diphenyl-2-buten-1-one.—1-Bromo-1-phenylpropene[29] was prepared in 56% yield by reaction of propylbenzene with a 2.5-mol excess of *N*-bromosuccinimide in refluxing carbon tetrachloride followed by dehydrobromination in 10% ethanolic KOH. Treatment of the bromide with magnesium in anhydrous ether followed by condensation with benzaldehyde gave 1,2-diphenyl-1-hydroxy-2-butene[14] in 50% yield. Oxidation of this alcohol with pyridinium dichromate[30] afforded a 65% yield of 1,2-diphenyl-2-buten-1-one.[14] Attempted allylic hydroxylation of this ketone with a 10-mol excess of SeO_2 in refluxing 95% ethanol for 96 hr resulted only in cis–trans isomerization. Treatment of the ketone with 1 equiv of *N*-bromosuccinimide in refluxing carbon tetrachloride afforded a quantitative yield of 1,2-diphenyl-4-bromo-2-buten-1-one.[14] Attempted hydrolysis of this bromide with a 2-mol excess of Na_2CO_3 in 80% aqueous acetone produced only 2,3-diphenylfuran (**5**) in 70% yield.

E. From 1,2-Diphenyl-3,4-epoxy-1-butanone (10).—Rearrangement of epoxide **10** with a 10-mol excess of potassium *tert*-butoxide in *tert*-butyl alcohol at room temperature produced a nearly quantitative yield of furan **5**. Treatment of the epoxide with a catalytic amount of triethylamine in benzene solution at room temperature for 5 hr gave no reaction.

Registry No.—**1**, 30953-21-0; **2**, 30953-22-1; **5**, 954-55-2; **6**, 30953-24-3; **7**, 30953-25-4; **8**, 30953-26-5; **9**, 30953-27-6; **10**, 30953-28-7.

Acknowledgment.—We are indebted to the National Science Foundation and the Computer Center at the University of Rochester for support of this work.

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(23) R. Scheffold and P. Dubs, *Helv. Chim. Acta*, **50**, 798 (1967).

(24) H. C. Brown and R. F. McFarlin, *J. Amer. Chem. Soc.*, **80**, 5372 (1958); H. C. Brown and B. C. Subba Rao, *ibid.*, **80**, 5377 (1958).

(25) S. W. Chaiken and W. G. Brown, *ibid.*, **71**, 122 (1949).

(26) R. Palm, H. Ohse, and H. Cherdron, *Angew. Chem., Int. Ed. Engl.*, **5**, 994 (1966).

(27) G. Swain, A. R. Todd, and W. S. Warring, *J. Chem. Soc.*, 548 (1944).

(28) L. A. Pablova, V. V. Belogorodskii, and E. D. Venus-Danilova, *J. Gen. Chem. USSR.*, **36**, 1396 (1966).

(29) D. Y. Curtin and J. W. Crump, *J. Amer. Chem. Soc.*, **80**, 1922 (1958).

(30) W. M. Coates and J. R. Corrigan, *Chem. Ind. (London)*, 1594 (1969).

Photolysis of Stilbene and 1,1-Diphenylethylene in the Presence of 2-Methyl-4,5-dihydrofuran

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Received February 24, 1971

trans-Stilbene reacts with 2-methyl-4,5-dihydrofuran on photolysis to yield two cycloaddition products. The reaction proceeds *via* the excited singlet state of stilbene. The rate constant for the quenching of *trans*-stilbene fluorescence by 2-methyl-4,5-dihydrofuran is reported and approximates the diffusion-controlled rate constant. 1,1-Diphenylethylene reacts *via* its excited triplet with 2-methyl-4,5-dihydrofuran to form two cycloaddition products in contrast to the previously reported reaction with 2,3-dihydropyran in which one cycloaddition product and two other adducts are observed.

We recently reported the results of our investigations of the photochemistry of stilbene (**1**) and 1,1-diphenylethylene (**2**) in the presence of 2,3-dihydropyran (**3**).[1] We found that stilbene gave two cycloadducts (**4** and **5**) in addition to two dimers (**6** and **7**) (Scheme I) and that this reaction could not be sensitized with triplet sensitizers.[1a] The ratio of the cycloadducts **4** and **5** was independent of the initial stilbene isomer ratio. 1,1-Diphenylethylene reacted *via* its triplet state[1b] to give one cycloadduct (**8**) and two addition products (**9** and **10**) (Scheme II), which were presumably formed in consecutive reactions initiated by hydrogen abstraction by the triplet of **2** followed by free-radical coupling.[2]

In this paper we report our results for the photolysis of **1** and **2** in the presence of 2-methyl-4,5-dihydrofuran (**11**). This study was conducted for the purpose of observing the influence of ring size of the cyclic vinyl ether substrate on the reaction.

Photolysis of Stilbene in 2-Methyl-4,5-dihydrofuran.—Solutions of *cis*- and *trans*-stilbene in 2-methyl-4,5-dihydrofuran (**11**) were photolyzed at 2537 Å. The solutions were irradiated for 48 hr in quartz vessels exposed to the atmosphere. Glpc analysis indicated that stilbene was consumed and that four products were produced whose yields were virtually independent of the stilbene isomer reactant. Two products were identified

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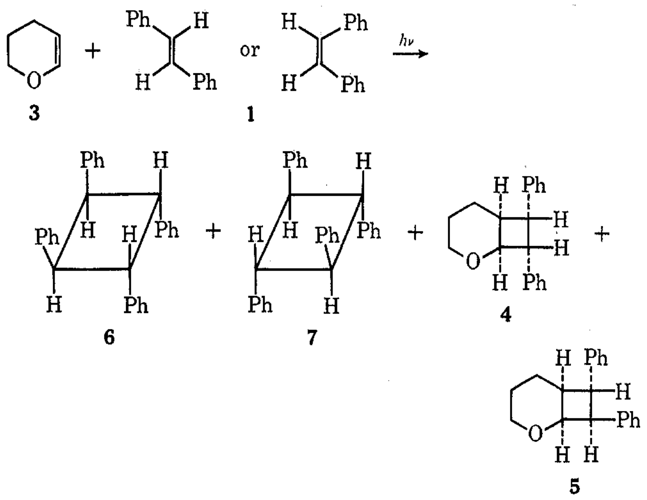
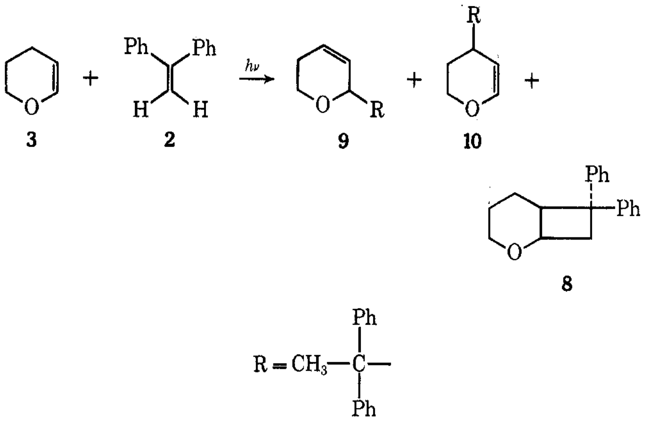
(1) (a) H. M. Rosenberg, R. Rondeau, and P. Servé, *J. Org. Chem.*, **34**, 471 (1969); (b) P. Servé, H. M. Rosenberg, and R. Rondeau, *Can. J. Chem.*, **47**, 4295 (1969).

(2) For additional examples of hydrogen abstraction by the triplet excited state of 1,1-diphenylethylene, see H. M. Rosenberg and P. Servé, *J. Amer. Chem. Soc.*, **92**, 4746 (1970), and T. S. Cantrell, *Chem. Commun.*, 1633 (1970).

SCHEME I



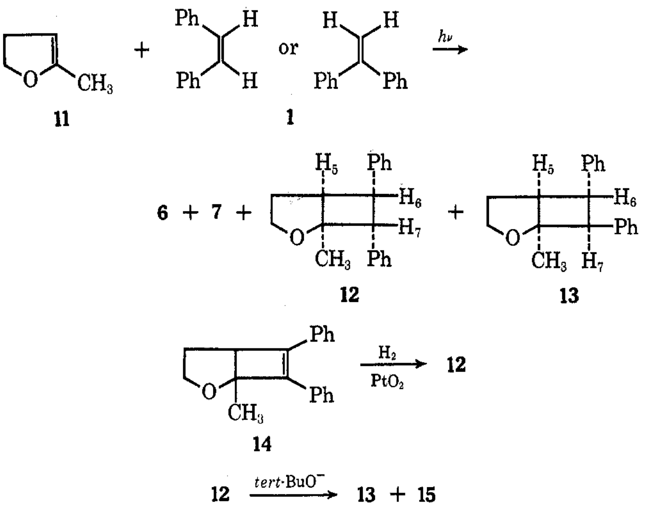
SCHEME II



as *cis,trans,cis*-1,2,3,4-tetraphenylcyclobutane (**6**) and *trans,trans,trans*-1,2,3,4-tetraphenylcyclobutane (**7**)[3] (combined yields 31%). The other products were characterized as 2-methyl-6,7-*cis*-*exo*-diphenyl-2-oxabicyclo[3.2.0]heptane (**12**, 18%) and 2-methyl-6-*exo*,7-*endo*-diphenyl-2-oxabicyclo[3.2.0]heptane (**13**, 29%).

The structural assignments of **12** and **13** were based on the following evidence (Scheme III). High-resolution mass spectra established that both products were 1:1 adducts of stilbene and **11**. Catalytic hydrogenation of 6,7-diphenyl-2-oxabicyclo[3.2.0]hept-6-ene (**14**)[4] yielded only one product whose glpc retention time and ir spectrum were identical with those of **12**. Compound **12**, on treatment with base, gave two products having similar but not identical glpc retention times. The product with the longer retention time was found to have the identical retention time with that of **13** on two different columns. A small amount of the product with the larger retention time was isolated, and its ir spectrum was identical with that of **13**. The other product (**15**) of the base isomerization was not isolated in sufficient quantity to permit positive identification. These data indicate that **12** and **13** are *cis*- and *trans*-diphenyl isomers, respectively, since catalytic hydrogen of **12** is expected to give the cis product, which should isomerize in base to the more stable trans isomers.

SCHEME III



An attempt was made to deduce the remaining stereochemical features of **12** and **13** from their nmr spectra, details of which are given in the Experimental Section. For compound **12** the pertinent coupling constants (Hz) were found to be $J_{5,6} = 2.5$, $J_{6,7} = 7.0$, which appear to be consistent with the *cis*-*exo*-diphenyl structure,[5,6] although there are considerable uncertainties in basing structural assignments in cyclobutane systems on coupling constants.[6] However, the anomalously high chemical shift, *ca.* τ 7.3, for the bridgehead hydrogen H_5 suggests shielding from the phenyl substituent cis to this proton and provides support for the assigned structure.[7] Similarly, the 6-*exo*,7-*endo*-diphenyl structure proposed for **13** rests on the chemical shift, τ 7.4, of C_5,[7] in addition to its formation in the base-catalyzed isomerization of the *cis*-diphenyl isomer (*vide supra*).

Solutions of *cis*- and *trans*-stilbene (0.1 *M*) in **11** exposed to the atmosphere were irradiated at 2537 and 3000 Å. The ratio of **12** to **13** (1:1.6) remained constant throughout all reactions and was independent of the initial *cis*-stilbene:*trans*-stilbene ratio. Isomerization of stilbenes was observed and the cis:trans isomer ratios varied continuously during the course of the reactions, as shown in Table I.

TABLE I

PHOTOLYSIS OF *cis*- AND *trans*-STILBENE IN 2-METHYL-4,5-DIHYDROFURAN AT 2537 Å

Time, min	[*cis*]/[*trans*]	Product yield, %
	cis-Stilbene	
15	26.1	6.2
30	12.8	19.8
45	7.6	24.0
75	7.0	40.1
	trans-Stilbene	
15	0.03	77.5
30	0.07	81.0
45	0.15	89.0
75	0.50	98.0

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[3] H. Schecter, W. J. Link and G. V. D. Tiers, *J. Amer. Chem. Soc.*, **85**, 1601 (1963).

[4] M. P. Servé and H. M. Rosenberg, *J. Org. Chem.*, **35**, 1237 (1970).

[5] I. Fleming and D. H. Williams, *Tetrahedron*, **23**, 2747 (1967).

[6] J. Krepinsky, Z. Samek, and F. Sorm, *Tetrahedron Lett.*, 3209 (1966).

[7] We are indebted to Professor L. A. Paquette, who provided us with the nmr spectrum of 2-oxabicyclo[3.2.0]heptane[8] in which the signal for H_5 was a multiplet centered at τ 6.9.

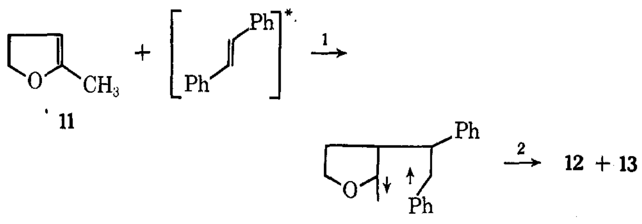
[8] L. A. Paquette, J. H. Barrett, R. P. Spitz, and R. Pitcher, *J. Amer. Chem. Soc.*, **87**, 3417 (1965).

The rate of product formation was significantly lower for the initial solution of *cis*-stilbene. Photolysis of a degassed solution of *trans*-stilbene (0.01 *M*) in **11** containing triphenylene (0.05 *M*) at 3500 Å provided evidence for sensitized isomerization of stilbene, but no sensitized formation of **12** and **13**.

The formation of cycloaddition products **12** and **13** therefore proceeds *via* the excited singlet states of stilbene. The shorter lifetime of the cis excited singlet[9] probably accounts for the lower reaction rate observed with *cis*-stilbene. The invariance of product ratio requires a common transition state or intermediate for cycloaddition reactions with *cis*- or *trans*-stilbene.

The formation of two isomeric products, in which the phenyl substituents at C_6 are exo, suggests a nonconcerted mechanism (Scheme IV). The first step appears to involve attack by the *trans*-stilbene excited singlet to give the more stable diradical, followed by ring closure to yield products. The nonconcerted process is consistent with the concept of nonplanar excited states of olefins.[10]

SCHEME IV



A linear Stern–Volmer plot obtained for the quenching of *trans*-stilbene (10^{-4} *M*) by **11** (4×10^{-2}–2×10^{-1} *M*) in *n*-hexane is consistent with the following process.[11]

$$t^0 \xrightarrow{h\nu} t'$$

$$t' \xrightarrow{k_f} t^0 + h\nu$$

$$t' \xrightarrow{k_d} c^0 + t^0$$

$$t' + 11 \xrightarrow{k_p} 12 + 13$$

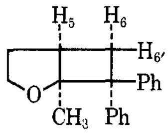
Where t and c are *trans*- and *cis*-stilbene respectively, k_f is the rate constant for fluorescence, k_d is the sum of all first-order radiationless decay processes,[13] and k_p is the bimolecular rate constant for quenching. Since the energy level of the *trans*-stilbene excited singlet is considerably lower than that of excited **11**, collisional energy transfer is unlikely and k_p may closely approximate the reaction rate constant in the absence of reversible excimer formation.

The linear plot of Φ_0/Φ *vs.* [**11**] gave slope 2.0 and intercept 1.0 in accordance with the following equation.

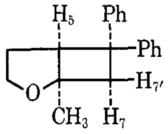
$$\Phi_0/\Phi = 1 + k_p[11](k_f + k_d)$$

Using the reported values of $\Phi_0 = 0.06$[14] and $k_f = 4 \times 10^8$ sec^{-1},[15] the computed value of k_p is 1×10^{10} l. mol^{-1} sec^{-1}. It is noted that k_p closely approximates the computed diffusion-controlled rate constant for hexane, 2.0×10^{10} l. mol^{-1} sec^{-1}.[16]

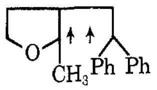
Photolysis of 1,1-Diphenylethylene in 2-Methyl-4,5-dihydrofuran.—Solutions of **2** in **11** were irradiated at 2537 and 3000 Å for 48 hr. Two major products were detected by glpc in each photolysis. The products were isolated by column chromatography and were identified from spectral data as 1-methyl-7,7-diphenyl-2-oxabicyclo[3.2.0]heptane (**16**) and 1-methyl-6,6-diphenyl-2-oxabicyclo[3.2.0]heptane (**17**). The ratio of **16**:**17** was 4.5:1. Separate irradiations of **16** and **17** showed that they were not interconvertible under the reaction conditions.



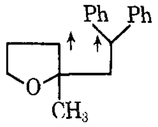
16



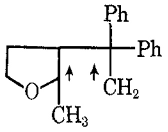
17



18



19



20

The parent peaks in the mass spectra of **16** and **17** corresponded to 1:1 adducts, $C_{19}H_{20}O$. The ir spectra of **16** and **17**, detailed in the Experimental Section, showed that the tetrahydrofuran ring system was still intact; both molecules also contained a methyl group and a monosubstituted phenyl. Notable by its absence was the band at 1750 cm^{-1} which was present in 2-methyl-4,5-dihydrofuran and is characteristic of an α,β-unsaturated ether.[17] The nmr spectra of **16** and **17** permitted final structure elucidations. The nmr of **16**, detailed in the Experimental Section, showed two sets of peaks each containing a doublet of doublets at τ 7.85 (J = 6.0 and 10.5 Hz) and 7.03 (J = 6.0 and 10.5 Hz). These peaks were assigned to hydrogens 6 and 6′. The nmr of **17** showed the presence of two doublets at τ 8.02 and 6.95 (J = 13 Hz); these were attributed to H_7 and $H_{7'}$. A doublet of doublets at τ 7.45 (J = 7.5 and 4.2 Hz) was assigned to H_5. Both **16** and **17** were assigned a cis ring fusion based on molecular models.

Sensitization and quenching experiments were performed in order to gain information regarding the reactive excited species. It was found that pyrene (E_T = 48.7 kcal/mol[18]) inhibited the reaction between 1,1-

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[9] G. N. Lewis, T. T. Magel, and D. Lepkin, *ibid.*, **62**, 2973 (1940); D. Schulte-Frohlinde, H. Blume, and H. Gusten, *J. Phys. Chem.*, **66**, 2486 (1962); A. A. Lamola, G. S. Hammond, and F. B. Mallory, *Photochem. Photobiol.*, **4**, 259 (1964).

[10] G. S. Hammond, *Advan. Photochem.*, **7**, 377 (1969).

[11] Chapman and Lura[12] propose exciplex formation in the photolysis of *trans*-stilbene in the presence of tetramethylethylene. More complex kinetics are required for this situation and our treatment may be oversimplified.

[12] O. L. Chapman and R. D. Lura, *J. Amer. Chem. Soc.*, **92**, 6352 (1970).

[13] Stilbene dimers **6** and **7** are not formed in these reactions because of the low stilbene concentration.

[14] D. Gegiou, K. A. Muszkat, and E. Fischer, *J. Amer. Chem. Soc.*, **90**, 3907 (1968).

[15] R. H. Dyck and D. S. McClure, *J. Chem. Phys.*, **36**, 2326 (1962).

[16] A. A. Lamola and N. J. Turro, "Energy Transfer and Organic Chemistry," Interscience, New York, N. Y., 1969, p 34.

[17] L. Bellamy, "The Infrared Spectra of Complex Molecules," 2nd ed, Wiley, New York, N. Y., 1966, p 119.

[18] W. G. Herkstroeter, A. A. Lamola, and G. S. Hammond, *J. Amer. Chem. Soc.*, **87**, 453 (1965).

diphenylethylene (E_T = 54.5 kcal/mol[19]) and 2-methyl-4,5-dihydrofuran at 2537 Å. Equimolar concentrations of 1,1-diphenylethylene and pyrene were used. Since their molar extinction coefficients are about the same at the absorption wavelength (log ϵ 4.1 at 2537 Å[20]), quenching was attributed to triplet energy transfer rather than absorption of light by pyrene. The use of triphenylene (E_T = 66.6 kcal/mol[18]) as sensitizer for the reaction run on a degassed sample in a Pyrex vessel at 3500 Å proved successful. The product distribution in the sensitized reaction was unchanged from the unsensitized reaction run at 2537 Å. The unsensitized reaction does not occur at 3500 Å.

The sensitization and quenching experiments indicate that the reaction proceeds by way of an excited triplet state of 1,1-diphenylethylene.

The product distribution can be rationalized on the basis of the expected differences in stability of the diradical intermediates, assuming that the transition states resemble the intermediates. Thus **16** most likely proceeds from **18** whereas **17** requires **19** or **20**, both of which should be less stable than **18**.

The following differences are noted for the reaction of 1,1-diphenylethylene triplet with 2-methyl-4,5-dihydropyran and 4,5-dihydropyran: addition products involving initial hydrogen abstraction are observed only for the six-membered vinyl ether ring; both isomeric cycloaddition products occur in the reaction with the five-membered ring and only one for the six-membered ring.

Experimental Section

Melting points are uncorrected. Photolyses were conducted in a Rayonet photochemical reactor at 2537, 3000, and 3500 Å as indicated. The infrared spectra were obtained on a Perkin-Elmer Model 621 spectrophotometer. High-resolution mass spectra were obtained on a CEC-21-110 instrument. Glpc were performed on a Varian Aerograph Model 1200 HYFI on 6-ft columns packed with 10% Apiezon L on Chromosorb W or 10% SE-30 on Chromosorb W. Nmr spectra were taken on a Varian DP-60-IL instrument. An Aminco–Bowman spectrofluorimeter was used for fluorescence measurements.

Reaction of *cis*- or *trans*-Stilbene with 2-Methyl-4,5-dihydrofuran.—In a quartz vessel a solution of 5.0 g (0.02 mol) of stilbene in 70 ml of **11** was irradiated at 2537 Å in a Rayonet photochemical reactor for 48 hr. After removal of unreacted **1** under reduced pressure, the crude reaction mixture was heated with petroleum ether (bp 30–60°) and then cooled. A white, crystalline product, 2.70 g (31% based on reacted stilbene) was isolated by filtration and characterized as a mixture of *cis,trans,cis*-1,2,3,4-tetraphenylcyclobutane (**6**) and *trans,trans,trans*-1,2,3,4-tetraphenylcyclobutane (**7**): nmr ($CDCl_3$) τ 2.75, 2.94 (20 H, singlets, aromatic protons), 5.54 and 6.33 (4 H, singlets, methine protons) (lit.[3] τ 2.95 and 5.60 for **6** and 2.79 and 6.37 for **7**). The filtrate was subjected to column chromatography on alumina (80–200 mesh). Elution with petroleum ether resulted first in 2-methyl-6,7-*cis-exo*-diphenyl-2-oxabicyclo[3.2.0]heptane (**12**): 0.8 g (18%); mp 38–40°; parent peak 264.1481 ($C_{19}H_{20}O$); ir (thin film) 3040 (aromatic CH), 2900 (aliphatic CH), 1610, 1500 (aromatic C═C), 1050 (COC), 740 and 659 cm^{-1} (monosubstituted phenyl); nmr ($CDCl_2$) τ 2.8–3.0 (10 H, multiplet, aromatic protons), 6.05 (1 H, doublet, H_7), 6.25 (2 H, multiplet, H_3 and $H_{3'}$), 6.40 (1 H, doublet of doublets, H_6), 7.35 (1 H, multiplet, H_5), 8.4–8.6 (2 H, multiplet, H_4 and $H_{4'}$), 8.60 (3 H, singlet, CH_3), $J_{5,6}$ = 2.5, $J_{6,7}$ = 7.0 Hz. Further elution with petroleum ether resulted in the isolation of 2-methyl-6-*exo*,7-*endo*-diphenyl-2-oxabicyclo[3.2.0]heptane (**13**): 1.55 g (29%); mp 52–54°; parent peak 264.1488 ($C_{19}H_{20}O$); ir (thin film) 3040 (aromatic CH), 2930 (aliphatic CH), 1640, 1480 (aromatic C═C), 1080 (COC), 735 and 700 cm^{-1} (monosubstituted phenyl); nmr ($CDCl_3$) τ 2.8–3.0 (10 H, multiplet, aromatic protons), 6.15 (2 H, multiplet, H_3 and $H_{3'}$), 6.55 (1 H, doublet of doublets, H_6), 6.70 (1 H, doublet, H_7), 7.40 (1 H, multiplet, H_5), 8.3 (2 H, multiplet, H_4 and $H_{4'}$), 8.85 (3 H, singlet, CH_3), $J_{5,6}$ = $J_{6,7}$ = 6.5 Hz.

Hydrogenation of 2-Methyl-6,7-diphenyl-2-oxabicyclo[3.2.0]-hept-6-ene.—A solution of 2.5 g (0.01 mol) of **14** in 15 ml of anhydrous ether was hydrogenated over prereduced platinum at atmospheric pressure. After 1 equiv of hydrogen had been absorbed, the hydrogenation was stopped, the solution was filtered, and the ether was carefully removed. The remaining oil was analyzed by glpc and was found to contain only one component. The retention time of the hydrogenation product was identical with that of **12** and its ir spectrum was superimposable on that of **12**.

Isomerization of 2-Methyl-6,7-*cis-exo*-diphenyl-2-oxabicyclo-[3.2.0]heptane.—A mixture of **12**, 1 g, and 2 g of potassium *tert*-butoxide in 50 ml of *tert*-butyl alcohol was refluxed for 12 hr. The solvent was removed under vacuum and the residue was taken up in ether which was then washed with water and dried over anhydrous sodium carbonate. The ether solution was analyzed by glpc. The chromatograph showed two new peaks with approximately equal areas. The retention times on two different columns of the component with the longer retention time was identical with that of **13**. Chromatography on alumina resulted in the isolation of a pure sample of the component with the longer retention time and its ir spectrum was identical with that of **13**.

Reaction of 1,1-Diphenylethylene with 2-Methyl-4,5-dihydrofuran.—A solution of 1,1-diphenylethylene (2 g, 0.011 mol) in 2-methyl-4,5-dihydrofuran (25 g, 0.3 mol) was placed in a quartz vessel. The solution was degassed using three alternative freeze–thaw sequences. The solution was then irradiated at 2537 Å for 48 hr. After removal of the unreacted dihydrofuran under reduced pressure, the remaining liquid was subjected to column chromatography on alumina (80–200 mesh). Elution with petroleum ether gave 1-methyl-7,7-diphenyl-2-oxabicyclo[3.2.0]heptane (**16**): 1.6 g (63%); parent peak 264.1071 ($C_{19}H_{20}O$); ir (thin film) 3028 (aromatic CH), 2860 (aliphatic CH), 1595 (aromatic C═C), 1090 (COC), and 745 and 690 cm^{-1} (monosubstituted phenyl); nmr (C_6D_6) τ 8.8 (3 H, singlet, CH_3), 8.4–8.6 (2 H, multiplet, H_4 and $H_{4'}$), 7.85 (1 H, two doublets, J = 6.0 and 10.5 Hz, H_6 or $H_{6'}$), 7.50 (1 H, multiplet, H_5), 7.03 (1 H, two doublets, J = 6.0 and 10.5 Hz, H_6 or $H_{6'}$), 6.3–6.4 (2 H, multiplet, H_3 and $H_{3'}$), 2.8 (10 H, multiplet, aromatic H).

Further elution with petroleum ether gave 1-methyl-6,6-diphenyl-2-oxabicyclo[3.2.0]heptane (**17**): 0.29 g (12%); parent peak 264.1129 ($C_{19}H_{20}O$); ir (thin film) 3035 (aromatic CH), 2870 (aliphathic CH), 1595 (aromatic C = C), 1090 (COC), 745 and 690 (monosubstituted phenyl); nmr (C_6D_6), τ 8.9 (3 H, singlet, CH_3), 8.4–8.6 (2 H, multiplet, H_4 and $H_{4'}$), 8.02 (1 H, doublet, J = 13 Hz, $H_{7'}$), 7.45 (1 H, two doublets, J = 7.5 and 4.5 Hz, H_5), 6.95 (1 H, doublet, J = 13 Hz, $H_{7'}$), 6.2–6.4 (2 H, multiplet, H_3 and $H_{3'}$), 2.80 (10 H, multiplet, aromatic H).

Registry No.—*cis*-**1**, 645-49-8; *trans*-**1**, 103-30-0; **2**, 530-48-3; **11**, 1487-15-6; **12**, 31020-04-9; **13**, 31020-05-0; **16**, 31020-06-1; **17**, 31020-07-2.

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