Tetrahydrofuran Decomposition. Condensation of Solvent Fragment with Benzophenone and Trityllithium[1a,b]

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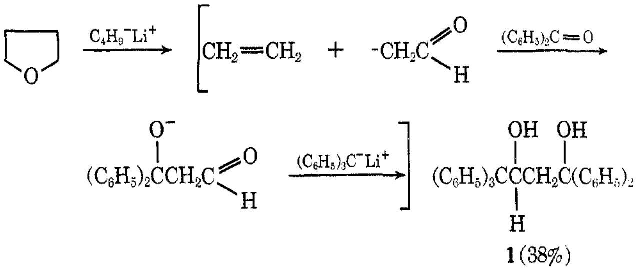
Decomposition of tetrahydrofuran by *n*-butyllithium producing ethylene and the enolate ion of acetaldehyde has been noted. Condensation of the latter with benzophenone and trityllithium results in the formation of significant quantities of 1,1,4,4,4-pentaphenyl-1,3-butanediol (1). Conformational analysis of diol 1 by nmr is discussed. None of the para-condensation product[2] of trityllithium with benzophenone is observed in this solvent.

This study of the decomposition of tetrahydrofuran (THF) resulted from an examination of the condensations of the bulky trityllithium reagent with several large molecules in different solvents. In our earlier work,[2] reaction of trityllithium with benzophenone in tetrahydropyran (THP) produced the para-condensation product *p*-(diphenylmethyl)diphenylhydroxymethylbenzene. The reduction of steric crowding achieved by para condensation of trityllithium was intriguing, but when the reaction solvent is THF a more unusual result is observed. THF decomposes in the presence of *n*-butyllithium and trityllithium, yielding ethylene and the enolate ion of acetaldehyde. The latter condenses with benzophenone and trityllithium, furnishing 1,1,4,4,4-pentaphenyl-1,3-butanediol (1) in yields as high as 38% (Scheme I). *THF decomposition with subsequent two-carbon insertion has not been reported previously.* Low yields of diol 1 were obtained when 2-methyltetrahydrofuran was employed as solvent.

SCHEME I



Structure of Condensation Product.—Base-catalyzed thermal decomposition of diol 1 yielded triphenylmethane (86%), benzophenone (41%), and acetaldehyde (10%). 1,1,2,4,4-Pentaphenyl-1,3-butadiene (3) (Scheme II) resulted (74%) from treatment of diol 1 with iodine in acetic acid for 1 hr; dehydration of the secondary hydroxyl function is accompanied by rearrangement. 1,1,1,4,4-Pentaphenylbut-3-en-2-one (2) was isolated (63%) after refluxing diol 1 with acetic acid and sodium dichromate for 2.5 hr. Base-catalyzed thermal decomposition of ketone 2 furnished triphenylmethane and benzophenone. Acidification of the aqueous layer of the above decomposition produced 3,3-diphenylacrylic acid (5) in an 82% yield. Diol 1 dissolved in methylene chloride and refluxed for 2 hr with acetic acid and chromium trioxide yielded 1,1,1,4,4-pentaphenyl-4-hydroxy-2-butanone (4) (56%). Apparently, because of milder reaction conditions used in this oxidation, dehydration to the butenone 2 does not occur.

Keto alcohol 4 refluxed with iodine in acetic acid furnished ketone 2. Base-catalyzed thermal decomposition of keto alcohol 4 yielded triphenylmethane and benzophenone. The mass and spectroscopic data support the proposed structure of diol 1. The mass spectrum of diol 1 was analyzed by an element-mapping technique.[3] Dehydration of diol 1 to butadiene 3 in the mass spectrometer prevented observation of the parent peak of the diol, but fragments of both diol 1 and butadiene 3 were found to be present in the element map.

Using *p*-phenylbenzophenone instead of benzophenone, the corresponding analog of diol 1, 1-*p*-biphenyl-1,4,4,4-tetraphenyl-1,3-butanediol (6), was formed; its identity was established by comparison of its spectral characteristics with those of diol 1.

Decomposition of THF.—Ring opening[4-6] and decomposition[7-11] of THF with various organometallic reagents have been reported, but significant quantities of products from decomposition reactions have never been isolated. Bates has recently reported THF decomposition by *n*-butyllithium forming ethylene and the enolate ion of acetaldehyde.[11] The reactions were carried out in an nmr sample tube and the products were identified spectroscopically. THF has been found to decompose in the presence of Grignard reagents under some conditions. Nmr spectra of strongly heated Grignard reagents frequently have sharp resonances at τ 4.64, which is indicative of ethylene dis-

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(1) (a) This research was supported in part by a Frederick Gardner Cottrell grant; (b) presented in part at the 155th National Meeting of the American Chemical Society, San Francisco, Calif., April 1968; (c) to whom correspondence should be addressed; (d) National Science Foundation Undergraduate Research Participant.

(2) P. Tomboulian and K. Stehower (Dumke), *J. Org. Chem.*, **33**, 1509 (1968).

(3) Data obtained from high-resolution mass spectrometry were converted by computer to the elemental composition of the ion fragments *via* their accurate masses. Computer selection of empirical formulas was within ±3 mmu. See K. Biemann, P. Bommer, and D. M. Desiderio, *Tetrahedron Lett.*, **26**, 1725 (1964). We are indebted to Dr. Ronald Hites for these spectral determinations.

(4) F. R. Jensen and R. L. Bedard, *J. Org. Chem.*, **24**, 874 (1959).

(5) W. J. Bailey and F. Marktscheffel, *ibid.*, **25**, 1797 (1960).

(6) H. Gilman and B. J. Gaj, *ibid.*, **28**, 1725 (1963).

(7) R. L. Letsinger and D. F. Pollart, *J. Amer. Chem. Soc.*, **78**, 6079 (1956).

(8) A. Rembaum, S.-P. Siao, and N. Indictor, *J. Polym. Sci.*, **56**, No. 163, S17 (1962).

(9) E. A. Hill, *J. Org. Chem.*, **31**, 20 (1966).

(10) S. C. Honeycutt, *J. Organometal. Chem.*, **29**, 1 (1971).

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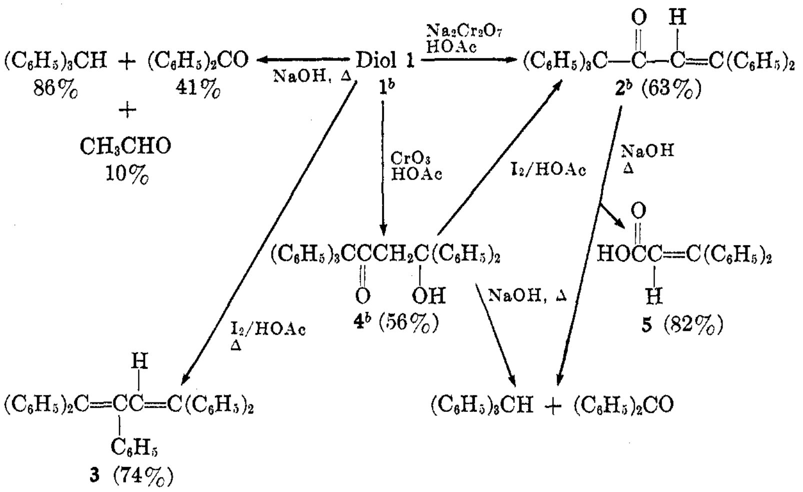
SCHEME II[a]



[a] Reactions of diol 1. [b] New compounds.

solved in a Grignard solution;[9] the enolate ion could not be identified by nmr spectroscopy.

THF decomposition products have been isolated from reactions involving substituted THF's and organometallic reagents. The production of ethylene (95.5%) and acetophenone (56%) has been observed when 2-phenyltetrahydrofuran was treated with phenyllithium and the mixture was hydrolyzed.[7] This reaction clearly is more likely to occur than a decomposition involving an unsubstituted THF. Less than a 5% yield of ethylene was obtained when propylsodium and THF were allowed to react in hexane at 50°.[7] Decomposition of THF in the presence of ethyllithium has been reported[8] but the reaction products were not clearly identified. In the above THF reactions only small quantities of decomposition products, if any, were actually isolated.

In our system a significant quantity of the decomposition products have been obtained. To our knowledge, this is the first reported condensation of the acetaldehyde fragment of THF. This unusual combination of reactants traps the enolate ion of acetaldehyde and incorporates it in a stable adduct, diol 1. The stabilized enolate ion resulting from THF decomposition has no counterpart in the THP solvent system, in which no diol is formed. The other product of THF decomposition, ethylene, was isolated following triphenylmethane addition to the n-butyllithium solution while the mixture was permitted to warm to room temperature. Infrared analysis of the gases released at this point indicated the presence of butane and ethylene.

The quantity of diol 1 produced indicates that solvent decomposition can be of major significance in systems containing n-butyllithium and THF. The calculated yield of the diol (38% based on triphenylmethane) assumes that the trityl anion plays little or no role in solvent decomposition. The basis for this assumption is that n-butyllithium is a stronger base than trityllithium and appears to be much less stable in THF.[10-14] The details of the solvent decomposition are as yet unclear, although mechanisms have been proposed.[7,8,11] Recent kinetic data suggest that the mechanism is complex; the rate of reaction of n-butyllithium was found to be first order in n-butyllithium but 2.5 order in THF.[10]

A possible reaction mechanism for the decomposition of THF is the nucleophilic abstraction of an α proton by n-butyllithium resulting in the cleavage of the solvent molecule to furnish ethylene and the enolate ion of acetaldehyde. Condensation of benzophenone, the enolate ion, and a trityl ion follows, as is evidenced by the formation of diol 1 (Scheme I). The order of condensation of these species has not been established. The inherent basicity of both the enolate and trityl ions would favor a condensation between benzophenone and the enolate followed by addition of the trityl anion to the new and less basic species.

Diol 1 results (9% yield) when commercial n-butyllithium in hydrocarbon solvent is added to a solution of triphenylmethane in THF, indicating that the THF decomposition reaction competes significantly with the metalation reaction. *Thus THF decomposition may be quite common in systems containing organolithium reagents*, and the involvement of THF in these systems probably has been underestimated.

This reaction scheme has been attempted in other solvents. When 2-methyltetrahydrofuran was employed, diol 1 was obtained in low yield (<1%), which can be attributed to the higher stability of this solvent. The half-life for α cleavage at 35° has been found to be 13 times longer for 2-methyltetrahydrofuran than for THF.[11] Attempted cleavage and condensation reactions in THF-d_4 and THF-d_8 failed; adequate yields of trityllithium were not obtained presumably owing to solvent impurities.

Conformational Studies by Nmr Spectroscopy.—The nonequivalency of the aliphatic protons of diol 1, as revealed by the nmr spectrum, suggested a further study of the conformation of this molecule.[15] 1,1-Diphenyl-4,4,4-trichloro-1,3-butanediol (7) was prepared and its nmr spectra were similar to those of diol 1. The mono- and dimethyl ethers of diol 7 also were synthe-

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(12) H. Gilman and B. J. Gaj, *J. Org. Chem.*, **22**, 1165 (1957).

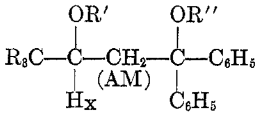
(13) R. Waack and P. West, *J. Amer. Chem. Soc.*, **86**, 4494 (1964).

(14) The deep red color of the trityl anion persists after stirring for 24 hr at room temperature.

(15) We are indebted to Dr. Steven Beare for these syntheses and nmr determinations.

TABLE I

NMR DATA FOR DIOLS AND THE MONO- AND DIMETHYL ETHER OF DIOL 7



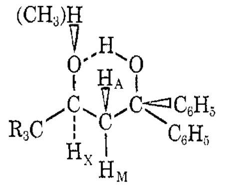
$R_3C{-}C(OR')(H_X){-}CH_2\,(AM){-}C(OR'')(C_6H_5){-}C_6H_5$

Solvent	R	R′	R″	Registry no.	J_{AX}, Hz	J_{MX}, Hz	J_{AM}, Hz	δ_{AM}, ppm	δ, ppm[a] H_A	H_M	H_X	OR′	OR″
Benzene	C_6H_5	H	H	36976-71-3	10.0		14.7		1.32	2.22	4.83	1.98[b]	3.67
$CDCl_3$[c]	C_6H_5	H	H		10.0	0.5	14.7	0.84	1.83	2.67	5.25		
CS_2[d]	C_6H_5[e]	H	H		10.0		14.7		1.7	2.7	5.3	2.3[f]	4.6
Acetone-d_6	Cl	H	H	36976-72-4	9.7	1.9	14.4	0.72	2.58	3.31	4.25	2.98	2.98
DMSO	Cl	H	H		8.1	≤ 1					1.55	4.33[g]	3.44
$CDCl_3$	Cl	H	H		10.1	1.8	14.7	0.57	2.69	3.26	4.22	3.90[h]	4.05
CCl_4	Cl	CH_3	H	36976-73-5	8.5	2.2	14.5	0.49	2.53	3.08	3.80	3.22	3.64
DMSO	Cl	CH_3	H		4.0[i]	4.0[i]					1.36	0.80	3.50
CCl_4	Cl	CH_3	CH_3	36976-74-6	5.1	3.4		0.007	2.90	2.90	3.58	3.17	3.41

[a] All spectra were obtained at 100 MHz and 27° unless otherwise stated, relative to TMS except for DMSO, where DMSO is used as an internal standard. [b] $J_{CHX OH}$ = 4.5 Hz. [c] D_2O was added to the nmr tube. [d] Spectra obtained at 60 MHz. [e] Diol 6. [f] $J_{CHX OH}$ = 5.0 Hz. [g] $J_{CHX OH}$ = 5.2 Hz. [h] $J_{CHX OH}$ = 3.0 Hz. [i] Measured from 1000-Hz sweep width.

sized in order to determine the extent of intramolecular hydrogen bonding[16] in this molecule.

The nmr spectral data indicate (Table I) that the conformation of these diols is indeed dependent on intramolecular hydrogen bonding, and the following model is suggested. This cyclic conformation would



place all of the aliphatic protons in dissimilar magnetic environments, and is also consistent with the observed coupling constants J_{AX}, J_{MX}, and J_{AM}.[17]

Evidence for intramolecular hydrogen bonding was obtained from the nmr spectra of diol **7** and its mono- and dimethyl ethers. The AMX pattern remained essentially unchanged in diol **7** and its monomethyl ether, indicating that significant intramolecular association exists in these compounds. The association is lost in the dimethyl ether, as evidenced by the chemical shift and coupling constant differences between the mono- and dimethyl ethers in the same solvent.[18] The intramolecular hydrogen bonding in the monomethyl ether, and presumably in diols **1** and **7**, occurs through the tertiary hydroxyl hydrogen and the secondary hydroxyl oxygen.[19]

Thus it appears that diols **1** and **7** and the monomethyl ether have similar conformations in nonpolar solvents. The same conformation is preserved for diol **7** in acetone-d_6 and DMSO; however, the monomethyl ether exhibits a loss of this conformation in DMSO. In the dimethyl ether there is no association, and the small difference in J_{AX} and J_{MX} is only a reflection of electronic interactions.

Experimental Section

General.—Melting points are corrected. Microanalyses were performed by Clark Microanalytical Laboratory, Urbana, Ill.; Galbraith Laboratories, Inc., Knoxville, Tenn.; and Spang Microanalytical Laboratory, Ann Arbor, Mich. Infrared spectra were measured with Beckman IR-5 and IR-12 spectrophotometers. Nmr spectra were recorded on Varian T-60, HA-100, and A-56/60 and Perkin-Elmer R-12 spectrometers. TMS was used as an internal standard except where noted otherwise.

Materials.—THF and 2-methyltetrahydrofuran were distilled from $LiAlH_4$ in an argon atmosphere. Argon was employed for all trityllithium reactions, and was purified by bubbling through a benzophenone–lithium ketyl mixture in THF.

1,1,4,4,4-Pentaphenyl-1,3-butanediol (1).—In a typical experiment trityllithium[2] was prepared by slowly adding 1.0 g (4.2 mmol) of triphenylmethane dissolved in 10 ml of THF to a 180% excess of freshly prepared *n*-butyllithium in THF and allowing the mixture to stir at room temperature for 3 hr. To the deep red mixture, 2.0 g (11 mmol) of benzophenone in 10 ml of THF was added. After 18 hr of stirring, the purple solution was treated with cold 9 *M* hydrochloric acid. Concentration of the organic layer furnished a solid, which when recrystallized yielded 0.76 g (38% based on triphenylmethane) of diol 1: mp 192–194°; ir (CS_2) 3467 (broad OH), 3079, 3050, 3022, 2964, 2929, 1069, 858, 770, 760, 743, and 591 cm^{-1}; nmr ($CDCl_3/D_2O$) δ 5.25 (m, 1, *J* = 10.0, 0.5 Hz, H_X), 2.67 (m, 1, *J* = 14.7, 0.5 Hz, H_M), 1.83 (m, 1, *J* = 14.7, 10.0 Hz, H_A); mass spectrum *m/e* (rel intensity) 434 (6), 357 (1), 356 (1), 279 (2), 267 (5), 244 (7), 243 (8), 183 (5), 180 (5); decomposition in spectrometer.

Anal. Calcd for $C_{34}H_{30}O_2$: C, 86.78; H, 6.43. Found: C, 87.17, 87.10; H, 6.19, 6.44.

Diol 1 from Preparation of Trityllithium Using Commercial *n*-Butyllithium.—Triphenylmethane (3.5 g, 14 mmol) was dissolved in 20 ml of THF. To this mixture 10 ml (16 mmol) of commercial *n*-butyllithium in hexane (Foote Mineral Co.) was added, causing the solution to warm and turn deep red. After the solution was stirred for 15 min, 2.6 g (14 mmol) of benzophenone was added over a period of 35 min. The mixture was stirred for 35 min and yielded 0.42 g (9% based on benzophenone) of white crystals, mp 195–196°. Infrared spectroscopic data indicate that this material is diol 1.

1-*p*-Biphenyl-1,4,4,4-tetraphenyl-1,3-butanediol (6).—A THF solution of 1.3 g (5.1 mmol) of *p*-phenylbenzophenone was added

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[16] Studies of intramolecular hydrogen bonding in 2,4-pentanediols have been reported previously. See (a) P. E. McMahon and W. C. Tincher, *J. Mol. Spectrosc.*, **15**, 180 (1965); (b) S. Fujiwara, Y. Fujiwara, K. Fujii, and T. Kukuroi, *ibid.*, **19**, 294 (1966).

[17] In substituted ethanes dihedral angles between vicinal protons of 180 and 70° correspond to coupling constants of 9 and 0.5 Hz, respectively. Geminal coupling constants of 14–15 Hz result if the H–C–H bond angle is 109°. See J. R. Dyer, "Applications of Absorption Spectroscopy of Organic Compounds," Prentice-Hall, Englewood Cliffs, N. J., 1965, pp 116–117.

[18] The most obvious differences were the J_{AX}–J_{MX} values of 6.3 and 1.7 Hz, respectively, and the chemical shift differences (δ_{AM}) of 0.49 and 0.00 ppm, respectively. Both of these observations are consonant with a favored conformation in the monomethyl ether.

[19] There is a substantial upfield shift of H_X (0.42 ppm) of the monomethyl ether relative to diol **7** which does not appear to be a solvent effect. When DMSO is used the nmr spectrum of the monomethyl ether exhibits a singlet hydroxyl proton, indicating the presence of a tertiary hydroxyl group. See O. L. Chapman and R. W. King, *J. Amer. Chem. Soc.*, **86**, 1256 (1964). Significantly, diol **7** gives rise to two different hydroxyl protons in DMSO, one showing doublet coupling to the carbinyl proton and the other showing no coupling.

to trityllithium (10.2 mmol) in 10 ml of THF and the mixture was allowed to stir overnight. Acidification, extraction of the organic layer followed by chromatographic separation on alumina, and subsequent recrystallization furnished a low yield (10.9 mg) of the analytical sample: mp 201–205°; ir (CS_2) 3481 (broad OH), 3095, 3068, 3040, 2978, 2936, 1070, 859, 769, 760, and 743 cm^{-1}; nmr (CS_2) δ 5.3 (m, 1, J = 10.0, 5.0 Hz, H_X), 2.7 (s, 1, J = 14.7 Hz, H_M), 1.7 (m, 1, J = 14.7, 10.0 Hz, H_A).

Anal. Calcd for $C_{40}H_{34}O_2$: C, 87.88; H, 6.27. Found: C, 88.48; H, 6.56.

Diol 1 from Decomposition of 2-Methyltetrahydrofuran.—Trityllithium was prepared from 3.5 g of triphenylmethane in freshly distilled 2-methyltetrahydrofuran and a stock solution of *n*-butyllithium (Foote Mineral Co.) at ice temperatures. A solution of 2.3 g of benzophenone in 10 ml of 2-methyltetrahydrofuran was slowly added after the reaction vessel had warmed to room temperature. The dark blue-green solution was stirred for 9 hr and was then hydrolyzed with 3 *M* hydrochloric acid. Extraction of the organic layer followed by crystallization furnished a solid sample; mixture melting point and infrared data indicated that this material was diol 1, 0.04 g (0.6%), mp 196–198°.

1,1-Diphenyl-4,4,4-trichloro-1,3-butanediol (7).—A mixture of 5.5 g (27 mmol) of racemic 4,4,4-trichloro-3-hydroxybutyric acid (mp 118–118.5°), 50 ml of methanol, and 0.5 ml of boron trifluoride etherate was refluxed for several hours. The solvent was distilled to furnish 5.7 g (97%) of off-white, glistening crystals. Recrystallization produced the colorless ester: mp 63–63.5° (lit.[20] mp 62–63°); the nmr spectrum showed the presence of CH_3, OH, CH_2, and CH absorptions with an AMX pattern for the methylene and carbinyl protons. A solution of 2.2 g (10 mmol) of the above ester in 10 ml of diethyl ether was added to phenylmagnesium bromide (41 mmol). The mixture was refluxed on a steam bath for 30 min. The organic layer yielded 1.2 g (35%) of diol 7, mp 174–175° (lit.[21] mp 178.5°).

Anal. Calcd for $C_{16}H_{15}Cl_3O_2$: C, 55.60; H, 4.37. Found: C, 55.41; H, 4.26.

The mono- and dimethyl ethers were prepared by refluxing diol 7 with a mixture of calcium sulfate, methyl iodide, and silver oxide in DMF for 2 days. The nmr spectrum indicated an equal mixture of the monomethyl and dimethyl ethers which was cleanly separated on a silica gel column with chloroform.

Stability of Diol 1 to Heat and Base.—Diol 1 was refluxed in 5 ml of benzene and 90 ml of 5% sodium ethoxide solution in ethanol for 22 hr. Column chromatography yielded 360 mg of triphenylmethane (86%) and 270 mg of benzophenone (41%).

Thermal decomposition was afforded by injecting 1.2 mg of diol 1 into a vapor phase chromatographic column (SE-30) maintained at 300°. Analysis of the chromatogram showed that triphenylmethane, benzophenone, and acetaldehyde account for 40, 32, and 10% of the recorded decomposition products, respectively. The presence of acetaldehyde was confirmed by heating 56 mg of diol 1 at 300° for 25 min in an argon atmosphere and treating the decomposition mixture with 2,4-dinitrophenylhydrazine. The orange derivative was recrystallized from ethanol, furnishing 3.4 mg (13%) of the analytical sample, mp 147–148°.

1,1,2,4,4-Pentaphenyl-1,3-butadiene (3) resulted after refluxing 0.35 g of diol 1 with 0.14 g of iodine for 1 hr in 19 ml of acetic acid. The organic layer yielded yellow prisms, 0.15 g (74%). (Lower yields of hydrocarbon **3** were obtained by dehydrating diol 1 with thionyl chloride or formic acid.) The hydrocarbon was identified by infrared, ultraviolet, and combustion data, which were consistent with literature values:[22] mp 169–170°; uv max (95% EtOH) 341 nm (ϵ 11,430), 244 (17,000).

Anal. Calcd for $C_{34}H_{26}$: C, 93.97; H, 6.03. Found: C, 93.78, 93.82; H 6.25, 6.13.

1,1,1,4,4-Pentaphenyl-4-hydroxy-2-butanone (4).—Diol 1 (172 mg) dissolved in methylene chloride was refluxed with 3 ml of chromium trioxide solution (1 g CrO_3 + 1 ml HOAc + 3 ml H_2O) for 2 hr. Crystallization of the organic layer provided 97 mg (56%) of white prisms: mp 144–145°; ir (CS_2) 3520 (broad OH), 1707 (C=O), 1070, 768, and 594 cm^{-1}; nmr ($CDCl_3$) δ 7.2, 7.1 (m, 25 H, aromatic), 5.3 (s, 1, OH), 3.5 (s, 2, CH_2).

Anal. Calcd for $C_{34}H_{28}O_2$: C, 87.15; H, 6.02. Found: C, 87.32; H, 6.14.

Stability of Keto Alcohol 4 to Heat and Base.—Keto alcohol 4 (11 mg) and 13 mg of sodium hydroxide were placed in a sealed tube in an argon atmosphere and heated to 157° for 1 hr. A carbon disulfide extract exhibited infrared absorption characteristic of a triphenylmethane and benzophenone mixture.

1,1,1,4,4-Pentaphenylbut-3-en-2-one (2).—Diol 1 (109 mg) was dissolved in 5 ml of acetic acid and a solution of 426 mg of sodium dichromate in 6 ml of acetic acid was added. The mixture was refluxed for 2.5 hr. Crystallization of the organic layer yielded a fine yellow powder (63% yield): mp 182–183.5°; ir (CS_2) 1705 (C=O), 1090, 675 cm^{-1}; uv max (95% EtOH) 308 nm (ϵ 8920), 272 (5960).

Anal. Calcd for $C_{34}H_{26}O$: C, 90.63; H, 5.82. Found: C, 91.00; H, 5.85.

Stability of Ketone 2 to Heat and Base.—Ketone 2 (31 mg) decomposed when heated at 200° for 1 hr with 38 mg of sodium hydroxide. The infrared spectrum indicated that the extracted organic layer contained triphenylmethane and benzophenone. Acidification of the basic solution produced 14 mg (82%) of 3,3-diphenylacrylic acid (5), mp 154–158°, compared to known sample.

Dehydration of Keto Alcohol 4.—Keto alcohol 4 (31 mg) was refluxed with 15 mg of iodine in 4 ml of acetic acid for 1 hr. Crystallization of the organic layer furnished 22 mg (75%) of ketone 2, mp 182–183°.

Detection of Ethylene and Butane.—After the addition of triphenylmethane in a typical trityllithium reaction, the gases were collected and qualitative infrared analysis indicated a mixture of butane and ethylene.

Registry No.—**2**, 36976-75-7; **3**, 2639-26-1; **4**, 36994-56-6; **6**, 36976-77-9; tetrahydrofuran, 109-99-9; benzophenone, 119-61-9; trityllithium, 733-904.

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[21] J. S. W. Boyle, A. McKenzie, and W. Mitchell, *Ber.*, **70B**, 2153 (1937).

[22] R. E. Lutz, R. G. Bass, and D. W. Boykin, Jr., *J. Org. Chem.*, **29**, 3660 (1964).