($CHCl_3$) 3600, 3450, 1590, 1575, 990, 925 $cm^{-1}$; nmr ($CDCl_3$) $\delta$ 1.5–2.1 (m, 4 H), 2.59 (s, 3 H), 2.80 (t, 2 H), 4.18 (q, 1 H), 4.39 (s, 1 H), 4.9–5.4 (m, 2 H), 5.65–6.2 (m, 1 H), 6.90 (d, 2 H), 7.38 (t, 1 H); *m/e* 191.

*Anal.* Calcd for $C_{12}H_{17}NO$: C, 75.35; H, 8.96; N, 7.32. Found: C, 74.95; H, 8.66; N, 7.21.

**Preparation of Pyridine Enone 11.** A solution containing 12.0 g (0.063 mol) of allylic alcohol **10** and 120 g of activated $MnO_2$[12] in 300 ml of $CH_2Cl_2$ was refluxed for 5 hr. The mixture was filtered, the precipitate was thoroughly washed with $CHCl_3$, and the combined washings were concentrated. Filtration of the concentrate through 200 g of silica gel using 3:1 hexane–ethyl acetate as the eluent afforded 10.5 g (88%) of the desired vinyl ketone **11** as a pale yellow oil: ir ($CHCl_3$) 1670, 1610, 1590, 1575 $cm^{-1}$; nmr ($CDCl_3$) $\delta$ 1.97 (m, 2 H), 2.47 (s, 3 H), 2.55–3.0 (m, 4 H), 5.66 (d of d, 1 H), 6.21 (m, 2 H), 6.90 (d, 2 H), 7.38 (m, 1 H); *m/e* 189.

*Anal.* Calcd for $C_{12}H_{15}NO$: C, 76.16; H, 7.99; N, 4.65. Found: C, 76.15; H, 7.65; N, 4.50.

**Coupling of Diketone 6 with Vinyl Ketone 11. Formation of Adduct 12.** To a solution containing 1.5 g (0.0118 mol) of 2-methyl-1,3-cyclohexanedione in 50 ml of dry DME (freshly distilled from $CaH_2$) under a nitrogen atmosphere was added 10 mg of NaH. After stirring for 10 min at room temperature, 2.0 g (0.0106 mol) of vinyl ketone **11** in 10 ml of dry DME was slowly added. The resulting solution was heated under reflux for 0.5 hr and cooled to room temperature, and 25 ml of $H_2O$ was added. The mixture was extracted with 3 × 25 ml of $CH_2Cl_2$ and the combined organic extracts were dried over anhydrous $Na_2SO_4$. After removal of the solvents, the residue was chromatographed on 100 g of silica gel. Elution with $CHCl_3$ afforded 3.18 g (95%) of trione **12** as an oil: ir ($CHCl_3$) 1710, 1690, 1590, 1575 $cm^{-1}$; nmr ($CDCl_3$) $\delta$ 1.27 (s, 3 H), 1.8–2.9 (m, 19 H), 6.90 (d, 2 H), 7.38 (t, 1 H); *m/e* 315.

*Anal.* Calcd for $C_{19}H_{25}NO_3$: C, 72.35; H, 7.99; N, 4.44. Found: C, 72.50; H, 8.15; N, 4.50.

**Cyclodehydration of Trione 12. Formation of Octalindione 2.** A solution containing 1.0 g (3.18 mmol) of trione **12**, 565 mg (6.36 mmol) of 3-aminopropionic acid, and 2.5 ml of 1 *N* $HClO_4$ in 25 ml of $CH_3CN$ was heated under reflux for 55 hr. The solution was cooled to room temperature, 25 ml of $H_2O$ was added, and the mixture was neutralized with $NaHCO_3$. This mixture was extracted with 4 × 25 ml of $CH_2Cl_2$, the combined organic extracts were dried, the solvents were evaporated, and the residue was chromatographed on 50 g of silica gel. Elution with $CHCl_3$ afforded 711 mg (75%) of **2** as an oil: ir ($CHCl_3$) 1705, 1665, 1590, 1575 $cm^{-1}$; nmr ($CDCl_3$) $\delta$ 1.38 (s, 3 H), 1.8–3.0 (m, 17 H), 6.90 (t, 2 H), 7.38 (t, 1 H); *m/e* 297.

**Reduction of Picolylethylated Octalindione. Preparation of Hydroxyoctalone 1.** To a solution containing 750 mg (2.5 mmol) of enedione **2** in 25 ml of absolute ethanol under an atmosphere of nitrogen at 0° was added 47.5 mg (1.25 mmol) of $NaBH_4$. The solution was stirred at 0° for 0.5 hr and then at room temperature for 1.5 hr. To the resulting solution was then added 10 ml of saturated KCl solution and the mixture was extracted with 4 × 25 ml of $CHCl_3$. The combined organic extracts were dried over anhydrous $Na_2SO_4$ and, after evaporation of the solvents, the residue was chromatographed on 50 g of silica gel. Elution with $CHCl_3$ afforded 682 mg (91%) of hydroxyenone **1** as white crystals, which were recrystallized from ethyl acetate–hexane: mp 103–104°;[1] ir ($CHCl_3$) 3600, 3400, 1660 1590, 1575 $cm^{-1}$; nmr ($CDCl_3$) $\delta$ 1.07 (s, 3 H), 1.5–2.9 (m, 17 H), 3.24 (t, 1 H), 3.31 (s, 1 H), 6.90 (t, 2 H), 7.38 (t, 1 H); *m/e* 299.

*Anal.* Calcd for $C_{19}H_{25}NO_2$: C, 76.22; H, 8.42; N, 4.68. Found: C, 76.14, H, 8.37; N, 4.52.

**One-Step Condensation of Vinyl Ketone 11 with Diketone 7. Formation of Enedione 14.** A solution containing 50 g (0.0264 mol) of vinyl ketone **11** and 3.88 g (0.0345 mol) of 2-methyl-1,3-cyclopentanedione (**7**) in 100 ml of aqueous 10% $H_2SO_4$ was heated under reflux for 20 hr. The solution was then cooled to room temperature, neutralized with $NaHCO_3$, and extracted with 3 × 25 ml of $CH_2Cl_2$. The combined organic extracts were dried over anhydrous $Na_2SO_4$ and, after evaporation of the solvent, the residue was chromatographed on 250 g of silica gel. Elution with $CHCl_3$ afforded 6.9 g (29%) of **14** as a pale yellow oil:[13] ir ($CHCl_3$) 1740, 1660, 1590, 1575 $cm^{-1}$; nmr ($CDCl_3$) $\delta$ 1.12 (s, 3 H), 1.9–3.0 (m, 15 H), 6.92 (t, 2 H), 7.41 (t, 1 H); *m/e* 283.

*Anal.* Calcd for $C_{18}H_{21}NO_2$: C, 76.30; H, 7.47; N, 4.94. Found: C, 76.21; H, 7.38; N, 4.80.

**Coupling of Diketone 7 with Vinyl Ketone 11. Formation of Adduct 13.** To a solution containing 5.0 g (0.0264 mol) of vinyl ketone **11** and 3.8 g (0.034 mol) of 2-methyl-1,3-cyclopentanedione in 20 ml of ethyl acetate was added 10 ml of 20% $Et_3N$ in ethyl acetate. After stirring at room temperature for 36 hr the resulting solution was added to 15 ml of $H_2O$. The mixture was extracted with 3 × 20 ml of $CHCl_3$ and the combined extracts were dried over anhydrous $Na_2SO_4$. Evaporation of the solvent afforded 7.88 g of crude trione **13**. Examination of the "crude" material by tlc (4:4:1 hexane–ethyl acetate–methanol) showed one spot, $R_f$ 0.43, and the absence of **11**, $R_f$ 0.56. A small spot at the origin appeared to be the only contamination. Trione **13** showed ir ($CHCl_3$) 1785 (shoulder), 1721, 1590, 1575 $cm^{-1}$; nmr ($CDCl_3$) $\delta$ 1.10 (s, 3 H), 1.6–2.2 (m, 4 H), 2.3–2.9 (m, 13 H), 6.90 (d, 2 H), 7.38 (t, 1 H); *m/e* 301.

**Preparation of Enedione 14 from Trione 13.** A solution containing 7.88 g (0.028 mol) of crude trione **13**, 4.45 g (0.050 mol) of 3-aminopropionic acid, and 10.5 ml of 1 *N* $HClO_4$ in 105 ml of $CH_3CN$ was heated under reflux for 55 hr. The resulting solution was cooled to room temperature, 50 ml of $H_2O$ was added, and the solution was neutralized with $NaHCO_3$. The solution was then extracted with 4 × 50 ml of $CH_2Cl_2$ and the combined organic extracts were dried over anhydrous $Na_2SO_4$. Evaporation of the solvents and chromatography on 500 g of silica gel afforded 7.04 g (7) of the enedione **14** after elution with $CHCl_3$.

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**Registry No.**—**1**, 51965-91-4; **2**, 51965-92-5; **6**, 1193-55-1; **7**, 765-69-5; **9**, 46119-04-4; **10**, 51965-93-6; **11**, 51965-94-7; **12**, 51965-95-8; **13**, 51965-96-9; **14**, 51965-97-0; 2,6-lutidine, 108-48-5; 3-chloropropionaldehyde diethyl acetal, 35573-93-4; 3-aminopropionic acid, 107-95-9.

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(11) Melting points and boiling points are uncorrected. Nmr spectra were measured on Varian T-60 or A-60D spectrometers using tetramethylsilane as an internal standard. Mass spectra were obtained on an L.K.B.-9000 system *via* direct insertion. Combustion analysis were performed by Galbraith Laboratories, Knoxville, Tenn. Phenyllithium and vinylmagnesium chloride were obtained from Alpha Inorganics.
(12) H. B. Henbest, E. R. H. Jones, and T. C. Owen, *J. Chem. Soc.*, 4909 (1957).
(13) It is interesting to note that the corresponding reaction, vinyl ketone **11** with $\beta$-diketone **6**, failed for reasons not understood.

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## Phenylation of Perchlorocyclobutenone

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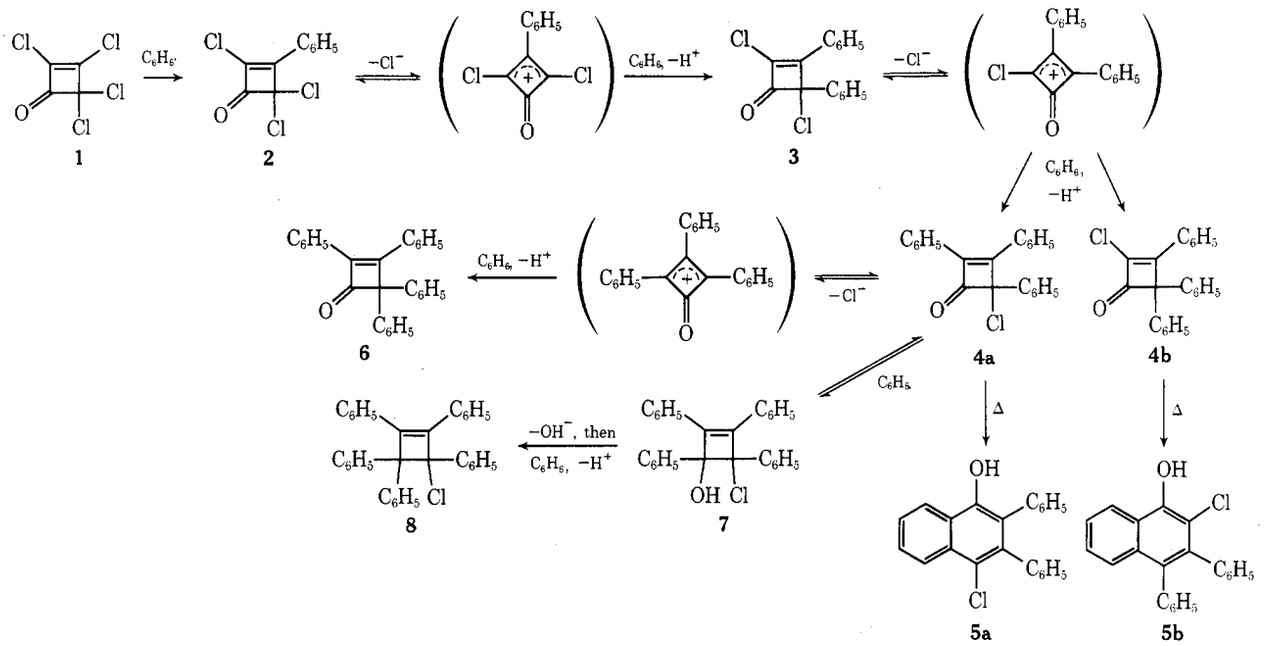
The interaction of perchlorocyclobutenone (1,2,3,3-tetrachlorocyclobutene-4-one, 1) with benzene under Friedel–Crafts conditions was first studied by De Selms, *et al.*,[1] who observed no phenylation in the presence of 1 molar equiv of aluminum chloride. Subsequently, Ried and Lantzsch,[2] employing 3 molar equiv of the same Lewis acid, obtained the two polyphenylated cyclobutene derivatives, 1,2,3,3-tetraphenylcyclobuten-4-one (**6**) and 3-chloro-1,2,3,4,4-pentaphenylcyclobutene (**8**), in respective yields of 25 and 50%.

**Chart I**[a]



[a] Lewis acid participation not shown.

As a study of the arylation of squaryl dichloride in this laboratory[3,4] required the independent synthesis of ketone **6**, we modified Ried's procedure, treating **1** in benzene solution with 4 mol of $AlCl_3$ at 6° (24 hr). Both **6** and **8** were formed, although in different yields (54 and 18%, respectively), under the conditions of our experiment. In addition to the two major products, separation by chromatography furnished in 0.5% yield a chlorodiphenylnaphthol melting at 149–152°, which proved identical with the 1-naphthol derivative obtained in previous work[4] from squaryl dichloride and benzene and assigned[4] one of the two isomeric structures **5** (Chart I). The compound most likely resulted from thermal electrocyclic ring opening of the corresponding chloro ketone precursor **4** and recyclization of the intermediary vinylketene.[5] On a tentative basis, we ascribe structure **4a** to the postulated precursor ketone and, correspondingly, structure **5a** to the separated naphthol with mp 149–152°. This decision is based on the isolation, in a separate, short-time experiment (1 hr at 10°, same reactant ratio), of a chloro ketone **4** (0.6%), for which we consider **4b** the most reasonable assignment on the basis of the 100-MHz nmr [acetone-$d_6$; three multiplets centered at $\delta$ 7.9 (2 H, ortho protons, phenyl group at C-2), 7.55 (3 H, meta and para protons, same group),[6] and 7.37 ppm (10 H, all other protons)], electronic [$\lambda_{max}$ (EtOH) 310 nm[7]], and mass [*m/e* 330 ($P^+$), correct isotopic distribution for one Cl atom] spectra. On thermal treatment (0.5 hr at 130°), the chloro ketone readily converted to a chlorodiphenylnaphthol (mp 145–148°), for which the mode of formation[5] then suggests structure **5b**. Since this compound, although similar in melting and spectral behavior, clearly proved nonidentical with the naphthol (mp 149–152°) obtained in the main experiment, we are left with the assignment of **5a** for the latter compound (and, hence, of **4a** for its hypothetical, nonintercepted chloro ketone precursor), no isomer structures other than **a** and **b** being conceivable in the present reaction scheme. Support for this reasoning is found in the observed indifference of the presumed **4b** toward further phenylation; upon treatment with excess $AlCl_3$ in benzene solution (24 hr at 6°), the chloro ketone was recovered almost quantitatively, and no traces of **6** or **8** were detected in that experiment.[9] Such behavior accords with the chemical inertness of the vinylic Cl atom at C-1, whereas with Cl at C-3, as in **4a**, fast Lewis acid assisted dissociation of $Cl^-$ and subsequent electrophilic attack on benzene (to give **6**), as well as (slower) benzene addition and following substitution at C-4 (to furnish **8** *via* **7**),[10] would be the expected consequence. In fact, the last-named two sequences are almost certainly the paths of formation of both **6** and **8** in the main experiment, although the unavailability of **4a** has so far prevented an experimental verification.

The formation of intermediates **4** may proceed *via* chlorides **2** and **3**. A likely pathway, involving the 1,3-dichloro-4-oxo-2-phenylcyclobutenyl and 1-chloro-4-oxo-2,3-diphenylcyclobutenyl cations, is suggested in Chart I.[11] We were able to isolate the key compound **2** (3%) in the aforementioned short-time experiment. The structure of **2** derives from the 60-MHz nmr [acetone-$d_6$: two signals centered at $\delta$ 7.8 (2 H, ortho phenyl protons) and 7.5 ppm (3 H, remaining phenyl protons)[6]] and electronic [$\lambda_{max}$ (EtOH) 301 nm, in accord with 2-chloro-1-phenylenone chromophore[12]] spectra and is corroborated by mass spectral evidence [*m/e* 246 ($P^+$), correct isotopic distribution for 3 Cl atoms]. The unique lability of the vinylogous chloro group at C-2 in **1**, demonstrated for such related systems as squaryl dichloride,[1,3,4,14] 2-chloro-1-phenylcyclobutene-3,4-dione,[8a] and 2-chloro-1-phenylcyclobuten-4-ones,[13] leaves no doubt as to the primary formation of **2** in the acylation of benzene with **1**. The early-stage interception of the compound and its elusiveness in the 24-hr experiment both lend support to this inference.

The isolation of **4b**, but not of **4a**, in initial stages and the collection of **5a**, but not of **5b**, in final stages of the reaction require discussion. Ionization of **4a** and formation of **6** is probably a fast reaction because of expected high stability of the oxotriphenylcyclobutenyl cation intermediate. Electrocyclic ring opening and cycloaddition to the naphthol **5a** likewise is a low activation energy process, as manifested in the formation of **5a** even at the low reaction temperature (6°) employed. Both factors combine to keep the instantaneous concentration of **4a** below a convenient isolation threshold, even though the compound is probably generated from the precursor cation appreciably faster[15] than is **4b**. Isomer **4b**, on the other hand, cannot readily ionize to a highly stabilized cation, nor is its conversion to **5b**, requiring either fusion (130°) or heating in benzene so-

lution (24 hr at 77°, no isomerization observed after 24 hr at 20°), a rapid process. Its instantaneous concentration, hence, will be higher than that of **4a**, permitting preparative separation. The ease of isomerization of **4a** to **5a** most likely results from relief of steric strain on ring opening in the 1,2-diphenyl derivative; no such (kinetic and thermodynamic) driving force exists for the conversion of **4b** to **5b**. A parallel situation can be found with the two ketones **6** and 1-hydroxy-2,3,3-triphenylcyclobuten-4-one.[3] While the former converts with great ease[5] to the corresponding triphenylnaphthol in boiling benzene, we have been unable to achieve (kinetically and energetically disfavored) naphthol formation from the strongly chelated[3] 1-hydroxy derivative under the same conditions.

## Experimental Section

**Friedel–Crafts Reaction of Perchlorocyclobuten-4-one with Benzene. A. 24 Hr at 6°.** The solution of 0.51 g (2.5 mmol) of **1**[1,16] in benzene (5 ml, predried and distilled from Na) was stirred with 1.35 g (10 mmol) of freshly sublimed $AlCl_3$ for 24 hr at 6° under dry $N_2$. The reaction mixture was shaken with ice-cold 0.1 *M* aqueous hydrochloric acid (25 ml) and benzene (25 ml), and the organic phase was thoroughly washed with water and dried ($Na_2SO_4$). Solvent removal under reduced pressure and chromatography of the residue (silica gel, Merck 7734, fractionation monitored by tlc) produced three fractions (eluents in parentheses), which, after a single recrystallization from hexane, gave crude product yields as stated.

Fraction I (hexane): 3-chloro-1,2,3,4,4-pentaphenylcyclobutene (**8**), 0.21 g (17.9%). Three times recrystallized from hexane, the compound melted at 153–153.5° (lit.[2] mp 161°), undepressed on admixture of authentic[2] **8**.

Fraction II (1:1 hexane–benzene): 4-chloro-2,3-diphenyl-1-naphthol (**5a**), 0.004 g (0.48%), mp 149–152° (no further recrystallization attempted), undepressed on admixture of naphthol derivative (mp 150–153°) from previous work,[4] ir (KBr) 3503 (s), 3480 $cm^{-1}$ (m) ($\nu_{OH}$).

Fraction III (benzene): 1,2,3,3-tetraphenylcyclobuten-4-one (**6**), 0.50 g (53.7%). Twice recrystallized from hexane, the compound had mp 128–129° (lit. mp 139,[2] 129–130° [3]), undepressed on admixture of authentic[3] **6**.

**B. 1 Hr at 10°.** An experiment was set up as under A, but was conducted for 1 hr at 10°. Chromatographic work-up as before yielded four slightly overlapping bands; these were rechromatographed, and corresponding fractions were combined and once recrystallized from hexane to give the product yields stated.

Fraction I (hexane): **8**, 0.003 g (0.26%).

Fraction II (hexane): 1,3,3-trichloro-2-phenylcyclobuten-4-one (**2**), 0.018 g (2.9%). The faintly yellow crystals, once more recrystallized from hexane, had mp 125–126°. *Anal.* Calcd for $C_{10}H_5Cl_3O$ (247.5): C, 48.52; H, 2.04. Found: C, 48.45; H, 1.91. Electronic spectrum $\lambda_{max}$ (EtOH) 301 nm ($\epsilon$ 30,000); ir (KBr) 1798 $cm^{-1}$ (s) ($\nu_{C=O}$); mass spectrum *m/e* 246 ($P^+$ for $^{35}Cl$).

Fraction III (1:1 hexane–benzene): 1-chloro-2,3,3-triphenylcyclobuten-4-one (**4b**), 0.005 g (0.6%), mp 129.5–130.5° (twice recrystallized from hexane). *Anal.* Calcd for $C_{22}H_{15}ClO$ (330.8): C, 79.87; H, 4.58. Found: C, 80.00; H, 4.70. Electronic spectrum $\lambda_{max}$ (EtOH) 310 nm ($\epsilon$ 21,000); ir (KBr) 1777, 1773 $cm^{-1}$ (s) ($\nu_{C=O}$); mass spectrum *m/e* 330 ($P^+$ for $^{35}Cl$).

Fraction IV (benzene): **6**, 0.075 g (8.1%).

**Isomerization of 4b.** A sample (0.002 g) of **4b** recovered from spectroscopic analysis was fused for 0.5 hr at 130° in a capillary tube, and the solidified product, 2-chloro-3,4-diphenyl-1-naphthol (**5b**), was once recrystallized from hexane: mp 145–148° (purity not optimized), depressed on admixture of **5a**; ir (KBr) 3470 $cm^{-1}$ (s) ($\nu_{OH}$), remaining details similar to, but not identical with, those in spectrum of **5a**; mass spectrum *m/e* 330 ($P^+$ for $^{35}Cl$).

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**Registry No.**—**1**, 3200-96-2; **2**, 51965-98-1; **4b**, 51965-99-2; **5a**, 51966-00-8; **5b**, 51966-01-9; benzene, 71-43-2.

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(4) B. R. Green and E. W. Neuse, *Synthesis*, 46 (1974).
(5) An almost quantitative yield of 2,3,4-triphenyl-1-naphthol can be obtained by electrocyclic ring opening of 1,2,3,3-tetraphenylcyclobuten-4-one. This reaction is brought about by heating the ketone in the melt or in benzene solution.
(6) We find (acetone-$d_6$, 60 MHz) the analogous multiplets for ortho and meta plus para protons, respectively, at $\delta$ 8.0 and 7.55 ppm in 1-phenylcyclobutene-3,4-dione[8a,d] and at 8.0 and 7.5 ppm in 1-hydroxy-2-phenylcyclobutene-3,4-dione[8a,c] (samples of both compounds kindly supplied by Professor Ried). A similar pattern has been reported[8d] for 1-hydroxy-2-phenyl-3-alkylcyclobuten-4-ones.
(7) The related 1-chloro-2-phenylcyclobutene-3,4-dione absorbs at 296 and 306 nm (isooctane),[8a] and maxima at 295 and 304 nm are found in the spectrum of 1-chloro-2,3-diphenylcyclobuten-4-one (J. D. Roberts, private communicatons). In contrast, a red shift by 10–15 nm would be expected for the 1,2-diphenylenone chromophore in **4a**. For example, both 1,2-diphenylcyclobutene-3,4-dione[8b] and **6**[2] show $\lambda_{max}$ (EtOH) at 322 nm.
(8) (a) E. J. Smutny, M. C. Caserio, and J. D. Roberts, *J. Amer. Chem. Soc.*, **82**, 1793 (1960); (b) A. T. Blomquist and F. A. LaLancette, *ibid.*, **83**, 1387 (1961); (c) W. Ried, W. Kunkel, and G. Isenbruck, *Chem. Ber.*, **102**, 2688 (1969); (d) W. Ried and H. Kohl, *ibid.*, **104**, 2896 (1971).
(9) The observed stability of **4b** suggests that the compound should have been among the products of the main experiment. The similarity of the $R_f$ values of **4b** and **6** possibly caused the chloro ketone to be "buried" in the large quantities of **6** present in that experiment (54% as against 8% in the 1-hr run) and so simply prevented its detection and isolation.
(10) One might argue that **4b**, although indifferent to phenylation at C-1, could react in an analogous fashion through benzene addition and subsequent substitution at C-4, giving first 1-chloro-4-hydroxy-2,3,3,4-tetraphenylcyclobutene and then 1-chloro-2,3,3,4,4-pentaphenylcyclobutene. We were unable, however, to detect either product in these reactions. It appears that *gem*-diphenyl substitution at C-3 provides sufficient steric hindrance for successful approach of a benzene molecule. The same situation holds for **6** and, going one step farther, for **8**: neither in previous work[2-5] nor in the present investigation were even traces of hexaphenylcyclobutene detected, which would have resulted from further Lewis acid catalyzed reaction with benzene.
(11) Sequences providing alternative pathways to **6** and **8** from **2** and **4a**, almost certainly of higher activation energy, have for reasons of clarity been omitted from Chart I.
(12) The closely related 1,3-dichloro-2-phenylcyclobuten-4-one shows $\lambda_{max}$ (cyclohexane) at 298 nm.[13] (See also ref 7.) For the alternative isomer structure, 1,2,3-trichloro-3-phenylcyclobuten-4-one, on the other hand, absorption at wavelengths below 250 nm can be predicted. Thus, we find **1** to absorb at 234 nm.
(13) M. C. Caserio, H. E. Simmons, Jr., A. E. Johnson, and J. D. Roberts, *J. Amer. Chem. Soc.*, **82**, 3102 (1960).
(14) (a) W. Ried and F. Bätz, *Justus Liebigs Ann. Chem.*, **755**, 32 (1972); (b) E. Neuse and B. Green, *ibid.*, 619 (1973); (c) E. W. Neuse and B. R. Green, *Polymer*, **15**, 339 (1974).
(15) Preferred attack of the precursor cation at benzene is expected to proceed through the C atom bearing the (Lewis acid complexed) Cl substituent.
(16) G. Maahs, *Justus Liebigs Ann. Chem.*, **686**, 55 (1965).

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# Enol Ethers and Monoketals of Biacetyl

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In extension of recent studies[1] on the photochemistry of various $\alpha$-methylene ketones (**1**) we were interested in examining the photochemical behavior of the formally related compounds bearing oxygen directly on the olefinic double bond. These substances (**2**) are alkyl enol ethers of biacetyl, and we were somewhat surprised to learn that such compounds have never been described. The photochemical behavior of these systems ultimately proved disappointing, but we report below an indirect procedure permitting