1630 cm^{-1} (CO); uv max (*i*-PrOH) 233 nm (44,750), 250 (28,750) 257 (28,400), 274 sh (10,500), 308 sh (14,300), 314 (15,000), 342 (10,550), 357 (8550).

Anal. Calcd for $C_{20}H_{22}N_4O$: C, 71.83; H, 6.63; N, 16.75. Found: C, 71.93; H, 6.87; N, 16.85.

The acetonitrile mother liquors were combined and evaporated to dryness. Trituration of the residue with ether afforded a light brown amorphous solid, which on recrystallizations from acetonitrile gave 2.40 g (36%) of **12a** as colorless prisms, mp 122–125°. This material was found to be identical with **12a** obtained above by tlc and comparison of infrared spectra.

The separation of **11** and **12a** were aided by tlc analyses. On silica gel plates developed in a mixture (1:1) of ethanol and ethyl acetate, **11** appeared at R_f 0.22 and **12a** at R_f 0.08.

Acknowledgment.—We thank Dr. R. P. W. Scott and his staff in our Physical Chemistry Department, in particular, Dr. F. Scheidl for elemental analyses, Dr. V. Toome for uv measurements, Mr. S. Traiman for ir spectra, and Dr. T. Williams for nmr spectra.

Registry No.—**6a**, 41526-19-6; **6b**, 1694-64-0; **7a**, 41895-15-2; **7b**, 41895-16-3; **8a**, 41895-17-4; **8b**, 41895-18-5; **11**, 41895-19-6; **12a**, 41895-20-9; **12b**, 41895-21-0; **13a** dihydrochloride, 41895-22-1; **13b** dihydrochloride, 41895-23-2; 2-(2-azidoacetamido-5-bromobenzoyl)pyridine, 41895-24-3.

Reaction of Polyarylated Carbinols. IV. Reactions of 1,2,3,4,5-Pentaphenyl-2,4-cyclopentadien-1-ol with Sodium Amide. Effect of Quenching Temperature on the Products Obtained

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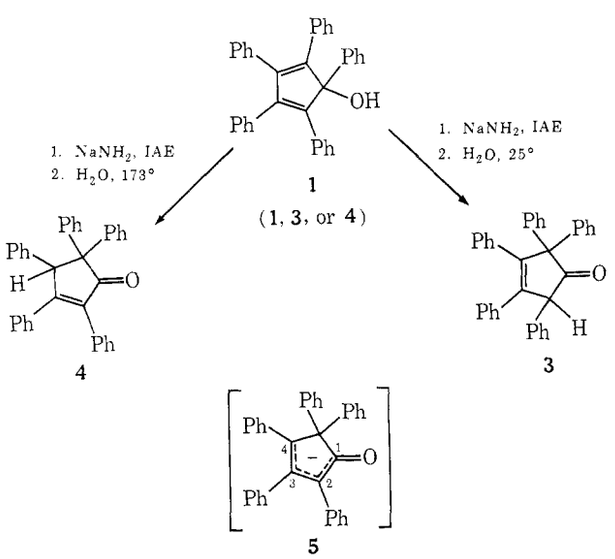
The reaction of 1,2,3,4,5-pentaphenyl-2,4-cyclopentadien-1-ol (1) with catalytic and equimolar amounts of sodium amide has been observed and its mechanism investigated. With catalytic amounts of sodium amide the reaction of **1** has been observed to occur *via* the same mechanism previously reported with other bases. With equimolar amounts of sodium amide 1,2,3,4,5-pentaphenyl-2,4-cyclopentadien-1-ol (1), 2,2,3,4,5-pentaphenyl-3-cyclopenten-1-one (**3**), and 2,3,4,5,5-pentaphenyl-2-cyclopenten-1-one (**4**) are all observed to produce exclusively **3**, the kinetically controlled product, if quenched with water at room temperature, and **4**, the thermodynamically controlled product, if quenched with water at 173°. A mechanism for production of these products involving initial formation of **3** in each case is proposed. Reaction of the anion formed when **1** is treated with equimolar amounts of sodium amide and quenched with benzoyl and benzyl chloride at both room temperature and at 173° is also discussed.

During our continuing study[1-3] of reactions of polyarylated carbinols we have observed[3] that heating 1,2,3,4,5-pentaphenyl-2,4-cyclopentadien-1-ol (**1**)[4,5] to 173° in isoamyl ether (IAE) in the presence of bases such as sodium hydroxide afforded a mixture of isomeric kinetically and thermodynamically controlled ketones, 2,2,3,4,5-pentaphenyl-3-cyclopenten-1-one (**3**)[6,7] and 2,3,4,5,5-pentaphenyl-2-cyclopenten-1-one (**4**),[6] respectively.

We now wish to report the results of this rearrangement when it is performed in the presence of sodium amide and offer some mechanistic explanation for the differences observed.

Treatment of dienol **1**, ketone **3**, or ketone **4** at 173° in IAE with 1 molar equiv of sodium amide followed by cooling of the anion solution to room temperature and quenching with water produces exclusively ketone **3**, the kinetically controlled product. However, if the anion solution is prepared in exactly the same manner from either dienol **1**, ketone **3**, or ketone **4**, but is quenched at 173° with water, ketone **4**, the thermodynamically controlled product, is exclusively produced.

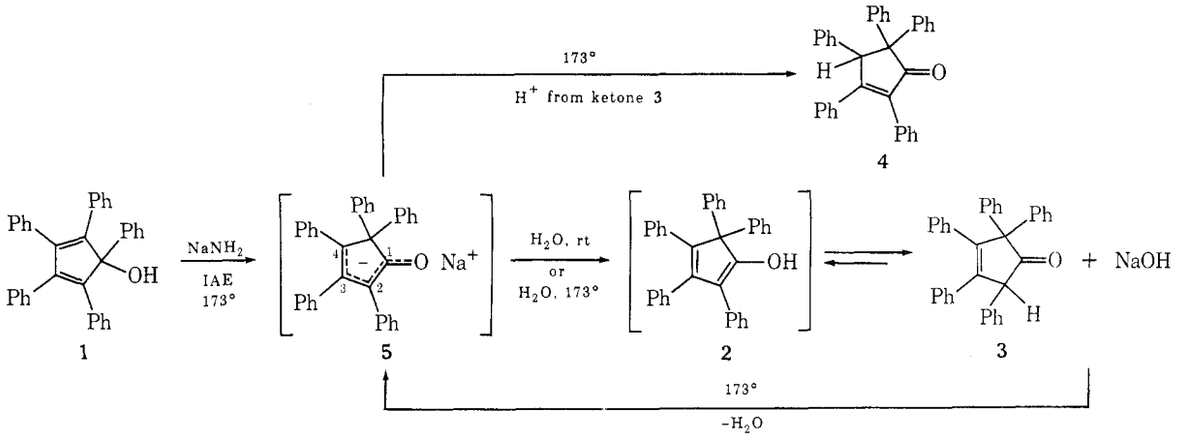
While these results with molar equivalents of sodium amide are different from the results obtained with sodium hydroxide,[3] the results obtained (Table I, Experimental Section) when catalytic amounts of sodium amide are used as base (molar ratio of 10 dienol **1**:1 $NaNH_2$) are identical with those obtained in the reaction of the dienol **1** in IAE with sodium hydroxide as the base.[3] Thus in the reaction of dienol **1** with catalytic amounts of sodium amide, the products formed are obtained by internal quenching and *via* the same mechanism previously described[3] for the sodium hydroxide catalyzed reaction.



This mechanism does not, however, apply in the case where equimolar amounts of sodium amide are employed. Since this quantity of base ensures complete

(1) A. K. Youssef and M. A. Ogliaruso, *J. Org. Chem.*, **37**, 2601 (1972).
(2) A. K. Youssef and M. A. Ogliaruso, *J. Org. Chem.*, **38**, 487 (1973).
(3) A. K. Youssef and M. A. Ogliaruso, *J. Org. Chem.*, **38**, 2023 (1973).
(4) K. Ziegler and B. Schnell, *Justus Liebigs Ann. Chem.*, **445**, 266 (1925).
(5) C. F. H. Allen and J. A. VanAllan, *J. Amer. Chem. Soc.*, **65**, 1384 (1943).
(6) C. Dufraisse, G. Rio, and A. Ranjon, *C. R. Acad. Sci.*, **253**, 2441 (1961).
(7) R. Breslow and H. W. Chang, *J. Amer. Chem. Soc.*, **83**, 3727 (1961).

SCHEME I



conversion of dienol **1** to the intermediate anion **5** and the proton sources, dienol **1** and ketone **3**, available for internal quenching in the case of the sodium hydroxide catalyzed reaction are no longer available in the reaction mixture, the proton source must be the water externally added. In this reaction the quenching temperature controls the site of protonation, either at the oxygen atom, at carbon 2, or at carbon 4, and the results may be explained on the basis of thermodynamically or kinetically controlled product formation.

Reaction of dienol **1** at 173° in IAE with an equimolar amount of sodium amide, cooling the solution containing anion **5** to 120°, and adding water dropwise at this temperature produced ketones **3** and **4** in a 4:6 ratio, respectively. These results agree with expectation that the products formed depended upon thermodynamic and kinetic control.

To establish if the kinetically controlled product, ketone **3**, was the first product formed when the quench was performed at 173° and then rearranged to the thermodynamically controlled product, ketone **4**, under the conditions of the reaction, the following reaction was performed. Dienol **1** was completely converted to anion **5** with molar amounts of sodium amide in IAE at 173° and the reaction was quenched by the addition of water. Immediately upon completion of the water addition a sample was removed and analyzed. The only product shown to be present in this sample by both ir and glpc was ketone **3**. Removal of a second sample from the same reaction mixture after 15 min showed ketone **4** to be the only product present. These observations indicate that the kinetically controlled product, ketone **3**, is formed first and that it is converted to the thermodynamically controlled product, ketone **4**, under the conditions of the reaction.

Attempts to rearrange **3** to **4** thermally were unsuccessful. However, since **3** is converted quantitatively to **4** if the same reaction is performed in the presence of base,[3] then the sodium hydroxide formed when anion **5** is quenched must be catalyzing the rearrangement of **3** to **4** at 173° in IAE completely and quantitatively *within* 15 min. The speed of this isomerization was established independently using a pure sample of **3**.

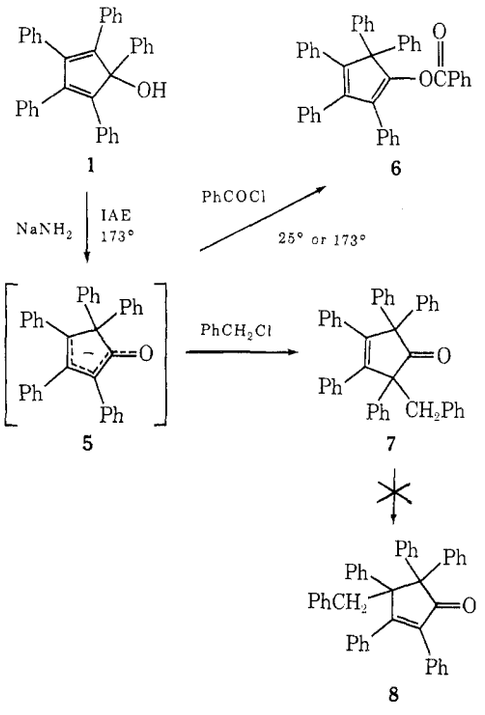
The mechanism proposed for the reaction of dienol **1** with equimolar amounts of sodium amide is illustrated in Scheme I and involves complete conversion of **1** to anion **5**, which upon quenching with water at 173° produces initially the kinetically controlled product, ketone **3**, which then quantitatively rearranges within 15 min to the thermodynamically controlled product, ketone **4**, upon standing in IAE at 173° in the presence of the sodium hydroxide formed. The sodium hydroxide reacts with small amounts of **3** to produce anion **5**, which is then quenched with unreacted **3** to produce **4**. The mechanism proposed for the production of ketone **3** when anion **5** is quenched with water at room temperature involves the same sequence as described above, except that **3** once formed does not undergo rearrangement to **4**. Even though the sodium hydroxide is still produced in this quench, the temperature is not sufficient to cause extensive reaction of the sodium hydroxide with ketone **3** to produce anion **5**.

Additional evidence in support of this proposed mechanism is obtained by quenching anion **5**, formed from dienol **1** at 173° in IAE and an excess of sodium amide, with benzoyl chloride. Using benzoyl chloride as the quenching agent and performing the addition at room temperature should result in the production of the kinetically controlled product, which should resemble structurally ketone **3**. However, since the benzoyl group is a good agent for O- *vs.* C-acylation[8] and since in anion **5** the electron density[3] is greater on oxygen than on C_2, it is not surprising that the major product (71%) isolated from this reaction using benzoyl chloride as the quenching agent at room temperature is 1-benzoyloxy-2,3,4,5,5-pentaphenyl-1,3-cyclopentadiene (**6**), the same product which is formed in 70% yield when anion **5** is quenched with benzoyl chloride at 173°.

These results are in direct contrast to the results reported by Dufraisse, *et al.*,[9] who quenched anion **5** with benzyl chloride and bromide. The product which they obtained was 5-benzyl-2,2,3,4,5-pentaphenyl-3-cyclopentadien-1-one (**7**). We have repeated their experiment quenching anion **5** with benzyl chloride at room temperature and at 173° and have obtained good yields (83–92%) of **7**. It is clear that in this case the absence of an allylic hydrogen prevented reaction of **7** with the sodium hydroxide formed and thus prevented **7**, which resembles the kinetically controlled product ketone **3**, from equilibrating to **8**, which resembles the thermodynamically controlled product ketone **4**. Product **7**

(8) H. O. House, "Modern Synthetic Reactions," 2nd ed, W. A. Benjamin, Menlo Park, Calif., 1972, pp 526–529, 763–765.

(9) C. Dufraisse, G. Rio, and A. Liberles, *C. R. Acad. Sci.*, **256**, 1873 (1963).



can be justified on the basis of the C- *vs.* O-alkylating selectivity of the benzyl group.[8]

From these results it appears that the site of acylation and/or alkylation is governed by both the electron density distribution in anion **5** and, to a greater extent, by the C *vs.* O selectivity of the acylating and/or alkylating agent. Reactions of anion **5** with other acylating and alkylating agents at various temperatures are currently under investigation.

Experimental Section

General.—The glpc analysis of samples was performed on a Bendix Model 2600 gas chromatograph and a Bendix Model 1200 recorder. The glpc was equipped with a 3 ft × 0.25 in. column packed with 3% QF-1 on Chromosorb W (H. P., mesh 100–120) support. Operating conditions were as follows: temperature of inlet 210°; detector 255°; injector 255°; column 210°; and a He carrier gas flow rate of 80 ml/min. Retention times of the dienol **1** was 6.25 min, of ketone **3** 13.75 min, and of ketone **4** 15.75 min. Analysis of the peak areas observed was determined by triangulation.[10] Elemental analyses were performed on a departmental F & M Model 185 C, H, and N analyzer.

Reaction of 1,2,3,4,5-Pentaphenyl-2,4-cyclopentadien-1-ol (1) with Equimolar Sodium Amide. I. Quenching with Water at 173°.—Into a 250-ml three-necked round-bottomed flask equipped with a reflux condenser, a magnetic stirrer, a serum cap, and a nitrogen inlet tube was placed 1.0 g (2.16 mmol) of 1,2,3,4,-5-pentaphenyl-2,4-cyclopentadien-1-ol (**1**)[1,3,4] and 50 ml of freshly distilled isoamyl ether (IAE) and the mixture was heated to reflux (173°). At this temperature 84.3 mg (2.16 mmol) of sodium amide was added very cautiously all at once. A vigorous reaction occurred immediately, affording a deep red-orange solution which was allowed to reflux for 15 min. After this time 10 ml of water was added all at once *via* syringe to the reaction mixture, which was then retained at 173° for an additional 30 min. At this point a 5-ml sample was removed for glpc analysis and the rest of the solution was cooled to room temperature. Analysis of the sample removed by glpc using the instrument and conditions described in the general section above showed only one product to be present, 2,3,4,5,5-pentaphenyl-2-cyclopenten-1-one (**4**). The remaining solution, now at room temperature, was poured into 100 ml of water, and the organic layer was separated, washed several times with water, and dried over anhydrous magnesium sulfate. The solvent was removed under vacuum to afford a viscous yellow oil which was crystallized from a mixture of benzene–petroleum ether (bp 30–60°) to give 992 mg (2.14 mmol, 99.1%) of pale yellow crystals of 2,3,4,5,5-pentaphenyl-2-cyclopenten-1-one (**4**), mp 169–170° (lit.[1,3,6] mp 169–170°). The ir,[6] uv,[6] and nmr[1] spectral data for this compound agreed with the literature.

II. Quenching with Water at Room Temperature.—The above experiment was repeated as described to the point of allowing the anion solution to reflux for 15 min. At this point the entire reaction mixture was then cooled to room temperature under nitrogen. When the solution attained room temperature, 10 ml of water was added at once *via* syringe to the stirred solution and immediately a 5-ml sample was removed for glpc analysis. Analysis of the sample removed by glpc using the instrument and conditions described in the general section above showed only one product to be present, 2,2,3,4,5-pentaphenyl-3-cyclopenten-1-one (**3**). The remaining solution was worked up as reported above to give 996 mg (2.15 mmol, 99.5%) of white crystals of **3**, mp 194.5–196° (lit. mp 194–195°,[6,7] 194.5–196°[1]). The ir,[1,6,7] uv,[6,7] and nmr[7] for this compound agreed with the literature.

Reaction of 2,2,3,4,5-Pentaphenyl-3-cyclopenten-1-one (3) with Equimolar Sodium Amide. III. Quenching with Water at 173°.—This experiment was performed as described above in I to the point of analyzing the sample removed by glpc, except that 1.0 g (2.16 mmol) of 2,2,3,4,5-pentaphenyl-3-cyclopenten-1-one (**3**) was used. The sample removed was subjected to glpc analysis using the instrument and conditions described in the general section and only one product, 2,3,4,5,5-pentaphenyl-2-cyclopenten-1-one (**4**), was shown to be present. The remaining solution, cooled to room temperature, was poured into 100 ml of water and worked up as described in I above to afford 996 mg (2.15 mmol, 99.5%) of white crystals of **4**, melting point and spectral data the same as those described in I above.

IV. Quenching with Water at Room Temperature.—This experiment was performed as described in II above, except that 2,2,3,4,5-pentaphenyl-3-cyclopenten-1-one (**3**) was used. The results were the same as reported above in II.

Reaction of 2,3,4,5,5-Pentaphenyl-2-cyclopenten-1-one (4) with Equimolar Sodium Amide. V. Quenching with Water at 173°.—This experiment was performed as described in I above except that 1.0 g (2.16 mmol) of 2,3,4,5,5-pentaphenyl-2-cyclopenten-1-one (**4**) was used. Glpc analysis of the sample removed again showed **4** to be the only product present. Work-up of the remaining solution afforded 994 mg (2.14 mmol, 99.1%) of **4** with melting point and spectral data the same as reported in I above.

VI. Quenching with Water at Room Temperature.—This experiment was performed as described in II above except that 2,3,4,5,5-pentaphenyl-2-cyclopenten-1-one (**4**) was used. The results were the same as reported above in II.

Reaction of 1,2,3,4,5-Pentaphenyl-2,4-cyclopentadien-1-ol (1) with Catalytic Sodium Amide.—Into a 100-ml, three-necked, round-bottomed flask equipped with a reflux condenser, a magnetic stirrer, a serum cap, and a nitrogen inlet tube was placed 50 ml of isoamyl ether (IAE) which was heated to 173°. At this point a mixture of 8.0 mg (0.2 mmol) of sodium amide and 1.0 g (2.16 mmol) of dienol **1** was added all at once. Samples of 1 ml each were taken at various times by inserting a hypodermic syringe through the serum cap. The samples thus removed were placed in separate containers and cooled by means of an ice–water bath. After all the required samples were collected, glpc analysis was carried out using the instrument and conditions described in the general section. Table I reports the per cent composition obtained from the peak areas and these percentages are plotted on the same graph *vs.* time. Qualitative ir analysis of each sample was also performed and for the sample taken after 13 min only two products were observed to be present, the dienol **1** with a hydroxyl peak at 3500 cm^{-1} and ketone **3** with a carbonyl peak at 1760 cm^{-1}. Analysis of all samples taken after 53 min and up to 533 min showed three distinct products to be present, dienol **1** (hydroxyl peak at 3500 cm^{-1}), ketone **3** (carbonyl peak at 1760 cm^{-1}), and ketone **4** (carbonyl peak at 1720 cm^{-1}). Fractional crystallization techniques using varying mixtures of benzene–petroleum ether allowed separation and isolation of both ketone **3** and ketone **4** from each of these intermediate samples. Analysis of samples taken after 533 min showed only one peak to be present in both ir and glpc corresponding to ketone **4**.

Reaction of 1,2,3,4,5-Pentaphenyl-2,4-cyclopentadien-1-ol (1) with Equimolar Sodium Amide. VII. Quenching with Water at

(10) As described in H. M. McNair and E. J. Bonelli, "Basic Gas Chromatography," Varian Associates, Palo Alto, Calif., 1969, p 154.

TABLE I

ISOMERIZATION REACTION OF 1,2,3,4,5-PENTAPHENYL-2,4-CYCLOPENTADIEN-1-OL IN ISOAMYL ETHER WITH CATALYTIC AMOUNTS OF SODIUM AMIDE

Reaction time, min	Ratio, %		
	Dienol 1	Ketone 3	Ketone 4
0	100	0	0
13	87.8	12.2	0
53	77.8	18.4	3.8
113	69.8	21.5	8.7
143	61.0	24.3	14.7
173	56.7	25.7	17.6
203	50.4	24.6	25.0
233	46.5	23.3	30.2
263	27.7	22.2	40.1
323	30.2	15.8	53.9
383	23.5	12.0	64.5
443	14.6	9.4	76.0
503	8.6	4.7	86.7
533	7.0	3.6	89.4

120°.—This experiment was performed with the same amounts of starting material and in the same manner as described in I above to the point of allowing the anion solution to reflux for 15 min. At this point the entire reaction mixture was cooled to 120° and 10 ml of water was added all at once *via* syringe to the reaction mixture, which was then retained at 120° for an additional 30 min. A 5-ml sample was then removed for glpc and ir analysis and the rest of the solution was cooled to room temperature. Analysis by ir of the sample removed showed two strong peaks, one for ketone **3** (carbonyl at 1760 cm^{-1}) and one for ketone **4** (carbonyl at 1720 cm^{-1}), while glpc analysis of the same sample further established **3** and **4** as the only products present. The remaining solution, now at room temperature, was worked up as described above in I and the residue was subjected to fractional crystallization using varying mixtures of benzene–petroleum ether which allowed separation and isolation of 0.42 g (0.9 mmol, 41%) of ketone **3** and 0.55 g (1.2 mmol, 58%) of ketone **4**. The melting points and spectral data for these compounds agreed with the literature values.

VIII. Quenching with Water at 90°.—This experiment was performed in the same manner as described in VII above except that the reaction mixture was cooled to 90° before being quenched with water. Analysis by ir of a sample removed showed one strong peak for ketone **3** (carbonyl at 1760 cm^{-1}) and one weak peak for ketone **4** (carbonyl at 1720 cm^{-1}), while glpc analysis of the same sample also showed both ketones as the only products present. Work-up of the remaining solution as described above afforded 0.83 g (1.8 mmol, 83%) of ketone **3** and 0.17 g (0.4 mmol, 17%) of ketone **4**.

Establishment of Ketone 3 as the Initial Product Formed When the Solution Is Quenched at 173°.—This experiment was performed using the same amounts of starting material and in the same manner as described in I above to the point of allowing the anion solution to reflux for 15 min. At this point a syringe containing 10 ml of water equipped with a needle long enough to extend below the level of the solution in the flask was inserted through the serum cap. On the down stroke of the plunger the 10 ml of water was added all at once to the reaction mixture and immediately a 5-ml sample was removed from the solution by an up stroke of the plunger. Glpc and ir analysis of this sample showed only ketone **3** to be present. A second sample removed from the solution, still at 173°, after 15 min subjected to ir and glpc analysis showed only ketone **4** to be present. Repeating this experiment several times always gave the same results.

Sodium Hydroxide Catalyzed Isomerization of Ketone 3 to Ketone 4.—Into a 100-ml, three-necked, round-bottomed flask equipped with a reflux condenser, a magnetic stirrer, and a serum cap was placed 50 ml of IAE which was heated to reflux (173°). At this point a concentrated aqueous solution of sodium hydroxide (85 mg of sodium hydroxide in 2 ml of water) was syringed into the boiling solvent. After the initial spattering subsided, small particles of base were observed to precipitate from the solvent. At this point 1.0 g (2.16 mmol) of 2,2,3,4,5-pentaphenyl-3-cyclopenten-1-one (**3**) was added as a solid all at once to the refluxing base mixture. Samples removed by syringe at 5-min intervals after the addition of **3** was complete showed by ir and glpc that **3** had been quantitatively converted to **4** within 15 min.

Attempted Thermal Rearrangement of Ketone 4.—Into a 100-ml, three-necked, round-bottomed flask equipped with a reflux condenser, a magnetic stirrer, and a serum cap was placed 1.0 g (2.16 mmol) of 2,2,3,4,5-pentaphenyl-3-cyclopenten-1-one (**3**) and 50 ml of freshly distilled IAE and the mixture was heated to reflux (173°). In 15-min intervals at the beginning of the reaction, and at 30-min intervals after 2 hr, over an 8-hr period, samples were removed and subjected to ir and glpc analysis. The only product shown to be present in every sample by both ir and glpc was ketone **3**.

Preparation of 1-Benzoyloxy-2,3,4,5,5-pentaphenyl-1,3-cyclopentadiene (6). Quenching Anion 5 with Benzoyl Chloride. IX. At 173°.—Into a 100-ml, three-necked, round-bottomed flask equipped with a reflux condenser, a nitrogen inlet tube, a magnetic stirrer, and a dropping funnel were placed 25 ml of freshly distilled IAE and 462 mg (1.0 mmol) of 1,2,3,4,5-pentaphenyl-2,4-cyclopentadien-1-ol (**1**) and the solution was heated to reflux (173°). At this point 39 mg (1.0 mmol) of sodium amide was added and the solution was allowed to reflux for 15 min. To this red-orange solution of anion **5** was added dropwise 140 mg (1.0 mmol) of freshly distilled benzoyl chloride, and the color of the anion solution was observed to completely discharge as the addition of the benzoyl chloride was completed (5 min). The reaction mixture was allowed to reflux for an additional 30 min and cooled to room temperature with stirring, and the solvent was removed under vacuum on the rotoevaporator. This afforded a viscous yellow oil which was crystallized from 95% ethanol to give 390 mg (0.7 mmol, 70%) of white crystals, mp 186–187°, ir (CCl_4) 1755 (carbonyl), 1175 cm^{-1} (ester).

Anal. Calcd for $C_{42}H_{30}O_2$: C, 89.02; H, 5.34; mol wt, 566. Found: C, 88.88; H, 5.68; mol wt, 566 (mass spectrum).

X. At Room Temperature.—This experiment was performed in the same manner as described in IX above except that the reaction mixture was cooled to room temperature with stirring under nitrogen before the benzoyl chloride was added. Removal of the solvent under vacuum on the rotoevaporator afforded a viscous yellow oil which was crystallized from 95% ethanol to yield 400 mg (0.705 mmol, 70.5%) of white, crystalline 1-benzoyloxy-2,3,4,5,5-pentaphenyl-1,3-cyclopentadiene (**6**), melting point, spectral data, and analysis the same as reported above. Concentration of the mother liquor from the crystallization afforded 115 mg (0.248 mmol, 24.8%) of ketone **3** which probably resulted from reaction of some of anion **5** with atmospheric moisture.

Preparation of 5-Benzyl-2,2,3,4,5-pentaphenyl-3-cyclopenten-1-one (7). Quenching Anion 5 with Benzyl Chloride. XI. At 173°.—This experiment was performed in the same manner as described in IX above using 1.0 g (2.16 mmol) of **1**, 30 ml of IAE, and 84.3 mg (2.16 mmol) of sodium amide. The refluxing anion solution was quenched with 5 ml of freshly distilled benzyl chloride added dropwise and the resulting solution was allowed to reflux for an additional 1 hr. At this point the solution was cooled to room temperature and poured into 100 ml of water, and the organic layer was separated, dried over magnesium sulfate, and concentrated under vacuum on the rotoevaporator. The resulting viscous yellow oil was crystallized from 95% ethanol to give 1.0 g (1.8 mmol, 83%) of white, crystalline solid, mp 196–197° (lit.[9] mp 197–198°).

XII. At Room Temperature.—This experiment was performed in the same manner as described in XI above except that the reaction mixture was cooled to room temperature with stirring under nitrogen before the benzyl chloride was added. Work-up and crystallization as described above afforded 1.1 g (1.99 mmol, 92%) of **7**.

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Registry No.—**1**, 2137-74-8; **3**, 34759-47-2; **4**, 34759-48-3; **6**, 42116-83-6; **7**, 42116-84-7; sodium amide, 7782-92-5.