Chemistry of Trityllithium. Condensation with Benzophenone in Tetrahydropyran

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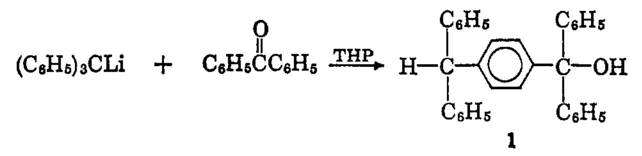
Methods for the preparation of trityllithium are described and compared; yields to 100% have been realized. Trityllithium and benzophenone react in tetrahydropyran to furnish the *para*-condensation product *p*-(diphenylmethyl)diphenylhydroxymethylbenzene (1); no normal addition is observed. Evidence for the instability of the pentaphenylethoxide ion is presented, based on the reactions of phenyllithium with benzopinacolone and methyl triphenylacetate; in both cases cleavage involving elimination of the trityl group is observed and triphenylmethane and triphenylcarbinol are usually obtained.

This study of the preparation and reactions of trityllithium is an extension of earlier investigations with the reagent.[2] Trityllithium has been employed as a proton-abstracting reagent, as in the preparation of enolates.[3] Its utility for this purpose is due to the difficulty with which the reagent participates in addition reactions.

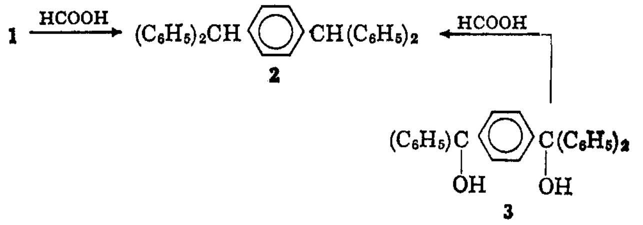
In our earlier work, preparation of trityllithium by direct reaction of trityl chloride with lithium afforded yields of the reagent up to 70% in 1,2-dimethoxyethane (DME) as solvent. Yields were determined by measuring triphenylacetic acid formed following carbonation. The chief disadvantage of this preparative method is the competitive self-condensation of the reagent producing the *p*-(diphenylmethyl)triphenylmethylbenzene anion. We have refined the original technique of metalation of triphenylmethane with butyllithium,[4] making the reaction quantitative using tetrahydrofuran or tetrahydro-2-methylfuran as solvent.

A variety of organolithium reagents can be used to effect the metalation and their rates of reaction with triphenylmethane have been measured.[5] In addition, other methods for the preparation of trityllithium are now known.[6,7] We recommend the butyllithium exchange procedure in preference to the other methods by virtue of the high and reproducible yields, the short reaction periods, and the absence of side products. If the exchange is carried out at room temperature, excess butyllithium is rapidly consumed by reaction with tetrahydrofuran.[5,8] If butyllithium is prepared using lithium in a solvent such as tetrahydrofuran, an excess of lithium should be avoided because of reducing nature of the mixture. We have noted that benzophenone is reduced by the action of lithium in tetrahydrofuran to form both benzohydrol and benzopinacol.[9]

We attempted to condense the bulky trityllithium reagent with several other large molecules. The reaction with benzophenone seemed significant since this ketone has no enolizable hydrogen atoms to react with the reagent.[10] Neither tritylsodium nor the trityl Grignard reagent condenses with benzophenone; with tritylsodium a color change is observed but no products are noted,[11] while the Grignard reagent reduces the ketone to benzopinacol.[12] In the present work using tetrahydropyran as solvent, trityllithium was found to react with benzophenone to form *p*-(diphenylmethyl)diphenylhydroxymethylbenzene (1). No evidence for any other alcohol was found following chromatographic separation of the reaction products.



Condensation of the trityl reagent at the *para* position has been previously noted: self-condensation yields *p*-(diphenylmethyl)triphenylmethylbenzene,[2] and tritylsodium is reported to react with ethyl benzoate in the presence of triphenylaluminum to furnish *p*-(diphenylmethyl)benzophenone.[13] A careful examination of the carbonation product of the trityllithium reagent showed no evidence for the *para*-condensation product, *p*-(diphenylmethyl)benzoic acid, which should be easily detectable by infrared analysis in the presence of triphenylacetic acid. Chemical structure proof for alcohol 1 was obtained by formic acid reduction to the known *p*-bis(diphenylmethyl)benzene, which was also prepared from *p*-bis(diphenylhydroxymethyl)benzene by reduction. Supporting spectral evidence for the proposed structure for alcohol 1 included infrared absorption bands similar to those of triphenylcarbinol and mass spectral data which established the molecular weight and included peaks due to fragmentation on either side of the *para*-substituted ring. This chemical reactivity of a *para* position in trityllithium is consistent with the evidence for high charge on the *para* position of trityllithium. Sandel and Freedman[14] concluded from pmr studies that interelectron repulsion



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[1] National Science Foundation Undergraduate Research Participant.

[2] P. Tomboulian, *J. Org. Chem.*, **24**, 229 (1959).

[3] H. O. House and B. M. Trost, *ibid.*, **30**, 1341 (1965).

[4] H. Gilman and R. V. Young, *ibid.*, **1**, 315 (1936).

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

[6] P. T. Lansbury and R. Thedford, *J. Org. Chem.*, **27**, 2383 (1962); H. Gilman and B. J. Gaj, *ibid.*, **28**, 1725 (1963).

[7] J. J. Eisch and W. C. Kaska, *ibid.*, **27**, 3745 (1962).

[8] After 1 hr at room temperature, 71% of butyllithium initially present has disappeared: H. Gilman and B. J. Gaj, *ibid.*, **22**, 1165 (1957). Trityllithium is remarkably stable in diethyl ether [H. Gilman, A. H. Haubein, and H. Hartzfeld, *ibid.*, **19**, 1034 (1954)], although it is much less stable in tetrahydrofuran.[7]

[9] The anomalous reaction of benzaldehyde with trityllithium in this mixture is probably due to the same property.[2]

[10] *Cf.* reaction with cyclohexanone[2] and with other ketones.[3]

[11] W. Schlenk and E. Bergmann, *Ann.*, **464**, 1 (1928).

[12] W. E. Bachmann, *J. Amer. Chem. Soc.*, **53**, 2758 (1931).

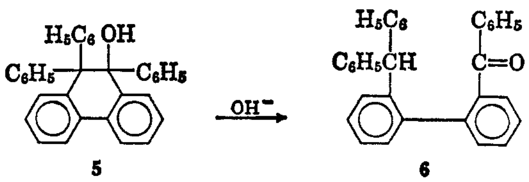
[13] G. Wittig and O. Bub, *Ann.*, **566**, 113 (1950).

[14] V. R. Sandel and H. H. Freeman, *J. Amer. Chem. Soc.*, **85**, 2328 (1963).

causes dispersion of charge to the extremities of the ion resulting in high charges on the *meta* (0.08 unit of charge) and *para* (0.13 unit) positions.

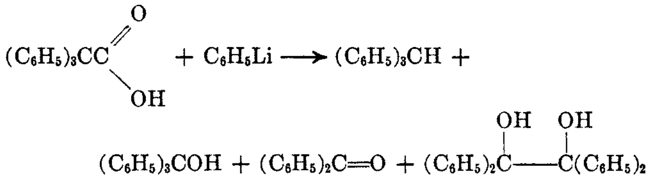
Reaction of trityllithium with benzophenone at a *para* position on one ring is thus strongly favored over reaction at the benzyl position. The latter would result in the sterically crowded pentaphenylethoxide ion; no evidence for an hydrolysis product of this ion was detected. To obtain further evidence for the stability of such an ion, the reaction between phenyllithium and benzopinacolone was examined carefully. Mosher and Huber[15] investigated this system and noted that the main products were triphenylmethane and triphenylcarbinol, with traces of benzophenone. In a number of trials with this reaction, our observations agree with those above. With an excess of phenyllithium, the only products are triphenylmethane and triphenylcarbinol. In the case of inverse addition at −65° with an excess of benzopinacolone, small amounts of triphenylmethane and triphenylcarbinol are obtained, along with the starting materials. No other crystalline products were isolated; no evidence was found for benzophenone or alcohol **1**. Thus, if formed initially, the pentaphenylethoxide ion (**4**) undergoes rapid elimination of the trityl anion. (Elimination possibly occurs by direct displacement without the formation of alkoxide **4**.) Addition of 1 mol more of phenyllithium occurs preferentially with the more reactive ketone, benzophenone, so that no appreciable quantities of benzophenone accumulate in the mixture.

In several similar systems, cleavage reactions have been noted. Instability due to steric crowding has been established in the polyphenylethane series. Pentaphenylethane, although not so reactive as hexaphenylethane, is split by the action of heat, hydrogen iodide, sodium–potassium alloy, and bromine.[16] We verified these observations by examining the action of bromine, N-bromosuccinimide, and chlorine on pentaphenylethane; only cleavage products were obtained. Polyphenylethoxide ions are similarly unstable, especially under basic conditions when steric strain may be relieved by elimination of a stabilized leaving group. Sodium 1,1,2,2-tetraphenylethoxide decomposes to furnish benzophenone and diphenylmethane.[17] The methyl Grignard reagent reacts with benzopinacolone to yield the addition compound acetophenone and the cleavage product triphenylmethane.[18] Similarly, lithium aluminum hydride treatment of benzopinacolone in pyridine results in extensive decomposition, forming triphenylmethane and benzyl alcohol, presumably *via* a 1,2,2,2-tetraphenylethoxide ion intermediate.[19] If the leaving group in such crowded systems is bonded at another site, the tendency for cleavage is decreased and bridged pentaphenylethoxide ions are much more stable. For instance, 9,10,10-triphenyl-9,10-dihydro-9-phenanthrol (**5**) is formed in high yield using phenyllithium.[20] Although inert to phenyllithium, this pentaphenylethanol analog may be decomposed by treatment with potassium hydroxide in ethanol to yield the ketone **6**.[21] Evidence that this is a base-catalyzed reaction was obtained by measuring decomposition rates in Pyrex and quartz tubes. The half-life of alcohol **5** at 275° is 7.5 min in a Pyrex tube and 60 min in a quartz tube.



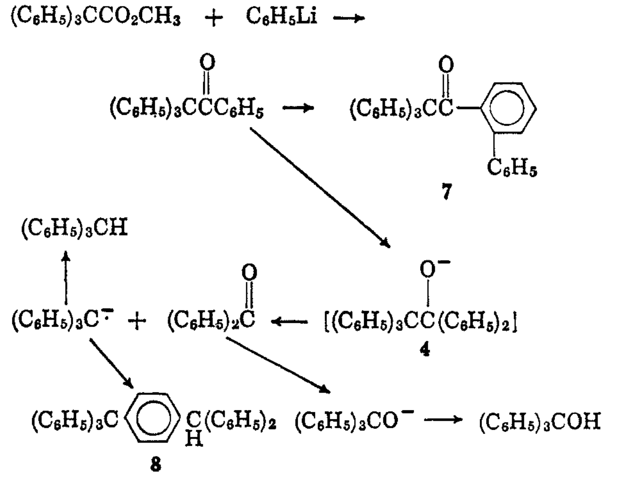
5 **6**

The recent report[22] of the condensation of phenyllithium with triphenylacetic acid or methyl triphenylacetate to form pentaphenylethanol prompted us to investigate this system under a variety of conditions. It is now clear[23] that the compound originally reported as pentaphenylethanol (mp 143–144°) is actually alcohol **1** (mp 141–142°), although we were unable to isolate any alcohol **1** from the reaction. A combination of chromatographic techniques and quantitative infrared methods was employed to analyze the reaction products. The reaction of triphenylacetic acid with excess phenyllithium yields mainly triphenylcarbinol



$$(C_6H_5)_3CCOOH + C_6H_5Li \longrightarrow (C_6H_5)_3CH + (C_6H_5)_3COH + (C_6H_5)_2C{=}O + (C_6H_5)_2\overset{OH}{C}-\overset{OH}{C}(C_6H_5)_2$$

and triphenylmethane, with some benzophenone and benzopinacol. Because of variations in the yields of the products obtained, the reaction of methyl triphenylacetate with phenyllithium was also investigated. Once again the results were in accord with predictions based on the reaction of phenyllithium with benzopinacolone, except that some conjugate addition and self-condensation were observed. With excess phenyl-



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[15] W. L. Huber, Ph.D. Thesis, University of Delaware, 1950.

[16] W. E. Bachmann, *J. Amer. Chem. Soc.*, **55**, 3005 (1933).

[17] P. J. Hamrick, Jr., and C. R. Hauser, *ibid.*, **81**, 2096, 3144 (1959). The condensation of sodium diphenylmethide with benzophenone was found to involve a rapid reversible condensation; instantaneous acidification of the mixture furnished 1,1,2,2-tetraphenylethanol, but gradual acidification resulted in the recovery of starting materials. See also W. G. Kofron, W. R. Dunnavant, and C. R. Hauser, *J. Org. Chem.*, **27**, 2737 (1962).

[18] W. A. Mosher, T. H. Fairbanks, Jr., and L. J. Prucino, Abstracts, 126th National Meeting of the American Chemical Society, New York, N. Y., Sept 1954, p 91-O.

[19] P. T. Lansbury, *J. Amer. Chem. Soc.*, **83**, 429 (1961). Elimination of the trityl anion was established by trapping with benzyl chloride to form 1,1,1,2-tetraphenylethane; see ref 6.

[20] R. C. Fuson and P. Tomboulian, *ibid.*, **79**, 956 (1957).

[21] W. A. Mosher and M. L. Huber, *ibid.*, **73**, 795 (1951).

[22] G. A. Olah, C. U. Pittman, Jr., E. Namanworth, and M. B. Comisarow, *ibid.*, **88**, 5571 (1966).

[23] G. A. Olah, private communication.

lithium, no benzopinacolone or benzophenone was found and the only products isolated were ketone **7** (16%), triphenylmethane (39%), hydrocarbon **8** (6%), and triphenylcarbinol (69%). When the phenyllithium reagent was not in excess, some starting material was recovered (25%) along with the reaction intermediates benzophenone (3.3%) and benzopinacolone (31%); triphenylmethane (24%), ketone **7** (4.8%), and triphenylcarbinol (31%) were also found. Some yellow polar carbonyl compounds were present, probably intermediates in the formation of ketone **7**.[21] The material balance indicates that no significant reaction product has been undetected in the analysis. These findings are completely consistent with the reaction pathway described above and lend further support to the hypothesis concerning the instability of the pentaphenylethoxide ion.

Experimental Section

Melting points are corrected. Microanalyses were performed by Clark Microanalytical Laboratory, Urbana, Ill., and Spang Microanalytical Laboratory, Ann Arbor, Mich. Infrared spectra were measured in carbon disulfide solution (unless otherwise indicated) with Beckman IR-5 and IR-12 spectrophotometers.

Ethereal solvents were distilled from lithium aluminum hydride into the reaction flask. An argon atmosphere was employed in all trityllithium reactions. Purification of the argon was accomplished best by passing it through a benzophenone–lithium ketyl mixture in the ether used for the reaction.

Preparation of Trityllithium.—A summary of the methods and conditions is presented in Table I. Results with commercial butyllithium seemed to be slightly less reproducible than those in which the butyllithium was prepared by direct reaction of lithium with butyl chloride in the reaction solvent.

TABLE I

YIELDS OF TRITYLLITHIUM

Excess of BuLi, %	Solvent[a]	Reaction time, hr	Temp, °C	Yield, %
150	THF	2	−25 to 25	100
50	TH2MF	24	25	100
25	THP	20	25	90
300	DME	1.5	25	50
50	TH2,5DMF	8	15	11
30	Dioxane	3	15	14

[a] THF is tetrahydrofuran, TH2MF is tetrahydro-2-methylfuran, THP is tetrahydropyran, DME is 1,2-dimethoxyethane, TH2,5DMF is tetrahydro-2,5-dimethylfuran.

Yields of trityllithium were determined by weighing the triphenylacetic acid formed following carbonation of the reagent.[2] Fractional crystallization of many samples of the carbonation product did not reveal the presence of an acid other than triphenylacetic acid, as indicated by infrared analysis.

***p*-(Diphenylmethyl)diphenylhydroxymethylbenzene (1) by Reaction of Trityllithium with Benzophenone in Tetrahydropyran.**—In a typical experiment, trityllithium was prepared from 6.13 g (25.1 mmol) of triphenylmethane and a 10% excess of *n*-butyllithium in 50 ml of tetrahydropyran at 15° during a 3-hr reaction period. To the reagent, 2.36 g (13.0 mmol) of solid benzophenone was added. After 9 hr at 21–25°, dilute hydrochloric acid was added to the deep red mixture. The organic layer was removed by methylene chloride extraction. Following distillation of the solvents, the yellow oily product was subjected to chromatography on Alcoa F-20 alumina. Triphenylmethane (3.88 g) and *p*-(diphenylmethyl)triphenylmethylbenzene (0.40 g) were obtained in the early fractions. Elution with ether furnished the alcohol **1** (1.85 g, 33% yield based on benzophenone), mp 130–135°. Recrystallization from hexane furnished the analytical sample, mp 141–142° with decomposition. (The crystalline alcohol has a marked tendency to trap benzene and carbon tetrachloride; removal of these solvents *in vacuo* is slow.)

Anal. Calcd for $C_{32}H_{26}O$: C, 90.10; H, 6.14. Found: C, 89.87, 90.35; H, 5.90, 6.50.

The infrared spectrum closely resembles that of triphenylcarbinol, with the addition of bands at 2880 (benzylic C–H), 1022, 840, 805, 732, 662, 607, and 510 cm^{-1}. The mass spectrum[24] exhibited a parent peak (M) at *m/e* 426; other principal peaks occurred at *m/e* 409 (M − OH), 349 (M − C_6H_5), 271 (M − $2C_6H_5$), and 259 [M − $(C_6H_5)_2CH$]. The pmr spectrum[24] had peaks for aromatic hydrogens at 7.26 and 7.19 (24 H), for a benzylic hydrogen at 5.53 (1 H), and for an hydroxyl proton at 2.77 (1 H).

Stability of Alcohol 1 to Base.—The effects of a variety of bases on the alcohol **1** were examined. Decomposition was detected by infrared analysis of the reaction products recovered from the base treatment. No decomposition was detected after (1) heating at 305° for 5 min in a soft glass tube, (2) heating to 255° for 4 min with powdered potassium hydroxide, (3) boiling for 16 hr in ethanol with sodium ethoxide, and (4) stirring with an excess of butyllithium at 25° for 2 days in hexane.

Reduction of Alcohol 1 with Formic Acid.—A 0.105-g sample of the alcohol **1** was heated under reflux with 0.5 ml of toluene and 3.0 ml of 98% formic acid for 4 hr. Removal of the solvent furnished 0.074 g of solid with mp 163–170°. Crystallization from hexane or sublimation yielded needles, mp 176–177°. Mixture melting point determinations and infrared comparisons of this hydrocarbon with authentic *p*-bis(diphenylmethyl)benzene (**2**) showed the two to be identical.

***p*-Bis(diphenylhydroxymethyl)benzene** (**3**) was prepared by treatment of 2.20 g (0.0113 mol) of dimethyl terephthalate with excess (0.06 mol) phenyllithium in 100 ml of ether for 20 hr at reflux temperature. The crude diol product (5.07 g) was crystallized from benzene, mp 167–170° (lit.[25,26] mp 170–171°, 175°). Infrared maxima (fluorocarbon mull) were at 2.89 (OH) and 12.1 μ (*para*-substituted benzene).

***p*-Bis(diphenylmethyl)benzene** (**2**) was prepared by boiling 0.92 g of the above diol with 7 ml of 98% formic acid and 3 ml of toluene for 8 hr. Removal of the solvent produced the hydrocarbon, mp 175.5–177° (lit.[27] mp 171°). The infrared spectrum resembled that of triphenylmethane with the addition of bands at 1022, 842, 793, 719, and 510 cm^{-1}.

The reaction of phenyllithium with benzopinacolone was repeated over 20 times with a variety of conditions and solvents. Product analysis employed gas–liquid partition and column chromatographic techniques, plus infrared analysis.

In a typical reaction at 30°, 1.20 g (2.9 mmol) of benzopinacolone dissolved in 20 ml of benzene was added to an excess (50 mmol) of phenyllithium in 30 ml of ether. The mixture turned first yellow, then dark red-brown. After 1.5 hr, hydrolysis was effected by addition to an ice–ammonium chloride mixture. Chromatographic separation of the products furnished triphenylmethane, triphenylcarbinol, and a trace amount of benzopinacolone. No other crystalline compounds were obtained; infrared analysis of the crude reaction product as well as chromatographic fractions did not indicate the presence of other materials. In similar reactions, gas–liquid partition chromatographic analysis did not disclose any additional compounds.

In an experiment involving inverse addition, 13 mmol of phenyllithium in 10 ml of ether and 20 ml of toluene was slowly added to 3.70 g (10.5 mmol) of benzopinacolone in 60 ml of toluene while the mixture was held in a Dry Ice bath at −65°. After 15 min, the mixture was added to dilute acetic acid. The crude reaction product was treated with a benzene–ligroin mixture and filtered. The insoluble fraction was unchanged benzopinacolone. The filtrate was concentrated and subjected to chromatography on alumina. (Infrared analysis of this fraction indicated an alcoholic component in addition to the ketone.) Triphenylmethane, benzopinacolone, and triphenylcarbinol were isolated in small quantities as crystalline compounds; no other crystalline components were obtained and no evidence for benzo-

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[24] The mass spectra were measured on an Hitachi RMU-6A instrument with a source temperature of 200°. We are indebted to Mr. Ronald Hites of the Massachusetts Institute of Technology for these spectral determinations. The pmr spectrum was determined in deuteriochloroform solution on a Varian Associates A56-60 spectrometer. Chemical shifts are reported in parts per million downfield from TMS. We are indebted to Mr. Steven Beare of the University of Illinois for this determination.

[25] G. J. Sloan and W. R. Vaughan, *J. Org. Chem.*, **22**, 750 (1957).

[26] R. A. Benkeser and W. Schroeder, *J. Amer. Chem. Soc.*, **80**, 3314 (1958).

[27] E. D. Bergmann, *ibid.*, **75**, 2761 (1953).

phenone was found. Trace amounts of phenolic oils and a carbonyl compound (absorption at 1735 cm^{-1}) were found in milligram quantities in the last (polar) fractions.

Attempted Halogenation of Pentaphenylethane.—The procedure of Bachmann[16] was followed, using bromine in carbon tetrachloride solution, and the products were examined by infrared analysis. No materials other than trityl bromide and dibromodiphenylmethane were indicated. The action of N-bromosuccinimide in carbon tetrachloride solution furnished no crystalline products. Only cleavage products were identified following photochlorination[28] with chlorine in carbon tetrachloride solution.

Decomposition of 9,10,10-Triphenyl-9,10-dihydro-9-phenanthrol (5).[20]—Small amounts of alcohol **5** were heated in both Pyrex and quartz tubes under a nitrogen atmosphere in an oil bath at 275°. Progress of the decomposition was followed by quantitative infrared spectroscopy, measuring the carbonyl band in the product (**6**). Observed half-lives were 7.5 and 60 min, respectively, in the Pyrex and quartz tubes. Essentially complete conversion to *o*-(α,α-diphenyl-*o*-tolyl)benzophenone (**6**) was observed in a Pyrex tube held at 300° for 30 min. The ketone **6** was isolated and identified by its melting point, 173–175° (lit.[21] mp 178–179°), and the infrared spectrum which exhibited typical benzophenone-type absorption bands at 1669, 1314, 1283, 1263, and 928 cm^{-1}, aliphatic CH absorption at 2900 cm^{-1}, and aromatic CH bands at 760, 753, 729, and 698 cm^{-1}.

Reaction of Triphenylacetic Acid with Phenyllithium.—In a typical reaction (one of three), 1.508 g (5.24 mmol) of triphenylacetic acid (three times crystallized from toluene) dissolved in 180 ml of ether was added to phenyllithium prepared from 7.4 ml (70 mmol) of bromobenzene. After stirring at 25° for 3 hr, the red-brown mixture was added to ice and water. Triphenylacetic acid (0.305 g) was recovered from the water layer. Removal of the organic solvent left 1.62 g of a yellow semisolid which furnished 0.42 g of crystals, mp 140–161°, upon treatment with ligroin (bp 30–60°). Infrared analysis indicated this to be mainly triphenylcarbinol, which was isolated from some of the runs (mp 160–166°). Crystallization of this impure material from benzene furnished a small amount of benzopinacol, mp 183–185°. Benzophenone and triphenylmethane were readily identified in the crystallization residues, but no other reaction products were indicated.

In the other runs, the same products were identified, although the yields were not consistently reproducible, but in no case was any evidence for alcohol **1** found.

Methyl triphenylacetate was prepared conveniently from triphenylacetyl chloride and methanol. Triphenylacetic acid, thionyl chloride, and hexane were boiled for 3 hr and the solvent was distilled below 80°. Methanol was added and the mixture was heated under reflux for 18 hr. Removal of the solvent followed by crystallization furnished 3.69 g (79% yield) of methyl triphenylacetate, mp 185–187°. An additional crystallization gave a sample with mp 187.5–189° (lit.[29] mp 185–187°). The infrared spectrum exhibits ester absorption at 1743 and 1222 cm^{-1}.

Reaction of Methyl Triphenylacetate with Phenyllithium. A.—To a solution of phenyllithium prepared from 0.100 mol of bromobenzene and 0.190 g-atom of lithium in 75 ml of ether was added 1.33 g (4.43 mmol) of the ester (mp 187.5–189°) as a slurry in 75 ml of ether. The resulting deep red mixture was stirred at 23° for 2 hr before hydrolysis was effected by addition to ice. Methylene chloride extraction furnished the crude product which was subjected to chromatographic analysis on activated Alcoa F-20 alumina. Analysis of the fractions employed gas–liquid partition chromatography (SE-30 on glass beads) and quantitative infrared spectroscopic techniques. In addition to biphenyl, the following reaction products were obtained (yields are based on the ester): triphenylmethane, 0.426 g (39%); *p*-(diphenylmethyl)triphenylmethylbenzene (**8**), 0.062 g (6%); *o*-biphenylyl triphenylmethyl ketone (**7**), 0.308 g (16%); triphenylcarbinol, 0.721 g (69%). No evidence for the presence of benzopinacolone, benzophenone, benzopinacol, or alcohol **1** was found. Products were identified by comparison with known samples.

When the reaction was repeated using four times as much ester (67 mmol of phenyllithium and 10.3 mmol of ester), essentially the same results were obtained.

B.—A freshly prepared solution of phenyllithium (17 mmol) was cooled to −70°. A slurry of 1.79 g (5.97 mmol) of methyl triphenylacetate was added in 100 ml of ether; no color change was observed. The stirred mixture was allowed to warm up to 20° during 1.5 hr and after 1 hr at 23° hydrolysis was effected with a mixture of ice and hydrochloric acid. Product analysis was by the same techniques as above: recovered ester, 0.456 g (25%); triphenylmethane, 0.344 g (24%); benzophenone, 0.036 g (3.3%); benzopinacolone, 0.646 g (31%); *o*-biphenylyl triphenylmethyl ketone (**7**), 0.125 g (4.8%); triphenylcarbinol, 0.498 g (31%). Infrared analysis did not indicate the presence of any other alcoholic compounds, except for trace amounts in the last polar fractions which were yellow oils with phenolic odors; carbonyl absorption bands (1701 and 1672 cm^{-1}) were also present.

Registry No.—Benzophenone, 119-61-9; tetrahydropyran, 142-68-7; **1**, 15591-47-6; trityllithium, 733-90-4; phenyllithium, 591-51-5; benzopinacolone, 466-37-5; triphenylacetic acid, 595-91-5; methyl triphenylacetate, 5467-21-0.

Acknowledgment.—The early phases of this research were supported by a Frederick Gardner Cottrell Grant from the Research Corporation.

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(28) R. E. Lovins and L. J. Andrews, *J. Org. Chem.*, **29**, 487 (1964).

(29) A. G. Brook and H. Gilman, *J. Amer. Chem. Soc.*, **76**, 77 (1954).