These solutions were transferred by syringe in the proper proportions to a reaction flask immersed in an ice bath. Total volumes of solution were approximately 20 ml. Typically the reductions were carried out using 0.1 ml. of epoxide, which was added dropwise to the reducing solution at 0°. For more accurate yield determinations, a few reductions were carried out on a larger scale.

The solution, after being stirred for 0.5 hr., was decomposed with excess dilute hydrochloric acid. The ethereal layer was separated, washed with water and bicarbonate solution, dried over magnesium sulfate, and evaporated. The residue was examined directly by vapor phase chromatography, using an 8-m. diethylene glycol succinate column at 140°.

The retention times of the axial alcohols (*trans*-3- and *cis*-4-*t*-butylcyclohexanol) were too similar for direct analysis, and consequently this analysis gave only axial to equatorial ratios. On this same column, however, the acetates of all four isomeric alcohols were cleanly separated, and these were used for exact product measurements.

Reduction of *trans*-4-*t*-Butylcyclohexene Oxide with Lithium Aluminum Deuteride.—To a solution of 2.0 g. (0.048 mole) of lithium aluminum deuteride[23] in ether at 0° was added 2.0 g. (0.013 mole) of *trans*-4-*t*-butylcyclohexene oxide over a period of 0.25 hr. The mixture was stirred for 2 hr., then decomposed, washed, dried, and evaporated as described for the normal isotope reduction. Preparative vapor phase chromatographic separation was accomplished using the 8-m. diethylene glycol succinate column and 0.2-ml. injections.

The major product (**1**) after collection was analyzed and found to contain 95.6 ± 0.5% *trans*-3-*t*-butylcyclohexanol and 4.4 ± 0.5% *cis*-3-*t*-butylcyclohexanol.

The minor product (**2**, 11% of original material) analyzed after collection contained 90.1 ± 0.3% *cis*-3-*t*-butylcyclohexanol and 9.9 ± 0.3% *trans*-3-*t*-butylcyclohexanol. Further analyses involved these somewhat isomerically impure materials **1** and **2**.

The nuclear magnetic resonance spectrum of **1** was very similar to that of *trans*-3-*t*-butylcyclohexanol prepared by reduction of 3-*t*-butylcyclohexanone and isolated by preparative v.p.c. The only notable difference was a decrease in the ring proton peak at *ca.* 1.65 p.p.m. (from tetramethylsilane), expected for substitution by deuterium. The carbinol carbon proton at 4.10 p.p.m. was present, and integration indicated no measurable deuterium at this position.

The spectrum of **2** contained no peak at 3.38 p.p.m. (C-1 proton), and showed slight differences in the ring proton region, when compared with that of an isotopically normal sample.

The infrared spectrum of **1** contained a sharp single peak at 4.63 ± 0.01 μ with a very weak shoulder at 4.71 ± 0.02 μ. The C–D stretching region for **2** showed a clear doublet, the stronger peak at 4.64 ± 0.01 μ, another at 4.74 ± 0.1 μ. A sample of 3-*t*-butylcyclohexanone was reduced with lithium aluminum deuteride, and the product (90% *cis*-3-*t*-butylcyclohexanol-1-*d*)[14] showed a somewhat broadened peak at 4.74 ± 0.01 μ with a weak shoulder at 4.59 ± 0.02 μ.

Since nuclear magnetic resonance had indicated C-1 deuterium substitution in **2**, borne out by the infrared absorption at 4.74 μ, the other C–D stretch must be assigned to the adjacent ring position. Although the excellent wave-length agreement for these peaks in **1** and **2** (4.63 and 4.64 μ) might suggest assignment of like (axial) conformations to the substituents, such speculation may not be valid.[24] Combustion analyses[25] of samples **1** and **2** indicate that **1** is 98% monodeuterated and **2** is 94% dideuterated.

Anal. Calcd.[26] atom % excess D for $C_{10}H_{19}DO$ (**1**): 5.06. Found: 4.96.

Anal. Calcd.[27] atom % excess D for $C_{10}H_{18}D_2O$ (**2**): 9.22. Found: 8.65, 8.70.

Controls.—Known mixtures of *cis*- and *trans*-4-*t*-butylcyclohexanol were added to lithium aluminum hydride and hydride–aluminum chloride (3:1) in ether. After 24 hr. these mixtures were recovered, by an extraction procedure identical with that described for the reductions. In all cases the alcohol mixtures were unchanged, thus precluding equilibration, preferential destruction, or loss in work-up of either isomer.

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[23] Metal Hydrides' 97% material was used; we wish to thank Professor G. O. Pritchard for a timely gift of this material.

[24] A. Streitwieser, Jr., and C. E. Coverdale [*J. Am. Chem. Soc.*, **81**, 4275 (1959)] have reported C–D stretching frequencies of 4.60 and 4.66 μ, respectively, for *trans*- and *cis*-cyclohexanol-2-*d*. These materials were prepared by lithium aluminum deuteride reduction of cyclohexene oxide and presumably contain about 10% C-1-deuterated material. These spectra were obtained from thin-film neat samples while we used carbon tetrachloride solutions, and hence direct comparison is not possible. Nevertheless, the very slight shift associated with deuterium conformation would make a structural assignment on such a basis very questionable.

[25] Analyses were by Josef Nemeth, Urbana, Ill.

[26] Corrected for the purity (97%) of lithium aluminum deuteride and for the inclusion of 4.4% $C_{10}H_{18}D_2O$ in the sample.

[27] Corrected for purity of lithium aluminum deuteride and for the inclusion of 9.9% of $C_{10}H_{19}DO$ in the sample. Isotope effects have necessarily been ignored. These corrections cannot alter the gross conclusion that **1** is monodeuterated and **2** is dideuterated.

Cleavage of 1,2-Diazetidinones by Bases. The Reaction of 1,2,4,4-Tetraphenyl-1,2-diazetidinone with Alkoxides, Organolithium Compounds, and Lithium Aluminum Hydride

J. HERBERT HALL

Department of Chemistry, Southern Illinois University, Carbondale, Illinois

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Reaction of 1,2,4,4-tetraphenyl-1,2-diazetidinone (I) with sodium ethoxide or sodium *t*-butoxide, followed by hydrolysis, results in cleavage of the C–N bonds to give azobenzene and diphenylacetic acid. In contrast, reaction of I with methyllithium results in cleavage of the N–N and C–C bonds to give benzophenone anil and acetanilide. Similarly, I reacts with phenyllithium to give benzophenone anil and benzanilide. Lithium aluminum hydride cleaves I to give azobenzene and diphenylacetaldehyde as the major products along with smaller amounts of 1,1-diphenylethylene glycol, 2,2-diphenylethanol, and aniline.

The type of products formed in the base-induced cleavage of the 1,2-diazetidinone ring system has been found to depend on the nature of the base used and the structure of the 1,2-diazetidinone. Ingold and Weaver[1] reported that ethyl 1,4,4-triphenyl-1,2-diazetidinone-2-carboxylate underwent rapid amide cleavage when treated with 2% sodium hydroxide in aqueous ethanol. The structure of their starting material has been recently reinvestigated by Bird[2] and has been shown to have the alternate ethyl 2,4,4-triphenyl-1,2-diazetidinone-1-carboxylate (IIa) structure, which on basic hydrolysis would give the acid IIIa. Schenck and Engelhard[3] reported that 1,2-diphenyl-1,2-diazetidinone (IIb) was rapidly cleaved by cold, dilute sodium hydroxide solution to give the acid IIIb. Similarly, Cook and Jones[4] reported the easy base hydrolysis of IIc to IIIc,

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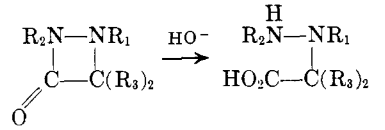
[1] C. K. Ingold and S. D. Weaver, *J. Chem. Soc.*, **127**, 378 (1925).

[2] C. W. Bird, *ibid.*, 674 (1963).

[3] G. O. Schenck and N. Engelhard, *Angew. Chem.*, **68**, 71 (1956).

[4] A. H. Cook and D. G. Jones, *J. Chem. Soc.*, 184 (1941).



II III

a, $R_1 = CO_2C_2H_5$; $R_2, R_3 = C_6H_5$
b, $R_1, R_2 = C_6H_5$; $R_3 = H$
c, $R_1 = p\text{-}ClC_6H_4$; $R_2 = C{\equiv}N$; $R_3 = C_6H_5$

but the structure assignments of these compounds are in doubt in light of the work of Bird.[2]

In contrast to these results, 1,2,4,4-tetraphenyl-1,2-diazetinone (I) was reported to be stable in cold alcoholic alkali,[4] but on prolonged heating with 50% sodium hydroxide it was cleaved into azobenzene and diphenylacetic acid.[5] Cook and Jones also reported that I was cleaved by refluxing with 10% sodium methoxide in methanol to give azobenzene.[4] No other products were isolated.

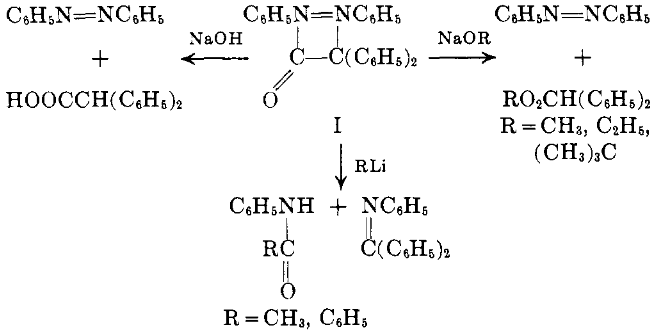
This alkoxide cleavage has been confirmed. For example, prolonged refluxing of I with 10% sodium ethoxide in ethanol, followed by rapid work-up in aqueous medium, gave azobenzene and diphenylacetic acid, isolated in 85 and 80% yields, respectively. Ethyl diphenylacetate, which would be one of the expected cleavage products, hydrolyzed in the aqueous solution before it could be isolated.

Analogous results were obtained by refluxing I in 6% sodium *t*-butoxide in *t*-butyl alcohol. Azobenzene and diphenylacetic acid were isolated in 79 and 80% yields.

When I was treated with methyllithium in ether solution, a different type of cleavage was observed. The isolated products, which were identified by their infrared spectra and mixture melting points, were acetanilide (73%), benzophenone anil (43%), azobenzene (<2%), and benzophenone (41%). The latter of these is a product of the facile hydrolysis of benzophenone anil during isolation of the products.

Similar results were obtained when I was treated with phenyllithium in ether. The products obtained were benzanilide (67%), benzophenone anil (43%), benzophenone (25%), and aniline (isolated as acetanilide). These results are summarized in Chart I.

CHART I



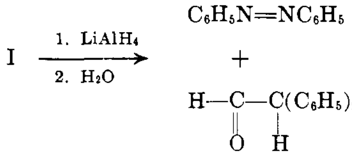
The reduction of I with excess lithium aluminum hydride was attempted with the expectation that the product of the reaction would be N-(1,1-diphenyl-2-hydroxyethyl)hydrazobenzene. Instead of the expected product, a mixture of products was obtained. The mixture was separated by chromatography on alumina. The products isolated and identified were azobenzene (76%), benzophenone (62%), 2,2-diphenylethanol (7%), 1,1-diphenylethylene glycol (9%), and aniline (3%). Two other unidentified products were also isolated, a white solid, m.p. 117–120°, and a bright yellow solid, m.p. 245°, each in <1% yield.

Similar results were obtained when 1,2-di(*o*-chlorophenyl)-4,4-diphenyl-1,2-diazetidinone was treated with lithium aluminum hydride. *o,o'*-Dichloroazobenzene was isolated in 58% yield and benzophenone was isolated in 40% yield as its 2,4-dinitrophenylhydrazone.

The infrared spectrum of the crude reaction mixture from the reduction of I with lithium aluminum hydride contained two carbonyl peaks, one at 1665 cm.$^{-1}$ due to benzophenone and the second at 1725 cm.$^{-1}$. When the lithium aluminum hydride reduction of I was carried out in a nitrogen atmosphere and the crude reaction mixture was protected from air, the benzophenone carbonyl peak at 1665 cm.$^{-1}$ was absent and the 1725-cm.$^{-1}$ peak was very strong. The crude reaction mixture gave positive aldehyde tests with Tollens and Schiffs reagents. Finally, treatment of the crude reaction mixture with 2,4-dinitrophenylhydrazine gave a 2,4-dinitrophenylhydrazone which was identical with that prepared from diphenylacetaldehyde.

While this work was in progress, Huffman and Elliott reported that diphenylacetaldehyde is oxidized in air to benzophenone.[6] They also noted that the oxidation is accelerated when the aldehyde is absorbed on alumina. Thus the benzophenone produced in these reactions is the result of air oxidation of diphenylacetaldehyde during the chromatographic separation of the products.

The azobenzene produced in the reaction of I with lithium aluminum hydride was not formed during the reaction, but was formed during hydrolysis of the reaction mixture. This was shown by the fact that the solution was colorless at the end of the reaction, and the red color of the azobenzene appeared after sufficient water had been added to destroy all of the excess lithium aluminum hydride. This result is in contrast to the reactions of I with sodium hydroxide and sodium alkoxides, where the azobenzene color appears during the reaction.



The reaction of I with sodium hydride in refluxing benzene was attempted, but only starting material was recovered. Also I failed to react with ethylmagnesium bromide in refluxing ether.

The formation of the main cleavage products in the above reactions can be understood by reference to Chart II. The addition of alkoxide to the carbonyl of I would give the intermediate VI which could decompose to give azobenzene and the enolate ion of the diphenylacetate ester (VII) by either a stepwise or concerted mechanism.

The reaction of I with lithium aluminum hydride is represented by the addition of hydride ion to the carbonyl of I to give the complex VIII. Apparently only one of the hydride ions of the lithium aluminum hydride is used in the reaction. This was indicated by the fact that, when I and lithium aluminum hydride were used

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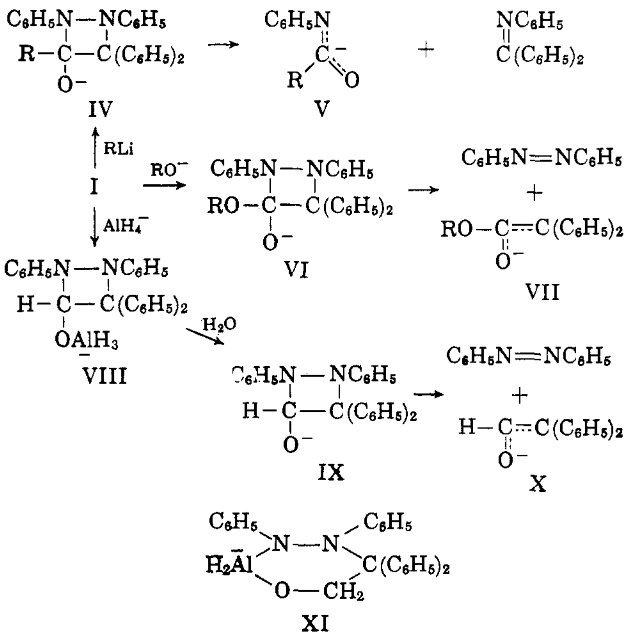
[5] L. Horner and E. Spietschka, *Ber.*, **89**, 2765 (1956).

[6] J. W. Huffman and R. R. Elliott, *Chem. Ind.* (London), 650 (1963).

CHART II



in a 4:1 molar ratio and the reaction time was extended to 24 hr., 80% of I was recovered unchanged. That complex VIII is stable is shown by the absence of the red azobenzene color. Addition of water destroys the complex and releases the anion IX which can then decompose by either a concerted process or stepwise process into azobenzene and the enolate ion of diphenylacetaldehyde (X).

The fact that a small amount of 2,2-diphenylethanol was formed in the reaction indicates that VIII to some small extent can suffer further reduction, perhaps with the formation of the cyclic complex XI (or a similar linear structure). If destruction of XI by water occurs by initial cleavage of the Al–N bond, the products should be azobenzene and 2,2-diphenylethanol. On the other hand, if the Al–O bond cleaves first, the expected products would be 1,1-diphenylethylene oxide and hydrazobenzene. Further hydrolysis of the epoxide would give the observed 1,1-diphenylethylene glycol. The presence of hydrazobenzene in the reaction mixture has not been verified. Attempts were made to detect its presence in the infrared spectrum of the reaction products, by its rearrangement to benzidine, and by chromatography, but all of these methods were too insensitive to show the presence of the small amount of hydrazobenzene that would be present in the reaction mixture.

The cleavage of I with methyllithium or phenyllithium would be expected to proceed by addition of the reagent to the carbonyl group to give IV. Decomposition of IV would then give benzophenone anil and the enolate ion of acetanilide or benzanilide (V).

Why cleavage of the N–N and C–C bonds in IV occurs, while cleavage of the C–N bonds in VI and IX occurs, is not fully understood. There are several factors that may influence the cleavage. In the first place, the cleavage of VI and IX occurs in the presence of the polar hydroxylic solvents ethanol and water, while the cleavage of IV occurs in the nonpolar ether. Secondly, the approach to the carbonyl carbon is somewhat hindered by the phenyl groups on C-4. Steric factors certainly would be expected to influence the rate of reaction, but this cannot be the major factor in deciding which bonds are to be cleaved. This is evident by the fact that the two bulkiest groups used, phenyl and *t*-butoxide, give different types of cleavage. Likewise, the two smallest groups, hydride and methide, cleave the ring in different ways. Finally, the reactions leading to cleavage of the C–N bonds may be the result of thermodynamic control, whereas the reactions leading to cleavage of the N–N and C–C bonds may be the result of a kinetically controlled concerted cleavage, in which case, structure IV would be a transition state rather than a discrete intermediate. Experiments are currently being designed to help evaluate some of these factors.

Experimental

Reaction of Lithium Aluminum Hydride with 1,2,4,4-Tetraphenyl-1,2-diazetidinone.—In 50 ml. of dry benzene was dissolved 2.48 g. (0.068 mole) of 1,2,4,4-tetraphenyl-1,2-diazetidinone.[5] This solution was added dropwise over a period of 50 min. to a stirred solution of 0.25 g. (0.066 mole) of lithium aluminum hydride in 75 ml. of dry ether. After 6 hr. of stirring, water was added to destroy the excess lithium aluminum hydride. No color was produced during the reaction, but after all of the lithium aluminum hydride was destroyed, the organic layer turned red due to the formation of azobenzene. The reaction mixture was acidified by addition of 5% hydrochloric acid. The ether–benzene layer was separated, washed with water, extracted with sodium bicarbonate solution, and dried over magnesium sulfate.

The acid layer from above was evaporated to dryness and redissolved in 10 ml. of 10% sodium hydroxide solution. Benzenesulfonyl chloride (1 ml.) was added and the solution was stirred for 2 hr. The aluminum hydroxide was filtered off and the filtrate was acidified. Extraction of the filtrate with benzene and evaporation of the solvent gave an oil which solidified on treatment with petroleum ether (b.p. 30–60°), yielding 0.04 g. (3%) of benzenesulfonanilide, m.p. 106–108°, m.m.p. 108–110°.

The ether benzene layer was evaporated to dryness. The red oil obtained was warmed with petroleum ether (b.p. 60–90°), and the petroleum ether solution was decanted. The insoluble residue was extracted with two more portions of petroleum ether.

The petroleum ether insoluble material was treated with benzene and the insoluble, white solid which formed was filtered off. The infrared spectrum of this solid was identical with that of a sample of 1,1-diphenylethylene glycol; yield 0.05 g. (4%), m.p. 121–122° (lit.[7] m.p. 121°).

The petroleum ether solution was placed on a 100-g. alumina (Fisher A-540) column. The column was eluted with a series of solvents to give the following fractions: 1, petroleum ether, 750 ml.; 2, 1:9 benzene–petroleum ether, 150 ml.; 3, benzene, 100 ml.; 4, benzene, 1 l.; 5, methylene chloride, 200 ml.; 6, methylene chloride, 200 ml.; and 7, 95% ethanol, 500 ml. Fraction 1 on evaporation gave 1.20 g. (70%) of azobenzene, m.p. 67–68°. Fraction 2 was concentrated to 2 ml. On standing, 0.01 g. of a bright yellow, unidentified compound crystallized; m.p. 245°. Fraction 3 on evaporation gave 0.70 g. (58%) of crude benzophenone, m.p. 41–45°, b.p. 303°. Recrystallization from petroleum ether raised this to m.p. 48–49°. Evaporation of the solvent from fraction 4 gave an oil whose infrared spectrum indicated that it was a mixture of 0.05 g. (4%) of benzophenone and 0.07 g. (6%) of azobenzene. Fraction 5 on evaporation gave an unknown solid, which after recrystallization from petroleum ether, gave 0.02 g., m.p. 117–120°. Fraction 6 on evaporation gave 0.10 g. (7%) of a colorless oil which refused to crystallize, but whose infrared spectrum was identical with that of an authentic sample of 2,2-diphenylethanol. This fraction was further identified by its reaction with phenyl isocyanate to give the same phenylurethan as that prepared from a sample of 2,2-diphenylethanol. Evaporation of the ethanol from fraction 7 gave a white solid. Recrystallization from benzene gave 0.07 g. (5%) of 1,1-diphenylethylene glycol, m.p. 122°. All of the solids

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[7] C. Paal and E. Weidenkaff, *Ber.*, **39**, 2063 (1906).

were identified by mixture melting points and/or by their infrared spectra.

1,2-Di(*o*-chlorophenyl)-4,4-diphenyl-1,2-diazetidinone.—Diphenylketene (0.97 g., 0.005 mole) and *trans-o,o'*-dichloroazobenzene were dissolved in 40 ml. of anhydrous ether in a Pyrex flask and the solution was irradiated with an ultraviolet U.A.-3 Uviarc mercury lamp until the dark red color was discharged (4 hr.). Removal of the solvent and recrystallization of the residue from aqueous acetone gave 2.0 g. (91%) of the diazetidinone. An analytical sample melted at 170°.

Anal. Calcd. for $C_{26}H_{18}Cl_2N_2O$: C, 70.12; H, 4.07. Found: C, 70.08; H, 4.03.

Reaction of Lithium Aluminum Hydride with 1,2-Di(*o*-chlorophenyl)-4,4-diphenyl-1,2-diazetidinone.—The above diazetidinone (0.746 g., 0.00164 mole) was dissolved in 35 ml. of dry benzene and this solution was added dropwise over a period of 1 hr. with stirring to 0.094 g. (0.002 mole) of lithium aluminum hydride in 35 ml. of dry ether. After 1.5 hr. of additional stirring, the excess lithium aluminum hydride was decomposed by addition of water. As soon as all of the lithium aluminum hydride was gone, the solution turned cherry red. The organic layer was separated, dried over magnesium sulfate, concentrated, and placed on a 25-g. alumina (Fisher A-540) column. The column was eluted with benzene. The first 30 ml. of eluent contained a red solid, which after recrystallization gave 0.24 g. (58%) of *o,o'*-dichloroazobenzene, m.p. 131–132° (lit.[8] m.p. 137°). This compound gave no melting point depression with an authentic sample. The next 250 ml. of eluent gave a light yellow liquid, whose infrared spectrum showed it to be primarily benzophenone. Treatment of the liquid with 2,4-dinitrophenylhydrazine gave 0.24 g. (40%) of benzophenone 2,4-dinitrophenylhydrazone, m.p. 237°.

Reaction of Sodium Ethoxide with 1,2,4,4-Tetraphenyl-1,2-diazetidinone.—A sodium ethoxide solution was prepared by dissolving 1.7 g. of sodium in 54 ml. of absolute ethanol. To this solution was added 0.83 g. (0.0022 mole) of 1,2,4,4-tetraphenyl-1,2-diazetidinone. The reaction mixture was refluxed for 2 days. During this time, the insoluble solid dissolved and a red oil appeared. The solution was concentrated to 15 ml. and diluted with water; the insoluble azobenzene was filtered off, yielding 0.34 g. (85%), m.p. 63–67°. Recrystallization from petroleum ether raised this to m.p. 69°. Acidification of the aqueous layer gave 0.34 g. (80%) of diphenylacetic acid, m.p. 147–148°. The azobenzene was identified by its infrared spectrum and the diphenylacetic acid by its mixture melting point with a known sample of the compound.

Reaction of Sodium *t*-Butoxide with 1,2,4,4-Tetraphenyl-1,2-diazetidinone.—A sodium *t*-butoxide solution was prepared by dissolving 2.4 g. of sodium in 182 ml. of *t*-butyl alcohol (dried by distillation from sodium). To 100 ml. of this solution was added 1.88 g. (0.005 mole) of 1,2,4,4-tetraphenyl-1,2-diazetidinone and the solution was refluxed for 20 hr. The *t*-butyl alcohol was then distilled and the residue was dissolved in water. The solution was quickly extracted with ether. Evaporation of the ether solution gave 0.72 g. (79%) of azobenzene, m.p. 67°. Acidification of the aqueous layer gave 0.85 g. (80%) of a white solid, m.p. 145°, which gave no melting point depression with diphenylacetic acid.

Reaction of Methyllithium with 1,2,4,4-Tetraphenyl-1,2-diazetidinone.—Methyllithium (0.004 mole) was prepared from 0.57 g. of methyl iodide and 0.06 g. of lithium in dry ether at −10°.[9] A slurry of 0.75 g. (0.002 mole) of 1,2,4,4-tetraphenyl-1,2-diazetidinone in 50 ml. of dry ether was added to the methyllithium solution all at once. The solution was stirred for a period of 18 hr., during which time it gradually warmed to room temperature. Water was added to destroy the excess methyllithium. The ether layer was separated, washed with water, and dried over magnesium sulfate. Evaporation of the ether gave a yellow viscous oil. This oil was heated with 10 ml. of petroleum ether, the solution was cooled, and the insoluble acetanilide was filtered off, yielding 0.17 g. (73%), m.p. 116°. This solid was identified by mixture melting point and infrared spectrum. The petroleum ether filtrate was concentrated to 5 ml. After standing several hours, two crystalline compounds separated. One was in the form of large brown crystals and the other as fine granules. The two compounds were easily separated mechanically. The large crystals, m.p. 110°, possessed an infrared spectrum which was identical with that of benzophenone anil; yield 0.19 g. (43%). Recrystallization from absolute ethanol raised the melting point to 113°. The fine granular material was identified, by its infrared spectrum and mixture melting point, as starting material; yield 0.06 g., m.p. 174–176°.

The petroleum ether filtrate left after the separation of the above products was subjected to chromatography on 10 g. of alumina (Fisher A-540). Elution with petroleum ether gave a trace of a red solid whose infrared spectrum showed it to be azobenzene. Elution of the column with benzene gave a fraction which on evaporation gave a semisolid. Treatment of the semisolid with 2 ml. of petroleum ether dissolved the oil and left 0.04 g. of insoluble solid, identified as starting material. Evaporation of the petroleum ether solution gave an oil which solidified on seeding with benzophenone; yield 0.13 g. (41%). Recrystallization of the solid from petroleum ether by cooling in ice gave a pure sample of benzophenone, m.p. 48°.

Reaction of Phenyllithium with 1,2,4,4-Tetraphenyl-1,2-diazetidinone.—Phenyllithium was prepared by reacting 1.56 g. (0.023 mole) of bromobenzene with 0.16 g. (0.023 mole) of lithium in 30 ml. of dry ether.[10] To this solution was added a slurry of 1.88 g. (0.005 mole) of 1,2,4,4-tetraphenyl-1,2-diazetidonone in 30 ml. of dry ether. On stirring, the diazetidinone dissolved rapidly. The reaction was allowed to stand overnight to insure complete reaction. The excess phenyllithium was destroyed by the addition of water. The reaction mixture was acidified with dilute hydrochloric acid and the emulsion was filtered to remove an insoluble solid. Recrystallization of this solid from ethanol gave 0.18 g. (18%) of benzanilide, m.p. 160–162°. The layers of the filtrate were then separated.

The aqueous layer was made basic with sodium hydroxide and treated with acetic anhydride. The precipitated solid was filtered off and identified as acetanilide by mixture melting point.

The ether layer was dried over magnesium sulfate and then evaporated to give an oil. This oil was extracted several times with 10-ml. portions of warm petroleum ether. The petroleum ether insoluble material was recrystallized from 95% ethanol and gave 0.41 g. (41%) of benzanilide, m.p. 159–162°. It gave no melting point depression with an authentic sample.

The petroleum ether extracts were concentrated to 20 ml., cooled, and decanted from the small amount of oil that separated. Seeding of this solution induced the crystallization of 0.44 g. (36%) of benzophenone anil, m.p. 107–111°. Recrystallization from ethanol raised this to m.p. 112° and this solid gave no melting point depression with a known sample of benzophenone anil.

The petroleum ether filtrate, after removal of the benzophenone anil, was placed on an alumina column and the column was eluted with petroleum ether to give 0.24 g. (25%) of benzophenone, identified by its infrared spectrum. Elution with benzene gave an additional 0.08 g. (8%) of benzanilide.

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(8) D. Volander and F. Meyer, *Ann.*, **320**, 130 (1902).

(9) The procedure for preparation of butyllithium reported by H. Gilman, J. A. Beel, C. G. Brannen, M. W. Bullock, G. E. Dunn, and L. S. Miller [*J. Am. Chem. Soc.*, **71**, 1499 (1949)] was used.

(10) H. Gilman and L. S. Miller, *Org. Reactions*, **6**, 353 (1951).