of 2-(dimethylaminomethyl)benzyl phenyl ketone (XXII), b.p. 143–146° (0.3 mm.).[19]

Anal. Calcd. for $C_{17}H_{19}NO$: C, 80.57; H, 7.56; N, 5.53. Found: C, 80.39; H, 7.64; N, 5.74.

The picrate, recrystallized twice from 95% ethanol, melted at 176–177.5°.

Anal. Calcd. for $C_{23}H_{22}N_4O_8$: C, 57.26; H, 4.60; N, 11.61. Found: C, 57.44; H, 4.67; N, 11.42.

The infrared spectrum of XXII showed a strong peak at 1690 cm.^{-1} for the carbonyl group,[20] at 845 cm.^{-1} for the dimethylaminomethyl group,[5] and at 753, 740, and 690 cm.^{-1} for four and five adjacent aromatic hydrogens.[21]

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[19] On standing several weeks, this light yellow oil became a red viscous material which resisted attempts at crystallization.

[20] See ref. 4, p. 132.

[21] See ref. 4, pp. 76–78.

Cyclization of Certain o-Chlorophenyl-β-dicarbonyl Compounds through Dicarbanion–Benzyne Intermediates[1]

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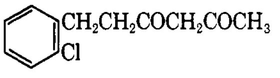
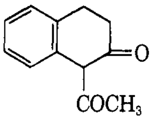
Bunnett's principle of ring closure involving the intramolecular reaction of an anion with the benzyne moiety was adapted to certain cyclizations in which the terminal methyl group of an o-chlorophenyl β-diketone or β-ketoaldehyde was condensed with the aromatic ring through a dicarbanion–benzyne intermediate. The cyclizations, which were effected by means of excess potassium amide in liquid ammonia, afforded five- and six-membered rings. One of these reactions was useful for the synthesis of 3-acetyl-2-tetralone (IX).

Bunnett,[2] Huisgen,[3] and co-workers have effected a number of cyclizations that involve the intramolecular condensation of an anion and the benzyne moiety. Bunnett has formulated this general principle[2a,b] of cyclization and has applied it to the synthesis of homocyclic,[2c] and heterocyclic[2b,d] products. One of these interesting cyclizations was observed with o-chlorophenyl β-diketone I, the methylene group of which was condensed intramolecularly with the aromatic ring to give II.[2c] This reaction was effected by excess potassium amide in liquid ammonia.



I

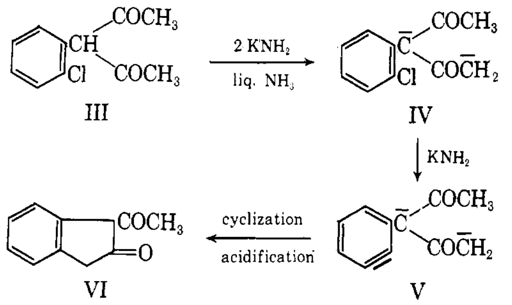


II

In the present investigation Bunnett's principle of ring closure was adapted to certain cyclizations in which a terminal methyl group of a β-diketone or β-ketoaldehyde was condensed intramolecularly with the aromatic ring through the intermediate formation of a dicarbanion and the benzyne moiety. The analogous intermolecular phenylation at the terminal methyl group of acetylacetone had been effected previously through the intermediate formation of benzyne and of the dicarbanion of the β-diketone.[4]

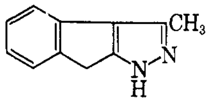
First, the o-chlorophenyl β-diketone III was cyclized to form the five-membered ring β-diketone VI in almost quantitative yield (Scheme A).

This reaction was effected by the gradual addition of excess[5] potassium amide in liquid ammonia to III in this medium, IV and V presumably being intermediates.



The intermediate formation of dicarbanion IV was indicated by a transient green color similar to that observed with the dicarbanion of 3-phenyl-2,4-pentanedione.[6] Indirect evidence, that the twofold ionization to form IV preceded dehydrohalogenation, was the lack of appreciable amination, which might have been expected if a benzyne were produced first.

Structure VI for the product was supported by analysis, by agreement of its melting point with the reported value,[7] by a positive enol test, and by cyclization with hydrazine to form pyrazole VII.



VII

Structure VI was confirmed by independent synthesis involving acetylation of β-indanone by means of boron trifluoride (eq. 1).

(1)

Similarly, o-chlorobenzyl β-diketone VIII was cyclized to form the six-membered ring β-diketone IX in 65% yield.

That the cyclization product was a carbocyclic derivative, not a heterocyclic derivative such as the

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[1] Supported by the National Science Foundation.

[2] (a) B. F. Hrutfiord and J. F. Bunnett, *J. Am. Chem. Soc.*, **80**, 2021 (1958); (b) J. F. Bunnett and B. F. Hrutfiord, *ibid.*, **83**, 1691 (1961); (c) J. F. Bunnett and J. A. Skorcz, *J. Org. Chem.*, **27**, 3836 (1962); (d) J. F. Bunnett, T. Kato, R. R. Flynn, and J. A. Skorcz, *ibid.*, **28**, 1 (1963).

[3] R. Huisgen and H. König, *Angew. Chem.*, **69**, 268 (1957); R. Huisgen and H. König, *Ber.*, **92**, 203, 429 (1959); R. Huisgen, H. König, and A. R. Lepley, *ibid.*, **93**, 1496 (1960).

[4] C. R. Hauser and T. M. Harris, *J. Am. Chem. Soc.*, **80**, 6360 (1958).

[5] At least three molecular equivalents of this reagent should be required to effect the reaction since the dicarbanion of product VI would probably be formed.

[6] W. I. O'Sullivan and C. R. Hauser, *J. Org. Chem.*, **25**, 1110 (1960).

[7] M. Konieczny, *Bull. acad. polon. sci., Ser. sci., chim., geol., geograph.*, **8**, 229 (1959).

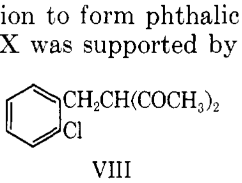
TABLE I

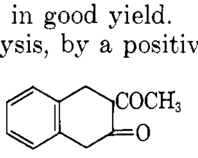
ULTRAVIOLET SPECTRA[a]

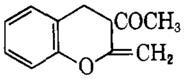
XV[b] λ ($\epsilon \times 10^{-3}$), mμ	IX λ ($\epsilon \times 10^{-3}$), mμ	2-Naphthol λ ($\epsilon \times 10^{-3}$), mμ	1,1′-Binaphthol[b] λ ($\epsilon \times 10^{-3}$), mμ
	228 (3.90) min		
261 (3.77) min	251 (6.50) max	248 (2.00) min	253 (3.02) min
267 (4.59) max	258 (6.19) sh	264 (3.86) max	268 (4.08) max
273 (3.95) min	268 (4.84) min	268 (3.52) min	270 (4.06) min
278 (4.59) max		275 (4.73) max	278 (4.73 max
286 (2.72) min		281 (2.74) min	284 (3.98) min
288 (2.77) max	291 (8.11) max	286 (3.30) max	288 (4.17) max
298 (0.77) min		295 (0.55) min	305 (1.55) min
320 (2.03) sh		318 (1.76) max	324 (2.81) sh
		323 (1.73) min	
332 (2.49) max	340 (0.11) min	335 (2.08) max	335 (3.65) max
	385 (0.25) max		

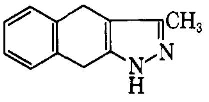
[a] max = maximum, min = minimum, and sh = shoulder. [b] The extinction coefficients given for these dimers are one-half the observed values. They were used to allow comparison with IX and 2-naphthol.

possible O-derivative X, was shown by permanganate oxidation to form phthalic acid in good yield. Structure IX was supported by analysis, by a positive enol test, by conversion to a copper chelate, and by formation of pyrazole XI and β-tetralone[8] with hydrazine and alkali, respectively.

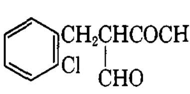

VIII

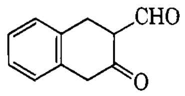

IX

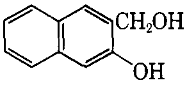

X

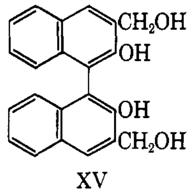

XI

Likewise, the *o*-chlorophenyl-β-ketoaldehyde XII was cyclized to form presumably the six-membered ring β-ketoaldehyde XIII, but this compound was not isolated. Evidently, XIII underwent aromatization to give carbinolnaphthol XIV followed by oxidative dimerization to afford XV, which was obtained in 29% yield.


XII

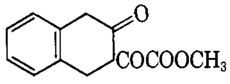

XIII

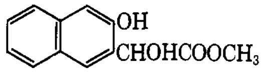

XIV


XV

Although the product isolated gave a positive enol test, the compound failed to form a copper chelate or a pyrazole. This suggested that it was a naphthol, such as XIV, rather than β-ketoaldehyde XIII. Moreover, the ultraviolet spectrum of the product was greatly different from that of IX, the keto analog of XIII, but resembled that of β-naphthol (Table I). However, the elemental analysis indicated slightly less hydrogen than is required for structure XIV and a molecular weight determination suggested that the compound was dimer XV. The ultraviolet spectrum correlated well with that of 1,1′-bi-2-naphthol (Table I). Such a dimerization would not be surprising, since ferric ion was employed in the preparation of the potassium amide used in the reaction and such metal ions are known to catalyze (in air) the oxidative dimerization of phenols and naphthols.[9]

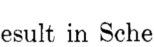
A precedent for the aromatization of XIII to form XIV is the observation that, although acylation of β-tetralone with methyl oxalate by sodium methoxide presumably affords oxalyl derivative XVI, the product isolated was the carbinolnaphthol XVII.[10]

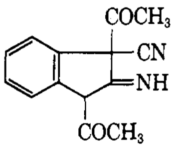

XVI


XVII

Discussion

The present method of cyclization is useful for the synthesis of 3-acetyl-2-tetralone (IX) and perhaps of dimer XV, both of which are new compounds. The method is satisfactory for the synthesis of 1-acetyl-2-indanone (VI) but the method of choice is probably that represented by eq. 1 even though the yield was only 35%. Both of these methods appear to be superior to one reported recently[7] involving reaction of dinitrile XVIII with ethyl acetate. The product, described as iminonitrile XIX, was hydrolyzed to give VI and β-indanone in unstated yields.


XVIII


XIX

The result in Scheme A is of interest in connection with those obtained by Bunnett and co-workers on *o*-chlorophenyl ketones XXa and XXb under similar conditions.[2b,c] Thus, whereas diketone III was converted to VI, no such carbocyclic product was isolated

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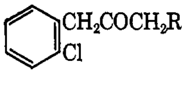
[8] A ketone acid resulting from cleavage at the carbonyl group of the ring may have been produced, but no attempt was made to isolate it.

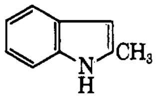
[9] See L. F. Fieser and M. Fieser, "Advanced Organic Chemistry," Reinhold Publishing Corp., New York, N. Y., 1961, p. 764.

[10] M. D. Soffer, R. A. Stewart, and G. I. Smith, *J. Am. Chem. Soc.*, **74**, 1556 (1952).

from the reactions of XXa and XXb. Apparently XXa was converted to its dicarbanion but not to a benzyne, and XXb was converted to a benzyne but not to a dicarbanion.[11] This resulted in recovery of XXa, and conversion of XXb to the heterocyclic product XXI.



XXa, R = C_6H_5
b, R = H



XXI

The cyclization of diketone VIII through its dicarbanion–benzyne to form IX is similar to that of diketone I to form II observed by Bunnett and Skorcz.[2c] Interestingly, the latter reaction also may involve the formation of a dicarbanion–benzyne intermediate. If so, this appears to be the first example of the less nucleophilic 3-position of such a dicarbanion reacting in preference to the more nucleophilic terminal position.[4] This would, of course, then be attributed to the greater ease of formation of a six-membered ring as compared with an eight-membered ring.

It should be mentioned that 4-(o-chlorophenyl)-2-butanone, which was formylated to form ketoaldehyde XII, was prepared by cleavage of diketone VIII,[12] rather than by the acetoacetic ester method employed previously.[2c] Formylation of the ketone in ethanol afforded a good yield of ketoaldehyde XII. Studies by Roch of similar systems indicated that formylation would be expected to occur at the methylene group rather than at the methyl group under these conditions.[13] The stability of the reaction product and the green color of the derived copper chelate confirmed that formylation had, in fact, occurred at the methylene position.[14]

Experimental[15]

3-(o-Chlorophenyl)-2,4-pentanedione (III).—Diketone III was prepared by the boron trifluoride method essentially as described for 3-phenyl-2,4-pentanedione.[16] From the reaction of a mixture of 122 g. (0.72 mole) of o-chlorophenylacetone,[2b] 110 g. (1.08 moles) of acetic anhydride, and 28 g. (0.15 mole) of p-toluenesulfonic acid was isolated 75 g. (43%) of the green copper chelate of 3-(o-chlorophenyl)-2,4-pentanedione (III), m.p. 258–264°, and m.p. 271–272° after recrystallization from benzene.

Anal. Calcd. for $C_{22}H_{20}O_4Cl_2Cu$: C, 54.72; H, 4.18. Found: C, 54.76; H, 4.18.

The chelate was decomposed with dilute hydrochloric acid to give 61.4 g. (40%) of 3-(o-chlorophenyl)-2,4-pentanedione (III), m.p. 44–49°, and m.p. 51–53° after recrystallization from cyclohexane–pentane.

Anal. Calcd. for $C_{11}H_{10}O_2Cl$: C, 62.76; H, 5.26; Cl, 16.83. Found: C, 62.65; H, 5.18; Cl, 17.01.

Cyclization of III to 1-Acetyl-2-indanone (VI).—In a 500-ml. inverse addition flask[17] was prepared a solution of 0.262 mole of potassium amide[4] from 10.2 g. of potassium in 400 ml. of commercial, anhydrous liquid ammonia. This solution was added over 10 min. to a solution of 10.52 g. (0.05 mole) of diketone III in 125 ml. of liquid ammonia and 125 ml. of anhydrous ether. During the course of the addition the reaction mixture changed from light amber through a transient green and finally to a yellow suspension. After 1 hr., ammonium chloride was added to neutralize the excess base. Ether was added and the ammonia was evaporated. Immediately dilute hydrochloric acid was added and the ether layer separated. Evaporation of the ether left 8.55 g. (98%) of 1-acetyl-2-indanone (VI) as a tan solid, m.p. 121.5–122.5°. Sublimation and recrystallization from ethanol gave m.p. 122.5–123.5°, lit.[7] m.p. 122°.

Anal. Calcd. for $C_{11}H_{10}O_2$: C, 75.84; H, 5.79. Found: C, 75.88; H, 5.84.

Diketone VI gave a blue enol test with ethanolic ferric chloride. Treatment of VI with hydrazine in refluxing ethanol gave pyrazole VII in 20% yield, m.p. 204–205° after two recrystallizations from ether–cyclohexane.

Anal. Calcd. for $C_{11}H_{10}N_2$: C, 77.62; H, 5.92; N, 16.46. Found: C, 77.62; H, 5.80; N, 16.41.

Diketone VI was independently synthesized from 26.4 g. (0.2 mole) of β-indanone[18] and 40.8 g. (0.4 mole) of acetic anhydride by means of the boron trifluoride–ethyl acetate complex prepared from 52.8 g. (0.6 mole) of the ester.[19] Sublimation afforded 12.0 g. (35%) of 1-acetyl-2-indanone (VI), m.p. 112–117°, and m.p. 122.5–123.5° after recrystallization from ethanol. A mixture melting point with diketone VI prepared from III was undepressed.

3-(o-Chlorobenzyl)-2,4-pentanedione (VIII).—A mixture of 100 g. (1.0 mole) of acetylacetone, 80.5 g. (0.05 mole) of o-chlorobenzyl chloride, 69 g. (0.05 mole) of potassium carbonate, and 250 ml. of absolute ethanol was refluxed gently on the steam bath for 1.5 hr. After the reaction mixture had cooled, sufficient water was added to dissolve the salts present, and the mixture was extracted with ether. The ethereal solution was distilled until a vapor temperature of 60° (0.5 mm.) was reached, and the residue was treated with aqueous cupric acetate to form 62.5 g. (53%) of the green copper chelate of 3-(o-chlorobenzyl)-2,4-pentanedione (VIII), m.p. 209–210°. The chelate was treated with dilute hydrochloric acid, and the diketone extracted into ether. Distillation afforded a good recovery of diketone VIII, b.p. 121–123° (0.7 mm.).

Anal. Calcd. for $C_{12}H_{13}O_2Cl$: C, 64.14; H, 5.83; Cl, 15.79. Found: C, 64.27; H, 5.86; Cl, 15.57.

Diketone VIII underwent crystallization under varying conditions to give a wide range of melting points from below room temperature to 69°. Different crops showed slight differences in the infrared spectrum, but all gave violet enol tests with ethanolic ferric chloride.

Treatment with cupric acetate gave a good yield of the green copper chelate, m.p. 223–225° after recrystallization from ethanol.

Anal. Calcd. for $C_{24}H_{24}O_4Cl_2Cu$: C, 56.24; H, 4.73; Cl, 13.88; Cu, 12.44. Found: C, 56.23; H, 5.23; Cl, 14.15; Cu, 12.30.

Treatment of diketone VIII with refluxing ethanolic hydrazine afforded in good yield 4-(o-chlorobenzyl)-3,5-dimethylpyrazole, m.p. 126–127° after recrystallization from ethanol.

Anal. Calcd. for $C_{12}H_{13}N_2Cl$: C, 65.30; H, 5.93; Cl, 16.07; N, 12.70. Found: C, 65.19; H, 5.81; Cl, 15.95; N, 12.72.

Cyclization of VIII to 3-Acetyl-2-tetralone (IX).—To a solution of 12.3 g. (0.056 mole) of diketone VIII in 250 ml. of ammonia was gradually added a solution of 0.33 mole of potassium amide (prepared from 13.0 g. of potassium) in 500 ml. of liquid ammonia. After 45 min., ammonium chloride was added to the green suspension which had formed in order to neutralize the excess base. The ammonia was evaporated and the residue was taken up in dilute hydrochloric acid and ether. The ethereal layer was separated and evaporated. The remaining oil was treated with ethanol to afford 6.7 g. (65%) of 3-acetyl-2-tetralone (IX), m.p. 45–49°, and m.p. 48–49° after recrystallization from ethanol.

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[11] For evidence that the related phenylacetone affords the methylene monocarbanion but not appreciable dicarbanion under similar conditions, see T. M. Harris and C. R. Hauser, *J. Am. Chem. Soc.*, **81**, 1154 (1959).

[12] It was found convenient not to isolate VIII. Thus, 4-(o-chlorophenyl)-2-butanone was isolated in 78% yield on refluxing a mixture of o-chlorobenzyl chloride, acetylacetone, and potassium carbonate in ethanol for 16 hr. The generality of this method is being investigated.

[13] L. M. Roch, *Ann. chim.* (Paris), **6**, 105 (1961).

[14] See T. M. Harris, S. Boatman, and C. R. Hauser, *J. Am. Chem. Soc.*, **85**, 3273 (1963).

[15] Melting points were taken on a capillary melting point apparatus and are uncorrected. Ultraviolet spectra were determined in 95% ethanol solution with a Cary 14 spectrophotometer. Elemental analyses and the molecular weight were by Galbraith Laboratories, Knoxville, Tenn.

[16] C. R. Hauser, F. W. Swamer, and J. T. Adams, *Org. Reactions*, **8**, 133 (1954).

[17] See C. R. Hauser, W. R. Brasen, P. S. Skell, S. W. Kantor, and A. E. Brodhag, *J. Am. Chem. Soc.*, **78**, 1653 (1956).

[18] J. E. Horan and R. W. Schiessler, *Org. Syn.*, **41**, 53 (1961).

[19] See ref. 16, pp. 131–132.

Anal. Calcd. for $C_{12}H_{12}O_2$: C, 76.57; H, 6.43. Found: C, 76.53; H, 6.21.

Diketone IX gave a red-violet enol test with ferric chloride. Oxidation of IX with alkaline potassium permanganate gave phthalic acid in 76% yield.

Treatment of diketone IX with refluxing ethanolic hydrazine gave a good yield of pyrazole XI, m.p. 173.5–174° after recrystallization from benzene–hexane.

Anal. Calcd. for $C_{12}H_{12}N_2$: C, 78.23; H, 6.57; N, 15.21. Found: C, 78.30; H, 6.67; N, 15.22.

A sample of diketone IX was refluxed with aqueous sodium hydroxide under a nitrogen atmosphere to give β-tetralone, which was isolated as its semicarbazone, m.p. 191–192° after three recrystallizations from ethanol, lit.[20] m.p. 193–194°. The odor of acetic acid could be detected in the acidified hydrolysate. When the hydrolysis was not conducted in a nitrogen atmosphere, the blue-black color, characteristic of the base-catalyzed air oxidation of β-tetralone,[21] was detected.

2-(*o*-Chlorobenzyl)-1,3-butanedione (XII).—To a cooled solution of 1.0 mole of sodium ethoxide (prepared from 23 g. of sodium) in 500 ml. of absolute ethanol was added a mixture of 172 g. (0.94 mole) of 4-(*o*-chlorophenyl)-2-butanone[12] and 80.0 g. (1.08 moles) of ethyl formate.[13] The solution was stirred at room temperature for 16 hr. Ethanol was removed under reduced pressure. To the residue was added ice–water, followed by 100 ml. of hydrochloric acid, and finally 500 ml. of ether. Crystals of XII (74 g., m.p. 120–122°) formed in the mixture, and were removed by filtration. The ethereal layer of the filtrate was separated and evaporated to a small volume to give an additional 42 g. of XII, m.p. 107–117°. The total yield of 2-(*o*-chlorobenzyl)-1,3-butanedione (XII) was 116 g. (58%). Recrystallization from methanol afforded crystals, m.p. 122–123°.

Anal. Calcd. for $C_{11}H_{11}ClO_2$: C, 62.76; H, 5.26; Cl, 16.83. Found: C, 62.69; H, 5.08; Cl, 17.04.

The green copper chelate of XII, m.p. 202–203° after recrystallization from benzene, was prepared by treatment with aqueous cupric acetate.

Anal. Calcd. for $C_{22}H_{20}Cl_2O_4Cu$: C, 54.72; H, 4.18; Cl, 14.69; Cu, 13.16. Found: C, 55.06; H, 4.10; Cl, 15.00; Cu, 13.46.

Formation of Binaphthol XV from XII.—To a solution of 13.2 g. (0.063 mole) of β-ketoaldehyde XII in 125 ml. of liquid ammonia and 125 ml. of anhydrous ether was added 0.264 mole of potassium amide (prepared from 10.3 g. of potassium) in 300 ml. of liquid ammonia. After 1 hr., ammonium chloride was added to the dark green solution to neutralize excess base and the ammonia was evaporated. The residue was extracted with dilute hydrochloric acid and ether. The ethereal solution was separated and evaporated. The residual solid was washed with benzene to leave 3.1 g. (29%) presumably of 2,2′,α,α′-tetrahydroxy-3,3′-dimethyl-1,1′-binaphthyl (XV), m.p. 216°, and m.p. 231–232° after chromatography on silica gel and recrystallization from ethyl acetate.

Anal. Calcd. for $C_{22}H_{18}O$: C, 75.84; H, 5.79; mol. wt., 346. Found: C, 76.03, 75.94; H, 5.07, 5.08; mol. wt., 356.[22]

This compound gave an enol test with ethanolic ferric chloride, but neither a copper chelate nor a pyrazole could be prepared.

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[20] N. M. Przhiyalgovskaya, L. N. Lavrisheva, and V. N. Belov, *J. Gen. Chem. USSR*, **27**, 1349 (1957).

[21] J. W. Cornforth, R. H. Cornforth, and R. Robinson, *J. Chem. Soc.*, 689 (1942).

[22] The molecular weight was determined at 37° in acetone solution employing a Mechrolab osmometer.

Benzylation and Dehydrogenation of 3-Phenylindan-1-one through Its α,β-Dicarbanion. Synthesis of 3-Phenylindenone[1]

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3-Phenylindan-1-one (I) underwent a twofold acid–base reaction with two molecular equivalents of sodium amide in liquid ammonia to form α,β-disodio salt II, which underwent alkylation with benzyl chloride to give β-benzyl derivative III. The monosodio salt of I was benzylated to afford the α-benzyl derivative V. III was reduced to give 1-benzyl-1-phenylindane. Dehydrogenation of I through its disodio salt II was effected with 2,3-dibromo-2,3-dimethylbutane to form 3-phenylindenone. Similar dehydrogenations of 2,3-diphenylindanone and 2,3,3-triphenylpropionitrile were effected through their dicarbanions to yield 2,3-diphenylindenone and 2,3,3-triphenylacrylonitrile, respectively. Possible mechanisms for this new method of dehydrogenation are mentioned.

Since 3-phenylindan-1-one (I) has not only α-hydrogens but also a β-hydrogen activated by two aryl groups, it might be expected to undergo a twofold acid–base reaction with sodium amide in liquid ammonia to form α,β-disodio salt II. This was realized as demonstrated not only by production of a deep red color like that of the diphenylmethide ion[2] but also by alkylation with a molecular equivalent of benzyl chloride to form β-benzyl derivative III in 73% yield (Scheme A).

Similar to the benzylation of sodium diphenylmethide in liquid ammonia,[2] that of disodio salt II was rapid as indicated by immediate discharge of the red color by the equivalent of halide.

That the conversion of I to II as represented in Scheme A was essentially complete was indicated not only by the high yield of III but also by the lack of formation of an isolable amount of stilbene, some of which would probably have been produced had there been an appreciable amount of amide ion present in the equilibrium.[3]

Although disodio salt II was generally prepared by addition of I to the reagent, the inverse addition procedure was also employed to show the intermediate formation of monosodio salt IV. Thus, when sodium amide in liquid ammonia was added slowly to I in ether the yellow-brown color of IV persisted until slightly more than a molecular equivalent had been added; then the red color of II became evident and deepened until addition of the second equivalent of the base was complete.

The product from the benzylation of II was shown *not* to be the possible α-benzyl derivative V, which was prepared from monosodio salt IV and benzyl chloride in diglyme (β,β′-dimethoxydiethyl ether) at 100°. Incidentally, IV failed to afford an isolable amount of

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[2] C. R. Hauser and P. J. Hamrick, *J. Am. Chem. Soc.*, **79**, 3142 (1957).

[3] The stilbene would have arisen through self-alkylation of the benzyl chloride; see C. R. Hauser, W. R. Brasen, P. S. Skell, S. W. Kantor, and A. E. Brodhag, *ibid.*, **78**, 1653 (1956).