Metal-Ammonia Reduction of Triptycene and Related Benzobarrelene Derivatives

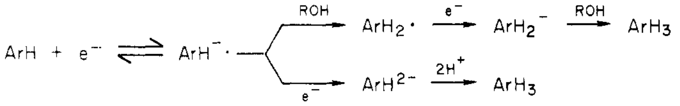
Peter W. Rabideau,* Donald W. Jessup, Jay W. Ponder, and George F. Beekman

Department of Chemistry, Indiana-Purdue University at Indianapolis, Indianapolis, Indiana 46205

Received May 14, 1979

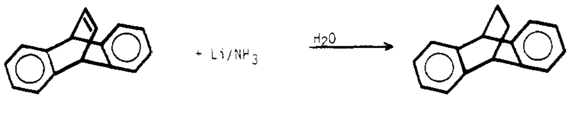
This paper describes the metal-ammonia reduction of several bicyclic compounds including dibenzobarrelene, triptycene, and symmetrical and unsymmetrical benztriptycenes. Although previous studies indicate that triptycene undergoes ring opening on reaction with potassium in THF, successful reduction in ammonia has been accomplished for all of these compounds with selection of proper conditions. Triptycene exhibits rather unusual regioselectivity in that the major product is the 2,4a-dihydro isomer with the expected 1,4-dihydro isomer produced in lesser amounts. This unanticipated result is discussed in terms of π-π conjugation in the intermediate radical anion (or dianion). As might be expected, the reduction of isomeric benztriptycenes appears to be dominated by the naphthalene moiety and results in both inner-ring and outer-ring reduction products. Furthermore, 1,2-benztriptycene shows a novel metal effect in that lithium produces ring cleavage whereas the use of sodium provides "normal" reduction products.

Electron addition to aromatic compounds in anhydrous liquid ammonia has provided an important method (Birch reduction) for the reduction of aromatic compounds. In the case of benzene derivatives, a radical anion is formed which may be protonated by alcohols to shift the unfavorable equilibrium (eq 1, upper path), whereas polynuclear aromatics can undergo a second electron addition resulting in a dianion (eq 1, lower path), which may then be protonated (or alkylated).[1] Hence, most interests have focused on polynuclear compounds, and much less attention has been given to polycyclic compounds which do not contain fused aromatic rings.

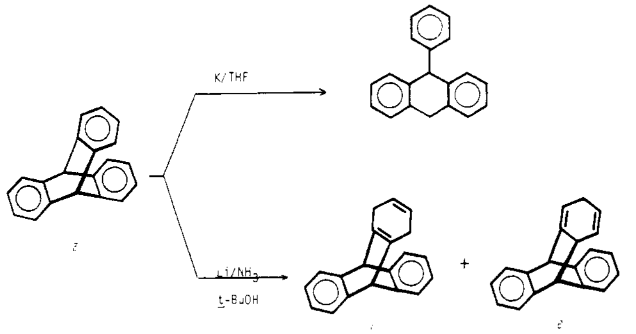


$$ArH + e^- \rightleftharpoons ArH^{-}\cdot \begin{cases} \xrightarrow{ROH} ArH_2\cdot \xrightarrow{e^-} ArH_2^- \xrightarrow{ROH} ArH_3 \\ \xrightarrow[e^-]{} ArH^{2-} \xrightarrow{2H^+} ArH_3 \end{cases}$$

Our interest in the metal-ammonia reduction of barrelene derivatives developed some time ago with the discovery[2] that dibenzobarrelene (1) reacts with lithium in ammonia to furnish the dihydro product 2. This must be regarded as unusual for the following reasons: (1) "isolated" double bonds are considered to be unreactive toward alkali metals in ammonia[1] (e.g., bicyclo[2.2.2]octene is completely unreactive under these conditions), and (2) alkali metal/ammonia solutions even fail to reduce benzene to any appreciable extent if water is used as a protonating agent (i.e., alcohols are necessary to shift the initial equilibrium).[1]



In an attempt to determine whether or not unusual reduction behavior might be common in barrelene derivatives, we chose to investigate the reaction of triptycene (3; i.e., tribenzobarrelene), although it had been reported that 3 gives only ring opening (i.e., 4) upon reaction with potassium in THF.[3] We considered the possibility that the reaction might take an entirely different course in ammonia. Accordingly, reaction with lithium/ammonia/*tert*-butyl alcohol does not produce ring cleavage but results in an 80% conversion to a product affording only one peak by GLC on several columns ($C_{20}H_{16}$ by mass spectroscopy and elemental analysis). NMR, however, suggested the presence of two isomers (2:1) since the reduction product of triptycene-*9,10-d*$_2$ indicated at least three distinct bridgehead protons. Ultimately, high-pressure liquid chromatography allowed a reasonable separation of two isomers assigned as 5 and 6 on the following basis. One of these $C_{20}H_{16}$ isomers showed equivalent bridgehead protons and only two olefinic protons in the NMR, and this narrows the possibilities to either a 1,4-dihydro or a 2,3-dihydro isomer. The chemical shift (δ 2.85) of the allylic protons is consistent only with the doubly allylic system, and structure 5 was assigned. The predominant isomer showed two bridgehead and three olefinic protons and could only be a 4,4a-dihydro or 2,4a-dihydro isomer. Once again, the chemical shift data suggest doubly allylic protons (δ 2.55), and this isomer was assigned as structure 6. Additional evidence supporting both assignments lies in the fact that the UV spectrum of the purified mixture of 5 and 6 did not indicate a 1,3-cyclohexadiene moiety for either of these isomers.



The metal-ammonia reduction of triptycene proved to be quite sensitive to reaction conditions. The use of calcium or potassium resulted mainly in the recovery of starting material with no detectable amounts of 5 and 6, whereas sodium afforded these products in only minor amounts. However, the use of excess lithium was found to give over 80% reduction when coupled with the use of

---

[1] (a) Birch, A. J.; Rao, G. Subba "Advances in Organic Chemistry, Methods and Results"; Taylor, E. C., Ed.; Wiley-Interscience: New York, 1972. (b) Smith, H. "Chemistry in Nonaqueous Ionizing Solvents"; Jander, G., Spandau, H., Addison, C. C., Eds.; Interscience: New York, 1963; Vol. I, Part 2. (c) Harvey, R. G. *Synthesis* **1970**, 161.

[2] (a) Rabideau, P. W.; Jessup, D. W. Abstracts of Papers, 8th Annual Great Lakes Regional Meeting of the American Chemical Society, West Lafayette, IN, 1974. (b) Butler, D. N.; Koves, G. *Synth. Commun.* **1975**, *5*, 471.

[3] (a) Theilacker, W.; Mollhoff, E. *Angew. Chem., Int. Ed. Engl.* **1962**, *1*, 596. (b) Walsh, T. D.; Ross, R. T. *Tetrahedron Lett.* **1968**, 3123.

0022-3263/79/1944-4594$01.00/0

Table I. Metal–Ammonia Reduction of Triptycene[a]

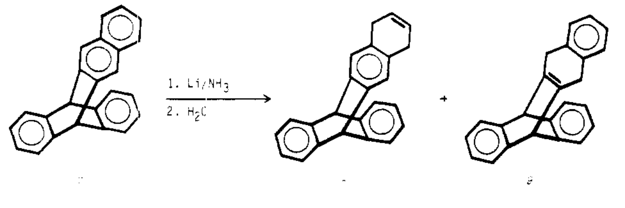
metal	proton source	% yield 5^b	 6^b	 unreacted 3	ratio 6/5
Li	$(CH_3)_3COH$	27	55	18	2.0
Li	$(CH_3)_2CHOH$	23	53	25	2.3
Li	$CH_3CH_2OH^{c,d}$	18	33	49	1.8
Li	CF_3CH_2OH	16	22	62	1.4
Li	$(CF_3)_2CHOH$	10	9	81	0.9
Na	$(CH_3)_3COH$	6	11	83	1.8

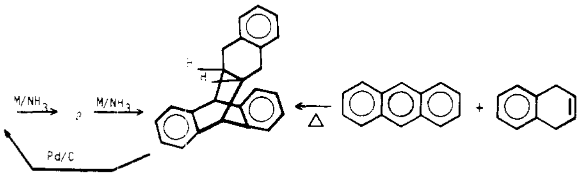
[a] Reactions run at −33 °C in THF/NH_3 (1:1.5) with 3.5 g-atoms of metal. [b] Determined by integrated areas of NMR spectra. [c] Identical results with rapid or dropwise addition. [d] Identical results when run at −78 °C.

THF as a cosolvent and *tert*-butyl alcohol as the protonating agent. In addition, there appeared to be considerable variation in the ratio of reduction products, as well as yield, with the nature of the protonating agent.

Thus, while improved yields with *tert*-butyl alcohol could be rationalized on the basis of its decreased acidity, it was not clear whether the increase in isomer **6** (relative to **5**) was also a function of acidity, or rather the larger *t*-BuOH might be producing more **6** due to steric effects encountered for protonation at the 1-position which leads to **5**. In an attempt to make this determination, the reaction was carried out with a variety of alcohols, and the results are summarized in Table I. There is a definite trend toward decreased yields as the acidity of the protonating agent increases, and, in fact, when water is used as a quench, very little reduction is observed at all. Furthermore, the formation of isomer **6** also appears to be related to acidity and not steric effects, since its yield drops with substitution of fluorine for hydrogen (with both ethanol and 2-propanol) which greatly increases acidity with presumably little effect on steric size. Hence, we conclude that the more acidic alcohols (and water) consume metal too rapidly (relative to protonation of the radical anion) which lowers the yield, and the more weakly acidic alcohols become more discriminate in protonation site, resulting in an increase in **6**.

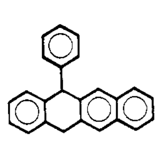
In an attempt to determine the effect of annelation on the triptycene system, 2,3-benztriptycene (**7**) was investigated, and reduction with lithium metal followed by a water quench resulted in two products. Compound **8** could be obtained in pure form (see below), and its structure assignment was straightforward on the basis of NMR. Compound **9** could not be separated from **8** and unreacted **7** by either fractional crystallization or gas chromatography, and its NMR was determined by difference. However, further evidence for this structure is provided by the fact that the use of excess metal in the reduction of **7** leads to the tetrahydro product which served as an intermediate in the preparation of **7** itself.

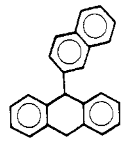


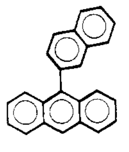


It is not surprising that the naphthalene moiety dominates reduction in this case, and in view of the ESR results with the 2,3-naphthobarrelene radical anion,[4] it is expected that considerable electron density should result in both of the naphthalene rings. Interestingly, the use of *t*-BuOH as a proton source results only in the formation of **8** without the production of **9**. Once again, less discrimination in protonation sites is observed with stronger proton sources. Similar behavior has been observed for *p*-terphenyl which gives both outer- and inner-ring reduction as the acidity of the protonating agent is increased.[5]

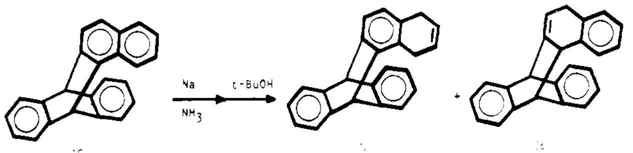
Interestingly, reduction of the isomeric 1,2-benztriptycene (**10**) with lithium/ammonia resulted in ring opening regardless of whether water or *t*-BuOH is used as a proton source: this represents the only barrelene derivative that we have investigated which has undergone ring opening in ammonia. In principle, ring opening could occur in two ways, with phenyl cleavage to form **11** or naphthyl cleavage to afford **12**. Unfortunately, little could be determined from the NMR spectrum except the presence of three benzylic protons and a very complex aromatic region. However, the product could be readily dehydrogenated over Pd/C to afford **13**, a known compound.





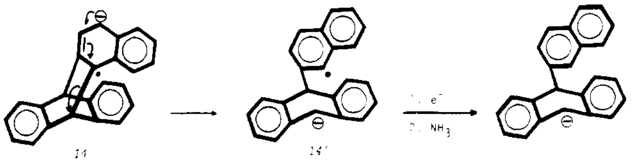


In contrast, when sodium is used for the reduction of **10**, *ring cleavage does not occur*, but two dihydro products **15** and **16** are produced. Hence, the reduction behavior of **10** represents a rather unusual pattern in view of different pathways with lithium and sodium, and this warrants some attempt at explanation.



10

We cannot readily determine whether ring opening proceeds through the initial formation of an "intact" radical anion such as **14** (only one resonance structure shown for illustrative purposes) or whether either **14** or **14′** results from a common transition state. In either case, **14′** is expected to be the more highly delocalized species due to the dihydroanthracenyl type anion.[6] Since lithium tends toward greater separation of ion pairs as compared with sodium, it follows that the structure with greater charge delocalization (i.e., **14′**) could be more important when lithium is the counterion.[7]



14
14′

The difference in the behavior of **7** and **10** remains as somewhat of a puzzle. However, there may be a consid-

---

[4] Dodd, J. R.; Sanzero, G. V. *J. Am. Chem. Soc.* **1975**, *97*, 4917.

[5] Harvey, R. G.; Lindow, D. F.; Rabideau, P. W. *J. Am. Chem. Soc.* **1972**, *94*, 5412.

[6] The relative positions of the radical and anion were deduced on the basis of ref 3b.

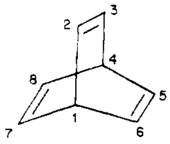
[7] Szwarc, M., Ed. "Ions and Ion Pairs in Organic Reactions"; Wiley-Interscience: New York, 1972.

erable difference in the distribution of charge densities in the radical anions derived from **7** and **10**. ESR studies with 2,3-naphthobarrelene,[4] for example, suggest considerable spin density in the unsubstituted ring, and this sort of effect may tend to keep the radical anion of **7** intact (see also ref 3b). In any event, factors which affect ring-opening processes in these and related systems certainly warrant further investigation.

Discussion

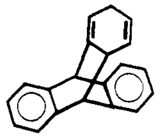
In considering the behavior of **1**, we note that the reduction of a bridged ethylene has been previously reported, and the conversion of norbornadiene to norbornene by the action of alkali metals in ammonia was attributed to the intermediacy of a nonclassical radial ion (**17**).[8,9] Subsequent theoretical considerations of similar topologies[10] lead to the classification of the probable intermediate as a homoaromatic radical anion or dianion (i.e., **18**). More recently bishomoconjugative stabilization was reported for the related carbanions **19** and **20**, although the presence of a third center in **20** does not result in substantial additional stability due to longicyclic stabilization (bicycloaromaticity).[11]

Most recently, barrelene itself (**21**) has been investigated in terms of the nature and extent of $\pi-\pi$ conjugation, and the first theoretical calculations (semiempirical SCF-C1-MO treatment) that reproduce the observed UV spectrum have been obtained.[12] In this case best results seem to be achieved by a geometry providing slight increases in bond lengths. Further support for this approach was obtained by force-field calculations which did indeed suggest a lower energy for this geometry. The results of this study indicate that through-space $\pi-\pi$ interactions as well as through-bond $\pi-\pi$ interactions are necessary to account for the UV spectrum of barrelene. This latter interaction must, of course, be transmitted through the σ framework.

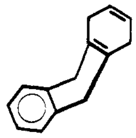


Thus, interaction of $C_5-C_6(\pi)$ with $C_7-C_8(\pi)$ is transmitted through $C_1-C_2(\sigma)$ and $C_4-C_3(\sigma)$, etc. This is, of course, consistent with the concept developed by Hoffmann[13] for $\pi-\pi$ interactions both through space and through bonds. It should also be noted that through-bond $\pi-\pi$ conjugation is accompanied by a weakening of the σ framework.[14] This may account in part for the ring-opening reactions observed for triptycene derivatives.

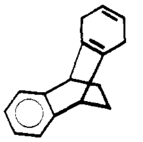
Thus, we expect significant $\pi-\pi$ orbital interactions with the compounds studied herein. Furthermore, the particular topology associated with the barrelene system should provide for a stabilizing interaction in the radical anion or dianion intermediates when all three π systems are involved.[10] This effect does appear to be important in this case since regioselectivity appears to depend on the third π bridge. Thus, in comparing the reduction products of triptycene, 9,10-dihydroanthracene, and 9,10-ethano-9,10-dihydroanthracene (**6**, **21**, and **22**, respectively), one observes that only triptycene which possesses a third π center provides any 2,4a-type reduction (i.e., **6**, as the major product). In the latter two cases, only **21** and **22** were detected (respectively) with no evidence for other dihydro products.



6



21



We conclude, therefore, that all three rings must be involved in the metal-ammonia reduction of triptycene and that this effect is responsible for the product distribution.

Experimental Section

General Methods. Metal-ammonia reductions were carried out as follows. The hydrocarbon was dissolved in 1 part of THF (distilled from $LiAlH_4$) and added to 1.5 parts of anhydrous ammonia under helium. The metal was then added in pieces, and the reaction was stirred at either -33 or -78 °C for about 20 min. A proton source was then added until the solution lost color followed by the addition of water and isolation by ether/water partition.

9,10-Ethano-9,10-dihydroanthracene (2). Dibenzobarrelene[15] (1.5 g, 7 mmol) was reacted with lithium (18 mmol) in 100 mL of NH_3 at -33 °C according to the general procedure, followed by quenching with water to afford **2**[16] in nearly quantitative yield: mp 138-140 °C; NMR ($CDCl_3$) δ 7.1 (m, 8 H), 4.3 (br s, 2), 1.7 (br s, 4).

9,10-Ethano-1,4,9,10-tetrahydroanthracene (22). The above product (**2**; 0.5 g, 2.4 mmol) was reacted with lithium (8.5 mmol) in 75 mL of NH_3 at -33 °C as above. After 30 min, dry *t*-BuOH was added quickly, and workup produced a white solid. Recrystallization from CCl_4 afforded **22** in 70% yield: mp 71-73 °C; NMR ($CDCl_3$) δ 7.0 (br s, 4), 5.6 (br s, 2), 3.55 (br s, 2), 2.8 (br s, 4), 1.5 (m, 4).

Anal. Calcd for $C_{16}H_{16}$: C, 92.26; H, 7.74. Found: C, 91.96; H, 7.98.

1,4- and 2,4a-Dihydrotriptycene (5 and 6). Triptycene was reduced under a variety of conditions as described in Table I. Separation of these isomers could not be achieved by standard methods, including GLC on at least ten different columns (partial separation in some cases), and elemental analysis was performed on the mixture of isomers. However, high-pressure LC with recycling on two 2-ft μ-Porasil columns provided substantial separation, and the NMR spectra were determined: **5**, δ 7.1 (m, 8), 5.8 (br s, 2), 4.65 (s, 2), 2.9 (br s, 4); **6**, 7.1 (m, 8), 5.7 (m, 3), 4.75 (s, 1), 4.3 (br s, 1), 2.55 (m, 3).

Anal. Calcd for $C_{20}H_{16}$: C, 93.74; H, 6.26. Found: C, 93.44; H, 6.12.

---

[8] (a) Ortiz de Montellano, B. R.; Loving, B. A.; Shields, T. C.; Gardner, P. D. *J. Am. Chem. Soc.* **1967**, *89*, 3365. (b) Birch, A. J.; Rao, G. Subba *Adv. Org. Chem.* **1972**, *8*, 17.

[9] See also ref 2b which includes additional examples and: Paddon-Row, M. N.; Butler, D. N.; Warrener, R. N. *J. Chem. Soc., Chem. Commun.* **1976**, 741.

[10] (a) Goldstein, M. J. *J. Am. Chem. Soc.* **1967**, *89*, 6357. (b) Goldstein, M. J.; Hoffmann, R. *Ibid.* **1971**, *93*, 6193.

[11] (a) Moncur, M. V.; Grutzner, J. B. *J. Am. Chem. Soc.* **1973**, *95*, 6449. (b) Goldstein, M. J.; Natowsky, S. *Ibid.* **1973**, *95*, 6451. See also: Grutzner, J. B.; Winstein, S. *Ibid.* **1972**, *94*, 2200.

[12] Meyer, A. Y.; Pasternak, R. *Tetrahedron* **1977**, *33*, 3239.

[13] Hoffmann, R. *Acc. Chem. Res.* **1971**, *4*, 1.

[14] Gleiter, R. *Angew. Chem., Int. Ed. Engl.* **1974**, *13*, 696.

[15] Rabideau, P. W.; Hamilton, J. B.; Friedman, L. *J. Am. Chem. Soc.* **1968**, *90*, 4465.

[16] Birnbaum, N.; Cookson, R. C.; Lewin, N. *J. Chem. Soc.* **1961**, 1224.

2a,3a-Dihydro-2,3-benztriptycene (8). 2,3-Benztriptycene[17] (0.25 g) was reduced with lithium according to the general procedure with *t*-BuOH as the proton source. This was a difficult reaction to carry out cleanly, but the use of somewhat lesser amounts of lithium (1.1 equiv) afforded a product consisting primarily of 8 together with unreacted starting material. Recrystallization from acetone–hexane provided 8: mp 209 °C; NMR ($CDCl_3$) δ 6.9 (m, 10), 5.7 (br s, 2), 5.2 (s, 2), 3.2 (br s, 4).

Anal. Calcd for $C_{24}H_{18}$: C, 94.08; H, 5.92. Found: C, 93.87; H, 5.90.

1,4-Dihydro-2,3-benztriptycene (9). When the above reaction was quenched with H_2O instead of *t*-BuOH, 8 and 9 were formed in a ratio of 1:2 together with $\sim$30% unreacted starting material. Purification of 9 was not possible, and the NMR spectrum was determined by subtracting out those of 7 and 8: NMR ($CDCl_3$) δ 6.9 (m, 12), 4.75 (s, 2), 3.5 (s, 4).

9-(2-Naphthyl)-9,10-dihydroanthracene. 1,2-Benztriptycene[17] (0.75 g, 2.5 mmol) was reacted with lithium metal (6.2 mmol) as usual with a *t*-BuOH quench. Recrystallization of the crude product from ethanol gave white crystals of 12: mp 128–129 °C (0.4 g, 53%); NMR ($CDCl_3$) δ 7.2 (m, 15), 5.35 (s, 1), 4.0 (br s, 2).

Anal. Calcd for $C_{24}H_{18}$: C, 94.08; H, 5.92. Found: C, 93.84; H, 6.07.

Dehydrogenation of 12 (0.2 g) with 5% Pd/C (0.12 g) at 350 °C for 1.5 h provided 9-(2-naphthyl)anthracene, mp 200–202 °C.[18]

5,8-Dihydro-1,2-benztriptycene (15). When the above reaction was carried out by using sodium metal at −78 °C, a mixture of 15 and 16 resulted. Two recrystallizations from ethanol provided 15 in 40% yield: mp 211–213 °C; NMR ($CDCl_3$) δ 7.0 (m, 10), 5.8 (br s, 2), 5.5 (br s, 1), 5.25 (s, 1), 3.4 (m, 4).

Anal. Calcd for $C_{24}H_{18}$: C, 94.08; H, 5.92. Found: C, 93.79; H, 6.15.

1,4-Dihydro-1,2-benztriptycene (16). This compound could not be obtained free from 15, but the NMR spectrum was determined by subtracting out the spectrum of 15: NMR ($CDCl_3$) δ 7.0 (m, 12), 5.9 (br s, 1), 5.0 (br s, 1), 4.8 (s, 1), 3.1 (br s, 3).

Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for partial support of this research and to the U.S. Department of Energy, Office of Basic Energy Sciences.

Registry No. **1**, 2734-13-6; **2**, 5675-64-9; **3**, 477-75-8; **5**, 71870-39-8; **6**, 71870-40-1; **7**, 13395-89-6; **8**, 71870-41-2; **9**, 71870-42-3; **10**, 69096-77-1; **12**, 71870-43-4; **13**, 7424-72-8; **15**, 71870-44-5; **16**, 71870-45-6; **22**, 71870-46-7.

---

(17) Hurd, C. D.; Juel, L. H. *J. Am. Chem. Soc.* **1955**, *77*, 601.

(18) Vingiello, F. A.; Borkovec, A.; Zajac, W., Jr. *J. Am. Chem. Soc.* **1958**, *80*, 1714.

Alkylations and Acylations of α-Aryl-4-morpholineacetonitriles (Masked Acyl Anion Equivalents) and Their Use in 1,4-Additions

Francis J. McEvoy and J. Donald Albright*

American Cyanamid Company, Medical Research Division, Lederle Laboratories, Pearl River, New York 10965

Received June 7, 1979

The anions of α-aryl-4-morpholineacetonitriles are alkylated with ethyl bromoacetate, epichlorohydrin, or allyl chloride and displace halogen on benzene derivatives containing electron-withdrawing groups to give benzophenones. These anions are acylated with ethyl chloroformate and benzoyl chlorides and add to ethyl acrylate and acrylonitrile to give good yields of 1,4-addition products.

Discussion

The use of masked functional groups such as masked acyl anion equivalents in the formation of carbon–carbon bonds has proved to be a powerful strategy in the development of new synthetic methods.[1] Stetter has studied the addition of aryl aldehydes to α,β-unsaturated esters, ketones, and nitriles by the use of anions from intermediate cyanohydrins.[2] The utility of O-alkylated cyanohydrins[3,4] and O-silylated cyanohydrins[5] in the synthesis of ketones has been reported. Related masked acyl anion equivalents are those derived from α-aryl-α-(dialkylamino)acetonitriles.[6] Alkylations with benzyl halides have been studied[6a,7] and Leete reported[8] the 1,4-addition of α-(3-pyridyl)-4-morpholineacetonitrile to acrylonitrile.

We have found that diverse α-aryl-4-morpholineacetonitriles add to acrylonitrile and ethyl acrylate to give good yields of 1,4-addition products.[9] With ethyl acrylate the yields are superior to those obtained from aryl-aldehyde cyanohydrins,[2,6] and with acrylonitrile yields are at least equivalent. The addition of α-aryl-4-morpholineacetonitriles to ethyl acrylate followed by hydrolysis of the masked acyl function is a useful high-yield route to ethyl 3-aroylpropionates[10] and 3-aroylpropionitriles (see Table III).

---

(1) (a) Seebach, D. *Angew. Chem., Int. Ed. Engl.* **1969**, *8*, 639. (b) Seebach, D.; Kolb, M. *Chem. Ind. (London)* **1974**, 687. (c) Lever, O. W., Jr. *Tetrahedron* **1976**, *32*, 1943. (d) Seebach, D.; Burstinghaus, R. *Angew. Chem., Int. Ed. Engl.* **1975**, *14*, 57. (e) Stetter, H.; Kuhlmann, H. *Ibid.* **1974**, *13*, 539. Stork, G.; Ozorio, A. A.; Leong, A. Y. W. *Tetrahedron Lett.* **1978**, 5175.

(2) Stetter, H. *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 639 and references therein.

(3) Stork, G.; Maldonado, L. *J. Am. Chem. Soc.* **1971**, *93*, 5286.

(4) Kalir, A.; Balderman, D. *Synthesis* **1973**, 358.

(5) Hunig, S.; Wehner, G. *Synthesis* **1974**, 180.

(6) (a) Hauser, C. R.; Taylor, H. M.; Ledford, T. G. *J. Am. Chem. Soc.* **1960**, *82*, 1786. Morris, G. F.; Hauser, C. R. *J. Org. Chem.* **1961**, *26*, 4741. Hauser, C. R.; Morris, G. F. *Ibid.* **1961**, *26*, 4740. (b) Bennett, D. J.; Kirby, G. W.; Moss, V. A. *Chem. Commun.* **1967**, 218; *J. Chem. Soc. C* **1970**, 2049.

(7) Dyke, S. F.; Tiley, E. P.; White, A. W. C.; Gale, D. P. *Tetrahedron* **1975**, *31*, 1219.

(8) (a) Leete, E.; Chedekel, M. P.; Boden, G. B. *J. Org. Chem.* **1972**, *37*, 4465. (b) Leete, E.; Boden, G. B. *J. Am. Chem. Soc.* **1976**, *98*, 6321.

(9) Albright, J. D.; McEvoy, F. J.; Moran, D. B. *J. Heterocycl. Chem.* **1978**, *15*, 881.

(10) McEvoy, F. J.; Allen, G. R. *J. Org. Chem.* **1973**, *38*, 4044.

0022-3263/79/1944-4597$01.00/0