
A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

# Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

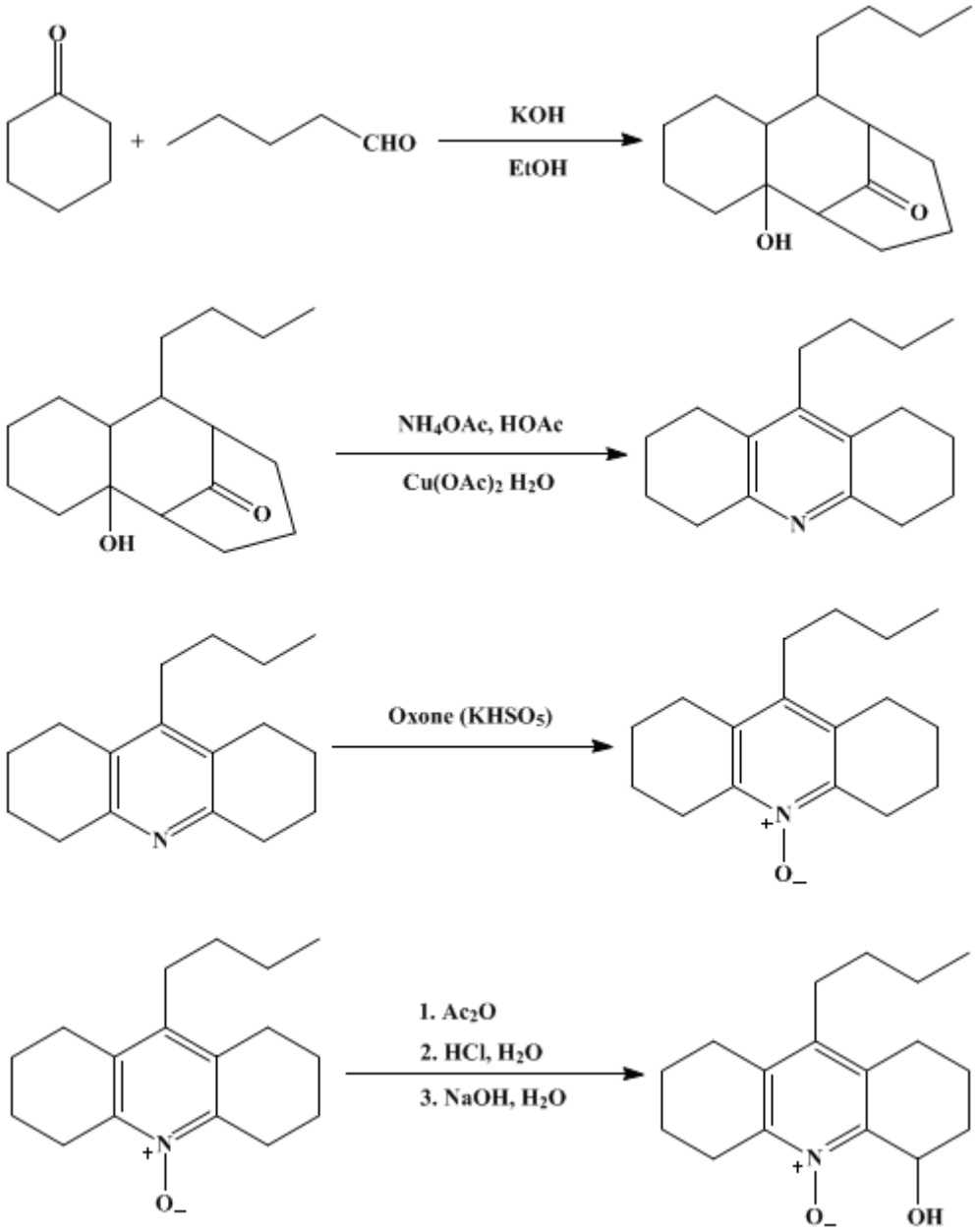
*These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

DOI:10.15227/orgsyn.069.0226

*Organic Syntheses, Coll. Vol. 8, p.87 (1993); Vol. 69, p.226 (1990).*

# 9-*n*-BUTYL-1,2,3,4,5,6,7,8-OCTAHYDROACRIDIN-4-OL

**[4-Acridinol; 9-butyl-1,2,3,4,5,6,7,8-octahydro]**




Submitted by Thomas W. Bell, Young-Moon Cho, Albert Firestone, Karin Healy, Jia Liu, Richard Ludwig, and Scott D. Rothenberger[1].
Checked by Edward R. Holler, Jr. and Bruce E. Smart.

## 1. Procedure

A. *8-n-Butyl-2-hydroxytricyclo*[*7.3.1.0*$^{2,7}$]tridecan-13-one. A 2-L, three-necked, round-bottomed flask is equipped with a magnetic stirring bar, a thermometer, a 500-mL pressure-equalizing dropping funnel, and a reflux condenser fitted with a nitrogen gas inlet tube that is attached to a mineral oil bubbler. The flask is flushed with nitrogen and then charged with 1.0 L (947 g, 9.65 mol) of

cyclohexanone (Note 1). The cyclohexanone is stirred and heated to 70–75°C under nitrogen, a solution of 9.0 g (0.14 mol) of potassium hydroxide (Note 2) in 150 mL of absolute ethanol is added in one portion, and then a solution of 150 mL (122 g, 1.4 mol) of pentanal (Note 3) in 140 mL of absolute ethanol is added dropwise over a 8-hr period while maintaining the reaction mixture at 70–75°C. The reaction mixture is stirred and held at 70–75°C for an additional 12 hr, and then allowed to cool to room temperature. The reaction flask is immersed in an ice bath, the inner wall of the flask is scratched with a glass rod to initiate crystallization, and the mixture is kept at 0°C for 4 hr to complete the crystallization. The colorless crude product is collected by vacuum filtration and washed with 200 mL of cold ether. The filtrates are combined and concentrated to approximately 200 mL with a rotary evaporator. The precipitated white solid is collected by filtration and washed with water (2 × 200 mL) and 200 mL of cold ether to give a second crop of crude product. The two crops ae combined and recrystallized by dissolving in hot methanol (3 mL per gram of solid), boiling until cloudiness occurs and cooling slowly to room temperature, then to 0°C. The white crystals are collected by vacuum filtration to give 228–230 g (61–62%) of 8-*n*-butyl-2-hydroxytricyclo[7.3.1.$0^{2,7}$]tridecan-13-one, mp 140–141°C (Note 4).

B. *9-n-Butyl-1,2,3,4,5,6,7,8-octahydroacridine.* A 2-L, three necked flask equipped with a mechanical stirrer, a glass stopper, and a reflux condenser is charged with 70 g (0.91 mol) of ammonium acetate, 309 g (1.55 mol) of cupric acetate monohydrate (Note 5) and 750 mL of glacial acetic acid. The mixture is stirred and heated at reflux for 15 min under nitrogen. The resulting solution is allowed to cool below reflux and 200 g (0.76 mol) of 8-*n*-butyl-2-hydroxytricyclo-[7.3.1.$0^{2,7}$]-tridecan-13-one is added in several portions. The blue-green reaction mixture is then refluxed under nitrogen for 3 hr with efficient stirring to control foaming. The mixture is allowed to cool to room temperature and then chilled in an ice bath for 3 hr (Note 6). The precipitated cuprous acetate is collected by vacuum filtration using a medium porosity fritted glass funnel and washed with 200 mL of ether. The combined filtrates are mixed with 500 g of ice in a 4-L beaker and stirred rapidly as 1600 mL of concentrated ammonium hydroxide is added slowly along with additional ice (ca. 300 g) to avoid local heating and boiling of the ether (final pH 10–11). The resulting mixture is separated and the aqueous layer is extracted with ether (400 mL, then 2 × 200 mL). The combined ether layers are washed with 200 mL of 20% aqueous ammonium hydroxide, followed by 200 mL of saturated aqueous sodium chloride, and dried over anhydrous magnesium sulfate. The drying agent is removed by filtration and washed with 200 mL of ether. The combined filtrates are concentrated to minimum volume with a rotary evaporator and the resulting solid is dried to constant weight under vacuum (1 mm) to give 173–177 g (91–96%) of beige solid, mp 36–38°C (Note 7). The product may be used directly in procedure C or further purified by adding a solution of material in dichloromethane to a column of Woelm neutral alumina and eluting with dichloromethane. The solvent is removed on a rotary evaporator to give 148 g (86%) of white crystalline solid, mp 41–43°C (Note 7).

C. *9-n-Butyl-1,2,4,5,6,7,8-octahydroacridine N-oxide.* A 2-L, round-bottomed flask equipped with a mechanical stirrer or a powerful magnetic stirrer and a reflux condenser fitted with a nitrogen inlet tube is flushed with nitrogen and charged with 38.0 g (0.16 mol) of 9-*n*-butyl-1,2,3,4,5,6,7,8-octahydroacridine, 42.7 g (0.51 mol) of sodium bicarbonate, and 890 mL of methanol. The resulting mixture is stirred as 110 g (0.18 mol) of Oxone® (Note 8) is added, followed by 360 mL of distilled water. The suspension is stirred under nitrogen at 48–50°C for 12–24 hr (Note 9). The mixture is cooled to room temperature and filtered, and the filter cake is washed with methanol (2 × 50 mL). The methanol is removed from the combined filtrates with a rotary evaporator, and the resulting mixture (volume: ca 200 mL) is extracted with dichloromethane (3 × 100 mL). The combined extracts are washed with water (2 × 50 mL) and dried over magnesium sulfate. The drying agent is removed by filtration, the filtrate is concentrated with a rotary evaporator, and the residual solid is dried under vacuum (0.1–0.5 mm) to give 40.0 g (99%) of 9-*n*-butyl-1,2,3,4,5,6,7,8-octahydroacridine-*N*-oxide as a cream colored solid, mp 92–94°C (Note 10),(Note 11),(Note 12).

D. *9-n-Butyl-1,2,3,4,5,6,7,8-octahydroacridin-4-ol.* Crude 9-*n*-butyl-1,2,3,4,5,6,7,8-octahydroacridine-*N*-oxide (38.9 g, 0.15 mol) is placed in a 1-L, three-necked, round-bottomed flask equipped with a magnetic stirrer, a glass stopper, and a reflux condenser fitted with a nitrogen inlet tube and a 500-mL addition funnel. Acetic anhydride (300 mL) is placed in the addition funnel, deaerated by sparging with helium for 30 min, and then added rapidly to the nitrogen-purged reaction flask. The reaction mixture is stirred and heated in a 100–110°C oil bath for 2 hr. The reflux condenser is replaced

by a simple distillation head and approximately 280 mL of acetic anhydride is removed by distillation at water-aspirator pressure (25–35 mm). To the brown residue is added 470 mL of 3 *M* aqueous hydrochloric acid, and the resulting mixture is refluxed under nitrogen for 1.5 hr. The mixture is allowed to cool to room temperature, chilled in an ice bath, and made alkaline (pH 12–13) by slowly adding about 550 mL of cold 4 *M* aqueous sodium hydroxide. The resulting cloudy mixture is extracted with chloroform (3 × 150 mL) and the combined extracts are dried over anhydrous sodium sulfate. The drying agent is removed by filtration and the filtrate is concentrated to dryness with a rotary evaporator. The brown residue is recrystallized from 75 mL of ethyl acetate, and the collected product is recrystallized again from 50 mL of ethyl acetate (Note 13) to give 26–28 g (67–72%) of 9-*n*-butyl-1,2,3,4,5,6,7,8-octahydroacridin-4-ol as a light beige solid, mp 105–106°C (Note 14).

## 2. Notes

1. Cyclohexanone (99.8%) was obtained from Aldrich Chemical Company, Inc. and was used without purification.
2. Certified-grade potassium hydroxide (86.6%) from Fisher Scientific was used.
3. Pentanal (99%) was obtained from Aldrich Chemical Company, Inc., redistilled under a static atmosphere of nitrogen (bp 103°C), and used immediately.
4. The product has the following spectroscopic properties: $^1$H NMR (300 MHz, $CDCl_3$) δ: 0.89 (t, 3 H, $J$ = 6, $CH_3$), 1.1–2.3 (m, 23 H, $CH_2$, CH), 2.44 (m, 1 H, $CH_2$), 2.72 (s, 1 H, OH); IR (KBr) cm$^{-1}$: 3413 (s), 2931 (s), 2857 (s), 1703 (s), 1455 (m), 1406 (m), 1378 (m), 1352 (m), 1285 (m), 1267 (m), 1206 (m), 1140 (m), 968 (m), 931 (m); mass spectrum *m/z* (relative abundance, 70 eV): 264 ($M^+$, 10), 167 (85), 166 (100). The submitters report that a second recrystallization gives analytically pure material, mp 141–142°C. Anal. calcd. for $C_{17}H_{28}O_2$: C, 77.22; H, 10,67. Found: C, 77.34; H, 10.51.
5. Ammonium acetate (99 + %) and copper(II) acetate monohydrate (98 + %) were obtained from Aldrich Chemical Company, Inc.
6. Alternatively, the stoppered reaction flask may be allowed to stand overnight in a refrigerator or freezer. In this case the flask should stand at room temperature for 0.5–1 hr prior to filtration in order to partially melt the acetic acid.
7. The product is pure by TLC analysis (Alumina GF Uniplate from Analtec, Inc., 1 : 1 ethyl acetate: hexane solvent, $R_f$ = 0.79) and $^1$H NMR analysis. The product has the following spectroscopic properties: $^1$H NMR (300 MHz, $CDCl_3$) δ: 0.96 (t, 3 H, $J$ = 6, $CH_3$), 1.42 (m, 4 H, $CH_2CH_2CH_3$), 1.7–1.9 (m, 8 H, H2, H3, H6, H7), 2.49 (m, 2 H, $ArCH_2$), 2.68 (m, 4 H, $ArCH_2$), 2.85 (m, 4 H, $ArCH_2$); IR (neat) cm$^{-1}$: 2932 (s), 2858 (s), 1565 (m), 1438 (m), 1409 (m), 1246 (w); mass spectrum, *m/z* (relative abundance 70, eV): 243 ($M^+$, 51), 228 (6), 214 (16), 201 (64), 200 (56), 186 (100). The submitters obtained an analytically pure sample by bulb-to-bulb distillation of the initial beige product. Anal. calcd. for $C_{17}H_{25}N$: C, 83.89; H, 10.35; N, 5.75. Found: C, 83.58; H, 10.07; N, 5.40.
8. Oxone®, the Du Pont Company trade name for potassium peroxymonosulfate, has the composition $2KHSO_5{\cdot}KHSO_4{\cdot}K_2SO_4$, and was purchased from Aldrich Chemical Company, Inc.
9. Oxidation may be monitored by thin-layer chromatography (Alumina GF Uniplate, 1 : 1 ethyl acetate : hexane). The $R_f$ values of the *N*-oxide product and starting material are 0.3 and 0.8, respectively. The reaction is approximately 99% complete after 12 hr, but reaction times varied with different lots of Oxone®
10. The submitters report obtaining 39 g (96%) of pale-yellow product, mp 89–92°C, when the beige starting material from Part B, mp 36–39°C, was used without further purification. The checkers, however, obtained only an 80% yield of *N*-oxide, mp 87–91°C, when crude starting material was used.
11. The product is sufficiently pure to be used directly in Part D, but may be further purified by recrystallization from ethyl acetate : hexane (1 : 6) to give colorless material, mp 99–101°C. The product has the following spectral properties : $^1$H NMR (300 MHz, $CDCl_3$) δ: 0.97 (t, 3 H, $J$ = 6, $CH_3$), 1.42 (m, 4 H, $CH_2CH_2CH_3$), 1.7–1.9 (m, 8H, H2, H3, H6, H7), 2.53 (m, 2 H, 9-$CH_2$), 2.69 (m, 4 H, H1, H8), 2.97 (m, 4 H, H4, H5); IR (KBr) cm$^{-1}$: 2942 (s), 2857 (s), 1477 (m), 1444 (m), 1424 (m), 1398 (m), 1350 (m), 1322 (m), 1286 (s), 1236 (m), 1096 (s).
12. The submitters provided the following alternative procedure for conducting the oxidation with *m*-chloroperoxybenzoic acid (MCPBA) in place of Oxone®. Into a 1-L, round-bottomed flask equipped with a magnetic stirrer, a reflux condenser, and a 250-mL addition funnel are placed 56.3 g (0.26 mol) of MCPBA (80%) and 350 mL of dichloromethane. The suspension is stirred and a solution of 38.0 g (0.16 mol) of 9-*n*-butyl-1,2,3,4,5,6,7,8-octahydroacridine in 120 mL of dichloromethane is added

rapidly (exotherm). When the reaction mixture ceases to boil gently from the heat of reaction, it is heated to extend the reflux period to a total of 2.5 hr. The reaction mixture is cooled to room temperature, extracted with 0.5 *M* aqueous sodium hydroxide (4 × 450 mL), and dried over anhydrous sodium sulfate. The drying agent is removed by filtration, the filtrate is concentrated with a rotary evaporator, and the residual solvent is removed at 0.1-mm pressure to afford 40 g (99%) of yellow crystalline product, mp 96–100°C.

13. For both recrystallizations, the solid is taken up in boiling ethyl acetate, rapidly filtered, and the filtrate is allowed to cool slowly to room temperature. It then is stored at −5°C overnight in a refrigerator prior to collecting the crystals.

14. The product ($R_f$ = 0.47) contains a trace of 9-*n*-butyl-1,2,3,4,5,6,7,8-octahydroacridine ($R_f$ = 0.79) by TLC analysis (Alumina GF Uniplate, 1 : 1 ethyl acetate : hexane). The checkers chromatographed a 10-g sample on a Woelm neutral alumina column (75 × 28 mm) using 300 mL of warm ethyl acetate as eluent to give 9.2 g of colorless product, mp 107–109°C. The original and chromatographed products have identical spectroscopic properties: $^1$NMR (300 MHz, $CDCl_3$) δ: 0.96 (t, 3 H, $J$ = 7, $CH_3$), 1.41 (m, 4 H, $CH_2CH_2CH_3$), 1.7–1.9 (m, 6 H, H2, H6, H7), 2.03 (m, 1 H, H3), 2.27 (m, 1 H, H3), 2.50 (m, 2 H, 9-$CH_2$), 2.70 (m, 4 H, H1, H8), 2.84 (m, 2 H, H5), 4.63 (m, 1 H, H4), 4.76 (s, 1 H, OH); IR (KBr) cm$^{-1}$: 3174 (s, br), 2942 (s), 2713 (m), 1569 (s), 1432 (s), 1407 (s), 1377 (m), 1338 (s), 1307 (s), 1253 (m), 1216 (m), 1169 (m), 1155 (s), 1094 (s), 1081 (s), 1005 (s), 962 (s), 939 (m), 893 (m). The submitters obtained an analytical sample, mp 104–105°C, by recrystallization from ethyl acetate and drying for 6 hr at room temperature (0.1 mm). Anal. calcd. for $C_{17}H_{25}NO$: C, 78.72; H, 9.71; N, 5.40. Found: C, 78.81, H, 9.53, N, 5.19.

## 3. Discussion

Taken together, Steps A and B of this procedure describe the most expedient, large-scale approach to 9-*n*-butyl-1,2,3,4,5,6,7,8-octahydroacridine, which is prepared in about 60% overall yield from inexpensive starting materials. This heterocycle is an important building block for "hexagonal lattice" receptors, which are relatively rigid, planar hosts for metal ions and organic molecules.[2 3 4 5 6 7 8 9]

Several methods exist for preparing pyridines that are annelated to nonaromatic rings in the [*b,e*]-positions,[10,11 12 13 14 15 16,17 18,19 20 21 22] but most do not also introduce an alkyl group in the 4-position. *n*-Butyl groups are found to lend solubility to higher molecular weight hexagonal lattice receptors. Step A of this procedure is a modification of the method of Tilichenko, who has reported the condensation of cyclohexanone with various aldehydes.[23] The reaction involves the following sequence: aldol condensation to form 2-pentylidenecyclohexanone, Michael addition of cyclohexanone enolate, and intramolecular aldol condensation of the resulting 1,5-diketone. Many aldol products are formed and the yield of keto alcohol depends strongly on (1) reaction temperature, (2) use of a large excess of cyclohexanone, and (3) prolonged addition of the aldehyde. The ease of product isolation is particularly dependent on its crystallinity and solubility.

If a substituent is not required in the 4-position of the new pyridine ring, then viable alternatives to the current procedure include trimethylhydrazonium-salt pyrolysis[17,18] and various methods for condensing ketones or enamines with formaldehyde or methyleneammonium salts.[11,16,22] The latter methods often involve isolation of the intermediate 1,5-diketone, which is condensed with ammonia or ammonium salts to form the pyridine ring.[12,13,14,16] The yield of this cyclization is limited by disproportionation of the intermediate dihydropyridine.[24] Hydrazine[15] or hydroxylamine[2,11] may also be used, but yields are similar to those obtained with ammonium acetate in acetic acid. The cupric acetate/ammonium acetate method described in Step B nearly quantitatively gives annelated pyridines from various 1,5-diketone equivalents.[24] Cupric acetate appears to be the oxidant of choice for intercepting dihydropyridines before disproportionation can occur.

Step C describes a method for oxidizing a pyridine to its *N*-oxide with Oxone® (potassium hydrogen persulfate). The more traditional oxidant, *m*-chloroperoxybenzoic acid (MCPBA), works equally well, but the availability of 80–85% pure MCPBA is now limited. Pyridine *N*-oxides may also be prepared with hydrogen peroxide in acetic acid,[25,26] but reaction time is variable and removal of acetic acid is inconvenient for large-scale preparations. Potassium hydrogen persulfate (Oxone®) is an inexpensive alternative to MCPBA in many oxidation reactions.[27 28 29 30 31 32] The oxidation procedure given here

avoids the formation of volatile peroxides, which occurs in ketone-catalyzed *N*-oxidation of pyridine by persulfate.[28,31] A 50% excess of Oxone® is used, assuming 100% activity. The submitters used Oxone® of 67–68% purity by iodometric titration. Less oxidant leads to incomplete reaction or inconveniently long reaction times.

Synthesis of annelated polypyridines or hexagonal lattice receptors from 1,2,3,4,5,6,7,8-octahydroacridines requires oxidative functionalization of the 4-position ($CH_2$ group bonded to the pyridine 2-position). In Step D this is accomplished by "Katada" or "Boekelheide" rearrangement of the *N*-oxide. This general reaction is commonly used for selective oxidation of alkylated pyridines, although the mechanism for conversion of the acetylated *N*-oxide to the 2-acetoxyalkylpyridine has not been fully elucidated.[33] [34] The current procedure reflects an empirical finding that deoxygenation of the acetic anhydride prior to addition results in slightly higher yields. Condensation of 2-alkylpyridines with benzaldehyde, followed by ozonolysis of the benzylidene intermediate, is a general, alternative route to 2-oxoalkylpyridines.[35] The *N*-oxide rearrangement described here is superior when monofunctionalization is required, because condensation of 9-*n*-butyl-1,2,3,4,5,6,7,8-octahydroacridine with 1 equiv of benzaldehyde gives a mixture of monobenzylidene and dibenzylidene derivatives.[2,3,4,5,6,7,8,9] Recent work by Tilichenko has shown that 1,5-diketones may be converted to monobenzylidene derivatives before forming the pyridine ring,[36] but overall yields are lower than those for the current procedure.

## References and Notes


1. Department of Chemistry, State University of New York, Stony Brook, New York 11794-3400.
2. Bell, T. W.; Firestone, A. *J. Org. Chem.* **1986**, *51*, 764–765;
3. Bell, T. W.; Firestone, A. J. *Am. Chem. Soc.* **1986**, *108*, 8109–8111;
4. Bell, T. W.; Firestone, A.; Guzzo, F.; Hu, L. Y. *J. Inclusion Phenom.* **1987**, *5*, 149–152;
5. Bell, T. W.; Liu, J. *J. Am. Chem. Soc.* **1988**, *110*, 3673–3674;
6. Bell, T. W.; Firestone, A.; Ludwig, R. *J. Chem. Soc., Chem. Commum.* **1989**, 1902–1904;
7. Bell, T. W.; Firestone, A.; Liu, J.; Ludwig, R.; Rothenberger, S. D. in "Inclusion Phenomena and Molecular Recognition," Atwood, J. L., Ed.; Plenum: New York, 1990, pp. 49–56;
8. Bell, T. W.; Liu, J. *Angew. Chem. Int. Ed. Engl.* **1990**, *29*, 923–925;
9. Bell, T. W.; Jousselin, J. *J. Am. Chem. Soc.* **1991**, *113*, 6283–6284.
10. Review: Thummel, R. P. in "Pyridine and its Derivatives," Newkome, G. R., Ed.; Wiley-Interscience: New York, 1984, Chapter 5.
11. Gill, N. S.; James, K. B.; Lions, F.; Potts, K. T. *J. Am. Chem. Soc.* **1952**, *74*, 4923–4928;
12. Colonge, J.; Dreux, J.; Delplace, H. *Bull. Soc. Chim. Fr.* **1957**, 447–449;
13. Hall, G. E.; Walker, J. *J. Chem. Soc. (C)* **1968**, 2237–2244;
14. Vysotskii, V. I.; Tilichenko, M. N. *Khim. Geterotsikl. Soedin.* (Engl. transl.) **1969**, 560–561; *Chem. Abstr.* **1970**, *72*, 31579*m*;
15. Moskovkina, T. V.; Kaminskii, V. A.; Vysotskii, V. I.; Tilichenko, M. N. *Khim. Geterotsikl. Soedin.* (Engl. transl.) **1973**, 759–762; *Chem. Abstr.* **1973**, *79*, 91958p;
16. Thummel, R. P.; Jahng, Y. *J. Org. Chem.* **1985**, *50*, 2407–2412.
17. Newkome, G. R.; Fishel, D. L. *J. Org. Chem.* **1972**, *37*, 1329–1336;
18. Partch, R. E.; Andrews, R. R.; Luellen, P.; Forsyth, D. A. *Tetrahedron Lett.* **1982**, *23*, 4447–4448.
19. Thummel, R. P.; Kohli, D. K. *J. Org. Chem.* **1977**, *42*, 2742–2747;
20. Katritzky, A. R.; Marson, C. M. *J. Am. Chem. Soc.* **1983**, *105*, 3279–3283;
21. Ransohoff, J. E. B.; Stabb, H. A. *Tetrahedron Lett.* **1985**, *26*, 6179–6182;
22. Risch, N.; Esser, A. *Synthesis* **1988**, 337–339.
23. Tilichenko, M. N. *Uch. Zap. Saratovsk. Gos. Univ.* **1962**, *75*, 60–65; *Chem. Abstr.* **1964**, *60*, 419*a*.
24. Bell, T. W.; Rothenberger, S. D. *Tetrahedron Lett.* **1987**, *28*, 4817–4820.
25. Katritzky, A. R.; Lagowski, J. M. "Chemistry of the Heterocyclic *N*-Oxides," Academic Press: New York, 1971.
26. Klimov, G. A.; Tilichenko, M. N. *Khim. Geterotsikl. Soedin. Sb. 1: Azotsoderzhashchie*

*Geterotsikey* **1967**, 306–309; *Chem. Abstr.* **1969**, *70*, 87539*z*.
**27.** Trost, B. M.; Curran, D. P. *Tetrahedron Lett.* **1981**, *22*, 1287–1290;
**28.** Gallopo, A. R.; Edwards. J. O. *J. Org. Chem.* **1981**, *46*, 1684–1688;
**29.** Cicala, G.; Curci, R.; Fiorentino, M.; Laricchiuta, O. *J. Org. Chem.* **1982**, *47*, 2670–2673;
**30.** Jeyaraman, R.; Murray, R. W. *J. Am. Chem. Soc.* **1984**, *106*, 2462–2463;
**31.** Murray, R. W.; Jeyaraman, R. *J. Org. Chem.* **1985**, *50*, 2847–2853;
**32.** Davis, F. A.; Chattopadhyay, S.; Towson, J. C.; Lal, S.; Reddy, T. *J. Org. Chem.* **1988**, *53*, 2087– 2089.
**33.** Cohen, T.; Deets, G. L. *J. Am. Chem. Soc.* **1972**, *94*, 932–938;
**34.** McKillop, A.; Bhagrath, M. K. *Heterocycles* **1985**, *23*, 1697–1701, and references cited therein.
**35.** Lodde, N.; Reimann, E. *Arch. Pharm. (Weinheim, Ger.)* **1979**, *312*, 940–950; *Chem. Abstr.* 1980, 163822*h*.
**36.** Minaeva, N. N.; Tilichenko, M. N. *Khim. Geterotsikl. Soedin.* (Engl. transl.) **1986**, *22*, 1720–1724.

---

## Appendix
## Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

pyridine N-oxides

potassium peroxymonosulfate

m-chloroperoxybenzoic acid (MCPBA)

1,2,3,4,5,6,7,8-octahydroacridines

4-Acridinol; 9-butyl-1,2,3,4,5,6,7,8-octahydro

ethanol (64-17-5)

hydrochloric acid (7647-01-0)

acetic acid (64-19-7)

ammonia (7664-41-7)

ethyl acetate (141-78-6)

methanol (67-56-1)

ether (60-29-7)

ammonium acetate (631-61-8)

acetic anhydride (108-24-7)

sodium hydroxide (1310-73-2)

formaldehyde (50-00-0)

chloroform (67-66-3)

oxone (37222-66-5)

sodium bicarbonate (144-55-8)

Cyclohexanone (108-94-1)

sodium chloride (7647-14-5)

sodium sulfate (7757-82-6)

nitrogen (7727-37-9)

benzaldehyde (100-52-7)

pyridine (110-86-1)

potassium hydroxide (1310-58-3)

hydrogen peroxide (7722-84-1)

ammonium hydroxide (1336-21-6)

cupric acetate (142-71-2)

benzylidene

persulfate (13445-49-3)

hydrazine (302-01-2)

hydroxylamine (7803-49-8)

dichloromethane (75-09-2)

magnesium sulfate (7487-88-9)

Ammonium (14798-03-9)

cupric acetate monohydrate,
copper(II) acetate monohydrate (6046-93-1)

hexane (110-54-3)

dihydropyridine

helium (7440-59-7)

methyleneammonium

cuprous acetate (598-54-9)

pentanal (110-62-3)

2-pentylidenecyclohexanone

cyclohexanone enolate

potassium hydrogen persulfate

m-chloroperoxybenzoic acid (937-14-4)

9-n-BUTYL-1,2,3,4,5,6,7,8-OCTAHYDROACRIDIN-4-OL (99922-91-5)

8-n-Butyl-2-hydroxytricyclo[7.3.1.$0^{2,7}$]tridecan-13-one (24133-22-0)

9-n-butyl-1,2,3,4,5,6,7,8-octahydroacridine (99922-90-4)

9-n-butyl-1,2,3,4,5,6,7,8-octahydroacridine-N-oxide (136528-61-5)

9-n-Butyl-1,2,4,5,6,7,8-octahydroacridine N-oxide