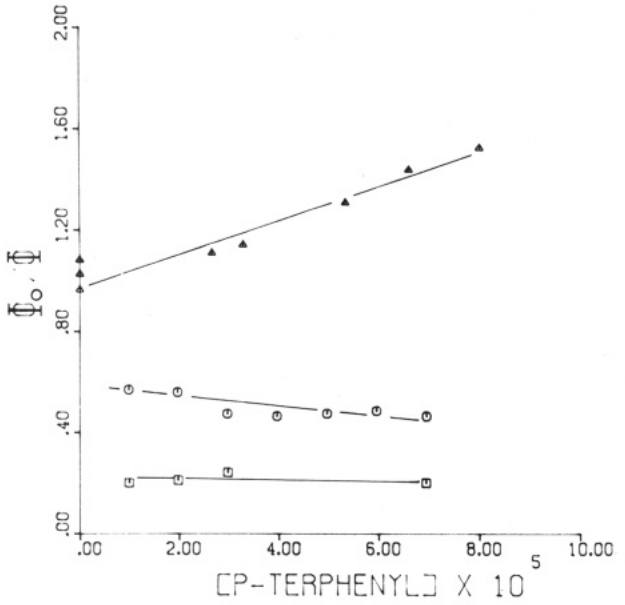

Figure 3. Effect of added tetrahydrofuran on electron-transfer quenching (△, no THF; ○, 5% THF; □, 10% THF).

that reduced the quantum yield by a factor of 2 and 4. When similarly prepared solutions and concentrations of quencher were used, there was no measurable quenching of product formation (see Figure 3), indicating a dramatic change in the $Me_2SO^{-}\cdot$ lifetime. Although the reason for the increased rate of decomposition of $Me_2SO^{-}\cdot$ is obscure, Walker and co-workers[8] have noted a dramatic effect on the lifetime of free electrons and other anionic species upon addition of water to Me_2SO.

The presence of a negative intercept for eq 3 is not surprising if one considers that in neat Me_2SO, the *effective* concentration of acceptor will already be at its limiting value, since very little diffusion needs to take place prior to electron transfer. Moreover, various aspects of relative product formation indicate that the $Me_2SO^{-}\cdot$ lifetime is *the* factor determining the remaining anomalous quantum yield behavior. We have previously noted that whereas the combination of methyl radical with trityl radical yields exclusively the product of *alpha* attack, TPE, the combination of methyl radical with trityl anion yields both TPE and the product of para attack DTM in a ratio 3:1 that is characteristic of the radical anion process.[2] This additional product may reflect the stability of the intermediate radical anion or the greater delocalization of trityl anion over trityl radical due to electron-electron repulsion.[12] When tetrahydrofuran is added, however, a decrease in DTM yield is noted.[13] Furthermore, the large increase in TPE yield at high THF concentrations is unaccompanied by a similar increase in TTF yield. Both these results indicate that the quantum yield inversion is due not to an increase in free methyl radical concentration, which would increase the yield of all products, but a cage effect dependent upon the competition between $Me_2SO^{-}\cdot$ decomposition and diffusion from the cage. Thus when $Me_2SO^{-}\cdot$ lifetime is short or, equivalently, the electron transfer to Me_2SO becomes dissociative, the back electron transfer represented by $k_{d'}$ becomes negligible, and the quantum yield of TPE formation increases. This corresponds in a change from the nonchain $S_{RN}1$ mechanism[14] to the S_{ET} mechanism.[15]

Steady-state treatments of electron-transfer reactions provide good models for observed rate constants when both donors and acceptors are well separated by a nonreacting solvent. At high concentrations of acceptors, and presumably donors, large deviations from linearity and even rate inversions are possible. These anomalies reflect partially the participation of the acceptor in the solvation sphere of the donor and partially the change in partition between the forward reaction and back electron transfer. Although we do not of course understand every aspect of this behavior, we are very excited by the prospect that high concentration studies may provide new details of nature of donor–acceptor interactions and may allow direct determination of the microscopic rate constants for electron transfer which are only now inferred.

Acknowledgment. Support by the Division of Chemical Sciences, Office of Basic Energy Sciences of the U.S. Department of Energy is gratefully acknowledged.

Registry No. Triphenylmethyl anion, 40006-86-8; biphenyl, 92-52-4; naphthalene, 91-20-3; picene, 213-46-7; *p*-terphenyl, 92-94-4; chrysene, 218-01-9; anthracene, 120-12-7; fluoranthene, 206-44-0.

(11) The involvement of methyl radical in the mechanism is demonstrated by the stability of tetrahydrofuran solutions of trilithium upon prolonged irradiation with no detectable yield of TTF.

(12) House, H. O.; Weeks, P. D. *J. Am. Chem. Soc.* **1975**, *97*, 2785.

(13) As a minor product, the yield of DTM was difficult to quantitate. However, it never exceeded 5% of the product mixture.

(14) Bunnett, J. F. *Acc. Chem. Res.* **1978**, *11*, 413.

(15) Russell, G. A.; Jawdosuik, M.; Makosza, M. *J. Am. Chem. Soc.* **1979**, *101*, 2355.

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An Efficient Synthesis of Ellipticine

Summary: A synthesis of the antitumor pyrido[4,3-*b*]-carbazole alkaloid ellipticine (**1a**) is described in which the key steps are, successively, a highly regioselective acylation of 2-lithio-1-(phenylsulfonyl)indole (**4**) with 3,4-pyridinedicarboxylic anhydride (**3**) and acetic anhydride induced ring closure to give keto lactam **8**. Further manipulation affords ellipticine in 54% overall yield from indole (**2a**).

Sir: The alkaloid ellipticine (**1a**) and its 9-oxygenated derivatives **1b** and **1c** show pronounced anticancer activity toward several experimental[1] and human[2] tumor systems. For example, 9-methoxyellipticine (**1c**) is capable of inducing complete remission in patients with acute myeloblastic leukemia[2] and 2-methyl-9-hydroxyellipticinium acetate has been successfully used to treat certain intractable human cancers unresponsive to other drug protocols.[3,4]

(1) (a) Svoboda, G. H.; Poore, G. A.; Montford, M. L. *J. Pharm. Sci.* **1968**, *57*, 1720. (b) Hayat, M.; Mathé, G. *Biomedicine* **1974**, *21*, 101. (c) Le Pecq, J. B.; Dat-Xuong, N.; Gosse, C.; Paoletti, C. *Proc. Natl. Acad. Sci. U.S.A.* **1974**, *71*, 5078. (d) Le Pecq, J. B.; Gosse, C.; Dat-Xuong, N.; Cros, S.; Paoletti, C. *Cancer Res.* **1976**, *36*, 3067. (e) Delbarre, A.; Roques, B. P.; Le Pecq, J. B.; Lallemand, J. Y.; Dat-Xuong, N. *Biophys. Chem.* **1976**, *4*, 275.

(2) Mathé, G.; Hayat, M.; De Vassal, F.; Schwarzenberg, L.; Schneider, M.; Schlumberger, J. R.; Jasmin, C.; Rosenfeld, C. *Rev. Eur. Etud. Clin. Biol.* **1970**, *15*, 541.

(3) (a) Juret, P.; Tanguy, A.; Girard, A.; Le Talaer, J. Y.; Abbatucci, J. S.; Dat-Xuong, N.; Le Pecq, J. B.; Paoletti, C. *Nouv. Presse Med.* **1979**, *8*, 1495. (b) Van-Bac, N.; Moisand, C.; Gouyette, A.; Muzard, G., Dat-Xuong, N.; Le Pecq, J. B.; Paoletti, C. *Cancer Treat. Rep.* **1980**, *64*, 879.

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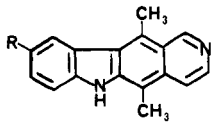
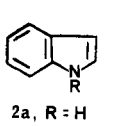
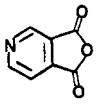


1a, R = H
1b, R = OH
1c, R = OCH_3

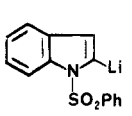
Although ellipticine has been the target of many synthetic studies,[5] the obvious need for an efficient method suitable for large-scale preparations of **1a** and its more active 9-substituted derivatives (e.g., **1b**, **1c**) encouraged us to undertake the present study. We projected that the pyrido[4,3-*b*]carbazole skeleton in **1a** would be readily attainable from the simple starting materials indole (**2a**) and 3,4-pyridinedicarboxylic anhydride (**3**;[6] cinchomeronic anhydride). We now report the realization of this strategy, resulting in a 54% overall yield of ellipticine from indole.



2a, R = H
2b, R = SO_2Ph



3



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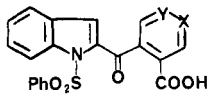
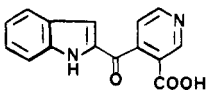


5a, X = N ; Y = CH
5b, X = CH ; Y = N



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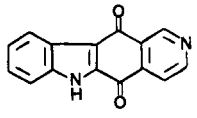

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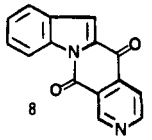


8

During the course of this study, Joule reported[5d] a conceptually similar synthesis of ellipticine, involving the acylation of 2-lithio-1-(phenylsulfonyl)indole (**4**) with 3-(hydroxymethyl)isonicotinic acid lactone. Subsequent operations, including a novel indole-3-nucleophilic substitution reaction, afforded ellipticine. Although the overall yield of **1a** from indole was respectable (ca. 36%), the preparation of the requisite pyridine lactone is by no means trivial.[7] Our approach obviates the need to prepare this lactone and instead utilizes **3** directly.

Indole (**2a**) was converted to 1-(phenylsulfonyl)indole (**2b**) in 91% yield by treatment with *n*-butyllithium in tetrahydrofuran (THF) followed by the addition of benzenesulfonyl chloride at −78 °C.[8] Regiospecific 2-lithiation of **2b** was achieved[8] with lithium diisopropylamide (LDA) (THF, −75 → 0 °C, 2 h) and the resulting solution of 2-lithio-1-(phenylsulfonyl)indole (**4**) was rapidly treated with **3** at −100 °C. This afforded a mixture of protected keto acids **5a**[9,10] and **5b**,[10] mp 252–254 °C dec, in 78% yield after crystallization from acetic acid, in a ratio of 92:8 (vide infra). A priori, we had anticipated[11] that the ring opening of **3** would be regioselective in the desired sense (→ **5a**), but this could not be proved until the completion of the synthesis. The mixture of **5a** and **5b** was easily separated by fractional crystallization from acetone since the minor isomer **5b**, mp 274–275 °C dec, is completely insoluble in this solvent. This solubility difference between **5a** and **5b** parallels that between nicotinic and isonicotinic acid, the latter being relatively more insoluble than the former,[12] and suggested to us that the ring opening of **3** had indeed occurred in the desired fashion.

Cleavage of the phenylsulfonyl protecting group in pure **5a**, mp 263–264 °C dec, was achieved with potassium carbonate (MeOH–H_2O, 3:1, reflux, 5 h) to afford keto acid **6**,[9,10] mp 159–162 °C dec (acetone), in essentially quantitative yield.[13] After several abortive attempts to cyclize **6** to the known[5b,e] ellipticine quinone **7**, using conventional Friedel–Crafts and other conditions, we found that heating **6** in neat acetic anhydride (80–85 °C, 24 h) smoothly effected cyclization to the bright yellow-orange keto lactam **8**,[9,10] mp 196–199 °C dec (acetone), in virtually quantitative yield. The structure of **8** is supported by its quantitative reconversion to **6** with aqueous base at room temperature,[14] and by its infrared spectrum, which shows carbonyl absorption at 1702 and 1675 cm^{-1} consistent with *N*-acyl-

(4) This new derivative has now entered phase II clinical trials for further testing; see ref 3b.

(5) (a) For two recent reviews, see: Sainsbury, M. *Synthesis* **1977**, 437. Barone, R.; Chanon, M. *Heterocycles* **1981**, *16*, 1357. (b) Taylor, D. A.; Baradarani, M. M.; Martinez, S. J.; Joule, J. A. *J. Chem. Res., Synop.* **1979**, 387; *J. Chem. Res., Miniprint* **1979**, 4801. (c) Taylor, D. A.; Joule, J. A. *J. Chem. Soc., Chem. Commun.* **1979**, 642. (d) Ashcroft, W. R.; Beal, M. G.; Joule, J. A. *Ibid.* **1981**, 994. (e) Watanabe, M.; Snieckus, V. *J. Am. Chem. Soc.* **1980**, *102*, 1457. (f) Kano, S.; Sugino, E.; Shibuya, S.; Hibino, S. *J. Org. Chem.* **1981**, *46*, 2979.

(6) This material is readily prepared from the commercially available diacid using the procedure of Bachman, G. B.; Barker, R. S. *J. Org. Chem.* **1949**, *14*, 97.

(7) Ashcroft, W. R.; Beal, M. G.; Joule, J. A. *J. Chem. Soc., Perkin Trans. 1* **1981**, 3012.

(8) Saulnier, M. G.; Gribble, G. W. *J. Org. Chem.* **1982**, *47*, 757.

(9) A satisfactory combustion analysis has been obtained for this compound.

(10) **5a**: IR (KBr) 3440, 2390,1735, 1680, 1605, 1535, 1450, 1185, 1045, 840, 585 cm^{-1}; ^1H NMR (Me_2SO-d_6) δ 7.26 (s, 1 H), 7.37 (t, 1 H), 7.55–7.80 (m, 6 H), 8.16–8.23 (m, 3 H), 8.92 (d, 1 H), 9.12 (s, 1 H); ^{13}C NMR (Me_2SO-d_6) δ 116.0, 122.9, 124.1, 124.6, 125.3, 126.2, 127.7, 127.9, 129.8, 130.1, 135.2, 137.6, 138.9, 139.8, 147.5, 151.1, 153.7, 166.6, 184.4; mass spectrum, *m/e* 406 (M^+), 265, 248, 141, 115, 77 (100%); UV (95% EtOH) λ_{max} 213 nm, 244 (sh), 301. **5b**: IR (KBr) 3440, 2450, 1720, 1670, 1530, 1355, 1175, 1045, 960, 725, 590 cm^{-1}; ^1H NMR (Me_2SO-d_6) δ 7.29 (s, 1 H), 7.38 (t, 1 H), 7.56–7.83 (m, 6 H), 8.13–8.20 (m, 3 H), 8.85 (s, 1 H), 8.95 (d, 1 H); ^{13}C NMR (Me_2SO-d_6) δ 115.9, 122.9, 123.5, 124.5, 125.4, 125.9, 127.6, 128.1, 129.5, 130.1, 133.4, 135.4, 138.3, 138.5, 139.3, 141.1, 153.7, 167.5, 184.4; mass spectrum, *m/e* 406 (M^+), 265, 248, 141, 115, 77 (100%); UV (95% EtOH) λ_{max} 214 nm, 245 (sh), 300. **6**: IR (KBr) 3475, 3320, 2440, 1715, 1645, 1525, 1310, 1250, 740, 670, 475 cm^{-1}; ^1H NMR (Me_2SO-d_6) δ 6.71 (s, 1 H), 7.08 (t, 1 H), 7.33 (t, 1 H), 7.49 (d, 1 H), 7.61–7.67 (m, 2 H), 8.93 (d, 1 H), 9.15 (s, 1 H), 12.10 (s, 1 H); ^{13}C NMR (Me_2SO-d_6) δ 113.0, 113.4, 121.4, 122.6, 123.6, 125.6, 127.0, 127.4, 135.3, 139.0, 148.4, 151.1, 153.6, 166.4, 186.6; mass spectrum, *m/e* 266 (M^+), 248, 220, 192, 164, 144, 116, 89 (100%); UV (95% EtOH) λ_{max} 209 nm, 228 (sh), 319. **8**: IR (KBr) 3460, 1702, 1675, 1550, 1370, 1340, 1245, 750, 720, 685 cm^{-1}; ^1H NMR (Me_2SO-d_6) δ 7.46 (t, 1 H), 7.68 (t, 1 H), 7.85 (s, 1 H), 7.90 (d, 1 H), 8.03 (d, 1 H), 8.53 (d, 1 H), 9.13 (d, 1 H), 9.49 (s, 1 H); ^{13}C NMR (Me_2SO-d_6) δ 116.3, 118.3, 124.1, 124.7, 125.3, 128.3, 130.0, 133.6, 136.3, 138.7, 150.0, 155.2, 158.3, 174.6; mass spectrum, *m/e* 248 (M^+, 100%) 220, 192, 164, 115, 88, 50; UV (95% EtOH) λ_{max} 217 nm, 240, 266 (sh); with added base the UV spectrum obtained is identical with that of **6**. **9**: IR (KBr) 3440, 3240, 1605, 1455, 1310, 1080, 1040, 910, 830, 740 cm^{-1}; mass spectrum, *m/e* 280 (M^+), 262, 247 (100%), 219, 148, 117, 89; UV (95% EtOH) λ_{max} 219 nm, 265, 282, 292 (sh). **1a**: IR (KBr) 3470, 1620, 1605, 1470, 1415, 1250, 1030, 845, 810, 740, 465 cm^{-1}; ^{13}C NMR (Me_2SO-d_6) δ 11.9, 14.3, 107.9, 110.7, 115.8, 119.1, 121.9, 123.1, 123.3, 123.7, 127.0, 127.9, 132.4, 140.5, 142.6, 149.6; mass spectrum, *m/e* 246 (M^+, 100%), 245, 231, 217, 123, 109, 96, 51; UV (MeOH) λ_{max} 219 nm, 274 (sh), 283.5, 292.5, 319.

(11) For a recent example of the regioselective ring opening of **3**, see Bottaro, J. C.; Berchtold, G. A. *J. Org. Chem.* **1980**, *45*, 1176.

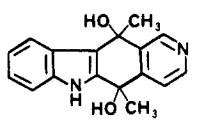
(12) We find that nicotinic acid is about 13 times more soluble in hot acetone than is isonicotinic acid (unpublished data); for similar data in other solvents, see Beilstein **1935**, *22*, 38, 45.

(13) A small amount of what is probably a methoxy keto acid, formed by conjugate addition of methoxide to the indole β-position of **5a**, is present by mass spectroscopy; for a similar reaction, see Cooper, M. M.; Hignett, G. J.; Joule, J. A. *J. Chem. Soc., Perkin Trans. 1* **1981**, 3008.

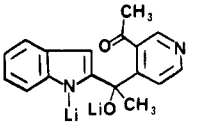
(14) *N*-Acylindoles are easily cleaved with base: Bergman, J.; Carlsson, R.; Misztal, S. *Acta Chem. Scand. B* **1976**, *30*, 853.

indole and aryl ketone chromophores, respectively.[15]

In accord with expectations, **8** reacted with methyllithium (2 equiv, -100 °C, THF) to afford diol **9**[10,16] as a mixture of diastereomers. This reaction presumably involves the successive generation of ketone **10** followed by cyclization to the indole β-position to afford **9**.



9



10

Without purification the crude diol mixture (**9**) was treated with sodium borohydride (EtOH, reflux)[5b] to afford ellipticine (**1a**), mp 311-315 °C dec, in 82% yield from **8** after flash chromatography[17] on silica gel (THF-EtOAc, 7:3) and in 54% overall yield from indole (**2a**). The material so obtained was identical in all respects (IR, TLC, UV, MS, mmp 311-315 °C dec) with an authentic sample of ellipticine.

Further studies to explore the generality of this pyrido[4,3-*b*]carbazole synthesis are in progress.

Acknowledgment. This investigation was supported in part by Merck Sharp and Dohme Research Laboratories, by Grant CH-200 from the American Cancer Society, and by Biomedical Research Support Grants RR-05392 and RR-07056 from the Biomedical Research Support Branch, Division of Research Facilities and Resources, National Institutes of Health. We also thank Dr. Catherine E. Costello (Massachusetts Institute of Technology) for the high-resolution mass spectra (National Institutes of Health Resource Grant FR00317 from the Division of Research Facilities and Resources), the Colorado State University Regional NMR Center, funded by National Science Foundation Grant No. CHE 78-18581, for high-resolution NMR spectra, Professors John A. Joule, Victor A. Snieckus, Jan Bergman, and Dr. Norbert Neuss for authentic samples and/or spectra of **1a** and **7**, and Mr. Al Barefoot for low-resolution mass spectra. We also thank Professor Joule for communicating his results to us prior to publication.

Registry No. **1a**, 519-23-3; **2a**, 120-72-9; **2b**, 40899-71-6; **3**, 4664-08-8; **4**, 40900-03-6; **5a**, 81940-21-8; **5b**, 81940-22-9; **6**, 81940-23-0; **7**, 73326-98-4; **8**, 81940-24-1; **9**, 81940-25-2; **10**, 81940-26-3.

(15) By way of analogy, the corresponding 2-methylpyrrole-phthalic anhydride derived keto lactam shows carbonyl absorption at 1708 and 1655 cm^{-1}: Cornforth, J. W.; Firth, M. E. *J. Chem. Soc.* **1958**, 1091.
(16) **9**: mass spectrum, *m/e* 280.1202 (M^+, calcd 280.1212).
(17) Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.

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Palladium-Assisted C-Glycosylation. Addition of Carbanions to Cyclic Enol Ethers

Summary: Pd(II) effects the regiospecific addition of carbanions to dihydrofuran at the ring oxygen-bearing carbon. A similar regiospecific alkylation of acetoxydihydropyrans catalyzed by Pd(0) is also reported. Initial examples of the stereoselectivity of these reactions are also given.

Sir: The alkylation of furanosides and pyranosides constitutes a very important step in the synthesis of a variety of biologically active natural products. Methodology for this purpose continues to be developed and applied to the stereoselective asymmetric syntheses of *C*-glycoside-containing natural products. *C*-Glycofuranosides can be viewed as precursors to C-nucleosides and other natural products containing alkylated tetrahydrofuran moieties. Some recent examples include syntheses of showdomycin[1] and the elaboration of chiral furenone components of germacranolide sesquiterpenes.[2-4] *C*-Glycosides have also been prepared by a variety of methods, including enolate Claisen rearrangements,[5,6] Lewis acid catalyzed nucleophilic additions to glycals,[7,8] hetero-Diels-Alder reactions,[9-12] and allyl stannane coupling with glycosyl halides.[13]

As a part of a general study directed toward the development of transition metal controlled asymmetric functionalization of carbohydrates, the palladium-assisted alkylation of cyclic enol ethers has been investigated.[14] Herein we report examples of two regiospecific methods for the alkylation of cyclic enol ethers, both of which alkylate exclusively at the ring oxygen-bearing carbon atom.

The alkylation of dihydrofurans was first investigated. Palladium(II) reagents were used in these reactions following the recently developed methodology to alkylate alkenes.[15,16] The alkylation of 2,3-dihydrofuran was carried out by activation with bis(acetonitrile)palladium(II) chloride. Formation of the Pd(II) π complex was accomplished by addition of 2,3-dihydrofuran to a solution of $Pd(CH_3CN)_2Cl_2$ in 1:1 THF/DMF at room temperature under argon. The complex was cooled to -78 °C followed by addition of Et_3N (2.0 equiv/Pd). A carbanion (1 equiv) (**1-7**, Table I) was introduced at -78 °C and the reaction

(1) Ito, Y.; Shibata, T.; Arita, M.; Sawai, H.; M. Ohno, *J. Am. Chem. Soc.* **1981**, *103*, 6739 and references therein.
(2) Sun, K. M.; Fraser-Reid, B.; Tam, T. F. *J. Am. Chem. Soc.* **1982**, *104*, 367.
(3) Tam, T. F.; Fraser-Reid, B. *Can. J. Chem.* **1979**, *51* 2818 and references therein.
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(6) Ireland, R. E.; McGarvey, G. J.; Anderson, R. C.; Badoud, R.; Fitzsimmons, B.; Thaisrivongs, S. *J. Am. Chem. Soc.* **1980**, *102*, 6178.
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(9) Danishefsky, S.; Kerwin, J. F., Jr; Kobayashi, S. *J. Am. Chem. Soc.* **1982**, *104*, 358.
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(11) Snider, B. B.; Phillips, G. B. *J. Am. Chem. Soc.* **1982**, *104*, 1113.
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