tetrahydrofuran slurry (12 mL) of potassium *tert*-butoxide (472 mg, 4.2 mmol). After 15 min, TLC [silica/$CHCl_3$/MeOH/NH_3 (9:1:.1)] indicated that starting material was consumed. The reaction mixture was added to a stirred mixture of ethyl acetate/water (40 mL:150 mL), and the pH was adjusted to 11.5 with aqueous sodium hydroxide (6 N). The organic layer was separated, washed with saturated sodium chloride solution, dried over anhydrous sodium sulfate, and concentrated in vacuo, affording crude **5** (900 mg). The crude material was crystallized from an acetone/water mix and recrystallized from the same solvent to afford pure **5** (800 mg, mp 132–133 °C): ^{1}H NMR ($CDCl_3$) δ 0.90 (t), 0.95 (d), 1.05 (d), 1.15–1.30 (m), 1.40 (d), 1.65 (m), 1.82 (dd, 11.9, 10.4), 2.05 (s), 2.30 (s), 2.60–2.81 (m), 3.45–3.55 (m), 3.75 (br s), 3.95 (m), 4.20 (br s), 4.30 (br s), 4.60 (AB q, J_{AB} = 12.8 Hz), 4.75–4.95 (m), 5.15 (dd, 10.1, 2.1), 5.60 (d, 1.8); ^{13}C NMR ($CDCl_3$) δ 177.2 (off-resonance, s), 169.5 (s), 143.5 (s), 118.7 (d), 99.8 (d), 94.2 (d), 85.4 (s), 83.9 (d), 80.1 (d), 78.3 (d), 76.4 (d), 74.4 (s), 73.9, 71.9, 70.8, 69.6, 68.8, 63.2, 46.2, 42.9, 41.7, 40.5, 38.4, 34.6, 32.1, 31.3, 25.7, 24.8, 21.2, 20.8, 18.5, 16.8, 15.8, 13.1, 10.9, 7.7.

Anal. Calcd for $C_{40}H_{69}NO_{13}$: C, 62.23; H, 9.01; N, 1.81. Found: C, 62.08; H, 8.94; N, 1.80.

Acknowledgment. We thank Mr. R. Ware, Dr. E. Whipple, and Dr. G. Chmurny for assistance in obtaining spectral data and invaluable discussions with regard to the interpretations.

Registry No. **2**, 63864-45-9; **3**, 83291-99-0; **4**, 27491-70-9; **5**, 83292-00-6; 9-dihydro-2′-acetylerythromycin A, 83311-47-1.

Isoquinolinium Cycloadditions: Regiospecific Synthesis of 1-Naphthaldehydes and Conversion to 1-Naphthylamines

Sukumar Manna and J. R. Falck*

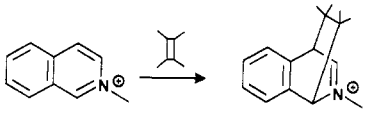
Department of Molecular Genetics, University of Texas Health Science Center at Dallas, Dallas, Texas 75235

Charles Mioskowski*

École Nationale Supérieure de Chimie, ERA du CNRS No. 687, Université Louis Pasteur, 67008 Strasbourg, France

Received May 25, 1982

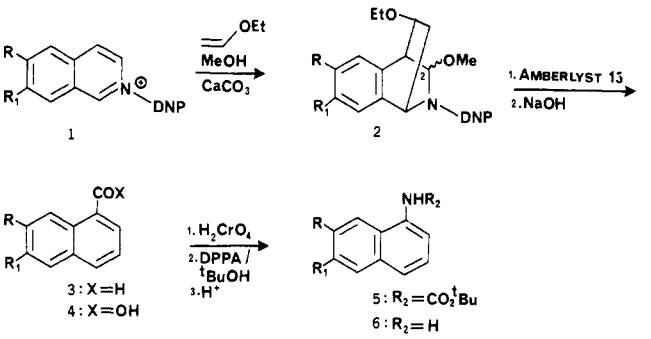
Polar cycloaddition of isoquinoline salts with electron-rich alkenes disrupts the aza aromatic ring, creating a tricyclic system containing up to four new stereocenters and an immonium ion capable of further chemistry (eq 1).



(1)

Due largely to the pioneering work of Bradsher,[1] these reactions are known to be virtually 100% regiospecific and often highly stereospecific. During our investigations into the general use of this intriguing reaction, we developed a modification that overcomes some prior synthetic limitations and gives good yields of adduct from 3-unsubstituted isoquinolines.[2] We report herein its application to a regiospecific synthesis of 1-naphthaldehydes and their conversion to 1-naphthylamines, useful benzophenanthridine alkaloid precursors.[3a,b,4]

Scheme I



a: R = R_1=OCH_2O; b: R=OCH_2Ph, R_1=OMe; c: R=H, R_1=OMe; d: R = R_1 = H

Treatment of dinitrophenyl (DNP) salt **1** (prepared from the corresponding isoquinoline[5] and 2,4-dinitrobromobenzene) in methanol with excess ethyl vinyl ether in the presence of powdered calcium carbonate for 24 h quantitatively generates adduct **2** as a mixture of C-2 epimers[1b] (Scheme I). The use of calcium carbonate is important for maximum yields. Other commonly used acid scavengers, inter alia, tertiary amines, alumina, glycidol, sodium bicarbonate, and sodium acetate, are either ineffective in preventing polymerization of the dienophile or incompatible with **1** under the reaction conditions. The foregoing epimeric mixture is hydrolyzed in tetrahydrofuran/water with Amberlyst-15 resin at 37 °C for 16 h and the isolated product immediately heated under reflux for 5 min with sodium hydroxide.[6] The entire procedure, best performed without purification of intermediates, affords naphthaldehyde **3** in 80–96% yield overall from **1** after chromatography.

Jones oxidation of **3** at 0 °C for 8 h gives naphthoic acid **4** (88–95%). Subsequent Curtius rearrangement by diphenylphosphoryl azide[7] (DPPA) and triethylamine in refluxing anhydrous *tert*-butyl alcohol leads to carbamate **5** (73–80%), which is hydrolyzed in tetrahydrofuran–water–concentrated hydrochloric acid to the easily oxidized naphthylamine (**6**; 73–86%).[8] Curtius rearrangement in dioxane with added benzyl alcohol followed by catalytic debenzylation is an equally effective alternative, e.g., **4c** to **6c** (71%).

Substituted isoquinoline methodide salts also give good yields of adduct but under more drastic contitions. For instance, 6,7-(methylenedioxy)isoquinoline methodide requires heating at 120 °C (sealed tube) for 2 days. More importantly, conversion of the adduct to naphthaldehyde proceeds in low overall yield (<30%).

Experimental Section

General Procedures. ^{1}H NMR spectra measured at 90 MHz on a JEOL FX-90Q spectrometer with tetramethylsilane as in-

(1) (a) Chen, T.-K.; Bradsher, C. K. *J. Org. Chem. 1979*, **44**, 4680–4683 and earlier references cited. (b) Intramolecular version: Gisby, G. P.; Sammes, P. G.; Watt, R. A. *J. Chem. Soc., Perkin Trans. 1* **1982**, 249–255.

(2) These limitations have been recognized: Day, F. H.; Bradsher, C. K.; Chen, T.-K. *J. Org. Chem.* **1975**, *40*, 1195–1198. Day, F. H.; Bradsher, C. K. *Tetrahedron Lett.* **1971**, 409–410.

(3) Examples of 1-naphthylamine synthesis: (a) Gillespie, J. P.; Amoros, L. G.; Stermitz, F. R. *J. Org. Chem.* **1974**, *39*, 3239–3241. (b) Begley, W. J.; Grimshaw, J. *J. Chem. Soc., Perkin Trans. 1* **1977**, 2324. (c) Campbell, K. N.; LaForge, R. A.; Campbell, B. K. *J. Org. Chem.* **1949**, *14*, 346–354.

(4) Reviews: Phillips, S. D.; Castle, R. N. *J. Heterocycl. Chem.* **1981**, *18*, 223–232. Hearn, M. J.; Swanson, S. L. *Ibid.* **1981**, *18*, 207–222.

(5) For a general isoquinoline synthesis, see Falck, J. R.; Manna, S.; Mioskowski, C. *J. Org. Chem.* **1981**, *46*, 3742–3745. Also, Birch, A. J.; Jackson, A. H.; Shannon, P. V. R.; Varma, P. S. P. *Tetrahedron Lett.* **1972**, 4789–4792.

(6) For related reaction leading to ketones, consult Day, F. H., Ph.D. Dissertation, Duke University, Durham, NC, 1973.

(7) Shioiri, T.; Ninomiya, K.; Yamada, S.-I. *J. Am. Chem. Soc.* **1972**, *94*, 6203–6205.

(8) Naphthylamines are best stored as their hydrochloride salt or cold under an inert atmosphere.

0022-3263/82/1947-5021$01.25/0

ternal standard. Mass (EI) and high-resolution mass spectra were obtained on a Finnigan 4000 and MAT-711, respectively. Infrared spectra were recorded on a Beckman Acculab 8. Melting-point determinations were preformed with an electrothermal melting point apparatus and are uncorrected. Microanalyses were conducted by Galbraith Laboratories, Inc., Knoxville, TN.

Preparation of 6,7-(Methylenedioxy)naphthaldehyde (3a). Cycloaddition and conversion to naphthaldehyde are best performed without purification of intermediates. To a stirred suspension of 2-(2,4-dinitrophenyl)-6,7-(methylenedioxy)isoquinolinium bromide (**1a**; 503 mg, 1.2 mmol) and powdered $CaCO_3$ (675 mg) in 4 mL of MeOH was added ethyl vinyl ether (864 mg, 12 mmol). After 12 h, another portion of vinyl ether (12 mmol) was added and stirring continued for an additional 12 h [TLC (SiO_2): 15% MeOH/CH_2Cl_2, R_f ~0.78]. The suspension was filtered over Celite, the filter cake washed with CH_2Cl_2, and the combined filtrate evaporated.

The crude adduct was stirred at 37 °C with Amberlyst-15 resin (300 mg) in 30 mL of THF and 3 mL of H_2O for 18 h. After filtration and evaporation, the residue was added immediately in 5 mL of THF to a solution of NaOH (400 mg) in 4 mL of H_2O and 10 mL of MeOH and then heated at reflux for 5 min. The resultant dark-red solution was concentrated under reduced pressure, diluted with H_2O, extracted with EtOAc, and chromatographed (SiO_2, 1:1 ether/hexanes) to give 230 mg of **3a** (96%): mp 121 °C (ether/hexane); NMR ($CDCl_3$) δ 5.86 (s, 2 H), 6.92 (s, 1 H), 7.22 (dd, J = 7, 7 Hz, 1 H), 7.56 (dd, J = 7, 1.5 Hz, 1 H), 7.64 (dd, J = 7, 1.5 Hz, 1 H), 8.46 (s, 1 H), 10.25 (s, 1 H); IR ($CHCl_3$) 1690, 1615, 1470, 1250 cm^{-1}; mass spectrum, m/e (rel intensity) 200 (M^+, 100), 171 (M^+ - CHO, 74); TLC (SiO_2, 2:1 ether/hexanes), R_f ~0.50; high-resolution mass spectrum calcd for $C_{12}H_8O_3$ 200.0473, found 200.0479.

6-Methoxy-7-(benzyloxy)naphthaldehyde (3b). By the method described for the preparation of **3a**, 2-(2,4-dinitrophenyl)-6-(benzyloxy)-7-methoxyisoquinolinium bromide (**1b**) was converted to **3b** (85%): mp 167–168 °C; NMR ($CDCl_3$) δ 3.78 (s, 3 H), 5.12 (s, 2 H), 6.94 (s, 1 H), 7.05–7.73 (m, 8 H), 8.62 (s, 1 H), 10.3 (s, 1 H); IR ($CHCl_3$) 1690, 1615, 1475 cm^{-1}; mass spectrum, m/e (rel intensity) 292 (M^+, 19), 263 (M^+ - CHO, 5), 91 (100); TLC (SiO_2; 2:1 ether/hexanes), R_f ~0.5; high-resolution mass spectrum calcd for $C_{19}H_{16}O_3$ 292.1099, found 292.1090.

6-Methoxynaphthaldehyde (3c). Following the procedure used to prepare **3a**, 2-(2,4-dinitrophenyl)-7-methoxyisoquinolinium bromide (**1c**) was transformed to **3c** (95%), mp 82–83 °C (lit.[9] mp 82–83 °C).

Naphthaldehyde (3d). With use of the standard procedure used to make **3a**, 2-(2,4-dinitrophenyl)isoquinolinium bromide (**1d**) gave **3d** (80%) as a pale-yellow oil identical in all respects with an authentic sample (Aldrich Chemical Co.).

Preparation of 6,7-(Methylenedioxy)naphthoic Acid (4a). To a 0 °C solution of naphthaldehyde **3a** (100 mg, 0.5 mmol) in 6 mL of acetone was added 2.67 M chromic acid solution (0.36 mL). The mixture was maintained at 0 °C until TLC analysis (SiO_2, 2:1 ether/hexanes, R_f ~0.28) showed no remaining starting material (8 h). Excess reagent was destroyed with isopropyl alcohol. Most of the acetone was evaporated and the residue poured into water and extracted with EtOAc. The combined organic extracts were concentrated, passed through a pad of silica gel, and evaporated, yielding 101 mg of **4a** (94%): mp 267–268 °C (CH_2Cl_2/MeOH); NMR (Me_2SO-d_6) δ 6.12 (s, 2 H), 7.25–7.48 (m, 2 H), 7.80–8.04 (m, 2 H), 8.24 (s, 1 H); IR ($CHCl_3$) 3100, 1690, 1615 cm^{-1}; mass spectrum, m/e (relative intensity) 216 (M^+, 100), 199 (25), 171 (M^+ - CO_2H, 2); high-resolution mass spectrum calcd for $C_{12}H_8O_4$ 216.0423, found 216.0417.

6-Methoxy-7-(benzyloxy)naphthoic Acid (4b). By the method described for the preparation of **4a**, naphthaldehyde **3b** was converted to **4b** (95%): mp 228–230 °C; NMR (Me_2SO-d_6) δ 3.75 (s, 3 H), 5.15 (s, 2 H), 7.04–8.10 (m, 9 H), 8.26 (s, 1 H); IR ($CHCl_3$) 3050, 1690, 1615 cm^{-1}; mass spectrum, m/e (relative intensity) 308 (M^+, 10), 91 (100); high-resolution mass spectrum calcd for $C_{19}H_{16}O_4$ 308.1049, found 308.1045.

6-Methoxynaphthoic Acid (4c). Following the method used to prepared **4a**, naphthaldehyde **3c** was oxidized to **4c** (94%), mp 166–167 °C (lit.[9] mp 167–168 °C).

Naphthanoic Acid (4d). Jones oxidation of naphthaldehyde (**3d**) as described for **4a** gave **4d** (88%) identical with an authentic sample.

***tert*-Butyl [6,7-(Methylenedioxy)naphthyl]carbamate (5a).** To a solution of naphthoic acid **4a** (100 mg, 0.463 mmol) and triethylamine (103 mg, 1.02 mmol) in 8 mL of anhydrous *t*-butyl alcohol was added diphenylphosphoryl azide (153 mg, 0.555 mmol). After the mixture was heated at reflux under argon for 5 h, the solvent was evaporated. The residue was dissolved in EtOAc, washed with brine, and then chromatographed (SiO_2, CH_2Cl_2) to give 107 mg of **5a** (80%): mp 159 °C; NMR ($CDCl_3$) δ 1.52 (s, 9 H), 5.96 (s, 2 H), 7.04–7.56 (m, 6 H); IR ($CHCl_3$) 3420, 1720, 1610, 1590 cm^{-1}; mass spectrum, m/e (relative intensity) 287 (M^+, 21), 231 (100), 213 (40), 187 (95); TLC (SiO_2, 2:1 ether/hexanes), R_f ~0.6; high-resolution mass spectrum calcd for $C_{16}H_{17}NO_4$ 287.1157, found 287.1165.

***tert*-Butyl [6-Methoxy-7-(benzyloxy)naphthyl]carbamate (5b).** By the method described for the preparation of **5a**, naphthoic acid **4b** was converted to **5b** (74%): mp 134–135 °C; NMR ($CDCl_3$) δ 1.52 (s, 9 H), 3.96 (s, 3 H), 4.98 (s, 1 H), 5.22 (s, 1 H), 7.02–7.84 (m, 11 H); IR ($CHCl_3$) 3425, 1720, 1615, 1590 cm^{-1}; mass spectrum, m/e (relative intensity) 379 (M^+, 18), 323 (100), 275 (23); TLC (SiO_2, 2:1 ether/hexanes), R_f ~0.4; high-resolution mass spectrum calcd for $C_{23}H_{25}NO_4$ 379.1783, found 379.1778.

***tert*-Butyl (6-Methoxynaphthyl)carbamate (5c).** Following the procedure used to prepare **5a**, naphthoic acid **4c** was transformed to **5c** (73%) isolated as a yellow glass: NMR ($CDCl_3$) δ 1.52 (s, 9 H), 3.84 (s, 3 H), 6.82–7.85 (m, 7 H); IR ($CHCl_3$) 3420, 1720, 1615 cm^{-1}; mass spectrum, m/e (relative intensity) 273 (M^+, 21), 257 (38), 215 (82); TLC (SiO_2, 2:1 ether/hexanes), R_f ~0.53; high-resolution mass spectrum calcd for $C_{16}H_{19}NO_3$ 273.1365, found 273.1358.

***tert*-Butyl Naphthylcarbamate (5d).** Curtius rearrangement of **4d** as described for **4a** gave **5d** (76%), mp 98–100 °C (lit.[10] 100–101 °C).

Benzyl (6-Methoxynaphthyl)carbamate (5c, R_2 = CO_2CH_2Ph). To a solution of naphthoic acid **4c** (85 mg, 0.423 mmol), triethylamine (94 mg, 0.926 mmol), and benzyl alcohol (91 mg, 0.841 mmol) in 7 mL of dry dioxane was added diphenylphosphoryl azide (139 mg, 0.505 mmol). After the mixture was refluxed for 5 h, the solvent was evaporated, the residue dissolved in EtOAc, washed with brine, and chromatographed, furnishing 92 mg of **5c** (R_2 = CO_2CH_2Ph) (71%): mp 120–121 °C; NMR ($CDCl_3$) δ 3.80 (s, 3 H), 5.16 (s, 2 H), 6.98–7.74 (m, 12 H); IR ($CHCl_3$) 3420, 1720, 1615 cm^{-1}; mass spectrum, m/e (relative intensity) 307 (M^+, 17), 263 (11), 199 (70), 156 (52); TLC (SiO_2, 2:1 ether/hexanes) R_f ~0.46. Anal. Calcd for $C_{19}H_{17}NO_3$: C, 74.25; H, 5.58; N, 4.56. Found: C, 74.32; H, 5.42; N, 4.68.

Preparation of 6,7-(Methylenedioxy)naphthylamine (6a). Carbamate **5a** (312 mg, 1.09 mmol) was heated at reflux in 15 mL of THF and 3 mL of H_2O with 3 mL of concentrated hydrochloric acid under argon for 40 min. After evaporation of the THF, the aqueous solution was basified at 0 °C with 3 N NH_4OH and extracted with CH_2Cl_2. The combined organic extracts were washed with brine, dried, and evaporated. Crystallization from ether/hexanes afforded 194 mg of **6a** (86%): mp 154–155 °C (lit.[3b] mp 154–155 °C); TLC (SiO_2, CH_2Cl_2), R_f ~0.27.

6-Methoxy-7-(benzyloxy)naphthylamine (6b). By the method used to prepared **6a**, carbamate **5b** was hydrolyzed to **6b** (79%) isolated as a yellow oil after chromatography: NMR ($CDCl_3$) δ 3.96 (s, 3 H), 5.24 (s, 2 H), 7.05–7.68 (m, 11 H); mass spectrum, m/e (relative intensity) 279 (M^+, 11), 188 (72), 105 (30); TLC (SiO_2, 2:1 ether/hexanes), R_f ~0.27; high-resolution mass spectrum calcd for $C_{18}H_{17}NO_2$ 279.1259, found 279.1268.

6-Methoxynaphthylamine (6c). Following the method used to prepare **6a**, carbamate **5c** was transformed to **6c** (79%): mp 63–64 °C (lit.[3c] mp 64–66 °C); NMR ($CDCl_3$) δ 3.84 (s, 3 H), 6.52 (dd, J = 7, 7 Hz, 1 H), 6.92–7.28 (m, 4 H), 7.64 (d, J = 9 Hz, 1 H); TLC (SiO_2, 2:1 ether/hexanes), R_f ~0.30.

Naphthylamine (6d). Hydrolysis of carbamate **5d** as described for **5a** furnished **6d** (73%) identical with an authentic sample (Aldrich Chemical Co.).

(9) Srivastava, K. C.; Dev, S. *Tetrahedron* **1972**, *28*, 1083–1091.

(10) *Beilstein* **1929**, *12*, 1237.

Hydrogenolysis of 5c (R_2 = CO_2CH_2Ph). Carbamate 5c (R_2 = CO_2CH_2Ph) (266 mg, 0.866 mmol) was stirred under a hydrogen atmosphere with 30 mg of 5% Pd/C in 6 mL of MeOH for 1 h. The catalyst was removed by filtration over Celite. Evaporation of the solvent gave 150 mg of 6c (100%) identical with the sample prepared above.

Acknowledgment. The Robert A. Welch Foundation and NATO (Grant No. RG 158.80) are thanked for generous financial support. Dr. David Sawyer is thanked for high-resolution mass spectra.

Registry No. 1a·Br^-, 83379-65-1; 1b·Br^-, 83379-66-2; 1c·Br^-, 83379-67-3; 1d·Br^-, 70211-56-2; 2a (isomer 1), 83379-77-5; 2a (isomer 2), 83461-43-2; 3a, 83379-68-4; 3b, 83379-69-5; 3c, 3597-42-0; 3d, 66-77-3; 4a, 83379-70-8; 4b, 83379-71-9; 4c, 36112-61-5; 4d, 86-55-5; 5a, 83379-72-0; 5b, 83379-73-1; 5c, 83379-74-2; 5c (R_2 = CO_2CH_2Ph), 83379-75-3; 5d, 72594-62-8; 6a, 53811-49-7; 6b, 83379-76-4; 6c, 5302-77-2; 6d, 134-32-7; ethyl vinyl ether, 109-92-2; diphenylphosphoryl azide, 26386-88-9.

Degradation of Aminimides Obtained from Enamines and (Ethoxycarbonyl)nitrene

Lucio Pellacani, Patrizio Pulcini, and Paolo A. Tardella*

Istituto di Chimica Organica dell'Università di Roma, I-00185 Roma, Italy

Received February 2, 1982

Recently, during our ongoing study of nitrene chemistry, we have found a very high ratio of addition to insertion product in the reaction of (ethoxycarbonyl)nitrene (EtOCON) with vinyl chlorides, as compared with the case of other unsubstituted olefins.[1] Aziridines are also the main products in the reaction of $EtOCON_3$ with enol ethers[2] and enol acetates.[3]

We have now extended our study of nitrene reactions to enamines. There are few examples of reactions between nitrenes and amines in the literature. Tertiary amines gave isolable or supposed N–N ylides (aminimides) by addition of electron-deficient nitrene to the nonbonded electron pair.[4] Enamines were, therefore, an interesting substrate for testing the preference of the nitrene to add to the double bond or to the electron pair to give substituted aziridines or aminimides, respectively.

We considered 1-(1-cyclopentenyl)piperidine (2), 1-(1-cyclohexenyl)piperidine (6), 1-(1-cyclopentenyl)pyrrolidine (9), and 1-(1-cyclohexenyl)pyrrolidine (12). EtOCON was generated by α elimination from *N*-[[(4-nitrophenyl)-sulfonyl]oxy]urethan (NBSU, 1) at room temperature.[5] The procedure was modified by adding a threefold excess of triethylamine to the solution of enamine in dichloromethane and then NBSU (1) portionwise, during 1 h.

The piperidine enamines 2 and 6 gave the corresponding aminimides (3 and 7) in 24% and 33% isolated yields, along with minor amounts of *N*-(ethoxycarbonyl)-2-aminocycloalkanones (4 and 8) and 1-[(ethoxycarbonyl)-amino]piperidine (5). Under the same conditions, the enamines containing the pyrrolidine ring gave lower yields of the expected aminimides and comparable or higher amounts of 4, 8, and 1-[(ethoxycarbonyl)amino]pyrrolidine (11). We were never able to isolate the primary product of the nitrene addition to the carbon–carbon double bond, while in the reaction of enamines and dichlorocarbene this was the only isolable product, and it was often stable[6] (Scheme I and experiment a of Table I).

Interestingly, when the reaction on 12 was run without triethylamine (experiment b of Table I), the basic function was fulfilled by the enamine itself, and we found only 8 and 11 (in almost a 1:1 ratio), which seemed thus to be generated from the protonated enamine. In fact, *N*-

Scheme I. Reaction of Enamines NBSU (1) and Et_3N in Dichloromethane at Room Temperature

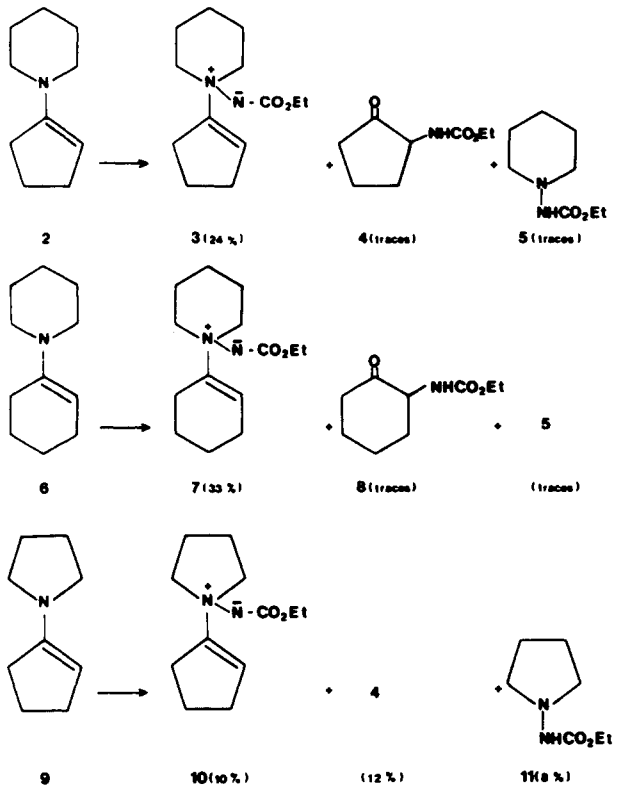
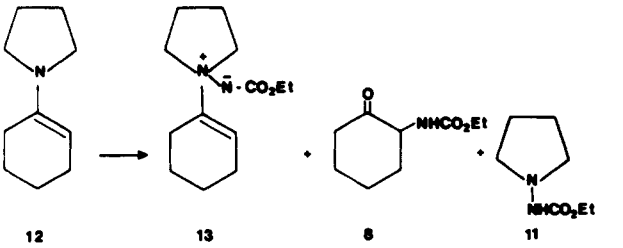


Table I. Reaction of 1-(1-Cyclohexenyl)pyrrolidine (12) or *N*-Cyclohexylidenepyrrolidinium Perchlorate (14) and NBSU (1) in Dichloromethane at Room Temperature under Varied Reaction Conditions



		rel yield, %		
expt	molar ratio	13	8	11
a	1:3 enamine/Et_3N	11	32	57
b	without Et_3N		49	51
c	1:3 perchlorate/Et_3N		36	64
d	1:10 enamine/Et_3N	58	30	12

(1) Pellacani, L.; Persia, F.; Tardella, P. A. *Tetrahedron Lett.* **1980**, *21*, 4967.
(2) Brown, I.; Edwards, O. E. *Can. J. Chem.* **1965**, *43*, 1266.
(3) Keana, J. F. W.; Keana, S. K.; Beetham, D. *J. Org. Chem.* **1967**, *32*, 3057.
(4) (a) Hafner, K.; Zinser, D.; Moritz, K. L. *Tetrahedron Lett.* **1964**, 1733. (b) Tsuchida, T.; Koyama, K.; Mitani, M.; Takeuchi, H. *Bull. Chem. Soc. Jpn.* **1980**, *53*, 1189. (c) Hutchins, M. G. K.; Swern, D. *Tetrahedron Lett.* **1981**, *22*, 4599.
(5) Lwowski, W.; Maricich, T. J. *J. Am. Chem. Soc.* **1965**, *87*, 3630.
(6) (a) Ohno, M. *Tetrahedron Lett.* **1963**, 1753. (b) Wolinsky, J.; Chan, D.; Novak, R. *Chem. Ind. (London)* **1965**, 720. (c) Bergman E. *J. Org. Chem.* **1963**, *28*, 2210.

0022-3263/82/1947-5023$01.25/0