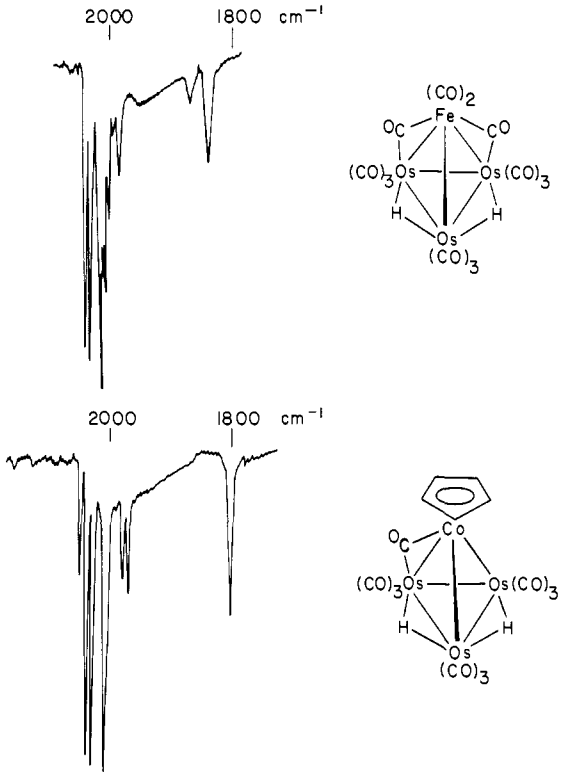

Figure 1. Infrared spectra and proposed structures of $H_2FeOs_3(CO)_{13}$ and $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$.

The classical valence bond representation of $H_2Os_3(CO)_{10}$ implies the presence of an osmium–osmium double bond. In an operative sense, the proposed intermediate, $H_2Os_3(CO)_{10}[Fe(CO)_4]$, can be likened to an $Fe(CO)_4$–olefin complex (eq 3).

$$Fe_2(CO)_9 + H_2Os(CO)_{10} \rightarrow Fe(CO)_5 + H_2Os_3(CO)_{10}[Fe(CO)_4] \rightarrow H_2FeOs_3(CO)_{13} + CO \quad (3)$$

Reaction pathways 1 and 2, especially the latter, afford significantly improved yields over earlier preparations of $H_2FeOs_3(CO)_{13}$. This cluster was reported as a side product isolated in 6% yield from the reaction between $H_2Os(CO)_4$ and $Fe_2(CO)_9$.[10] A later report[5] revealed that the reaction between the electron-precise cluster $Os_3(CO)_{12}$ and $Fe(CO)_4^{2-}$ followed by protonation gives $H_2FeOs_3(CO)_{13}$ in 9% yield.

In view of the success of reaction 2 as a high-yield pathway to an iron–osmium cluster, we extended this general procedure to the preparation of a new cobalt–osmium cluster via the displacement reaction 4.

$$(\eta^5\text{-}C_5H_5)Co(CO)_2 + H_2Os_3(CO)_{10} \rightarrow H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10} + 2CO \quad (4)$$

An excess of $(\pi^5\text{-}C_5H_5)Co(CO)_2$ will slowly react at 90 °C with $H_2Os_3(CO)_{10}$ in toluene to yield the new cluster $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$ in 60% yield. This compound is isolatable by column chromatography (silica gel 80:20 hexane/benzene) as an air-stable black-green solid. Its mass spectrum exhibits a high-mass cutoff at m/e 982, and the relative intensity distributions of the parent-ion cluster and those fragments due to loss of CO are in good agreement with the calculated intensity values for a three-osmium system.[11] High-resolution mass measurement on the m/e 982 ion confirms an elemental composition of $^1H_7{}^{12}C_{15}{}^{16}O_{10}{}^{59}Co^{192}Os_3$ (obsd 981.8223; calcd 981.8207).

The infrared spectrum of $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$ in cyclohexane consists of the following bands in the carbonyl stretching region: 2095 (m), 2068 (vs), 2050 (vs), 2012 (vs), 2000 (sh), 1977 (m), 1968 (m), 1800 (s) cm^{-1}. On the basis of comparison of infrared spectra, Geoffroy and Gladfelter[5] assign a tetrahedral structure of $H_2FeOs_3(CO)_{13}$ analogous to the crystallographically established tetrahedral structure of $H_2FeRu_3(CO)_{13}$.[12] Variable temperature ^{13}C NMR spectra of $H_2FeOs_3(CO)_{13}$ are fully consistent with spectra reported[5] for $H_2FeRu_3(CO)_{13}$. In Figure 1, the infrared spectrum and structure of $H_2FeOs_3(CO)_{13}$ are compared with the infrared spectrum and our proposed structure of $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$. The bridging carbonyl regions of these spectra differ significantly. In the case of $H_2FeOs_3(CO)_{13}$, two bands are observed at 1875 and 1848 cm^{-1} which Geoffroy and Gladfelter[5] attribute to symmetric and asymmetric stretching of the two equivalent bridging CO groups. The single, sharp bridging CO band at 1800 cm^{-1} in the infrared spectrum of $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$ is consistent with the presence of only one bridging CO ligand in this molecule.

In addition to one sharp resonance of intensity five at τ 4.53, due to the cyclopentadienyl hydrogens, the low-temperature (−80 °C) 90-MHz ^1H NMR spectrum of $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$ reveals the presence of two distinct bridging hydrogen resonances at τ 27.17 and τ 30.89. This observation permits distinction between the proposed structure and one in which the positions of hydrogens generate a cluster structure with apparent C_s symmetry. As the temperature is raised, the hydride resonances broaden and at 22 °C coalesce into one weak broad peak centered at τ 29.03. The cyclopentadienyl resonance is temperature independent. The ^{13}C NMR spectrum at −80 °C $\{^1H\}$ shows one bridge carbonyl and nine nonequivalent terminal carbonyls.

A new pentanuclear iron–osmium cluster, $H_2Fe_2Os_3(CO)_{16}$, has been isolated in low yield (<2%) from reaction 1. The low-resolution mass spectrum of this material exhibits a cutoff at m/e 1138 while high-resolution mass measurements confirm an elemental composition of $^1H_2{}^{12}C_{16}{}^{16}O_{16}{}^{56}Fe_2{}^{192}Os_3$ (calcd 1137.6877; obsd 1137.6897). Its infrared spectrum contains bands only in the terminal carbonyl region at 2086 (vs), 2070 (vs), 2062 (vs), 2040 (w), 2030 (s), 2005 (m), and 1983 (w) cm^{-1}. The room-temperature 90-MHz ^1H NMR spectrum of $H_2Fe_2Os_3(CO)_{16}$ consists of a single resonance at τ 31.4. Work is currently directed towards improving the yield of this pentanuclear compound.

Acknowledgment. We thank the National Science Foundation for support of this work through Grant CHE79-18148. D.G.A. thanks The Ohio State University Graduate School for a Postdoctoral Fellowship.

(10) J. R. Moss and W. A. G. Graham, *J. Organomet. Chem.*, **23**, C23 (1970).

(11) M. A. Andrews, S. W. Kirtley, and H. D. Kaesz, *Adv. Chem. Ser.*, *No. 167*, 215 (1974).

(12) C. J. Gilmore and P. Woodward, *J. Chem. Soc., Chem. Commun.*, 1463 (1970).

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Received March 21, 1980

General Methodology for *cis*-Hydroisoquinoline Synthesis: Synthesis of Reserpine[1]

Sir:

Reserpine (**1**) has figured prominently during the last 3 decades as a compound of considerable medicinal importance, due largely to its extensive use in the treatment of hypertension and mental disorders.[2] Its isolation from the roots of the tropical shrub

(1) Taken in part from the Ph.D Thesis of J. M. S., Harvard University, 1980.

(2) (a) Woodson, R. E.; Younken, H. W.; Schlittler, E.; Schneider, J. A. "Rauwolfia: Botany Pharmacognosy, Chemistry and Pharmacology"; Little, Brown and Co.: Boston, 1957. (b) Monachino, J. *Econ. Bot.* **1954**, *8*, 349. (c) Chatterjee, A.; Pakrashi, S.; Werner, G. *Fortschr. Chem. Org. Naturst.* **1956**, *13*, 346.

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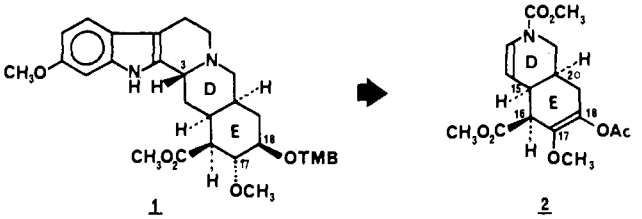


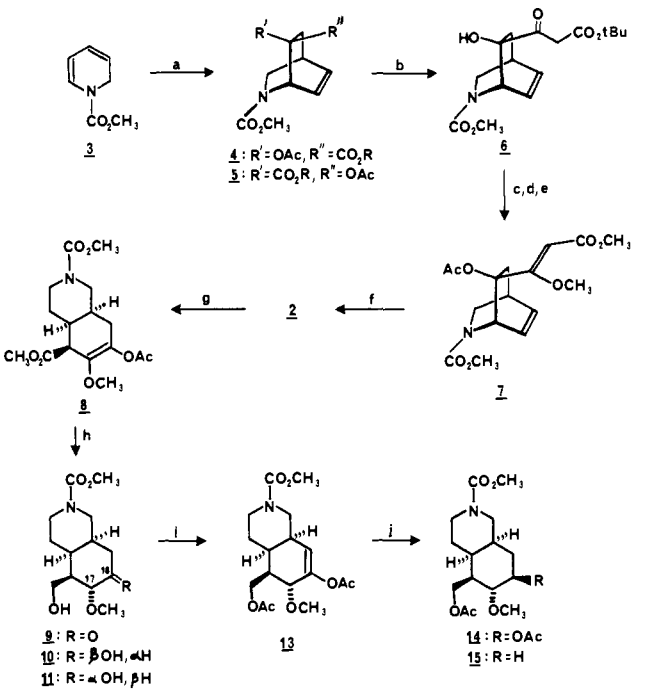
1 2

Rauwolfia serpentina Benth. was first reported in 1952 by Schlittler et al.,[3] and its structure and stereochemistry[4] were established soon thereafter through the elegant investigations of the groups of Schlittler, van Tamelen, Weisenborn, and Wenkert.[5] Hardly a year after these reports became public, Woodward and co-workers[6] reported the first synthesis of this pentacyclic indole alkaloid. Following a hiatus of over 20 years, a second synthesis of reserpine was completed by Pearlman.[7] Described herein is a synthesis of reserpine which is based on our previous studies[8a] of a method for *cis*-hydroisoquinoline synthesis.

For the application of this method to a synthesis of reserpine, it was expected that *cis*-hydroisoquinoline (**2**)[9] could serve as a key D,E-ring precursor since cleavage of its acetate group would introduce a ketone at C-18 which could be used to control the C-17 stereochemistry. Subsequent reduction of this ketone from the convex face would establish the fifth E-ring stereocenter and leave coupling of the derived product to a 6-methoxytryptophyl unit as the final phase of the synthesis.

The investigation of the initial phase of this strategy focused on the preparation of hydroisoquinoline **2**, which we found could be readily accomplished (Scheme I: **3** → **4** → **6** → **7** → **2**) by extension of the previously reported[8] Diels–Alder/Cope rearrangement sequence. Thus, cycloaddition of either the methyl or the ethyl ester of 2-acetoxyacrylic acid and methyl 1,2-dihydropyridine-1-carboxylate (**3**)[10] provided esters **4** and **5** in a ratio of 2:1 [R = Me (76%); R = Et (58%)].[11] Condensation of **4** with the lithium enolate of *tert*-butyl acetate afforded keto ester **6** in 89% yield. The acetylation[12] of **6** followed by sequential treatment of the resulting acetoxy enol acetate with trifluoroacetic acid and ethereal diazomethane in methanol[13] provided the isoquinuclidene

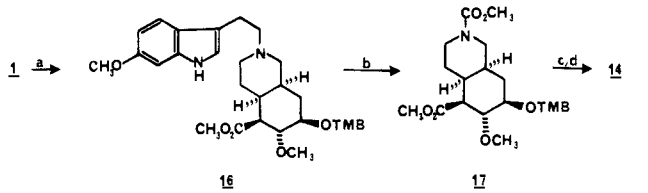
Scheme I



3; **4**: R′ = OAc, R″ = CO_2R; **5**: R′ = CO_2R, R″ = OAc; **6**; **7**; **8**; **9**: R = O; **10**: R = βOH, αH; **11**: R = αOH, βH; **13**; **14**: R = OAc; **15**: R = H

[a] CH_2=C(OAc)CO_2R, $PhCH_3$, hydroquinone, 120 °C, 56 h. [b] $LiCH_2CO_2$-*t*-Bu, THF, -78 °C → room temperature. [c] Ac_2O, NEt_3, DMAP. [d] CF_3CO_2H, room temperature. [e] CH_2N_2, CH_3OH, Et_2O. [f] Xylene solution, 243 °C, 3 h. [g] H_2, Pd/C, EtOAc. [h] LAH, Et_2O, 0 °C, 2 min. [i] $LiN(SiMe_3)_2$, -10 °C, THF; then AcCl, -78 °C. [j] H_2, 10% Pd/C, EtOAc.

Scheme II



16; **17**

[a] $HCONH_2$, HCO_2H, Δ.[19] [b] $ClCO_2CH_3$, C_6H_6, Δ.[19] [c] LAH. [d] Ac_2O, NEt_3, DMAP.

7 in 65% overall yield. Thermolysis of a xylene solution of **7** in a resealable Pyrex tube afforded the key hydroisoquinoline **2** in 78% yield.

Partial hydrogenation of **2** was effected at this juncture in order to allow for subsequent correlation (vide infra) of the product of the synthetic sequence with a degradation product of reserpine. The reduction product **8** so obtained (95%) when treated with lithium aluminum hydride followed by addition of ethyl acetate and basic workup[14] provided a *single ketone*, **9** (78%), along with diols **10** and **11** (ca. 4% each). Since epimerization of **9** with sodium methoxide produced a 6:1 mixture of **9** and its C-17 epimer **12**, the stereospecificity observed in the formation of **9** is presumably the result of kinetic protonation[15] of the enol aluminate intermediate derived from **8**.

Introduction of the final E-ring stereocenter by catalytic hydrogenation of **9** provided the desired diol **10**[16] as the major product accompanied by its C-18 epimer **11**[17] (**10**:**11**, 2:1). In

(3) Müller, J. M.; Schlittler, E.; Bein, H. J. *Experientia* **1952**, *8*, 338.

(4) The abbreviation TMB will be used throughout this paper to designate the 3,4,5-trimethoxybenzoyl group.

(5) (a) Dorfman, L.; Furlenmeier, A.; Huebner, C. F.; Lucas, R.; MacPhillamy, H. B.; Mueller, J. M.; Schlittler, E.; Schwyzer, R.; St. Andre, A. F. *Helv. Chim. Acta* **1954**, *37*, 59. (b) Huebner, C. F.; MacPhillamy, H. B.; Schlittler, E.; St. Andre, A. F. *Experientia* **1955**, *11*, 303. (c) van Tamelen, E. E.; Hance, P. D. *J. Am. Chem. Soc.* **1955**, *77*, 4692. (d) Wenkert, E.; Liu, L. H. *Experientia* **1955**, *11*, 302. Huebner, C. F.; Wenkert, E. *J. Am. Chem. Soc.* **1955**, *77*, 4180. (e) Diassi, P. A.; Weisenborn, F.; Dylion, C. M.; Wintersteiner, O. *Ibid.* **1955**, *77*, 4687.

(6) Woodward, R. B.; Bader, F. E.; Bickel, H.; Frey, A. J.; Kierstead, R. W. *J. Am. Chem. Soc.* **1956**, *78*, 2023, 2657; *Tetrahedron* **1958**, *2*, 1.

(7) Pearlman, B. *J. Am. Chem. Soc.* **1979**, *101*, 6398, 6404.

(8) (a) Wender, P. A.; Schaus, J. M.; Torney, D. C. *Tetrahedron Lett.* **1979**, 2485; cf. ref 8b. (b) A conceptually related approach to reserpine has been reported by Stevens, R. V.; Moran, J. R. ACS/CJS Chemical Congress, April 1–6, 1979; ORGN 289.

(9) For the hydroisoquinoline derivatives described herein, the numbering scheme is as indicated for **2** and is a correlation with the numbering scheme used for the yohimbine alkaloids.

(10) Fowler, F. W. *J. Org. Chem.* **1972**, *37*, 1321.

(11) All compounds reported were homogeneous by TLC and gave satisfactory IR and NMR spectra and exact mass and/or combustion analyses. Partial spectroscopic data for selected intermediates are as follows. **2**: NMR ($CDCl_3$) δ 6.99–6.58 (m, 1 H), 4.92–4.53 (m, 1 H), 3.75 (s, 3 H), 3.68 (s, 3 H), 3.59 (s, 3 H), 2.15 (s, 3 H). **9**: NMR ($CDCl_3$) δ 3.68 (s, 3 H), 3.49 (s, 3 H). **10**: NMR ($CDCl_3$) δ 3.66 (s, 3 H), 3.57 (s, 3 H). **11**: NMR ($CDCl_3$) δ 3.67 (s, 3 H), 3.41 (s, 3 H). **14**: NMR ($CDCl_3$) δ 4.96–4.54 (m, 1 H), 3.66 (s, 3 H), 3.39 (s, 3 H), 2.05 (s, 6 H). **23**: NMR ($CDCl_3$) δ 9.87 (d, J = 1 Hz, 1 H), 7.64 (br s, 1 H), 7.36–7.13 (m, 1 H), 7.28 (s, 2 H), 6.81 (s, 1 H), 6.72 (dd, J = 2, 8 Hz, 1 H), 5.31–4.90 (m, 1 H), 3.89 (s, 3 H), 3.88 (s, 6 H), 3.83 (s, 3 H), 3.49 (s, 3 H). **25**: NMR ($CDCl_3$) δ 7.37–7.14 (m, 2 H), 7.31 (s, 2 H), 6.77 (dd, J = 2, 8 Hz, 1 H), 5.03 (br d, J = 11 Hz, 1 H), 3.91 (s, 9 H), 3.85 (s, 3 H), 3.77 (s, 3 H), 3.53 (s, 3 H); IR (KBr) 1735, 1710, 1588 cm^{-1}; UV (EtOH) (λ_{max}, log ε) 233 (4.3), 267 (4.1), 295 (3.9 nm). **26**: NMR ($CDCl_3$) δ 9.88 (d, J = 0.7 Hz, 1 H), 7.36–7.14 (m, 2 H), 7.31 (s, 2 H), 6.26 (dd, J = 2, 8 Hz, 1 H), 5.27–4.86 (m, 1 H), 3.91 (s, 9 H), 3.85 (s, 3 H), 3.56 (s, 3 H).

(12) Hassner, A.; Krepski, L.; Alexanian, V. *Tetrahedron* **1978**, *34*, 2069.

(13) Eistert, B.; Arndt, F.; Loewe, L.; Ayca, A. *Ber.* **1951**, *84*, 156.

(14) Fieser, L. F; Fieser, M. "Reagents for Organic Synthesis", Wiley: New York, 1967; Vol. 1, p 584.

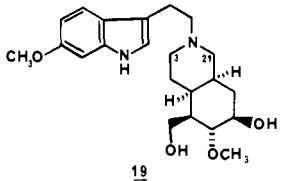
(15) This stereochemistry may result from *intramolecular* delivery of a proton to the C-17 carbon by the C-16 hydroxymethyl substituent.

(16) This assignment is based on a spectroscopic comparison of **10** with an authentic sample derived from (-)-reserpine (Boehringer, Ingelheim). Cf. Scheme II.

(17) In accord with this assignment, $NaBH_4$ reduction of **9** gave **11**. Cf.: Kowalski, C.; Creary, X.; Rollin, A.; Burke, M. *J. Org. Chem.* **1978**, *43*, 2601.

accord with the expectation that the stereoselectivity of the hydrogenation would be improved if the π system to be reduced were closer to the structural feature which induces the stereoselectivity (the cis-ring fusion), enol acetate **13**[18] provided, upon hydrogenation [H_2 (1 atmosphere), Pd/C, EtOAc], *a single diacetate* **14** (79%) along with the hydrogenolysis product **15** (14%). The stereochemistry of diacetate **14** was established by spectroscopic comparison of this compound with an authentic sample derived from (-)-reserpine, according to the degradation sequence[19] delineated in Scheme II. Thus, from the Diels-Alder adduct **4**, the introduction of all the E-ring stereocenters was achieved *with full stereocontrol.*

Completion of the synthesis based on **14**[20] required introduction of the methoxytryptophyl moiety and adjustment of the E-ring appendages. The former objective was accomplished by conversion of **14** with (trimethylsilyl) iodide[21] to the corresponding free amine **18** (90%), which upon alkylation with 6-methoxytryptophyl bromide[22] gave 2,3-secoreserpinediol (**19**) in 85% yield. Oxidative



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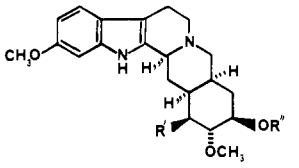
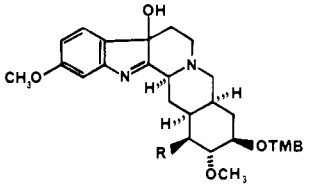


20: R' = CH_2OH, R'' = H
21: R' = CH_2OH, R'' = TMB
22: R' = CH_2OTMB, R'' = TMB
23: R' = CHO, R'' = TMB
24: R' = CO_2CH_3, R'' = TMB



25: R = CO_2CH_3
26: R = CHO

cyclization[23] of this compound followed by $NaBH_4$ reduction produced isoreserpinediol (**20**,[24] 45%) and an isomeric diol (30%) which is presumed to be an inside reserpinediol.[25] Monoester **21** was prepared by treatment of **20** with excess 3,4,5-trimethoxybenzoyl chloride (53%) followed by selective hydrolysis (0.3 M KOH, MeOH, 25 °C, 5 min, 62%) of the resulting diester **22**. Oxidation of **21** with $Me_2SO/DCC/H_3PO_4$[26] gave aldehyde **23** (65%) and a product (20%) resulting from Pummerer rearrangement. The aldehyde, when treated with acetone cyanohydrin in the presence of triethylamine, gave in 86% yield the cyanohydrin which reacted with Me_2SO/oxalyl chloride[27] to provide, after addition of methanol, overoxidized products assigned as ester **25** (33%) and aldehyde **26** (43%). Reduction of ester **25** with $NaBH_4$ followed by treatment with acid afforded, in 85% yield, isoreserpine (**24**).[28] Since four methods are available for the conversion of isoreserpine to reserpine,[29] the described synthesis constitutes a formal, stereospecific synthesis of reserpine based on the Diels-Alder adduct **4**. Efforts to extend this method of hydroisoquinoline synthesis and to more fully exploit the advantages inherent in this general strategy for alkaloid synthesis are in progress.

Acknowledgment. We thank the National Science Foundation for support of this research (CHE-7821463).

(18) **13** was prepared by reaction of **9** with $LiN(SiMe_3)_2$ followed by quenching with excess acetyl chloride at -78 °C.

(19) Sakai, S.; Ogawa, M. *Chem. Pharm. Bull.* **1978**, *26*, 678; *Heterocycles* **1978**, *10*, 67.

(20) Due to the concomitant development of the synthetic and degradative work, all subsequent transformations were performed with material derived from (-)-reserpine.

(21) Jung, M. E.; Lyster, M. A. *J. Am. Chem. Soc.* **1977**, *99*, 968; *J. Chem. Soc., Chem. Commun.* **1978**, 315.

(22) Hydrolysis of the acetate subunits occurred under the conditions of the alkylation [6-methoxytryptophyl bromide (3 equiv)/MeOH/K_2CO_3/reflux/25 h]. 6-Methoxytryptophyl bromide was prepared by treatment of 6-methoxytryptophol with PBr_3. This alcohol was prepared by $LiAlH_4$ reduction of the methyl ester corresponding to the known[6] 2-(3-indolyl)-2-oxoacetyl chloride.

(23) (a) Wenkert, E.; Wickberg, B. *J. Am. Chem. Soc.* **1962**, *84*, 4914. (b) Morrison, G. C.; Cetenko, W.; Shavel, J., Jr. *J. Org. Chem.* **1967**, *32*, 4089. (c) Gutzwiller, J.; Pizzolato, G.; Uskokovic, M. *J. Am. Chem. Soc.* **1971**, *93*, 5908. (d) Stork, G.; Guthikonda, N. *Ibid.* **1972**, *94*, 5110. (e) Aimi, N.; Yamanaka, E.; Endo, J.; Sakai, S.; Haginiwa, J. *Tetrahedron Lett.* **1972**, 1081. (f) *Tetrahedron* **1973**, *29*, 2015.

(24) Identical by NMR with material derived from the lithium aluminum hydride reduction of (-)-isoreserpine: MacPhillamy, H. B.; Huebner, C. F.; Schlittler, E.; St. Andre, A. F.; Ulshafer, P. R. *J. Am. Chem. Soc.* **1955**, *77*, 4335.

(25) In accord with convention (cf. ref 23e,f), inside reserpinediol is that product which arises from cyclization of the iminium salt derived by oxidation of **19** at C-21.

(26) Albright, J. D.; Goldman, L. *J. Org. Chem.* **1965**, *30*, 1107.

(27) Swern, D.; Omura, K. *Tetrahedron* **1978**, *34*, 1651.

(28) Identical with an authentic sample of (-)-isoreserpine (Gaskell, A. J.; Joule, J. A. *Tetrahedron* **1967**, *23*, 4053) by NMR and IR spectroscopy, thin-layer chromatography, and melting point. A mixture melting point was undepressed.

(29) (a) Huebner, C. F.; Kuehne, M. E.; Korzun, B.; Schlittler, E. *Experientia* **1956**, *12*, 249. (b) Weisenborn, F. L.; Diassi, P. A. *J. Am. Chem. Soc.* **1956**, *78*, 2022.

(30) Fellow of the Alfred P. Sloan Foundation, 1979-1981.

(31) National Science Foundation Fellow, 1975-1978.

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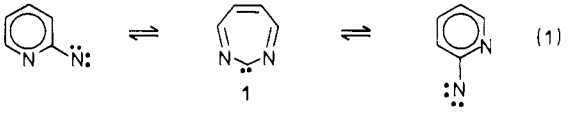
Received May 27, 1980

Isolation of Diazacycloheptatetraenes from Thermal Nitrene-Nitrene Rearrangements[1]

Sir:

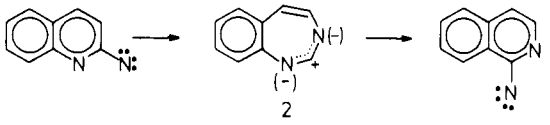
The first examples of nitrene-nitrene rearrangements were reported in 1969, when we demonstrated the thermal gas-phase interconversion of 2-pyridylnitrenes via an intermediate which has "an arrangement of atoms as in 2,7-diazatropylidene" (**1**).[3] Since



(1)

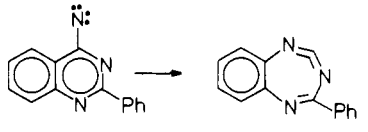
1

the rearrangement took place just as easily in benzo-annelated systems (quinolines and phenanthridines), we subsequently formulated the seven-membered ring intermediates as resonance forms of cyclic carbodiimides,[4,5] e.g., **2**.[4]



2

In 1975, we submitted evidence for the thermal ring expansion of the nitrene **3** to the carbodiimide **4**.[6] In further work, a rearranged dimer of **4** was isolated,[7] and, finally, **4** itself was



3 **4**

(1) Part VIII of the series "Hetarylnitrenes". Part VII: see ref 2. The financial support of the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie is gratefully acknowledged.

(2) Wentrup, C.; Thētaz, C.; Tagliaferri, E.; Lindner, H. J.; Kitschke, B.; Winter, H.-W.; Reisenauer, H. P. *Angew. Chem.* **1980**, *92*, 556-557.

(3) Crow, W. D.; Wentrup, C. *J. Chem. Soc. D* **1969**, 1387.

(4) Wentrup, C. *Tetrahedron* **1971**, *27*, 367-374.

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