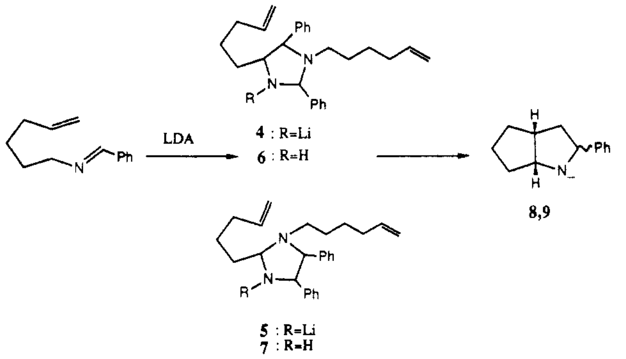
In order to quantify this trend, kinetics were performed on the cyclization in entry 2. The expected first-order kinetics were not observed; in fact, evidence for an intermediate was obtained. Performing the deprotonation in THF-d_8 allowed observation of the rapid buildup of two new compounds by ^{1}H NMR, which were identified as the lithioimidazolidines **4** and **5**. These were slowly converted to the bicyclic pyrrolidines **8** and **9** after several hours at room temperature. Apparently, initial deprotonation is slow enough to allow intermolecular cycloaddition with the imine portion of a molecule of starting material.[10] In a separate experiment, workup at partial conversion allowed the isolation of the protio derivatives **6** and **7** of undefined stereochemistry. Resubjection of these imidazolidines to the reaction conditions (LDA, THF, room temperature) also gave **8** and **9**. Hence, for the first time, it has been demonstrated that lithioimidazolidines are subject to anionic cycloreversion to 2-azaallyl anions.[11] Interestingly, this may allow a *new route to 2-azaallyl anions which does not rely on imine deprotonation*,[12] thereby obviating our reliance on one or more aryl groups in the anion.[13]



In summary, the first examples of intramolecular 2-azaallyl anion cycloadditions are reported. High stereoselectivity and a tolerance for olefin substitution makes the method very promising for synthetic endeavors. Our results in the imidazolidine area will be reported shortly, as well as our efforts directed toward natural products synthesis.

Acknowledgment. We are grateful to the Camille and Henry Dreyfus Foundation and to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for the support of this research. We would also like to thank the University of Michigan for a Baer Fellowship awarded to M.A.W.

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(10) Cycloaddition of 2-azaallyl anions with imines to give imidazolidines has been previously observed.[5] For more recent examples, see: (a) Vo-Quang, L.; Vo-Quang, Y. *J. Heterocycl. Chem.* **1982**, *19*, 145. (b) Gracheva, R. A.; Potapov, V. M. Sivov, B. A.; Sivova, L. I. *J. Org. Chem. USSR* (*Engl. Transl.*) **1982**, *17*, 1963.

(11) Although the cycloreversion of lithioimidazolidines to 2-azaallyl anions has not been previously reported, similar anionic cycloreversions are known; see ref 5 and: (a) Bianchi, G.; De Micheli, C.; Gandolfi, R. *Angew. Chem., Int. Ed. Engl.* **1979**, *18*, 721. (b) Kauffmann, T.; Busch, A.; Habersaat, K.; Scheerer, B. *Tetrahedron Lett.* **1973**, 4047. Imidazolidines have been reported to undergo cycloreversion to azomethine ylides: Amornraksa, K.; Grigg, R. *Tetrahedron Lett.* **1980**, *21*, 2197.

(12) For example, imidazolidines may be synthesized from carbonyl compounds and vicinal diamines. Anionic cycloreversion would then provide 2-azaallyl anions.

(13) Alternatively, oxidative cleavage of benzylic amines to amino acids[a] followed by decarboxylation[b] provides a route to the unsubstituted bicyclic pyrrolidines: (a) Hill, R. K.; Prakash, S. R.; Zydowsky, T. M. *J. Org. Chem.* **1984**, *49*, 1666. Ayres, D. C. *J. Chem. Soc., Perkin Trans. 1* **1978**, 585. (b) Dean, R. T.; Padgett, H. C.; Rapport, H. *J. Am. Chem. Soc.* **1976**, *98*, 7448.

(14) (a) Entries 1–3 use 5-hexenylamine: Cogdell, T. J. *J. Org. Chem.* **1972**, *37*, 2541. (b) Entry 4: from 5-hepten-1-ol (Ohloff, G.; Vial, C.; Naf, F.; Pawlak, M. *Helv. Chim. Acta* **1977**, *60*, 1161) by the sequence TsCl, pyr; NaN_3, Me_2SO; LAH, ether. (c) Entry 5: from 4-benzoylbutyric acid (Aldrich) by the sequence MeOH, HCl; Ph_3PCH_3Br, KO-*t*-Bu, THF; LAH, ether; TsCl, pyr; NaN_3, Me_2SO; LAH, ether. (d) Entry 6: same sequence as (c), except NaCN instead of NaN_3. (e) Entry 7: 6-bromo-1-hexene, NaCN, Me_2SO; LAH, ether.

(15) Lithium hydride elimination has been proposed to explain nonstereospecificity in some 2-azaallyl anion cycloadditions. See ref 10 and: (a) Vo-Quang, L.; Gaessler, H.; Vo-Quang, Y. *Angew. Chem., Int. Ed. Engl.* **1981**, *20*, 880. (b) Vo-Quang, L.; Vo-Quang, Y.; Pouet, M. J.; Simonnin, M. P. *Tetrahedron* **1981**, *37*, 4343.

A Well-Characterized, Highly Active, Lewis Acid Free Olefin Metathesis Catalyst[1]

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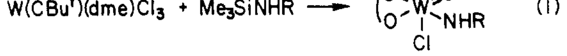
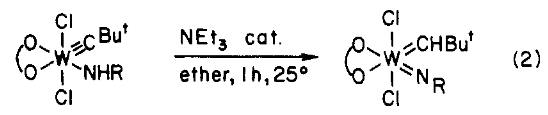
For almost 5 years it has been known that W(VI) alkylidene complexes will metathesize olefins.[2] Osborn has shown that what are likely to be highly electrophilic, cationic alkylidene complexes are formed in the presence of Lewis acids and that several of these systems will metathesize *cis*-2-pentene extremely efficiently.[2c,d] But olefins that contain basic functionalities probably will not be metathesized by catalysts that depend on a Lewis acid cocatalyst for activity. We report here the synthesis and reactivity of the first well-characterized, highly active, neutral olefin metathesis catalyst.

The design of efficient acetylene metathesis catalysts of the type $W(C\text{-}t\text{-}Bu)(OR'')_3$,[3] and the isolation of distorted TBP tungstenacyclobutadiene complexes of the type $W(C_3R'_3)(OR'')_3$,[3] led us to propose that alkylidene complexes of the type $W(CH\text{-}t\text{-}Bu)(NR)(OR'')_2$ would metathesize olefins via TBP tungstenacyclobutane intermediates if R and R'' are chosen carefully. By analogy with $W(C\text{-}t\text{-}Bu)(O\text{-}2,6\text{-}C_6H_3\text{-}i\text{-}Pr_2)_3$[3a] and $W(C\text{-}t\text{-}Bu)[OCMe(CF_3)_2]_3$,[3b] we decided that $W(CH\text{-}t\text{-}Bu)(N\text{-}2,6\text{-}C_6H_3\text{-}i\text{-}Pr_2)[OCMe(CF_3)_2]_2$ would be a good candidate. It was finally prepared via the sequence of reactions shown in eq 1–3.



R = 2,6-diisopropylphenyl; O‿O = dme



All reactions proceed in high yield and the crystalline products

$$W(CH\text{-}t\text{-}Bu)(NR)(dme)Cl_2 + 2LiOR_F \xrightarrow{\text{ether}} W(CH\text{-}t\text{-}Bu)(NR)(OR_F)_2 \quad (3)$$

$$OR_F = OCMe(CF_3)_2$$

have all been characterized fully.[4a] The crucial reaction shown in eq 2 is based upon earlier work.[5] We assume $W(CH\text{-}t\text{-}Bu)(NR)(OR_F)_2$ to a pseudotetrahedral monomer in which H_α and C_β of the neopentylidene ligand lie in the C_α–W–N plane as a result of the strong donation of the imido ligand's π-electron pair. The alkylidene ligand does seem to be somewhat distorted toward a large W–C_α–C_β angle, as judged by $\delta(H_\alpha) = 8.87$ and $J_{CH_\alpha} = 110$ Hz,[4b] presumably as a result of the somewhat electrophilic and electronically unsaturated nature of the metal center.[6]

$W(CH\text{-}t\text{-}Bu)(NR)(OR_F)_2$ in pentane reacts rapidly with ethylene (2.4 equiv, 0 °C, 20 min) or with vinyltrimethylsilane

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(1) Multiple Metal-Carbon Bonds. 43. For part 42, see: Freudenberger, J. H.; Schrock, R. R. *Organometallics*, in press.

(2) (a) Schrock, R. R.; Rocklage, S. M.; Wengrovius, J.; Rupprecht, G.; Fellmann, J. *J. Mol. Catal.* **1980**, *8*, 73. (b) Wengrovius, J.; Schrock, R. R.; Churchill, M. R.; Missert, J. R.; Youngs, W. J. *J. Am. Chem. Soc.* **1980**, *102*, 4515. (c) Kress, J.; Wesolek, M.; LeNy, J.-P.; Osborn, J. A. *J. Chem. Soc., Chem. Commun.* **1981**, 1039. (d) Kress, J.; Wesolek, M.; Osborn, J. A. *J. Chem. Soc., Chem. Commun.* **1982**, 514.

(3) (a) Churchill, M. R.; Ziller, J. W.; Freudenberger, J. H.; Schrock, R. R. *Organometallics* **1984**, *3*, 1554. (b) Freudenberger, J. H.; Schrock, R. R.; Churchill, M. R.; Rheingold, A. L.; Ziller, J. W. *Organometallics* **1984**, *3*, 1563.

(4) (a) Full preparative, analytical, and spectroscopic details are provided as supplementary material. (b) In $W(CH\text{-}t\text{-}Bu)(NR)(OR_F)_2$ $\delta(C_\alpha) = 253.9$, $J_{CW} = 198$ Hz, $J_{HW} = 13$ Hz.

(5) Rocklage, S. M.; Schrock, R. R.; Churchill, M. R.; Wasserman, H. J. *Organometallics* **1982**, *1*, 1332.

(6) Schrock, R. R. *Acc. Chem. Res.* **1976**, *12*, 98.

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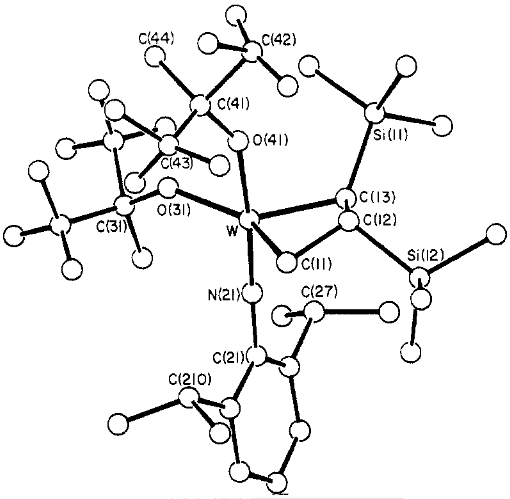

Figure 1. Drawing of $\overline{\mathrm{WCH(Me_3Si)CH(Me_3Si)CH_2}}$(N-2,6-$C_6H_3$-*i*-$Pr_2$)[$OCMe(CF_3)_2$]$_2$. For clarity, atoms are depicted as spheres of arbitrary radius and hydrogen atoms are omitted. The axial alkoxide ligand is disordered.[8] W–C(11) = 2.099 (11) Å, W–C(13) = 2.066 (11) Å, W···C(12) = 2.372 (11) Å, WC(11)C(13)/C(11)C(12)C(13) dihedral angle = 24.8°.

(2 equiv, 25 °C, 10 min) to give 1 equiv of *tert*-butylethylene (*only*) and the tungstenacyclobutane complexes, ($\overline{\mathrm{WCH_2CH_2CH_2}}$)(NR)($OR_F$)$_2$ and [$\overline{\mathrm{WCH(Me_3Si)CH(Me_3Si)CH_2}}$](NR)($OR_F$)$_2$, virtually quantitatively.[4,7] The X-ray structure of the latter[8] shows it to be a distorted trigonal bipyramid with and equatorial WC_3 ring and an axial imido group (Figure 1). The W–C_α bonds are somewhat shorter than typical W–C single bonds (2.099 (11) and 2.066 (11) Å), and the C_α–C_β bonds (1.60–1.63 Å) are quite long compared to typical C–C bonds. Note that the WC_3 ring is not planar; the dihedral angle between the C(11)–W–C(13) and C(11)–C(12)–C(13) planes is 25°. The ring is likely to be distorted easily in general and in this case perhaps in part because of steric interaction between the β-Me_3Si group and the bulky imido ligand. This WC_3 ring should be compared to the planar TiC_3 rings in bis(cyclopentadienyl) titanocyclobutane complexes studied in the past few years by Grubbs,[9] the only other examples of d^0 metallacyclobutane complexes. The fact that no metathesis products of vinyltrimethylsilane are observed requires that the $\overline{\mathrm{WCH(Me_3Si)CH(Me_3Si)CH_2}}$ ring lose only vinyltrimethylsilane when it breaks up, never $Me_3SiCH{=}CHSiMe_3$.

W(CH-*t*-Bu)(NR)(OR_F)$_2$ reacts rapidly with 20 equiv of *cis*-3-hexene to give ~1 equiv of *trans*-5,5-dimethyl-3-hexene (by GLC) and (by ^1H and ^{13}C NMR) what appears to be W(CHEt)(NR)(OR_F)$_2$ mixed with some residual W(CH-*t*-Bu)(NR)(OR_F)$_2$. Two alkylidene H_α signals (triplets) are observed for W(CHEt)(NR)(OR_F)$_2$ at 9.21 and 9.29 ppm in a ratio that varies from experiment to experiment. We propose that the isomers result from the two possible orientations of the propylidene ligand (eq 4), one of which is not so especially favored as in the

(4)

neopentylidene complex, and that they do not interconvert rapidly on the chemical time scale. This proposal is consistent with NMR results in related W and Re systems.[10] W(CHEt)(NR)(OR_F)$_2$ slowly decomposes over a period of 72 h, according to ^1H NMR studies; we do not yet know how. So far we have not been able to isolate it.

W(CH-*t*-Bu)(NR)(OR_F)$_2$ reacts with excess *cis*-2-pentene to give the expected initial cleavage products and *cis*- and *trans*-2-butene and 3-hexene. If 3700 equiv of *cis*-2-pentene are employed the expected equilibrium mixture is formed in ≤5 min at 25 °C. Therefore, a lower limit for metathesis activity of *cis*-2-pentene is ~1000 turnovers per minute at 25 °C. If 50 equiv of *cis*-2-pentene is added to W(CH-*t*-Bu)(NR)(OR_F)$_2$ in pentane and all volatile material removed in vacuo 15 min later, W(CHEt)(NR)(OR_F)$_2$ (primarily one isomer) is observed as the only major species by ^1H NMR.

Preliminary observations suggest that styrene, 1-octene, and allyltrimethylsilane are metathesized qualitatively much more slowly than *cis*-2-pentene. Many organometallic compounds are observed in such systems. Only the parent unsubstituted tungstenacyclobutane complex can be identified with certainty.

W(CH-*t*-Bu)(NR)(OR_F)$_2$ will catalyze the metathesis of methyl oleate (*cis*-methyl-9-octadecenoate). Between 200 and 300 equiv of methyl oleate are metathesized in 2–3 h at 25 °C; thereafter the system is inactive. During this process no new alkylidene H_α signal is observed, only signals ascribable to W(O)(NR)(OR_F)$_2$.[11] If only 1 equiv of methyl oleate is added to W(CH-*t*-Bu)(NR)(OR_F)$_2$ then 16 h are required for the reactions that yield W(O)(NR)(OR_F)$_2$ to be complete. Since excess ethyl acetate reacts rapidly only with W(CHEt)(NR)(OR_F)$_2$ in a mixture of it and W(CH-*t*-Bu)(NR)(OR_F)$_2$, we assume that the catalyst lifetime is limited by the reaction shown in eq 5 (R′ ≠ *t*-Bu; R‴ can be one of several possibilities), one that has precedent in tantalum,[12] titanium,[13] and zirconium[14] chemistry. W(CH-*t*-Bu)(NR)(OR_F)$_2$ also will catalyze predominantly the cross metathesis of methyl oleate with a several-fold excess of *cis*-3-hexene and *cis*-5-decene. Again activity is limited, probably by the reaction shown in eq 5.

$$\mathrm{W{=}CHR' + R'''CO_2Me \rightarrow W{=}O + R'CH{=}CR'''(OMe)} \qquad (5)$$

We feel we are now in a position to address many questions about olefin metathesis that have not been answered to date. Among the features of this system that should prove valuable is the option of varying the steric and electronic characteristics of the alkoxide ligand and potentially also those of the imido ligand. It is also interesting to note that in this apparently "classical"

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(7) In the $\overline{\mathrm{WCH_2CH_2CH_2}}$ complex $\delta(C_\alpha)$ 100.7, $\delta(C_\beta)$ −5.1, $\delta(H_\alpha)$ 4.50, $\delta(H'_\alpha)$ 4.66, $\delta(H_\beta)$ −0.79, and $\delta(H'_\beta)$ −1.39. In the $\overline{\mathrm{WCH(Me_3Si)CH(Me_3Si)CH_2}}$ complex $\delta(C_\alpha)$ 110.8, $\delta(C'_\alpha)$ 105.7, and $\delta(C_\beta)$, 5.18.

(8) The compound crystallizes in the space group $P2_1/n$ with $Z = 4$. Other crystal data are $a = 18.049$ (4) Å, $b = 12.224$ (4) Å, $c = 18.877$ (5) Å $\beta = 114.86$ (2)°, $V = 3778.9$ Å^3, $M_r = 906.85$, ρ(calcd) = 1.594 g cm^{-3}, and $\mu = 30.3$ cm^{-1}. Data were collected at −20 °C on an Enraf-Nonius CAD4F-11 diffractometer equipped with a liquid nitrogen low temperature device and using Mo K_α radiation. Data reduction and refinement have been explained in detail elsewhere.[8b] An empirical absorption correction was applied. A total of 6623 reflections were collected in the range 3° ≤ 2θ ≤ 50° with the 4209 having $F_o < 4\sigma(F_o)$ being used in the structure refinement by full-matrix least-squares techniques (424 variables) using SHELX. Final $R_1 = 0.54$ and $R_2 = 0.058$. The structure was refined without complications except for the axial OR_F group, which was not well-behaved. No suitable model for this disorder was forthcoming. C(43) and C(44) were refined isotropically and hydrogen atoms were not included on C(44). All remaining non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in calculated positions (C–H = 0.95 Å) and constrained to ride on their respective carbon atoms. A final difference Fourier map showed no significant features. (b) Silverman, L. D.; Dewan, J. C.; Giandomenico, C. M.; Lippard, S. J. *Inorg. Chem.* **1980**, *19*, 3379.

(9) Straus, D. A.; Grubbs, R. H. *Organometallics* **1982**, *1*, 1658.

(10) (a) There is one precedent; Re(N-*t*-Bu)$_2$(CHBu)(CH_2-*t*-Bu)[10b] shows four different *tert*-butyl signals in its ^1H NMR spectrum, and the α protons in the neopentyl ligand are inequivalent. For similar reasons the neopentyl methylene protons in W(CH-*t*-Bu)(NR)(CH_2-*t*-Bu)$_2$ are diastereotopic (C. J.S., preliminary results). (b) Edwards, D. S.; Biondi, L. V.; Ziller, J. W.; Churchill, M. R.; Schrock, R. R. *Organometallics* **1983**, *2*, 1505.

(11) Addition of 1 equiv of acetone to W(CH-*t*-Bu)(NR)(OR_F)$_2$ gave 2,4,4-trimethyl-2-pentene virtually quantitatively (by GLC) and pentane-soluble, orange W(O)(NR)(OR_F)$_2$ (according to elemental analysis and ^1H NMR spectrum; see supplementary material). We have not yet identified the organic product of any other Wittig-like reaction involving W(CH-*t*-Bu)(NR)(OR_F)$_2$.

(12) Schrock, R. R. *J. Am. Chem. Soc.* **1976**, *98*, 5399.

(13) Pine, S. H.; Zahler, R.; Evans, D. A.; Grubbs, R. H. *J. Am. Chem. Soc.* **1980**, *102*, 3270.

(14) (a) Clift, S. M.; Schwartz, J. *J. Am. Chem. Soc.* **1984**, *106*, 8300. (b) Hartner, F. W., Jr.; Schwartz, J.; Clift, S. M. *J. Am. Chem. Soc.* **1983**, *105*, 640.

system, tungstenacyclobutane complexes are close in energy to alkylidene complexes, the actual species that predominates depending upon the extent of substitution of the tungstencyclobutane ring.

Acknowledgment. R.R.S. thanks the National Science Foundation for support through Grant CHE 84-02892. C.J.S. thanks the Science and Engineering Research Council (U.K.) for a NATO postdoctoral fellowship. We also thank the Biomedical Research Support Shared Instumentation Grant Program, Division of Research Resources, for funds to purchase the X-ray diffraction equipment (NIH Grant S10RR02243-01).

Registry No. W(CBu-*t*)(NHR)(dimethoxyethane)Cl_2, 101347-96-0; W(CHBu-*t*)(NR)(dimethoxyethane)Cl_2, 101375-06-8; W(CHBu-*t*)(NR)$(OR_F)_2$, 101249-40-5; $CH_2CH_2CH_2W(NR)(OR_F)_2$, 101249-41-6; $W[CH(SiMe_3)CH(SiMe_3)CH_2](NR)(OR_F)_2$, 101347-97-1; W(CBu-*t*)(dimethoxyethane)Cl_3, 83416-70-0; Me_3SiNH-2,6-C_6H_3Pr-i_2, 78923-65-6; $LiOCMe(CF_3)_2$, 98171-13-2; W(CHEt)(NR)$(OR_F)_2$, 101249-42-7; W(O)(NR)$(OR_F)_2$, 101315-93-9; ethylene, 74-85-1; *tert*-butylethylene, 558-37-2; vinyltrimethylsilane, 754-05-2; *cis*-3-hexene, 7642-09-3; *trans*-5,5-dimethyl-3-hexene, 690-93-7; *cis*-2-pentene, 627-20-3; *cis*-2-butene, 590-18-1; *trans*-2-butene, 624-64-6; 3-hexene, 592-47-2; methyl oleate, 112-62-9.

Supplementary Material Available: Preparative, analytical, and spectroscopic details for all isolable compounds, along with final atomic coordinates and structure factor tables (31 pages). Ordering information is given on any current masthead page.

6-Cyanotetracyclo[5.5.0.0^{2,4}.0^{3,5}]dodeca-6,8,10,12-tetraene: A Novel Heptalene Valence Isomer

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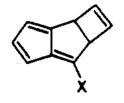
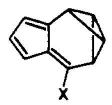
Received October 28, 1985

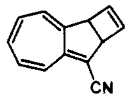
Recent research in this laboratory, beginning with the first synthesis of a pleiadiene isomer[1] in 1973, has been directed toward attainment of a synthetic representation of valence isomers of nonalternant hydrocarbons. Among the relevant species are the azulene isomers **1a**,[2] **1b**,[3] **2a**,[4] and **2b**[3] and the heptalene isomer **3**,[5,6] which contain nonalternant pentafulvene and heptafulvene chromophores,[7] respectively. Therefore, these studies are complementary to those of the well-documented benzene isomers.[8] In this paper we disclose the successful synthesis and some properties of the fourth example of our series, 6-cyanotetracyclo[5.5.0.0^{2,4}.0^{3,5}]dodeca-6,8,10,12-tetraene (cyanoheptalvalene)[9] (**4**), which is significant since this carbon skeleton has recently been postulated as an intermediate in thermal heptalene–heptalene transformations[10] but has never been isolated. The synthetic achievement completes a series of valence isomers of azulene and heptalene and opens the way for comparative studies on the detailed chemical and physical properties of these prototype molecules.



1a, X = OCH_3
1b, X = H



2a, X = OCH_3
2b, X = H



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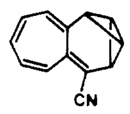

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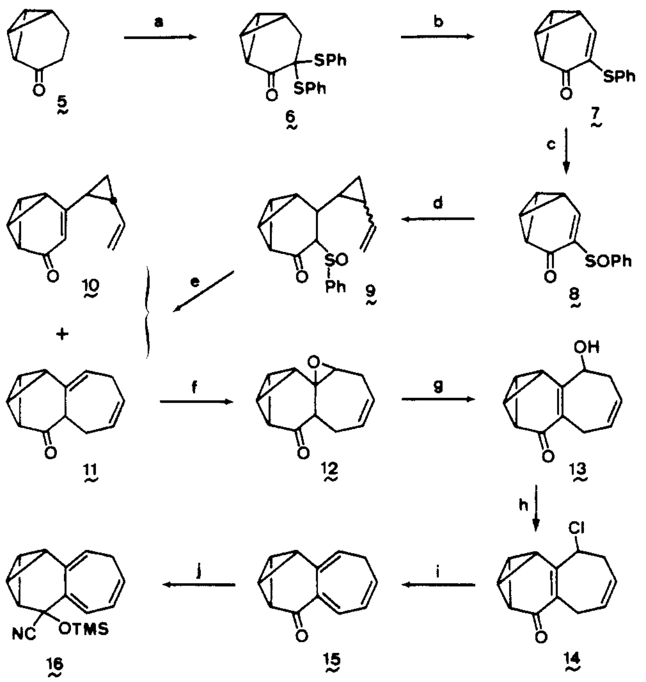
Scheme I[a]



[a] (a) 2.1 equiv of $LiN(i\text{-}C_3H_7)C_6H_{11}$ in THF–HMPA, then 2.2 equiv of $PhSSO_2Ph$ in THF, 0 °C, 1 h, 45%; (b) 1.1 equiv of mCPBA in CH_2Cl_2, −30 °C, 3 h, 62%; (c) 1.1 equiv of mCPBA in CH_2Cl_2, −78 °C, 1 h, 84%; (d) 3.0 equiv of 1-lithio-2-vinylcyclopropane in ether–THF, −78 °C, 1 h, 96%; (e) 0.3 equiv of $(MeO)_3P$ in benzene, 70 °C, 2.5 h, 77%; (f) 1.1 equiv of mCPBA in CH_2Cl_2, $-78 \rightarrow 0$ °C, 2 days, 65%; (g) 3.2 equiv of *t*-BuOK in ether, −78 °C, 1 h, 80%; (h) 2.8 equiv of *n*-Bu_3P and 5.9 equiv of CCl_4 in CH_2Cl_2, room temperature, 0.5 h, 88%; (i) 4.4 equiv of DBU in THF, room temperature, 3 h, 90%; (j) 2.7 equiv of Me_3SiCN, KCN/18-crown-6 complex in benzene, room temperature, 1 h, 99%.

Initially we planned to construct the bicyclobutane skeleton required for the synthesis of **4** through oxa-di-π-methane rearrangement[11] of an appropriately designed tricyclic β,γ-unsaturated ketone in an approach modeled after our previous synthesis of **2a**[4] and **2b**.[3] However, all attempts at these photoconversions gave unsatisfactory results. Consequently, in our successful approach outlined in Scheme I,[12] tricyclo[4.1.0.0^{2,7}]heptan-3-one (**5**),[13] which contains a bicyclobutane system, was used as a starting material.

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(1) (a) Murata, I.; Nakasuji, K. *Tetrahedron Lett.* **1973**, 47. (b) Pagni, R. M.; Watson, C. R., Jr. *Ibid.* **1973**, 59. (c) For a Dewar-type isomer, see: Meinwald, J.; Samuelson, G. E.; Ikeda, M. *J. Am. Chem. Soc.* **1970**, *92*, 7604.

(2) Sugihara, Y.; Sugimura, T.; Murata, I. *J. Am. Chem. Soc.* **1982**, *104*, 4295.

(3) Sugihara, Y.; Sugimura, T.; Murata, I. *J. Am. Chem. Soc.* **1984**, *106*, 7268.

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