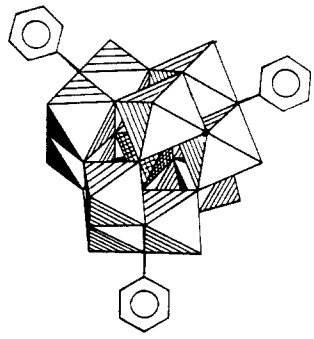

Figure 1. Proposed structure of $Ph_4SiW_{12}O_{40}$. The central SiO_4 unit is cross-hatched. One of the phenyl rings lies behind the plane of the paper.

$(aryl)_{8-n}XM_{12}O_{40}$. For example, addition of hydrated $(H_3O)_4SiW_{12}O_{40}$ to an acetonitrile solution of $PhN_2^+PF_6^-$ yields white, insoluble crystalline $(PhN_2)_4SiW_{12}O_{40}$ (**1**: IR $\nu_{N\equiv N}$ 2269 cm^{-1}; ^{13}C CPMAS NMR 114 (C_{ipso}), 134 ($C_{meta\ and\ ortho}$), 143, 147 (C_{para}) ppm. On heating at 75 °C under vacuum, **1** undergoes rapid, quantitative loss of nitrogen to provide tan $Ph_4SiW_{12}O_{40}$ (**2**).[14] This synthetic method has broad general applicability and has been extended to a wide variety of aryldiazonium salts, readily obtainable from the corresponding anilines, and other molecular metal oxide clusters such as $SiMo_{12}O_{40}^{4-}$, $PW_{12}O_{40}^{3-}$, and $PMo_{12}O_{40}^{3-}$.

Aryldiazonium Keggin ion salts bearing electron-withdrawing ring substituents have significantly higher thermal stability but can be decomposed photochemically. Thus, for example, $(p\text{-}CH_3OPhNHPhN_2)_3PW_{12}O_{40}$ (**3**) undergoes nitrogen loss at 138 °C. Irradiation into the broad absorption band centered at 450 nm results in elimination of nitrogen and formation of $(p\text{-}CH_3OPhNHph)_3PW_{12}O_{40}$ (**4**). The photochemical syntheses are not quantitative, however, because of self-absorption of incident radiation.

The Keggin ion $PMo_{12}O_{40}^{3-}$ undergoes monoalkylation by $(CH_3)_3O^+BF_4^-$ and the X-ray structure of $CH_3PMo_{12}O_{40}^{2-}$ disclosed that the methyl group is attached to an oxygen atom which bridges two edge-shared molybdenum octahedra.[15] This compound provides a structural paradigm for $(aryl)_{8-n}XM_{12}O_{40}$, and we consider that the aryl substituents are similarly attached to bridging oxygen atoms in the cluster. The ^{13}C CPMAS NMR spectrum of **2** demonstrates peaks at 166 (C_{ipso}), 133 (C_{meta} and C_{para}), and 122 (C_{ortho}) ppm relative to $(CH_3)_4Si$. These chemical shift values are typical of oxygen-substituted aryl compounds such as anisole for which the corresponding shifts are 159.9, 129.5, 120.7, and 114.1 ppm[16] and furnish evidence that the aryl groups are in fact covalently bonded to the $SiW_{12}O_{40}$ cage, cf. Figure 1. Consistent with this is the observation that the infrared spectra of **1** and **2** in the W–O stretching region are essentially the same which argues against a W=O bonding site.

Compound **2** and its analogues are poorly soluble in nonreactive organic solvents. Typical of inorganic esters, it reacts with aqueous acetonitrile to form phenol, in 97% yield, and $SiW_{12}O_{40}^{4-}$. A search for anionic, partially hydrolyzed clusters has not been successful. We consider that the hydrolysis involves nucleophilic attack by hydroxide ion with cleavage of W–O bonds for, when this reaction is carried out with $H_2{}^{18}O$, the phenol does not incorporate the ^{18}O label. Remarkably, the hydrolysis also produces a ca. 3% yield of hydroxybiphenyls which also do not contain ^{18}O. The ortho/meta/para isomer ratio is 1:0.24:0.31 which is strongly suggestive of a free radical coupling process.[17] When **2** and $(p\text{-tolyl})_4SiO_{12}O_{40}$ are cohydrolyzed, cross-coupling products, viz., methylhydroxybiphenyls, are not detected. Similarly, they are not formed when *p*-cresol is added to the hydrolysis mixture. This indicates that the aryl groups which combine to give hydroxybiphenyl are confined to a region close to the surface of the $SiW_{12}O_{40}$ cluster and are not freely diffusing in solution.

We are continuing to investigate the chemistry of these and related hydrocarbyl oxymetalates in an effort to better understand the reaction mechanisms of the pendant aryl groups.

Acknowledgment. We are grateful to W. T. Conway, 3M Analytical and Properties Research Laboratory, for the thermogravimetric analyses.

(14) **1**: Anal. Calcd C, 8.7; H, 0.6; N, 3.4. Found C, 8.7; H, 0.6, N, 3.4. **2**: Anal. Calcd C, 9.0; H, 0.6; N, 0.0; found: C, 8.9; H, 0.7; N <0.1. Temperatures required for thermal elimination of N_2 from **1** and further quantitation of N_2 loss was achieved by thermogravimetric analysis.
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Synthesis of a 1,3-Bridged β-Lactam: A Novel, Anti-Bredt β-Lactam

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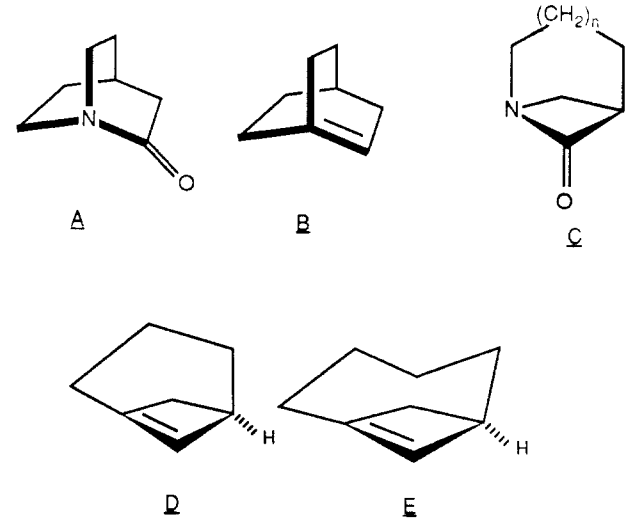
The β-lactam antibiotics have figured prominently in chemistry due to their desirable medicinal properties as chemotherapeutic agents, their structural novelty, and their attendant rich chemistry.[1] In the past few years, a plethora of structurally novel β-lactam antibiotics have been discovered from natural sources as well as the laboratories of academic and industrial scientists. In conjunction with an ongoing project in our laboratories, we desired the preparation of bicyclic amides containing the amide nitrogen in a bridgehead disposition. Hall[2] and others[3] have employed Bredt's rule, later modified by Wiseman,[4] Kobrich,[5] and Schleyer,[6] as a guide for predicting the stability and attendant isolability of N-bridgehead amides. However, due to the capacity of the nitrogen atom to assume a tetrahedral geometry, several "anti-Bredt amides" have been synthesized[7] such as 1-aza-bicyclo-[2.2.2]octan-2-one (A): the corresponding olefin B is predicted



to be an unstable olefin[8] with an olefin strain energy (OS)[6] of >40

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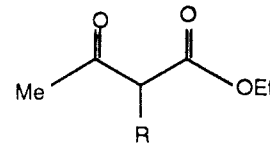
kcal/mol. Indeed, amide A displayed unusual properties, such as an anomalously high infrared absorption at ~1750 cm^{-1} for the carbonyl, high pK_a values, and susceptibility to hydrolysis and polymerization. Although several examples of these types of "acylamines" have been reported in the literature, there are no reports of a bicyclic β-lactam bridging the amide nitrogen to the opposite carbon in a 1,3-relationship (i.e., C). The bridgehead disposition of the β-lactam nitrogen atom in such a structure is expected to impart unusual and interesting properties to this system relative to the corresponding traditional monocyclic and bicyclic structures. For comparison purposes, the bicyclo[3.1.1]hept-1-(6)-ene (D) and bicyclo[4.1.1]oct-1(7)-ene (E) are predicted to be unstable bridgehead olefins with OS values of 39.1 and 37.8 kcal/mol, respectively. In this preliminary account is described a straightforward synthesis of a 1,3-bridged β-lactam[9] (3,7-dioxo-2-(*tert*-butylcarboxy)-8-methyl-1-azabicyclo[4.1.1]octane).

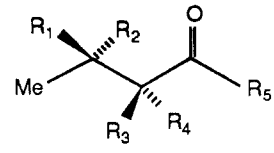
Ethyl acetoacetate is alkylated with 4-bromo-1-butene (NaOEt, EtOH, 50%) to afford the β-keto ester **2**. Sodium borohydride reduction[10] provided an inseparable mixture of the alcohols **3** and **4** (syn/anti, 1:2) which were directly subjected to a Mitsunobu inversion[11] (HCO_2H, Ph_3P, DEAD, THF) to furnish the syn-formate **5** in 20% overall yield from **2**. Hydrolysis to the acid **6** (1 N NaOH, THF) followed by reaction with 1-ethyl-3-(3-(dimethylamino)propyl)carbodiimide hydrochloride (1.3 equiv) and NH_2OCH_3·HCl (1.1 equiv) at pH 4.2 for 1 h at 25 °C furnished the *N*-methoxy amide[12] **7** (94%, mp 64–66.5 °C). Cyclization to the β-lactam **8** according to Miller[12] was accomplished with Ph_3P/DEAD (THF, 25 °C, 75%). Reductive cleavage of the N–O bond[13] was readily accomplished by dissolving metal reduction (Na^0, 2.4 equiv, NH_3(l), THF, −40 °C, 10 min) to furnish the desired β-lactam **9** (80%).

Oxidative cleavage of the olefin **9** to the acid **10** could be accomplished in one step by treatment with $NaIO_4$ in the presence of a catalytic amount of $RuCl_3$[14] (MeCN, CCl_4, H_2O) but was not practical for the preparation of multigram quantities of **10**. Therefore, a stepwise oxidation proved to be more convenient. Ozonolysis of **9** (MeOH, −78 °C, then Me_2S) followed by Jones oxidation[15] (8 N, acetone, 0 °C) furnished the acid **10** (80%). This material was homologated to the β-keto ester **11** utilizing the procedure of Brooks, Lu, and Masamune.[16] Reaction of **10** with carbonyldiimidazole (THF, 25 °C, 6 h) followed by condensation with $Mg(CO_2CH_2CO_2Bu^+)_2$ (0.55 equiv, 12 h, 25 °C) cleanly furnished **11** (20–40%). Diazotization under the standard conditions[17] (TsN_3, MeCN, 25 °C) gave the relatively labile diazo β-keto ester **12** (80%). Reaction of **12** with a catalytic amount of $Rh_2(OAc)_4$ for 1 h in refluxing benzene followed by filtration and evaporation of the solvent and chromatography on silica gel (PTLC, THF/hexane, 1:1) provided the bicyclic compound **13** as a solid in 50% yield.



1, R=H

2, R=$CH_2CH_2CH{=}CH_2$



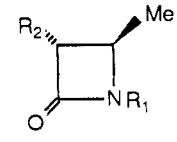
3, R_1=OH, R_2=H, R_3=$CH_2CH_2CH{=}CH_2$, R_4=H, R_5=OEt

4, R_1=OH, R_2=H, R_3=H, R_4=$CH_2CH_2CH{=}CH_2$, R_5=OEt

5, R_1=H, R_2=OCHO, R_3=H, R_4=$CH_2CH_2CH{=}CH_2$, R_5=OEt

6, R_2=OH, R_1=R_3=H, R_4=$CH_2CH_2CH{=}CH_2$, R_5=OH

7, R_2=OH, R_1=R_3=H, R_4=$CH_2CH_2CH{=}CH_2$, R_5=$NHOCH_3$



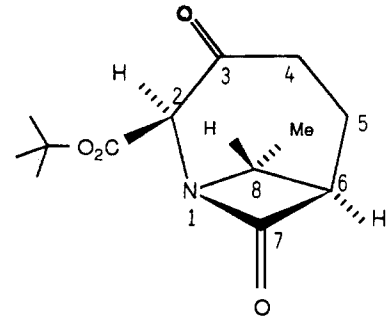
8, R_1=OCH_3, R_2=$CH_2CH_2CH{=}CH_2$

9, R_1=H, R_2=$CH_2CH_2CH{=}CH_2$

10, R_1=H, R_2=$CH_2CH_2CO_2H$

11, R_1=H, R_2=$CH_2CH_2COCH_2CO_2Bu^+$

12, R_1=H, R_2=$CH_2CH_2COC(N_2)CO_2Bu^+$

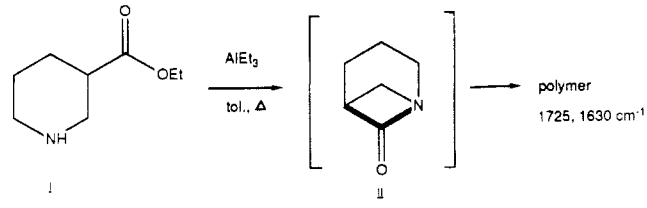


13

The structure of **13** was evident by examination of the spectroscopic properties.[18,19] The infrared exhibited carbonyl ab-

(8) Maier and Schleyer have proposed the following empirical rule regarding the relationship between the calculated olefinic strain (OS) and predicted experimental observability: (a) isolable bridgehead olefins OS ≤17 kcal/mol; (b) observable bridgehead olefins 17 kcal/mol ≤ OS ≤ 21 kcal/mol; (c) unstable bridgehead olefins OS ≥ 21 kcal/mol.

(9) Attempts at synthesizing a bicyclo[3.1.1] β-lactam ii from i afforded an unstable product (IR (neat) 1935, 1850, 1795, 1725 cm^{-1}) that rapidly polymerized (IR (neat) 1725, 1630 cm^{-1}).



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(18) ^1H NMR (270 MHz, $CDCl_3$) δ (Me_4Si) 1.50 (9 H, s), 1.53 (3 H, d, *J* = 6.0 Hz), 2.3–2.5 (2 H, m), 2.51–2.80 (2 H, m), 2.93 (1 H, dq, *J* ~ 1, *J* = 6.0 Hz), 3.13 (1 H, dd, *J* = 4.0, *J* = 1.3 Hz), 4.68 (1 H, s).

(19) The corresponding *p*-nitrobenzyl ester series was also synthesized and cyclized to the 1,3-bridged β-lactams as a 1:1 mixture of diastereomers epimeric at the β-keto ester methine position. The *tert*-butyl ester reported herein gave a single diastereomer that has been tentatively assigned the indicated stereochemistry.

sorptions at 1795, 1750, and 1730 cm^{-1} (NaCl, neat) for the β-lactam, ester, and ketone moieties, respectively. The mass spectrum (CH_4, CI) gave peaks at m/z 254 and 198 for ($M^+ + 1$) and ($M^+ - C_4H_9$), respectively. The ^1H NMR exhibited the characteristic one-proton singlet at δ 4.7 for the C-2 methine; a multiplet centered at δ 2.9 for the C-8 methine reflects a 0.6 ppm upfield shift of this resonance from the monocyclic precursors **9–12** and probably reflects both increased sp^3 character on nitrogen as well as shielding by the C-3 carbonyl.

The relatively surprising stability of **13** opens the possibility that numerous, stable "anti-Bredt" β-lactams can be synthesized and studied for novel chemical and possibly biological properties. Investigations along these lines are in progress in these laboratories and shall be reported on in due course.

Acknowledgment. We acknowledge the National Institutes of Health and the National Science Foundation for their generous support of our programs.

Supplementary Material Available: Physical data for all new compounds (2 pages). Ordering information is given on any current masthead page.

Oxazoline Route to Azomethine Ylides

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Recent progress[1,2] in generation of nonstabilized azomethine ylides allows synthesis of relatively simple five-membered nitrogen rings by 2 + 3 dipolar cycloaddition. Acyl-stabilized analogues **3** can be similarly used to prepare more complex cycloadducts,[3,4] but the most practical method to date for their generation via aziridine pyrolysis has limitations. Only the most reactive dipolarophiles can trap aziridine-derived dipoles stereospecifically,[3] and the behavior of alkyl-substituted aziridines (**2**, $R^1 = CH_3$, etc.) is surprisingly complex in certain systems.[5,6]

An alternative route to **3** from 4-oxazolines **4** has been contemplated[7] ever since the reverse reaction was demonstrated in a study of the valence bond tautomers **1–4**.[8] However, we can find only one example where pyrolysis of **4** with a dipolarophile is claimed to result in adduct formation.[7f] The other known 4-oxazolines **4**[7,8] are remarkably resistant to this process. They are also relatively inaccessible, and so far, all have been made directly or indirectly by acylaziridine pyrolysis, as in **2** → **3** → **4**.

We have developed an independent route to 4-oxazolines **4** by reduction of oxazolium salts **5** with $PhSiH_3/CsF$.[9] This reagent provides active hydride under aprotic, essentially neutral conditions, and overreduction of **4** is easily avoided. Most important, the silane/fluoride reagent selectively reduces oxazolium salts in the presence of dipolarophiles such as acrylate or acetylenedicarboxylate and permits in situ trapping of unstable oxazolines.

Isolation of the 4-oxazolines from oxazolium salt reductions has not been achieved due to their hydrolytic sensitivity but the 4,5-diphenyl derivative **4a** is stable in solution at room temperature (NMR, CD_3CN, CH_3CH, 5.02 ppm, quartet, J = 5.5 Hz). Addition of dimethyl acetylenedicarboxylate (DMAD) results in conversion into a 1:1 cycloadduct to which we assign the bicyclic structure **6** based on NMR evidence.[10] Similar adducts are obtained from other 4,5-disubstituted 4-oxazolines made in situ (Table I).

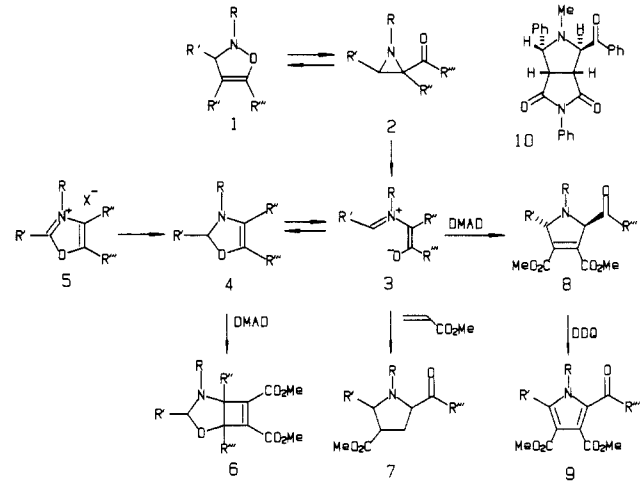
Table I. 2 + 2 Cycloadducts from 4-Substituted 4-Oxazolines (R = CH_3)

entry	R′	R″	R‴	product	yield
a	CH_3	Ph	Ph	**6a**	70%
b	CH_3	CH_3	CH_3	**6b**	82%
c	Ph	CH_3	Ph	**6c**	81%

Table II. 2 + 3 Cycloadducts from 4-Unsubstituted 4-Oxazolines (R = CH_3)

				yields	
				7	**9**[a]
d	Ph	H	Ph	55%[b]	95%
e	Ph	H	CH_3	87%	90%
f	Ph	H	OC_2H_5	63%[b]	93%
g	Ph	H	H	57%	
h	CH_3	H	OC_2H_5	61%	64%
i	CH_3	H	Ph	40%[b]	85%

[a] After DDQ oxidation of the crude product. [b] The other regioisomer (single stereoisomer) is formed in (d) 9%, (f) 10%, (i) 20%.



In contrast, several oxazolium salts having no substituent at C_4 are reduced by the in situ method ($PhSiH_3/CsF$/dipolarophile)

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(10) For example, **6b**: ^1H NMR ($CDCl_3$, ppm) 3.94 (1 H, q, J = 5.2 Hz), 3.78 (6 H, s), 1.43 (3 H, s), 1.33 (3 H, s), 1.31 (3 H, d, J = 5.2 Hz); ^{13}C NMR ($CDCl_3$, ppm) 167.2 (s), 162.6 (s), 142.9 (s), 140.8 (s), 89.0 (d), 85.7 (s), 74.5 (s), 52.0 (q), 51.9 (q), 31.6 (q), 17.9 (q), 16.1 (q), 15.9 (q).

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