have led to a number of misformulated polyoxoanions.[8a] Our own work, aimed at developing heteropolyanions for use as soluble metal oxide analogues in catalysis,[6] has made us keenly aware of the need for a rapid and accurate method for determining the elemental compositions and molecular weights of polyoxoanions.

Herein we report the use of fast atom bombardment mass spectroscopy (FABMS)[9,10] to obtain the first mass spectra of nonvolatile, high molecular weight salts of polyoxoanion complexes such as $SiW_{12}O_{40}^{4-}$, $SiW_9V_3O_{40}^{7-}$, and the previously unreported organometallic-polyoxoanion complex $CpTiSiW_9V_3O_{40}^{4-}$.[6b] The success we have experienced suggests that FABMS constitutes a significant new development in the ability to characterize polyoxoanions and similar inorganic polyanions or -cations.

FABMS were obtained on samples of $H_4SiW_{12}O_{40}\cdot 26H_2O$,[11a] $K_4H_3SiW_9V_3O_{40}\cdot 3H_2O$,[6b,11b] and $(Bu_4N)_4CpTiSiW_9V_3O_{40}$[6b] in thioglycerol in both the negative and positive ion modes,[12] as shown in Figure 1. The parent or molecular ion peaks show an isotopic distribution which agrees with the calculated envelope, as shown in Figure 2.

Cation exchange processes are clearly observed in several of our FABMS spectra. For example, K^+ replacement of H^+ in negative-ion FABMS spectra of $K_4H_3SiW_9V_3O_{40}$ produces the progression shown in Figure 1C corresponding to $K_4H_2SiW_9V_3O_{40}^-$, $K_5HSiW_9V_3O_{40}^-$, and $K_6SiW_9V_3O_{40}^-$. Similarly, Bu_4N^+ exchange with H^+ in positive ion FABMS of $(Bu_4N)_4CpTiSiW_9V_3O_{40}$ produces the series shown in Figure 1D corresponding to $(Bu_4N)_3H_2CpTiSiW_9V_3O_{40}^+$, $(Bu_4N)_4HCpTiSiW_9V_3O_{40}^+$, and $(Bu_4N)_5CpTiSiW_9V_3O_{40}^+$. The latter FABMS spectrum is particularly significant, since it illustrates the ability of this technique to characterize the previously unknown $CpTiSiW_9V_3O_{40}^{4-}$, which we have synthesized as a discrete, soluble analogue of $CpTi^{3+}$ supported on metal oxide surfaces.[6b]

The fragmentation patterns are dominated by multiple loss of O (m/z 16), consistent with other mass spectral data[10c,13] and by multiple loss of WO_3 (m/z 232). The former is seen in all FABMS spectra, both positive and negative ion, which we have examined, whereas the latter has not been observed with Bu_4N^+ salts (i.e., Figure 1D). The observation of positive-ion FABMS is remarkable due to the high acidity of some of these ions, and the recent report that FABMS ion currents can reflect solution concentrations.[9h] It appears from the data in Figure 1 and from FABMS spectral data on other related complexes that optimal spectra are obtained for positive ions from Bu_4N^+ salts.

In summary, fast atom bombardment mass spectroscopy has provided the first mass spectra of high molecular weight, nonvolatile salts of polyoxoanion complexes. The results obtained suggest that FABMS constitutes a significant development in the ability to characterize polyoxoanions and similar inorganic materials.

Acknowledgment. Support at Oregon by NSF Grant CHE-8313459 and a Dreyfus Teacher–Scholar (1982–1987) and Alfred P. Sloan Foundation Fellowship (1982–1984) to R.G.F. and at Illinois by NSF CHE-8020006 to K.S.S. are gratefully acknowledged. FAB mass spectra were obtained in the mass spectroscopy facility of the School of Chemical Sciences, University of Illinois, supported in part by NIH GM-27029; the ZAB mass spectrometer was purchased in part by grants from the Division of Research Resources, NIH (RR01575), and from the NSF (PCM-8121494).

Registry No. $H_4SiW_{12}O_{40}$, 12027-38-2; $K_4H_3SiW_9V_3O_{40}$, 91523-04-5.

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[7] For potential applications as electron microscopy labels, see: (a) Zonnevijlle, F.; Pope, M. T. *J. Am. Chem. Soc.* **1979**, *101*, 2731. (b) Mann, S.; Williams, R. J. P.; Sethuraman, P. R.; Pope, M. T. *J. Chem. Soc., Chem. Commun.* **1981**, 1083. (c) Ogan, M. D.; Keana, J. F. W. *Abstr. Pap.-Am. Chem. Soc.* **1983**, *185th*, ORGN 244.

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[12] All FABMS spectra were obtained on a VG Analytical ZAB-HF ultrahigh-resolution 8-kV mass spectrometer with 11250 data system. Milligram samples were dissolved in CH_3CN and a few microliters added to 100 μL of thioglycerol or other low-volatility matrix. Other matrices that have worked well with these polyoxoanions include triethylenetetraamine and 5:1 dithiothreitol/dithioerythritol. In some cases, sample preparation leads to the formation of so-called "polyoxoanion blues",[3a] which does not, however, appear to significantly affect the FABMS.

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Polar Transition State for the [1,5]-Sigmatropic Shift in Cyclopentadienes

Katrina S. Replogle and Barry K. Carpenter*

Department of Chemistry, Baker Laboratory
Cornell University, Ithaca, New York 14853

Received April 6, 1984

The model that we have developed[1] for analyzing substituent effects on thermal pericyclic reactions suggests that the transition state for the [1,5]-sigmatropic shift in cyclopentadienes should be π-isoconjugate[2] with bicyclo[3.1.0]hexatriene, which, being a lower homologue of azulene, should have a dipole moment with the negative end directed toward the five-membered ring (see structure at the top of Figure 2). In this communication we present data that support this idea. In addition we show that the same model provides a convenient way to rationalize and predict the regiochemistry of [1,5] migrations in substituted cyclopentadienes.

The parent[3] **1** and five-substituted[4] spiro[4.4]nona-1,3-dienes **2–6** were synthesized and subjected to thermal rearrangement. The products underwent facile [1,5]-hydrogen shifts, as antici-

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[4] Replogle, K. S. Ph.D. Dissertation, Cornell University, Ithaca, NY, 1984. The following spectroscopic information was obtained for the new compounds **2–6**. **2**: ^{1}H NMR ($CDCl_3$) δ 7.1–7.3 (m, 5 H), 5.9–6.5 (m, 4 H), 3.4 (t, $J = 6$ Hz, 1 H), 1.5–3.0 (m, 6 H); ^{13}C NMR ($CDCl_3$) δ 142.6, 141.1, 139.8, 129.3, 129.1, 125.7, 67.8, 51.5, 32.3, 31.6, 23.8; HRMS, m/z 196.1253 (calcd for $C_{15}H_{16}$ 196.1252). **3**: ^{1}H NMR δ 7.0–7.6 (m, 5 H), 6.5–6.7 (m, 2 H), 6.2 (dd, $J = 5$, 2 Hz, 1 H), 1.5–2.3 (m, 8 H); ^{13}C NMR δ 151.5, 147.4, 135.5, 128.2, 127.6, 126.5, 126.3, 126.2, 63.3, 32.7, 36.9. **4**: ^{1}H NMR δ 7.0–7.6 (m, 5 H), 6.3–3.8 (m, 3 H), 1.5–2.4 (m, 8 H); ^{13}C NMR δ 144.9, 137.1, 128.4, 128.3, 127.8, 127.0, 126.4, 125.9, 64.8, 33.2, 26.0. **5**: ^{1}H NMR δ 7.0 (dd, $J = 2$, 1.5 Hz, 1 H), 6.6 (dd, $J = 5$, 1.5 Hz, 1 H), 6.2 (dd, $J = 5$, 2 Hz, 1 H), 1.4–2.3 (m, 8 H); ^{13}C NMR δ 149.8 (d), 142.7 (d), 126.4 (d), 124.4 (s), 116.7 (s), 66.0 (s), 31.5 (t), 25.5 (t); IR 2200 cm^{-1}; HRMS, m/z 145.0883 (calcd for $C_{10}H_{11}N$ 145.0891). **6**: ^{1}H NMR δ 6.3 (dd, $J = 5$, 3 Hz, 1 H), 6.0 (dd, $J = 5$, 2 Hz, 1 H), 5.3 (m, 1 H), 3.6 (s, 3 H), 1.5–2.1 (m, 8 H); ^{13}C NMR δ 159.1 (s), 144.6 (d), 125.6 (d), 106.1 (d), 60.7 (s), 56.3 (q), 33.8 (t), 25.4 (t); HRMS, m/z 150.1028 (calcd for $C_{10}H_{14}O$ 150.1045).

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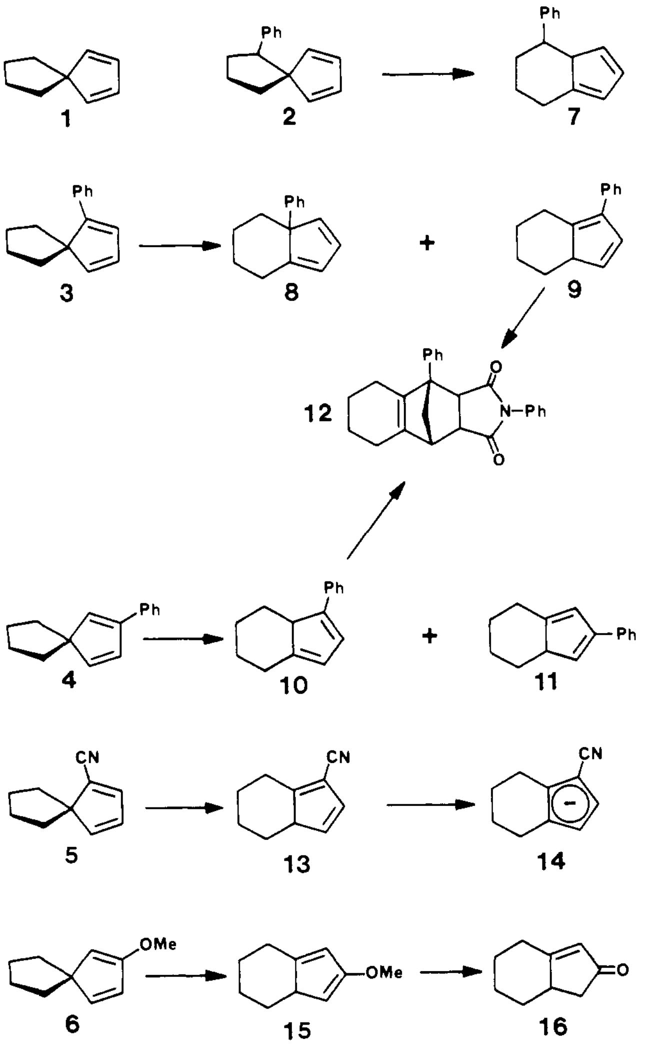

Figure 1. Six spiro[4.4]nonadienes used in this study (**1**–**6**) and the products of their thermal rearrangement. See text for further explanation.

pated, but, nevertheless, the regiochemistry of each rearrangement could be determined (Figure 1) by adduct **12** formation for **9** and **10**,[5] by ^{13}C NMR analysis of the symmetry of the anion **14** from **13**[5] and by hydrolysis of **15**[5] to the known[6] enone **16**. Compound **8** was formed reversibly from **3**, as might have been anticipated given the location of the phenyl group in **8**.

The kinetics of rearrangement of compounds **1**–**6** were studied on dilute solutions (typically 5×10^{-3} M) in anhydrous, degassed isooctane. In addition, studies were undertaken on rearrangements of **1** and **5** in isopropyl alcohol. The concentrations of reactants and products were followed by capillary gas chromatography. Control experiments showed that the compounds were stable to the chromatography conditions. Rate constants were determined at five temperatures (six for **1**) and the activation enthalpy and entropy calculated from the Eyring equation. The results are summarized in Table I.[7]

Table I. Kinetic Data for Compounds **1**–**6**

compd	temp range, °C	solvent	$\Delta H^{\ddagger}$, kcal/mol	$\Delta S^{\ddagger}$, cal/(mol K)	k_{rel} (150 °C)
1	141–196	isooctane	32.0 ± 0.2	-5.1 ± 0.5	[1]
1	136–175	isopropyl alcohol	30.9 ± 0.6	-6.7 ± 1.3	1.7
2	110–151	isooctane	28.3 ± 0.1	-6.8 ± 0.3	35
3 → **8**	146–185	isooctane	35.4 ± 1.1	-0.4 ± 2.6	0.2
3 → **9**	146–185	isooctane	32.6 ± 0.6	-4.3 ± 1.3	0.7
4 → **10**	146–185	isooctane	32.4 ± 0.5	-4.8 ± 1.0	0.7
4 → **11**	146–185	isooctane	34.4 ± 0.6	-0.3 ± 1.2	0.6
5	120–160	isooctane	29.6 ± 0.2	-5.7 ± 0.5	13
5	106–146	isopropyl alcohol	26.6 ± 0.2	-9.9 ± 0.4	55
6	160–200	isooctane	32.8 ± 0.4	-4.8 ± 0.5	0.4

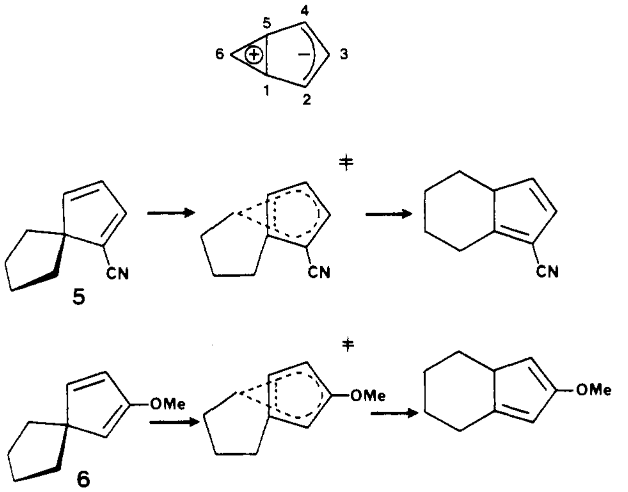

Figure 2. Explanation for the regiochemistry of rearrangement of **5** and **6**.

The idea of partial charge development[8] in the transition state for the rearrangement is supported by the reduction in both $\Delta H^{\ddagger}$ and $\Delta S^{\ddagger}$ that accompanies the change in solvent from isooctane to isopropyl alcohol. Further support comes from the effect of the cyano group which reduces $\Delta H^{\ddagger}$ by 2.4 kcal/mol in isooctane and 4.3 kcal/mol in isopropyl alcohol. This substituent effect cannot be due to the steric, conjugating, or radical-stabilizing properties of a cyano group since a phenyl at the same location has virtually no effect on $\Delta H^{\ddagger}$. The effect of the cyano group must be a consequence of its electron-withdrawing properties.

The predicted direction of the dipole moment in the transition state is consistent with the observation that a cyano group on the five-membered ring causes a reduction in $\Delta H^{\ddagger}$ whereas a methoxy group causes a small increase. The more modest effect of the donor substituent is to be expected when one considers the regiochemistry of the rearrangement (vide infra).

The fact that with a phenyl substituent on the cyclopentadiene ring one observes both possible regiochemistries for the [1,5] shift, whereas with methoxy and cyano substituents one finds only a single product, suggests that the regioselectivity observed in the latter two cases is electronic rather than steric in origin and, furthermore, that it has to do with the polarity of the substituents rather than their conjugating abilities. The charge distribution in bicyclo[3.1.0]hexatriene, as calculated by the Hückel molecular orbital model, is like that of an allyl anion fused to a cyclopropenium ion. Thus C2 and C4 bear the largest negative charges, C3 has a very small negative charge, and C1, C5, and C6 bear positive charges (Figure 2). In the rearrangement of **5** the two possible regiochemistries correspond to placing the acceptor substituent at C1 or C2; clearly one would expect C2 to be preferred, and this is what is observed. For **6** the choice is between C2 and C3. Since **6** bears a π-donor substituent, one would expect the site of smaller negative charge, i.e., C3, to be preferred. Again, this is what is observed. The modest effect of the methoxy substituent on the activation enthalpy for the reaction can now be

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(5) In fact, the reactions were performed on the indicated compounds plus their H-shifted isomers.

(6) Becker, K. B. *Helv. Chim. Acta* **1977**, *60*, 68–80.

(7) The activation parameters for conversion of **4** to **11** are probably somewhat less reliable than the calculated uncertainties because some difficulty was encountered in measuring the concentration of **11** with satisfactory reproducibility. Thus, the apparently higher activation entropy for this reaction might well be an artifact. If one assumed that the $\Delta S^{\ddagger}$ was really about -5 cal/(mol K) the activation enthalpy would be reduced to 32.3 kcal/mol.

(8) Independent evidence in support of a polar transition state in cyclopentadiene [1,5] shifts has come from studies of pressure effects on the reaction: Schulman, E. M.; Merbach, A. E.; Turin, M.; Wedinger, R.; le Noble, W. J. *J. Am. Chem. Soc.* **1983**, *105*, 3988–3991.

seen to be consistent with its being attached to a site of small charge.

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Synthesis of Amines through Nucleophilic Addition on Nitrogen

Robert A. Hagopian, Michael J. Therien, and
Joseph R. Murdoch*

Department of Chemistry, University of California
Los Angeles, California 90024

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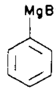
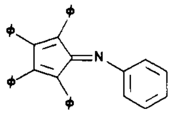
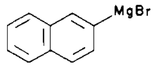
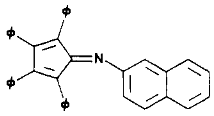
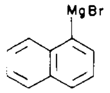
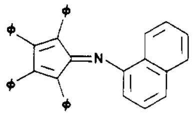
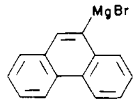
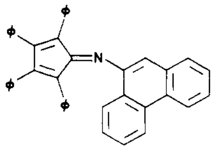
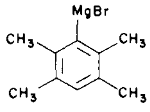
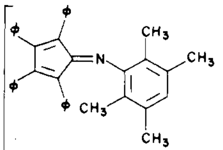
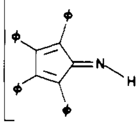
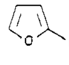
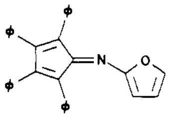
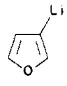
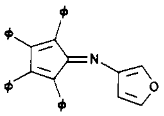
Nucleophilic displacements at carbon are well-known and form the basis of many useful synthetic reactions.[1] However, similar reactions at quaternary nitrogen proceed with difficulty[2] if at all.[3] There are a few reports of nucleophilic attack by alkyl Grignard reagents on trivalent nitrogen compounds including *O*-methylhydroxylamine[4] and chloramine[5] (0–90% yields). Addition of nucleophilic reagents to unsaturated nitrogen has also been used as a means of synthesizing amines from organometallic compounds. For example, Grignard reagents react with tosyl azide to form salts of tosyl triazenes which can be reduced to amines.[6] Reactions of aryl Grignards and aryllithiums with acetone oxime give the corresponding arylamines in yields of 12–70%.[7] [*O*-(*p*-Tolylsulfonyl)isonitroso]malononitrile reacts with malononitrile under basic conditions to yield salts of 1,1,3,3-tetracyano-2-azopropenide.[8] An unusual feature of this reaction is the addition of the nucleophile to the more electronegative atom of the C=X bond, but the usefulness of the method as a general synthetic route to amines is limited. Nucleophilic additions to the hetero atom of C=X bonds have been reported where the hetero atom is nitrogen,[6-9a,b,c] sulfur,[10a,b] or oxygen.[11] In all cases yields vary from 30% to 80%. Other methodologies for primary arylamine synthesis in useful yields involve the use of methoxyamine and methyllithium for the conversion of aromatic organometallics to primary amines. The yields are promising for aryllithiums (55–97%) but low for phenyl Grignard (37%).[12a,b] The reaction of aromatic organometallics with azidomethyl phenyl sulfide also yields the primary arylamine (50–98%). Present drawbacks of this elegant and valuable approach include the difficulty in preparing the starting material and the unpleasant aroma of phenyl thiol.[13a,b,c] Primary arylamines have also been obtained in 69–79% yield from the reaction of (trimethylsilyl)methyl azide (TMSMA) with aryl Grignards. Yields are poor for aryllithium reagents (35–41%).[14]

Table I

organometallic	imine	mp, °C[18]	yield, %[a]
1. 		225-226	95[e]
2. 		163-165	78[e,f]
3. 		224-225	70[b,e,f]
4. 		202-205	83[e,f]
5. 		174-175	65-68[b,e,f]
		170-173	20[c,e]
6. 		103-105	81[e,f,g]
7. 		178-180	78[e,f,g]
8a. benzanilide			90[d]
8b. tetraphenycyclopentadienone oxime			95[d]

[a] Yield of product recovered after liquid chromatography. [b] Identified by elemental analysis. [c] Reduction product of the reaction of (2,3,5,6-tetramethylphenyl)magnesium bromide with the oxime *O*-tosylate. [d] Identified by comparison with authentic sample. [e] Identified via high-resolution mass spectrometry. [f] Yield is not optimized. [g] Yield of recrystallized product. No chromatographic separation.

In the present paper, we report an efficient method for the amination of organometallic reagents. A novel feature of this approach is a "catalytic" cycle in which the amination reagent (hydroxylamine) is activated by oxime tosylate formation (Figure 1).

The starting material of the present work is easy to prepare (Figure 1): tetraphenylcyclopentadienone is synthetically and

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