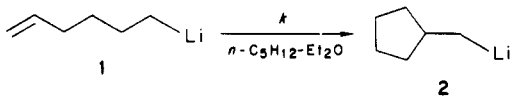
Table I. First-Order Rate Constants and Activation Parameters for the Cyclization of 5-Hexenyllithium (1) to (Cyclopentylmethyl)lithium (2)[a]

temp, °C	10^4k, s^{-1}	$\Delta H^{\ddagger}$, kcal/mol	$\Delta S^{\ddagger}$, eu
−11.1	1.75 ± 0.02	11.8 ± 0.5	−30 ± 2
−9.4	1.77 ± 0.02		
−0.5	4.18 ± 0.03		
0.8	5.71 ± 0.09		
9.4[b]	10.3 ± 0.1		
20.0[c]	20.6 ± 0.2		

[a] Errors are reported as standard deviations. [b] Average of two experiments. [c] Average of three experiments.

the kinetics of the cyclization of 5-hexenyllithium (CH_2=$CHCH_2CH_2CH_2CH_2Li$, **1**)[15] to (cyclopentylmethyl)lithium (c-$C_5H_9CH_2Li$, **2**)[15] by direct observation of the organolithiums using ^1H NMR spectroscopy. The results of these experiments are summarized in Table I.

Treatment of a 0.5 M solution of 6-iodo-1-hexene in *n*-pentane–diethyl ether (3:2 by volume)[14] with 2-equiv of freshly prepared *tert*-butyllithium[16] (*t*-BuLi) at −78 °C under argon affords an essentially quantitative yield of **1**. The isomerization of **1** to **2** was monitored at four tem-



1 **2**

peratures between −10 °C and +20 °C by NMR observation[17] of the CH_2Li region of the spectrum (Figure 1). The CH_2Li protons of **1** appear as a triplet at δ −1.11 (J = 8.67 Hz) while those of **2** appear as a doublet at δ −0.95 (J = 7.18 Hz). This latter assignment was confirmed by synthesis of **2** in quantitative yield upon treatment of a solution of cyclopentylmethyl iodide in *n*-C_5H_{12}–Et_2O (3:2 by volume) with 2 equiv of *t*-BuLi at −78 °C.[14]

The conversion of **1** to **2** is a clean first-order process when care is taken to exclude moisture and oxygen from the reaction mixture. In contrast to the behavior of other 5-hexenylalkalis in ethereal solvents,[13] there was no evidence for prototropic rearrangement of **1** to a 1-propylallyl species. The cyclization was followed through 3–4 half-lives by integration of the CH_2Li patterns of **1** and **2**. These data were fit by nonlinear least-squares analysis to the standard exponential form of the first-order rate expression (Figure 2) to give the rate constants reported in Table I. Activation parameters were determined by application of the Eyring equation: a linear plot of ln (k/T) vs. $1/T$ gave (Figure 3) $\Delta H^{\ddagger}$ = 11.8 kcal/mol and $\Delta S^{\ddagger}$ = −30 eu. The corresponding Arrhenius parameters were also determined: E_a = 12.6 ± 0.6 kcal/mol and ln A = 15.5 ± 1.2.

The data in Table I indicate that although the conversion of **1** to **2** is very much slower (by a factor of 10^8–10^{10}) than the cyclization of the 5-hexenyl radical,[2-4] it is, as suggested by the qualitative observations of Oliver and co-workers,[11] much faster than the analogous isomerization

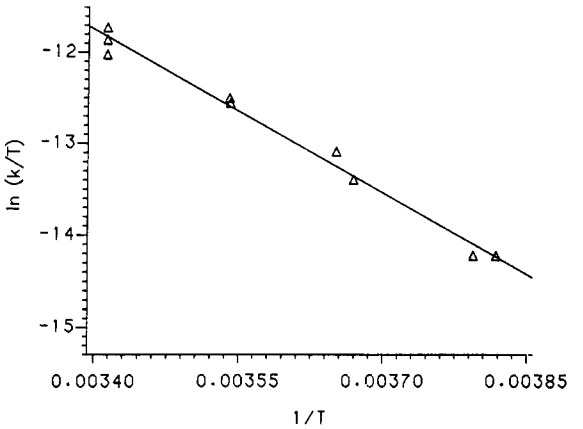


Figure 3. Plot of ln (k/T) vs. $1/T$ (K) for the conversion of **1** to **2**. Data from Table I.

of the 5-hexenyl Grignard.[10] Indeed, nonnegligible quantities of product containing the cyclopentylmethyl group may arise from cyclization of **1** to **2** since the half-life for this process at temperatures above 0 °C ($t_{1/2}$ ~ 23 min at 0 °C, 5.5 min at 23 °C) is short relative to the time scale of many experiments that seek to probe for radical intermediates. Be that as it may, the 5-hexenyl-to-cyclopentylmethyl cyclization remains a useful probe for radical intermediates even in reactions that produce **1**,[14] provided account is taken of the relative rates of isomerization of the radical and the organolithium.

The results noted above, and those recently reported by Garst and Hines for (1-methyl-5-hexenyl)sodium,[12] serve to emphasize the caveat that observation of products containing the cyclopentylmethyl group from reactions employing 5-hexenyl substrates is not sufficient evidence to establish the intermediacy of radicals particularly when organometallic species may be involved.

Acknowledgment. We are grateful to Dr. Mark Granville of the University of Connecticut for helpful advice on the kinetic analyses. This investigation was supported in part by the donors of the Petroleum Research Fund, administered by the American Chemical Society, and in part by a generous grant from the Humphrey Chemical Company, North Haven, CT.

William F. Bailey,* Jeffrey J. Patricia
Vincent C. DelGobbo
Department of Chemistry
University of Connecticut
Storrs, Connecticut 06268

Ronald M. Jarret, Paul J. Okarma
Department of Chemistry
Yale University
New Haven, Connecticut 06511
Received February 13, 1985

(14) Bailey, W. F.; Gagnier, R. P.; Patricia, J. J. *J. Org. Chem.* **1984**, *49*, 2098.

(15) Although organolithiums are often (as here) represented as monomeric, they are in fact aggregates whose degree of association may be affected by such factors as solvent, concentration, and temperature [Wakefield, B. J. "The Chemistry of Organolithium Compounds"; Pergamon Press: New York, 1974]. The degree of aggregation of **1** and **2** under the reaction conditions is unknown.

(16) Kamienski, C. W.; Esmay, D. L. *J. Org. Chem.* **1960**, *25*, 1807.

(17) ^1H NMR spectra were recorded at the Northeast Regional NMR Facility at Yale University on a Bruker WM-500 spectrometer operating in the FT-mode. The isomerization of **1** to **2** was monitored for 3 to 4 half-lives (70–90 points per experiment). Temperatures were measured as described by Van Geet [*Anal. Chem.* **1970**, *42*, 679] and are considered accurate to ±0.5°.

Stereochemistry of Crotylboronate Additions to α,β-Dialkoxy Aldehydes

Summary: The stereochemistry of the reactions of crotylboronates **1–4** with chiral α,β-dialkoxy aldehydes **5** and **6** is described.

Sir: A transformation with broad significance for control

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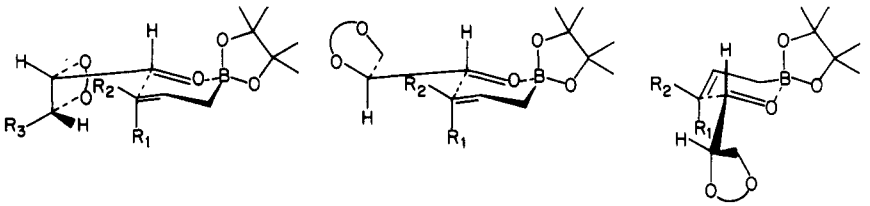
Table I[a]

entry	aldehyde	boronate	isomeric[b] purity, %	product ratios[c-e] 7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
1	**5**	1	98	91	5	1	3												
2	**5**	1	89	80	11	6	3												
3	**6**	1	97					96	2	1	1								
4	**6**	1	89					84	10	6	–								
5	**5**	2	95									90	4	2	4				
6	**5**	2	87									83	6	7	4				
7	**6**	2	93													93	3	3	1
8	**5**	3	98	6	52	42	–												
9	**5**	3	87	14	46	40	–												
10	**6**	3	98					2	52	48	–								
11	**6**	3	89					11	50	39	–								
12	**5**	4	>98									–	44	56	–				
13	**5**	4	93									6	43	51	–				
14	**6**	4	97													1	40	59	–
15	**6**	4	93													4	40	56	–

[a] All reactions were performed by addition of a slight excess of the aldehyde to the boronate in CH_2Cl_2 at −78 °C. The reactions were allowed to warm to room temperature and were stirred until complete (typically 24–48 h). Workup involved dilution with ether and extraction with water. [b] Isomeric purity of **1**–**4** was determined by capillary GC (OV-101 fused silica column). [c] Product ratios determined by GC analysis before chromatographic separation. Mixtures of **16** and **17** are easily separated by using a 0.25 in. × 9 ft 4.1% Carbowax/Chrom G column. All other analyses were performed by using a 9-ft capillary dimethylsilicone fused silica gel column (**16** and **17** coelute on this column but do resolve from **15**) or a 50-ft SE-54 capillary column. [d] The yield of products isolated by chromatography generally ranged from 75% to 85%. [e] See ref 7.



of acyclic stereochemistry is the reaction of chiral carbonyl compounds with organometallic reagents.[1] Crotylmetal compounds[2] (and related propionate aldol equivalents)[3] which generate two new stereochemical relationships in the C–C bond forming step are of considerable interest in this context. Of the numerous crotylmetal reagents which have been studied,[2] crotylboronates seem particularly attractive for many applications since stereochemically defined *Z* or *E* reagents are readily accessible[2h-j,4] and because the olefinic geometry is transmitted predictably to a syn or anti relationship in the product via cyclic transition states.[2a] Remarkably, however, relatively little information is available regarding the stereochemistry of crotylboronate reactions with chiral aldehydes.[5] In connection with several synthetic investigations we have had occasion to study the reactions of boronates **1**–**4**[6] with α,β-dialkoxy aldehydes **5** and **6**, the results of which are described in this communication (see Table I).[7,8]

(1) (a) Bartlett, P. A. *Tetrahedron* **1980**, *36*, 2. (b) McGarvey, G. J.; Kimura, M.; Oh, T.; Williams, J. M. *J. Carbohydr. Chem.* **1984**, *3*, 125. (c) Reetz, M. T. *Angew. Chem., Int. Ed. Engl.* **1984**, *23*, 556. (d) Eliel, E. L. In "Asymmetric Synthesis"; Morrison, J. D., Ed.; Academic Press: New York, 1983; Vol. 2, p 125.

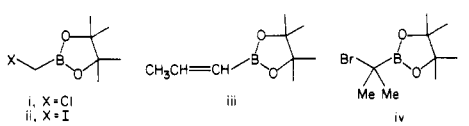
(2) (a) Hoffmann, R. W. *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 555. (b) Yamamoto, Y.; Maruyama, K. *Heterocycles* **1982**, *18*, 357. For other leading references, see: (c) Yamamoto, Y.; Yatogai, H.; Ishihara, Y.; Maeda, N.; Maruyama, K. *Tetrahedron* **1984**, *40*, 2239. (d) Keck, G. E.; Abbott, D. E.; Boden, E. P.; Enholm, E. J. *Tetrahedron Lett.* **1984**, *25*, 3927. (e) Hoffmann, R. W.; Landmann, B. *Ibid.* **1983**, *24*, 3209. (f) Hayashi, T.; Kabeta, K.; Hamachi, I.; Kumada, M. *Ibid.* **1983**, *24*, 2865. (g) Reetz, M. T.; Sauerwald, M. *J. Org. Chem.* **1984**, *49*, 2292. (h) Wuts, P. G. M.; Thompson, P. A.; Callen, G. R. *Ibid.* **1983**, *48*, 5400. (i) Hoffmann, R. W.; Zeiss, H. J. *Ibid.* **1981**, *46*, 1309. (j) Fujita, K.; Schlosser, M. *Helv. Chim. Acta* **1982**, *65*, 1258.

(3) Heathcock, C. H. In "Asymmetric Synthesis"; Morrison, J. D., Ed.; Academic Press: New York, 1984; Vol. 3, p 111.

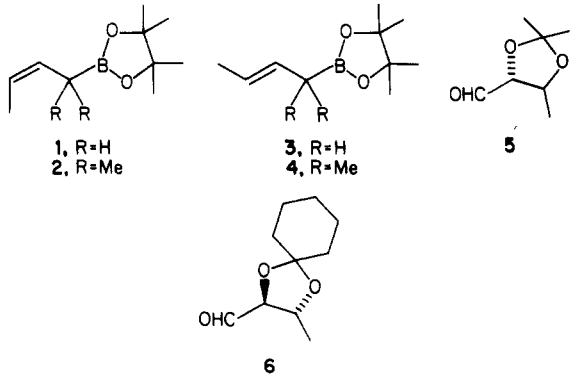
(4) Brown, H. C.; DeLue, N. R.; Yamamoto, Y.; Maruyama, K.; Kasahara, T.; Murahashi, S.; Sonoda, A. *J. Org. Chem.* **1977**, *42*, 4088.

(5) (a) Roush, W. R.; Peseckis, S. M.; Walts, A. E. *J. Org. Chem.* **1984**, *49*, 3429. (b) Roush, W. R.; Harris, D. J.; Lesur, B. M. *Tetrahedron Lett.* **1983**, *24*, 2227. (c) Wuts, P. G. M.; Bigelow, S. S. *J. Org. Chem.* **1983**, *48*, 3489. (d) Hoffmann, R. W.; Endesfelder, A.; Zeiss, H.-J. *Carbohydr. Res.* **1983**, *123*, 320. (e) Hoffmann, R. W.; Zeiss, H.-J.; Ladner, W.; Tabche, S. *Chem. Ber.* **1982**, *115*, 2357. (f) Hoffmann, R. W. *Chem. Scr.*, in press. We thank Professor Hoffmann for providing a copy of this manuscript prior to publication.

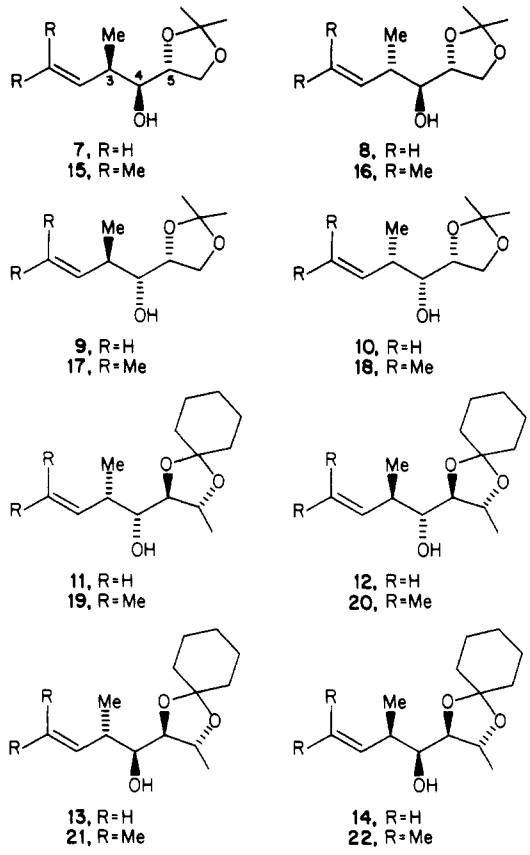
(6) (*Z*)-Crotylboronate **1** was prepared in 97% isomeric purity from (*Z*)-2-butene ((i) KO-*t*-Bu, *n*-BuLi, THF, −78 °C; (ii) $FB(OMe)_2$; (iii) H_3O^+; (iv) pinacol, CH_2Cl_2) by modification of Schlosser's general procedure (ref 2j). Reagent **1** was also prepared but with lower isomeric purity (89–95%) by addition of (*Z*)-propenyllithium (Whitesides, G. M.; Casey, C. P.; Krieger, J. K. *J. Am. Chem. Soc.* **1971**, *93*, 1379) or (*Z*)-$CH_3CH{=}CHMgBr$ (Martin, G. J.; Mêchin, B.; Martin, M. L. *C.R. Acad. Sci. Paris, Ser. C* **1968**, *267*, 986) to chloride i or iodide ii (see ref 2h). It should be noted that the propenyl Grignard route (75–80% yield of **1**) generally gave best results since the reaction of propenyllithium with i or ii afforded product mixtures in variable and nonreproducible yields containing at least 20–40% of propenylboronate iii. (*E*)-Crotylboronate **3** was best prepared (70% yield, 98% isomeric purity) by the reaction of (*E*)-propenyllithium with i (isomeric purity was <90% when propenyl Grignard was used). Finally, reagents **2** and **4** were prepared (77–85%) by addition of (*Z*)- or (*E*)-propenyllithium to bromide iv (see ref 2i, 4).



(7) The stereochemical assignments for **7**–**17** and **19**–**21** were established rigorously by correlation with compounds synthesized from epoxides of known configuration. The details of these correlations will be reported separately. The assignments for **18** and **22** are by analogy to **10** and **14**.



It is immediately striking that the reactions of (*Z*)-crotylboronates **1** and **2** with aldehydes **5** and **6** are highly stereoselective, with selectivity for the major 3,4-syn, 4,5-anti adducts **7**, **11**, **15**, and **19** approaching the limit defined



by the isomeric purity of the reagents. Although four products were detected in these experiments (entries 1–7), the minor anti–anti (**8**, **12**, **16**, **20**) and anti–syn (**9**, **13**, **17**, **21**) adducts can be attributed primarily to the contaminating olefin isomers present in **1** and **2**.[9] The fourth set of products, namely, the syn–syn isomers **10**, **14**, **18**, and **22**, serve to define the level of aldehyde facial selectivity in these cases: at least 20:1 in reactions with glyceraldehyde acetonide (**5**) and >90:1 with threonine-derived aldehyde **6**.

In contrast, the reactions involving (*E*)-crotylboronates **3** and **4** are much less stereoselective (entries 8–15). These experiments afforded approximate 1:1 mixtures of anti–anti (**8**, **12**, **16**, **20**) and anti–syn (**9**, **13**, **17**, **21**) adducts differing in the facial sense of reagent addition to the aldehyde. The 3,4-anti stereochemistry in both sets of products, as well as the 3,4-syn relationship in the major products generated from **1** and **2**, is in agreement with expectations based on chair-like transition states in which R of RCHO occupies an equatorial position.[2a] Consequently, the feature which clearly distinguishes the reactions of *E* boronates **3** and **4** from the *Z* isomers is that exceptional aldehyde facial discrimination occurs with **1** and **2** but not with **3** and **4**.

At the outset of these studies we had anticipated that both sets of boronate isomers would display excellent anti (Felkin–Ahn)[10] selectivity in these carbonyl addition reactions. First, reagents **1**–**4** can coordinate with only one ligand (the aldehydic carbonyl group) and so α- or β-chelate controlled pathways would not be possible. Second, aldehydes **5** and **6** are highly electrophilic and have a marked tendency to undergo anti nucleophilic addition even with reagents capable of chelation.[1b,c] Although the data for *Z* boronates **1**, **2**, and related systems[5a–d] are superficially consistent with Felkin–Ahn transition states (e.g., A_Z), the data for **3** and **4** (compare A_Z and A_E) suggests that the excellent results with **1** and **2** may be fortuitous and that factors other than conventional acyclic stereochemical considerations[10] must play an important role in determining the stereochemical course of these transformations.

Consider first the reactions of (*Z*)-crotylboronates **1** and **2**. Although one would ordinarily expect that Felkin–Ahn transition state A_Z should be preferred on the basis of favorable stereoelectronic interactions[10] between $\sigma^*_{C(2)-OR}$ and the developing C–C bond, examination of space-filling molecular models reveals that serious steric interactions occur between the axial vinylic methyl group (R_1) and C(3) of the aldehydic reactant. After consideration of other possible transition-state structures (generated by 120° rotations about the aldehydic C(1)–C(2) bond such that anti-periplanar relationships are maintained and by reversing the face of the carbonyl exposed to the reagent), it is apparent that B_Z contains fewer nonbonded interactions involving R_1, R_2, and the aldehydic C(2) or C(3) substituents than any of the other reasonable alternatives. It is probably this Cornforth-like transition state, and not A_Z, that accounts for formation of the major 3,4-syn, 4,5-anti adducts in entries 1–7 of Table I. The level of asymmetric induction is very high since the syn-selective transition states (e.g., C_Z) leading to the minor syn–syn isomers are disfavored by the interactions highlighted here.

The lower degree of aldehyde facial selectivity in the reactions of **3** and **4** (entries 8–15) can be rationalized by comparing transition states B_E and C_E with B_Z and C_Z. Transition state B_E is clearly more congested and less accessible than B_Z (1,3-interaction between R_2 and aldehydic C(2)-OR in B_E) whereas C_E is analogously less crowded and more accessible than C_Z. In the aggregate, no great preference exists for addition of **3**/**4** to either face of **5**/**6**.

In summary, we suggest that these reactions should be viewed as [3,3]-sigmatropic rearrangements of intermediate allylic boronate–aldehyde complexes (a bis-hetero-1,5-diene system) in which the stereochemical outcome is strongly influenced by steric effects. The Felkin–Ahn model[10] for 1,2-asymmetric induction may not be as important in these

(8) While our work was in progress Hoffmann described the reactions of **5** with the *E* and *Z* isomers of 2-*exo*,3-*exo*-[(2-butenylborylene)dioxy]*endo*-3-phenylbornane (see ref 5d). Our results (Table I) show that the chiral auxiliary employed in Hoffmann's study has very little effect on the overall stereoselectivity.

(9) These minor products are also potential products of minor reaction pathways involving **1** and **2** in which R of RCHO adopts an axial position in the transition state (see ref 2a).

(10) (a) Chêrest, M.; Felkin, H.; Prudent, N. *Tetrahedron Lett.* **1968**, 2199. (b) Ahn, N. T.; Eisenstein, O. *Nouv. J. Chim.* **1977**, *1*, 61. (c) Ahn, N. T. *Top. Curr. Chem.* **1980**, *88*, 145.

reactions as in other systems since the crotylboronates are very weak (soft) nucleophiles and because the trajectory of reagent approach to the carbonyl is constrained to a considerably smaller value than in biomolecular carbonyl addition reactions.[11] Studies probing the generality of these conclusions are in progress and will be reported in due course.

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Registry No. 1, 69611-01-4; 2, 76347-14-3; 3, 69611-02-5; 4, 96041-10-0; 5, 22323-80-4; 6, 87305-35-9; 7, 88406-01-3; 7 (acetate), 96041-14-4; 8, 88424-95-7; 8 (acetate), 96094-53-0; 9, 88424-94-6; 9 (acetate), 96094-54-1; 10, 96094-43-8; 11, 96041-11-1; 11 (acetate), 96041-15-5; 12, 96094-44-9; 12 (acetate), 96094-55-2; 13, 96094-45-0; 13 (acetate), 96094-56-3; 14, 96094-46-1; 15, 96041-12-2; 15 (acetate), 96041-16-6; 16, 96094-47-2; 16 (acetate), 96094-57-4; 17, 96094-48-3; 17 (acetate), 96094-58-5; 18, 96094-49-4; 19, 96041-13-3; 19 (acetate), 96041-17-7; 20, 96094-50-7; 20 (acetate), 96094-59-6; 21, 96094-51-8; 21 (acetate), 96094-60-9; 22, 96094-52-9.

Supplementary Material Available: Spectroscopic data and physical constants for 7-17 and 19-21 (5 pages). Ordering information is given on any current masthead page.

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(12) Holder of the Firmenich Career Development Chair in Natural Products Chemistry, 1981-84; Fellow of the Alfred P. Sloan Foundation, 1982-84.

William R. Roush,*[12] Michael A. Adam
David J. Harris
Department of Chemistry
Massachusetts Institute of Technology
Cambridge, Massachusetts 02139
Received January 25, 1985

Cascade Molecules: A New Approach to Micelles.[1a] A [27]-Arborol[1b]

Summary: The preliminary synthesis and spectral characterization of monocascade spheres (Arborols) which possess a three-dimensional microenvironment having the outer surface covered with polar functional groups is described.

Sir: In quest of novel micellar structures, we herein report a new series of micelles derived from an architectural model of trees,[2,3] specifically the Leeuwenberg model. This cascade[4] design generates a molecular structure, having an outer surface covered with polar functional groups. Since this model is based on a simple mathematical progression [$X_n = E^{n-1}$], it denotes a new class of cascade structures.[5]

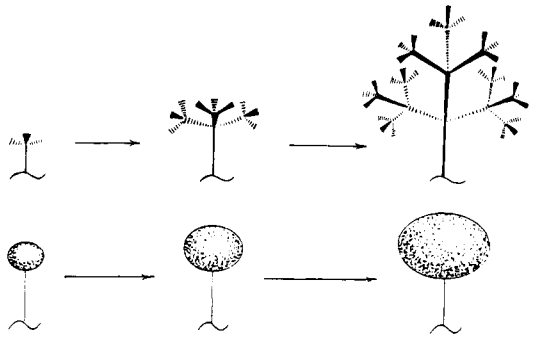
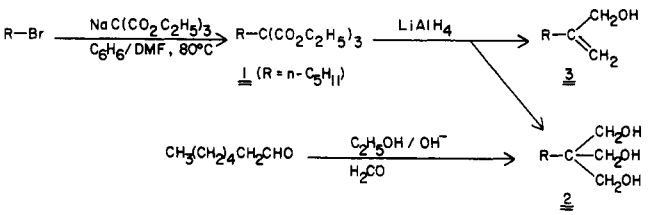


Figure 1.

Figure 1 shows the pictorial representation of the Leeuwenberg model as applied to micellar construction. The expansion of this one-directional cascade model to that of a two-directional cascade (sylvanols)[1c] affords entrance to potential "unimolecular" micelles, which possess an expandable parabolic cavity capable of surface inclusion.[14] Such a two-directional model is in essence an anticrown ether, since absorption is on the outer surface possessing the negative curvature. We herein communicate the preliminary synthesis and spectral characterization of the simplest examples of monocascade spheres.

Treatment of typical primary alkyl halides, for example 1-bromopentane, with $NaC(CO_2Et)_3$[15] afforded (83%) the tris-ester 1 [oil; bp 115-120 °C (3 mm); ^{13}C NMR δ 65.9



($C^{4°}$), 167.6 (CO)],[16] which can be reduced with either $LiAlH_4$ or $LiBH_4$ in ether to give triol 2 [white crystals; mp 65-65.5 °C; ^{13}C NMR δ 42.8 ($C^{4°}$), 66.7 (CCH_2O)][16] in low yield. The major unexpected product from this reduction is olefin 3 [^{13}C NMR δ 149.8 (C=CH_2), 109.4 (C=CH_2), 66.2 (CH_2O)], which arises by a facile Grob fragmentation.[17] In view of this deleterious result, a

(1) (a) Micelles. Part 1. (b) Since these cascades are based on arboreal design, they are logically called *arborols*. Sylvanols are thus the polyspherical cascade analogues. (c) Visiting Scholar from the Lanzhou Institute of Chemical Physics, Academia Sinica, China, 1983-1985.

(2) Hallé, F.; Oldeman, R. A. A. In "Essai sur l'architecture et la dynamique de croissance des arbes tropicaux", Paris; Masson et cie, 1970.

(3) Hallé, F.; Oldeman, R. A. A.; Tomlinson, P. B. "Tropical Trees and Forests: An Architectural Analysis", Springer-Verlag, Berlin, 1982.

(4) For cascade terminology, see: Buhleier, E.; Wehner, W.; Vögtle, F. *Synthesis* **1978**, 155.

(5) There are very few examples[6] of true cascade molecules which follow such a mathematical progression. Molecules, such as polypods,[7] hydrophilic lipids,[8] octopus ["hexapus"] molecules,[9] tentacle molecules,[10] hexahosts,[11] branched polyamines,[12] and "many-armed acyclic polyethers,[13] are related to, but do not fit, a cascade formulation.

(6) Denkewalter, R. G.; Kolc, J. F.; Lukasavage, W. J. U.S. Pat. 4410688, **1983**; *Chem. Abstr.* **1984**, *100*, 103907p. Aharoni, S. M.; Crosby, C. R., III; Walsh, E. K. *Macromolecules* **1982**, *15*, 1093.

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