as a colorless oil: $[\alpha]_D$ +28.6° (c 1.03, $CHCl_3$); IR ν 3500, 2950, 2870, 1740, 1460, 1450, 1380, 1350, 1220, 1180, 1120 cm^{-1}; ^1H NMR δ 0.83, 0.88, 0.94 (C-4 and C-10 Me), 1.26 (C-13 Me), 1.55 (C-8 Me), 2.50 (s, H-14), 3.70 (s, OMe); mass spectrum, m/e (relative intensity) 336 (M^+, 1), 335 (8), 317 (6), 304 (3), 285 (2), 277 (5), 259 (6), 243 (8), 233 (22), 226 (70), 210 (14), 205 (25), 197 (12), 189 (16), 177 (24), 163 (25), 147 (28), 135 (100), 123 (80), 117 (75), 109 (90), 95 (82), 69 (73), 43 (87); found for M^+ m/e 336.2710 ($C_{21}H_{36}O_3$ requires m/e 336.2664).

Compound 6. A solution of **7b** (118 mg, 0.35 mmol) in acetone (2.5 mL) was stirred overnight, in the cold (0 °C), with Jones' reagent (1 mL). Usual workup gave an oily residue (102 mg), which was purified by silica gel column chromatography (5 g, hexane and hexane–EtOAc), affording **6** (71 mg, 0.21 mmol, 60%) as a colorless oil: $[\alpha]_D$ +16.3° (c 2.5, $CHCl_3$); IR ν 3500, 2980–2860, 1730, 1660, 1610, 1450, 1380, 1340, 1210, 1090, 920, 740 cm^{-1}; ^1H NMR δ 0.88, 0.92, 1.10 (C-4 and C-10 Me), 1.30 (C-13 Me), 1.75 (C-8 Me), 2.53 (s, H-14), 3.73 (s, OMe); mass spectrum, m/e (relative intensity) 350 (M^+, 7), 331 (13), 316 (5), 300 (2), 284 (2), 272 (1), 258 (4), 234 (16), 232 (24), 227 (41), 225 (100), 205 (18), 176 (17), 161 (13), 152 (15), 134 (29), 133 (19), 123 (14), 116 (32), 104 (18), 43 (58); found for M^+ m/e 350.2455 ($C_{21}H_{34}O_4$ requires m/e 350.2457).

Compound 5a. To a solution of **6** (70 mg, 0.2 mmol) in MeOH (5 mL) was added $NaBH_4$ (10 mg, 0.26 mmol), the mixture was stirred at room temperature, and additional amounts of $NaBH_4$ (10 mg, 0.26 mmol each) were added every hour. The progress of the reaction was monitored by TLC. After 3 h, the reaction was complete. Brine (10 mL) was added, and the mixture was extracted with Et_2O (2 × 15 mL). The combined Et_2O extract was washed with brine (2 × 10 mL), dried (Na_2SO_4), and evaporated, affording an oily residue (65 mg, 0.18 mmol, 90%) of compound **5a**: $[\alpha]_D$ +39.3° (c 1.14, $CHCl_3$); IR ν 3400, 2980–2850, 1735, 1450, 1380, 1340, 1210, 1110, 1020, 920, 740 cm^{-1}; ^1H NMR δ 0.85, 0.88, 1.01 (C-4 and C-10 Me), 1.27 (C-13 Me), 1.66 (C-8 Me), 2.50 (s, H-14), 3.71 (s, OMe), 4.01 (m, $W_{1/2}$ = 25.6 Hz, H-7); mass spectrum, m/e (relative intensity) 352 (M^+, 30), 350 (32), 334 (15), 318 (17), 315 (20), 302 (19), 300 (21), 227 (13), 219 (50), 210 (28), 206 (13), 189 (12), 176 (9), 163 (8), 150 (9), 135 (15), 117 (18), 109 (100), 43 (60); found for M^+ m/e 352.2653 ($C_{21}H_{36}O_4$ requires m/e 352.2613).

Compound 5b. A solution of compound **5a** (80 mg, 0.23 mmol) and Ac_2O (0.2 mL) in pyridine (2 mL) was stirred overnight at room temperature. The mixture was then poured into ice and extracted with Et_2O (2 × 10 mL). The Et_2O solution was washed with 2 N HCl (2 × 10 mL) and brine (until neutral), dried (Na_2SO_4), and evaporated. Flash chromatography over silica gel (hexane and hexane–EtOAc) afforded **5b** (74 mg, 0.19 mmol, 83%): $[\alpha]_D$ +31.4° (c 0.6, $CHCl_3$); IR ν 3500, 2930, 1740, 1730, 1450, 1380, 1250, 1030 cm^{-1}; ^1H NMR δ 0.83, 0.88, 1.03 (C-4 and C-10 Me), 1.26 (C-13 Me), 1.51 (C-8 Me), 2.07 (CH_3COO-), 2.50 (s, H-14), 3.71 (s, OMe), 5.32 (t, J = 8 Hz, H-7); mass spectrum, m/e (relative intensity) 394 (M^+, 2), 376 (2), 375 (6), 374 (2), 365 (1), 352 (1), 335 (6), 334 (6), 315 (10), 301 (8), 262 (12), 220 (100), 210 (85), 189 (38), 173 (17), 159 (15), 147 (15), 133 (22), 119 (51), 117 (62), 109 (36), 105 (18), 43 (51); found for M^+ m/e 394.2788 ($C_{23}H_{38}O_5$ requires m/e 394.2719).

Compound 9. A stream of O_2/O_3 was bubbled through a cold (−30 °C) solution of **5b** (50 mg, 0.13 mmol) in a 1:1 mixture of CH_2Cl_2–MeOH (4 mL) during 2 h. The excess of O_3 was then displaced by a N_2 stream, and the mixture was treated with $(MeO)_3P$ (0.1 mL) at room temperature for 30 min. The solvent was evaporated, and the residue (55 mg) was purified by preparative TLC (silica gel, 70:30 hexane–EtOAc), yielding **9** (43 mg, 0.1 mmol, 77%): $[\alpha]_D$ −9.47° (c 0.8, $CHCl_3$); IR ν 3450, 3000–2900, 1760–1720, 1450, 1380, 1230, 1110, 1060, 920, 750 cm^{-1}; ^1H NMR δ 0.87, 0.95, 1.23 (C-4, C-10, and C-13 Me), 2.12 (s, 6 H, C-8 Me and CH_3COO), 2.50 (s, H-14), 2.57–2.85 (m, 2 H, H-11), 3.72 (s, OMe), 4.50 (dd, J = 5.8 and 8.3 Hz, H-7); mass spectrum, m/e (relative intensity) 382 ($M^+ - CO_2$, 5), 364 (6), 322 (20), 304 (3), 291 (5), 265 (7), 249 (18), 227 (21), 195 (19), 167 (25), 141 (28), 123 (85), 109 (100), 95 (55), 69 (83), 43 (65).

Compound 3b. A solution of **9** (70 mg, 0.16 mmol), K_2CO_3 (75 mg, 0.54 mmol), and $NaIO_4$ (150 mg, 0.70 mmol) in a 1:1 t-BuOH–H_2O mixture (10 mL) was stirred at room temperature. Additional amounts of K_2CO_3 and $NaIO_4$ was added after 24 and 48 h. After 72 h, the solvent was evaporated, and the residue was taken up in H_2O (5 mL). The mixture was saturated with NaCl, brought to pH 3 (10% HCl), and extracted with Et_2O (2 × 15 mL). The combined Et_2O extract was washed with brine until neutral and dried (Na_2SO_4). After being filtered, it was treated with excess ethereal solution of CH_2N_2 at 0 °C for 30 min. The solvent was evaporated, and the residue was redissolved in CH_2Cl_2 (5 mL) and treated with PCC/Al_2O_3[12] (160 mg). The mixture was stirred at room temperature overnight. It was then filtered through Celite, and the solvent was evaporated. The residue (53 mg) was purified by a silica gel column chromatography (3 g, hexane and hexane–EtOAc) to afford **3b** (42 mg, 0.12 mmol, 75% from **9**) as an oil: $[\alpha]_D$ −26.1 (c 0.8, $CHCl_3$); IR ν 2960, 1750, 1740, 1450, 1340, 1270, 1250, 1200, 960, 900, 780 cm^{-1}; ^1H NMR δ 0.88, 1.03 (C-4 and C-10 Me), 1.30 (C-13 Me), 2.37–2.57 (m, 2 H, H-6), 2.72 (s, H-14), 3.66 (s, OMe); mass spectrum, m/e (relative intensity) 294 ($M^+ - CO_2$, 9), 265 (6), 247 (4), 223 (15), 197 (21), 173 (51), 165 (18), 155 (23), 123 (35), 109 (100), 95 (39), 69 (54), 55 (26), 43 (19).

Acknowledgment. We thank Professor F. Bohlmann for carrying out the direct comparison of the synthetic and natural products, Dr. O. S. Giordano (Universidad Nacional de San Luis) for the high-resolution mass spectra determinations, Dr. J. C. Oberti (Universidad Nacional de Córdoba) for the low-resolution mass spectral data, and CONICET (Consejo Nacional de Investigaciones Científicas y Técnicas) and UNR (Universidad Nacional de Rosario) for financial support and fellowship (A.C.O.).

Registry No. **1a**, 1438-57-9; **1b**, 1438-58-0; **3a**, 81916-08-7; **3b**, 81916-38-3; **5a**, 102505-49-7; **5b**, 102505-51-1; **6**, 102505-48-6; **7a**, 102505-54-4; **7b**, 102505-47-5; **8** (isomer 1), 102505-50-0; **8** (isomer 2), 102572-03-2; **9**, 102505-52-2; **10** (isomer 1), 102505-53-3; **10** (isomer 2), 102572-04-3; **10** (methyl ester) (isomer 1), 102505-55-5; **10** (methyl ester) (isomer 2), 102572-05-4.

(12) Cheng, Y.-S.; Liu, W.-L.; Chen, S. *Synthesis* **1980**, 223.

Nafion-H† Catalyzed Reductive Cleavage of Acetals and Ketals to Ethers with Triethylsilane[1]

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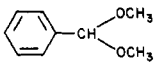
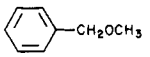
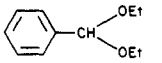
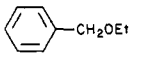
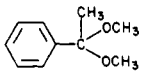
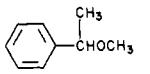
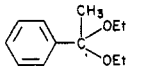
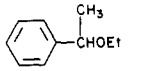
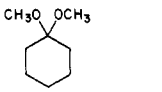
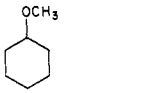
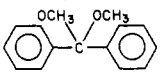
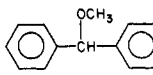
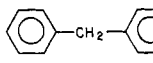
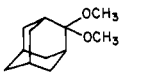
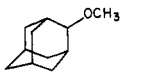
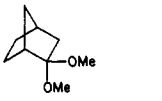
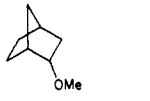
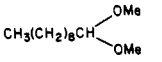
Acetals and ketals are protected carbonyl compounds which can be reduced to either ethers or hydrocarbons under a variety of conditions.[2] Noyori and co-workers[3] have shown that acetals can be conveniently reduced to ethers with triethyl- or trimethylsilane catalyzed by trimethylsilyl trifluoromethanesulfonate. More recently Mukaiyama and co-workers have used[4] trityl perchlorate as a catalyst to convert carbonyl compounds to symmetrical and unsymmetrical ethers with triethylsilane. Over the years we have shown that Nafion-H[5] a superacidic perfluororesinsulfonic acid is a convenient catalyst for a variety of acid-catalyzed synthetic transformations. The selectivity, high catalytic activity, and ease of regeneration frequently makes Nafion-H the acid catalyst of choice.

We wish to report now that acetals and ketals are reductively cleaved very efficiently to the corresponding ethers under Nafion-H catalysis with triethylsilane in refluxing dichloromethane solution. Both benzylic as well

† Nafion is a registered trademark of the Du Pont Company.

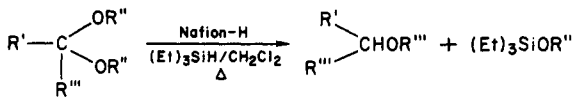
0022-3263/86/1951-2826$01.50/0

Table I. Nafion-H Catalyzed Hydrogenation of Acetals and Ketals

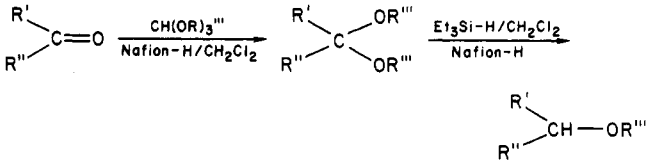
acetal/ketal	reacn time, h	product[a]	yield, %	mp °C or bp °C (torr) found	mp °C or bp °C (torr) reported[ref]
	2		96.0	66–63 (14)	62–70 (15)[10]
	1		97.0	70–72 (14)	78 (18)[10]
	2		91.2	173–175	60 (12)[8]
	2		96.7	67–68 (3)	88–90 (15)[7]
	4		91.7[b]		135[10]
	3		92.3	96–98 (0.25)	147–148 (17)[10]
	4[d]		81.5	115–117 (5)	158 (35)[10]
	2		94.5	58–60 (25)	47 (12)[9]
	2		99.0[b]	140–141	*c*
	2	$CH_2(CH_2)_8CH_2OCH_3$	95.3	189	90–92 (10)[7]

[a] Identity and purity (≥98%) established by GLC (25 ft × 0.25 mm i.d. glass capillary column, OV-101, temperature 70–190 °C), IR, and ^1H NMR and ^{13}C NMR spectrometry. [b] Yields were determined by GLC analysis. [c] Mixture of exo and endo isomers. [d] With 2.2 equiv of triethylsilane.

as aliphatic ketals and acetals are reduced in high yields (Table I).



Previously we have shown[6] that acetals and ketals can readily prepared by reacting carbonyl compound with trialkyl orthoformates in the presence of Nafion-H in dichloromethane solution. Utilizing this reaction we have also developed a one-pot procedure to convert carbonyl compounds directly to their alkyl ethers. Treatment of the carbonyl compound with trialkyl orthoformate (without isolation of acetals or ketals) followed by addition of triethylsilane and reflux gives the alkyl ethers in high yields (Table II).



The presently developed procedures offer advantages over previously reported methods. Nafion-H catalyst can be easily regenerated after the reaction by simple washing with acetone and deionized water followed by drying at 105 °C. Unlike trimethylsilyl trifluoromethanesulfonate, Nafion-H is stable in the presence of moisture. In fact the catalytic activity of Nafion-H significantly improves in the presence of small amount of water.

Experimental Section

General Procedure of the Nafion-H Catalyzed Reductive Cleavage of Acetals and Ketals with Trimethylsilane. A mixture of acetal or ketal (10 mmol), triethylsilane (1.28 g, 11 mmol), and Nafion-H (250 mg) in dichloromethane (20 mL) was refluxed until completion of the reaction, as monitored by GLC analysis (OV-101 column). The solid resinsulfonic acid catalyst was then filtered off and the solvent evaporated. The crude

(1) Catalysis by Solid Superacids. 20. For part 19, see: Olah, G. A.; Husain, A.; Singh, B. P. *Synthesis* **1983**, 892.

(2) (a) $LiAlH_4$–BF_3 (ether, refluxing temperature): Abdun-Nur, A. R.; Issidorides, C. H. *J. Org. Chem.* **1962**, *27*, 67. (b) $LiAlH_4$–$AlCl_3$ (ether, room temperature): Eliel, E. L.; Badding, V. G.; Rerick, M. N. *J. Am. Chem. Soc.* **1962**, *84*, 2371. (c) Diisobutylaluminum hydride (benzene, 70–80 °C): Zakharkin, L. I.; Khorlina, I. M. *Izy. Akad. Nauk SSSR, Ser. Khim.* **1959**, 2255; *Chem. Abstr.* **1960**, *54*, 10837h. (d) B_2H_6 (THF, up to 50 °C): Fleming, B.; Bolker, H. I. *Can. J. Chem.* **1974**, *52*, 888. (e) $NaB(CN)H_3$–HCl (methanol, 0 °C): Horne, D. A.; Jordan, A. *Tetrahedron Lett.* **1978**, 1357. (f) $(C_2H_5)_3SiH$ in CF_3COOH (50 °C): Loim, N. M.; Parnes, Z. N.; Vasil'eva, S. P.; Nursanov, D. N. *Zh. Org. Khim.* **1972**, *8*, 896; *Chem. Abstr.* **1972**, *77*, 125841w. Doyle, M. P.; DeBruyn, D. J.; Kooistra, D. A. *J. Am. Chem. Soc.* **1972**, *94*, 3659. For a review on the ionic hydrogenation with silanes, see: Kursanov, D. N.; Parnes, Z. N.; Loim, N. M. *Synthesis* **1974**, 633. (g) H_2 over Rh on alumina–HCl (room temperature to 80 °C): Howard, W. L.; Brown, J. H., Jr. *J. Org. Chem.* **1961**, *26*, 1026.

(3) Tsunoda, T.; Suzuki, M.; Noyori, R. *Tetrahedron Lett.* **1979**, *48*, 4679.

(4) Kato, J.; Iwasawa, N.; Mukaiyama, T. *Chem. Lett.* **1985**, *6*, 743.

(5) For comprehensive review on Nafion-H catalysis, see: Olah, G. A.; Iyer, P. S.; Prakash, G. K. S. *Synthesis*, in press.

(6) Olah, G. A.; Narang, S. C.; Meidar, D.; Salem, G. *Synthesis* **1981**, 282.

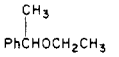
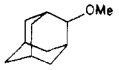
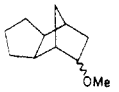
(7) McKillop, A.; Ford, M. E. *Tetrahedron*, **1974**, *30*, 2467.

(8) Yoshida, M.; Weiss, R. G. *Tetrahedron*, **1975**, *31*, 1801.

(9) Udding, A. C.; Strating, J.; Wyberg, H. *Tetrahedron Lett.* **1968**, *11*, 1345.

(10) *Dictionary of Organic Compounds*, 5th ed.; Bukingham, J., Ed.; Chapman & Hall: New York, 1982.

Table II. Nafion-H Catalyzed Reductive Conversion of Aldehydes and Ketones into Ethers

compound	reacn temp[e]	reacn time, h	product	yield, %[a]
PhCHO	rt	2[b]	$PhCH_2OCH_2CH_3$	94.6
	reflux	3[c]		
	rt	2[b]		91.0
	reflux	3[c]		
	rt	2[b]		93.0
	reflux	3[c]		
	rt	4[b]		88.0[d]
	reflux	6[c]		

[a] Isolated yield are shown. [b] With orthoformate. [c] With triethylsilane. [d] Bp 84 °C (2.2 mm), endo/exo mixture (93:7). [e] Room temperature = rt.

product was purified by distillation to provide corresponding ethers.

One-Pot Conversion of Benzaldehyde into Benzyl Ethyl Ether. A mixture of benzaldehyde (3.18 g, 30 mmol), triethyl orthoformate (4.44 g, 30 mmol), and Nafion-H (500 mg) in dichloromethane (20 mL) was stirred at room temperature for 2 h. Then to the reaction mixture was added triethylsilane (3.84 g, 33 mmol) followed by gentle reflux for 3 h. The solid resinsulfonic acid was filtered, and the solvent was evaporated. The crude product was distilled to obtain benzyl ethyl ether: yield, 3.8 g (95%); bp 67–68 °C (3 torr).

Regeneration of Nafion-H Catalyst. The used catalyst was washed several times with acetone and deionized water, followed by drying at 105 °C for 10 h. The catalytic activity of regenerated catalyst was as good as that of fresh catalyst.

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Registry No. Nafion-H, 63937-00-8; triethylsilane, 617-86-7; (dimethoxymethyl)benzene, 1125-88-8; (diethoxymethyl)benzene, 774-48-1; (1,1-dimethoxyethyl)benzene, 4316-35-2; (1,1-diethoxyethyl)benzene, 4316-37-4; 1,1-dimethoxycyclohexane, 933-40-4; 1,1′-(dimethoxymethylene)bisbenzene, 2235-01-0; 2,2-dimethoxytricyclo[3.3.1.^{3,7}]decane, 52776-45-1; 2,2-dimethoxybicyclo-[2.2.1]heptane, 10395-51-4; 1,1-dimethoxydecane, 7779-41-1; (methoxymethyl)benzene, 538-86-3; (ethoxymethyl)benzene, 539-30-0; (1-methoxyethyl)benzene, 4013-34-7; (1-ethoxyethyl)-benzene, 3299-05-6; methoxycyclohexane, 931-56-6; 1,1′-(methoxymethylene)bisbenzene, 1016-09-7; 2-methoxytricyclo-[3.3.1.1^{3,7}]decane, 19066-23-0; *endo*-2-methoxybicyclo[2.2.1]heptane, 10395-55-8; 1-methoxydecane, 7289-52-3; 1,1′-methylenebisbenzene, 101-81-5; *exo*-2-methoxybicyclo[2.2.1]heptane, 10395-53-6; benzaldehyde, 100-52-7; 1-phenylethanone, 98-86-2; tricyclo[3.3.1.1^{3,7}]decanone, 700-58-3; (3aα,4α,7α,7aα)octahydro-4,7-methano-2*H*-inden-5-one, 19138-60-4; (3aα,4α,5β,7α,7aα)-5-methoxyoctahydro-4,7-methano-2*H*-indene, 102518-93-4; (3aα,4α,5α,7α,7aα)-5-methoxyoctahydro-4,7,-methano-2*H*-indene, 102518-94-5.

Regio- and Diastereoselective Reactions of Dithio-Substituted Crotyllithium and Aldehydes

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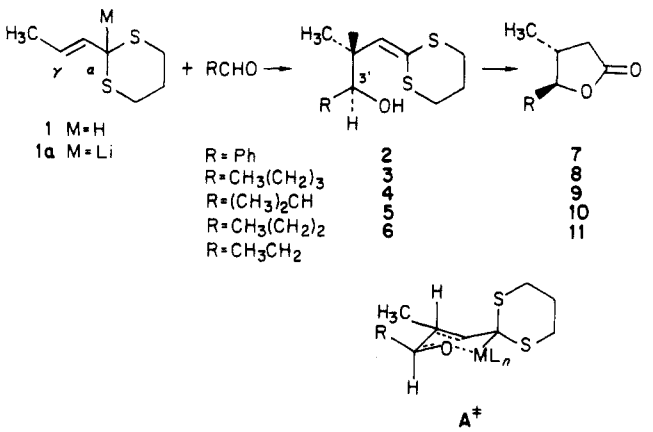
In syntheses of macrolide and polyether antibiotics, crotyl anion, especially with counter cations of boron, aluminum, tin, and titanium, has been shown to react with aldehydes in a highly diastereoselective fashion.[1] However, the corresponding crotyllithium compound, presumably existing as an equilibrating E/Z mixture, reacts less selectively with aldehydes. We thought the process of E/Z isomerization might be impeded by the dithioacetal substituent in the crotyl ion **1a** derived from (*E*)-2-(1-propen-1-yl)-1,3-dithiane,[2] and thus the **1a** ion would react stereoselectively with aldehydes. According to precedent,[3,4] the ambident crotyl ion **1a** should react regioselectively at the γ-site, and it would function as a useful synthon of a β-butyrate anion equivalent. The above speculation was realized as demonstrated in this note.

Scheme I



The desired vinylogous dithiane **1** was prepared by treating crotonaldehyde with an equivalent amount of 1,3-propanedithiol in the presence of magnesium perchlorate. The purity of the resultant dithiane, containing 98% *E* isomer, was superior to that prepared under conventional conditions (BF_3-Et_2O, HOAc).[2] Contrary to the counterpart of 2-propylidene-1,3-dithiane,[4] metalation of vinylogous dithiane **1** was readily effected by treatment with *n*-BuLi (THF, −30 °C, 1 h) in the absence of HMPA.[5] Thus, subsequent reaction with appropriate aldehydes (−78 °C, 20 min) was achieved in a highly regio- and stereoselective manner without the interference of HMPA.[5]

In all cases (Scheme I), crotyllithium **1a** reacted with the aldehyde exclusively at the γ-site as evidenced by ^{1}H NMR analysis. The reaction with benzaldehyde gave a diastereomeric mixture (77:23) of the γ-addition products **2** (95% total yield) as revealed by HPLC and ^{1}H NMR analyses. Since both the H-3′ resonances (C*H*OH) of diastereomers **2** (at δ 4.40 and 4.60) exhibited similar coupling constants (6 Hz), the structural elucidation was based on elaboration of **2** to γ-lactones **7**. Hydrolysis ($HgCl_2$, aqueous MeOH) of the minor isomer (**2**-*erythro*) gave **7**-*cis* lactone, which was characterized by the unusually high field of the methyl group (at δ 0.66) owing to the shielding effect of the adjacent phenyl group.[6] On the other hand, hydrolysis of the major isomer (**2**-*threo*) afforded the **7**-*trans* lactone, which displayed the methyl resonance at the normal position (δ 1.17). The γ-H resonance of **7**-*trans* lactone, presumably shielded by the β-CH_3 group, exhibited the resonance (d, J = 9 Hz) at relatively higher field (δ 4.95) than that of **7**-*cis* lactone (δ 5.62, d, J = 6 Hz).

The reaction of crotyl ion **1a** and pentanal gave exclusively threo product **3** in 90% yield; no erythro isomer

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