Notes

A Convenient Synthesis of *N*-Tosylimines

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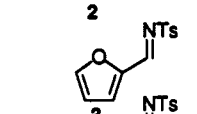
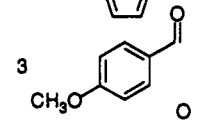
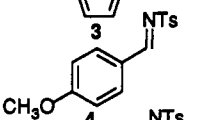
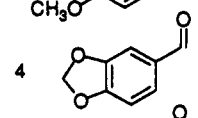
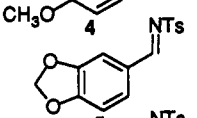
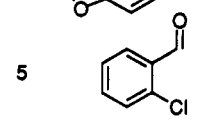
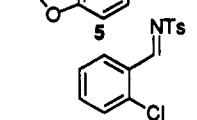
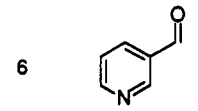
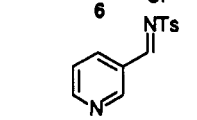
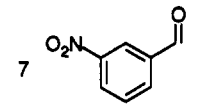
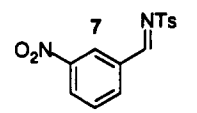
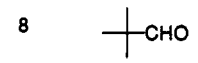
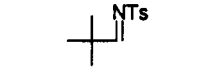
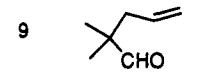
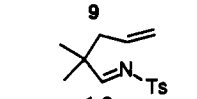
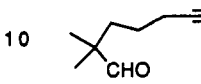
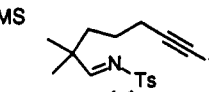
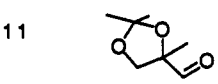
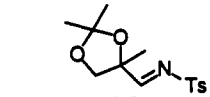
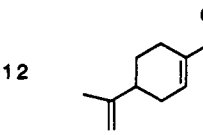
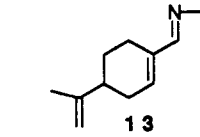
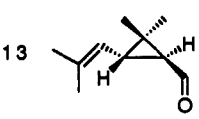
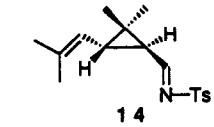
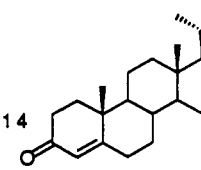
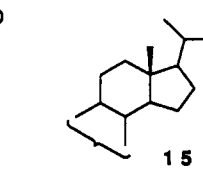
Sulfonimines have been increasing in importance because they are one of the few types of electron-deficient imines that are stable enough to be isolated but reactive enough to undergo addition reactions.[1] The lack of reactivity of simple imines toward our Pd-catalyzed [3 + 2] cycloaddition[2] induced us to examine this class of acceptors. Lichtenburger and Kretar first prepared such compounds by a Lewis acid catalyzed direct condensation,[3-5] a method that appears to be limited to aromatic aldehydes. Rearrangements of sulfinate esters of oximes have also served as a convenient approach. Kresze pioneered an imine-transfer reaction with aromatic aldehydes utilizing sulfinylsulfonamides[5a-6]—a procedure that was adopted more recently for aliphatic aldehydes.[6e] *N,N'*-Bis(arylsulfonylthio)- and selenodiimide[6c] have also been effective but suffer from lack of chemoselectivity since they undergo Diels–Alder and ene reactions. Diaryl- and dialkyltellurium analogues appear more useful but less convenient.[6b,d] We wish to report a simple protocol that makes these valuable building blocks readily available.

N,N'-Ditosyltelluordiimide (1), which previously was prepared by reacting tellurium tetrachloride and *N,N*-bis(trimethylsilyl)tosylamide,[7] appeared attractive to us since it fails to undergo Diels–Alder or ene reactions. Synthetically, we find a convenient protocol involves in situ formation of the reagent (or a synthetic equivalent thereof) by reaction of tellurium metal with chloramine T.[8] Polar and nonpolar solvents including acetonitrile, THF, chloroform, methylene chloride, 1,2-dichloroethane, and toluene were examined. While reaction proceeded

Table I. Synthesis of *N*-Tosylimines from Aldehydes

1	PhCHO	PhCH=NTs (2)	90-100%
2		 (3)	90-100%
3		 (4)	90-100%
4		 (5)	90-100%
5		 (6)	90-100%
6			93%
7		 (8)	90-100%
8		 (9)	90%
9		 (10)	97%
10		 (11)	98%
11		 (12)	90-100%
12		 (13)	84%
13		 (14)	100%
14		 (15)	100%

(1) For recent examples see: Melnick, M. D.; Freyer, A. J.; Weinreb, S. M. *Tetrahedron Lett.* **1989**, *29*, 3891. Sisko, J.; Weinreb, S. M. *Tetrahedron Lett.* **1989**, *29*, 3037. Ralbovsky, J. L.; Kinsella, M. A.; Sisko, J.; Weinreb, S. M. *Synth. Commun.* **1990**, *20*, 573. Alexander, M. D.; Anderson, R. E.; Sisko, J.; Weinreb, S. M. *J. Org. Chem.* **1990**, *55*, 2563. Sisko, J.; Weinreb, S. M. *J. Org. Chem.* **1990**, *55*, 393. Saigo, K.; Shimada, S.; Hasegawa, H. *Chem. Lett.* **1990**, 905. Boger, D. L.; Corbett, W. L.; Curran, T. T.; Kasper, A. M. *J. Am. Chem. Soc.* **1991**, *113*, 1713. Reetz, M. T.; Jaeger, R.; Drewlies, R.; Hübel, M. *Angew. Chem., Int. Ed. Engl.* **1991**, *30*, 103.

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(3) Lichtenburger, J.; Fleury, S.; Barrett, B. *Bull. Soc. Chim. Fr.* **1955**, 669.

(4) Also see: (a) Kretov, A. E.; Abrazhanova, E. A. *Russ. Gen. Chem.* **1957**, *27*, 2051. (b) Jennings, W. B.; Lovely, C. S. *Tetrahedron Lett.* **1988**, *29*, 3725. (c) Vishwakarma, L. C.; Stringer, O. D.; Davis, F. A. *Org. Synth.* **1986**, *66*, 203.

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(8) For selenium analogue, see: Sharpless, K. B.; Hori, T. S.; Truesdale, L. K.; Dietrich, C. O. *J. Am. Chem. Soc.* **1976**, *98*, 269. Sharpless, K. B.; Singer, S. P. *J. Org. Chem.* **1976**, *41*, 2504.

0022-3263/91/1956-6468$02.50/0

Table II. Experimental Details

entry	aldehyde (mg, mmol)	time (h)	GC ret time[a] (min)	imine (% yield)	mp (°C)	ref
				2[b]		
1	106, 1.0	1	9.8	230, 93	107	5a
				3[b]		
2	96, 1.0	1	9.1	250, 100	101–2	5a
				4[b]		
3	150, 1.0	1	11.9	290, 100	128–9	5a
				5[b]		
4	150, 1.0	1	12.7	290, 96	114–6	
				6		
5	140, 1.0	2	11.7	293, 100	128–9	
				7		
6	3.34 g, 31.2	4	10.5	8.22 g, 93	70–1	
				8		
7	152, 1.0	5	15.2	310, 100	101–2	4b
				9		
8	430, 5.0	0.5	7.1	106 g, 90	102–3	
				10		
9	112, 1.0	1	7.7	258, 97	oil	
				11		
10	210, 1.1	1	10.9	360, 98	oil	
				12		
11	144, 1.0	1	8.2	320, q	oil	
				13		
12	150, 1.0	1	11.9	272, 90	oil	
				14		
13	152, 1.0	6		307, 100	oil	
				15		
14	327, 1.0	3		500, q	oil	
				18		
15	260, 1.0	1		310, 75	oil	

[a] Analytical gas chromatography performed on a Varian Model 3700 gas chromatograph using an Alltech 25 m × 0.25 mm i.d. SE-30 column with flame ionization detection with a temperature program of T_i = 70 °C, 2 min, T_f = 250 °C; rate = 40 °C/min. [b] Purified by flash chromatography with 4:1 ether/hexane.

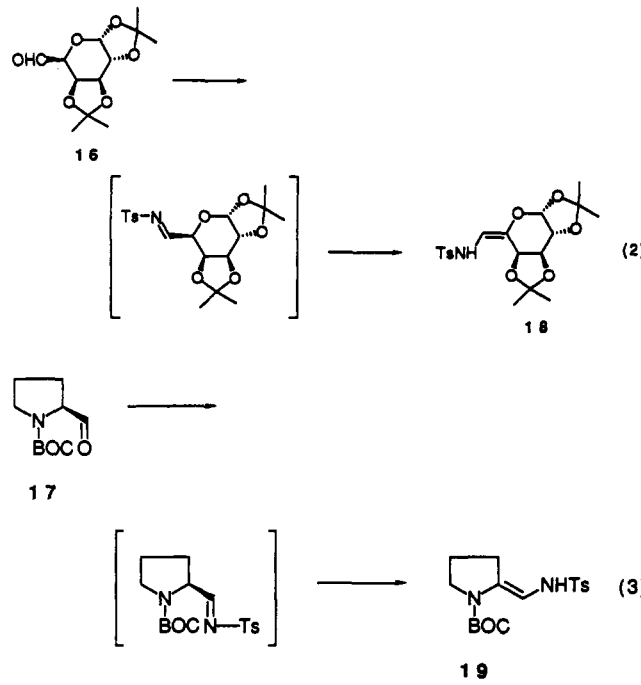
virtually in all solvents at their reflux temperature toluene appears best.

$$2RCHO + 2TsN(Cl)Na + Te \rightarrow 2RCH{=}NTs + 2NaCl + TeO_2 \quad (1)$$

The reaction proceeds stoichiometrically according to eq 1. The effectiveness of the reaction is somewhat surprising since it is heterogeneous throughout. Tellurium, chloramine T, and the diimide 1 are all insoluble in toluene. The byproducts, sodium chloride and tellurium dioxide, are also insoluble, which facilitates their removal by filtration. Unlike the case of selenium, the dioxide byproduct is not an oxidant itself and no foul-smelling colored residues are produced. Table I lists the range of aldehydes examined.

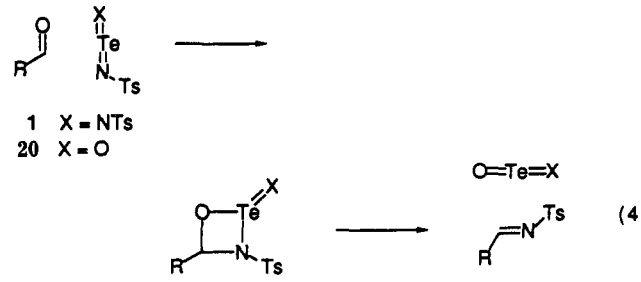
The examples show the excellent chemoselectivity of the process. The order of reactivity is aliphatic (<30 min) > electron-rich aromatic (1–2 h) > electron-poor aromatic (~5 h). Mass balance is normally quantitative, and the only impurity appears to be some tosylamide (0–5%) derived from an impurity in the chloramine T. While some enolizable aldehydes succeed (Table I, entries 12–14), enolization is clearly a problem. For example, the steroid case of entry 14 shows epimerization of the product accompanied its formation. The galactose aldehyde **16** and the prolinal **17** produce the enamines **18** and **19**, respectively, derived by tautomerization of the initial *N*-tosylimines upon workup.

The nature of the reagent has not been discerned. While the bisimide is a likely candidate, there is controversy in the corresponding selenium series.[9] At a minimum,



whatever the structure may be, it is at least a functional equivalent of the bisimide. For the following discussion, we employ **1** as a convenient formalism.

The mechanism of this reaction presumably involves a cycloaddition to a four-membered ring followed by cycloreversion to the observed product (eq 4). The fact that



1 mol of **1** converts 2 mol of aldehyde suggests that the initial byproduct **20** is approximately equivalent to **1** in its reactivity. The fact that the rate increases in less polar non-Lewis basic solvents and with electron-rich aldehydes suggests the initial interaction between the two reaction partners is that of a Lewis base–Lewis acid and therefore the initial cycloaddition is either highly asynchronous or nonconcerted.

Experimental Section

General Procedure. A suspension of 0.070 g (0.55 mmol) of tellurium metal and 0.24 g (1.05 mmol) of anhydrous chloramine-T in 2 mL of toluene was heated at reflux for 1 h, at which time the suspension became gray. The aldehyde (1.0 mmol) was added and heating continued for the stated time. The gray suspension became white. Methylene chloride was added and the reaction filtered through Celite. Removal of solvent in vacuo gave the *N*-tosylimine suitable for further use. Analytical samples were purified by recrystallization or flash chromatography. Table II lists the experimental details.

Spectral Data. 6: IR ($CHCl_3$) 1590, 1160, 1090 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.5 (s, 1 H), 8.17 (d, J = 7 Hz, 1 H), 7.92 (d, J = 10 Hz, 2 H), 7.15 (m, 2 H), 7.35 (m, 3 H), 2.45 (s, 3 H); ^{13}C (75 MHz, $CDCl_3$) δ 167.0, 135.8, 130.7, 130.4, 130.0 (2 C), 128.5 (2 C), 127.5, 21.5 (missing signals due to weak signal of quaternary aromatic carbons); MS calcd for $C_{14}H_{12}NO_2O$ (M^+ – Cl) 258.0590, found 259.0587. Anal. Calcd for $C_{14}H_{12}ClNO_2S$: C, 57.34; H, 4.12; N, 4.77. Found: C, 57.15; H, 4.27; N, 4.53.

(9) Derkach, N. Y.; Brashenkov, G. G. *Zh. Org. Khim.* **1986**, *22*, 1189. Derkach, N. Y.; Levchenko, E. S. *Russ. Chem. Rev.* **1989**, *58*, 507.

7: IR ($CHCl_3$) 1790, 1610, 1470, 1380, 1325, 1160, 1090 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 9.1 (d, J = 12 Hz, 2 H), 8.85 (s, 1 H), 8.3 (d, J = 8 Hz, 1 H), 7.90 (d, J = 8 Hz, 2 H), 7.5–7.15 (m, 3 H), 2.45 (s, 3 H); ^{13}C (75 MHz, $CDCl_3$) δ 167.9, 155.4, 153.3, 137.1, 130.1, 128.5, 124.3, 21.5; MS calcd for $C_{13}H_{12}N_2O_2S$ 260.0620, found 260.0617. Anal. Calcd for $C_{13}H_{12}N_2O_2S$-0.5H_2O: C, 57.97; H, 4.86; N, 10.40. Found: C, 57.71; H, 4.69; N, 10.38.

9: IR ($CHCl_3$) 1632, 1317, 1168, 1090, 1014 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.6 (s, 1 H), 7.7 (d, J = 10 Hz, 2 H), 7.3 (d, J = 10 Hz, 2 H), 2.4 (s, 3 H), 1.1 (s, 9 H); ^{13}C (100 MHz) δ 183.8, 144.5, 129.6 (2 C), 129.4 (2 C), 127.8, 37.7, 25.6, 21.5; MS calcd for $C_{12}H_{17}NO_2S$ 239.0980, found 239.0999.

10: IR ($CHCl_3$) 1635, 1325, 1160, 1090 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.4 (s, 1 H), 7.8 (d, J = 12 Hz, 2 H), 7.35 (d, J = 12 Hz, 2 H), 5.75–5.5 (m, 1 H), 5.02 (d, J = 14 Hz, 1 H), 5.0 (d, J = 19 Hz, 1 H), 2.45 (s, 3 H), 2.20 (d, J = 12 Hz, 2 H), 1.10 (s, 6 H); ^{13}C (75 MHz, $CDCl_3$) δ 184.0, 132.9, 129.9 (2 C), 128.2 (2 C), 119.0, 43.7, 23.4 (2 C), 2.15; MS calcd for $C_{13}H_{16}NO_2S$ ($M^+ - CH_3$) 250.0902, found 250.0888.

11: IR ($CHCl_3$) 1635, 1320, 1160, 840 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 1 H), 7.8 (d, J = 8 Hz, 2 H), 7.35 (d, J = 8 Hz, 2 H), 2.45 (s, 3 H), 2.15 (t, J = 7 Hz, 2 H), 1.61–1.58 (m, 2 H), 1.41–1.37 (m, 2 H), 1.10 (s, 6 H), 0.18 (s, 9 H); ^{13}C (100 MHz, $CDCl_3$) δ 183.7, 129.8 (2 C), 128.0 (2 C), 106.4, 40.8, 38.6, 23.6, 23.5 (2 C), 21.7, 20.2, 0.1; MS calcd for $C_{19}H_{29}NO_2SSi$ (M^+) 363.1690, found 363.1688.

12: IR ($CHCl_3$) 1635, 1600, 1350, 1165, 1062 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.5 (s, 1 H), 7.8 (d, J = 10 Hz, 2 H), 7.35 (d, J = 10 Hz, 2 H), 4.25 (d, J = 12 Hz, 1 H), 3.8 (d, J = 12 Hz, 1 H), 2.42 (s, 3 H), 1.41 (s, 6 H), 1.30 (s, 3 H); ^{13}C (75 MHz, $CDCl_3$) δ 178.4, 130.1, 129.7 (2 C), 129.6 (2 C), 128.4, 127.3, 72.2, 26.6, 26.3, 21.6; MS calcd for $C_{13}H_{16}NO_4S$ ($M^+ - CH_3$) 282.0800, found 282.0786.

13: IR ($CDCl_3$) 1630, 1570, 1320, 1160, 1090 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.5 (s, 1 H), 7.8 (d, J = 10 Hz, 2 H), 7.3 (d, J = 10 Hz, 2 H), 6.85 (s, 1 H), 4.8 (s, 1 H), 4.70 (s, 1 H), 2.52 (br, 1 H), 2.49 (s, 3 H), 2.31 (br, 1 H), 2.2 (m, 3 H), 1.9 (m, 1 H), 1.8 (s, 3 H), 1.45 (m, 1 H); ^{13}C (50 MHz, $CDCl_3$) δ 172.1, 153.1, 148.3, 144.4, 137.1, 135.8, 129.8 (2 C), 128.0 (2 C), 109.7, 40.1, 32.3, 26.0, 22.79, 21.4, 20.4; MS calcd for $C_{17}H_{21}NO_2S$ 303.1293, found 303.1286. Anal. Calcd for $C_{17}H_{21}NO_2S$-0.45H_2O: C, 65.54; H, 7.09; N, 4.50. Found: C, 65.22; H, 6.70; N, 4.25.

14: IR ($CDCl_3$) 1600, 1330, 1160, 1085 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, J = 7 Hz, 1 H), 7.80 (d, J = 8 Hz, 2 H), 7.30 (d, J = 8 Hz, 2 H), 4.90 (d, J = 6 Hz, 1 H), 2.43 (s, 3 H), 1.70–1.50 (m, 7 H), 1.30 (s, 3 H), 1.29–1.18 (m, 1 H), 1.17 (s, 3 H); ^{13}C (75 MHz, $CDCl_3$) δ 179.4, 143.4, 136.9, 129.7 (2 C), 128.1 (2 C), 127.8 (2 C), 119.8, 40.7, 38.0, 33.0, 25.6, 22.6, 22.1, 21.6, 18.5; MS calcd for $C_{17}H_{23}NO_2S$ 305.1449, found 305.1453.

15: 2:1 dr; IR ($CDCl_3$) 1635, 1325, 1160, 1090 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.41 (d, J = 8 Hz, 0.33 H), 8.39 (d, J = 8 Hz, 0.67 H), 7.9–7.8 (m, 2 H), 7.7 (d, J = 8 Hz, 2 H), 7.35–7.2 (m, 4 H), 7.17–7.13 (m, 1 H), 5.97–5.90 (m, 1 H), 5.73 (s, 1 H), 2.60–2.20 (m, 8 H), 2.10–0.80 (m, 16 H), 0.71 (s, 1.5 H), 0.61 (s, 1.5 H), 0.49 (s, 1.5 H); ^{13}C (75 MHz, $CDCl_3$) δ 182.1, 130.0, 129.9, 129.8, 128.3, 128.2, 126.9, 124.0, 123.9, 118.6, 56.3, 55.0, 54.9, 54.1, 53.8, 53.4, 42.4, 38.1, 35.7, 35.5, 35.3, 33.7, 32.6, 32.5, 31.7, 31.6, 26.7, 24.1, 23.8, 23.7, 21.3, 20.7, 20.5, 17.1, 17.0, 16.0, 12.7, 12.3; MS calcd for $C_{29}H_{39}NO_3S$ 481.2652, found 481.2646.

18: IR ($CDCl_3$) 3370, 1710, 1420, 1385, 1377, 1350, 1320, 1165, 1012 cm^{-1}; ^1H NMR (300 Mhz, $CDCl_3$) δ 7.71 (d, J = 10 Hz, 2 H), 7.29 (d, J = 10 Hz, 2 H), 6.7 (d, J = 11 Hz, 1 H), 6.05 (d, J = 11 Hz, 1 H0, 5.55 (d, J = 4 Hz, 1 H), 4.55 (dd, J = 12.2 Hz, 1 H), 4.47 (d, J = 7 Hz, 1 H), 4.28 (m, 1 H); ^{13}C (75 MHz, $CDCl_3$) δ 129.9, 126.9, 112.4, 110.3, 109.5, 97.5, 72.5, 70.6, 70.5, 26.0, 25.9, 25.0, 23.9, 21.2; MS calcd for $C_{19}H_{25}NO_7S$ 411.1356, found 411.1348.

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Registry No. 1, 65537-76-0; 2, 51608-60-7; 2 aldehyde, 100-52-7; 3, 135822-87-6; 3 aldehyde, 98-01-1; 4, 135822-88-7; 4 aldehyde, 123-11-5; 5, 135822-89-8; 5 aldehyde, 120-57-0; 6, 135822-90-1; 6 aldehyde, 89-98-5; 7, 135822-91-2; 7 aldehyde, 500-22-1; 8, 73845-02-0; 8 aldehyde, 99-61-6; 9, 135822-92-3; 9 aldehyde, 630-19-3; 10, 135822-93-4; 10 aldehyde, 5497-67-6; 11, 135822-94-5; 11 aldehyde, 135823-01-7; 12, 135822-95-6; 12 aldehyde, 68691-67-8; 13, 135822-96-7; 13 aldehyde, 2111-75-3; 14, 135822-97-8; 14 aldehyde, 20104-05-6; (20*R*)-15, 135823-00-6; (20*S*)-15, 135822-98-9; 15 aldehyde, 3986-89-8; 16, 4933-77-1; 17, 69610-41-9; 18, 135822-99-0; 19, 135823-02-8; Te, 13494-80-9; chloramine T, 127-65-1.

Synthesis of a New Macromolecular Ionophore with 2,5-Anhydro-D-glucitol Units via Cyclopolymerization of 1,2:5,6-Dianhydro-3,4-di-*O*-ethyl-D-mannitol

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Ionophores form a lipophilic complex with cations and transport the cations across the membrane by ion complex–decomplex formation. The naturally occurring ionophores include the polyether antibiotics such as monensin and nigericin consisting of a formally linear array of tetrahydrofuranyl and tetrahydropyranyl rings.

Several poly(cyclooxalkanediyl)s have been reported to act as so-called synthetic polyether ionophores. α,ω-Poly(cyclooxalkanediyl) was prepared through the ring expansion of the oxiranes deriving from the polymers of butadiene or cyclopentene, and the striking ability to bind with various size of cations, for example, Li^+, Ba^{2+}, and methylene blue was shown.[1] Poly(7-oxanorbornene) obtained from the metathesis polymerization was characterized by complexing with various cations containing methylene blue and rhodamine 6G as well.[2] Unlike the crown ethers, these acyclic ionophores are supposed to form helical conformers capable of varying pitch and cavity size to optimize multidentate coordination with a given cation. In this work, we report on the synthesis of a new macromolecular ionophore via stereoselective cyclopolymerization of diepoxide.[3]

Results and Discussion

Monomer **1**, which was synthesized from D-mannitol by the method reported by Kuszmann,[4] was polymerized with BF_3·OEt_2 in CH_2Cl_2. The reaction system was homogeneous, thereby eliminating any tendency to gelation. The product **2** obtained was a sticky semisolid soluble in $CHCl_3$,

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