Amines obtained by this method were essentially pure, as indicated by NMR spectroscopy, GLC, and TLC and were identified by comparison of their properties with literature values.

From these results, it is clear that enolizable as well as nonenolizable *N*-diisobutylaluminum imines react with a variety of organometallic reagents to afford good to excellent yields of primary amines after aqueous workup. The stoichiometry of the reaction generally required the use of 2 equiv of organometallic reagents against 1 equiv of imine due to the competitive attack of the organometallic reagent on the aluminum atom.

The method presented herein provides rapid access to primary amines from nitriles. The foundation is now established for a future study of an asymmetric variant of this chemistry as well as its applications to α-heterosubstituted aluminum imines. We are actively investigating these matters and will report on additional developments in due course.

Experimental Section

^{1}H NMR spectra were recorded at 90 or 300 MHz. All reactions were carried out under a blanket of argon. Column flash chromatography was performed on Merck silica gel (70–230 mesh). Butyllithium and *sec*-butyllithium were purchased from Aldrich as 15% hexane solutions. *tert*-Butyllithium was purchased from Ric-Roc. i-Bu_2AlH was purchased from Fluka. The purity of all title compounds was judged to be >95% by GC, GC-mass spectra, and ^{1}H NMR determinations.

Procedures for the preparation of selected primary amines are provided below. Known compounds gave spectral data according to those reported in literature and to the assigned structure.

Preparation of *N*-Diisobutylaluminum Imines. *N*-(Diisobutylaluminio)benzaldimine (2a). In a 50-mL, two-necked flask equipped with magnetic stirring bar and with nitrogen and syringe inlets was placed 5 mmol (0.516 g) of benzonitrile **2** in 10 mL of dry pentane. *i*-Bu_2AlH (5 mmol, 0.781 g) was added via syringe at −78 °C. The solution was maintained at the same temperature for 3 h, and the pentane removed under vacuum.[8] A sample of this material was subjected to spectral identification: IR ($CHCl_3$) 1635 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) 8.97 (s, 1 H), 7.76–7.50 (m, 5 H), 1.77 (m, 2 H), 1.5–0.8 (m, 16 H).

One-Pot Preparation of Primary Amines from Nitriles. 1-Phenyl-3-butenamine (4b). Method A. From the *N*-(diisobutylaluminio)benzaldimine solution prepared as described above, the pentane was completely removed under vacuum, 8 mL of anhydrous THF and 2 mL of an ethereal 2 M solution of allylmagnesium chloride[9] were added at −78 °C. This solution was allowed to reach room temperature (overnight) and stirred for a further 48 h. The reaction mixture was then cautiously hydrolyzed with 50 mL of water and extracted with ethyl acetate. After drying over $MgSO_4$, removal of the solvent gave 0.720 g (98%) of an oil essentially constituted by the amine as indicated by its NMR and GLC-MS.

Method B. To the reaction mixture of the amine, obtained as described in the method A, was added saturated NH_4Cl (10 mL), followed by concentrated ammonium hydroxide (10 mL) at 0 °C. After being stirred for 1 h, the mixture was extracted with diethyl ether (3 × 30 mL). The combined organic phases were washed with 1 M NaOH, dried over $MgSO_4$, and evaporated. The residue was dissolved in anhydrous diethyl ether (10 mL) and a saturated ethereal HCl solution was added to precipitate the amine hydrochloride. The product was filtered off and washed with anhydrous diethyl ether and acetone to give the amine hydrochloride as a white solid (mp 222 °C; lit.[10] mp 224–226 °C) (95%). The free amine was quantitatively obtained upon treatment with NaOH (1 N) and extraction with ether: IR (film) 3370, 3290, 3080, 1640, 1600, 1490 cm^{-1}; ^{1}H NMR ($CDCl_3$, 90 MHz) 7.3 (m, 5 H), 5.6 (m, 1 H), 5.1 (m, 2 H), 4.0 (dd, J_1 = 6 Hz, J_2 = 7.5 Hz, 1 H), 2.4 (m, 2 H), 1.9 (s, 2 H, NH_2); GC/MS 147, 146 (100) (M^+ − 1). Anal. Calcd for $C_{10}H_{13}N$:C, 81.59; H, 8.9. Found: C, 81.67; H, 8.92.

1-(2-Thienyl)pentanamine (4h): IR (film) 3360, 3280, 3100, 3060 cm^{-1}; ^{1}H NMR ($CDCl_3$) 7.0 (m, 1 H), 6.8 (m, 2 H), 4.1 (t, 1 H, J = 6 Hz), 2.0 (bs, 2 H, NH_2), 1.7–0.7 (m, 9 H); GC/MS 169 (M^+), 112 (M^+ − butyl) (100). Anal. Calcd for $C_9H_{15}NS$: C, 63.86, H, 8.93. Found: C, 63.97; H, 8.92.

1-(2-Thienyl)-3-butenamine (4i): IR (film) 3360, 3280, 3100, 3060, 1650, 1630 cm^{-1}; ^{1}H NMR ($CDCl_3$, 90 MHz) 7.2 (m, 1 H), 6.9 (m, 2 H), 5.7 (m, 1 H), 5.1 (m, 2 H), 4.3 (dd, J_1 = 7.5 Hz, J_2 = 6 Hz, 1 H), 2.5 (m, 2 H), 1.7 (bs, 2 H, NH_2); GC/MS 152 (M^+ − 1), 112 (M^+ − allyl) (100). Anal. Calcd for $C_8H_{11}NS$: C, 62.7; H, 7.24. Found: C, 62.56; H, 7.25.

1-Dodecen-4-amine (4l): IR (film) 3360, 3280, 3070 cm^{-1}; ^{1}H NMR ($CDCl_3$, 90 MHz) 5.7 (m, 1 H), 5.1 (m, 2 H), 2.8 (m, 1 H), 2.2 (m, 2 H), 2.0–0.7 (m, 17 H, and NH_2); GC/MS 142 (100) (M^+ − allyl). Anal. Calcd for $C_{12}H_{25}N$: C: 78.62; H, 13.74. Found: C, 78.43; H, 13.67.

***N*^{1}-(Triphenylmethyl)-8-nonene-1,6-diamine (4m):** IR (film) 3380, 3320, 3280, 1650, 1630, 1590 cm^{-1}; ^{1}H NMR ($CDCl_3$) 7.3 (m, 15 H), 5.7 (m, 1 H), 5.1 (m, 2 H), 3.4 (m, 1 H), 2.2 (m, 4 H), 1.4 (m, 8 H, NH_2, NH). The crude amine was converted to the benzamide for the purpose of identification. Spectra are as follows.

***N*^{1}-(Triphenylmethyl)-*N*^{6}-(benzyloxycarbonyl)-8-nonene-1,6-diamine (4m'):** mp 112–114 °C; IR ($CHCl_3$) 3440, 3080, 3060, 3000, 1650 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz) 7.7 (m, 1 H); 7.5–7.1 (m, 19 H), 5.8 (m, 1 H, N*H*CO), 5.1 (m, 2 H), 4.2 (m, 1 H), 2.3 (m, 2 H), 2.1 (m, 2 H), 1.7–1.2 (m, 8 H, Ph_3NH); exact mass calcd for $C_{35}H_{38}N_2O$ m/e 502.29841, found 502.29852. Anal. Calcd for $C_{35}H_{38}N_2O$: C, 83.63; H, 7.62; Found: C, 83.35; H, 7.71.

1-Octen-4-amine (4n): IR (film) 3360, 3280, 3080; ^{1}H NMR ($CDCl_3$) 5.7 (m, 1 H), 5.1 (m, 2 H), 2.8 (m, 1 H), 2.1 (m, 2 H), 1.8–0.7 (m, 9 H, NH_2); GC/MS 126 (M^+ − 1); 86 (M^+ − allyl) (100). Anal. Calcd. for $C_8H_{17}N$: C, 75.51; H, 13.48. Found: C, 75.43; H, 13.44.

Acknowledgment. This investigation was supported by Ministero Pubblica Istruzione (Fondi 60%) and Progetto Finalizzato Chimica Fine e Secondaria II.

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(8) Generally speaking, after addition of *i*-Bu_2AlH the reactions were monitored by GC and further processed after complete disappearance of the starting nitrile (2–3 h).

(9) Allylmagnesium chloride was purchased from Aldrich. No difference could be noted with freshly prepared reagent.

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Thioaldehydes and Thioketones from 1,3-Dithiolane *S*-Oxides

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Received September 27, 1989

Thiocarbonyl compounds such as thioaldehydes or thioketones are highly reactive and, consequently, interesting synthetic intermediates. However, unless sterically or electronically stabilized, they show a pronounced tendency to di-, oligo-, or even polymerize.[1] Therefore, a prerequisite for synthetic uses of reactive thiocarbonyl compounds is their generation under conditions that avoid oligo- or polymerization and, preferably, also minimize

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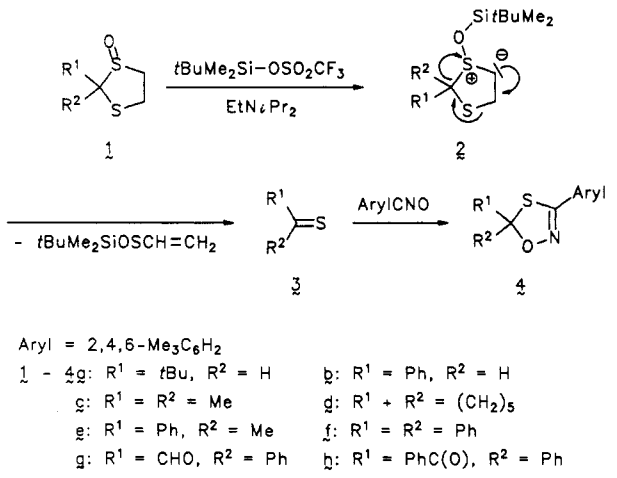
Scheme I



unpleasant odors. Possible routes include the Norrish II type cleavage of ω-alkylthio acetophenones[2] or elimination reactions from appropriate sulfides.[3] Our previous work has shown that the base-induced [3 + 2]cycloreversion of 1,3-dithiolane derivatives offers a convenient solution to this problem[4] with the 4,5-dicarboxylates being particularly useful as precursors of thioaldehydes[5] as well as thioketones.[6]

We now report that 1,3-dithiolane *S*-oxides **1**, which are readily accessible by oxidation of the parent dithiolanes, offer particularly facile access to such thiocarbonyl targets, employing a sequence of silylation and deprotonation. In the silylation step, trimethylsilyl iodide or the triflate can be used; however, the best yields are obtained using *tert*-butyldimethylsilyl triflate.[9] Hünig base is employed for deprotonation giving the *S* ylides **2** as likely intermediates. Interestingly, in contrast to acyclic sulfoxides,[7] under our conditions compounds **1** give no elimination reaction that would provide 1-alkenyl sulfides, i.e., here 2*H*-1,3-dithioles. However, this elimination does occur in the presence of a double excess of base at −78 °C.[8]

The presence of thioketones **3e,f,h** that are relatively stable is obvious from the intense color of the reaction mixture. However, as in the other cases, we did not attempt to isolate these products but determined the yields using the particularly efficient 1,3-dipolar cycloaddition with mesitonitrile oxide giving the heterocycles **4**. The silyl sulfenate that is the other expected fragment in the cycloreversion of **2** does not interfere with this trapping reaction.

Thus, the relatively stable thione **3f** is generated and converted into heterocycle **4f**[6] in 47% (using trimethylsilyl iodide) or 56% yield (using *tert*-butyldimethylsilyl triflate). Moreover, the approach allows—obviously by kinetically controlled deprotonation of **1a,b** on C-5—trapping of thioaldehydes **3a,b** as **4a**[5] (44%) and **4b**[5] (39%), respectively. The successful cycloadditions giving **4c**[6] (34%), **4d**[6] (65%), and **4e**[6] (67%) demonstrate that thioketones with α-hydrogens are not deprotonated to give enethiolates. It is noteworthy that in the generation of **3c** use of *tert*-butyldimethylsilyl triflate as silylating agent is essential.

Interestingly, the present method even offers access to products that have so far not been obtained via dithiolane cycloreversion.[6] Thus, as examples of thioketones with an additional functional group, the oxo-substituted derivatives **3g,h** were generated and trapped as **4g** (crude yield 95%) and **4h** (85%), respectively.

Experimental Section

Infrared spectra were recorded on either a Perkin-Elmer 257 or 399 spectrophotometer. ^1H and ^{13}C NMR spectra were taken on Varian T60, Bruker WP80-FT, WH 270, or WM 400 spectrometers. Proton chemical shifts are reported in ppm relative to internal TMS and carbon shifts relative to the center peak of $CDCl_3$ (δ = 77.0). Low-resolution electron-impact mass spectra were obtained with a CH7 or CH311 Varian Mat mass spectrometer operating at 70 eV. The exact mass measurement was obtained on a VG Analytical 70-250S instrument.

Dithiolane *S*-Oxides 1. Synthesis of **1a,b**[10] and **1c–f**[6] by oxidation of the corresponding 1,3-dithiolanes with 3-chloroperbenzoic acid was carried out as reported previously. The same procedure supplied **1g** [yield 50%, mp 110 °C; IR (KBr): 1705 (C=O), 1045 cm^{-1} (S=O); ^1H NMR (270 MHz, $CDCl_3$) δ 3.25–3.35, 3.35–3.48 (each m, 1 H, SCH), 3.58–3.70, 3.82–3.93 (each m, 1 H, S(O)CH), 7.5–7.6 (m, 5 H, H Ar), 9.43 (s, 1 H, CHO) (major diastereomer); 2.70–2.85, 3.3–3.4 (each m, 1 H, SCH), 3.60–3.72, 4.03–4.16 (each m, 1 H, S(O)CH), 7.35–7.6 (m, 5 H, H Ar), 9.76 (s, 1 H, CHO) (minor diastereomer, ratio 10:1); ^{13}C NMR ($CDCl_3$ δ 33.43 (SCH_2), 58.76 ($S(O)CH_2$), 90.61 (CS_2), 126.61, 129.15, 129.23, 130.26 (C Ar), 189.28 (CHO) (major diastereomer); 33.99 (SCH_2), 53.77 ($S(O)CH_2$), 90.61 (CS_2), 127.14, 128.78, 129.47, 129.53 (C Ar), 190.68 (CHO) (minor diastereomer)] as well as **1h** [yield 97% (ratio of diastereomers 11:5), mp 130–135 °C (major diastereomer), 180–182 °C (minor diastereomer); IR (KBr) 1655 (C=O), 1060 cm^{-1} (S=O); ^1H NMR (250 MHz, $CDCl_3$) δ 3.41, 3.69, 4.02 (m, 2 H, 1 H, 1H, SCH, S(O)CH), 7.2–7.7 (m, 10 H, H Ar) (major diastereomer); 2.59 (ddd, 1 H, J = 8.1, 11.3, 13.8 Hz, SCH), 3.06 (ddd, 1 H, J = 1.1, 6.2, 13.8 Hz, SCH), 3.62 (ddd, 1 H, J = 1.1, 8.1, 10.8 Hz, SCH), 4.12 (ddd, 1 H, J = 6.2, 10.8, 11.3 Hz, SCH), 7.2–7.9 (m, 10 H, H Ar) (minor diastereomer); ^{13}C NMR ($CDCl_3$) δ 35.2 (sec, CH_2S), 57.6 (sec, CH_2SO), 91.0 (quart, CS_2), 128.3, 128.7, 129.1, 129.8, 130.4 (tert, arom C), 131.1, 134.9 (quart, arom C), 194.7 (quart, CO) (major diastereomer); 35.1 (sec, CH_2S), 48.1 (sec, CH_2SO), 97.8 (quart, CS_2), 127.2, 128.3, 129.6, 129.9, 133.6 (tert, arom C), 133.0, 135.7 (quart, arom C), 191.1 (quart, CO) (minor diastereomer)].

General Procedure for Generation and Trapping of 3. Under nitrogen and protected from moisture, *tert*-butyldimethylsilyl triflate[9] (0.48 mL, 2 mmol) in CH_2Cl_2 (20 mL) was added to diisopropylethylamine (0.32 mL, 1.65 mmol) at 20 °C. Then *S*-oxide **1** (1 mmol) was added without solvent and subsequently mesitonitrile oxide (160 mg, 1 mmol). In the case of very sensitive thiones, *S*-oxide **1** and the trapping agent may be introduced simultaneously. After stirring for 1 h, aqueous NH_4Cl and then saturated aqueous NaCl are added. Product **4** is isolated by column chromatography; if separation of the adduct from unreacted nitrile oxide should prove difficult (as for **4d**), the latter is transformed into an isoxazole derivative by addition of 1-hexene and stirring for 15–20 h prior to chromatography on silica (eluents ethyl acetate/petroleum ether 1:13).[11] For isolated yields of pure

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products, vide supra. Products **4a,b**[5] as well as **4c-f**[6] have been reported previously.

4g: oil; IR (neat) 1730 (C=O), 1610, 1575 cm^{-1} (C=N); ^1H NMR (270 MHz, $CDCl_3$) δ 2.23, 2.29 (s, 3 + 6 H, Me), 6.88 (s, 2 H, mesityl-H), 7.4-7.6 (m, 5 H, phenyl H), 9.71 (s, 1 H, CHO); mass spectrum m/e 311 (1, M^+). Anal. Calcd for $[C_{17}H_{16}NOS]^+$ (M - CHO) m/z 282.09444, found m/z 282.09450.

4h: mp 103-104 °C; IR (KBr): 1662 (C=O), 1600, 1585 cm^{-1} (C=N); ^1H NMR (80 MHz, $CDCl_3$): δ 2.15, 2.23 (s, 3 + 6 H, Me), 6.87 (s, 2 H, mesityl H), 7.0-8.2 (m, 10 H, phenyl H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 19.31, 21.04 (Me), 112.4 (C-5), 122.3-139.97 (aryl C), 155.66 (C-3), 193.42 (C=O). Anal. Calcd for $C_{24}H_{21}NO_2S$: C, 74.39; H, 5.46; N, 3.62; S, 8.27. Found: C, 74.04; H, 5.54; N, 3.67; S, 8.13.

Acknowledgment. We thank the Deutsche Forschungsgemeinschaft (Scha 231/5-2) and the Fonds der Chemischen Industrie, Frankfurt, for financial support.

Competitive Intramolecular Cyclizations of Epoxides to Aromatic and Double Bond Positions

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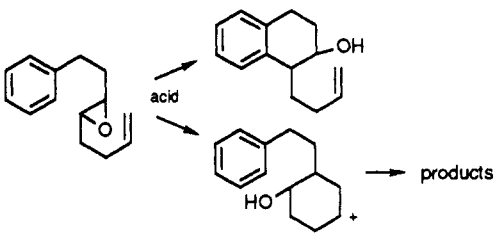
Received February 3, 1989 (Revised Manuscript Received March 13, 1990)

Epoxy-ene cyclizations have been extensively investigated,[1] and epoxy-arene cyclizations have received considerable attention recently.[2,3] In both the former[4] and latter[2,3,5] transformations, Baldwin's rules have been useful in predicting cyclization preferences and relative yields.[2-4]

We felt it would be instructive to investigate epoxides which can undergo intramolecular cyclization to either a double bond or aromatic position (eq 1). The determi-



(1)

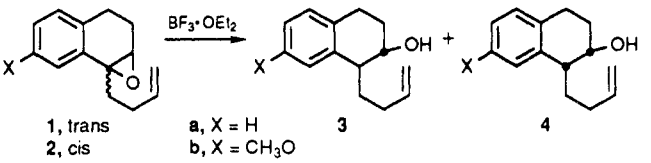
nation of the relative facility of these two processes, under conditions where stereoelectronic effects can play a major role, is the thrust of this report. We believe this is the first systematic report of this nature. In certain cases, it is shown that Baldwin's rules can be used to predict which ring formation process will occur (even though these rules heretofore have not been used to compare the relative propensity of epoxy-ene versus epoxy-arene cyclizations). In other cases, no predictions can be made based on Baldwin's rules, and our work is a first step in elucidating the cyclization preferences in these types of situations.

Results and Discussion

Since five-membered ring formation is less favorable than six-membered ring formation,[1-5] eq 1 represents the most probable transformations of **1a** and **2a,b** (Table I). When treated with $AlCl_3$, $SnCl_4$, or BF_3·OEt_2, all of these

† Student research participants from Olivet Nazarene University.

Table I. Products of the Reaction of BF_3·OEt_2 and 3,4-Epoxy-1-aryl-7-octenes



epoxide	trans:cis	ratio of 3:4[a]	yield,[b] % of 3 + 4
1a	>99:1	99:1	72
2a	8:92	7:93	62
2b	10:90	13:87	91

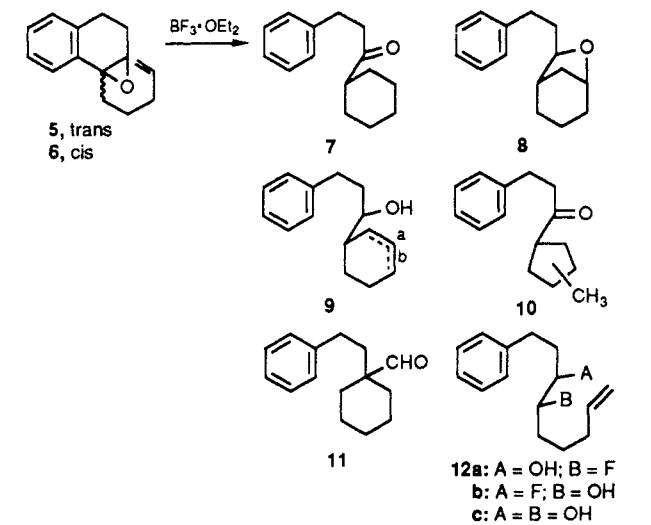
[a] Ratios were determined by capillary GC. [b] NMR yields.

Table II. Product Composition for the Reactions of 5 and 6

		percent composition[a]							
epoxide	trans:cis	7	8	9a[b]	9b	10	11	12a + b	12c
5	>99:1	8	41	12	11	10	<1	6	12
6	8:92	63	2	1	1	5	20	8	-

[a] Area percents by flame ionization detector GC. [b] A small quantity of an allylic alcohol, **6c** the 1-substituted cyclohexenyl isomer of **9c**, was detected at 5 min reaction time, but this compound disappeared rapidly after that.

Scheme I



epoxides cyclize predominantly to the aromatic group. However, BF_3·OEt_2 gave the highest yields and smallest amounts of halohydrin side products. A catalytic quantity of BF_3·OEt_2 in CH_2Cl_2 is sufficient to transform epoxides **1a**, **2a** and **2b** in good yields to the products shown in Table I. These compounds account for 95% or greater of the volatile products. It should also be noted that the reactions are highly stereospecific as cis epoxides lead to cis-disubstituted tetralols and trans epoxides give the

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