



A Publication  
of Reliable Methods  
for the Preparation  
of Organic Compounds

## Working with Hazardous Chemicals

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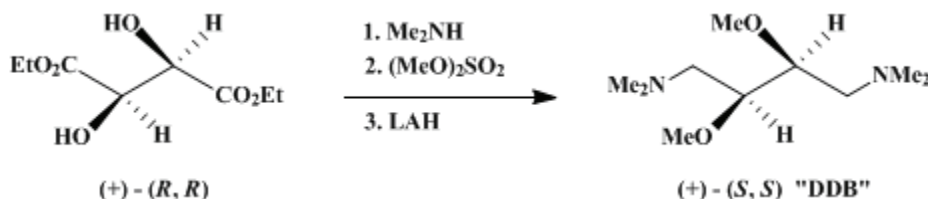
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*These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

*Organic Syntheses, Coll. Vol. 7, p.41 (1990); Vol. 61, p.24 (1983).*

**CHIRAL MEDIA FOR ASYMMETRIC SOLVENT INDUCTIONS.  
(*S,S*)-( + )-1,4-BIS(DIMETHYLAMINO)-2,3-DIMETHOXYBUTANE  
FROM (*R,R*)-( + )-DIETHYL TARTRATE**

[1,4-Butanediamine, 2,3-dimethoxy-*N,N,N',N'*-tetramethyl-*[S,S]*-]



Submitted by Dieter Seebach, Hans-Otto Kalinowski, Werner Langer, Gerhard Crass, and Eva-Maria Wilka<sup>1</sup>.

Checked by M. F. Semmelhack and Diane Facciolo.

### 1. Procedure

*Caution! Because of the high toxicity and carcinogenicity of dimethyl sulfate (Step B), this step should be carried out in a well-ventilated hood.*

A. (*R,R*)-( + )-*N,N,N',N'*-Tetramethyltartaric acid diamide. Into a mixture of 618 g (3 mol) of diethyl tartrate (Note 1) and 600 mL of freshly distilled methanol in a 2-L Erlenmeyer flask is poured at least 450 mL (7 mol) of liquid, anhydrous, cold ( $-78^{\circ}\text{C}$ ) dimethylamine (Note 2). The mixture is swirled briefly and then allowed to stand in a hood for 3 days with a drying tube in place. After seeding (Note 3) and cooling in a refrigerator overnight, the massive crystals are collected by suction filtration. The filtrate is concentrated, seeded, and cooled to yield a second crop. The combined crystals are washed with cold methanol ( $-30^{\circ}\text{C}$ ) and dried under reduced pressure at  $70$ – $100^{\circ}\text{C}$  (oil bath). The diamide thus obtained is sufficiently pure to be used in the following step. The yield is 570–580 g (93–95%). Recrystallization from methanol–ethyl acetate furnishes an analytically pure sample, mp  $189$ – $190^{\circ}\text{C}$ ,  $[\alpha]_{\text{D}} +43^{\circ}$  (ethanol,  $c$  3.0).

B. (*R,R*)-( + )-2,3-Dimethoxy-*N,N,N',N'*-tetramethylsuccinic acid diamide. Into a 4-L, three-necked, round-bottomed flask, fitted with a mechanical stirrer, reflux condenser, and stopper, are introduced 240 mL of 50% aqueous sodium hydroxide (3 mol), 1.5 L of methylene chloride, 0.2 g of benzyltriethylammonium chloride (TEBA), and then 260 g (2.06 mol) of dimethyl sulfate (Note 4). The mixture is stirred vigorously (Note 5), and a total of 204 g (1 mol) of the powdered tartaric acid diamide is added in portions at such a rate as to maintain refluxing (Note 6). Stirring is continued for 24 hr without heating, whereupon 1 L of water is added. Separation of the organic phase, extraction of the aqueous layer with three 300-mL portions of methylene chloride, drying of the combined organic solutions over sodium sulfate, and removal of the solvent in a rotary evaporator (bath temperature below  $80^{\circ}\text{C}$ , water aspirator vacuum) furnishes a slightly yellow oil that crystallizes at  $25^{\circ}\text{C}$  and is sufficiently pure for use in the following reduction step. Recrystallization from cyclohexane/benzene yields 220 g (95%, (Note 7)) of colorless prisms, mp  $63.2$ – $63.5^{\circ}\text{C}$ ,  $[\alpha]_{\text{D}} +116^{\circ}$  (benzene,  $c$  3).

C. (*S,S*)-( + )-1,4-Bis (dimethylamino)-2,3-dimethoxybutane (DDB). A 4-L, three-necked, round-bottomed flask is fitted with a heating jacket, mechanical stirrer, reflux condenser with drying tube, and a stoppered, pressure-equalizing dropping funnel, flushed with nitrogen or argon, and charged with 2.2 L of dry tetrahydrofuran (THF, (Note 8)) and 60 g (1.6 mol) of lithium aluminum hydride ( $\text{LiAlH}_4$ , (Note 9)). A mixture of 250 mL of THF and 232 g (1.0 mol) of the diamide is added, with stirring, at a rate sufficient to reach and maintain refluxing. After the addition is completed, the reaction mixture is kept boiling for 2 hr. The flask is immersed in an ice bath, and 60 mL of water, 180 mL of 10% aqueous potassium hydroxide, and again 60 mL of water are added cautiously with very vigorous stirring. The hydrogen gas that is generated is led well above the stirring motor into the hood exhaust. Temporarily,

the slurry becomes viscous and difficult to stir; during this period addition has to be made extremely carefully. The pale yellow, completely hydrolyzed slurry is filtered by suction, the filter cake extracted twice by refluxing with THF in a round-bottomed flask, and the combined solutions are concentrated in a rotary evaporator. The residual liquid is distilled through a 20-cm Vigreux column; bp 62–64°C (3 mm), yield 180 g (88%). For use in organometallic reactions, DDB is freshly distilled from  $\text{LiAlH}_4$ ,  $[\alpha]_D^{20} + 14.7^\circ$  (neat),  $d_4^{20}$  0.896 (Note 10).

## 2. Notes

1. Commercial (*R,R*)-( + )-diethyl tartrate can be used. The submitters prepared it from (*R,R*)-( + )-tartaric acid (Firma Benckiser, D-Ludwigshafen, or Firma Boehringer, D-Ingelheim),  $[\alpha]_D^{20} + 12.7^\circ$  (water, *c* 17): a 4-L, three-necked, round-bottomed flask equipped with a mechanical stirrer, water separator for organic solvents heavier than water, and a stopper is charged with 1.5 kg (10 mol) of tartaric acid, 1.5 L (26 mol) of 96% ethanol, 1 L of chloroform, and 30 g of freshly activated (1 *N* HCl), highly acidic ion-exchange resin (Lewatit 3333). The stirred mixture is heated at reflux until no more water separates (up to 60 hr). Filtration, evaporation, and vacuum distillation (oil bath temperature must not exceed 145°C, no column, fast distillation) yield 1.85 kg (90%) of the ester,  $[\alpha]_D^{20} + 8.16^\circ$  (neat).
2. Dimethylamine (bp 6°C) is condensed into a 1-L flask cooled to –78°C and fitted with an inlet tube and an opening protected from atmospheric moisture with a silica gel drying tube. It is either taken from a cylinder or freed from 1.5 L of a stirred 40% aqueous solution by heating at 60–80°C with 50 g of potassium hydroxide and leading the amine vapors first through a reflux condenser, then through a 50-cm (2 cm i.d.) drying tube filled with potassium hydroxide pellets, and finally into the receiver flask cooled to –78°C.
3. Sometimes spontaneous crystallization occurs; if it does not, a small amount of the solution is withdrawn and evaporated on a watch glass, and the crystals that are obtained by scratching with a glass rod are used for seeding.
4. Dimethyl sulfate was purchased from Riedel de Haen, D-Seelze-Hannover, and used without purification.
5. Since the reaction mixture becomes very gelatinous on addition of the tartaric acid amide, a powerful motor and a large stirring blade are necessary.
6. Since dimethyl sulfate decomposes rapidly in concentrated alkaline medium, addition of the powdered tartaric acid amide must begin *immediately* after the dimethyl sulfate is introduced. The amide should be added *as fast as possible* (ca. 20–30 min) within the limits of the capacity of the reflux condenser and the mechanical stirrer. The amount of dimethyl sulfate can be increased up to 2.5 equivalents and fresh benzyltriethylammonium chloride can be added toward the end of the addition. With less rapid addition and stirring, the yield drops to 45–55%.
7. The yield obtained by the checkers was 78%.
8. Tetrahydrofuran (THF) was obtained from BASF AG, D-Ludwigshafen, and was distilled twice from potassium hydroxide pellets.
9. Lithium aluminum hydride ( $\text{LiAlH}_4$ ) was used as a white powder purchased from the Metallgesellschaft AG, D-Frankfurt.
10. The specific rotation is highly sensitive to the water content of the DDB; only material distilled from  $\text{LiAlH}_4$  shows this value.

## 3. Discussion

The three compounds whose syntheses are described in the present procedure have been reported previously by the submitters.<sup>2,3</sup>

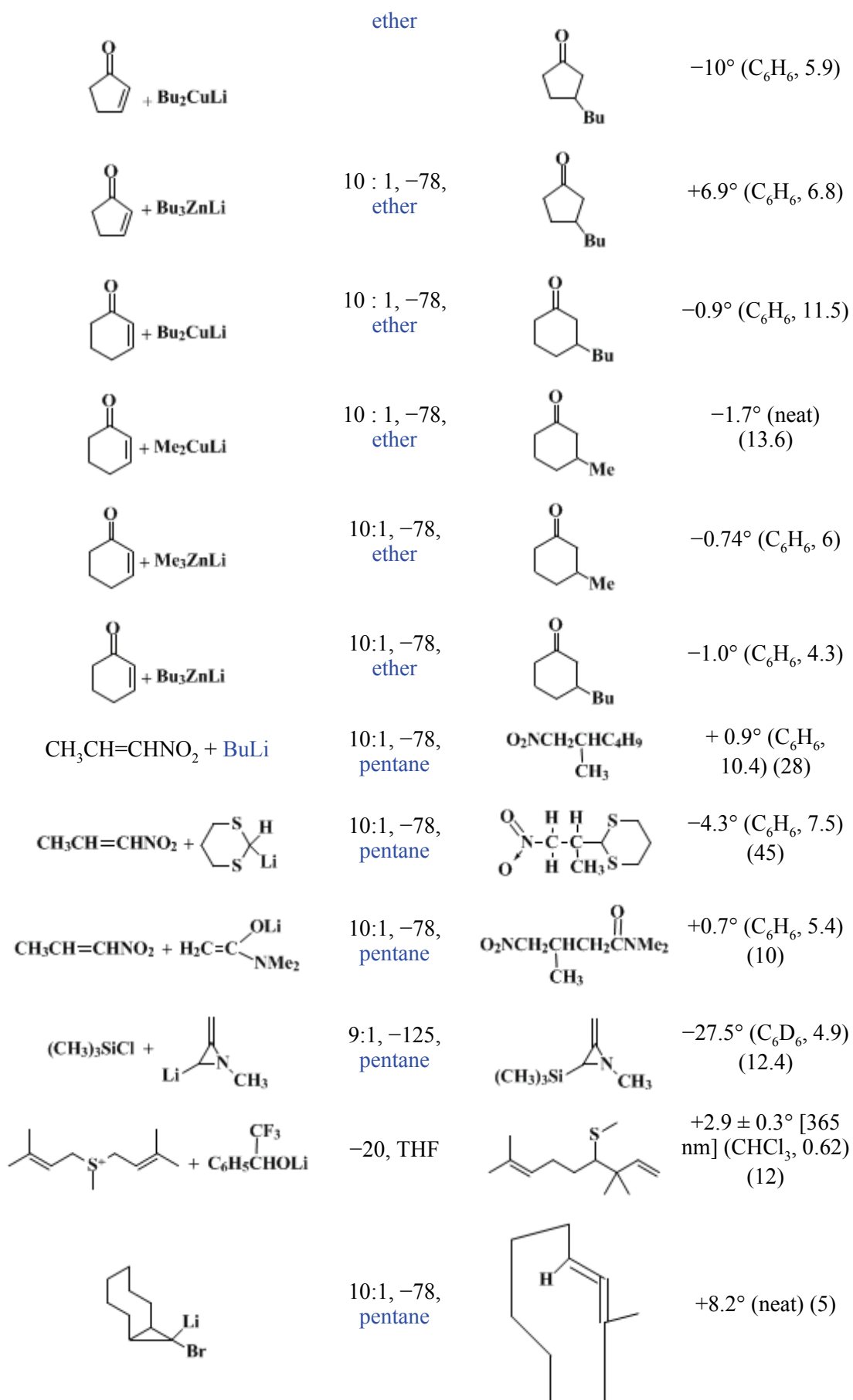
The amino ether DDB has been used extensively as a chiral solvent for asymmetric syntheses.<sup>2,3,4,5,6,7,8</sup> It is readily available on a large scale in both enantiomeric forms: starting from the unnatural (*S,S*)-( – )-tartaric acid,<sup>9</sup> ( – )-DDB is equally accessible<sup>3</sup> following the procedures described herein.

As demonstrated by the examples listed in Table I, DDB induces chirality in enantioface, enantiotope, and enantiomer differentiating<sup>10</sup> reactions in which it acts as a metal (Li, Mg, Cu, Zn) complexing ligand, as a hydrogen-bond mediating component, and as a base catalyst. It can be used at

temperatures as low as  $-150^{\circ}\text{C}$  if mixed with appropriate cosolvents.<sup>3</sup> It is readily recovered and separated from products by acid extraction during workup. The enantiomeric excess (e.e.) obtained in this asymmetric induction is generally in the range of 10–20%; in optimized and/or fortuitous cases, optical yields of up to 50% have been obtained. The chemical yields are as high as in conventional achiral solvent systems. An application of DDB is described on p. 000 of this volume.

TABLE 1  
ASYMMETRIC SYNTHESIS WITH (+)-DDB AS A CHIRAL AUXILIARY AGENT<sup>2,3,4,5,6,7,8</sup>

| Reagents  | Conditions<br>(DDB :<br>Reagent,<br>Temp. $^{\circ}\text{C}$ ,<br>Solvent) | Product | $[\alpha]_{\text{D}}$ (Solvent, $c$ )<br>(%, e.e)        |
|---|--|---------|--|
| $\text{C}_6\text{H}_5\text{CHO} + \text{Bu}_2\text{Mg}$   | 2 : 1, $-78$ ,<br>ether  |         | $-2.5^{\circ}$ ( $\text{C}_6\text{H}_6$ , 7.0)<br>(8)    |
| $\text{C}_6\text{H}_5\text{CHO} + \text{BuLi}$  | 10 : 1, $-150$ ,<br>pentane  |         | $+7^{\circ}$ (neat) (40)                                 |
| $\text{C}_6\text{H}_5\text{CHO} + i\text{-PrLi}$  | 4 : 1, $-120$ ,<br>pentane   |         | $+6.1^{\circ}$ (ether, 7.5)<br>(14)                      |
| $\text{C}_6\text{H}_5\text{CHO} + (\text{C}_6\text{H}_5\text{S})_3\text{CLi}$   | 10 : 1, $-78$ ,<br>pentane   |         | $+23^{\circ}$ ( $\text{C}_6\text{H}_6$ , 1.03)<br>(12)   |
| $\text{C}_6\text{H}_5\text{CHO} + \text{CH}_3\text{N}(\text{CH}_2\text{Li})\text{NO}$   | 10 : 1, $-78$ ,<br>pentane   |         | $+6.5$ ( $\text{CH}_2\text{Cl}_2$ , 3.0)<br>(14.8)       |
| $\text{C}_6\text{H}_5\text{CHO} + \text{CH}_2=\text{C}(\text{O}^-\text{Li}^+)(\text{O}^-\text{C}(\text{CH}_3)_3)$                                   | 10 : 1, $-78$ ,<br>pentane   |         | $+5.1^{\circ}$ ( $\text{C}_6\text{H}_6$ , 11.1)          |
| $\text{C}_6\text{H}_5\text{CHO} + \text{CH}_2=\text{C}(\text{OLi})(\text{CNMe}_2)$  | 10 : 1, $-78$ ,<br>pentane   |         | $+9.1^{\circ}$ ( $\text{C}_6\text{H}_6$ , 12.4)<br>(14)  |
| $(\text{C}_6\text{H}_5)_2\text{CO} + \text{CH}_3\text{CH}(\text{OLi})\text{CNMe}_2$   | 10 : 1, $-78$ ,<br>pentane   |         | $+8.5^{\circ}$ ( $\text{C}_6\text{H}_6$ , 11.3)<br>(~22) |
| $\text{C}_6\text{H}_5\text{COCH}_3 + \text{LiAlH}_4$  | $-78$ , pentane  |         | $+4.7^{\circ}$ (neat) (11)                               |
| $\text{C}_6\text{H}_5\text{CH}=\text{NC}_6\text{H}_5 + \text{Li}^+\text{S}(\text{CH}_2)_2\text{S}(\text{CH}_2)_2\text{S}(\text{CH}_2)_2\text{Li}^+$ | 3 : 1, $-30$ ,<br>hexane   |         | $+15.5^{\circ}$ ( $\text{CH}_2\text{Cl}_2$ ,<br>18.7)    |
|   | 10 : 1, $-78$ ,  |         |  |



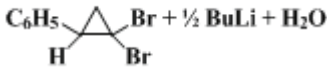
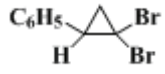
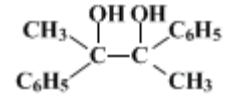
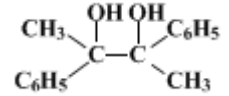
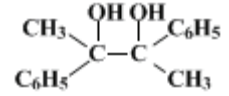
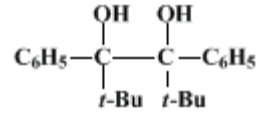
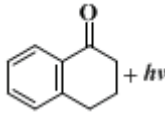
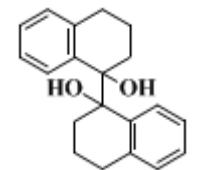
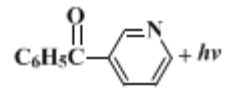
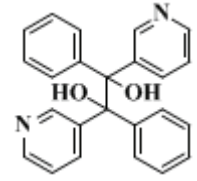
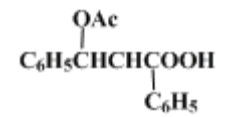
|   |                        |  |   |
|---|------------------------|--|---|
|    | 10:1, -100,<br>pentane |    | +1.9° (neat)  |
| $C_6H_5COCH_3 + hv$   | 7.5:1, -72,<br>pentane |    | +8.0° (C <sub>2</sub> H <sub>5</sub> OH,<br>5.0) (23.5)                                       |
| $C_6H_5COCH_3 + \text{cathodic, electrochemical reduction}$                         | 24, methanol           |    | +2.2° (C <sub>2</sub> H <sub>5</sub> OH,<br>5.0) (6.4)  |
| $C_6H_5COCH_3 + \text{Li/naphthalene}$  | 6:1, 25,               |    | 2.5° (C <sub>2</sub> H <sub>5</sub> OH,<br>5.0) (7.3)   |
| $C_6H_5CO-t-Bu + hv$  | -30, neat DDB          |    | -1.2° [(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> O,<br>5.0]                               |
|    | -35, pentane           |    | +22.3° (CHCl <sub>3</sub> ,<br>5.0)   |
|  | -15, neat DDB          |   | -20.0°<br>(CH <sub>3</sub> SOCH <sub>3</sub> , 2.0)   |
| $C_6H_5CHO + C_6H_5CH_2COOH + \text{AcOAc}$   | 6:1, -25               |  | diastereomer a<br>+5.0°,<br>diastereomer b<br>+26.0° (C <sub>2</sub> H <sub>5</sub> OH,<br>3) |

TABLE II  
COMPARISON OF TMB<sup>a</sup> WITH DDB<sup>b</sup> USED AS COSOLVENTS IN THE  
ADDITION OF BUTYLLITHIUM TO VARIOUS ALDEHYDES AT -78°C IN  
PENTANE<sup>3</sup>

| R  | RCHO + C <sub>4</sub> H <sub>9</sub> Li → RCH(OH)C <sub>4</sub> H <sub>9</sub> |                       | Sense of Rotation, Absolute Configuration |
|--|--|-----------------------|---|
|  | With TMB <sup>a</sup>  | With DDB <sup>b</sup> |   |
| CH <sub>3</sub>                                  | 1.2  | 7.5                   | (+)-S                                     |
| C <sub>2</sub> H <sub>5</sub>                    | 8.8  | 11.5                  | (+)-S                                     |
| <i>i</i> -C <sub>3</sub> H <sub>7</sub>          | 18.0   | 19.0                  | (+)-R                                     |
| <i>t</i> -C <sub>4</sub> H <sub>9</sub>          | 22.8   | 13.5                  | (+)-R                                     |
| (C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> CH | 20.0   | 19.0                  | (+)                                       |
| <i>c</i> -C <sub>6</sub> H <sub>11</sub>         | 25.0   | 22.5                  | (+)-R                                     |
| C <sub>6</sub> H <sub>5</sub>                    | 30.0   | 19.0                  | (+)-R                                     |

|  |      |      |     |
|--|------|------|-----|
| 4-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>                     | 32.5 | 11.5 | (+) |
| 2-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>                     | 45.3 | 10.5 | (+) |
| 2,4,6-(CH <sub>3</sub> ) <sub>3</sub> -C <sub>6</sub> H <sub>2</sub> | 23.0 | 2.3  | (+) |

<sup>a</sup>TMB = (*S,S*)-(–)-1,2,3,4-tetramethoxybutane.

<sup>b</sup>DDB = (*S,S*)-(+)–1,4-bis (dimethylamino)-2,3-dimethoxybutane.

Another chiral cosolvent, which is less readily separated from low-boiling and/or water-soluble products and is somewhat less stable toward organolithium reagents, is (*S,S*)-(–)-1,2,3,4-tetramethoxybutane (TMB).<sup>3</sup> As is shown in Table II, it is a cosolvent that is superior to DDB in differentiating between the enantiotopic faces of aldehydes with organolithium reagents.<sup>3</sup> Finally, the octamethyl-1,4-diamino-2,3-bis (2-aminoethoxy)butane (DEB)<sup>5</sup> can be used in a 2 : 1 ratio with alkyllithium reagents to produce carbinols in even higher enantiomeric yields.

DDB, TMB, and DEB are far superior to other neutral chiral auxiliary agents used in the same reactions.<sup>3,10,11,12,13</sup>

This preparation is referenced from:

- *Org. Syn. Coll. Vol. 10*, 613
- *Org. Syn. Coll. Vol. 10*, 115

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## References and Notes

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## Appendix

### Chemical Abstracts Nomenclature (Collective Index Number);

## (Registry Number)

(S,S)-( + )-1,4-Bis (dimethylamino)-2,3-dimethoxybutane (DDB)

Lithium aluminum hydride (LiAlH<sub>4</sub>)

C<sub>6</sub>H<sub>5</sub>CHO

Bu<sub>2</sub>Mg

i-PrLi

(C<sub>6</sub>H<sub>5</sub>S)<sub>3</sub>CLi

C<sub>6</sub>H<sub>5</sub>COCH<sub>3</sub>

CH<sub>3</sub>CH=CHNO<sub>2</sub>

C<sub>6</sub>H<sub>5</sub>CO-t-Bu

C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>COOH

AcOAc

octamethyl-1,4-diamino-2,3-bis (2-aminoethoxy)butane (DEB)

Li

[ethanol \(64-17-5\)](#)

[HCl \(7647-01-0\)](#)

[Benzene \(71-43-2\)](#)

[ethyl acetate \(141-78-6\)](#)

[methanol \(67-56-1\)](#)

[ether \(60-29-7\)](#)

[hydrogen \(1333-74-0\)](#)

[sodium hydroxide \(1310-73-2\)](#)

[chloroform \(67-66-3\)](#)

[sodium sulfate \(7757-82-6\)](#)

[nitrogen \(7727-37-9\)](#)



dimethyl sulfate (77-78-1)

cyclohexane (110-82-7)

potassium hydroxide (1310-58-3)

Naphthalene (91-20-3)

tartaric acid,  
(87-69-4)

dimethylamine (124-40-3)

Pentane (109-66-0)

methylene chloride (75-09-2)

BuLi (109-72-8)

Tetrahydrofuran,  
THF (109-99-9)

lithium aluminum hydride,  
 $\text{LiAlH}_4$  (16853-85-3)

hexane (110-54-3)

argon (7440-37-1)

benzyltriethylammonium chloride (56-37-1)

diethyl tartrate

(R,R)-( + )-tartaric acid (87-69-4)

(R,R)-( + )-N,N,N',N'-Tetramethyltartaric acid diamide (26549-65-5)

(S,S)-( + )-1,4-BIS(DIMETHYLAMINO)-2,3-DIMETHOXYBUTANE,  
1,4-Butanediamine, 2,3-dimethoxy-N,N,N',N'-tetramethyl-[S,S]-,  
(S,S)-( + )-1,4-bis (dimethylamino)-2,3-dimethoxybutane (26549-21-3)

(R,R)-( + )-DIETHYL TARTRATE (87-91-2)

(R,R)-( + )-2,3-Dimethoxy-N,N,N',N'-tetramethylsuccinic acid diamide (26549-29-1)