# Carbohydrate as a chiral template: optical resolution of *N-tert*-butanesulfinamides

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## Abstract

*N-tert*-butanesulfinamides, a class of amines bearing a sulfinyl group attached to nitrogen, exhibit pyramidal bonding where the non-bonded electron pair located at the sulfur atom acts as a fourth ligand. These compounds are configurationally sufficiently stable to be separated into *R*- and *S*- enantiomers. Enantiopure *N-tert*-butanesulfinamides are important auxiliaries in asymmetric synthesis, and some of them also have useful biological properties. In this paper, a new efficient strategy for enantioselective synthesis of *N-tert*-butanesulfinamides with very goods yields and excellent enantiomeric excess *via* the hydrolysis reaction of *N*-glycosidic bonds that were formed from D-ribose, under basic conditions was described.

**Keywords.** *N-tert*-butanesulfinamide, *N*-glycoside, D-ribose, enantiomer, asymmetric synthesis.

## 1. INTRODUCTION

The past two decades have seen an explosion in interest in the synthesis and utility of molecules containing a stereogenic sulfur center [1]. Sulfoxides are found in a variety of natural products. They have also been employed as chiral auxiliaries in a range of reaction classes, and more recently as chiral ligands. Especially, chiral *N-tert*-butanesulfinamides are increasingly being utilized as versitale chiral nitrogen intermediates for the preparation of amines, aminoacids [2]. These amides are useful synthons for asymmetric synthesis of biogically active molecules [3]. Moreover, *tert*-butanesulfinamide is increasingly being applied across many additional research areas, including the development of agrochemicals, natural product synthesis, and the preparation of chemical tools for a wide range of biological investigations. *N-tert*-butanesulfinamides exists in two forms as an enantiostereomeric mixture: (*R*)-**1** and (*S*)-**1** (figure 1).
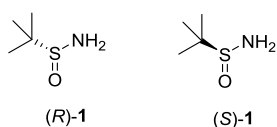


*Figure 1*: Two enantiopures of *N-tert*-butanesulfinamides

The synthesis and isolation of enantiomerically pure *N-tert*-butanesulfinamide (**1**) was first reported by Ellman and coworkers in 1997 [4]. Since then, several different approaches to the synthesis of this compound have been reported, including enantioselective oxidation [5], resolution of racemic material [6], diastereoselective synthesis utilizing stoichiometric chiral auxiliaries [7], and catalytic enantioselective sulfinyl transfer [8].

In this paper, we described a new efficient strategy for enantioselective synthesis of *N-tert*-butanesulfinamides with very goods yields and excellent enantiomeric excess *via* the hydrolysis reaction of *N*-glycosidic bonds that were formed from D-ribose, under basic conditions.

## 2. EXPERIMENTAL

All reagents were obtained commercially and used without further purification. All reactions have been carried out under a nitrogen atmosphere and dry conditions. The solvents used were freshly distilled under anhydrous conditions, unless otherwise specified. The reaction mixtures have been magnetically stirred with Teflon stirring bars, and the temperatures were measured externally. The reactions have been monitored by thin layer chromatography (TLC) with detection by UV light, or a p-anisaldehyde staining solution. Acros silica gel (60, particle size 0.040-0.063 mm) was used for column chromatography. Nuclear magnetic

resonance (NMR) spectra have been recorded with Bruker Avance 500 spectrometers. The optical rotation values have been measured with a Perkin-Elmer 141 Polarimeter, at 589 nm. The concentration was reported in gram per milliliter (c, g. ml$^{-1}$).

**Racemic sulfinamides** (±)-**1** *m*-CPBA (*m*-Chloroperoxybenzoic acid) (7 g, 31 mmol) in CH$_2$Cl$_2$ (50 mL) was added dropwise to a stirred solution of *tert*-butanedisulfide (5 g, 28 mmol) in CH$_2$Cl$_2$ (15 mL) at 0 °C over 15 min. The solution was stirred for 30 min at 0 °C, thenat room temperature until the reaction was complete by TLC (3 h). The reaction mixture was poured into a separatory funnel containing CH$_2$Cl$_2$ (50 mL) and saturrated NaHCO$_3$ (50 mL) and further extracted with CH$_2$Cl$_2$ (2×50 mL). The organic layer was removed and washed with saturated NaHCO$_3$ (3×50 mL), saturated NaCl (50 mL), dried over Na$_2$SO$_4$ and concentrated in vacuo to give 5.17 g (95 %) of *tert*-butylthiosulfinate: $^1$H NMR (300 MHz) δ 1.32 (sulfide) 1.39 (s, 9H, (CH$_3$)$_3$S), 1.57 (s, 9H, (CH$_3$)$_3$S=O). This intermediate was dissolved in CH$_2$Cl$_2$ (15 mL) and a solution of SO$_2$Cl$_2$ (3.6 g, 27 mmol) in CH$_2$Cl$_2$ (5 mL) was added dropwise at 0°C. The resulting yellow solution was stirred for 1 h allowing it to gradually reach room temperature. Excess SO$_2$Cl$_2$ was removed under vacuum and the resulting product, *tert*-butylsufinyl chloride, was diluted in CH$_2$Cl$_2$ (50 mL) and added dropwise to NH$_4$OH (100 mL) at 0 °C over 30 min. After stirring for 30 min at room temperature, the reaction mixture was saturated with NaCl and extracted with CH$_2$Cl$_2$ (3x50 mL). The combined organic layers were washed with saturated NaCl (100 mL), dried over Na$_2$SO$_4$ and concentrated in vacuo to give the crude sulfinamide. Purification using flash chromatography (4:1 *n*-pentane/EtOAc) gave the title compound (±)-**1** (0.71 g, 22 %) as a white solid: mp 98-100 °C $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm): 2.01 (s, br, 2H (NH$_2$)); 1.32 (s, 9H C(CH$_3$)$_3$). $^{13}$C NMR (125MHz, CDCl$_3$) δ (ppm): 26.6; 62.9.

**2,3-O-Isopropylidene-D-ribose, (2)** To a stirred suspension of D-ribose (40 g, 266 mmol) in acetone (500 mL) was added dropwise concentrated H$_2$SO$_4$ (1.5 mL) at room temperature and the reaction mixture was stirred at this temperature for 2.5 h. The mixture was neutralized with solid NaHCO$_3$, filtered and evaporated under reduced pressure to give a colorless syrup. The residue was purified by silica gel column chromatography using n-hexane and ethyl acetate (1:2) as the eluent to afford **2** as a colorless syrup (47.1 g, 93 %): $^1$H NMR (500 MHz, MeOH-d$_4$), δ (ppm): 5.26 (s, 1H, *anomeric H*), 4.77 (d, 1H, *J* = 6.0, *OH*), 4.52 (d, 1H, *J* = 6.0, CH$_2$*OH*),

4.19 (td, 1H, *J* = 4.4 Hz, *J* = 5.2 Hz, *CH*CH$_2$OH), 3.63 (dd, 1H, *J* = 4.8 Hz, *J* = 12.0 Hz, HOCH(CO)*CH*), 3.59 (dd, 1H, *J* = 5.6 Hz, *J* = 12.0 Hz, HOCH$_2$CH*CH*), 1.44 (s, 3H, *CH$_3$*), 1.31 (s, 3H, *CH$_3$*). [α]$^{23}$$_D$ = -36.1 (c 1.45, acetone).

**1-[(4R,5S)-5-((1S)-1-Hydroxyallyl)-2,2-dimethyl [1,3] dioxolan-4-yl]ethane-1,2-diol (3)** To a stirred solution of **2** (1.2 g, 5,4 mmol) in THF (40 mL) was added dropwise vinylmagnesium bromide (24 mL, 24 mmol, 1.0 M solution in THF) at -78 °C and the reaction mixture was stirred at 0 °C for 3 h. After adding water (10 mL) at 0 °C, the resulting precipitate was removed through a pad of Celite. The filtrate was extracted with ethyl acetate (25 mL), dried, filtered, and evaporated under reduced pressure to give an oil, which was purified by silica gel column chromatography using *n*-hexane and ethyl acetate (1:2) as the eluent to afford **3** as a white solid (0.98 g, 81 %): mp: 73-74 °C; $^1$H NMR (500 MHz, MeOH-d$_4$), δ (ppm): 5.97 (m, 1H, CH$_2$=*CH*), 5.31 (td, 1H, *J* = 1.6, 17.2, *CH*H=CH), 5.17 (td, 1H, *J* = 1.6 Hz, 10.8 Hz, *CH*H=CH), 4.24 (m, 1H, CH$_2$=CH*CH*OH), 4.11 (dd, 1H, *J* = 5.6 Hz, *J* = 9.6 Hz,CH$_2$=CHCH*OH*), 3.96 (dd. 1H, *J* = 5.2 Hz, *J* = 9.6 Hz, HOCH$_2$CH(*OH*)), 3.84 (m, 1H, *HO*CH$_2$CH(OH)), 3.77 (dd, 1H, *J* = 2.4 Hz, *J* = 11.2 Hz, (CH$_3$)$_2$CO*CH*), 3.59 (dd, 1H, *J* = 6.0, *J* = 11.2 Hz, (CH$_3$)$_2$CO*CH*), 1.35 (s, 3H, *CH$_3$*), 1.28 (s, 3H, *CH$_3$*). [α]$^{25}$$_D$ = -30.5 (c 1.23, CHCl$_3$).

**(3aS,4R,6S,6aS)- and (3aS,4S,6S,6aS)-2,2-Dimethyl-6-vinyltetrahydrofuro[3,4-d][1,3]dioxol-4-ol (4)** To a stirred solution of **3** (0.19 g, 0.9 mmol) in methylene chloride (10 mL) was added dropwise an aqueous solution of NaIO$_4$ (2.1 mL, 0.13 mmol, 0.65 M solution) at 0 °C and the reaction mixture was stirred at room temperature for 40 min. After water (5 mL) was added, the mixture was extracted with methylene chloride (10 mL), dried, filtered, and evaporated under reduced pressure to give an oil, which was purified by silica gel column chromatography using hexane and ethyl acetate (2:1) as the eluent to give vinylic lactol **4** as a colorless oil (140 mg, 85 %). $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm): 6.01 (m, 0.8 H), 5.79 (m, 0.2 H), 5.50 (d, *J* = 2.8 Hz, 0.8 H), 5.43-5.16 (m, 2.2 H), 4.70-4.56 (m, 3 H), 3.93 (d, *J* = 10.4 Hz, 0.2 H), 2.66 (d, *J* = 2.8 Hz, 0.8 H), 1.59 (s, 0.6 H), 1.51 (s, 2.4 H), 1.39 (s, 0.6 H), 1.33 (s, 2.4 H).

**Synthesis of compounds 5 and 6.** The mixture of racemic *N-tert*-butanesulfinamide (336 mg, 2.77 mmol) and the vinylic lactol **4** [9] (516 mg, 2.77 mmol) were dissolved in CH$_2$Cl$_2$ (15 mL), then Cs$_2$CO$_3$ (1.35 g, 4.2 mmol) was added. The mixture was refluxed for 3h, cooled, and filtered through a pad of celite. The solids were washed with CH$_2$Cl$_2$,

and the combined filtrates were evaporated in vacuo to afford a mixture of two diastereoisomers **5** and **6** (ration: 50/50 by ^{1}H NMR spectroscopy of crude product, 803 mg, 98 %). The two diastereoisomers were separated by chromatography on silica gel. 400 mg of each isomer for next step (eluent: *n*-pentan/EtOAc: 2/3) was obtained.

Only compound **5** (118.5 mg, quantitative) was obtained by condensation reaction between vinylic lactol **4** (76 mg, 0.41 mmol) and (*S*)-*N*-*tert*-butanesulfinamide (50 mg, 0.41 mmol) in presence of $Cs_2CO_3$ (202 mg, 0.62 mmol).

With the same method, the condensation reaction between vinylic lactol **4** (50 mg, 0.25 mmol) and *(R)*-*N*-*tert*-butanesulfinamide (30.2 mg, 0.25 mmol) in presence of $Cs_2CO_3$ (123 mg, 0.38 mmol) affords only a compound **6** (72.2 mg, quantitative).

**(*S*)-*N*-((3a*S*,4*R*,6*S*,6a*S*)-2,2-dimethyl-6-vinyl tetrahydrofuro [3,4-d] [1,3] dioxol-4-yl)-2-methylpropane-2-sulfinamide (5).** Rf = 0.35; ^{1}H NMR (500 MHz, CDCl$_3$) δ (ppm) 5.77 (ddd, *J* = 4.0 Hz, *J* = 10.7 Hz, *J* = 17.4 Hz, 1H (*CH*=CH$_2$)); 5.44 (ddd, *J* = 1.4 Hz, *J* = 1.8, Hz, *J* = 17.4 Hz, 1H (CH=*CH$_2$*)); 5.18 (ddd, *J* = 1.4 Hz, *J* = 1.8, Hz, *J* = 10.7 Hz, 1H (CH=*CH$_2$*)); 5.06 (dd, *J* = 3.8 Hz, *J* = 11.1 Hz, 1H (*CH*NH)); 4.61 (dd, *J* = 0.3 Hz, *J* = 6.0 Hz, 1H (*CH*-CH-CH=CH$_2$)); 4.55 (dd, *J* =0.3 Hz, *J* = 4.0 Hz, 1H (*CH*-CH-CH=CH$_2$)); 4.48 (dd, 1H, *J* = 3.8 Hz, *J* =6.0 Hz, 1H (*CH*-CHNH)); 4.45 (d, *J* = 11.1 Hz, 1H (*NH*)); 1.51 (s, 3H, *CH$_3$*); 1.33 (s, 3H, *CH$_3$*); 1.23 (s, 9H, (*CH$_3$*)$_3$). ^{13}C NMR (125 MHz, CDCl$_3$) δ (ppm) 22.3, 24.8, 26.0, 56.3, 81.2, 83.5, 86.5, 113.0, 116.6, 134.1. $[α]^{18}_D$ = +34.3 (c 0.32, CHCl$_3$).

**(*R*)–*N*-((3a*S*,4*S*,6*S*,6a*S*)-2,2-dimethyl-6-vinyl tetrahydrofuro [3,4-d] [1,3] dioxol-4-yl)-2-methylpropane-2-sulfinamide (6).** Rf = 0.32; ^{1}H NMR (500 MHz, CDCl$_3$) δ (ppm) 5.99 (ddd, *J* = 10.6 Hz, *J* = 17.2 Hz, 1H (*CH*=CH$_2$)); 5.42 (dd, *J* = 1.4 Hz, *J* = 17.2 Hz, 1H (CH=*CH$_2$*)); 5.26 (dd, *J* = 1.4 Hz, *J* = 10.6 Hz, 1H (CH=*CH$_2$*)); 5.21 (dd, *J* = 2.8 Hz, *J* = 8.4 Hz, 1H (*CH*NH)); 4.92 (d, *J* = 8.4 Hz, 1H (*NH*)); 4.67 (dd, *J* = 2.8 Hz, *J* = 6.4 Hz, 1H (*CH*-CHNH)); 4.52 (dd, *J* = 3.2 Hz, *J* = 5.9 Hz, 1H, (*CH*-CH=CH$_2$)); 3.38 (dd, *J* = 3.2 Hz, *J* = 6.4 Hz, 1H (*CH*-CH-CH=CH$_2$)); 1.54 (s, 3H, *CH$_3$*); 1.34 (s, 3H, *CH$_3$*); 1.24 (s, 9H, (*CH$_3$*)$_3$). ^{13}C NMR (125 MHz, CDCl$_3$) δ (ppm) 22.4, 25.3, 26.9, 56.2, 83.8, 85.3, 85.7, 92.5, 114.1, 117.1, 136.2; $[α]^{18}_D$ = -23.8 (c 0.26, CHCl$_3$).

**(*S*)-*N*-*tert*-butanesulfinamide ((*S*)-1)** A solution of compound **5** (116 mg, 0.4 mmol) in dry methanol (10 mL) was treated with freshly prepared (0.5 M) solution of sodium methoxide (2 mL). The reaction

mixture was stirred for 4 h (TLC control). Then a few drops of water were added to the mixture, and the mixture was neutralized with acetic acid. The mixture was stirred at room temperature for another 4 h. The mixture was extracted with EtOAc (3x5 mL) and the organic phase was washed with saturated aqueous NaHCO$_3$ (3x5 mL), dried over MgSO$_4$, filtered, and concentrated in vacuo to yield the crude product. The residue was purified by column chromatography on silica gel *n*-pentane/EtOAc = 2/1, with 0.1 % Et$_3$N], yielding the corresponding compound (*S*)-**1** (47 mg, 97 % yield) as a white solid; mp = 100-102 °C; ^{1}H NMR (500 MHz, CDCl$_3$) δ (ppm): 2.01 (s, br, 2H (*NH$_2$*)); 1.32 (s, 9H C(*CH$_3$*)$_3$). ^{13}C NMR (125 MHz, CDCl$_3$) δ (ppm): 26.6; 62.9; $[α]^{20}_D$ = -4,9 (c 1.0, CHCl$_3$).

**(*R*)-*N*-*tert*-butanesulfinamide ((*R*)-1)** In the same method, the hydrolysis reaction of compound **6** (73 mg, 0.25 mmol) in MeOH (10 mL) in presence of MeONa 0.5 M (1.2 mL, 0.6 mmol) affords a compound (*R*)-**1** (30.2 mg, quantitative); mp = 100-102°C, $[α]^{20}_D$ = +4.9 (c 1.0 CHCl$_3$) In both cases, the compound **4** will be recovered in quantitave yield.

## 3. RESULTS AND DISCUSSION

Racemic sulfinamides from *tert*-butanedisulfide were prepared by very simple route ain 3 steps [10]. The oxidation of *tert*-butanedisulfide by *m*-CPBA (*m*-Chloroperoxybenzoic acid) in CH$_2$Cl$_2$ gave thioester in quantitative yield. Without purification, this intermediate was chlorinated by SO$_2$Cl$_2$ solution to give tert-butanesulfinyl chloride, following the nucleophilic substitution with NH$_4$OH to afford racemic *N*-*tert*-butanesulfinamide as a white solid in 22 % overall yield (figure 2).

Lactol **4** was easily prepared in three steps in 55 % overall yield from D-ribose by following a literature procedure (figure 3) [9].

The protection of the two secondary hydroxyl groups as an acetonide was accomplished by reaction of D-ribose with acetone in the presence of concentrated H$_2$SO$_4$ at room temperature, afforded **2** in 80 % yield. The addition of vinylmagnesium bromide to the aldehyde **2** gave the triol **3** in 81 % yield. Finally, the desired product **4** was obtained in 85 % yield by the cleavage of the diol with sodium periodate. ^{1}H NMR analysis of the crude product showed that the vinyl lactol **4** is in the form of a 80/20 mixture of two diastereoisomers. In this paper, characteristics of all known intermediates were confirmed by ^{1}H NMR of crude product.
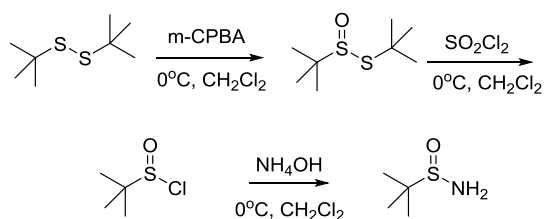
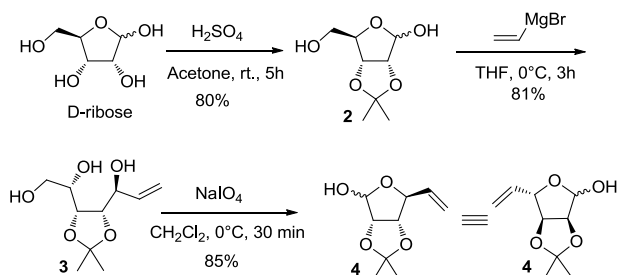*Figure 2*: Synthesis of racemic
*N-tert*-butanesulfinamide



*Figure 3*: Synthesis of vinylic lactol **4**

Condensation of lactol **4** with racemic *N-tert*-butanesulfinamide (±)-**1** afforded, in quantitative yield, a 1:1 mixture of sulfinamides **5** and **6**, which were easily separated by chromatography.

On the other hand, reactions were performed with each enantiomer of starting sulfinamide independently: we observed that the (*S*)-**1** enantiomer yielded only **5**, whereas diastereoisomer **6** was obtained quantitatively from the (*R*)-**1** enantiomer. Therefore, this condensation was stereospecific and was determined by the configuration of the starting sulfinamide (figure 4).
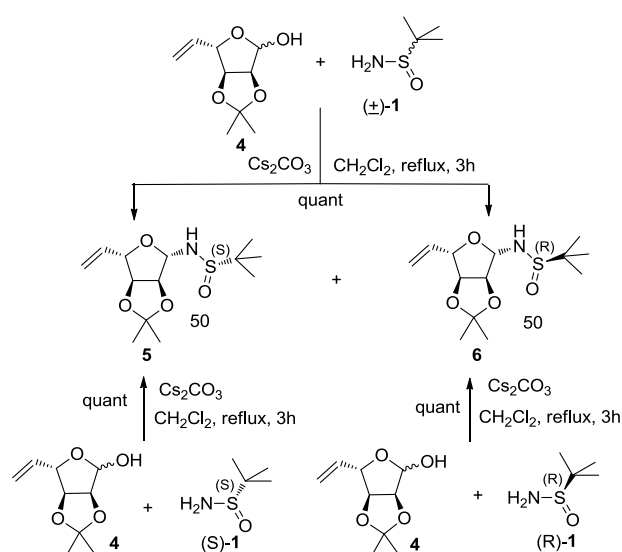


*Figure 4*: Preparation of chiral intermediates by the
condensation reaction with
*N-tert*-butanesulfinylamides

The stereochemistry of derivatives **5** and **6** were established by $^1$H NMR for the protons of the tetrahydrofuran rings (table 1): the *anti*-diastereoisomer $^3J_{1,2}$ and $^3J_{3,4}$ coupling constants were smaller (0.3-3.8 Hz) than the *syn* one (6.0-6.4 Hz).

Finally, synthesis of each enantiomerically pure of *N-tert*-butanesulfinamides from compound **5** and **6** were easily established by cleveage of the *N*-glycosidic bond under mild basic condition. Thus, to demonstrate the efficient removal of the auxiliary, compound **5** was treated with a solution of sodium methoxide in methanol to give (*S*)-**1** in quantitative yield.



*Table 1*: Stereochemistry and coupling contants of
vicinal protons of compounds **5** and **6**

| Compounds | $^3J_{1\text{-}2}$ | $^3J_{2\text{-}3}$ | $^3J_{3\text{-}4}$ |
|-----------|--------|--------|--------|
| **5** | 0.3 Hz | 6.0 Hz | 3.8 Hz |
| **6** | 3.2 Hz | 6.4 Hz | 2.8 Hz |

In the same way, enantiomer (*R*)-**1** was obtained in quantitative yield by treatment of compound **6** with solution of sodium methoxide in methanol (figure 5).

The $^1$H, $^{13}$C NMR spectra, melting point and optical rotation of two final products (*R*)-**1** and (*S*)-**1** were in complete agreement with the data reported in the literature [6, 7].
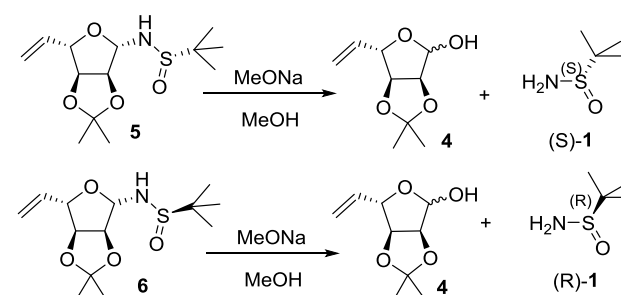


*Figure 5:* Synthesis of two enantiomerically pure
*N-tert*-butanesulfinamides

Further in both cases, the chiral auxilliary vinylic lactol **4** can be recovered in quantitative yield and this molecule can be recycled.

## 4. CONCLUSION

In summary, we have developed a new and efficient procedure to synthesis of enantiopure (*R*)- and (*S*)-*N-tert*-butanesulfinamide, starting from D-ribose and racemic *N-tert*-butanesulfinamide using hydrolytic cleavage of the *N*-glycosidic bond as a key step. This procedure proved to be good for large scale synthesis of each enantiomerically pure of *N-tert*-butanesulfinamides.

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