A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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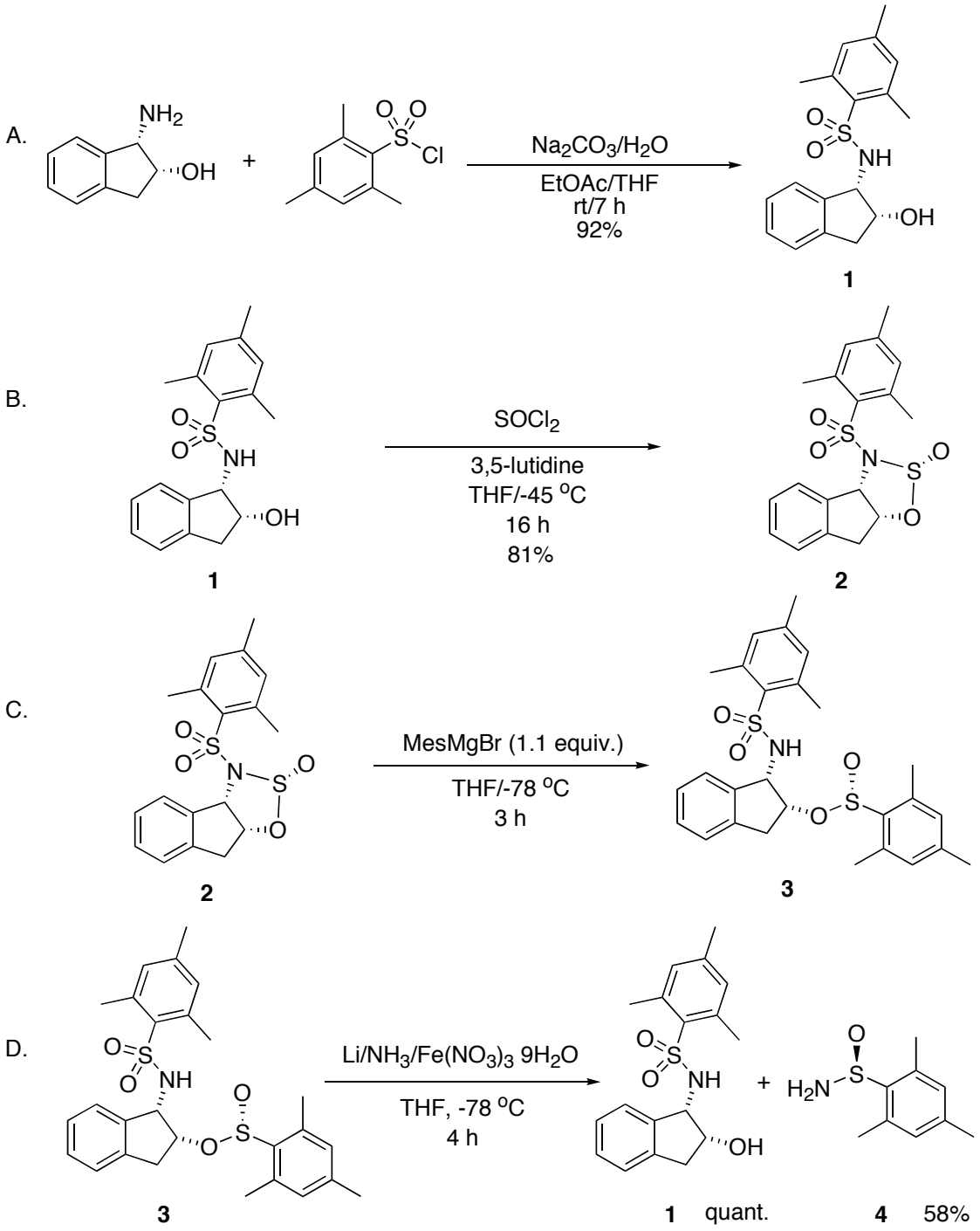
(*S*)-(+)-2,4,6-TRIMETHYLBENZENESULFINAMIDE



Submitted by Tokala Ramachandar, Yongzhong Wu, Junyi Zhang, and Franklin A. Davis.[1]
Checked by Shinji Harada, Riichiro Tsuji, and Masakatsu Shibasaki.

1. Procedure

*A. (1S,2R)-(+)-N-(2-Hydroxyindan-1-yl)-2,4,6-trimethylbenzenesulfonamide (**1**).*[2] A 1-L, 2-necked, round-bottomed flask equipped with a magnetic stirring bar, a 100-mL pressure-equalizing addition funnel, and a glass stopper is charged with sodium carbonate (21.34 g, 0.2 mol) and water (100 mL). The reaction mixture is stirred for 20 min. (1*S*,2*R*)-(–)-*cis*-1-Amino-2-indanol (15.0 g, 0.1 mol) (Note 1) is added, followed by ethyl acetate (250 mL) (Note 2). After 30 min, the addition funnel is charged with 2,4,6-trimethylbenzenesulfonyl chloride (21.3 g, 0.097 mol) (Note 1) in 50 mL of ethyl acetate and tetrahydrofuran (1:1) (Note 3), which is added dropwise to the mixture within 20 min. After the reaction mixture is stirred at ambient temperature for 7 h, water (100 mL) and ethyl acetate (200 mL) are slowly added. The resulting mixture is transferred to a 2 L separatory funnel. The reaction flask is rinsed with ethyl acetate (50 mL) and the ethyl acetate solution is added to the separatory funnel. The mixture is shaken, the phases are separated and the aqueous phase is extracted with ethyl acetate (2 × 200 mL). The combined organic phase is washed with water (100 mL), 1 N hydrochloric acid (2 × 100 mL), water (100 mL), brine (50 mL), dried over anhydrous sodium sulfate, and filtered by gravity. The solvent is removed under reduced pressure at 30 °C or lower to give a white/slightly yellow solid, which is dissolved in a 1 L Erlenmeyer flask containing ethyl acetate (150 mL). The solution is warmed to 40 °C in a water bath, and *n*-hexane (300 mL) (Note 4) is added. The solution is kept at –20 °C (Note 5) for 16 h and the resulting 30.9 g (92%) of white crystalline solid of **1** (Note 6) is collected.

*B. 3-(2,4,6-Trimethylbenzenesulfonyl)-3,3a,8,8a-tetrahydro-2H-1-oxa-2λ^4-thia-3-aza-cyclopenta[a]inden-2-ol (**2**).*[2] An oven-dried, 1-L three-necked, round-bottomed flask (Note 7) equipped with a magnetic stirring bar, temperature probe, an argon inlet, and a rubber septum is charged with **1** (30.0 g, 0.090 mol). Tetrahydrofuran (180 mL) (Note 3) is added via syringe at room temperature and the solution is cooled to –45 °C (Note 8). To the colorless solution is added thionyl chloride (9.9 mL, 0.135 mol) (Note 9) *via* syringe over a 5 min period. A tetrahydrofuran (150 mL) solution containing 3,5-lutidine (25.8 mL, 0.226 mol) (Note 9) is added to the reaction mixture over 60 minutes *via* cannula at –45 °C. A white precipitate appears almost immediately. After stirring for 16 h at –45 °C, the reaction mixture is quenched at this temperature by dropwise addition of

a saturated aqueous sodium bicarbonate solution (120 mL) using a 250-mL dropping funnel. The mixture is diluted with ethyl acetate (150 mL) and warmed to room temperature with stirring. The mixture is shaken, the phases are separated and the aqueous phase is extracted with ethyl acetate (2 × 200 mL). The reaction flask is rinsed with ethyl acetate (50 mL) and the combined organic phases are transferred to a 2-L separatory funnel. The organic phase is washed with brine (100 mL), dried over anhydrous sodium sulfate, and filtered into a 500-mL round-bottomed flask. The solvent is removed under reduced pressure at 30 °C or lower. The resulting light yellow solid is stirred with *n*-heptane (250 mL) (Note 10) for 2 h affording a white precipitate. The slurry is filtered and the white cake is washed with *n*-heptane (75 mL) to give 33.0 g (yield 96.5%) of white powder. The white powder is dissolved in ethyl acetate (450 mL) in a 1-L Erlenmeyer flask at 40 °C and *n*-hexane (200 mL) is added. The solution is cooled to –20 °C (Note 6) for 16 h and filtered to give 27.5 g (81%) of a white crystalline solid **2** (Note 11).

*C. (R_S,1S,2R)-(–)-2,4,6-Trimethylbenzenesulfinic acid 1-(2,4,6-trimethylbenzenesulfonylamino)-2-indan-2-yl ester (**3**)*.[2] An oven-dried, 1-L three-necked, round-bottomed flask (Note 7) equipped with a magnetic stirring bar, a temperature probe and an argon inlet is charged with white crystalline **2** (20.0 g, 0.053 mol). Tetrahydrofuran (400 mL) is added via syringe at room temperature and the solution is cooled to –78 °C. After 5 min, 2-mesitylmagnesium bromide (58.4 mL, 1.0 M solution in tetrahydrofuran, 0.058 mol) (Note 12) is added to the solution over a 5 min period. The slightly yellow reaction mixture is stirred at –78 °C for 3 h and quenched at this temperature by addition of a saturated sodium bicarbonate aqueous solution (100 mL) via a 250-mL dropping funnel. The mixture is diluted with ethyl acetate (150 mL), warmed to room temperature with stirring, and transferred to a 2-L separatory funnel. The mixture is shaken, the phases are separated, and the aqueous phase is extracted with ethyl acetate (2 × 200 mL). The combined organic phases are washed with brine (100 mL), dried over anhydrous sodium sulfate, and filtered. The solvent is removed under reduced pressure at 30 °C or lower to give 26.8 g (quantitative) of a white foam, which is impure **3** (Note 13). The resulting white foam is used without further purification.

*D. (S)-(+)-2,4,6-Trimethylphenylsulfinamide (**4**)*.[2] An oven-dried, 1-L 3-necked round-bottomed flask (Note 7) is equipped with a magnetic

stirring bar. One neck of the reaction flask is fitted with an acetone-dry ice-cooled cold finger condenser bearing an argon inlet/outlet vented through a mineral oil bubbler. Another neck is fitted with a gas inlet valve, and the third neck is fitted with a dropping funnel. The flask is cooled to –78 °C using an external acetone-dry ice bath. Ammonia (400 mL) is condensed into the flask through the gas inlet valve. The gas inlet is replaced with a stopper and crystalline iron(III) nitrate (0.1 g, 0.25 mmol) (Note 14) is added. The solution develops a brown (Note 15) color immediately. Then lithium wire (10.0 g, 1.443 mol) (Note 16) is added portion wise over 30 min and the resulting gray (Note 17) solution is stirred at –78 °C for 90 min. A solution containing the white foam **3** (20.0 g, 0.039 mol) in THF (100 mL) is added over a 30 min period via dropping funnel. The reaction mixture is stirred at this temperature for 4 h, and quenched at this temperature by addition of solid NH_4Cl (25.0 g) portion-wise over 20 min. The mixture is warmed to room temperature and the ammonia is allowed to evaporate to give a dark solid residue, to which is added Et_2O (200 mL). The mixture is filtered under vacuum into a 1-L round-bottomed flask. The solid residue is thoroughly washed with excess Et_2O (2 × 250 mL) and the combined organic solutions are concentrated. Flash chromatography (SiO_2, EtOAc:hexanes, 1:1) (Note 18) affords 13.3 g (100%) of chiral auxiliary **1** (product in *Part A*) and 4.95 g of crude **4**. This material is dissolved in EtOAc (30 mL) and *n*-hexane (30 mL) is added. The solution is kept at –20 °C (Note 6) for 24 h and then filtered to give 4.25 g (58%) of white crystalline **4** (Note 19 and 20).

2. Notes

1. (1*S*,2*R*)-(–)-*cis*-1-Amino-2-indanol and 2,4,6-trimethylbenzene-sulfonyl chloride, were purchased from Aldrich Chemical Company, Inc.

2. The checkers purchased ethyl acetate from Wako Pure Chemical Industries, Ltd. and used it as received. The submitters purchased ethyl acetate from Fisher Scientific Company.

3. Reagent grade tetrahydrofuran was purified by passing through columns packed with activated alumina and supported copper catalyst (Glass Contour, Irvine, CA).

4. The checkers purchased *n*-hexane from Wako Pure Chemical Industries, Ltd. and used it as received. The submitters purchased *n*-hexane from Fisher Scientific Company.

5. A refrigerator having a freezer compartment at –20 °C was used.

6. Compound (+)-**1** exhibits the following physical and spectroscopic properties: mp 149–150 °C; $[\alpha]_D$ +17.9 (*c* 1.09, $CHCl_3$); IR (KBr): 3476, 3331, 2949, 1601, 1331, 1154 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 2.33 (s, 3 H), 2.72 (s, 6 H), 2.91 (d, *J* = 17.0 Hz, 1 H), 3.08 (dd, *J* = 5.0, 16.5 Hz, 1 H), 4.40–4.43 (m, 1 H), 4.62 (dd, *J* = 5.0, 10.0 Hz, 1 H), 5.34 (d, *J* = 9.5 Hz, 1 H), 7.00 (s, 2 H), 7.12–7.24 (m, 4 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 20.9, 23.0, 39.4, 61.1, 72.9, 124.6, 125.3, 127.2, 128.5, 132.0, 133.7, 139.3, 139.4, 139.6, 142.6; Anal. Calcd for $C_{18}H_{21}NO_3S$: C, 65.23; H, 6.39; N, 4.23. Found: C, 65.16; H, 6.38; N, 4.11.

7. All glassware was dried at 120 °C for at least 4 h and cooled to room temperature in a desiccator prior to use.

8. An acetonitrile/dry ice bath was used.

9. Thionyl chloride and 3,5-lutidine were purchased from Aldrich Chemical Company, Inc and used as received. The checkers found that 1.5 equiv of $SOCl_2$ is necessary to drive the reaction to completion.

10. The checkers purchased *n*-heptane from Wako Pure Chemical Industries, Ltd. and used it as received. The submitters purchased it from Fisher Scientific Company.

11. The known compound (–)-**2**[3] exhibits the following physical and spectroscopic properties: mp 170–171 °C; $[\alpha]_D$ –2.6 (*c* 1.0, $CHCl_3$), IR (KBr): 2937, 1601, 1335, 1158 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 2.38 (s, 3 H), 2.73 (s, 6 H), 3.42 (dd, *J* = 7.0, 18.5 Hz, 1 H), 3.57 (d, *J* = 17.5 Hz, 1 H), 5.54 (d, *J* = 6.5 Hz, 1 H), 5.85 (dt, *J* = 1.5, 7.0 Hz, 1 H), 6.59 (d, *J* = 8.0 Hz, 1 H), 7.07–7.27 (m, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.1, 23.1, 39.3, 66.2, 95.6, 124.7, 125.4, 127.8, 129.4, 131.9, 132.5, 138.41, 138.45, 140.9, 144.7.

12. 2-Mesitylmagnesium bromide (1.0 M solution in tetrahydrofuran) was purchased from Aldrich Chemical Company, Inc.

13. The submitter's report an isolated yield of 23.7 g (90%). A portion of compound **3** was purified for the purpose of characterization by flash chromatography (SiO_2, EtOAc:hexanes, 3:7) as follows: The product (approximately 0.1 g) is charged on a column (40 x 2.5 cm) of 50 g of silica gel and eluted with 200 mL of hexanes. At this point, fraction collection (25-mL fractions) begins, and elution is continued with 400 mL of 15% EtOAc-hexane until compound **3** is obtained in fractions 5-10. These

fractions are concentrated by rotary evaporation (25 °C, 15 mmHg) to give 0.112 g (85%) of a white crystalline solid. The R_f of compound **3** is 0.57. The known compound (–)-**3**[3] exhibits the following physical and spectroscopic properties: mp 145–146 °C; $[\alpha]_D$ –58.8 (*c* 1.0, $CHCl_3$), IR (KBr): 2970, 1602, 1332, 1154 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 2.25 (s, 3 H), 2.32 (s, 3 H), 2.43 (s, 6 H), 2.70 (s, 6 H), 3.08 (dd, *J* = 4.5, 17.0 Hz, 1 H), 3.16 (d, J = 16.5 Hz, 1 H), 4.85 (dd, *J* = 4.5, 9.0 Hz, 1 H), 4.96–4.98 (m, 1 H), 5.53 (d, *J* = 9.5 Hz, 1 H), 6.79 (s, 2 H), 6.98 (s, 2 H), 7.13–7.25 (m, 4 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 18.8, 20.9, 21.1, 23.1, 37.2, 60.2, 81.0, 124.4, 124.7, 127.4, 128.2, 130.7, 132.0, 134.2, 137.4, 137.6, 137.8, 139.3, 140.0, 142.3.

14. Iron(III) nitrate was purchased from Acros Organics, Inc. and was used as received.

15. The submitters reported that a gray color is formed.

16. Lithium wire (3.2 mm diameter in mineral oil, 99.9%, Catalog No: 220914) was purchased from Aldrich Chemical Company, Inc., and was cut into small pieces.

17. The submitter reported that a blue color is formed.

18. The product is charged on a column (60 x 7.5 cm) of 400 g of silica gel and eluted with 500 mL of hexane. At that point, fraction collection (75-mL fractions) begins, and elution is continued with 1 L of 20% EtOAc-hexane until auxiliary **1** is obtained in fractions 10-15. At this time the column is eluted with 1.5 L of 80% of EtOAc-hexane until sulfinamide **4** was obtained in fractions 15-25. The products fractions are concentrated by rotary evaporation (25 °C, 15 mmHg). The R_fs of the two compounds are 0.51 (for chiral auxiliary **1**) and 0.24 (for **4**). The resulting chiral auxiliary **1** was obtained in 9.2 g (69%) by submitters. Compound **1** was used without further purification in *Part B* and the purity of the recovered auxiliary was evaluated by comparison of its physical properties with an authentic sample: mp 149–150 °C; $[\alpha]_D$ +17.9 (*c* 1.09, $CHCl_3$).

19. The known compound (+)-**4**[2] exhibits the following physical and spectroscopic properties: mp 120–122 °C; $[\alpha]_D$ + 303 (*c* 0.46, $CHCl_3$), IR (KBr): 3287, 3103, 1601, 1450, 1062, 1027, 848 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 2.27 (s, 3 H), 2.59 (s, 6 H), 4.46 (brs, 2 H), 6.85 (s, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 19.1, 20.9, 130.8, 136.1, 138.7, 140.7. The enantiomeric excess (97%) was determined by using a Chiralcel OD, 4-6 x 250 mm, 10 μm; 9:1 (hexane/*i*-PrOH), 1.0 mL/min, 250 nm; (*S*)-**4**, r_t = 17.5 min; the minor isomer, (*R*)-**4**, r_t = 23.5 min.

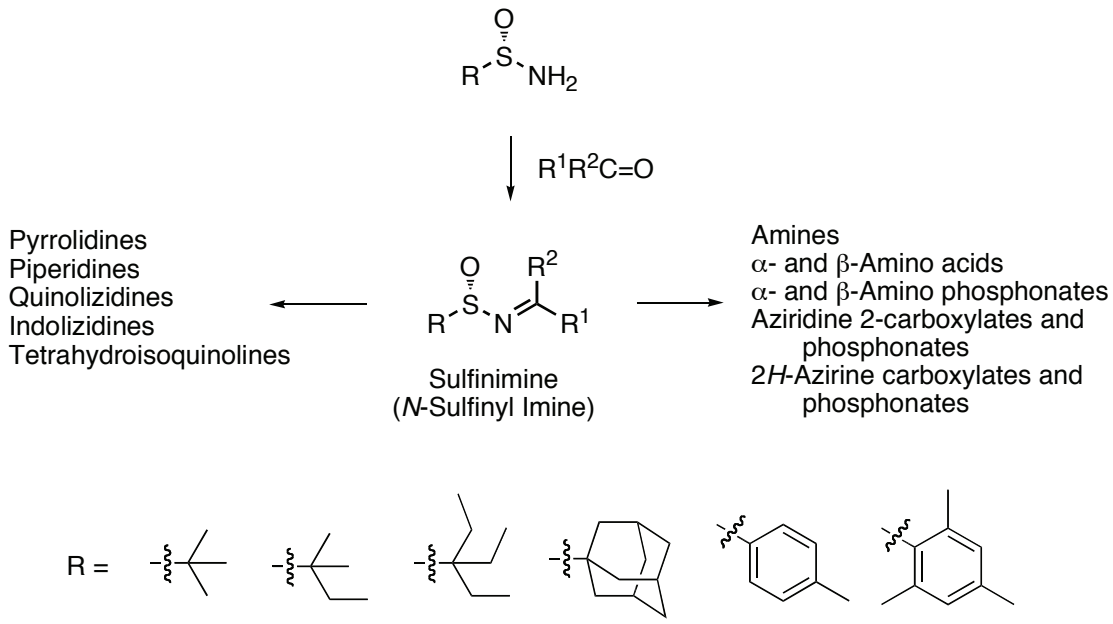
20. The submitters report the use of LiHMDS to effect the conversion of **3** to **4** on a 22.4 gram scale resulted in 11.2 g (50%) of starting material **3**, 6.8 g (46%) of the chiral auxiliary **1** and 3.8 g (46%) sulfinamide **4**. On a 1.0 g scale the LiHMDS procedure gave **4** in 70% yield.[3]

Waste Disposal Information

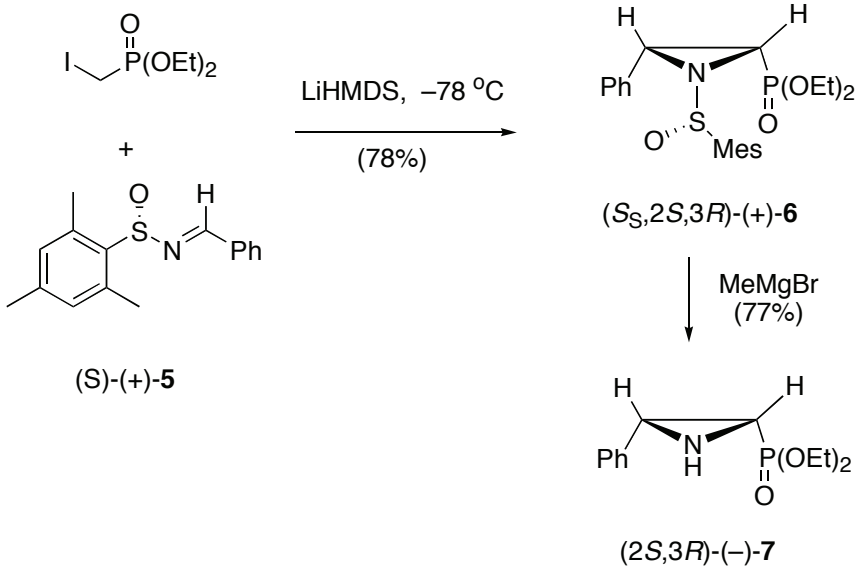
All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

The most direct and reliable method for the asymmetric synthesis of diverse amine derivatives is the addition of an organometallic reagent across the C-N bondof an imine having a chiral *N*-sulfinyl auxiliary.[4] Sulfinimines (*N*-sulfinylimines) provide a general solution to the problem of addition of organometallic reagents across the C-N bond of chiral imines because the sulfinyl group activates the imine for addition, is highly stereodirecting, and easily removed in the sulfinamide product giving the enantiomerically pure amine product of known stereochemistry. Generally sulfinimines are prepared by condensation of an enantiopure (*S*)- or (*R*)-sulfinamide (RS(O)-NH_2) with an aldehyde or ketone in the presence of a weak Lewis acid dehydrating agent such as $Ti(OEt)_4$.[4] For this purpose the commercially available and easily prepared *p*-toluenesulfinamide and *N*-*tert*-butane-sulfinamide are generally employed. However, a useful addition to the sulfinimine protocols would be the ability to "tune" the reactivity of the sulfinimine by varying the steric and electronic properties of the *N*-sulfinyl auxiliary.[5] The method described here for the asymmetric synthesis of (*S*)-(+)-2,4,6-trimethylphenylsulfinamide **4** is a representative general procedure for the asymmetric synthesis of structurally diverse sulfinamides developed by Senanayake and co-workers.[2] In this procedure *N*-sulfonyl-1,2-3-oxathiazolidines-2-oxide **2** is treated with Grignard reagents to afford the sulfenate ester **3**, which on reaction with Li/NH_3 or LiHMDS affords the corresponding enantiopure sulfinamide **4**.[2,3] The reaction of Grignard reagents with **3** has been used to prepared enantiomerically pure sulfoxides.[6]



In the aza-Darzens reaction of (*S*)-(+)-*N*-(benzylidene)-2,4,6-trimethyphenylsulfinamide **5** (prepared using (*S*)-(+)-2,4,6-trimethyl-phenylsulfinamide (**4**) with lithium diethyl iodomethylphosphonate) a single diastereomeric aziridine 2-phosphonate (+)-**6** was obtained.[3] Other *N*-sulfinyl auxiliaries gave mixtures of products or could not be removed with MeMgBr to give the corresponding NH-aziridine 2-phosphonate (–)-**7**. NH-Aziridine 2-phosphonates are valuable chiral building block for the synthesis of α-amino phosphonates[3,7] and the 2*H*-azirine 3-phosphonate which are chiral imino dienophiles.[8] An example of the application of 2,4,6-trimethylsulfinamide has been reported.[9]



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Appendix
Chemical Abstracts Nomenclature; (Registry Number)

(1*S*,2*R*)-(–)-*cis*-Aminoindanol: 1*H*-Inden-2-ol, 1-amino-2,3-dihydro-: (1*S*,2*R*)- (126456-43-7)

2,4,6-Trimethylbenzenesulfonyl chloride; (773-64-8)

(1*S*,2*R*)-(–)-*N*-(2-Hydroxy-indan-1-yl)-2,4,6-trimethyl-benzenesulfonamide: Benzenesulfonamide, *N*-[(1*S*,2*R*)-2,3-dihydro-2-hydroxy-1*H*-inden-1-yl]-2,4,6-trimethyl-; (473554-01-7)

3-(2,4,6-Trimethylbenzenesulfonyl)-3,3a,8,8a-tetrahydro-2*H*-1-oxa-2λ^4-thia-3-aza-cyclopenta[a]inden-2-ol: Indeno[1,2-d]-1,2,3-oxathiazole, 3,3a,8,8a-tetrahydro-3-[(2,4,6-trimethylphenyl)sulfonyl]-, 2-oxide, (2*R*,3a*S*,8a*R*)-; (473554-02-8)

(R_S,1*S*,2*R*)-(–)-2,4,6-Trimethylbenzenesulfinic acid 1-(2,4,6-trimethylbenzenesulfonylamino)-2-indan-2-yl ester: Benzenesulfinic acid, 2,4,6-trimethyl-, (1*S*,2*R*)-2,3-dihydro-1-[[(2,4,6-trimethylphenyl)sulfonyl]amino]-1*H*-inden-2-yl ester, [$S_{(S)}$]-; (607729-49-7)

2-Mesitylmagnesium bromide: Magnesium, bromo(2,4,6-trimethylphenyl)-; (2633-66-1)

(*S*)-(+)-2,4,6-Trimethylbenzenesulfinamide: Benzenesulfinamide, 2,4,6-trimethyl-, [$S_{(S}$]-; (607729-50-0)

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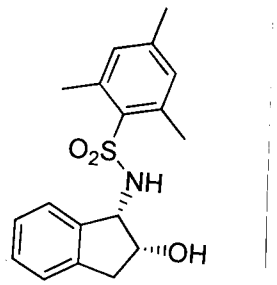

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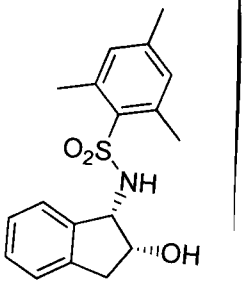

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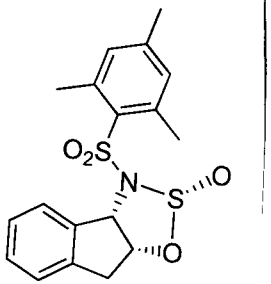

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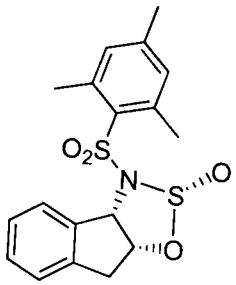

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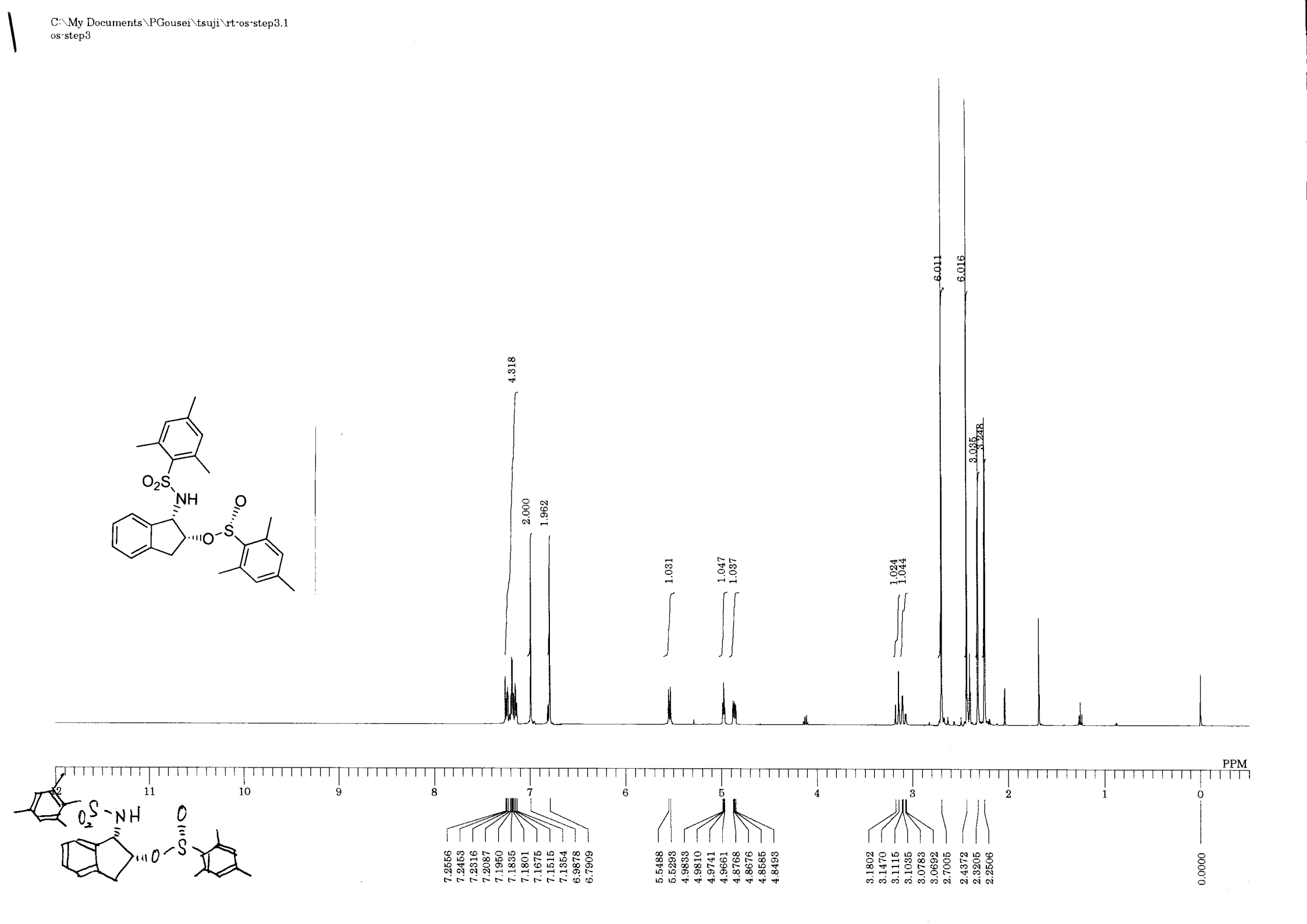

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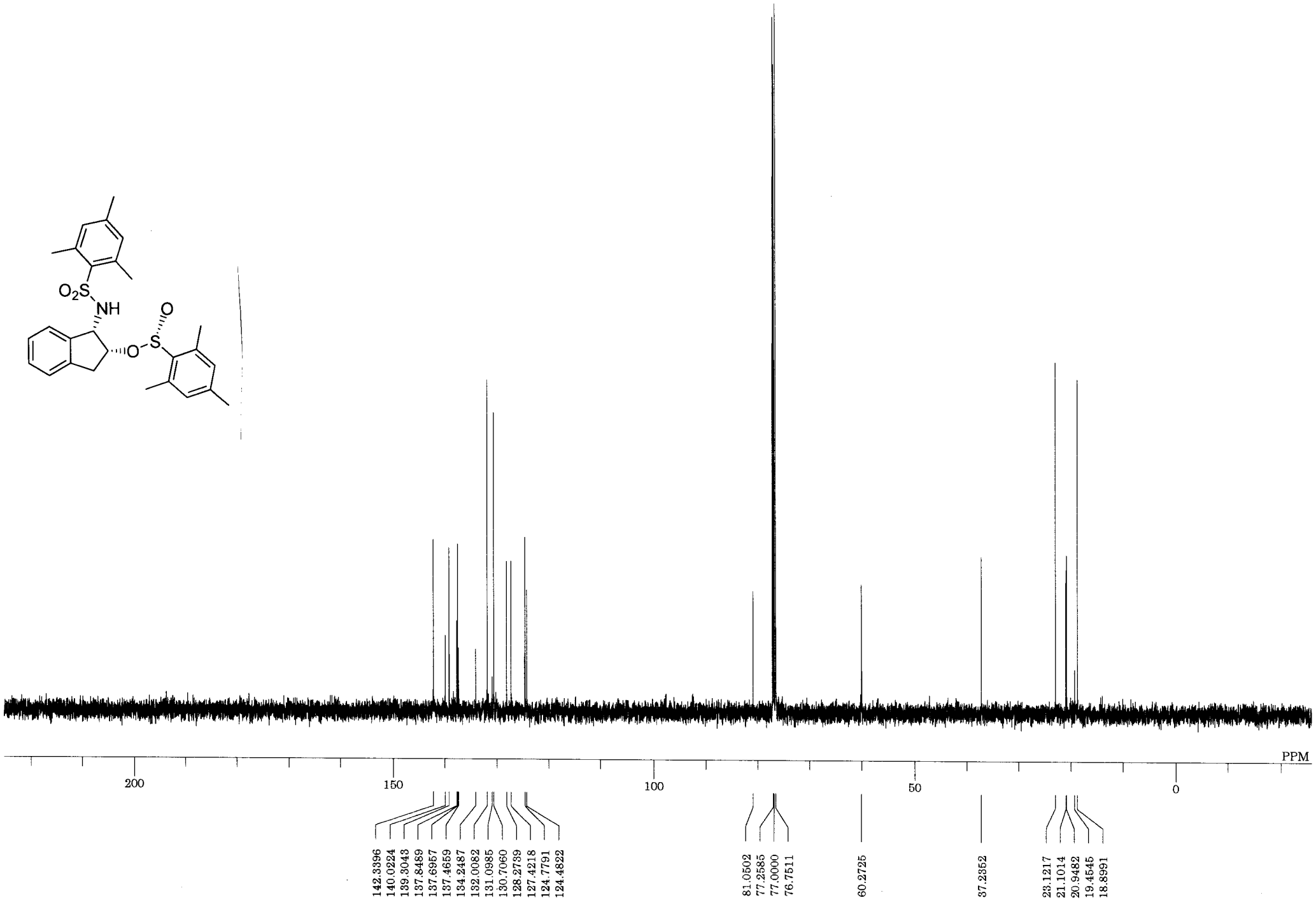

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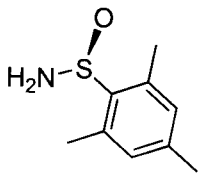
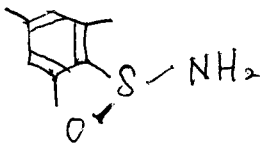
mes-sulfineamide





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mes-sulfineamide