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Diastereoselective Pomeranz–Fritsch–Bobbitt synthesis of (*S*)-(−)-salsolidine using (*R*)-*N*-*tert*-butanesulfinylimine as a substrate

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Abstract—The highly diastereoselective addition of methyl Grignard reagent to chiral (*R*)-*N*-*tert*-butanesulfinylimine **8** was the key step in the synthesis of (*S*)-(−)-salsolidine **1** of high enantiomeric purity. The resulting addition product, *tert*-butanesulfinylamide **9**, was then subjected to cyclization via amine **10** and Pomeranz–Fritsch aminoacetal **11**.

1. Introduction

The recent outgrowth of the sulfinimine-mediated asymmetric synthesis of chiral nonracemic amines and their derivatives, developed by Davis et al.[1] and Ellman et al.,[2] has prompted us to apply this methodology to a stereoselective modification of the Pomeranz–Fritsch–Bobbit synthesis of isoquinoline alkaloids.

The diastereoselective addition of various carbon nucleophiles to enantiopure *N*-sulfinaldimines and ketimines has been extensively exploited as an efficient method for the synthesis of nitrogen-containing natural products and biologically active compounds and has also been the subject of recent reviews.[1,2] Due to the unique reactivity and excellent stereoselectivity, imines bearing *N*-*p*-toluenesulfinyl and *N*-*tert*-butanesulfinyl auxiliaries have been chosen in most of the asymmetric syntheses reported; they can be readily prepared from the commercially available (*R*)- and (*S*)-*p*-toluenesulfinylamides and (*R*)- and (*S*)-*tert*-butanesulfinylamides, respectively.

A number of chiral nonracemic cyclic amines, including alkaloids, have been prepared by employing the sulfinimine approach; the literature concerning the syntheses of various heterocyclic amines using the *p*-toluenesulfinylimines, has been reviewed in 2004.[1]

Several other reports describing the synthesis of aziridines,[3] pyrrolidines,[4] pyrrolizidines,[5] piperidines,[6] indolizidines,[7] tetrahydroisoquinolines[8] and tetrahydropyrimidines,[9] using mostly *N*-*tert*-butanesulfinylimines, both as electrophiles and nucleophiles, have also appeared in the literature.

It should be mentioned that isoquinoline alkaloids and their precursors have been accessed by the sulfinimine-mediated synthesis with success. Ellman et al.[8] using *N*-*tert*-butanesulfinylimine bound to a polymer as substrate, synthesized pavine and the isopavine skeleton in an eight-step reaction sequence using the Pomeranz–Fritsch cyclization at the final step of the synthesis. (*S*)-(−)-Xylopinine[10] and several 3-mono- and 3,4-disubstituted isoquinoline derivatives[11] were prepared by Davis et al. by the addition of laterally lithiated *o*-tolunitriles[10,12] *o*-toluamide[13] and phthalide[13] to chiral *N*-*p*-toluenesulfinylimines.

2. Results and discussion

The stereoselective modification of the Pomeranz–Fritsch–Bobbitt methodology that we have realized so far involved either enantioselective synthesis employing external inductors of chirality[14] or diastereoselective synthesis, using chiral building blocks.[15] Both synthetic strategies are shown in Scheme 1.

In the enantioselective synthesis, (*S*)-(−)-salsolidine **1** and (*S*)-(−)-carnegine (*N*-methylsalsolidine) were prepared by the addition of methyllithium to prochiral imine **2** in the presence of oxazolines of type **3**. The key addition product **4** was obtained with enantioselectivity of up to 76% ee.[14] A diastereoselective approach was applied in the synthesis of both enantiomers of (*S*)-(−)- and (*R*)-(+)-*O*-methylbharat-

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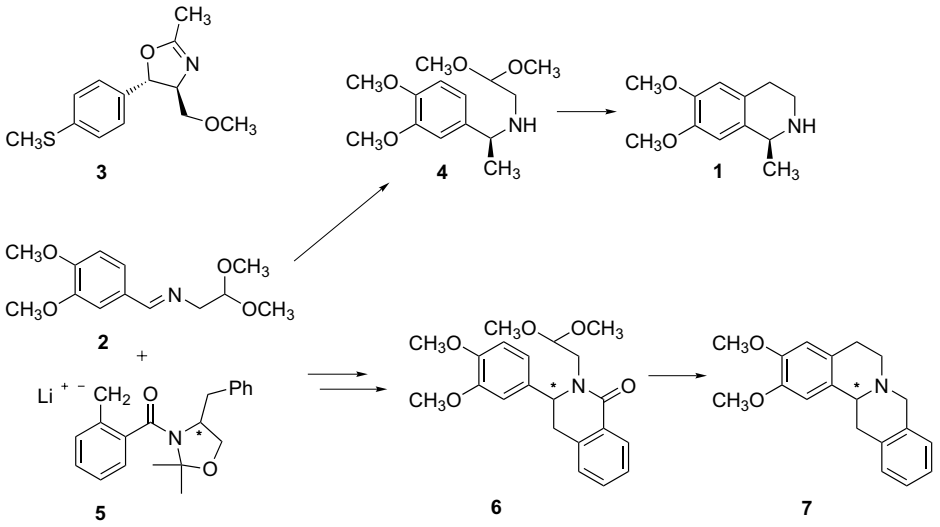
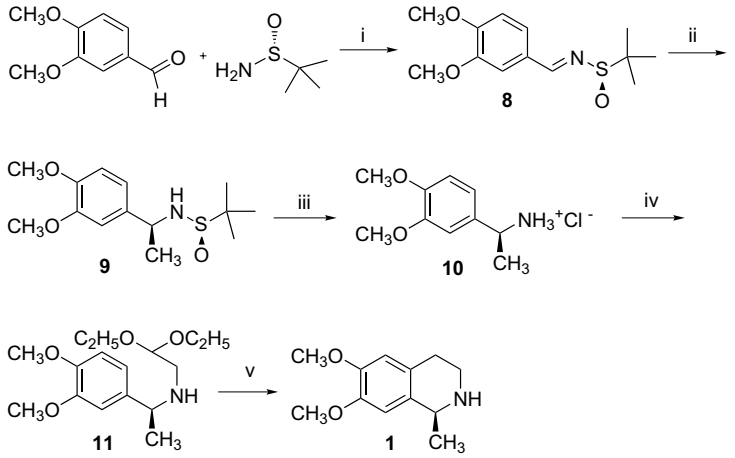


Scheme 1.

amine **7**.[15] Chiral *o*-toluamide **5**, containing either the (*S*)- or (*R*)-phenylalaninol as a component of the amide group, was laterally lithiated and added to imine **2**, to afford the corresponding isoquinolones **6**, in satisfactory yield and with good diastereoselectivity. In both cases the synthesis was completed by following the modified Bobbitt methodology[16] involving a one-pot cyclization/reduction of the intermediate aminoacetals **4** and **6**.

Herein another diastereoselective synthetic approach to the Pomeranz–Fritsch–Bobbitt methodology is also presented, which is based on the addition of methyl organometallic reagents to (*R*)-*N*-*tert*-butanesulfinylimine **8**, another building block in which the chiral auxiliary was attached to the imine nitrogen, and transformation of the resulting addition product **9** into (*S*)-(−)-salsolidine **1** (Scheme 2).

The synthesis was initiated by the condensation of 3,4-dimethoxybenzaldehyde with (*R*)-*tert*-butanesulfinylamide in the presence of Ti(O-*i*-Pr)$_4$ according to Ellmans procedure.[17] Sulfinimine **8**, obtained in 84% yield, mp 77–78 °C, $[\alpha]_D = -19.1$, was then used in the key step of the synthesis, in the addition of methyl organometallic reagents to the imine C=N double bond, during which a new stereogenic centre was created. To optimize the reaction conditions of this crucial step, several experiments were carried out with the results shown in Table 1. At first when methyllithium and methyllithium/methylaluminium reagents were used, disappointing results, with regards to the yield and diastereoselectivity, were obtained (entries 1–3). The corresponding Grignard reagent was applied in the reaction conditions following Ellman's[18] protocol (methylene chloride, −48 °C). However, no progress in the reaction was observed at this low temperature. After a gradual increase in temperature up to rt, product **9** was formed after 24 h (entry 4). At higher temperatures, between −16 and −5 °C, the reaction was completed within 5 h, but a lower yield was obtained (entry 5). When THF was used as the solvent, the addition of a Grignard reagent was completed within 4 h at ca. −27 °C and 1.5 h at +3 °C, affording sulfinamide **9** in satisfactory yield and with 97:3 and 98:2 dr, respectively (entries 6 and 7). The diastereomeric ratio



Scheme 2. Reagents and conditions: (i) Ti(O-*i*-Pr)$_4$, THF, 65 °C; (ii) CH_3MgBr, THF, −27 °C; (iii) HCl/EtOH; (iv) $(EtO)_2CHCH_2Br$, K_2CO_3, CH_3CN, 80 °C; (v) HCl, $NaBH_4$, TFA, CH_2Cl_2.

Table 1. Addition of CH_3M to imine **8**

Entry	CH_3M	Solvent	Temperature (°C)	Time (h)	*Y* (%) **9**	dr[a]
1	CH_3Li[b]	Toluene	−68	1.5	88	64:36
2	$CH_3Li/Al(CH_3)_3$[c]	Toluene	−68	1.5	38[g]	50:50
3	CH_3Li[b]	THF	−68	1.5	80	60:40
4	CH_3MgBr[d]	CH_2Cl_2	−48→rt[e]	24	70	92:8
5	CH_3MgBr[d]	CH_2Cl_2	−16→5	5	56[g]	95:5
6	CH_3MgBr[d]	THF	−27	4	89	97:3[f]
7	CH_3MgBr[d]	THF	+3	1.5	85	98:2

[a] HPLC, Chiralcel OD-H, *i*-PrOH/hexane 10:90, 0.5 ml/min, 204.5 nm.
[b] 1:1.5 molar ratio.
[c] 1:1.5:1.5 molar ratio.
[d] 10 equiv.
[e] No progress in the reaction at −48 °C.
[f] 99:1 after crystallization from *i*-Pr_2O/hexane.
[g] Starting material recovered.

could be increased to 99:1 after one crystallization from isopropyl ether/hexane, to afford **9**, with an mp 111–113 °C, $[\alpha]_D = -100.0$. The (*S*)-absolute configuration of the newly generated stereogenic centre was deduced only at the end of the synthesis, on the basis of the negative sign of the specific rotation of the synthesized (−)-salsolidine **1**, whose (*S*) configuration was already known.[19]

Removal of the *N*-sulfinyl auxiliary was carried by treatment of sulfinamide **9** in ethanol with hydrochloric acid at 0 °C, followed by stirring of the mixture at rt for 2 h.[20] The primary amine was isolated as a hydrochloride salt, **10**·HCl, in 92% yield, mp 201–203 °C (lit.[21] 214–215 °C for racemic **10**·HCl), $[\alpha]_D = -6.9$. Amine **10** was next N-alkylated with bromoacetaldehyde diethylacetal/potassium carbonate in DMF at 110 °C for 24 h to give the Pomeranz–Fritsch amine **11** as an oil, $[\alpha]_D = -29.6$, in only 62% yield. Attempts undertaken to improve the yield of this step of the synthesis by using acetonitrile at reflux as the solvent, various ratios of reagents and prolonged reaction times up to 35 h, did not improve the outcome of this step.

The synthesis was then completed by a one-pot two-step procedure in which aminoacetal **11** was treated with 6 M hydrochloric acid for 20 h, and then reduced with $NaBH_4$/trifluoroacetic acid. As a result, levorotatory (−)-salsolidine **1** was isolated in 55% yield and with 98% ee (HPLC). Its spectral characteristics corresponded to that reported in the literature.[19] Mp 228–230 °C and $[\alpha]_D = -25.4$ of its hydrochloride salt, **1**·HCl, were also in accordance with the literature data.[22,23]

3. Conclusion

In this work another approach to the diastereoselective modification of the Pomeranz–Fritsch–Bobbitt synthesis of isoquinoline alkaloids[24] is described. It is based on the addition of a methyl Grignard reagent to a chiral imine in which *N*-*tert*-butanesulfinyl auxiliary attached to the nitrogen atom controls the steric course of the reaction. By this method, the target alkaloid, (*S*)-(−)-salsolidine **1** was obtained in a five-step reaction sequence in 24% overall yield and with 98% ee.

This work also makes a contribution to the developing applicability of chiral *N*-sulfinylimines in various types of asymmetric organic synthesis.

4. Experimental

4.1. General

Melting points were determined on a Koffler block and are uncorrected. IR spectra: Perkin–Elmer 180 in KBr pellets. NMR spectra: Varian Gemini 300, with TMS as internal standard. Mass spectra (EI): instrument AMD-402. Specific rotation: Perkin–Elmer polarimeter 243B at 20 °C. Analytical HPLC: Waters HPLC system with Mallinkrodt–Baker Chiralcel OD-H column. Merck DC-Alufolien Kieselgel 60_{254} were used for TLC. Merck Kieselgel 60 (70–230 mesh) were used for column chromatography.

THF was freshly distilled from $LiAlH_4$; CH_2Cl_2 was freshly distilled from CaH_2. Ti(O-*i*-Pr)$_4$ was purchased from Fluka; *tert*-butanesulfinamide, CH_3Li and CH_3MgBr, were purchased from Aldrich and used as received.

4.1.1. (*R*)-(−)-*N*-(3,4-Dimethoxybenzylidene)-2-methylpropanesulfinamide 8. To a solution of veratraladehyde (166 mg, 1 mmol) and (*R*)-*tert*-butanesulfinamide (121 mg, 1 mmol) in anhydrous THF (2.5 ml), Ti(O-*i*-Pr)$_4$ (0.74 ml, 2.5 mmol) was added. The mixture was heated at reflux for 6 h under an argon atmosphere and left for 24 h at room temperature. Water (5 ml) was added to the solution with rapid stirring. The reaction mixture was filtered through a pad of Celite®, the filter cake washed with CH_2Cl_2 and phases then separated. The organic phase was washed with brine, dried over anhydrous Na_2SO_4 and the solvent evaporated. The crude reaction product was purified by silica gel column chromatography (CH_2Cl_2). Yield: 84%; mp 77–78 °C (*i*-Pr_2O); $[\alpha]_D = -19.1$ (*c* 0.185, CH_2Cl_2). IR (KBr), ν (cm^{-1}): 1593, 1520, 1272. ^1H NMR ($CDCl_3$): δ 1.27 (s, 9H, $C(CH_3)_3$), 3.95 (s, 6H, $2\times OCH_3$), 6.94 (d, $J = 8.52$ Hz, 1H, ArH), 7.38 (dd, $J = 2$, 8.24 Hz, 1H, ArH), 7.45 (d, $J = 2$ Hz, 1H, ArH), 8.49 (s, 1H, C*H*=N). ^{13}C NMR ($CDCl_3$): 22.53, 55.89, 56.03, 57.57, 109.69, 110.60, 125.03, 127.39, 149.38, 152.81, 161.95. EI MS *m*/*z* (%): 269 (M^+,

1), 213 (74), 165 (100), 150 (9), 137 (5). Anal. Calcd for $C_{13}H_{19}NO_3S$: (269.1) C, 57.97; H, 7.12; N, 5.20; S, 11.88. Found: C, 57.80; H, 7.18; N, 5.33; S, 12.16.

4.1.2. (R_S,*S*)-(−)-*N*-[1-(3,4-Dimethoxyphenylethyl)]-2-methylpropanesulfinamide 9. A solution of aldimine **8** (26 mg, 0.096 mmol) in anhydrous THF (2.5 ml) was cooled to −27 °C under an argon atmosphere. Methylmagnesium bromide (3 M in diethyl ether) (0.32 ml, 0.96 mmol) was then added and the reaction mixture stirred at −27 °C for 4 h, after which 20% NH_4Cl (2 ml) was added and after reaching rt the phases were separated. The aqueous phase was extracted three times with ethyl ether. The combined organic extracts were dried over anhydrous Na_2SO_4 and the solvent evaporated. The crude reaction product was purified by silica gel column chromatography (CH_2Cl_2/MeOH 0.3%). Yield: 89%, dr 97:3 (after crystallization from *i*-Pr_2O/hexane dr 99:1); mp 111–113 °C; $[\alpha]_D = -100.0$ (*c* 1.105, $CHCl_3$). IR (KBr), *v* (cm^{-1}): 3209, 2974, 1520, 1266, 1063. ^1H NMR ($CDCl_3$): δ 1.21 (s, 9H, $C(CH_3)_3$), 1.52 (d, $J = 6.59$ Hz, 3H, CH_3CH), 3.35 (d, 1H, disappears on treatment with D_2O, N*H*), 3.87 (s, 6H, $2 \times OCH_3$), 4.52 (m, 1H, $CHCH_3$; after treatment with D_2O: q, $J = 6.59$ Hz), 6.81–6.90 (m, 3H, Ar*H*). ^{13}C NMR ($CDCl_3$): δ 22.64, 25.18, 54.30, 55.44, 55.81, 55.88, 109.99, 110.91, 119.04, 135.83, 148.26, 148.86. EI MS *m*/*z* (%): 285 (M^+, 0.6), 229 (6), 213 (2), 179 (1), 165 (100), 150 (5). Anal. Calcd for $C_{14}H_{23}NSO_3$: (285.1) C, 58.92; H, 8.13; N, 4.91; S, 11.21. Found: C, 58.94; H, 8.15; N, 4.88; S, 10.95.

4.1.3. (*S*)-(−)-1-(3,4-Dimethoxyphenyl)ethylamine hydrochloride 10·HCl. Sulfinamide **9** (90 mg, 0.32 mmol) was dissolved in EtOH (2 ml) and the solution cooled to 0 °C. Concentrated HCl (94 μl) was added and the reaction mixture stirred at room temperature for ca. 2 h. Saturated K_2CO_3 (7 ml) was added, and the reaction mixture extracted twice with ethyl acetate. The combined organic extracts were dried over anhydrous Na_2SO_4 and solvents evaporated. To the crude reaction product, a 3% solution of HCl in MeOH (2 ml) was added and after 1 h the solvents were evaporated. The residue was washed a few times with ethyl acetate and dried in air to provide a white solid (65 mg). Yield: 92%; mp 201–203 °C (lit.[21] 214–215 °C for racemic **10**·HCl), $[\alpha]_D = -6.9$ (*c* 0.7, MeOH). IR (KBr), *v* (cm^{-1}): 3000, 2696, 1514, 1262. ^1H NMR (DMSO-d_6): δ 1.49 (d, $J = 6.8$ Hz, 3H, $CHCH_3$), 3.75 (s, 3H, OCH_3), 3.78 (s, 3H, OCH_3), 4.31 (q, $J = 6.8$ Hz 1H, $CHCH_3$), 6.98–6.99 (m, 2H, Ar*H*), 7.22 (d, 1H, Ar*H*), 8.44 (s, 1H, disappears on treatment with D_2O, N*H*). ^{13}C NMR (DMSO-d_6): 20.77, 49.84, 55.58, 55.65, 110.89, 111.58, 119.07, 131.76, 148.69, 148.74. EI MS *m*/*z* (%): 181 (M^+−HCl, 18), 166 (100), 150 (15), 139 (14), 105 (5). Anal. Calcd for $C_{10}H_{15}NO_2 \cdot HCl \cdot 1/3H_2O$: (223.5) C, 53.68; H, 7.51; N, 6.26. Found: C, 53.44; H, 7.74; N, 6.17.

4.1.4. (*S*)-(−)-*N*-(2,2-Diethoxyethyl)-1-(3,4-dimethoxyphenyl)ethylamine 11. To a solution of amine **10** (100 mg, 0.52 mmol), prepared from the amine hydrochloride **10**·HCl by treatment with 20% NaOH, in DMF (3 ml), anhydrous K_2CO_3 (66 mg, 0.48 mmol) was added followed by 2-bromo-1,1-diethoxyethane (0.7 mmol, 0,1 ml). The reaction mixture was heated at 110 °C for 24 h under the argon atmosphere. During that time an additional amount of K_2CO_3 (66 mg, 0.48 mmol) and 2-bromo-1,1-diethoxyetane (0.7 mmol, 0.1 ml) were added. After completion of the reaction, the mixture was poured into ice and after reaching rt. the mixture was extracted three times with ethyl ether. The organic extracts were dried and the solvent was evaporated to afford crude **11**, which after purification by silica gel column chromatography yielded oily amine **11**. Yield: 62%; $[\alpha]_D = -29.6$ (*c* 0.8, $CHCl_3$); IR (KBr), *v* (cm^{-1}): 2974, 1517, 1262, 1139. ^1H NMR ($CDCl_3$): δ 1.15–1.21 (m, 6H, $2 \times CH_2CH_3$), 1.36 (d, $J = 6.59$ Hz, 3H, CH_3CH), 1.60 (br s, 1H, disappears on treatment with D_2O, N*H*), 2.53–2.67 (dABq, $J =$ ca. 5, 12.1 Hz, 2H, CH_2NH), 3.44–3.76 (m, 5H, $2 \times CH_3CH_2$, $CH(OEt)_2$), 3.87 (s, 3H, OCH_3), 3.89 (s, 3H, OCH_3), 4.58 (t, $J = 5.76$ Hz, 1H, $CHCH_3$), 6.82 (m, 2H, Ar*H*), 6.90 (s, 1H, Ar*H*). ^{13}C NMR ($CDCl_3$): δ 15.33, 24.22, 49.78, 55.84, 57.99, 62.01, 62.52, 101.91, 109.34, 110.86, 118.85, 137.48, 147.92, 149.03. EI MS *m*/*z* (%): 297 (M^+, 2.9), 281 (6), 236 (2), 206 (11), 180 (21), 165 (100), 150 (5), 103 (32). Anal. Calcd for $C_{16}H_{27}NO_4$: (297.2) C, 64.60; H, 9.16; N, 4.71. Found: C, 64.60; H, 9.32; N, 4.75.

4.1.5. (*S*)-(−)-Salsolidine 1. A solution of aminoacetal (40 mg, 0.13 mmol) and 5 M aqueous HCl (1.3 ml) were stirred overnight at rt. The mixture was basified with 20% aqueous NaOH and extracted three times with dichloromethane. The combined organic extracts were dried over anhydrous Na_2SO_4 and evaporated to give a solid (27 mg), which was dissolved in dichloromethane (6 ml) and cooled to 0 °C. To this solution $NaBH_4$ (60 mg, 1,6 mmol) was added along with the slow introduction of trifluoroacetic acid (0.93 ml, 12.2 mmol) in dichloromethane (2 ml). The reaction was stirred at rt for 24 h, then the solvent was removed under reduced pressure. The resultant precipitate was dissolved in water (4 ml), basified with 20% aqueous NaOH and extracted three times with dichloromethane. The organic extract was dried over anhydrous Na_2SO_4 and evaporated yielding levorotatory (−)-salsolidine **1** as an oil. Yield: 55%, ee 98% (HPLC). This was dissolved in methanolic HCl to give crystalline hydrochloride salt, **1**·HCl; mp 230–232 °C, $[\alpha]_D = -25.9$ (*c* 0.32, EtOH), lit.[22] mp 235–236 °C, $[\alpha]_D^{22} = -18$ (*c* 1, EtOH), $[\alpha]_D^{23} = -26.6$ (*c* 4, EtOH). ^1H NMR (DMSO-d_6): δ 1.55 (d, $J = 6.59$ Hz, 3H, $CHCH_3$), 2.90 (m, 2H, CH_2CH_2NH), 3.23 (m, 1H, CH*H*NH), 3.38 (m, 1H, C*H*HNH), 3.71 (s, 6H, $2 \times OCH_3$), 4.43 (q, $J = 6.59$ Hz, 1H, $CHCH_3$), 6.76 (s, 1H, Ar*H*), 6.80 (s, 1H, Ar*H*).

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