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TETRAHEDRON: *ASYMMETRY*

# Facile synthesis of homochiral derivatives of 10-bornane sulfinates, sulfinamides and sulfinimines

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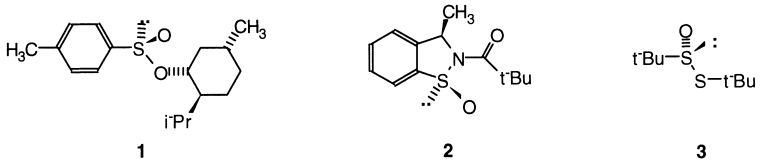
**Abstract**

A convenient route for the synthesis of enantiopure 10-bornanesulfinic acid derivatives from (1*S*)-camphorsulfonyl chloride is described. An interesting epimerization at sulfur in five-membered sultines is observed, leading to diastereoisomerically pure ($S_S$)-10-isobornyl sulfinates. A large difference in solubility in organic solvents allowed ($R_S$)-2-oxo-10-bornanesulfinamide to be obtained as the major diastereoisomer. Cyclization of 2-oxo-10-bornanesulfinamides gave five-membered sulfinimines. 

## 1. Introduction

There is great interest in the asymmetric synthesis using chiral organosulfur compounds.[1] Recent years have shown that among them, optically active sulfinimines are of great value for the stereoselective synthesis of amino compounds.[2] The most convenient precursors for the preparation of homochiral sulfinimines are derivatives of sulfinic acids. Diastereoisomerically pure menthyl esters,[3] especially (−)-menthyl *p*-toluenesulfinate **1**, have found many applications (Scheme 1). Some other useful reagents have been discovered, e.g. derivatives of benzisothiazole *S*-oxide[4] **2** or (*R*)-(+)-*t*-butyl *t*-butanethiosulfinate[5] **3**. The latter especially seems to have superior properties. Nevertheless, the search for new reagents is still an important task for organic chemists.




**1** **2** **3**

Scheme 1.

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It has been shown in recent publications that the 10-bornylsulfinyl group serves as a useful chiral auxiliary.[6] For example, alkenyl bornyl sulfoxides **4** were successfully used as dienophiles in Diels–Alder cycloadditions (Scheme 2).[6b] For this purpose, optically active sulfoxides were obtained by highly stereoselective oxidation of corresponding bornyl sulfides with *m*-CPBA. However, the high stereoselectivity was observed only in the presence of a 2-*exo*-hydroxy group in the bornyl moiety.[6b]


**4** **5**

Scheme 2.

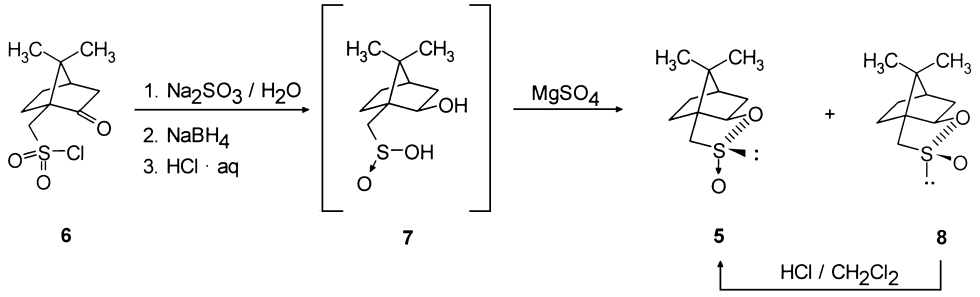
A different approach was applied in our laboratory. A number of homochiral 10-isobornyl sulfoxides were prepared by addition of organometallic reagents to 10-isobornyl sulfinate **5**.[7] This methodology was also very useful for the preparation of 10-bornylsulfinamide, a key compound for the synthesis of sulfinimines.[8] Previously, sultine **5** was prepared by stereoselective reduction of the corresponding sultone with $LiAlH_4$ in THF.[9] Since the preparation of camphorsultone is rather troublesome on a large scale, we developed a facile and short synthesis of sultine **5** which can be carried out in one-pot and in aqueous solution.

## 2. Results and discussion

The camphorsulfonyl chloride was reduced to camphorsulfinic acid with sodium sulfite in water. The resulting solution of sodium sulfinate was subsequently reduced with sodium borohydride. The main product of this reaction after aqueous acidic workup consisted of 2-hydroxy-10-bornanesulfinic acid **7** which slowly cyclized to sultines **5** and **8** (Scheme 3). The ratio of diastereoisomers varies from 1.5:1 to 1.8:1. Both stereoisomers can be separated by column chromatography, however, sultine **8** was easily converted to sultine **5** by epimerization at sulfur induced by HCl. This process is quite fast but can be monitored by TLC or NMR. Within 1 h after addition of HCl in $CH_2Cl_2$ at rt more than 90% of the sultine **8** was already epimerized, and a few hours later no trace of sultine **8** could be detected. The only side product (ca. 10%) of the above mentioned procedure arose from non-stereoselective reduction of the carbonyl group by $NaBH_4$ and was tentatively assigned as 2-*endo*-hydroxycamphorsulfinic acid. The amount of this unwanted compound can be decreased by reduction of camphorsulfinic acid with $NaBH_4$ in methanol at low temperatures, instead of using water solution. However, this highly polar impurity is easily removed by sodium bicarbonate solution, so an aqueous one-pot procedure seems to be more facile. The overall yield of **5** from camphorsulfonyl chloride was 67%.

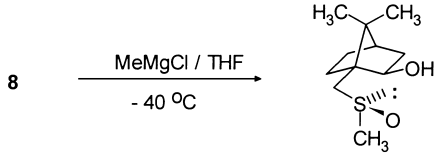
The configuration at sulfur in **5** was previously determined as *S*.[7] In a similar way, the configuration of **8** was established by reaction with methylmagnesium chloride (Scheme 4), which proceeds with inversion at sulfur.[10] Known ($S_S$)-methylisobornyl sulfoxide[11] **9** was obtained proving the $R_S$ configuration in **8**.

The interesting conversion of **8** to sultine **5** originates from the higher stability of sultine **5** caused by stereoelectronic effects. The pseudo-axial sulfinyl oxygen atom in **5** is close to an antiperiplanar position with respect to a lone pair on the oxygen at C2 giving extra stabilization (ca. 2.0 kcal/mol for six-membered sultines[12]). This conformation is confirmed by $^1H$ NMR spectra which reveal an H-2 atom




6 7 5 8

Scheme 3.

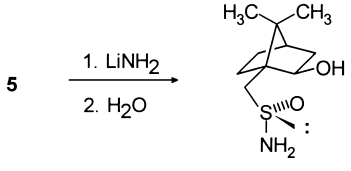



8 9

Scheme 4.

highly deshielded (*syn*-axial effect)[12] by the sulfinyl oxygen atom (5.0 ppm in sultine **5** versus 4.3 ppm in **8**). Sultine **8** is relatively stable and can be stored for months in a refrigerator. The recently described[13] epimerization of sultines with iodine in benzene does not occur with **8**. Isomerization in the presence of $H_2{}^{18}O$ showed that there is no oxygen exchange in sultine **5**.

Previously we have described the synthesis of 10-isobornylsulfinamide by addition of LHMDS to sultine **5**.[8] The same product may be synthesized conveniently on a large scale and in good yield with lithium amide in liquid ammonia (Scheme 5).

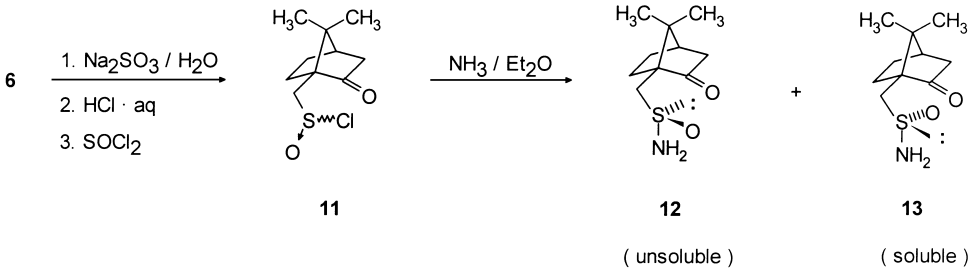



5 10

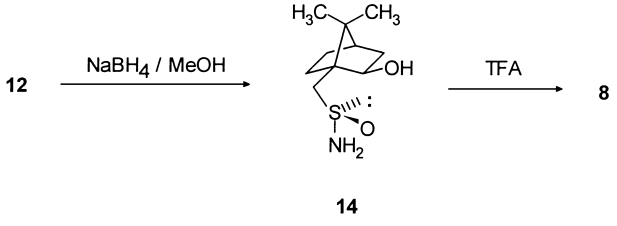
Scheme 5.

Sultine **8** would be a very desirable starting material for bornanesulfinamides with an *R* configuration at the sulfur atom. However, the necessity of chromatographic separation of this diastereoisomer excludes the above mentioned procedure for use on a larger scale. Therefore, we found that the easily accessible sulfinamide **12** is a very convenient chiral precursor. The starting material was, as previously, camphorsulfonyl chloride, which was reduced with sodium sulfite to camphorsulfinic acid. The crude acid was converted to camphorsulfinyl chloride with thionyl chloride. The $^1$H NMR spectrum of this product revealed the presence of two diastereoisomers in a ratio of 1:3.4. Subsequent reaction with ethereal ammonia solution gave a mixture of two diastereoisomeric sulfinamides **12** and **13** (Scheme 6).

Fortunately, the major diastereoisomer was hardly soluble in organic solvents in contrast to the minor diastereoisomer. Crystallization from methanol gave (*R*)-sulfinamide **12** in 45% yield from **6**. The configuration of this product was established by reduction of the carbonyl function with $NaBH_4$ (Scheme 7). Spectral data, which were different from **10**, indicated that the configuration at the sulfur atom is *R*. Furthermore, sulfinamide **14** can be quantitatively converted to **8** in the presence of TFA.

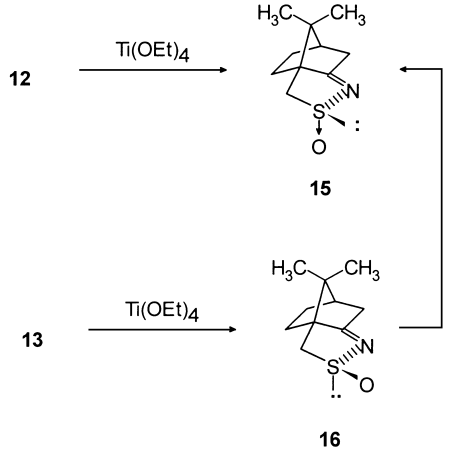



Scheme 6.




Scheme 7.

The diastereoisomerically pure sulfinamides **12** and **13** are very interesting starting materials for the synthesis of sulfinimines (Scheme 8). For example, condensation in the presence of $Ti(OEt)_4$[14] gives sulfinimines **15** and **16**. To our knowledge these are the first cyclic *N*-sulfinyl imines prepared to date. Interestingly, sulfinimine **16** slowly epimerizes to **15**, similarly to sultine **8**.




Scheme 8.

## 3. Conclusion

This methodology is a convenient way to synthesize homochiral derivatives of 10-bornanesulfinic acid. Since both enantiomers of camphorsulfonic acid are available, the presented method allows the preparation of all stereoisomers of the title compounds. The isomerization of ($R_S$)-sultine **8** to ($S_S$)-sultine **5** is described.

## 4. Experimental

### 4.1. General

NMR spectra were recorded at 500 or 200 MHz ($^1$H). All spectra were referenced to residual solvent peak (chloroform 7.26 and 77.0 ppm, DMSO 2.49 and 39.5 ppm for $^1$H and $^{13}$C, respectively). THF was distilled from sodium in the presence of sodium benzophenone ketyl. Camphorsulfonyl chloride was prepared from (1*S*)-(+)-camphorsulfonic acid monohydrate (Merck) by a known procedure[15] and was immediately used without drying. The amount of water was assumed as 12%. Commercially available, dry reagent may be used instead. Melting points are uncorrected. TLC plates were developed in $KMnO_4$ solution or phosphormolybdic acid.

### 4.2. (1S,5R,7R,S$_S$)-10,10-Dimethyl-4-oxo-3-thiatricyclo[5.2.1.0$^{1,5}$]decane-3-oxide **5**

(+)-10-Camphorsulfonyl chloride (9.5 g, 38 mmol) was added to the solution of sodium bicarbonate (6.37 g, 76 mmol) and sodium sulfite (4.78 g, 38 mmol) in water (95 mL) at rt. The mixture was stirred vigorously until the solution became clear and then kept for a further 2 h. The reaction mixture was transferred to a 1 L beaker immersed in an ice bath and cooled to 10°C. Sodium borohydride (6.5 g, 171 mmol) was added in one portion. The stirring at 12°C was continued for 20 min and the reaction mixture was acidified carefully (hood) with 10% aqueous HCl (70 mL). The temperature was kept below 25°C by addition of crushed ice to the reaction mixture from time to time. Concentrated HCl (12 mL), $CH_2Cl_2$ (40 mL) were added and enough water to dissolve the white precipitate. The reaction mixture was extracted four times with $CH_2Cl_2$ (40 mL). Concentrated HCl (1 mL) was added to combined organic layers and the solution was dried with $MgSO_4$ for at least 16 h. The solution was filtered and evaporated to give 6.6 g of crude sultine **5**. This product was dissolved in hexane (75 mL) and washed twice with saturated $Na_2CO_3$ solution (12 mL) and once with water. The organic layer was dried, filtered and evaporated. The resulting white solid was recrystallized from hexane to give 5.0 g (67%) of pure **5**. Mp 151–153°C. Lit.[9] mp 145–148°C. $[\alpha]_D^{25}$=+35.8 (c 1.33, $CHCl_3$). Lit.[7] $[\alpha]_D^{20}$=+35.0 (c 2.26, $CHCl_3$).

### 4.3. (1S,5R,7R,S$_S$)-10,10-Dimethyl-4-oxa-3-thiatricyclo[5.2.1.0$^{1,5}$]decane-3-oxide **8**

This compound was obtained by chromatographic separation (silica gel, $CH_2Cl_2$) of sultines obtained as described above, without epimerization with conc. HCl (after at least 16 h drying with $MgSO_4$). Sultine **8** has higher $R_f$ value than sultine **5**. A white solid: mp ca. 168°C. $[\alpha]_D^{22}$=+2.5 (c 1.0, $CHCl_3$). IR (KBr) 1046, 1143, 2957 cm$^{-1}$. $^1$H NMR ($CDCl_3$) δ 0.93 (s, 3H), 1.1–1.3 (m, 2H), 1.24 (s, 3H), 1.62–2.02 (m, 4H), 2.28–2.40 (m, 1H), 2.68 and 3.24 (AB, *J*=13.9 Hz, 2H), 4.31 (dd, *J*=3.7, 7.9 Hz, 1H). $^{13}$C NMR ($CDCl_3$) δ 20.2, 22.0, 26.5, 30.3, 37.5, 46.3, 47.4, 56.4, 58.9, 95.2. HR LSIMS calcd for $C_{10}H_{17}O_2S$ $(M+H)^+$ 201.0949. Found 201.0945. Anal. calcd for $C_{10}H_{16}O_2S$: C, 59.97; H, 8.05; S, 16.01. Found: C, 60.03; H, 8.16; S, 16.13.

### 4.4. (1S,2R,4R,S$_S$)-7,7-Dimethyl-1-[(methylsulfinyl)methyl]bicyclo[2.2.1]heptan-2-ol **9**

Assignment of the configuration of sultine **8**: To a solution of **8** (200 mg, 1.0 mmol) in THF (6 mL) was added a solution of methylmagnesium chloride (0.5 mL, 1 mmol) in THF at −40°C. The reaction mixture was stirred for 30 min and quenched with saturated $NH_4Cl$ solution. The organic layer was separated and the aqueous phase was extracted twice with $CH_2Cl_2$ (5 mL). The combined organic extracts were dried

with $MgSO_4$ and evaporated. The solid residue was recrystallized from hexane to give 163 mg (75%) of colorless crystals. Mp 80–82°C, $[\alpha]_D^{22}$=+31.5 (c 1.16, $CHCl_3$). Lit.[11] mp 79–81°C, $[\alpha]_D^{26}$=+31.7 (c 1.0, $CHCl_3$). $^1$H NMR data are in agreement with those previously reported.[11] $^{13}$C NMR ($CDCl_3$) δ 19.8, 20.4, 27.0, 30.8, 38.3, 39.8, 44.8, 47.9, 51.3, 55.3, 76.7.

### *4.5. (1*S,*2*R,*4*R,S$_S$*)-2-Hydroxy-10-bornanesulfinamide **10***

To the suspension of lithium amide, prepared by addition of small lithium pieces (215 mg, 30.7 mmol) to liquid ammonia (ca. 20 mL) in the presence of a catalytic amount of $FeCl_3 \cdot 6H_2O$, was added at −45°C a solution of sultine **5** (0.75 g, 3.75 mmol) in THF (10 mL). The reaction mixture was stirred at −60°C for 3 h and carefully quenched with solid $NH_4Cl$ (4.6 g). The ammonia was evaporated and the residue was dissolved in 15 mL of $H_2O$ and 25 mL of $CH_2Cl_2$. The organic layer was separated and the aqueous solution was extracted twice with $CH_2Cl_2$. The combined organic extracts were dried and evaporated to give a white solid (730 mg). Crystallization from a mixture of chloroform and hexane gave 620 mg (76%) of white crystals. Mp 114–127°C. Lit.[8] mp 123–129°C. Spectral data are identical with those previously reported.[8]

### *4.6. (1*S,*4*R,R$_S$*)-2-Oxo-10-bornanesulfinamide **12***

Camphorsulfonyl chloride (wet, 10.85 g, ca. 38 mmol) was added to a vigorously stirred solution of sodium sulfite (5.05 g, 40 mmol) and sodium hydrogen carbonate (6.7 g, 80 mmol) in water (100 mL). The stirring was continued until the mixture became homogeneous and then for a further 2 h. The reaction mixture was acidified first with 10% HCl (20 mL) and then with 15 mL of conc. HCl. After cooling, the mixture was extracted once with $CH_2Cl_2$ (30 mL). After separation of the organic layer, the aqueous phase was saturated with sodium chloride and extracted three times with $CH_2Cl_2$ (25 mL). The combined organic layers were dried with $MgSO_4$ for 1 h and evaporated. Benzene (15 mL) was added and the solution was again evaporated to give an oil (8.4 g), which solidified after 30 min. 10-Camphorsulfinic acid was used immediately in the next step. Thionyl chloride (19 g, 160 mmol) was added to the acid at rt. The reaction mixture was stirred for 2 h. The excess of $SOCl_2$ was removed in vacuo at rt. The residue was dissolved in 5 mL of benzene and the solvent evaporated. This procedure was repeated to remove traces of $SOCl_2$. The crystalline residue, consisting of two diastereoisomers of 10-camphorsulfinic chloride (1:3.4) as revealed by $^1$H NMR, was dissolved in diethyl ether (30 mL) and added at −30°C to a saturated solution of ammonia in $Et_2O$ (150 mL). The mixture was warmed to rt and stirred for 30 min. Water (25 mL) was added and the reaction mixture was filtered. The ‘cake’ was washed three times with water (10 mL). The precipitate was dried under vacuum at rt to give 5.2 g of a white solid. Crystallization from methanol gave 3.1 g (1st crop) and 0.55 g (2nd crop) of colorless crystals (45% from 10-camphorsulfonyl chloride) of sulfinamide **12**. A white solid: mp 166–180°C (MeOH). $[\alpha]_D^{25}$=+1.6 (c 0.82, MeOH). IR (KBr) 1035, 1053, 1726, 3311 cm$^{-1}$. $^1$H NMR (DMSO-$d_6$) δ 0.81 (s, 3H), 0.96 (s, 3H), 1.32 (m, 2H), 1.81–1.91 (m, 2H), 2.01–2.14 (m, 2H), 2.28–2.34 (m, 1H), 2.67 and 2.76 (AB, *J*=13.5 Hz, 2H), 5.72 (br, 2H). $^{13}$C NMR (DMSO-$d_6$) δ 19.6, 19.7, 25.9, 26.5, 42.4, 42.5, 47.7, 55.4, 58.2, 216.1. MS (EI): *m/z*=197 (5), 180 (5), 81 (100). HR LSIMS calcd for $C_{10}H_{18}NO_2S$ $(M+H)^+$ 216.1058. Found 216.1078.

The mother liquid was evaporated to dryness and extracted several times with boiling $Et_2O$. Combined etheral extracts were evaporated to give 1.1 g (13%) of sulfinamide (1*S*,4*R*,*S*$_S$)-2-oxo-10-bornanesulfinamide **13**. A white solid: mp 118–123°C. $[\alpha]_D^{25}$=−41.1 (c 1.16, $CHCl_3$). IR (KBr) 1045, 1071, 1732, 3268 cm$^{-1}$. $^1$H NMR ($CDCl_3$) δ 0.87 (s, 3H), 1.01 (s, 3H), 1.35–1.55 (m, 1H), 1.65–2.15

(m, 5H), 2.38 (ddd, *J*=2.6, 4.5, 18.6 Hz, 1H), 2.63 and 3.09 (AB, *J*=13.6 Hz, 2H), 4.39 (br, 2H). $^{13}$C NMR ($CDCl_3$) δ 19.2, 19.6, 25.7, 26.9, 42.3, 42.6, 48.2, 55.2, 58.2, 216.5. Anal. calcd for $C_{10}H_{17}NO_2S$: C, 55.78; H, 7.96; N, 6.51; S, 14.89. Found: C, 55.66; H, 7.95; N, 6.72; S, 14.78.

### *4.7. (1S,2R,4R,R$_S$)-2-Hydroxy-10-bornanesulfinamide* **14**

To the suspension of sulfinamide **12** (217 mg, 1 mmol) in methanol (4 mL) was added sodium borohydride (128 mg, 3.4 mmol) in four portions at rt. The reaction mixture became clear. The stirring was continued for 3 h and the solution was evaporated. Water (10 mL) was added and the reaction mixture was extracted three times with $CH_2Cl_2$ (5 mL), dried with $MgSO_4$ and evaporated to give 196 mg (90%) of white crystals. An analytical sample was obtained by crystallization from $CH_2Cl_2$ and hexane.

A white solid: mp 153–158°C ($CH_2Cl_2$ and hexane). $[\alpha]_D^{22}$=+30.1 (c 1.35, $CHCl_3$). IR (KBr) 1032, 2941, 3349 cm$^{-1}$. $^1$H NMR ($CDCl_3$) δ 0.83 (s, 3H), 1.05–1.20 (m, 1H), 1.09 (s, 3H), 1.4–1.9 (m, 6H), 2.68 and 3.27 (AB, *J*=12.8 Hz, 2H), 3.82 (d, *J*=3.4 Hz, 1H), 4.02 (ddd, *J*=7.0, 3.4, 3.6 Hz, 1H), 4.29 (br, 2H). $^{13}$C NMR ($CDCl_3$) δ 20.0, 20.4, 27.1, 30.6, 38.5, 44.6, 48.1, 50.5, 57.3, 76.4. Anal. calcd for $C_{10}H_{19}NO_2S$: C, 55.27; H, 8.81; N, 6.45; S, 14.75. Found: C, 55.03; H, 9.10; N, 6.17; S, 14.78.

### *4.8. (1S,7R,R$_S$)-10,10-Dimethyl-3-thia-4-azatricyclo[5.2.1.0$^{1,5}$]dec-4-ene 3-oxide* **15**

To the suspension of sulfinamide **12** (380 mg, 1.75 mmol) in THF (8 mL) was added titanium tetraethoxide (616 mg, 2.7 mmol) and the mixture was refluxed under argon for 16 h. The reaction mixture was cooled to rt and 8 mL of saturated aqueous KCl solution was added. The mixture was filtered through Celite and the precipitate was washed several times with $CH_2Cl_2$. The organic phase was dried and evaporated to give white crystals which were recrystallized from $CH_2Cl_2$ and hexane. Yield 250 mg (72%). A white solid: mp 153–154°C ($CH_2Cl_2$ and hexane). $[\alpha]_D^{27}$=+41.3 (c 1.09, $CHCl_3$). IR (KBr) 1077, 1629, 2951 cm$^{-1}$. $^1$H NMR ($CDCl_3$) δ 0.78 (s, 3H), 1.00 (s, 3H), 1.40–1.60 (m, 1H), 1.90–2.35 (m, 5H), 2.45 (1/2AB, *J*=13.7 Hz, 1H), 2.61 (ddd, *J*=2.4, 4.5, 19.0 Hz, 1H), 2.75 (1/2AB, *J*=13.7 Hz, 1H). $^{13}$C NMR ($CDCl_3$) δ 19.0, 19.6, 26.8, 31.4, 36.0, 46.9, 47.7, 50.9, 72.1, 205.5. HR LSIMS calcd for $C_{10}H_{16}ONS$ $(M+H)^+$ 198.0953. Found 198.0950.

### *4.9. (1S,7R,S$_S$)-10,10-Dimethyl-3-thia-4-azatricyclo[5.2.1.0$^{1,5}$]dec-4-ene 3-oxide* **16**

Yield 59%, mp 120–126°C. $[\alpha]_D^{25}$=−274.8 (c 1.25, acetone). IR (KBr) 1087, 1636, 2962 cm$^{-1}$. $^1$H NMR ($CDCl_3$) δ 0.98 (s, 3H), 1.03 (s, 3H), 1.30–1.60 (m, 2H), 1.90–2.12 (m, 3H), 2.11 (d, *J*=18.8 Hz, 1H), 2.26 (dd, *J*=4.3, 4.3 Hz, 1H), 2.37 (d, *J*=13 Hz, 1H), 2.78 (ddd, *J*=2.1, 4.3, 18.8 Hz, 1H), 3.39 (d, *J*=13 Hz, 1H). $^{13}$C NMR ($CDCl_3$) δ 18.8, 19.6, 26.5, 29.0, 35.4, 46.4, 46.5, 56.4, 71.7, 197.5. MS (EI): *m/z*=197 (9, $M^+$), 180 (10), 134 (100). Anal. calcd for $C_{10}H_{15}NOS$: C, 60.88; H, 7.66; N, 7.10; S, 16.25. Found: C, 60.78; H, 7.80; N, 7.03; S, 16.41.

### *4.10. Sultine* **8** *by cyclization of sulfinamide* **14**

To the solution of sulfinamide **14** (36 mg, 0.16 mmol) in $CH_2Cl_2$ (2 mL) was added trifluoroacetic acid (0.05 mL). After 2 minutes, water (2 mL) and saturated $NaHCO_3$ solution (2 mL) were added to the cloudy solution. The reaction mixture was stirred vigorously for a few minutes. The organic phase was separated and the aqueous layer was extracted twice with $CH_2Cl_2$. The organic extracts were dried and evaporated to give 30 mg (91%) of pure **8**.

## Acknowledgements

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