Enantioselective Organocatalyzed α-Sulfenylation of Aldehydes

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General. The $^1$H NMR and $^{13}$C NMR spectra were recorded at 400 MHz and 100 MHz, respectively. The chemical shifts are reported in ppm relative to CHCl$_3$ ($\delta = 7.26$) for $^1$H NMR and relative to the central CDCl$_3$ resonance ($\delta = 77.0$) for $^{13}$C NMR. Flash chromatography (FC) was carried out using Merck silica gel 60 (230-400 mesh). Optical rotation values were measured on a Perkin-Elmer 241 polarimeter. NMR data of known compounds are in agreement with literature values.

Materials. Commercially available aldehydes and organocatalysts were used without further purifications. The catalysts 4c,d were synthesized according to literature procedures. All solvents were of p.a. quality and used without further purification.

1-Benzylsulfanyl-[1,2,4]triazole 2e: 20 mmol of sulfuryl chloride were added dropwise to a solution of dibenzyl disulfide (20 mmol) in CH$_2$Cl$_2$ (20 mL). After 15 min the resulting solution was added dropwise to a second solution of 1,2,4-triazole (50 mmol) and NET$_3$ (44 mmol) in CH$_2$Cl$_2$ (20 mL). The solvent was removed in vacuo at 20°C and the product
was extracted from the crude mixture using pentane (2 × 100 mL) and pentane/CH₂Cl₂ 30 vol% (100 mL). The product was isolated in 62% yield by quick FC (gradient pentane/Et₂O 1/1 to Et₂O) in order to minimize decomposition. The product was stored under inert atmosphere at 0°C. ¹H NMR δ 8.03 (s, 1H, CH₃), 7.63 (s, 1H, CH₂), 7.27 (m, 3H, ArH), 7.03 (m, 2H, ArH), 4.22 (s, 2H, SCH₂Ph); ¹³C NMR δ 154.1, 151.4, 134.2, 128.9, 128.8, 128.1, 45.2.

General procedure for the preparation of catalysts 4e-h: TMSOTf (10 mmol, 1.3 eq.) was added at 0°C to a solution of the appropriate amino alcohol (7.6 mmol) and NEt₃ (10 mmol, 1.3 eq.) in CH₂Cl₂ (50 mL). The reaction was then allowed to reach ambient temperature and was stirred for 1 h until full conversion of the starting material was confirmed by TLC analysis. The reaction was quenched with water. The product was extracted 3 times with CH₂Cl₂ and dried over Na₂SO₄. After evaporation of the solvent the catalysts were isolated by FC in near quantitative yields.

(S)-2-(Diphenyl-trimethylsilanyloxy-methyl)-pyrrolidine 4e: The title compound was prepared following the general procedure. ¹H NMR δ 7.36-7.30 (m, 10H, ArH), 4.86 (br, 1H, NH), 4.50 (t, 1H, J = 7.2, NCH₂), 3.10 (dt, 1H, J = 10.7, 6.7, NCH₂), 2.84 (dt, 1H, J = 10.7, 7.0, NCH₂), 2.05-1.99 (m, 1H, CH₂), 1.96-1.82 (m, 2H, CH₂), 1.58-1.51 (m, 1H, CH₂), -0.01 (s, 9H, Si(CH₃)₃); ¹³C NMR δ 143.7, 143.7, 128.3, 128.1, 128.0, 127.9, 127.8, 127.7, 82.4, 66.4, 47.1, 27.3, 24.6, 1.8; [α]₀ = -30.0 (c = 1.0, CH₂Cl₂).
(S)-2-(Dinaphtalen-2-yl-trimethylsilanyloxy-methyl)-pyrrolidine 4f: The title compound was isolated following the general procedure. $^1$H NMR $\delta$ 8.07 (s, 1H, ArH), 8.01 (s, 1H, ArH), 7.97-7.66 (m, 6H, ArH), 7.54-7.24 (m, 6H, ArH), 4.51 (br, 1H, NCH), 2.96 (br, 1H, NCH$_2$), 2.75 (br, 1H, NCH$_2$), 2.05-1.55 (m, 5H, CH$_2$, NH), -0.05 (s, 9H, Si(CH$_3$)$_3$; $^{13}$C NMR $\delta$ 142.2, 141.9, 132.7, 132.5, 128.8, 128.4, 127.6, 127.5, 127.4, 126.8, 126.5, 126.3, 126.2, 126.1, 125.9, 83.1, 65.8, 47.0, 27.5, 24.9, 2.2; $[\alpha]_D = -62.6$ (c = 0.9, CH$_2$Cl$_2$).

(S)-2-[Bis-(3-5-dimethyl-phenyl)-trimethylsilanyloxy-methyl]-pyrrolidine 4g: The title compound was prepared following the general procedure. $^1$H NMR $\delta$ 7.00 (s, 2H, ArH), 6.94 (s, 2H, ArH), 6.84 (s, 1H, ArH), 6.81 (s, 1H, ArH), 3.97 (t, 1H, J = 7.6, NCH), 2.88-2.76 (m, 2H, NCH$_2$), 2.27 (s, 6H, CH$_3$), 2.26 (s, 6H, CH$_3$), 1.90 (br, 1H, NH), 1.61-1.52 (m, 3H, CH$_2$), 1.48-1.39 (m, 1H, CH$_2$), -0.10 (s, 9H, Si(CH$_3$)$_3$); $^{13}$C NMR $\delta$ 146.6, 145.7, 136.8, 128.4, 128.2, 125.9, 125.3, 83.2, 65.4, 47.1, 27.5, 25.1, 21.5, 21.5, 2.2; $[\alpha]_D = -73.3$ (c = 1.5, CH$_2$Cl$_2$).

(S)-2-[Bis-(3-5-bistrifluoromethyl-phenyl)-trimethylsilanyloxy-methyl]-pyrrolidine 4h: The title compound was prepared following the general procedure. $^1$H NMR $\delta$ 8.04 (s, 2H, ArH), 7.84 (s, 1H, ArH), 7.83 (s, 1H, ArH), 7.79 (s, 2H, ArH), 4.25 (t, 1H, J = 7.2, NCH), 2.93 (dt, 1H, J = 10.0, 6.8, NCH$_2$), 2.59 (dt, J = 10.0, 6.0, NCH$_2$), 1.75 (br, 1H, NH), 1.75-1.68 (m, 1H, CH$_2$), 1.60-1.42 (m, 2H, CH$_2$), 1.17-1.08 (m, 1H, CH$_2$), -0.08 (s, 9H, Si(CH$_3$)$_3$); $^{13}$C NMR $\delta$ 148.7, 146.7, 131.8 (q, $^2$J$_{C,F} = 33.4$), 131.1 (q, $^2$J$_{C,F} = 33.4$), 128.9 (q,
$^3J_{C,F} = 3.1), 128.3 \ (q, \ ^3J_{C,F} = 3.1), 123.6 \ (q, \ ^1J_{C,F} = 271), 128.1 \ (q, \ ^1J_{C,F} = 271), 121.9 \ (\text{hept}, \ ^3J_{C,F} = 3.8), 121.7 \ (\text{hept}, \ ^3J_{C,F} = 3.8), 82.6, 64.5, 47.5, 27.8, 25.5, 2.0; [\alpha]_D = -6.8 \ (c = 1.2, \ CH_2Cl_2)$.

**General procedure for the organocatalytic α-sulfenylation of aldehydes and reduction to the corresponding alcohols.** The catalyst (0.05 mmol, 10 mol%) and the aldehyde (0.5 mmol) were stirred at ambient temperature in toluene (1.0 mL) for 5 min before the addition of 1-benzylsulfanyl-1,2,4-triazole (115 mg, 0.60 mmol, 1.2 eq.). The reaction mixture was then stirred for 3 h. After dilution of the reaction mixture with MeOH (4.0 mL), NaBH$_4$ (0.6 mmol) was added at ambient temperature. After 20 min, the reaction was quenched with 1 M aqueous KHSO$_4$ (3 mL). After standard aqueous workup, the products were purified by FC.

(2S)-Benzylsulfanyl-3-methyl-butyraldehyde 3a: The title compound was prepared according to the general procedure without subsequent reduction by NaBH$_4$. $^1$H NMR $\delta$ 9.20 (d, 1H, $J = 5.8$, CHO), 7.36-7.25 (m, 5H, ArH), 3.61 (d, 1H, $J = 3.4$, $CH_2$Ph), 3.48 (d, 1H, $J = 3.4$, $CH_2$Ph), 2.70 (dd, 1H, $J = 9.0$, $J = 5.7$, CHS), 1.95 (m, 1H, $CH(CH_3)_2$), 0.99 (d, 3H, $J = 6.3$, $CH(CH_3)_2$), 0.98 (d, 3H, $J = 6.3$, $CH(CH_3)_2); ^{13}$C NMR $\delta$ 193.1, 137.1, 129.1, 128.5, 127.2, 59.8, 34.7, 27.1, 20.8, 19.8. The ee of 3a in crude reaction mixtures was determined by GC on a Chrompak CP-ChiralSil Dex CB-column. Temperature program: 140°C isotherm for 10 min followed by a temperature ramp 140°C to 200°C at a rate of 10°C/min, $R_t$ (min): 12.91 ((S)-enantiomer), 12.99 ((R)-enantiomer).

(2S)-Benzylsulfanyl-3-methylbutan-1-ol 5a: The title compound was isolated following the general procedure. The spectroscopic data were in agreement with
literature values. The ee was determined by HPLC on a Daicel Chiralpak AD column with hexane/i-PrOH (99:1) as the eluent. Rₜ (min): 12.1 (major enantiomer), 13.2 (minor enantiomer); [α]₀ = -26.0 (c = 2.1, CH₂Cl₂, 98% ee). The absolute configuration was determined by optical rotation and compared with literature values.

(2S)-2-Benzylsulfanyl-propan-1-ol 5b: The title compound was isolated following the general procedure. ¹H NMR δ 7.36-7.25 (m, 5H, ArH), 3.79 (d, 1H, J = 7.4, CH₂Ph), 3.73 (d, 1H, J = 7.4, CH₂Ph), 3.61 (m, 1H, CH₂OH), 3.48 (m, 1H, CH₂OH), 2.85 (m, 1H, CHS), 2.17 (br, 1H, CH₂OH), 1.26 (d, 3H, J = 7.0, CHCH₃); ¹³C NMR δ 138.2, 128.7, 128.5, 127.1, 65.4, 42.8, 34.6, 17.8. The ee was determined by HPLC on a Daicel Chiralpak AD column with hexane/i-PrOH (98:2) as the eluent. Rₜ (min): 13.9 (minor enantiomer), 14.9 (major enantiomer); [α]₀ = -32.3 (c = 1.6, CHCl₃, 95% ee). The absolute configuration was determined by optical rotation and compared with literature values.

2,2-Bis-benzylsulfanyl-propan-1-ol: The title compound was isolated as a by-product following the general procedure. ¹H NMR δ 7.36-7.25 (m, 10H, ArH), 3.91 (d, 2H, J = 2.6, CH₂Ph), 3.87 (d, 2H, J = 2.6, CH₂Ph), 3.50 (s, 2H, CH₂OH), 2.10 (br, 1H, CH₂OH), 1.62 (s, 3H, CCH₃); ¹³C NMR δ 137.5, 129.0, 128.7, 127.2, 67.7, 63.2, 33.9, 25.2.

2-Benzylsulfanyl-butan-1-ol 5c: The title compound was isolated following the general procedure. ¹H NMR δ 7.36-7.25 (m, 5H, ArH), 3.79 (d, 1H, J = 7.4, CH₂Ph), 3.74 (d, 1H, J = 7.4, CH₂Ph), 3.67 (m, 1H, CH₂OH), 3.48 (m, 1H, CH₂OH), 2.63 (m, 1H, CHS), 2.17 (br, 1H, CH₂OH), 1.63 (m, 1H, CH₂CH₃), 1.52 (m, 1H, CH₂CH₃), 0.97 (m, 3H, CH₂CH₃); ¹³C NMR δ 138.3, 128.7, 128.5,
127.1, 63.4, 50.5, 34.8, 24.5, 11.6. The ee was determined by HPLC on 2 Daicel Chiralpak AS columns in a row with hexane/i-PrOH (99:1) as the eluent. R<sub>t</sub> (min): 19.9 (minor enantiomer), 21.2 (major enantiomer).

2-Benzylsulfanyl-3-phenyl-propan-1-ol 5d: The title compound was isolated following the general procedure. 

$^1$H NMR δ 7.36-7.25 (m, 8H, ArH), 7.15 (d, 2H, $J = 6.8$, ArH), 3.65 (m, 3H, SCH$_2$Ph and CH$_2$OH), 3.45 (m, 1H, CH$_2$OH), 2.85 (m, 3H, CHS and CCH$_2$Ph), 2.08 (br, 1H, CH$_2$OH); $^{13}$C NMR δ 138.7, 138.0, 129.2, 128.8, 128.6, 128.4, 127.2, 126.5, 62.9, 49.9, 38.3, 35.4. The ee was determined by HPLC on a Daicel Chiralpak AD column with hexane/i-PrOH (99:1) as the eluent. R<sub>t</sub> (min): 23.4 (major enantiomer), 25.6 (minor enantiomer); [$\alpha$]<sub>D</sub> = -9.0 (c = 2.3, CH$_2$Cl$_2$, 97% ee).

2-Benzylsulfanyl-pent-4-en-1-ol 5e: The title compound was isolated following the general procedure. 

$^1$H NMR δ 7.36-7.25 (m, 5H, ArH), 5.77 (m, 1H, CH=CH$_2$), 5.08 (m, 2H, CH=CH$_2$), 3.78 (d, 1H, $J = 6.2$, CH$_2$Ph), 3.75 (d, 1H, $J = 6.2$, CH$_2$Ph), 3.64 (m, 1H, CH$_2$OH), 3.52 (m, 1H, CH$_2$OH), 2.76 (m, 1H, CHS), 2.32 (m, 2H, CH$_2$), 2.05 (br, 1H, CH$_2$OH); $^{13}$C NMR δ 138.1, 135.0, 128.8, 128.6, 127.2, 117.3, 63.3, 47.9, 36.2, 35.1. The ee was determined by HPLC on 2 Daicel Chiralpak AD columns in a row with hexane/i-PrOH (99:1) as the eluent. R<sub>t</sub> (min): 32.0 (major enantiomer), 30.6 (minor enantiomer); [$\alpha$]<sub>D</sub> = -18.4 (c = 1.1, CH$_2$Cl$_2$, 96% ee).

2-Benzylsulfanyl-3,3-dimethyl-butan-1-ol 5f: The title compound was isolated following the general procedure.

$^1$H NMR δ 7.36-7.25 (m, 5H, ArH), 3.80-3.65 (m, 3H, SCH$_2$Ph and CH$_2$OH), 3.36 (ddd, 1H, $J = 11.3$, $J = 9.1$, $J = 3.9$, CH$_2$OH), 2.41
(dd, 1H, J = 9.1, J = 4.1, CHS), 2.22 (dd, 1H, J = 9.5, J = 3.9, CH$_2$OH), 0.91 (s, 9H, C(CH$_3$)$_3$); $^{13}$C NMR $\delta$ 138.4, 129.0, 128.6, 127.2, 61.9, 61.5, 38.5, 34.5, 27.9. The ee was determined by HPLC after conversion to 4-nitro-benzoic acid 2-benzylsulfanyl-3,3-dimethyl-butyl ester (see below); $[\alpha]_D = +18.8$ (c = 1.9, CH$_2$Cl$_2$, 95% ee).

![Image of chemical structure]

4-Nitro-benzoic acid 2-benzylsulfanyl-3,3-dimethyl-butyl ester: The title compound was prepared by adding an excess of 4-nitro benzoyl chloride and NEt$_3$ to an aliquot of 5f in CH$_2$Cl$_2$ and was purified by column chromatography. $^1$H NMR $\delta$ 8.29 (d, J = 8.9, 2H, ArH), 8.14 (d, J = 8.9, 2H, ArH), 7.33-7.17 (m, 5H, ArH), 4.62 (dd, 1H, J = 11.7, J = 5.4, CH$_2$O), 4.50 (dd, 1H, J = 11.7, J = 5.4, CH$_2$O), 3.73 (s, 2H, CH$_2$Ph), 2.62 (app. t, J = 5.9, 1H, CHS), 1.04 (s, 9H, C(CH$_3$)$_3$); $^{13}$C NMR $\delta$ 164.6, 150.5, 138.2, 135.5, 130.7, 129.0, 128.4, 127.1, 123.6, 66.7, 54.9, 37.7, 34.8, 28.0. The ee was determined on a Daicel Chiralpak AD column with hexane/i-PrOH (98:2) as the eluent. R$_t$ (min): 8.1 (major enantiomer); 8.9 (minor enantiomer); $[\alpha]_D = -67.1$ (c = 1.1, CH$_2$Cl$_2$, 96% ee).

![Image of chemical structure]

2-Benzylsulfanyl-2-phenyl-propan-1-ol 5g: The title compound was isolated following the general procedure. $^1$H NMR $\delta$ 7.55 (dd, 2H, J = 8.1, J = 1.5, ArH), 7.42-7.36 (m, 2H, ArH), 7.31-7.10 (m, 6H, ArH), 3.87 (d, 2H, J = 7.0, CH$_2$OH), 3.43 (d, 1H, J = 12.1, CH$_2$Ph), 3.39 (d, 1H, J = 12.1, CH$_2$Ph), 2.20 (t, 1H, J = 7.0, CH$_2$OH), 1.70 (s, 3H, CH$_3$); $^{13}$C NMR $\delta$ 142.5, 137.7, 128.7, 128.5, 128.4, 127.1, 127.0, 127.0, 68.0, 55.1, 33.7, 25.4. The ee was determined by HPLC on 2 Daicel Chiralpak AS columns in a row with hexane/i-PrOH (99:1) as the eluent. R$_t$ (min): 21.1 (minor enantiomer), 29.5 (major enantiomer); $[\alpha]_D = -25.2$ (c = 1.0, CH$_2$Cl$_2$, 61% ee).
Dibenzyl-(2-benzylsulfanyl-3-methyl-butyl-amine 6: 

The catalyst (0.05 mmol, 10 mol%) and the aldehyde 1a were stirred at ambient temperature in toluene (1.0 mL) for 5 min before the addition of 1-benzylsulfanyl-1,2,4-triazole (115 mg, 0.60 mmol, 1.2 eq.). The reaction mixture was then stirred for 3 h. The reaction mixture was quenched by filtration through a plug of silica using CH₂Cl₂ as the solvent. The solvent was removed in vacuo and the crude product was transferred using CH₂Cl₂ (1 mL) to a vial containing dibenzylamine (193 µL, 1.0 mmol) and sodium triacetoxyborohydride (212 mg, 1.0 mmol) in CH₂Cl₂ (3 mL). After stirring for 6 h, the resulting solution was quenched with saturated aq. NaHCO₃, extracted with EtOAc (3 × 10 mL), dried over Na₂SO₄, filtered, concentrated in vacuo and purified by FC (5% EtOAc/pentane). ¹H NMR δ 7.39-7.28 (m, 15H, Ar H), 3.70-3.56 (m, 4H, CH₂Ph), 3.27 (d, 2H, J = 13.6, CH₂Ph), 2.69 (dd, 1H, J = 12.8, J = 10.4, CH₂N), 2.58 (m, 1H, CHS), 2.55 (dd, 1H, J = 12.8, J = 4.8, CH₂N), 2.29 (m, 1H, CH(CH₃)₂), 0.86 (d, 3H, J = 6.8, CH(CH₃)₂), 0.49 (d, 3H, J = 6.8, CH(CH₃)₂). ¹³C NMR δ 139.5, 138.8, 129.0, 128.3, 128.1, 126.9, 59.0, 57.3, 50.4, 36.5, 28.0, 20.8, 16.3. The ee was determined by HPLC after conversion to 3-benzyl-5-isopropyl-thiazolidin-2-one (see below).

3-Benzyl-5-isopropyl-thiazolidin-2-one: Sodium metal was added in small portions to 6 in liquid NH₃ at -35°C until a blue color remained for 6 h. Then NH₄Cl (aq.) was added to decompose excess sodium, and the reaction mixture was allowed to warm up for removal NH₃. Toluene (2 mL) and K₂CO₃ (2 eq.) were added and the remaining NH₃ was removed by gentle heating. The reaction mixture was cooled to 0°C and a 2M solution of phosgene in toluene (1 mL, 2 eq.) was added. After being
stirred vigorously for 30 min the reaction was extracted with EtOAc, dried on Na₂SO₄ and concentrated in vacuo. The compound was purified by FC (gradient from CH₂Cl₂ to 5% Et₂O in CH₂Cl₂). ¹H NMR δ 7.38-7.25 (m, 5H, ArH), 4.50 (d, 1H, J = 14.8, CH₂Ph), 4.43 (d, 1H, J = 14.8, CH₂Ph), 3.50 (m, 2H, CH₂N), 3.20 (m, 1H, CHS), 1.82 (m, 1H, CH(CH₃)₃), 0.96 (d, 3H, J = 6.7, CH(CH₃)₃), 0.90 (d, 3H, J = 6.7, CH(CH₃)₃); ¹³C NMR δ 171.9, 135.9, 128.7, 128.1, 127.8, 51.9, 49.0, 48.4, 33.2, 20.1, 19.6. The ee was determined by HPLC on Daicel Chiralpak AS column with hexane/i-PrOH (95:5) as the eluent. Rₜ (min): 19.9 (major enantiomer); 22.8 (minor enantiomer).

(2S)-(2-Benzylsulfanyl-3-methyl-butoxy)-tert-butyl-dimethyl-silane 7: NEt₃ (1.2 eq) and TBDMSOTf (1.2 eq) were added to a solution of 5a (0.5 mmol) in CH₂Cl₂ and stirred overnight. After standard aqueous workup, the product was isolated by FC. ¹H NMR δ 7.28-7.17 (m, 5H, ArH), 3.69 (s, 2H, CH₂Ph), 3.59 (m, 2H, CH₂O), 2.46 (m, 1H, CHS), 2.06 (m, 1H, CH(CH₃)₂), 0.89-0.77 (m, 15H, CH(CH₃)₂ and Si(C(CH₃)₃)), -0.02 (s, 6H, Si(CH₃)₂); ¹³C NMR δ 138.9, 128.9, 128.3, 126.8, 65.1, 54.1, 36.9, 27.8, 25.9, 20.6, 18.2, 17.3, -5.3, -5.4.

Thioacetic acid S-[1-(tert-butyl-dimethyl-silyloxymethyl)-2-methyl-propyl] ester: Sodium metal was added in small portions to a solution of (2S)-(2-benzylsulfanyl-3-methyl-butoxy)-tert-butyl-dimethyl-silane (0.25 mmol) (see above) in liquid NH₃ at -35°C until a blue colour persisted for 1 h. Then NH₄Cl was added to decompose the excess sodium, and the reaction mixture was allowed to warm up to room temperature for removal of NH₃. CH₂Cl₂ (2 mL) was added to the solid residue. Pyridine (3 eq) and acetyl chloride (3 eq) were added to this solution at RT The mixture was stirred overnight. The resulting suspension was filtered and the
solvents were removed in vacuo. The title compound was purified by FC (65% yield). \(^1\)H NMR \(\delta\) 3.71 (dd, 1H, \(J = 10.0, J = 4.9, \text{CH}_2\text{O}\)), 3.64-3.54 (m, 2H, \(\text{CH}_2\text{O}\) and \(\text{CHS}\)), 2.33 (s, 3H, \(\text{CH}_3\text{CO}\)), 2.19 (m, 1H, \(\text{CH}(\text{CH}_3)_2\)), 0.97 (d, 3H, \(J = 6.8, \text{CH}(\text{CH}_3)_2\)), 0.93-0.85 (m, 12H, \(\text{CH}(\text{CH}_3)_2\) and \(\text{Si}(\text{C}(\text{CH}_3)_3)\)), 0.06 (s, 3H, \(\text{Si}(\text{C}(\text{CH}_3)_3)\)), 0.05 (s, 3H, \(\text{Si}(\text{C}(\text{CH}_3)_3)\)); \(^{13}\)C NMR \(\delta\) 195.7, 63.7, 52.5, 30.9, 27.5, 25.8, 20.6, 18.6, 18.2, -5.4, -5.5.

(2S)-1-(tert-Butyl-dimethyl-silanyloxy)-3-methyl-butane-2-thiol 8: An analytical sample of the title compound was isolated prior to acetylation of the thioacetic acid ester (see above) and purified by repeated FC.\(^3\) \(^1\)H NMR \(\delta\) 3.64 (d, 2H, \(J = 5.5, \text{CH}_2\text{O}\)), 2.83 (m, 1H, \(\text{CHS}\)), 2.04 (m, 1H, \(\text{CH}(\text{CH}_3)_2\)), 1.39 (d, 1H, \(J = 7.4, \text{SH}\)), 0.98 (d, 3H, \(J = 6.9, \text{CH}(\text{CH}_3)_2\)), 0.91-0.88 (m, 12H, \(\text{CH}(\text{CH}_3)_2\) and \(\text{Si}(\text{C}(\text{CH}_3)_3)\)), 0.05 (s, 6H, \(\text{Si}(\text{C}(\text{CH}_3)_3)\)); \(^{13}\)C NMR \(\delta\) 66.7, 49.3, 29.2, 25.8, 20.8, 18.2, 17.3, -5.3, -5.4.

References


[3] In order to facilitate workup and purification procedures, 8 was acetylated in situ.