Diastereoselective Synthesis of 1,3-syn Oxazines via a Tandem Hemiaminalization-Tsuji-Trost Reaction

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General Remarks: NMR spectra were recorded in CDCl3 on a Bruker AM 300, AM 500 spectrometer. EI and DCI mass spectra (reactant gas ammonia) were obtained on a Finnigan MAT 95 spectrometer, high resolution data were acquired using peak matching (M/DM = 10000). Analytical TLC (TLC polyester sheets silica gel Si 60 F254 (Merck), solvent: mixtures of ethylacetate/petroleum ether, detection: UV absorption at 254 nm, dark blue spots on staining with vanillin or cerium(IV)sulfate- phosphomolybdic acid in sulfuric acid followed by charring.
I. Experimental Details and Characterization Data.

Synthesis of homoallylic alcohol

Homoallylic alcohols were prepared in a one-pot procedure from the corresponding aldehydes by allyl Grignard addition.\(^1\)

Preparation of \((E)-N\)-benzylidene-4-methylbenzenesulfonamide 10\(^2\)

\[
\begin{align*}
\text{Chemical Formula: } & C_{14}H_{13}NO_2S \\
\text{Exact Mass: } & 259.06670 \\
\text{Molecular Weight: } & 259.32352
\end{align*}
\]

According to the literature:
A mixture of benzaldehyde (10 mmol), arenesulfonamide (10 mmol) and sodium \(p\)-toluenesulfinate (1.78 g, 10 mmol) or sodium benzenesulfinate (1.82 g, 11 mmol) in formic acid (15 mL) and \(H_2O\) (15 mL) was stirred for 12 h at r.t. The resulting white precipitate was filtered off, washed with \(H_2O\) (2 x 10 mL), then pentane (10 mL), and dissolved in \(CH_2Cl_2\) (100 mL). Sat. aq \(NaHCO_3\) or \(Na_2CO_3\) (70 mL) was added and the solution was well stirred for 2 h at r.t. The organic phase was decanted, the aqueous phase extracted with \(CH_2Cl_2\) (70 mL) and the combined organic layers dried (\(NaHCO_3\)), filtered off and the solvent removed under vacuum to yield the corresponding sulfonylimine 10.

Cross coupling of alcohols with alkenes\(^1,3\)

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Representative preparative procedure of compound substrate-15

Under argon and room temperature, 1-phenylbut-3-en-1-ol (adduct 1) (0.836 g, 5.6 mmol) and (Z)-but-2-ene-1,4-diyl tert-butyl dicarbonate (adduct 2) (3.246 g, 11.3 mmol) were dissolved in 16 mL anhydrous dichloromethane. To this solution was added the solution of Grubbs catalyst 2nd (0.239 g, 0.28 mmol) in 8 mL anhydrous dichloromethane. The reaction mixture was refluxed overnight, TLC monitoring (Rf (adduct 1) = 0.27, Rf (substrate-15) = 0.10, ethyl acetate/hexane = 1:6) indicated the terminal of reaction. Solvent was removed in vacuo and the residue was purified on the column of silica gel, eluted with the gradient of petroleum ether and ethyl acetate, from 10:1, 7:1 to 4:1 to afford 1.116 g (4.01 mmol) the compound of substrate-15 as an viscous pale-brown oil. Yield: 71 %. The spectroscopic data was identical to those previously reported.1, 2

Representative procedure for preparation of oxazinane 11

Under argon, (E)-N-benzyldiene-4-methylbenzenesulfonamide 10 (62.2 mg, 0.24 mmol), allylpalladium(II) chloride dimer (7.3 mg, 10 mol%) and triphenylphosphine (15.8 mg, 30 mol%) added to a well-dried Schlenk flask. Then, a solution of (E)-tert-butyl (5-hydroxy-5-(p-tolyl)pent-2-en-1-yl) carbonate 9 (58.4 mg, 0.2 mmol) in dried toluene (0.33 M, 0.61 mL) added to the flask, stirred until all solids are dissolved. Cooled down to -78 °C, the potassium bis(trimethylsilyl)amide solution (0.5 M in toluene) was dropwise added (40 μL, 10 mol%). 15 minutes after addition, warmed it up to room temperature, stirred it until TLC (Rf (9) = 0.49, Rf (11) = 0.61, ethyl acetate/hexane = 1:4) showed the terminal of reaction. 2 mL saturated solution of NH₄Cl was added, the residue was extracted with ethyl acetate three
times, the combined organic phase was washed with water, brine, respectively, then was dried over sodium sulfate. The solvent was removed in vacuo and the residue was purified by column chromatography on silica gel with ethyl acetate/hexane = 1:16 as eluent to afford the product 11a (58 mg, 67%) as a colorless oil together with its diastereomers 11b, 11c, 11d (see below).

**HPLC separation conditions:**

Column: KNAUER Eurospher II 100 RP C-18; 5 μm; 250 × 16,0 mm.
Flow rate: 15.0 ml/min, Pressure: 103-83 bar.

Eluent Gradient:
- Start: 75% A (Acetonitrile) + 25 % B (Water),
- 15 Min: 85% A + 15% B
- 17 Min: 85% A + 15% B
- 18 Min: 75% A + 25% B
- 20 Min: 75% A + 25% B

For results please see part II of supporting information.

2-phenyl-6-(p-tolyl)-3-tosyl-4-vinyl-1,3-oxazinane (11a)

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Chemical Formula: C_{26}H_{27}NO_{3}S
Exact Mass: 433.17
Molecular Weight: 433.56
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(dr 3.9: 1.0: 2.7 : 1.5; combined yield: 67%)
Data for 11a: $^1$H NMR (500.13MHz, CDCl$_3$) $\delta$ = 7.97 (d, $J$ = 8.5 Hz, 1 H), 7.95 (d, $J$ = 8.5 Hz, 1 H) 7.63 (d, $J$ = 8.2 Hz, 1 H), 7.58 (d, $J$ = 8.2 Hz, 1 H), 7.29-7.42 (m, 5 H), 7.12-7.19 (m, 2 H), 7.07 (d, $J$ = 7.9 Hz, 1 H), 7.00 (d, $J$ = 7.9 Hz, 1 H), 6.75 (s, 1 H), 5.78 (ddd, $J$ = 17.3, 10.4, 7.7 Hz, 1 H), 5.02 (d, $J$ = 17.3 Hz, 1 H), 4.96 (d, $J$ = 10.3 Hz, 1 H), 4.40 (ddd, $J$ = 8.5, 8.2, 7.7 Hz, 1 H), 3.86 (dd, $J$ = 10.2, 4.7 Hz, 1 H), 2.50 (s, 3 H), 2.35 (s, 3 H), 1.93 (ddd, $J$ = 13.7, 10.2, 8.2 Hz, 1 H), 1.54 (ddd, $J$ = 13.7, 8.5, 4.7 Hz, 1 H); $^{13}$C NMR (125.76MHz, CDCl$_3$) $\delta$ = 143.93, 141.52, 139.12, 137.74, 137.36, 129.78, 129.19, 128.02, 127.82, 127.63, 126.81, 126.17, 125.71, 115.73, 83.83, 73.15, 56.10, 33.62, 21.58, 21.08; HRMS (ESI) m/z : [M+H]$^+$ Calcd for C$_{26}$H$_{28}$NO$_3$S 434.17844; Found 434.17839.

6-(2-methoxyphenyl)-2-phenyl-3-tosyl-4-vinyl-1,3-oxazinane (12a)

Chemical Formula: C$_{26}$H$_{27}$NO$_4$S
Exact Mass: 448.17
Molecular Weight: 449.56

Data for 12a: $^1$H NMR (500.13MHz, CDCl$_3$) $\delta$ = 7.91-7.96 (m, 4 H), 7.71 (d, $J$ = 7.7 Hz, 2 H), 7.50 (d, $J$ = 7.7 Hz, 2 H), 7.32-7.40 (m, 5 H), 6.73 (s, 1 H), 5.78 (ddd, $J$ = 17.3, 10.4, 6.6 Hz, 1 H), 5.02 (d, $J$ = 17.3 Hz, 1 H), 4.94 (d, $J$ = 10.4 Hz, 1 H), 4.50 (ddd, $J$ = 8.2, 8.0, 6.6 Hz, 1 H), 4.34 (dd, $J$ = 11.5, 2.2 Hz, 1 H), 3.71 (s, 3 H), 2.48 (s, 3 H), 2.07 (ddd, $J$ = 13.7, 8.0, 2.2 Hz, 1 H), 1.82 (ddd, $J$ = 13.7, 11.5, 8.2 Hz, 1 H); $^{13}$C NMR (125.77MHz, CDCl$_3$) $\delta$ = 170.03, 155.85, 143.35, 141.45, 139.34, 131.19, 129.72, 129.06, 128.57, 128.00, 127.90, 127.65, 126.89, 120.62, 115.46, 110.29, 84.36, 68.03, 55.78, 55.09, 33.19, 21.54; HRMS (ESI) m/z : [M+K]$^+$ Calcd for C$_{26}$H$_{27}$NO$_4$SK 488.12924; found 488.12948.
6-(4-methoxyphenyl)-2-phenyl-3-tosyl-4-vinyl-1,3-oxazinane (13a)

Chemical Formula: C_{26}H_{27}NO_4S
Exact Mass: 449.17
Molecular Weight: 449.56

Data for 13a: \(^1\)H NMR (500.13 MHz, CDCl\(_3\)) \(\delta = 7.90-7.98\) (m, 4 H), 7.62 (d, \(J = 7.6\) Hz, 2 H), 7.42 (d, \(J = 7.9\) Hz, 2 H), 7.36 (d, \(J = 7.9\) Hz, 2 H), 7.09 (d, \(J = 8.5\) Hz, 1 H), 6.88 (d, \(J = 8.8\) Hz, 2 H), 6.75 (s, 1 H), 5.78 (dddd, \(J = 17.0, 10.4, 6.6\) Hz, 1 H), 5.03 (d, \(J = 17.0\) Hz, 1 H), 4.96 (d, \(J = 10.4\) Hz, 1 H), 4.40 (td, \(J = 8.2, 8.2, 6.6\) Hz, 1 H), 3.83 (dd, \(J = 13.9, 2.8\) Hz, 1 H), 3.81 (s, 3H), 2.51 (s, 3 H), 1.91 (ddd, \(J = 16.7, 8.2, 2.8\) Hz, 1 H), 1.52 (ddd, \(J = 16.7, 13.9, 8.2\) Hz, 1 H); \(^{13}\)C NMR (125.77 MHz, CDCl\(_3\)) \(\delta = 170.06, 159.33, 143.91, 141.54, 139.12, 131.24, 129.76, 129.09, 128.01, 127.83, 127.59, 126.77, 115.72, 113.90, 83.81, 73.01, 56.14, 55.26, 33.54, 21.58; HRMS (ESI) m/z: [M+K]^+ Calcd for C_{26}H_{27}NO_4SK 488.12924; found 488.12947.

6-(naphthalen-2-yl)-2-phenyl-3-tosyl-4-vinyl-1,3-oxazinane (14a)
Data for 14a: $^1$H NMR (300.08MHz, CDCl₃) δ = 8.0 (t, $J = 7.7$ Hz, 2 H), 7.79-7.85 (m, 4 H), 7.62-7.69 (m, 4 H), 7.38-7.52 (m, 4 H) 7.18 (d, $J = 8.5$ Hz, 2 H), 7.05 (s, 1 H), 5.83 (ddd, $J = 17.1$, 10.4, 6.3 Hz, 1 H), 5.12 (d, $J = 17.1$ Hz, 1 H), 4.96 (d, $J = 10.4$ Hz, 1 H), 4.48 (td, $J = 8.8$, 8.8, 6.3 Hz, 1 H), 4.09 (dd, $J = 9.6, 4.9$ Hz, 1 H), 2.52 (s, 3 H), 2.07 (ddd, $J = 13.9, 9.6, 8.8$ Hz, 1 H), 1.60 (ddd, $J = 13.9, 8.8, 4.9$ Hz, 1 H); $^{13}$C NMR (75.46MHz, CDCl₃) δ = 144.00, 141.41, 137.80, 137.33, 136.38, 135.83, 133.14, 129.85, 129.2, 128.85, 128.34, 128.08, 127.84, 127.68, 127.26, 126.82, 126.28, 126.07, 124.60, 124.08, 123.59, 115.87, 83.97, 73.32, 56.08, 33.62, 21.61; HRMS (ESI) m/z : [M+Na]$^+$ Calcd for C$_{29}$H$_{27}$NO$_3$SNa 492.16055; Found 492.16090.

2,6-diphenyl-3-tosyl-4-vinyl-1,3-oxazinane (15a)
**Data for 15a:** $^1$H NMR (300.13MHz, CDCl$_3$) $\delta$ = 7.97 (d, $J$ = 8.0 Hz, 2 H), 7.64 (d, $J$ = 7.7 Hz, 2 H), 7.28 - 7.45 (m, 8 H), 7.20 (d, $J$ = 6.9 Hz, 2 H), 6.77 (s, 1 H), 6.03 (s, 1 H), 5.78 (ddd, $J$ = 17.3, 10.4, 6.6 Hz, 1 H), 5.07 (d, $J$ = 17.3 Hz, 1 H), 4.92 (d, $J$ = 10.4 Hz, 1 H), 4.68 (dd, $J$ = 9.1, 3.3 Hz, 1 H), 3.97 (ddd, $J$ = 8.8, 6.6, 5.5 Hz, 1 H), 2.00 (ddd, $J$ = 14.3, 9.1, 8.8 Hz, 1 H), 2.50 (s, 3 H), 1.77 (ddd, $J$ = 14.3, 5.5, 3.3 Hz, 1 H); $^{13}$C NMR (75.46MHz, CDCl$_3$) $\delta$ = 143.76, 141.43, 139.08, 137.33, 129.82, 128.49, 128.31, 128.05, 127.64, 127.27, 126.20, 125.97, 125.75, 115.80, 83.89, 73.32, 56.05, 33.71, 21.58; HRMS (ESI) m/z : [M+Na]$^+$ Calcd for C$_{25}$H$_{25}$NO$_3$SNa 442.14474; Found: 442.14497.

6-phenethyl-2-phenyl-3-tosyl-4-vinyl-1,3-oxazinane (16a)

![Chemical Structure of 16a](image)

**Chemical Formula:** C$_{26}$H$_{26}$NO$_3$S

**Exact Mass:** 447.18681

**Molecular Weight:** 447.58906

(dr 9.8: 2.1: 3.9: 1.0; combined yield 63%)

**Data for 16a:** $^1$H NMR (500.13MHz, CDCl$_3$) $\delta$ = 7.82 (d, $J$ = 8.2 Hz, 2 H), 7.59 (d, $J$ = 8.0 Hz, 2 H), 7.37 (t, $J$ = 7.4 Hz, 2 H), 7.29-7.33 (m, 6 H), 7.14 (d, $J$ = 6.9 Hz, 2 H), 6.57 (s, 1 H), 5.78 (ddd, $J$ = 17.3, 10.4, 6.9 Hz, 1 H), 5.01 (d, $J$ = 17.3 Hz, 1 H), 4.96 (d, $J$ = 10.4 Hz, 1 H), 4.21 (ddd, $J$ = 8.5, 7.7, 6.9 Hz, 1 H), 2.99 (ddt, $J$ = 11.8, 8.0, 3.8, 3.8 Hz, 1 H), 2.83 (ddd, $J$ = 14.3, 10.7, 5.5 Hz, 1 H), 2.73 (ddd, $J$ = 14.3, 9.1, 5.2 Hz, 1 H), 2.61-2.67 (m, 1 H), 2.53-2.60 (m, 1 H), 2.46 (s, 3 H), 1.87-1.95 (m, 1 H), 1.66 (ddd, $J$ = 14.3, 7.7, 3.8 Hz, 1 H), 1.64-1.69 (m, 1 H), 1.54 (ddd, $J$ = 14.3, 11.8, 8.5 Hz, 1 H); $^{13}$C NMR (125.76MHz, CDCl$_3$) $\delta$ = 143.59, 141.75, 141.44, 139.51, 129.60, 128.36, 128.32, 128.16, 128.05, 127.67, 127.24, 126.65, 125.87, 115.47, 83.76, 70.70, 55.44, 32.74, 21.58; HRMS (ESI) m/z : [M+H]$^+$ Calcd for C$_{27}$H$_{30}$NO$_3$S 448.19409; Found: 448.19416.
6-cyclohexyl-2-phenyl-3-tosyl-4-vinyl-1,3-oxazinane (17a)

\[
\text{Chemical Formula: } C_{25}H_{31}NO_3S \\
\text{Exact Mass: } 425.20 \\
\text{Molecular Weight: } 425.58
\]

\[
\text{(dr 5.2: 1.0: 1.1: 1.1; combined yield 51%)}
\]

**Data for 17a:** \(^1\)H NMR (500.13MHz, CDCl\(_3\)) \(\delta = 7.78\) (d, \(J = 8.2\) Hz, 2 H), 7.51 (d, \(J = 8.0\) Hz, 2 H), 7.21-7.33 (m, 5 H), 6.45 (s, 1 H), 5.68 (ddd, \(J = 17.2, 10.2, 7.4\) Hz, 1 H), 4.94 (d, \(J = 17.2\) Hz, 1 H), 4.86 (d, \(J = 10.2\) Hz, 1 H), 4.20 (dt, \(J = 8.2, 8.2, 7.4\) Hz, 1 H), 2.54 (ddd, \(J = 11.5, 7.1, 3.3\) Hz, 1 H), 2.41 (s, 3 H), 1.67 (ddd, \(J = 13.7, 8.2, 3.3\) Hz, 1 H), 1.56-1.64 (m, 2 H), 1.43 (ddd, \(J = 13.7, 11.5, 8.2\) Hz, 1 H), 1.28 (td, \(J = 7.2, 7.2, 3.4\) Hz, 1 H), 1.04-1.20 (m, 4 H), 0.68-0.96 (m, 4 H); \(^{13}\)C NMR (125.76MHz, CDCl\(_3\)) \(\delta = 143.61, 141.57, 139.64, 129.52, 128.78, 127.87, 127.75, 127.66, 126.74, 115.28, 84.10, 75.84, 55.62, 42.50, 30.55, 28.37, 26.33, 25.57, 21.46; HRMS (ESI) m/z : [M+H]\(^+\) Calcd for C\(_{25}\)H\(_{32}\)NO\(_3\)S 426.20974; Found: 426.20979.

6-pentyl-2-phenyl-3-tosyl-4-vinyl-1,3-oxazinane (18a)

\[
\text{Chemical Formula: } C_{26}H_{31}NO_3S \\
\text{Exact Mass: } 413.20 \\
\text{Molecular Weight: } 413.57
Data for 18a: $^1$H NMR (500.13MHz, CDCl$_3$) δ = 7.86 (d, $J = 8.2$ Hz, 2 H), 7.56 (d, $J = 8.2$ Hz, 2 H), 7.28-7.37 (m, 5 H), 6.56 (s, 1 H), 5.73 (ddd, $J = 17.3$, 10.4, 7.7 Hz, 1 H), 4.99 (d, $J = 17.3$ Hz, 1 H), 4.93 (d, $J = 10.4$ Hz, 1 H), 4.20 (dt, $J = 8.8$, 7.7, 7.7 Hz, 1 H), 2.87 (ddddd, $J = 11.5$, 7.7, 4.7, 3.0 Hz, 1 H), 2.46 (s, 3 H), 1.64 (ddd, $J = 14.3$, 7.7, 3.0 Hz, 1 H), 1.48 (ddd, $J = 14.3$, 11.5, 8.8 Hz, 1 H), 1.22-1.36 (m, 6 H), 1.13-1.20 (m, 2 H), 0.89 (t, $J = 7.4$ Hz, 3 H); $^{13}$C NMR (125.76MHz, CDCl$_3$) δ = 143.63, 141.85, 139.50, 129.59, 128.32, 127.96, 127.75, 127.49, 126.68, 115.36, 83.49, 71.31, 55.71, 36.09, 32.69, 31.27, 24.72, 22.49, 21.53, 13.97; HRMS (ESI) m/z : [M+H]$^+$ Calcd for C$_{24}$H$_{32}$NO$_3$S 414.20974; Found: 414.20994.

3-(benzylamino)-1-(p-tolyl)pent-4-en-1-ol (19)

Oxazine 11 (52 mg, 0.12 mmol, 1.0 equiv) as a diastereomeric mixture ($syn/anti = 1.3:1$) dissolved in 2 mL dry methanol was added to a oven-dried schlenk tube. Then, under the flow of argon, it was heated up to reflux for 8 hours. TLC monitoring ($R_f$ (18) = 0.61, $R_f$ (19) = 0.16, ethyl acetate/hexane = 1:4) indicated the terminal of reaction. 50 mL water and 25 mL chloroform were added and stirred for another 15 minutes. The organic phase was separated, the inorganic phase were further extracted with chloroform (3 × 25 mL), the combined organic phase was dried over MgSO$_4$. Solvent was removed in vacuo and the residue was purified on the column of silica gel, eluted with the gradient of petroleum ether and ethyl acetate (0.2% Et$_3$N), from 16:1, 8:1, 4:1 to 2:1 to afford 21.7 mg product 19 as a pale-yellow oil. Yield: 64%. The diastereomeric ratio of syn to anti is 2.6 : 1.0.

syn-diastereomer 19a
1H NMR (400.18 MHz, CDCl3) δ = 1.67 (dt, J = 14.5, 10.6 Hz, 1 H), 1.80 (dt, J = 14.5, 2.6 Hz, 1 H), 2.32 (s, 3 H), 3.43 (ddd, J = 10.6, 8.1, 2.6 Hz, 1 H), 3.71 (d, J = 12.7 Hz, 1 H), 3.92 (d, J = 12.7 Hz, 1 H), 4.89 (dd, J = 10.6, 2.6 Hz, 1 H), 5.14 (dt, J = 17.2, 0.9 Hz, 1 H), 5.20 (dt, J = 10.3, 0.9 Hz, 1 H), 5.65 (ddd, J = 17.2, 10.3, 8.1 Hz, 1 H), 7.12 (d, J = 7.9 Hz, 2 H), 7.24 (d, J = 7.9 Hz, 2 H), 7.32-7.37 (m, 5 H). 13C NMR (100.64 MHz, CDCl3) δ = 21.07, 44.13, 50.72, 61.47, 74.81, 116.09, 125.44, 127.35, 128.51, 128.59, 128.90, 136.59, 138.79, 138.85, 141.98. HRMS (ESI) m/z : [M+H]+ Calcd for C19H24NO2 282.18524; Found: 282.18524.

anti-diastereomer 19b

1H NMR (400.18 MHz, CDCl3) δ = 1.94 (ddd, J = 5.9, 4.4, 1.1 Hz, 1 H), 1.94 (ddd, J = 6.4, 4.4, 1.8 Hz, 1 H), 2.34 (s, 3 H), 3.33 (ddd, J = 7.7, 6.4, 4.4 Hz, 1 H), 3.68 (d, J = 12.8 Hz, 1 H), 3.80 (d, J = 12.8 Hz, 1 H), 5.02 (dd, J = 5.9, 4.4 Hz, 1 H), 5.11 (dt, J = 10.6, 1.3 Hz, 1 H), 5.12 (dt, J = 17.2, 1.3 Hz, 1 H), 5.84 (ddd, J = 17.2, 10.6, 7.7 Hz, 1 H), 7.17 (d, J = 8.1 Hz, 2 H), 7.24 (d, J = 8.1 Hz, 2 H), 7.31-7.37 (m, 5 H). 13C NMR (100.64 MHz, CDCl3) δ = 21.07, 41.81, 50.95, 58.04, 71.85, 116.34, 125.44, 127.35, 128.51, 128.59, 128.90, 136.59, 138.57, 138.85, 141.98.
Chemical Shift (ppm)
WL334R.ESP
Chemical Shift (ppm)
Chemical Shift (ppm)

OH

OBoc
Chemical Shift (ppm)
Chemical Shift (ppm)

$\text{dr} 18 : 4.0 : 1.0$
Chemical Shift (ppm)

$dr_{18:4.0:1.0}$
Chemical Shift (ppm)

\[ dr \ 9.2 : 1.0 : 2.1 \]
Chemical Shift (ppm)

$dr \ 9.2 : 1.0 : 2.1$
Chemical Shift (ppm)

dr 1.0 : 1.5 : 4.8
Chemical Shift (ppm)

$\delta 1.0 : 1.5 : 4.8$
Chemical Shift (ppm)
d.r 6.5 : 1.0 : 1.5

ON T s
Ph
ON T s
Ph
ON T s
Ph

ON
Ph
ON
Ph
ON
Ph
ON
Ph

$\text{dr } 3.9: 2.7: 1.0: 1.5$
Chemical Shift (ppm)

$dr \ 3.9 : 2.7 : 1.0 : 1.5$
Chemical Shift (ppm)

$dr_{10.1: 2.7: 1.0: 2.8}$
dr 10.1: 2.7: 1.0: 2.8
Chemical Shift (ppm)

\[ dr \ 8.0: 4.5: 1.7: 1.0 \]
Chemical Shift (ppm)

$dr \ 8.0: 4.5: 1.7: 1.0$
$dr \, 1.9: \, 2.7: \, 1.5: \, 1.0$
Chemical Shift (ppm)

$dr = 1.9: 2.7: 1.5: 1.0$

Supporting Information
Chemical Shift (ppm)

$dr \ 1.6: 2.3: 1.1: 1.0$
Chemical Shift (ppm)

Dr 1.6: 2.3: 1.1: 1.0
$\text{Chemical Shift (ppm)}$

$\text{dr \ 9.8: 2.1: 3.9: 1.0}$
Chemical Shift (ppm)

$dr \ 9.8: 2.1: 3.9: 1.0$
dr 5.2: 1.0: 1.1: 1.1
dr 5.2: 1.0: 1.1: 1.1
Chemical Shift (ppm)

$dr 5.2 : 1.5 : 1.0 : 2.2$
Chemical Shift (ppm)

dr 5.2 : 1.5 : 1.0 : 2.2
Chemical Shift (ppm)

$19a : 19b = 2.6 : 1.0$
19a : 19b = 2.6 : 1.0