Transition metal free free Asymmetric and Diastereoselective Allylic Alkylation using Grignard reagents: Construction of vicinal stereogenic centers via Kinetic resolution.

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SUPPORTING INFORMATION AVAILABLE


2. Catalysis adducts characterization and preparation of substrates........3-19.

3. GC traces............................................................................................................20-61.

4. NMR spectra.....................................................................................................62-95.
General Remarks.

All reactions were not carried out under nitrogen or argon atmosphere in flame-dried glassware and with dry solvents. Solvents (THF, Et₂O, Toluene, DCM, MTBE and CH₃CN) were dried over alumina (activated at 350 °C under nitrogen atmosphere for 12 h). Yields refer to chromatographically and spectroscopically ¹H NMR homogeneous materials, unless otherwise stated. Reactions were monitored by GC-MS Hewlet Packard (EI mode) HP6890-5973 on an HP6890 or by TLC carried out on 0.25 mm E. Merck silica gel plates (60F-254) using UV lamp as visualizing agent and KMnO₄ solution as developing agents. Flash chromatographies were performed using silica gel (particle size 32-63 µm, 60 Å). ¹H (300, 400 MHz or 500 MHz) and ¹³C (75 or 100 MHz) NMR spectra were recorded on Bruker AMX-300 or 400 instrument in CDCl₃ and calibrated using residual undeuterated solvent as an internal reference. Chemical shift (δ) are given in ppm relative to tetramethylsilane (0 ppm). Multiplicity is indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), br s (broad singlet). Coupling constants J are reported in Hz. Mass spectra (MS) and High resolution mass spectra (HRMS) were obtained by Electrospray Ionisation (ESI) or by electronic impact (EI, 70 eV). Optical rotations were measured at 20°C in a 10 cm cell in the stated solvent; [α]D values are given in 10-1 deg.cm2 g⁻¹ (concentration c given as g/100 mL). Enantiomeric excesses were determined either by chiral GC measurement on a HP6890 (H₂ as vector gas) or HP6850 (He or H₂ as vector gas). Temperature programs are described as follows; initial T (°C) – initial times (min) – temperature gradient (°C/min) – final T (°C); retention times (RT) are given in min.). or by SFC with supercritical carbon dioxide as vector and iPrOH or MeOH. Programme sequence as followed: Name of the column, percentage of iPrOH or MeOH. retention time 1 and retention time 2.
Optimization of the ligand.

![Reaction Scheme]

**Ligands**

- **L1**: \( R^1 = \text{Et}, R^2 = \text{Et}, R^3 = \text{H} \)
- **L2**: \( R^1 = \text{Et}, R^2 = \text{Me}, R^3 = \text{R}^4 = \text{H} \)
- **L3**: \( R^1 = \text{Et}, R^2 = \text{Me}, R^3 = \text{H}, R^4 = \text{Ph} \)
- **L4**: \( R^1 = \text{Et}, R^2 = \text{Me}, R^3 = \text{H}, R^4 = 1\text{-Naphthyl} \)
- **L5**: \( R^1 = \text{Et}, R^2 = \text{Me}, R^3 = \text{H}, R^4 = 2\text{-Naphthyl} \)
- **L6**: \( R^1 = \text{Et}, R^2 = \text{Me}, R^3 = \text{H}, R^4 = 4\text{-FPh} \)
- **L7**: \( R^1 = \text{R}^2 = \text{R}^3 = \text{Me}, R^4 = \text{H} \)

### Table 1

<table>
<thead>
<tr>
<th>Entry</th>
<th>( L )</th>
<th>Conv% ([^a])</th>
<th>( S_{N2}/S_{N2} )^[^a]</th>
<th>anti/syn ([^a])</th>
<th>e:e % ([^b])</th>
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<td>100/0</td>
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<tr>
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<td>100/0</td>
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<td>100/0</td>
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</tr>
<tr>
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<td>100/0</td>
<td>100/0</td>
<td>85:15</td>
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<tr>
<td>5</td>
<td>L4</td>
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<td>100/0</td>
<td>100/0</td>
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<tr>
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<td>50</td>
<td>100/0</td>
<td>100/0</td>
<td>85:15</td>
</tr>
</tbody>
</table>

**Table 1**: \([^a]\) Ratio determined by \(^1\)H-(NMR). \([^b]\) Determined using chiral GC. \([^c]\) Isolated yield after silica gel column chromatography on 0.8 mmol scale.

The screening of the ligand library began by examination of the importance of the blocking part of the ligand (left part). The switch from two methyl \( L7 \) to an ethyl/methyl \( L2 \) pattern or an ethyl/ethyl \( L1 \) pattern allowed to improve the enantioselectivity greatly (Tables 1. Entry 1 vs Entries 2,3). However no difference in terms of enantioselectivity can be measured using \( L1 \) or \( L2 \). Having these results in hand the screen of the moiety in para position of the hydroxyl group on the chelating part of the ligand was then probed (right part). The introduction of a phenyl group \( L3 \) was deleterious on the enantiomeric excess (Table 1. Entry 4). On the opposite the use of more bulky \( L4 \) with a 1-Naphthyl group afforded the same level of enantiomeric excess than \( L1 \) or \( L2 \) (Table 1. Entries 2,3 vs Entry 5). A slight modification of the ligand morphology by using a 2-Naphthyl group \( L6 \) led to a decrease of the ee value to 76 % ee (Table 1. Entry 6). The introduction of a fluorine group led to a decrease of the enantioselectivity to 70 % ee (Table 1. Entry 7). The ligands \( L1 \), \( L2 \) and \( L4 \) were compared...
by isolating the product after silica gel chromatography. To our surprise the ligand **L4** allowed us to isolate the desired adduct in 35 % isolated yield versus 20 and 25 % isolated yield respectively for the ligand **L1** and **L2**. To conclude the enantioselectivity and the isolated yield can be controlled by tuning in an appropriate way the NHC ligand. The diastereoselectivity and the regioselectivity are not affected whatever is the structure of the NHC ligand employed.

The desired methyl ketones were commercially available or prepared according to the following procedure (M. Mc Intosh and al, *J. Org. Chem*, **1983**, 48, 2987.). The other ketone were prepared according to the procedure (G. B. Trimitis and al, *Org. Lett*, **2010**, 9, 1996).

**Preparation of substrate.**

Into a conditioned two necked round bottomed flask the appropriate ketone (1.5 g,1 eq) is added and dissolved in 25 mL of dry THF. The round bottomed flask is placed into a bath containing crushed ice. The vinyl magnesium chloride (1,5 eq) is added dropwise and the ice bath is removed and the mixture is stirred at room temperature for 2 h. After completion by TLC the round bottomed flask is placed into an ice bath and 20 mL of saturated aqueous solution of ammonium chloride is added. 30 mL of ether is added and the aqueous phase is extracted two times with 20 mL portion of ether each. The organic phases are combined and dried over anhydrous sodium sulfate. The solvent is removed using a rotavapor and dried over high vacuum using an oil pump. The $^1$H NMR analysis of the crude revealed a pure compound which is used in the next step without further purification. The tertiary alcohol (1 eq) is diluted in a mixture of pentane/ether 9/1 and HBr (48 % aq,5 mL) is added under vigorous stirring. After 30 min (crucial to respect the exact time) the aqueous phase is removed using a separation funnel. The organic phase is washed with saturated solution of sodium bicarbonate. The organic phase is dried over sodium sulfate. The solvent is evaporated and the product is dried under high vacuum. Products are recovered 100 % E isomer and are silica gel, basic alumina and neutral alumina sensitive product in 55-62 % yield over two steps.

![Compound](image-url)

Compound is obtained as brown oil with 70 % purity (55 % yield based on purity) >99/1 % E isomer and is used as crude and stored in the freezer at -30°C. $^1$H (NMR) (400 MHz,
$^{1}C$ NMR (75 MHz, $C_{6}H_{6}$) $\delta$ 158.5, 146.5, 136, 128.9 120.1, 113.8, 54.5, 46.5, 28.92, 20.1, 17.1.

Compound is obtained as brown oil with 75 % purity (58 % yield based on purity) >99/1 % E isomer and is used as crude and stored in the freezer at -30°C. $^{1}H$ (NMR) (400 MHz, $C_{6}H_{6}$) $\delta$ 7.50-7.20 (m, 2H) 6.90-6.80 (m, 2H) 5.70 (t, 1H, J = 8.2 Hz) 4.03 (d, 2H, J=8.4 Hz) 3.75 (q, 1H, J= 7 Hz) 1.61 (s, 3H) 1.41 (d, 3 H, J= 6.2 Hz). $^{13}C$ NMR (75 MHz, $C_{6}H_{6}$) $\delta$ 145, 143, 129.9, 129.8, 128.3, 127.7, 127.5, 126.3, 126.2, 47.4, 28.7, 19, 14.3. HRMS (EI + mode) m/z expected: 315,9462 observed: 315,9463.

Compound is obtained as brown oil with 80 % purity (62 % yield based on purity) >99/1 % E isomer and is used as crude and stored in the freezer at -30°C. $^{1}H$ (NMR) (400 MHz, $C_{6}H_{6}$) $\delta$ 7.50-7.20 (m, 5H) 5.81 (t, 1H, J= 8.5 Hz) 4.11 (d, 2H, J=8.4 Hz) 3.50 (q, 1H, J= 7.1 Hz) 1.61 (s, 3H) 1.41 (d, 3 H, J= 6.2 Hz). $^{13}C$ NMR (75 MHz, $C_{6}H_{6}$) $\delta$ 146, 144.2, 128.5, 128.3, 127.7, 127.5, 126.3, 126.2, 47.4, 28.7, 19, 14.3. HRMS (EI + mode) m/z expected: 238,0357 observed: 238,0358.

Compound is obtained as brown oil with 70 % purity (52 % yield based on purity) >99/1 % E isomer and is used as crude and stored in the freezer at -30°C. $^{1}H$ (NMR) (400 MHz, $C_{6}H_{6}$) $\delta$ 7.50-7.20 (m, 5H) 5.78 (t, 1H, J= 8.4 Hz) 4.09 (d, 2H, J=8.4 Hz) 3.85 (q, 1H, J= 7.1 Hz) 1.59 (s, 3H) 1.38 (d, 3 H, J= 6.22 Hz). $^{13}C$ NMR (75 MHz, $C_{6}H_{6}$) $\delta$ 162 (dd, J=10.8 Hz and J=243.9 Hz), 146, 140 (d, J=3.2 Hz), 139, 129.5 (d, J=7.6 Hz), 128.6 (d, J=7.8 Hz), 121, 115 (dd, J=4.7 Hz and J=21 Hz), 46.4, 28.6, 18.9, 14. HRMS (EI + mode) m/z expected: 256,0263 observed: 256,0262.
\[^{13}\text{C} \text{NMR} (75 \text{ MHz, } \text{C}_6\text{H}_6) \delta 144.9, 142.7, 129, 128.2, 127.8, 126.3, 120.9, 55.5, 28.7, 25.1, 13.8, 12.1. \text{HRMS (EI + mode) m/z expected: 252,0514 observed: 252,0512.}\] 

Compound is obtained as brown oil with 70% purity (55% yield based on purity) >99/1% E isomer and is used as crude and stored in the freezer at -30°C. \[^1\text{H} \text{NMR} (400 \text{ MHz, } \text{C}_6\text{H}_6) \delta 7.50-7.20 (m, 5H) 5.82 (t, 1H, J= 8.3 Hz) 4.09 (d, 2H, J=8.5 Hz) 3.90 (t, 1H, J= 7.5 Hz) 1.60-1.50 (m, 4H) 1.4-1.30 (m, 2H) 1.30 (s, 3H) 0.8-0.7 (m, 3H). \[^{13}\text{C} \text{NMR} (75 \text{ MHz, } \text{C}_6\text{H}_6) \delta 145.1, 142.9, 128.9, 127.7, 126.4, 126.2, 120.8, 53.7, 31.9, 29.8, 28.7, 22.8, 13.9, 13.8. \text{HRMS (EI + mode) m/z expected: 280,0827 observed: 280,0829.}\] 

Compound is obtained as brown oil with 70% purity (52% yield based on purity) >99/1% E isomer and is used as crude and stored in the freezer at -30°C. \[^1\text{H} \text{NMR} (400 \text{ MHz, } \text{C}_6\text{H}_6) \delta 7.50-7.20 (m, 5H) 5.85-5.75 (m, 1H) 5.1-4.9 (m, 3H) 4.12 (t, 2H, J=8.3 Hz) 3.9 (t, 1H, J= 7.8 Hz) 1.60 (s, 3H) 1.25 (t, 2H, J= 6.6 Hz). \[^{13}\text{C} \text{NMR} (75 \text{ MHz, } \text{C}_6\text{H}_6) \delta 144.3, 142.2, 136.9, 129, 128.2, 128.1, 126.4, 121.2, 115.9, 53.4, 36.8, 28.6, 20.8. \text{HRMS (EI + mode) m/z expected: 264,0514 observed: 264,0514.}\] 

Compound is obtained as brown oil with 75% purity (58% yield based on purity) >99/1% E isomer and is used as crude and stored in the freezer at -30°C. \[^1\text{H} \text{NMR} (400 \text{ MHz, } \text{C}_6\text{H}_6) \delta 7.50-7.20 (m, 5H) 5.80-5.70 (m, 1H) 5-4.90 (m, 3H) 4.15 (t, 2H, J=8.3 Hz) 3.88 (t, 1H, J= 7.8 Hz) 1.62 (s, 3H) 1.40-1.25 (m, 4H). \[^{13}\text{C} \text{NMR} (75 \text{ MHz, } \text{C}_6\text{H}_6) \delta 144.3, 142.2, 136.9, 129, 128.2, 128.1, 126.4, 121.2, 115.9, 53.4, 37.2, 36.8, 28.6, 20.8. \text{HRMS (EI + mode) m/z expected: 278,0670 observed: 278,0671.}\] 

Compound is obtained as brown oil with 75% purity (58% yield based on purity) >99/1% E isomer and is used as crude and stored in the freezer at -30°C. \[^1\text{H} \text{NMR} (400 \text{ MHz, } \text{C}_6\text{H}_6) \delta 7.50-7.20 (m, 5H) 5.80-5.60 (m, 1H) 5-4.90 (m, 3H) 4.17 (t, 2H, J=8.3 Hz) 3.86 (t, 1H, J= 7.8 Hz) 1.65 (s,2H) 1.50-1.25 (m, 6H). \[^{13}\text{C} \text{NMR} (75 \text{ MHz, } \text{C}_6\text{H}_6) \delta 144.3, 142.2, 136.9, 129, 128.2, 128.1, 126.4, 121.2, 115.9, 53.4, 37.2, 36.8, 28.6, 20.8. \text{HRMS (EI + mode) m/z expected: 292,0827 observed: 292,0828.}\]
Compound is obtained as brown oil with 70 % purity (61 % yield based on purity) >99/1 % E isomer and is used as crude and stored in the freezer at -30°C. $^1$H (NMR) (400 MHz, C$_6$H$_6$) $\delta$ 7.30-7.20 (m, 2H) 6.90- 6.80 (m, 2H) 5.71 (t, 1H, J= 8.3 Hz) 4.01 (d, 2H, J=8.3 Hz) 3.80 (q, 1H, J= 7.2 Hz) 1.51 (s, 3H) 1.31 (d, 3H, J= 7.1 Hz). $^{13}$C NMR (75 MHz, C$_6$H$_6$) $\delta$ 145.2, 142, 129.6, 129.4, 121.8, 120.4, 47.2, 29.2, 20.1, 18.3. HRMS (EI + mode) m/z expected: 315,9462 observed: 315,9462.

Compound is obtained as brown oil with 70 % purity (55 % yield based on purity) >99/1 % E isomer and is used as crude and stored in the freezer at -30°C. $^1$H (NMR) (400 MHz, C$_6$H$_6$) $\delta$ 7.40-7.30 (m, 2H) 6.95-6.85 (m, 2H) 5.74 (t, 1H, J= 8.3 Hz) 4.05 (d, 2H, J=8.4 Hz) 3.44 (q, 1H, J= 7.3 Hz) 1.55 (s, 3H) 1.34 (d, 3H, J= 7.15 Hz). $^{13}$C NMR (75 MHz, C$_6$H$_6$) $\delta$ 145.2, 142, 134, 129.6, 129.4, 121.8, 120.4, 53.3, 36.8, 28.8, 20.8. HRMS (EI + mode) m/z expected: 271,9967 observed: 271,9965.

Compound is obtained as brown oil with 80 % purity (65 % yield based on purity) >99/1 % E isomer and is used as crude and stored in the freezer at -30°C. $^1$H (NMR) (400 MHz, C$_6$H$_6$) $\delta$ 7.5-7.20 (m, 5H) 5.70 (t, 1H, J= 8Hz) 4.10 (d, 2H, J=8.2 Hz) 3.89 (q, 1H, J=6.9 Hz) 2.36 (s, 3H) 1.61 (s, 3H) 1.40 (d, 3H, J= 7.1 Hz). $^{13}$C NMR (75 MHz, C$_6$H$_6$) $\delta$ 146.5, 136, 128.9 120.1, 113.8, 46.5, 28.92, 21.8, 21.3, 20.1, 17.1.

Compound is obtained as brown oil with 70 % purity (62 % yield based on purity) >99/1 % E isomer and is used as crude and stored in the freezer at -30°C. $^1$H (NMR) (400 MHz, C$_6$H$_6$) $\delta$ 7.50-7.20 (m, 5H) 5.70 (t, 1H, J= 8Hz) 4.02 (d, 2H, J=8.2 Hz) 3.78 (q, 1H, J=6.9 Hz) 2.70 (d, 1H, J= 11.7 Hz ) 1.76 (s, 3H) 1.33 (d, 3H, J= 7.1 Hz). $^{13}$C NMR (75 MHz, C$_6$H$_6$) $\delta$ 146, 141.8, 141.7, 130.2, 128.8, 127.9, 127.8, 127.4, 126 1, 120.1, 113.8, 55.5, 28.92, 21.8, 21.3, 20.1, 17.1.
Preparation of Grignard reagent.

Into a conditioned two necked flask equipped with a reflux condenser and an addition funnel magnesium turnings (2.72 g, 1.3 eq) are introduced. The system is connected to high vaccum of the manifold and heat to 600 °C for 15 minutes using a heating gun. The magnesium turnings are stirred vigorously while cooling down into vaccum. The system is then backfilled with nitrogen and the cycle is repeated two times. The corresponding bromide (1 eq) is dissolved into 30 mL of dry ether and placed into the addition funnel. A few millilitres of the solution are poured and the Grignard formation starts by boiling of the ether. The rest of the solution is added over 45 minutes and stirring is continued for 2 hours. The liquid is cannulated into a conditioned bottle and titrated using phenanthroline/menthol. The typical concentrations obtained are between 1.8 M and 2.3 M.

Catalysis procedure for racemate.

In a flame-dried Schlenk, under N₂ atmosphere, the racemic allylic bromide (0.8 mmol) and racemic NHC ligand L (2 mol %) are suspended in 3.2 mL of dry Et₂O (crucial to respect the dilution) and cooled to -15°C. RMgBr x M in Et₂O (0.8 eq) is added dropwise over 4 min. After stirring overnight full conversion is reached, the mixture is quenched by addition of a saturated solution of NH₄Cl (2 ml). The aqueous layer is separated and extracted with Et₂O (3 × 3 ml). The combined organic fractions are dried over Na₂SO₄, filtered and concentrated in vaccuo. The residue is purified by flash column chromatography using pentane (100 %) as eluant (0.5 cm diameter and 10 cm high column).

Determination of the absolute configuration.

The optically pure (S)-allylic substrate was prepared starting from 2 g of optically pure (R)-Styrene oxide and transformed into the desired allylic bromide by reported procedure. The catalysis was then performed using both (R, R) and (S, S) chiral NHC ligand. The absolute configuration of the product below could be attributed by analogy.
(R)-Styrene oxide

\[
\text{(Al)Me}_3 \cdot \text{Toluene} \quad 70 \%
\]


Jones oxidation

\[
\text{75}\%
\]


**Catalysis procedure.**

In a flame-dried Schlenk, under N\(_2\) atmosphere, the racemic allylic bromide (0.8 mmol) and chiral NHC ligand (prepared from (S,S) Corey diamine see ref below) \(\text{L}\) (2 mol %) are suspended in 3.2 mL of dry Et\(_2\)O (crucial to respect the dilution) and cooled to -40°C. RMgBr \(\times\) M in Et\(_2\)O (0.8 eq) is added dropwise over 4 min. After half conversion, the mixture is quenched by addition of a saturated solution of NH\(_4\)Cl (2 ml). The aqueous layer is separated
and extracted with Et₂O (3 × 3 ml). The combined organic fractions are dried over Na₂SO₄, filtered and concentrated in vacuo. The residue is purified by flash column chromatography using pentane (100 %) as eluant (0.5 cm diameter and 6 cm high column).


Catalysis procedure for scale up.

In a flame-dried Schlenk, under N₂ atmosphere, the racemic allylic bromide (5 mmol) and chiral NHC ligand (prepared from (S,S) Corey diamine see ref below) L (2 mol %) are suspended in 20 mL of dry Et₂O (crucial to respect the dilution) and cooled to -40°C. RMgBr x M in Et₂O (0.8 eq) is added dropwise over 4 min. After half conversion, the mixture is quenched by addition of a saturated solution of NH₄Cl (2 ml). The aqueous layer is separated and extracted with Et₂O (3 × 3 ml). The combined organic fractions are dried over Na₂SO₄, filtered and concentrated in vacuo. The residue is purified by flash column chromatography using pentane (100 %) as eluant (2 cm diameter and 8 cm high column).

\[1a\] Reaction time: 1 h. Product isolated as colourless oil in 36 % yield, >99/1 anti/syn ratio, >99/1 S₉₂'/S₉₂ ratio with 92 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture.

GC: CP-CHIRASIL DEX CB: 60-0-1-170-5: t1= 55.20 min, t2=55.80 min

\(^{1}\)H (NMR) (400 MHz, CDCl₃) \(\delta\) 7.40-7.25 (m, 5H) 5.80 (q, 1H, J= 10.9 Hz) 5.12 (dd, 1H, J= 1.5 Hz and J=10.8 Hz) 4.93 (d, J=1.5 Hz and J=17.5 Hz) 1.50-1.40 (m, 2H) 1.25 (d, 3H, J= 6.9 Hz) 0.80 (s, 3H) 0.65 (t, 3H, J=7.5 Hz). \(^{13}\)C NMR (75 MHz, CDCl₃) \(\delta\) 145.7, 144.3, 129.4, 127.4, 125.9, 112.4, 48.3, 43.2, 31.6, 17.9, 16.1, 8.5. HRMS (EI + mode) m/z expected: 242,2035 observed: 242,2037. \([\alpha] = +13.2\) (c 1.33, CHCl₃).

\[1b\] Reaction time: 1h 30. Product isolated as colourless oil in 32 % yield, >99/1 anti/syn ratio, >99/1 S₉₂'/S₉₂ ratio with 86 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: t1=52.80 min, t2= 53.40 min.

\(^{1}\)H (NMR) (400 MHz, CDCl₃) \(\delta\) 7.40-7.25 (m, 5H) 5.80 (q, 1H, J= 10.9 Hz) 5.12 (dd, 1H, J= 1.5 Hz and J=10.8 Hz) 4.93 (d, J=1.5 Hz and J=17.5 Hz) 1.36-1.25 (m, 7H) 0.80-0.70
(m, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 145.7, 144.7, 129.4, 127.4, 125.9, 112.9, 48.6, 43.1, 41.8, 18.6, 17.4, 16.2, 14.9. HRMS (EI + mode) m/z expected: 202,1722 observed: 202,1723. \([\alpha] = +8.1\ (c\ 1.33,\ CHCl_3)\).

**1c.** Reaction time: 1h 30. Product isolated as colourless oil in 35 % yield, >99/1 anti/syn ratio, >99/1 $\text{S}_2$/S$\text{N}_2$ ratio with 84 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: CP-CHIRASIL DEX CB: 60-0-1-170-5: t1 = 68.8 min, t2 = 69.40 min $^1$H (NMR) (400 MHz, CDCl$_3$) $\delta$ 7.40-7.10 (m, 5H) 5.75 (q, 1H, J=10.9 Hz) 5.15 (dd, 1H, J= 1.55 Hz and J= 10.8 Hz) 2.70 (q, 1H, J= 7.2 Hz) 1.45-1.20 (m, 9 H) 0.80-0.70 (m, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 145.7, 144.3, 129.4, 127.4, 125.9, 112.4, 48.6, 43, 39.2, 26.4, 23.5, 18.7, 16.2, 14.2. HRMS (EI + mode) m/z expected: 216,1878 observed: 216,1877. \([\alpha] = +9.6\ (c\ 1.33,\ CHCl_3)\).

**1d.** Reaction time: 1h 30. Product isolated as colourless oil in 36 % yield, >99/1 anti/syn ratio, >99/1 $\text{S}_2$/S$\text{N}_2$ ratio with 91 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: CP-CHIRASIL DEX CB: 60-0-1-170-5: t1 = 78.90 min, t2 = 79.30 min. $^1$H (NMR) (400 MHz, CDCl$_3$) $\delta$ 7.40-7.20 (m, 5H) 5.80 (q, 1H, J= 10.9 Hz) 5.12 (d, 1H, J=10.90 Hz) 4.92 (d, J=17.5 Hz) 2.70 (q, 1H, J= 7.2 Hz) 1.35-1.25 (m, 12H) 0.80 (t, 3H, J=7.4 Hz) 0.75 (t, 3H, J=7.5 Hz). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 145.7, 144.3, 129.4, 127.4, 125.9, 112.9, 48.6, 43.1, 39.4, 32.8, 23.9, 22.7, 18.7, 16.2, 14.1. HRMS (EI + mode) m/z expected: 230,2035 observed: 230,2037. \([\alpha] = +10.6\ (c\ 1.33,\ CHCl_3)\).

**1e.** Reaction time: 1h 30. Product isolated as colourless oil in 36 % yield, >99/1 anti/syn ratio, >99/1 $\text{S}_2$/S$\text{N}_2$ ratio with 90 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: t1 = 88.10 min, t2 = 88.50 min. $^1$H (NMR) (400 MHz, CDCl$_3$) $\delta$ 7.40-7.20 (m, 5H) 5.80 (q, 1H, J= 10.9 Hz) 5.12 (dd, 1H, J= 1.5 Hz and J=10.8 Hz) 4.93 (d, J=1.5 Hz and J=17.5 Hz) 2.70 (q, 1H, J= 7.2 Hz) 1.40-1.32 (m, 15H) 0.80-0.70 (m, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 145.7, 144.3, 129.4, 127.4, 125.9, 112.9, 48.6, 43, 39.5, 31.9, 30.2, 24.2, 22.7, 18.7, 16.2, 14.1.
14.1. HRMS (EI + mode) m/z expected: 244.2191 observed: 244.2192. $[\alpha] = +10.1$ (c 1.33, CHCl$_3$).

**1f.** Reaction time: 1h 30. Product isolated as colourless oil in 36% yield, >99/1 anti/syn ratio, >99/1 $S_N2'/S_N2$ ratio with 86% ee after column chromatography on silica gel using 100% pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: $t_1$=78.60 min, $t_2$= 79.10 min. $^1$H (NMR) (400 MHz, CDCl$_3$) $\delta$ 7.40-7.10 (m, 5H) 5.84-5.78 (m, 2H) 5.14 (dd, 1H, J= 1.5 Hz and J=10.9 Hz) 5.05-4.95 (m, 3H) 2.70 (q, 1H, J= 7.2 Hz) 2.10-2 (m, 2H) 1.60-1.50 (m, 3H) 1.25 (s, 3H) 1.20 (s, 3H) 0.75 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 145.3, 143, 139.6, 129.4, 127.5, 126, 113.9, 113.8, 48.8, 43, 38.6, 28.8, 18.3, 16.2. HRMS (EI + mode) m/z expected: 214.1722 observed: 214.1724. $[\alpha] = +8.2$ (c 1.33, CHCl$_3$).

**1g.** Reaction time: 1h 45. Product isolated as colourless oil in 34% yield, >99/1 anti/syn ratio, >99/1 $S_N2'/S_N2$ ratio with 84% ee after column chromatography on silica gel using 100% pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: $t_1$=75.50 min, $t_2$= 76.10 min.$^1$H (NMR) (400 MHz, CDCl$_3$) $\delta$ 7.40-7.10 (m, 5H) 5.85-5.78 (m, 2H) 5.10 (dd, 1H, J= 1.5 Hz and J=10.9 Hz) 4.97 (q, 2H, J=19.2 Hz) 4.90 (q, 1H, J= 17.6 Hz) 2.70 (q, 1H, J= 7.2 Hz) 2 (d, 2H, J=7 Hz) 1.35-1.25 (m, 7H) 0.75 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 145.3, 139, 129.4, 127.4, 125.9, 114.2, 113.1, 48.7, 38.9, 34.5, 23.7, 18.7, 16.2. HRMS (EI + mode) m/z expected: 228.1878 observed: 228.1880. $[\alpha] = +8.6$ (c 1.33, CHCl$_3$).

**1h.** Reaction time: 1h 30. Product isolated as colourless oil in 37% yield, >99/1 anti/syn ratio, >99/1 $S_N2'/S_N2$ ratio with 86% ee after column chromatography on silica gel using 100% pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: $t_1$=88 min, $t_2$= 88.40 min. $^1$H (NMR) (400 MHz, CDCl$_3$) $\delta$ 7.40-7.10 (m, 5H) 5.86-5.78 (m, 2H) 5.10 (dd, 1H, J= 1.5 Hz and J=10.9 Hz) 4.97 (q, 2H, J=19.2 Hz) 4.90 (q, 1H, J= 17.6 Hz) 2.70 (q, 1H, J= 7.2 Hz) 2.10-2 (m, 2H) 1.40-1.25 (m, 9H) 0.75 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 145.6, 144.2, 139.1, 129.4, 128.9, 128, 127.9, 127.4, 125.9, 114.1, 113, 48.6,
43, 39.2, 33.8, 29.8, 25.6, 23.7, 18.7, 16.2. HRMS (EI + mode) m/z expected: 242,2035 observed: 242,2033. $[\alpha] = +7.7$ (c 1.33, CHCl$_3$).

**II.** Reaction time: 4h 30. Product isolated as colourless oil in 35% yield, >99/1 anti/syn ratio, >99/1 $S_N2'/S_N2$ ratio with 22% ee after column chromatography on silica gel using 100% pentane as solvent mixture. $^1$H (NMR) (400 MHz, CDCl$_3$) $\delta$ 7.4-7.1 (m, 5H) 6.9 (m, 2H) 5.90 (q, 1H, $J=10.9$ Hz) 5.20 (d, 1H, $J=1.7$ Hz and $J=10.9$ Hz) 4.90 (d, 1H, $J=1.60$ Hz and $J=17.5$ Hz) 2.86 (q, 1H, $J=7.2$ Hz) 1.90-1.50 (m, 8H) 1.30 (s, 6H) 1.0-0.9 (m, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 144.7, 143.6, 129.3, 127.4, 125.9, 114, 46.6, 45.8, 45.3, 27.2, 25.9, 25.8, 16.7, 16.2. HRMS (EI + mode) m/z expected: 228,1878 observed: 228,1881. $[\alpha] = +1.6$ (c 1.33, CHCl$_3$).

**lj.** Reaction time: 45 min. Product isolated as colourless oil in 32% yield, >99/1 anti/syn ratio, >99/1 $S_N2'/S_N2$ ratio with 86% ee after column chromatography on silica gel using 100% pentane as solvent mixture. GC: CP-CHIRASIL DEX CB: 60-0-1-170-5: $t_1$=64.8 min, $t_2$=65.50 min. $^1$H (NMR) (400 MHz, CDCl$_3$) $\delta$ 7.30-7.20 (s, 4H) 5.70 (q, 1H, $J=10.8$ Hz) 5.11 (dd, 1H, $J=1.5$ Hz and $J=10.9$ Hz) 4.90 (dd, 1H, $J=1.5$ Hz and $J=17.5$ Hz) 2.70 (q, 1H, $J=7.3$ Hz) 2.35 (s, 3H) 1.5-1.4 (m, 3H) 1.25 (d, 3H, $J=7.3$ Hz) 0.80 (s, 3H) 0.70 (t, 3H, $J=7.4$ Hz). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 145.6, 141.2, 135.3, 129.3, 126.1, 113.1, 48, 43.3, 31.7, 21 17.7, 16.3, 8.5. HRMS (EI + mode) m/z expected: 202,1722 observed: 202,1723. $[\alpha] = +8.6$ (c 1.33, CHCl$_3$).

**lk.** Reaction time: 45 min. Product isolated as colourless oil in 34% yield, >99/1 anti/syn ratio, >99/1 $S_N2'/S_N2$ ratio with 92% ee after column chromatography on silica gel using 100% pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: $t_1$=86.60 min, $t_2$= 87.60 min.$^1$H (NMR) (400 MHz, CDCl$_3$) $\delta$ 7.30 (d, 2H, $J=7.7$ Hz) 7 (d, 2H, $J=7.6$ Hz) 5.80 (q, 1H, $J=10.9$ Hz) 5.12 (dd, 1H, $J=1.5$ Hz and $J=10.8$ Hz) 4.93 (d, $J=1.6$ Hz and $J=17.5$ Hz) 2.70 (q, 1H, $J=7.2$ Hz) 1.70-1.60 (m, 5H) 0.80 (s, 3H) 0.70 (t, 3H, $J=7.5$ Hz). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 144.8, 143.3, 131, 130.5, 119.7, 113.7, 48, 43.1, 31.6, 18,
16.1, 8.5. HRMS (EI + mode) m/z expected: 266,0670 observed: 266,0669. [α] = +11.9 (c 1.33, CHCl3).

**11.** Reaction time: 40 min. Product isolated as colourless oil in 35 % yield, >99/1 anti/syn ratio, 100/0 SN2/SN2 ratio with 94 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: t1=51.40 min, t2= 52.70 min. $^1$H (NMR) (400 MHz, CDCl3) δ 7.20-7.30 (m, 2H) 7-6.90 (m, 2H) 5.80 (q, 1H, J= 10.9 Hz) 5.12 (dd, 1H, J= 1.5 Hz and J=10.8 Hz) 4.93 (d, J=1.5 Hz and J=17.5 Hz) 2.70 (q, 1H, J= 7.2 Hz) 1.45-1.35 (m, 2H) 1.25 (d, 3H, J= 6.8 Hz) 0.80 (s, 3H) 0.65 (t, 3H, J=7.5 Hz). $^{13}$C NMR (75 MHz, CDCl3) δ 162.5, 160.1, 145, 139.7, 130.6, 114.9, 114.7, 114.2 114, 113.5, 47.2, 43.2, 31.6, 17.9, 16.3, 8.5. HRMS (EI + mode) m/z expected: 206,1471 observed: 206,1472. [α] = +12.6 (c 1.33, CHCl3).

**1m.** Reaction time: 50 min. Product isolated as colourless oil in 38 % yield, >99/1 anti/syn ratio, 100/0 SN2/SN2 ratio with 78 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: t1=79.20 min, t2= 79.80 min. $^1$H (NMR) (400 MHz, CDCl3) δ 7.20-7.30 (m, 2H) 6.90-6.80 (m, 2H) 5.80 (q, 1H, J= 10.9 Hz) 5.12 (dd, 1H, J= 1.5 Hz and J=10.8 Hz) 4.93 (d, J=1.6 Hz and J=17.5 Hz) 3.82 (s, 3H) 2.70 (q, 1H, J= 7.2 Hz) 1.70-1.60 (m, 5H) 0.80 (s, 3H) 0.70 (t, 3H, J=7.5 Hz). $^{13}$C NMR (75 MHz, CDCl3) δ 157.8, 145.3, 136.4, 130.5, 128.3, 113.4, 113.1, 112.7, 55.2, 47.6, 43.4, 31.6, 17.7, 16.4, 8.6. HRMS (EI + mode) m/z expected: 218,1671 observed: 218,1673. [α] = +4.5 (c 1.33, CHCl3).

**1n.** Reaction time: 50 min. Product isolated as colourless oil in 32 % yield, >99/1 anti/syn ratio, >99/1 SN2/SN2 ratio with 84 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: t1=70.70 min, t2= 71.70 min. $^1$H (NMR) (400 MHz, CDCl3) δ 7.50 (dd, 1H, J= 1.3 Hz and J= 7.9 Hz) 7.25 (m, 2H) 7-6.90 (m, 1H) 5.80 (q, 1H, J= 10.9 Hz) 5.12 (dd, 1H, J= 1.5 Hz and J=10.8 Hz) 4.93 (d, J=1.5 Hz and J=17.50 Hz) 3.4 (q, 1H, J= 7.17 Hz) 1.5-1.4 (m, 2H) 1.25 (d, 3H, J= 6.9 Hz)
0.80 (s, 3H) 0.65 (t, 3H, J=7.5 Hz). $^{13}$C NMR (75 MHz, CDCl$_3$) δ 144.2, 144, 132.7, 129.8, 127.6, 126.6, 126.3, 114, 45.2, 31.3, 17.8, 16.5, 8.5. HRMS (EI + mode) m/z expected: 266,0670 observed: 266,0672. [α] = +8.1 (c 1.33, CHCl$_3$).

**1o.** Reaction time: 45 min. Product isolated as colourless oil in 31 % yield, >99/1 anti/syn ratio, >99/1 S$_N$2'/S$_N$2 ratio with 92 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: t1=68.90 min, t2= 69.40 min.$^1$H (NMR) (400 MHz, CDCl$_3$) δ 7.40-7.30 (m, 4H) 7.20-7.10 (m, 1H) 5.80 (q, 1H, J= 10.9 Hz) 5.12 (dd, 1H, J= 1.5 Hz and J=10.9 Hz) 4.93 (d, J=1.5 Hz and J=17.5 Hz) 2.70 (q, 1H, J= 7.2 Hz) 1.70-1.60 (m, 5H) 0.80 (s, 3H) 0.70 (t, 3H, J=7.5 Hz). $^{13}$C NMR (75 MHz, CDCl$_3$) δ 144.2, 144, 132.7, 129.8, 127.6, 126.6, 126.3, 114, 45.2, 31.3, 17.8, 16.5, 8.5. HRMS (EI + mode) m/z expected: 266,0670 observed: 266,0672. [α] = +8.1 (c 1.33, CHCl$_3$).

**1p.** Reaction time: 50 min. Product isolated as colourless oil in 36 % yield, >99/1 anti/syn ratio, >99/1 S$_N$2'/S$_N$2 ratio with 82 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: CP-CHIRASIL DEX CB: 60-0-1-170-5: t1= 57 min, t2=57.50 min. $^1$H (NMR) (400 MHz, CDCl$_3$) δ 7.4-7.1 (m, 5H) 5.66 (q, 1H, J= 10.9 Hz) 5 (dd, 1H, J= 1.60 Hz and J=10.9 Hz ) 4.80 (dd, 1H, J=1.6 Hz and J=17.6 Hz ) 2.24 (dd, 1H, J= 2.8 Hz and J=12 Hz) 1.70-1.50 (m, 2H) 1.25-1.10 (m, 2H) 0.75 (s, 3H) 0.70 (t, 3H, J=7.4 Hz) 0.6 (t, 3H, J=7.4 Hz). $^{13}$C NMR (75 MHz, CDCl$_3$) δ 146.1, 142.1, 129.9, 127.9, 127.8, 127.4, 125.9, 113.2, 57.4, 43.7, 32.2, 22.7, 17.7, 13.1, 8.5. HRMS (EI + mode) m/z expected: 202,1722 observed: 202,1720. [α] = +7.6 (c 1.33, CHCl$_3$).

**1q.** Reaction time: 1h. Product isolated as colourless oil in 35 % yield, >99/1 anti/syn ratio, >99/1 S$_N$2'/S$_N$2 ratio with 82 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: t1=66.20 min, t2= 66.60 min. $^1$H (NMR) (400 MHz, CDCl$_3$) δ 7.40-7.10 (m, 5H) 5.75 (q, 1H, J= 10.9 Hz) 5 (dd, 1H, J= 1.5 Hz and J=10.8 Hz) 4.90 (dd, 1H, J=1.5 Hz and J=17.5 Hz) 2.40 (dd, 1H, J= 2.8 Hz and J=11.9 Hz) 1.70-1.60 (m, 2H) 1.40-1.30 (m, 6H) 1.1-1 (m, 3H) 0.90 (s, 3H) 0.80
(t, 3H, J=7.3 Hz) 0.70 (t, 3H, J=7.5 Hz). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 146.1, 142.5, 127.4, 125.9, 113.2, 55.2, 43.7, 32.2, 30.6, 29.6, 22.7, 17.8, 14, 8.5. HRMS (EI + mode) m/z expected: 230,2035 observed: 230,2036. $[\alpha] = +6.8$ (c 1.33, CHCl$_3$).

**1s.** Reaction time: 45 min. Product isolated as colourless oil in 37 % yield, >99/1 anti/syn ratio, >99/1 S$_{N}$2'/S$_{N}$2 ratio with 90 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: t$_1$=78.1 min, t$_2$=78.4 min.$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.40-7.20 (m, 5H) 5.84-5.75 (m, 2H) 5.73 (q, 1H, J= 6.7 Hz) 5.73 (q, 1H, J= 6.7 Hz) 5.82-5.70 (m, 1H) 5.16 (dd, 1H, J= 1.3 Hz and J= 10.8 Hz) 4.94 (dd, 1H, J= 1.5 Hz and J= 11.5 Hz) 4.92-4.80 (m, 2H) 2.49 (dd, 1H, J= 2.1 Hz and J= 11.5 Hz) 2-1.7 (m, 4H) 1.30-1.20 (m, 3H) 1 (dd, 1H, J= 3.3 Hz and J= 6.6 Hz) 0.88 (s, 3H) 0.74 (t, 3H, J= 7.4 Hz). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 146, 142.2, 138.9, 128, 127.7, 127.5, 125.9, 114.1, 113.3, 55.1, 43.6, 33.8, 32.2, 29.3, 27.6, 17.7, 8.4. HRMS (EI + mode) m/z expected: 242,2035 observed: 242,2037. $[\alpha] = +11.1$ (c 1.33, CHCl$_3$).

**1t.** Reaction time: 45 min. Product isolated as colourless oil in 34 % yield, >99/1 anti/syn ratio, >99/1 S$_{N}$2'/S$_{N}$2 ratio with 91 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: HYDRODEX B-6 TBDMS: 60-0-1-170-5: t$_1$=66.5 min, t$_2$=66.8 min.$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.40-7.20 (m, 5H) 5.82-5.75 (m, 2H) 5.73 (q, 1H, J= 6.7 Hz) 5.70-5.60 (m, 1H) 5.16 (dd, 1H, J= 1.3 Hz and J= 10.8 Hz) 4.94 (dd, 1H, J= 1.5 Hz and J= 11.5 Hz) 4.92 (dd, 1H, J= 2.1 Hz and J= 11.5 Hz) 1.30-1.20 (m, 3H) 1 (dd, 1H, J= 3.3 Hz and J= 6.6 Hz) 0.88 (s, 3H) 0.74 (t, 3H, J= 7.4 Hz). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 145.9, 141.9, 138.9, 128, 127.5, 126, 114.3, 113.3, 54.4, 43.5, 32.3, 32.2, 29.1, 17.7, 8.4. HRMS (EI + mode) m/z expected: 228,2878 observed: 228,2880. $[\alpha] = +12.2$ (c 1.33, CHCl$_3$).

**1u.** Reaction time: 1h. Product isolated as colourless oil in 35 % yield, >99/1 anti/syn ratio, >99/1 S$_{N}$2'/S$_{N}$2 ratio with 92 % ee after
column chromatography on silica gel using 100 % pentane as solvent mixture. SFC: AZ-column with 2 % MeOH and 98 % CO₂: t₁= 4.20 min, t₂= 4.40 min. H (NMR) (400 MHz, CDCl₃) δ 7.40-7.10 (m, 8H) 7-6.90 (m, 2H) 5.90 (q, 1H, J= 10.9 Hz ) 5.30 (d, 1H, J=10.8 Hz ) 5.10 (d, 1H, J= 17.5 Hz) 3.20 (d, 1H, J= 13.3 Hz) 2.90 (t, 1H, J= 11.8 Hz) 2.70 (d, 1H, J= 11.7 Hz ) 1.60-1.50 (m, 3H) 1 (s, 3H) 0.80 (t, 3H, J= 7.5 Hz). C NMR (75 MHz, CDCl₃) δ 146, 141.8, 141.7, 130.2, 128.8, 127.9, 127.8, 127.4, 126.1, 125.3, 114, 57.6, 44.1, 36.8, 32.3, 17.6, 8.6. HRMS (EI + mode) m/z expected: 264,1878 observed: 264,1879. [α] = +11.6 (c 1.33, CHCl₃).

1v. Reaction time: 12 h. Product isolated as colourless oil in 31 % yield, 8.5/1 anti/syn ratio, >99/1 S₂N₂/S₂N₂ ratio with 85 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. GC: CP-CHIRASIL DEX CB: 60-0-1-170-5: t₁= 41.90 min, t₂=43.20 min. H NMR (300 MHz, CDCl₃) δ 7.32-7.18 (m, 5H) 5.58 (quin, 1H, J= 9.5 Hz) 5.12 (d, 1H, J=10. 3 Hz) 5.09 (d, 1H, J=17.2 Hz) 2.62 (quin, 1H, J=6.2 Hz) 2.09 (m, 1H) 1.20 (d, 3H, J=7.1 Hz) 1.08-0.95 (m, 2H) 0.77 (t, 3H, J=7.5 Hz). C NMR (101 MHz, CDCl₃) δ 146.6, 141.2, 128.2, 127.7, 125.8, 115.9, 53.10, 44, 25.4, 20.1, 11.8. HRMS (EI + mode) m/z expected: 174,1409 observed: 174,1410. [α] = +7.3 (c 1.33, CHCl₃).

1w. Reaction time: 12 h. Product isolated as colourless oil in 36 % yield, >99/1 anti/syn ratio, >99/1 S₂N₂/S₂N₂ ratio with 85 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. ee determined after lithium/bromide exchange and quench with water. GC: CP-CHIRASIL DEX CB: 60-0-1-170-5: t₁= 41.7 min, t₂= 42.9 min. H (NMR) (400 MHz, CDCl₃) δ 7.30-7.10 (m, 5H) 5.75 (s, 1H) 5.60 (s, 1H) 2.85-2.75 (m, 1H) 2 (td, 1H, J= 3.4 Hz and J=10.6 Hz) 1.35-1.25 (m, 4H) 1.20-1.10 (m, 1H) 0.75 (t, 3H, J= 7.4 Hz). C NMR (75 MHz, CDCl₃) δ 145.5, 138.6, 128.4, 127.8, 126.2, 118.8, 58.4, 43, 24, 21.1, 11.9. HRMS (EI + mode) m/z expected: 252.0514 observed: 252.0512.

1x. Reaction time: 12 h. Product isolated as colourless oil in 36 % yield, >99/1 syn/anti ratio, >99/1 S₂N₂/S₂N₂ ratio with 77 % ee after column chromatography on silica gel using 100 % pentane as solvent mixture. ee determined after lithium/bromide exchange and quench with water. GC: CP-CHIRASIL DEX CB: 60-0-1-170-5: t₁= 42.6 min, t₂= 43.4 min.
1H (NMR) (400 MHz, CDCl₃) δ 7.30-7.10 (m, 5H) 5.25 (s, 1H) 5.22 (s, 1H) 3-2.90 (m, 1H) 2.25 (td, 1H, J= 3.4 Hz and J=10.6 Hz) 1.76-1.31 (m, 5H) 0.90 (t, 3H, J= 7.4 Hz). 13C NMR (75 MHz, CDCl₃) δ 146, 138.1, 128.6, 128, 127.5, 126.8, 118.5, 58.3, 42.7, 22.8, 19, 12. HRMS (EI + mode) m/z expected: 252.0514 observed: 252.0512.

Application.

Into a conditioned Young Valve with Grubbs second generation catalyst (5 mol %) the desired catalysis adduct (0.2 mmol) is dissolved into 1 mL of dry and degassed dichloromethane. The flask is sealed and heated at reflux for 24 h. After cooling to room temperature the solvent is evaporated in vacuo and the crude material is purified via flash column chromatography using pentane (100 %) as eluant (0.5 cm diameter and 6 cm high column).

2a. Product isolated as colourless oil in 68 % yield, >99/1 anti/syn ratio with 82 % ee after column chromatography on silica gel using 100 % pentane as solvent. 1H (NMR) (400 MHz, CDCl₃) δ 7.40-7.10 (m, 5H) 5.75-5.65 (m, 2H) 2.60 (q, 1H, J=7.10 Hz) 2.35-2.25 (m, 2H) 1.60-1.50 (m, 1H) 1.25 (d, 3H, J=7.1 Hz) 1 (s, 3H). 13C NMR (75 MHz, CDCl₃) δ 145.2, 138.3, 129.4, 128.8, 128.7, 127.7, 127.6, 125.8, 52.6, 48.6, 35.9, 32, 25.5, 16.4. HRMS (EI + mode) m/z expected: 186,1409 observed: 186,1410. [α] = +7.8 (c 1.33, CHCl₃).

2b. Product isolated as colourless oil in 72 % yield, >99/1 anti/syn ratio with 84 % ee after column chromatography on silica gel using 100 % pentane as solvent. 1H (NMR) (400 MHz, CDCl₃) δ 7.40-7.20 (m, 5H) 5.80-5.70 (m, 2H) 2.8 (q, 1H, J=7.1 Hz) 1.90 (m, 2H) 1.70-1.60 (m, 4H) 1.30 (d, 3H, J=7.1 Hz) 0.90 (s, 3H). 13C NMR (75 MHz, CDCl₃) δ 144.7, 135.4, 129.2, 127.5, 125.9, 125.8, 48.2, 37.5, 33.7, 25.2, 24.4, 19.1, 15.6. HRMS (EI + mode) m/z expected: 200,1565 observed: 200,1566. [α] = +8.3 (c 1.33, CHCl₃).
2c. Product isolated as colourless oil in 67 % yield, >99/1 anti/syn ratio with 90 % ee after column chromatography on silica gel using 100 % pentane as solvent. $^1$H (NMR) (400 MHz, CDCl$_3$) δ 7.40-7.20 (m, 5H) 5.80-5.70 (m, 2H) 2.8 (q, 1H, J=7.10 Hz) 1.90-1.60 (m, 8H) 1.30 (d, 3H, J=7.1 Hz) 0.90 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) δ 144.7, 135.4, 129.2, 127.5, 125.9, 125.8, 48.2, 37.5, 33.7, 28, 25.2, 24.4, 19.1, 15.6. HRMS (EI + mode) m/z expected: 214,1722 observed: 214,1722. [α] = +11.2 (c 1.33, CHCl$_3$).

2d. Product isolated as colourless oil in 75 % yield, >99/1 anti/syn ratio with 90 % ee after column chromatography on silica gel using 100 % pentane as solvent. $^1$H (NMR) (400 MHz, CDCl$_3$) δ 7.40-7.10 (m,5H) 5.80-5.63 (m, 2H) 3.22 ( t, 1H, J= 8.3 Hz) 2.83-2.75 (m, 2H) 1.60-1.50 (m, 2H) 1 (t, 3H, J=7.4 Hz) 0.70 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) δ 142.2, 139.4, 130.4, 128.8, 127.9, 127.8, 127.6, 126, 52, 51.7, 37.8, 32.9, 22, 9.5. HRMS (EI + mode) m/z expected: 186,1409 observed: 186,1410. [α] = +8.7 (c 1.33, CHCl$_3$).

2e. Product isolated as colourless oil in 65 % yield, >99/1 anti/syn ratio with 91 % ee after column chromatography on silica gel using 100 % pentane as solvent. $^1$H (NMR) (400 MHz, CDCl$_3$) δ 7.40-7.10 (m,5H) 5.80-5.63 (m, 2H) 3.22 ( t, 1H, J= 8.3 Hz) 2.80-2.75 (m, 2H) 1.50-1.20 (m, 4H) 1 (t, 3H, J=7.4 Hz) 0.70 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) δ 143.8, 136.8, 129.2, 127.5, 125.9, 125.5, 45.6, 39.1, 32.6, 26.2, 25.3, 23.5, 8.6. HRMS (EI + mode) m/z expected: 200,1565 observed: 200,1566. [α] = +10.6 (c 1.33, CHCl$_3$).

2f. Product isolated as colourless oil in 62 % yield, >99/1 anti/syn ratio with 91 % ee after column chromatography on silica gel using 100 % pentane as solvent. $^1$H (NMR) (400 MHz, CDCl$_3$) δ 7.40-7.10 (m,5H) 5.80-5.63 (m, 2H) 3.22 ( t, 1H, J= 8.3 Hz) 2.85-2.75 (m, 2H) 1.50-1.10 (m, 6H) 1 (t, 3H, J=7.4 Hz) 0.70 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) δ 143.8, 136.8, 129.2, 127.5, 125.9, 125.5, 45.6, 39.1, 32.6, 27.4, 26.2, 25.3, 23.5, 8.6. HRMS (EI + mode) m/z expected: 214,1722 observed: 214,1720. [α] = +11.1 (c 1.33, CHCl$_3$).
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Acq On: 14.10.2013 06:46:46 PM
Sample: GR1483
Misc:
ALS vial: 1    Sample Multiplier: 1

Integration File: autoint1.e

Method: C:\MSDCHEM\2\METHODS\F60-1.M
Title:

Signal: FID1A.CH

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Sum of corrected areas: 19618986
### GC Analysis Data

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**Misc:**  
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**Title:**  
**Signal:** FID1A.CH  

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Acq On : 22-Oct-2013 06:59:03 PM
Sample : D1496
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Integration File: autoint1.e
Method : C:\MIDCHEM\METHODS\P60-1.M
Title : 
Signal : FIDIA.CH
peak R.T. Start End PK peak peak % of
# min min min TV height area % max. total
--- --- --- --- --- --- --- --- --- --- ---- ---- ----
1 58.024 57.624 58.271 M 92911 9808728 100.00% 95.427%
2 58.652 58.513 58.766 M 5559 455689 4.79% 4.573%
Sum of corrected areas: 9964417
Data Path: E:\GC 546 Janvier 2013 - Octobre 2013
Data File: gnl490.d
Signal(s): FID1A.CH
Acq On: 18.10.2013 01:24:18 PM
Sample: gnl490
Misc:
ALS Vial: 1 Sample Multiplier: 1
Integration File: autoint1.e
Method: C:\MSDCHEM\METHODS\F60-1.M
Title:

Signal: FID1A.CH

| peak | R.T. | Start | End  | Fk peak | peak | peak | % of  
|------|------|-------|------|---------|------|------|-------
|     | min  | min   | min  | TY height | area | % max. | total |
| 1   | 59.027 | 57.755 | 59.263 | M 93339 | 10123970 | 100.00% | 59.938% |
| 2   | 59.634 | 58.385 | 59.975 | M 93179 | 9751091 | 96.32% | 49.062% |

Sum of corrected areas: 19675061
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Acq On : 29.10.2013 09:07:30 PM
Sample : GRA1502
Misc :
ALS Vial : 1 Sample Multiplier : 1
Integration File : autoint1.e
Method : C:\MSOCHEM\2\METHODS\F69-1.M
Title :
Signal : FIDIA.CH

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Data File: DG1494.D 
Signal(s): FID1A.CH 
Acq On : 22.10.2013 09:17:11 AM 
Sample : DG1494 
Misc 
ALS Vial : 1  Sample Multiplier: 1 

Integration File: autoint1.e 
Method : C:\MSDCHEM\2\METHODS\F60-1.M 
Title : 
Signal : FID1A.CH 

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Acq On : 27.10.2013 05:33:54 PM
Sample : GR1499
Misc :
ALS Vial : 1 Sample Multiplier : 1

Integration File: autointl.e
Method : C:\MSDCHEM2\METHODS\F60-1.M
Title :

Signal : FID1A.CH

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Sample : GR1517
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Integration File: autoInt1.e

Method : C:\MSDChem\2\METHODS\F60-1.M
Title :

Signal : FIDIA.CH

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Sample : GR1520
Misc : 
ALS Vial : 1  Sample Multiplier: 1

Integration File: autoint1.exe

Method : C:\MSDCHEM\2\METHODS\F60-1.M
Title : 

Signal : FIDIA.CH

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Acq On: 08.11.2013 06:46:22 PM
Sample: 1518G
Misc:
ALS Vial: 3 Sample Multiplier: 1

Integration File: autoint.l.e

Method: C:\MSDCHM\2\METHODS\F60-1.M
Title:

Signal: FID1A.CH

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Sum of corrected areas: 12072365

60-1.M Thu Nov 14 17:55:52 2013
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<th>peak TY</th>
<th>height</th>
<th>area (arb. units)</th>
<th>% max. total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>68.993</td>
<td>68.782</td>
<td>69.180</td>
<td>M</td>
<td>67108</td>
<td>6538677</td>
<td>100.00%</td>
<td>51.557%</td>
</tr>
<tr>
<td>2</td>
<td>69.367</td>
<td>69.184</td>
<td>69.595</td>
<td>M</td>
<td>62583</td>
<td>6143855</td>
<td>93.96%</td>
<td>48.443%</td>
</tr>
</tbody>
</table>

Sum of corrected areas: 12682532
<table>
<thead>
<tr>
<th>#</th>
<th>R.T. min</th>
<th>Start min</th>
<th>End min</th>
<th>PK min</th>
<th>peak TY</th>
<th>height</th>
<th>area % max.</th>
<th>% total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>52.845</td>
<td>52.640</td>
<td>53.107</td>
<td>M</td>
<td>155698</td>
<td>15544692</td>
<td>100.00%</td>
<td>92.809%</td>
</tr>
<tr>
<td>2</td>
<td>53.417</td>
<td>53.283</td>
<td>53.561</td>
<td>M</td>
<td>14131</td>
<td>1204412</td>
<td>7.75%</td>
<td>7.191%</td>
</tr>
</tbody>
</table>

Sum of corrected areas: 16749105
<table>
<thead>
<tr>
<th>peak</th>
<th>R.T.</th>
<th>Start</th>
<th>End</th>
<th>PK peak</th>
<th>peak</th>
<th>peak</th>
<th>% of total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>52.850</td>
<td>52.665</td>
<td>53.095</td>
<td>M</td>
<td>54975</td>
<td>5718408</td>
<td>100.00%</td>
</tr>
<tr>
<td>2</td>
<td>53.421</td>
<td>53.189</td>
<td>53.924</td>
<td>M</td>
<td>48392</td>
<td>5047854</td>
<td>88.27%</td>
</tr>
</tbody>
</table>

Sum of corrected areas: 10766262
<table>
<thead>
<tr>
<th>#</th>
<th>R.T.</th>
<th>Start</th>
<th>End</th>
<th>PK</th>
<th>peak</th>
<th>peak</th>
<th>% of max. total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>75.548</td>
<td>75.332</td>
<td>75.860</td>
<td>M</td>
<td>62897</td>
<td>6532851</td>
<td>100.00% 91.791%</td>
</tr>
<tr>
<td>2</td>
<td>76.163</td>
<td>76.044</td>
<td>76.289</td>
<td>M</td>
<td>7245</td>
<td>584262</td>
<td>8.94% 8.209%</td>
</tr>
</tbody>
</table>

Sum of corrected areas: 7117112
<table>
<thead>
<tr>
<th>peak</th>
<th>R.T.</th>
<th>Start</th>
<th>End</th>
<th>PK</th>
<th>peak</th>
<th>peak</th>
<th>peak % of</th>
<th>% of total</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>min</td>
<td>min</td>
<td>min</td>
<td>TY</td>
<td>height</td>
<td>area</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>75.587</td>
<td>75.310</td>
<td>75.839</td>
<td>M</td>
<td>228687</td>
<td>24144654</td>
<td>100.00%</td>
<td>75.718%</td>
</tr>
<tr>
<td>2</td>
<td>76.190</td>
<td>75.993</td>
<td>76.457</td>
<td>M</td>
<td>77990</td>
<td>7743045</td>
<td>32.07%</td>
<td>24.282%</td>
</tr>
</tbody>
</table>

Sum of corrected areas: 31887699
Injection Date : 08-Oct-13 5:23:22 PM
Sample Name : GR1474
Acq. Operator : dg
Acq. Method : C:\HECHEM\METHODS\60-1-170.M
Last changed : 17-Jan-13 6:37:28 PM by DG
Analysis Method : C:\HECHEM\METHODS\140-150.M
Last changed : 18-Nov-13 1:01:48 PM by pg
(modified after loading)

---

Area Percent Report
---

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width [min]</th>
<th>Area [pA*s]</th>
<th>Height [pA]</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>68.832</td>
<td>BB</td>
<td>0.1248</td>
<td>656.95709</td>
<td>66.77818</td>
<td>91.86192</td>
</tr>
<tr>
<td>2</td>
<td>69.357</td>
<td>MM</td>
<td>0.1362</td>
<td>58.20006</td>
<td>7.12276</td>
<td>8.13808</td>
</tr>
</tbody>
</table>

---
Injection Date: 20-Sep-13 7:34:39 PM  
Sample Name: gt1415  
Seq. Line: 1  
Acq. Operator: DG  
Location: Vial 1  
Inj Volume: 1 µl  
Acq. Method: C:\RCHMA\METHODS\60-1.M  
Last changed: 19-Mar-13 8:33:43 PM by LH  
Analysis Method: C:\RCHMA\METHODS\40-150.M  
Last changed: 18-Nov-13 12:58:11 PM by pg  
(modified after loading)

FID1 A, (DGCHM415.Id)

Area Percent Report

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>#</th>
<th>Ret. Time (min)</th>
<th>Width (min)</th>
<th>Area [pA]</th>
<th>Height [pA]</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.1604</td>
<td>290.89505</td>
<td>30.21913</td>
<td>50.80902</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.1617</td>
<td>281.63138</td>
<td>29.02872</td>
<td>49.19098</td>
<td></td>
</tr>
</tbody>
</table>

Total 99.9%
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: PID λ,

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>64.813 MM</td>
<td>0.1750</td>
<td>351.20416</td>
<td>33.44104</td>
<td>93.28302</td>
</tr>
<tr>
<td>1</td>
<td>65.429 MM</td>
<td>0.1306</td>
<td>25.28898</td>
<td>3.22723</td>
<td>6.71698</td>
</tr>
</tbody>
</table>

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Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area [pA^s]</th>
<th>[pA]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>53.819</td>
<td>MM</td>
<td>0.1511</td>
<td>715.43890</td>
<td>78.89700</td>
<td>70.02864</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>54.384</td>
<td>MM</td>
<td>0.1470</td>
<td>306.19861</td>
<td>34.71799</td>
<td>29.97136</td>
<td></td>
</tr>
</tbody>
</table>
Injection Date : 01-Nov-13 9:08:47 AM  
Sample Name : DPEN  
Acq. Operator : DG  
Acq. Method : C:\HPChem\I\METHODS\60-1.M  
Last changed : 19-Mar-13 8:33:19 PM by LH  
Analysis Method : C:\HPChem\I\METHODS\140-150.M  
Last Changed : 18-Nov-13 12:53:56 PM by pg  
(modified after loading)

---

Area Percent Report
---

Sorted By : Signal  
Multiplier : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>78.877 MM</td>
<td>0.1997</td>
<td>1126.74939</td>
<td>94.03220</td>
<td>95.59128</td>
</tr>
<tr>
<td>2</td>
<td>79.318 MM</td>
<td>0.1425</td>
<td>51.96627</td>
<td>6.07661</td>
<td>4.40872</td>
</tr>
</tbody>
</table>

---
Injection Date: 21-Oct-13 7:57:51 PM
Sample Name: dgl493
Location: Vial 1
Acq. Operator: dg
Inj Volume: 1 μl
Acq. Method: C:\HPCHEM\1\METHODS\60-1.M
Analysis Method: C:\HPCHEM\1\METHODS\140-150.M
Last changed: 18-Nov-13 1:39:01 PM by pg
(modified after loading)

Area Percent Report

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>78.935</td>
<td>BV</td>
<td>0.1252</td>
<td>456.17191</td>
<td>47.86106</td>
<td>71.53850</td>
</tr>
<tr>
<td>2</td>
<td>79.306</td>
<td>VB</td>
<td>0.1177</td>
<td>181.48737</td>
<td>18.78806</td>
<td>28.46150</td>
</tr>
</tbody>
</table>

Signal: [Chemical Structure Image]
**Area Percent Report**

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type Width</th>
<th>Area</th>
<th>Height</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>56.995</td>
<td>MM 0.124</td>
<td>32.2</td>
<td>4.2</td>
<td>0.71</td>
</tr>
<tr>
<td>2</td>
<td>57.447</td>
<td>MM 0.1627</td>
<td>337</td>
<td>34.6</td>
<td>9.28</td>
</tr>
</tbody>
</table>
Injection Date: 20-Sep-13 9:35:26 PM  
Seq. Line: 2  
Sample Name: gr1425  
Sample Location: Vial 2  
Acq. Operator: DG  
Inj. Volume: 1 μl  
Acq. Method: C:\NFCHEM\METHODS\60-1,M  
Last changed: 19-Mar-13 8:33:19 PM by LH  
Analysis Method: C:\NFCHEM\METHODS\140-150,M  
Last changed: 18-Nov-13 1:39:48 PM by pg  
(modified after loading)

MIDA (DGGHR+45E)+

Area Percent Report

Sorted By: Signal  
Multiplier: 1.0000  
Dilution: 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,  
Peak Ret.Time Type Width Area Height Area %  
# (min) [min] [pA*s] [pA]  
--- --- ------ --- --- ---  
1 56.974 BB 0.1375 462.33231 41.72952 49.98594  
2 57.493 BB 0.1216 402.55859 39.92019 50.01406

---

49
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>#</th>
<th>Ret Time</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>55.227</td>
<td>0.116</td>
<td>8.46873</td>
<td>1.26523</td>
<td>4.13751</td>
</tr>
<tr>
<td>2</td>
<td>55.795</td>
<td>0.1716</td>
<td>196.21310</td>
<td>19.05769</td>
<td>95.86249</td>
</tr>
</tbody>
</table>
## Signal: FIDIA.CH

<table>
<thead>
<tr>
<th>Peak</th>
<th align="right">R.T.</th>
<th align="right">Start</th>
<th align="right">End</th>
<th>PK</th>
<th align="right">Peak</th>
<th align="right">Peak</th>
<th align="right">Peak</th>
<th align="right">% of</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td align="right">min</td>
<td align="right">min</td>
<td align="right">min</td>
<td>TY</td>
<td align="right">height</td>
<td align="right">area</td>
<td align="right">% max.</td>
<td align="right">total</td>
</tr>
<tr>
<td>------</td>
<td align="right">------</td>
<td align="right">------</td>
<td align="right">------</td>
<td>----</td>
<td align="right">--------</td>
<td align="right">------</td>
<td align="right">--------</td>
<td align="right">-------</td>
</tr>
<tr>
<td>1</td>
<td align="right">66.238</td>
<td align="right">66.067</td>
<td align="right">66.360</td>
<td>M</td>
<td align="right">33778</td>
<td align="right">3299134</td>
<td align="right">10.33%</td>
<td align="right">9.366%</td>
</tr>
<tr>
<td>2</td>
<td align="right">66.590</td>
<td align="right">66.372</td>
<td align="right">66.820</td>
<td>M</td>
<td align="right">306724</td>
<td align="right">31925474</td>
<td align="right">100.00%</td>
<td align="right">90.634%</td>
</tr>
</tbody>
</table>

Sum of corrected areas: 35224608
<table>
<thead>
<tr>
<th>Peak</th>
<th>R.T.</th>
<th>Start</th>
<th>End</th>
<th>PK</th>
<th>peak height</th>
<th>area %</th>
<th>max. %</th>
<th>total %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>66.197</td>
<td>65.964</td>
<td>66.328</td>
<td>M</td>
<td>39024</td>
<td>3846647</td>
<td>89.95%</td>
<td>47.356%</td>
</tr>
<tr>
<td>2</td>
<td>66.542</td>
<td>66.336</td>
<td>66.751</td>
<td>M</td>
<td>40564</td>
<td>4276210</td>
<td>100.00%</td>
<td>52.644%</td>
</tr>
</tbody>
</table>

Sum of corrected areas: 9122856
Injection Date: 28-Nov-13 6:39:10 PM
Sample Name: dg1535
Location: Vial 1
Acq. Operator: dg
Inj: 1
Inj Volume: 1 µl
Acq. Method: C:\\HPCHEM\\METHODS\\60-1.M
Last changed: 19-Mar-13 8:33:19 PM by LH
Analysis Method: C:\\HPCHEM\\METHODS\\110-1.M
Last changed: 01-Dec-13 4:32:12 PM by stef
(modified after loading)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal: FID1 A,

Peak RetTime Type Width Area Height Area %
# [min] [min] [pA*s] [pA] [pA] %
1 41.040 MM 0.1500 156.02570 17.43066 91.56020
2 42.994 MM 0.1239 15.31548 2.08620 8.43172
Totals: 181.4618 19.49880
Injection Date : 28-Nov-13 9:24:49 PM
Sample Name : dgj537
Acq. Operator : dg
Location : Vial 1
Inj Volume : 1 µl
Acq. Method : C:\HPChem\METHODS\60-1.M
Last changed : 19-Mar-13 8:33:19 PM by LH
Analysis Method : C:\HPChem\METHODS\110-1.M
Last changed : 01 Dec 13 9:32:12 PM by otof
(modified after loading)

FID1 A, (DGDG1537.D)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A.
Peak RetTime Type Width Area Height Area
# | [min] | [min] | [µA*s] | [µA] | %
---|-------|-------|--------|------|-----
1 | 41.788 BB | 0.1294 | 217.64574 | 22.78954 | 39.08769 |
2 | 42.687 BV | 0.1047 | 55.04097 | 7.49064 | 9.86498 |
3 | 43.863 VB | 0.1320 | 232.36348 | 21.89451 | 39.74896 |
4 | 43.599 BB | 0.1290 | 62.79955 | 7.11193 | 11.27837 |

[Chemical structure diagram]
Data Path: C:\MSDChem\2\DATA\nData File: GR1536.D
Signal(s): PID1A.CH
Acq On: 28.11.2013 05:40:21 PM
Sample: GR1536
Misc:
ALS Vial: 1 Sample Multiplier: 1

Integration File: autoint1.e

Method: C:\MSDCHEM\2\METHODS\F60-1.M
Title:

Signal: PID1A.CH

<table>
<thead>
<tr>
<th>Peak</th>
<th>R.T.</th>
<th>Start</th>
<th>End</th>
<th>PK peak</th>
<th>peak</th>
<th>peak</th>
<th>% of</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>min</td>
<td>min</td>
<td>min</td>
<td>TY height</td>
<td>area</td>
<td>% max.</td>
<td>total</td>
</tr>
<tr>
<td>1</td>
<td>66.565</td>
<td>66.359</td>
<td>66.752</td>
<td>43999</td>
<td>4603993</td>
<td>100.00%</td>
<td>95.527%</td>
</tr>
<tr>
<td>2</td>
<td>66.824</td>
<td>66.760</td>
<td>66.975</td>
<td>2783</td>
<td>215603</td>
<td>4.68%</td>
<td>4.473%</td>
</tr>
</tbody>
</table>

Sum of corrected areas: 4819595
**Integration File:** autoint1.e

**Method:** C:\MSDCHEM\2\METHODS\P60-1.M

**Signal:** FID1A.CH

<table>
<thead>
<tr>
<th>#</th>
<th>R.T.</th>
<th>Start</th>
<th>End</th>
<th>PK</th>
<th>peak TY</th>
<th>height</th>
<th>area</th>
<th>% max.</th>
<th>total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>66.572</td>
<td>66.336</td>
<td>66.692</td>
<td>M</td>
<td>90013</td>
<td>8940768</td>
<td>100.00%</td>
<td>64.421%</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>66.839</td>
<td>66.699</td>
<td>67.069</td>
<td>M</td>
<td>46301</td>
<td>4937948</td>
<td>55.23%</td>
<td>35.579%</td>
<td></td>
</tr>
</tbody>
</table>

*Sum of corrected areas: 13878716*
Data Path : C:\MSDChe\2\DATA\nData File : GR1532.D
Signal(s) : FID1A.CH
Acq On : 26.11.2013 04:11:16 PM
Sample : GR1532
Misc :
ALS Vial : 1 Sample Multiplier: 1

Integration File: autoint1.e
Method : C:\MSDCHEM\2\METHODS\P60-1.M
Title :
Signal : FID1A.CH

<table>
<thead>
<tr>
<th>peak</th>
<th>R.T.</th>
<th>Start</th>
<th>End</th>
<th>PK peak</th>
<th>peak</th>
<th>peak</th>
<th>% of</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>78.103</td>
<td>77.973</td>
<td>78.311</td>
<td>M 63892</td>
<td>6603782</td>
<td>100.00%</td>
<td>95.33%</td>
</tr>
<tr>
<td>2</td>
<td>78.350</td>
<td>78.331</td>
<td>78.544</td>
<td>M 4800</td>
<td>322892</td>
<td>4.89%</td>
<td>4.66%</td>
</tr>
</tbody>
</table>

Sum of correct areas: 6926674
Area Percent Report

Data Path : C:\NEDChem\2\DATA\Data File : GR1531.D
Signal(s) : PID1A.CH
Ago On : 20-11-2013 07:30:11 PM
Sample : GR1531
Misc : 
ALS Vial : 1 Sample Multiplier: 1
Integration File: autoint1.e
Method : C:\NEDCHEM\2\METHODS\P60-1.M
Title : PID1A.CH

<table>
<thead>
<tr>
<th>peak</th>
<th>R.T.</th>
<th>Start</th>
<th>End</th>
<th>PK peak</th>
<th>peak</th>
<th>peak</th>
<th>% of</th>
</tr>
</thead>
<tbody>
<tr>
<td>---</td>
<td>----</td>
<td>-----</td>
<td>---</td>
<td>--------</td>
<td>-----</td>
<td>-----</td>
<td>----</td>
</tr>
<tr>
<td>1</td>
<td>78.124</td>
<td>77.969</td>
<td>78.243</td>
<td>M 79611</td>
<td>#10076</td>
<td>100.00%</td>
<td>69.007%</td>
</tr>
<tr>
<td>2</td>
<td>78.361</td>
<td>78.263</td>
<td>78.557</td>
<td>M 36090</td>
<td>3548390</td>
<td>43.25%</td>
<td>30.19%</td>
</tr>
</tbody>
</table>

Sum of corrected areas: 11735085

P60-1.M Sun Dec 01 16:41:49 2013

![Chemical Structure](image)

![Graph](image)

Minutes

59
<table>
<thead>
<tr>
<th>Peak Name</th>
<th>RT</th>
<th>Area</th>
<th>% Area</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Peak1</td>
<td>4.216</td>
<td>264367</td>
<td>3.99</td>
<td>36891</td>
</tr>
<tr>
<td>2 Peak2</td>
<td>4.428</td>
<td>6364670</td>
<td>96.01</td>
<td>489515</td>
</tr>
</tbody>
</table>

![Graphical representation of peaks and a structural formula of a compound with a bromine (Br) atom]
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID A,

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>[min]</td>
<td>[min]</td>
<td>[pA*s]</td>
<td>[pA]</td>
</tr>
<tr>
<td>1</td>
<td>41.663</td>
<td>0.1890</td>
<td>460.329</td>
<td>40.58437</td>
</tr>
<tr>
<td>2</td>
<td>42.870</td>
<td>0.1157</td>
<td>36.90282</td>
<td>5.31766</td>
</tr>
</tbody>
</table>
Injection Date: 03-Jan-14 5:26:59 PM
Sample Name: 1576
Acq. Operator: dg
Location: Vial 1
Inj Volume: 1 µl
Acq. Method: C:\HPChem\1\METHODS\60-1-115.M
Last changed: 03-Jan-14 4:04:33 PM by dg
(modified after loading)
Analysis Method: C:\HPChem\1\METHODS\60-1.M
Last changed: 17-Jan-14 4:07:30 PM by pg
(modified after loading)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>Peak RetTime Type Width</th>
<th>Area [pA*s]</th>
<th>Height [pA]</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 42.625 MM 0.1238</td>
<td>67.93658</td>
<td>9.14555</td>
<td>11.38321</td>
</tr>
<tr>
<td>2 43.363 MM 0.2068</td>
<td>528.87720</td>
<td>42.61638</td>
<td>88.61679</td>
</tr>
</tbody>
</table>

Totals: 596.81376 51.76193