Supporting Information

Dehydroxylation of alcohols for nucleophilic substitution

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Content

1. General information .................................................................2

2. Evidence for the proposed mechanism .................................2

3. Screening reaction conditions of dehydroxy-amination of alcohols ..........4

4. General procedure for dehydroxy-functionalization of alcohols .............4

5. The synthesis of $^{18}$O-1a ..........................................................15

6. Isolation of Ph$_3$P=O ..............................................................17

7. Inversion of configuration ....................................................17

8. References and notes ..........................................................20

9. Copies of $^1$H NMR, $^{19}$F NMR, $^{13}$C NMR and $^{31}$P NMR spectra ..............22
1. General information

Solvents and reagents were purchased from commercial sources and used as received unless otherwise noted. \(^1\)H, \(^{13}\)C, \(^{19}\)F and \(^{31}\)P NMR spectra were detected on a 500 MHz, 400MHz or 300 MHz NMR spectrometer. Data for \(^1\)H NMR, \(^{13}\)C NMR, \(^{19}\)F NMR and \(^{31}\)P NMR were recorded as follows: chemical shift (\(\delta\), ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, coupling constant \((J)\) in Hz). Mass spectra were obtained on a GC-MS or LC-MS. High resolution mass data were recorded on a high resolution mass spectrometer in the EI, ESI or DART mode.

2. Evidence for the proposed mechanism

Usually, \(\text{Ph}_3\text{P}\) would readily undergo alkylation with alkyl iodides to give alkylphosphonium salts. Surprisingly, \(\text{Ph}_3\text{P}\) was almost completely transformed into \(\text{Ph}_3\text{P} = \text{O}\), and neither monophosphonium salt \((\text{Ph}_3\text{P}^+\text{CH}_2\text{CH}_2\text{I}^-)\) nor diphosphonium salt \((\text{Ph}_3\text{P}^+\text{CH}_2\text{CH}_2\text{P}^+\text{Ph}_3^-\text{I}^-)\) was observed in the dehydroxy-functionalization reaction. Furthermore, diphosphonium salt \((\text{Ph}_3\text{P}^+\text{CH}_2\text{CH}_2\text{P}^+\text{Ph}_3^-\text{I}^-)\) could not promote dehydroxy-functionalization, suggesting that the alkylation process did not occur. NMR spectroscopy was then used to monitor the reaction of \(\text{Ph}_3\text{P}\) with \(\text{ICH}_2\text{CH}_2\text{I}\). Mixing \(\text{Ph}_3\text{P}\) (0.2 mmol) and \(\text{ICH}_2\text{CH}_2\text{I}\) (0.2 mmol) in DMF (2 mL) at room temperature would immediately lead to the rapid evolution of gas and the complete conversion of both \(\text{ICH}_2\text{CH}_2\text{I}\) and \(\text{Ph}_3\text{P}\). \(^1\)H NMR analysis of the reaction mixture revealed that \(\text{ICH}_2\text{CH}_2\text{I}\) was converted into ethylene \((\text{CH}_2=\text{CH}_2)\) (Figure S1). \(\text{Ph}_3\text{P}\) was transformed into two new species (A and B), the signals of which detected by \(^{31}\)P NMR spectroscopy appeared at 25.5 ppm and 11.9 ppm, respectively. The signal at 25.5 ppm was assigned to \(\text{Ph}_3\text{P} = \text{O}\), as the intensity of this signal in \(^{31}\)P NMR spectrum was increased by external addition of \(\text{Ph}_3\text{P} = \text{O}\) into the system. Intermediate B is so fragile that the attempts at its isolation or further characterization were unsuccessful. Its structure is proposed to be a pentacoordinate species (Figure S1). \(\text{Ph}_3\text{P} = \text{O}\) should be formed from this species with the simultaneous formation of Vilsmeier–Haack type intermediate C. After intermediates A and B were generated, the subsequent addition of phenylbutanol and sodium phenolate gave the desired product in 28% yield, indicating that both species B and C are key intermediates of the reaction. This low yield is because these two species are unstable and would readily undergo side reactions in the absence of substrates. If substrates are added before intermediates B and C are formed, high yields would be obtained, as shown in Tables 1 and 2.
Figure S1. The reaction of Ph₃P with ICH₂CH₂I in DMF. *The yield was determined by ¹H NMR spectroscopy.

The reaction of Ph₃P with ICH₂CH₂I to furnish intermediate B occurs so fast that no direct evidence could be collected to elucidate this process. Fortunately, we found that the conversion in chloroform proceeded slowly. Therefore, the reaction in CDCl₃ in a NMR tube was monitored by ³¹P NMR spectroscopy. New phosphorus species D and E were detected immediately after mixing Ph₃P and ICH₂CH₂I (Figure S2-A). They were too unstable to be isolated for characterization, and thus their structures were determined based on their ³¹P NMR signals. The formation of intermediates D and E should be because of a strong P-I halogen bond.¹ In intermediate D, the phosphorus atom carries slightly positive charge, therefore a downfield shift was observed in ³¹P NMR spectroscopy. Intermediate E is close to a pentacoordinate phosphorus species, thus its signal would move upfield.² This intermediate could be either a linear or a cyclic structure, depending on the interaction of terminal Ph₃P and CH₂I moiety. Intermediates D and E were slowly transformed into two new species F and G, with ³¹P NMR signals appearing at +45.8 ppm and -18.7 ppm, respectively (Figure S2-B). F and G were determined to be Ph₃PI₂ and [Ph₃P⁺I⁻I⁻], respectively, based on the reported phosphorus signals of Ph₃PI₃ and [Ph₃P⁺I⁻I⁻].³⁴

Figure S2-A

Figure S2-B

Figure S2. The reaction of Ph₃P with ICH₂CH₂I in CDCl₃.
3. Screening reaction conditions of dehydroxy-amination of alcohols

\[
\begin{align*}
\text{Ph} & \text{-} \text{OH} + \text{Ph} & \text{N} & \text{Me} \quad \text{Ph}_3\text{P} & \text{ICH}_2\text{CH}_2\text{I} \\
\text{DMF, T, t} & \quad \rightarrow \text{Ph} & \text{N} & \text{Me} & \text{Ph} \\
\end{align*}
\]

<table>
<thead>
<tr>
<th>Entry</th>
<th>Ratio(^a)</th>
<th>T (°C)</th>
<th>t (hour)</th>
<th>Yield (%)(^b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1:3:1.2:1.2</td>
<td>r.t.</td>
<td>10 h</td>
<td>66</td>
</tr>
<tr>
<td>2</td>
<td>1:3:1.2:1.2</td>
<td>50</td>
<td>10 h</td>
<td>63</td>
</tr>
<tr>
<td>3</td>
<td>1:3:1.2:1.2</td>
<td>80</td>
<td>10 h</td>
<td>62</td>
</tr>
<tr>
<td>4</td>
<td>1:3.5:1.2:1.2</td>
<td>r.t.</td>
<td>10 h</td>
<td>68</td>
</tr>
<tr>
<td>5</td>
<td>1:4:1.2:1.2</td>
<td>r.t.</td>
<td>10 h</td>
<td>75</td>
</tr>
<tr>
<td>6</td>
<td>1:4:1.1:1.1</td>
<td>r.t.</td>
<td>10 h</td>
<td>55</td>
</tr>
<tr>
<td>7</td>
<td>1:4:1.3:1.3</td>
<td>r.t.</td>
<td>10 h</td>
<td>70</td>
</tr>
<tr>
<td>8</td>
<td>1:4:1.5:1.5</td>
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<td>10 h</td>
<td>72</td>
</tr>
<tr>
<td>9</td>
<td>1:4:1.2:1.2</td>
<td>r.t.</td>
<td>2</td>
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<tr>
<td>10</td>
<td>1:4:1.2:1.2</td>
<td>r.t.</td>
<td>4</td>
<td>45</td>
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<tr>
<td>11</td>
<td>1:4:1.2:1.2</td>
<td>r.t.</td>
<td>6</td>
<td>56</td>
</tr>
</tbody>
</table>

Reaction conditions: 1 (0.5 mmol), 2, Ph\(_3\)P and ICH\(_2\)CH\(_2\)I in DMF (5 mL). \(^a\)Molar ratio of 1:2:Ph\(_3\)P:ICH\(_2\)CH\(_2\)I. \(^b\)Determined by \(^1\)H NMR with the use of anisole as an internal standard.

4. General procedure for dehydroxy-functionalization of alcohols

4.1. General procedure for dehydroxylation of alcohols to form C-N and C-S bonds

Alcohol 1a (1.0 equiv, 0.5 mmol, 92.1 mg), triphenylphosphine (1.2 equiv, 0.6 mmol, 157.4 mg) and anhydrous DMF (5.0 mL) were added into a 10 mL sealed tube under a N\(_2\) atmosphere. 1,2-Diiodoethane (1.2 equiv, 0.6 mmol, 169.1 mg) was then added and the resulting mixture was stirred for around 1 min until the 1,2-diiodoethane was completely dissolved. Amine 2a (3.0 equiv, 1.5 mmol, 181.8 mg) was added subsequently and the mixture was stirred at room temperature for 10-12 h. Dichloromethane (20 mL) was added and the resulting solution was washed with water (20 mL x 3). The organic layer was dried over Na\(_2\)SO\(_4\). After filtration, the solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography to afford the pure product 3a.

\[
\begin{align*}
\text{Ph} & \text{-} \text{N} & \text{Me} & \text{Ph} \\
\end{align*}
\]

1-[[1,1’-biphenyl]-4-yl]-N-benzyl-N-methylmethanamine (3a):\(^5\) 96%; white solid. \(^1\)H NMR (400 MHz, chloroform-\(d\)) \(\delta\) 7.64 – 7.55 (m, 4H), 7.49 – 7.38 (m, 6H), 7.38 –
7.31 (m, 3H), 7.30 – 7.23 (m, 1H), 3.58 (s, 2H), 3.57 (s, 2H), 2.24 (s, 3H). LC-MS (ESI) Calcd. for C$_{21}$H$_{22}$N [M+H]$^+$ 288.2; found 288.2.

$\text{Ph}^-\text{N}^-\text{Ph}$

$N$-[[1,1'-biphenyl]-4-ylmethyl]aniline (3b). 4 equiv of N-nucleophile was used as the nucleophile; the reaction temperature was 50 °C. 90%; white solid. $^1$H NMR (400 MHz, chloroform- $d$) $\delta$ 7.57 (t, $J = 7.0$ Hz, 4H), 7.46 – 7.39 (m, 4H), 7.33 (t, $J = 7.3$ Hz, 1H), 7.18 (t, $J = 7.8$ Hz, 2H), 6.72 (t, $J = 7.3$ Hz, 1H), 6.65 (d, $J = 7.9$ Hz, 2H), 4.36 (s, 2H), 4.05 (s, 1H).

$\text{Ph}^-\text{N}^-\text{Me}$

$N$-[[1,1'-biphenyl]-4-ylmethyl]-4-methylaniline (3c). 4 equiv of N-nucleophile was used as the nucleophile; the reaction temperature was 50 °C. 94%; white solid. m.p. 122 – 123 °C. $^1$H NMR (400 MHz, chloroform- $d$) $\delta$ 7.62 – 7.52 (m, 4H), 7.49 – 7.39 (m, 4H), 7.39 – 7.29 (tt, $J = 7.4$, 1.6 Hz, 1H), 6.99 (d, $J = 8.0$ Hz, 2H), 6.59 (d, $J = 8.4$ Hz, 2H), 4.35 (s, 2H), 3.94 (s, 1H), 2.24 (s, 3H). $^{13}$C NMR (101 MHz, chloroform-$d$) $\delta$ 145.9, 140.9, 140.1, 138.8, 129.8, 128.8, 127.9, 127.4, 127.2, 127.1, 126.8, 113.0, 48.3, 20.4. IR (neat) $\nu$ = 3392, 2924, 2852, 1612, 1520, 1406, 1928, 1156, 1080, 826, 815, 771, 727, 697, 512 cm$^{-1}$. HRMS (EI) Calcd. for C$_{20}$H$_{19}$N $[M]^+$: 273.1517; found 273.1517.

$\text{Ph}^-\text{N}^-\text{Cl}$

$N$-[[1,1'-biphenyl]-4-ylmethyl]-4-chloroaniline (3d). 4 equiv of N-nucleophile was used as the nucleophile; the reaction temperature was 50 °C. 82%; white solid. m.p. 124 – 125 °C. $^1$H NMR (400 MHz, chloroform- $d$) $\delta$ 7.68 – 7.51 (m, 4H), 7.50 – 7.38 (m, 4H), 7.35 (t, $J = 7.3$ Hz, 1H), 7.12 (d, $J = 8.7$ Hz, 2H), 6.57 (d, $J = 8.7$ Hz, 2H), 4.34 (s, 2H), 4.09 (s, 1H). $^{13}$C NMR (101 MHz, chloroform-$d$) $\delta$ 146.6, 140.8, 140.4, 138.0, 129.1, 128.8, 127.8, 127.45, 127.35, 127.1, 122.2, 114.0, 48.1. IR (neat) $\nu$ = 3383, 3026, 2843, 1594, 1494, 1400, 1339, 1157, 1077, 1006, 815, 762, 689, 669, 505 cm$^{-1}$. HRMS (EI) Calcd. for C$_{19}$H$_{16}$NCl $[M]^+$: 293.0971; found 293.0963.

$\text{Ph}^-\text{Me}$

$N$-[[1,1'-biphenyl]-4-ylmethyl]-N-methylaniline (3e). 4 equiv of N-nucleophile was used as the nucleophile; the reaction temperature was 50 °C. quantitative; white solid.
$^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.63 – 7.52 (m, 4H), 7.44 (t, $J = 7.6$ Hz, 2H), 7.39 – 7.28 (m, 3H), 7.29 – 7.17 (m, 2H), 6.80 (d, $J = 8.0$ Hz, 2H), 6.74 (t, $J = 7.3$ Hz, 1H), 4.59 (s, 2H), 3.06 (s, 3H).

4-azidomethyl)-1,1'-biphenyl (3f). $^7$ NaN$_3$ was used as the nucleophile. Quantitative; white solid. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.65 – 7.52 (m, 4H), 7.49 – 7.42 (m, 2H), 7.41 – 7.31 (m, 3H), 4.38 (s, 2H).

N-(4-phenylbutyl)aniline (3h). $^9$ 4 equiv of N-nucleophile was used as the nucleophile; the reaction temperature was 50 °C. 71%; yellow liquid. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.32 – 7.25 (m, 2H), 7.25 – 7.12 (m, 5H), 6.68 (t, $J = 7.1$ Hz, 1H), 6.58 (d, $J = 8.1$ Hz, 2H), 3.57 (s, 1H), 3.10 (t, $J = 6.9$ Hz, 2H), 2.66 (t, $J = 7.4$ Hz, 2H), 1.80 – 1.59 (m, 4H).

N-benzyl-N-methyl-4-phenylbutan-1-amine (3i). $^10$ 4 equiv of N-nucleophile was used as the nucleophile. 59%; colorless liquid. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.34 – 7.28 (m, 4H), 7.28 – 7.21 (m, 3H), 7.20 – 7.13 (m, 3H), 3.47 (s, 2H), 2.61 (t, $J = 7.5$ Hz, 2H), 2.38 (t, $J = 7.2$ Hz, 2H), 2.17 (s, 3H), 1.73 – 1.48 (m, 4H).

4-methyl-N-(4-phenylbutyl)aniline (3j). 4 equiv of N-nucleophile was used as the nucleophile; the reaction temperature was 50 °C. 74%; white solid. m.p. 32 – 33 °C. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.32 – 7.25 (m, 2H), 7.22 – 7.11 (m, 3H), 6.98 (d, $J = 8.0$ Hz, 2H), 6.52 (d, $J = 8.3$ Hz, 2H), 3.44 (s, 1H), 3.10 (t, $J = 6.9$ Hz, 2H), 2.65 (t, $J = 7.4$ Hz, 2H), 2.23 (s, 3H), 1.79 – 1.58 (m, 4H). $^{13}$C NMR (101 MHz, chloroform-$d$) $\delta$ 146.2, 142.2, 129.7, 128.4, 128.3, 126.4, 125.8, 112.9, 44.2, 35.7,
29.2, 29.0, 20.4. IR (neat) ν = 3406, 3061, 3025, 2935, 2857, 1600, 1502, 1475, 1401, 1320, 1248, 1177, 1090, 814, 748, 699, 669, 504 cm⁻¹. HRMS (EI) Calcd. for C₁₇H₂₁N [M]+: 239.1674; found 239.1681.

4-chloro-N-(4-phenylbutyl)aniline (3k). 4 equiv of N-nucleophile was used as the nucleophile; the reaction temperature was 50 °C. 53%; yellow liquid. ¹H NMR (400 MHz, chloroform-d) δ 7.29 (t, J = 7.4 Hz, 2H), 7.23 – 7.15 (m, 3H), 7.10 (d, J = 8.5 Hz, 2H), 6.49 (d, J = 8.4 Hz, 2H), 3.57 (s, 1H), 3.09 (t, J = 7.0 Hz, 2H), 2.66 (t, J = 7.5 Hz, 2H), 1.81 – 1.57 (m, 4H). ¹³C NMR (101 MHz, chloroform-d) δ 147.0, 142.1, 129.0, 128.4, 125.9, 121.6, 113.7, 44.0, 35.6, 29.0, 28.9. IR (neat) ν = 3415, 3261, 3061, 3025, 2935, 2857, 1600, 1502, 1475, 1401, 1320, 1248, 1177, 1090, 814, 748, 699, 669, 504 cm⁻¹. HRMS (EI) Calcd. for C₁₆H₁₈NCl [M]+: 259.1128; found 259.1123.

N-methyl-N-(4-phenylbutyl)aniline (3l).⁴ 4 equiv of N-nucleophile was used as the nucleophile; the reaction temperature was 50 °C. 80%; yellow liquid. ¹H NMR (400 MHz, chloroform-d) δ 7.32 – 7.26 (m, 2H), 7.26 – 7.13 (m, 5H), 6.72 – 6.64 (m, 3H), 3.33 (t, J = 6.9 Hz, 2H), 2.91 (s, 3H), 2.64 (t, J = 7.3 Hz, 2H), 1.73 – 1.56 (m, 4H).

(4-azidobutyl)benzene (3m).¹² NaN₃ was used as the nucleophile. Irrespective of whether a base (2 equiv of 2,6-lutidine) was used or not, a quantitative yield was obtained. If the base is used, it should be added after NaN₃. colorless liquid. ¹H NMR (400 MHz, chloroform-d) δ 7.28 (t, J = 7.4 Hz, 2H), 7.22 – 7.14 (m, 3H), 3.27 (t, J = 6.6 Hz, 2H), 2.64 (t, J = 7.4 Hz, 2H), 1.77 – 1.57 (m, 4H).

(3-azidobutyl)benzene (3n).¹³ NaN₃ was used as the nucleophile. 98%; colorless liquid. ¹H NMR (400 MHz, chloroform-d) δ 7.28 (t, J = 7.4 Hz, 2H), 7.22 – 7.15 (m, 3H), 3.43 (h, J = 6.6 Hz, 1H), 2.80 – 2.59 (m, 2H), 1.88 – 1.69 (m, 2H), 1.28 (d, J = 6.5 Hz, 3H).
1-(4-phenylbutyl)-1H-imidazole (3o). 14 equiv of N-nucleophile was used as the nucleophile; the reaction temperature was 50 °C. 82%; yellow liquid. \(^1\)H NMR (400 MHz, chloroform-\(d\)) \(\delta\) 7.92 (s, 1H), 7.34 – 7.25 (m, 2H), 7.20 (tt, \(J = 7.3, 1.5\) Hz, 1H), 7.17 – 7.10 (m, 3H), 6.92 (s, 1H), 3.96 (t, \(J = 7.2\) Hz, 2H), 2.64 (t, \(J = 7.2\) Hz, 2H), 1.82 (p, \(J = 7.3\) Hz, 2H), 1.63 (p, \(J = 7.5\) Hz, 2H).

![Structure of 1-(4-phenylbutyl)-1H-imidazole (3o)](image)

([1,1′-biphenyl]-4-ylmethyl)(phenyl)sulfane (3p). PhSNa\(^+\) was used as the nucleophile. 93%; white solid. \(^1\)H NMR (400 MHz, chloroform-\(d\)) \(\delta\) 7.56 (d, \(J = 7.5\) Hz, 2H), 7.51 (d, \(J = 8.2\) Hz, 2H), 7.42 (t, \(J = 7.7\) Hz, 2H), 7.38 – 7.29 (m, 5H), 7.29 – 7.22 (m, 3H), 7.21 – 7.14 (m, 1H), 4.15 (s, 2H). GC-MS (El) Calcd. for C\(_{19}\)H\(_{16}\)S [M]\(^+\): 276.1; found 276.1.

![Structure of ([1,1′-biphenyl]-4-ylmethyl)(phenyl)sulfane (3p)](image)

([1,1′-biphenyl]-4-ylmethyl)(4-(trifluoromethyl)phenyl)sulfane (3q): 85%; white solid. m.p. 161 – 162 °C. \(^1\)H NMR (400 MHz, chloroform-\(d\)) \(\delta\) 7.63 – 7.52 (m, 4H), 7.49 (d, \(J = 8.2\) Hz, 2H), 7.47 – 7.31 (m, 7H), 4.22 (s, 2H). \(^19\)F NMR (376 MHz, chloroform-\(d\)) \(\delta\) -62.45 (s, 3F). \(^13\)C NMR (101 MHz, chloroform-\(d\)) \(\delta\) 142.0, 142.0, 140.55, 140.48, 135.4, 129.2, 128.8, 128.0, 127.8 (q, \(J = 33.3\) Hz), 127.4, 127.0, 125.7 (q, \(J = 3.8\) Hz), 124.1 (q, \(J = 272.7\) Hz), 37.4. IR (neat) \(\nu\) = 1654, 1403, 1322, 1176, 1157, 1118, 845, 823, 773, 745, 689, 590, 495 cm\(^{-1}\). HRMS (El) Calcd. for C\(_{20}\)H\(_{15}\)F\(_3\)S [M]\(^+\): 344.0847; found 344.0835.

![Structure of ([1,1′-biphenyl]-4-ylmethyl)(4-(trifluoromethyl)phenyl)sulfane (3q)](image)

phenyl(4-phenylbutyl)sulfane (3r). PhSNa\(^+\) was used as the nucleophile. 95%; colorless liquid. \(^1\)H NMR (400 MHz, chloroform-\(d\)) \(\delta\) 7.34 – 7.23 (m, 4H), 7.21 – 7.13 (m, 4H), 2.93 (t, \(J = 7.1\) Hz, 2H), 2.62 (t, \(J = 7.4\) Hz, 2H), 1.85 – 1.62 (m, 4H). GC-MS (El) Calcd. for C\(_{16}\)H\(_{18}\)S [M]\(^+\): 242.1; found 242.1.

![Structure of phenyl(4-phenylbutyl)sulfane (3r)](image)

(4-methoxyphenyl)(4-phenylbutyl)sulfane (3s): 96%; colorless liquid. \(^1\)H NMR (400 MHz, chloroform-\(d\)) \(\delta\) 7.36 – 7.22 (m, 4H), 7.21 – 7.09 (m, 3H), 6.83 (d, \(J = 8.7\) Hz, 2H), 3.79 (s, 3H), 2.83 (t, \(J = 7.2\) Hz, 2H), 2.60 (t, \(J = 7.6\) Hz, 2H), 1.73 (p, \(J = 7.0\) Hz, 2H), 1.62 (p, \(J = 7.2\) Hz, 2H). \(^13\)C NMR (101 MHz, chloroform-\(d\)) \(\delta\) 158.8, 142.2, 133.2, 128.4, 128.3, 126.7, 125.8, 114.5, 55.3, 35.8, 35.4, 30.4, 28.8. IR (neat) \(\nu\) = 3061, 3025, 3001, 2934, 2855, 2835, 1592, 1570, 1493, 1461, 1440, 1284, 1245, 1179,
(4-phenylbutyl)(4-(trifluoromethyl)phenyl)sulfane (3t): quantitative; colorless liquid. 

$^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.49 (d, $J = 8.2$ Hz, 2H), 7.36 – 7.22 (m, 4H), 7.23 – 7.10 (m, 3H), 2.97 (t, $J = 7.0$ Hz, 2H), 2.64 (t, $J = 7.3$ Hz, 2H), 1.90 – 1.62 (m, 4H). $^{19}$F NMR (376 MHz, chloroform-$d$) $\delta$ -62.41 (s, 3F). $^{13}$C NMR (101 MHz, chloroform-$d$) $\delta$ 142.5, 142.5, 141.8, 128.4, 127.3, 127.2 (q, $J = 33.3$ Hz), 125.9, 125.6 (q, $J = 3.8$ Hz), 124.2 (q, $J = 272.7$ Hz), 35.3, 32.4, 30.4, 28.3. IR (neat) $\nu = 3086, 3063, 3027, 2934, 2858, 1607, 1497, 1454, 1402, 1328, 1166, 1126, 1095, 1030, 1013, 824, 745, 699, 590, 494$ cm$^{-1}$. HRMS (EI) Calcd. for C$_{17}$H$_{20}$OS [M$^+$]: 272.1235; found 272.1229.

phenyl(4-phenylbutan-2-yl)sulfane (3u).$^{16}$ PhS$^-$Na$^+$ was used as the nucleophile. 40%; colorless liquid. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.39 – 7.33 (m, 2H), 7.31 – 7.24 (m, 4H), 7.24 – 7.14 (m, 4H), 3.20 (h, $J = 6.7$ Hz, 1H), 2.87 – 2.70 (m, 2H), 1.99 – 1.87 (m, 1H), 1.87 – 1.76 (m, 1H), 1.32 (d, $J = 6.7$ Hz, 3H).

dodecyl(4-phenylbutyl)sulfane (3v): 80%; colorless liquid. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.30 – 7.23 (m, 2H), 7.20 – 7.12 (m, 3H), 2.62 (t, $J = 7.5$ Hz, 2H), 2.51 (t, $J = 7.3$ Hz, 2H), 2.47 (t, $J = 7.4$ Hz, 2H), 1.77 – 1.66 (m, 2H), 1.66 – 1.58 (m, 2H), 1.58 – 1.50 (m, 2H), 1.40 – 1.19 (m, 18H), 0.87 (t, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 142.3, 128.4, 128.3, 125.7, 35.5, 32.2, 32.0, 31.9, 30.6, 29.7, 29.68, 29.65, 29.63, 29.56, 29.4, 29.28, 29.26, 29.0, 22.7, 14.1. IR (neat) $\nu = 3085, 3062, 3026, 2925, 2853, 1604, 1496, 1454, 1377, 1261, 1030, 743, 698$ cm$^{-1}$. HRMS (EI) Calcd. for C$_{22}$H$_{38}$S [M$^+$]: 334.2694; found 334.2699.

4.2. General procedure for dehydroxylation of alcohols to form C-O and C-X (X = Cl, Br, I) bonds

Alcohol 1a (1.0 equiv, 0.5 mmol, 92.1 mg), triphenylphosphone (1.2 equiv, 0.6 mmol, 157.4 mg) and anhydrous DMF (5.0 mL) were added into a 10 mL sealed tube under a N$_2$ atmosphere. 1,2-Diiodooethane (1.2 equiv, 0.6 mmol, 169.1 mg) was then added and the resulting mixture was stirred for around 1 min until the 1,2-diiodoethane was completely dissolved. Sodium phenolate 4a (3.0 equiv, 1.5 mmol, 174.1 mg) was added subsequently and the mixture was stirred at room temperature for 10-12 h.
Dichloromethane (20 mL) was added and the resulting solution was washed with water (20 mL x 3). The organic layer was dried over Na$_2$SO$_4$. After filtration, the solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography to afford the pure product 5a.

4-(phenoxy)methyl)-1,1'-biphenyl (5a):$^{17}$ 98%; white solid. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.60 (t, $J = 7.4$ Hz, 4H), 7.51 (d, $J = 8.1$ Hz, 2H), 7.44 (t, $J = 7.5$ Hz, 2H), 7.38 – 7.26 (m, 3H), 7.04 – 6.93 (m, 3H), 5.10 (s, 2H). GC-MS (EI) Calcd. for C$_{19}$H$_{16}$O [M]$^+$ 260.1; found 260.1.

4-((4-methoxyphenoxy)methyl)-1,1'-biphenyl (5b): 83%; white solid. m.p. 162 – 163 ºC. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.64 – 7.57 (m, 4H), 7.50 (d, $J = 8.0$ Hz, 2H), 7.44 (t, $J = 7.5$ Hz, 2H), 7.35 (t, $J = 7.3$ Hz, 1H), 6.94 (d, $J = 9.0$ Hz, 2H), 6.85 (d, $J = 9.1$ Hz, 2H), 5.05 (s, 2H), 3.77 (s, 3H). $^{13}$C NMR (101 MHz, chloroform-$d$) $\delta$ 154.0, 153.0, 140.9, 140.8, 136.3, 128.8, 128.0, 127.37, 127.35, 127.2, 115.9, 114.7, 70.5, 55.7. IR (neat) $\nu$ = 2995, 2958, 2927, 2833, 1506, 1488, 1438, 1407, 1388, 1290, 1232, 1220, 1178, 1127, 1112, 1039, 1016, 877, 824, 767, 745, 717, 688, 524 cm$^{-1}$. HRMS (EI) Calcd. for C$_{20}$H$_{18}$O$_2$ [M]$^+$: 290.1307; found 290.1310.

4-((4-nitrophenoxy)methyl)-1,1'-biphenyl (5c):$^{18}$ 76%; yellow solid. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 8.21 (d, $J = 9.1$ Hz, 2H), 7.63 (d, $J = 8.1$ Hz, 2H), 7.59 (d, $J = 7.5$ Hz, 2H), 7.52 – 7.41 (m, 4H), 7.36 (t, $J = 7.3$ Hz, 1H), 7.04 (d, $J = 9.1$ Hz, 2H), 5.19 (s, 2H).

4-(methoxymethyl)-1,1'-biphenyl (5d):$^{19}$ 92%; colorless liquid. $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.62 – 7.54 (m, 4H), 7.47 – 7.38 (m, 4H), 7.34 (tt, $J = 7.3$, 1.5 Hz, 1H), 4.50 (s, 2H), 3.42 (s, 3H).
[1,1′-biphenyl]-4-ylmethyl acetate (5e). CH₃CO₂K⁺ was used as the nucleophile. 99%; colorless liquid. ¹H NMR (400 MHz, chloroform-d) δ 7.62 – 7.55 (m, 4H), 7.48 – 7.40 (m, 4H), 7.38 – 7.32 (m, 1H), 5.15 (s, 2H), 2.12 (s, 3H).

Methyl 4-(acetoxy)methyl)benzoate (5f). CH₃CO₂K⁺ was used as the nucleophile. 96%; colorless liquid. ¹H NMR (400 MHz, Chloroform-d) δ 8.01 (d, J = 8.2 Hz, 2H), 7.39 (d, J = 8.2 Hz, 2H), 5.13 (s, 2H), 3.90 (s, 3H), 2.11 (s, 3H).

[1,1′-biphenyl]-4-ylmethyl benzoate (5g): quantitative; white solid. ¹H NMR (400 MHz, chloroform-d) δ 8.14 – 8.06 (m, 2H), 7.64 – 7.57 (m, 4H), 7.57 – 7.50 (m, 3H), 7.48 – 7.41 (m, 4H), 7.35 (tt, J = 7.3, 1.6 Hz, 1H), 5.41 (s, 2H).

(4-phenoxybutyl)benzene (5h): quantitative; white solid. ¹H NMR (400 MHz, chloroform-d) δ 7.32 – 7.23 (m, 4H), 7.23 – 7.15 (m, 3H), 6.96 – 6.86 (m, 3H), 3.96 (t, J = 5.9 Hz, 2H), 2.69 (t, J = 7.0 Hz, 2H), 1.96 – 1.67 (m, 4H).

1-methoxy-4-(4-phenylbutoxy)benzene (5i): quantitative; colorless liquid. ¹H NMR (400 MHz, chloroform-d) δ 7.28 (t, J = 7.5 Hz, 2H), 7.22 – 7.15 (m, 3H), 6.84 – 6.80 (m, 4H), 3.92 (t, J = 4.9 Hz, 2H), 3.76 (s, 3H), 2.68 (t, J = 6.5 Hz, 2H), 1.85 – 1.76 (m, 4H). ¹³C NMR (101 MHz, chloroform-d) δ 153.7, 153.2, 142.3, 128.4, 128.3, 125.8, 115.4, 114.6, 68.4, 55.8, 35.6, 29.0, 27.9. IR (neat) ν = 3025, 2999, 2939, 2861, 2832, 1508, 1465, 1389, 1288, 1232, 1180, 1107, 1040, 825, 745, 699, 524 cm⁻¹. HRMS (EI) Calcd. for C₁₇H₂₀O₂ [M⁺]: 256.1463; found 256.1470.
1-nitro-4-(4-phenylbutoxy)benzene (5j):⁴⁶% yellow liquid. 

\[ ^1\text{H NMR (400 MHz, chloroform-}d\text{)} \delta 8.17 (d, J = 9.3 Hz, 2H), 7.32 – 7.25 (m, 2H), 7.22 – 7.16 (m, 3H), 6.91 (d, J = 9.2 Hz, 2H), 4.04 (t, J = 5.8 Hz, 2H), 2.69 (t, J = 7.0 Hz, 2H), 1.92 – 1.74 (m, 4H). \]

(4-methoxybutyl)benzene (5k):⁴⁰% colorless liquid. 

\[ ^1\text{H NMR (400 MHz, chloroform-}d\text{)} \delta 7.30 – 7.23 (m, 2H), 7.19 – 7.13 (m, 3H), 3.37 (t, J = 6.3 Hz, 2H), 3.31 (s, 3H), 2.62 (t, J = 7.5 Hz, 2H), 1.73 – 1.57 (m, 4H). \]

4-phenylbutyl acetate (5l):⁶⁶ CH₃CO₂K⁺ was used as the nucleophile. quantitative; colorless liquid. 

\[ ^1\text{H NMR (400 MHz, chloroform-}d\text{)} \delta 7.30 – 7.25 (m, 2H), 7.21 – 7.14 (m, 3H), 4.07 (t, J = 6.2 Hz, 2H), 2.63 (t, J = 7.1 Hz, 2H), 2.03 (s, 3H), 1.73 – 1.62 (m, 4H). \]

4-phenylbutyl benzoate (5m):⁶⁰% colorless liquid. 

\[ ^1\text{H NMR (400 MHz, chloroform-}d\text{)} \delta 8.06 – 8.00 (m, 2H), 7.54 (t, J = 7.4 Hz, 1H), 7.43 (t, J = 7.8 Hz, 2H), 7.31 – 7.25 (m, 2H), 7.22 – 7.15 (m, 3H), 4.33 (t, J = 6.0 Hz, 2H), 2.68 (t, J = 7.0 Hz, 2H), 1.88 – 1.73 (m, 4H). \]

cinnamyl acetate (5n):⁹⁹ CH₃CO₂K⁺ was used as the nucleophile. 99% colorless liquid. 

\[ ^1\text{H NMR (400 MHz, chloroform-}d\text{)} \delta 7.42 – 7.35 (m, 2H), 7.35 – 7.28 (m, 2H), 7.28 – 7.20 (m, 1H), 6.64 (d, J = 15.9 Hz, 1H), 6.27 (dt, J = 15.9, 6.5 Hz, 1H), 4.71 (dd, J = 6.5, 1.1 Hz, 2H), 2.09 (s, 3H). \]

(3-phenoxybutyl)benzene (5o):³²% colorless liquid. 

\[ ^1\text{H NMR (400 MHz, chloroform-}d\text{)} \delta 7.30 – 7.25 (m, 4H), 7.21 – 7.14 (m, 3H), 6.92 (t, J = 7.3 Hz, 1H), 6.86 (d, J = 7.9 Hz, 2H), 4.36 (h, J = 6.0 Hz, 1H), 2.87 – 2.66 (m, 2H), 2.14 – 2.01 (m,
1H), 1.96 – 1.82 (m, 1H), 1.32 (d, J = 6.1 Hz, 3H).

4-phenylbutan-2-yl acetate (5p).\textsuperscript{16} CH$_3$CO$_2$K$^+$ was used as the nucleophile. 40%; colorless liquid. \textsuperscript{1}H NMR (400 MHz, chloroform-d) $\delta$ 7.30 – 7.23 (m, 2H), 7.20 – 7.13 (m, 3H), 4.92 (h, J = 6.3 Hz, 1H), 2.73 – 2.53 (m, 2H), 2.01 (s, 3H), 1.98 – 1.86 (m, 1H), 1.86 – 1.73 (m, 1H), 1.23 (d, J = 6.3 Hz, 3H).

2-methyl-4-phenylbutan-2-yl acetate (5q).\textsuperscript{16} CH$_3$CO$_2$K$^+$ was used as the nucleophile; 60 °C of reaction temperature and 5 h of reaction time were used. 44%; colorless liquid. \textsuperscript{1}H NMR (400 MHz, Chloroform-d) $\delta$ 7.30 – 7.23 (m, 2H), 7.21 – 7.13 (m, 3H), 2.67 – 2.59 (m, 2H), 2.10 – 2.01 (m, 2H), 1.96 (s, 3H), 1.49 (s, 6H).

tert-butyl 3-(phenoxy)methylpiperidine-1-carboxylate (5r): 55%; white solid. m.p. 57 – 58 °C. \textsuperscript{1}H NMR (400 MHz, DMSO-d$_6$) $\delta$ 7.29 (t, J = 7.8 Hz, 2H), 6.98 – 6.88 (m, 3H), 4.21 – 3.53 (m, 4H), 2.89 (br, 2H), 1.95 – 1.73 (m, 2H), 1.72 – 1.56 (m, 1H), 1.56 – 1.05 (m, 11H). \textsuperscript{13}C NMR (101 MHz, chloroform-d) $\delta$ 158.9, 155.0, 129.4, 120.7, 114.5, 79.4, 69.9, 47.2, 44.5, 35.9, 28.4, 27.4, 24.5. IR (neat) $\nu$ = 3447, 2975, 2930, 2857, 1692, 1598, 1497, 1469, 1421, 1365, 1266, 1242, 1175, 1148, 1041, 753, 691, 668 cm$^{-1}$. HRMS (EI) Calcd. for C$_{17}$H$_{25}$NO$_3$ [M]$^+$: 291.1834; found 291.1842.

N-(4-(4-fluorophenyl)-6-isopropyl-5-(phenoxy)methyl)pyrimidin-2-yl)-N methylmethanesulfonamide (5s): 77%; white solid. m.p. 129 – 130 °C. \textsuperscript{1}H NMR (400 MHz, chloroform-d) $\delta$ 7.77 – 7.69 (m, 2H), 7.37 – 7.29 (m, 2H), 7.08 (t, J = 8.7 Hz, 2H), 7.03 (t, J = 7.4 Hz, 1H), 6.94 (d, J = 8.6 Hz, 2H), 4.89 (s, 2H), 3.59 (s, 3H), 3.52 (s, 3H), 3.40 – 3.26 (m, 1H), 1.32 (d, J = 6.7 Hz, 6H). \textsuperscript{19}F NMR (376 MHz, chloroform-d) $\delta$ -110.85 – -110.98 (m, 1F). \textsuperscript{13}C NMR (101 MHz, chloroform-d) $\delta$
178.4, 166.6, 163.8 (d, $J = 250.3$ Hz), 158.4, 158.0, 133.8 (d, $J = 3.1$ Hz), 131.4 (d, $J = 21.7$ Hz), 114.7, 63.3, 42.5, 33.1, 31.8, 22.1. IR (neat) $\nu = 2974, 2931, 1872, 1605, 1594, 1553, 1480, 1339, 1231, 1155, 1069, 1029, 957, 896, 848, 820, 772, 756, 691, 600, 575, 521, 495$ cm$^{-1}$. HRMS (EI) Calcd. for C$_{22}$H$_{24}$N$_3$O$_3$FS [M$^+$]: 429.1522; found 429.1528.

1-(chloromethyl)-4-phenoxybenzene (5t). $^{29}$ $^{\text{Bu}_4\text{N}}^+$Cl$^-$ was used as the nucleophile. 55%; colorless liquid. $^1$H NMR (400 MHz, chloroform-d) $\delta$ 7.40 – 7.30 (m, 4H), 7.12 (t, $J = 7.4$ Hz, 1H), 7.02 (d, $J = 7.7$ Hz, 2H), 6.97 (d, $J = 8.6$ Hz, 2H), 4.58 (s, 2H).

1-(bromomethyl)-4-nitrobenzene (5u). $^{30}$ $^{\text{Bu}_4\text{N}}^+$Br$^-$ was used as the nucleophile. quantitative; white solid. $^1$H NMR (400 MHz, chloroform-d) $\delta$ 8.14 (d, $J = 8.7$ Hz, 2H), 7.51 (d, $J = 8.7$ Hz, 2H), 4.46 (s, 2H).

1-bromo-4-(iodomethyl)benzene (5v). $^{31}$ NaI was not required as the iodide anion was generated from ICH$_2$CH$_2$I; 60 °C of reaction temperature and 2 h of reaction time in CH$_3$CN were used. 92%; white solid. $^1$H NMR (500 MHz, chloroform-d) $\delta$ 7.44 – 7.40 (m, 2H), 7.26 – 7.15 (m, 3H), 4.40 (s, 2H).

(4-iodobutyl)benzene (5w). $^{12}$ NaI was not required as the iodide anion was generated from ICH$_2$CH$_2$I; 60 °C of reaction temperature and 2 h of reaction time in CH$_3$CN were used. 85%; yellow liquid. $^1$H NMR (400 MHz, chloroform-d) $\delta$ 7.28 (t, $J = 7.4$ Hz, 2H), 7.23 – 7.15 (m, 3H), 3.20 (t, $J = 6.9$ Hz, 2H), 2.63 (t, $J = 7.5$ Hz, 2H), 1.92 – 1.80 (m, 2H), 1.80 – 1.68 (m, 2H).

(3-chlorobutyl)benzene (5x). $^{32}$ $^{\text{Bu}_4\text{N}}^+$Cl$^-$ was used as the nucleophile. 79%; colorless liquid. $^1$H NMR (400 MHz, chloroform-d) $\delta$ 7.33 – 7.25 (m, 2H), 7.23 – 7.16 (m, 3H), 3.99 (h, $J = 6.5$ Hz, 1H), 2.90 – 2.80 (m, 1H), 2.79 – 2.69 (m, 1H), 2.05 – 1.97 (m,
(3-bromobutyl)benzene (5y).\textsuperscript{33} \textsuperscript{a}Bu4NB\textsuperscript{-}Br was used as the nucleophile. 62%; colorless liquid. \textsuperscript{1}H NMR (400 MHz, chloroform-\textit{d}) \(\delta\) 7.32 – 7.26 (m, 2H), 7.23 – 7.16 (m, 3H), 4.13 – 4.02 (m, 1H), 2.91 – 2.81 (m, 1H), 2.79 – 2.69 (m, 1H), 2.19 – 1.98 (m, 2H), 1.72 (d, \(J = 6.7\) Hz, 3H).

(3-iodobutyl)benzene (5z).\textsuperscript{34} NaI was not required as the iodide anion was generated from ICH\textsubscript{2}CH\textsubscript{2}I; 60 °C of reaction temperature and 2 h of reaction time in CH\textsubscript{3}CN were used. 35%; colorless liquid. \textsuperscript{1}H NMR (400 MHz, chloroform-\textit{d}) \(\delta\) 7.32 – 7.26 (m, 2H), 7.24 – 7.16 (m, 3H), 4.19 – 4.05 (m, 1H), 2.90 – 2.80 (m, 1H), 2.75 – 2.64 (m, 1H), 2.22 – 2.10 (m, 1H), 1.95 (d, \(J = 6.8\) Hz, 3H), 1.93 – 1.82 (m, 1H).

5 The synthesis of \textsuperscript{18}O-1a

NaH (0.6 mmol, 14.4 mg) was added into a solution of H\textsubscript{2}\textsuperscript{18}O (\textsuperscript{18}O content: 95%, 0.6 mmol, 12.0 mg) in anhydrous DMF (1.0 mL) under N\textsubscript{2} atmosphere. The mixture was stirred for around 3 min to afford the suspension of Na\textsuperscript{18}OH in anhydrous DMF.

Alcohol 1a (1.0 equiv, 0.2 mmol, 36.8 mg), triphenylphosphine (1.2 equiv, 0.24 mmol, 62.9 mg) and anhydrous DMF (1.0 mL) were added into a 10 mL sealed tube under a N\textsubscript{2} atmosphere. 1,2-Diiodoethane (1.2 equiv, 0.24 mmol, 67.6 mg) was then added and the resulting mixture was stirred for around 1 min until the 1,2-diiodoethane was completely dissolved. The above Na\textsuperscript{18}OH suspension was added subsequently and the mixture was stirred at 50 °C for 5 h. Dichloromethane (20 mL) was added and the resulting solution was washed with water (20 mL x 3). The organic layer was dried over Na\textsubscript{2}SO\textsubscript{4}. After filtration, the solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography to afford product \textsuperscript{18}O-1a (25.0 mg, 67%). MS-EI analysis of the product showed that \(^{18}\text{O}:^{16}\text{O} = 50:50\).
MS-EI spectrum of the $^{18}$O-product:

![MS-EI spectrum of the $^{18}$O-product](image)

MS-EI spectrum of the substrate $1a$:

![MS-EI spectrum of the substrate $1a$](image)
6. Isolation of Ph$_3$P=O

\[
\begin{align*}
\text{Ph} & \quad \text{OH} & \quad \text{Ph}_3\text{P} (0.24 \text{ mmol}) \\
& \quad \text{+ KOAc} & \quad \text{ICH}_2\text{CH}_2\text{I} (0.24 \text{ mmol}) \\
\text{Ph}_3\text{P=O} & \quad \text{Ph} & \quad \text{OAc} \quad \text{+ Ph}_3\text{P=O} \\
\text{1a (0.2 mmol)} & \quad \text{4 (0.6 mmol)} & \quad \text{5a} & \quad (79\%) \\
\end{align*}
\]

Esterification of alcohol 1a (0.2 mmol) with KOAc occurred well under the optimal conditions. After the reaction was completed, 53.0 mg of Ph$_3$P=O was isolated in 79% yield (based on Ph$_3$P). $^1$H NMR (400 MHz, chloroform-$d$) $\delta$ 7.72 – 7.60 (m, 6H), 7.58 – 7.48 (m, 3H), 7.49 – 7.37 (m, 6H). $^{31}$P NMR (162 MHz, chloroform-$d$) $\delta$ 29.86 (s, 1P).

7. Inversion of configuration

\[
\begin{align*}
\text{Ph} & \quad \text{OH} & \quad \text{optimal conditions} & \quad \text{Cl} & \quad \text{Me} \\
\text{S-1w} & \quad \text{Me} & \quad 5w (75\%, \text{R/S} = 81/19) \\
\end{align*}
\]

$S$-$1w$ (0.5 mmol) was converted into $R$-$5w$ under the optimized conditions. Enantiomeric excess was determined by chiral GC with a CP-chirasil-DEX CB column (length 25 m, diameter 0.25 mm, 0.25 µm layer thickness) and nitrogen as carrier gas. Injector temperature was 100 °C. Major enantiomer rt = 36.417 min, minor enantiomeric rt = 37.465 min. GC analysis from the isolated product showed an er of 81:19.

$[\alpha]_{D}^{25} = -37.4$ (c = 1.15 g/100 mL, CHCl$_3$, er 81:19).

Lit. $[\alpha]_{D}^{25} = -36.4$ (c = 1.00 g/100 mL, CHCl$_3$, $R$-ent, no enantiopurity given).
Gas chromatogram of a racemic sample:

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Inj Volume : Manually

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(modified after loading)

Analysis Method : C:\CHEM32\METHODS\TEST2-1.M
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(modified after loading)

Additional Info : Peak(s) manually integrated

FID1 A, 脉冲信号 (2017SIG1000303.D)

Area Percent Report

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

Signal 1: FID1 A, 脉冲信号

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Totals : 89.75812 4.31946

*** End of Report ***

S18
Gas chromatogram of the enantioenriched sample:

Area Percent Report

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Totals: 36.79947 1.83555

*** End of Report ***
8. References and notes


9. Copies of $^1$H NMR, $^{19}$F NMR, $^{13}$C NMR and $^{31}$P NMR spectra

$^1$H NMR

$^1$H NMR
$^1$H NMR

$^1$H NMR
$^1$H NMR

$^{19}$F NMR
$^{13}\text{C NMR}$

$^1\text{H NMR}$
$^1$H NMR

$^{13}$C NMR
$^{13}$C NMR

$^1$H NMR

5c
1H NMR

$5f$

1H NMR

$5g$
$^1$H NMR

$^1$H NMR
13C NMR

5i

1H NMR

5j
$^1$H NMR

5m

$^1$H NMR

5n
$^1$H NMR

$^1$H NMR
$^{13}$C NMR

$^1$H NMR
$^1$H NMR

$^1$H NMR