Selenocyanobenziodoxolone: A Practical Electrophilic Selenocyanation Reagent and Its Application for Solid-state Synthesis of α-Carbonyl Selenocyanates

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Supporting Information

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1. General Information

Unless otherwise noted, all the reagents were purchased from commercial suppliers and used without further purification. $^1$H NMR spectra were recorded at 300 MHz. The chemical shifts were recorded in ppm relative to tetramethylsilane and with the solvent resonance as the internal standard. Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration. $^{13}$C NMR data were collected at 75 MHz with complete proton decoupling. Chemical shifts were reported in ppm from the tetramethylsilane with the solvent resonance as internal standard. Infrared spectra (IR) were measured by FT-IR apparatus. $^{19}$F NMR data were collected at 282 MHz with complete proton decoupling. High resolution mass spectroscopy (HRMS) was recorded on TOF MS ESI$^+$ mass spectrometer and acetonitrile was used to dissolve the sample. Column chromatography was carried out on silica gel (200-300 mesh). All solvents and commercially available reagents were either purified via literature procedures or used without further purification.

The $N$-acetyl-$\alpha$-arylenamines derivatives and silyl enol ethers derivatives were prepared according to the reported procedure.[1], [2]

Caution: the hypervalent iodine compounds have a hazardous of explosion after grinding. Thus, a safety shield is necessary before carrying out the reaction.

2. Experimental Procedures and Characterization Data

2.1 Synthetic procedure for the synthesis of selenocyanobenziodoxolone 3 (BI-SeCN) and thiocyanobenziodoxolone 6 (BI-SCN)

\[
\begin{align*}
\text{COOH} & \quad \overset{\text{BuOCl, MeOH, 10 min}}{\longrightarrow} \quad \overset{\text{AgXCN, CHCl}_3, \text{Ar}}{\longrightarrow} \\
1 & \quad 2 \ (82\%) & \quad 3, X = \text{Se}, 86\% & \quad 6, X = \text{S}, 73\%
\end{align*}
\]

To a solution of 2-iodobenzoic acid 1 (3 g, 12.1 mmol) in anhydrous methanol (50 mL) was added $\text{tert}$-butyl hypochlorite. The mixture was stirred vigorously at room temperature for 10 min. Then, the white precipitate was filtered and dried under vacuum to yield 1-chlorobenziodoxolone 2 as white solid (2.8 g, 9.9 mmol, 82% yield).

To a stir solution of 1-chlorobenziodoxolone 2 (2 g, 7.1 mmol) in dichloromethane (35 mL), silver selenocyanate (1.8 g, 8.5 mmol, 1.2 equiv.) was added under Ar atmosphere. The flask was covered with thin-foil and stirred at room temperature for 48 h. After the completion of the reaction, the undissolved solid was filtered off and the residue was concentrated under high vacuum to give the BI-SeCN as a yellow solid (2.2 g, 6.1 mmol). Synthesized using the same
procedure with silver thiocyanate in 7.1 mmol scale to afford the BI-SCN as a white solid (1.6 g, 5.2 mmol). Those reagents were used in the next step without further purification and preserved at 0 °C.

**Selenocyanobenziodoxolone (3):** yellow solid (86% yield, 2.2 g, 6.1 mmol); m.p. 97-99 °C; IR (KBr) ν 2517, 1799, 1654, 1467, 1270, 1018, 879, 732 cm⁻¹; 

\[ ^1\text{H} \text{NMR} \ (300 \text{ MHz, DMSO-d}_6) \ \delta \ 7.99 \ (d, J = 7.8 \text{ Hz, } 1\text{H}), \ 7.71 \ (d, J = 7.5 \text{ Hz, } 1\text{H}), \ 7.48 \ (t, J = 7.2 \text{ Hz, } 1\text{H}), \ 7.24 \ (t, J = 7.8 \text{ Hz, } 1\text{H}); \ ]

\[ ^{13}\text{C} \text{NMR} \ (75 \text{ MHz, DMSO-d}_6) \ \delta \ 168.6, \ 141.0, \ 137.4, \ 132.9, \ 130.5, \ 128.6, \ 102.4, \ 94.5; \ ]

HRMS (TOF-ESI⁺) m/z: calcd for C₈H₄NO₂SeINa [M+Na]⁺ 375.8344, found 375.8340.

**Thiocyanobenziodoxolone (6):** white solid (73% yield, 1.6 g, 5.2 mmol); m.p. 74-75 °C; IR (KBr) ν 2053, 1794, 1679, 1465, 1270, 1008, 894, 732 cm⁻¹; 

\[ ^1\text{H} \text{NMR} \ (300 \text{ MHz, CDCl}_3) \ \delta \ 8.03-8.10 \ (m, 2\text{H}), \ 7.48 \ (td, J = 7.5, \ 1.2 \text{ Hz, } 1\text{H}), \ 7.23 \ (td, J = 7.8, \ 1.7 \text{ Hz, } 1\text{H}); \ ]

\[ ^{13}\text{C} \text{NMR} \ (75 \text{ MHz, CDCl}_3) \ \delta \ 171.2, \ 142.0, \ 133.6, \ 133.1, \ 132.1, \ 128.1, \ 107.0, \ 94.7; \ ]

HRMS (TOF-ESI⁺) m/z: calcd for C₈H₄NO₂SINa [M+Na]⁺ 327.8907, found 327.8907.

**2.2 General procedure for the synthesis of α-carbonyl selenocyanate compounds.**

*General Procedure 1*

![NHAc](NHAc.png) → BI-SeCN → ![SeCN](SeCN.png)

The N-acetyl-α-arylenamine (0.1 mmol) and BI-SeCN (52.9 mg, 0.15 mmol, 1.5 equiv.) was ground together with agate mortar at room temperature for the given time. After the completion of the reaction, the mixture was dissolved in EtOAc (2 mL). Then the mixture was extracted with EtOAc (2×2 mL) and washed with saturated aqueous NaHCO₃. The combined organic phase were dried (Na₂SO₄) and concentrated in vacuo. The resulting residue was purified by flash column chromatography (PE:EtOAc = 9:1-5:1) to afford the desired selenocyanate compound as a white solid.

*Caution: This reaction should be carried out behind a safety shield!*

*General Procedure 2*

![OTBS](OTBS.png) → BI-SeCN → ![SeCN](SeCN.png)
The silyl enol ether (0.1 mmol) and BI-SeCN (52.9 mg, 0.15 mmol, 1.5 equiv.) was ground together with agate mortar at room temperature for the given time. After the completion of the reaction, the mixture was dissolved in EtOAc (2 mL). Then the mixture was extracted with EtOAc (2 × 2 mL) and washed with saturated aqueous NaHCO₃. The combined organic phase were dried (Na₂SO₄) and concentrated in vacuo. The resulting residue was purified by flash column chromatography (PE:EtOAc = 9:1-5:1) to afford the desired selenocyanate compound as a white solid.

**Caution: This reaction should be carried out behind a safety shield!**

**General Procedure 3**

\[
\begin{array}{c}
\text{COOR} \\
\underline{\text{4}} \\
\text{BI-SeCN} \\
\underline{\text{5}} \\
\text{COOR}
\end{array}
\]

The β-keto ester (0.1 mmol) and BI-SeCN (52.9 mg, 0.15 mmol, 1.5 equiv.) was ground together with agate mortar at room temperature for the given time. After the completion of the reaction, the mixture was dissolved in EtOAc (2 mL). Then the mixture was extracted with EtOAc (2 × 2 mL) and washed with saturated aqueous NaHCO₃. The combined organic phase were dried (Na₂SO₄) and concentrated in vacuo. The resulting residue was purified by flash column chromatography with PE:EtOAc = 9:1-5:1 to afford the desired selenocyanate compound as a white solid.

**Caution: This reaction should be carried out behind a safety shield!**

**General Procedure 4**

\[
\begin{array}{c}
\text{CO} \\
\underline{\text{4}} \\
\text{BI-SeCN} \\
\underline{\text{5}} \\
\text{LA} \\
\text{SeCN}
\end{array}
\]

The ketone (0.1 mmol), BI-SeCN (52.9 mg, 0.15 mmol, 1.5 equiv.) and Lewis acid (0.01 mmol) was ground together with agate mortar at room temperature for the given time. After the completion of the reaction, the mixture was dissolved in EtOAc (2 mL). Then the mixture was extracted with EtOAc (2 × 2 mL) and washed with saturated aqueous NaHCO₃. The combined organic phase were dried (Na₂SO₄) and concentrated in vacuo. The resulting residue was purified by flash column chromatography with (PE:EtOAc = 9:1-5:1) to afford the desired selenocyanate compound as a white solid.
Various lewis acids were screened under grinding conditions. Pleasingly, scandium(III) triflate performed the best and afforded the desired product 5aa in 92% yield (Table S-1, entry 5).

Table S-1 Screening of the lewis acids

<table>
<thead>
<tr>
<th>entry</th>
<th>LA</th>
<th>t (h)</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Mg(OTf)₂</td>
<td>0.5</td>
<td>nr</td>
</tr>
<tr>
<td>2</td>
<td>Zn(OTf)₂</td>
<td>0.5</td>
<td>64</td>
</tr>
<tr>
<td>3</td>
<td>Cu(OTf)₂</td>
<td>0.5</td>
<td>71</td>
</tr>
<tr>
<td>4</td>
<td>Yb(OTf)₂</td>
<td>0.5</td>
<td>80</td>
</tr>
<tr>
<td>5</td>
<td>Sc(OTf)₂</td>
<td>0.5</td>
<td>92</td>
</tr>
</tbody>
</table>

Caution: This reaction should be carried out behind a safety shield!

General Procedure 5

To a stir solution of N-acetyl-α-arylenamines/silyl enol ethers/β-keto esters (0.1 mmol) in DCE (1 mL), BI-SeCN (52.9 mg, 0.15 mmol, 1.5 equiv.) was added. After the completion of the reaction, the mixture was extracted with EtOAc (2×2 mL) and washed with saturated aqueous NaHCO₃. The combined organic phase were dried (Na₂SO₄) and concentrated in vacuo. The resulting residue was purified by flash column chromatography (PE:EtOAc = 9:1-5:1) to afford the desired selenocyanate compound as a white solid.

Caution: This reaction should be carried out behind a safety shield!

2-Selenocyanato-3,4-dihydronaphthalen-1(2H)-one (5aa): Obtained in 99% yield (24.8 mg, 0.099 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 79-80 °C (lit. 82-83 °C); IR (KBr) ν 2906, 2512, 2148, 1797, 1594, 1457, 896, 739 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 7.90 (d, J = 8.1 Hz, 1H), 7.65 (t, J = 7.2 Hz, 1H), 7.41 (t, J = 6.0 Hz, 2H), 5.31 (dd, J = 12.6, 5.1 Hz, 1H), 3.16-3.27 (m, 1H), 3.07-3.12 (m, 1H), 2.64-2.70 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 194.2, 144.1, 135.0, 129.0, 127.8, 127.4, 102.5, 53.2, 32.6, 30.3; HRMS (TOF-ESI⁺) m/z: calcd for C₁₁H₉NOSeNa [M+Na⁺] 273.9742, found 273.9753.
Ethyl 1-Oxo-2-selenocyanato-2,3-dihydro-1H-indene-2-carboxylate (5ab): Obtained in 83% yield (25.6 mg, 0.083 mmol) according to procedure 3 after column chromatography as a white solid by using β-keto ester; m.p. 114-115 °C; IR (KBr) ν 2938, 2509, 2148, 1791, 1599, 1472, 1180, 751, 694 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.91 (d, J = 7.8 Hz, 1H), 7.76 (t, J = 7.5 Hz, 1H), 7.49-7.56 (m, 2H), 4.19-4.34 (m, 3H), 3.84 (d, J = 18.6 Hz, 1H), 1.29 (t, J = 7.2 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 196.0, 167.4, 151.1, 136.9, 132.9, 128.9, 126.2, 125.8, 100.7, 64.3, 61.6, 41.0, 13.9; HRMS (TOF-ESI⁺) m/z: calcd for C₁₃H₁₁NO₃SeNa [M+Na]⁺ 331.9796, found 331.9813.

Ethyl 1-Oxo-2-selenocyanato-5-bromo-2,3-dihydro-1H-indene-2-carboxylate (5ac): Obtained in 65% yield (25.1 mg, 0.065 mmol) according to procedure 3 after column chromatography as a white solid by using β-keto ester; m.p. 117-118 °C; IR (KBr) ν 2983, 2148, 1716, 1594, 1427, 1183, 829, 575 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.02 (s, 1H), 7.84 (d, J = 8.4 Hz, 1H), 7.44 (d, J = 8.1 Hz, 1H), 4.30 (q, J = 7.2 Hz, 2H), 4.15 (d, J = 18.6 Hz, 1H), 3.77 (d, J = 18.6 Hz, 1H), 1.28 (t, J = 6.9 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 194.8, 167.0, 149.7, 139.7, 134.7, 128.5, 127.7, 123.1, 100.6, 64.5, 61.6, 40.6, 13.9; HRMS (TOF-ESI⁺) m/z: calcd for C₁₃H₁₀NO₃SeBrNa [M+Na]⁺ 409.8901, found 409.8900.

Ethyl 1-Oxo-2-selenocyanato-5-methyl-2,3-dihydro-1H-indene-2-carboxylate (5ad): Obtained in 93% yield (30.0 mg, 0.093 mmol) according to procedure 3 after column chromatography as a white solid by using β-keto ester; m.p. 147-148 °C; IR (KBr) ν 2990, 2519, 1764, 1577,1465, 1013, 874, 732 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.70 (s, 1H), 7.57 (d, J = 7.8 Hz, 1H), 7.43 (d, J = 7.8 Hz, 1H), 4.29 (q, J = 6.9 Hz, 2H), 4.16 (d, J = 18.3 Hz, 1H), 3.78 (d, J = 18.3 Hz, 1H), 2.46 (s, 3H), 1.28 (t, J = 6.9 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 196.0, 167.6, 148.6, 133.1, 125.8, 125.6, 100.8, 64.2, 62.1, 40.7, 21.1, 13.9; HRMS (TOF-ESI⁺) m/z: calcd for C₁₄H₁₃NO₃SeNa [M+Na]⁺ 345.9953, found 345.9944.

Methyl 1-Oxo-2-selenocyanato-1,2,3,4-tetrahydronaphthalene-2-carboxylate (5ae): Obtained in 88% yield (28.4 mg, 0.088 mmol) according to procedure 3 after column chromatography as a white solid by using β-keto ester; m.p. 141-143 °C; IR (KBr) ν 2958, 2153, 1767, 1477, 1308, 961, 879 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.04 (d, J = 7.8 Hz, 1H), 7.60 (t, J = 7.5 Hz, 1H), 7.39 (t, J = 7.5 Hz, 1H), 7.30 (d, J = 7.8 Hz, 1H), 3.82 (s, 3H), 3.25-3.33 (m, 1H), 3.06-3.19 (m, 2H), 2.72-2.82 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 191.5, 167.8, 143.5, 135.3, 130.0, 129.1, 128.4, 127.5, 101.8, 64.2, 54.0, 35.0, 27.9; HRMS (TOF-ESI⁺) m/z: C₁₃H₁₃NO₃SeNa [M+Na]⁺ 331.9796, found 331.9803.

Ethyl 1-Oxo-2-selenocyanato-6-methoxy-1,2,3,4-tetrahydronaphthalene-2-carboxylate (5af): Obtained in 79% yield (27.9 mg, 0.079 mmol) according to procedure 3 after column chromatography as a white solid by...
using β-keto ester; m.p. 132-133 °C; IR (KBr) v 2947, 2516, 2153, 1657, 1342, 1067, 1013, 883 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.01 (d, J = 9.0 Hz, 1H), 6.89 (dd, J = 2.1, 8.7 Hz, 1H), 6.72 (s, 1H), 4.25-4.34 (m, 2H), 3.89 (s, 3H), 3.21-3.29 (m, 1H), 2.99-3.15 (m, 2H), 2.69-2.79 (m, 1H), 1.27 (t, J = 6.9 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 190.0, 167.6, 165.1, 146.2, 130.9, 123.5, 114.4, 112.7, 102.3, 64.5, 63.2, 35.0, 28.4, 13.8; HRMS (TOF-ESI⁺) m/z: calcd for C₁₅H₁₅NO₄SeNa [M+Na]+ 376.0039, found 376.0039.

Ethyl 1-selenocyanato-2-oxo-cyclohexanecarboxylate (5ag): Obtained in 81% yield (22.3 mg, 0.081 mmol) according to procedure 3 after column chromatography as a colorless oil by using β-keto ester; IR (KBr) ν 2923, 2856, 2155, 1796, 1454, 1203, 869, 547 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 4.25-4.31 (m, 2H), 2.90 (dd, J = 12.9, 2.4 Hz, 1H), 2.50-2.69 (m, 2H), 2.25 (td, J = 13.2, 3.6 Hz, 1H), 2.03-2.07 (m, 1H), 1.85 (d, J = 13.2 Hz, 1H), 1.66-1.75 (m, 1H), 1.45-1.59 (m, 1H), 1.22 (t, J = 6.9 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 205.5, 167.6, 102.7, 66.8, 63.3, 38.7, 26.7, 23.9, 14.1; HRMS (TOF-ESI⁺) m/z: calcd for C₁₀H₁₃NO₃SeNa [M+Na]+ 297.9953, found 297.9953.

Ethyl 3-oxo-3-phenyl-2-selenocyanatopropanoate (5ah): Obtained in 84% yield (25.0 mg, 0.084 mmol) according to procedure 3 after column chromatography as a colorless oil by using β-keto ester; IR (KBr) ν 2978, 2155, 1796, 1734, 1450, 1235, 876, 684 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 8.01 (d, J = 7.5 Hz, 2H), 7.75 (t, J = 7.2 Hz, 1H), 7.60 (t, J = 7.5 Hz, 2H), 6.37 (s, 1H), 4.17-4.23 (m, 2H), 1.15 (t, J = 7.2 Hz, 3H); ¹³C NMR (75 MHz, DMSO-d₆) δ 191.5, 167.2, 135.3, 133.9, 129.6, 129.4, 103.3, 63.0, 51.8, 14.2; HRMS (TOF-ESI⁺) m/z: calcd for C₁₂H₁₁NO₃SeNa [M+Na]+ 319.9800.

1-Phenyl-2-selenocyanatoethanone (5ai): Obtained in 89% yield (20.0 mg, 0.089 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 81-82 °C (lit. 48-49 °C); IR (KBr) ν 2955, 2514, 2155, 1796, 1462, 1268, 884, 734 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.97 (d, J = 7.5 Hz, 2H), 7.70 (t, J = 7.2 Hz, 1H), 7.60 (t, J = 7.5 Hz, 2H), 7.55 (t, J = 7.5 Hz, 2H), 4.96 (s, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 193.2, 134.9, 133.8, 129.2, 128.8, 101.9, 38.5; HRMS (TOF-ESI⁺) m/z: calcd for C₉H₇NOSeNa [M+Na]+ 247.9585, found 247.9571.

1-(4-Fluorophenyl)-2-selenocyanatoethanone (5aj): Obtained in 83% yield (20.2 mg, 0.083 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 118-119 °C (lit. 112-113 °C); IR (KBr) ν 2990, 2158, 1657, 1412, 1290, 1000, 824, 581 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.99-8.04 (m, 2H), 7.22 (t, J = 8.4 Hz, 2H), 4.92 (s, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 191.6, 166.7 (d, Jₑ,F = 257.3 Hz), 131.6 (d, ²Jₑ,F = 9.8 Hz), 130.3 (d, ³Jₑ,F = 3.0 Hz), 116.5 (d, ²Jₑ,F = 22.5 Hz),...
1-(4-Chlorophenyl)-2-selenocyanatoethanone (5ak): Obtained in 71% yield (18.4 mg, 0.071 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 162-164 °C; IR (KBr) ν 2985, 2155, 1796, 1657, 1399, 1178, 816, 579 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 8.04-8.15 (m, 2H), 7.65 (d, J = 8.7 Hz, 1H), 7.41 (t, J = 8.7 Hz, 1H), 4.79 (d, J = 7.2 Hz, 2H); ¹³C NMR (75 MHz, DMSO-d₆) δ 193.4 (d, J = 29.3 Hz), 139.5, 133.4, 132.4 (d, J = 9.8 Hz), 131.2, 129.5, 116.4 (d, J = 21.8 Hz), 104.3, 35.6 (d, J = 12.0 Hz); HRMS (TOF-ESI⁺) m/z: calcd for C₉H₆NOSeFNa [M+Na]⁺ 265.9491, found 265.9499.

1-(4-Bromophenyl)-2-selenocyanatoethanone (5al): Obtained in 66% yield (20.0 mg, 0.066 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 184-186 °C (lit. 144-145 °C); IR (KBr) ν 2509, 1794, 1652, 1465, 1397, 1008, 871, 739 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 7.97 (d, J = 8.1 Hz, 2H), 7.79 (d, J = 8.1 Hz, 2H), 4.77 (s, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 192.2, 132.6, 132.5, 130.5, 130.1, 101.5, 37.9; HRMS (TOF-ESI⁺) m/z: calcd for C₉H₆NOSeBrNa [M+Na]⁺ 325.8690, found 325.8679.

1-(3-Methylphenyl)-2-selenocyanatoethanone (5am): Obtained in 84% yield (20.1 mg, 0.084 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 84-85 °C (lit. 43-44 °C); IR (KBr) ν 2983, 2153, 1799, 1380, 1290, 1043, 786 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.76-7.78 (m, 2H), 7.51 (d, J = 7.2 Hz, 1H), 7.43 (t, J = 7.5 Hz, 1H), 4.95 (s, 2H), 2.46 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 193.4, 139.2, 135.7, 133.8, 129.2, 129.0, 126.0, 101.9, 38.6, 21.3; HRMS (TOF-ESI⁺) m/z: calcd for C₁₀H₉NOSeNa [M+Na]⁺ 261.9742, found 261.9742.

2-Selenocyanato-1-(thiophen-2-yl)ethanone (5an): Obtained in 99% yield (22.9 mg, 0.099 mmol) according to procedure 2 after column chromatography as a white solid by using silyl enol ethers; m.p. 101-102 °C (lit. 100-101 °C); IR (KBr) ν 2936, 2158, 1794, 1429, 941, 836 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.82 (d, J = 3.9 Hz, 2H), 7.23 (t, J = 3.9 Hz, 1H), 4.77 (s, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 185.4, 140.2, 136.3, 134.3, 128.8, 101.4, 36.7; HRMS (TOF-ESI⁺) m/z: calcd for C₇H₅NOSSeNa [M+Na]⁺ 253.9143, found 253.9143.

1-(4-Chlorophenyl)-2-selenocyanatopropan-1-one (5ao): Obtained in 95% yield (25.9 mg, 0.095 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 72-73 °C (lit. 70-71 °C); IR (KBr) ν 2921, 2514, 2160, 1791, 1440, 1230, 844, 752 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 8.04-8.15 (m, 2H), 7.65 (d, J = 8.7 Hz, 1H), 7.41 (t, J = 8.7 Hz, 1H), 4.79 (d, J = 7.2 Hz, 2H); ¹³C NMR (75 MHz, DMSO-d₆) δ 193.4 (d, J = 29.3 Hz), 139.5, 133.4, 132.4 (d, J = 9.8 Hz), 131.2, 129.5, 116.4 (d, J = 21.8 Hz), 104.3, 35.6 (d, J = 12.0 Hz); HRMS (TOF-ESI⁺) m/z: calcd for C₉H₆NOSeFNa [M+Na]⁺ 265.9491, found 265.9499.
**DMSO-\textit{d}_6** \(\delta\) 8.08 (d, \(J = 8.4\) Hz, 2H), 7.64 (d, \(J = 8.4\) Hz, 2H), 5.18 (dd, \(J = 13.2, 6.6\) Hz, 1H), 1.77 (dd, \(J = 6.6\) Hz, 3H); \(^{13}\text{C NMR (75 MHz, CDCl}_3\) \(\delta\) 195.8, 141.4, 131.1, 130.3, 129.6, 102.3, 48.3, 21.2; \text{HRMS (TOF-ESI\textsuperscript{+}) \(m/z\)}: calcd for \(\text{C}_{10}\text{H}_8\text{NOSeClNa [M+Na]}^+\) \(295.9352\), found 295.9332.

2-Selenocyanato-2,3-dihydro-1H-inden-1-one (\textit{5ap}): Obtained in 93\% yield (22.0 mg, 0.093 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 131-133 °C; \text{IR (KBr)} \(\nu\) 2965, 2517, 2153, 1794, 1604, 1460, 1325, 754 cm\(^{-1}\); \(\text{\textit{1H NMR (300 MHz, DMSO-\textit{d}_6)}}\) \(\delta\) 7.75 (d, \(J = 7.5\) Hz, 2H), 7.61 (d, \(J = 7.5\) Hz, 1H), 7.49 (t, \(J = 7.2\) Hz, 1H), 4.45 (dd, \(J = 8.1, 3.6\) Hz, 1H), 3.88 (dd, \(J = 18.0, 8.1\) Hz, 1H), 3.29-3.30 (m, 1H); \(\text{\textit{13C NMR (75 MHz, CDCl}_3\) \(\delta\) 199.8, 151.7, 136.4, 134.0, 128.6, 126.5, 125.0, 99.8, 44.2, 35.6; \text{HRMS (TOF-ESI\textsuperscript{+}) \(m/z\)}: calcd for \(\text{C}_{10}\text{H}_7\text{NOSeNa [M+Na]}^+\) \(259.9578\), found 259.9578.

5-Bromo-2-selenocyanato-2,3-dihydro-1H-inden-1-one (\textit{5aq}): Obtained in 86\% yield (27.1 mg, 0.086 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 123-125 °C; \text{IR (KBr)} \(\nu\) 2923, 2856, 2155, 1796, 1445, 1245, 869, 547 cm\(^{-1}\); \(\text{\textit{1H NMR (300 MHz, DMSO-\textit{d}_6)}}\) \(\delta\) 7.93 (dd, \(J = 8.1, 1.5\) Hz,, 1H), 7.88 (s, 1H), 7.59 (d, \(J = 8.1\) Hz,1H), 4.47 (dd, \(J = 8.1, 3.6\) Hz, 1H), 3.84 (dd, \(J = 18.3, 8.1\) Hz, 1H), 3.25 (d, \(J = 3.0\) Hz, 1H); \(\text{\textit{13C NMR (75 MHz, CDCl}_3\) \(\delta\) 198.5, 150.2, 139.1, 135.8, 128.0, 127.8, 122.8, 99.5, 43.9, 35.3; \text{HRMS (TOF-ESI\textsuperscript{+}) \(m/z\)}: calcd for \(\text{C}_{10}\text{H}_6\text{NOSeBrNa [M+Na]}^+\) \(337.8690\), found 337.8683.

5-Methyl-2-selenocyanato-2,3-dihydro-1H-inden-1-one (\textit{5ar}): Obtained in 82\% yield (20.6 mg, 0.082 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 104-106 °C; \text{IR (KBr)} \(\nu\) 2965, 2153, 1574, 1278, 1110, 826, 724 cm\(^{-1}\); \(\text{\textit{1H NMR (300 MHz, CDCl}_3\) \(\delta\) 7.65 (s, 1H), 7.54 (d, \(J = 7.8\) Hz, 1H), 7.41 (d, \(J = 7.8\) Hz, 1H), 4.47 (dd, \(J = 8.1, 3.6\) Hz, 1H), 3.84 (dd, \(J = 18.0, 7.8\) Hz, 1H), 3.44-3.50 (m, 1H), 2.45 (s, 3H); \(\text{\textit{13C NMR (75 MHz, CDCl}_3\) \(\delta\) 199.8, 151.7, 136.4, 134.0, 128.6, 126.5, 125.0, 99.8, 44.2, 35.6; \text{HRMS (TOF-ESI\textsuperscript{+}) \(m/z\)}: calcd for \(\text{C}_{11}\text{H}_9\text{NOSeNa [M+Na]}^+\) \(273.9748\).}

6-Methoxyl-2-selenocyanato-3,4-dihydronaphthalen-1(2\textit{H})-one (\textit{5as}): Obtained in 99\% yield (27.8 mg, 0.099 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 125-126 °C; \text{IR (KBr)} \(\nu\) 2511, 2149, 1793, 1651, 1592, 1356, 1255, 1104,1021, 893 cm\(^{-1}\); \(\text{\textit{1H NMR (300 MHz, CDCl}_3\) \(\delta\) 7.99 (d, \(J = 9.0\) Hz, 1H), 6.89 (dd, \(J = 8.7, 1.8\) Hz, 1H), 6.76 (s, 1H), 4.99 (dd, \(J = 13.5, 5.1\) Hz, 1H), 3.90 (s, 3H), 3.13-3.17 (m, 2H), 2.87-2.92 (m, 1H), 2.48-2.60 (m, 1H); \(\text{\textit{13C NMR (75 MHz, CDCl}_3\) \(\delta\) 192.6, 164.9, 146.8, 130.4, 124.0, 114.1, 112.8, 102.8, 55.7, 53.1, 274.9748.}
2-Selenocyanato chromanone (5at): Obtained in 91% yield (23.0 mg, 0.091 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 93-94 °C; IR (KBr) ν 2928, 2851, 2153, 1799, 1664, 1602, 1203, 1140, 998, 599 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 7.83 (d, J = 8.4 Hz, 1H), 7.64 (t, J = 8.4 Hz, 2H), 4.92 (dd, J = 11.7, 4.2 Hz, 1H), 4.82-4.86 (m, 1H), 4.70 (dd, J = 11.4, 7.2 Hz, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 187.7, 161.7, 137.5, 127.6, 122.6, 119.2, 118.2, 99.6, 71.2, 47.1; HRMS (TOF-ESI⁺) m/z: calcd for C₁₀H₁₁NO₂SeNa [M+Na]⁺ 275.9534, found 275.9520.

N-Tosyl-2-selenocyanato-2,3-dihydro-4(1H)-quinolinone (5au): Obtained in 47% yield (19.1 mg, 0.047 mmol) according to procedure 1 after column chromatography as a white solid by using enamine; m.p. 114-116 °C; IR (KBr) ν 2921, 2155, 1679, 1475, 1370, 1223, 1163, 754 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.95 (t, J = 8.7 Hz, 2H), 7.65-7.72 (m, 3H), 7.35 (t, J = 7.8 Hz, 3H), 4.88 (dd, J = 14.1, 4.8 Hz, 1H), 4.46 (dd, J = 13.2, 5.1 Hz, 1H), 4.20 (t, J = 18.9 Hz, 1H), 2.46 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 189.2, 145.4, 142.6, 136.2, 130.6, 128.4, 127.0, 126.0, 124.3, 123.1, 99.7, 52.2, 47.0, 21.7; HRMS (TOF-ESI⁺) m/z: calcd for C₁₇H₁₄N₂O₃SSeNa [M+Na]⁺ 428.9739, found 428.9737.

2-Selenocyanato cyclohexanone (5av): Obtained in 86% yield (17.4 mg, 0.086 mmol) according to procedure 1 after column chromatography as a colorless oil by using enamine; IR (KBr) ν 2933, 2856, 2148, 1794, 1450, 1340, 1098, 939, 891 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 5.04 (dd, J = 12.6, 6.3 Hz, 1H), 2.54-2.59 (m, 2H), 2.40-2.45 (m, 1H), 1.87-1.91 (m, 1H), 1.62-1.77 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 207.8, 103.6, 55.9, 41.2, 37.0, 26.8, 25.6; HRMS (TOF-ESI⁺) m/z: calcd for C₇H₉NOSeNa [M+Na]⁺ 225.9742, found 225.9741.

2-Selenocyanato cycloheptanone (5aw): Obtained in 81% yield (17.6 mg, 0.081 mmol) according to procedure 1 after column chromatography as a colorless oil by using enamine; IR (KBr) ν 2923, 2861, 2150, 1794, 1450, 1340, 1200, 1098, 939, 891 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 5.22 (dd, J = 10.5, 3.6 Hz, 1H), 2.63-2.72 (m, 1H), 2.36-2.47 (m, 2H), 2.02 (dd, J = 25.5, 12.0 Hz, 1H), 1.80-1.84 (m, 2H), 1.68-1.72 (m, 2H), 1.53-1.61 (m, 1H), 1.34-1.45 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 207.8, 103.6, 55.9, 41.2, 37.0, 26.8, 25.6; HRMS (TOF-ESI⁺) m/z: calcd for C₈H₁₁NOSeK [M+K]⁺ 255.9637, found 255.9657.

1-Cyclohexyl-1-selenocyanatoethanone (5ax): Obtained in 71% yield (16.4 mg, 0.071 mmol) according to procedure 1 after column chromatography as a white solid
by using enamine; m.p. 43-44 °C; IR (KBr) v 2930, 2856, 2145, 1699, 1455, 1360, 1183, 1123, 894, 654 cm⁻¹; \(^1\)H NMR (300 MHz, DMSO-\(d_6\)) \(\delta\) 2.35 (s, 3H), 2.01-2.08 (m, 4H), 1.55-1.66 (m, 2H), 1.35-1.46 (m, 4H); \(^13\)C NMR (75 MHz, CDCl₃) \(\delta\) 203.1, 102.4, 64.9, 32.6, 24.8, 24.7, 23.8; HRMS (TOF-ESI⁺) m/z: calcd for C₉H₁₃NOSeNa [M+Na]⁺ 254.0055, found 254.0061.

3-Selenocyanato-4-phenyl-2-butanone (5ay): Obtained in 83% yield (21.0 mg, 0.083 mmol) according to procedure 1 after column chromatography as a white solid, by using enamine; m.p. 57-58 °C; IR (KBr) v 2921, 2846, 2517, 2155, 1799, 1647, 1465, 1270, 874, 694 cm⁻¹; \(^1\)H NMR (300 MHz, DMSO-\(d_6\)) \(\delta\) 7.31-7.35 (m, 2H), 7.24-7.27 (m, 3H), 4.41 (dd, \(J = 9.0, 6.0\) Hz, 1H), 3.38-3.43 (m, 1H), 3.01 (dd, \(J = 14.7, 9.3\) Hz, 1H), 2.32 (s, 3H); \(^13\)C NMR (75 MHz, DMSO-\(d_6\)) \(\delta\) 202.7, 138.5, 129.5, 128.8, 127.2, 103.5, 53.6, 36.1, 28.0; HRMS (TOF-ESI⁺) m/z: calcd for C₁₁H₁₁NOSeNa [M+Na]⁺ 275.9899, found 275.9899.

Pregnenolone acetate selenocyanate derivative (5az): Obtained in 92% yield (42.6 mg, 0.092 mmol) according to procedure 2 after column chromatography as a white solid by using silyl enol ethers; m.p. 153-154 °C; IR (KBr) v 2940, 2155, 1799, 1724, 1465, 1370, 1248, 1028, 874, 607 cm⁻¹; \(^1\)H NMR (300 MHz, CDCl₃) \(\delta\) 5.39 (d, \(J = 3.6\) Hz, 1H), 4.61-4.64 (m, 1H), 4.30 (s, 2H), 2.67 (t, \(J = 8.1\) Hz, 1H), 2.34-2.36 (m, 2H), 2.15-2.21 (m, 1H), 2.05 (s, 3H), 2.00-2.04 (m, 2H), 1.88-1.91 (m, 2H), 1.77-1.79 (m, 2H), 1.62-1.65 (m, 3H), 1.53-1.56 (m, 1H), 1.47-1.50 (m, 2H), 1.18-1.32 (m, 3H), 1.04 (s, 3H); \(^13\)C NMR (75 MHz, CDCl₃) \(\delta\) 204.7, 170.6, 139.7, 122.1, 101.8, 73.8, 65.7, 56.7, 49.7, 41.4, 38.7, 38.0, 37.0, 36.6, 31.8, 27.7, 24.4, 23.4, 21.4, 21.0, 19.3, 13.5; HRMS (TOF-ESI⁺) m/z: calcd for C₂₄H₃₃NO₃SeK [M+K]⁺ 502.1257, found 502.1244.

Estrone selenocyanate derivative (5ba): Obtained in 87% yield (33.8 mg, 0.087 mmol) according to procedure 2 after column chromatography as a white solid by using silyl enol ethers; m.p. 134-135 °C; IR (KBr) v 2928, 2153, 1796, 1724, 1609, 1499, 1377, 1255, 869, 669 cm⁻¹; \(^1\)H NMR (300 MHz, CDCl₃) \(\delta\) 7.21 (d, \(J = 8.4\) Hz, 1H), 6.75 (d, \(J = 7.3\) Hz, 1H), 6.68 (s, 1H), 4.65 (d, \(J = 6.8\) Hz, 1H), 3.80 (s, 3H), 2.93-2.94 (m, 2H), 2.43-2.50 (m, 2H), 2.32-2.39 (m, 1H), 1.91-2.05 (m, 3H), 1.45-1.67 (m, 5H), 1.07 (d, \(J = 13.8\) Hz, 3H); \(^13\)C NMR (75 MHz, CDCl₃) \(\delta\) 213.5, 157.8, 137.5, 131.4, 126.3, 113.9, 111.7, 101.3, 55.2, 48.4, 47.5, 45.7, 43.7, 37.9, 31.7, 31.3, 29.4, 26.3, 25.6, 14.0; HRMS (TOF-ESI⁺) m/z: calcd for C₂₀H₂₃NO₂SeNa [M+Na]⁺ 412.0786, found 412.0758.

Cholesterol selenocyanate derivative (5bb): Obtained in 73% yield (35.7 mg, 0.073 mmol) according to procedure 2 after column chromatography as a white solid by using silyl enol ethers; m.p. 100-101 °C; IR (KBr) v 2938, 2148, 1799, 1649, 1562, 1467, 1228, 1013,
874, 667 cm\(^{-1}\); \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 5.95 (s, 1H), 4.83 (s, 1H), 2.54-2.63 (m, 2H), 2.08-2.22 (m, 3H), 1.52-1.89 (m, 8H), 1.43 (s, 3H), 1.28-1.35 (m, 6H), 1.09-1.19 (m, 6H), 1.01-1.06 (m, 2H), 0.94 (d, \(J = 6.3\) Hz, 3H), 0.90 (s, 3H), 0.88 (s, 3H), 0.77 (s, 3H); \(^13\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 198.5, 163.1, 128.4, 102.0, 56.1, 55.2, 52.6, 49.8, 42.5, 39.5, 39.3, 38.2, 37.4, 37.1, 36.1, 35.7, 34.1, 31.6, 28.0, 24.1, 23.8, 22.8, 22.5, 21.2, 20.9, 18.6, 12.0; HRMS (TOF-ESI\(^+\)) m/z: calcd for C\(_{28}\)H\(_{43}\)NOSeNa [M+Na]\(^+\) 512.2402, found 512.2417.

2.3 Synthetic procedure for the synthesis of 2-thiocyanato-3,4-dihydronaphthalen-1(2\(H\))-one (7)

The N-acetyl-\(\alpha\)-arylenamines 4a (0.1 mmol) and Bi-SCN (36.6 mg, 0.15 mmol, 1.5 equiv.) was ground together with agate mortar at room temperature for the given time. After the completion of the reaction, the mixture was dissolved in EtOAc (2 mL). Then the mixture was extracted with EtOAc (2 \(\times\) 2 mL) and washed with saturated aqueous NaHCO\(_3\). The combined organic phase were dried (Na\(_2\)SO\(_4\)) and concentrated in vacuo. The resulting residue was purified by flash column chromatography (PE:EtOAc = 9:1-5:1) to afford the desired selenocyanate compound as a white solid.

\textit{Caution: This reaction should be carried out behind a safety shield!}

2-thiocyanato-3,4-dihydronaphthalen-1(2\(H\))-one (7): white solid (64% yield, 13.0 mg, 0.064 mmol); m.p. 56-57 °C (lit. 56-58 °C); IR (KBr) \(\nu\) 2923, 1796, 1607, 1460, 1225, 1150, 983, 764 cm\(^{-1}\); \(^1\)H NMR (300 MHz, DMSO-d\(_6\)) \(\delta\) 7.91 (d, \(J = 8.1\) Hz, 1H), 7.65 (t, \(J = 7.5\) Hz, 1H), 7.41-7.43 (m, 2H), 5.17 (dd, \(J = 13.2, 4.8\) Hz, 1H), 3.22 (dd, \(J = 12.3, 4.2\) Hz, 1H), 3.09-3.14 (m, 1H), 2.61-2.66 (m, 1H), 2.33 (dd, \(J = 12.3, 4.2\) Hz, 1H); \(^13\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 191.7, 143.5, 134.9, 130.6, 129.0, 128.0, 127.5, 111.8, 55.8, 31.5, 29.1; HRMS (TOF-ESI\(^+\)) m/z: calcd for C\(_{11}\)H\(_9\)NOSK [M+K]\(^+\) 242.0036, found 242.0026.

2.4 Synthetic procedure for the synthesis of 2-trifluoromethylselenocyanato-3,4-dihydronaphthalen-1(2\(H\))-one (8)

To a dry two-neck flask were added \(\alpha\)-selenocyanate tetralone (25.0 mg, 0.1 mmol) and dry THF (2 mL). The flask was evacuated and refilled with Ar three times. After the reaction was cooled to 0 °C, TMSCF\(_3\) (28.4 mg, 0.2 mmol, 2.0 equiv.) and TBAF in 1 M THF (20 \(\mu\)L, 0.02
mmol, 0.2 equiv.) were added dropwise respectively. After 30 min at 0 °C under Ar atmosphere, the reaction was allowed to warm to 23 °C and stirred for 12 h. The reaction mixture was then extracted with EtOAc (2 × 2 mL) and washed with brine. The combined organic layers were dried (MgSO$_4$) and concentrated in vacuo. The resulting residue was purified by flash column chromatography (PE:EtOAc = 19:1) to afford the desired product as a colorless liquid.

2-Trifluoromethylselenocyanato-3,4-dihydronaphthalen-1(2H)-one (8): colorless oil (83% yield, 24.4 mg, 0.083 mmol); IR (KBr) ν 2923, 2512, 2153, 1671, 1450, 1320, 891, 742 cm$^{-1}$; $^1$H NMR (300 MHz, DMSO-d$_6$) δ 7.90 (d, $J$ = 7.5 Hz, 1H), 7.62 (t, $J$ = 7.2 Hz, 1H), 7.41 (t, $J$ = 7.2 Hz, 2H), 5.13 (dd, $J$ = 11.1, 4.5 Hz, 1H), 3.15-3.18 (m, 1H), 3.01-3.06 (m, 1H), 2.60-2.66 (m, 1H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 193.6, 143.2, 134.3, 131.0, 128.8, 128.1, 127.2, 122.9 (d, $^1J_{C,F}$ = 329.2 Hz), 49.4, 31.5, 29.0; $^{19}$F NMR (282 MHz, CDCl$_3$) δ -32.2 (s); HRMS (TOF-ESI$^+$) m/z: calcd for C$_{11}$H$_9$F$_3$OSeNa [M+Na]$^+$ 316.9663, found 316.9649.

3. References


4. Spectra

\[ \text{3 (DMSO-}d_6\text{, 300 MHz)} \]

\[ \text{3 (DMSO-}d_6\text{, 75 MHz)} \]
S19

SeCN

COOEt

$\text{Sad (CDCl}_3, 300 \text{ MHz)}$

SeCN

COOEt

$\text{Sad (CDCl}_3, 75 \text{ MHz)}$
S20

SeCN
COOMe

5ae (CDCl₃, 300 MHz)

SeCN
COOMe

5ae (CDCl₃, 75 MHz)
$$\text{SeCN}$$

$$(\text{DMSO-d}_6, 300 \text{ MHz})$$

$$(\text{CDCl}_3, 75 \text{ MHz})$$
$\text{SeCN}$

5ai (CDCl$_3$, 300 MHz)

$\text{SeCN}$

5ai (CDCl$_3$, 75 MHz)
5ak (DMSO-d$_6$, 300 MHz)

5ak (DMSO-d$_6$, 75 MHz)
$\text{SeCN}$

5am (CDCl$_3$, 300 MHz)

$\text{SeCN}$

5am (CDCl$_3$, 75 MHz)

S28
$\text{BrSeCN}$

5aq (DMSO-$d_6$, 300 MHz)

5aq (CDCl$_3$, 75 MHz)
$\text{SeCN}$

$5\text{ax}$ (DMSO-$d_6$, 300 MHz)

$\text{SeCN}$

$5\text{ax}$ (CDCl$_3$, 75 MHz)
5ay (DMSO-d$_6$, 300 MHz)

5ay (DMSO-d$_6$, 75 MHz)
$\text{AcO}$

$\text{H}$

$\text{H}$

$\text{H}$

$\text{SeCN}$

$\text{5az}$ (CDCl$_3$, 300 MHz)

$\text{AcO}$

$\text{H}$

$\text{H}$

$\text{H}$

$\text{SeCN}$

$\text{5az}$ (CDCl$_3$, 75 MHz)
$\text{MeO}$

$5\text{ba}$ (CDCl$_3$, 300 MHz)

$\text{MeO}$

$5\text{ba}$ (CDCl$_3$, 75 MHz)
7 (DMSO-$_d_6$, 300 MHz)

7 (CDCl$_3$, 75 MHz)
$^{19}$F NMR 
$\delta$ (CDCl$_3$, 282 MHz)
5. X-Ray Crystal Structures

![Chemical structure](image)

**Fig. S1** X-ray structures of 5am (with 20% probability level)

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**Fig. S2** X-ray structures of **5az** (with 20% probability level)

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<td>Cell volume</td>
<td>1146.62(13)</td>
<td>Space group: P2(1)</td>
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</tr>
<tr>
<td>Data completeness</td>
<td>1.78/0.96</td>
<td>Theta(max) = 27.550</td>
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<tr>
<td>R(reflections)</td>
<td>0.0362( 4061)</td>
<td>WR2(reflections) = 0.0849( 5088)</td>
<td></td>
</tr>
<tr>
<td>S</td>
<td>1.023</td>
<td>Radiation type: MoK'a</td>
<td></td>
</tr>
<tr>
<td>Measurement device type:</td>
<td>CCD area detector</td>
<td>Measurement method: phi and omega scans</td>
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<tr>
<td>Structure solution</td>
<td>SHELXS-97</td>
<td>Structure refinement: SHELXL-97</td>
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<tr>
<td>Solution primary</td>
<td>direct</td>
<td>Solution secondary: difmap</td>
<td></td>
</tr>
<tr>
<td>Solution hydrogens</td>
<td>geom</td>
<td>Hydrogen treatment: mixed</td>
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