Supplementary information

Efficient synthesis of multicyclic spirooxindoles via a cascade Michael/Michael/oxa-Michael reaction of curcumins and isatylidene malononitriles

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1. Screening of chiral organocatalysts and reaction solvents

Table 1 Screening of chiral organocatalysts and solvents

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>Solvent</th>
<th>dr&lt;sup&gt;b&lt;/sup&gt;</th>
<th>Time (h)</th>
<th>Yield (%)&lt;sup&gt;c&lt;/sup&gt;</th>
<th>Ee (%)&lt;sup&gt;d&lt;/sup&gt;</th>
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<td>4a</td>
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<td>24</td>
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<tr>
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<td>24</td>
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<sup>a</sup> The reactions were carried out with 1a (0.05 mmol), 2a (0.05 mmol) and catalyst (0.01 mmol) in solvent (1 mL) at room temperature.  
<sup>b</sup> Determined by <sup>1</sup>H NMR analysis of the crude product.  
<sup>c</sup> Isolated yields after column chromatography.  
<sup>d</sup> Determined by HPLC with a Chiralpak AS-H.  
<sup>e</sup> Not determined.  
<sup>f</sup> No reaction.
Chiral HPLC chromatogram

Chirapak AS-H column (4.6 mm × 25 cm), hexane/2-PrOH = 90: 10, λ = 254 nm, 1.0 mL/min; t\textsubscript{major} = 36.6 min, t\textsubscript{minor} = 49.4 min).

Racemic 3a

\begin{center}
\includegraphics[width=\textwidth]{chart1.png}
\end{center}

\begin{tabular}{|c|c|c|c|c|c|}
\hline
Pen & Ret. Time & Area & Height & Area % & Height % \\
\hline
1 & 36.643 & 3408671 & 15078 & 79.212 & 81.486 \\
2 & 49.420 & 894042 & 1326 & 20.788 & 18.514 \\
\hline
Total & 430713 & 18504 & 100.000 & 100.000 \\
\hline
\end{tabular}

3a (58% ee)

\begin{center}
\includegraphics[width=\textwidth]{chart2.png}
\end{center}

\begin{tabular}{|c|c|c|c|c|c|}
\hline
Pen & Ret. Time & Area & Height & Area % & Height % \\
\hline
1 & 36.643 & 3408671 & 15078 & 79.212 & 81.486 \\
2 & 49.420 & 894042 & 1326 & 20.788 & 18.514 \\
\hline
Total & 430713 & 18504 & 100.000 & 100.000 \\
\hline
\end{tabular}

1. General Methods

\(^1\)H and \(^{13}\)C NMR spectra were recorded on a Bruker Advance 400 MHz spectrometer as solutions in CDCl\textsubscript{3} or DMSO-d\textsubscript{6}. Chemical shifts in \(^1\)H NMR spectra are reported in parts per million (ppm, \(\delta\)) downfield from the internal standard Me\textsubscript{4}Si (TMS, \(\delta = 0\) ppm). Chemical shifts in \(^{13}\)C NMR spectra are reported relative to the central line of the chloroform signal (\(\delta = 77.0\) ppm). The following abbreviations are used to designate chemical shift multiplicities: \textit{s} = singlet, \textit{d} = doublet, \textit{m} = multiplet.

High-resolution mass spectra were obtained with Shimadzu LCMS-IT-TOF mass spectrometer. Optical rotations were measured on a Perkin-Elmer 341 digital polarimeter and are reported as \([\alpha]_D^{20}\) (c in gram per 100 mL of solvent). Infrared (IR) spectra were recorded on a Bruker Tensor 37 spectrophotometer. Data are represented as follows: frequency of absorption (cm\(^{-1}\)), intensity of absorption (\textit{s} = strong, \textit{m} = medium, \textit{w} = weak). The crystallographic data were obtained with
Oxford Diffraction Xcalibur Nova diffractometer. Melting points were recorded on an electrothermal digital melting point apparatus and were uncorrected. TLC analysis was performed on precoated silica gel GF254 slides, and visualised by either UV irradiation. The flash column chromatography was carried out over silica gel (230–400 mesh), purchased from Qingdao Haiyang Chemical Co. Ltd. Unless otherwise stated, all reagents were obtained from commercial sources and used as received. The solvents were used as commercial anhydrous grade without further purification. Enantiomeric excesses were determined by HPLC using a Daicel Chiralpak AS-H column (4.6 mm × 25 cm) and eluting with hexane/2-ProH solution. Curcumins and isatylidene malononitriles were prepared according to the reported procedures.1,2

2. Typical procedures

2.1 Typical procedure for the reaction of isatylidene malononitriles and curcumins

A mixture of DMAP (0.01 mmol), isatylidene malononitrile 1a (0.05 mmol) and curcumin 2a (0.05 mmol) in ethanol (1 mL) was stirred at room temperature for 18 h. After the solvent was evaporated under vacuum, the residue was purified by flash column chromatography over silica gel (petroleum ether/EtOAc = 2: 1) to provide product 3a as a yellow solid.

2.2 Asymmetric reaction of isatylidene malononitrile 1a and curcumin 2a

A mixture of Takemoto’s catalyst (0.01 mmol), isatylidene malononitrile 1a (0.05 mmol) and curcumin 2a (0.05 mmol) in dichloromethane (1 mL) was stirred at room temperature for 48 h. After the solvent was evaporated under vacuum, the residue was purified by flash column chromatography over silica gel (petroleum ether/AcOEt = 2: 1) to provide product 3a as a yellow solid. The enantiomeric excess of 3a was determined by HPLC with a Chiralpak AS-H column (4.6 mm × 25 cm) (hexane/2-ProH = 90: 10, λ = 254 nm, 1.0 mL/min); t_major= 27.2 min, t_minor = 35.1 min, 58% ee.

3. Spectroscopic data of 3a-3n

3.1 2',4-dioxo-2,7-diphenyl-3,4,7,8-tetrahydrospiro[chromene-5,3'-indoline]-6,6(2H)-dicarbonitrile (3a)

Yellow solid, mp 174-176 °C ; 1H NMR (400 MHz, CDCl3) δ 8.23 (s, 1H), 7.59 – 7.31 (m, 12H), 7.13 (t, J = 7.7 Hz, 1H), 6.93 (d, J = 7.7 Hz, 1H), 5.50 (dd, J = 14.9, 3.0 Hz, 1H), 4.72 (dd, J = 12.4, 4.7 Hz, 1H), 3.41 (dd, J = 19.4, 12.4 Hz, 1H), 3.01 (dd, J = 19.4, 4.7 Hz, 1H), 2.91 (dd, J = 17.9, 15.0 Hz, 1H), 2.61 (dd, J = 17.9, 3.0 Hz, 1H); 13C NMR (100 MHz, CDCl3) δ 188.45, 174.58, 173.30, 168.48, 141.54, 136.76, 134.62, 130.32, 129.58, 129.38, 129.28, 129.00, 128.75, 127.20, 126.37, 124.52, 123.20, 112.34, 111.44, 111.10, 110.80, 81.10, 53.38, 47.73, 42.75, 39.13, 31.79; IR (KBr) ν/cm⁻¹: 3450, 2350, 1739, 1679; HRMS (ESI) calcd for C30H20N3O3 (M-H)-: 470.1504, found: 470.1503.

3.2 5'-methoxy-2',4-dioxo-2,7-diphenyl-3,4,7,8-tetrahydrospiro[chromene-5,3'-indoline]-6,6(2H)-dicarbonitrile (3b)

Yellow solid. mp 174-176 °C ; 1H NMR (400 MHz, DMSO) δ 11.07 (s, 1H), 7.69 – 7.35 (m, 10H), 7.08 (s, 1H), 6.92 (d, J = 1.2 Hz, 2H), 5.80 (dd, J = 14.6, 2.8 Hz, 1H), 4.60 (dd, J = 12.3, 4.8 Hz, 1H), 3.75 (s, 3H), 3.47 – 3.39 (m, 1H), 3.16 (dd, J = 19.4, 4.8 Hz, 1H), 2.93 (dd, J = 17.6, 14.8 Hz, 1H), 2.59 (dd, J = 17.6, 3.1 Hz, 1H); 13C NMR (100 MHz, DMSO) δ 188.05, 174.43, 172.55, 154.74, 137.50, 136.07, 135.10, 129.22, 129.00, 128.81, 128.70, 128.56, 126.81, 113.77, 112.20, 112.11, 110.47, 110.07, 80.15, 79.12, 55.40, 53.03, 47.61, 41.93, 30.51; IR (KBr) ν/cm⁻¹: 3490,3035,2961,2250,1729,1677;HRMS (ESI) calcd for C31H22N3O4 (M-H): 470.1504, found: 470.1503.

3.3 5'-chloro-2',4-dioxo-2,7-diphenyl-3,4,7,8-tetrahydrospiro[chromene-5,3'-indoline]-6,6(2H)-dicarbonitrile(3c)
3.4 7’-bromo-2’,4-dioxo-2,7-diphenyl-3,4,7,8-tetrahydrospiro[chromene-5,3’-indoline]-6,6(2H)-dicarbonitrile (3d)

Yellow solid. mp 153-155°C; 1H NMR (400 MHz, CDCl3) δ 8.28 (s, 1H), 7.58 – 7.40 (m, 12H), 7.06 (t, J = 7.8 Hz, 1H), 5.52 (d, J = 14.8 Hz, 1H), 4.75 – 4.65 (m, 1H), 3.43 (dd, J = 19.3, 12.5 Hz, 1H), 3.08 – 2.86 (m, 2H), 2.64 (d, J = 17.9 Hz, 1H); 13C NMR (100 MHz, CDCl3) δ 188.28, 173.63, 173.32, 141.05, 136.65, 134.37, 133.13, 129.70, 129.44, 129.35, 129.04, 128.75, 128.50, 126.37, 124.37, 123.39, 112.17, 111.13, 110.74, 103.75, 81.18, 54.70, 47.48, 42.59, 39.18, 31.72; IR (KBr) ν/cm⁻¹: 3482, 2922, 2852, 2360, 1732, 1678; HRMS (ESI) calcd for C30H20N3O3Br (M-H)⁻: 548.0615, found: 548.0608.

3.5 6’-methoxy-2’,4-dioxo-2,7-diphenyl-3,4,7,8-tetrahydrospiro[chromene-5,3’-indoline]-6,6(2H)-dicarbonitrile (3e)

Yellow solid. mp 169-171°C; 1H NMR (400 MHz, DMSO) δ 11.41 (s, 1H), 7.57 (d, J = 7.0 Hz, 2H), 7.53 – 7.38 (m, 10H), 7.04 (d, J = 8.3 Hz, 1H), 5.87 (dd, J = 14.6, 2.7 Hz, 1H), 4.56 (dd, d, J = 12.2, 4.8 Hz, 1H), 3.50 – 3.41 (m, 1H), 2.95 (dd, d, J = 14.6, 2.7 Hz, 1H), 2.61 (dd, d, J = 17.6, 3.0 Hz, 1H); 13C NMR (100 MHz, DMSO) δ 188.27, 174.35, 173.00, 141.91, 137.44, 134.88, 129.87, 129.46, 129.31, 129.04, 128.84, 128.71, 128.57, 128.63, 125.93, 124.57, 112.10, 111.97, 111.62, 109.49, 80.21, 52.89, 47.33, 41.76, 30.44; IR (KBr) ν/cm⁻¹: 3490, 2366, 1721, 1675; HRMS (ESI) calcd for C30H19N3O3Cl (M-H)⁻: 504.1115, found: 504.1111.

3.6 1’-methyl-2’,4-dioxo-2,7-diphenyl-3,4,7,8-tetrahydrospiro[chromene-5,3’-indoline]-6,6(2H)-dicarbonitrile (3f)

Yellow solid. mp 140-142°C; 1H NMR (400 MHz, CDCl3) δ 7.60 – 7.37 (m, 12H), 7.16 (td, J = 7.7, 0.8 Hz, 1H), 7.00 (d, J = 7.8 Hz, 1H), 5.48 (dd, J = 15.0, 3.1 Hz, 1H), 4.78 (dd, d, J = 12.5, 4.9 Hz, 1H), 3.41 (dd, d, J = 19.3, 12.5 Hz, 1H), 3.00 (dd, d, J = 19.3, 4.9 Hz, 1H), 2.86 (dd, d, J = 17.8, 15.0 Hz, 1H), 2.56 (dd, d, J = 17.8, 3.2 Hz, 1H); 13C NMR (100 MHz, CDCl3) δ 187.97, 173.44, 173.03, 144.43, 136.85, 134.68, 130.36, 129.56, 129.35, 129.25, 129.01, 128.77, 126.75, 126.31, 124.15, 123.27, 112.42, 111.48, 110.93, 109.14, 81.06, 53.11, 47.80, 42.80, 39.17, 31.73, 27.08; IR (KBr) ν/cm⁻¹: 3480, 3061, 2922, 1720, 1678; IR (KBr) ν/cm⁻¹: 3480, 3061, 2922, 1720, 1678; HRMS (ESI) calcd for C31H22N3O3 (M-H)⁻: 484.1661, found: 484.1657.

3.7 1’-benzyl-2’,4-dioxo-2,7-diphenyl-3,4,7,8-tetrahydrospiro[chromene-5,3’-indoline]-6,6(2H)-dicarbonitrile (3g)

White solid. mp 135-137°C; 1H NMR (400 MHz, CDCl3) δ 7.72 – 7.28 (m, 17H), 7.16 (td, J = 7.6, 1H), 6.81 (d, J = 7.8 Hz, 1H), 5.53 (dd, J = 14.9, 2.8 Hz, 1H), 5.20 (d, J = 16.1 Hz, 1H), 5.02 (d, J = 16.1 Hz, 1H), 4.86 (dd, J = 12.4, 4.8 Hz, 1H), 3.47 (dd, J = 19.3, 12.4 Hz, 1H), 3.05 (dd, J = 19.3, 4.9 Hz, 1H), 2.95 (dd, J = 17.7, 15.1 Hz, 1H), 2.63 (dd, J = 17.8, 3.0 Hz, 1H); 13C NMR (100 MHz, CDCl3) δ 187.97, 173.84, 173.03, 143.85, 136.85, 134.86, 134.68, 130.24, 129.59, 129.38, 129.28, 129.03, 128.82, 127.64, 127.21, 126.74, 126.38, 124.17, 123.33, 112.45, 111.71, 111.18, 110.47, 81.11, 53.08, 47.84, 45.21, 42.82, 39.30, 31.81; IR (KBr) ν/cm⁻¹: 3480, 3064, 3033, 2921, 2852, 2250, 1729, 1677; HRMS
3.8 2,7-bis(4-methoxyphenyl)-2',4-dioxo-3,4,7,8-tetrahydrospiro[chromene-5,3'-indoline]-6,6(2H)-dicarbonitrile(3h)

Yellow solid. mp 191-194 °C; 1H NMR (400 MHz, CDCl3) δ 8.43 (br, 1H), 7.43 (d, J = 25.7, 21.0, 7.7 Hz, 6H), 7.14 (t, J = 7.6 Hz, 1H), 7.04 – 6.86 (m, 5H), 5.46 (dd, J = 15.0, 2.7 Hz, 1H), 4.68 (dd, J = 12.3, 4.7 Hz, 1H), 3.84 (s, 3H), 3.82 (s, 3H), 3.37 (dd, J = 19.4, 12.5 Hz, 1H), 3.07 – 2.86 (m, 2H), 2.59 (dd, J = 17.9, 2.9 Hz, 1H); 13C NMR (100 MHz, CDCl3) δ 188.83, 174.74, 173.55, 160.44, 160.39, 141.62, 130.24, 129.93, 128.73, 128.14, 127.35, 126.54, 124.44, 123.12, 114.63, 114.36, 112.46, 111.65, 110.95, 110.83, 80.90, 55.38, 55.28, 53.39, 48.11, 42.45, 38.52, 31.93; HRMS (ESI) calcd for C32H24N3O5 (M-H)-: 530.1716, found: 530.1718.

3.9 2,7-bis(4-chlorophenyl)-2',4-dioxo-3,4,7,8-tetrahydrospiro[chromene-5,3'-indoline]-6,6(2H)-dicarbonitrile(3i)

Yellow solid. mp 191-194 °C; 1H NMR (400 MHz, CDCl3) δ 8.45 (s, 1H), 7.51 (d, J = 7.6 Hz, 1H), 7.49 – 7.27 (m, 9H), 7.13 (t, J = 7.6 Hz, 1H), 6.91 (d, J = 7.8 Hz, 1H), 5.48 (dd, J = 14.8, 2.9 Hz, 1H), 4.71 (dd, J = 12.4, 4.8 Hz, 1H), 3.35 (dd, J = 19.3, 12.4 Hz, 1H), 2.99 (dd, J = 19.3, 4.9 Hz, 1H), 2.86 (dd, J = 17.8, 15.0 Hz, 1H), 2.59 (dd, J = 17.9, 3.2 Hz, 1H). 13C NMR (100 MHz, CDCl3) δ 188.12, 174.54, 172.86, 141.57, 135.78, 135.38, 135.14, 132.98, 130.47, 130.07, 129.58, 129.26, 127.74, 126.94, 124.44, 123.29, 112.12, 111.30, 111.20, 110.96, 80.32, 53.28, 47.57, 42.58, 38.58, 31.67; IR(KBr) ν/cm⁻¹: 3450, 2926, 1729, 1678; HRMS (ESI) calcd for C30H18N3O3Cl2 (M-H)-: 538.0725, found: 538.0722.

3.10 2,7-bis(2-methoxyphenyl)-2',4-dioxo-3,4,7,8-tetrahydrospiro[chromene-5,3'-indoline]-6,6(2H)-dicarbonitrile(3j)

Yellow solid. mp 244°C, decompose; 1H NMR (400 MHz, CDCl3) δ 8.02 (s, 1H), 7.56 (d, J = 7.5 Hz, 2H), 7.47 (d, J = 7.5 Hz, 1H), 7.35 (dd, J = 13.1, 6.7 Hz, 3H), 7.13 (t, J = 7.7 Hz, 1H), 7.06 – 7.00 (m, 2H), 6.99 – 6.88 (m, 3H), 5.85 (dd, J = 13.7, 4.2 Hz, 1H), 5.47 (dd, J = 12.5, 4.6 Hz, 1H), 3.86 (s, 3H), 3.85 (s, 3H), 3.36 (dd, J = 19.0, 12.6 Hz, 1H), 2.93 (dd, J = 19.2, 4.7 Hz, 1H), 2.71 (m, 2H); 13C NMR (100 MHz, CDCl3) δ 189.09, 174.45, 173.90, 157.78, 155.89, 141.55, 130.34, 130.11, 129.92, 127.51, 126.51, 125.70, 124.66, 123.27, 123.00, 120.94, 112.68, 111.67, 111.29, 110.99, 110.64, 110.46, 76.22, 55.57, 55.35, 53.51, 47.17, 41.89, 31.38; IR (KBr) ν/cm⁻¹: 3479, 3414, 2357, 1732, 1637, 1618; HRMS (ESI) calcd for C28H20N2O5 (M-H)-: 530.1716, found: 530.1716.

3.11 2,7-bis(2-chlorophenyl)-2',4-dioxo-3,4,7,8-tetrahydrospiro[chromene-5,3'-indoline]-6,6(2H)-dicarbonitrile(3k)

Yellow solid. mp 206-209°C; 1H NMR (400 MHz, CDCl3) δ 8.29 (s, 1H), 7.77 (d, J = 7.5 Hz, 1H), 7.65 – 7.32 (m, 9H), 7.17 (t, J = 7.7 Hz, 1H), 6.98 (d, J = 7.8 Hz, 1H), 5.93 (dd, J = 13.2, 4.7 Hz, 1H), 5.63 (dd, J = 12.3, 4.9 Hz, 1H), 3.34 (dd, J = 19.2, 12.3 Hz, 1H), 3.06 (dd, J = 19.2, 4.9 Hz, 1H), 2.86 – 2.69 (m, 2H); 13C NMR (100 MHz, CDCl3) δ 187.95, 174.07, 172.80, 141.63, 135.74, 134.92, 132.62, 131.76, 130.72, 130.49, 130.44, 130.14, 129.88, 127.98, 127.72, 127.58, 127.21, 126.82, 124.61, 123.21, 112.42, 111.38, 110.90, 110.83, 77.91, 53.35, 46.59, 41.65, 34.38, 32.03; HRMS (ESI) calcd for C26H18N2O5Cl (M-H)-: 530.0725, found: 530.0725.

3.12 2,7-bis(2,6-dimethoxyphenyl)-2',4-dioxo-3,4,7,8-tetrahydrospiro[chromene-5,3'-indolin]-6,6(2H)-dicarbonitrile(3l)

Yellow solid. mp 175-177°C; 1H NMR (400 MHz, DMSO) δ 11.15 (s, 1H), 7.43 (d, J = 7.5 Hz, 1H), 7.34 (dd, J = 7.7, 1.0 Hz, 1H), 7.17 (dd, J = 11.1, 2.7 Hz, 2H), 7.05 (m, 3H), 6.96 (m, 3H), 5.85 (dd, J = 14.6, 3.1 Hz, 1H), 5.26 (dd, J = 12.5, 4.7 Hz, 1H), 3.78 (s, 3H), 3.74 (s, 3H), 3.73 (s, 3H), 3.73 (s, 3H),
3.52 – 3.40 (m, 1H), 3.05 (dd, J = 19.4, 4.9 Hz, 1H), 2.92 (dd, J = 17.7, 14.7 Hz, 1H), 2.52 – 2.49 (m, 1H); ¹³C NMR (100 MHz, DMSO) δ 188.04, 174.17, 173.11, 153.26, 153.15, 151.46, 150.01, 143.00, 129.77, 127.61, 126.16, 124.31, 124.23, 121.78, 114.62, 114.42, 113.84, 113.19, 112.83, 112.71, 112.54, 112.16, 110.01, 109.90, 79.12, 75.30, 56.10, 55.46, 55.37, 52.74, 46.99, 40.96, 30.28; HRMS (ESI) calcd for C₃₄H₂₈N₃O₇ (M-H): 590.1927, found: 590.1928.

3.13 2,7-di(furan-2-yl)-2',4-dioxo-3,4,7,8-tetrahydrospiro[chromene-5,3'-indoline]-6,6(2H)-dicarbonitrile (3m)
Yellow solid. mp 155-157°C ; ¹H NMR (400 MHz, CDCl₃) δ 8.11 (s, 1H), 7.50 (d, J = 7.7 Hz, 3H), 7.36 (d, J = 7.2 Hz, 1H), 7.14 (d, J = 7.1 Hz, 1H), 6.97 (d, J = 7.2 Hz, 1H), 6.63 – 6.33 (m, 4H), 5.52 (dd, J = 14.9, 2.1 Hz, 1H), 4.86 (dd, J = 11.9, 5.0 Hz, 1H), 3.39 (dd, J = 19.4, 12.0 Hz, 1H), 3.14 (dd, J = 17.8, 14.9 Hz, 1H), 2.98 (dd, J = 19.4, 5.0 Hz, 1H), 2.61 (dd, J = 17.8, 2.1 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 187.70, 174.29, 172.19, 148.92, 148.14, 144.01, 143.90, 141.42, 130.38, 126.85, 124.45, 123.28, 111.86, 111.28, 110.82, 110.74, 110.49, 110.37, 73.54, 52.85, 46.16, 39.06, 34.03, 30.56; IR (KBr) ν/cm⁻¹: 3419,2924,1728,1618; HRMS (ESI) calcd for C₂₆H₁₆N₃O₅ (M-H): 450.1090, found: 450.1091.

3.14 2',4-dioxo-2,7-di(thiophen-2-yl)-3,4,7,8-tetrahydrospiro[chromene-5,3'-indoline]-6,6(2H)-dicarbonitrile (3n)
Yellow solid. mp 244°C, decompose; ¹H NMR (400 MHz, DMSO) δ 11.25 (s, 1H), 7.72 – 7.56 (m, 2H), 7.44 (d, J = 7.5 Hz, 3H), 7.39 – 7.28 (m, 3H), 7.18 – 6.96 (m, 4H), 6.02 (dd, J = 14.5, 3.1 Hz, 1H), 4.86 (dd, J = 11.7, 5.3 Hz, 1H), 3.32 – 3.16 (m, 2H), 3.01 (dd, J = 17.7, 14.6 Hz, 1H), 2.71 (dd, J = 17.7, 3.2 Hz, 1H); ¹³C NMR (100 MHz, DMSO) δ 187.53, 174.47, 171.54, 142.92, 139.37, 137.18, 129.95, 128.13, 127.50, 127.44, 127.30, 127.07, 127.04, 124.32, 121.97, 112.15, 112.10, 110.22, 75.56, 52.61, 48.30, 41.98, 34.78, 32.53; IR (KBr) ν/cm⁻¹: 3478,2924,2354,1728,1670,1618; HRMS (ESI) calcd for C₂₆H₁₆N₃O₃S₂ (M-H): 482.0633, found: 482.0636.
3H NMR of Sn

13C NMR of Sn
References of supplementary information


2 For the synthesis of isatylidene malononitriles, see: H. Liu, G. L. Dou and D. Q. Shi, J. Comb. Chem., 2010, 12, 292.