Design, synthesis and biological evaluation of esculetin derivatives as anti-tumour agents

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Table of Contents

<table>
<thead>
<tr>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. General Experimental</td>
<td>S2</td>
</tr>
<tr>
<td>2. $^1$H NMR and $^{13}$C NMR spectra for compound 2</td>
<td>S2</td>
</tr>
<tr>
<td>3. $^1$H NMR spectra for compound 3</td>
<td>S3</td>
</tr>
<tr>
<td>4. $^1$H NMR and $^{13}$C NMR spectra for compound 4</td>
<td>S4</td>
</tr>
<tr>
<td>5. $^1$H NMR spectra for compound 5</td>
<td>S5</td>
</tr>
<tr>
<td>6. $^1$H NMR spectra for compound 6</td>
<td>S5</td>
</tr>
<tr>
<td>7. $^1$H NMR, $^{13}$C NMR, HSQC, HMBC and HRMS spectra for compound 8</td>
<td>S6</td>
</tr>
<tr>
<td>8. $^1$H NMR, $^{13}$C NMR, HSQC, HMBC and HRMS spectra for compound 9</td>
<td>S8</td>
</tr>
<tr>
<td>9. $^1$H NMR, $^{13}$C NMR and HRMS spectra for compound 10</td>
<td>S10</td>
</tr>
<tr>
<td>10. $^1$H NMR, $^{13}$C NMR and HRMS spectra for compound 11</td>
<td>S11</td>
</tr>
<tr>
<td>11. $^1$H NMR spectra for compound 12</td>
<td>S12</td>
</tr>
<tr>
<td>12. $^1$H NMR, $^{13}$C NMR and HRMS spectra for compound 13</td>
<td>S13</td>
</tr>
<tr>
<td>13. $^1$H NMR, $^{13}$C NMR and HRMS spectra for compound 14</td>
<td>S15</td>
</tr>
<tr>
<td>14. $^1$H NMR, $^{13}$C NMR and HRMS spectra for compound 15</td>
<td>S16</td>
</tr>
</tbody>
</table>
1. General Experimental

The $^1$H NMR and $^{13}$C NMR spectra were recorded in DMSO-$d_6$ using a Bruker ARX 400 spectrometer (400 MHz for $^1$HNMR and 100 MHz for $^{13}$CNMR), and chemical shifts were expressed as ppm against TMS as an internal reference. High-resolution mass spectral (HRMS) analyses were measured with Hybrid Ion Trap-Orbitrap Mass Spectrometer (LTQ Orbitrap XL, Thermo). A UFLC system (Shimadzu, Kyoto, Japan) with tandem mass spectrometry (2010EV), using electrospray ionization (ESI) interface and a computer equipped with UFLC-MS solution software (version 3.41; Shimadzu). All reagents used in the synthesis were obtained commercially and used without further purification. The reactions were monitored by thin layer chromatography (TLC) on glass-packed precoated silica gel GF$_{254}$ plates and visualized in an iodine chamber or with a UV lamp. Flash column chromatography was performed using silica gel (200–300 mesh) purchased from Qingdao Haiyang Chemical Co. Ltd.

2. $^1$H NMR and $^{13}$C NMR spectra for compound 2

$^1$H NMR (400 MHz, DMSO-$d_6$)
3. $^{1}H$ NMR Spectra for compound 3

$^{13}C$ NMR (100 MHz, DMSO-$d_6$)

$^{1}H$ NMR (400 MHz, DMSO-$d_6$)
4. $^1$H NMR and $^{13}$C NMR Spectra for compound 4

$^1$H NMR (400 MHz, DMSO-$d_6$)

$^{13}$C NMR (100 MHz, DMSO-$d_6$)
5. $^1$H NMR Spectra for compound 5

$^1$H NMR (400 MHz, DMSO-$d_6$)

6. $^1$H NMR spectra for compound 6

$^1$H NMR (400 MHz, DMSO-$d_6$)
7. $^1$H NMR, $^{13}$C NMR, HSQC, HMBC and HRMS spectra for compound 8

$^1$H NMR (400 MHz, DMSO-$d_6$)

$^{13}$C NMR (100 MHz, DMSO-$d_6$)
8. $^1$H NMR, $^{13}$C NMR, HSQC, HMBC and HRMS spectra for compound 9

$^1$H NMR (400 MHz, DMSO-$d_6$)

$^{13}$C NMR (100 MHz, DMSO-$d_6$)
9. \(^1\)H NMR, \(^{13}\)C NMR and HRMS spectra for compound 10

\(^1\)H NMR (400 MHz, DMSO-\(d_6\))

\(^{13}\)C NMR (100 MHz, DMSO-\(d_6\))
10. $^1$H NMR spectra for compound 11

$^1$H NMR (400 MHz, DMSO-$d_6$)
11. $^1$H NMR, $^{13}$C NMR and HRMS spectra for compound 12

$^1$H NMR (400 MHz, DMSO-$d_6$)

$^{13}$C NMR (100 MHz, DMSO-$d_6$)
12. $^1$H NMR, $^{13}$C NMR and HRMS spectra for compound 13

$^1$H NMR (400 MHz, DMSO-$d_6$)
$^{13}$C NMR (100 MHz, DMSO-$d_6$)

HRMS (ESI)
13. $^1$H NMR, $^{13}$C NMR and HRMS spectra for compound 14

$^1$H NMR (400 MHz, DMSO-$d_6$)

$^{13}$C NMR (100 MHz, DMSO-$d_6$)
14. $^1$H NMR, $^{13}$C NMR and HRMS spectra for compound 15

$^1$H NMR (400 MHz, DMSO-$d_6$)