

# Supplementary Information

## **Rapid Mining of Novel $\alpha$ -Glucosidase and Lipase Inhibitors from *Streptomyces* sp. HO1518 Using UPLC-QTOF-MS/MS**

**Jianlin Xu <sup>1,2,3,†</sup>, Zhifeng Liu <sup>2,3,†</sup>, Zhanguang Feng <sup>2,3</sup>, Yuhong Ren <sup>1</sup>, Haili Liu <sup>2,\*</sup> and Yong Wang <sup>1,2,\*</sup>**

<sup>1</sup>*State Key Laboratory of Bioreactor Engineering, East China University of Science and Technology, Shanghai 200237, China*

<sup>2</sup>*CAS-Key Laboratory of Synthetic Biology, CAS Center for Excellence in Molecular Plant Sciences, Institute of Plant Physiology and Ecology, Chinese Academy of Sciences, Shanghai 200032, China*

<sup>3</sup>*University of Chinese Academy of Sciences, Beijing 100039, China*

\*Corresponding authors. Tel./fax: +86 21 54924295.

E-mail addresses: yongwang@cemps.ac.cn (Yong Wang); hlliu@cemps.ac.cn (Haili Liu).

† These authors contributed equally to this work.

## Table of Contents

<b>Table S1</b> $^1\text{H}$ (500 MHz) and $^{13}\text{C}$ (125 MHz) NMR data of <b>10–13</b> ( $\delta$ ppm) in $\text{D}_2\text{O}$ .....	8
<b>Table S2</b> $^1\text{H}$ (500 MHz) and $^{13}\text{C}$ (125 MHz) NMR data of <b>14–16</b> ( $\delta$ ppm) in $\text{D}_2\text{O}$ . ....	10
<b>Table S3</b> $^1\text{H}$ (500 MHz) and $^{13}\text{C}$ (125 MHz) NMR data of <b>10</b> ( $\delta$ ppm) in $\text{D}_2\text{O}$ .....	12
<b>Table S4</b> $^1\text{H}$ (500 MHz) and $^{13}\text{C}$ (125 MHz) NMR data of <b>11</b> ( $\delta$ ppm) in $\text{D}_2\text{O}$ .....	14
<b>Table S5</b> $^1\text{H}$ (500 MHz) and $^{13}\text{C}$ (125 MHz) NMR data of <b>12</b> ( $\delta$ ppm) in $\text{D}_2\text{O}$ .....	16
<b>Table S6</b> $^1\text{H}$ (500 MHz) and $^{13}\text{C}$ (125 MHz) NMR data of <b>13</b> ( $\delta$ ppm) in $\text{D}_2\text{O}$ .....	18
<b>Table S7</b> $^1\text{H}$ (500 MHz) and $^{13}\text{C}$ (125 MHz) NMR data of <b>14</b> ( $\delta$ ppm) in $\text{D}_2\text{O}$ .....	20
<b>Table S8</b> $^1\text{H}$ (500 MHz) and $^{13}\text{C}$ (125 MHz) NMR data of <b>15</b> ( $\delta$ ppm) in $\text{D}_2\text{O}$ .....	22
<b>Table S9</b> $^1\text{H}$ (500 MHz) and $^{13}\text{C}$ (125 MHz) NMR data of <b>16</b> ( $\delta$ ppm) in $\text{D}_2\text{O}$ .....	24
<b>Table S10</b> Acarviostatins with glucose(s) at the reducing terminus (Aca-glu) from <i>Streptomyces</i> sp. HO1518. ....	26
<b>Table S11</b> Acarviostatins with glucose(s) at the reducing and nonreducing terminus (glu-Aca-glu) from <i>Streptomyces</i> sp. HO1518 <sup>a</sup> .....	28
<b>Table S12</b> Acarviostatins with an incomplete pseudo-trisaccharide at the nonreducing terminus (incAca-glu) from <i>Streptomyces</i> sp. HO1518 <sup>a</sup> . ....	29
<b>Figure S1</b> HRESI-MS/MS spectra of compounds <b>1–9</b> .....	31
<b>Figure S2.</b> Positive HRESIMS/MS fragmentation and spectra of <b>9</b> and <b>14–16</b> . (A) Positive-ion HRESIMS/MS fragmentation patterns of <b>9</b> and <b>14–16</b> ; (B–E) HRESIMS/MS spectra of <b>9</b> and <b>14–16</b> .....	32
<b>Figure S3</b> The inhibitory activities of <b>10–16</b> against three digestive enzymes. (A) The inhibitory activities of <b>10–16</b> against PPA. (B) The inhibitory activities of <b>10–16</b> against sucrase. (C) The inhibitory activities of <b>10–16</b> against PL. ....	33
<b>Figure S4</b> HRESI-MS/MS spectra of compounds <b>17–20</b> .....	34
<b>Figure S5</b> The total ion chromatograms of fraction 1 and 2 derived from <i>Streptomyces</i> sp. HO1518. ....	34
<b>Figure S6</b> The EIC of acarviostatins with glucose at the reducing terminus from <i>Streptomyces</i> sp. HO1518.....	36
<b>Figure S7</b> The EIC of acarviostatins with glucose at the reducing and nonreducing terminus from <i>Streptomyces</i> sp. HO1518.....	36
<b>Figure S8</b> The EIC of acarviostatins with an incomplete <i>pseudo</i> -tetrasaccharide at the nonreducing terminus from <i>Streptomyces</i> sp. HO1518.....	37
<b>Figure S9</b> Positive HRESIMS/MS fragmentation and spectra of Ac-Aca I12 and Ac-Aca I03. (A) Positive-ion HRESIMS/MS fragmentation patterns of Ac-Aca I12 and Ac-Aca I03; (B) The extracted ion chromatogram of aminooligosaccharides at $m/z$ 1012; (C) HRESIMS/MS spectrum of Ac-Aca I12; (D) HRESIMS/MS spectrum of Ac-Aca I03.....	38
<b>Figure S10</b> $^1\text{H}$ NMR spectrum of compound <b>10</b> (500 MHz, $\text{D}_2\text{O}$ ).....	39
<b>Figure S11</b> 1D-selective TOCSY spectrum of compound <b>10</b> (500 MHz, $\text{D}_2\text{O}$ , excitation at $\delta$ 5.23, H-A1 $\alpha$ ). ....	40
<b>Figure S12</b> 1D-selective TOCSY spectrum of compound <b>10</b> (500 MHz, $\text{D}_2\text{O}$ , excitation at $\delta$ 4.65, H-A1 $\beta$ ).....	41
<b>Figure S13</b> 1D-selective TOCSY spectrum of compound <b>10</b> (500 MHz, $\text{D}_2\text{O}$ , excitation at $\delta$ 5.41, H-B1, and H-C1). ....	42

<b>Figure S14</b> 1D-selective TOCSY spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.22, H-C6).....	43
<b>Figure S15</b> 1D-selective TOCSY spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.33, H-D1 and H-G1). .....	44
<b>Figure S16</b> 1D-selective TOCSY spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.98, H-E7).....	45
<b>Figure S17</b> 1D-selective TOCSY spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.38, H-F1).....	46
<b>Figure S18</b> 1D-selective TOCSY spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.90, H-H7). .....	47
<b>Figure S19</b> <sup>13</sup> C NMR spectrum of compound <b>10</b> (125 MHz, D <sub>2</sub> O).....	48
<b>Figure S20</b> DEPT-135 spectrum of compound <b>10</b> (125 MHz, D <sub>2</sub> O).....	49
<b>Figure S21</b> HSQC spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O). .....	50
<b>Figure S22</b> <sup>1</sup> H- <sup>1</sup> H COSY spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O).....	51
<b>Figure S23</b> 2D-TOCSY spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O).....	52
<b>Figure S24</b> HSQC-TOCSY spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O).....	53
<b>Figure S25</b> HMBC spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O). .....	54
<b>Figure S26</b> NOESY spectrum of compound <b>10</b> (500 MHz, D <sub>2</sub> O).....	55
<b>Figure S27</b> HRESIMS spectrum of compound <b>10</b> . .....	56
<b>Figure S28</b> UV spectrum of compound <b>10</b> . .....	56
<b>Figure S29</b> IR spectrum of compound <b>10</b> .....	57
<b>Figure S30</b> <sup>1</sup> H NMR spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O).....	58
<b>Figure S31</b> 1D-selective TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.66, H-A1 $\beta$ ).....	59
<b>Figure S32</b> 1D-selective TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.41, H-B1 and H-C1). .....	60
<b>Figure S33</b> 1D-selective TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.44, H-C6a).....	61
<b>Figure S34</b> 1D-selective TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.29, H-D1).....	62
<b>Figure S35</b> 1D-selective TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.98, H-E7).....	63
<b>Figure S36</b> 1D-selective TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.38, H-F1).....	64
<b>Figure S37</b> 1D-selective TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.32, H-G1). .....	65
<b>Figure S38</b> 1D-selective TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.90, H-H7). .....	66
<b>Figure S39</b> <sup>13</sup> C NMR spectrum of compound <b>11</b> (125 MHz, D <sub>2</sub> O).....	67
<b>Figure S40</b> DEPT-135 spectrum of compound <b>11</b> (125 MHz, D <sub>2</sub> O).....	68
<b>Figure S41</b> HSQC spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O). .....	69
<b>Figure S42</b> <sup>1</sup> H- <sup>1</sup> H COSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O).....	70
<b>Figure S43</b> 2D-TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O).....	71
<b>Figure S44</b> HSQC-TOCSY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O).....	72

<b>Figure S45</b> HMBC spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O).	73
<b>Figure S46</b> NOESY spectrum of compound <b>11</b> (500 MHz, D <sub>2</sub> O).	74
<b>Figure S47</b> HRESIMS spectrum of compound <b>11</b> .	75
<b>Figure S48</b> UV spectrum of compound <b>11</b> .	75
<b>Figure S49</b> IR spectrum of compound <b>11</b> .	76
<b>Figure S50</b> <sup>1</sup> H NMR spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O).	77
<b>Figure S51</b> 1D-selective TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.24, H-A1 $\alpha$ ).	78
<b>Figure S52</b> 1D-selective TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.66, H-A1 $\beta$ ).	79
<b>Figure S53</b> 1D-selective TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.38, H-B1 and H-C1).	80
<b>Figure S54</b> 1D-selective TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.44, H-C6a).	81
<b>Figure S55</b> 1D-selective TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.28, H-D1).	82
<b>Figure S56</b> 1D-selective TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.98, H-E1).	83
<b>Figure S57</b> 1D-selective TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.42, H-F1).	84
<b>Figure S58</b> 1D-selective TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.33, H-G1).	85
<b>Figure S59</b> 1D-selective TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.91, H-H1).	86
<b>Figure S60</b> <sup>13</sup> C NMR spectrum of compound <b>12</b> (125 MHz, D <sub>2</sub> O).	87
<b>Figure S61</b> DEPT-135 spectrum of compound <b>12</b> (125 MHz, D <sub>2</sub> O).	88
<b>Figure S62</b> HSQC spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O).	89
<b>Figure S63</b> <sup>1</sup> H- <sup>1</sup> H COSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O).	90
<b>Figure S64</b> 2D-TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O).	91
<b>Figure S65</b> HSQC-TOCSY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O).	92
<b>Figure S66</b> HMBC spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O).	93
<b>Figure S67</b> NOESY spectrum of compound <b>12</b> (500 MHz, D <sub>2</sub> O).	94
<b>Figure S68</b> HRESIMS spectrum of compound <b>12</b> .	95
<b>Figure S69</b> UV spectrum of compound <b>12</b> .	95
<b>Figure S70</b> IR spectrum of compound <b>12</b> .	96
<b>Figure S71</b> <sup>1</sup> H NMR spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O).	97
<b>Figure S72</b> 1D-selective TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.24, H-A1 $\alpha$ ).	98
<b>Figure S73</b> 1D-selective TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.66, H-A1 $\beta$ ).	99
<b>Figure S74</b> 1D-selective TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.41, H-B1 and H-C1).	100
<b>Figure S75</b> 1D-selective TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.49, H-C6).	101



<b>Figure S76</b> 1D-selective TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.29, H-D1).....	102
<b>Figure S77</b> 1D-selective TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.99, H-E7).....	103
<b>Figure S78</b> 1D-selective TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.38, H-F1).....	104
<b>Figure S79</b> 1D-selective TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.33, H-G1). ....	105
<b>Figure S80</b> 1D-selective TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.91, H-H7). ....	106
<b>Figure S81</b> <sup>13</sup> C NMR spectrum of compound <b>13</b> (125 MHz, D <sub>2</sub> O).....	107
<b>Figure S82</b> DEPT-135 spectrum of compound <b>13</b> (125 MHz, D <sub>2</sub> O).....	108
<b>Figure S83</b> HSQC spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O). ....	109
<b>Figure S84</b> <sup>1</sup> H- <sup>1</sup> H COSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O).....	110
<b>Figure S85</b> 2D-TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O).....	111
<b>Figure S86</b> HSQC-TOCSY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O).....	112
<b>Figure S87</b> HMBC spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O). ....	113
<b>Figure S88</b> NOESY spectrum of compound <b>13</b> (500 MHz, D <sub>2</sub> O).....	114
<b>Figure S89</b> HRESIMS spectrum of compound <b>13</b> . ....	115
<b>Figure S90</b> UV spectrum of compound <b>13</b> . ....	115
<b>Figure S91</b> IR spectrum of compound <b>13</b> .....	116
<b>Figure S92</b> <sup>1</sup> H NMR spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O).....	117
<b>Figure S93</b> 1D-selective TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.20, H-A1 $\alpha$ ). ....	118
<b>Figure S94</b> 1D-selective TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.62, H-A1 $\beta$ ).....	119
<b>Figure S95</b> 1D-selective TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.38, H-B1, H-C1, and H-D1).....	120
<b>Figure S96</b> 1D-selective TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.40, H-D6a).....	121
<b>Figure S97</b> 1D-selective TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.25, H-E1).....	122
<b>Figure S98</b> 1D-selective TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.94, H-F7).....	123
<b>Figure S99</b> 1D-selective TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.34, H-G1). ....	124
<b>Figure S100</b> 1D-selective TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.29, H-H1). ....	125
<b>Figure S101</b> 1D-selective TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.86, H-I1).....	126
<b>Figure S102</b> <sup>13</sup> C NMR spectrum of compound <b>14</b> (125 MHz, D <sub>2</sub> O).....	127
<b>Figure S103</b> DEPT-135 spectrum of compound <b>14</b> (125 MHz, D <sub>2</sub> O).....	128
<b>Figure S104</b> HSQC spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O). ....	129
<b>Figure S105</b> <sup>1</sup> H- <sup>1</sup> H COSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O).....	130

<b>Figure S106</b> 2D-TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O).....	131
<b>Figure S107</b> HSQC-TOCSY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O).....	132
<b>Figure S108</b> HMBC spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O). ....	133
<b>Figure S109</b> NOESY spectrum of compound <b>14</b> (500 MHz, D <sub>2</sub> O).....	134
<b>Figure S110</b> HRESIMS spectrum of compound <b>14</b> . ....	135
<b>Figure S111</b> UV spectrum of compound <b>14</b> . ....	135
<b>Figure S112</b> IR spectrum of compound <b>14</b> . ....	136
<b>Figure S113</b> <sup>1</sup> H NMR spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O).....	137
<b>Figure S114</b> 1D-selective TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.23, H-A1 $\alpha$ ). ....	138
<b>Figure S115</b> 1D-selective TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.65, H-A1 $\beta$ ).....	139
<b>Figure S116</b> 1D-selective TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.40, H-B1, C1, and D1). ....	140
<b>Figure S117</b> 1D-selective TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.45, H-D6a).....	141
<b>Figure S118</b> 1D-selective TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.28, H-E1).....	142
<b>Figure S119</b> 1D-selective TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.97, H-F1). ....	143
<b>Figure S120</b> 1D-selective TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.37, H-G1). ....	144
<b>Figure S121</b> 1D-selective TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.32, H-H1). ....	145
<b>Figure S122</b> 1D-selective TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.90, H-I1). ....	146
<b>Figure S123</b> <sup>13</sup> C NMR spectrum of compound <b>15</b> (125 MHz, D <sub>2</sub> O).....	147
<b>Figure S124</b> DEPT-135 spectrum of compound <b>15</b> (125 MHz, D <sub>2</sub> O).....	148
<b>Figure S125</b> HSQC spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O). ....	149
<b>Figure S126</b> <sup>1</sup> H- <sup>1</sup> H COSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O).....	150
<b>Figure S127</b> 2D-TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O).....	151
<b>Figure S128</b> HSQC-TOCSY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O).....	152
<b>Figure S129</b> HMBC spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O). ....	153
<b>Figure S130</b> NOESY spectrum of compound <b>15</b> (500 MHz, D <sub>2</sub> O).....	154
<b>Figure S131</b> HRESIMS spectrum of compound <b>15</b> . ....	155
<b>Figure S132</b> UV spectrum of compound <b>15</b> . ....	155
<b>Figure S133</b> IR spectrum of compound <b>15</b> . ....	156
<b>Figure S134</b> <sup>1</sup> H NMR spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O).....	157
<b>Figure S135</b> 1D-selective TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.20, H-A1 $\alpha$ ). ....	158
<b>Figure S136</b> 1D-selective TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.63, H-A1 $\beta$ ).....	159
<b>Figure S137</b> 1D-selective TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.38, H-B1, H-C1, H-D1).....	160

<b>Figure S138</b> 1D-selective TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 4.43, H-D6a).....	161
<b>Figure S139</b> 1D-selective TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.24, H-E1).....	162
<b>Figure S140</b> 1D-selective TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.94, H-F7).....	163
<b>Figure S141</b> 1D-selective TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.34, H-G1). ....	164
<b>Figure S142</b> 1D-selective TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.30, H-H1). ....	165
<b>Figure S143</b> 1D-selective TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O, excitation at $\delta$ 5.87, H-I7).....	166
<b>Figure S144</b> <sup>13</sup> C NMR spectrum of compound <b>16</b> (125 MHz, D <sub>2</sub> O).....	167
<b>Figure S145</b> DEPT-135 spectrum of compound <b>16</b> (125 MHz, D <sub>2</sub> O).....	168
<b>Figure S146</b> HSQC spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O). ....	169
<b>Figure S147</b> <sup>1</sup> H- <sup>1</sup> H COSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O).....	170
<b>Figure S148</b> 2D-TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O).....	171
<b>Figure S149</b> HSQC-TOCSY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O).....	172
<b>Figure S150</b> HMBC spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O). ....	173
<b>Figure S151</b> NOESY spectrum of compound <b>16</b> (500 MHz, D <sub>2</sub> O).....	174
<b>Figure S152</b> HRESIMS spectrum of compound <b>16</b> . ....	175
<b>Figure S153</b> UV spectrum of compound <b>16</b> . ....	175
<b>Figure S154</b> IR spectrum of compound <b>16</b> .....	176

**Table S1.** <sup>1</sup>H (500 MHz) and <sup>13</sup>C (125 MHz) NMR data of **10–13** (ppm) in D<sub>2</sub>O.

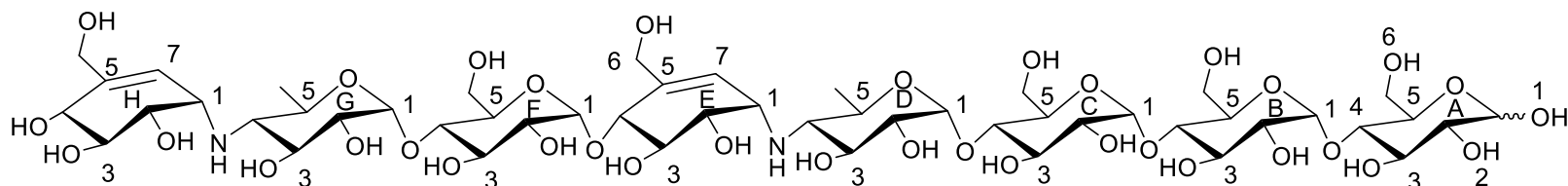
No.	10		11		12		13	
	$\delta_C$	$\delta_H$	$\delta_C$	$\delta_H$	$\delta_C$	$\delta_H$	$\delta_C$	$\delta_H$
A1 $\alpha$	93.4, CH	5.23, d (3.5, 1H)	95.3, CH	5.23, d (3.5, 1H)	91.9, CH	5.24, d (3.4, 1H)	92.0, CH	5.24, d (3.5, 1H)
A2 $\alpha$	71.2, CH	3.57, m (1H)	74.8, CH	3.58, m (1H)	71.3, CH	3.52, m (1H)	71.4, CH	3.51, m (1H)
A3 $\alpha$	73.2, CH	4.09, m (1H)	76.7, CH	3.97, m (1H)	73.3, CH	4.16, m (1H)	73.4, CH	3.92, m (1H)
A4 $\alpha$	76.8, CH	3.68, m (1H)	80.2, CH	3.65, m (1H)	76.8, CH	3.69, m (1H)	76.9, CH	3.69, m (1H)
A5 $\alpha$	70.0, CH	3.97, m (1H)	73.3, CH	3.87, m (1H)	70.0, CH	3.90, m (1H)	70.0, CH	3.82, m (1H)
A6 $\alpha$	60.5, CH <sub>2</sub>	3.85, m (2H)	63.8, CH <sub>2</sub>	3.87, m (2H)	60.5, CH <sub>2</sub>	3.74, m (2H)	60.5, CH <sub>2</sub>	3.79, m (2H)
A1 $\beta$	95.8, CH	4.65, d (8.0, 1H)	99.2, CH	4.66, d (8.0, 1H)	95.8, CH	4.66, d (8.0, 1H)	95.8, CH	4.66, d (7.8, 1H)
A2 $\beta$	74.0, CH	3.27, t (9.0, 1H)	77.4, CH	3.28, t (9.0, 1H)	74.0, CH	3.29, m (1H)	74.0, CH	3.28, m (1H)
A3 $\beta$	76.2, CH	3.76, m (1H)	79.7, CH	3.77, m (1H)	76.3, CH	3.78, m (1H)	76.2, CH	3.77, m (1H)
A4 $\beta$	76.9, CH	3.65, m (1H)	80.2, CH	3.66, m (1H)	76.8, CH	3.67, m (1H)	76.8, CH	3.66, m (1H)
A5 $\beta$	74.6, CH	3.60, m (1H)	77.9, CH	3.62, m (1H)	74.6, CH	3.62, m (1H)	74.6, CH	3.60, m (1H)
A6 $\beta$	60.5, CH <sub>2</sub>	3.90, m (2H)	64.1, CH <sub>2</sub>	3.91, m (2H)	60.6, CH <sub>2</sub>	3.92, m (2H)	60.5, CH <sub>2</sub>	3.90, m (2H)
B1	99.5, CH	5.41, d (3.5, 1H)	102.8, CH	5.41, d (3.5, 1H)	99.6, CH	5.38, d (3.5, 1H)	99.4, CH	5.41, d (3.5, 1H)
B2	71.5, CH	3.62, m (1H)	74.9, CH	3.64, m (1H)	71.6, CH	3.58, m (1H)	71.6, CH	3.62, m (1H)
B3	73.4, CH	3.95, m (1H)	76.7, CH	3.96, m (1H)	73.4, CH	3.94, m (1H)	73.2, CH	3.96, m (1H)
B4	77.0, CH	3.67, m (1H)	80.4, CH	3.67, m (1H)	77.1, CH	3.63, m (1H)	77.1, CH	3.64, m (1H)
B5	71.2, CH	3.85, m (1H)	74.6, CH	3.85, m (1H)	71.2, CH	3.80, m (1H)	71.3, CH	3.85, m (1H)
B6	60.7, CH <sub>2</sub>	3.85, m (2H)	63.9, CH <sub>2</sub>	3.85, m (2H)	60.7, CH <sub>2</sub>	3.80, m (2H)	60.7, CH <sub>2</sub>	3.85, m (2H)
C1	99.4, CH	5.41, d (3.5, 1H)	103.0, CH	5.41, d (3.5, 1H)	99.4, CH	5.38, d (3.5, 1H)	99.6, CH	5.41, d (3.5, 1H)
C2	71.0, CH	3.66, m (1H)	74.5, CH	3.67, m (1H)	71.1, CH	3.62, m (1H)	71.2, CH	3.62, m (1H)
C3	73.3, CH	3.83, m (1H)	76.6, CH	3.95, m (1H)	73.2, CH	3.92, m (1H)	73.2, CH	3.96, m (1H)
C4	77.0, CH	3.68, m (1H)	81.3, CH	3.67, m (1H)	78.2, CH	3.62, m (1H)	78.0, CH	3.64, m (1H)
C5	69.4, CH	4.04, d (12.0, 1H)	72.3, CH	4.05, d (12.0, 1H)	69.0, CH	4.03, d (12.0, 1H)	69.0, CH	4.05, d (12.0, 1H)
C6a	63.2, CH <sub>2</sub>	4.15, m (1H)	63.8, CH <sub>2</sub>	4.22, m (1H)	63.5, CH <sub>2</sub>	4.20, m (1H)	63.4, CH <sub>2</sub>	4.23, m (1H)
C6b		4.23, d (12.0, 1H)		4.44, d (12.0, 1H)		4.45, d (12.0, 1H)		4.49, d (12.0, 1H)
D1	99.9, CH	5.33, d (3.5, 1H)	104.0, CH	5.29, d (3.5, 1H)	100.7, CH	5.28, d (3.5, 1H)	100.6, CH	5.29, d (3.5, 1H)
D2	71.2, CH	3.56, m (1H)	74.6, CH	3.56, m (1H)	71.2, CH	3.54, m (1H)	71.3, CH	3.58, m (1H)
D3	72.7, CH	3.59, m (1H)	76.3, CH	3.59, m (1H)	73.0, CH	3.56, m (1H)	73.0, CH	3.60, m (1H)
D4	64.2, CH	2.48, t (9.0, 1H)	67.7, CH	2.48, t (9.0, 1H)	64.3, CH	2.47, t (8.8, 1H)	64.3, CH	2.47, t (9.0, 1H)
D5	69.7, CH	3.76, m (1H)	73.2, CH	3.73, m (1H)	69.8, CH	3.71, m (1H)	69.8, CH	3.75, m (1H)
D6	17.4, CH <sub>3</sub>	1.34, d (5.7, 3H)	20.7, CH <sub>3</sub>	1.30, d (5.7, 3H)	17.4, CH <sub>3</sub>	1.31, d (5.6, 3H)	17.4, CH <sub>3</sub>	1.32, d (5.6, 3H)
E1	55.0, CH	3.54, m (1H)	58.4, CH	3.53, m (1H)	55.0, CH	3.53, m (1H)	55.0, CH	3.48, m (1H)
E2	70.7, CH	3.82, m (1H)	73.0, CH	3.80, m (1H)	70.8, CH	3.80, m (1H)	69.7, CH	3.79, m (1H)
E3	70.8, CH	4.13, m (1H)	74.1, CH	4.13, m (1H)	70.9, CH	4.14, m (1H)	70.8, CH	4.14, m (1H)
E4	76.1, CH	4.22, m (1H)	79.6, CH	4.22, m (1H)	76.2, CH	4.23, m (1H)	76.4, CH	4.23, m (1H)
E5	136.5, C		139.9, C		136.5, C		136.5, C	
E6a	62.0, CH <sub>2</sub>	4.13, m (1H)	65.4, CH <sub>2</sub>	4.13, m (1H)	62.0, CH <sub>2</sub>	4.14, m (1H)	62.0, CH <sub>2</sub>	4.14, m (1H)
E6b		4.22, m (1H)		4.22, m (1H)		4.23, m (1H)		4.23, m (1H)

E7	126.3, CH	5.98, d (3.2, 1H)	129.7, CH	5.98, d (3.2, 1H)	126.3, CH	5.98, d (3.1, 1H)	126.4, CH	5.99, d (3.2, 1H)
F1	97.5, CH	5.38, d (3.4, 1H)	101.0, CH	5.38, d (3.4, 1H)	97.8, CH	5.42, d (3.5, 1H)	97.6, CH	5.38, d (3.5, 1H)
F2	72.9, CH	3.58, m (1H)	74.9, CH	3.62, m (1H)	73.2, CH	3.68, m (1H)	72.7, CH	3.61, m (1H)
F3	73.5, CH	3.93, m (1H)	76.9, CH	3.93, m (1H)	73.5, CH	3.94, m (1H)	73.5, CH	3.93, m (1H)
F4	70.8, CH	3.60, m (1H)	74.2, CH	3.64, m (1H)	70.9, CH	3.66, m (1H)	70.9, CH	3.61, m (1H)
F5	71.1, CH	3.95, m (1H)	74.5, CH	3.93, m (1H)	71.2, CH	3.96, m (1H)	71.2, CH	3.93, m (1H)
F6	60.4, CH <sub>2</sub>	3.81, m (2H)	66.9, CH <sub>2</sub>	3.81, m (2H)	60.4, CH <sub>2</sub>	3.82, m (2H)	60.7, CH <sub>2</sub>	3.82, m (2H)
G1	99.6, CH	5.33, d (3.4, 1H)	103.3, CH	5.32, d (3.4, 1H)	99.9, CH	5.33, d (3.4, 1H)	100.0, CH	5.33, d (3.4, 1H)
G2	71.3, CH	3.56, m (1H)	74.7, CH	3.58, m (1H)	71.2, CH	3.61, m (1H)	71.5, CH	3.58, m (1H)
G3	72.2, CH	3.59, m (1H)	75.9, CH	3.62, m (1H)	72.6, CH	3.61, m (1H)	72.6, CH	3.60, m (1H)
G4	64.9, CH	2.48, t (9.0, 1H)	68.3, CH	2.48, t (9.0, 1H)	64.9, CH	2.47, t (8.8, 1H)	65.0, CH	2.47, t (9.0, 1H)
G5	69.6, CH	3.86, m (1H)	73.0, CH	3.76, m (1H)	69.6, CH	3.77, m (1H)	69.6, CH	3.75, m (1H)
G6	17.3, CH <sub>3</sub>	1.34, d (5.7, 3H)	20.8, CH <sub>3</sub>	1.34, d (5.7, 3H)	17.3, CH <sub>3</sub>	1.35, d (5.6, 3H)	17.4, CH <sub>3</sub>	1.35, d (5.6, 3H)
H1	56.0, CH	3.52, m (1H)	59.4, CH	3.54, m (1H)	56.0, CH	3.54, m (1H)	56.0, CH	3.48, m (1H)
H2	72.6, CH	3.72, m (1H)	76.1, CH	3.68, m (1H)	72.7, CH	3.69, m (1H)	72.8, CH	3.67, m (1H)
H3	72.6, CH	3.75, m (1H)	76.3, CH	3.77, m (1H)	72.9, CH	3.77, m (1H)	73.0, CH	3.76, m (1H)
H4	70.9, CH	4.07, d (12.0, 1H)	74.3, CH	4.04, d (12.0, 1H)	71.0, CH	4.06, d (12.0, 1H)	71.0, CH	4.05, d (12.0, 1H)
H5	139.0, C		142.3, C		139.0, C		139.0, C	
H6a				4.12, m (1H)		4.13, m (1H)		4.12, m (1H)
H6b	61.6, CH <sub>2</sub>	4.12, m (2H)	65.0 CH <sub>2</sub>	4.22, m (1H)	61.6, CH <sub>2</sub>	4.25, m (1H)	61.6, CH <sub>2</sub>	4.23, m (1H)
H7	123.7, CH	5.90, d (3.5, 1H)	127.2, CH	5.90, d (3.5, 1H)	123.8, CH	5.91, d (3.4, 1H)	123.8, CH	5.91, d (3.4, 1H)
1'			180.8, C=O		180.1, C=O		176.2, C=O	
2'			30.6, CH <sub>2</sub>	2.48, m (2H)	33.9, CH	2.72, m (2H)	42.9, CH <sub>2</sub>	2.36, m (2H)
3'			11.7, CH <sub>3</sub>	1.13, d (7.2, 3H)	18.3, CH <sub>3</sub>	1.19, d (3.0, 3H)	25.5, CH	2.08, m (1H)
4'					18.2, CH <sub>3</sub>	1.19, d (3.0, 3H)	21.7, CH <sub>3</sub>	0.96, d (6.6, 3H)
5'							21.7, CH <sub>3</sub>	0.96, d (6.6, 3H)

**Table S2.** <sup>1</sup>H (500 MHz) and <sup>13</sup>C (125 MHz) NMR data of **14–16** ( ppm) in D<sub>2</sub>O.

No.	14		15		16	
	$\delta_C$	$\delta_H$	$\delta_C$	$\delta_H$	$\delta_C$	$\delta_H$
A1 $\alpha$	94.7, CH	5.20, d (3.5, 1H)	91.9, CH	5.22, d (3.5, 1H)	91.9, CH	5.21, d (3.5, 1H)
A2 $\alpha$	74.2, CH	3.53, m (1H)	71.4, CH	3.52, m (1H)	71.3, CH	3.50, m (1H)
A3 $\alpha$	76.1, CH	3.93, m (1H)	73.3, CH	3.93, m (1H)	73.3, CH	3.92, m (1H)
A4 $\alpha$	79.6, CH	3.61, m (1H)	76.8, CH	3.62, m (1H)	76.8, CH	3.64, m (1H)
A5 $\alpha$	72.8, CH	3.85, m (1H)	69.9, CH	3.85, m (1H)	69.9, CH	3.84, m (1H)
A6 $\alpha$	63.2, CH <sub>2</sub>	3.78, m (2H)	60.4, CH <sub>2</sub>	3.79, m (2H)	60.4, CH <sub>2</sub>	3.78, m (2H)
A1 $\beta$	98.6, CH	4.62, d (8.0, 1H)	95.8, CH	4.65, d (8.0, 1H)	95.8, CH	4.63, d (8.0, 1H)
A2 $\beta$	76.8, CH	3.24, t (9.0, 1H)	74.0, CH	3.27, m (1H)	74.0, CH	3.25, m (1H)
A3 $\beta$	79.1, CH	3.74, m (1H)	76.3, CH	3.77, m (1H)	76.2, CH	3.73, m (1H)
A4 $\beta$	79.7, CH	3.63, m (1H)	76.8, CH	3.65, m (1H)	76.8, CH	3.64, m (1H)
A5 $\beta$	77.4, CH	3.61, m (1H)	74.5, CH	3.60, m (1H)	74.5, CH	3.61, m (1H)
A6 $\beta$	63.3, CH <sub>2</sub>	3.88, m (2H)	60.5, CH <sub>2</sub>	3.89, m (2H)	60.4, CH <sub>2</sub>	3.88, m (2H)
B1	102.2, CH	5.38, d (3.5, 1H)	99.4, CH	5.40, d (3.5, 1H)	99.4, CH	5.38, d (3.5, 1H)
B2	74.4, CH	3.61, m (1H)	71.5, CH	3.60, m (1H)	71.6, CH	3.59, m (1H)
B3	76.2, CH	3.93, m (1H)	73.3, CH	3.95, m (1H)	73.2, CH	3.94, m (1H)
B4	79.9, CH	3.63, m (1H)	77.0, CH	3.64, m (1H)	77.0, CH	3.64, m (1H)
B5	74.0, CH	3.82, m (1H)	71.2, CH	3.82, m (1H)	71.2, CH	3.82, m (1H)
B6	63.4, CH <sub>2</sub>	3.82, m (2H)	60.7, CH <sub>2</sub>	3.84, m (2H)	60.5, CH <sub>2</sub>	3.82, m (2H)
C1	102.3, CH	5.38, d (3.5, 1H)	99.5, CH	5.40, d (3.5, 1H)	99.5, CH	5.38, d (3.5, 1H)
C2	74.3, CH	3.61, m (1H)	71.5, CH	3.60, m (1H)	71.5, CH	3.59, m (1H)
C3	76.1, CH	3.93, m (1H)	73.2, CH	3.95, m (1H)	73.3, CH	3.94, m (1H)
C4	79.9, CH	3.63, m (1H)	77.0, CH	3.64, m (1H)	76.9, CH	3.64, m (1H)
C5	74.1, CH	3.82, m (1H)	71.2, CH	3.82, m (1H)	71.2, CH	3.82, m (1H)
C6	63.5, CH <sub>2</sub>	3.82, m (2H)	60.8, CH <sub>2</sub>	3.84, m (2H)	60.7, CH <sub>2</sub>	3.82, m (2H)
D1	102.4, CH	5.38, d (3.5, 1H)	99.6, CH	5.40, d (3.5, 1H)	99.6, CH	5.38, d (3.5, 1H)
D2	73.9, CH	3.63, m (1H)	71.1, CH	3.63, m (1H)	71.1, CH	3.64, m (1H)
D3	76.0, CH	3.93, m (1H)	73.2, CH	3.94, m (1H)	73.1, CH	3.93, m (1H)
D4	80.7, CH	3.63, m (1H)	77.9, CH	3.63, m (1H)	78.4, CH	3.64, m (1H)
D5	71.7, CH	4.01, d (12.0, 1H)	68.9, CH	4.04, d (12.0, 1H)	69.0, CH	4.01, d (12.0, 1H)
D6a	67.1, CH <sub>2</sub>	4.19, m (1H)	64.3, CH <sub>2</sub>	4.22, m (1H)	63.4, CH <sub>2</sub>	4.19, m (1H)
D6b		4.44, d (12.0, 1H)		4.45, dd (12.0, 3.0, 1H)		4.43, d (12.0, 1H)
E1	103.4, CH	5.25, d (3.5, 1H)	100.5, CH	5.28, d (3.4, 1H)	100.7, CH	5.24, d (3.5, 1H)
E2	74.1, CH	3.57, m (1H)	71.3, CH	3.53, m (1H)	71.3, CH	3.50, m (1H)
E3	75.8, CH	3.61, m (1H)	72.9, CH	3.58, m (1H)	72.9, CH	3.52, m (1H)
E4	67.8, CH	2.43, t (9.0, 1H)	64.9, CH	2.45, t (9.0, 1H)	64.3, CH	2.43, t (9.0, 1H)
E5	72.6, CH	3.73, m (1H)	69.8, CH	3.73, m (1H)	69.7, CH	3.71, m (1H)
E6	20.2, CH <sub>3</sub>	1.27, d (5.6, 3H)	17.4, CH <sub>3</sub>	1.30, d (6.0, 3H)	17.4, CH <sub>3</sub>	1.29, d (5.6, 3H)
F1	57.8, CH	3.49, m (1H)	55.0, CH	3.53, m (1H)	55.0, CH	3.45, m (1H)

F2	72.5, CH	3.77, m (1H)	69.7, CH	3.79, m (1H)	69.7, CH	3.76, m (1H)
F3	73.6, CH	4.09, m (1H)	70.8, CH	4.13, m (1H)	70.8, CH	4.09, m (1H)
F4	79.0, CH	4.19, m (1H)	76.2, CH	4.22, m (1H)	76.3, CH	4.19, m (1H)
F5	139.3, C		136.5, C		136.5, C	
F6a		4.09, m (1H)		4.13, m (1H)		4.09, m (1H)
F6b	64.8, CH <sub>2</sub>	4.19, m (1H)	62.0, CH <sub>2</sub>	4.22, m (1H)	62.0, CH <sub>2</sub>	4.19, m (1H)
F7	129.1, CH	5.94, d (3.2, 1H)	126.3, CH	5.97, d (3.2, 1H)	126.6, CH	5.94, d (3.1, 1H)
G1	100.4, CH	5.34, d (3.4, 1H)	97.6, CH	5.37, d (3.6, 1H)	97.5, CH	5.34, d (3.5, 1H)
G2	74.4, CH	3.61, m (1H)	71.5, CH	3.63, m (1H)	71.7, CH	3.58, m (1H)
G3	76.3, CH	3.91, m (1H)	73.5, CH	3.92, m (1H)	73.5, CH	3.89, m (1H)
G4	73.7, CH	3.61, m (1H)	70.8, CH	3.63, m (1H)	70.8, CH	3.59, m (1H)
G5	74.0, CH	3.91, m (1H)	71.1, CH	3.92, m (1H)	71.1, CH	3.89, m (1H)
G6	66.3, CH <sub>2</sub>	3.86, m (2H)	63.4, CH <sub>2</sub>	3.86, m (2H)	62.5, CH <sub>2</sub>	3.83, m (2H)
H1	102.7, CH	5.29, d (3.4, 1H)	99.9, CH	5.32, d (3.4, 1H)	99.9, CH	5.30, d (3.4, 1H)
H2	74.4, CH	3.57, m (1H)	71.6, CH	3.61, m (1H)	71.5, CH	3.57, m (1H)
H3	75.4, CH	3.61, m (1H)	72.5, CH	3.65, m (1H)	72.6, CH	3.57, m (1H)
H4	67.8, CH	2.43, t (9.0, 1H)	64.9, CH	2.45, t (9.0, 1H)	64.9, CH	2.43, t (9.0, 1H)
H5	72.4, CH	3.74, m (1H)	69.6, CH	3.78, m (1H)	69.6, CH	3.73, m (1H)
H6	20.2, CH <sub>3</sub>	1.31, d (5.6, 3H)	17.3, CH <sub>3</sub>	1.34, d (6.0, 3H)	17.3, CH <sub>3</sub>	1.30, d (5.6, 3H)
I1	58.8, CH	3.49, m (1H)	56.0, CH	3.53, m (1H)	56.0, CH	3.45, m (1H)
I2	75.6, CH	3.67, m (1H)	72.7, CH	3.66, m (1H)	72.7, CH	3.63, m (1H)
I3	75.8, CH	3.77, m (1H)	72.9, CH	3.78, m (1H)	72.9, CH	3.73, m (1H)
I4	73.8, CH	4.01, d (12.0, 1H)	70.9, CH	4.04, d (12.0, 1H)	70.9, CH	4.01, d (12.0, 1H)
I5	141.8, C		138.9, C		138.9, C	
I6a		4.09, m (1H)		4.13, m (1H)		4.09, m (1H)
I6b	64.4, CH <sub>2</sub>	4.19, m (1H)	61.6, CH <sub>2</sub>	4.22, m (1H)	61.6, CH <sub>2</sub>	4.19, m (1H)
I7	126.6, CH	5.86, d (3.3, 1H)	123.8, CH	5.90, d (3.4, 1H)	123.8, CH	5.87, d (3.5, 1H)
1'	180.2, C=O		176.7, C=O		179.8, C=O	
2'	30.0, CH <sub>2</sub>	2.43, m (2H)	35.6, CH <sub>2</sub>	2.45, m (2H)	40.9, CH	2.52, m (1H)
3'	11.1, CH <sub>3</sub>	1.09, d (7.0, 3H)	17.9, CH <sub>2</sub>	1.18, m (1H)	26.4, CH <sub>2</sub>	1.51, m (1H)
4'				1.65, m (1H)		1.63, m (1H)
5'			12.9, CH <sub>3</sub>	0.94, t (7.2, 3H)	10.9, CH <sub>3</sub>	0.88, t (7.0, 3H)
					15.7, CH <sub>3</sub>	1.13, d (7.0, 3H)



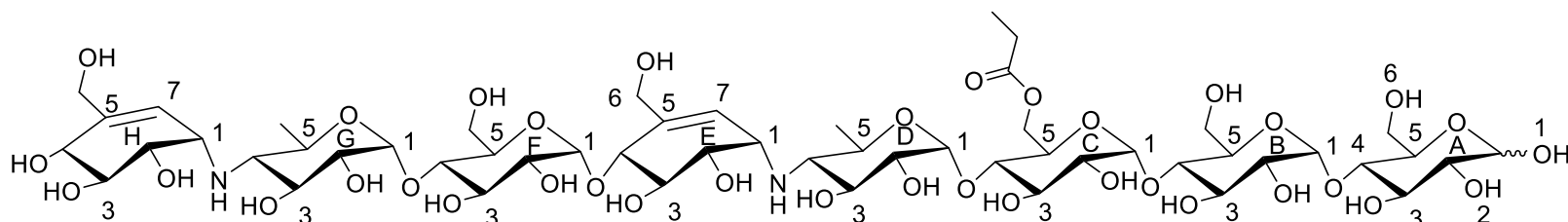
10

**Table S3.**  $^1\text{H}$  (500 MHz) and  $^{13}\text{C}$  (125 MHz) NMR data of **10** ( $\delta$  ppm) in  $\text{D}_2\text{O}$ .

No.	$\delta_{\text{C}}$	$\delta_{\text{H}}$	HMBC ( $^1\text{H} \rightarrow ^{13}\text{C}$ )	COSY ( $^1\text{H} \rightarrow ^1\text{H}$ )	TOCSY ( $^1\text{H} \rightarrow ^1\text{H}$ )
A1 $\alpha$	93.4, CH	5.23, d (3.5, 1H)	A2 $\alpha$ , A5 $\alpha$	A2 $\alpha$	A2 $\alpha$ , A5 $\alpha$
A2 $\alpha$	71.2, CH	3.57, m (1H)		A1 $\alpha$ , A3 $\alpha$	A1 $\alpha$ , A3 $\alpha$
A3 $\alpha$	73.2, CH	4.09, m (1H)		A2 $\alpha$ , A4 $\alpha$	A2 $\alpha$ , A4 $\alpha$
A4 $\alpha$	76.8, CH	3.68, m (1H)	B1	A3 $\alpha$ , A5 $\alpha$	A3 $\alpha$ , A5 $\alpha$
A5 $\alpha$	70.0, CH	3.97, m (1H)		A4 $\alpha$ , A6 $\alpha$	A4 $\alpha$ , A6 $\alpha$
A6 $\alpha$	60.5, CH <sub>2</sub>	3.85, m (2H)		A5 $\alpha$	A5 $\alpha$
A1 $\beta$	95.8, CH	4.65, d (8.0, 1H)	A2 $\beta$ , A3 $\beta$ , A5 $\beta$	A2 $\beta$	A2 $\beta$ , A3 $\beta$ , A5 $\beta$
A2 $\beta$	74.0, CH	3.27, t (9.0, 1H)		A1 $\beta$ , A3 $\beta$	A1 $\beta$ , A3 $\beta$
A3 $\beta$	76.2, CH	3.76, m (1H)		A2 $\beta$ , A4 $\beta$	A2 $\beta$ , A4 $\beta$
A4 $\beta$	76.9, CH	3.65, m (1H)	B1	A3 $\beta$	A3 $\beta$ , A5 $\beta$
A5 $\beta$	74.6, CH	3.60, m (1H)		A4 $\beta$ , A6 $\beta$	A4 $\beta$ , A6 $\beta$
A6 $\beta$	60.5, CH <sub>2</sub>	3.90, m (2H)		A5 $\beta$	A5 $\beta$
B1	99.5, CH	5.41, d (3.5, 1H)	B2, B3	B2	B2, B3, B5
B2	71.5, CH	3.62, m (1H)		B1, B3	B1, B3
B3	73.4, CH	3.95, m (1H)		B2, B4	B2, B4
B4	77.0, CH	3.67, m (1H)	C1	B3, B5	B3, B5
B5	71.2, CH	3.85, m (1H)		B4	B4
B6	60.7, CH <sub>2</sub>	3.85, m (2H)		overlapped	overlapped
C1	99.4, CH	5.41, d (3.5, 1H)	C2, C3	C2	C2, C3
C2	71.0, CH	3.66, m (1H)		C1, C3	C1, C3
C3	73.3, CH	3.83, m (1H)		C2, C4	C2, C4
C4	77.0, CH	3.68, m (1H)	D1	C3, C5	C3, C5
C5	69.4, CH	4.04, d (12.0, 1H)		C4, C6a, C6b	C4, C6a, C6b
C6a	63.2, CH <sub>2</sub>	4.15, m (1H)		C5, C6b	C5, C6b
C6b		4.23, d (12.0, 1H)		C5, C6a	C5, C6a
D1	99.9, CH	5.33, d (3.5, 1H)	D2, D5	D2	D2, D3, D5
D2	71.2, CH	3.56, m (1H)		D1, D3	D1, D3
D3	72.7, CH	3.59, m (1H)		D2, D4	D2, D4
D4	64.2, CH	2.48, t (9.0, 1H)	D1, D3, D5, E1	D3, D5	D3, D5, D6



D5	69.7, CH	3.76, m (1H)		D4, D6	D4, D6
D6	17.4, CH <sub>3</sub>	1.34, d (5.7, 3H)	D4, D5	D5	D3, D4, D5
E1	55.0, CH	3.54, m (1H)	E6	E2, E7	E2, E7
E2	70.7, CH	3.82, m (1H)		E1, E3	E1, E3
E3	70.8, CH	4.13, m (1H)		E2, E4	E2, E4
E4	76.1, CH	4.22, m (1H)	F1	E3	E3
E5	136.5, C				
E6a		4.13, m (1H)		E6b	E6b
E6b	62.0, CH <sub>2</sub>	4.22, m (1H)		E6a	E6a
E7	126.3, CH	5.98, d (3.2, 1H)	E1, E2, E6	E1	E1, E2, E6b
F1	97.5, CH	5.38, d (3.4, 1H)		F2	F2
F2	72.9, CH	3.58, m (1H)		F1, F3	F1, F3
F3	73.5, CH	3.93, m (1H)		F2, F4	F2, F4
F4	70.8, CH	3.60, m (1H)	G1	F3, F5	F3, F5
F5	71.1, CH	3.95, m (1H)		F4, F6	F4, F6
F6	60.4, CH <sub>2</sub>	3.81, m (2H)		F5	F5
G1	99.6, CH	5.33, d (3.4, 1H)	G2, G3	G2	G2, G3, G5
G2	71.3, CH	3.56, m (1H)		G1	G1, G3
G3	72.2, CH	3.59, m (1H)		G4	G4, G3
G4	64.9, CH	2.48, t (9.0, 1H)	H1	G3, G5	G3, G5, G6
G5	69.6, CH	3.86, m (1H)		G4, G6	G4, G6
G6	17.3, CH <sub>3</sub>	1.34, d (5.7, 3H)	G4, G5	G5	G3, G4, G5
H1	56.0, CH	3.52, m (1H)	H6	H2, H7	H2, H7
H2	72.6, CH	3.72, m (1H)		H1, H3	H1, H3
H3	72.6, CH	3.75, m (1H)		H2, H4	H2, H4
H4	70.9, CH	4.07, d (12.0, 1H)		H3	H3
H5	139.0, C				
H6a					
H6b	61.6, CH <sub>2</sub>	4.12, m (2H)			
H7	123.7, CH	5.90, d (3.5, 1H)	H1, H2, H6	H1	H1, H2, H6a, H6b

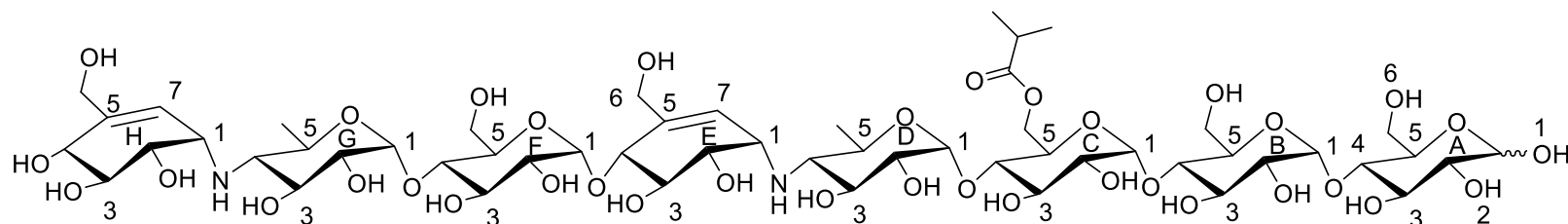


11

**Table S4.**  $^1\text{H}$  (500 MHz) and  $^{13}\text{C}$  (125 MHz) NMR data of **11** ( $\delta$  ppm) in  $\text{D}_2\text{O}$ .

No.	$\delta_{\text{C}}$	$\delta_{\text{H}}$	HMBC ( $^1\text{H} \rightarrow ^{13}\text{C}$ )	COSY ( $^1\text{H} \rightarrow ^1\text{H}$ )	TOCSY ( $^1\text{H} \rightarrow ^1\text{H}$ )
A1 $\alpha$	95.3, CH	5.23, d (3.5, 1H)	A2 $\alpha$ , A5 $\alpha$	A2 $\alpha$	A2 $\alpha$ , A5 $\alpha$
A2 $\alpha$	74.8, CH	3.58, m (1H)		A1 $\alpha$ , A3 $\alpha$	A1 $\alpha$ , A3 $\alpha$
A3 $\alpha$	76.7, CH	3.97, m (1H)		A2 $\alpha$ , A4 $\alpha$	A2 $\alpha$ , A4 $\alpha$
A4 $\alpha$	80.2, CH	3.65, m (1H)	B1	A3 $\alpha$ , A5 $\alpha$	A3 $\alpha$ , A5 $\alpha$
A5 $\alpha$	73.3, CH	3.87, m (1H)		A4 $\alpha$	A4 $\alpha$ , A6 $\alpha$
A6 $\alpha$	63.8, CH <sub>2</sub>	3.87, m (2H)		overlapped	A5 $\alpha$
A1 $\beta$	99.2, CH	4.66, d (8.0, 1H)	A2 $\beta$ , A5 $\beta$	A2 $\beta$	A2 $\beta$ , A3 $\beta$ , A5 $\beta$
A2 $\beta$	77.4, CH	3.28, t (9.0, 1H)		A1 $\beta$ , A3 $\beta$	A1 $\beta$ , A3 $\beta$
A3 $\beta$	79.7, CH	3.77, m (1H)		A2 $\beta$ , A4 $\beta$	A2 $\beta$ , A4 $\beta$
A4 $\beta$	80.2, CH	3.66, m (1H)	B1	A3 $\beta$ , A5 $\beta$	A3 $\beta$ , A5 $\beta$
A5 $\beta$	77.9, CH	3.62, m (1H)		A4 $\beta$ , A6 $\beta$	A4 $\beta$ , A6 $\beta$
A6 $\beta$	64.1, CH <sub>2</sub>	3.91, m (2H)		A5 $\beta$	A5 $\beta$
B1	102.8, CH	5.41, d (3.5, 1H)	B3, B4, B5	B2	B2, B3, B5
B2	74.9, CH	3.64, m (1H)		B1, B3	B1, B3
B3	76.7, CH	3.96, m (1H)		B2, B4	B2, B4
B4	80.4, CH	3.67, m (1H)	C1	B3, B5	B3, B5
B5	74.6, CH	3.85, m (1H)		B4	B4
B6	63.9, CH <sub>2</sub>	3.85, m (2H)		overlapped	overlapped
C1	103.0, CH	5.41, d (3.5, 1H)	C3, C5	C2	C2, C3
C2	74.5, CH	3.67, m (1H)		C1, C3	C1, C3
C3	76.6, CH	3.95, m (1H)		C2, C4	C2, C4
C4	81.3, CH	3.67, m (1H)	D1	C3, C5	C3, C5
C5	72.3, CH	4.05, d (12.0, 1H)		C4, C6a, C6b	C4, C6a, C6b
C6a	63.8, CH <sub>2</sub>	4.22, m (1H)		C5, C6b	C5, C6b
C6b		4.44, d (12.0, 1H)		C5, C6a	C5, C6a
D1	104.0, CH	5.29, d (3.5, 1H)	D2, D5	D2	D2, D3, D5
D2	74.6, CH	3.56, m (1H)		D1, D3	D1, D3
D3	76.3, CH	3.59, m (1H)		D2, D4	D2, D4
D4	67.7, CH	2.48, t (9.0, 1H)	D3, D5, D6, E1	D3, D5	D2, D3, D5, D6

D5	73.2, CH	3.73, m (1H)		D4, D6	D4, D6
D6	20.7, CH <sub>3</sub>	1.30, d (5.7, 3H)	D4, D5	D5	D3, D4, D5
E1	58.4, CH	3.53, m (1H)	E6	E2, E7	E2, E7
E2	73.0, CH	3.80, m (1H)		E1, E3	E1, E3
E3	74.1, CH	4.13, m (1H)		E2, E4	E2, E4
E4	79.6, CH	4.22, m (1H)	F1	E3	E3
E5	139.9, C				
E6a		4.13, m (1H)		E6b	E6b
E6b	65.4, CH <sub>2</sub>	4.22, m (1H)		E6a	E6a
E7	129.7, CH	5.98, d (3.2, 1H)	E1, E2, E4, E6	E1	E1, E2, E6b
F1	101.0, CH	5.38, d (3.4, 1H)		F2	F2
F2	74.9, CH	3.62, m (1H)		F1, F3	F1, F3
F3	76.9, CH	3.93, m (1H)		F2, F4	F2, F4
F4	74.2, CH	3.64, m (1H)	G1	F3	F3, F5
F5	74.5, CH	3.93, m (1H)		F4, F6	F4, F6
F6	66.9, CH <sub>2</sub>	3.81, m (2H)		F5	F5
G1	103.3, CH	5.32, d (3.4, 1H)	G2, G3	G2	G2, G4, G5
G2	74.7, CH	3.58, m (1H)		G1, G3	G1, G3
G3	75.9, CH	3.62, m (1H)		G2, G4	G2, G4
G4	68.3, CH	2.48, t (9.0, 1H)	H1	G3, G5	G2, G3, G5, G6
G5	73.0, CH	3.76, m (1H)		G4, G6	G4, G6
G6	20.8, CH <sub>3</sub>	1.34, d (5.7, 3H)	G4, G5	G5	G3, G4, G5
H1	59.4, CH	3.54, m (1H)	H6	H2, H7	H2, H7
H2	76.1, CH	3.68, m (1H)		H1, H3	H1, H3
H3	76.3, CH	3.77, m (1H)		H2, H4	H2, H4
H4	74.3, CH	4.04, d (12.0, 1H)		H3	H3
H5	142.3, C				
H6a		4.12, m (1H)		H6b	
H6b	65.0 CH <sub>2</sub>	4.22, m (1H)		H6a	
H7	127.2, CH	5.90, d (3.5, 1H)	H1, H2, H6	H1	H1, H2, H6a, H6b
1'	180.8, C=O				
2'	30.6, CH <sub>2</sub>	2.48, m (2H)	1', 3'	3'	3'
3'	11.7, CH <sub>3</sub>	1.13, d (7.2, 3H)	2', 3'	2'	2'

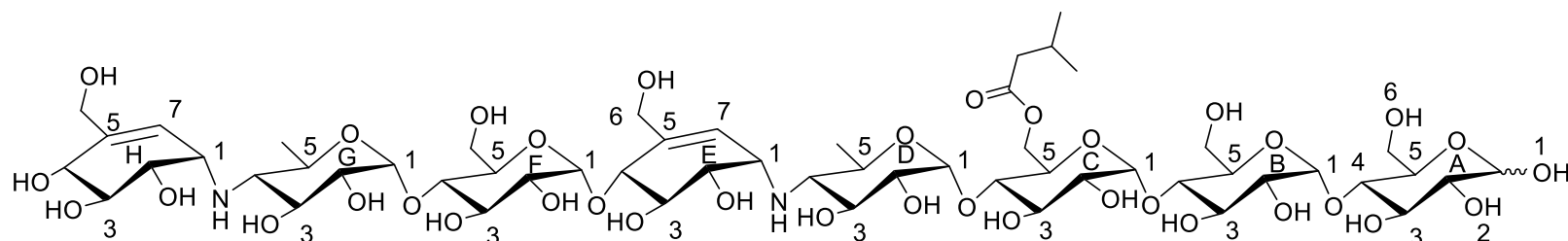


12

**Table S5.**  $^1\text{H}$  (500 MHz) and  $^{13}\text{C}$  (125 MHz) NMR data of **12** ( $\delta$  ppm) in  $\text{D}_2\text{O}$ .

No.	$\delta_{\text{C}}$	$\delta_{\text{H}}$	HMBC ( $^1\text{H} \rightarrow ^{13}\text{C}$ )	COSY ( $^1\text{H} \rightarrow ^1\text{H}$ )	TOCSY ( $^1\text{H} \rightarrow ^1\text{H}$ )
A1 $\alpha$	91.9, CH	5.24, d (3.4, 1H)	A2 $\alpha$ , A5 $\alpha$	A2 $\alpha$	A2 $\alpha$ , A5 $\alpha$
A2 $\alpha$	71.3, CH	3.52, m (1H)		A1 $\alpha$ , A3 $\alpha$	A1 $\alpha$ , A3 $\alpha$
A3 $\alpha$	73.3, CH	4.16, m (1H)		A2 $\alpha$ , A4 $\alpha$	A2 $\alpha$ , A4 $\alpha$
A4 $\alpha$	76.8, CH	3.69, m (1H)	B1	A3 $\alpha$ , A5 $\alpha$	A3 $\alpha$ , A5 $\alpha$
A5 $\alpha$	70.0, CH	3.90, m (1H)		A4 $\alpha$ , A6 $\alpha$ ,	A4 $\alpha$ , A6 $\alpha$
A6 $\alpha$	60.5, CH <sub>2</sub>	3.74, m (2H)		A5 $\alpha$	A5 $\alpha$
A1 $\beta$	95.8, CH	4.66, d (8.0, 1H)	A2 $\beta$ , A5 $\beta$	A2 $\beta$	A2 $\beta$ , A3 $\beta$ , A5 $\beta$
A2 $\beta$	74.0, CH	3.29, m (1H)		A1 $\beta$ , A3 $\beta$	A1 $\beta$ , A3 $\beta$
A3 $\beta$	76.3, CH	3.78, m (1H)		A2 $\beta$ , A4 $\beta$	A2 $\beta$ , A4 $\beta$
A4 $\beta$	76.8, CH	3.67, m (1H)	B1	A3 $\beta$ , A5 $\beta$	A3 $\beta$ , A5 $\beta$
A5 $\beta$	74.6, CH	3.62, m (1H)		A4 $\beta$ , A6 $\beta$	A4 $\beta$ , A6 $\beta$
A6 $\beta$	60.6, CH <sub>2</sub>	3.92, m (2H)		A5 $\beta$	A5 $\beta$
B1	99.6, CH	5.38, d (3.5, 1H)	B3, B5	B2	B3, B4
B2	71.6, CH	3.58, m (1H)		B1, B3	B1, B3
B3	73.4, CH	3.94, m (1H)		B2, B4	B2, B4
B4	77.1, CH	3.63, m (1H)	C1	B3, B5	B3, B5
B5	71.2, CH	3.80, m (1H)		B4	B4
B6	60.7, CH <sub>2</sub>	3.80, m (2H)		overlapped	overlapped
C1	99.4, CH	5.38, d (3.5, 1H)	C3, C5	C2	C2, C3
C2	71.1, CH	3.62, m (1H)		C1, C3	C1, C3
C3	73.2, CH	3.92, m (1H)		C2, C4	C2, C4
C4	78.2, CH	3.62, m (1H)	D1	C3, C5	C3, C5
C5	69.0, CH	4.03, d (12.0, 1H)		C4, C6a, C6b	C4, C6a, C6b
C6a	63.5, CH <sub>2</sub>	4.20, m (1H)		C5, C6b	C5, C6b
C6b		4.45, d (12.0, 1H)		C5, C6a	C5, C6a
D1	100.7, CH	5.28, d (3.5, 1H)	D2, D5	D2	D2, D3, D4
D2	71.2, CH	3.54, m (1H)		D1	D1, D3
D3	73.0, CH	3.56, m (1H)		D4	D2, D4
D4	64.3, CH	2.47, t (8.8, 1H)	D3, D5, E1	D3, D5	D1, D2, D3, D5

D5	69.8, CH	3.71, m (1H)		D4, D6	D4, D6
D6	17.4, CH <sub>3</sub>	1.31, d (5.6, 3H)	D4, D5	D5	D3, D4, D5
E1	55.0, CH	3.53, m (1H)	E6	E2, E7	E2, E7
E2	70.8, CH	3.80, m (1H)		E1, E3	E1, E3
E3	70.9, CH	4.14, m (1H)		E2, E4	E2, E4
E4	76.2, CH	4.23, m (1H)	F1	E3	E3
E5	136.5, C				
E6a		4.14, m (1H)		E6b	E6b
E6b	62.0, CH <sub>2</sub>	4.23, m (1H)		E6a	E6a
E7	126.3, CH	5.98, d (3.1, 1H)	E2, E6	E1	E1, E2, E6b
F1	97.8, CH	5.42, d (3.5, 1H)		F2	F2
F2	73.2, CH	3.68, m (1H)		F1, F3	F1, F3
F3	73.5, CH	3.94, m (1H)		F2, F4	F2, F4
F4	70.9, CH	3.66, m (1H)	G1	F3, F5	F3, F5
F5	71.2, CH	3.96, m (1H)		F4, F6	F4, F6
F6	60.4, CH <sub>2</sub>	3.82, m (2H)		F5	F5
G1	99.9, CH	5.33, d (3.4, 1H)	G2, G3	G2	G2, G4
G2	71.2, CH	3.61, m (1H)		G1	G1, G3
G3	72.6, CH	3.61, m (1H)		G4	G2, G4
G4	64.9, CH	2.47, t (8.8, 1H)	G3, G5, H1	G3, G5	G2, G3, G5
G5	69.6, CH	3.77, m (1H)		G4, G6	G4, G6
G6	17.3, CH <sub>3</sub>	1.35, d (5.6, 3H)	G4, G5	G5	G3, G4, G5
H1	56.0, CH	3.54, m (1H)	H6	H2, H7	H2, H7
H2	72.7, CH	3.69, m (1H)		H1, H3	H1, H3
H3	72.9, CH	3.77, m (1H)		H2, H4	H2, H4
H4	71.0, CH	4.06, d (12.0, 1H)		H3	H3
H5	139.0, C				
H6a		4.13, m (1H)		H6b	
H6b	61.6, CH <sub>2</sub>	4.25, m (1H)		H6a	
H7	123.8, CH	5.91, d (3.4, 1H)	H1, H2, H6	H1	H1, H2, H6a, H6b
1'	180.1, C=O				
2'	33.9, CH	2.72, m (2H)	1', 3', 4'	3'	3', 4'
3'	18.3, CH <sub>3</sub>	1.19, d (3.0, 3H)	1, 2', 4'	2'	2'
4'	18.2, CH <sub>3</sub>	1.19, d (3.0, 3H)	1, 2', 3'	Overlapped	2'

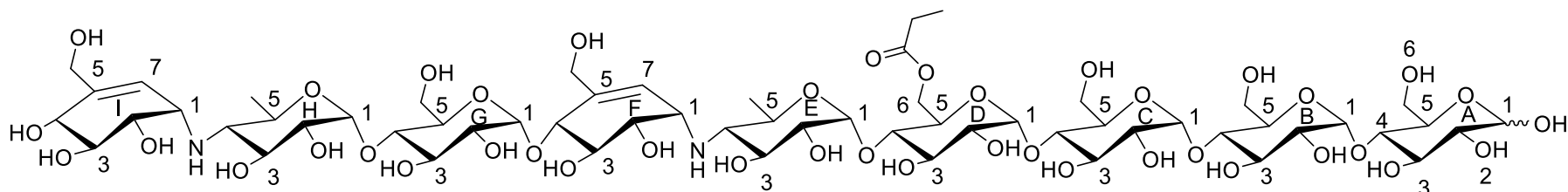


**13**

**Table S6.**  $^1\text{H}$  (500 MHz) and  $^{13}\text{C}$  (125 MHz) NMR data of **13** ( $\delta$ ppm) in  $\text{D}_2\text{O}$ .

No.	$\delta_{\text{C}}$	$\delta_{\text{H}}$	HMBC ( $^1\text{H} \rightarrow ^{13}\text{C}$ )	COSY ( $^1\text{H} \rightarrow ^1\text{H}$ )	TOCSY ( $^1\text{H} \rightarrow ^1\text{H}$ )
A1 $\alpha$	92.0, CH	5.24, d (3.5, 1H)	A2 $\alpha$ , A5 $\alpha$	A2 $\alpha$	A2 $\alpha$ , A5 $\alpha$
A2 $\alpha$	71.4, CH	3.51, m (1H)		A1 $\alpha$ , A3 $\alpha$	A1 $\alpha$ , A3 $\alpha$
A3 $\alpha$	73.4, CH	3.92, m (1H)		A2 $\alpha$ , A4 $\alpha$	A2 $\alpha$ , A4 $\alpha$
A4 $\alpha$	76.9, CH	3.69, m (1H)	B1	A3 $\alpha$ , A5 $\alpha$	A3 $\alpha$ , A5 $\alpha$
A5 $\alpha$	70.0, CH	3.82, m (1H)		A4 $\alpha$ , A6 $\alpha$ ,	A4 $\alpha$ , A6 $\alpha$
A6 $\alpha$	60.5, CH <sub>2</sub>	3.79, m (2H)		A5 $\alpha$	A5 $\alpha$
A1 $\beta$	95.8, CH	4.66, d (7.8, 1H)	A2 $\beta$ , A5 $\beta$	A2 $\beta$	A2 $\beta$ , A4 $\beta$ , A5 $\beta$
A2 $\beta$	74.0, CH	3.28, m (1H)		A1 $\beta$ , A3 $\beta$	A1 $\beta$ , A3 $\beta$
A3 $\beta$	76.2, CH	3.77, m (1H)		A2 $\beta$ , A4 $\beta$	A2 $\beta$ , A4 $\beta$
A4 $\beta$	76.8, CH	3.66, m (1H)	B1	A3 $\beta$ , A5 $\beta$	A3 $\beta$ , A5 $\beta$
A5 $\beta$	74.6, CH	3.60, m (1H)		A4 $\beta$ , A6 $\beta$	A4 $\beta$ , A6 $\beta$
A6 $\beta$	60.5, CH <sub>2</sub>	3.90, m (2H)		A5 $\beta$	A5 $\beta$
B1	99.4, CH	5.41, d (3.5, 1H)	B2, B3, B5	B2	B2, B3, B5
B2	71.6, CH	3.62, m (1H)		B1, B3	B1, B3
B3	73.2, CH	3.96, m (1H)		B2, B4	B2, B4
B4	77.1, CH	3.64, m (1H)	C1	B3, B5	B3, B5
B5	71.3, CH	3.85, m (1H)		B4	B4
B6	60.7, CH <sub>2</sub>	3.85, m (2H)		overlapped	overlapped
C1	99.6, CH	5.41, d (3.5, 1H)	C2, C3, C5	C2	C2, C3
C2	71.2, CH	3.62, m (1H)		C1, C3	C1, C3
C3	73.2, CH	3.96, m (1H)		C2, C4	C2, C4
C4	78.0, CH	3.64, m (1H)	D1	C3, C5	C3, C5
C5	69.0, CH	4.05, d (12.0, 1H)		C4, C6a, C6b	C4, C6a, C6b
C6a	63.4, CH <sub>2</sub>	4.23, m (1H)		C5, C6b	C5, C6b
C6b		4.49, d (12.0, 1H)		C5, C6a	C5, C6a
D1	100.6, CH	5.29, d (3.5, 1H)	D2, D5	D2	D2, D4, D5
D2	71.3, CH	3.58, m (1H)		D1	D1
D3	73.0, CH	3.60, m (1H)		D4	D4

D4	64.3, CH	2.47, t (9.0, 1H)	D3, D5, D6, E1	D3, D5	D1, D3, D5, D6
D5	69.8, CH	3.75, m (1H)		D4, D6	D4, D6
D6	17.4, CH <sub>3</sub>	1.32, d (5.6, 3H)	D4, D5	D5	D3, D4, D5
E1	55.0, CH	3.48, m (1H)	E6	E2, E7	E2, E7
E2	69.7, CH	3.79, m (1H)		E1, E3	E1, E3
E3	70.8, CH	4.14, m (1H)		E2, E4	E2, E4
E4	76.4, CH	4.23, m (1H)	F1	E3	E3
E5	136.5, C				
E6a		4.14, m (1H)		E6b	E6b
E6b	62.0, CH <sub>2</sub>	4.23, m (1H)		E6a	E6a
E7	126.4, CH	5.99, d (3.2, 1H)	E2, E6	E1	E1, E2, E6b
F1	97.6, CH	5.38, d (3.5, 1H)		F2	F2, F5
F2	72.7, CH	3.61, m (1H)		F1, F3	F1, F3
F3	73.5, CH	3.93, m (1H)		F2	F2, F4
F4	70.9, CH	3.61, m (1H)	G1	F3	F3, F5
F5	71.2, CH	3.93, m (1H)		F4, F6	F4, F6
F6	60.7, CH <sub>2</sub>	3.82, m (2H)		F5	F5
G1	100.0, CH	5.33, d (3.4, 1H)	G2, G3	G2	G2, G4
G2	71.5, CH	3.58, m (1H)		G1	G1, G3
G3	72.6, CH	3.60, m (1H)		G4	G2, G4
G4	65.0, CH	2.47, t (9.0, 1H)	G3, G5, H1	G3, G5	G1, G2, G5, G6
G5	69.6, CH	3.75, m (1H)		G4, G6	G4, G6
G6	17.4, CH <sub>3</sub>	1.35, d (5.6, 3H)	G4, G5	G5	G3, G4, G5
H1	56.0, CH	3.48, m (1H)	H6	H2, H7	H2, H7
H2	72.8, CH	3.67, m (1H)		H1, H3	H1, H3
H3	73.0, CH	3.76, m (1H)		H2, H4	H2, H4
H4	71.0, CH	4.05, d (12.0, 1H)		H3	H3
H5	139.0, C				
H6a		4.12, m (1H)		H6b	
H6b	61.6, CH <sub>2</sub>	4.23, m (1H)		H6a	
H7	123.8, CH	5.91, d (3.4, 1H)	H1, H2, H6	H1	H1, H2, H6a, H6b
1'	176.2, C=O				
2'	42.9, CH <sub>2</sub>	2.36, m (2H)	1', 3', 4'	3'	3', 4'
3'	25.5, CH	2.08, m (1H)	1, 2', 4'	2', 4'	2', 4'
4'	21.7, CH <sub>3</sub>	0.96, d (6.6, 3H)	2', 3'	3'	3'
5'	21.7, CH <sub>3</sub>	0.96, d (6.6, 3H)		overlapped	overlapped



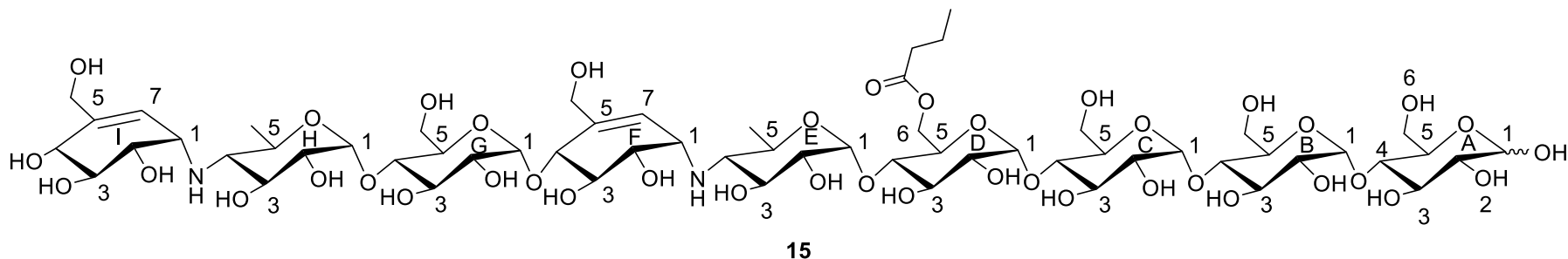
14

**Table S7.**  $^1\text{H}$  (500 MHz) and  $^{13}\text{C}$  (125 MHz) NMR data of **14** ( $\delta$  ppm) in  $\text{D}_2\text{O}$ .

No.	$\delta_{\text{C}}$	$\delta_{\text{H}}$	HMBC ( $^1\text{H} \rightarrow ^{13}\text{C}$ )	COSY ( $^1\text{H} \rightarrow ^1\text{H}$ )	TOCSY ( $^1\text{H} \rightarrow ^1\text{H}$ )
A1 $\alpha$	94.7, CH	5.20, d (3.5, 1H)	A2 $\alpha$ , A5 $\alpha$	A2 $\alpha$	A2 $\alpha$ , A5 $\alpha$
A2 $\alpha$	74.2, CH	3.53, m (1H)		A1 $\alpha$ , A3 $\alpha$	A1 $\alpha$ , A3 $\alpha$
A3 $\alpha$	76.1, CH	3.93, m (1H)		A2 $\alpha$ , A4 $\alpha$	A2 $\alpha$ , A4 $\alpha$
A4 $\alpha$	79.6, CH	3.61, m (1H)	B1	A3 $\alpha$ , A5 $\alpha$	A3 $\alpha$ , A5 $\alpha$
A5 $\alpha$	72.8, CH	3.85, m (1H)		A4 $\alpha$ , A6 $\alpha$ ,	A4 $\alpha$ , A6 $\alpha$ ,
A6 $\alpha$	63.2, CH <sub>2</sub>	3.78, m (2H)		A5 $\alpha$	A5 $\alpha$
A1 $\beta$	98.6, CH	4.62, d (8.0, 1H)	A5 $\beta$	A2 $\beta$	A2 $\beta$ , A3 $\beta$ , A5 $\beta$
A2 $\beta$	76.8, CH	3.24, t (9.0, 1H)		A1 $\beta$ , A3 $\beta$	A1 $\beta$ , A3 $\beta$
A3 $\beta$	79.1, CH	3.74, m (1H)		A2 $\beta$ , A4 $\beta$	A2 $\beta$ , A4 $\beta$
A4 $\beta$	79.7, CH	3.63, m (1H)	B1	A3 $\beta$	A3 $\beta$ ,
A5 $\beta$	77.4, CH	3.61, m (1H)		A6 $\beta$	A6 $\beta$
A6 $\beta$	63.3, CH <sub>2</sub>	3.88, m (2H)		A5 $\beta$	A5 $\beta$
B1	102.2, CH	5.38, d (3.5, 1H)	B2, B3, B5	B2	B2, B3
B2	74.4, CH	3.61, m (1H)		B1, B3	B1, B3
B3	76.2, CH	3.93, m (1H)		B2, B4	B2, B4
B4	79.9, CH	3.63, m (1H)	C1	B3, B5	B3, B5
B5	74.0, CH	3.82, m (1H)		B4	B4
B6	63.4, CH <sub>2</sub>	3.82, m (2H)		overlapped	overlapped
C1	102.3, CH	5.38, d (3.5, 1H)	C2, C3, C5	C2	C2, C3
C2	74.3, CH	3.61, m (1H)		C1, C3	C1, C3
C3	76.1, CH	3.93, m (1H)		C2, C4	C2, C4
C4	79.9, CH	3.63, m (1H)	D1	C3, C5	C3, C5
C5	74.1, CH	3.82, m (1H)		C4	C4
C6	63.5, CH <sub>2</sub>	3.82, m (2H)		overlapped	overlapped
D1	102.4, CH	5.38, d (3.5, 1H)	D2, D3, D5	D2	D2, D3
D2	73.9, CH	3.63, m (1H)		D1, D3	D1, D3
D3	76.0, CH	3.93, m (1H)		D2	D2
D4	80.7, CH	3.63, m (1H)	E1	D3, D5	D3, D5
D5	71.7, CH	4.01, d (12.0, 1H)		D4, D6a, D6b	D4, D6a, D6b



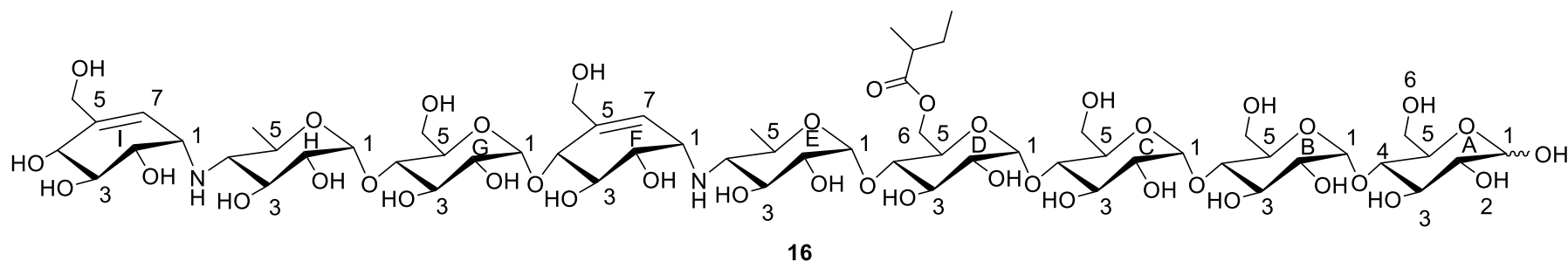
D6a		4.19, m (1H)		D5, D6b	D5, D6b
D6b	67.1, CH <sub>2</sub>	4.44, d (12.0, 1H)		D5, D6a	D5, D6a
E1	103.4, CH	5.25, d (3.5, 1H)	E3, E5	E2	E2, E4
E2	74.1, CH	3.57, m (1H)		E1, E3	E1, E3, E4, E6
E3	75.8, CH	3.61, m (1H)		E2, E4	E2, E4
E4	67.8, CH	2.43, t (9.0, 1H)	E2, E5, E6, F1	E3, E5	E3, E5, E6
E5	72.6, CH	3.73, m (1H)		E4, E6	E4, E6
E6	20.2, CH <sub>3</sub>	1.27, d (5.6, 3H)	E4, E5	E5	E4, E5
F1	57.8, CH	3.49, m (1H)	F2, F6	F2, F7	F2, F3, F7
F2	72.5, CH	3.77, m (1H)		F1, F3	F1, F3
F3	73.6, CH	4.09, m (1H)		F2, F4	F2, F4
F4	79.0, CH	4.19, m (1H)	G1	F3	F3
F5	139.3, C				
F6a		4.09, m (1H)		F6b	F6b
F6b	64.8, CH <sub>2</sub>	4.19, m (1H)		F6a	F6a
F7	129.1, CH	5.94, d (3.2, 1H)	F2, F6	F1	F1, F2, F4
G1	100.4, CH	5.34, d (3.4, 1H)	G3, G5	G2	G2, G5
G2	74.4, CH	3.61, m (1H)		G1, G3	G1, G3
G3	76.3, CH	3.91, m (1H)		G2	G2
G4	73.7, CH	3.61, m (1H)	H1	G3	G3
G5	74.0, CH	3.91, m (1H)		G4, G6	G4, G6
G6	66.3, CH <sub>2</sub>	3.86, m (2H)		G5	G5
H1	102.7, CH	5.29, d (3.4, 1H)	H3, H5	H2	H2
H2	74.4, CH	3.57, m (1H)		H1, H3	H1, H3, H4, H6
H3	75.4, CH	3.61, m (1H)		H2, H4	H2, H4
H4	67.8, CH	2.43, t (9.0, 1H)	H1, H2, H5, H6	H3, H5	H3, H5, H6
H5	72.4, CH	3.74, m (1H)		H4, H6	H4, H6
H6	20.2, CH <sub>3</sub>	1.31, d (5.6, 3H)	H4, H5	H5	H4, H5
I1	58.8, CH	3.49, m (1H)	I2, I6	I2, I7	I2, I3, I7
I2	75.6, CH	3.67, m (1H)		I1, I3	I1, I3
I3	75.8, CH	3.77, m (1H)		I2, I4	I2, I4
I4	73.8, CH	4.01, d (12.0, 1H)		I3	I3
I5	141.8, C				
I6a		4.09, m (1H)		I6b	I6b
I6b	64.4, CH <sub>2</sub>	4.19, m (1H)		I6a	I6a
I7	126.6, CH	5.86, d (3.3, 1H)	I1, I2, I6	I1	I1
1'	180.2, C=O				
2'	30.0, CH <sub>2</sub>	2.43, m (2H)	1', 3'	3'	3'
3'	11.1, CH <sub>3</sub>	1.09, d (7.0, 3H)	1', 2'	2'	2'



**Table S8.**  $^1\text{H}$  (500 MHz) and  $^{13}\text{C}$  (125 MHz) NMR data of **15** ( $\delta$ ppm) in  $\text{D}_2\text{O}$ .

No.	$\delta_{\text{C}}$	$\delta_{\text{H}}$	HMBC ( $^1\text{H} \rightarrow ^{13}\text{C}$ )	COSY ( $^1\text{H} \rightarrow ^1\text{H}$ )	TOCSY ( $^1\text{H} \rightarrow ^1\text{H}$ )
A1 $\alpha$	91.9, CH	5.22, d (3.5, 1H)	A2 $\alpha$ , A5 $\alpha$	A2 $\alpha$	A2 $\alpha$ , A5 $\alpha$
A2 $\alpha$	71.4, CH	3.52, m (1H)		A1 $\alpha$ , A3 $\alpha$	A1 $\alpha$ , A3 $\alpha$
A3 $\alpha$	73.3, CH	3.93, m (1H)		A2 $\alpha$ , A4 $\alpha$	A2 $\alpha$ , A4 $\alpha$
A4 $\alpha$	76.8, CH	3.62, m (1H)	B1	A3 $\alpha$ , A5 $\alpha$	A3 $\alpha$ , A5 $\alpha$
A5 $\alpha$	69.9, CH	3.85, m (1H)		A4 $\alpha$ , A6 $\alpha$ ,	A4 $\alpha$ , A6 $\alpha$ ,
A6 $\alpha$	60.4, CH <sub>2</sub>	3.79, m (2H)		A5 $\alpha$	A5 $\alpha$
A1 $\beta$	95.8, CH	4.65, d (8.0, 1H)	A2 $\beta$ , A5 $\beta$	A2 $\beta$	A2 $\beta$ , A3 $\beta$ , A5 $\beta$
A2 $\beta$	74.0, CH	3.27, m (1H)		A1 $\beta$ , A3 $\beta$	A1 $\beta$ , A3 $\beta$
A3 $\beta$	76.3, CH	3.77, m (1H)		A2 $\beta$ , A4 $\beta$	A2 $\beta$ , A4 $\beta$
A4 $\beta$	76.8, CH	3.65, m (1H)	B1	A3 $\beta$ , A5 $\beta$	A3 $\beta$ , A5 $\beta$
A5 $\beta$	74.5, CH	3.60, m (1H)		A4 $\beta$ , A6 $\beta$	A4 $\beta$ , A6 $\beta$
A6 $\beta$	60.5, CH <sub>2</sub>	3.89, m (2H)		A5 $\beta$	A5 $\beta$
B1	99.4, CH	5.40, d (3.5, 1H)	B2, B3, B5	B2	B2, B3, B5
B2	71.5, CH	3.60, m (1H)		B1, B3	B1, B3
B3	73.3, CH	3.95, m (1H)		B2, B4	B2, B4
B4	77.0, CH	3.64, m (1H)	C1	B3, B5	B3, B5
B5	71.2, CH	3.82, m (1H)		B4	B4
B6	60.7, CH <sub>2</sub>	3.84, m (2H)		overlapped	overlapped
C1	99.5, CH	5.40, d (3.5, 1H)	C2, C3, C5	C2	C2, C3, C5
C2	71.5, CH	3.60, m (1H)		C1, C3	C1, C3
C3	73.2, CH	3.95, m (1H)		C2, C4	C2, C4
C4	77.0, CH	3.64, m (1H)	D1	C3, C5	C3, C5
C5	71.2, CH	3.82, m (1H)		C4	C4
C6	60.8, CH <sub>2</sub>	3.84, m (2H)		overlapped	overlapped
D1	99.6, CH	5.40, d (3.5, 1H)	D2, D3, D5	D2	D2, D3, D5
D2	71.1, CH	3.63, m (1H)		D1, D3	D1, D3
D3	73.2, CH	3.94, m (1H)		D2	D2
D4	77.9, CH	3.63, m (1H)	E1	D3, D5	D3, D5

D5	68.9, CH	4.04, d (12.0, 1H)		D4, D6a, D6b	D4, D6a, D6b
D6a		4.22, m (1H)		D5, D6b	D5, D6b
D6b	64.3, CH <sub>2</sub>	4.45, dd (12.0, 3.0, 1H)		D5, D6a	D5, D6a
E1	100.5, CH	5.28, d (3.4, 1H)	E3, E5	E2	E2, E3, E4
E2	71.3, CH	3.53, m (1H)		E1, E3	E1, E3
E3	72.9, CH	3.58, m (1H)		E2, E4	E1, E2, E4, E6
E4	64.9, CH	2.45, t (9.0, 1H)	E3, E5, E6, F1	E3, E5	E3, E5, E6
E5	69.8, CH	3.73, m (1H)		E4, E6	E4, E6
E6	17.4, CH <sub>3</sub>	1.30, d (6.0, 3H)	E4, E5	E5	E4, E5
F1	55.0, CH	3.53, m (1H)	F2, F5	F2, F7	F2, F7
F2	69.7, CH	3.79, m (1H)		F1, F3	F1, F3
F3	70.8, CH	4.13, m (1H)		F2, F4	F2, F4
F4	76.2, CH	4.22, m (1H)	G1	F3	F3
F5	136.5, C				
F6a		4.13, m (1H)		F6b	F6b
F6b	62.0, CH <sub>2</sub>	4.22, m (1H)		F6a	F6a
F7	126.3, CH	5.97, d (3.2, 1H)	F2, F6	F1	F1, F2, F6b
G1	97.6, CH	5.37, d (3.6, 1H)	G3, G5	G2	G2, G5
G2	71.5, CH	3.63, m (1H)		G1, G3	G1, G3
G3	73.5, CH	3.92, m (1H)		G2	G2
G4	70.8, CH	3.63, m (1H)	H1	G3	G3
G5	71.1, CH	3.92, m (1H)		G4, G6	G4, G6
G6	63.4, CH <sub>2</sub>	3.86, m (2H)		G5	G5
H1	99.9, CH	5.32, d (3.4, 1H)	H3, H5	H2	H2, H4, H5
H2	71.6, CH	3.61, m (1H)		H1, H3	H1, H3
H3	72.5, CH	3.65, m (1H)		H2, H4	H1, H2, H4, H6
H4	64.9, CH	2.45, t (9.0, 1H)	H3, H5, H6	H3, H5	H3, H5, H6
H5	69.6, CH	3.78, m (1H)		H4, H6	H4, H6
H6	17.3, CH <sub>3</sub>	1.34, d (6.0, 3H)	H4, H5	H5	H2, H4, H5
I1	56.0, CH	3.53, m (1H)	I2, I6	I2, I7	I2, I7
I2	72.7, CH	3.66, m (1H)		I1, I3	I1, I3
I3	72.9, CH	3.78, m (1H)		I2, I4	I2, I4
I4	70.9, CH	4.04, d (12.0, 1H)		I3	I3
I5	138.9, C				
I6a		4.13, m (1H)		I6b	I6b
I6b	61.6, CH <sub>2</sub>	4.22, m (1H)		I6a	I6a
I7	123.8, CH	5.90, d (3.4, 1H)	I1, I2, I6	I1	I1, I6a, I6b
1'	176.7, C=O				
2'	35.6, CH <sub>2</sub>	2.45, m (2H)	1', 3', 4'	3'b	3'b, 4'
3'a		1.18, m (1H)	2'		2'
3'b	17.9, CH <sub>2</sub>	1.65, m (1H)	1', 2', 4'	2', 4'	2', 4'
4'	12.9, CH <sub>3</sub>	0.94, t (7.2, 3H)	2', 3'	3'b	2', 3'b



**Table S9.**  $^1\text{H}$  (500 MHz) and  $^{13}\text{C}$  (125 MHz) NMR data of **16** ( $\delta$ ppm) in  $\text{D}_2\text{O}$ .

No.	$\delta_{\text{C}}$	$\delta_{\text{H}}$	HMBC ( $^1\text{H} \rightarrow ^{13}\text{C}$ )	COSY ( $^1\text{H} \rightarrow ^1\text{H}$ )	TOCSY ( $^1\text{H} \rightarrow ^1\text{H}$ )
A1 $\alpha$	91.9, CH	5.21, d (3.5, 1H)	A2 $\alpha$ , A5 $\alpha$	A2 $\alpha$	A2 $\alpha$ , A5 $\alpha$
A2 $\alpha$	71.3, CH	3.50, m (1H)		A1 $\alpha$ , A3 $\alpha$	A1 $\alpha$ , A3 $\alpha$
A3 $\alpha$	73.3, CH	3.92, m (1H)		A2 $\alpha$ , A4 $\alpha$	A2 $\alpha$ , A4 $\alpha$
A4 $\alpha$	76.8, CH	3.64, m (1H)	B1	A3 $\alpha$ , A5 $\alpha$	A3 $\alpha$ , A5 $\alpha$
A5 $\alpha$	69.9, CH	3.84, m (1H)		A4 $\alpha$ , A6 $\alpha$ ,	A4 $\alpha$ , A6 $\alpha$ ,
A6 $\alpha$	60.4, CH <sub>2</sub>	3.78, m (2H)		A5 $\alpha$	A5 $\alpha$
A1 $\beta$	95.8, CH	4.63, d (8.0, 1H)	A2 $\beta$ , A3 $\beta$	A2 $\beta$	A3 $\beta$ , A5 $\beta$
A2 $\beta$	74.0, CH	3.25, m (1H)		A1 $\beta$ , A3 $\beta$	A1 $\beta$ , A3 $\beta$
A3 $\beta$	76.2, CH	3.73, m (1H)		A2 $\beta$ , A4 $\beta$	A2 $\beta$ , A4 $\beta$
A4 $\beta$	76.8, CH	3.64, m (1H)	B1	A3 $\beta$ , A5 $\beta$	A3 $\beta$ , A5 $\beta$
A5 $\beta$	74.5, CH	3.61, m (1H)		A4 $\beta$ , A6 $\beta$	A4 $\beta$ , A6 $\beta$
A6 $\beta$	60.4, CH <sub>2</sub>	3.88, m (2H)		A5 $\beta$	A5 $\beta$
B1	99.4, CH	5.38, d (3.5, 1H)	B2, B4, B5	B2	B2, B3, B5
B2	71.6, CH	3.59, m (1H)		B1, B3	B1, B3
B3	73.2, CH	3.94, m (1H)		B2, B4	B2, B4
B4	77.0, CH	3.64, m (1H)	C1	B3, B5	B3, B5
B5	71.2, CH	3.82, m (1H)		B4	B4
B6	60.5, CH <sub>2</sub>	3.82, m (2H)		overlapped	overlapped
C1	99.5, CH	5.38, d (3.5, 1H)	C2, C4, C5	C2	C2, C3, C5
C2	71.5, CH	3.59, m (1H)		C1, C3	C1, C3
C3	73.3, CH	3.94, m (1H)		C2, C4	C2, C4
C4	76.9, CH	3.64, m (1H)	D1	C3, C5	C3, C5
C5	71.2, CH	3.82, m (1H)		C4	C4
C6	60.7, CH <sub>2</sub>	3.82, m (2H)		overlapped	overlapped
D1	99.6, CH	5.38, d (3.5, 1H)	D2, D4, D5	D2	D2, D3, D5
D2	71.1, CH	3.64, m (1H)		D1, D3	D1, D3
D3	73.1, CH	3.93, m (1H)		D2	D2
D4	78.4, CH	3.64, m (1H)	E1	D3, D5	D3, D5

D5	69.0, CH	4.01, d (12.0, 1H)		D4, D6a, D6b	D4, D6a, D6b
D6a	63.4, CH <sub>2</sub>	4.19, m (1H)		D5, D6b	D5, D6b
D6b		4.43, d (12.0, 1H)		D5, D6a	D5, D6a
E1	100.7, CH	5.24, d (3.5, 1H)	E3, E5	E2	E2, E3, E4
E2	71.3, CH	3.50, m (1H)		E1, E3	E1, E3
E3	72.9, CH	3.52, m (1H)		E2, E4	E1, E2, E4
E4	64.3, CH	2.43, t (9.0, 1H)	E3, E5, E6, F1	E3, E5	E3, E5, E6
E5	69.7, CH	3.71, m (1H)		E4, E6	E4, E6
E6	17.4, CH <sub>3</sub>	1.29, d (5.6, 3H)	E4, E5	E5	E4, E5
F1	55.0, CH	3.45, m (1H)	F2, F5	F2, F7	F2, F7
F2	69.7, CH	3.76, m (1H)		F1, F3	F1, F3
F3	70.8, CH	4.09, m (1H)		F2, F4	F2, F4
F4	76.3, CH	4.19, m (1H)	G1	F3	F3
F5	136.5, C				
F6a		4.09, m (1H)		F6b	F6b
F6b	62.0, CH <sub>2</sub>	4.19, m (1H)		F6a	F6a
F7	126.6, CH	5.94, d (3.1, 1H)	F2, F6	F1	F1, F2, F6b
G1	97.5, CH	5.34, d (3.5, 1H)	G3, G5	G2	G2, G5
G2	71.7, CH	3.58, m (1H)		G1, G3	G1, G3
G3	73.5, CH	3.89, m (1H)		G2	G2
G4	70.8, CH	3.59, m (1H)	H1	G3	G3
G5	71.1, CH	3.89, m (1H)		G4, G6	G4, G6
G6	62.5, CH <sub>2</sub>	3.83, m (2H)		G5	G5
H1	99.9, CH	5.30, d (3.4, 1H)	H3, H5	H2	H2, H4, H5
H2	71.5, CH	3.57, m (1H)		H1	H1
H3	72.6, CH	3.57, m (1H)		H4	H1, H4, H6
H4	64.9, CH	2.43, t (9.0, 1H)	H3, H5, H6	H3, H5	H3, H5, H6
H5	69.6, CH	3.73, m (1H)		H4, H6	H4, H6
H6	17.3, CH <sub>3</sub>	1.30, d (5.6, 3H)	H4, H5	H5	H2, H4, H5
I1	56.0, CH	3.45, m (1H)	I2, I6	I2, I7	I2, I7
I2	72.7, CH	3.63, m (1H)		I1, I3	I1, I3
I3	72.9, CH	3.73, m (1H)		I2, I4	I2, I4
I4	70.9, CH	4.01, d (12.0, 1H)		I3	I3
I5	138.9, C				
I6a		4.09, m (1H)		I6b	I6b
I6b	61.6, CH <sub>2</sub>	4.19, m (1H)		I6a	I6a
I7	123.8, CH	5.87, d (3.5, 1H)	I1, I2, I6	I1	I1, I6a, I6b
1'	179.8, C=O				
2'	40.9, CH	2.52, m (1H)	3', 4', 5'	3'b, 5'	3'a, 3'b, 4', 5'
3'a		1.51, m (1H)	2', 4', 5'	4'	2', 3'b, 4', 5'
3'b	26.4, CH <sub>2</sub>	1.63, m (1H)	2', 4', 5'	2', 4'	2', 3'a, 4', 5'
4'	10.9, CH <sub>3</sub>	0.88, t (7.0, 3H)	2', 3'	3'a, 3'b	3'a, 3'b, 5'
5'	15.7, CH <sub>3</sub>	1.13, d (7.0, 3H)	2', 3'	2'	2', 4'

**Table S10.** Acarviostatins with glucose(s) at the reducing terminus (Aca-glu) from *Streptomyces* sp. HO1518.

No.	Compounds	Formula	$\Delta$ (ppm)	$t_R$ (min)	$[M + H]^+$	Characterized fragment ions	Ref.
1	Aca I01	C <sub>25</sub> H <sub>43</sub> NO <sub>18</sub>	0.2	6.00	646.2553	304.1392, 646.2554	1
2	Aca I02	C <sub>31</sub> H <sub>53</sub> NO <sub>23</sub>	1.7	6.14	808.3081	304.1395, 808.3095	2
3	Aca I03 ( <b>17</b> )	C <sub>37</sub> H <sub>63</sub> NO <sub>28</sub>	0.1	6.55	970.3609	304.1397, 970.3610	3
4	Aca II00	C <sub>38</sub> H <sub>64</sub> N <sub>2</sub> O <sub>25</sub>	1.4	6.82	949.3871	304.1398, 769.3251, 949.3884	4
5	Aca II01	C <sub>44</sub> H <sub>74</sub> N <sub>2</sub> O <sub>30</sub>	−0.4	6.93	1111.4399	304.1401, 769.3245, 808.3090, 1111.4395	5
6	Aca II02 ( <b>10</b> )	C <sub>50</sub> H <sub>84</sub> N <sub>2</sub> O <sub>35</sub>	0.2	7.81	1273.4927	304.1394, 769.3244, 970.3609, 1273.4929	—
7	Aca II03 ( <b>9</b> )	C <sub>56</sub> H <sub>94</sub> N <sub>2</sub> O <sub>40</sub>	−2.0	9.27	1435.5456	304.1386, 769.3219, 1132.4094, 1435.5427	3
8	Aca II04	C <sub>62</sub> H <sub>104</sub> N <sub>2</sub> O <sub>45</sub>	0.1	7.74	1597.5984	304.1389, 769.3233, 1294.4610, 1597.5986	—
9	Aca II05	C <sub>68</sub> H <sub>114</sub> N <sub>2</sub> O <sub>50</sub>	−1.2	9.18	1759.6512	304.1389, 769.3233, 1759.6491	—
10	Ac-Aca I01	C <sub>27</sub> H <sub>45</sub> NO <sub>19</sub>	−0.1	10.71	688.2659	304.1396, 688.2658	—
11	Ac-Aca I02	C <sub>33</sub> H <sub>55</sub> NO <sub>24</sub>	−1.9	10.93	850.3187	304.1393, 850.3171	—
12	Ac-Aca I03 ( <b>1</b> )	C <sub>39</sub> H <sub>65</sub> NO <sub>29</sub>	−1.2	10.99	1012.3715	304.1395, 1012.3703	6
13	Ac-Aca II01	C <sub>46</sub> H <sub>76</sub> N <sub>2</sub> O <sub>31</sub>	−0.5	10.31	1153.4505	304.1392, 769.3233, 850.3168, 1153.4499	—
14	Ac-Aca II02	C <sub>52</sub> H <sub>86</sub> N <sub>2</sub> O <sub>36</sub>	−1.8	10.80	1315.5033	304.1390, 769.3231, 1012.3683, 1315.5009	—
15	Ac-Aca II03 ( <b>7</b> )	C <sub>58</sub> H <sub>97</sub> N <sub>2</sub> O <sub>41</sub>	−1.2	11.11	1477.5561	304.1389, 769.3231, 1174.4232, 1477.5543	7
16	Hac-Aca I03	C <sub>39</sub> H <sub>65</sub> NO <sub>30</sub>	−1.0	10.29	1028.3664	304.1391, 1028.3654	—
17	Hac-Aca II03	C <sub>58</sub> H <sub>96</sub> N <sub>2</sub> O <sub>42</sub>	2.4	11.80	1493.5510	304.1392, 769.3219, 1493.5546	—
18	Pr-Aca I01	C <sub>28</sub> H <sub>47</sub> NO <sub>19</sub>	−3.4	12.39	702.2815	304.1385, 702.2791	—
19	Pr-Aca I02	C <sub>34</sub> H <sub>57</sub> NO <sub>24</sub>	−2.0	12.59	864.3343	304.1379, 864.3326	—
20	Pr-Aca I03 ( <b>2</b> )	C <sub>40</sub> H <sub>67</sub> NO <sub>29</sub>	−1.0	13.10	1026.3872	304.1395, 1026.3862	6
21	Pr-Aca II01	C <sub>47</sub> H <sub>78</sub> N <sub>2</sub> O <sub>31</sub>	−0.6	11.63	1167.4661	304.1394, 769.3236, 864.3338, 1167.4654	—
22	Pr-Aca II02 ( <b>11</b> )	C <sub>53</sub> H <sub>88</sub> N <sub>2</sub> O <sub>36</sub>	−0.7	11.86	1329.5190	304.1395, 769.3233, 1026.3870, 1329.5181	—
23	Pr-Aca II03 ( <b>14</b> )	C <sub>59</sub> H <sub>98</sub> N <sub>2</sub> O <sub>41</sub>	−0.2	12.40	1491.5718	304.1377, 769.3239, 1188.4393, 1491.5715	—
24	Pr-Aca II04	C <sub>65</sub> H <sub>108</sub> N <sub>2</sub> O <sub>46</sub>	−4.0	12.15	1653.6246	304.1394, 769.3229, 1653.6180	—
25	Pr-Aca III03	C <sub>78</sub> H <sub>129</sub> N <sub>3</sub> O <sub>53</sub>	−2.8	11.95	1956.7564	304.1382, 769.3221, 1188.4364, 1956.7510	—
26	Hpr-Aca I02	C <sub>34</sub> H <sub>57</sub> NO <sub>25</sub>	−1.4	12.07	880.3292	304.1389, 880.3280	—
27	Hpr-Aca I03	C <sub>40</sub> H <sub>67</sub> NO <sub>30</sub>	−2.9	12.81	1042.3821	304.1381, 769.3215, 1042.3791	—
28	Hpr-Aca II03	C <sub>59</sub> H <sub>98</sub> N <sub>2</sub> O <sub>42</sub>	1.8	13.61	1507.5667	304.1387, 769.3211, 1204.4598, 1507.5694	—
29	isoBu-Aca I01	C <sub>29</sub> H <sub>49</sub> NO <sub>19</sub>	−0.4	14.14	716.2972	304.1383, 716.2969	—

30	isoBu-Aca I02	C <sub>35</sub> H <sub>59</sub> NO <sub>24</sub>	-1.1	14.56	878.3500	304.1390, 878.3490	-
31	isoBu-Aca I03 (3)	C <sub>41</sub> H <sub>69</sub> NO <sub>29</sub>	0.6	15.43	1040.4028	304.1481, 1040.4034	7
32	Bu-Aca I03 (18)	C <sub>41</sub> H <sub>69</sub> NO <sub>29</sub>	-0.1	15.53	1040.4028	304.1391, 1040.4027	8
33	Bu-Aca II01	C <sub>48</sub> H <sub>80</sub> N <sub>2</sub> O <sub>31</sub>	-1.1	13.55	1181.4818	304.1380, 769.3232, 878.3489, 1181.4805	8
34	isoBu-Aca II02 (12)	C <sub>54</sub> H <sub>90</sub> N <sub>2</sub> O <sub>36</sub>	-2.9	13.75	1343.5346	304.1386, 769.3255, 1040.4000, 1343.5307	-
35	isoBu-Aca II03 (8)	C <sub>60</sub> H <sub>100</sub> N <sub>2</sub> O <sub>41</sub>	0.2	14.40	1505.5874	304.1391, 769.3228, 1202.4547, 1505.5877	7
36	Bu-Aca II03 (15)	C <sub>60</sub> H <sub>100</sub> N <sub>2</sub> O <sub>41</sub>	2.2	14.48	1505.5874	304.1392, 769.3238, 1202.4570, 1505.5907	-
37	isoBu-Aca II04	C <sub>66</sub> H <sub>110</sub> N <sub>2</sub> O <sub>46</sub>	-3.8	15.76	1667.6403	304.1392, 769.3230, 1667.6339	-
38	Hbu-Aca I01	C <sub>29</sub> H <sub>49</sub> NO <sub>20</sub>	-2.4	9.71	732.2921	304.1383, 732.2903	-
39	Hbu-Aca I02	C <sub>35</sub> H <sub>59</sub> NO <sub>25</sub>	-0.3	10.15	894.3449	304.1397, 894.3446	-
40	Hbu-Aca I03 (4)	C <sub>41</sub> H <sub>70</sub> NO <sub>30</sub>	-3.2	11.31	1056.3977	304.1386, 1056.3943	6
41	Hbu-Aca II01	C <sub>48</sub> H <sub>80</sub> N <sub>2</sub> O <sub>32</sub>	-2.4	10.35	1197.4767	304.1392, 769.3224, 894.3440, 1197.4738	-
42	Hbu-Aca II02	C <sub>54</sub> H <sub>90</sub> N <sub>2</sub> O <sub>37</sub>	-1.2	10.49	1359.5295	304.1382, 769.3230, 1056.3944, 1359.5279	-
43	Hbu-Aca II03 (19)	C <sub>60</sub> H <sub>100</sub> N <sub>2</sub> O <sub>42</sub>	-0.8	10.85	1521.5823	304.1383, 769.3228, 1218.4477, 1521.5810	6
44	isoVa-Aca I01	C <sub>30</sub> H <sub>51</sub> NO <sub>19</sub>	-1.6	15.83	730.3128	304.1385, 730.3116	-
45	isoVa-Aca I02	C <sub>36</sub> H <sub>61</sub> NO <sub>24</sub>	-0.4	16.78	892.3656	304.1408, 892.3660	-
46	Mbu-Aca I03 (5)	C <sub>42</sub> H <sub>71</sub> NO <sub>29</sub>	-1.1	17.93`	1054.4184	304.1388, 1054.4172	7
47	isoVa-Aca I03 (6)	C <sub>42</sub> H <sub>71</sub> NO <sub>29</sub>	1.5	18.04	1054.4184	304.1388, 1054.4200	9
48	isoVa-Aca II01	C <sub>49</sub> H <sub>82</sub> N <sub>2</sub> O <sub>30</sub>	-2.4	14.91	1195.4974	304.1300, 769.3226, 1195.4945	-
49	isoVa-Aca II02 (13)	C <sub>55</sub> H <sub>92</sub> N <sub>2</sub> O <sub>36</sub>	0.1	16.06	1357.5503	304.1390, 769.3219, 1054.4195, 1357.5505	-
50	Mbu-Aca II03 (16)	C <sub>61</sub> H <sub>102</sub> N <sub>2</sub> O <sub>41</sub>	0.6	16.65	1519.6031	304.1391, 769.3234, 1216.4712, 1519.6040	-
51	isoVa-Aca II03 (20)	C <sub>61</sub> H <sub>102</sub> N <sub>2</sub> O <sub>41</sub>	0.6	16.79	1519.6031	304.1389, 769.3226, 1216.4704, 1519.6041	10
52	Hva-Aca I01	C <sub>30</sub> H <sub>51</sub> NO <sub>20</sub>	1.6	10.90	746.3077	304.1378, 746.3089	-
53	Hva-Aca I02	C <sub>36</sub> H <sub>61</sub> NO <sub>25</sub>	-4.5	11.93	908.3605	304.1369, 908.3564	-
54	Hva-Aca I03	C <sub>42</sub> H <sub>71</sub> NO <sub>30</sub>	-1.8	13.08	1070.4134	304.1372, 1070.4115	-
55	He-Aca I01	C <sub>31</sub> H <sub>53</sub> NO <sub>19</sub>	-0.7	17.50	744.3285	304.1377, 744.3280	-
56	He-Aca I02	C <sub>37</sub> H <sub>63</sub> NO <sub>24</sub>	-1.3	19.34	906.3813	304.1379, 906.3801	-
57	He-Aca I03	C <sub>43</sub> H <sub>73</sub> NO <sub>29</sub>	-1.1	21.06	1068.4341	304.1378, 1068.4329	-
58	He-Aca II02	C <sub>56</sub> H <sub>94</sub> N <sub>2</sub> O <sub>36</sub>	-3.4	18.04	1371.5659	304.1392, 769.3182, 1371.5612	-
59	He-Aca II03	C <sub>62</sub> H <sub>104</sub> N <sub>2</sub> O <sub>41</sub>	-3.9	19.55	1533.6187	304.1379, 769.3215, 1230.4826, 1533.6127	-
60	Hhe-Aca I03	C <sub>43</sub> H <sub>73</sub> NO <sub>30</sub>	-1.3	18.97	1084.4290	304.1396, 1084.4276	-
61	Hhe-Aca II01	C <sub>50</sub> H <sub>84</sub> N <sub>2</sub> O <sub>32</sub>	0.7	17.80	1225.5080	304.1394, 769.3238, 922.3765, 1225.5089	-

62	Hhe-Aca II02	C <sub>56</sub> H <sub>94</sub> N <sub>2</sub> O <sub>37</sub>	−0.2	17.93	1387.5608	304.1392, 769.3233, 1084.4272, 1387.5605	–
63	diHva-Aca I03	C <sub>42</sub> H <sub>71</sub> NO <sub>31</sub>	−3.4	8.96	1086.4083	304.1390, 1086.4046	–

Aca, acarviostatin; Ac, acetyl; Hac, hydroxyacetyl; Pr, propionyl; Hpr, hydroxypropionyl; Bu, butyryl; isoBu, isobutyryl; Hbu, hydroxybutyryl; Mbu, 2-methyl-butyl; isoVa, isovaleryl; Hva, hydroxyvaleryl; He, hexanoyl; Hhe, hydroxyhexanoyl; diHva, dihydroxyvaleryl. Acarviostatins, marked with “–” in Ref. column, are potential new compounds.

**Table S11.** Acarviostatins with glucose(s) at the reducing and nonreducing terminus (glu-Aca-glu) from *Streptomyces* sp. HO1518<sup>a</sup>.

No.	Compounds	Formula	Δ (ppm)	t <sub>R</sub> (min)	[M + H] <sup>+</sup>	Characterized fragment ions
1	Aca I13	C <sub>43</sub> H <sub>73</sub> NO <sub>33</sub>	−0.7	6.80	1132.4138	466.1933, 1132.4130
2	Ac-Aca I10	C <sub>27</sub> H <sub>45</sub> NO <sub>19</sub>	1.6	10.34	688.2659	466.1900, 688.2670
3	Ac-Aca I12	C <sub>39</sub> H <sub>65</sub> NO <sub>29</sub>	−1.7	10.19	1012.3715	466.1918, 1012.3698
4	Ac-Aca I13	C <sub>45</sub> H <sub>75</sub> NO <sub>34</sub>	−1.1	10.50	1174.4243	466.1921, 1174.4230
5	Ac-Aca II11	C <sub>52</sub> H <sub>86</sub> N <sub>2</sub> O <sub>36</sub>	0.2	10.30	1315.5033	466.1922, 850.3170, 931.3730, 1315.5035
6	Pr-Aca I10	C <sub>28</sub> H <sub>47</sub> NO <sub>19</sub>	1.1	12.09	702.2815	466.1920, 702.2823
7	Pr-Aca I13	C <sub>46</sub> H <sub>77</sub> NO <sub>34</sub>	−1.3	12.20	1188.4400	466.1915, 1188.4384
8	Hpr-Aca II12	C <sub>59</sub> H <sub>98</sub> N <sub>2</sub> O <sub>42</sub>	−1.6	11.83	1507.5667	466.1921, 1042.3769, 931.3743, 1507.5642
9	Bu-Aca I13	C <sub>47</sub> H <sub>79</sub> NO <sub>34</sub>	−1.7	13.44	1202.4556	466.1910, 1202.4536
10	Hbu-Aca I10	C <sub>29</sub> H <sub>49</sub> NO <sub>20</sub>	−1.8	9.45	732.2921	466.1920, 732.2908
11	Hbu-Aca I12	C <sub>41</sub> H <sub>70</sub> NO <sub>30</sub>	−0.5	10.13	1056.3977	466.1916, 1056.3972
12	Hbu-Aca I13	C <sub>47</sub> H <sub>79</sub> NO <sub>35</sub>	−0.6	10.02	1218.4505	466.1920, 1218.4498
13	Hbu-Aca II10	C <sub>48</sub> H <sub>80</sub> N <sub>2</sub> O <sub>32</sub>	−2.0	9.97	1197.4767	466.1919, 931.3626, 1197.4743
14	isoVa-Aca I13	C <sub>48</sub> H <sub>81</sub> NO <sub>34</sub>	−2.0	15.10	1216.4713	466.1908, 1216.4688
15	Hva-Aca I11	C <sub>36</sub> H <sub>61</sub> NO <sub>25</sub>	−2.3	11.34	908.3605	466.1902, 908.3584

Aca, acarviostatin; Ac, acetyl; Pr, propionyl; Hpr, hydroxypropionyl; Hbu, hydroxybutyryl; Hva, hydroxyvaleryl. <sup>a</sup> all isolates are potential new compounds.



**Table S12.** Acarviostatins with an incomplete pseudo-trisaccharide at the nonreducing terminus (incAca-glu) from *Streptomyces* sp. HO1518<sup>a</sup>.

No.	Compounds	Formula	$\Delta$ (ppm)	$t_R$ (min)	$[M + H]^+$	Characterized fragment ions
1	Aca I(−1)2	C <sub>30</sub> H <sub>53</sub> NO <sub>24</sub>	0.1	5.90	812.3030	146.0813, 812.3031
2	Aca II(−1)2	C <sub>43</sub> H <sub>74</sub> N <sub>2</sub> O <sub>31</sub>	−1.5	7.56	1115.4348	146.0811, 611.2658, 1115.4331
3	Aca II(−1)3	C <sub>49</sub> H <sub>84</sub> N <sub>2</sub> O <sub>36</sub>	−2.0	8.94	1277.4877	146.0804, 611.2647, 1277.4851
4	Ac-Aca I(−1)3	C <sub>32</sub> H <sub>55</sub> NO <sub>25</sub>	−2.2	10.44	854.3136	146.0810, 854.3117
5	Ac-Aca II(−1)2	C <sub>45</sub> H <sub>76</sub> N <sub>2</sub> O <sub>32</sub>	2.5	10.23	1157.4454	146.0810, 611.2639, 1157.4483
6	Ac-Aca II(−1)3	C <sub>51</sub> H <sub>86</sub> N <sub>2</sub> O <sub>37</sub>	−2.2	10.60	1319.4982	146.0811, 611.2655, 1174.4167, 1319.4953
7	Hac-Aca I(−1)3	C <sub>32</sub> H <sub>55</sub> NO <sub>26</sub>	−2.0	9.74	870.3085	146.0809, 870.3067
8	Pr-Aca I(−1)3	C <sub>34</sub> H <sub>57</sub> NO <sub>24</sub>	−1.3	12.42	868.3292	146.0811, 868.3281
9	Pr-Aca II(−1)2	C <sub>46</sub> H <sub>78</sub> N <sub>2</sub> O <sub>32</sub>	0.8	11.89	1171.4610	146.0809, 611.2623, 1026.3845, 1171.4600
10	Pr-Aca II(−1)3	C <sub>52</sub> H <sub>88</sub> N <sub>2</sub> O <sub>37</sub>	−4.4	12.37	1333.5139	146.0810, 611.2648, 1188.4208, 1333.5080
11	Hpr-Aca II(−1)3	C <sub>52</sub> H <sub>88</sub> N <sub>2</sub> O <sub>38</sub>	−1.6	12.50	1349.5088	146.0810, 611.2655, 1204.4326, 1349.5066
12	Bu-Aca II(−1)2	C <sub>47</sub> H <sub>80</sub> N <sub>2</sub> O <sub>32</sub>	3.8	13.72	1185.4767	146.0800, 611.2651, 1185.4812
13	Bu-Aca II(−1)3	C <sub>53</sub> H <sub>90</sub> N <sub>2</sub> O <sub>37</sub>	−3.8	14.10	1347.5295	146.0799, 611.2649, 1202.4555, 1347.5245
14	Hbu-Aca I(−1)3	C <sub>34</sub> H <sub>59</sub> NO <sub>26</sub>	−2.4	9.64	898.3398	146.0803, 898.3376
15	Hbu-Aca II(−1)2	C <sub>47</sub> H <sub>80</sub> N <sub>2</sub> O <sub>33</sub>	−3.4	10.34	1201.4716	146.0812, 611.2656, 1056.3941, 1201.4675
16	isoVa-Aca I(−1)3	C <sub>35</sub> H <sub>61</sub> NO <sub>25</sub>	−2.4	15.90	896.3605	146.0805, 896.3583
17	isoVa-Aca II(−1)2	C <sub>48</sub> H <sub>82</sub> N <sub>2</sub> O <sub>32</sub>	−1.8	15.72	1199.4923	146.0810, 611.2651, 1054.4174, 1199.4901
18	isoVa-Aca II(−1)3	C <sub>54</sub> H <sub>92</sub> N <sub>2</sub> O <sub>37</sub>	1.0	16.13	1361.5452	146.0810, 611.2637, 1216.4712, 1361.5465
19	Hva-Aca II(−1)2	C <sub>48</sub> H <sub>82</sub> N <sub>2</sub> O <sub>33</sub>	−2.4	11.20	1215.4873	146.0797, 611.2645, 1215.4844
20	Hva-Aca II(−1)3	C <sub>54</sub> H <sub>92</sub> N <sub>2</sub> O <sub>38</sub>	−3.0	11.41	1377.5401	146.0797, 611.2644, 1218.4834, 1377.5360

Aca, acarviostatin; Ac, acetyl; Hac, hydroxyacetyl; Pr, propionyl; Hpr, hydroxypropionyl; Bu, butyryl; Hbu, hydroxybutyryl; isoVa, isovaleryl; Hva, hydroxyvaleryl.

<sup>a</sup> all isolates are potential new compounds.

## References

- Schmidt, D.D.; Frommer, W.; Junge, B.; Müller, L.; Wingender, W.; Truscheit, E.; Schäfer, D.  $\alpha$ -Glucosidase inhibitors. New complex oligosaccharides of microbial origin. *Naturwissenschaften* **1977**, *64*, 535–536.

2. Fukuhara, K.; Murai, H.; Murao, S. Isolation and structure-activity relationship of some amylostatins (F-Ib Fraction) produced by *Streptomyces diastaticus* subsp *amylostaticus* No. 9410. *Agric. Biol. Chem.* **1982**, *46*, 1941–1945.
3. Geng, P.; Qiu, F.; Zhu, Y.Y.; Bai, G. Four acarviosin-containing oligosaccharides identified from *Streptomyces coelicoflavus* ZG0656 are potent inhibitors of  $\alpha$ -amylase. *Carbohydr. Res.* **2008**, *343*, 882–892.
4. Qian, M.X.; Nahoum, V.; Bonicel, J.; Bischoff, H.; Henrissat, B.; Payan, F. Enzyme-catalyzed condensation reaction in a mammalian  $\alpha$ -amylase. High resolution structural analysis of an enzymeinhibitor complex. *Biochemistry* **2001**, *40*, 7700–7709.
5. Weiss, S.C.; Skerra, A.; Schiefner, A. Structural basis for the interconversion of maltodextrins by MalQ, the amyломaltase of *Escherichia coli*. *J. Biol. Chem.* **2015**, *290*, 21352–21364.
6. Liu, H.L.; E, H.C.; Xie, D.A.; Cheng, W.B.; Tao, W.Q.; Wang, Y. Acylated aminooligosaccharides with inhibitory effects against  $\alpha$ -amylase from *Streptomyces* sp. HO1518. *Mar. Drugs* **2018**, *16*, 403.
7. Xu, J.L.; Liu, H.L.; Liu, Z.F.; Ren, Y.H.; Wang, Y. Acylated aminooligosaccharides from the yellow sea *Streptomyces* sp. HO1518 as both  $\alpha$ -glucosidase and lipase inhibitors. *Mar. Drugs* **2020**, *18*, 576.
8. Si, D.Y.; Zhong, D.F.; Xu, Q.M. Two butylated aminooligosaccharides isolated from the culture filtrate of *Streptomyces luteogriseus*. *Carbohydr. Res.* **2001**, *335*, 127–132.
9. Zhong, D.F.; Si, D.Y.; He, W.Y.; Zhao, L.M.; Xu, Q.M. Structural revision of isovalertatins M03, M13, and M23 isolated from the culture of *Streptomyces luteogriseus*. *Carbohydr. Res.* **2001**, *331*, 69–75.
10. Si, D.Y.; Zhong, D.F.; He, W.Y.; Zhao, L.M. Structural revision of isovalertatins D03 and D23 isolated from the culture filtrate of *Streptomyces luteogriseus*. *Chin. Chem. Lett.* **2001**, *12*, 327–330.

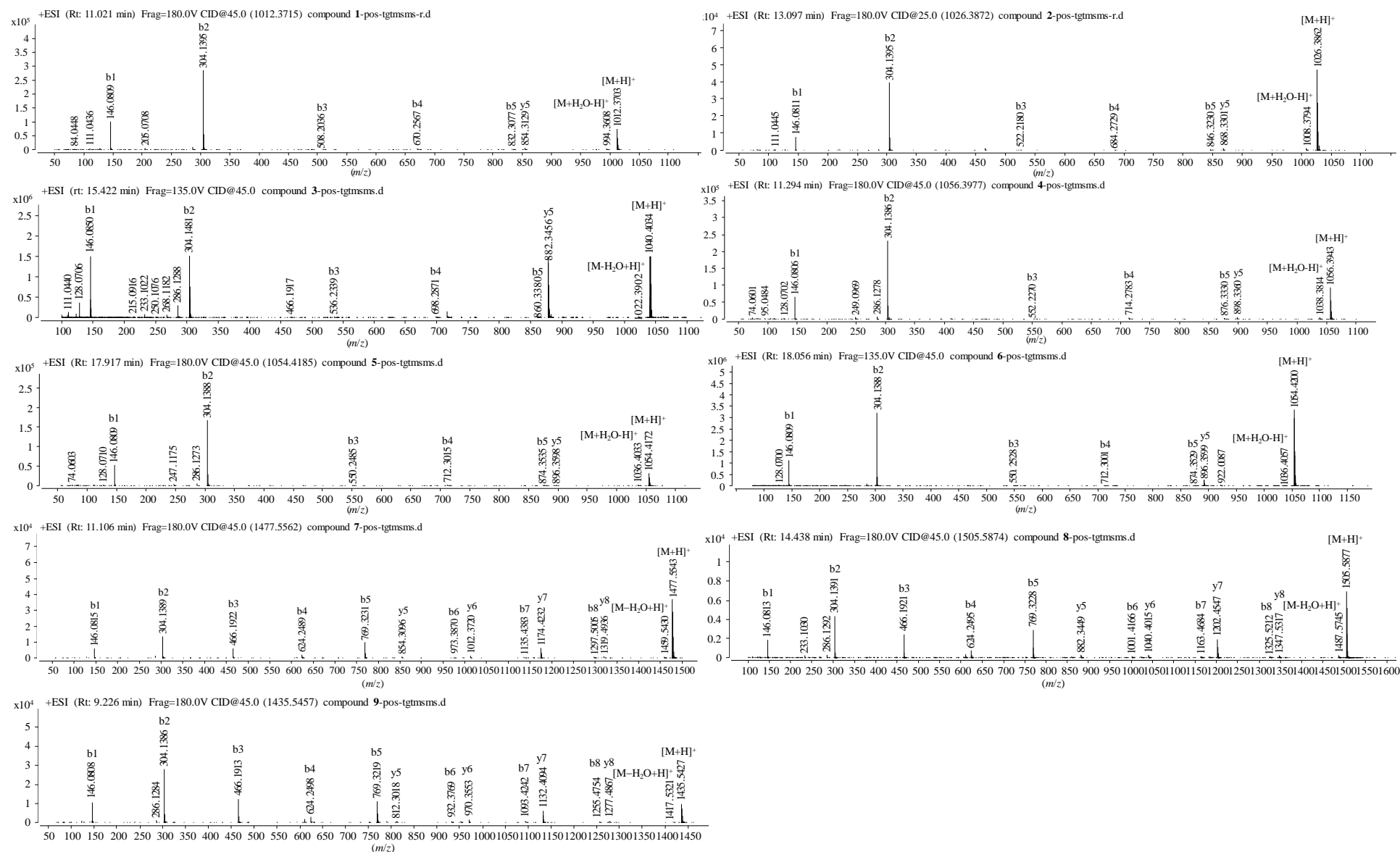
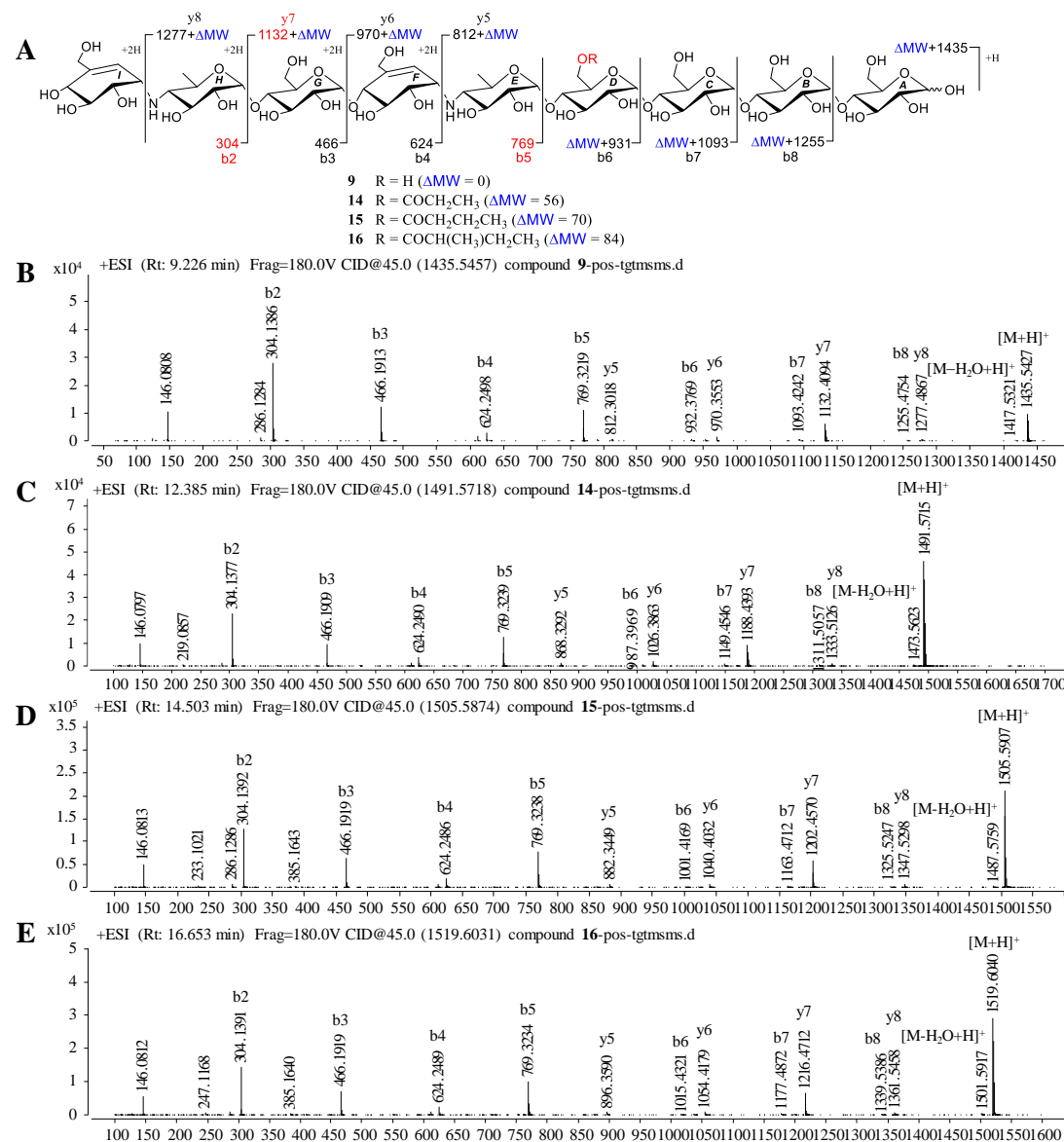
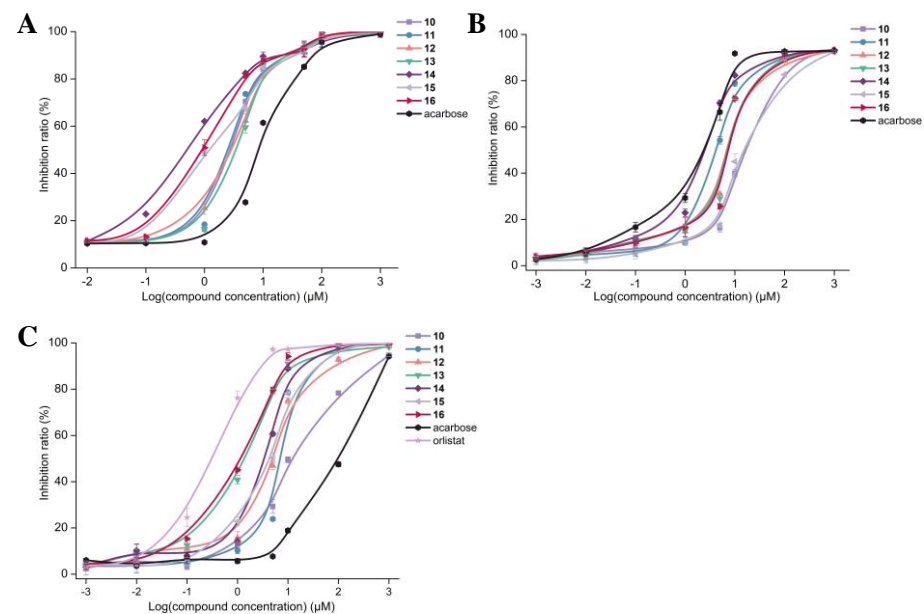


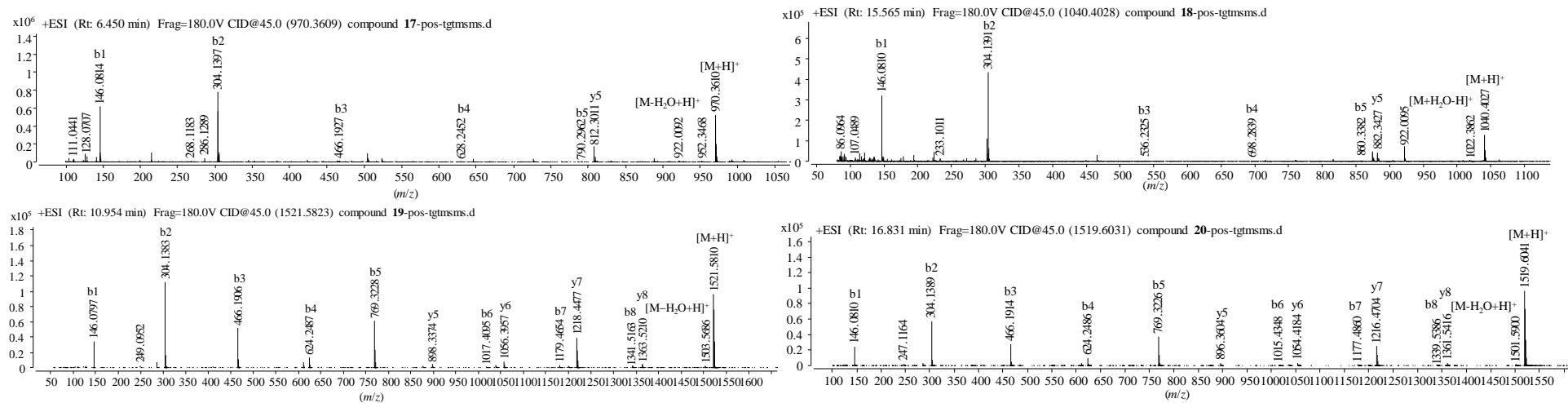
Figure S1. HRESI-MS/MS spectra of compounds 1-9.



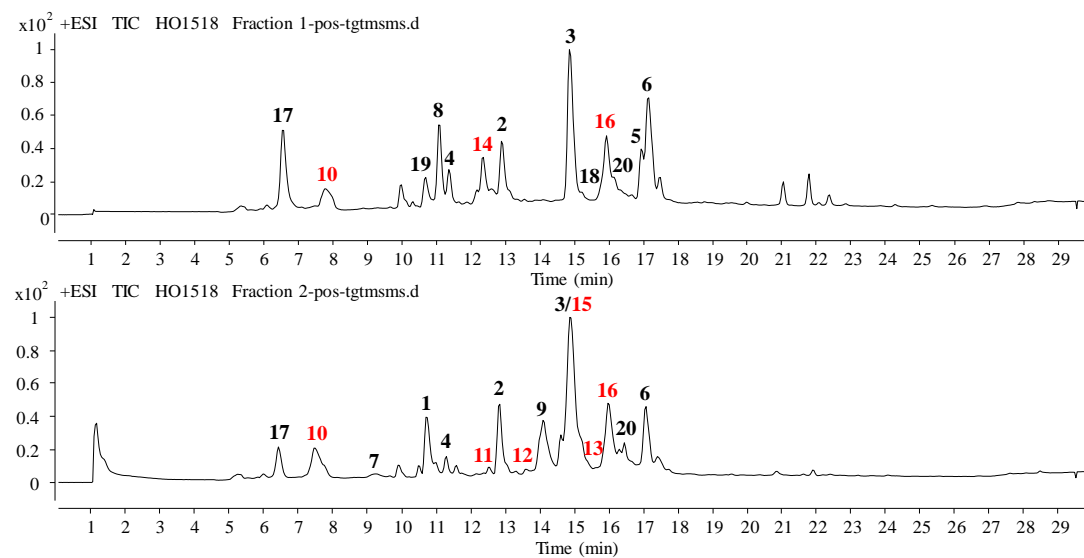
**Figure S2.** Positive HRESIMS/MS fragmentation and spectra of **9** and **14-16**. (A) Positive-ion HRESIMS/MS fragmentation patterns of **9** and **14-16**; (B–E) HRESIMS/MS spectra of **9** and **14-16**.



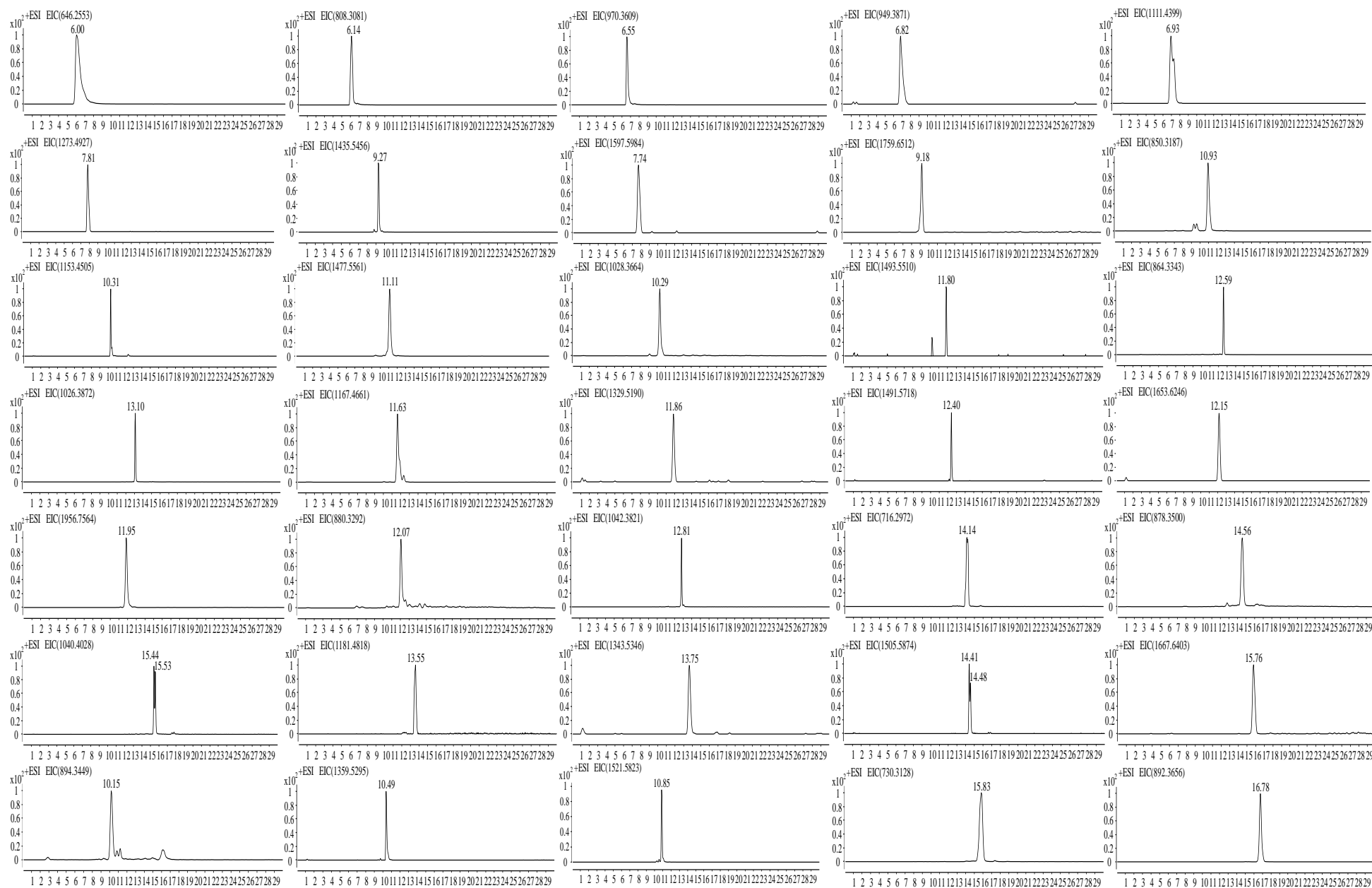
**Figure S3.** The inhibitory activities of **10-16** against three digestive enzymes. (A) The inhibitory activities of **10-16** against PPA. (B) The inhibitory activities of **10-16** against sucrase. (C) The inhibitory activities of **10-16** against PL.

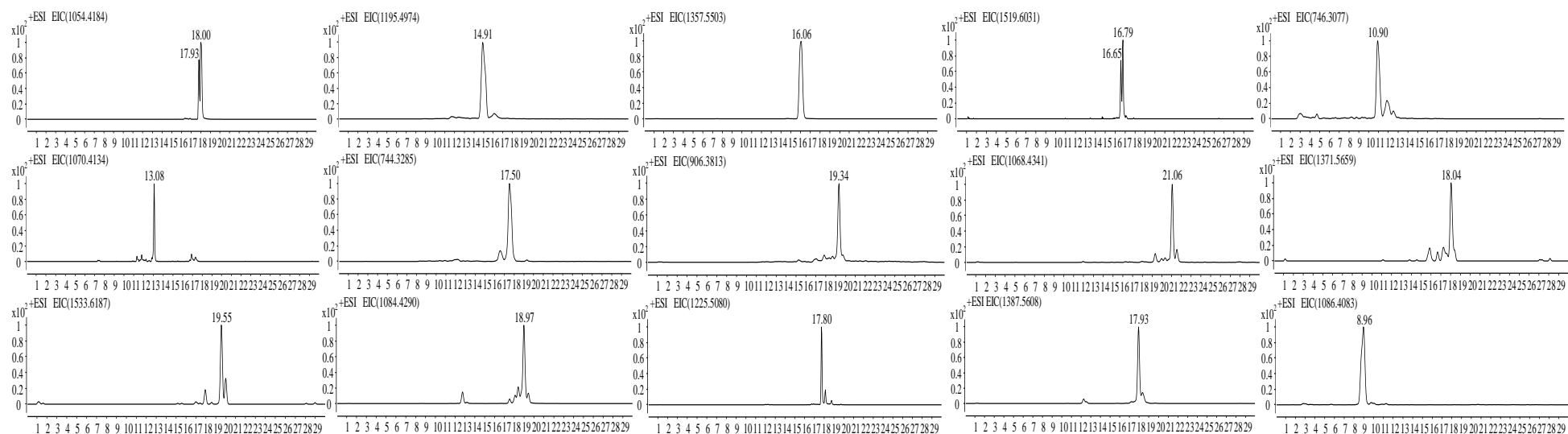


**Figure S4.** HRESI-MS/MS spectra of compounds **17-20**.

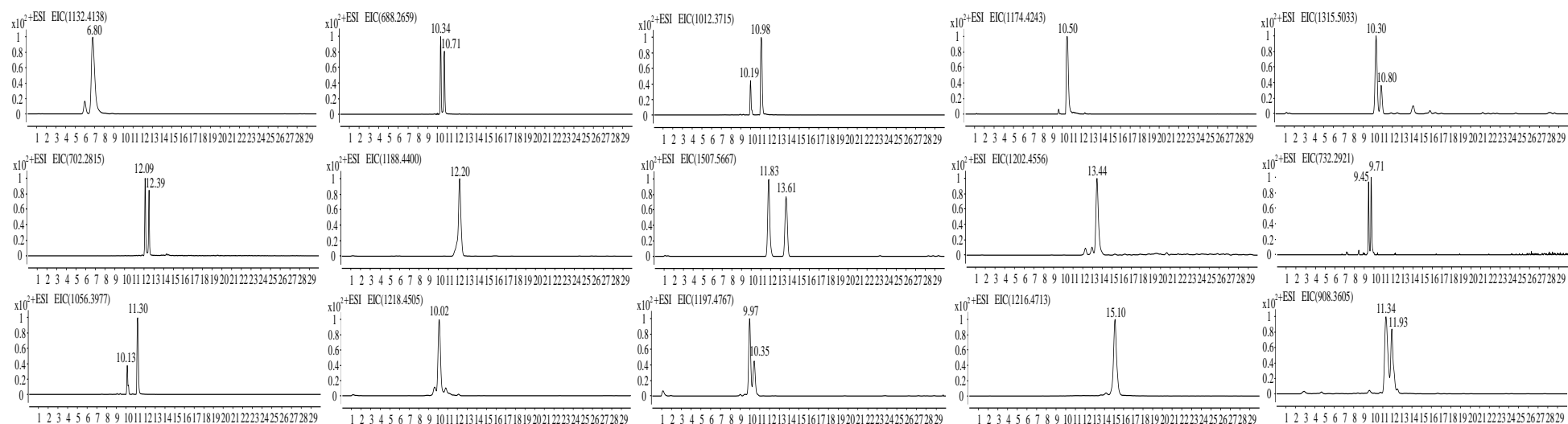


**Figure S5.** The total ion chromatograms of fraction 1 and 2 derived from *Streptomyces* sp. HO1518.



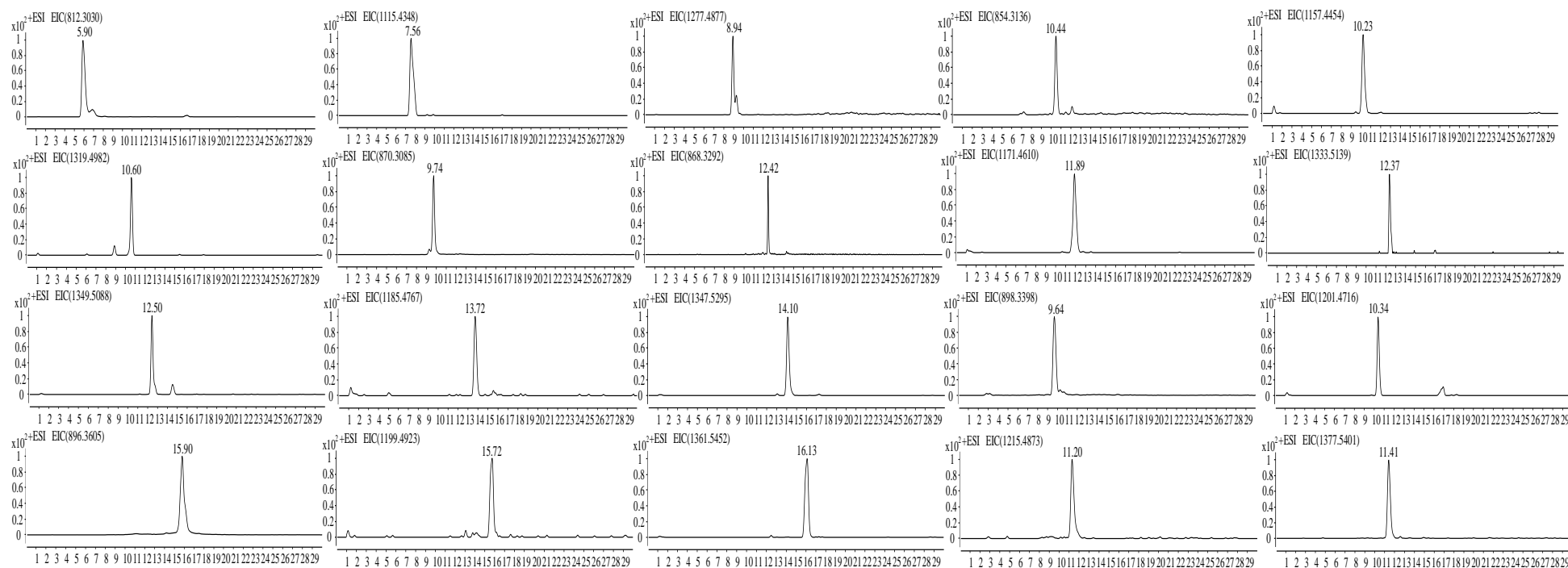


**Figure S6.** The EIC of acarviostatins with glucose at the reducing terminus from *Streptomyces* sp. HO1518.

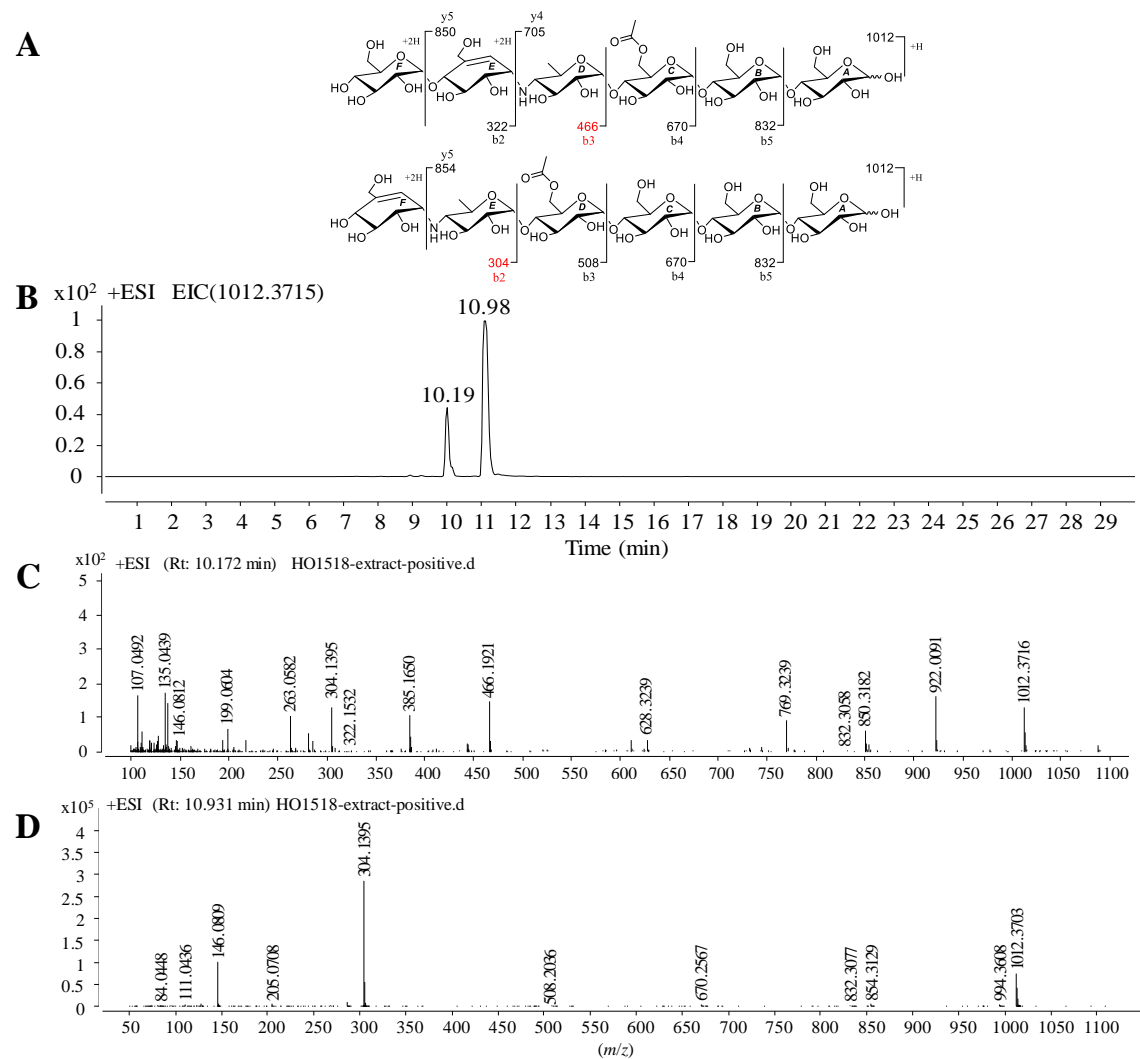


**Figure S7.** The EIC of acarviostatins with glucose at the reducing and nonreducing terminus from *Streptomyces* sp. HO1518.

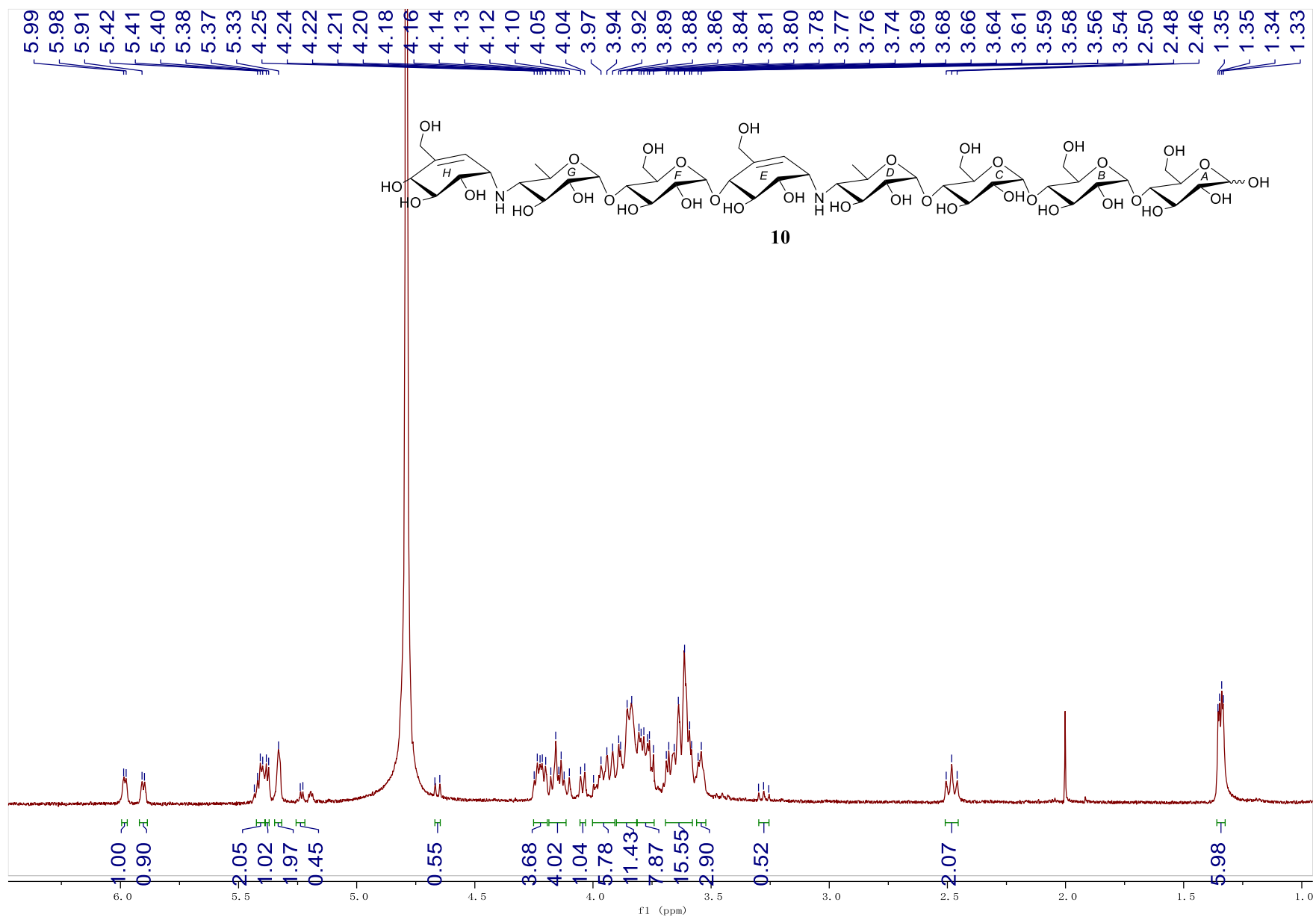




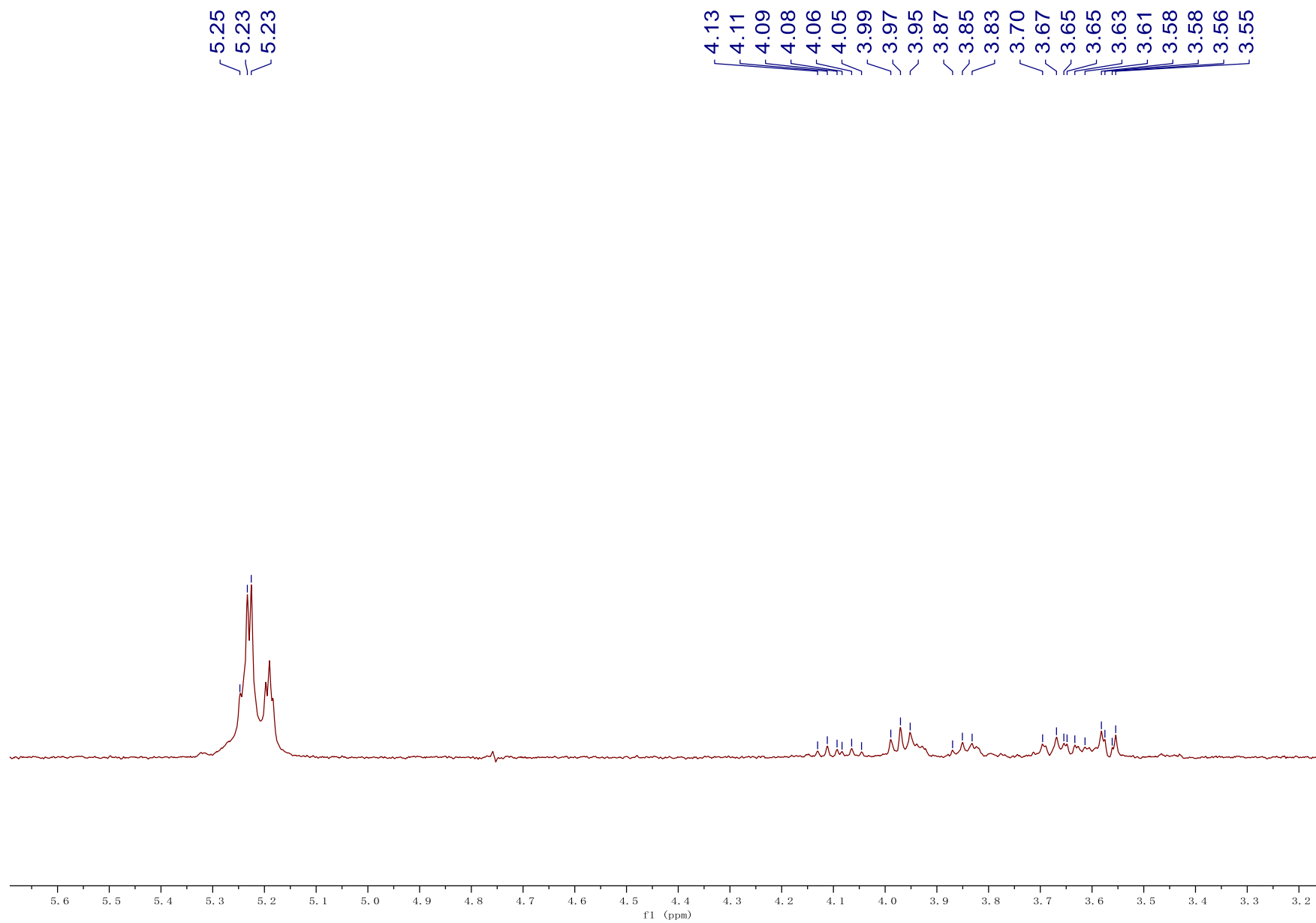
**Figure S8.** The EIC of acarviostatins with an incomplete *pseudo*-tetrasaccharide at the nonreducing terminus from *Streptomyces* sp. HO1518.



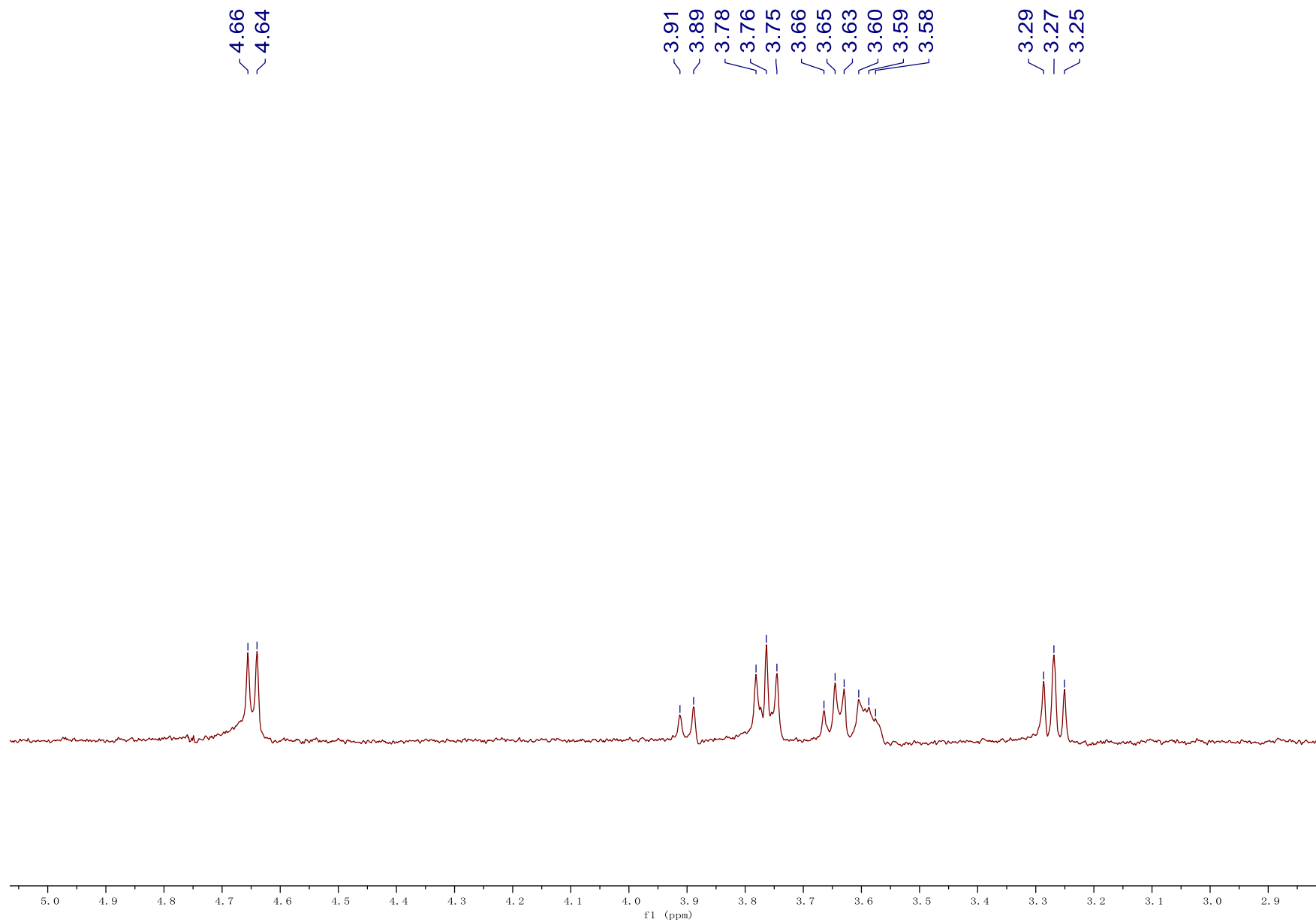
**Figure S9.** Positive HRESIMS/MS fragmentation and spectra of Ac-Aca I12 and Ac-Aca I03. (A) Positive-ion HRESIMS/MS fragmentation patterns of Ac-Aca I12 and Ac-Aca I03; (B) The extracted ion chromatogram of aminooligosaccharides at  $m/z$  1012; (C) HRESIMS/MS spectrum of Ac-Aca I12; (D) HRESIMS/MS spectrum of Ac-Aca I03.



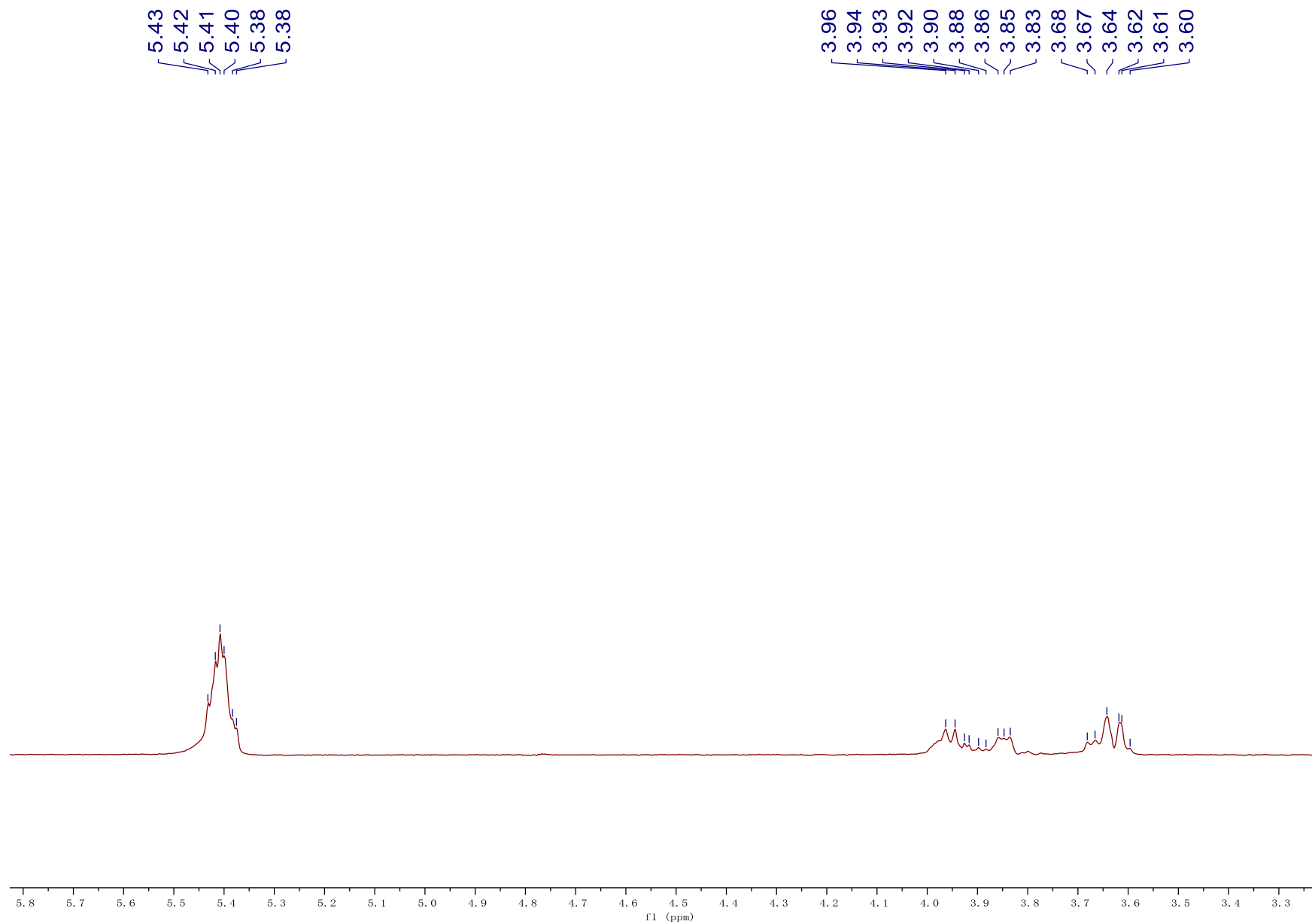
**Figure S10.**  $^1\text{H}$  NMR spectrum of compound **10** (500 MHz,  $\text{D}_2\text{O}$ ).



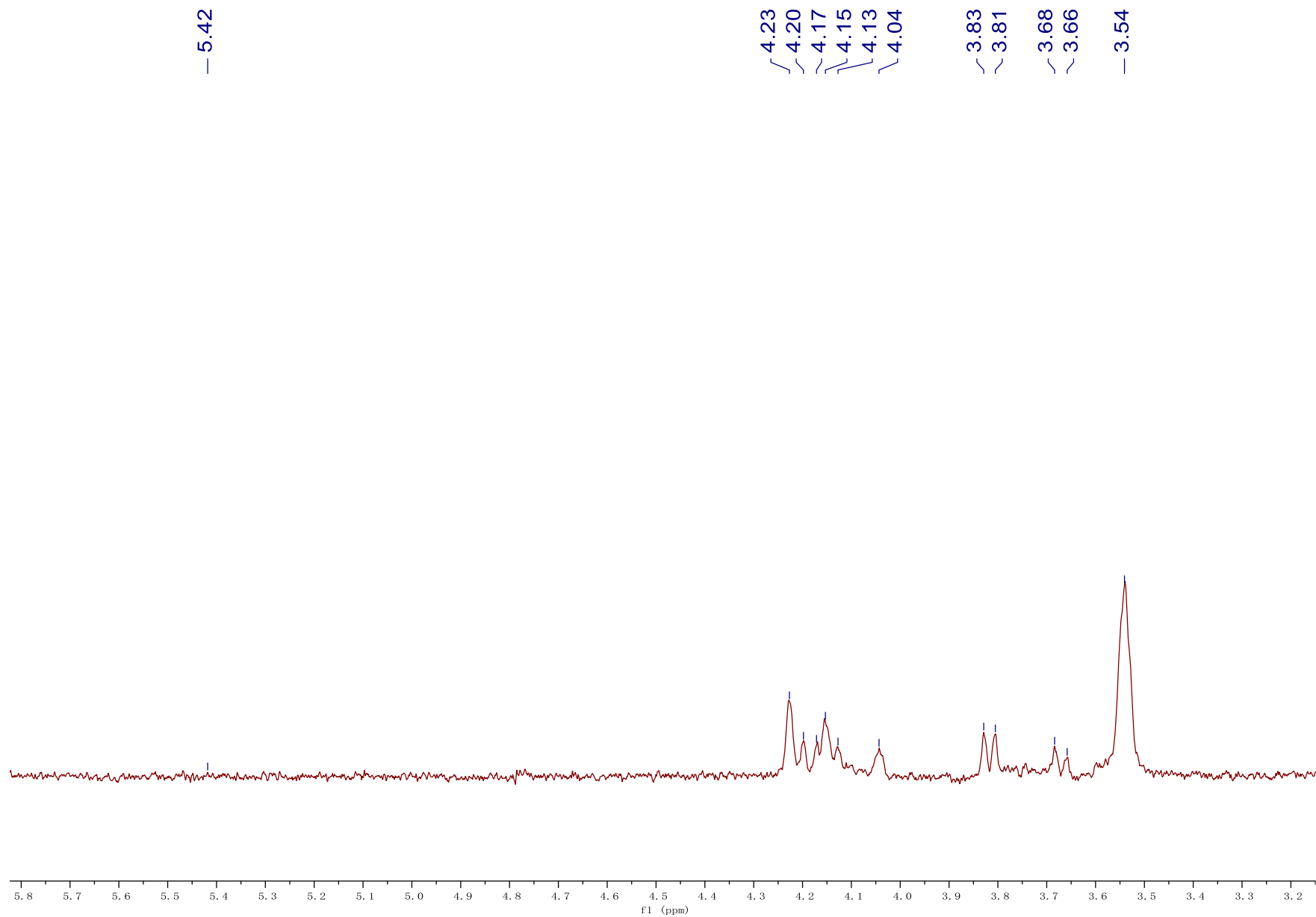
**Figure S11.** 1D-selective TOCSY spectrum of compound **10** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.23, H-A1 $\alpha$ ).



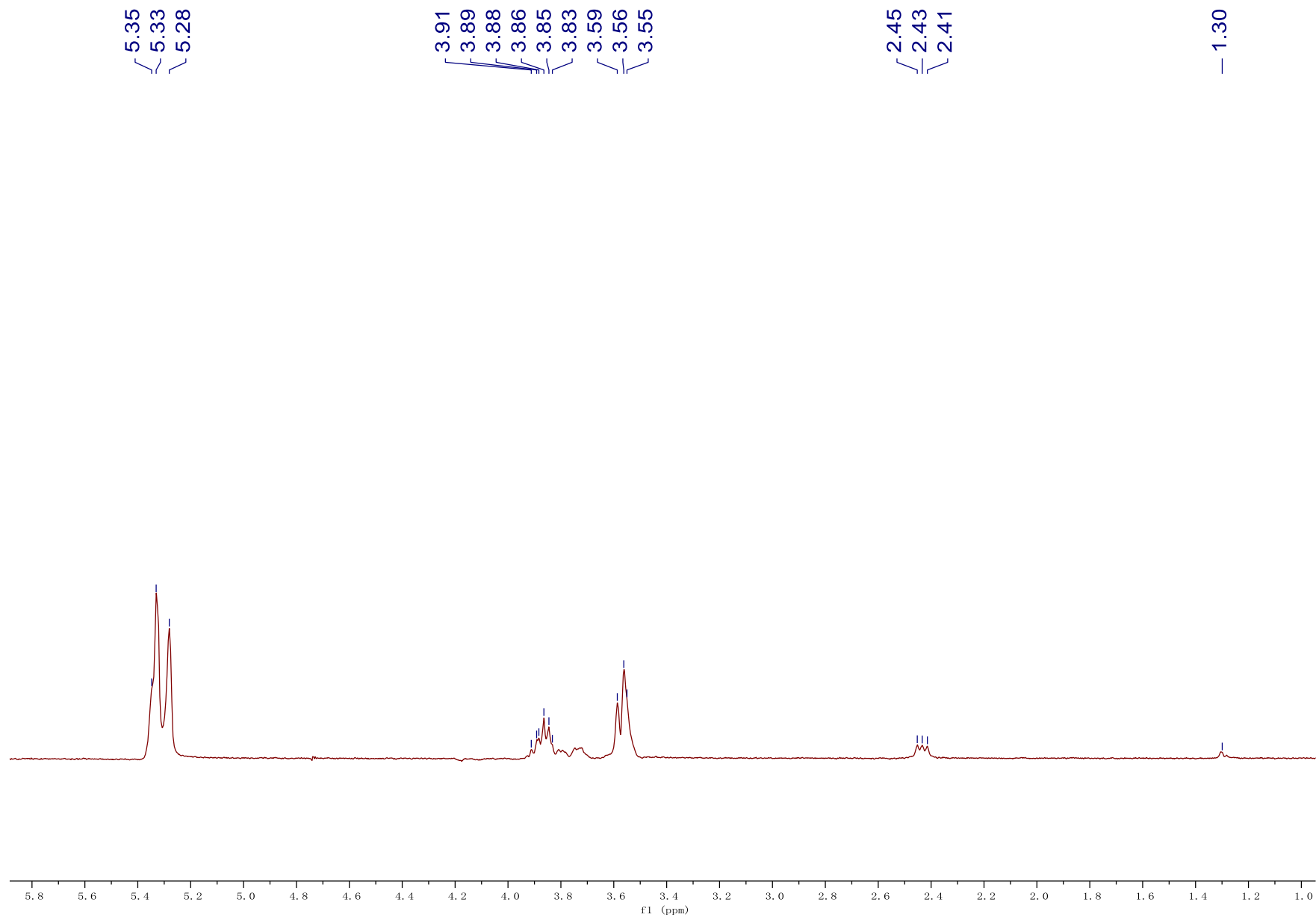
**Figure S12.** 1D-selective TOCSY spectrum of compound **10** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.65, H-A1 $\beta$ ).



**Figure S13.** 1D-selective TOCSY spectrum of compound **10** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.41, H-**B1**, and H-**C1**).

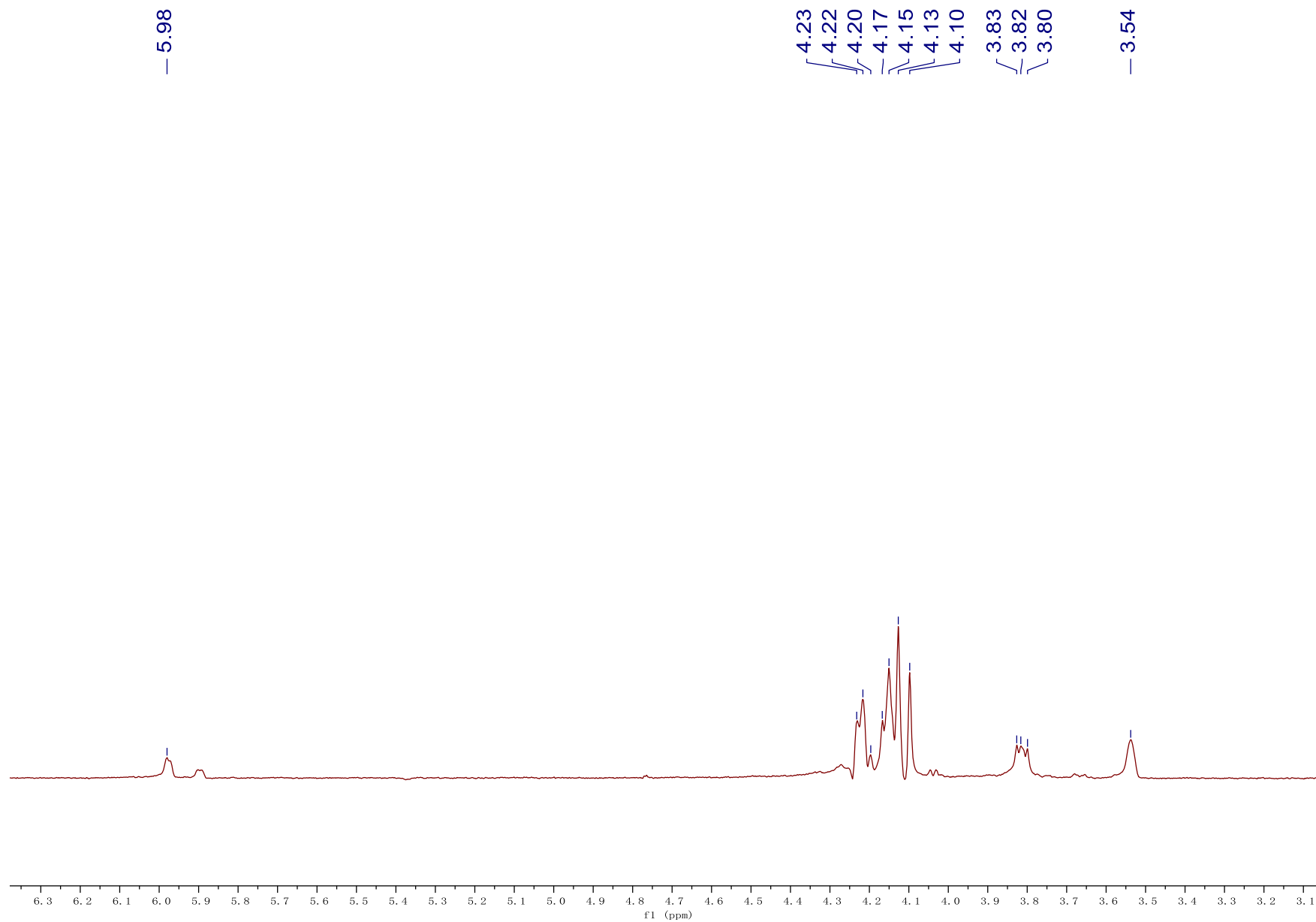


**Figure S14.** 1D-selective TOCSY spectrum of compound **10** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.22, H-C6).

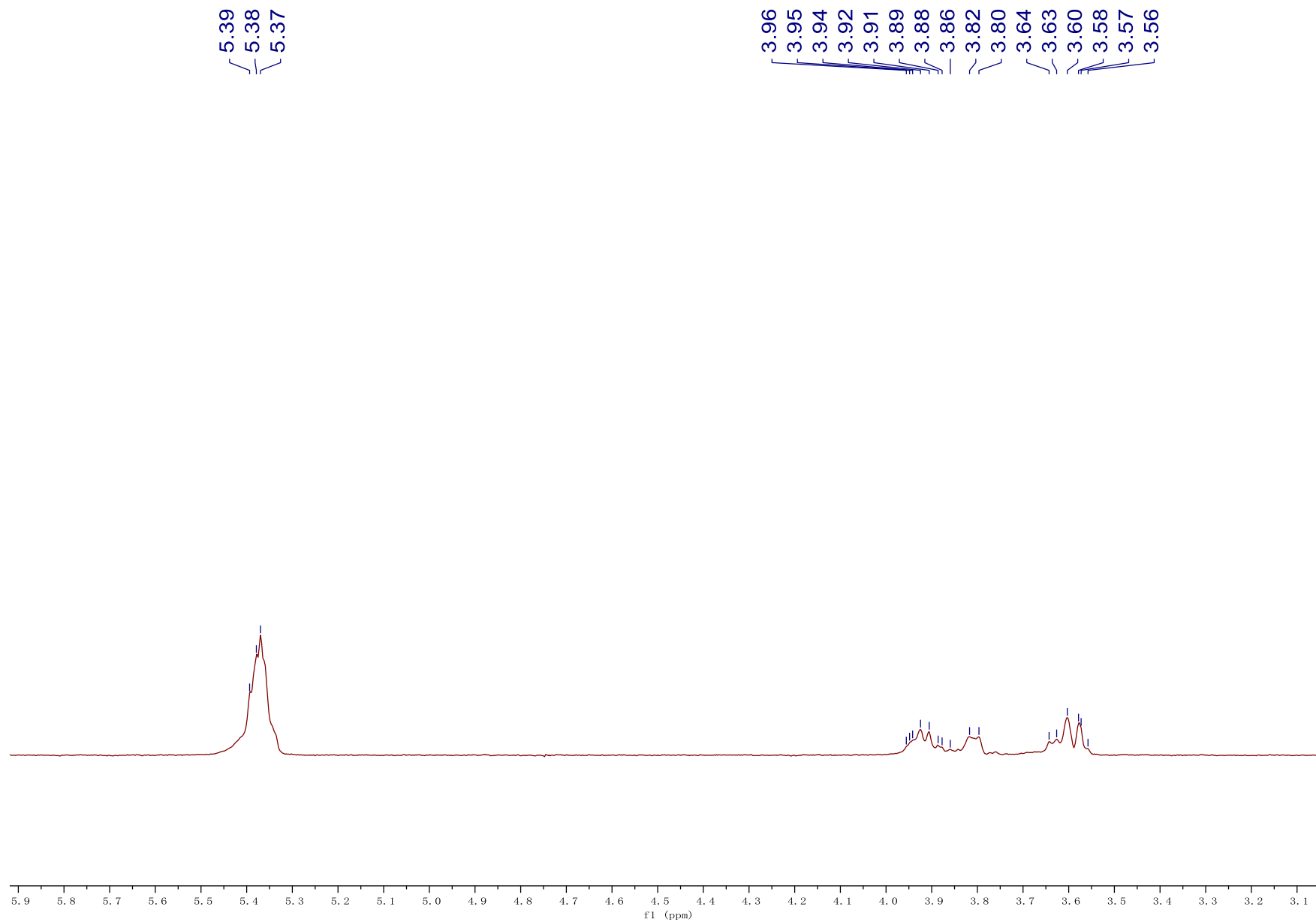


**Figure S15.** 1D-selective TOCSY spectrum of compound **10** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.33, H-**D1** and H-**G1**).

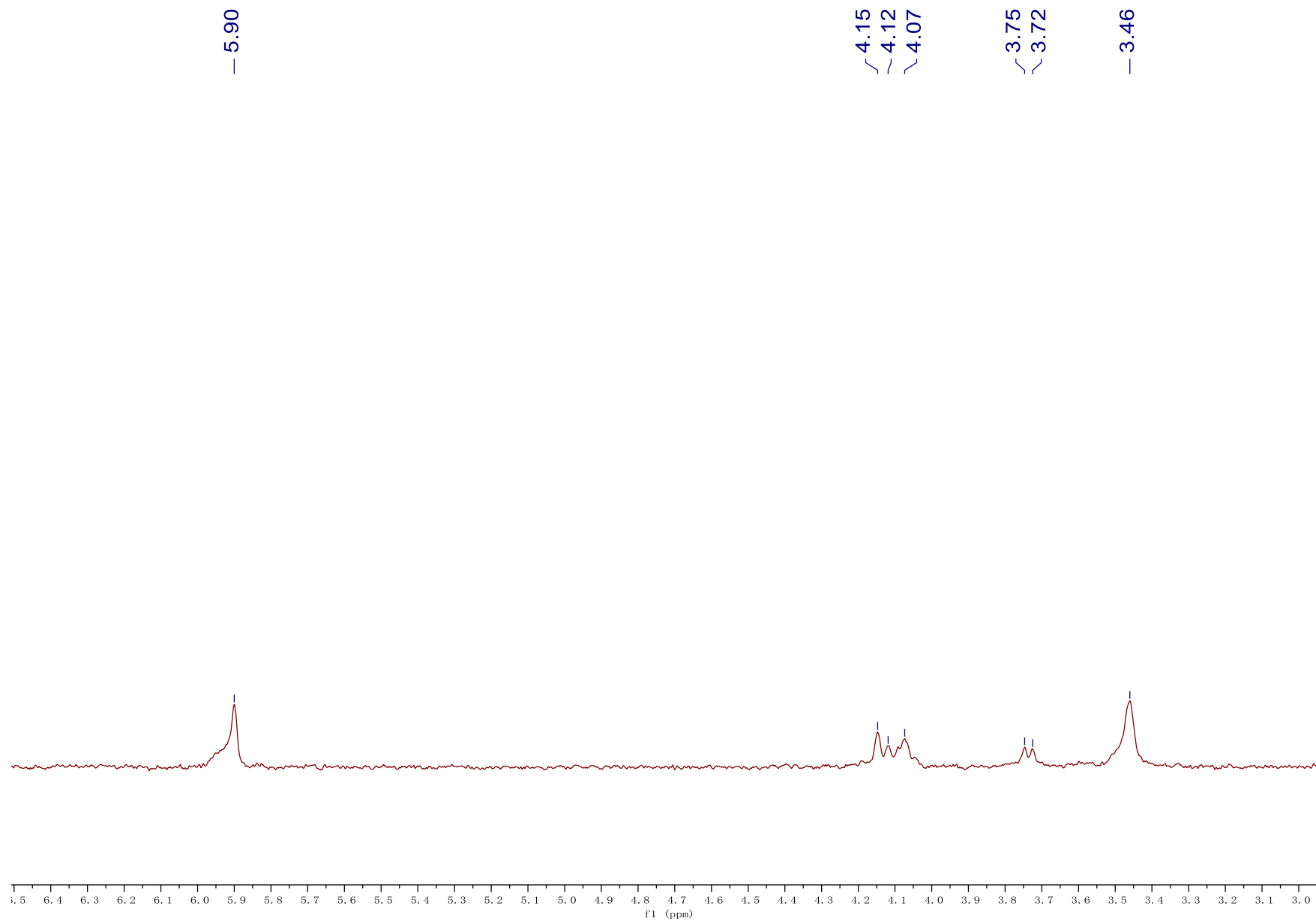




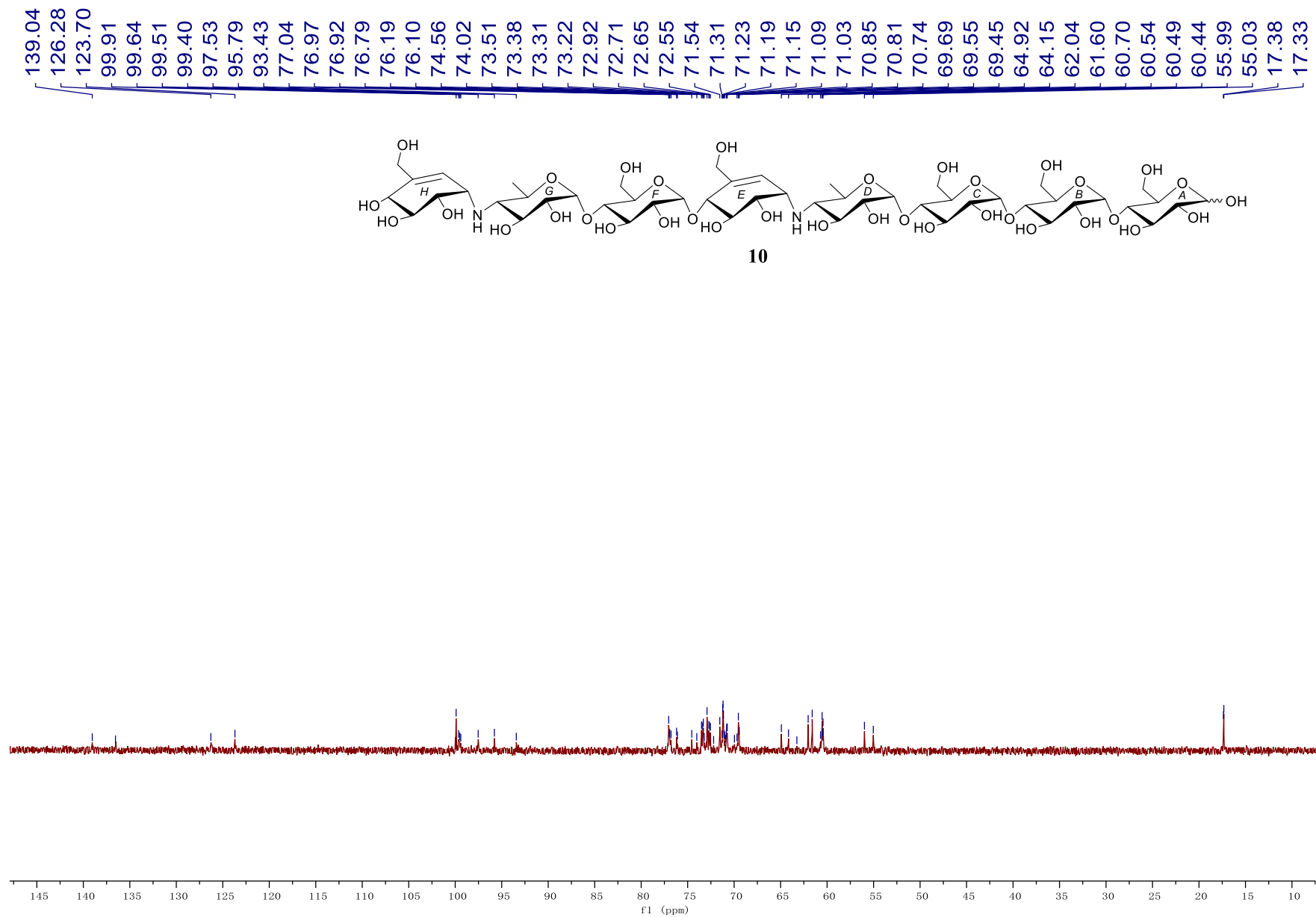
**Figure S16.** 1D-selective TOCSY spectrum of compound **10** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.98, H-E7).



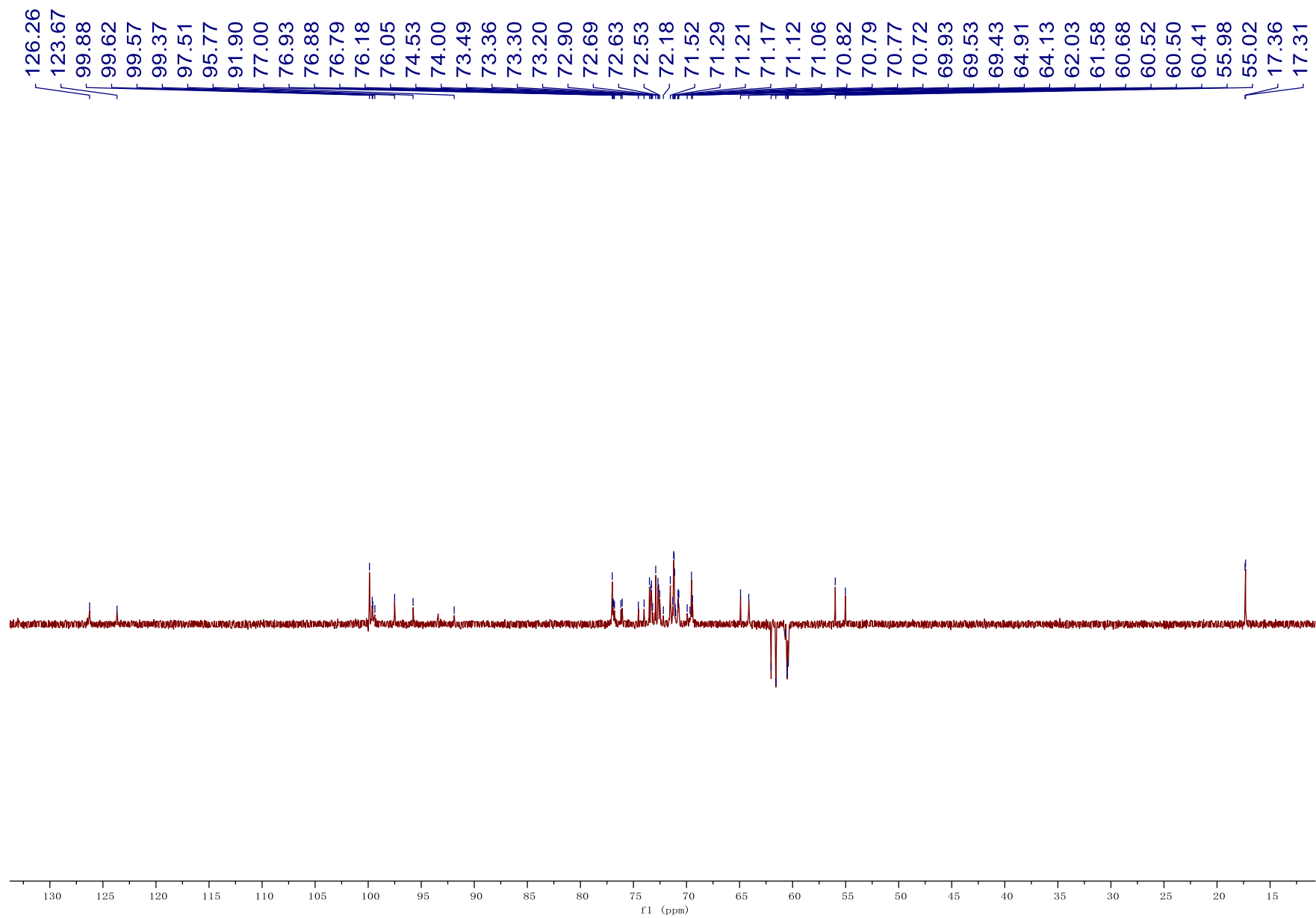
**Figure S17.** 1D-selective TOCSY spectrum of compound **10** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.38, H-F1).



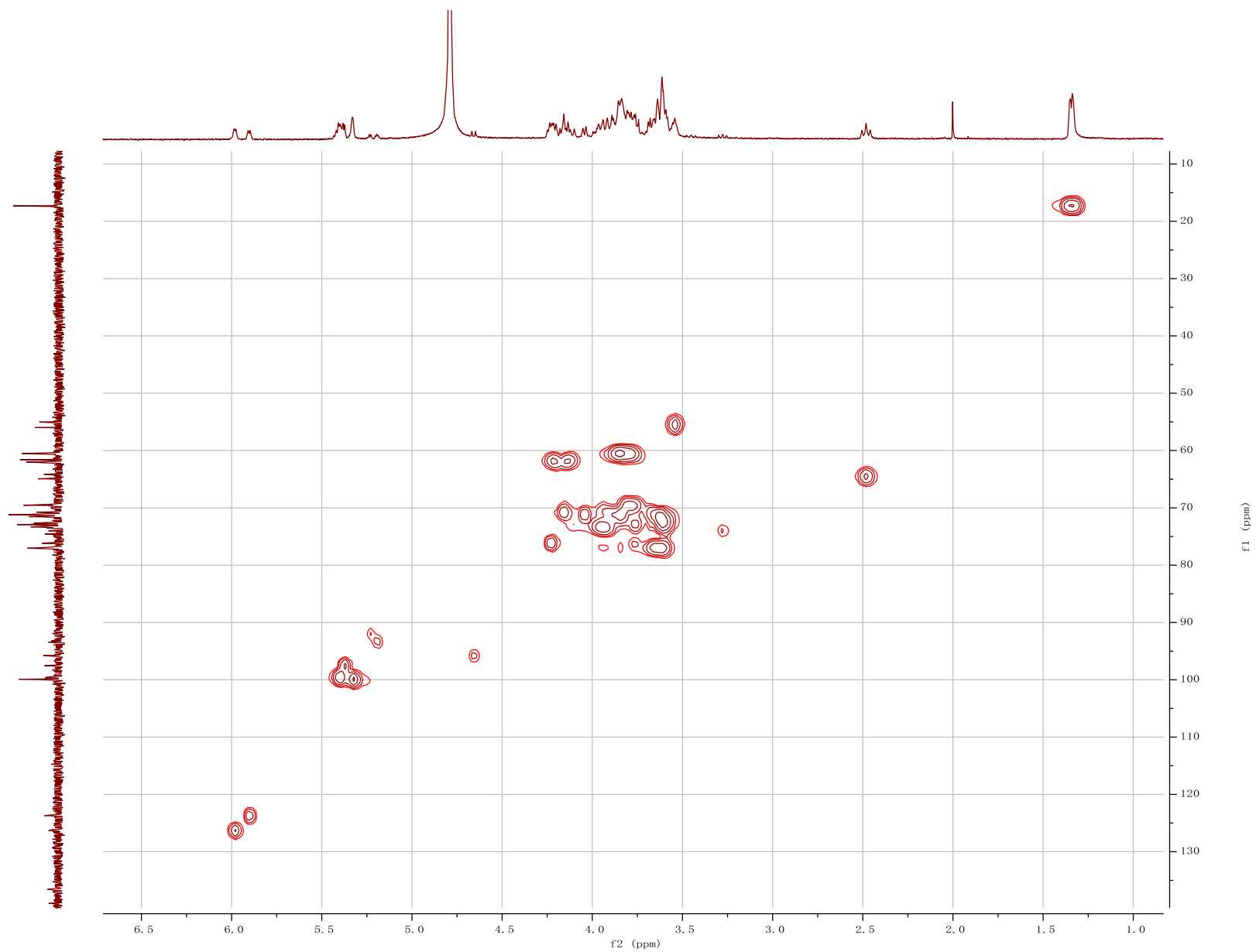
**Figure S18.** 1D-selective TOCSY spectrum of compound **10** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.90, H-**H**7).



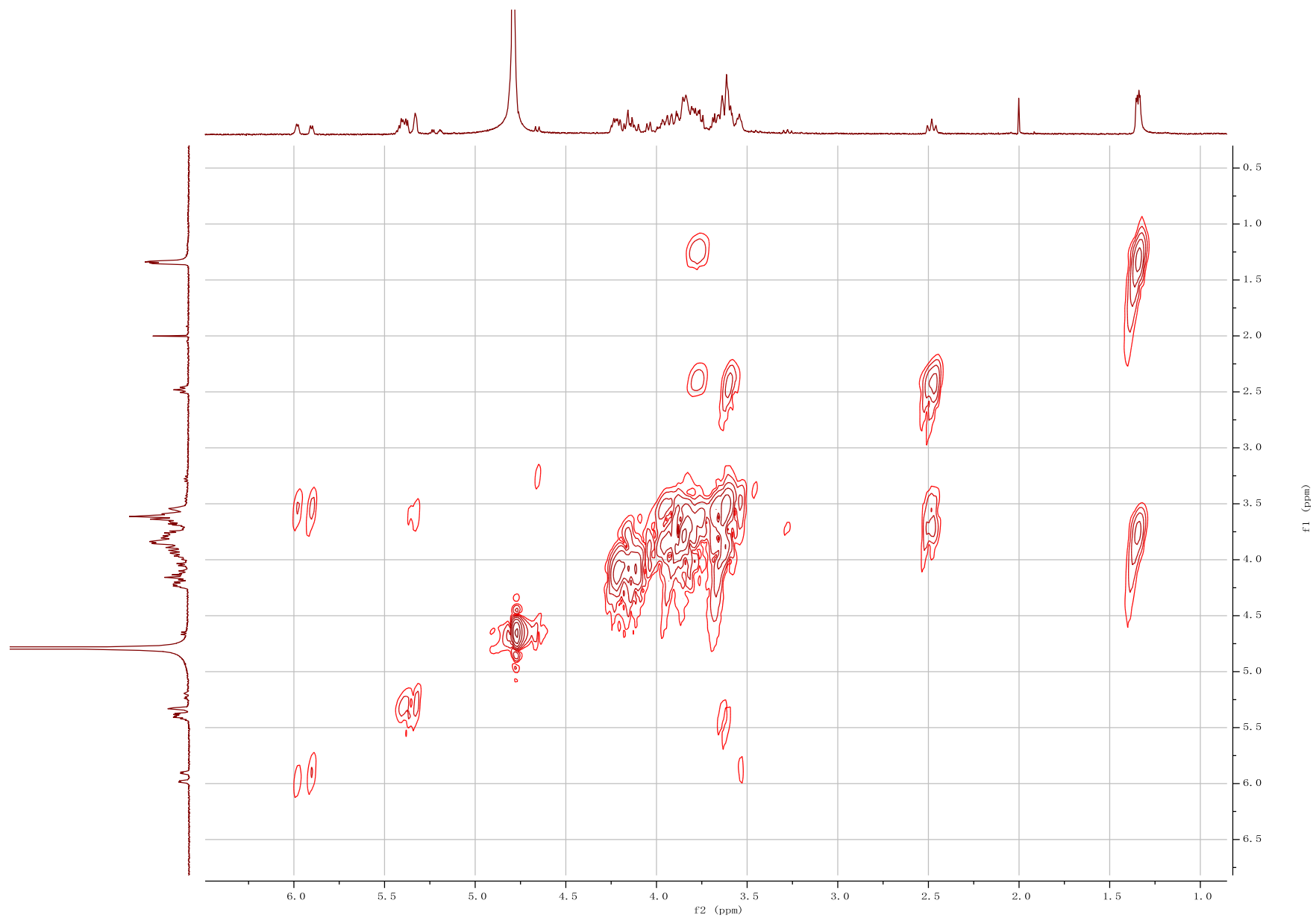
**Figure S19.**  $^{13}\text{C}$  NMR spectrum of compound **10** (125 MHz,  $\text{D}_2\text{O}$ ).



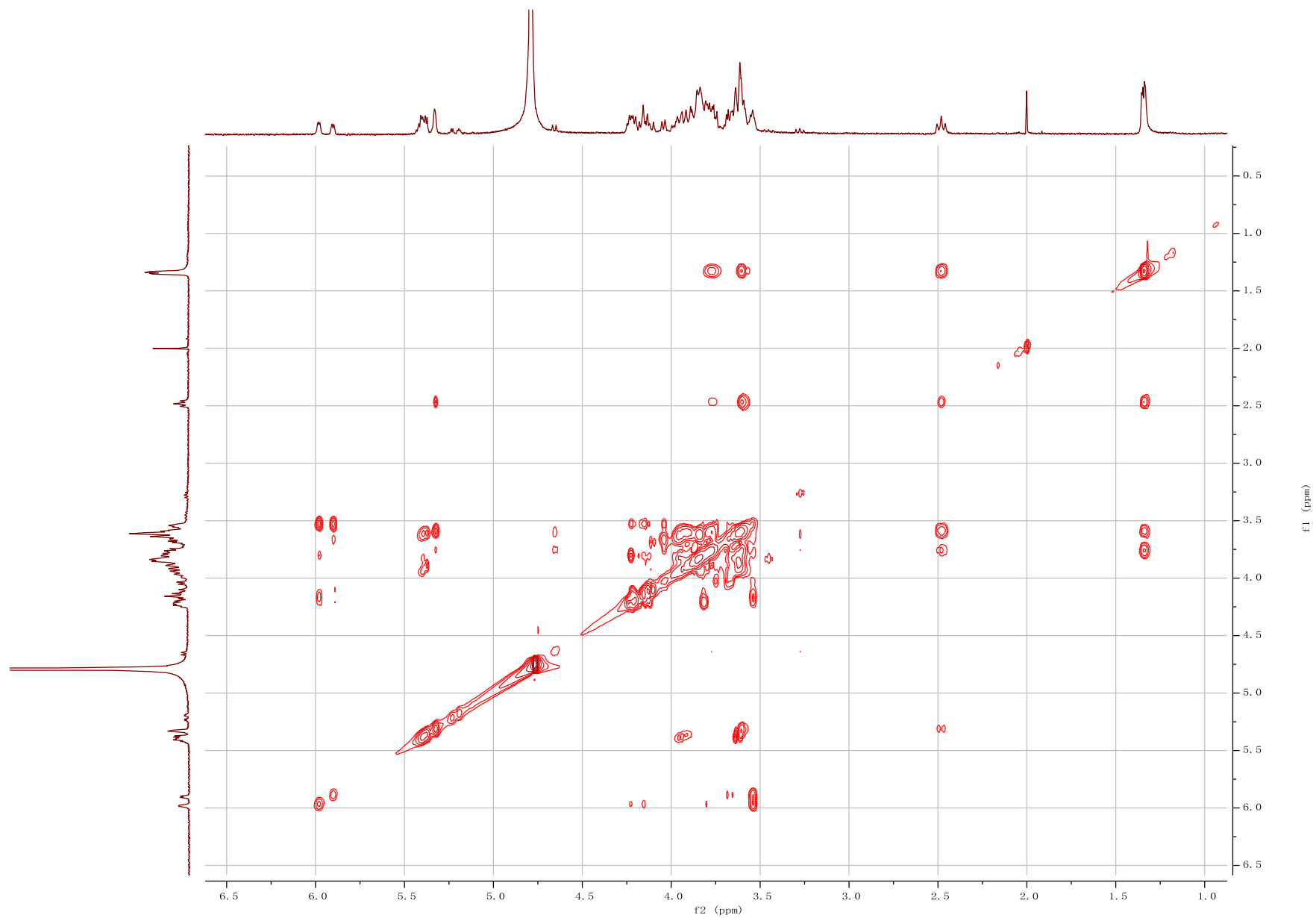
**Figure S20.** DEPT-135 spectrum of compound **10** (125 MHz, D<sub>2</sub>O).



**Figure S21.** HSQC spectrum of compound **10** (500 MHz, D<sub>2</sub>O).

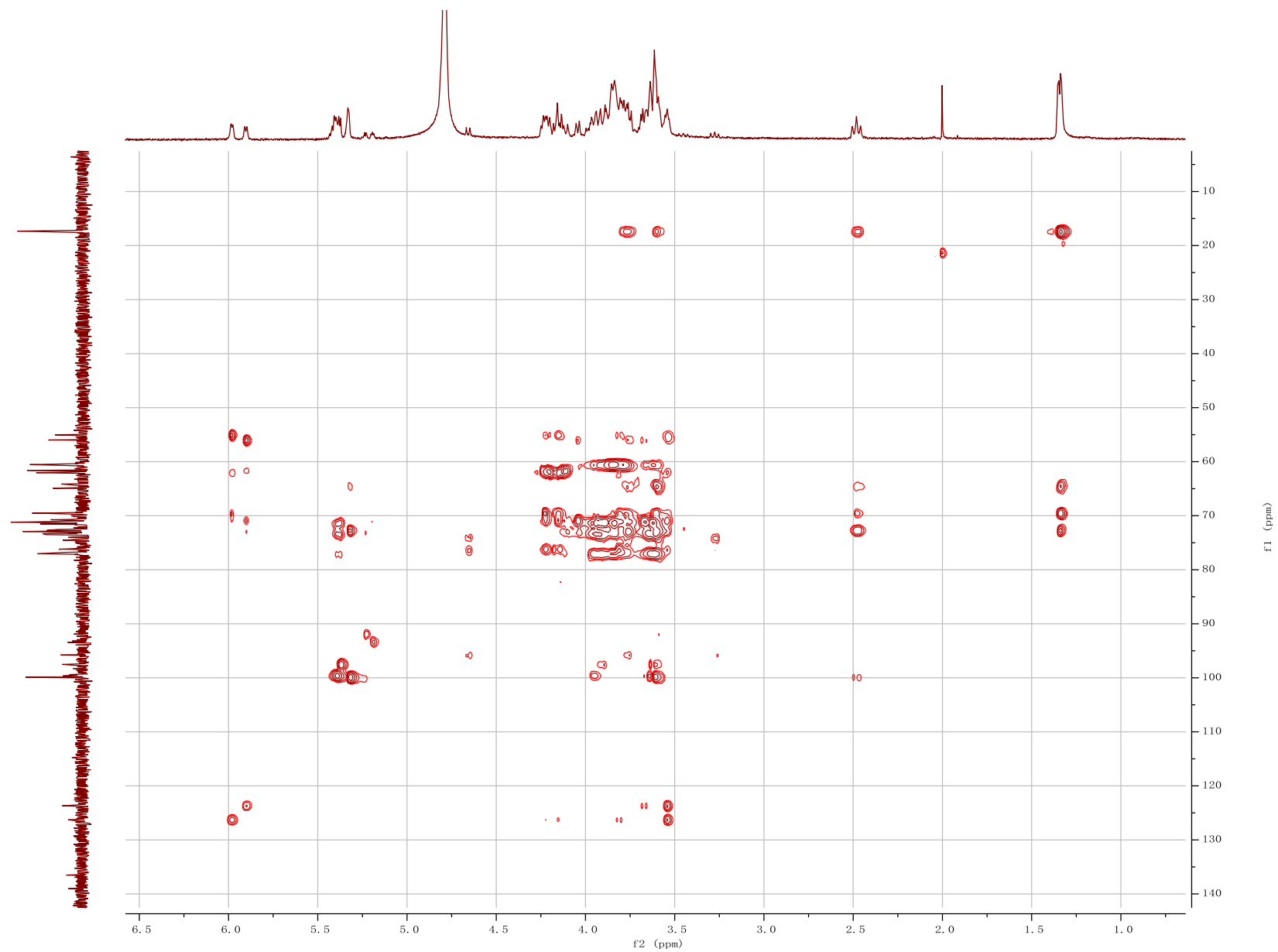


**Figure S22.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound **10** (500 MHz,  $\text{D}_2\text{O}$ ).

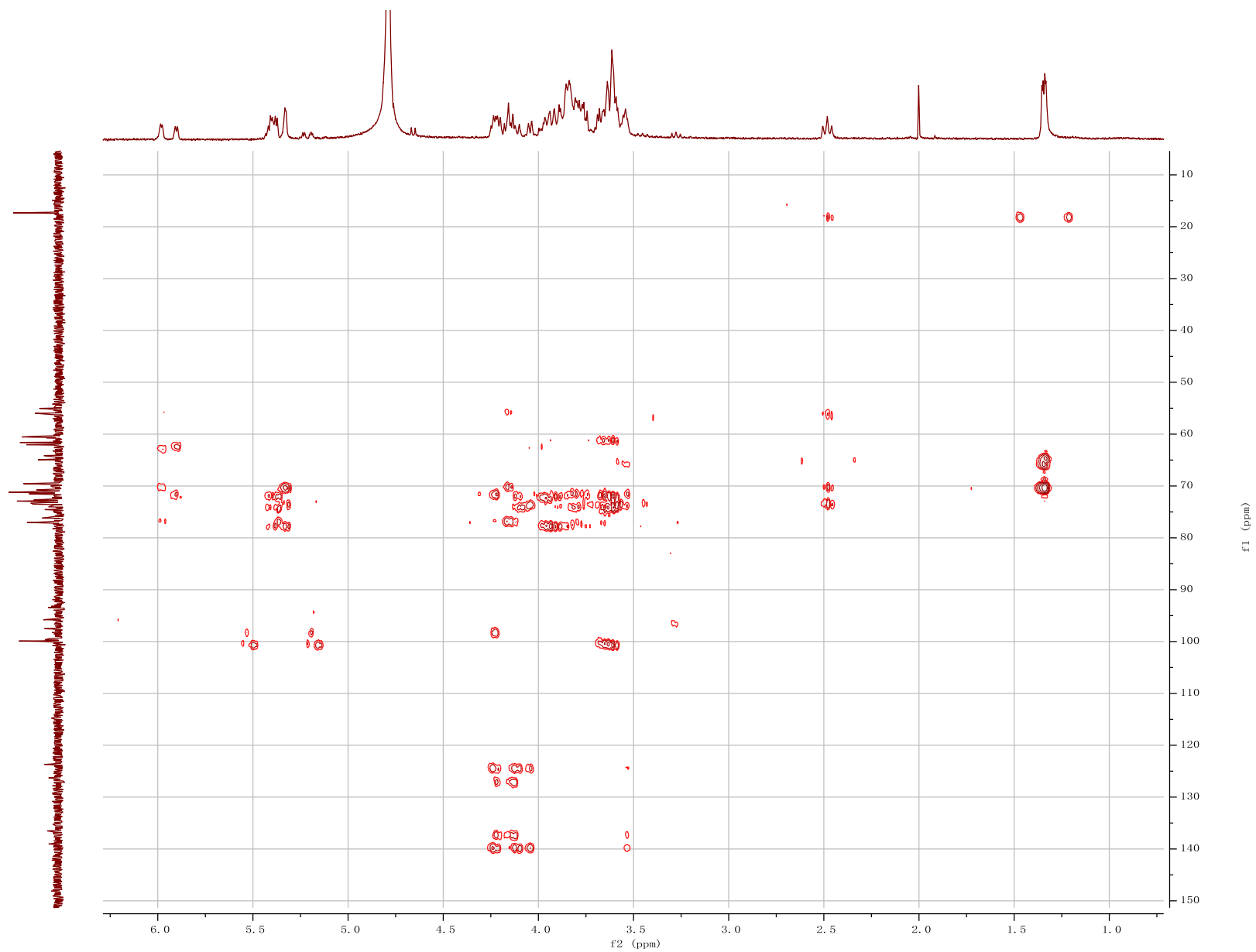


**Figure S23.** 2D-TOCSY spectrum of compound **10** (500 MHz, D<sub>2</sub>O).

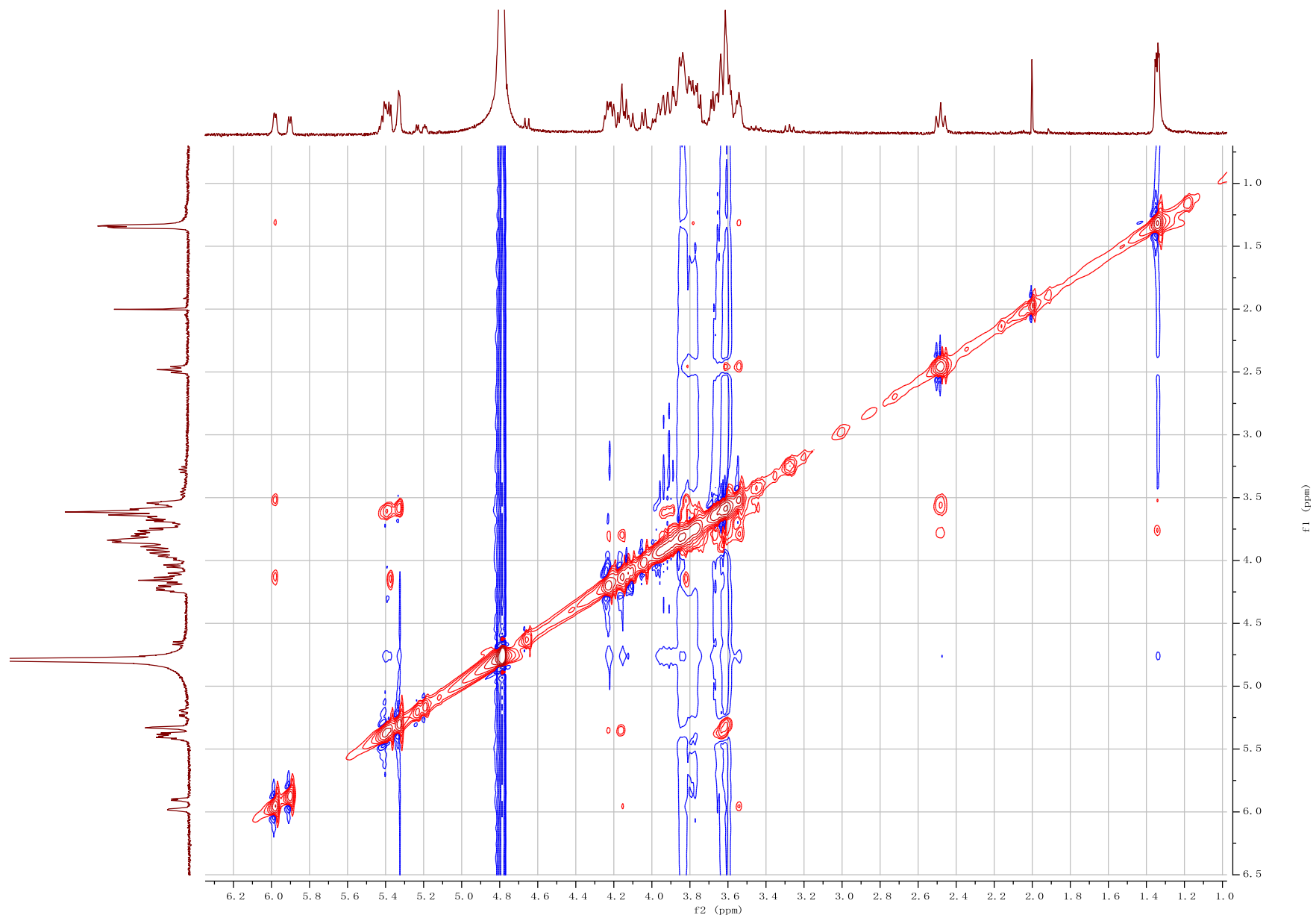




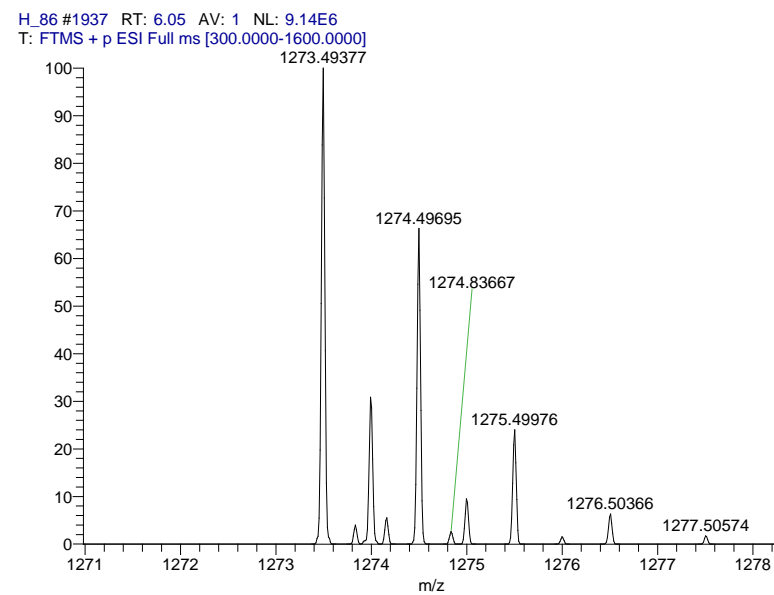
**Figure S24.** HSQC-TOCSY spectrum of compound **10** (500 MHz,  $\text{D}_2\text{O}$ ).



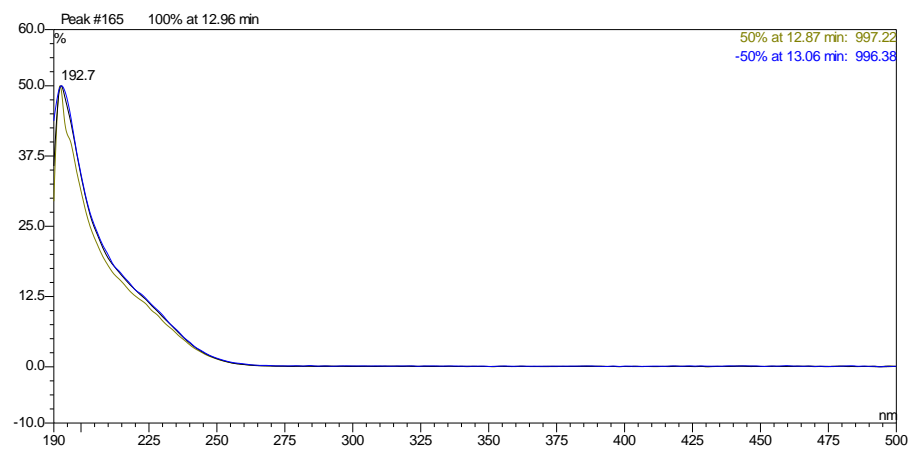
**Figure S25.** HMBC spectrum of compound **10** (500 MHz, D<sub>2</sub>O).



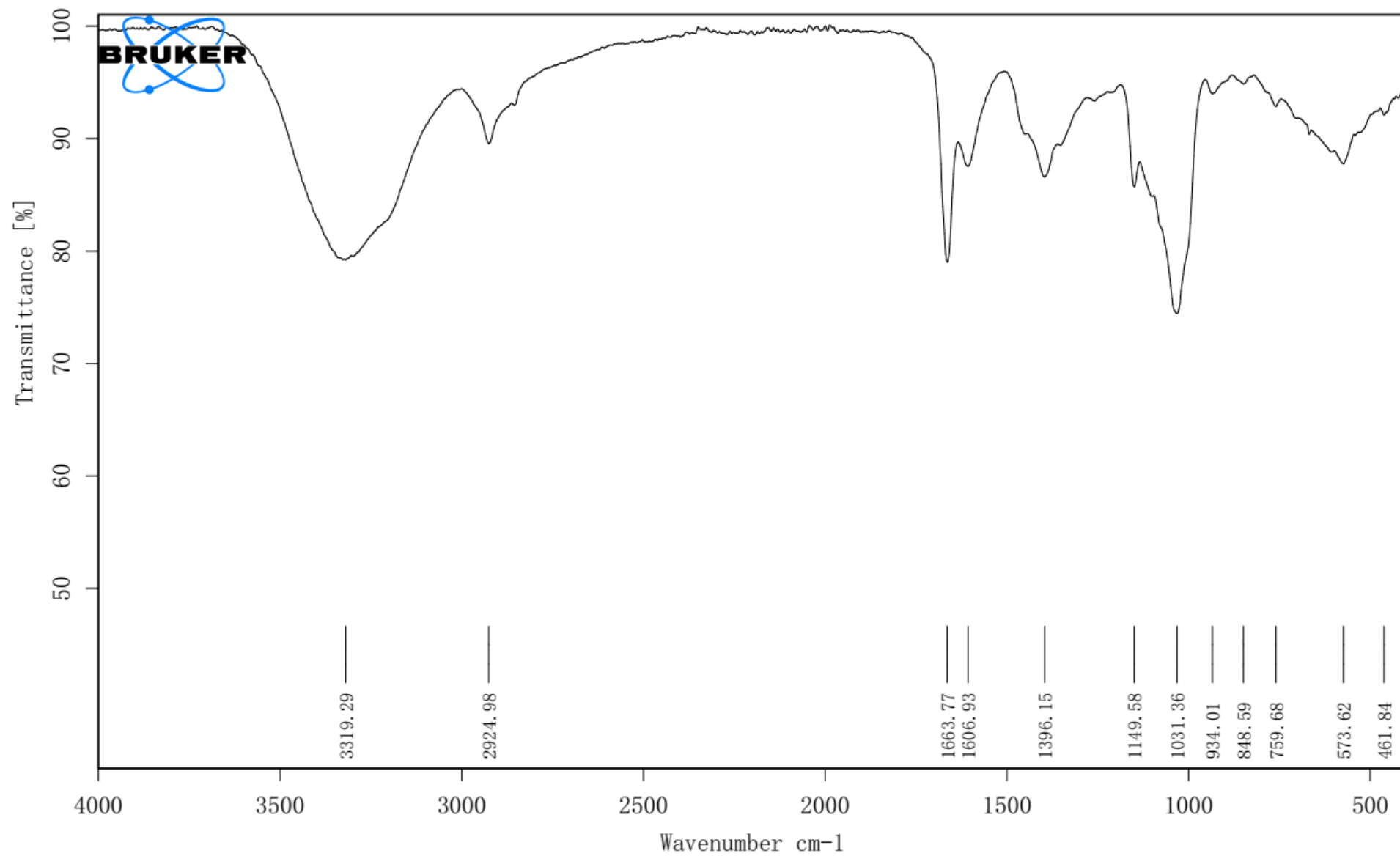
**Figure S26.** NOESY spectrum of compound **10** (500 MHz, D<sub>2</sub>O).



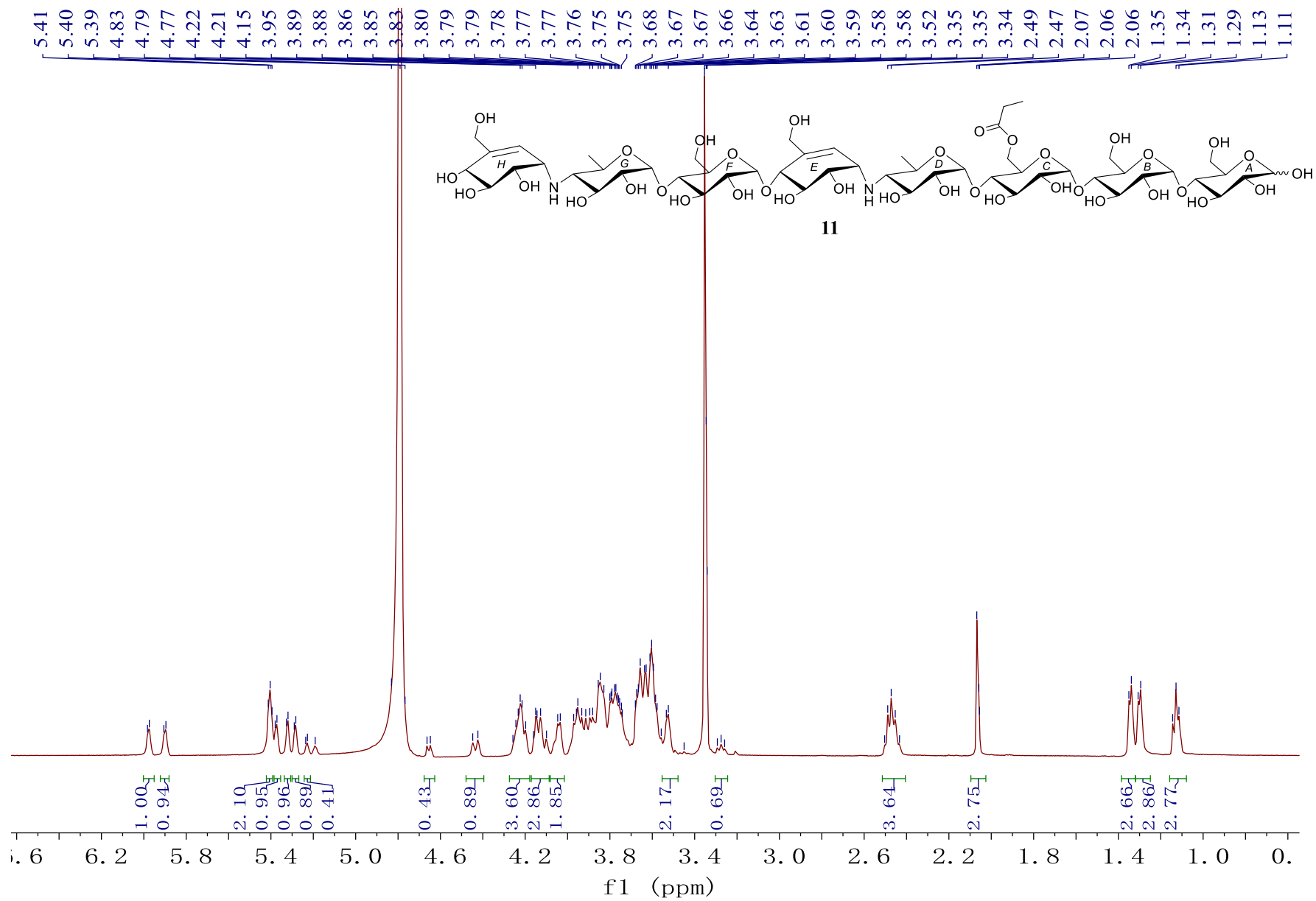
**Figure S27.** HRESIMS spectrum of compound **10**.



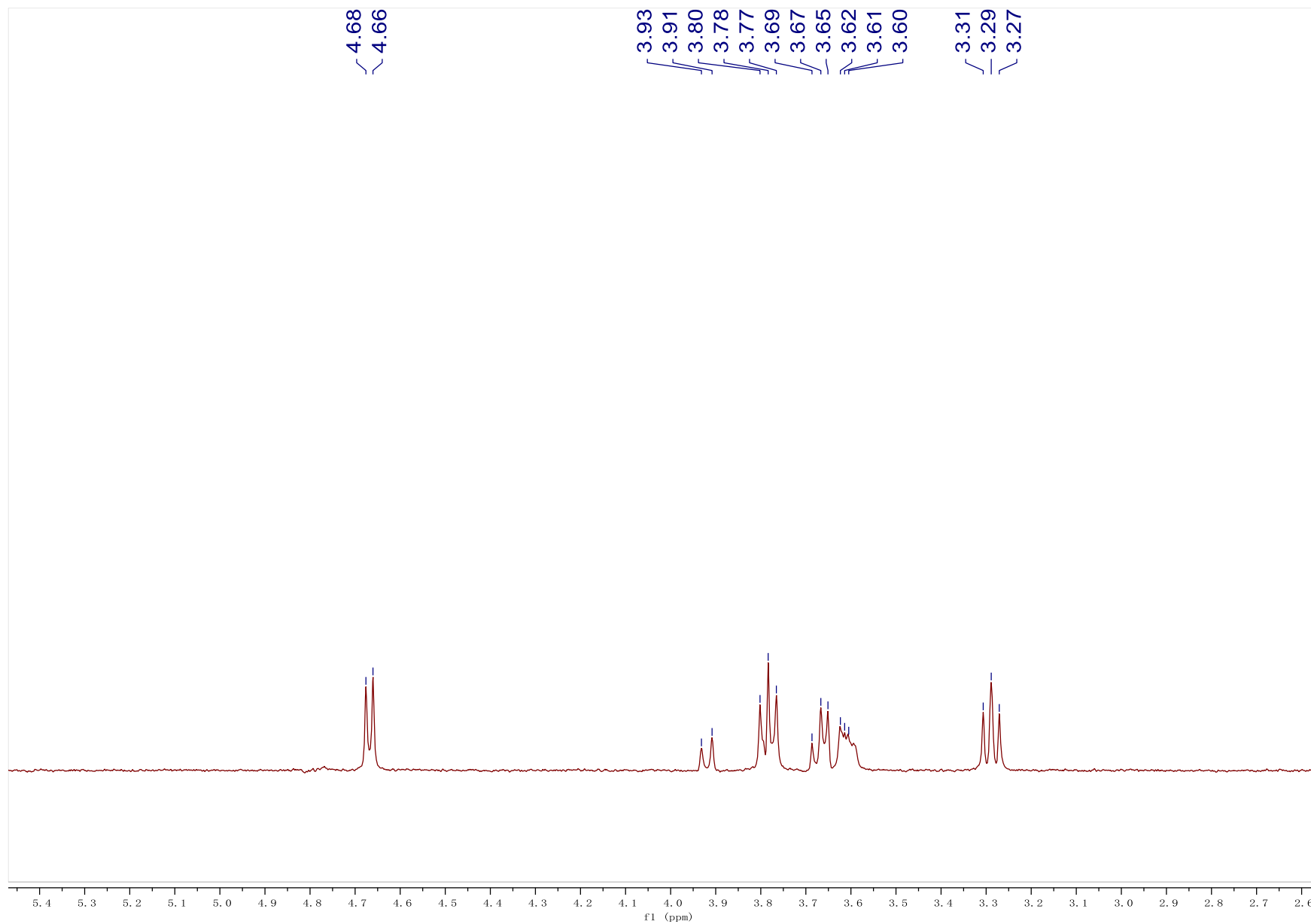
**Figure S28.** UV spectrum of compound **10**.



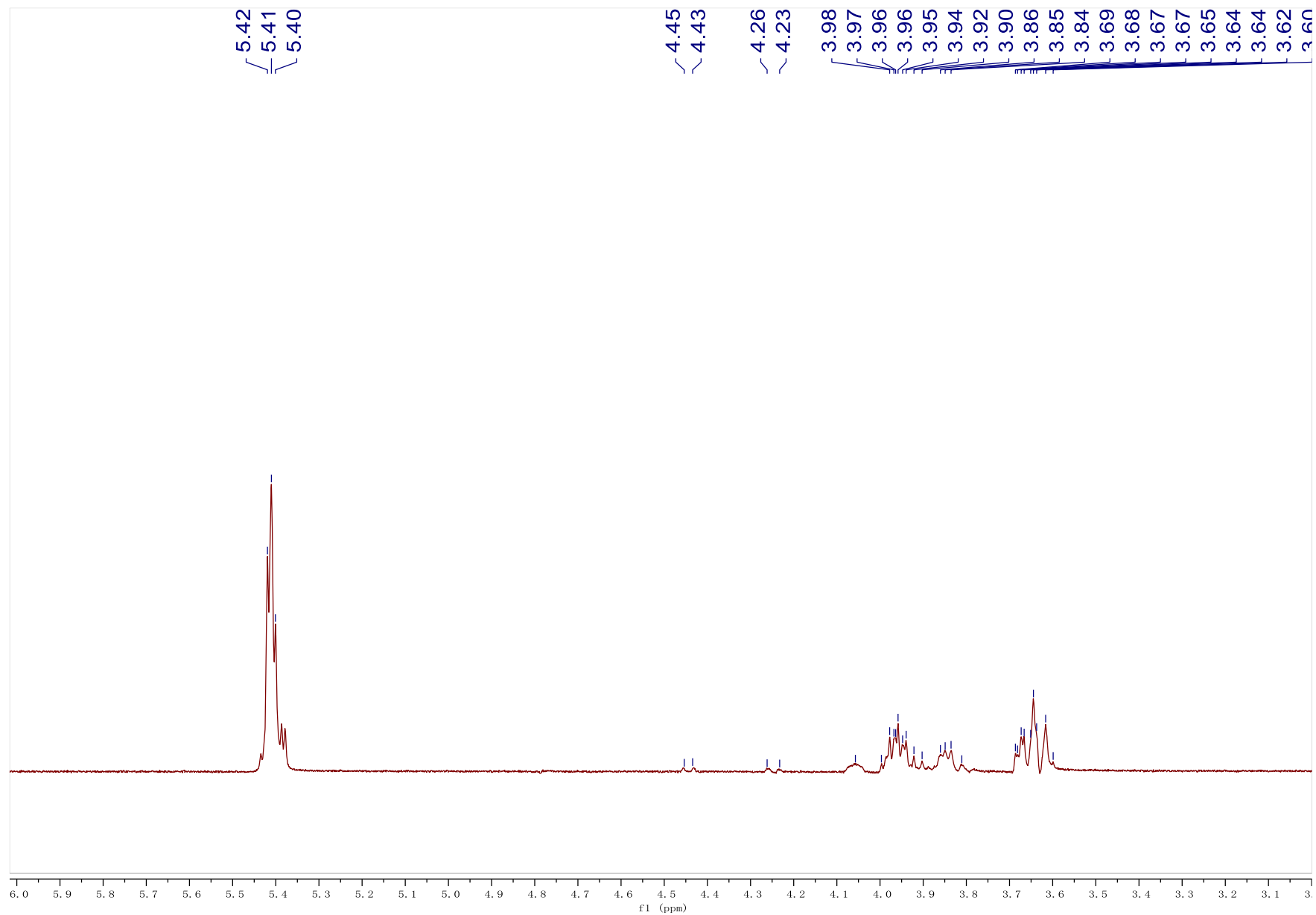
**Figure S29.** IR spectrum of compound **10**.



**Figure S30.**  $^1\text{H}$  NMR spectrum of compound **11** (500 MHz,  $\text{D}_2\text{O}$ ).

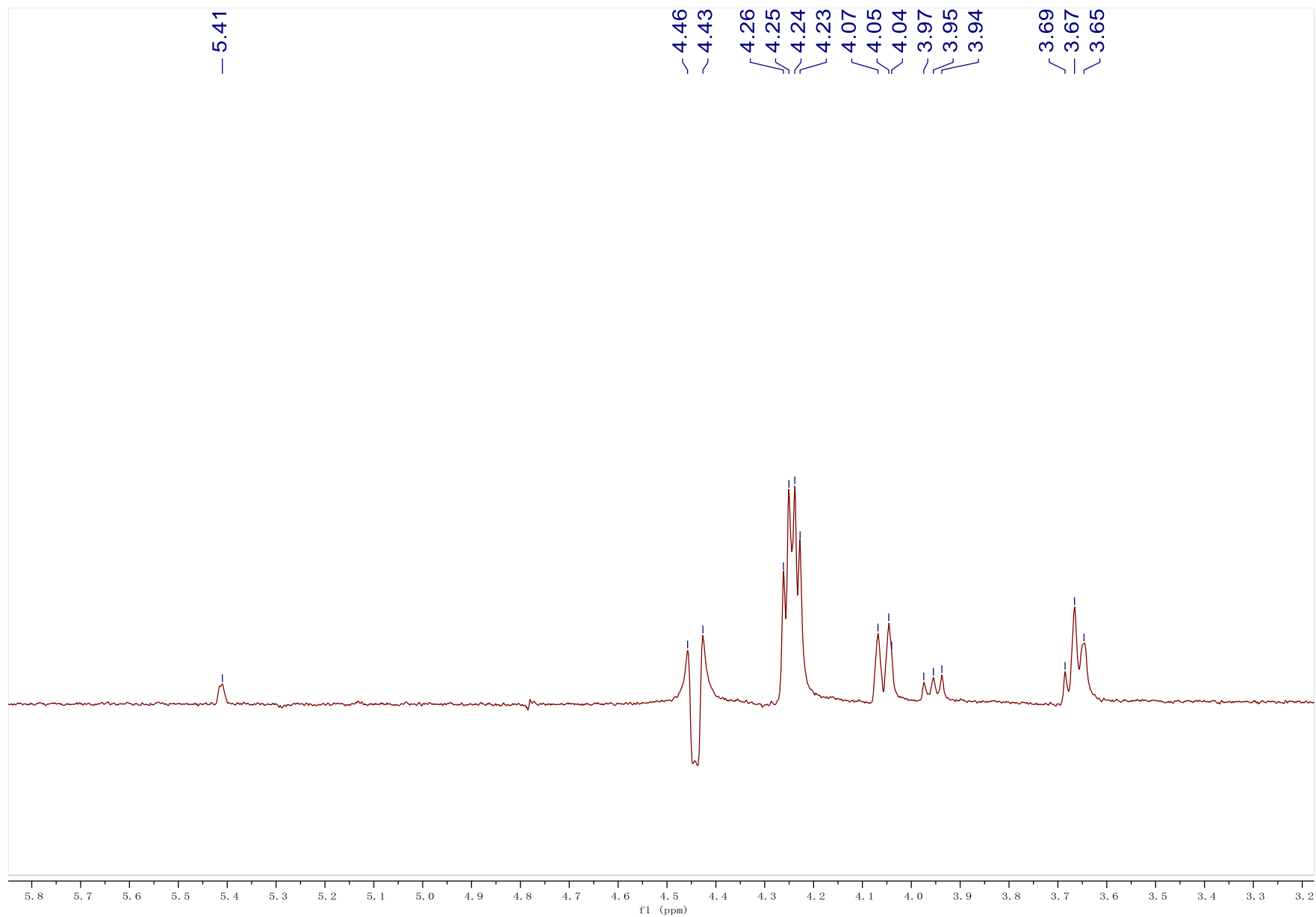


**Figure S31.** 1D-selective TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.66, H-A1 $\beta$ ).

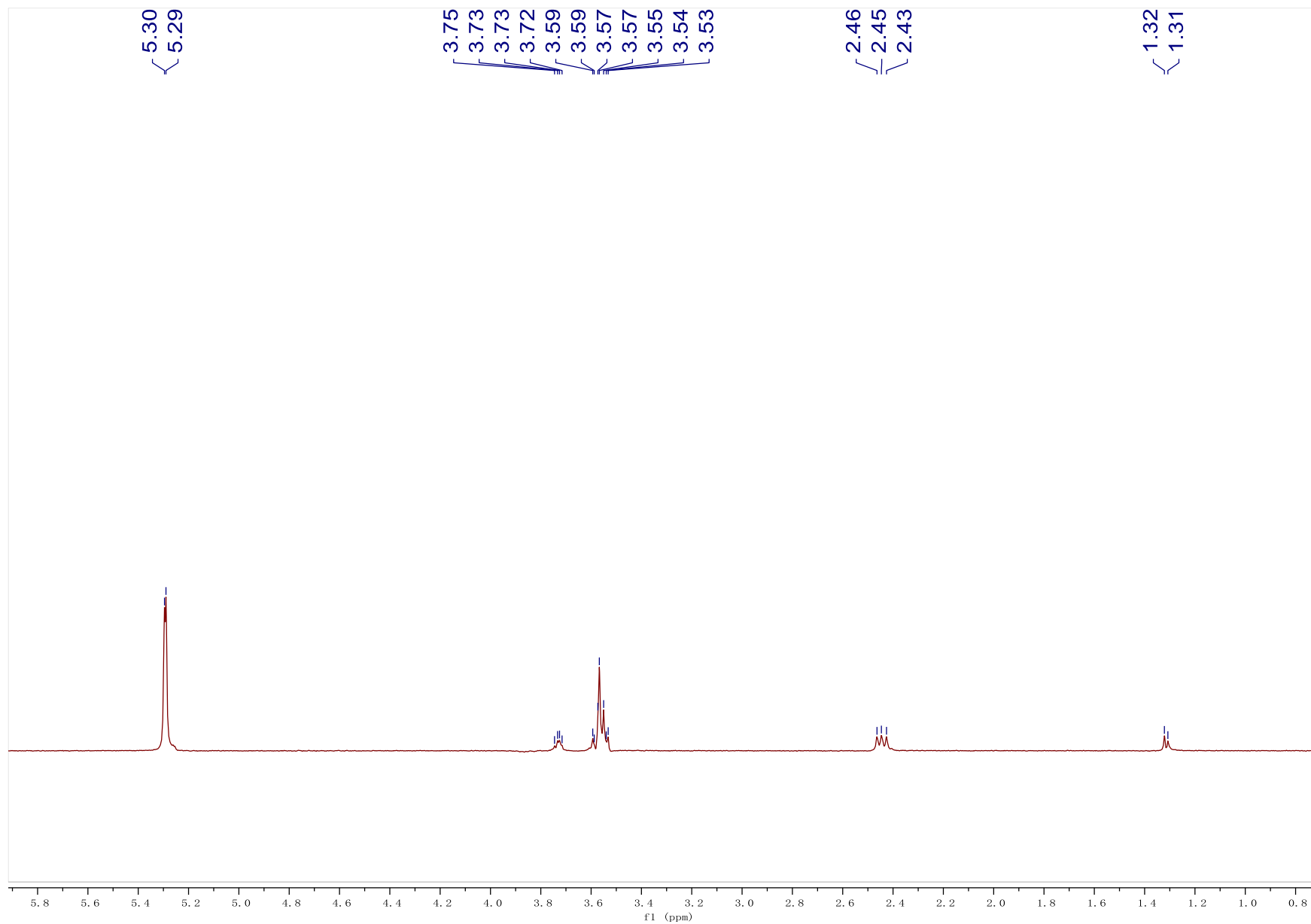


**Figure S32.** 1D-selective TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.41, H-**B1** and H-**C1**).

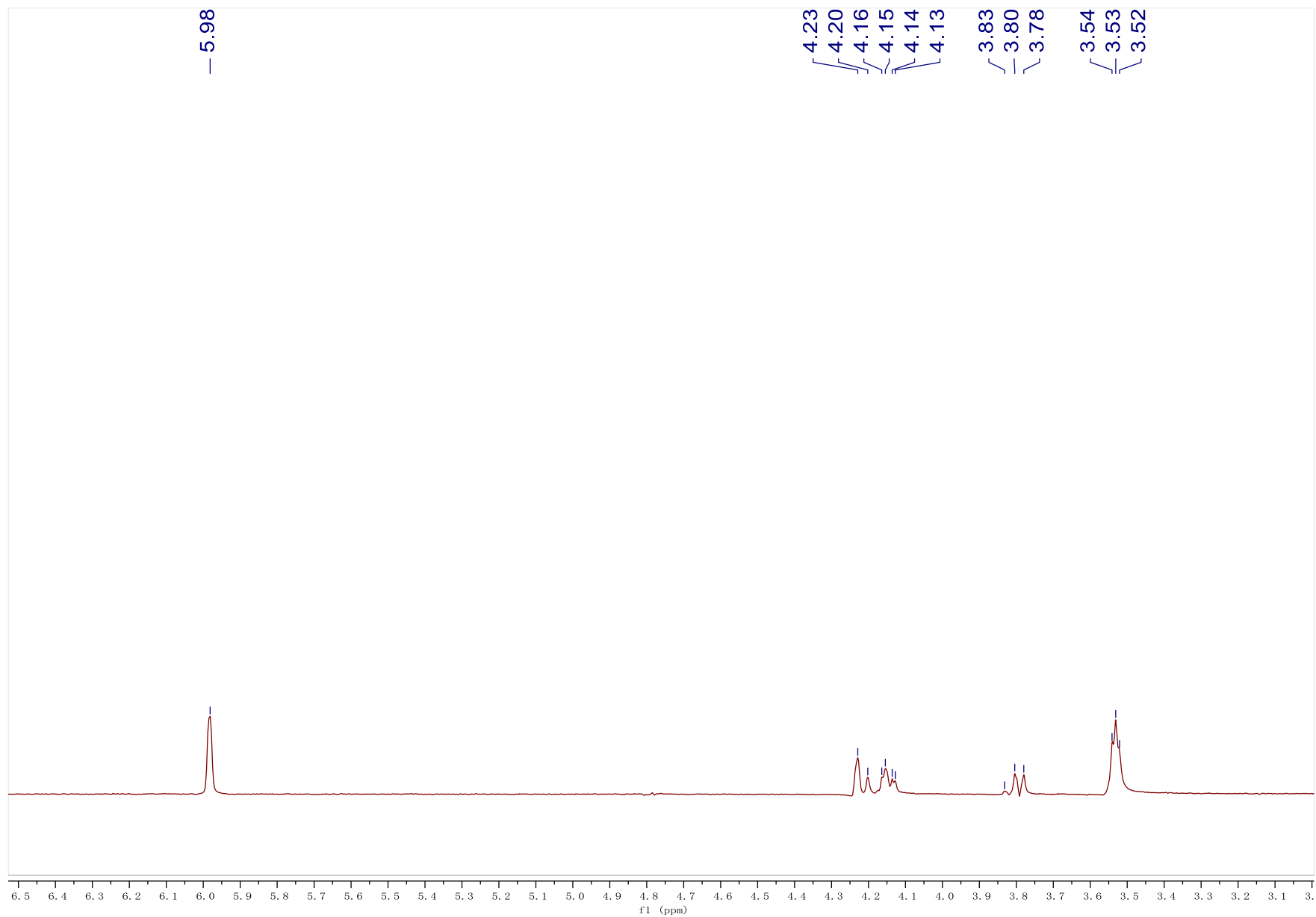




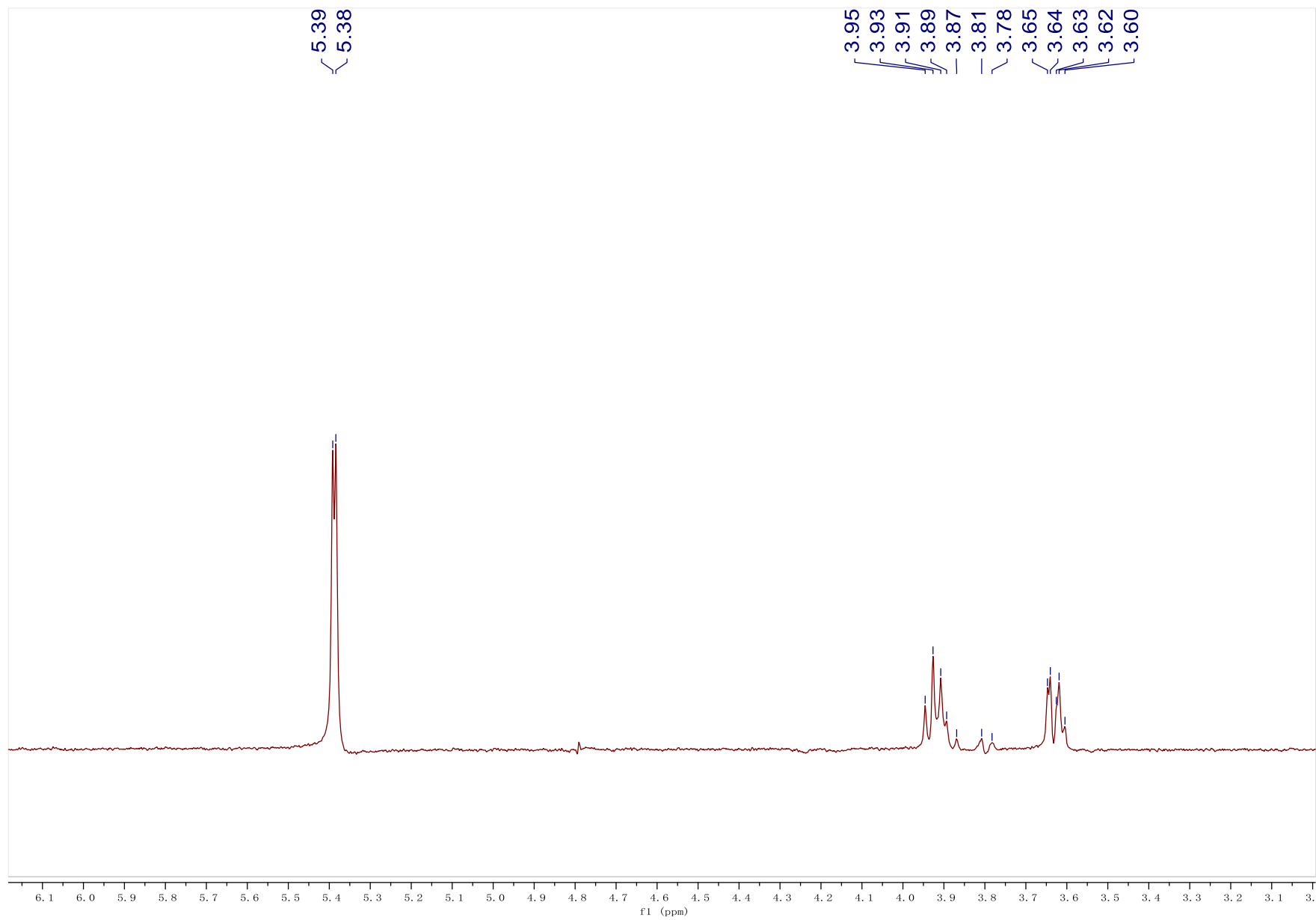
**Figure S33.** 1D-selective TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.44, H-C6a).



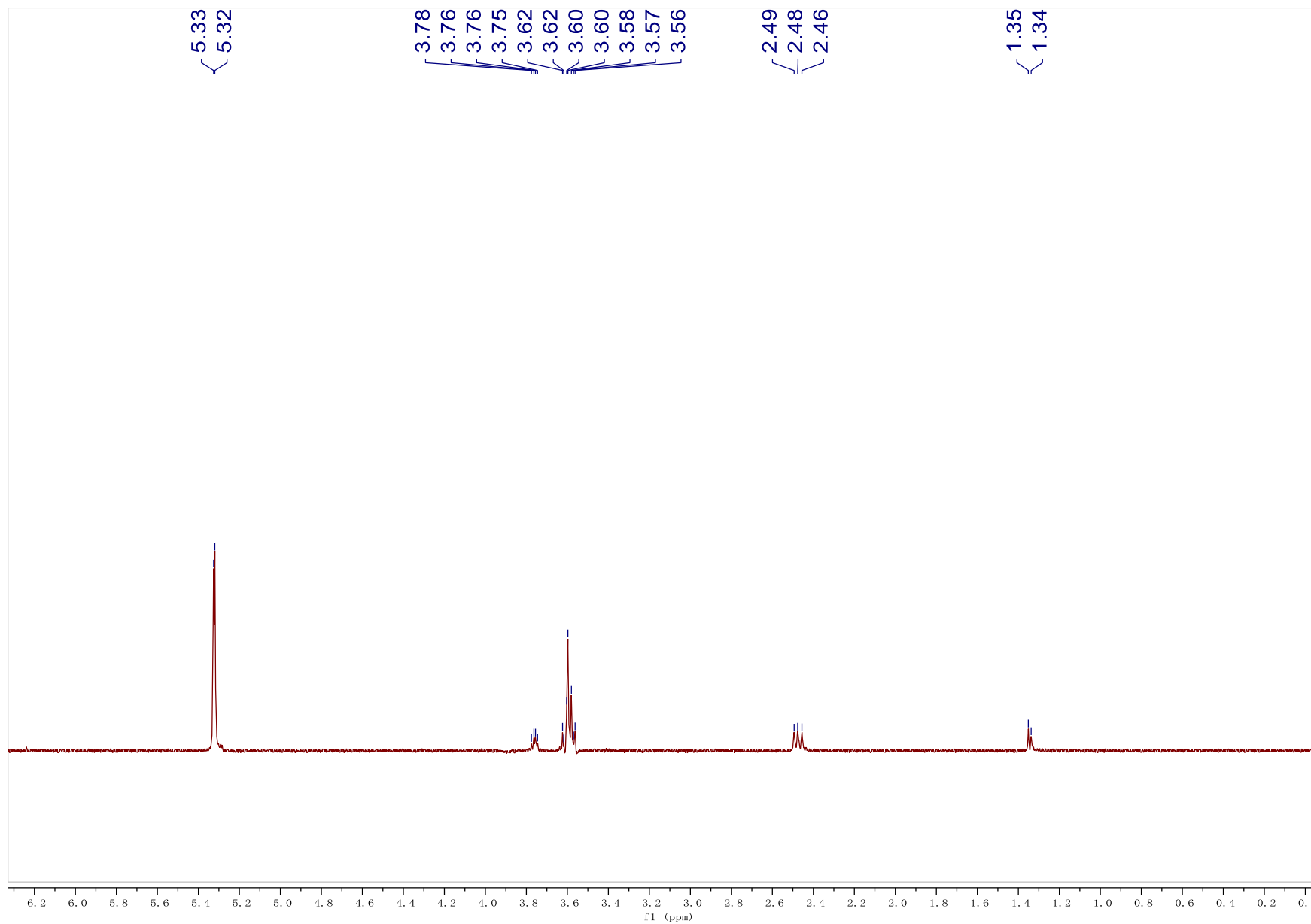
**Figure S34.** 1D-selective TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.29, H-D1).



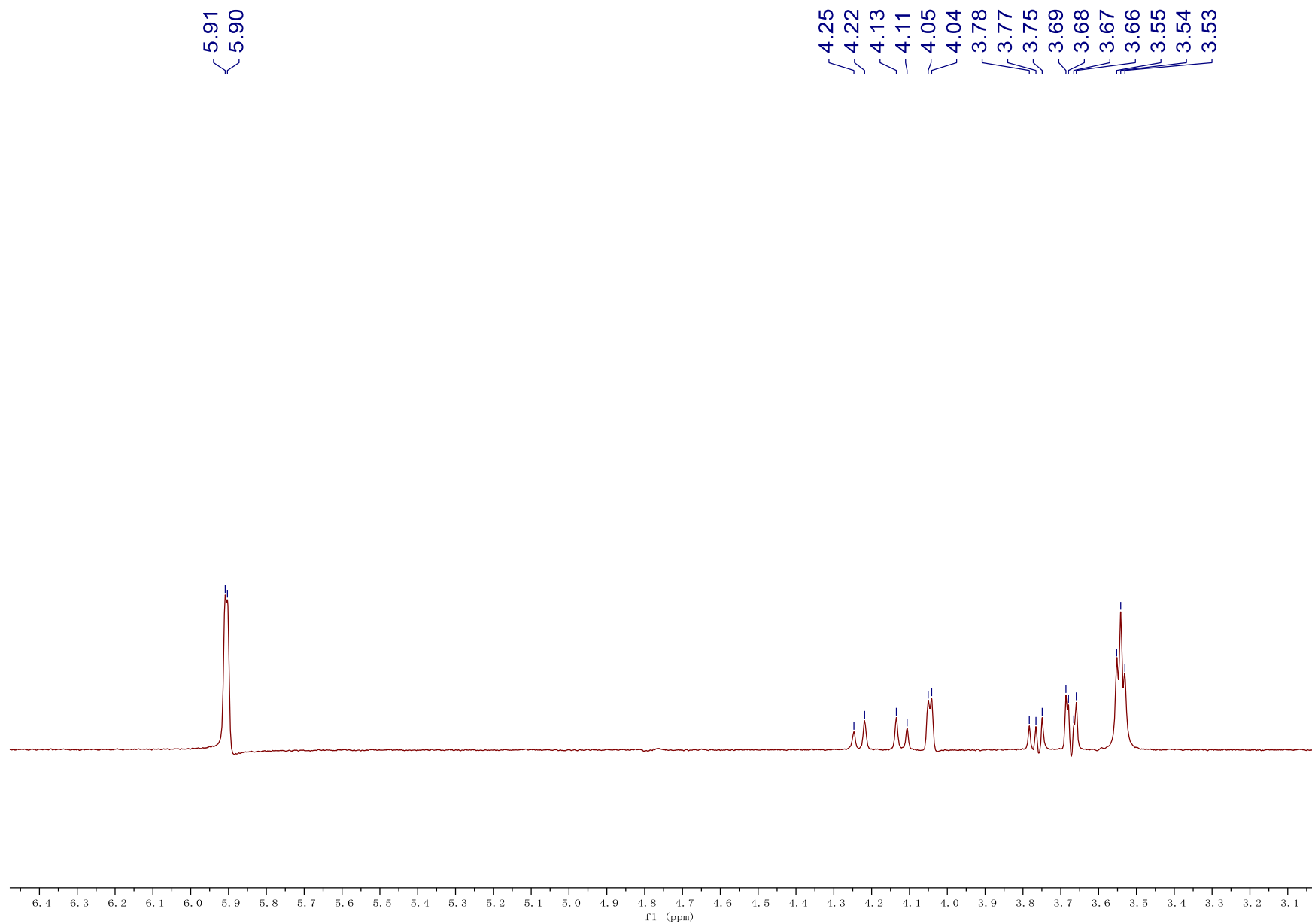
**Figure S35.** 1D-selective TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.98, H-E7).



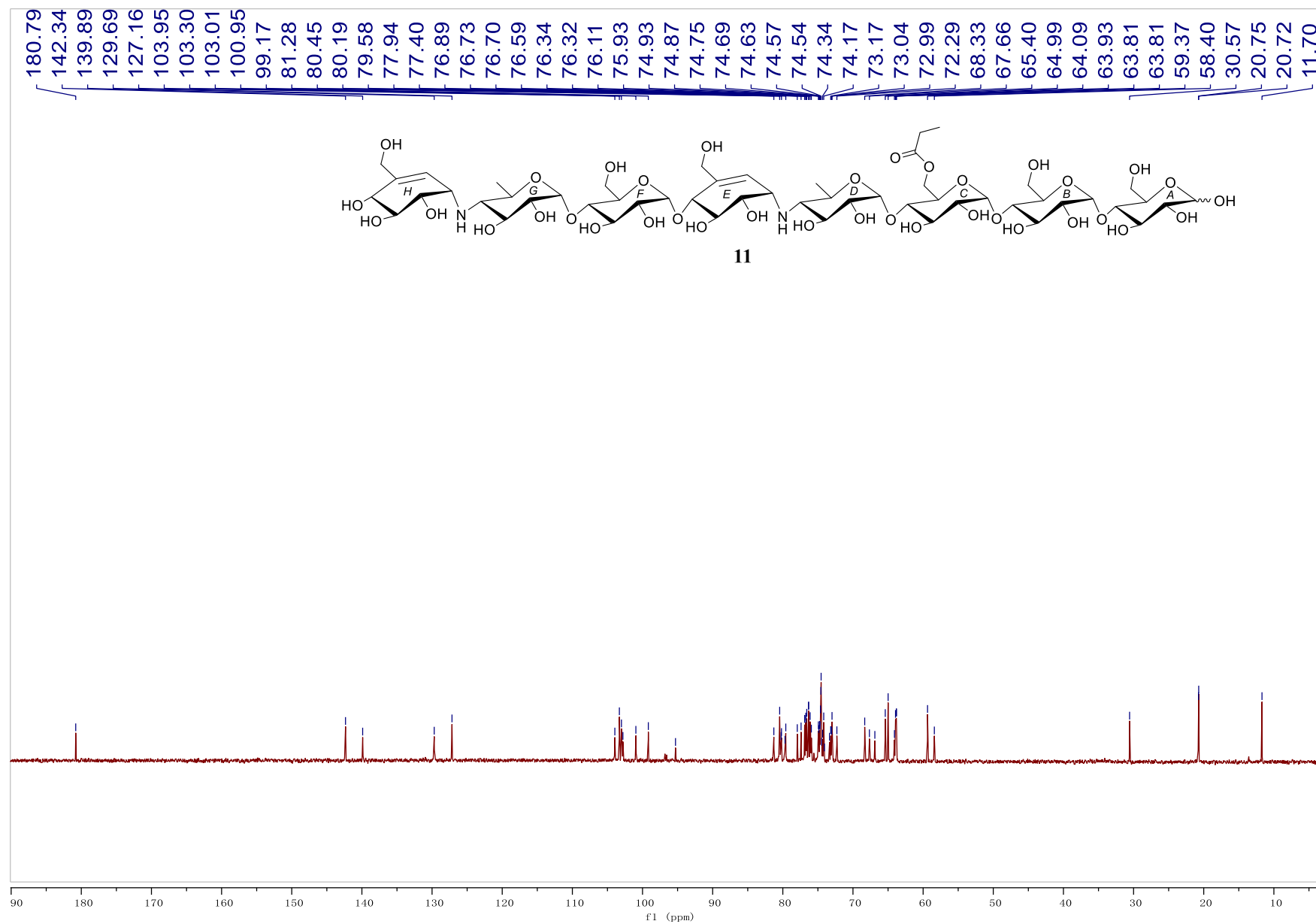
**Figure S36.** 1D-selective TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.38, H-F1).



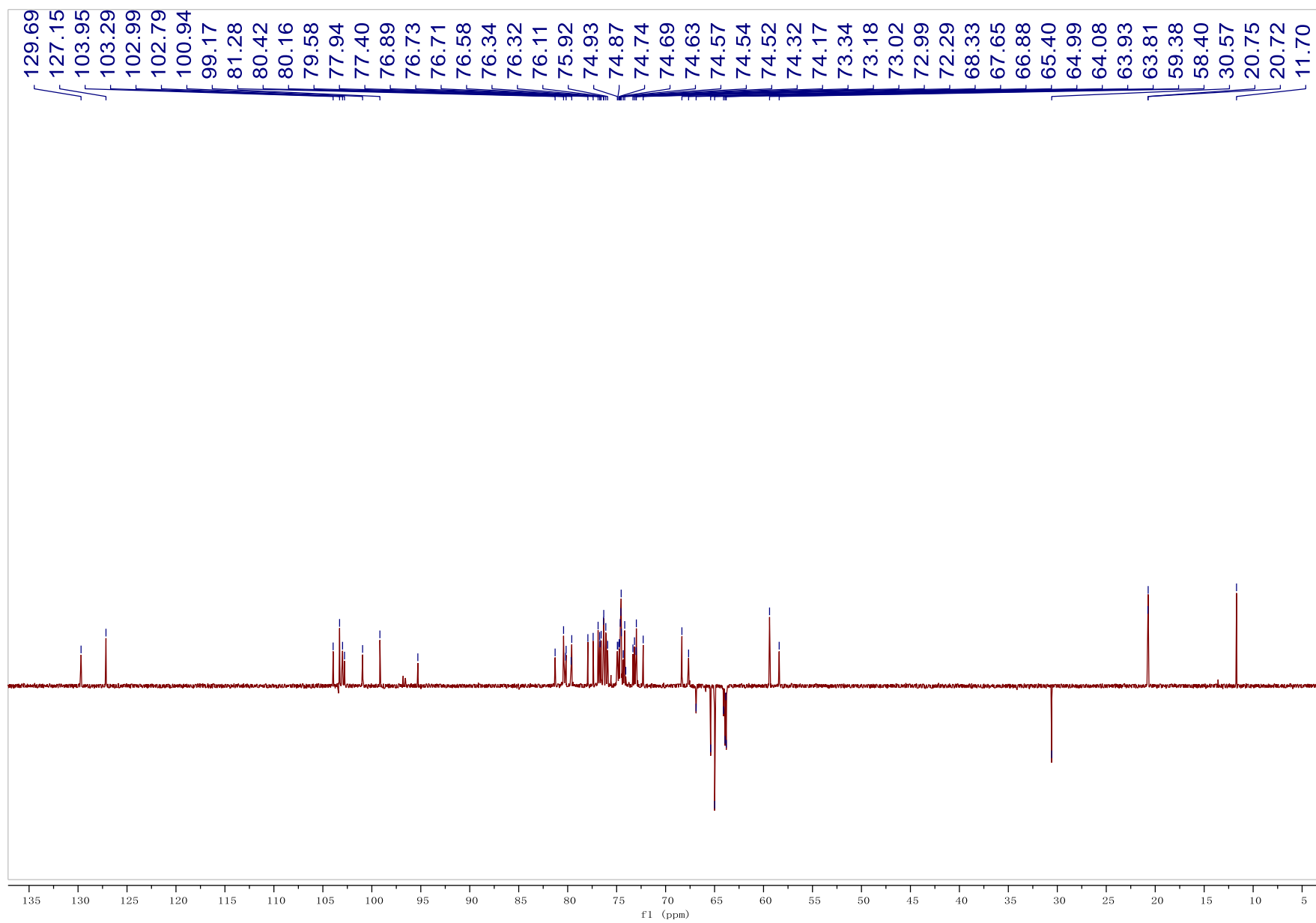
**Figure S37.** 1D-selective TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.32, H-G1).



**Figure S38.** 1D-selective TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.90, H-**H7**).

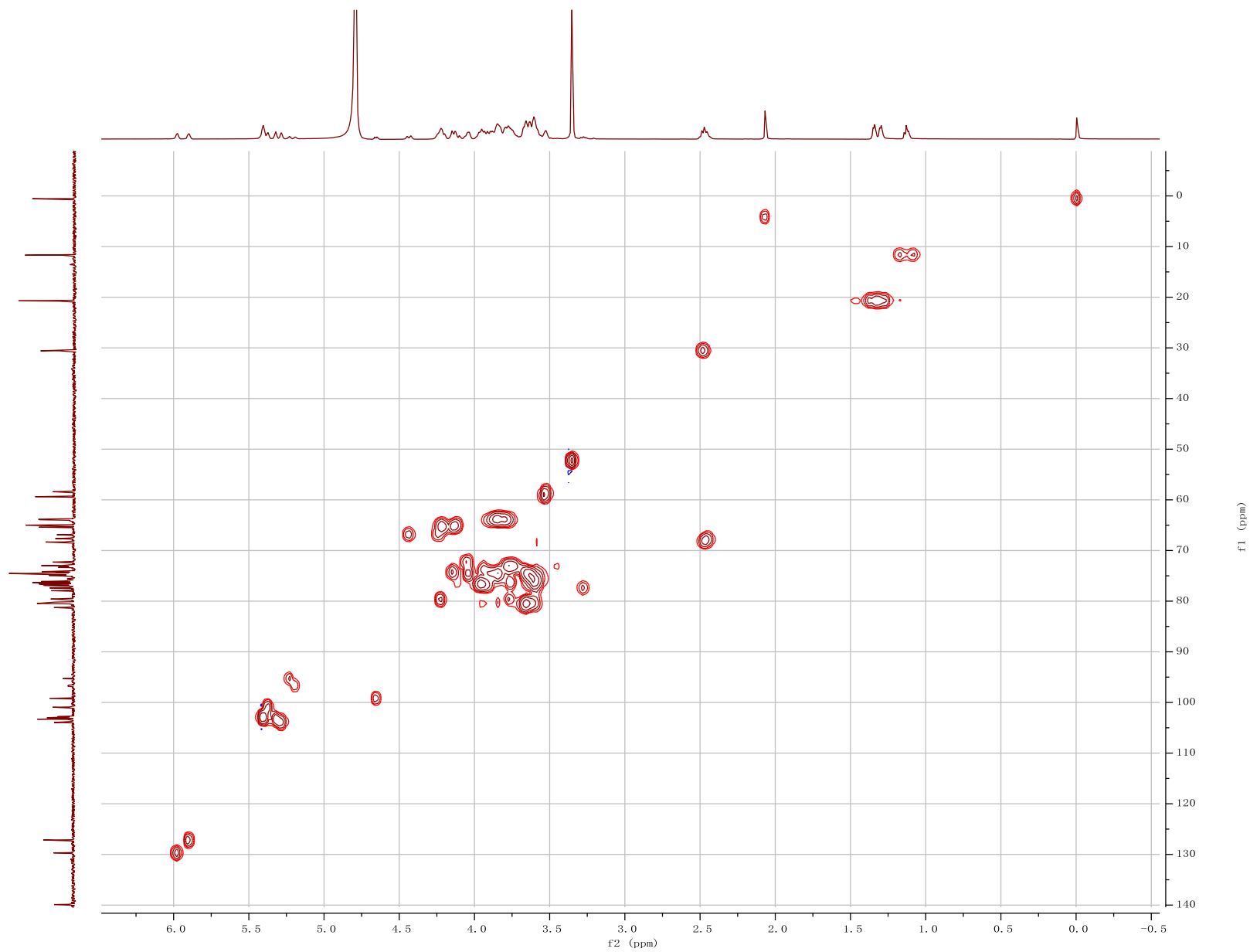


**Figure S39.**  $^{13}\text{C}$  NMR spectrum of compound **11** (125 MHz,  $\text{D}_2\text{O}$ ).

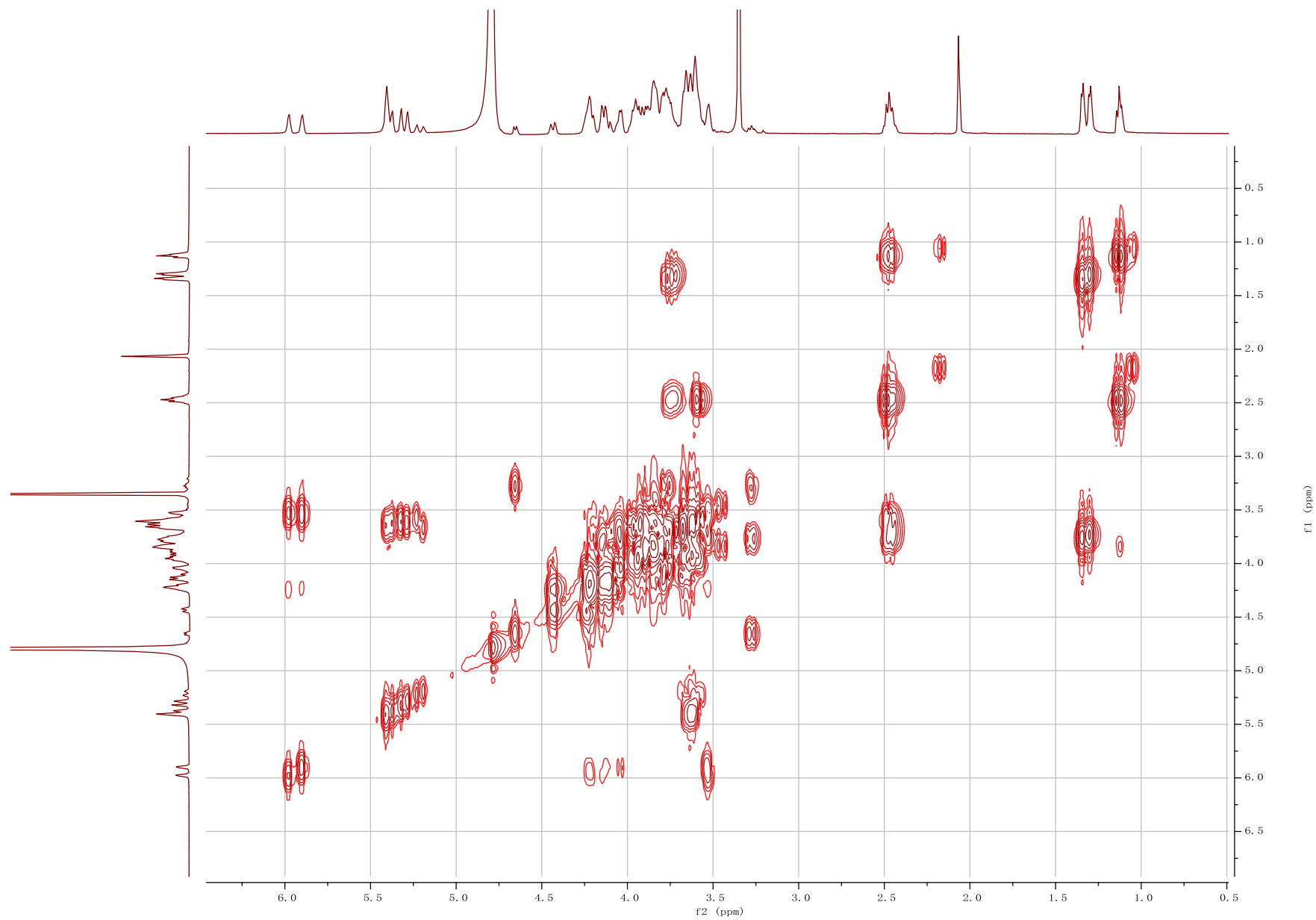


**Figure S40.** DEPT-135 spectrum of compound **11** (125 MHz, D<sub>2</sub>O).

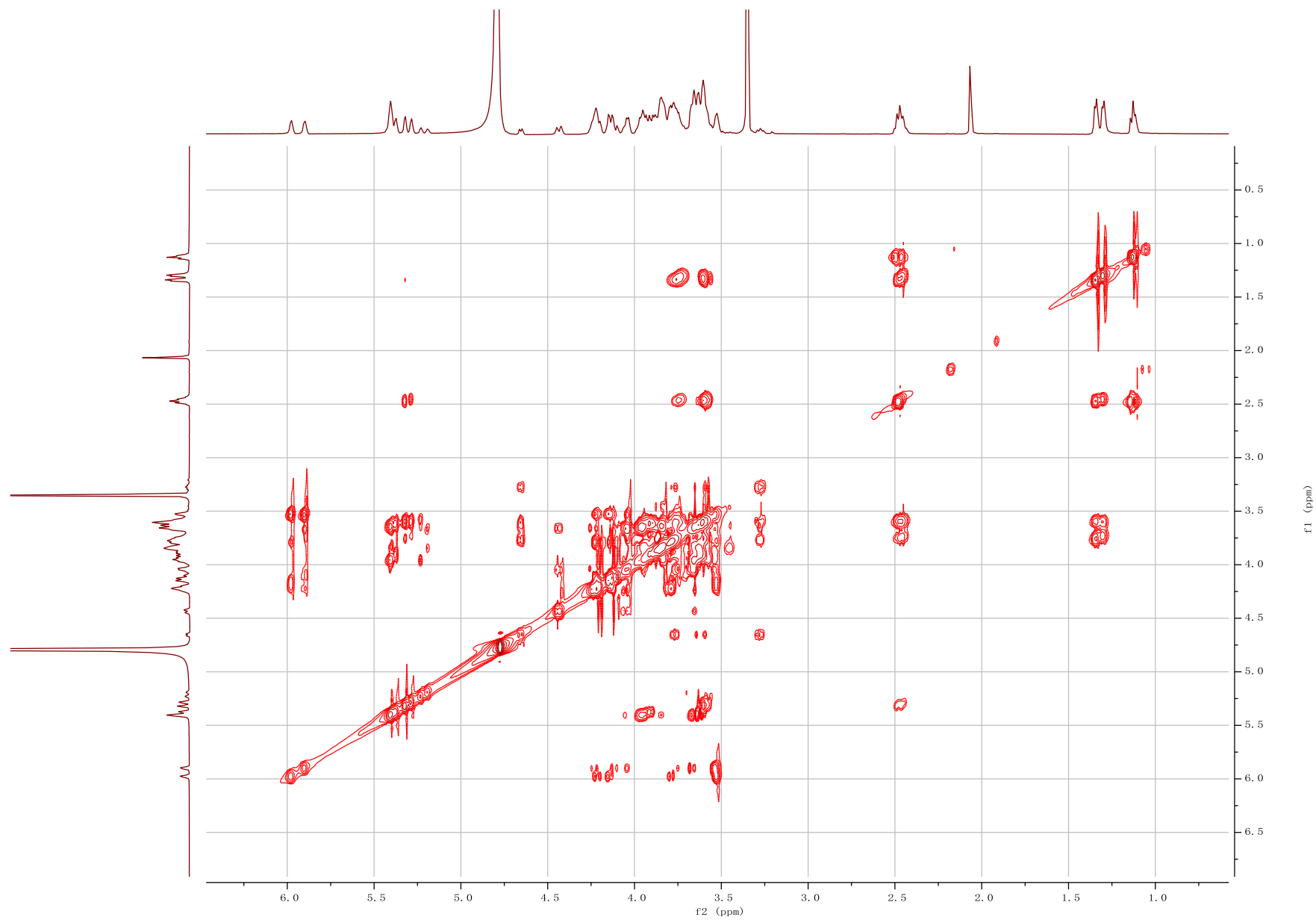




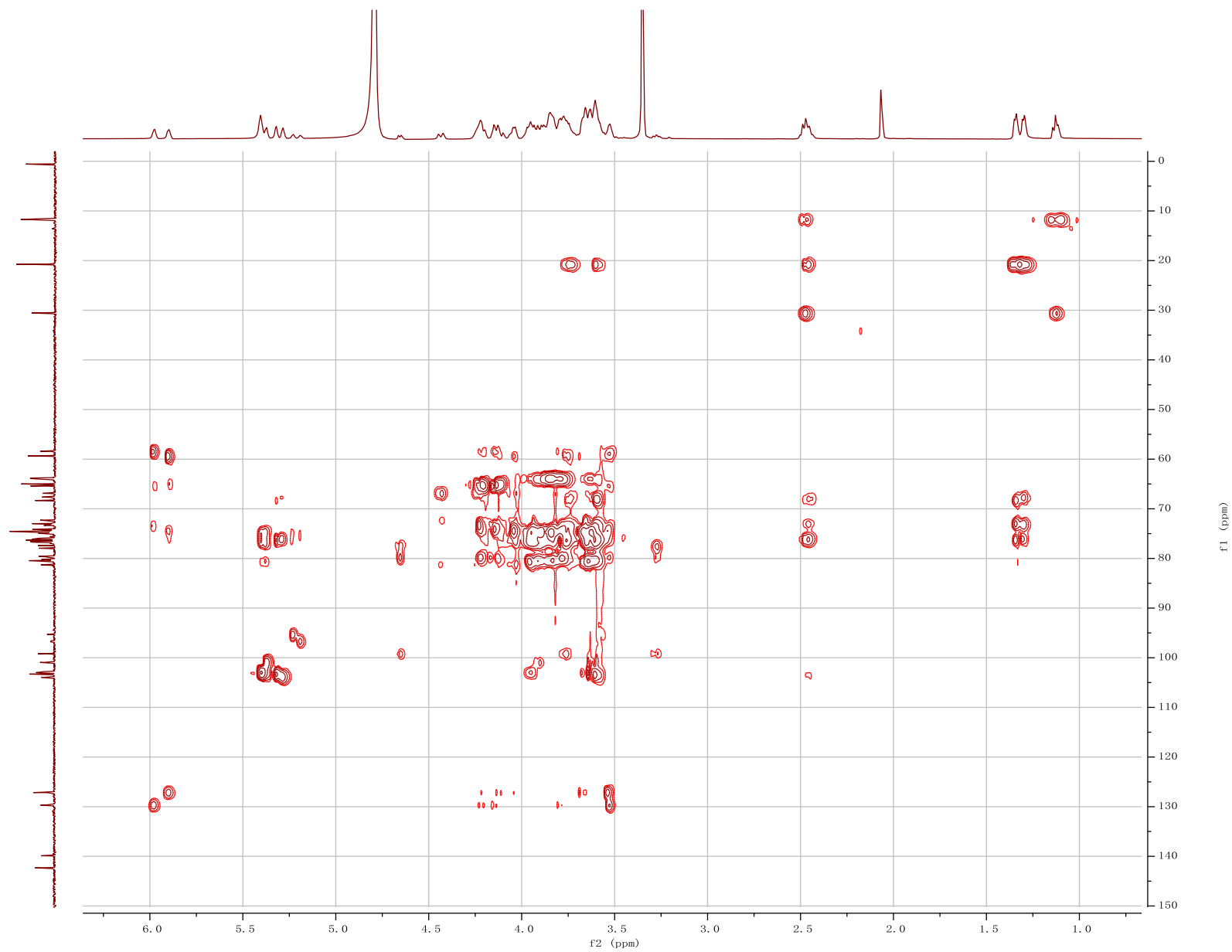
**Figure S41.** HSQC spectrum of compound **11** (500 MHz, D<sub>2</sub>O).



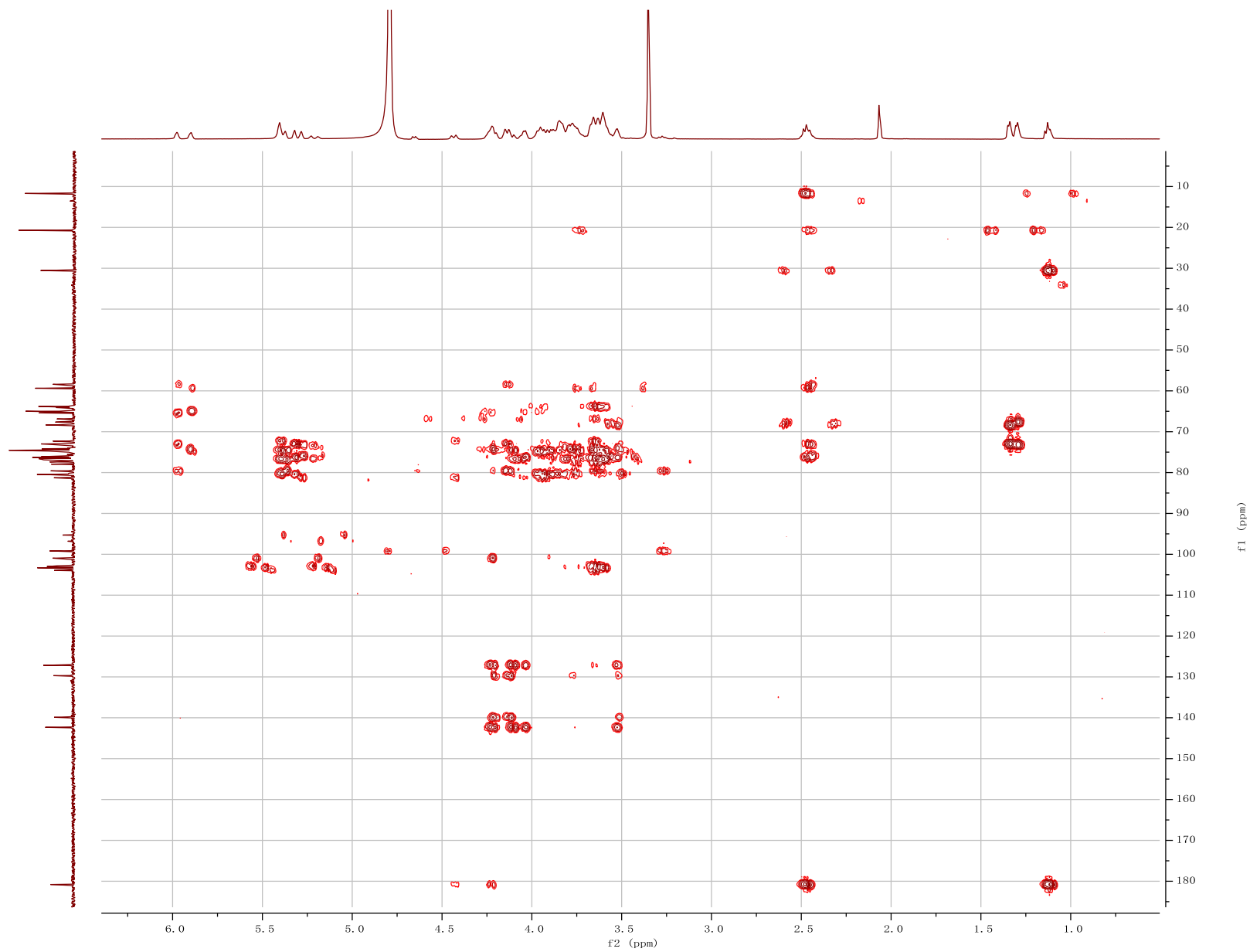
**Figure S42.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound **11** (500 MHz,  $\text{D}_2\text{O}$ ).



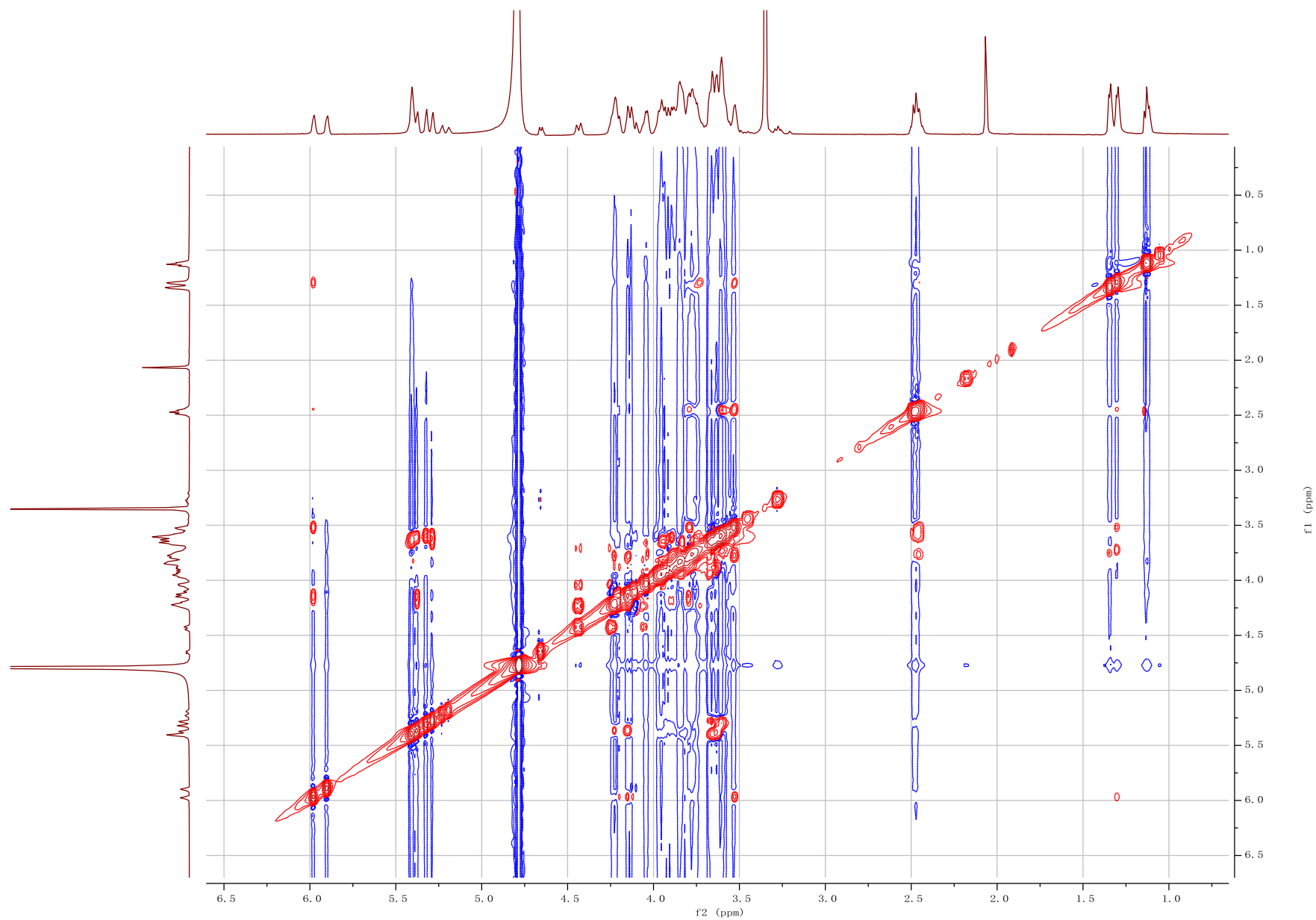
**Figure S43.** 2D-TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O).



**Figure S44.** HSQC-TOCSY spectrum of compound **11** (500 MHz, D<sub>2</sub>O).

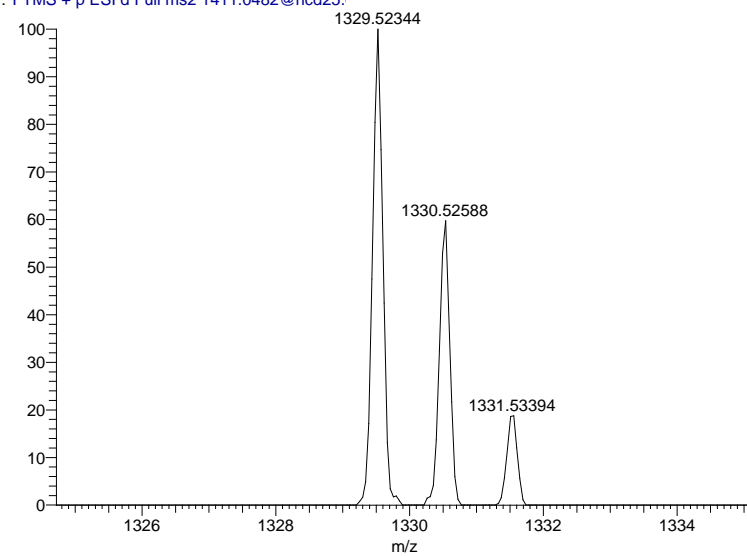


**Figure S45.** HMBC spectrum of compound **11** (500 MHz, D<sub>2</sub>O).

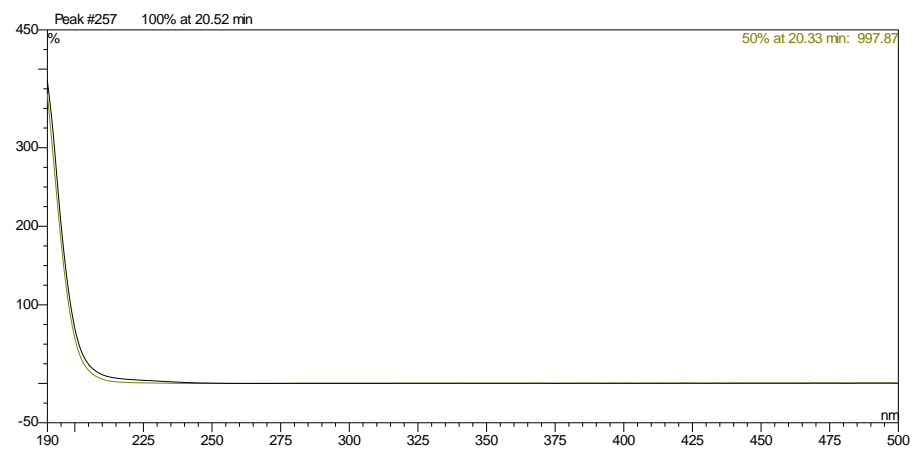


**Figure S46.** NOESY spectrum of compound **11** (500 MHz, D<sub>2</sub>O).

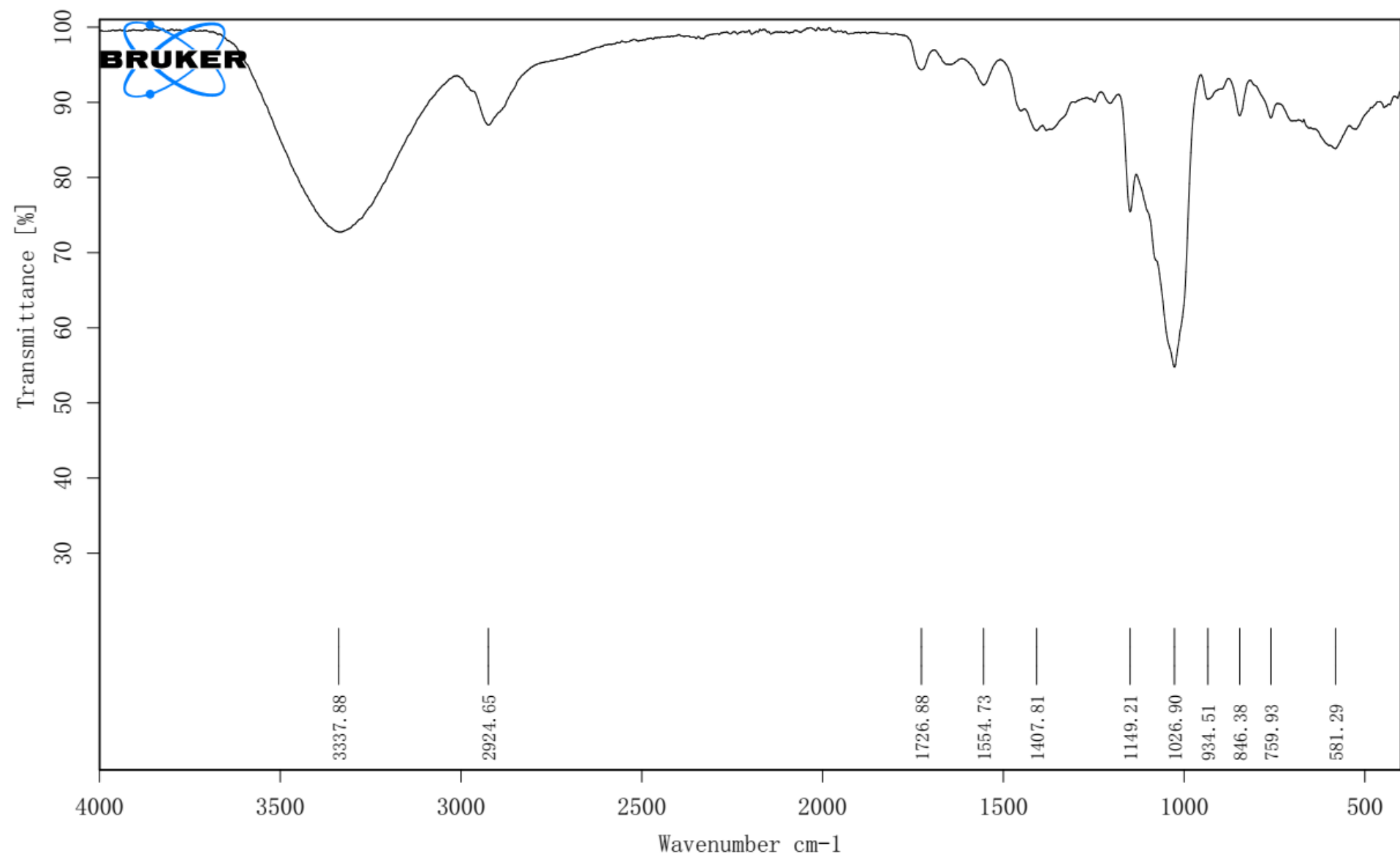
H\_77a #2908 RT: 9.86 AV: 1 NL: 4.02E5  
T: FTMS + p ESI d Full ms2 1411.0482@hcd25.



**Figure S47.** HRESIMS spectrum of compound **11**.

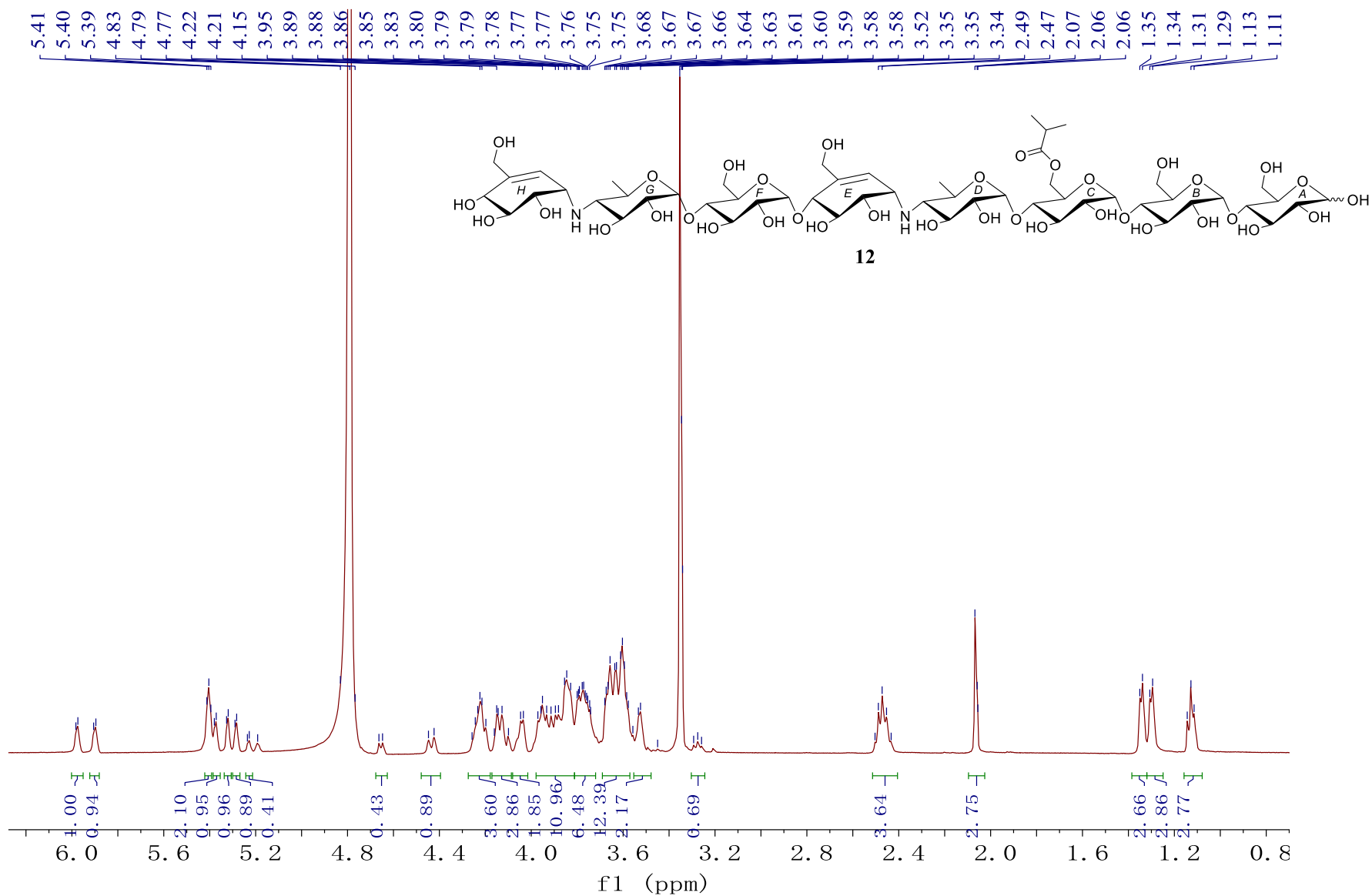


**Figure S48.** UV spectrum of compound **11**.

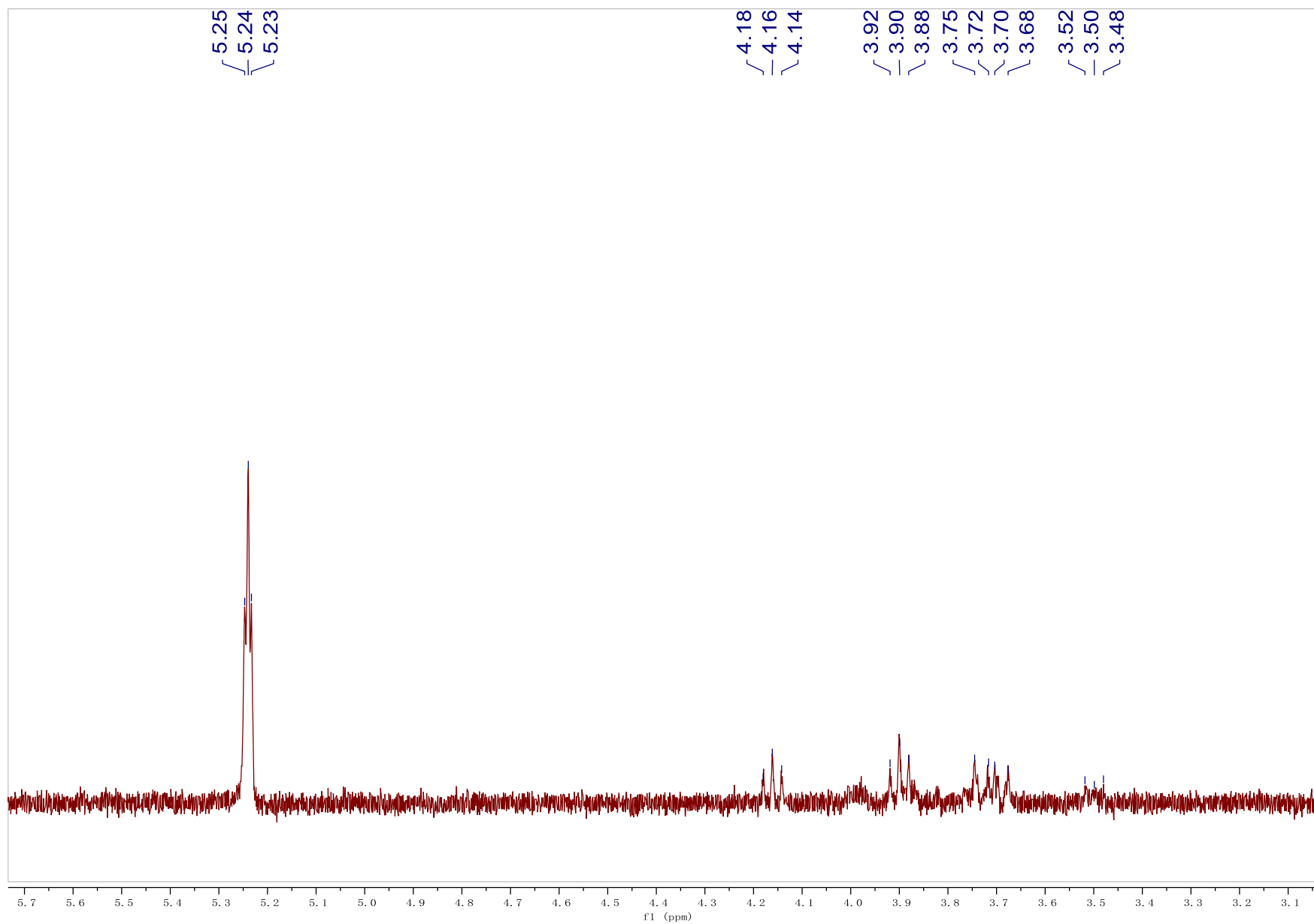


**Figure S49.** IR spectrum of compound **11**.

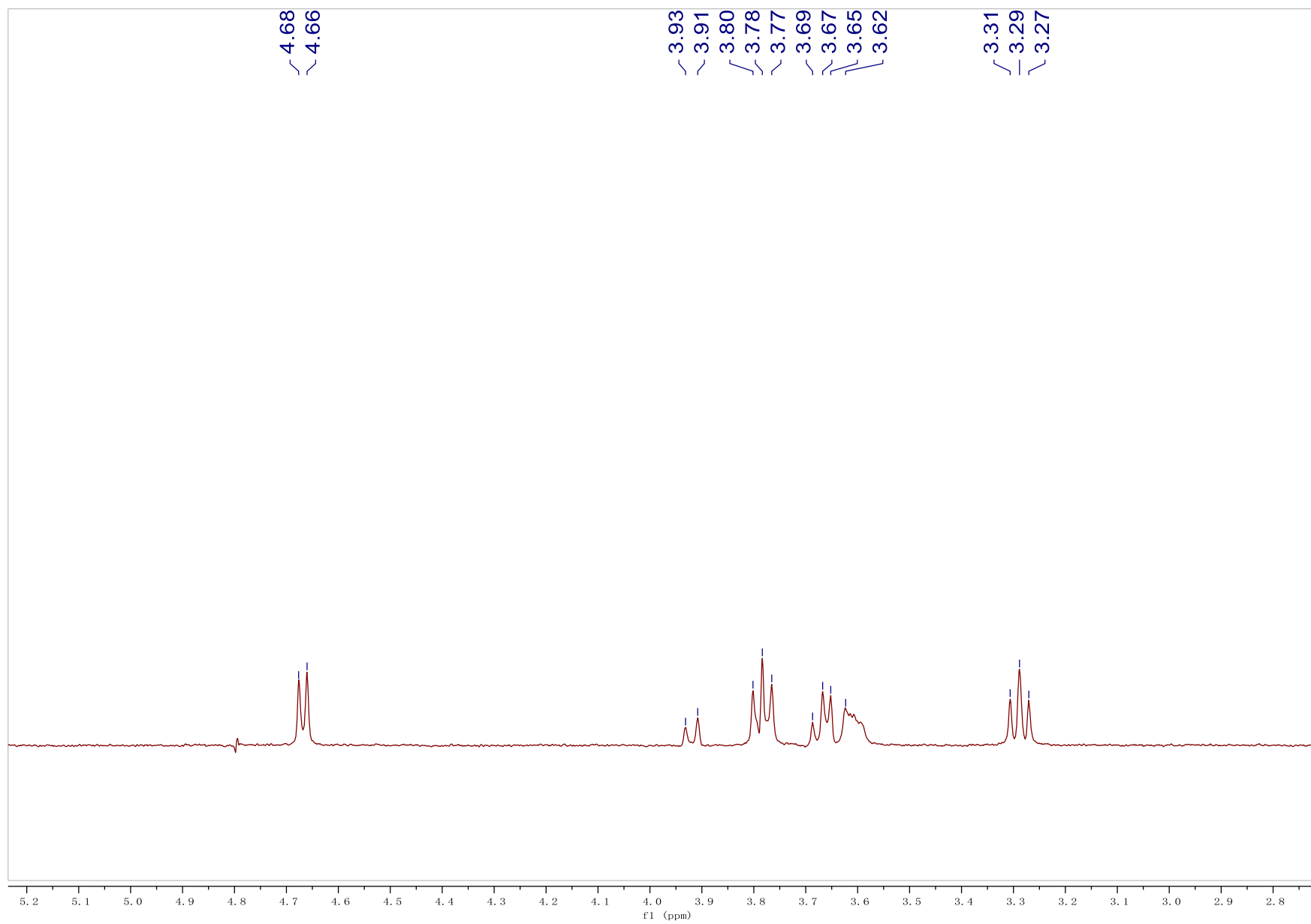




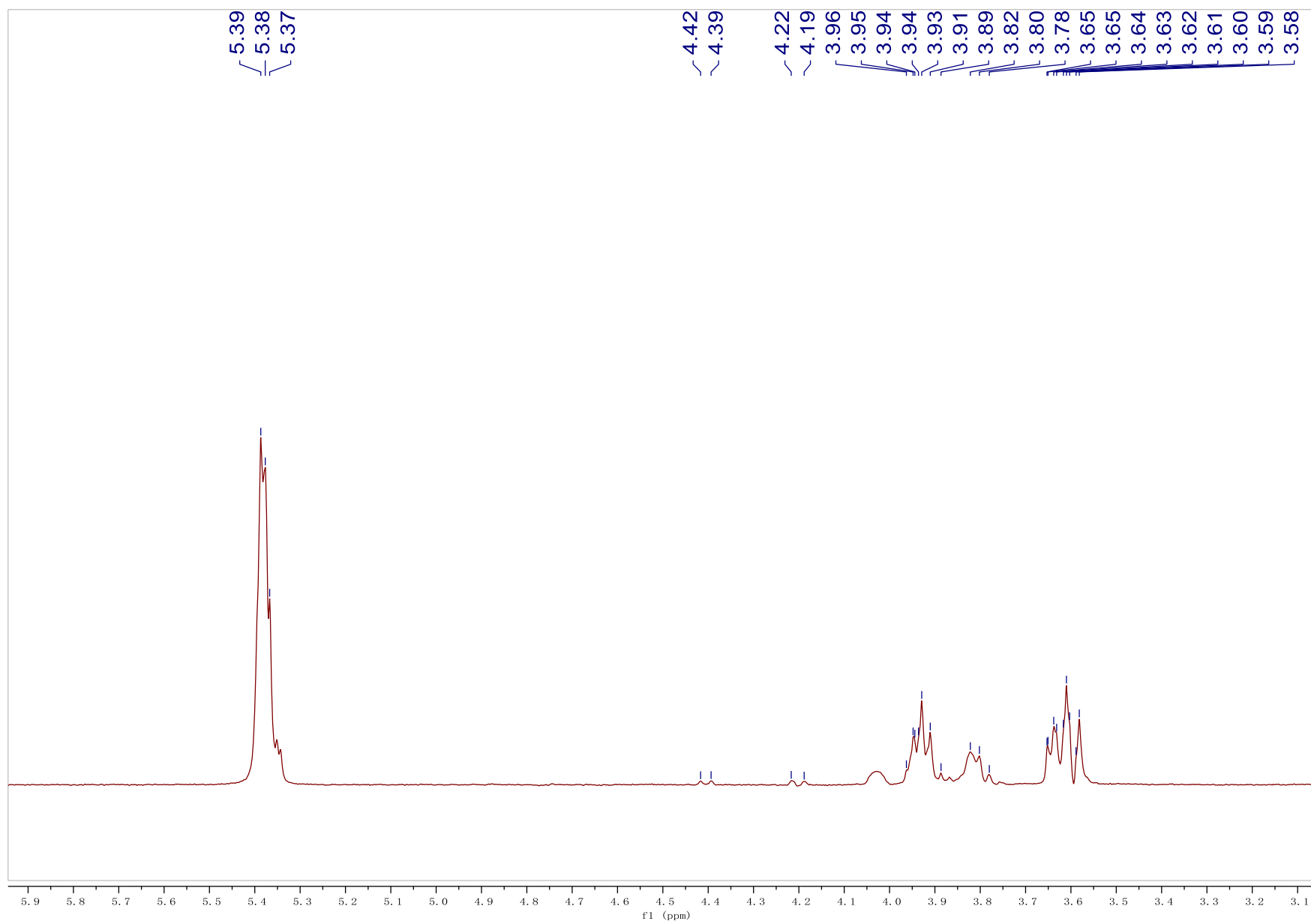
**Figure S50.**  $^1\text{H}$  NMR spectrum of compound **12** (500 MHz,  $\text{D}_2\text{O}$ ).



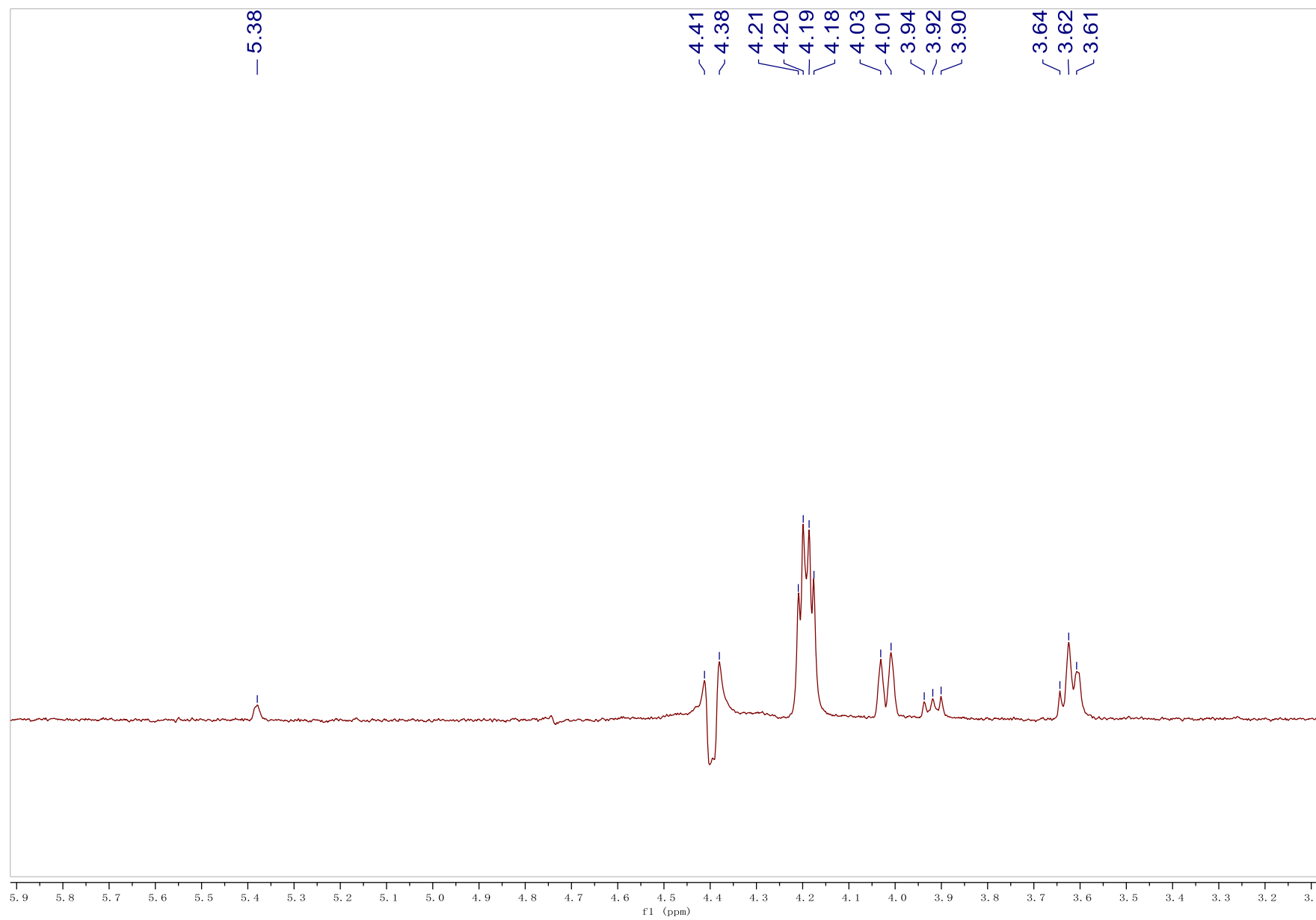
**Figure S51.** 1D-selective TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.24, H-A1 $\alpha$ ).



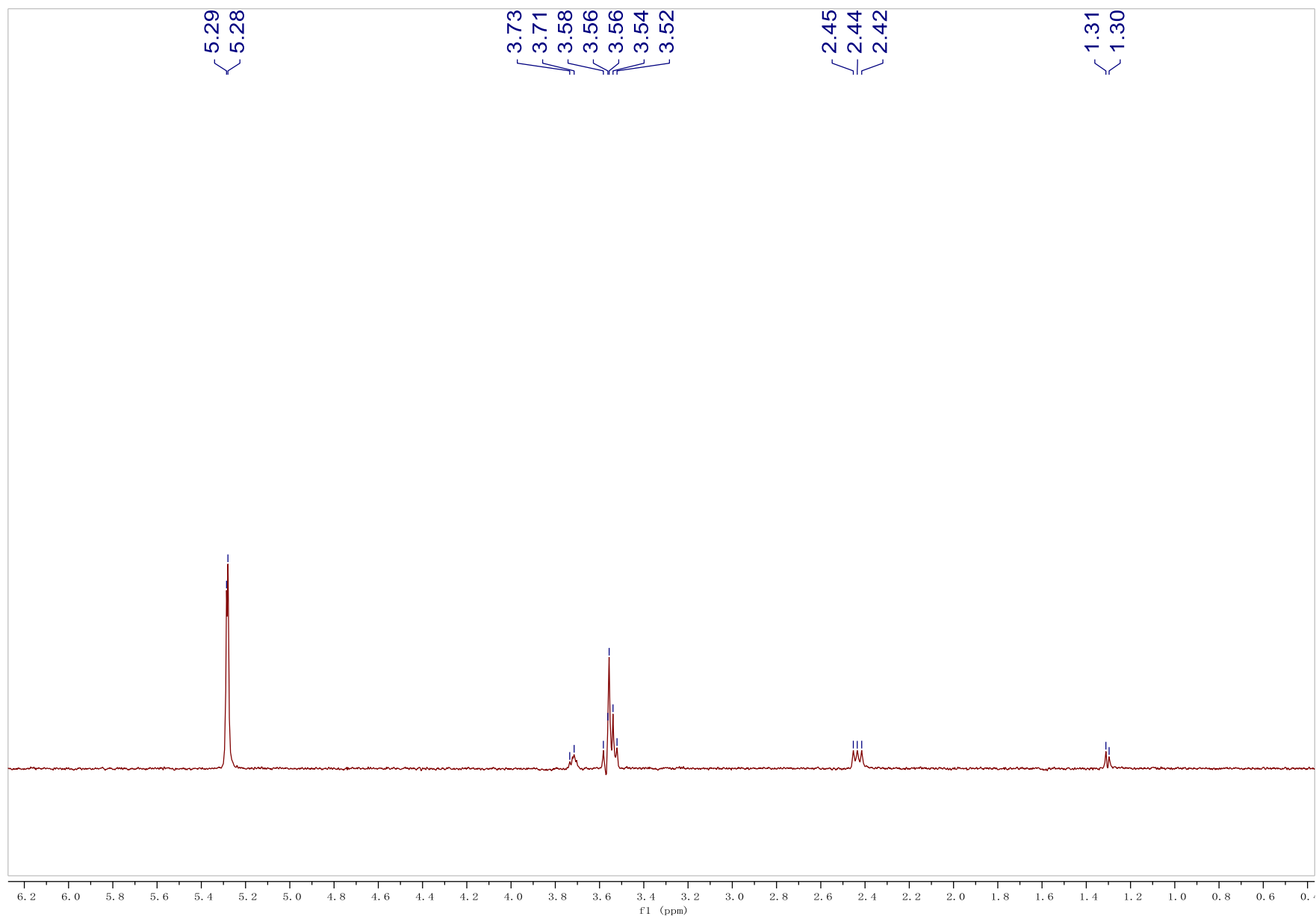
**Figure S52.** 1D-selective TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.66, H-A1 $\beta$ ).



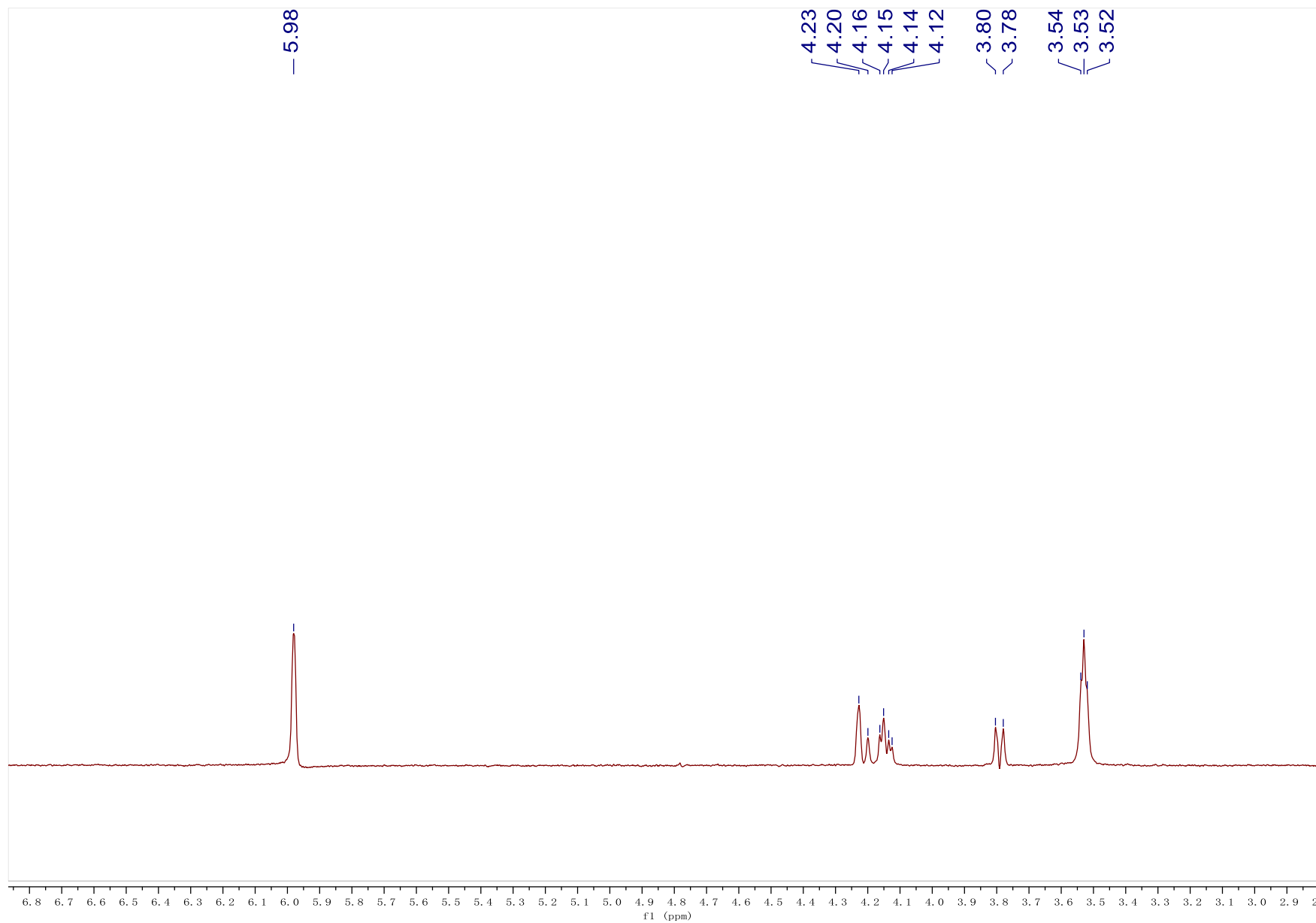
**Figure S53.** 1D-selective TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.38, H-**B1** and H-**C1**).



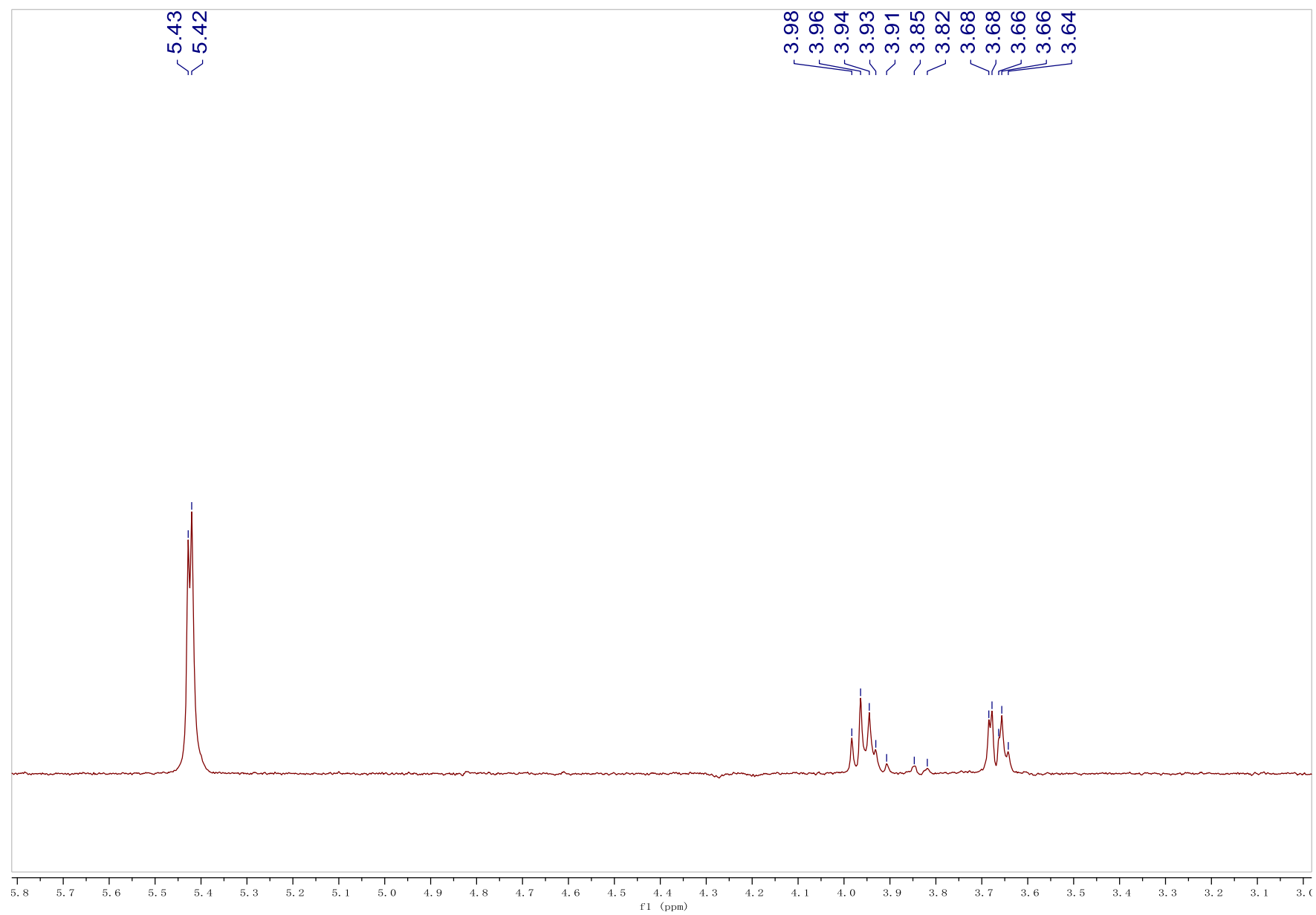
**Figure S54.** 1D-selective TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.44, H-C6a).



**Figure S55.** 1D-selective TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.28, H-D1).

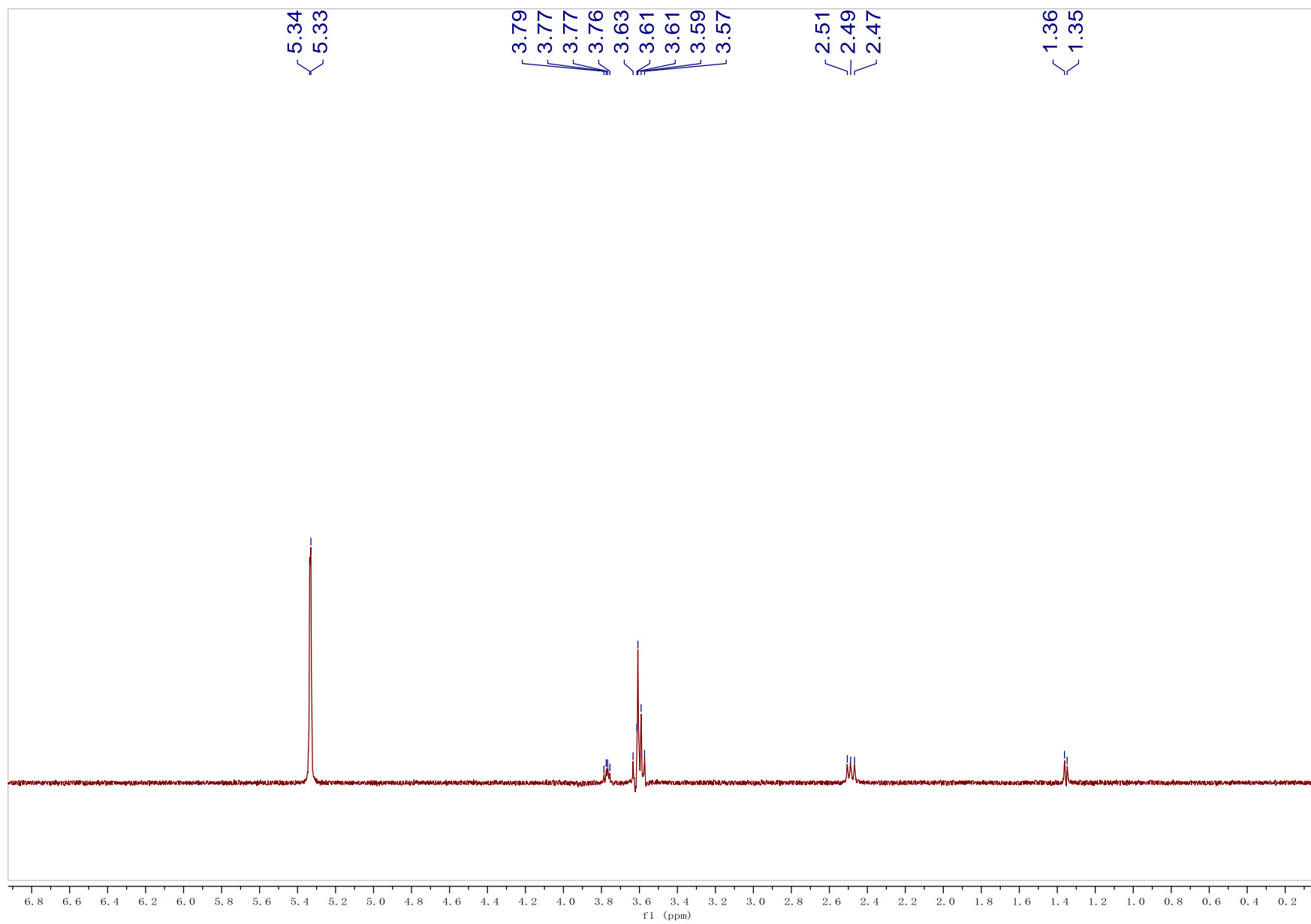


**Figure S56.** 1D-selective TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.98, H-E1).

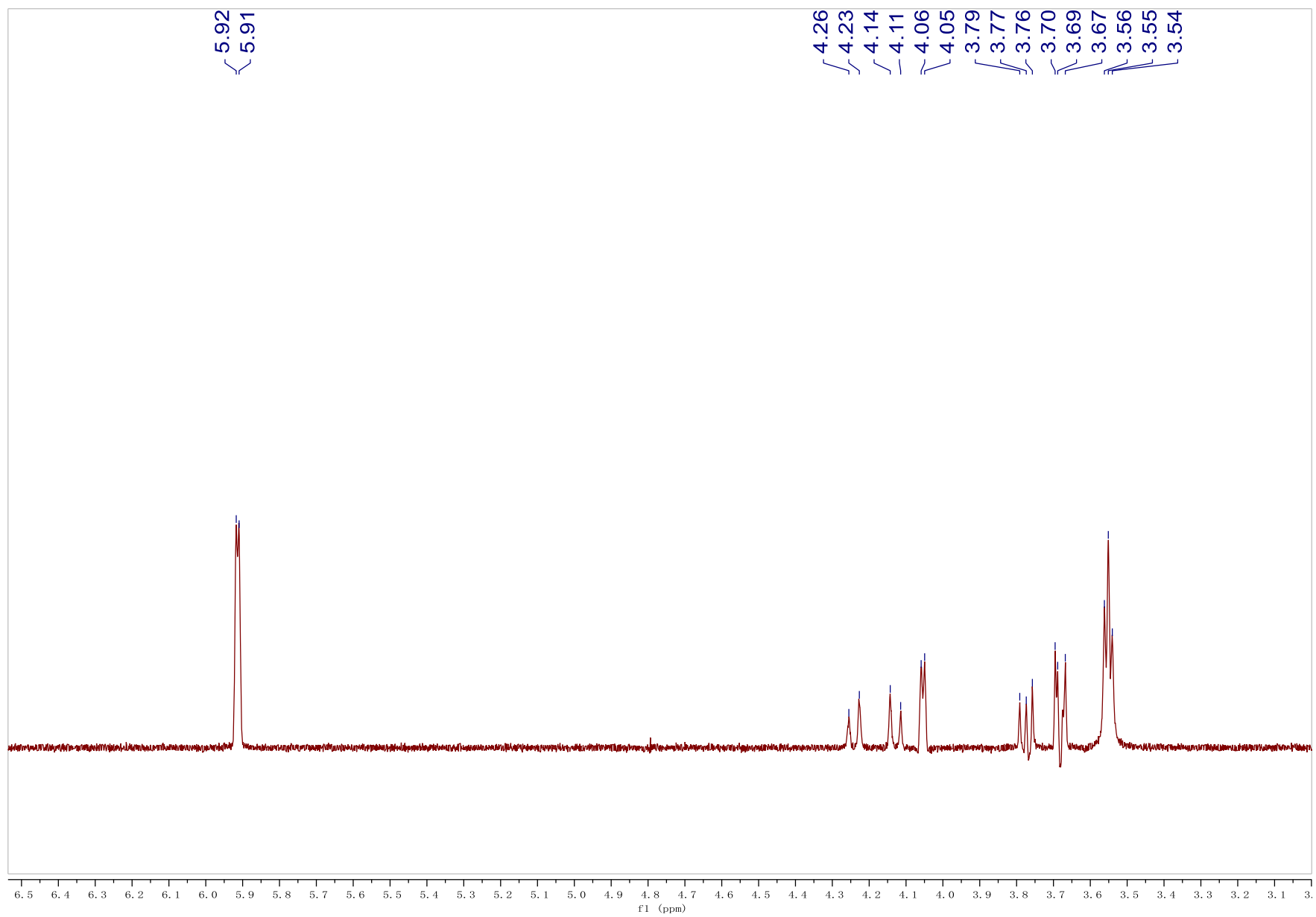


**Figure S57.** 1D-selective TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.42, H-F1).

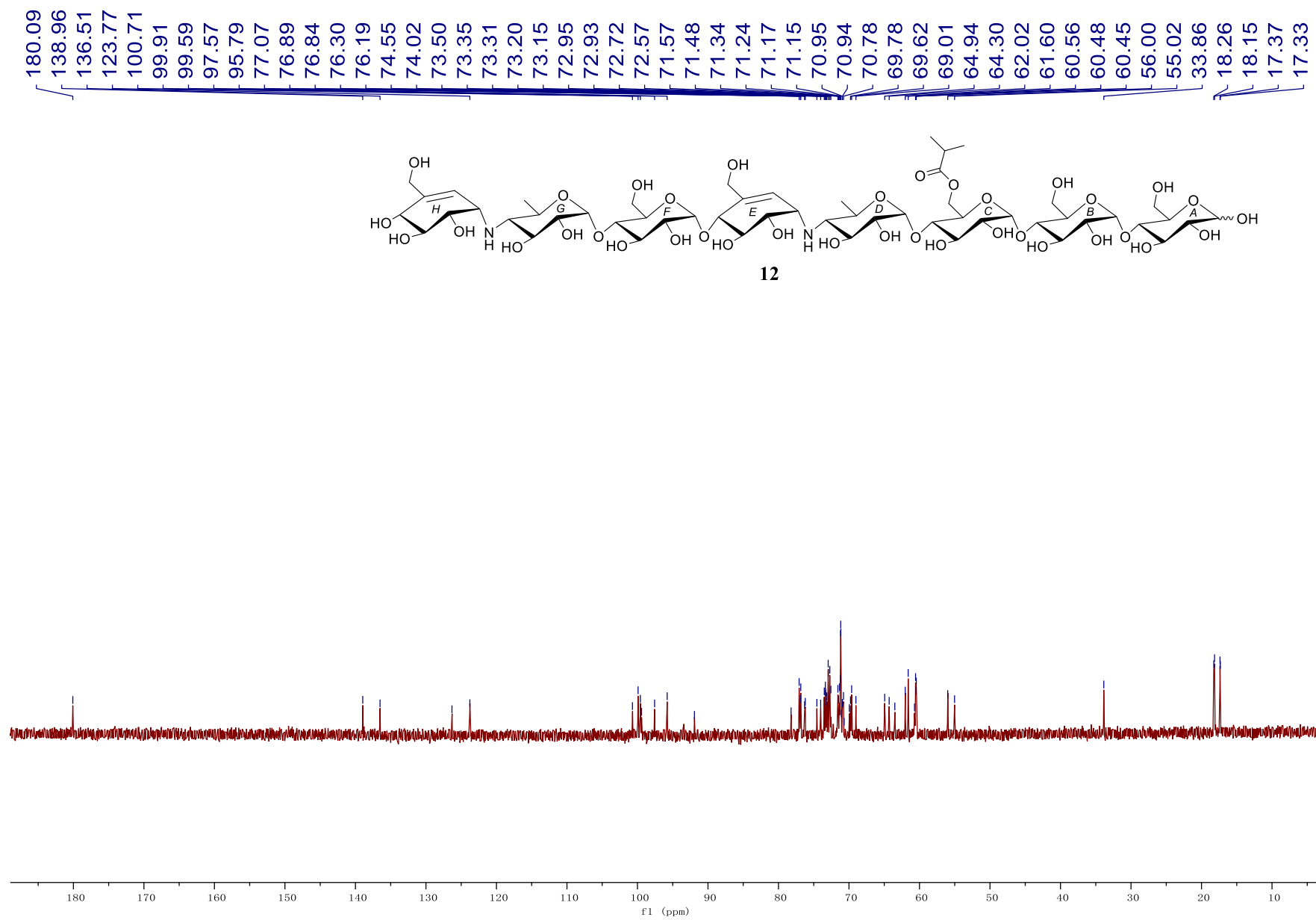




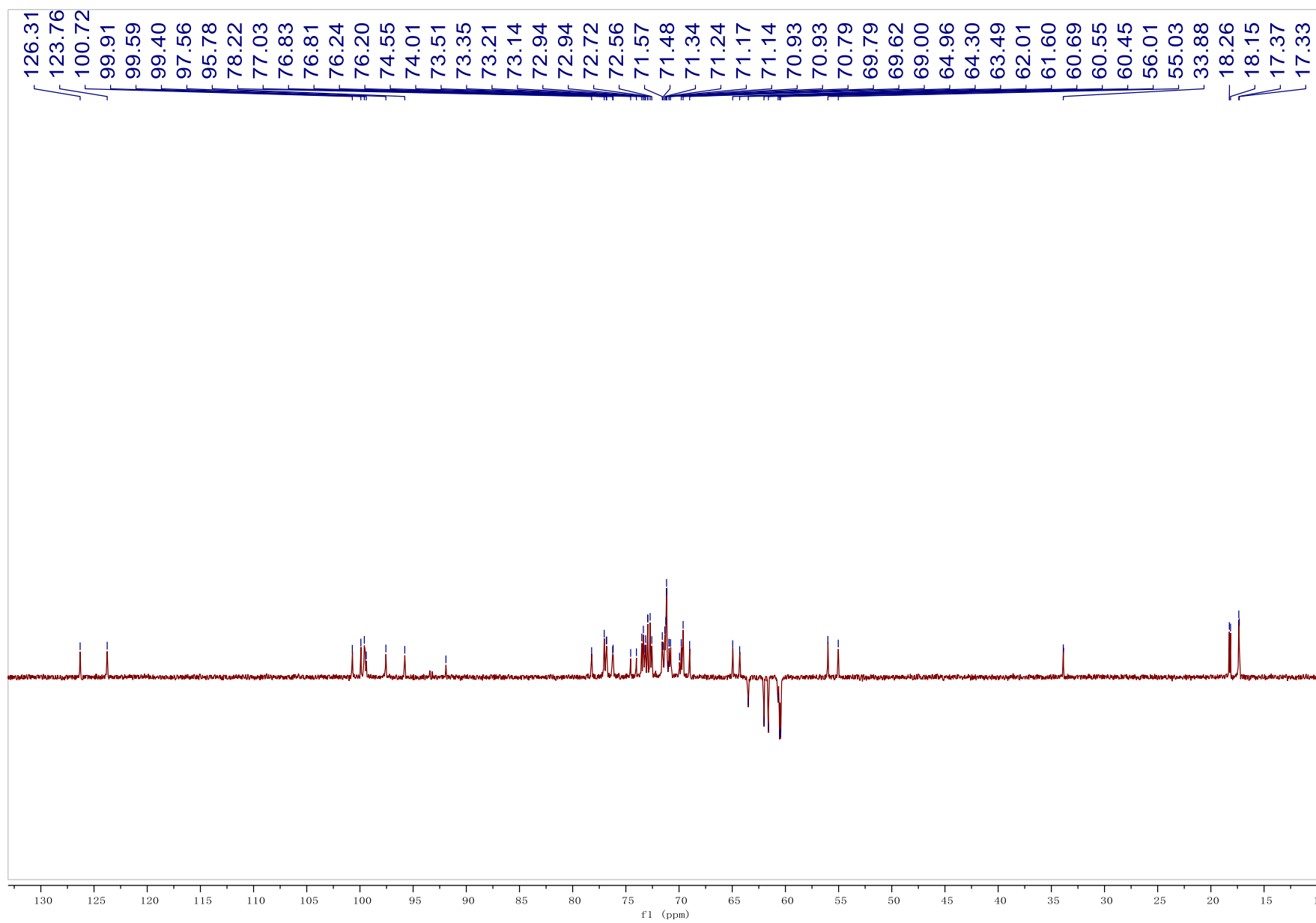
**Figure S58.** 1D-selective TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.33, H-G1).



**Figure S59.** 1D-selective TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.91, H-H1).



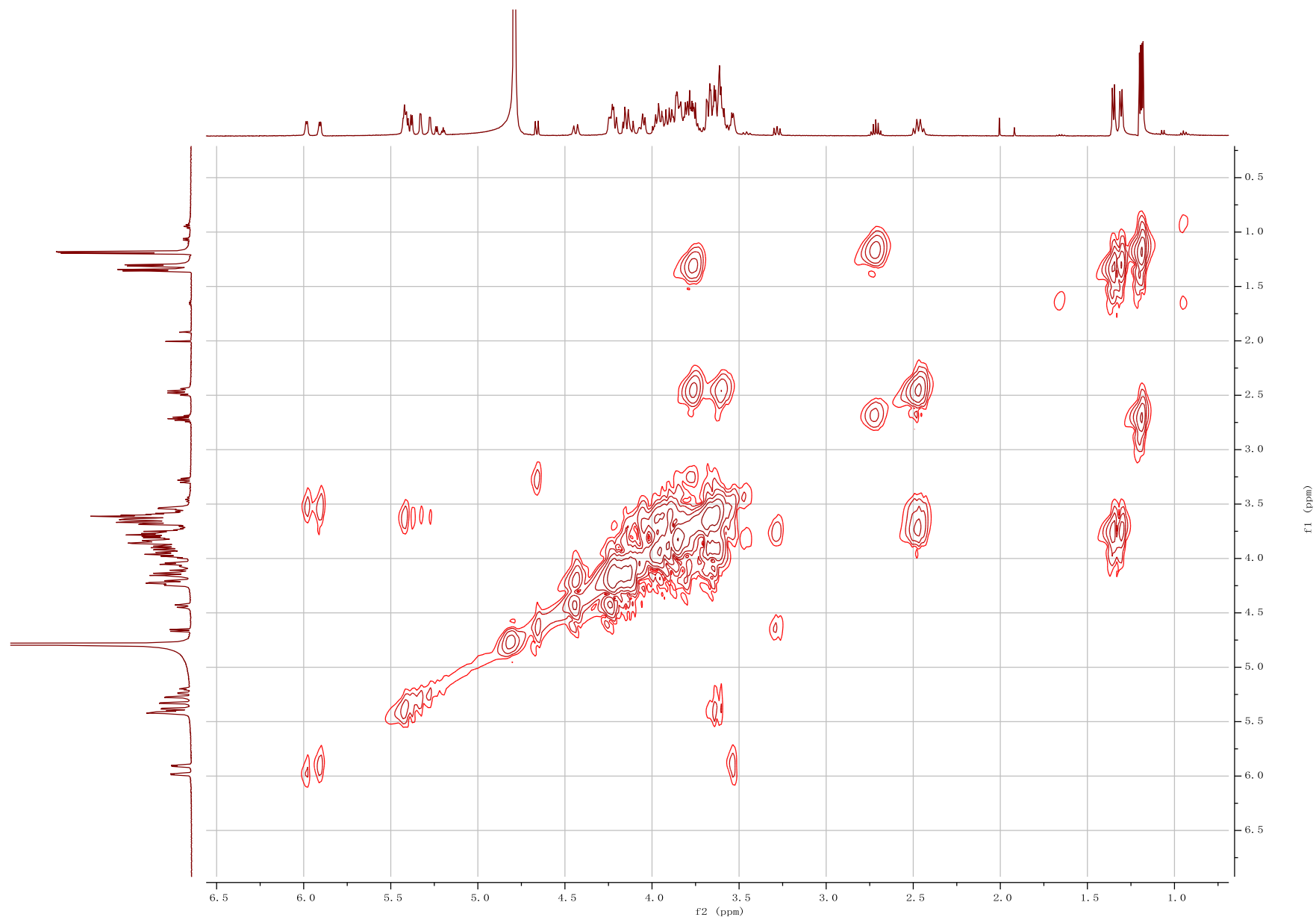
**Figure S60.**  $^{13}\text{C}$  NMR spectrum of compound **12** (125 MHz,  $\text{D}_2\text{O}$ ).



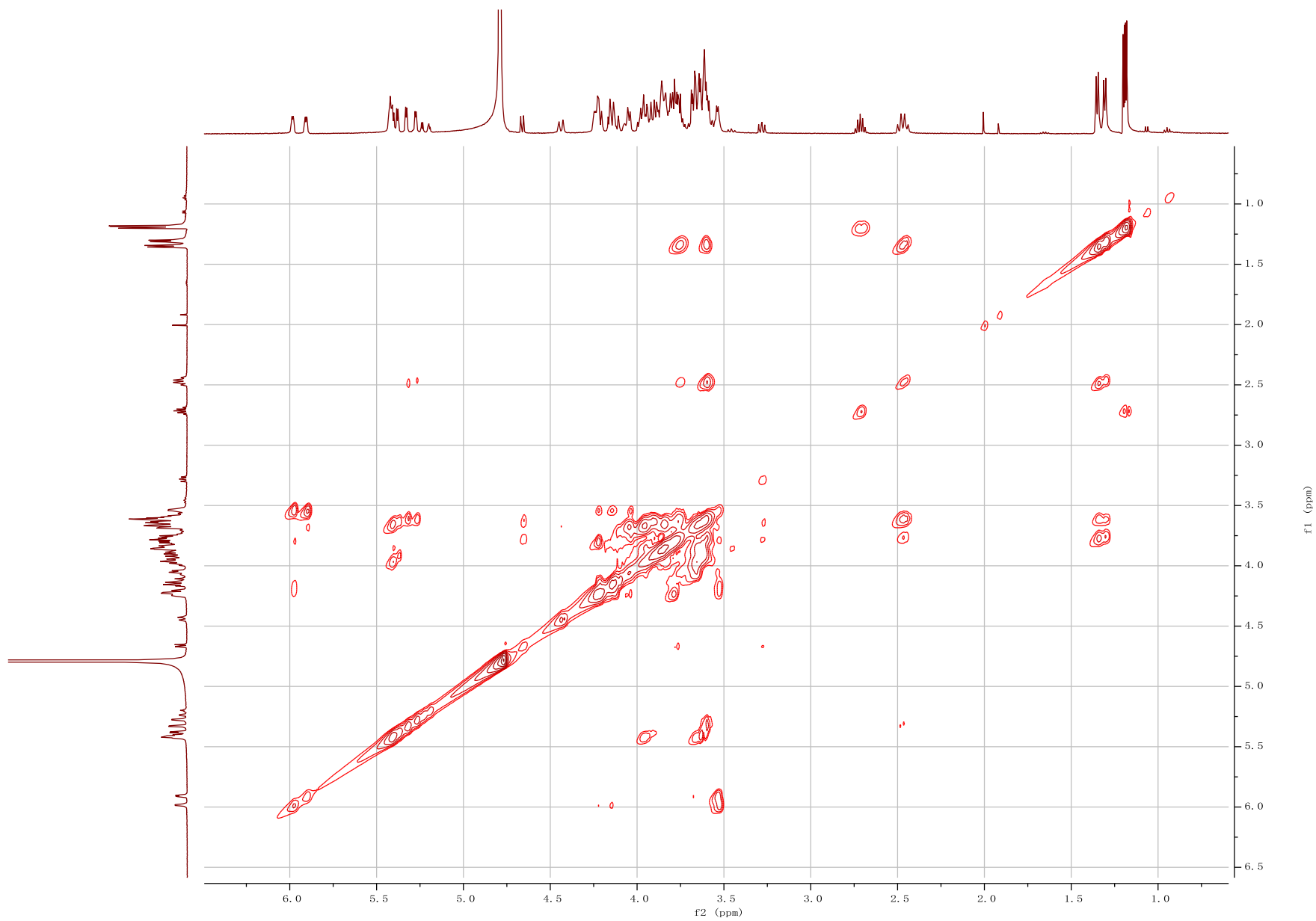
**Figure S61.** DEPT-135 spectrum of compound **12** (125 MHz, D<sub>2</sub>O).



**Figure S62.** HSQC spectrum of compound **12** (500 MHz, D<sub>2</sub>O).



**Figure S63.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound **12** (500 MHz,  $\text{D}_2\text{O}$ ).



**Figure S64.** 2D-TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O).

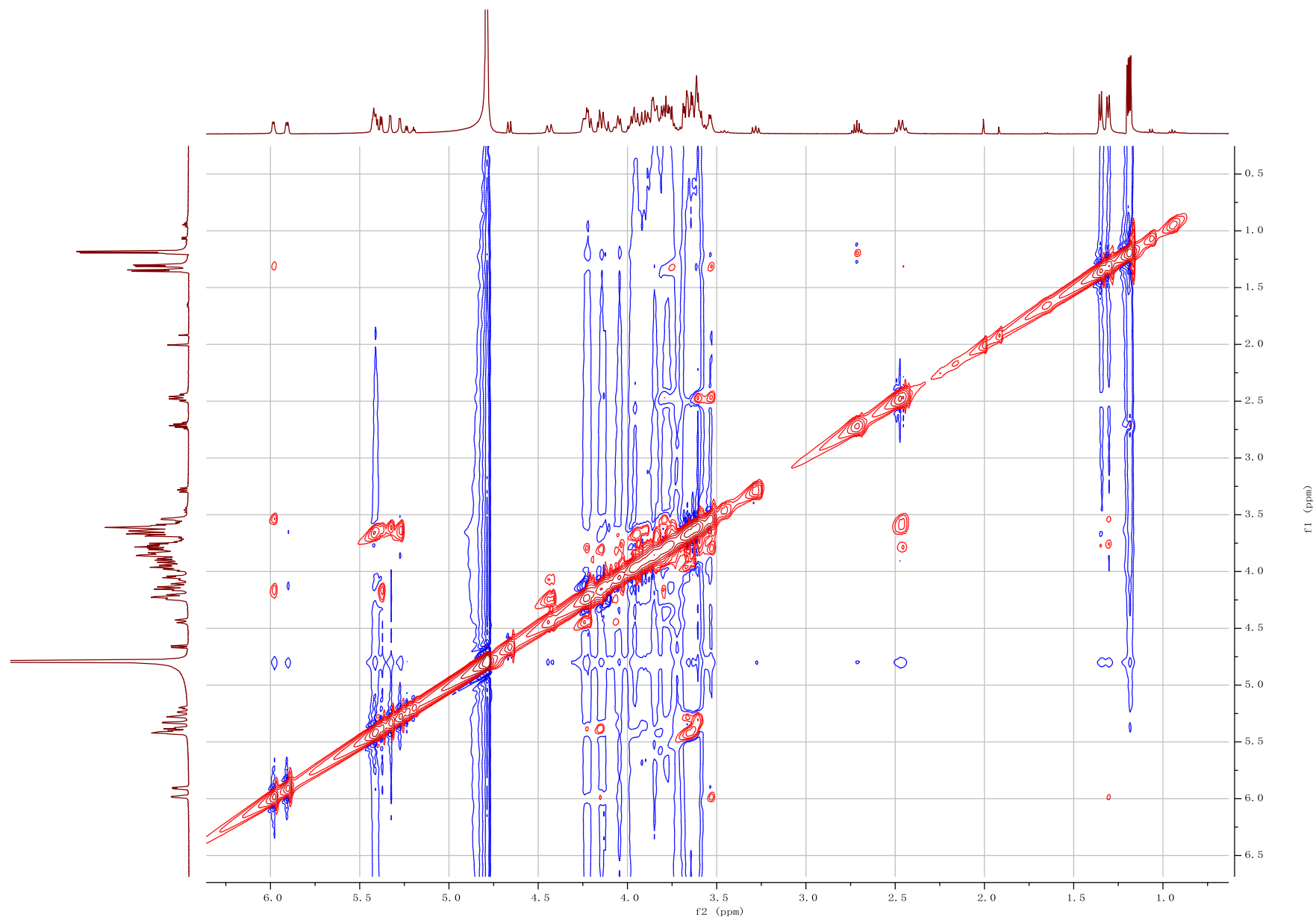


**Figure S65.** HSQC-TOCSY spectrum of compound **12** (500 MHz, D<sub>2</sub>O).

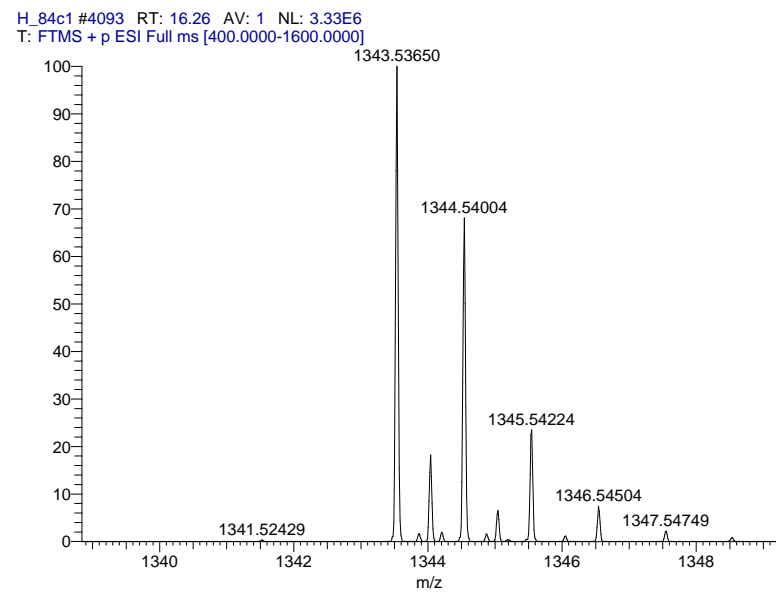




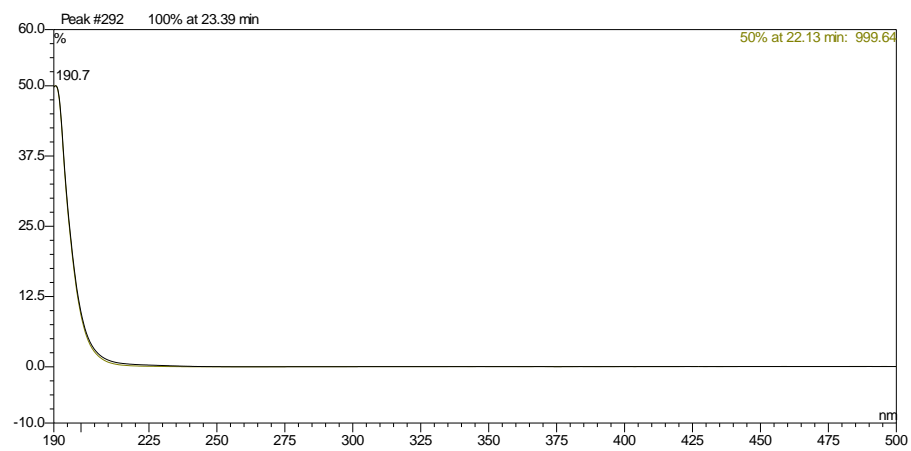
**Figure S66.** HMBC spectrum of compound **12** (500 MHz, D<sub>2</sub>O).



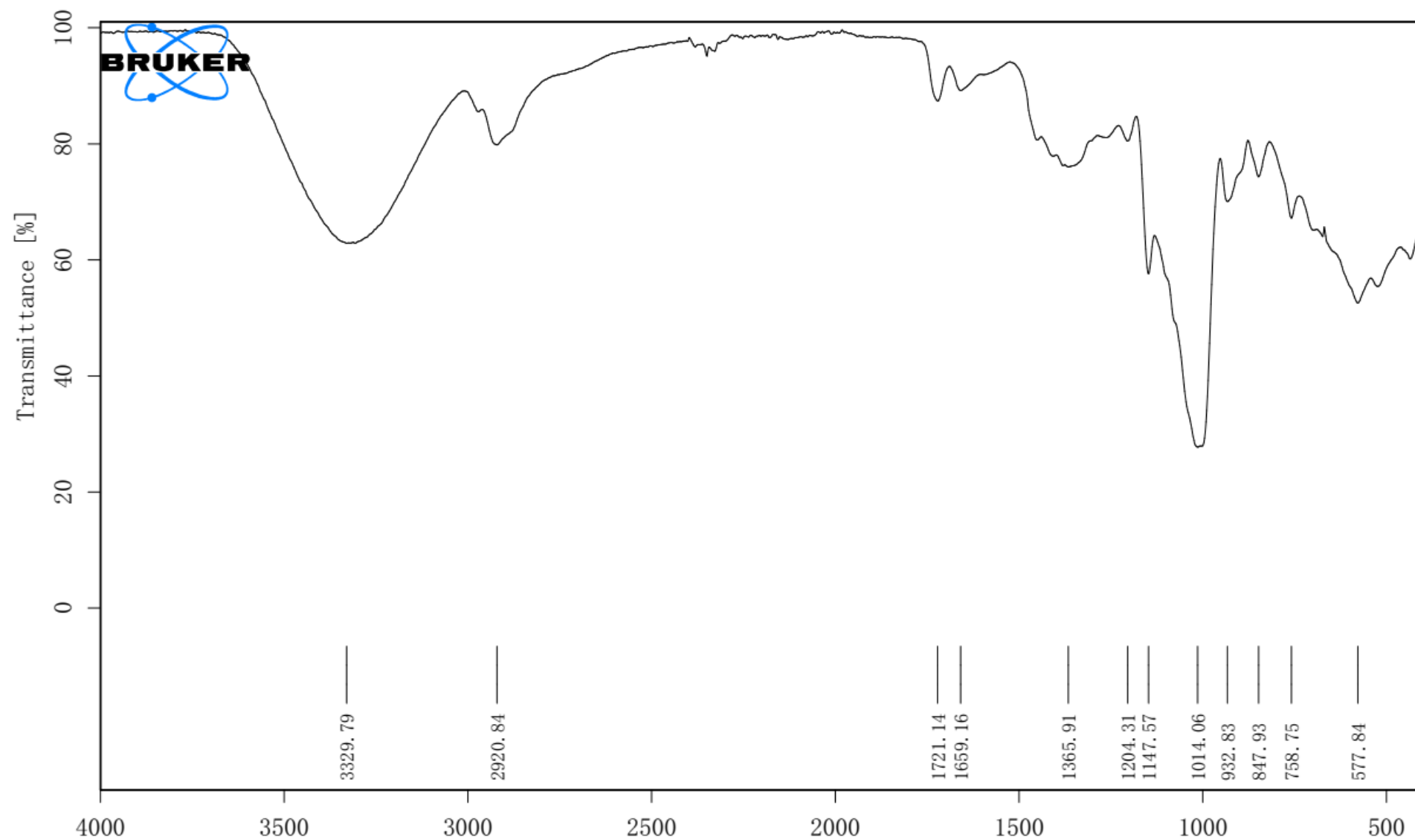
**Figure S67.** NOESY spectrum of compound **12** (500 MHz, D<sub>2</sub>O).



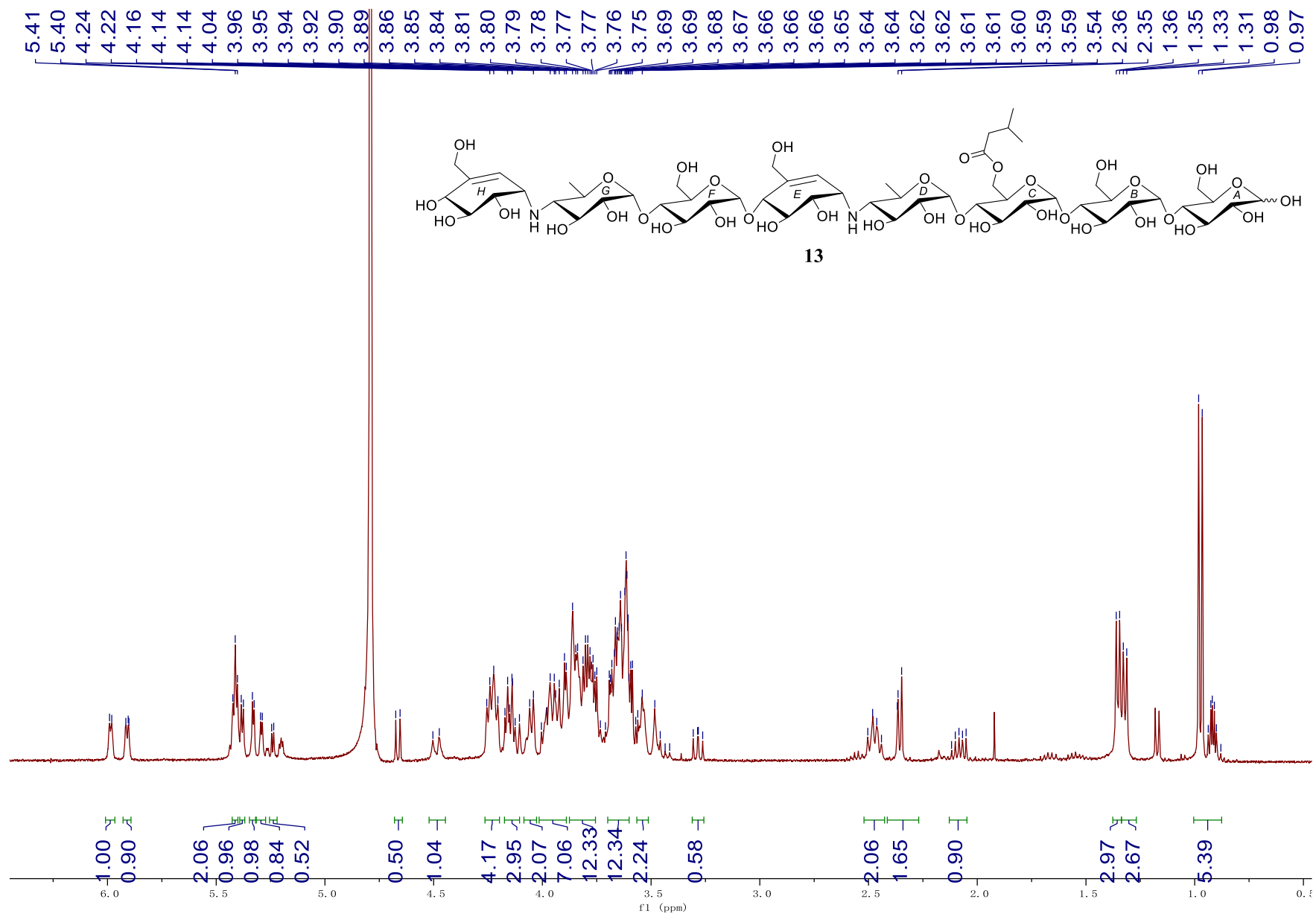
**Figure S68.** HRESIMS spectrum of compound **12**.



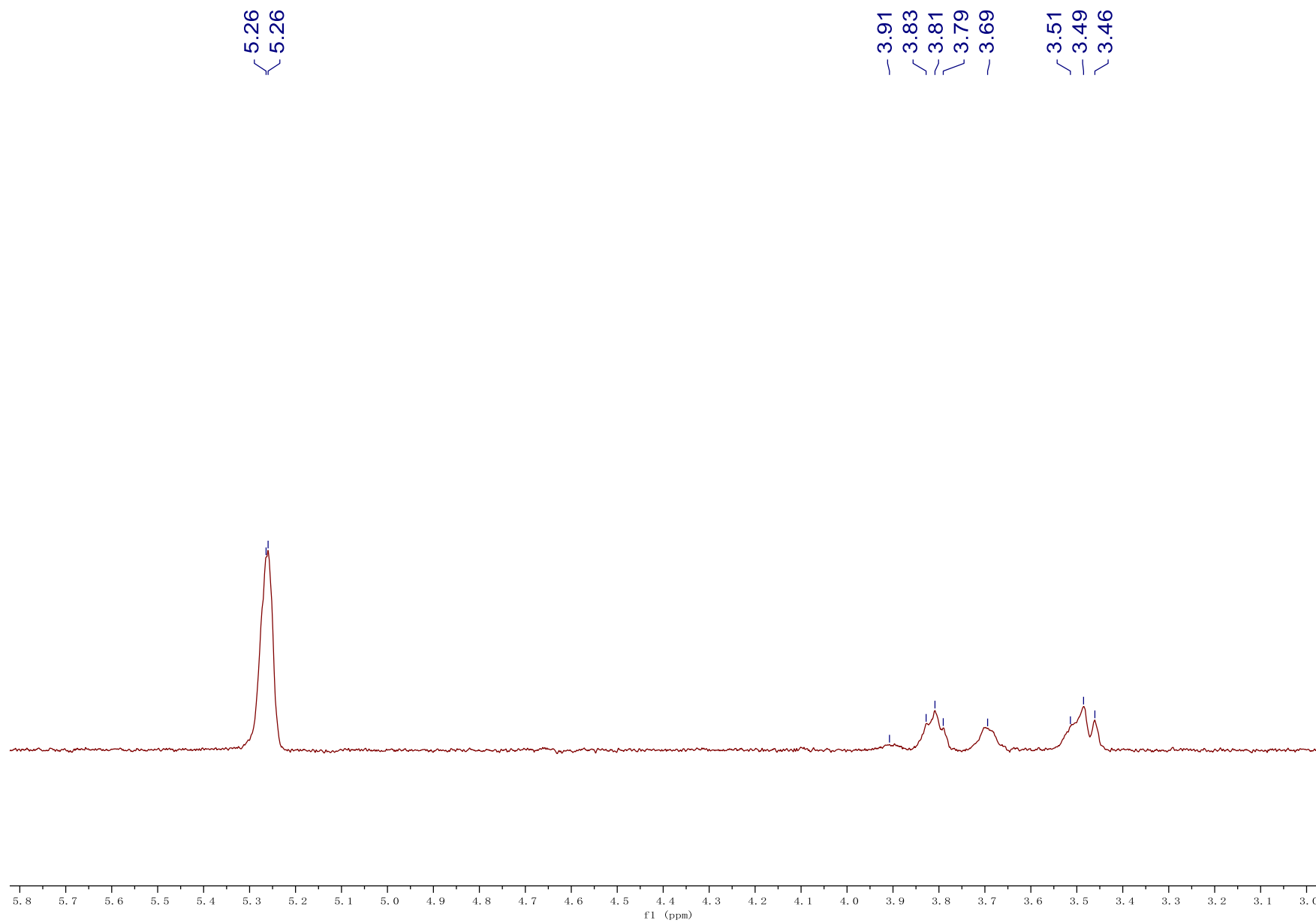
**Figure S69.** UV spectrum of compound **12**.



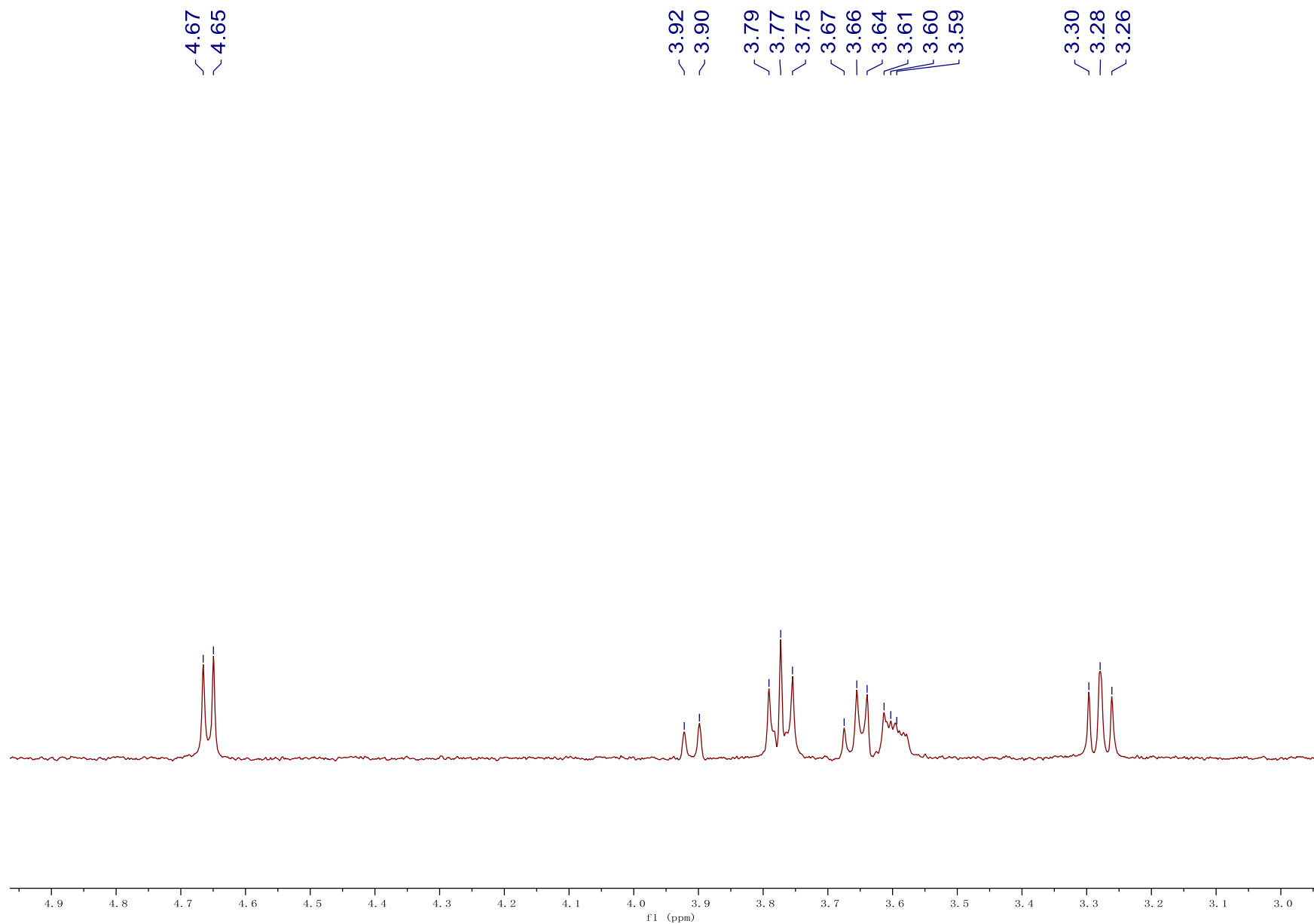
**Figure S70.** IR spectrum of compound **12**.



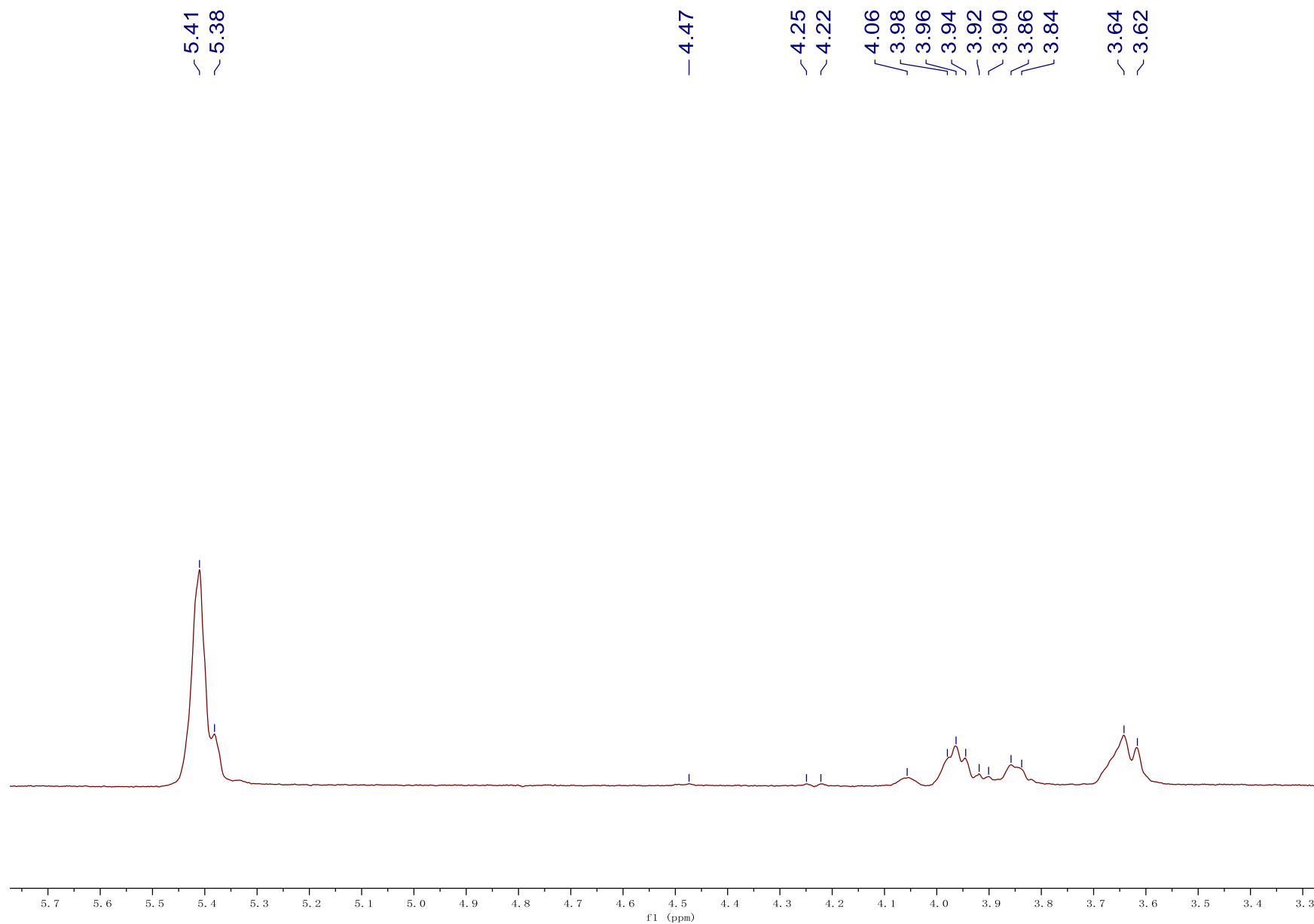
**Figure S71.**  $^1\text{H}$  NMR spectrum of compound **13** (500 MHz,  $\text{D}_2\text{O}$ ).



**Figure S72.** 1D-selective TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.24, H-A1 $\alpha$ ).

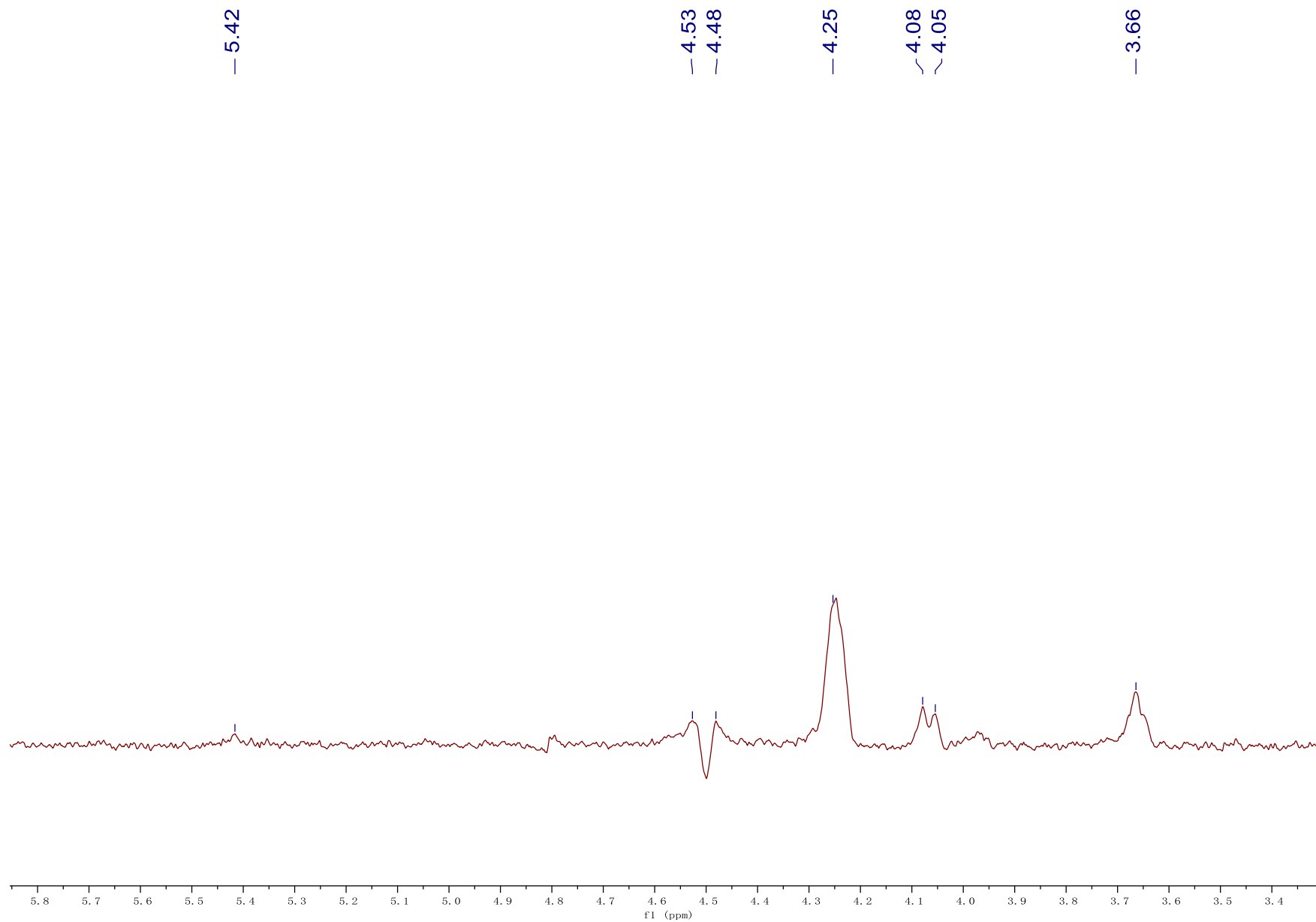


**Figure S73.** 1D-selective TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.66, H-A1 $\beta$ ).

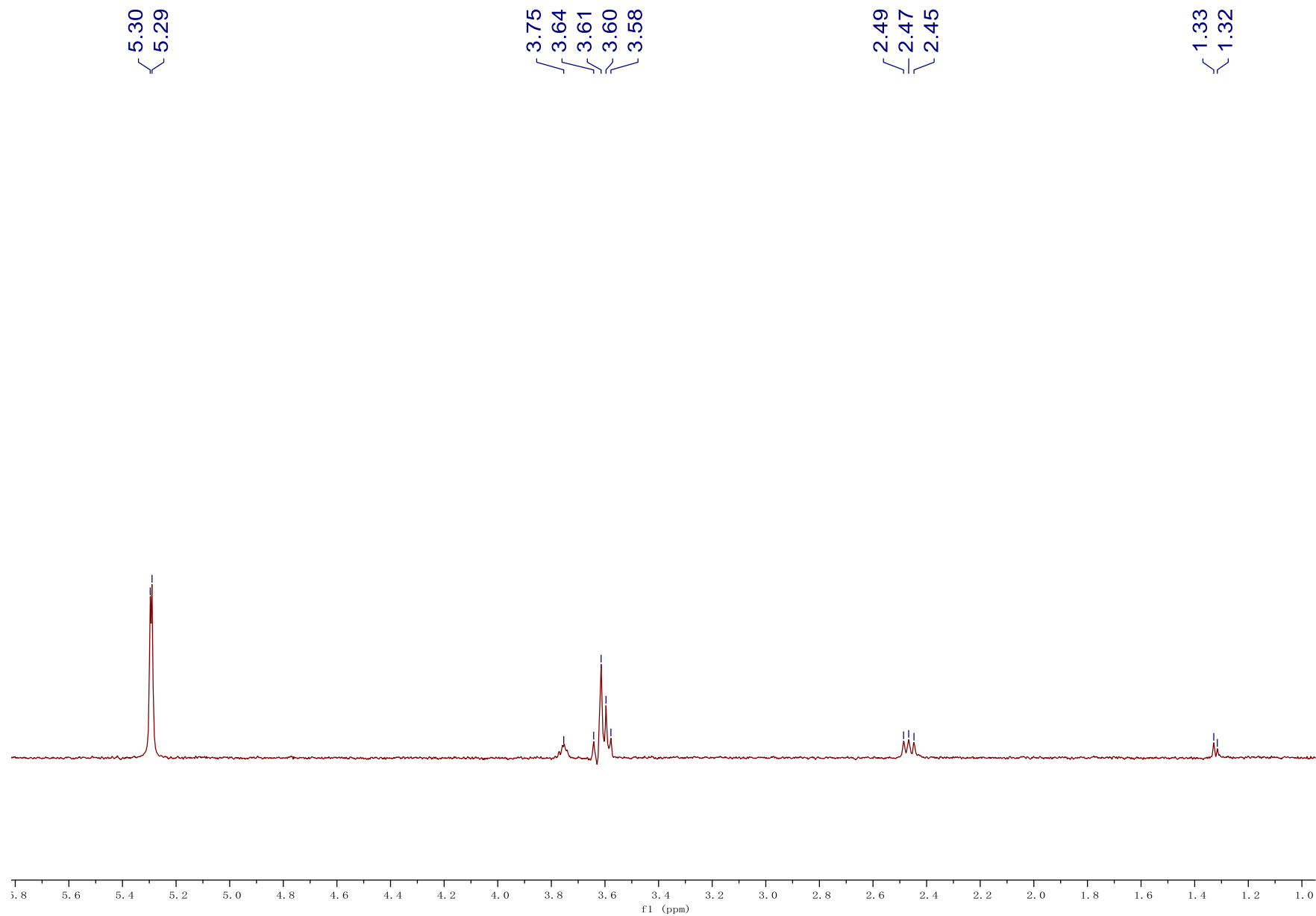


**Figure S74.** 1D-selective TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.41, H-**B1** and H-**C1**).

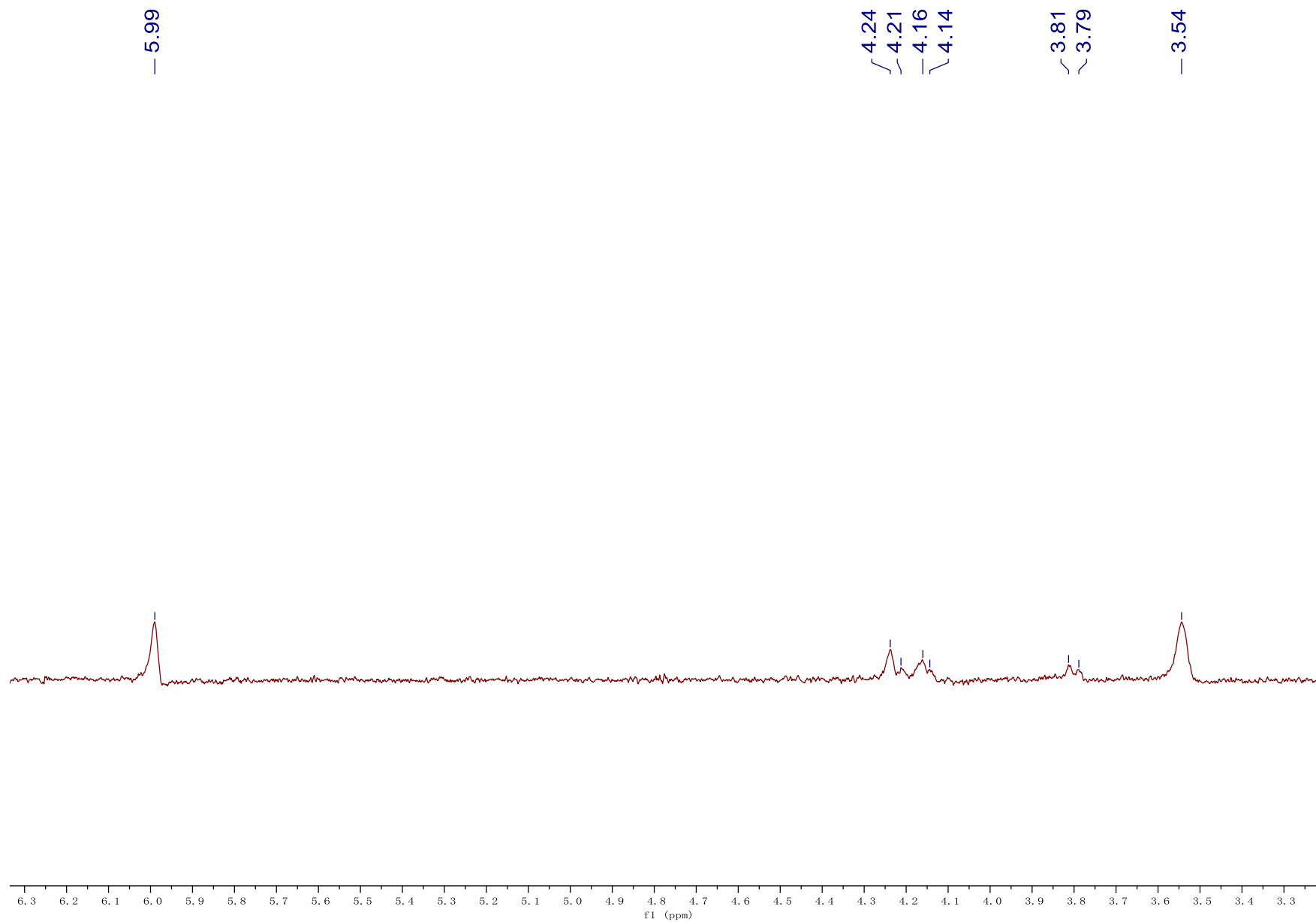




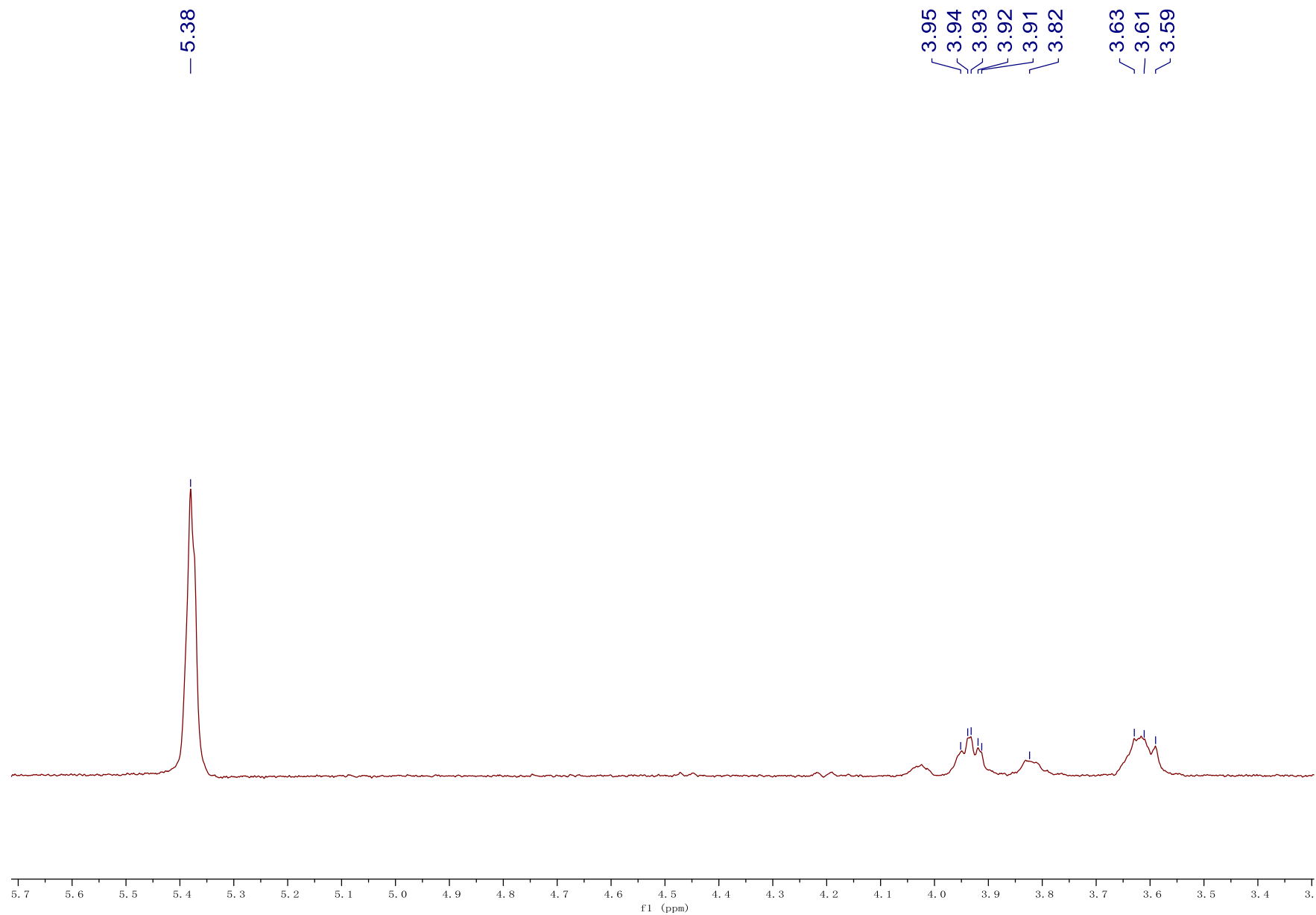
**Figure S75.** 1D-selective TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.49, H-C6).



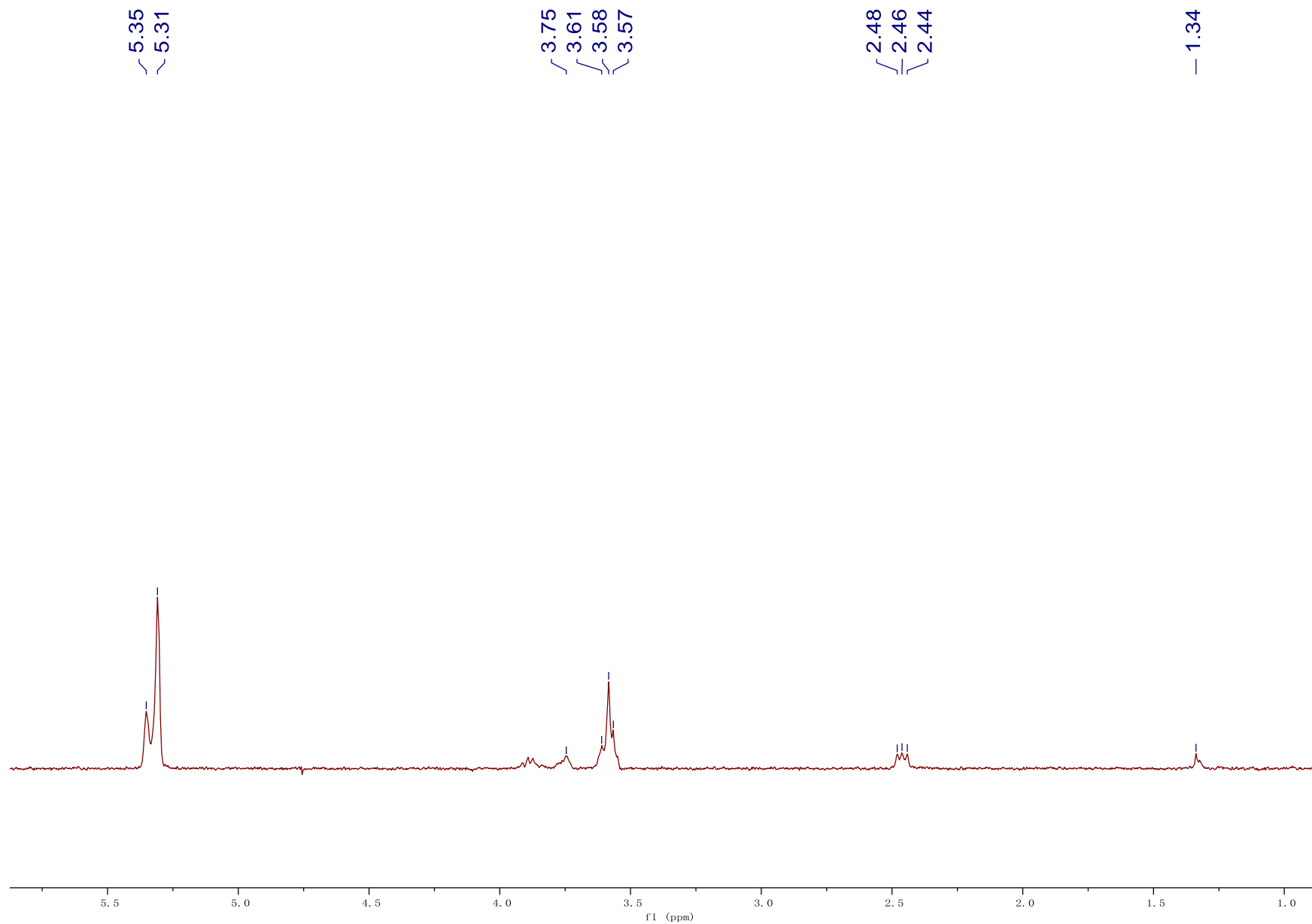
**Figure S76.** 1D-selective TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.29, H-**D1**).



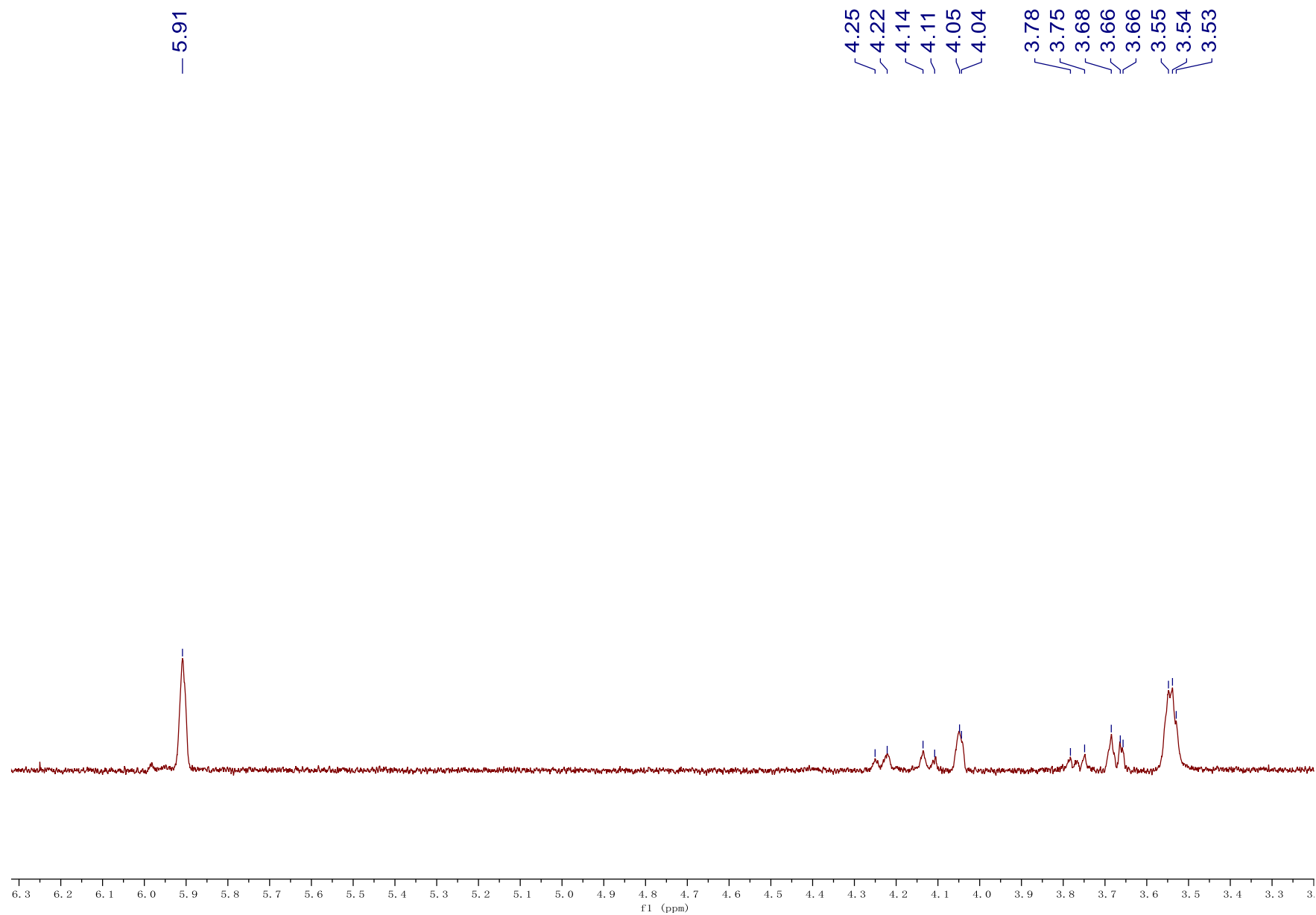
**Figure S77.** 1D-selective TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.99, H-E7).



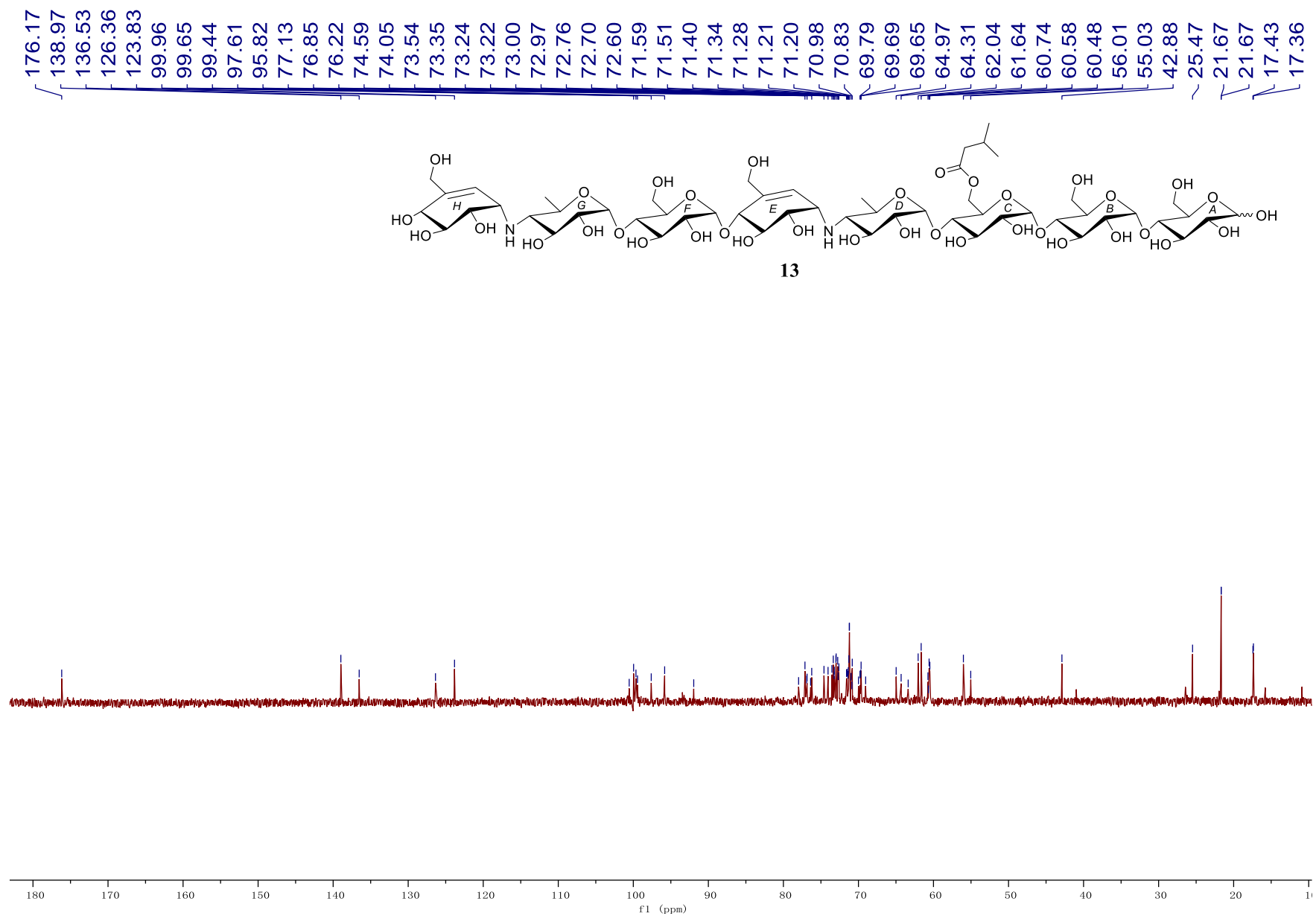
**Figure S78.** 1D-selective TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.38, H-F1).



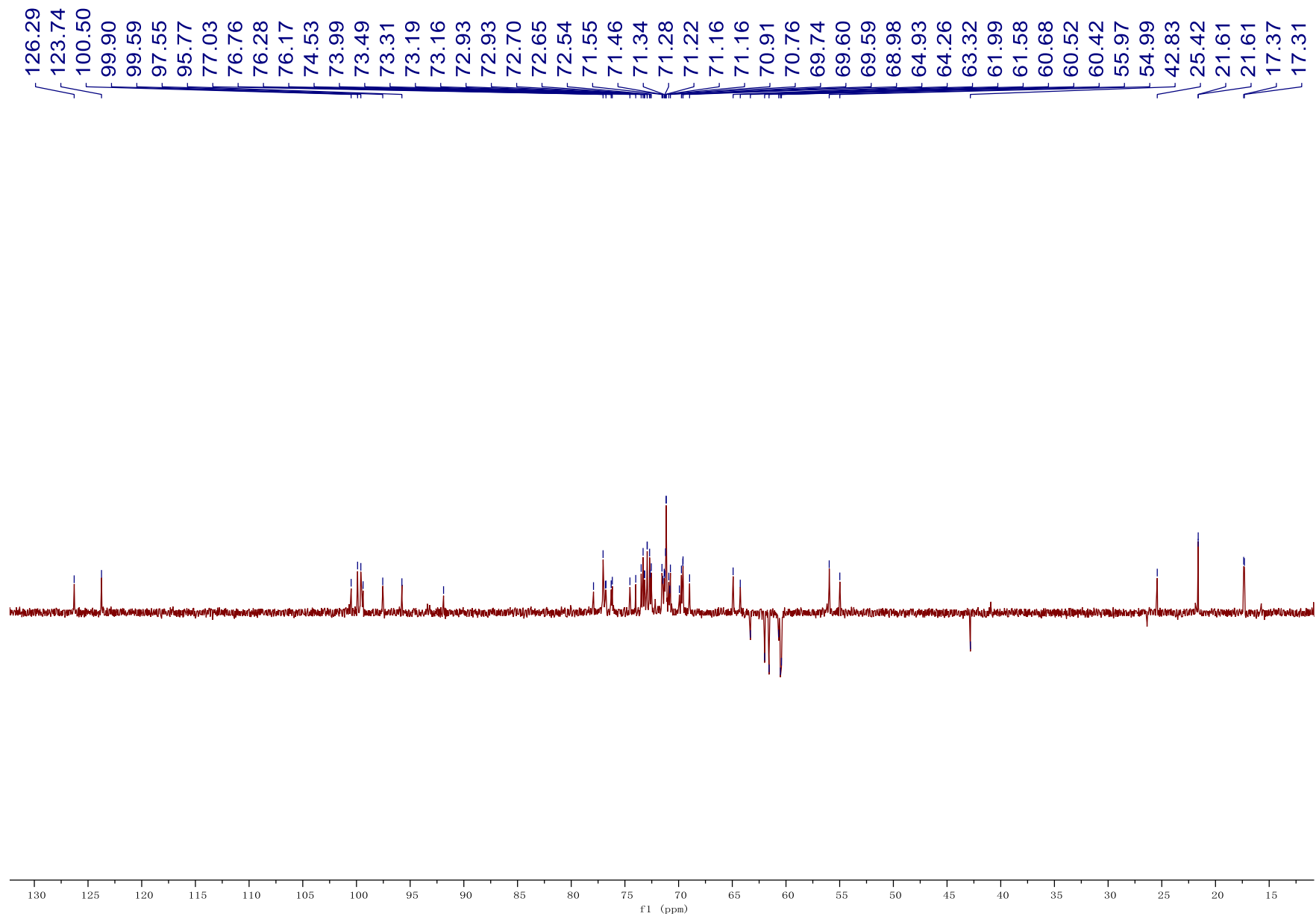
**Figure S79.** 1D-selective TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.33, H-**G1**).



**Figure S80.** 1D-selective TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.91, H-**H7**).

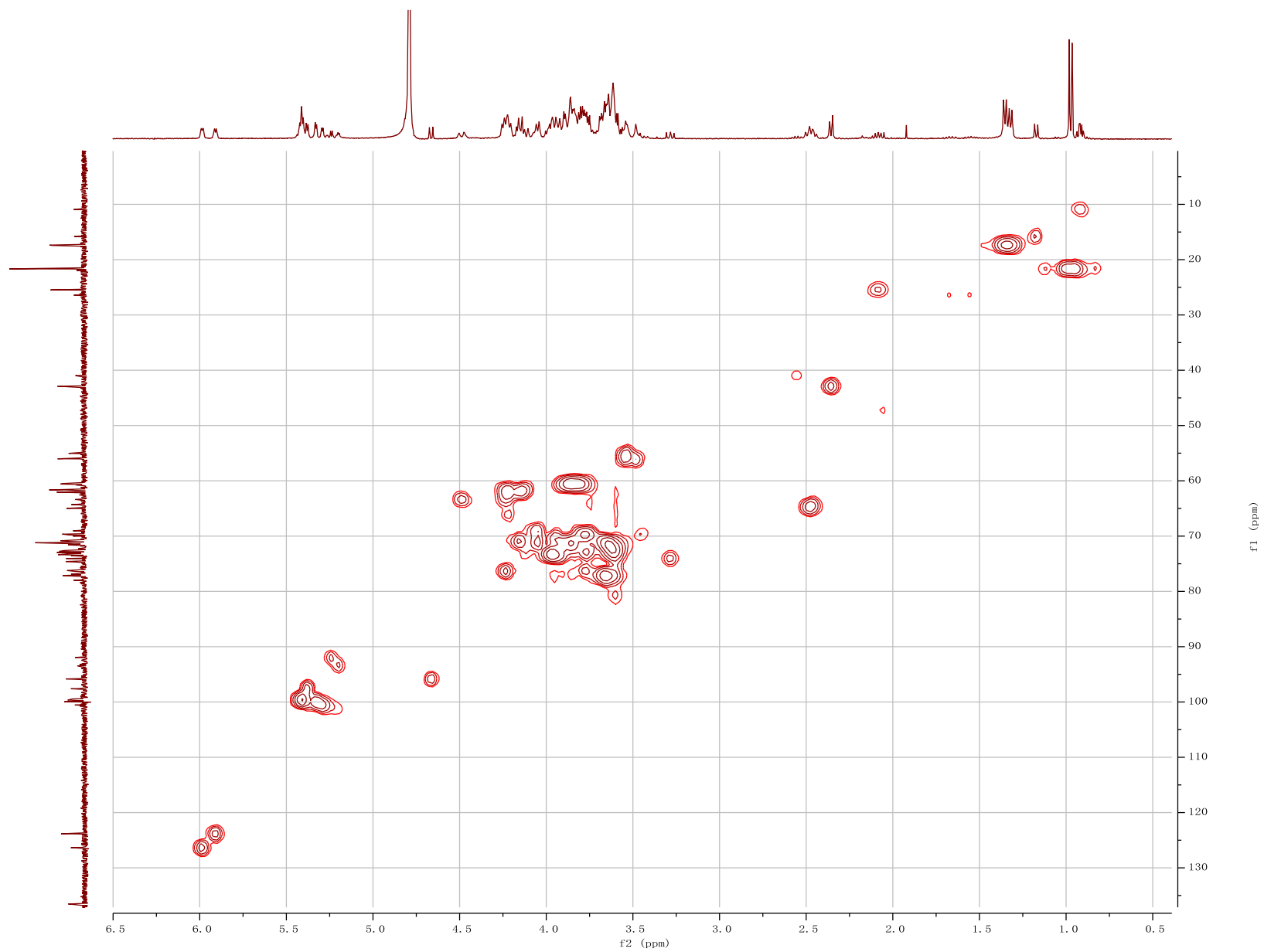


**Figure S81.**  $^{13}\text{C}$  NMR spectrum of compound **13** (125 MHz,  $\text{D}_2\text{O}$ ).

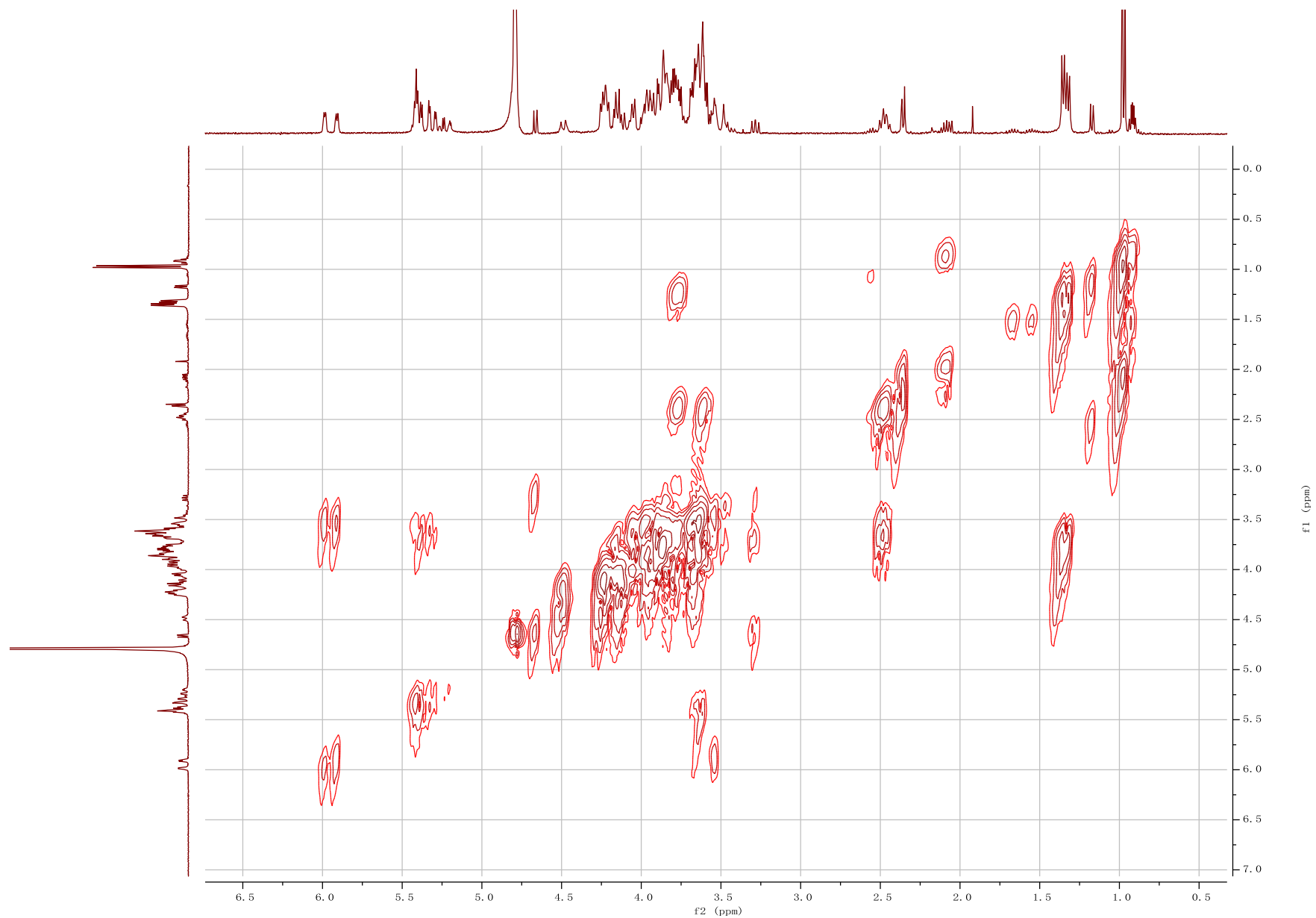


**Figure S82.** DEPT-135 spectrum of compound **13** (125 MHz, D<sub>2</sub>O).

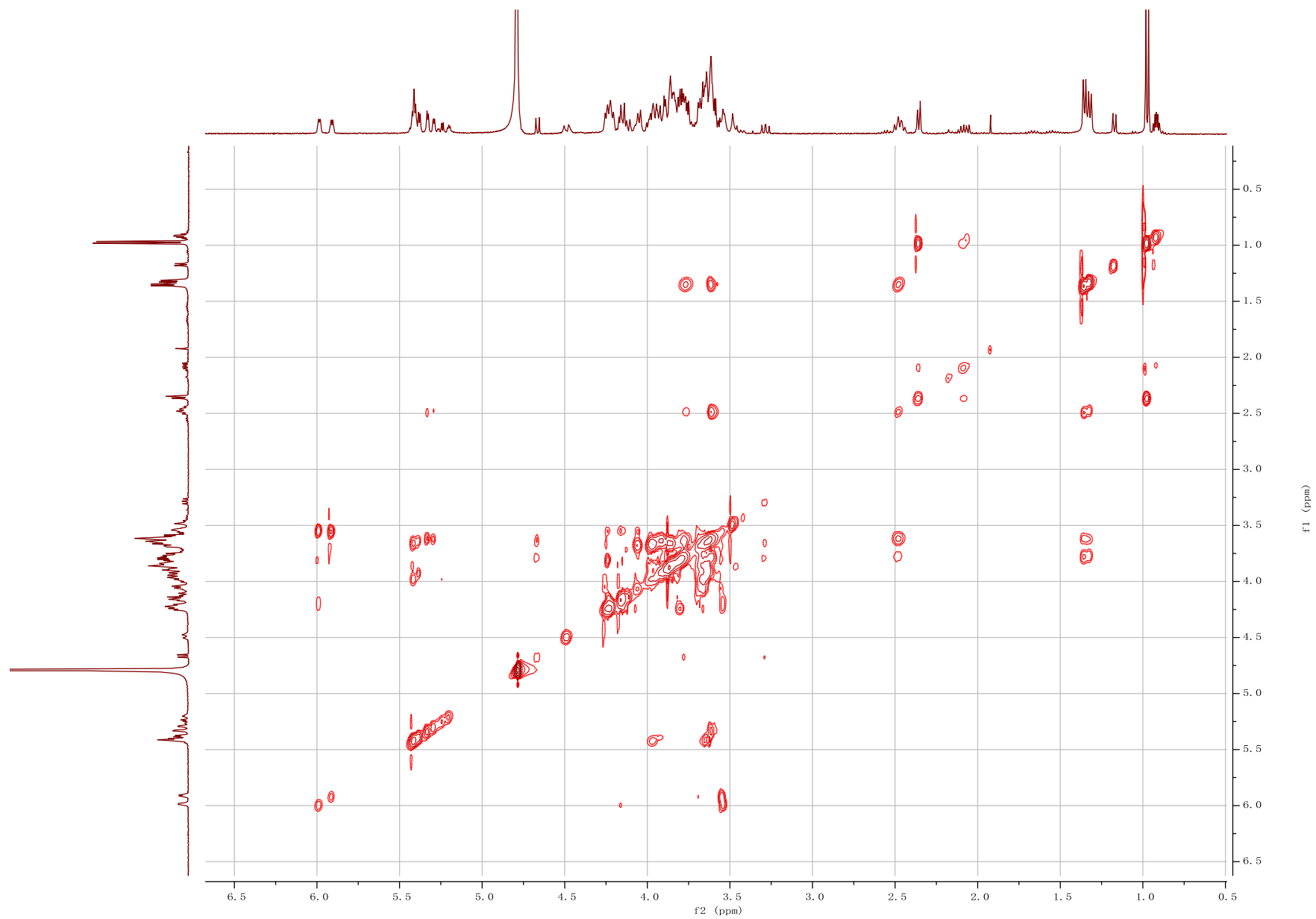




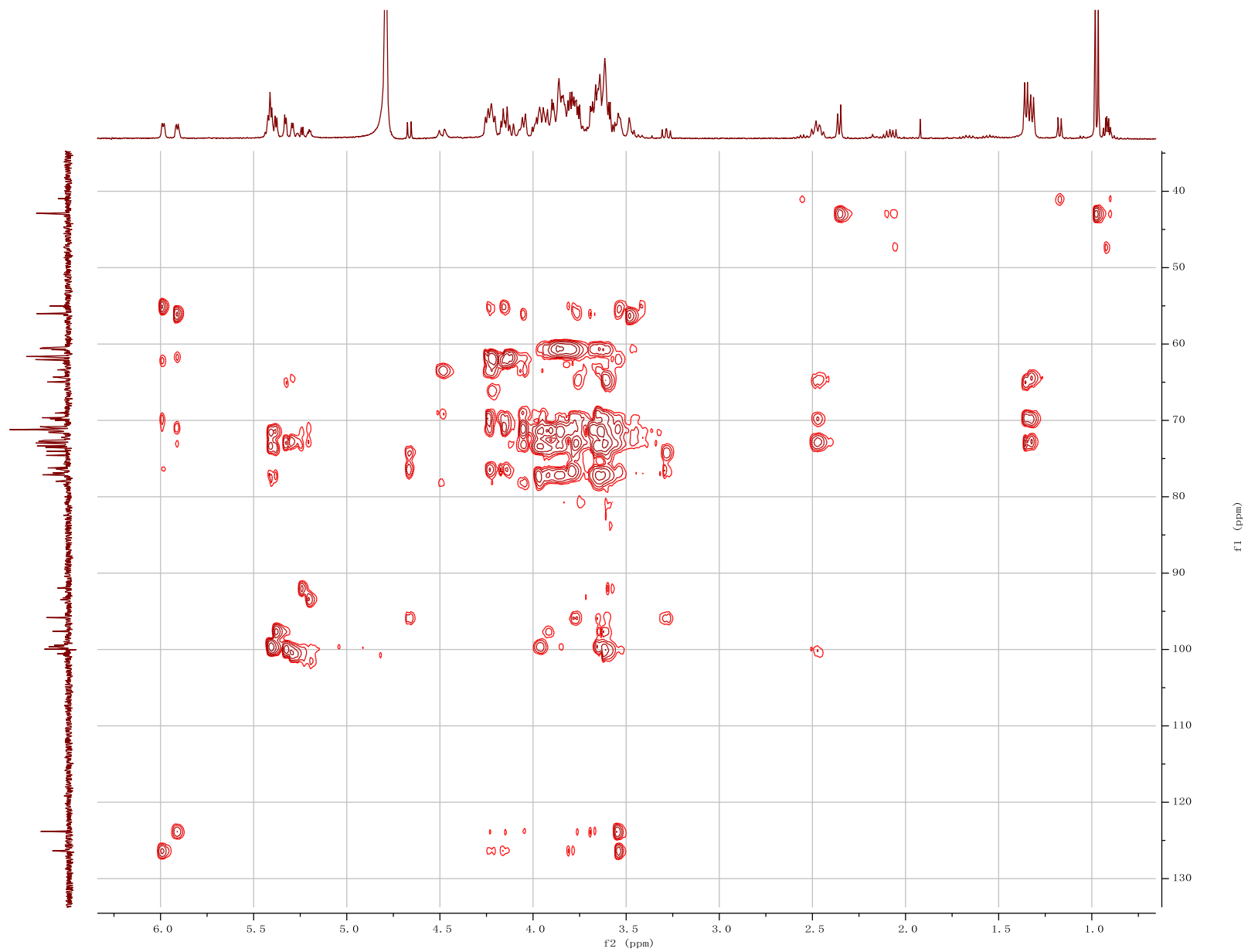
**Figure S83.** HSQC spectrum of compound **13** (500 MHz, D<sub>2</sub>O).



**Figure S84.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound **13** (500 MHz,  $\text{D}_2\text{O}$ ).



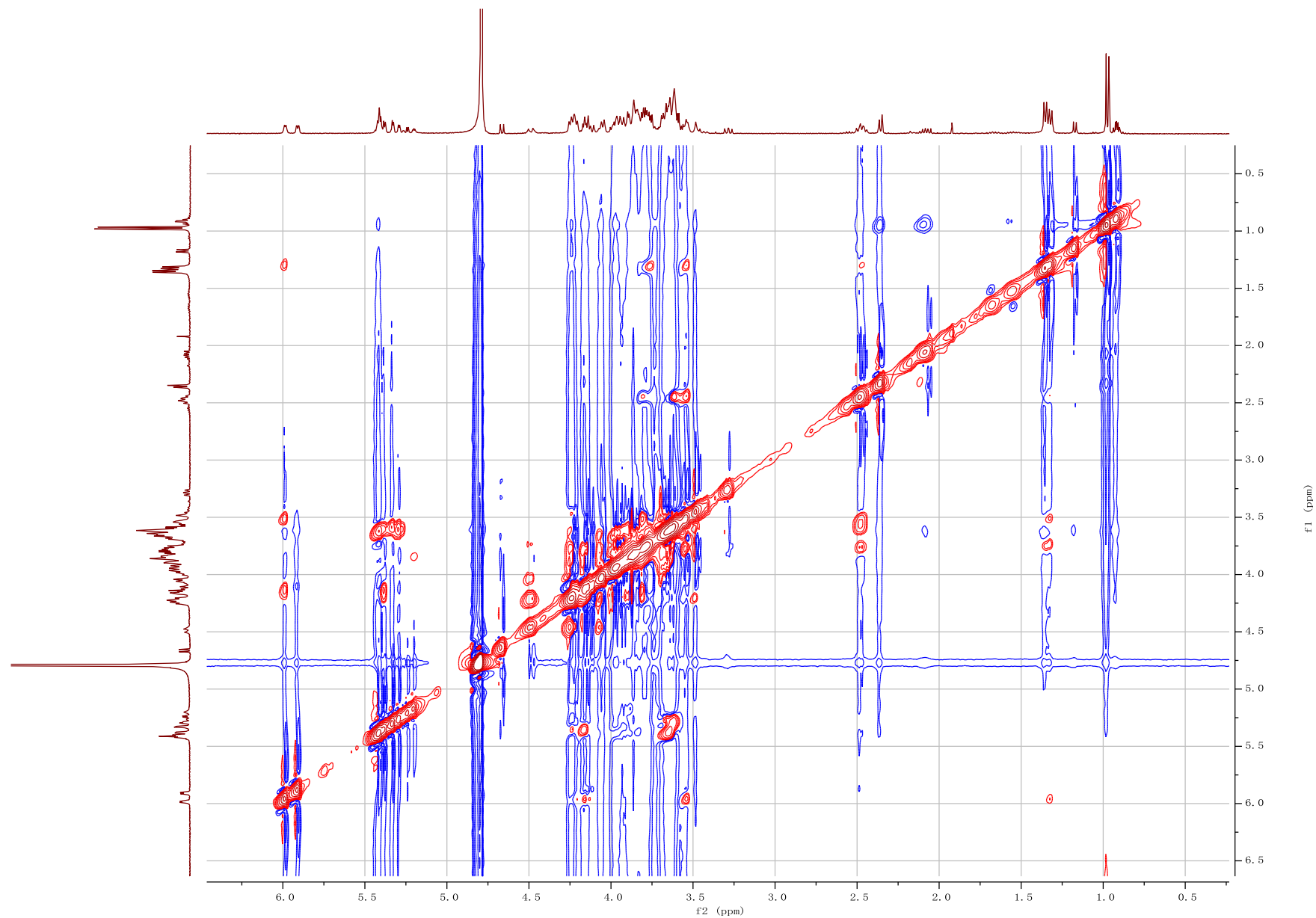
**Figure S85.** 2D-TOCSY spectrum of compound **13** (500 MHz, D<sub>2</sub>O).



**Figure S86.** HSQC-TOCSY spectrum of compound **13** (500 MHz,  $\text{D}_2\text{O}$ ).

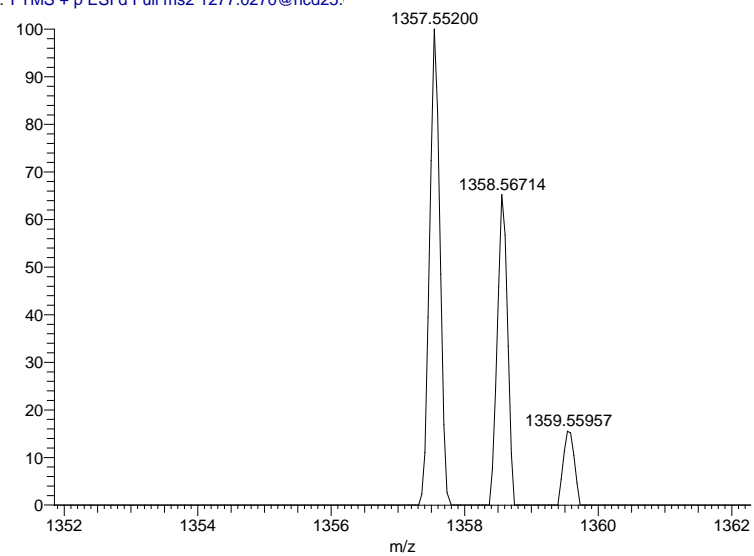


**Figure S87.** HMBC spectrum of compound **13** (500 MHz, D<sub>2</sub>O).

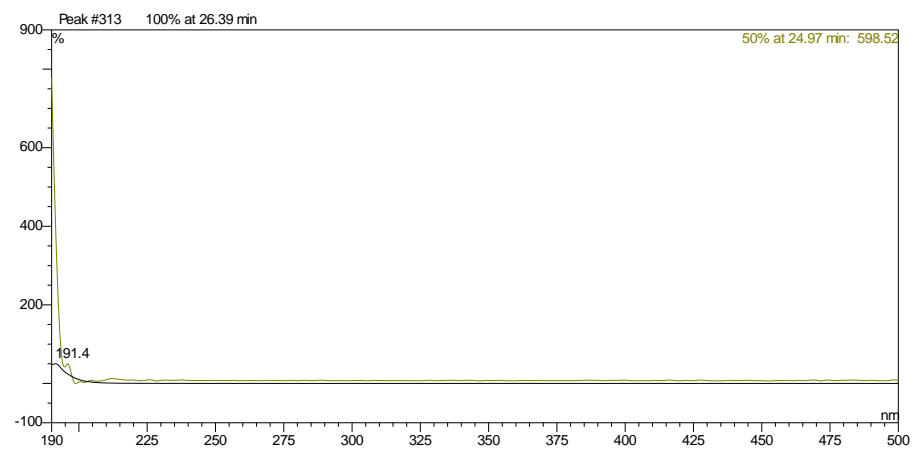


**Figure S88.** NOESY spectrum of compound **13** (500 MHz, D<sub>2</sub>O).

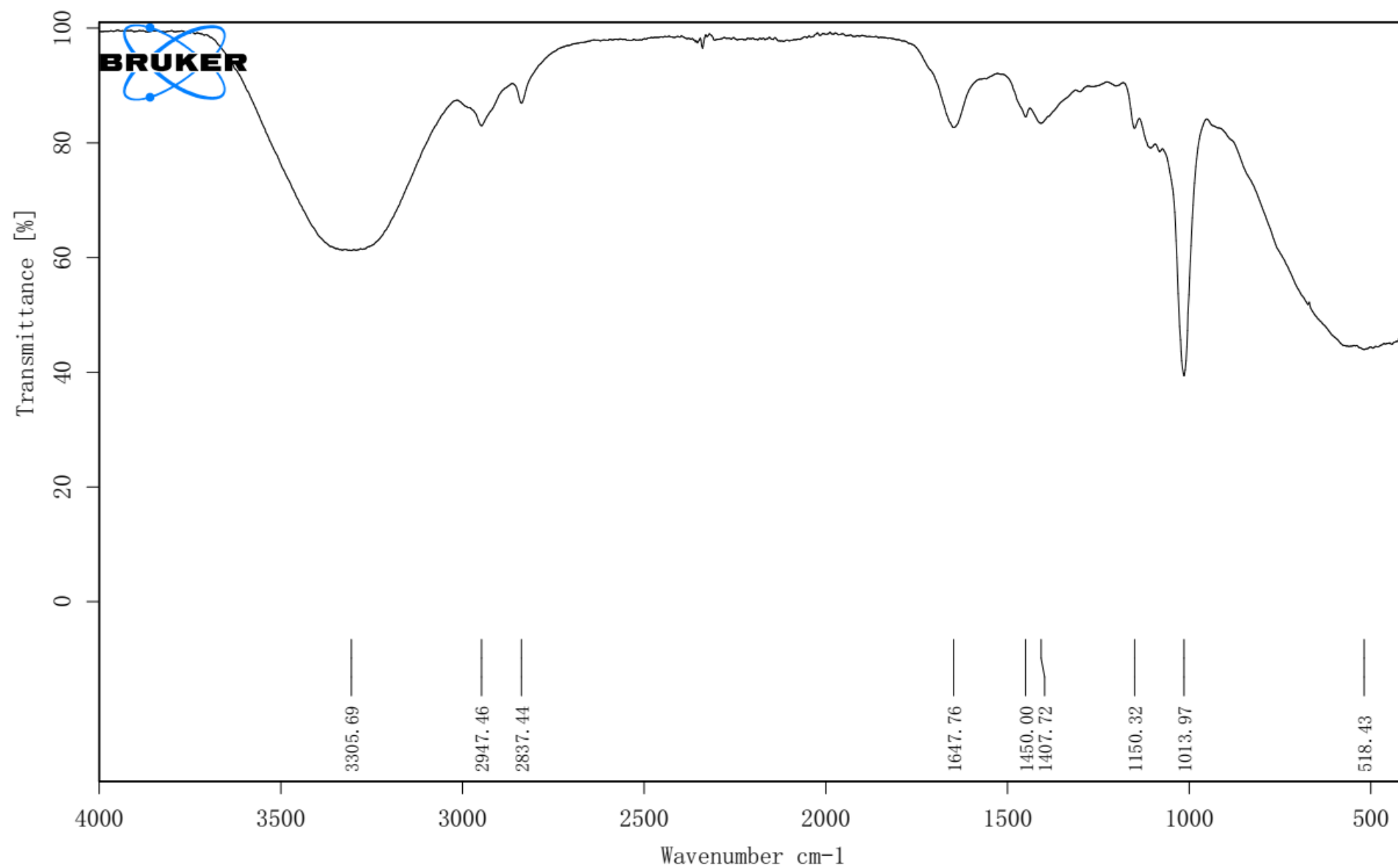
H\_40a #3804 RT: 12.74 AV: 1 NL: 5.41E4  
T: FTMS + p ESI d Full ms2 1277.0270@hcd25



**Figure S89.** HRESIMS spectrum of compound **13**.

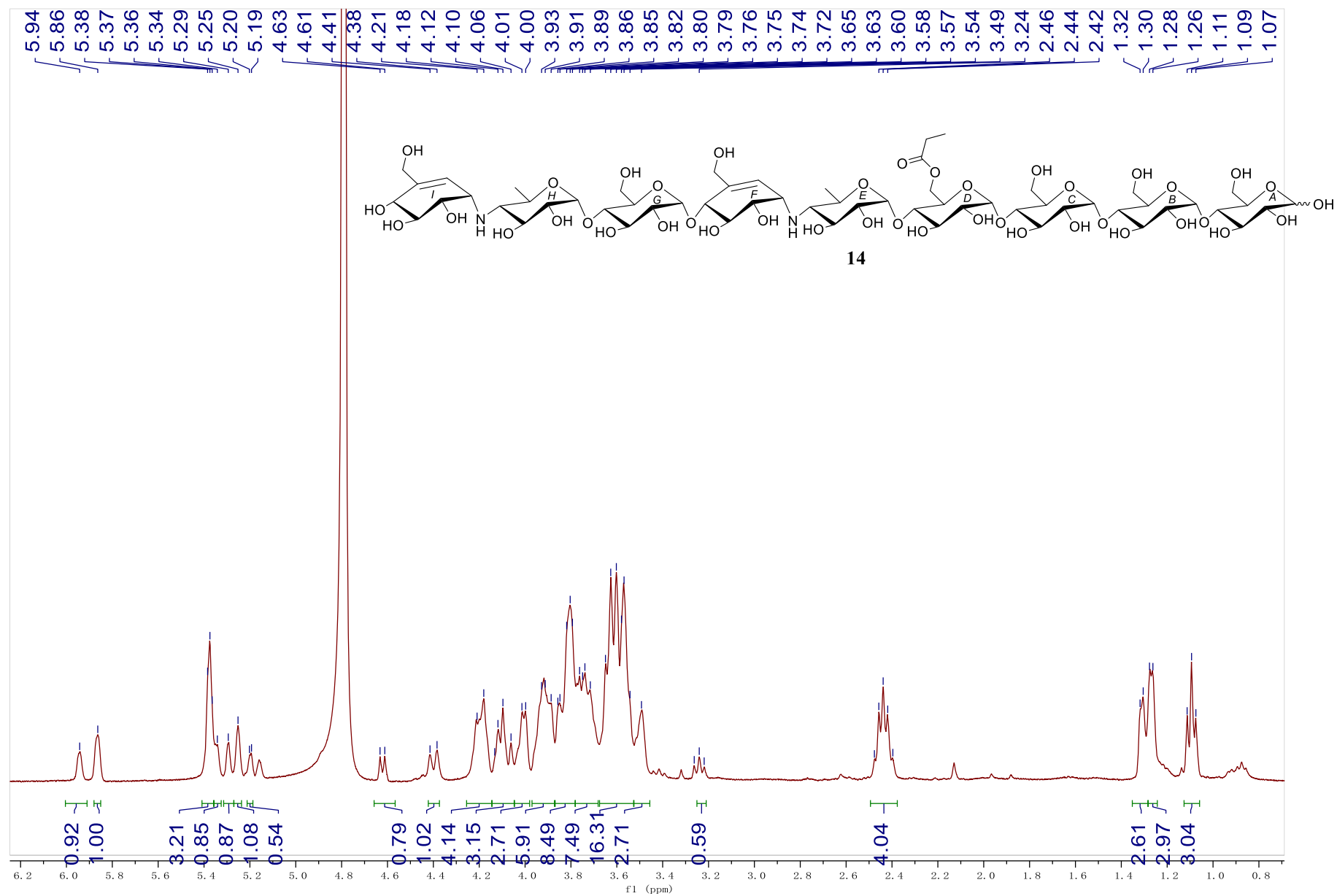


**Figure S90.** UV spectrum of compound **13**.

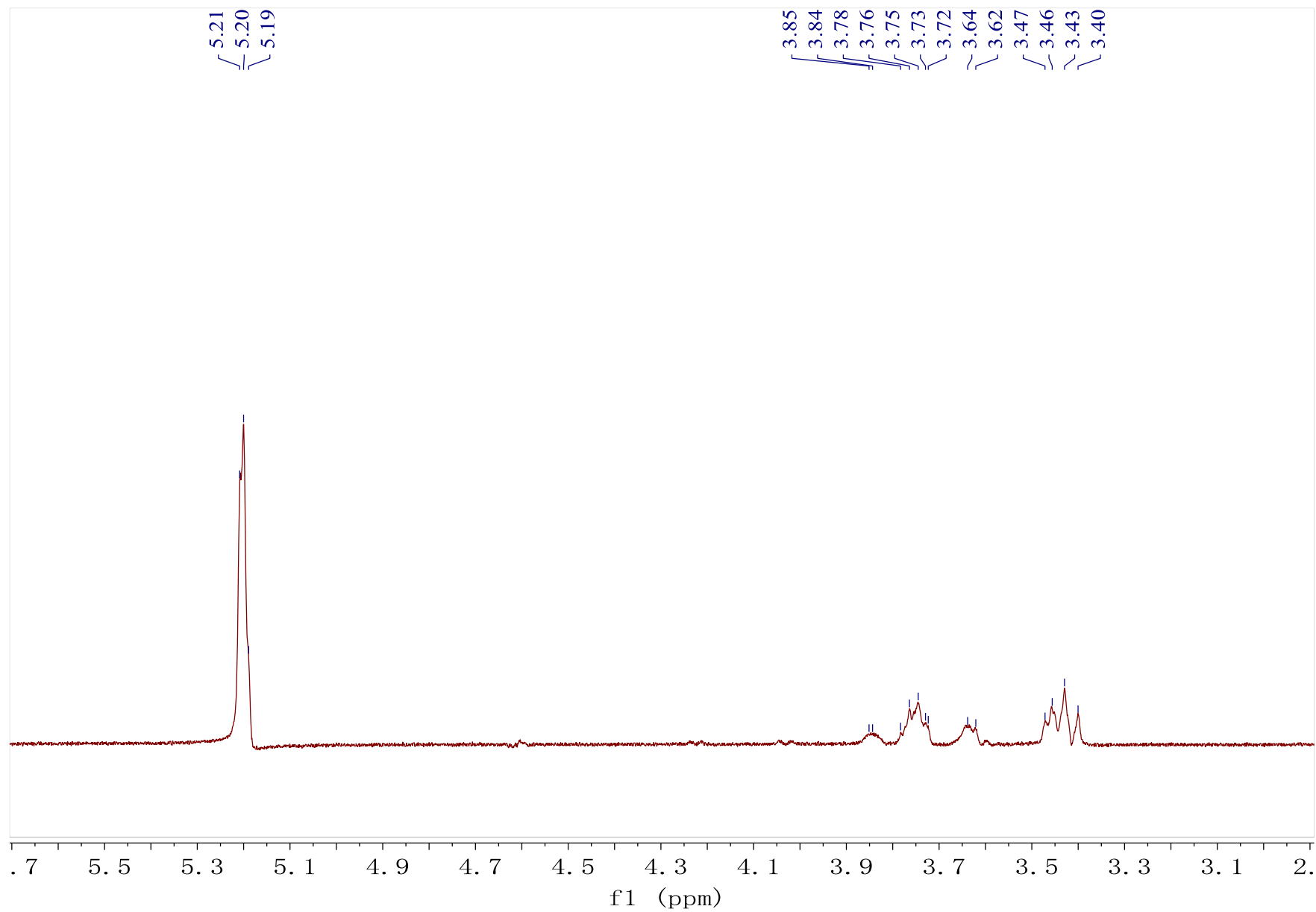


**Figure S91.** IR spectrum of compound **13**.

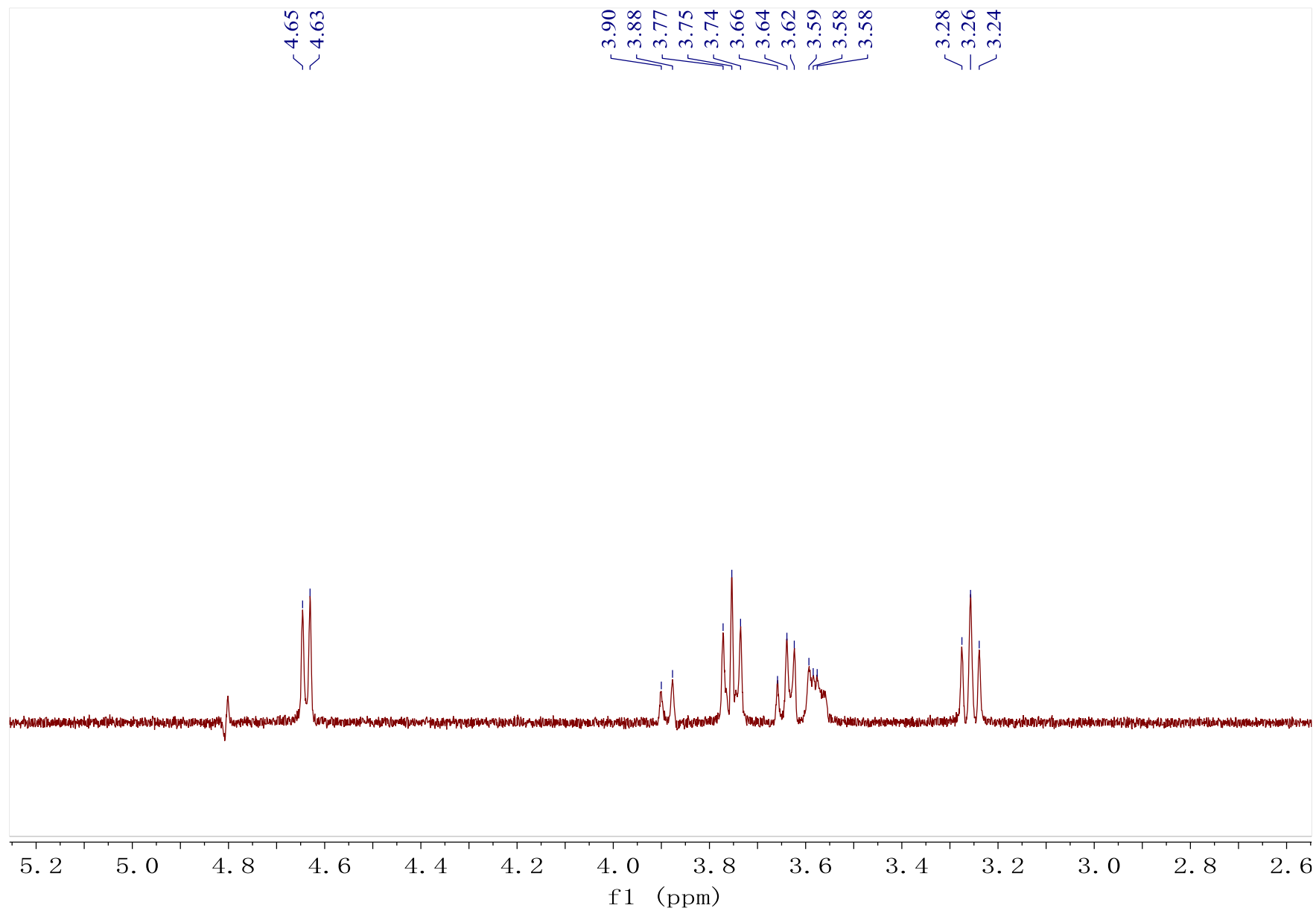




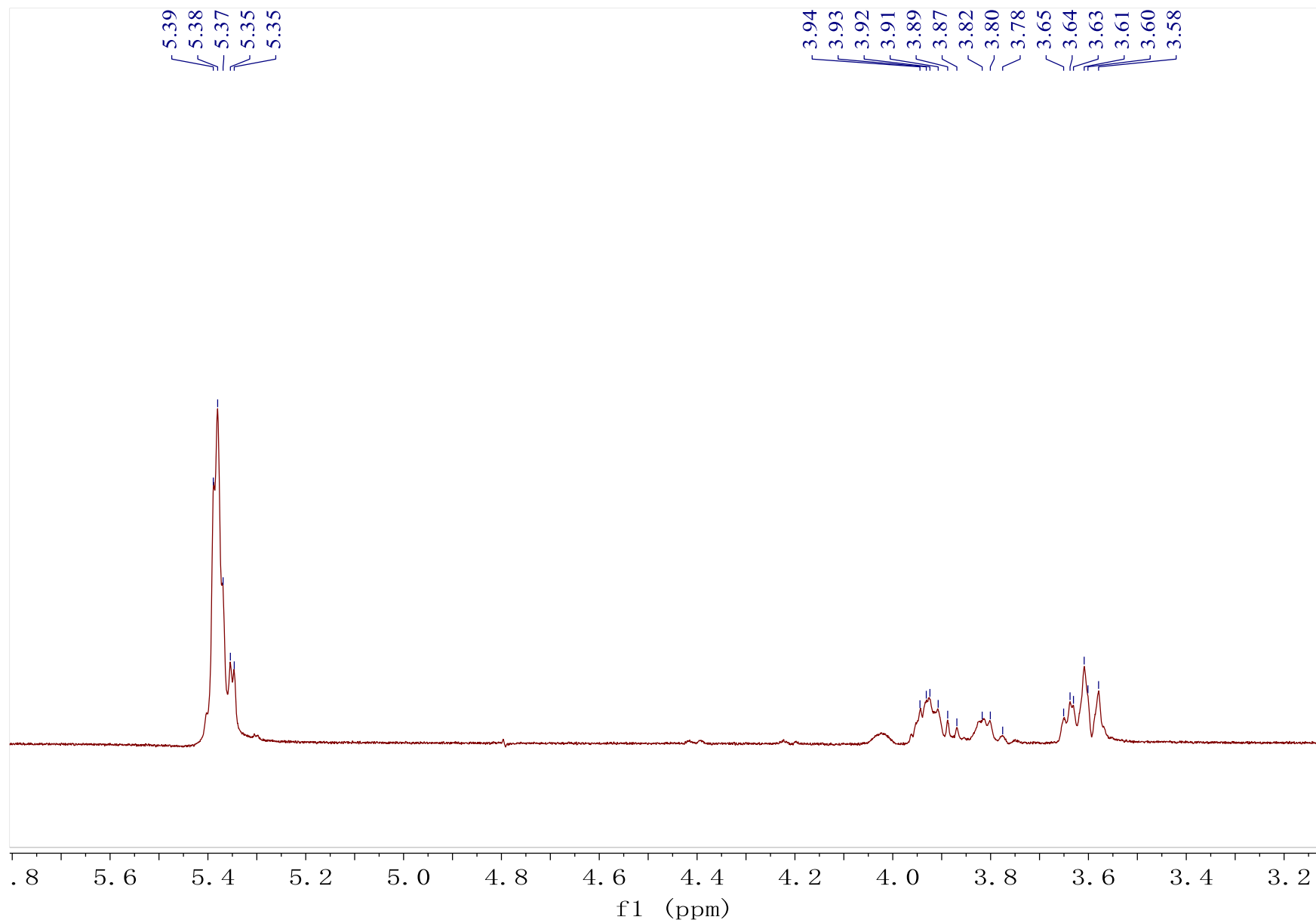
**Figure S92.**  $^1\text{H}$  NMR spectrum of compound **14** (500 MHz,  $\text{D}_2\text{O}$ ).



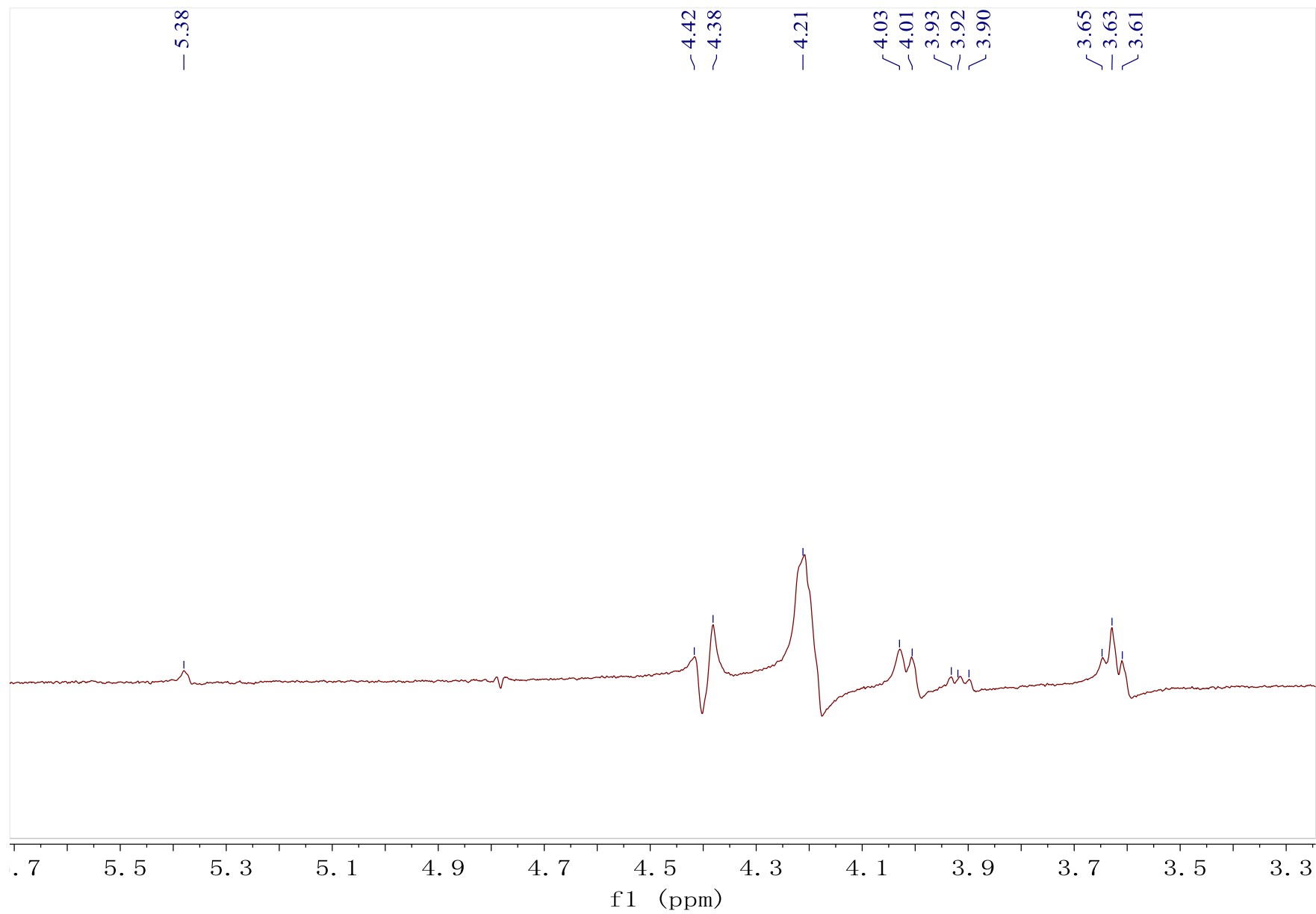
**Figure S93.** 1D-selective TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.20, H-A1 $\alpha$ ).



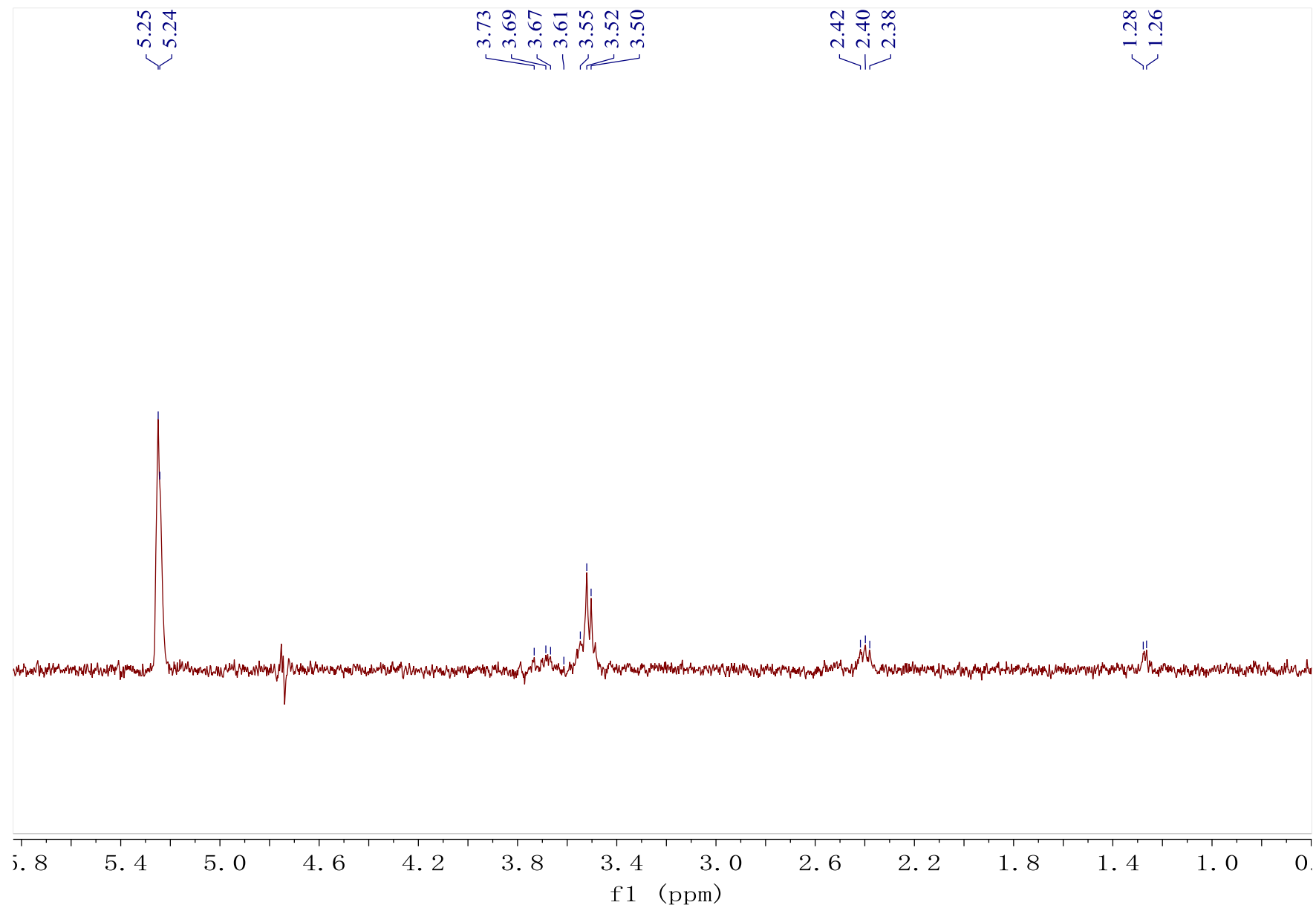
**Figure S94.** 1D-selective TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.62, H-A1 $\beta$ ).



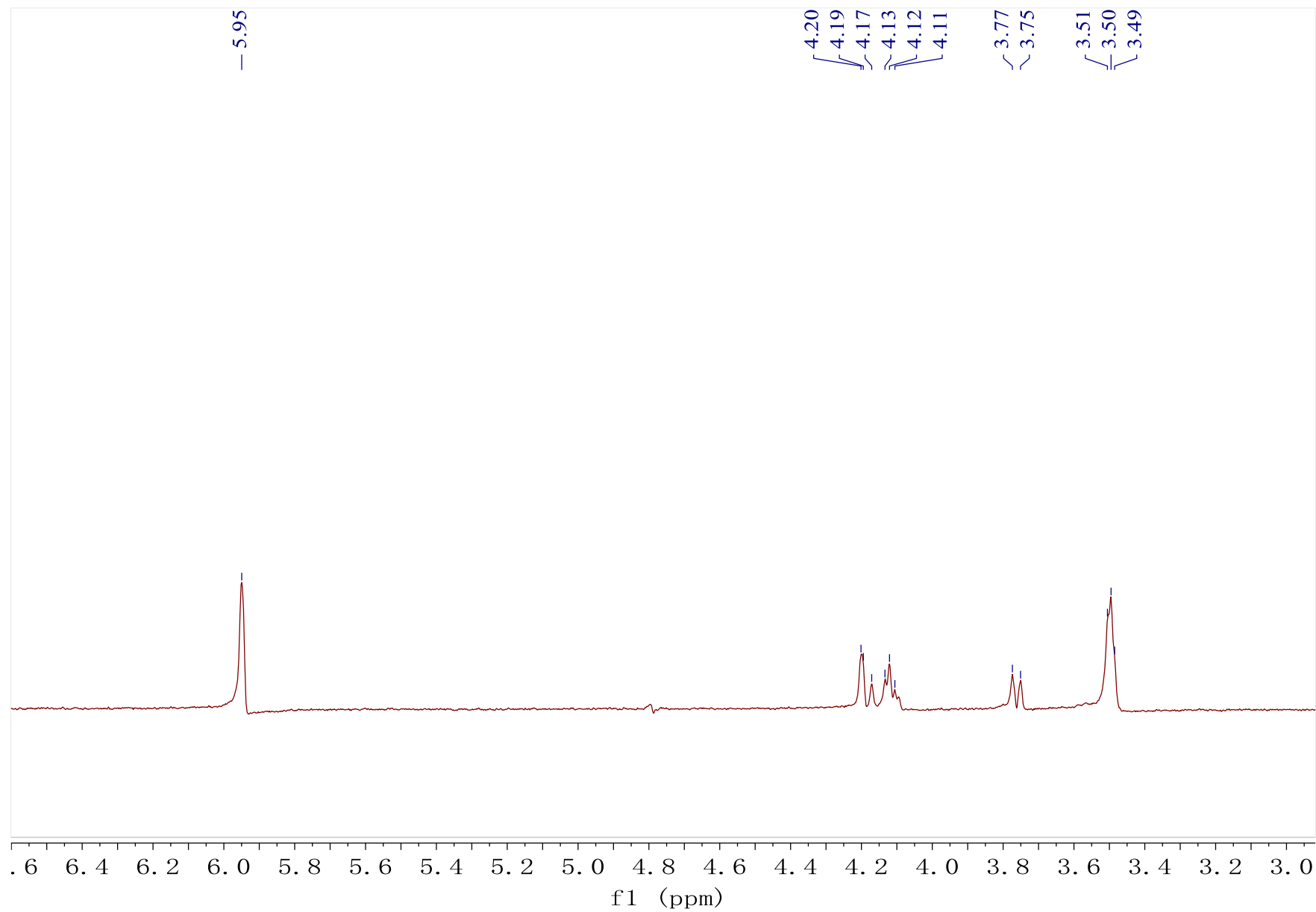
**Figure S95.** 1D-selective TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.38, H-**B1**, H-**C1**, and H-**D1**).



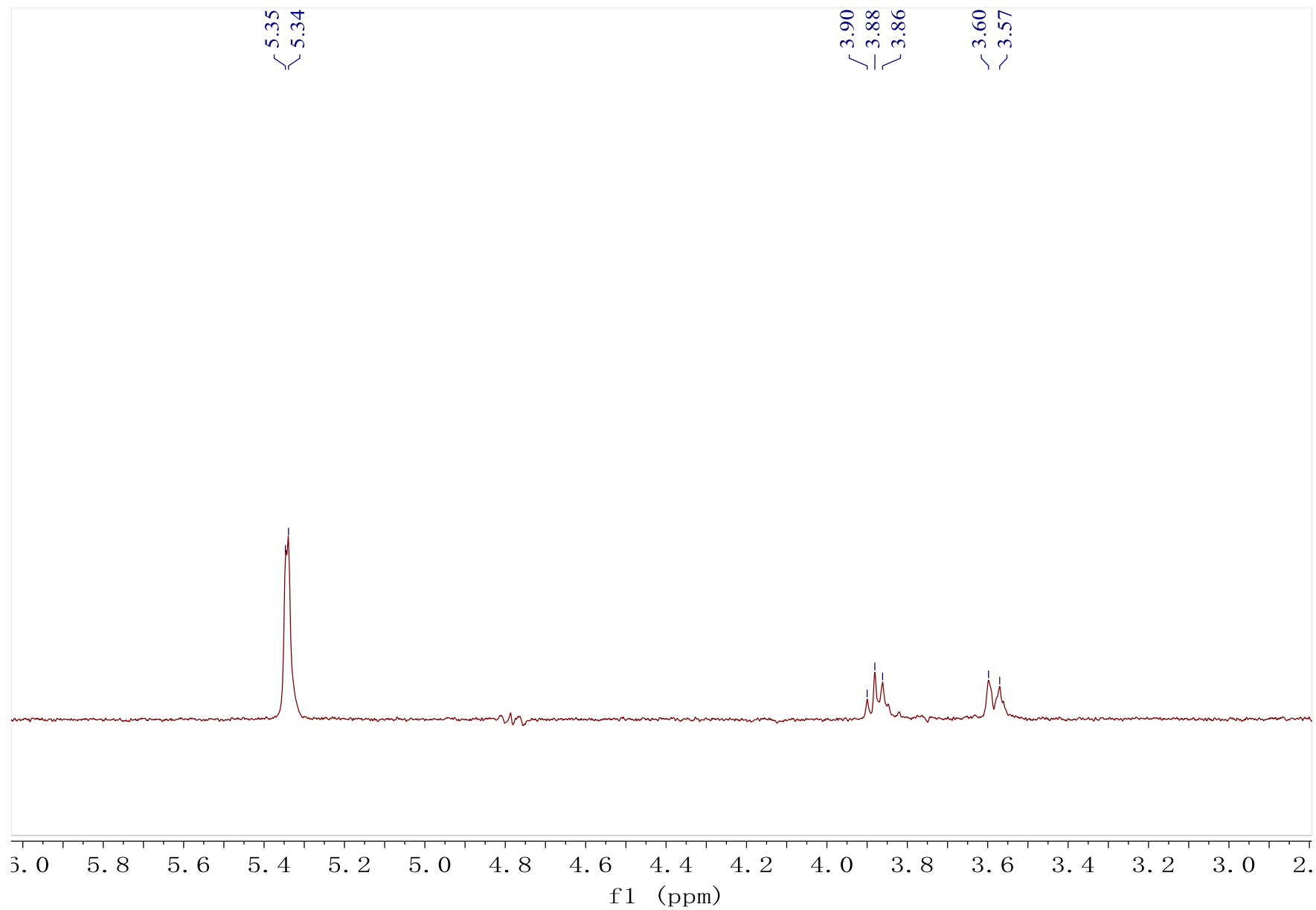
**Figure S96.** 1D-selective TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.40, H-**D**6a).



**Figure S97.** 1D-selective TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.25, H-E1).

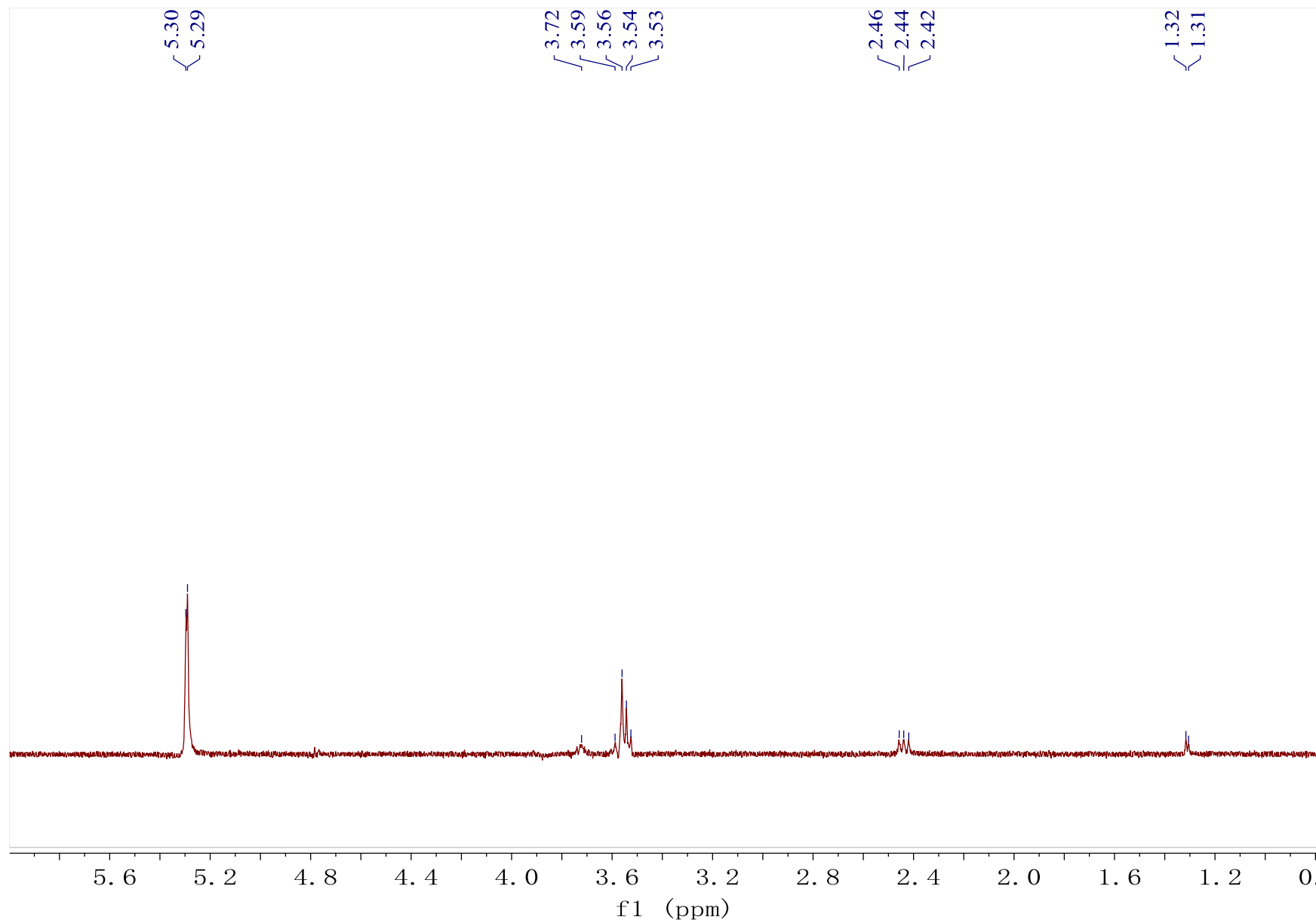


**Figure S98.** 1D-selective TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.94, H-F7).

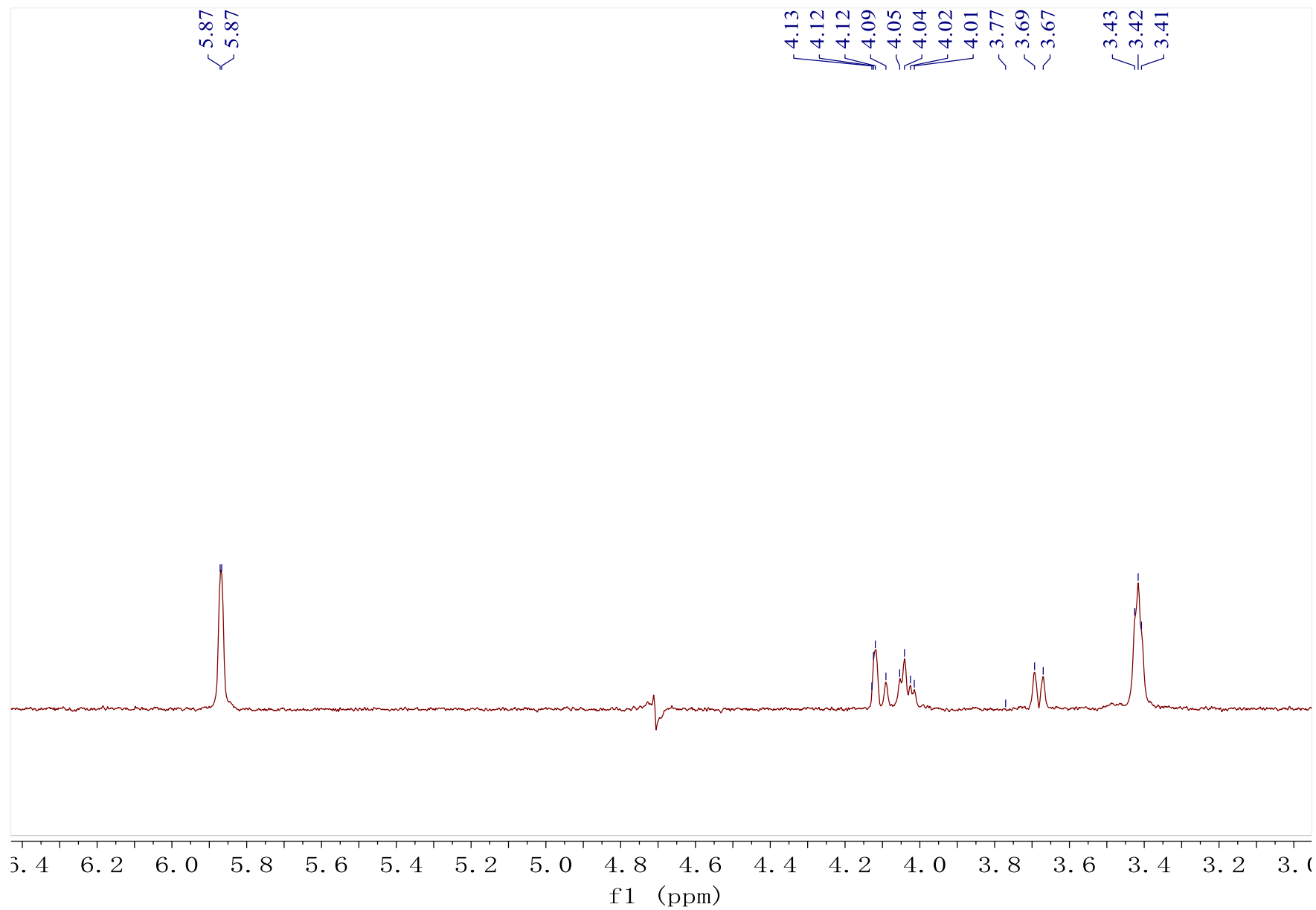


**Figure S99.** 1D-selective TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.34, H-G1).

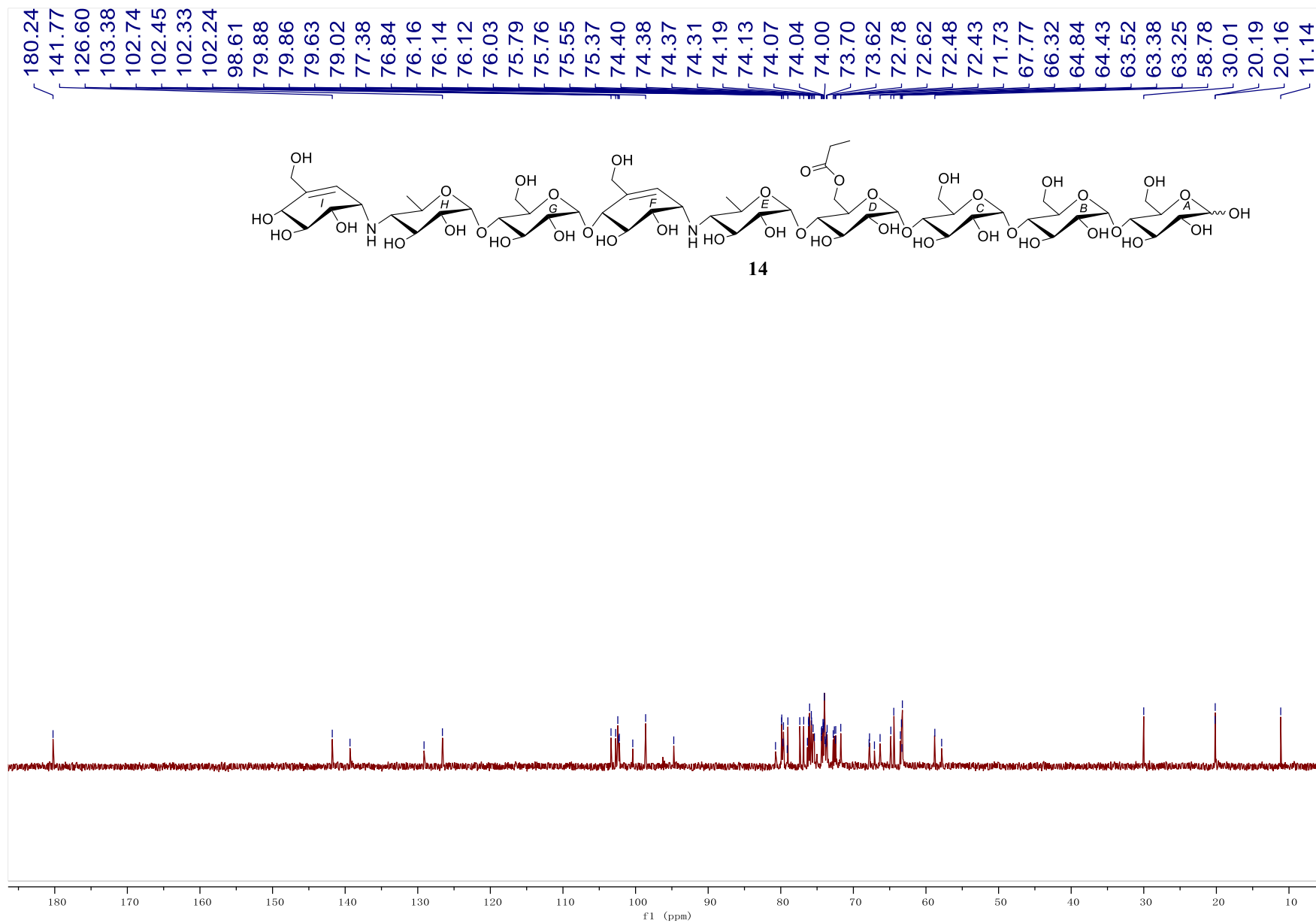




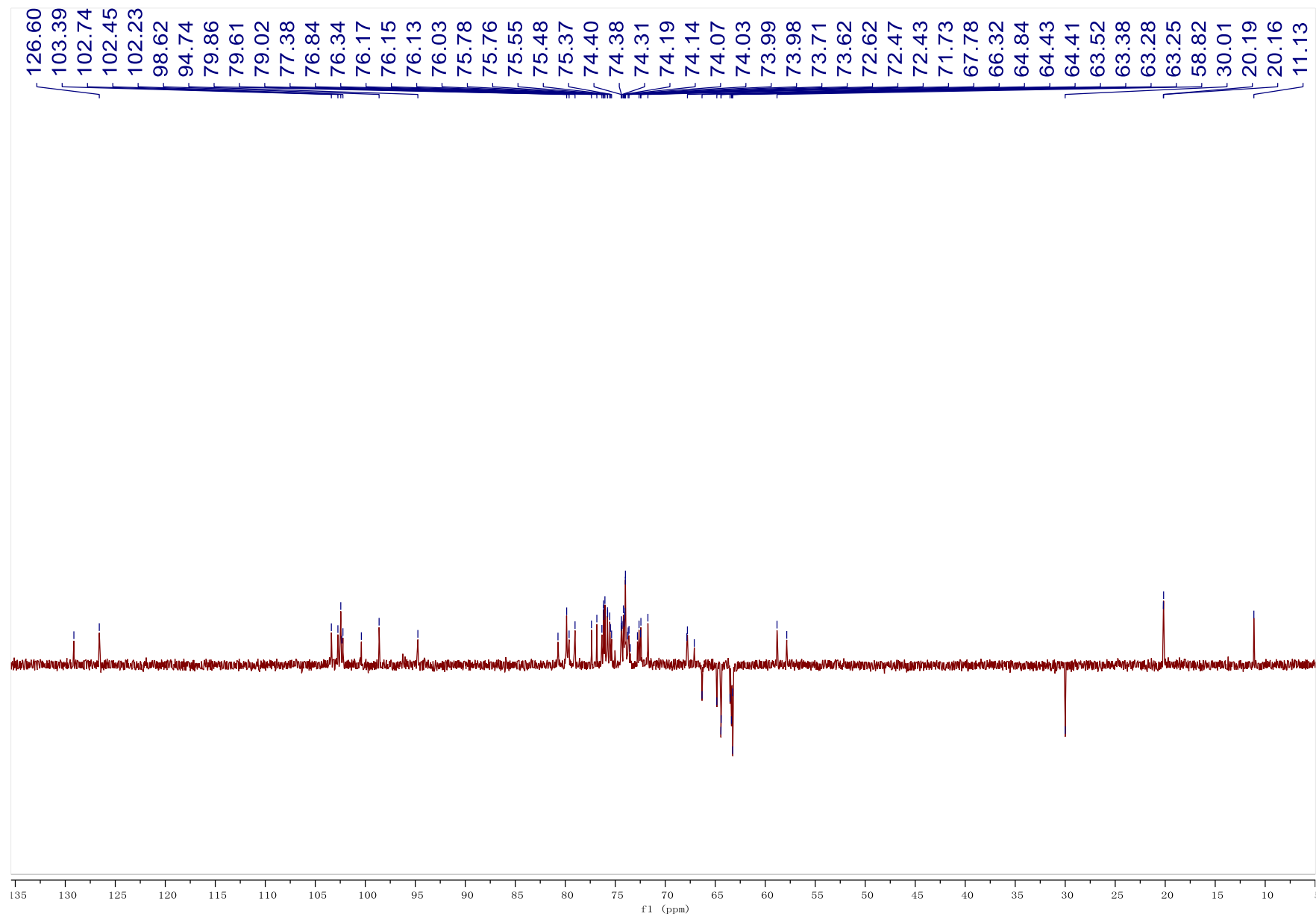
**Figure S100.** 1D-selective TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.29, H-**H1**).



**Figure S101.** 1D-selective TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.86, H-**II**).



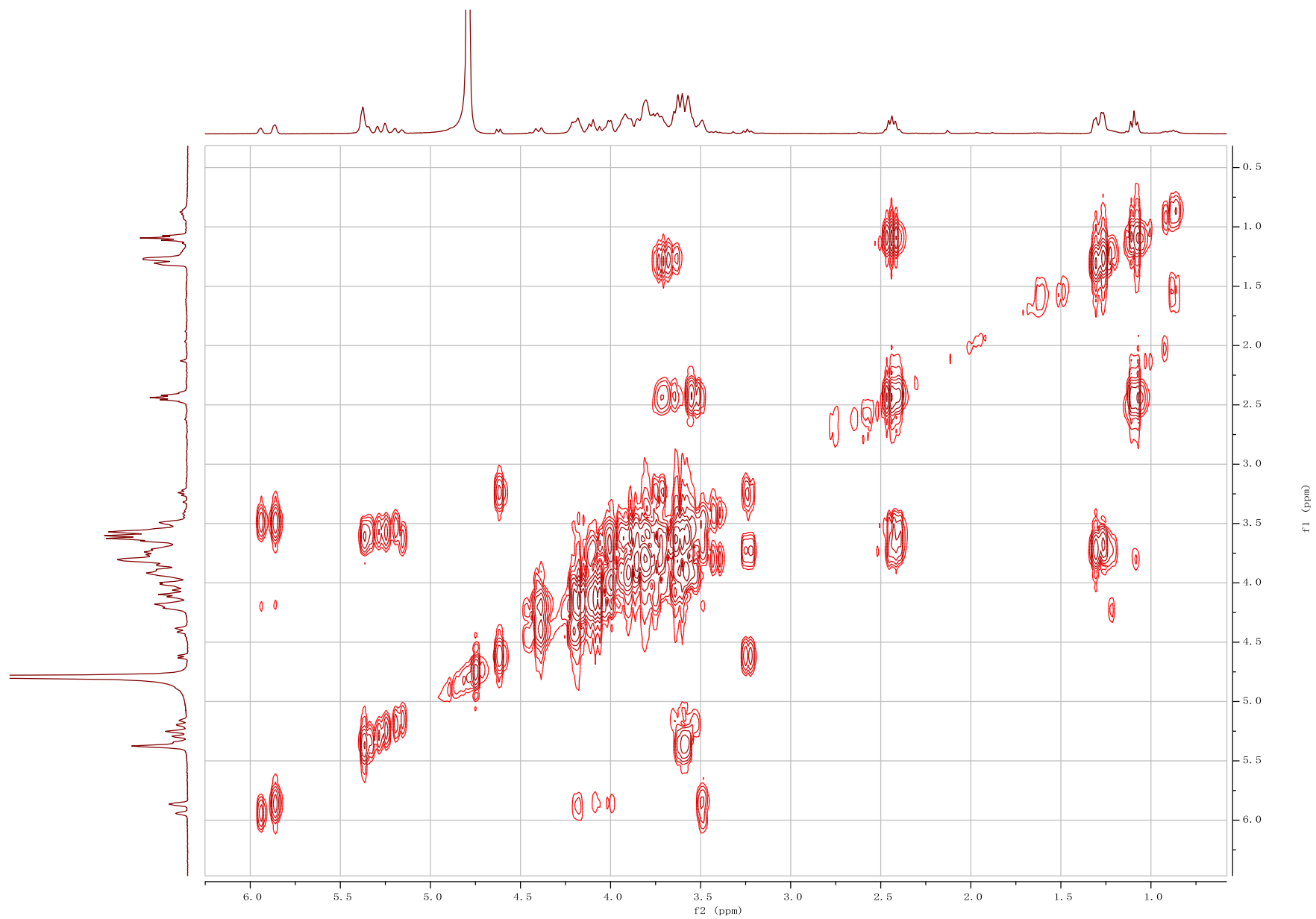
**Figure S102.**  $^{13}\text{C}$  NMR spectrum of compound **14** (125 MHz,  $\text{D}_2\text{O}$ ).



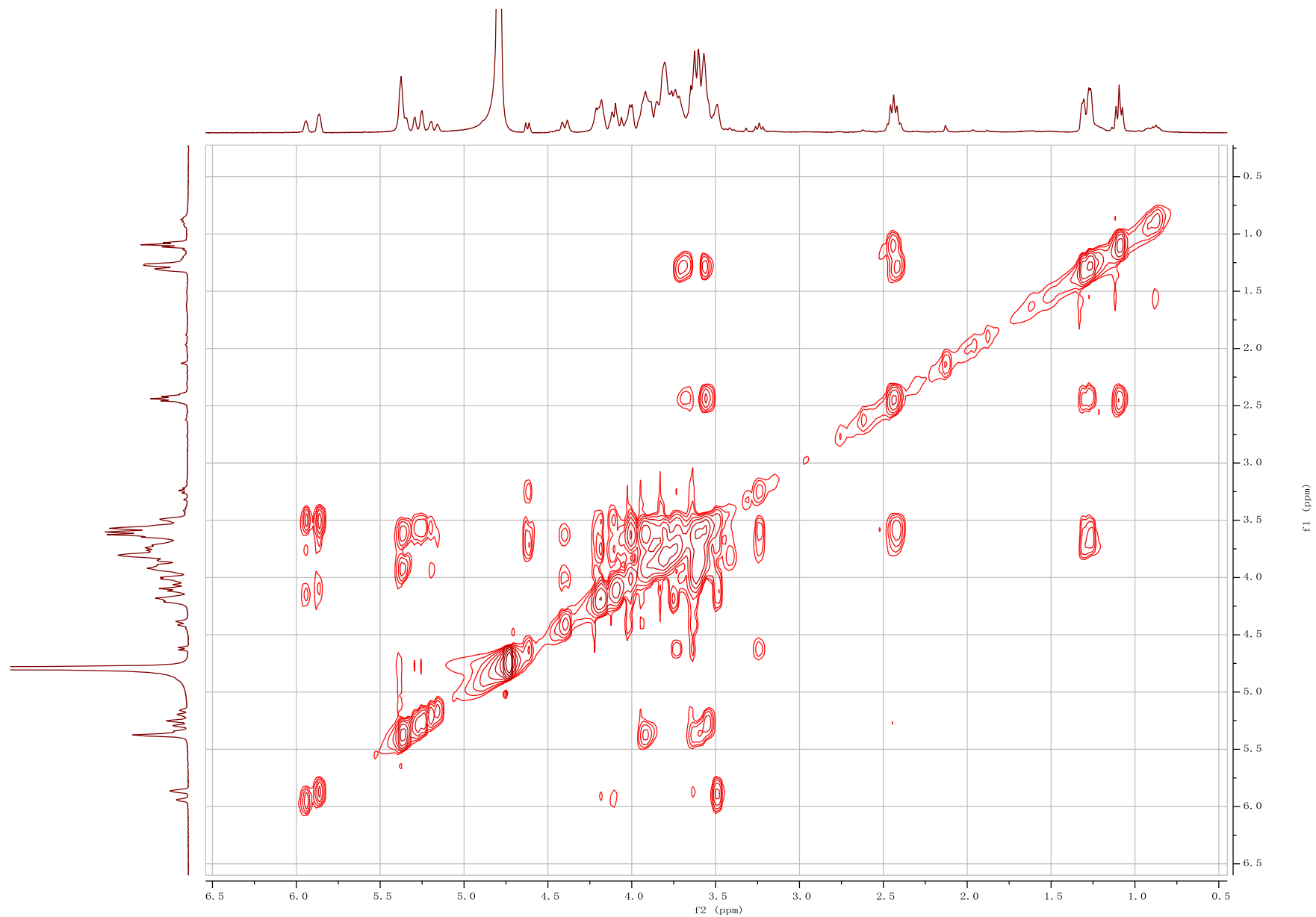
**Figure S103.** DEPT-135 spectrum of compound **14** (125 MHz, D<sub>2</sub>O).



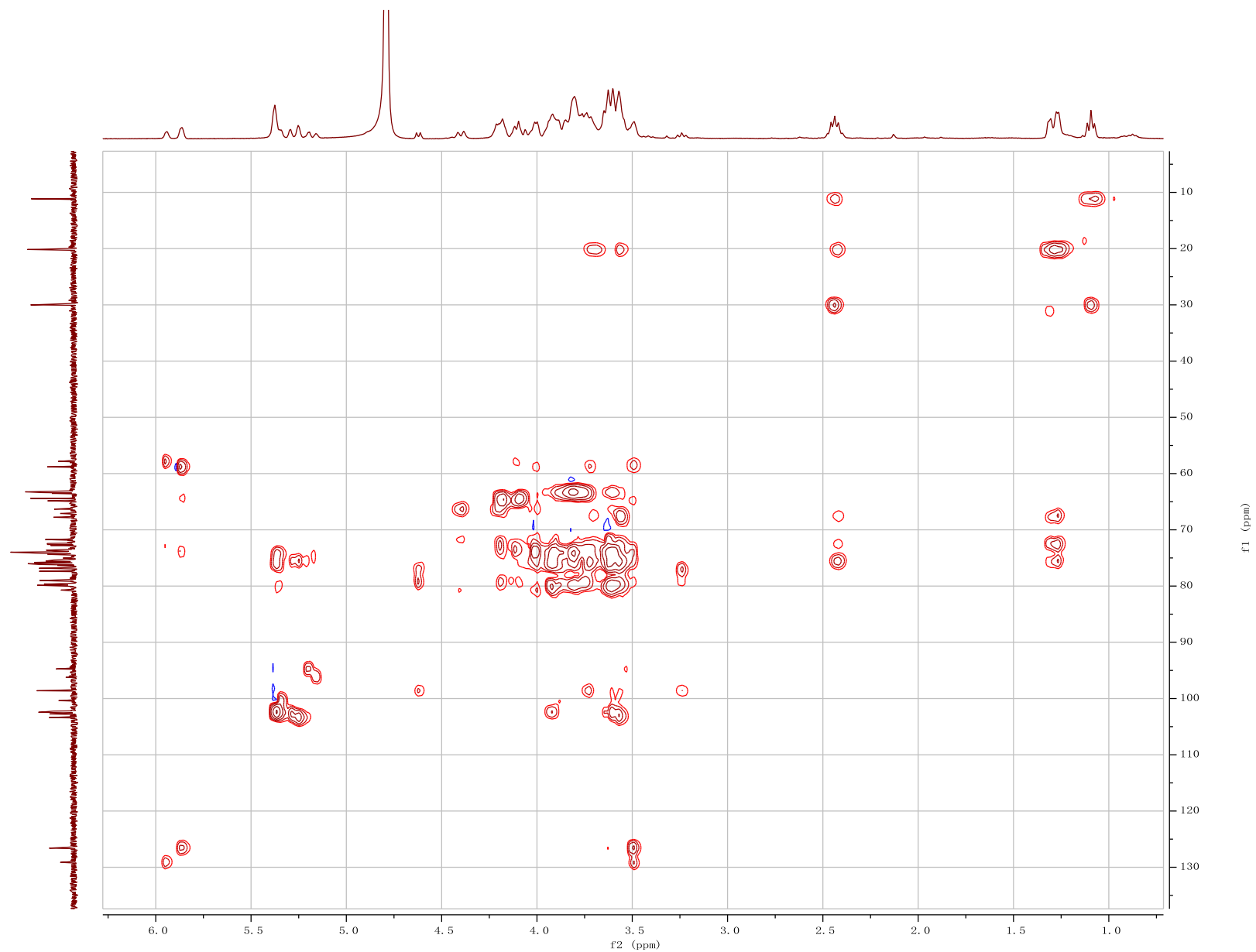
**Figure S104.** HSQC spectrum of compound **14** (500 MHz, D<sub>2</sub>O).



**Figure S105.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound **14** (500 MHz,  $\text{D}_2\text{O}$ ).

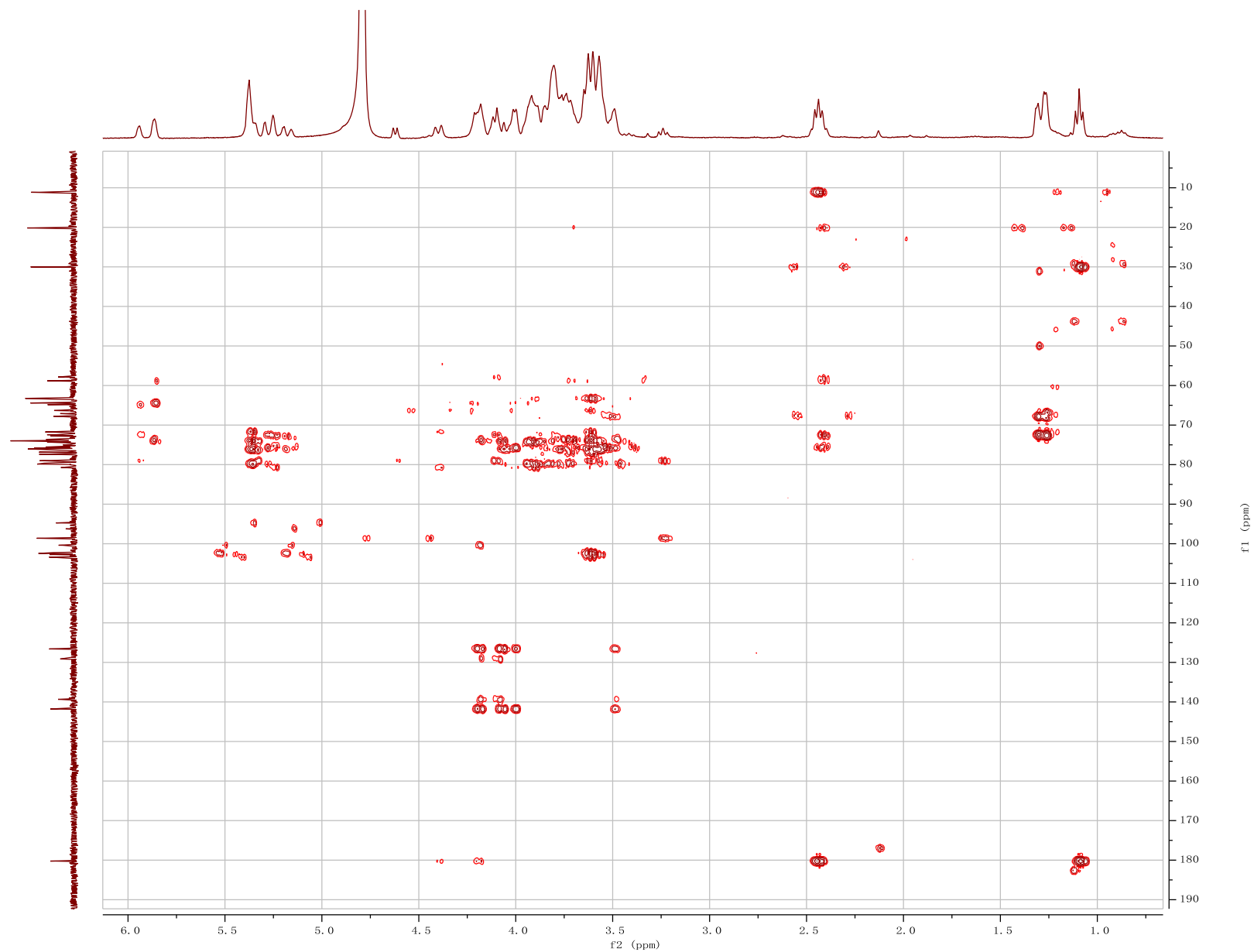


**Figure S106.** 2D-TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O).

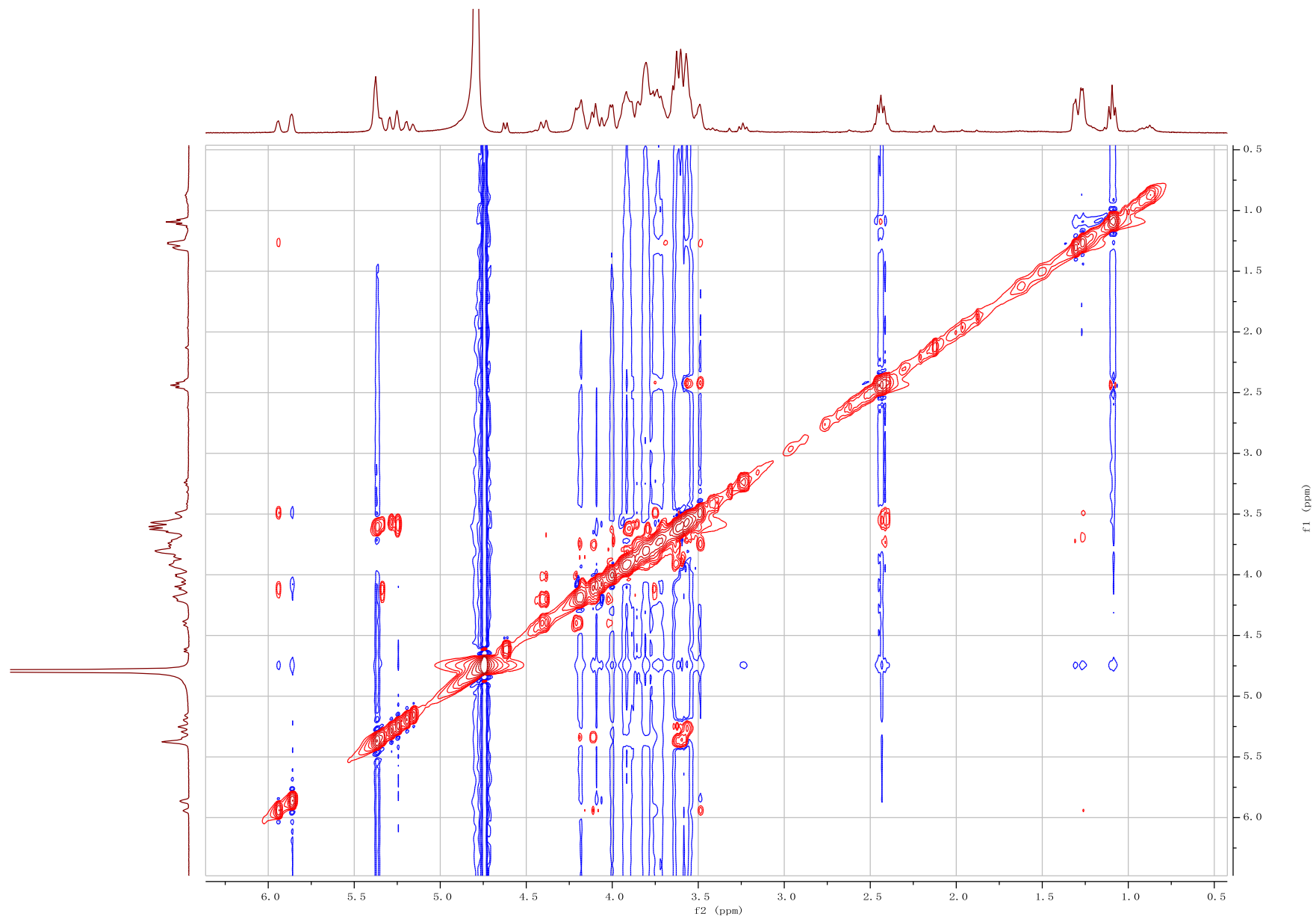


**Figure S107.** HSQC-TOCSY spectrum of compound **14** (500 MHz, D<sub>2</sub>O).

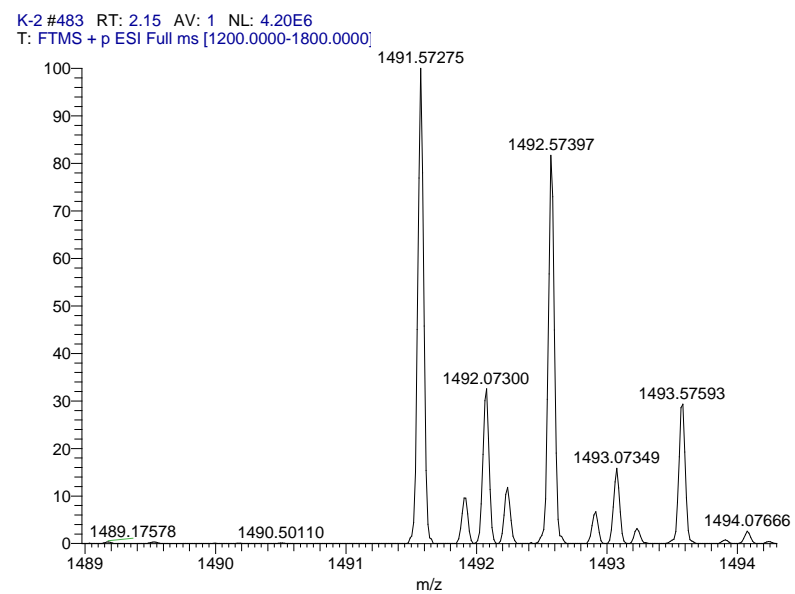




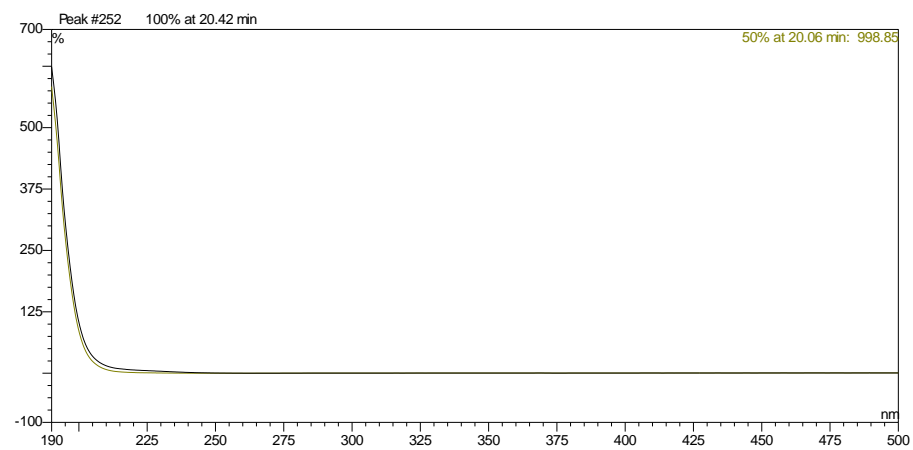
**Figure S108.** HMBC spectrum of compound **14** (500 MHz, D<sub>2</sub>O).



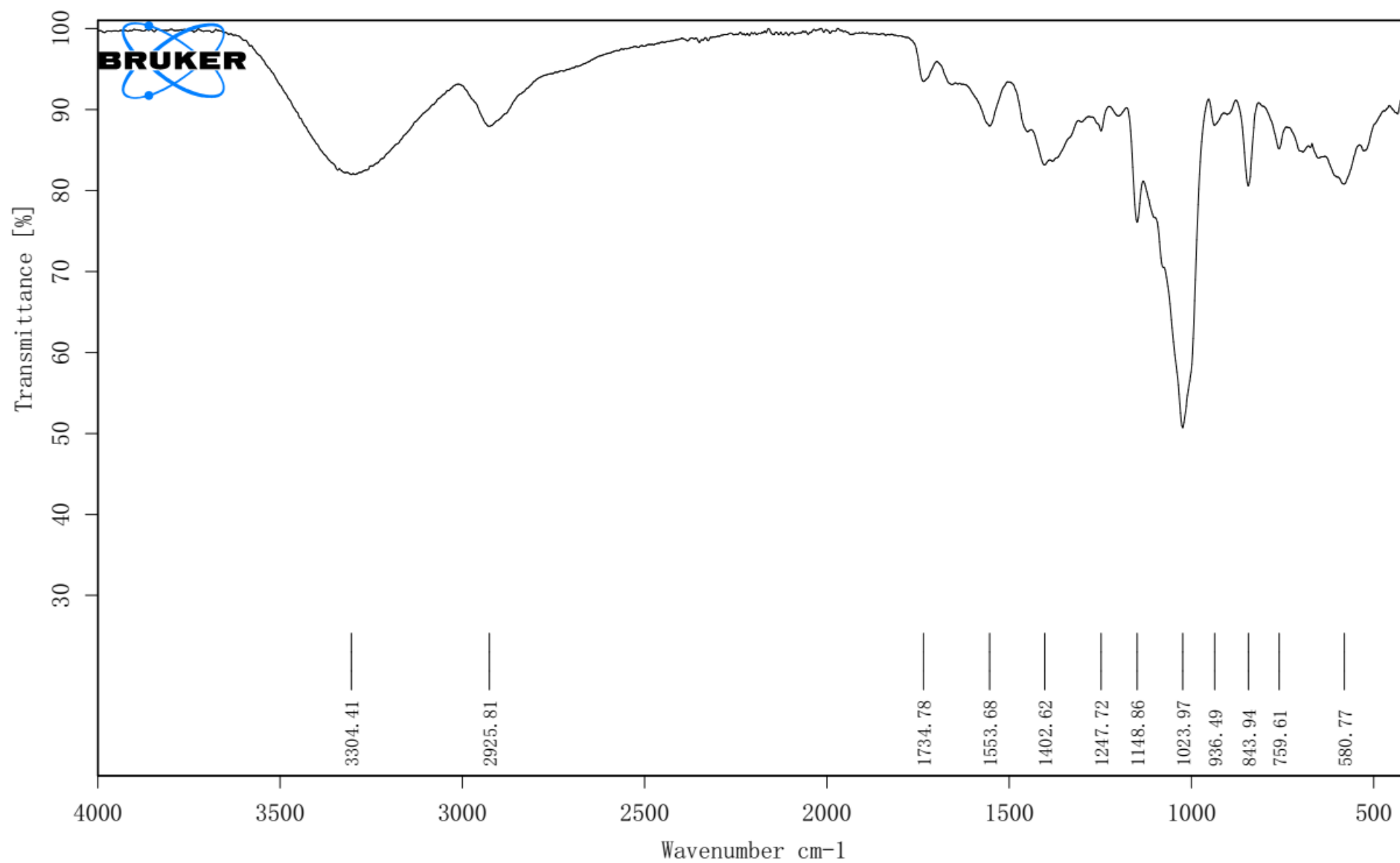
**Figure S109.** NOESY spectrum of compound **14** (500 MHz, D<sub>2</sub>O).



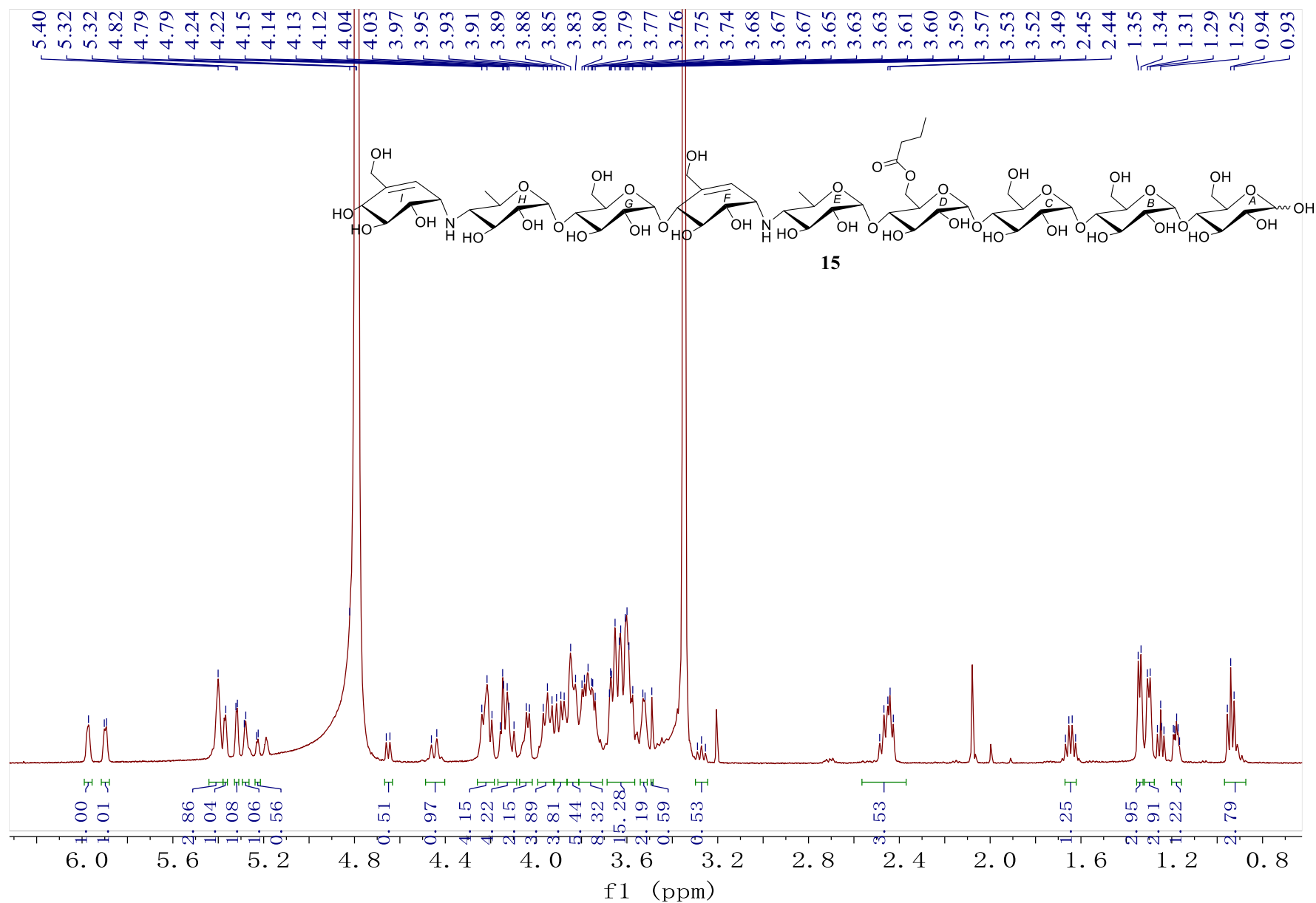
**Figure S110.** HRESIMS spectrum of compound **14**.



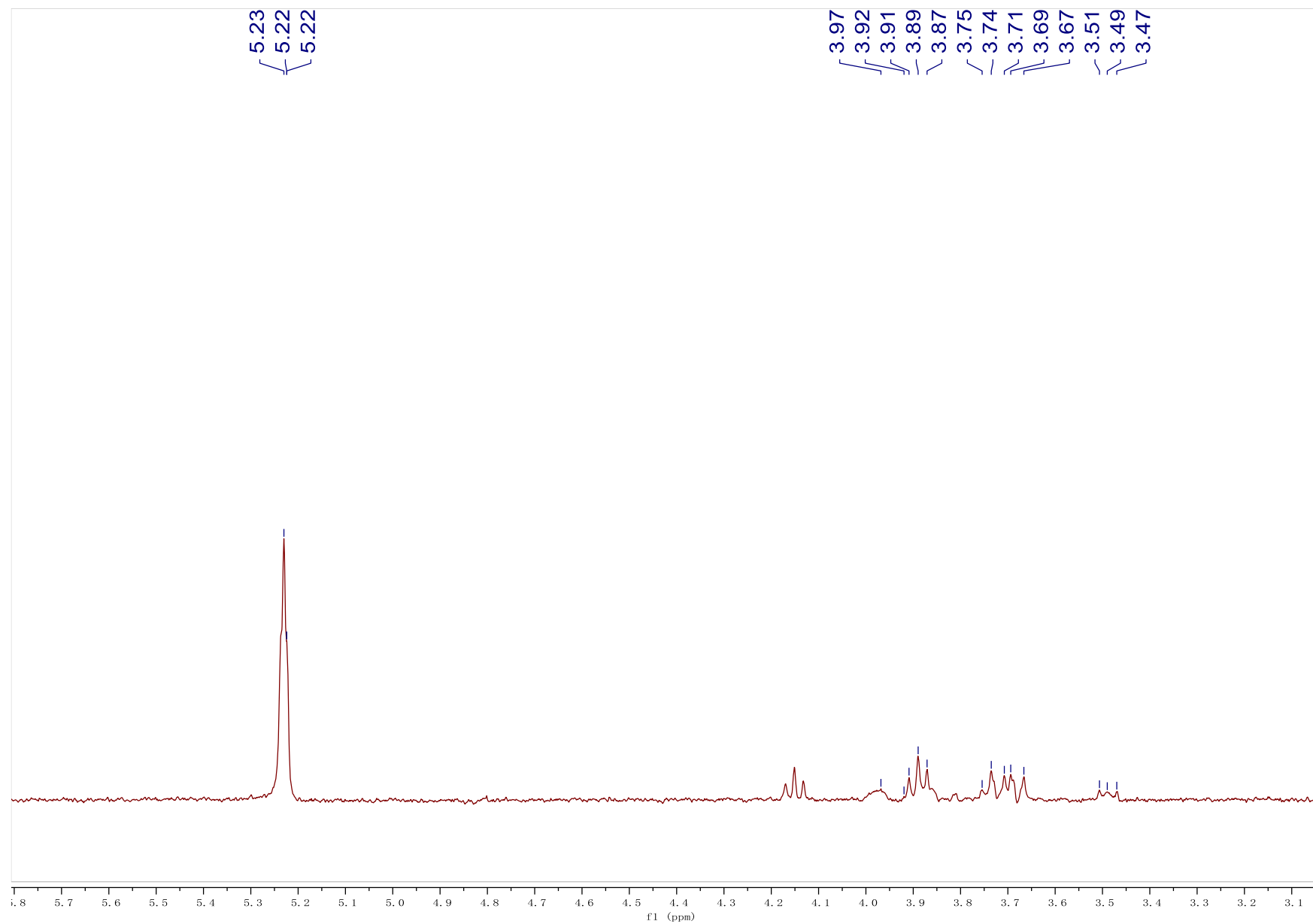
**Figure S111.** UV spectrum of compound **14**.



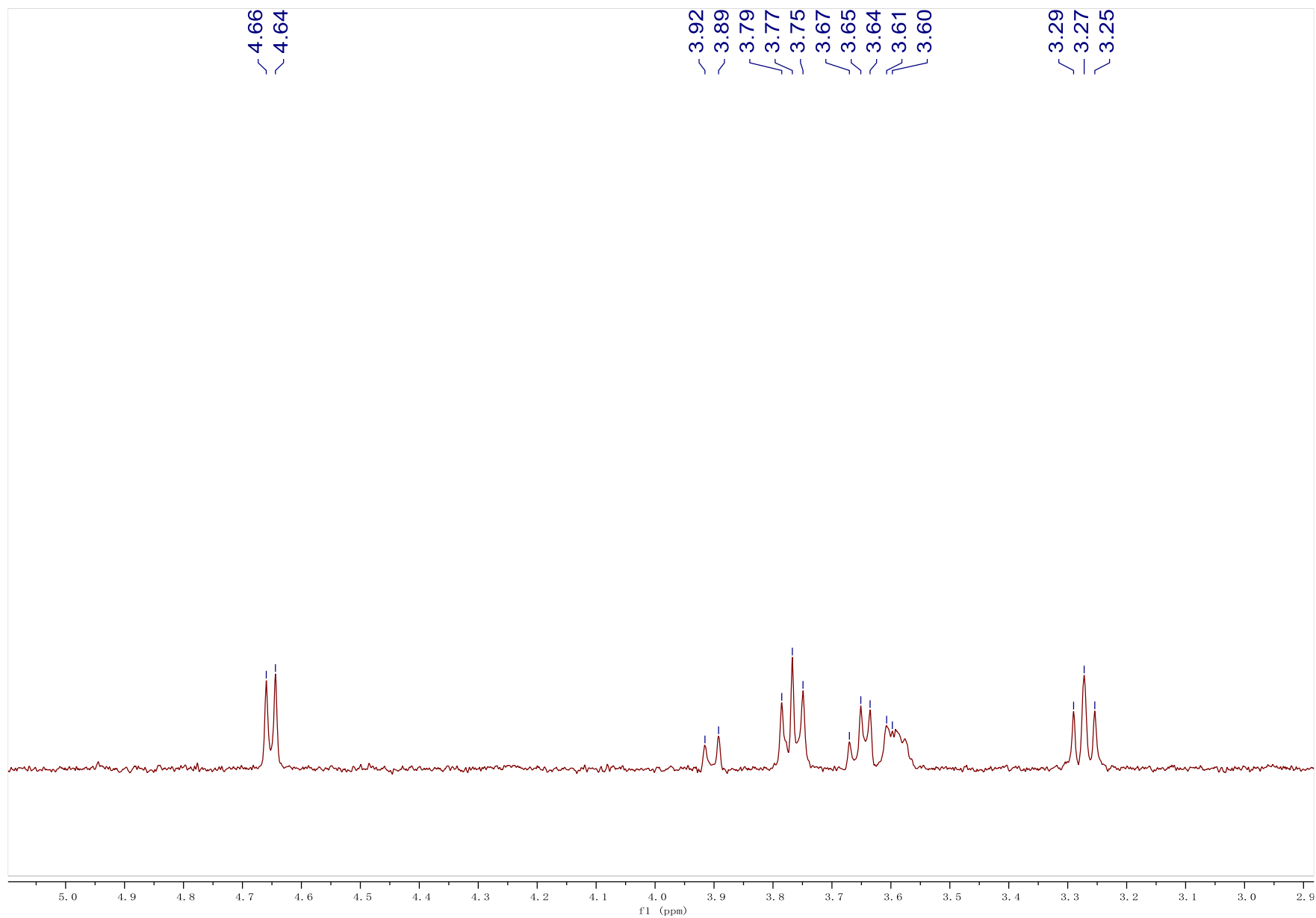
**Figure S112.** IR spectrum of compound **14**.



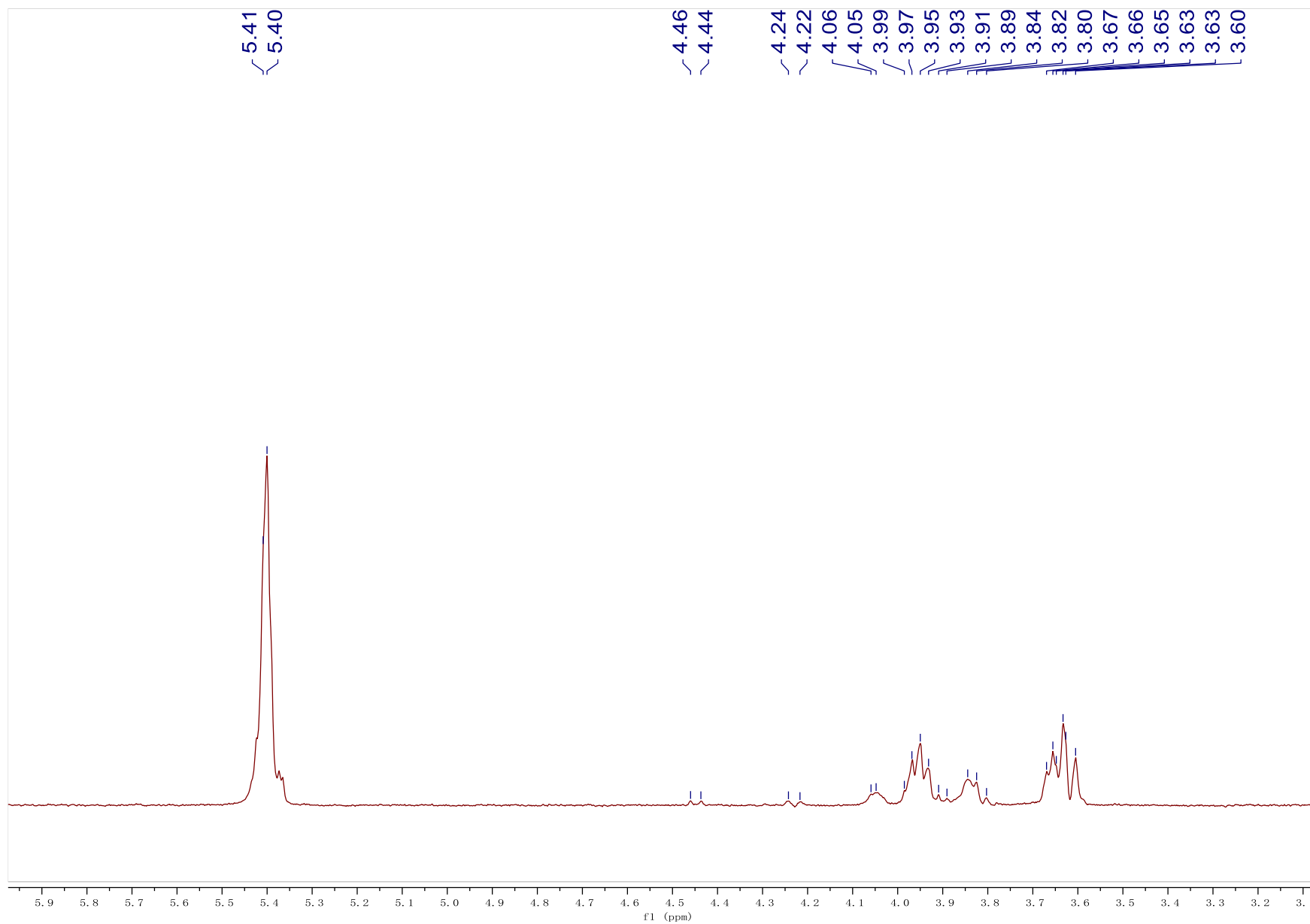
**Figure S113.**  $^1\text{H}$  NMR spectrum of compound **15** (500 MHz,  $\text{D}_2\text{O}$ ). 137



**Figure S114.** 1D-selective TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.23, H-A1 $\alpha$ ).

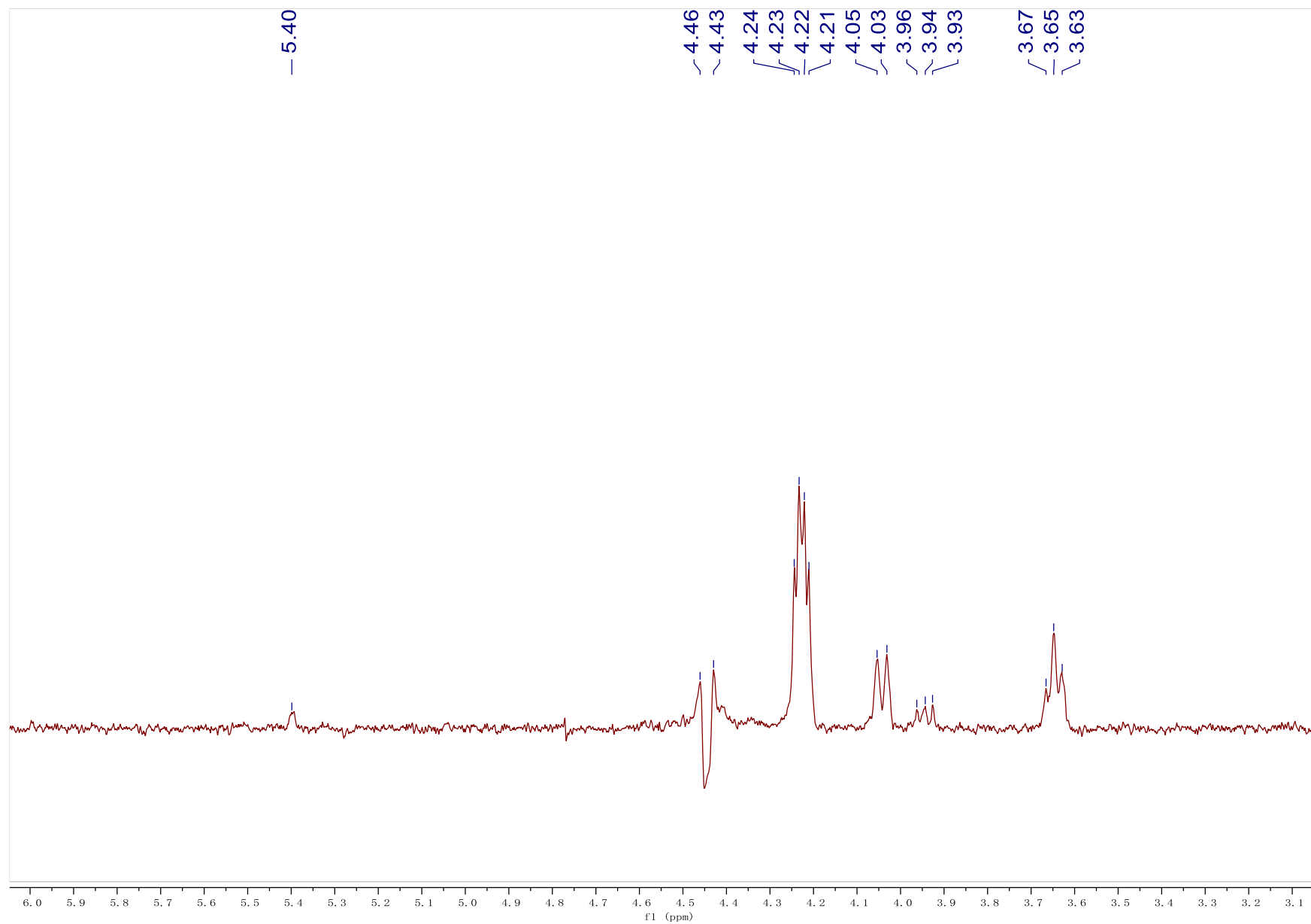


**Figure S115.** 1D-selective TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.65, H-A1 $\beta$ ).

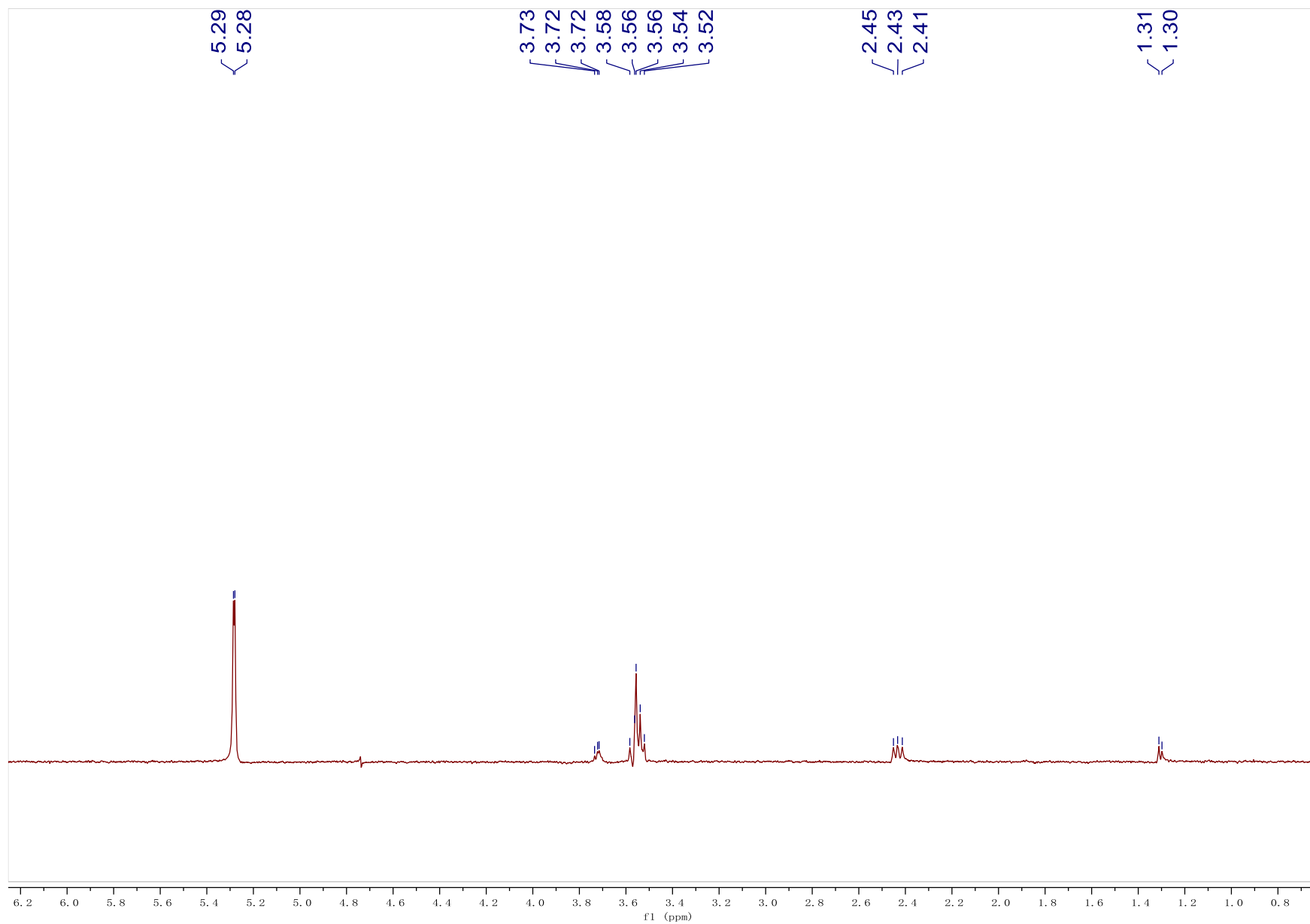


**Figure S116.** 1D-selective TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.40, H-B1, C1, and D1).

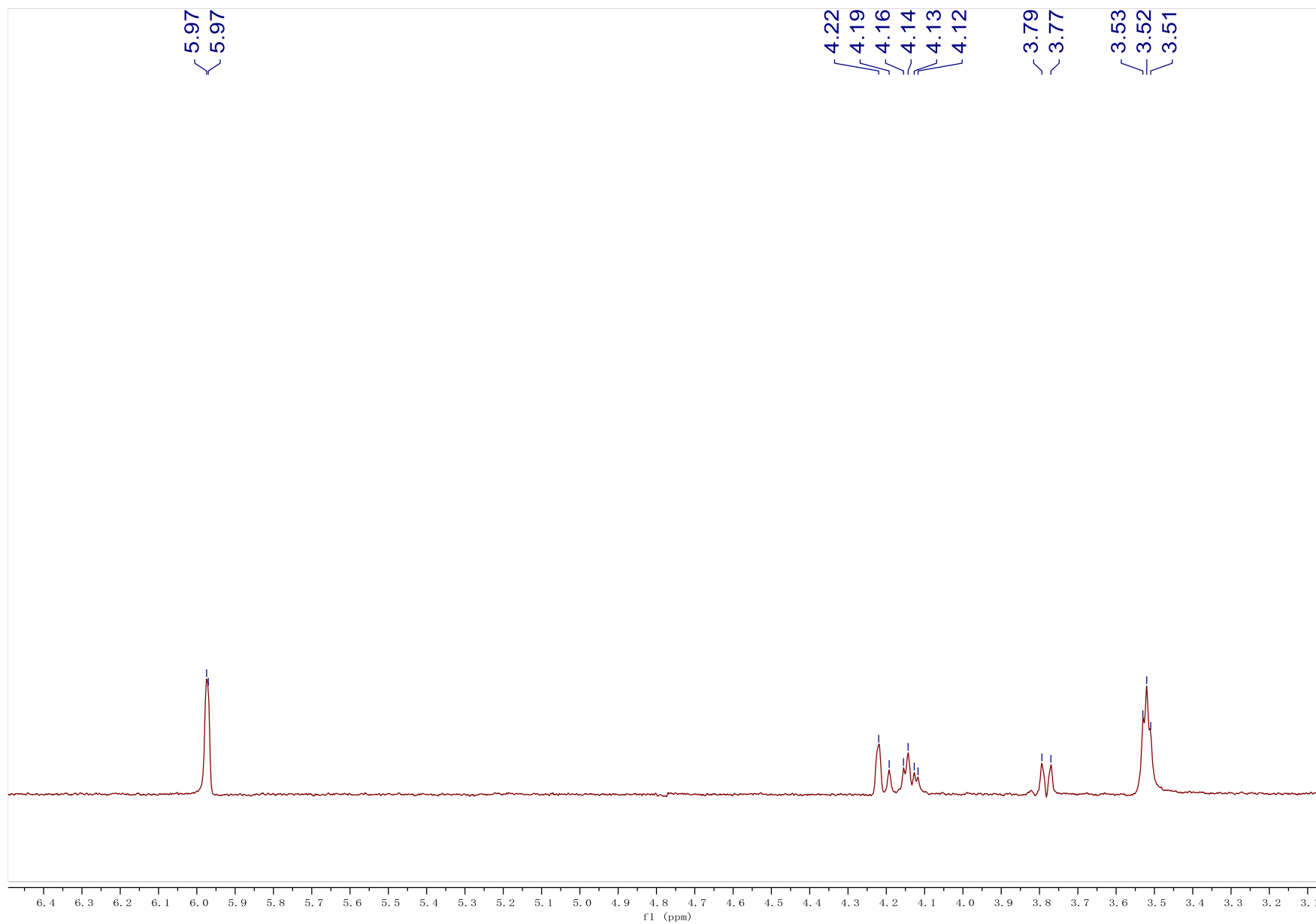




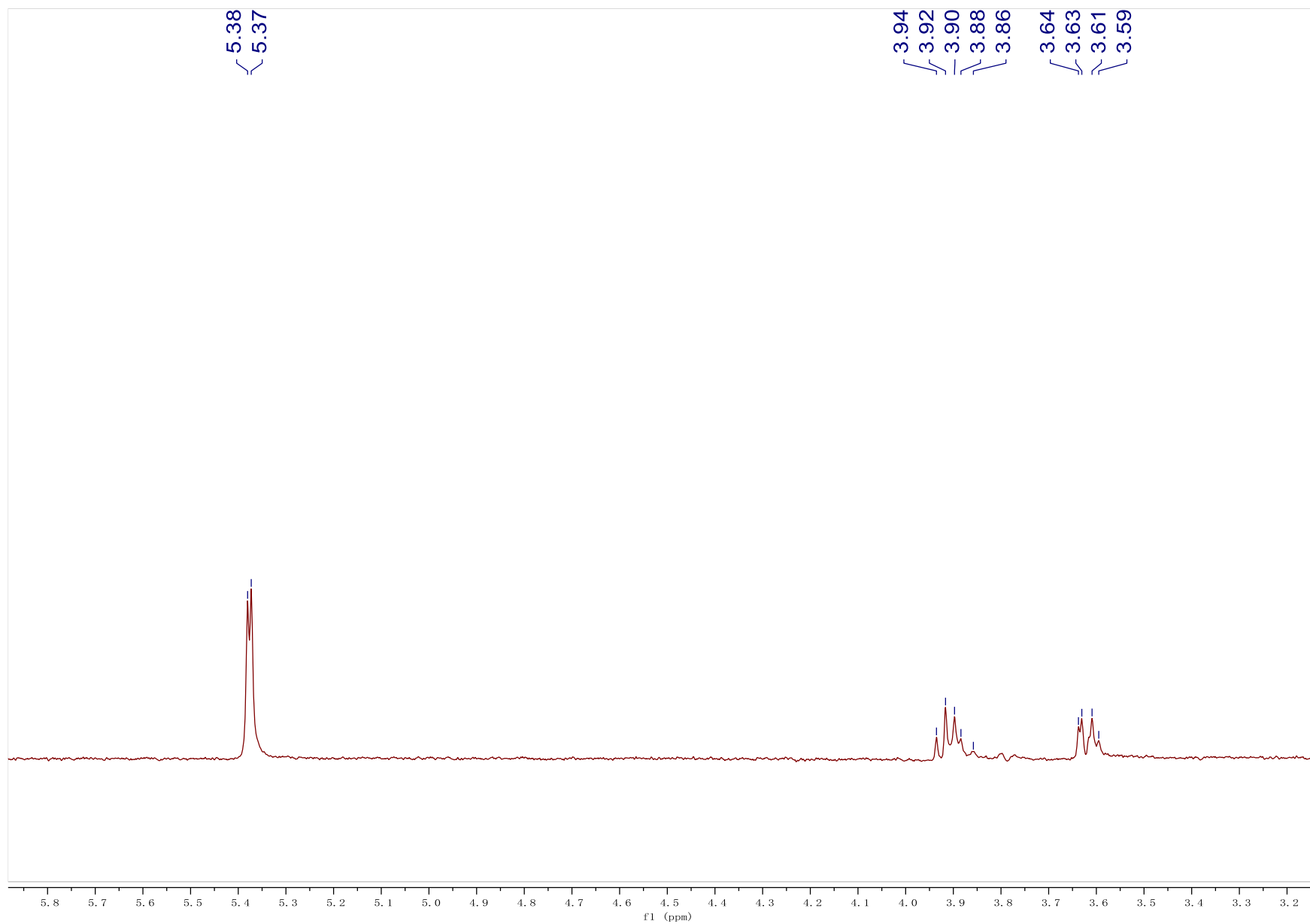
**Figure S117.** 1D-selective TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.45, H-D6a).



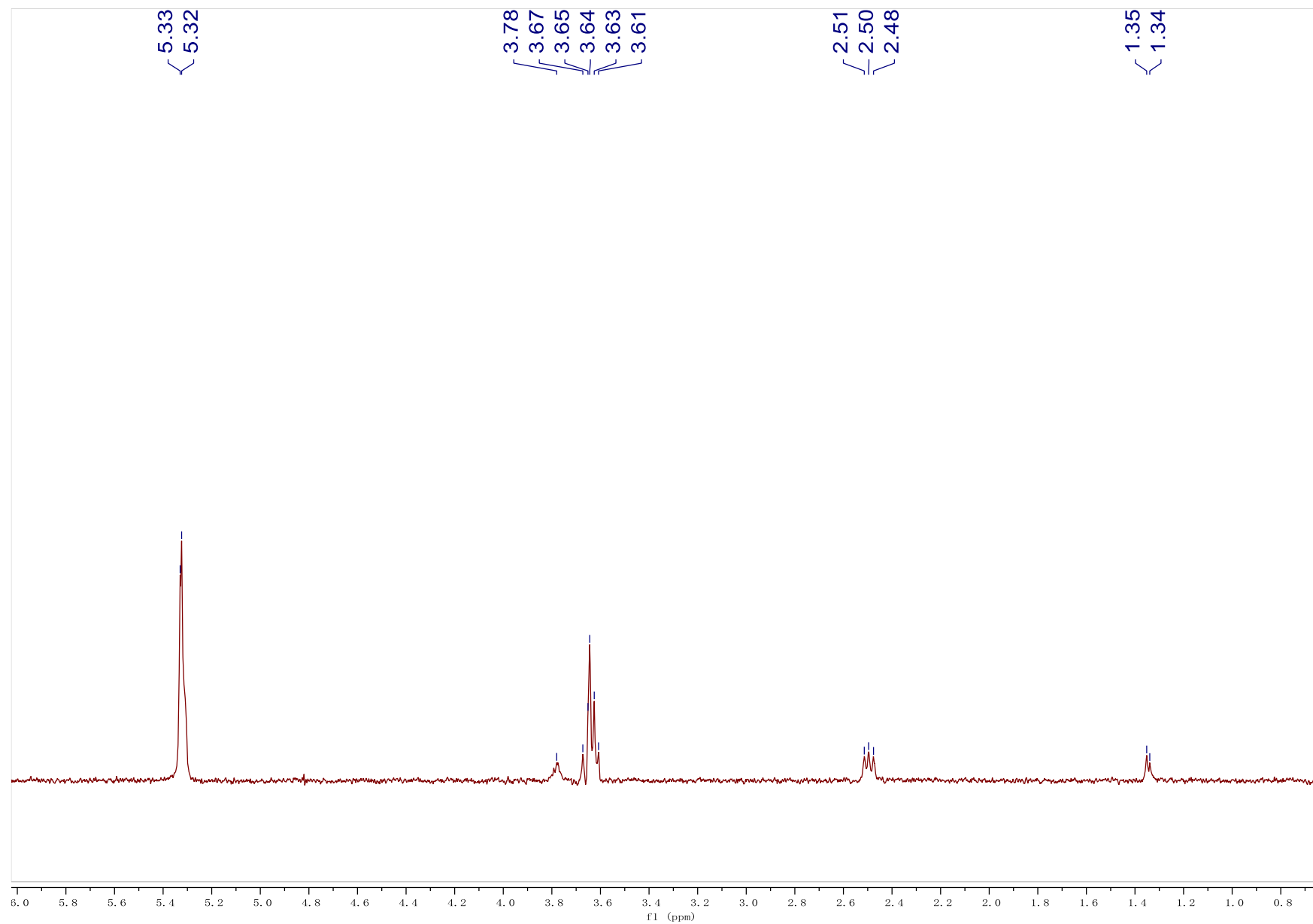
**Figure S118.** 1D-selective TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.28, H-E1).



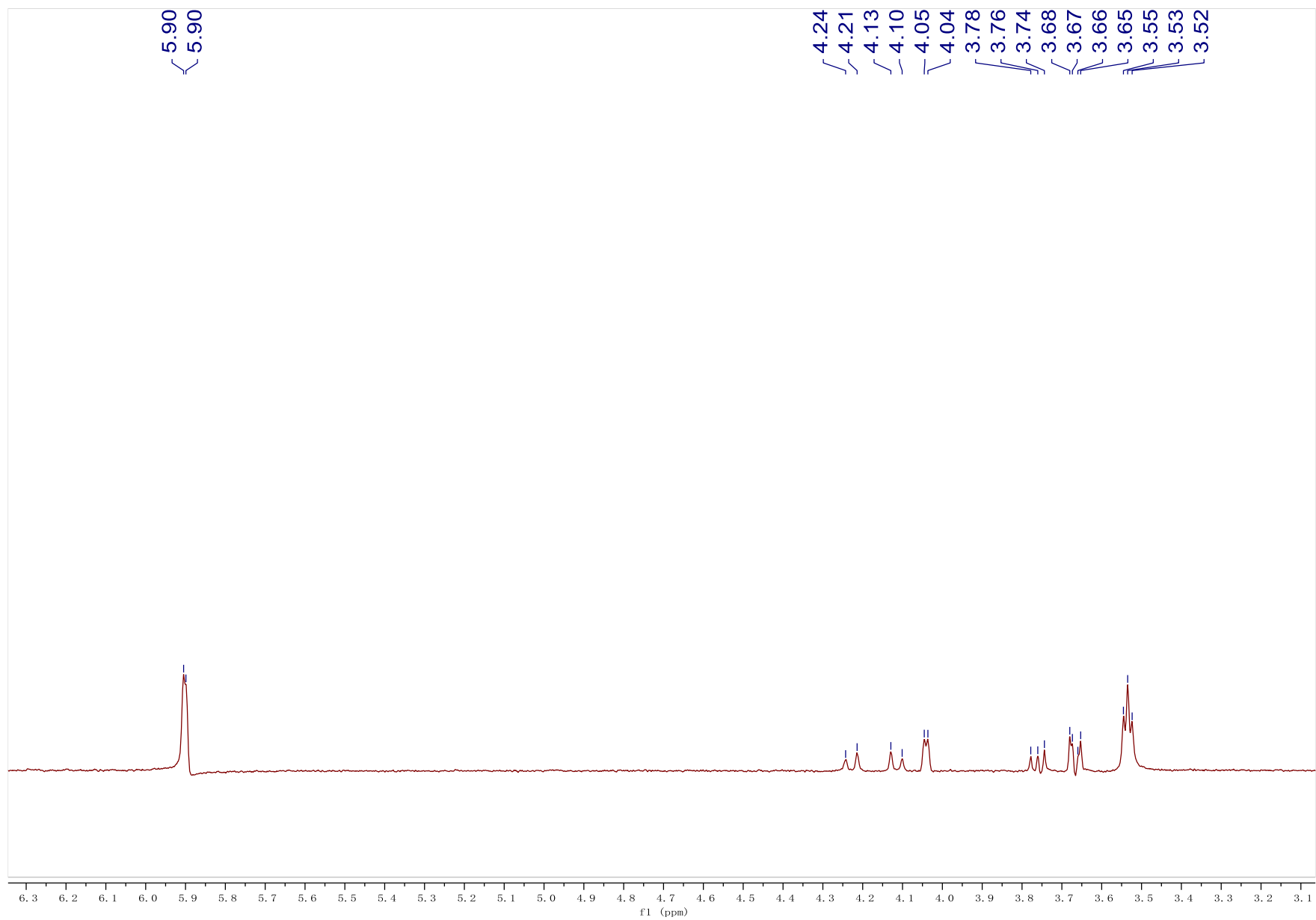
**Figure S119.** 1D-selective TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.97, H-F1).



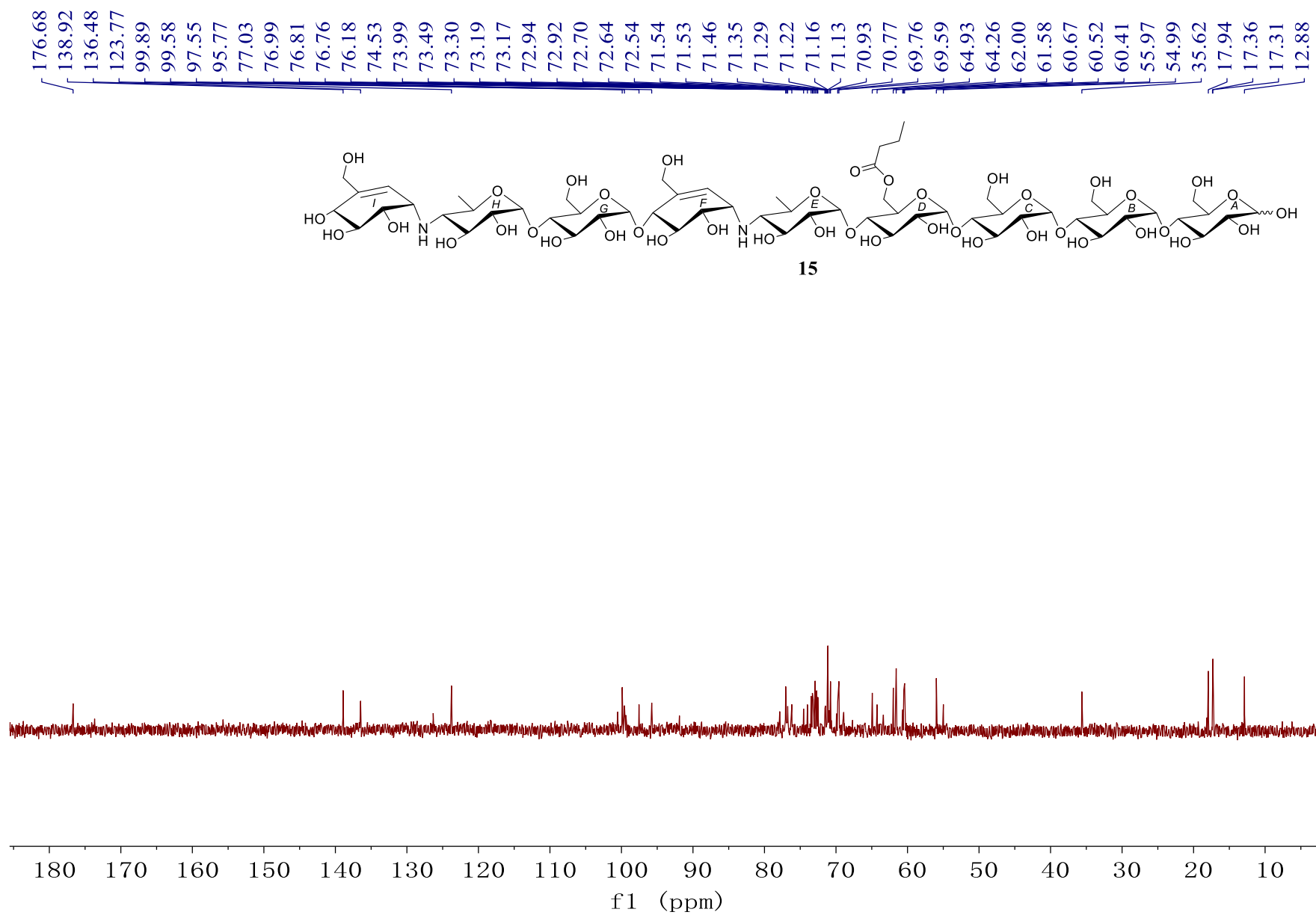
**Figure S120.** 1D-selective TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.37, H-G1).



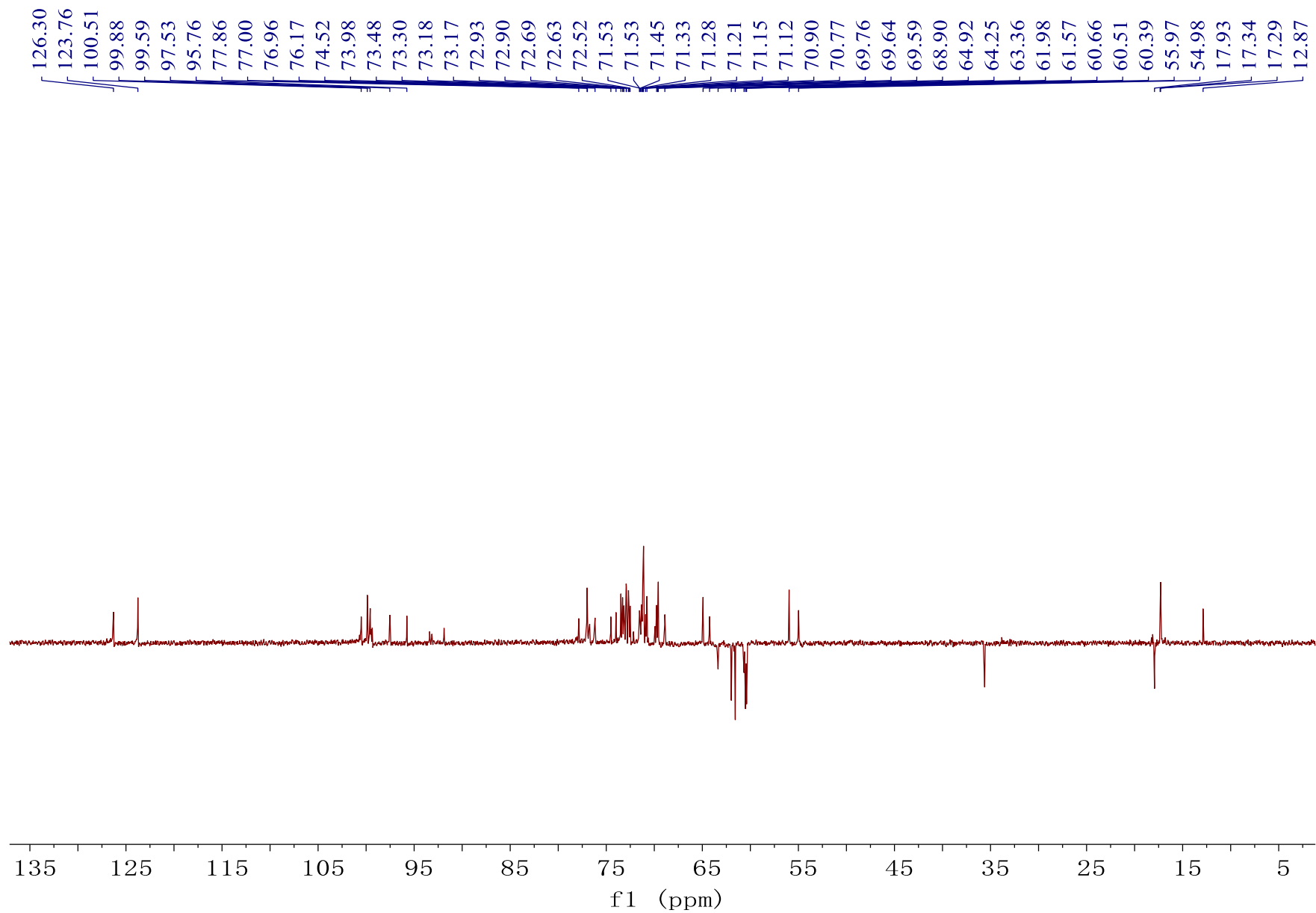
**Figure S121.** 1D-selective TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.32, H-**H1**).



**Figure S122.** 1D-selective TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.90, H-I1).



**Figure S123.**  $^{13}\text{C}$  NMR spectrum of compound **15** (125 MHz,  $\text{D}_2\text{O}$ ).

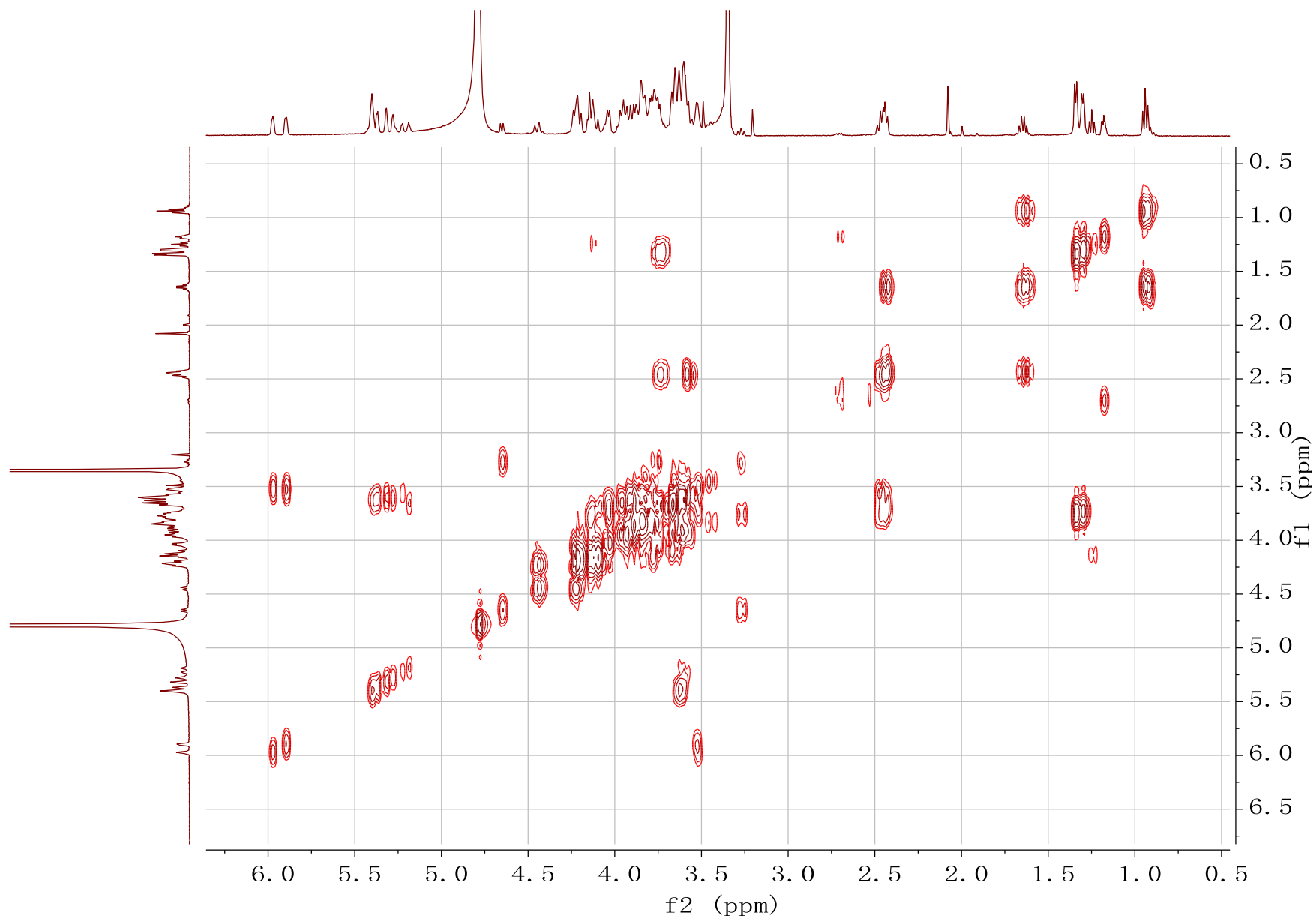


**Figure S124.** DEPT-135 spectrum of compound **15** (125 MHz, D<sub>2</sub>O).

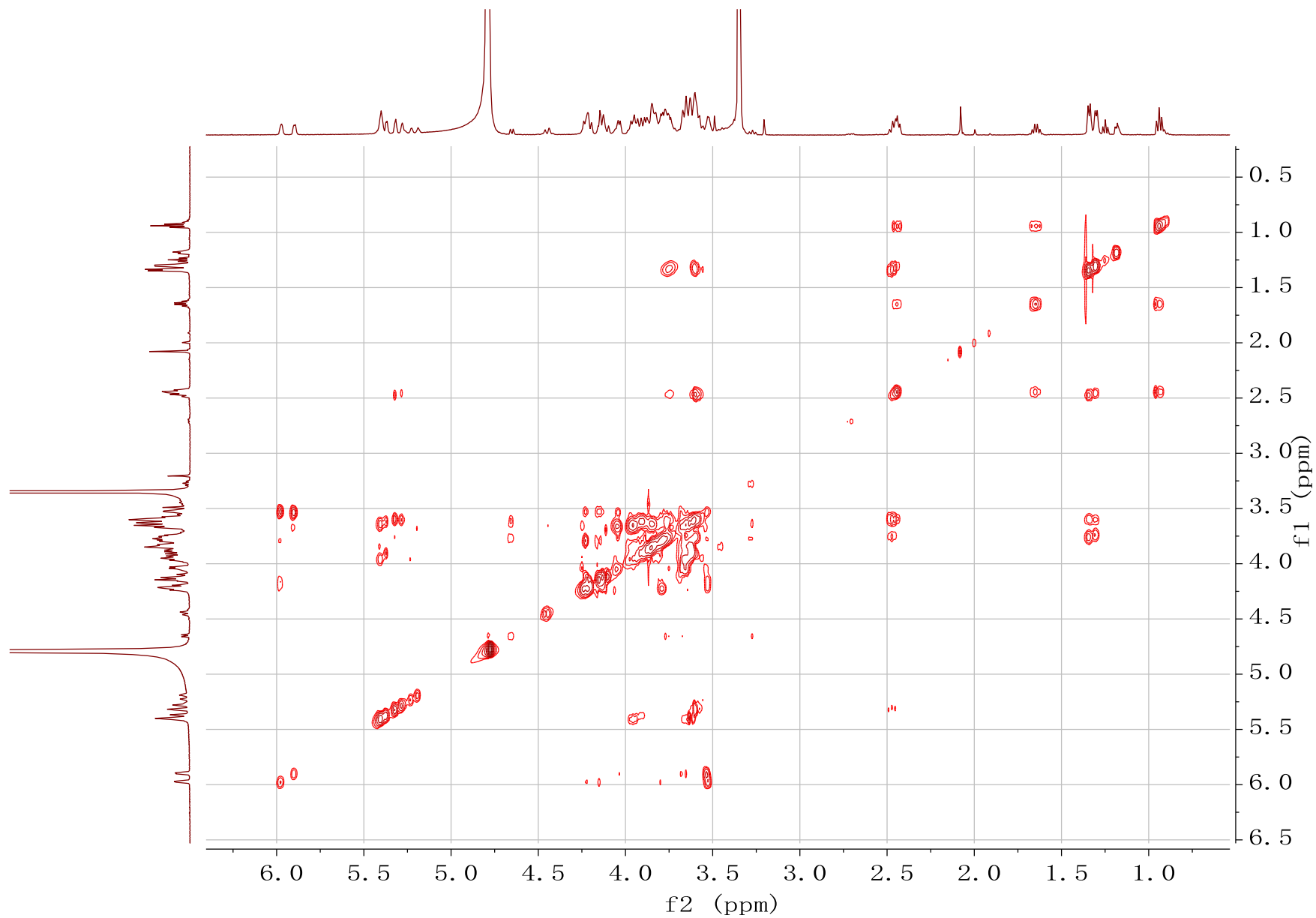




**Figure S125.** HSQC spectrum of compound **15** (500 MHz, D<sub>2</sub>O).



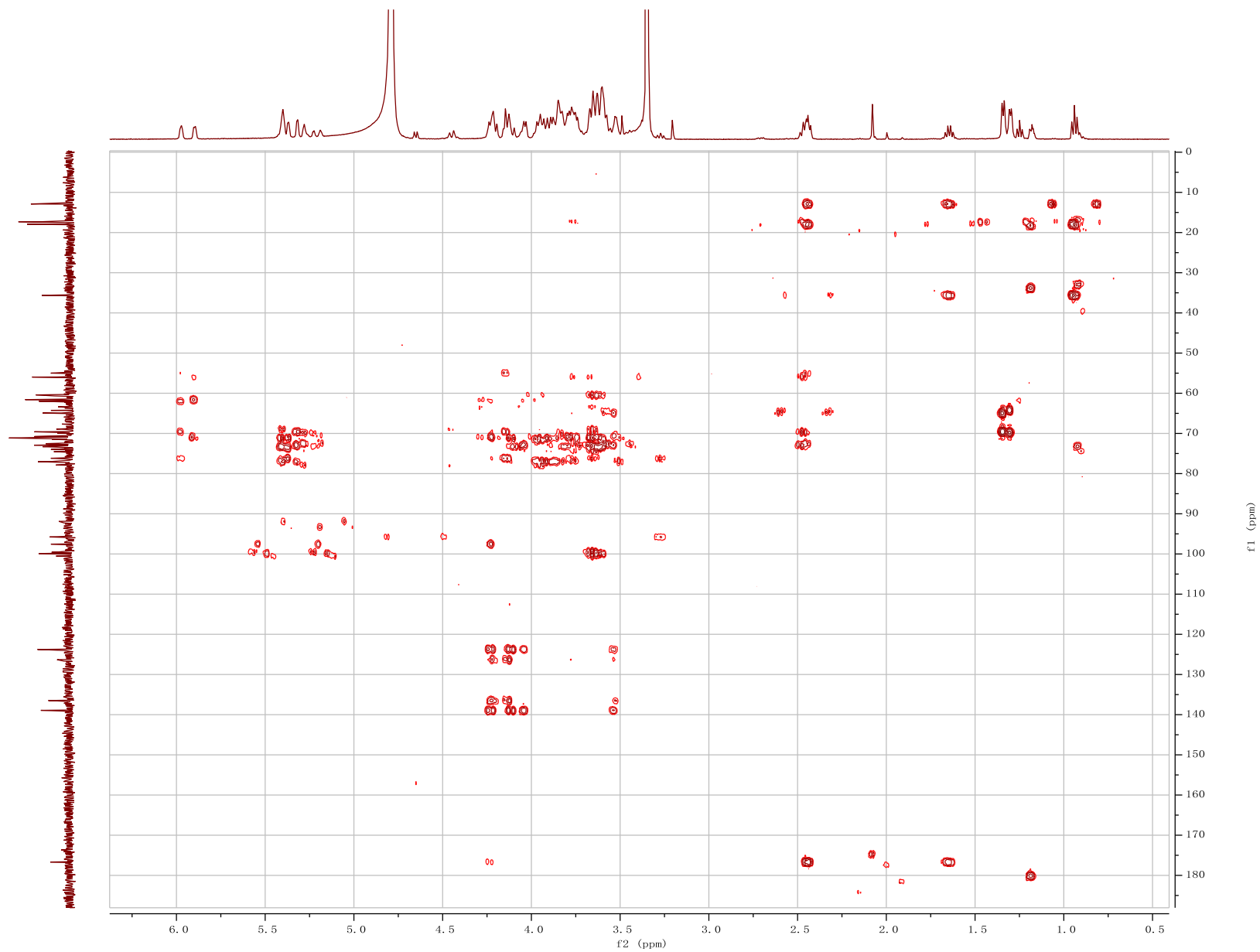
**Figure S126.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound **15** (500 MHz,  $\text{D}_2\text{O}$ ).



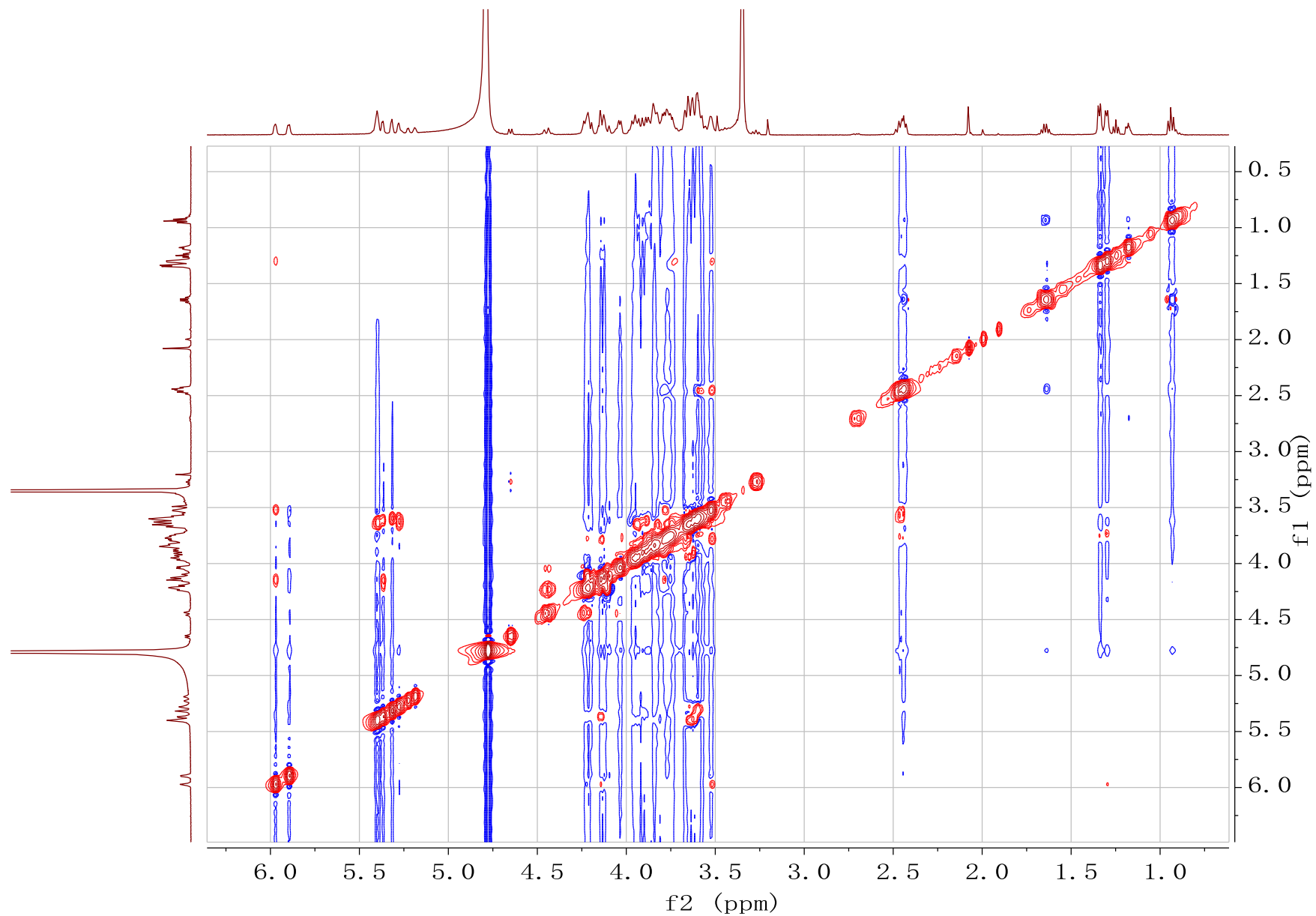
**Figure S127.** 2D-TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O).



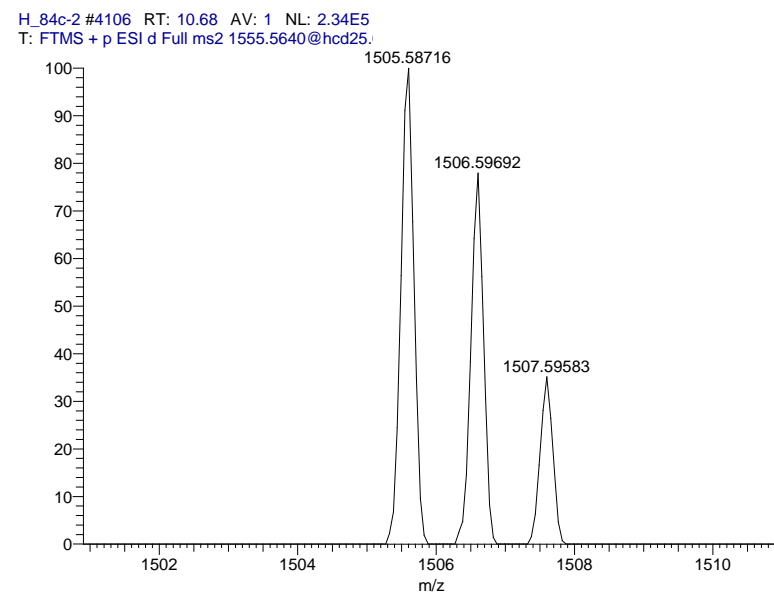
**Figure S128.** HSQC-TOCSY spectrum of compound **15** (500 MHz, D<sub>2</sub>O).



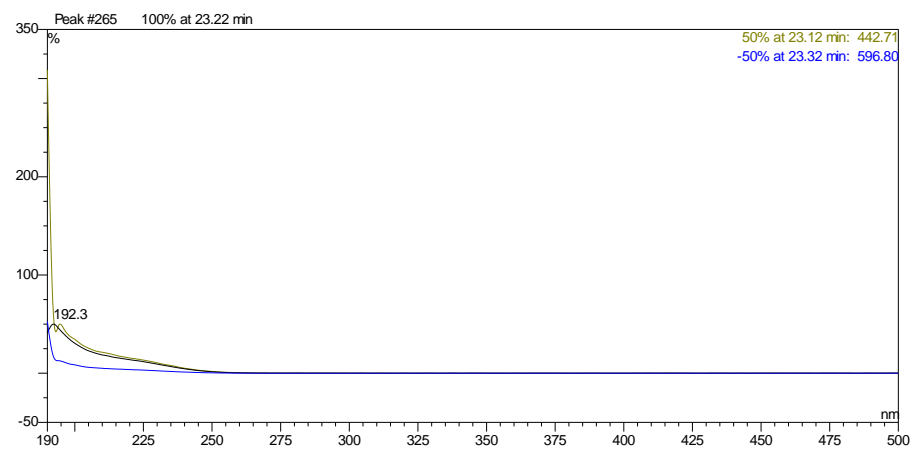
**Figure S129.** HMBC spectrum of compound **15** (500 MHz, D<sub>2</sub>O).



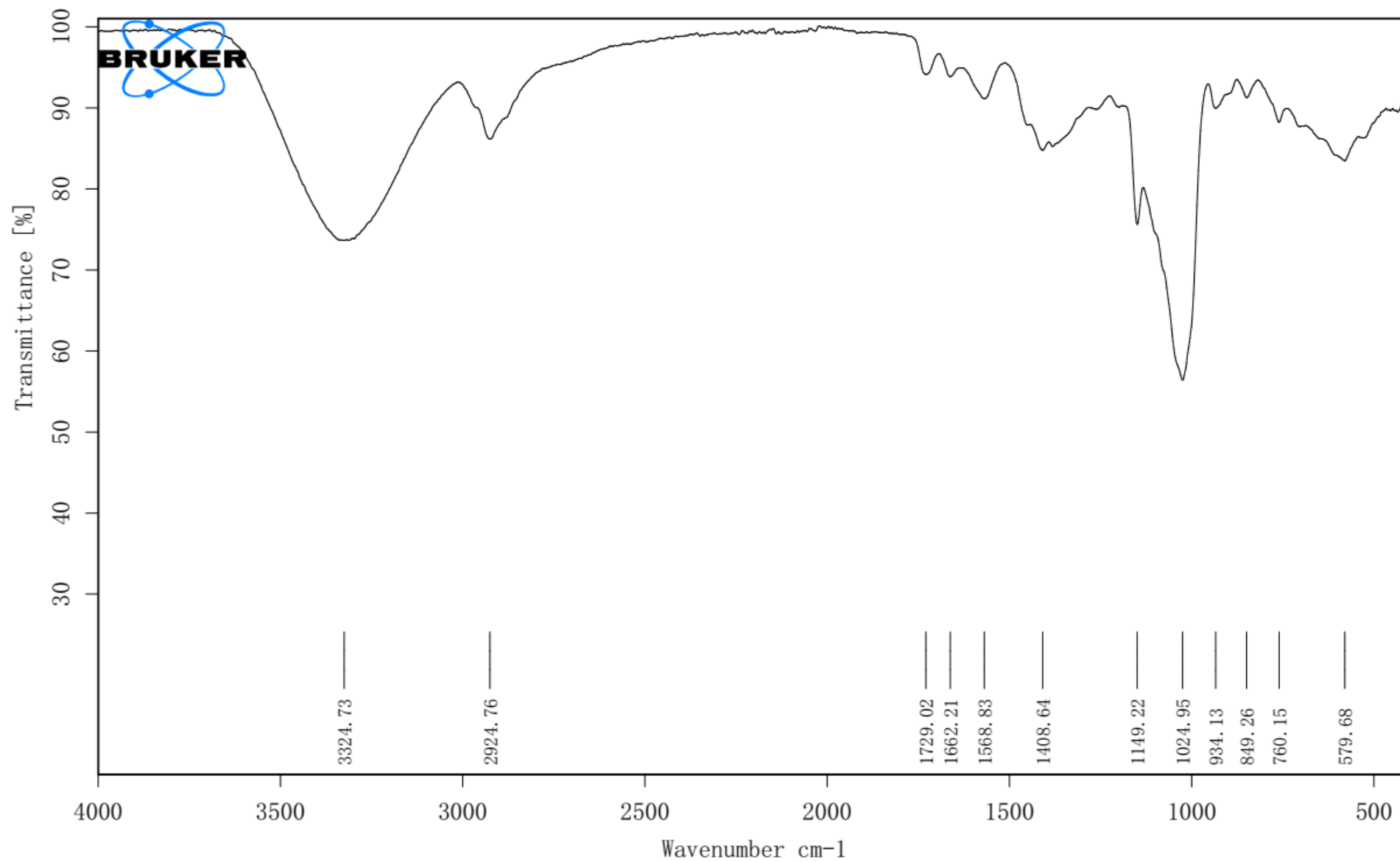
**Figure S130.** NOESY spectrum of compound **15** (500 MHz, D<sub>2</sub>O).



**Figure S131.** HRESIMS spectrum of compound **15**.

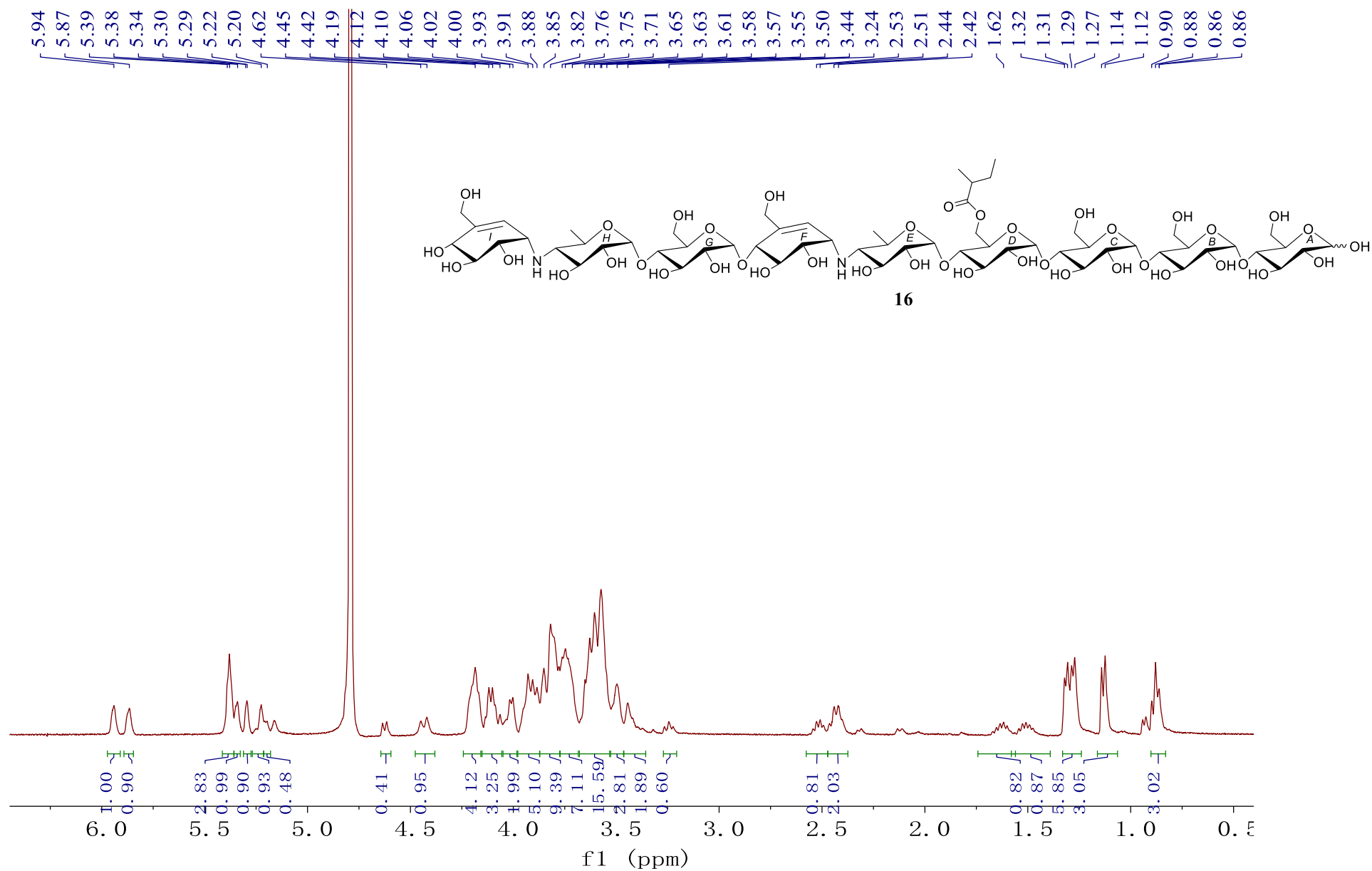


**Figure S132.** UV spectrum of compound **15**.

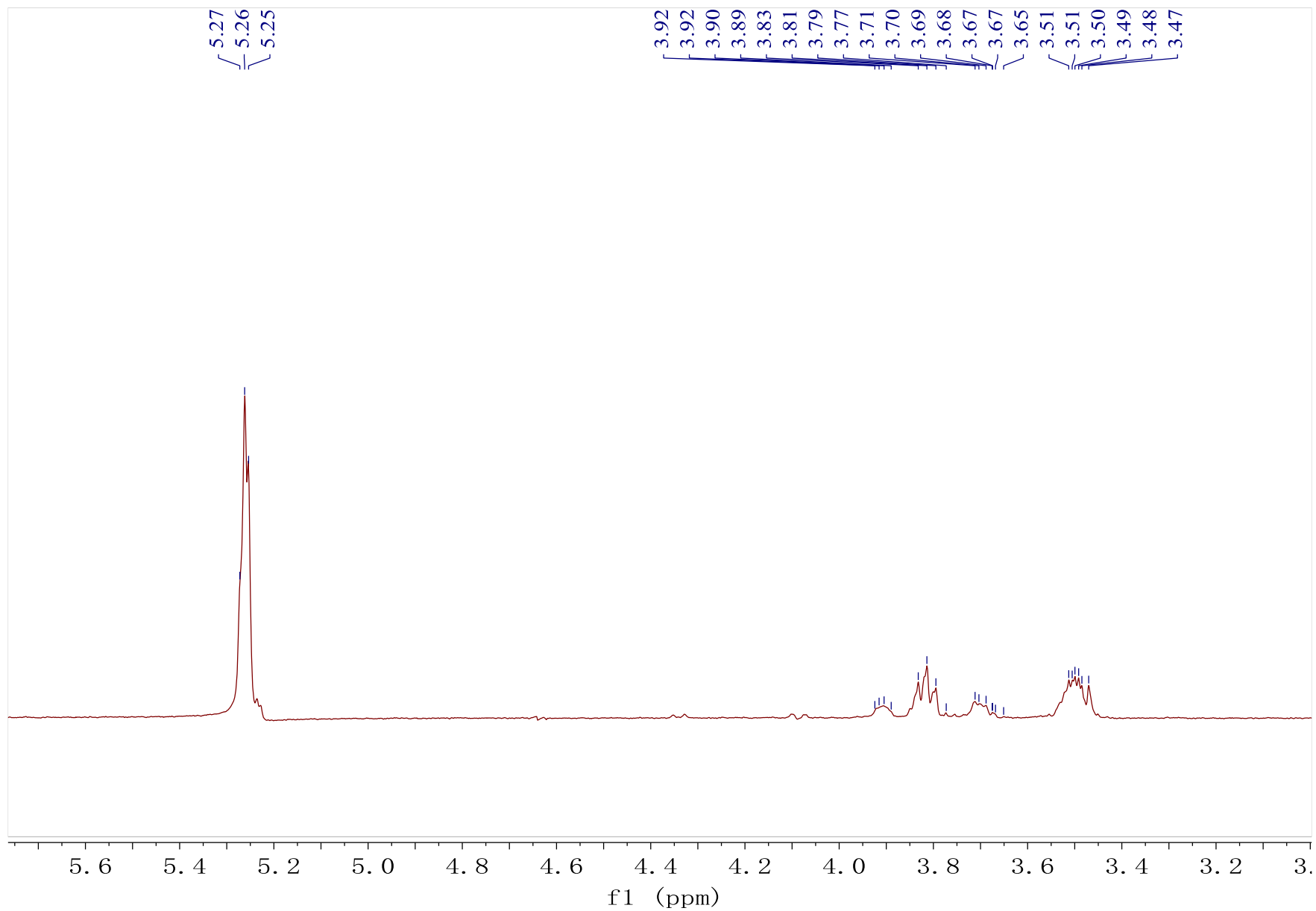


**Figure S133.** IR spectrum of compound **15**.

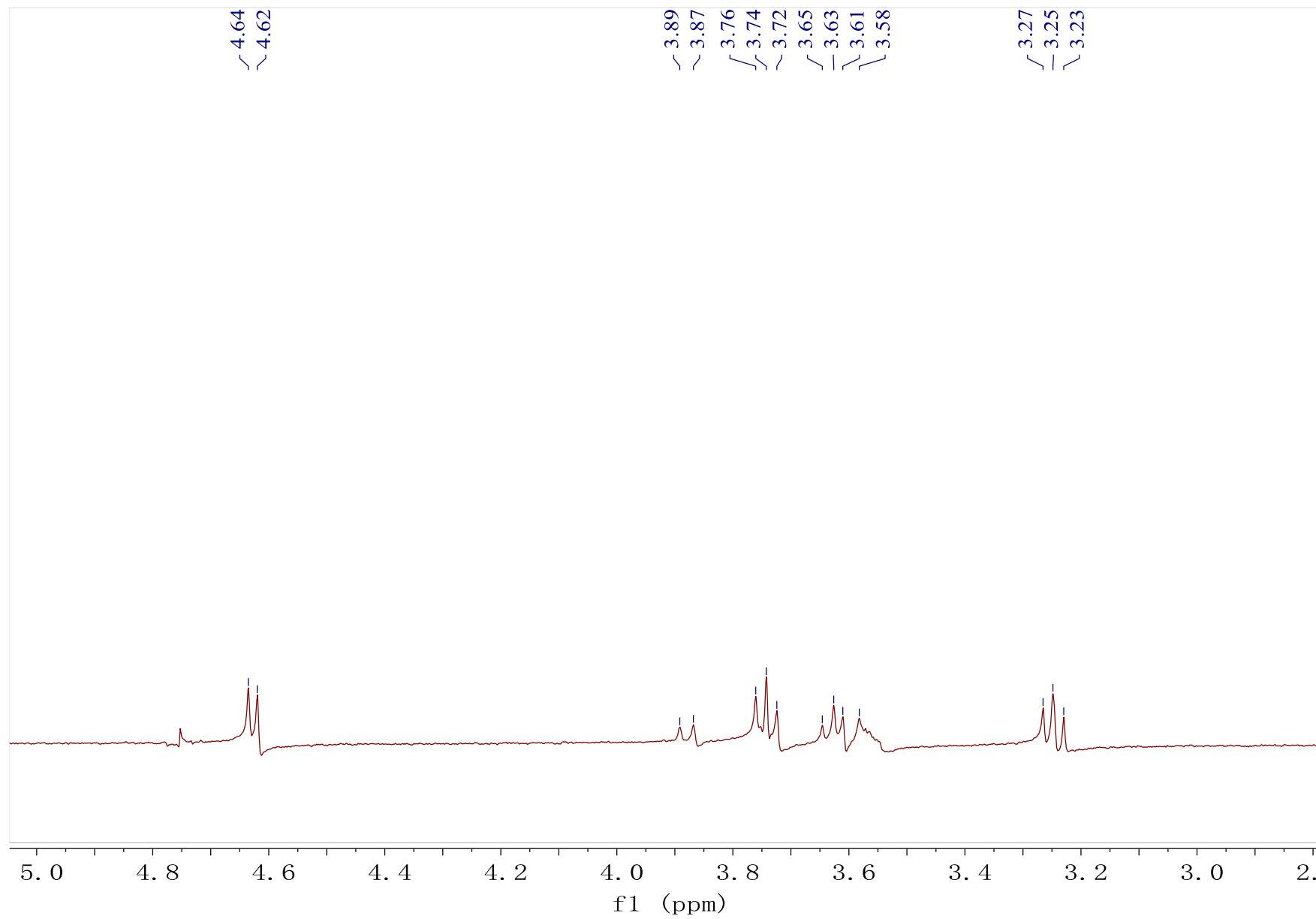




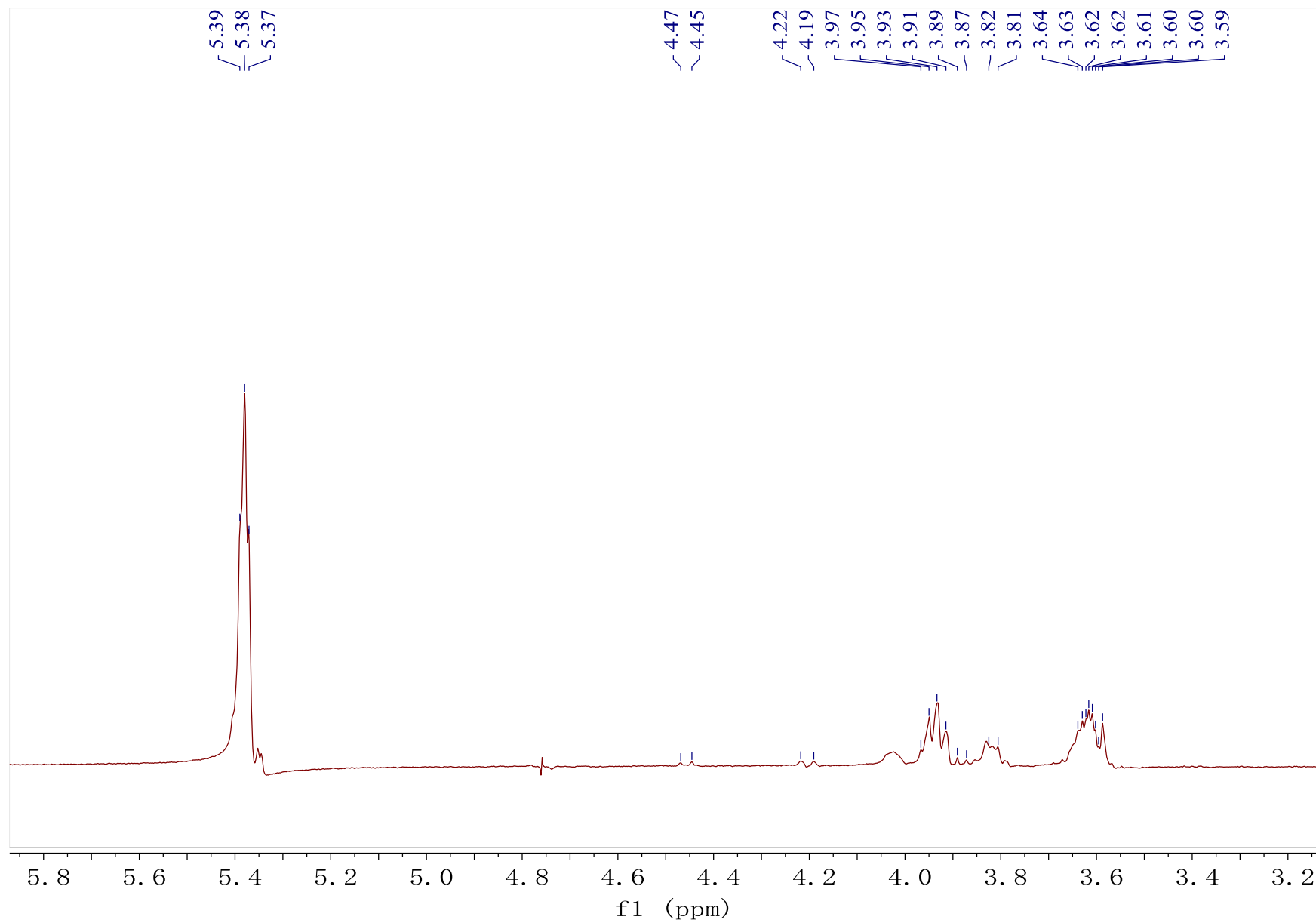
**Figure S134.**  $^1\text{H}$  NMR spectrum of compound **16** (500 MHz,  $\text{D}_2\text{O}$ ).



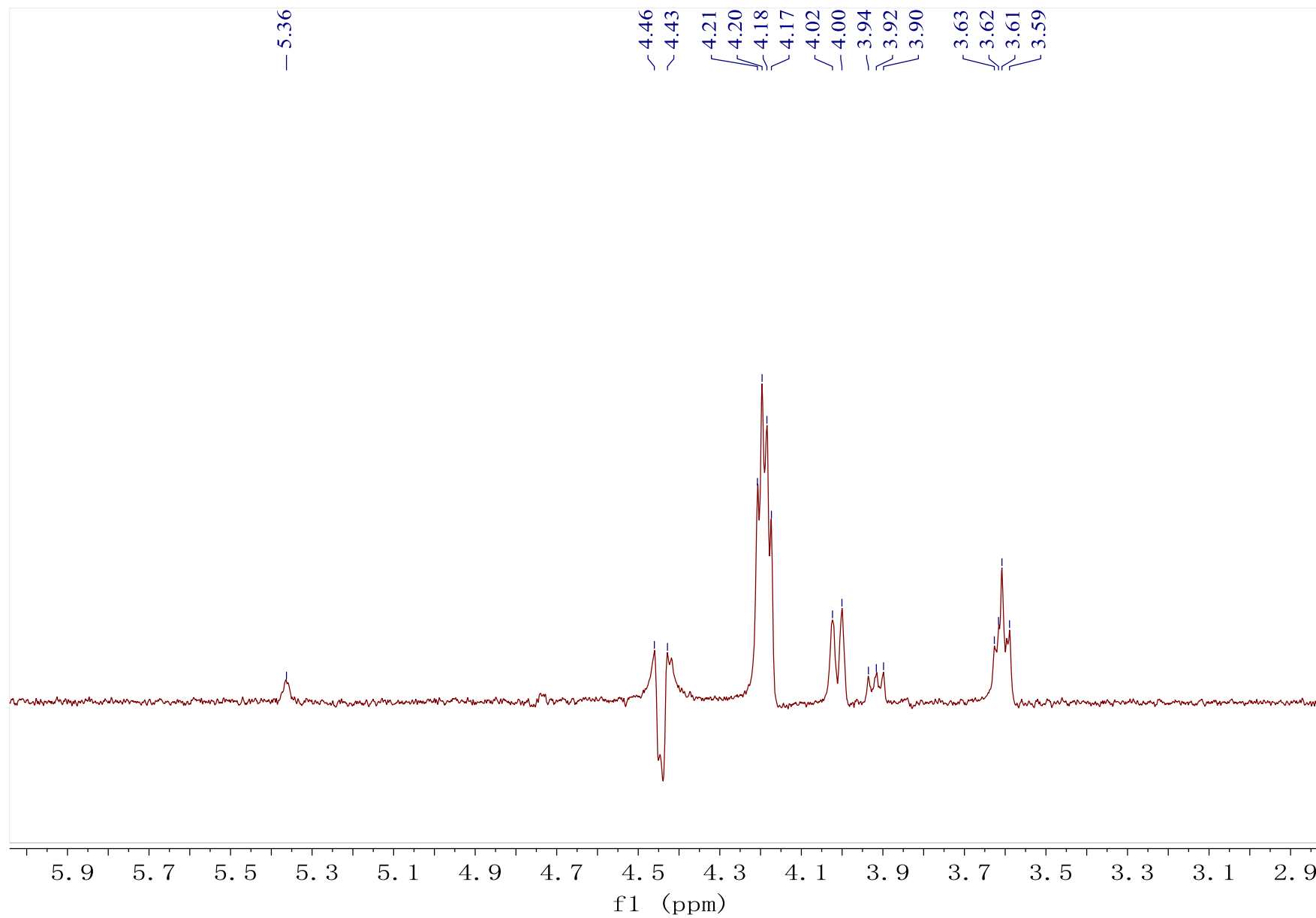
**Figure S135.** 1D-selective TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.20, H-A1 $\alpha$ ).



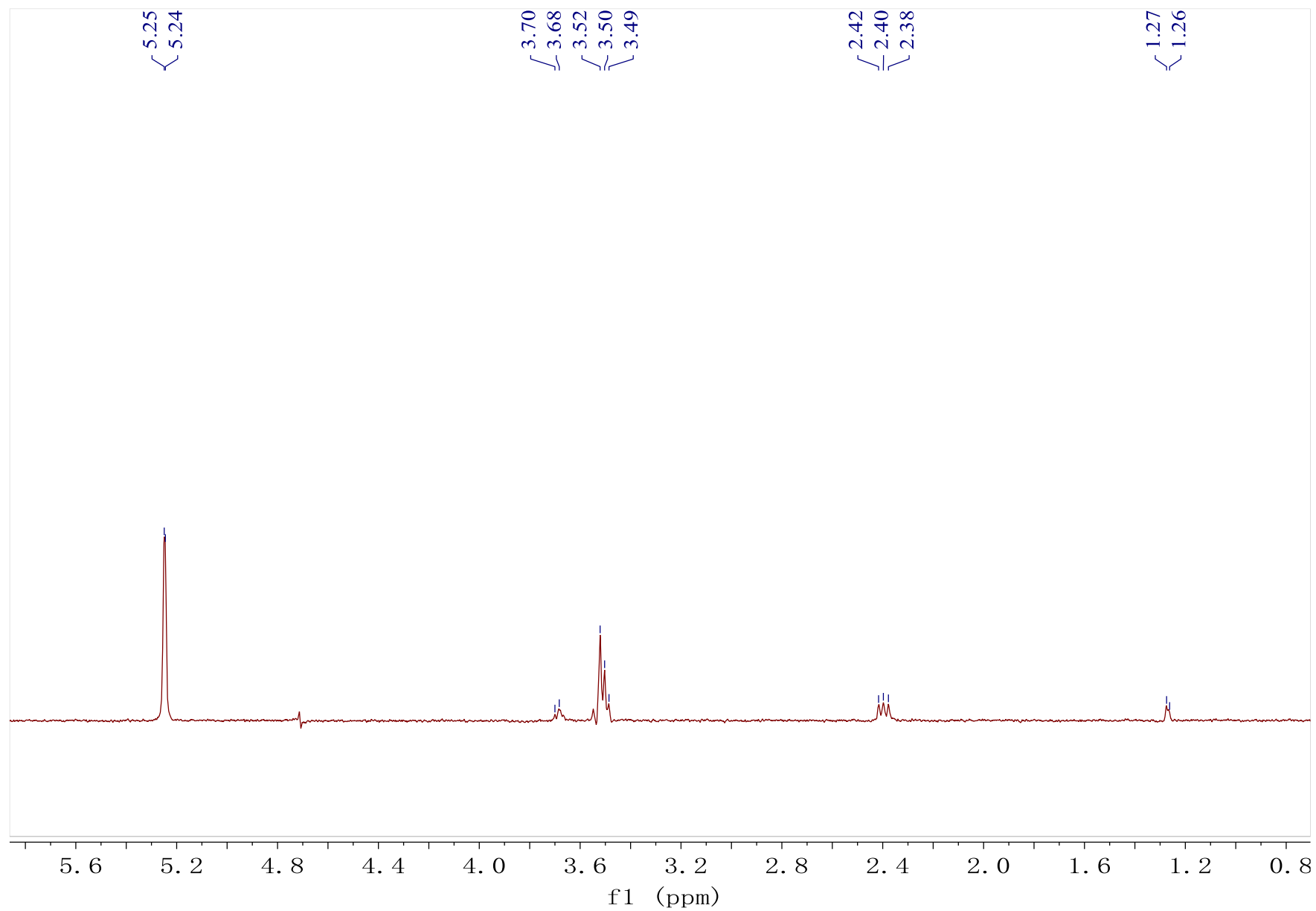
**Figure S136.** 1D-selective TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.63, H-A1 $\beta$ ).



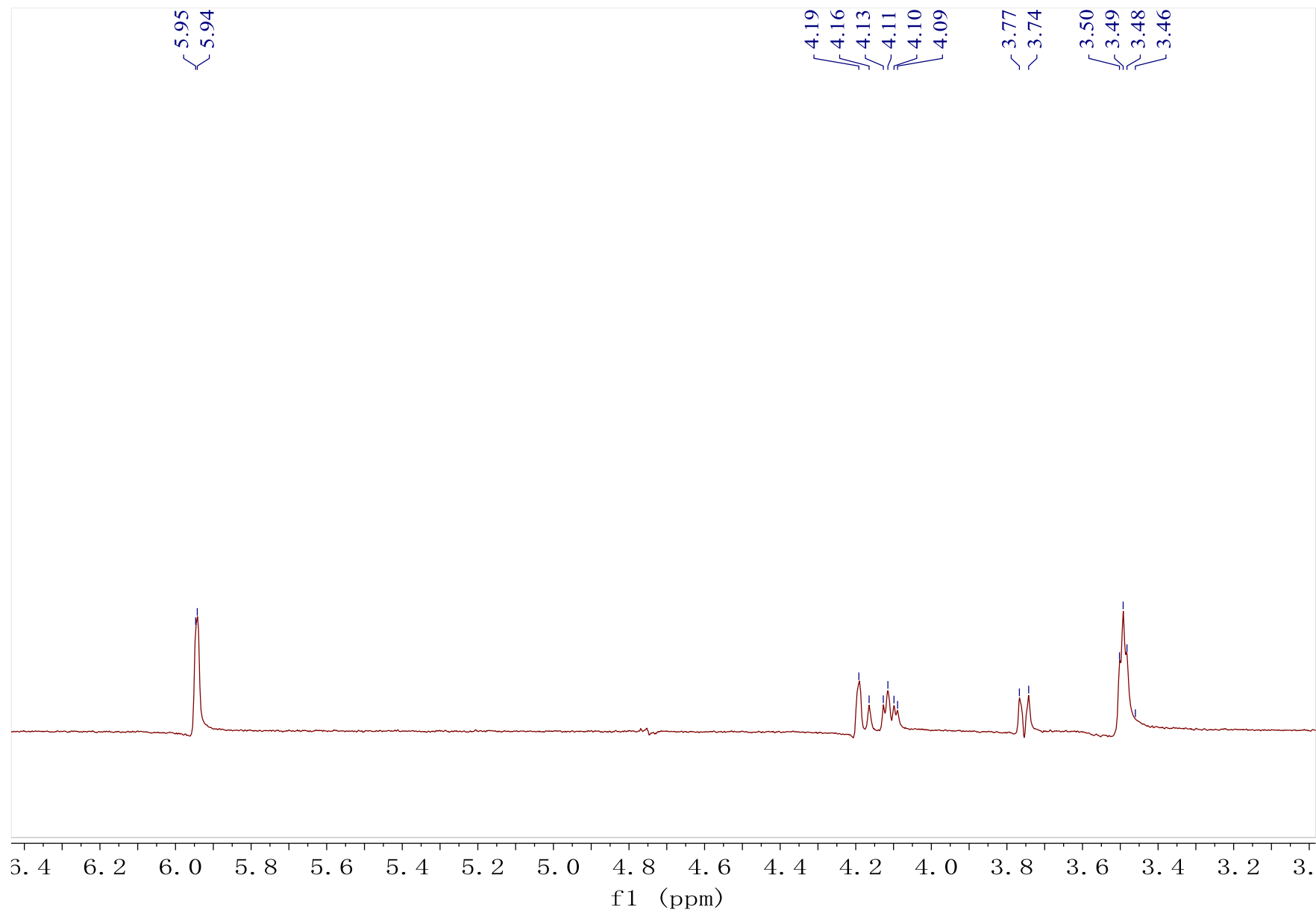
**Figure S137.** 1D-selective TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.38, H-**B1**, H-**C1**, H-**D1**).



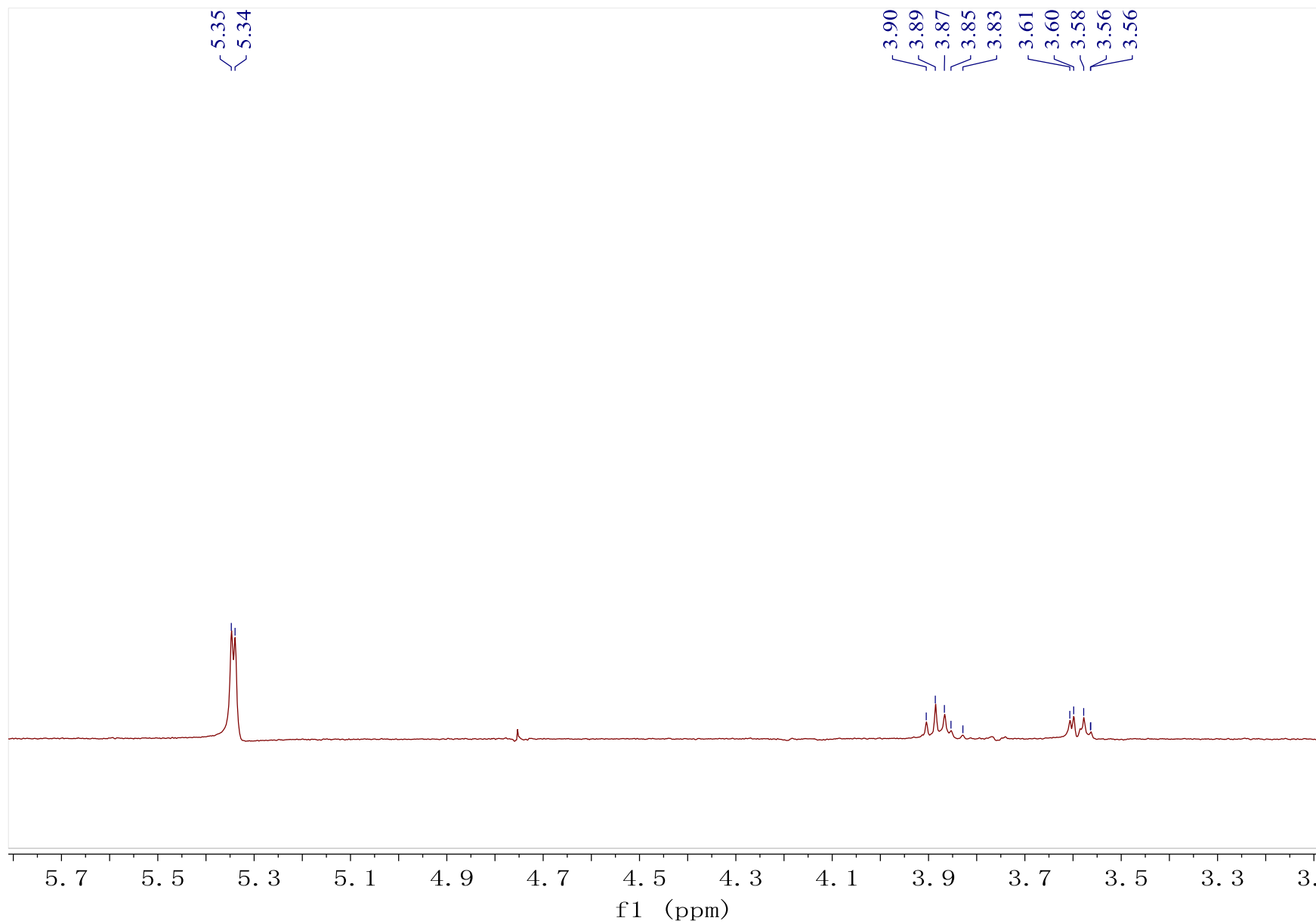
**Figure S138.** 1D-selective TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  4.43, H-D6a).



**Figure S139.** 1D-selective TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.24, H-E1).

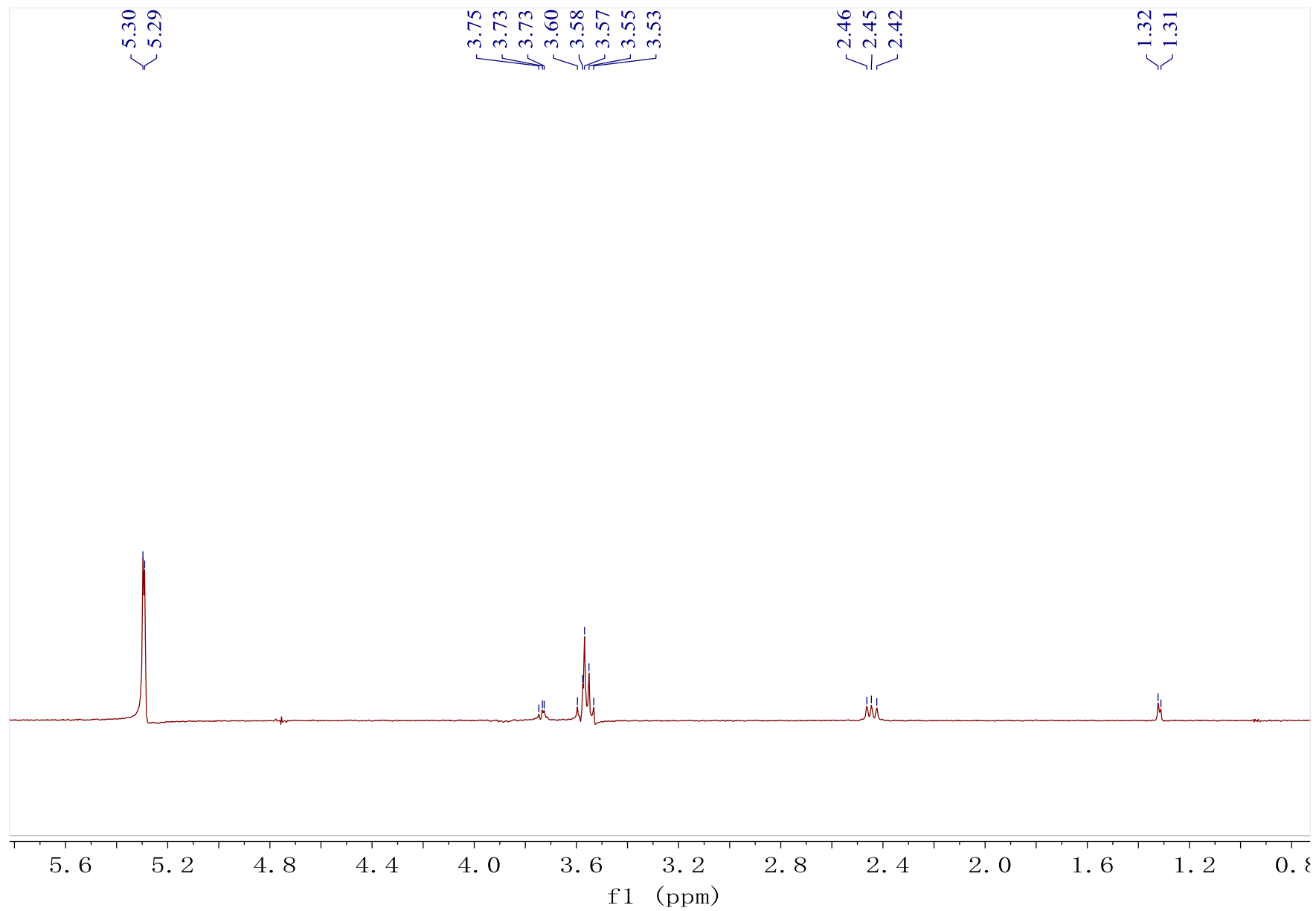


**Figure S140.** 1D-selective TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.94, H-F7).

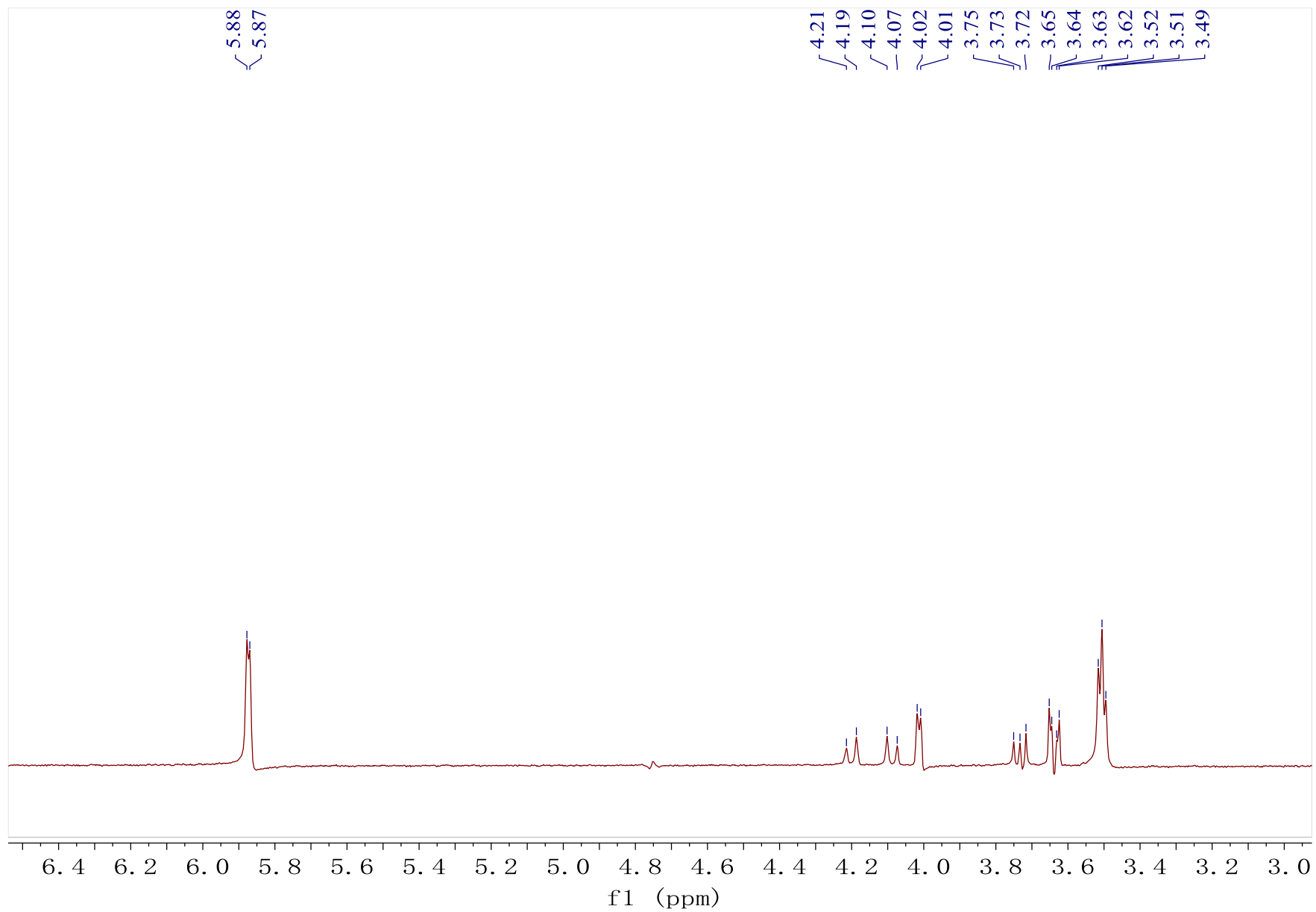


**Figure S141.** 1D-selective TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.34, H-G1).

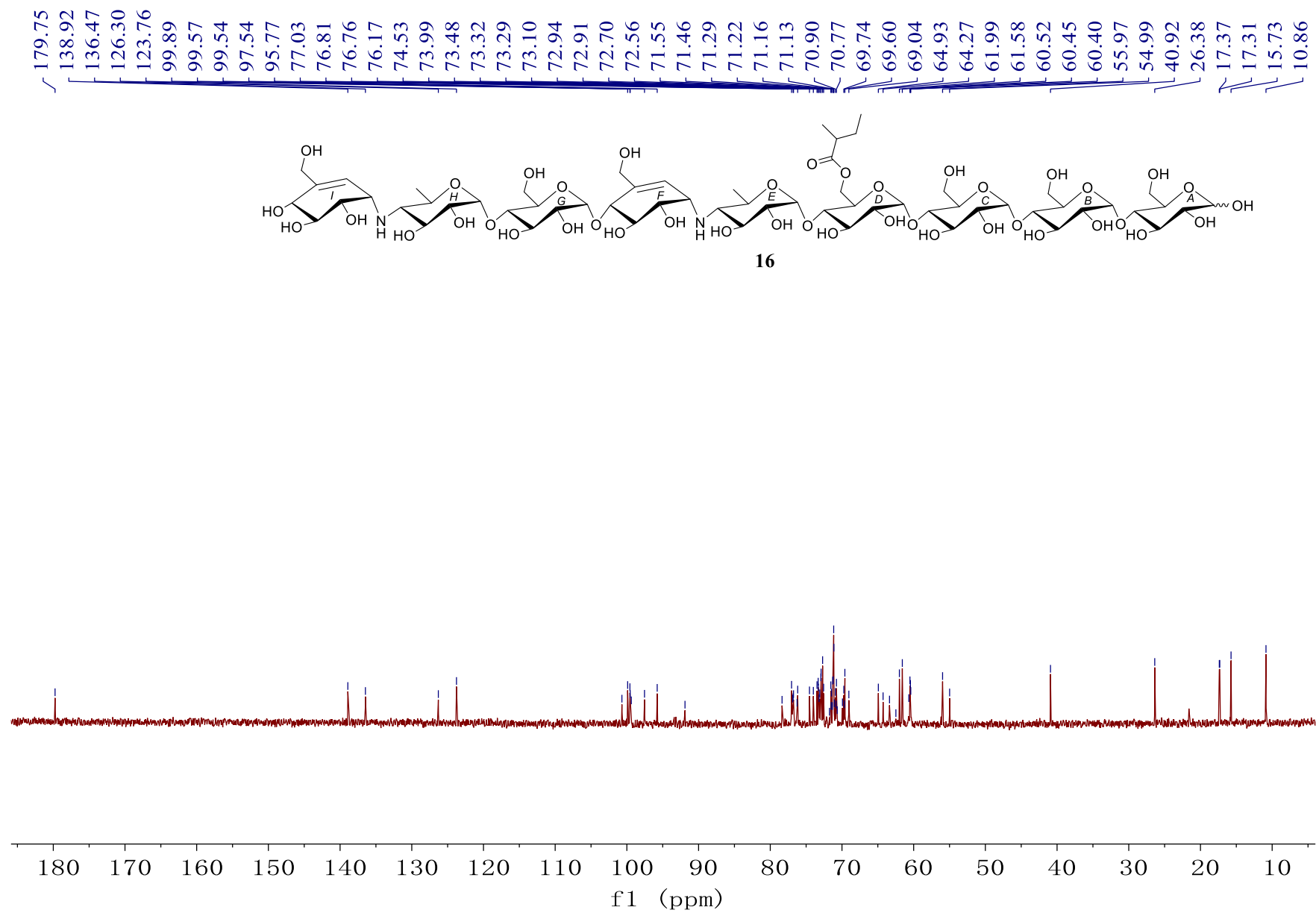




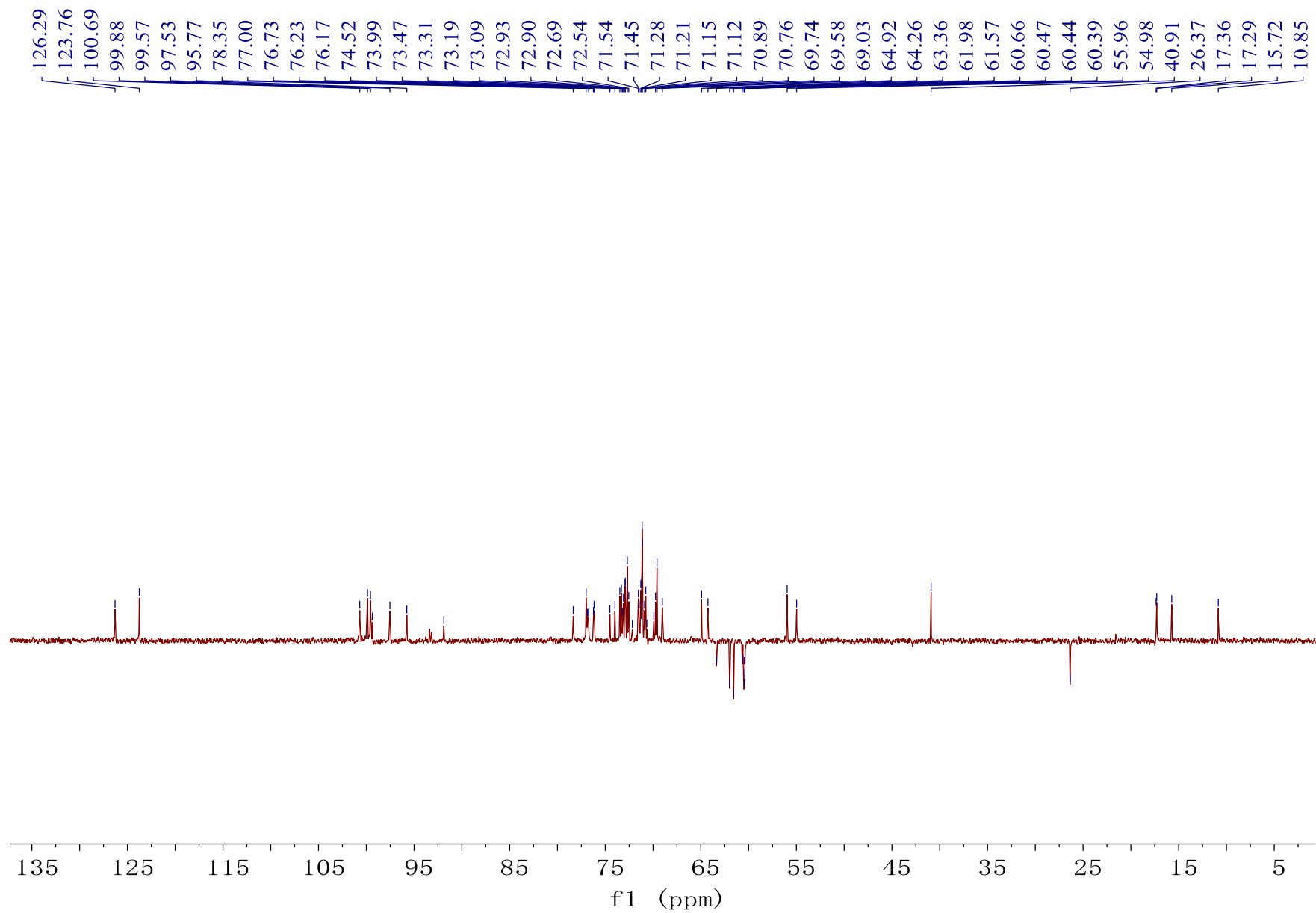
**Figure S142.** 1D-selective TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.30, H-**H1**).



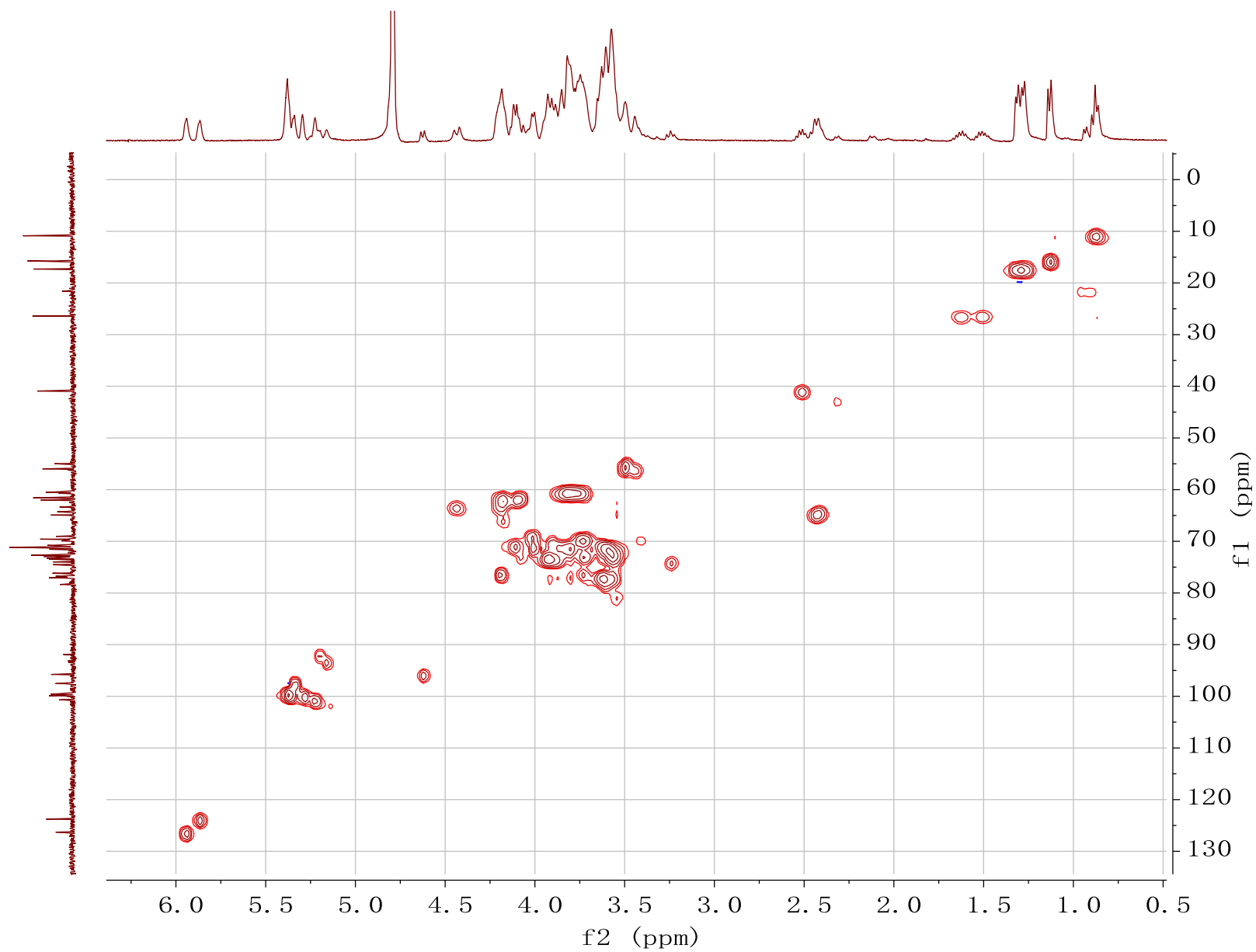
**Figure S143.** 1D-selective TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O, excitation at  $\delta$  5.87, H-**I7**).



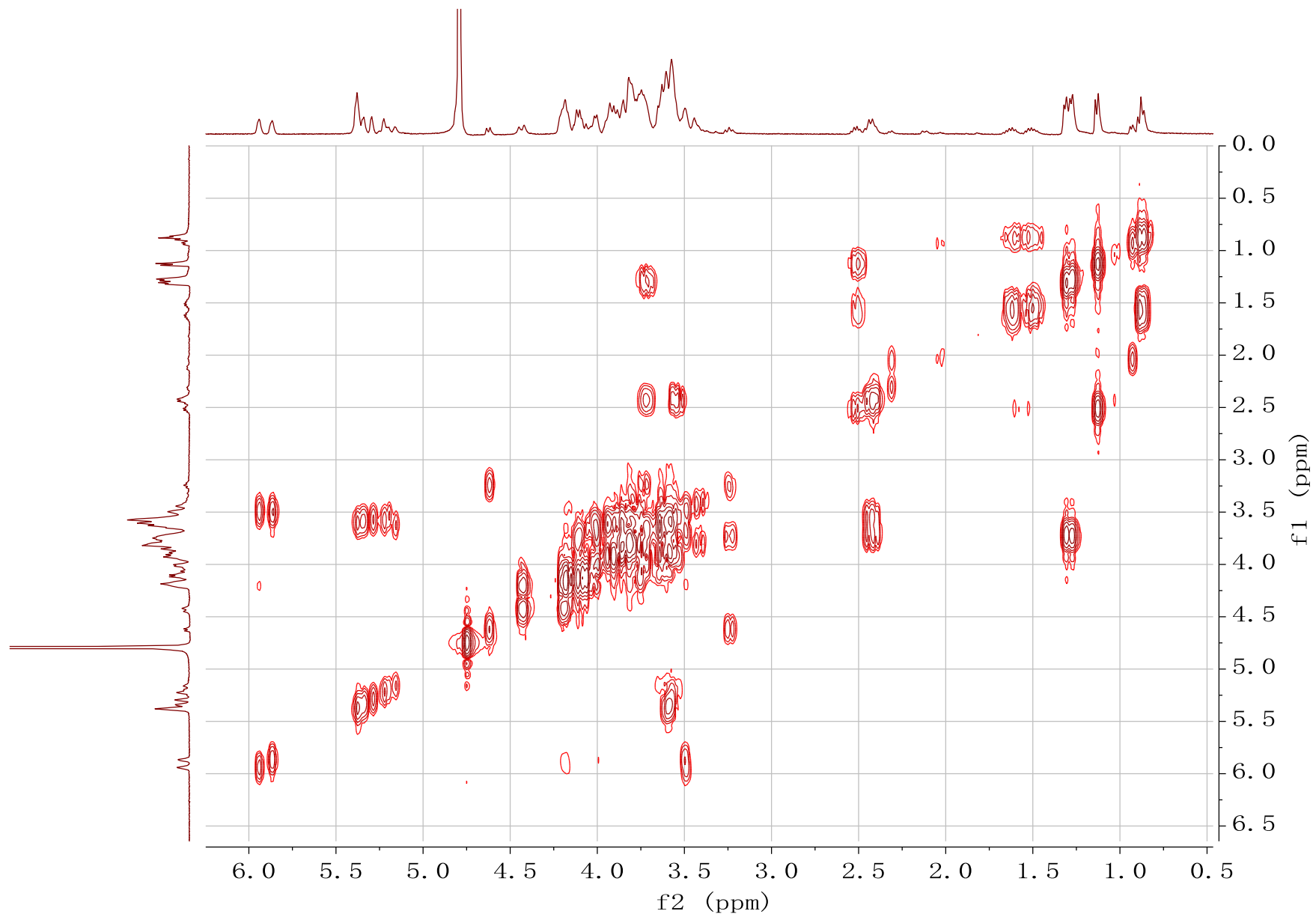
**Figure S144.**  $^{13}\text{C}$  NMR spectrum of compound **16** (125 MHz,  $\text{D}_2\text{O}$ ).



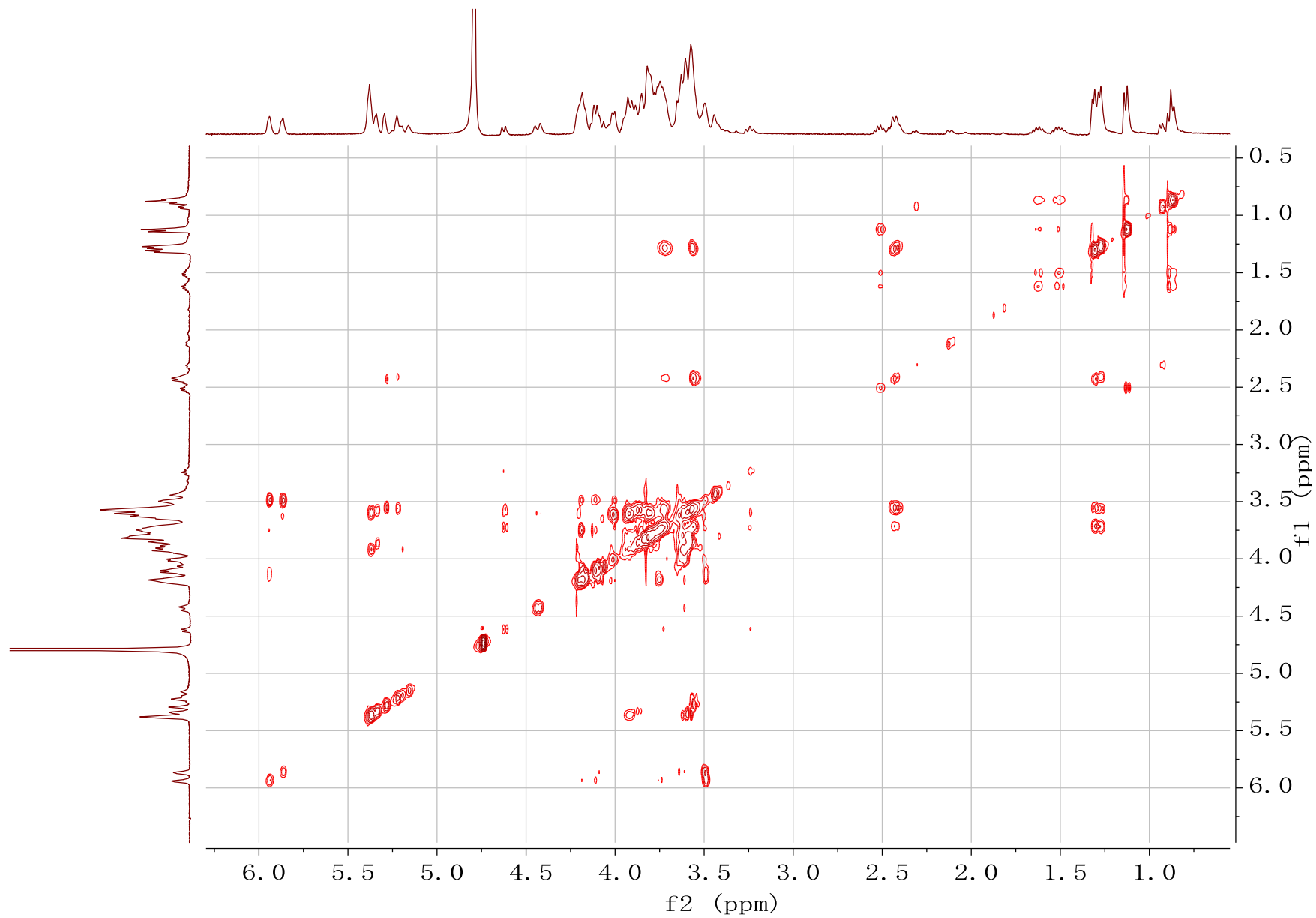
**Figure S145.** DEPT-135 spectrum of compound **16** (125 MHz, D<sub>2</sub>O).



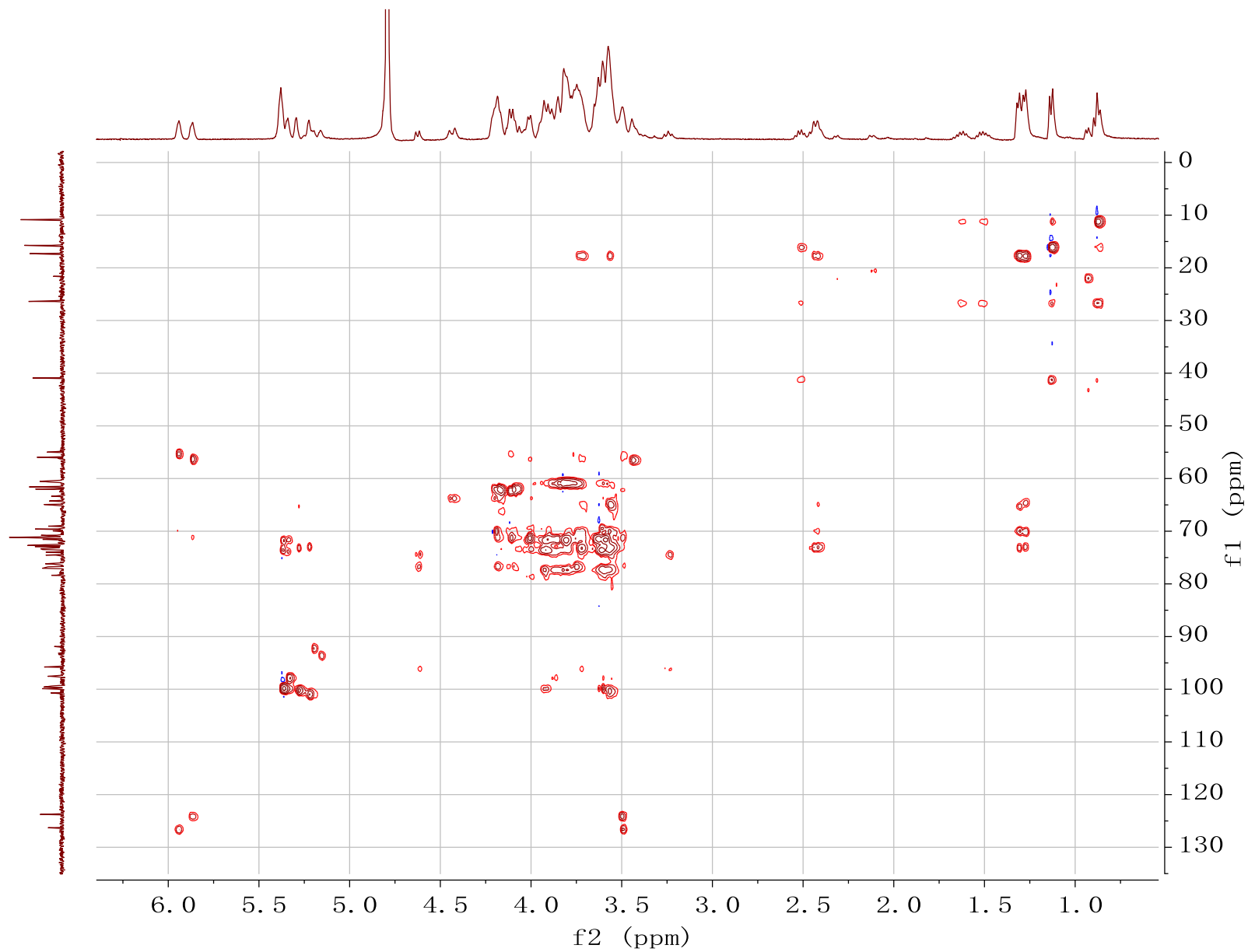
**Figure S146.** HSQC spectrum of compound **16** (500 MHz,  $\text{D}_2\text{O}$ ).



**Figure S147.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound **16** (500 MHz,  $\text{D}_2\text{O}$ ).

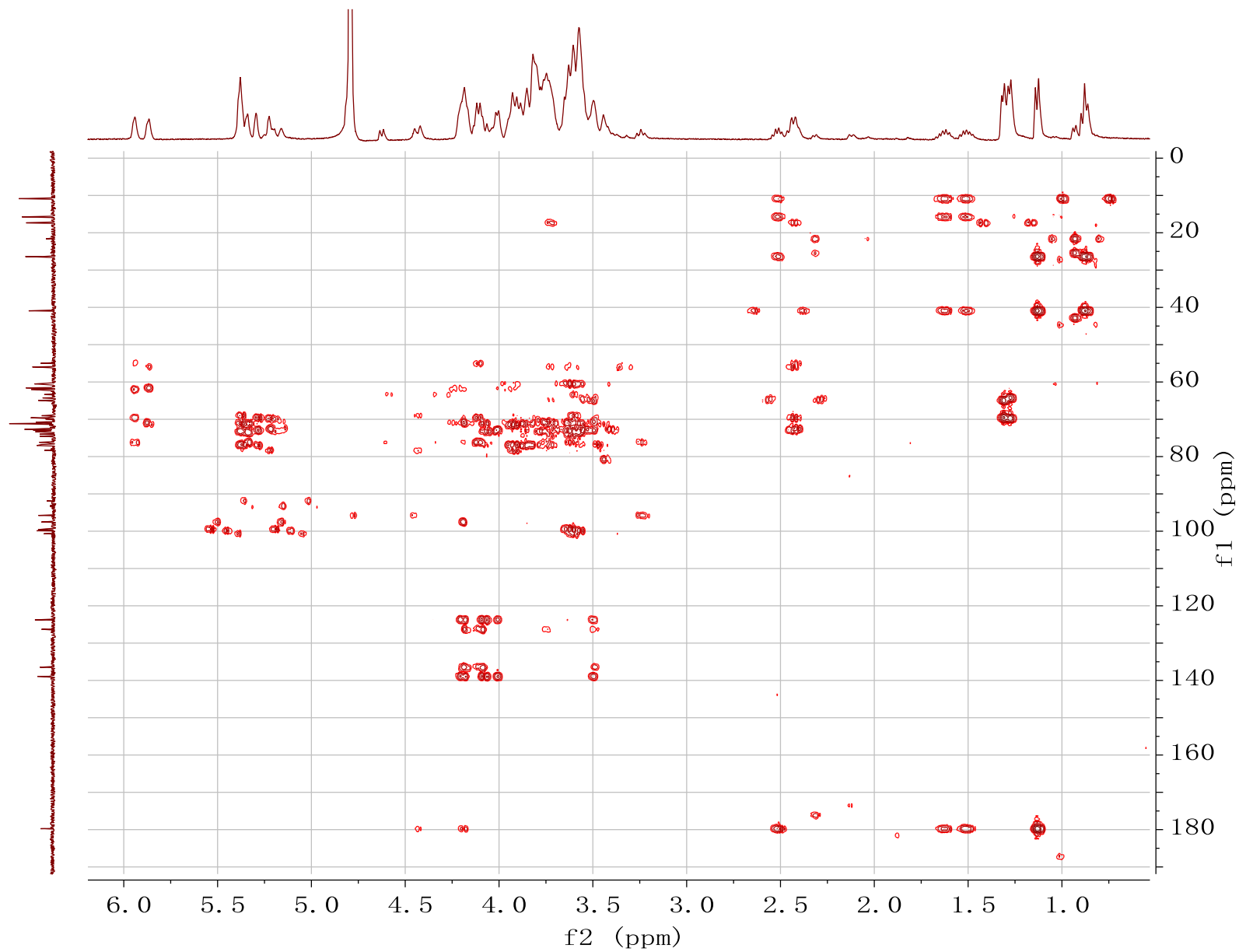


**Figure S148.** 2D-TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O).

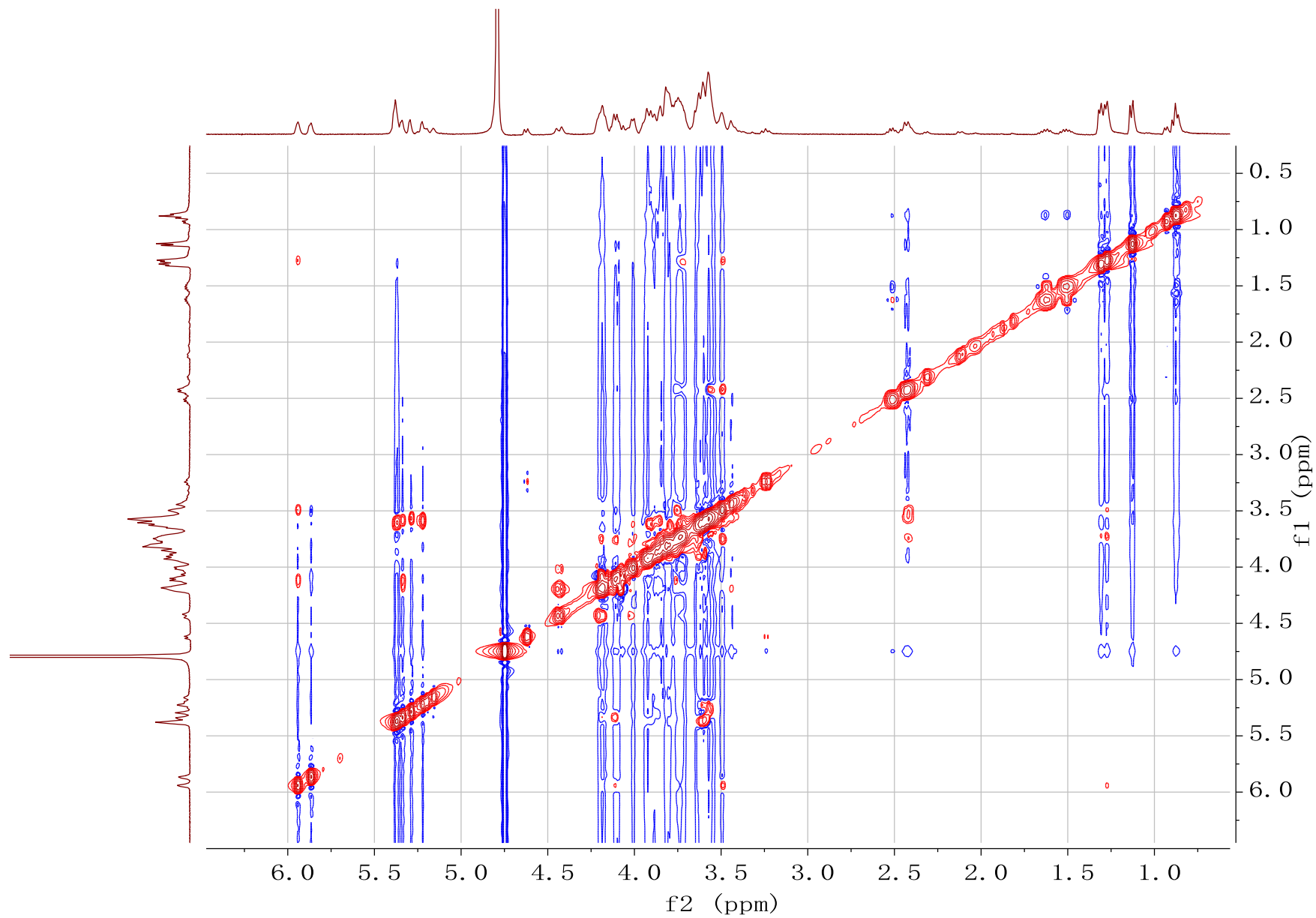


**Figure S149.** HSQC-TOCSY spectrum of compound **16** (500 MHz, D<sub>2</sub>O).

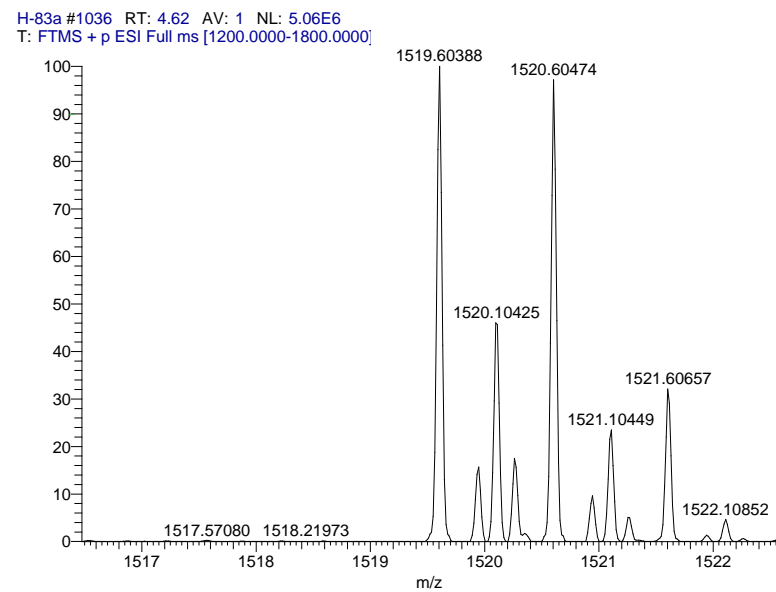




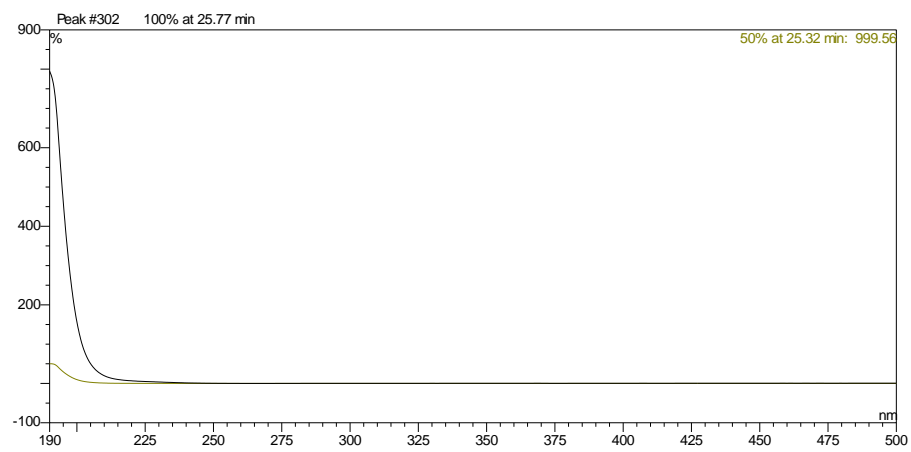
**Figure S150.** HMBC spectrum of compound **16** (500 MHz,  $D_2O$ ).



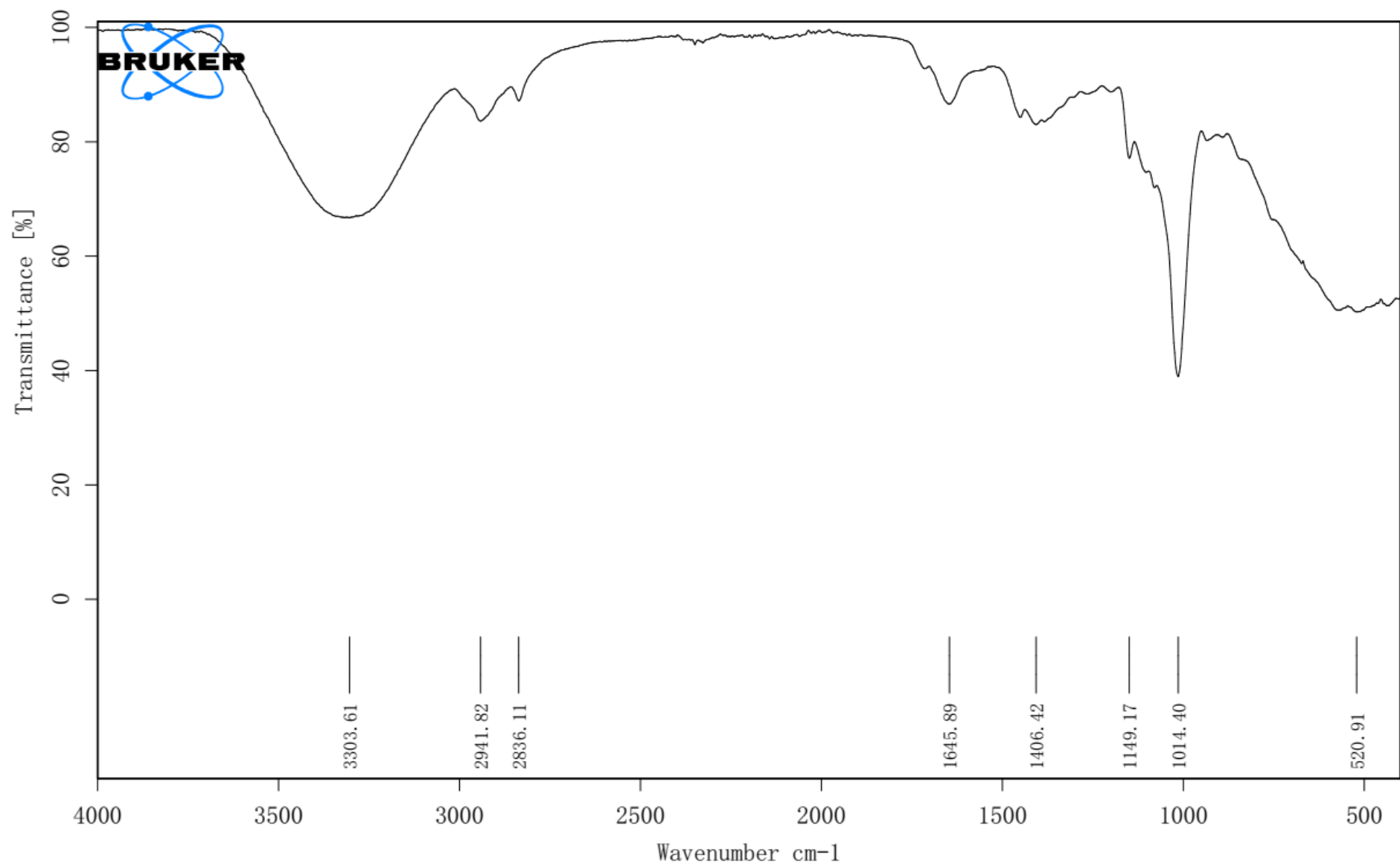
**Figure S151.** NOESY spectrum of compound **16** (500 MHz, D<sub>2</sub>O).



**Figure S152.** HRESIMS spectrum of compound **16**.



**Figure S153.** UV spectrum of compound **16**.



**Figure S154.** IR spectrum of compound **16**.