

Supplementary Data

O-Aminoalkyl-O-Trimethyl-2,3-Dehydrosilybins: Synthesis and *in vitro* Effects toward Prostate Cancer Cells

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7-O-(N,N-Diethylaminopropyl)-3,5,20-O-trimethyl-2,3-dehydrosilybin (**10**). 29% yield, yellow solid, m.p. 107-109 °C. IR (film) ν_{max} : 3419, 3182, 2981, 2844, 1624, 1602 cm⁻¹; ¹H NMR (300 MHz, CD₃OD): δ 7.59-7.48 (overlapped, 1H, H-15), 7.51 (s, 1H, H-13), 7.03 (s, 1H, H-18), 7.00-6.94 (overlapped, 2H, H-16 and H-22), 6.93 (d, J = 8.4 Hz, 1H, H-21), 6.36 (s, 1H, H-8), 6.23 (s, 1H, H-6), 4.89-4.86 (overlapped, 1H, H-11), 4.09-3.92 (overlapped, 3H, H-10 & 7-OCH₂), 3.84 (s, 3H, OCH₃), 3.83 (s, 3H, OCH₃), 3.78 (s, 3H, OCH₃), 3.75 (s, 3H, OCH₃), 3.69 (dd, J = 12.9, 2.4 Hz, 1H, H-23), 3.43 (dd, J = 12.3, 3.9 Hz, 1H, H-23), 2.74 (t, J = 7.5 Hz, 2H, 7-O-CH₂CH₂CH₂-), 2.70 (t, J = 7.2 Hz, 2H, NCH₂), 2.67 (t, J = 7.2 Hz, 2H, NCH₂), 2.02-1.86 (m, 2H, 7-O-CH₂CH₂CH₂-), 1.10 (t, J = 7.2 Hz, 6H, 2 × NCH₂CH₃); ¹³C NMR (75 MHz, CD₃OD): δ 175.5 (C-4), 165.0 (C-7), 161.7 (C-5), 159.7 (C-8a), 153.9 (C-19), 151.1 (C-20), 150.6 (C-16a), 147.2 (C-2), 145.0 (C-12a), 141.9 (C-3), 130.3 (C-17), 124.4 (C-14), 123.0 (C-15), 121.6 (C-22), 118.1 (C-16), 117.9 (C-13), 112.8 (C-21), 112.3 (C-18), 109.6 (C-4a), 97.0 (C-6), 94.1 (C-8), 80.3 (C-10), 77.5 (C-11), 67.9 (7-OCH₂), 61.9 (C-23), 60.2 (OCH₃), 56.6 (OCH₃), 56.5 (OCH₃), 56.4 (OCH₃), 50.3 (NCH₂), 47.9 (7-O-CH₂CH₂CH₂-), 26.5 (7-O-CH₂CH₂CH₂-), 11.0 (NCH₂CH₃); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₅H₄₂NO₁₀: 636.2808, found: 636.2801.

7-O-(N,N-Dibutylaminopropyl)-3,5,20-O-trimethyl-2,3-dehydrosilybin (**11**). 25% yield, yellow solid, mp. 69-71 °C. IR (film) ν_{max} : 3404, 2930, 2871, 1625, 1602, 1505 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.75 (d, J = 1.8 Hz, 1H, H-13), 7.72 (dd, J = 8.4, 2.1 Hz, 1H, H-15), 7.04 (d, J = 8.4 Hz, 1H, H-16), 7.03 (dd, J = 8.1, 1.8 Hz, 1H, H-22), 6.97 (d, J = 1.8 Hz, 1H, H-18), 6.92 (d, J = 8.4 Hz, 1H, H-21), 6.46 (d, J = 2.1 Hz, 1H, H-8), 6.31 (d, J = 2.1 Hz, 1H, H-6), 5.02 (d, J = 8.4 Hz, 1H, H-11), 4.13 (dt, J = 8.1, 2.7 Hz, 1H, H-10), 4.08 (t, J = 6.3 Hz, 2H, 7-OCH₂), 3.94 (s, 3H, OCH₃), 3.92 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.88 (s, 3H, OCH₃), 3.84 (dd, J = 12.6, 2.7 Hz, 1H, H-23), 3.57 (dd, J = 12.3, 3.6 Hz, 1H, H-23), 2.66 (t, J = 6.3 Hz, 2H, 7-O-CH₂CH₂CH₂-), 2.49 (t, J = 7.2 Hz, 4H, 2 × NCH₂), 1.98 (quin, J = 6.3 Hz, 2H, 7-O-CH₂CH₂CH₂-), 1.50-1.40 (m, 4H, 2 × NCH₂CH₂CH₂CH₃), 1.36-1.24 (m, 4H, 2 × NCH₂CH₂CH₂CH₃), 0.89 (t, J = 7.2 Hz, 6H, 2 × NCH₂CH₂CH₂CH₃); ¹³C NMR (75 MHz, CDCl₃): δ 174.2 (C-4), 163.4 (C-7), 161.1 (C-5), 158.8 (C-8a), 152.0 (C-19), 149.9 (C-20), 149.6 (C-16a), 145.3 (C-2), 143.8 (C-12a), 141.5 (C-3), 128.5 (C-17), 124.4 (C-14), 122.3 (C-15), 120.3 (C-22), 117.3 (C-16), 117.2 (C-13), 111.5 (C-21), 110.3 (C-18), 109.5 (C-4a), 96.1 (C-6), 93.0 (C-8), 78.8 (C-10), 76.4 (C-11), 66.8 (7-OCH₂), 61.7 (C-23), 60.0 (OCH₃), 56.6 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 53.9 (NCH₂), 50.5 (7-O-CH₂CH₂CH₂-), 28.9 (NCH₂CH₂CH₂CH₃), 26.7 (7-O-CH₂CH₂CH₂-), 20.8 (NCH₂CH₂CH₂CH₃), 14.2 (NCH₂CH₂CH₂CH₃); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₉H₅₀NO₁₀: 692.3434, found: 692.3431.

7-O-Pyrrolidinopropyl-3,5,20-O-trimethyl-2,3-dehydrosilybin (**12**). 29% yield, yellow solid, mp. 111-113 °C. IR (film) ν_{max} : 3415, 2933, 1625, 1605, 1578, 1506 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.74 (d, J = 1.8 Hz, 1H, H-13), 7.71 (dd, J = 8.7, 2.1 Hz, 1H, H-15), 7.03 (d, J = 8.7 Hz, 2H, H-16 & H-22), 6.97 (d, J = 1.5 Hz, 1H, H-18), 6.91 (d, J = 8.1 Hz, 1H, H-21), 6.46 (d, J = 2.1 Hz, 1H, H-8), 6.31 (d, J = 2.1 Hz, 1H, H-6), 5.01 (d, J = 8.1 Hz, 1H, H-11), 4.16-4.12 (m, 1H, H-10), 4.09 (t, J = 6.3 Hz, 2H, 7-OCH₂), 3.94 (s, 3H, OCH₃), 3.92 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.88 (s, 3H, OCH₃), 3.84 (dd, J = 12.9, 2.4 Hz, 1H, H-23), 3.56 (dd, J = 12.6, 3.9 Hz, 1H, H-23), 2.71 (t, J = 7.2 Hz, 2H, 7-O-CH₂CH₂CH₂-), 2.67-2.57 (m, 4H, 2 × pyrrolidino-CH₂), 2.07 (quin, J = 6.0 Hz, 2H, 7-O-CH₂CH₂CH₂-), 1.88-1.79 (m, 4H, 2 × pyrrolidino-CH₂); ¹³C NMR (75 MHz, CDCl₃): δ 174.2 (C-4), 163.3 (C-7), 161.0 (C-5), 158.8 (C-8a), 152.1 (C-19), 149.9 (C-20), 149.6 (C-16a), 145.3 (C-2), 143.8 (C-12a), 141.4 (C-3), 128.5 (C-17), 124.3 (C-14), 122.2 (C-15), 120.3 (C-22), 117.3 (C-16), 117.2 (C-13), 111.4 (C-21), 110.2 (C-18), 109.5 (C-4a), 96.1 (C-6), 93.0 (C-8), 78.8 (C-10), 76.4 (C-11), 66.7 (7-OCH₂), 61.7 (C-23), 60.0 (OCH₃), 56.5 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 54.3 (NCH₂), 53.0 (pyrrolidino-CH₂), 28.0 (7-O-CH₂CH₂CH₂-), 23.6 (pyrrolidino-CH₂); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₅H₄₀NO₁₀: 634.2652, found: 634.2650.

7-O-Mopholinopropyl-3,5,20-O-trimethyl-2,3-dehydrosilybin (**13**). 19% yield, yellow solid. mp. 132-133 °C. IR (film) ν_{max} : 3406, 2932, 1625, 1606, 1506, 1464, 1443 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.73 (s, 1H, H-13), 7.72 (d, J = 10.8 Hz, 1H, H-15), 7.03 (d, J = 8.4 Hz, 2H, H-16 & H-22), 6.97 (s, 1H, H-18), 6.91 (d, J = 8.1 Hz, 1H, H-21), 6.44 (s, 1H, H-8), 6.29 (s, 1H, H-6), 5.02 (d, J = 8.1 Hz, 1H, H-11), 4.18-4.04 (overlapped, 3H, H-10 & 7-OCH₂), 3.93 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.87 (s, 3H, OCH₃), 3.87-3.76 (overlapped, 4H, 2 × OCH₂ from mopholino), 3.57 (dd, J = 12.3, 3.3 Hz, 1H, H-23), 3.06-2.99 (m, 1H, H-23), 2.79-2.57 (overlapped, 6H, 3 × NCH₂), 2.18-2.05 (overlapped, 2H, 7-O-CH₂CH₂CH₂-); ¹³C NMR (75 MHz, CDCl₃): δ 174.2 (C-4), 163.2 (C-7), 161.0 (C-

5), 158.8 (C-8a), 152.1 (C-19), 149.9 (C-20), 149.6 (C-16a), 145.3 (C-2), 143.8 (C-12a), 141.4 (C-3), 128.4 (C-17), 124.3 (C-14), 122.2 (C-15), 120.3 (C-22), 117.23 (C-16), 117.17 (C-13), 111.4 (C-21), 110.2 (C-18), 109.5 (C-4a), 96.1 (C-6), 92.9 (C-8), 78.8 (C-10), 76.4 (C-11), 66.4 (7-OCH₂), 66.2 OCH₂ from morpholino), 61.7 (C-23), 60.0 (OCH₃), 56.5 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 55.4 (NCH₂), 53.4 (NCH₂), 42.3 (7-O-CH₂CH₂CH₂-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₅H₄₀NO₁₁: 650.2601, found: 650.2598.

7-O-(4-Methylpiperazin-1-yl)propyl-3,5,20-O-trimethyl-2,3-dehydrosilybin (14). 10% yield, light yellow solid. Mp. 142-143 °C. IR (film) ν_{max} : 3406, 2917, 2850, 1625, 1604, 1505 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.77-7.71 (overlapped, 2H, H-13 & H-15), 7.06 (d, *J* = 9.0 Hz, 1H, H-16), 7.04 (dd, *J* = 10.8, 1.5 Hz, 1H, H-22), 6.97 (d, *J* = 1.5 Hz, 1H, H-18), 6.92 (d, *J* = 8.4 Hz, 1H, H-21), 6.44 (d, *J* = 2.1 Hz, 1H, H-8), 6.30 (d, *J* = 1.8 Hz, 1H, H-6), 5.02 (d, *J* = 8.4 Hz, 1H, H-11), 4.17-4.01 (m, 1H, H-10), 4.06 (t, *J* = 6.0 Hz, 2H, 7-OCH₂), 3.94 (s, 3H, OCH₃), 3.92 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.89 (s, 3H, OCH₃), 3.85 (dd, *J* = 12.3, 2.1 Hz, 1H, H-23), 3.57 (dd, *J* = 12.6, 3.9 Hz, 1H, H-23), 2.95-2.72 (overlapped, 8H, 4 × NCH₂ from piperazin), 2.67 (t, *J* = 7.2 Hz, 2H, NCH₂), 2.55 (s, 3H, NCH₃), 2.07-2.02 (m, 2H, 7-O-CH₂CH₂CH₂-); ¹³C NMR (75 MHz, CDCl₃): δ 174.2 (C-4), 163.2 (C-7), 161.1 (C-5), 158.8 (C-8a), 152.0 (C-19), 150.0 (C-20), 149.6 (C-16a), 145.3 (C-2), 143.8 (C-12a), 141.5 (C-3), 128.4 (C-17), 124.4 (C-14), 122.4 (C-15), 120.3 (C-22), 117.2 (C-16), 117.2 (C-13), 111.5 (C-21), 110.3 (C-18), 109.6 (C-4a), 96.2 (C-6), 92.8 (C-8), 78.8 (C-10), 76.5 (C-11), 66.3 (7-OCH₂), 61.7 (C-23), 60.1 (OCH₃), 56.6 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 54.3 (NCH₂), 54.0 (NCH₂ from piperazin), 50.9 (NCH₂ from piperazin), 44.5 (NCH₃), 26.1 (7-O-CH₂CH₂CH₂-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₆H₄₃N₂O₁₀: 663.2917, found: 663.2917.

3-O-(4'-Bromo)butyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (18). 62% Yield, ¹H NMR (300 MHz, CDCl₃): δ 7.71 (s, 1H), 7.69 (dd, *J* = 7.2, 1.5 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 1.8 Hz, 1H), 6.92 (d, *J* = 8.1 Hz, 1H), 6.46 (d, *J* = 2.4 Hz, 1H), 6.32 (d, *J* = 2.4 Hz, 1H), 5.03 (d, *J* = 8.1 Hz, 1H), 4.16-4.09 (m, 1H), 4.03 (t, *J* = 6.0 Hz, 2H), 3.93 (s, 3H), 3.91 (s, 3H), 3.90 (s, 3H), 3.86 (s, 3H), 3.81 (dd, *J* = 7.2, 2.4 Hz, 1H), 3.57 (dd, *J* = 12.3, 3.6 Hz, 1H), 3.46 (t, *J* = 6.6 Hz, 2H), 2.09-2.00 (m, 2H), 1.90-1.81 (m, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 174.2, 164.1, 161.1, 158.9, 152.6, 149.9, 149.6, 145.3, 143.8, 140.4, 128.5, 124.4, 122.4, 120.3, 117.3, 117.1, 111.4, 110.3, 109.5, 95.9, 92.5, 78.8, 76.4, 61.7, 56.5, 56.2, 56.1, 55.9, 34.1, 28.8.

3-O-(5'-Bromo)pentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (19). 77% yield, ¹H NMR (300 MHz, CDCl₃): δ 7.72 (s, 1H), 7.71 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.03 (d, *J* = 8.1 Hz, 2H), 6.97 (d, *J* = 1.5 Hz, 1H), 6.92 (d, *J* = 8.4 Hz, 1H), 6.45 (d, *J* = 2.1 Hz, 1H), 6.31 (d, *J* = 2.1 Hz, 1H), 5.03 (d, *J* = 8.4 Hz, 1H), 4.14-4.11 (m, 1H), 4.02 (t, *J* = 6.3 Hz, 2H), 3.93 (s, 3H), 3.91 (s, 3H), 3.90 (s, 3H), 3.85 (s, 3H), 3.85 (d, *J* = 12.6 Hz, 1H), 3.57 (d, *J* = 11.7 Hz, 1H), 3.34 (t, *J* = 6.9 Hz, 2H), 1.85 (quin, *J* = 7.2 Hz, 2H), 1.74 (quin, *J* = 7.2 Hz, 2H), 1.59-1.49 (m, 2H).

3-O-(N,N-Dimethylamino)butyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (21). 86% yield, yellow wax. IR (film) ν_{max} : 3397, 2938, 1625, 1603, 1505, 1460 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.72 (dd, *J* = 8.7 Hz, 2.1 Hz, 1H, H-15), 7.72 (d, *J* = 1.8 Hz, 1H, H-13), 7.04 (d, *J* = 9.3 Hz, 1H, H-16), 7.03 (dd, *J* = 8.1, 1.8 Hz, 1H, H-22), 6.97 (d, *J* = 1.5 Hz, 1H, H-18), 6.91 (d, *J* = 8.1 Hz, 1H, H-21), 6.45 (d, *J* = 2.1 Hz, 1H, H-8), 6.31 (d, *J* = 2.1 Hz, 1H, H-6), 5.04 (d, *J* = 8.1 Hz, 1H, H-11), 4.13 (dt, *J* = 7.8, 3.3 Hz, 1H, H-10), 4.05-401 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.86 (s, 3H, OCH₃), 3.85-3.81 (overlapped, 1H, H-23), 3.57 (dd, *J* = 12.3, 3.9 Hz, 1H, H-23), 2.45 (t, *J* = 7.5 Hz, 2H, NCH₂), 2.29 (s, 6H, 2 × NCH₃), 1.77-1.65 (m, 4H, 3-OCH₂CH₂CH₂); ¹³C NMR (75 MHz, CDCl₃): δ 174.1 (C-4), 163.9 (C-7), 161.0 (C-5), 158.8 (C-8a), 152.3 (C-16a), 149.7 (C-19), 149.4 (C-20), 145.2 (C-2), 143.6 (C-12a), 140.4 (C-3), 128.5 (C-17), 124.3 (C-14), 122.4 (C-15), 120.2 (C-22), 117.2 (C-13), 117.0 (C-16), 111.3 (C-21), 110.2 (C-18), 109.4 (C-4a), 95.8 (C-6), 92.3 (C-8), 78.7 (C-10), 76.2 (C-11), 71.8 (3-OCH₂), 61.5 (C-23), 59.1 (OCH₃), 56.4 (OCH₃), 56.1 (OCH₃), 56.0 (OCH₃), 55.7 (NCH₂), 44.8 (NCH₃), 27.9 (3-O-CH₂CH₂CH₂-), 23.5 (3-O-CH₂CH₂CH₂-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₄H₄₀NO₁₀: 622.2652, found: 622.2651.

3-O-(*N,N*-Dimethylamino)pentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (22). 76% yield, yellow solid, mp. 90-91 °C. IR (film) ν_{max} : 3398, 2919, 1625, 1608, 1509 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.74-7.67 (overlapped, 2H, H-15 & H-13), 7.03 (d, J = 8.7 Hz, 2H, H-16 & H-22), 6.98 (d, J = 1.5 Hz, 1H, H-18), 6.91 (d, J = 8.1 Hz, 1H, H-21), 6.45 (d, J = 2.1 Hz, 1H, H-8), 6.31 (d, J = 2.1 Hz, 1H, H-6), 5.06 (d, J = 7.8 Hz, 1H, H-11), 4.14 (dt, J = 7.8, 3.3 Hz, 1H, H-10), 4.03-3.98 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.89 (s, 3H, OCH₃), 3.85 (s, 3H, OCH₃), 3.85-3.81 (overlapped, 1H, H-23), 3.57 (dd, J = 12.6, 3.9 Hz, 1H, H-23), 2.42 (t, J = 7.5 Hz, 2H, NCH₂), 2.34 (s, 6H, 2 × NCH₃), 1.72 (quin, J = 6.9 Hz, 2H, 3-OCH₂CH₂CH₂CH₂CH₂), 1.61-1.51 (m, 2H, 3-OCH₂CH₂CH₂CH₂CH₂), 1.47-1.37 (m, 2H, 3-OCH₂CH₂CH₂CH₂CH₂); ¹³C NMR (75 MHz, CDCl₃): δ 174.1 (C-4), 163.9 (C-7), 161.0 (C-5), 158.8 (C-8a), 152.3 (C-16a), 149.7 (C-19), 149.4 (C-20), 145.1 (C-2), 143.6 (C-12a), 140.5 (C-3), 128.5 (C-17), 124.3 (C-14), 122.4 (C-15), 120.2 (C-22), 117.2 (C-13), 116.9 (C-16), 111.3 (C-21), 110.2 (C-18), 109.4 (C-4a), 95.8 (C-6), 92.3 (C-8), 78.7 (C-10), 76.1 (C-11), 72.0 (3-OCH₂), 61.5 (C-23), 59.1 (OCH₃), 56.4 (OCH₃), 56.0 (OCH₃), 55.9 (OCH₃), 55.7 (NCH₂), 44.5 (NCH₃), 29.9 (3-OCH₂CH₂CH₂CH₂CH₂), 26.1 (3-OCH₂CH₂CH₂CH₂CH₂); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₅H₄₂NO₁₀: 636.2809, found: 636.2807.

3-O-(*N,N*-Diethylamino)propyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (23). 90% yield, yellow wax. IR (film) ν_{max} : 3375, 2936, 2837, 1624, 1604, 1579, 1517, 1505, 1492, 1462 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 7.81-7.56 (overlapped, 1H, H-15), 7.68 (s, 1H, H-13), 7.06 (d, J = 9.0 Hz, 1H, H-16), 7.03 (d, J = 9.3 Hz, 1H, H-22), 6.97 (s, 1H, H-18), 6.91 (d, J = 8.1 Hz, 1H, H-21), 6.47 (s, 1H, H-8), 6.33 (s, 1H, H-6), 5.03 (d, J = 7.9 Hz, 1H, H-11), 4.16-4.13 (m, 1H, H-10), 4.04-4.01 (m, 2H, 3-OCH₂), 3.94 (s, 3H, OCH₃), 3.91 (s, 6H, 2 \times OCH₃), 3.86 (s, 3H, OCH₃), 3.88-3.84 (overlapped, 1H, H-23), 3.57 (dd, J = 12.9, 2.7 Hz, 1H, H-23), 3.07 (t, J = 6.9 Hz, 2H, NCH₂), 2.90-2.83 (m, 4H, 2 \times NCH₂CH₃), 2.86-2.78 (m, 2H, 3-OCH₂CH₂), 1.19 (t, J = 6.9 Hz, 6H, 2 \times NCH₂CH₃); ^{13}C NMR (75 MHz, CDCl_3): δ 174.2 (C-4), 164.2 (C-7), 161.1 (C-5), 159.0 (C-8a), 152.9 (C-16a), 149.9 (C-19), 149.6 (C-20), 145.6 (C-2), 143.9 (C-12a), 140.2 (C-3), 128.5 (C-17), 124.1 (C-14), 122.4 (C-15), 120.4 (C-22), 117.4 (C-13), 117.3 (C-16), 111.5 (C-21), 110.3 (C-18), 109.4 (C-4a), 96.1 (C-6), 92.5 (C-8), 78.9 (C-10), 76.5 (C-11), 70.0 (3-OCH₂), 61.7 (C-23), 56.6 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 55.9 (OCH₃), 49.9 (NCH₂), 46.9 (NCH₂CH₃), 26.4 (3-O-CH₂CH₂CH₂-), 10.1 (NCH₂CH₃); HRMS-ESI m/z [M+H]⁺ calcd for C₃₅H₄₂NO₁₀: 636.2809, found: 636.2806.

3-O-(N,N-Diethylamino)butyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (24). 69% yield, yellow solid, mp. 64–65 °C. IR (film) ν_{max} : 3397, 2938, 1625, 1606, 1506, 1463 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.72 (d, J = 6.3 Hz, 1H, H-15), 7.71 (s, 1H, H-13), 7.03 (d, J = 9.0 Hz, 2H, H-16 & H-22), 6.97 (s, 1H, H-18), 6.91 (d, J = 8.1 Hz, 1H, H-21), 6.45 (s, 1H, H-8), 6.31 (s, 1H, H-6), 5.03 (d, J = 7.8 Hz, 1H, H-11), 4.15–4.10 (m, 1H, H-10), 4.02–4.98 (m, 2H, 3-OCH₂), 3.95 (s, 3H, OCH₃), 3.93 (s, 6H, 2 × OCH₃), 3.90 (s, 3H, OCH₃), 3.85–3.81 (overlapped, 1H, H-23), 3.56 (dd, J = 12.3, 3.3 Hz, 1H, H-23), 2.96 (br.s, 2H, NCH₂), 2.63 (q, J = 7.2 Hz, 4H, 2 × NCH₂CH₃), 1.80–1.59 (m, 4H, 3-OCH₂CH₂CH₂), 1.06 (t, J = 7.2 Hz, 6H, 2 × NCH₂CH₃); ¹³C NMR (75 MHz, CDCl₃): δ 174.8 (C-4), 164.5 (C-7), 161.6 (C-5), 159.4 (C-8a), 153.0 (C-16a), 150.3 (C-19), 150.0 (C-20), 145.8 (C-2), 144.2 (C-12a), 141.0 (C-3), 129.1 (C-17), 124.9 (C-14), 123.0 (C-15), 120.8 (C-22), 117.7 (C-13), 117.6 (C-16), 111.9 (C-21), 110.8 (C-18), 110.0 (C-4a), 96.4 (C-6), 92.9 (C-8), 79.3 (C-10), 76.8 (C-11), 72.5 (3-OCH₂), 62.1 (C-23), 57.0 (OCH₃), 56.6 (OCH₃), 56.4 (OCH₃), 52.7 (NCH₂), 47.1 (NCH₂CH₃), 28.7 (3-O-CH₂CH₂CH₂CH₂), 22.9 (3-O-CH₂CH₂CH₂CH₂CH₂), 11.3 (NCH₂CH₃); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₆H₄₄NO₁₀: 650.2966, found 650.2963.

3-O-(*N,N*-Diethylamino)pentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (25). 33% yield, yellow wax. IR (film) ν_{max} : 3397, 2938, 1624, 1606, 1507, 1459 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 7.74 (dd, $J = 9.3, 2.1 \text{ Hz}$, 1H, H-15), 7.74 (d, $J = 2.1 \text{ Hz}$, 1H, H-13), 7.05 (d, $J = 9.3 \text{ Hz}$, 1H, H-16), 7.04 (dd, $J = 8.4, 2.1 \text{ Hz}$, 1H, H-22), 6.98 (d, $J = 1.8 \text{ Hz}$, 1H, H-18), 6.92 (d, $J = 8.1 \text{ Hz}$, 1H, H-21), 6.46 (d, $J = 2.1 \text{ Hz}$, 1H, H-8), 6.32 (d, $J = 2.1 \text{ Hz}$, 1H, H-6), 5.07 (d, $J = 8.1 \text{ Hz}$, 1H, H-11), 4.16 (dt, $J = 7.2, 2.4 \text{ Hz}$, 1H, H-10), 4.05-3.98 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.86 (s, 3H, OCH₃), 3.85 (dd, $J = 12.6, 2.7 \text{ Hz}$, 1H, H-23), 3.58 (dd, $J = 12.6, 3.9 \text{ Hz}$, 1H, H-23), 2.75 (q, $J = 7.2 \text{ Hz}$, 4H, 2 \times NCH₂CH₃), 2.63 (t, $J = 8.4 \text{ Hz}$, 2H, NCH₂), 1.73 (quin, $J = 6.9 \text{ Hz}$, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.56-1.51 (m, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.46-1.41 (m, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.13 (t, $J = 7.2 \text{ Hz}$, 6H, 2 \times

NCH_2CH_3); ^{13}C NMR (75 MHz, CDCl_3): δ 174.1 (C-4), 163.9 (C-7), 161.0 (C-5), 158.8 (C-8a), 152.5 (C-16a), 149.7 (C-19), 149.4 (C-20), 145.3 (C-2), 143.6 (C-12a), 140.3 (C-3), 128.5 (C-17), 124.2 (C-14), 122.3 (C-15), 120.2 (C-22), 117.2 (C-13), 117.0 (C-16), 111.3 (C-21), 110.2 (C-18), 109.4 (C-4a), 95.8 (C-6), 92.3 (C-8), 78.7 (C-10), 76.1 (C-11), 71.7 (3-OCH₂), 61.5 (C-23), 56.4 (OCH₃), 56.1 (OCH₃), 56.0 (OCH₃), 55.7 (OCH₃), 51.1 (NCH₂), 46.3 (NCH₂CH₃), 29.5 3-O-CH₂CH₂CH₂CH₂N-), 23.7 3-O-CH₂CH₂CH₂CH₂N-), 9.4 (NCH₂CH₃); HRMS-ESI m/z [M+H]⁺ calcd for $\text{C}_{37}\text{H}_{46}\text{NO}_{10}$: 663.3122, found: 664.3116.

3-O-(3-(N,N-Dipropyl)propyl)-5,7,20-O-trimethyl-2,3-dehydrosilybin (26). 78% yield, yellow wax. IR (film) ν_{max} : 3338, 3052, 2958, 2934, 2873, 2837, 1624, 1603, 1578, 1517, 1505, 1456 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 7.70 (d, $J = 7.7$ Hz, 1H, H-15), 7.68 (s, 1H, H-13), 7.05 (d, $J = 7.9$ Hz, 1H, H-16), 7.03 (d, $J = 6.8$ Hz, 1H, H-22), 6.97 (s, 1H, H-18), 6.91 (d, $J = 8.2$ Hz, 1H, H-21), 6.46 (s, 1H, H-8), 6.32 (s, 1H, H-6), 5.03 (d, $J = 8.0$ Hz, 1H, H-11), 4.13 (d, $J = 7.8$ Hz, 1H, H-10), 4.08-4.00 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.90 (s, 6H, 2 × OCH₃), 3.85 (s, 3H, OCH₃), 3.85-3.82 (overlapped, 1H, H-23), 3.57 (dd, $J = 12.4$, 3.3 Hz, 1H, H-23), 2.97-2.85 (m, 2H, 3-O-CH₂CH₂CH₂N-), 2.59 (t, $J = 7.4$ Hz, 4H, 2 × NCH₂CH₂CH₃), 2.05-2.00 (m, 2H, 3-O-CH₂CH₂CH₂N-), 1.56 (sextet, $J = 6.8$ Hz, 4H, 2 × NCH₂CH₂CH₃), 0.86 (t, $J = 7.2$ Hz, 6H, 2 × NCH₂CH₂CH₃); ^{13}C NMR (75 MHz, CDCl_3): δ 174.2 (C-4), 164.1 (C-7), 161.1 (C-5), 158.9 (C-8a), 152.7 (C-16a), 149.9 (C-19), 149.6 (C-20), 145.5 (C-2), 143.8 (C-12a), 140.3 (C-3), 128.5 (C-17), 124.2 (C-14), 122.5 (C-15), 120.3 (C-22), 117.3 (C-13), 117.2 (C-16), 111.5 (C-21), 110.3 (C-18), 109.5 (C-4a), 96.0 (C-6), 92.5 (C-8), 78.9 (C-10), 76.4 (C-11), 70.3 (3-OCH₂), 61.6 (C-23), 56.5 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 55.9 (OCH₃), 55.4 (3-O-CH₂CH₂CH₂N-), 51.0 (NCH₂CH₂CH₃), 26.7 (3-O-CH₂CH₂CH₂N-), 18.7 (NCH₂CH₂CH₃), 11.8 (NCH₂CH₂CH₃); HRMS-ESI m/z [M+H]⁺ calcd for $\text{C}_{37}\text{H}_{46}\text{NO}_{10}$: 664.3122, found: 664.3118.

3-O-(N,N-dipropylamino)butyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (27). 59% yield, yellow solid, mp. 80-81 °C. IR (film) ν_{max} : 3415, 2956, 1625, 1607, 1507, 1462, 1433 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 7.75 (d, $J = 10.2$ Hz, 1H, H-15), 7.73 (s, 1H, H-13), 7.04 (d, $J = 8.4$ Hz, 1H, H-16), 7.03 (d, $J = 8.4$ Hz, 1H, H-22), 6.97 (s, 1H, H-18), 6.92 (d, $J = 8.1$ Hz, 1H, H-21), 6.46 (s, 1H, H-8), 6.32 (s, 1H, H-6), 5.04 (d, $J = 8.1$ Hz, 1H, H-11), 4.15-4.12 (m, 1H, H-10), 4.05-3.99 (m, 2H, 3-OCH₂), 3.94 (s, 3H, OCH₃), 3.91 (s, 6H, 2 × OCH₃), 3.86 (s, 3H, OCH₃), 3.88-3.82 (overlapped, 1H, H-23), 3.57 (dd, $J = 12.3$, 3.3 Hz, 1H, H-23), 2.55 (t, $J = 5.7$ Hz, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 2.44 (t, $J = 7.5$ Hz, 4H, 2 × NCH₂CH₂CH₃), 1.80-1.56 (m, 4H, 2 × NCH₂CH₂CH₃), 1.52-1.43 (m, 4H, 3-O-CH₂CH₂CH₂CH₂N-), 0.85 (t, $J = 7.2$ Hz, 6H, 2 × NCH₂CH₂CH₃); ^{13}C NMR (75 MHz, CDCl_3): δ 174.8 (C-4), 164.5 (C-7), 161.6 (C-5), 159.4 (C-8a), 152.9 (C-16a), 150.4 (C-19), 150.0 (C-20), 145.7 (C-2), 144.2 (C-12a), 141.1 (C-3), 129.0 (C-17), 125.0 (C-14), 123.1 (C-15), 120.8 (C-22), 117.7 (C-13), 117.6 (C-16), 111.9 (C-21), 110.7 (C-18), 110.1 (C-4a), 96.4 (C-6), 92.9 (C-8), 79.3 (C-10), 76.9 (C-11), 72.7 (3-OCH₂), 62.2 (C-23), 57.0 (OCH₃), 56.7 (OCH₃), 56.6 (OCH₃), 56.4 (OCH₃), 56.2 (3-O-CH₂CH₂CH₂CH₂N-), 54.1 (NCH₂CH₂CH₃), 28.7 (3-O-CH₂CH₂CH₂CH₂N-), 23.3 (3-O-CH₂CH₂CH₂CH₂N-), 20.1 (NCH₂CH₂CH₃), 12.5 (NCH₂CH₂CH₃); HRMS-ESI m/z [M+H]⁺ calcd for $\text{C}_{38}\text{H}_{48}\text{NO}_{10}$: 678.3279, found: 678.3275.

3-O-(N,N-Dipropylamino)pentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (28). 60% yield, white solid, mp. 94.0-94.5 °C. IR (film) ν_{max} : 3388, 2936, 2873, 2838, 1624, 1605, 578, 1490, 1462 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 7.73 (d, $J = 7.2$ Hz, 1H, H-15), 7.72 (s, 1H, H-13), 7.06 (d, $J = 9.3$ Hz, 2H, H-16 & H-22), 6.99 (s, 1H, H-18), 6.93 (d, $J = 8.4$ Hz, 1H, H-21), 6.47 (s, 1H, H-8), 6.33 (s, 1H, H-6), 5.07 (d, $J = 7.8$ Hz, 1H, H-11), 4.16 (d, $J = 7.8$ Hz, 1H, H-10), 4.03 (t, $J = 6.0$ Hz, 2H, 3-OCH₂), 3.94 (s, 3H, OCH₃), 3.91 (s, 6H, 2 × OCH₃), 3.87 (s, 3H, OCH₃), 3.87-3.83 (overlapped, 1H, H-23), 3.67 (dd, $J = 12.3$, 3.0 Hz, 1H, H-23), 2.68-2.51 (overlapped, 6H, 3-O-CH₂CH₂CH₂CH₂N- & 2 × NCH₂CH₂CH₃), 1.75 (quin, $J = 6.9$ Hz, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.60-1.50 (m, 6H, 3-O-CH₂CH₂CH₂CH₂N- & 2 × NCH₂CH₂CH₃), 1.43 (quin, $J = 6.9$ Hz, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 0.90 (t, $J = 7.5$ Hz, 6H, 2 × NCH₂CH₂CH₃); ^{13}C NMR (75 MHz, CDCl_3): δ 174.1 (C-4), 163.9 (C-7), 161.0 (C-5), 158.8 (C-8a), 152.3 (C-16a), 149.7 (C-19), 149.4 (C-20), 145.1 (C-2), 143.6 (C-12a), 140.4 (C-3), 128.4 (C-17), 124.4 (C-14), 122.4 (C-15), 120.1 (C-22), 117.2 (C-13), 116.9 (C-16), 111.3 (C-21), 110.2 (C-18), 109.5 (C-4a), 95.8 (C-6), 92.3 (C-8), 78.6 (C-10), 76.2 (C-11), 72.0 (3-OCH₂), 61.5 (C-23), 56.4 (OCH₃), 56.1 (OCH₃), 56.0 (OCH₃), 55.7 (OCH₃), 55.2 (3-O-CH₂CH₂CH₂CH₂N-), 53.2 (NCH₂CH₂CH₃), 29.7 (3-O-

$\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 23.8 (3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 18.8 ($\text{NCH}_2\text{CH}_2\text{CH}_3$), 18.7 (3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$). 11.7 ($\text{NCH}_2\text{CH}_2\text{CH}_3$); HRMS-ESI m/z [M+H]⁺ calcd for $\text{C}_{39}\text{H}_{50}\text{NO}_{10}$: 692.3435, found: 692.3436.

3-O-(3-(*N,N*-Dibutylamino)propyl)-5,7,20-O-trimethyl-2,3-dehydrosilybin (29). 79% yield, yellow wax. IR (film) ν_{max} : 3352, 2957, 2933, 2872, 2838, 1624, 1604, 1578, 1505, 1491, 1457 cm^{-1} ; ¹H NMR (300 MHz, CDCl_3): δ 7.67 (d, J = 7.2 Hz, 1H, H-15), 7.65 (s, 1H, H-13), 7.04 (d, J = 8.7 Hz, 1H, H-16), 6.98 (d, J = 7.2 Hz, 1H, H-22), 6.96 (s, 1H, H-18), 6.89 (d, J = 8.1 Hz, 1H, H-21), 6.45 (s, 1H, H-8), 6.31 (s, 1H, H-6), 5.02 (d, J = 8.1 Hz, 1H, H-11), 4.12 (d, J = 7.8 Hz, 1H, H-10), 4.05-3.95 (m, 2H, 3-OCH₂), 3.92 (s, 3H, OCH₃), 3.89 (s, 3H, OCH₃), 3.88 (s, 3H, OCH₃), 3.84 (s, 3H, OCH₃), 3.84-3.81 (overlapped, 1H, H-23), 3.55 (dd, J = 12.6, 3.3 Hz, 1H, H-23), 3.04 (t, J = 6.9 Hz, 2H, 3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 2.72 (t, J = 7.8 Hz, 4H, 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 2.13-2.05 (m, 2H, 3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 1.62-1.51 (m, 4H, 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.30 (quin, J = 7.2 Hz, 4H, 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 0.89 (t, J = 7.5 Hz, 6H, 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$); ¹³C NMR (75 MHz, CDCl_3): δ 174.2 (C-4), 164.2 (C-7), 161.0 (C-5), 158.9 (C-8a), 152.8 (C-16a), 149.9 (C-19), 149.5 (C-20), 145.5 (C-2), 143.9 (C-12a), 140.2 (C-3), 128.5 (C-17), 124.1 (C-14), 122.5 (C-15), 120.3 (C-22), 117.3 (C-13), 117.2 (C-16), 111.4 (C-21), 110.3 (C-18), 109.4 (C-4a), 96.0 (C-6), 92.5 (C-8), 78.9 (C-10), 76.4 (C-11), 70.1 (3-OCH₂), 61.6 (C-23), 56.6 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 55.9 (OCH₃), 53.3 (3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 51.1 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 27.1 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 26.5 (3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 20.6 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$); HRMS-ESI m/z [M+H]⁺ calcd for $\text{C}_{39}\text{H}_{50}\text{NO}_{10}$: 692.3435, found: 692.3427.

3-O-(*N,N*-dibutylamino)butyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (30). 74% yield, yellow wax. IR (film) ν_{max} : 3397, 2931, 2870, 1624, 1605, 1579, 1506, 1460 cm^{-1} ; ¹H NMR (300 MHz, CDCl_3): δ 7.75 (dd, J = 9.9, 1.8 Hz, 1H, H-15), 7.73 (s, 1H, H-13), 7.05 (d, J = 8.4 Hz, 1H, H-16), 7.03 (d, J = 8.1 Hz, 1H, H-22), 6.97 (s, 1H, H-18), 6.92 (d, J = 8.1 Hz, 1H, H-21), 6.46 (d, J = 1.8 Hz, 1H, H-8), 6.32 (d, J = 2.1 Hz, 1H, H-6), 5.05 (d, J = 8.1 Hz, 1H, H-11), 4.16-4.12 (m, 1H, H-10), 4.02 (t, J = 5.7 Hz, 2H, 3-OCH₂), 3.94 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.86 (s, 3H, OCH₃), 3.86-3.81 (overlapped, 1H, H-23), 3.58 (dd, J = 12.6, 3.9 Hz, 1H, H-23), 2.59 (t, J = 7.5 Hz, 2H, 3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 2.51 (t, J = 7.5 Hz, 4H, 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.78-1.60 (m, 4H, 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.50-1.40 (m, 4H, 3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 1.33-1.21 (m, 4H, 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 0.89 (t, J = 7.2 Hz, 6H, 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$); ¹³C NMR (75 MHz, CDCl_3): δ 174.1 (C-4), 163.9 (C-7), 161.0 (C-5), 158.8 (C-8a), 152.3 (C-16a), 149.7 (C-19), 149.4 (C-20), 145.1 (C-2), 143.6 (C-12a), 140.4 (C-3), 128.4 (C-17), 124.4 (C-14), 122.4 (C-15), 120.2 (C-22), 117.1 (C-13), 117.0 (C-16), 111.3 (C-21), 110.1 (C-18), 109.4 (C-4a), 96.8 (C-6), 92.3 (C-8), 78.6 (C-10), 76.2 (C-11), 72.0 (3-OCH₂), 61.5 (C-23), 56.4 (OCH₃), 56.0 (OCH₃), 55.9 (OCH₃), 55.7 (OCH₃), 53.3 (3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 53.2 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 28.1 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 22.5 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 20.6 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 14.0 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$); HRMS-ESI m/z [M+H]⁺ calcd for $\text{C}_{40}\text{H}_{52}\text{NO}_{10}$: 706.3592, found: 706.3591.

3-O-(*N,N*-Dibutylamino)pentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (31). 85% yield, yellow wax. IR (film) ν_{max} : 3398, 2931, 1625, 1606, 1506, 1462 cm^{-1} ; ¹H NMR (300 MHz, CDCl_3): δ 7.72 (dd, J = 8.1, 2.1 Hz, 1H, H-15), 7.71 (s, 1H, H-13), 7.04 (d, J = 8.1 Hz, 2H, H-16 & H-22), 6.98 (d, J = 1.8 Hz, 1H, H-18), 6.91 (d, J = 8.1 Hz, 1H, H-21), 6.46 (d, J = 2.1 Hz, 1H, H-8), 6.31 (d, J = 2.1 Hz, 1H, H-6), 5.06 (d, J = 8.1 Hz, 1H, H-11), 4.17-4.13 (m, 1H, H-10), 4.02 (t, J = 6.3 Hz, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.86 (s, 3H, OCH₃), 3.86-3.81 (overlapped, 1H, H-11), 3.58 (dd, J = 12.3, 3.9 Hz, 1H, H-10), 2.54 (t, J = 7.5 Hz, 6H, 3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$ & 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.74 (quin, J = 7.5 Hz, 2H, 3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 1.51-1.25 (m, 12H, 3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$ & 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 0.90 (t, J = 7.2 Hz, 6H, 2 × $\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$); ¹³C NMR (75 MHz, CDCl_3): δ 174.8 (C-4), 164.5 (C-7), 161.6 (C-5), 159.4 (C-8a), 153.0 (C-16a), 150.3 (C-19), 150.0 (C-20), 145.8 (C-2), 144.2 (C-12a), 141.1 (C-3), 129.1 (C-17), 124.9 (C-14), 123.0 (C-15), 120.8 (C-22), 117.8 (C-13), 117.6 (C-16), 111.9 (C-21), 110.8 (C-18), 110.0 (C-4a), 96.4 (C-6), 92.9 (C-8), 79.3 (C-10), 76.8 (C-11), 72.7 (3-OCH₂), 62.1 (C-23), 57.0 (OCH₃), 56.7 (OCH₃), 56.6 (OCH₃), 56.4 (OCH₃), 53.9 (3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 53.8 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 30.5 (3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 28.3 (3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 25.7 (3-O- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{N}-$), 24.5 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 21.2 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 14.6 ($\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$); HRMS-ESI m/z [M+H]⁺ calcd for $\text{C}_{41}\text{H}_{54}\text{NO}_{10}$: 720.3748, found: 720.3741.

3-O-Pyrrolidinopropyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (**32**). 84% yield, yellow oil. IR (film) ν_{max} : 3361, 2935, 2879, 2837, 1668, 1624, 1604, 1578, 1517, 1505, 1459 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.69 (s, 1H, H-13), 7.68 (dd, J = 7.8, 2.1 Hz, 1H, H-15), 7.06-7.01 (overlapped, 2H, H-16 & H-22), 6.96 (d, J = 1.5 Hz, 1H, H-18), 6.91 (d, J = 8.4 Hz, 1H, H-21), 6.45 (d, J = 2.1 Hz, 1H, H-8), 6.32 (d, J = 2.1 Hz, 1H, H-6), 5.03 (d, J = 8.4 Hz, 1H, H-11), 4.13 (dt, J = 7.8, 3.3 Hz, 1H, H-10), 4.07-3.96 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.85 (s, 3H, OCH₃), 3.85-3.81 (overlapped, 1H, H-23), 3.56 (dd, J = 12.3, 3.6 Hz, 1H, H-23), 3.05 (t, J = 7.8 Hz, 2H, 3-O-CH₂CH₂CH₂N-), 2.95-2.83 (m, 4H, 2 \times CH₂ from pyrrolidino), 2.16-2.00 (m, 2H, 3-O-CH₂CH₂CH₂N-), 1.92 – 1.83 (m, 4H, 2 \times CH₂ from pyrrolidino); ¹³C NMR (75 MHz, CDCl₃): δ 174.2 (C-4), 164.2 (C-7), 161.1 (C-5), 159.0 (C-8a), 152.8 (C-16a), 149.9 (C-19), 149.6 (C-20), 145.5 (C-2), 143.8 (C-12a), 140.2 (C-3), 128.5 (C-17), 124.2 (C-14), 122.4 (C-15), 120.4 (C-22), 117.3 (C-13), 117.3 (C-16), 111.5 (C-21), 110.4 (C-18), 109.5 (C-4a), 96.0 (C-6), 92.5 (C-8), 78.9 (C-10), 76.4 (C-11), 70.0 (3-OCH₂), 61.6 (C-23), 56.6 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 55.9 (OCH₃), 53.7 (3-O-CH₂CH₂CH₂N-), 53.3 (CH₂ from pyrrolidino), 28.2 (3-O-CH₂CH₂CH₂N-), 23.6 (CH₂ from pyrrolidino); HRMS-ESI m/z [M+H]⁺ calcd for C₃₅H₄₀NO₁₀: 634.2652, found: 634.2648.

3-O-Pyrrolidinobutyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (**33**). 69% yield, yellow solid, mp. 109-110 °C. IR (film) ν_{max} : 3398, 2936, 1625, 1606, 1507, 1458, 1433 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.70 (s, 1H, H-13), 7.69 (d, J = 5.4 Hz, 1H, H-15), 7.04 (d, J = 9.0 Hz, 1H, H-16)), 7.03 (d, J = 8.4 Hz, 1H, H-22), 6.97 (s, 1H, H-18), 6.91 (d, J = 8.1 Hz, 1H, H-21), 6.45 (s, 1H, H-8), 6.31 (s, 1H, H-6), 5.05 (d, J = 8.1 Hz, 1H, H-11), 4.15-4.12 (m, 1H, H-10), 4.00-3.90 (overlapped, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.90 (s, 6H, 2 \times OCH₃), 3.85 (s, 3H, OCH₃), 3.85-3.81 (overlapped, 1H, H-23), 3.56 (dd, J = 12.6, 3.6 Hz, 1H, H-23), 3.35-3.19 (m, 2H, 3-O-CH₂CH₂CH₂N-), 2.88-2.71 (m, 4H, 2 \times CH₂ from pyrrolidino), 1.95-1.76 (m, 8H, 3-O-CH₂CH₂CH₂N- & 2 \times CH₂ from pyrrolidino); ¹³C NMR (75 MHz, CDCl₃): δ 174.8 (C-4), 164.6 (C-7), 161.5 (C-5), 159.4 (C-8a), 153.2 (C-16a), 150.3 (C-19), 150.0 (C-20), 145.9 (C-2), 144.2 (C-12a), 140.9 (C-3), 129.1 (C-17), 124.8 (C-14), 123.0 (C-15), 120.8 (C-22), 117.74 (C-13), 117.70 (C-16), 111.9 (C-21), 110.8 (C-18), 110.0 (C-4a), 96.4 (C-6), 93.0 (C-8), 79.3 (C-10), 76.8 (C-11), 72.2 (3-OCH₂), 62.0 (C-23), 57.0 (OCH₃), 56.7 (OCH₃), 56.6 (OCH₃), 56.4 (OCH₃), 56.3 (3-O-CH₂CH₂CH₂N-), 54.2 (2 \times CH₂ from pyrrolidino), 28.4 (3-O-CH₂CH₂CH₂N-), 24.8 (3-O-CH₂CH₂CH₂N-), 24.0 (2 \times CH₂ from pyrrolidino); HRMS-ESI m/z [M+H]⁺ calcd for C₃₆H₄₂NO₁₀: 648.2809, found: 648.2806.

3-O-Pyrrolidinopentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (**34**). 77% yield, reddish wax. IR (film) ν_{max} : 3348, 2935, 2874, 2838, 2802, 1667, 1623, 1604, 1578, 1517, 1505, 1460 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.70 (s, 1H, H-13), 7.69 (dd, J = 7.3, 2.0 Hz, 1H, H-15), 7.03 (d, J = 9.3 Hz, 2H, H-16 & H-22), 6.98 (d, J = 1.3 Hz, 1H, H-18), 6.90 (d, J = 8.3 Hz, 1H, H-21), 6.45 (d, J = 2.1 Hz, 1H, H-8), 6.31 (d, J = 2.1 Hz, 1H H-6), 5.06 (d, J = 7.9 Hz, 1H, H-11), 4.16-4.13 (m, 1H, H-10), 4.03-3.96 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.89 (s, 3H, OCH₃), 3.85 (s, 3H, OCH₃), 3.85-3.81 (overlapped, 1H, H-23), 3.57 (dd, J = 12.5, 3.8 Hz, 1H, H-23), 2.74 (t, J = 6.6 Hz, 4H, 2 \times CH₂ from pyrrolidino), 2.58 (t, J = 7.9 Hz, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.90-1.81 (m, 4H, 2 \times CH₂ from pyrrolidino), 1.76-1.55 (m, 4H, 3-O-CH₂CH₂CH₂CH₂N-), 1.42 (quin, J = 7.8 Hz, 2H, 3-O-CH₂CH₂CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.1 (C-4), 163.9 (C-7), 161.0 (C-5), 158.8 (C-8a), 152.4 (C-16a), 149.7 (C-19), 149.4 (C-20), 145.2 (C-2), 143.5 (C-12a), 140.5 (C-3), 128.6 (C-17), 124.3 (C-14), 122.4 (C-15), 120.1 (C-22), 117.2 (C-13), 116.9 (C-16), 111.3 (C-21), 110.2 (C-18), 109.4 (C-4a), 95.8 (C-6), 92.3 (C-8), 78.7 (C-10), 76.1 (C-11), 72.1 (3-OCH₂), 61.4 (C-23), 56.4 (OCH₃), 56.0 (OCH₃), 55.9 (OCH₃), 55.8 (OCH₃), 55.7 (3-O-CH₂CH₂CH₂CH₂N-), 53.5 (CH₂ from pyrrolidino), 29.8 (3-O-CH₂CH₂CH₂CH₂N-), 27.1 (3-O-CH₂CH₂CH₂CH₂N-), 23.9 (CH₂ from pyrrolidino), 23.4 (3-O-CH₂CH₂CH₂CH₂N-); HRMS-ESI m/z [M+H]⁺ calcd for C₃₇H₄₄NO₁₀: 662.2965, found: 662.2970.

3-O-Piperidinopropyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (**35**). 51% yield, yellow wax. IR (film) ν_{max} : 3380, 2934, 2853, 1702, 1626, 1606, 1578, 1518, 1506, 1460 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.70 (s, 1H, H-13), 7.69 (d, J = 7.2 Hz, 1H, H-15), 7.06 (d, J = 9.0 Hz, 1H, H-16), 7.03 (d, J = 8.0 Hz, 1H, H-22), 6.97 (s, 1H, H-18), 6.92 (d, J = 8.2 Hz, 1H, H-21), 6.46 (d, J = 1.6 Hz, 1H, H-8), 6.32 (d, J = 1.7 Hz, 1H, H-6), 5.03 (d, J = 8.1 Hz, 1H, H-11), 4.14 (dd, J = 11.2, 3.2 Hz, 1H, H-10), 4.08-3.97 (m, 2H, 3-OCH₂), 3.94 (s, 3H, OCH₃), 3.92 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.86 (s, 3H, OCH₃), 3.86-3.82 (overlapped, 1H, H-23), 3.57 (dd, J =

12.5, 3.7 Hz, 1H, H-23), 2.94-2.83 (m, 2H, 3-O-CH₂CH₂CH₂N-), 2.76-2.63 (m, 4H, 2 × NCH₂ from piperidino), 2.12-2.08 (m, 2H, 3-O-CH₂CH₂CH₂N-), 1.79-1.76 (m, 4H, 2 × CH₂ from piperidino), 1.54-1.43 (m, 2H, CH₂ from piperidino); ¹³C NMR (75 MHz, CDCl₃): δ 174.1 (C-4), 164.0 (C-7), 160.9 (C-5), 158.8 (C-8a), 152.6 (C-16a), 149.8 (C-19), 149.4 (C-20), 145.3 (C-2), 143.7 (C-12a), 140.1 (C-3), 128.3 (C-17), 124.0 (C-14), 122.3 (C-15), 120.2 (C-22), 117.2 (C-13), 117.2 (C-16), 111.3 (C-21), 110.2 (C-18), 109.3 (C-4a), 95.9 (C-6), 92.4 (C-8), 78.7 (C-10), 76.3 (C-11), 70.1 (3-OCH₂), 61.5 (C-23), 56.4 (OCH₃), 56.1 (OCH₃), 56.0 (OCH₃), 55.77 (OCH₃), 55.76 (3-O-CH₂CH₂CH₂N-), 53.9 (NCH₂ from piperidino), 26.2 (3-O-CH₂CH₂CH₂N-), 24.3 (CH₂ from piperidino), 23.4 (CH₂ from piperidino); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₆H₄₂NO₁₀: 648.2809, found: 648.2807.

3-O-Piperidinobutyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (36). 78% yield, yellow solid, mp. 91-92 °C. IR (film) ν_{max} : 3392, 2937, 2837, 1625, 1606, 1506, 1458 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.70-7.65 (overlapped, 2H, H-13 & H-15), 7.02 (d, *J* = 9.0 Hz, 2H, H-16 & H-22), 6.97 (s, 1H, H-18), 6.90 (d, *J* = 8.1 Hz, 1H, H-21), 6.44 (s, 1H, H-8), 6.30 (s, 1H, H-6), 5.03 (d, *J* = 7.8 Hz, 1H, H-11), 4.15-4.10 (m, 1H, H-10), 3.97 (t, *J* = 5.7 Hz, 2H, 3-OCH₂), 3.92 (s, 3H, OCH₃), 3.90 (s, 3H OCH₃), 3.89 (s, 3H, OCH₃), 3.84 (s, 3H, OCH₃), 3.84-3.81 (overlapped, 1H, H-23), 3.56 (dd, *J* = 12.3, 3.6 Hz, 1H, H-23), 2.70-2.42 (overlapped, 6H, 3-O-CH₂CH₂CH₂N- & 2 × NCH₂ from piperidino), 1.74-1.64 (m, 8H, 3-O-CH₂CH₂CH₂N- & 2 × CH₂ from piperidino), 1.52-1.36 (m, 2H, CH₂ from piperidino); ¹³C NMR (75 MHz, CDCl₃): δ 174.8 (C-4), 164.6 (C-7), 161.5 (C-5), 159.4 (C-8a), 153.0 (C-16a), 150.3 (C-19), 150.0 (C-20), 145.9 (C-2), 144.2 (C-12a), 141.0 (C-3), 129.1 (C-17), 124.8 (C-14), 123.0 (C-15), 120.8 (C-22), 117.71 (C-13), 117.65 (C-16), 111.9 (C-21), 110.8 (C-18), 110.0 (C-4a), 96.4 (C-6), 93.0 (C-8), 79.3 (C-10), 76.8 (C-11), 72.4 (3-OCH₂), 62.0 (C-23), 59.1 (OCH₃), 57.0 (OCH₃), 56.7 (OCH₃), 56.6 (OCH₃), 56.4 (3-O-CH₂CH₂CH₂N-), 54.5 (NCH₂ from piperidino), 28.6 (3-O-CH₂CH₂CH₂N-), 25.4 (3-O-CH₂CH₂CH₂N-), 24.3 (CH₂ from piperidino), 22.9 (CH₂ from piperidino); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₇H₄₄NO₁₀: 662.2966, found: 662.2959.

3.10.18. 3-O-Piperidinopentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (37). 91% yield, white solid, mp. 89.5-90.0 °C. IR (film) ν_{max} : 3404, 2935, 2858, 1624, 1605, 1578, 1538, 1517, 1490, 1462 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.71 (s, 1H, H-13), 7.70 (d, *J* = 6.5 Hz, 1H, H-15), 7.03 (d, *J* = 8.2 Hz, 2H, H-16 & H-22), 6.98 (s, 1H, H-18), 6.92 (d, *J* = 8.1 Hz, 1H, H-21), 6.45 (s, 1H, H-8), 6.31 (s, 1H, H-6), 5.06 (d, *J* = 7.9 Hz, 1H, H-11), 4.16-4.13 (m, 1H, H-10), 4.03-3.97 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.90 (s, 6H, 2 × OCH₃), 3.85 (s, 3H, OCH₃), 3.85-3.82 (overlapped, 1H, H-23), 3.58 (dd, *J* = 11.8, 2.1 Hz, 1H, H-23), 2.59-2.45 (m, 4H, 2 × NCH₂ from piperidino), 2.41 (t, *J* = 7.1 Hz, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.76-1.62 (m, 6H, 3-O-CH₂CH₂CH₂CH₂N- & 2 × CH₂ from piperidino), 1.60-1.52 (m, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.51-1.37 (m, 4H, CH₂ from piperidino & 3-O-CH₂CH₂CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.3 (C-4), 164.0 (C-7), 161.1 (C-5), 158.9 (C-8a), 152.5 (C-16a), 149.9 (C-19), 149.6 (C-20), 145.3 (C-2), 143.7 (C-12a), 140.6 (C-3), 128.7 (C-17), 124.5 (C-14), 122.6 (C-15), 120.3 (C-22), 117.4 (C-13), 117.1 (C-16), 111.5 (C-21), 110.3 (C-18), 109.6 (C-4a), 95.9 (C-6), 92.5 (C-8), 78.8 (C-10), 76.3 (C-11), 72.3 (3-OCH₂), 61.6 (C-23), 58.9 (OCH₃), 56.5 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 55.9 (3-O-CH₂CH₂CH₂CH₂N-), 54.1 (NCH₂ from piperidino), 30.0 (3-O-CH₂CH₂CH₂CH₂N-), 25.6 (3-O-CH₂CH₂CH₂CH₂N-), 25.0 (3-O-CH₂CH₂CH₂CH₂N-), 24.2 (CH₂ from piperidino), 23.9 (CH₂ from piperidino); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₈H₄₆NO₁₀: 676.3122, found: 676.3117.

3-O-Morpholinopropyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (38). 54% yield, yellow wax. IR (film) ν_{max} : 3379, 2937, 2838, 1625, 1606, 1518, 1506, 1493, 1462 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.69 (s, 1H, H-13), 7.68 (d, *J* = 8.6 Hz, 1H, H-15), 7.07 (d, *J* = 8.9 Hz, 1H, H-16), 7.04 (d, *J* = 10.8 Hz, 1H, H-22), 6.97 (s, 1H, H-18), 6.92 (d, *J* = 8.2 Hz, 1H, H-21), 6.48 (s, 1H, H-8), 6.34 (s, 1H, H-6), 5.03 (d, *J* = 8.3 Hz, 1H, H-11), 4.16-4.14 (m, 1H, H-10), 4.02-3.90 (overlapped, 2H, 3-OCH₂), 3.94 (s, 3H, OCH₃), 3.91 (s, 6H, 2 × OCH₃), 3.87 (s, 3H, OCH₃), 3.94-3.87 (overlapped, 4H, 2 × OCH₂ from morpholino), 3.87-3.83 (overlapped, 1H, H-23), 3.58 (dd, *J* = 12.2, 2.9 Hz, 1H, H-23), 3.24-3.10 (m, 2H, 3-O-CH₂CH₂CH₂N-), 3.03-2.83 (m, 4H, 2 × NCH₂ from morpholino), 2.29-2.13 (m, 2H, 3-O-CH₂CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.2 (C-4), 164.3 (C-7), 161.1 (C-5), 159.0 (C-8a), 153.0 (C-16a), 150.0 (C-19), 149.6 (C-20), 145.6 (C-2), 143.9 (C-12a), 140.1 (C-3), 128.4 (C-17), 124.0 (C-14), 122.4 (C-15), 120.4 (C-22), 117.4 (C-13), 117.3 (C-16), 111.5 (C-21), 110.4 (C-18), 109.4 (C-4a), 96.1 (C-6), 92.6 (C-

8), 78.9 (C-10), 76.5 (C-11), 69.6 (3-OCH₂ from morpholino), 65.0 (OCH₂ from morpholino), 61.7 (C-23), 56.6 (OCH₃), 56.2 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 56.0 (3-O-CH₂CH₂CH₂N-), 52.9 (NCH₂ from morpholino), 25.6 (3-O-CH₂CH₂CH₂N-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₅H₄₀NO₁₁: 650.2601, found: 650.2602.

3-O-Morpholinobutyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (39). 90% yield, yellow wax. IR (film) ν_{max} : 3415, 2938, 1624, 1605, 1579, 1491 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.73 (d, *J* = 6.9 Hz, 1H, H-15), 7.72 (s, 1H, H-13), 7.02 (d, *J* = 9.0 Hz, 2H, H-16 & H-22), 6.96 (s, 1H, H-18), 6.91 (d, *J* = 8.4 Hz, 1H, H-21), 6.45 (d, *J* = 1.5 Hz, 1H, H-8), 6.31 (s, 1H, H-6), 5.03 (d, *J* = 8.1 Hz, 1H, H-11), 4.13-4.10 (m, 1H, H-10), 4.07-4.01 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.85 (s, 3H, OCH₃), 3.85-3.82 (overlapped, 1H, H-23), 3.68 (t, *J* = 4.5 Hz, 4H, 2 × OCH₂ from morpholino), 3.56 (dd, *J* = 12.3, 3.6 Hz, H-23), 2.42-2.35 (overlapped, 6H, 3-O-CH₂CH₂CH₂N- & 2 × NCH₂ from morpholino), 1.77-1.62 (m, 4H, 3-O-CH₂CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.8 (C-4), 164.5 (C-7), 161.6 (C-5), 159.4 (C-8a), 152.8 (C-16a), 150.4 (C-19), 150.0 (C-20), 145.8 (C-2), 144.2 (C-12a), 141.1 (C-3), 129.0 (C-17), 125.0 (C-14), 123.0 (C-15), 120.8 (C-22), 117.8 (C-13), 117.6 (C-16), 111.9 (C-21), 110.7 (C-18), 110.0 (C-4a), 96.4 (C-6), 92.9 (C-8), 79.3 (C-10), 76.9 (C-11), 72.6 (3-OCH₂), 67.4 (OCH₂ from morpholino), 62.2 (C-23), 59.3 (OCH₃), 57.0 (OCH₃), 56.7 (OCH₃), 56.6 (OCH₃), 56.4 (3-O-CH₂CH₂CH₂N-), 54.1 (NCH₂ from morpholino), 28.7 (3-O-CH₂CH₂CH₂N-), 23.3 (3-O-CH₂CH₂CH₂N-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₆H₄₂NO₁₁: 664.2758, found: 664.2757.

3-O-Morpholinopentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (40). 77% yield, light yellow wax. IR (film) ν_{max} : 3397, 2936, 1625, 1603, 1505, 1456, 1428 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.72-7.69 (overlapped, 2H, H-15 & H-13), 7.02 (dd, *J* = 8.4, 1.8 Hz, 1H, H-16), 7.01 (d, *J* = 9.3 Hz, 1H, H-22), 6.97 (s, 1H, H-18), 6.90 (d, *J* = 8.1 Hz, 1H, H-21), 6.44 (d, *J* = 2.1 Hz, 1H, H-8), 6.30 (d, *J* = 2.1 Hz, 1H, H-6), 5.03 (d, *J* = 8.1 Hz, 1H, H-11), 4.12 (dt, *J* = 8.1, 4.8 Hz, 1H, H-10), 4.01-3.98 (m, 2H, 3-OCH₂), 3.92 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.89 (s, 3H, OCH₃), 3.84 (s, 3H, OCH₃), 3.84-3.80 (overlapped, 1H, H-23), 3.72 (t, *J* = 4.5 Hz, 4H, 2 × OCH₂ from morpholino), 3.56 (dd, *J* = 12.6, 3.9 Hz, 1H, H-23), 2.47 (br,s, 4H, 2 × NCH₂ from morpholino), 2.35 (t, *J* = 7.2 Hz, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.73 (quin, *J* = 6.9 Hz, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.53-1.23 (m, 4H, 3-O-CH₂CH₂CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.1 (C-4), 163.9 (C-7), 160.9 (C-5), 158.7 (C-8a), 152.2 (C-16a), 149.7 (C-19), 149.4 (C-20), 145.1 (C-2), 143.6 (C-12a), 140.5 (C-3), 128.4 (C-17), 124.4 (C-14), 122.4 (C-15), 120.2 (C-22), 117.1 (C-13), 116.9 (C-16), 111.3 (C-21), 110.2 (C-18), 109.4 (C-4a), 95.7 (C-6), 92.3 (C-8), 78.7 (C-10), 76.2 (C-11), 72.0 (3-OCH₂), 66.5 (OCH₂ from morpholino), 61.5 (C-23), 58.9 (OCH₃), 56.3 (OCH₃), 56.0 (OCH₃), 55.9 (OCH₃), 55.7 (3-O-CH₂CH₂CH₂CH₂N-), 53.4 (NCH₂ from morpholino), 29.9 (3-O-CH₂CH₂CH₂CH₂N-), 25.7 (3-O-CH₂CH₂CH₂CH₂N-), 23.8 (3-O-CH₂CH₂CH₂CH₂N-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₇H₄₄NO₁₁: 678.2914, found: 678.2915.

3-O-(Piperazin-1-yl)propyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (41). 74% yield, white solid, mp. 158-159 °C. IR (film) ν_{max} : 3347, 3207, 2937, 2836, 1623, 1604, 1578, 1517, 1490, 1458 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.67 (s, 1H, H-13), 7.66 (d, *J* = 8.1 Hz, 1H, H-15), 7.02 (d, *J* = 8.1 Hz, 2H, H-16 & H-22), 6.97 (s, 1H, H-18), 6.90 (d, *J* = 8.1 Hz, 1H, H-21), 6.44 (s, 1H, H-8), 6.31 (s, 1H H-6), 5.73 (br.s, 1H, NH), 5.04 (d, *J* = 7.8 Hz, 1H, H-11), 4.13 (d, *J* = 7.5 Hz, 1H, H-10), 4.04 (t, *J* = 5.4 Hz, 2H, 3-OCH₂), 3.92 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.89 (s, 3H, OCH₃), 3.85 (s, 3H, OCH₃), 3.85-3.82 (overlapped, 1H, H-23), 3.55 (dd, *J* = 12.3, 3.0 Hz, 1H H-23), 2.97 (br.s, 4H, 2 × NCH₂ from piperazine), 2.49 (br.s., 6H, 3-O-CH₂CH₂CH₂N- & 2 × NCH₂ from piperazine), 1.90-1.79 (m, 2H, 3-O-CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.1 (C-4), 163.9 (C-7), 160.9 (C-5), 158.8 (C-8a), 152.5 (C-16a), 149.7 (C-19), 149.3 (C-20), 145.3 (C-2), 143.5 (C-12a), 140.3 (C-3), 128.5 (C-17), 124.1 (C-14), 122.5 (C-15), 120.2 (C-22), 117.3 (C-13), 116.9 (C-16), 111.3 (C-21), 110.2 (C-18), 109.4 (C-4a), 95.8 (C-6), 92.3 (C-8), 78.7 (C-10), 76.1 (C-11), 70.4 (3-OCH₂), 61.2 (C-23), 56.4 (OCH₃), 56.0 (OCH₃), 55.9 (OCH₃), 55.8 (OCH₃), 55.3 (3-O-CH₂CH₂CH₂N-), 51.7 (NCH₂ from piperazine), 44.2 (NCH₂ from piperazine), 27.2 (3-O-CH₂CH₂CH₂N-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₅H₄₁N₂O₁₀: 649.2761, found: 649.2764.

3-O-(Piperazin-1-yl)butyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (42). 68% yield, yellow wax. IR (film) ν_{max} : 3379, 2934, 1625, 1604, 1505, 1456 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.68 (d, *J* = 7.8 Hz, 1H, H-15), 7.66 (s, 1H, H-13), 7.03 (d, *J* = 7.8 Hz, 2H, H-16 & H-22), 6.99 (s, 1H, H-18), 6.91 (d, *J* = 8.1 Hz, 1H, H-21),

6.45 (s, 1H, H-8), 6.32 (s, 1H, H-6), 5.06 (d, J = 7.8 Hz, 1H, H-11), 4.17-4.09 (m, 1H, H-10), 4.09-3.99 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.89 (s, 3H, OCH₃), 3.86 (s, 3H, OCH₃), 3.85-3.79 (overlapped, 1H, H-23), 3.56 (d, J = 11.1 Hz, 1H, H-23), 3.11-2.99 (m, 2H, 3-O-CH₂CH₂CH₂N-), 2.61-2.43 (m, 4H, 2 × NCH₂ from piperazine), 2.39-2.26 (m, 4H, 2 × NCH₂ from piperazine), 1.74-1.47 (m, 4H, 3-O-CH₂CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.8 (C-4), 164.5 (C-7), 161.6 (C-5), 159.4 (C-8a), 153.2 (C-16a), 150.3 (C-19), 150.0 (C-20), 145.8 (C-2), 144.2 (C-12a), 141.0 (C-3), 129.1 (C-17), 124.9 (C-14), 123.3 (C-15), 120.8 (C-22), 117.9 (C-13), 117.5 (C-16), 111.9 (C-21), 110.8 (C-18), 110.0 (C-4a), 96.4 (C-6), 92.9 (C-8), 79.4 (C-10), 76.7 (C-11), 72.7 (3-OCH₂), 61.8 (C-23), 58.6 (OCH₃), 57.0 (OCH₃), 56.7 (OCH₃), 56.6 (OCH₃), 56.4 (3-O-CH₂CH₂CH₂N-), 52.0 (NCH₂ from piperazine), 44.9 (NCH₂ from piperazine), 28.4 (3-O-CH₂CH₂CH₂N-), 23.7 (3-O-CH₂CH₂CH₂N-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₆H₄₃N₂O₁₀: 663.2918, found: 663.2912.

3-O-(Piperazin-1-yl)pentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (**43**). 98% yield, light yellow wax. IR (film) ν_{max} : 3365, 3003, 2937, 2861, 2836, 1667, 1623, 1604, 1578, 1517, 1491, 1459 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.72 (d, J = 8.1 Hz, 1H, H-15), 7.67 (s, 1H, H-13), 7.04 (d, J = 8.1 Hz, 2H, H-16 & H-22), 6.98 (s, 1H, H-18), 6.91 (d, J = 8.1 Hz, 1H, H-21), 6.46 (s, 1H, H-8), 6.32 (s, 1H, H-6), 5.05 (d, J = 7.8 Hz, 1H, H-11), 4.12 (d, J = 7.2 Hz, 1H, H-10), 4.02 (t, J = 4.5 Hz, 2H, 3-OCH₂), 3.93 (s, 6H, 2 × OCH₃), 3.91 (s, 6H, 2 × OCH₃), 3.91-3.83 (overlapped, 1H, H-23), 3.56 (d, J = 12.3 Hz, 1H, H-23), 3.28-2.98 (m, 4H, 2 × NCH₂ from piperazine), 2.62-2.50 (m, 4H, 2 × NCH₂ from piperazine), 2.41-2.25 (m, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.78-1.60 (m, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.50-1.33 (m, 4H, 3-O-CH₂CH₂CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.3 (C-4), 164.1 (C-7), 161.1 (C-5), 159.0 (C-8a), 152.7 (C-16a), 149.9 (C-19), 149.6 (C-20), 145.3 (C-2), 143.8 (C-12a), 140.6 (C-3), 128.7 (C-17), 124.5 (C-14), 122.8 (C-15), 120.4 (C-22), 117.3 (C-13), 117.0 (C-16), 111.5 (C-21), 110.4 (C-18), 109.6 (C-4a), 95.9 (C-6), 92.5 (C-8), 79.0 (C-10), 76.4 (C-11), 72.2 (3-OCH₂), 61.3 (C-23), 58.5 (OCH₃), 56.5 (OCH₃), 56.2 (OCH₃), 56.1 (OCH₃), 55.9 (3-O-CH₂CH₂CH₂CH₂N-), 51.0 (NCH₂ from piperazine), 43.9 (NCH₂ from piperazine), 30.0 (3-O-CH₂CH₂CH₂CH₂N-), 26.2 (3-O-CH₂CH₂CH₂CH₂N-), 24.1 (3-O-CH₂CH₂CH₂CH₂N-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₇H₄₅N₂O₁₀: 677.3074, found: 677.3064.

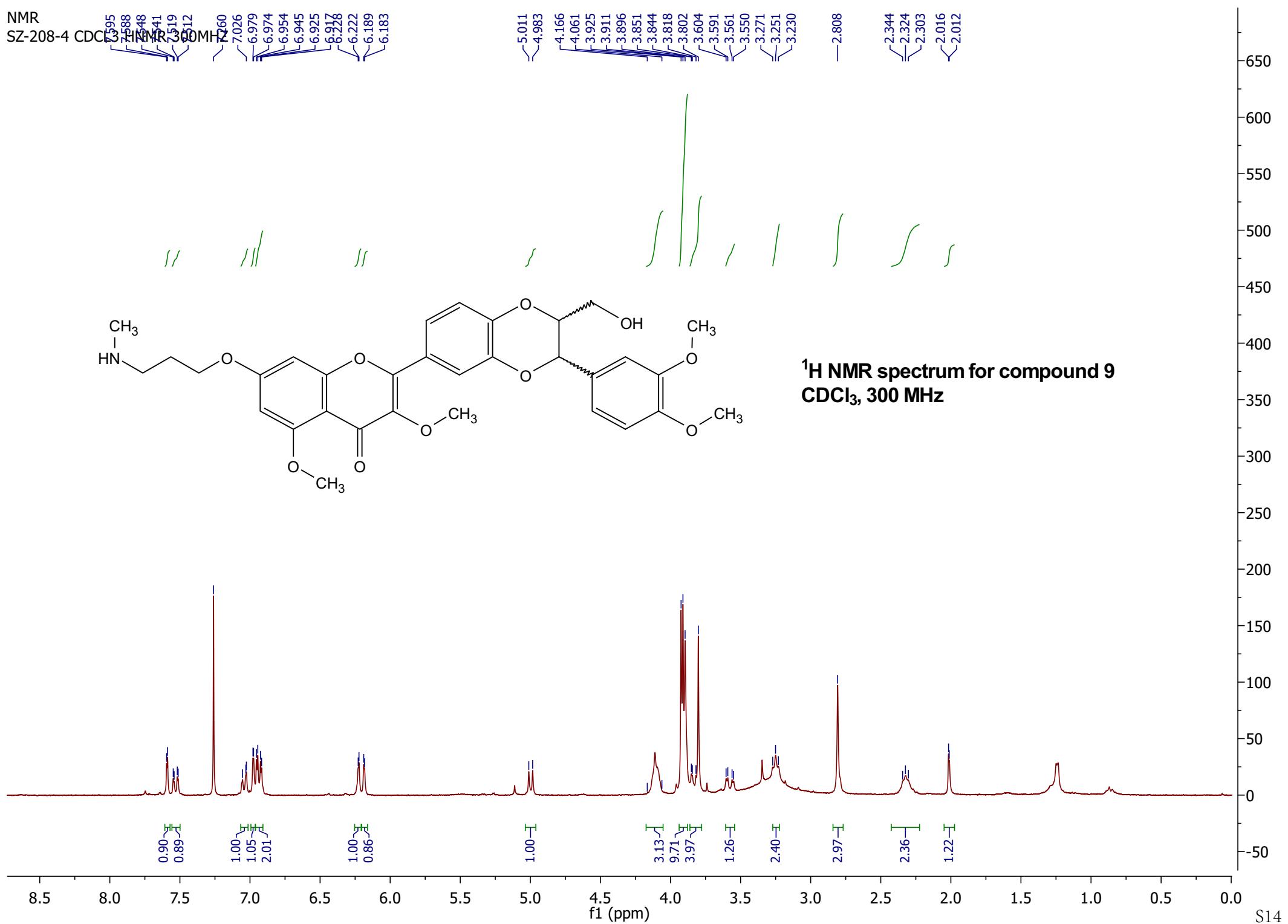
3-O-(4-Methylpiperazin-1-yl)propyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (**44**). 75% yield, yellow wax. IR (film) ν_{max} : 3398, 3002, 2937, 2879, 2836, 2806, 1625, 1606, 1578, 1517, 1506, 1491, 1460 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.70 (s, 1H, H-13), 7.68 (dd, J = 8.7, 2.1 Hz, 1H, H-15), 7.03 (d, J = 8.4 Hz, 2H, H-16 & H-22), 6.97 (d, J = 1.5 Hz, 1H, H-18), 6.91 (d, J = 8.2 Hz, 1H, H-21), 6.45 (d, J = 2.1 Hz, 1H, H-8), 6.31 (d, J = 2.1 Hz, 1H, H-6), 5.04 (d, J = 8.1 Hz, 1H, H-11), 4.17-4.12 (m, 1H, H-10), 4.10-4.04 (m, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.86 (s, 3H, OCH₃), 3.85-3.82 (overlapped, 1H, H-23), 3.58 (dd, J = 12.3, 3.9 Hz, 1H, H-23), 2.62-2.47 (overlapped, 10H, 4 × NCH₂ from 4-methylpiperazine and 3-O-CH₂CH₂CH₂N-), 2.32 (s, 3H, NCH₃), 1.93 (quin, J = 6.6 Hz, 2H, 3-O-CH₂CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.0 (C-4), 163.9 (C-7), 161.0 (C-5), 158.8 (C-8a), 152.3 (C-16a), 149.7 (C-19), 149.4 (C-20), 145.2 (C-2), 143.6 (C-12a), 140.3 (C-3), 128.5 (C-17), 124.2 (C-14), 122.4 (C-15), 120.2 (C-22), 117.3 (C-13), 117.0 (C-16), 111.3 (C-21), 110.2 (C-18), 109.4 (C-4a), 95.8 (C-6), 92.3 (C-8), 78.7 (C-10), 76.2 (C-11), 70.4 (3-OCH₂), 61.4 (C-23), 56.4 (OCH₃), 56.1 (OCH₃), 56.0 (OCH₃), 55.7 (OCH₃), 55.2 (3-O-CH₂CH₂CH₂N-), 54.3 (NCH₂ from 4-methylpiperazine), 52.3 (NCH₂ from 4-methylpiperazine), 45.4 (NCH₃), 27.2 (3-O-CH₂CH₂CH₂N-); HRMS-ESI *m/z* [M+H]⁺ calcd for C₃₆H₄₃N₂O₁₀: 663.2918, found: 663.2914.

3-O-(4-Methylpiperazin-1-yl)pentyl-5,7,20-O-trimethyl-2,3-dehydrosilybin (**45**). 93% yield, light yellow wax. IR (film) ν_{max} : 3384, 2941, 2838, 1624, 1606, 1579, 1517, 1506, 1491, 1463 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.72 (dd, J = 7.7, 2.1 Hz, 1H, H-15), 7.70 (s, 1H, H-13), 7.05-7.01 (overlapped, 2H, H-16 & H-22), 6.98 (d, J = 1.8 Hz, 1H, H-18), 6.91 (d, J = 8.3 Hz, 1H, H-21), 6.46 (d, J = 2.2 Hz, 1H, H-8), 6.31 (d, J = 2.2 Hz, 1H, H-6), 5.06 (d, J = 8.1 Hz, 1H, H-11), 4.13 (dt, J = 7.9, 3.1 Hz, 1H, H-10), 4.02 (t, J = 6.4 Hz, 2H, 3-OCH₂), 3.93 (s, 3H, OCH₃), 3.91 (s, 3H, OCH₃), 3.90 (s, 3H, OCH₃), 3.86 (s, 3H, OCH₃), 3.85 (dd, J = 12.5, 2.6 Hz, 1H, H-23), 3.56 (dd, J = 12.5, 3.8 Hz, 1H, H-23), 2.89-2.70 (overlapped, 4H, 2 × NCH₂ from 4-methylpiperazine), 2.63-2.45 (m, 6H, 2 × NCH₂ from 4-methylpiperazine and 3-O-CH₂CH₂CH₂CH₂N-), 2.32 (s, 3H, NCH₃), 1.71 (quin, J = 6.3 Hz, 2H, 3-O-CH₂CH₂CH₂CH₂N-), 1.51-1.35 (m, 4H, 3-O-CH₂CH₂CH₂CH₂N-); ¹³C NMR (75 MHz, CDCl₃): δ 174.1 (C-4), 163.9 (C-7), 161.0 (C-5), 158.8 (C-8a), 152.3 (C-16a), 149.7 (C-19), 149.4 (C-20), 145.1 (C-2),

143.6 (C-12a), 140.5 (C-3), 128.5 (C-17), 124.4 (C-14), 122.5 (C-15), 120.2 (C-22), 117.2 (C-13), 116.9 (C-16), 111.3 (C-21), 110.2 (C-18), 109.5 (C-4a), 95.8 (C-6), 92.3 (C-8), 78.7 (C-10), 76.1 (C-11), 72.2 (3-OCH₂), 61.4 (C-23), 58.4 (OCH₃), 56.4 (OCH₃), 56.1 (OCH₃), 56.0 (OCH₃), 55.7 (3-O-CH₂CH₂CH₂CH₂N-), 54.5 (NCH₂ from 4-methylpiperazine), 52.5 (NCH₂ from 4-methylpiperazine), 45.6 (NCH₃), 29.9 (3-O-CH₂CH₂CH₂CH₂N-), 26.1 (3-O-CH₂CH₂CH₂CH₂CH₂N-), 23.9 (3-O-CH₂CH₂CH₂CH₂N-); HRMS-ESI m/z [M+H]⁺ calcd for C₃₈H₄₇N₂O₁₀: 691.3231, found 691.3233.

NMR

SZ-208-4 CDCl₃ ¹³C NMR, 300MHz



NMR

SZ-208-4 CDCl₃ CNMR 75MHz

—184.4
—182.9
—160.4
—158.1
—152.6
—150.0
—149.6
—145.6
—143.7
—140.9

—128.3
—123.5
—122.0
—120.3
—117.2
—117.1
—111.5
—110.4
—108.7

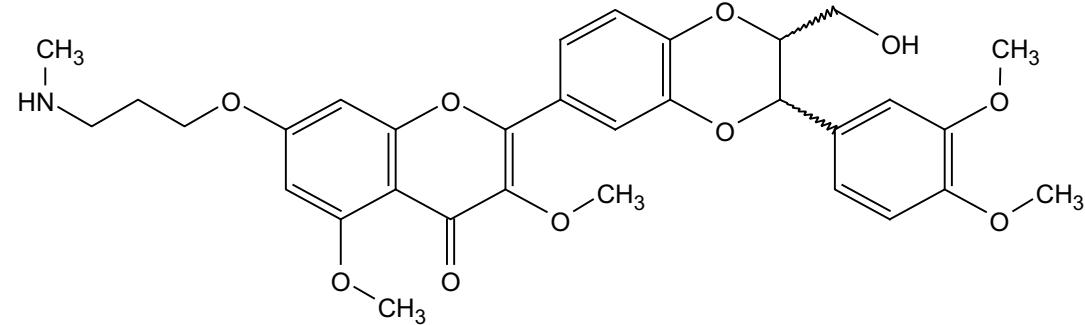
—96.3
—92.9

78.9
77.6
77.2
76.7
76.3

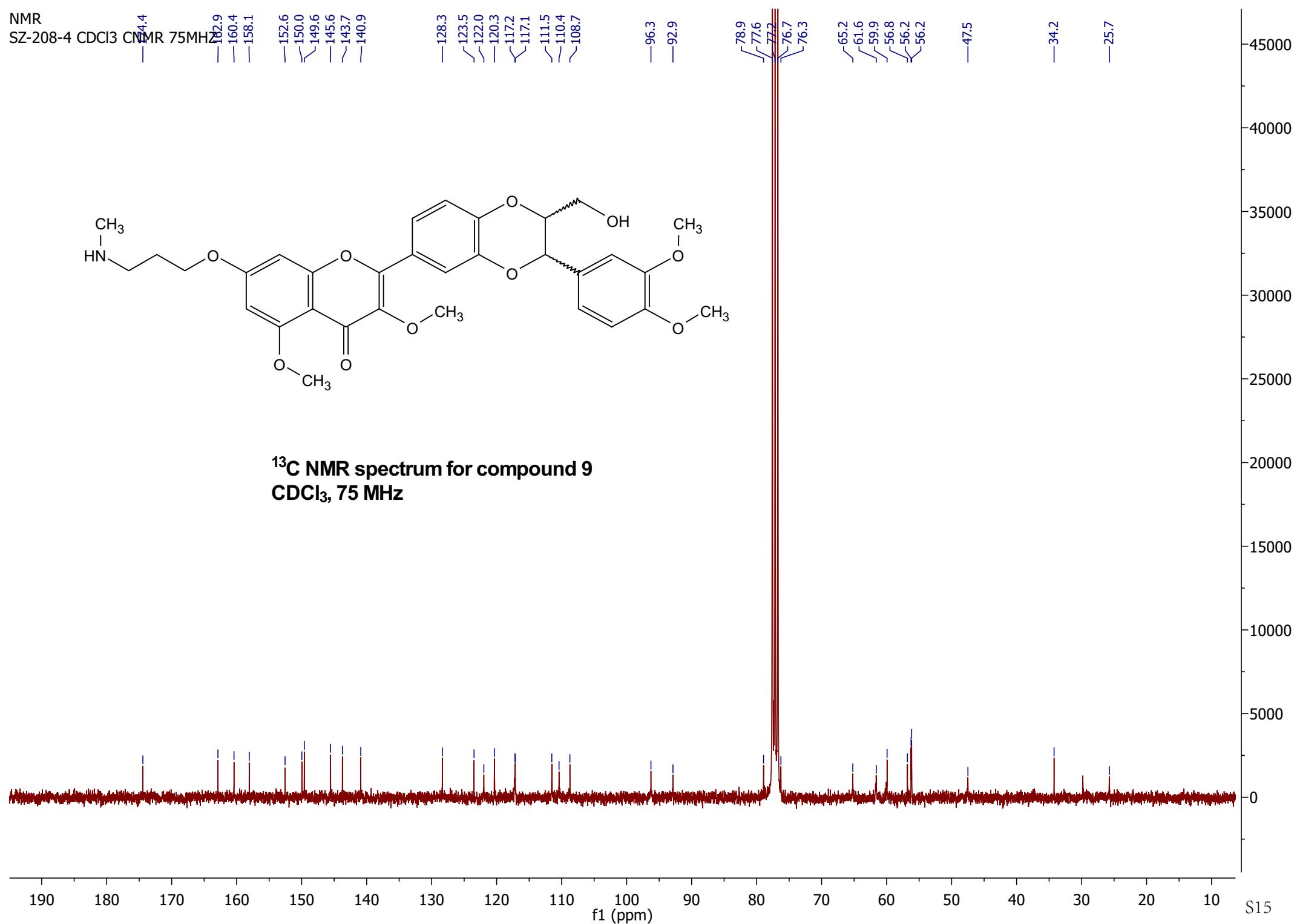
65.2
61.6
59.9
56.8
56.2
56.1

—47.5

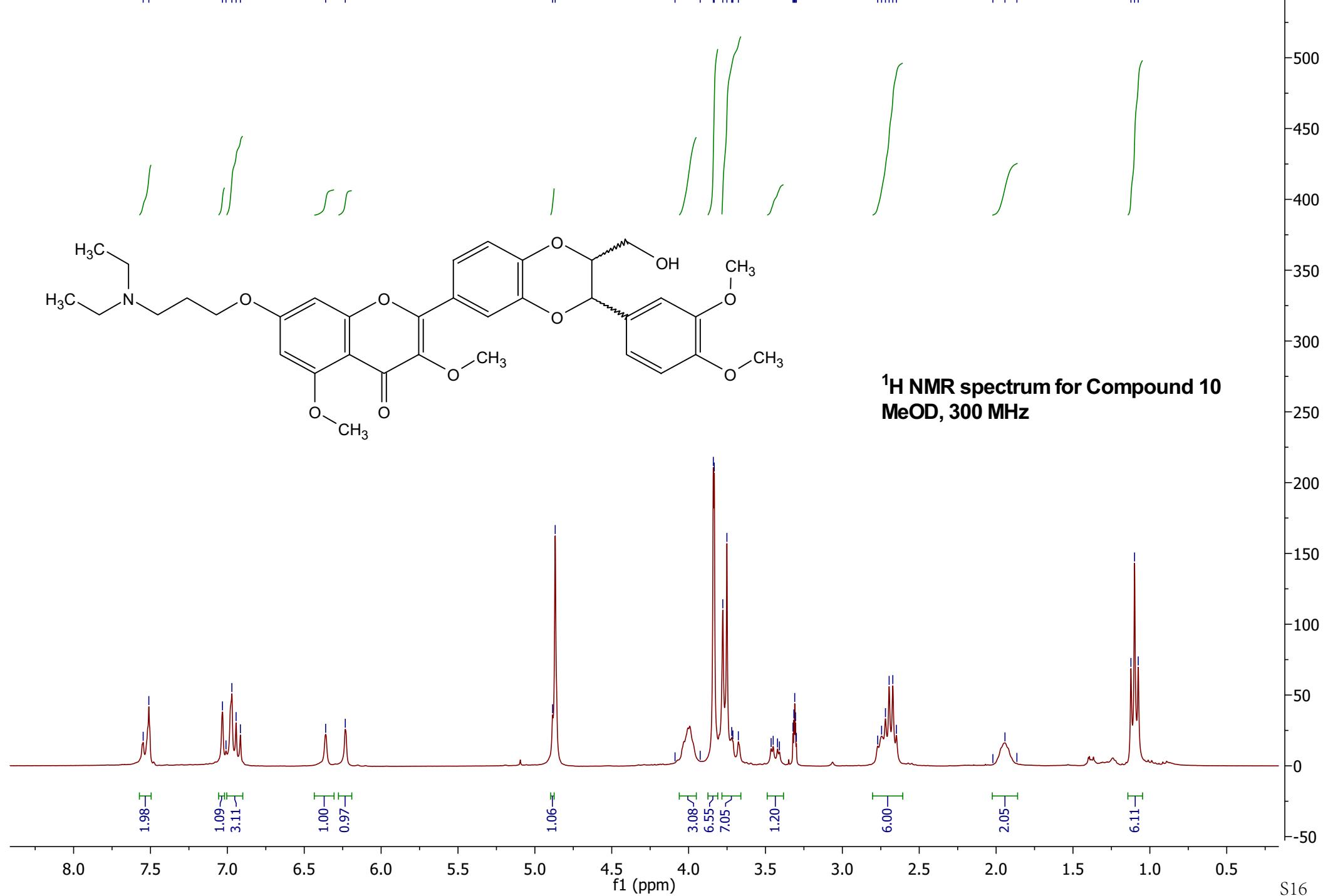
—34.2
—25.7



¹³C NMR spectrum for compound 9
CDCl₃, 75 MHz



NMR

SZ-19-138 MeOD
1H NMR 300 MHz

**¹H NMR spectrum for Compound 10
MeOD, 300 MHz**

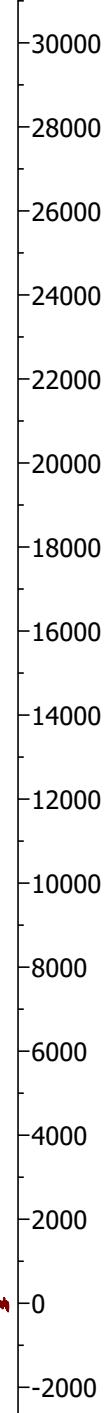
NMR

SZ-19-138 MeOD 75 MHz

¹³C NMR 75 MHzC5.5
-155.0
-151.7
-159.7
-153.9
-151.1
-150.6
-147.2
-145.0
-141.9-130.3
-124.4
-123.0
-121.6
-118.1
-117.9
-112.8
-112.3
-109.6-97.0
-94.1-80.3
-77.5-67.9
-61.9
-60.2
-56.6
-56.5
-56.4
-50.3
-47.9

-26.5

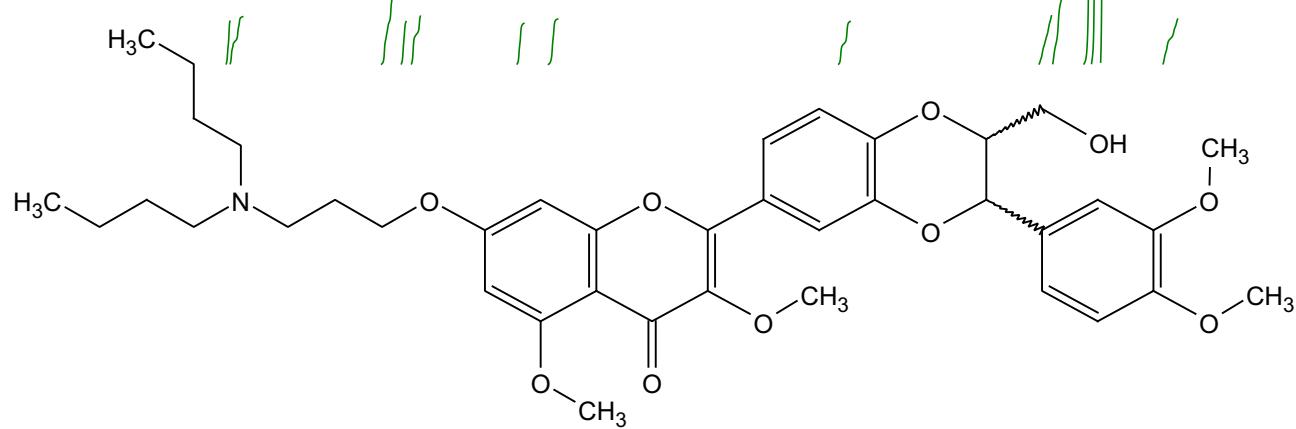
-11.0



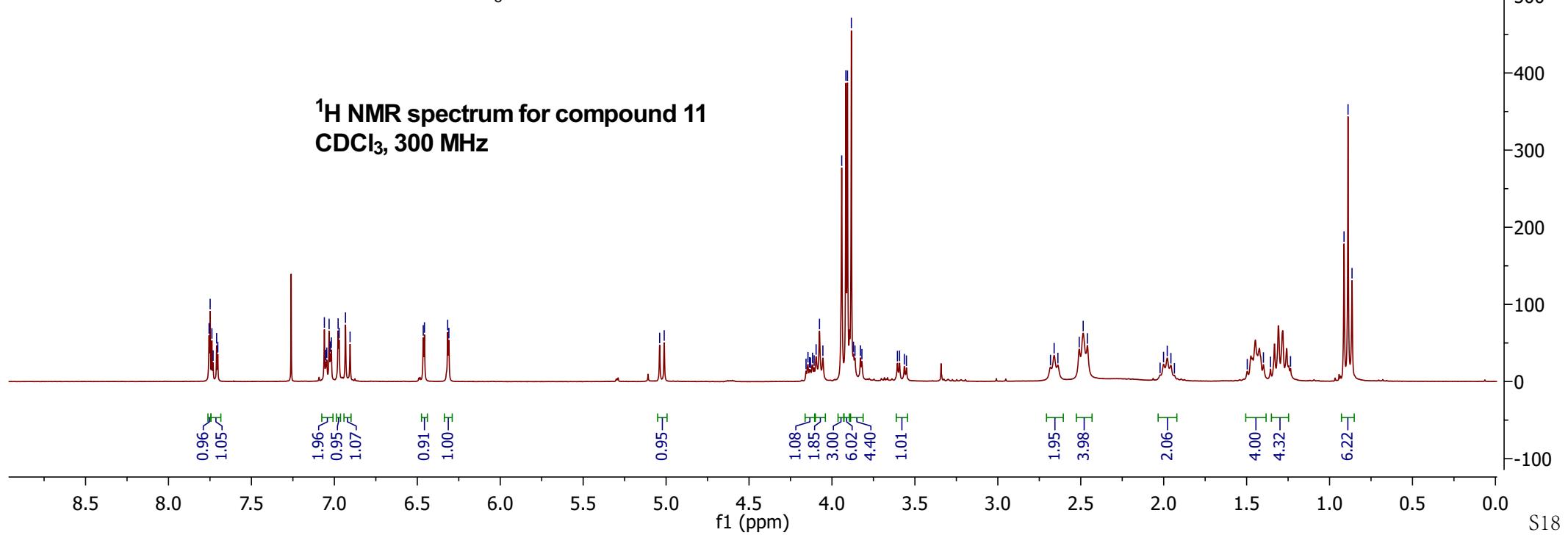
¹³C NMR spectrum for compound 10
MeOD, 75 MHz

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 S17

NMR

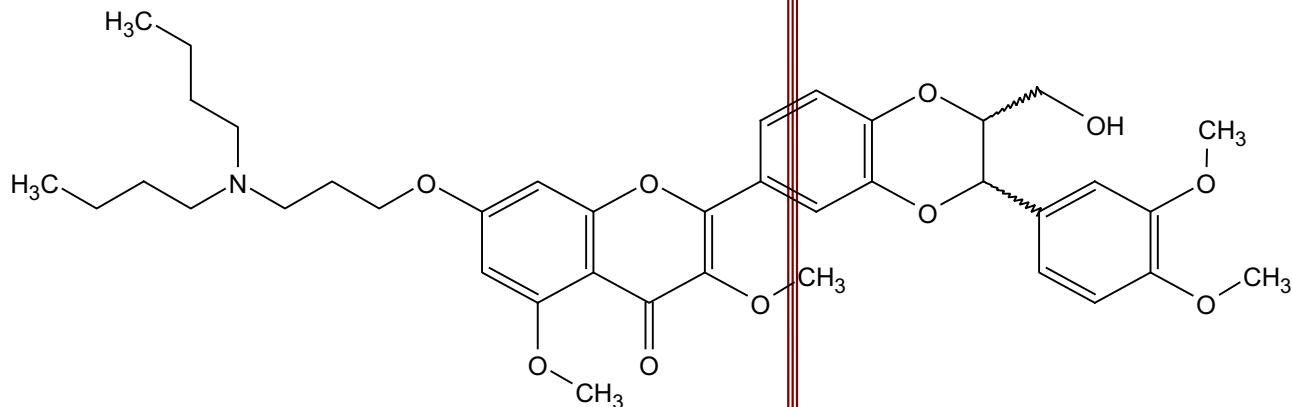
SZ-19-165 CDCl₃ 300 MHz

¹H NMR spectrum for compound 11
CDCl₃, 300 MHz

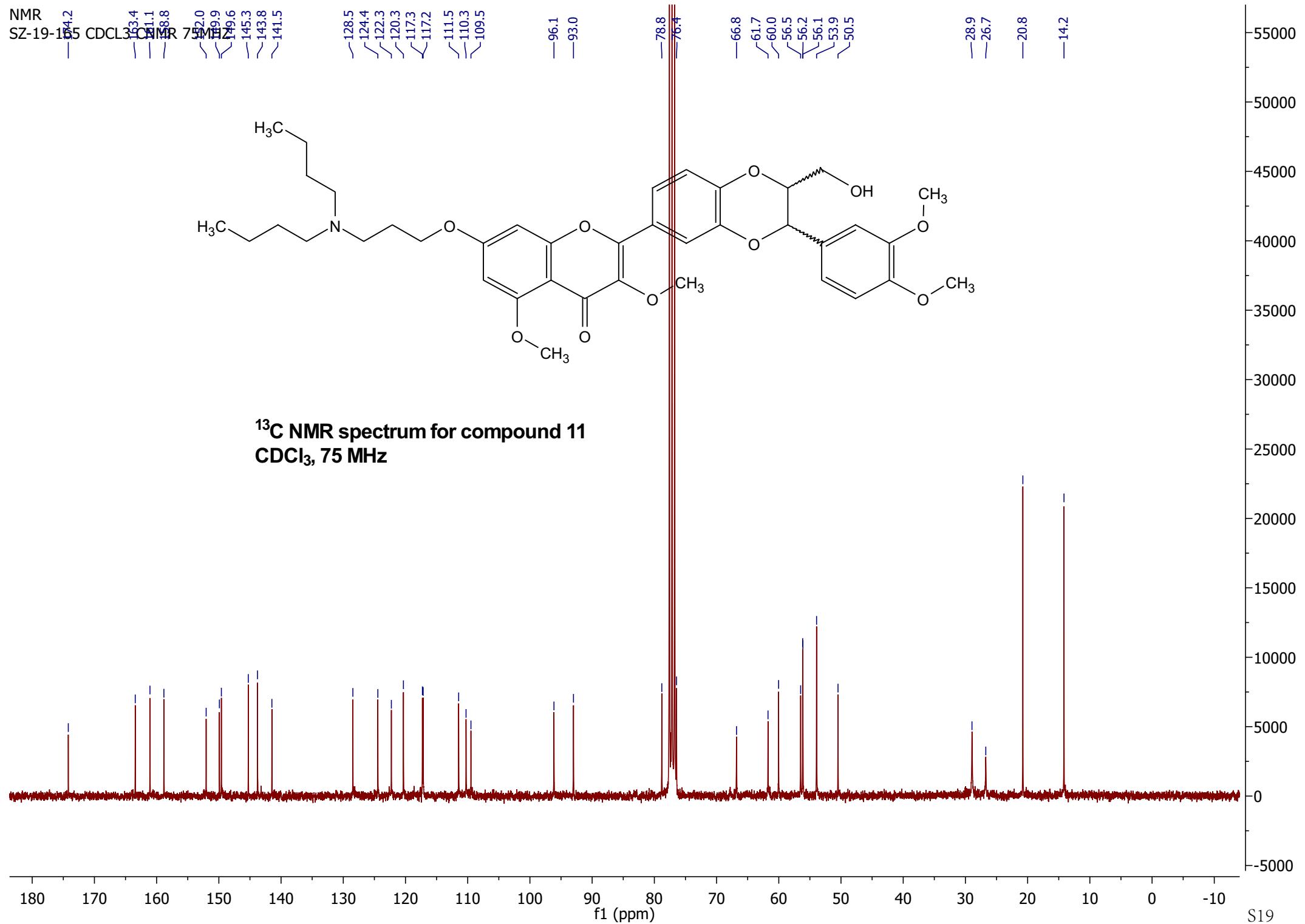


NMR
SZ-19

SZ-19-105 CDCL5 FAMIK / 3M12 1 1 1



**¹³C NMR spectrum for compound 11
CDCl₃, 75 MHz**



NMR

SZ-19-177-2 CDCl₃, 300 MHz

7.46
7.27
7.20
7.198
7.19
7.691
3.00
2.60

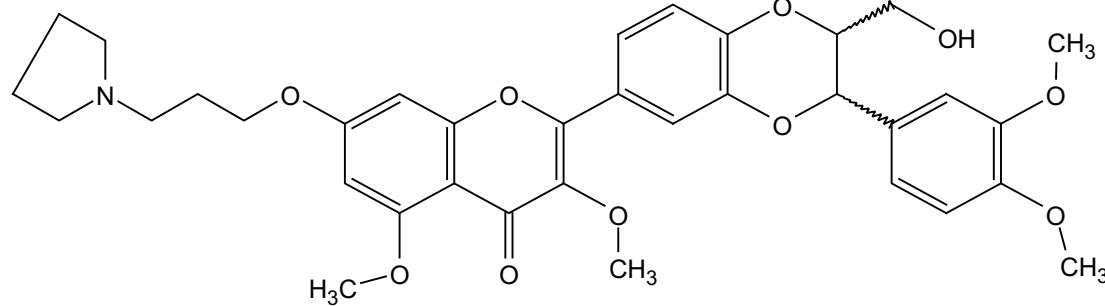
7.050
7.021
6.974
6.969
6.928
6.462
6.316
6.309

5.035
5.008

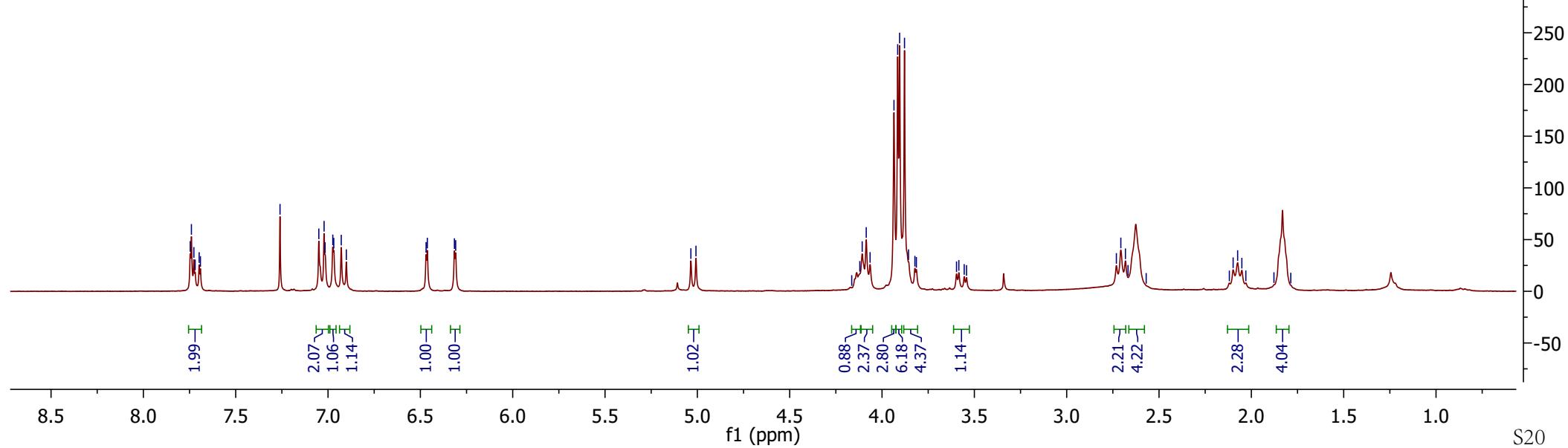
4.120
4.107
4.086
4.065
3.935
3.915
3.905
3.878
3.857
3.822
3.814
3.584
3.555
3.542

2.731
2.707
2.682
2.668
2.569

2.119
2.099
2.074
2.051
2.030
1.878
1.787



¹H NMR spectrum for compound 12
CDCl₃, 300 MHz



NMR

SZ-177-2 CDCl₃ CN₃R 75MHz

—134.2

—183.3

—161.0

—158.8

—152.1

—149.9

—149.6

—145.3

—143.8

—141.4

—128.5
—124.3
—122.2
—120.3
—117.3
—117.2
—111.4
—110.2
—109.5

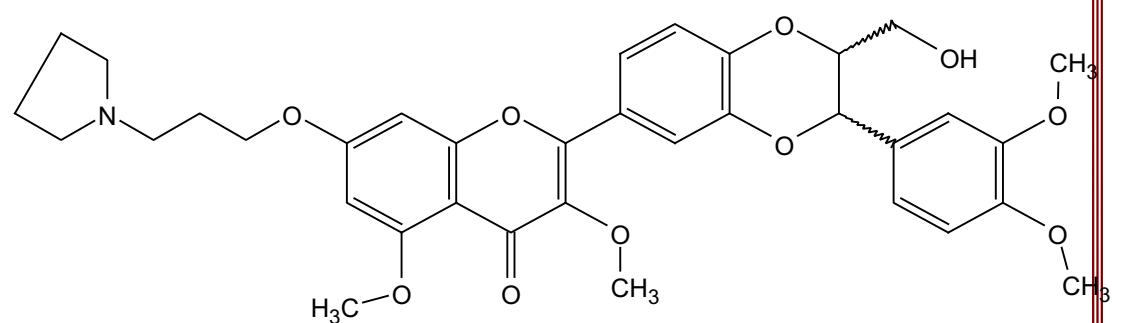
—96.1

—93.0

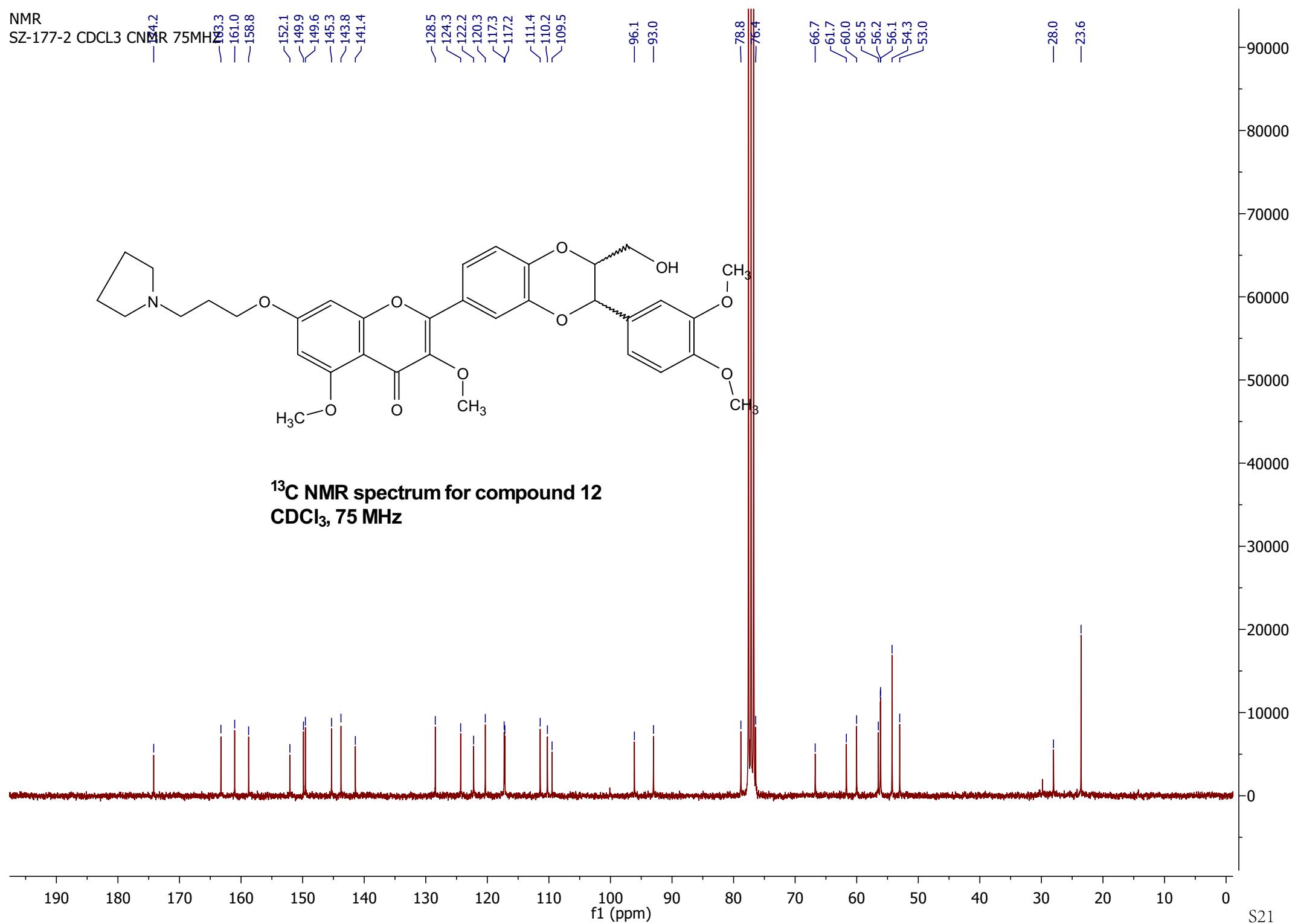
—78.8
—76.4—66.7
—61.7
—60.0
—56.5
—56.2
—56.1
—54.3
—53.0

—28.0

—23.6



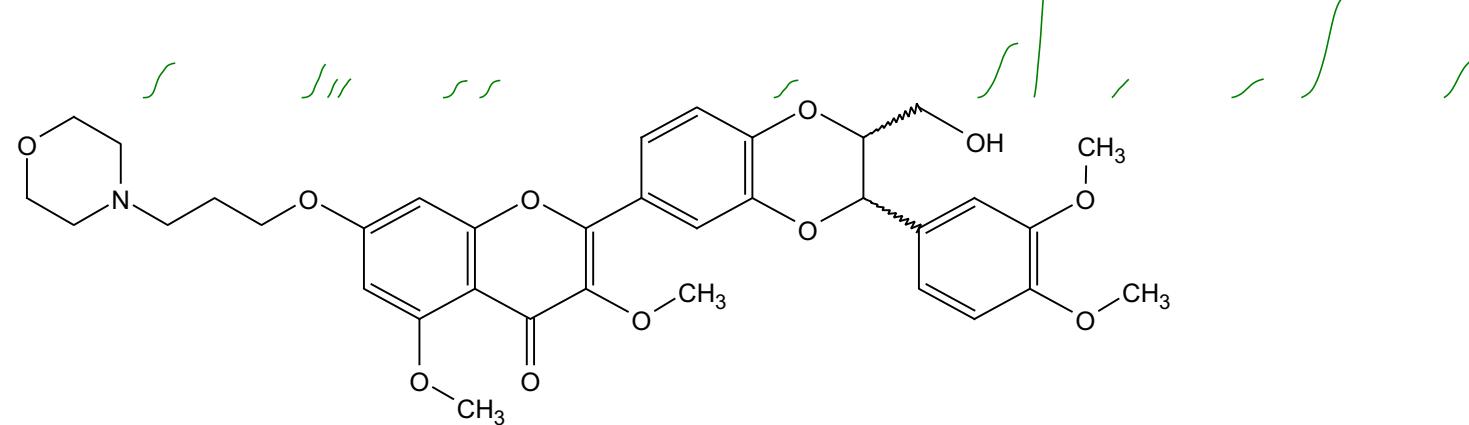
¹³C NMR spectrum for compound 12
CDCl₃, 75 MHz



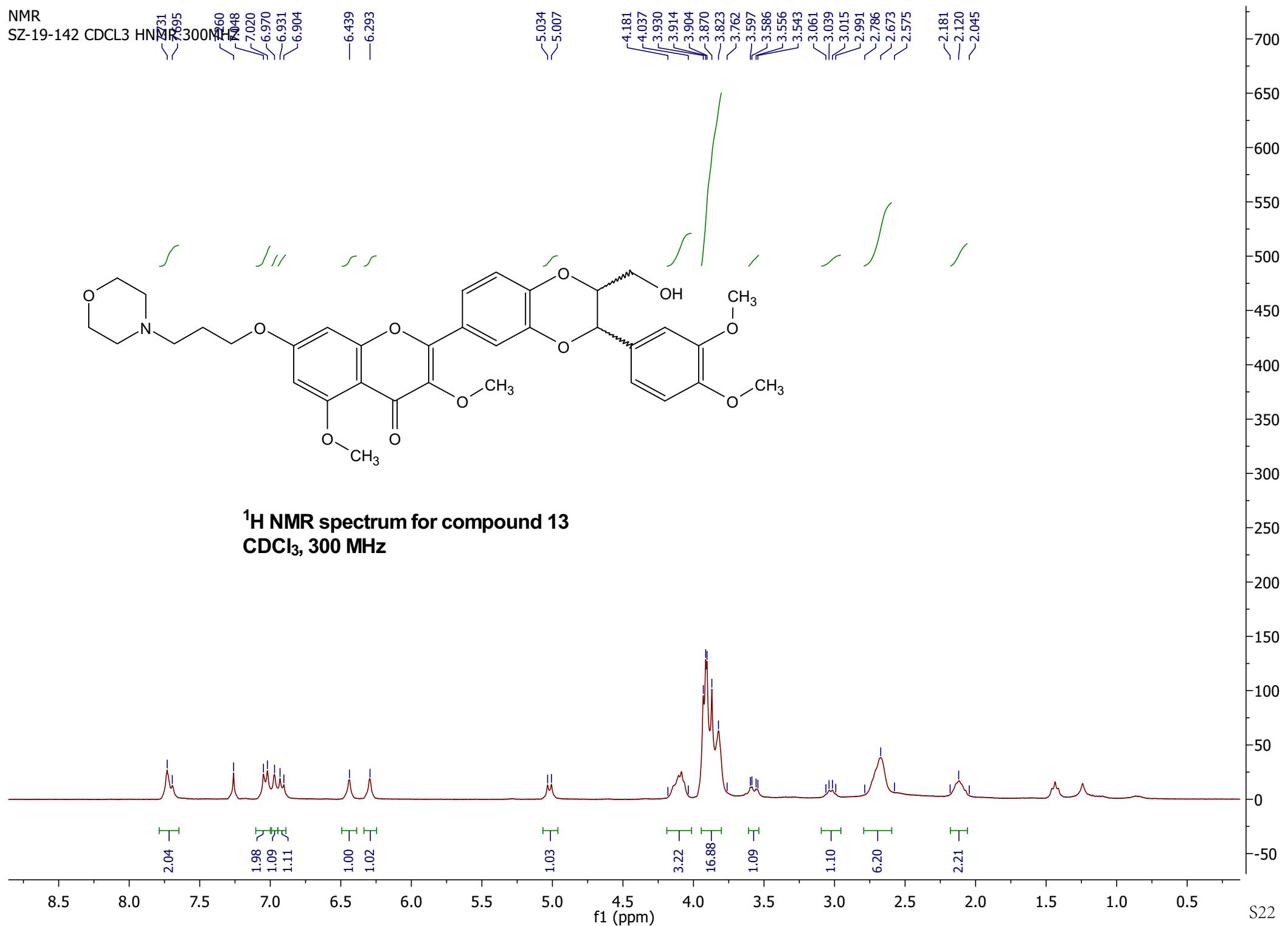
NMR

SZ-19-142 CDCl₃ HN_{300MHz}

7.31
 7.095
 7.260
 7.048
 7.020
 6.970
 6.931
 6.904
 —6.439
 —6.293



¹H NMR spectrum for compound 13
CDCl₃, 300 MHz



NMR

SZ-19-142 CDCl₃ CN¹³IR 75MHz

—144.2

—163.2

—161.0

—158.8

—152.1

—149.9

—149.6

—145.3

—143.8

—141.4

—128.4

—124.3

—122.2

—120.3

—117.2

—117.2

—117.2

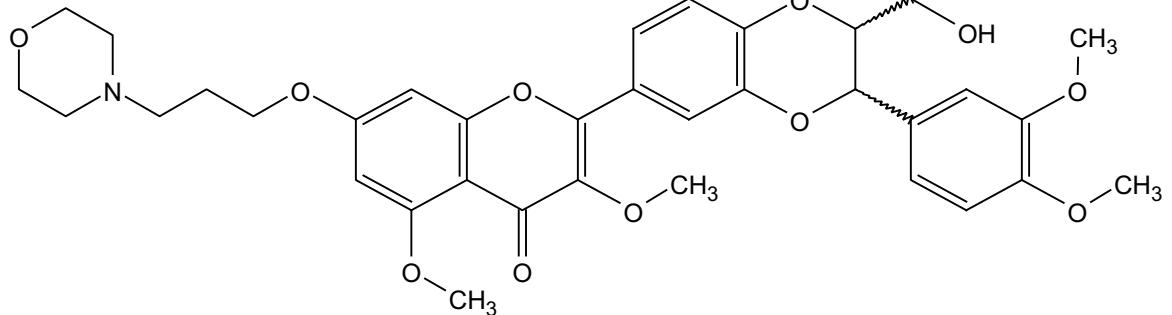
—96.1

—92.9

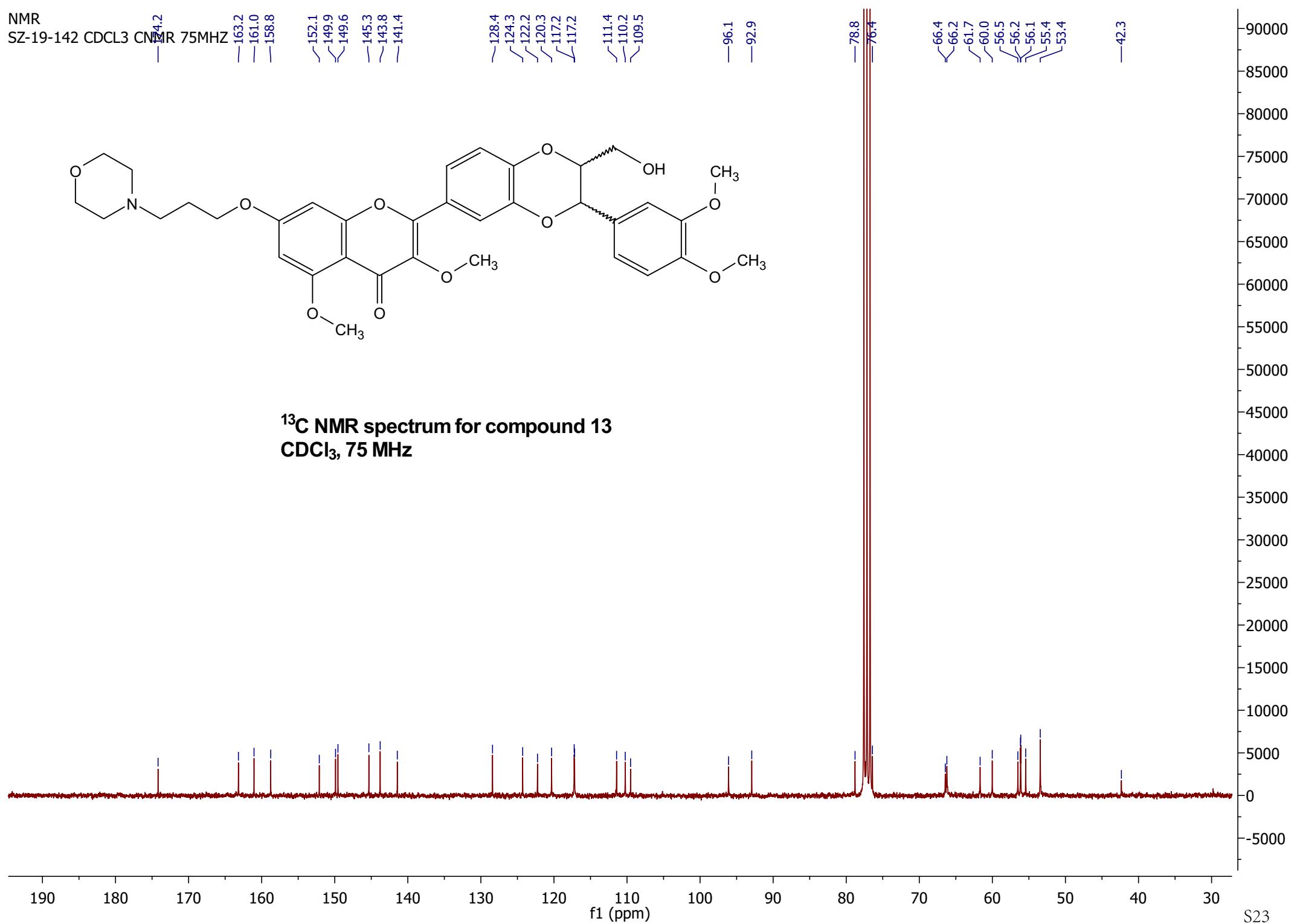
—78.8

—76.4

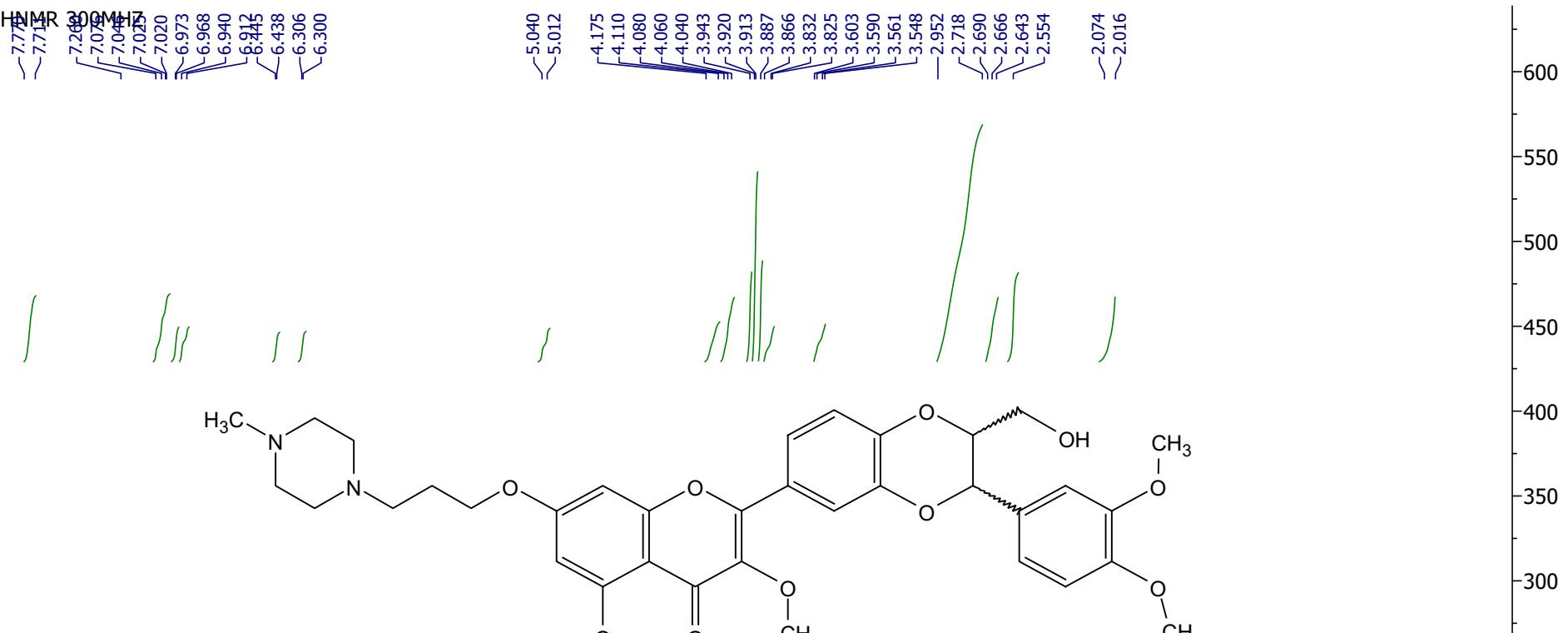
—42.3



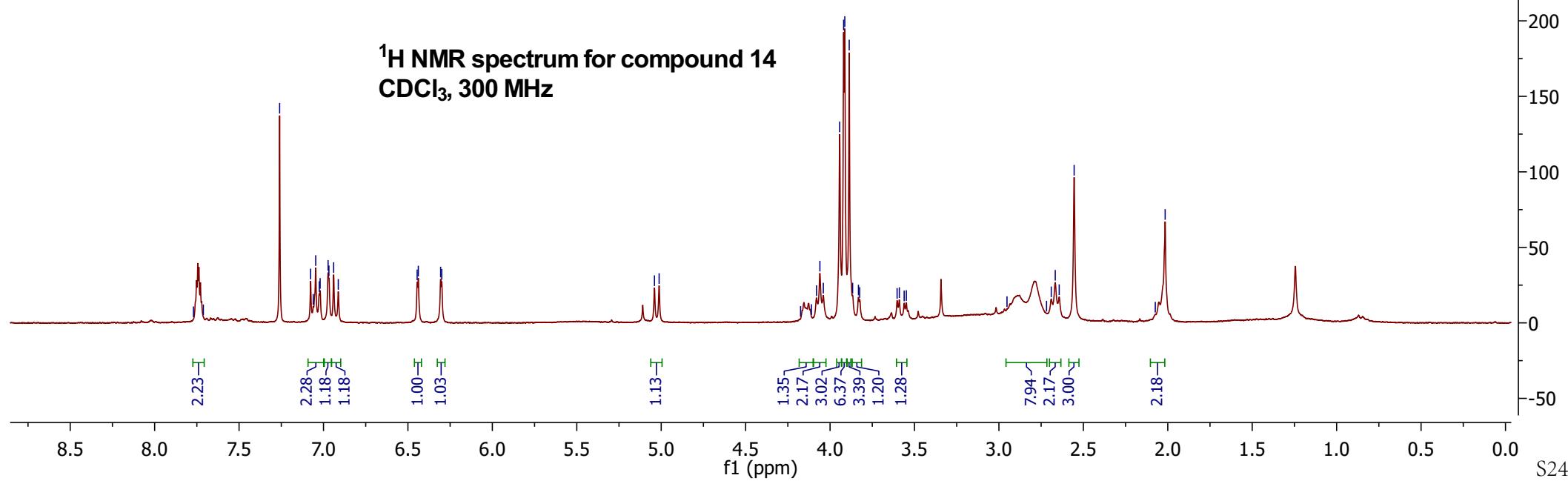
¹³C NMR spectrum for compound 13
CDCl₃, 75 MHz



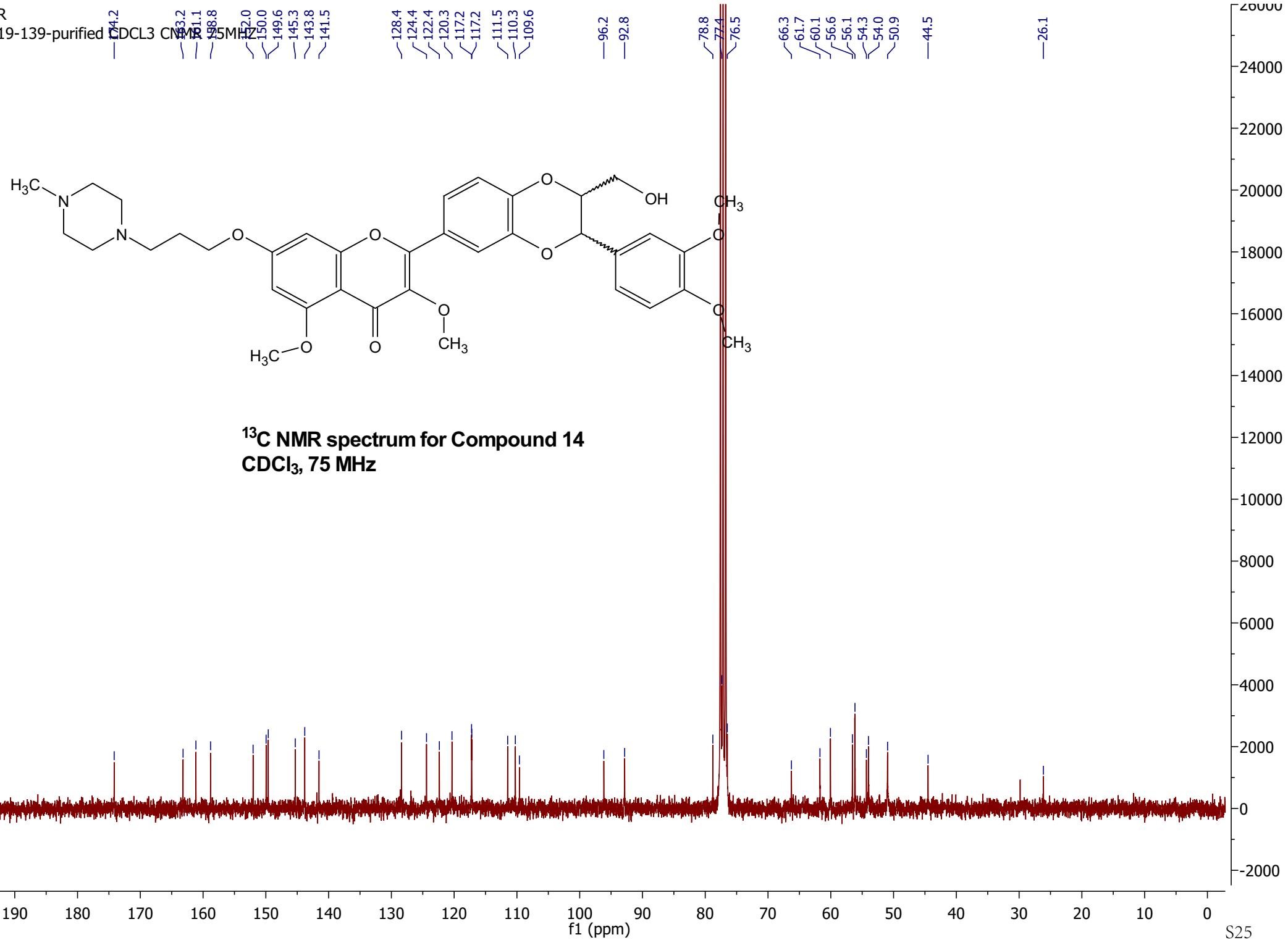
SZ-19-139-purified HNMR 300MHz

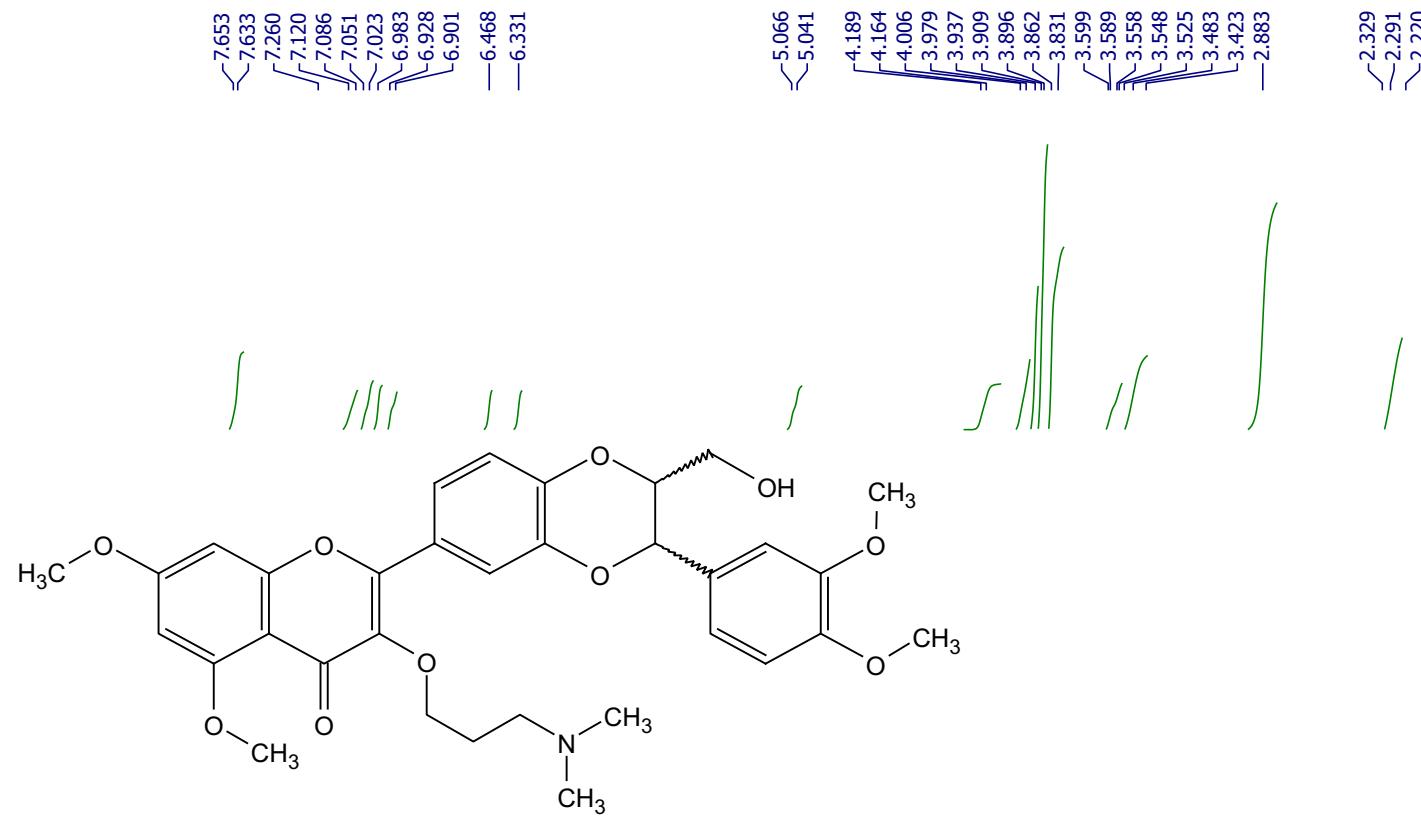


^1H NMR spectrum for compound 14
 CDCl_3 , 300 MHz

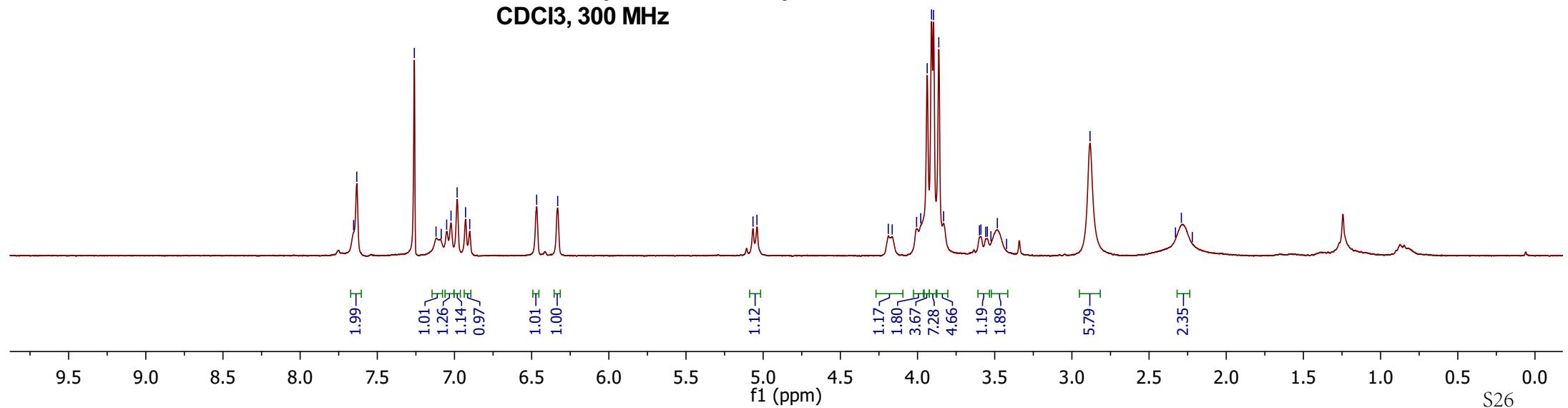


NMR

SZ-19-139-purified CDCl_3 CNM¹³C 75 MHz



1H NMR spectrum for compound 20
CDCl₃, 300 MHz



linker nmr
bv-17-23-r2- cdcl₃

—174.54

—164.60
—160.94
—159.06
—153.57
—149.93
—149.55
—145.99
—144.01
—139.73

—128.36
—123.46
—122.33
—120.38
—117.66
—117.16
—111.51
—110.42
—109.04

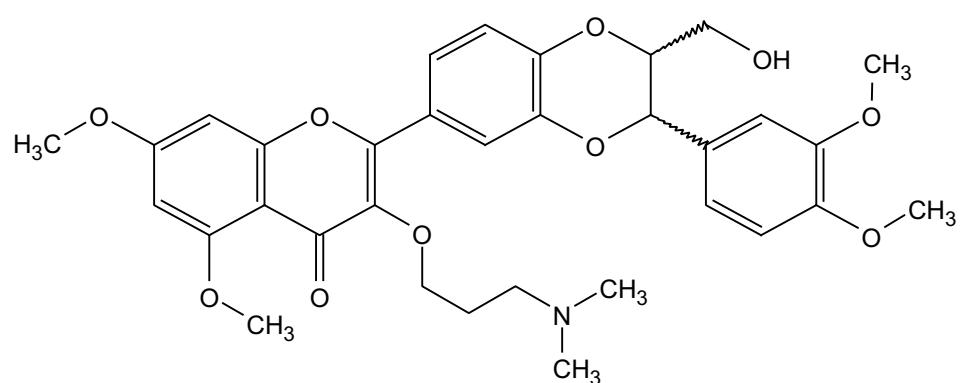
—96.33
—92.68

—78.89
—77.62
—77.20
—76.78
—76.47
—69.43

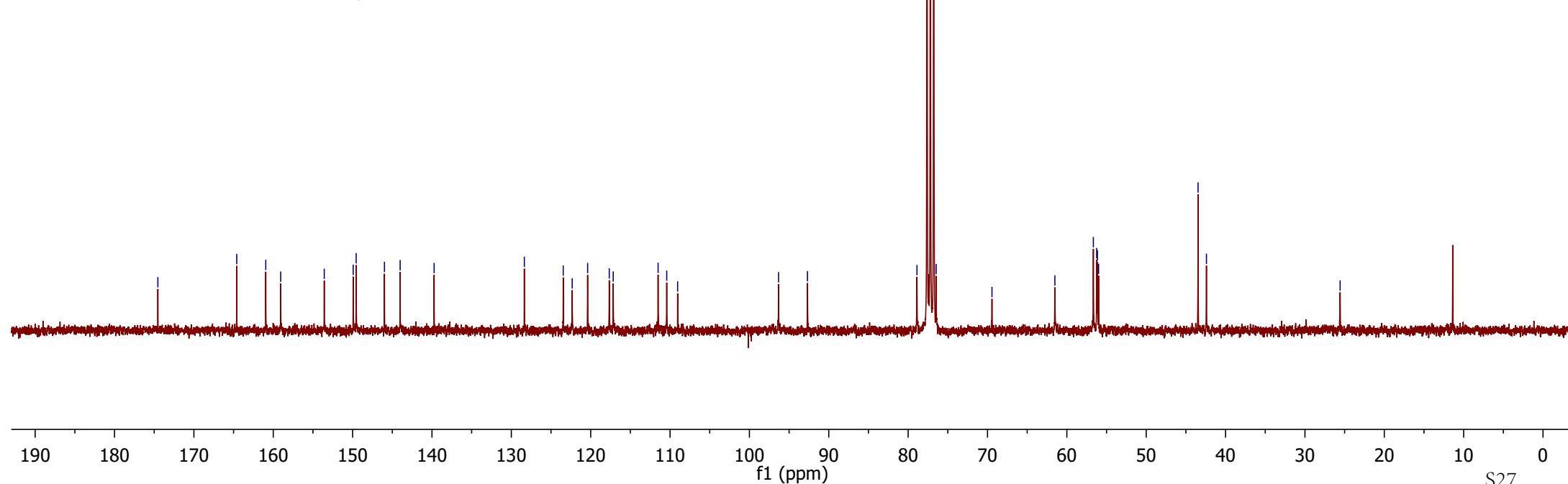
—61.51
—56.66
—56.25
—56.14
—56.00

—43.47
—42.41

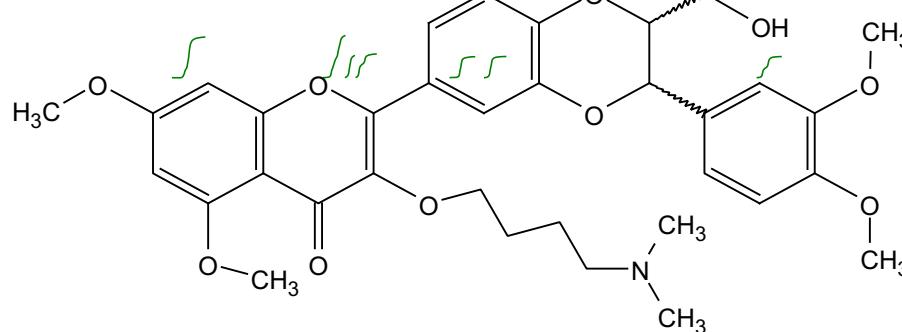
—25.57



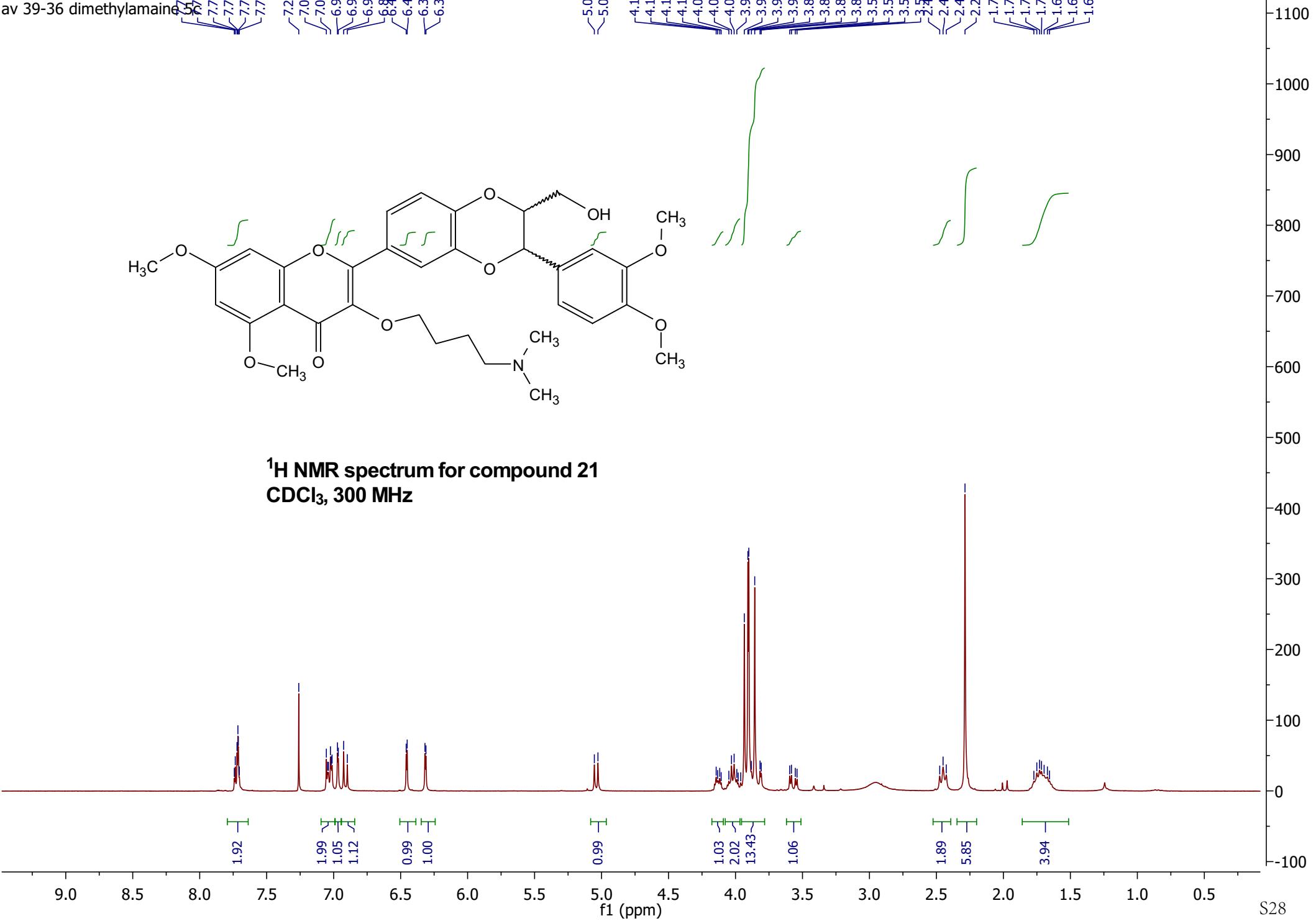
¹³C NMR spectrum for compound 20
CDCl₃, 75 MHz



andre vignau
av 39-36 dimethylamine



¹H NMR spectrum for compound 21
CDCl₃, 300 MHz



andre vignau
av 39-36 dimethylamine

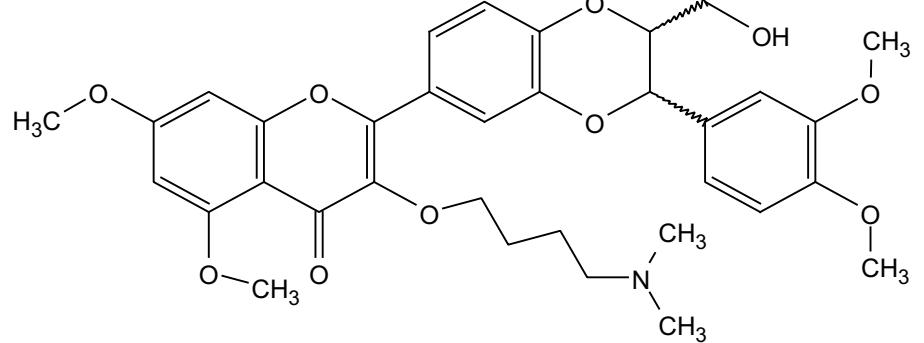
—144.11
—163.89
—160.96
—158.77
—152.32
—149.72
—149.40
—145.16
—143.60
—140.40

—128.45
—124.31
—122.36
—120.18
—117.15
—116.98
—111.30
—110.16
—109.41

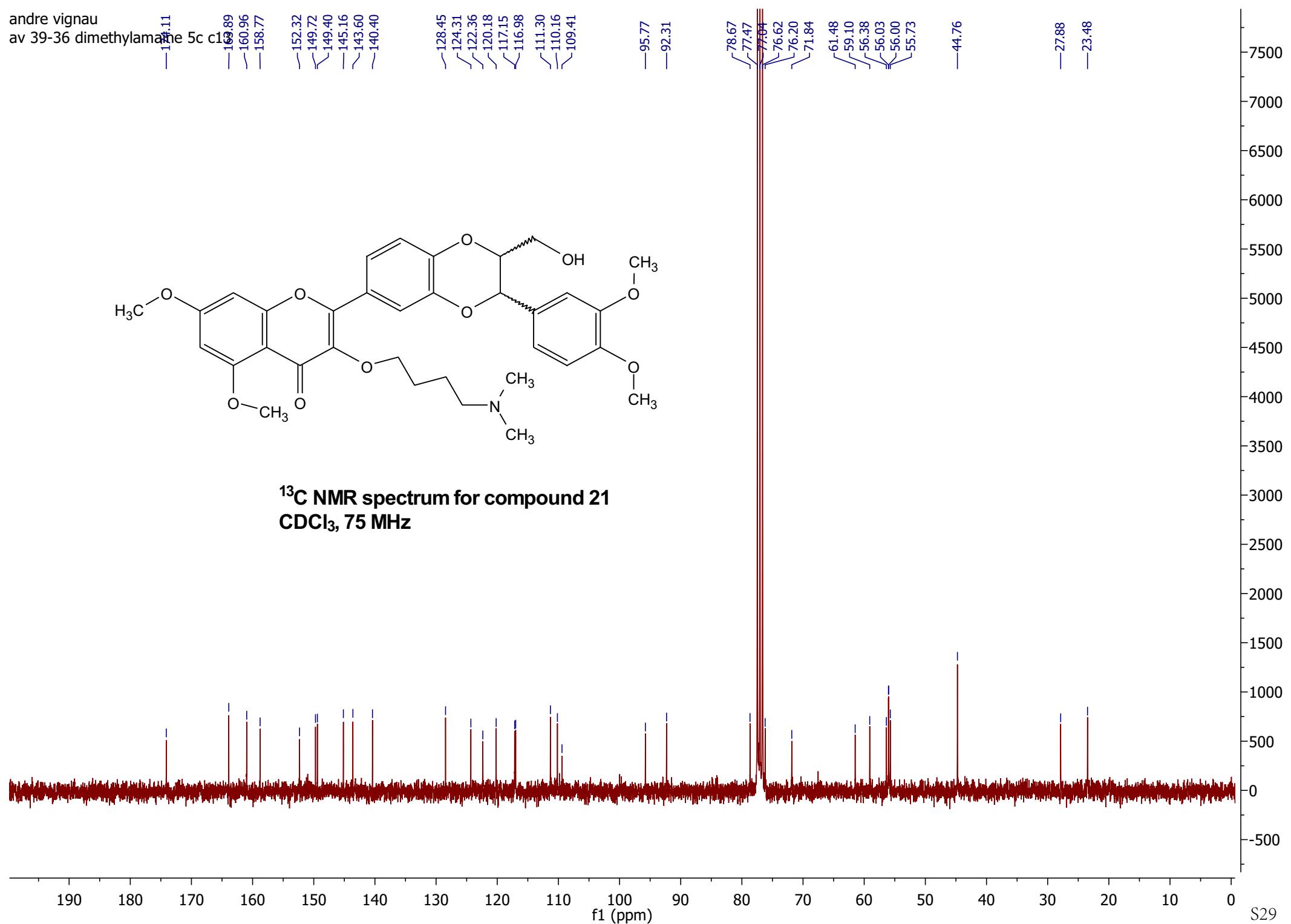
—95.77
—92.31

—78.67
—77.47
—77.04
—76.62
—76.20
—71.84
—61.48
—59.10
—56.38
—56.03
—56.00
—55.73

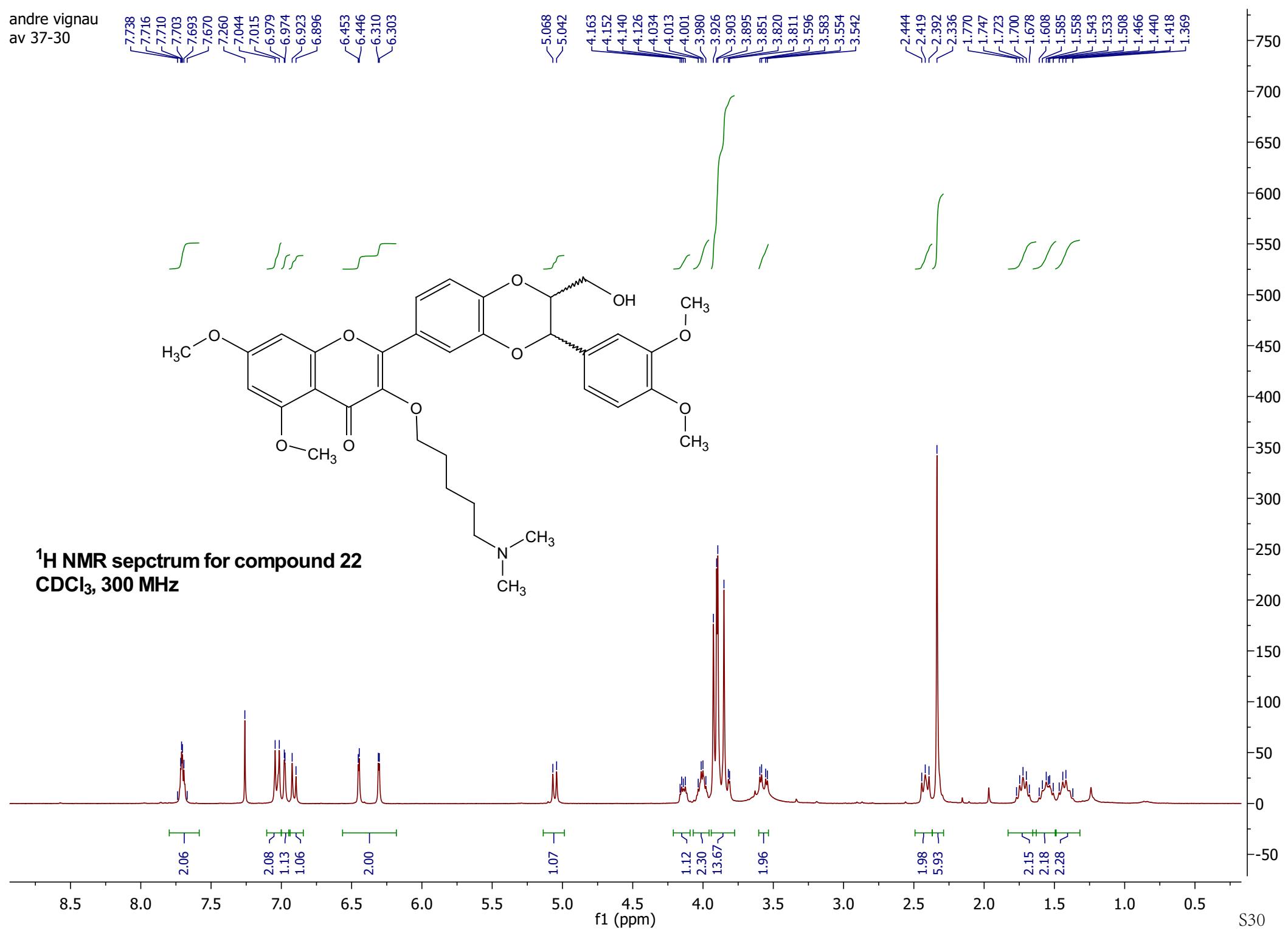
—44.76
—27.88
—23.48



¹³C NMR spectrum for compound 21
CDCl₃, 75 MHz



andre vignau
av 37-30



andre vignau
av 37-30 c13

-174.10

-163.87
-160.96
-158.77
-152.32
-149.69
-149.39
-145.14
-143.55
-140.47

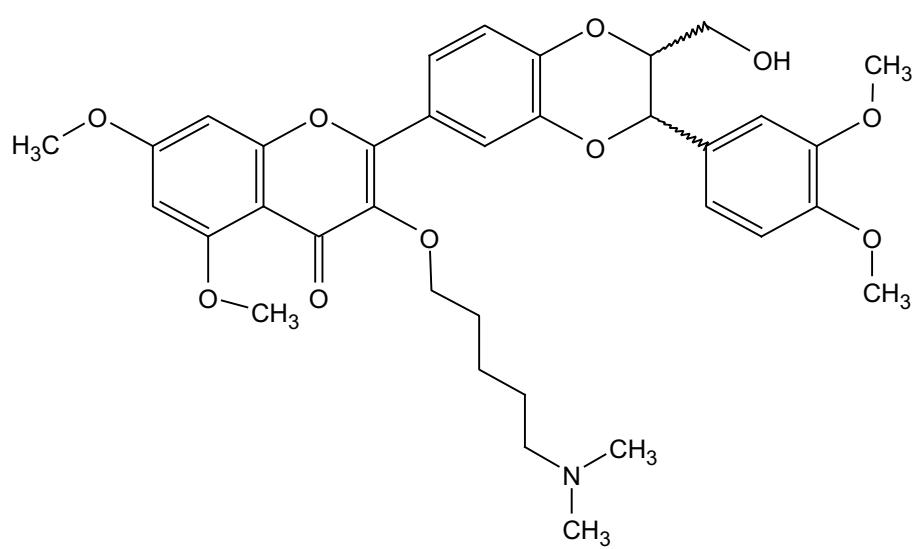
-128.53
-124.32
-122.38
-120.15
-117.18
-116.92
-111.31
-110.17
-109.42

-95.75
-92.30

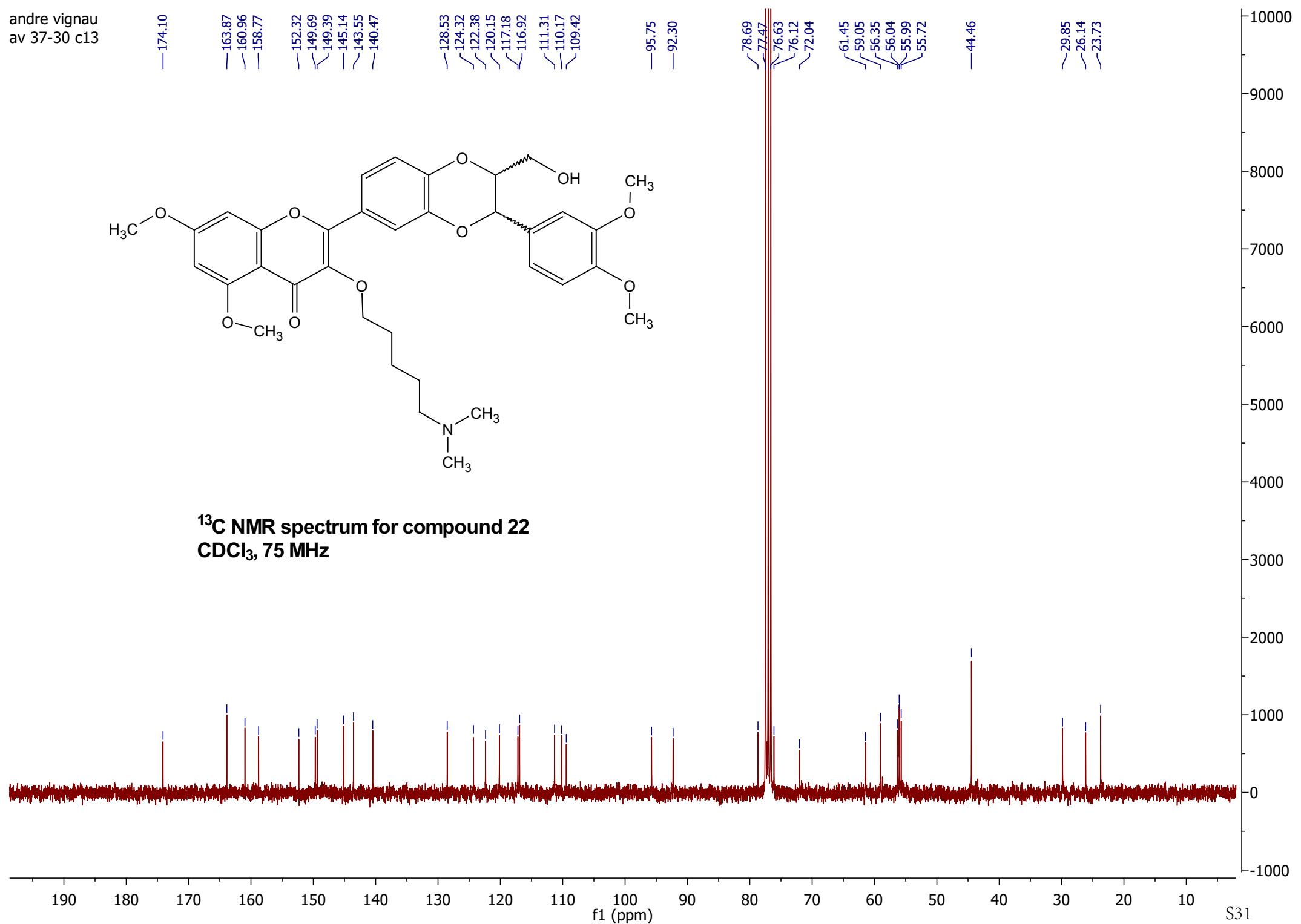
78.69
77.47
76.63
76.12
72.04
61.45
59.05
56.35
56.04
55.99
55.72

-44.46

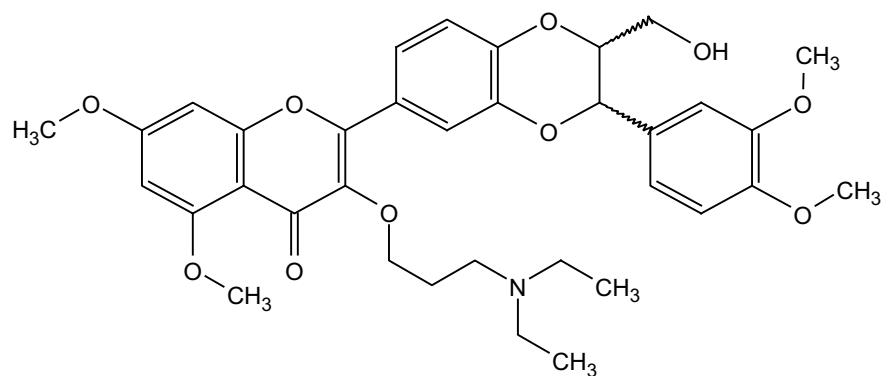
~29.85
~26.14
~23.73



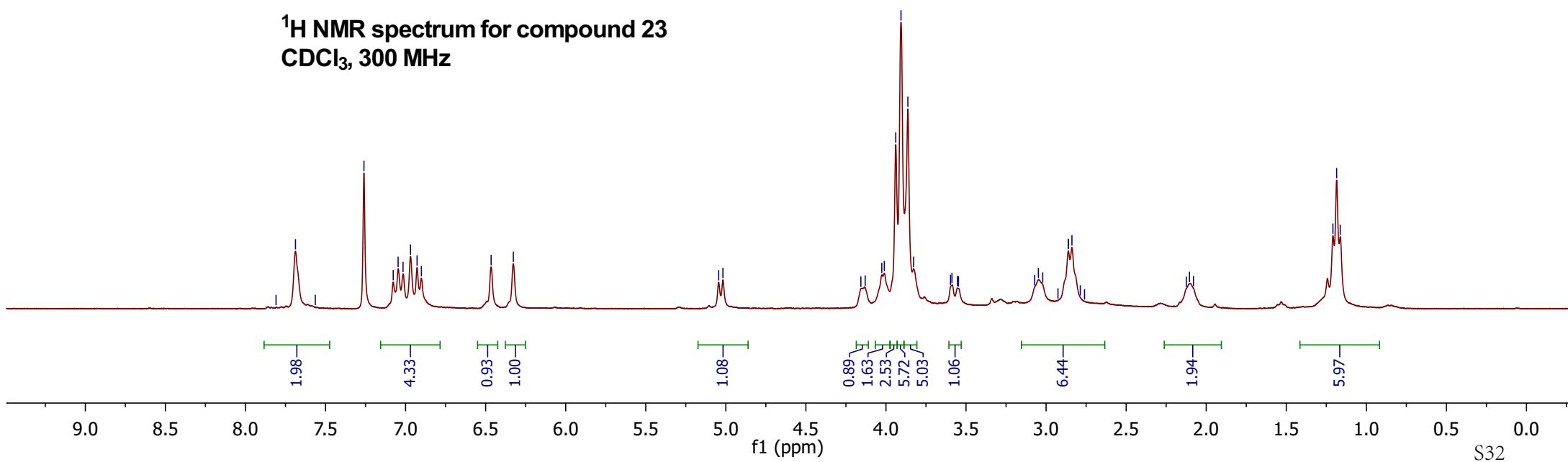
¹³C NMR spectrum for compound 22
CDCl₃, 75 MHz



linker nmr
bv-17-232 cdcl3

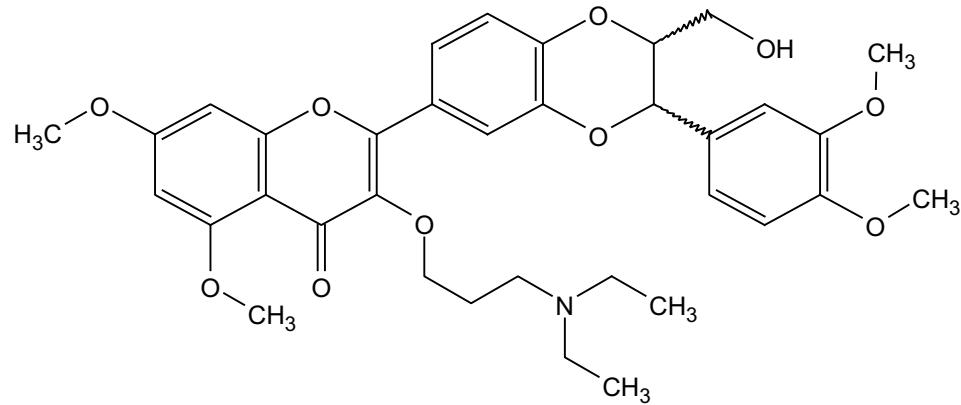


^1H NMR spectrum for compound 23
 CDCl_3 , 300 MHz

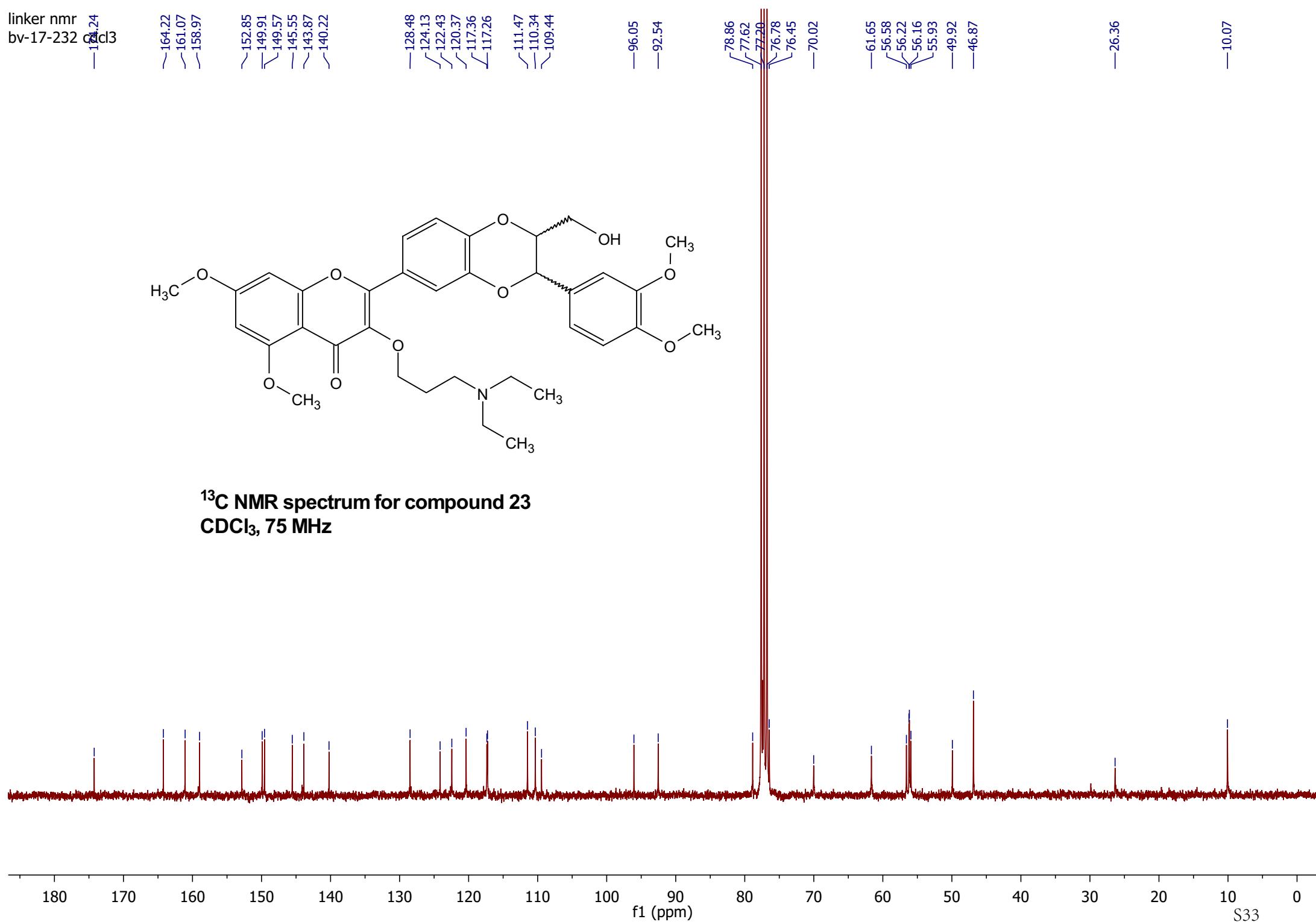


linker nmr
bv-17-232 cd3

-164.22
-161.07
-158.97
-152.85
-149.91
-149.57
-145.55
-143.87
-140.22
-128.48
-124.13
-122.43
-120.37
-117.36
-117.26
-111.47
-110.34
-109.44
-96.05
-92.54
-78.86
-77.62
-77.20
-76.78
-76.45
-70.02
-61.65
-56.58
-56.22
-56.16
-55.93
-49.92
-46.87
-26.36
-10.07

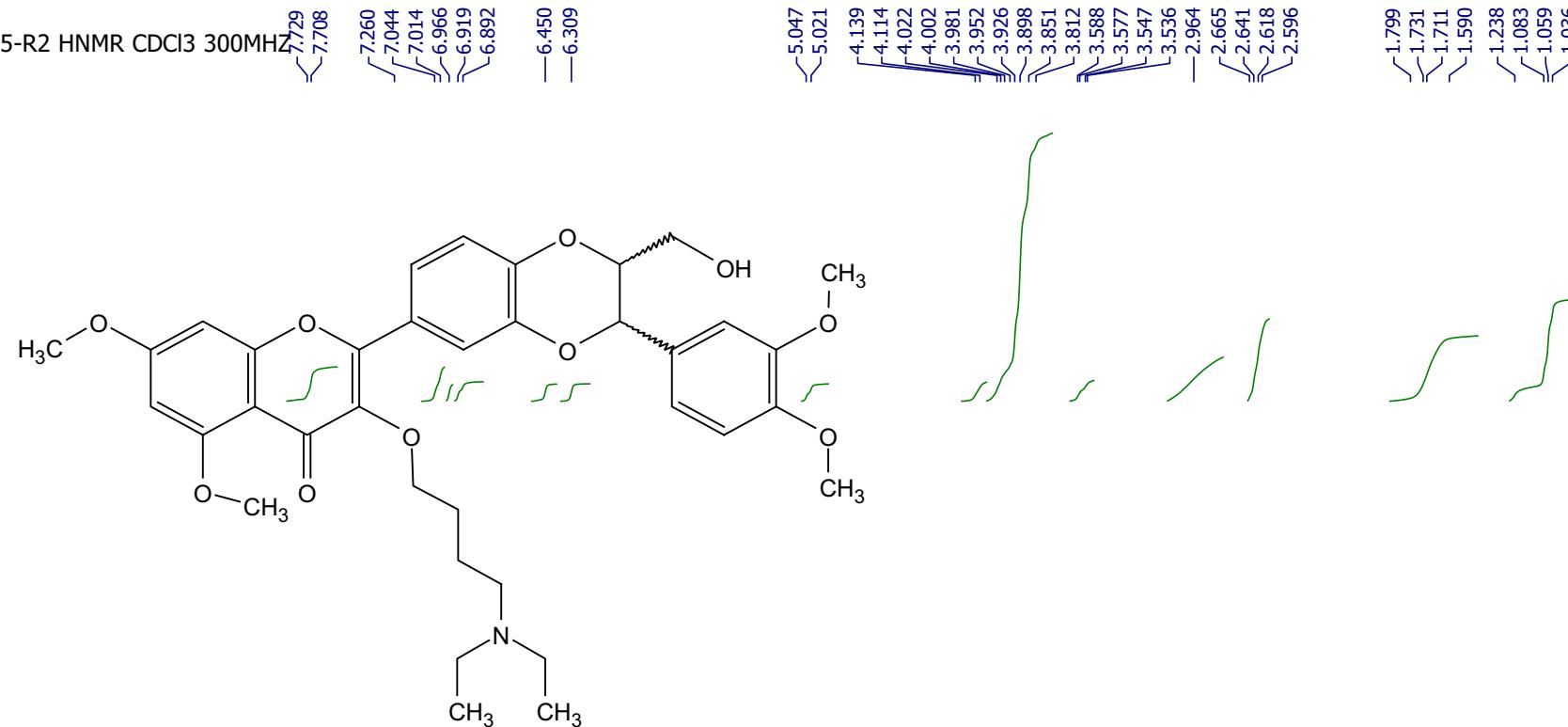


¹³C NMR spectrum for compound 23
CDCl₃, 75 MHz

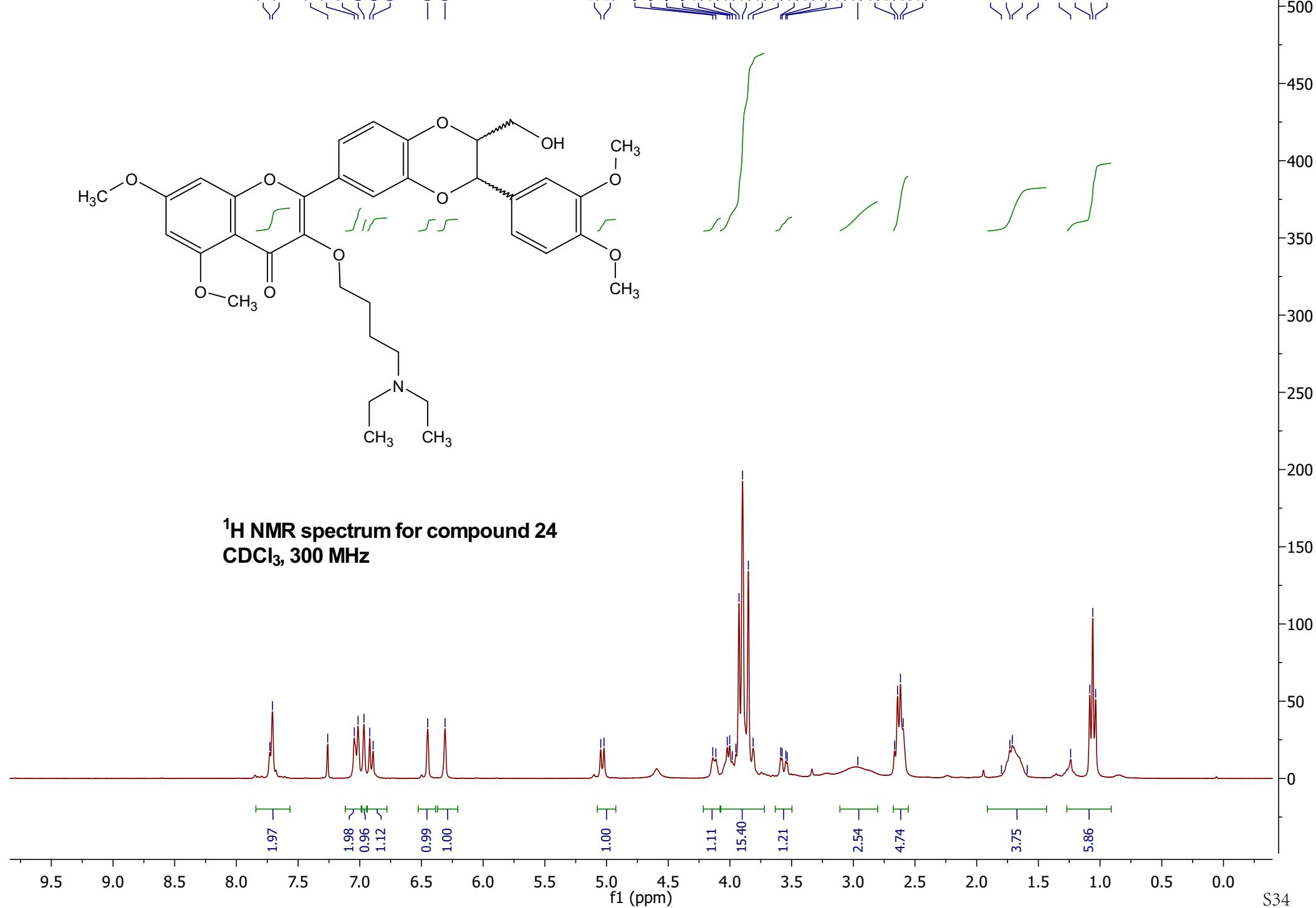


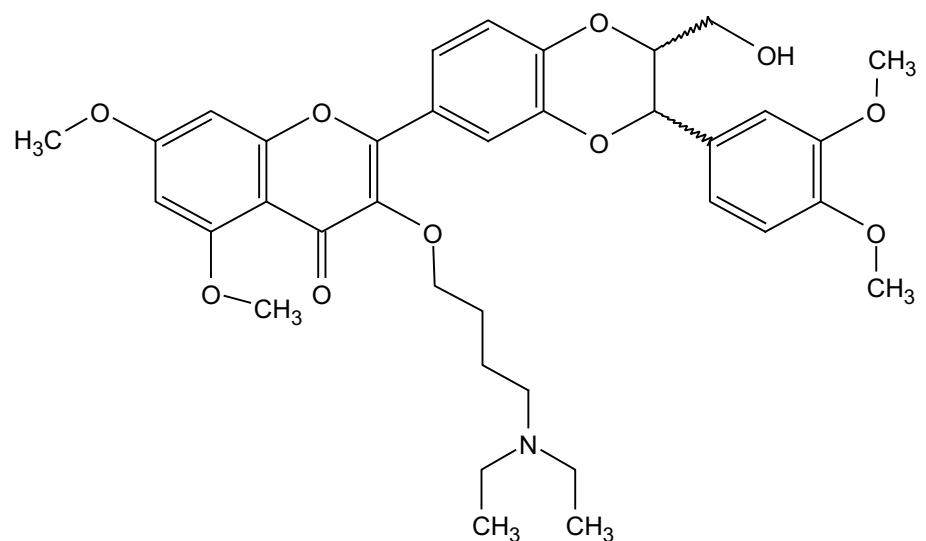
Andre

AV-39-35-R2 HNMR CDCl₃ 300MHz

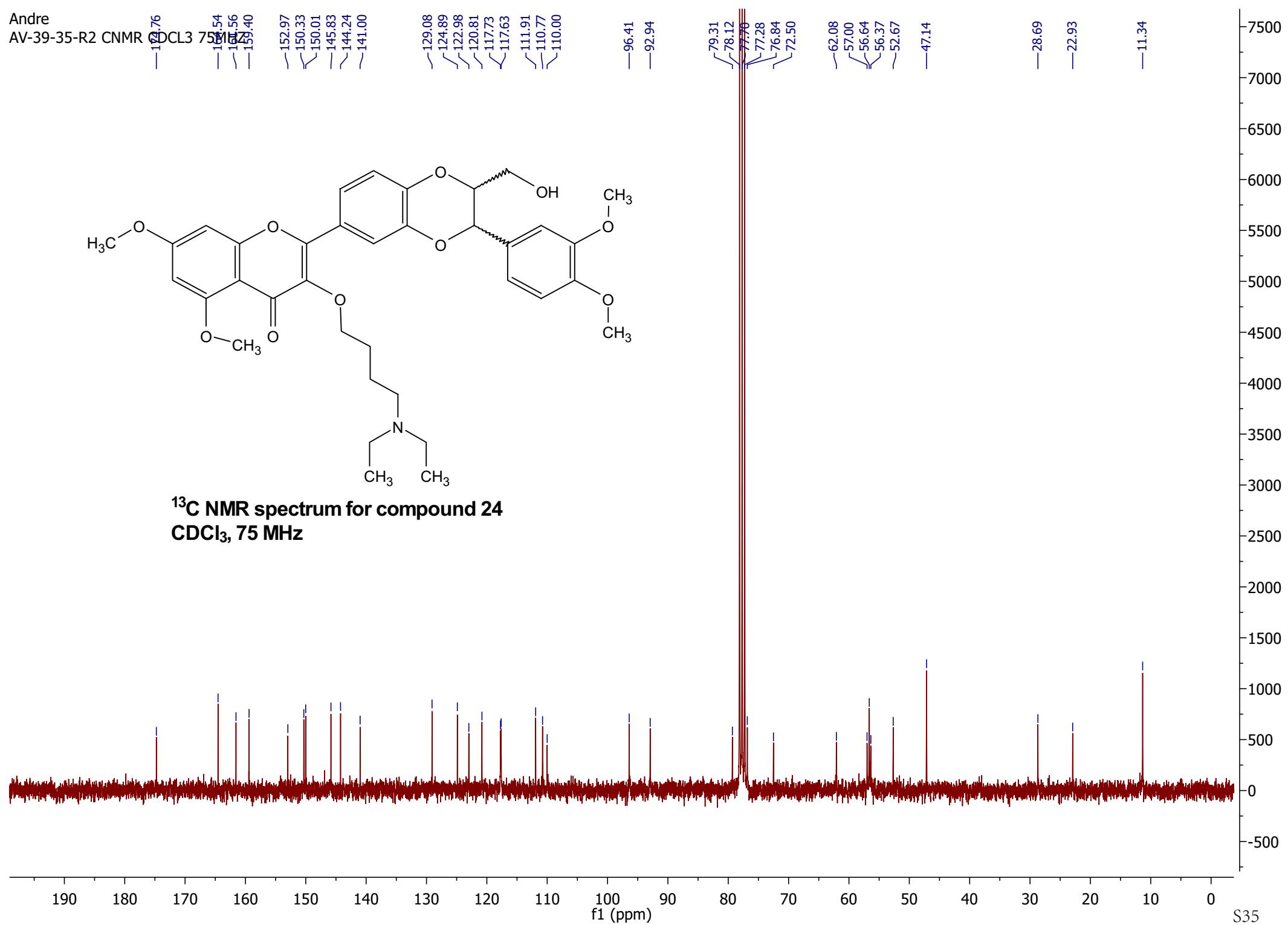


¹H NMR spectrum for compound 24
CDCl₃, 300 MHz





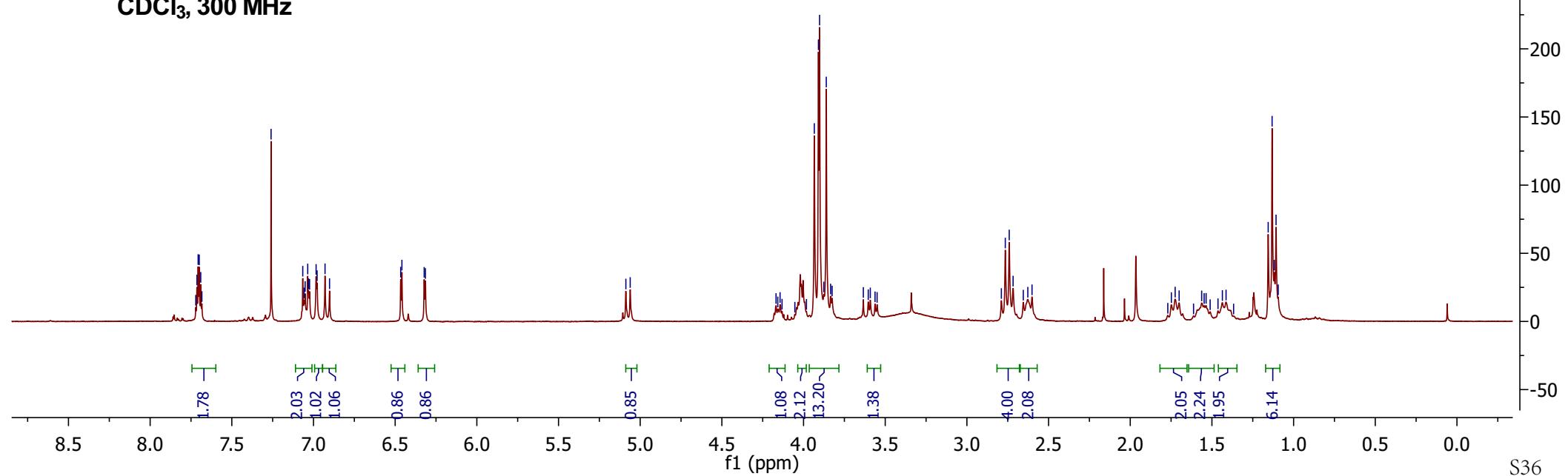
**¹³C NMR spectrum for compound 24
CDCl₃, 75 MHz**



andre vignau
av 37-16 diethylamine



¹H NMR spectrum for compound 25
CDCl₃, 300 MHz



-174.07

-163.92
-160.96
-158.81
-152.53
-149.70
-149.39
-145.26
-143.58
-140.33

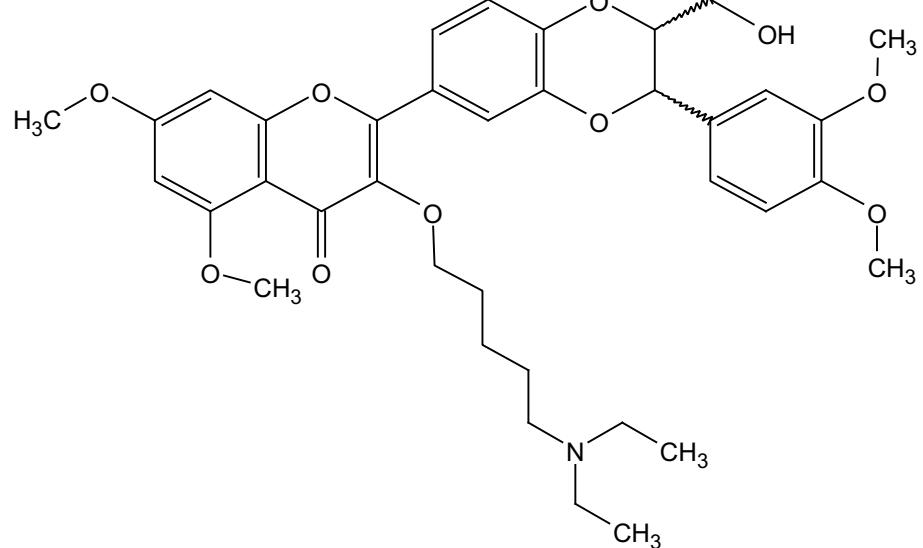
-128.51
-124.18
-122.34
-120.15
-117.23
-117.00
-111.33
-110.21
-109.43

-95.82
-92.34

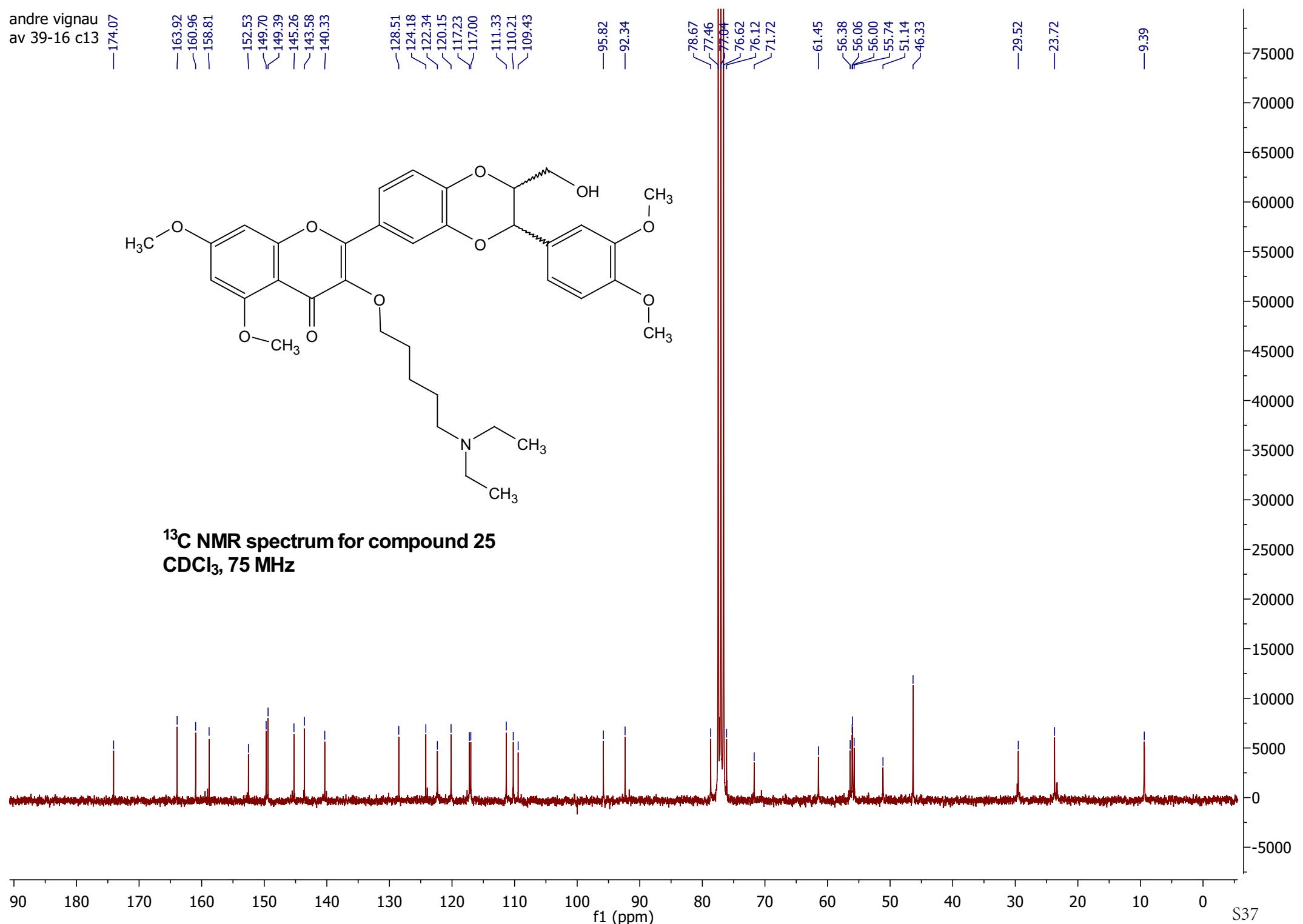
78.67
77.46
77.04
76.62
76.12
71.72
-61.45
56.38
56.06
56.00
55.74
51.14
-46.33

-29.52
-23.72

9.39



¹³C NMR spectrum for compound 25
CDCl₃, 75 MHz



linker nmr
bv-48-52-r1 cpl

7.082

7.2597
7.0630
7.0367
7.0141
6.9685
6.9224
6.8951

6.4576
6.3198

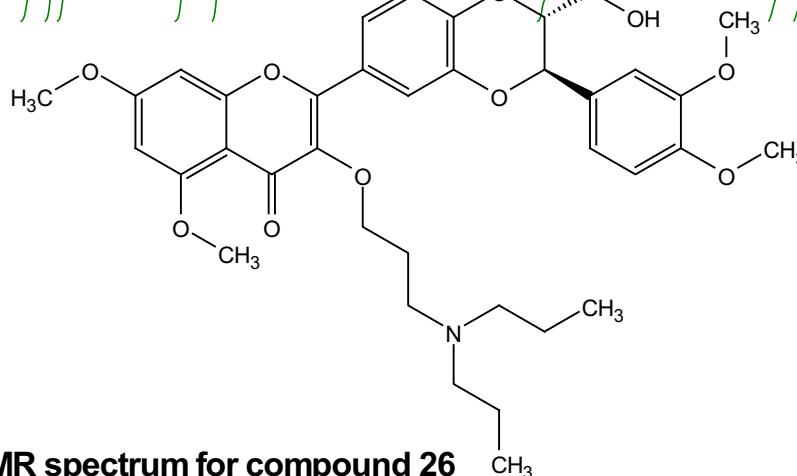
5.0456
5.0188

4.1446
4.1185
4.0791
4.0338
4.0160
4.0002
3.9336
3.9036
3.8549
3.8217
3.5931
3.5821
3.5518
3.5404

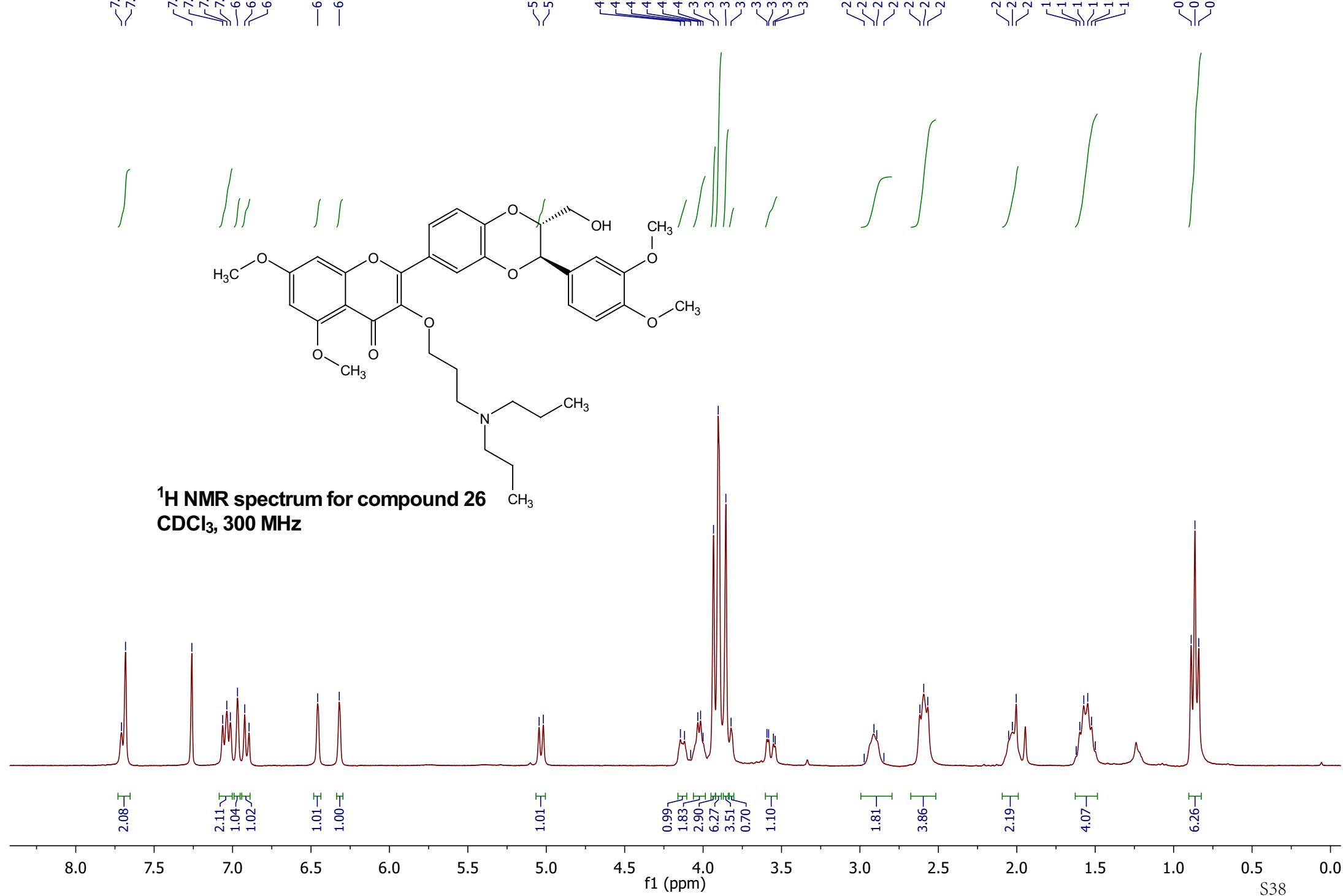
2.9730
2.9107
2.8928
2.8470
2.6177
2.5932
2.5675

2.0522
2.0278
2.0037
1.6210
1.5983
1.5727
1.5477
1.5233
1.4993

0.8882
0.8641
0.8399

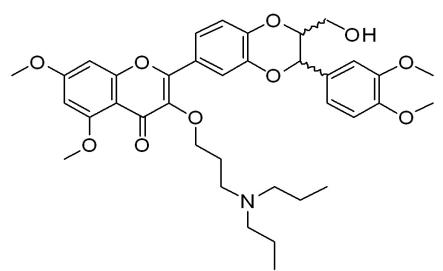


¹H NMR spectrum for compound 26
CDCl₃, 300 MHz

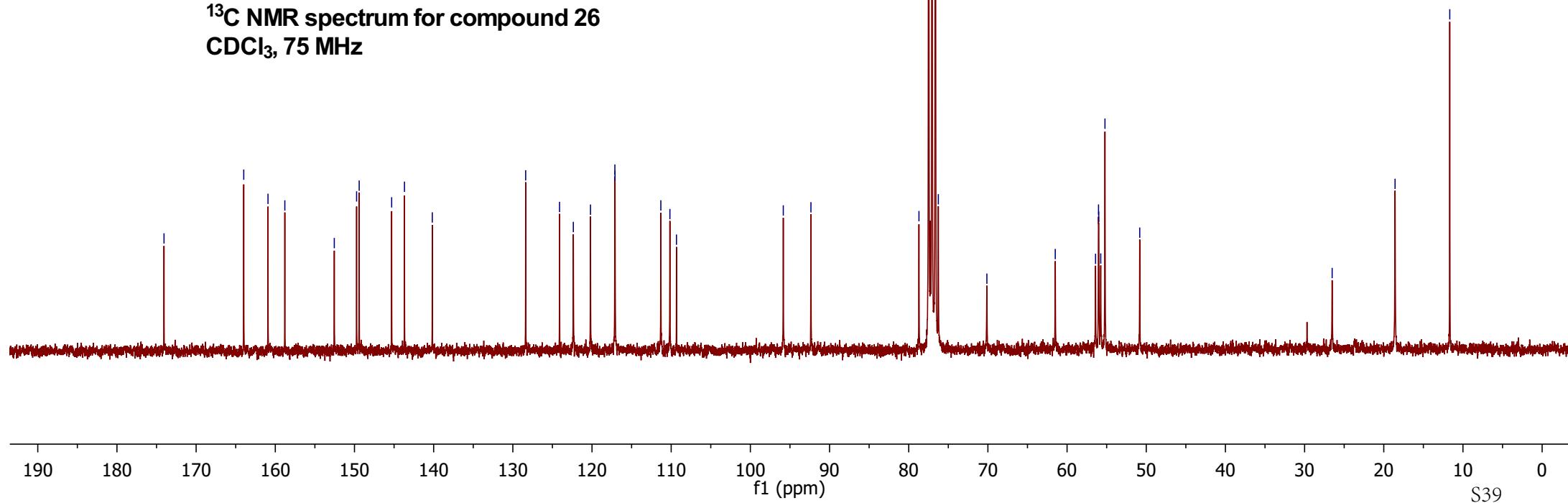


linker nmr
bv-48-52-r1 cdcl₃

-174.06
-163.99
-160.92
-158.80
-152.55
-149.73
-149.40
-145.30
-143.69
-140.17
-128.38
-124.10
-122.36
-120.19
-117.11
-117.09
-111.31
-110.16
-109.33
-95.84
-92.35
-78.71
-76.76
-70.13
-61.49
-56.40
-56.03
-56.00
-55.75
-55.21
-50.82
-26.52
-18.58
-11.66



¹³C NMR spectrum for compound 26
CDCl₃, 75 MHz

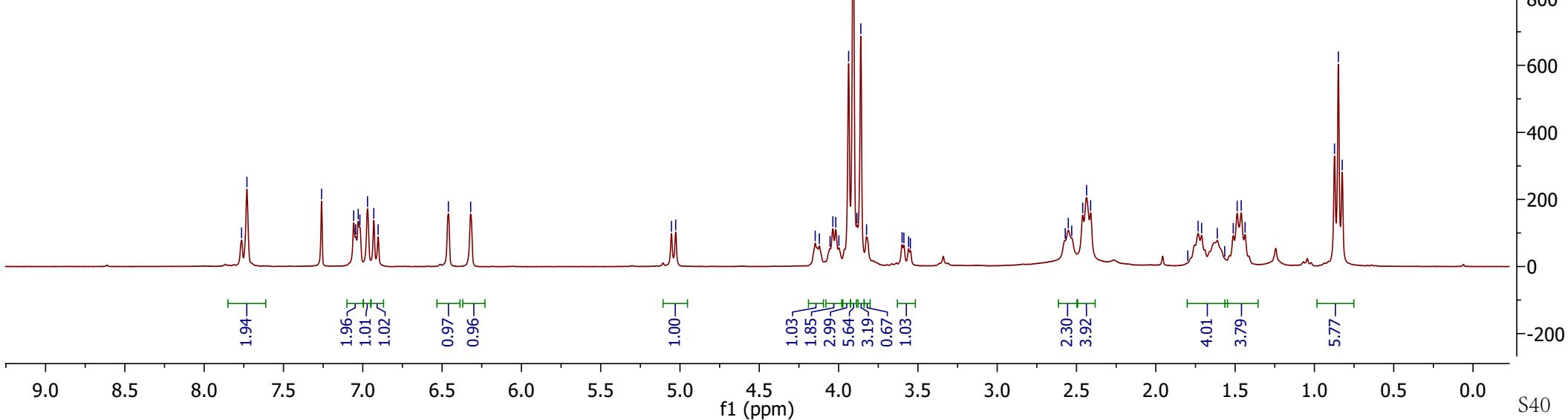


Andre

AV-39-41-R2 HNMR CDCl₃ 300 MHz



¹H NMR spectrum for compound 27
CDCl₃, 300 MHz



Andre

AV-39-41-R2 CNMR CDCl₃ 75MHz

-14.76

-137.51

-136.60

-152.88

-150.35

-150.04

-145.74

-144.24

-141.09

-129.03
-125.02
-123.07
-120.80
-117.73
-117.61
-111.92
-110.73
-110.05

-96.40

-92.92

79.27

78.11

77.68

77.26

76.85

72.72

62.17

57.01

56.65

56.63

56.37

56.20

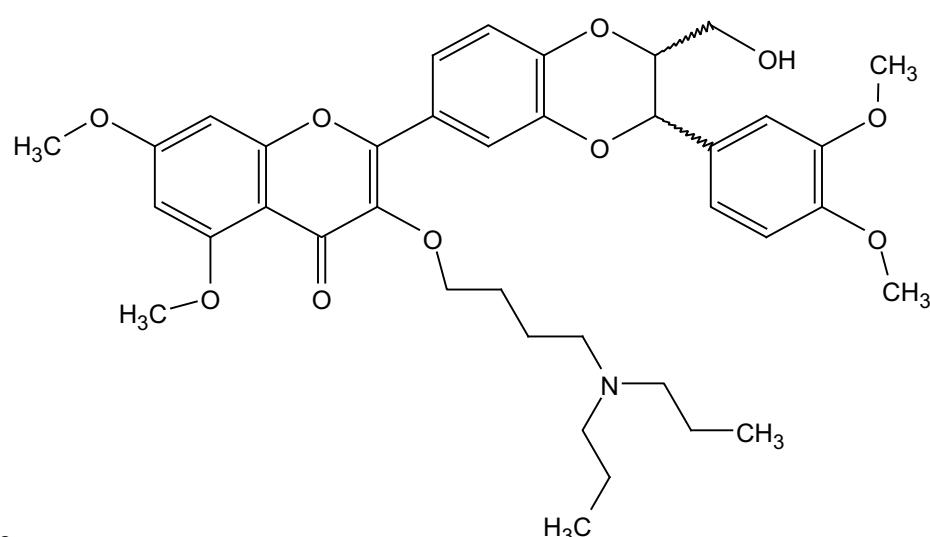
54.10

-28.71

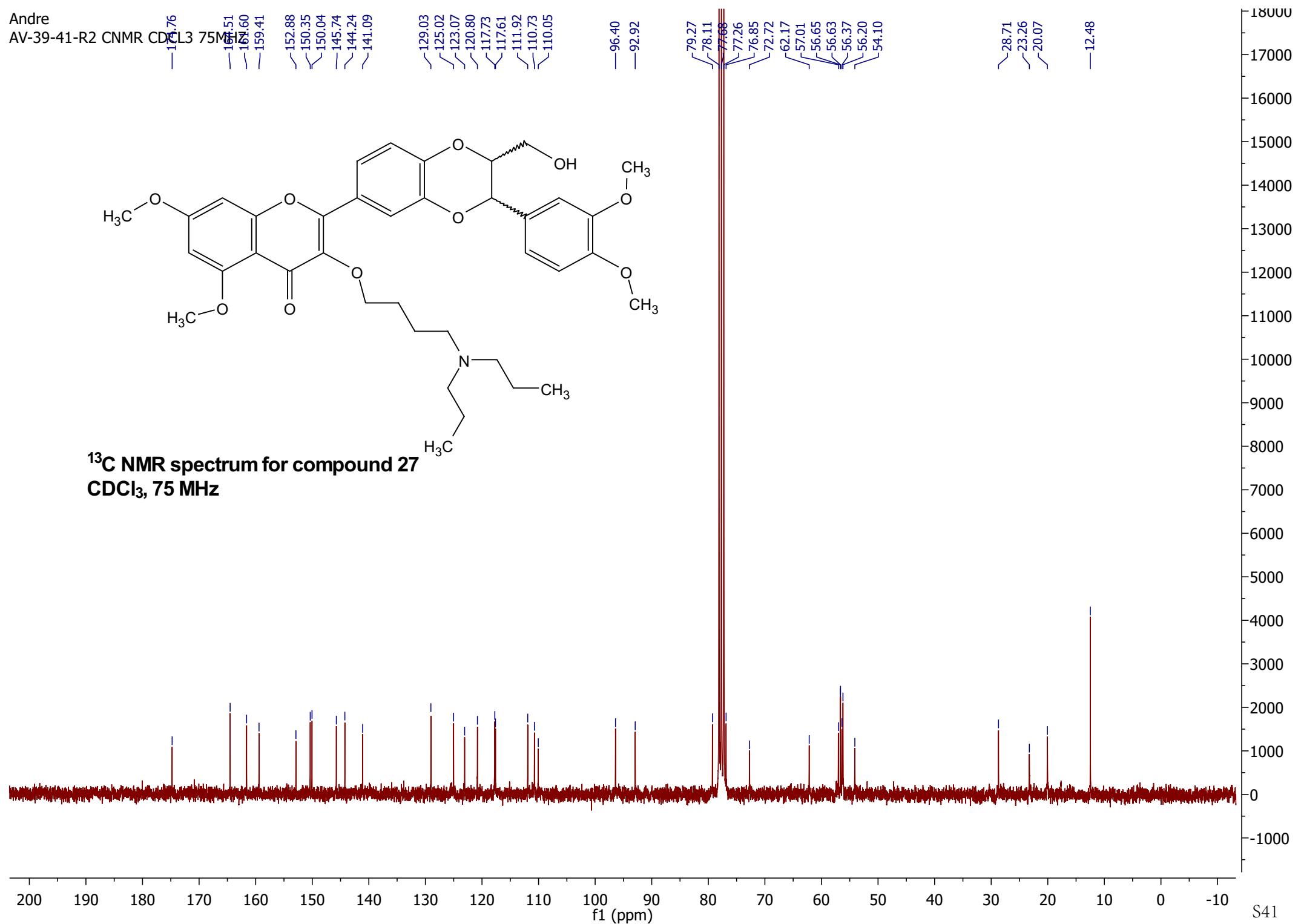
-23.26

-20.07

-12.48



¹³C NMR spectrum for compound 27
CDCl₃, 75 MHz



linker nmr
BV-48-36-r1

7.739

7.260

7.071

7.040

6.992

6.940

6.912

6.470

6.327

5.080

5.054

4.174

4.148

4.050

4.030

4.011

3.939

3.914

3.866

3.836

3.614

3.604

3.573

-2.682

-2.589

-2.508

1.791

1.768

1.745

1.722

1.699

1.676

1.601

1.503

1.474

1.448

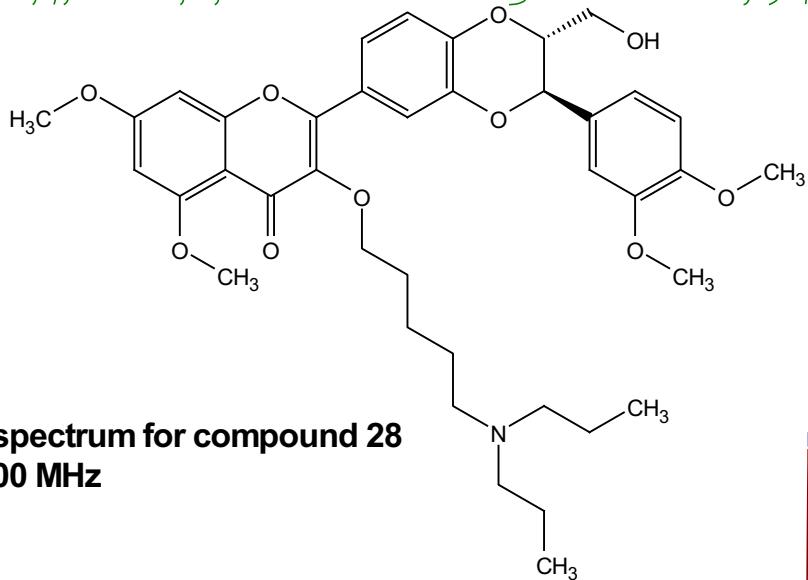
1.425

1.401

0.923

0.898

0.874



¹H NMR spectrum for compound 28
CDCl₃, 300 MHz

1.97

2.09

0.92

1.16

1.03

1.00

1.14

1.10

2.25

3.48

0.64

1.06

6.28

2.46

6.05

2.36

1.19

8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

f1 (ppm)

S42

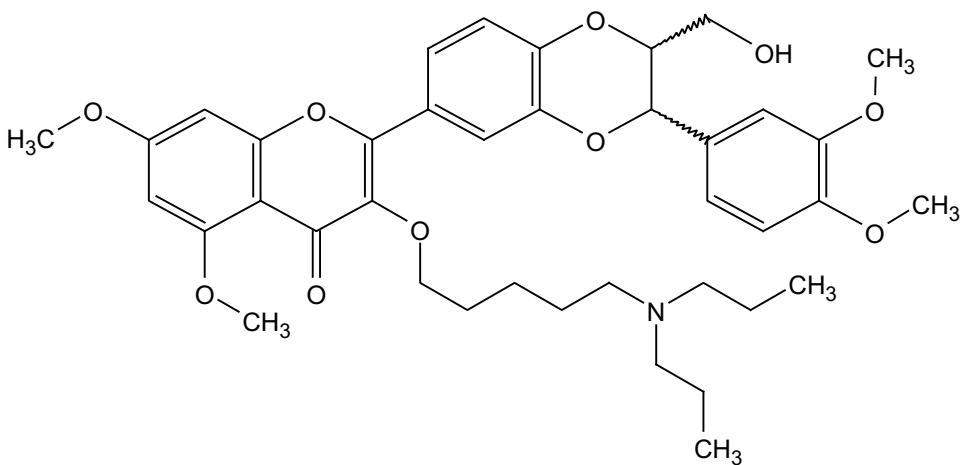
linker nmr

BV-48-36-r1 cdcl₃

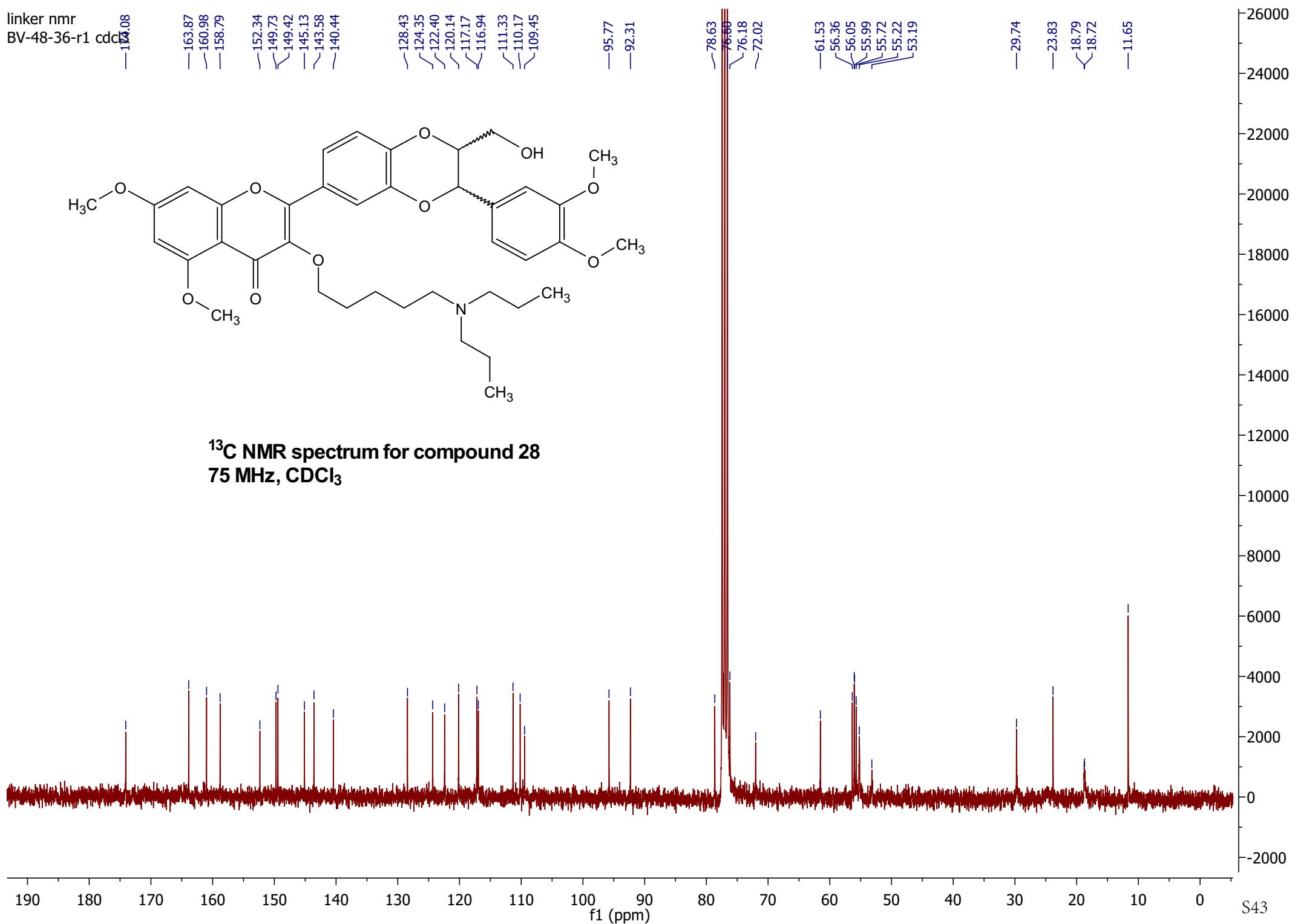
-163.87
-160.98
-158.79
-152.34
-149.73
-149.42
-145.13
-143.58
-140.44

-128.43
-124.35
-122.40
-120.14
-117.17
-116.94
-111.33
-110.17
-109.45

-95.77
-92.31

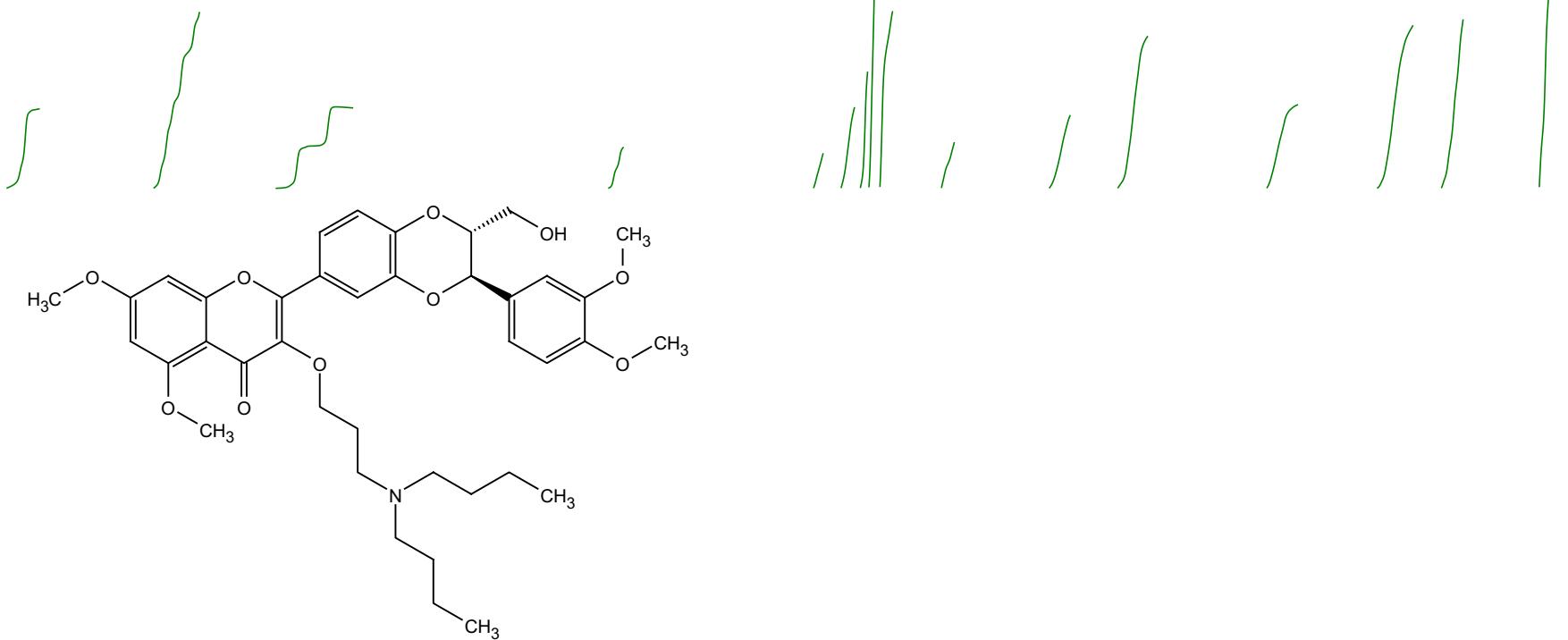


¹³C NMR spectrum for compound 28
75 MHz, CDCl₃

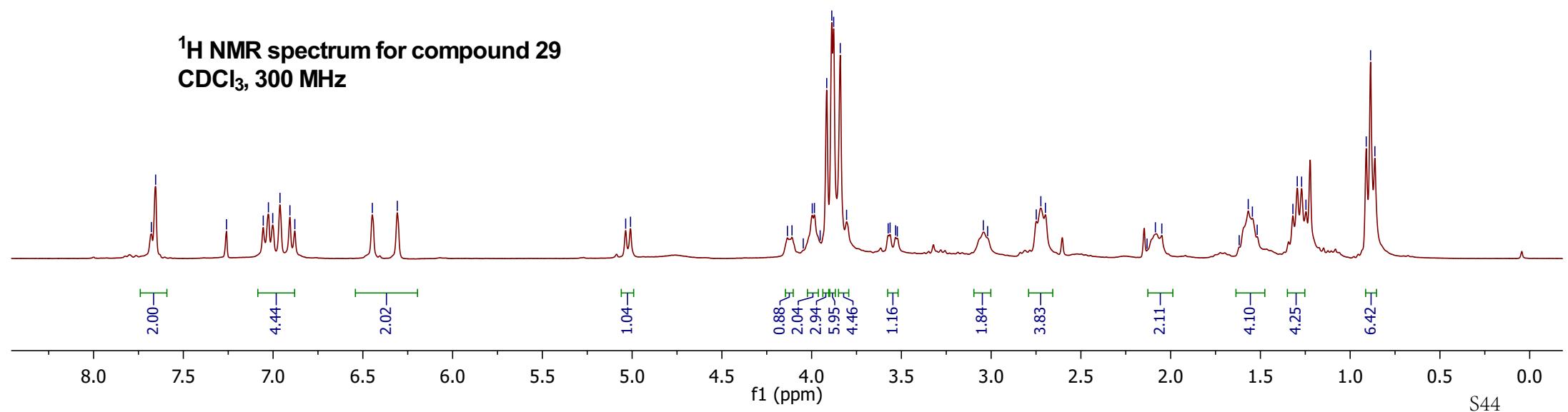


linker nmr
bv-48-13-r2 cdc

3.678
~7.654
7.260
7.055
~7.026
7.002
6.962
6.906
6.879
~6.447
~6.308
~5.036
5.009
4.135
4.109
4.047
3.997
3.984
3.984
3.952
3.916
3.888
3.877
3.840
3.805
3.574
3.563
3.532
3.523
3.043
3.020
2.749
2.723
2.698
2.132
2.085
2.049
1.618
1.568
1.545
1.518
1.319
1.295
1.270
1.246
0.911
0.886
0.862

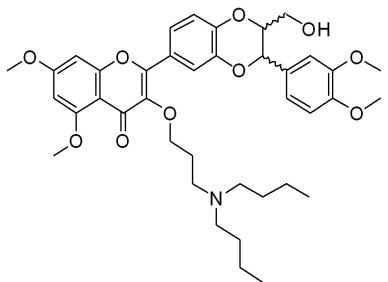


¹H NMR spectrum for compound 29
CDCl₃, 300 MHz

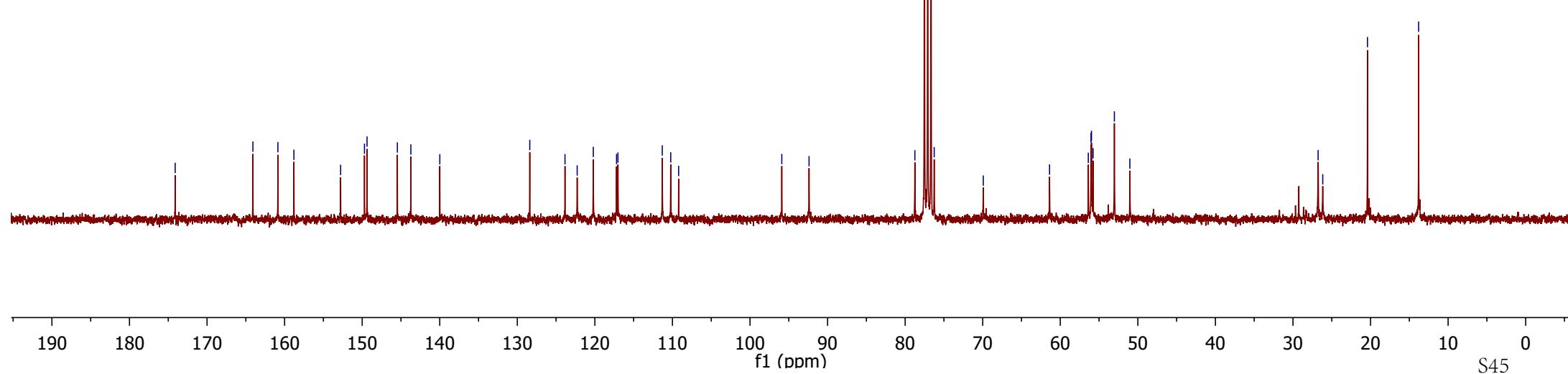


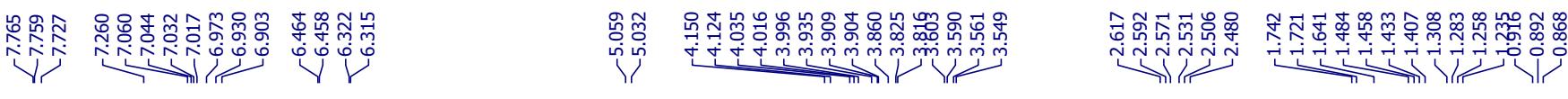
linker nmr
bv-48-13-r2 cdcl₃

—174.11
—164.09
—160.87
—158.80
—152.78
—149.71
—149.37
—145.46
—143.72
—139.99
—128.39
—123.85
—122.28
—120.20
—117.21
—117.03
—111.31
—110.22
—109.20
—95.90
—92.39
—78.74
—76.25
—69.93
—61.38
—56.39
—56.04
—55.98
—55.77
—53.02
—51.03
—26.76
—26.16
—20.40
—13.80

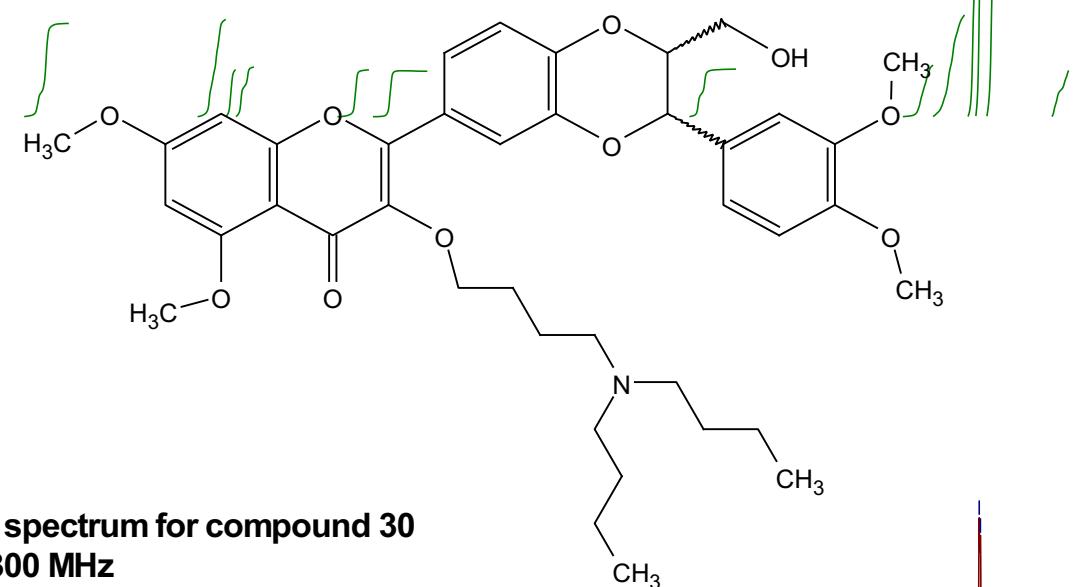


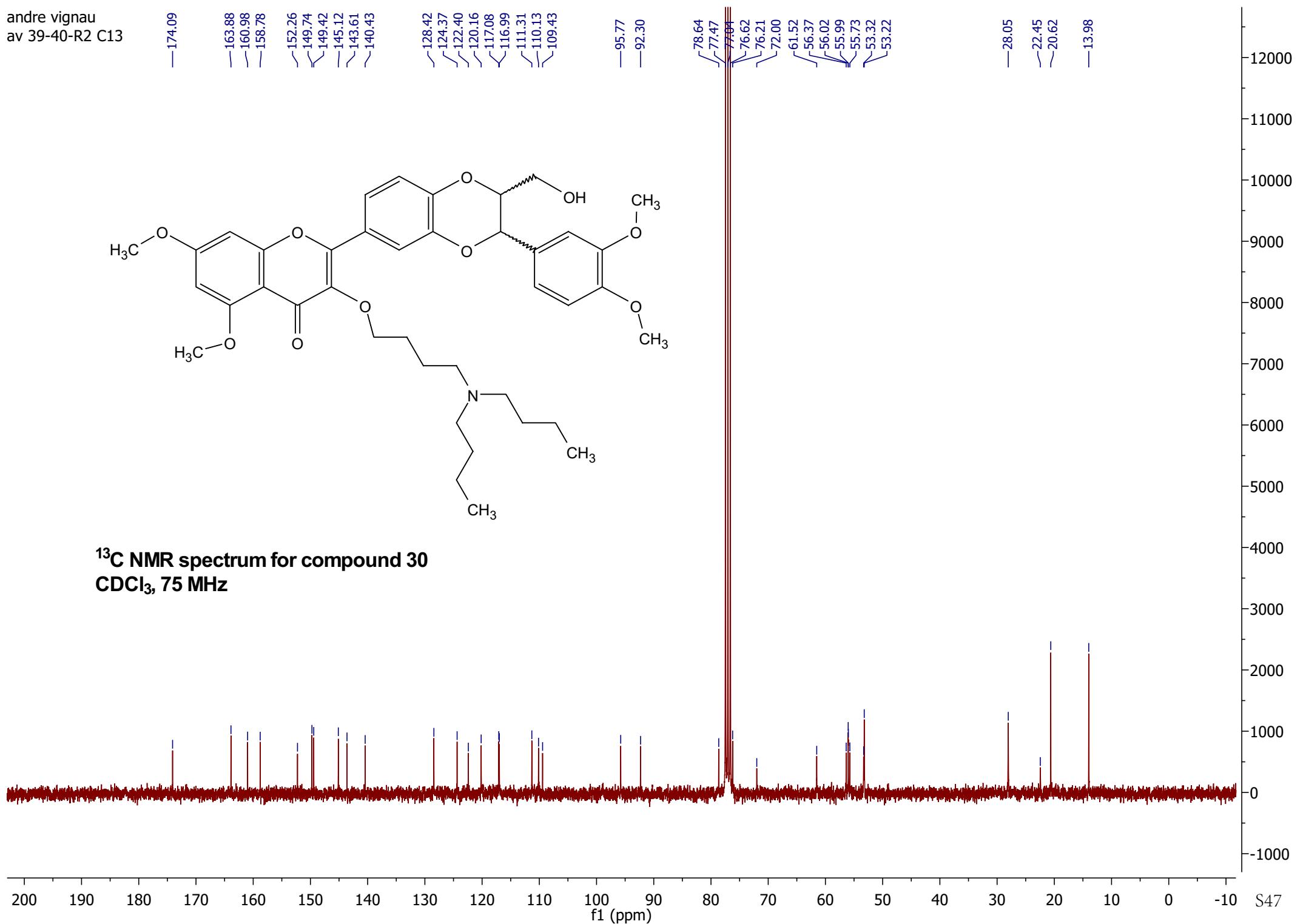
¹³C NMR spectrum for compound 29
CDCl₃, 75 MHz

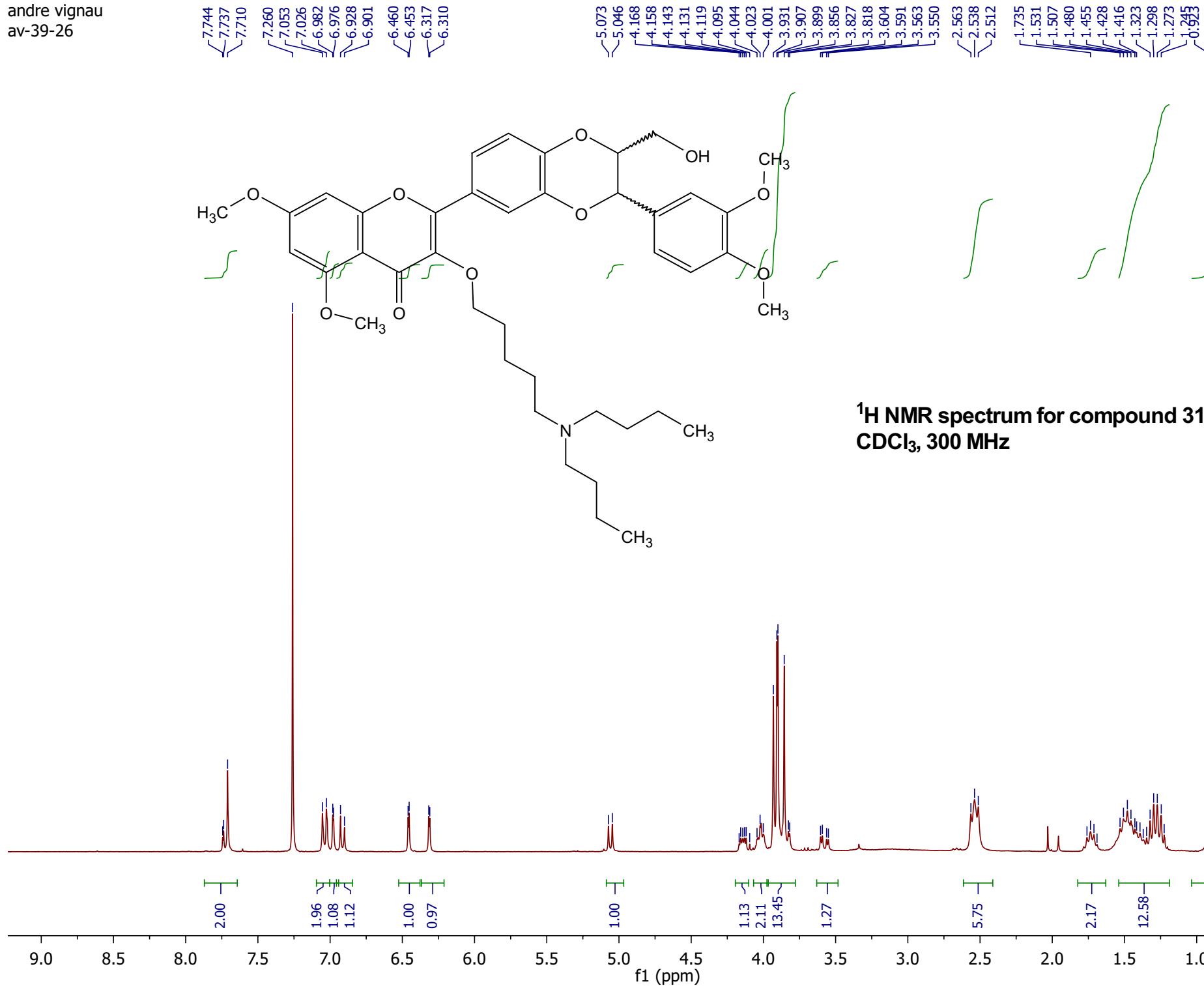




¹H NMR spectrum for compound 30
CDCl₃, 300 MHz







Andre

AV-39-26 CNMR CDCl₃ 75MHz

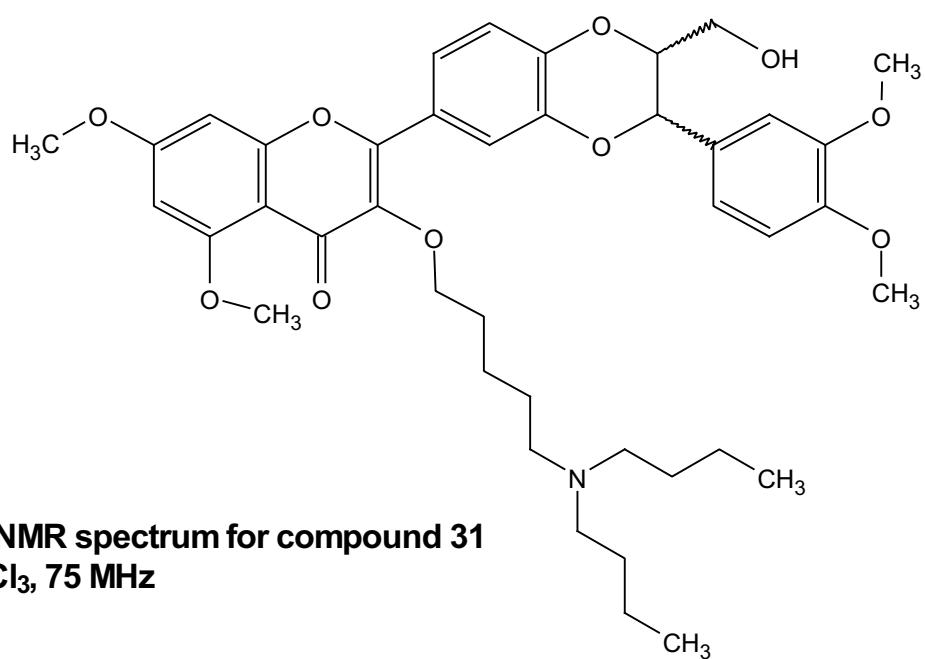
-174.76
-164.51
-161.58
-159.40
-152.99
-150.30
-150.00
-145.80
-144.20
-141.07

~129.12
~124.91
~123.03
~120.78
~117.75
~117.59
~111.91
~110.77
~110.03

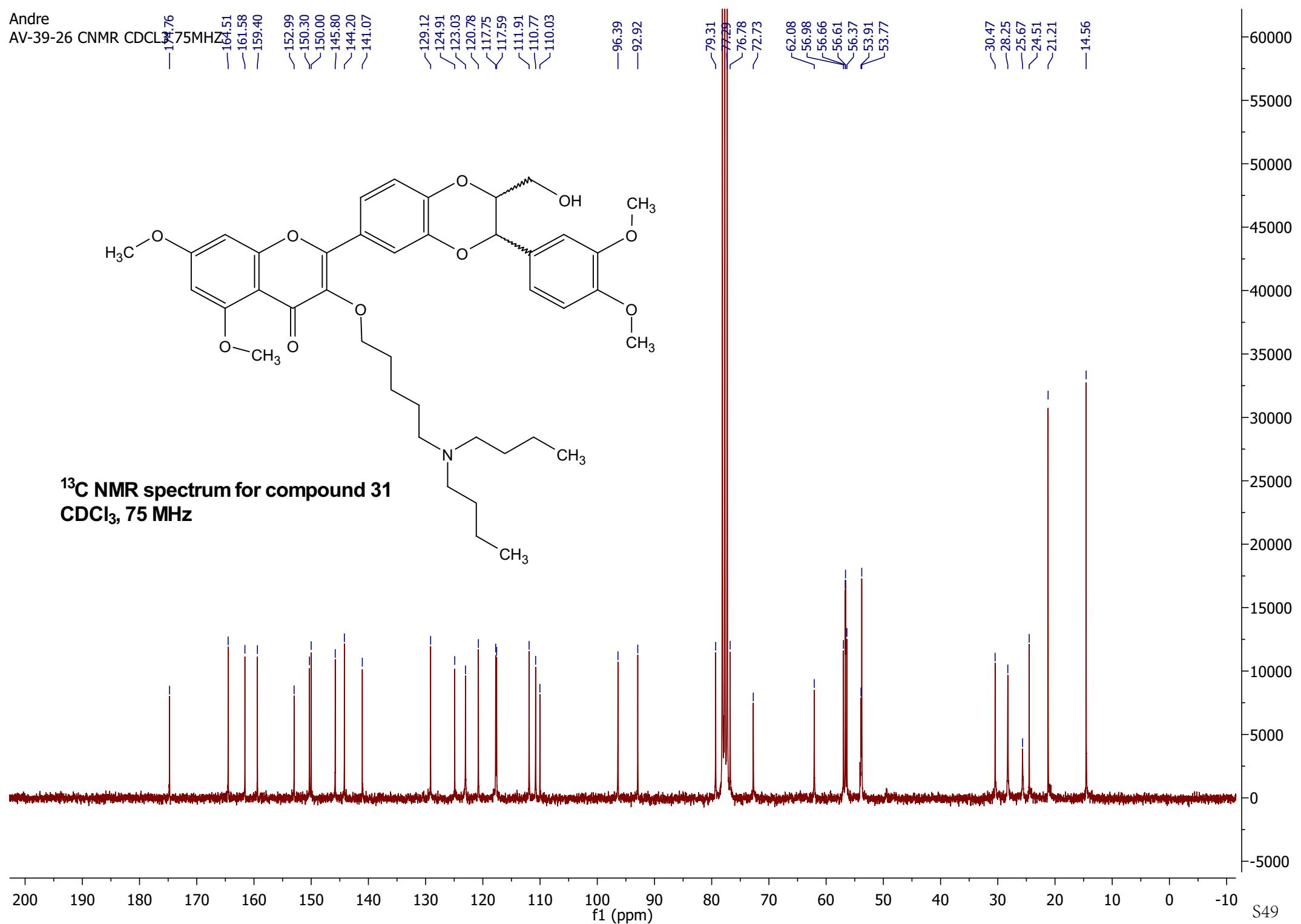
-96.39
-92.92

~79.31
~77.29
~76.78
~72.73
62.08
~56.98
~56.66
~56.61
~56.37
~53.91
~53.77

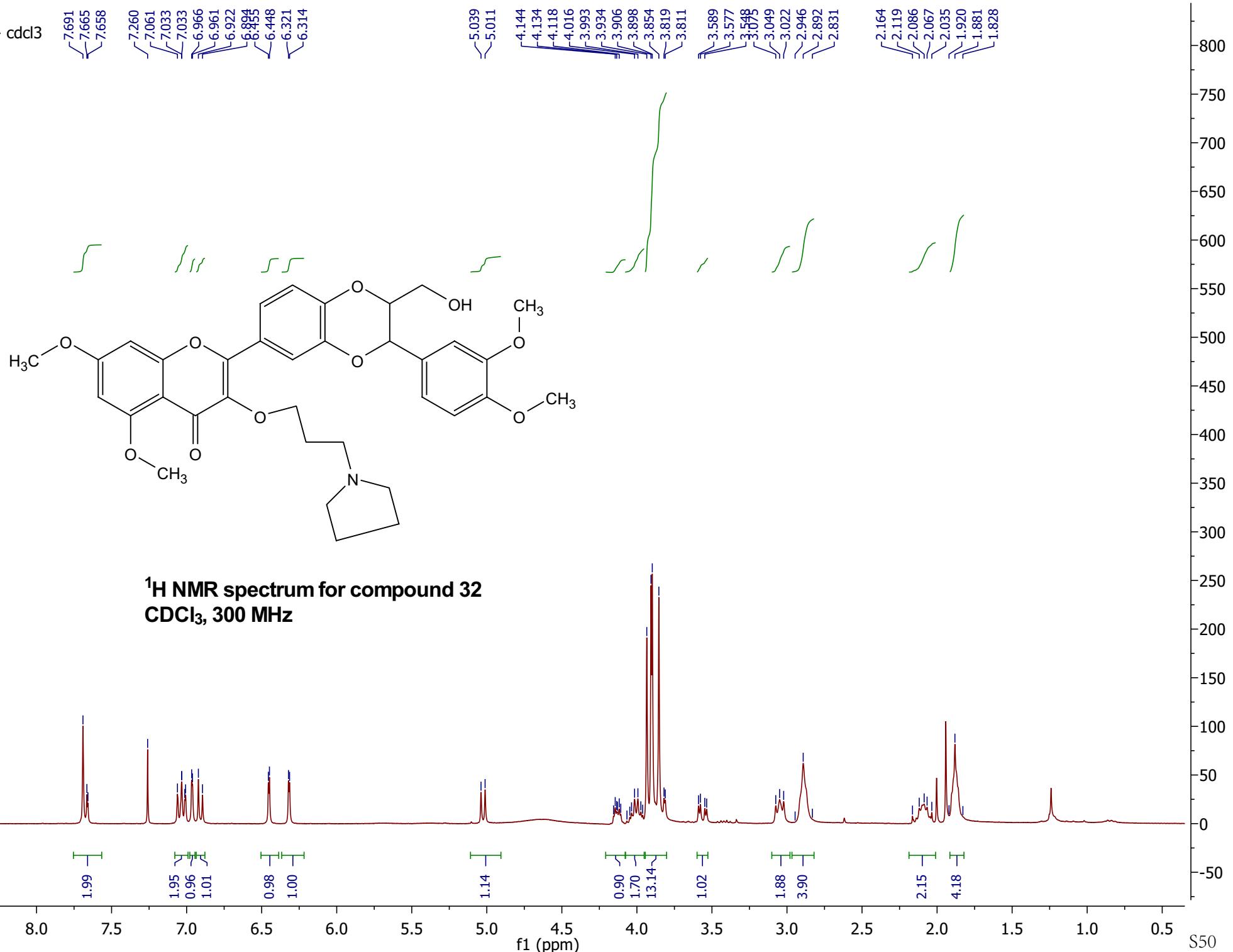
~30.47
~28.25
~25.67
~24.51
~21.21
-14.56



¹³C NMR spectrum for compound 31
CDCl₃, 75 MHz

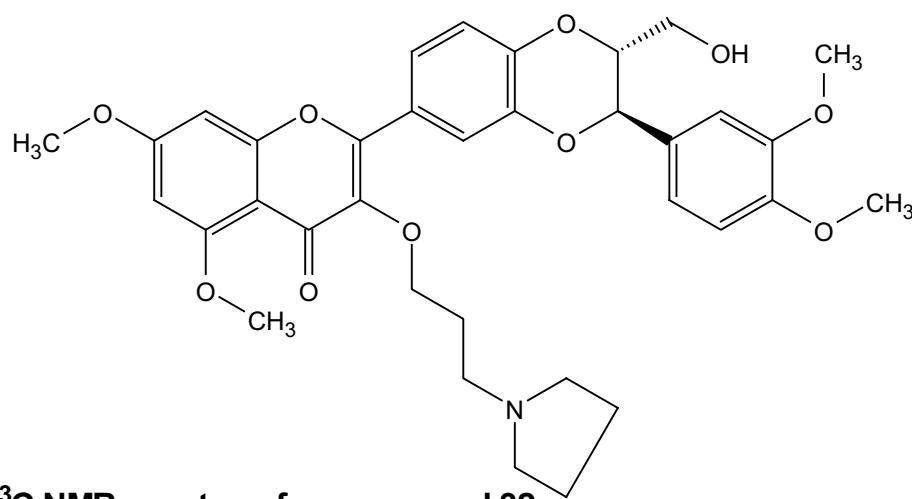


linker nmr
bv-17-37-r1- cdd3

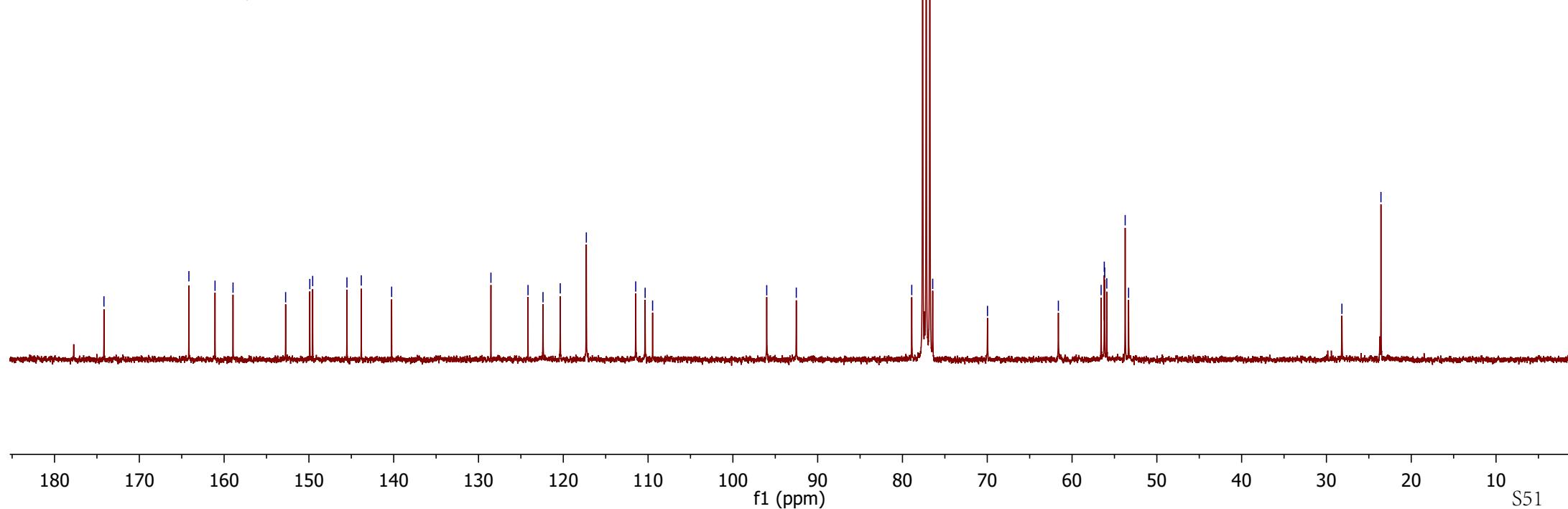


linker nmr
bv-17-37-r¹³C,1H
cccl3

—174.17
—164.15
—161.07
—158.95
—152.75
—149.89
—149.56
—145.51
—143.82
—140.24
—128.53
—124.16
—122.39
—120.36
—117.28
—111.46
—110.35
—109.47
—96.01
—92.52
—78.91
—77.62
—77.20
—76.78
—76.43
—69.97
—61.61
—56.57
—56.20
—56.15
—55.91
—53.73
—53.33
—28.18
—23.57



¹³C NMR spectrum for compound 32
CDCl₃, 75 MHz

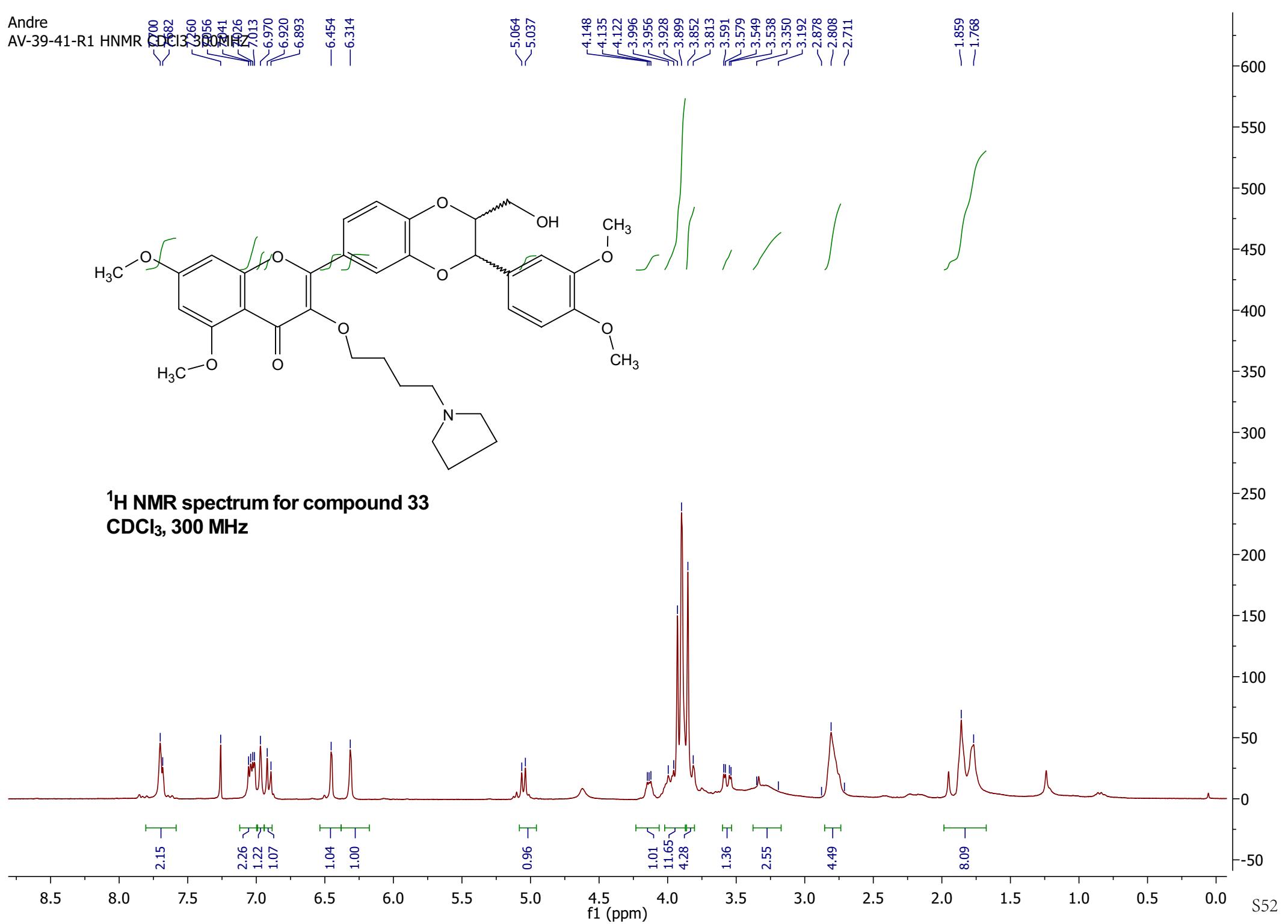


Andre

AV-39-41-R1 HNMR
CDCl₃, 300 MHz



¹H NMR spectrum for compound 33
CDCl₃, 300 MHz



Andre

AV-39-41-R1 CNMR CDCl₃ 75 MHz

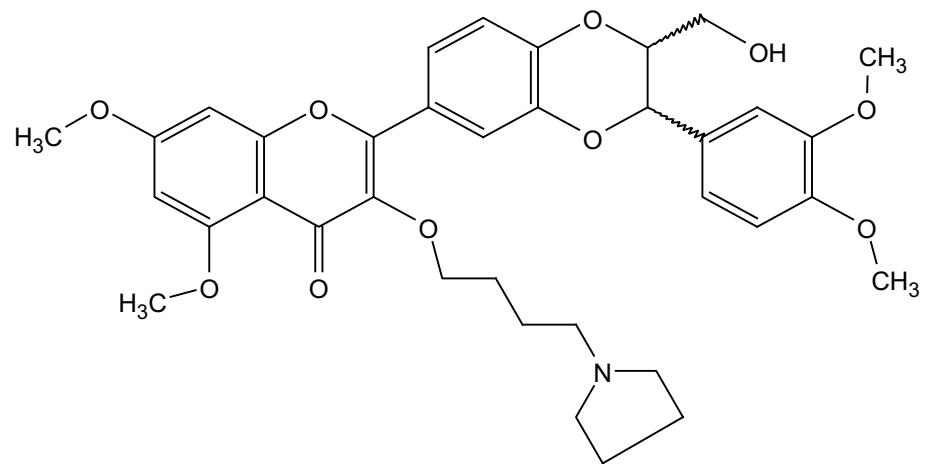
-160.77
-157.57
-154.54
-152.72
-150.42
-153.15
-150.31
-150.01
-145.88
-144.23
-140.93

-129.13
-124.78
-122.96
-120.78
-117.74
-117.70
-111.92
-110.78
-110.00

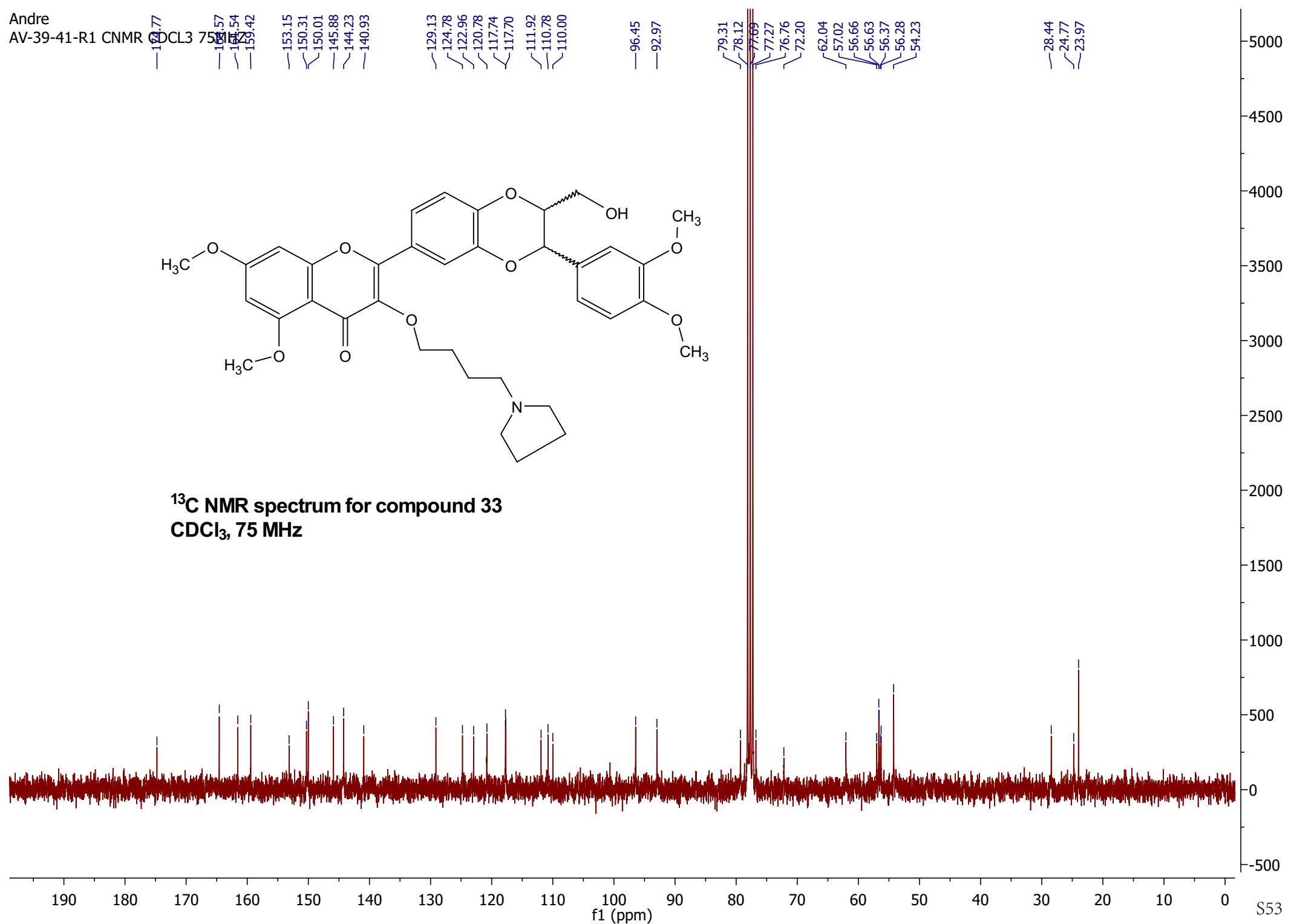
-96.45
-92.97

79.31
78.12
77.69
77.27
76.76
72.20
62.04
57.02
56.66
56.63
56.37
56.28
54.23

-28.44
-24.77
-23.97



¹³C NMR spectrum for compound 33
CDCl₃, 75 MHz



linker nmr
bv-17-36-r2- ccd3

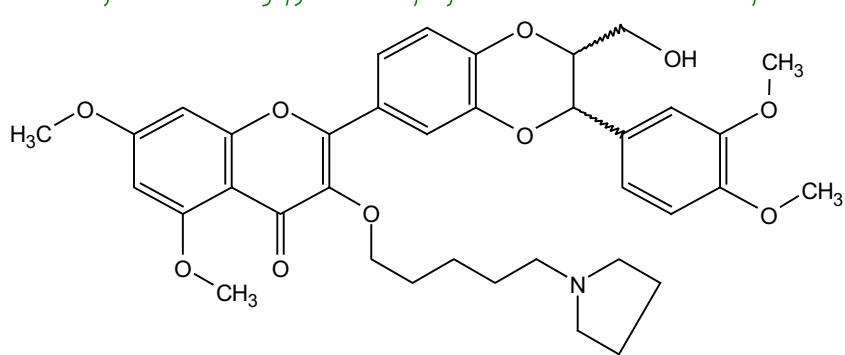
7.7096
7.6854
7.6786

-7.2605

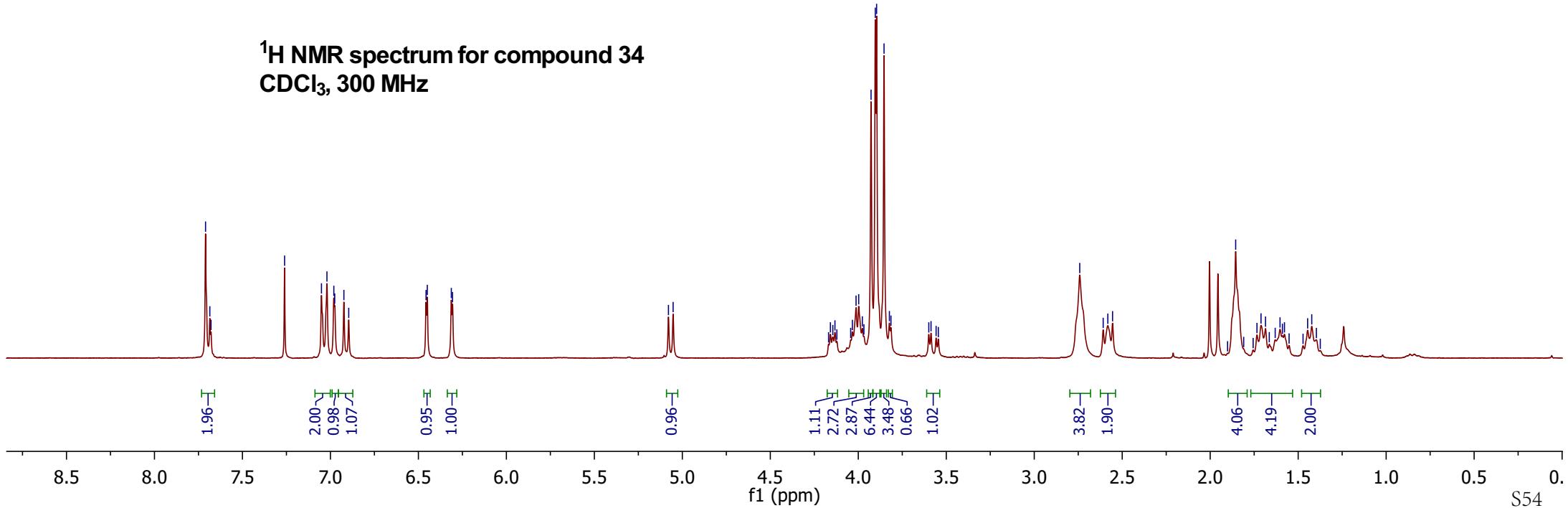
7.0509
7.0198
6.9806
6.9756
6.9234
6.9584
6.4494
6.3129
6.3059

5.0788
5.0523
4.1584
4.1327
4.0335
4.0127
3.9976
3.9770
3.9274
3.9034
3.8959
3.8536
3.8232
3.8144
3.5993
3.5866
3.5577
3.5451

2.7414
2.6082
2.5818
2.5549
1.9012
1.8552
1.8096
1.7559
1.7342
1.7112
1.6856
1.6642
1.6306
1.6032
1.5896
1.5779
1.5517
1.4723
1.4462
1.4228
1.3964
1.3743



¹H NMR spectrum for compound 34
CDCl₃, 300 MHz



linker nmr
bv-17-36-r2- cdd3

-174.09

-163.86
-160.96
-158.77

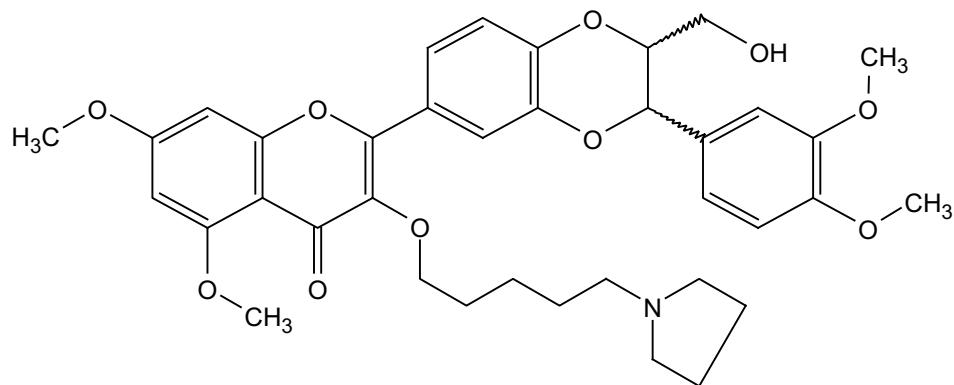
-152.37
-149.66
-149.38
-145.16
-143.52
-140.46

-128.58
-124.27
-122.36
-120.12
-117.22
-116.93

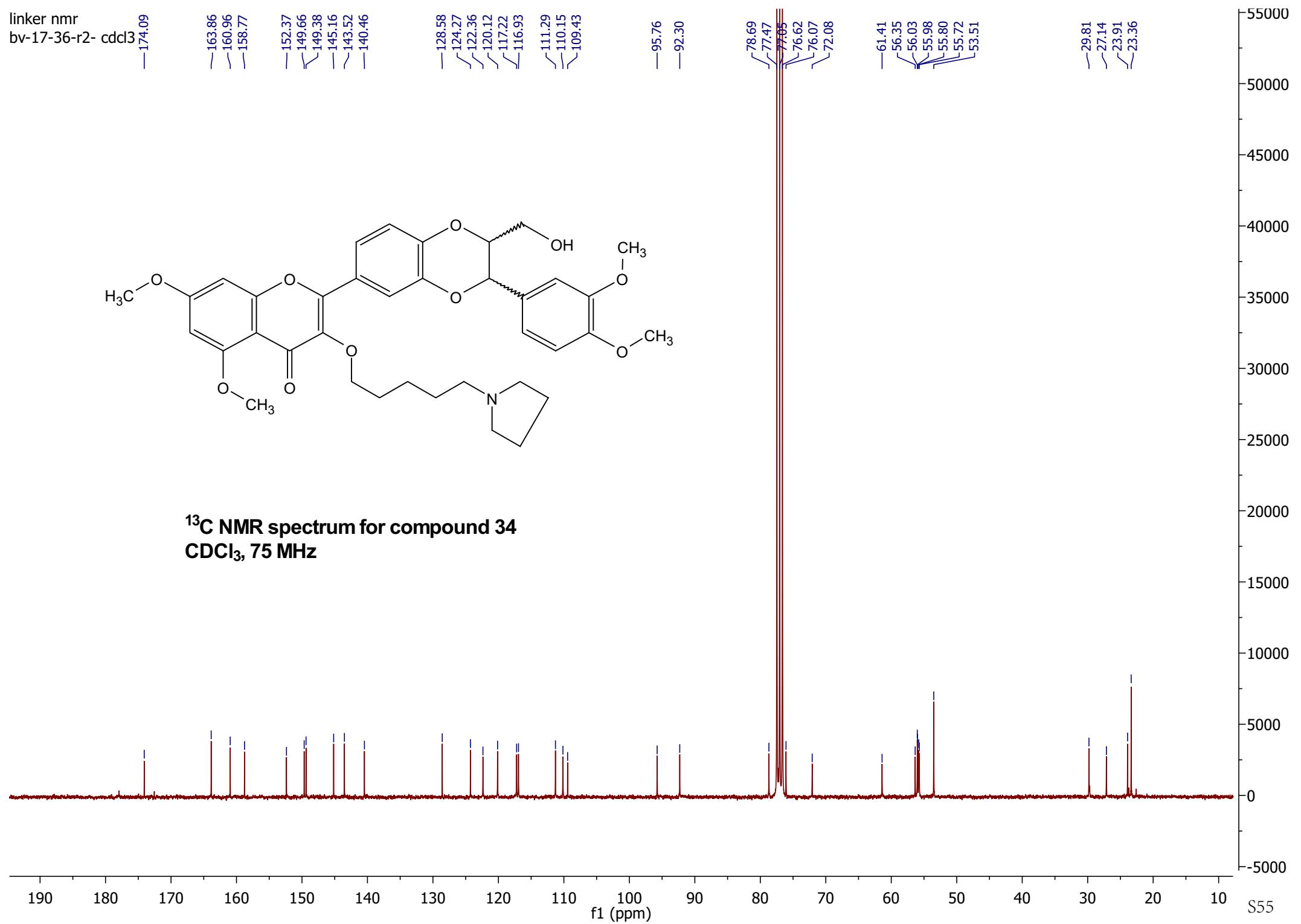
-111.29
-110.15
-109.43

-95.76
-92.30

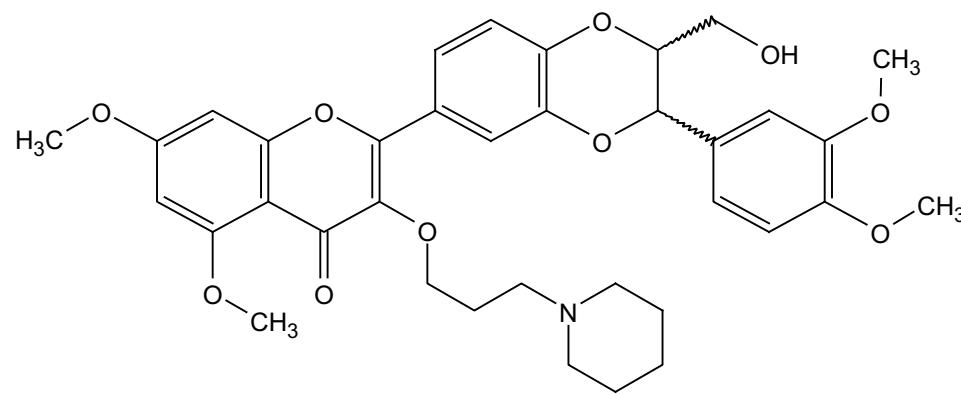
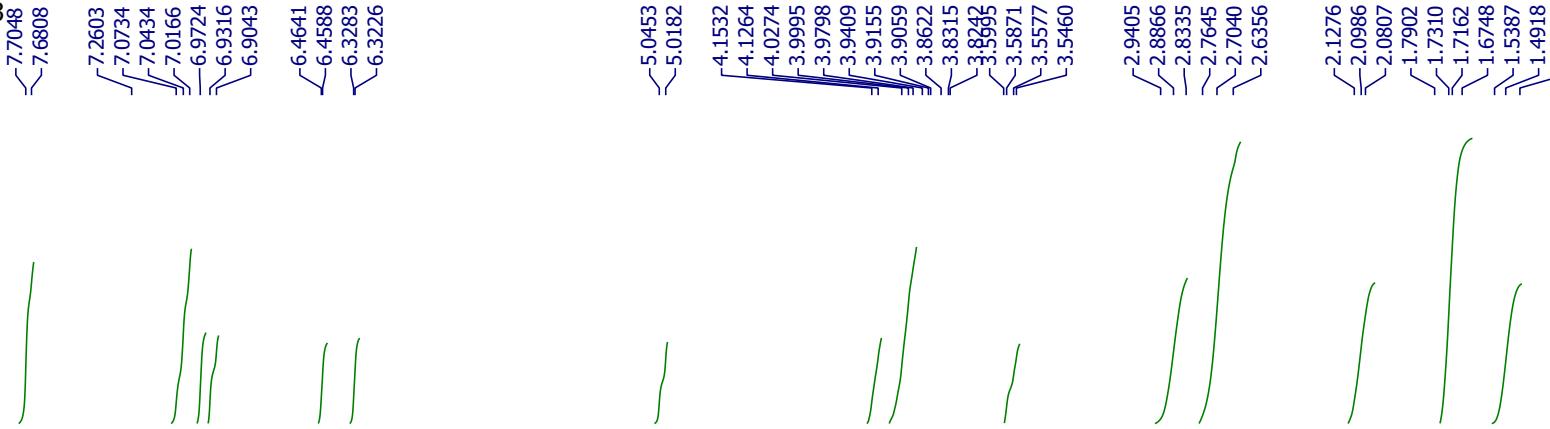
-29.81
-27.14
-23.91
-23.36



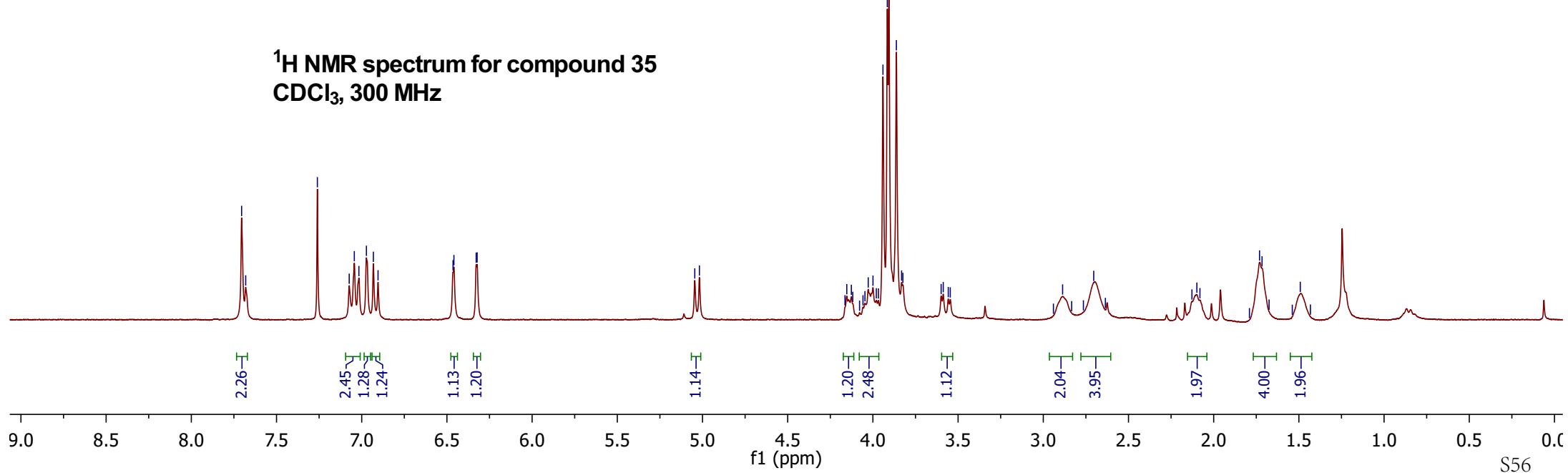
¹³C NMR spectrum for compound 34
CDCl₃, 75 MHz



BV-48-32-R2_CNMR CDCl₃



¹H NMR spectrum for compound 35
CDCl₃, 300 MHz



linker nmr
bv-17-32-r2- cdd3

-174.06

-164.02
-160.93
-158.80

-152.60
-149.76
-149.42
-145.33
-143.68
-140.09

-128.34
-124.04
-122.28
-120.20
-117.15

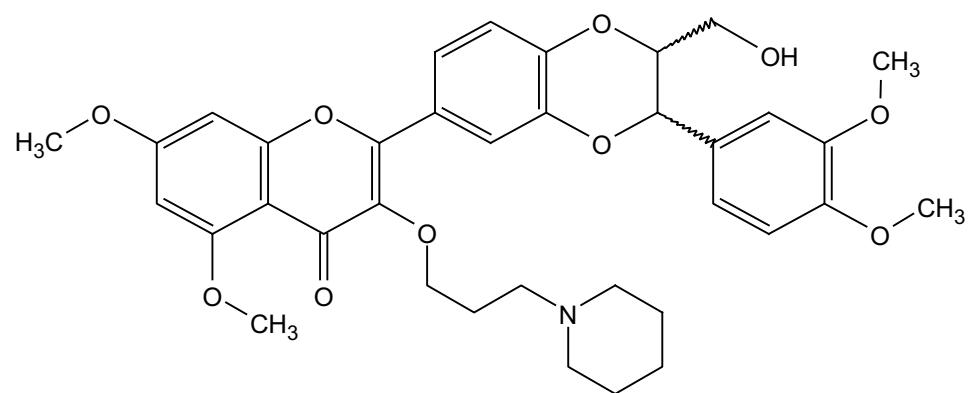
-111.32
-110.18
-109.30

-95.87
-92.36

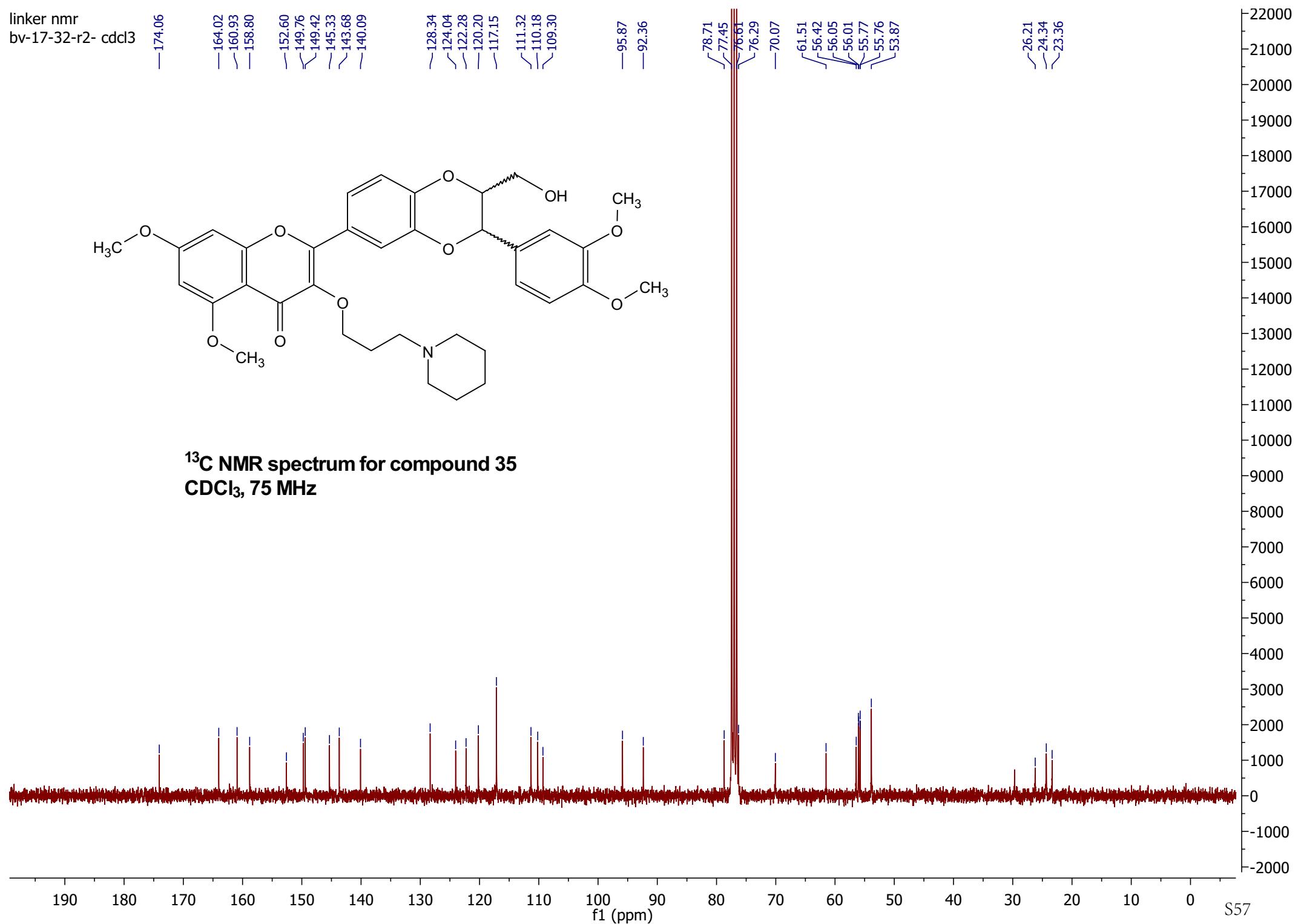
78.71
77.45
76.61
76.29
-70.07

61.51
56.42
56.05
56.01
55.77
55.76
53.87

-26.21
-24.34
-23.36



¹³C NMR spectrum for compound 35
CDCl₃, 75 MHz



Andre

AV-39-42-R1 HNMR CDCl₃ 300 MHz

7.703
7.687
7.687
7.260
7.034
7.004
6.965
6.913
6.886

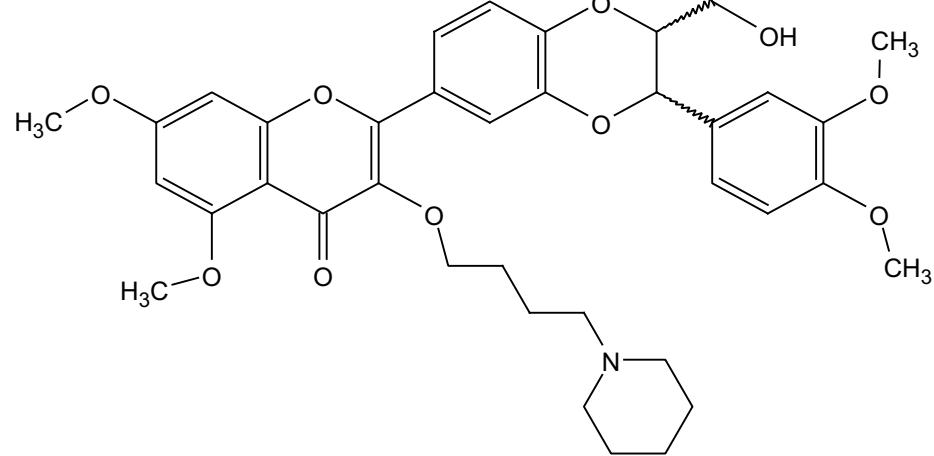
—6.441
—6.303

5.047
5.021

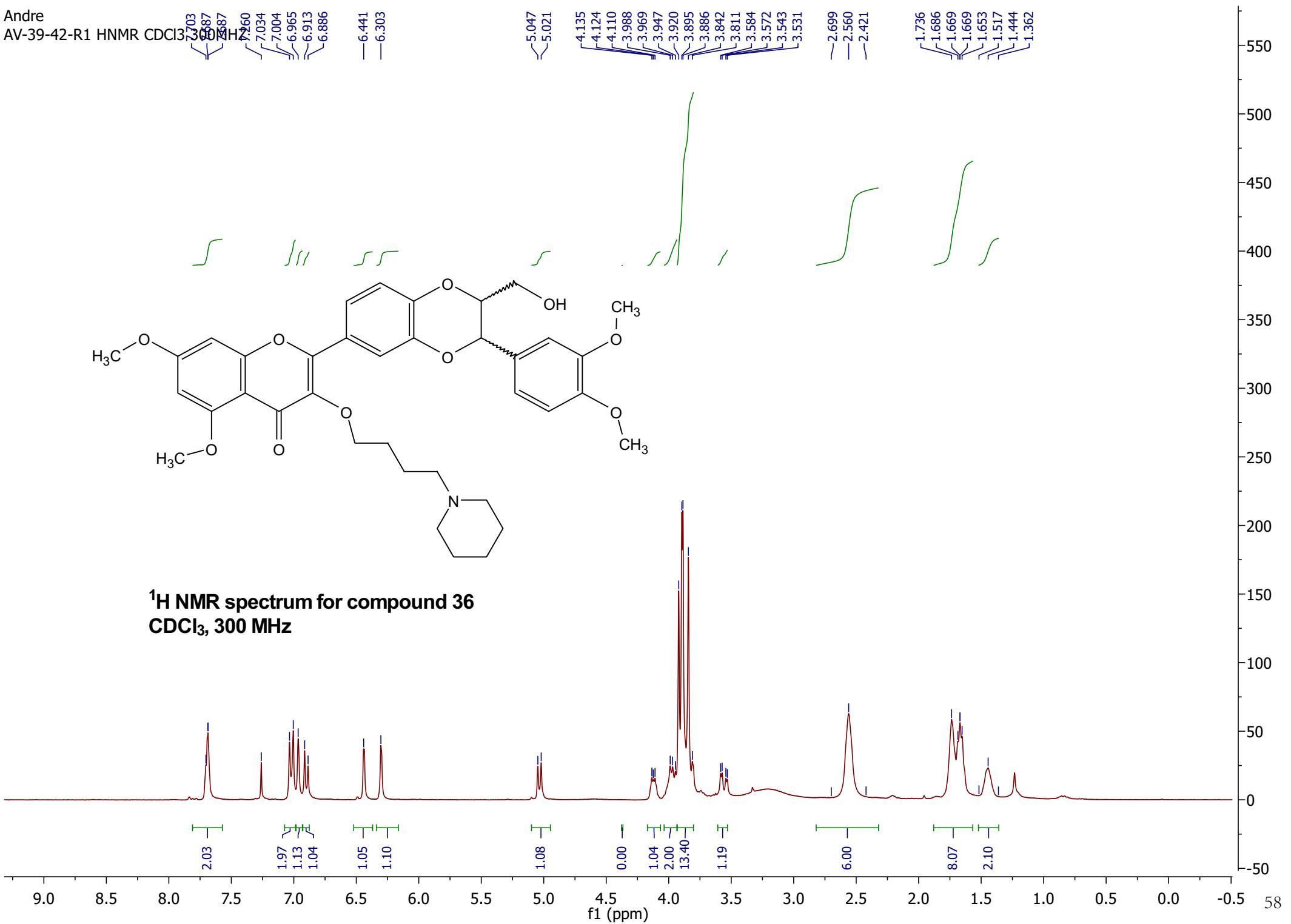
4.135
4.124
4.110
3.988
3.969
3.947
3.920
3.895
3.886
3.842
3.811
3.584
3.572
3.543
3.531

—2.699
—2.560
—2.421

1.736
1.686
1.669
1.669
1.653
1.517
1.444
1.362



¹H NMR spectrum for compound 36
CDCl₃, 300 MHz



Andre

AV-39-42-R1 CNMR CDCl₃ 75MHz

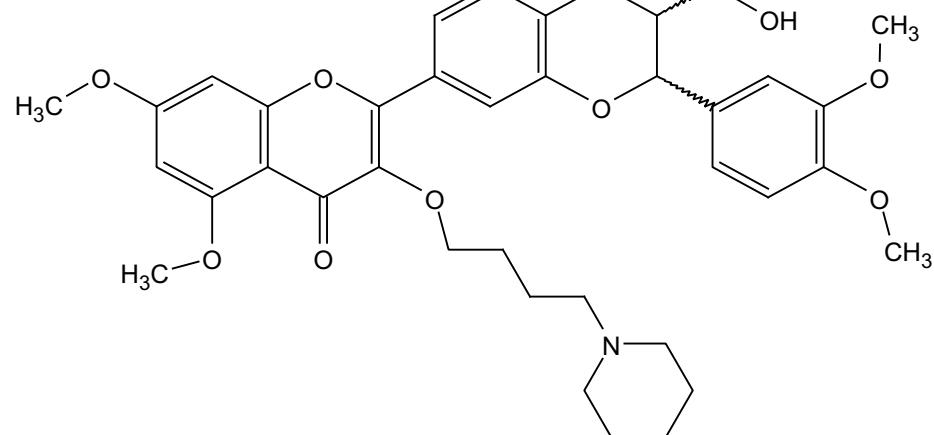
—171.78
—165.55
—164.53
—159.39
—153.04
—150.31
—150.00
—145.87
—144.23
—140.96

—129.11
—124.80
—122.95
—120.80
—117.71
—117.65
—111.92
—110.78
—109.97

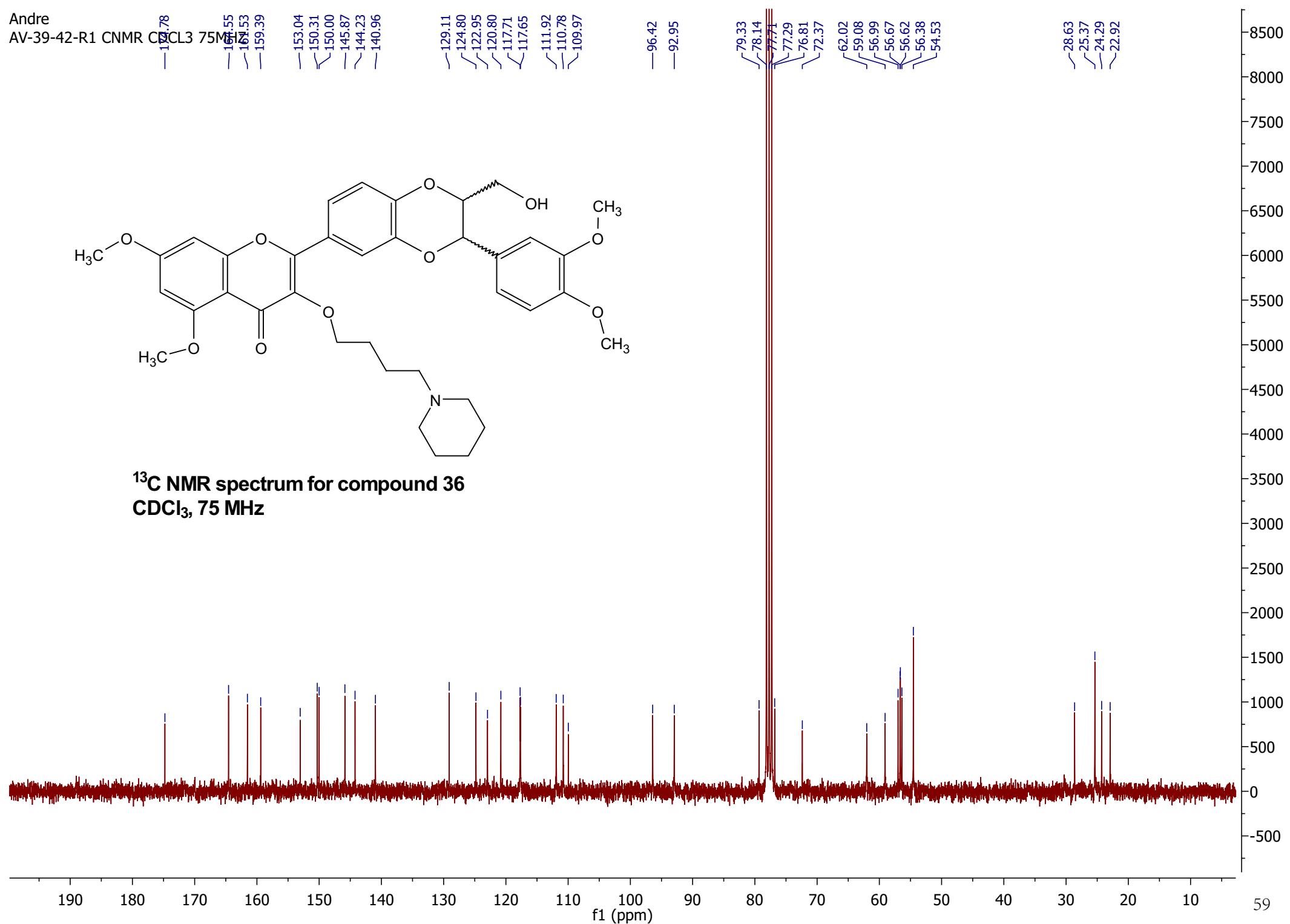
—96.42
—92.95

—79.33
—78.14
—77.71
—77.29
—76.81
—72.37
—62.02
—59.08
—56.99
—56.67
—56.62
—56.38
—54.53

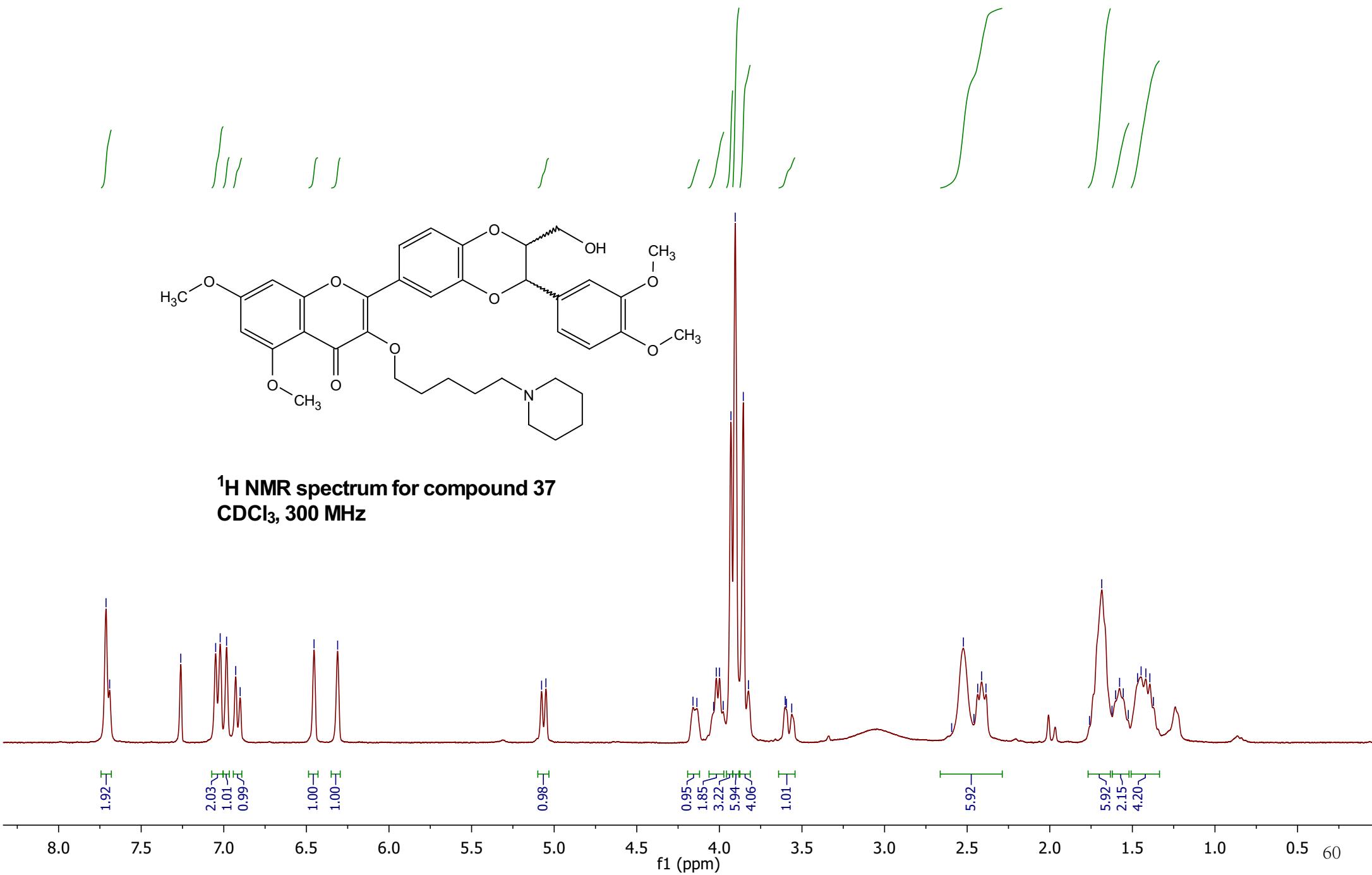
—28.63
—25.37
—24.29
—22.92



¹³C NMR spectrum for compound 36
CDCl₃, 75 MHz



linker nmr
BV-48-37-r2  7127 7909



E:
BV-48-37-r2

CDCl₃

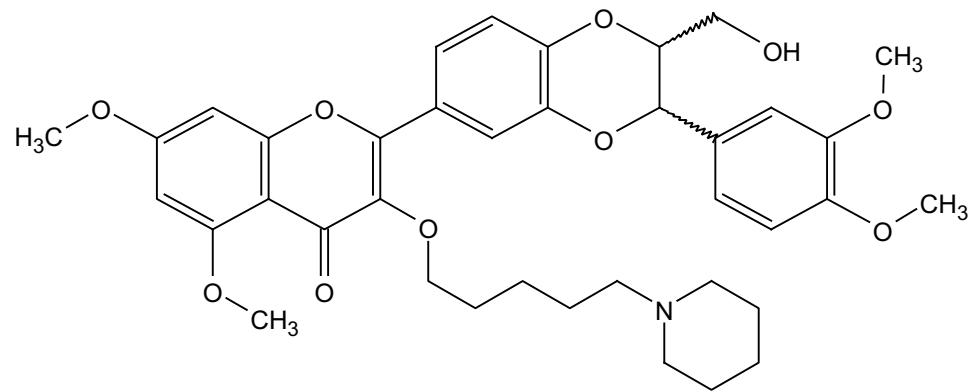
—164.02
—161.13
—158.93
—152.49
—149.85
—149.56
—145.28
—143.69
—140.64

—128.69
—124.49
—122.55
—120.29
—117.38
—117.07
—111.47
—110.33
—109.60

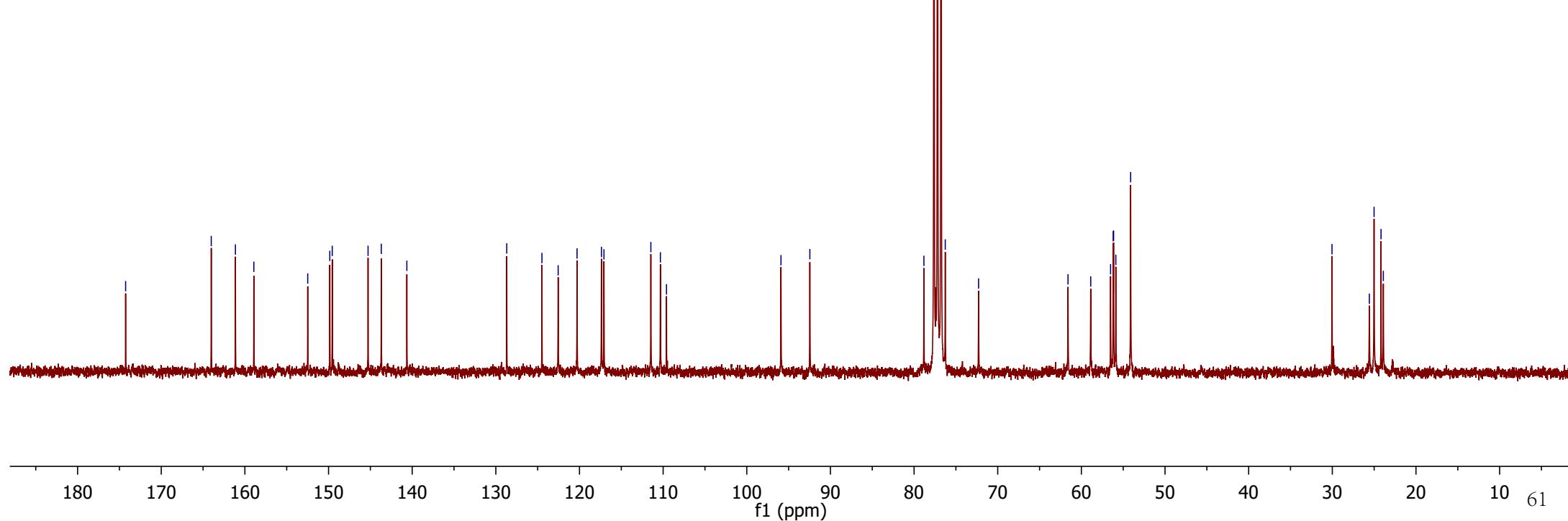
—95.91
—92.46

—78.82
—77.62
—77.26
—76.78
—76.27
—72.29
—61.60
—58.88
—56.51
—56.20
—56.15
—55.88
—54.11

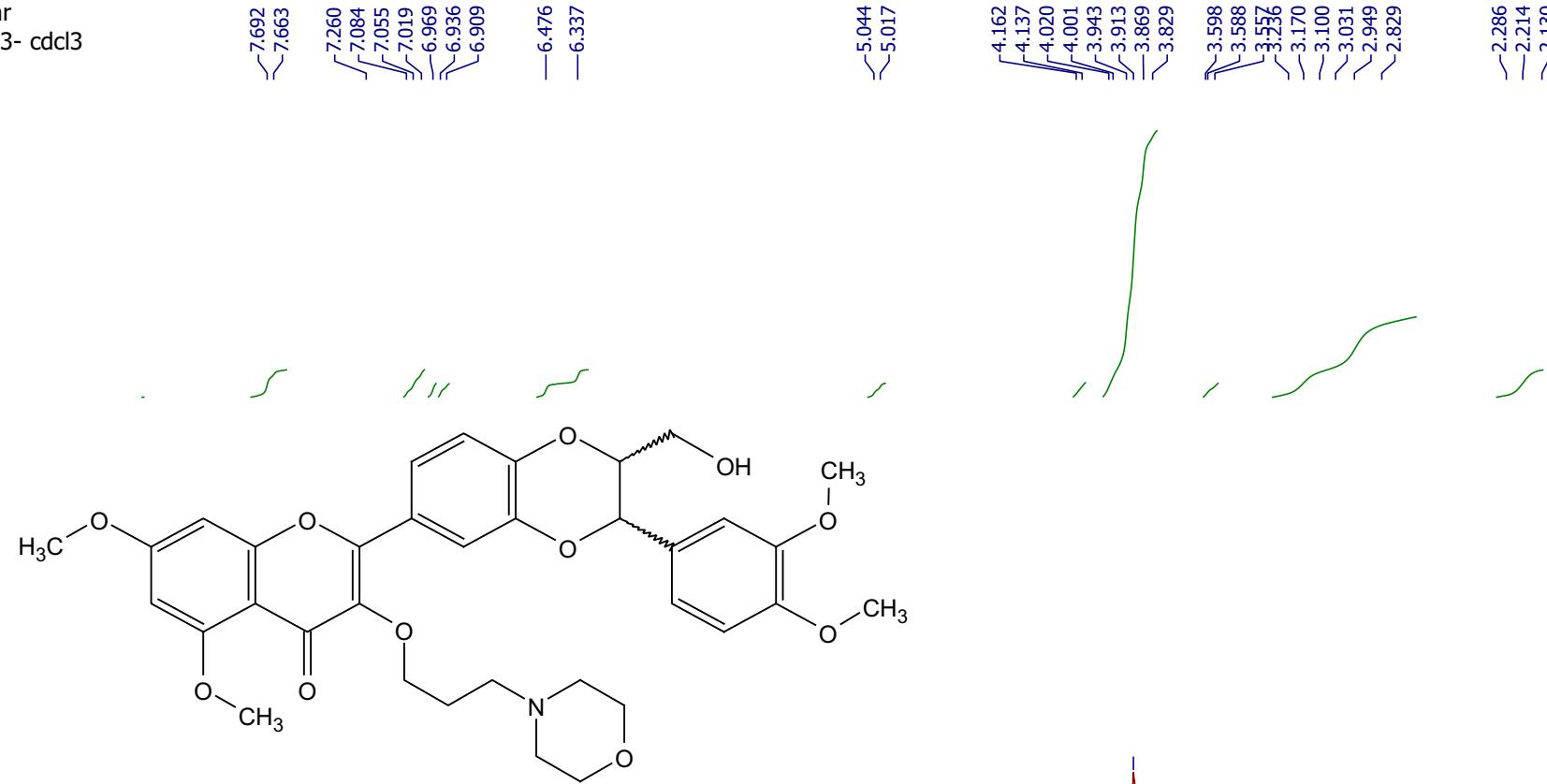
—30.04
—25.57
—25.01
—24.18
—23.90



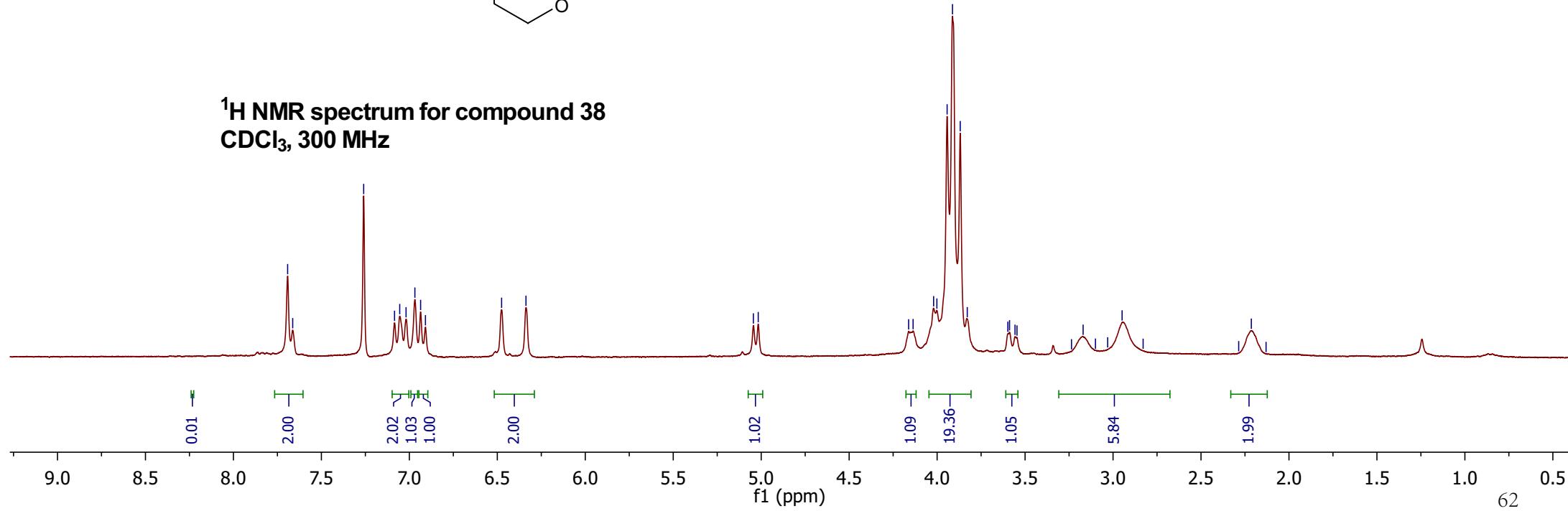
¹³C NMR spectrum for compound 37
CDCl₃, 75 MHz



linker nmr
bv-17-243- cdcl3

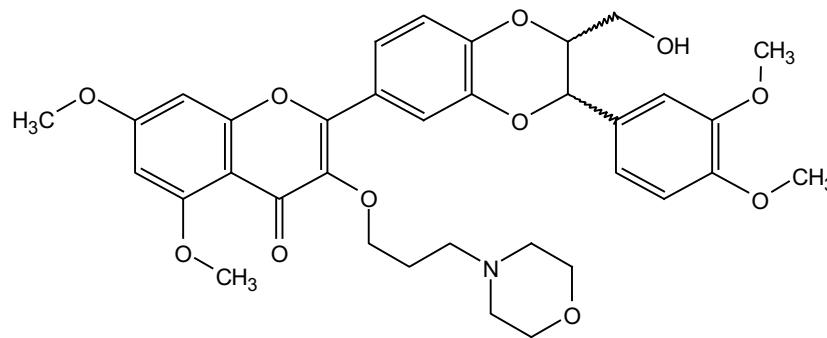


¹H NMR spectrum for compound 38
CDCl₃, 300 MHz

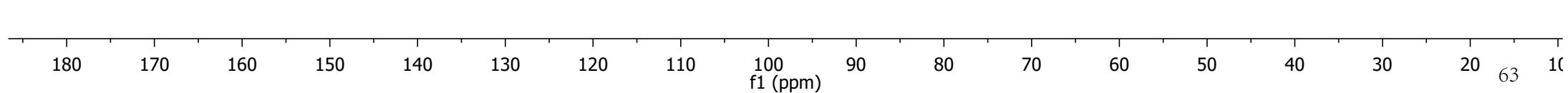


linker nmr
bv-17-243- CDCl₃

—124.24
~164.34
~161.06
~159.00
~152.97
~149.99
~149.61
~145.64
~143.91
~140.06
—128.36
~124.02
~122.37
~120.37
~117.43
~117.31
~111.51
~110.37
~109.36
—96.14
—92.59
—69.57
—64.96
—61.69
~56.63
~56.24
~56.17
~56.15
~55.96
~52.85
—25.57

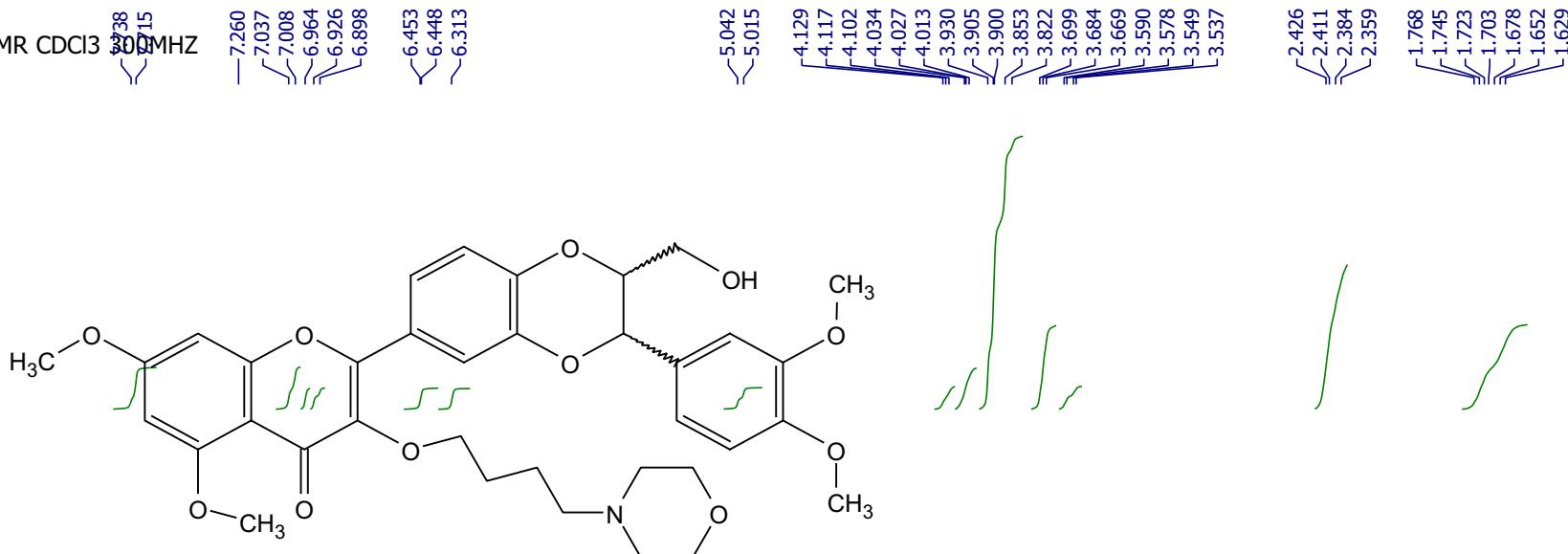


¹³C NMR spectrum for compound 38
CDCl₃, 300 MHz

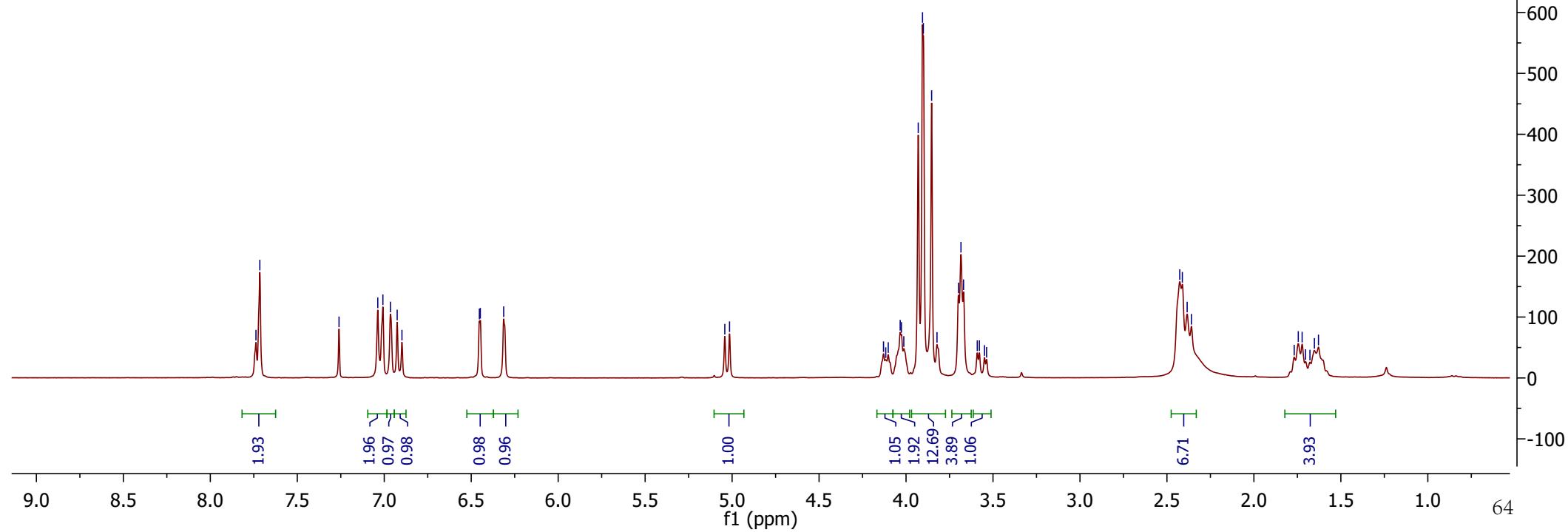


Andre

AV-39-40-R1 HNMR CDCl₃



¹H NMR spectrum for compound 39
CDCl₃, 300 MHz



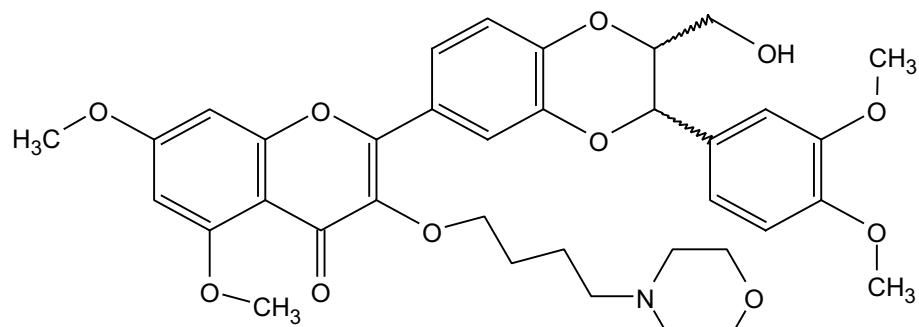
Andre

AV-39-40-R1 CMR CDCl₃, 75 MHz

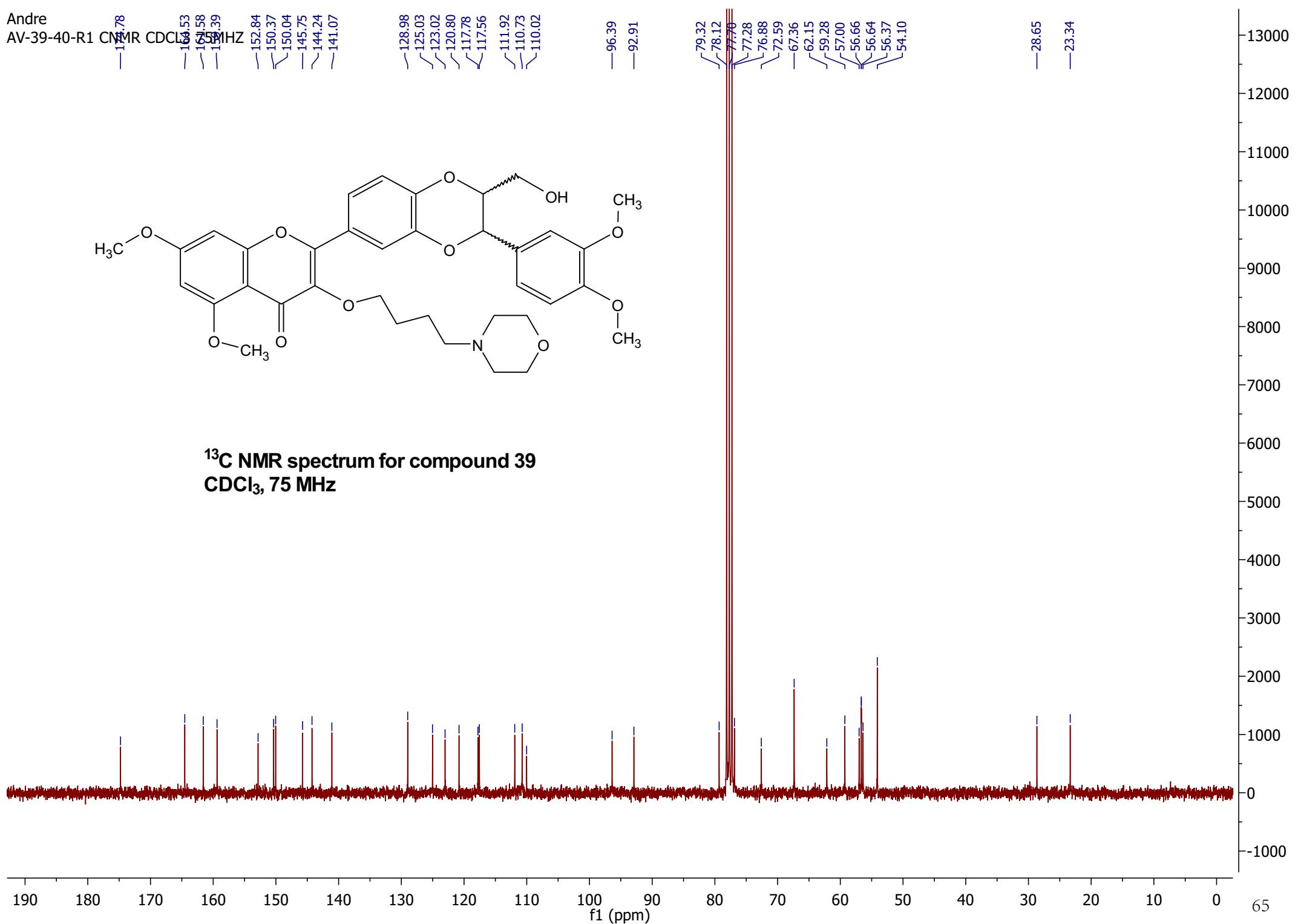
-117.78
-164.53
-167.58
-175.39
152.84
150.37
150.04
145.75
144.24
141.07

-128.98
-125.03
-123.02
-120.80
-117.78
-117.56
-111.92
-110.73
-110.02

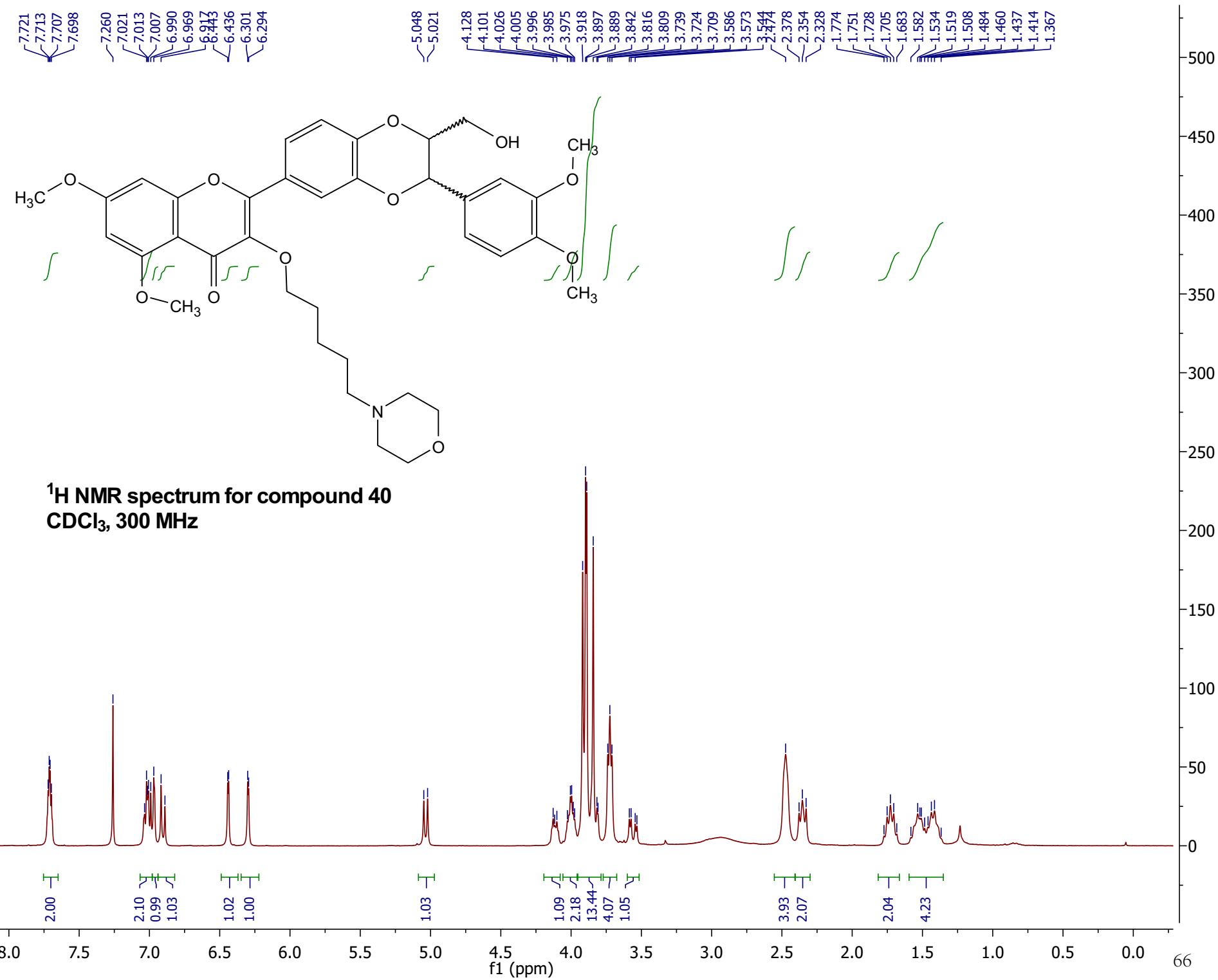
-96.39
-92.91



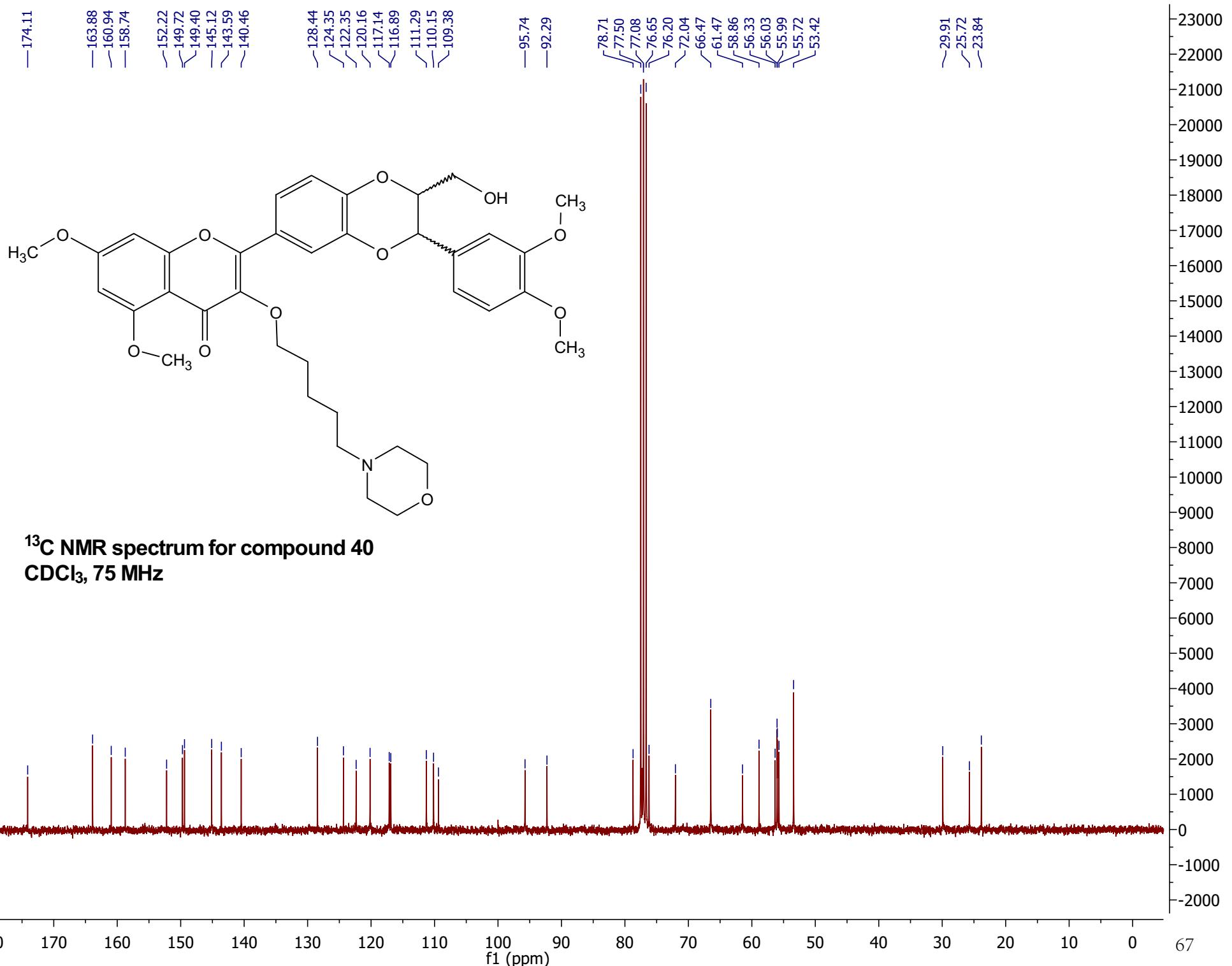
¹³C NMR spectrum for compound 39
CDCl₃, 75 MHz



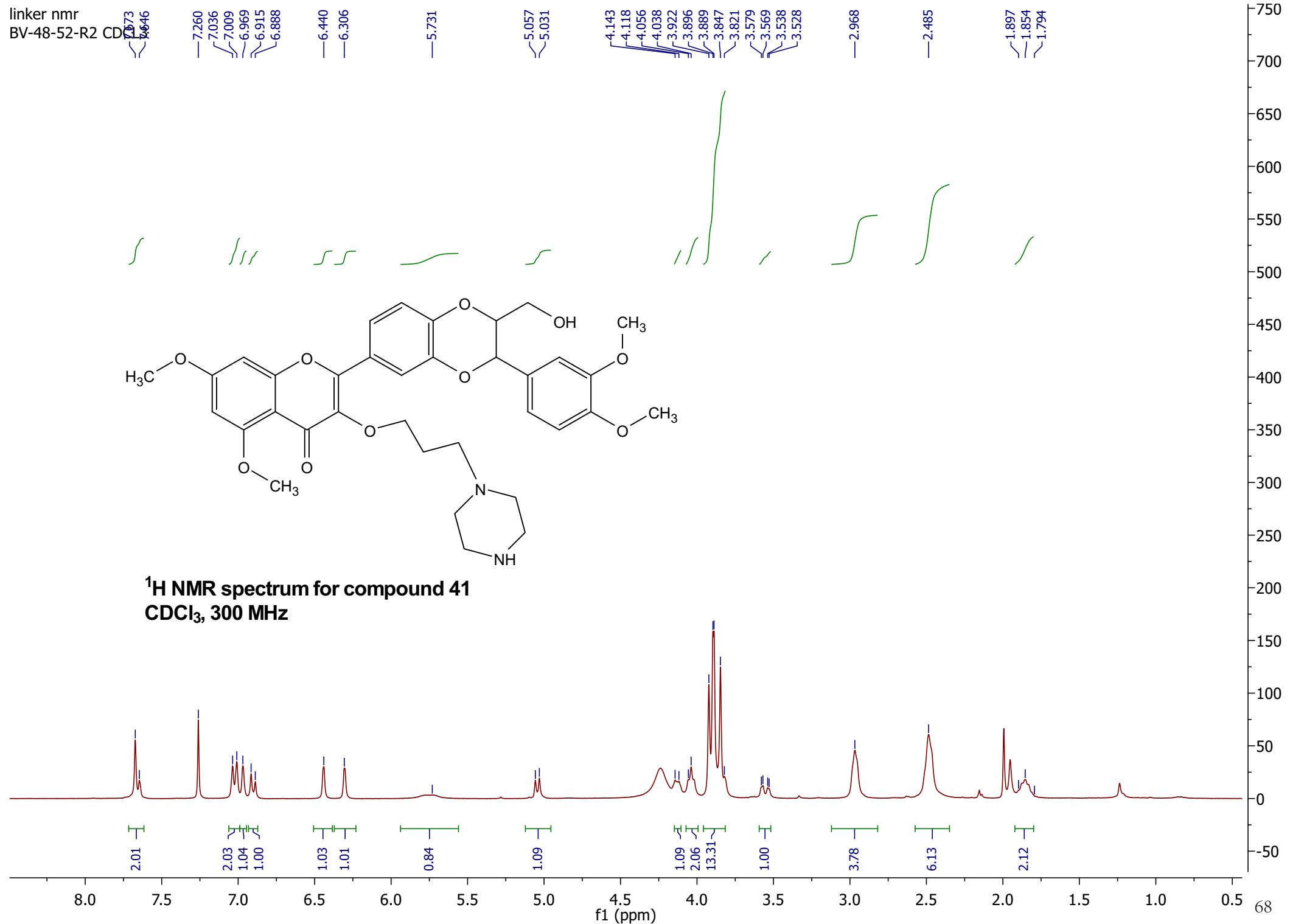
andre vignau
av 37-31



andre vignau
av 37-31 c13



linker nmr
BV-48-52-R2



linker nmr
BV-48-52-R24.07
CDCl₃

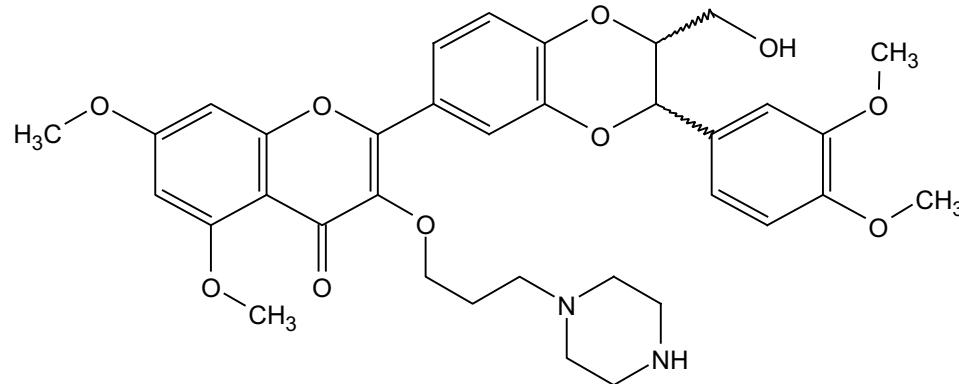
-163.93
-160.93
-158.78
-152.52
-149.65
-149.34
-145.27
-143.54
-140.31

-128.51
-124.14
-122.45
-120.16
-117.31
-116.94
-111.27
-110.18
-109.35

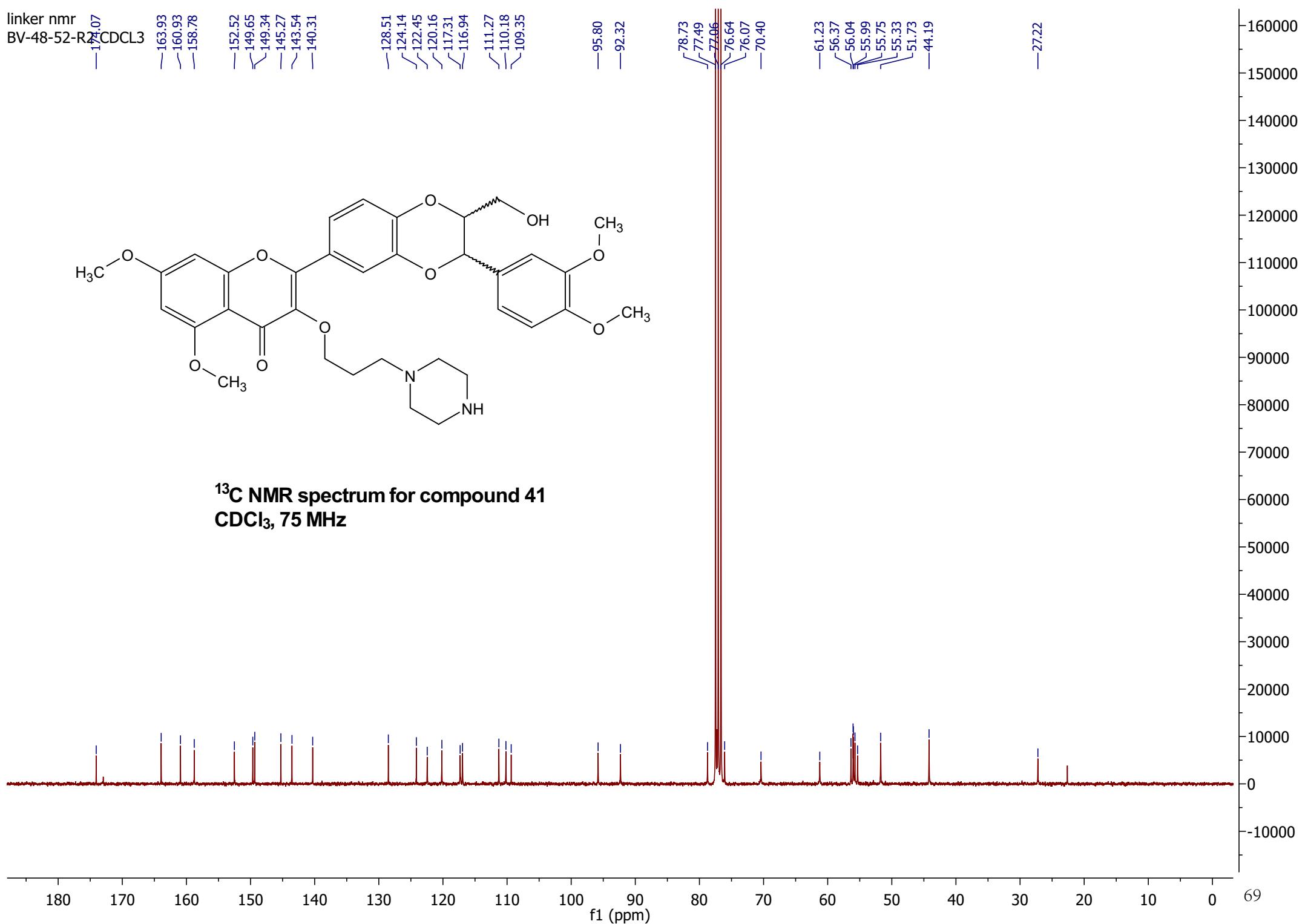
-95.80
-92.32

-61.23
-56.37
-56.04
-55.99
-55.75
-55.33
-51.73
-44.19

-27.22

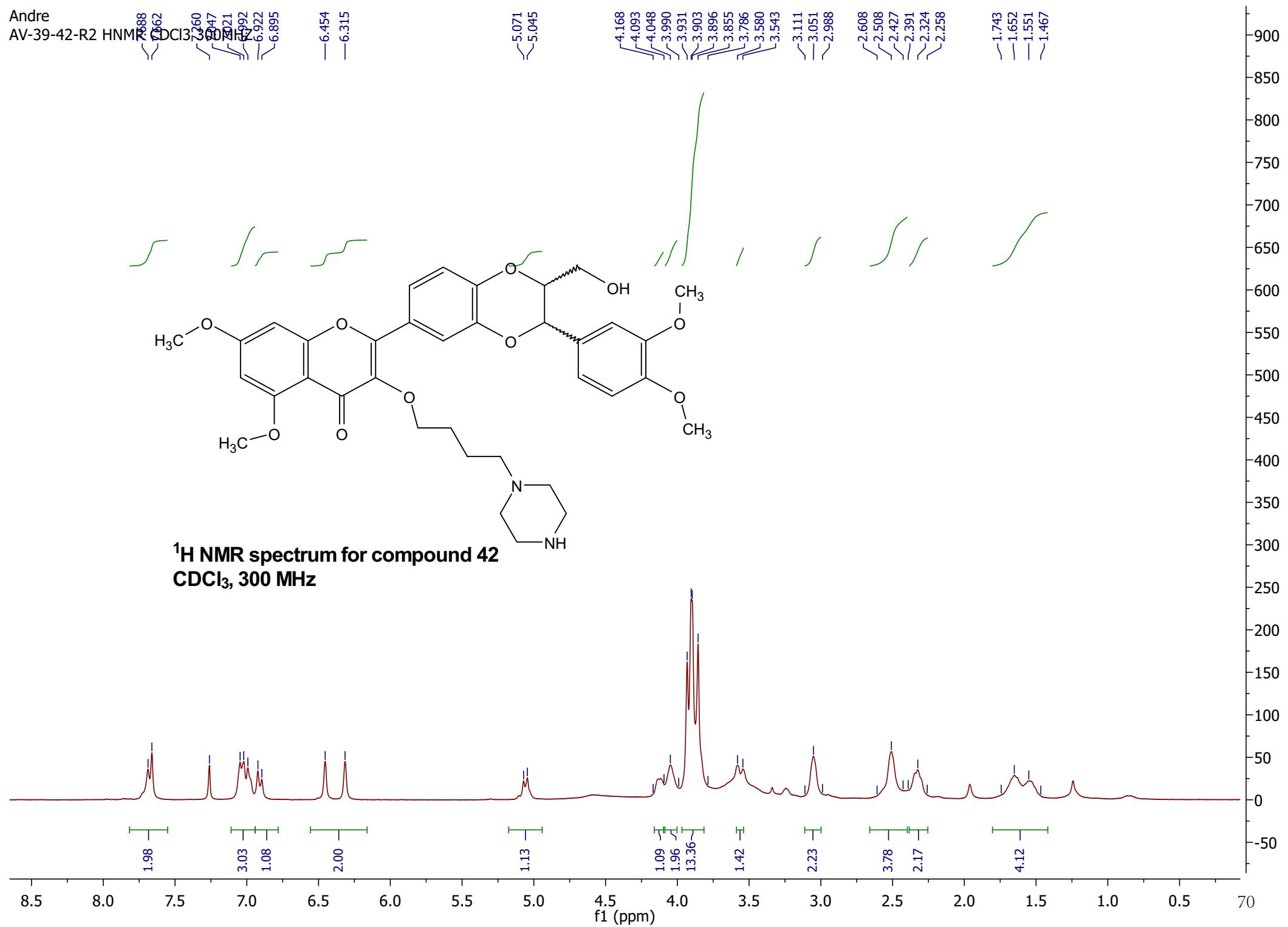


¹³C NMR spectrum for compound 41
CDCl₃, 75 MHz



Andre

AV-39-42-R2 HNMR CDCl₃ 300 MHz



Andre

AV-39-42-R2 CNMR CDCl₃ 75MHz

-171.79
-167.54
-161.59
-159.44

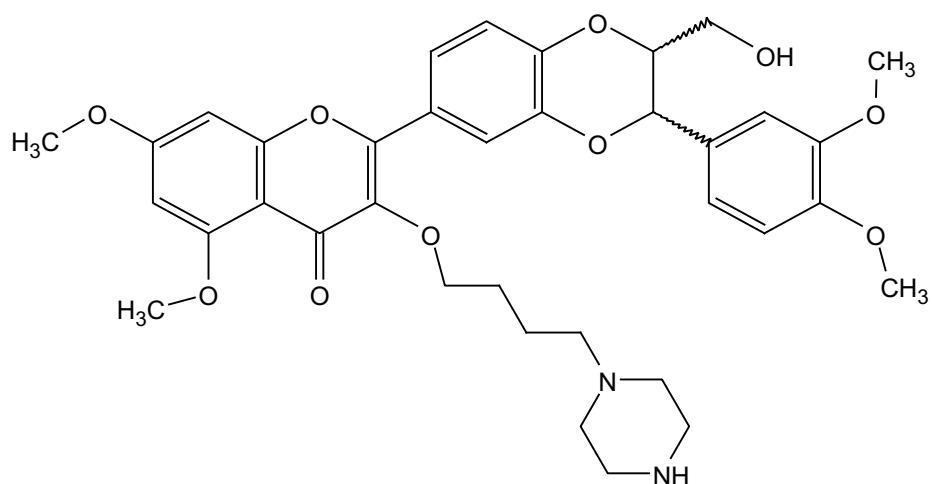
-153.19
-150.29
-149.99
-145.84
-144.19
-140.99

-129.14
-124.90
-123.28
-120.83
-117.91
-117.53
-111.90
-110.81
-110.04

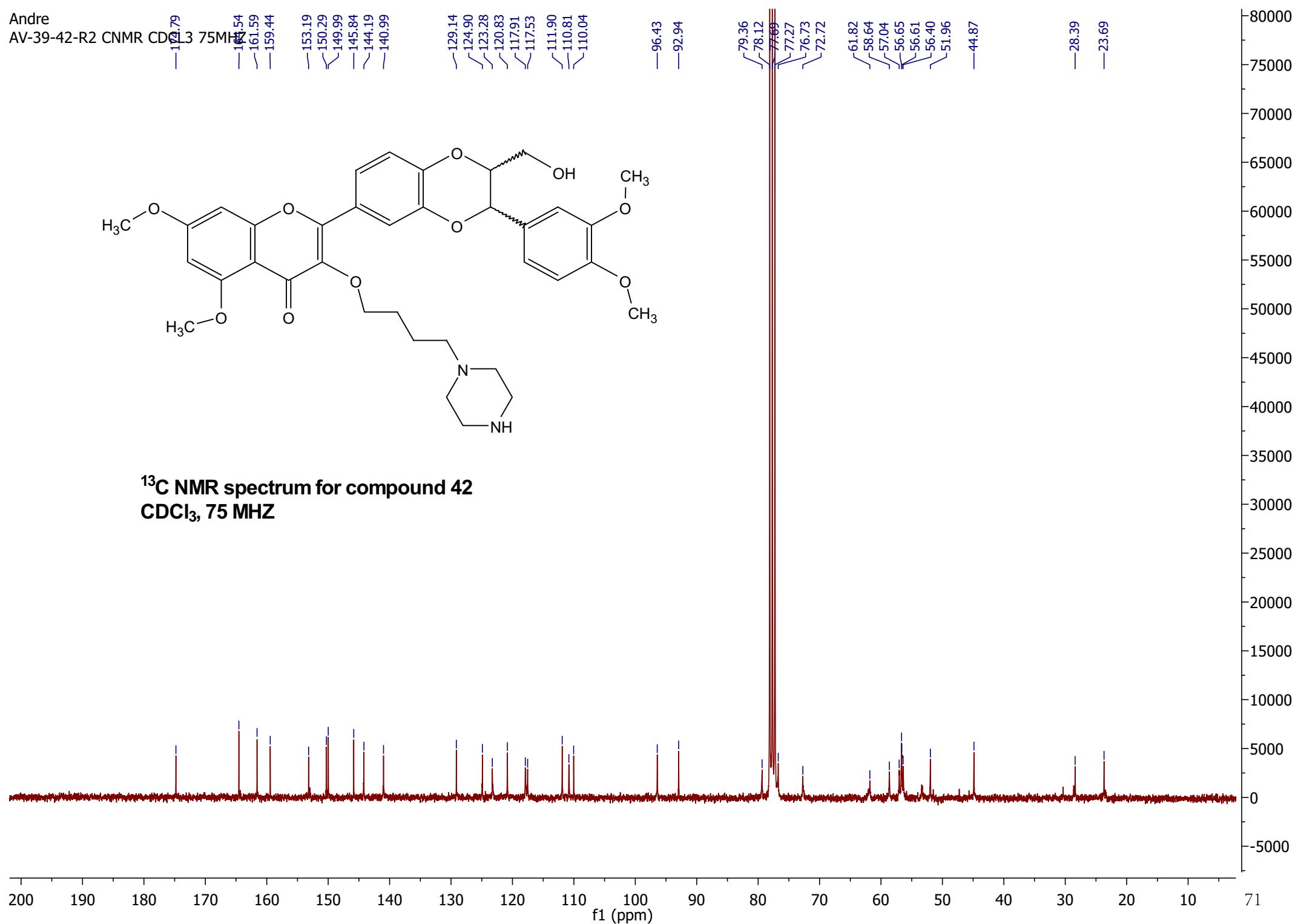
-96.43
-92.94

79.36
78.12
77.69
77.27
76.73
72.72
61.82
58.64
57.04
56.65
56.61
56.40
51.96
44.87

-28.39
-23.69

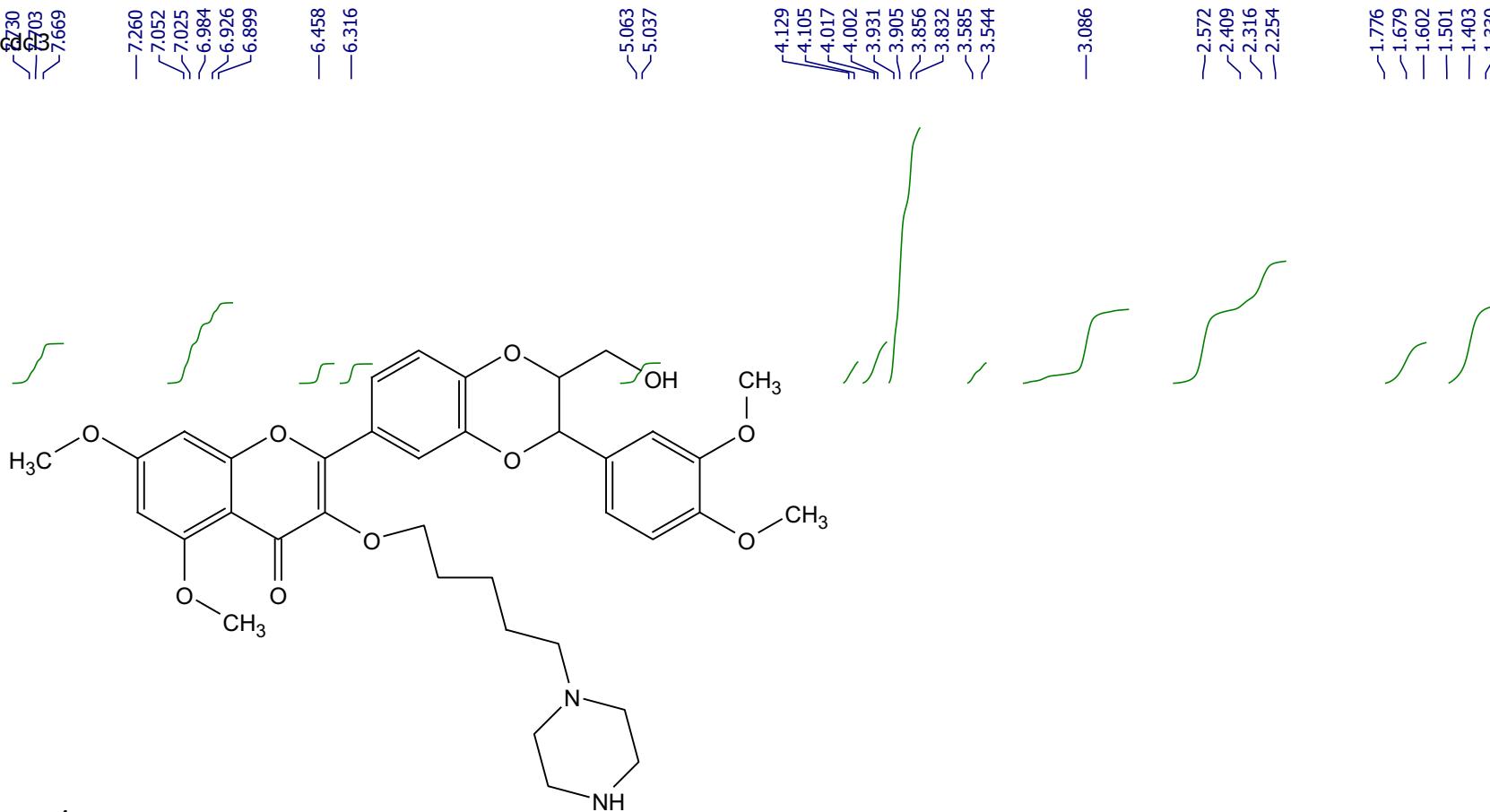


¹³C NMR spectrum for compound 42
CDCl₃, 75 MHz



linker nmr

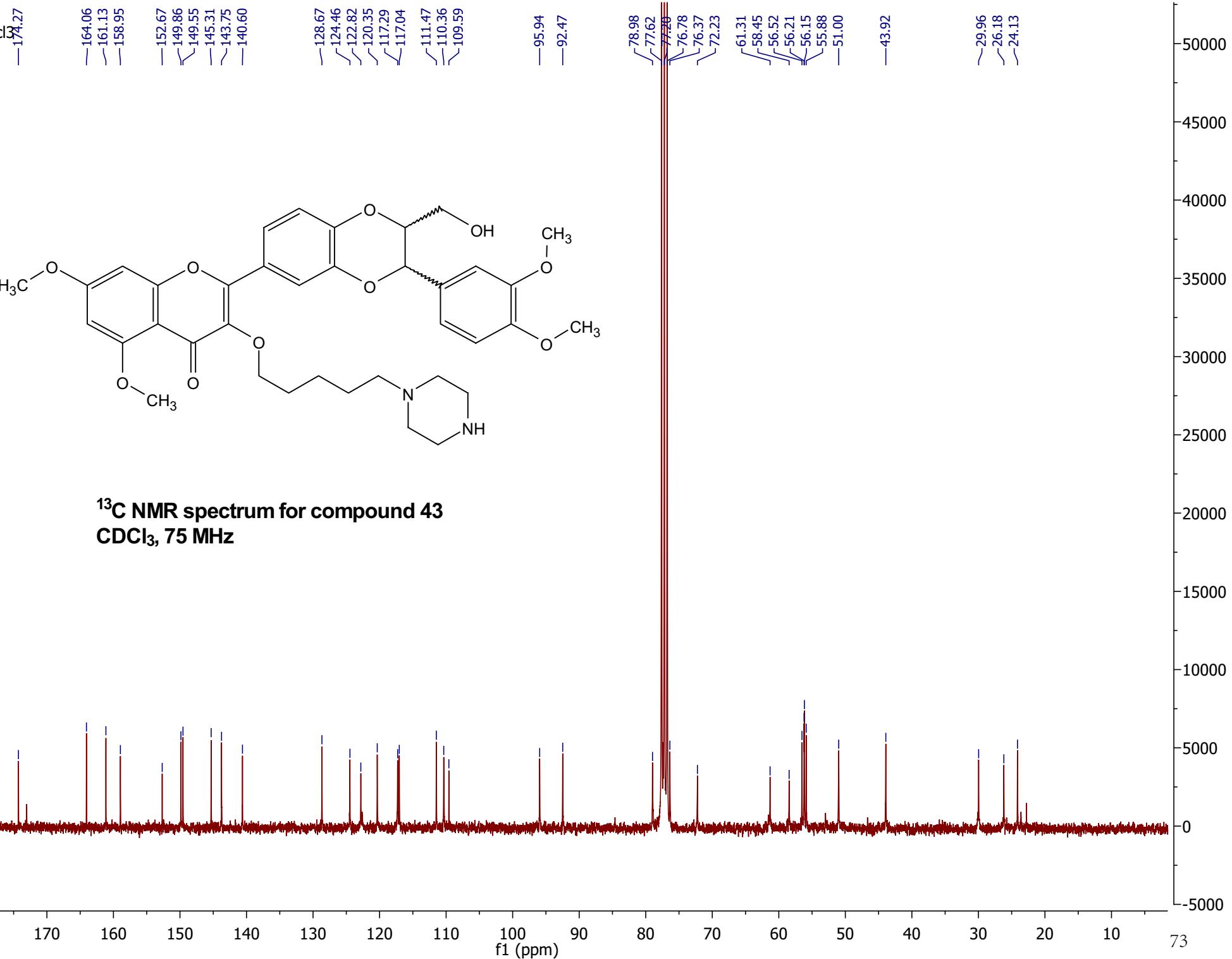
BV-48-38-r1 cdd3



**¹H NMR spectrum for compound 43
CDCl₃, 300 MHz**

linker nmr

BV-48-38-r1 cdcl₃



¹³C NMR spectrum for compound 43
CDCl₃, 75 MHz

linker nmr

BV-48-32-r1
δ7.02
δ6.73
δ6.666

7.260
7.048
7.020
6.973
6.968
6.928
6.900

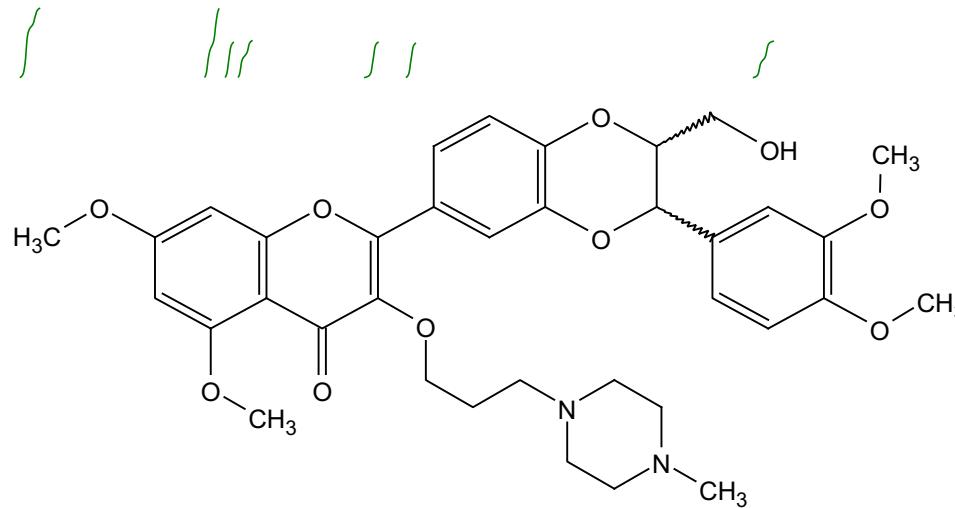
6.453
6.446
6.316
6.309

5.056
5.029

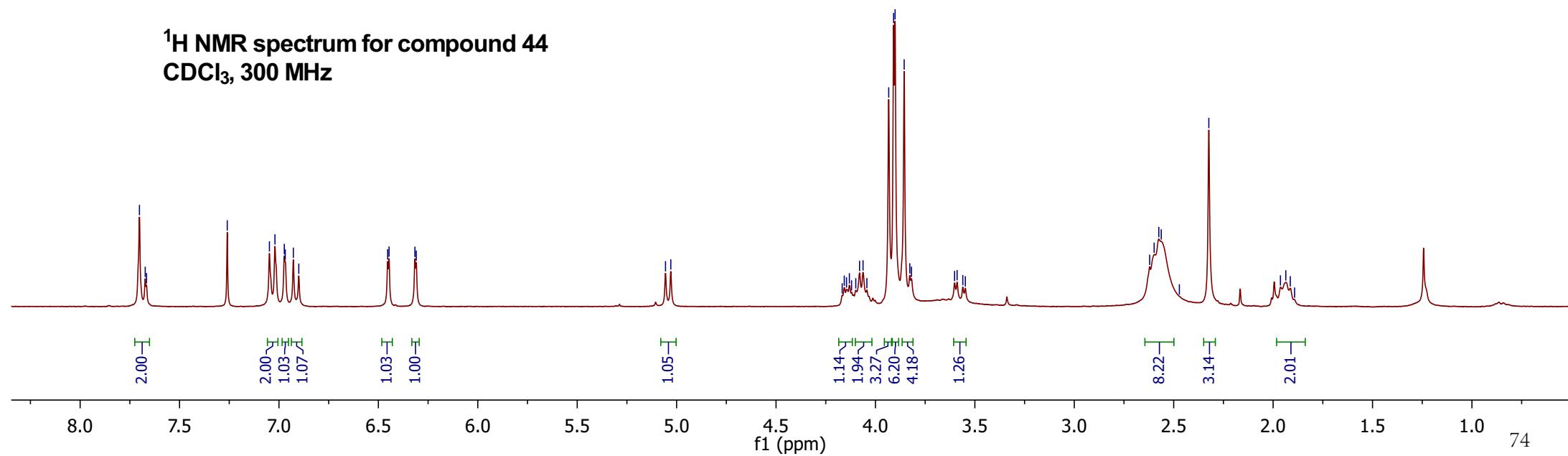
4.157
4.146
4.131
4.099
4.080
4.062
4.043
3.933
3.909
3.901
3.855
3.827
3.819
3.589
3.561
3.548

2.621
2.598
2.575
2.563
2.471
2.323

1.963
1.936
1.914
1.892

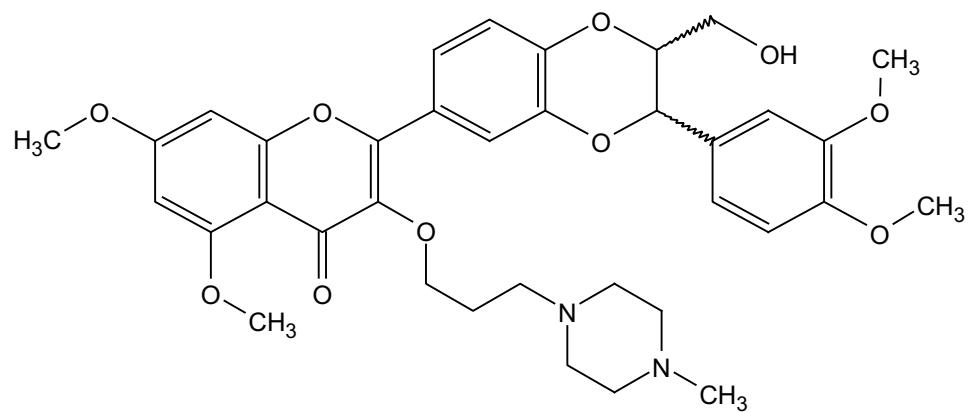


¹H NMR spectrum for compound 44
CDCl₃, 300 MHz

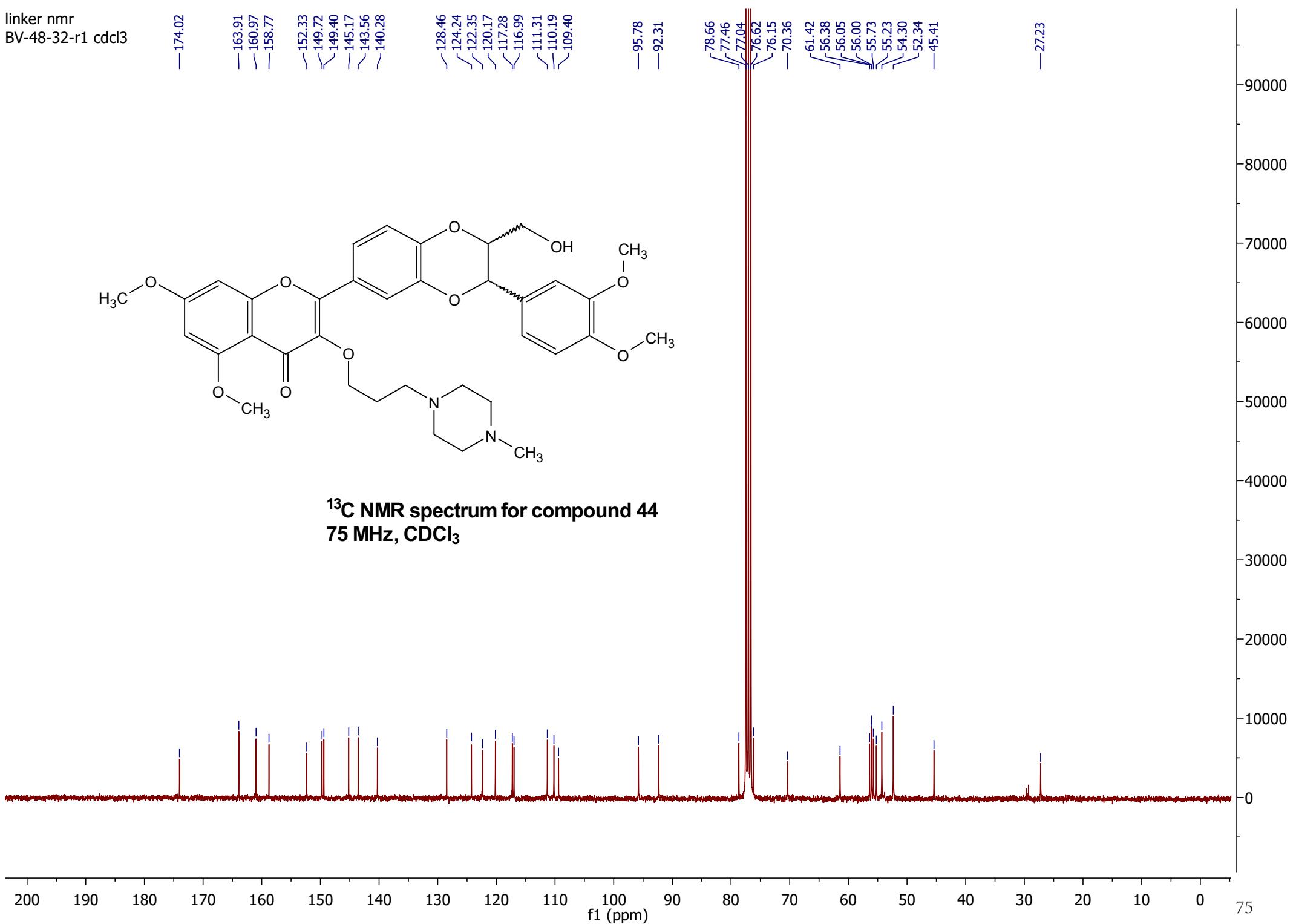


linker nmr
BV-48-32-r1 cdcl₃

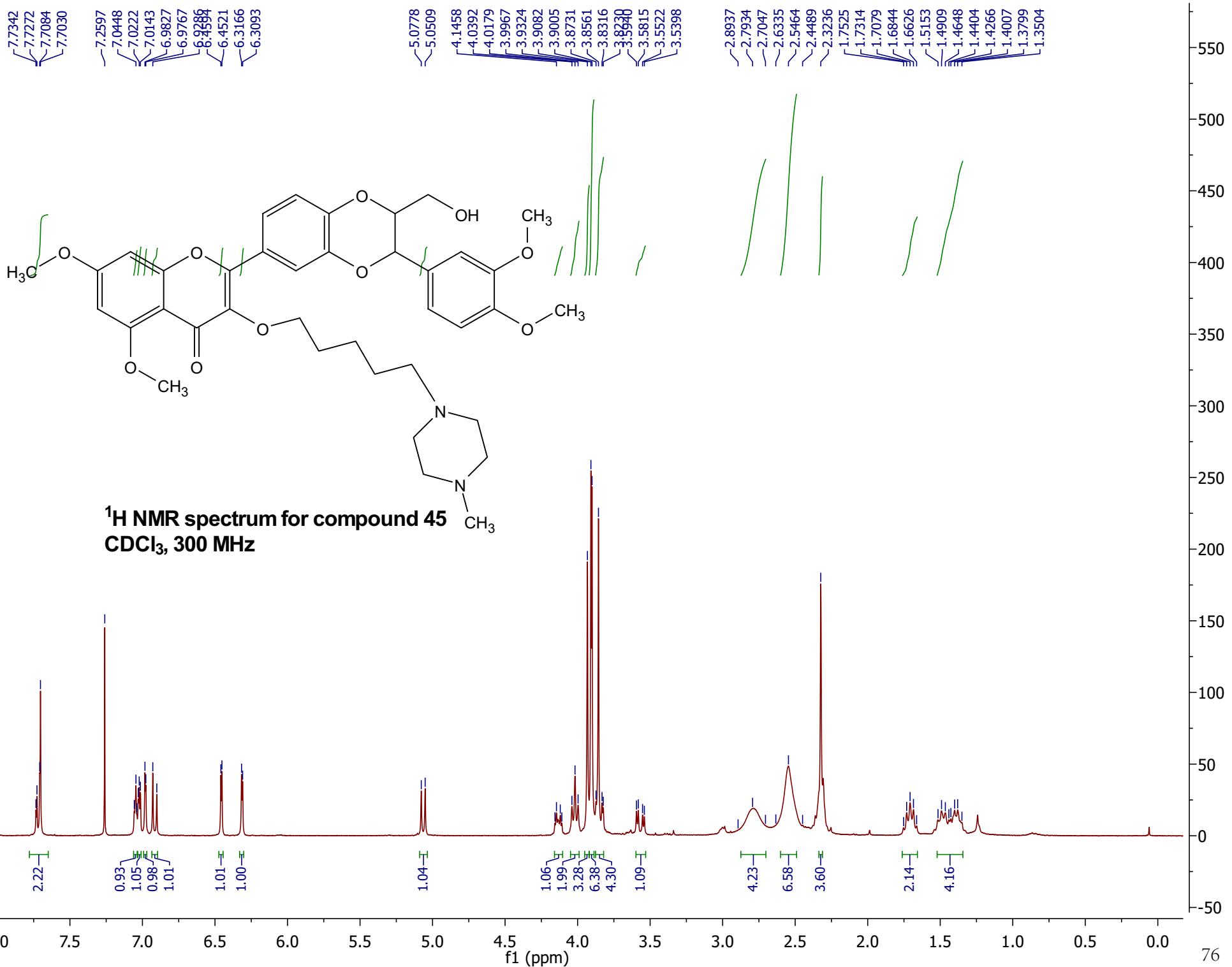
-174.02
-163.91
-160.97
-158.77
-152.33
-149.72
-149.40
-145.17
-143.56
-140.28
-128.46
-124.24
-122.35
-120.17
-117.28
-116.99
-111.31
-110.19
-109.40
-95.78
-92.31
-78.66
-77.46
-77.04
-76.62
-76.15
-70.36
-61.42
-56.38
-56.05
-56.00
-55.73
-55.23
-54.30
-52.34
-45.41
-27.23



¹³C NMR spectrum for compound 44
75 MHz, CDCl₃



bv-48-92



bv-48-92

