Sustainable Protocol for the Synthesis of 2',3'-Dideoxynucleoside and 2',3'-Didehydro-2',3'-dideoxynucleoside Derivatives

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5′-*O*-Levulinyl-β-D-uridine (**2a**). ^{1}H-NMR (300.13 MHz, $CDCl_3$): δ 2.18 (s, 3H, *Me*-Lev), 2.58 (t, 2H, C*H*$_2$-Lev, *J* = 6.2 Hz), 2.78 (t, 2H, C*H*$_2$-Lev, *J* = 6.0 Hz), 4.19 (m, 1H, H$_{3'}$), 4.27 (m, 2H, H$_{2'}$ + H$_{4'}$), 4.36 (m, 2H, H$_{5'}$), 5.78 (d, 1H, H$_5$, *J* = 8.1 Hz), 5.82 (d, 1H, H$_{1'}$, *J* = 3.6 Hz), 7.66 (d, 1H, H$_6$, *J* = 8.1 Hz) ppm. ^{13}C-NMR (75.5 MHz, $CDCl_3$): δ 28.0 (*C*H$_2$-Lev), 29.9 (*C*H$_3$-Lev), 38.0 (*C*H$_2$-Lev), 63.6 (C$_{5'}$), 70.3 (C$_{3'}$), 75.0 (C$_{2'}$), 82.3 (C$_{4'}$), 90.6 (C$_{1'}$), 102.7 (C$_5$), 140.5 (C$_6$), 151.4 (C$_2$), 164.2 (C$_4$), 172.8 (*C*O-Lev), 207.6 (*C*O-Lev) ppm.

5′-*O*-Levulinyl-β-D-5-methyluridine (**2b**). ^{1}H-NMR (300.13 MHz, $CDCl_3$): δ 1.88 (s, 3H, *Me*), 2.19 (s, 3H, *Me*-Lev), 2.59 (t, 2H, C*H*$_2$-Lev, *J* = 6.3), 2.80 (t, 2H, C*H*$_2$-Lev, *J* = 6.3), 4.19 (m, 1H, H$_{3'}$), 4.23-4.37 (several m, 3H, H$_{2'}$ + H$_{4'}$ + H$_{5'}$), 4.45 (dd, 1H, H$_{5'}$, *J* = 12.3, 3.7 Hz), 5.84 (d, 1H, H$_{1'}$, *J* = 3.7 Hz), 7.41 (s, 1H, H$_6$) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ 12.5 (*C*H$_3$), 28.9 (*C*H$_2$-Lev), 29.6 (*C*H$_3$-Lev), 38.6 (*C*H$_2$-Lev), 64.8 (C$_{5'}$), 71.3 (C$_{3'}$), 74.9 (C$_{2'}$), 83.1 (C$_{4'}$), 91.1 (C$_{1'}$), 111.8 (C$_5$), 138.0 (C$_6$), 152.5 (C$_2$), 166.3 (C$_4$), 174.2 (*C*O-Lev), 209.5 (*C*O-Lev) ppm.

5′-*O*-Levulinyl-β-D-cytidine (**2c**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 2.17 (s, 3H, *Me*-Lev), 2.59 (t, 2H, C*H*$_2$-Lev, *J* = 5.9 Hz), 2.84 (t, 2H, C*H*$_2$-Lev, *J* = 6.8 Hz), 4.08 (dd, 1H, H$_{3'}$, *J* = 6.3, 5.3 Hz), 4.16 (m, 2H, H$_{2'}$ + H$_{4'}$), 4.33 (dd, 1H, H$_{5'}$, *J* = 12.3, 4.1 Hz), 4.40 (dd, 1H, H$_{5'}$, *J* = 12.4, 2.9 Hz), 5.84 (d, 1H, H$_5$, *J* = 3.2 Hz), 5.95 (d, 1H, H$_{1'}$, *J* = 7.5 Hz), 7.81 (d, 1H, H$_6$, *J* = 7.5 Hz) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ 28.8 (*C*H$_2$-Lev), 29.6 (*C*H$_3$-Lev), 38.6 (*C*H$_2$-Lev), 64.5 (C$_{5'}$), 71.0 (C$_{3'}$), 75.9 (C$_{2'}$), 82.7 (C$_{4'}$), 92.6 (C$_{1'}$), 96.2 (C$_5$), 142.5 (C$_6$), 158.2 (C$_2$), 167.6 (C$_4$), 174.2 (*C*O-Lev), 209.5 (*C*O-Lev) ppm.

N^4-Benzoyl-5′-*O*-levulinyl-β-D-cytidine (**2d**). ^{1}H-NMR (300.13 MHz, DMSO-d_6): δ 2.12 (s, 3H, *Me*-Lev), 2.54 (t, 2H, C*H*$_2$-Lev, *J* = 6.3 Hz), 2.75 (t, 2H, C*H*$_2$-Lev, *J* = 6.3 Hz), 3.97 (m, 1H, H$_{4'}$), 4.09 (m, 2H, H$_{2'}$ + H$_{3'}$), 4.25 (dd, 1H, H$_{5'}$, *J* = 12.2, 5.1 Hz), 4.35 (dd, 1H, H$_{5'}$, *J* = 12.1, 2.3 Hz), 5.82 (d, 1H, H$_{1'}$, *J* = 2.7 Hz), 7.38 (d, 1H, H$_5$, *J* = 7.5 Hz), 7.51 (m, 2H, H$_{arom}$), 7.63 (m, 1H, H$_{arom}$), 8.01 (m, 2H, H$_{arom}$), 8.16 (d, 1H, H$_6$, *J* = 7.5 Hz) ppm. ^{13}C-NMR (75.5 MHz, DMSO-d_6): δ 28.0 (*C*H$_2$-Lev), 30.0 (*C*H$_3$-Lev), 37.9 (*C*H$_2$-Lev), 63.9 (C$_{5'}$), 69.7 (C$_{4'}$), 74.3 (C$_{2'}$), 81.3 (C$_{3'}$), 91.4 (C$_{1'}$), 96.9 (C$_5$), 128.9 (4CH$_{arom}$), 133.2, 133.6 (CH$_{arom}$ + C$_{arom}$), 145.5 (C$_6$), 154.9 (C$_2$), 163.5 (C$_4$), 168.0 (*C*=O), 172.2 (*C*O-Lev), 207.4 (*C*O-Lev) ppm.

5′-*O*-Levulinyl-β-D-adenosine (**2e**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 2.14 (s, 3H, *Me*-Lev), 2.56 (t, 2H, C*H*$_2$-Lev, *J* = 6.4 Hz), 2.78 (m, 2H, C*H*$_2$-Lev, *J* = 6.4 Hz), 4.25 (q, 1H, H$_{4'}$, *J* = 4.7 Hz), 4.37 (m, 3H, H$_{3'}$ + H$_{5'}$), 4.74 (t, 2H, H$_{2'}$, *J* = 4.9 Hz), 6.03 (d, 1H, H$_{1'}$, *J* = 4.5 Hz), 8.21 (s, 1H, H$_2$), 8.31 (s, 1H, H$_8$) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ 28.8 (*C*H$_2$-Lev), 29.6 (*C*H$_3$-Lev), 38.6 (*C*H$_2$-Lev), 64.8 (C$_{5'}$), 71.8 (C$_{3'}$), 75.2 (C$_{2'}$), 83.5 (C$_{4'}$), 90.2 (C$_{1'}$), 120.5 (C$_5$, cross peak with H$_8$ in HMBC), 141.2 (C$_8$), 150.6 (C$_4$),153.9 (C$_2$), 157.3 (C$_6$), 174.2 (*C*O-Lev), 209.5 (*C*O-Lev) ppm.

5′-*O*-Levulinyl-β-D-inosine (**2f**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 2.15 (s, 3H, *Me*-Lev), 2.57 (t, 2H, C*H*$_2$-Lev, *J* = 6.4 Hz), 2.80 (t, 2H, C*H*$_2$-Lev, *J* = 6.4 Hz), 4.25 (q, 1H, H$_{4'}$, *J* = 4.3 Hz), 4.37 (m, 3H, H$_{3'}$ + H$_{5'}$), 4.70 (t, 2H, H$_{2'}$, *J* = 4.8 Hz), 6.03 (d, 1H, H$_{1'}$, *J* = 4.6 Hz), 8.07 (s, 1H, H$_2$), 8.27 (s, 1H, H$_8$) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ 28.8 (*C*H$_2$-Lev), 29.6 (*C*H$_3$-Lev), 38.6 (*C*H$_2$-Lev), 64.8 (C$_{5'}$), 71.7 (C$_{3'}$), 75.5 (C$_{2'}$), 83.7 (C$_{4'}$), 90.4 (C$_{1'}$), 125.9 (C$_5$), 140.7 (C$_8$), 146.8 (C$_2$), 149.9 (C$_4$, cross peak with H$_{1'}$ in HMBC), 158.9 (C$_6$), 174.2 (*C*O-Lev), 209.5 (*C*O-Lev) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-β-d-uridine (**5a**). ^{1}H-NMR (300.13 MHz, $CDCl_3$): δ 0.10 (s, 6H, Si-*Me*), 0.91 (s, 9H, Si-^{t}*Bu*), 3.83 (dd, 1H, H$_{5'}$, *J* = 11.7, 1.5 Hz), 4.01 (dd, 1H, H$_{5'}$, *J* = 11.8, 1.8 Hz), 4.12 (m, 1H, H$_{4'}$), 4.23 (m, 2H, H$_{2'}$ + H$_{3'}$), 5.64 (d, 1H, H$_5$, *J* = 8.1 Hz), 5.90 (d, 1H, H$_{1'}$, *J* = 2.2 Hz), 8.05 (d, 1H, H$_6$, *J* = 8.1 Hz) ppm. ^{13}C-NMR (75.5 MHz, $CDCl_3$): δ -5.4 (Si-*C*H$_3$), 18.6 (Si*C*Me$_3$), 26.1 (^{t}Bu-*C*H$_3$), 62.0 (C$_{5'}$), 69.5 (C$_{2'}$), 75.8 (C$_{3'}$), 85.1 (C$_{4'}$), 90.3 (C$_{1'}$), 102.3 (C$_5$), 140.6 (C$_6$), 151.5 (C$_2$), 164.1 (C$_4$) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-β-d-5-methyluridine (**5b**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 0.15 (s, 3H, Si-*Me*), 0.16 (s, 3H, Si-*Me*), 0.97 (s, 9H, Si-^{t}*Bu*), 1.88 (d, 3H, Me, *J* = 1.1 Hz), 3.85 (dd, 1H, H$_{5'}$, *J* = 11.6, 2.7 Hz), 3.94 (dd, 1H, H$_{5'}$, *J* = 11.6, 2.3 Hz), 4.02 (q, 1H, H$_{4'}$, *J* = 2.6 Hz), 4.13 (m, 2H, H$_{2'}$ + H$_{3'}$), 5.95 (d, 1H, H$_{1'}$, *J* = 5.0 Hz), 7.58 (d, 1H, H$_6$, *J* = 1.1 Hz) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ -5.2 (Si-*C*H$_3$), 12.6 (*C*H$_3$), 19.4 (Si*C*Me$_3$), 26.5 (^{t}Bu-*C*H$_3$), 64.3 (C$_{5'}$), 71.7 (C$_{2'}$), 75.7 (C$_{3'}$), 86.4 (C$_{4'}$), 89.6 (C$_{1'}$), 111.6 (C$_5$), 137.4 (C$_6$), 152.6 (C$_2$), 166.2 (C$_4$) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-β-d-cytidine (**5c**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 0.15 (s, 6H, Si-*Me*), 0.96 (s, 9H, Si-^{t}*Bu*), 3.87 (dd, 1H, H$_{5'}$, *J* = 12.0, 2.1 Hz), 4.02-4.16 (several m, 4H, H$_{2'}$ + H$_{3'}$ + H$_{4'}$ + H$_{5'}$), 5.85 (d, 1H, H$_5$, *J* = 7.6 Hz), 5.88 (d, 1H, H$_{1'}$, *J* = 2.7 Hz), 8.11 (d, 1H, H$_6$, *J* = 7.5 Hz) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ -5.39 (Si-*C*H$_3$), -5.36 (Si-*C*H$_3$), 19.3 (Si*C*Me$_3$), 26.5 (^{t}Bu-*C*H$_3$), 63.0 (C$_{5'}$), 70.1 (C$_{2'}$), 76.8 (C$_{3'}$), 85.3 (C$_{4'}$), 91.8 (C$_{1'}$), 95.71 (C$_5$), 142.4 (C$_6$), 158.4 (C$_2$), 167.6 (C$_4$) ppm.

N^4-Benzoyl-5′-*O*-(*tert*-butyldimethylsilyl)-β-d-cytidine (**5d**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 0.17 (s, 3H, Si-*Me*), 0.19 (s, 3H, Si-*Me*), 0.99 (s, 9H, Si-^{t}*Bu*), 3.91 (dd, 1H, H$_{5'}$, *J* = 12.2, 2.1 Hz), 4.15 (several m, 4H, H$_{2'}$+ H$_{3'}$+ H$_{4'}$+ H$_{5'}$), 5.90 (d, 1H, H$_{1'}$, *J* = 1.5 Hz), 7.58 (m, 4H, H$_5$ + H$_{arom}$), 7.98 (m, 2H, H$_{arom}$), 8.64 (d, 1H, H$_6$, *J* = 7.5 Hz) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ -5.3 (Si-*C*H$_3$), 19.4 (Si*C*Me$_3$), 26.5 (^{t}Bu-*C*H$_3$), 62.6 (C$_{5'}$), 69.5 (C$_{2'}$), 76.8 (C$_{3'}$), 85.5 (C$_{4'}$), 92.9 (C$_{1'}$), 98.1 (C$_5$), 129.2 (CH$_{arom}$), 129.8 (CH$_{arom}$), 134.1 (CH$_{arom}$), 134.7 (C$_{arom}$), 146.3 (C$_6$), 157.9 (C$_2$), 164.8 (C$_4$), 169.2 (*C*=O) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-β-d-adenosine (**5e**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 0.11 (s, 6H, Si-*Me*), 0.92 (s, 9H, Si-^{t}*Bu*), 3.87 (dd, 1H, H$_{5'}$, *J* = 11.6, 3.0 Hz), 4.01 (dd, 1H, H$_{5'}$, *J* = 11.6, 3.0 Hz), 4.13 (m, 1H, H$_{4'}$), 4.36 (t, 1H, H$_{3'}$, *J* = 5.0 Hz), 4.56 (t, 1H, H$_{2'}$, *J* = 4.6 Hz), 6.05 (d, 1H, H$_{1'}$, *J* = 4.2 Hz), 8.20 (s, 1H, H$_2$), 8.38 (s, 1H, H$_8$) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ -5.33 (Si-*C*H$_3$), -5.31 (Si-*C*H$_3$), 19.3 (Si*C*Me$_3$), 26.5 (^{t}Bu-*C*H$_3$), 63.8 (C$_{5'}$), 71.3 (C$_{3'}$), 76.3 (C$_{2'}$), 86.3 (C$_{4'}$), 90.1 (C$_{1'}$), 120.4 (C$_5$), 140.7 (C$_8$), 150.5 (C$_4$), 153.9 (C$_2$), 157.3 (C$_6$) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-β-d-inosine (**5f**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 0.10 (s, 6H, Si-*Me*), 0.91 (s, 9H, Si-^{t}*Bu*), 3.86 (dd, 1H, H$_{5'}$, *J* = 11.6, 3.1 Hz), 3.99 (dd, 1H, H$_{5'}$, *J* = 11.6, 3.0 Hz), 4.14 (m, 1H, H$_{4'}$), 4.36 (t, 1H, H$_{3'}$, *J* = 4.8 Hz), 4.56 (t, 1H, H$_{2'}$, *J* = 4.8 Hz), 6.07 (d, 1H, H$_{1'}$, *J* = 4.5 Hz), 8.07 (s, 1H, H$_2$), 8.31 (s, 1H, H$_8$) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ -5.33 (Si-*C*H$_3$), -5.29 (Si-*C*H$_3$), 19.3 (Si*C*Me$_3$), 26.5 (^{t}Bu-*C*H$_3$), 63.9 (C$_{5'}$), 71.5 (C$_{3'}$), 76.6 (C$_{2'}$), 86.6 (C$_{4'}$), 90.0 (C$_{1'}$), 125.6 (C$_5$), 140.1 (C$_8$), 146.8 (C$_2$), 149.9 (C$_4$), 158.9 (C$_6$) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-bis-*O*-[(ethylthio)thiocarbonyl]-β-d-uridine (**6a**). ^{1}H-NMR (300.13 MHz, CDCl$_3$): δ 0.15 (s, 3H, Si-*Me*), 0.17 (s, 3H, Si-*Me*), 0.95 (s, 9H, Si-^{t}*Bu*), 1.35 (t, 3H, SCH$_2$-*Me*, *J* = 7.4 Hz), 1.37 (t, 3H, SCH$_2$-*Me*, *J* = 7.4 Hz), 3.13 (m, 4H, S-C*H*$_2$), 3.92 (dd, 1H, H$_{5'}$, *J* = 11.4, 1.7 Hz), 4.03 (dd, 1H, H$_{5'}$, *J* = 11.4, 1.5 Hz), 4.44 (m, 1H, H$_{4'}$), 5.75 (d, 1H, H$_5$, *J* = 8.2 Hz), 6.07 (dd, 1H, H$_{2'}$, *J* = 7.4, 5.7 Hz), 6.22 (dd, 1H, H$_{3'}$, *J* = 5.6, 1.1 Hz), 6.56 (d, 1H, H$_{1'}$, *J* = 7.4 Hz), 7.87 (d, 1H, H$_6$, *J* = 8.2 Hz), 9.09 (br s, 1H, NH) ppm. ^{13}C-NMR (75.5 MHz, CDCl$_3$): δ -5.5 (Si-*C*H$_3$), -5.2 (Si-*C*H$_3$), 13.3 (SCH$_2$-*C*H$_3$), 13.5 (SCH$_2$-*C*H$_3$), 18.5 (Si*C*Me$_3$), 26.1 (^{t}Bu-*C*H$_3$), 30.8 (S-*C*H$_2$), 63.5 (C$_{5'}$), 78.9 (C$_{3'}$), 79.6 (C$_{2'}$), 84.6 (C$_{4'}$), 85.4 (C$_{1'}$), 103.6 (C$_5$), 139.7 (C$_6$), 150.4 (C$_2$), 163.0 (C$_4$), 213.7 (*C*=S), 214.1 (*C*=S) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-bis-*O*-[(ethylthio)thiocarbonyl]-β-d-5-methyluridine (**6b**). ^{1}H-NMR (300.13 MHz, CDCl$_3$): δ 0.19 (s, 6H, Si-*Me*), 0.98 (s, 9H, Si-^{t}*Bu*), 1.35 (t, 3H, SCH$_2$-*Me*, *J* = 7.4 Hz), 1.37 (t, 3H, SCH$_2$-*Me*, *J* = 7.4 Hz), 1.94 (d, 3H, Me, *J* = 1.1 Hz), 3.14 (m, 4H, S-C*H*$_2$), 3.93 (dd, 1H, H$_{5'}$, *J* = 11.4, 1.7 Hz), 4.07 (dd, 1H, H$_{5'}$, *J* = 11.4, 1.7 Hz), 4.42 (m, 1H, H$_{4'}$), 6.03 (dd, 1H, H$_{2'}$, *J* = 7.8, 5.6 Hz), 6.22 (dd, 1H, H$_{3'}$, *J* = 5.6, 1.0 Hz), 6.56 (d, 1H, H$_{1'}$, *J* = 7.9 Hz), 7.54 (d, 1H, H$_6$, *J* = 1.2 Hz), 8.16 (br s, 1H, NH) ppm. ^{13}C-NMR (75.5 MHz, CDCl$_3$): δ -5.3 (Si-*C*H$_3$), -4.9 (Si-*C*H$_3$), 12.6 (*C*H$_3$), 13.4 (SCH$_2$-*C*H$_3$), 13.5 (SCH$_2$-*C*H$_3$), 18.6 (Si*C*Me$_3$), 26.2 (^{t}Bu-*C*H$_3$), 30.79 (S-*C*H$_2$), 30.83 (S-*C*H$_2$), 63.5 (C$_{5'}$), 78.7 (C$_{3'}$), 79.1 (C$_{2'}$), 84.4 (C$_{4'}$), 84.7 (C$_{1'}$), 112.2 (C$_5$), 134.9 (C$_6$), 150.5 (C$_2$), 163.5 (C$_4$), 213.7 (*C*=S), 214.2 (*C*=S) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-bis-*O*-[(ethylthio)thiocarbonyl]-β-d-cytidine (**6c**). ^{1}H-NMR (300.13 MHz, CDCl$_3$): δ 0.14 (s, 3H, Si-*Me*), 0.15 (s, 3H, Si-*Me*), 0.94 (s, 9H, Si-^{t}*Bu*), 1.33 (t, 3H, SCH$_2$-*Me*, *J* = 6.1 Hz), 1.35 (t, 3H, SCH$_2$-*Me*, *J* = 6.1 Hz), 3.13 (m, 4H, S-C*H*$_2$), 3.92 (dd, 1H, H$_{5'}$, *J* = 11.3, 1.8 Hz), 4.01 (dd, 1H, H$_{5'}$, *J* = 11.3, 1.7 Hz), 4.41 (m, 1H, H$_{4'}$), 5.76 (d, 1H, H$_5$, *J* = 7.4 Hz), 6.04 (dd, 1H, H$_{2'}$, *J* = 7.0, 5.6 Hz), 6.24 (dd, 1H, H$_{3'}$, *J* = 5.5, 2.0 Hz), 6.67 (d, 1H, H$_{1'}$, *J* = 7.0 Hz), 7.84 (d, 1H, H$_6$, *J* = 7.4 Hz) ppm. ^{13}C-NMR (75.5 MHz, CDCl$_3$): δ -5.4 (Si-*C*H$_3$), -5.2 (Si-*C*H$_3$), 13.4 (SCH$_2$-*C*H$_3$), 13.6 (SCH$_2$-*C*H$_3$), 18.5 (Si*C*Me$_3$), 26.2 (^{t}Bu-*C*H$_3$), 30.66 (S-*C*H$_2$), 30.71 (S-*C*H$_2$), 63.4 (C$_{5'}$), 78.8 (C$_{3'}$), 80.4 (C$_{2'}$), 83.9 (C$_{4'}$), 86.1 (C$_{1'}$), 95.6 (C$_5$), 141.3 (C$_6$), 155.8 (C$_2$), 165.7 (C$_4$), 213.7 (*C*=S), 214.1 (*C*=S) ppm.

N^4-Benzoyl-5′-*O*-(*tert*-butyldimethylsilyl)-2′,3′-bis-*O*-[(ethylthio)thiocarbonyl]-β-d-cytidine (**6d**). ^{1}H-NMR (300.13 MHz, CDCl$_3$): δ 0.18 (s, 6H, Si-*Me*), 0.97 (s, 9H, Si-^{t}*Bu*), 1.34 (t, 3H, SCH$_2$-*Me*, *J* = 6.1 Hz), 1.37 (t, 3H,

SCH_2-*Me*, *J* = 6.1 Hz), 3.14 (m, 4H, S-C*H*$_2$), 3.98 (dd, 1H, $H_{5'}$, *J* = 11.5, 1.8 Hz), 4.05 (dd, 1H, $H_{5'}$, *J* = 11.5, 1.6 Hz), 4.49 (m, 1H, $H_{4'}$), 6.15 (dd, 1H, $H_{2'}$, *J* = 6.8, 5.6 Hz), 6.27 (dd, 1H, $H_{3'}$, *J* = 5.5, 1.8 Hz), 6.74 (d, 1H, $H_{1'}$, *J* = 6.9 Hz), 7.50 (m, 3H, H_5 + H_{arom}), 7.61 (m, 1H, H_{arom}), 7.91 (m, 2H, H_{arom}), 8.30 (d, 1H, H_6, *J* = 7.6 Hz), 8.69 (br s, 1H, NH) ppm. ^{13}C-NMR (75.5 MHz, $CDCl_3$): δ -5.4 (Si-*C*H_3), -5.2 (Si-*C*H_3), 13.3 (SCH_2-*C*H_3), 13.5 (SCH_2-*C*H_3), 18.5 (Si*C*Me_3), 26.1 (^{t}Bu-*C*H_3), 30.65 (S-*C*H_2), 30.74 (S-*C*H_2), 63.2 ($C_{5'}$), 78.8 ($C_{3'}$), 80.7 ($C_{2'}$), 84.6 ($C_{4'}$), 86.5 ($C_{1'}$), 97.8 (C_5), 127.8 (CH_{arom}), 129.1 (CH_{arom}), 133.2 (C_{arom}), 133.3 (CH_{arom}), 144.4 (C_6), 154.6 (C_2), 162.5 (C_4), 167.1 (*C*=O), 213.6 (*C*=S), 214.0 (*C*=S) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-bis-*O*-[(ethylthio)thiocarbonyl]-β-d-adenosine (**6e**). ^{1}H-NMR (300.13 MHz, $CDCl_3$): δ 0.16 (s, 6H, Si-*Me*), 0.95 (s, 9H, Si-^{t}*Bu*), 1.29 (t, 3H, SCH_2-*Me*, *J* = 7.4 Hz), 1.37 (t, 3H, SCH_2-*Me*, *J* = 7.4 Hz), 3.07 (q, 2H, S-C*H*$_2$, *J* = 7.4 Hz), 3.17 (q, 2H, S-C*H*$_2$, *J* = 7.3 Hz), 3.95 (dd, 1H, $H_{5'}$, *J* = 11.4, 2.1 Hz), 4.05 (dd, 1H, $H_{5'}$, *J* = 11.3, 2.1 Hz), 4.52 (m, 1H, $H_{4'}$), 6.14 (br s, 2H, NH_2), 6.41 (dd, 1H, $H_{3'}$, *J* = 5.4, 1.9 Hz), 6.48 (dd, 1H, $H_{2'}$, *J* = 6.8, 5.5 Hz), 6.60 (d, 1H, $H_{1'}$, *J* = 6.9 Hz), 8.22 (s, 1H, H_8), 8.35 (s, 1H, H_2) ppm. ^{13}C-NMR (75.5 MHz, $CDCl_3$): δ -5.3 (Si-*C*H_3), -5.1 (Si-*C*H_3), 13.3 (SCH_2-*C*H_3), 18.6 (Si*C*Me_3), 26.2 (^{t}Bu-*C*H_3), 30.8 (S-*C*H_2), 63.4 ($C_{5'}$), 78.8 ($C_{3'}$), 80.7 ($C_{2'}$), 84.7 ($C_{1'}$ + $C_{4'}$), 119.6 (C_5), 138.5 (C_8), 150.3 (C_4), 153.4 (C_2), 155.7 (C_6), 213.6 (*C*=S), 214.0 (*C*=S) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-bis-*O*-[(ethylthio)thiocarbonyl]-β-d-inosine (**6f**). ^{1}H-NMR (300.13 MHz, $CDCl_3$): δ 0.17 (s, 6H, Si-*Me*), 0.96 (s, 9H, Si-^{t}*Bu*), 1.31 (t, 3H, SCH_2-*Me*, *J* = 7.4 Hz), 1.39 (t, 3H, SCH_2-*Me*, *J* = 7.4 Hz), 3.09 (q, 2H, S-C*H*$_2$, *J* = 7.4 Hz), 3.18 (q, 2H, S-C*H*$_2$, *J* = 7.4 Hz), 3.97 (dd, 1H, $H_{5'}$, *J* = 11.4, 2.0 Hz), 4.06 (dd, 1H, $H_{5'}$, *J* = 11.4, 2.0 Hz), 4.55 (m, 1H, $H_{4'}$), 6.40 (dd, 1H, $H_{3'}$, *J* = 5.3, 2.0 Hz), 6.46 (m, 1H, $H_{2'}$), 6.53 (d, 1H, $H_{1'}$, *J* = 6.7 Hz), 8.20 (s, 1H, H_2), 8.24 (s, 1H, H_8), 13.10 (br s, 1H, NH) ppm. ^{13}C-NMR (75.5 MHz, $CDCl_3$): δ -5.3 (Si-*C*H_3), -5.1 (Si-*C*H_3), 13.3 (SCH_2-*C*H_3), 13.4 (SCH_2-*C*H_3), 18.6 (Si*C*Me_3), 26.2 (^{t}Bu-*C*H_3), 30.86 (S-*C*H_2), 30.93 (S-*C*H_2), 63.3 ($C_{5'}$), 78.7 ($C_{3'}$), 80.9 ($C_{2'}$), 84.9 ($C_{4'}$), 85.2 ($C_{1'}$), 124.9 (C_5), 138.3 (C_8), 145.6 (C_2), 149.3 (C_4), 159.3 (C_6), 213.4 (*C*=S), 214.0 (*C*=S) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-didehydro-2′,3′-dideoxy-β-d-uridine (**7a**). ^{1}H-NMR (300.13 MHz, DMSO-d_6): δ 0.03 (s, 6H, Si-*Me*), 0.85 (s, 9H, Si-^{t}*Bu*), 3.75 (dd, 1H, $H_{5'}$, *J* = 11.7, 3.7 Hz), 3.81 (dd, 1H, $H_{5'}$, *J* = 11.7, 3.0 Hz), 4.82 (m, 1H, $H_{4'}$), 5.52 (d, 1H, H_5, *J* = 8.0 Hz), 5.95 (dt, 1H, $H_{2'}$, *J* = 6.0, 1.8 Hz), 6.39 (dt, 1H, $H_{3'}$, *J* = 6.0, 1.6 Hz), 6.80 (m, 1H, $H_{1'}$), 7.63 (d, 1H, H_6, *J* = 8.0 Hz), 11.35 (br s, 1H, NH) ppm. ^{13}C-NMR (75.5 MHz, DMSO-d_6): δ -5.43 (Si-*C*H_3), -5.38 (Si-*C*H_3), 18.2 (Si*C*Me_3), 25.8 (^{t}Bu-*C*H_3), 64.3 ($C_{5'}$), 86.9 ($C_{4'}$), 89.1 ($C_{1'}$), 101.6 (C_5), 126.2 ($C_{2'}$), 134.5 ($C_{3'}$), 140.8 (C_6), 150.7 (C_2), 163.1 (C_4) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-didehydro-3′-deoxy-β-d-5-thymidine (**7b**). ^{1}H-NMR (300.13 MHz, DMSO-d_6): δ 0.03 (s, 6H, Si-*Me*), 0.85 (s, 9H, Si-^{t}*Bu*), 1.75 (d, 3H, Me, *J* = 0.9 Hz), 3.72 (dd, 1H, $H_{5'}$, *J* = 11.5, 4.6 Hz), 3.80 (dd, 1H, $H_{5'}$, *J* = 11.6, 3.2 Hz), 4.78 (m, 1H, $H_{4'}$), 5.96 (dt, 1H, $H_{2'}$, *J* = 6.0, 2.0 Hz), 6.38 (dt, 1H, $H_{3'}$, *J* = 6.0, 1.6 Hz), 6.78 (m, 1H, $H_{1'}$), 7.28 (d, 1H, H_6, *J* = 1.2 Hz), 11.36 (br s, 1H, NH) ppm. ^{13}C-NMR (75.5 MHz, DMSO-d_6): δ -5.3 (Si-*C*H_3), -5.2 (Si-*C*H_3), 12.2 (*C*H_3), 18.3 (Si*C*Me_3), 25.9 (^{t}Bu-*C*H_3), 64.7 ($C_{5'}$), 86.7 ($C_{4'}$), 89.2 ($C_{1'}$), 109.4 (C_5), 126.2 ($C_{2'}$), 134.4 ($C_{3'}$), 135.9 (C_6), 150.8 (C_2), 163.8 (C_4) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-didehydro-2′,3′-dideoxy-β-d-cytidine (**7c**). ^{1}H-NMR (300.13 MHz, $CDCl_3$): δ 0.066 (s, 3H, Si-*Me*), 0.070 (s, 3H, Si-*Me*), 0.90 (s, 9H, Si-^{t}*Bu*), 3.83 (dd, 1H, $H_{5'}$, *J* = 11.6, 2.8 Hz), 3.92 (dd, 1H, $H_{5'}$, *J* = 11.5, 3.0 Hz), 4.90 (m, 1H, $H_{4'}$), 5.62 (d, 1H, H_5, *J* = 7.4 Hz), 5.91 (m, 1H, $H_{2'}$), 6.14 (dt, 1H, $H_{3'}$, *J* = 6.0, 1.6 Hz), 7.07 (m, 1H, $H_{1'}$), 7.94 (d, 1H, H_6, *J* = 7.4 Hz) ppm. ^{13}C-NMR (75.5 MHz, $CDCl_3$): δ -5.3 (Si-*C*H_3), -5.2 (Si-*C*H_3), 18.6 (Si*C*Me_3), 26.1 (^{t}Bu-*C*H_3), 64.5 ($C_{5'}$), 87.3 ($C_{4'}$), 91.0 ($C_{1'}$), 94.0 (C_5), 128.2 ($C_{2'}$), 132.7 ($C_{3'}$), 142.8 (C_6), 156.3 (C_2), 165.6 (C_4) ppm.

N^4-Benzoyl-5′-*O*-(*tert*-butyldimethylsilyl)-2′,3′-didehydro-2′,3′-dideoxy-β-d-cytidine (**7d**). ^{1}H-NMR (300.13 MHz, $CDCl_3$): δ 0.08 (s, 3H, Si-*Me*), 0.10 (s, 3H, Si-*Me*), 0.90 (s, 9H, Si-^{t}*Bu*), 3.86 (dd, 1H, $H_{5'}$, *J* = 11.7, 2.6 Hz), 3.99 (dd, 1H, $H_{5'}$, *J* = 11.7, 2.7 Hz), 4.99 (m, 1H, $H_{4'}$), 6.02 (d, 1H, $H_{2'}$, *J* = 5.7 Hz), 6.18 (dt, 1H, $H_{3'}$, *J* = 6.0, 1.4 Hz), 7.03 (m, 1H, $H_{1'}$), 7.49 (m, 3H, H_5 + H_{arom}), 7.60 (m, 1H, H_{arom}), 7.93 (d, 2H, H_{arom}, *J* = 7.3 Hz), 8.40 (d, 1H, H_6, *J* = 7.5 Hz), 8.71 (br s, 1H, NH) ppm. ^{13}C-NMR (75.5 MHz, $CDCl_3$): δ -5.4 (Si-*C*H_3), -5.2 (Si-*C*H_3), 18.6

(SiCMe$_3$), 26.0 (^{t}Bu-CH$_3$), 64.2 (C$_{5'}$), 88.0 (C$_{4'}$), 91.8 (C$_{1'}$), 96.9 (C$_5$), 127.67 (C$_{2'}$), 127.71 (CH$_{arom}$), 129.1 (CH$_{arom}$), 133.0 (C$_{3'}$), 133.2 (CH$_{arom}$), 133.4 (C$_{arom}$), 145.7 (C$_6$), 155.1 (C$_2$), 162.3 (C$_4$), 167.3 (*C*=O) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-didehydro-2′,3′-dideoxy-β-d-adenosine (**7e**). ^{1}H-NMR (300.13 MHz, CDCl$_3$): δ 0.03 (s, 3H, Si-*Me*), 0.04 (s, 3H, Si-*Me*), 0.87 (s, 9H, Si-^{t}*Bu*), 3.81 (d, 2H, H$_{5'}$, *J* = 3.8 Hz), 4.97 (m, 1H, H$_{4'}$), 5.95 (br s, 2H, NH$_2$), 6.03 (dt, 1H, H$_{2'}$, *J* = 6.0, 1.9 Hz), 6.38 (dt, 1H, H$_{3'}$, *J* = 6.0, 1.6 Hz), 7.09 (m, 1H, H$_{1'}$), 8.09 (s, 1H, H$_8$), 8.34 (s, 1H, H$_2$) ppm. ^{13}C-NMR (75.5 MHz, CDCl$_3$): δ -5.3 (Si-*C*H$_3$), -5.2 (Si-*C*H$_3$), 18.7 (SiCMe$_3$), 26.1 (^{t}Bu-*C*H$_3$), 64.9 (C$_{5'}$), 87.9 (C$_{4'}$), 88.4 (C$_{1'}$), 119.2 (C$_5$), 125.7 (C$_{2'}$), 134.7 (C$_{3'}$), 139.4 (C$_8$), 149.7 (C$_4$), 152.9 (C$_2$), 155.7 (C$_6$) ppm.

5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-didehydro-2′,3′-dideoxy-β-d-inosine (**7f**). ^{1}H-NMR (300.13 MHz, CDCl$_3$): δ 0.03 (s, 3H, Si-*Me*), 0.04 (s, 3H, Si-*Me*), 0.87 (s, 9H, Si-^{t}*Bu*), 3.82 (m, 2H, H$_{5'}$), 4.99 (m, 1H, H$_{4'}$), 6.05 (dt, 1H, H$_{2'}$, *J* = 5.9, 1.8 Hz), 6.42 (dt, 1H, H$_{3'}$, *J* = 5.8, 1.7 Hz), 7.04 (m, 1H, H$_{1'}$), 8.09 (s, 1H, H$_8$), 8.23 (s, 1H, H$_2$), 13.23 (br s, 1H, NH) ppm. ^{13}C-NMR (75.5 MHz, CDCl$_3$): δ -5.3 (Si-*C*H$_3$), -5.2 (Si-*C*H$_3$), 18.7 (SiCMe$_3$), 26.1 (^{t}Bu-*C*H$_3$), 64.9 (C$_{5'}$), 88.1 (C$_{4'}$), 88.6 (C$_{1'}$), 124.7 (C$_5$), 125.3 (C$_{2'}$), 135.0 (C$_{3'}$), 139.1 (C$_8$), 145.3 (C$_2$), 149.0 (C$_4$), 159.5 (C$_6$) ppm.

2′,3′-Didehydro-2′,3′-dideoxy-β-d-uridine (**8a**). ^{1}H-NMR (300.13 MHz, MeOH-*d*$_4$): δ 3.73 (m, 2H, H$_{5'}$), 4.87 (m, 1H, H$_{4'}$), 5.65 (d, 1H, H$_5$, *J* = 8.1 Hz), 5.92 (m, 1H, H$_{2'}$), 6.41 (dt, 1H, H$_{3'}$, *J* = 6.0, 1.7 Hz), 6.94 (dt, 1H, H$_{1'}$, *J* = 3.1, 1.5 Hz), 7.89 (d, 1H, H$_6$, *J* = 8.1 Hz) ppm. ^{13}C-NMR (75.5 MHz, MeOH-*d*$_4$): δ 63.8 (C$_{5'}$), 89.1 (C$_{4'}$), 91.3 (C$_{1'}$), 102.5 (C$_5$), 127.0 (C$_{2'}$), 136.1 (C$_{3'}$), 143.1 (C$_6$), 152.7 (C$_2$), 166.3 (C$_4$) ppm.

2′,3′-Didehydro-3′-deoxy-β-d-5-thymidine (**8b**). ^{1}H-NMR (300.13 MHz, MeOH-*d*$_4$): δ 1.84 (d, 3H, Me, *J* = 1.1 Hz), 3.76 (m, 2H, H$_{5'}$), 4.86 (debajo de H$_2$O, 1H, H$_{4'}$), 5.90 (dt, 1H, H$_{2'}$, *J* = 6.0, 2.0 Hz), 6.40 (dt, 1H, H$_{3'}$, *J* = 6.0, 1.7 Hz), 6.95 (dt, 1H, H$_{1'}$, *J* = 3.4, 1.7 Hz), 7.75 (d, 1H, H$_6$, *J* = 1.2 Hz) ppm. ^{13}C-NMR (75.5 MHz, MeOH-*d*$_4$): δ 12.4 (*C*H$_3$), 63.8 (C$_{5'}$), 89.0 (C$_{4'}$), 91.0 (C$_{1'}$), 111.2 (C$_5$), 127.3 (C$_{2'}$), 135.9 (C$_{3'}$), 138.9 (C$_6$), 152.9 (C$_2$), 166.6 (C$_4$) ppm.

N^4-Benzoyl-2′,3′-didehydro-2′,3′-dideoxy-β-d-cytidine (**8d**). ^{1}H-NMR (300.13 MHz, MeOH-*d*$_4$): δ 3.78 (m, 2H, H$_{5'}$), 4.99 (m, 1H, H$_{4'}$), 6.05 (m, 1H, H$_{2'}$), 6.41 (dt, 1H, H$_{3'}$, *J* = 6.0, 1.6 Hz), 7.00 (dt, 1H, H$_{1'}$, *J* = 2.9, 1.4 Hz), 7.54 (m, 3H, H$_5$ + H$_{arom}$), 7.64 (m, 1H, H$_{arom}$), 7.97 (m, 2H, H$_{arom}$), 8.46 (d, 1H, H$_6$, *J* = 7.5 Hz) ppm. ^{13}C-NMR (75.5 MHz, MeOH-*d*$_4$): δ 63.7 (C$_{5'}$), 89.9 (C$_{4'}$), 93.4 (C$_{1'}$), 98.5 (C$_5$), 127.6 (C$_{2'}$), 129.2 (CH$_{arom}$), 129.8 (CH$_{arom}$), 134.1 (CH$_{arom}$), 134.7 (C$_{arom}$), 135.5 (C$_{3'}$), 147.3 (C$_6$), 158.3 (C$_2$), 165.0 (C$_4$), 169.1 (*C*=O) ppm.

2′,3′-Didehydro-2′,3′-dideoxy-β-d-adenosine (**8e**). ^{1}H-NMR (300.13 MHz, MeOH-*d*$_4$): δ 3.75 (m, 2H, H$_{5'}$), 5.01 (m, 1H, H$_{4'}$), 6.13 (m, 1H, H$_{2'}$), 6.48 (dt, 1H, H$_{3'}$, *J* = 6.0, 1.7 Hz), 7.04 (dt, 1H, H$_{1'}$, *J* = 3.2, 1.7 Hz), 8.20 (s, 1H, H$_2$), 8.28 (s, 1H, H$_8$) ppm. ^{13}C-NMR (75.5 MHz, MeOH-*d*$_4$): δ 64.2 (C$_{5'}$), 89.8 (C$_{1'}$), 90.3 (C$_{4'}$), 120.2 (C$_5$), 126.8 (C$_{2'}$), 135.7 (C$_{3'}$), 141.3 (C$_8$), 150.3 (C$_4$), 153.8 (C$_2$), 157.4 (C$_6$) ppm.

2′,3′-Didehydro-2′,3′-dideoxy-β-d-inosine (**8f**). ^{1}H-NMR (300.13 MHz, DMSO-*d*$_6$): δ 3.56 (m, 2H, H$_{5'}$), 4.89 (m, 1H, H$_{4'}$), 4.98 (t, OH$_{5'}$, *J* = 5.4 Hz), 6.13 (m, 1H, H$_{2'}$), 6.48 (dd, 1H, H$_{3'}$, *J* = 6.0, 1.5 Hz), 6.90 (m, 1H, H$_{1'}$), 8.08 (s, 1H, H$_2$), 8.11 (s, 1H, H$_8$), 12.39 (br s, 1H, NH) ppm. ^{13}C-NMR (75.5 MHz, DMSO-*d*$_6$): δ 62.7 (C$_{5'}$), 88.1 (C$_{1'}$), 88.3 (C$_{4'}$), 124.1 (C$_5$), 125.3 (C$_{2'}$), 134.8 (C$_{3'}$), 138.6 (C$_8$), 146.0 (C$_2$), 148.1 (C$_4$), 156.7 (C$_6$) ppm.

2′,3′-Dideoxy-β-d-uridine (**9a**). ^{1}H-NMR (300.13 MHz, MeOH-*d*$_4$): δ 1.96 (m, 2H, H$_{3'}$), 2.08 (m, 1H, H$_{2'}$), 2.40 (m, 1H, H$_{2'}$), 3.68 (dd, 1H, H$_{5'}$, *J* = 12.2, 4.0 Hz), 3.86 (dd, 1H, H$_{5'}$, *J* = 12.2, 3.1 Hz), 4.14 (m, 1H, H$_{4'}$), 5.66 (d, 1H, H$_5$, *J* = 8.1 Hz), 6.04 (dd, 1H, H$_{1'}$, *J* = 6.8, 3.4 Hz), 8.08 (d, 1H, H$_6$, *J* = 8.1 Hz) ppm. ^{13}C-NMR (75.5 MHz, MeOH-*d*$_4$): δ 25.9 (C$_{3'}$), 33.5 (C$_{2'}$), 63.7 (C$_{5'}$), 83.5 (C$_{4'}$), 87.7 (C$_{1'}$), 101.9 (C$_5$), 142.6 (C$_6$), 152.2 (C$_2$), 166.4 (C$_4$) ppm.

3′-Deoxy-β-d-5-thymidine (**9b**). ^{1}H-NMR (300.13 MHz, MeOH-*d*$_4$): δ 1.88 (d, 3H, Me, *J* = 1.1 Hz), 1.98 (m, 2H, H$_{3'}$), 2.07 (m, 1H, H$_{2'}$), 2.38 (m, 1H, H$_{2'}$), 3.68 (dd, 1H, H$_{5'}$, *J* = 12.2, 3.8 Hz), 3.88 (dd, 1H, H$_{5'}$, *J* = 12.2, 2.9

Hz), 4.13 (m, 1H, $H_{4'}$), 6.05 (dd, 1H, $H_{1'}$, J = 6.8, 3.5 Hz), 7.92 (d, 1H, H_6, J = 1.1 Hz) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ 12.4 (CH_3), 25.9 ($C_{3'}$), 33.3 ($C_{2'}$), 63.7 ($C_{5'}$), 83.2 ($C_{4'}$), 87.3 ($C_{1'}$), 110.8 (C_5), 138.4 (C_6), 152.4 (C_2), 166.6 (C_4) ppm.

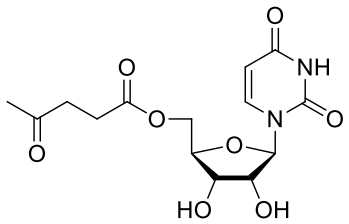
2′,3′-Dideoxy-β-d-cytidine (**9c**). ^{1}H-NMR (300.13 MHz, D_2O): δ 1.78 (m, 1H, $H_{3'}$), 2.05 (m, 2H, $H_{2'}$ + $H_{3'}$), 2.45 (m, 1H, $H_{2'}$), 3.72 (dd, 1H, $H_{5'}$, J = 12.4, 5.4 Hz), 3.87 (dd, 1H, $H_{5'}$, J = 12.4, 3.1 Hz), 4.23 (m, 1H, $H_{4'}$), 6.00 (d, 1H, H_5, J = 7.5 Hz), 6.05 (dd, 1H, $H_{1'}$, J = 7.0, 2.7 Hz), 7.89 (d, 1H, H_6, J = 7.5 Hz) ppm. ^{13}C-NMR (75.5 MHz, D_2O): δ 24.7 ($C_{3'}$), 31.9 ($C_{2'}$), 62.6 ($C_{5'}$), 82.2 ($C_{4'}$), 87.0 ($C_{1'}$), 95.5 (C_5), 141.6 (C_6), 157.3 (C_2), 165.9 (C_4) ppm.

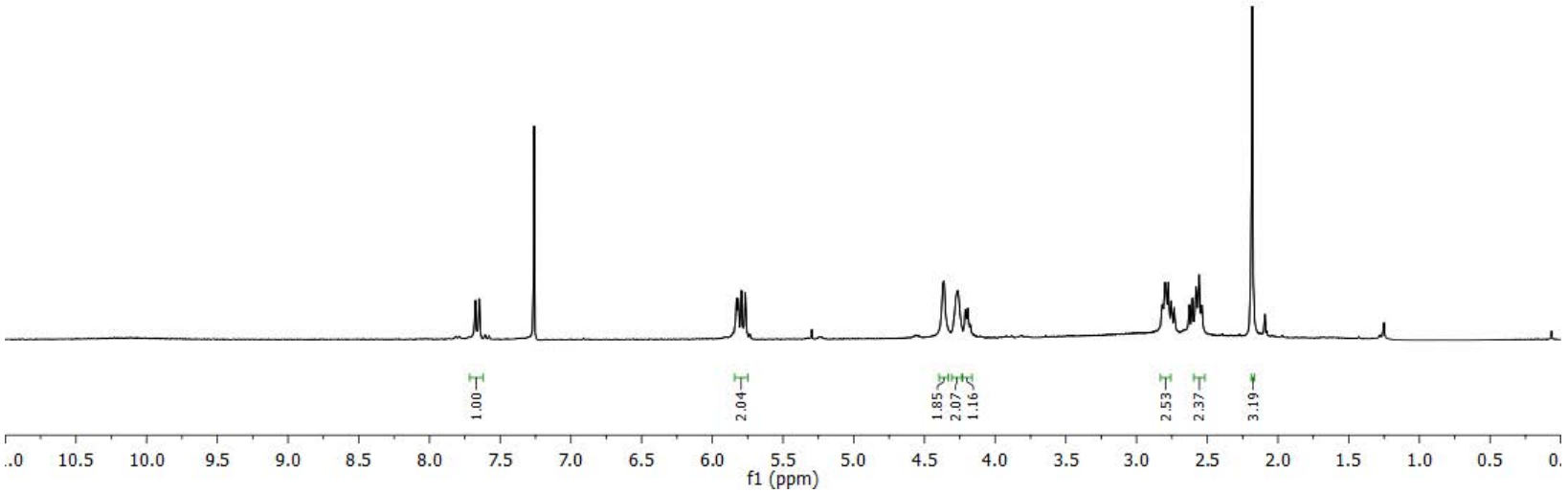
2′,3′-Dideoxy-β-d-adenosine (**9e**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 2.15 (m, 2H, $H_{3'}$), 2.51 (m, 2H, $H_{2'}$), 3.67 (dd, 1H, $H_{5'}$, J = 12.2, 3.9 Hz), 3.87 (dd, 1H, $H_{5'}$, J = 12.2, 3.0 Hz), 4.27 (m, 1H, $H_{4'}$), 6.28 (dd, 1H, $H_{1'}$, J = 5.8, 4.8 Hz), 8.18 (s, 1H, H_2), 8.41 (s, 1H, H_8) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ 26.5 ($C_{3'}$), 33.7 ($C_{2'}$), 64.5 ($C_{5'}$), 83.6 ($C_{4'}$), 87.4 ($C_{1'}$), 120.5 (C_5), 141.0 (C_2), 149.8 (C_4), 153.5 (C_8), 157.3 (C_6) ppm.

2′,3′-Dideoxy-β-d-inosine (**9f**). ^{1}H-NMR (300.13 MHz, MeOH-d_4): δ 2.12 (m, 2H, $H_{3'}$), 2.52 (m, 2H, $H_{2'}$), 3.67 (dd, 1H, $H_{5'}$, J = 12.1, 4.2 Hz), 3.84 (dd, 1H, $H_{5'}$, J = 12.1, 3.2 Hz), 4.26 (m, 1H, $H_{4'}$), 6.31 (dd, 1H, $H_{1'}$, J = 6.5, 3.5 Hz), 8.04 (s, 1H, H_2), 8.39 (s, 1H, H_8) ppm. ^{13}C-NMR (75.5 MHz, MeOH-d_4): δ 26.4 ($C_{3'}$), 33.9 ($C_{2'}$), 64.3 ($C_{5'}$), 83.9 ($C_{4'}$), 87.3 ($C_{1'}$), 125.7 (C_5), 140.4 (C_8), 146.6 (C_2), 149.3 (C_4), 159.0 (C_6) ppm.

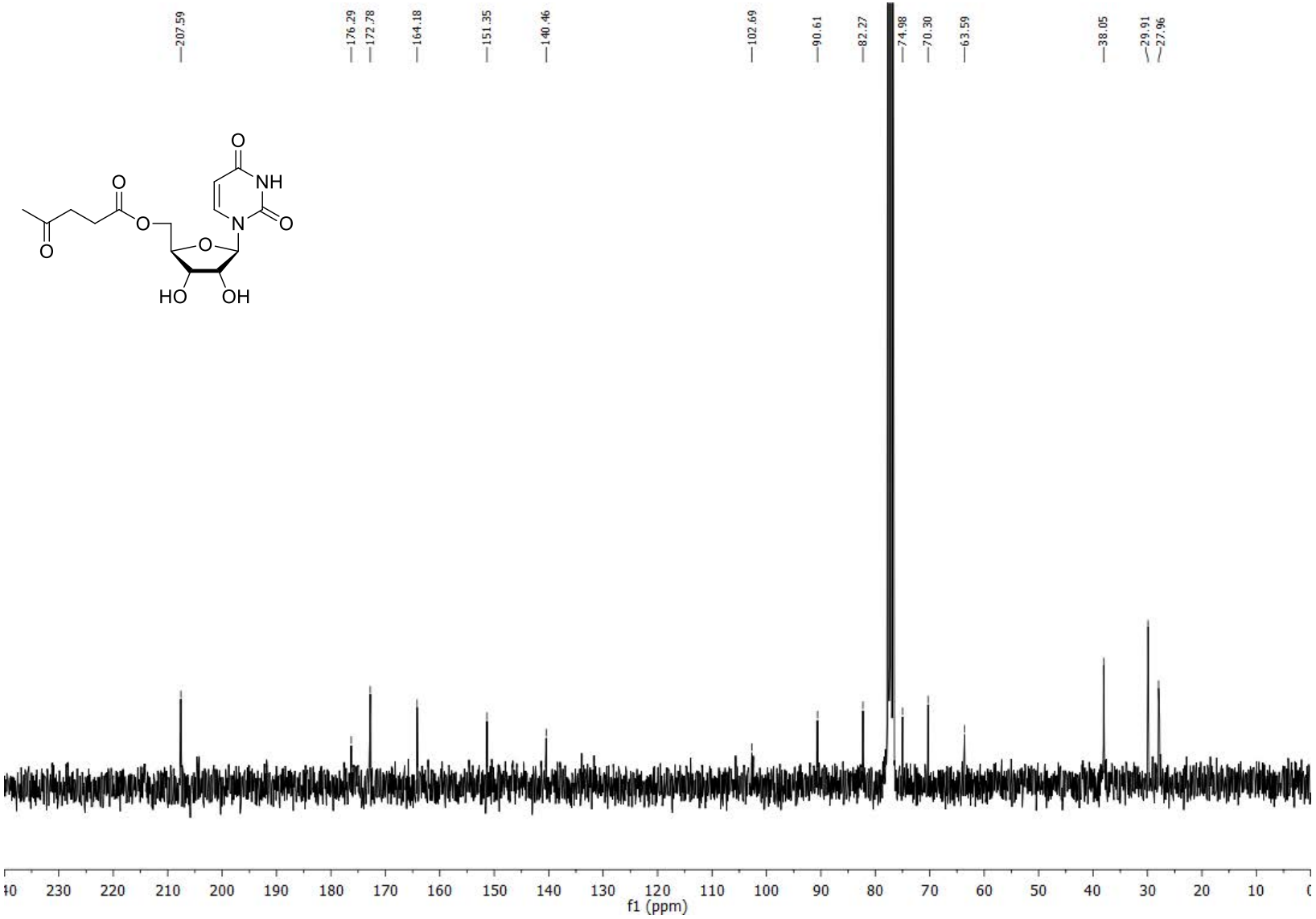
5'-*O*-Levulinyl-β-D-uridine (2a)

^{1}H-NMR (300.13 MHz, $CDCl_3$)

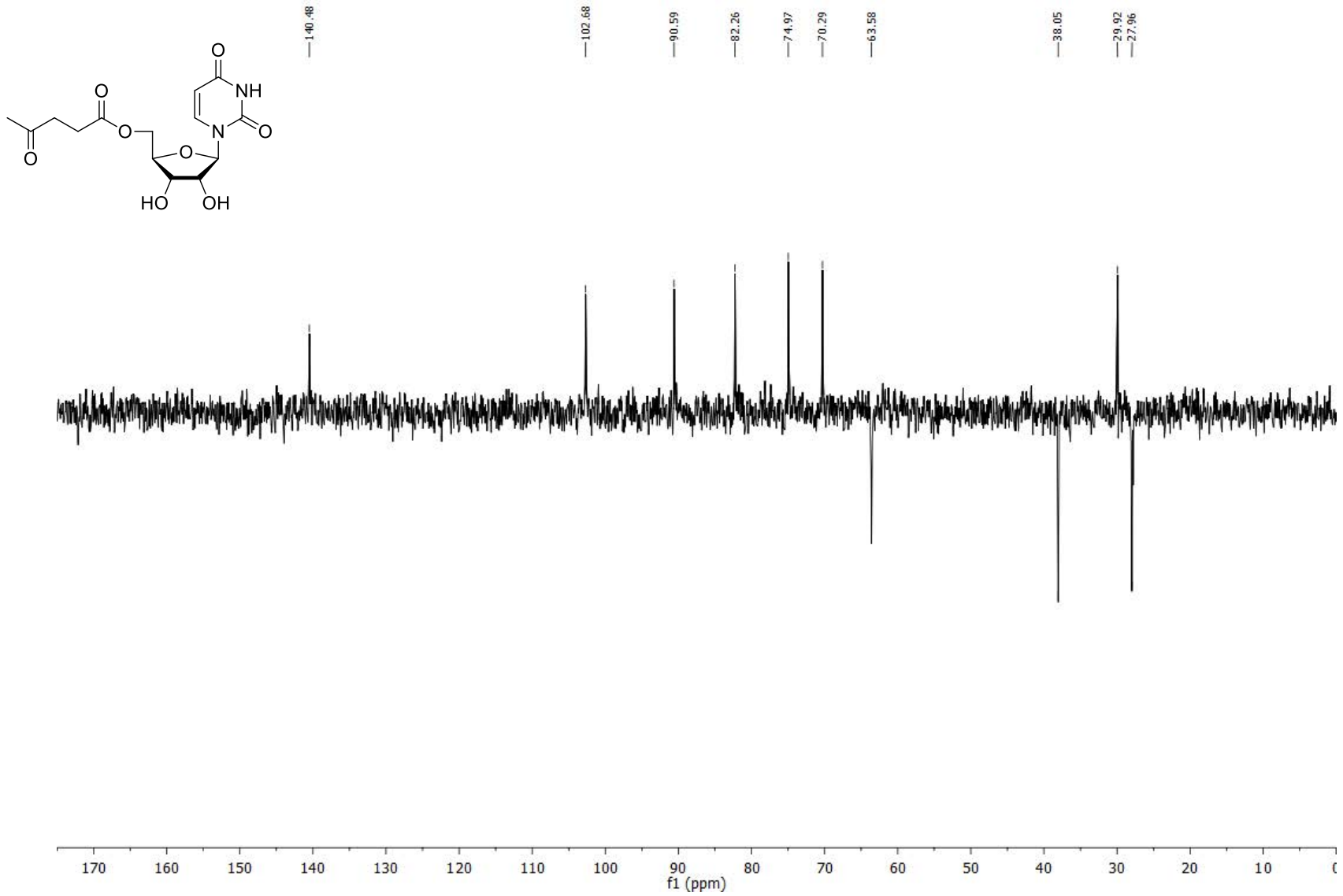




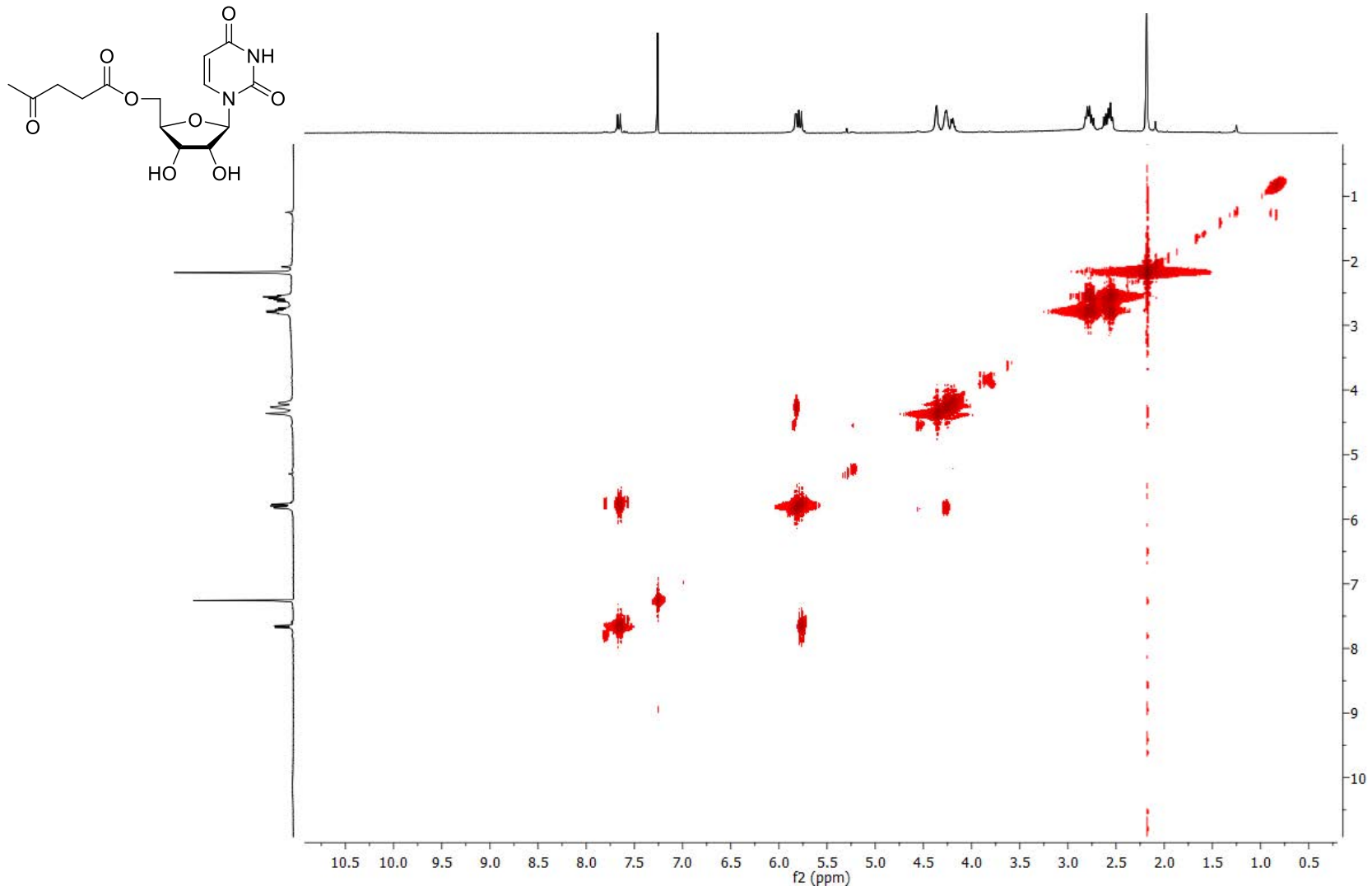
^{13}C-NMR (75.5 MHz, $CDCl_3$)



DEPT NMR (75.5 MHz, $CDCl_3$)

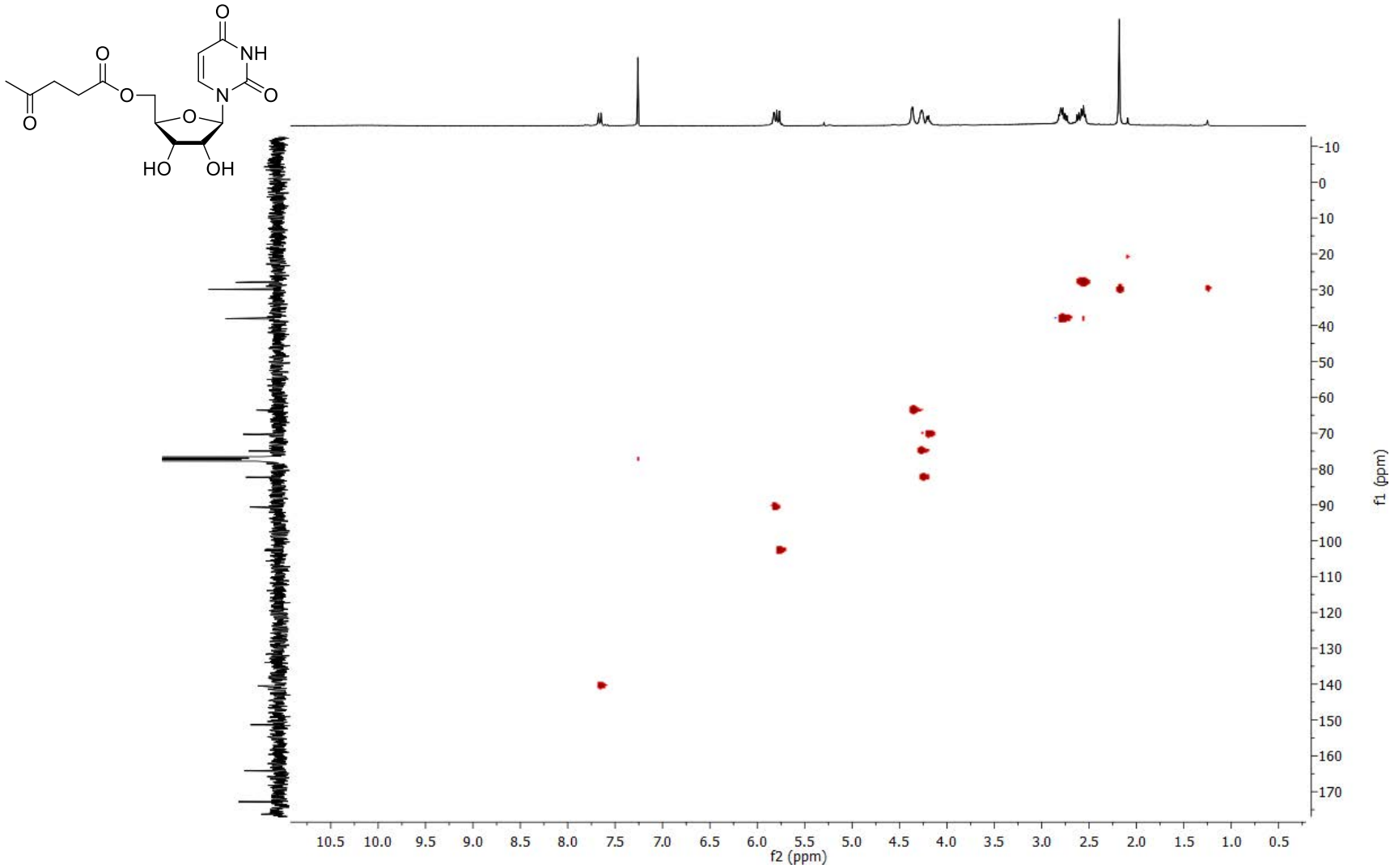


COSY NMR ($CDCl_3$)



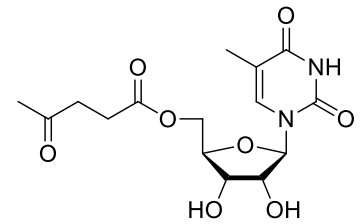
5'-*O*-Levulinyl-β-D-uridine (2a)

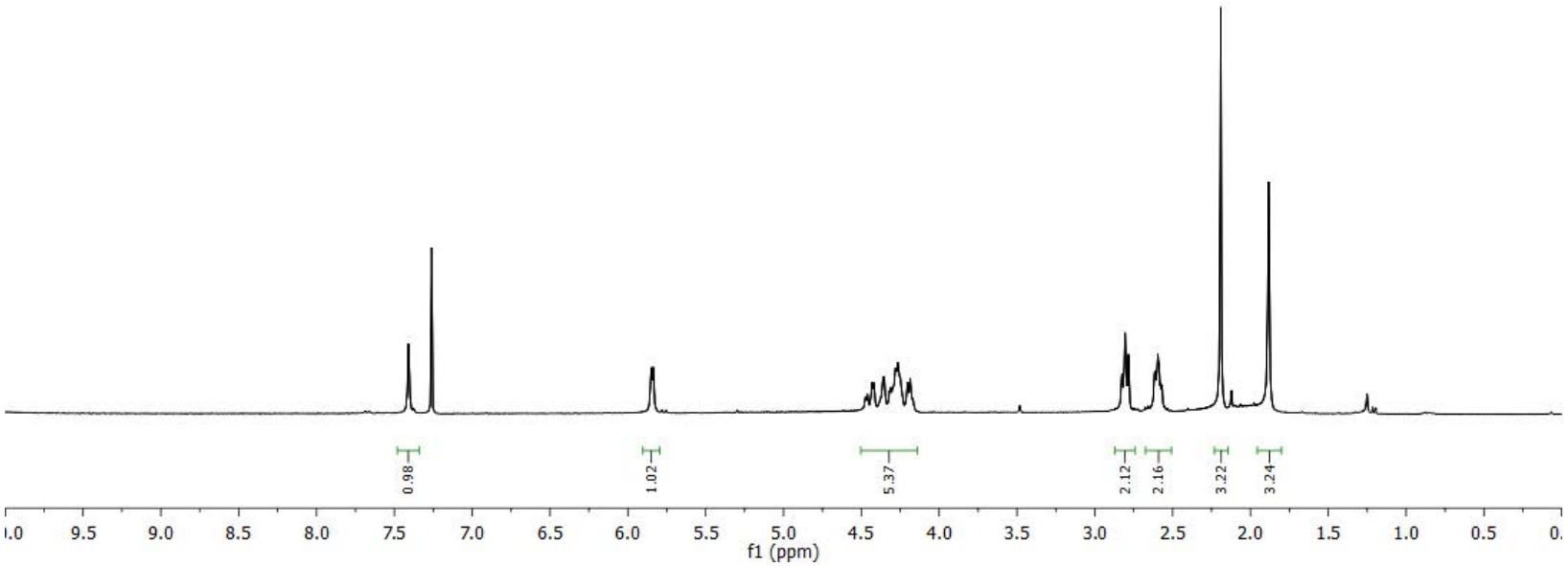
HSQC NMR ($CDCl_3$)



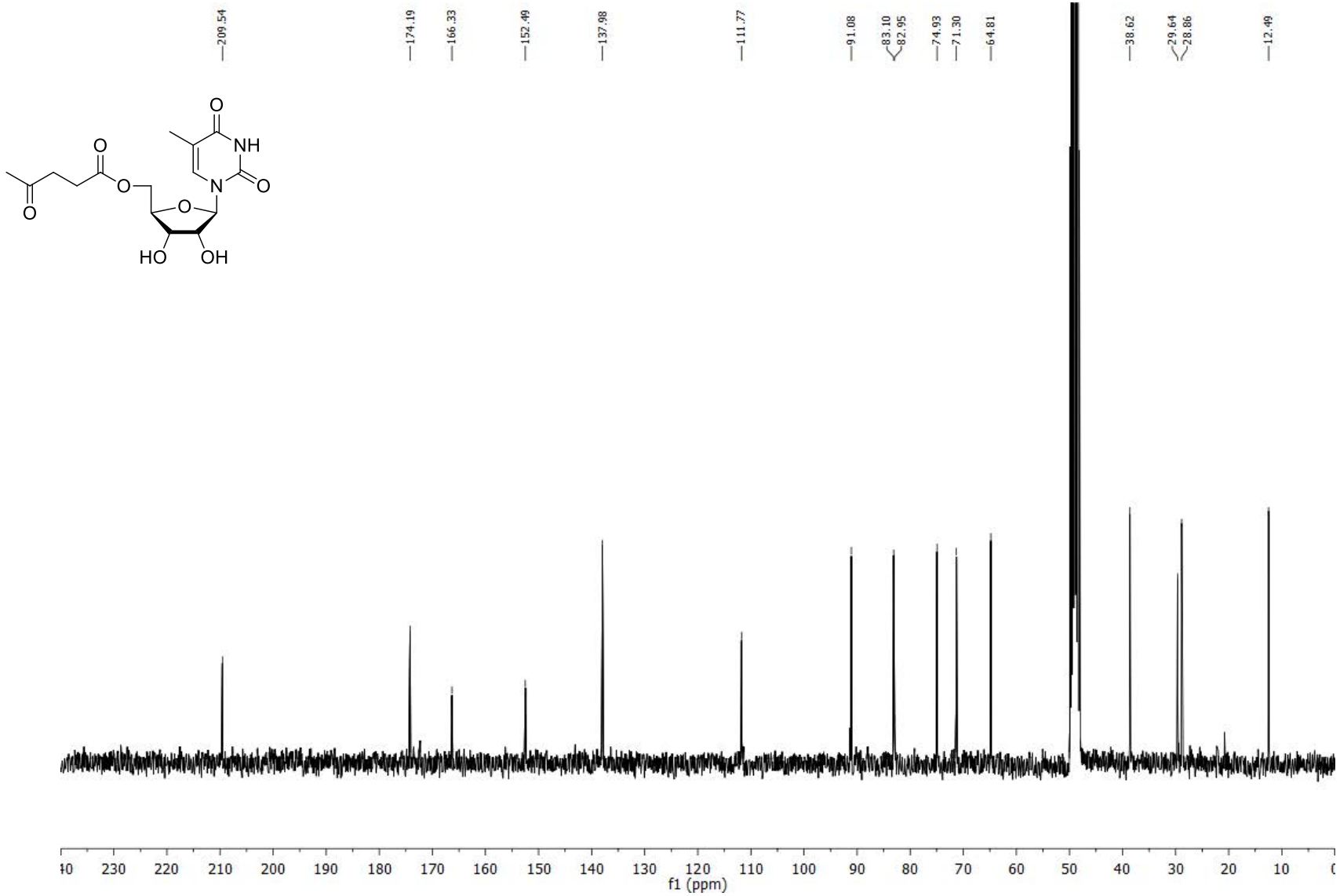
5'-*O*-Levulinyl-β-D-5-methyluridine (2b)

^{1}H-NMR (300.13 MHz, $CDCl_3$)

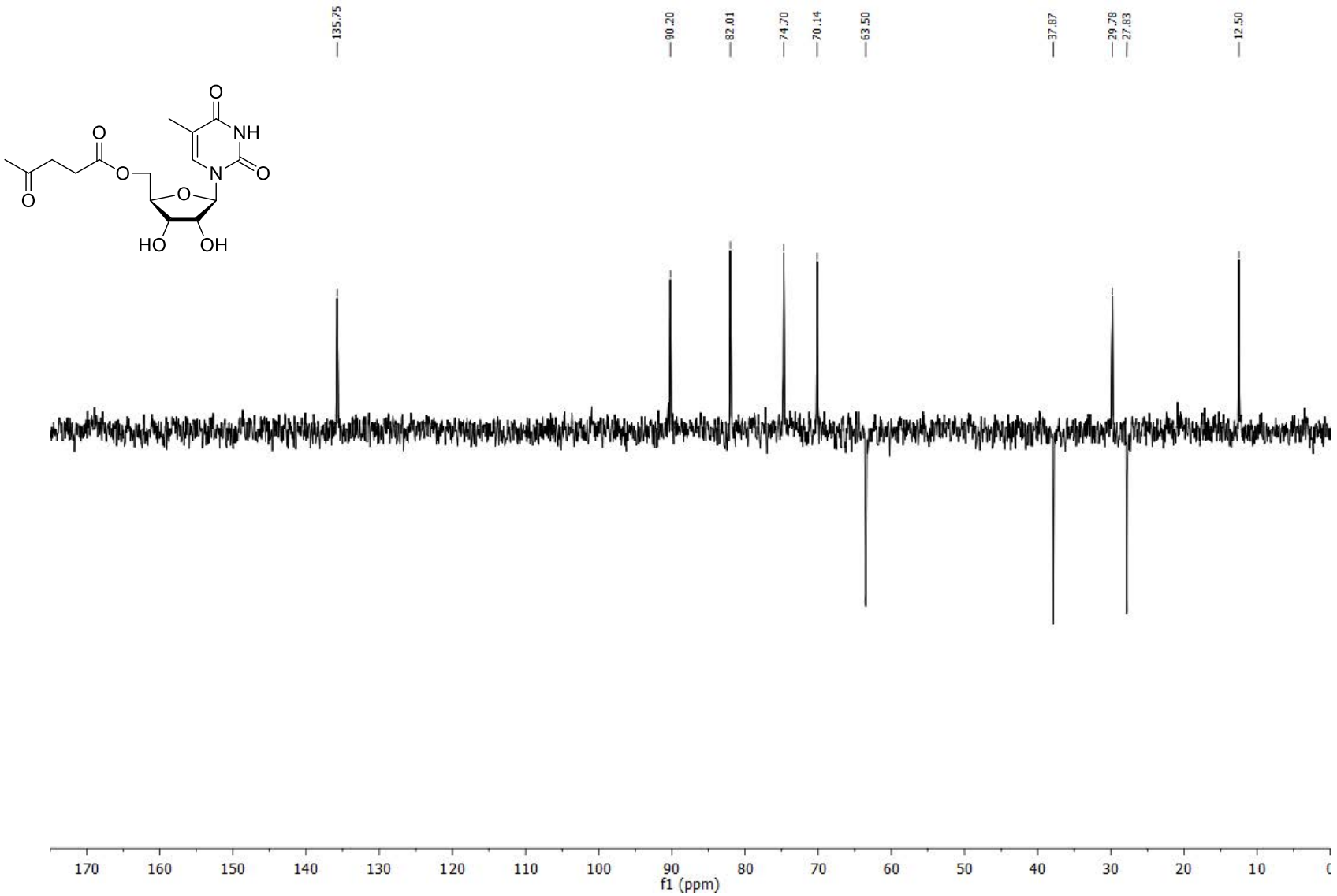




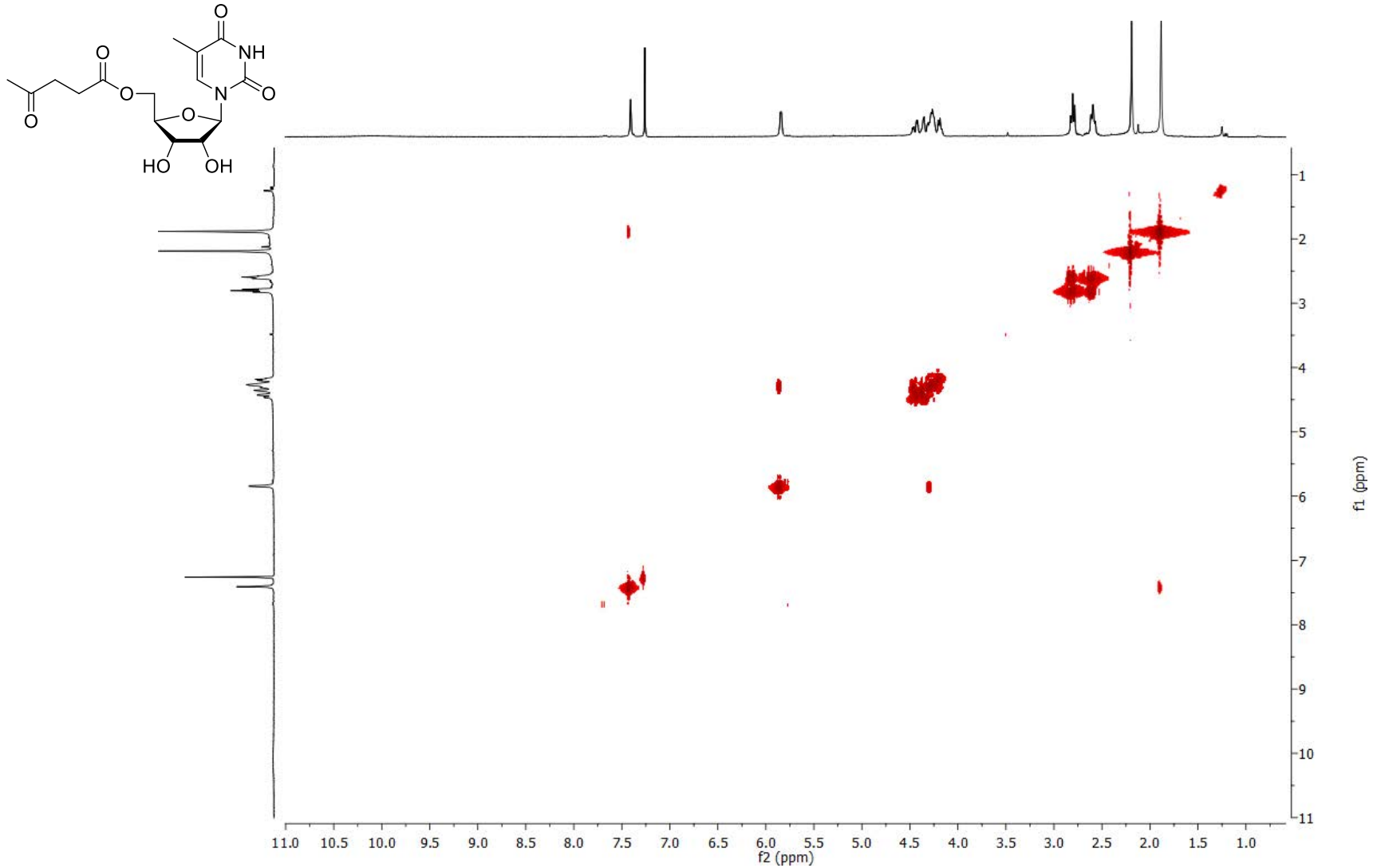
^{13}C-NMR (75.5 MHz, MeOH-d_4)



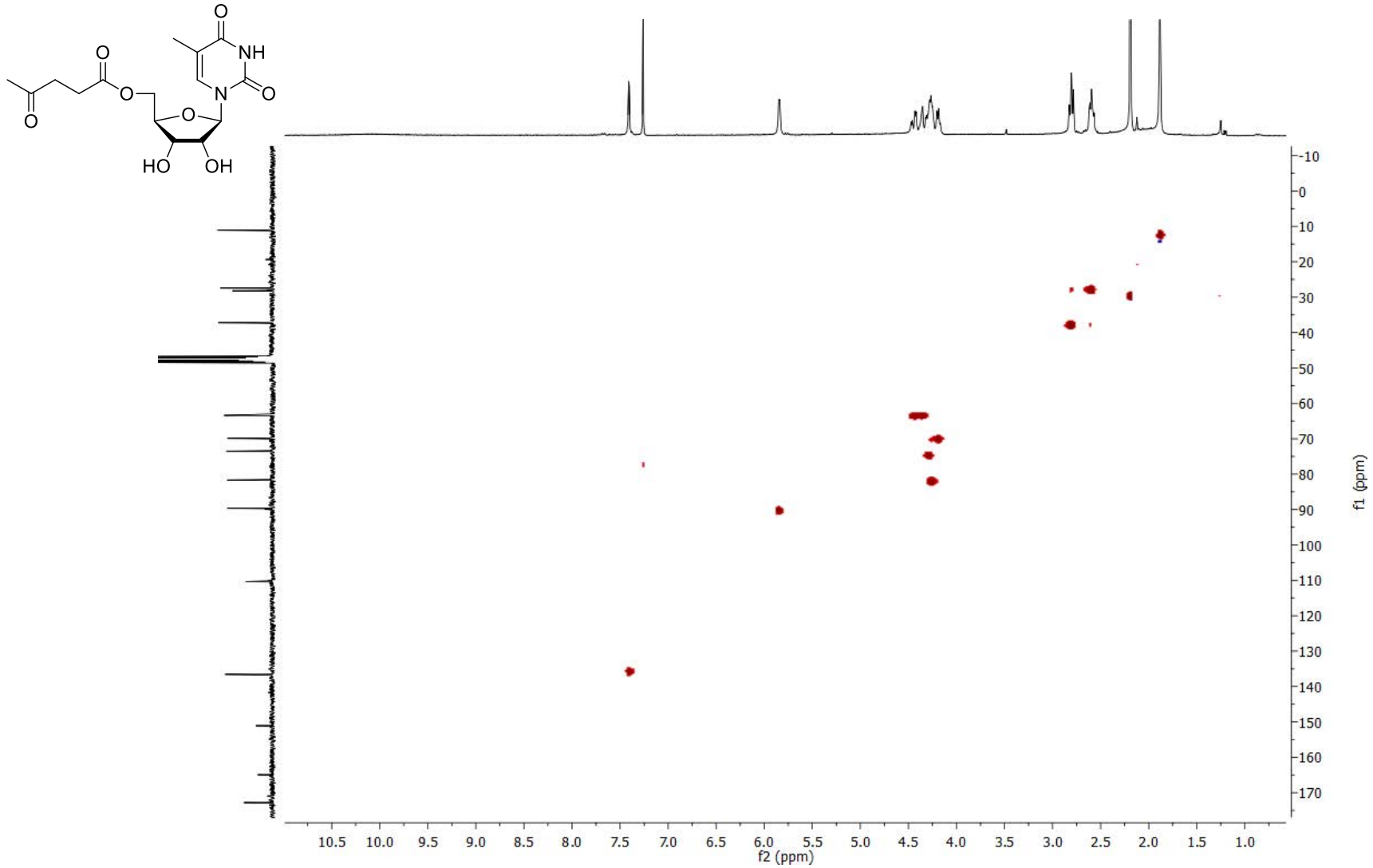
DEPT NMR (75.5 MHz, $CDCl_3$)



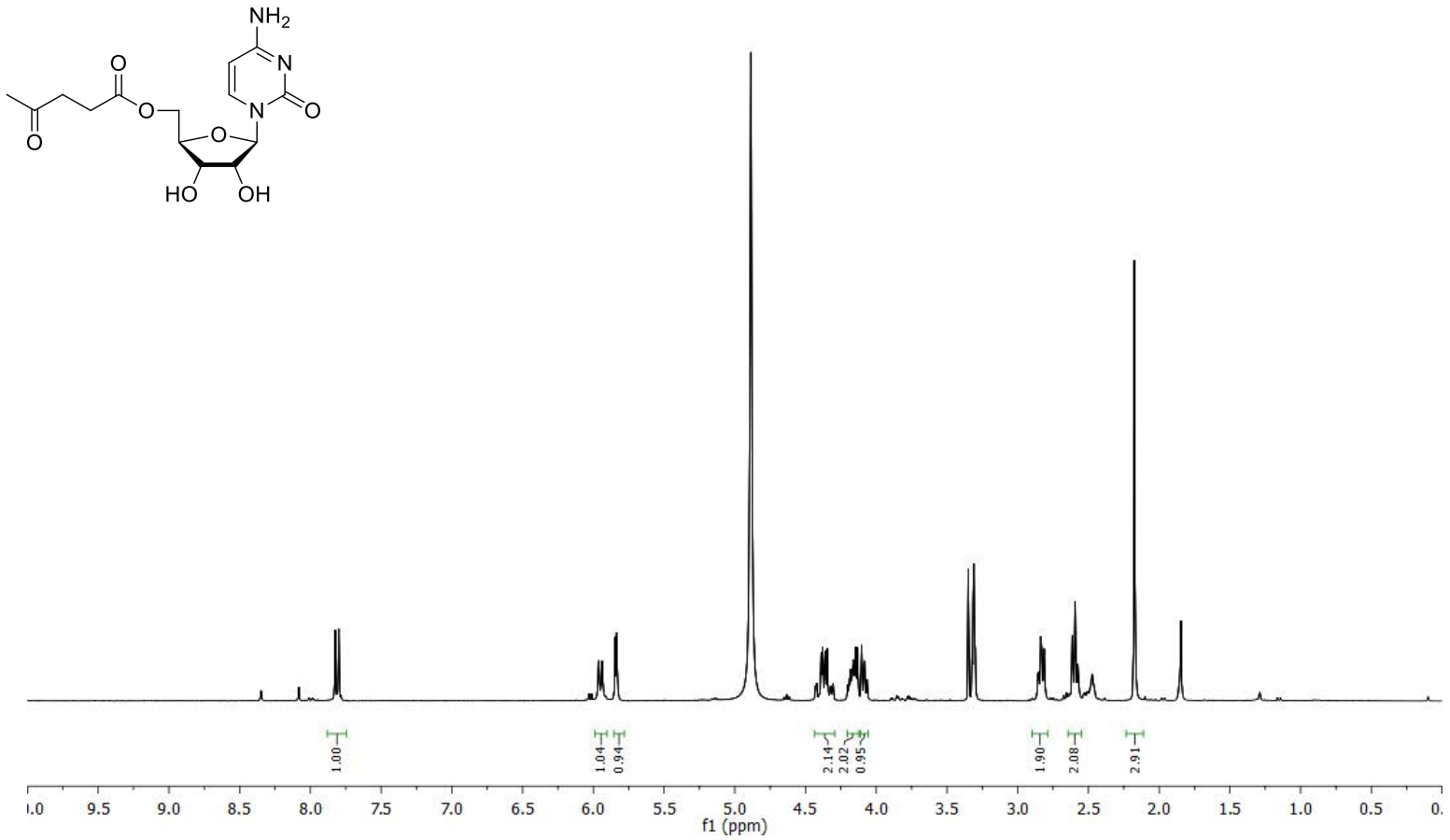
COSY NMR ($CDCl_3$)



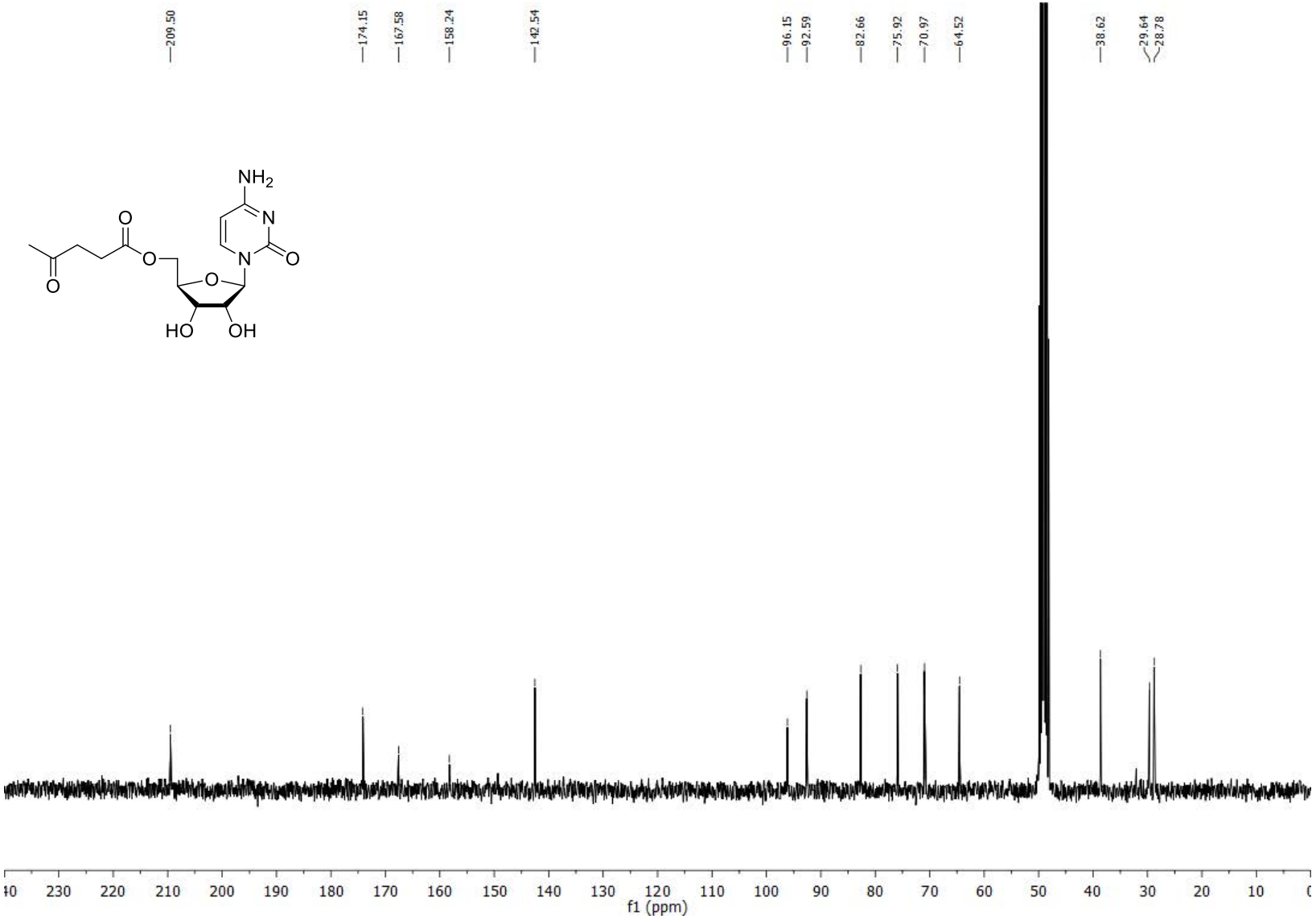
HSQC NMR ($CDCl_3$)



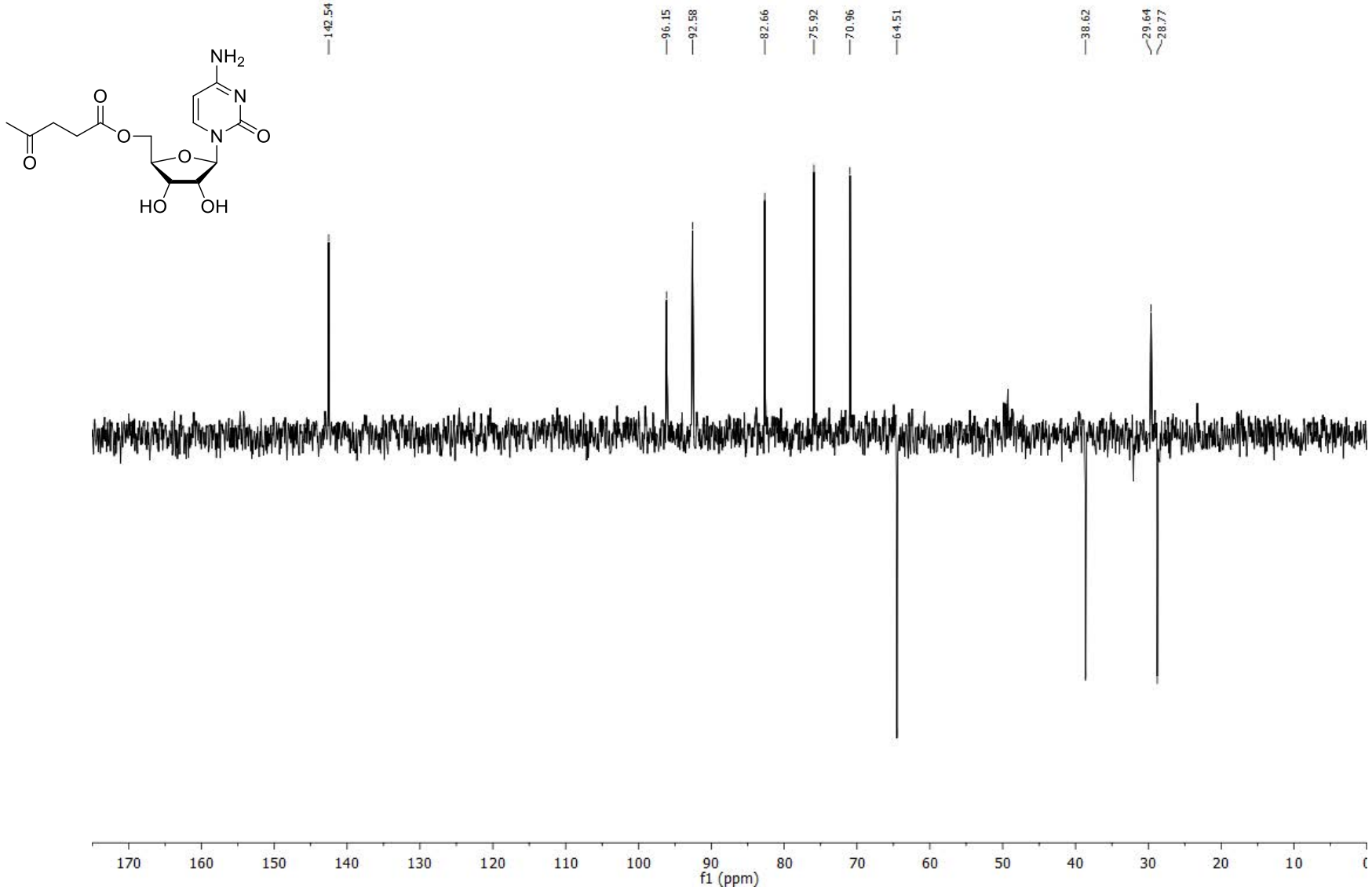
^{1}H-NMR (300.13 MHz, MeOD-d_4)



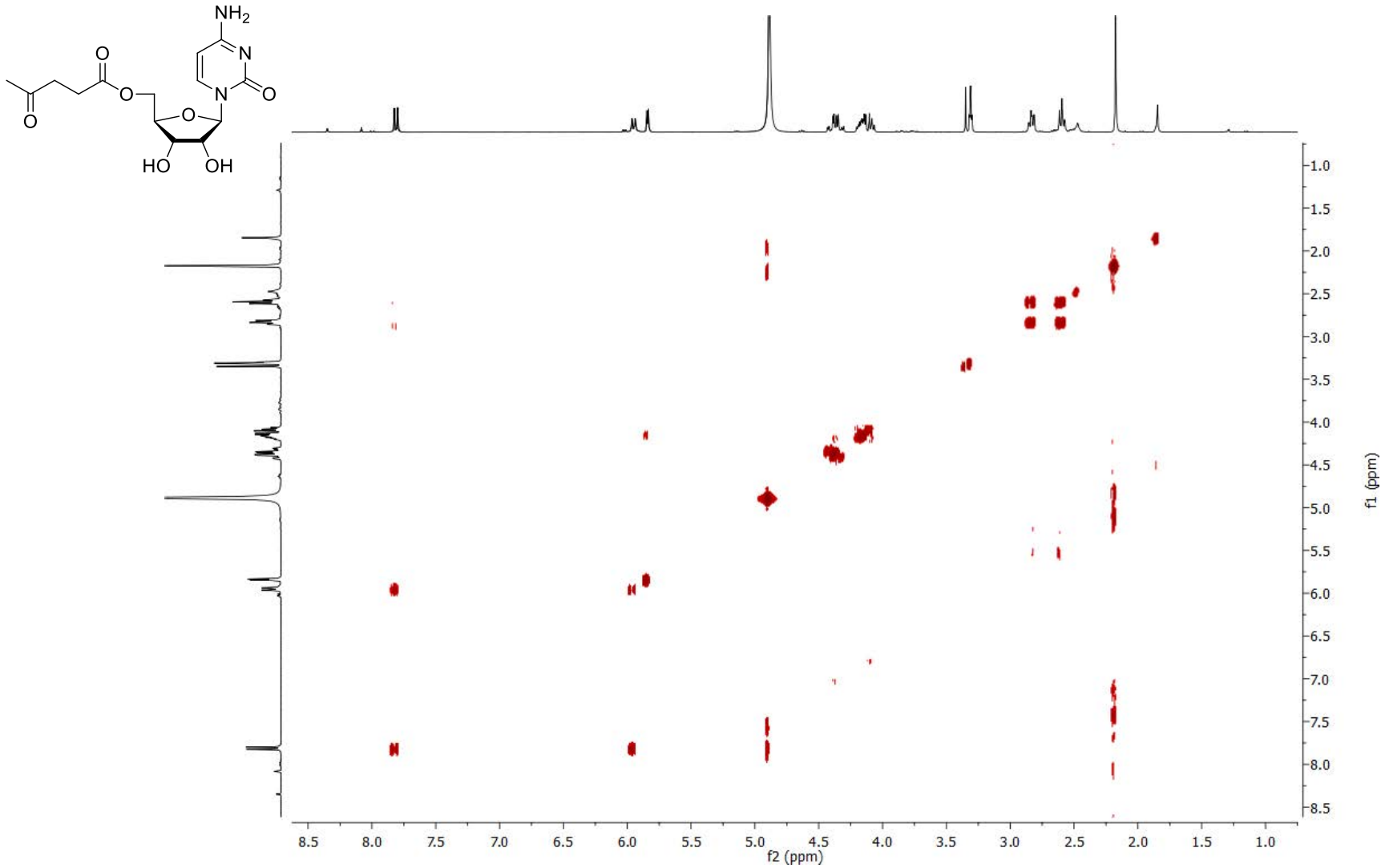
^{13}C-NMR (75.5 MHz, MeOD-d_4)



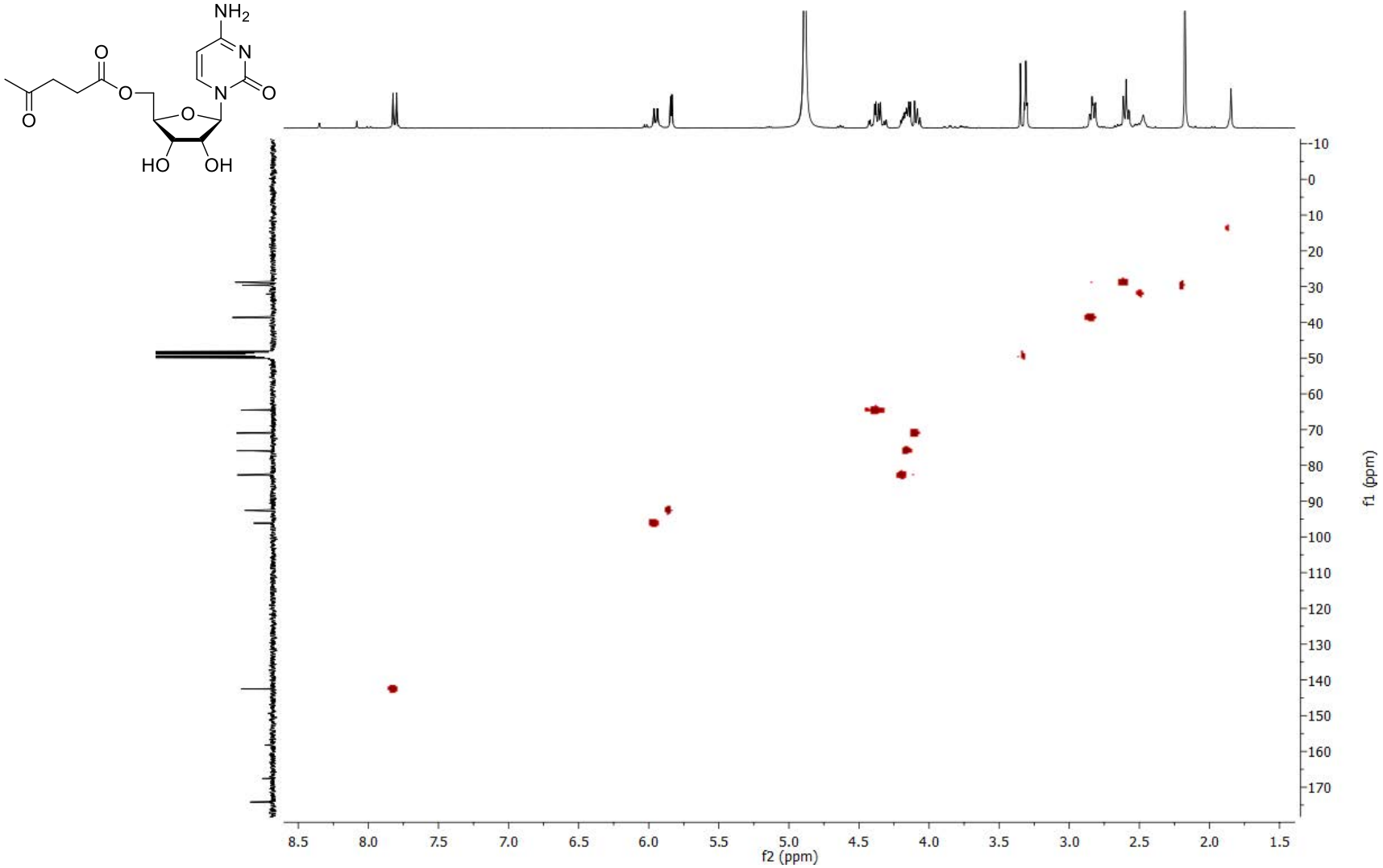
DEPT NMR (75.5 MHz, MeOD-d_4)



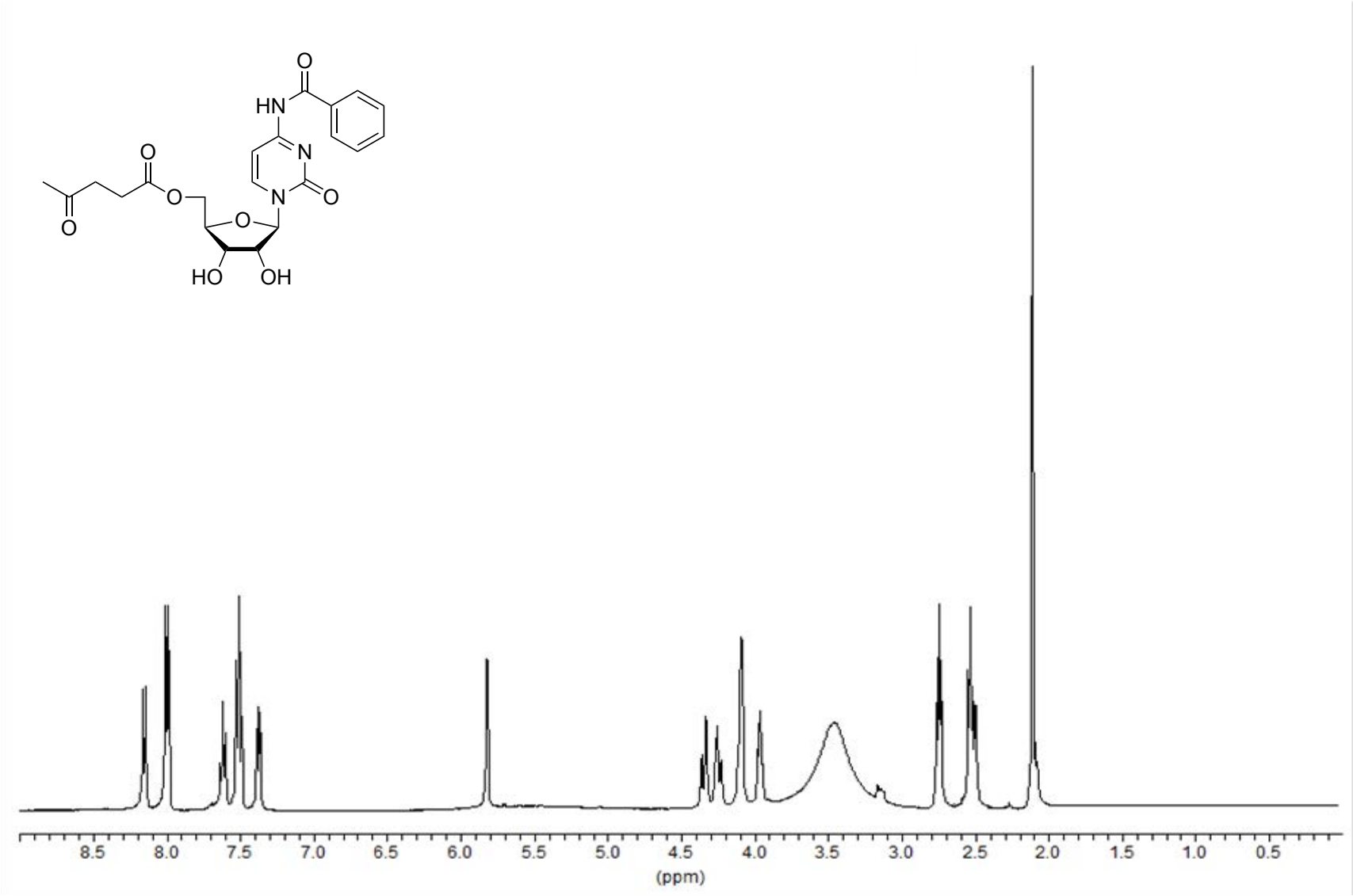
COSY NMR (MeOD-d_4)



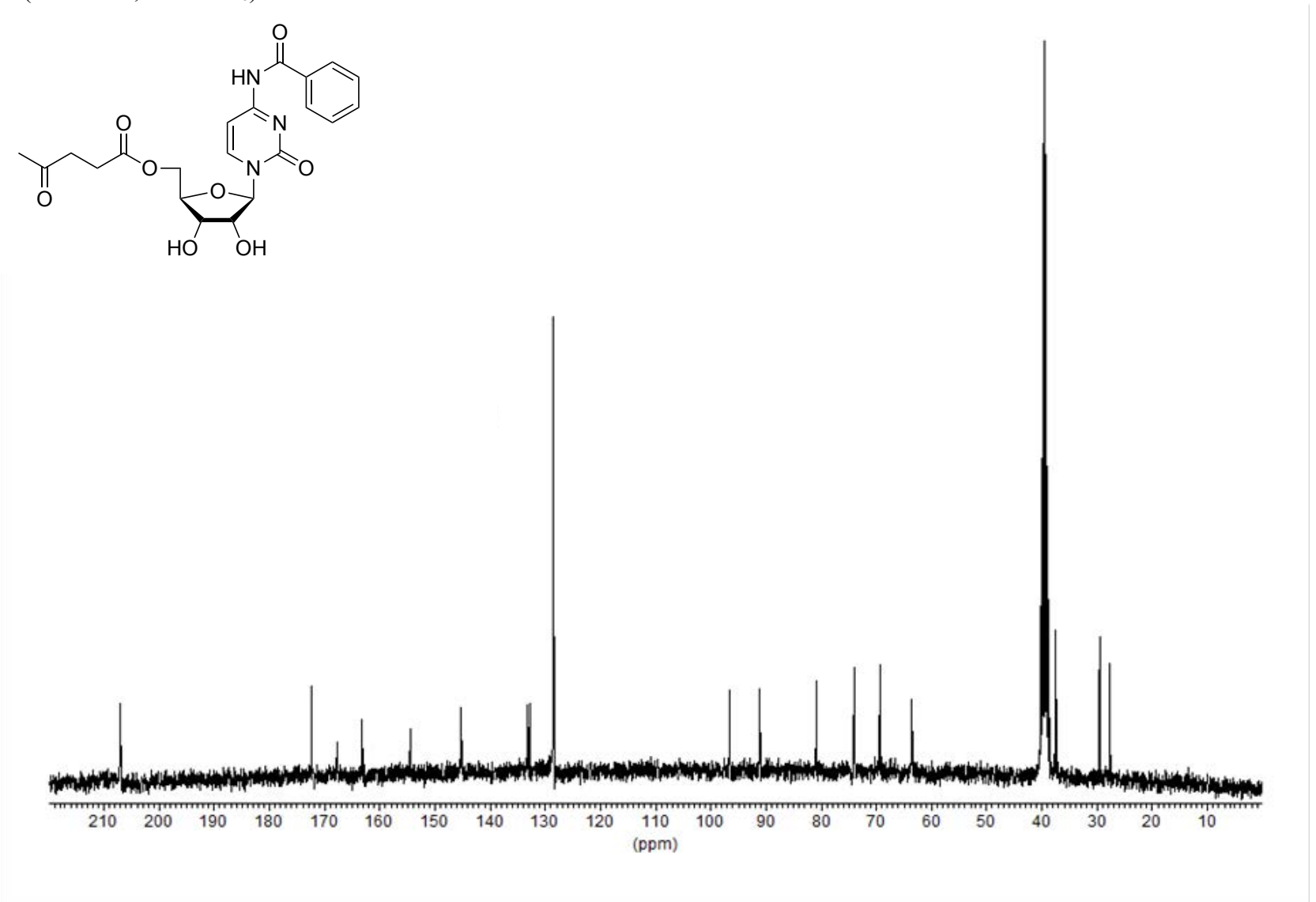
HSQC NMR (MeOD-d_4)



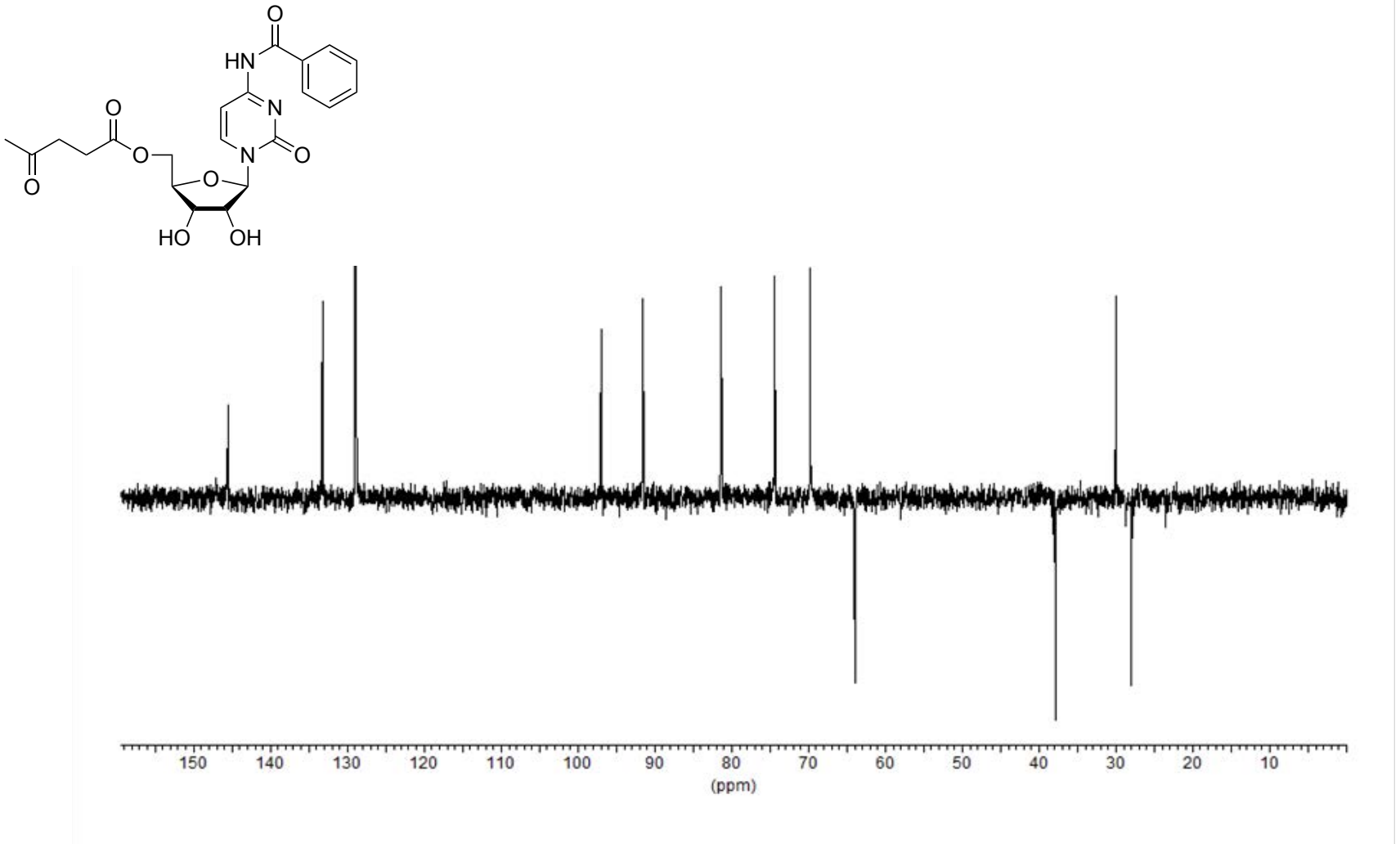
^{1}H-NMR (300.13 MHz, DMSO-d_6)



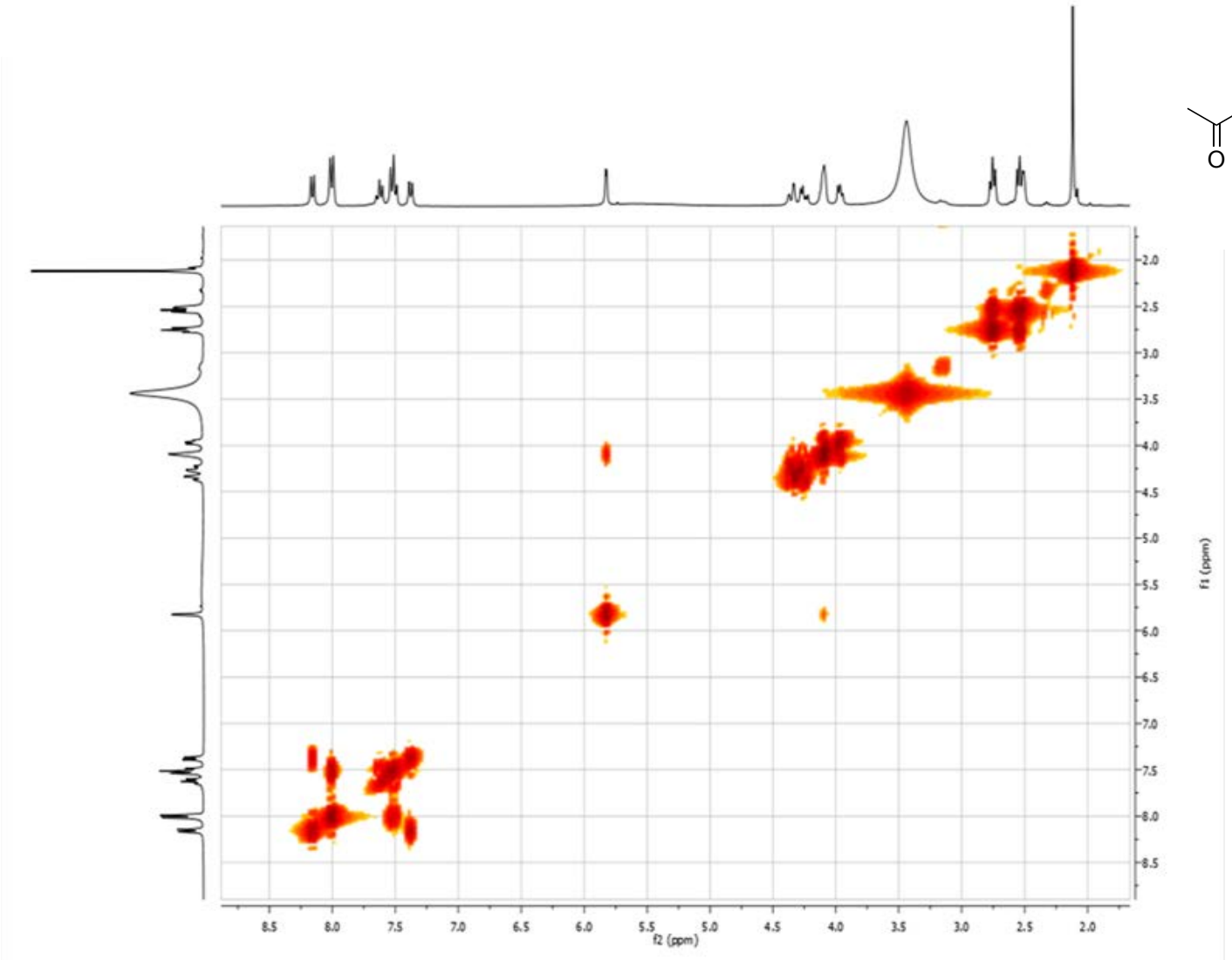
^{13}C-NMR (75.5 MHz, DMSO-d_6)

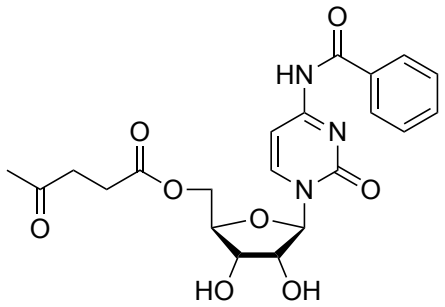


DEPT NMR (75.5 MHz, DMSO-d_6)

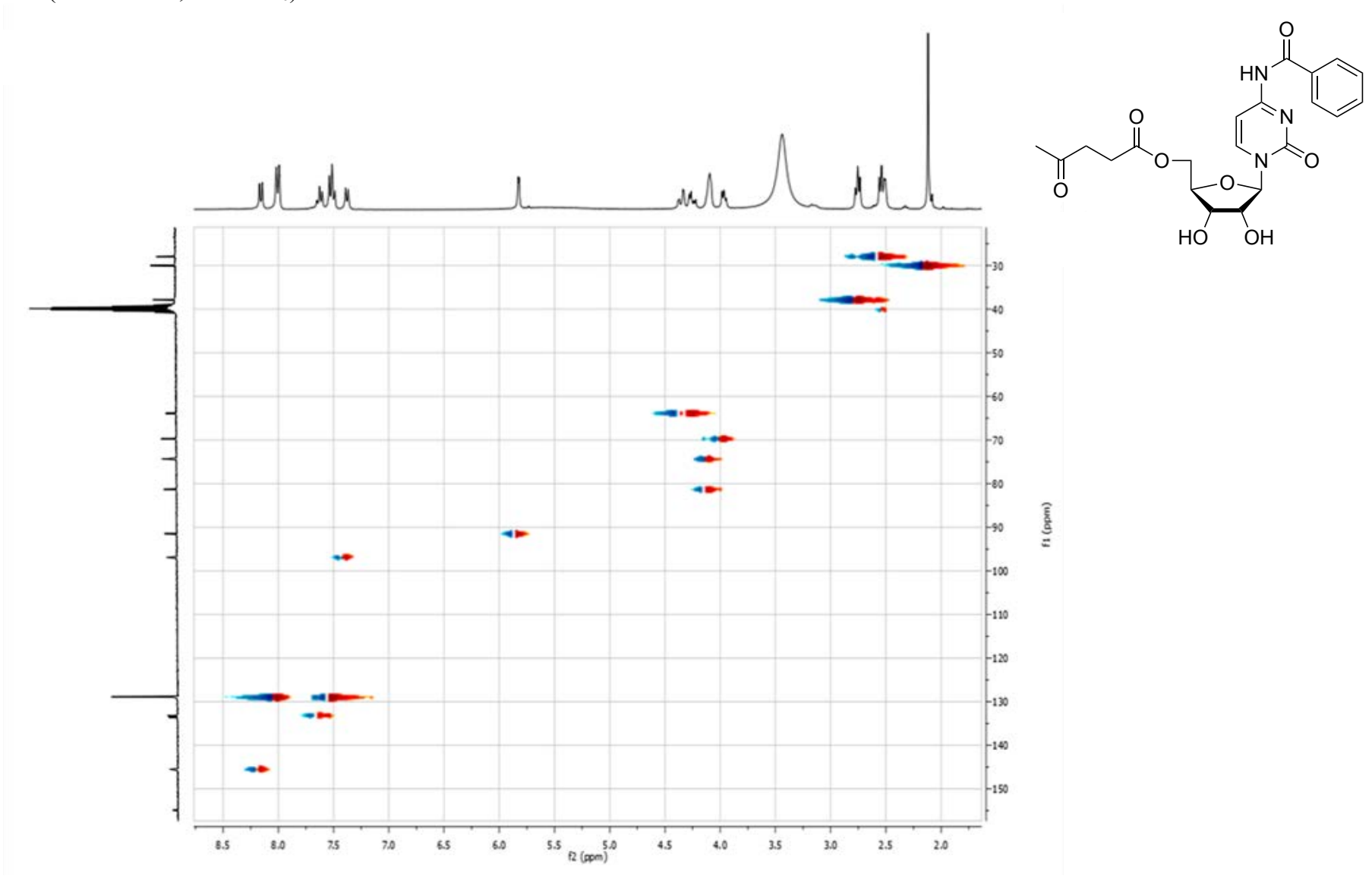


COSY NMR (300.13 MHz, DMSO-d_6)

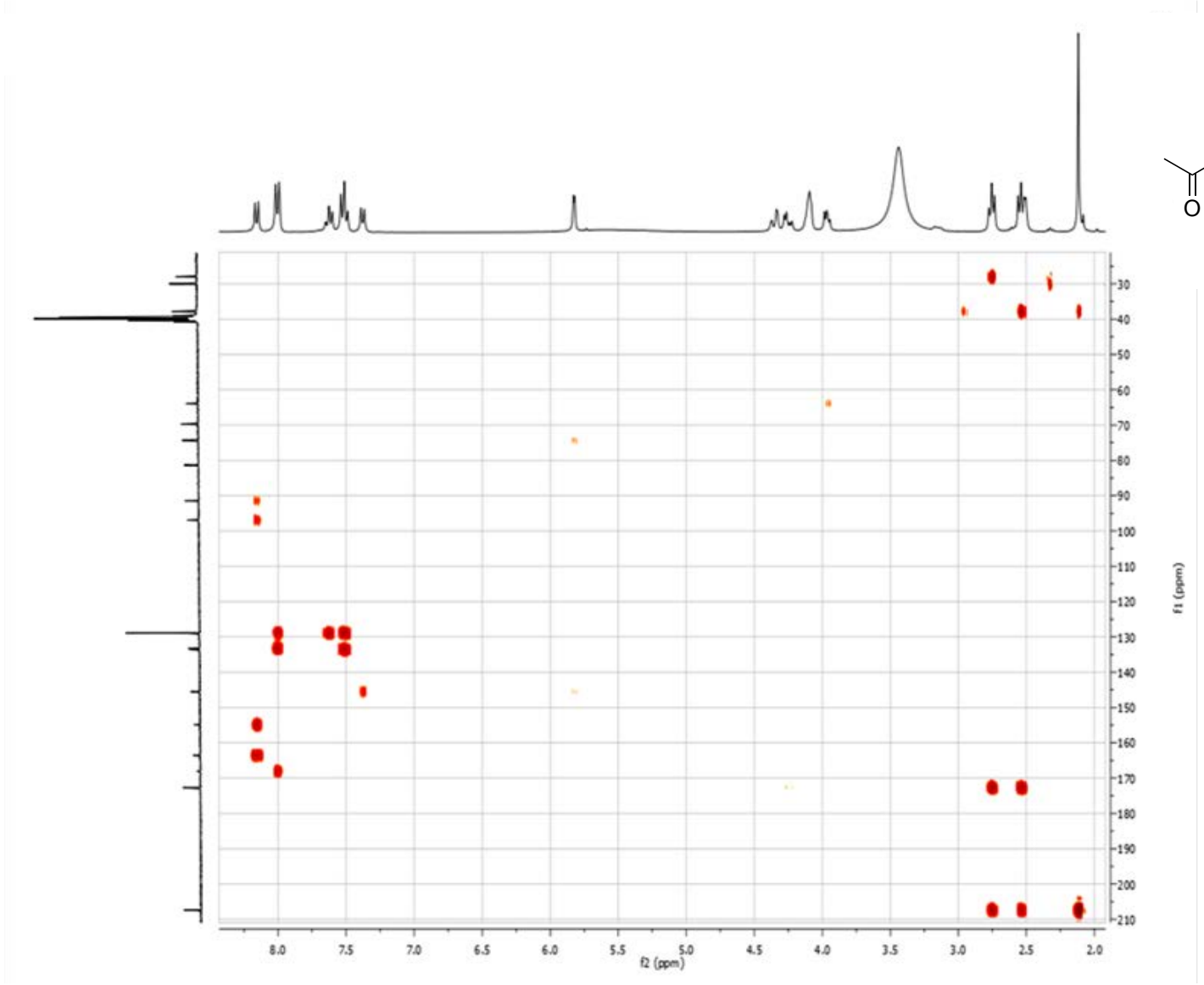


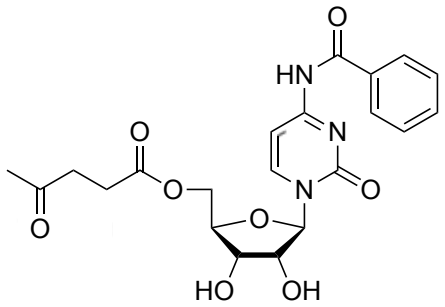


HSQC NMR (300.13 MHz, DMSO-d_6)

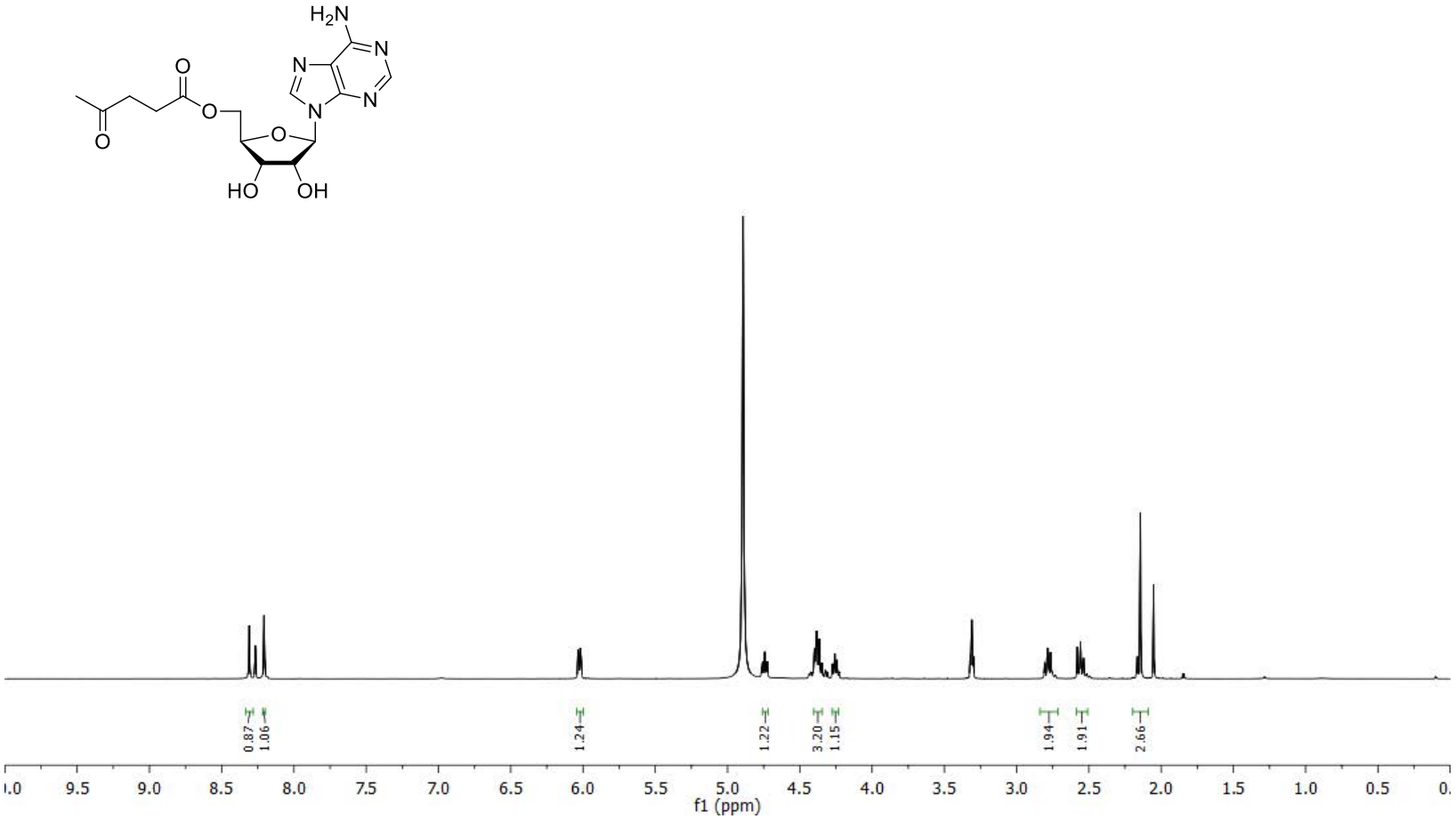


HMBC NMR (300.13 MHz, DMSO-d_6)

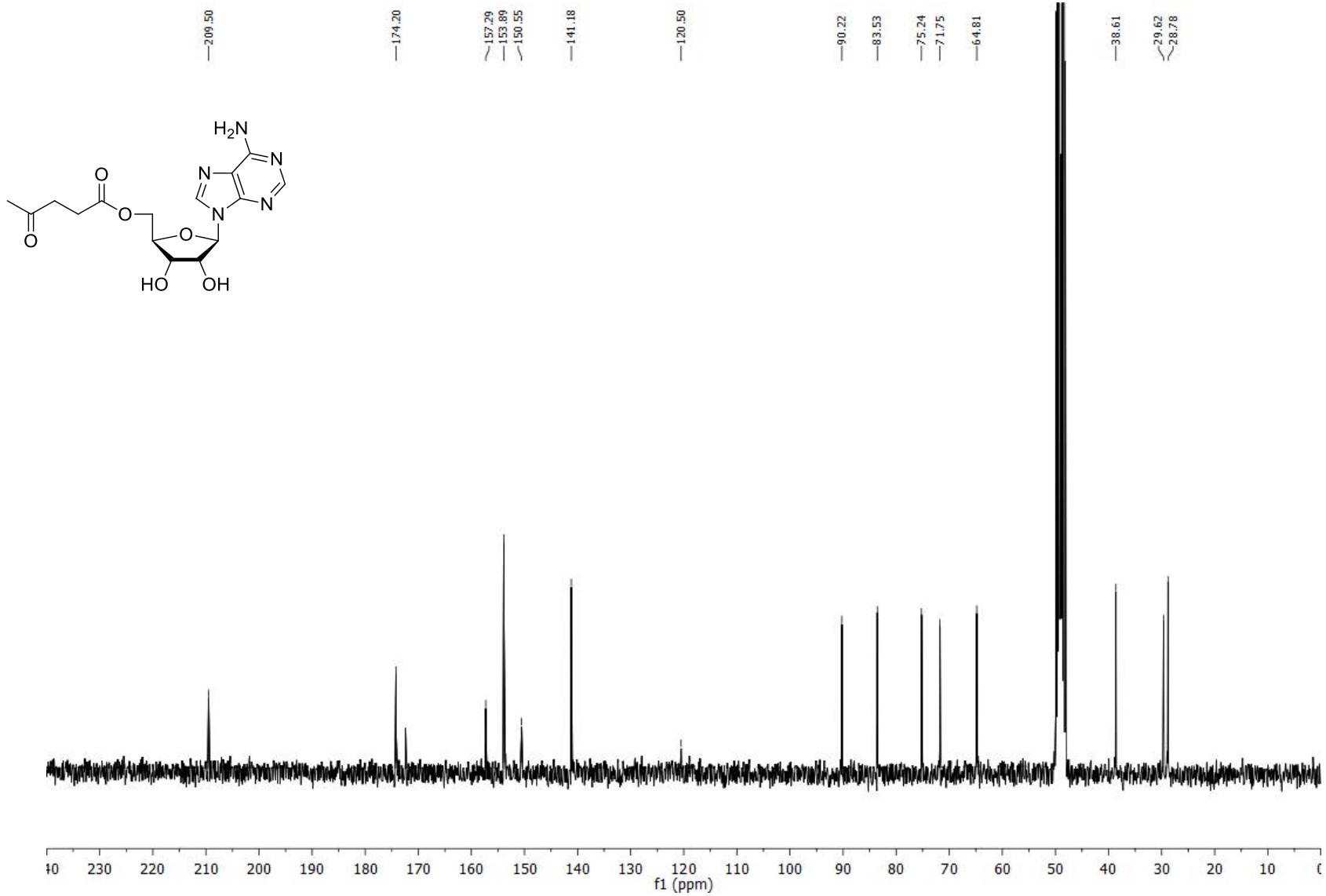




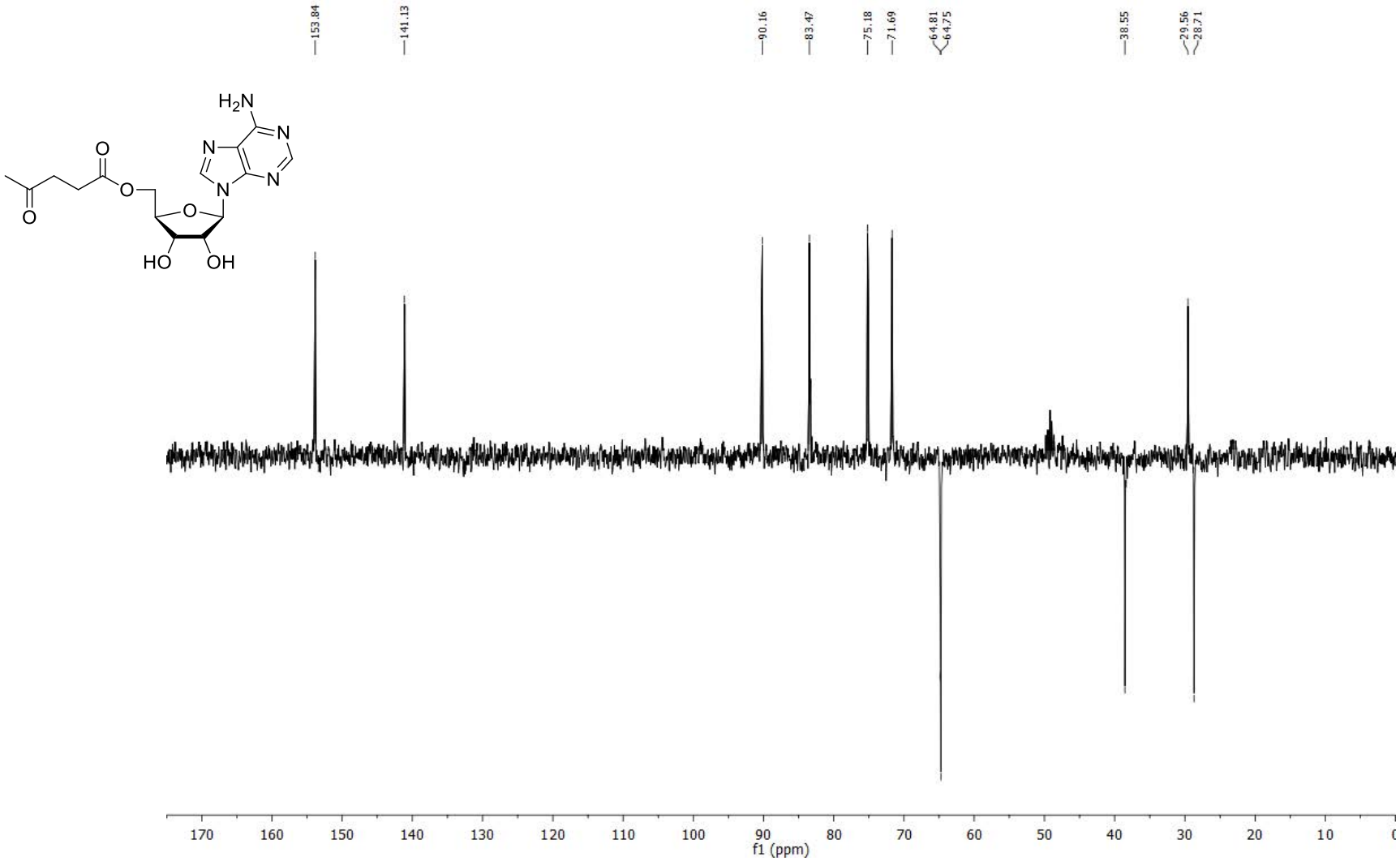
^{1}H-NMR (300.13 MHz, MeOD-d_4)



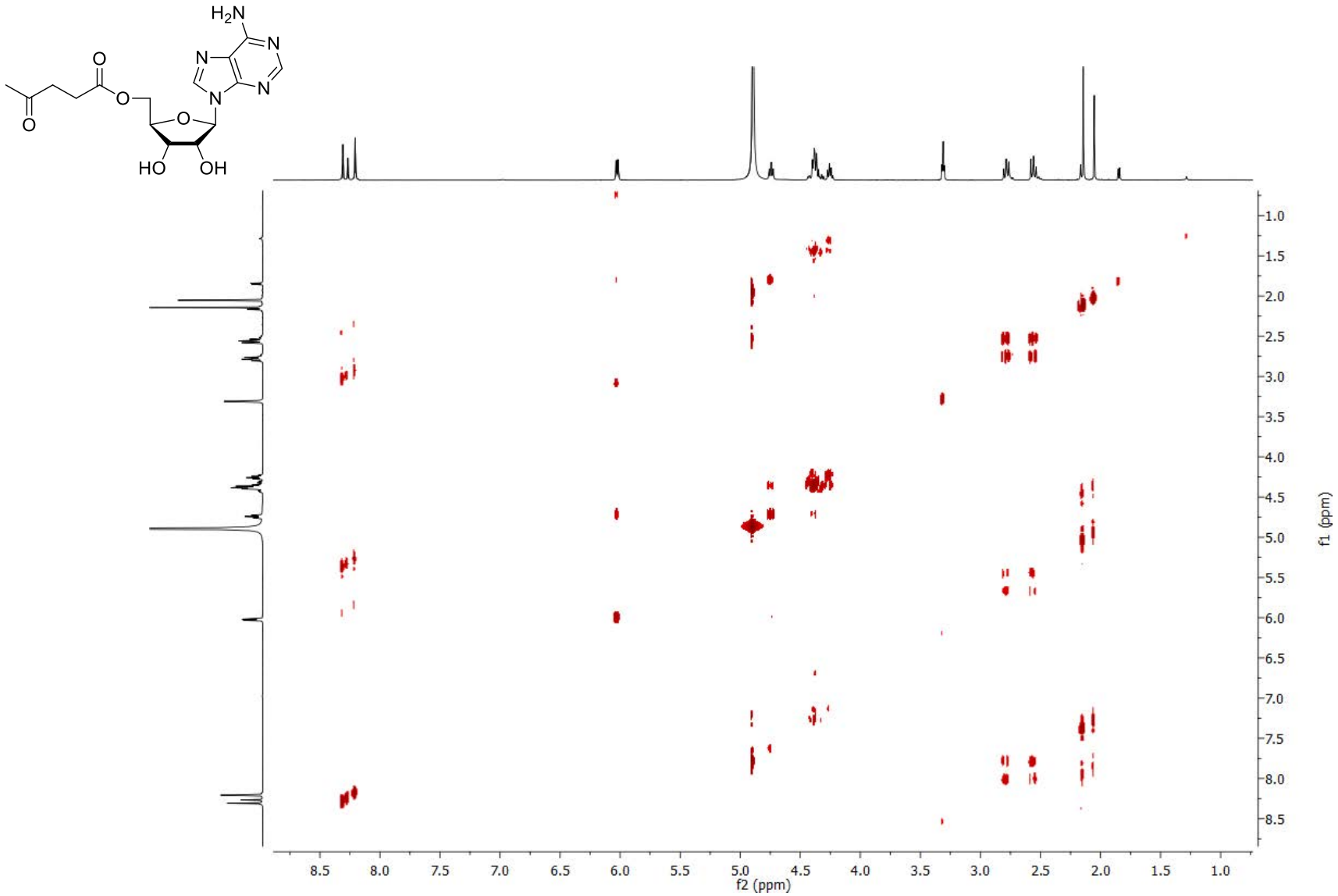
^{13}C-NMR (75.5 MHz, MeOD-d_4)



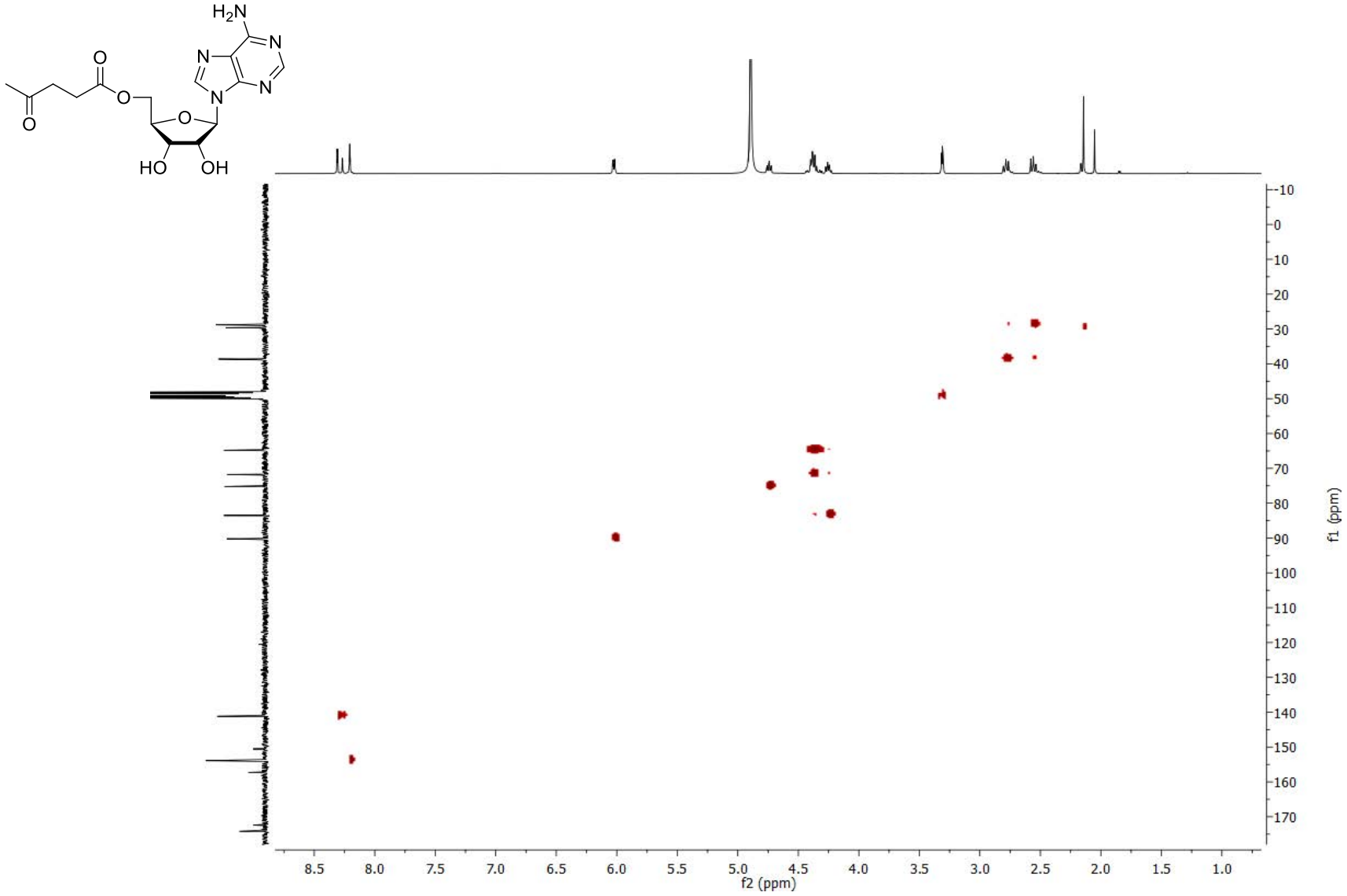
DEPT NMR (75.5 MHz, MeOD-d_4)



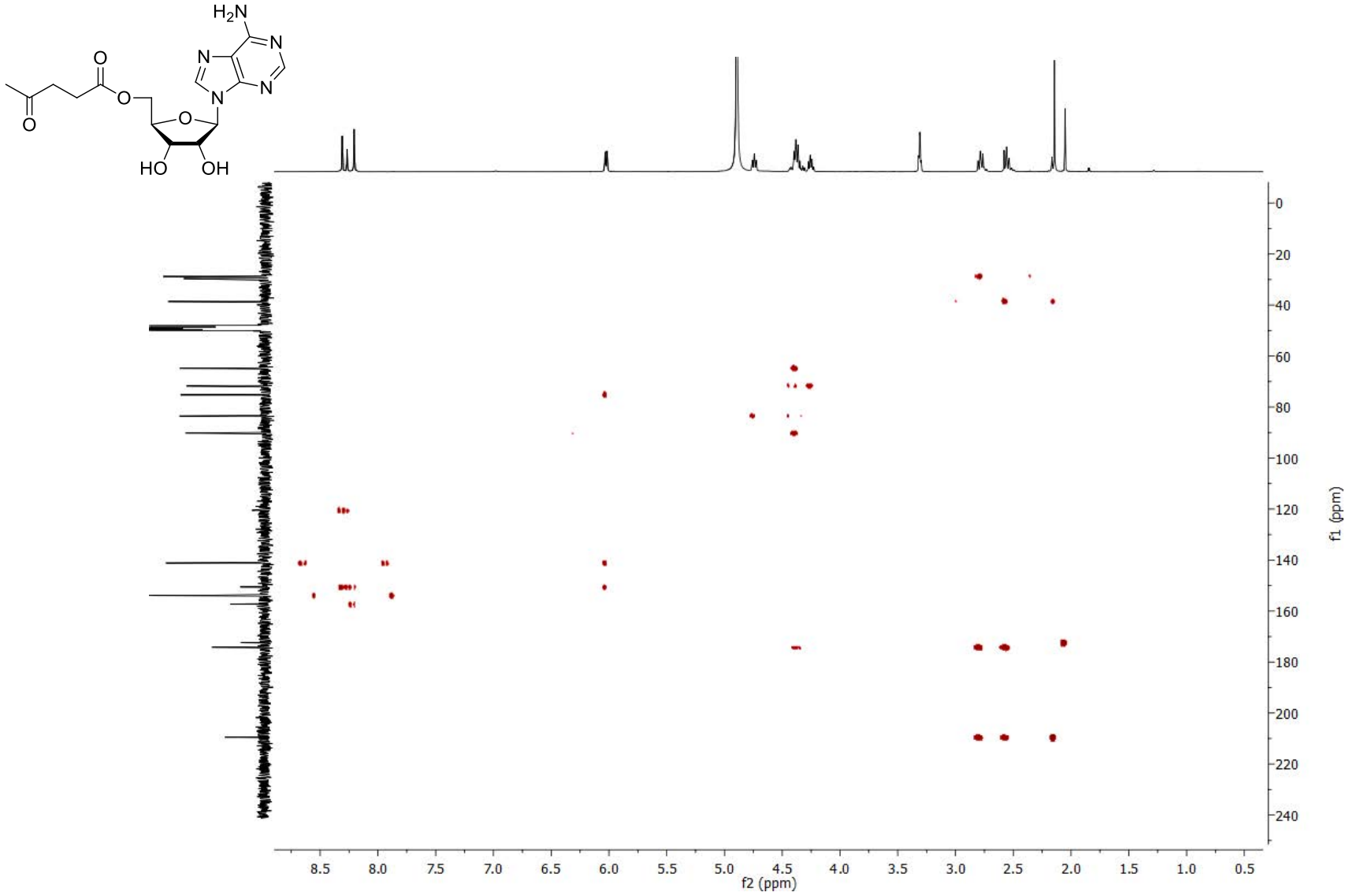
COSY NMR (MeOD-d_4)



HSQC NMR (MeOD-d_4)

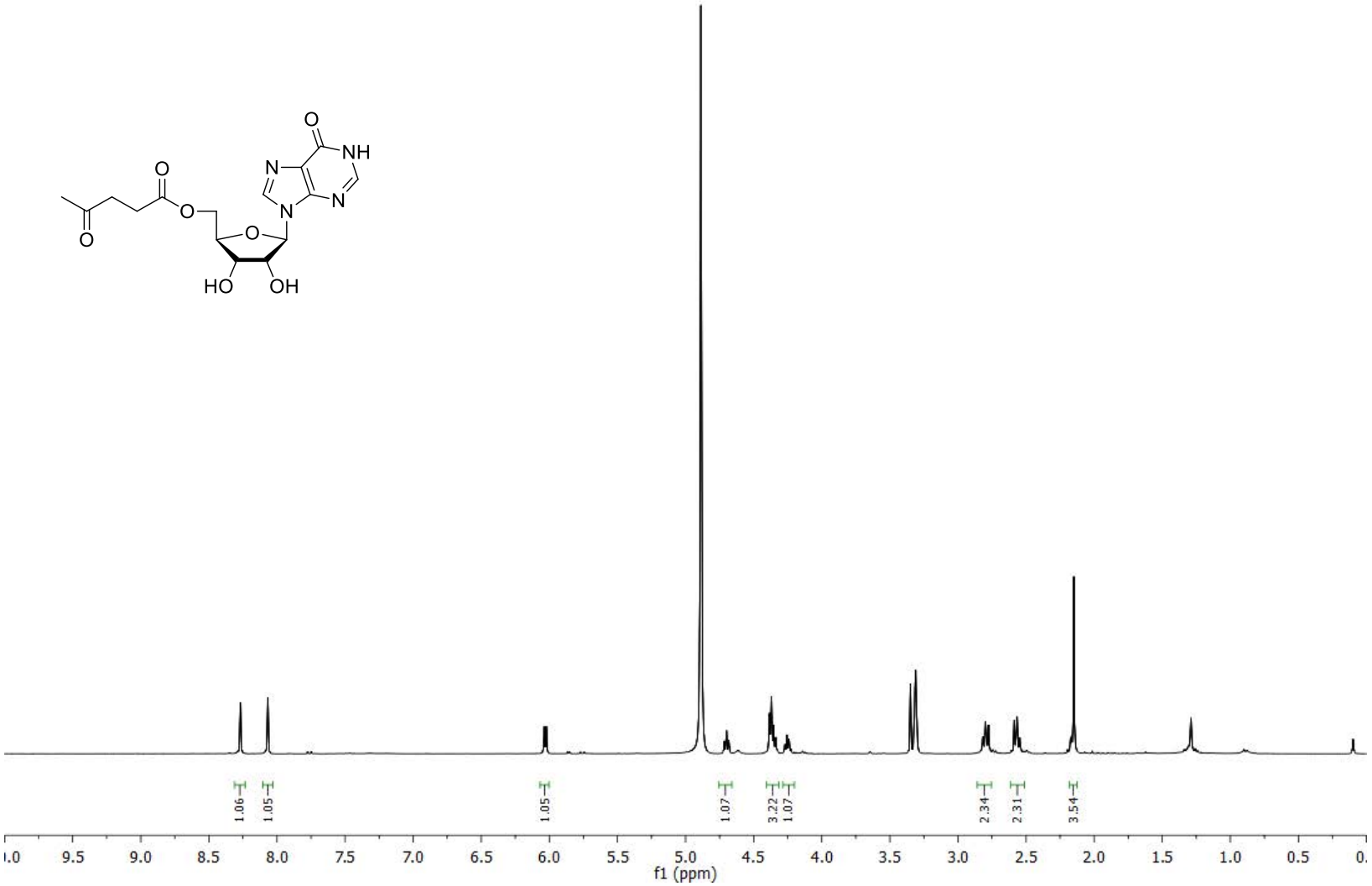


HMBC NMR (MeOD-d_4)

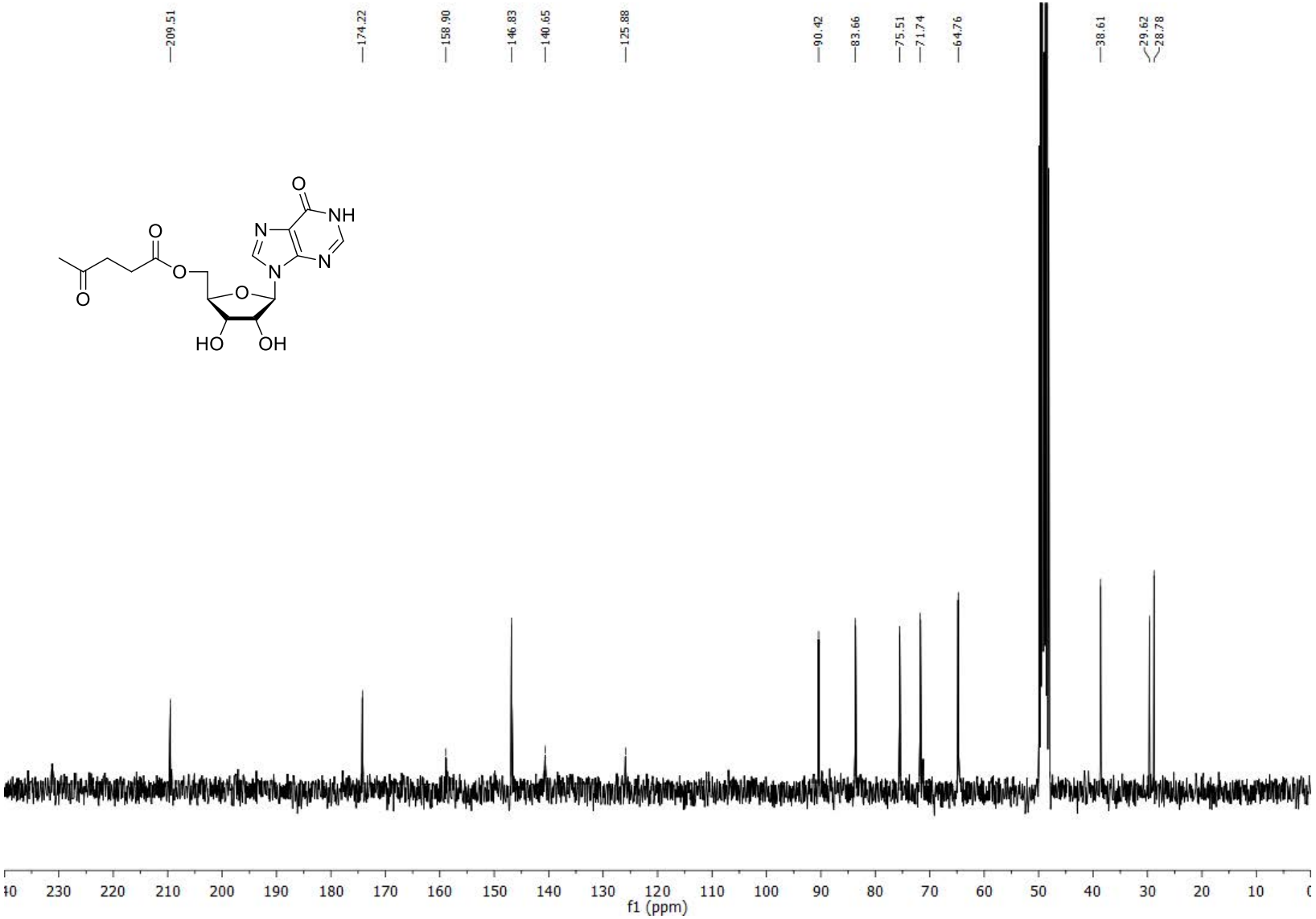


5'-*O*-Levulinyl-β-D-inosine (2f)

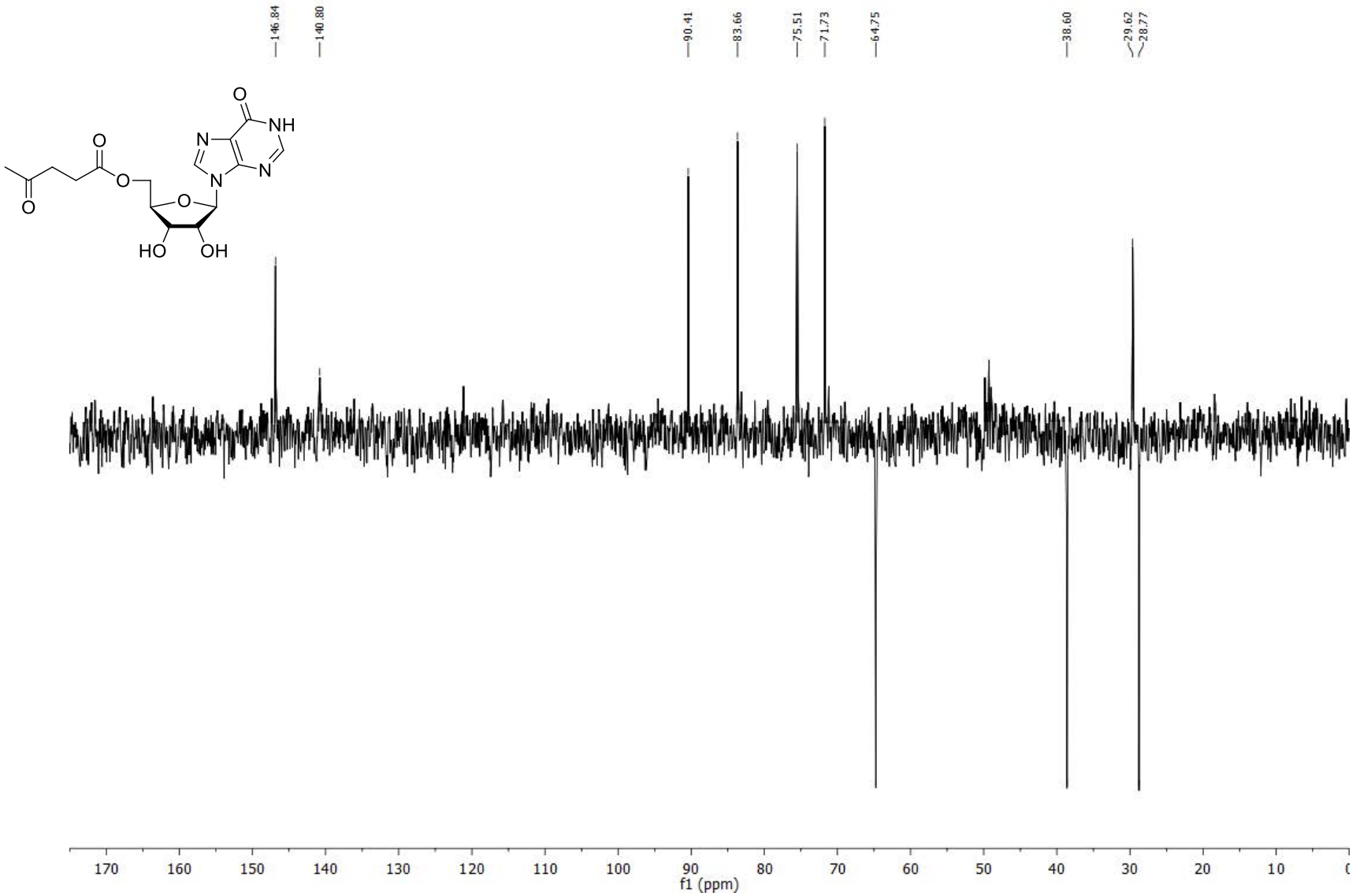
^{1}H-NMR (300.13 MHz, MeOD-d_4)



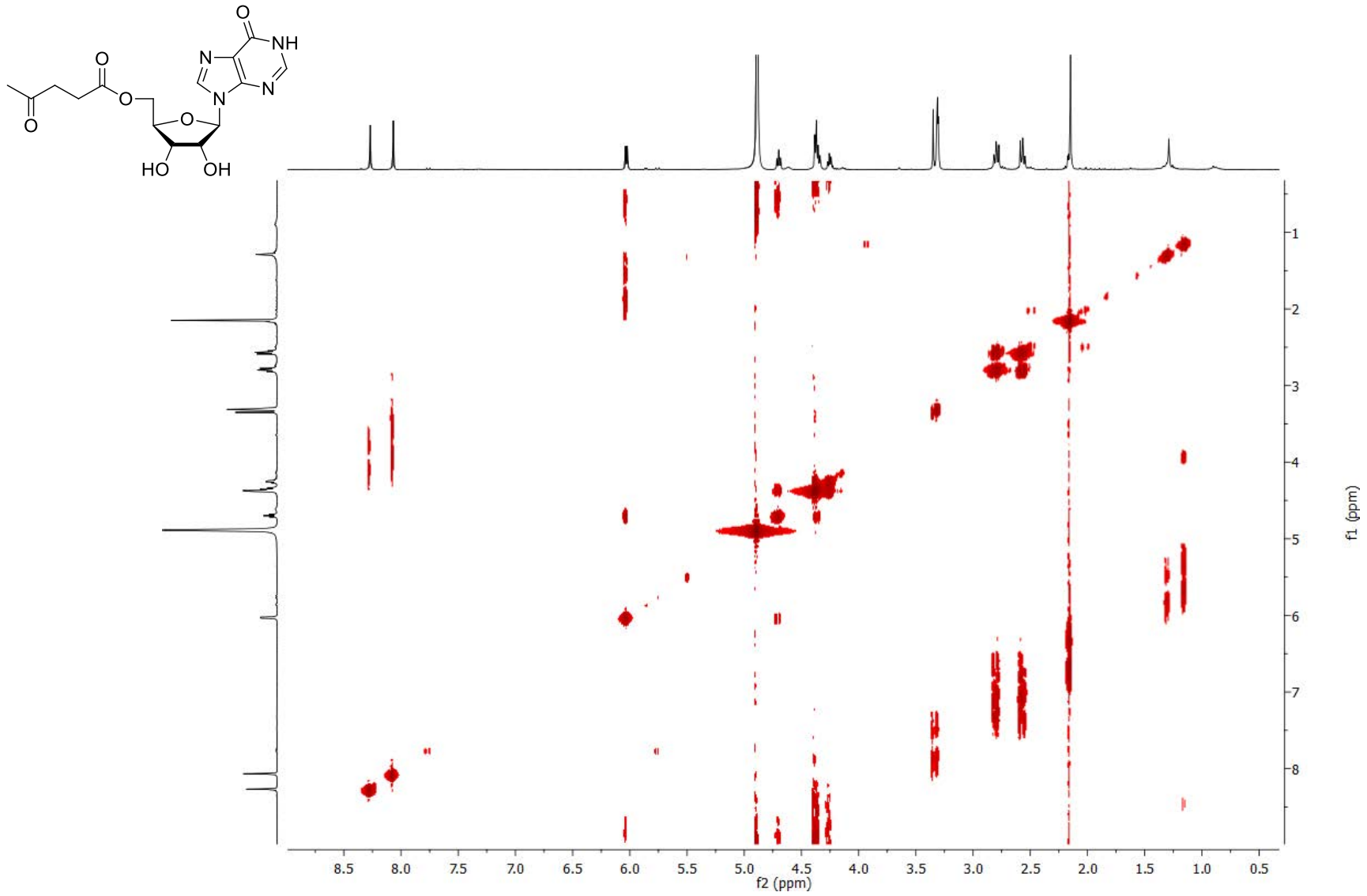
^{13}C-NMR (75.5 MHz, MeOD-d_4)



DEPT NMR (75.5 MHz, MeOD-d_4)



COSY NMR (MeOD-d_4)



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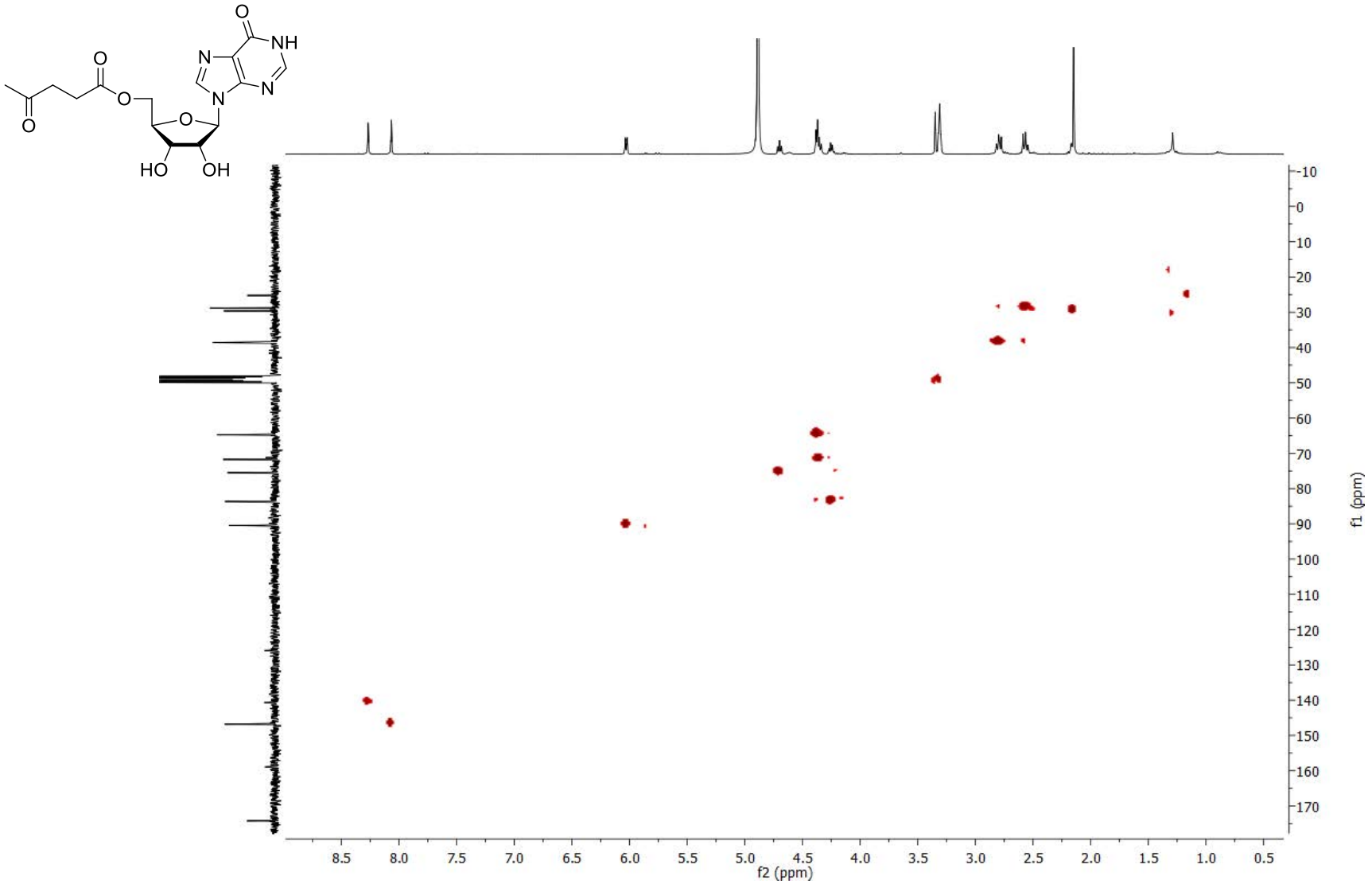
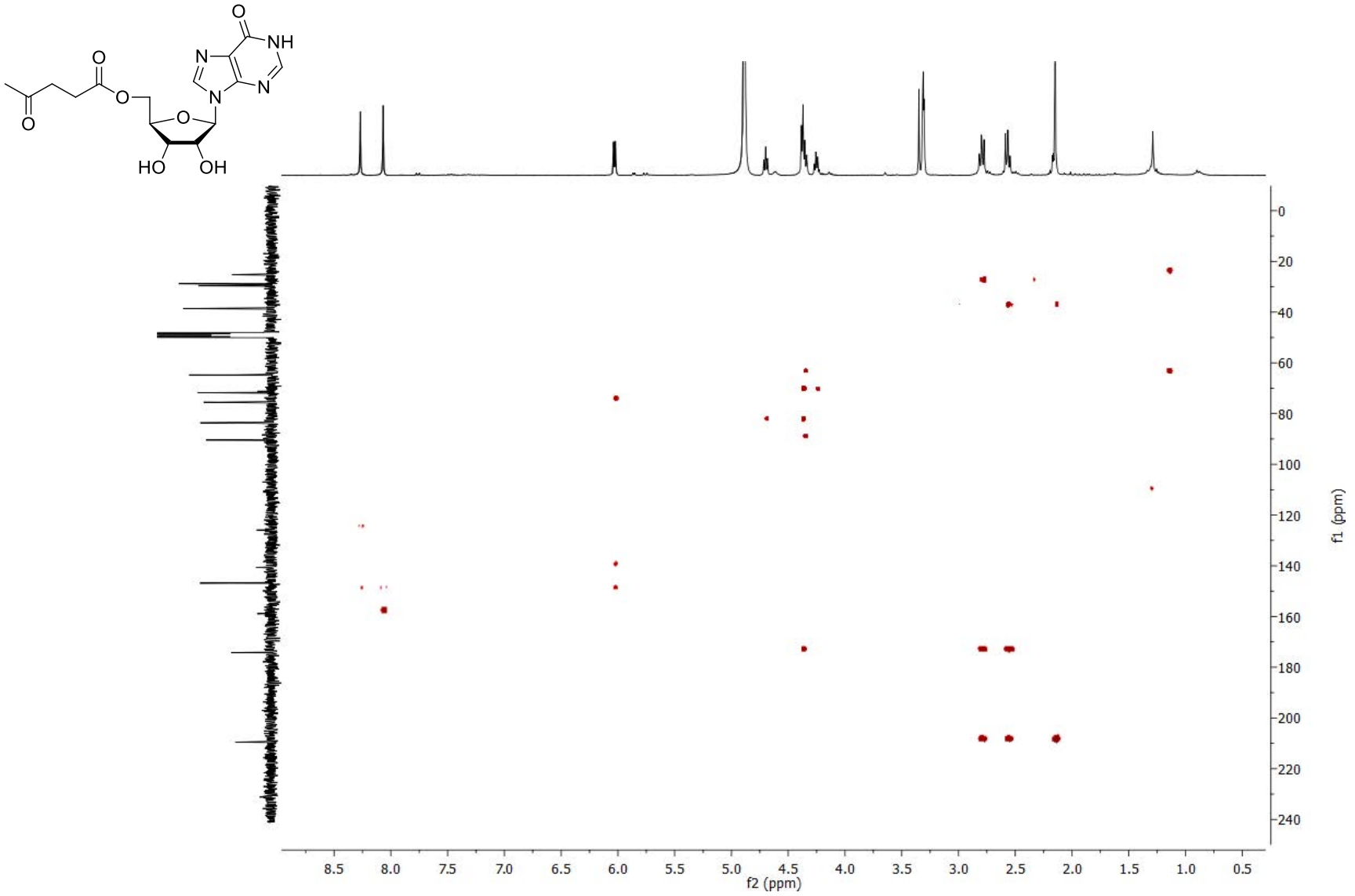
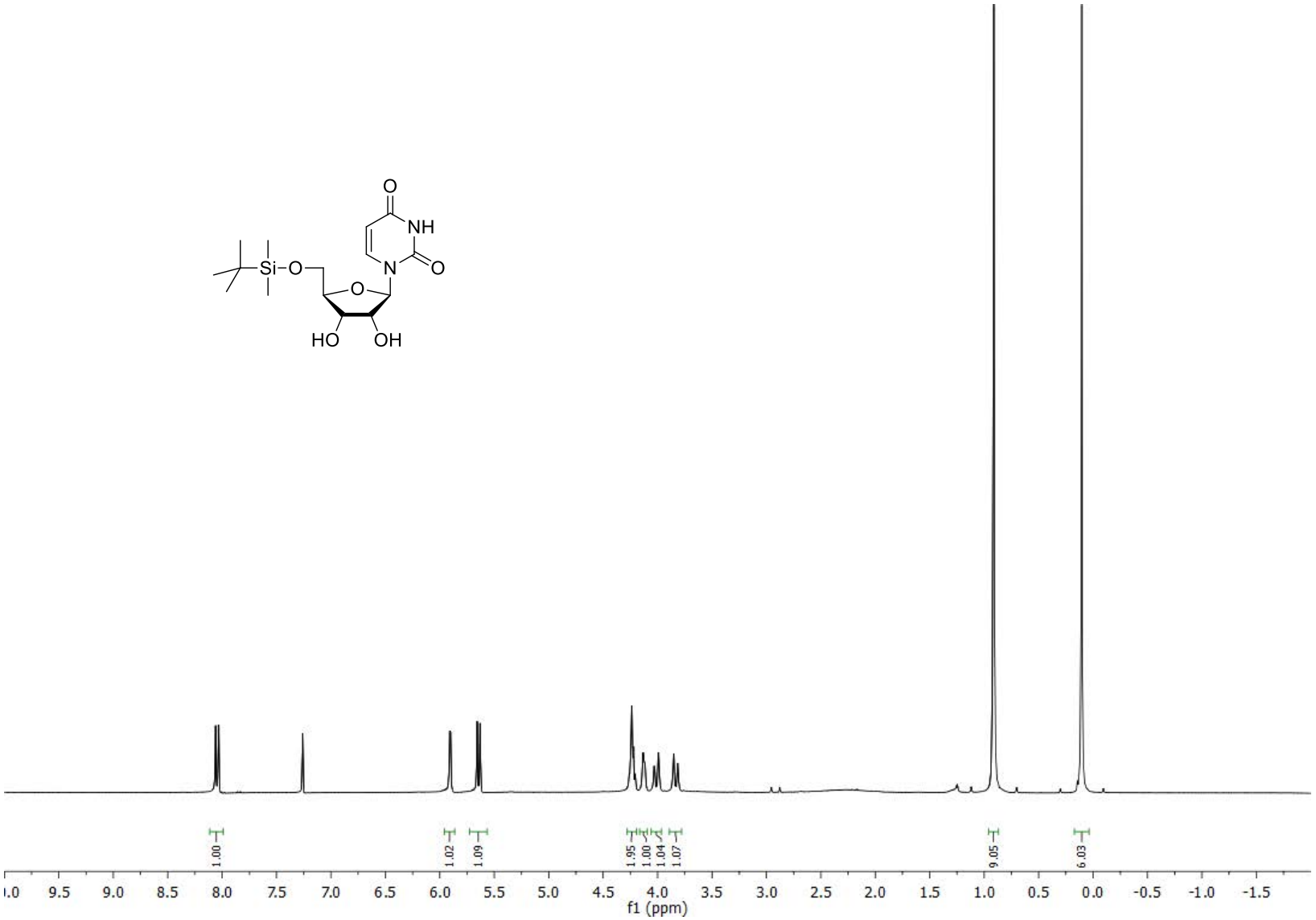


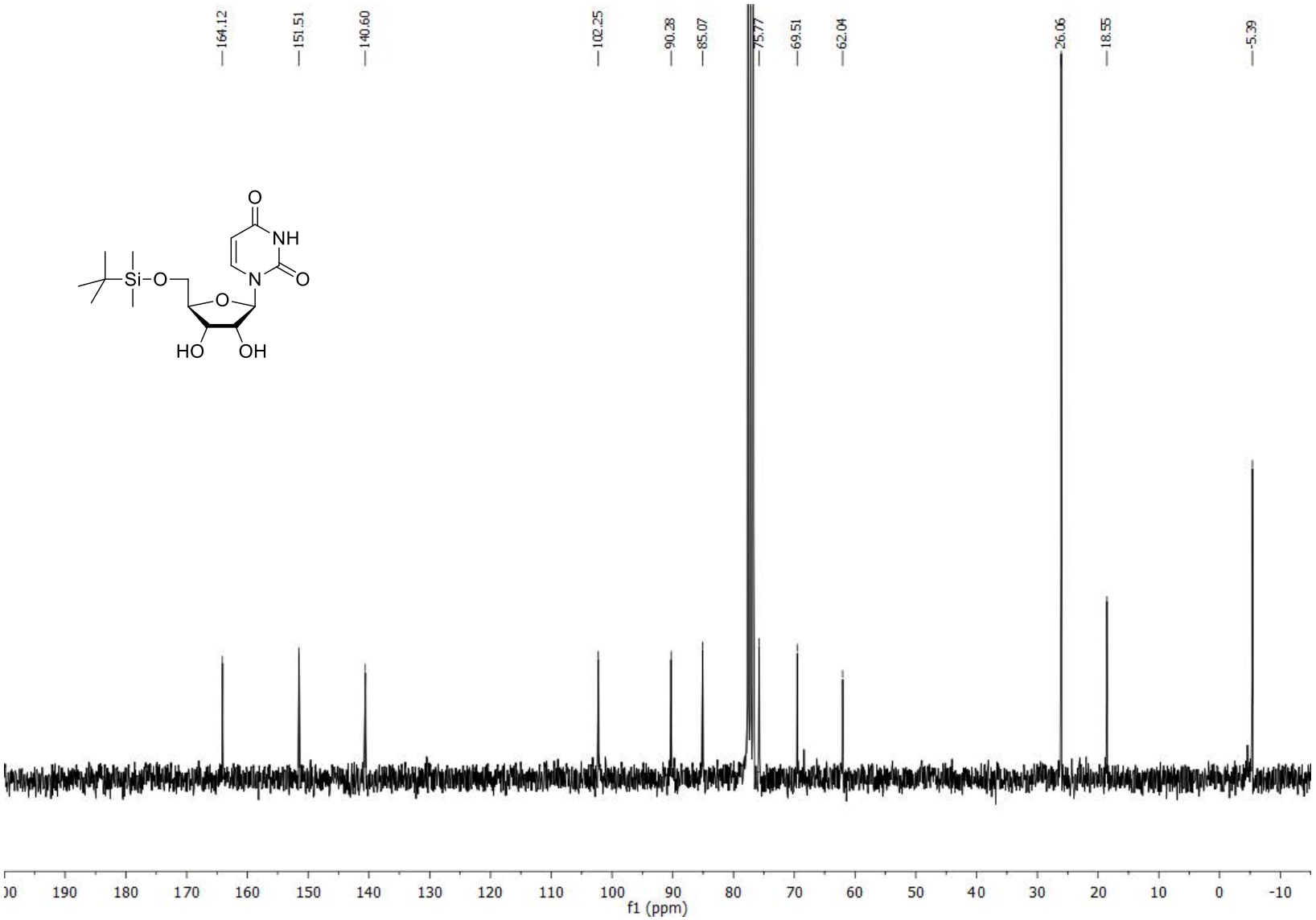
HMBC NMR (MeOD-d_4)



^{1}H-NMR (300.13 MHz, $CDCl_3$)

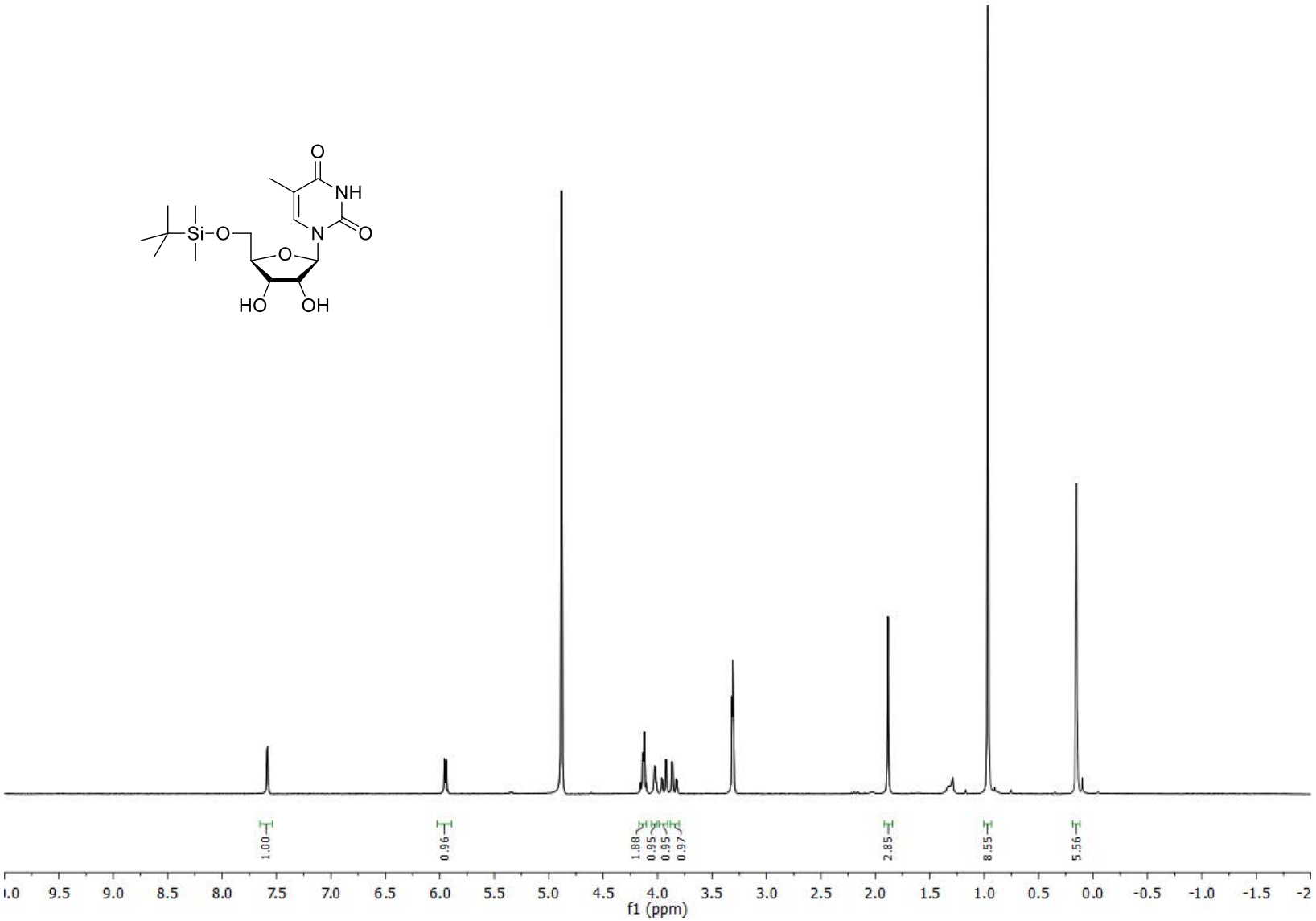


^{13}C-NMR (300.13 MHz, $CDCl_3$)

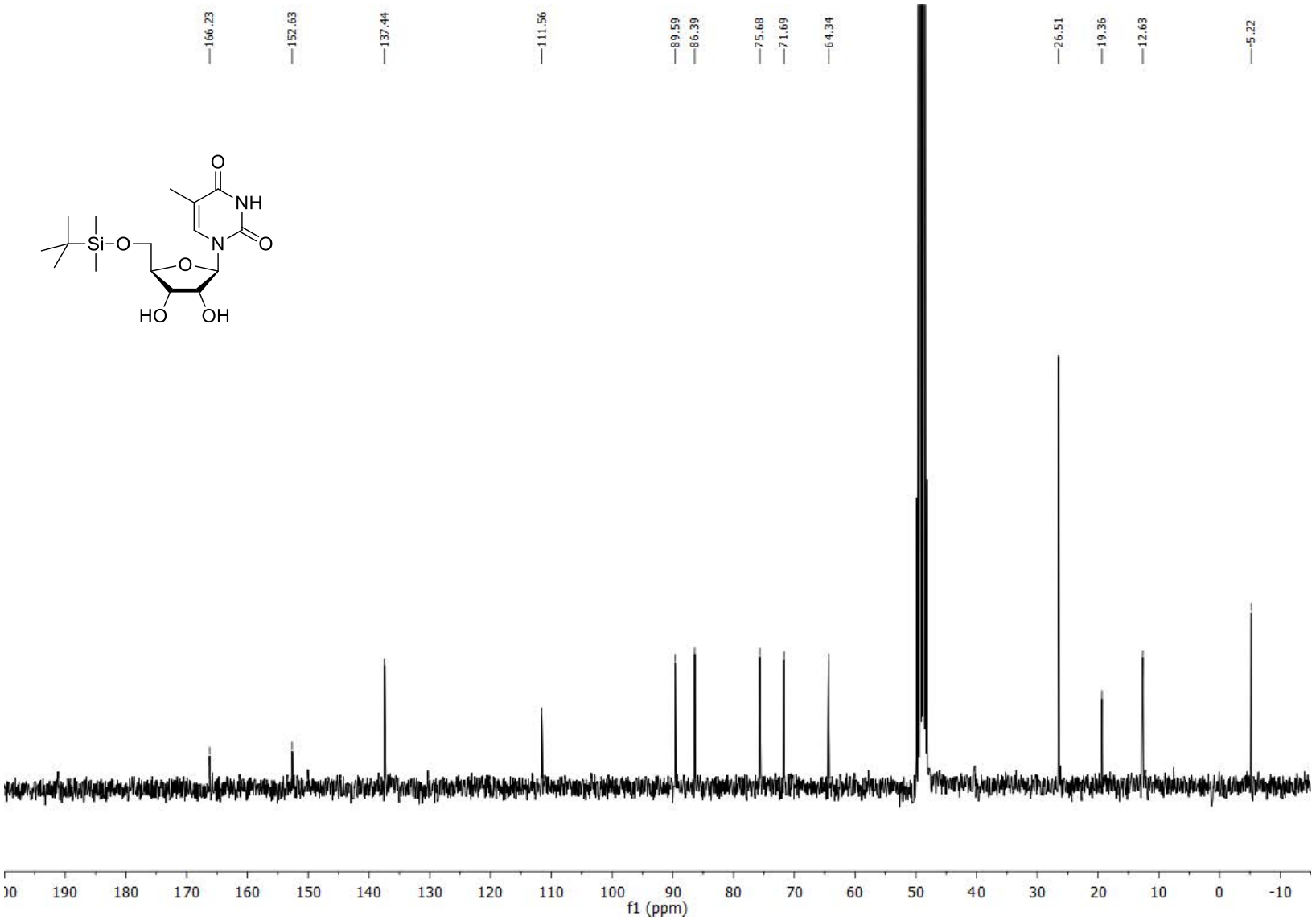


5’-*O*-(*tert*-Butyldimethylsilyl)-β-D-5-methyluridine (5b)

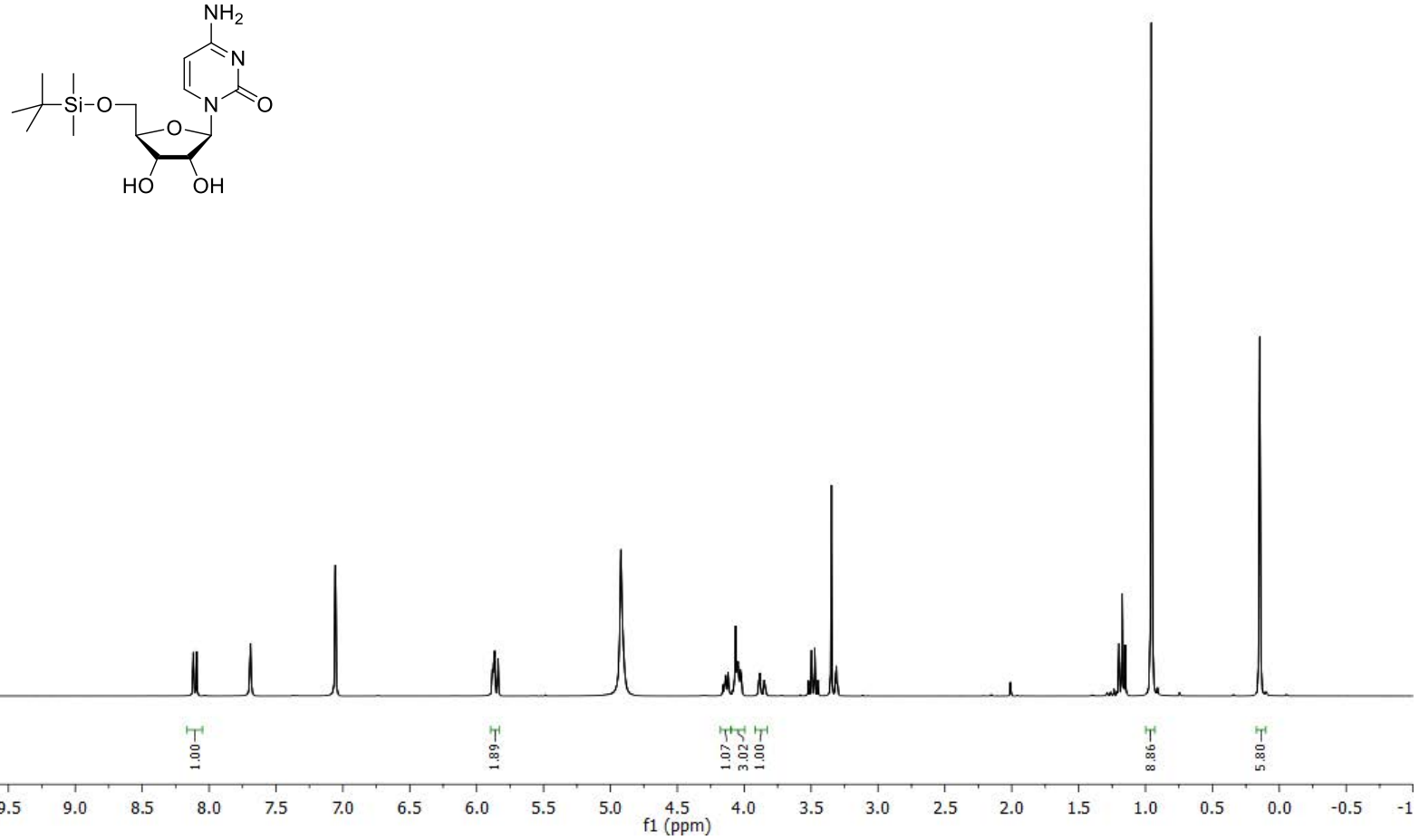
^{1}H-NMR (300.13 MHz, $CDCl_3$)



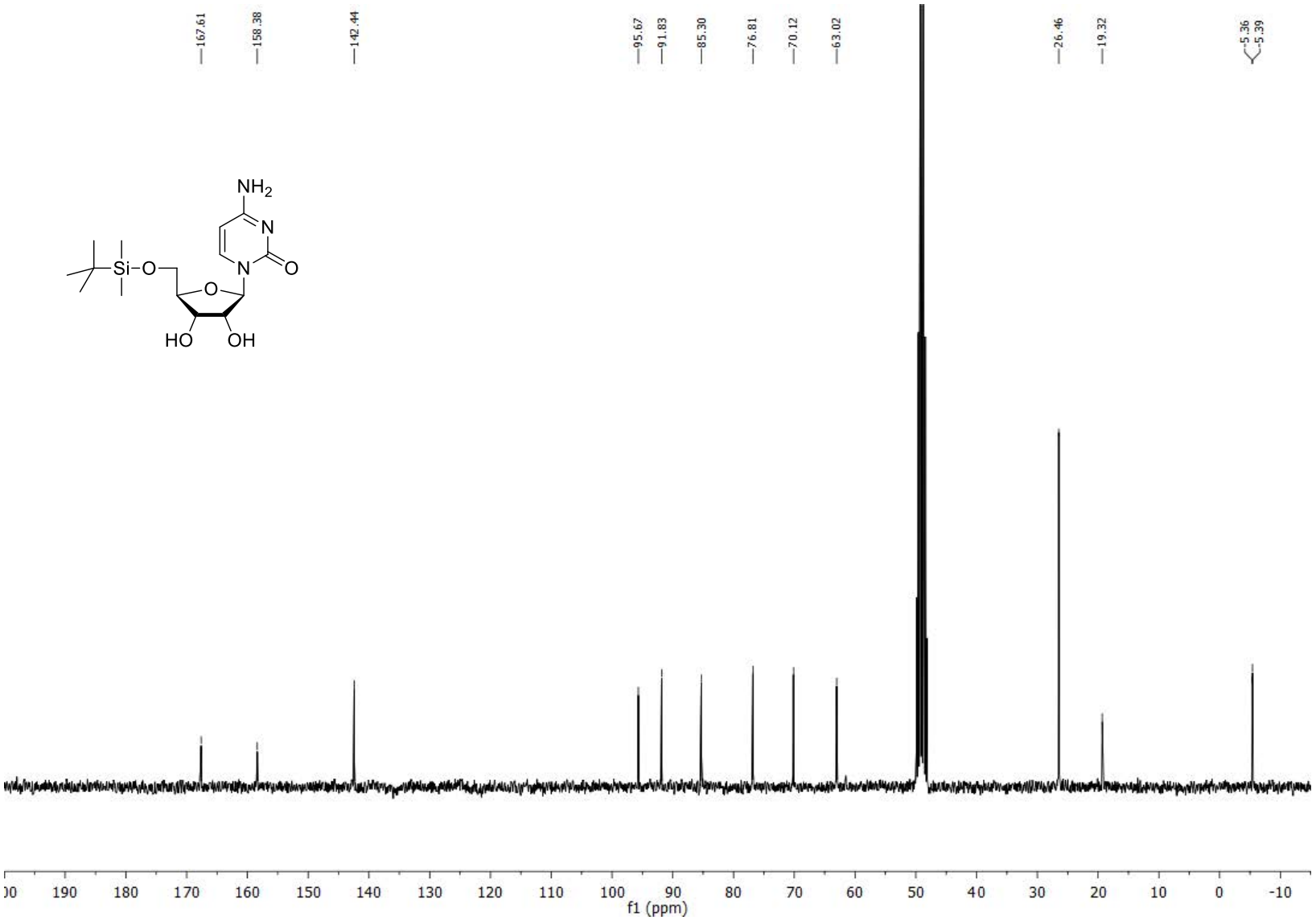
^{13}C-NMR (75.5 MHz, $CDCl_3$)



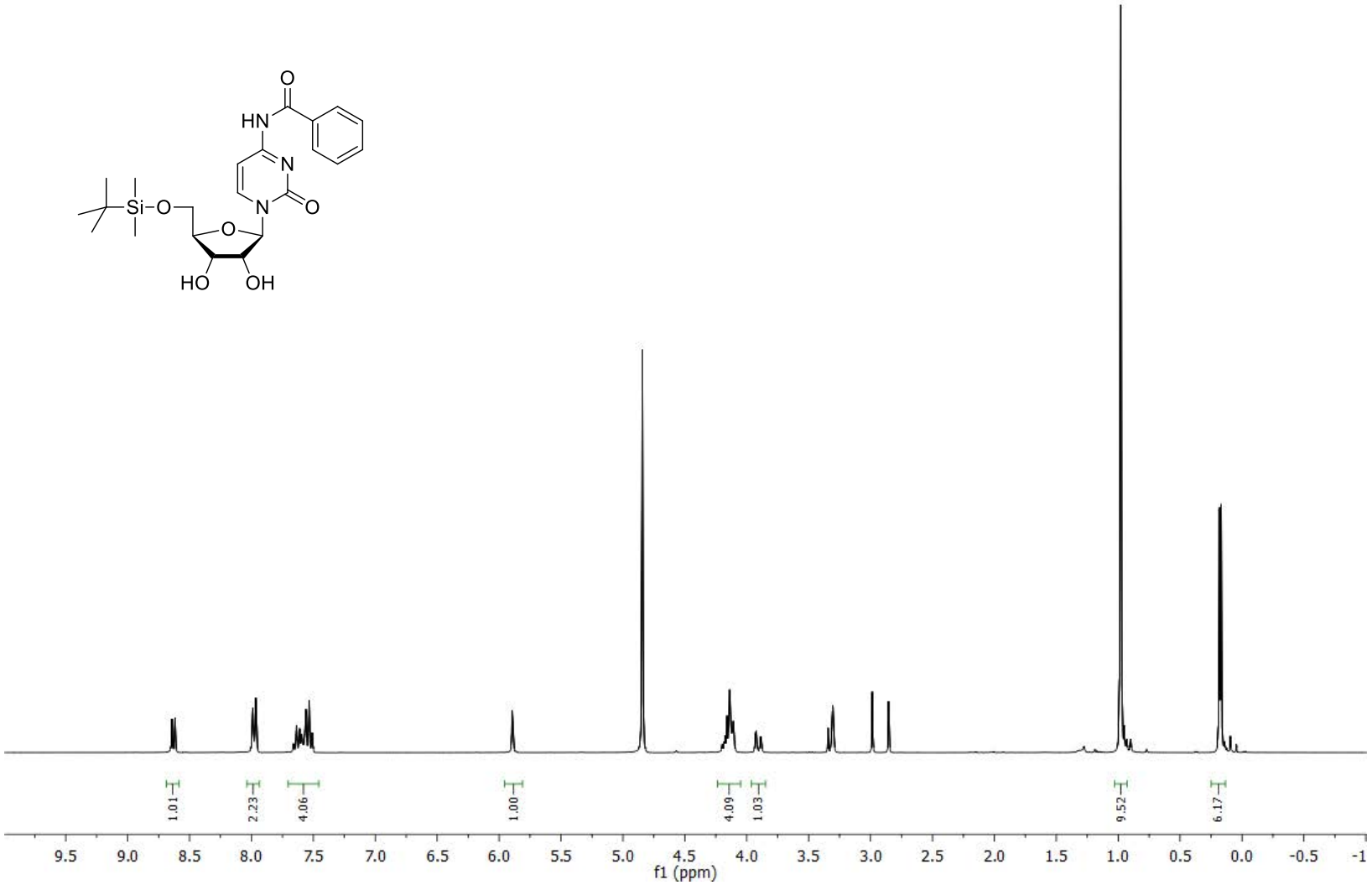
^{1}H-NMR (300.13 MHz, MeOH-d_4)



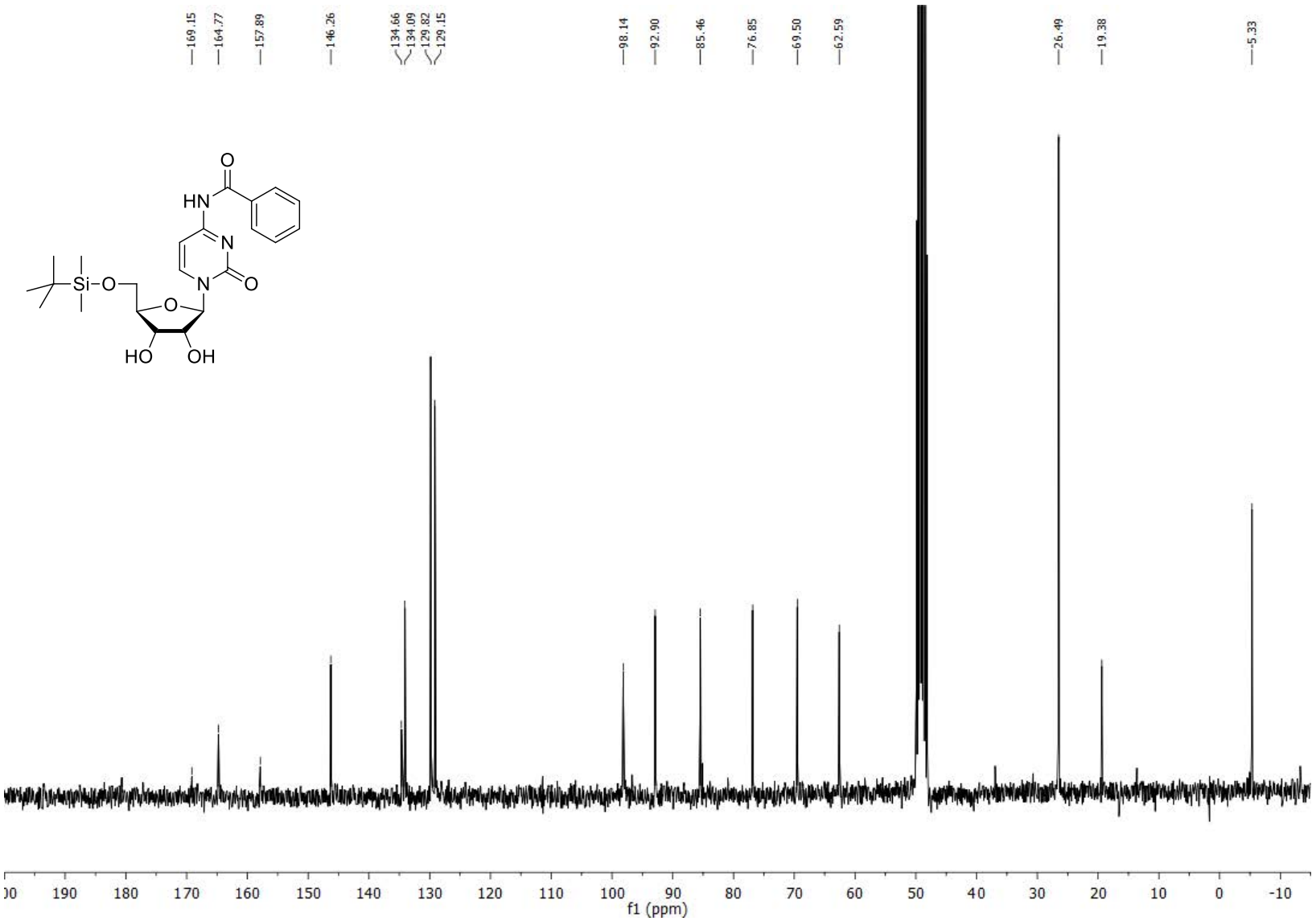
^{13}C-NMR (75.5 MHz, MeOH-d_4)



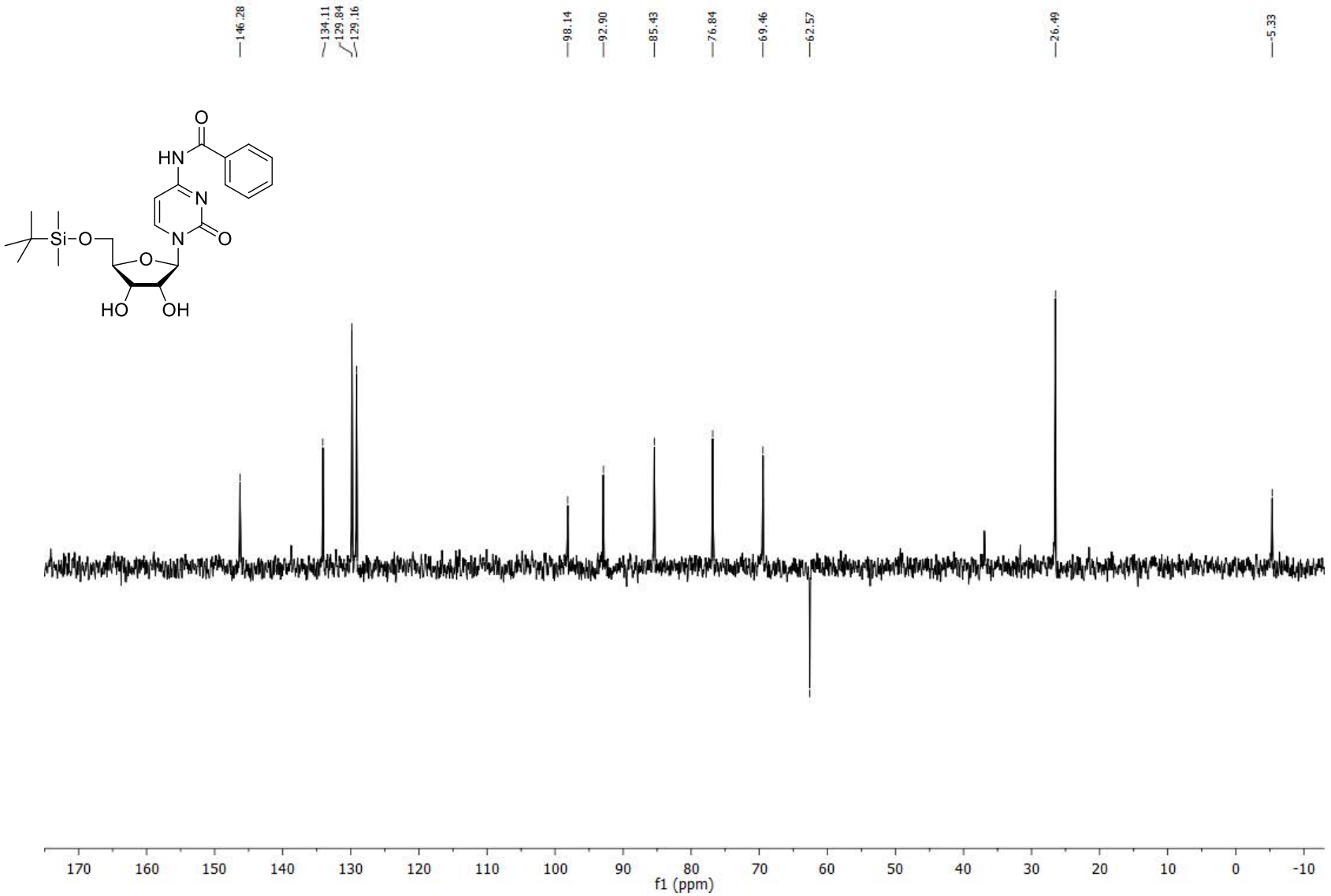
^{1}H-NMR (300.13 MHz, MeOH-d_4)



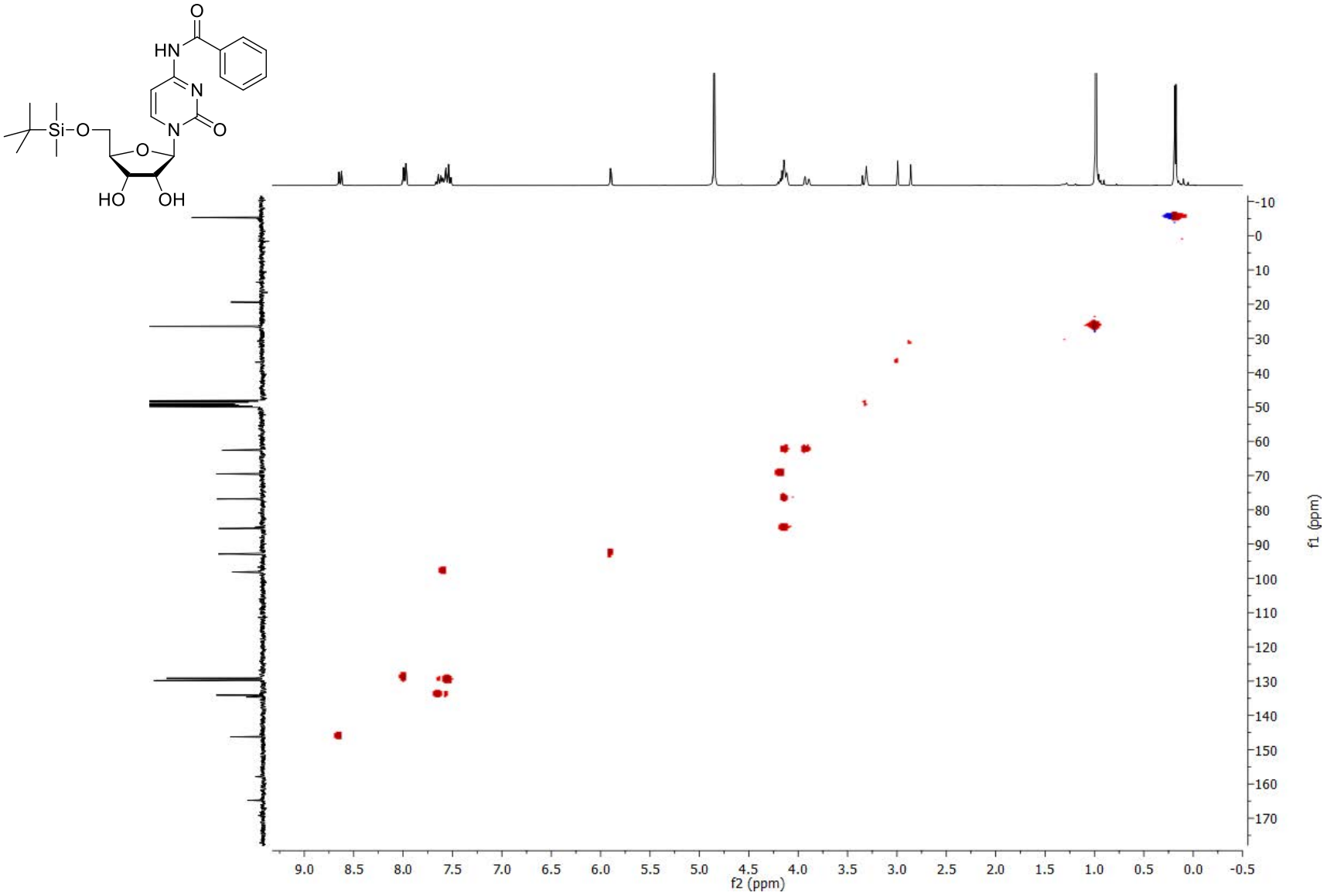
^{13}C-NMR (75.5 MHz, MeOH-d_4)



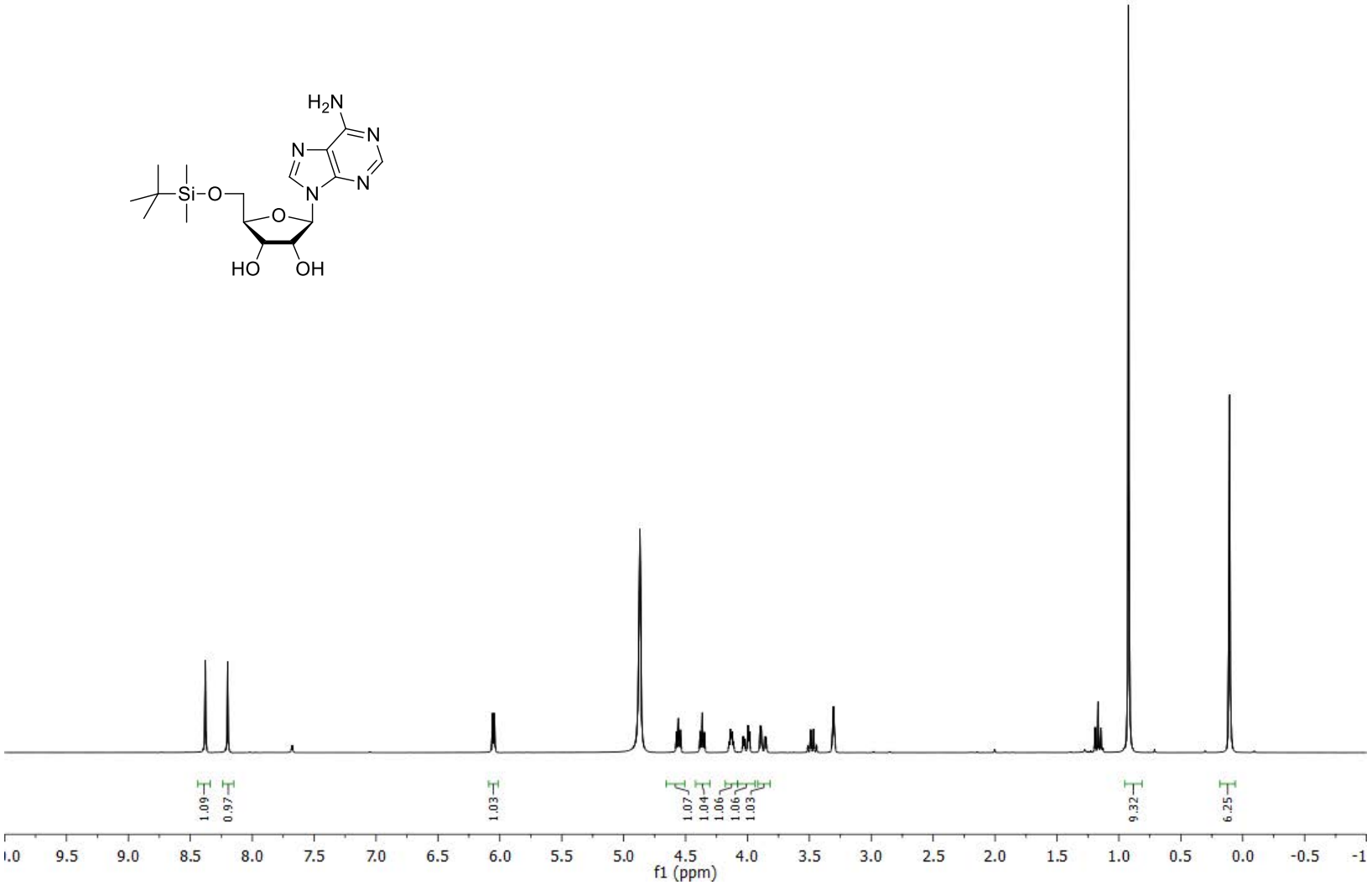
DEPT NMR (75.5 MHz, MeOH-d_4)



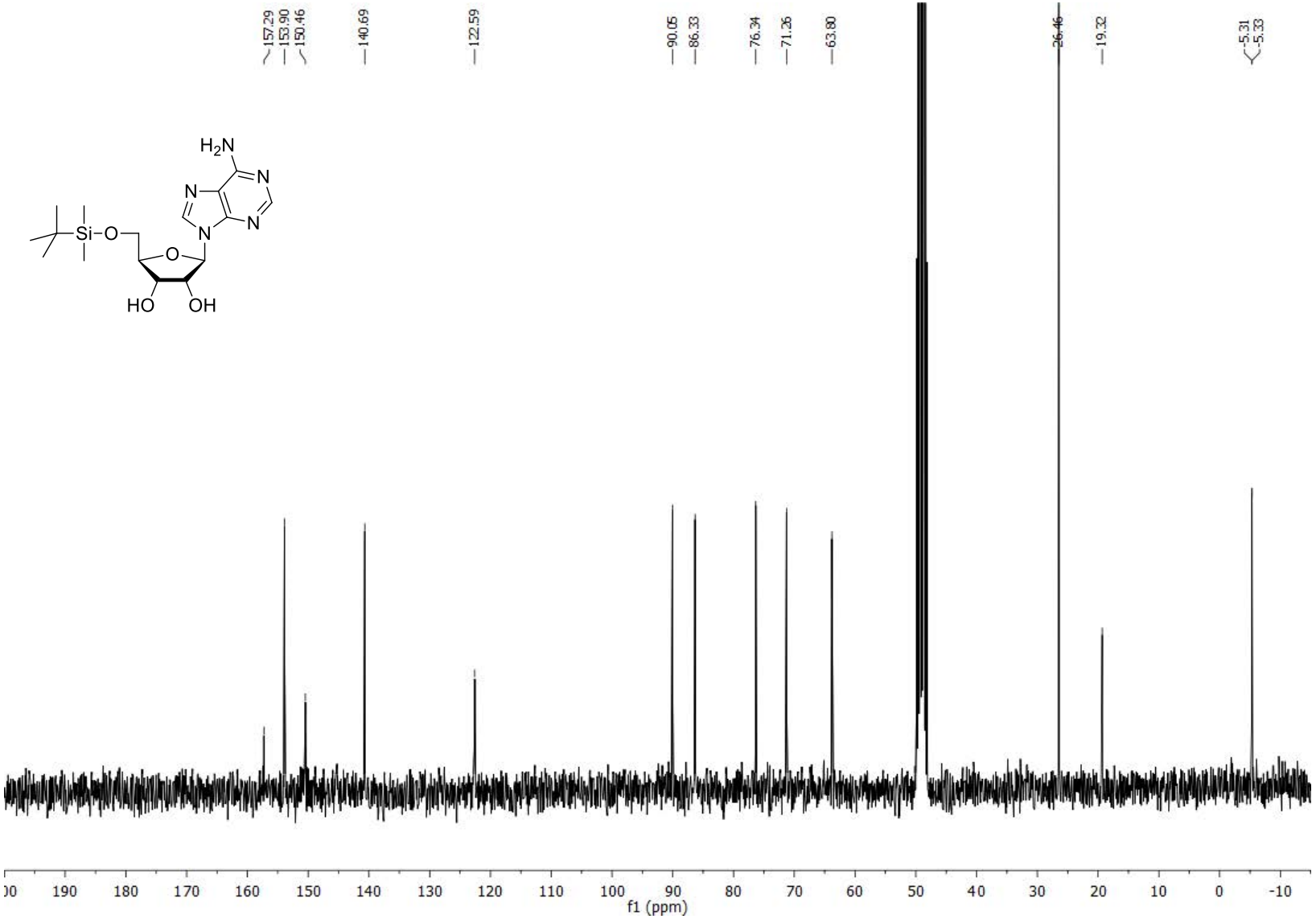
HSQC NMR (MeOH-d_4)



^{1}H-NMR (300.13 MHz, MeOH-d_4)

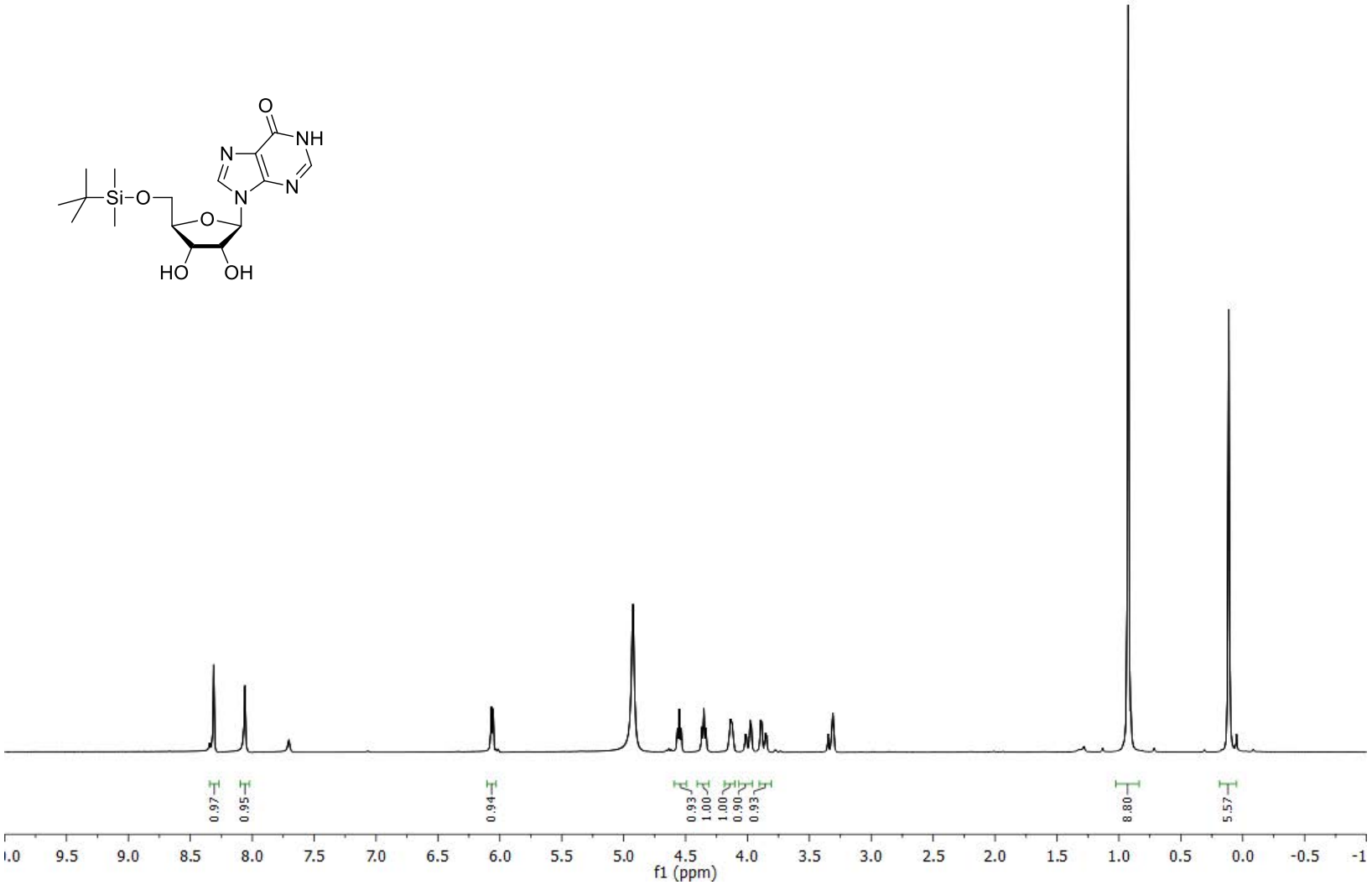


^{13}C-NMR (75.5 MHz, MeOH-d_4)

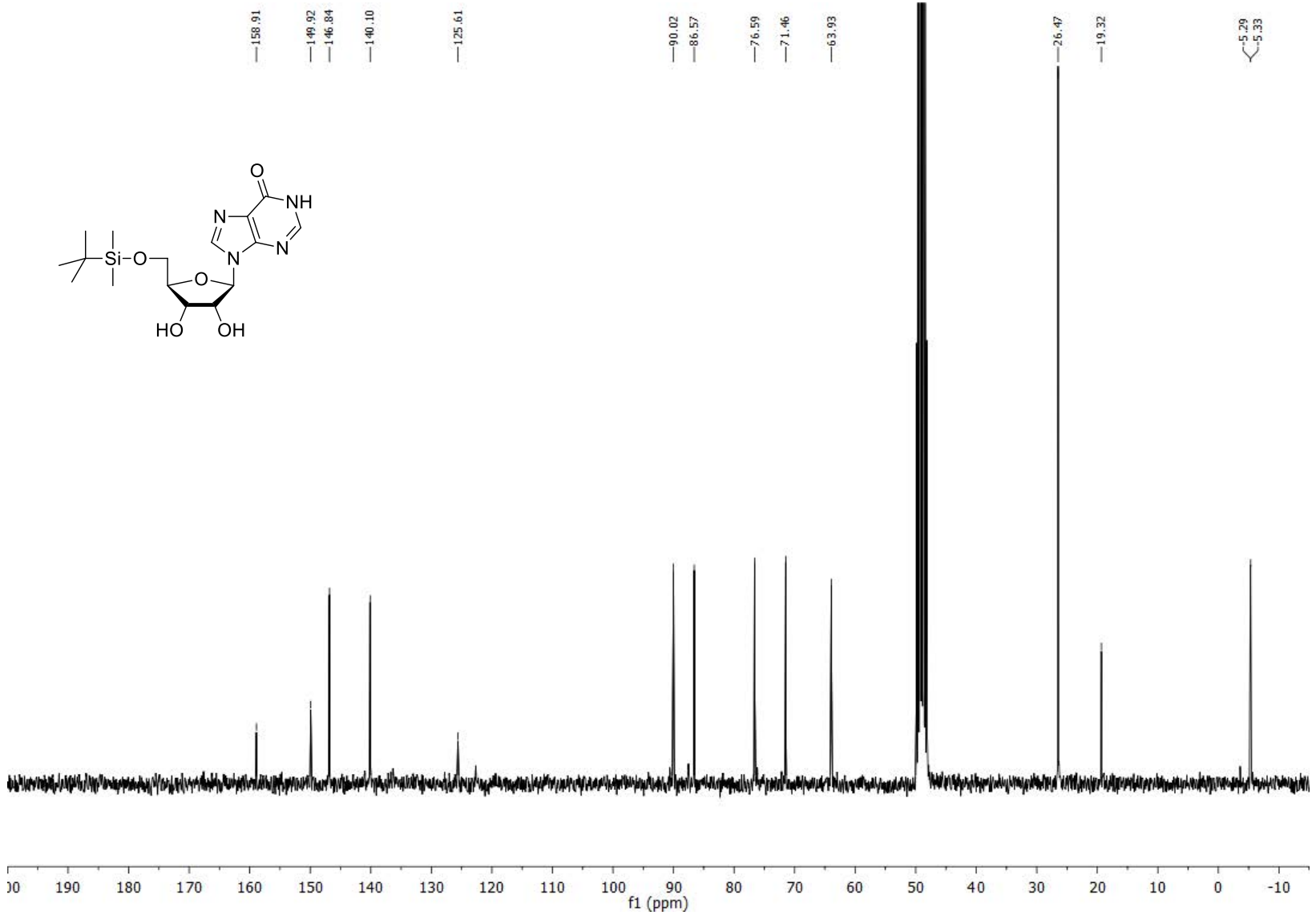


5'-*O*-(*tert*-Butyldimethylsilyl)-β-D-inosine (5f)

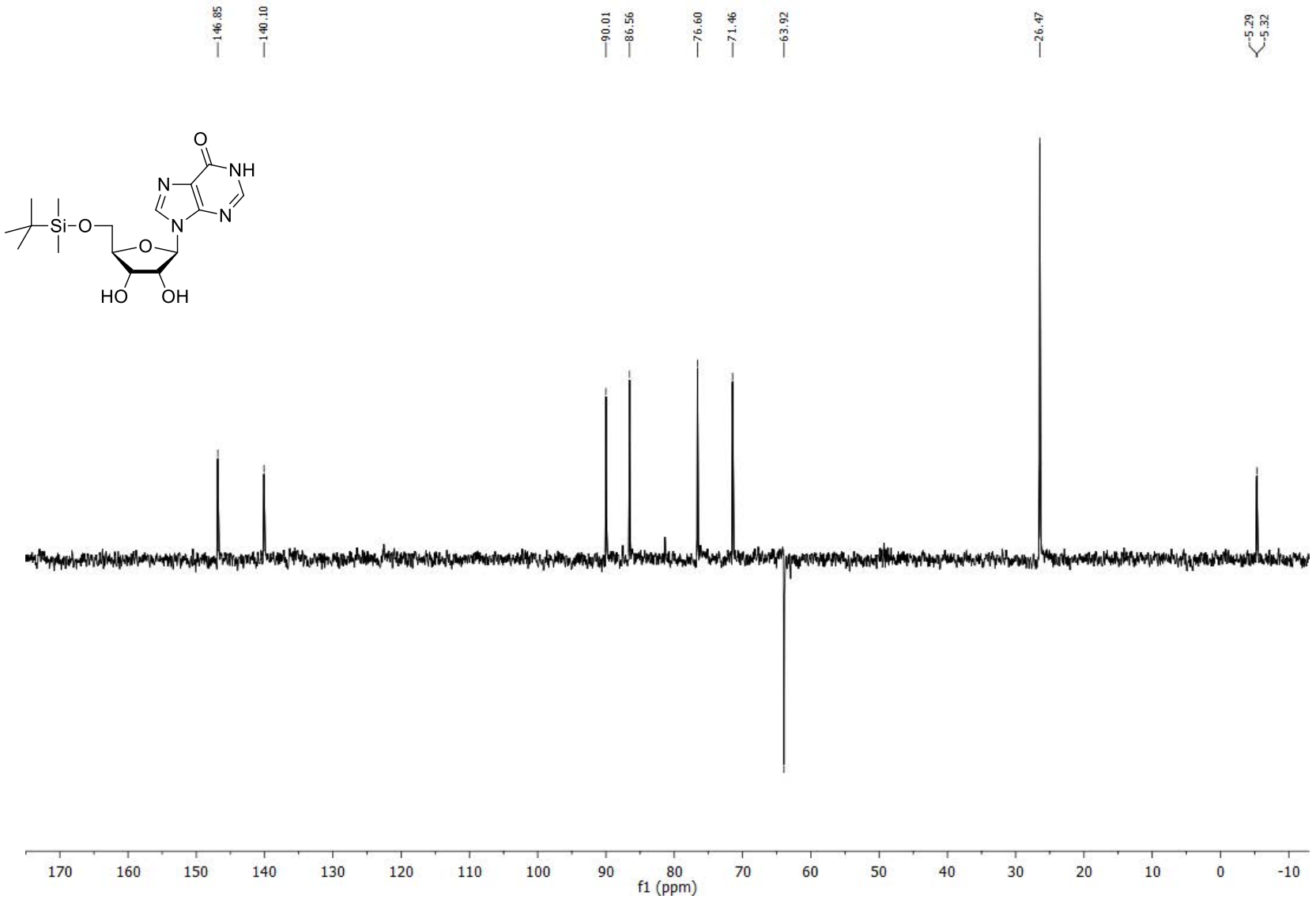
^{1}H-NMR (300.13 MHz, MeOH-d_4)



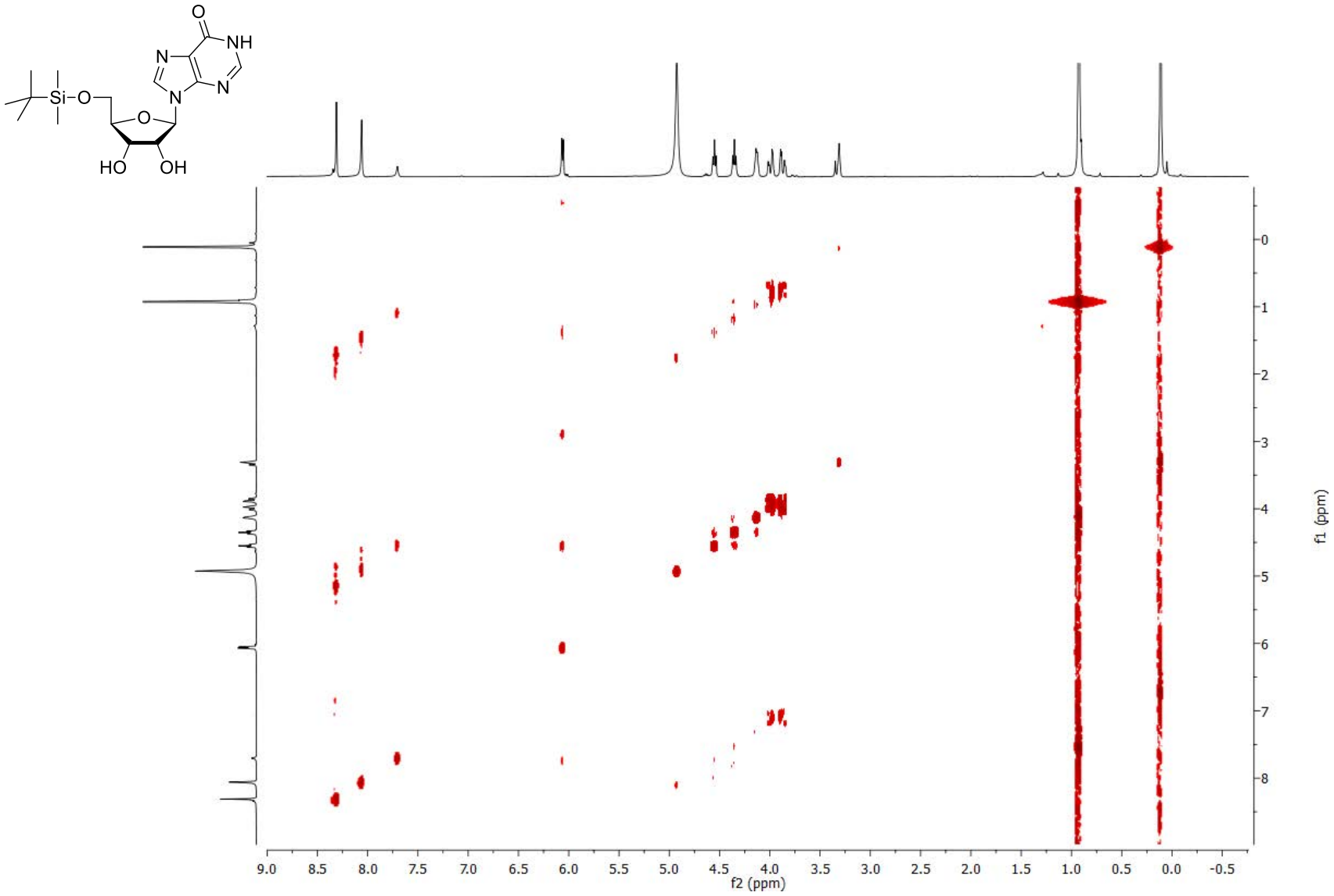
^{13}C-NMR (75.5 MHz, MeOH-d_4)



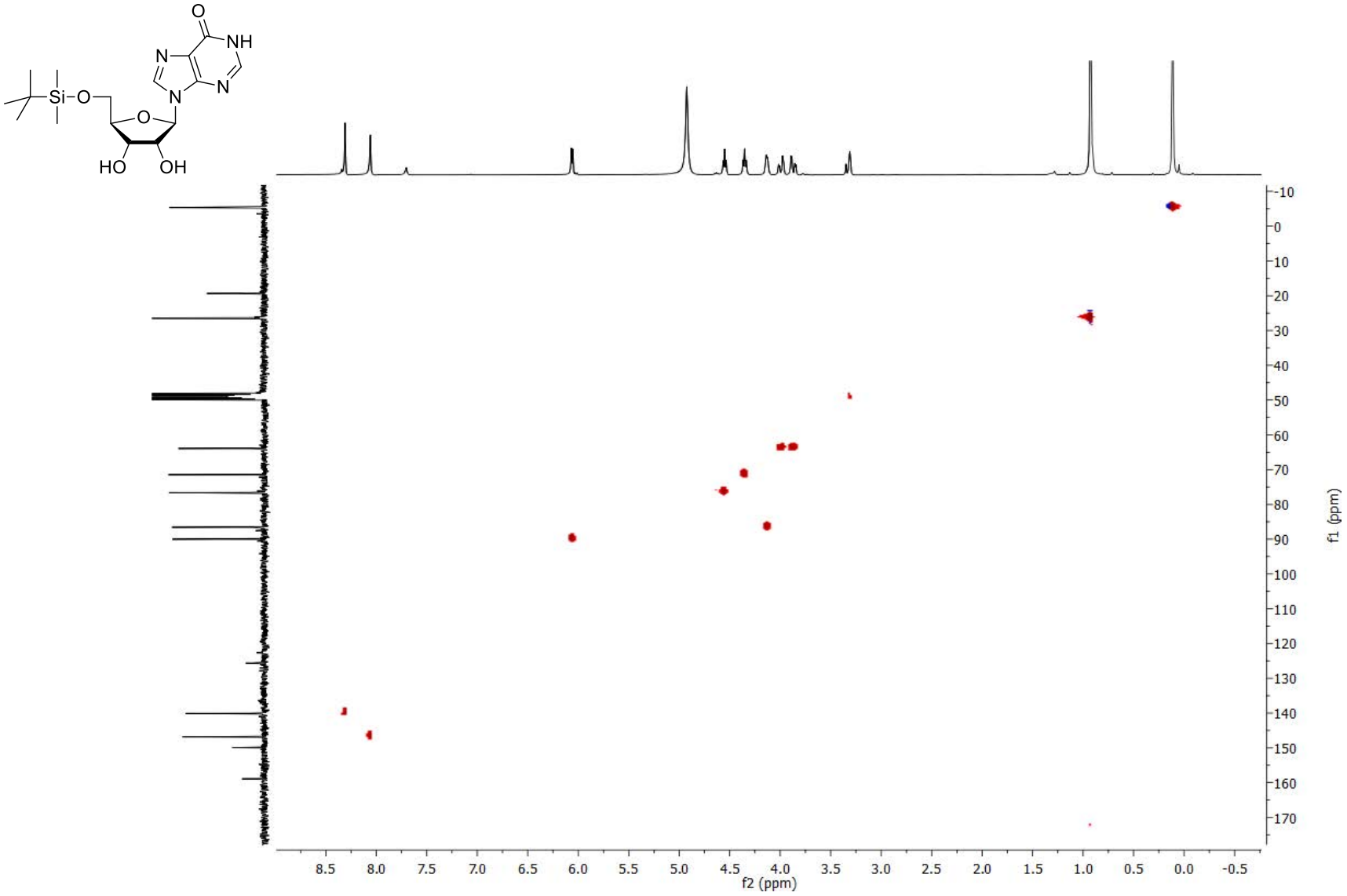
DEPT NMR (75.5 MHz, MeOH-d_4)



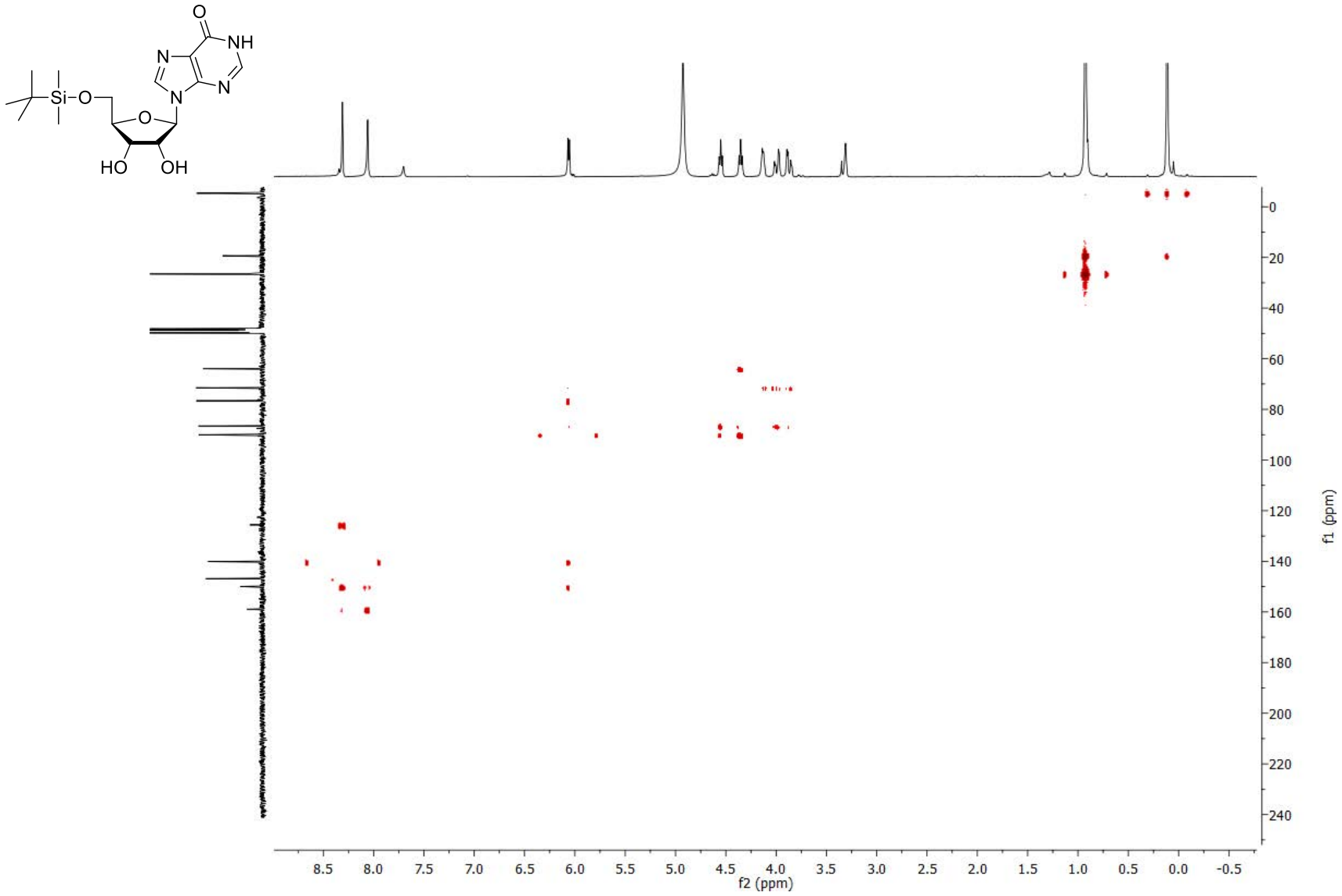
COSY NMR (MeOH-d_4)



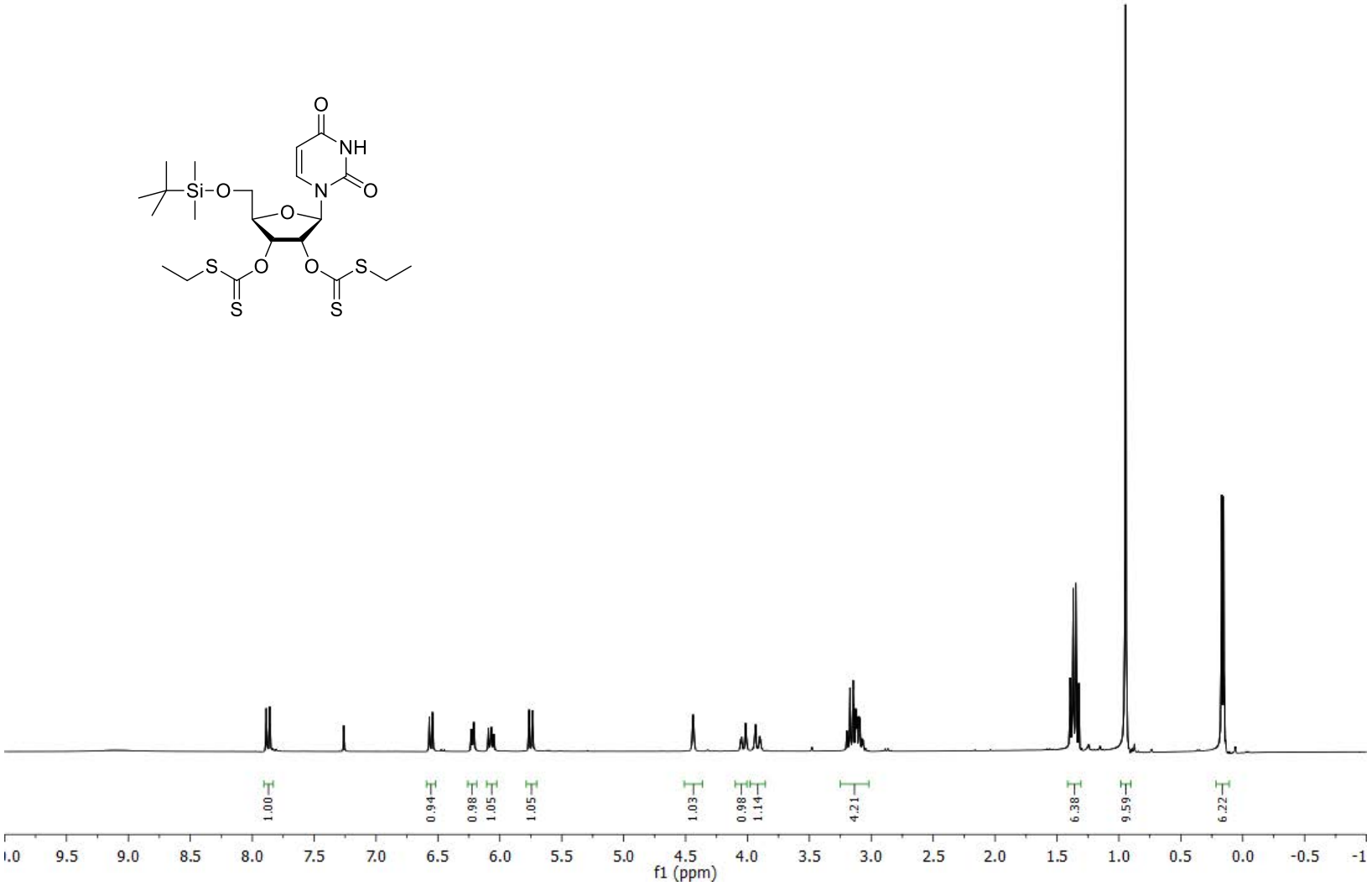
HSQC NMR (MeOH-d_4)



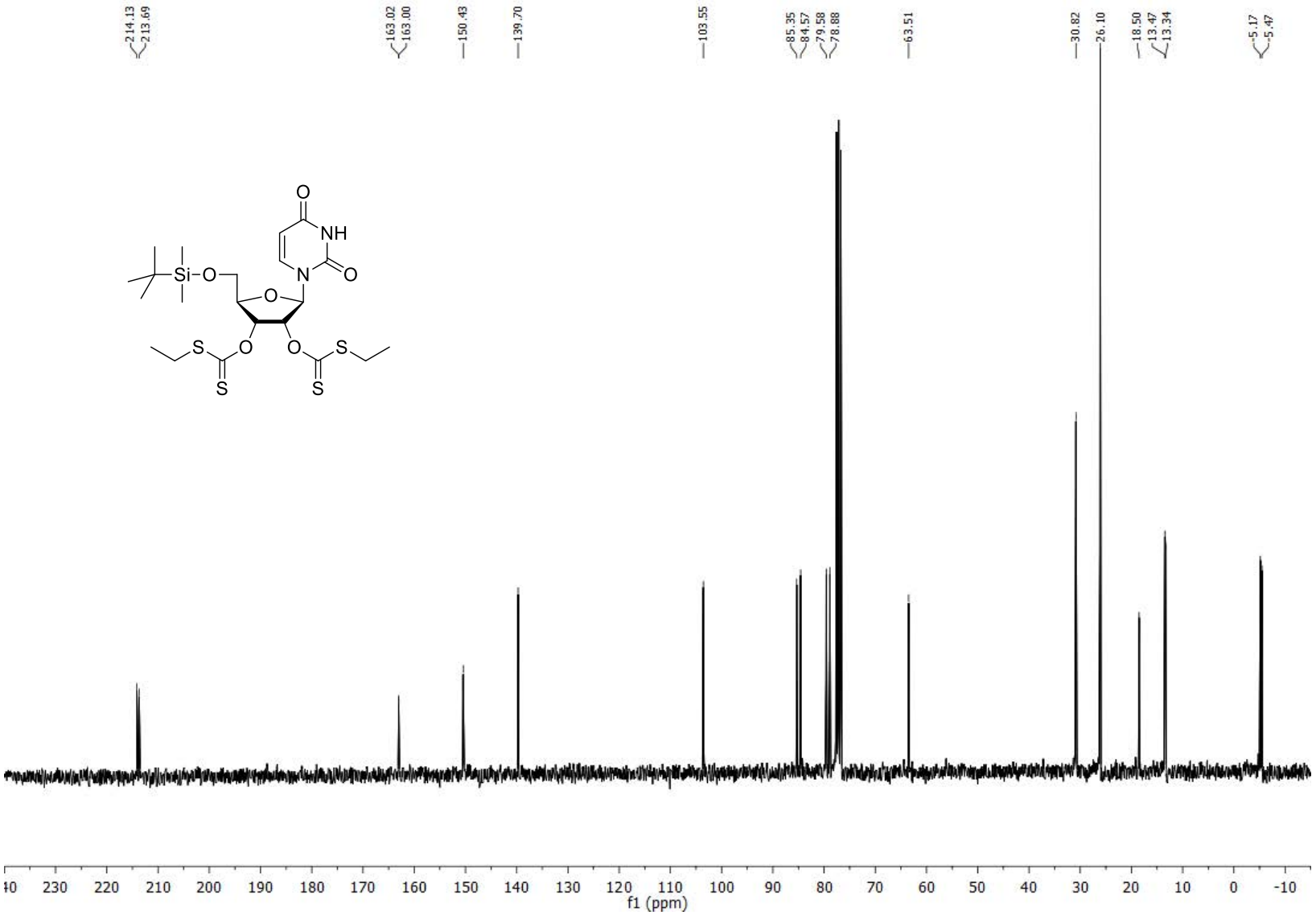
HMBC NMR (MeOH-d_4)



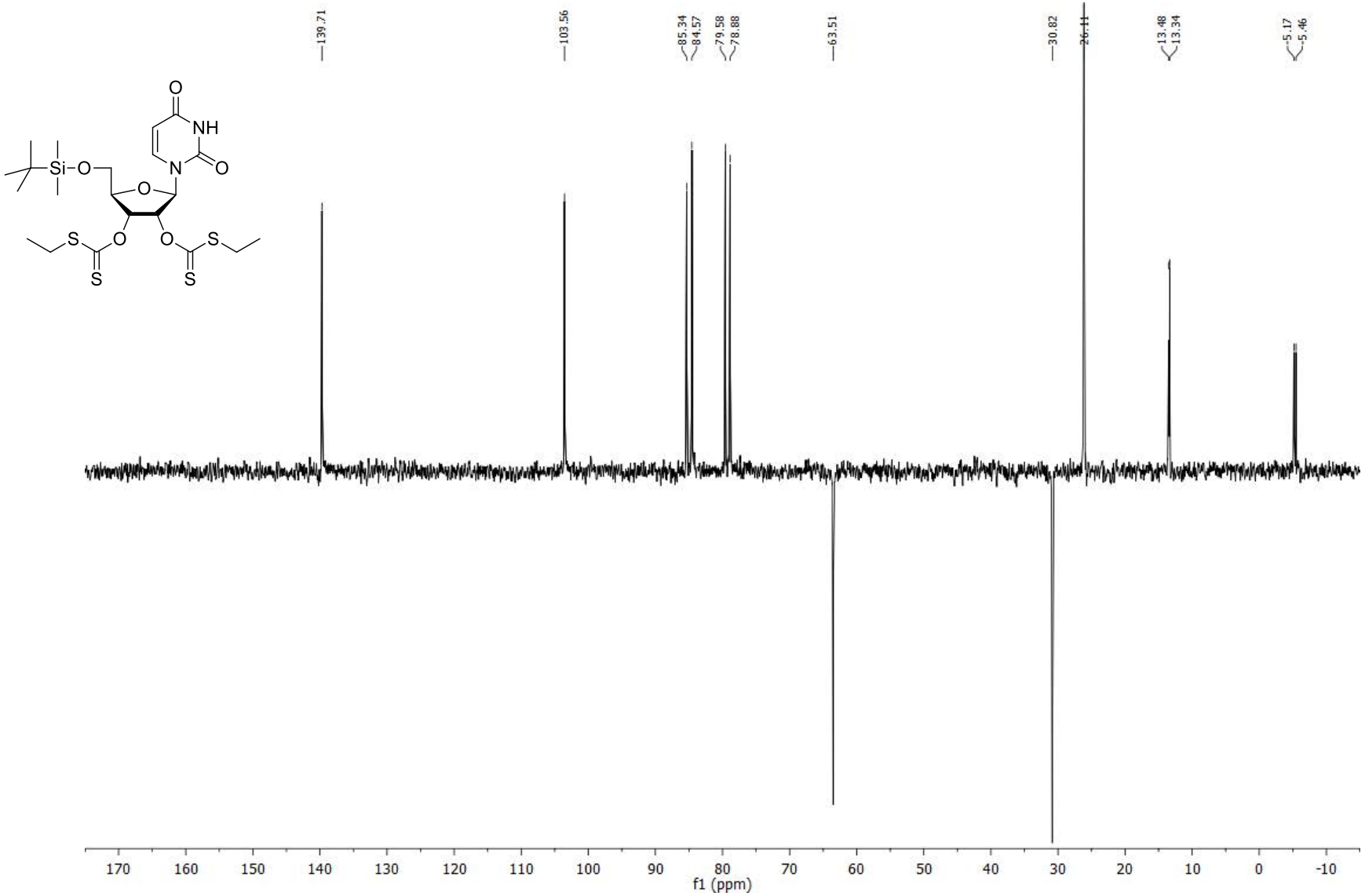
^{1}H-NMR (300.13 MHz, $CDCl_3$)



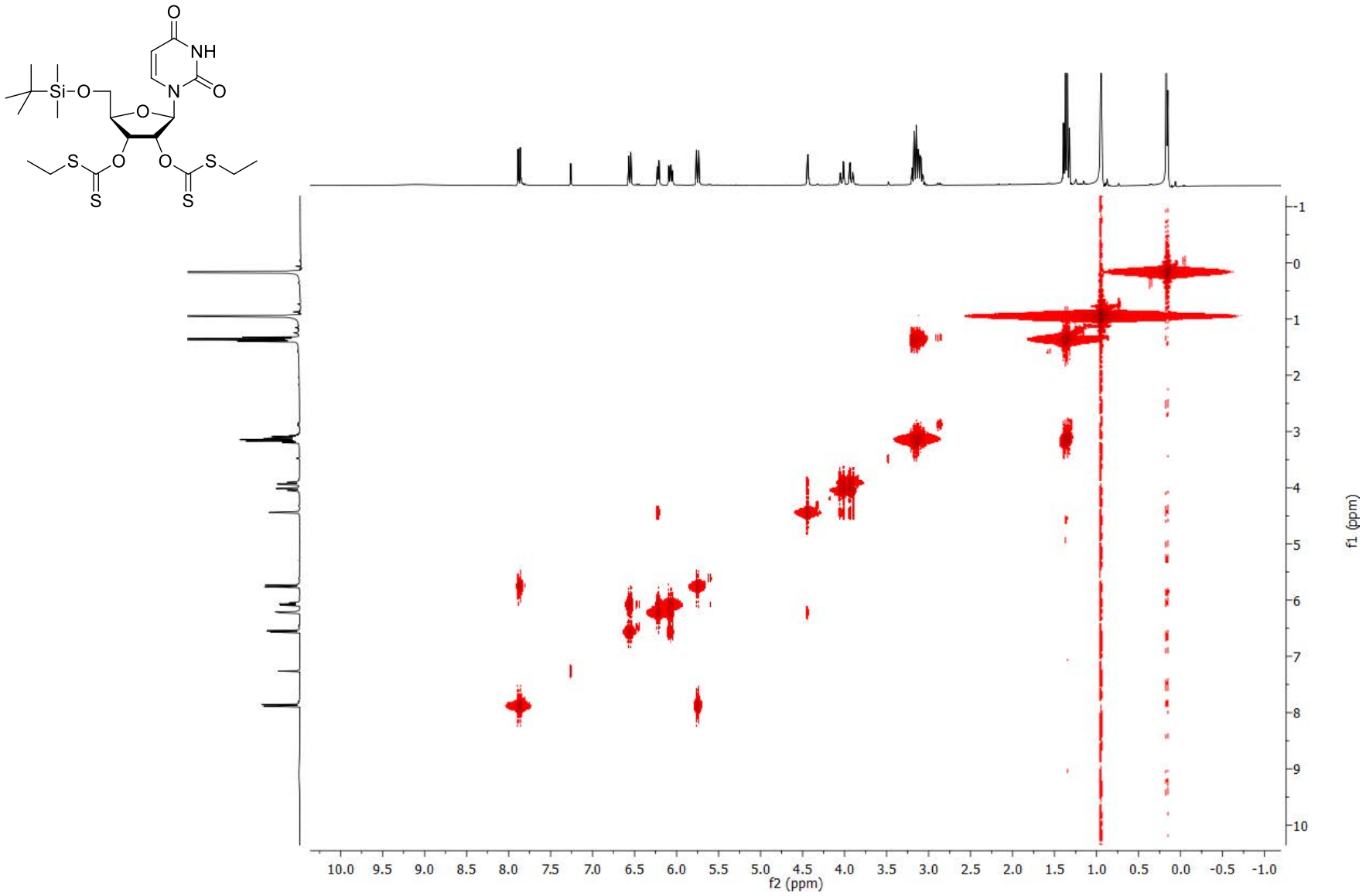
^{13}C-NMR (75.5 MHz, $CDCl_3$)



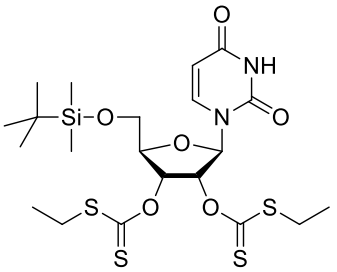
^{13}C-NMR (75.5 MHz, $CDCl_3$)

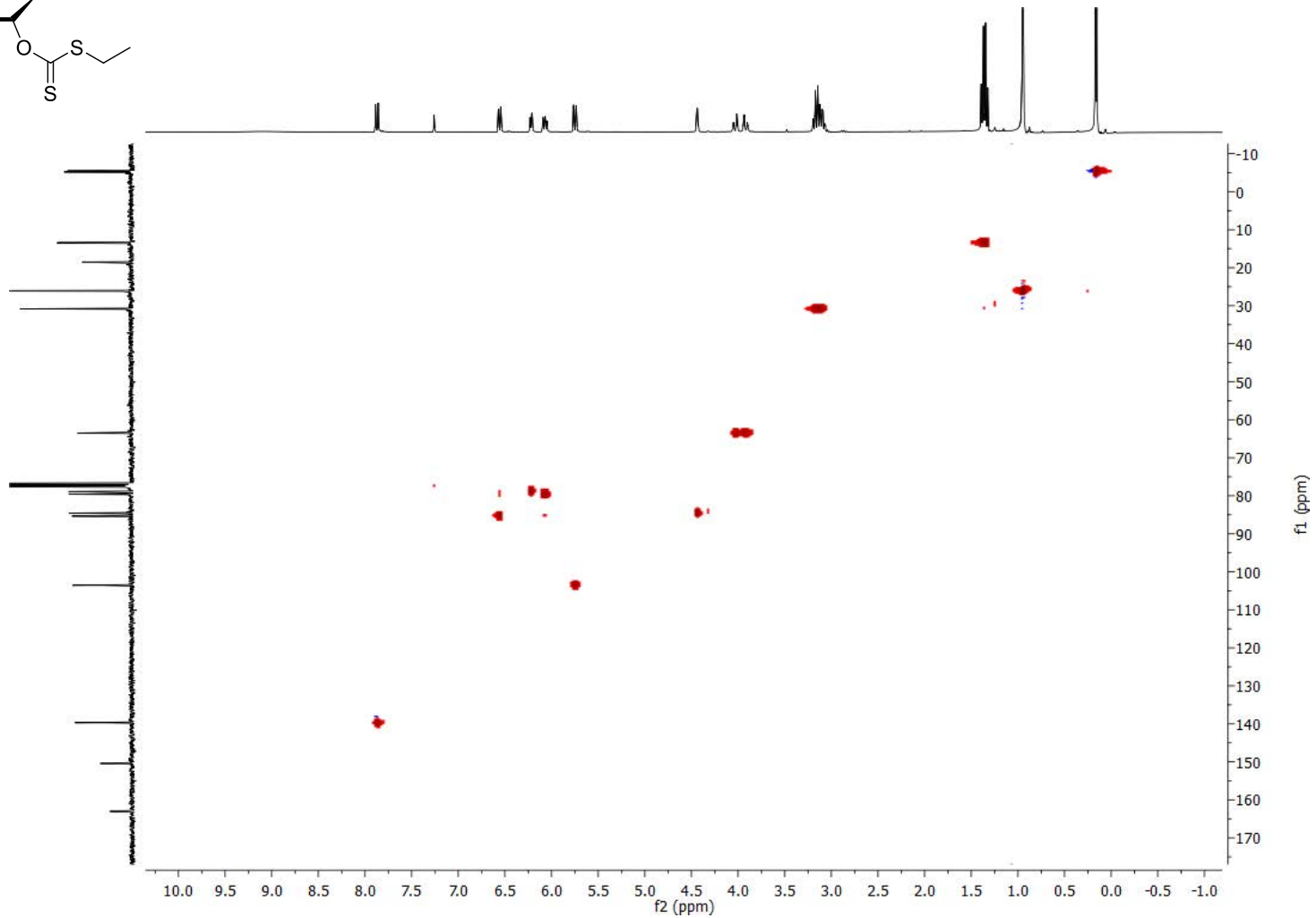


COSY NMR ($CDCl_3$)



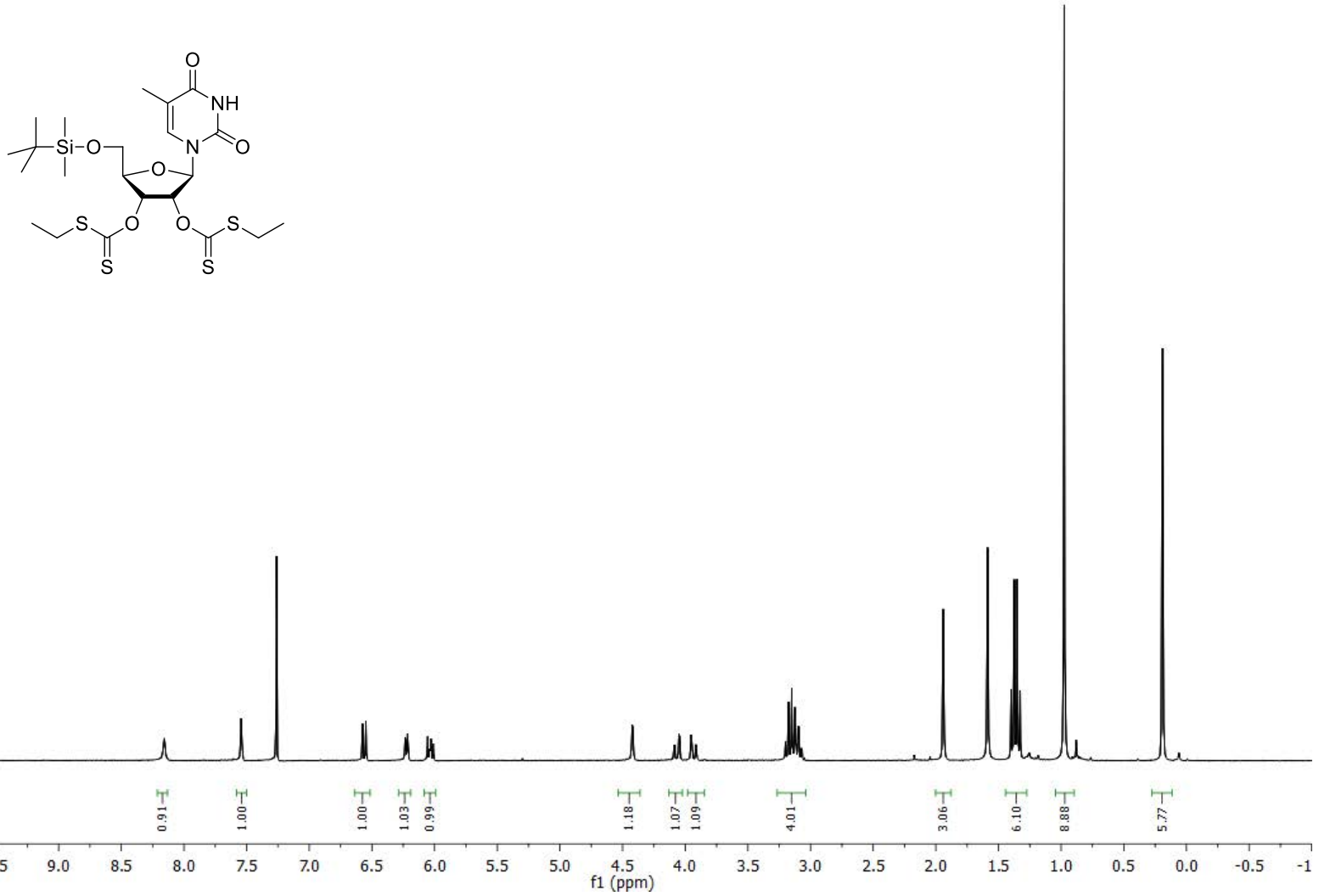
HSQC NMR ($CDCl_3$)



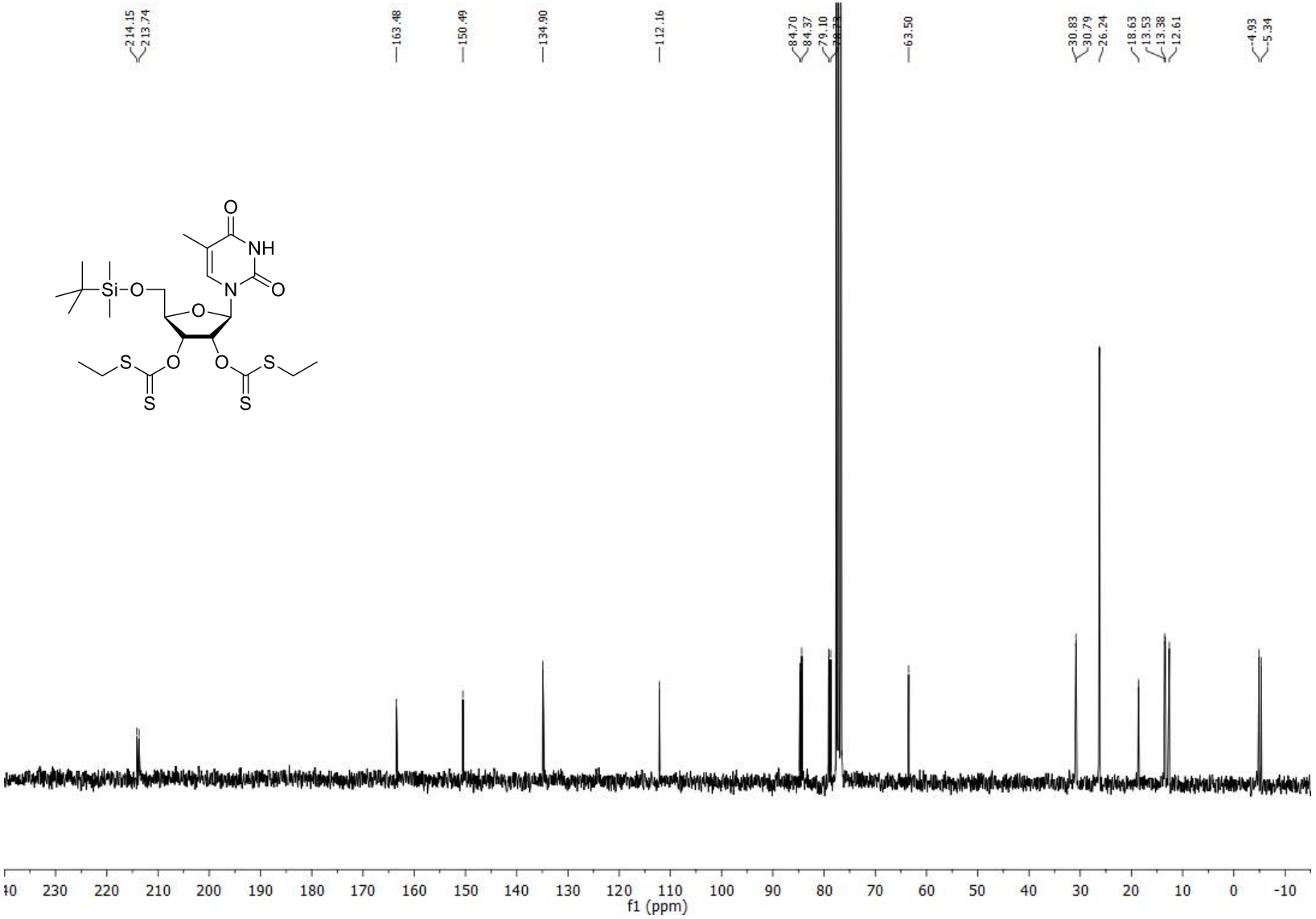


5'-*O*-(*tert*-Butyldimethylsilyl)-2',3'-bis-*O*-[(ethylthio)thiocarbonyl]-β-D-5-methyluridine (6b)

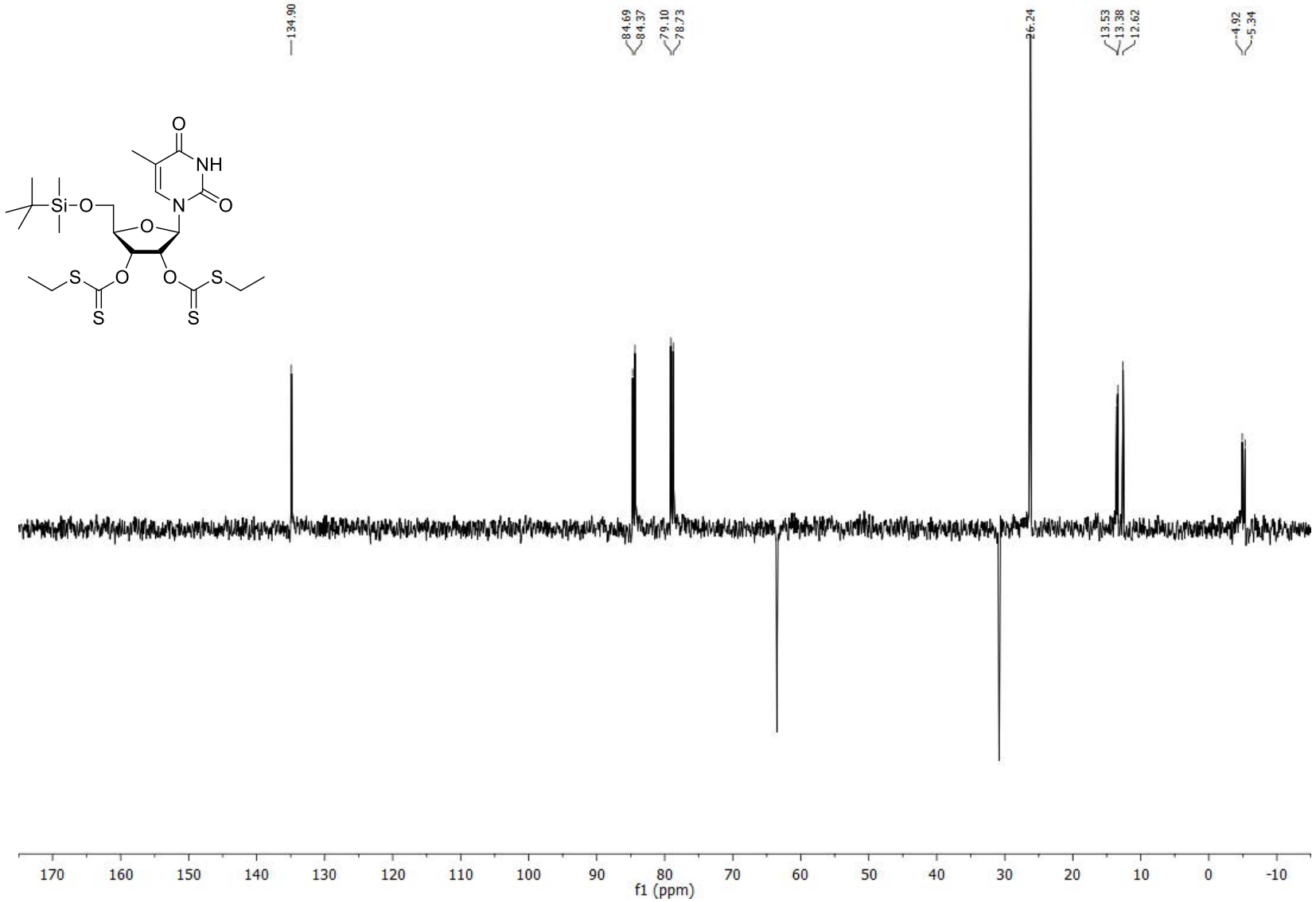
^{1}H-NMR (300.13 MHz, $CDCl_3$)



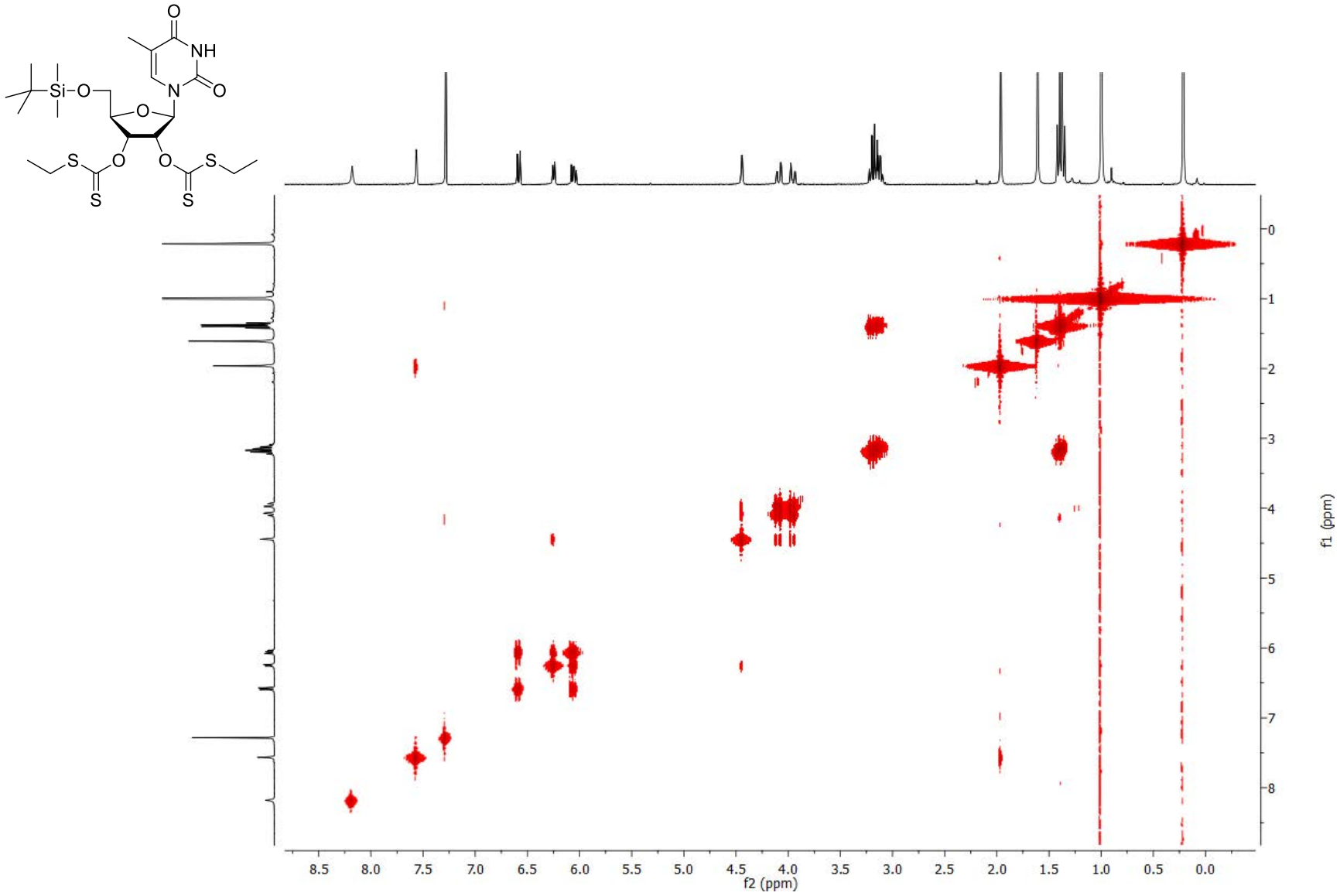
^{13}C-NMR (75.5 MHz, $CDCl_3$)



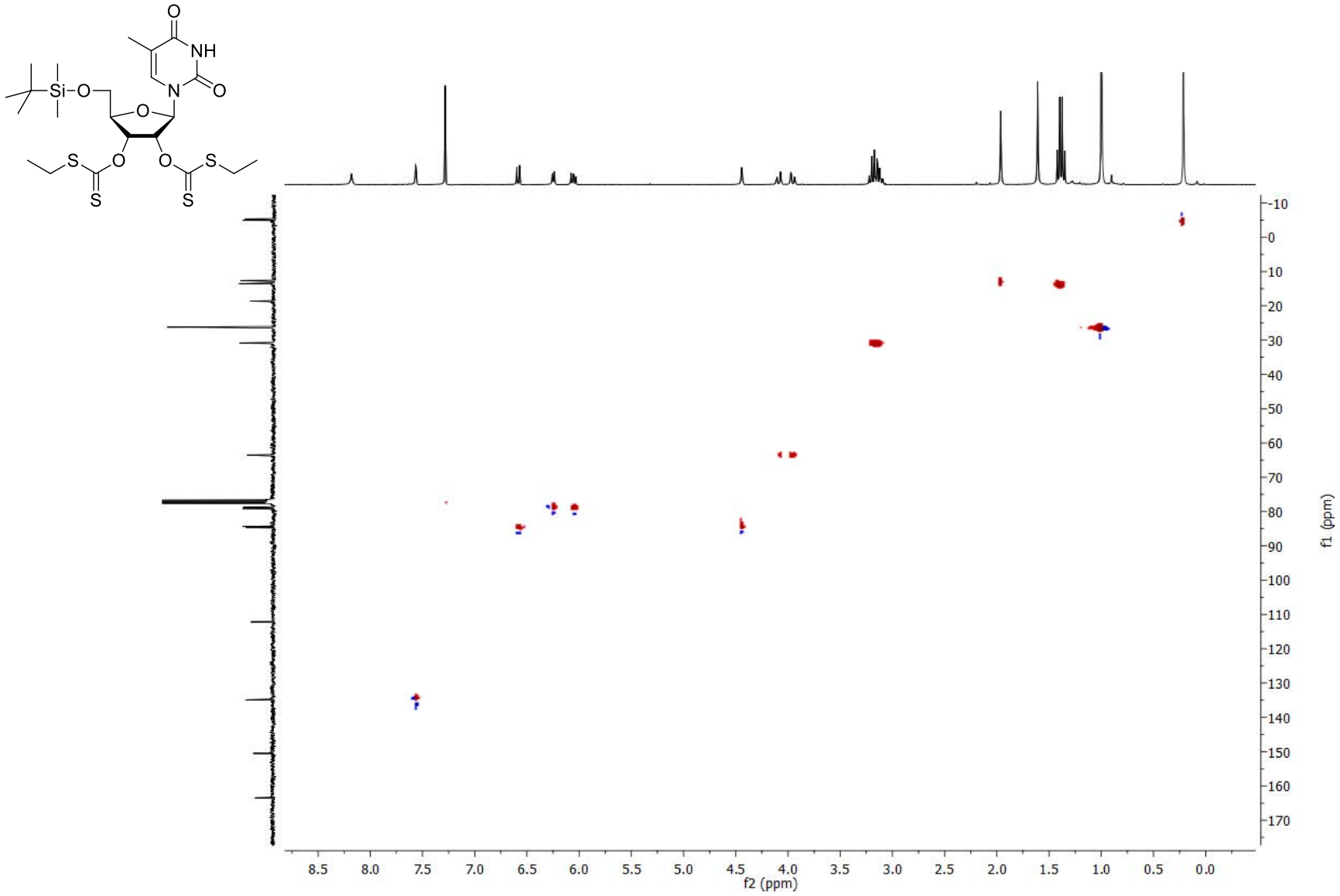
DEPT NMR (75.5 MHz, $CDCl_3$)



COSY NMR ($CDCl_3$)

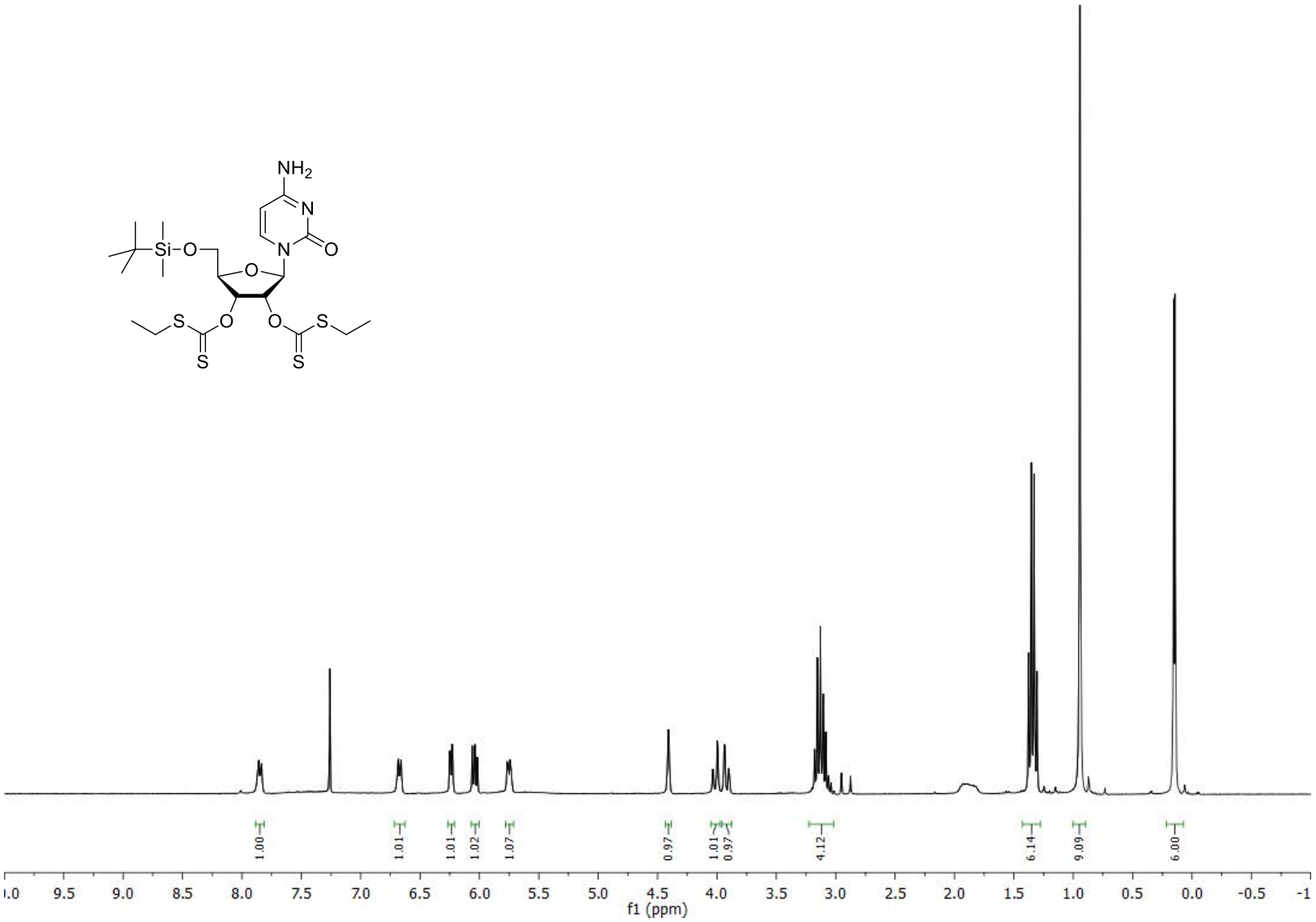


HSQC NMR ($CDCl_3$)

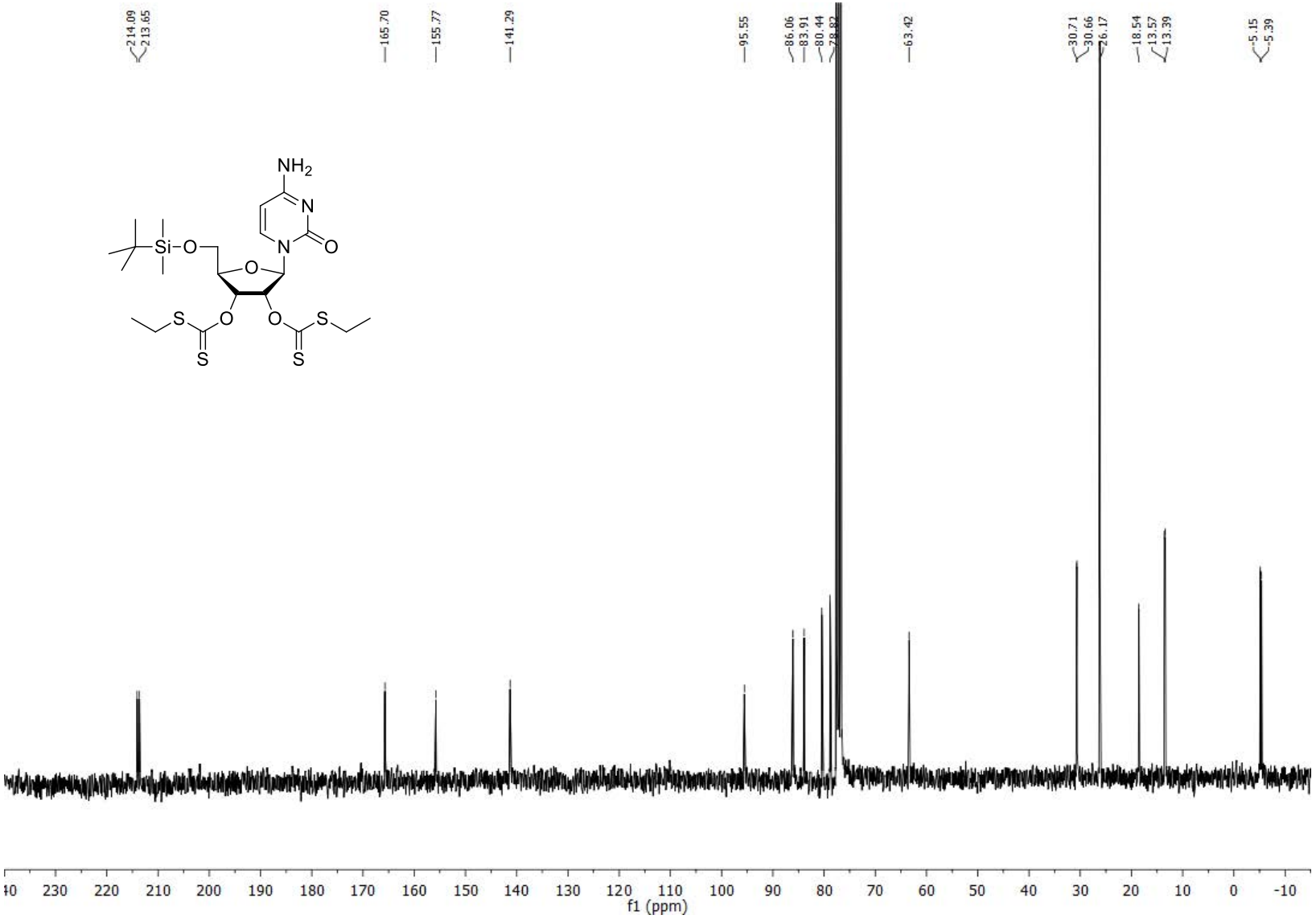


5'-*O*-(*tert*-Butyldimethylsilyl)-2',3'-bis-*O*-[(ethylthio)thiocarbonyl]-β-D-cytidine (6c)

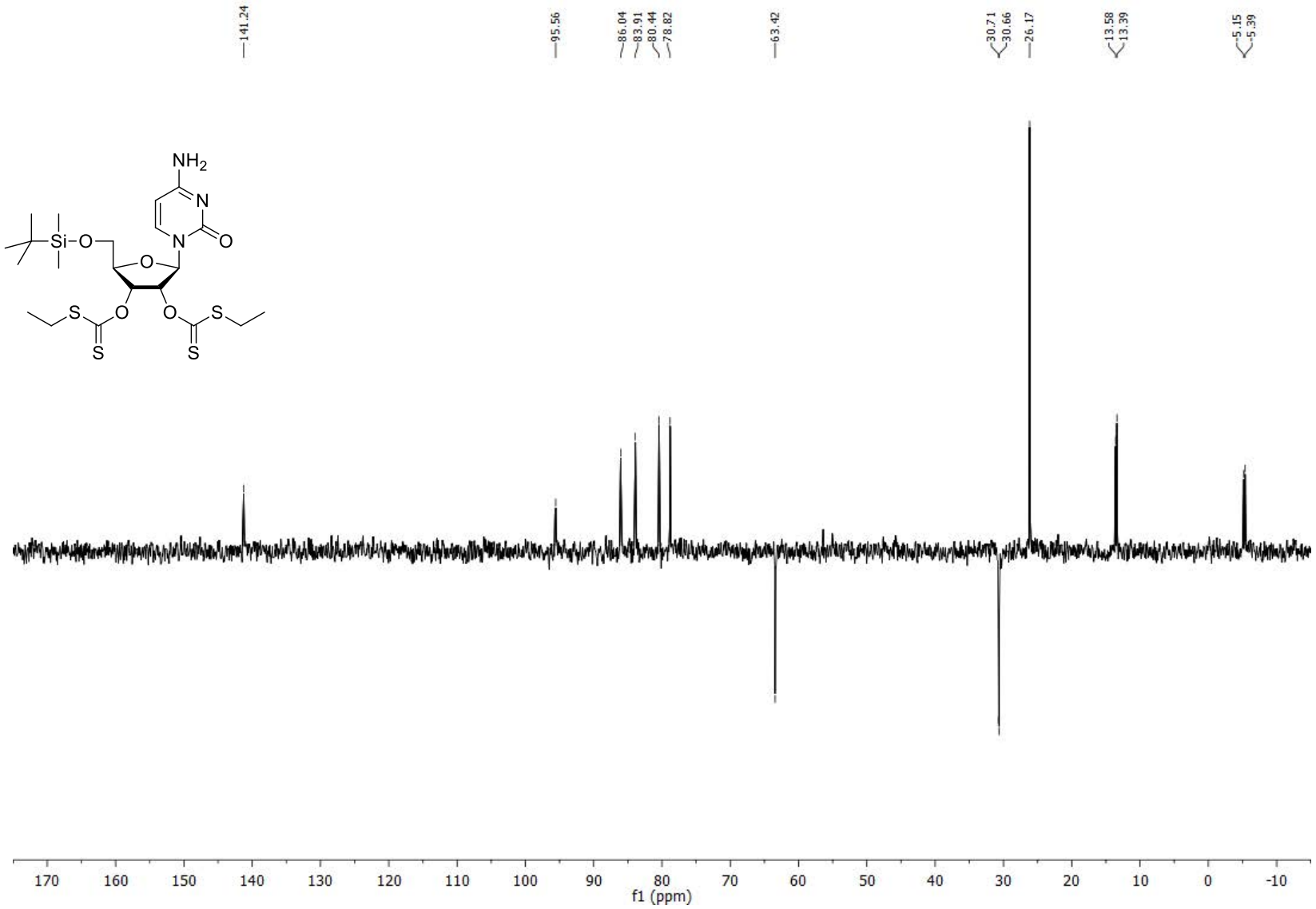
^{1}H-NMR (300.13 MHz, $CDCl_3$)



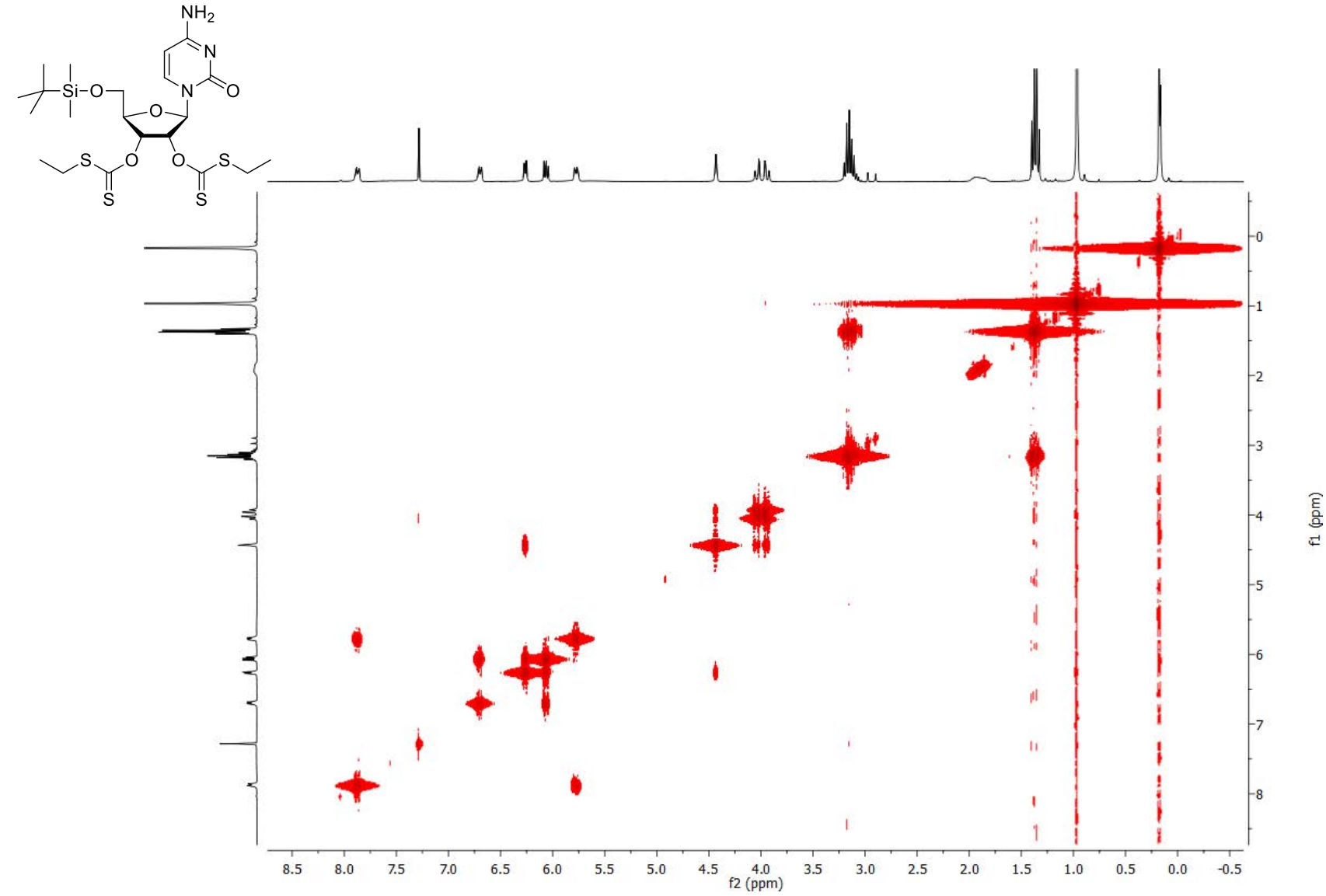
^{13}C-NMR (75.5 MHz, $CDCl_3$)



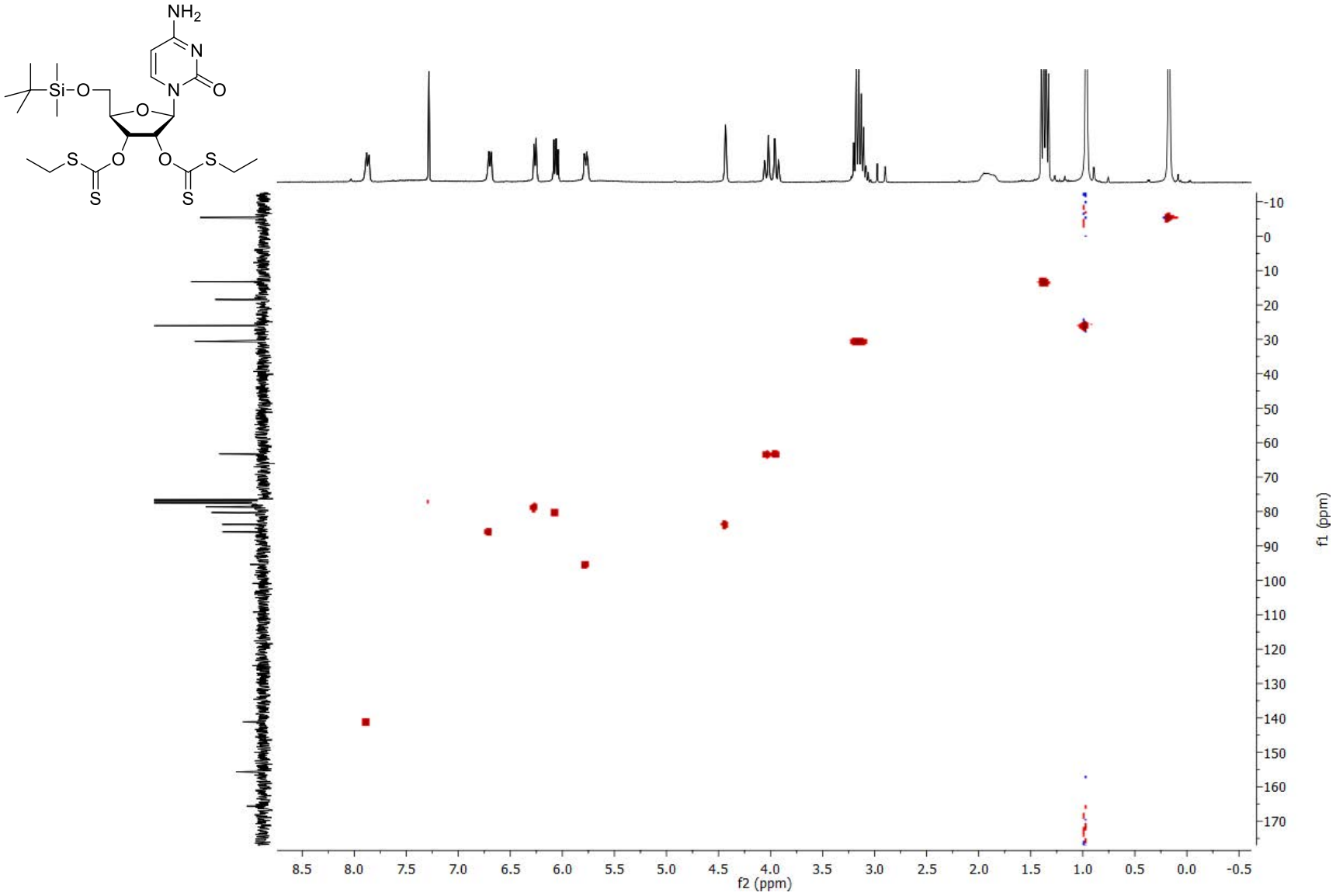
DEPT NMR (75.5 MHz, $CDCl_3$)



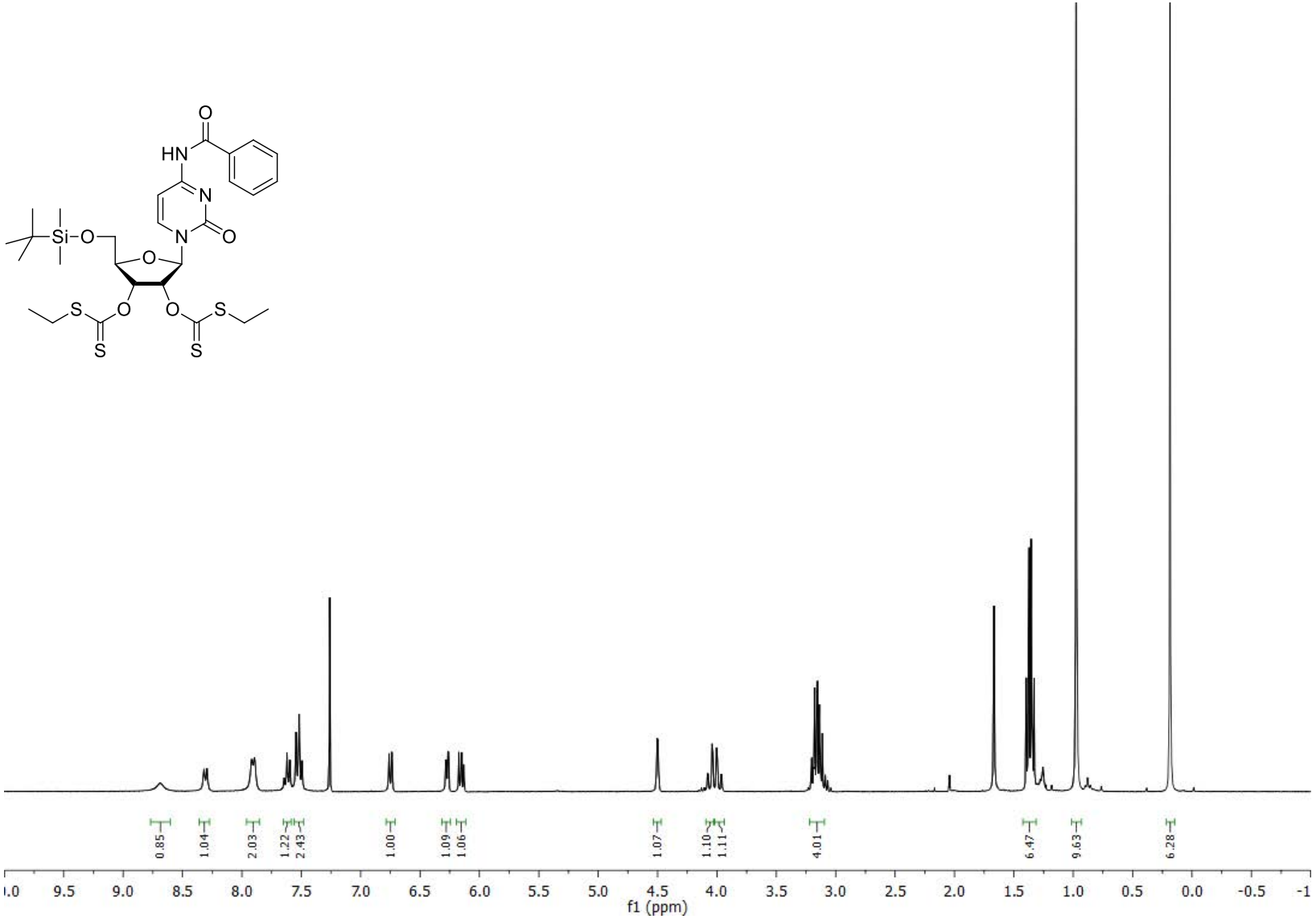
COSY NMR ($CDCl_3$)



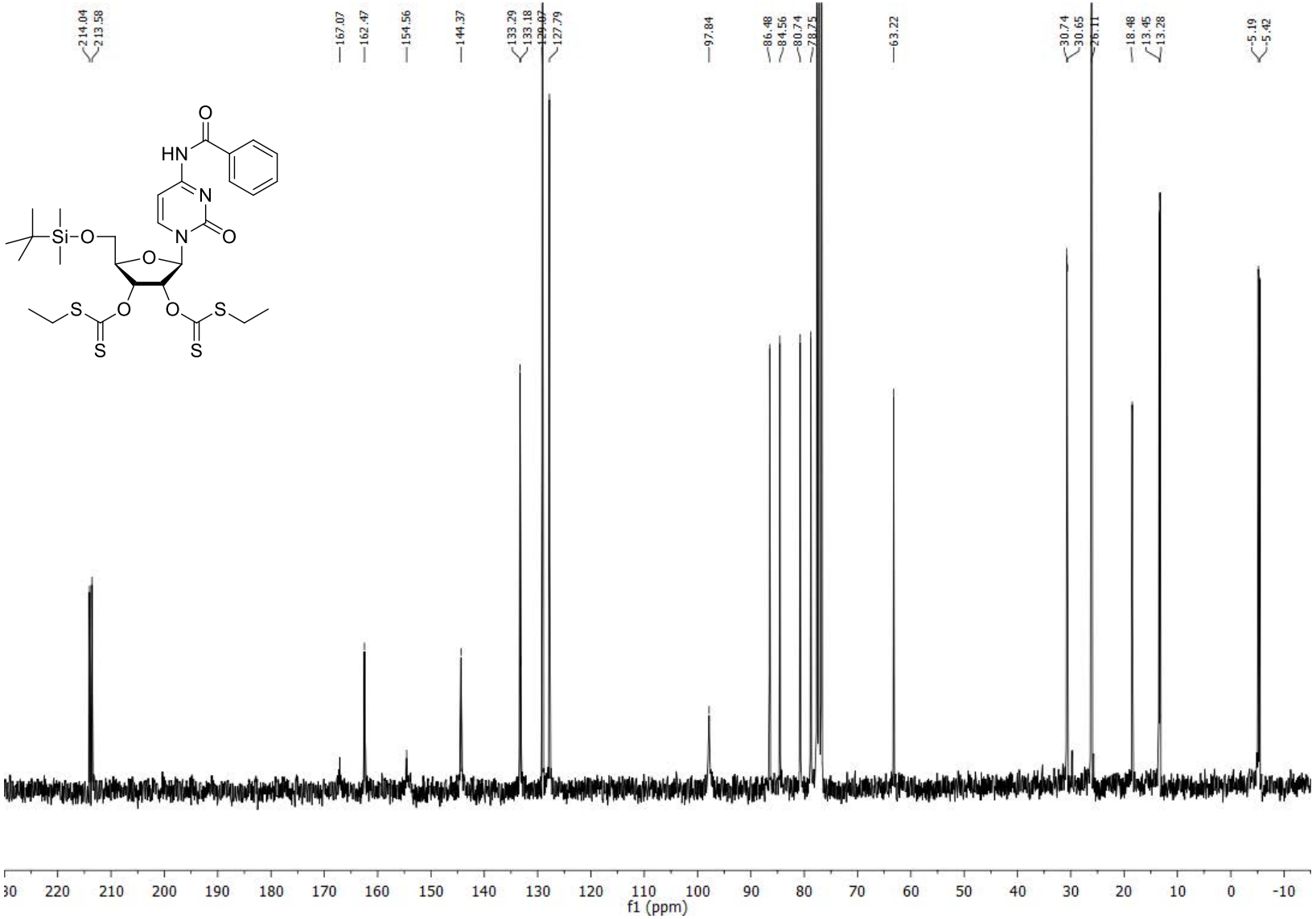
HSQC NMR ($CDCl_3$)



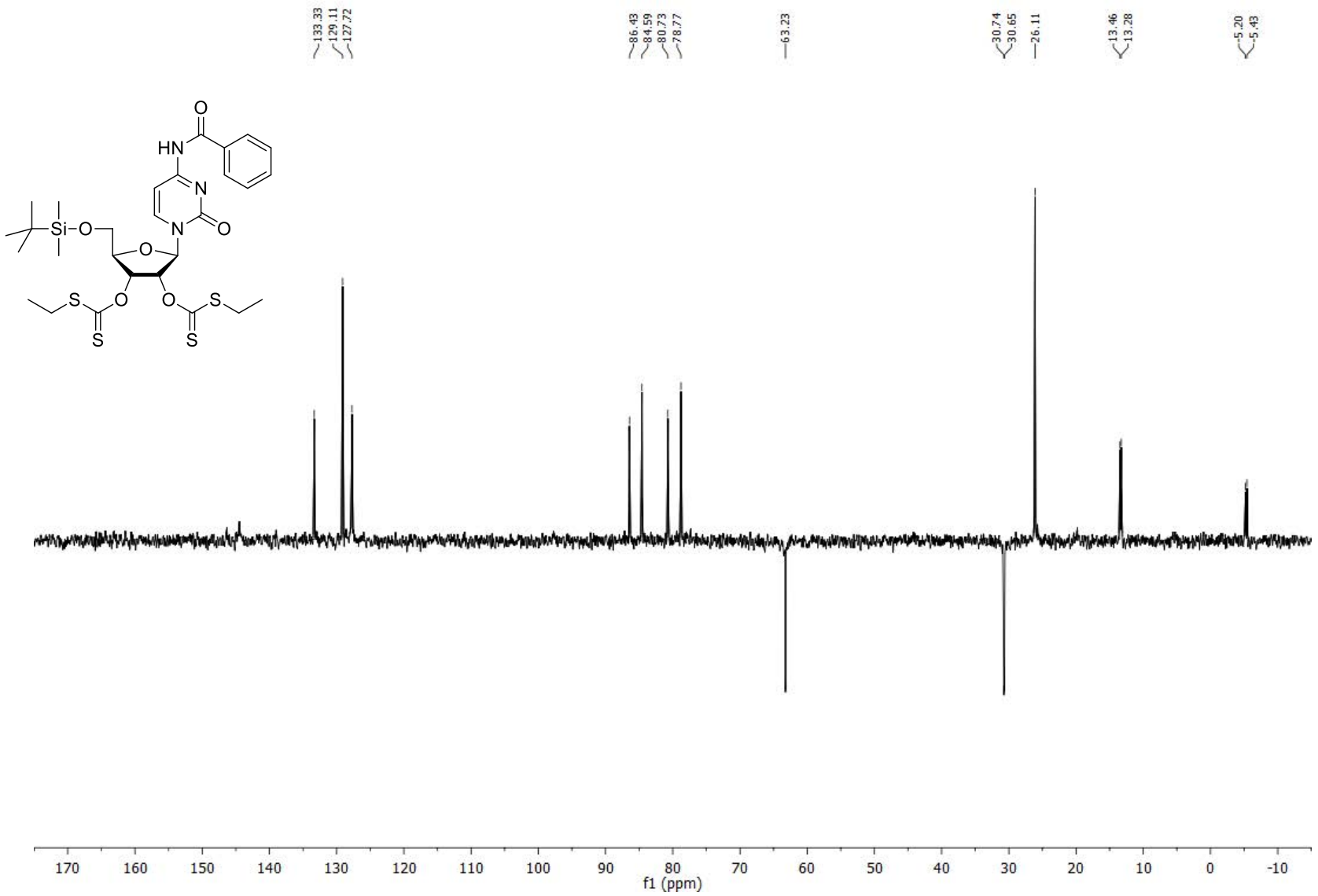
^{1}H-NMR (300.13 MHz, $CDCl_3$)



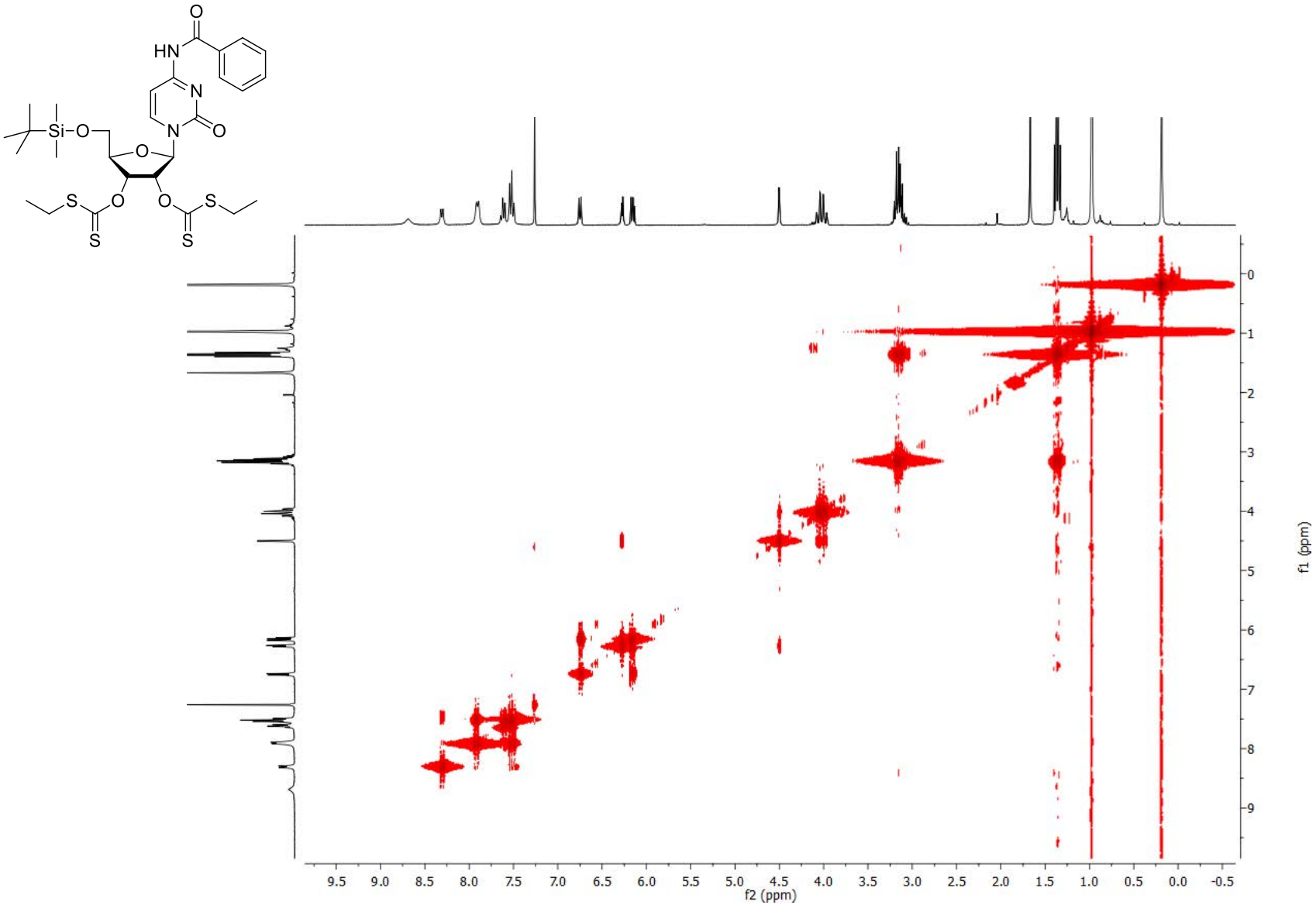
^{13}C-NMR (75.5 MHz, $CDCl_3$)



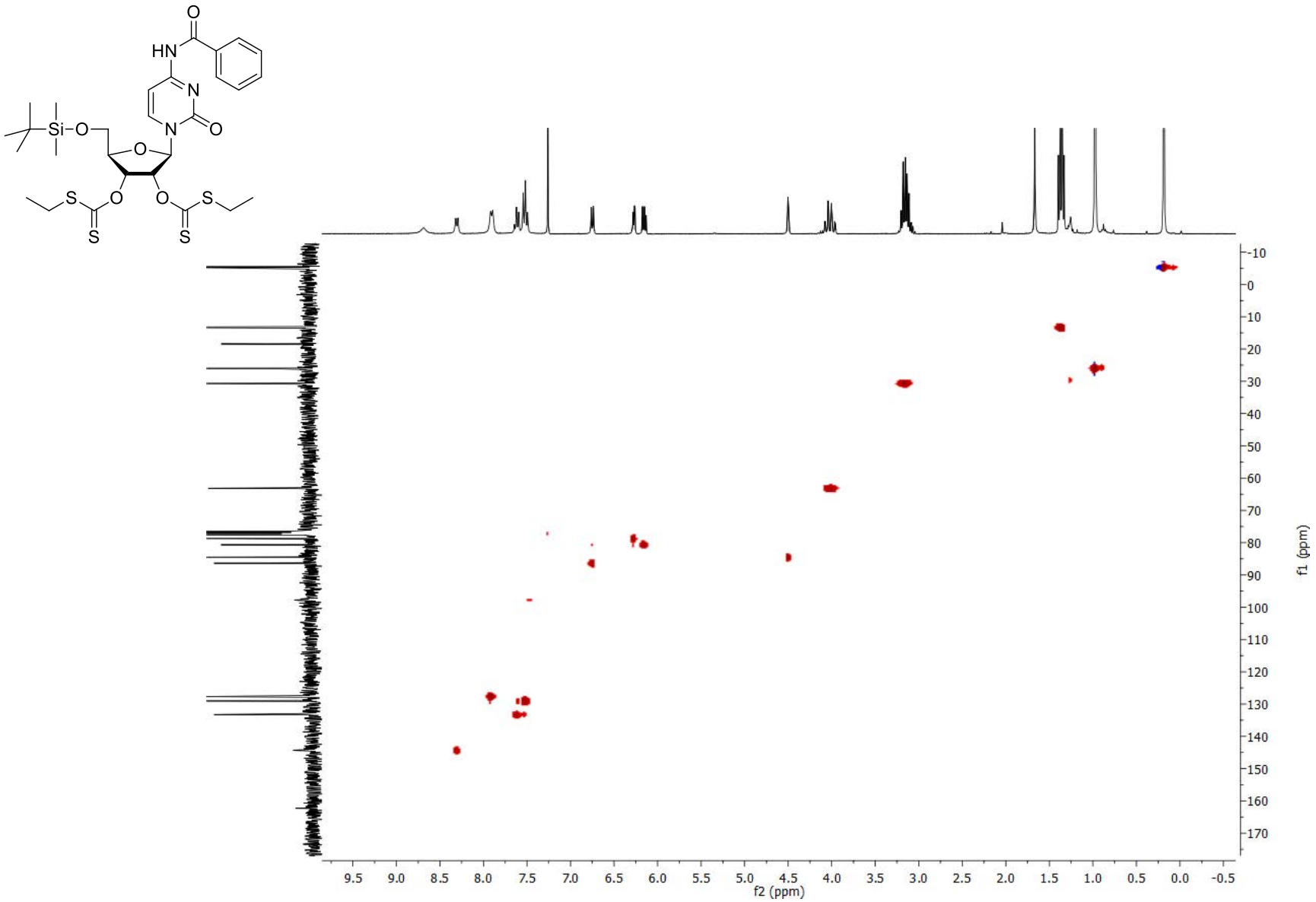
DEPT NMR (75.5 MHz, $CDCl_3$)



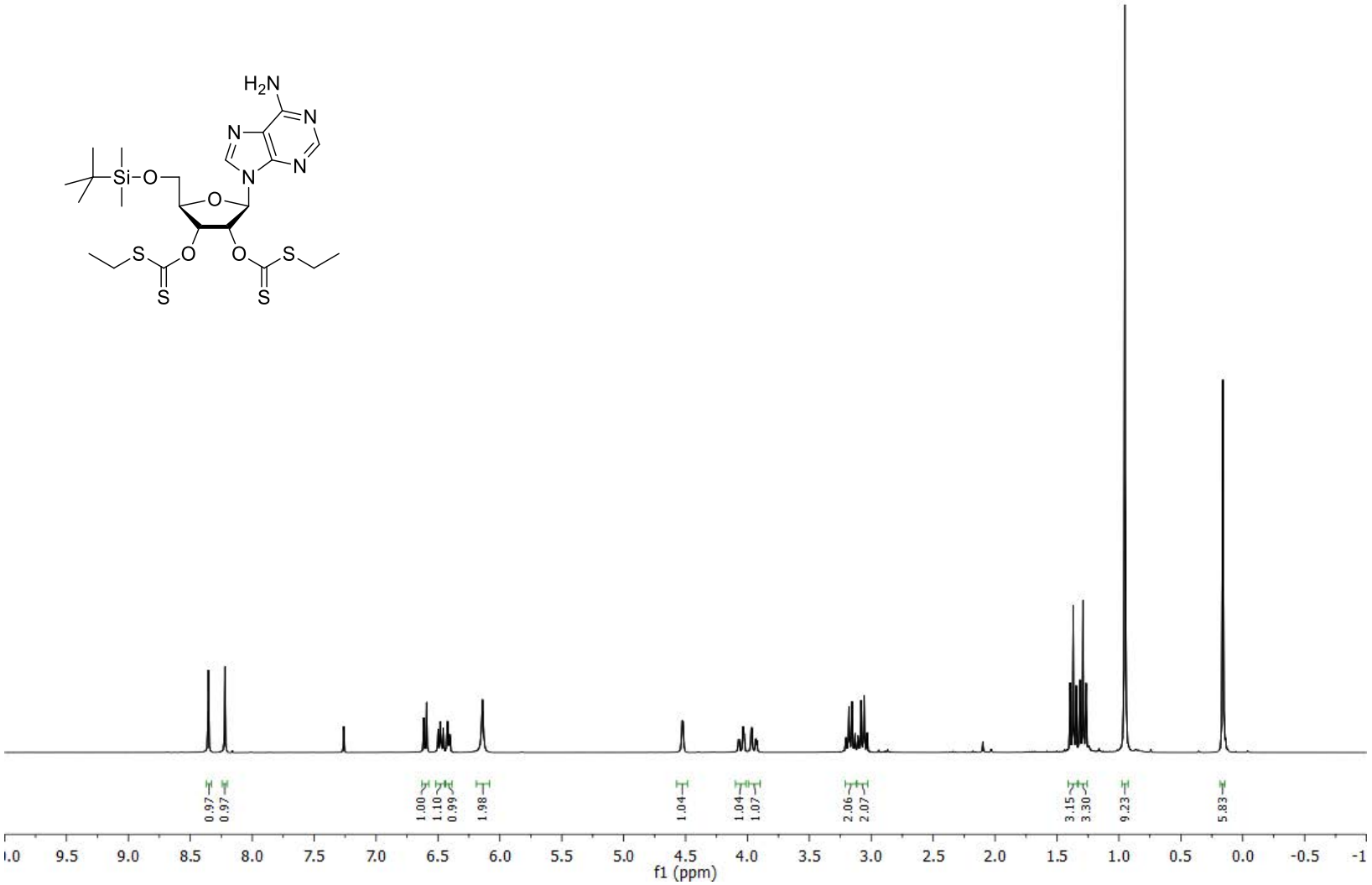
COSY NMR (75.5 MHz, $CDCl_3$)



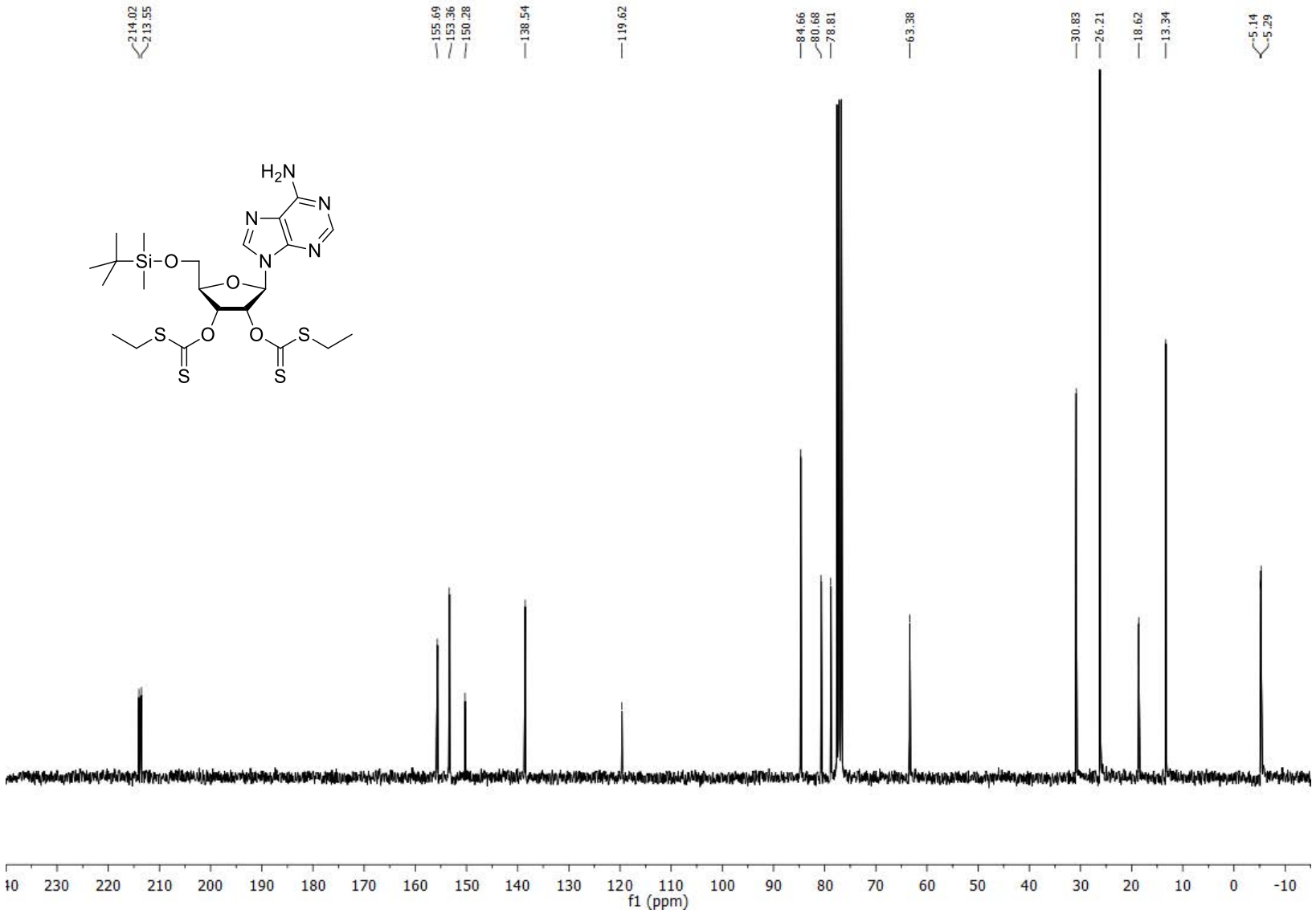
HSQC NMR (75.5 MHz, $CDCl_3$)



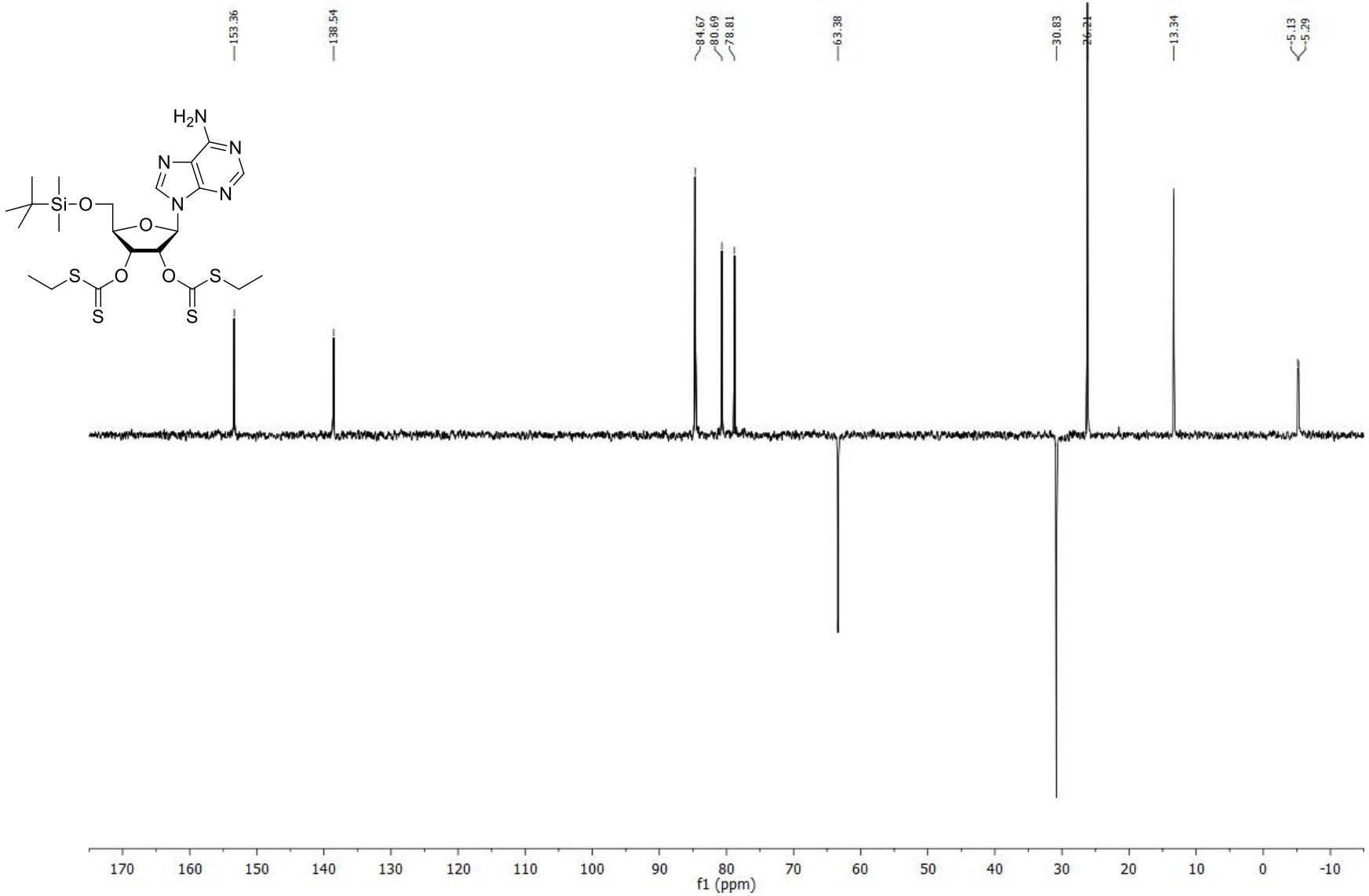
^{1}H-NMR (300.13 MHz, $CDCl_3$)



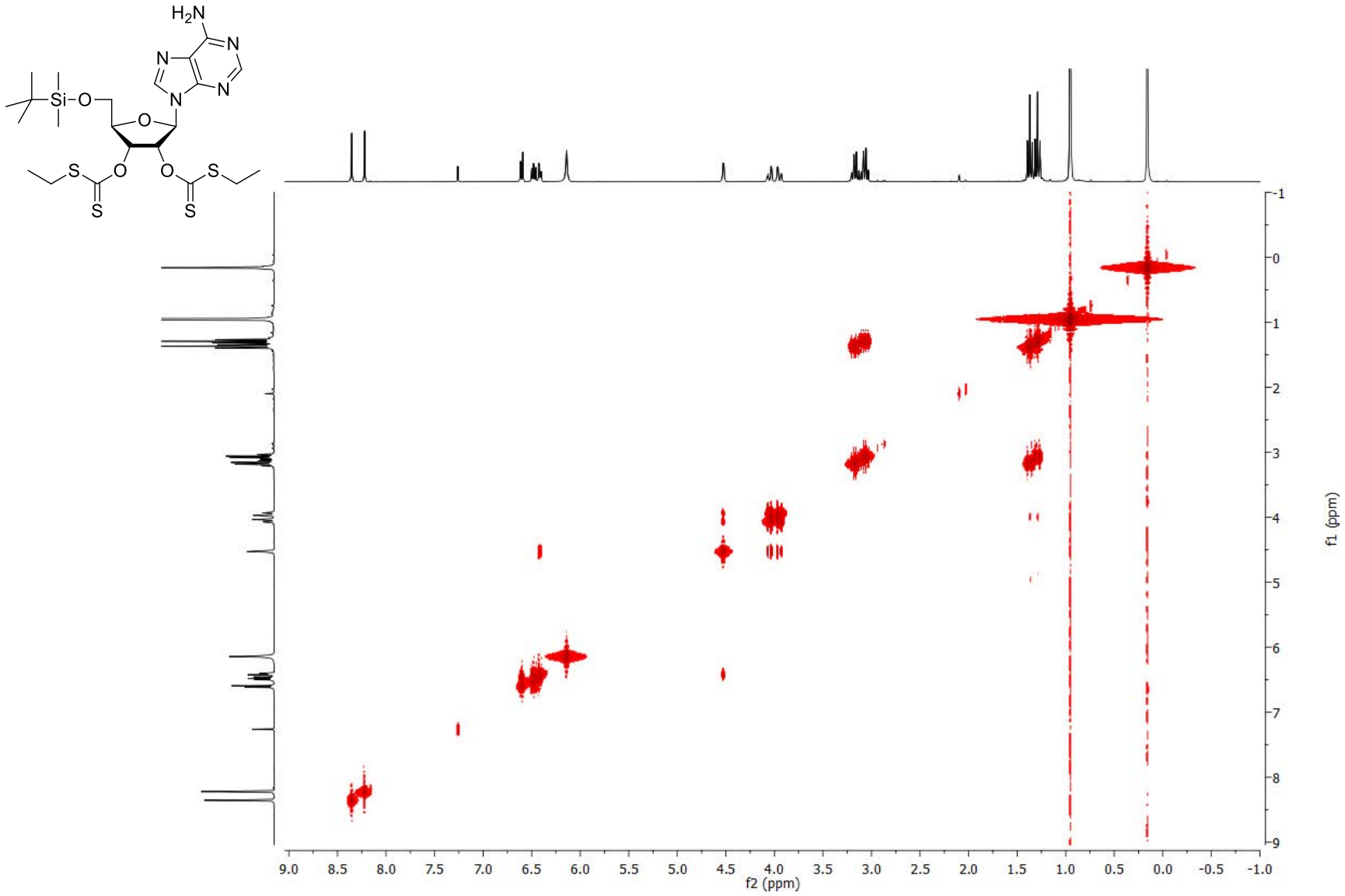
^{13}C-NMR (75.5 MHz, $CDCl_3$)



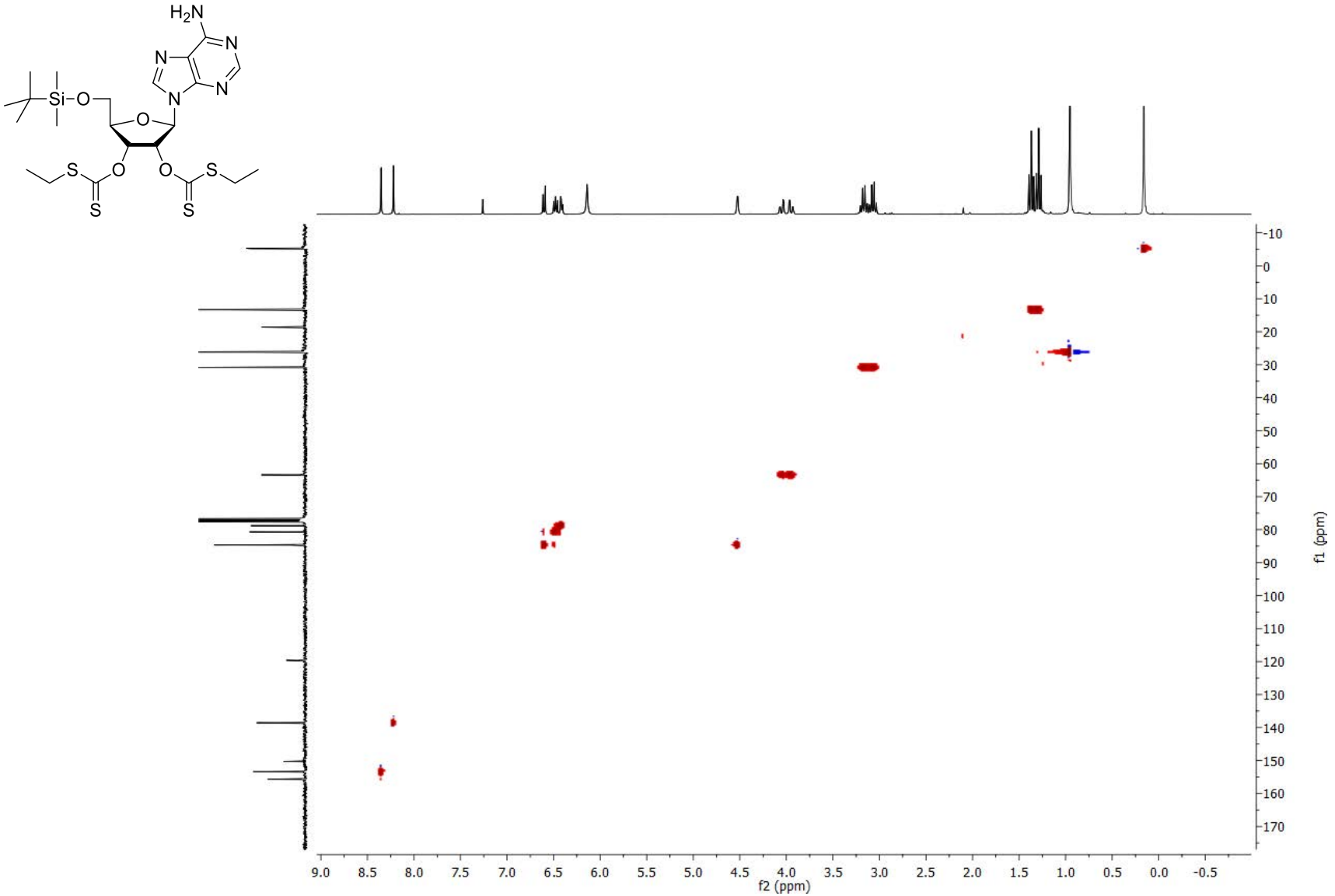
DEPT NMR (75.5 MHz, $CDCl_3$)



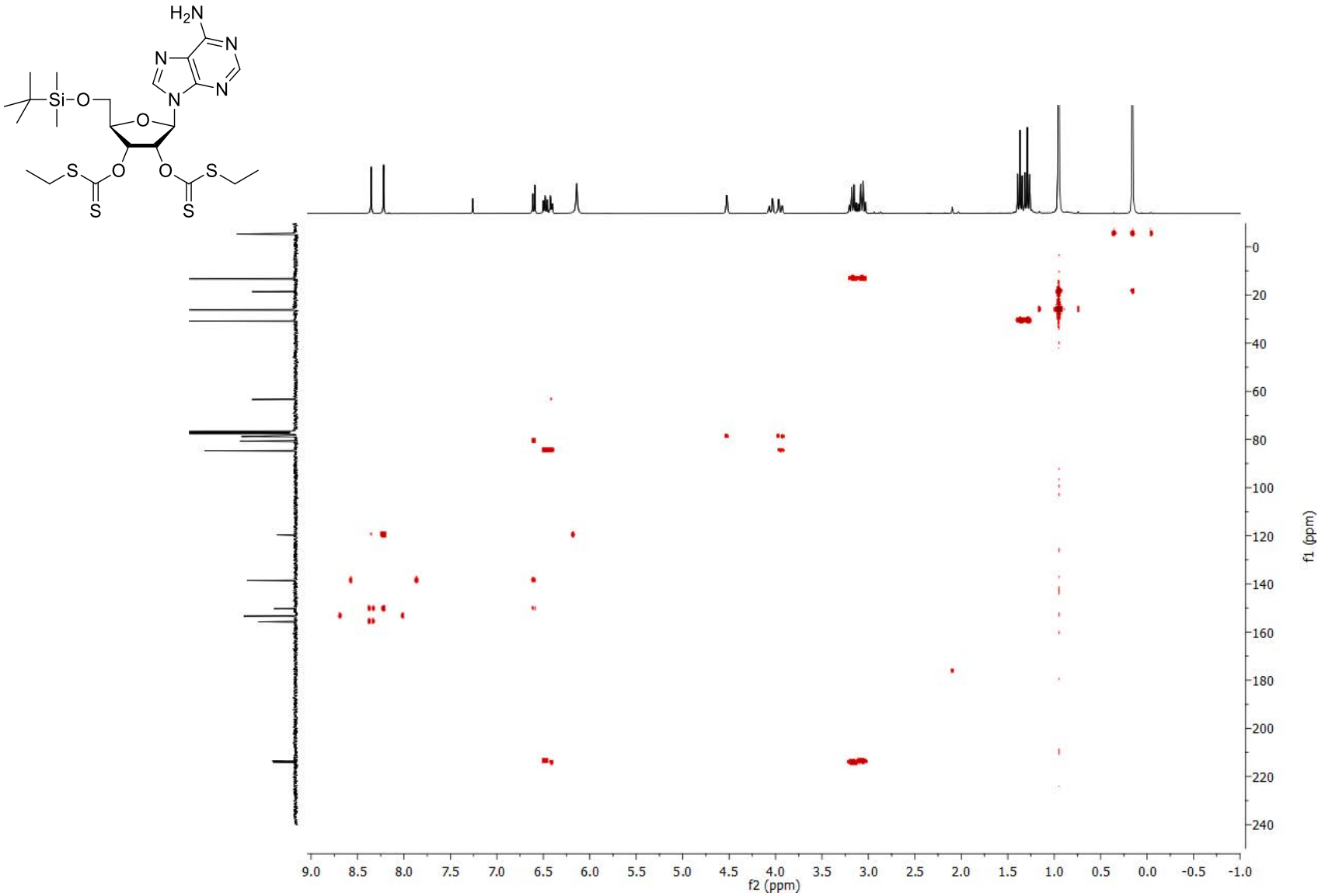
COSY NMR ($CDCl_3$)



HSQC NMR ($CDCl_3$)

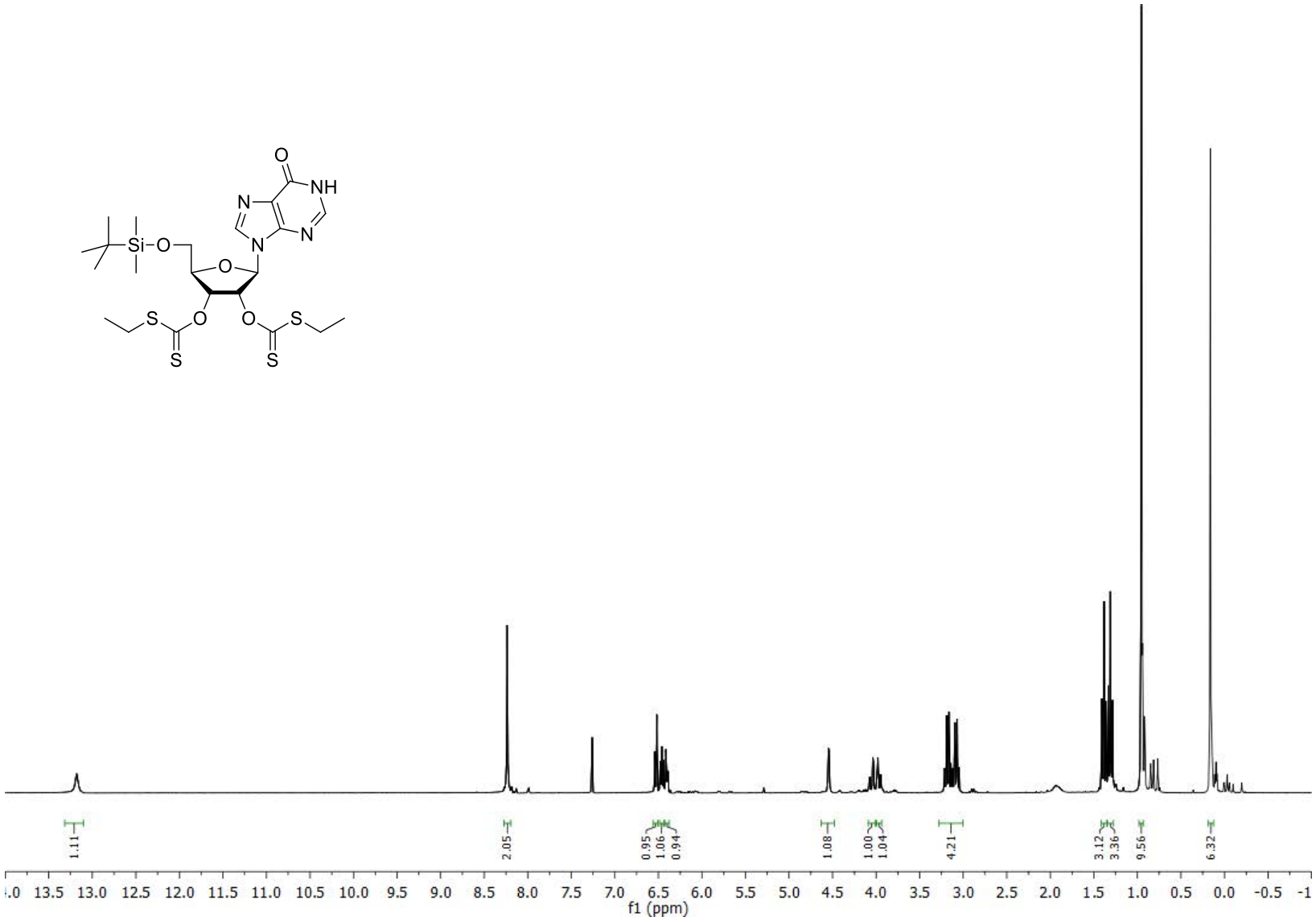


HMBC NMR ($CDCl_3$)

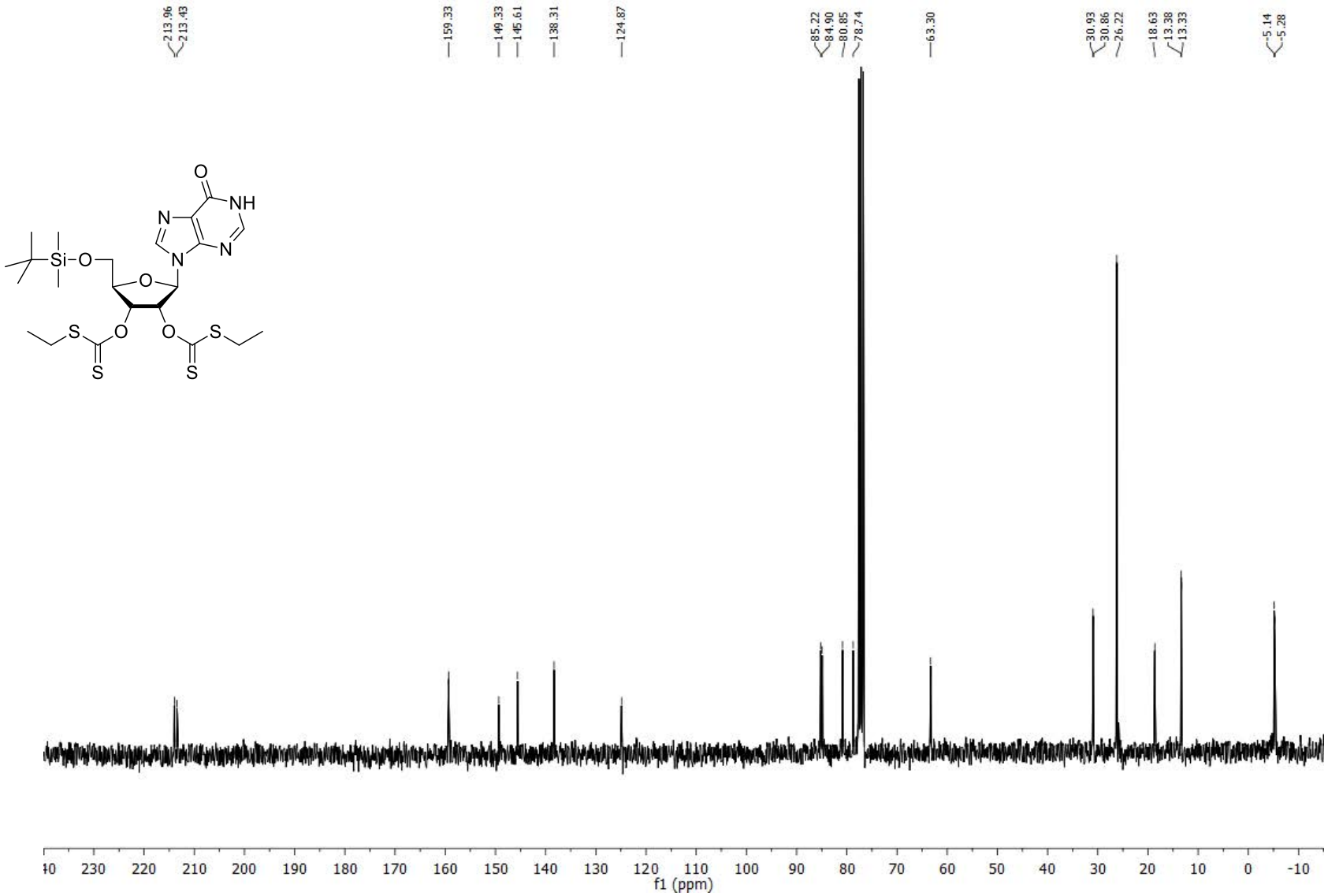


5'-*O*-(*tert*-Butyldimethylsilyl)-2',3'-bis-*O*-[(ethylthio)thiocarbonyl]-β-D-inosine (6f)

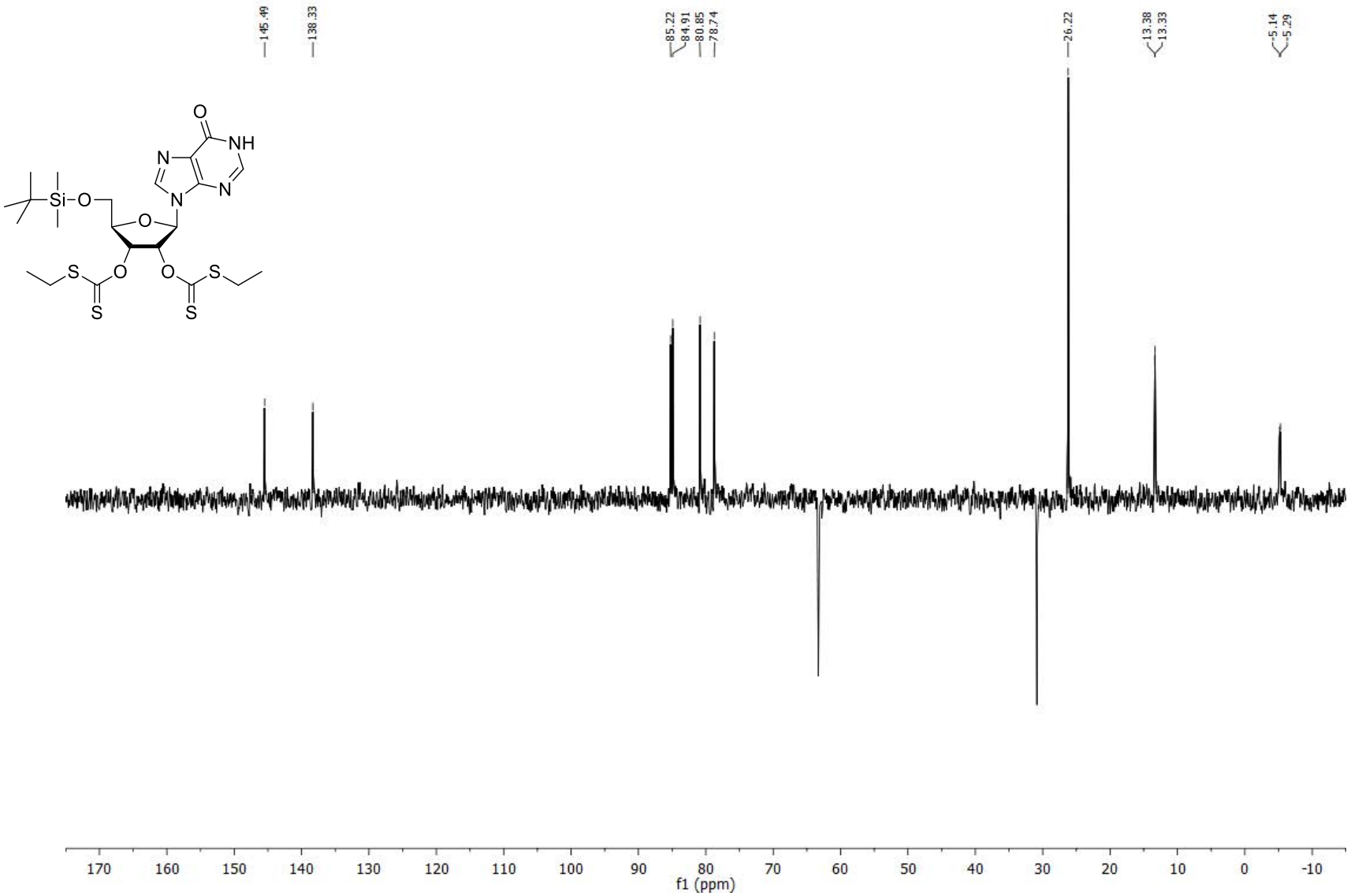
^{1}H-NMR (300.13 MHz, $CDCl_3$)



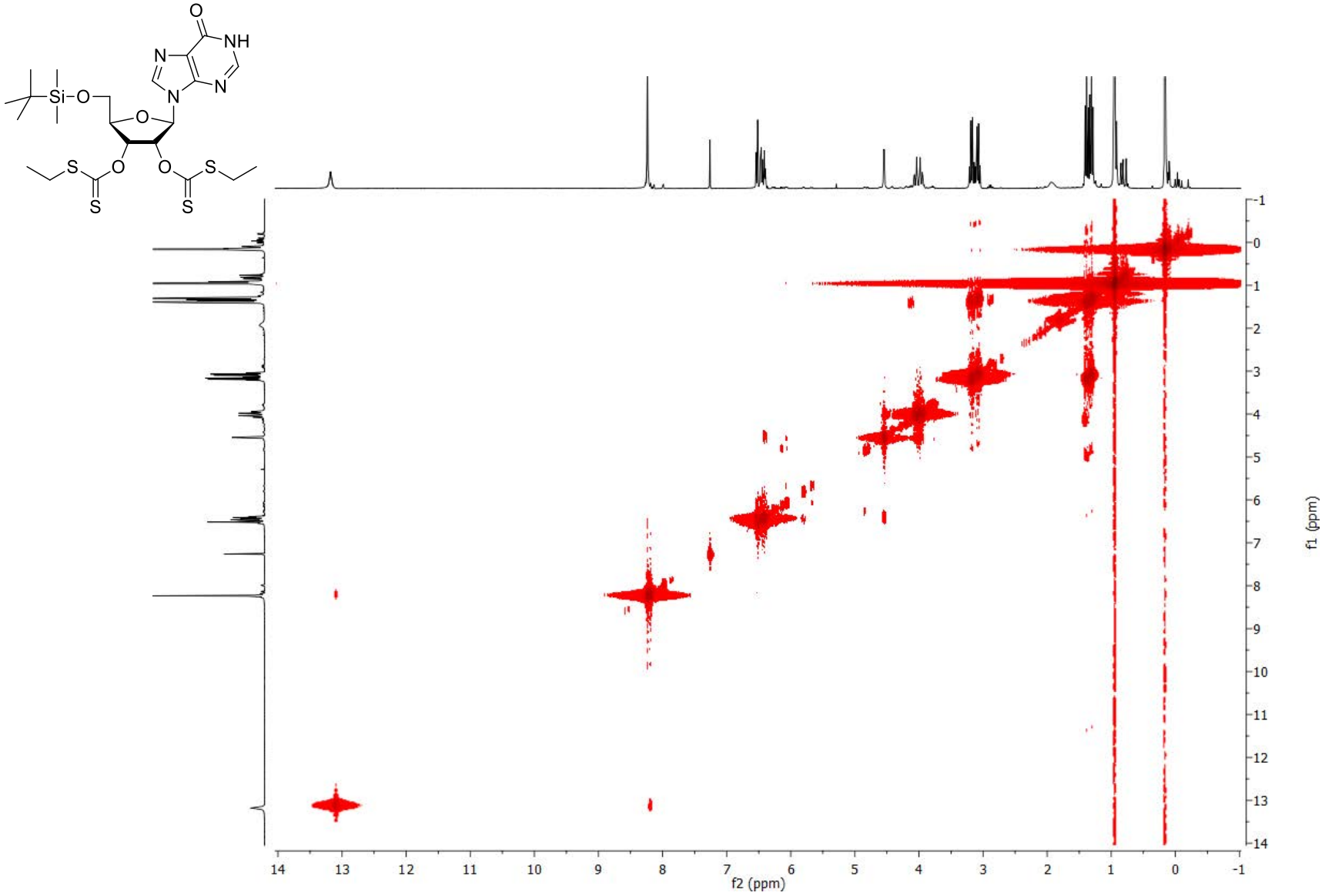
^{13}C-NMR (75.5 MHz, $CDCl_3$)



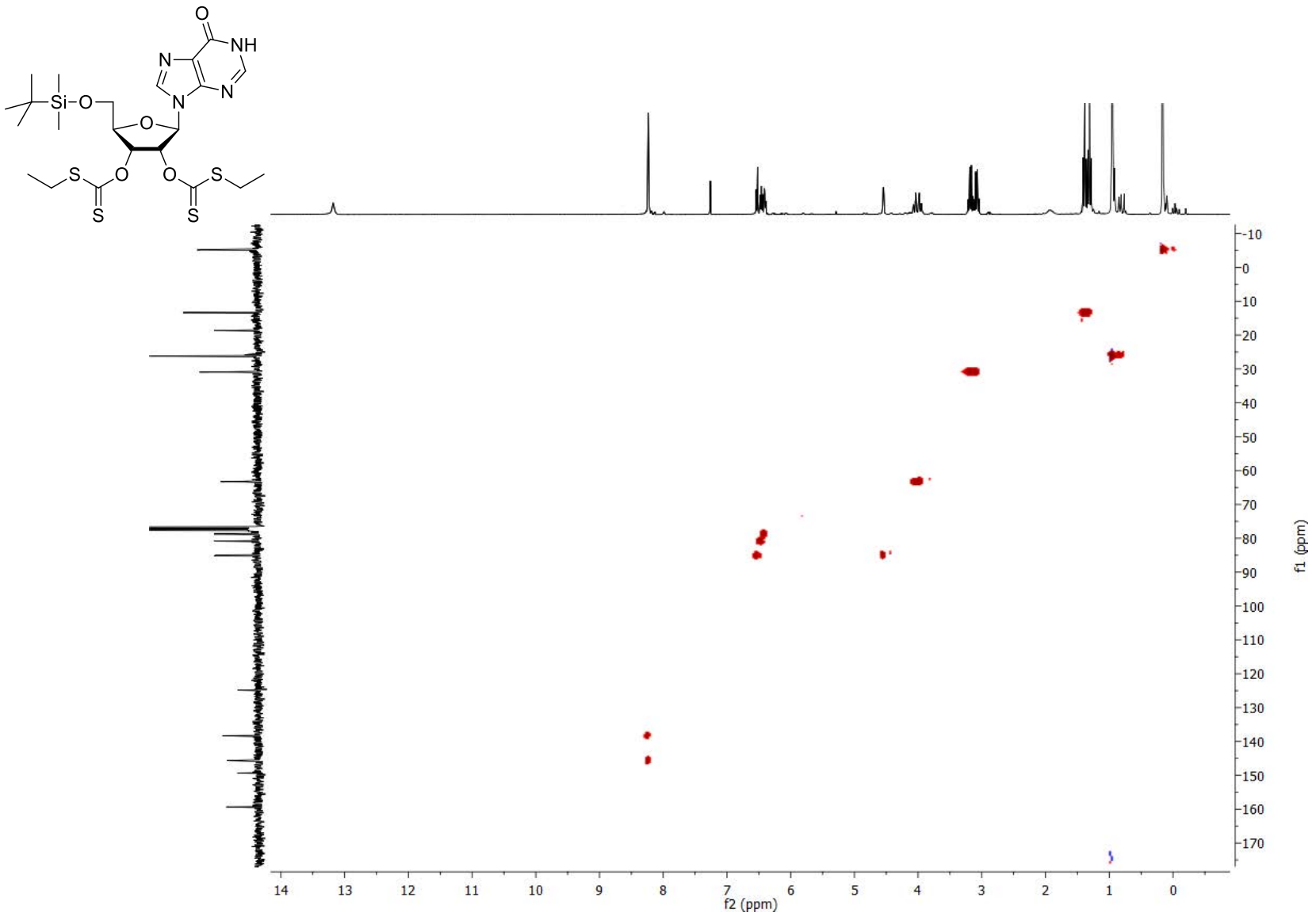
DEPT NMR (75.5 MHz, $CDCl_3$)



COSY NMR ($CDCl_3$)

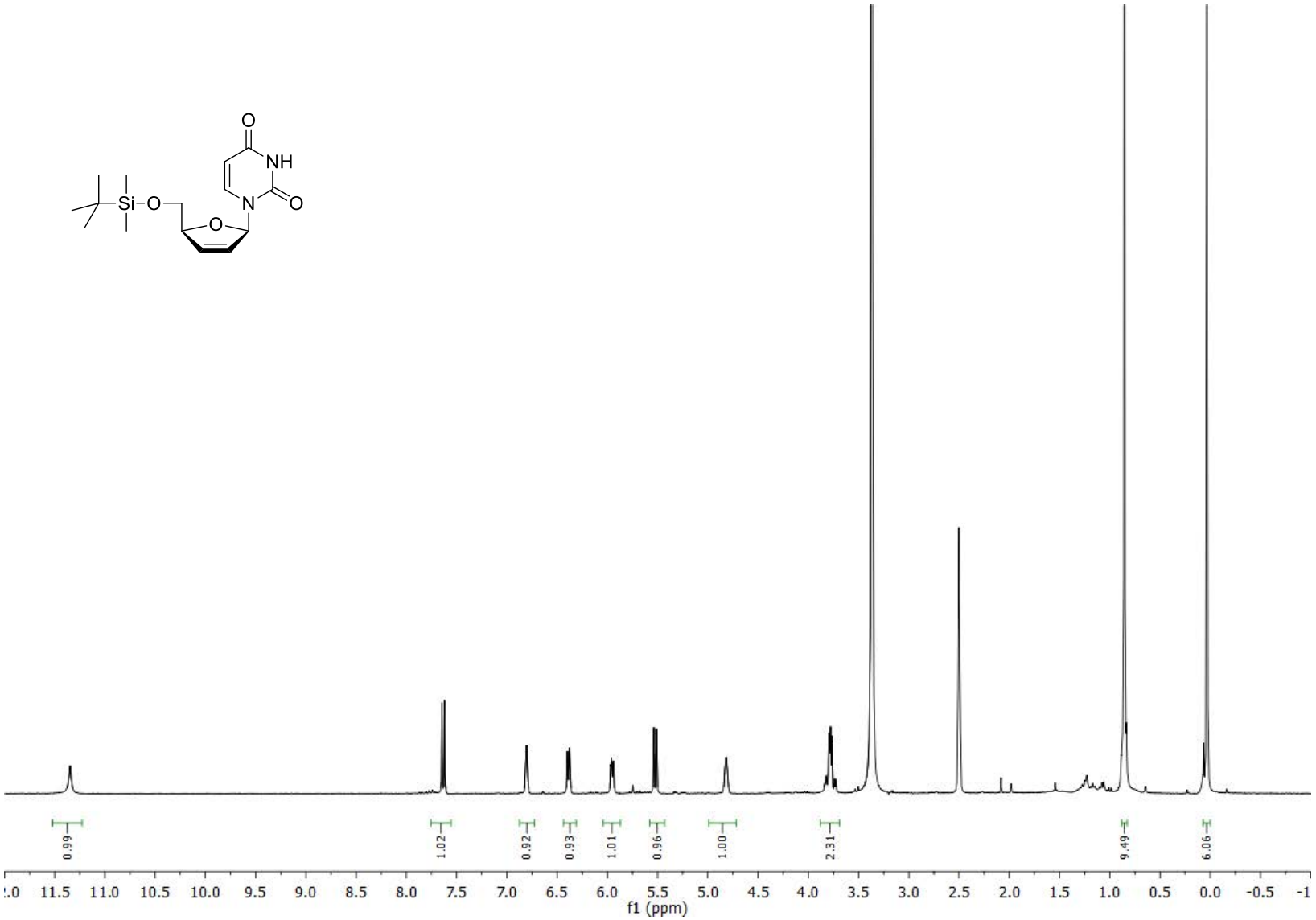


HSQC NMR ($CDCl_3$)

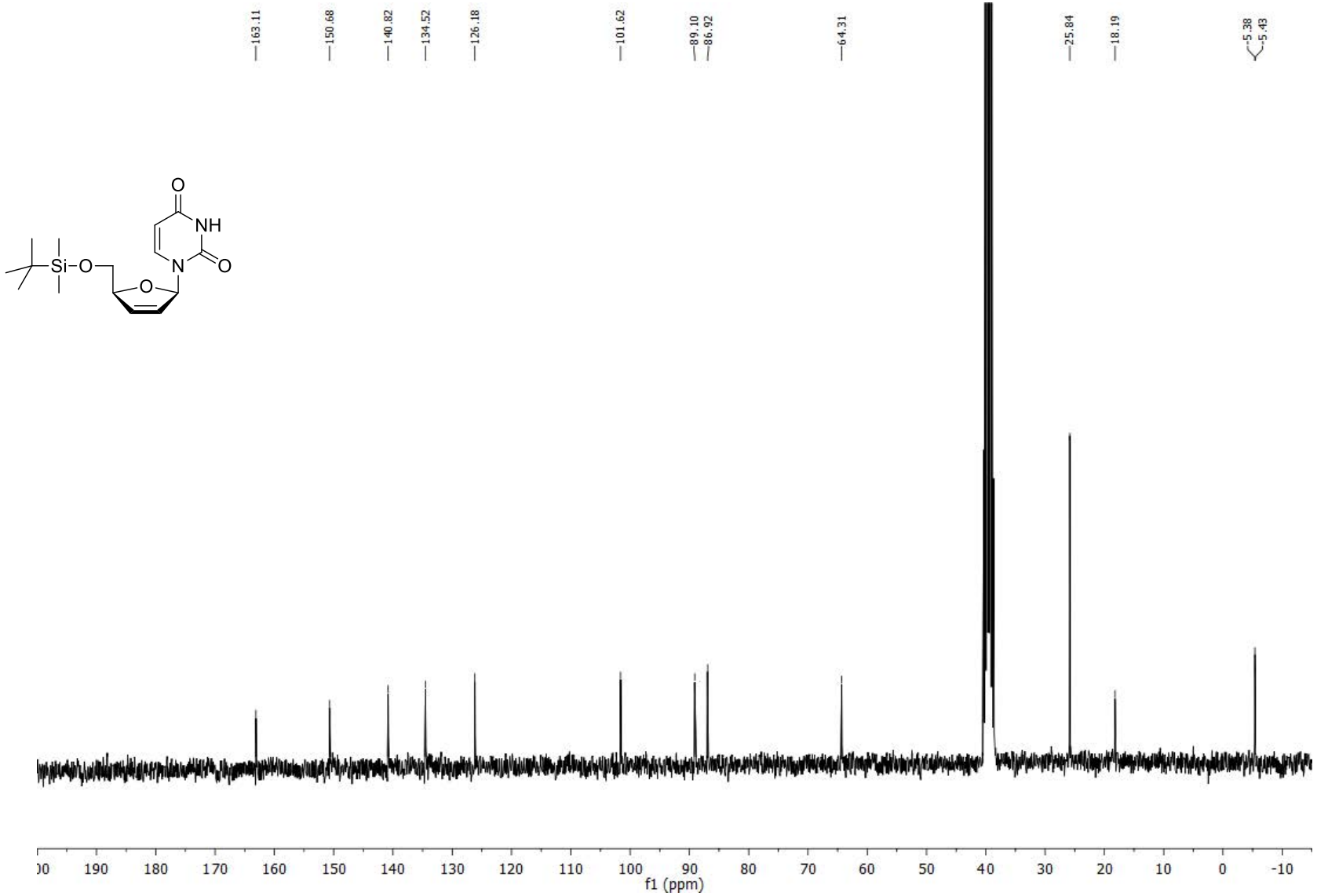


5'-*O*-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy-β-D-uridine (7a)

^{1}H-NMR (300.13 MHz, DMSO-d_6)

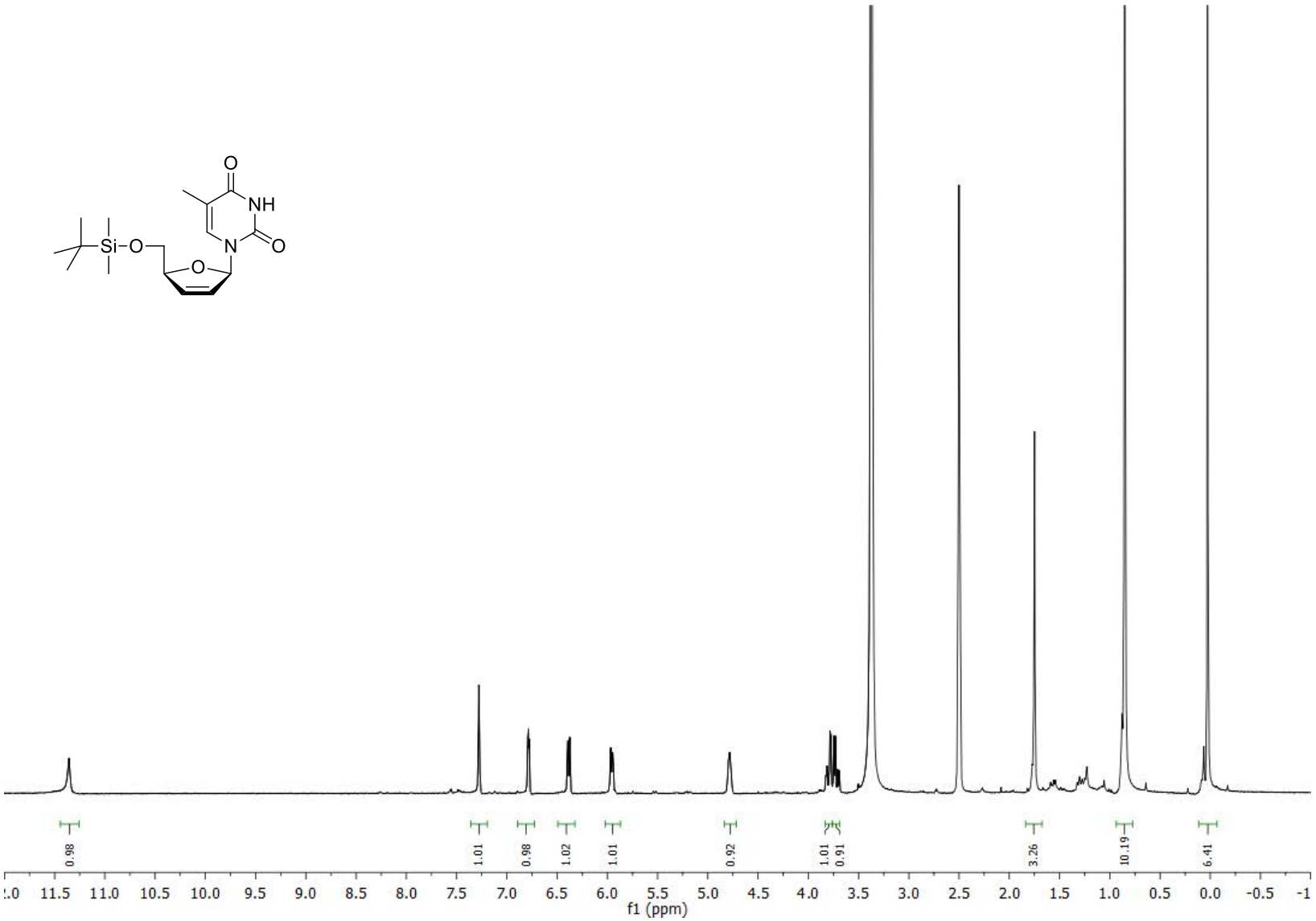


^{13}C-NMR (75.5 MHz, DMSO-d_6)

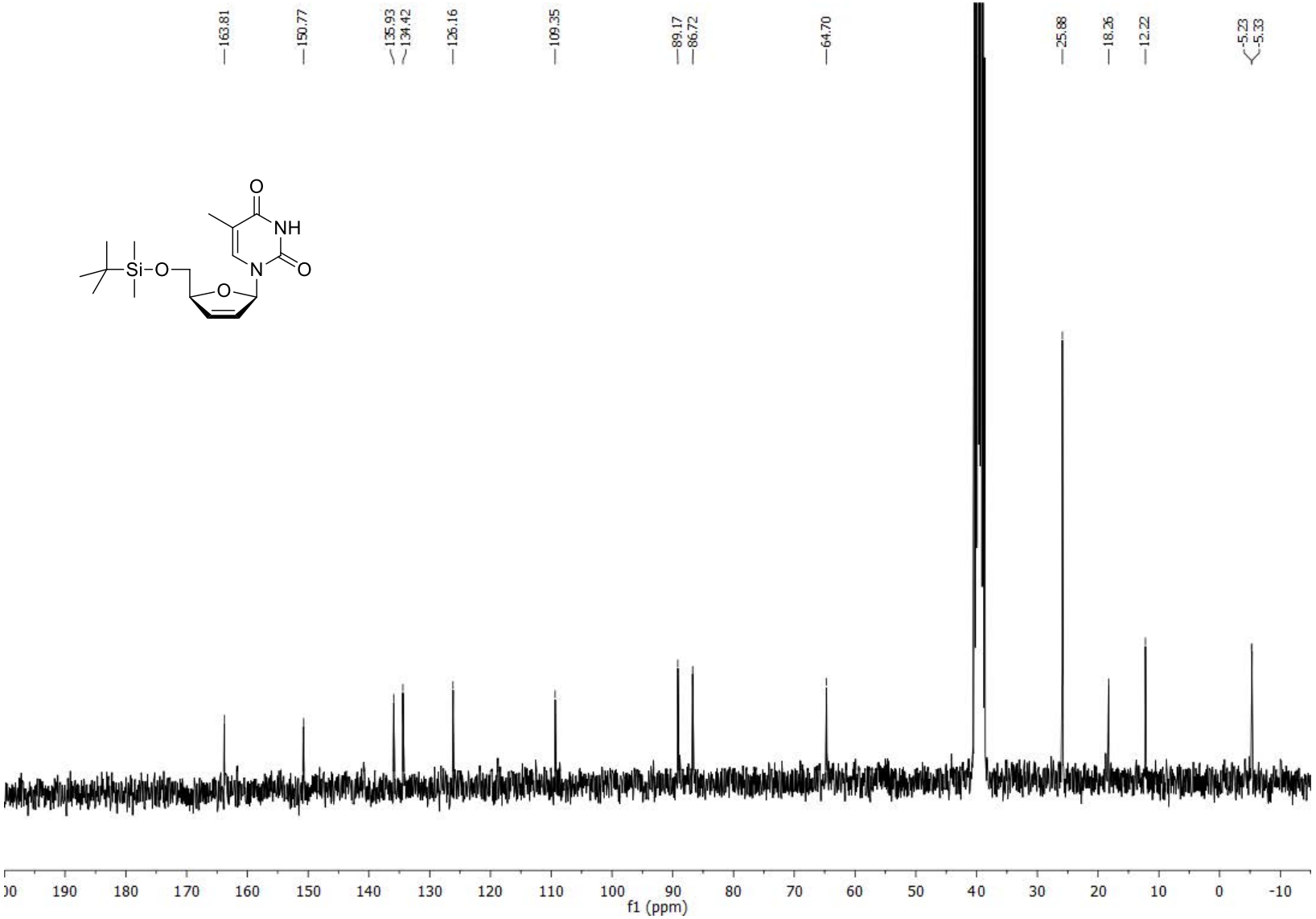


5'-*O*-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-3'-deoxy-β-D-5-thymidine (7b)

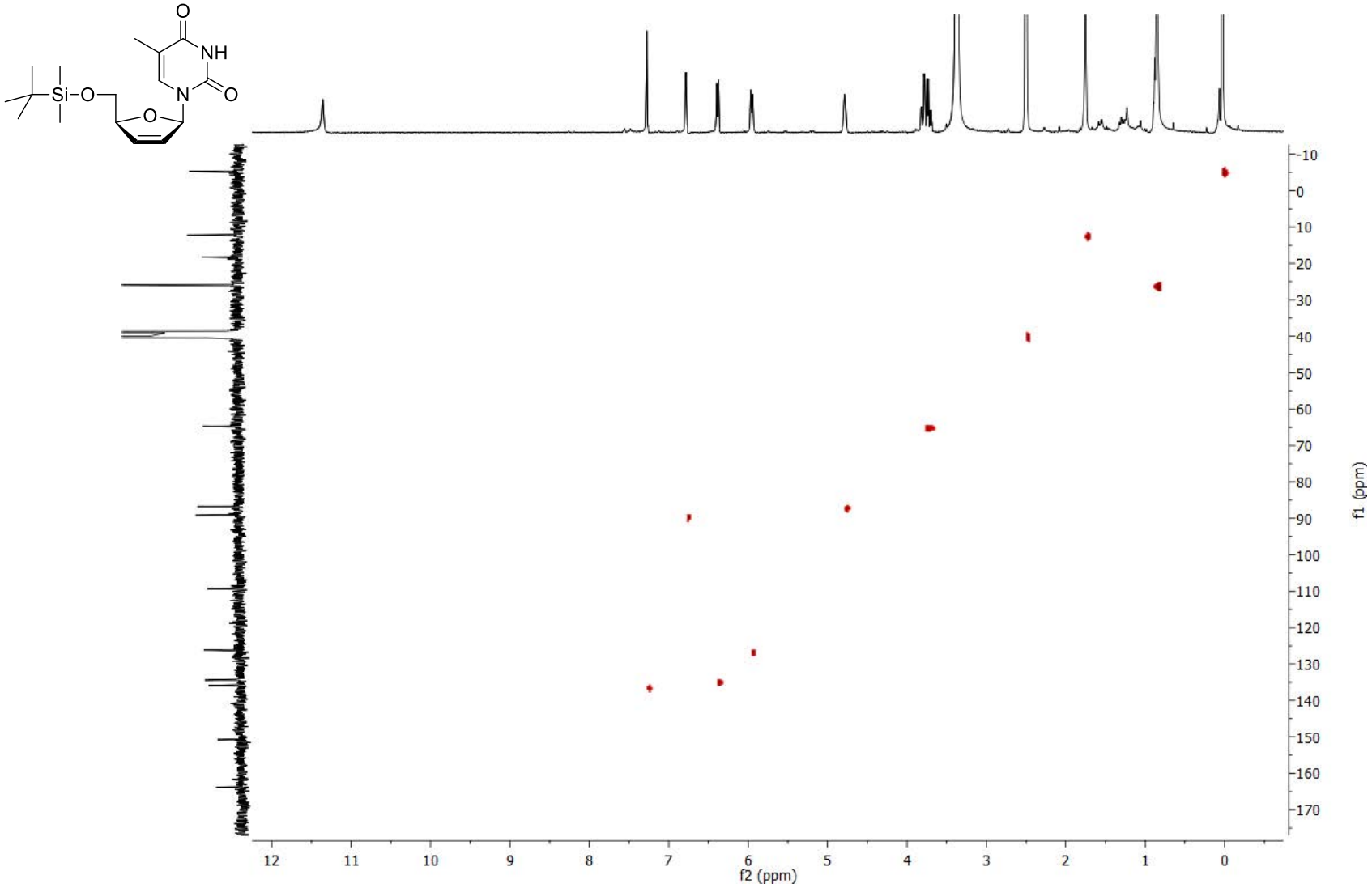
^{1}H-NMR (300.13 MHz, DMSO-d_6)



^{13}C-NMR (75.5 MHz, DMSO-d_6)

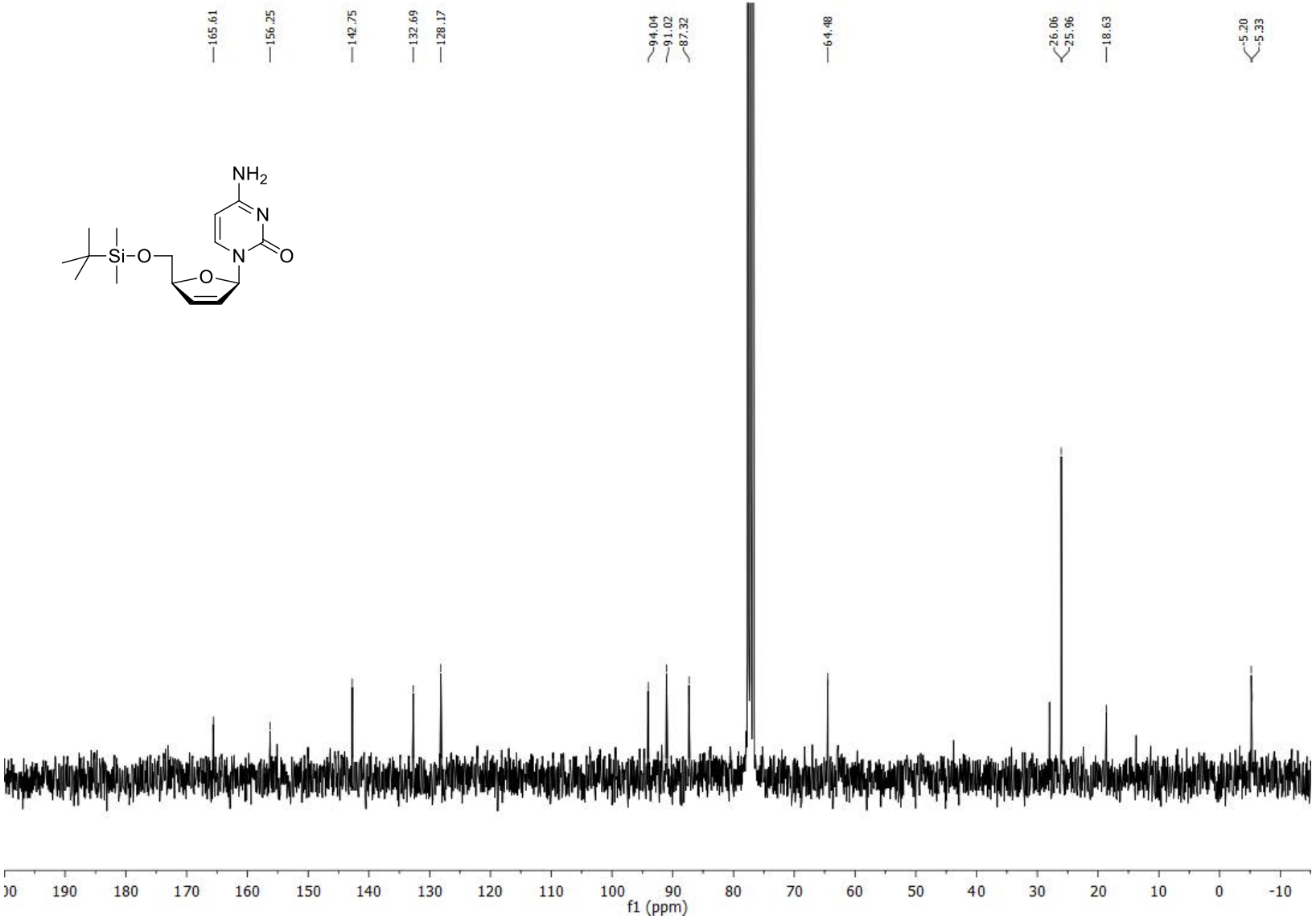


HSQC NMR (DMSO-d_6)

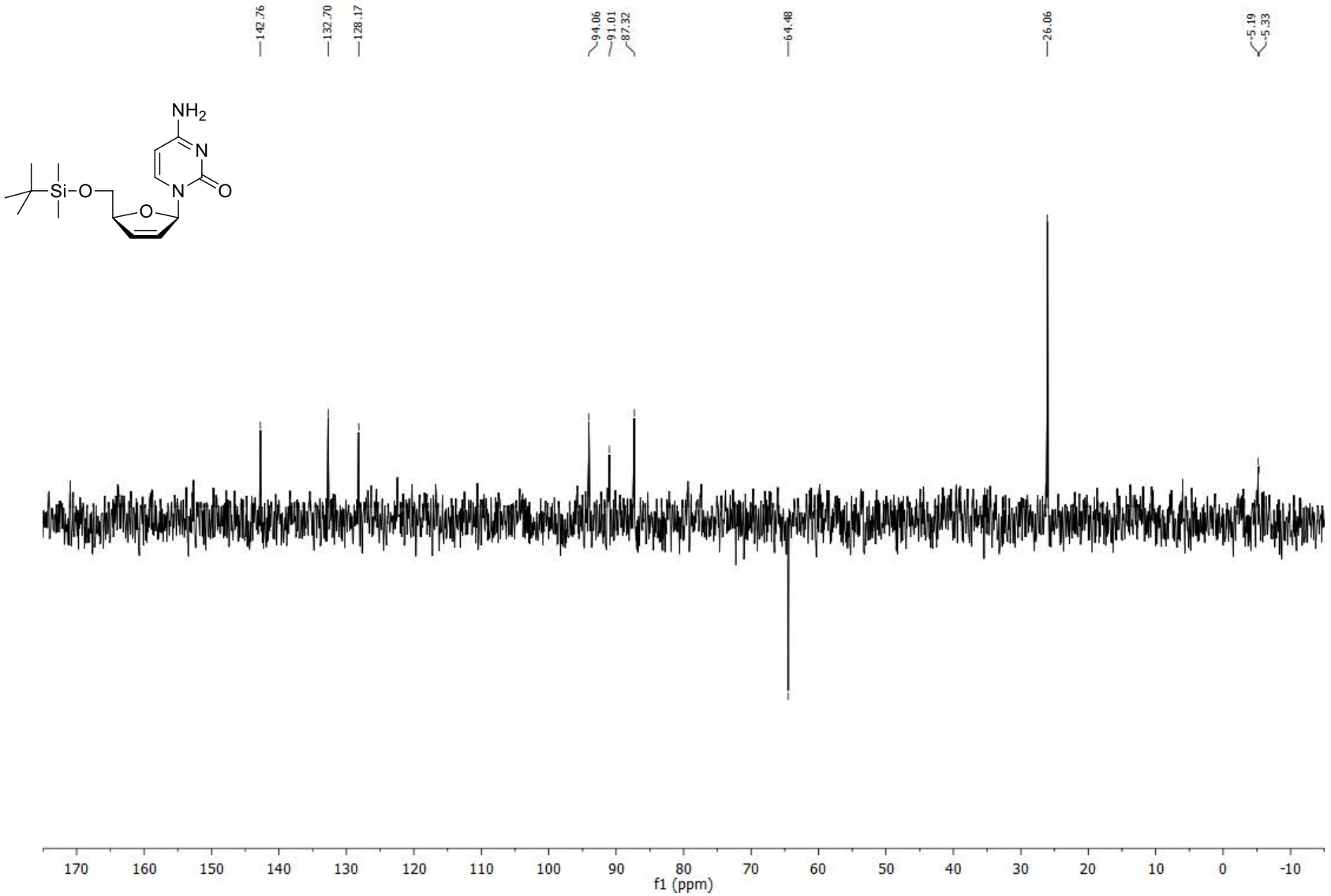


^{1}H-NMR (300.13 MHz, $CDCl_3$)

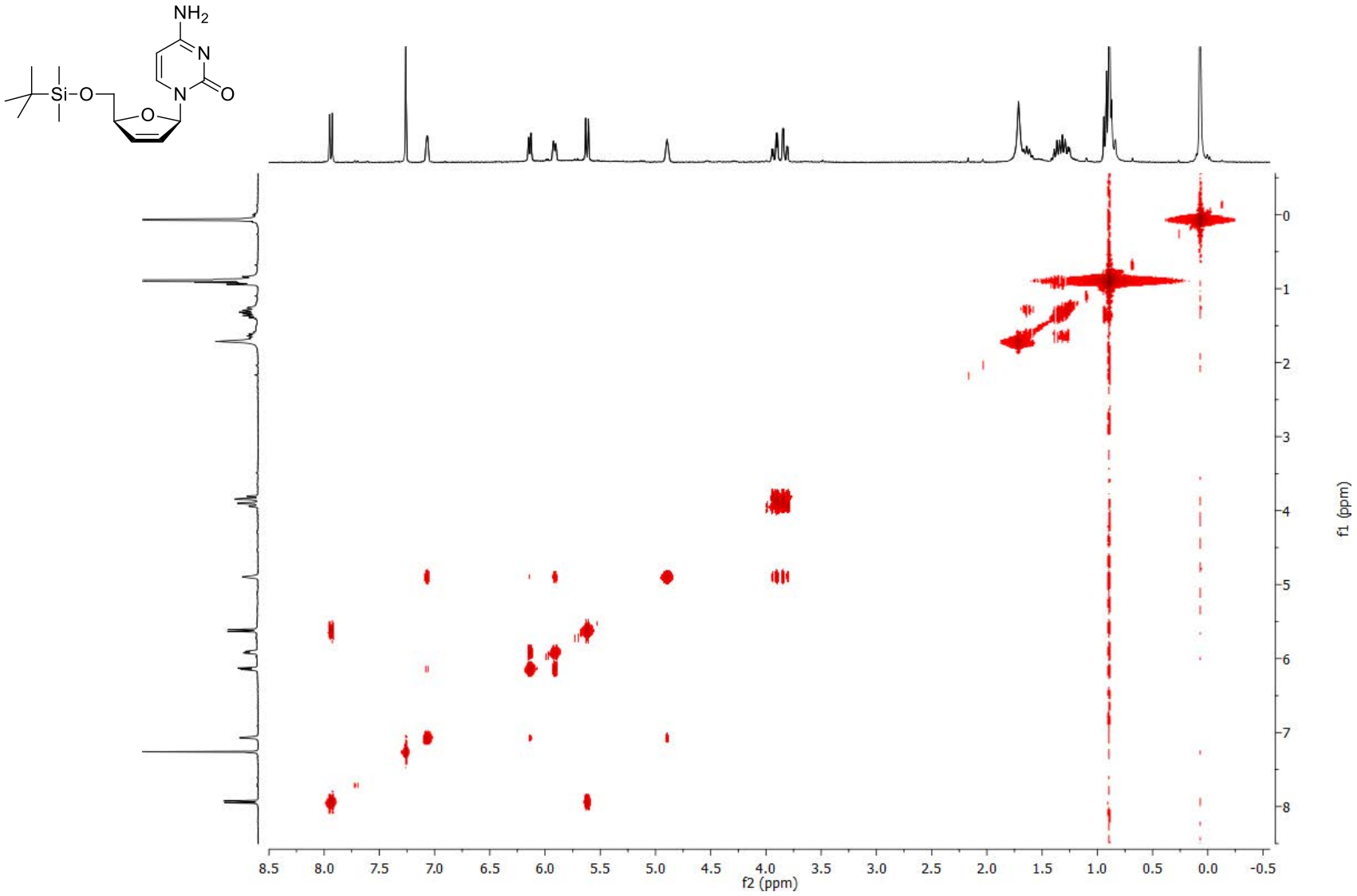
^{13}C-NMR (75.5 MHz, $CDCl_3$)



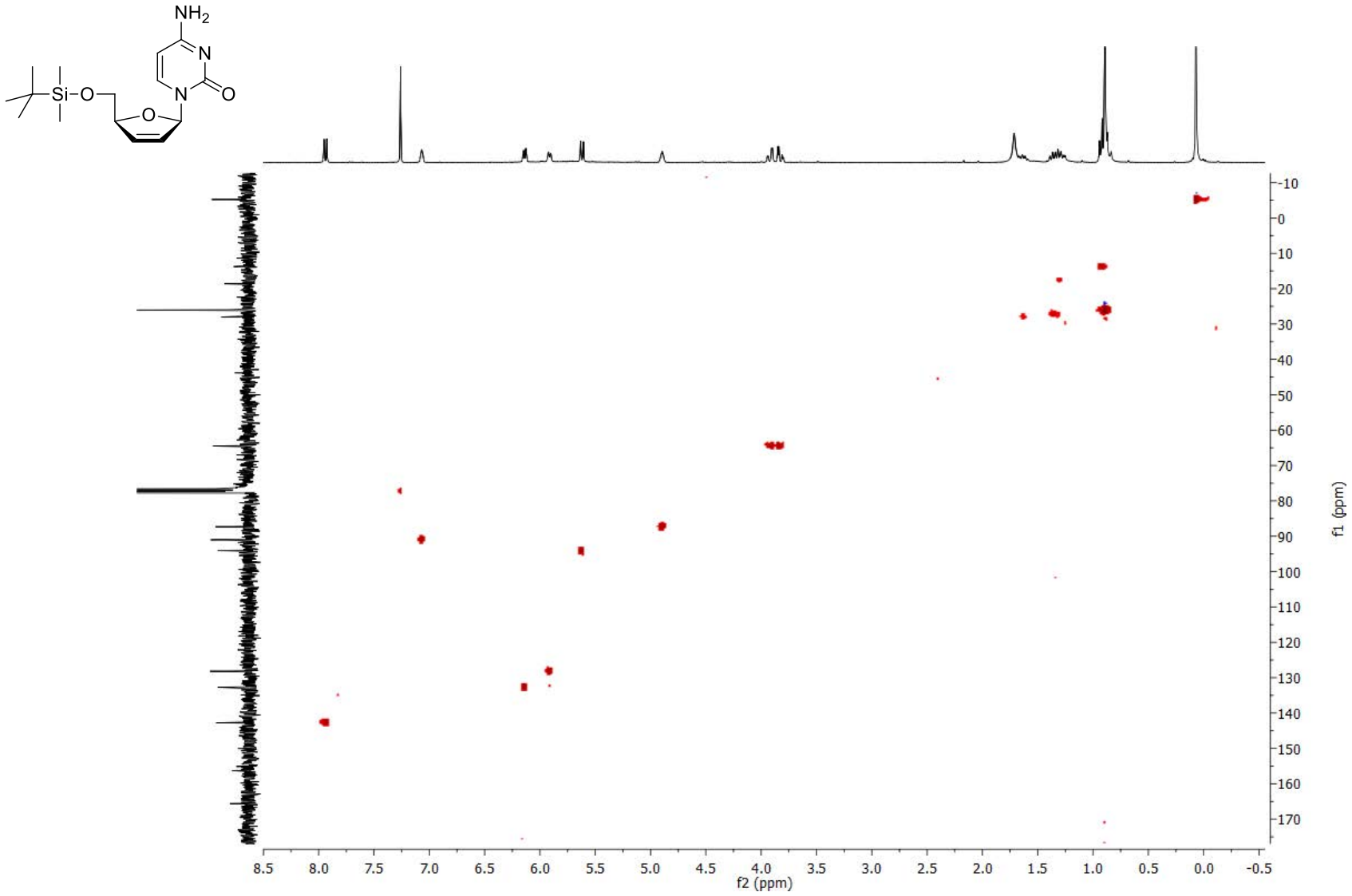
DEPT NMR (75.5 MHz, $CDCl_3$)



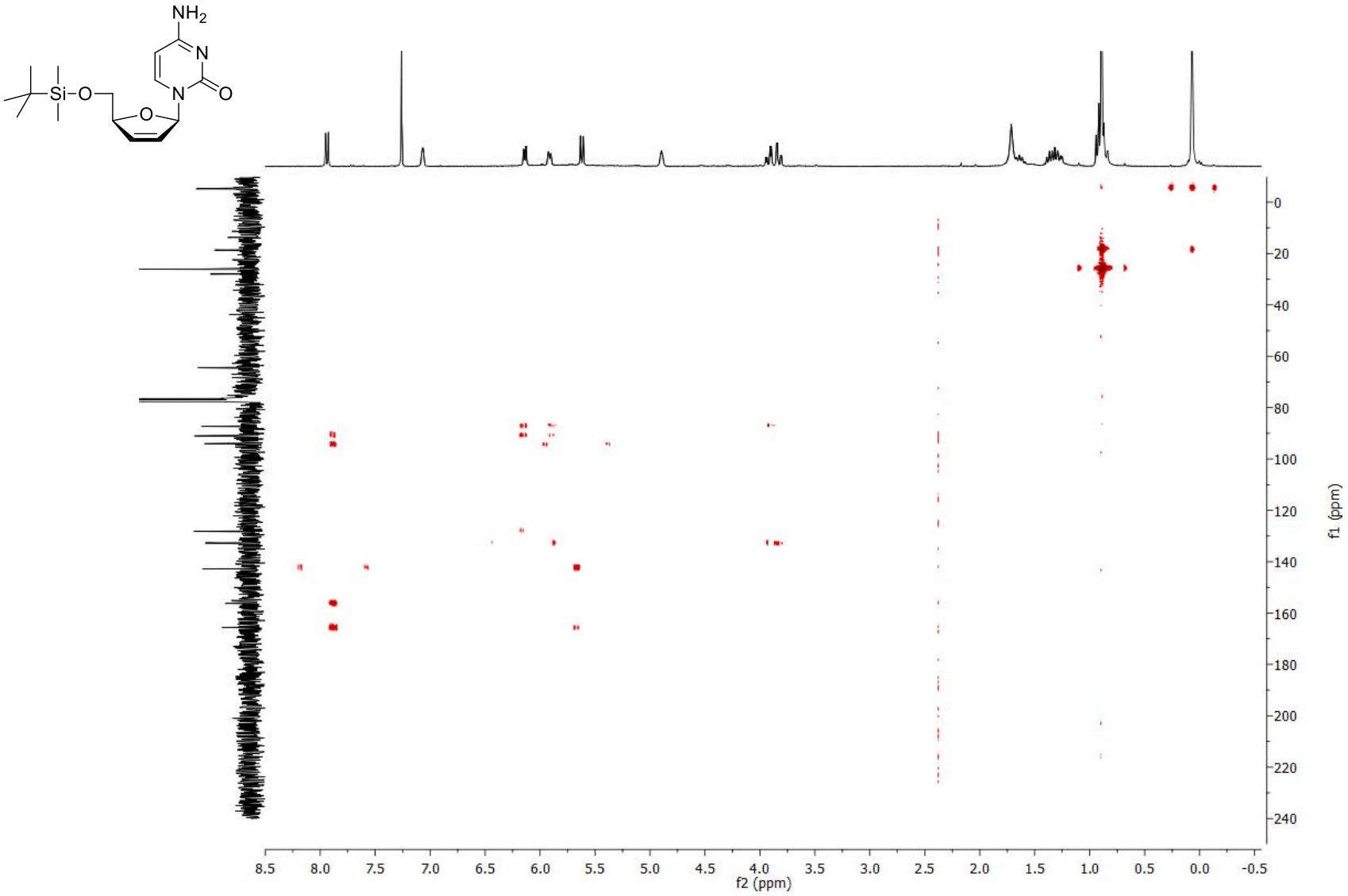
COSY NMR ($CDCl_3$)



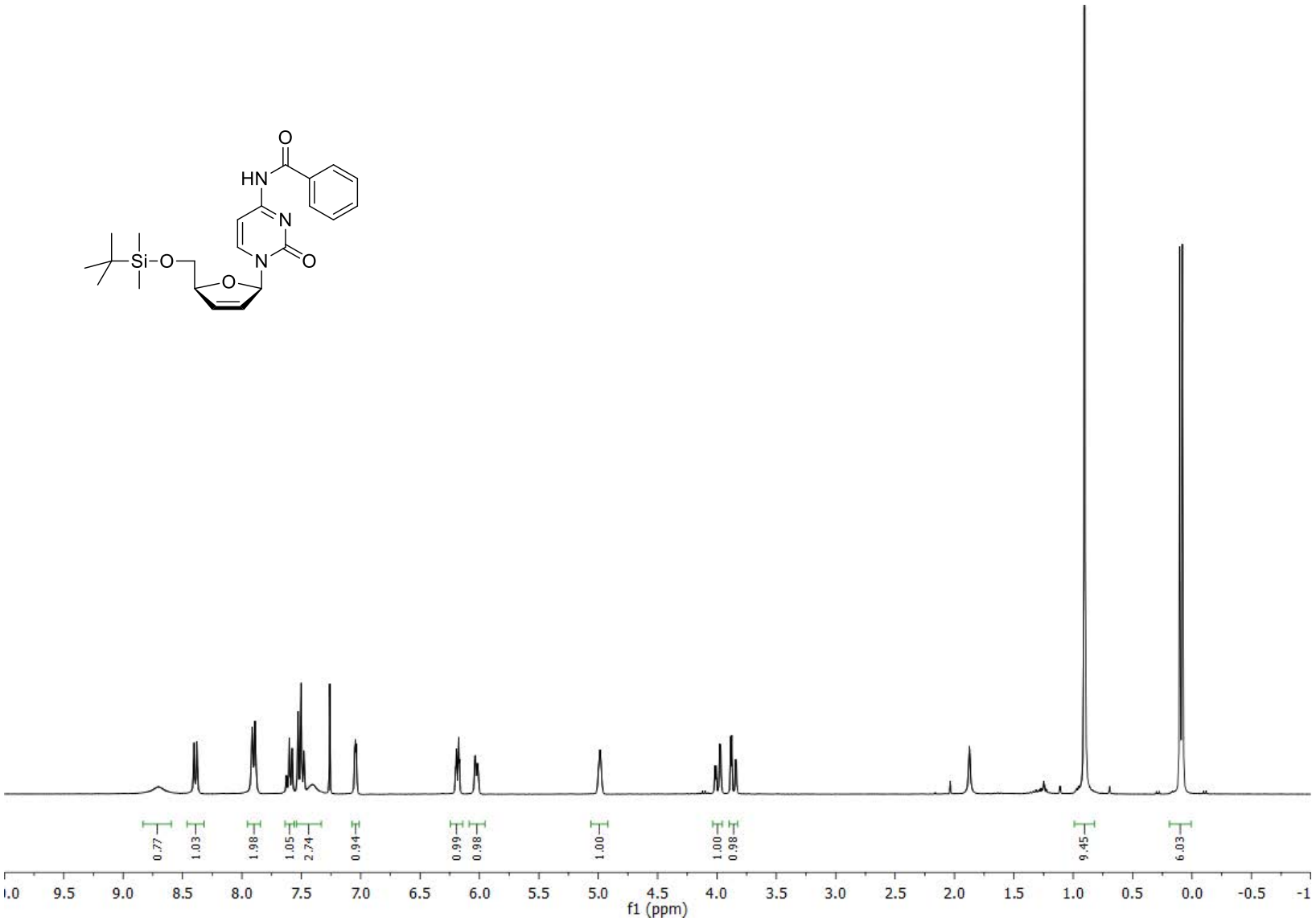
HSQC NMR ($CDCl_3$)



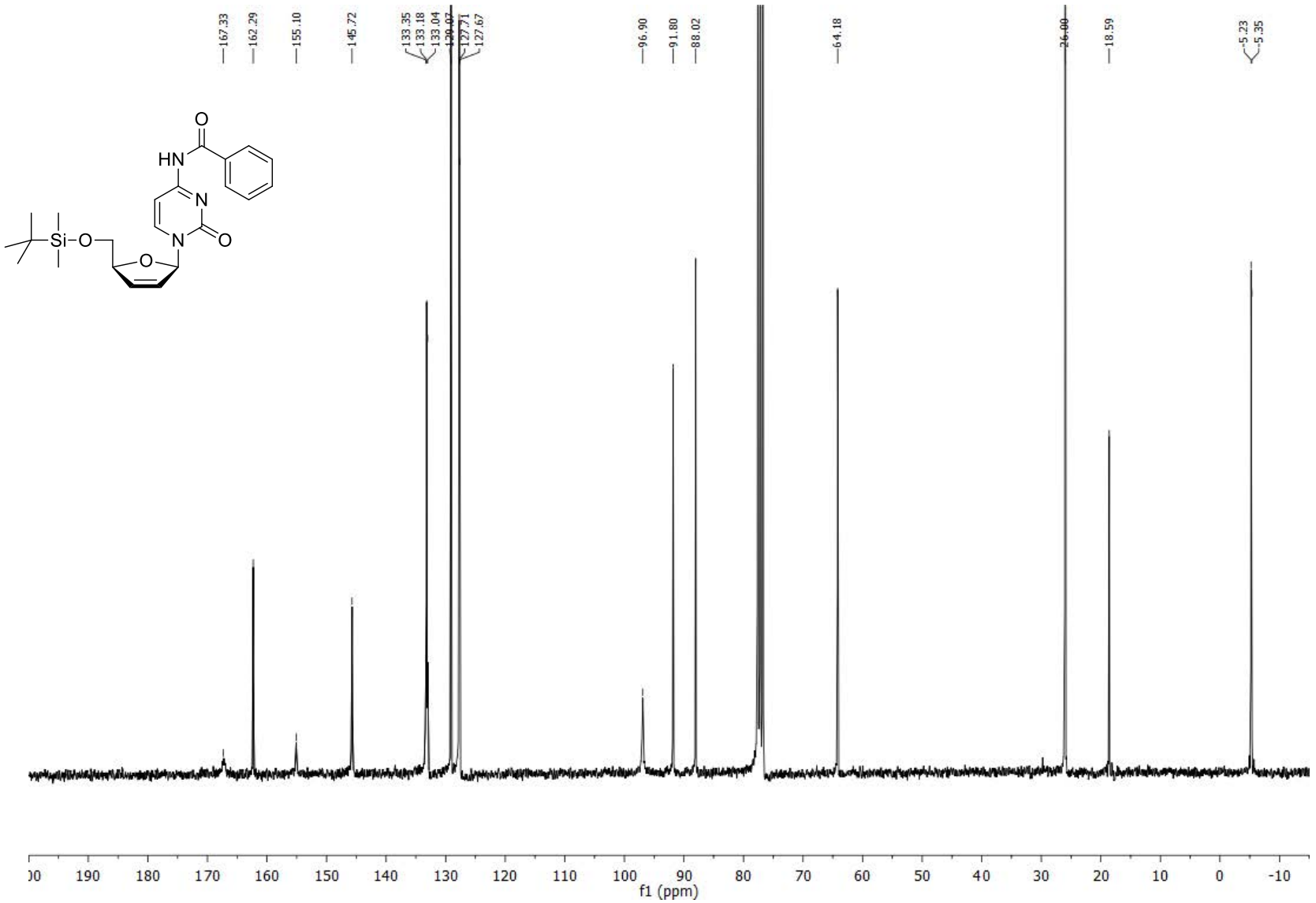
HMBC NMR ($CDCl_3$)



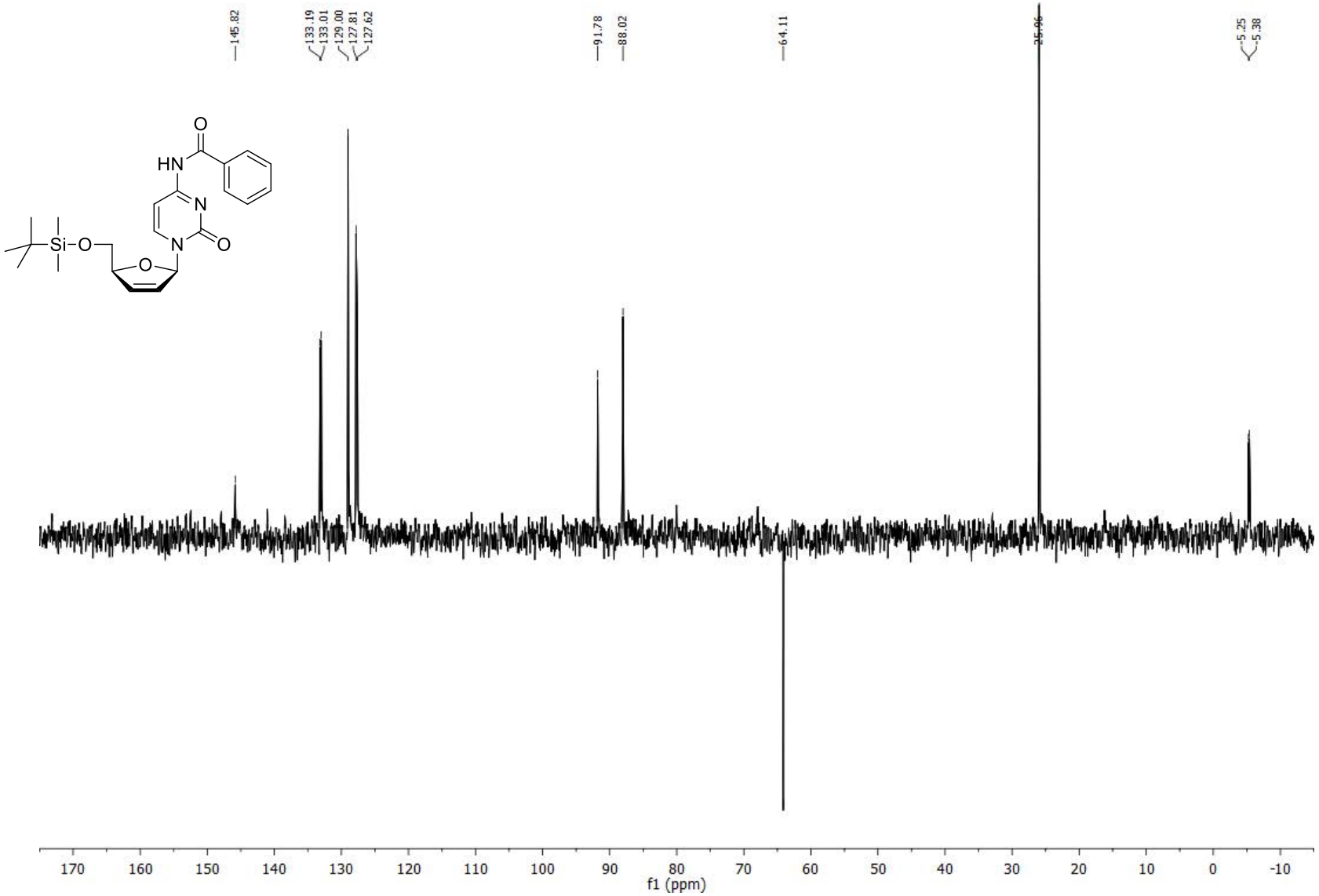
^{1}H-NMR (300.13 MHz, $CDCl_3$)



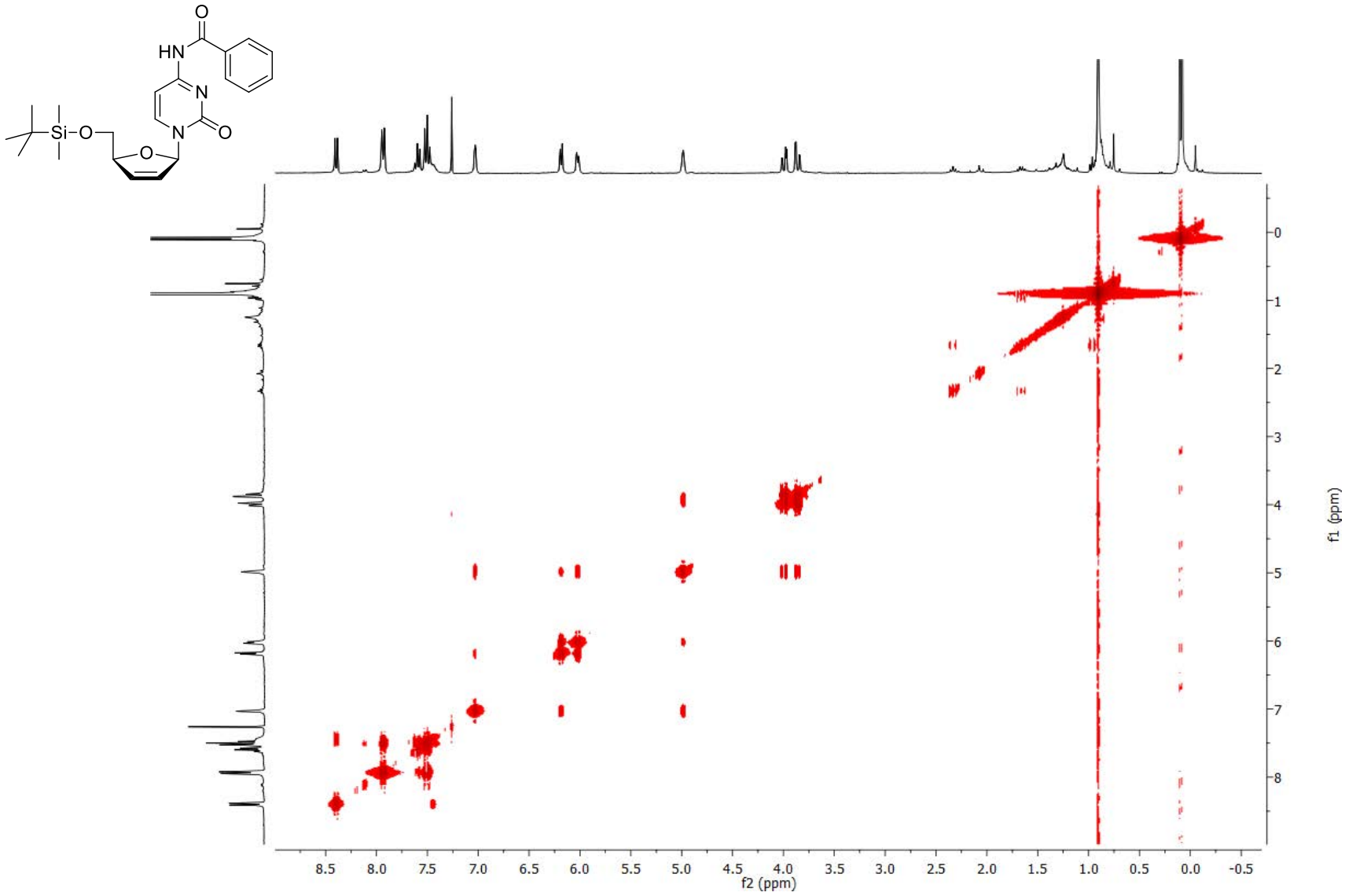
^{13}C-NMR (75.5 MHz, $CDCl_3$)



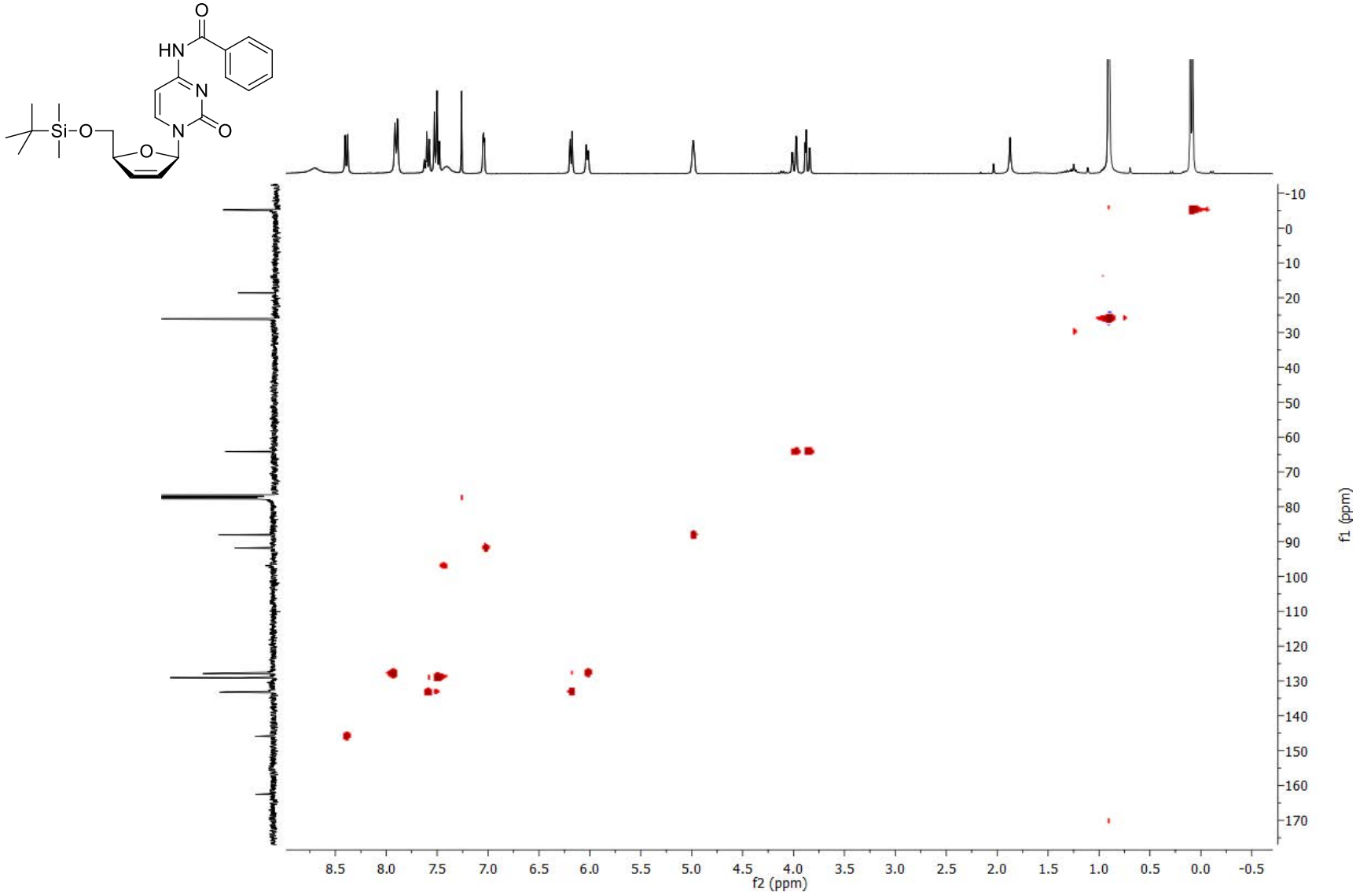
DEPT NMR (75.5 MHz, $CDCl_3$)



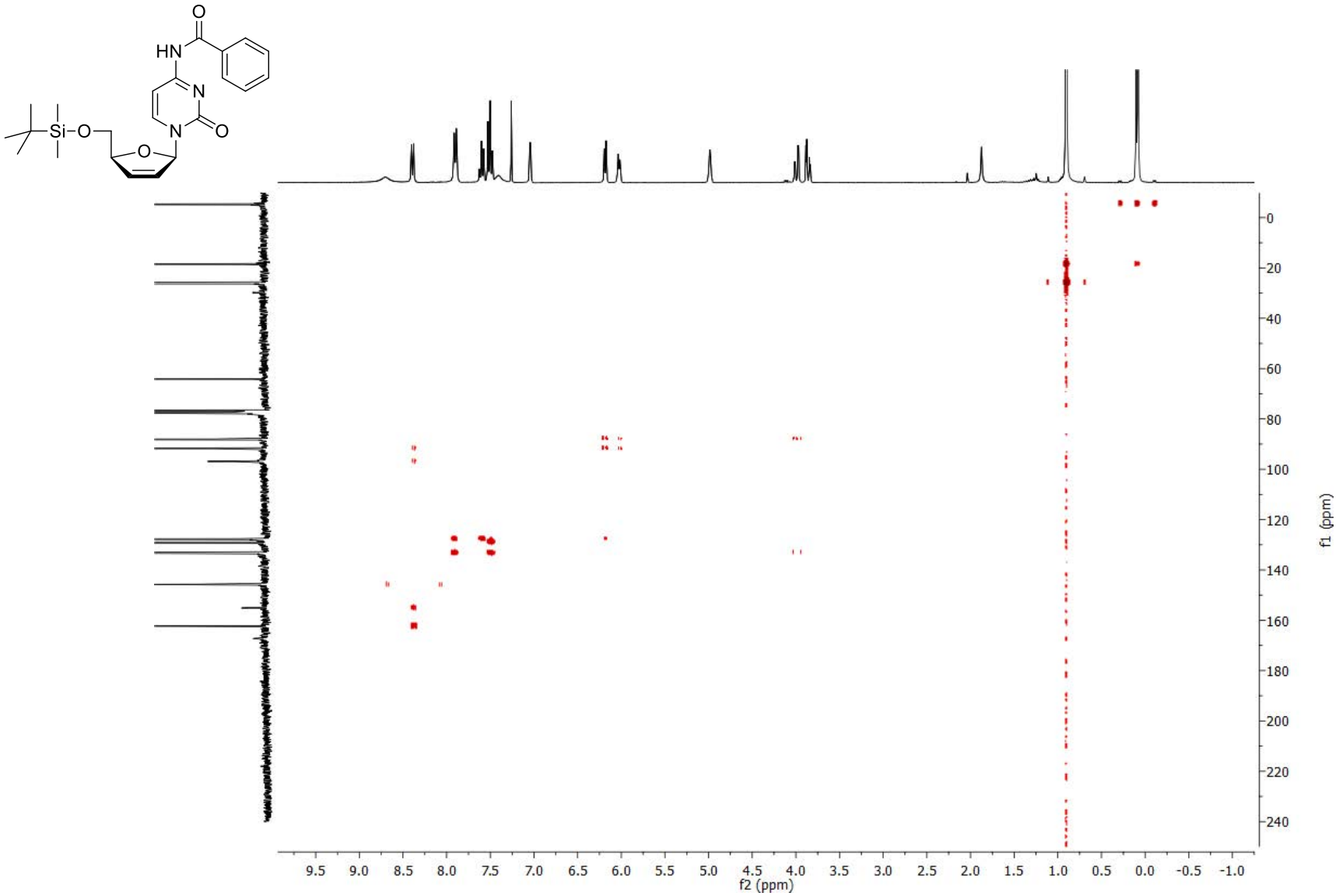
COSY NMR ($CDCl_3$)



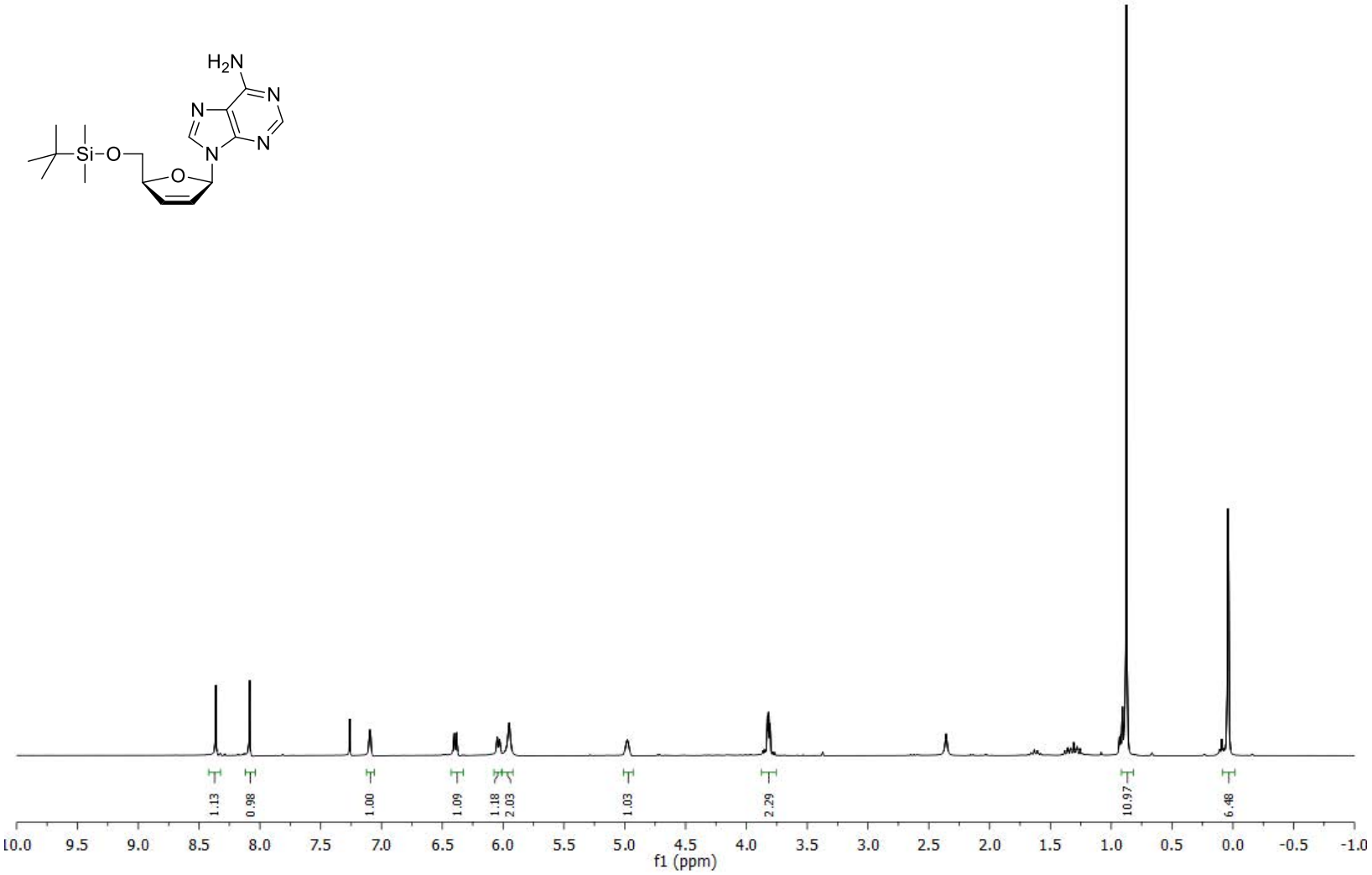
HSQC NMR ($CDCl_3$)



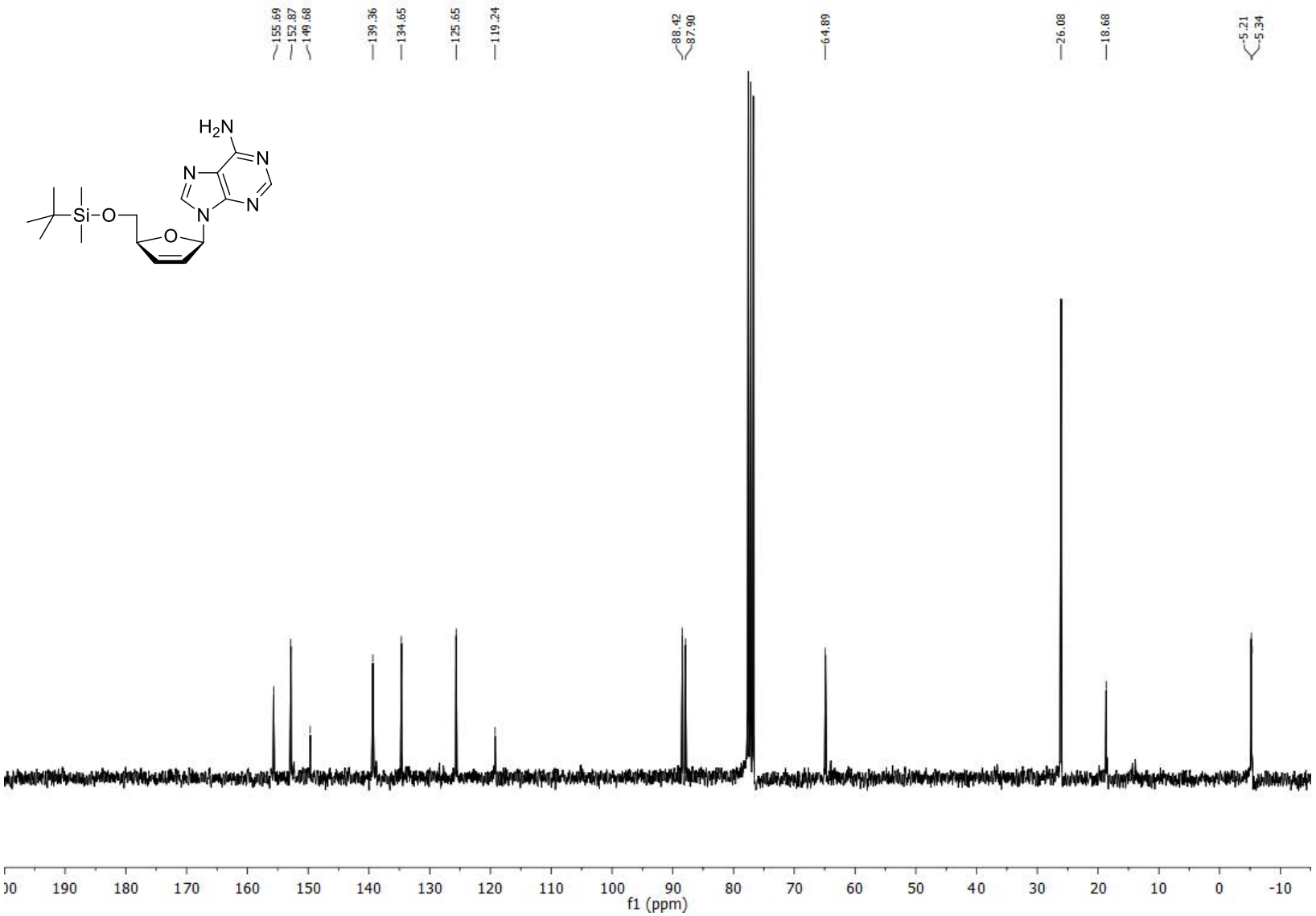
HMBC NMR ($CDCl_3$)



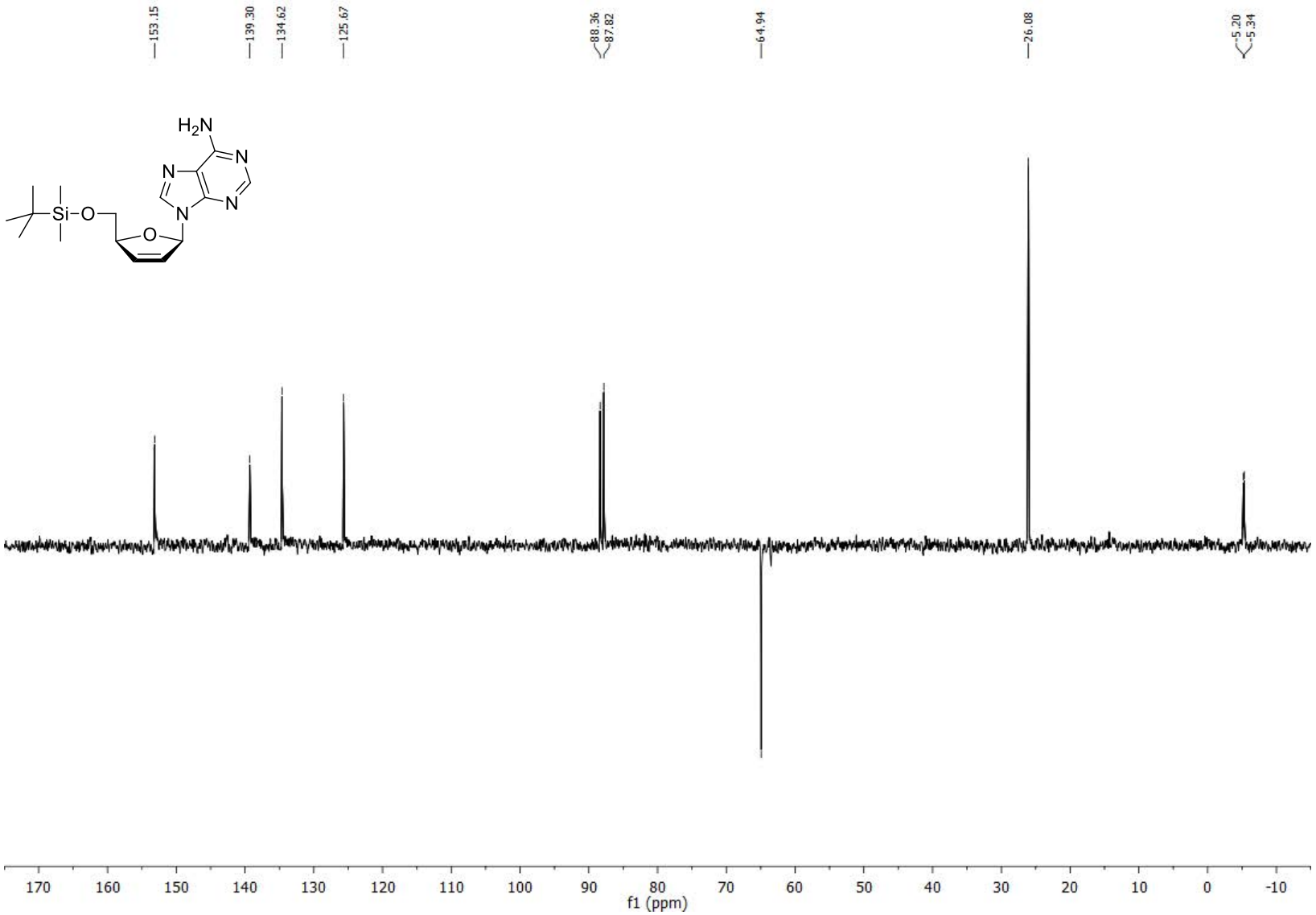
^{1}H-NMR (300.13 MHz, $CDCl_3$)



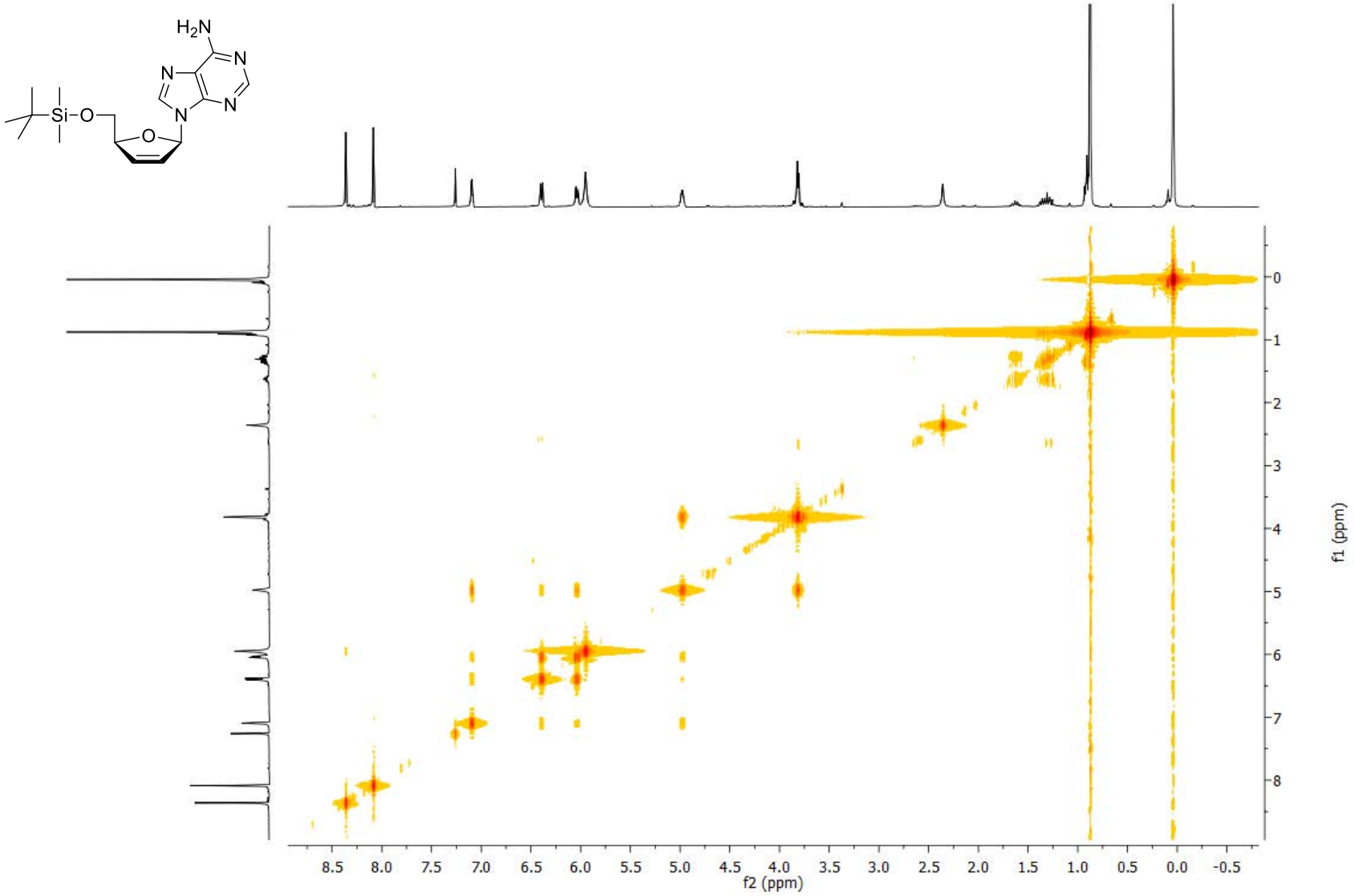
^{13}C-NMR (75.5 MHz, $CDCl_3$)



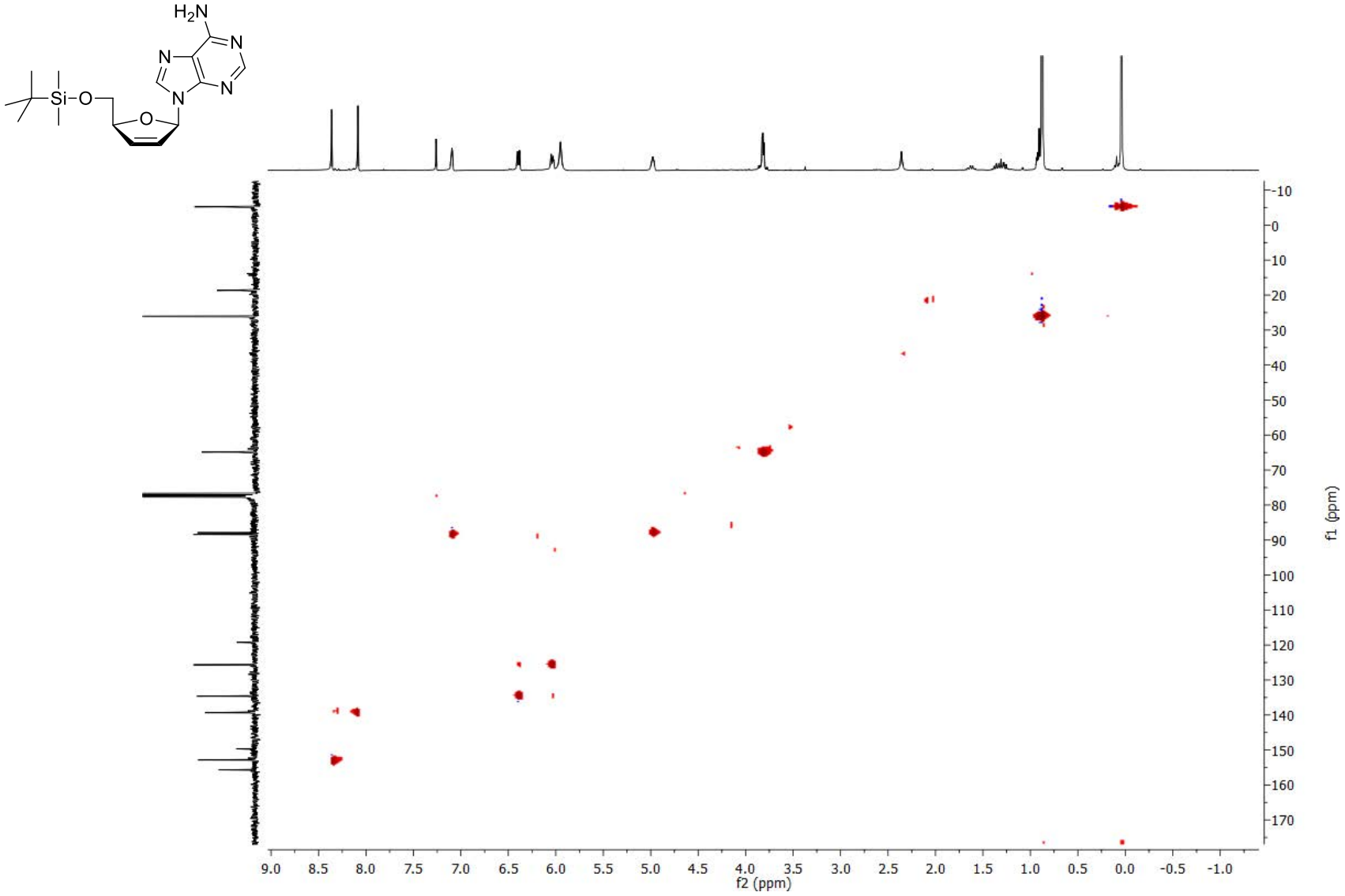
DEPT NMR (75.5 MHz, $CDCl_3$)



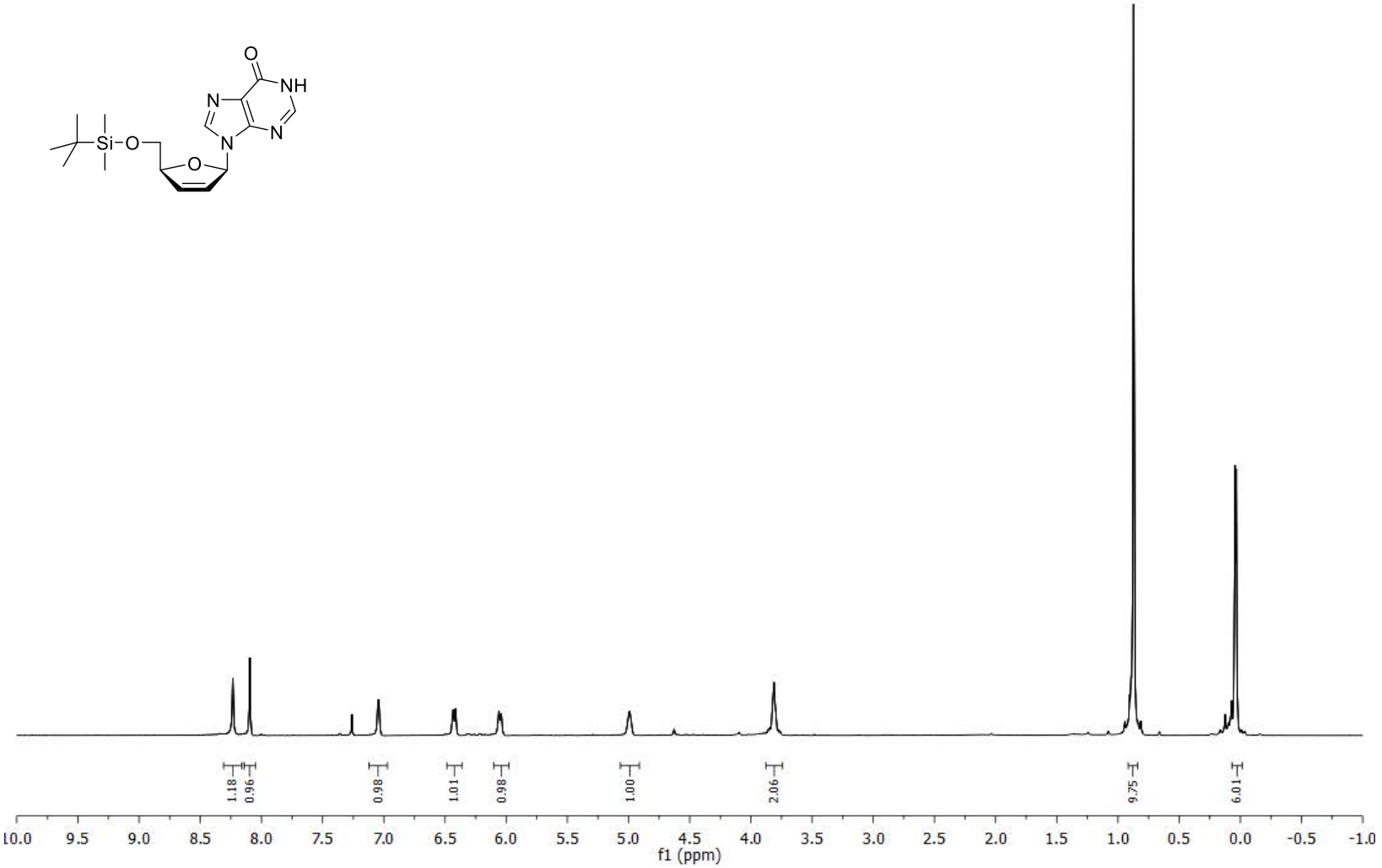
COSY NMR ($CDCl_3$)



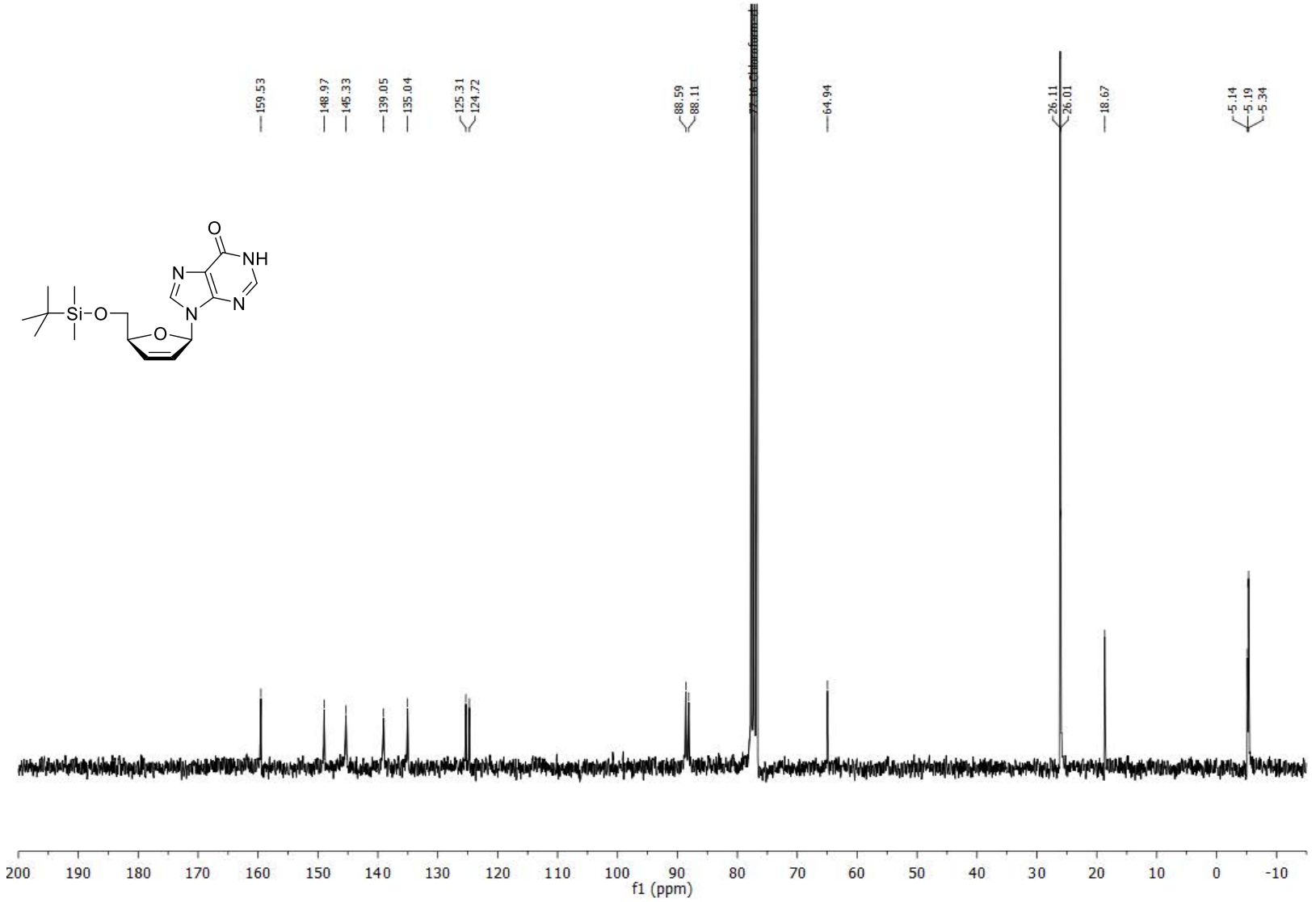
HSQC NMR ($CDCl_3$)



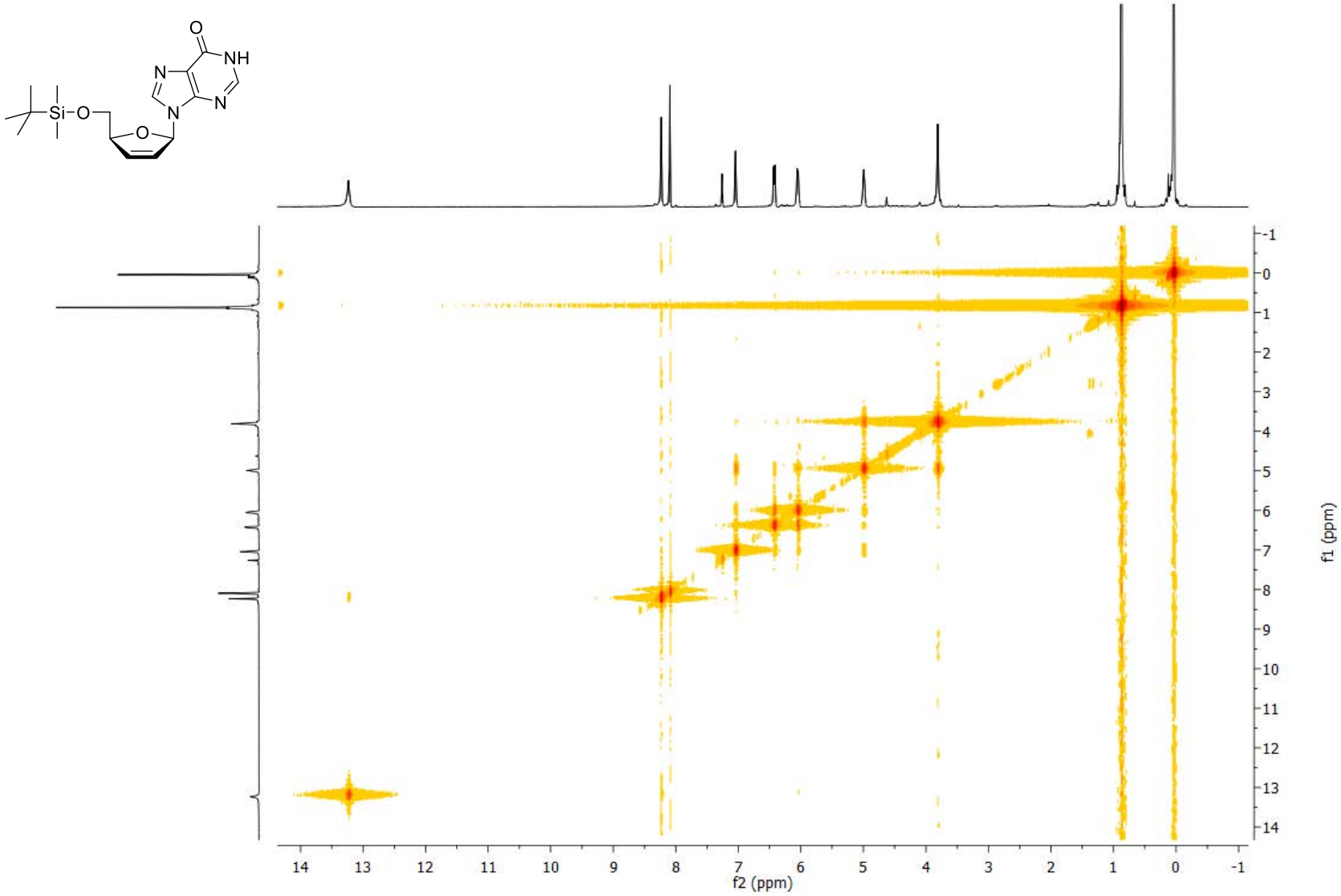
^{1}H-NMR (300.13 MHz, $CDCl_3$)



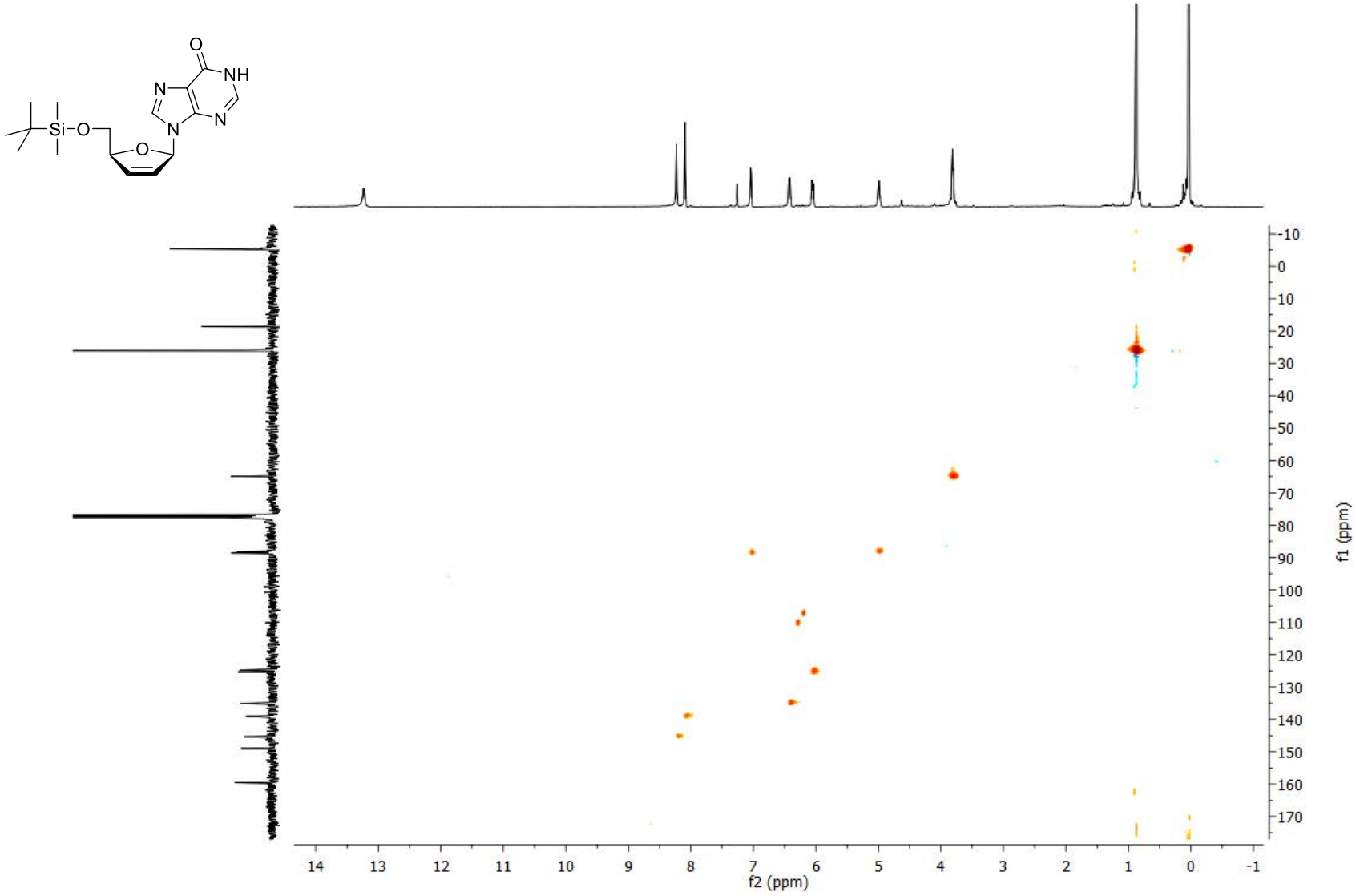
^{13}C-NMR (75.5 MHz, $CDCl_3$)



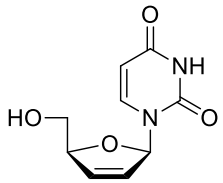
COSY NMR ($CDCl_3$)

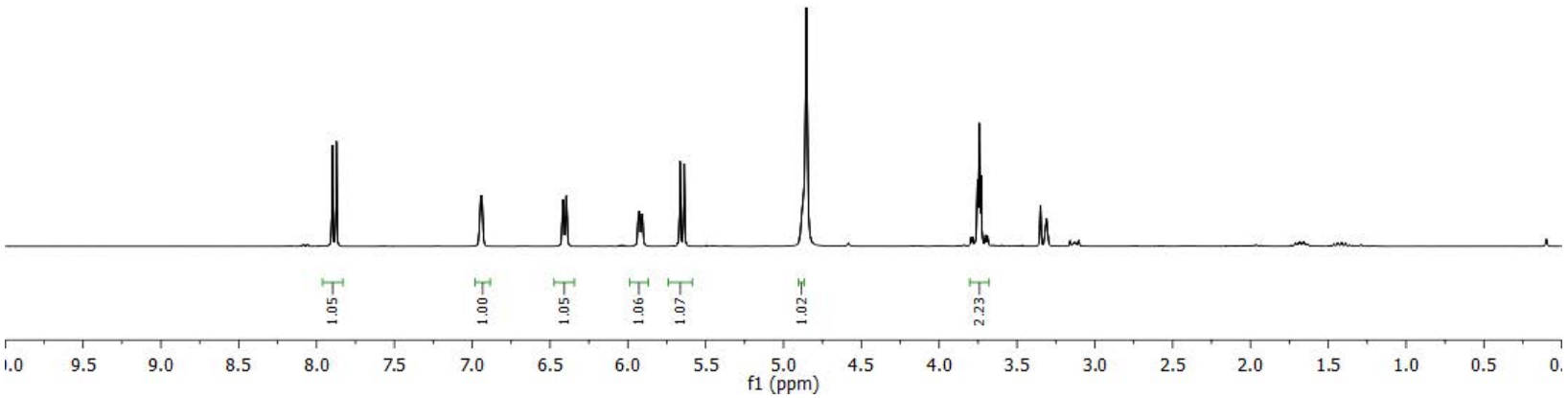


HSQC NMR ($CDCl_3$)

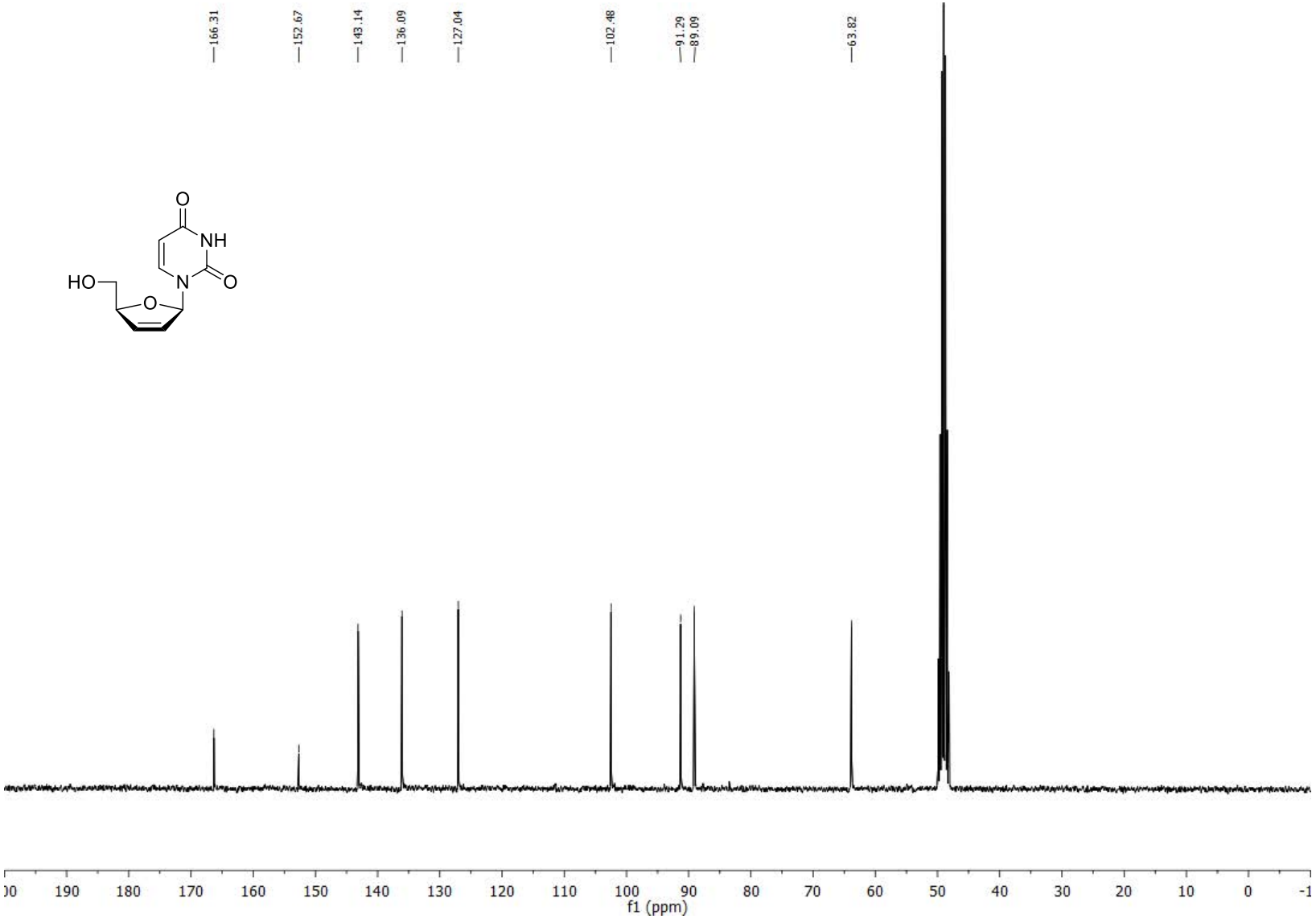


^{1}H-NMR (300.13 MHz, MeOH-d_4)

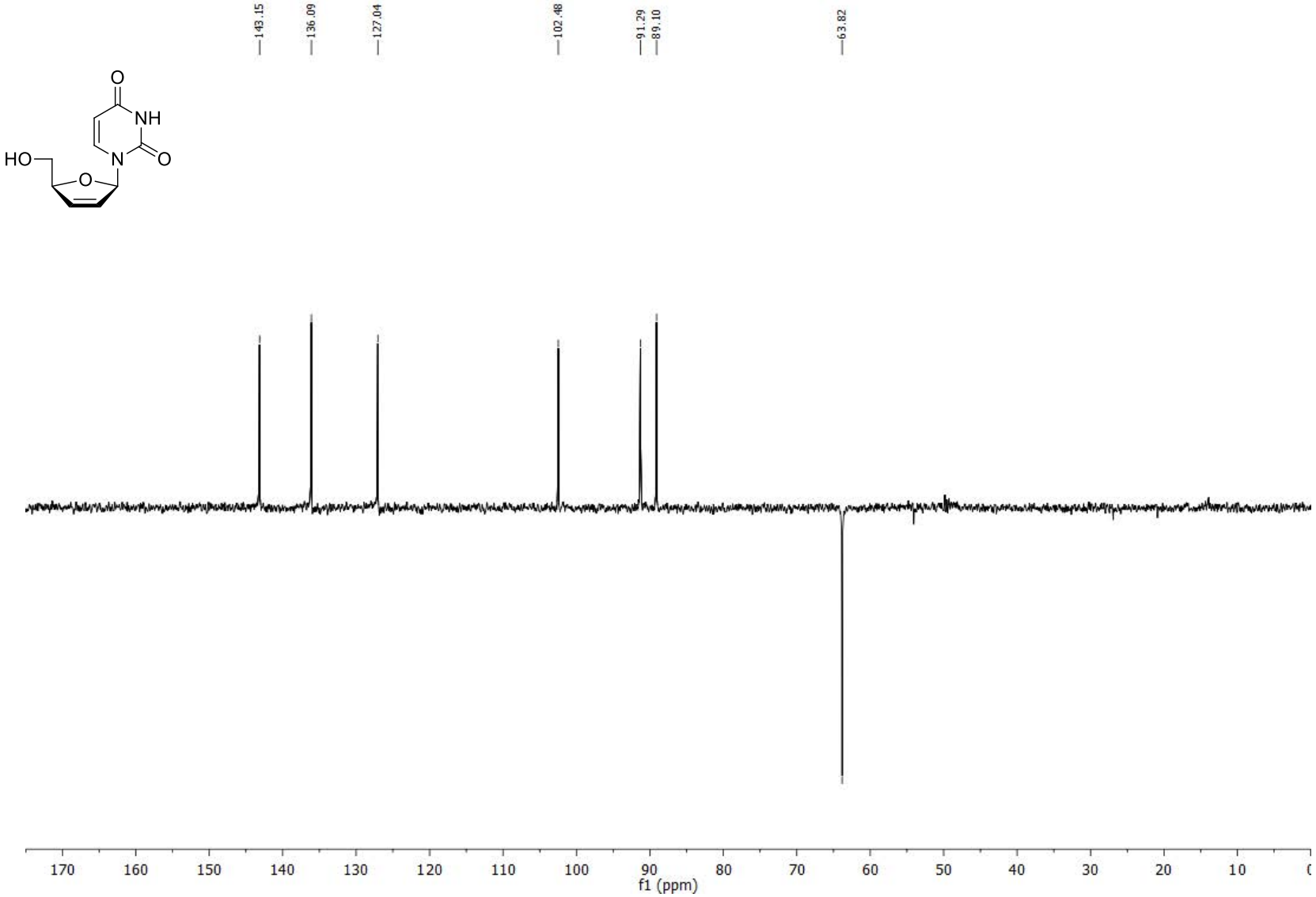




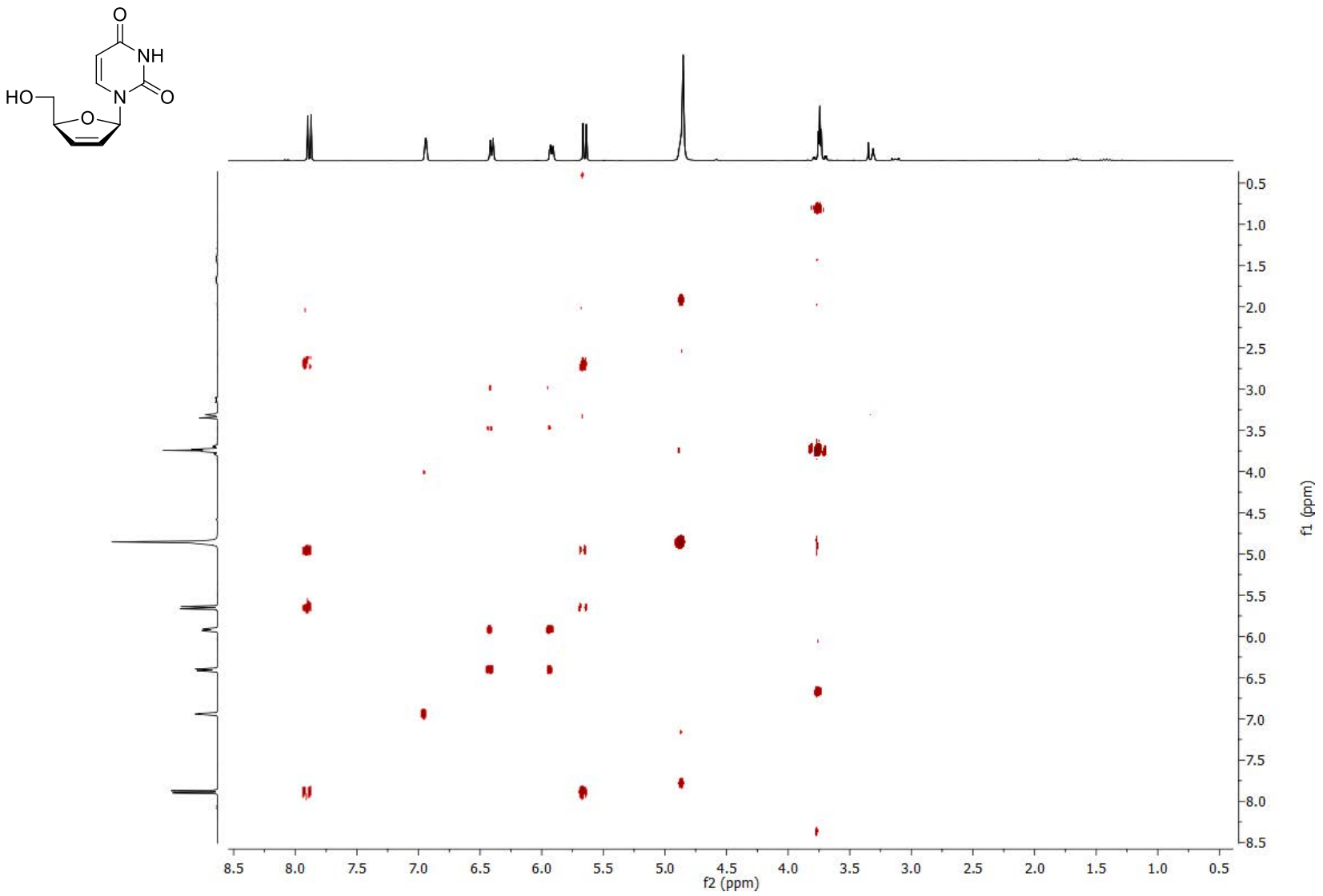
^{13}C-NMR (75.5 MHz, MeOH-d_4)



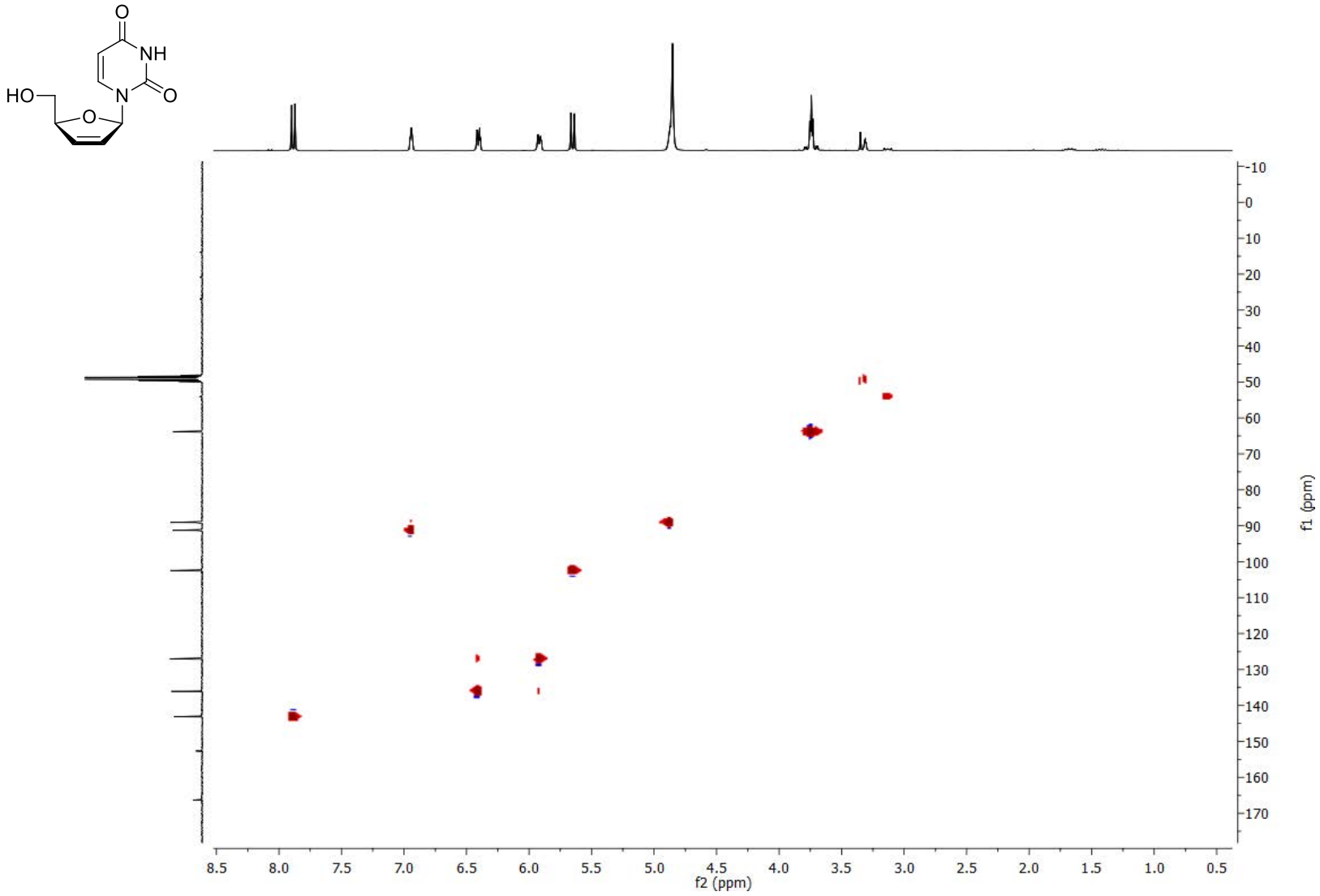
DEPT NMR (75.5 MHz, MeOH-d_4)



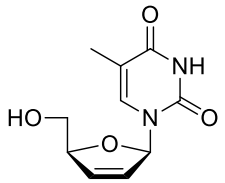
COSY NMR (MeOH-d_4)

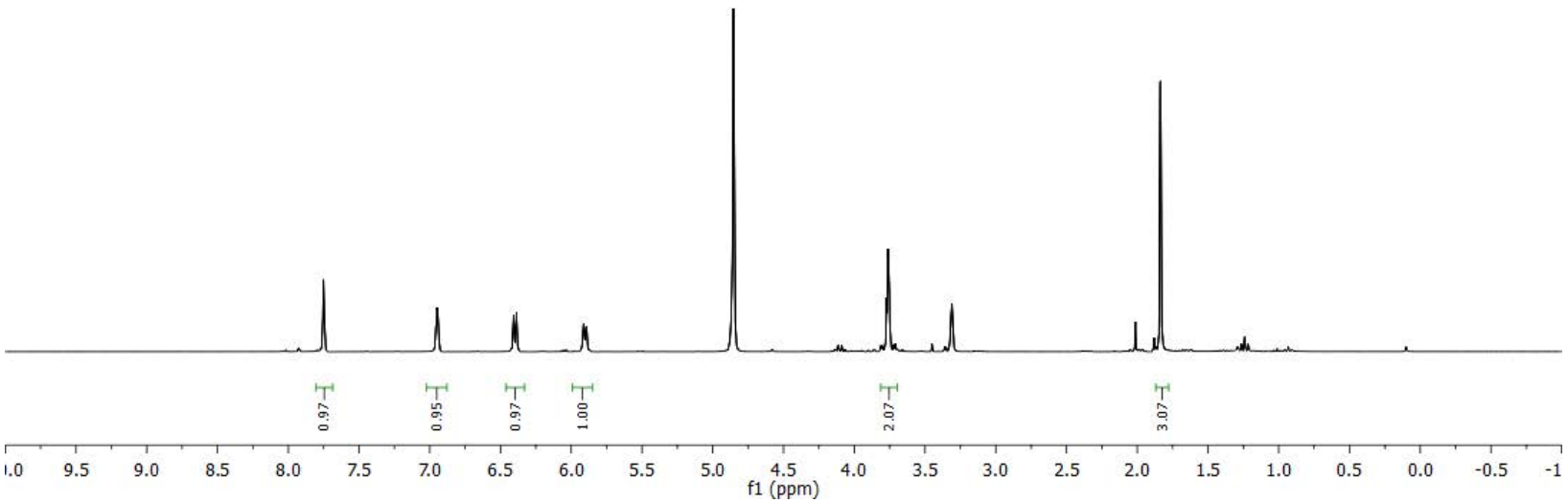


HSQC NMR (MeOH-d_4)

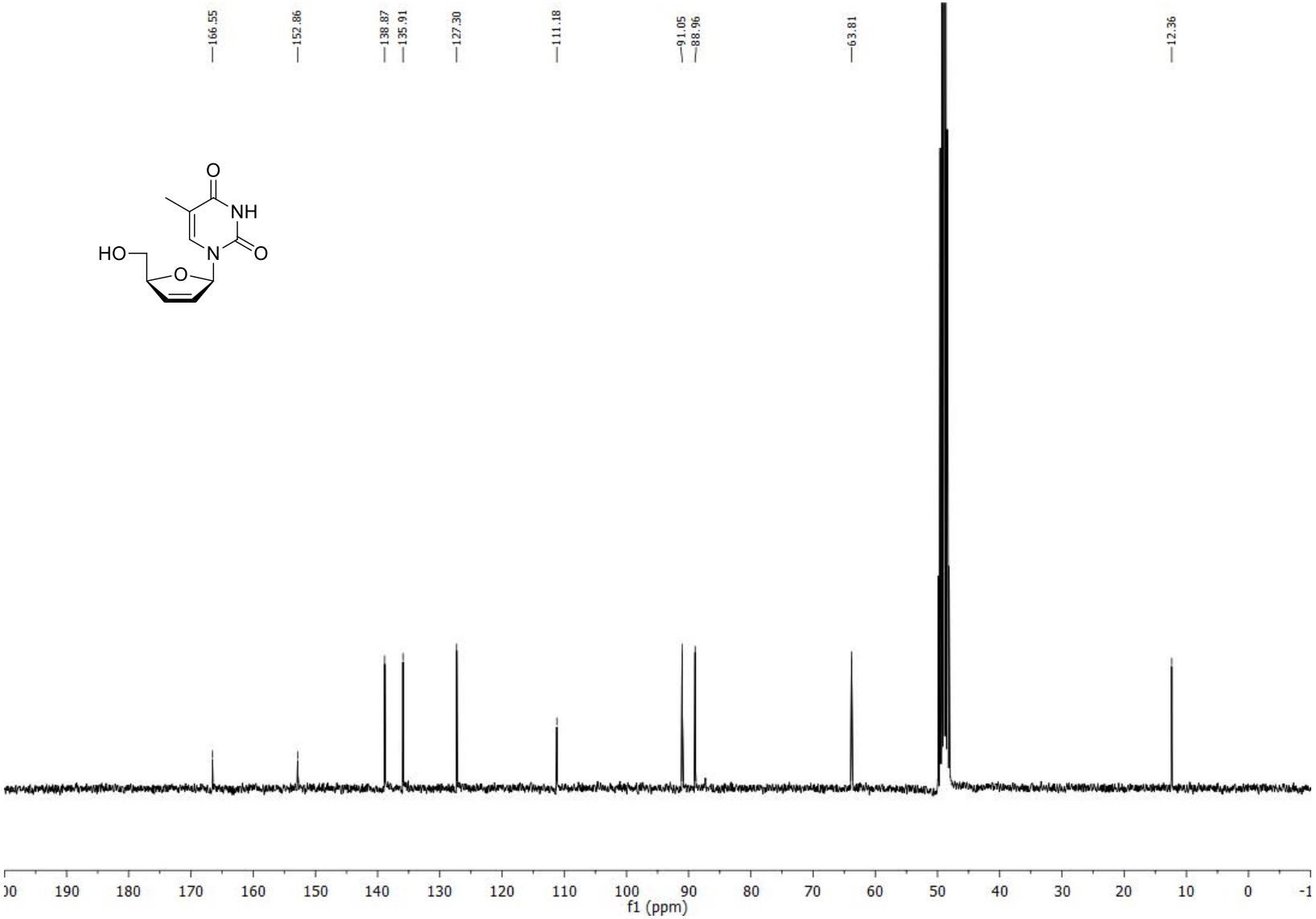


^{1}H-NMR (300.13 MHz, MeOH-d_4)

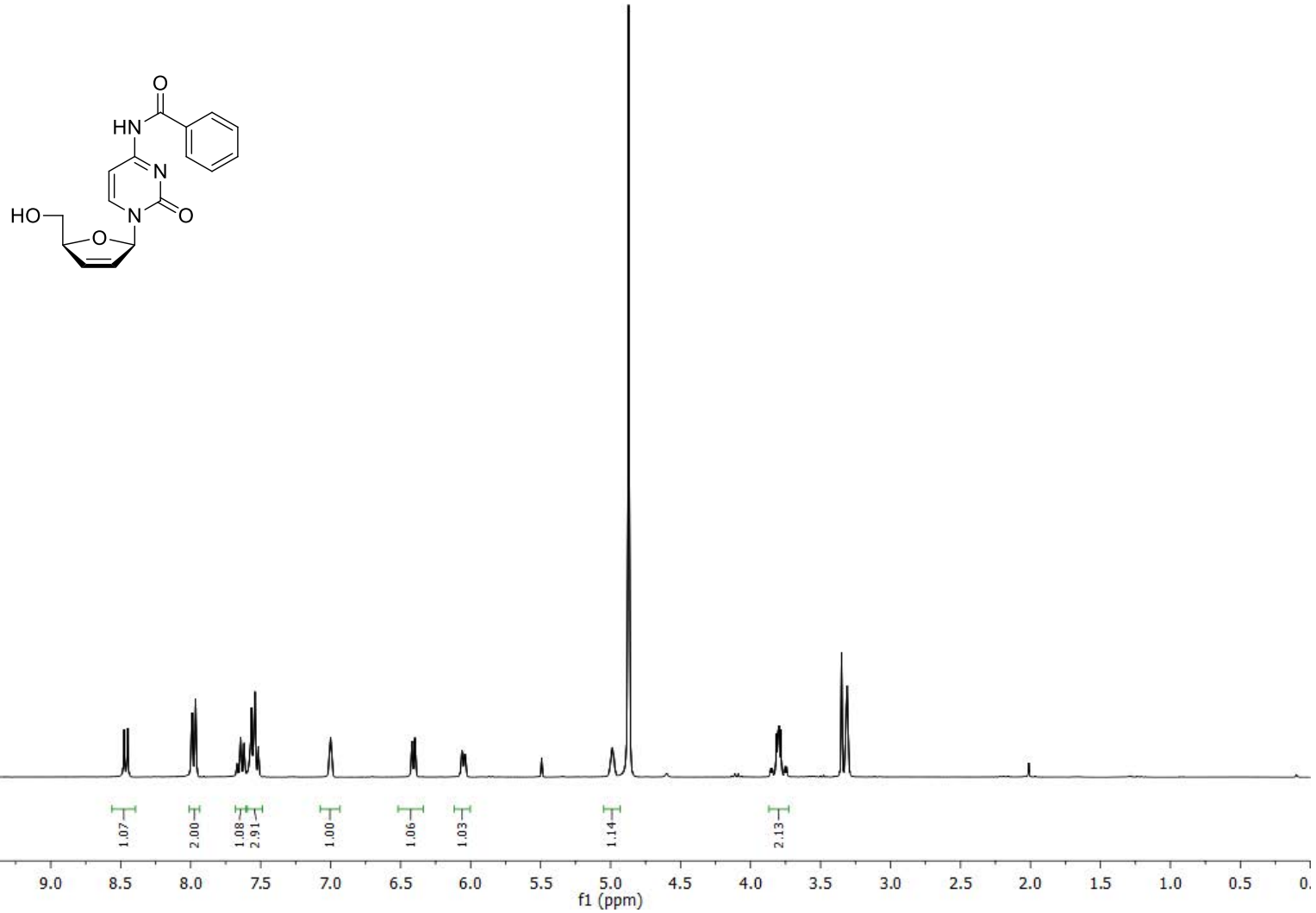




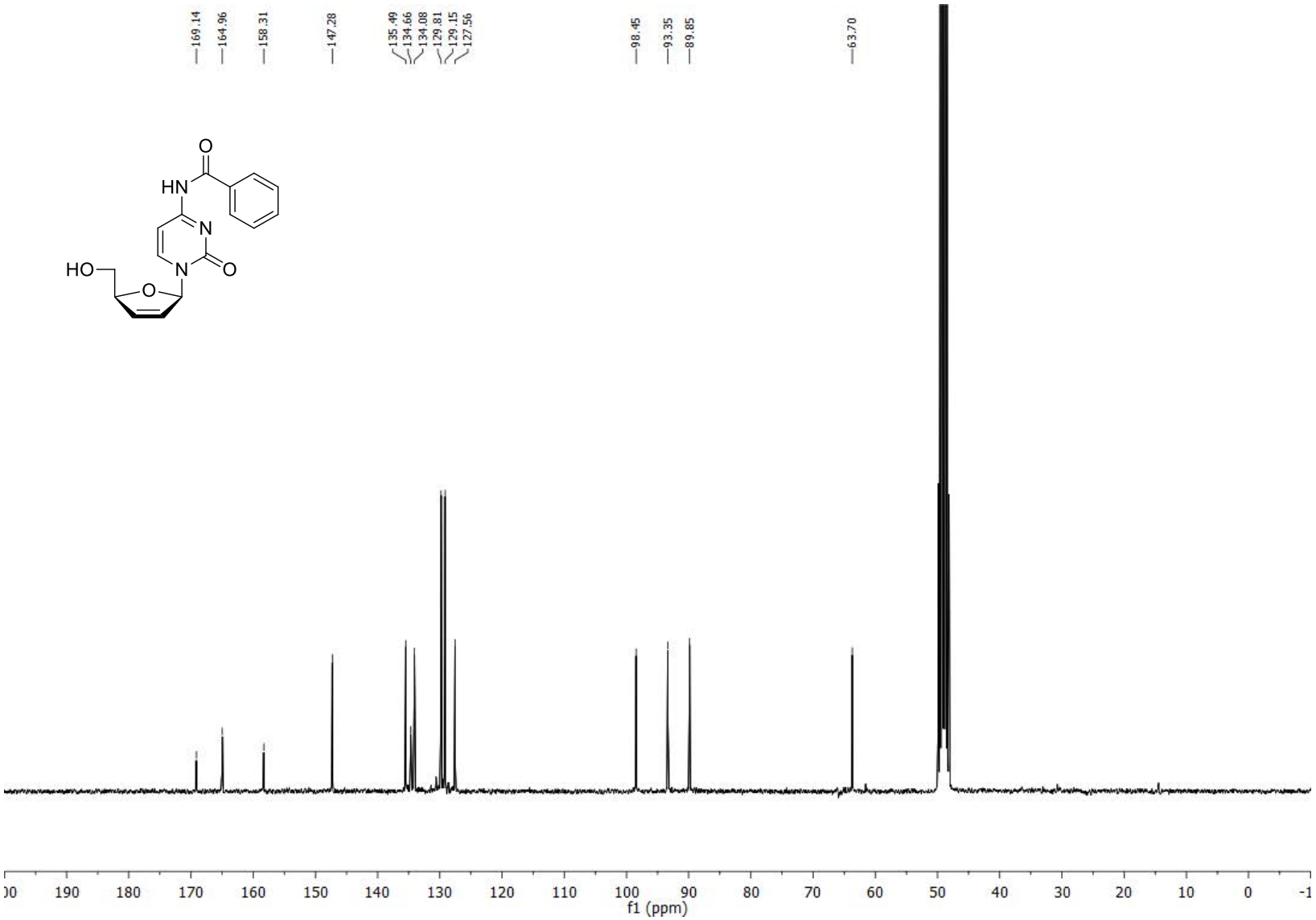
^{13}C-NMR (75.5 MHz, MeOH-d_4)



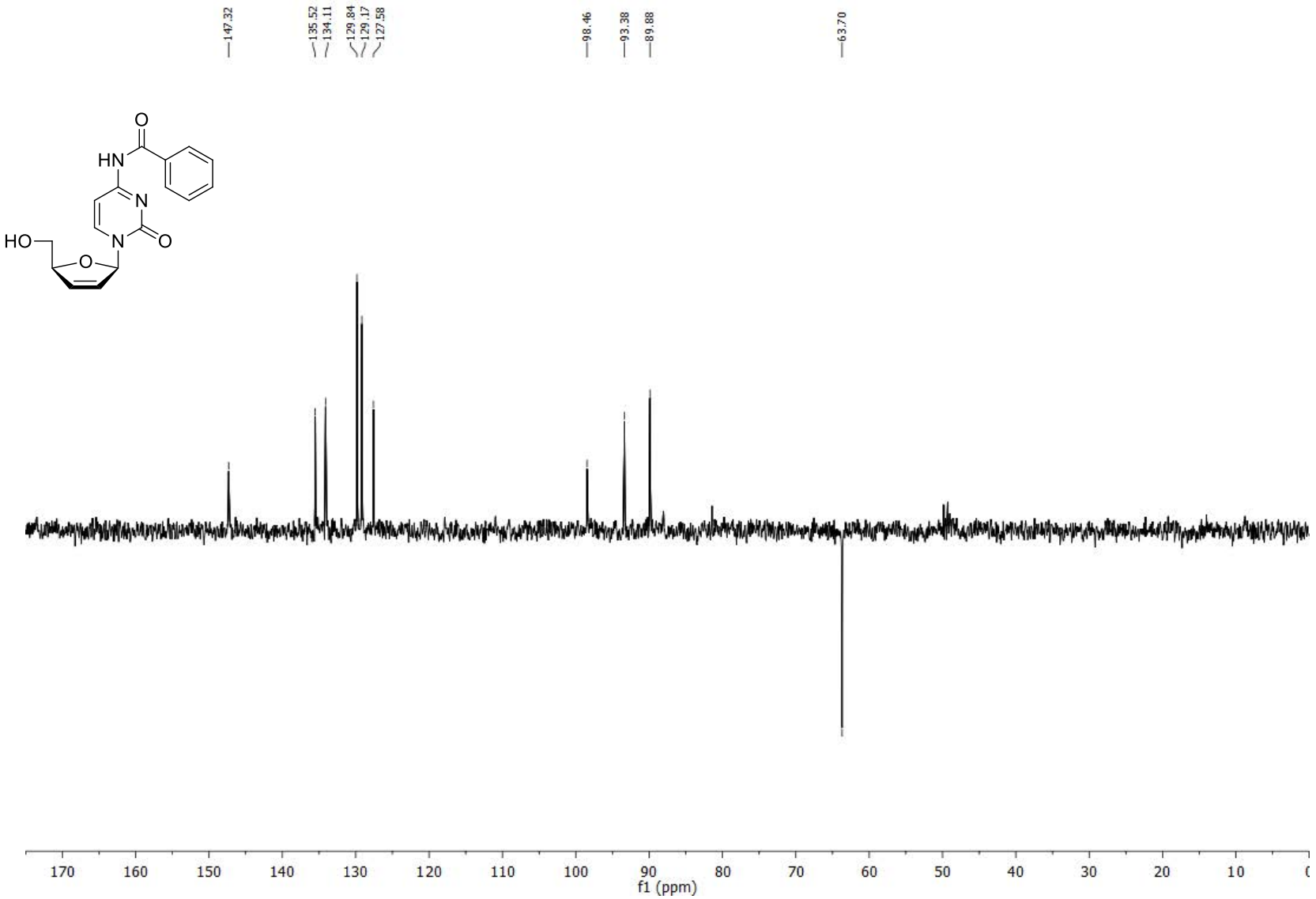
^{1}H-NMR (300.13 MHz, MeOH-d_4)



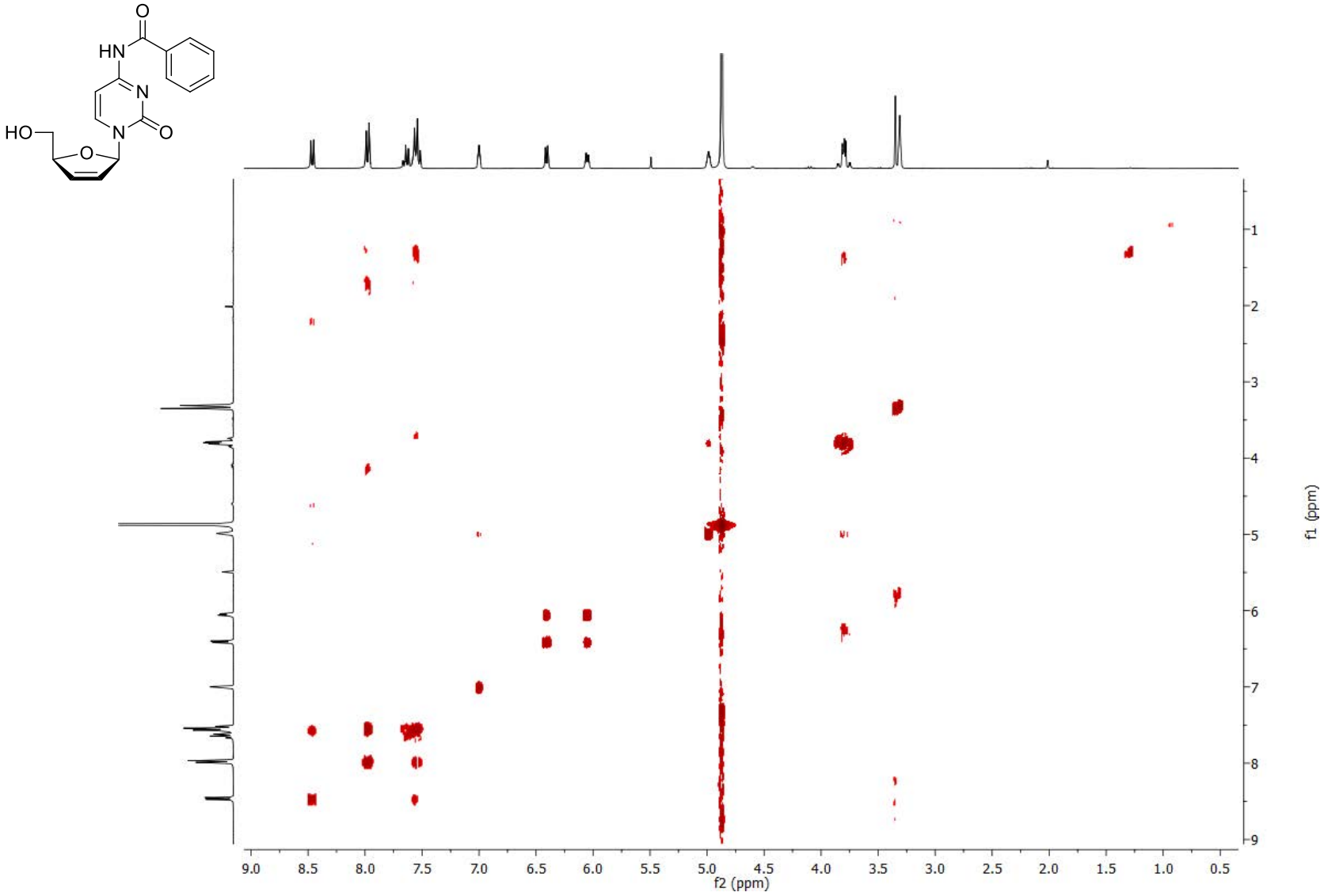
^{13}C-NMR (75.5 MHz, MeOH-d_4)



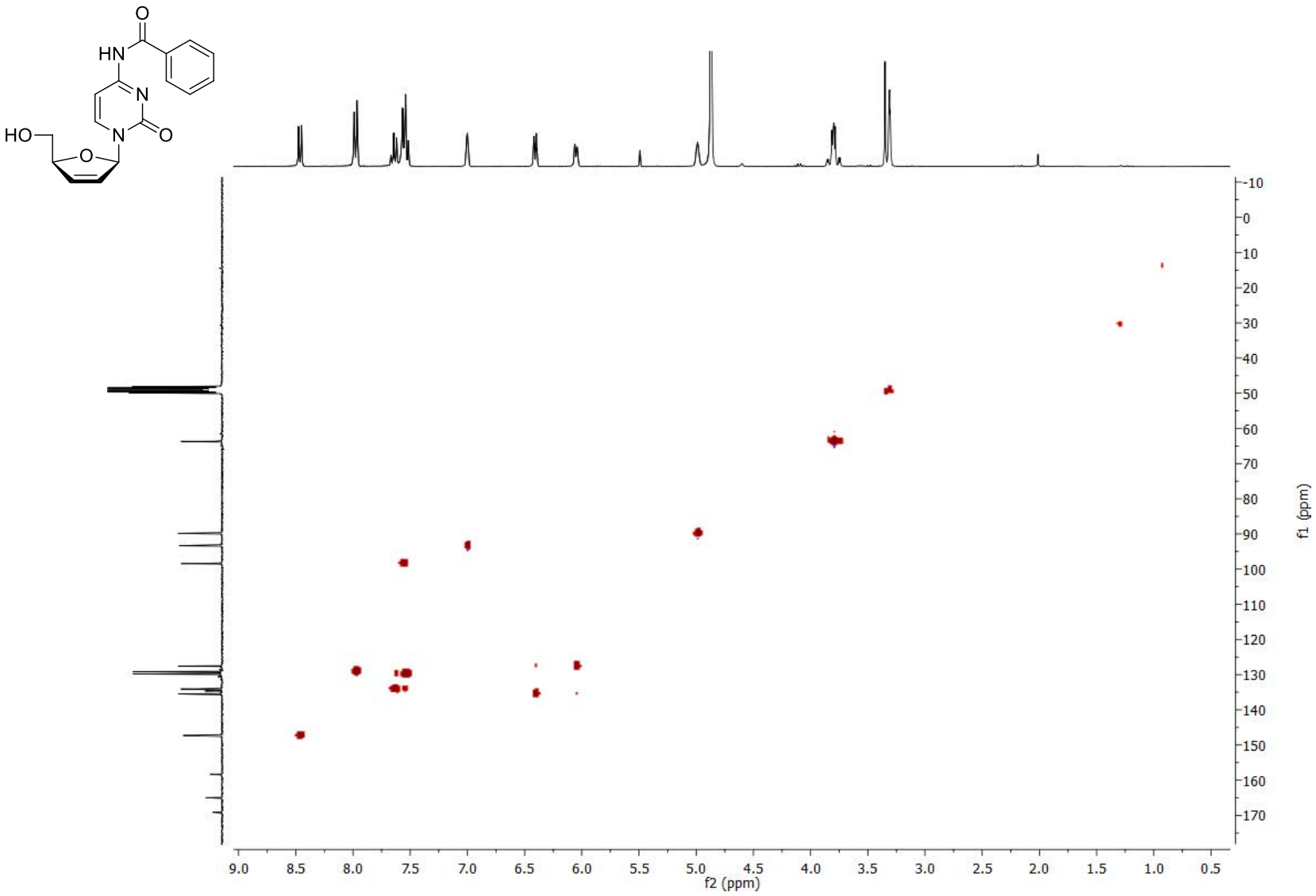
DEPT NMR (75.5 MHz, MeOH-d_4)



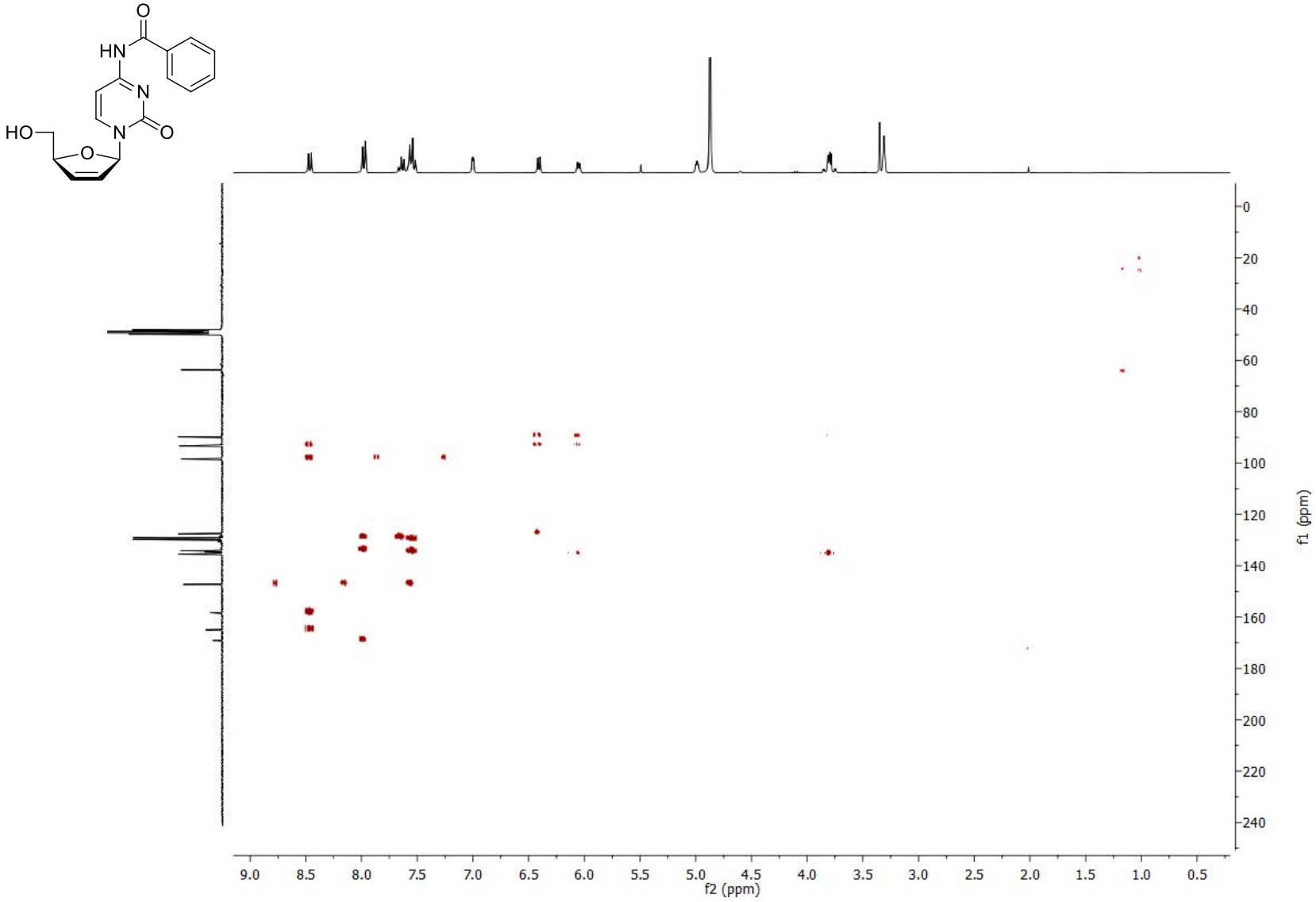
COSY NMR (MeOH-d_4)



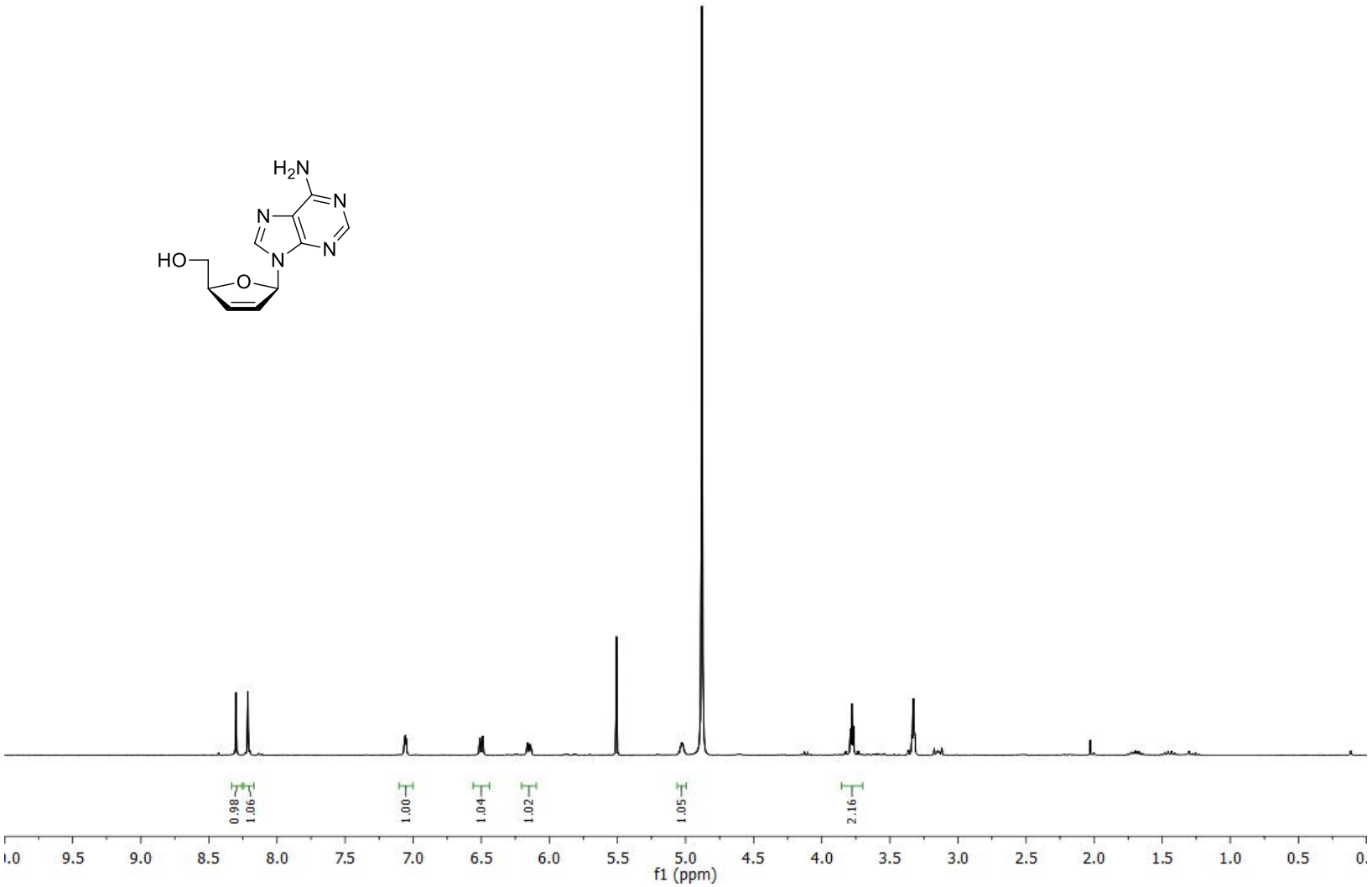
HSQC NMR (MeOH-d_4)



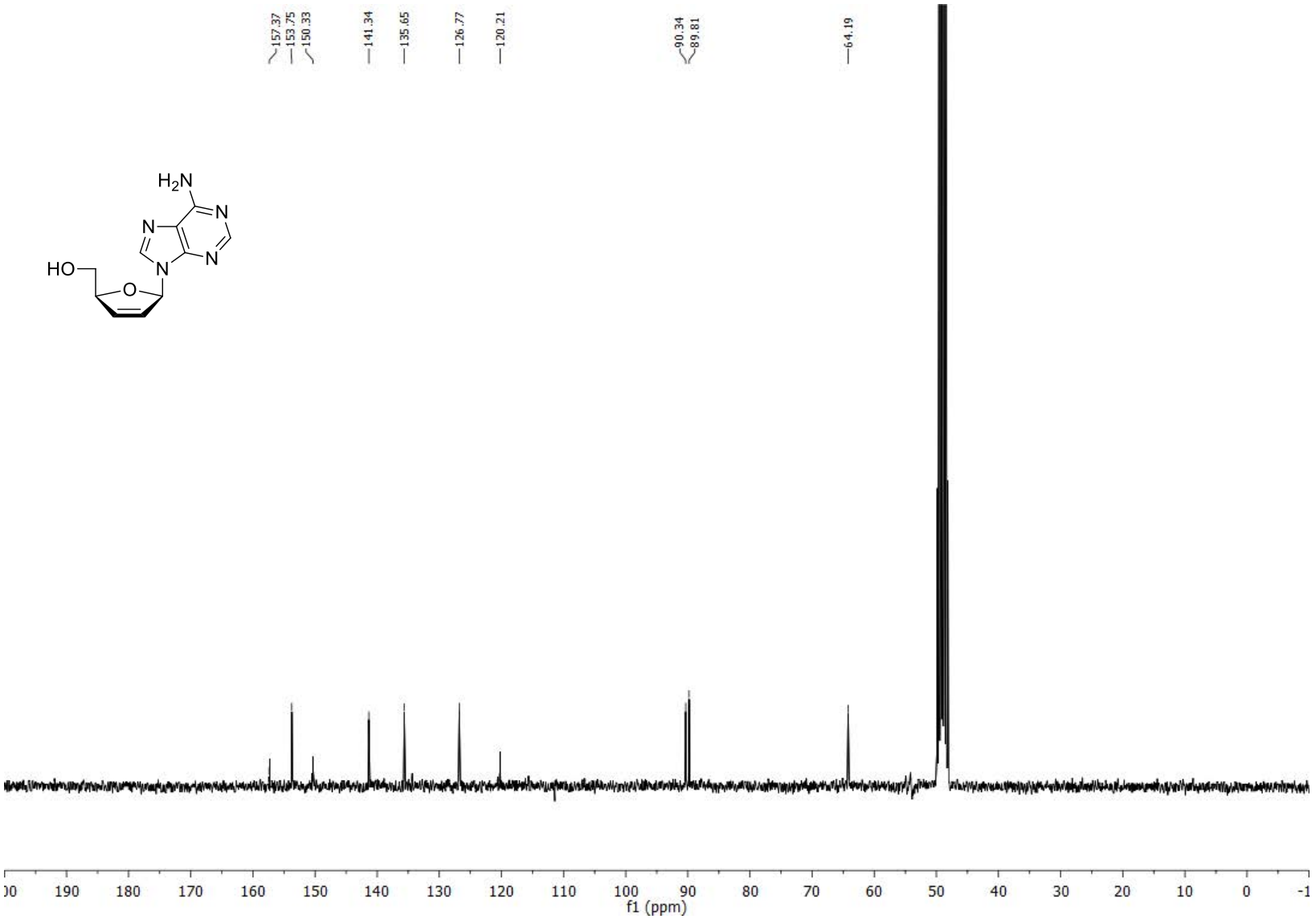
HMBC NMR (MeOH-d_4)



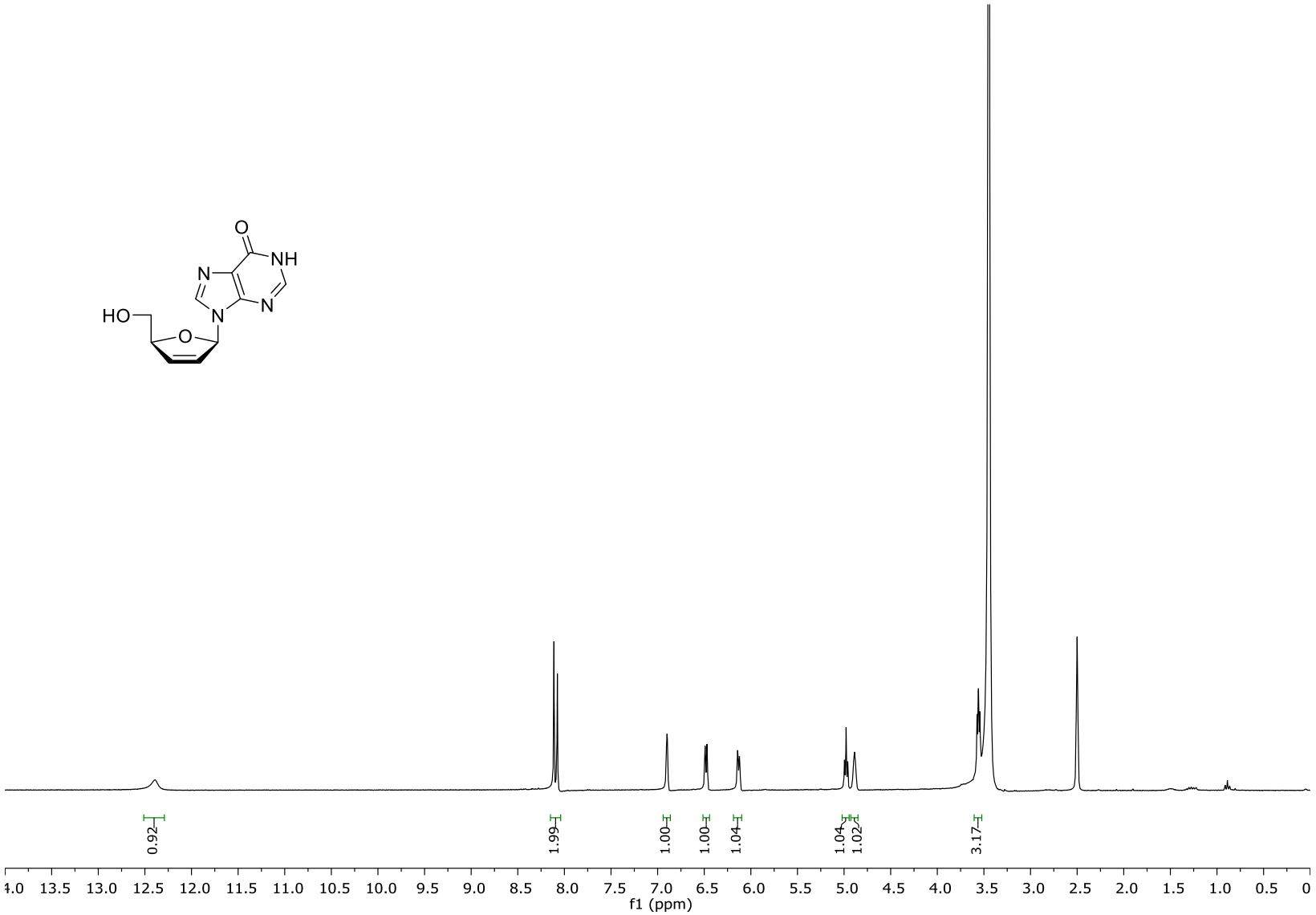
^{1}H-NMR (300.13 MHz, MeOH-d_4)



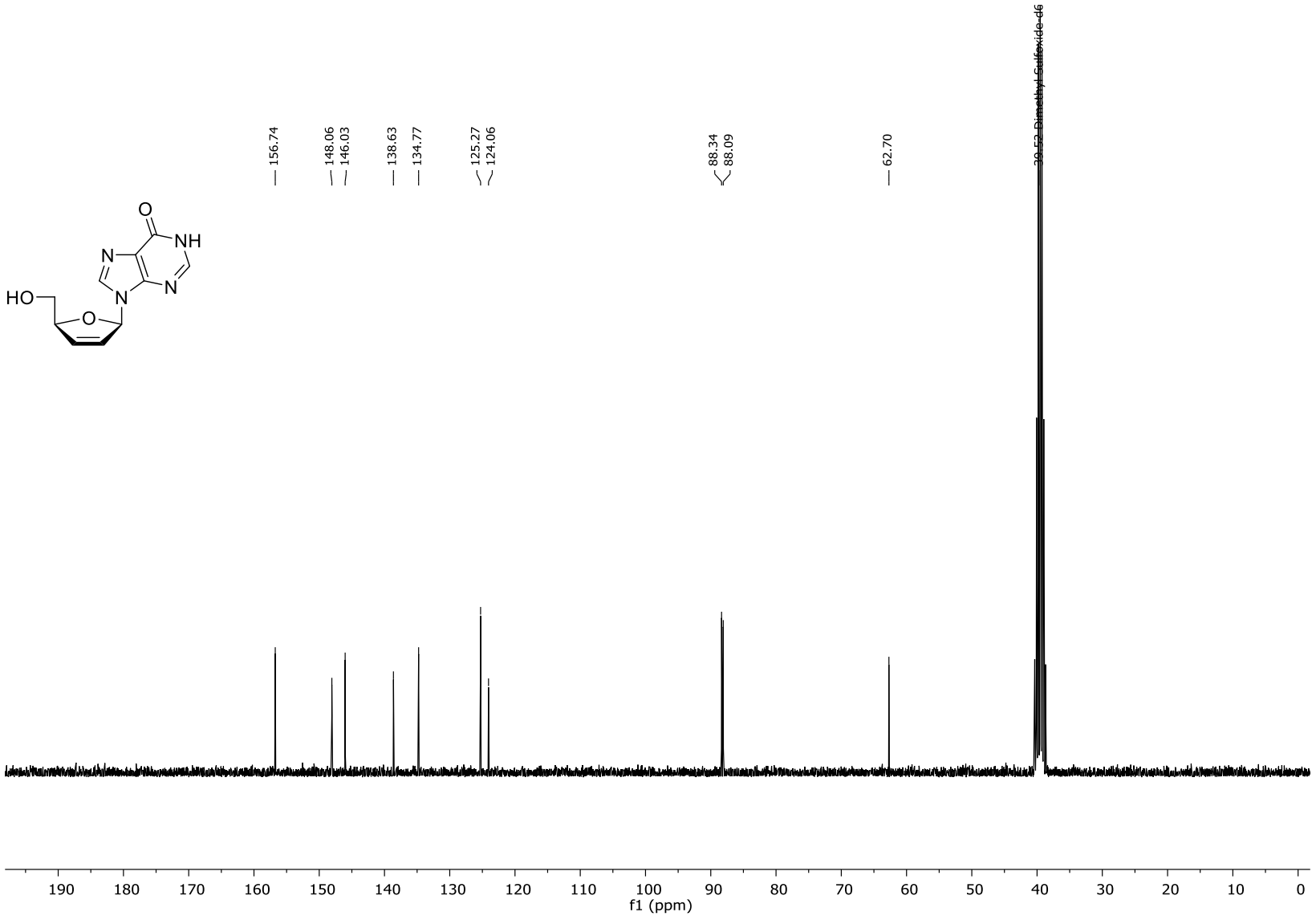
^{13}C-NMR (75.5 MHz, MeOH-d_4)



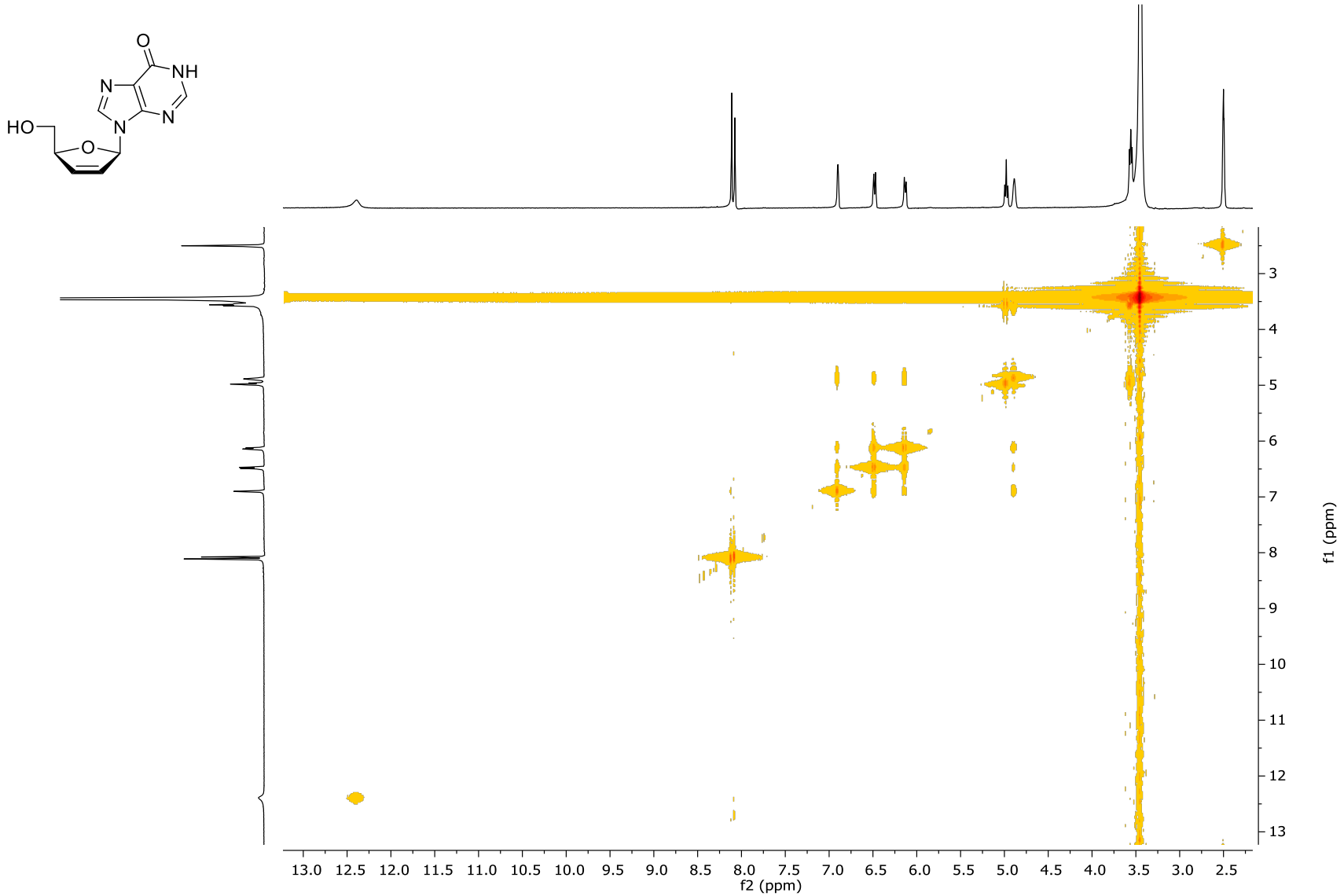
^{1}H-NMR (300.13 MHz, DMSO-d_6)



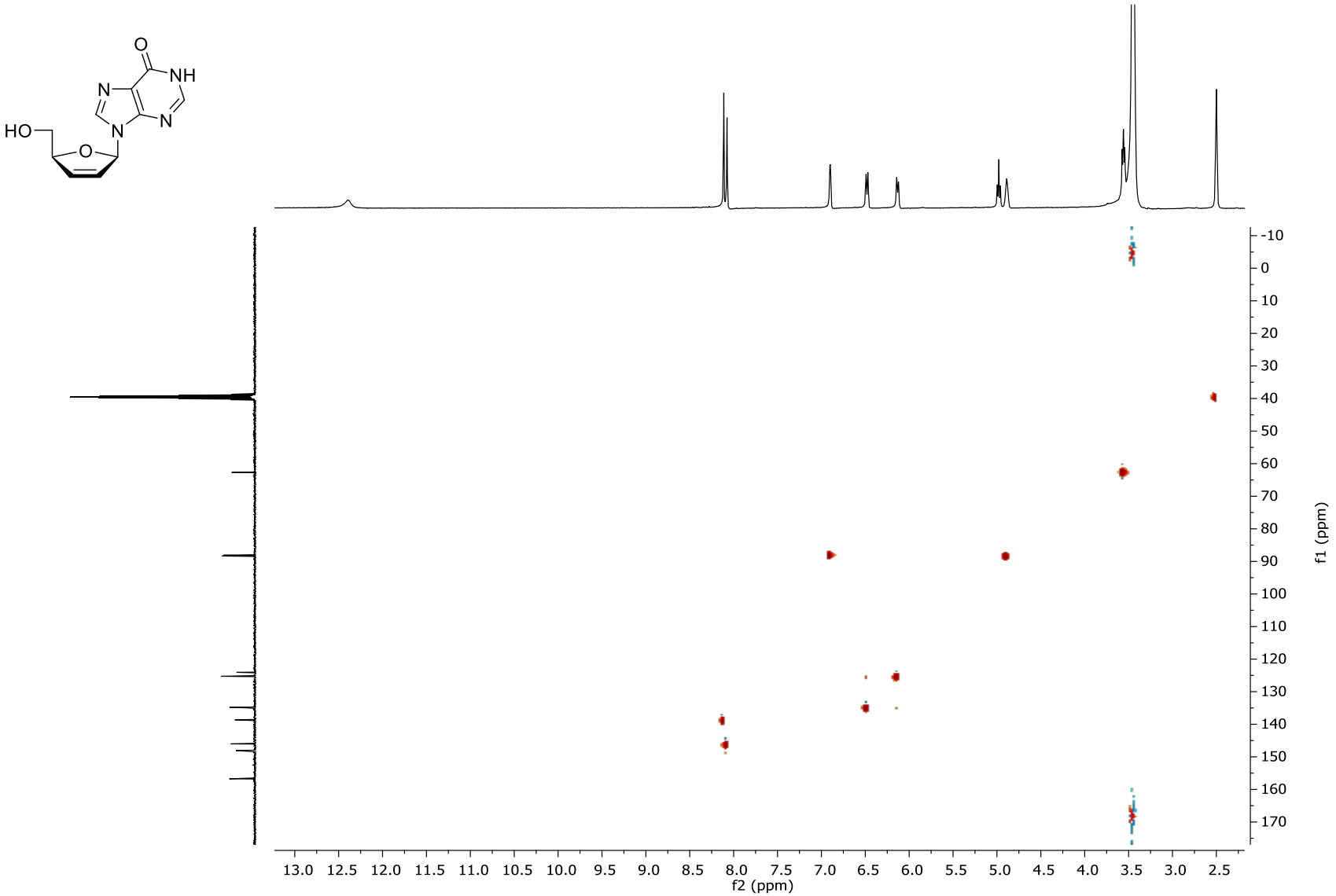
^{13}C-NMR (75.5 MHz, DMSO-d_6)



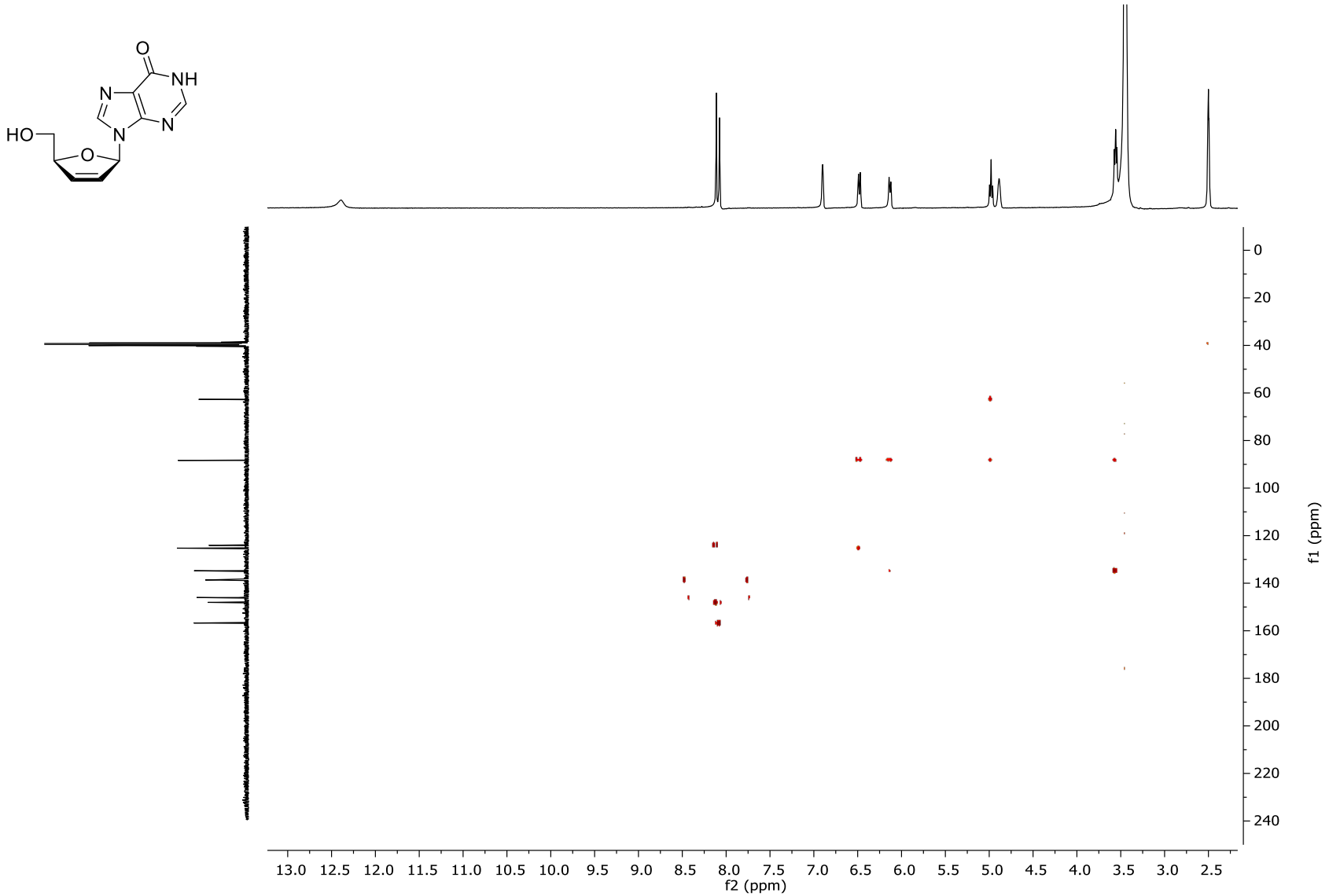
COSY NMR (DMSO-d_6)



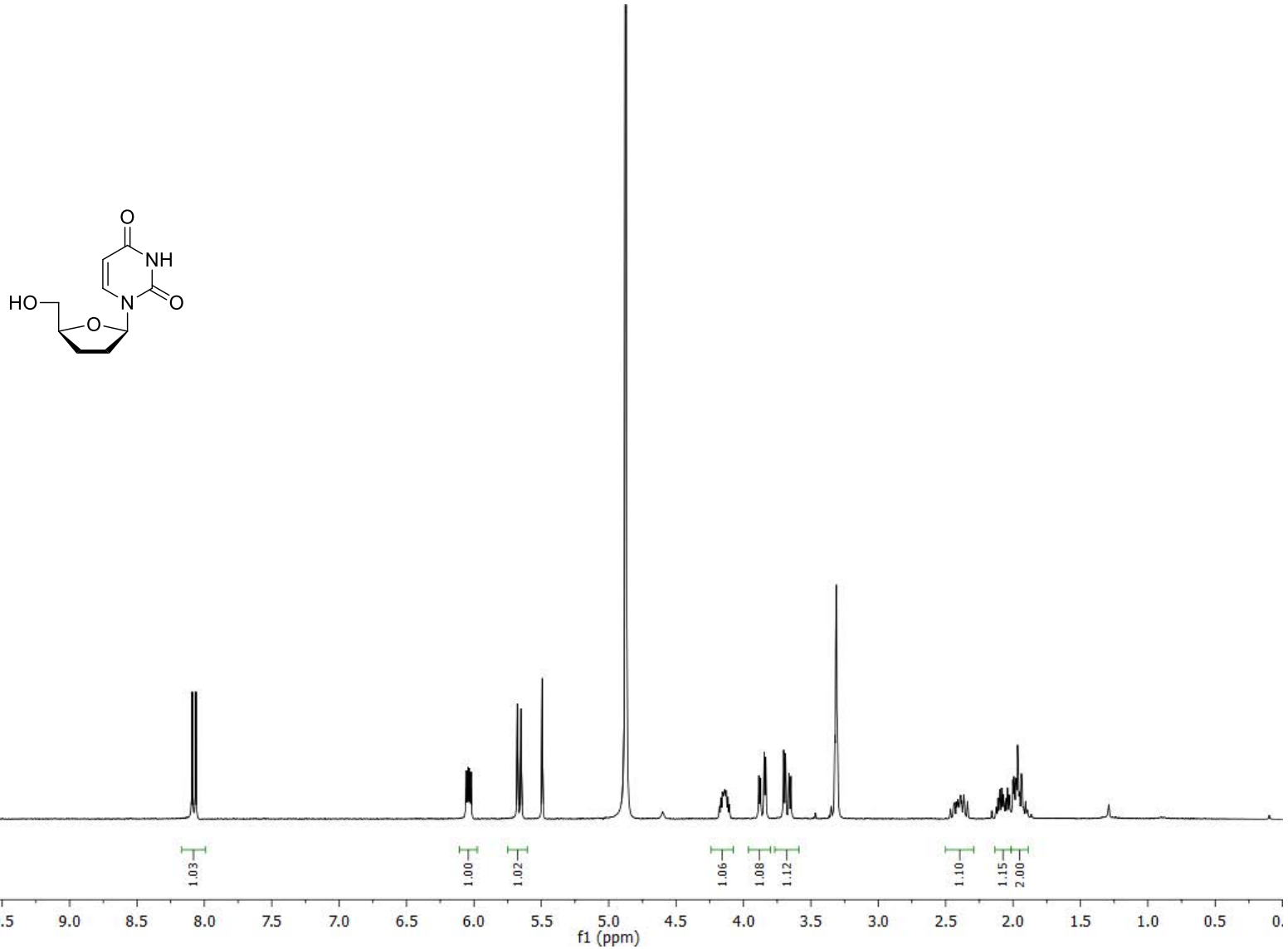
HSQC NMR (DMSO-d_6)



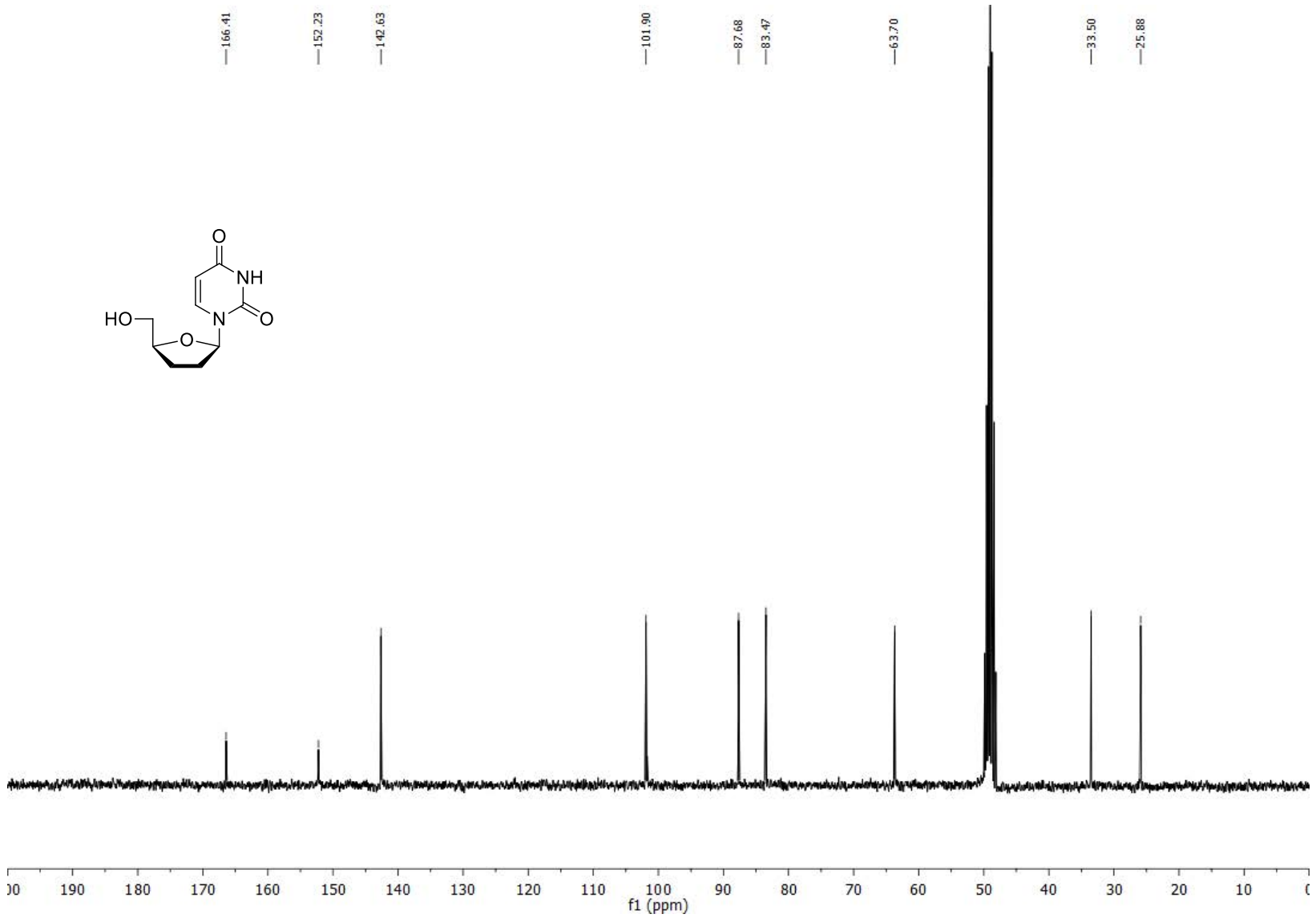
HMBC NMR (DMSO-d_6)



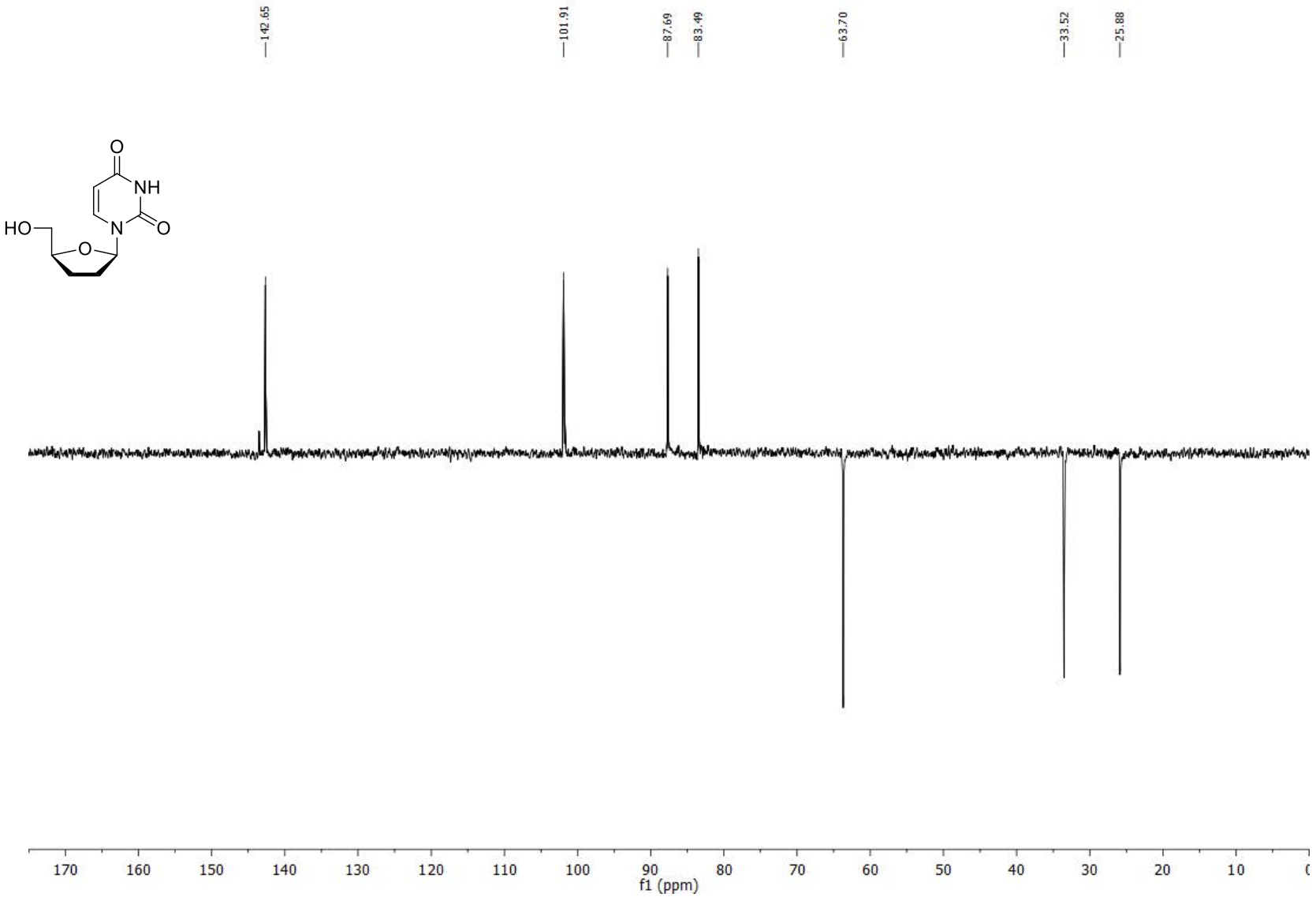
^{1}H-NMR (300.13 MHz, MeOH-d_4)



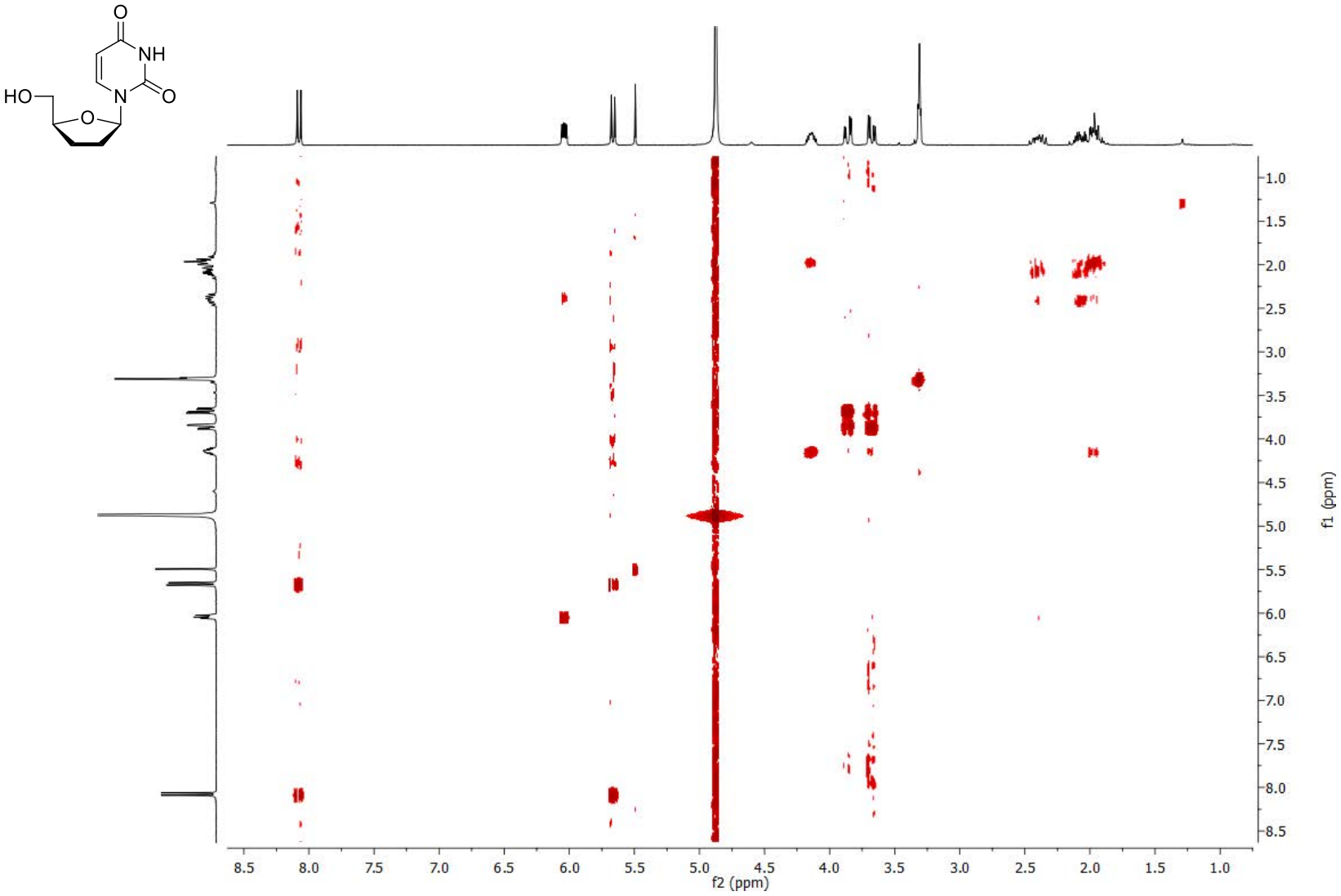
^{13}C-NMR (75.5 MHz, MeOH-d_4)



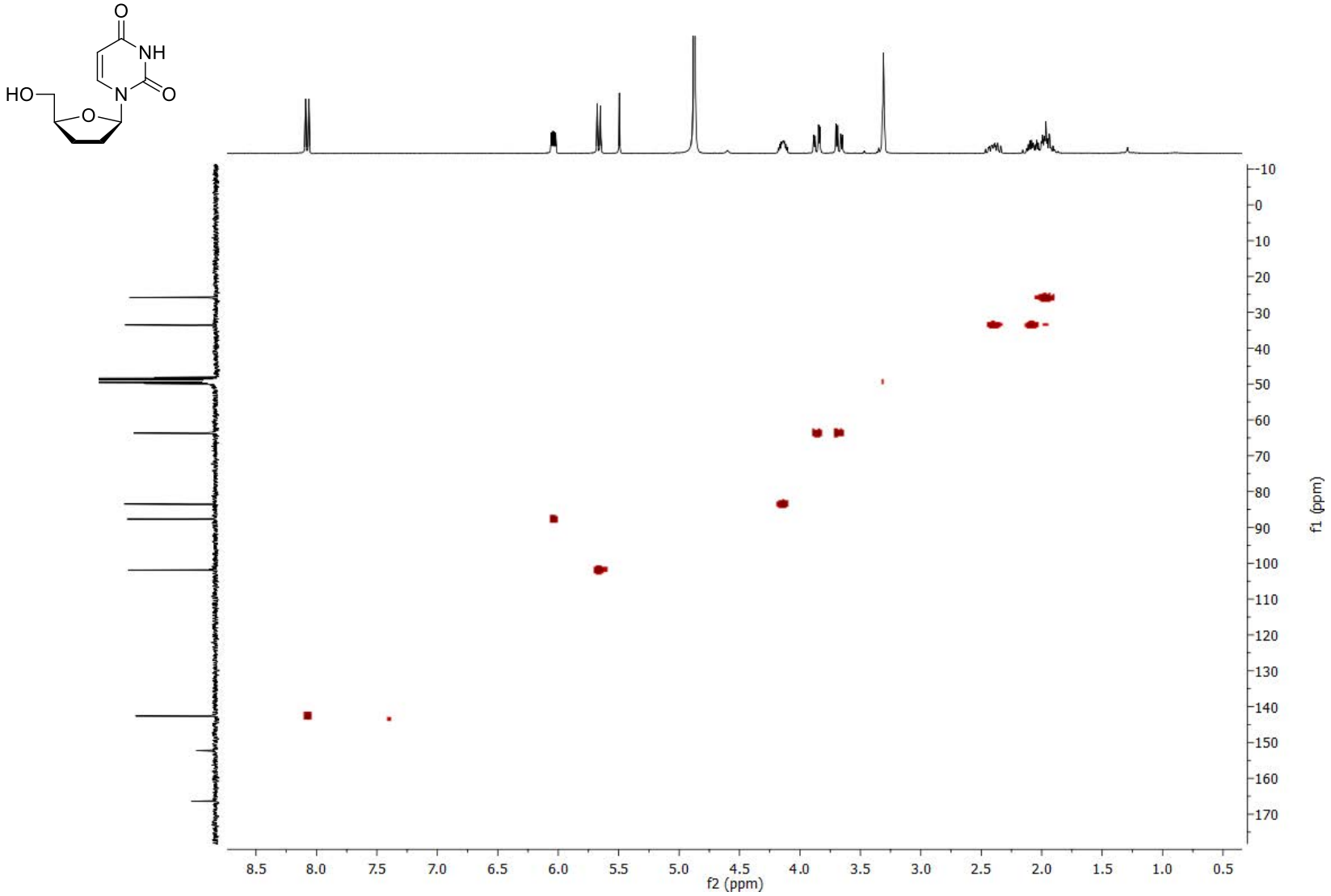
DEPT NMR (75.5 MHz, MeOH-d_4)



COSY NMR (MeOH-d_4)

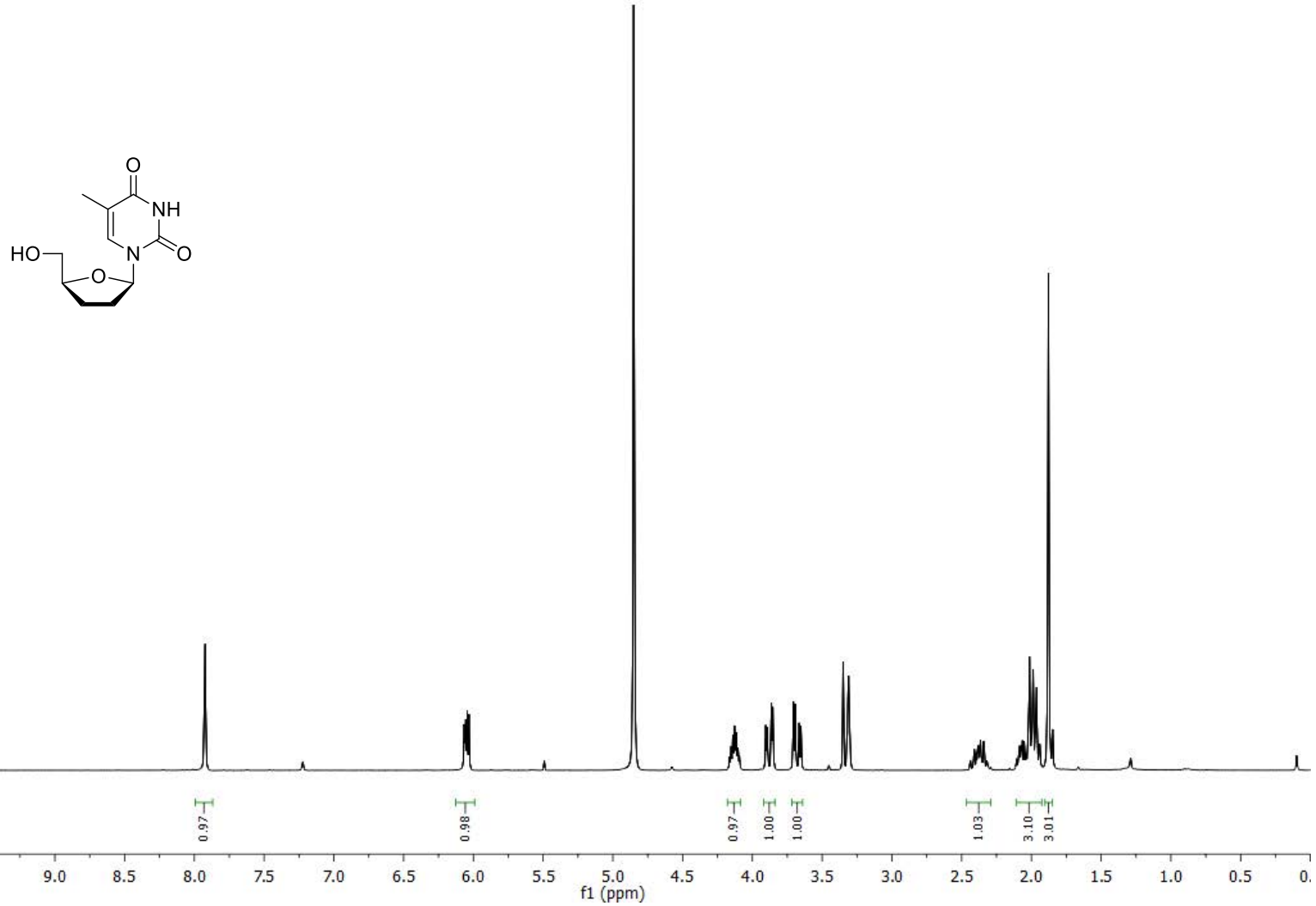


HSQC NMR (MeOH-d_4)

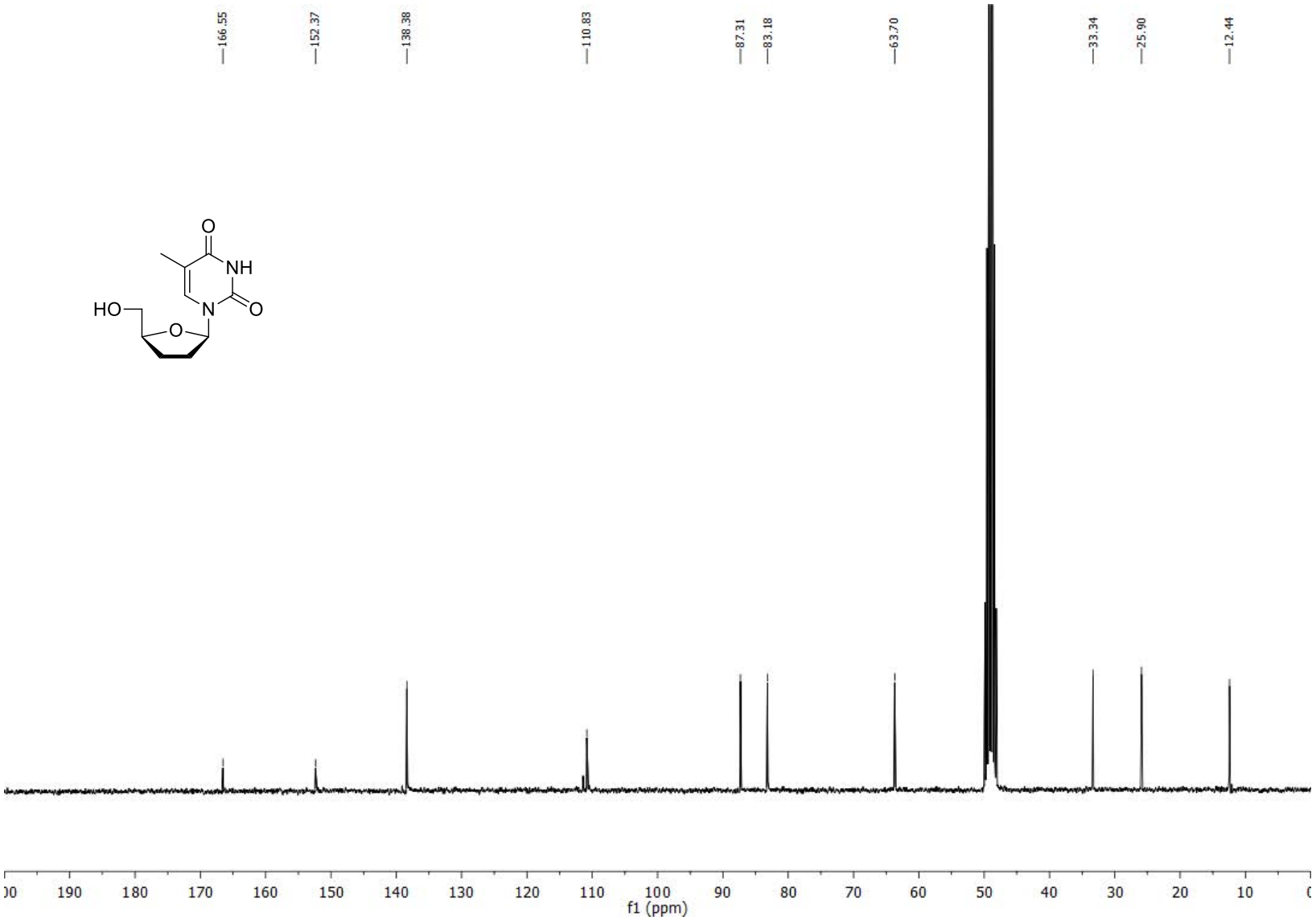


3’-Deoxy-β-D-5-thymidine (9b)

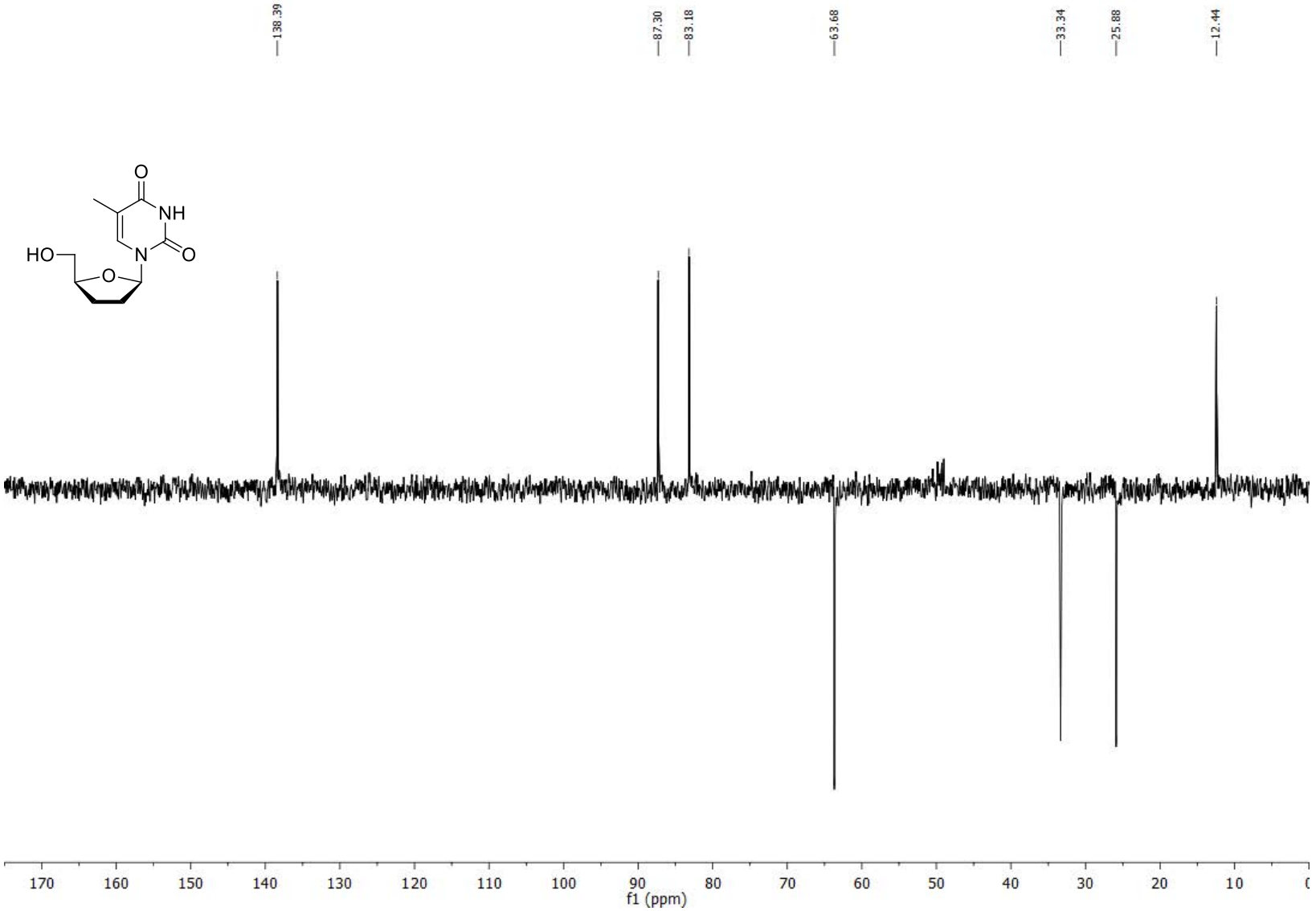
^{1}H-NMR (300.13 MHz, MeOH-d_4)



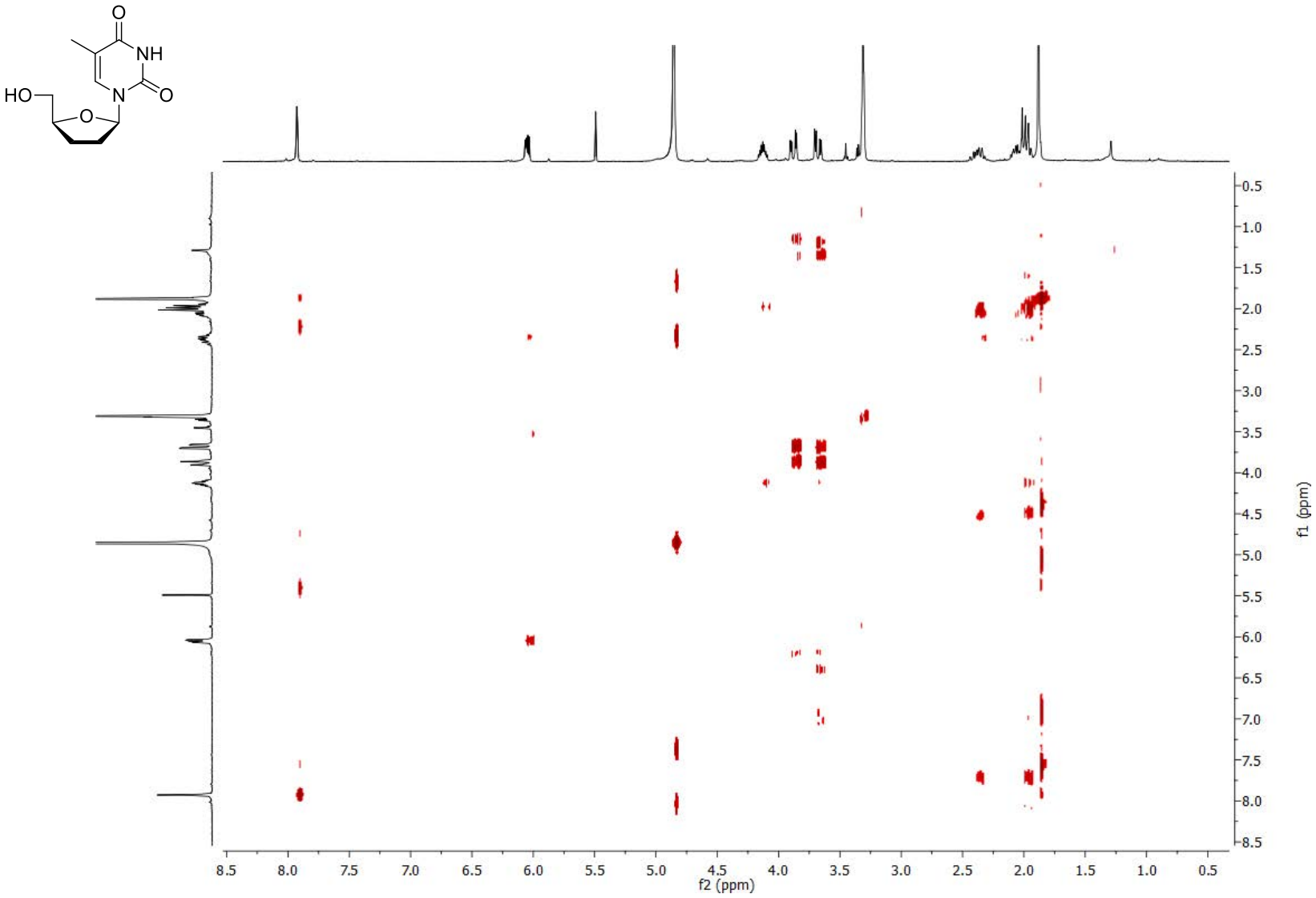
^{13}C-NMR (75.5 MHz, MeOH-d_4)



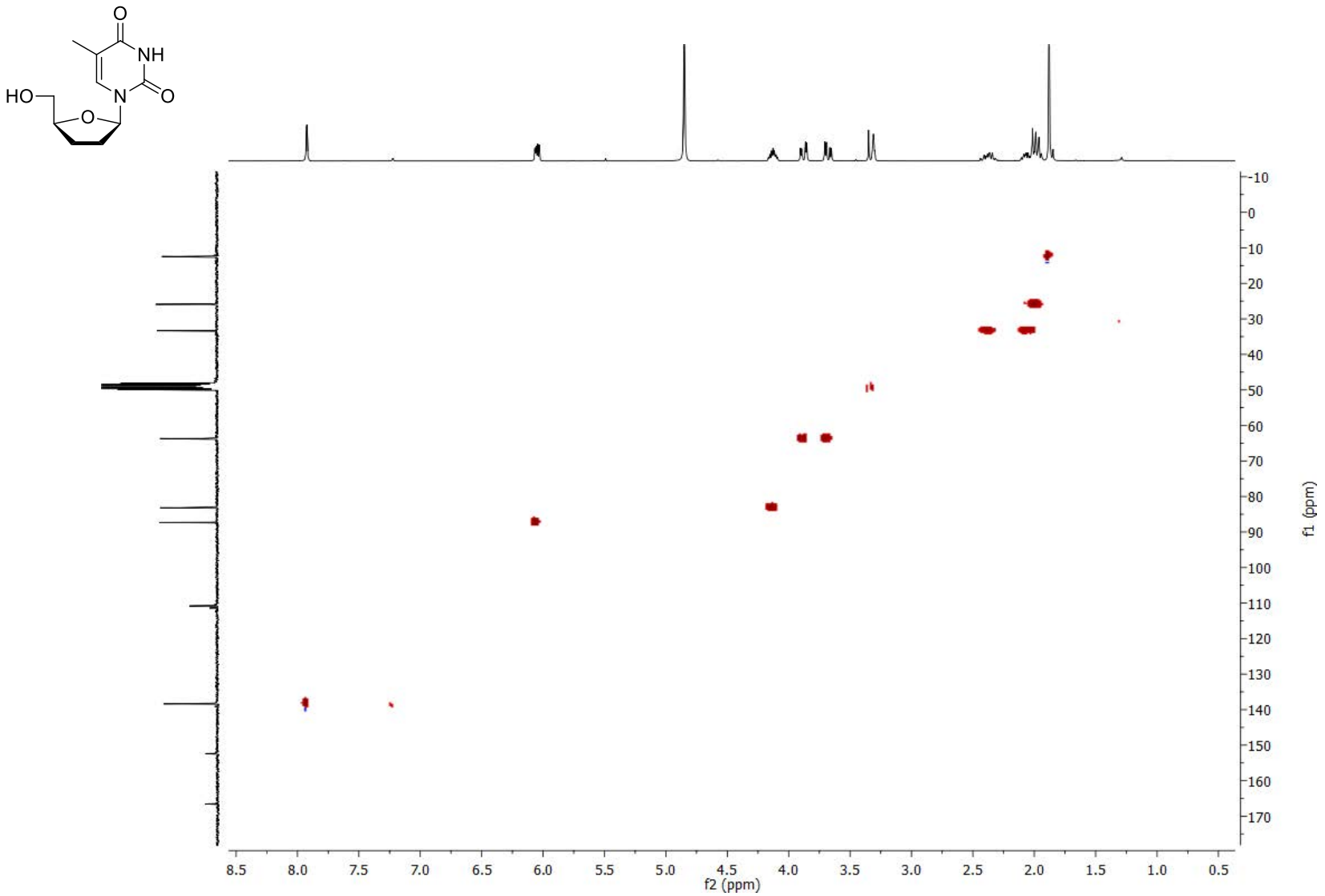
DEPT NMR (75.5 MHz, MeOH-d_4)



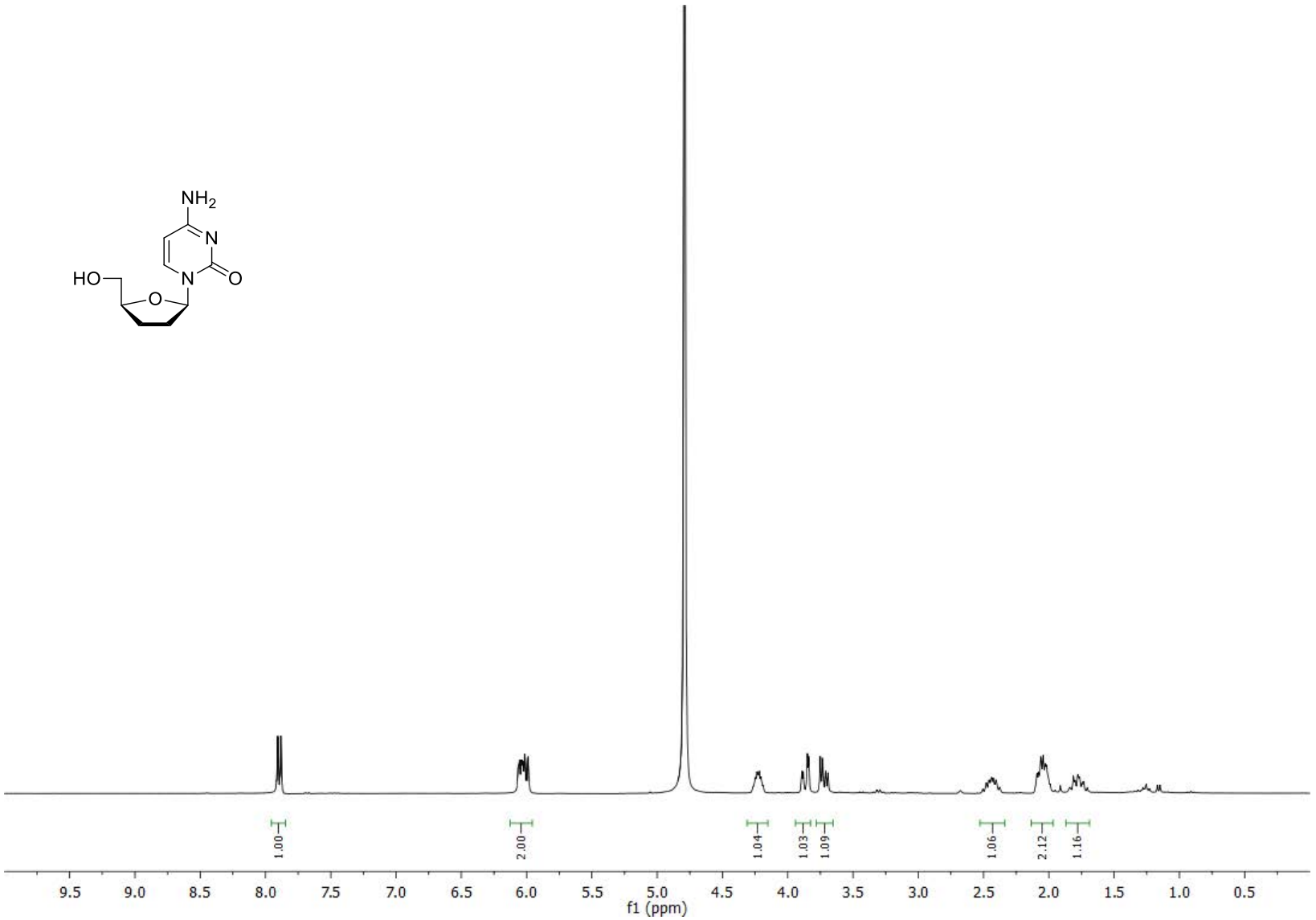
COSY NMR (MeOH-d_4)



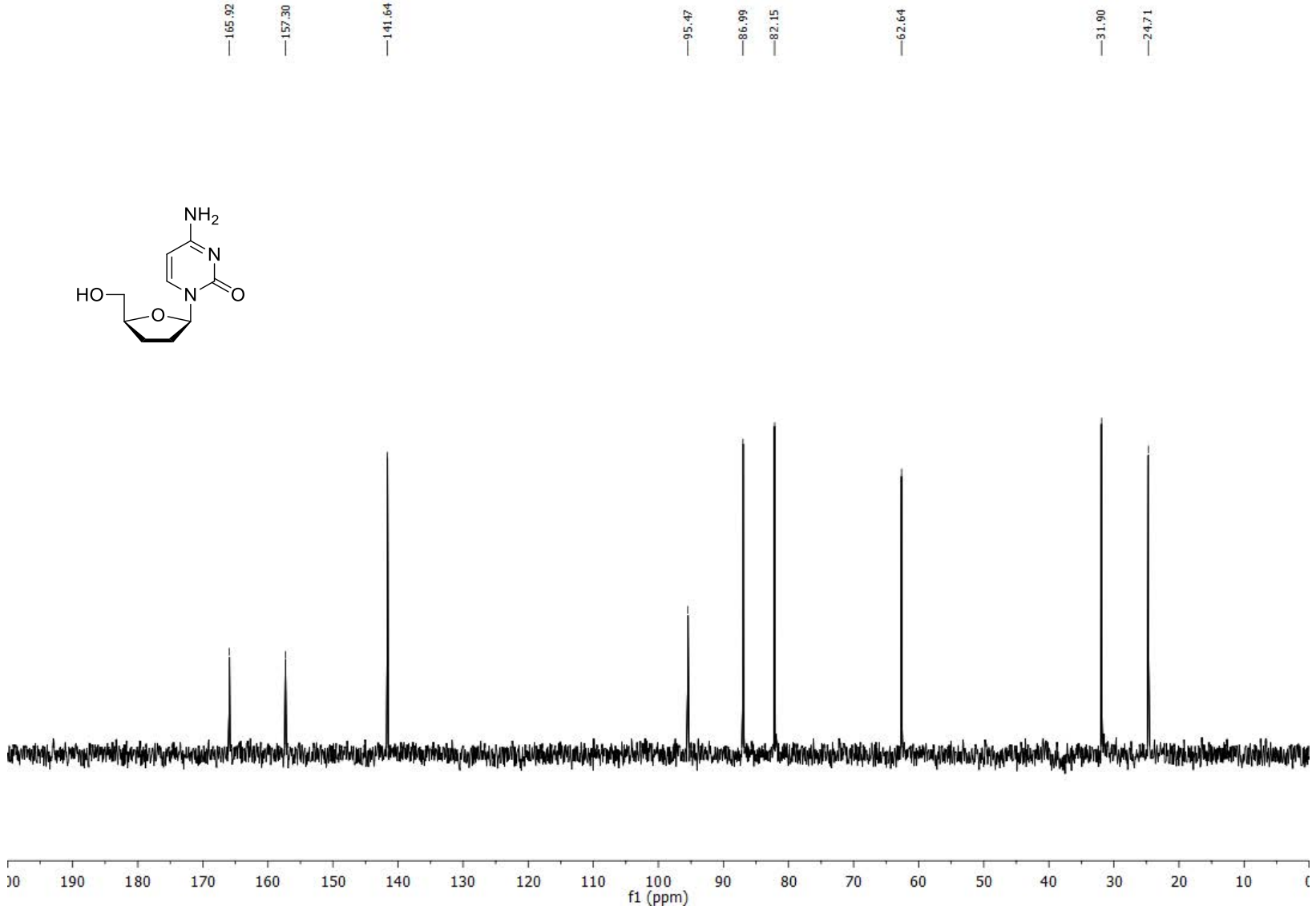
HSQC NMR (MeOH-d_4)



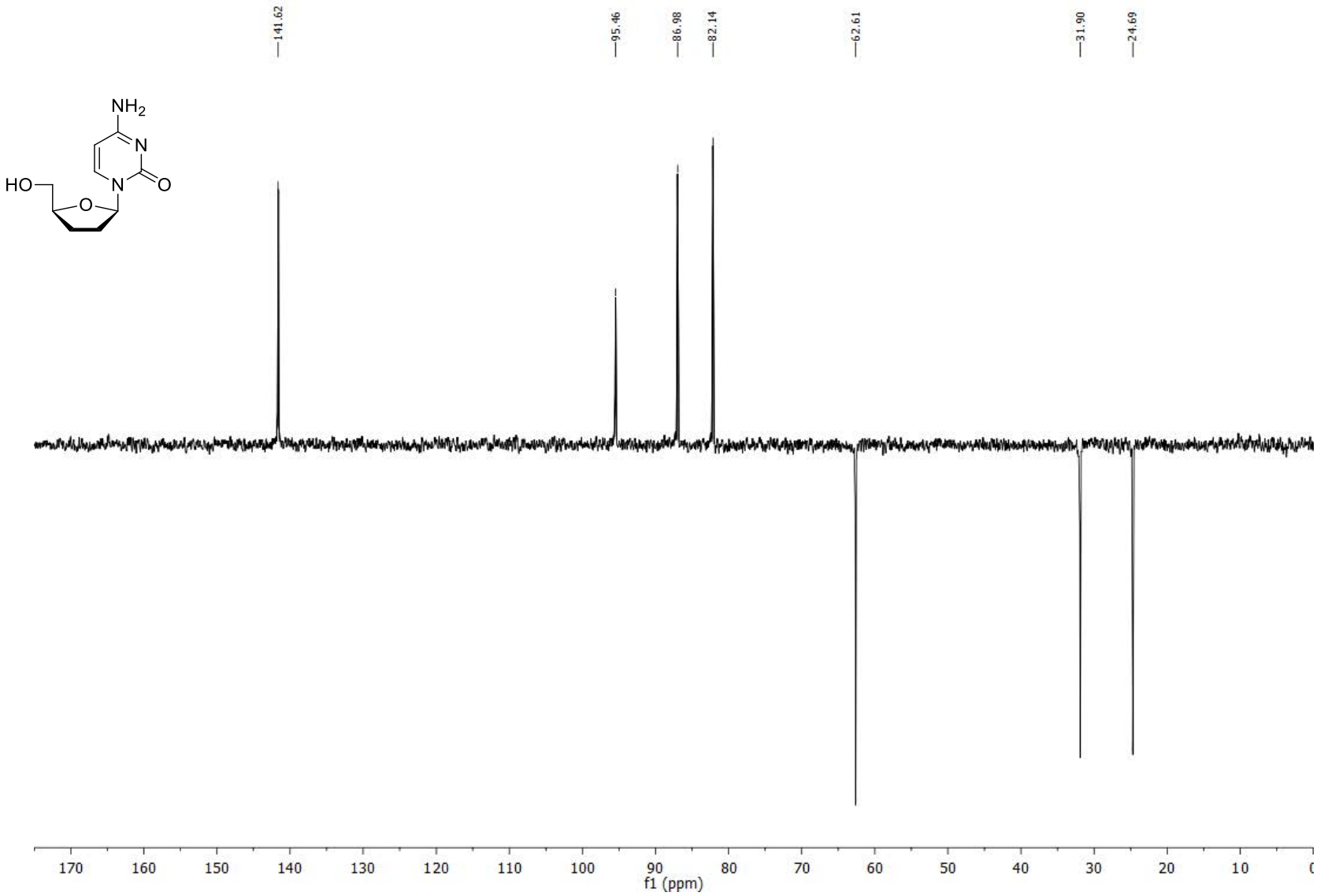
^{1}H-NMR (300.13 MHz, D_2O)



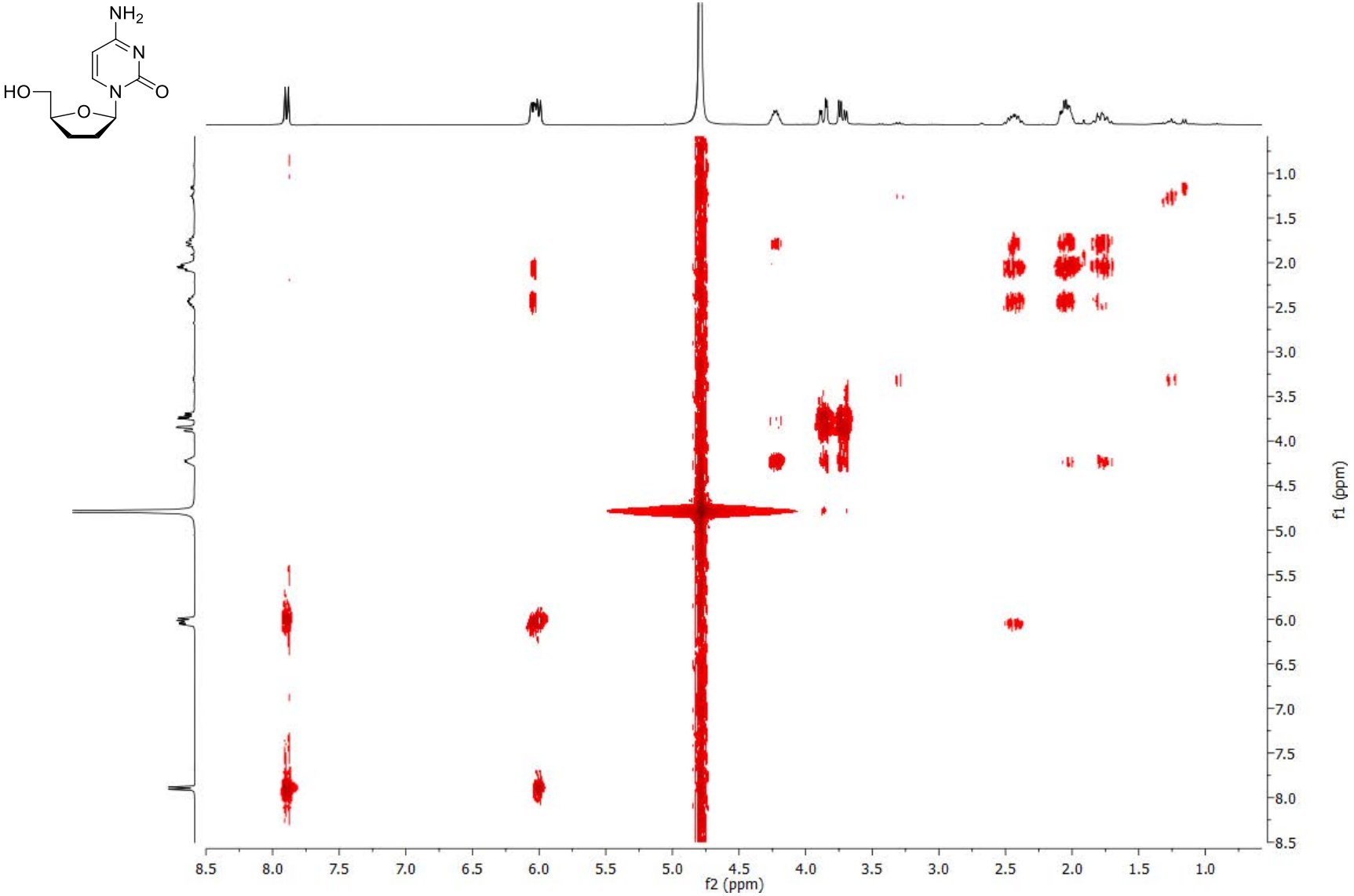
^{13}C-NMR (75.5 MHz, D_2O)



DEPT (75.5 MHz, D_2O)

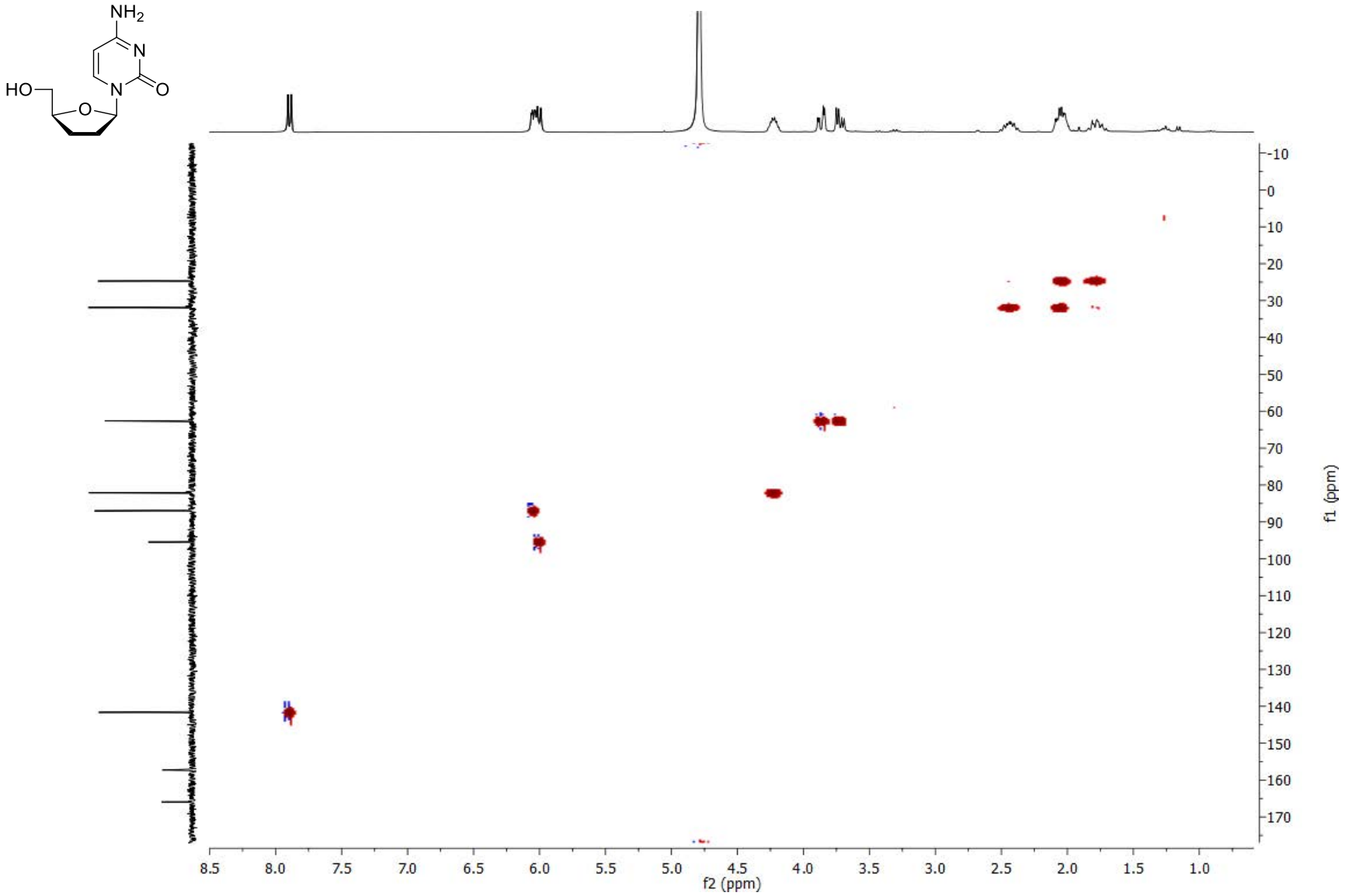


COSY NMR (D_2O)

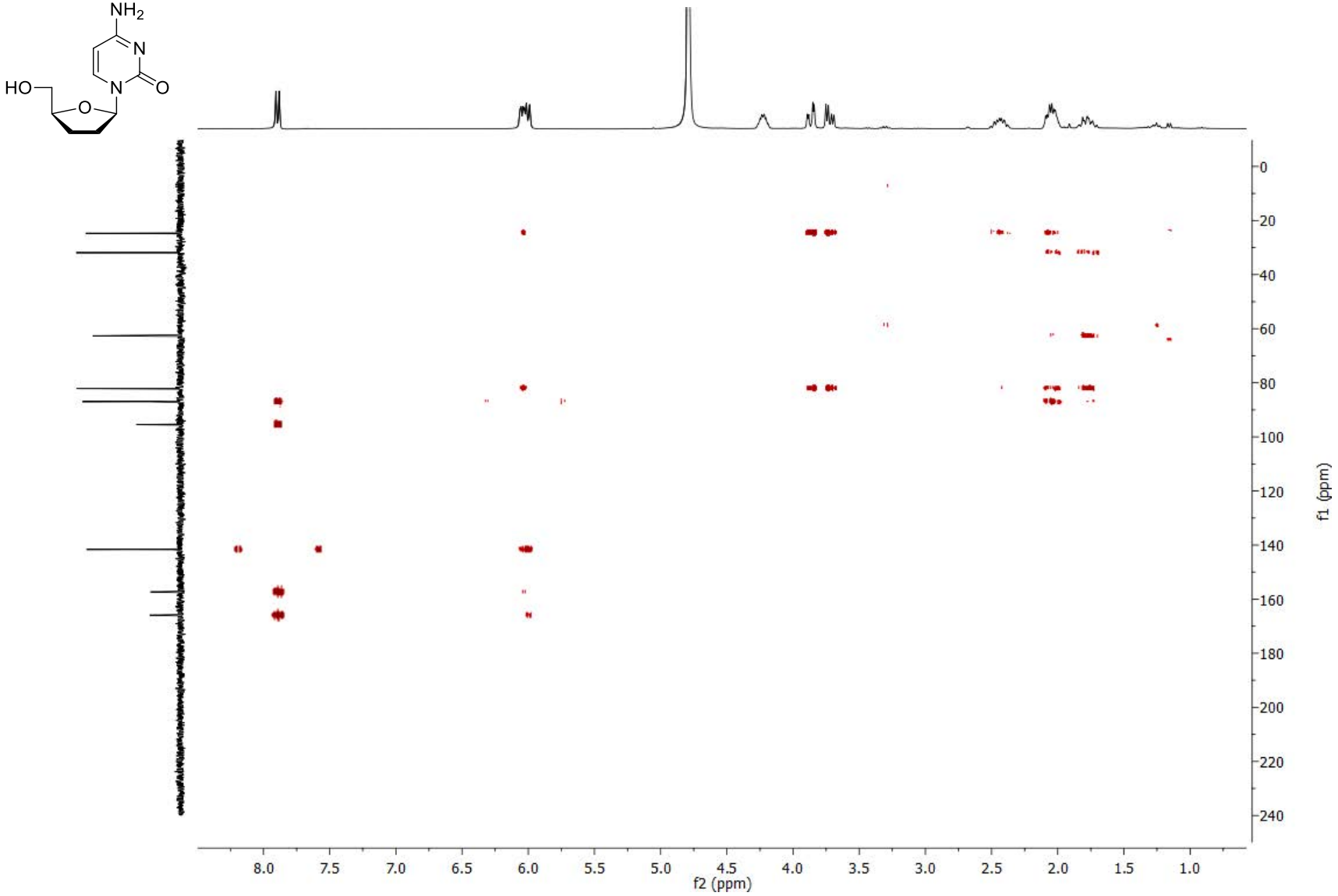


2’,3’-Dideoxy-β-D-cytidine (9c)

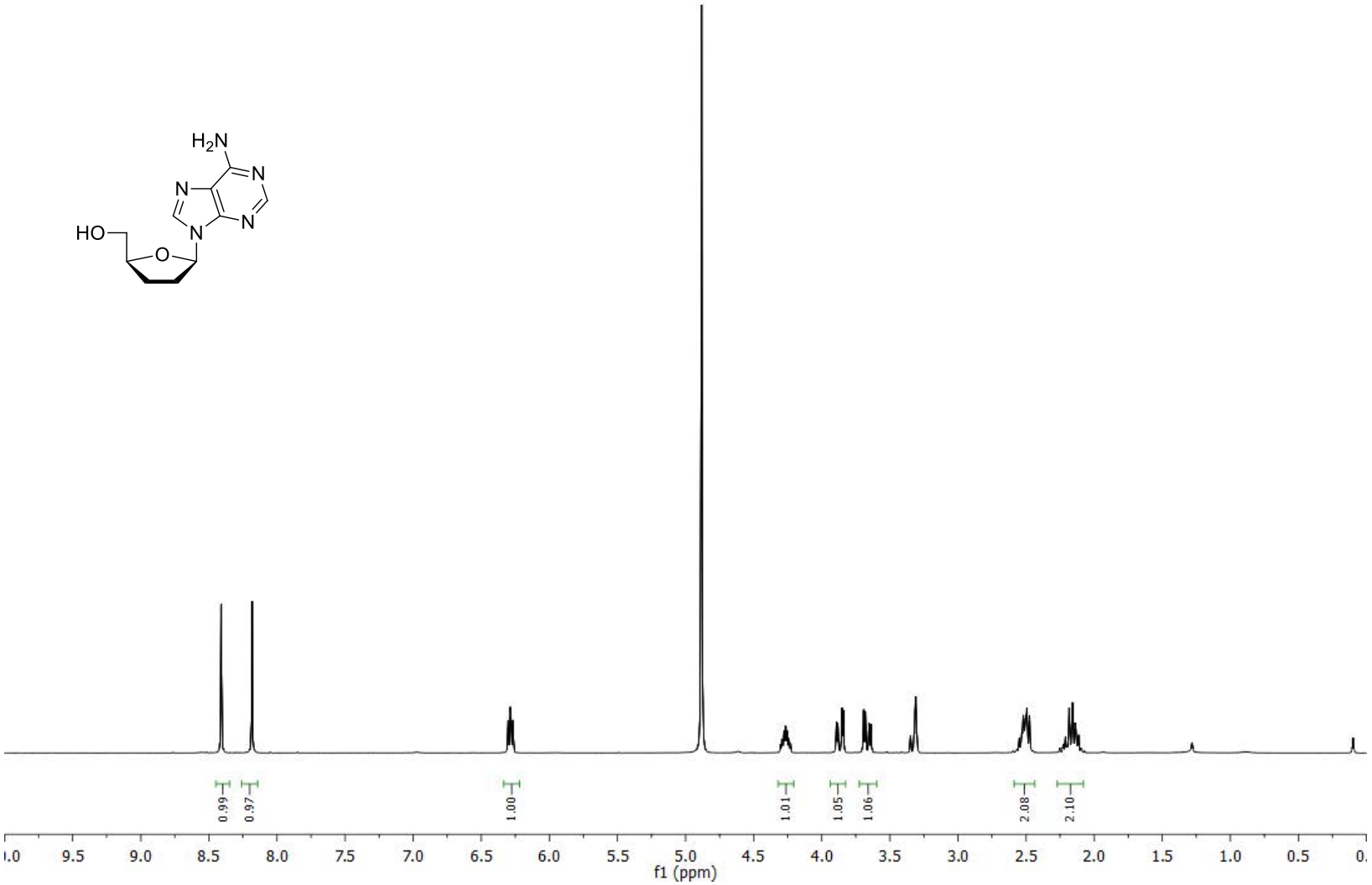
HSQC NMR (D_2O)



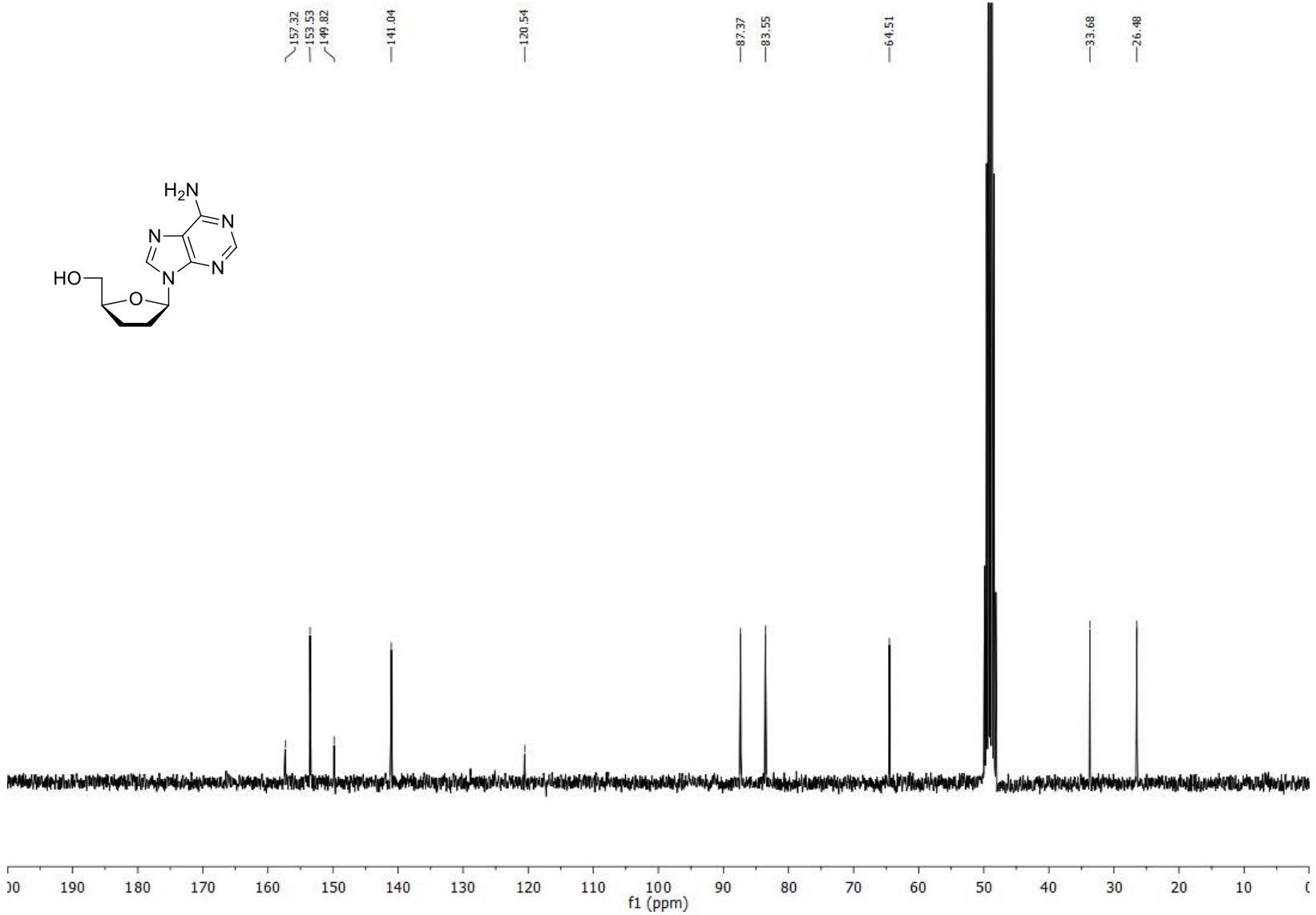
HMBC NMR (D_2O)


NH2
N
HO
N
O
O
0
20
40
60
80
100
120
140
160
180
200
220
240
f1 (ppm)
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
f2 (ppm)

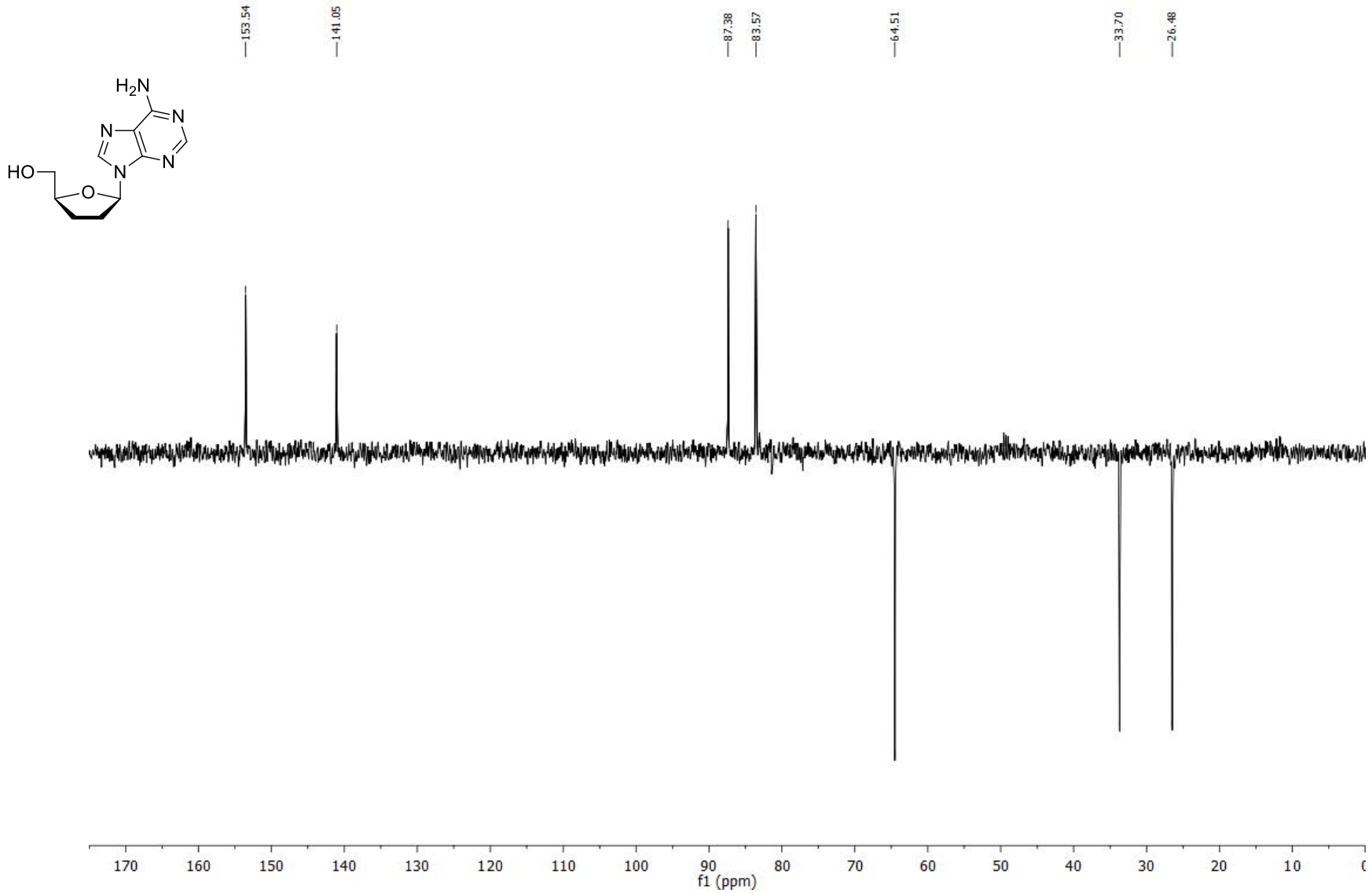
^{1}H-NMR (300.13 MHz, MeOH-d_4)



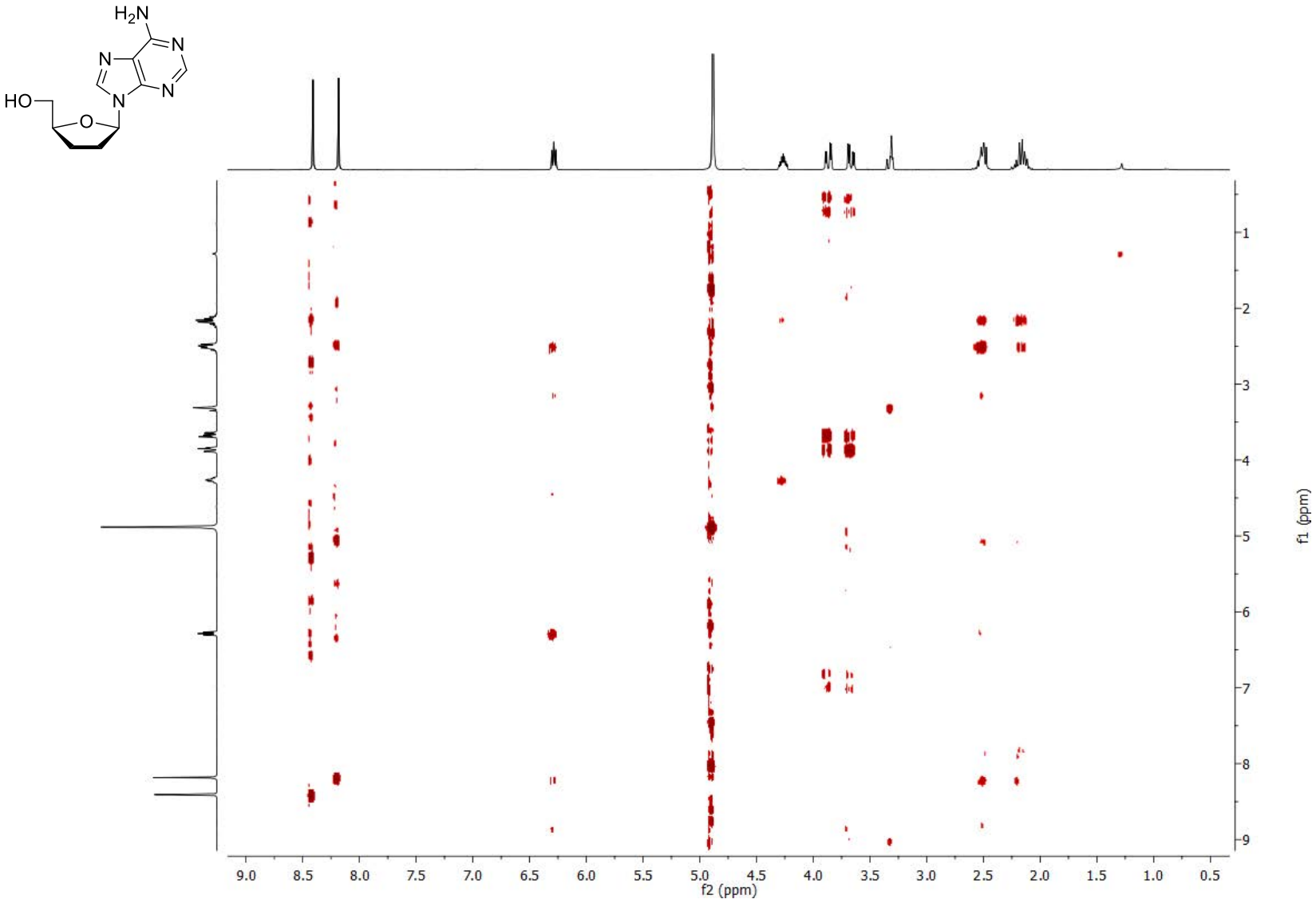
^{13}C-NMR (75.5 MHz, MeOH-d_4)



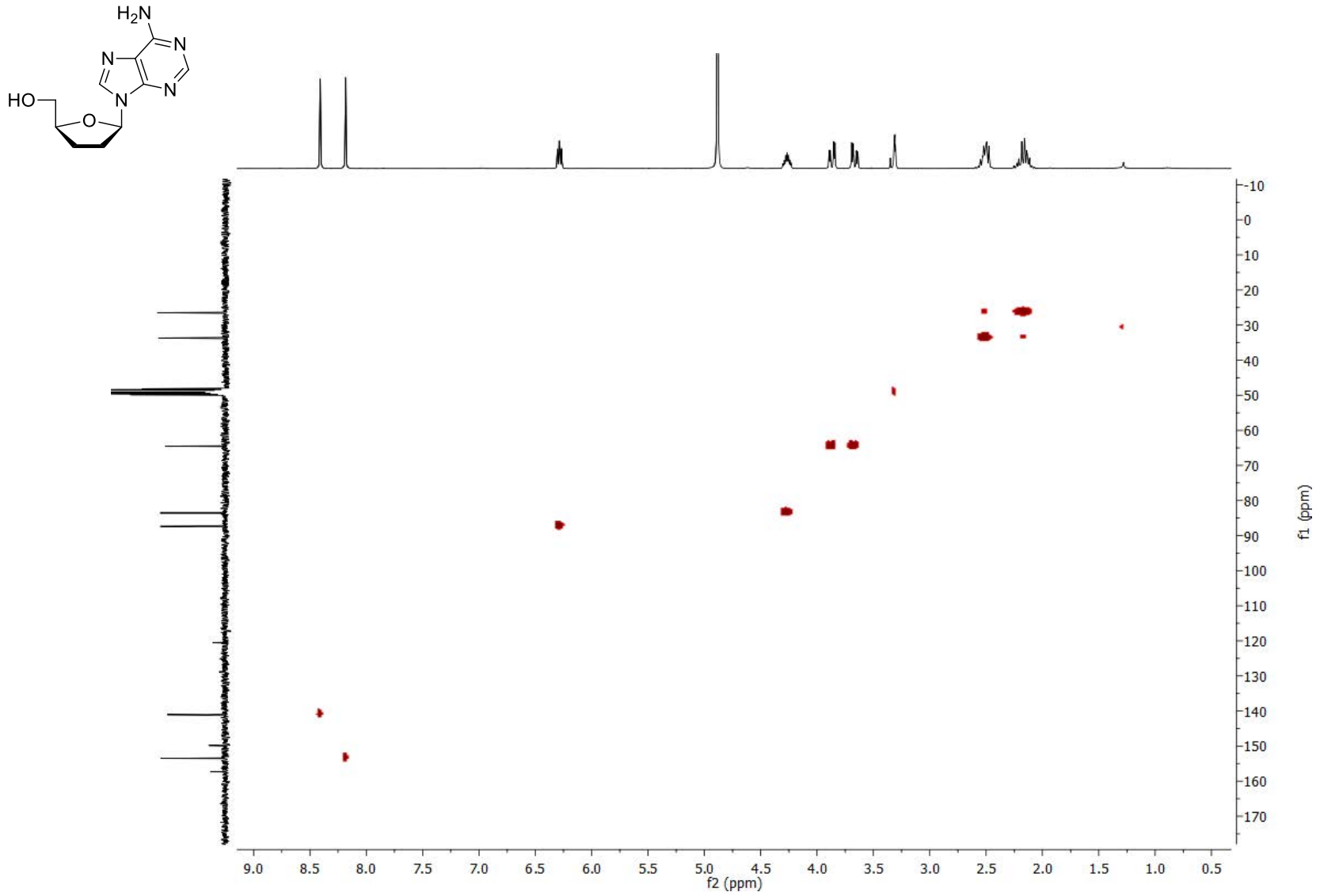
DEPT NMR (75.5 MHz, MeOH-d_4)



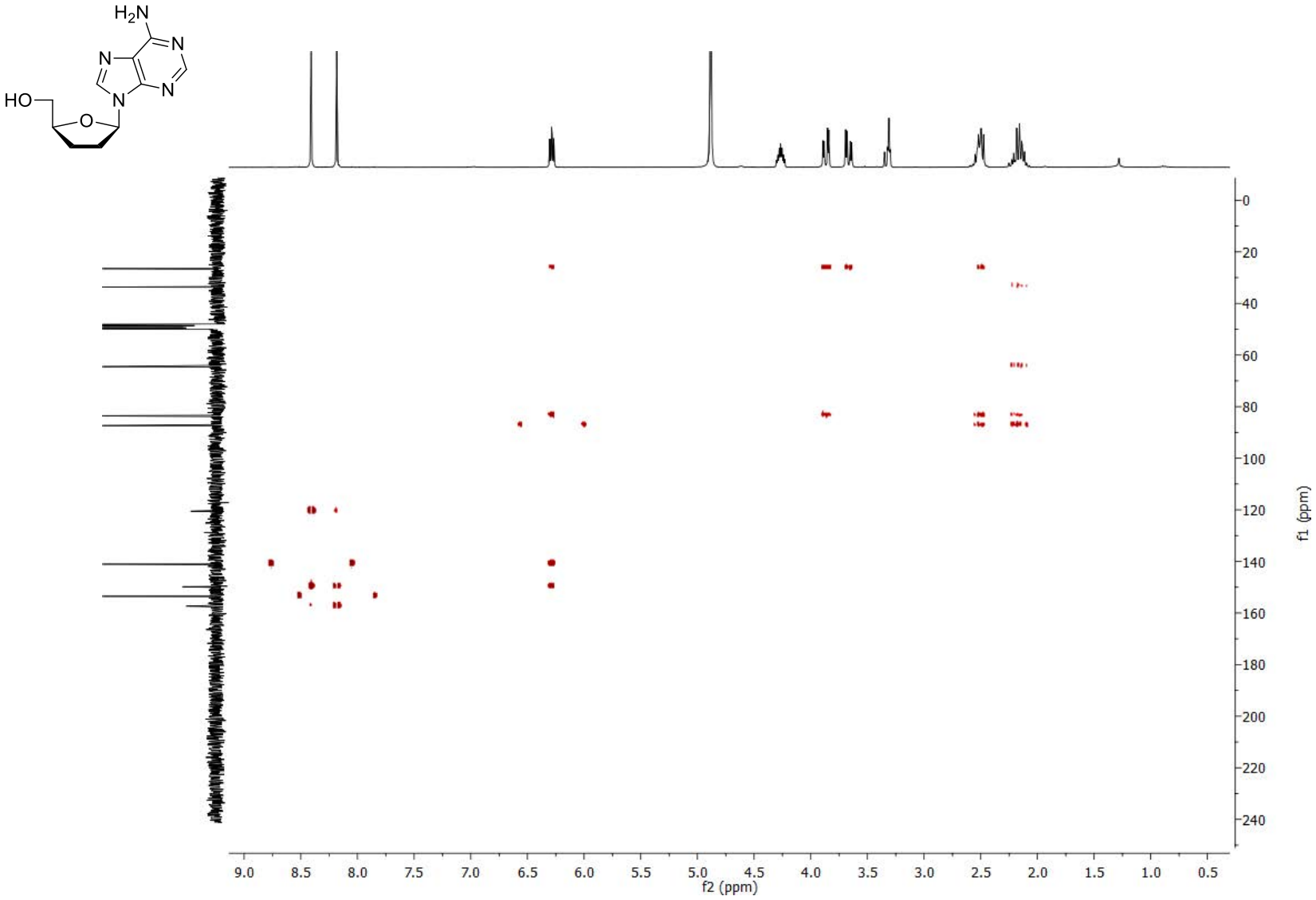
COSY NMR (MeOH-d_4)



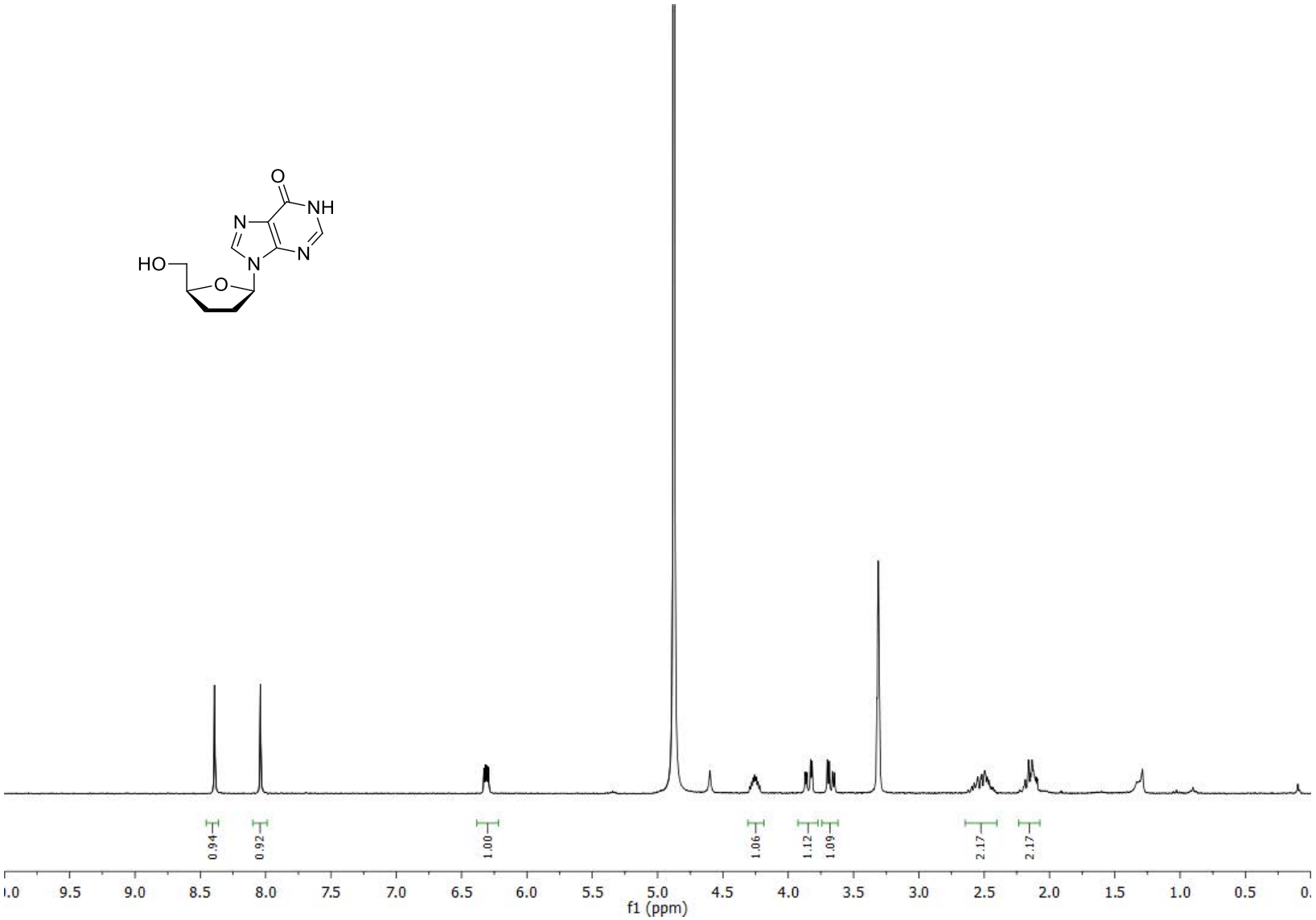
HSQC NMR (MeOH-d_4)



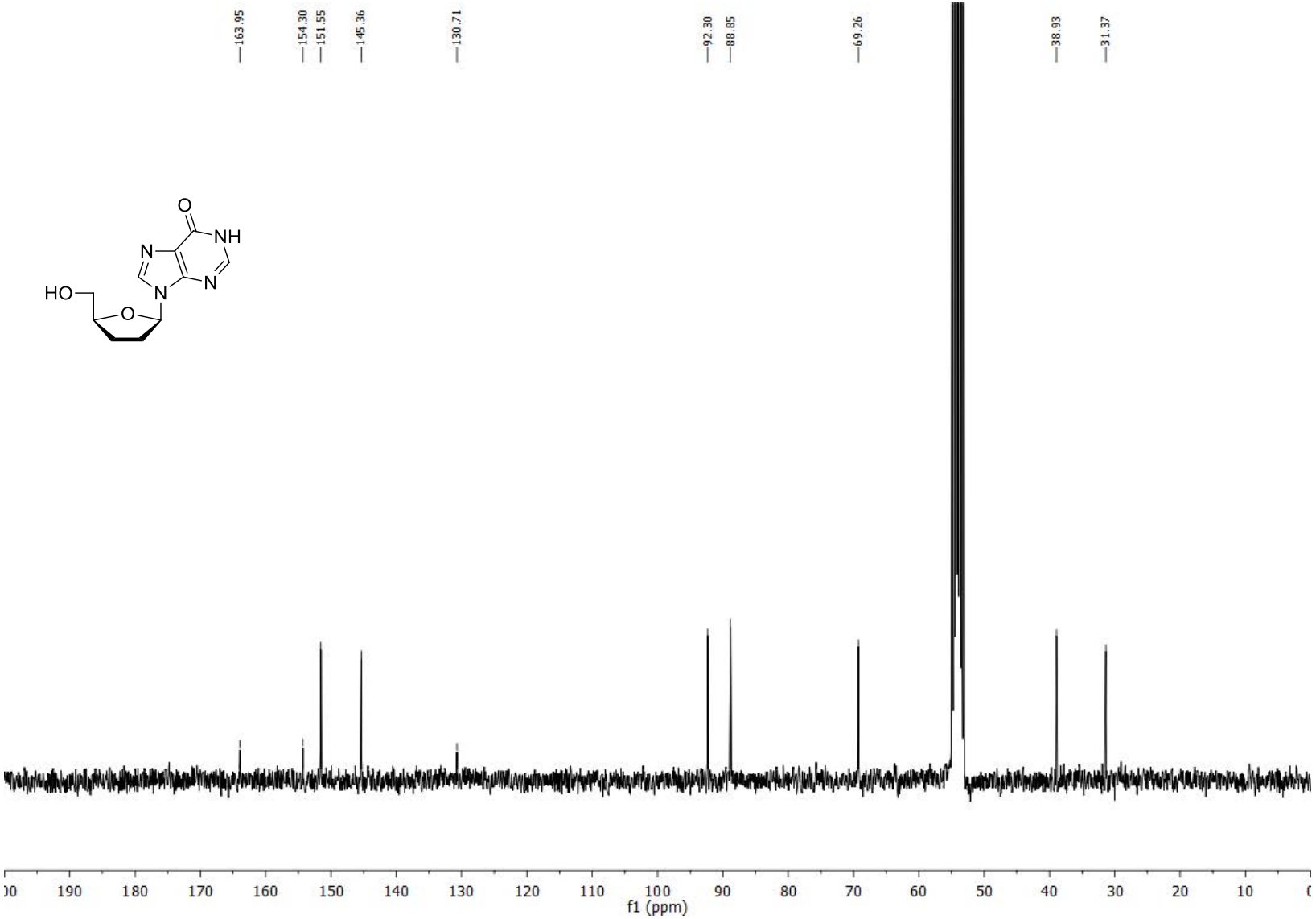
HMBC NMR (MeOH-d_4)



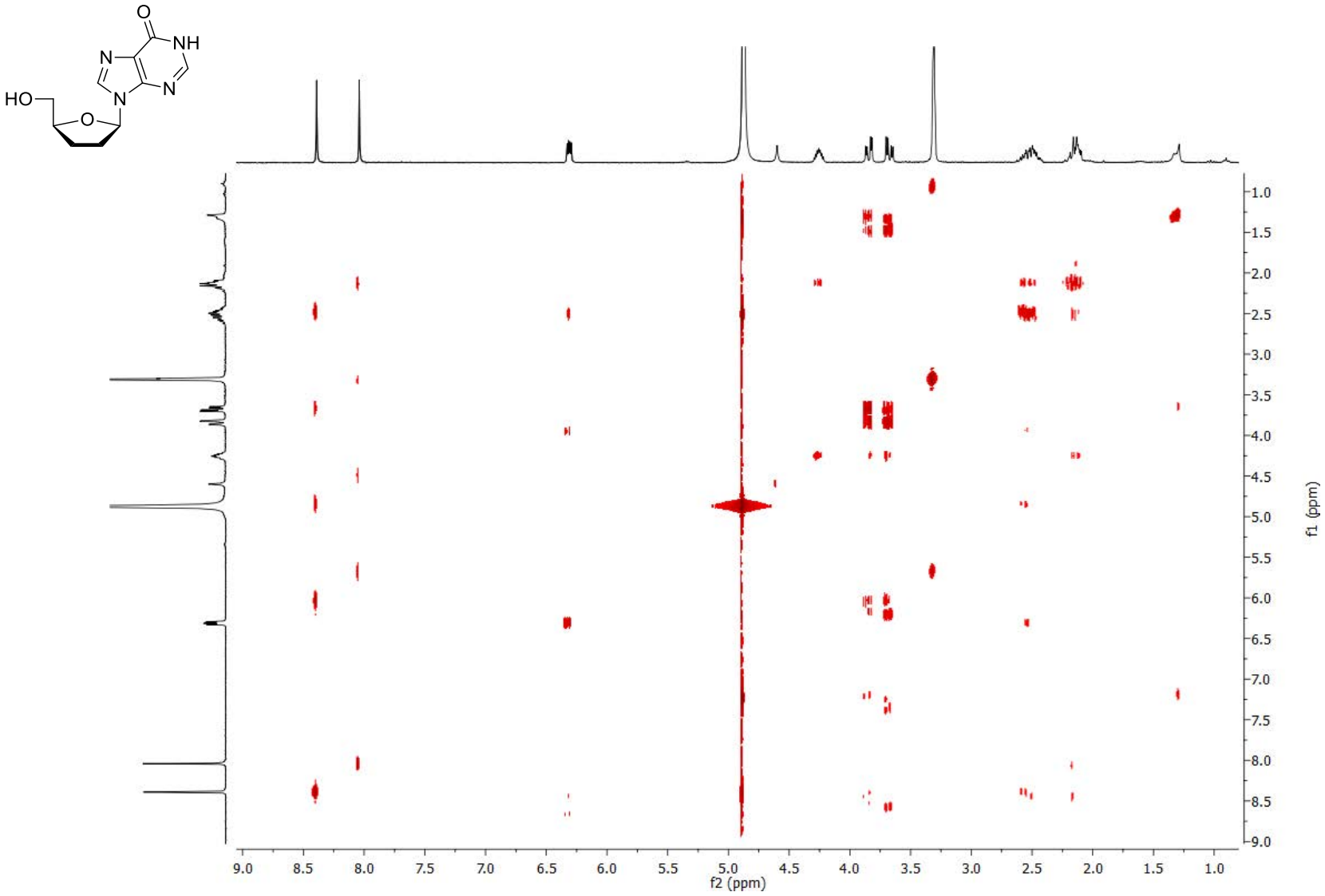
^{1}H-NMR (300.13 MHz, MeOH-d_4)



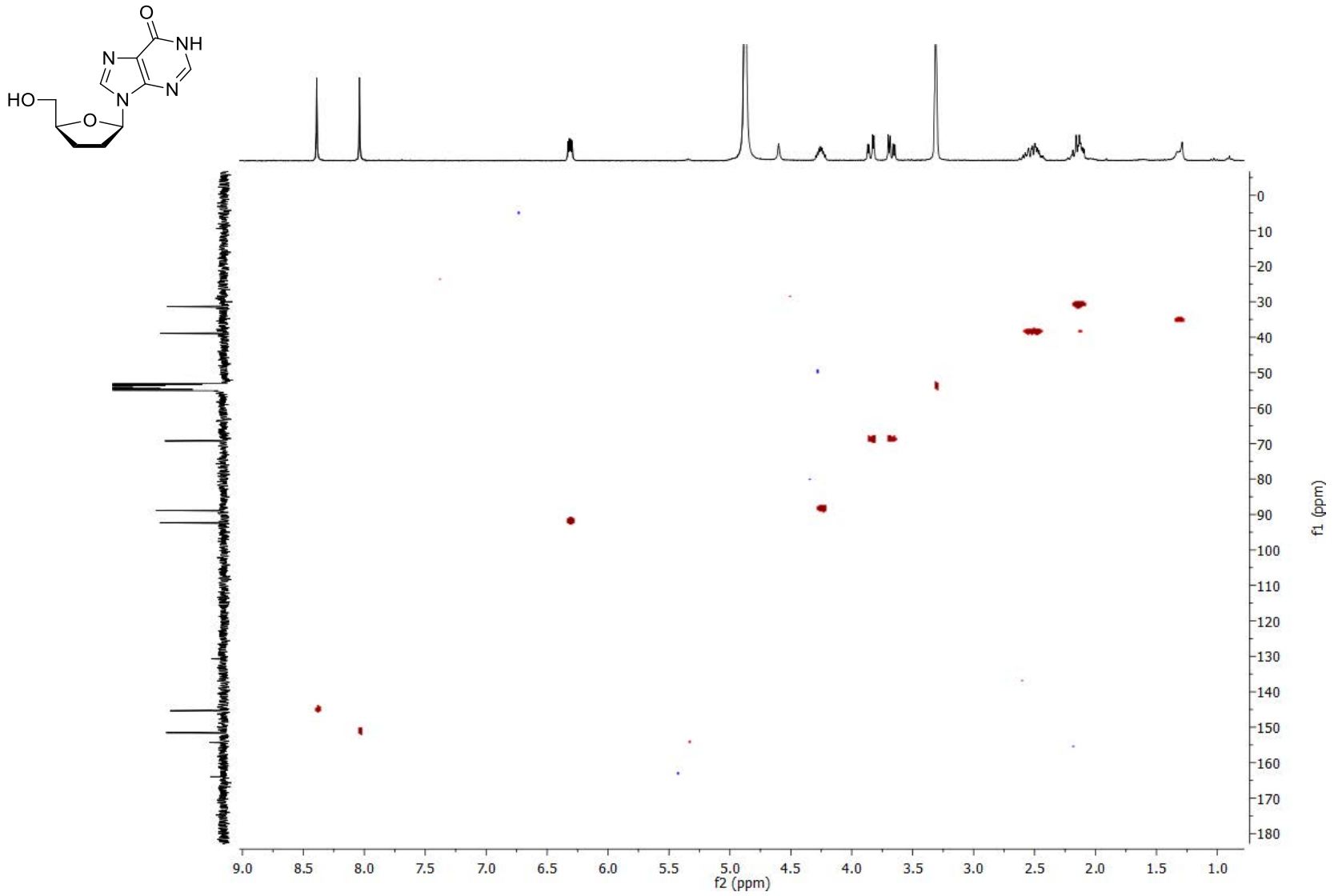
^{13}C-NMR (75.5 MHz, MeOH-d_4)



COSY NMR (MeOH-d_4)



HSQC NMR (MeOH-d_4)



HMBC NMR (MeOH-d_4)