

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

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**5'-O-Levulinyl- $\beta$ -D-uridine (**2a**).**  $^1\text{H-NMR}$  (300.13 MHz,  $\text{CDCl}_3$ ):  $\delta$  2.18 (s, 3H, *Me*-Lev), 2.58 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.2 Hz), 2.78 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.0 Hz), 4.19 (m, 1H,  $\text{H}_{3'}$ ), 4.27 (m, 2H,  $\text{H}_{2'} + \text{H}_{4'}$ ), 4.36 (m, 2H,  $\text{H}_{5'}$ ), 5.78 (d, 1H,  $\text{H}_5$ ,  $J$  = 8.1 Hz), 5.82 (d, 1H,  $\text{H}_1$ ,  $J$  = 3.6 Hz), 7.66 (d, 1H,  $\text{H}_6$ ,  $J$  = 8.1 Hz) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz,  $\text{CDCl}_3$ ):  $\delta$  28.0 ( $\text{CH}_2$ -Lev), 29.9 ( $\text{CH}_3$ -Lev), 38.0 ( $\text{CH}_2$ -Lev), 63.6 ( $\text{C}_{5'}$ ), 70.3 ( $\text{C}_{3'}$ ), 75.0 ( $\text{C}_2$ ), 82.3 ( $\text{C}_{4'}$ ), 90.6 ( $\text{C}_1'$ ), 102.7 ( $\text{C}_5$ ), 140.5 ( $\text{C}_6$ ), 151.4 ( $\text{C}_2$ ), 164.2 ( $\text{C}_4$ ), 172.8 (CO-Lev), 207.6 (CO-Lev) ppm.

**5'-O-Levulinyl- $\beta$ -D-5-methyluridine (**2b**).**  $^1\text{H-NMR}$  (300.13 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.88 (s, 3H, *Me*), 2.19 (s, 3H, *Me*-Lev), 2.59 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.3), 2.80 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.3), 4.19 (m, 1H,  $\text{H}_{3'}$ ), 4.23-4.37 (several m, 3H,  $\text{H}_{2'} + \text{H}_{4'} + \text{H}_{5'}$ ), 4.45 (dd, 1H,  $\text{H}_5$ ,  $J$  = 12.3, 3.7 Hz), 5.84 (d, 1H,  $\text{H}_1$ ,  $J$  = 3.7 Hz), 7.41 (s, 1H,  $\text{H}_6$ ) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz,  $\text{MeOH-}d_4$ ):  $\delta$  12.5 ( $\text{CH}_3$ ), 28.9 ( $\text{CH}_2$ -Lev), 29.6 ( $\text{CH}_3$ -Lev), 38.6 ( $\text{CH}_2$ -Lev), 64.8 ( $\text{C}_{5'}$ ), 71.3 ( $\text{C}_{3'}$ ), 74.9 ( $\text{C}_2$ ), 83.1 ( $\text{C}_4'$ ), 91.1 ( $\text{C}_1'$ ), 111.8 ( $\text{C}_5$ ), 138.0 ( $\text{C}_6$ ), 152.5 ( $\text{C}_2$ ), 166.3 ( $\text{C}_4$ ), 174.2 (CO-Lev), 209.5 (CO-Lev) ppm.

**5'-O-Levulinyl- $\beta$ -D-cytidine (**2c**).**  $^1\text{H-NMR}$  (300.13 MHz,  $\text{MeOH-}d_4$ ):  $\delta$  2.17 (s, 3H, *Me*-Lev), 2.59 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 5.9 Hz), 2.84 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.8 Hz), 4.08 (dd, 1H,  $\text{H}_{3'}$ ,  $J$  = 6.3, 5.3 Hz), 4.16 (m, 2H,  $\text{H}_{2'} + \text{H}_{4'}$ ), 4.33 (dd, 1H,  $\text{H}_5$ ,  $J$  = 12.3, 4.1 Hz), 4.40 (dd, 1H,  $\text{H}_5$ ,  $J$  = 12.4, 2.9 Hz), 5.84 (d, 1H,  $\text{H}_5$ ,  $J$  = 3.2 Hz), 5.95 (d, 1H,  $\text{H}_1$ ,  $J$  = 7.5 Hz), 7.81 (d, 1H,  $\text{H}_6$ ,  $J$  = 7.5 Hz) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz,  $\text{MeOH-}d_4$ ):  $\delta$  28.8 ( $\text{CH}_2$ -Lev), 29.6 ( $\text{CH}_3$ -Lev), 38.6 ( $\text{CH}_2$ -Lev), 64.5 ( $\text{C}_{5'}$ ), 71.0 ( $\text{C}_{3'}$ ), 75.9 ( $\text{C}_2$ ), 82.7 ( $\text{C}_4'$ ), 92.6 ( $\text{C}_1'$ ), 96.2 ( $\text{C}_5$ ), 142.5 ( $\text{C}_6$ ), 158.2 ( $\text{C}_2$ ), 167.6 ( $\text{C}_4$ ), 174.2 (CO-Lev), 209.5 (CO-Lev) ppm.

***N*<sup>4</sup>-Benzoyl-5'-O-levulinyl- $\beta$ -D-cytidine (**2d**).**  $^1\text{H-NMR}$  (300.13 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  2.12 (s, 3H, *Me*-Lev), 2.54 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.3 Hz), 2.75 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.3 Hz), 3.97 (m, 1H,  $\text{H}_{4'}$ ), 4.09 (m, 2H,  $\text{H}_{2'} + \text{H}_{3'}$ ), 4.25 (dd, 1H,  $\text{H}_5$ ,  $J$  = 12.2, 5.1 Hz), 4.35 (dd, 1H,  $\text{H}_5$ ,  $J$  = 12.1, 2.3 Hz), 5.82 (d, 1H,  $\text{H}_1$ ,  $J$  = 2.7 Hz), 7.38 (d, 1H,  $\text{H}_5$ ,  $J$  = 7.5 Hz), 7.51 (m, 2H,  $\text{H}_{\text{arom}}$ ), 7.63 (m, 1H,  $\text{H}_{\text{arom}}$ ), 8.01 (m, 2H,  $\text{H}_{\text{arom}}$ ), 8.16 (d, 1H,  $\text{H}_6$ ,  $J$  = 7.5 Hz) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  28.0 ( $\text{CH}_2$ -Lev), 30.0 ( $\text{CH}_3$ -Lev), 37.9 ( $\text{CH}_2$ -Lev), 63.9 ( $\text{C}_{5'}$ ), 69.7 ( $\text{C}_4'$ ), 74.3 ( $\text{C}_2$ ), 81.3 ( $\text{C}_{3'}$ ), 91.4 ( $\text{C}_1'$ ), 96.9 ( $\text{C}_5$ ), 128.9 (4 $\text{CH}_{\text{arom}}$ ), 133.2, 133.6 ( $\text{CH}_{\text{arom}} + \text{C}_{\text{arom}}$ ), 145.5 ( $\text{C}_6$ ), 154.9 ( $\text{C}_2$ ), 163.5 ( $\text{C}_4$ ), 168.0 ( $\text{C=O}$ ), 172.2 (CO-Lev), 207.4 (CO-Lev) ppm.

**5'-O-Levulinyl- $\beta$ -D-adenosine (**2e**).**  $^1\text{H-NMR}$  (300.13 MHz,  $\text{MeOH-}d_4$ ):  $\delta$  2.14 (s, 3H, *Me*-Lev), 2.56 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.4 Hz), 2.78 (m, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.4 Hz), 4.25 (q, 1H,  $\text{H}_{4'}$ ,  $J$  = 4.7 Hz), 4.37 (m, 3H,  $\text{H}_{3'} + \text{H}_{5'}$ ), 4.74 (t, 2H,  $\text{H}_{2'}$ ,  $J$  = 4.9 Hz), 6.03 (d, 1H,  $\text{H}_1$ ,  $J$  = 4.5 Hz), 8.21 (s, 1H,  $\text{H}_2$ ), 8.31 (s, 1H,  $\text{H}_8$ ) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz,  $\text{MeOH-}d_4$ ):  $\delta$  28.8 ( $\text{CH}_2$ -Lev), 29.6 ( $\text{CH}_3$ -Lev), 38.6 ( $\text{CH}_2$ -Lev), 64.8 ( $\text{C}_{5'}$ ), 71.8 ( $\text{C}_{3'}$ ), 75.2 ( $\text{C}_2$ ), 83.5 ( $\text{C}_4'$ ), 90.2 ( $\text{C}_1'$ ), 120.5 ( $\text{C}_5$ , cross peak with  $\text{H}_8$  in HMBC), 141.2 ( $\text{C}_8$ ), 150.6 ( $\text{C}_4$ ), 153.9 ( $\text{C}_2$ ), 157.3 ( $\text{C}_6$ ), 174.2 (CO-Lev), 209.5 (CO-Lev) ppm.

**5'-O-Levulinyl- $\beta$ -D-inosine (**2f**).**  $^1\text{H-NMR}$  (300.13 MHz,  $\text{MeOH-}d_4$ ):  $\delta$  2.15 (s, 3H, *Me*-Lev), 2.57 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.4 Hz), 2.80 (t, 2H,  $\text{CH}_2$ -Lev,  $J$  = 6.4 Hz), 4.25 (q, 1H,  $\text{H}_{4'}$ ,  $J$  = 4.3 Hz), 4.37 (m, 3H,  $\text{H}_{3'} + \text{H}_{5'}$ ), 4.70 (t, 2H,  $\text{H}_{2'}$ ,  $J$  = 4.8 Hz), 6.03 (d, 1H,  $\text{H}_1$ ,  $J$  = 4.6 Hz), 8.07 (s, 1H,  $\text{H}_2$ ), 8.27 (s, 1H,  $\text{H}_8$ ) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz,  $\text{MeOH-}d_4$ ):  $\delta$  28.8 ( $\text{CH}_2$ -Lev), 29.6 ( $\text{CH}_3$ -Lev), 38.6 ( $\text{CH}_2$ -Lev), 64.8 ( $\text{C}_{5'}$ ), 71.7 ( $\text{C}_{3'}$ ), 75.5 ( $\text{C}_2$ ), 83.7 ( $\text{C}_4'$ ), 90.4 ( $\text{C}_1'$ ), 125.9 ( $\text{C}_5$ ), 140.7 ( $\text{C}_8$ ), 146.8 ( $\text{C}_2$ ), 149.9 ( $\text{C}_4$ , cross peak with  $\text{H}_1$  in HMBC), 158.9 ( $\text{C}_6$ ), 174.2 (CO-Lev), 209.5 (CO-Lev) ppm.

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**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -D-uridine (**5a**).**  $^1\text{H-NMR}$  (300.13 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.10 (s, 6H, *Si-Me*), 0.91 (s, 9H, *Si-tBu*), 3.83 (dd, 1H,  $\text{H}_{5'}$ ,  $J$  = 11.7, 1.5 Hz), 4.01 (dd, 1H,  $\text{H}_5$ ,  $J$  = 11.8, 1.8 Hz), 4.12 (m, 1H,  $\text{H}_{4'}$ ), 4.23 (m, 2H,  $\text{H}_{2'} + \text{H}_{3'}$ ), 5.64 (d, 1H,  $\text{H}_5$ ,  $J$  = 8.1 Hz), 5.90 (d, 1H,  $\text{H}_1$ ,  $J$  = 2.2 Hz), 8.05 (d, 1H,  $\text{H}_6$ ,  $J$  = 8.1 Hz) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz,  $\text{CDCl}_3$ ):  $\delta$  -5.4 (*Si-CH*<sub>3</sub>), 18.6 (*SiCMe*<sub>3</sub>), 26.1 (*tBu-CH*<sub>3</sub>), 62.0 ( $\text{C}_{5'}$ ), 69.5 ( $\text{C}_2$ ), 75.8 ( $\text{C}_{3'}$ ), 85.1 ( $\text{C}_4'$ ), 90.3 ( $\text{C}_1'$ ), 102.3 ( $\text{C}_5$ ), 140.6 ( $\text{C}_6$ ), 151.5 ( $\text{C}_2$ ), 164.1 ( $\text{C}_4$ ) ppm.

**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -D-5-methyluridine (**5b**).**  $^1\text{H-NMR}$  (300.13 MHz,  $\text{MeOH-}d_4$ ):  $\delta$  0.15 (s, 3H, *Si-Me*), 0.16 (s, 3H, *Si-Me*), 0.97 (s, 9H, *Si-tBu*), 1.88 (d, 3H, *Me*,  $J$  = 1.1 Hz), 3.85 (dd, 1H,  $\text{H}_5$ ,  $J$  = 11.6, 2.7 Hz), 3.94 (dd, 1H,  $\text{H}_5$ ,  $J$  = 11.6, 2.3 Hz), 4.02 (q, 1H,  $\text{H}_{4'}$ ,  $J$  = 2.6 Hz), 4.13 (m, 2H,  $\text{H}_{2'} + \text{H}_{3'}$ ), 5.95 (d, 1H,  $\text{H}_1$ ,  $J$  = 5.0 Hz), 7.58 (d, 1H,  $\text{H}_6$ ,  $J$  = 1.1 Hz) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz,  $\text{MeOH-}d_4$ ):  $\delta$  -5.2 (*Si-CH*<sub>3</sub>), 12.6 ( $\text{CH}_3$ ), 19.4 (*SiCMe*<sub>3</sub>), 26.5 (*tBu-CH*<sub>3</sub>), 64.3 ( $\text{C}_{5'}$ ), 71.7 ( $\text{C}_2$ ), 75.7 ( $\text{C}_{3'}$ ), 86.4 ( $\text{C}_4'$ ), 89.6 ( $\text{C}_1'$ ), 111.6 ( $\text{C}_5$ ), 137.4 ( $\text{C}_6$ ), 152.6 ( $\text{C}_2$ ), 166.2 ( $\text{C}_4$ ) ppm.

**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -d-cytidine (**5c**).**  $^1\text{H-NMR}$  (300.13 MHz, MeOH-*d*<sub>4</sub>):  $\delta$  0.15 (s, 6H, Si-Me), 0.96 (s, 9H, Si-*t*Bu), 3.87 (dd, 1H, H<sub>5'</sub>,  $J$  = 12.0, 2.1 Hz), 4.02-4.16 (several m, 4H, H<sub>2'</sub> + H<sub>3'</sub> + H<sub>4'</sub> + H<sub>5'</sub>), 5.85 (d, 1H, H<sub>5'</sub>,  $J$  = 7.6 Hz), 5.88 (d, 1H, H<sub>1'</sub>,  $J$  = 2.7 Hz), 8.11 (d, 1H, H<sub>6'</sub>,  $J$  = 7.5 Hz) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz, MeOH-*d*<sub>4</sub>):  $\delta$  -5.39 (Si-CH<sub>3</sub>), -5.36 (Si-CH<sub>3</sub>), 19.3 (SiCMe<sub>3</sub>), 26.5 (*t*Bu-CH<sub>3</sub>), 63.0 (C<sub>5'</sub>), 70.1 (C<sub>2'</sub>), 76.8 (C<sub>3'</sub>), 85.3 (C<sub>4'</sub>), 91.8 (C<sub>1'</sub>), 95.71 (C<sub>5'</sub>), 142.4 (C<sub>6'</sub>), 158.4 (C<sub>2'</sub>), 167.6 (C<sub>4'</sub>) ppm.

**N<sup>4</sup>-Benzoyl-5'-O-(*tert*-butyldimethylsilyl)- $\beta$ -d-cytidine (**5d**).**  $^1\text{H-NMR}$  (300.13 MHz, MeOH-*d*<sub>4</sub>):  $\delta$  0.17 (s, 3H, Si-Me), 0.19 (s, 3H, Si-Me), 0.99 (s, 9H, Si-*t*Bu), 3.91 (dd, 1H, H<sub>5'</sub>,  $J$  = 12.2, 2.1 Hz), 4.15 (several m, 4H, H<sub>2'</sub> + H<sub>3'</sub> + H<sub>4'</sub> + H<sub>5'</sub>), 5.90 (d, 1H, H<sub>1'</sub>,  $J$  = 1.5 Hz), 7.58 (m, 4H, H<sub>5</sub> + H<sub>arom</sub>), 7.98 (m, 2H, H<sub>arom</sub>), 8.64 (d, 1H, H<sub>6'</sub>,  $J$  = 7.5 Hz) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz, MeOH-*d*<sub>4</sub>):  $\delta$  -5.3 (Si-CH<sub>3</sub>), 19.4 (SiCMe<sub>3</sub>), 26.5 (*t*Bu-CH<sub>3</sub>), 62.6 (C<sub>5'</sub>), 69.5 (C<sub>2'</sub>), 76.8 (C<sub>3'</sub>), 85.5 (C<sub>4'</sub>), 92.9 (C<sub>1'</sub>), 98.1 (C<sub>5'</sub>), 129.2 (CH<sub>arom</sub>), 129.8 (CH<sub>arom</sub>), 134.1 (CH<sub>arom</sub>), 134.7 (CH<sub>arom</sub>), 146.3 (C<sub>6'</sub>), 157.9 (C<sub>2'</sub>), 164.8 (C<sub>4'</sub>), 169.2 (C=O) ppm.

**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -d-adenosine (**5e**).**  $^1\text{H-NMR}$  (300.13 MHz, MeOH-*d*<sub>4</sub>):  $\delta$  0.11 (s, 6H, Si-Me), 0.92 (s, 9H, Si-*t*Bu), 3.87 (dd, 1H, H<sub>5'</sub>,  $J$  = 11.6, 3.0 Hz), 4.01 (dd, 1H, H<sub>5'</sub>,  $J$  = 11.6, 3.0 Hz), 4.13 (m, 1H, H<sub>4'</sub>), 4.36 (t, 1H, H<sub>3'</sub>,  $J$  = 5.0 Hz), 4.56 (t, 1H, H<sub>2'</sub>,  $J$  = 4.6 Hz), 6.05 (d, 1H, H<sub>1'</sub>,  $J$  = 4.2 Hz), 8.20 (s, 1H, H<sub>2'</sub>), 8.38 (s, 1H, H<sub>8'</sub>) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz, MeOH-*d*<sub>4</sub>):  $\delta$  -5.33 (Si-CH<sub>3</sub>), -5.31 (Si-CH<sub>3</sub>), 19.3 (SiCMe<sub>3</sub>), 26.5 (*t*Bu-CH<sub>3</sub>), 63.8 (C<sub>5'</sub>), 71.3 (C<sub>3'</sub>), 76.3 (C<sub>2'</sub>), 86.3 (C<sub>4'</sub>), 90.1 (C<sub>1'</sub>), 120.4 (C<sub>5'</sub>), 140.7 (C<sub>8'</sub>), 150.5 (C<sub>4'</sub>), 153.9 (C<sub>2'</sub>), 157.3 (C<sub>6'</sub>) ppm.

**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -d-inosine (**5f**).**  $^1\text{H-NMR}$  (300.13 MHz, MeOH-*d*<sub>4</sub>):  $\delta$  0.10 (s, 6H, Si-Me), 0.91 (s, 9H, Si-*t*Bu), 3.86 (dd, 1H, H<sub>5'</sub>,  $J$  = 11.6, 3.1 Hz), 3.99 (dd, 1H, H<sub>5'</sub>,  $J$  = 11.6, 3.0 Hz), 4.14 (m, 1H, H<sub>4'</sub>), 4.36 (t, 1H, H<sub>3'</sub>,  $J$  = 4.8 Hz), 4.56 (t, 1H, H<sub>2'</sub>,  $J$  = 4.8 Hz), 6.07 (d, 1H, H<sub>1'</sub>,  $J$  = 4.5 Hz), 8.07 (s, 1H, H<sub>2'</sub>), 8.31 (s, 1H, H<sub>8'</sub>) ppm.  $^{13}\text{C-NMR}$  (75.5 MHz, MeOH-*d*<sub>4</sub>):  $\delta$  -5.33 (Si-CH<sub>3</sub>), -5.29 (Si-CH<sub>3</sub>), 19.3 (SiCMe<sub>3</sub>), 26.5 (*t*Bu-CH<sub>3</sub>), 63.9 (C<sub>5'</sub>), 71.5 (C<sub>3'</sub>), 76.6 (C<sub>2'</sub>), 86.6 (C<sub>4'</sub>), 90.0 (C<sub>1'</sub>), 125.6 (C<sub>5'</sub>), 140.1 (C<sub>8'</sub>), 146.8 (C<sub>2'</sub>), 149.9 (C<sub>4'</sub>), 158.9 (C<sub>6'</sub>) ppm.

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

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

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

**N<sup>4</sup>-Benzoyl-5'-O-(*tert*-butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -d-cytidine (**6d**).**  $^1\text{H-NMR}$  (300.13 MHz, CDCl<sub>3</sub>):  $\delta$  0.18 (s, 6H, Si-Me), 0.97 (s, 9H, Si-*t*Bu), 1.34 (t, 3H, SCH<sub>2</sub>-Me,  $J$  = 6.1 Hz), 1.37 (t, 3H,

$\text{SCH}_2\text{-Me}$ ,  $J = 6.1$  Hz), 3.14 (m, 4H, S- $\text{CH}_2$ ), 3.98 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.5, 1.8$  Hz), 4.05 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.5, 1.6$  Hz), 4.49 (m, 1H,  $\text{H}_{4'}$ ), 6.15 (dd, 1H,  $\text{H}_{2'}$ ,  $J = 6.8, 5.6$  Hz), 6.27 (dd, 1H,  $\text{H}_{3'}$ ,  $J = 5.5, 1.8$  Hz), 6.74 (d, 1H,  $\text{H}_{1'}$ ,  $J = 6.9$  Hz), 7.50 (m, 3H,  $\text{H}_5 + \text{H}_{\text{arom}}$ ), 7.61 (m, 1H,  $\text{H}_{\text{arom}}$ ), 7.91 (m, 2H,  $\text{H}_{\text{arom}}$ ), 8.30 (d, 1H,  $\text{H}_6$ ,  $J = 7.6$  Hz), 8.69 (br s, 1H, NH) ppm.  $^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ ):  $\delta$  -5.4 (Si- $\text{CH}_3$ ), -5.2 (Si- $\text{CH}_3$ ), 13.3 ( $\text{SCH}_2\text{-CH}_3$ ), 13.5 ( $\text{SCH}_2\text{-CH}_3$ ), 18.5 (SiCMe<sub>3</sub>), 26.1 ( $t\text{Bu-CH}_3$ ), 30.65 (S- $\text{CH}_2$ ), 30.74 (S- $\text{CH}_2$ ), 63.2 ( $\text{C}_{5'}$ ), 78.8 ( $\text{C}_{3'}$ ), 80.7 ( $\text{C}_{2'}$ ), 84.6 ( $\text{C}_{4'}$ ), 86.5 ( $\text{C}_{1'}$ ), 97.8 ( $\text{C}_5$ ), 127.8 ( $\text{CH}_{\text{arom}}$ ), 129.1 ( $\text{CH}_{\text{arom}}$ ), 133.2 ( $\text{C}_{\text{arom}}$ ), 133.3 ( $\text{CH}_{\text{arom}}$ ), 144.4 ( $\text{C}_6$ ), 154.6 ( $\text{C}_2$ ), 162.5 ( $\text{C}_4$ ), 167.1 ( $\text{C=O}$ ), 213.6 ( $\text{C=S}$ ), 214.0 ( $\text{C=S}$ ) ppm.

5'-O-(*tert*-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -d-adenosine (**6e**).  $^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.16 (s, 6H, Si-Me), 0.95 (s, 9H, Si- $t\text{Bu}$ ), 1.29 (t, 3H,  $\text{SCH}_2\text{-Me}$ ,  $J = 7.4$  Hz), 1.37 (t, 3H,  $\text{SCH}_2\text{-Me}$ ,  $J = 7.4$  Hz), 3.07 (q, 2H, S- $\text{CH}_2$ ,  $J = 7.4$  Hz), 3.17 (q, 2H, S- $\text{CH}_2$ ,  $J = 7.3$  Hz), 3.95 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.4, 2.1$  Hz), 4.05 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.3, 2.1$  Hz), 4.52 (m, 1H,  $\text{H}_{4'}$ ), 6.14 (br s, 2H, NH<sub>2</sub>), 6.41 (dd, 1H,  $\text{H}_{3'}$ ,  $J = 5.4, 1.9$  Hz), 6.48 (dd, 1H,  $\text{H}_{2'}$ ,  $J = 6.8, 5.5$  Hz), 6.60 (d, 1H,  $\text{H}_{1'}$ ,  $J = 6.9$  Hz), 8.22 (s, 1H, H<sub>8</sub>), 8.35 (s, 1H, H<sub>2</sub>) ppm.  $^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ ):  $\delta$  -5.3 (Si- $\text{CH}_3$ ), -5.1 (Si- $\text{CH}_3$ ), 13.3 ( $\text{SCH}_2\text{-CH}_3$ ), 18.6 (SiCMe<sub>3</sub>), 26.2 ( $t\text{Bu-CH}_3$ ), 30.8 (S- $\text{CH}_2$ ), 63.4 ( $\text{C}_{5'}$ ), 78.8 ( $\text{C}_{3'}$ ), 80.7 ( $\text{C}_{2'}$ ), 84.7 ( $\text{C}_{1'} + \text{C}_{4'}$ ), 119.6 ( $\text{C}_5$ ), 138.5 ( $\text{C}_8$ ), 150.3 ( $\text{C}_4$ ), 153.4 ( $\text{C}_2$ ), 155.7 ( $\text{C}_6$ ), 213.6 ( $\text{C=S}$ ), 214.0 ( $\text{C=S}$ ) ppm.

5'-O-(*tert*-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -d-inosine (**6f**).  $^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.17 (s, 6H, Si-Me), 0.96 (s, 9H, Si- $t\text{Bu}$ ), 1.31 (t, 3H,  $\text{SCH}_2\text{-Me}$ ,  $J = 7.4$  Hz), 1.39 (t, 3H,  $\text{SCH}_2\text{-Me}$ ,  $J = 7.4$  Hz), 3.09 (q, 2H, S- $\text{CH}_2$ ,  $J = 7.4$  Hz), 3.18 (q, 2H, S- $\text{CH}_2$ ,  $J = 7.4$  Hz), 3.97 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.4, 2.0$  Hz), 4.06 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.4, 2.0$  Hz), 4.55 (m, 1H,  $\text{H}_{4'}$ ), 6.40 (dd, 1H,  $\text{H}_{3'}$ ,  $J = 5.3, 2.0$  Hz), 6.46 (m, 1H, H<sub>2</sub>), 6.53 (d, 1H,  $\text{H}_{1'}$ ,  $J = 6.7$  Hz), 8.20 (s, 1H, H<sub>2</sub>), 8.24 (s, 1H, H<sub>8</sub>), 13.10 (br s, 1H, NH) ppm.  $^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ ):  $\delta$  -5.3 (Si- $\text{CH}_3$ ), -5.1 (Si- $\text{CH}_3$ ), 13.3 ( $\text{SCH}_2\text{-CH}_3$ ), 13.4 ( $\text{SCH}_2\text{-CH}_3$ ), 18.6 (SiCMe<sub>3</sub>), 26.2 ( $t\text{Bu-CH}_3$ ), 30.86 (S- $\text{CH}_2$ ), 30.93 (S- $\text{CH}_2$ ), 63.3 ( $\text{C}_{5'}$ ), 78.7 ( $\text{C}_{3'}$ ), 80.9 ( $\text{C}_{2'}$ ), 84.9 ( $\text{C}_{1'}$ ), 85.2 ( $\text{C}_{5'}$ ), 124.9 ( $\text{C}_5$ ), 138.3 ( $\text{C}_8$ ), 145.6 ( $\text{C}_2$ ), 149.3 ( $\text{C}_4$ ), 159.3 ( $\text{C}_6$ ), 213.4 ( $\text{C=S}$ ), 214.0 ( $\text{C=S}$ ) ppm.

5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -d-uridine (**7a**).  $^1\text{H}$ -NMR (300.13 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  0.03 (s, 6H, Si-Me), 0.85 (s, 9H, Si- $t\text{Bu}$ ), 3.75 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.7, 3.7$  Hz), 3.81 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.7, 3.0$  Hz), 4.82 (m, 1H,  $\text{H}_{4'}$ ), 5.52 (d, 1H,  $\text{H}_5$ ,  $J = 8.0$  Hz), 5.95 (dt, 1H, H<sub>2'</sub>,  $J = 6.0, 1.8$  Hz), 6.39 (dt, 1H,  $\text{H}_{3'}$ ,  $J = 6.0, 1.6$  Hz), 6.80 (m, 1H,  $\text{H}_{1'}$ ), 7.63 (d, 1H,  $\text{H}_6$ ,  $J = 8.0$  Hz), 11.35 (br s, 1H, NH) ppm.  $^{13}\text{C}$ -NMR (75.5 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  -5.43 (Si- $\text{CH}_3$ ), -5.38 (Si- $\text{CH}_3$ ), 18.2 (SiCMe<sub>3</sub>), 25.8 ( $t\text{Bu-CH}_3$ ), 64.3 ( $\text{C}_{5'}$ ), 86.9 ( $\text{C}_{4'}$ ), 89.1 ( $\text{C}_{1'}$ ), 101.6 ( $\text{C}_5$ ), 126.2 ( $\text{C}_{2'}$ ), 134.5 ( $\text{C}_{3'}$ ), 140.8 ( $\text{C}_6$ ), 150.7 ( $\text{C}_2$ ), 163.1 ( $\text{C}_4$ ) ppm.

5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-3'-deoxy- $\beta$ -d-5-thymidine (**7b**).  $^1\text{H}$ -NMR (300.13 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  0.03 (s, 6H, Si-Me), 0.85 (s, 9H, Si- $t\text{Bu}$ ), 1.75 (d, 3H, Me,  $J = 0.9$  Hz), 3.72 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.5, 4.6$  Hz), 3.80 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.6, 3.2$  Hz), 4.78 (m, 1H,  $\text{H}_{4'}$ ), 5.96 (dt, 1H, H<sub>2'</sub>,  $J = 6.0, 2.0$  Hz), 6.38 (dt, 1H,  $\text{H}_{3'}$ ,  $J = 6.0, 1.6$  Hz), 6.78 (m, 1H,  $\text{H}_{1'}$ ), 7.28 (d, 1H,  $\text{H}_6$ ,  $J = 1.2$  Hz), 11.36 (br s, 1H, NH) ppm.  $^{13}\text{C}$ -NMR (75.5 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  -5.3 (Si- $\text{CH}_3$ ), -5.2 (Si- $\text{CH}_3$ ), 12.2 (CH<sub>3</sub>), 18.3 (SiCMe<sub>3</sub>), 25.9 ( $t\text{Bu-CH}_3$ ), 64.7 ( $\text{C}_{5'}$ ), 86.7 ( $\text{C}_{4'}$ ), 89.2 ( $\text{C}_{1'}$ ), 109.4 ( $\text{C}_5$ ), 126.2 ( $\text{C}_{2'}$ ), 134.4 ( $\text{C}_{3'}$ ), 135.9 ( $\text{C}_6$ ), 150.8 ( $\text{C}_2$ ), 163.8 ( $\text{C}_4$ ) ppm.

5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -d-cytidine (**7c**).  $^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.066 (s, 3H, Si-Me), 0.070 (s, 3H, Si-Me), 0.90 (s, 9H, Si- $t\text{Bu}$ ), 3.83 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.6, 2.8$  Hz), 3.92 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.5, 3.0$  Hz), 4.90 (m, 1H,  $\text{H}_{4'}$ ), 5.62 (d, 1H,  $\text{H}_5$ ,  $J = 7.4$  Hz), 5.91 (m, 1H, H<sub>2'</sub>), 6.14 (dt, 1H,  $\text{H}_{3'}$ ,  $J = 6.0, 1.6$  Hz), 7.07 (m, 1H,  $\text{H}_{1'}$ ), 7.94 (d, 1H,  $\text{H}_6$ ,  $J = 7.4$  Hz) ppm.  $^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ ):  $\delta$  -5.3 (Si- $\text{CH}_3$ ), -5.2 (Si- $\text{CH}_3$ ), 18.6 (SiCMe<sub>3</sub>), 26.1 ( $t\text{Bu-CH}_3$ ), 64.5 ( $\text{C}_{5'}$ ), 87.3 ( $\text{C}_{4'}$ ), 91.0 ( $\text{C}_{1'}$ ), 94.0 ( $\text{C}_5$ ), 128.2 ( $\text{C}_{2'}$ ), 132.7 ( $\text{C}_{3'}$ ), 142.8 ( $\text{C}_6$ ), 156.3 ( $\text{C}_2$ ), 165.6 ( $\text{C}_4$ ) ppm.

*N*<sup>4</sup>-Benzoyl-5'-O-(*tert*-butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -d-cytidine (**7d**).  $^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.08 (s, 3H, Si-Me), 0.10 (s, 3H, Si-Me), 0.90 (s, 9H, Si- $t\text{Bu}$ ), 3.86 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.7, 2.6$  Hz), 3.99 (dd, 1H,  $\text{H}_{5'}$ ,  $J = 11.7, 2.7$  Hz), 4.99 (m, 1H,  $\text{H}_{4'}$ ), 6.02 (d, 1H, H<sub>2'</sub>,  $J = 5.7$  Hz), 6.18 (dt, 1H,  $\text{H}_{3'}$ ,  $J = 6.0, 1.4$  Hz), 7.03 (m, 1H,  $\text{H}_{1'}$ ), 7.49 (m, 3H,  $\text{H}_5 + \text{H}_{\text{arom}}$ ), 7.60 (m, 1H,  $\text{H}_{\text{arom}}$ ), 7.93 (d, 2H,  $\text{H}_{\text{arom}}$ ,  $J = 7.3$  Hz), 8.40 (d, 1H,  $\text{H}_6$ ,  $J = 7.5$  Hz), 8.71 (br s, 1H, NH) ppm.  $^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ ):  $\delta$  -5.4 (Si- $\text{CH}_3$ ), -5.2 (Si- $\text{CH}_3$ ), 18.6

(SiCMe<sub>3</sub>), 26.0 (<sup>t</sup>Bu-CH<sub>3</sub>), 64.2 (C<sub>5'</sub>), 88.0 (C<sub>4'</sub>), 91.8 (C<sub>1'</sub>), 96.9 (C<sub>5</sub>), 127.67 (C<sub>2'</sub>), 127.71 (CH<sub>arom</sub>), 129.1 (CH<sub>arom</sub>), 133.0 (C<sub>3'</sub>), 133.2 (CH<sub>arom</sub>), 133.4 (C<sub>arom</sub>), 145.7 (C<sub>6</sub>), 155.1 (C<sub>2</sub>), 162.3 (C<sub>4</sub>), 167.3 (C=O) ppm.

**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy-β-d-adenosine (7e).** <sup>1</sup>H-NMR (300.13 MHz, CDCl<sub>3</sub>): δ 0.03 (s, 3H, Si-Me), 0.04 (s, 3H, Si-Me), 0.87 (s, 9H, Si-<sup>t</sup>Bu), 3.81 (d, 2H, H<sub>5'</sub>, J = 3.8 Hz), 4.97 (m, 1H, H<sub>4'</sub>), 5.95 (br s, 2H, NH<sub>2</sub>), 6.03 (dt, 1H, H<sub>2'</sub>, J = 6.0, 1.9 Hz), 6.38 (dt, 1H, H<sub>3'</sub>, J = 6.0, 1.6 Hz), 7.09 (m, 1H, H<sub>1'</sub>), 8.09 (s, 1H, H<sub>8</sub>), 8.34 (s, 1H, H<sub>2</sub>) ppm. <sup>13</sup>C-NMR (75.5 MHz, CDCl<sub>3</sub>): δ -5.3 (Si-CH<sub>3</sub>), -5.2 (Si-CH<sub>3</sub>), 18.7 (SiCMe<sub>3</sub>), 26.1 (<sup>t</sup>Bu-CH<sub>3</sub>), 64.9 (C<sub>5'</sub>), 87.9 (C<sub>4'</sub>), 88.4 (C<sub>1'</sub>), 119.2 (C<sub>5</sub>), 125.7 (C<sub>2'</sub>), 134.7 (C<sub>3'</sub>), 139.4 (C<sub>8</sub>), 149.7 (C<sub>4</sub>), 152.9 (C<sub>2</sub>), 155.7 (C<sub>6</sub>) ppm.

**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy-β-d-inosine (7f).** <sup>1</sup>H-NMR (300.13 MHz, CDCl<sub>3</sub>): δ 0.03 (s, 3H, Si-Me), 0.04 (s, 3H, Si-Me), 0.87 (s, 9H, Si-<sup>t</sup>Bu), 3.82 (m, 2H, H<sub>5'</sub>), 4.99 (m, 1H, H<sub>4'</sub>), 6.05 (dt, 1H, H<sub>2'</sub>, J = 5.9, 1.8 Hz), 6.42 (dt, 1H, H<sub>3'</sub>, J = 5.8, 1.7 Hz), 7.04 (m, 1H, H<sub>1'</sub>), 8.09 (s, 1H, H<sub>8</sub>), 8.23 (s, 1H, H<sub>2</sub>), 13.23 (br s, 1H, NH) ppm. <sup>13</sup>C-NMR (75.5 MHz, CDCl<sub>3</sub>): δ -5.3 (Si-CH<sub>3</sub>), -5.2 (Si-CH<sub>3</sub>), 18.7 (SiCMe<sub>3</sub>), 26.1 (<sup>t</sup>Bu-CH<sub>3</sub>), 64.9 (C<sub>5'</sub>), 88.1 (C<sub>4'</sub>), 88.6 (C<sub>1'</sub>), 124.7 (C<sub>5</sub>), 125.3 (C<sub>2'</sub>), 135.0 (C<sub>3'</sub>), 139.1 (C<sub>8</sub>), 145.3 (C<sub>2</sub>), 149.0 (C<sub>4</sub>), 159.5 (C<sub>6</sub>) ppm.

**2',3'-Didehydro-2',3'-dideoxy-β-d-uridine (8a).** <sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>): δ 3.73 (m, 2H, H<sub>5'</sub>), 4.87 (m, 1H, H<sub>4'</sub>), 5.65 (d, 1H, H<sub>5</sub>, J = 8.1 Hz), 5.92 (m, 1H, H<sub>2'</sub>), 6.41 (dt, 1H, H<sub>3'</sub>, J = 6.0, 1.7 Hz), 6.94 (dt, 1H, H<sub>1'</sub>, J = 3.1, 1.5 Hz), 7.89 (d, 1H, H<sub>6</sub>, J = 8.1 Hz) ppm. <sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>): δ 63.8 (C<sub>5'</sub>), 89.1 (C<sub>4'</sub>), 91.3 (C<sub>1'</sub>), 102.5 (C<sub>5</sub>), 127.0 (C<sub>2'</sub>), 136.1 (C<sub>3'</sub>), 143.1 (C<sub>6</sub>), 152.7 (C<sub>2</sub>), 166.3 (C<sub>4</sub>) ppm.

**2',3'-Didehydro-3'-deoxy-β-d-5-thymidine (8b).** <sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>): δ 1.84 (d, 3H, Me, J = 1.1 Hz), 3.76 (m, 2H, H<sub>5'</sub>), 4.86 (debajo de H<sub>2</sub>O, 1H, H<sub>4'</sub>), 5.90 (dt, 1H, H<sub>2'</sub>, J = 6.0, 2.0 Hz), 6.40 (dt, 1H, H<sub>3'</sub>, J = 6.0, 1.7 Hz), 6.95 (dt, 1H, H<sub>1'</sub>, J = 3.4, 1.7 Hz), 7.75 (d, 1H, H<sub>6</sub>, J = 1.2 Hz) ppm. <sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>): δ 12.4 (CH<sub>3</sub>), 63.8 (C<sub>5'</sub>), 89.0 (C<sub>4'</sub>), 91.0 (C<sub>1'</sub>), 111.2 (C<sub>5</sub>), 127.3 (C<sub>2</sub>), 135.9 (C<sub>3</sub>), 138.9 (C<sub>6</sub>), 152.9 (C<sub>2</sub>), 166.6 (C<sub>4</sub>) ppm.

**N<sup>4</sup>-Benzoyl-2',3'-didehydro-2',3'-dideoxy-β-d-cytidine (8d).** <sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>): δ 3.78 (m, 2H, H<sub>5'</sub>), 4.99 (m, 1H, H<sub>4'</sub>), 6.05 (m, 1H, H<sub>2'</sub>), 6.41 (dt, 1H, H<sub>3'</sub>, J = 6.0, 1.6 Hz), 7.00 (dt, 1H, H<sub>1'</sub>, J = 2.9, 1.4 Hz), 7.54 (m, 3H, H<sub>5</sub> + H<sub>arom</sub>), 7.64 (m, 1H, H<sub>arom</sub>), 7.97 (m, 2H, H<sub>arom</sub>), 8.46 (d, 1H, H<sub>6</sub>, J = 7.5 Hz) ppm. <sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>): δ 63.7 (C<sub>5'</sub>), 89.9 (C<sub>4'</sub>), 93.4 (C<sub>1'</sub>), 98.5 (C<sub>5</sub>), 127.6 (C<sub>2</sub>), 129.2 (CH<sub>arom</sub>), 129.8 (CH<sub>arom</sub>), 134.1 (CH<sub>arom</sub>), 134.7 (C<sub>arom</sub>), 135.5 (C<sub>3'</sub>), 147.3 (C<sub>6</sub>), 158.3 (C<sub>2</sub>), 165.0 (C<sub>4</sub>), 169.1 (C=O) ppm.

**2',3'-Didehydro-2',3'-dideoxy-β-d-adenosine (8e).** <sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>): δ 3.75 (m, 2H, H<sub>5'</sub>), 5.01 (m, 1H, H<sub>4'</sub>), 6.13 (m, 1H, H<sub>2'</sub>), 6.48 (dt, 1H, H<sub>3'</sub>, J = 6.0, 1.7 Hz), 7.04 (dt, 1H, H<sub>1'</sub>, J = 3.2, 1.7 Hz), 8.20 (s, 1H, H<sub>2</sub>), 8.28 (s, 1H, H<sub>8</sub>) ppm. <sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>): δ 64.2 (C<sub>5'</sub>), 89.8 (C<sub>1'</sub>), 90.3 (C<sub>4'</sub>), 120.2 (C<sub>5</sub>), 126.8 (C<sub>2'</sub>), 135.7 (C<sub>3'</sub>), 141.3 (C<sub>8</sub>), 150.3 (C<sub>4</sub>), 153.8 (C<sub>2</sub>), 157.4 (C<sub>6</sub>) ppm.

**2',3'-Didehydro-2',3'-dideoxy-β-d-inosine (8f).** <sup>1</sup>H-NMR (300.13 MHz, DMSO-*d*<sub>6</sub>): δ 3.56 (m, 2H, H<sub>5'</sub>), 4.89 (m, 1H, H<sub>4'</sub>), 4.98 (t, OH<sub>5'</sub>, J = 5.4 Hz), 6.13 (m, 1H, H<sub>2'</sub>), 6.48 (dd, 1H, H<sub>3'</sub>, J = 6.0, 1.5 Hz), 6.90 (m, 1H, H<sub>1'</sub>), 8.08 (s, 1H, H<sub>2</sub>), 8.11 (s, 1H, H<sub>8</sub>), 12.39 (br s, 1H, NH) ppm. <sup>13</sup>C-NMR (75.5 MHz, DMSO-*d*<sub>6</sub>): δ 62.7 (C<sub>5'</sub>), 88.1 (C<sub>1'</sub>), 88.3 (C<sub>4'</sub>), 124.1 (C<sub>5</sub>), 125.3 (C<sub>2'</sub>), 134.8 (C<sub>3'</sub>), 138.6 (C<sub>8</sub>), 146.0 (C<sub>2</sub>), 148.1 (C<sub>4</sub>), 156.7 (C<sub>6</sub>) ppm.

**2',3'-Dideoxy-β-d-uridine (9a).** <sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>): δ 1.96 (m, 2H, H<sub>3'</sub>), 2.08 (m, 1H, H<sub>2'</sub>), 2.40 (m, 1H, H<sub>2</sub>), 3.68 (dd, 1H, H<sub>5'</sub>, J = 12.2, 4.0 Hz), 3.86 (dd, 1H, H<sub>5'</sub>, J = 12.2, 3.1 Hz), 4.14 (m, 1H, H<sub>4'</sub>), 5.66 (d, 1H, H<sub>5</sub>, J = 8.1 Hz), 6.04 (dd, 1H, H<sub>1'</sub>, J = 6.8, 3.4 Hz), 8.08 (d, 1H, H<sub>6</sub>, J = 8.1 Hz) ppm. <sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>): δ 25.9 (C<sub>3'</sub>), 33.5 (C<sub>2'</sub>), 63.7 (C<sub>5'</sub>), 83.5 (C<sub>4'</sub>), 87.7 (C<sub>1'</sub>), 101.9 (C<sub>5</sub>), 142.6 (C<sub>6</sub>), 152.2 (C<sub>2</sub>), 166.4 (C<sub>4</sub>) ppm.

**3'-Deoxy-β-d-5-thymidine (9b).** <sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>): δ 1.88 (d, 3H, Me, J = 1.1 Hz), 1.98 (m, 2H, H<sub>3'</sub>), 2.07 (m, 1H, H<sub>2'</sub>), 2.38 (m, 1H, H<sub>2</sub>), 3.68 (dd, 1H, H<sub>5'</sub>, J = 12.2, 3.8 Hz), 3.88 (dd, 1H, H<sub>5'</sub>, J = 12.2, 2.9

Hz), 4.13 (m, 1H, H<sub>4'</sub>), 6.05 (dd, 1H, H<sub>1'</sub>, *J* = 6.8, 3.5 Hz), 7.92 (d, 1H, H<sub>6</sub>, *J* = 1.1 Hz) ppm. <sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>): δ 12.4 (CH<sub>3</sub>), 25.9 (C<sub>3'</sub>), 33.3 (C<sub>2'</sub>), 63.7 (C<sub>5'</sub>), 83.2 (C<sub>4'</sub>), 87.3 (C<sub>1'</sub>), 110.8 (C<sub>5</sub>), 138.4 (C<sub>6</sub>), 152.4 (C<sub>2</sub>), 166.6 (C<sub>4</sub>) ppm.

2',3'-Dideoxy-β-d-cytidine (**9c**). <sup>1</sup>H-NMR (300.13 MHz, D<sub>2</sub>O): δ 1.78 (m, 1H, H<sub>3'</sub>), 2.05 (m, 2H, H<sub>2'</sub> + H<sub>3'</sub>), 2.45 (m, 1H, H<sub>2'</sub>), 3.72 (dd, 1H, H<sub>5'</sub>, *J* = 12.4, 5.4 Hz), 3.87 (dd, 1H, H<sub>5'</sub>, *J* = 12.4, 3.1 Hz), 4.23 (m, 1H, H<sub>4'</sub>), 6.00 (d, 1H, H<sub>5</sub>, *J* = 7.5 Hz), 6.05 (dd, 1H, H<sub>1'</sub>, *J* = 7.0, 2.7 Hz), 7.89 (d, 1H, H<sub>6</sub>, *J* = 7.5 Hz) ppm. <sup>13</sup>C-NMR (75.5 MHz, D<sub>2</sub>O): δ 24.7 (C<sub>3'</sub>), 31.9 (C<sub>2'</sub>), 62.6 (C<sub>5'</sub>), 82.2 (C<sub>4'</sub>), 87.0 (C<sub>1'</sub>), 95.5 (C<sub>5</sub>), 141.6 (C<sub>6</sub>), 157.3 (C<sub>2</sub>), 165.9 (C<sub>4</sub>) ppm.

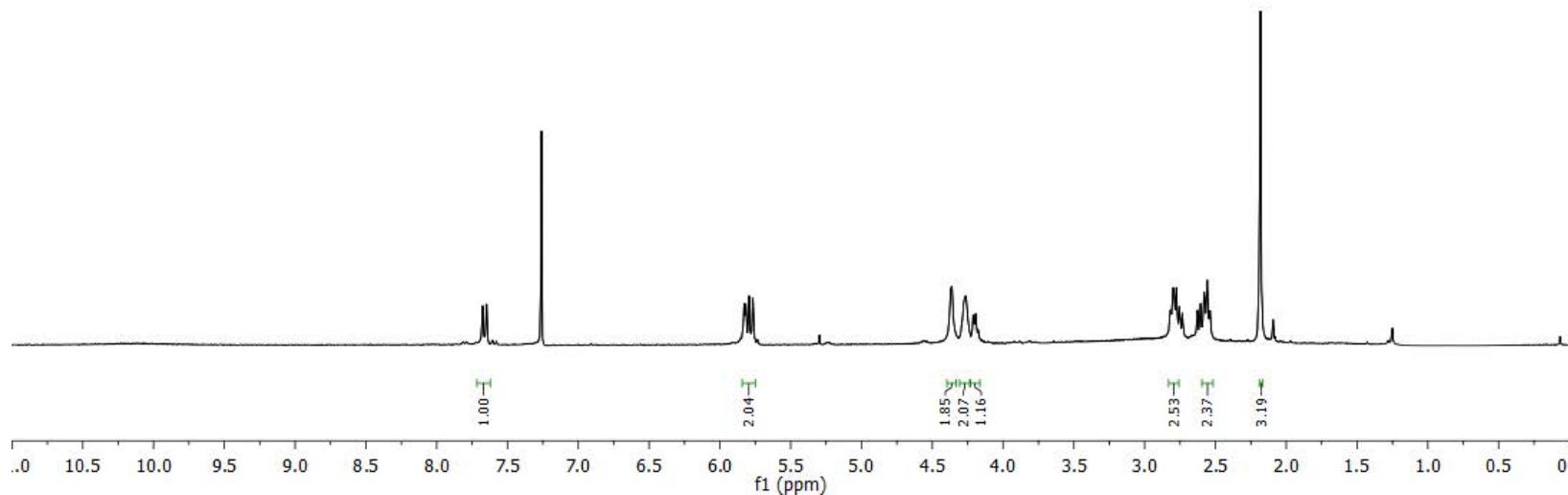
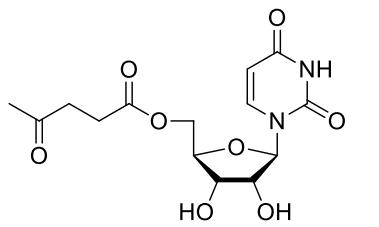
2',3'-Dideoxy-β-d-adenosine (**9e**). <sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>): δ 2.15 (m, 2H, H<sub>3'</sub>), 2.51 (m, 2H, H<sub>2'</sub>), 3.67 (dd, 1H, H<sub>5'</sub>, *J* = 12.2, 3.9 Hz), 3.87 (dd, 1H, H<sub>5'</sub>, *J* = 12.2, 3.0 Hz), 4.27 (m, 1H, H<sub>4'</sub>), 6.28 (dd, 1H, H<sub>1'</sub>, *J* = 5.8, 4.8 Hz), 8.18 (s, 1H, H<sub>2</sub>), 8.41 (s, 1H, H<sub>8</sub>) ppm. <sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>): δ 26.5 (C<sub>3'</sub>), 33.7 (C<sub>2'</sub>), 64.5 (C<sub>5'</sub>), 83.6 (C<sub>4'</sub>), 87.4 (C<sub>1'</sub>), 120.5 (C<sub>5</sub>), 141.0 (C<sub>2</sub>), 149.8 (C<sub>4</sub>), 153.5 (C<sub>8</sub>), 157.3 (C<sub>6</sub>) ppm.

2',3'-Dideoxy-β-d-inosine (**9f**). <sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>): δ 2.12 (m, 2H, H<sub>3'</sub>), 2.52 (m, 2H, H<sub>2'</sub>), 3.67 (dd, 1H, H<sub>5'</sub>, *J* = 12.1, 4.2 Hz), 3.84 (dd, 1H, H<sub>5'</sub>, *J* = 12.1, 3.2 Hz), 4.26 (m, 1H, H<sub>4'</sub>), 6.31 (dd, 1H, H<sub>1'</sub>, *J* = 6.5, 3.5 Hz), 8.04 (s, 1H, H<sub>2</sub>), 8.39 (s, 1H, H<sub>8</sub>) ppm. <sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>): δ 26.4 (C<sub>3'</sub>), 33.9 (C<sub>2'</sub>), 64.3 (C<sub>5'</sub>), 83.9 (C<sub>4'</sub>), 87.3 (C<sub>1'</sub>), 125.7 (C<sub>5</sub>), 140.4 (C<sub>8</sub>), 146.6 (C<sub>2</sub>), 149.3 (C<sub>4</sub>), 159.0 (C<sub>6</sub>) ppm.

**5'-O-Levulinyl- $\beta$ -D-uridine (2a)**

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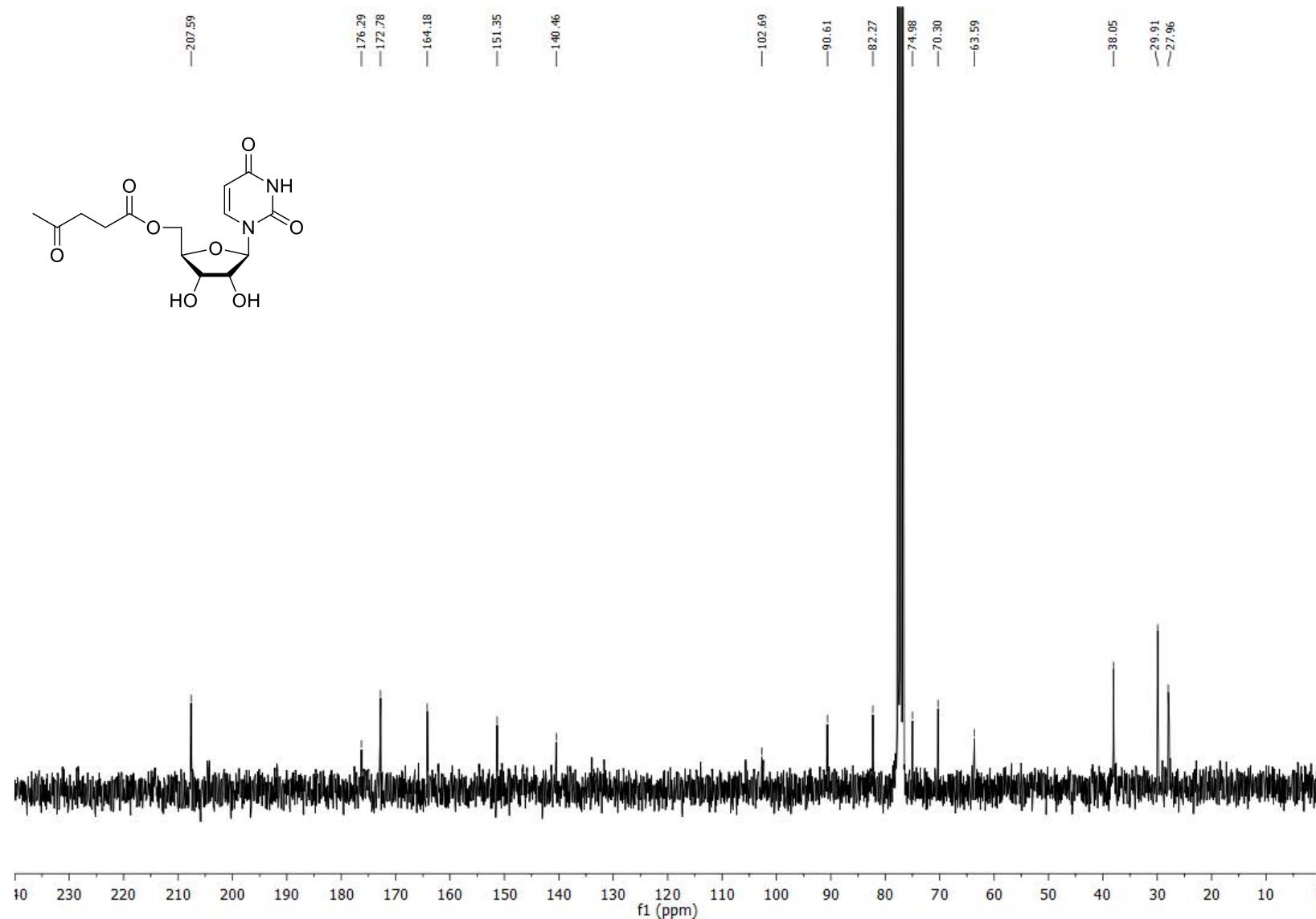
$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



**5'-O-Levulinyl- $\beta$ -D-uridine (2a)**

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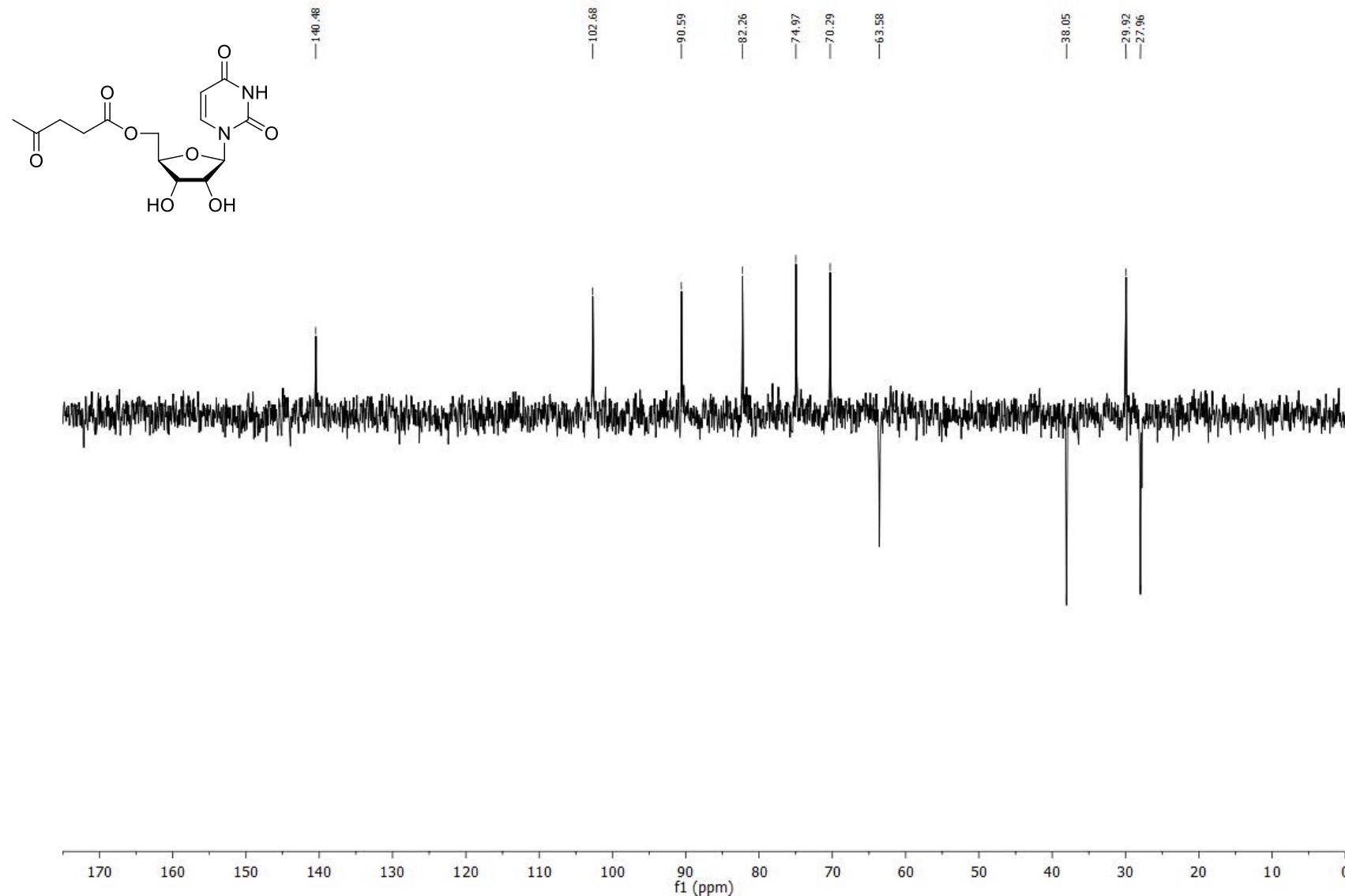
$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



**5'-O-Levulinyl- $\beta$ -D-uridine (2a)**

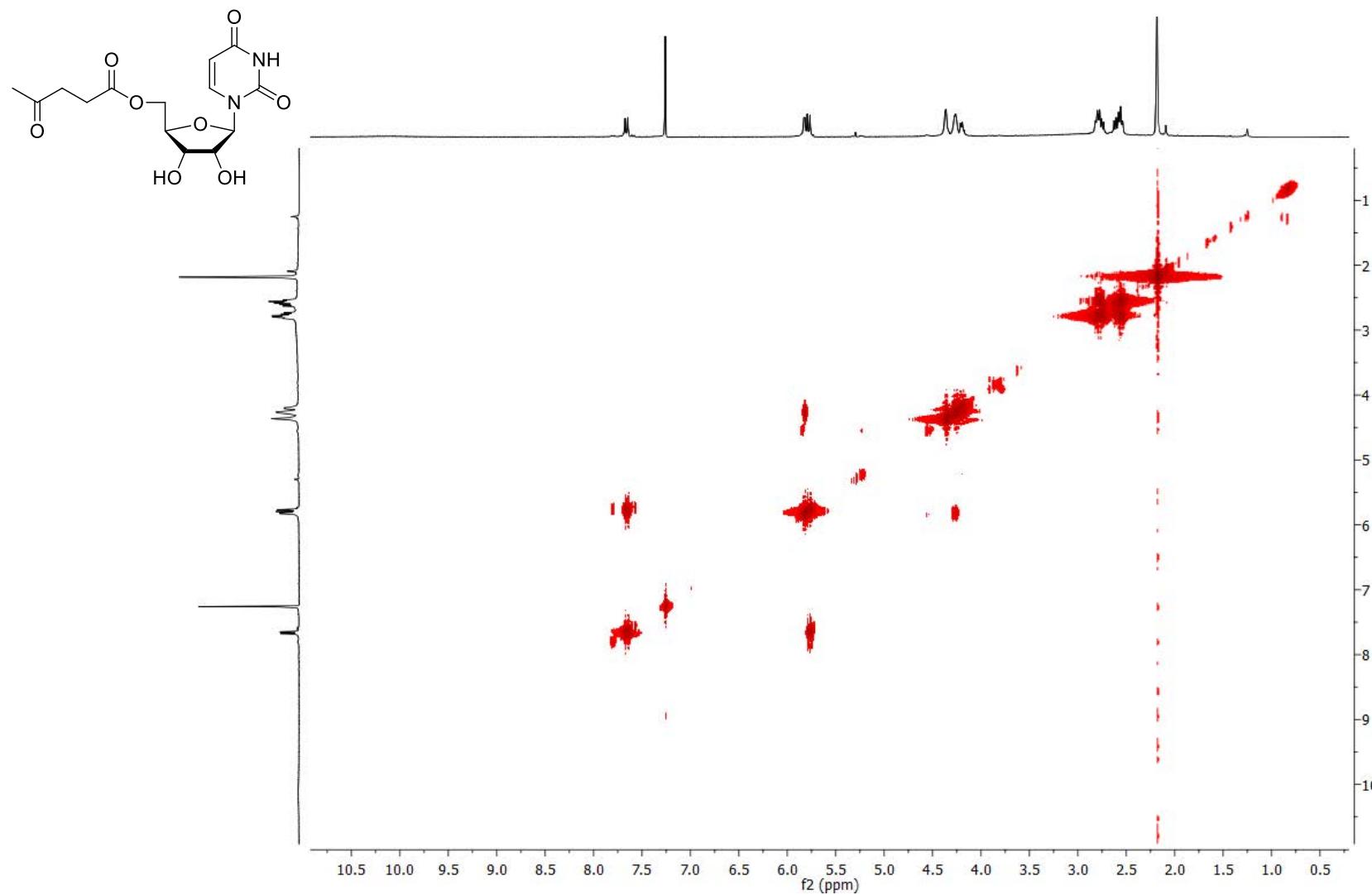
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DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



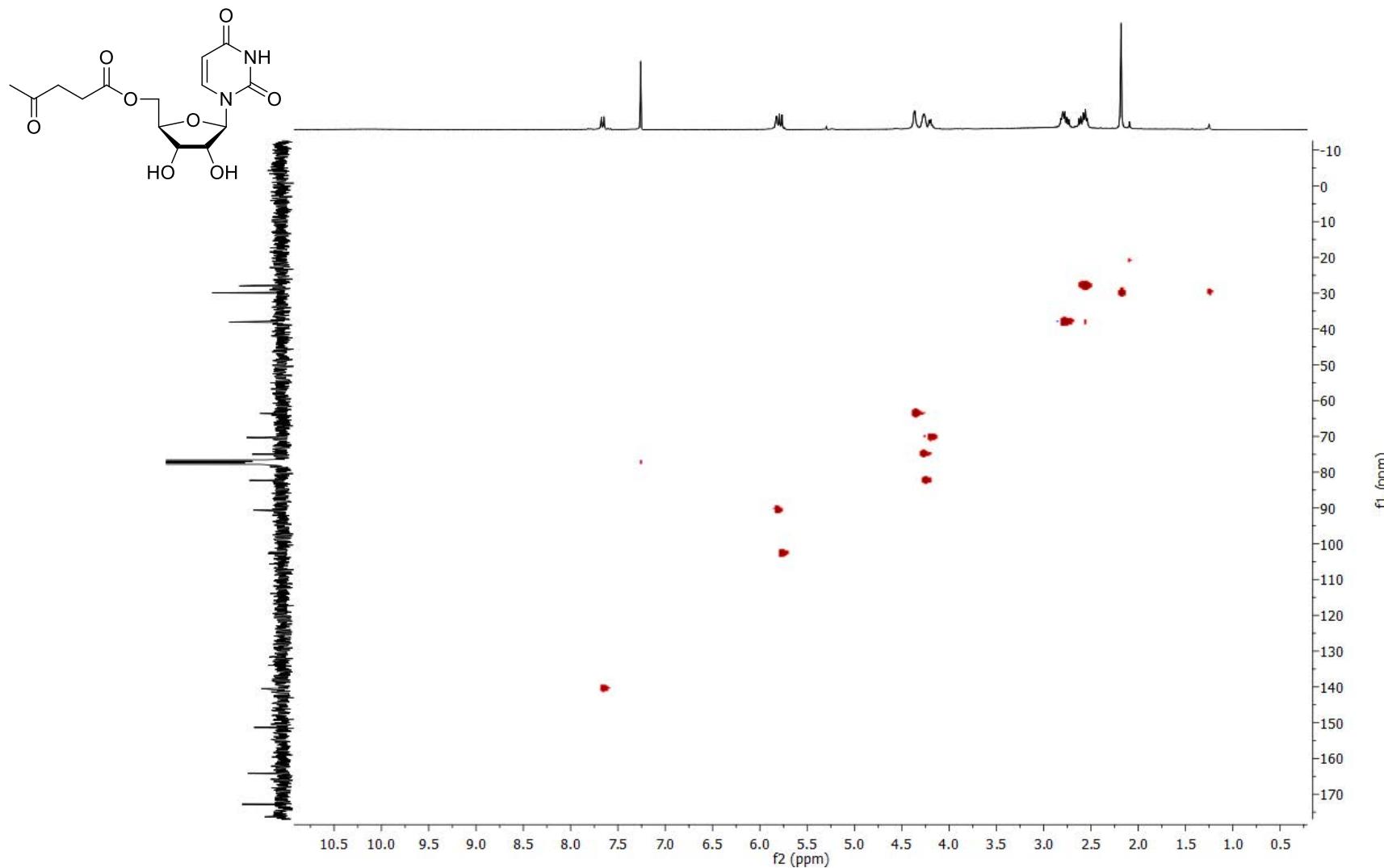
**5'-O-Levulinyl- $\beta$ -D-uridine (2a)**

COSY NMR ( $\text{CDCl}_3$ )



**5'-O-Levulinyl- $\beta$ -D-uridine (2a)**

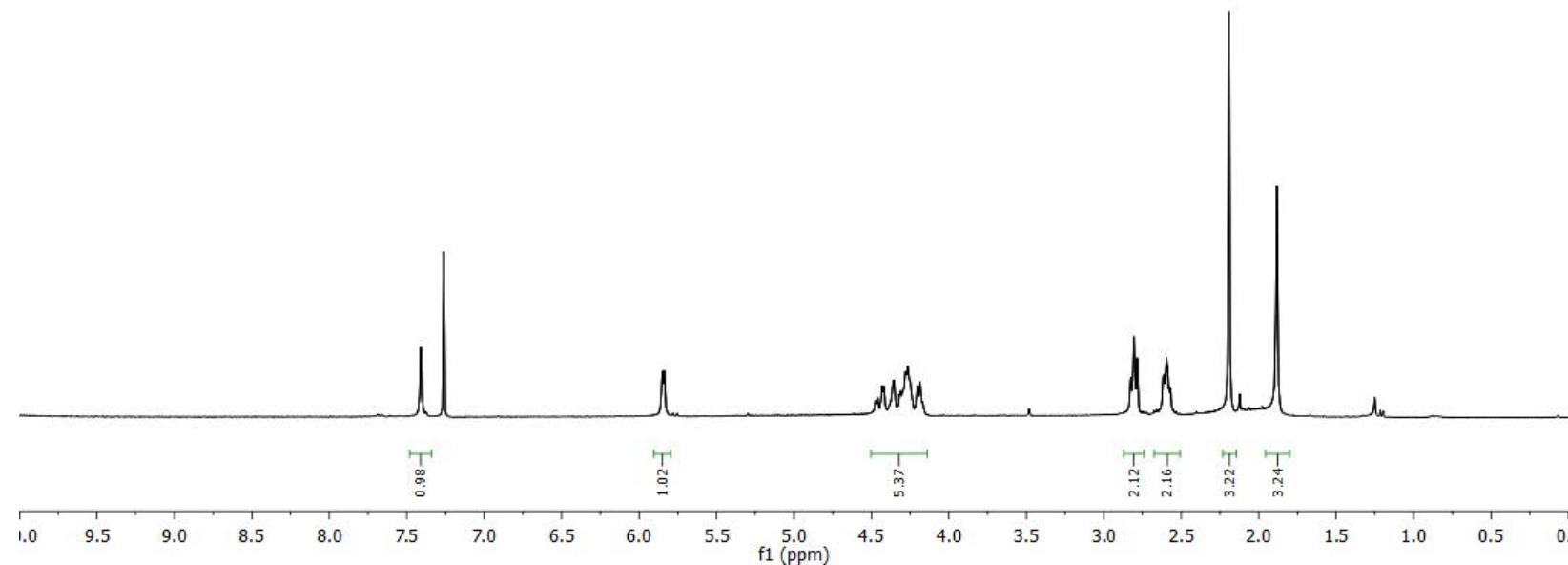
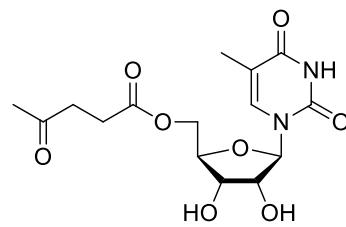
HSQC NMR ( $\text{CDCl}_3$ )



**5'-O-Levulinyl- $\beta$ -D-5-methyluridine (2b)**

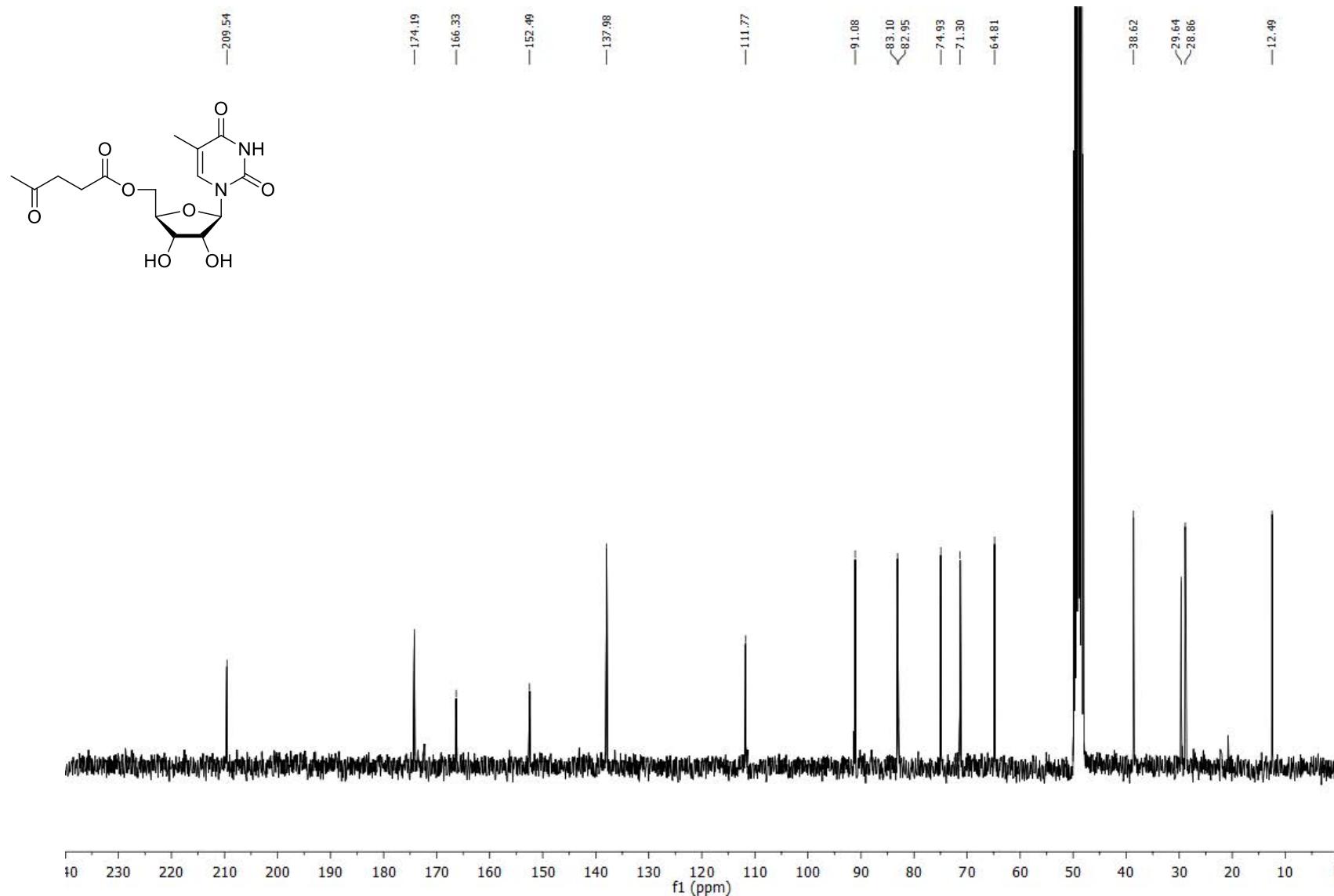
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$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



**5'-O-Levulinyl- $\beta$ -D-5-methyluridine (2b)**

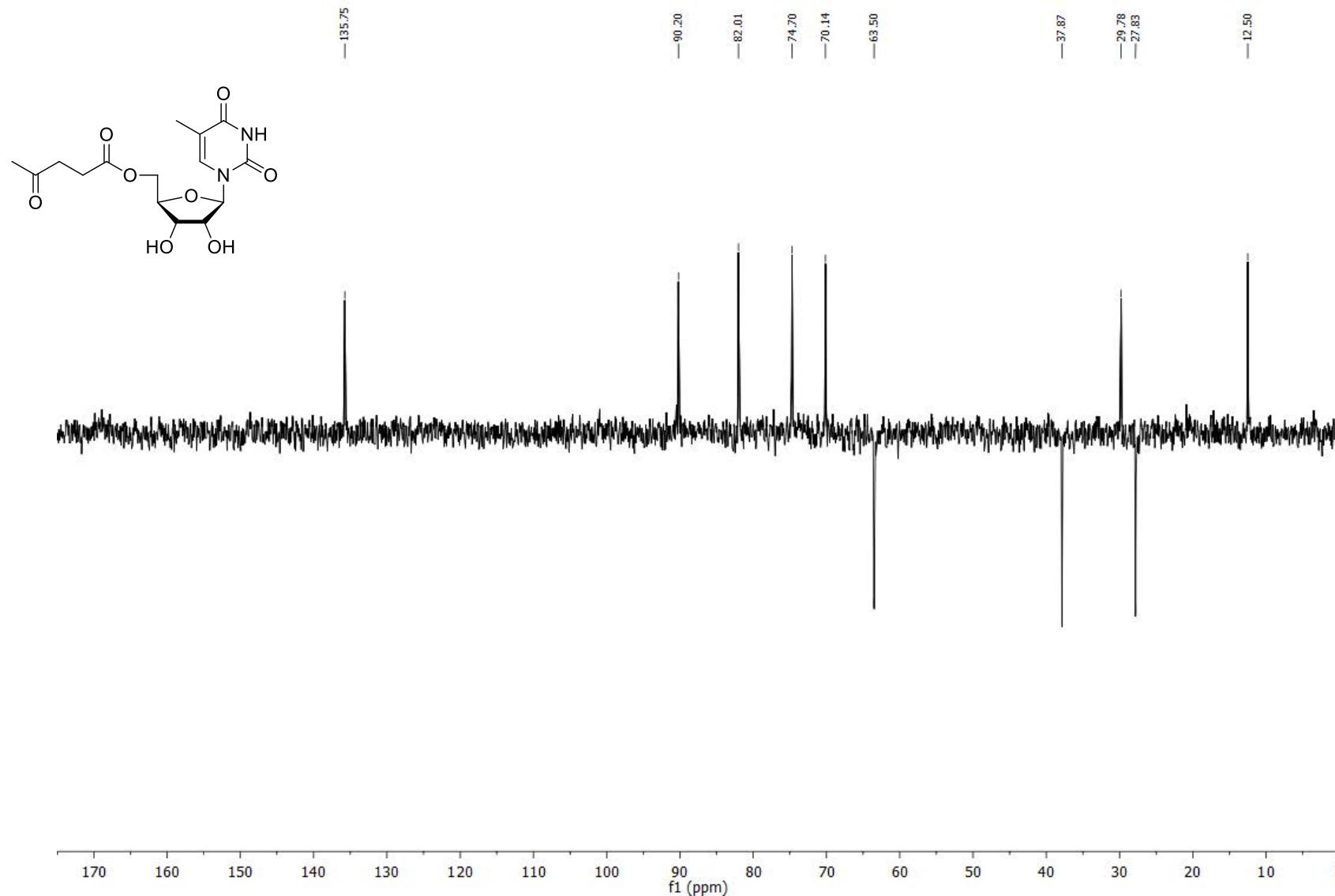
$^{13}\text{C}$ -NMR (75.5 MHz, MeOH- $d_4$ )



**5'-O-Levulinyl- $\beta$ -D-5-methyluridine (2b)**

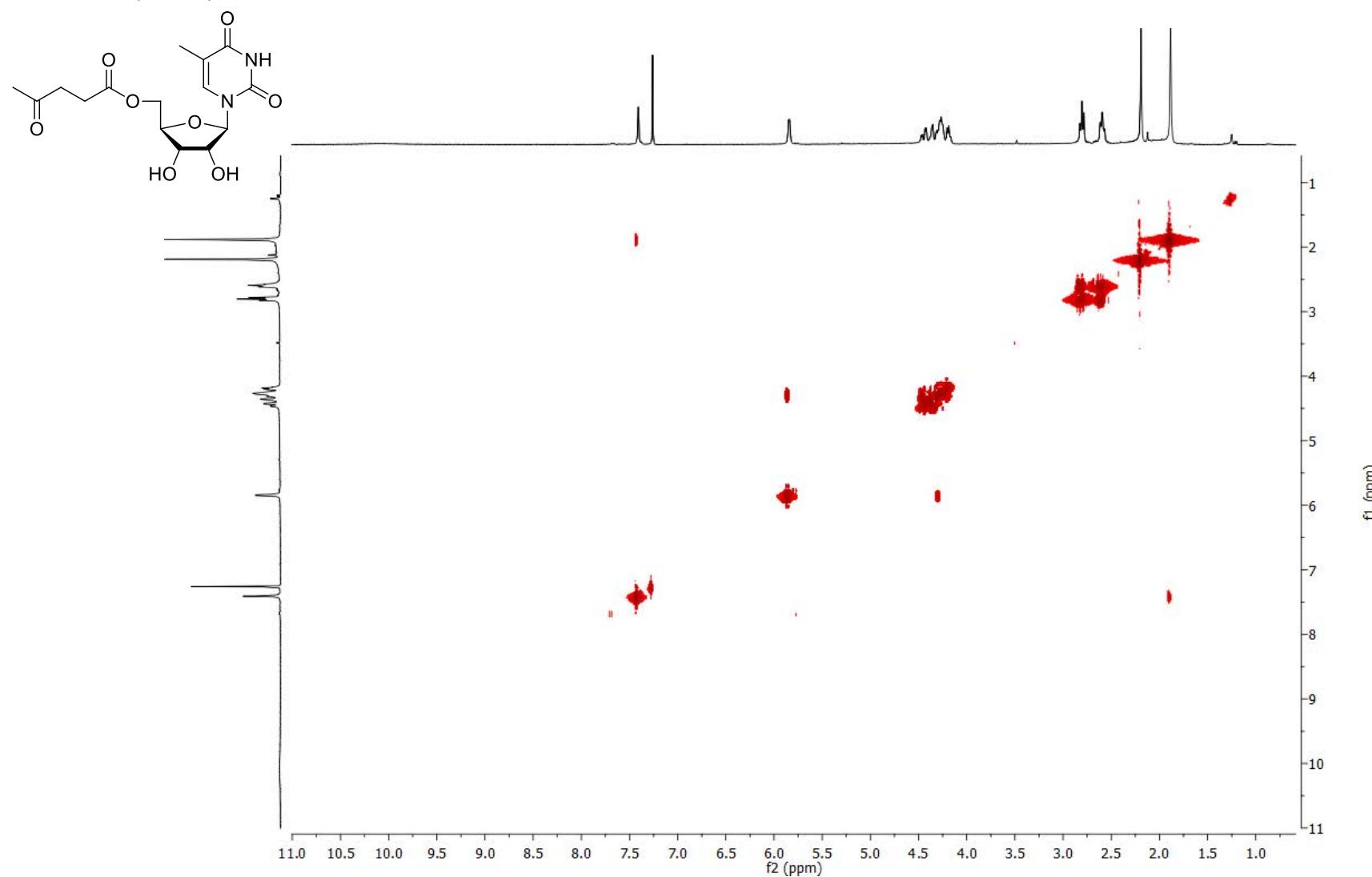
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DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



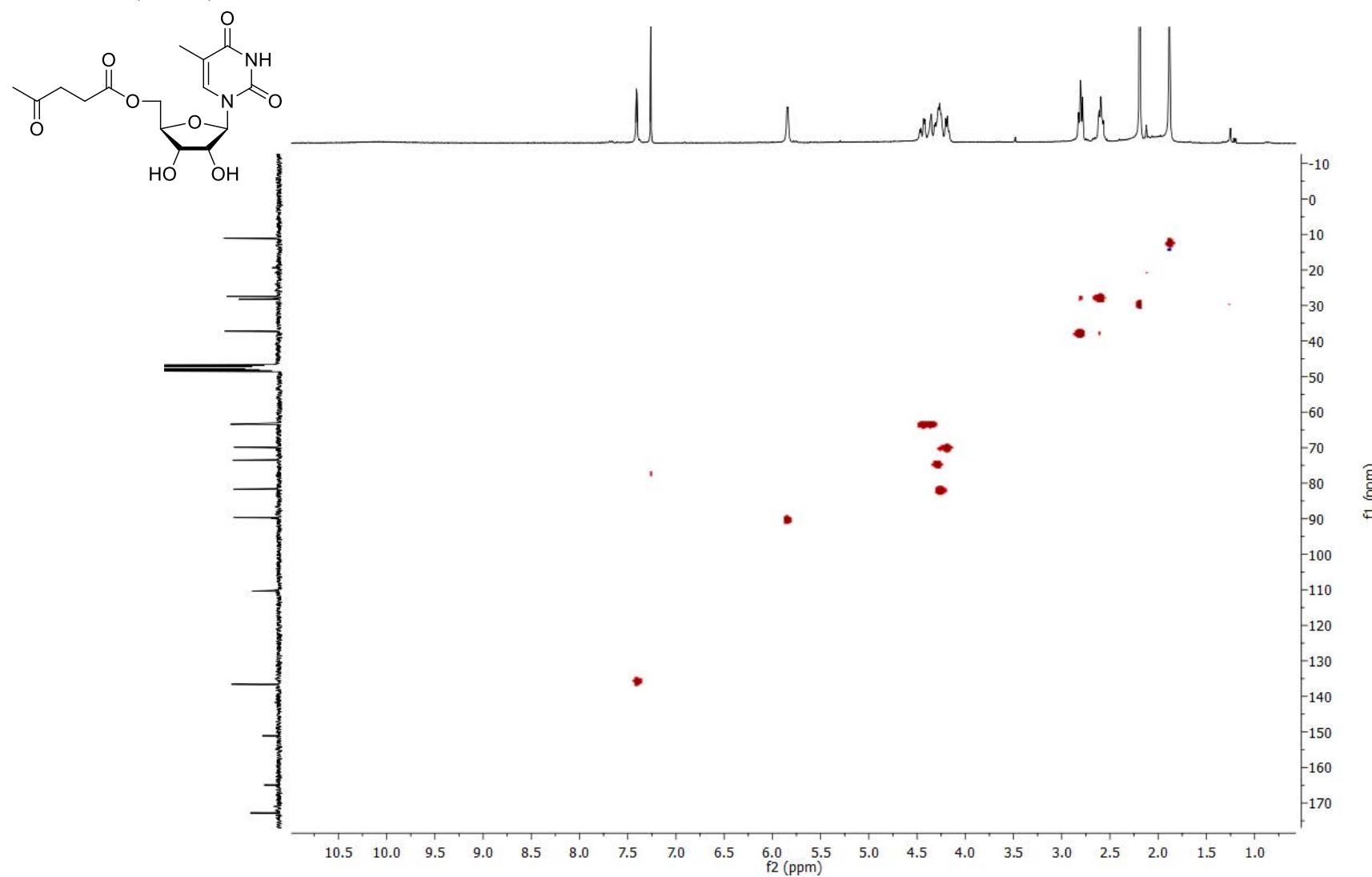
**5'-O-Levulinyl- $\beta$ -D-5-methyluridine (2b)**

COSY NMR ( $\text{CDCl}_3$ )



**5'-O-Levulinyl- $\beta$ -D-5-methyluridine (2b)**

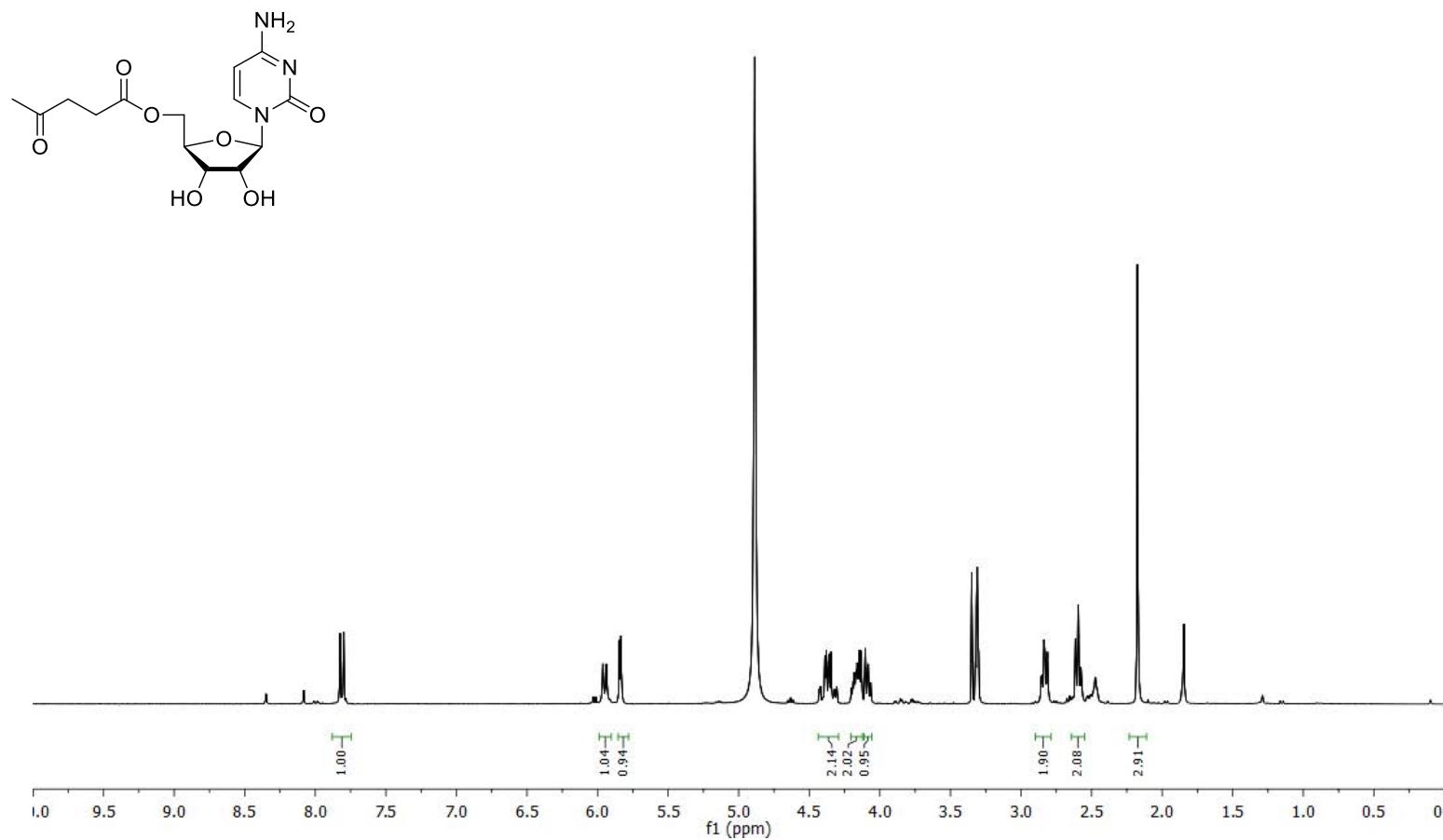
HSQC NMR ( $\text{CDCl}_3$ )



**5'-O-Levulinyl- $\beta$ -D-cytidine (2c)**

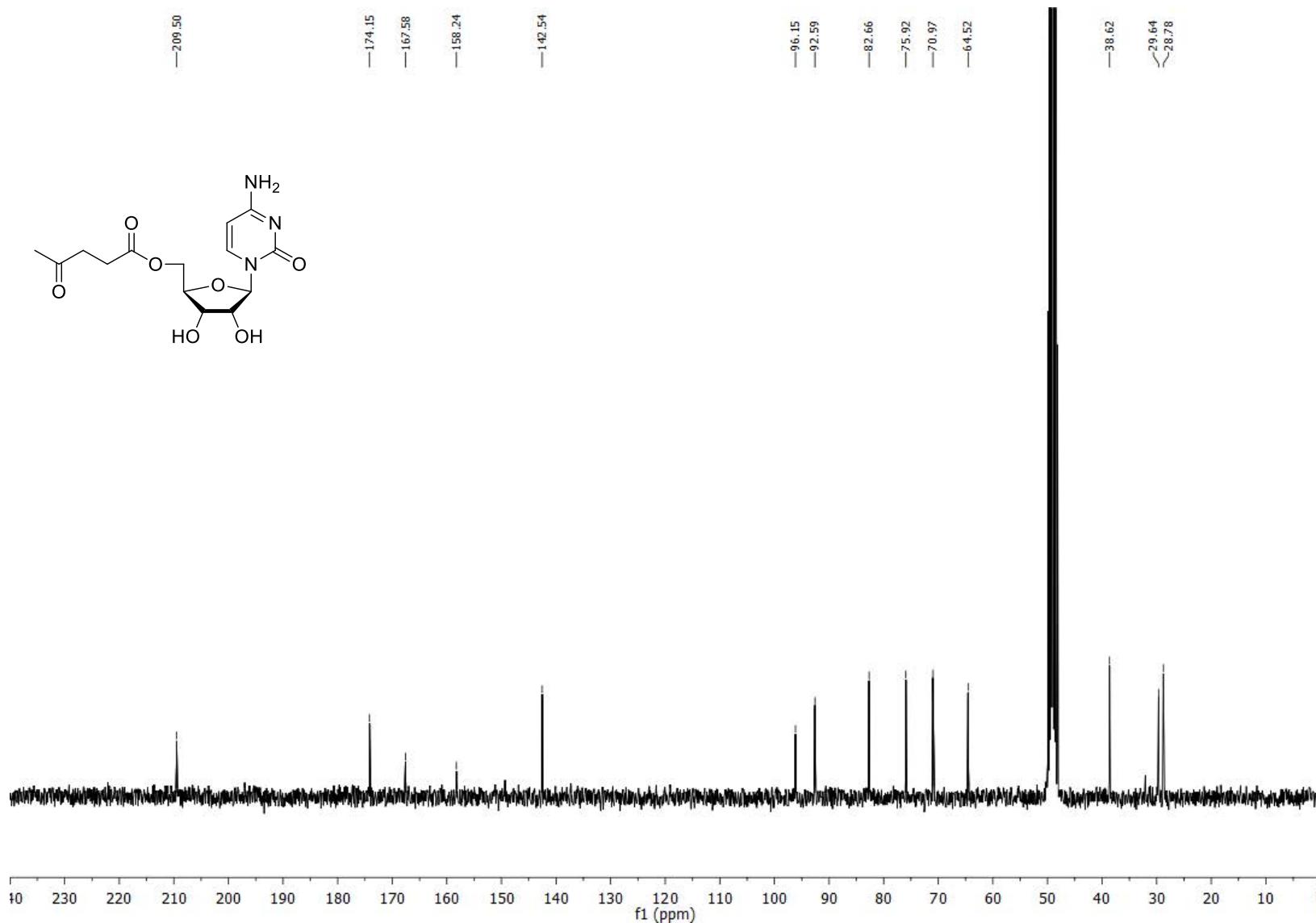
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$^1\text{H-NMR}$  (300.13 MHz, MeOD- $d_4$ )



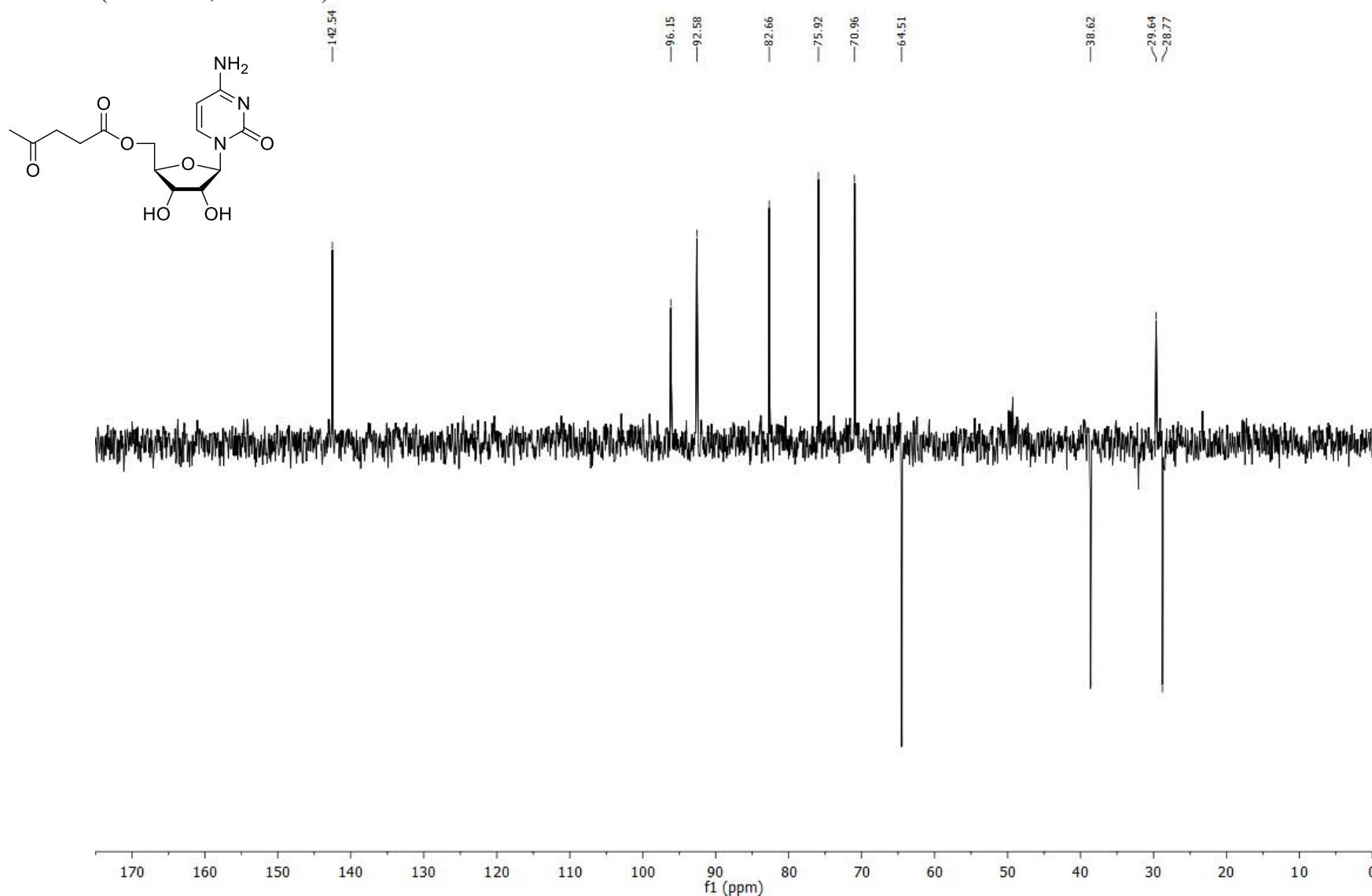
**5'-O-Levulinyl- $\beta$ -D-cytidine (2c)**

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



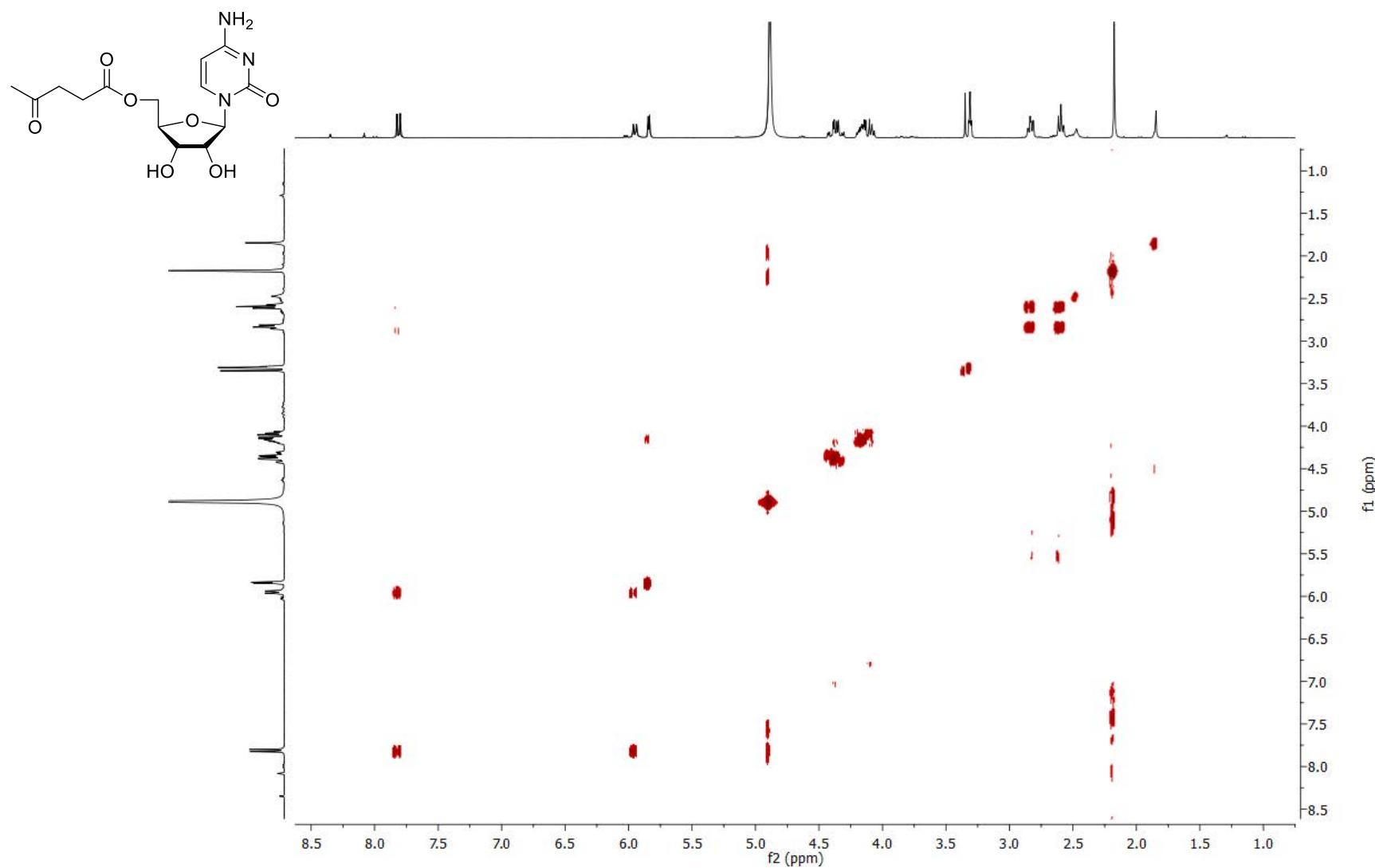
**5'-O-Levulinyl- $\beta$ -D-cytidine (2c)**

DEPT NMR (75.5 MHz, MeOD-*d*<sub>4</sub>)



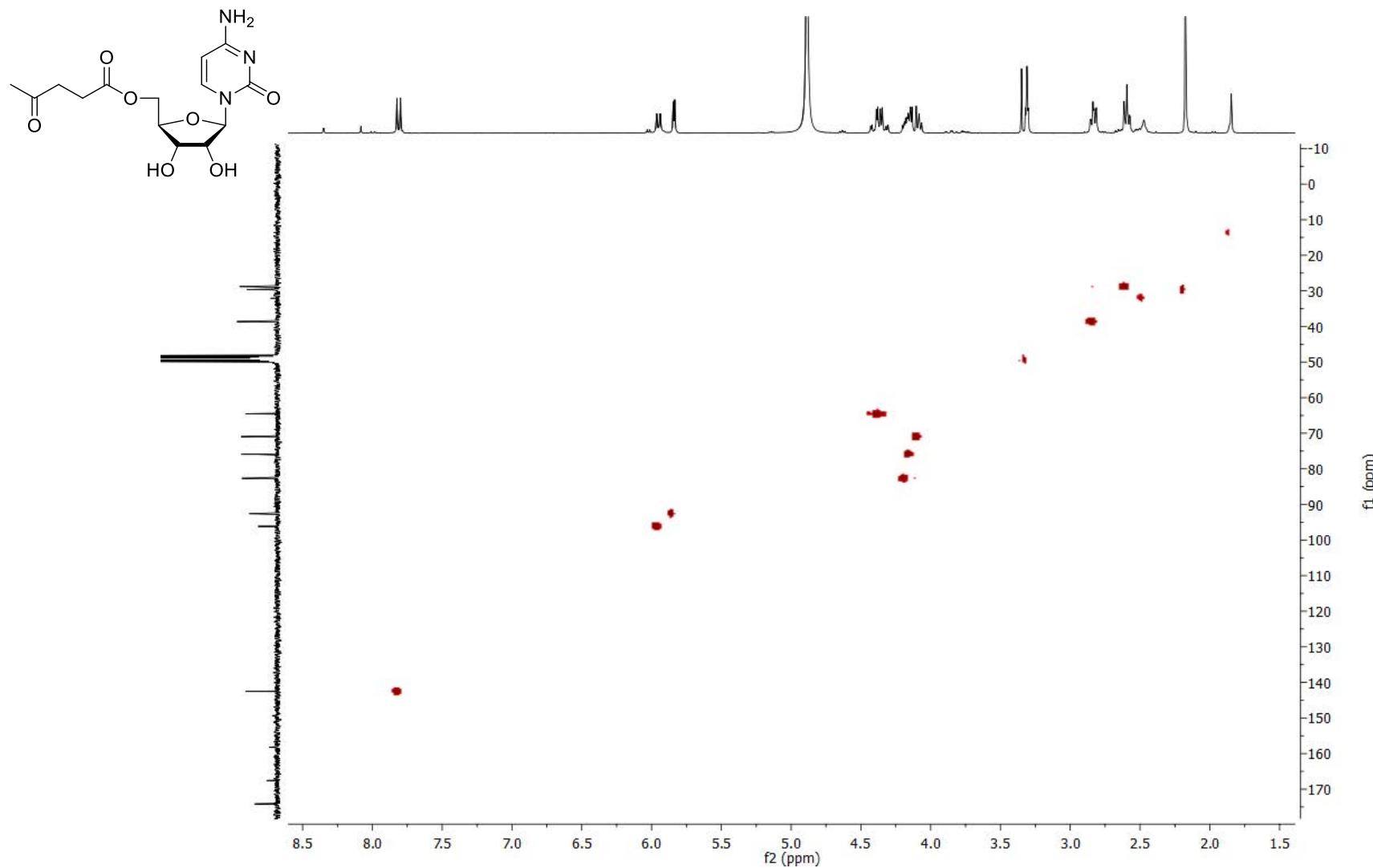
**5'-O-Levulinyl- $\beta$ -D-cytidine (2c)**

COSY NMR (MeOD-*d*<sub>4</sub>)



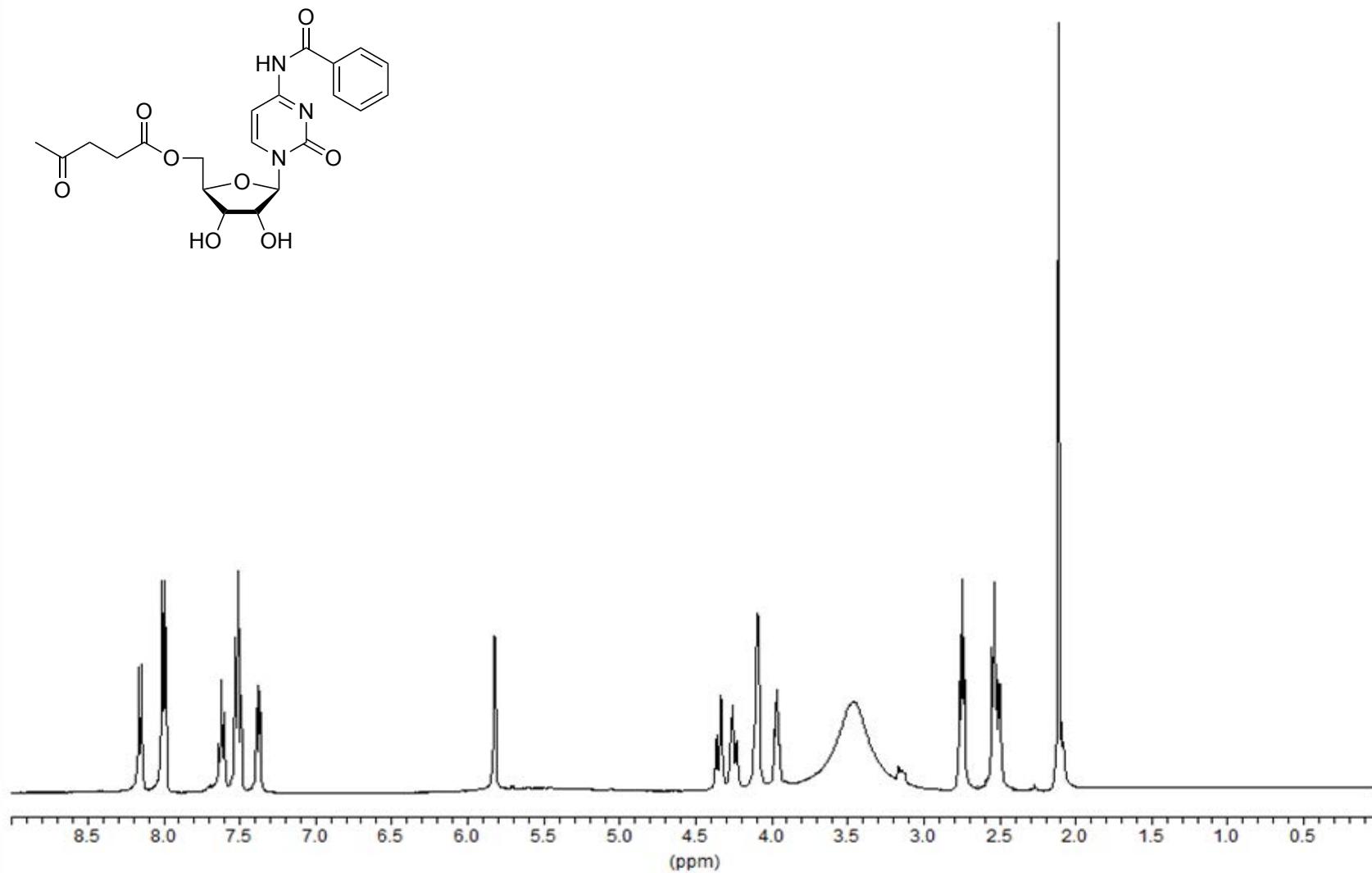
**5'-O-Levulinyl- $\beta$ -D-cytidine (2c)**

HSQC NMR (MeOD-*d*<sub>4</sub>)



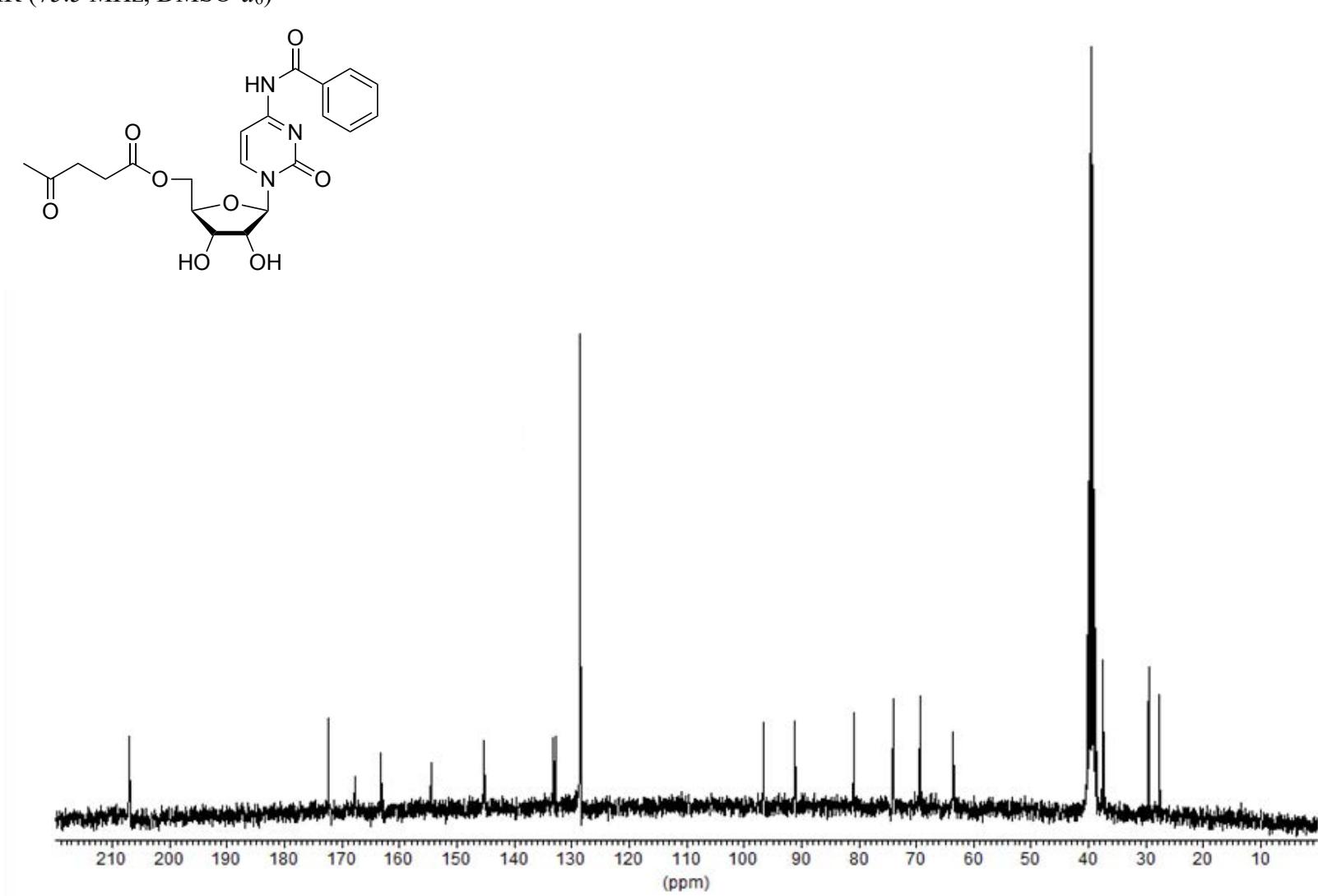
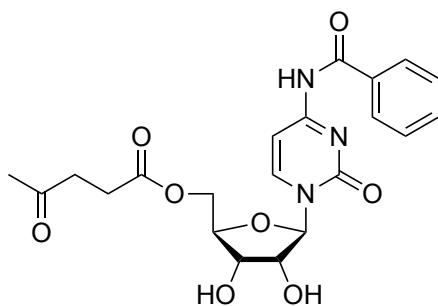
*N*-Benzoyl-5'-*O*-Levulinyl- $\beta$ -D-cytidine (2d)

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



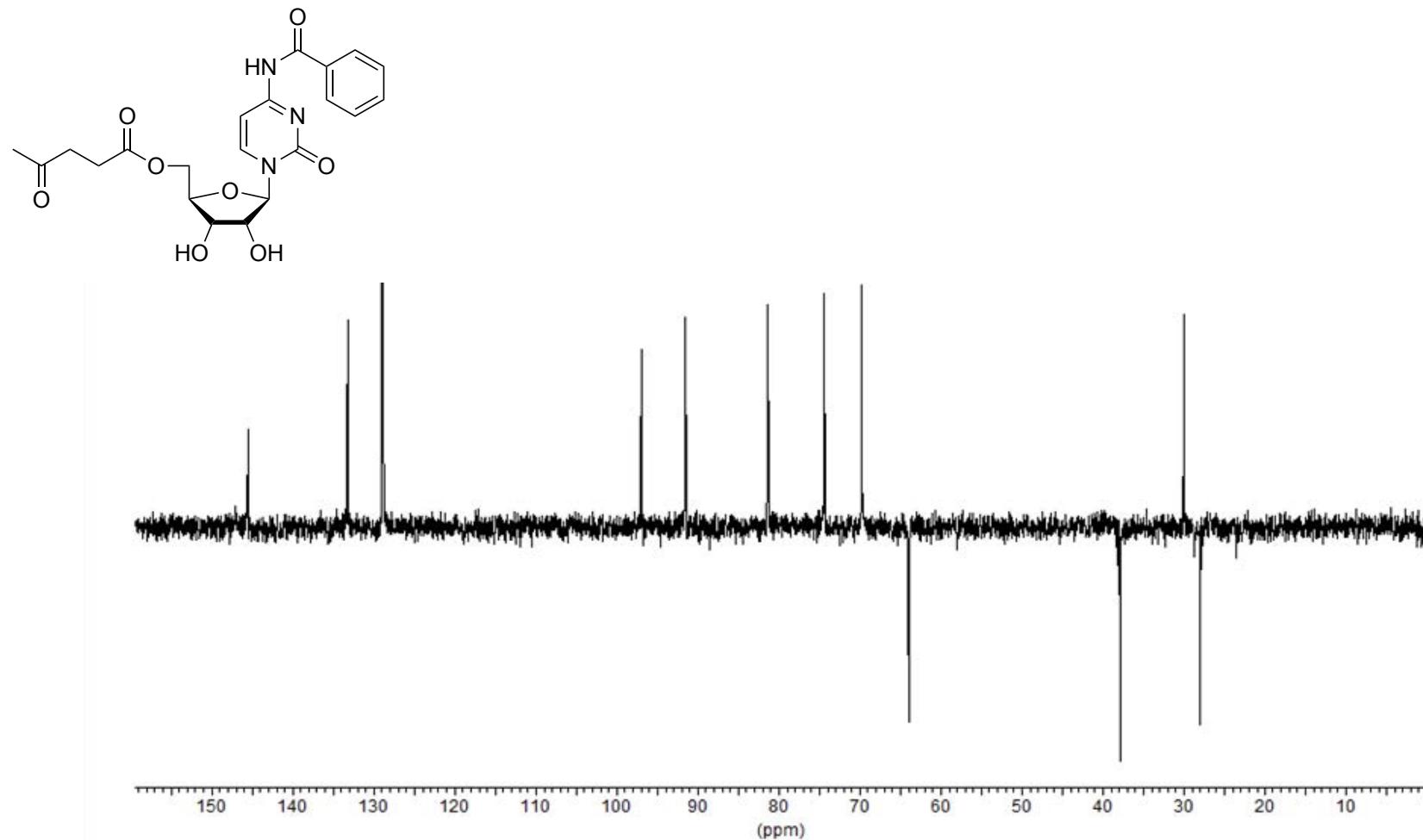
*N*-Benzoyl-5'-*O*-Levulinyl- $\beta$ -D-cytidine (2d)

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



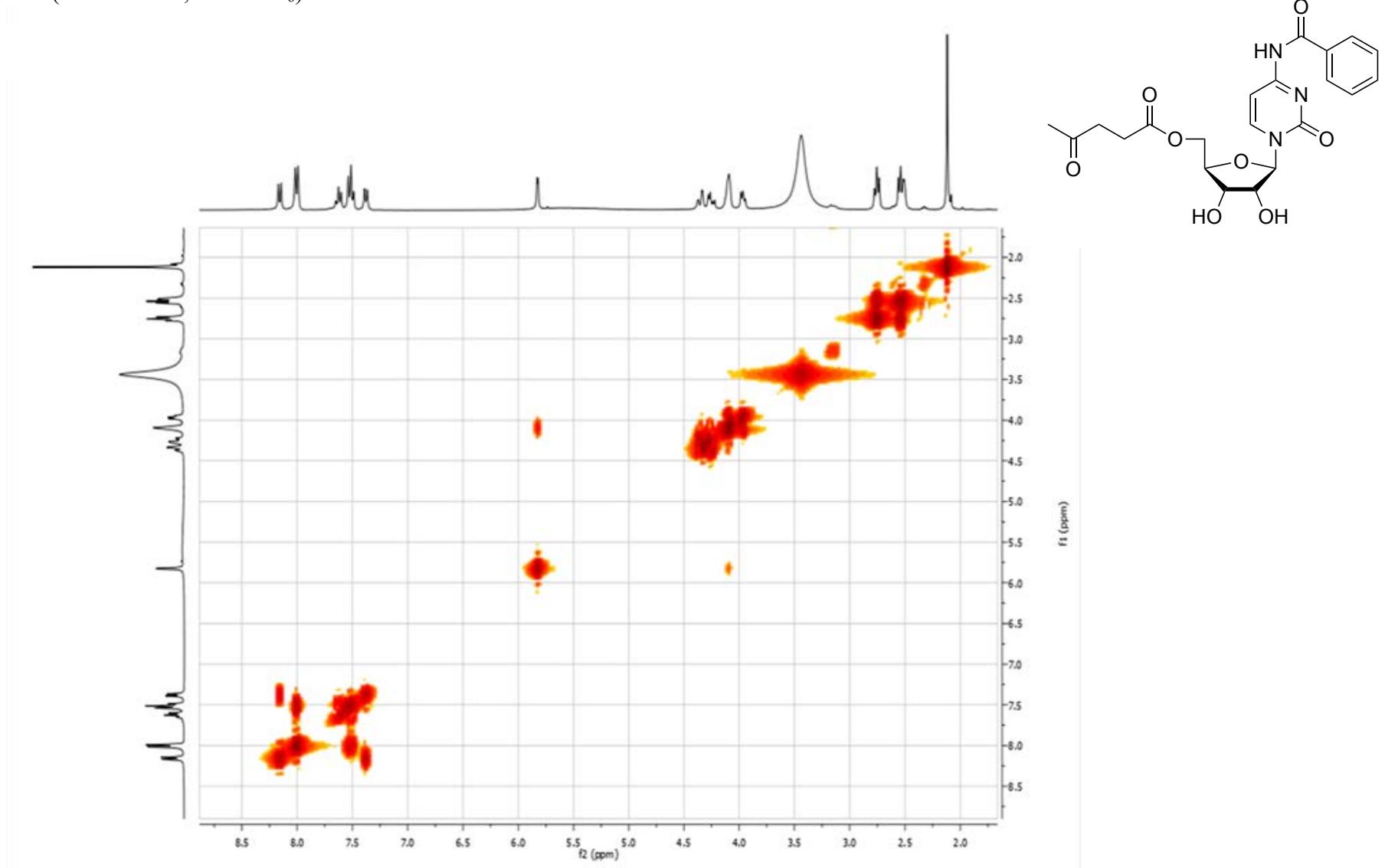
*N*-Benzoyl-5'-*O*-Levulinyl- $\beta$ -D-cytidine (2d)

DEPT NMR (75.5 MHz, DMSO-*d*<sub>6</sub>)



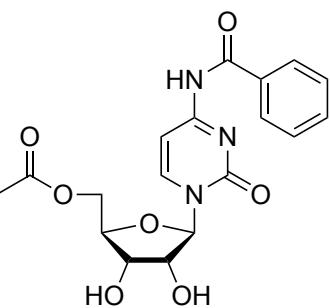
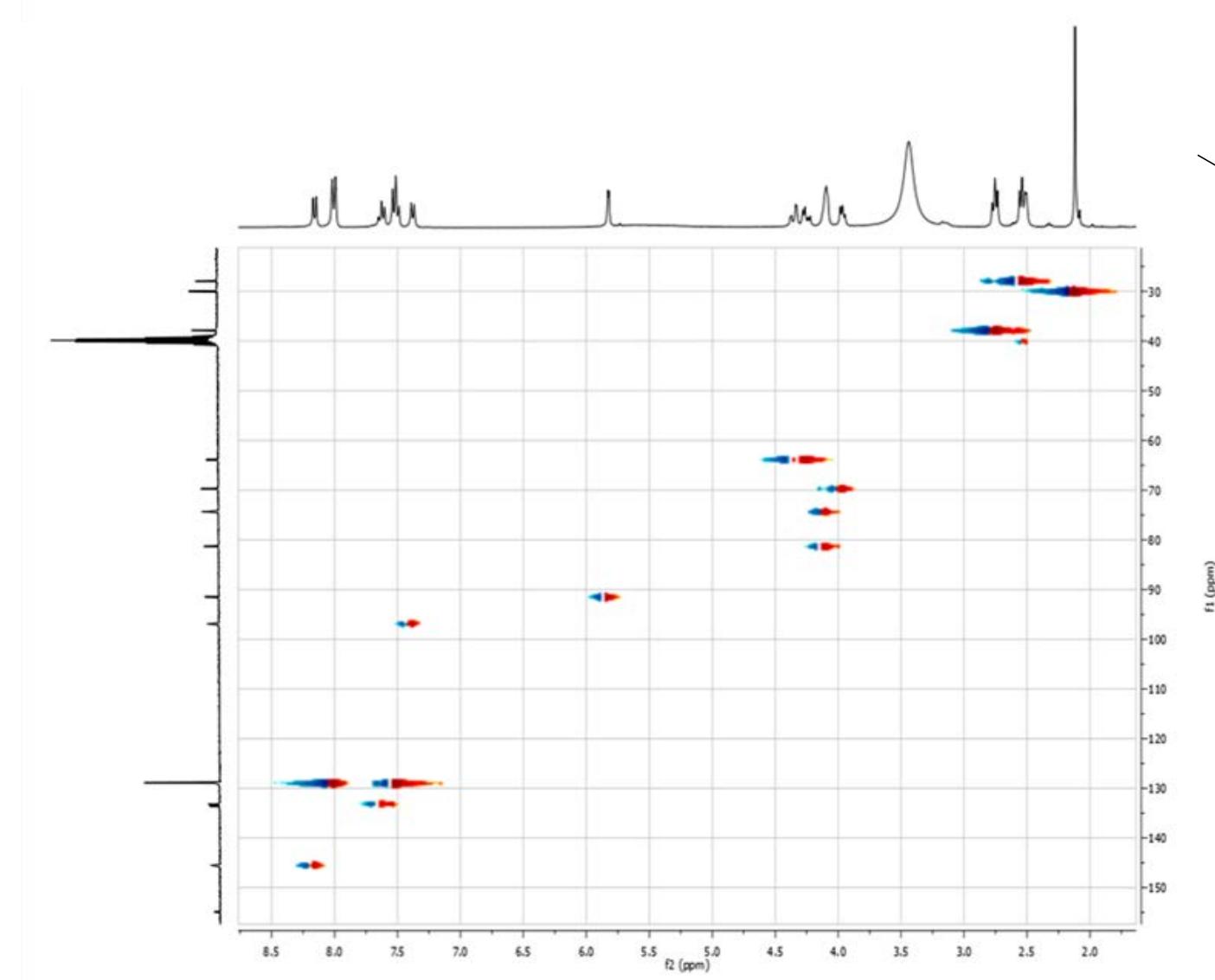
*N*-Benzoyl-5'-*O*-Levulinyl- $\beta$ -D-cytidine (2d)

COSY NMR (300.13 MHz, DMSO-*d*<sub>6</sub>)



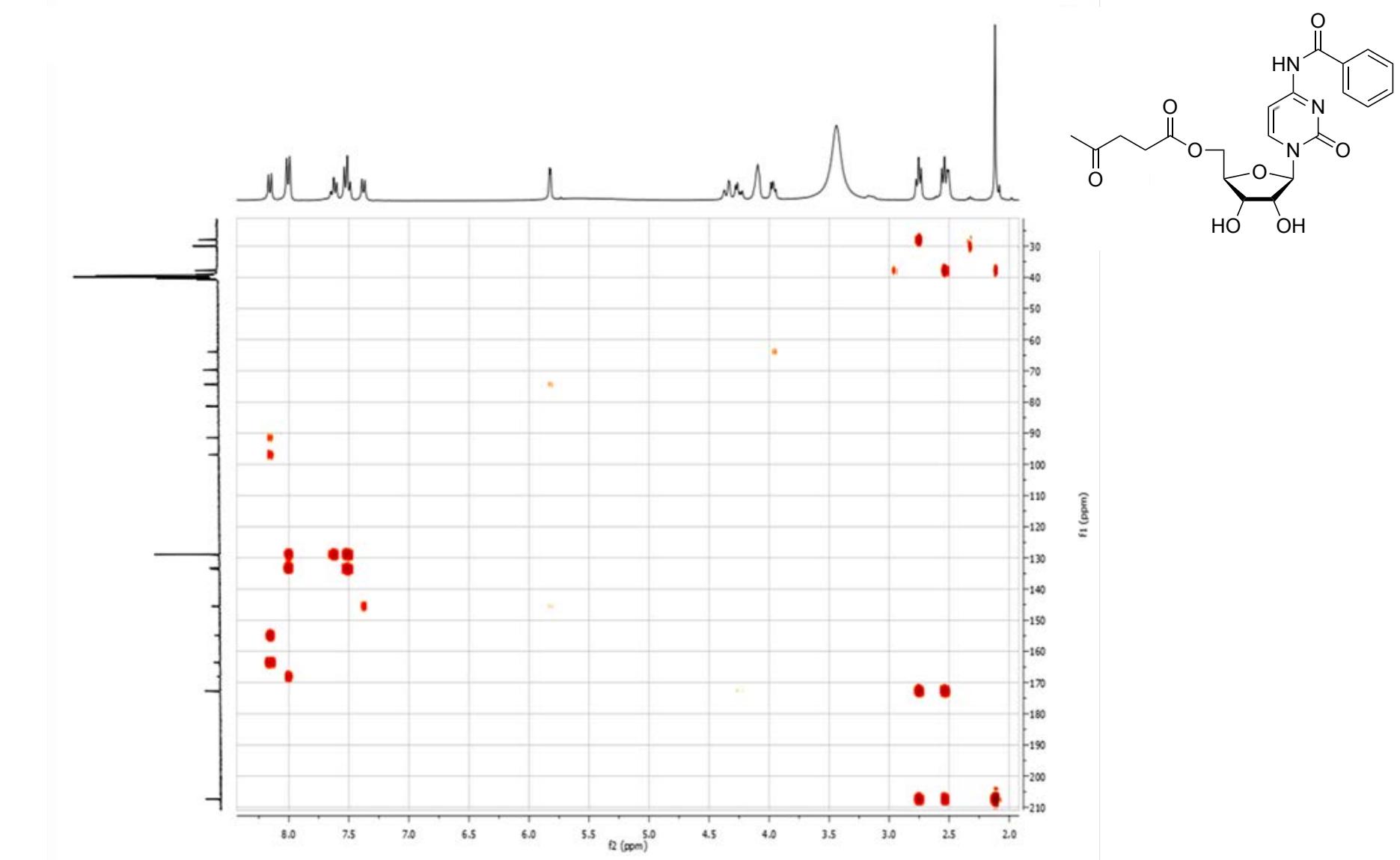
*N*-Benzoyl-5'-*O*-Levulinyl- $\beta$ -D-cytidine (2d)

HSQC NMR (300.13 MHz, DMSO-*d*<sub>6</sub>)



*N*-Benzoyl-5'-*O*-Levulinyl- $\beta$ -D-cytidine (2d)

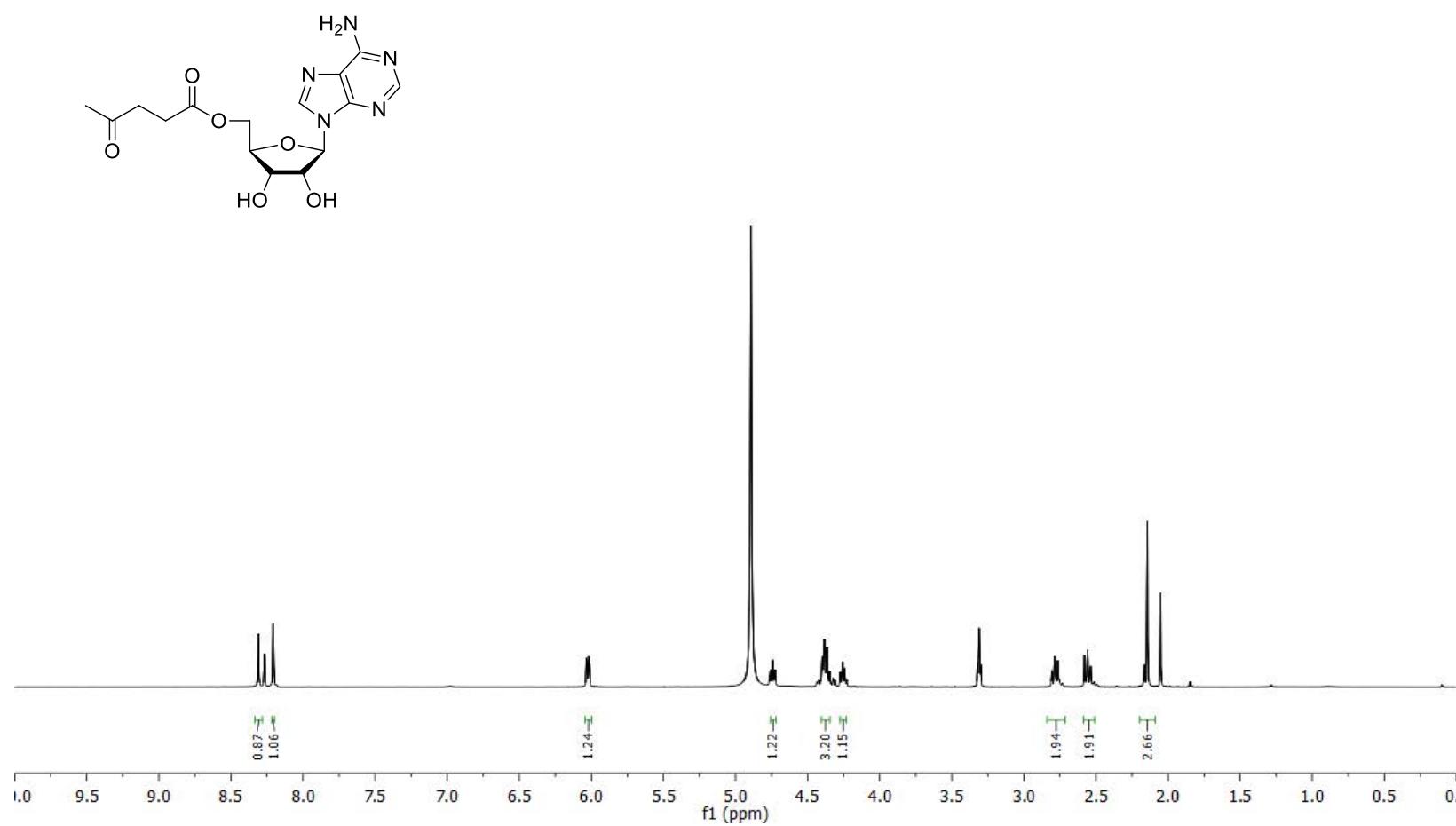
HMBC NMR (300.13 MHz, DMSO-*d*<sub>6</sub>)



**5'-O-Levulinyl- $\beta$ -D-adenosine (2e)**

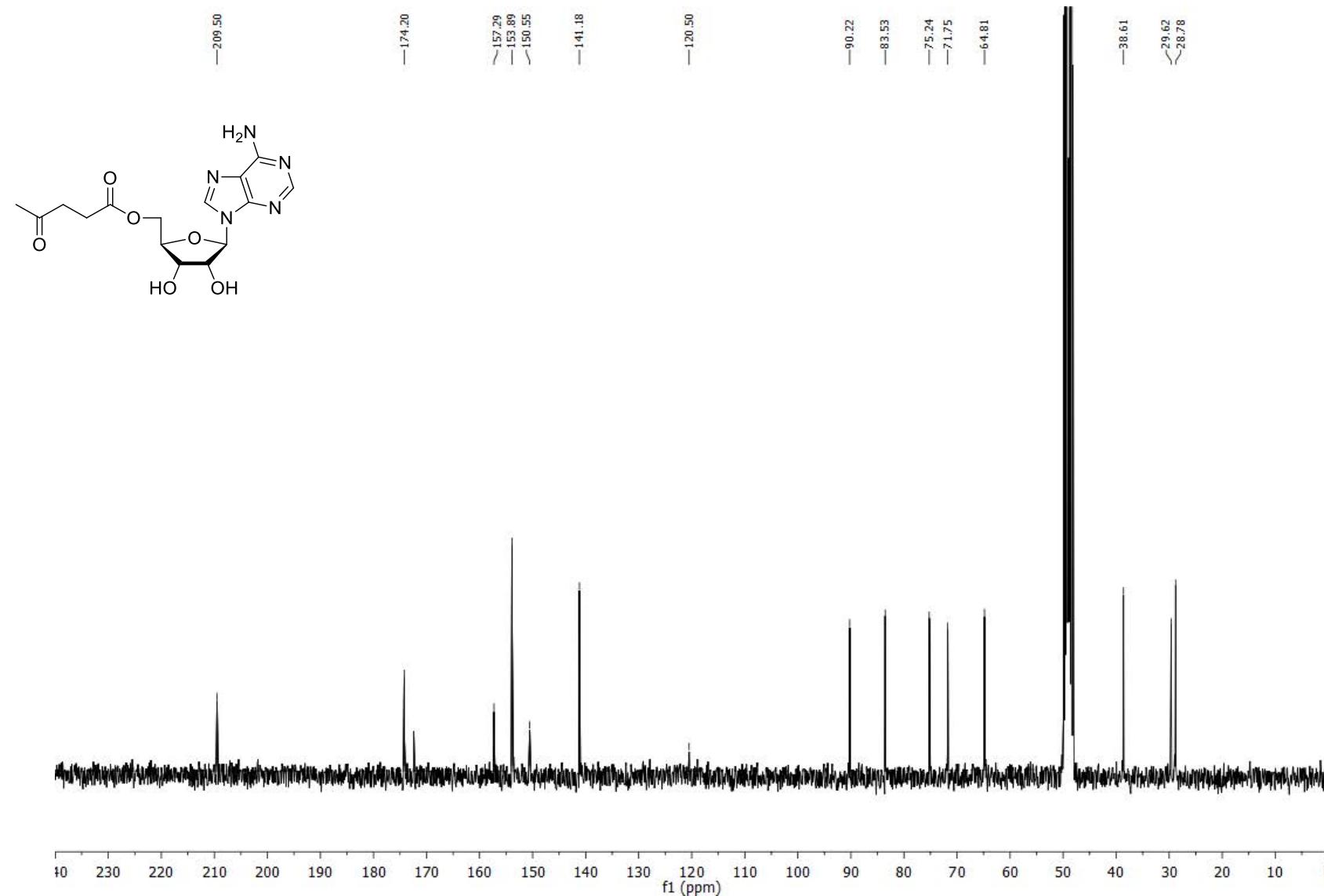
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$^1\text{H}$ -NMR (300.13 MHz, MeOD- $d_4$ )



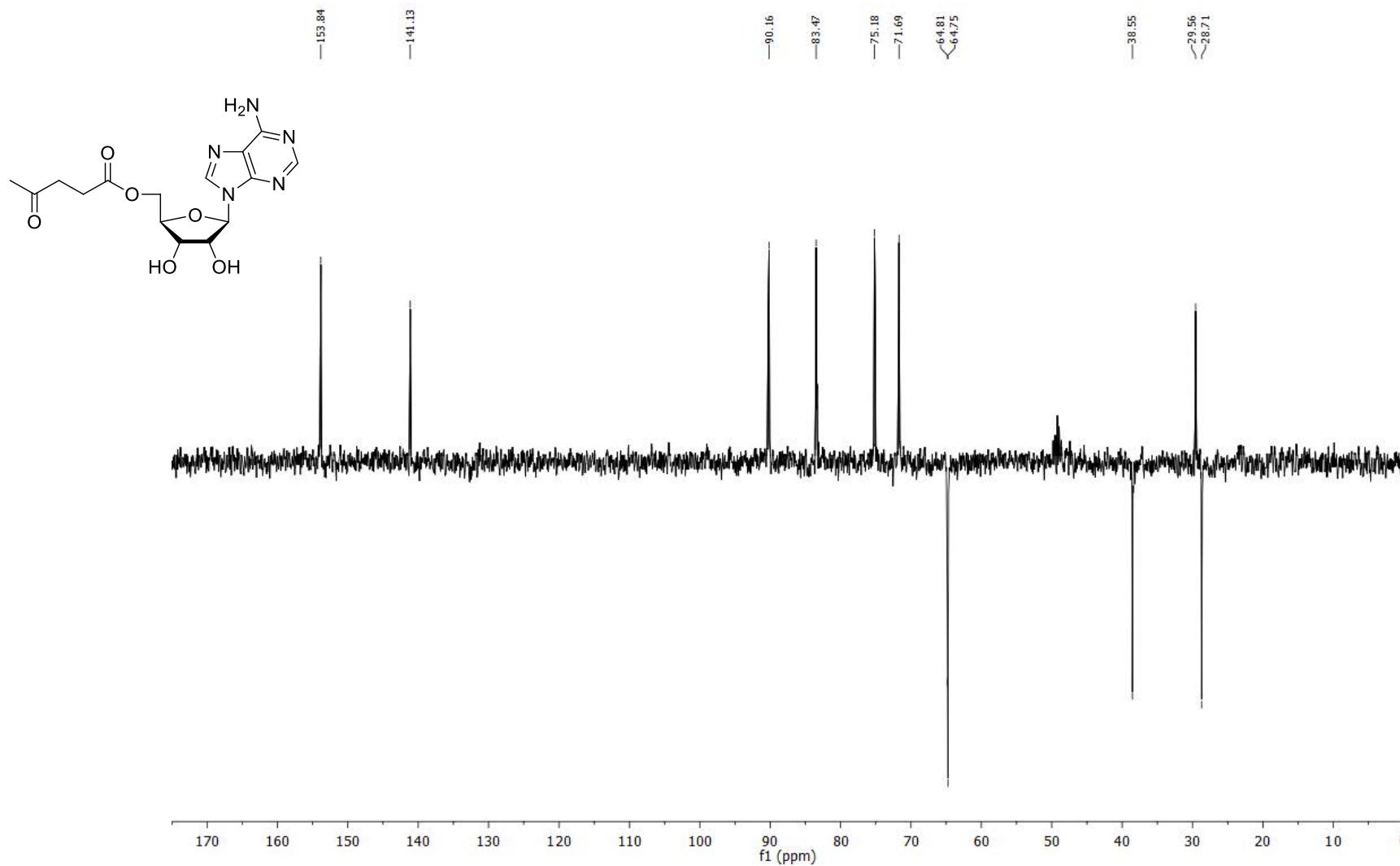
**5'-O-Levulinyl- $\beta$ -D-adenosine (2e)**

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



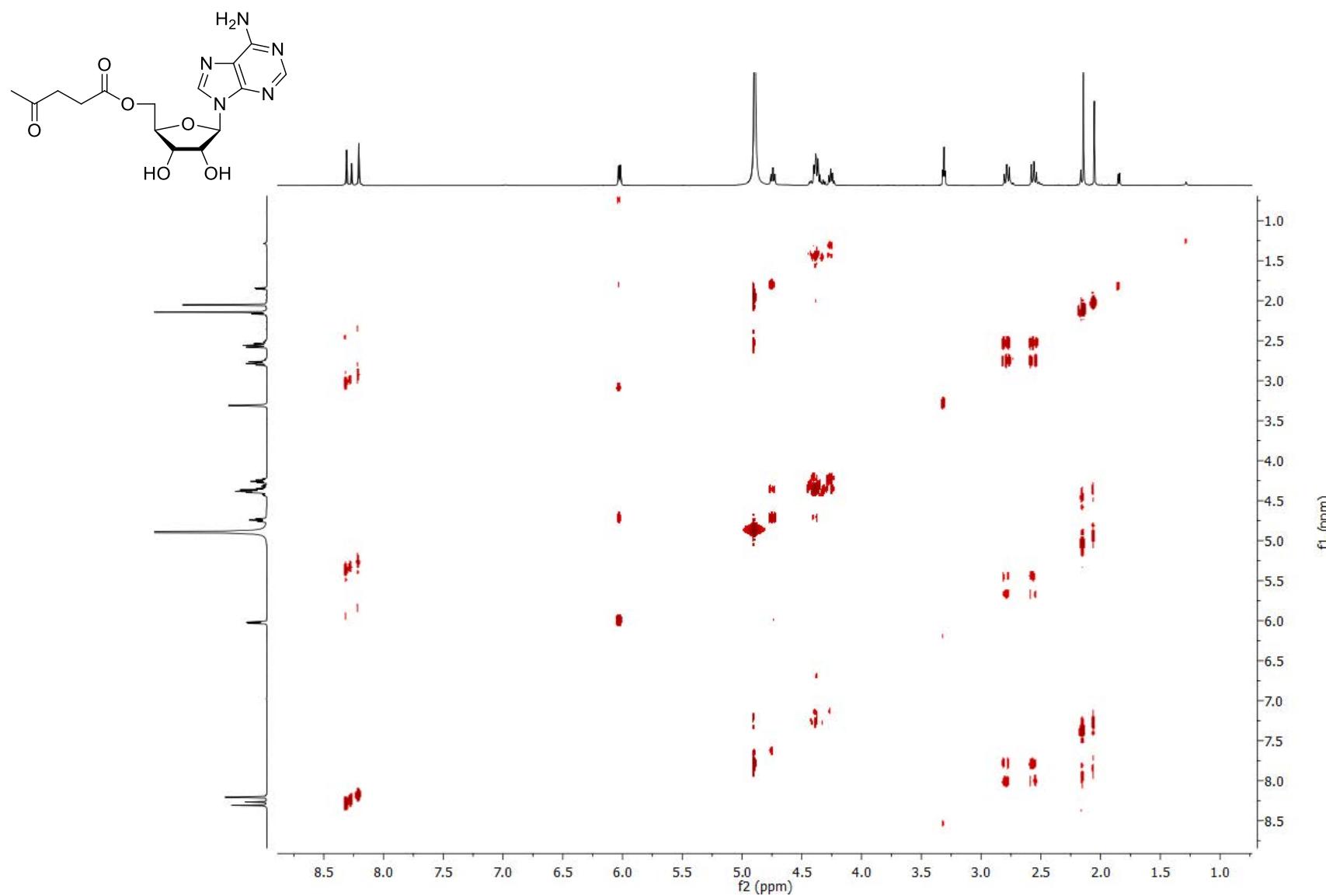
**5'-O-Levulinyl- $\beta$ -D-adenosine (2e)**

DEPT NMR (75.5 MHz, MeOD- $d_4$ )



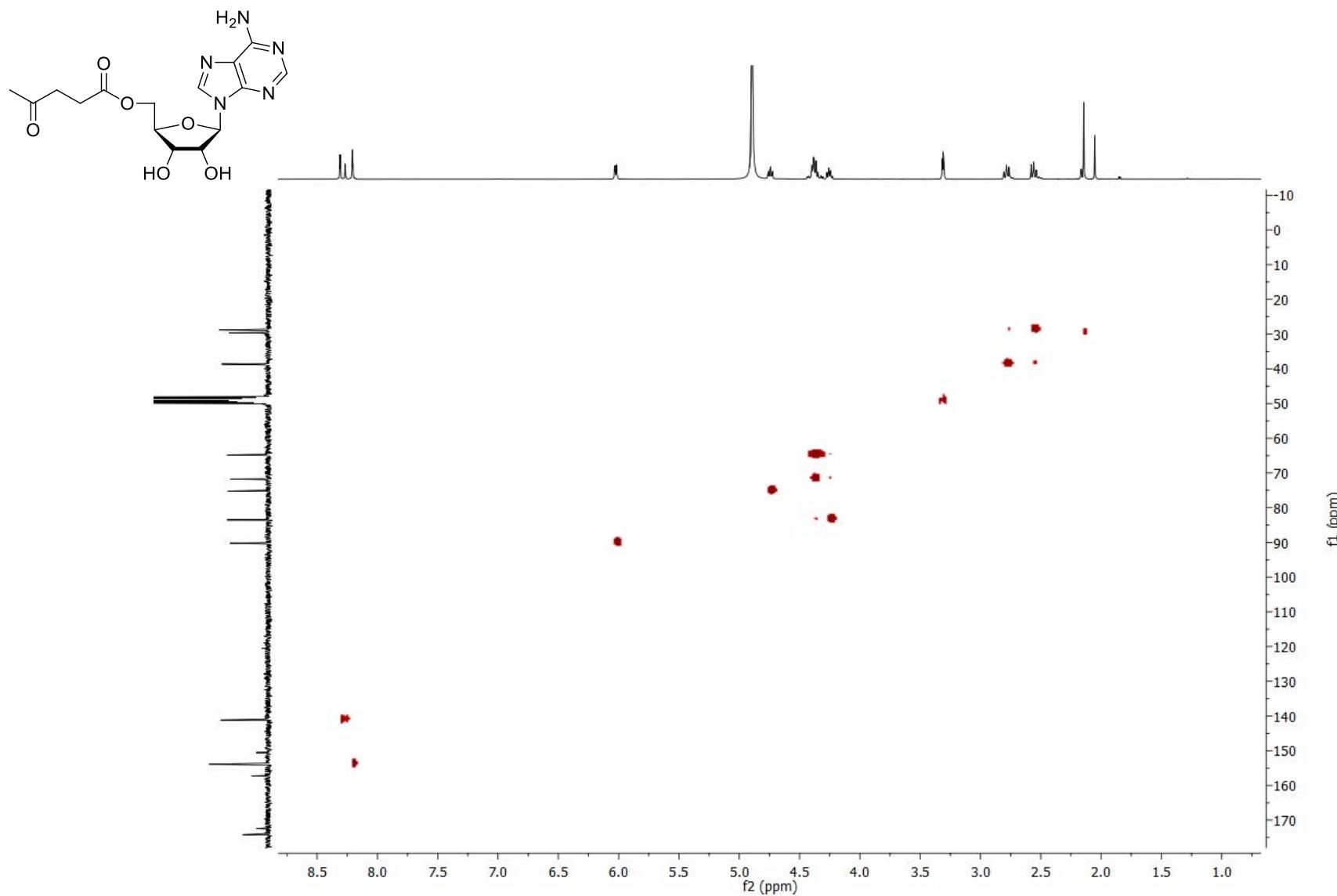
**5'-O-Levulinyl- $\beta$ -D-adenosine (2e)**

COSY NMR (MeOD-*d*<sub>4</sub>)



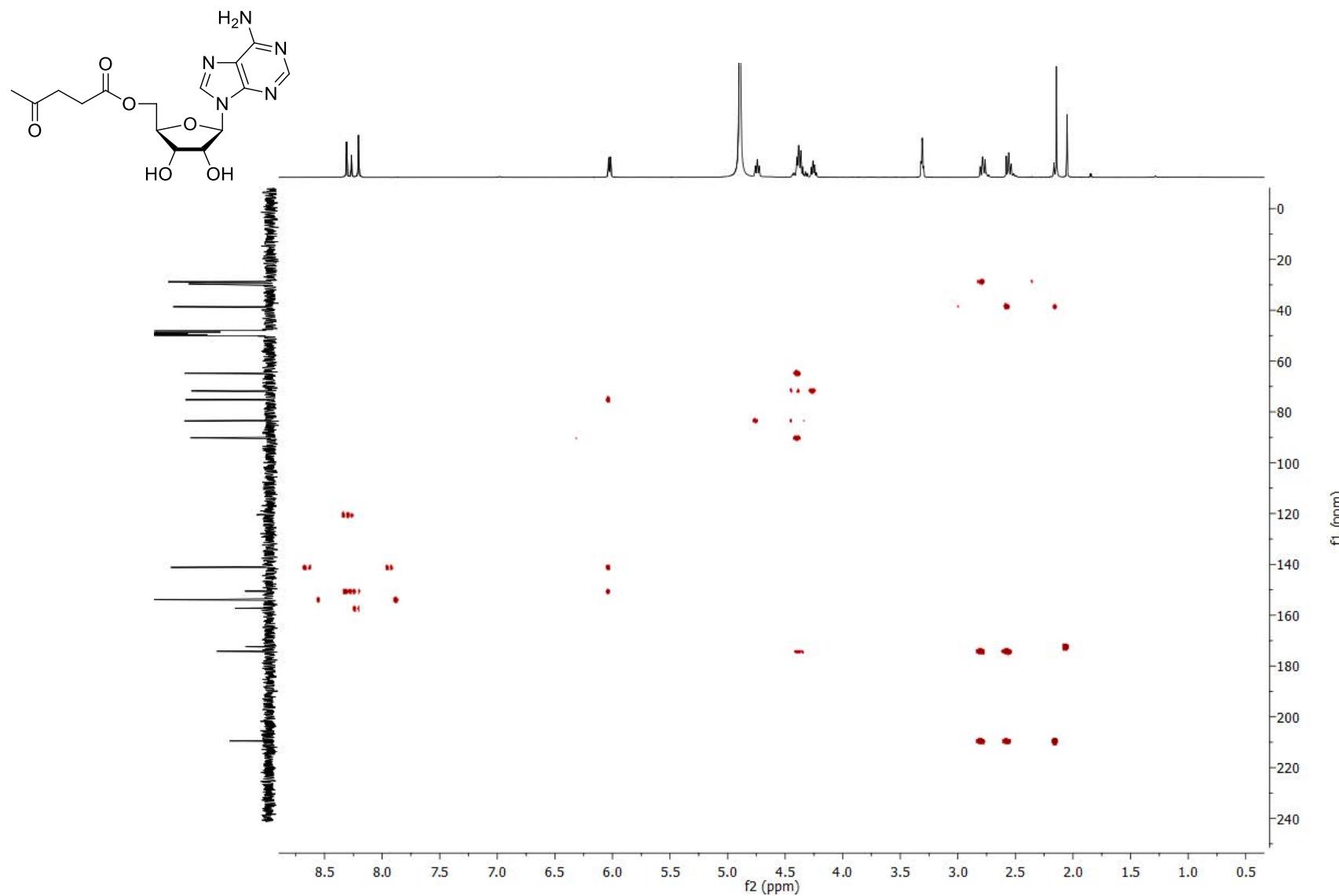
**5'-O-Levulinyl- $\beta$ -D-adenosine (2e)**

HSQC NMR ( $\text{MeOD}-d_4$ )



**5'-O-Levulinyl- $\beta$ -D-adenosine (2e)**

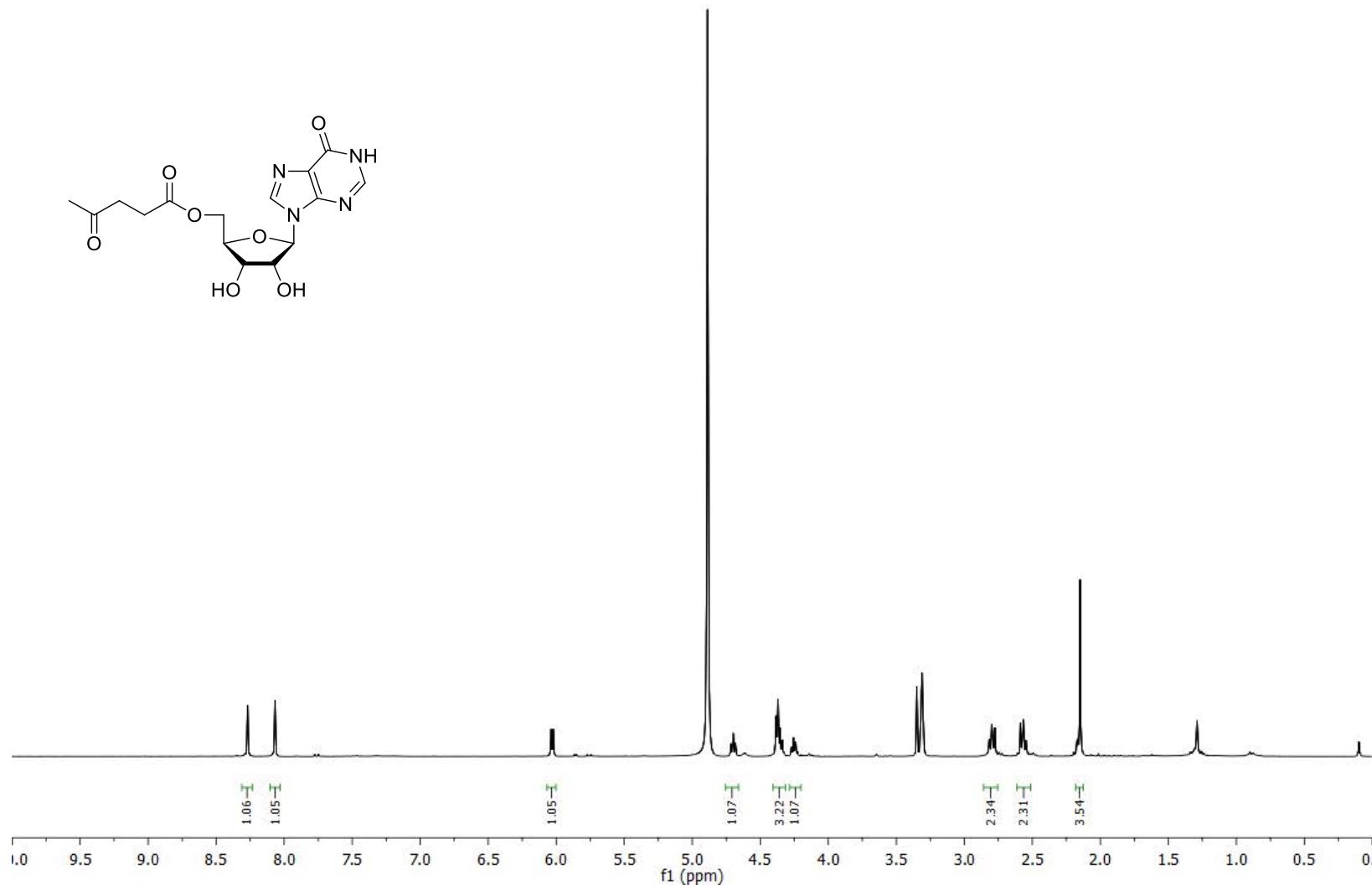
HMBC NMR ( $\text{MeOD}-d_4$ )



**5'-O-Levulinyl- $\beta$ -D-inosine (2f)**

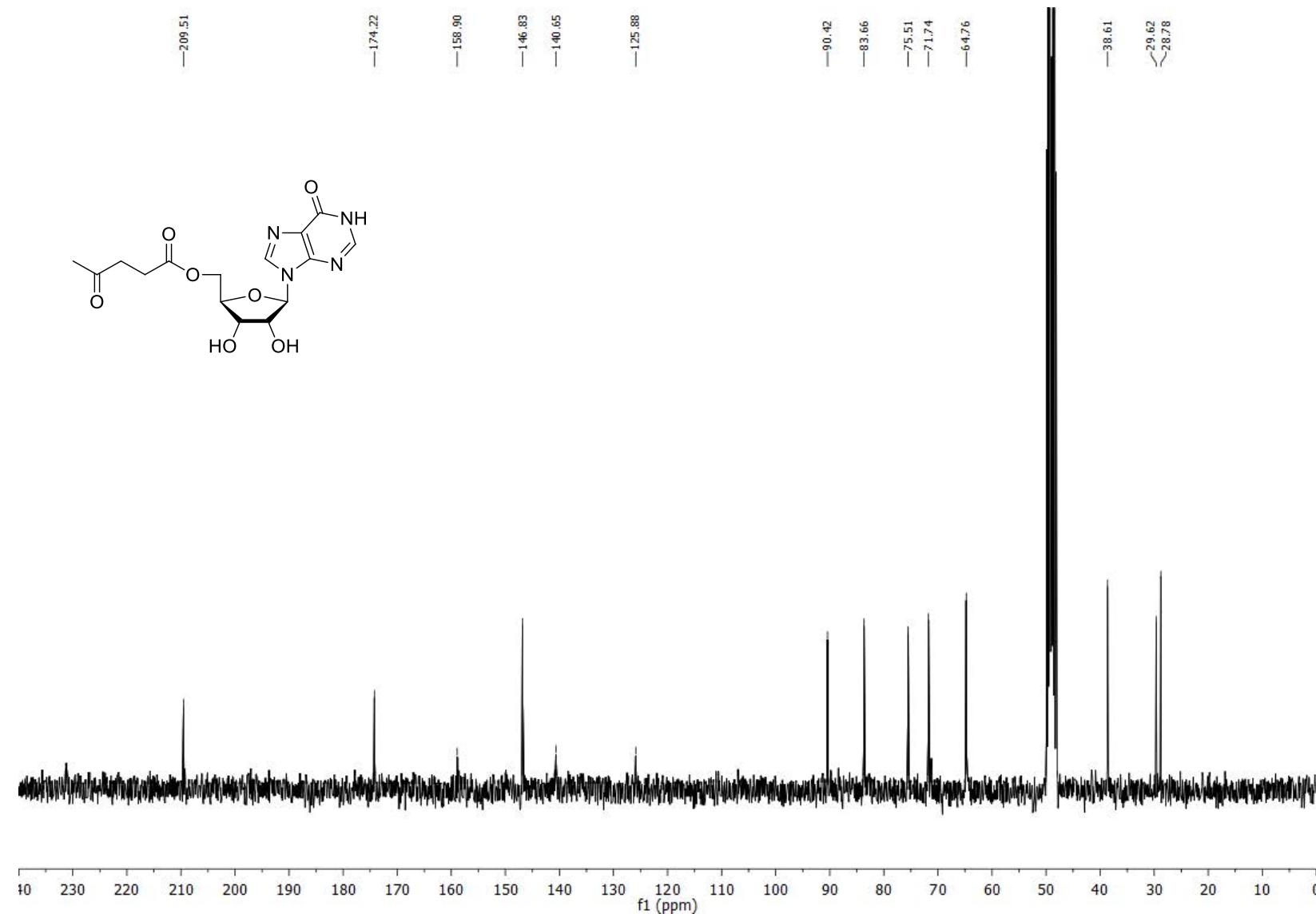
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$^1\text{H}$ -NMR (300.13 MHz, MeOD- $d_4$ )



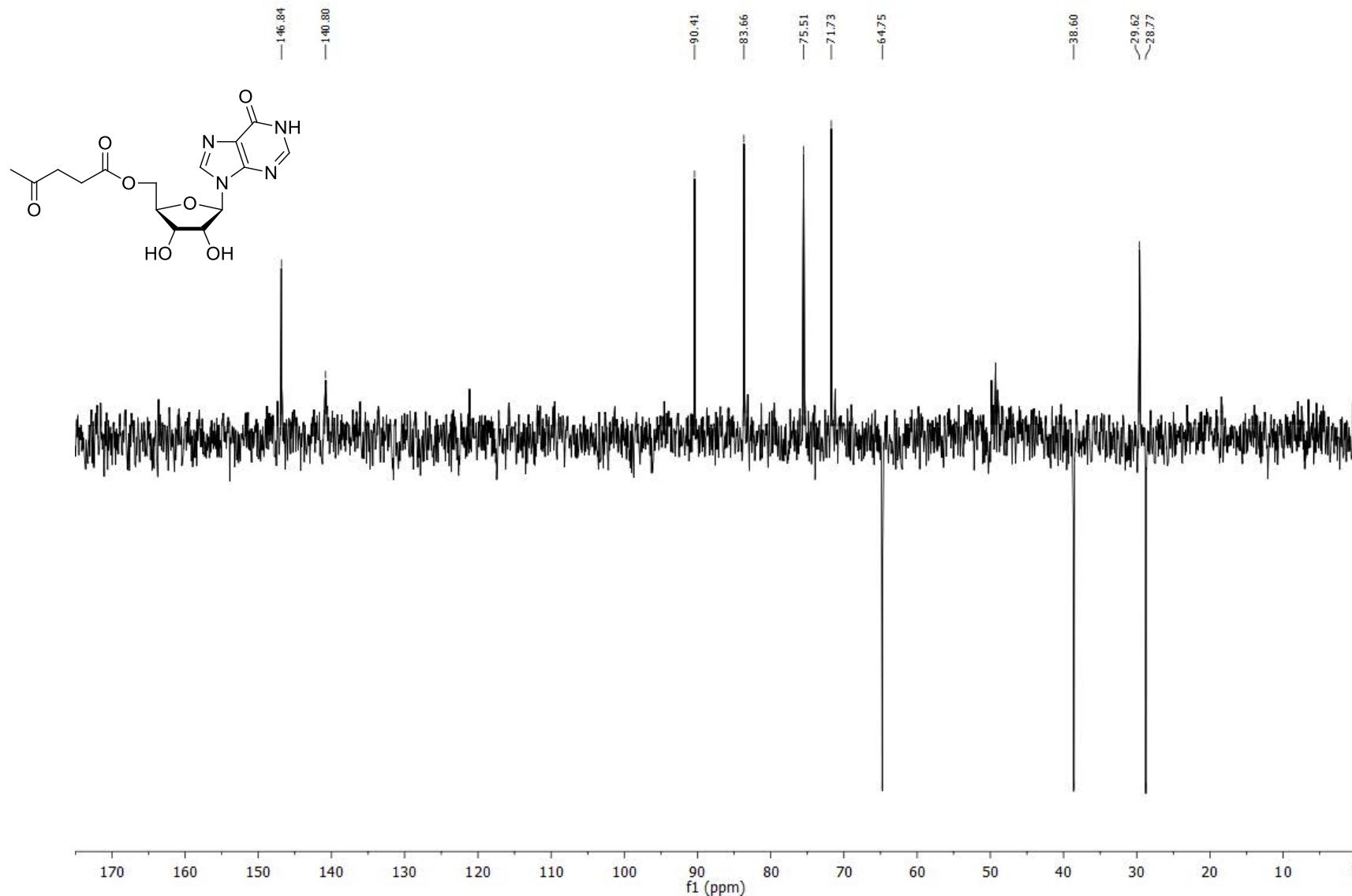
**5'-O-Levulinyl- $\beta$ -D-inosine (2f)**

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



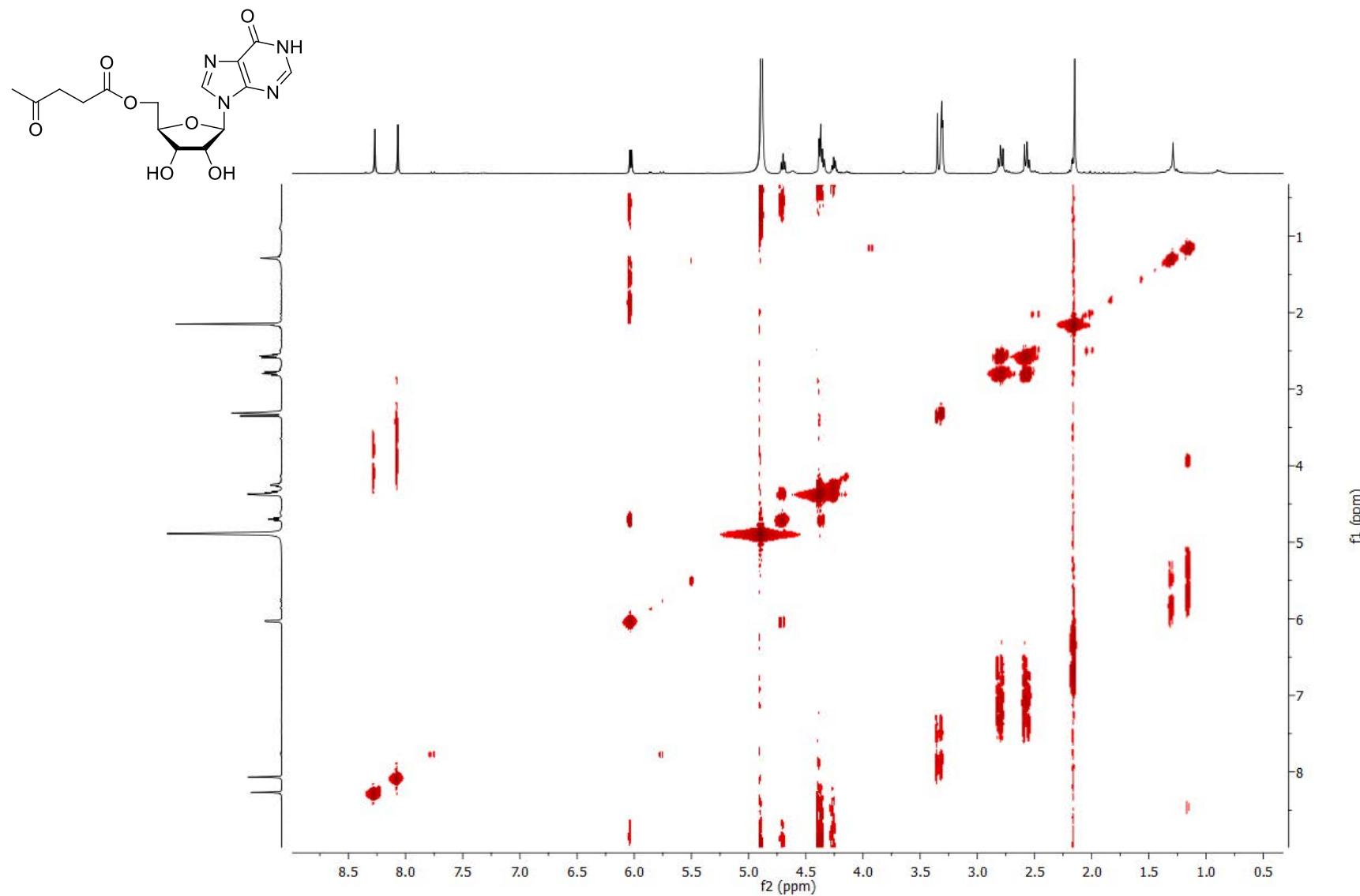
**5'-O-Levulinyl- $\beta$ -D-inosine (2f)**

DEPT NMR (75.5 MHz, MeOD-*d*<sub>4</sub>)



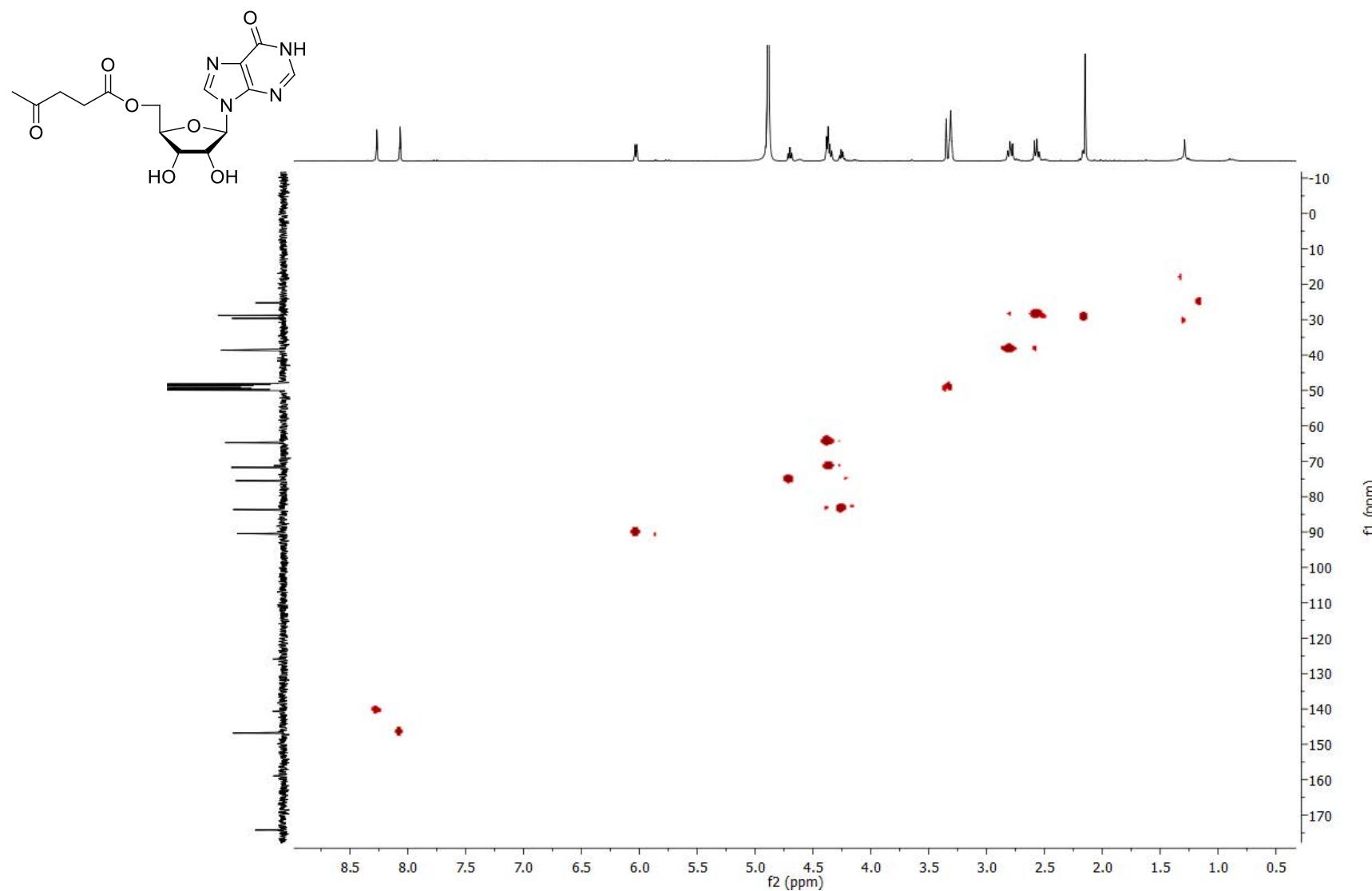
**5'-O-Levulinyl- $\beta$ -D-inosine (2f)**

COSY NMR (MeOD-*d*<sub>4</sub>)



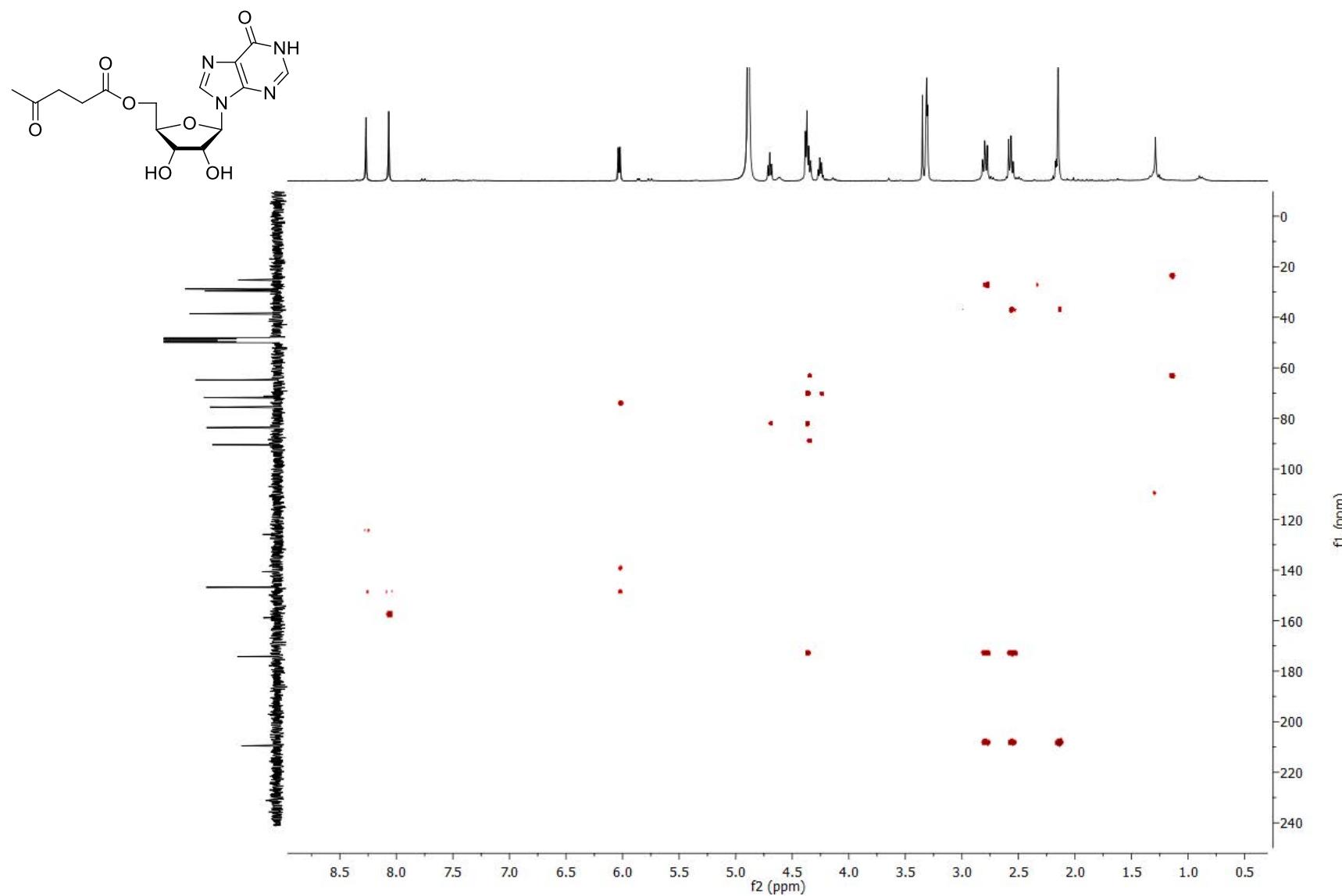
**5'-O-Levulinyl- $\beta$ -D-inosine (2f)**

HSQC NMR ( $\text{MeOD}-d_4$ )



**5'-O-Levulinyl- $\beta$ -D-inosine (2f)**

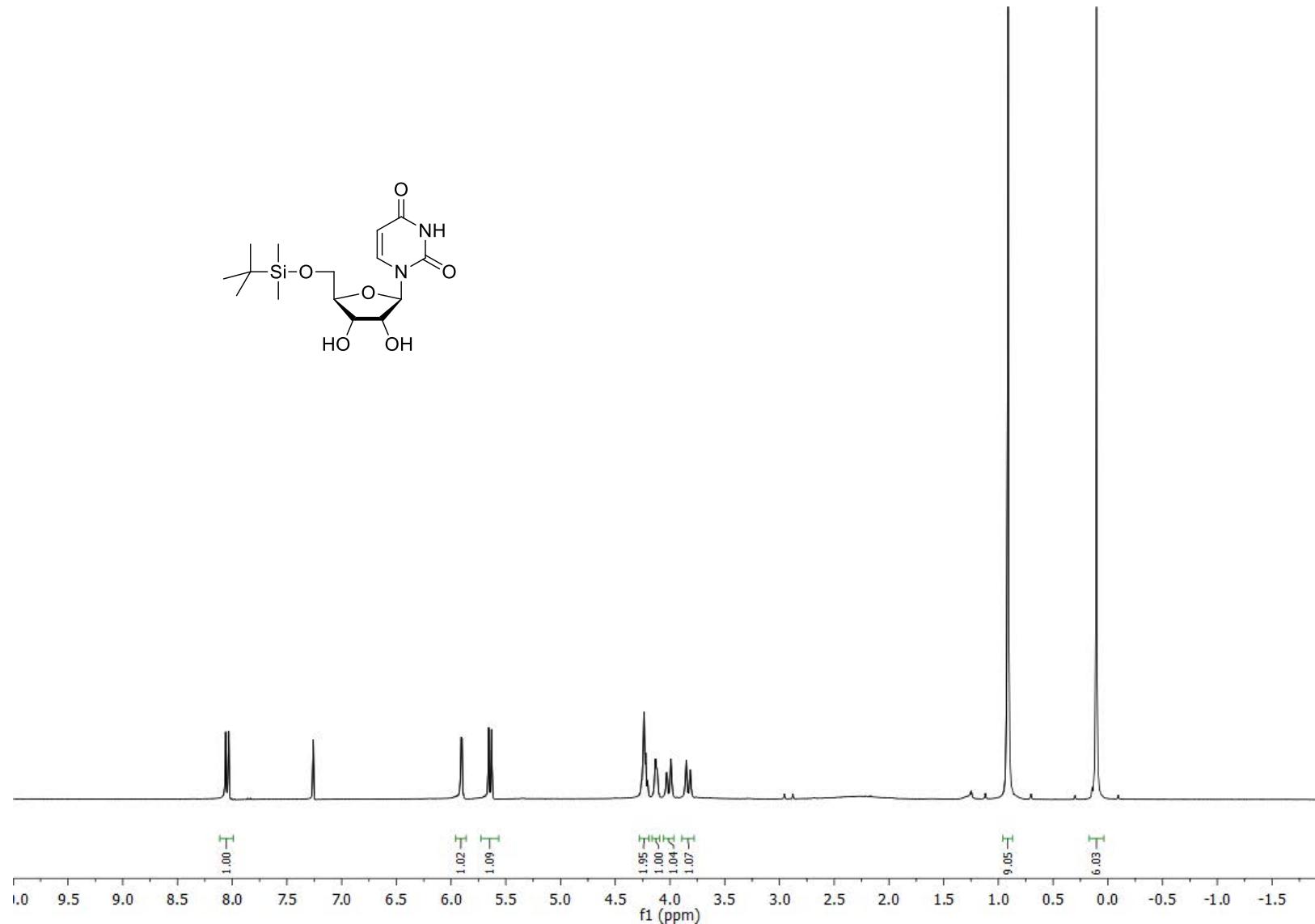
HMBC NMR ( $\text{MeOD}-d_4$ )



**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -D-uridine (**5a**)**

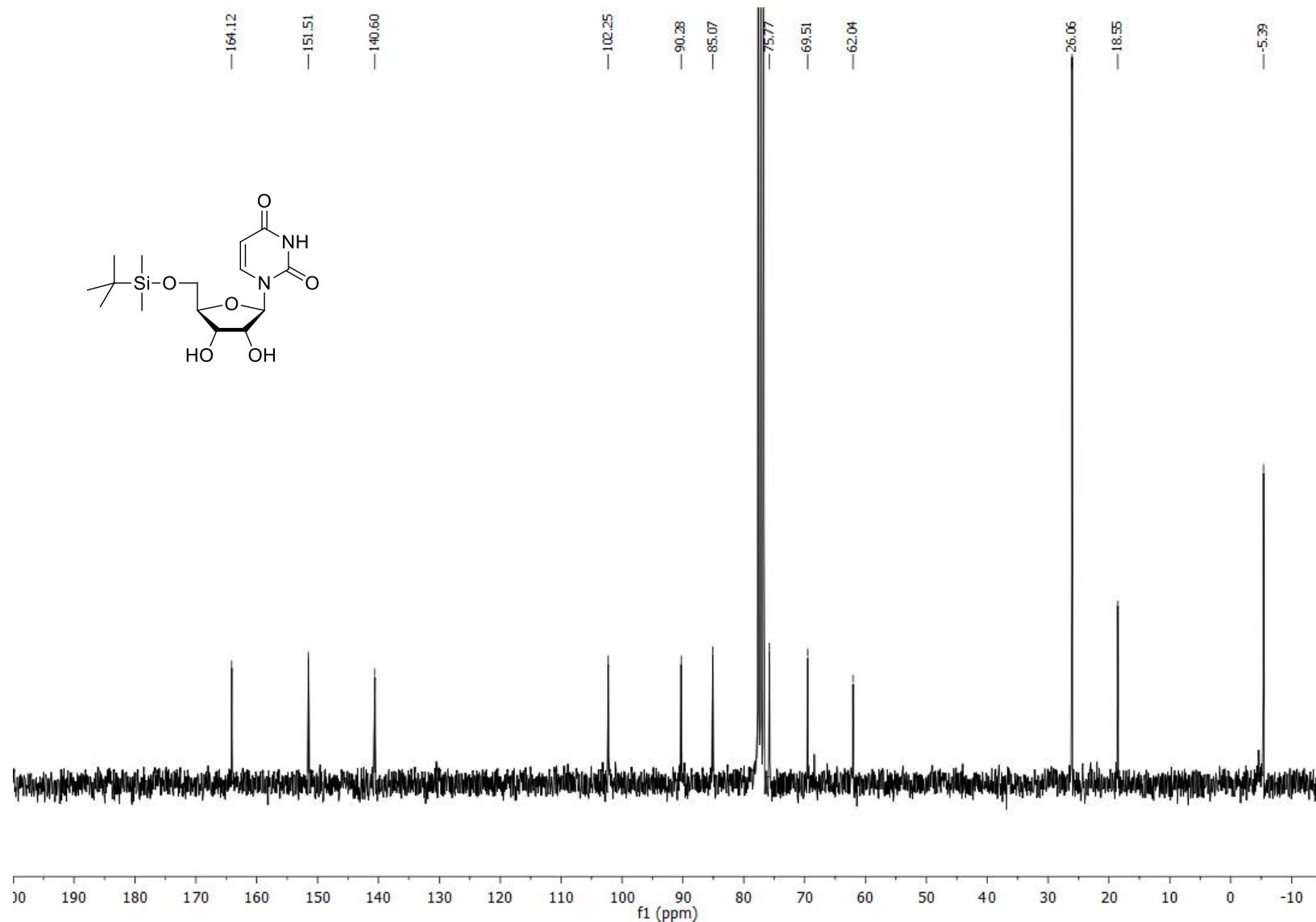
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$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -D-uridine (**5a**)**

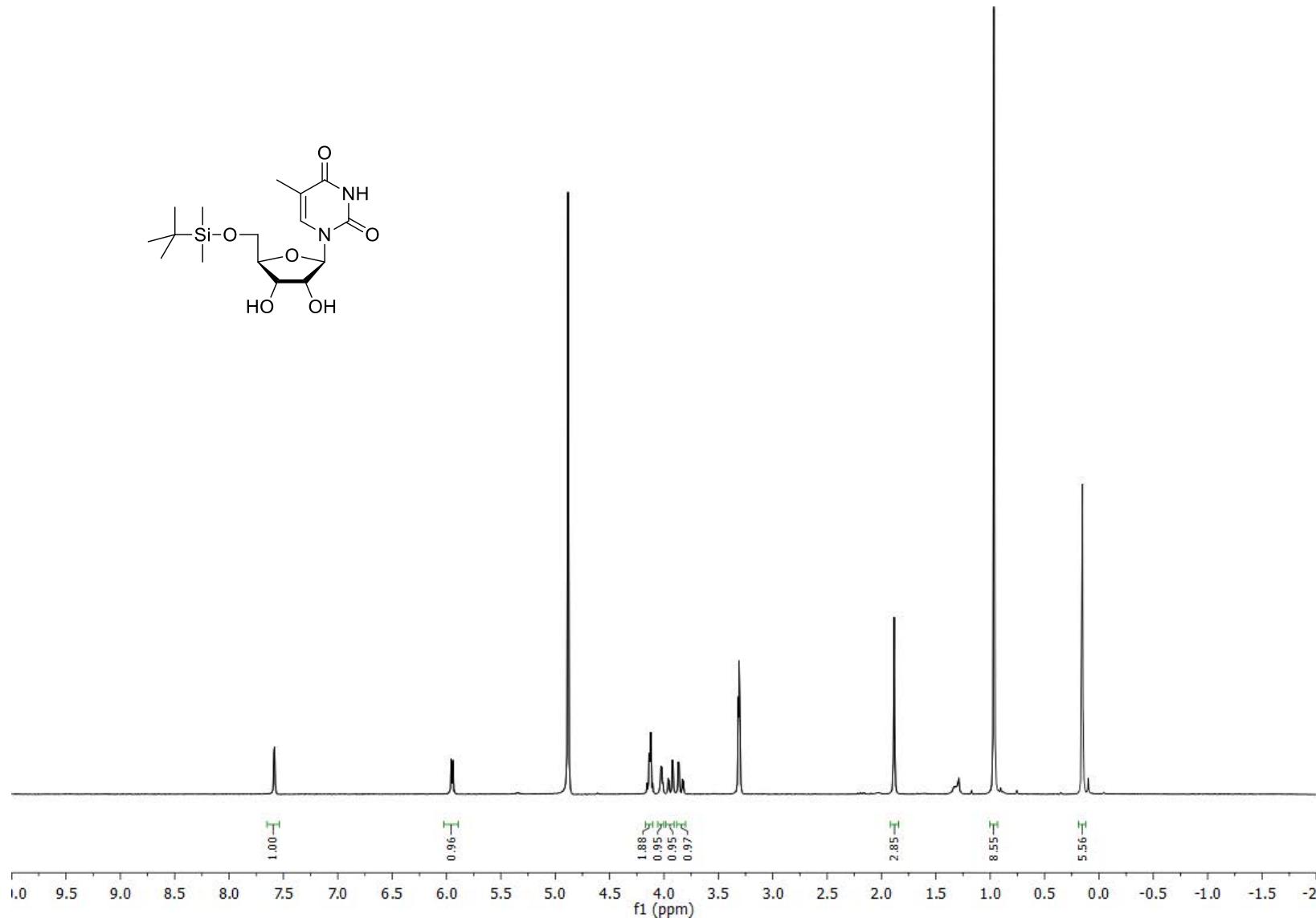
$^{13}\text{C}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -D-5-methyluridine (5b)**

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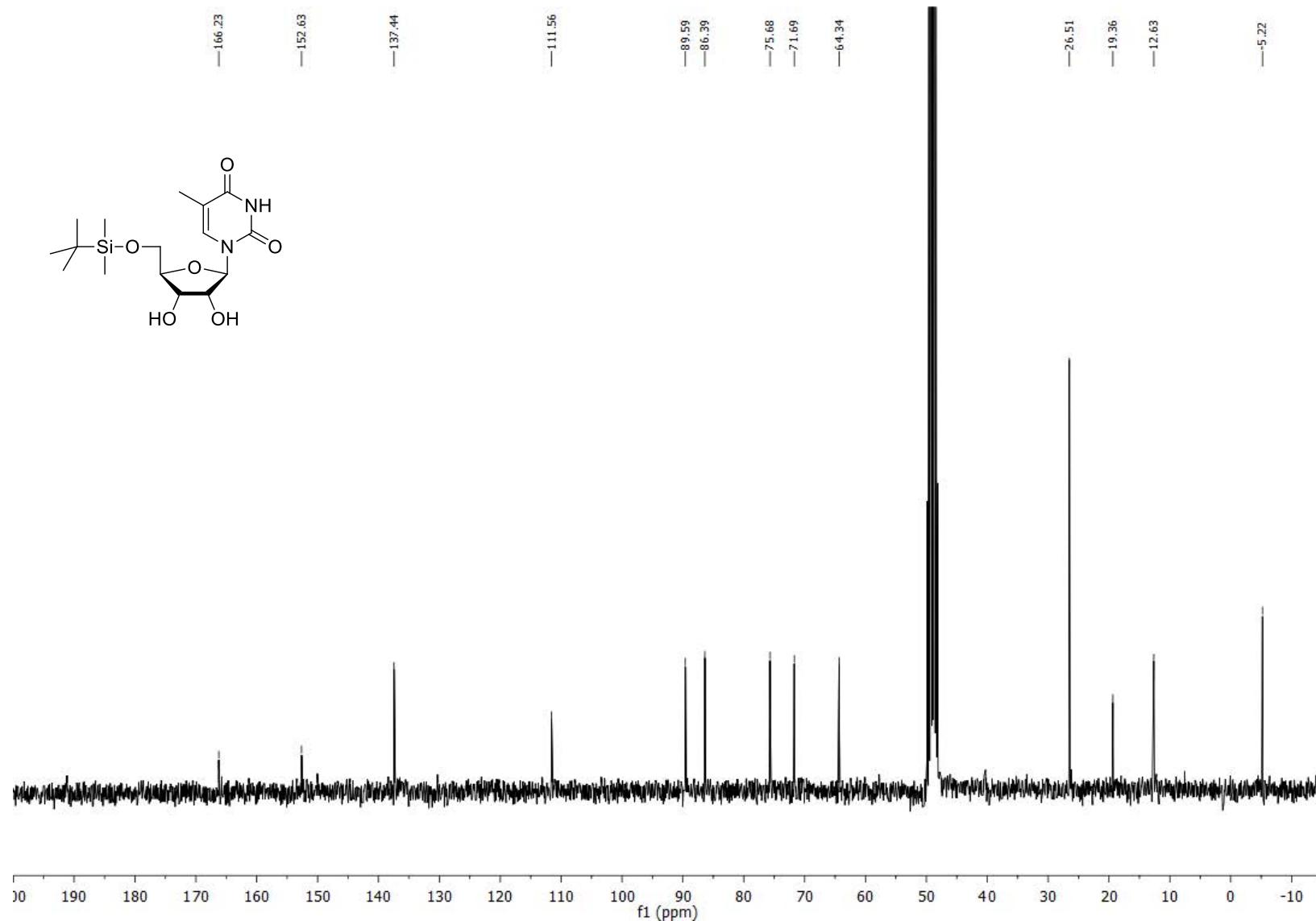
$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -D-5-methyluridine (5b)**

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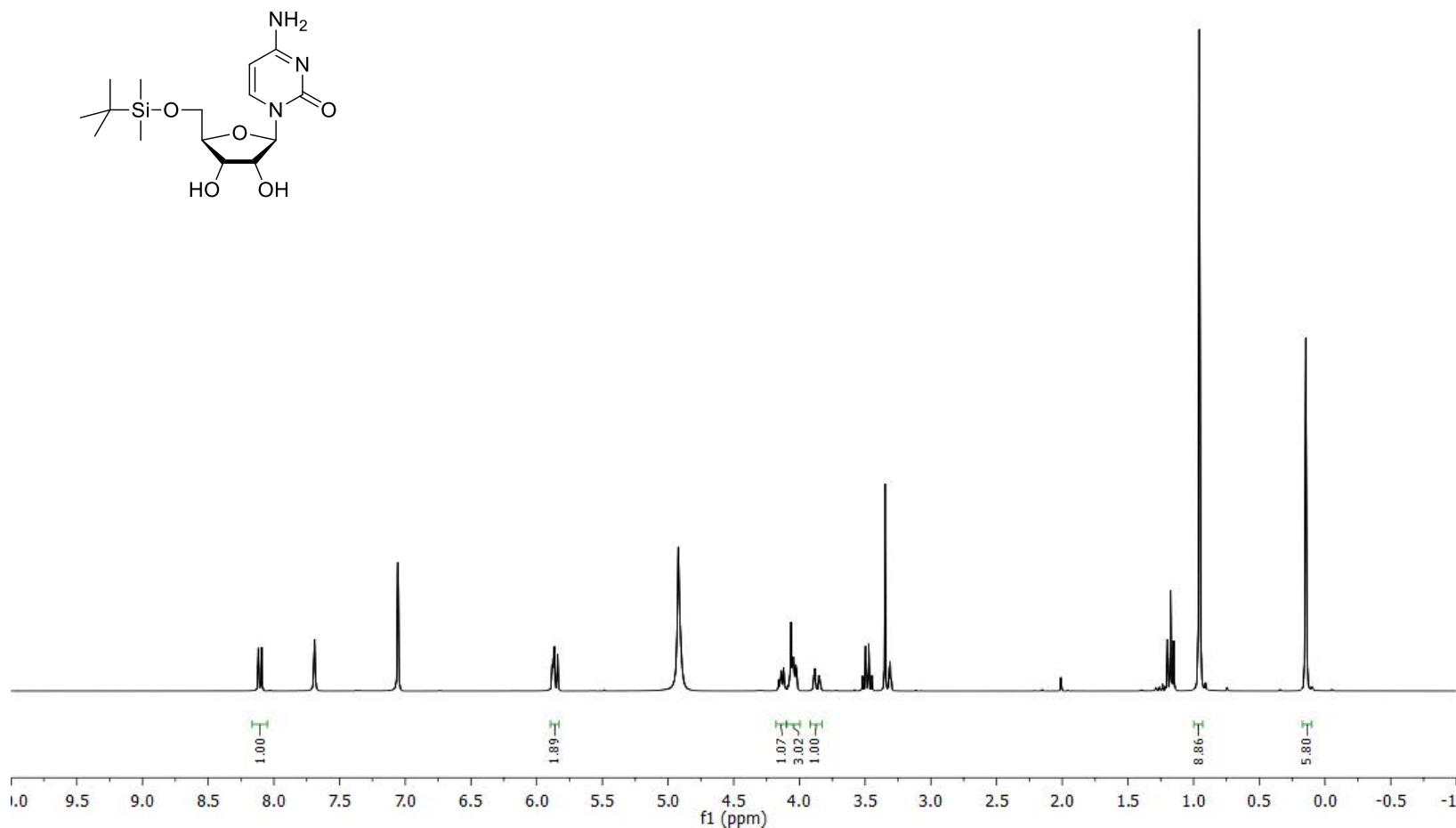
$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



**5'-O-(tert-Butyldimethylsilyl)- $\beta$ -D-cytidine (5c)**

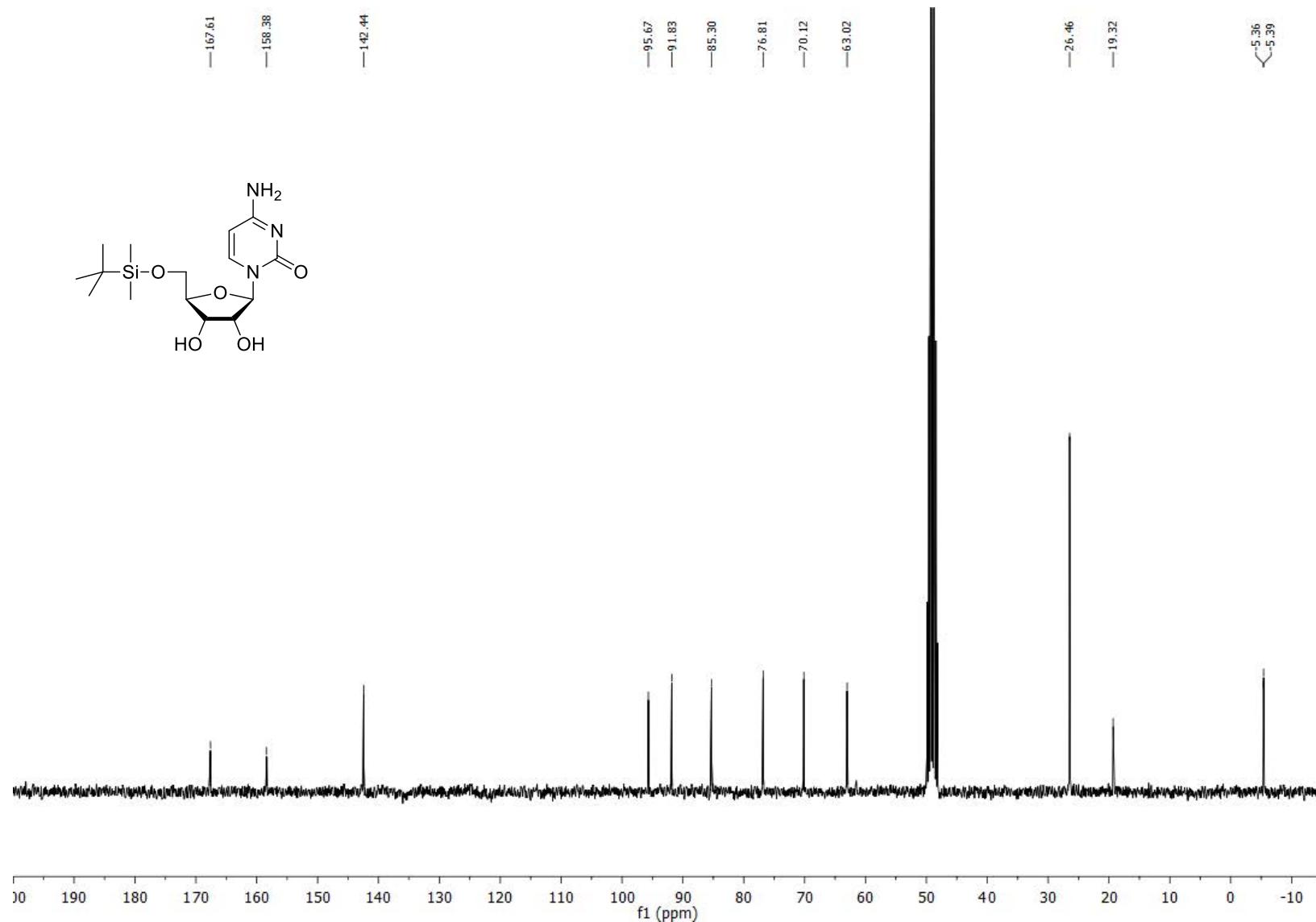
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$^1\text{H}$ -NMR (300.13 MHz, MeOH- $d_4$ )



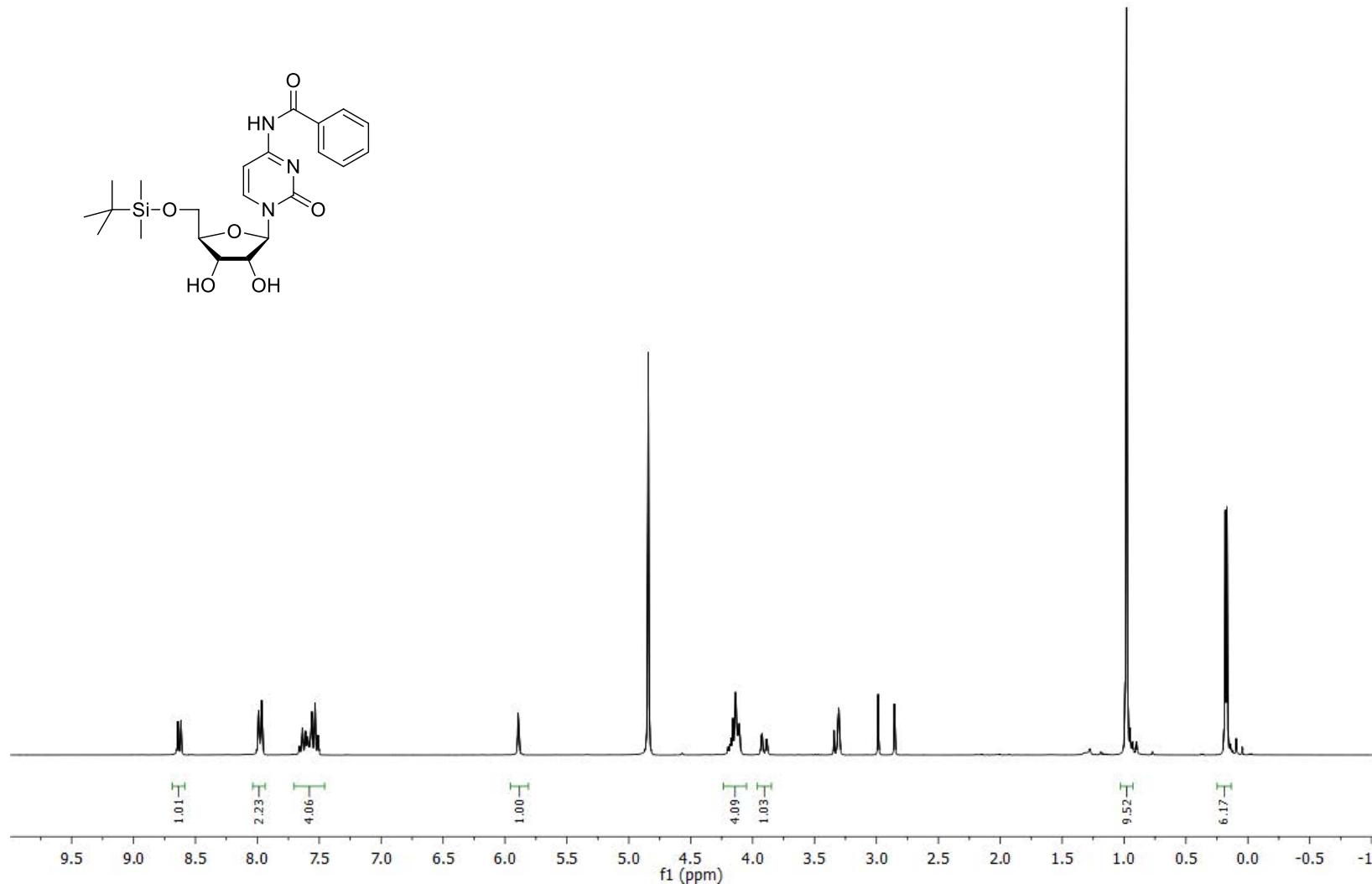
**5'-O-(tert-Butyldimethylsilyl)- $\beta$ -D-cytidine (5c)**

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



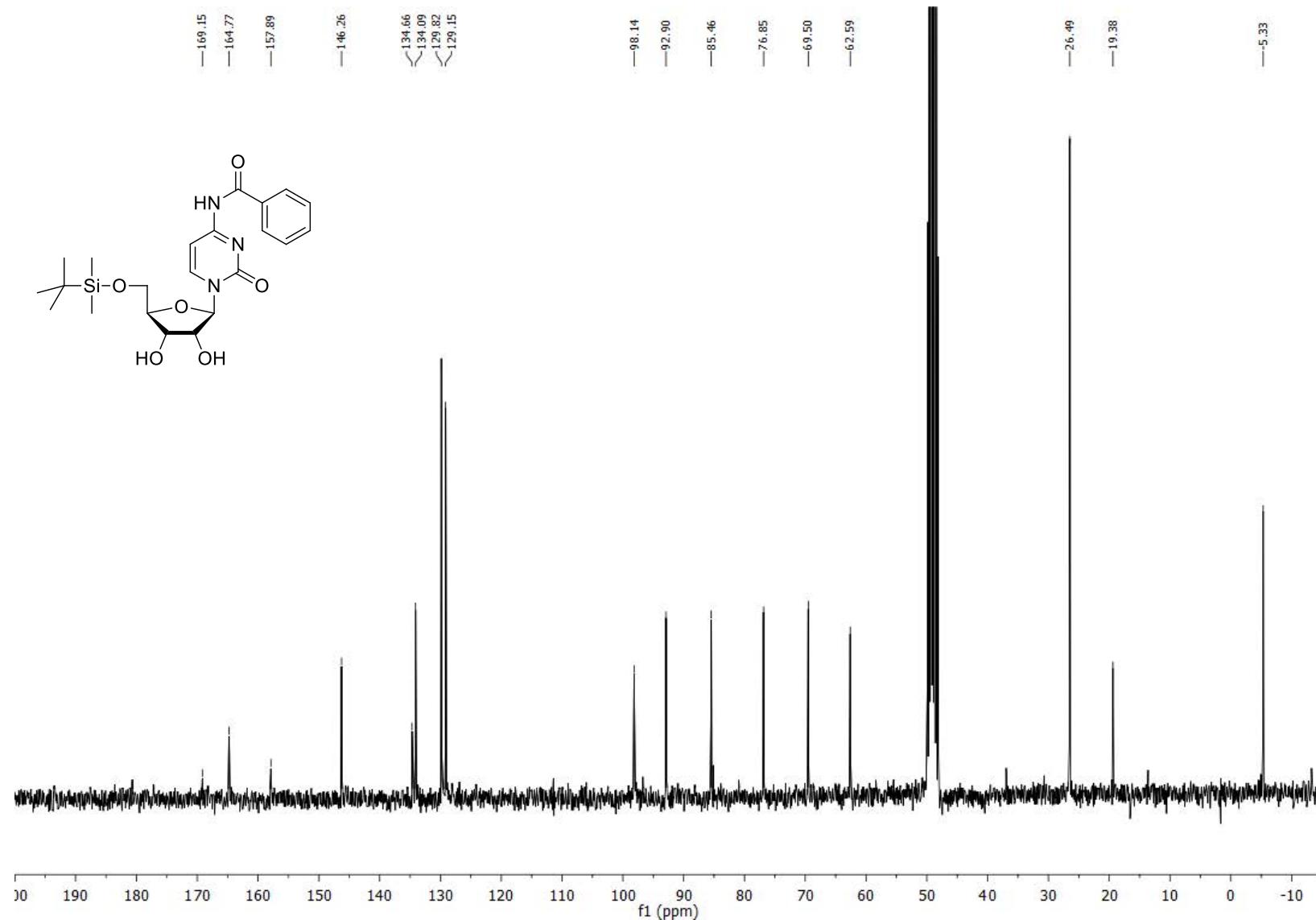
*N*<sup>4</sup>-Benzoyl-5'-O-(*tert*-butyldimethylsilyl)- $\beta$ -D-cytidine (**5d**)

<sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>)



*N*<sup>4</sup>-Benzoyl-5'-O-(*tert*-butyldimethylsilyl)- $\beta$ -D-cytidine (**5d**)

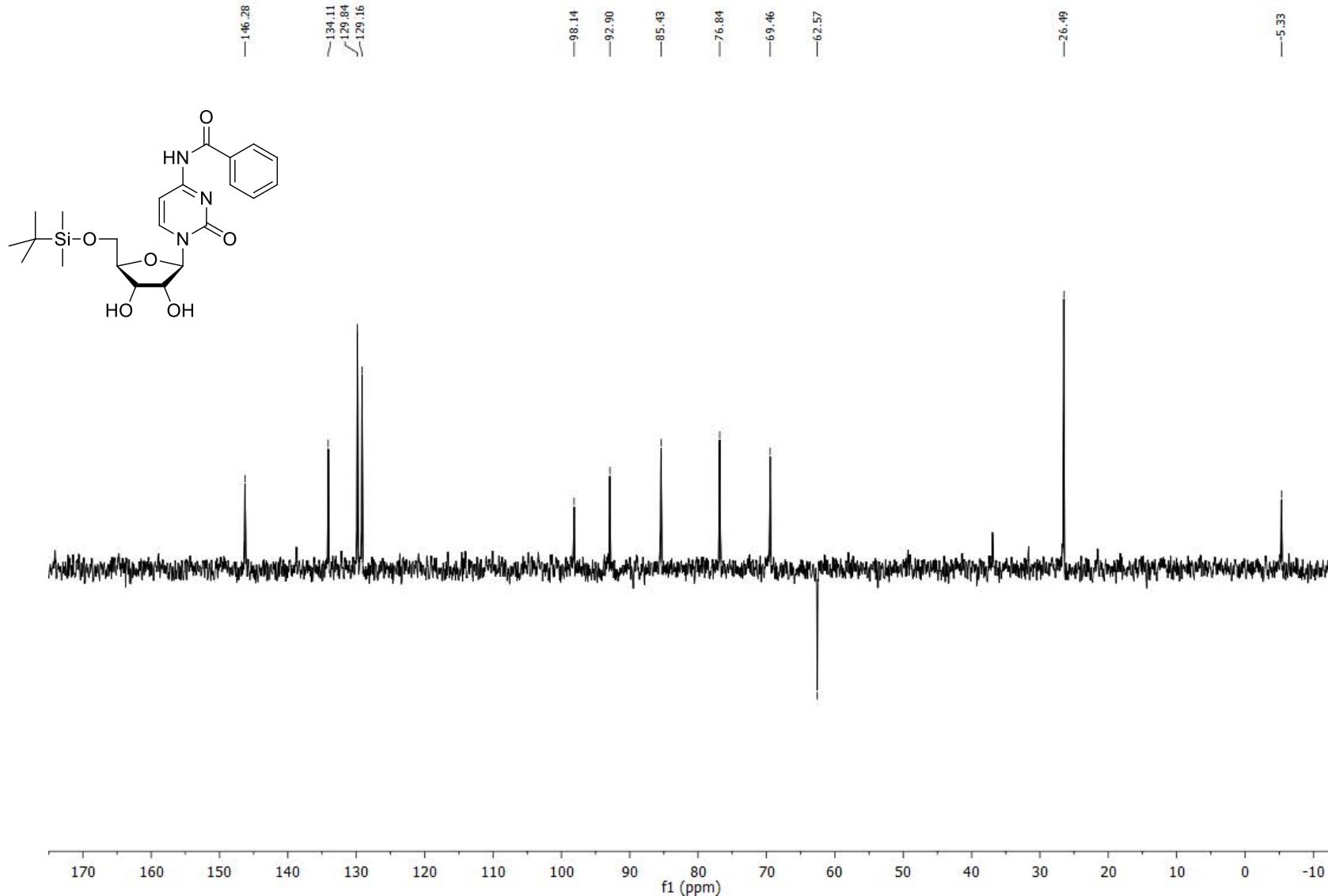
<sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>)



*N<sup>4</sup>-Benzoyl-5'-O-(tert-butyldimethylsilyl)-β-D-cytidine (5d)*

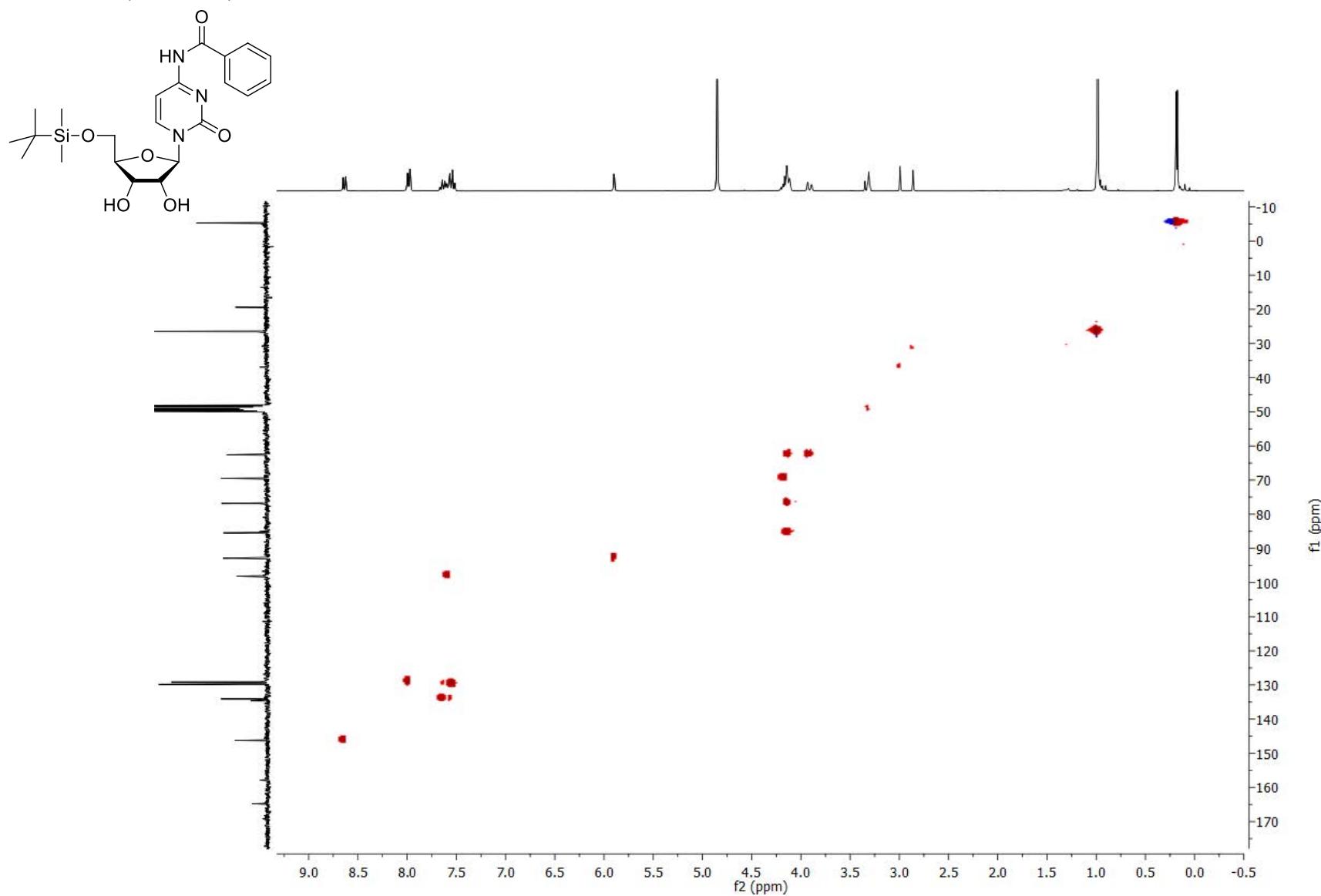
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DEPT NMR (75.5 MHz, MeOH-*d*<sub>4</sub>)



*N*<sup>4</sup>-Benzoyl-5'-O-(*tert*-butyldimethylsilyl)- $\beta$ -D-cytidine (**5d**)

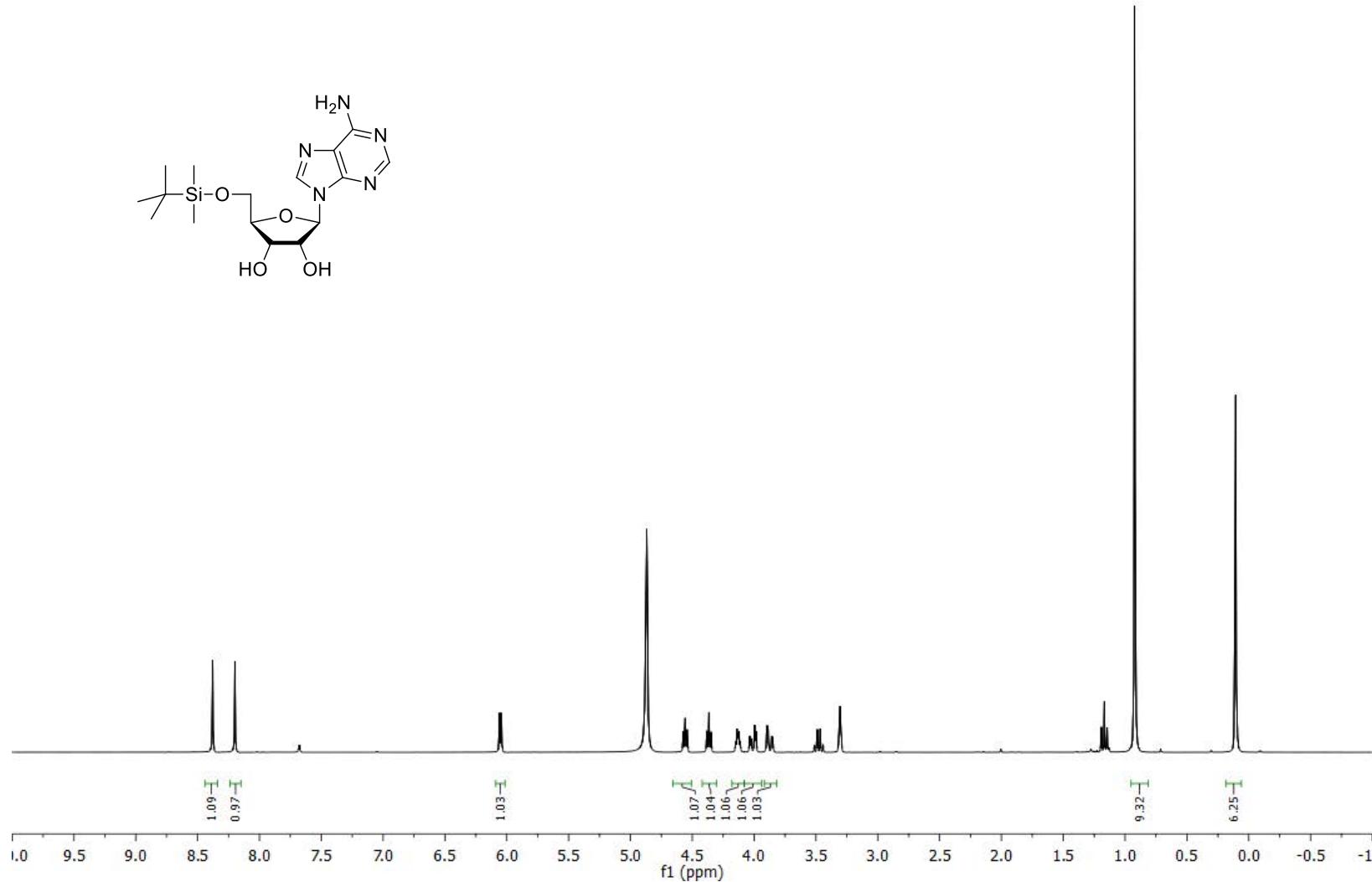
HSQC NMR (MeOH-*d*<sub>4</sub>)



**5'-O-(tert-Butyldimethylsilyl)- $\beta$ -D-adenosine (5e)**

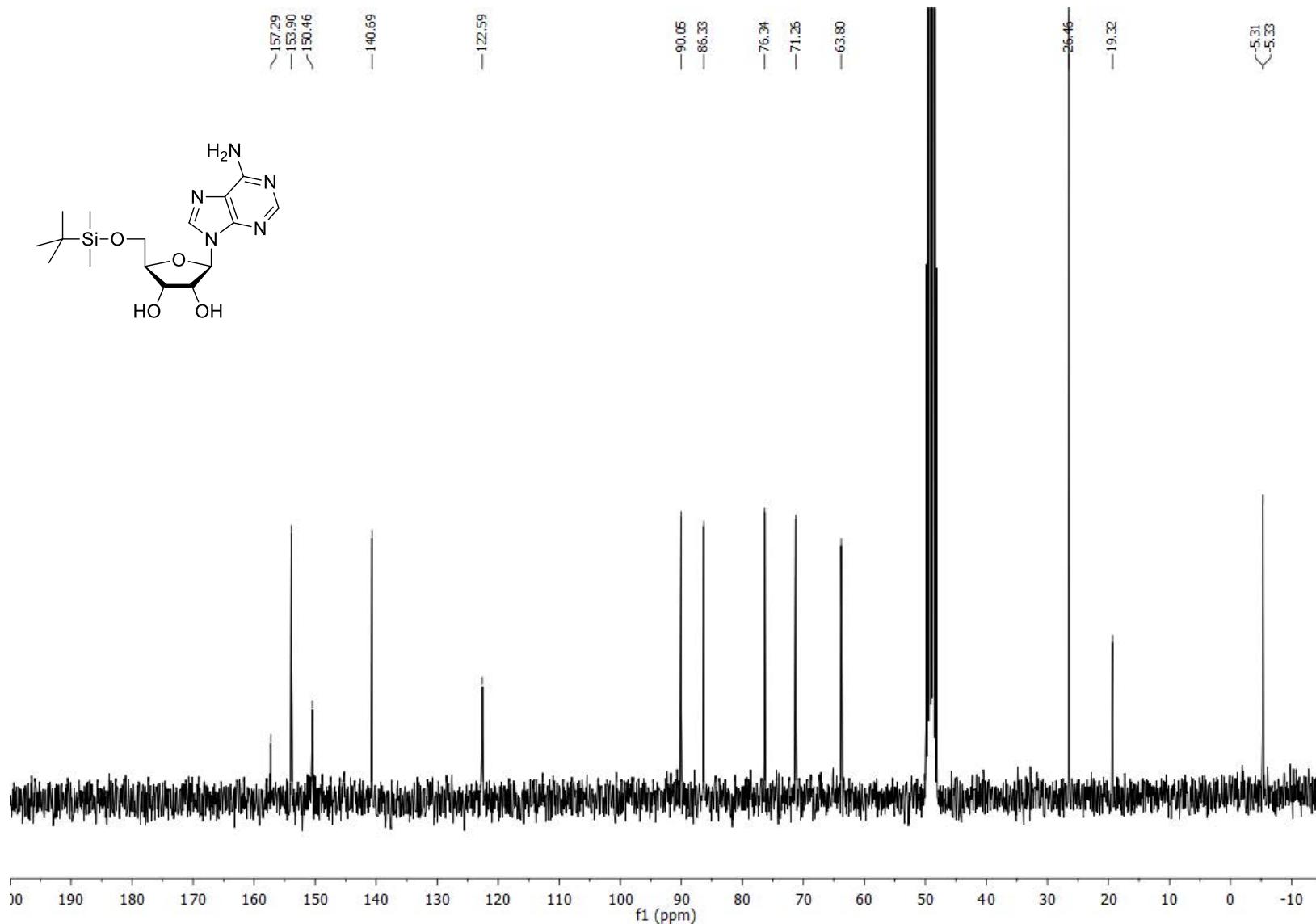
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$^1\text{H-NMR}$  (300.13 MHz, MeOH- $d_4$ )



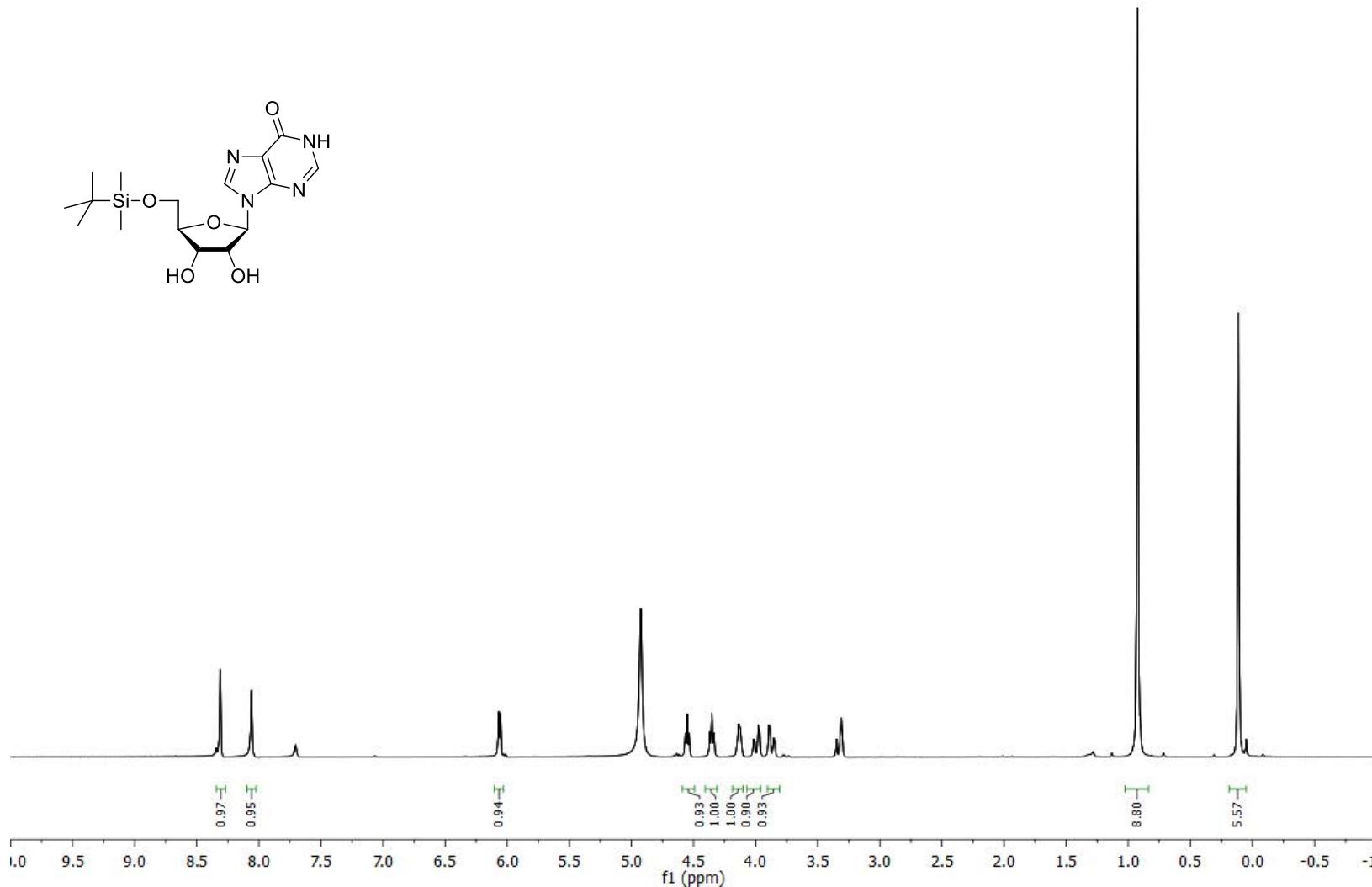
**5'-O-(tert-Butyldimethylsilyl)- $\beta$ -D-adenosine (5e)**

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



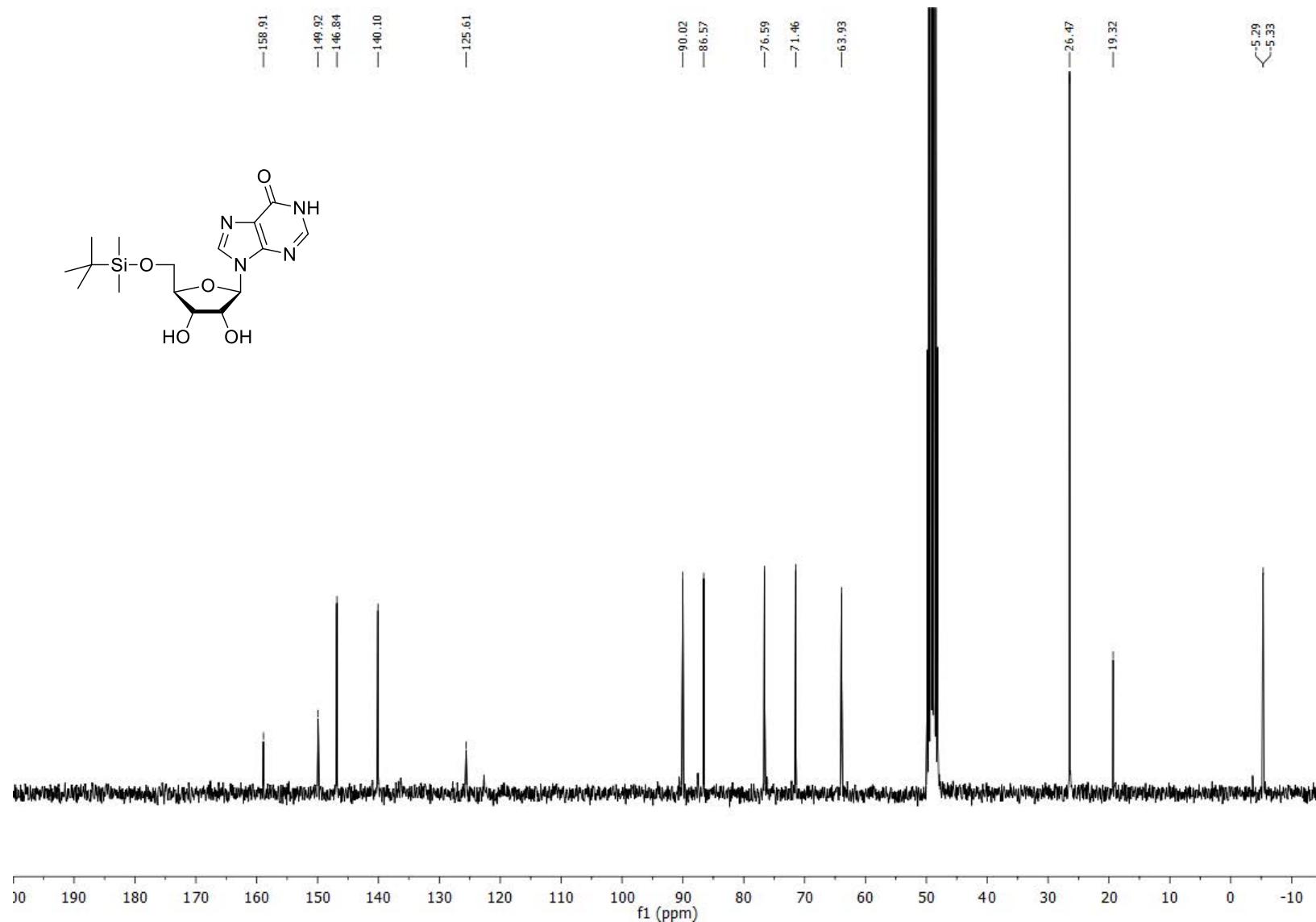
**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -D-inosine (5f)**

$^1\text{H}$ -NMR (300.13 MHz, MeOH-*d*<sub>4</sub>)



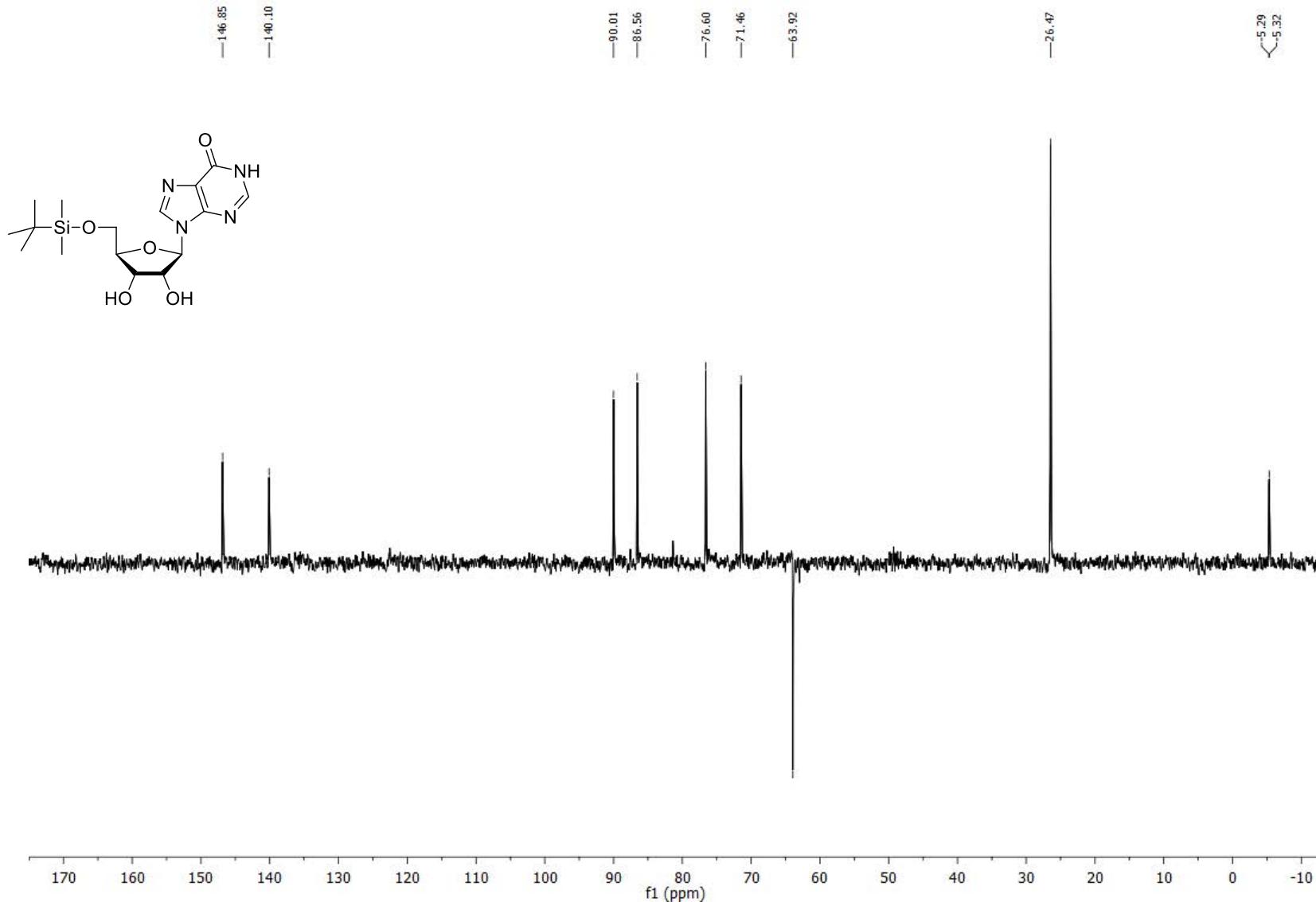
**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -D-inosine (5f)**

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



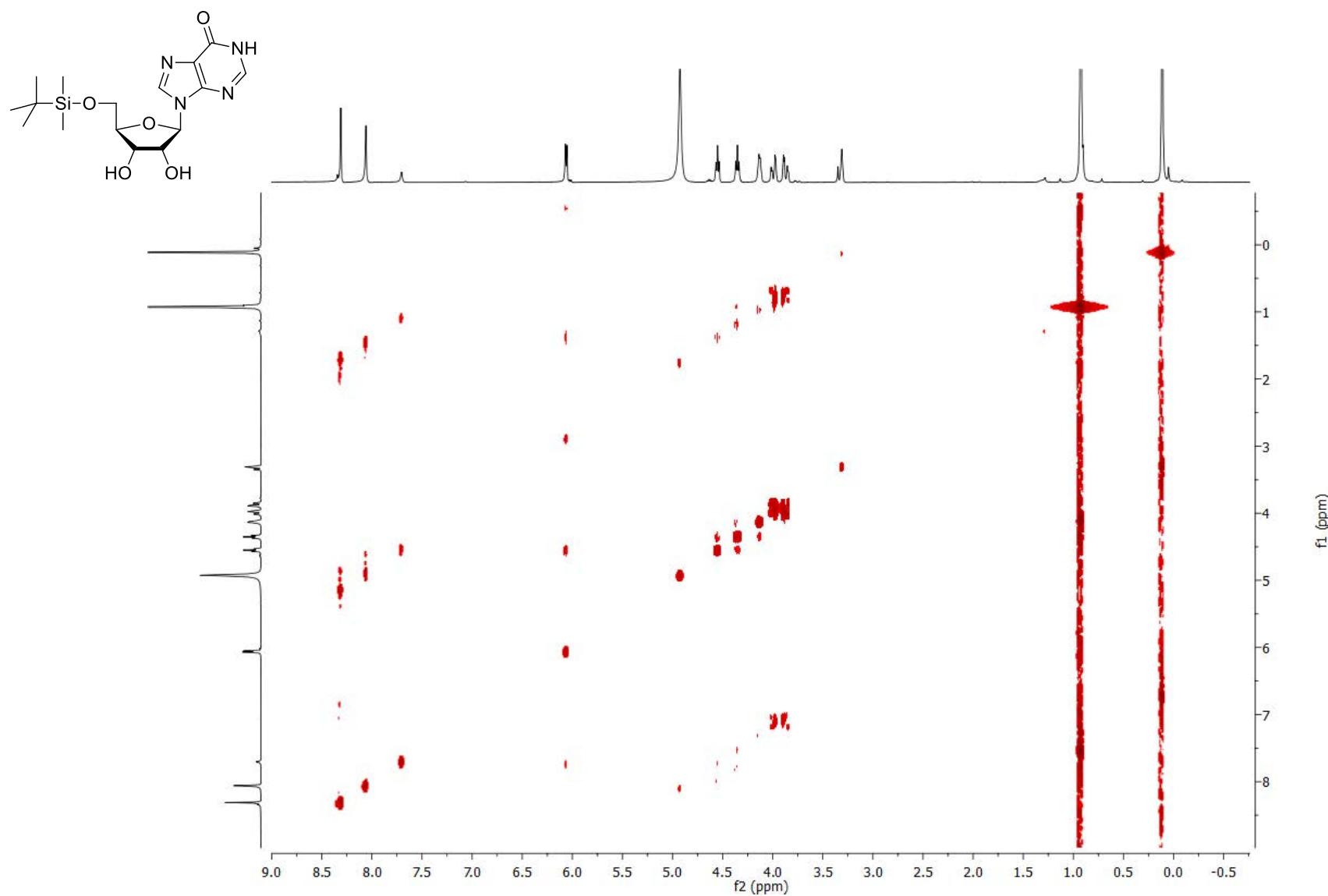
**5'-O-(*tert*-Butyldimethylsilyl)- $\beta$ -D-inosine (5f)**

DEPT NMR (75.5 MHz, MeOH-*d*<sub>4</sub>)



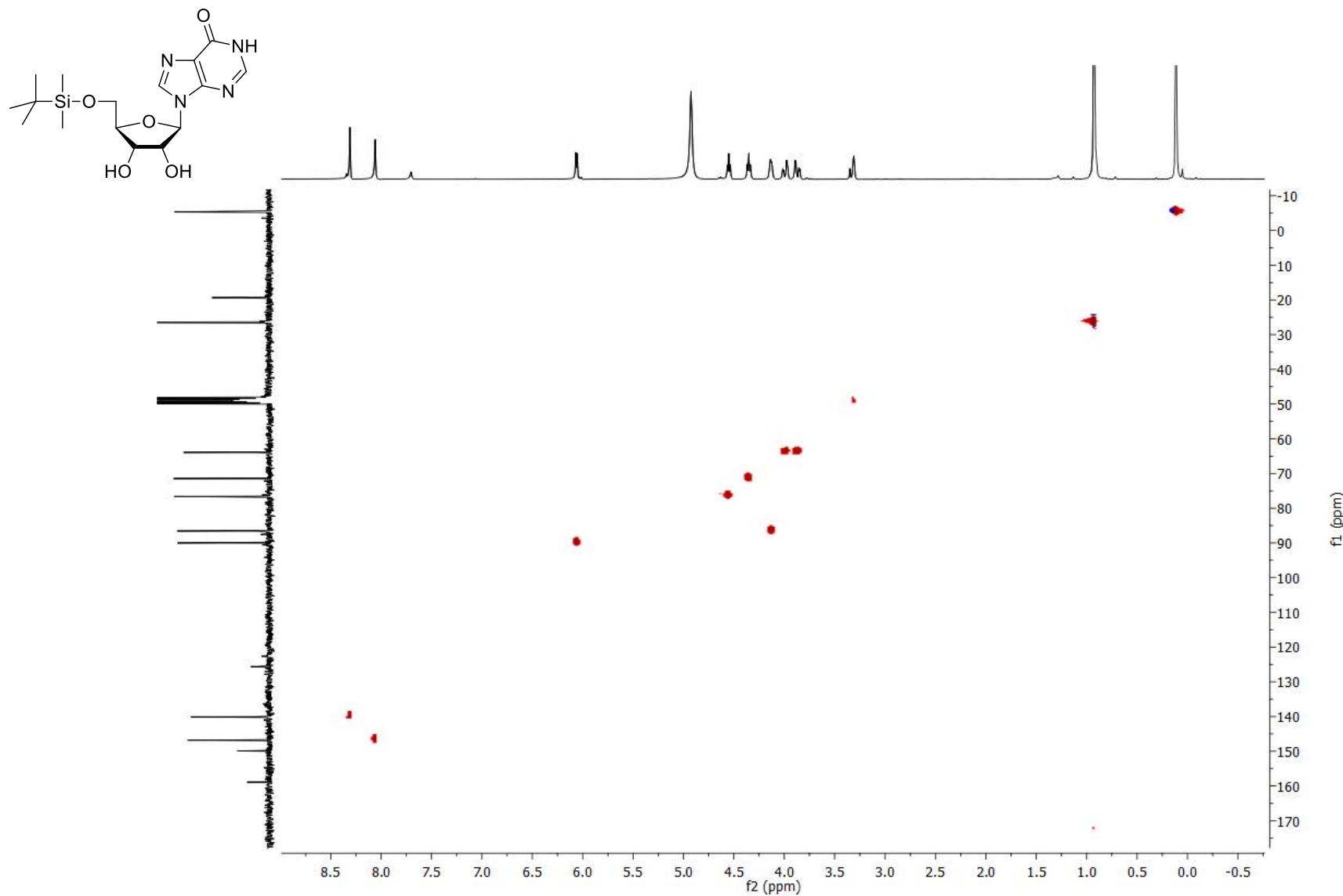
**5'-O-(tert-Butyldimethylsilyl)- $\beta$ -D-inosine (5f)**

COSY NMR (MeOH-*d*<sub>4</sub>)



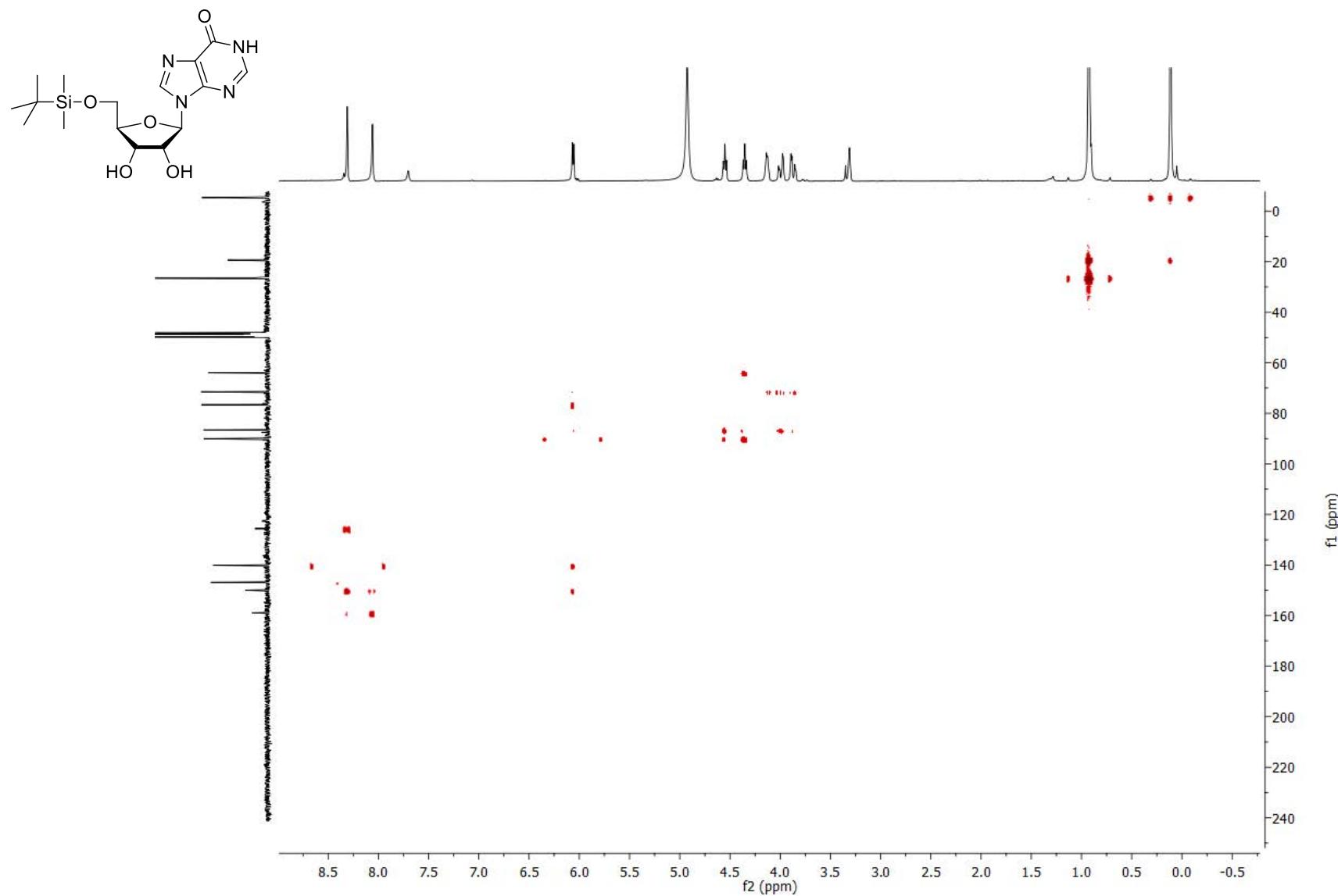
**5'-O-(tert-Butyldimethylsilyl)- $\beta$ -D-inosine (5f)**

HSQC NMR ( $\text{MeOH}-d_4$ )



**5'-O-(tert-Butyldimethylsilyl)- $\beta$ -D-inosine (5f)**

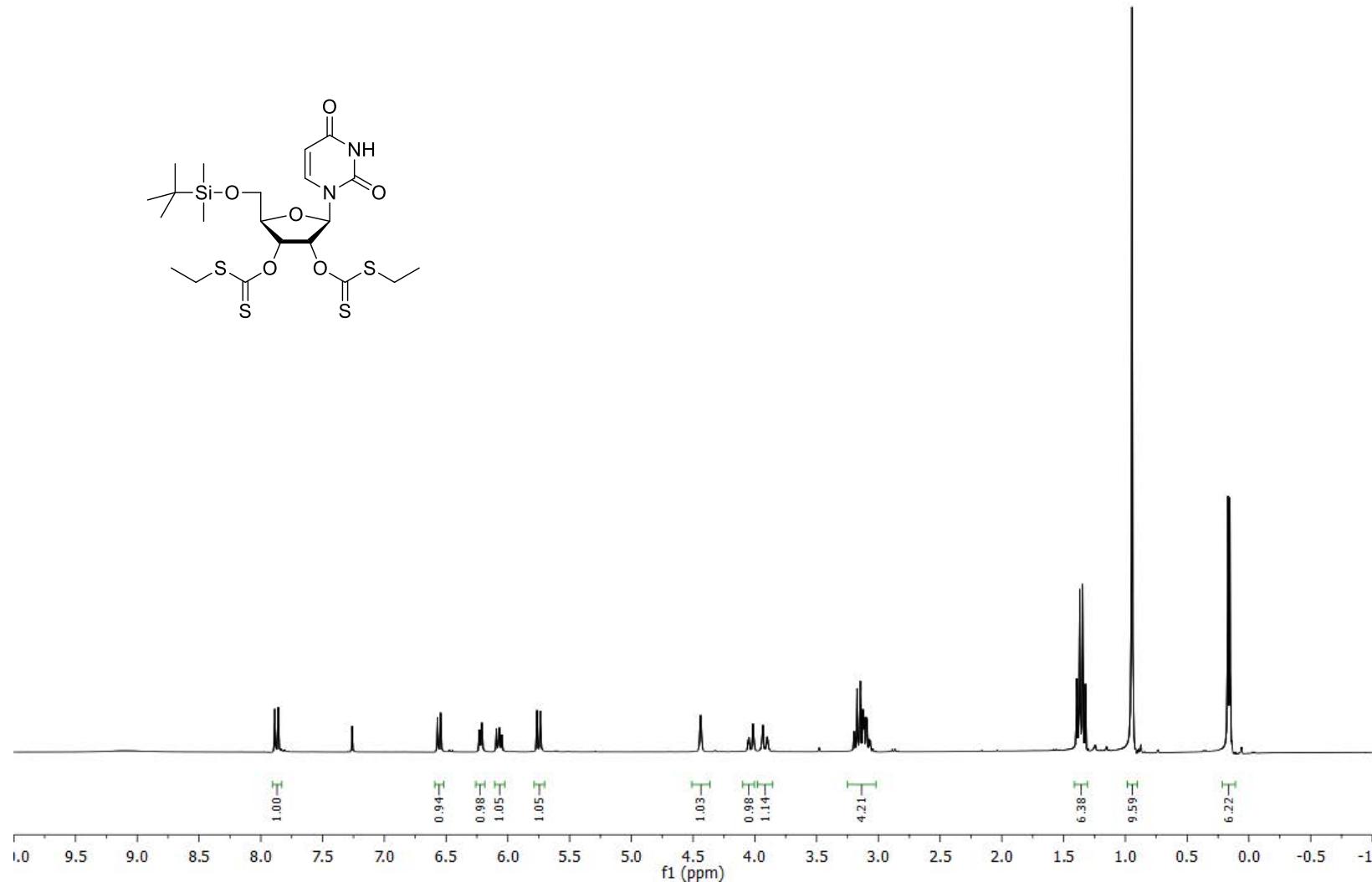
HMBC NMR ( $\text{MeOH}-d_4$ )



**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-uridine (6a)**

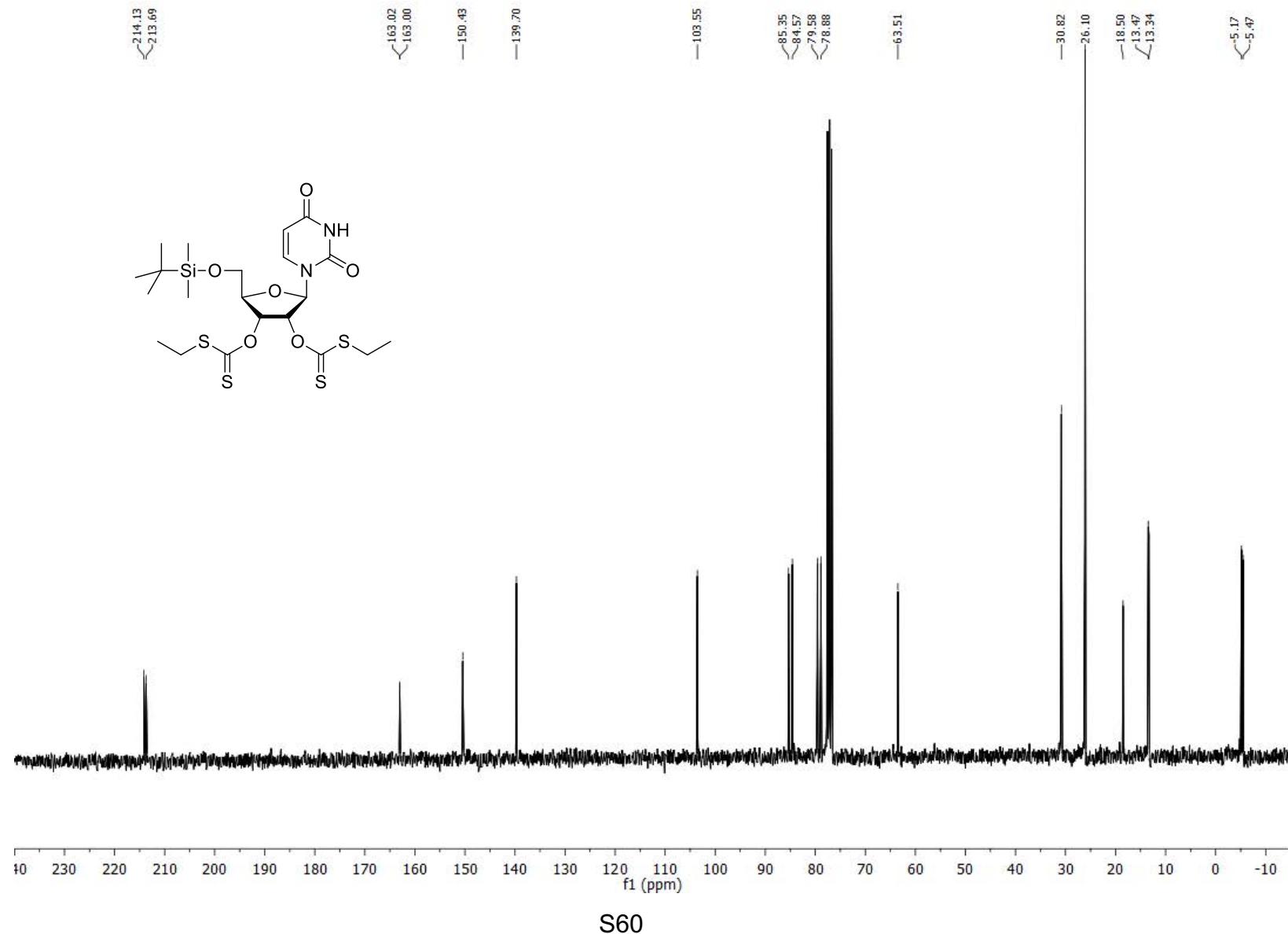
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$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



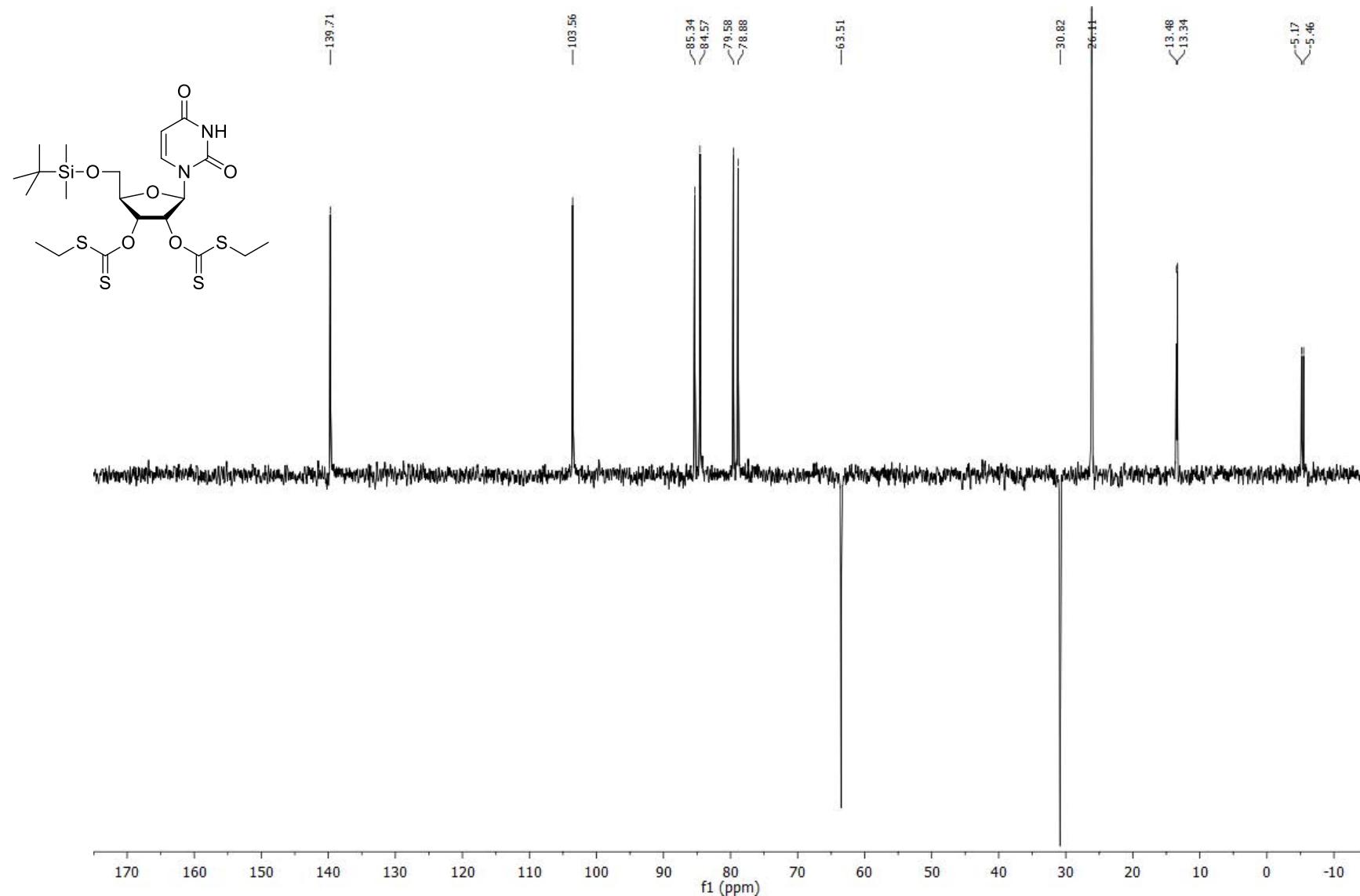
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-uridine (6a)**

$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



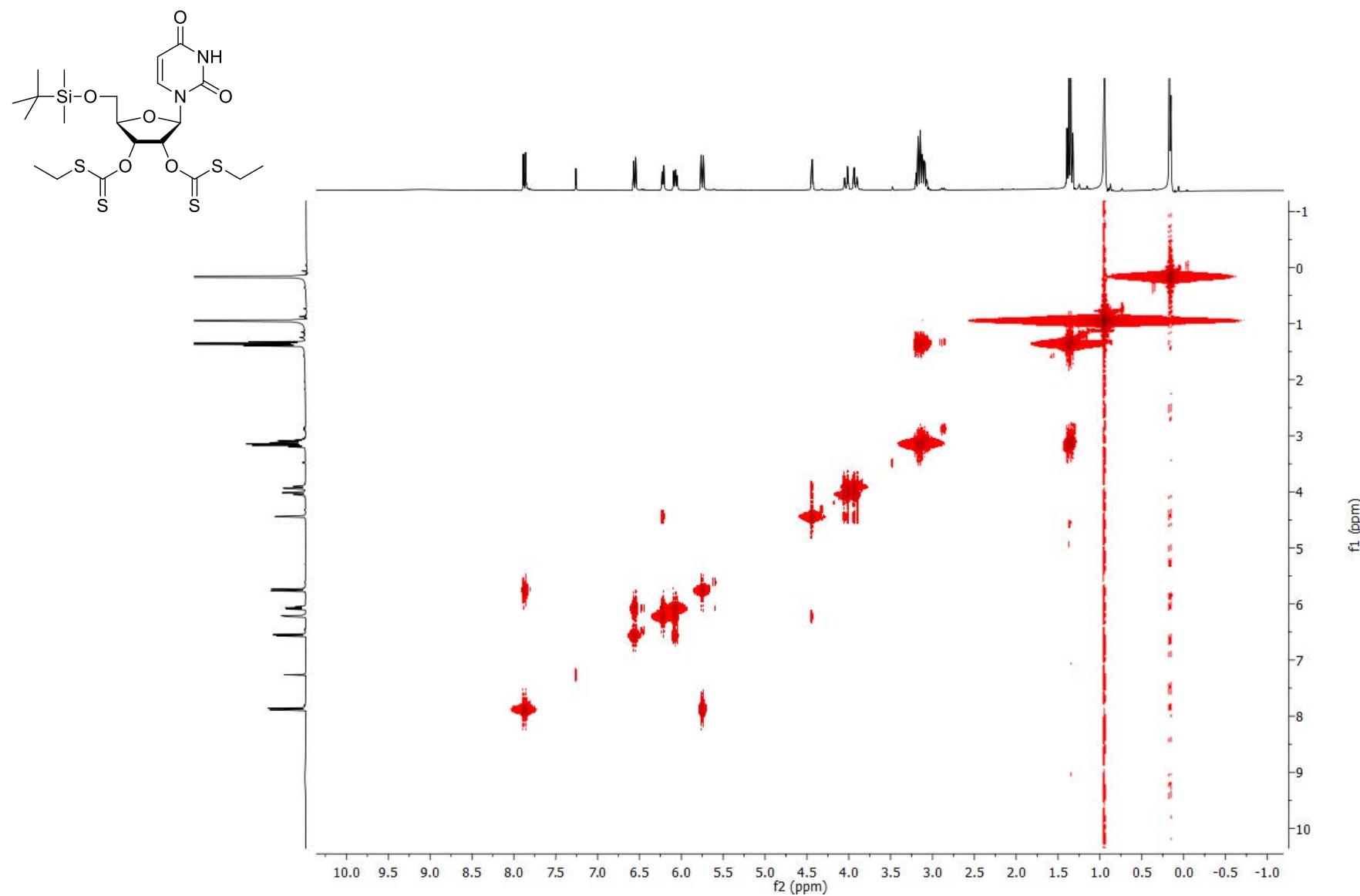
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-uridine (6a)**

$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



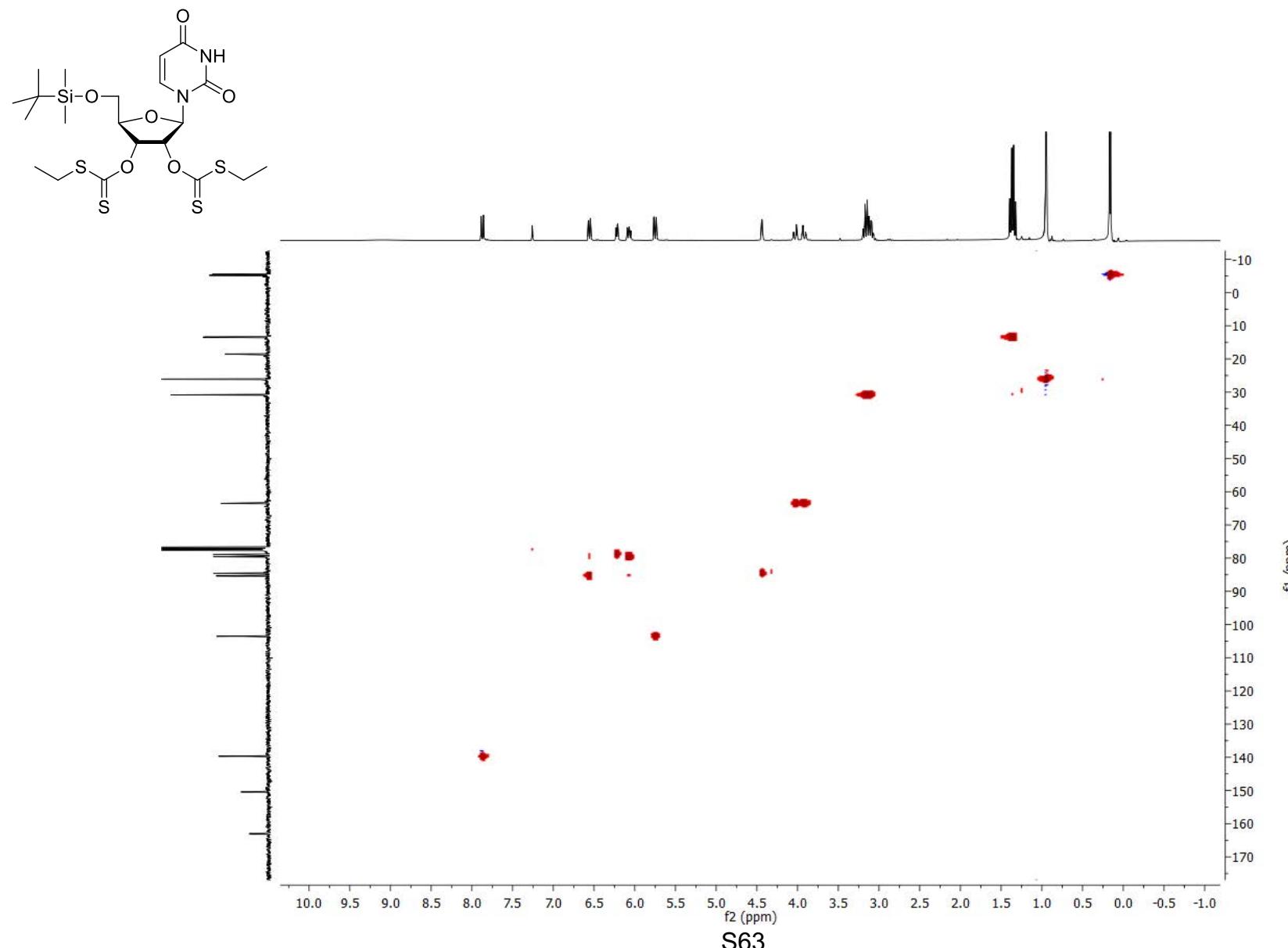
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-uridine (6a)**

COSY NMR ( $\text{CDCl}_3$ )



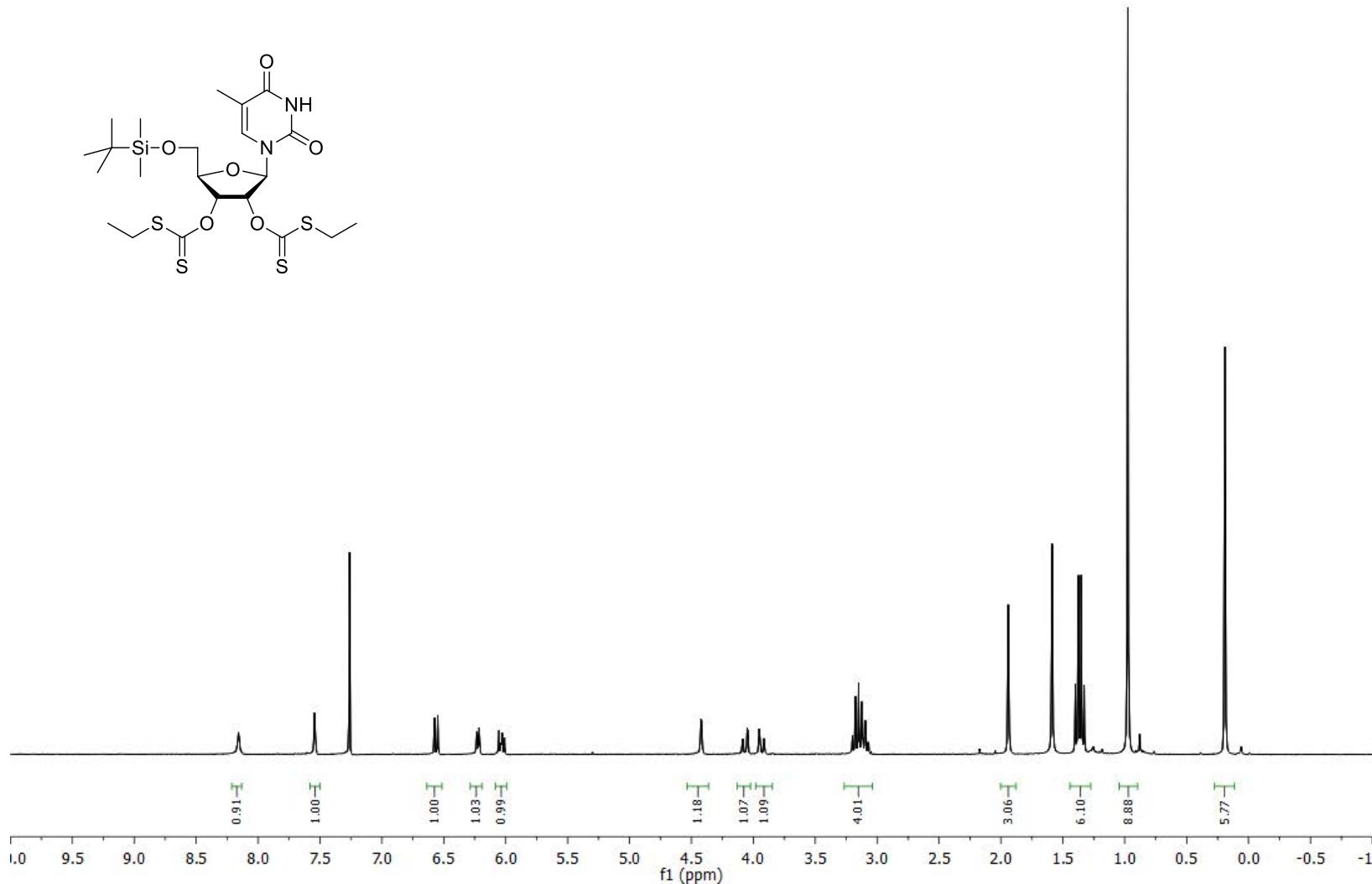
**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-bis-*O*-[(ethylthio)thiocarbonyl]- $\beta$ -D-uridine (6a)**

HSQC NMR ( $\text{CDCl}_3$ )



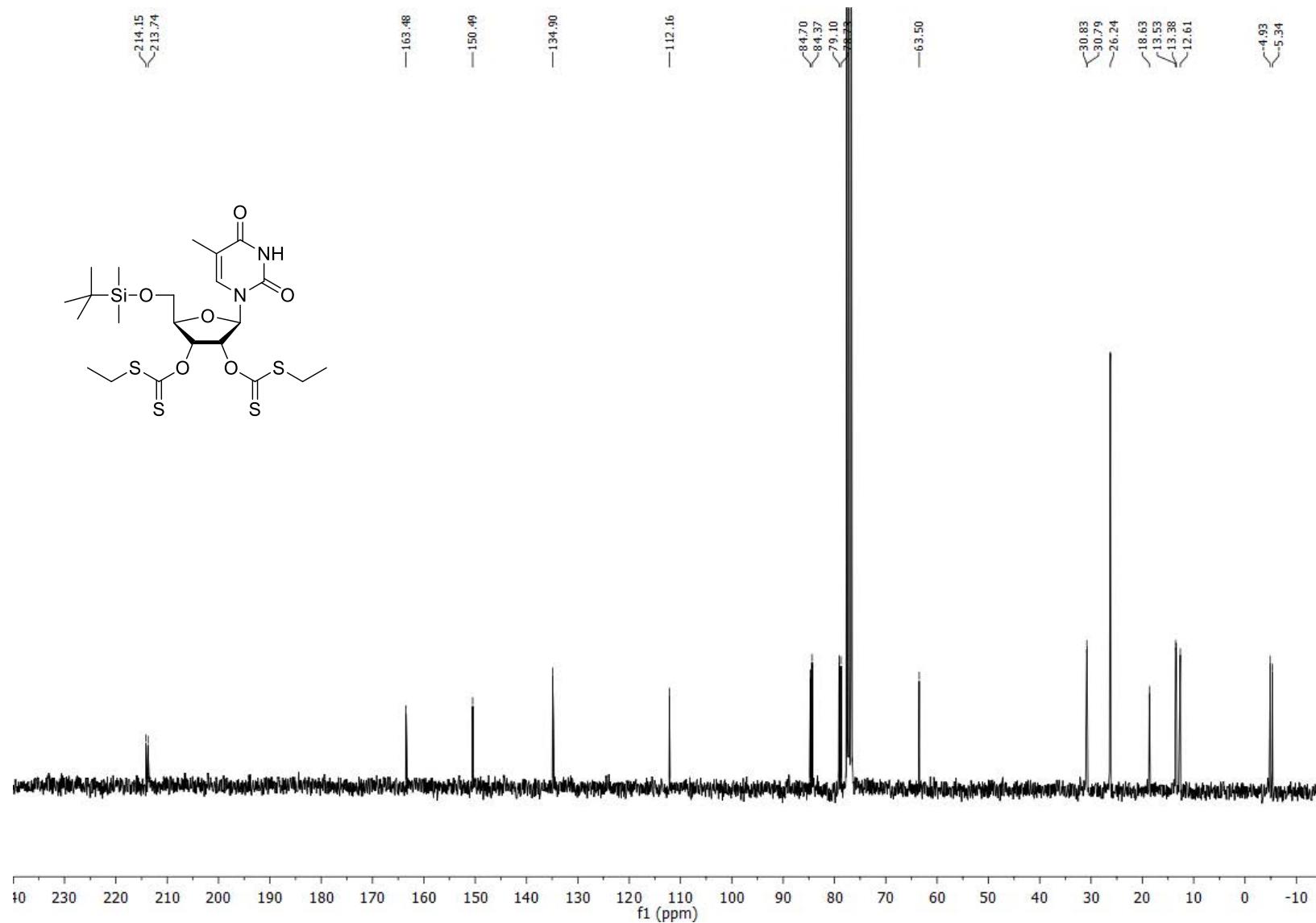
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-5-methyluridine (6b)**

$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



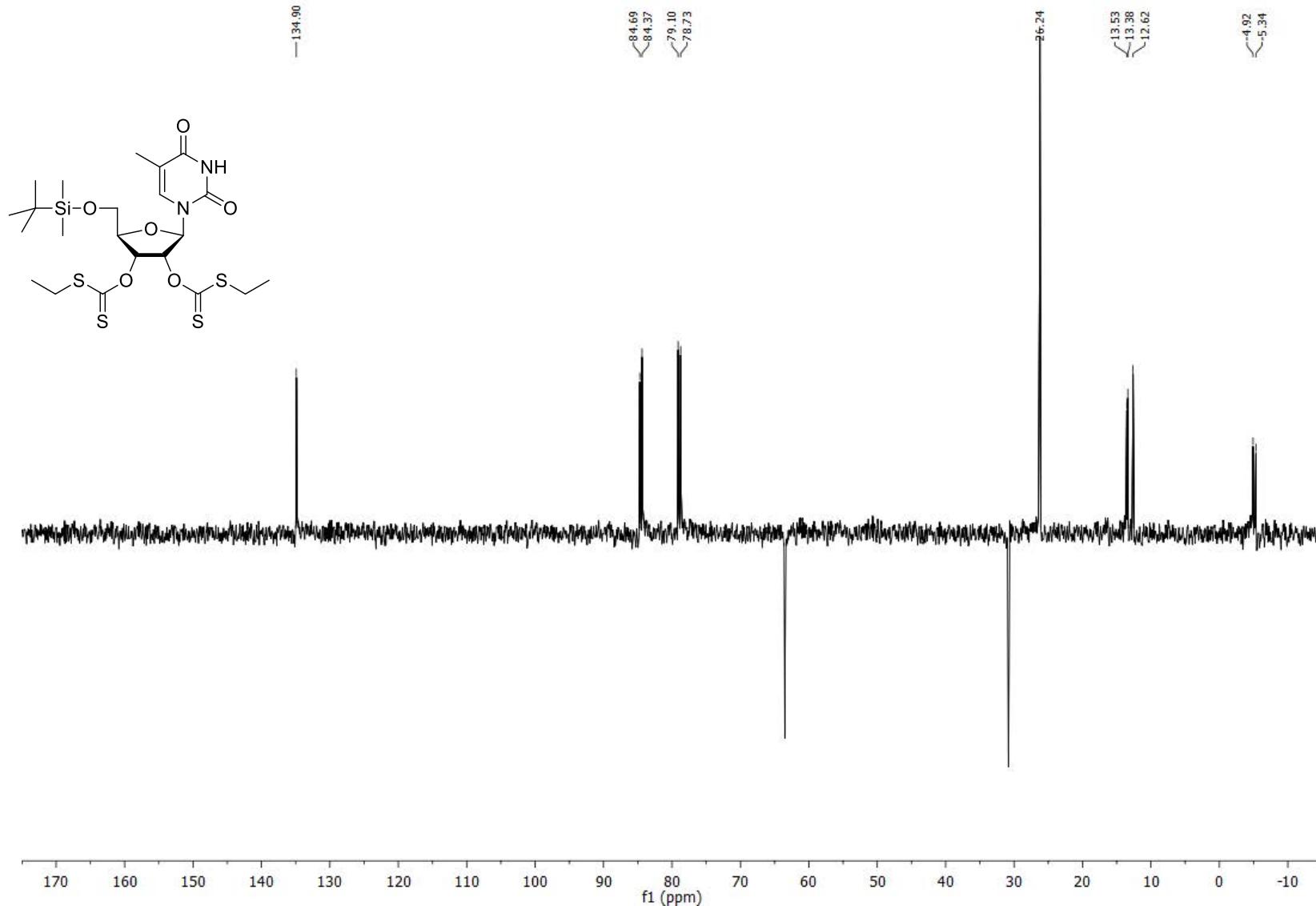
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-5-methyluridine (6b)**

$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



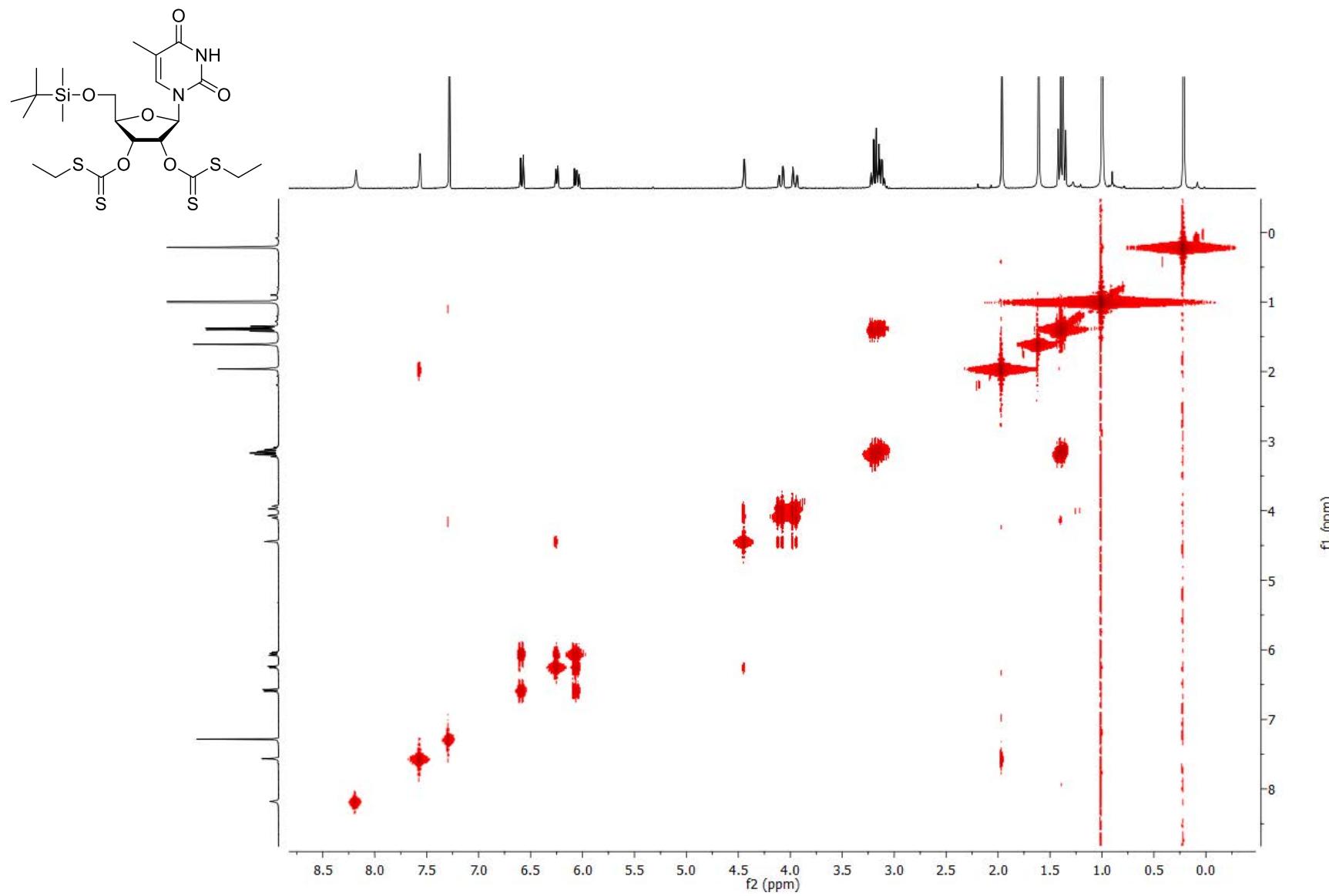
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-5-methyluridine (6b)**

DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



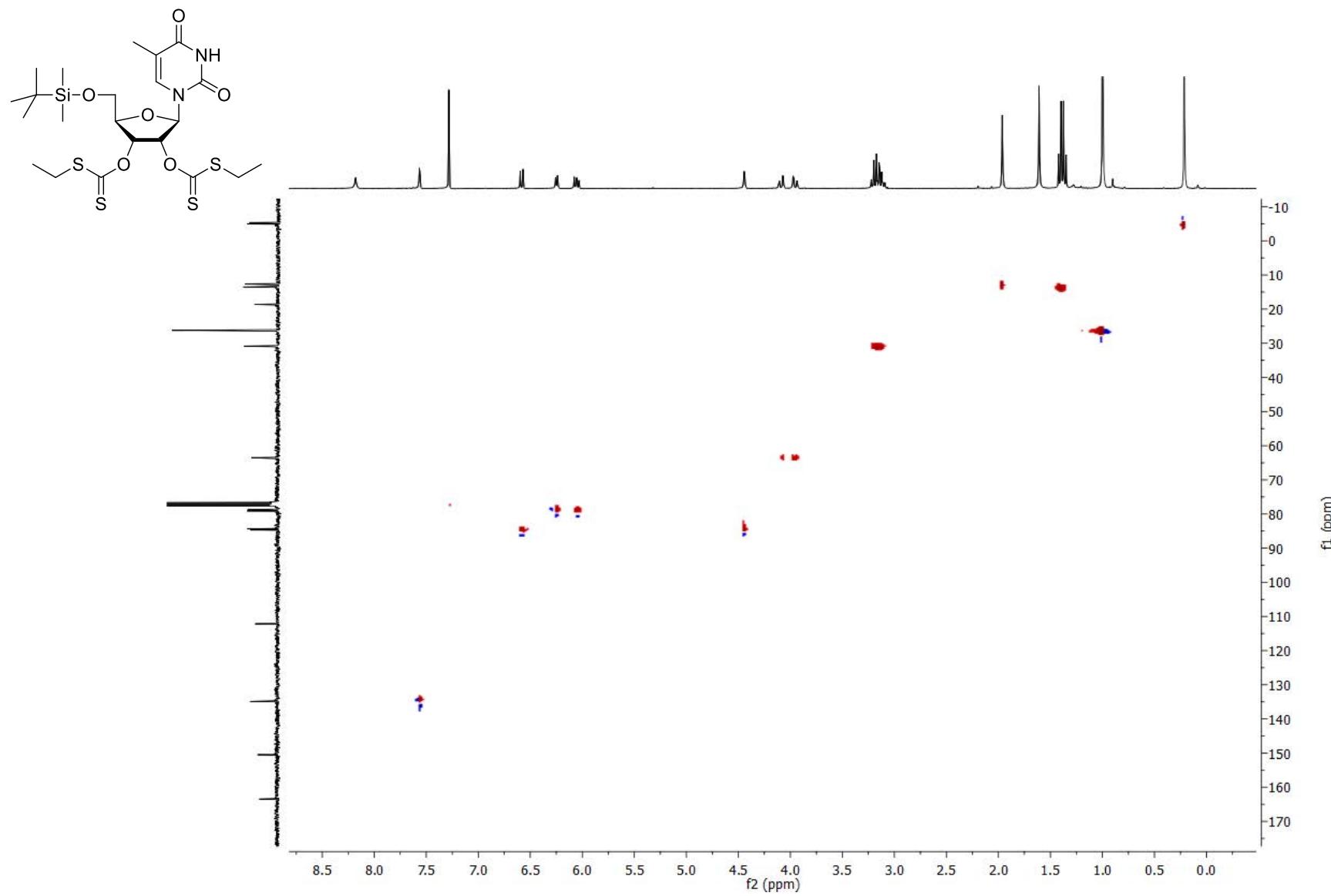
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[ethylthio]thiocarbonyl- $\beta$ -D-5-methyluridine (6b)**

COSY NMR ( $\text{CDCl}_3$ )



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

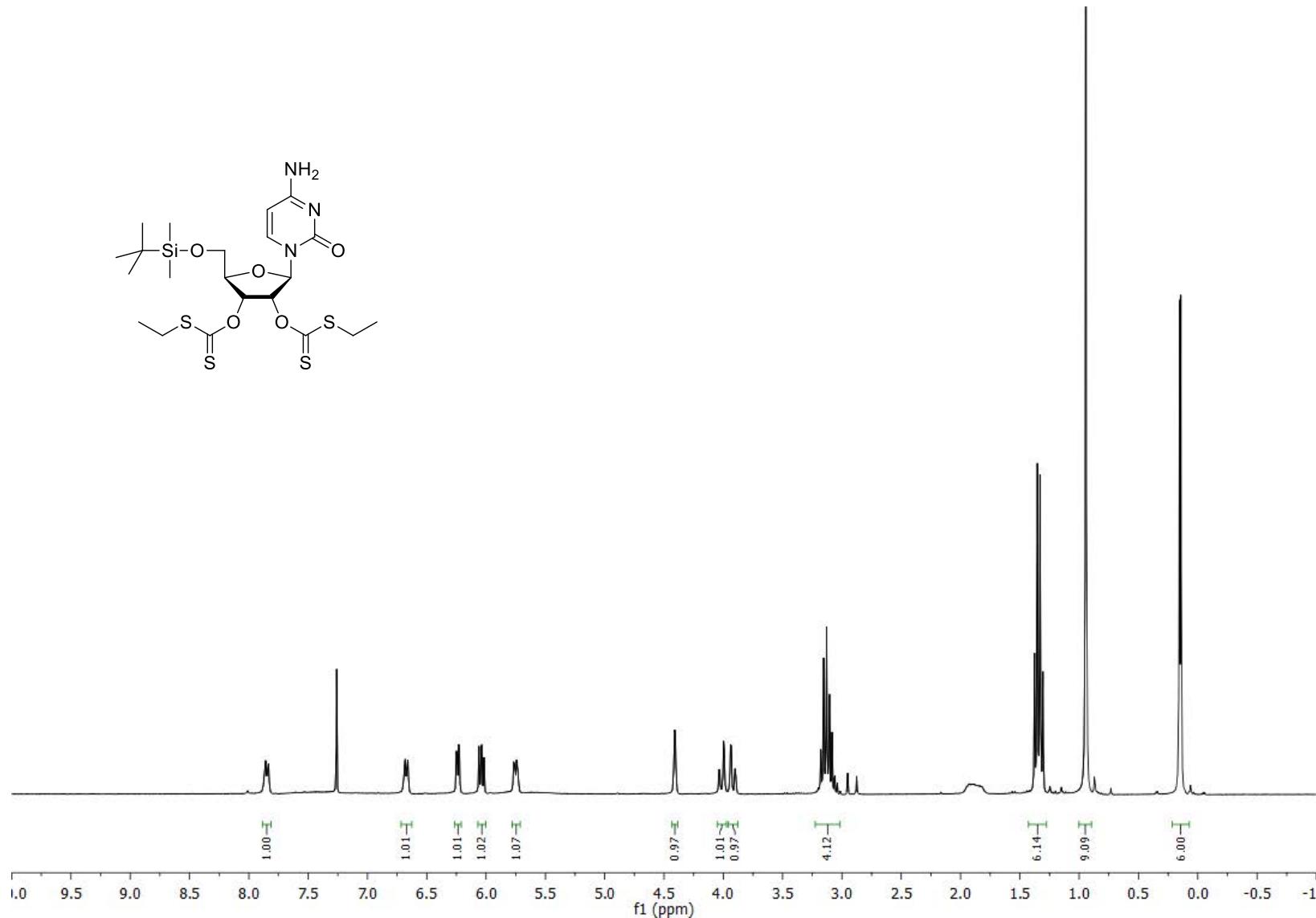
HSQC NMR ( $\text{CDCl}_3$ )



**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -d-cytidine (6c)**

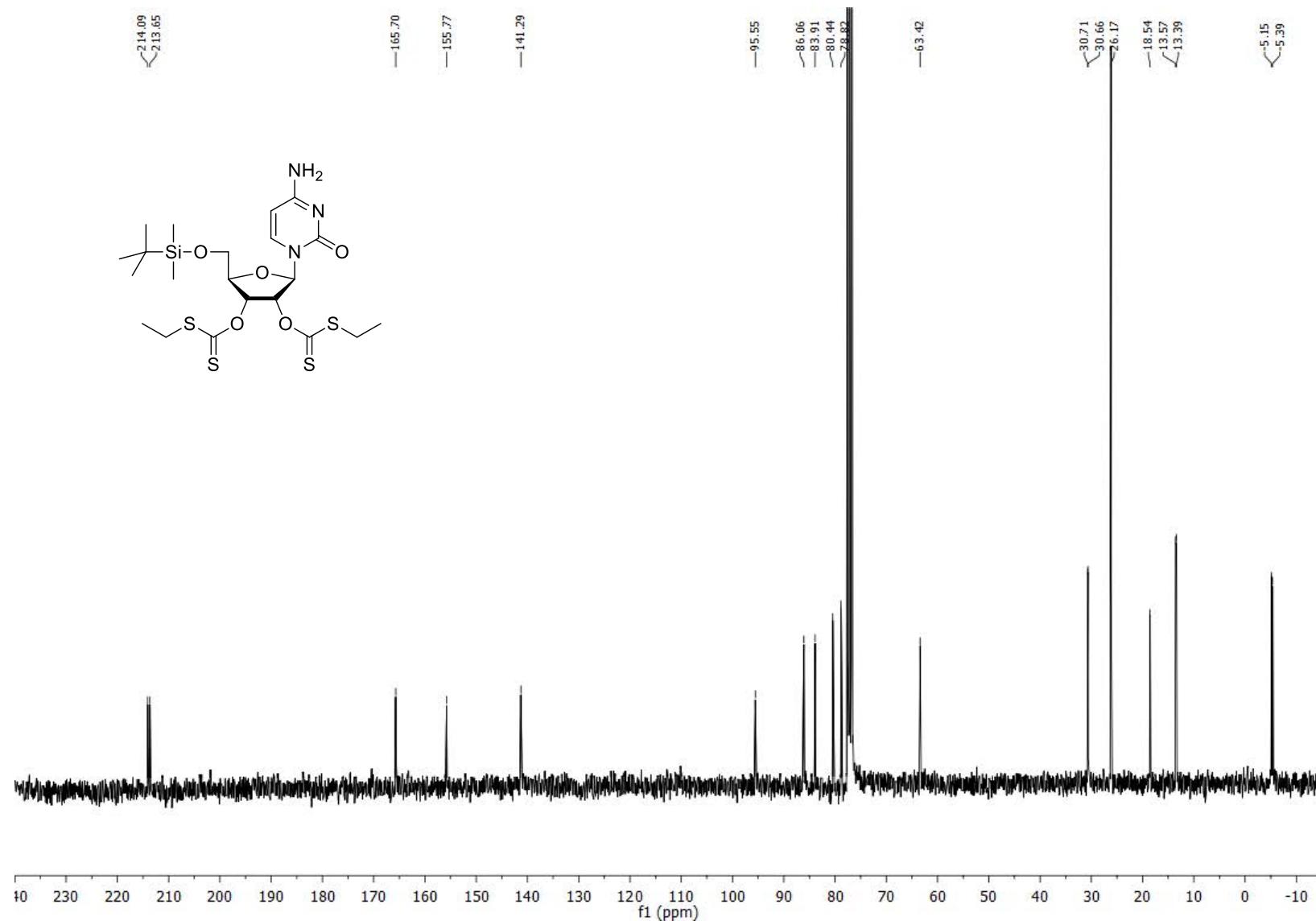
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$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



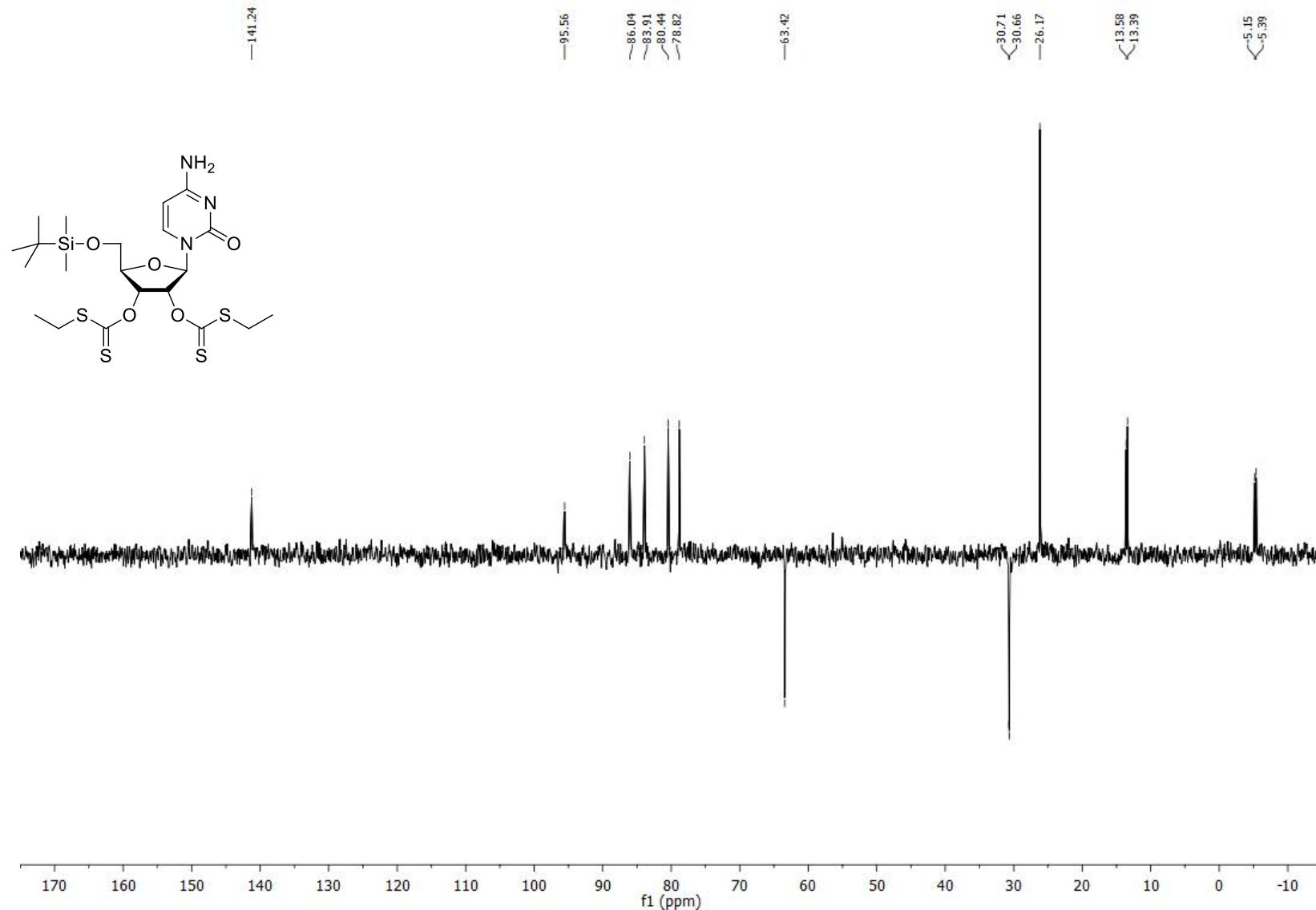
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-cytidine (6c)**

$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



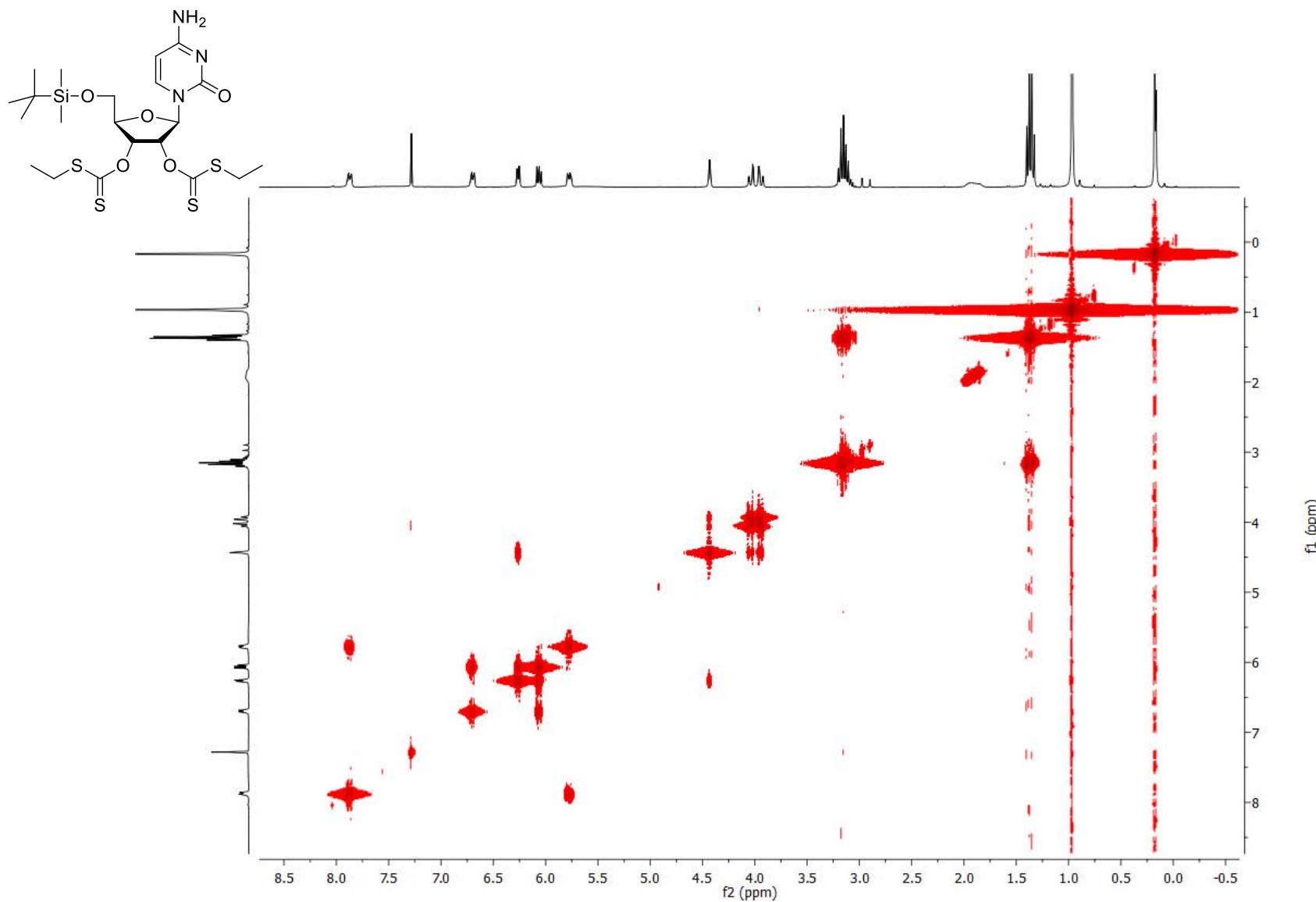
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-cytidine (6c)**

DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



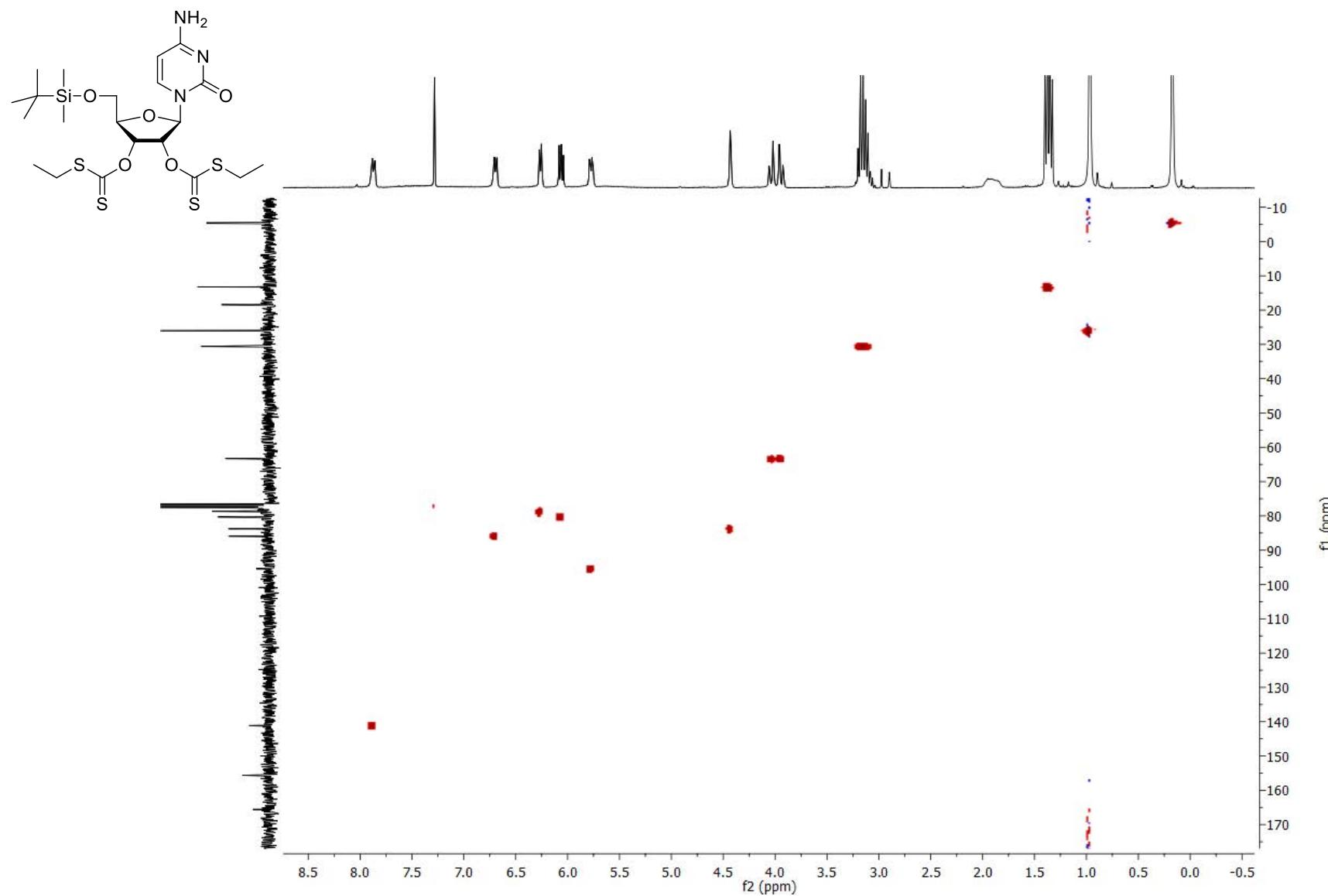
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -d-cytidine (6c)**

COSY NMR ( $\text{CDCl}_3$ )



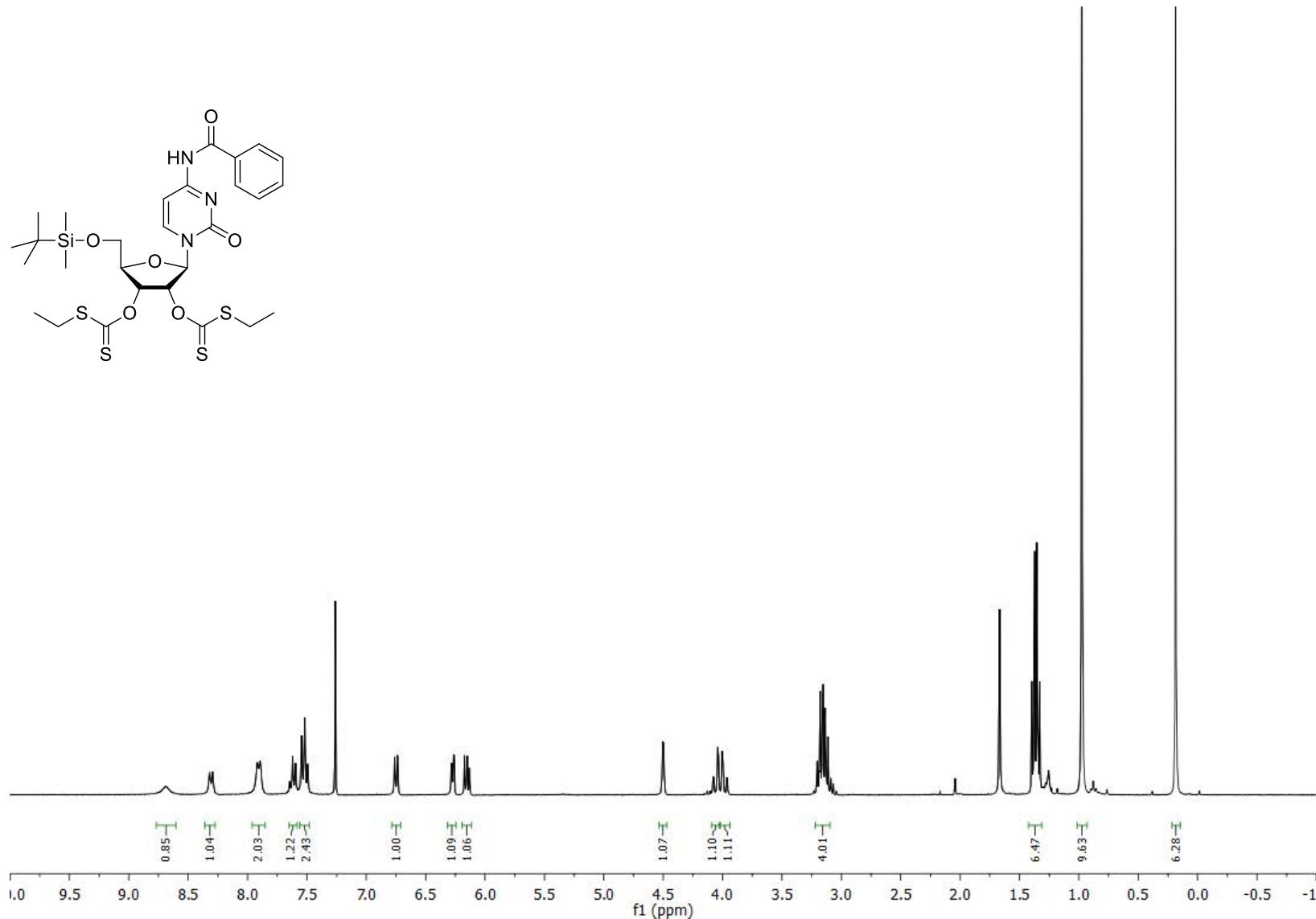
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -d-cytidine (6c)**

HSQC NMR ( $\text{CDCl}_3$ )



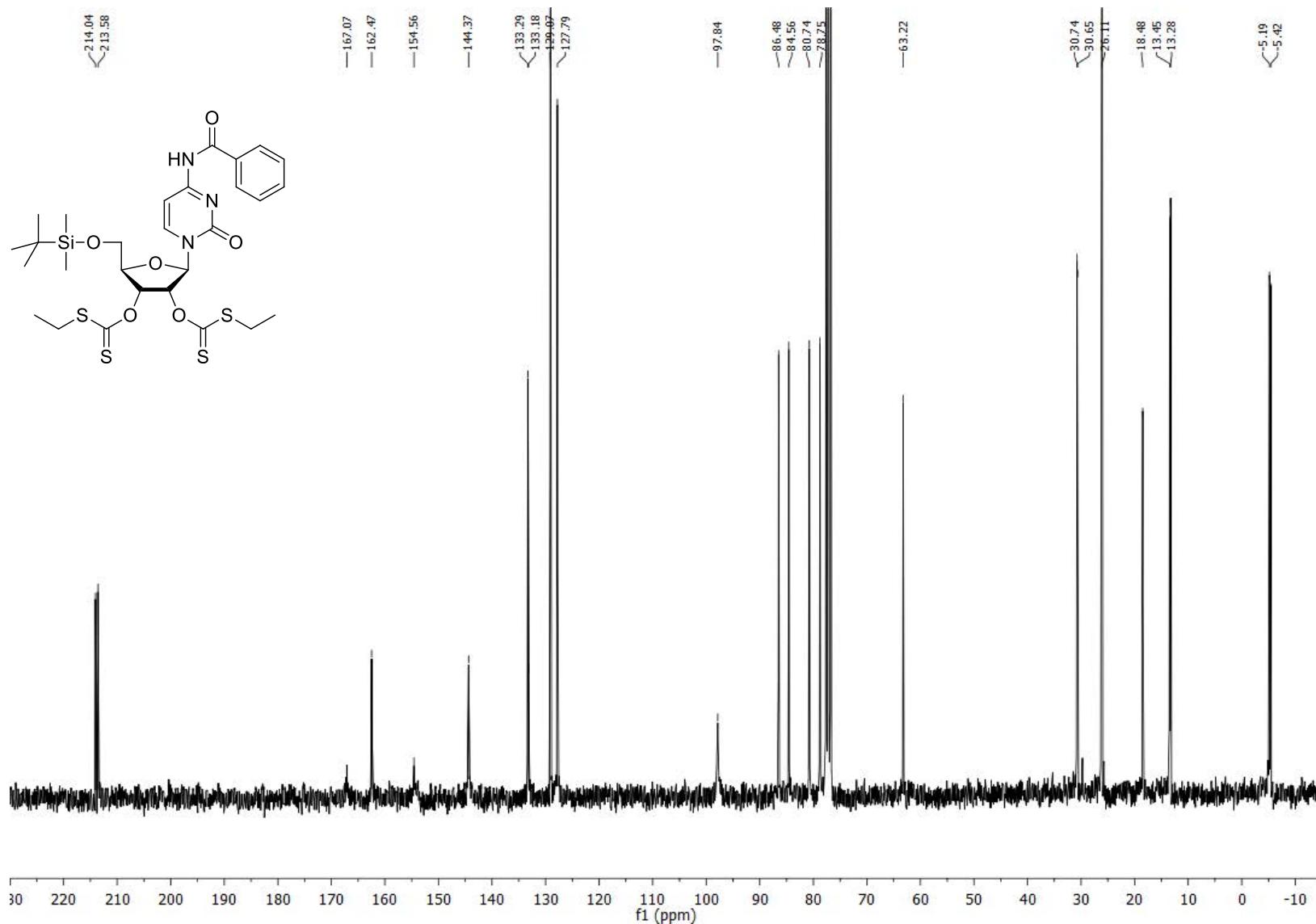
*N<sup>4</sup>-Benzoyl-5'-O-(tert-butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-cytidine (6d)*

<sup>1</sup>H-NMR (300.13 MHz, CDCl<sub>3</sub>)



***N*<sup>4</sup>-Benzoyl-5'-*O*-(tert-butyldimethylsilyl)-2',3'-bis-*O*-[(ethylthio)thiocarbonyl]- $\beta$ -D-cytidine (6d)**

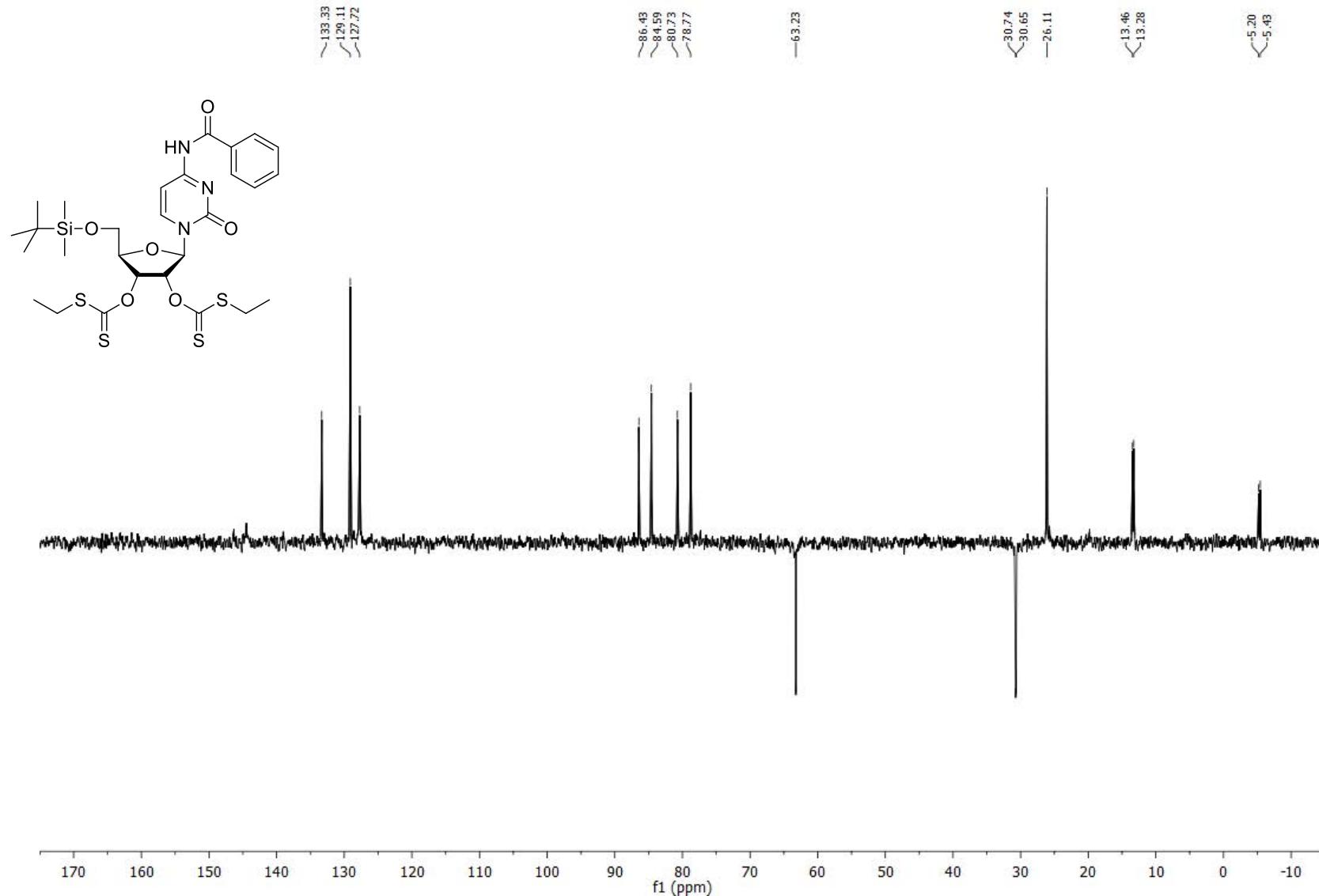
<sup>13</sup>C-NMR (75.5 MHz, CDCl<sub>3</sub>)



***N*<sup>4</sup>-Benzoyl-5'-*O*-(tert-butyldimethylsilyl)-2',3'-bis-*O*-[(ethylthio)thiocarbonyl]- $\beta$ -D-cytidine (6d)**

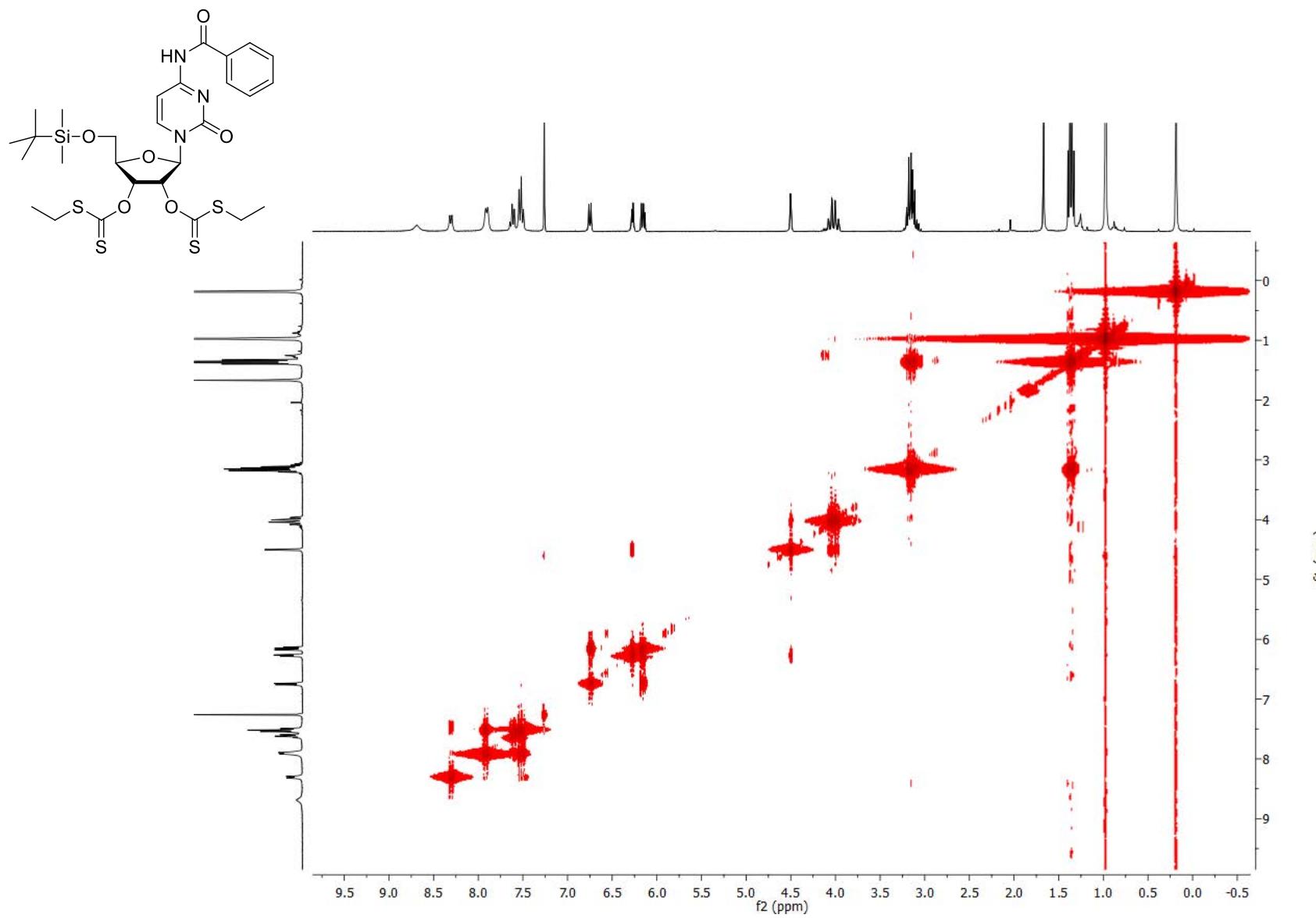
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DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



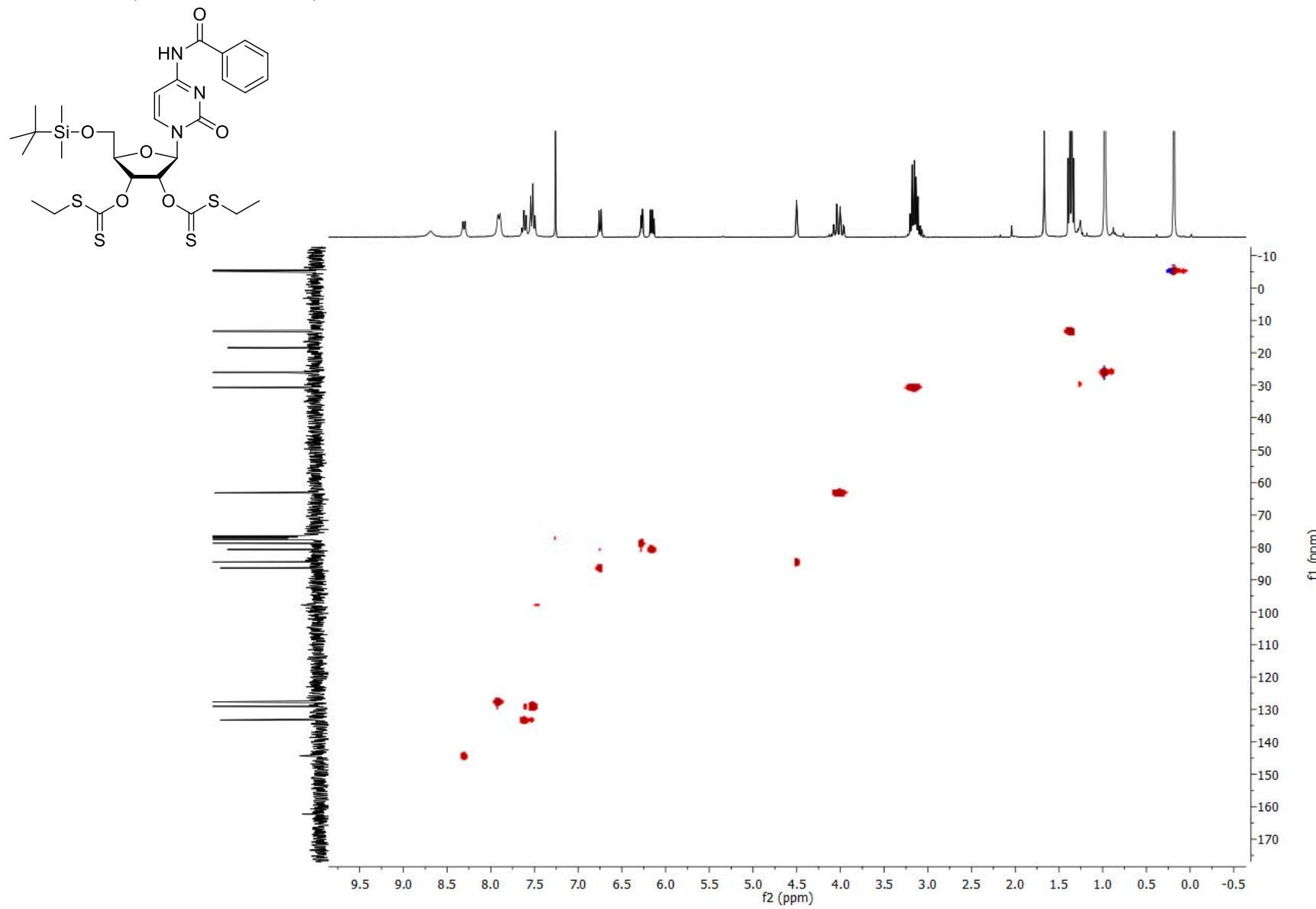
***N*<sup>4</sup>-Benzoyl-5'-*O*-(tert-butyldimethylsilyl)-2',3'-bis-*O*-[(ethylthio)thiocarbonyl]- $\beta$ -D-cytidine (6d)**

COSY NMR (75.5 MHz, CDCl<sub>3</sub>)



*N*<sup>4</sup>-Benzoyl-5'-*O*-(*tert*-butyldimethylsilyl)-2',3'-bis-*O*-[(ethylthio)thiocarbonyl]- $\beta$ -D-cytidine (6d)

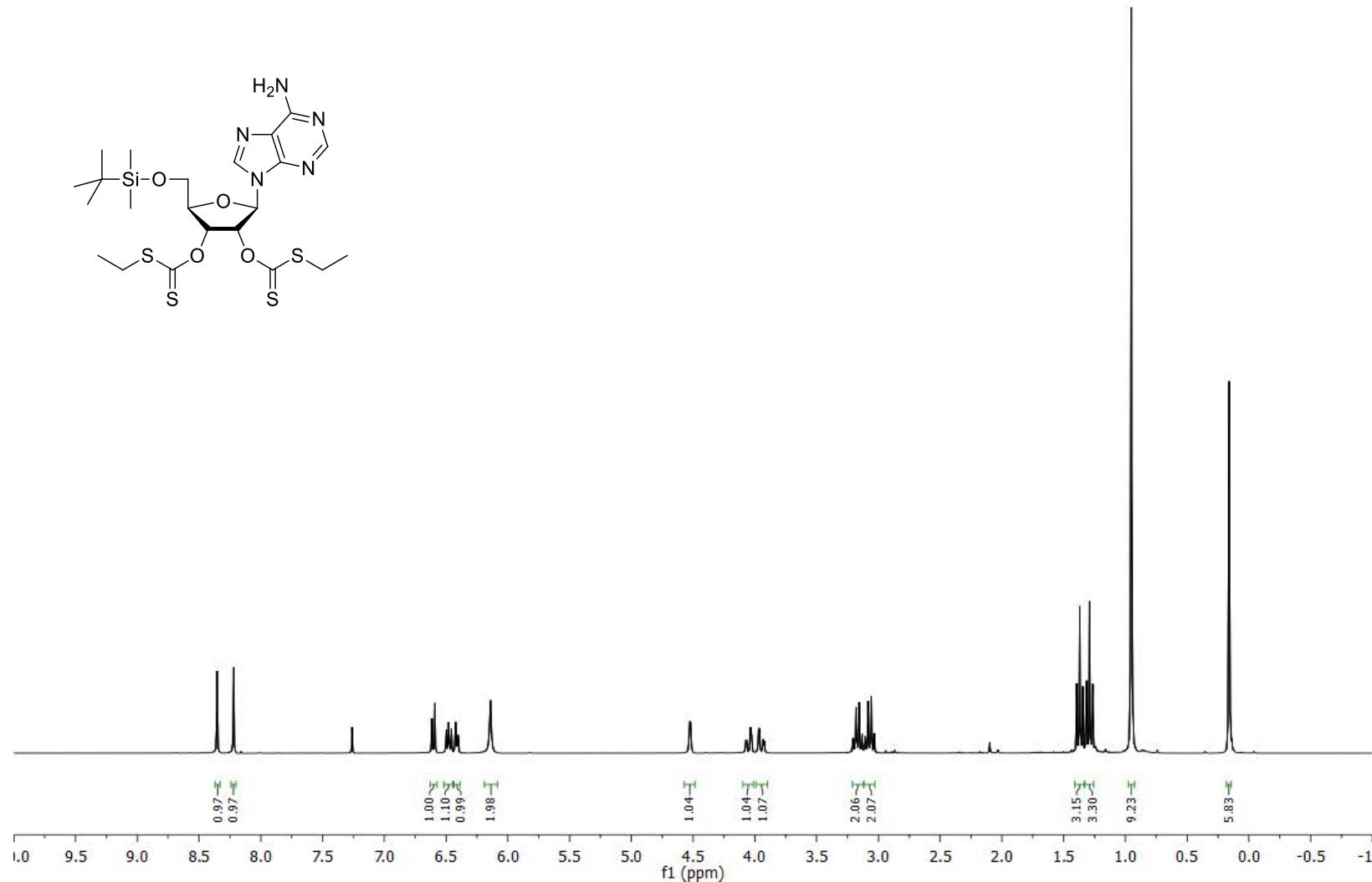
HSQC NMR (75.5 MHz, CDCl<sub>3</sub>)



**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[ethylthio]thiocarbonyl]- $\beta$ -D-adenosine (6e)**

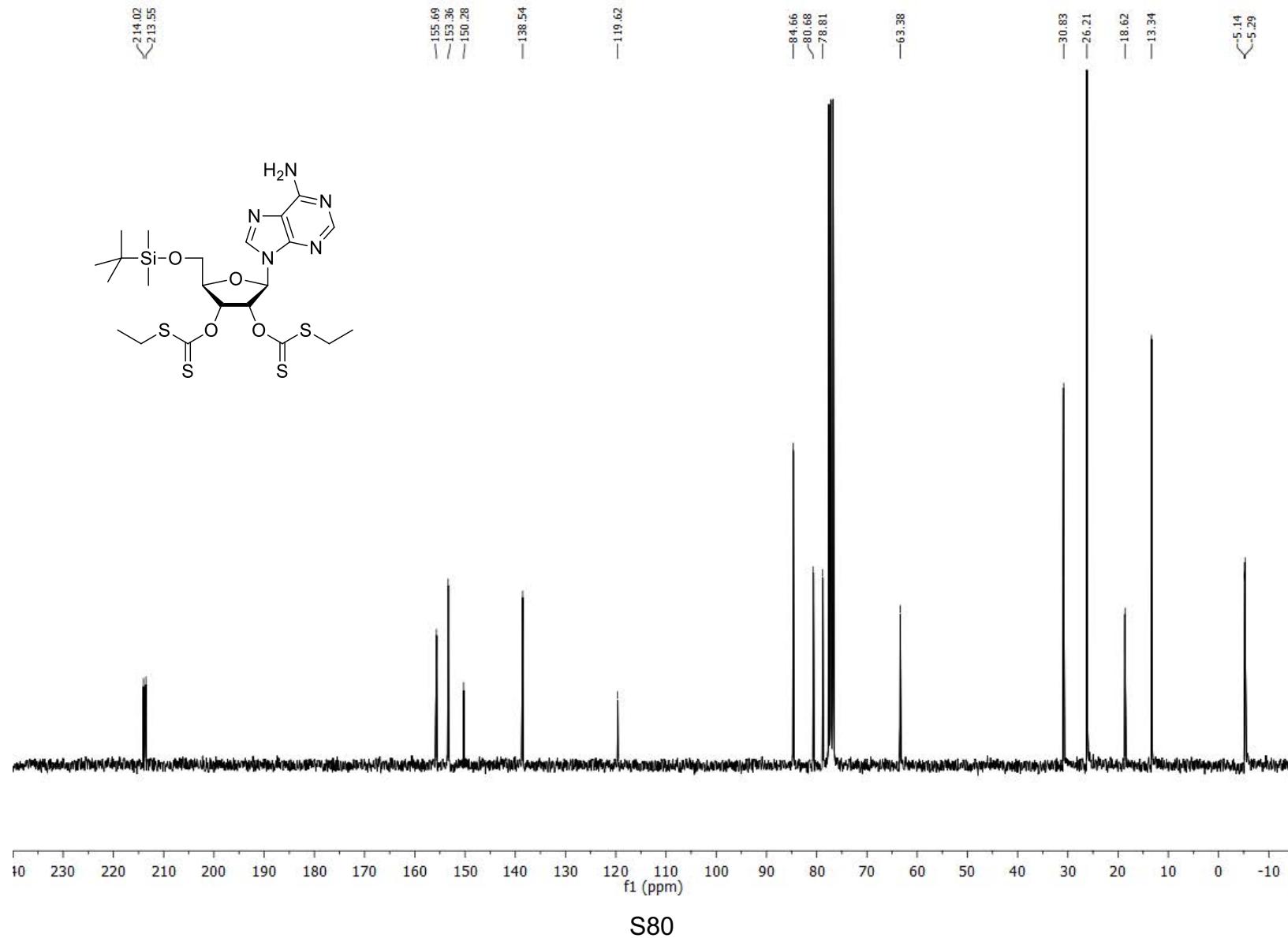
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$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[ethylthio)thiocarbonyl]- $\beta$ -D-adenosine (6e)**

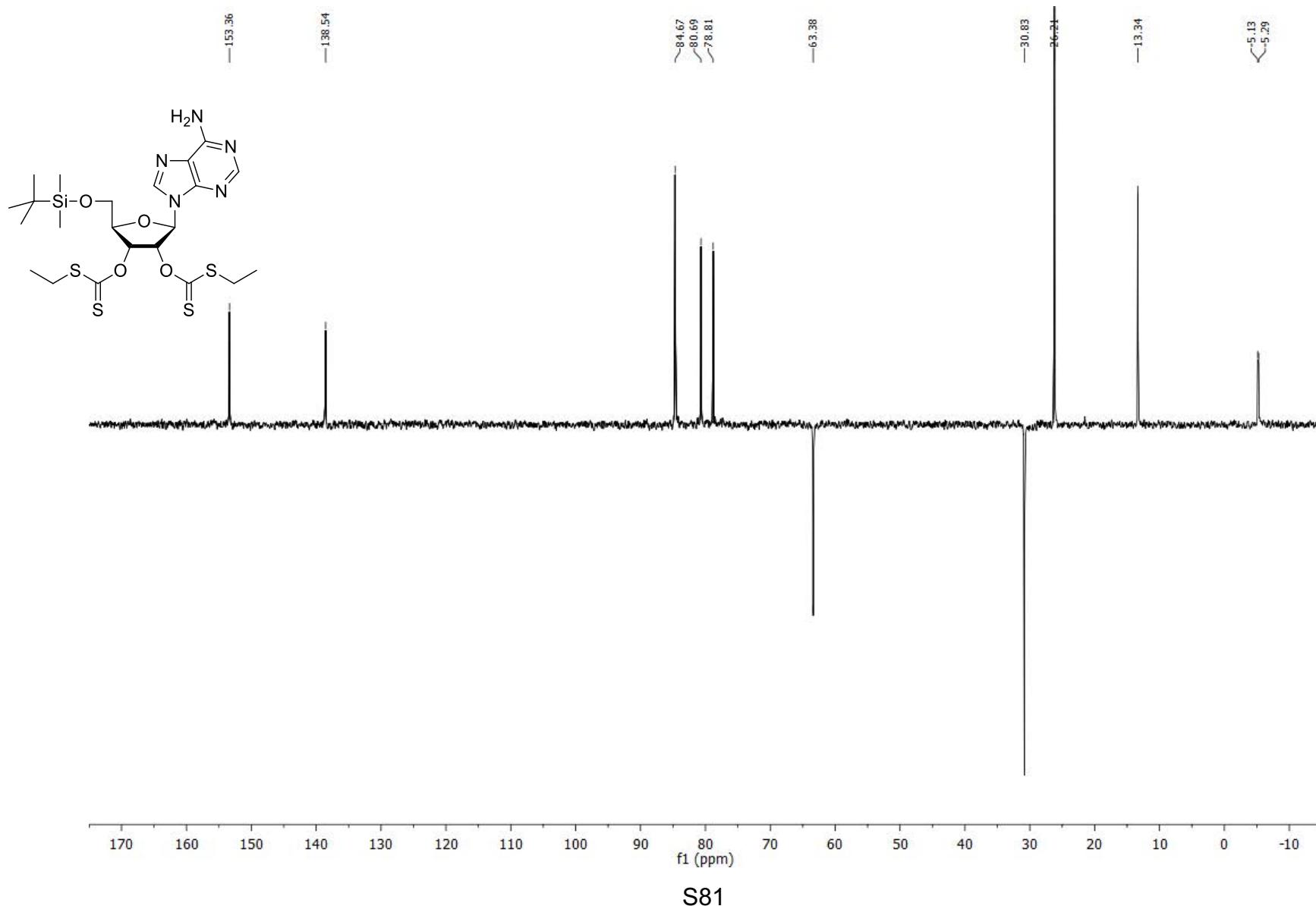
$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[ethylthio)thiocarbonyl]- $\beta$ -D-adenosine (6e)**

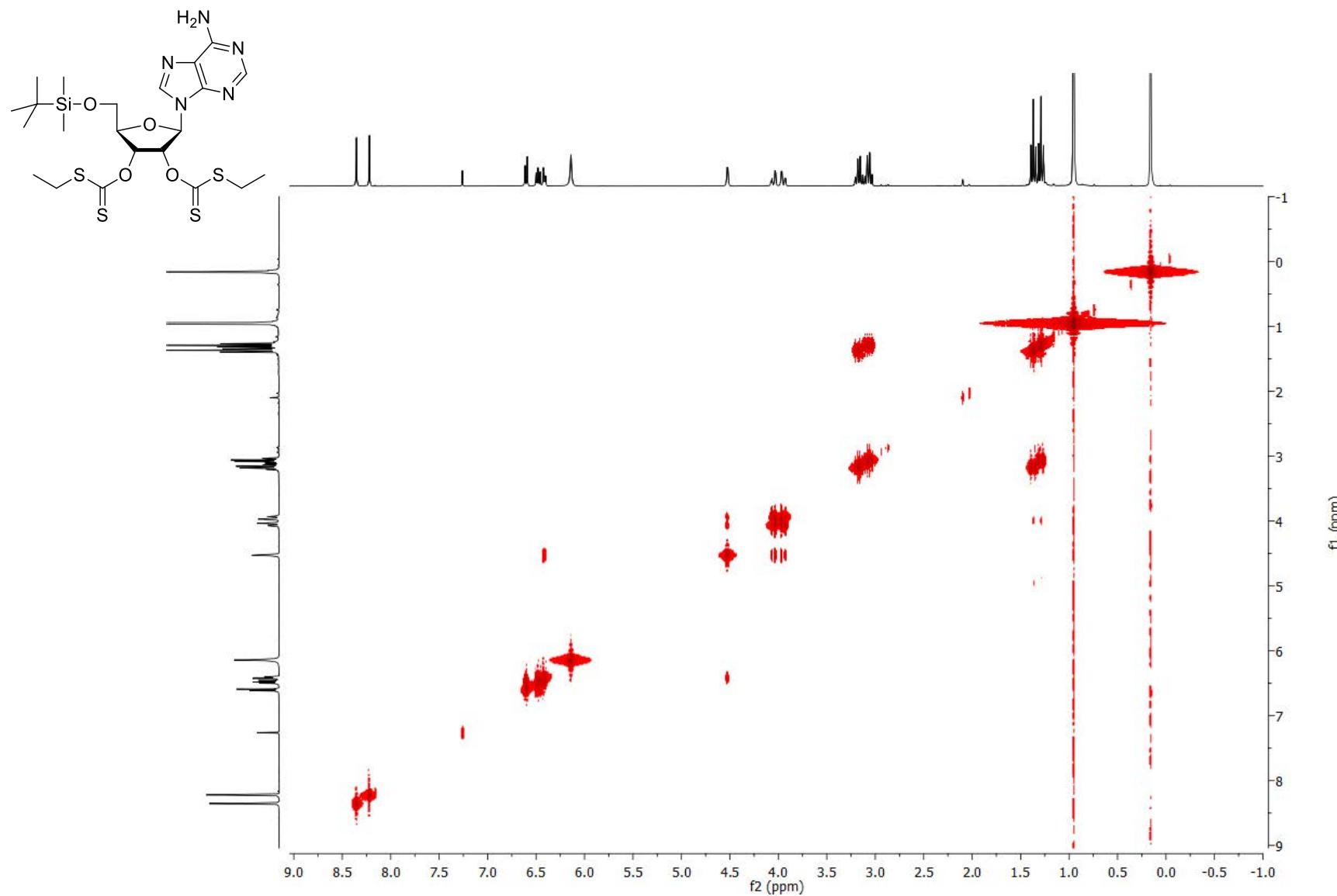
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DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



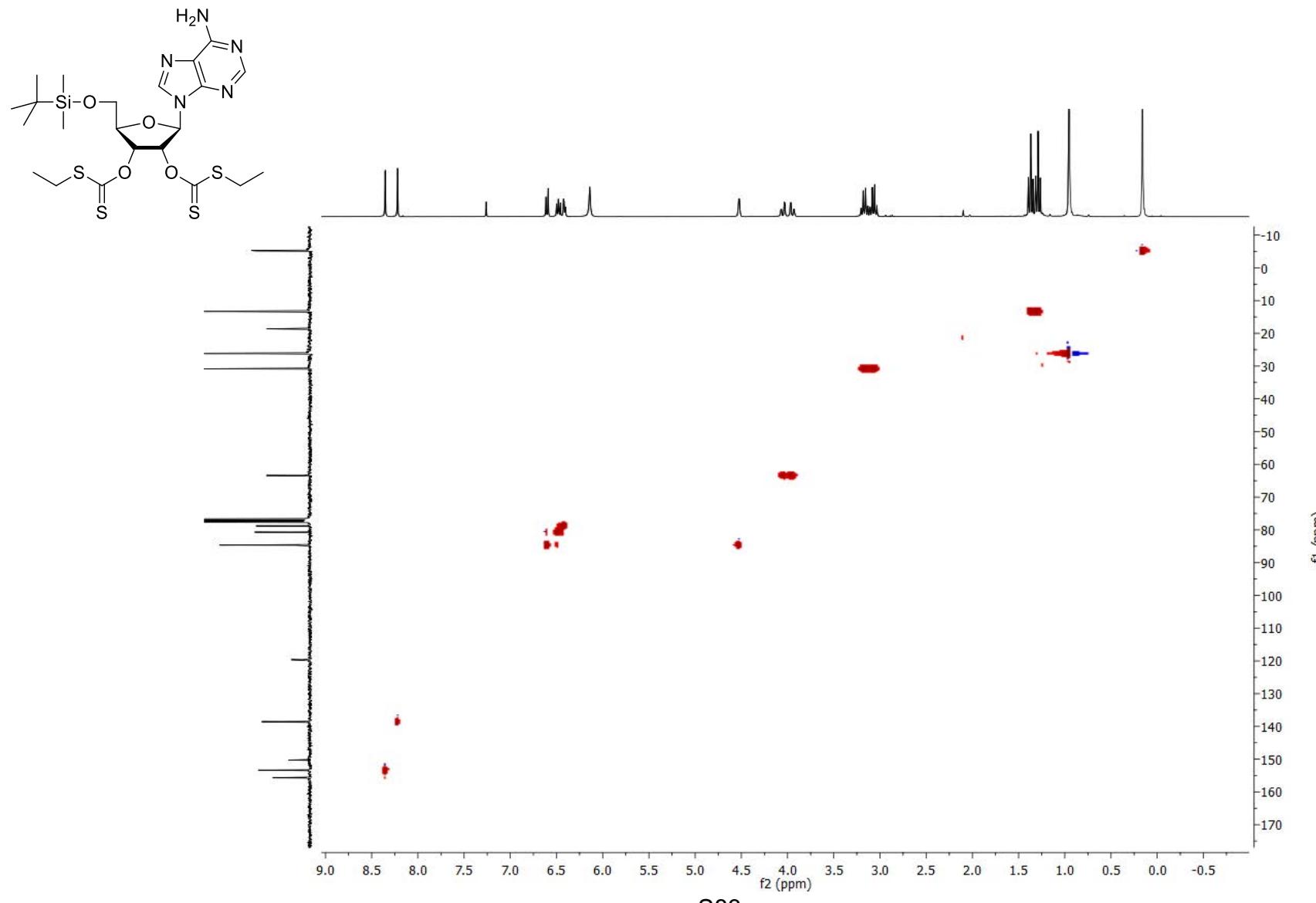
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[ethylthio)thiocarbonyl]- $\beta$ -D-adenosine (6e)**

COSY NMR ( $\text{CDCl}_3$ )



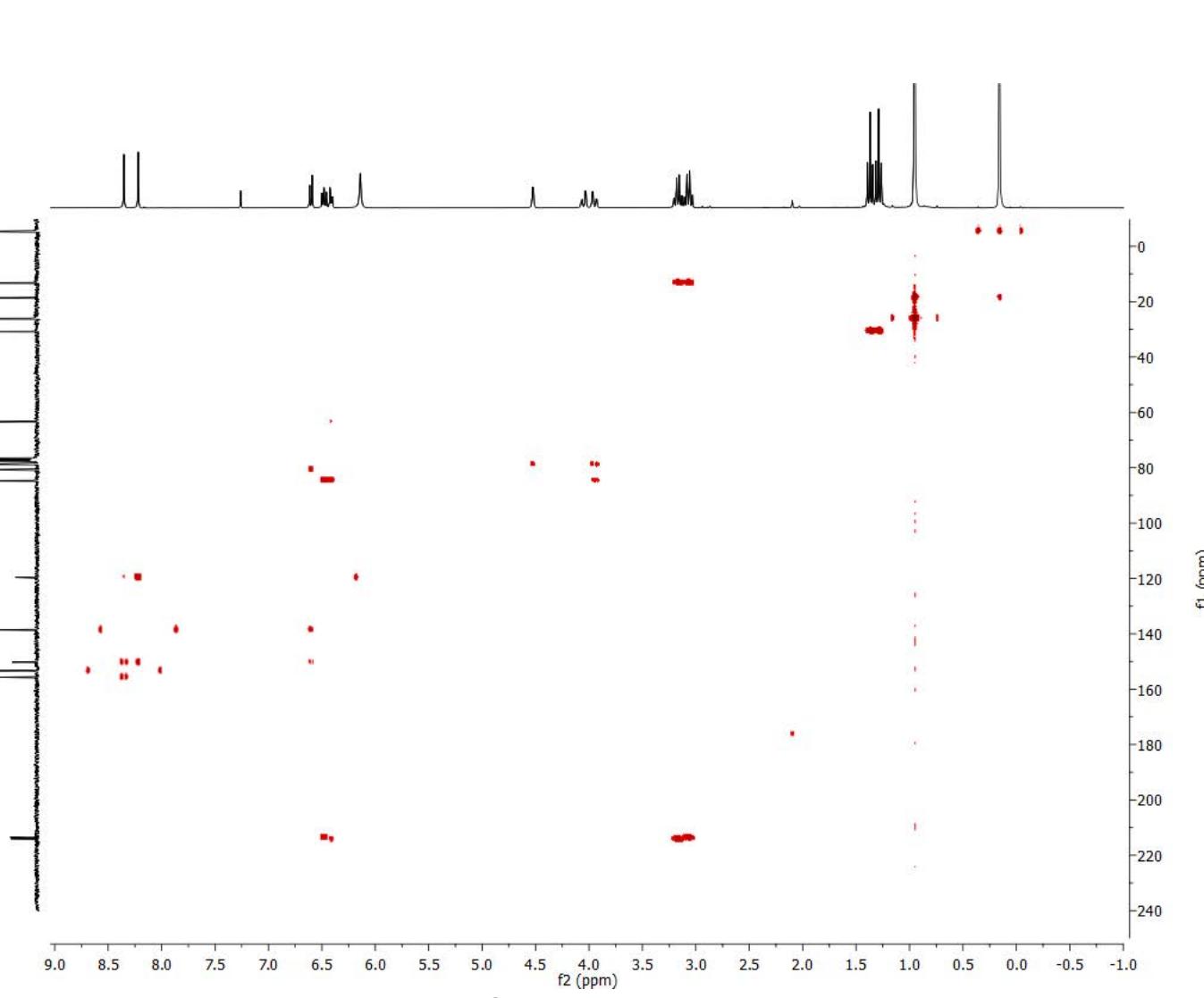
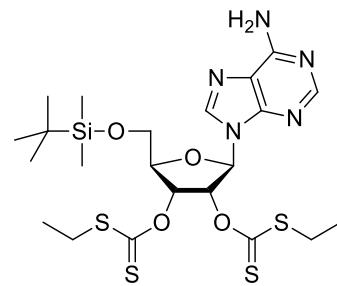
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[ethylthio)thiocarbonyl]- $\beta$ -D-adenosine (6e)**

HSQC NMR ( $\text{CDCl}_3$ )



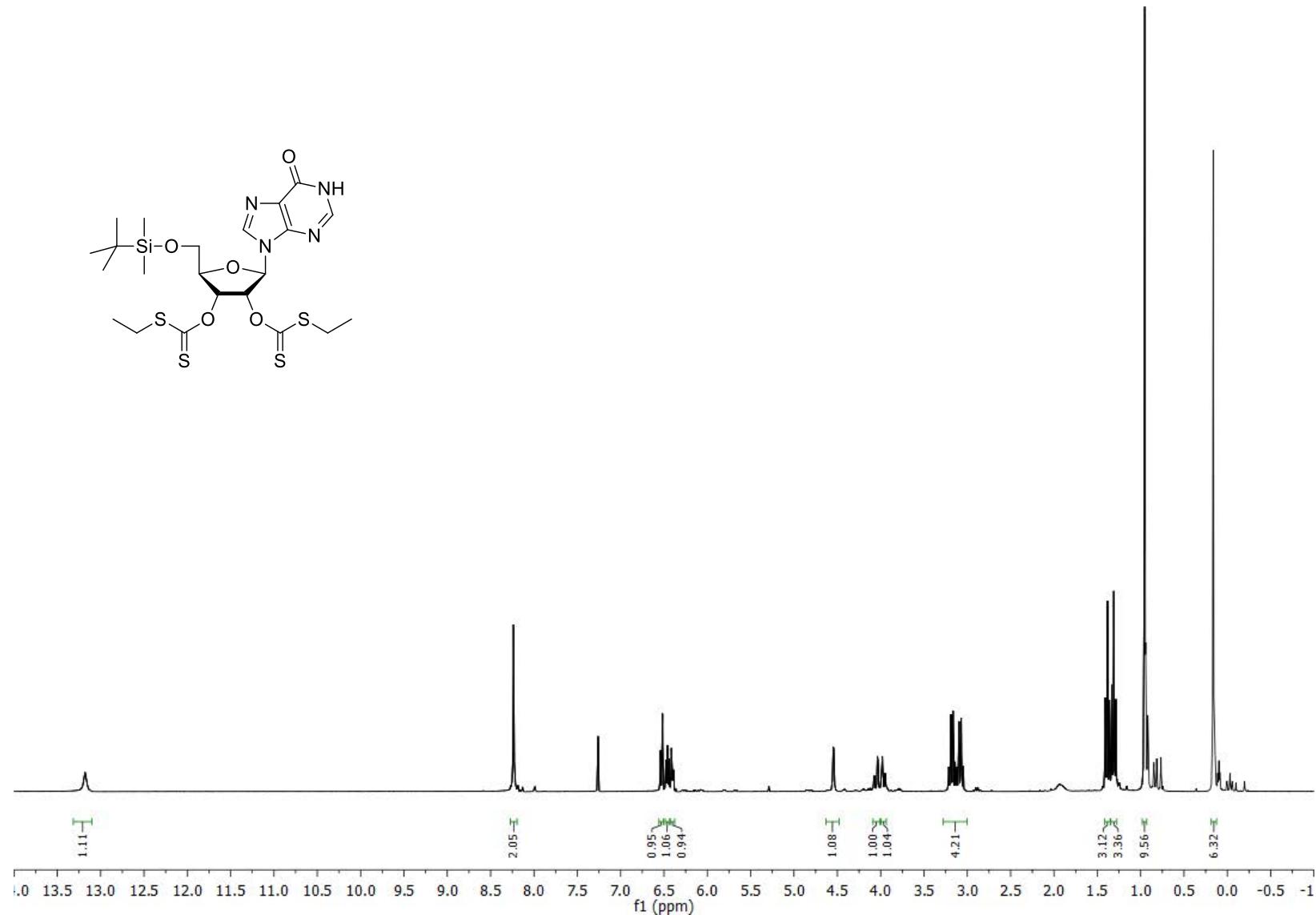
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-adenosine (6e)**

### HMBC NMR ( $\text{CDCl}_3$ )



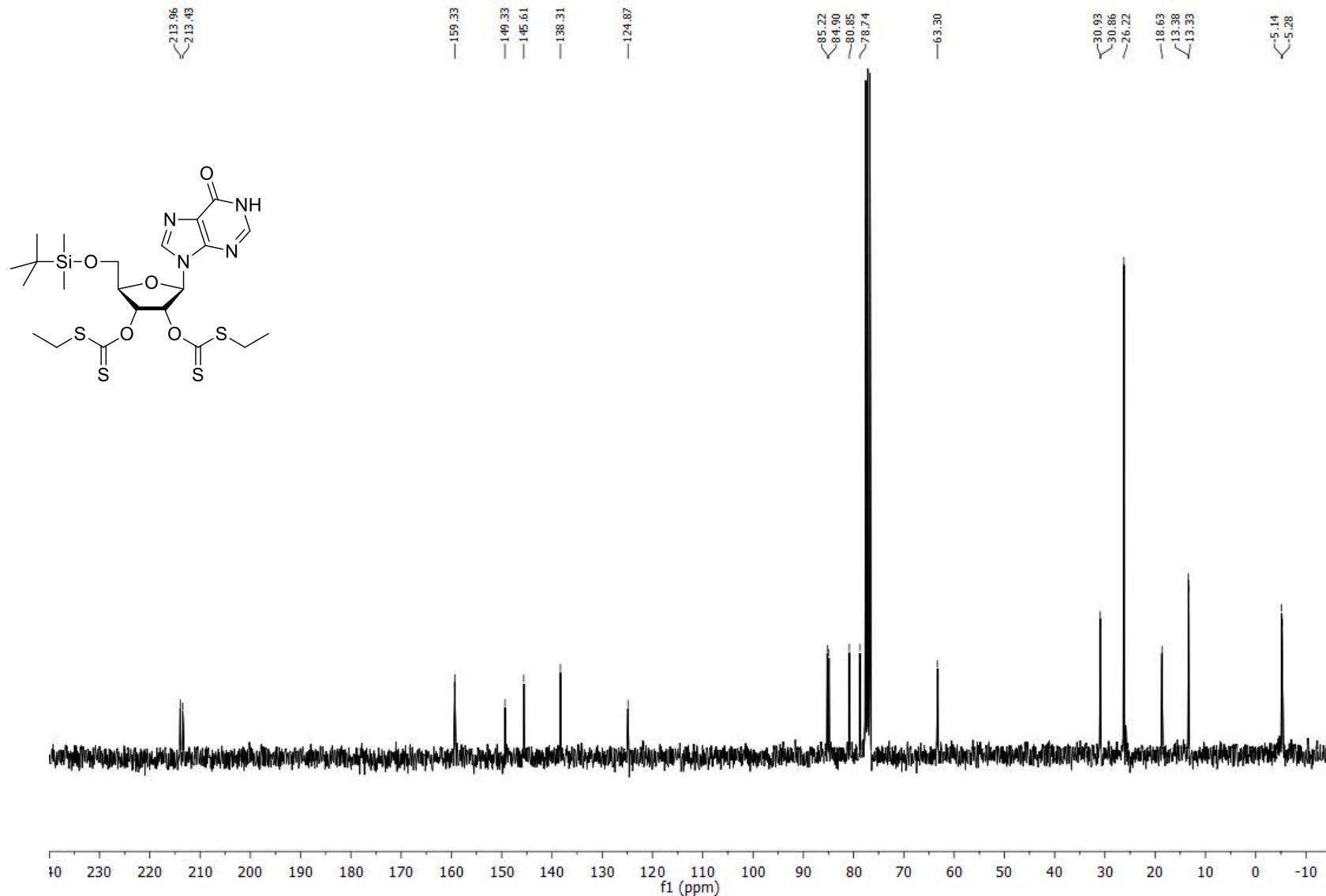
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-inosine (6f)**

$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



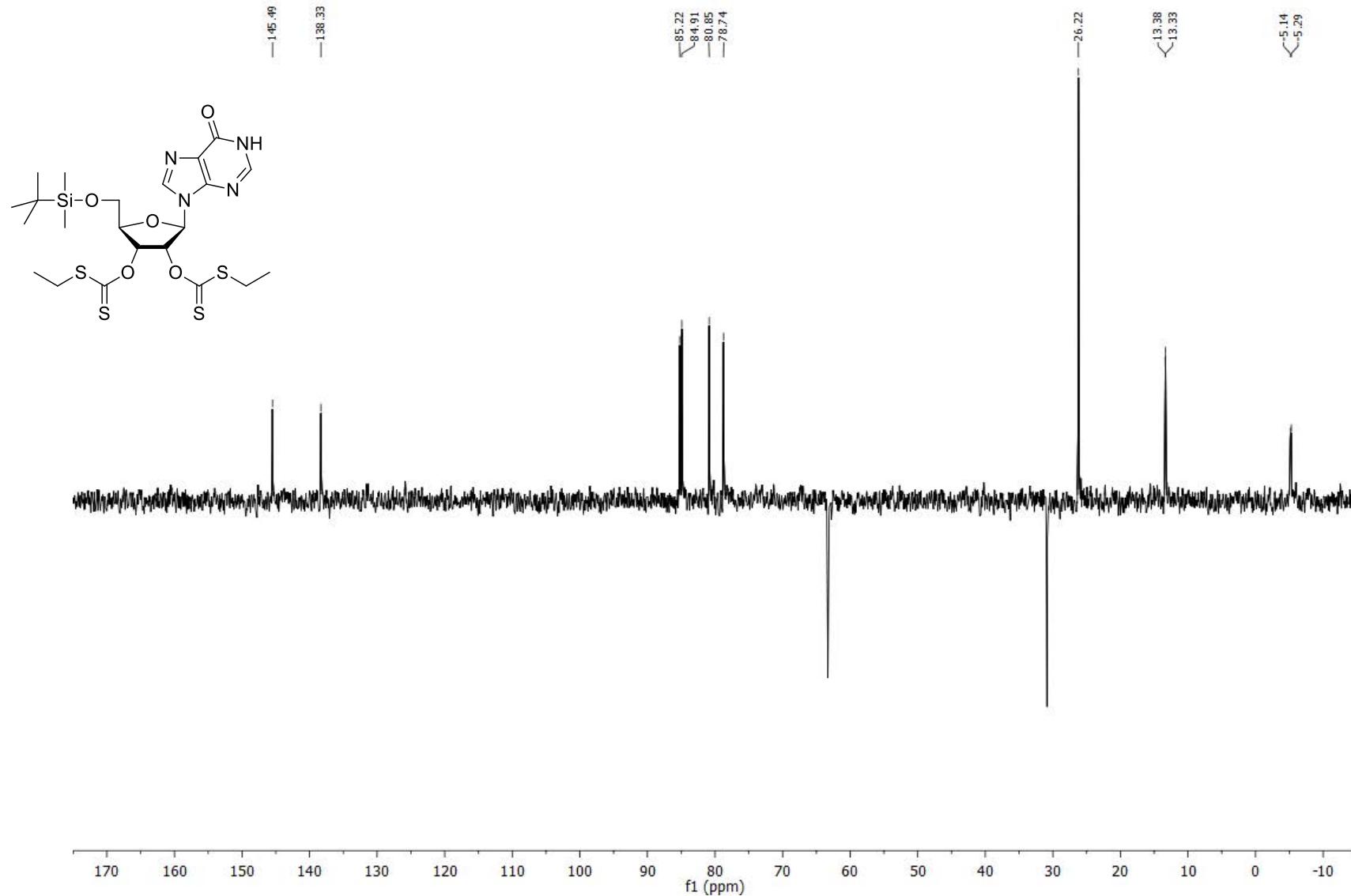
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-inosine (6f)**

$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



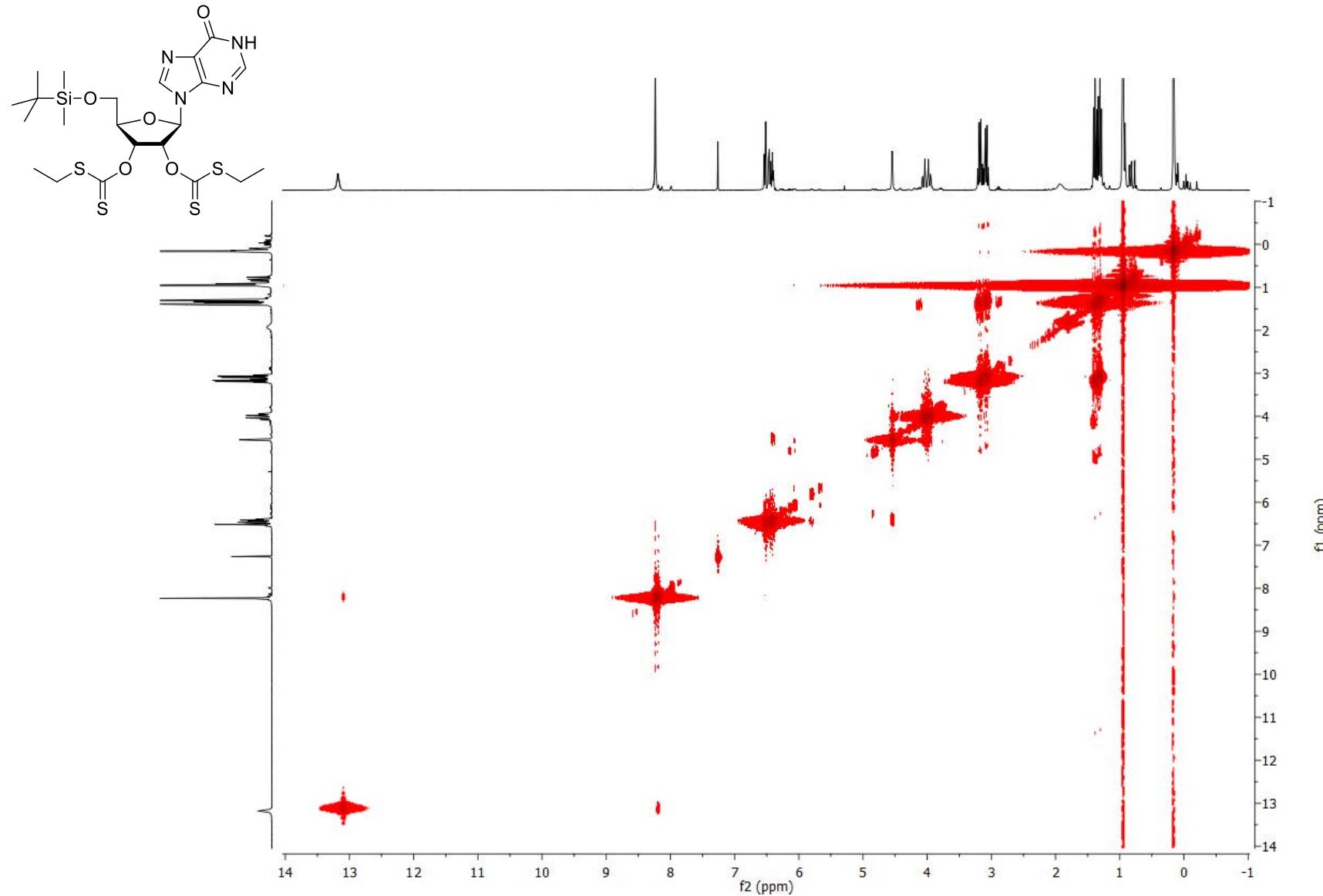
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-inosine (6f)**

DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



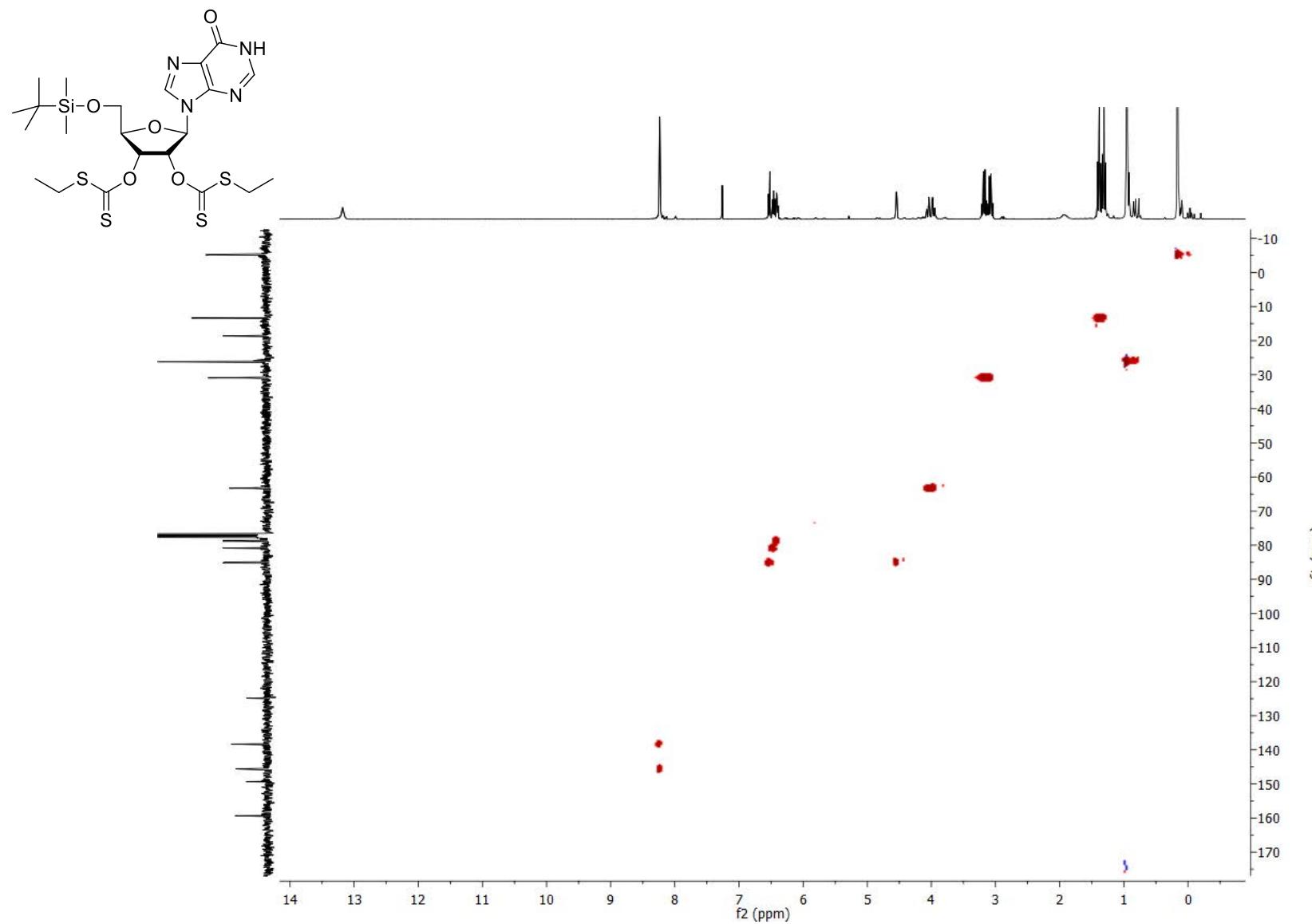
**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-bis-*O*-[(ethylthio)thiocarbonyl]- $\beta$ -D-inosine (6f)**

COSY NMR ( $\text{CDCl}_3$ )



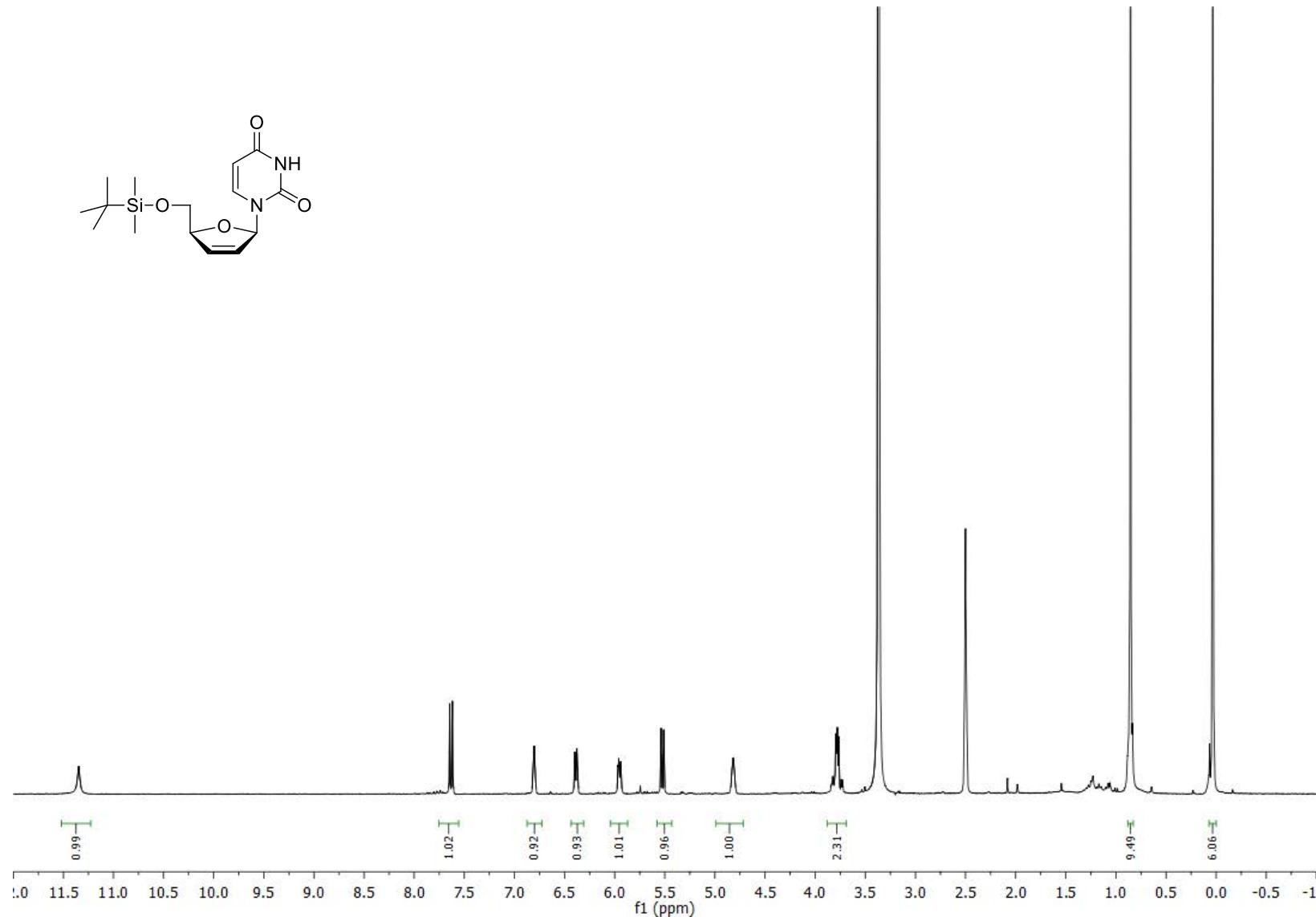
**5'-O-(tert-Butyldimethylsilyl)-2',3'-bis-O-[(ethylthio)thiocarbonyl]- $\beta$ -D-inosine (6f)**

HSQC NMR ( $\text{CDCl}_3$ )



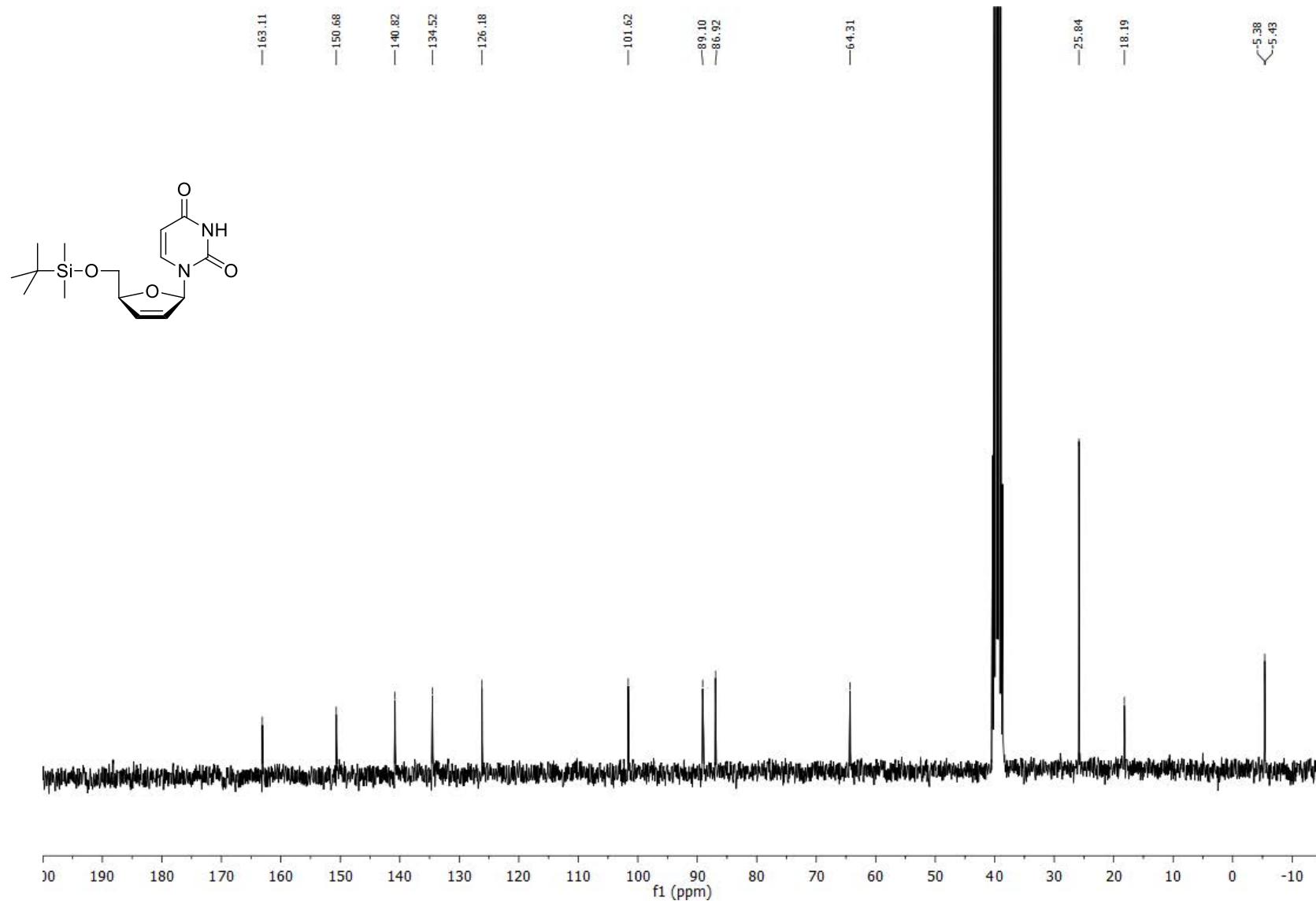
**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-uridine (7a)**

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



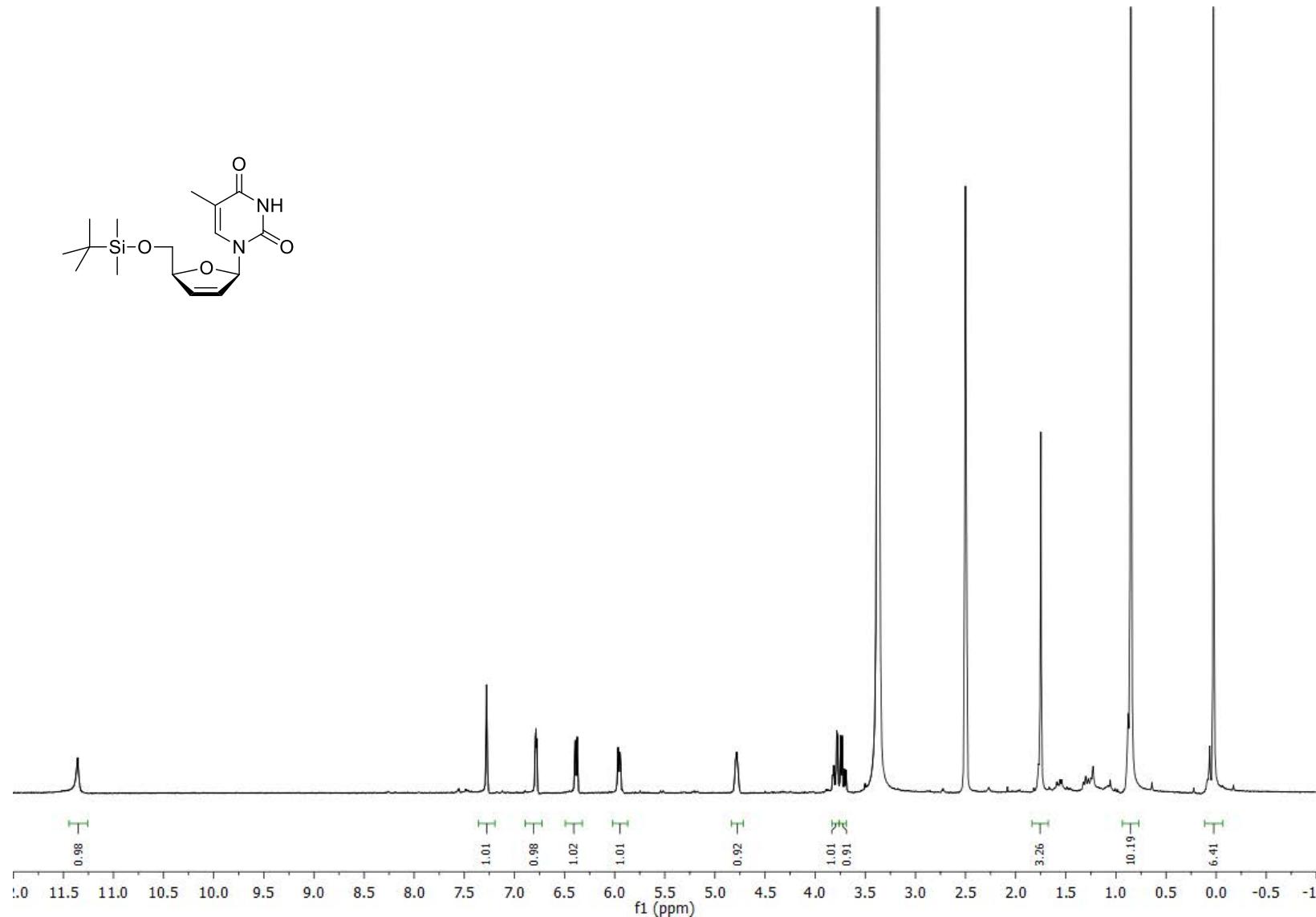
**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-uridine (7a)**

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



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

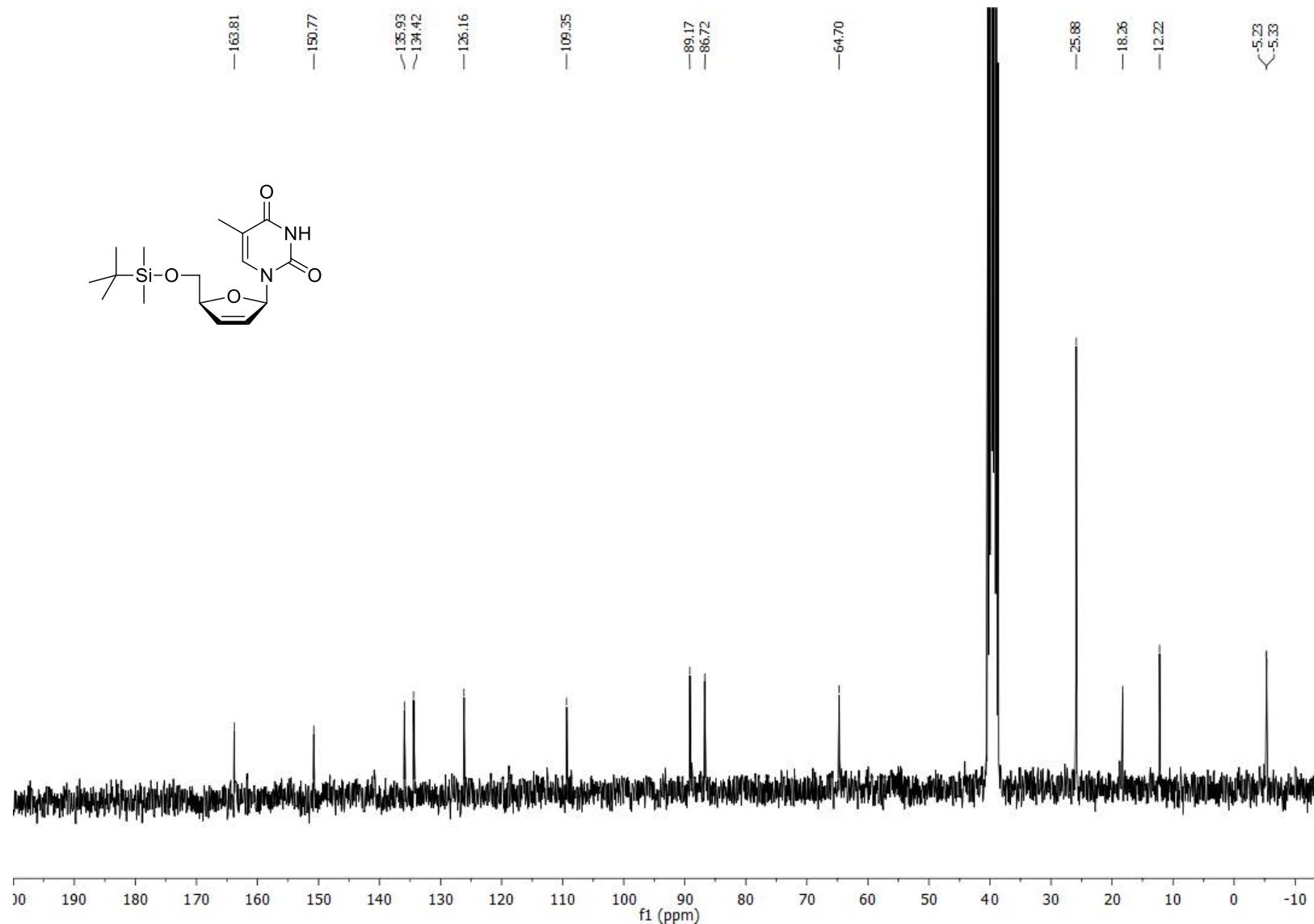
$^1\text{H}$ -NMR (300.13 MHz, DMSO- $d_6$ )



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

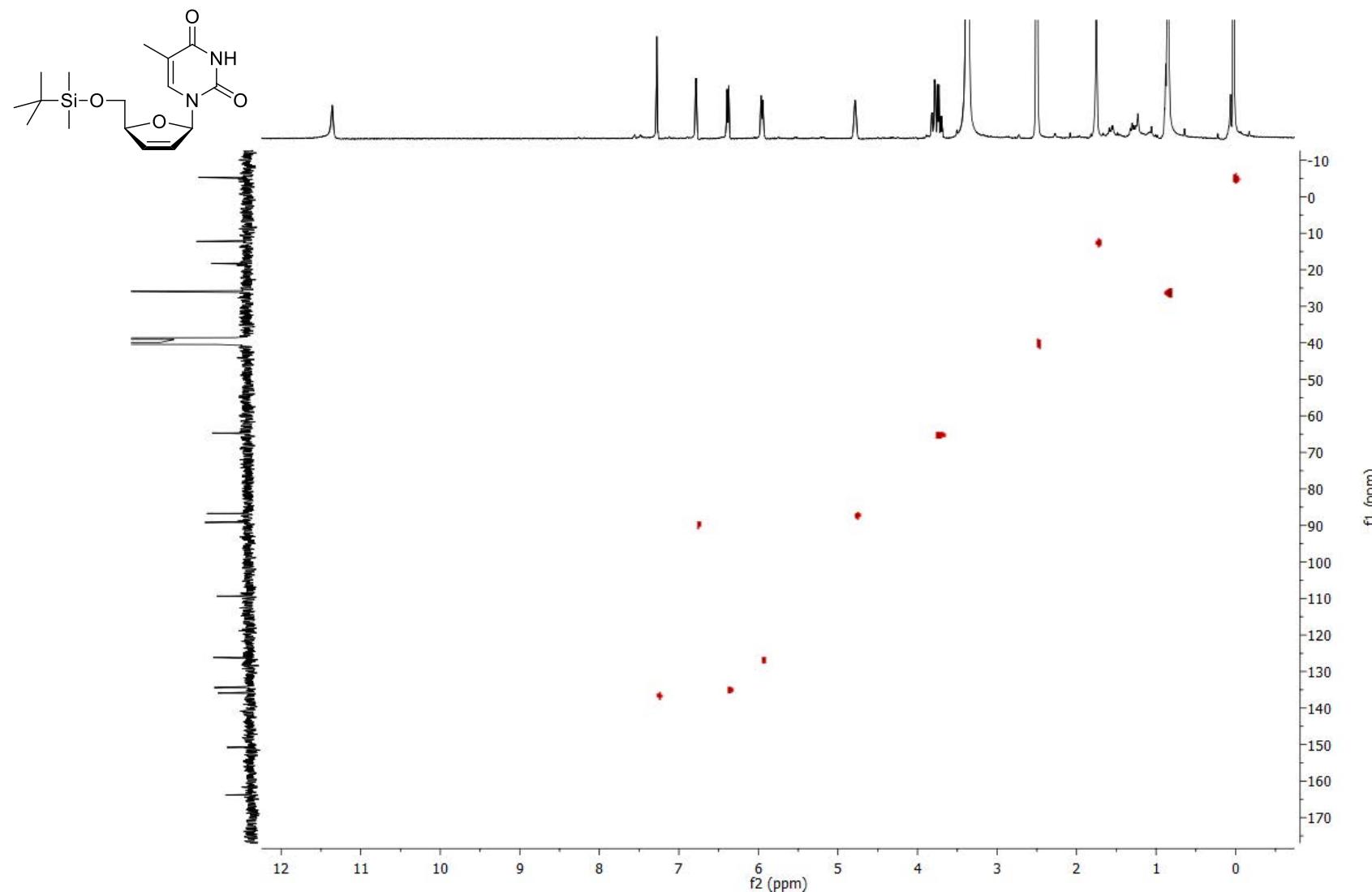
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$^{13}\text{C}$ -NMR (75.5 MHz, DMSO- $d_6$ )



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

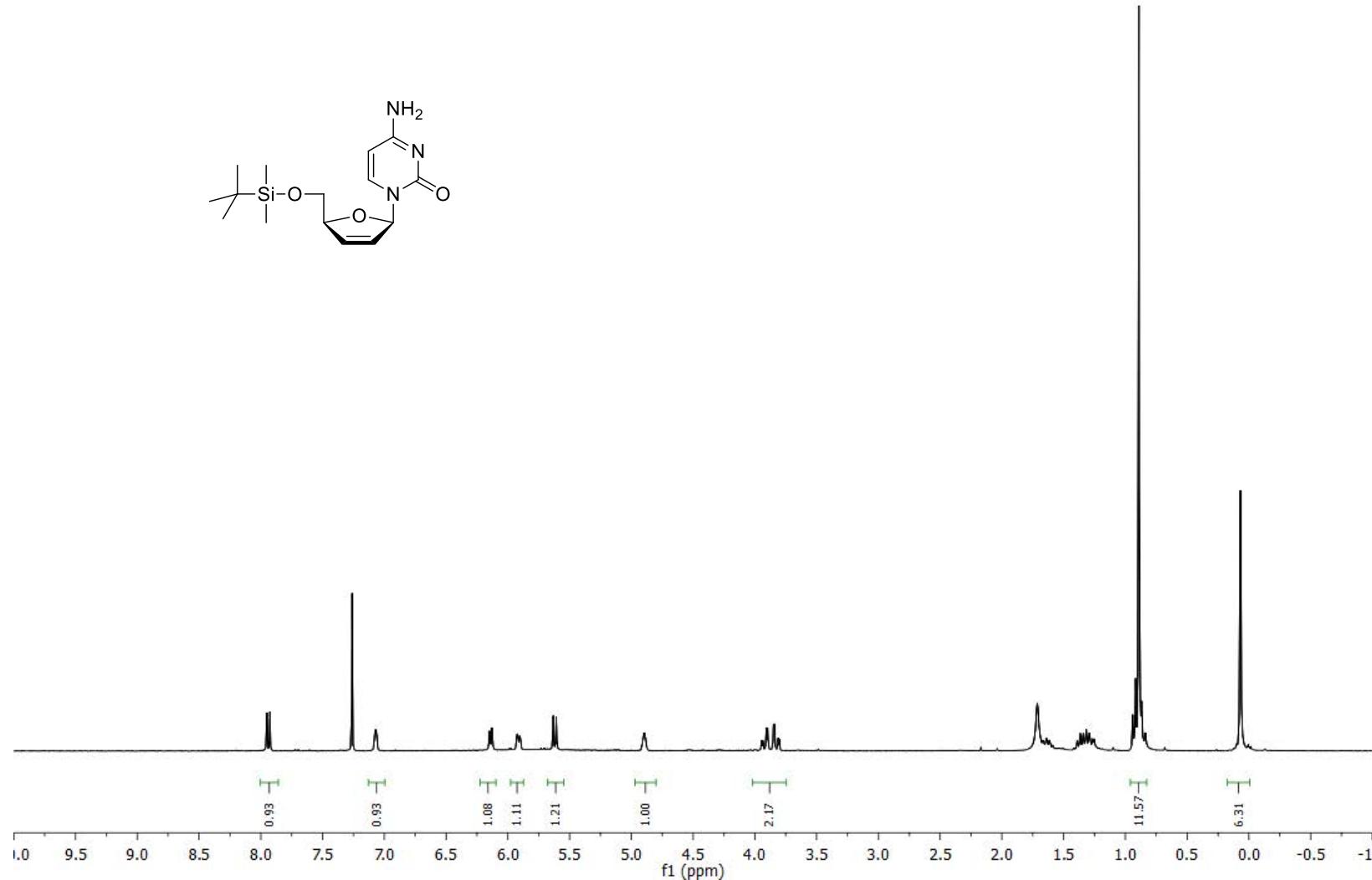
HSQC NMR (DMSO-*d*<sub>6</sub>)



**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7c)**

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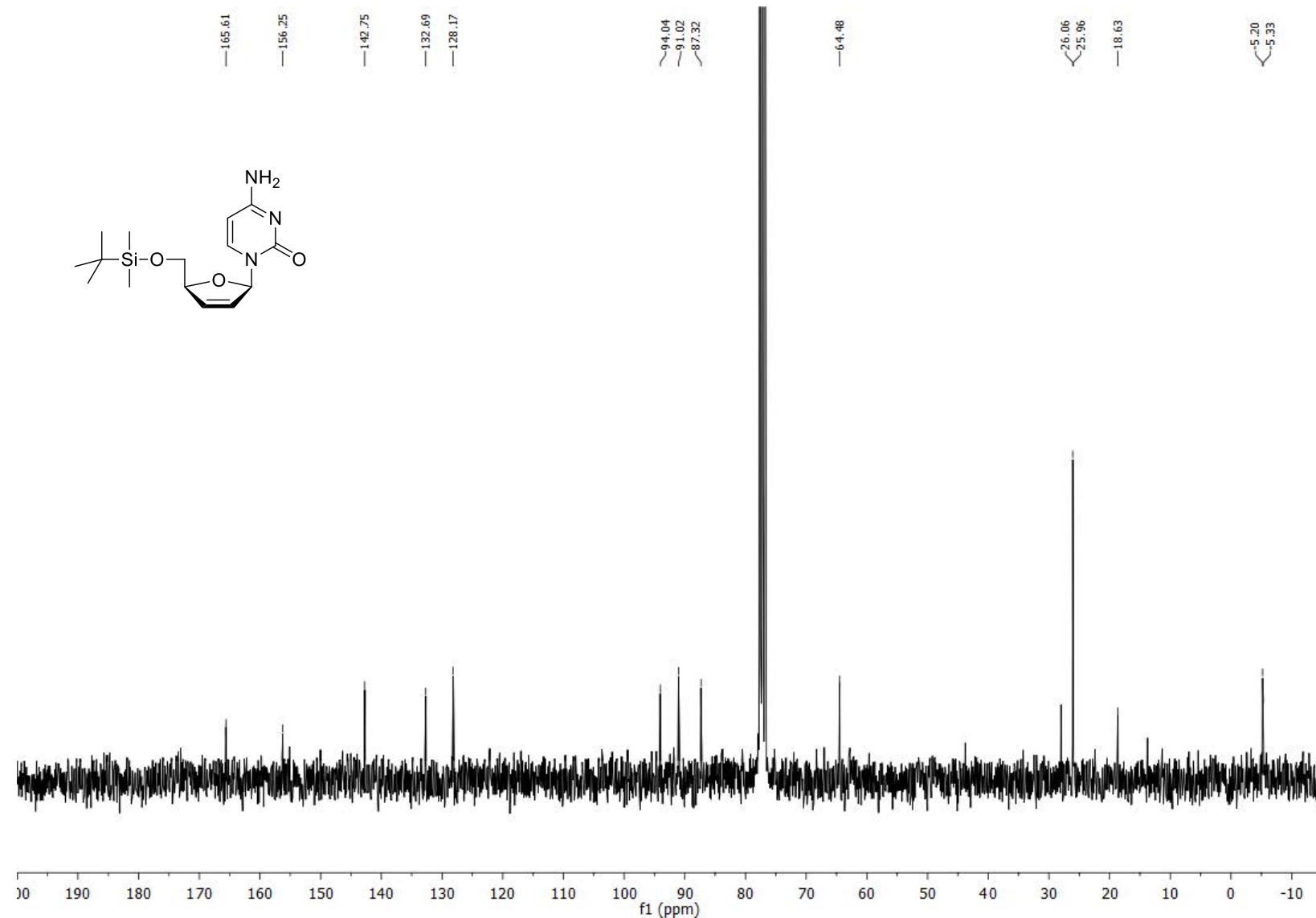
$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7c)**

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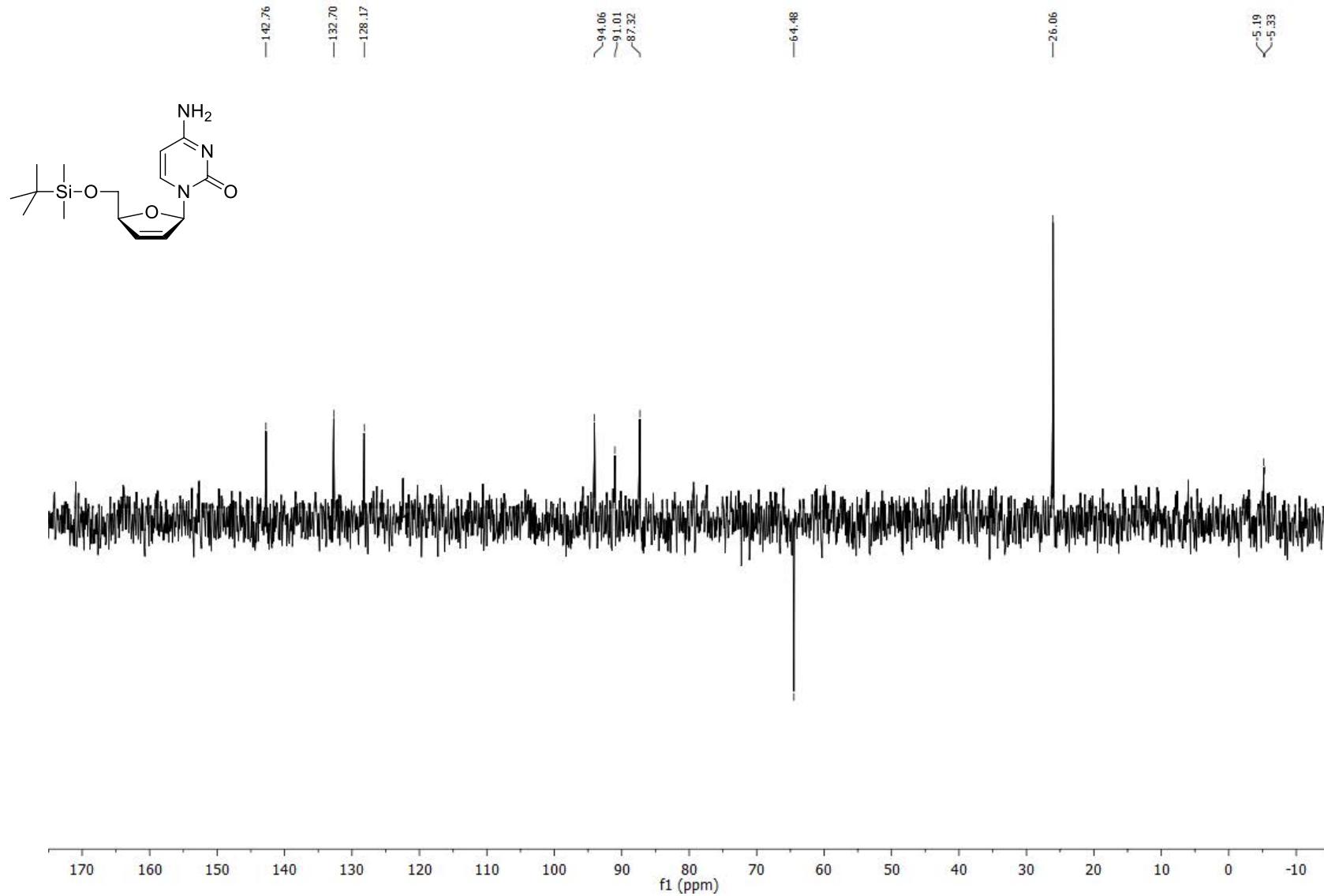
$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7c)**

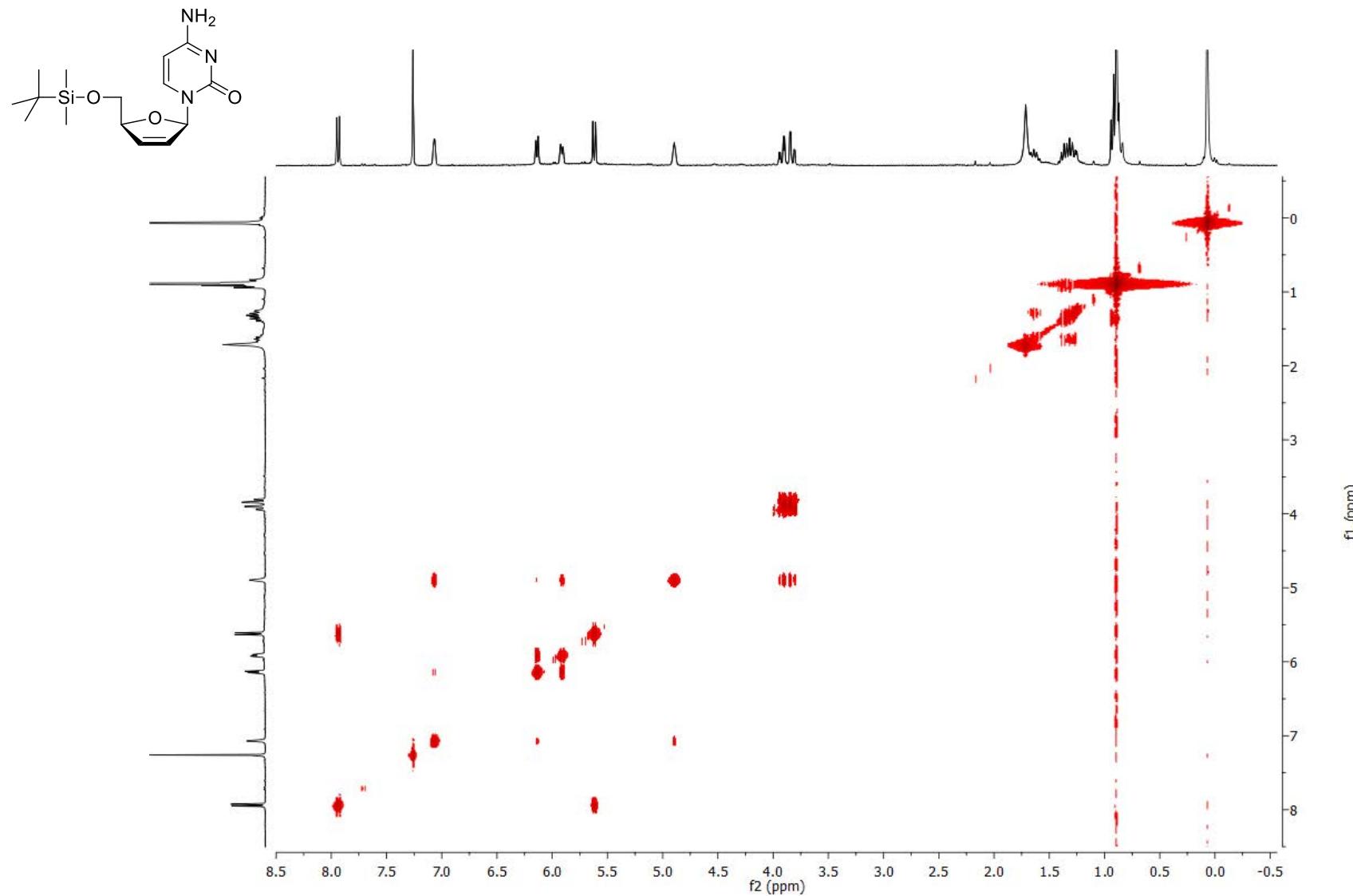
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DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



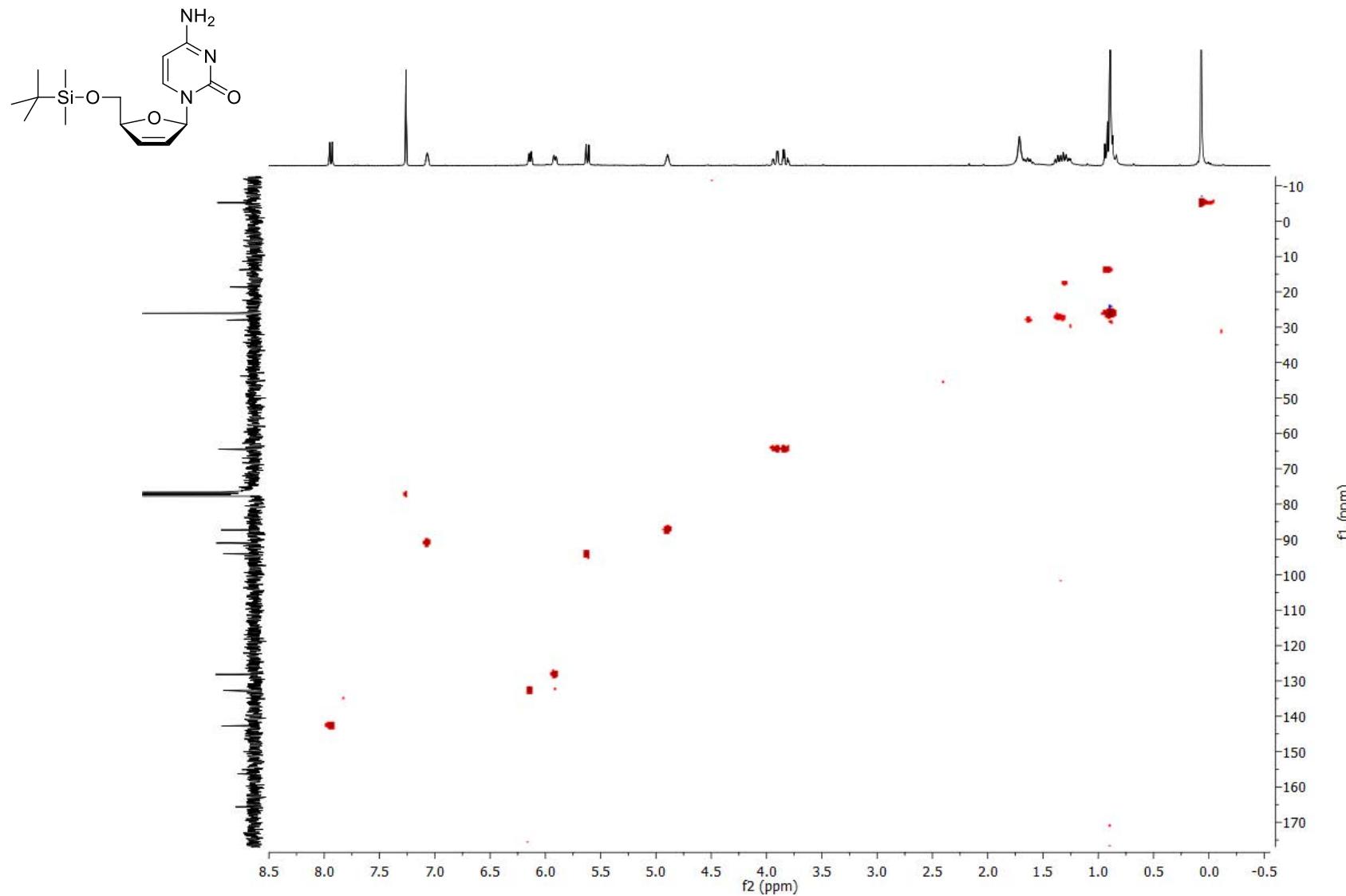
**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7c)**

COSY NMR ( $\text{CDCl}_3$ )



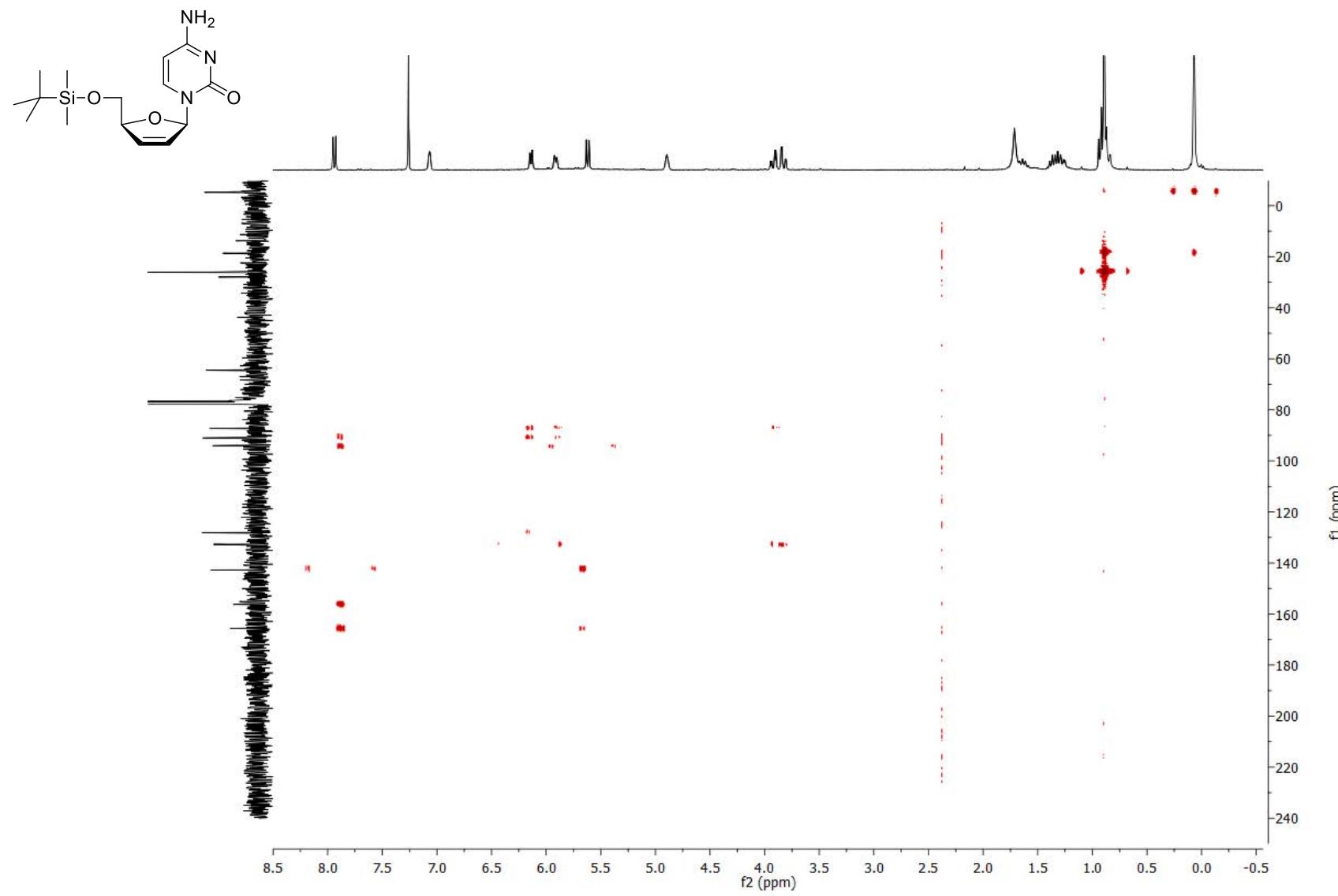
**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7c)**

HSQC NMR ( $\text{CDCl}_3$ )



**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7c)**

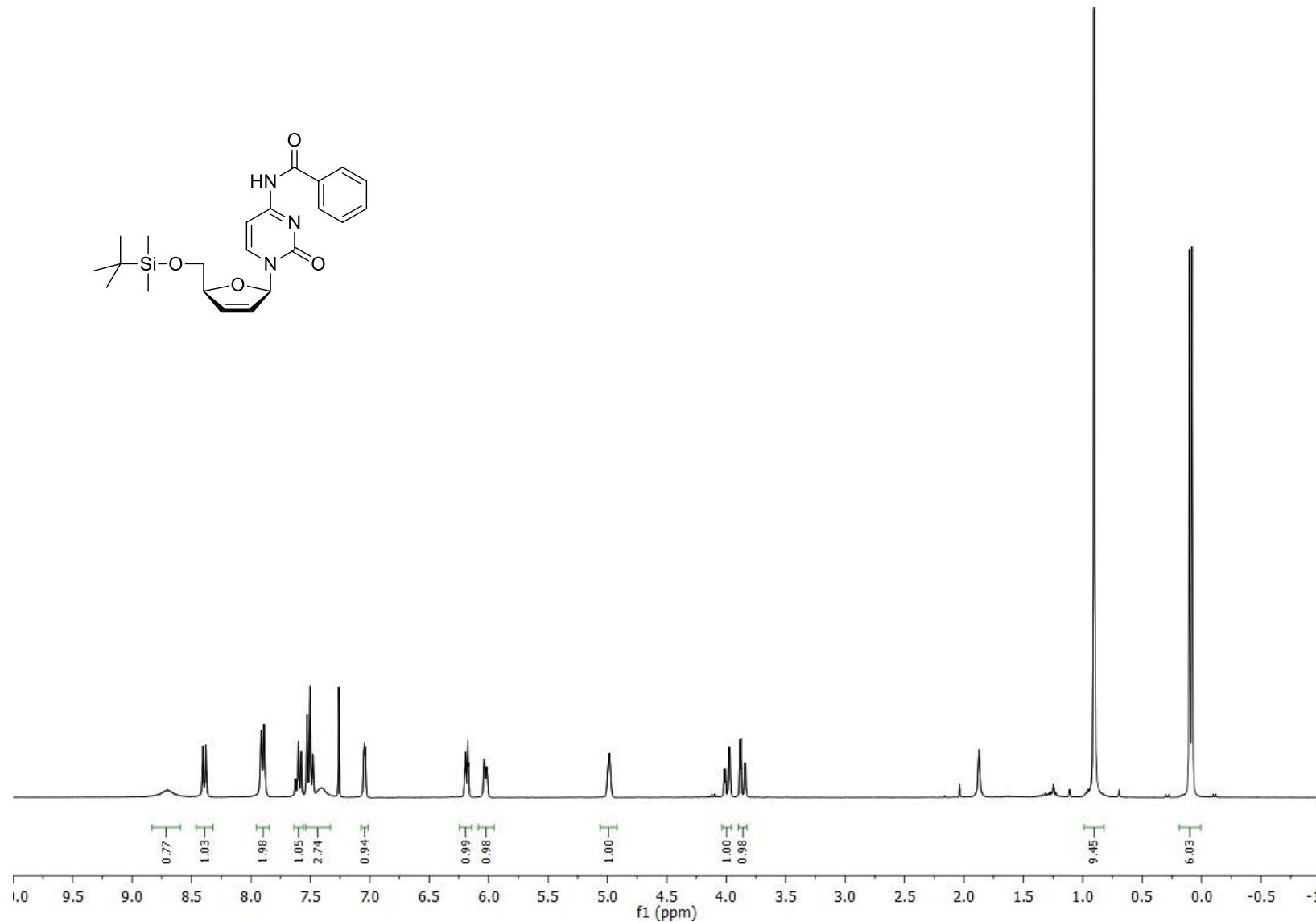
HMBC NMR ( $\text{CDCl}_3$ )



S100

*N*<sup>4</sup>-Benzoyl-5'-*O*-(*tert*-butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7d)

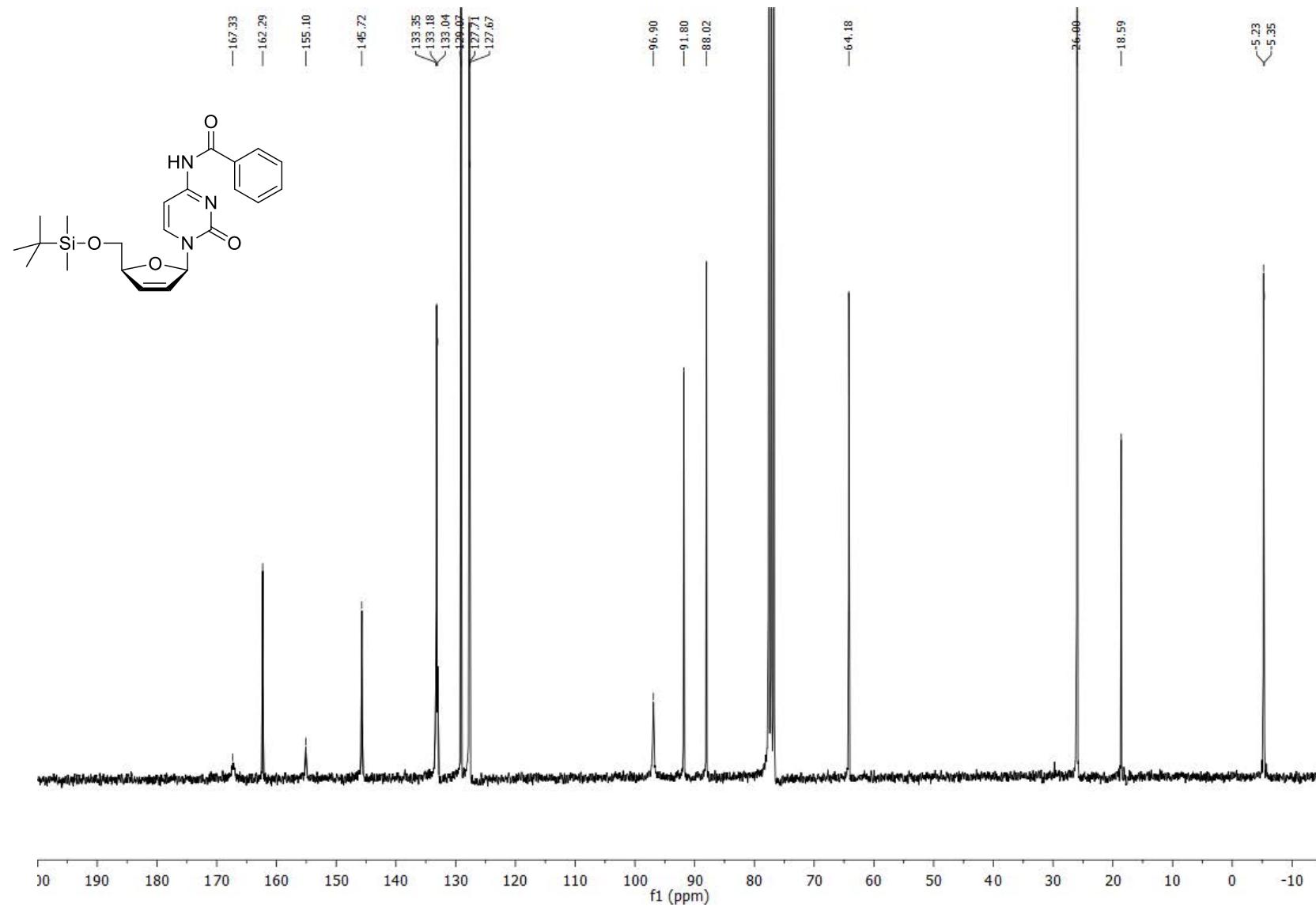
<sup>1</sup>H-NMR (300.13 MHz, CDCl<sub>3</sub>)



S101

***N*<sup>4</sup>-Benzoyl-5'-O-(*tert*-butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7d)**

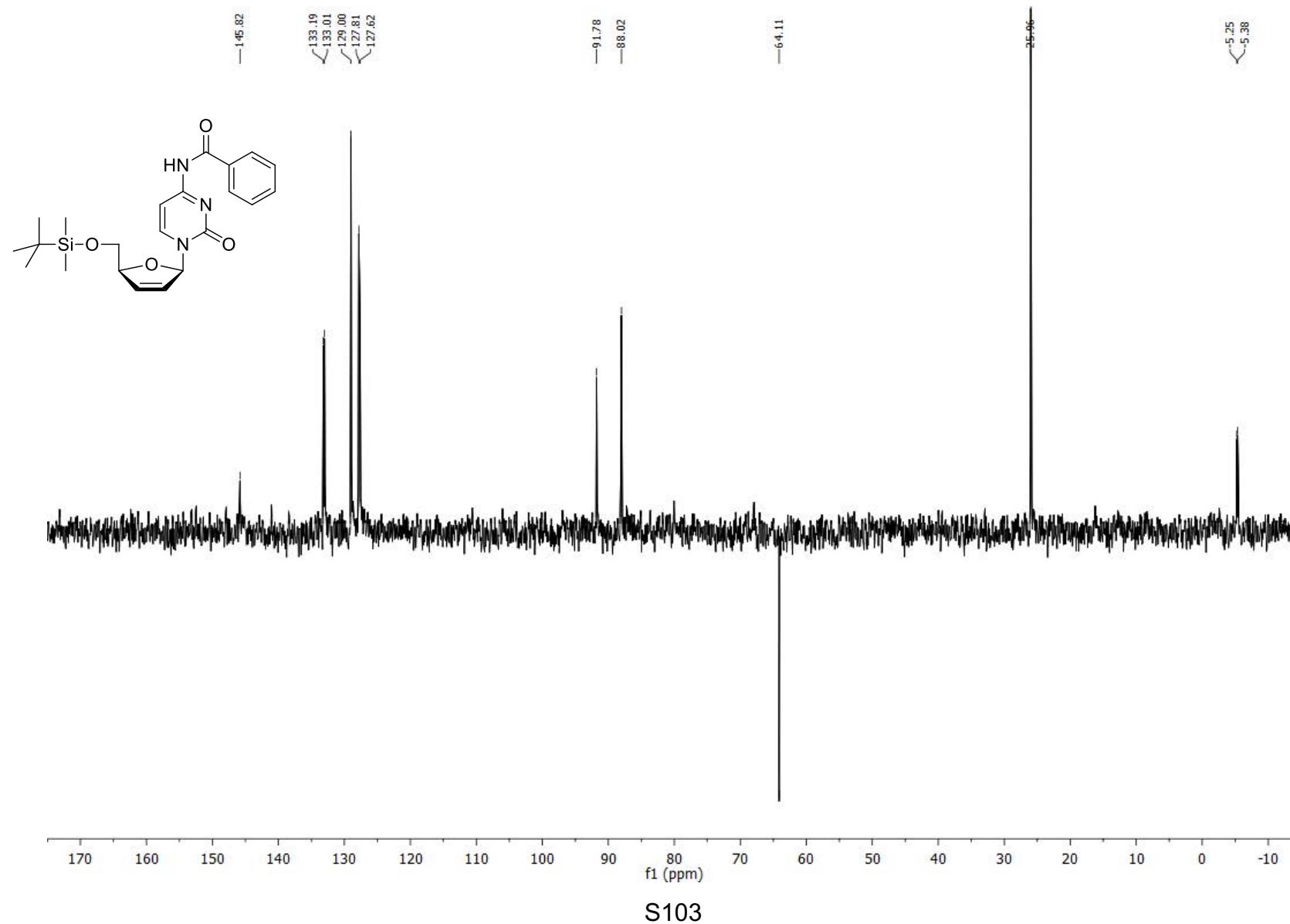
<sup>13</sup>C-NMR (75.5 MHz, CDCl<sub>3</sub>)



***N*<sup>4</sup>-Benzoyl-5'-*O*-(*tert*-butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7d)**

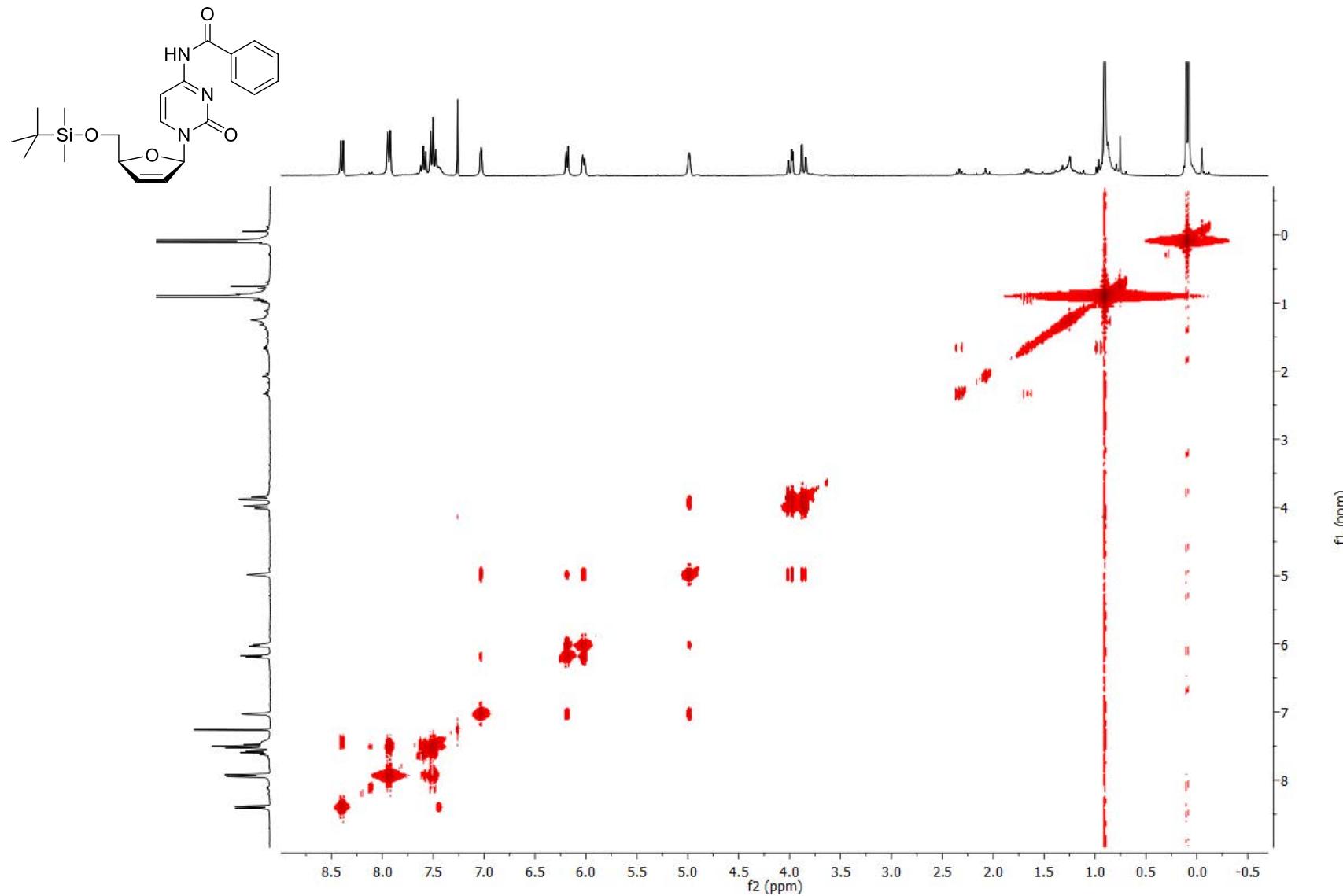
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DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



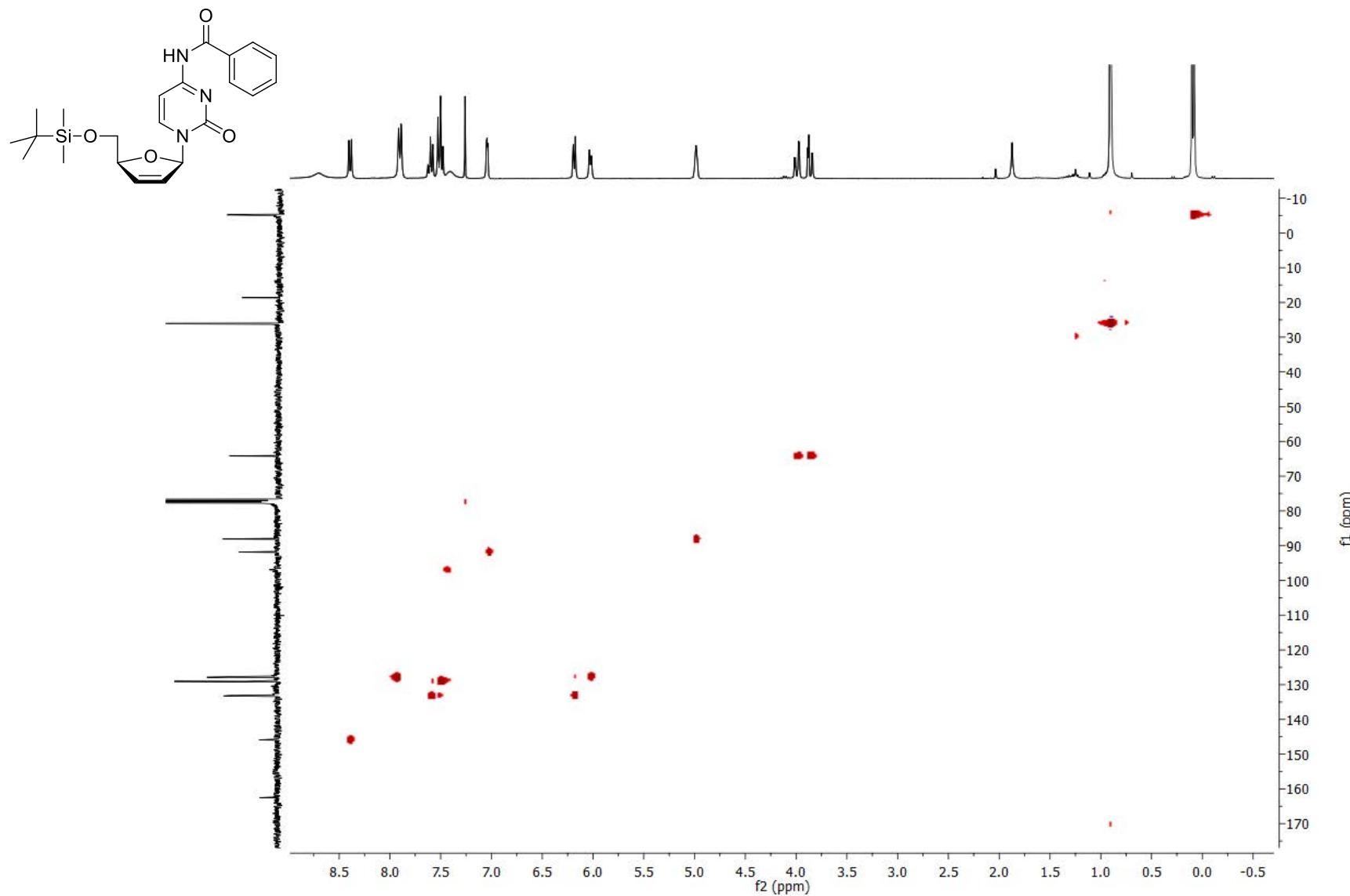
*N*<sup>4</sup>-Benzoyl-5'-*O*-(*tert*-butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7d)

COSY NMR ( $\text{CDCl}_3$ )



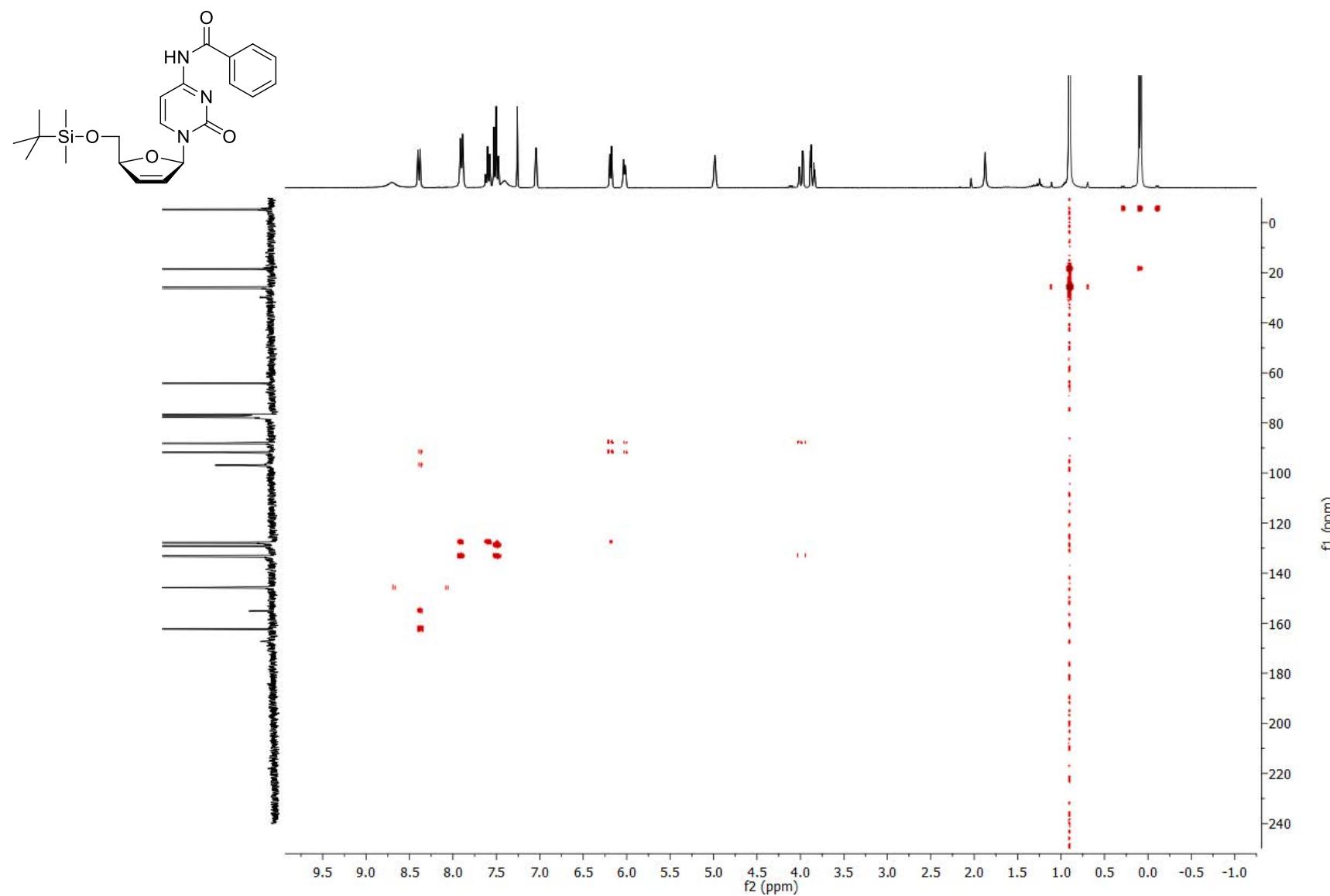
***N*<sup>4</sup>-Benzoyl-5'-*O*-(tert-butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7d)**

HSQC NMR ( $\text{CDCl}_3$ )



***N*<sup>4</sup>-Benzoyl-5'-*O*-(*tert*-butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (7d)**

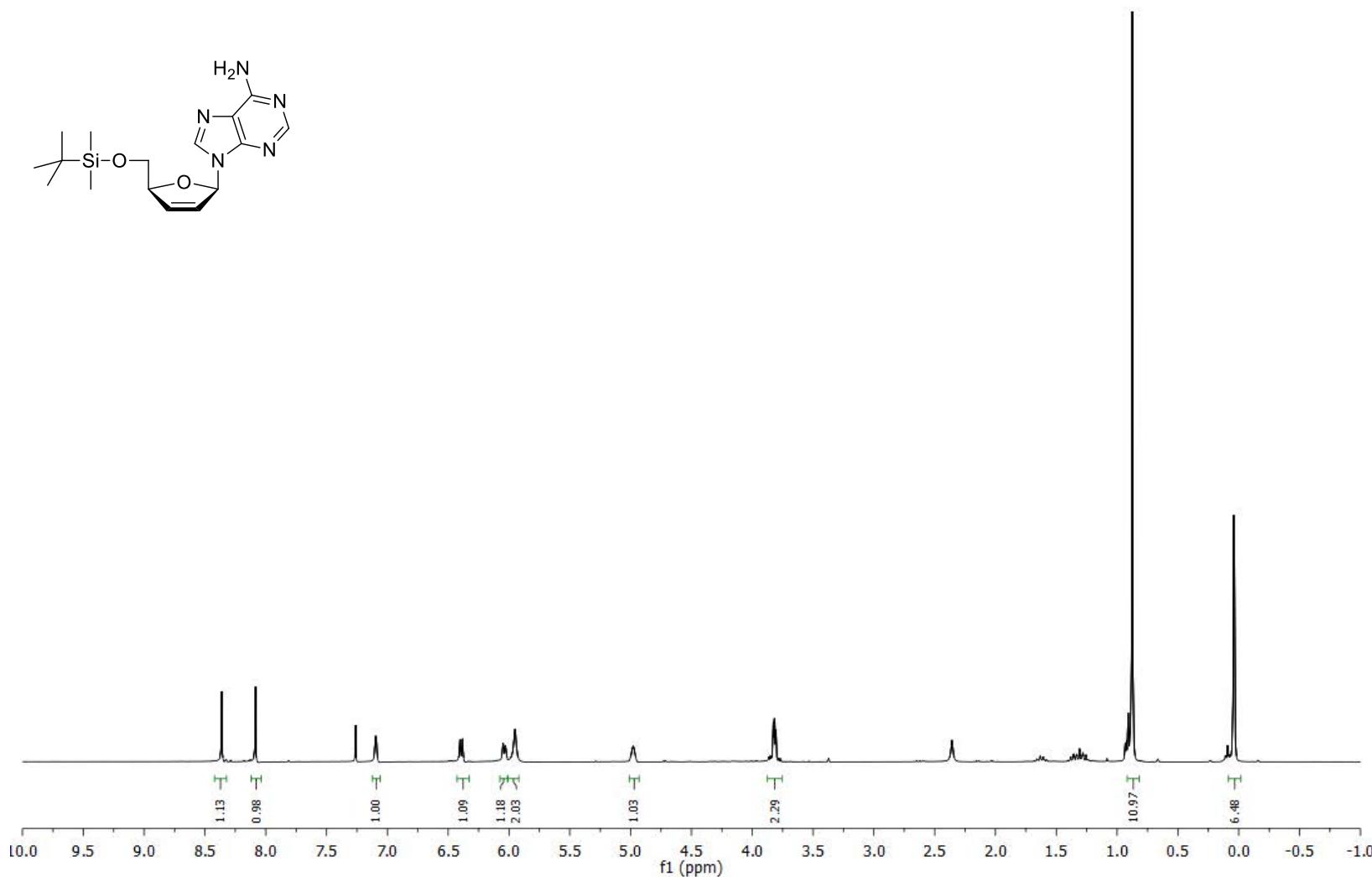
HMBC NMR ( $\text{CDCl}_3$ )



**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-adenosine (7e)**

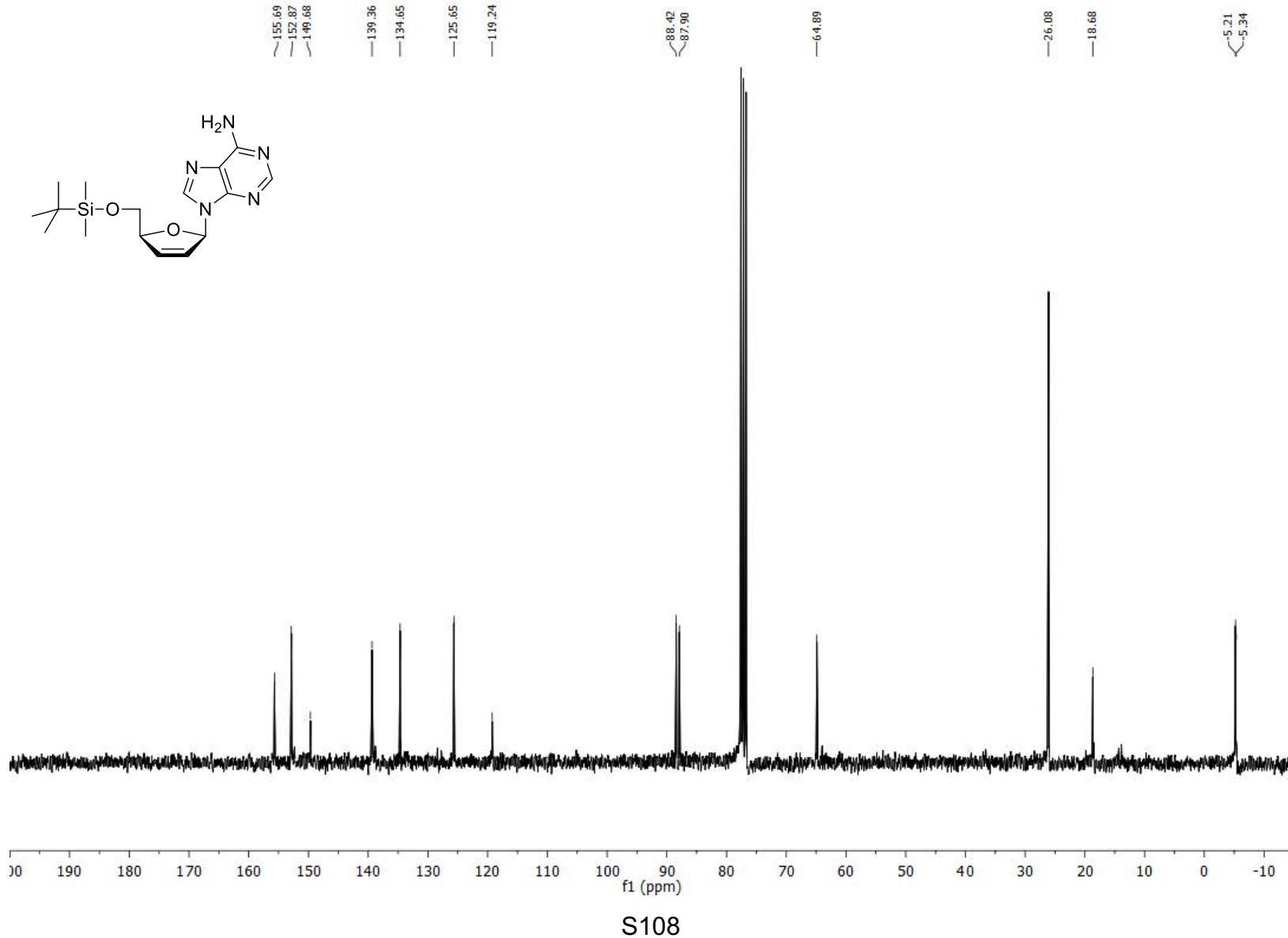
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$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



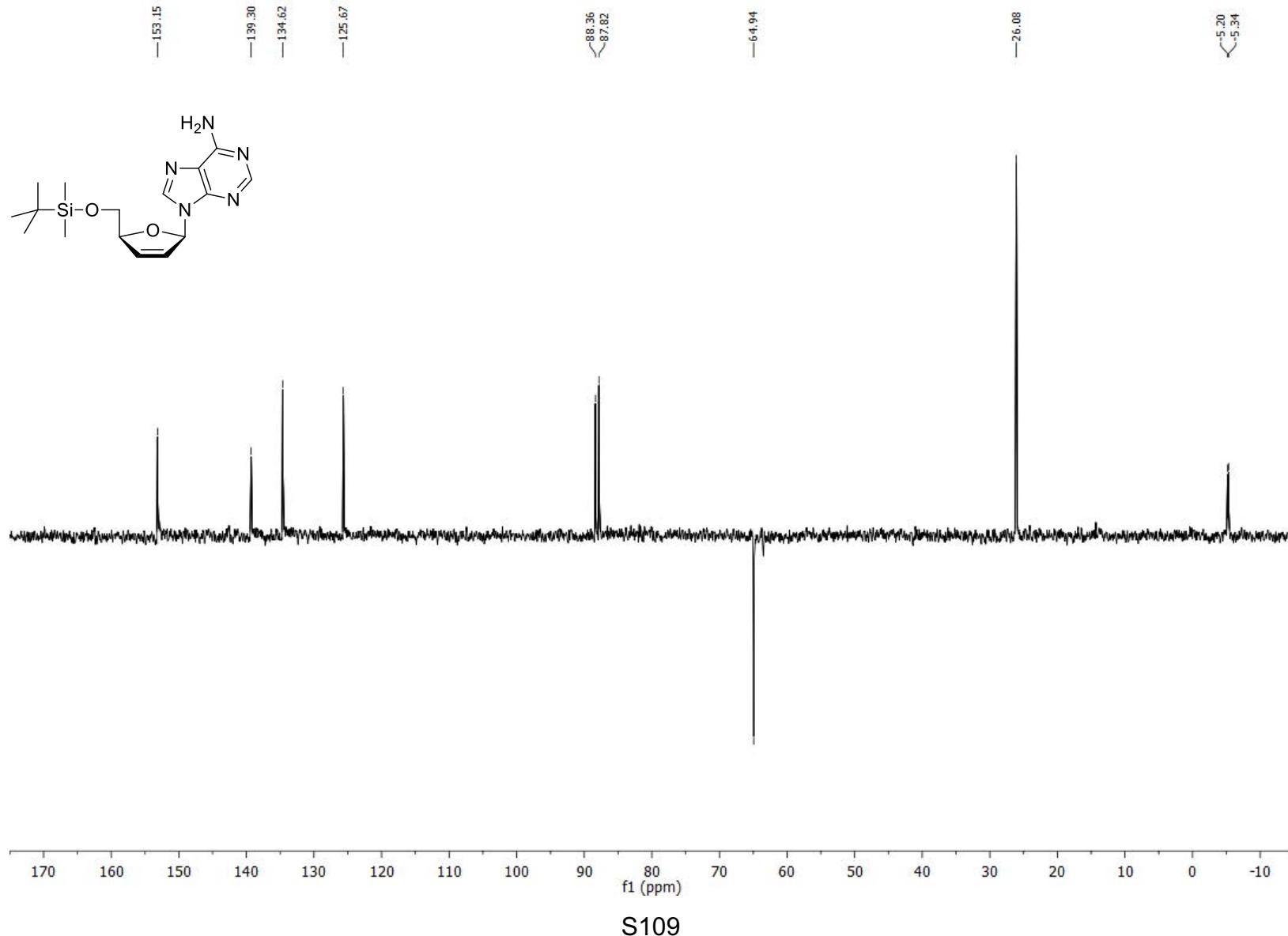
**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-adenosine (7e)**

$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



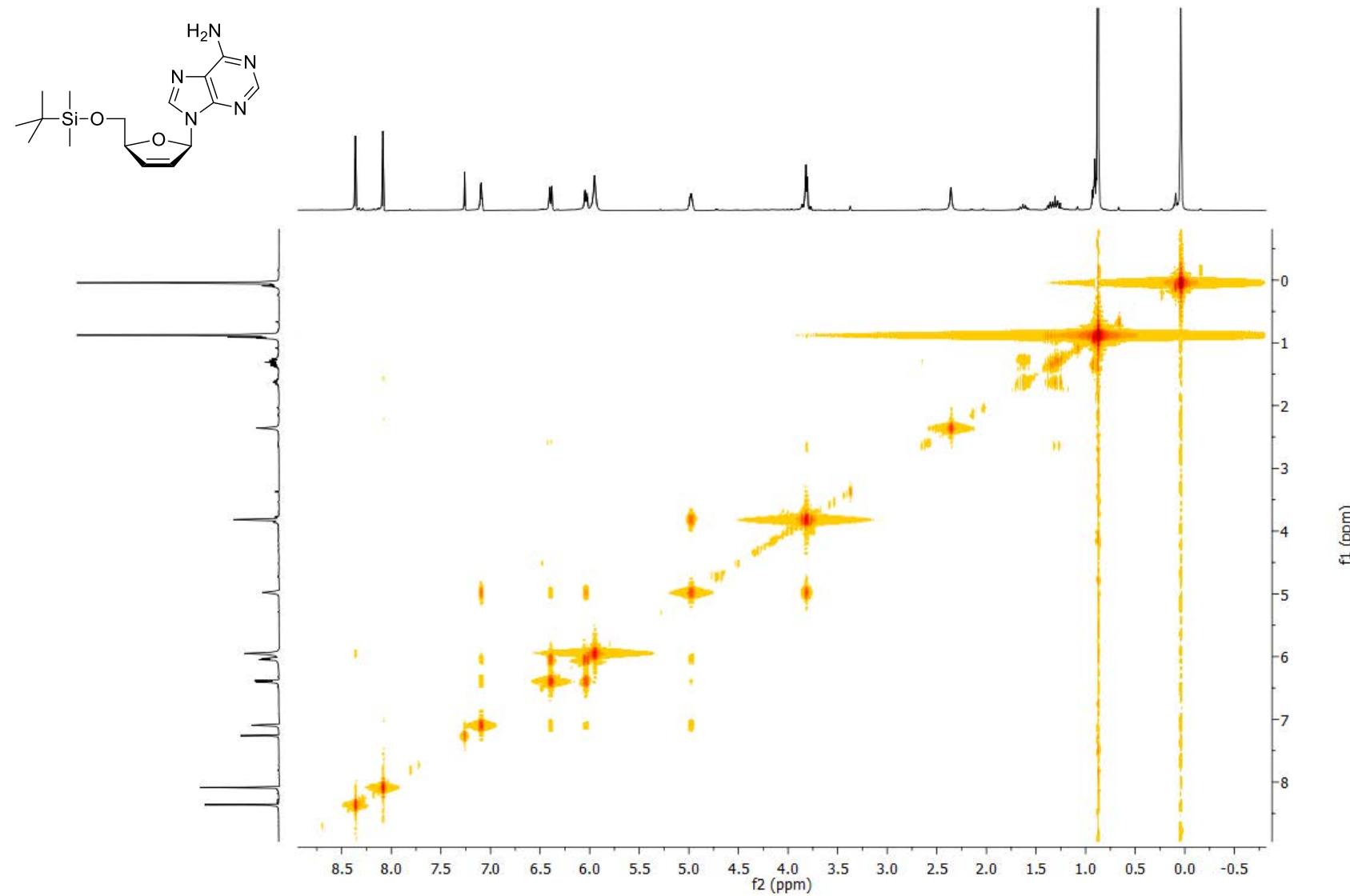
**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-adenosine (7e)**

DEPT NMR (75.5 MHz, CDCl<sub>3</sub>)



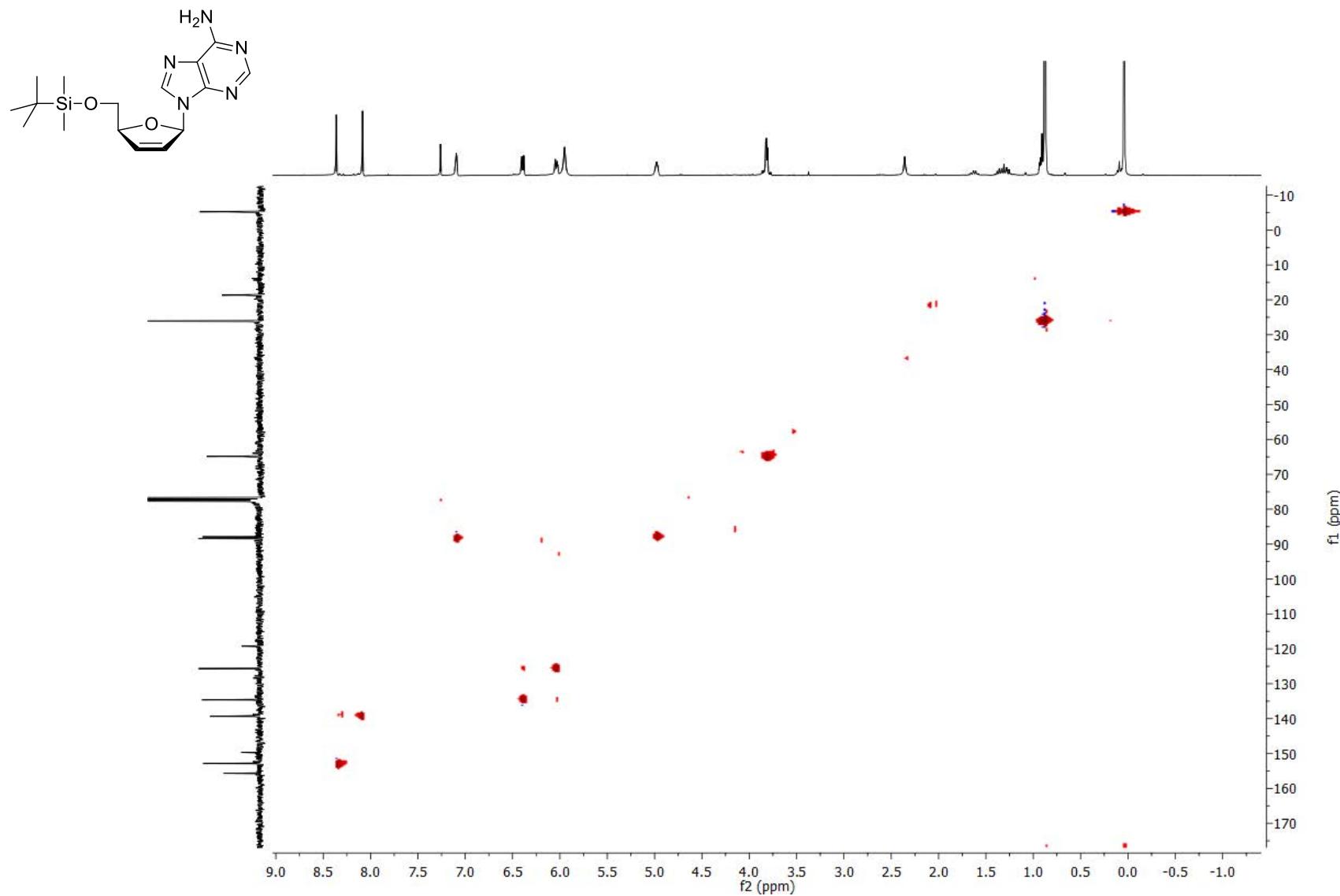
**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-adenosine (7e)**

COSY NMR ( $\text{CDCl}_3$ )



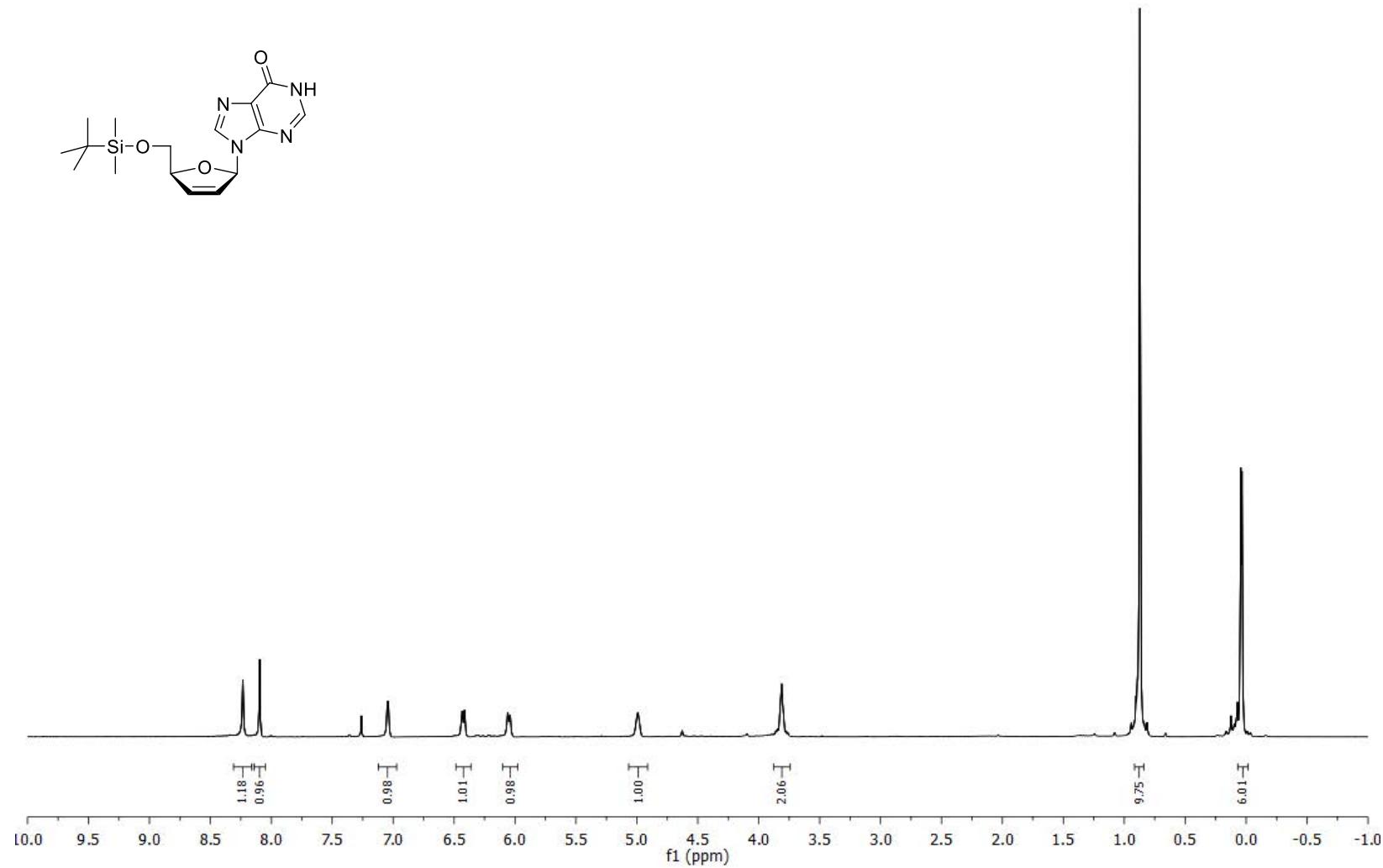
**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-adenosine (7e)**

HSQC NMR ( $\text{CDCl}_3$ )



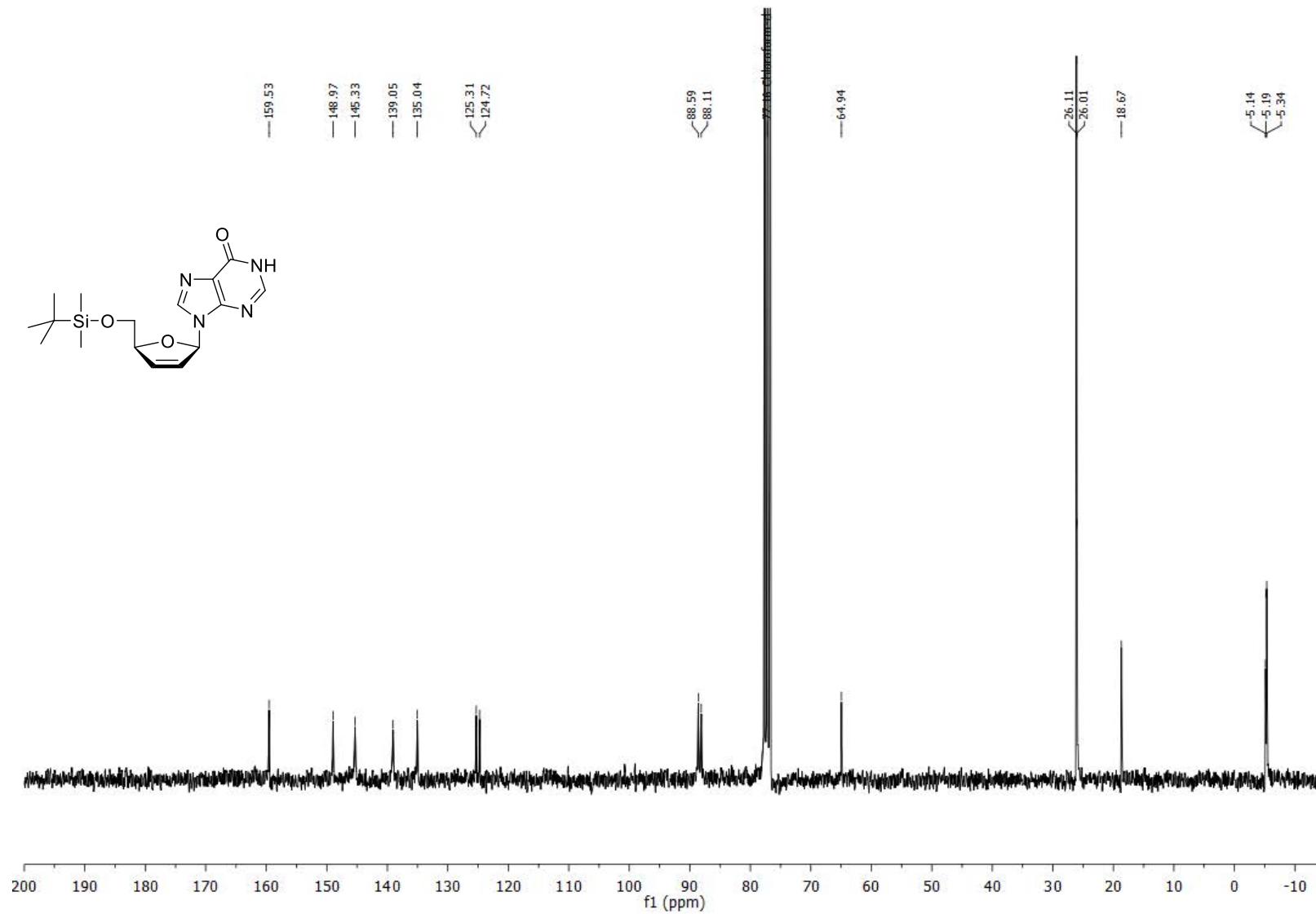
**5'-O-(*tert*-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-inosine (7f)**

$^1\text{H}$ -NMR (300.13 MHz,  $\text{CDCl}_3$ )



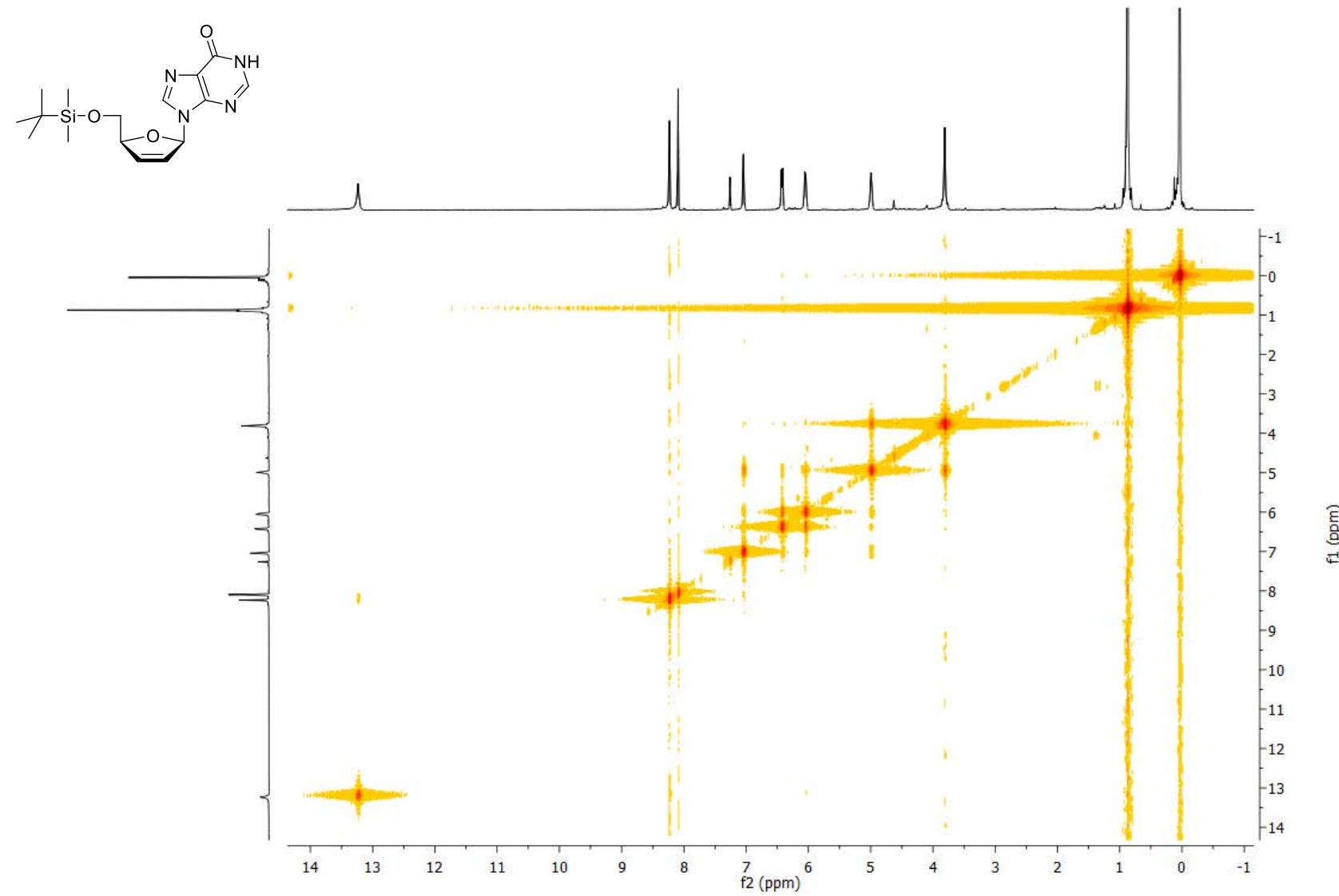
**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-inosine (7f)**

$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{CDCl}_3$ )



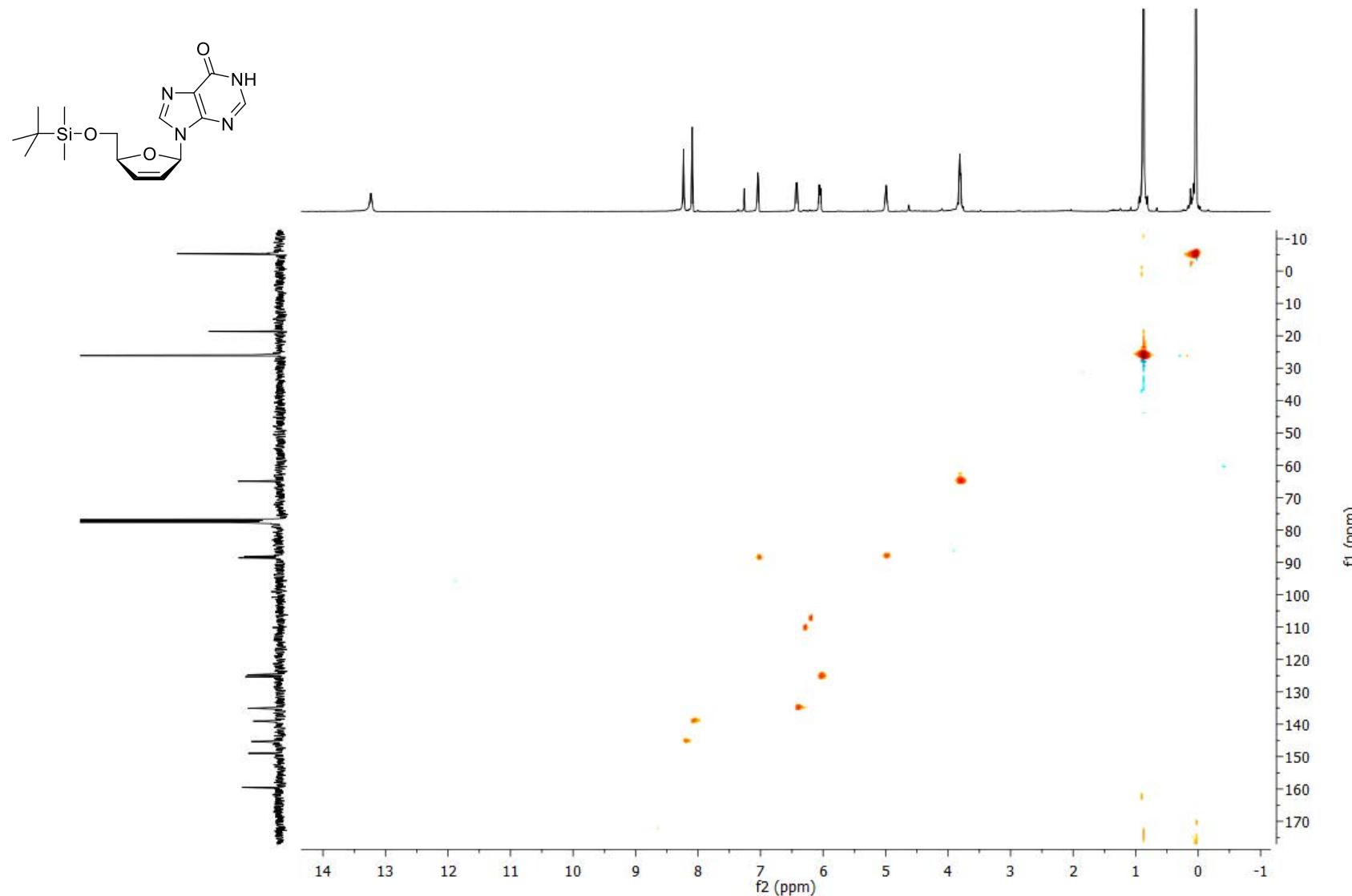
**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-inosine (7f)**

COSY NMR ( $\text{CDCl}_3$ )



**5'-O-(tert-Butyldimethylsilyl)-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-inosine (7f)**

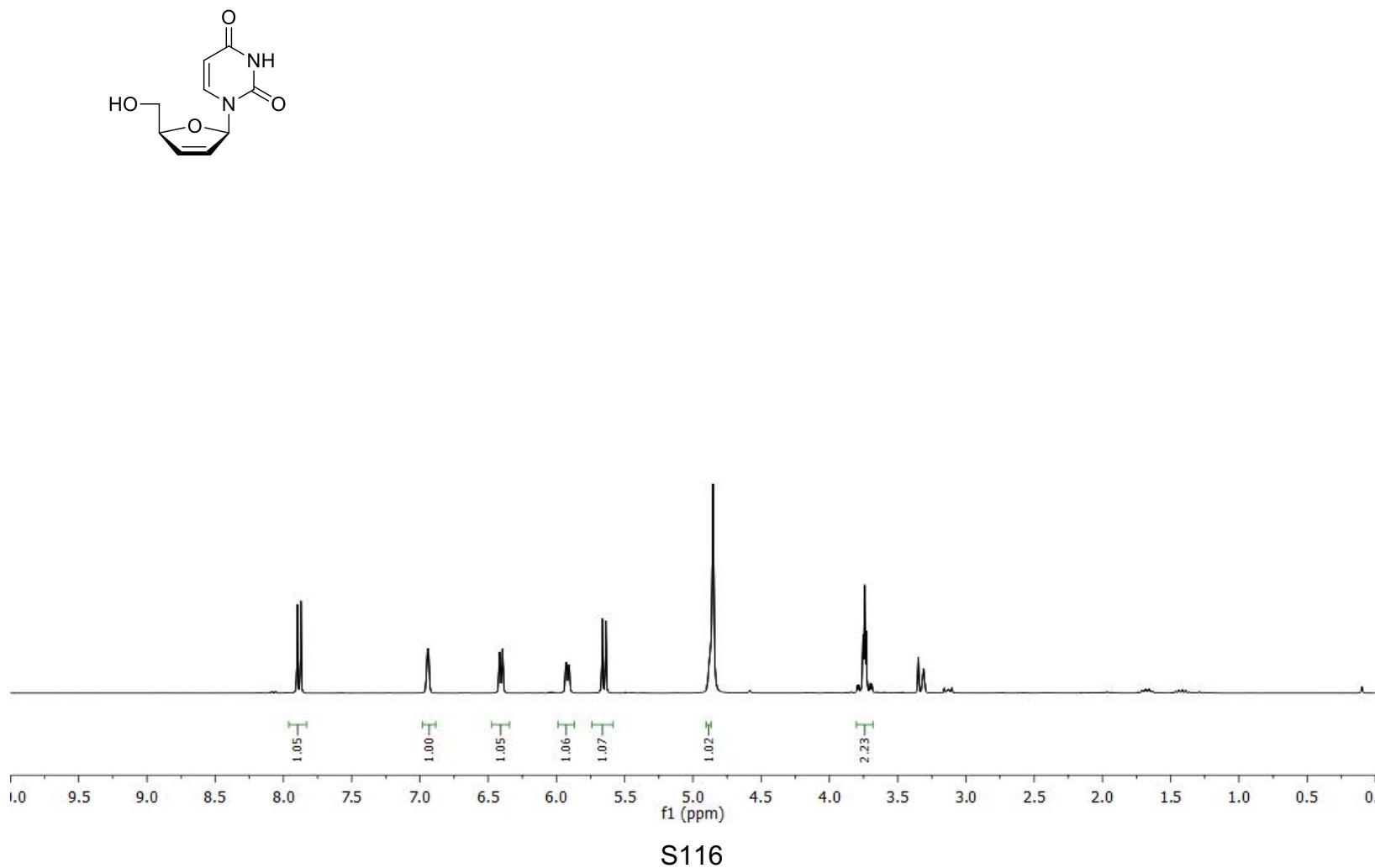
HSQC NMR ( $\text{CDCl}_3$ )



**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-uridine (8a)**

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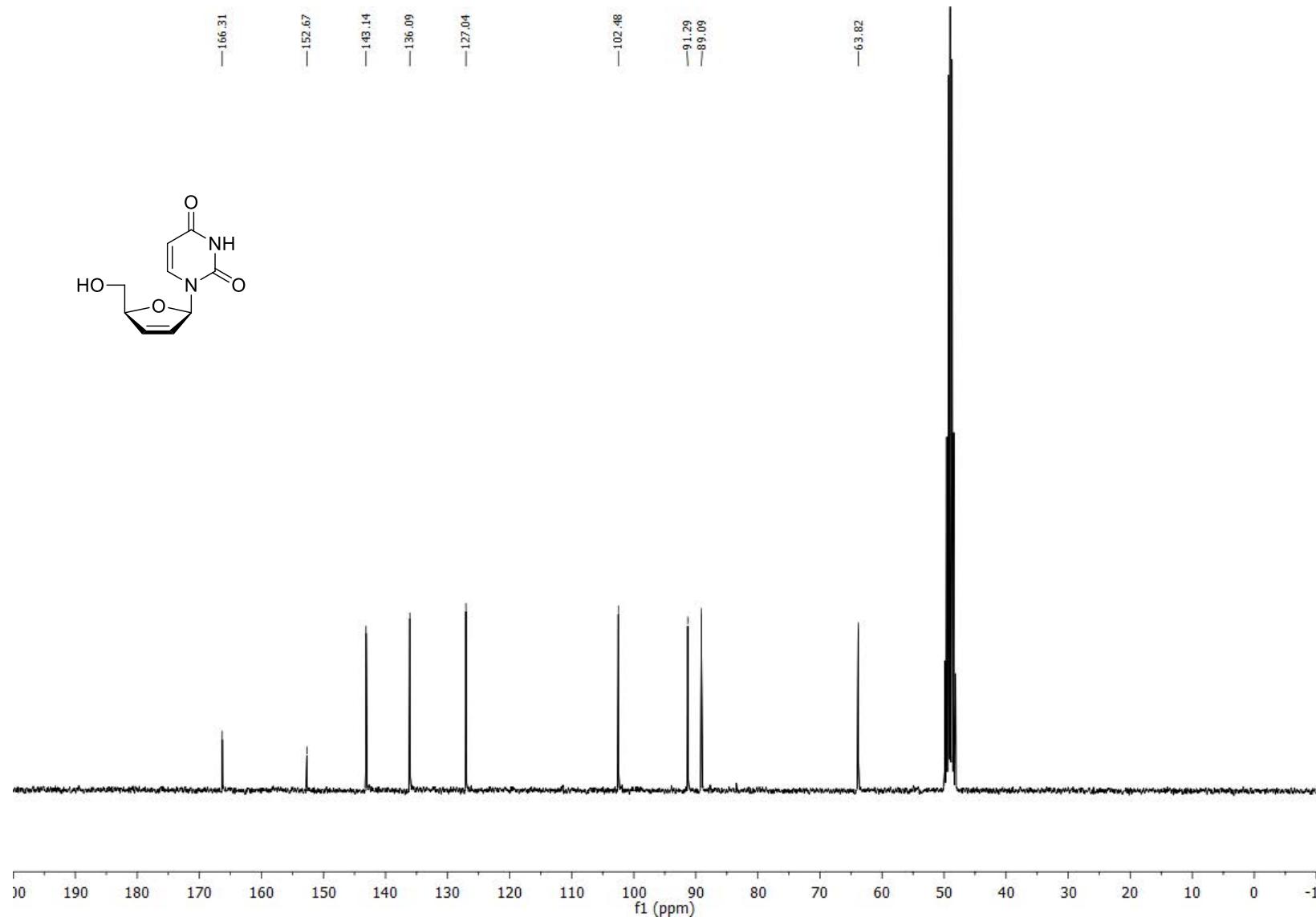
$^1\text{H}$ -NMR (300.13 MHz, MeOH- $d_4$ )



**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-uridine (8a)**

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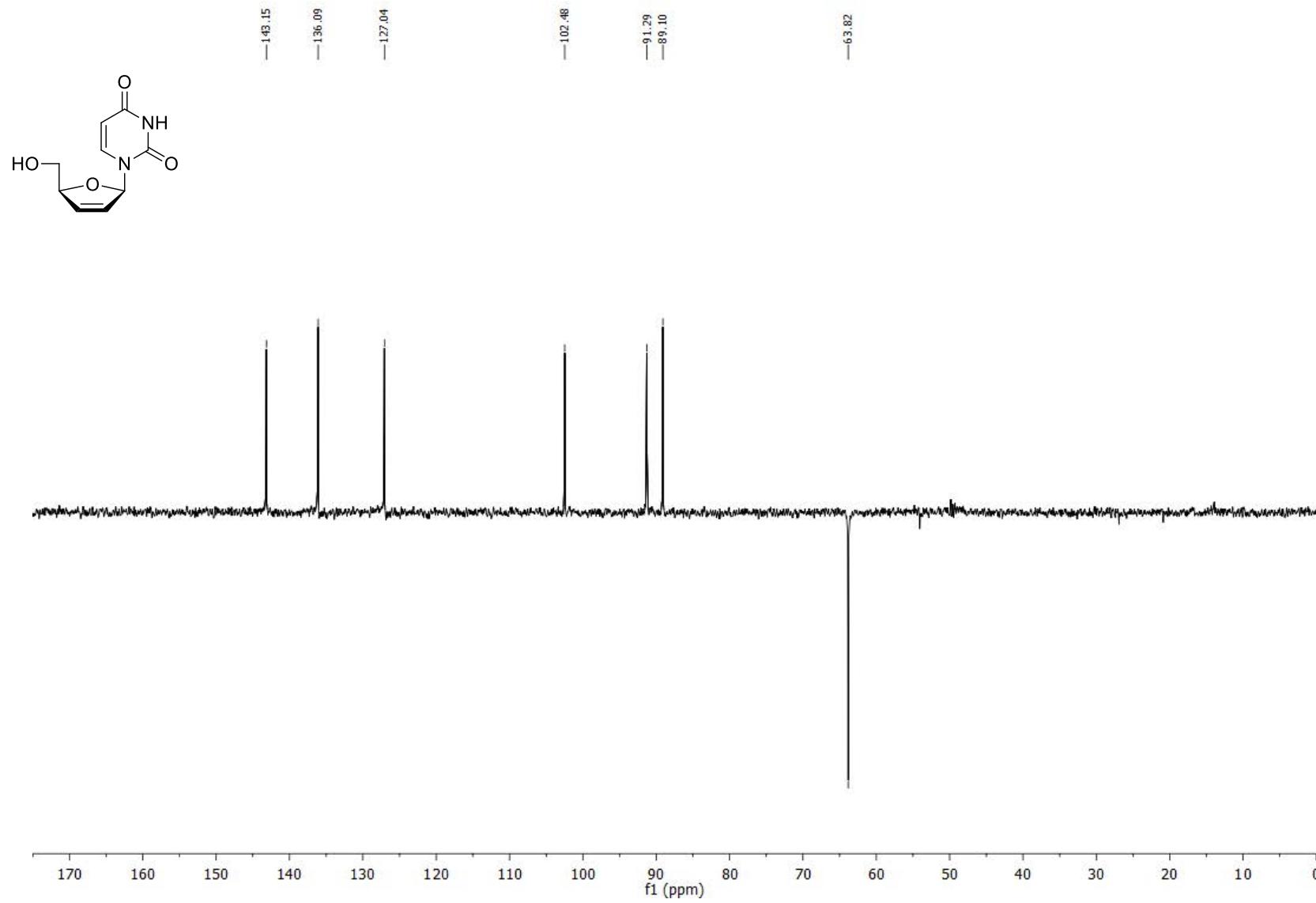
$^{13}\text{C}$ -NMR (75.5 MHz, MeOH- $d_4$ )



**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-uridine (8a)**

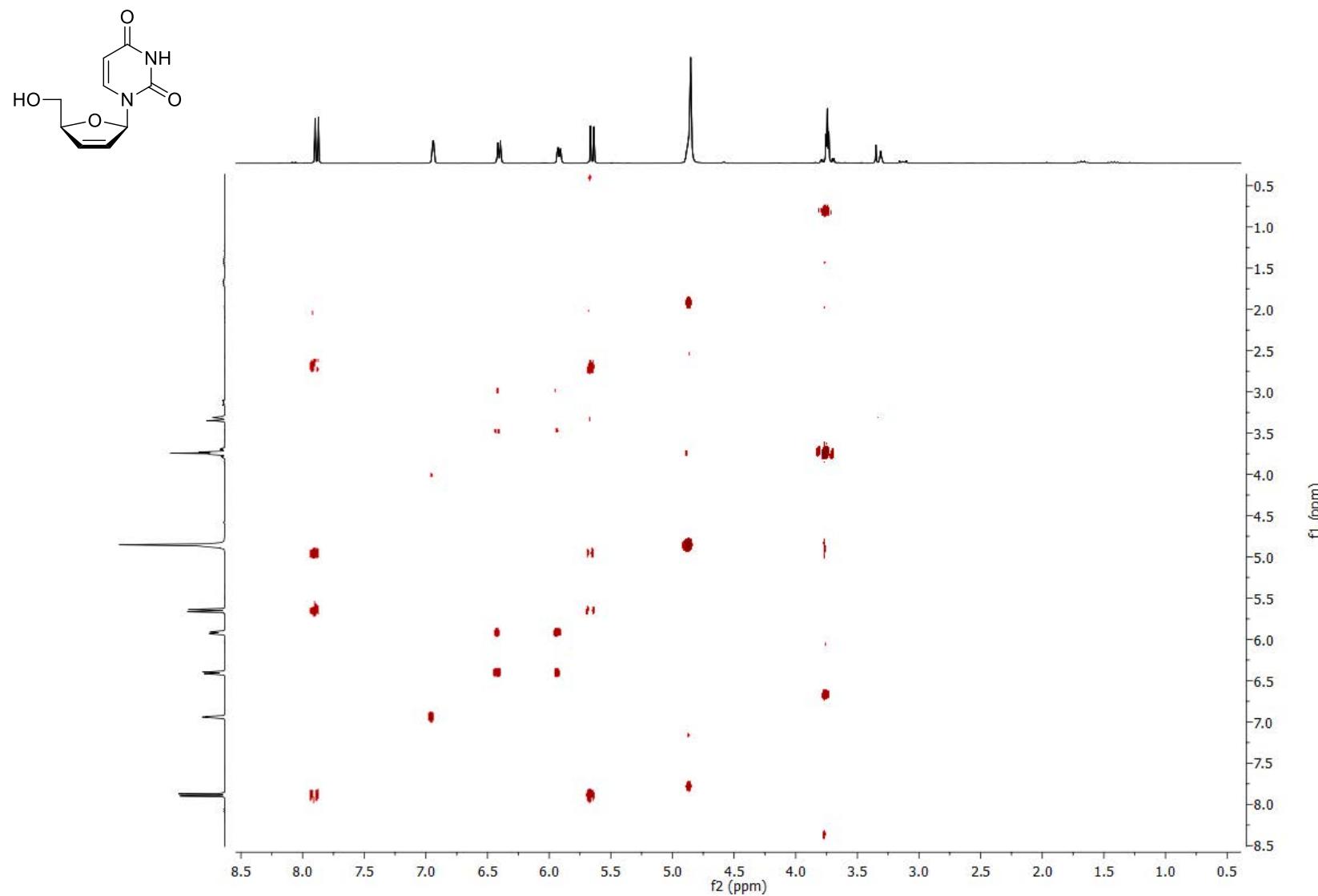
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DEPT NMR (75.5 MHz, MeOH-*d*<sub>4</sub>)



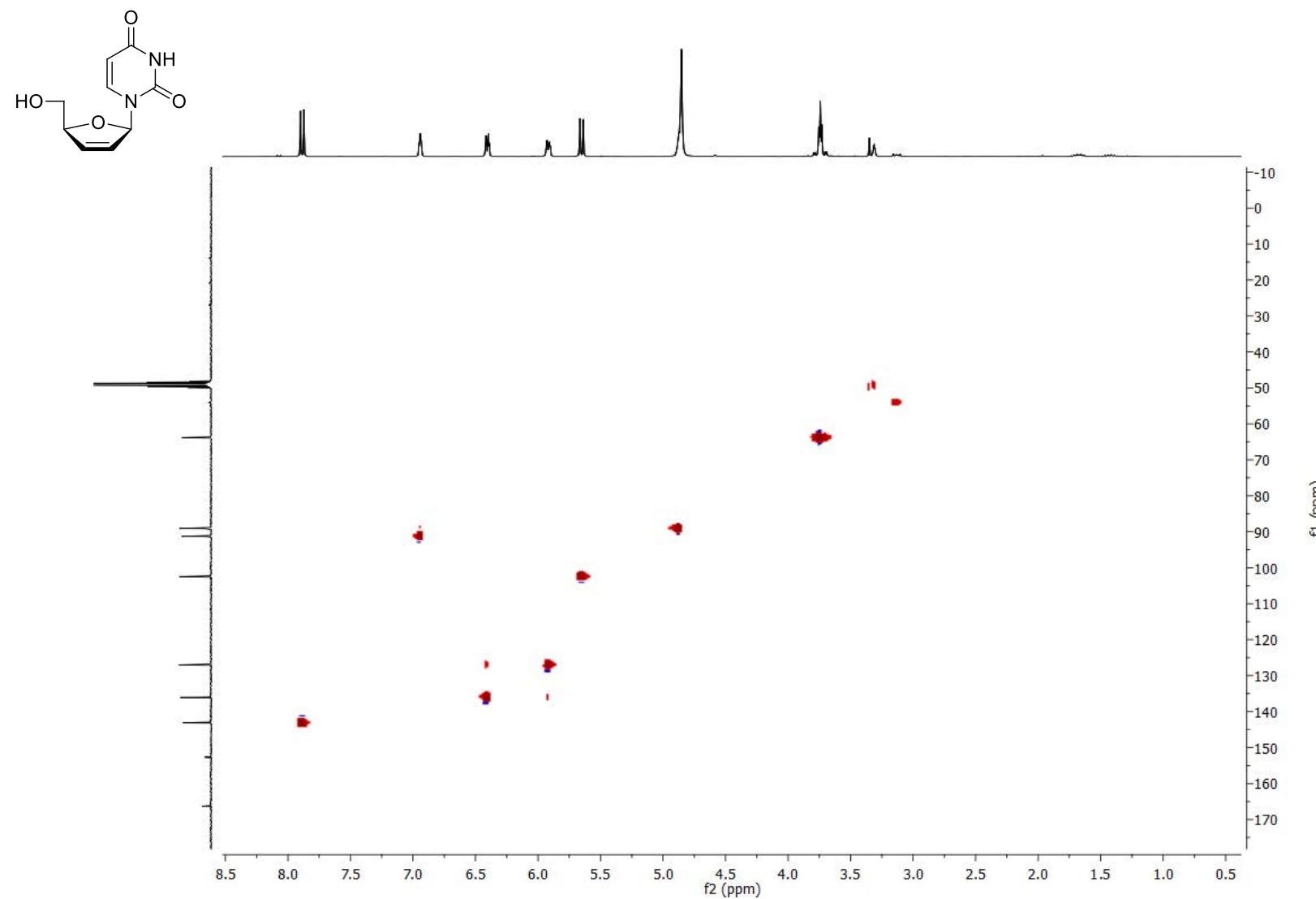
**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-uridine (8a)**

COSY NMR (MeOH-*d*<sub>4</sub>)



**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-uridine (8a)**

HSQC NMR ( $\text{MeOH}-d_4$ )

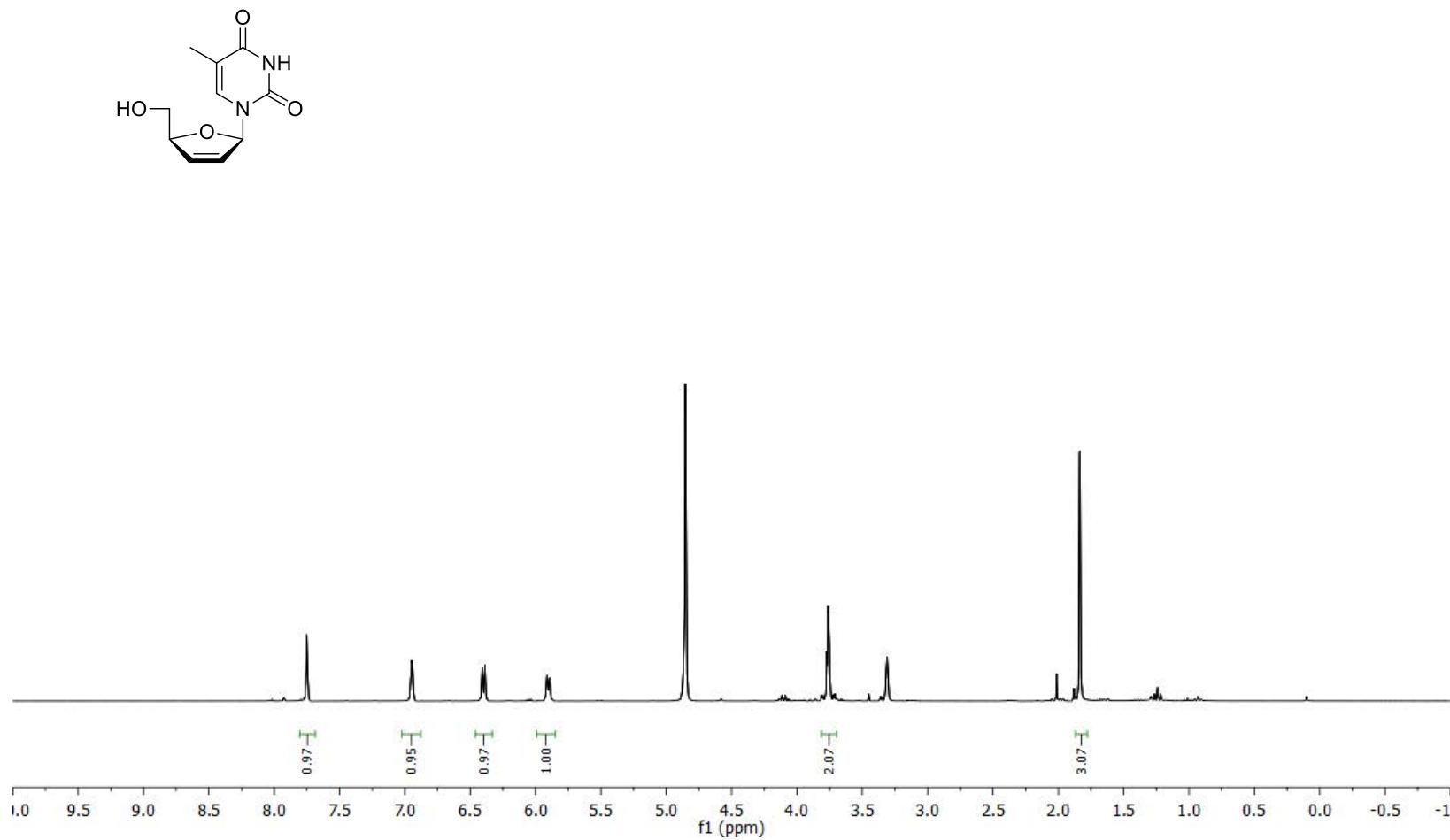


S120

**2',3'-Didehydro-3'-deoxy- $\beta$ -D-5-thymidine (8b)**

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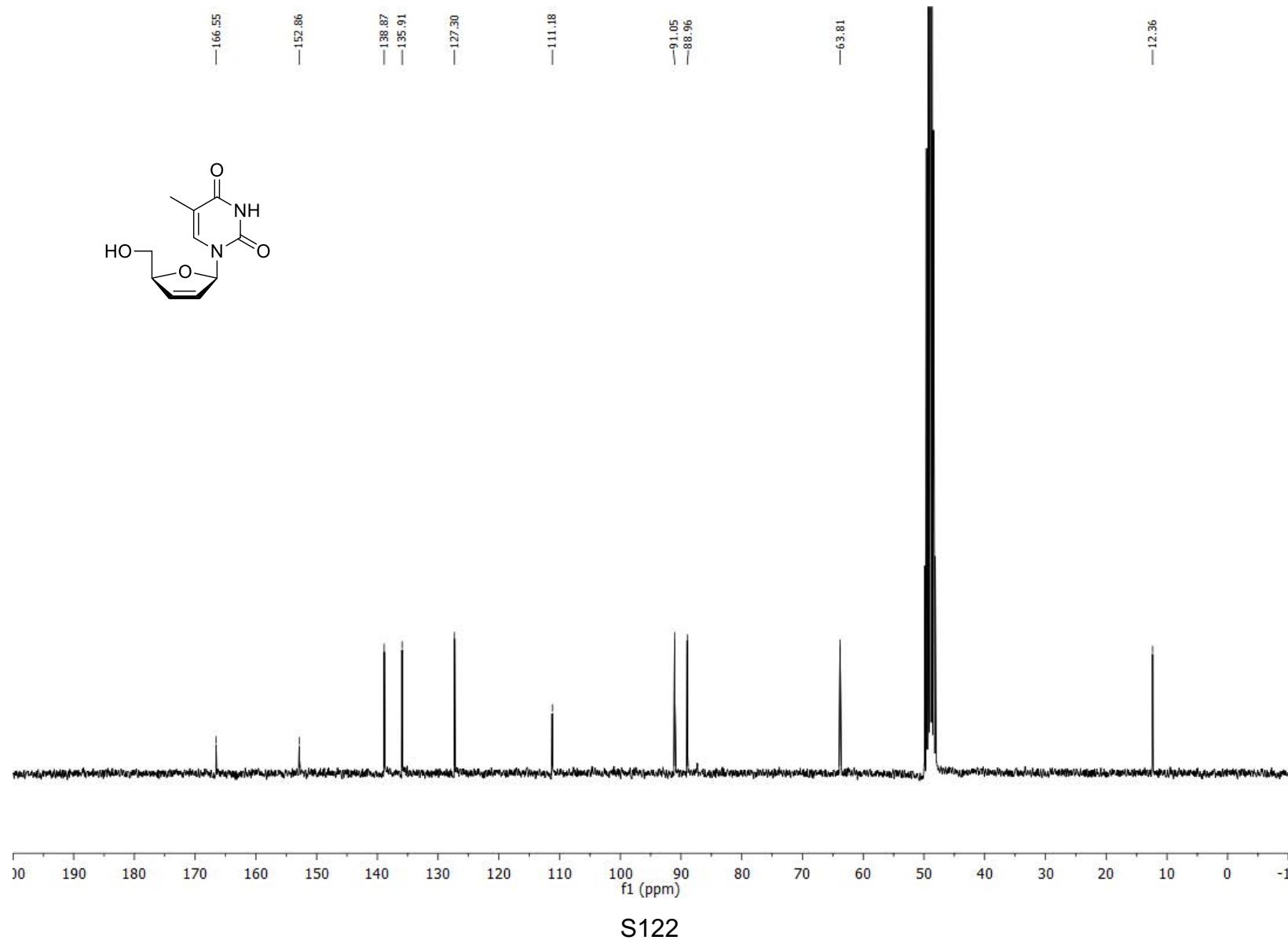
$^1\text{H}$ -NMR (300.13 MHz, MeOH- $d_4$ )



S121

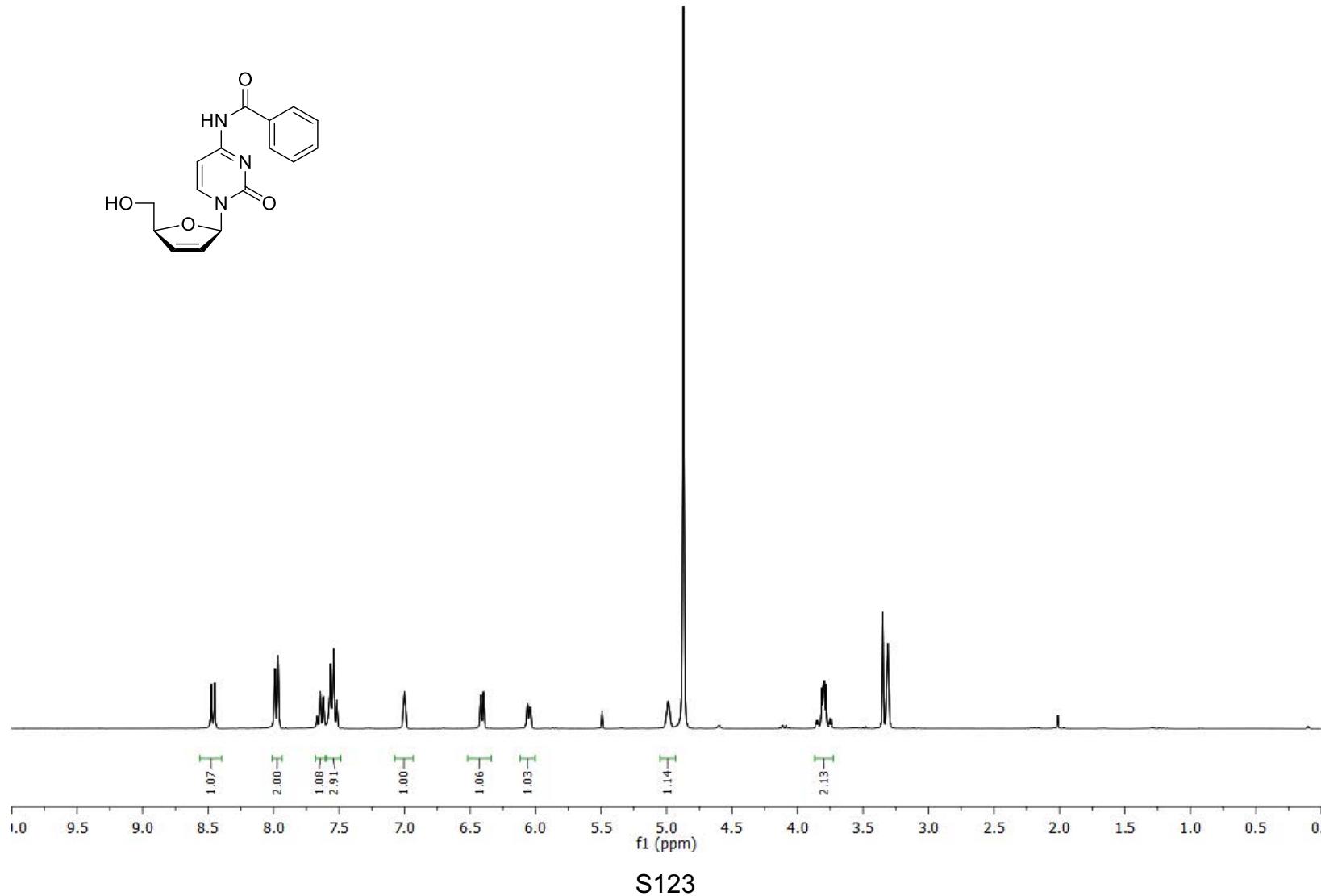
**2',3'-Didehydro-3'-deoxy- $\beta$ -D-5-thymidine (8b)**

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



*N*<sup>4</sup>-Benzoyl-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (8d)

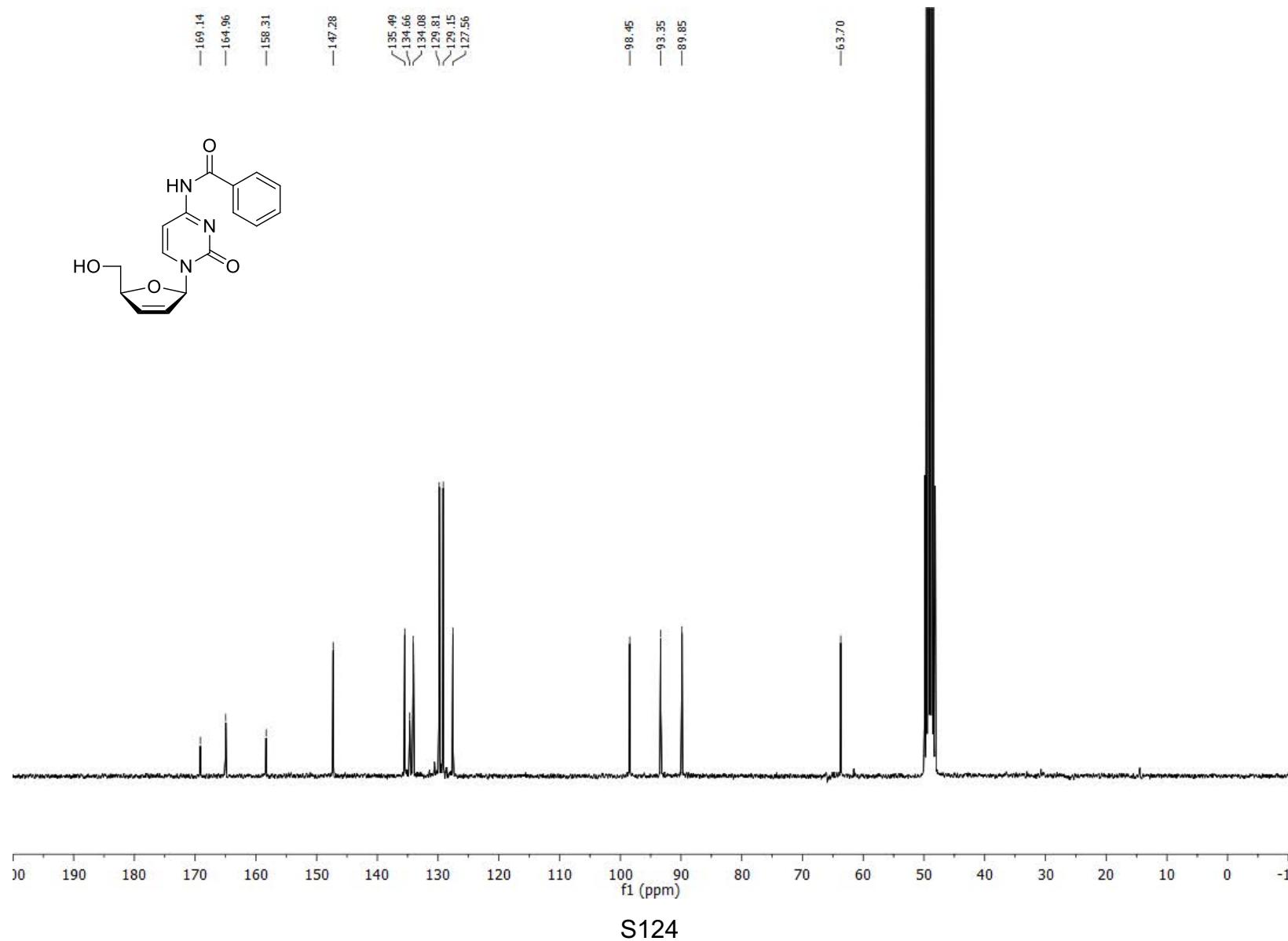
<sup>1</sup>H-NMR (300.13 MHz, MeOH-*d*<sub>4</sub>)



S123

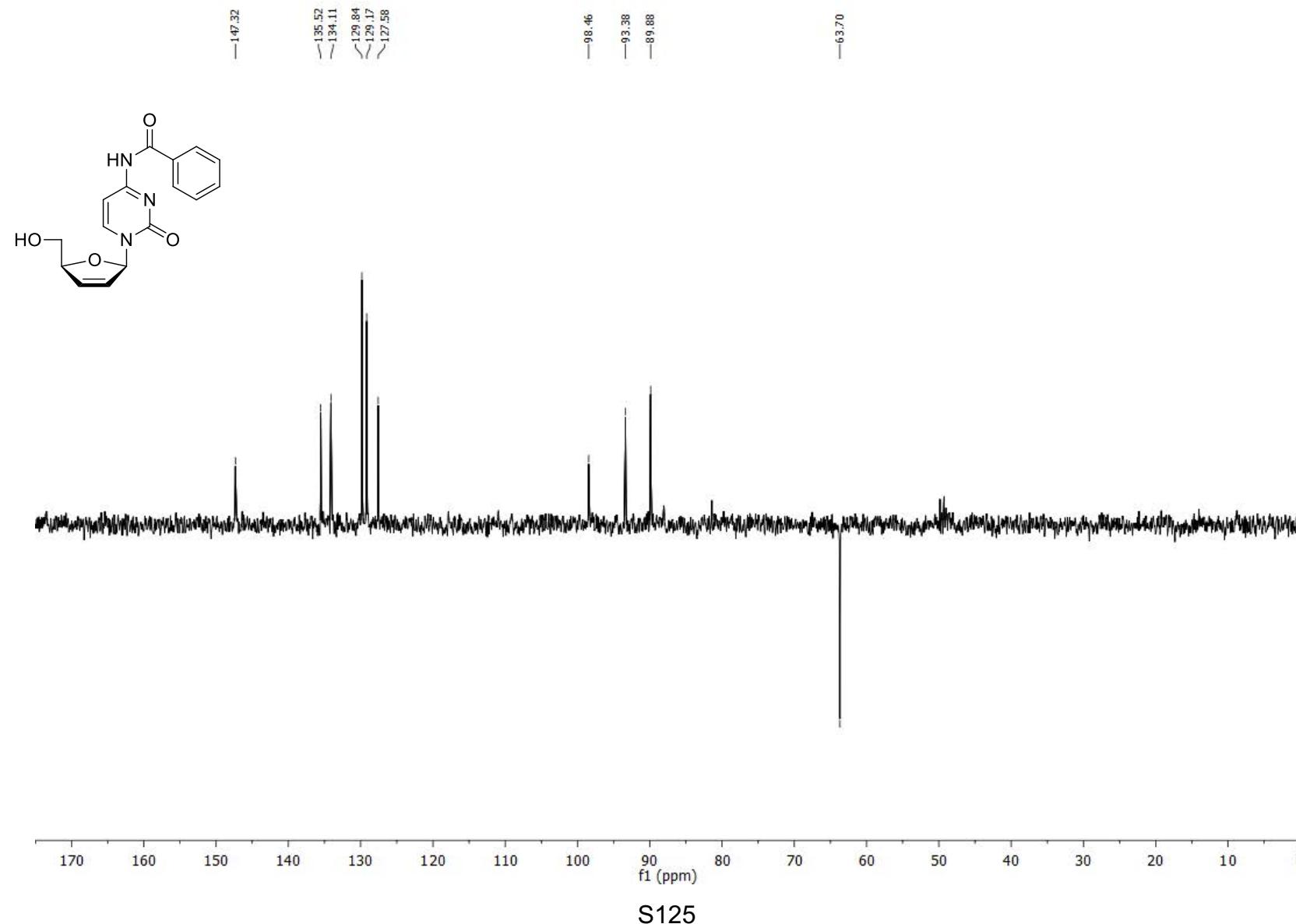
***N*<sup>4</sup>-Benzoyl-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (8d)**

<sup>13</sup>C-NMR (75.5 MHz, MeOH-*d*<sub>4</sub>)



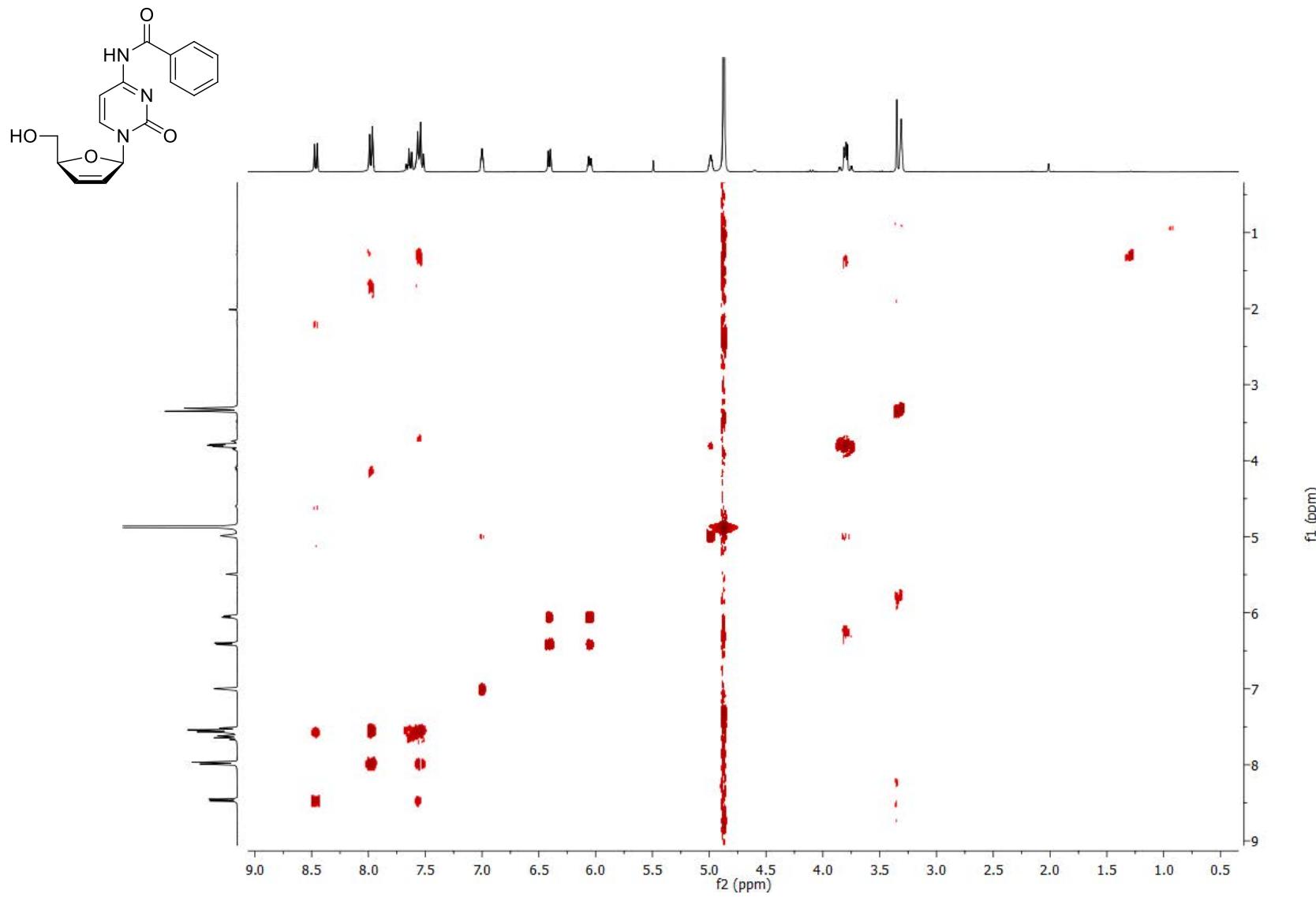
***N*<sup>4</sup>-Benzoyl-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (8d)**

DEPT NMR (75.5 MHz, MeOH-*d*<sub>4</sub>)



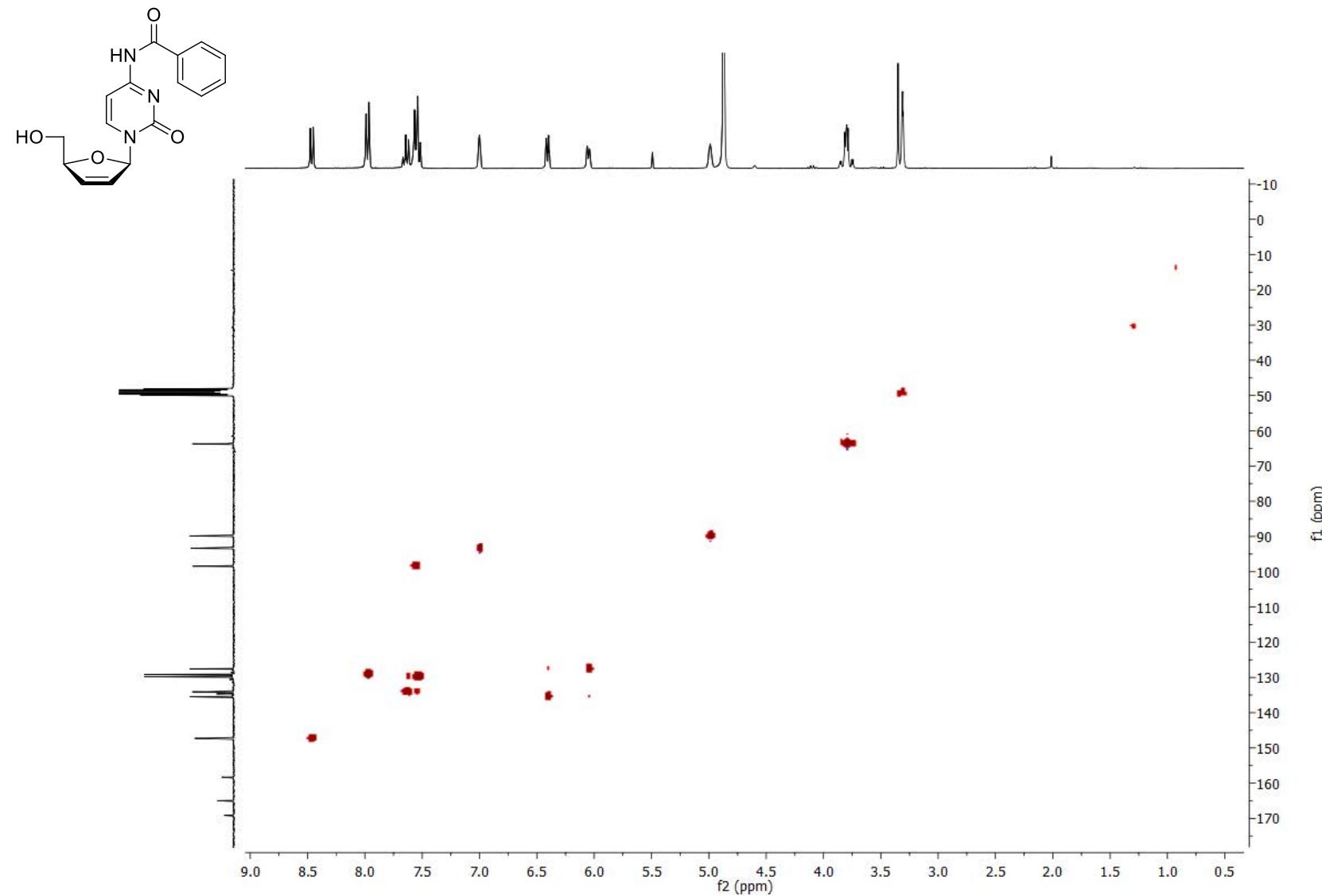
*N*<sup>4</sup>-Benzoyl-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (8d)

COSY NMR (MeOH-*d*<sub>4</sub>)



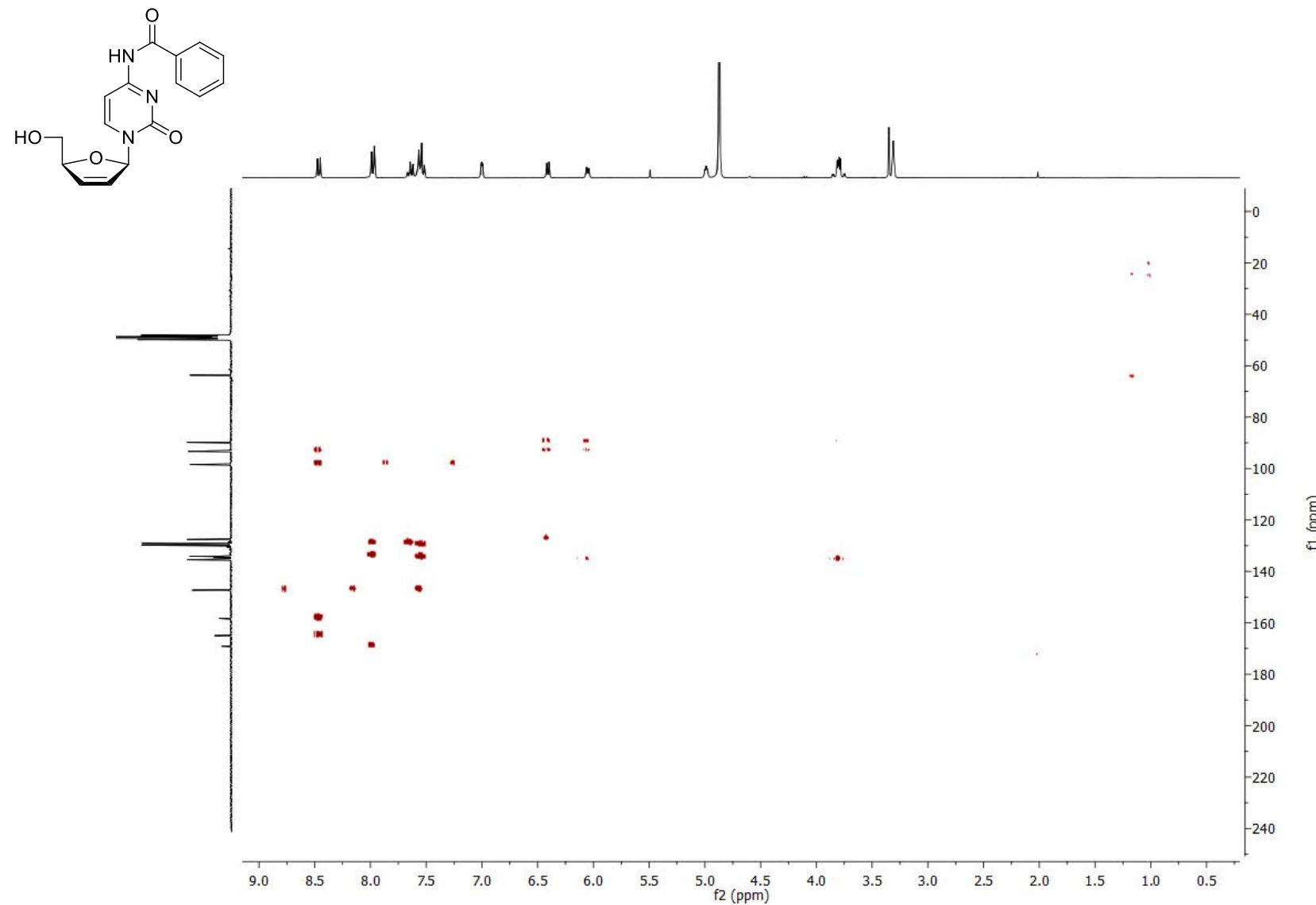
*N*<sup>4</sup>-Benzoyl-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (8d)

HSQC NMR (MeOH-*d*<sub>4</sub>)



*N*<sup>4</sup>-Benzoyl-2',3'-didehydro-2',3'-dideoxy- $\beta$ -D-cytidine (8d)

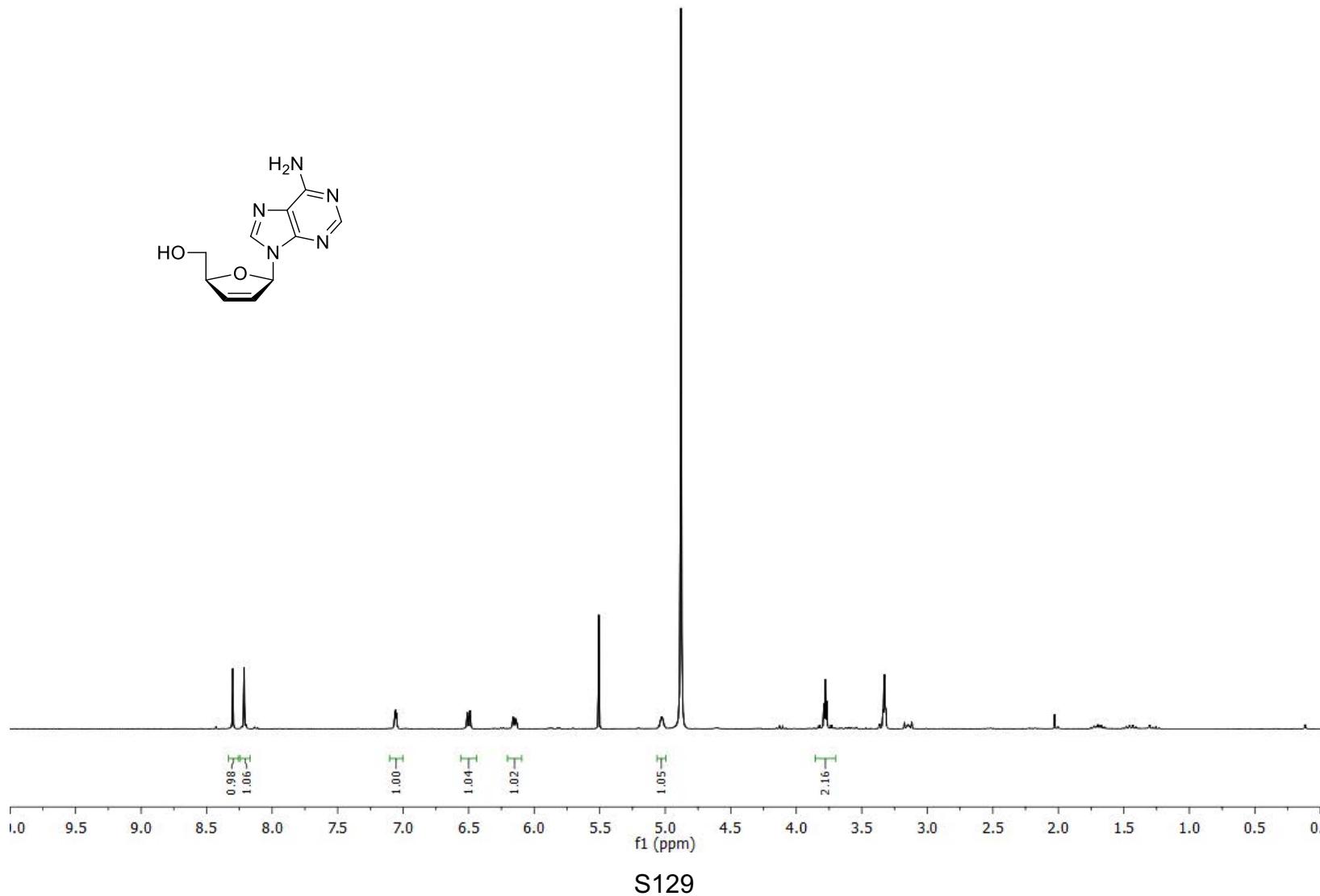
HMBC NMR (MeOH-*d*<sub>4</sub>)



**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-adenosine (8e)**

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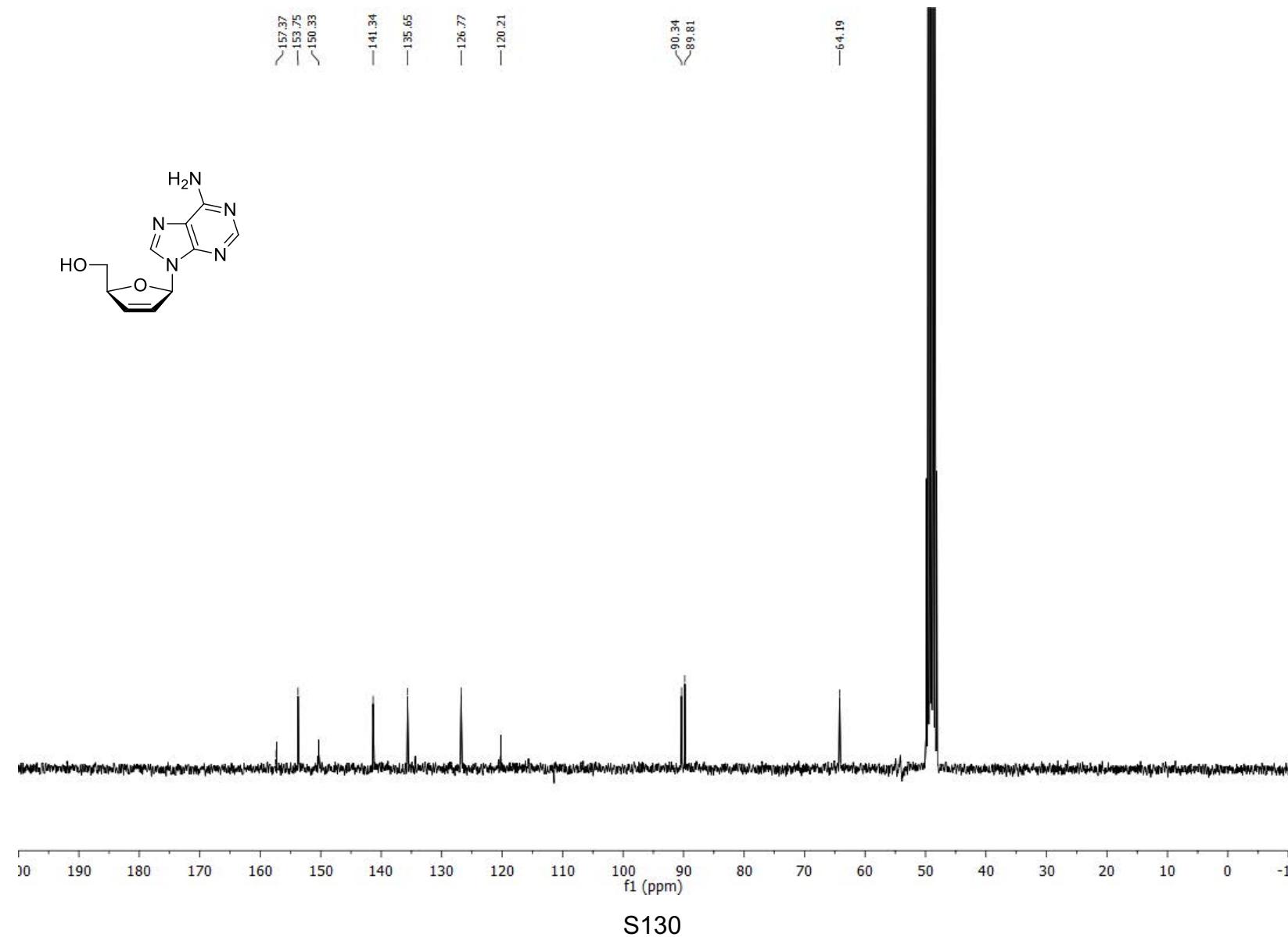
$^1\text{H}$ -NMR (300.13 MHz, MeOH- $d_4$ )



**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-adenosine (8e)**

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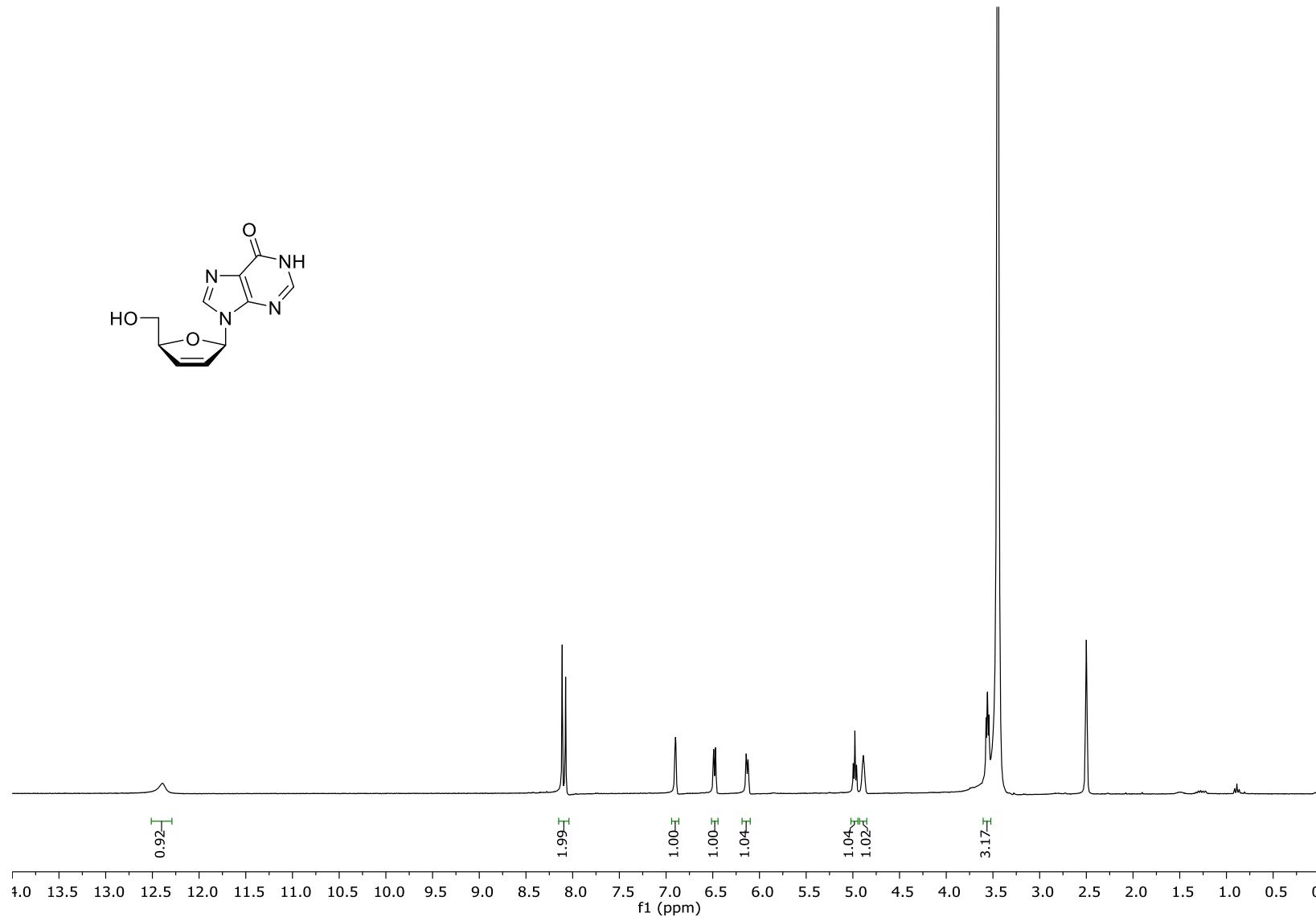
$^{13}\text{C}$ -NMR (75.5 MHz, MeOH- $d_4$ )



**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-inosine (8f)**

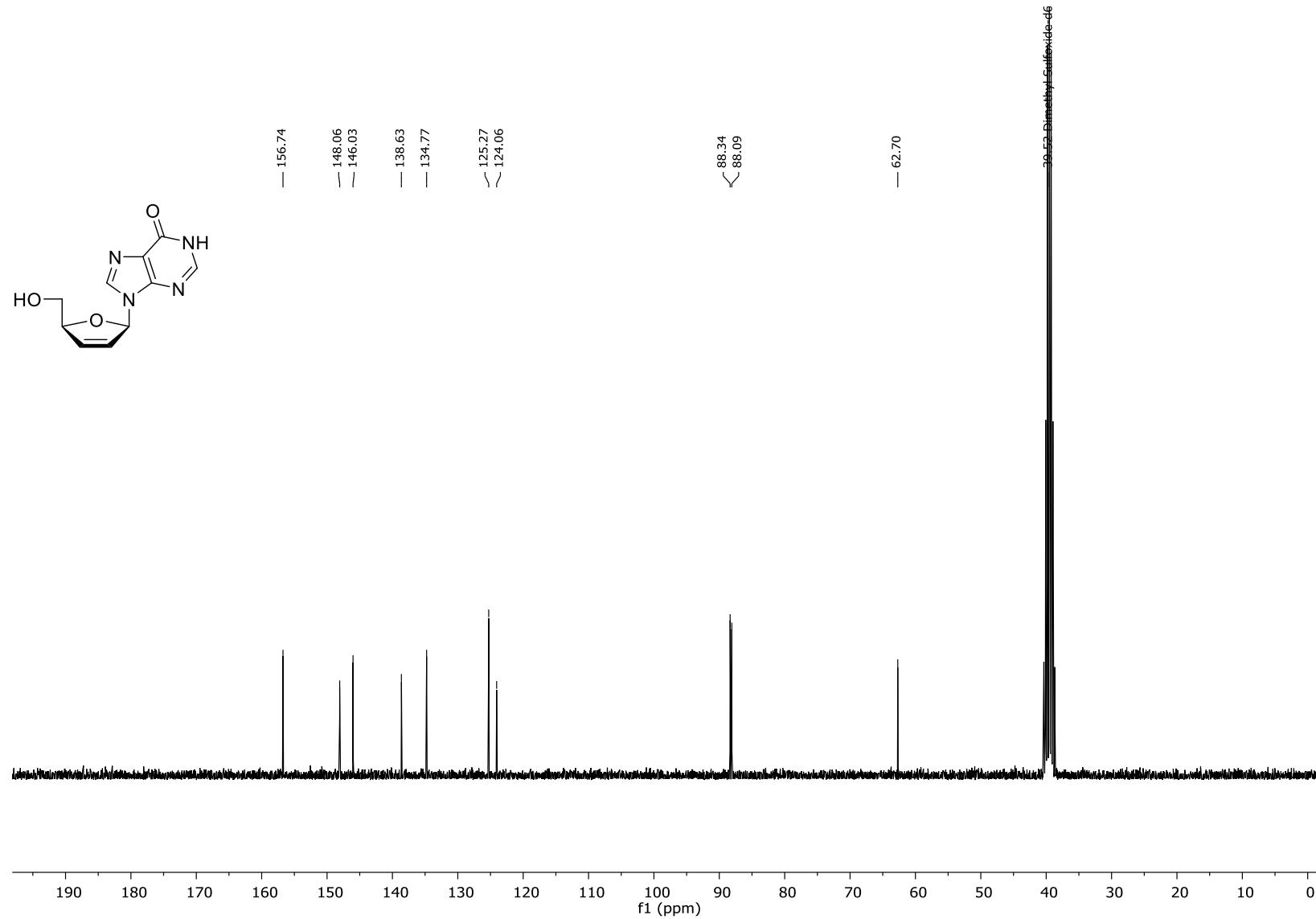
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$^1\text{H}$ -NMR (300.13 MHz, DMSO- $d_6$ )



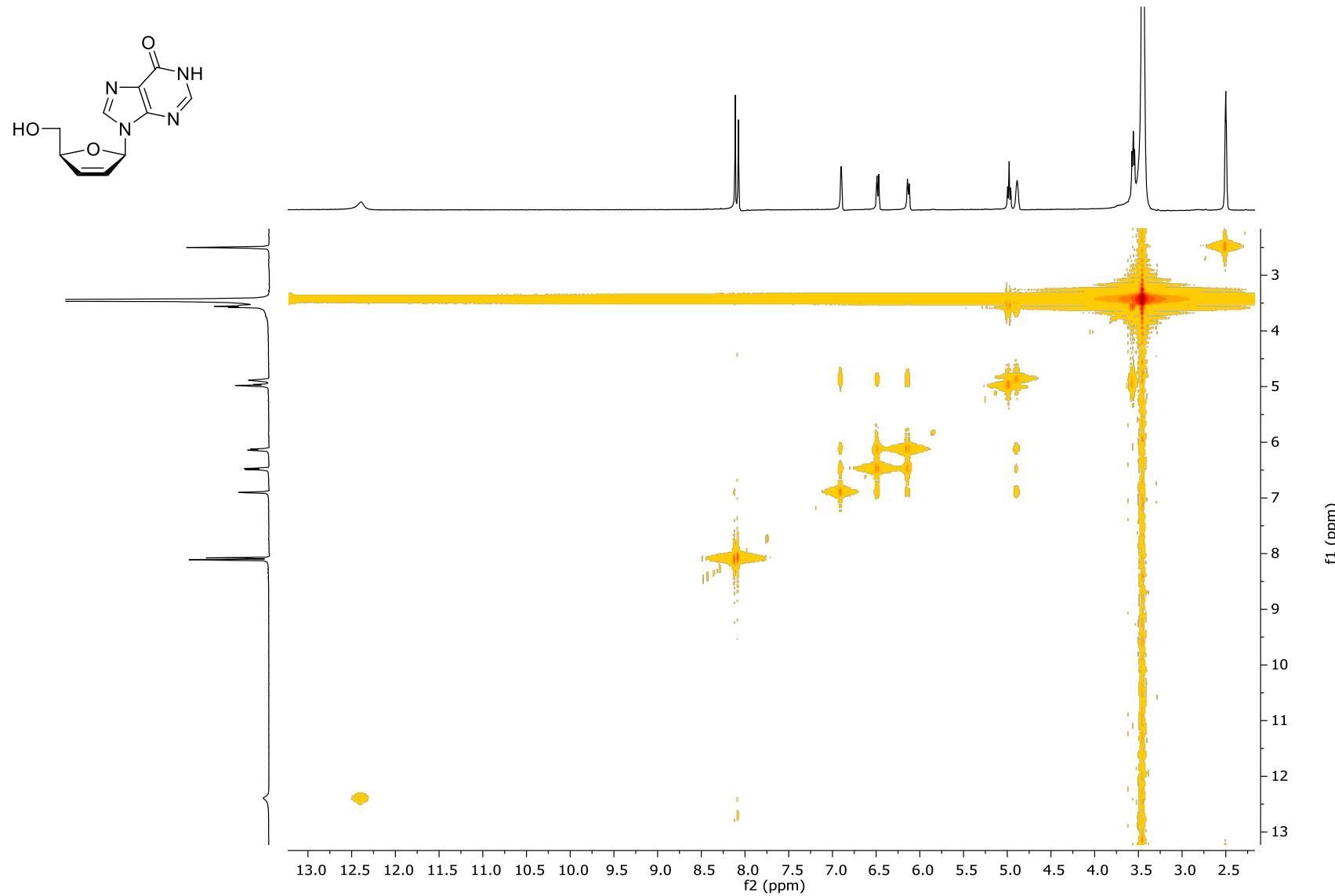
**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-inosine (8f)**

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



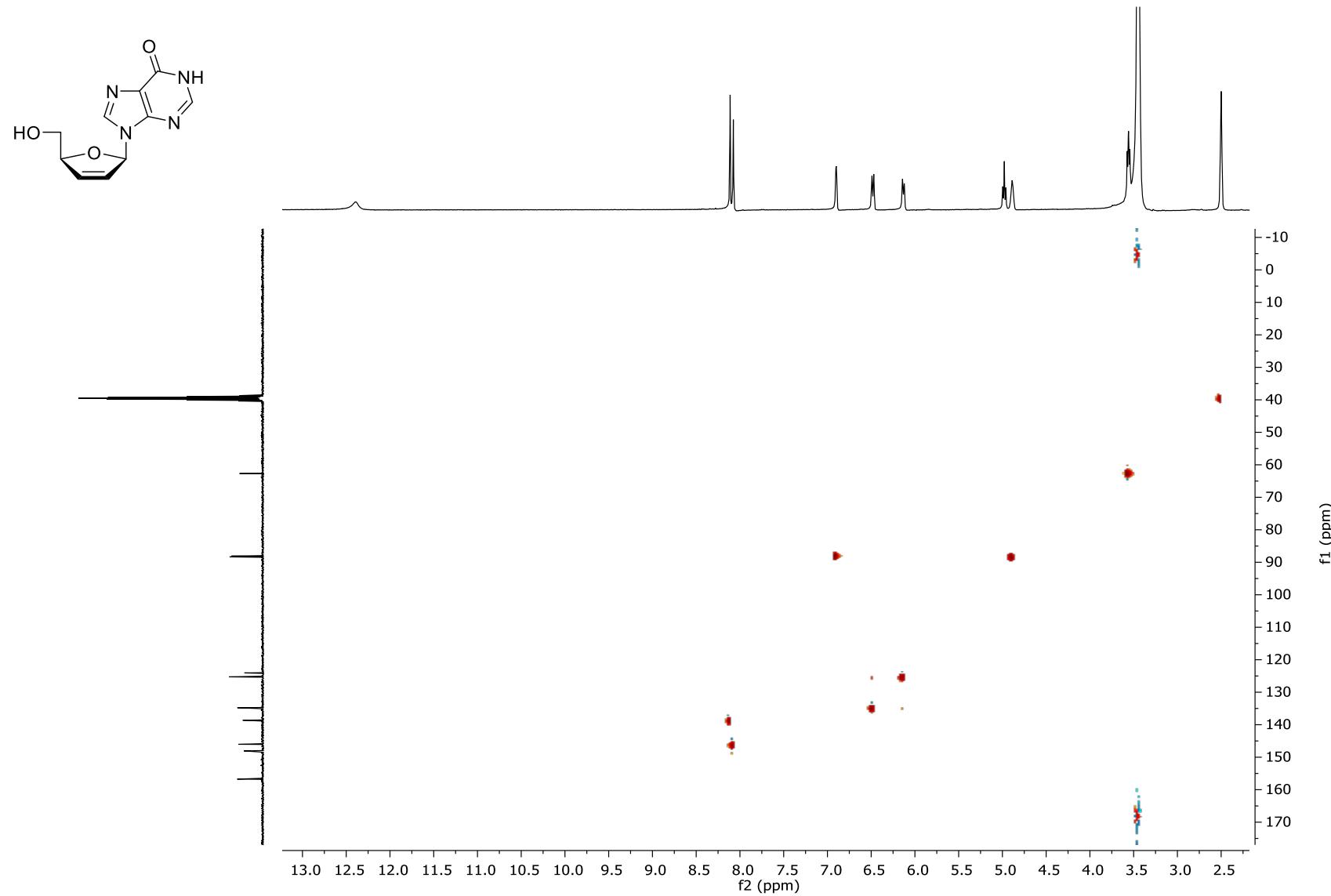
**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-inosine (8f)**

COSY NMR (DMSO- $d_6$ )



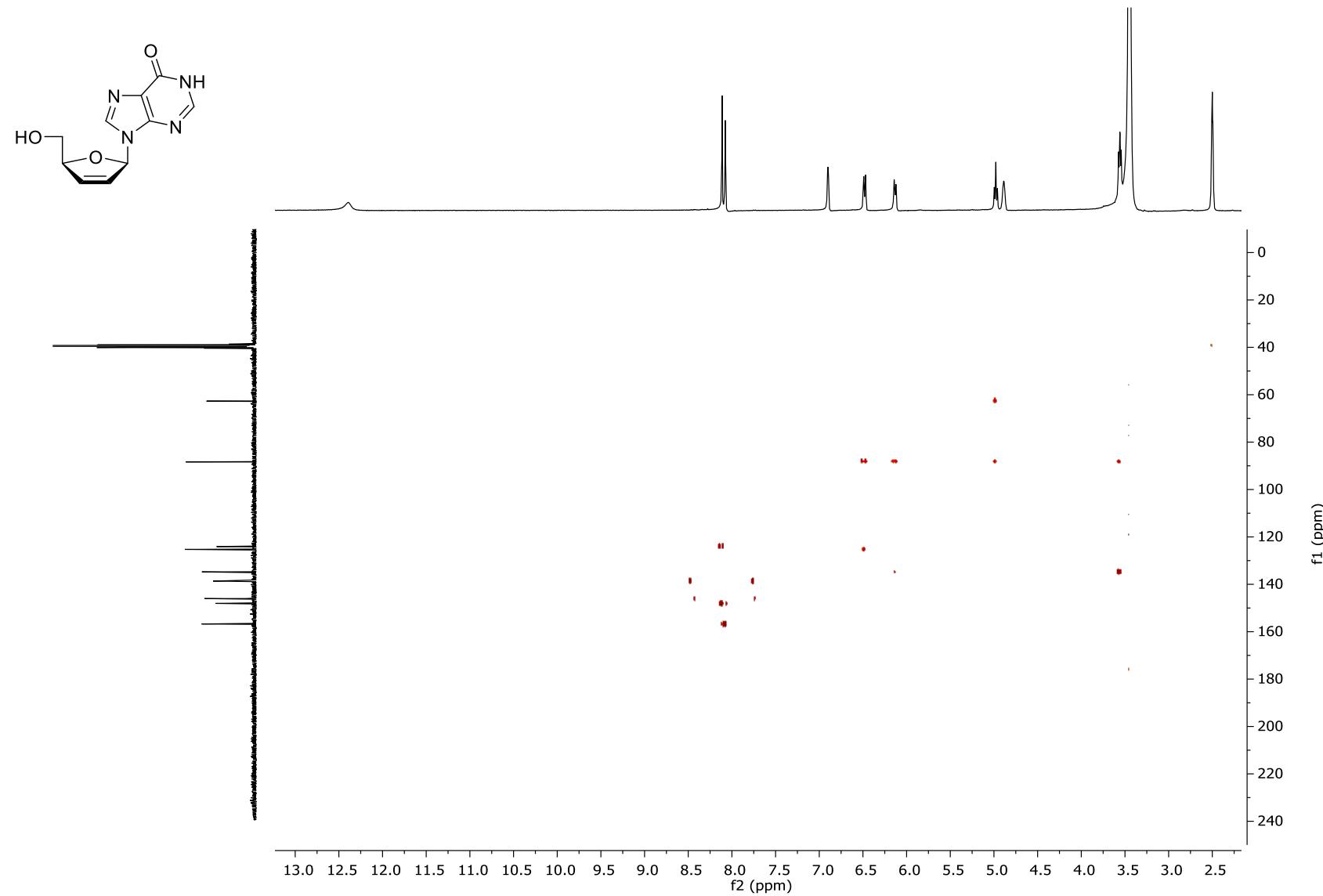
**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-inosine (8f)**

HSQC NMR (DMSO- $d_6$ )



**2',3'-Didehydro-2',3'-dideoxy- $\beta$ -D-inosine (8f)**

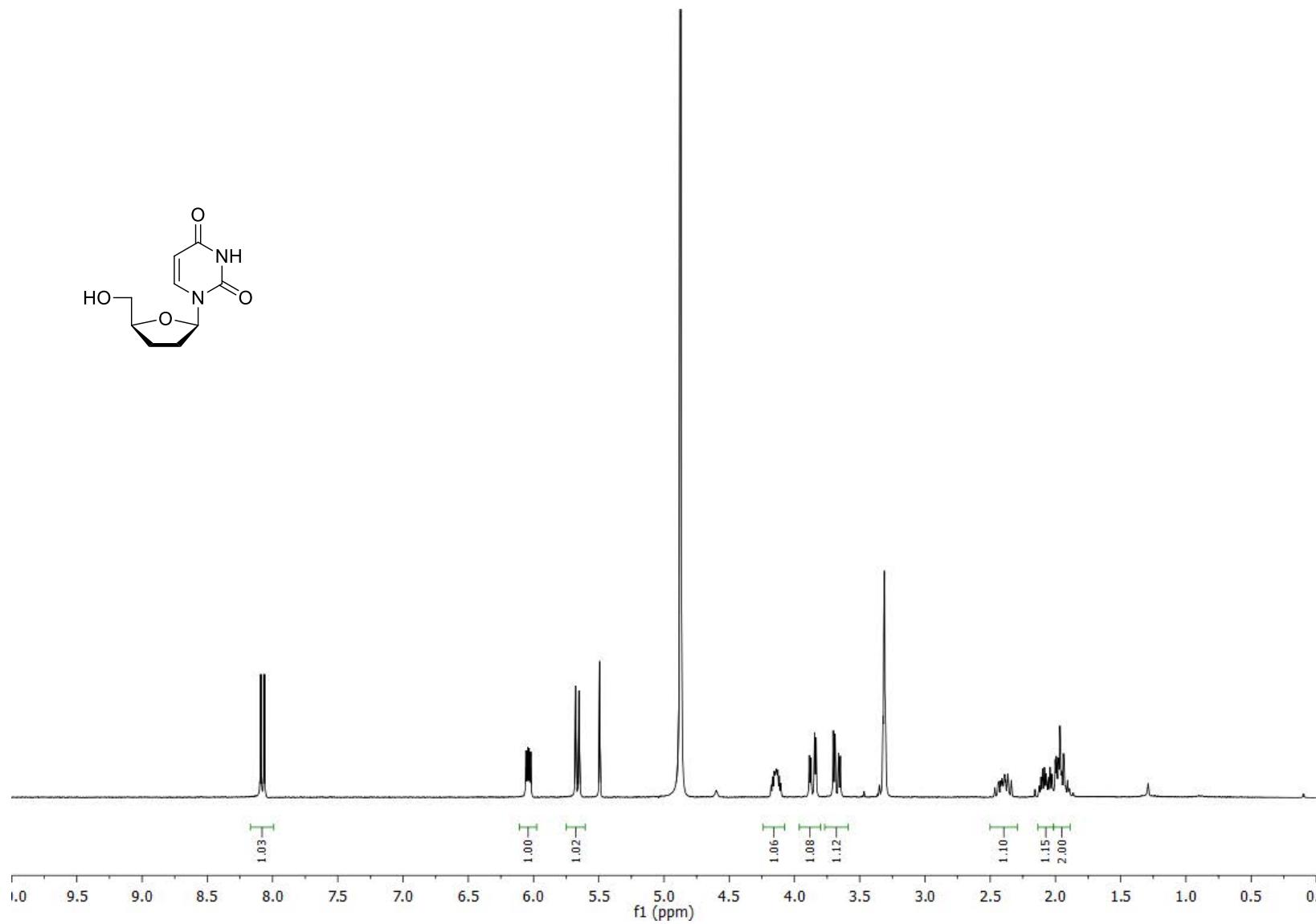
HMBC NMR (DMSO-*d*<sub>6</sub>)



**2',3'-Dideoxy- $\beta$ -D-uridine (9a)**

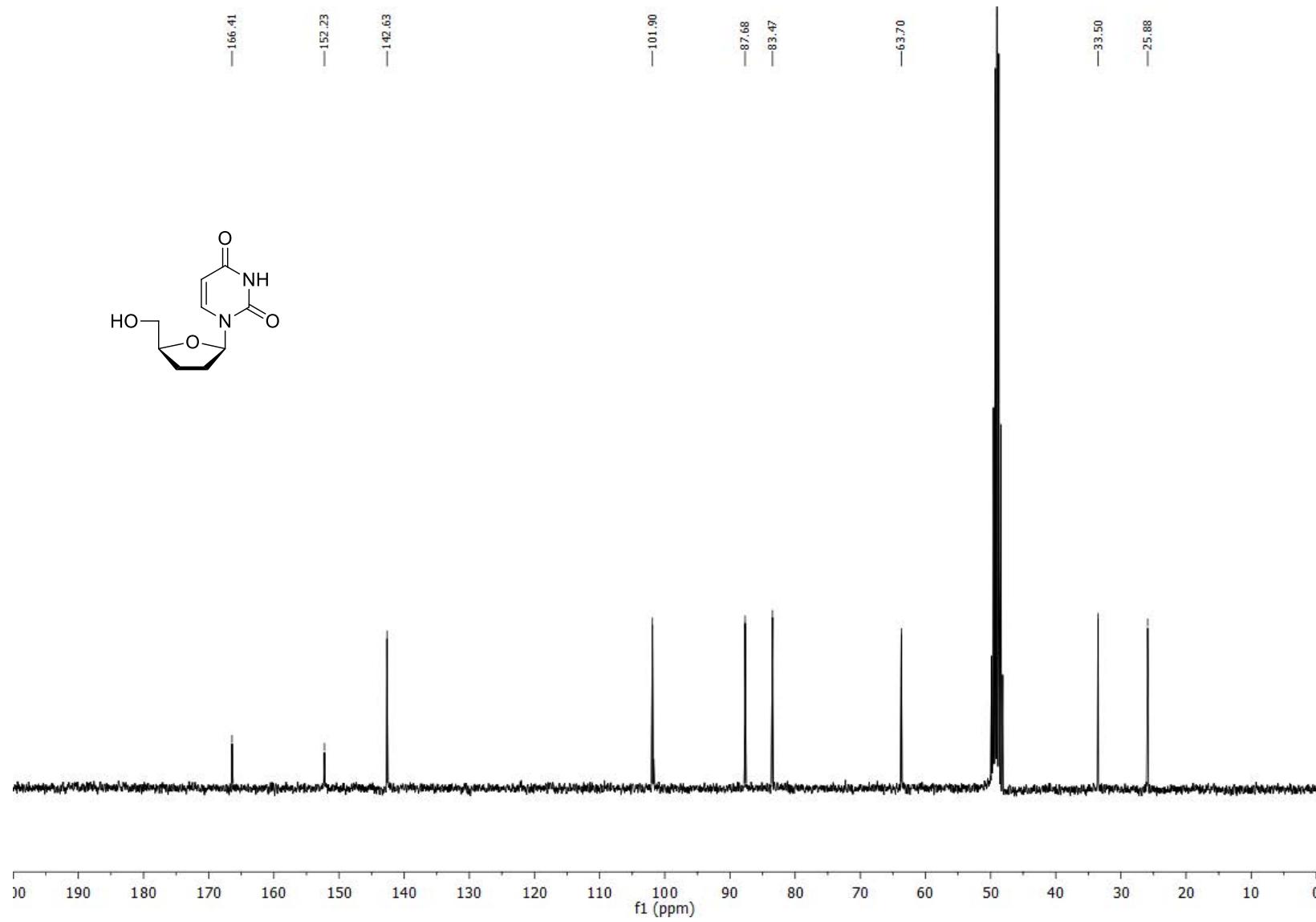
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$^1\text{H-NMR}$  (300.13 MHz, MeOH- $d_4$ )



**2',3'-Dideoxy- $\beta$ -D-uridine (9a)**

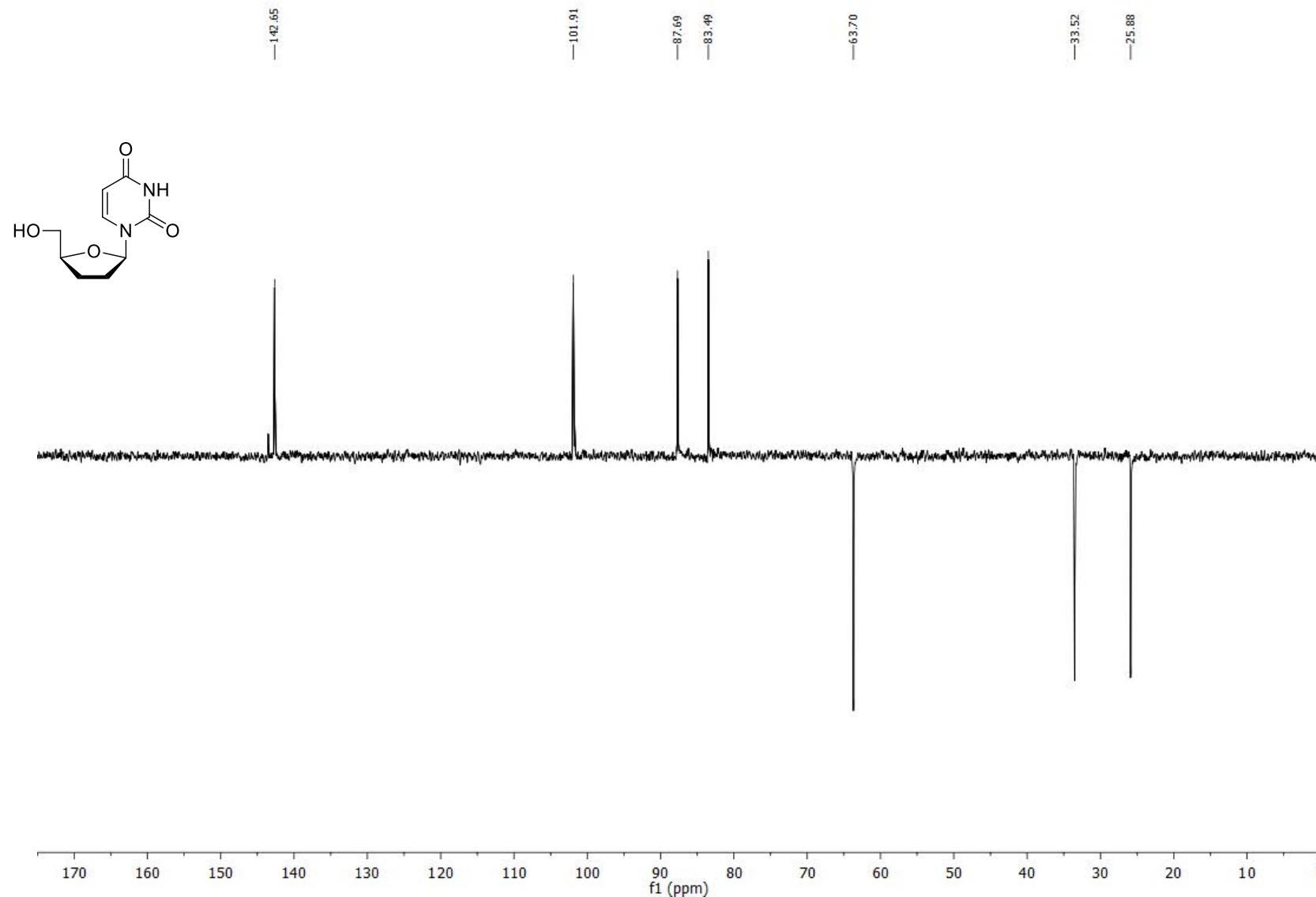
$^{13}\text{C}$ -NMR (75.5 MHz, MeOH- $d_4$ )



**2',3'-Dideoxy- $\beta$ -D-uridine (9a)**

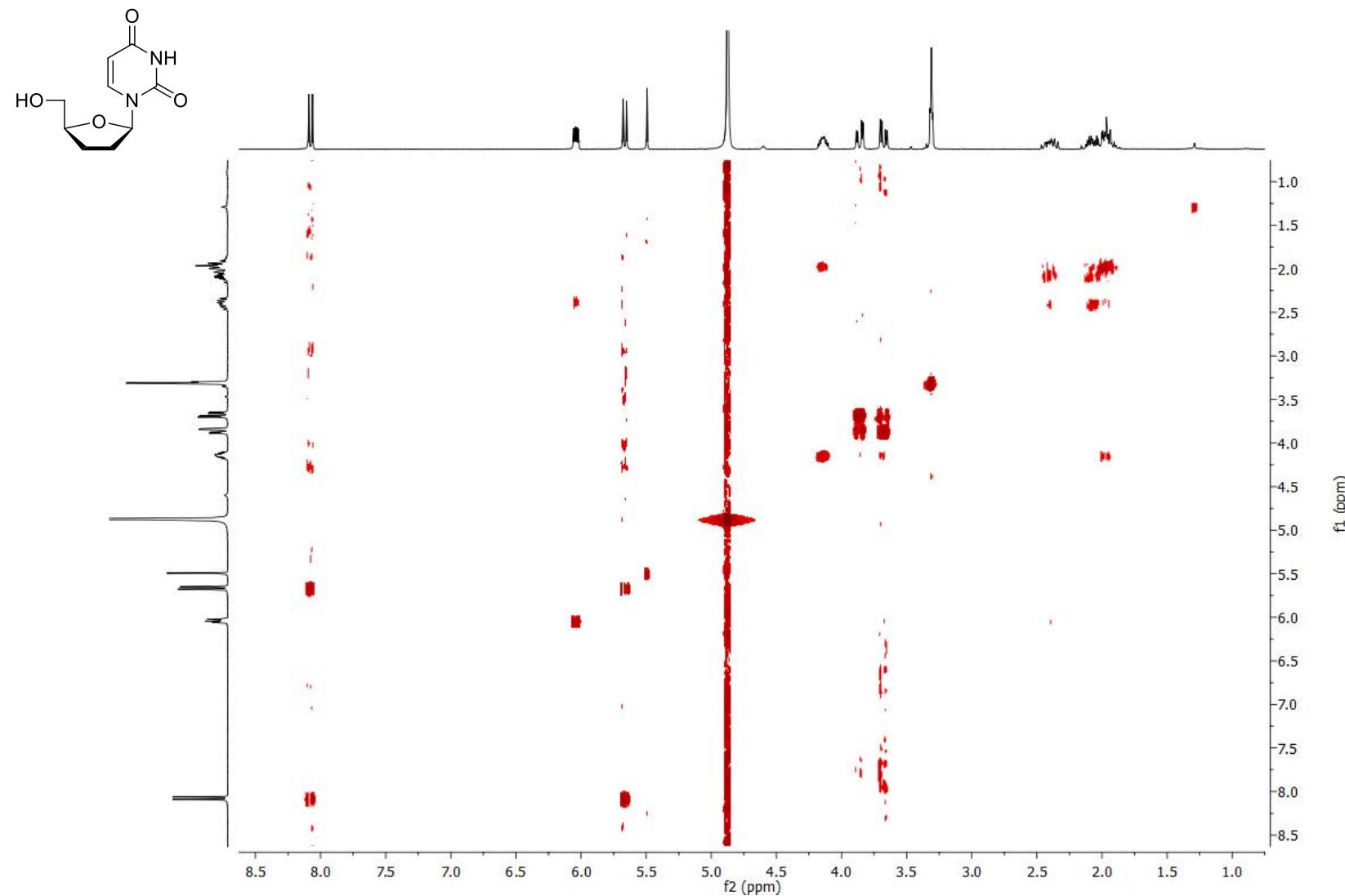
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DEPT NMR (75.5 MHz, MeOH-*d*<sub>4</sub>)



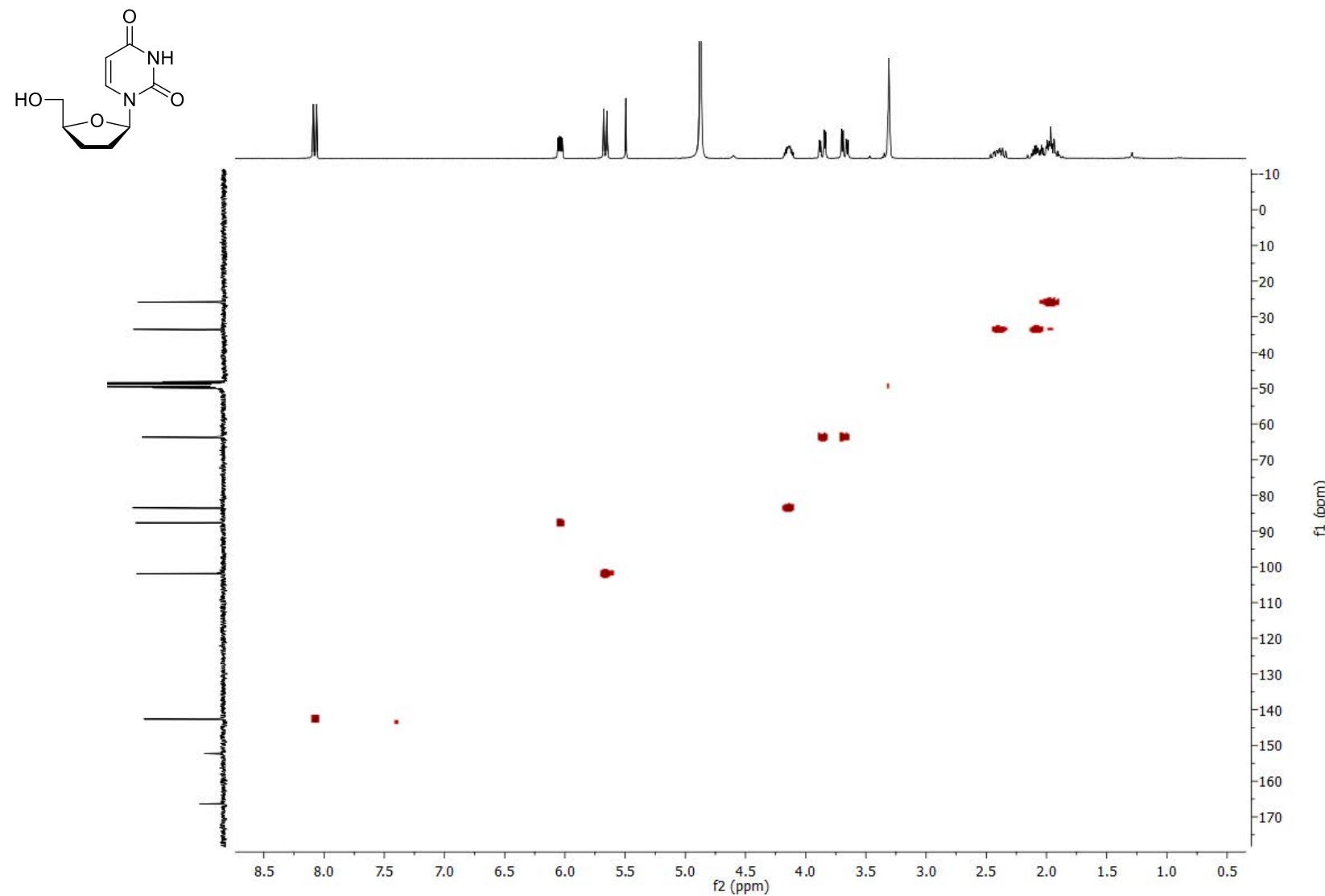
**2',3'-Dideoxy- $\beta$ -D-uridine (9a)**

COSY NMR (MeOH-*d*<sub>4</sub>)



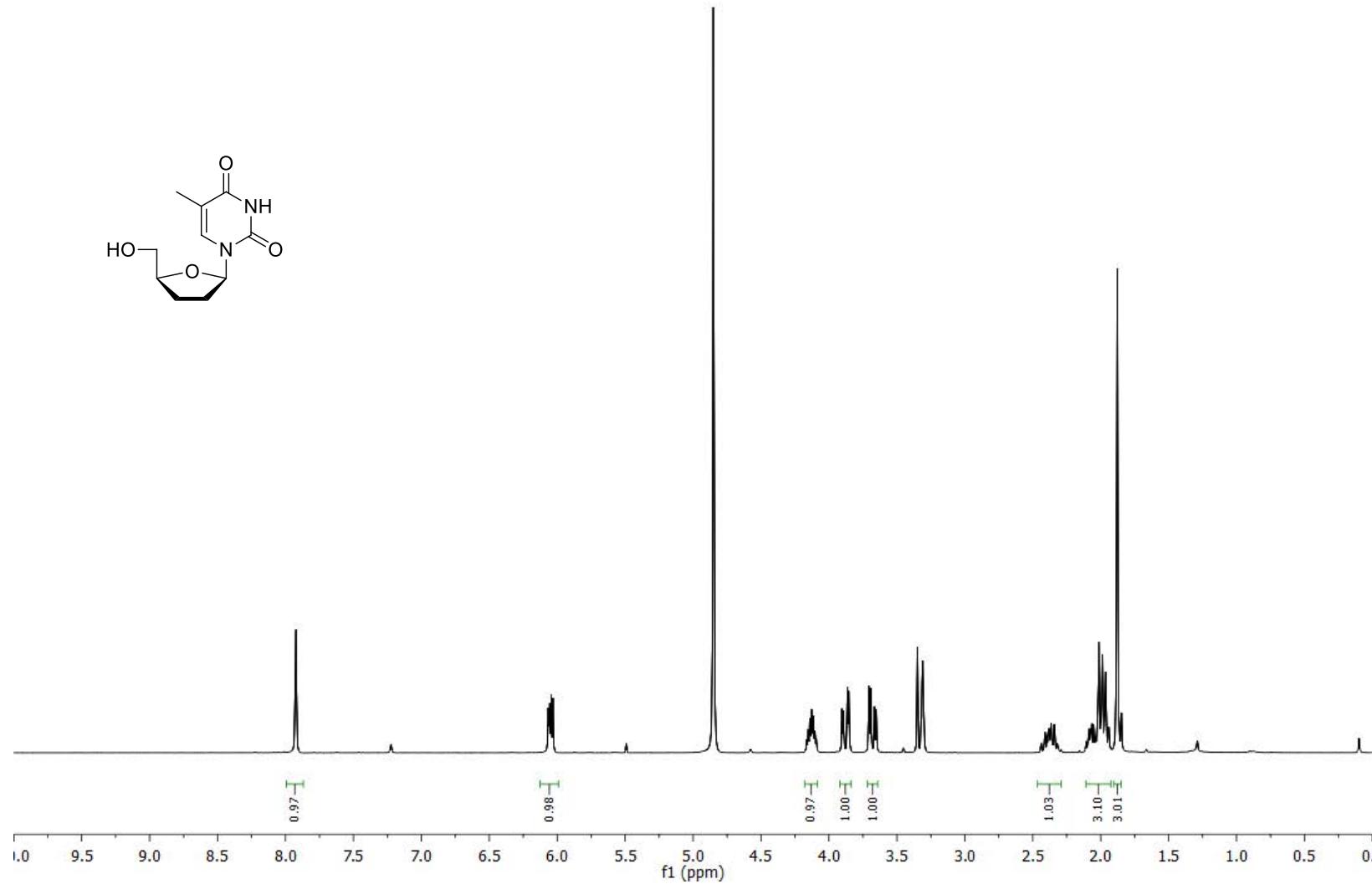
**2',3'-Dideoxy- $\beta$ -D-uridine (9a)**

HSQC NMR ( $\text{MeOH}-d_4$ )



**3'-Deoxy- $\beta$ -D-5-thymidine (9b)**

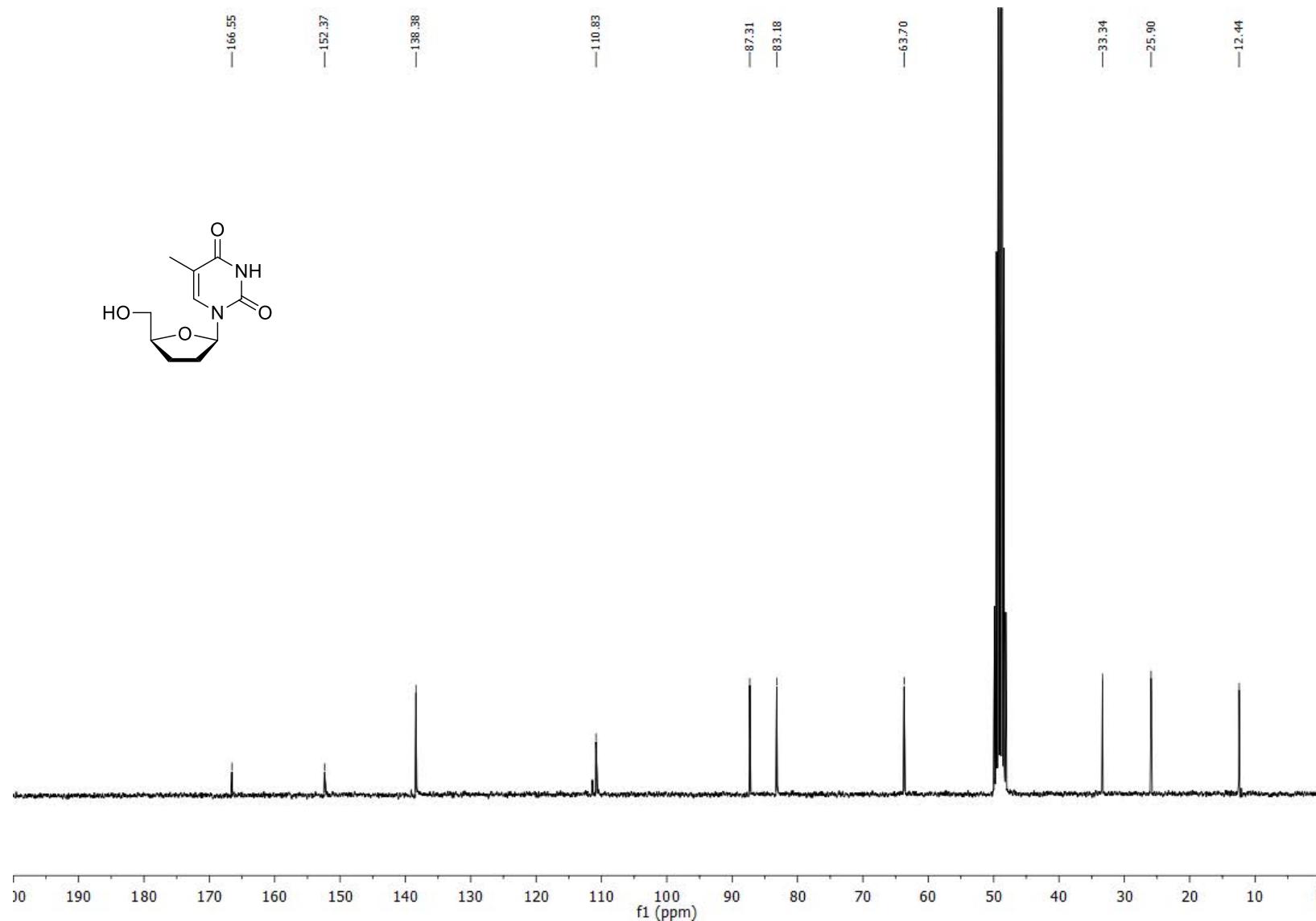
$^1\text{H-NMR}$  (300.13 MHz, MeOH- $d_4$ )



S141

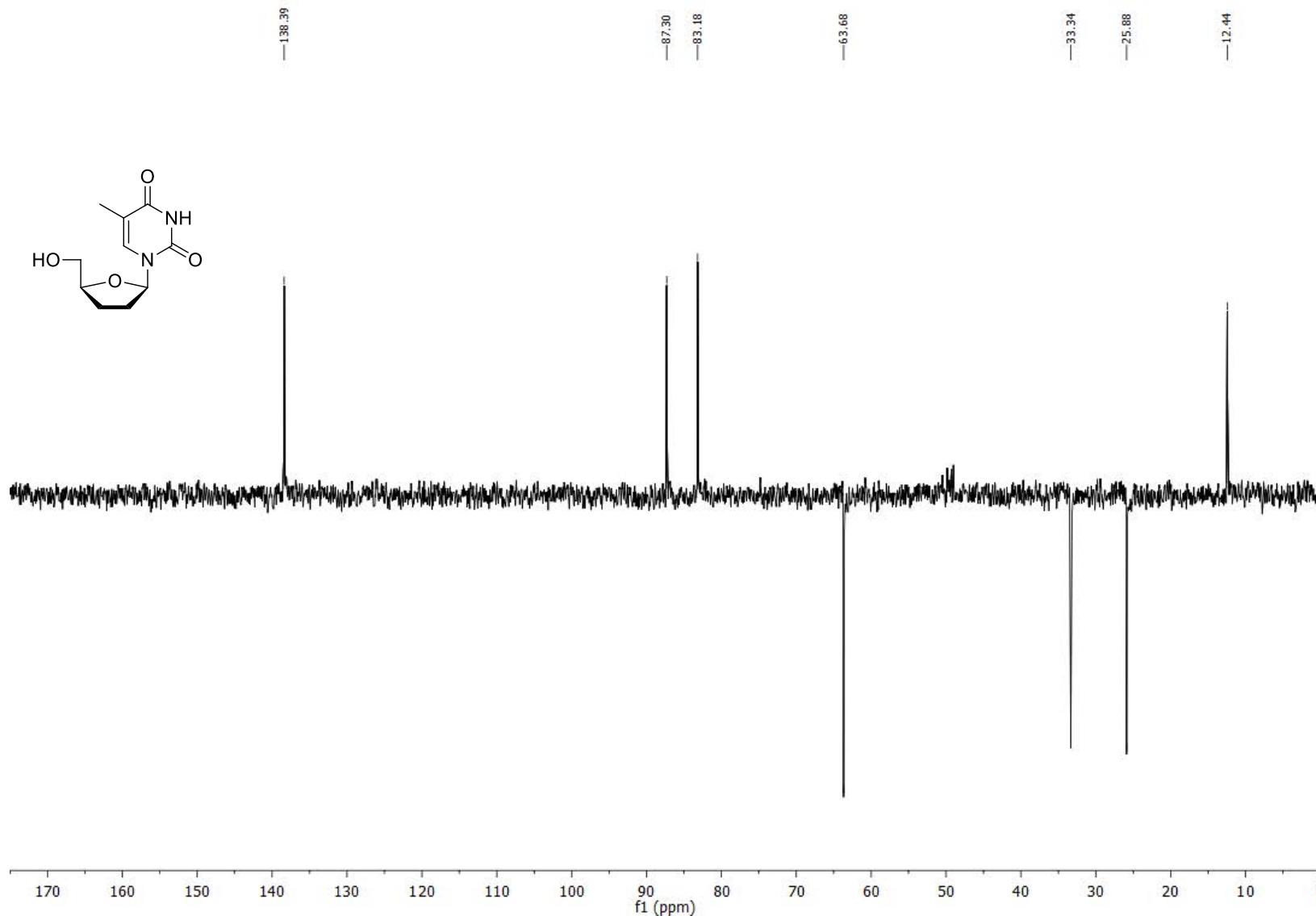
**3'-Deoxy- $\beta$ -D-5-thymidine (9b)**

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



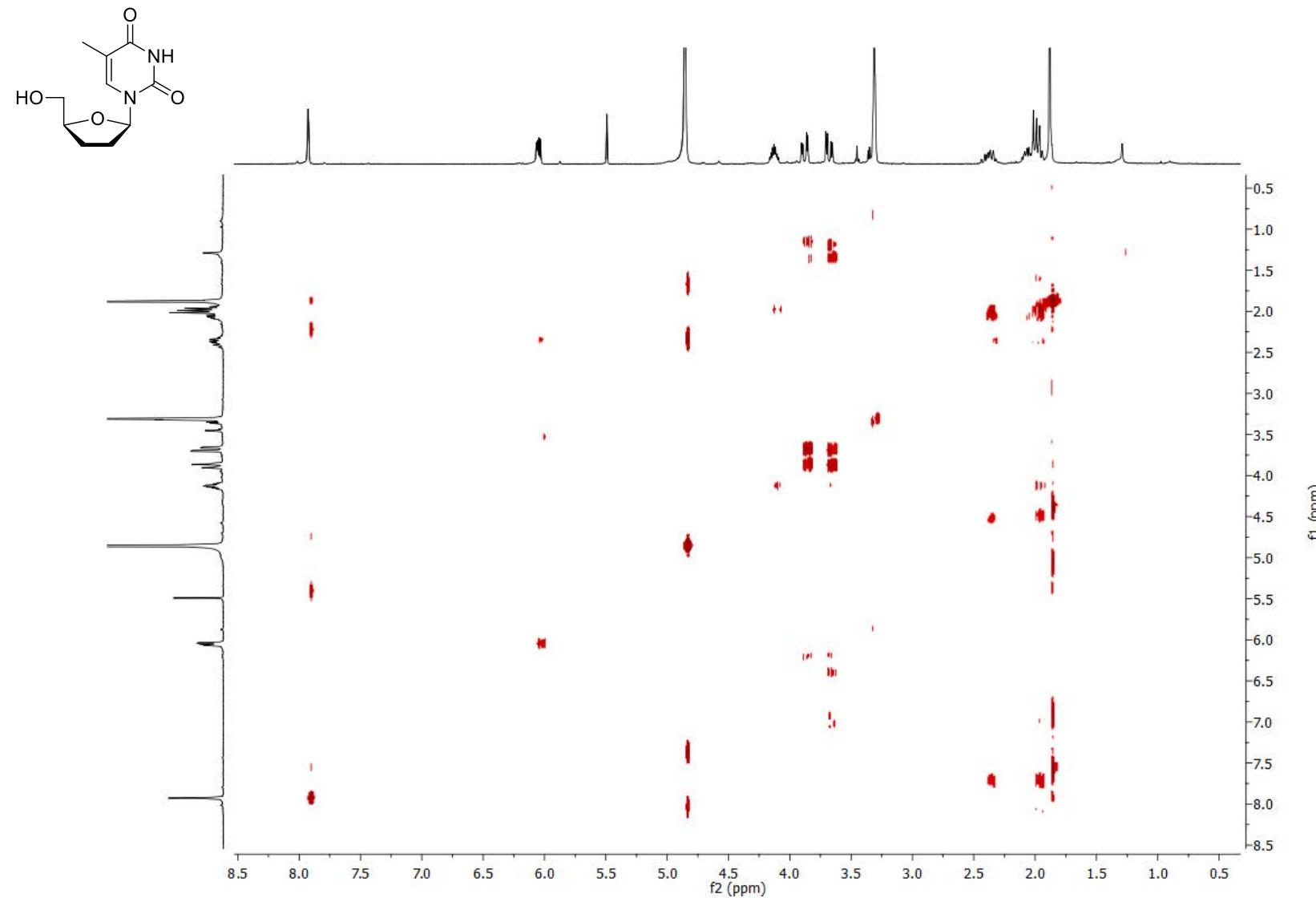
**3'-Deoxy- $\beta$ -D-5-thymidine (9b)**

DEPT NMR (75.5 MHz, MeOH-*d*<sub>4</sub>)



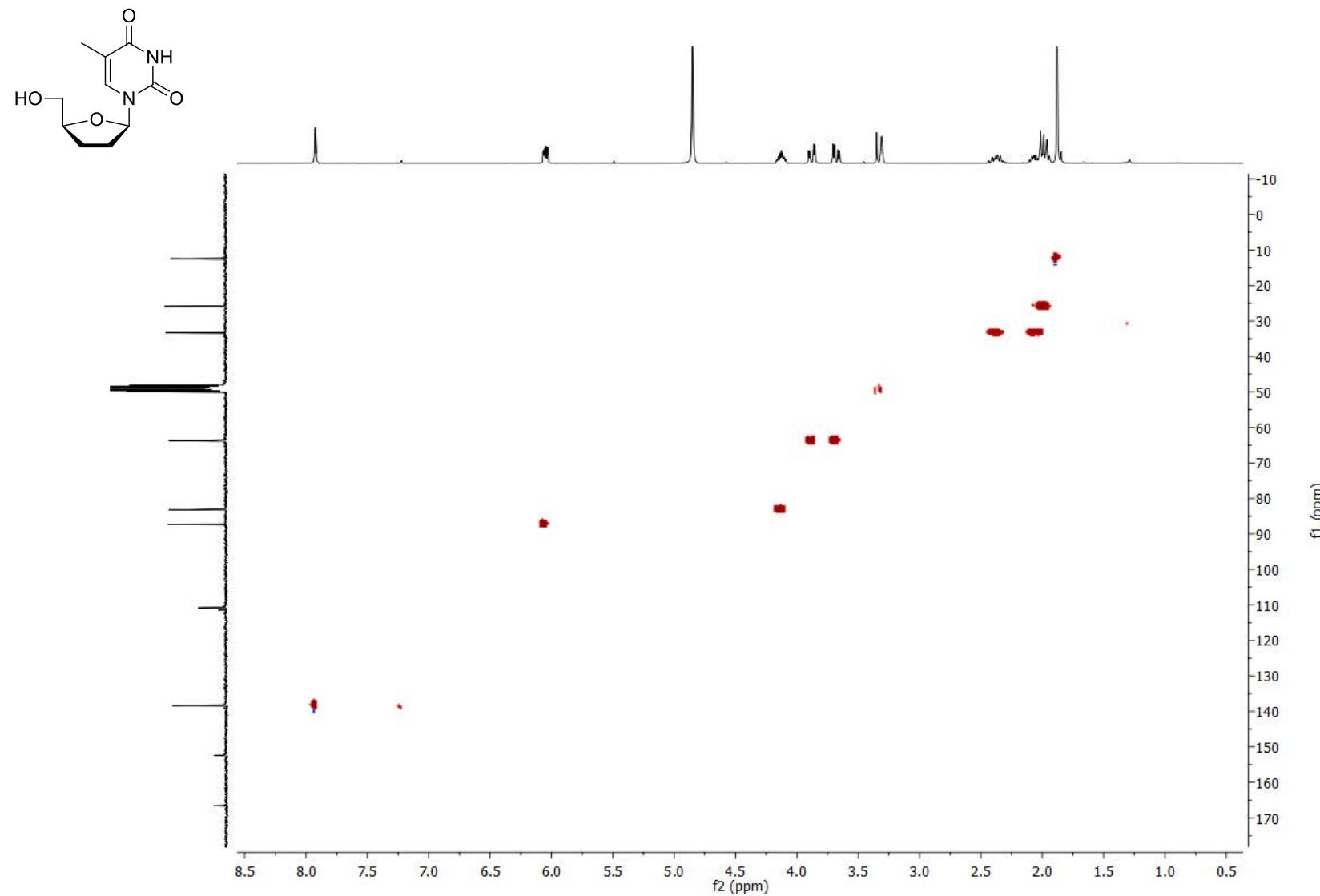
**3'-Deoxy- $\beta$ -D-5-thymidine (9b)**

COSY NMR (MeOH-*d*<sub>4</sub>)



**3'-Deoxy- $\beta$ -D-5-thymidine (9b)**

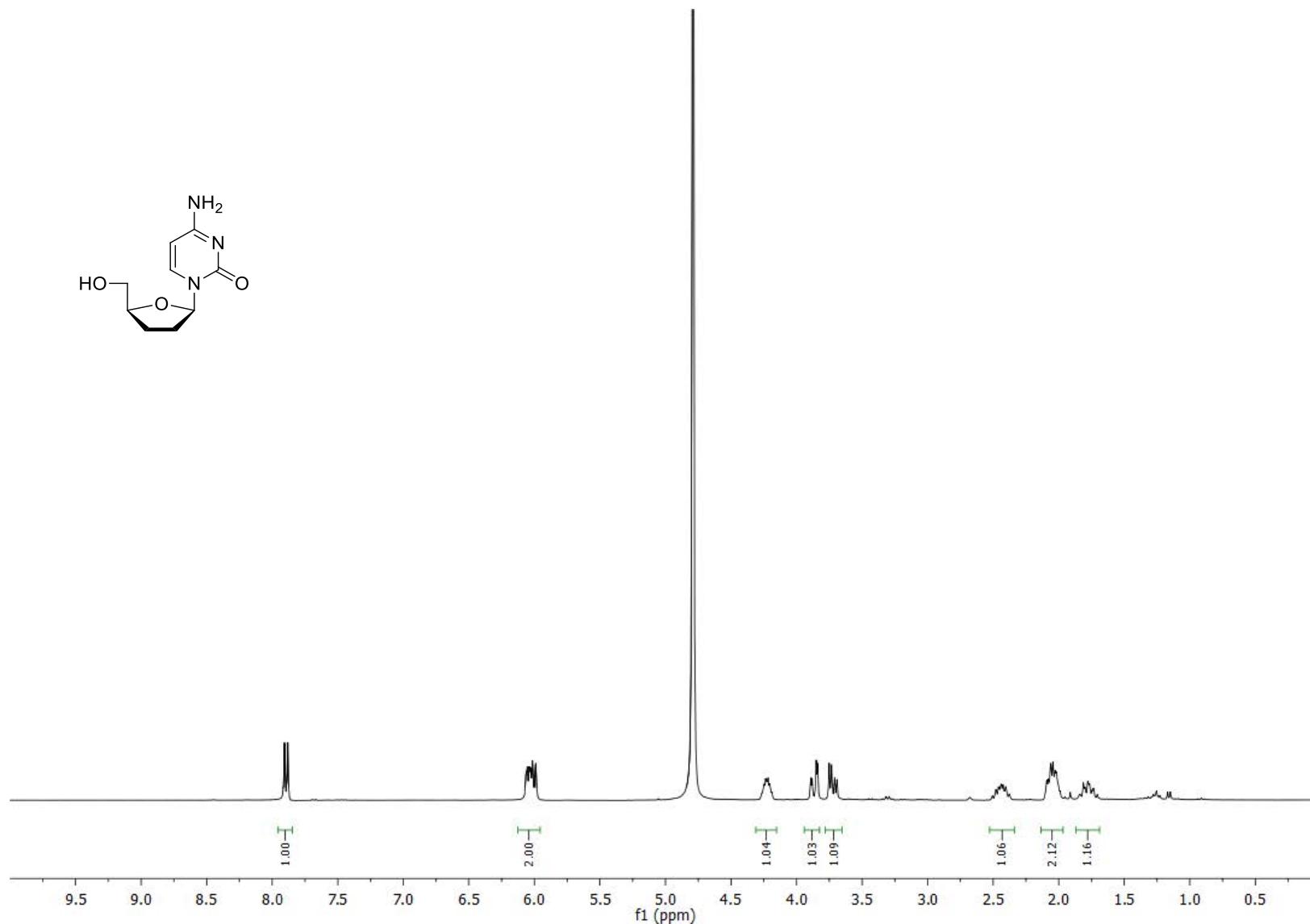
HSQC NMR ( $\text{MeOH}-d_4$ )



**2',3'-Dideoxy- $\beta$ -D-cytidine (9c)**

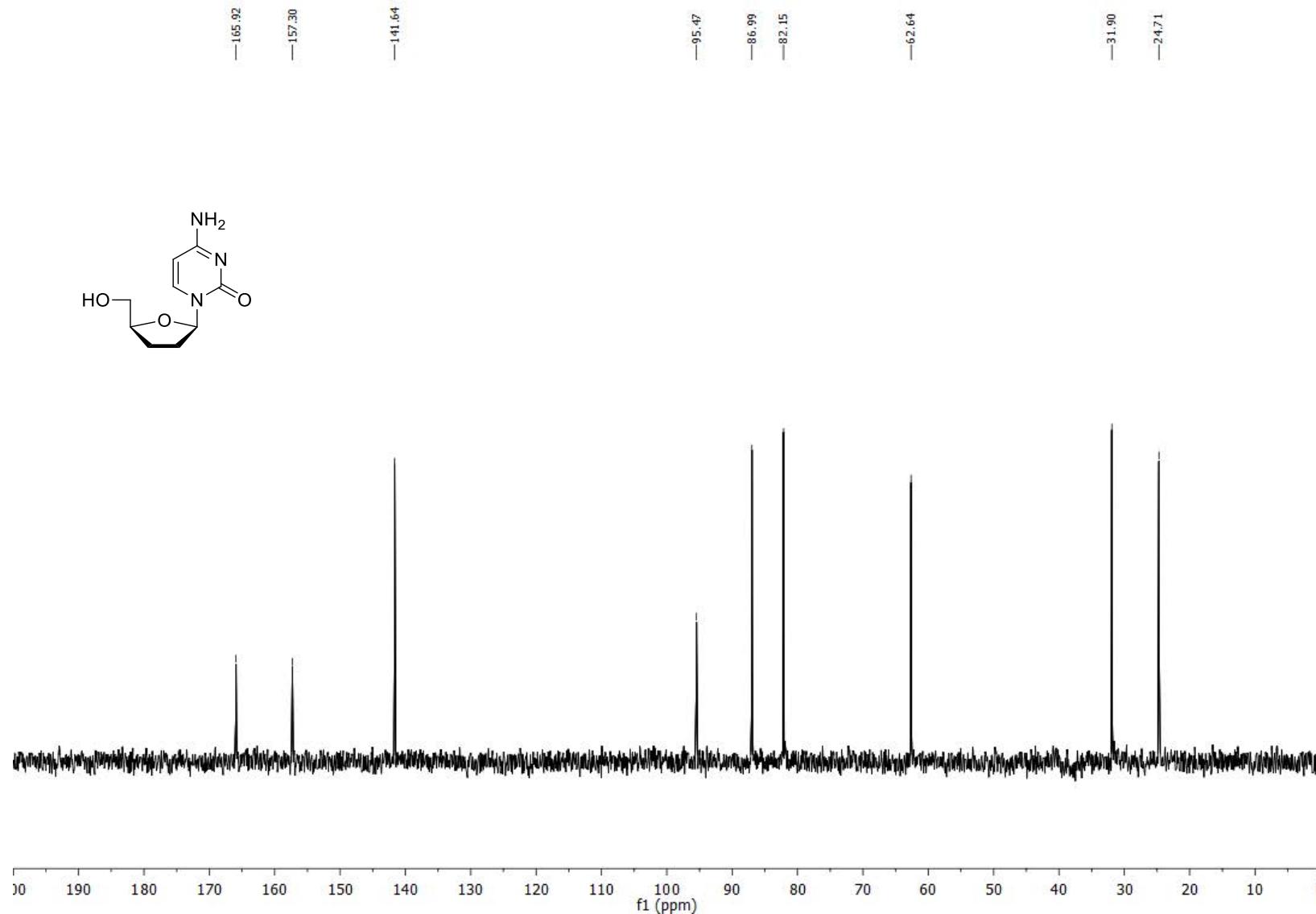
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$^1\text{H}$ -NMR (300.13 MHz,  $\text{D}_2\text{O}$ )



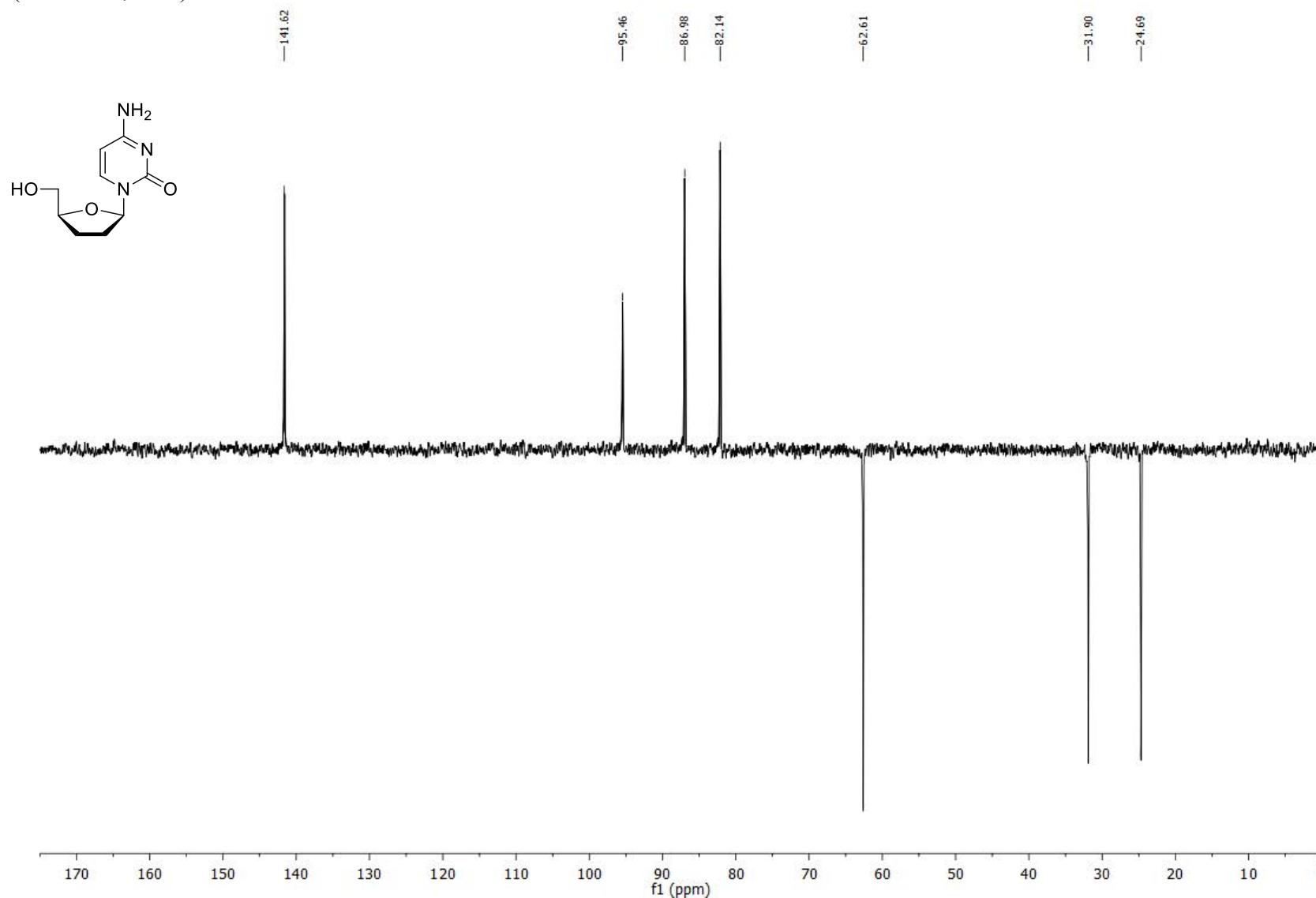
**2',3'-Dideoxy- $\beta$ -D-cytidine (9c)**

$^{13}\text{C}$ -NMR (75.5 MHz,  $\text{D}_2\text{O}$ )



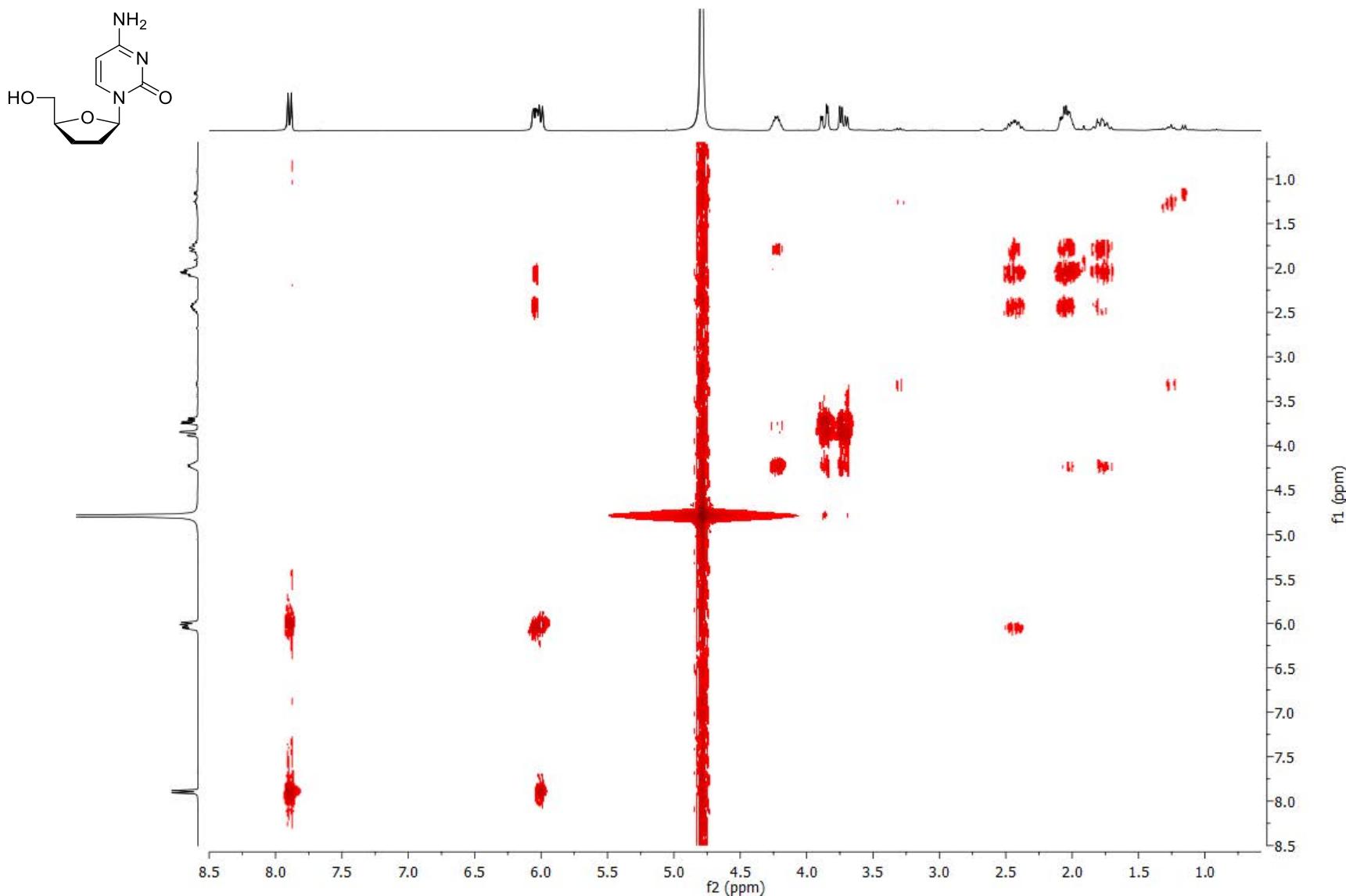
**2',3'-Dideoxy- $\beta$ -D-cytidine (9c)**

DEPT (75.5 MHz, D<sub>2</sub>O)



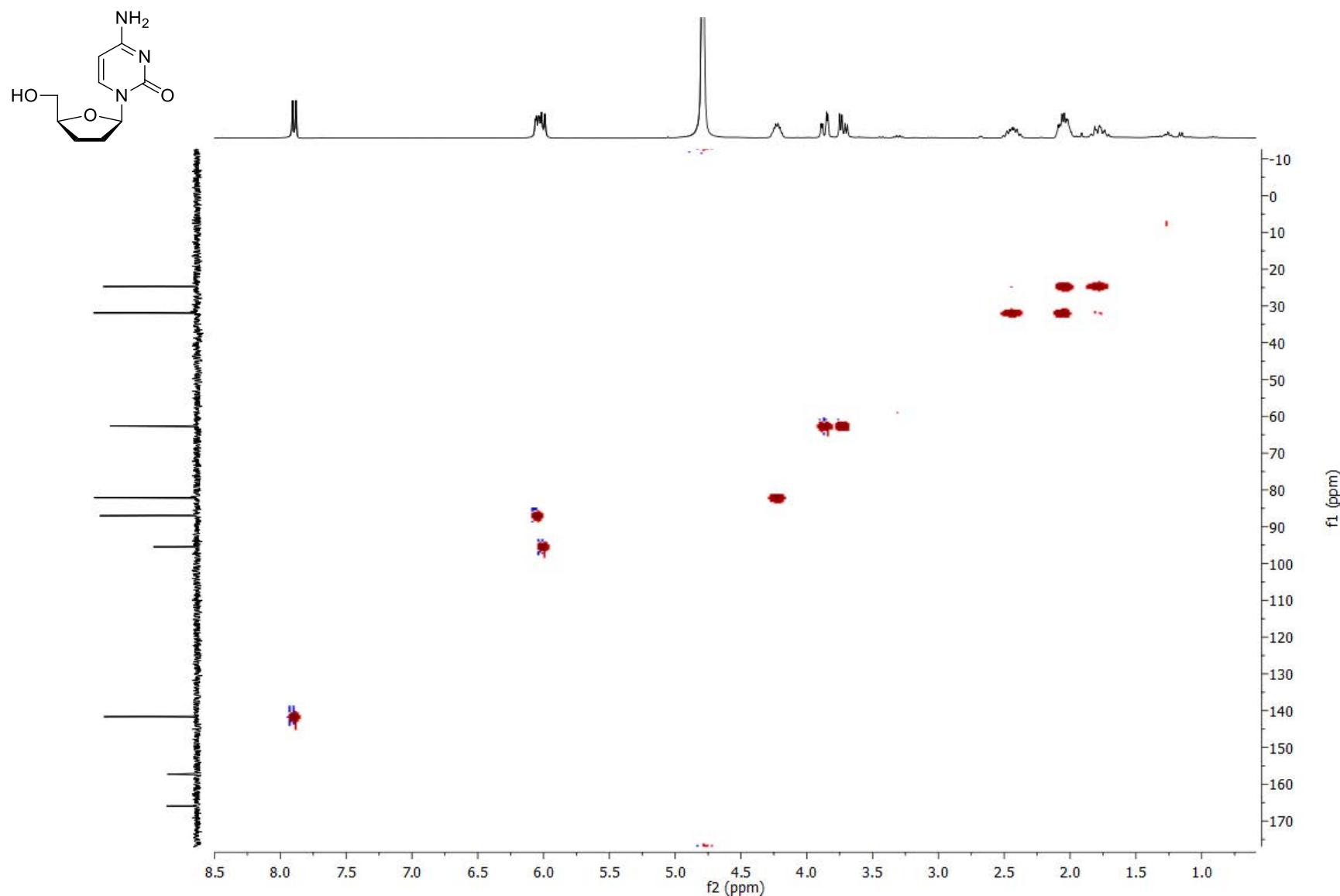
**2',3'-Dideoxy- $\beta$ -D-cytidine (9c)**

COSY NMR ( $D_2O$ )



**2',3'-Dideoxy- $\beta$ -D-cytidine (9c)**

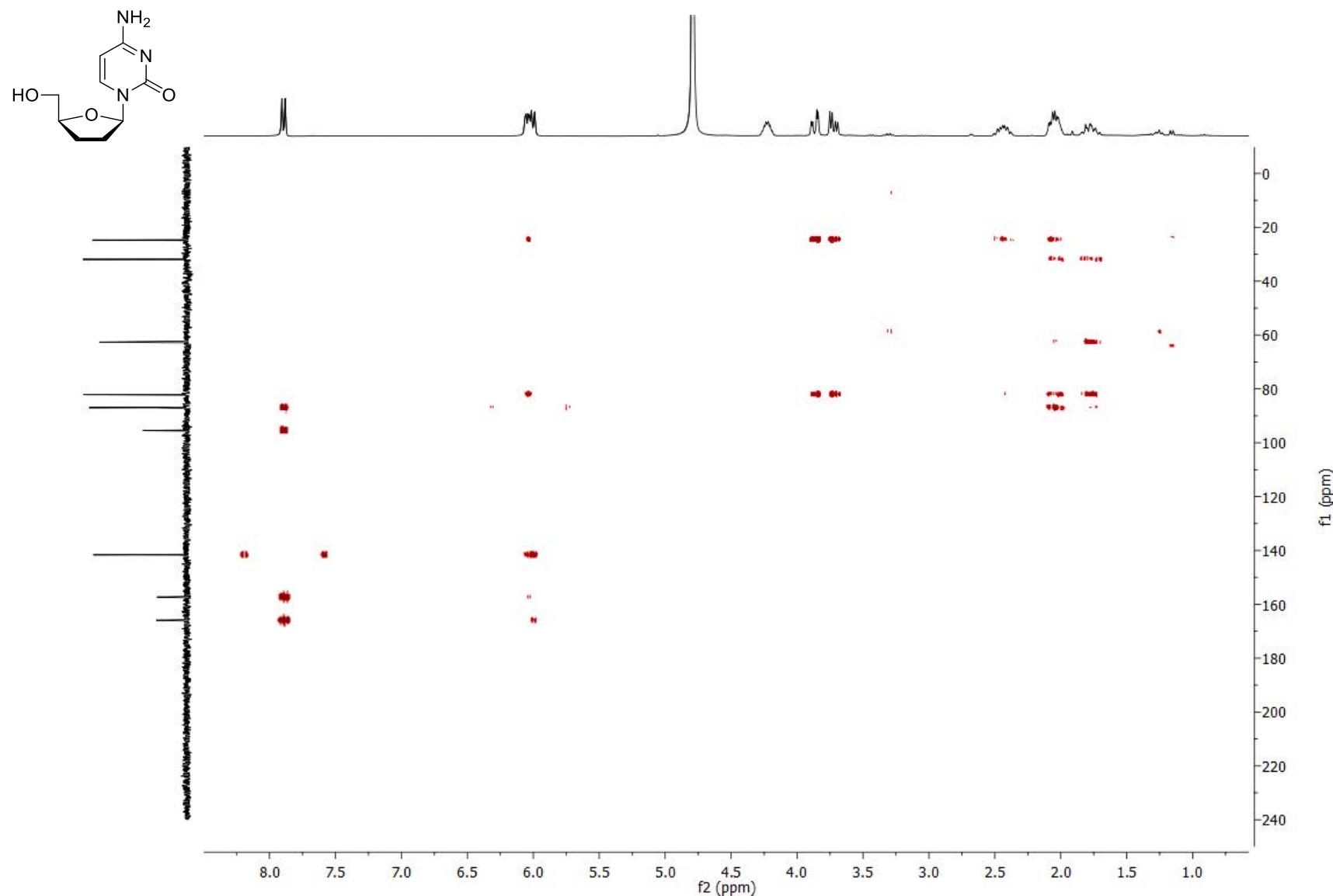
HSQC NMR ( $D_2O$ )



S150

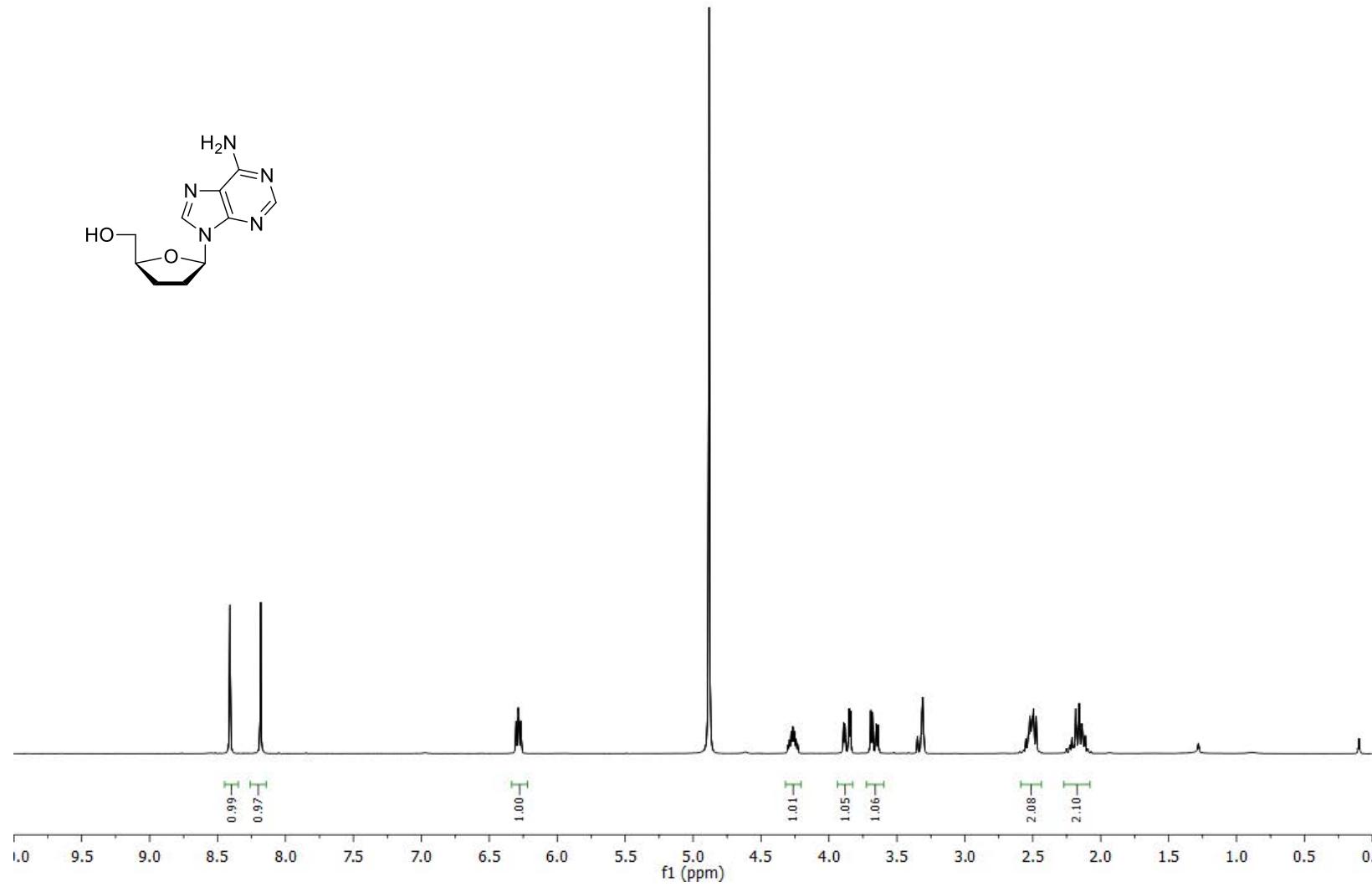
**2',3'-Dideoxy- $\beta$ -D-cytidine (9c)**

HMBC NMR ( $D_2O$ )



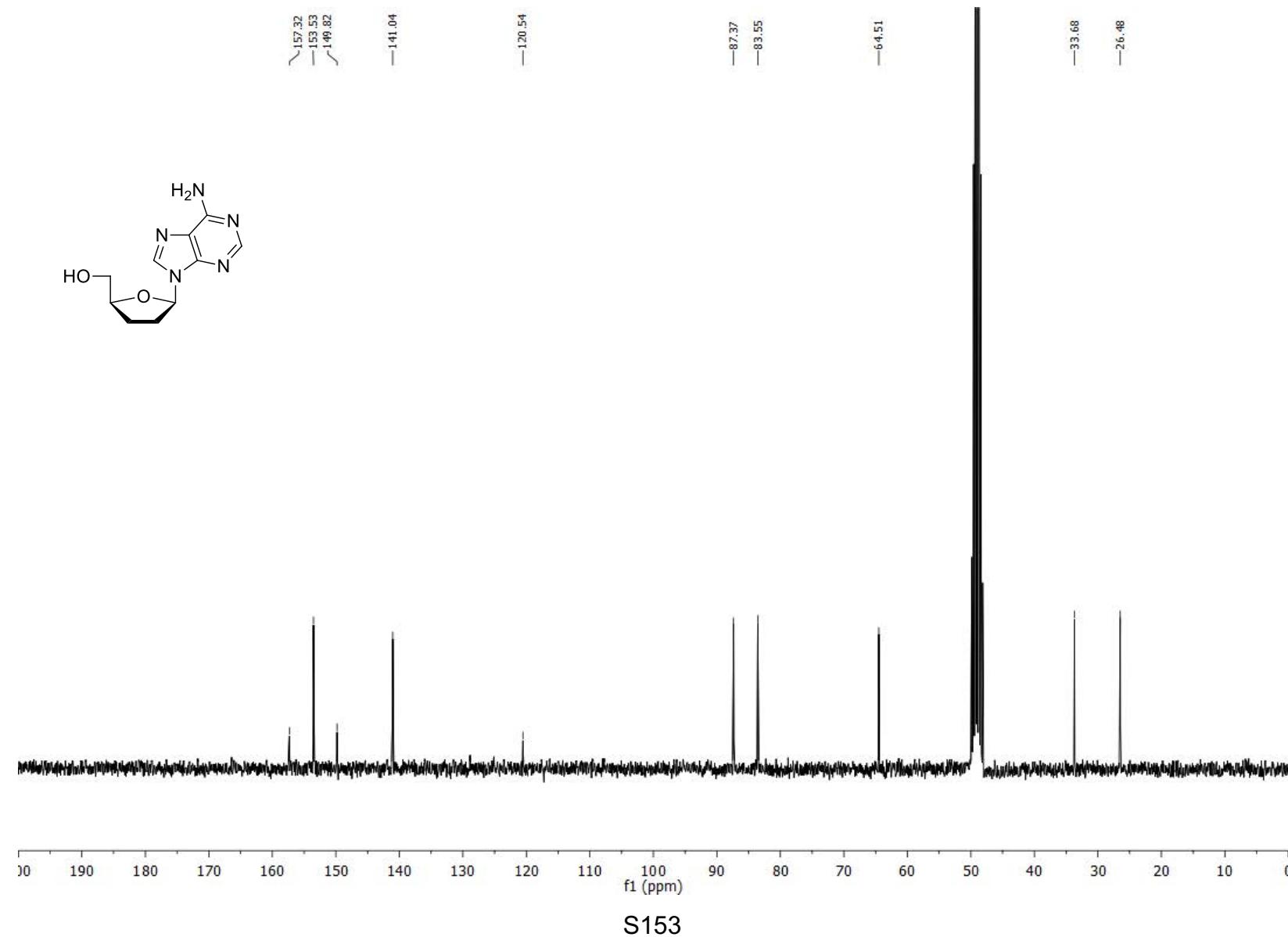
**2',3'-Dideoxy- $\beta$ -D-adenosine (9e)**

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



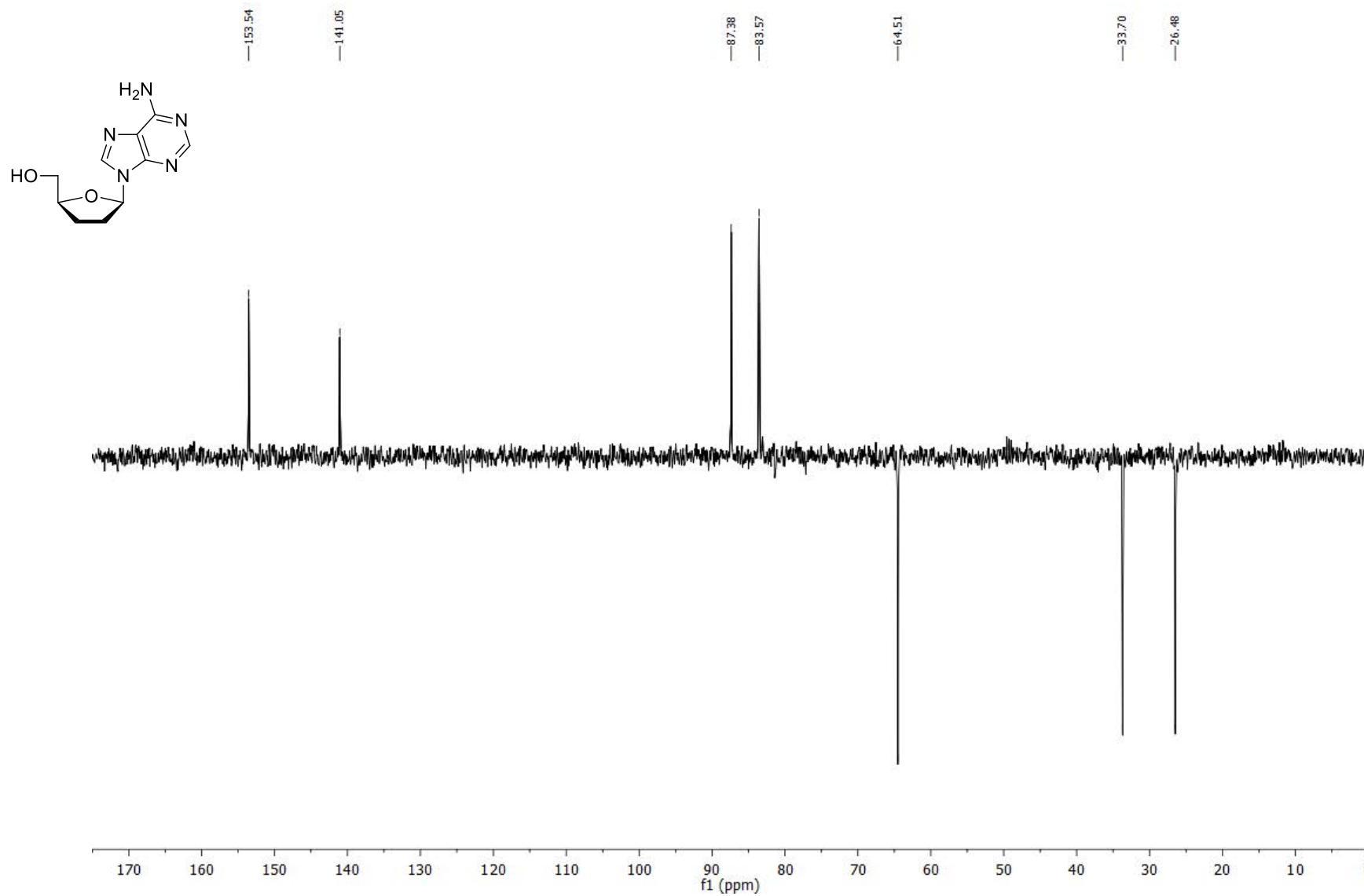
**2',3'-Dideoxy- $\beta$ -D-adenosine (9e)**

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



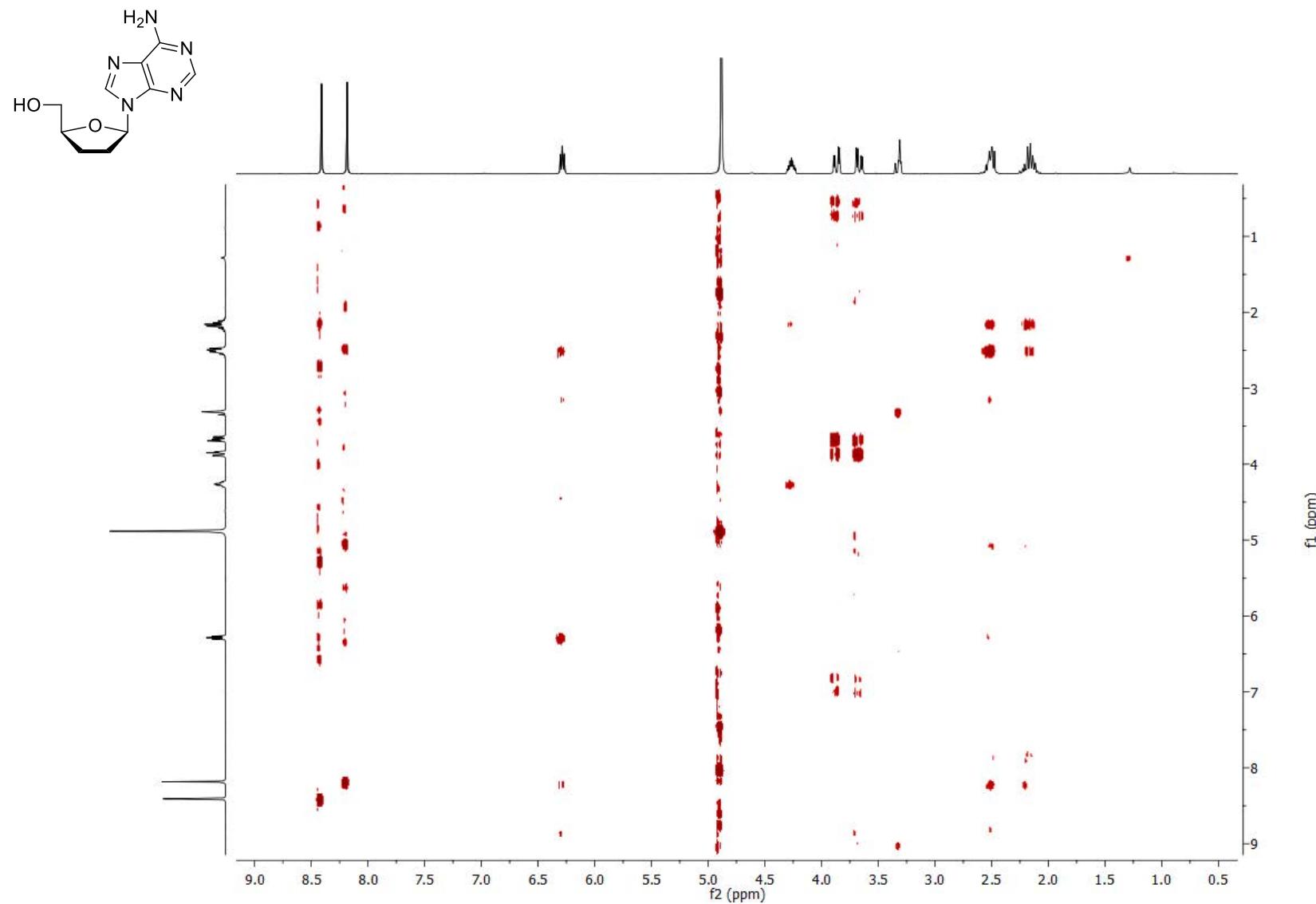
**2',3'-Dideoxy- $\beta$ -D-adenosine (9e)**

DEPT NMR (75.5 MHz, MeOH-*d*<sub>4</sub>)



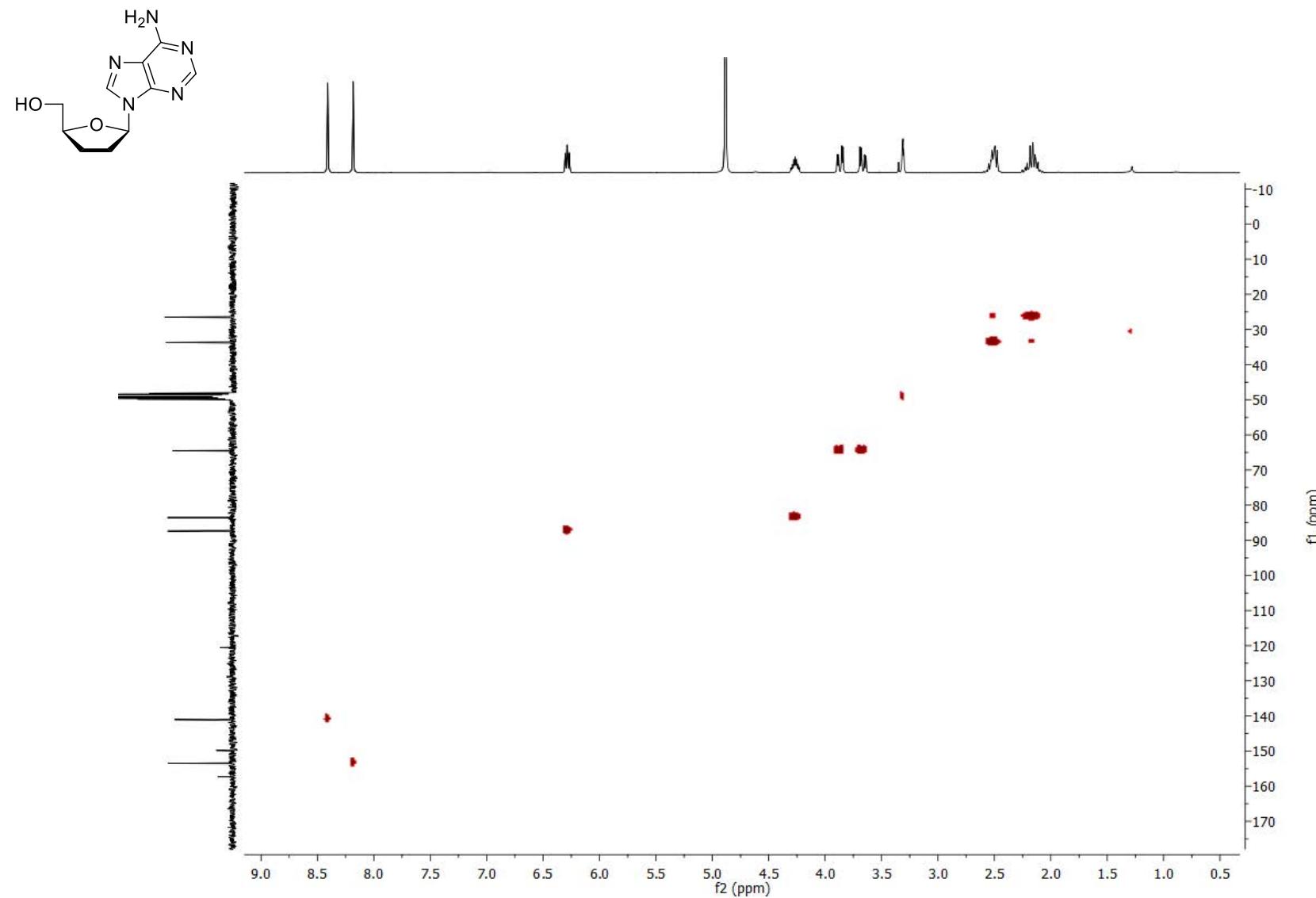
**2',3'-Dideoxy- $\beta$ -D-adenosine (9e)**

COSY NMR (MeOH-*d*<sub>4</sub>)



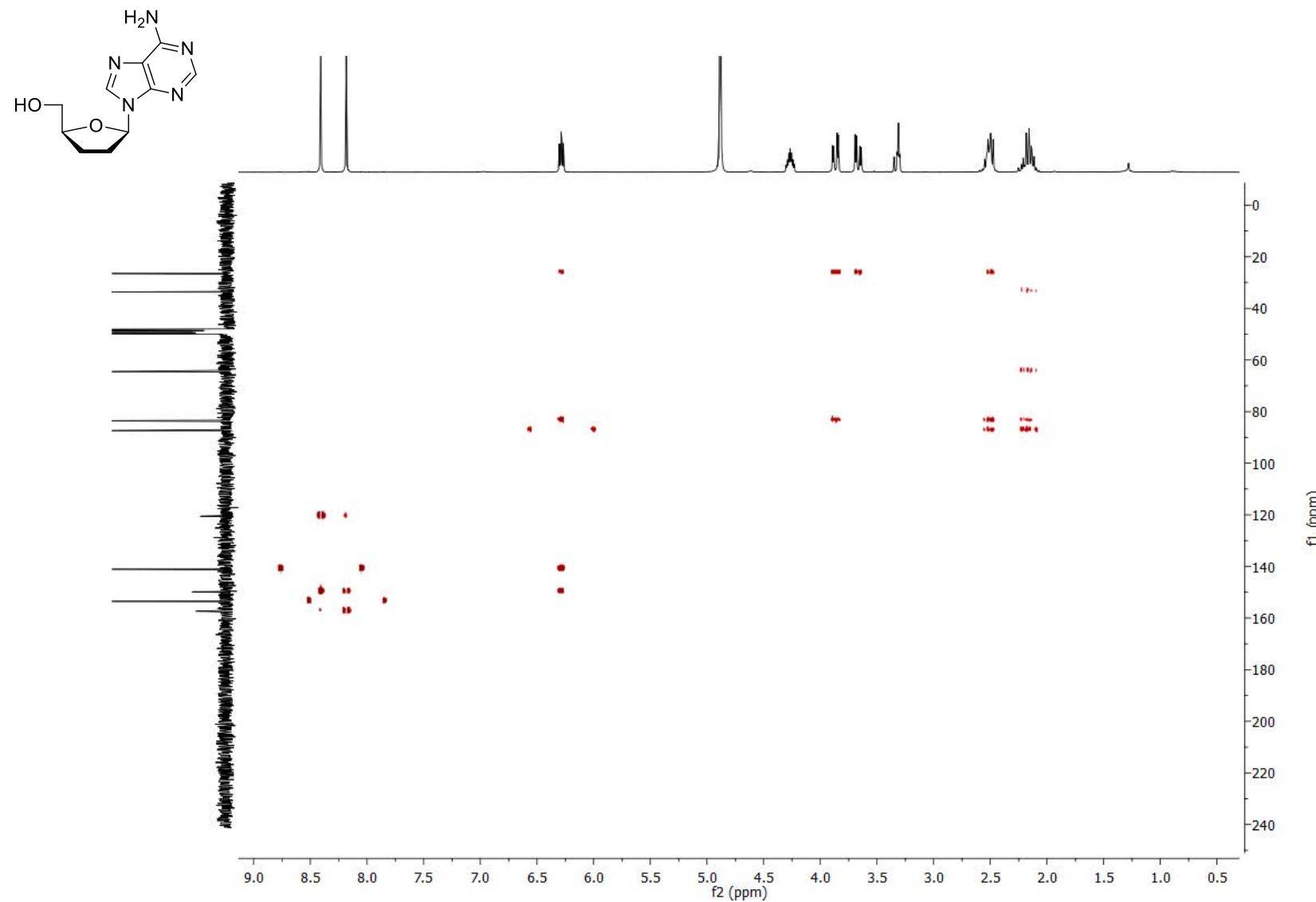
**2',3'-Dideoxy- $\beta$ -D-adenosine (9e)**

HSQC NMR ( $\text{MeOH}-d_4$ )



**2',3'-Dideoxy- $\beta$ -D-adenosine (9e)**

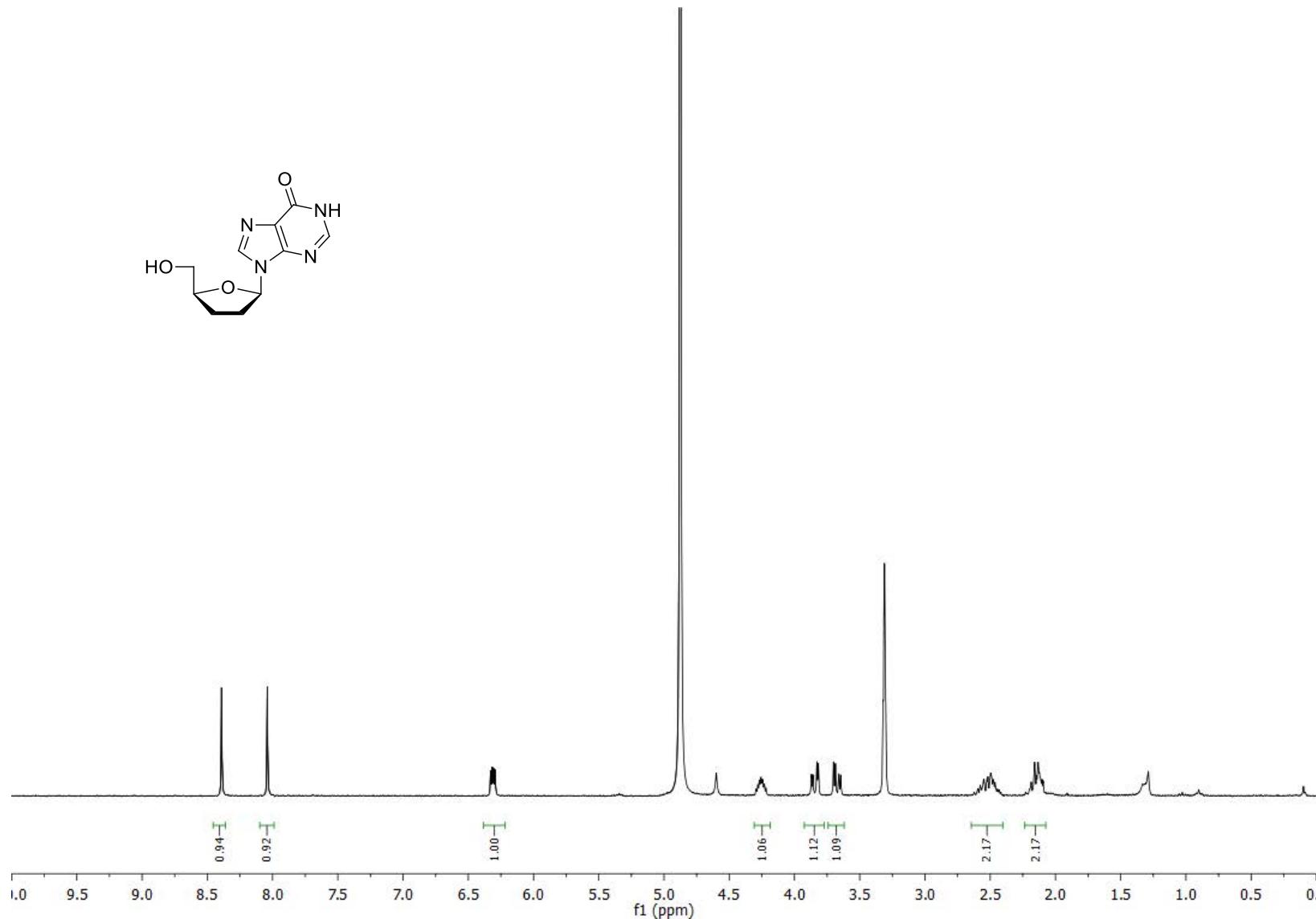
HMBC NMR ( $\text{MeOH}-d_4$ )



**2',3'-Dideoxy- $\beta$ -D-inosine (9f)**

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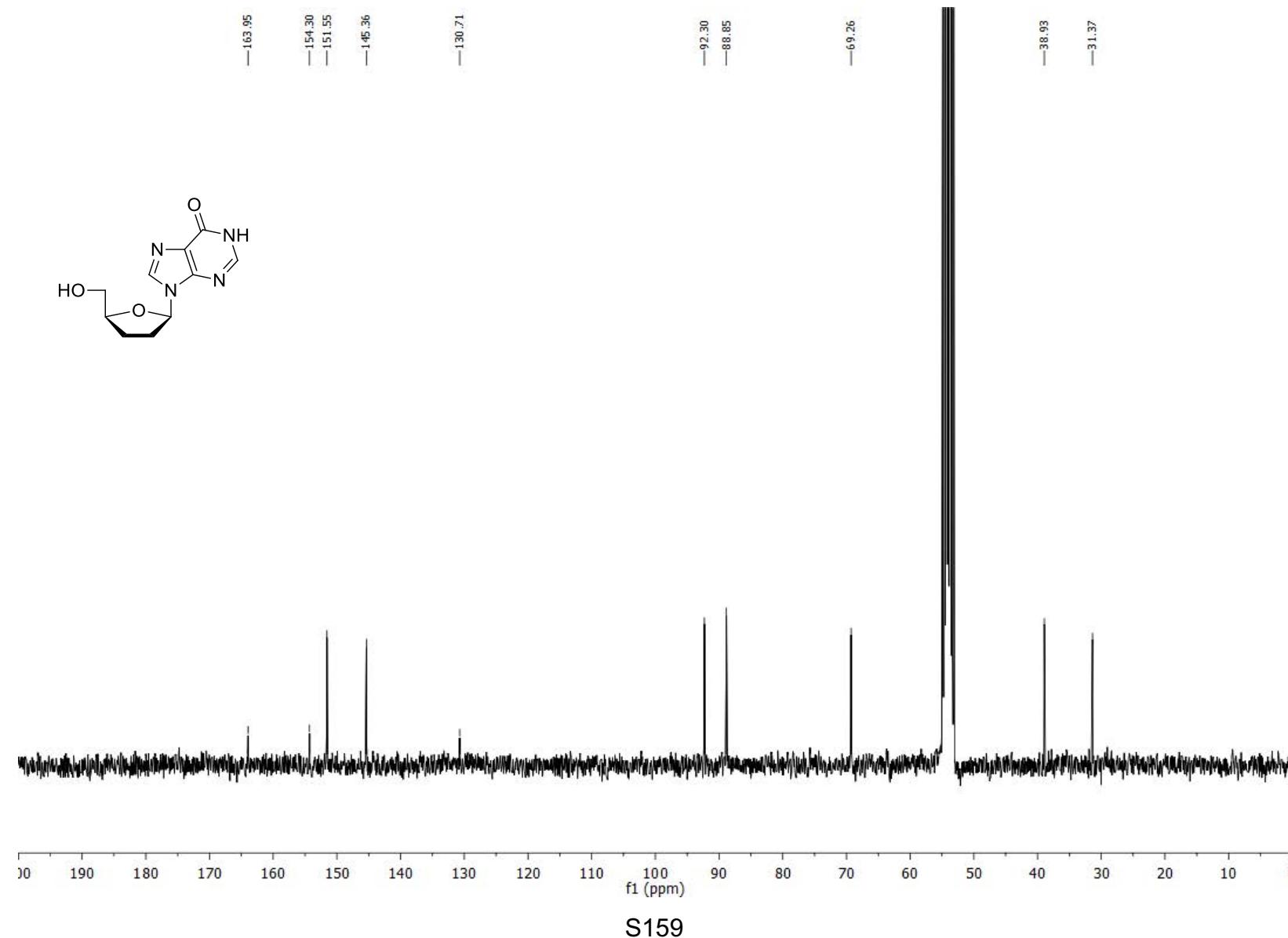
$^1\text{H-NMR}$  (300.13 MHz, MeOH- $d_4$ )



**2',3'-Dideoxy- $\beta$ -D-inosine (9f)**

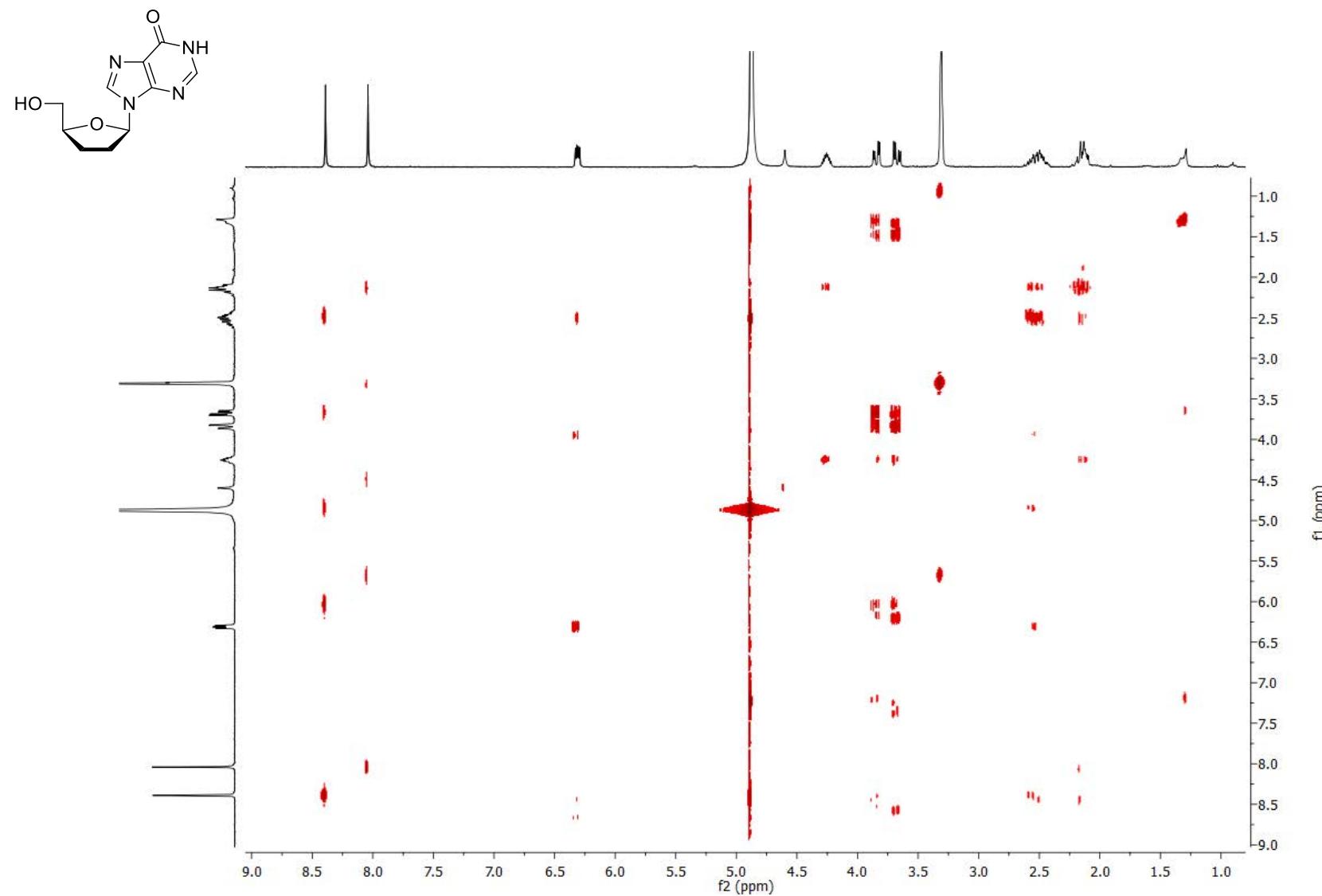
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$^{13}\text{C}$ -NMR (75.5 MHz, MeOH- $d_4$ )



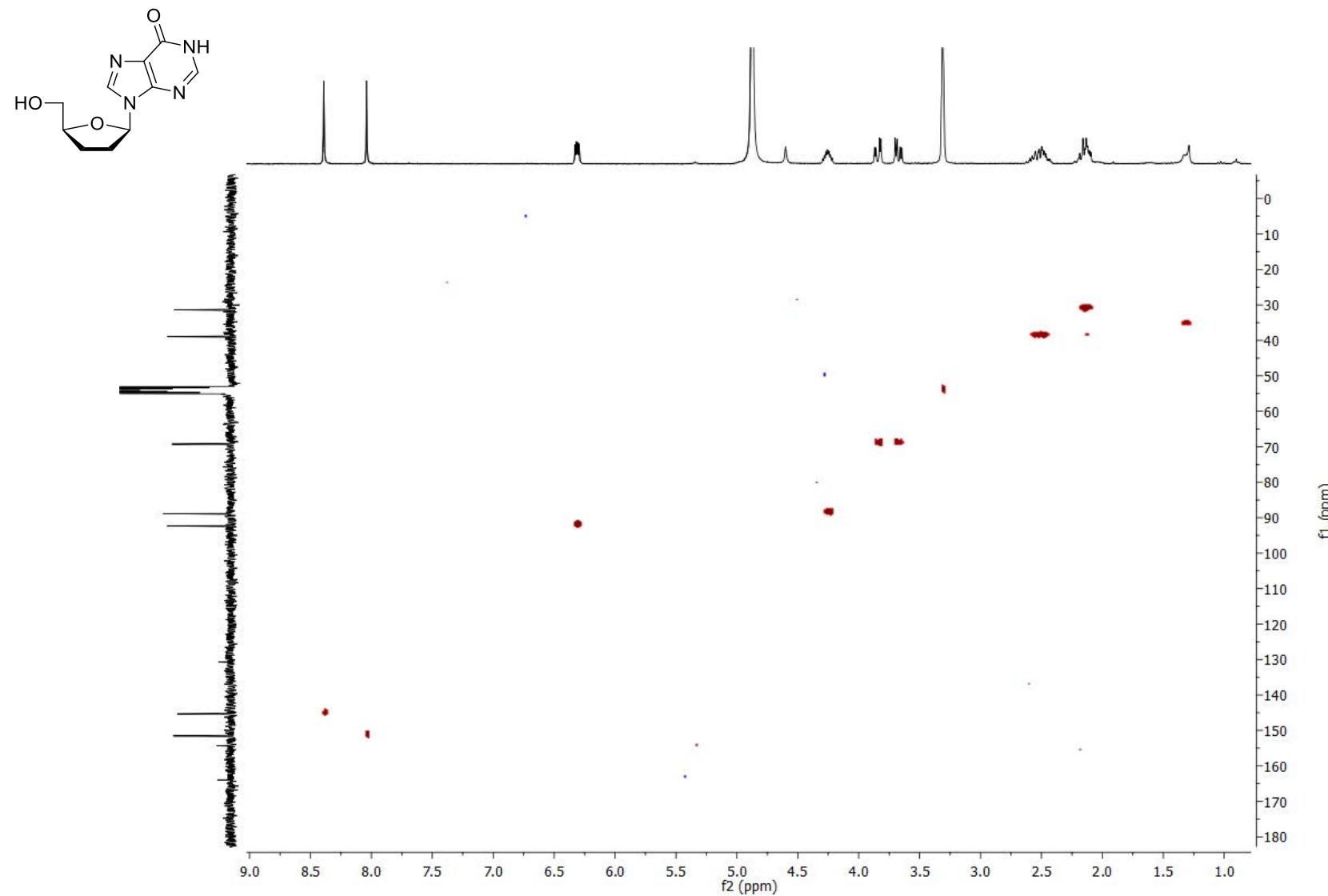
**2',3'-Dideoxy- $\beta$ -D-inosine (9f)**

COSY NMR (MeOH-*d*<sub>4</sub>)



**2',3'-Dideoxy- $\beta$ -D-inosine (9f)**

HSQC NMR ( $\text{MeOH}-d_4$ )



**2',3'-Dideoxy- $\beta$ -D-inosine (9f)**

HMBC NMR ( $\text{MeOH}-d_4$ )

