# Supporting Information for

# "Enantiomerically Pure Isophorone Diamine [3-(Aminomethyl)-3,5,5-trimethylcyclohexylamine]: A Chiral 1,4-Diamine Building Block Made Available on Large Scale"

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**General Methods.** All commercially available reagents were used without further purification. Technical isophorone diamine (**3**-*mix*) and all solvents were distilled prior to use. Dry solvents were freshly distilled under an argon atmosphere from either $CaH_2$ or Na/benzophenone. Optical rotations were measured on a precision automated polarimeter. NMR spectra were recorded on a 300 MHz spectrometer, chemical shifts are reported in ppm, coupling constants (*J* values) are reported in Hz. IR spectra were recorded on a FT-IR spectrometer. GC-MS experiment were performed using GC with a mass selective detector. Melting points were determined in an open capillary and are uncorrected. Analytical and preparative HPLC separations were performed on a UV-detector HPLC system. Chiral stationary phases were applied.

**General procedure for the preparation of IPDA *Schiff*-base ligands 10b, 10d, 10e and 10f.** To a solution of (2*R*,3*R*)-2,3-Bis(benzoyloxy)butanedioic acid (1*S*,5*R*)-(5-amino-1,3,3-trimethylcyclohexyl)-methaneamine salt (1:1) **9** (1.00 eq) and $K_2CO_3$ (2.00 eq) in water was added EtOH and a solution of the salicylic aldehyde (2.00 eq) in EtOH. A yellow precipitation was formed immediately. The reaction mixture was allowed to stir at rt for an additional hour, then water was added and the mixture cooled to 5 °C for 1 hour. The solid was filtered off, washed with EtOH and water, then dissolved in $CH_2Cl_2$. The organic layer was washed with water and brine, dried over $Na_2SO_4$, filtered and the solvent removed under reduced pressure.

**2-((*E*)-(((1*S*,5*R*)-5-((*E*)-3,5-di-*tert*-butyl-2-hydroxybenzylideneamino)-1,3,3-trimethyl-cyclohexyl)methylimino)methyl)-4,6-di-*tert*-butylphenol 10b.** The product **10b** was obtained as a yellow solid (940 mg, 63 %) with >90 % purity. Mp 110 °C. IR (ATR) 2951, 2908, 2868, 1627, 1595, 1465, 1439, 1388, 1360, 1272, 1249, 1213, 1200, 1171, 1052, 1025, 908, 876, 827, 802, 755, 731, 644 $cm^{-1}$. $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 13.95 (s(br); 1H), 13.78 (s(br); 1H), 8.45 (s; 1H), 8.32 (s; 1H), 7.43-7.45 (m; 2H), 7.14-7.08 (m; 2H), 3.66-3.52 (m; 1H), 3.35 (s; 2H), 1.73-1.60 (m; 2H), 1.56-1.37 (m; 21H), 1.37-1.28 (m; 18H), 1.28-1.20 (m; 3H), 1.19-0.98 (m; 7H). $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 166.5

(d), 164.1 (d), 158.2 (s), 158.1 (s), 139.9 (s), 139.8 (s), 136.7 (s), 136.6 (s), 126.9 (d), 126.6 (d), 126.0 (d), 125.8 (d), 178.0 (s), 177.9 (s), 75.5 (t), 61.9 (d), 48.2 (t), 47.5 (t), 43.9 (t), 36.1 (s), 35.3 (q), 35.1 (s), 35.0 (s) 34.2 (s), 31.6 (q), 29.5 (q), 28.1 (q), 24.6 (q). HRMS (EI) calcd for $C_{40}H_{62}N_2O_2^+$: 602.4811, found: 602.4810.

**2-((*E*)-(((1*S*,5*R*)-5-((*E*)-3,5-dichloro-2-hydroxybenzylideneamino)-1,3,3-trimethylcyclohexyl)methylimino)methyl)-4,6-dichlorophenol 10d.** The product **10d** was obtained as a yellow solid (1.08 g, 72 %) with >90 % purity. Mp 90 °C. IR (ATR) 2954, 2917, 2845, 1631, 1451, 1383, 1365, 1291, 1212, 1177, 1049, 1026, 906, 865, 853, 729, 698, 666 $cm^{-1}$. $^1H$ NMR (300 MHz, $CDCl_3$) $\delta$ 14.83-14.35 (m; 2H), 8.35-8.27 (m; 1H), 8.26-8.17 (m; 1H), 7.45-7.35 (m; 2H), 7.20-7.07 (m; 2H), 3.65 (tt; $J$ = 11.6 Hz, $J$ = 3.8 Hz, 1H), 3.46-3.29 (m; 2 H), 1.73-1.56 (m; 2H), 1.51-1.27 (m; 3H), 1.27-0.88 (m; 10H). $^{13}C$ NMR (75 MHz, $CDCl_3$) $\delta$164.1 (d), 161.6 (d), 157.7 (s), 157.3 (s), 132.3 (d), 132.2 (d), 129.1 (d), 128.8 (d), 123.2 (s), 123.0 (s), 122.4 (s), 122.0 (s), 119.1 (s), 119.0 (s), 73.8 (t), 60.8 (d), 47.7 (t), 46.8 (t), 43.3 (t), 36.0 (s), 35.0 (q), 31.5 (s), 27.8 (q), 24.2 (q). HRMS (EI) calcd for $C_{24}H_{26}Cl_4N_2O_2^+$: 514.0748, found: 514.0745.

**1-((*E*)-(((1*S*,3*R*)-3-(((*E*)-(2-hydroxynaphthalen-1-yl)methyleneamino)methyl-3,5,5-trimethylcyclohexylimino)methyl)naphthalen-2-ol 10e.** The product **10e** was obtained as a brown solid (780 mg, 52 %) with >90 % purity. Mp 78 °C. IR (ATR) 2951, 2919, 1708, 1623, 1543, 1523, 1353, 1208, 1139, 1033, 833, 749 $cm^{-1}$. $^1H$ NMR (300 MHz, $CDCl_3$) $\delta$ 14.85 (s(br); 1H), 14.63 (s(br); 1H), 9.03-8.59 (m; 2H), 8.02-7.82 (m; 2H), 7.77-7.57 (m; 4H), 7.52-7.37 (m; 2H), 7.34-7.16 (m; 2H), 7.04-6.89 (m; 2H), 3.85-3.64 (m; 1H), 3.47-3.22 (m; 2H), 1.97-1.75 (m; 2H), 1.51-1.30 (m; 3H), 1.30-0.87 (m; 10H). $^{13}C$ NMR (75 MHz, $CDCl_3$) $\delta$175.3 (s), 158.9 (d), 156.2 (d), 137.1 (d), 136.9 (d), 133.6 (s), 133.5 (s), 129.2 (d), 129.1 (d), 128.0 (d), 127.8 (d), 126.3 (d), 126.2 (d), 124.5 (d), 124.3 (d), 122.8 (d), 122.7 (d), 118.0 (d), 117.9 (d), 106.8 (s), 106.6 (s), 68.9 (t), 56.8 (d), 47.2 (t), 46.8 (t), 43.1 (t), 36.2 (s), 34.8 (q), 31.7 (s), 27.7 (q), 23.7 (q). HRMS (EI) calcd for $C_{32}H_{34}N_2O_2^+$: 478.2620, found: 478.2621.

**2-((*E*)-(((1*S*,5*R*)-5-((*E*)-5-bromo-2-hydroxy-3-methoxybenzylideneamino)-1,3,3-trimethylcyclohexyl)methylimino)methyl)-4-bromo-6-methoxyphenol 10f.** The product **10f** was obtained as of a yellow solid (1.12 g, 75 %) with 90 % purity. Mp 85 °C. IR (ATR) 2912, 1628, 1470, 1440, 1385, 1249, 1098, 1050, 976, 863, 839, 761, 734, 102, 666 $cm^{-1}$. $^{1}H$ NMR (300 MHz, $CDCl_3$) $\delta$ 14.24 (s(br); 1H), 14.09 (s(br); 1H), 8.33-8.24 (m; 1H), 8.24-8.13 (m; 1H), 7.05-6.88 (m; 4H), 3.94-3.83 (m; 6H), 3.60 (tt; $J$ = 11.6 Hz, $J$ = 3.8 Hz, 1H), 3.42-3.38 (m; 2H), 1.70-1.57 (m; 2H), 1.50-1.29 (m; 3H), 1.28-0.91 (m; 10H). $^{13}C$ NMR (75 MHz, $CDCl_3$) $\delta$ 164.3 (d), 162.0 (d), 152.4 (s), 149.7 (s), 124.8 (d), 124.7 (d), 119.0 (s), 118.8 (s), 116.8 (d), 116.7 (d), 108.9 (s), 108.8 (s), 74.0 (t), 61.1 (d), 56.3 (q), 56.2 (q), 47.8 (t), 47.0 (t), 43.5 (t), 36.0 (s), 35.0 (q), 31.5 (s), 27.9 (q), 24.3 (q). HRMS (EI) calcd for $C_{26}H_{32}Br_2N_2O_4^+$: 596.0710, found: 596.0706.

**X-Ray Crystal structures:** X-ray crystallographic data for compounds *rac*-**4**-*cis*, **4**-*cis*, *ent*-**4**-*cis*, *rac*-**5**-*cis*, *rac*-**6**-*cis*, *rac*-**7**-*cis*, *rac*-**8**-*cis*, **9**, **10**a, **10**c and *rac*-**11** were collected as summarized in Table 1. The Table also states the depository numbers at the Cambridge Crystallographic Data Centre. From there, the full set of data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

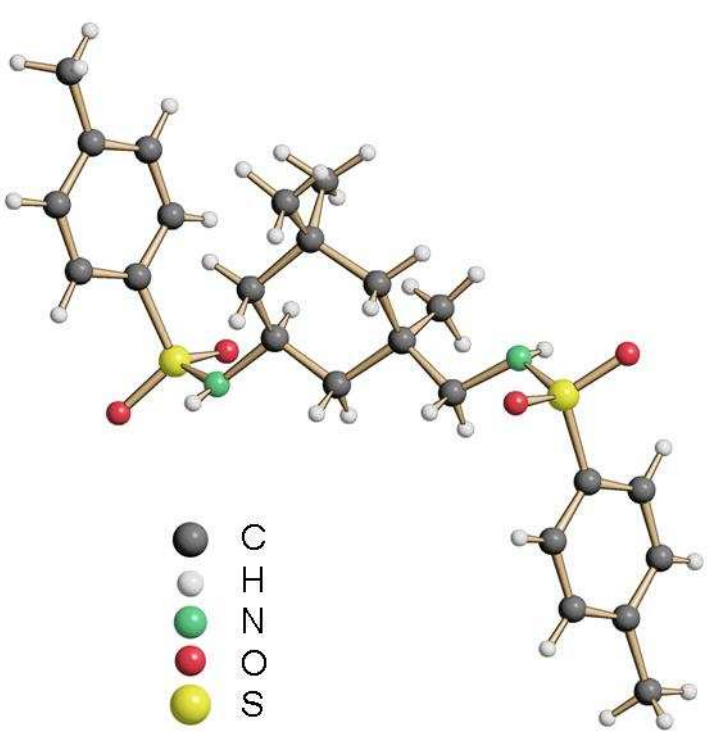
**TABLE 1.** X-Ray crystallographic data of compounds *rac*-**4**, **4**-*cis*, *ent*-**4**-*cis*, *rac*-**5**-*cis*, *rac*-**6**-*cis*, *rac*-**7**-*cis*.

| | *rac*-**4**-*cis* | **4**-*cis* | *ent*-**4**-*cis* | *rac*-**5**-*cis* | *rac*-**6**-*cis* | *rac*-**7**-*cis* |
|---|---|---|---|---|---|---|
| formula | $C_{24}H_{34}N_2O_4S_2$ | $C_{24}H_{34}N_2O_4S_2$ | $C_{24}H_{34}N_2O_4S_2$ | $C_{20}H_{38}N_2O_4$ | $C_{40}H_{42}N_2O_4$ | $C_{26}H_{34}N_2O_4$ |
| $M_r$ | 478.65 | 478.65 | 478.65 | 370.52 | 614.76 | 438.55 |
| crystal dimensions [mm] | 0.33×0.18×0.14 | 0.34×0.26×0.15 | 0.30×0.22×0.17 | 0.10×0.05×0.05 | 0.25×0.20×0.15 | 0.45×0.42×0.40 |
| crystal system | monoclinic | orthorhombic | orthorhombic | orthorhombic | monoclinic | triclinic |
| space group | $P2_1/c$ (no. 14) | $P2_12_12_1$ (no. 19) | $P2_12_12_1$ (no. 19) | Pcab (no. 61) | $P2_1/c$ (no. 14) | P1 (no. 1) |
| *a* [Å] | 8.405 (2) | 9.916 (3) | 9.919 (2) | 10.103 (7) | 9.800 (10) | 9.677 (3) |
| *b* [Å] | 29.088 (10) | 10.309 (3) | 10.308 (2) | 11.362 (7) | 28.333 (10) | 10.315 (5) |
| *c* [Å] | 10.437 (4) | 24.762 (10) | 24.801 (8) | 40.919 (4) | 12.161 (10) | 12.630 (5) |
| α [°] | 90 | 90 | 90 | 90 | 90 | 74.201 (10) |
| β [°] | 104.443 (10) | 90 | 90 | 90 | 98.82 (10) | 83.154 (10) |
| γ [°] | 90 | 90 | 90 | 90 | 90 | 75.354 (10) |
| *V* [Å$^3$] | 2471.09 (14) | 2531.22 (15) | 2535.8 (11) | 4697.1 (6) | 3336.7 (5) | 1171.86 (8) |
| $\rho_{calcd}$ [gcm$^{-3}$] | 1.287 | 1.256 | 1.254 | 1.048 | 1.224 | 1.243 |
| *Z* | 4 | 4 | 4 | 8 | 4 | 2 |
| radiation | Mo-$K_\alpha$ | Mo-$K_\alpha$ | Mo-$K_\alpha$ | Mo-$K_\alpha$ | Mo-$K_\alpha$ | Mo-$K_\alpha$ |
| scan mode | φ/ω | φ/ω | φ/ω | φ/ω | φ/ω | φ/ω |
| 2 $\Theta_{max}$ [°] | 54 | 54 | 54 | 54 | 54 | 54 |
| unique reflections | 5219 | 5419 | 5498 | 4002 | 5639 | 5079 |
| observed reflections | 2783 | 3868 | 4285 | 1474 | 2266 | 2970 |
| *R*1 | 0.055 | 0.045 | 0.043 | 0.075 | 0.072 | 0.048 |
| *wR*2 | 0.090 | 0.073 | 0.081 | 0.147 | 0.132 | 0.083 |
| $\rho_{fin}$(max) [eÅ$^{-3}$] | 0.416 | 0.283 | 0.250 | 0.190 | 0.185 | 0.229 |
| CCDC depository no. | 610536 | 610535 | 610531 | 610537 | 610538 | 610539 |

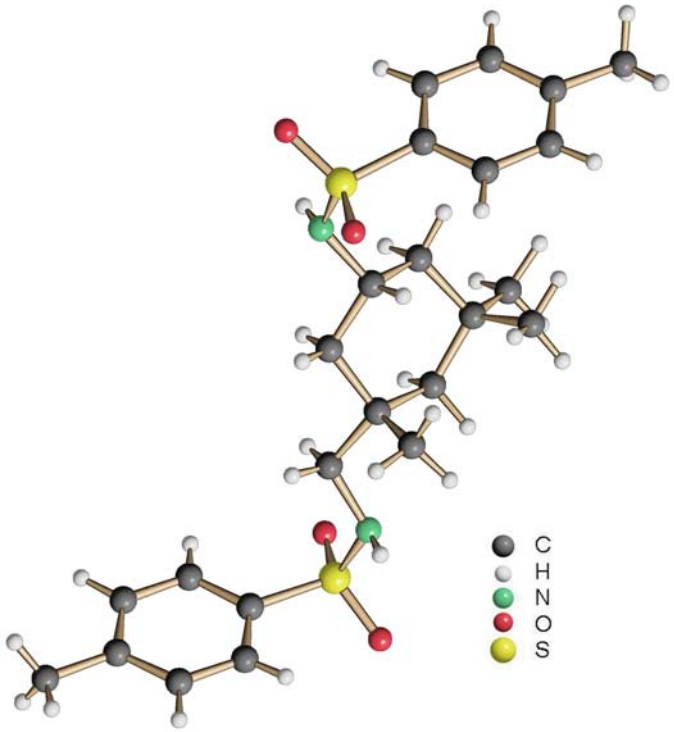
**TABLE 1, contnd.** X-Ray crystallographic data of compounds *rac*-**8**-*cis*, **9**, **10**a, **10**c, *rac*-**11**.

| | *rac*-**8**-*cis* | **9** | **10**a | **10**c | *rac*-**11***) |
|---|---|---|---|---|---|
| formula | $C_{11}H_{24}N_2O_3$ | $C_{28}H_{38}N_2O_9$ | $C_{24}H_{30}N_2O_2$ | $C_{24}H_{28}Cl_2N_2O_2$ | $C_{99}H_{127}Cl_{11}N_8Ni_4O_{17}$ |
| $M_r$ | 232.32 | 546.60 | 378.50 | 447.38 | 2325.88 |
| crystal dimensions [mm] | 0.30×0.30×0.10 | 0.20×0.20×0.05 | 0.20×0.20×0.01 | 0.20×0.05×0.02 | 0.10×0.10×0.05 |
| crystal system | orthorhombic | orthorhombic | triclinic | orthorhombic | monoclinic |
| space group | $P2_1ab$ (no. 29) | $P2_12_12_1$ (no. 19) | P1 (no. 1) | $P2_12_12_1$ (no. 19) | P21/n (no. ) |
| *a* [Å] | 7.995 (3) | 8.308 (2) | 6.084 (11) | 6.005 (3) | 17.851 (10) |
| *b* [Å] | 10.922 (3) | 9.579 (2) | 12.859 (4) | 14.508 (10) | 31.830 (10) |
| *c* [Å] | 14.461 (9) | 35.876 (10) | 14.354 (4) | 25.355 (2) | 20.587 (10) |
| α [°] | 90 | 90 | 72.851 (10) | 90 | 90 |
| β [°] | 90 | 90 | 89.224 (10) | 90 | 108.161 (5) |
| γ [°] | 90 | 90 | 77.700 (10) | 90 | 90 |
| *V* [Å$^3$] | 1262.8 (10) | 2855.3 (12) | 1047.0 (5) | 2209.0 (3) | 11115.0(9) |
| $\rho_{calcd}$ [g_cm$^{-3}$] | 1.222 | 1.272 | 1.201 | 1.345 | 1.390 |
| *Z* | 4 | 4 | 2 | 4 | 4 |
| radiation | Mo-$K_\alpha$ | Mo-$K_\alpha$ | Mo-$K_\alpha$ | Mo-$K_\alpha$ | Mo-$K_\alpha$ |
| scan mode | ω/θ | ω/θ | ω/θ | ω/θ | ω/θ |
| $2\theta_{max}$ [°] | 54 | 54 | 54 | 54 | 54 |
| unique reflections | 1481 | 3511 | 5557 | 4580 | 23327 |
| observed reflections | 1092 | 2944 | 4095 | 3122 | 12888 |
| *R*1 | 0.040 | 0.032 | 0.052 | 0.049 | 0.077 |
| *wR*2 | 0.084 | 0.065 | 0.105 | 0.088 | 0.224 |
| $\rho_{fin}$(max) [e_Å$^{-3}$] | 0.133 | 0.314 | 0.202 | 0.363 | 2.478 |
| CCDC depository no. | 610540 | 610532 | 610533 | 610534 | 610541 |

*)Best analysis possible, as the crystal was small and contained disordered solvent molecules (chloroform, ethanol, water).




**4**-*cis*

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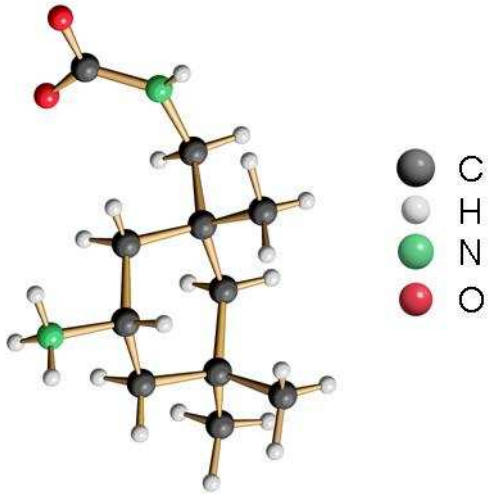


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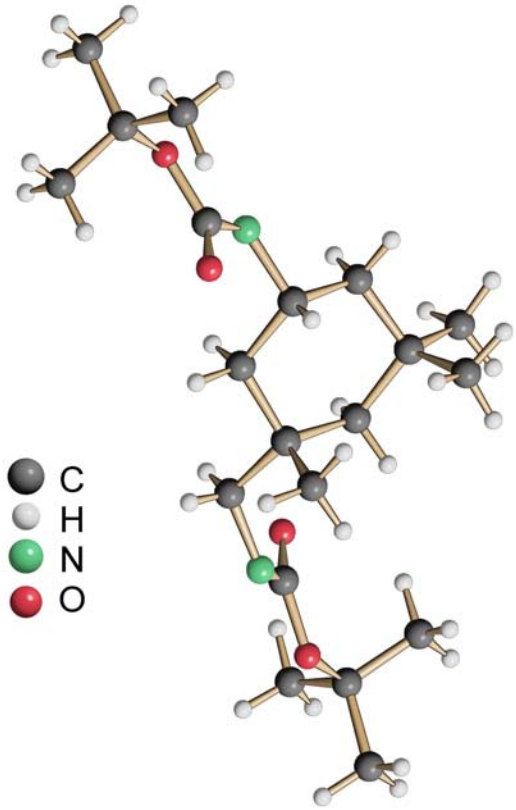


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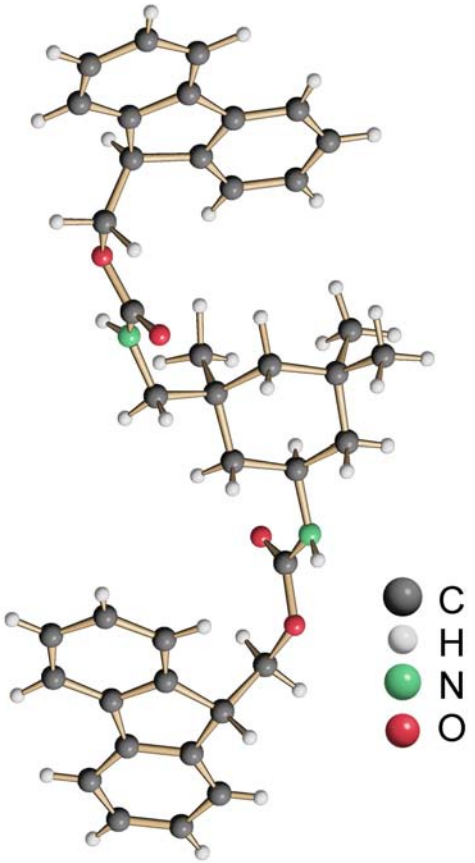
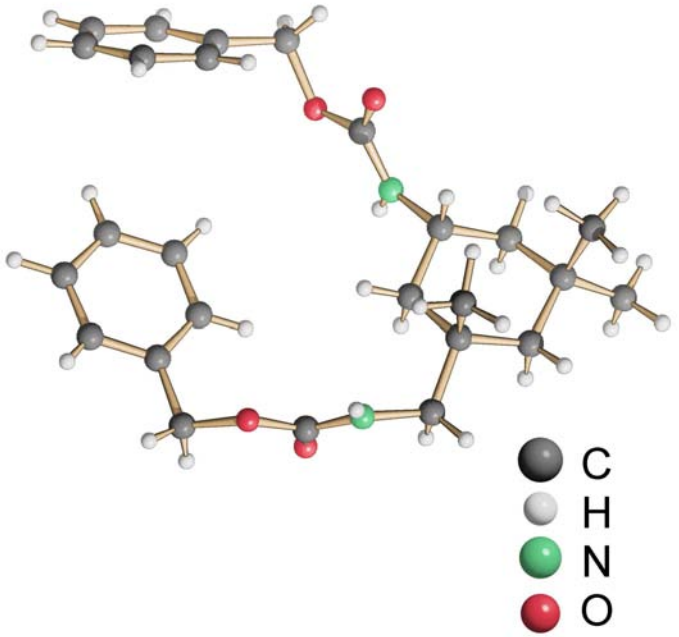
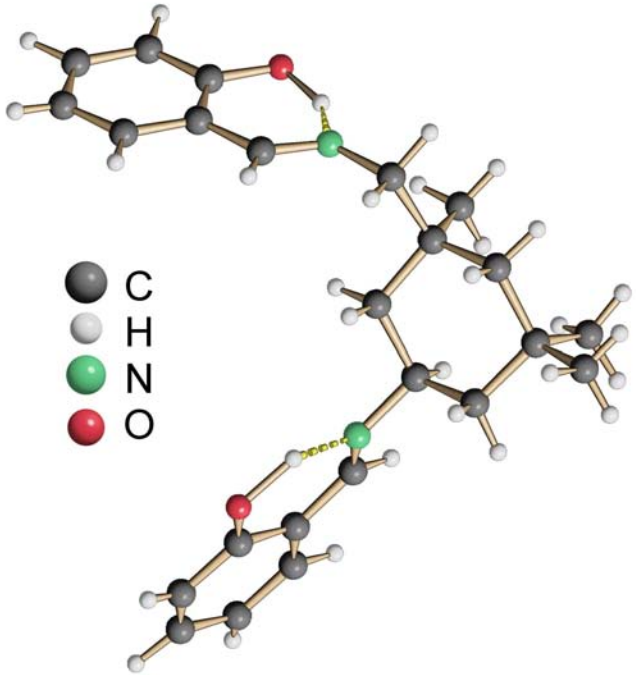


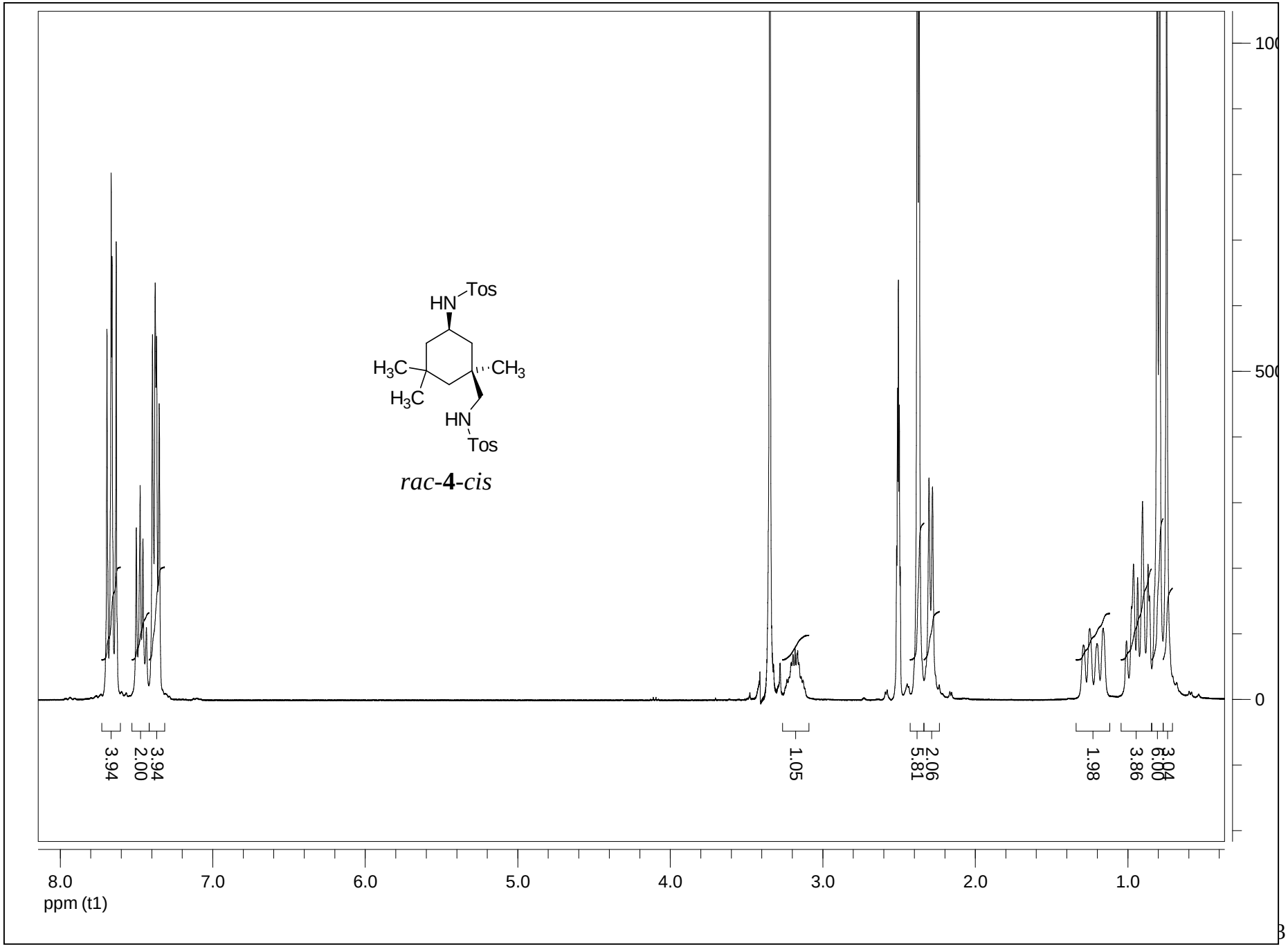

*rac*-**6**-*cis*




*rac*-**7**-*cis*




**10a**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HN
Tos
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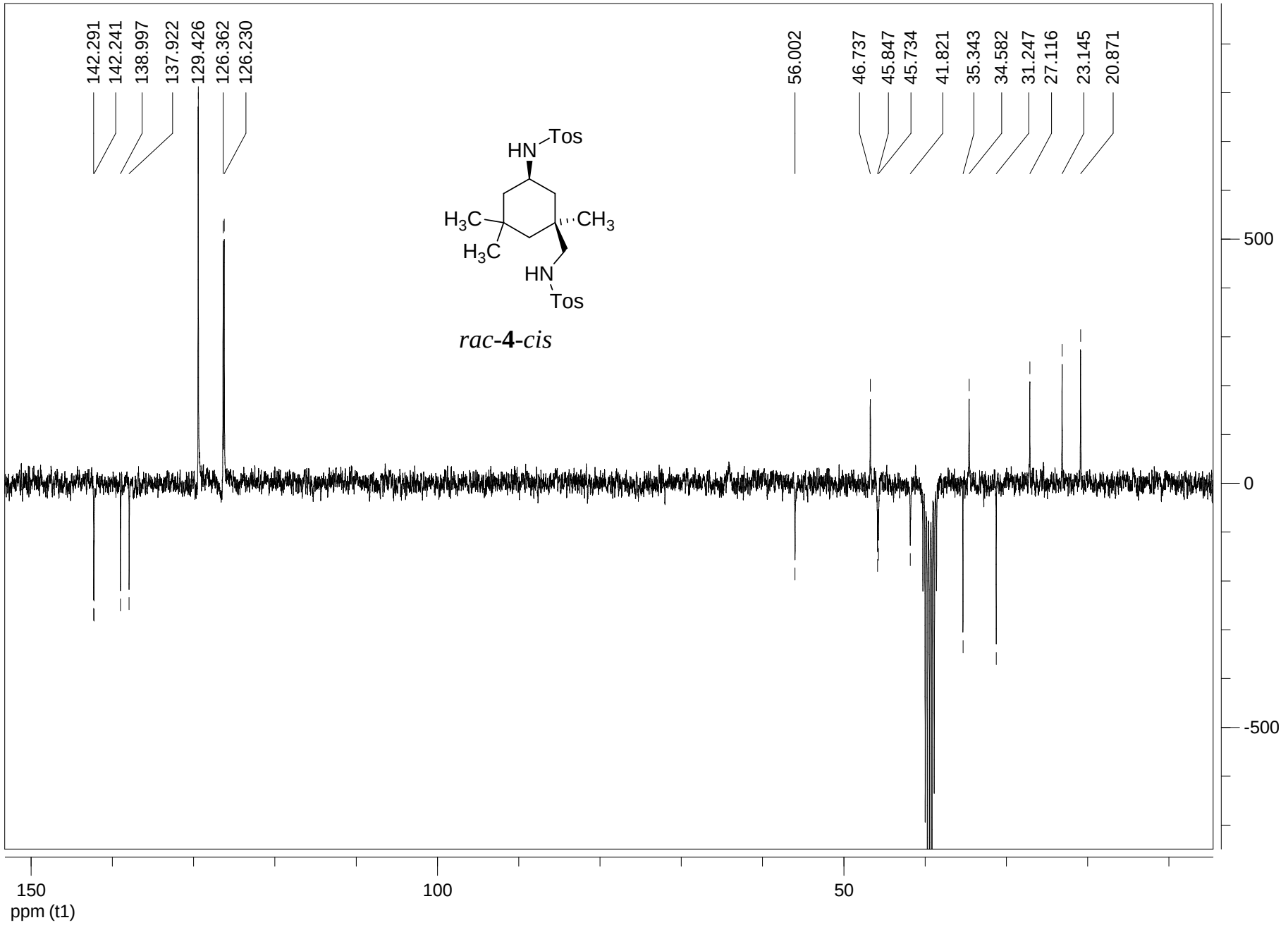
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34.582
31.247
27.116
23.145
20.871
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H3C
HN
Tos
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0
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150
ppm (t1)
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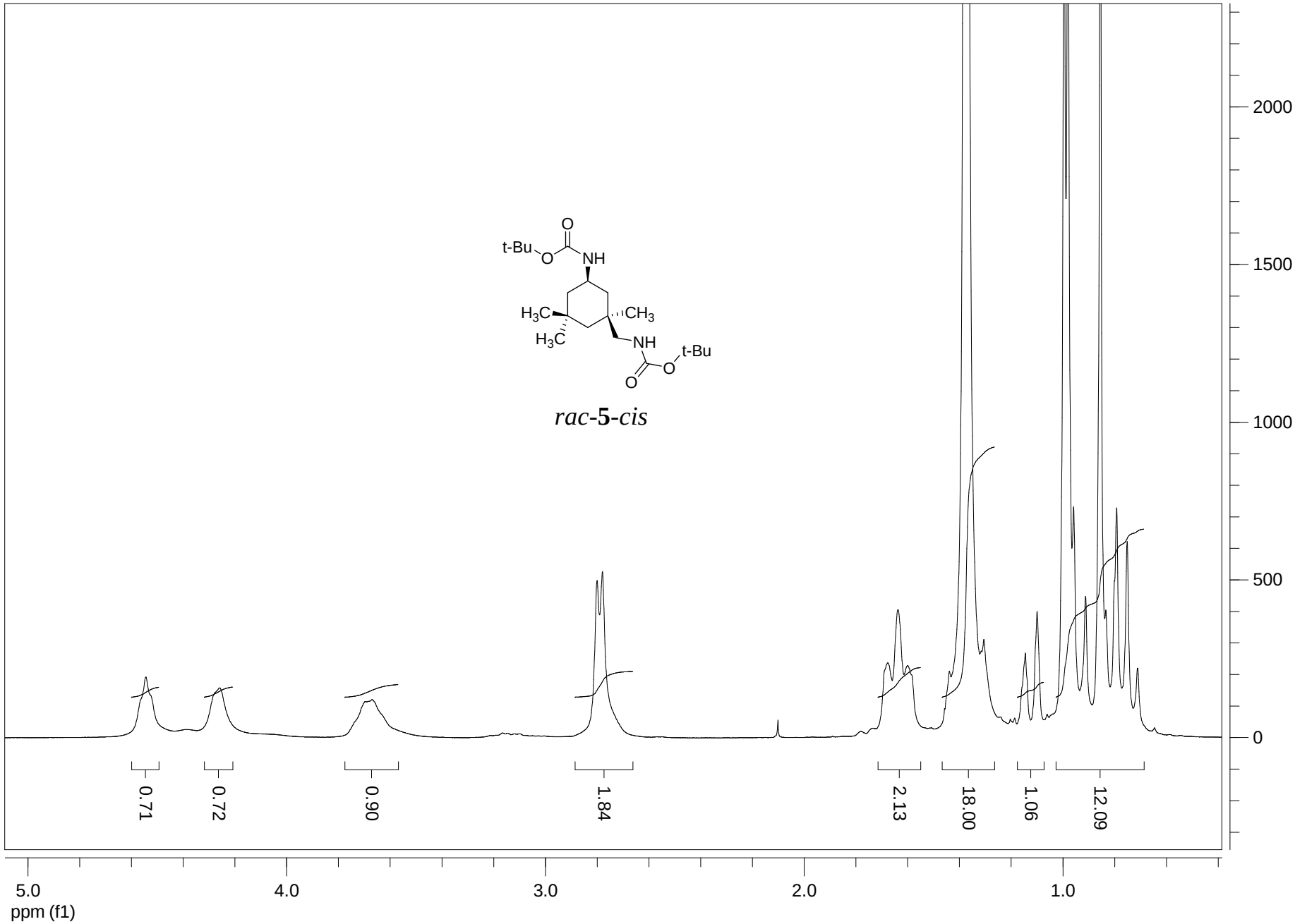
*rac*-**4**-*cis*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CH3
H3C
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t-Bu
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O
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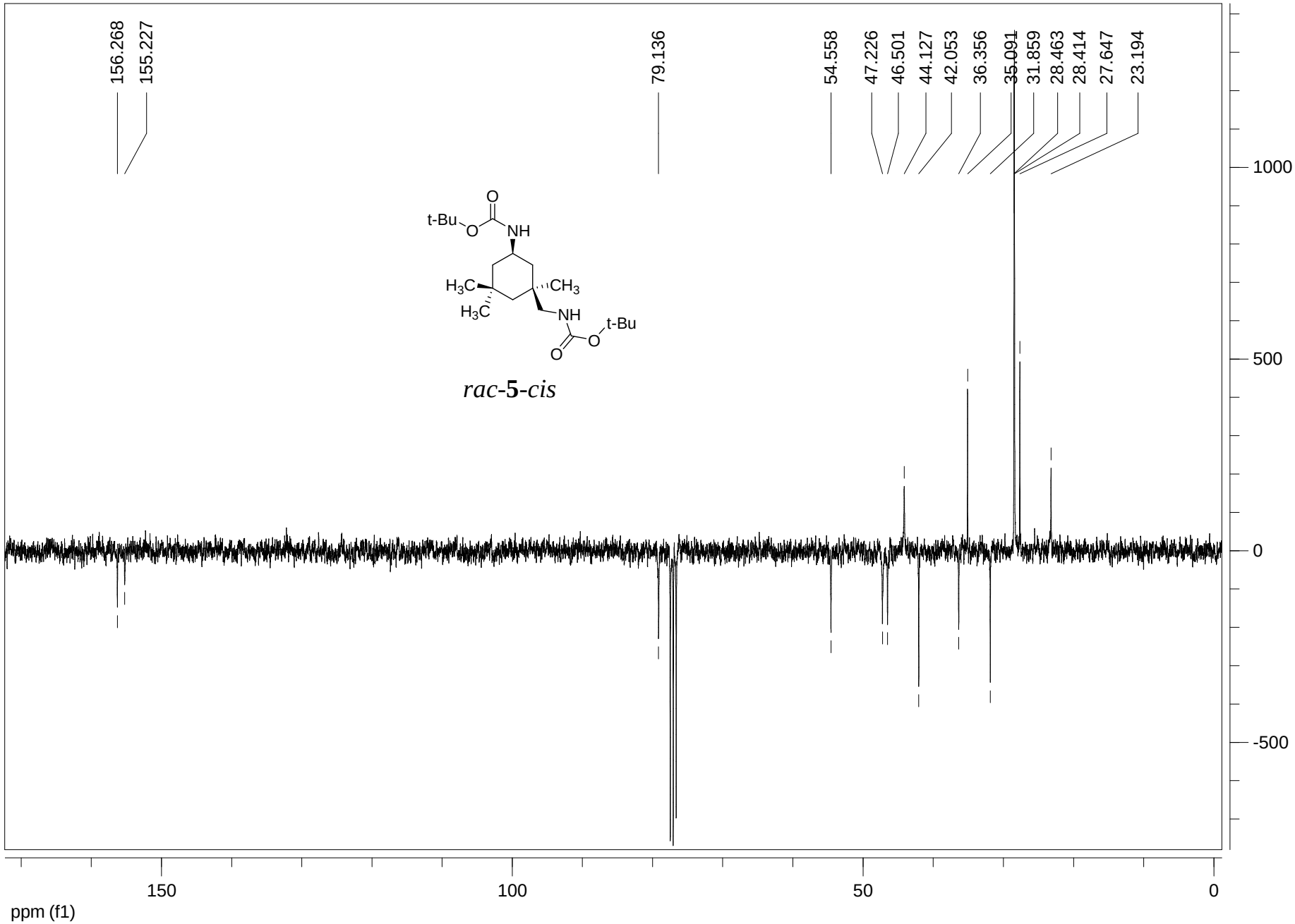
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H3C
CH3
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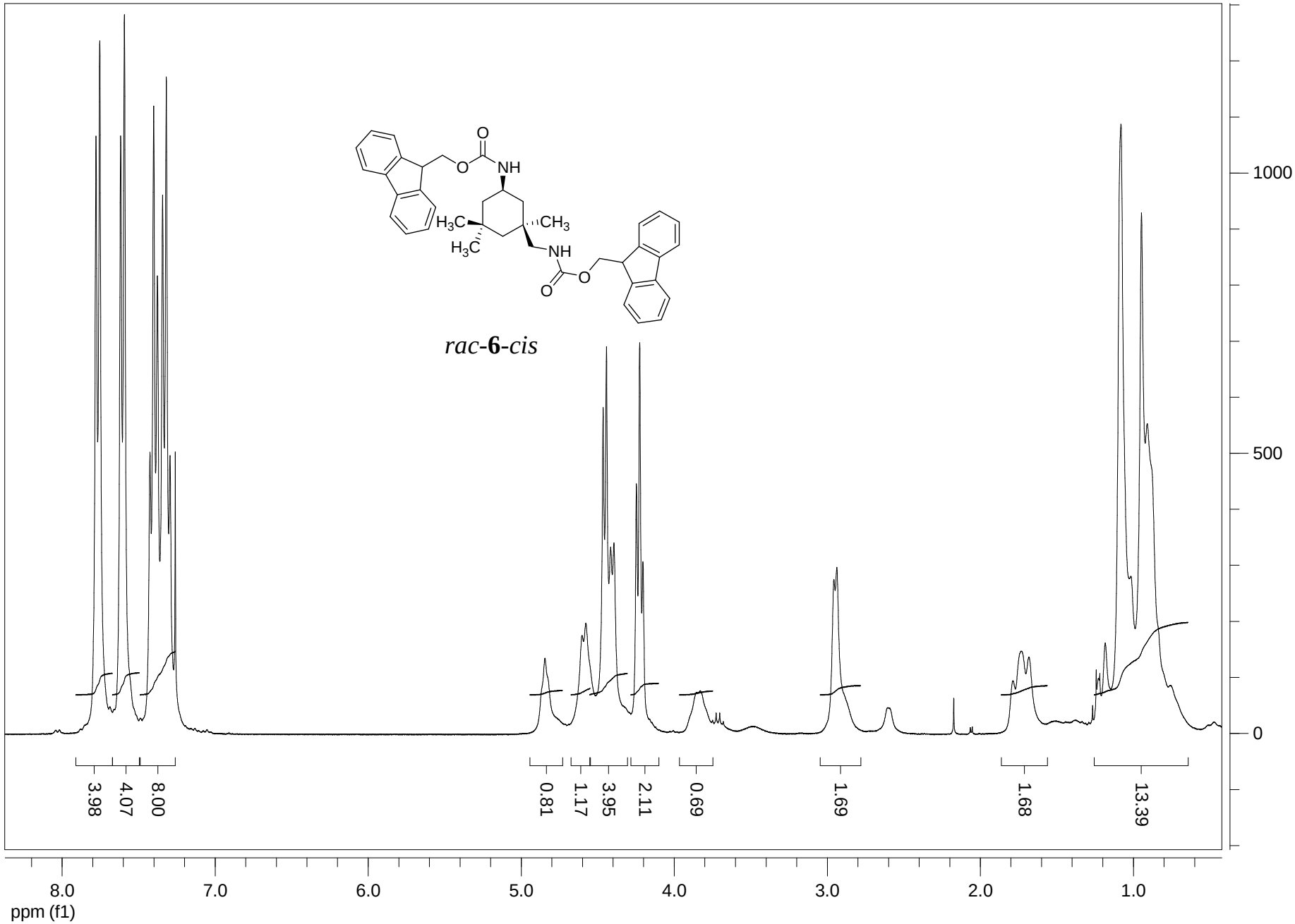
-500
150
100
50
0
ppm (f1)

*rac*-**5**-*cis*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NH
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3.95
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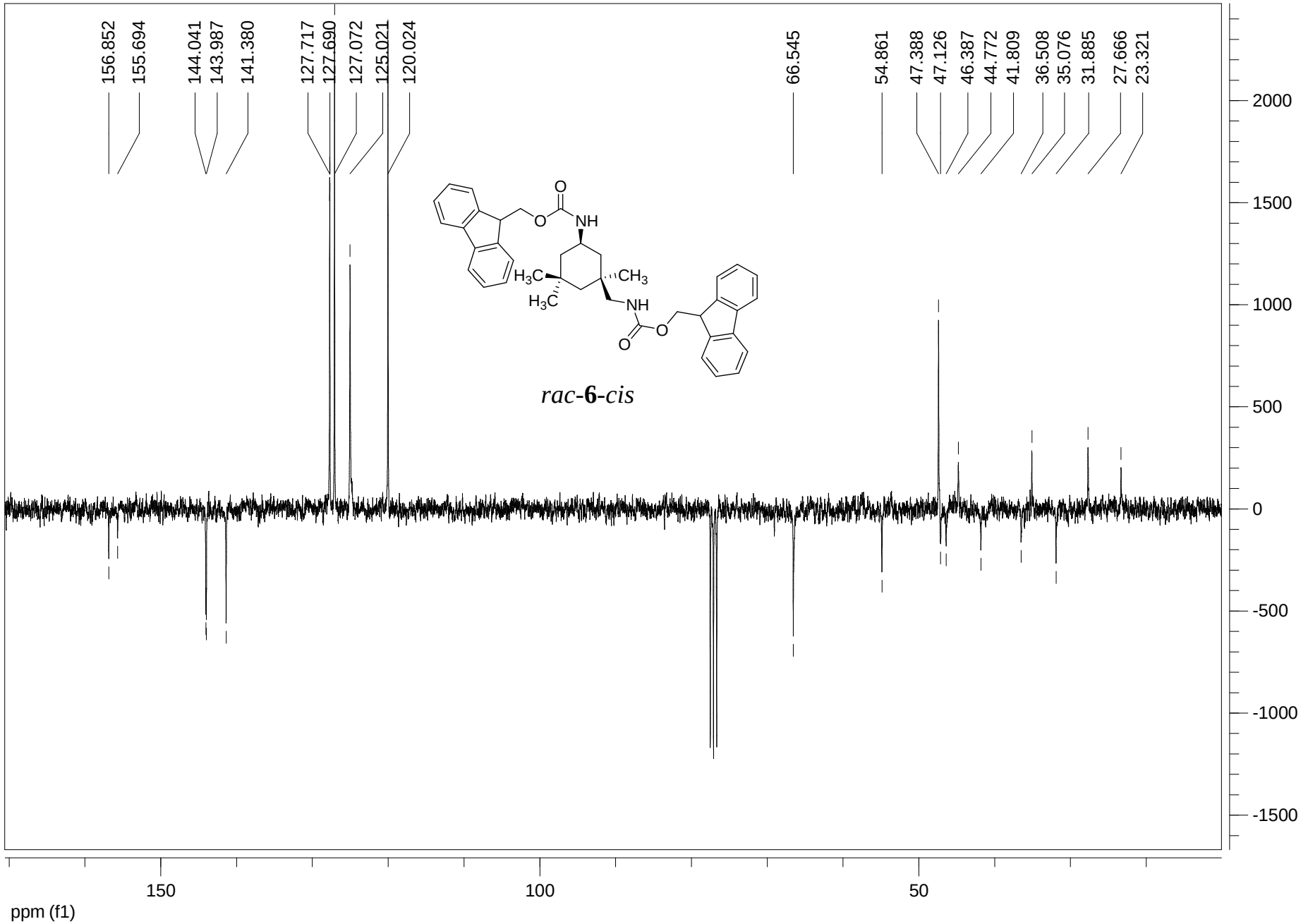
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27.666
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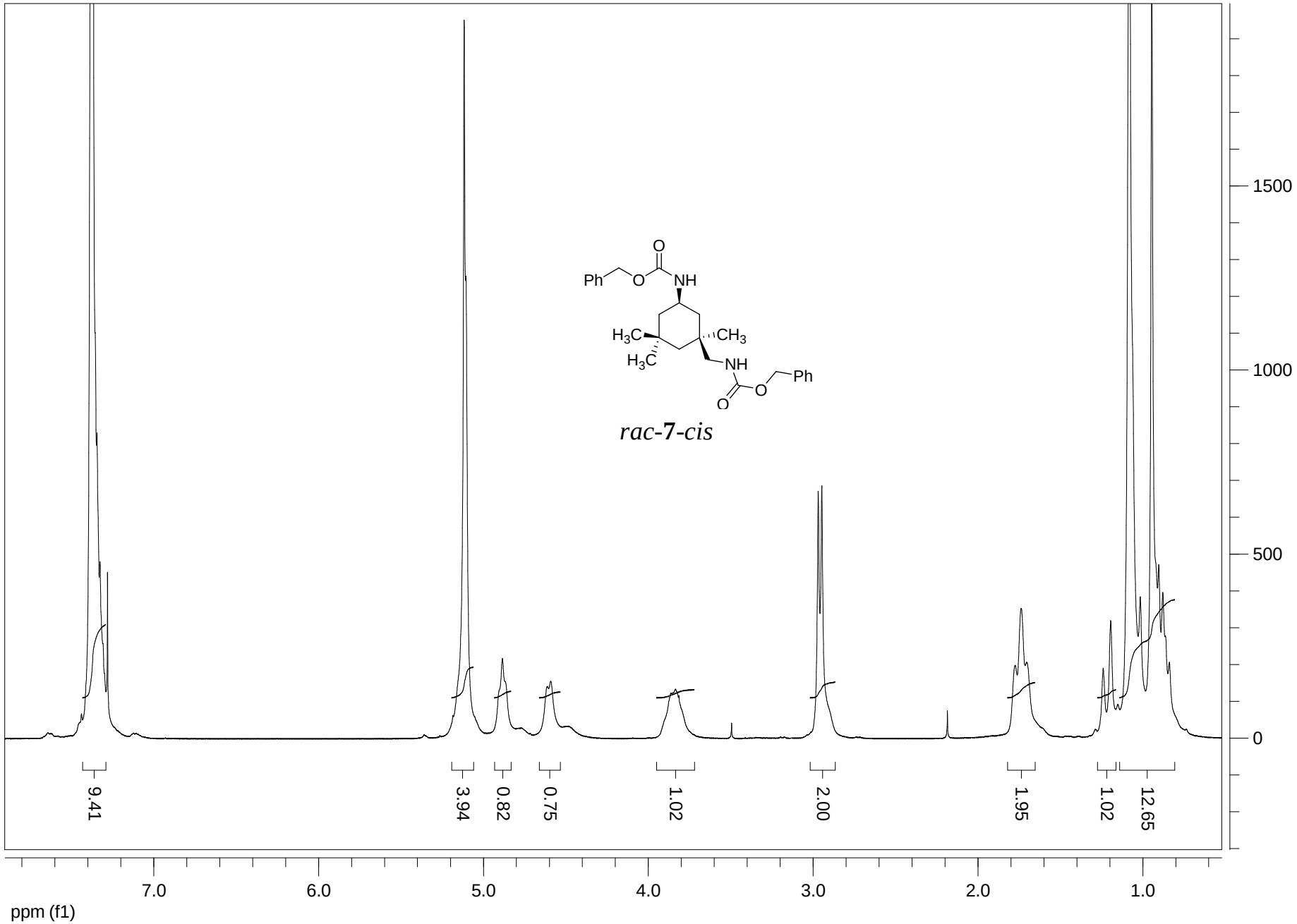
50
ppm (f1)

*rac*-**6**-*cis*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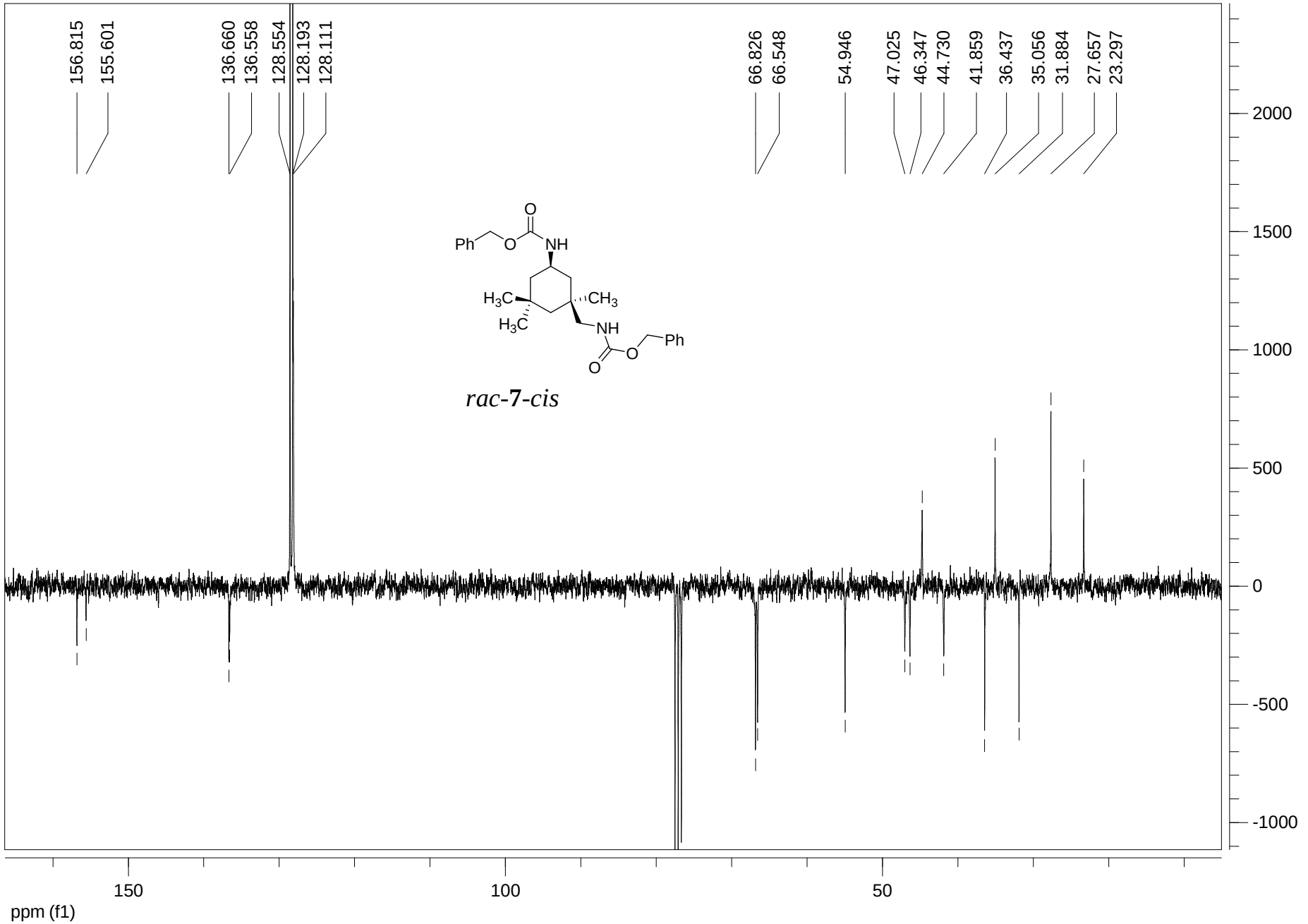
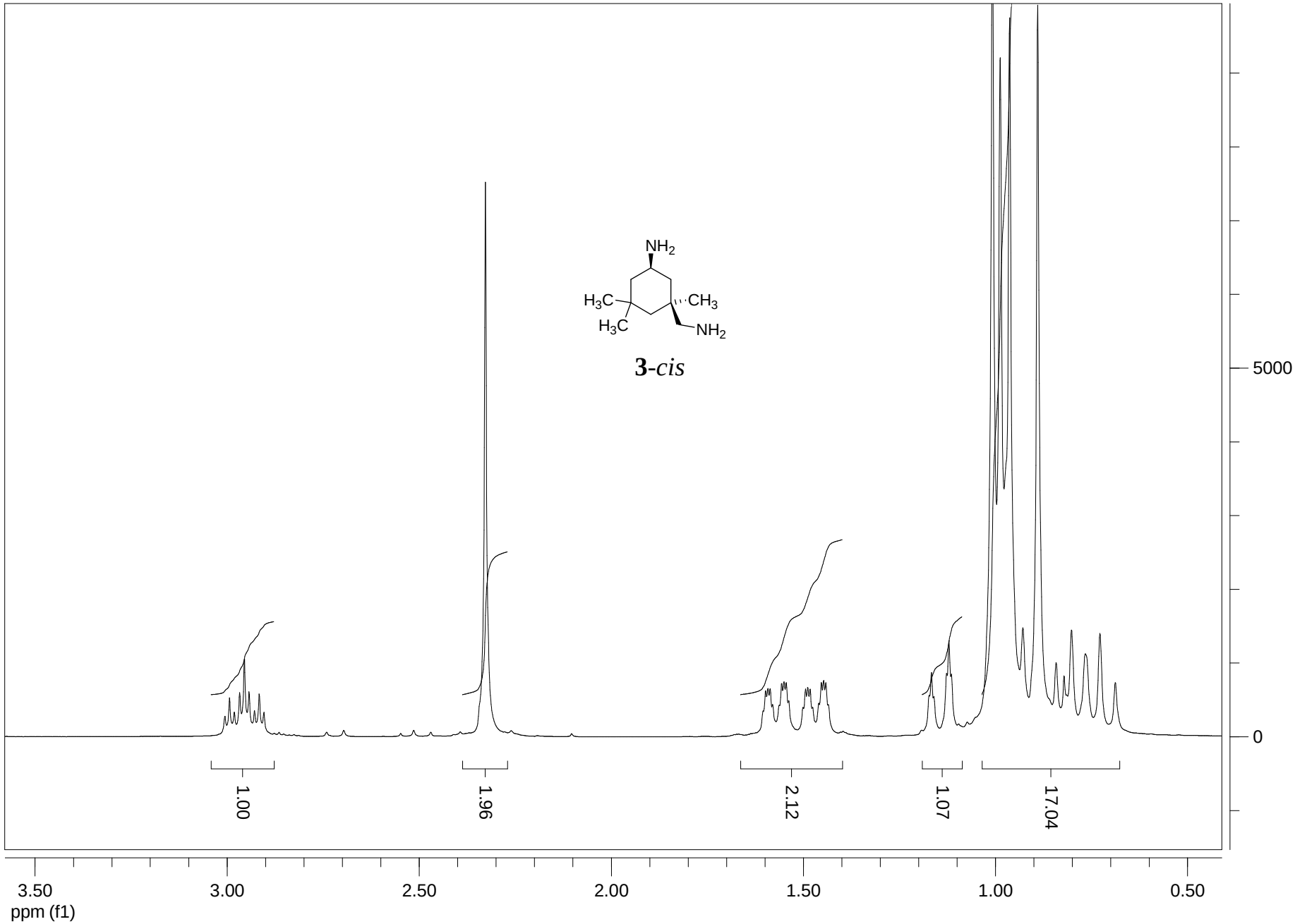
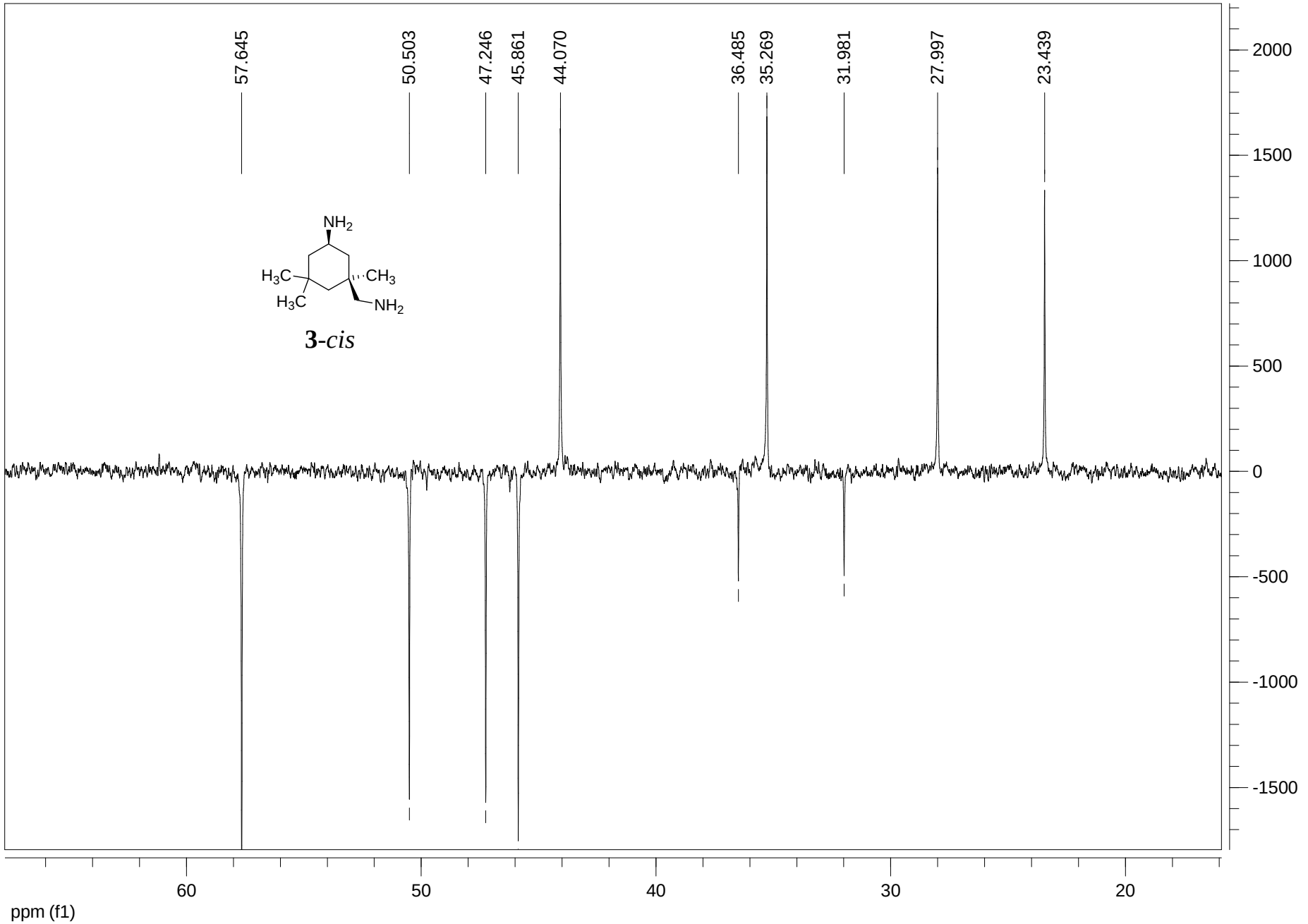
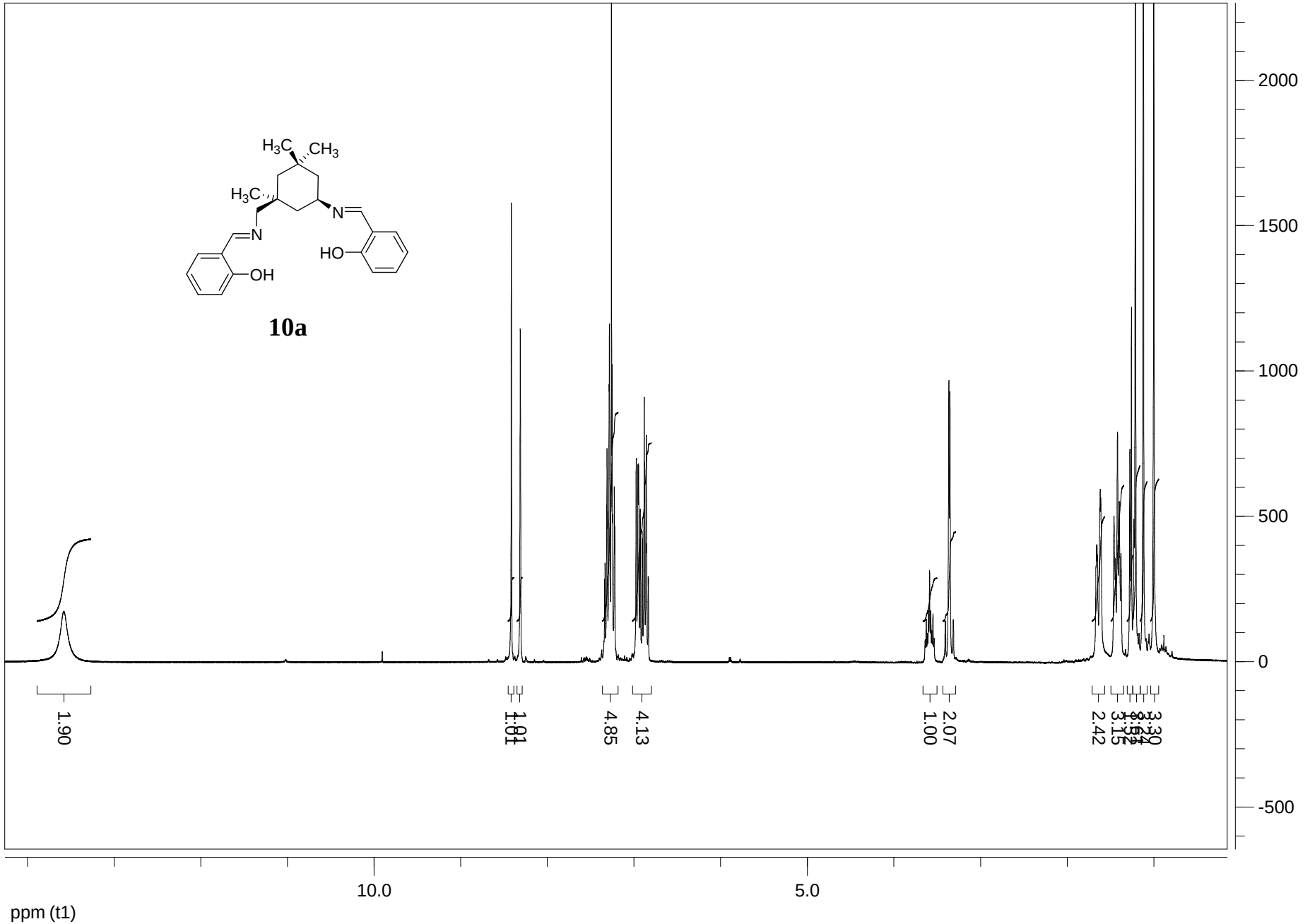
1.0
ppm (f1)

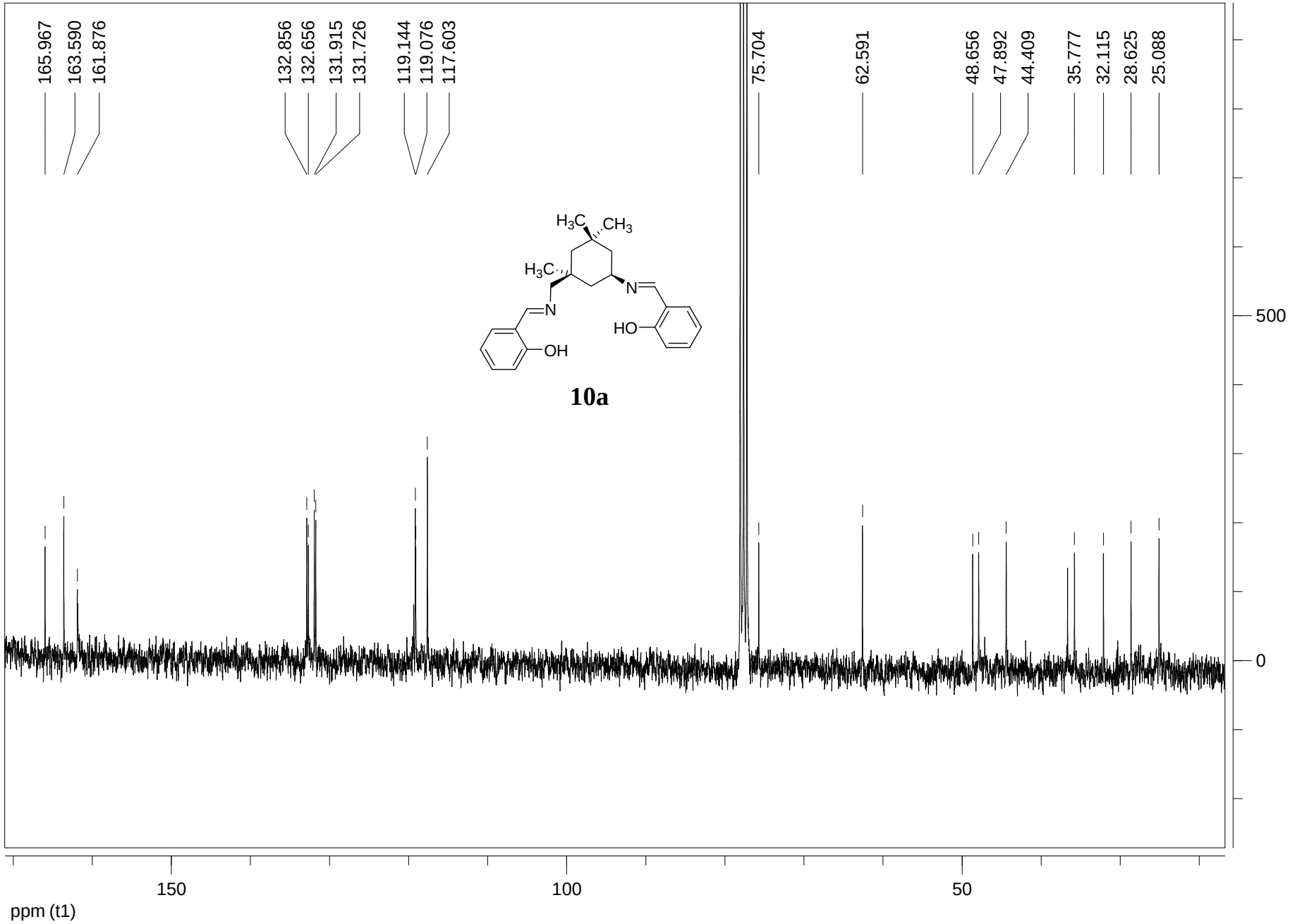

156.815
155.601
136.660
136.558
128.554
128.193
128.111
66.826
66.548
54.946
47.025
46.347
44.730
41.859
36.437
35.056
31.884
27.657
23.297
2000
1500
1000
500
0
-500
-1000
150
100
50
ppm (f1)

*rac*-**7**-*cis*

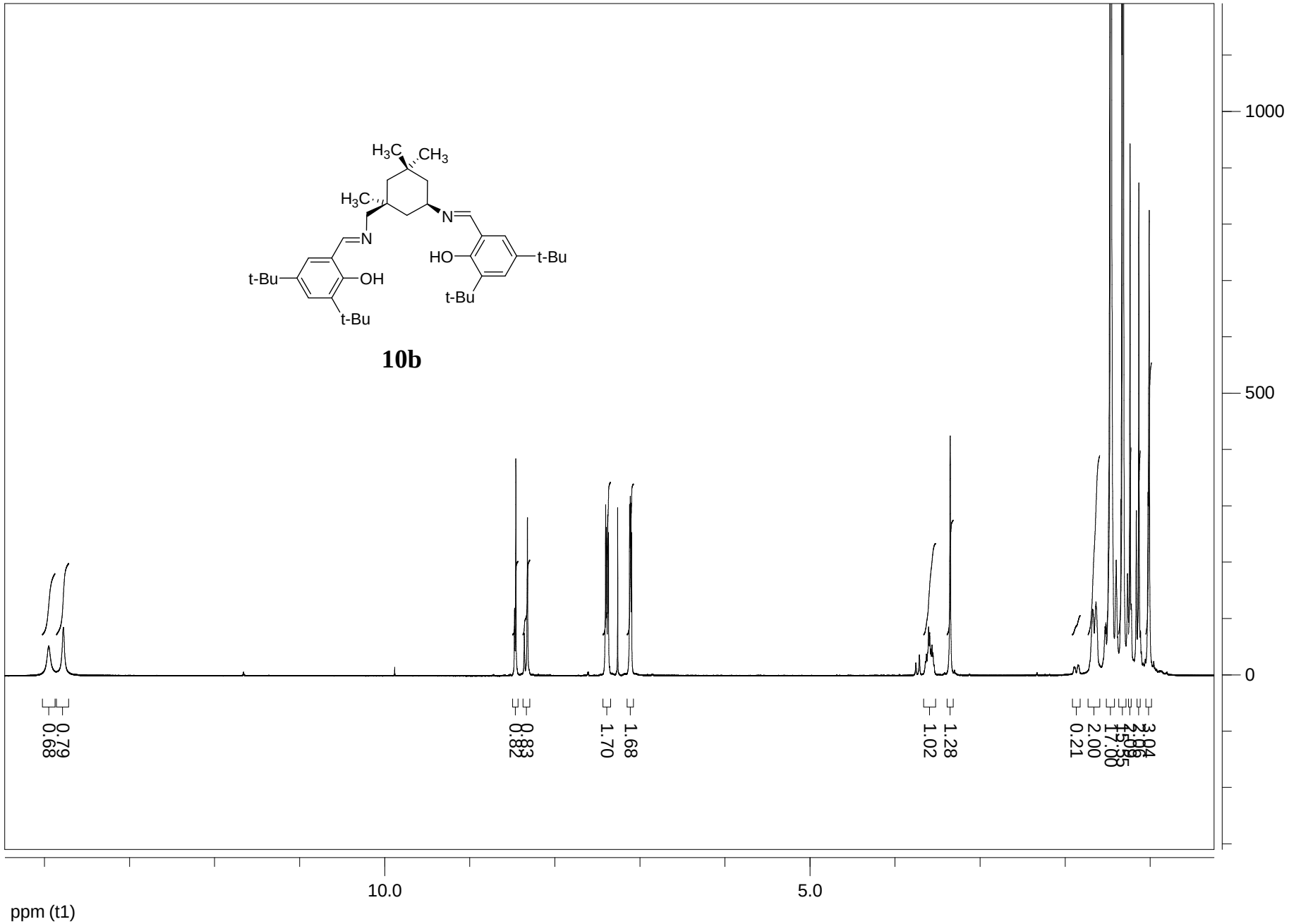

NH2
H3C
CH3
H3C
NH2
3-cis
5000
0
1.00
1.96
2.12
1.07
17.04
3.50
3.00
2.50
2.00
1.50
1.00
0.50
ppm (f1)

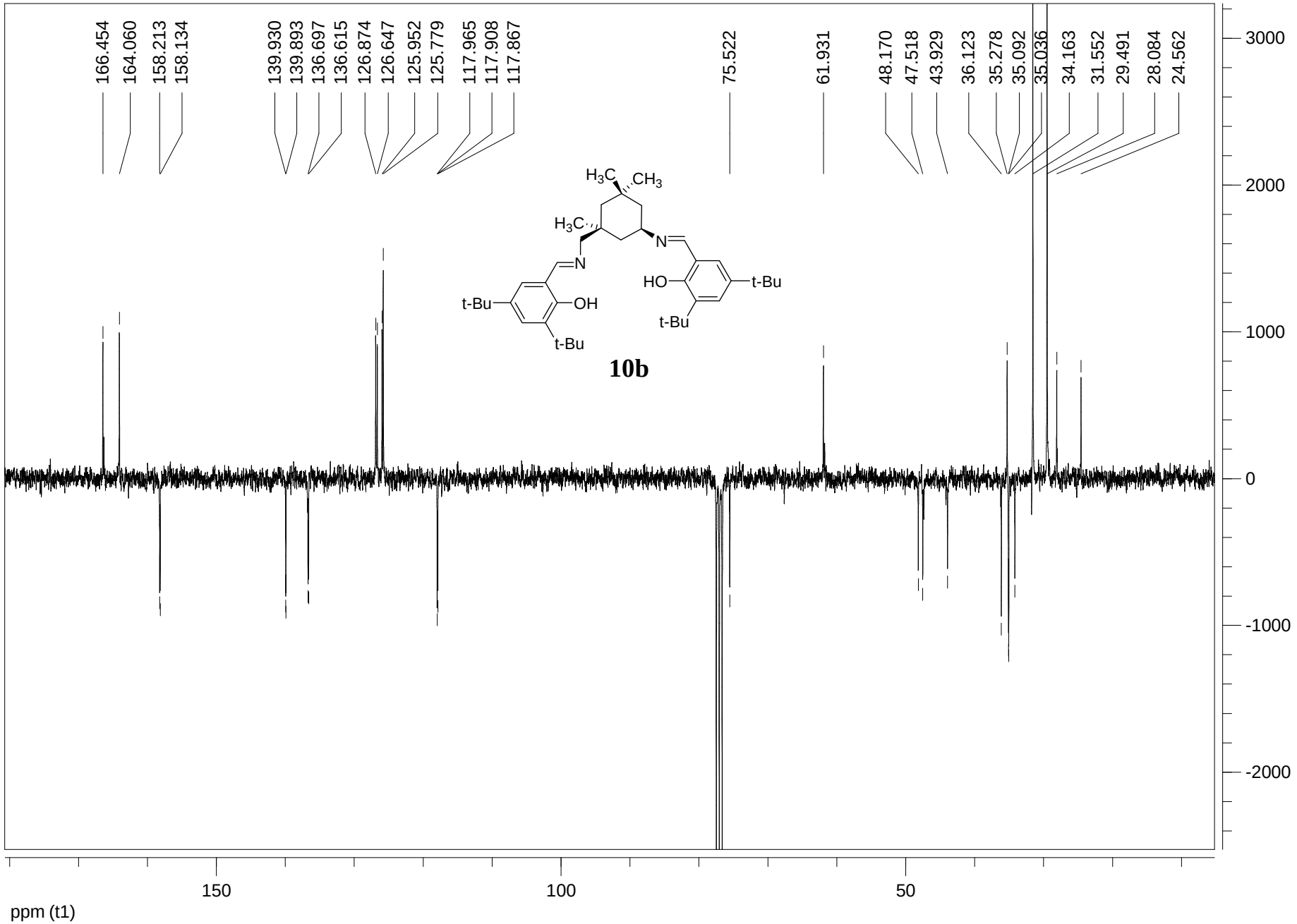

57.645
50.503
47.246
45.861
44.070
36.485
35.269
31.981
27.997
23.439
NH2
H3C
CH3
H3C
NH2
3-cis
2000
1500
1000
500
0
-500
-1000
-1500
60
50
40
30
20
ppm (f1)

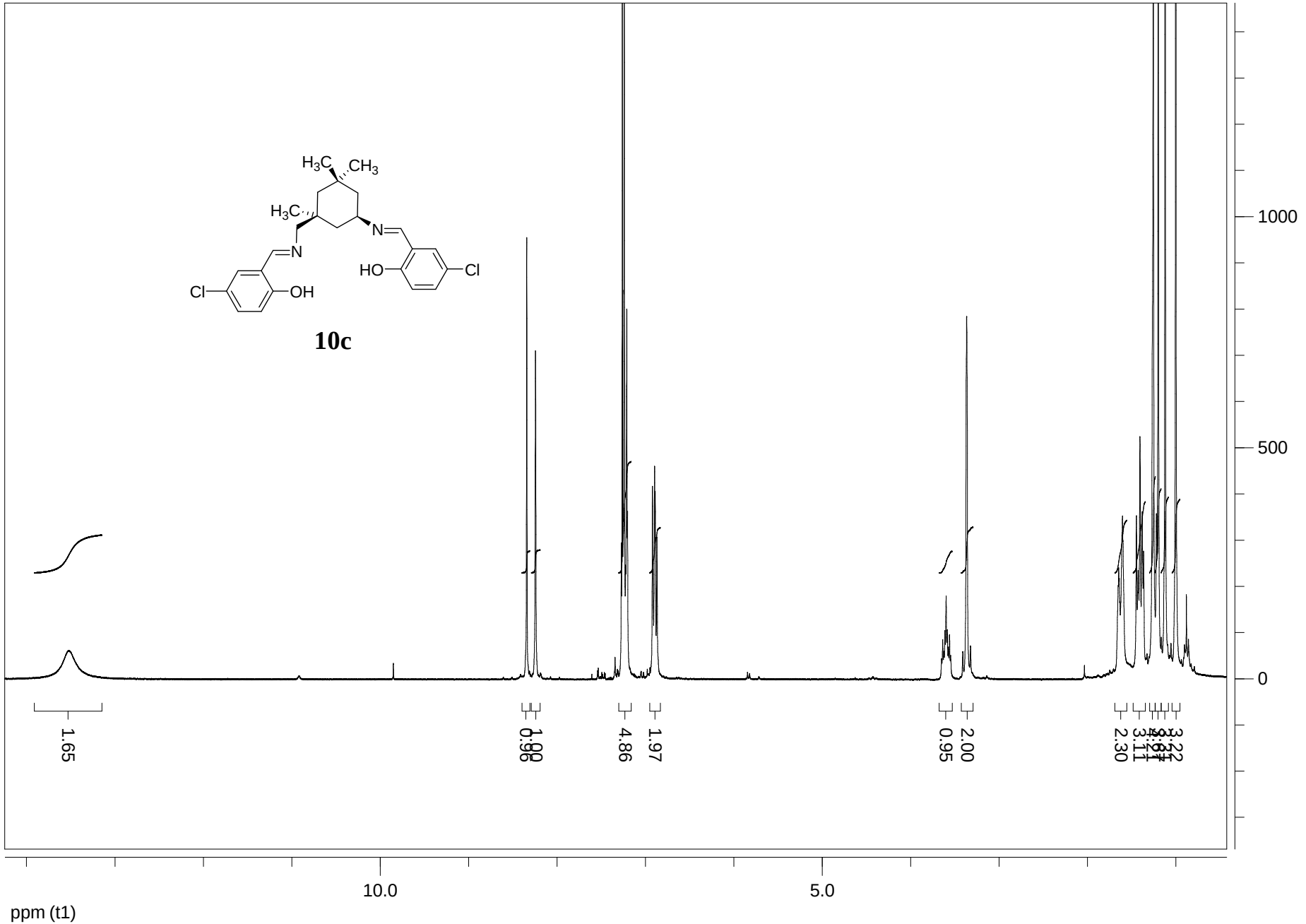

H3C
CH3
H3C
N
N
HO
OH
10a
2000
1500
1000
500
0
-500
1.90
1.01
1.01
4.85
4.13
1.00
2.07
2.42
3.15
3.30
10.0
5.0
ppm (t1)

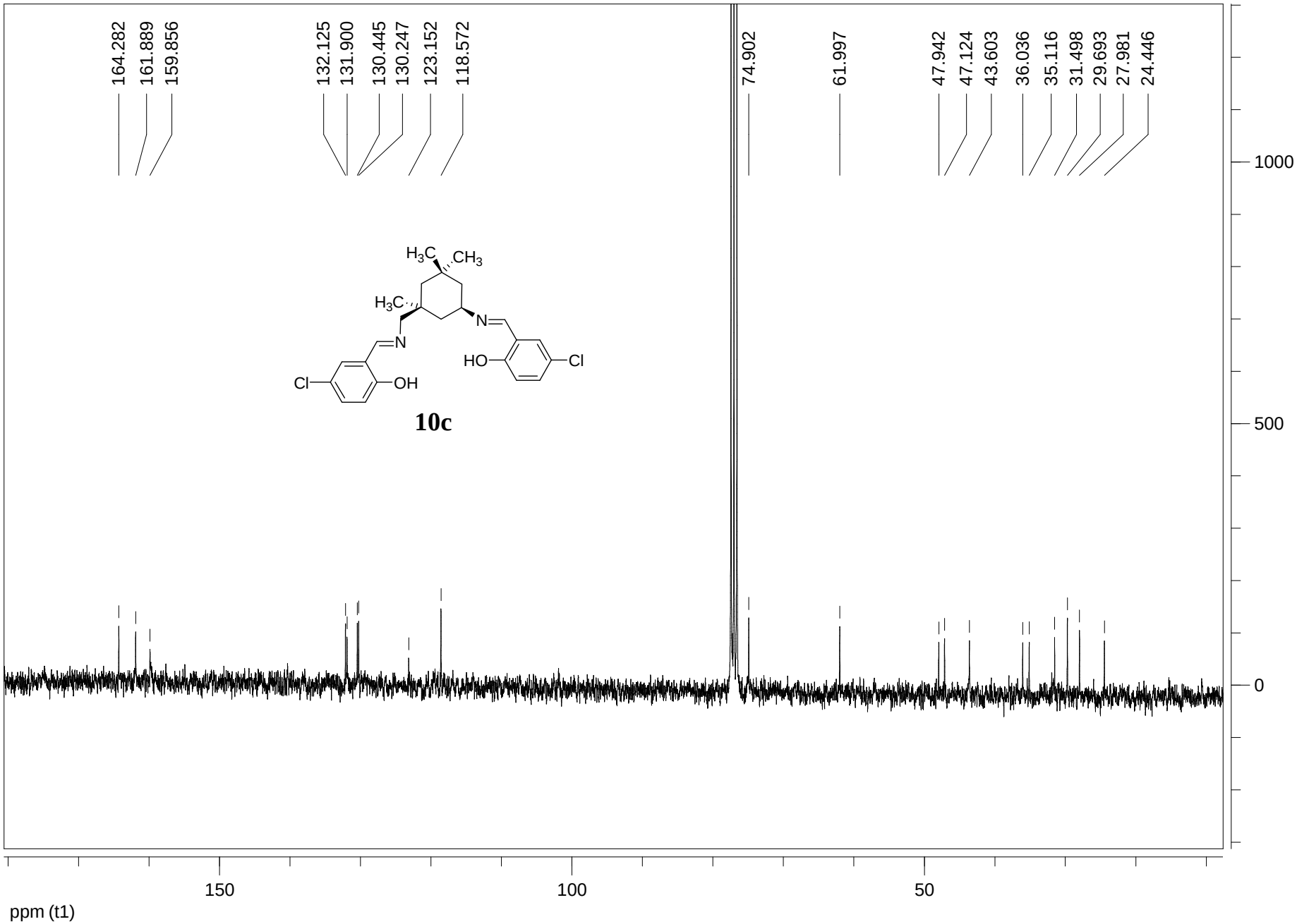

165.967
163.590
161.876
132.856
132.656
131.915
131.726
119.144
119.076
117.603
75.704
62.591
48.656
47.892
44.409
35.777
32.115
28.625
25.088
H3C
CH3
H3C
N
N
HO
OH
500
0
150
100
50
ppm (t1)

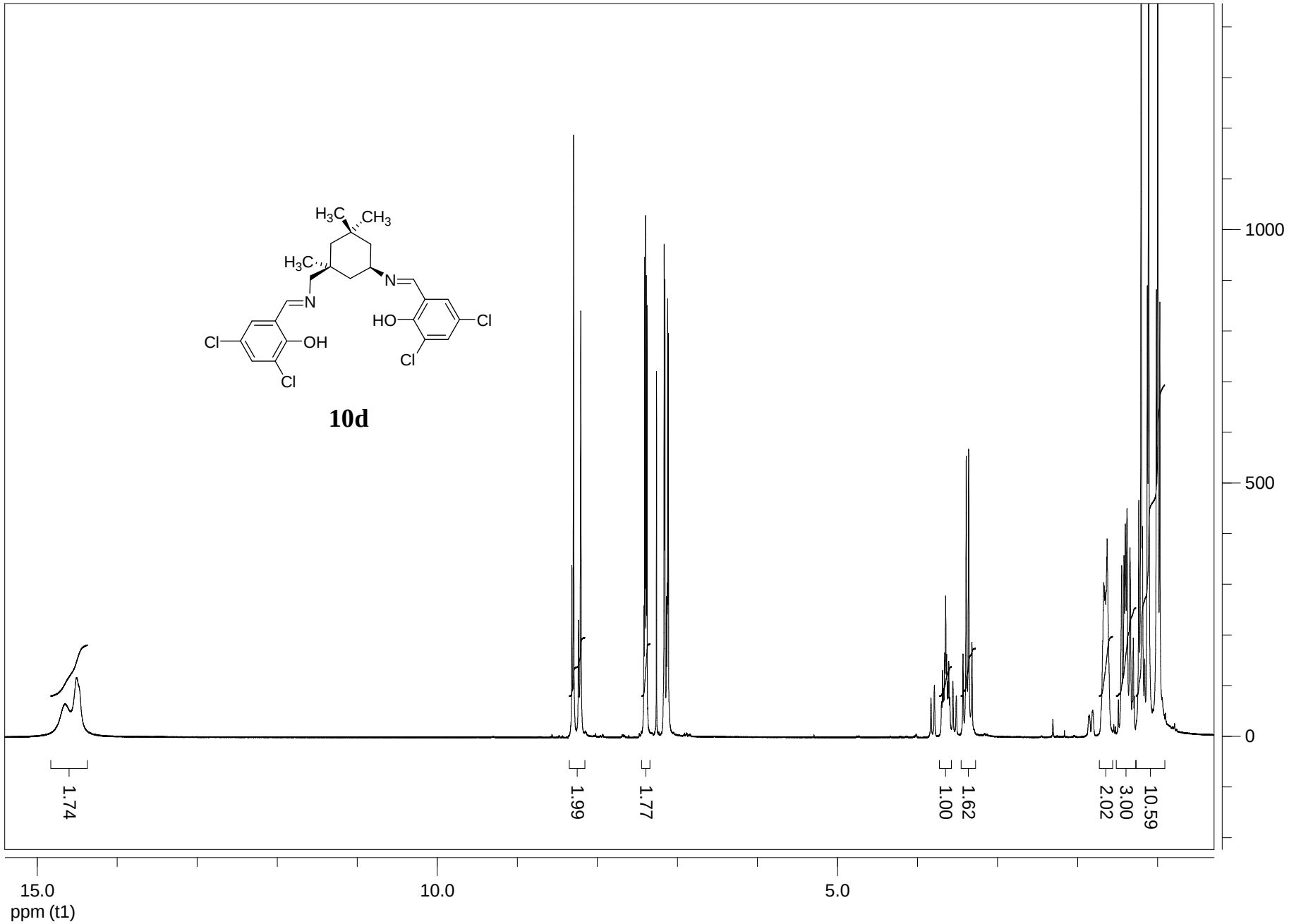
**10a**


H3C
CH3
H3C
N
N
HO
t-Bu
t-Bu
OH
t-Bu
t-Bu
10b
1000
500
0
0.68
0.79
0.82
0.83
1.70
1.68
1.02
1.28
0.21
2.00
17.00
10.0
5.0
ppm (t1)

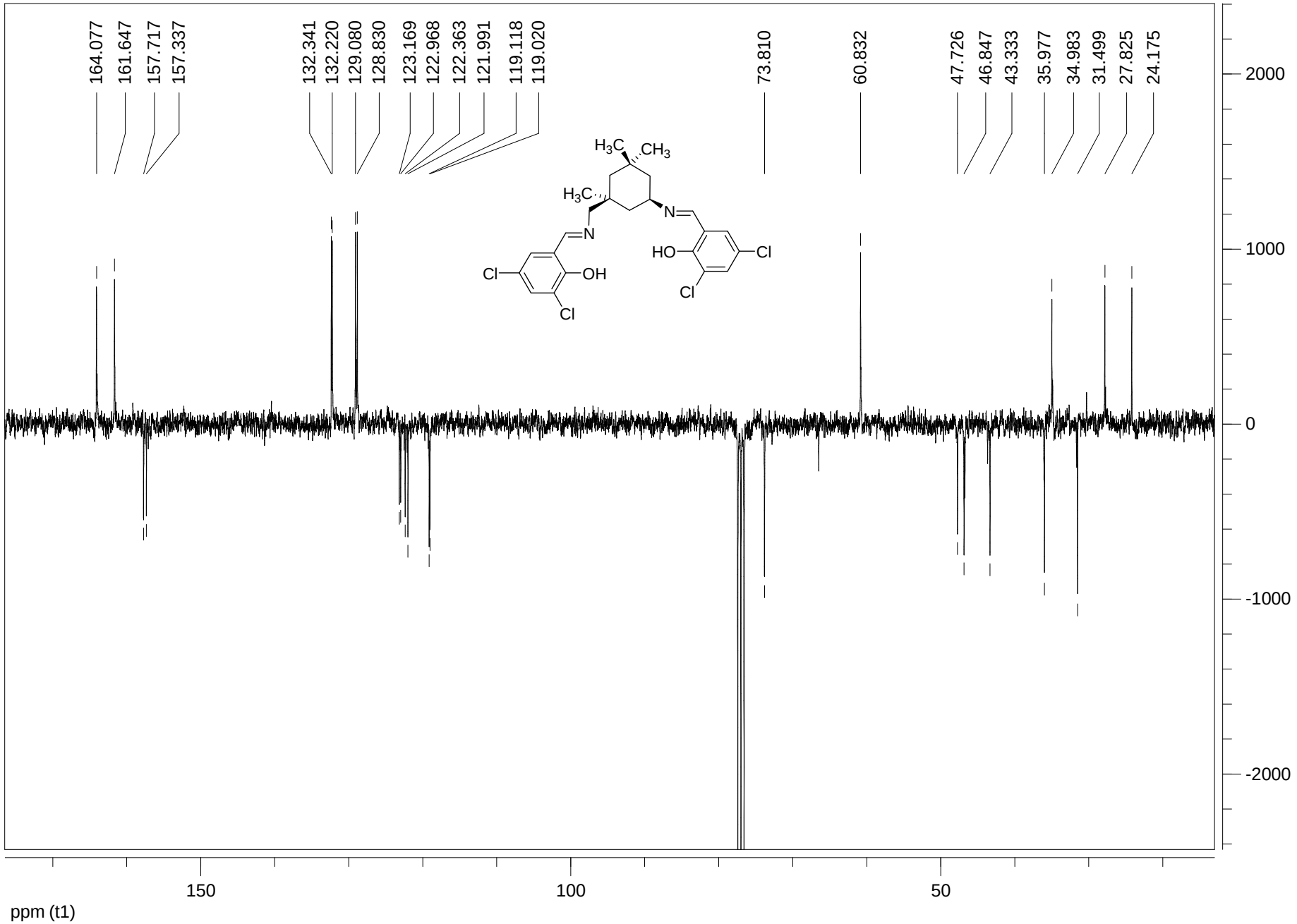

166.454
164.060
158.213
158.134
139.930
139.893
136.697
136.615
126.874
126.647
125.952
125.779
117.965
117.908
117.867
75.522
61.931
48.170
47.518
43.929
36.123
35.278
35.092
35.036
34.163
31.552
29.491
28.084
24.562
H3C
CH3
H3C
N
N
HO
t-Bu
t-Bu
t-Bu
OH
t-Bu
10b
3000
2000
1000
0
-1000
-2000
150
100
50
ppm (t1)

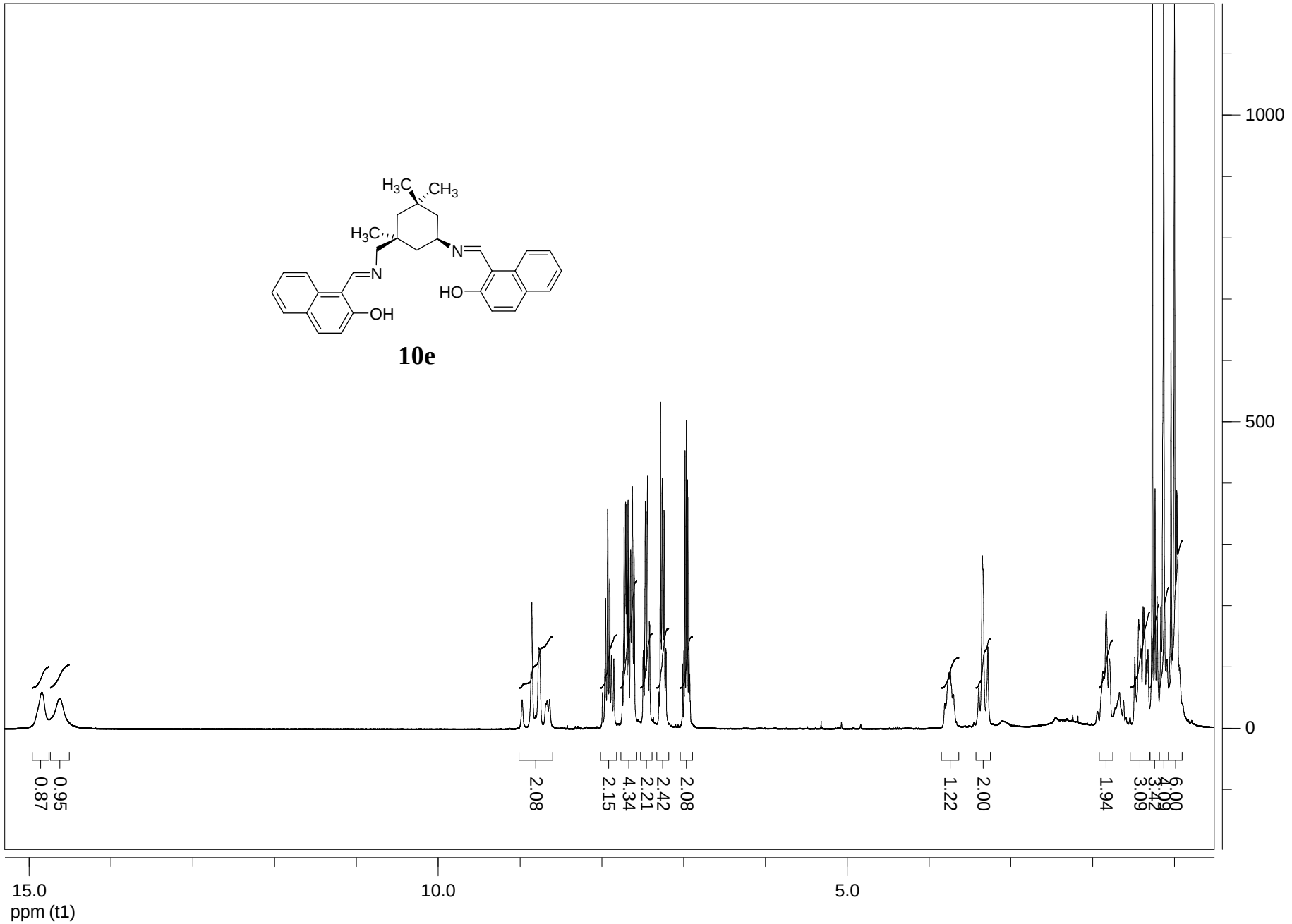

H3C
CH3
H3C
N
N
HO
Cl
Cl
OH
10c
1000
500
0
1.65
0.98
1.00
4.86
1.97
0.95
2.00
2.30
3.11
3.22
10.0
5.0
ppm (t1)

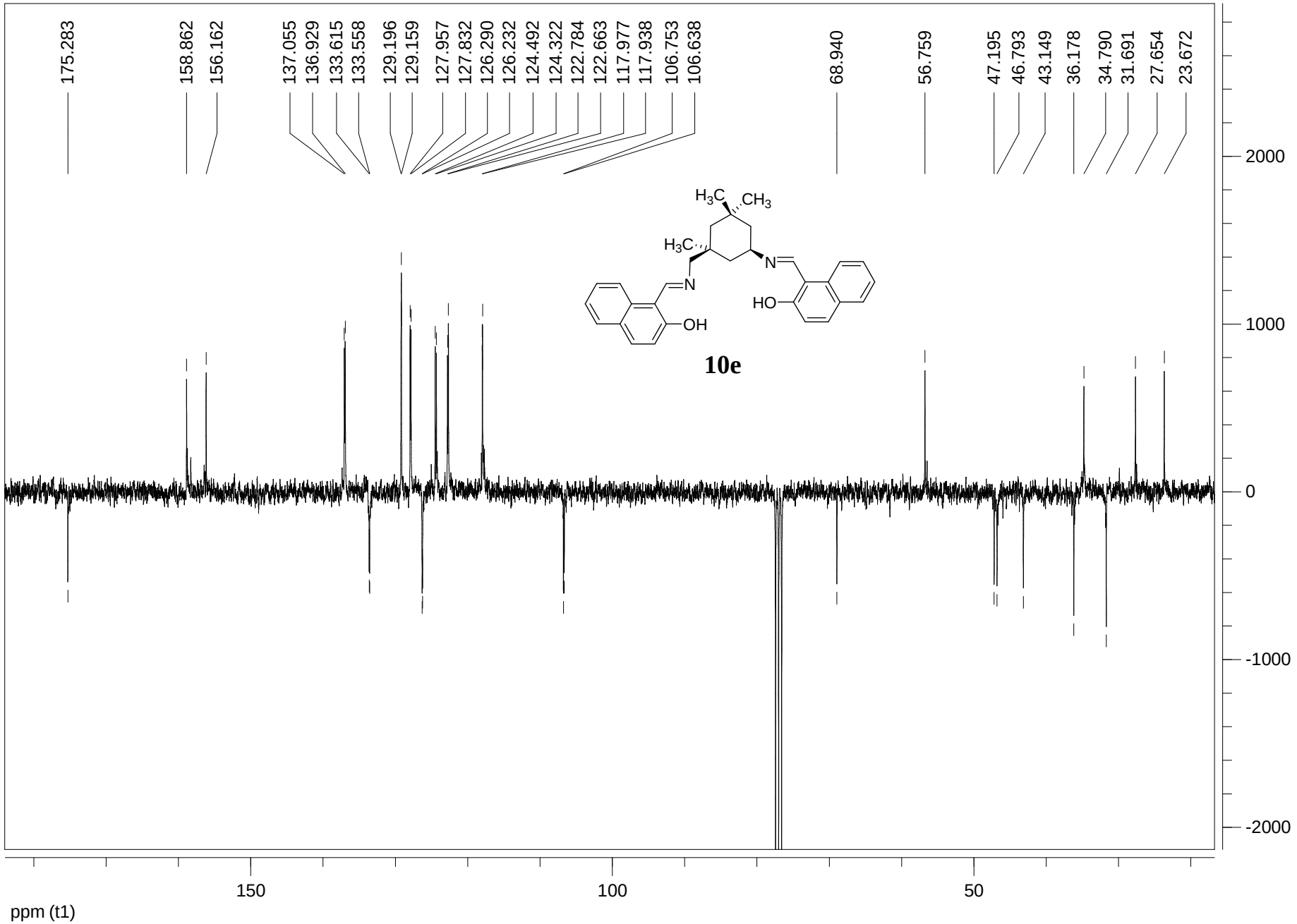

164.282
161.889
159.856
132.125
131.900
130.445
130.247
123.152
118.572
74.902
61.997
47.942
47.124
43.603
36.036
35.116
31.498
29.693
27.981
24.446
H3C
CH3
H3C
N
N
HO
Cl
Cl
OH
10c
1000
500
0
150
100
50
ppm (t1)


H3C
CH3
H3C
N
N
HO
Cl
Cl
Cl
OH
Cl
1000
500
0
1.74
1.99
1.77
1.00
1.62
2.02
3.00
10.59
15.0
ppm (t1)
10.0
5.0

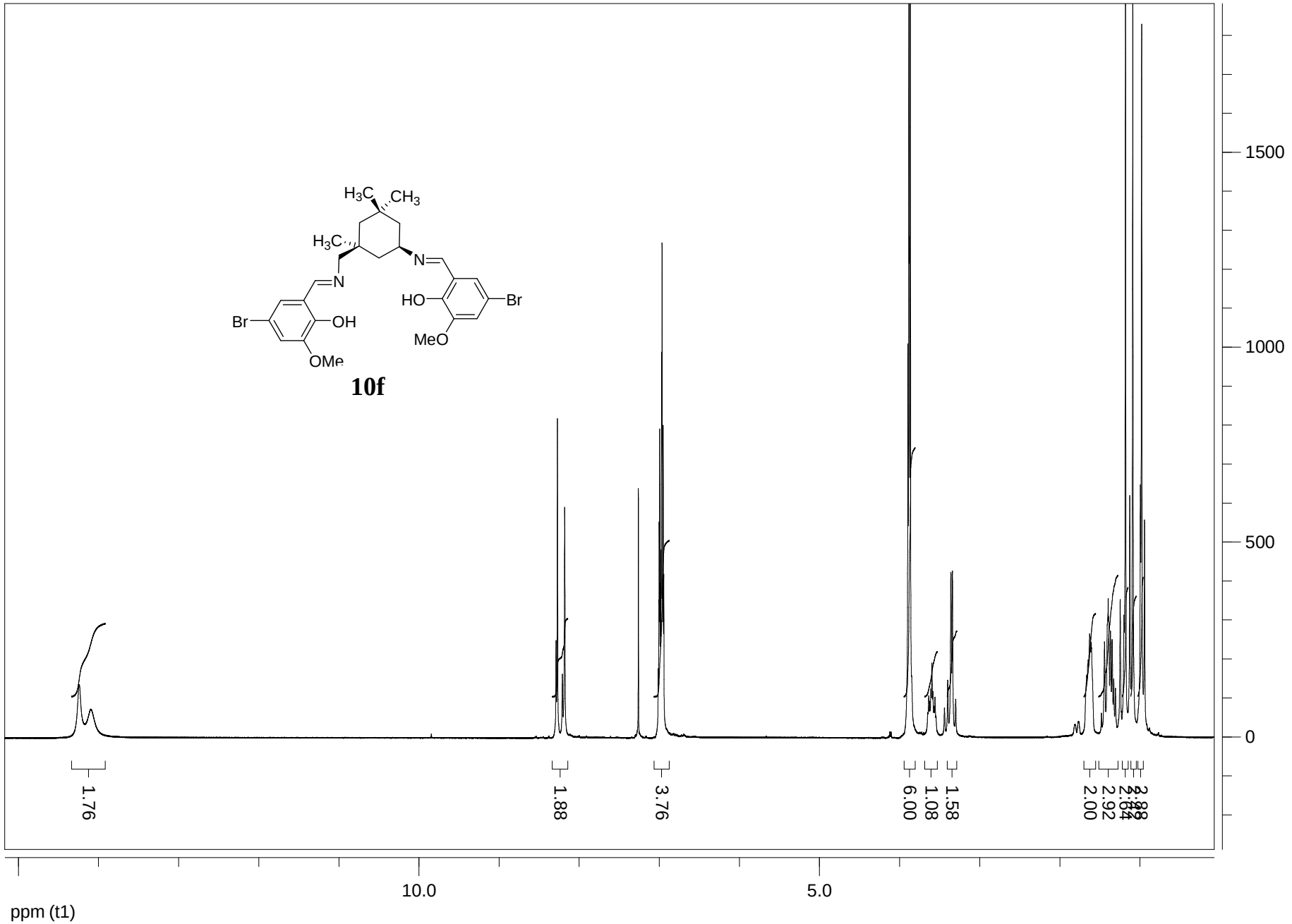
**10d**

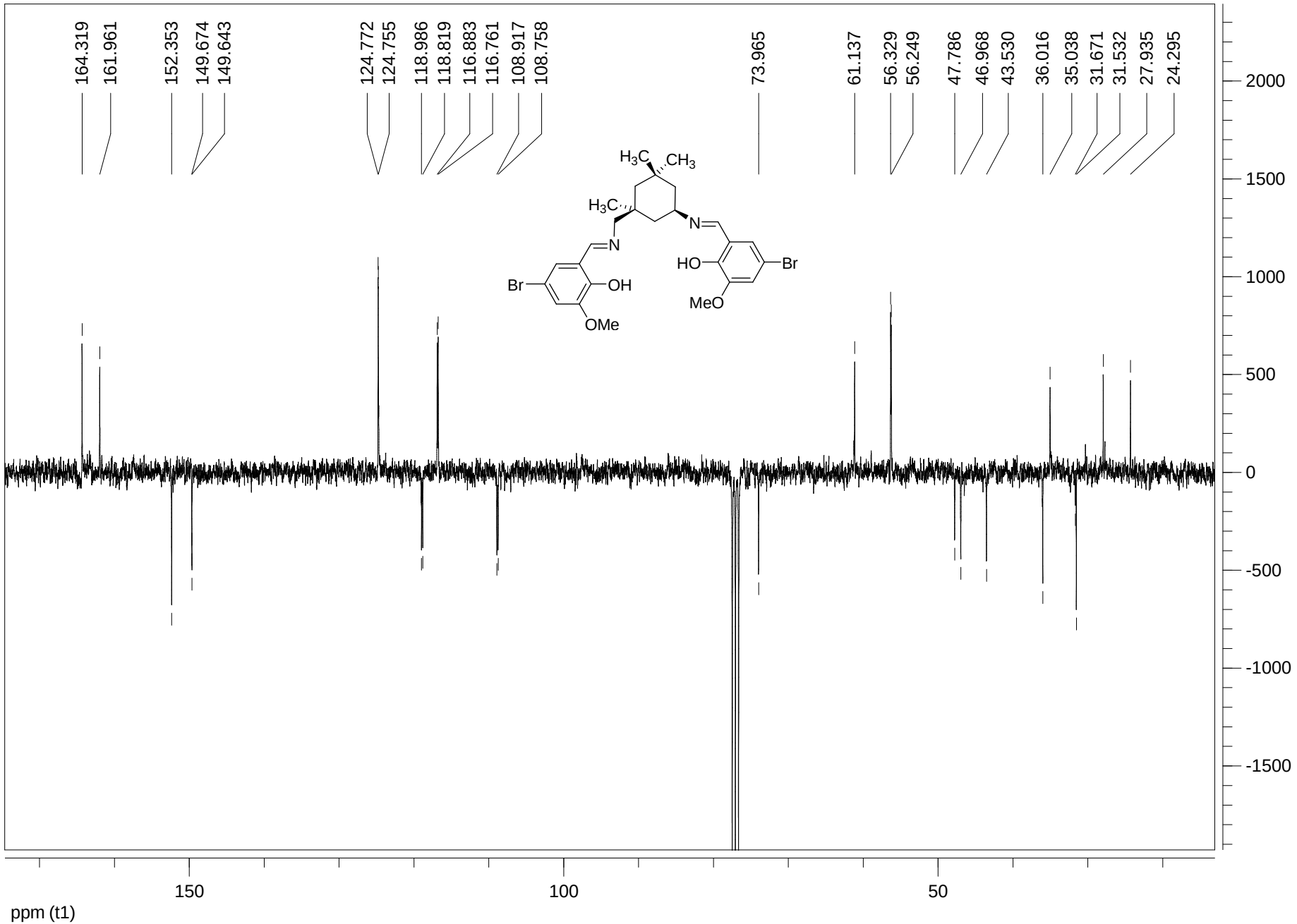

164.077
161.647
157.717
157.337
132.341
132.220
129.080
128.830
123.169
122.968
122.363
121.991
119.118
119.020
73.810
60.832
47.726
46.847
43.333
35.977
34.983
31.499
27.825
24.175
H3C
CH3
H3C
N
N
HO
Cl
Cl
Cl
OH
Cl
2000
1000
0
-1000
-2000
150
100
50
ppm (t1)

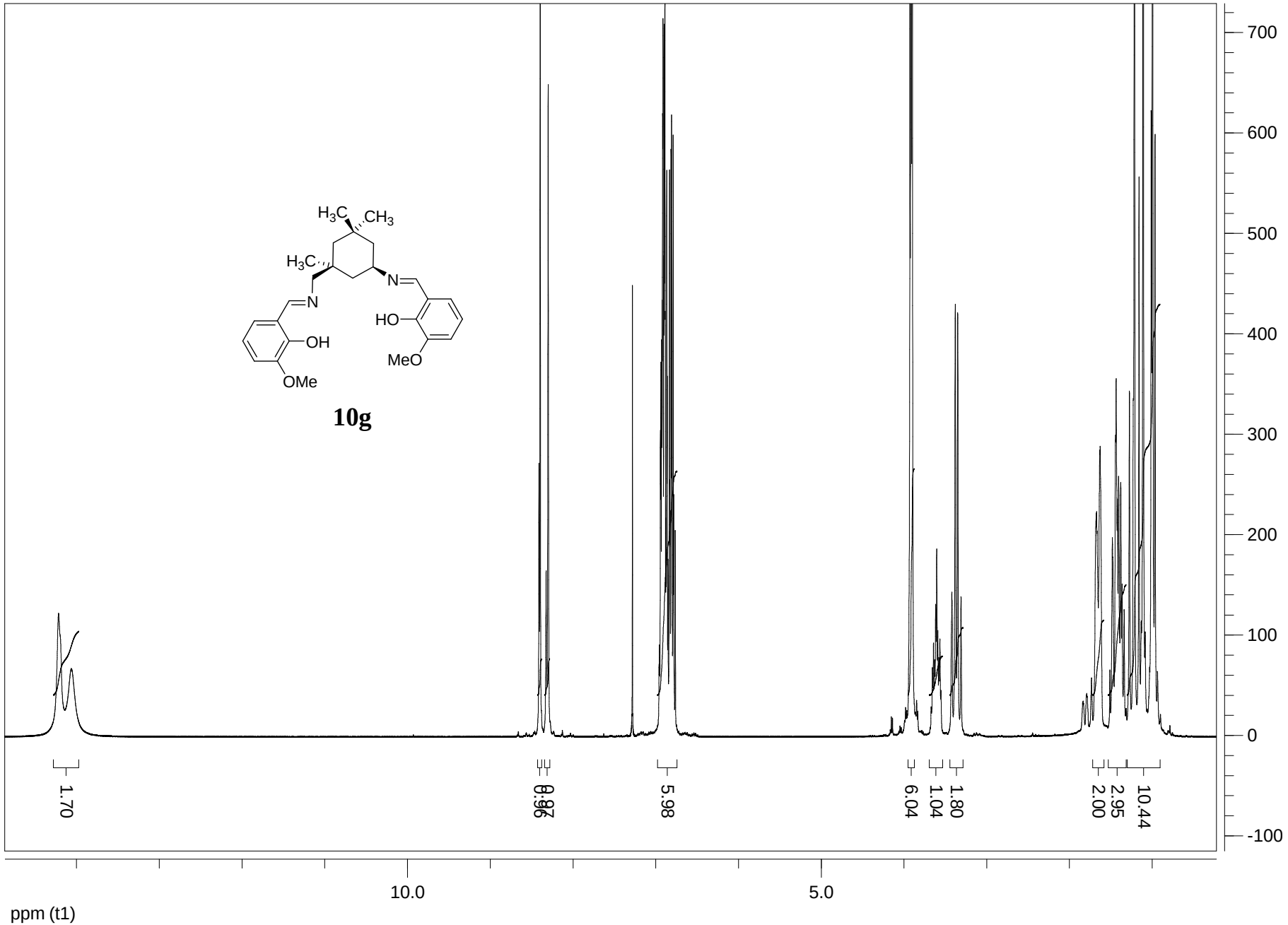

H3C
CH3
H3C
N
N
HO
OH
10e
1000
500
0
0.87
0.95
2.08
2.15
4.34
2.21
2.42
2.08
1.22
2.00
1.94
3.09
15.0
ppm (t1)
10.0
5.0

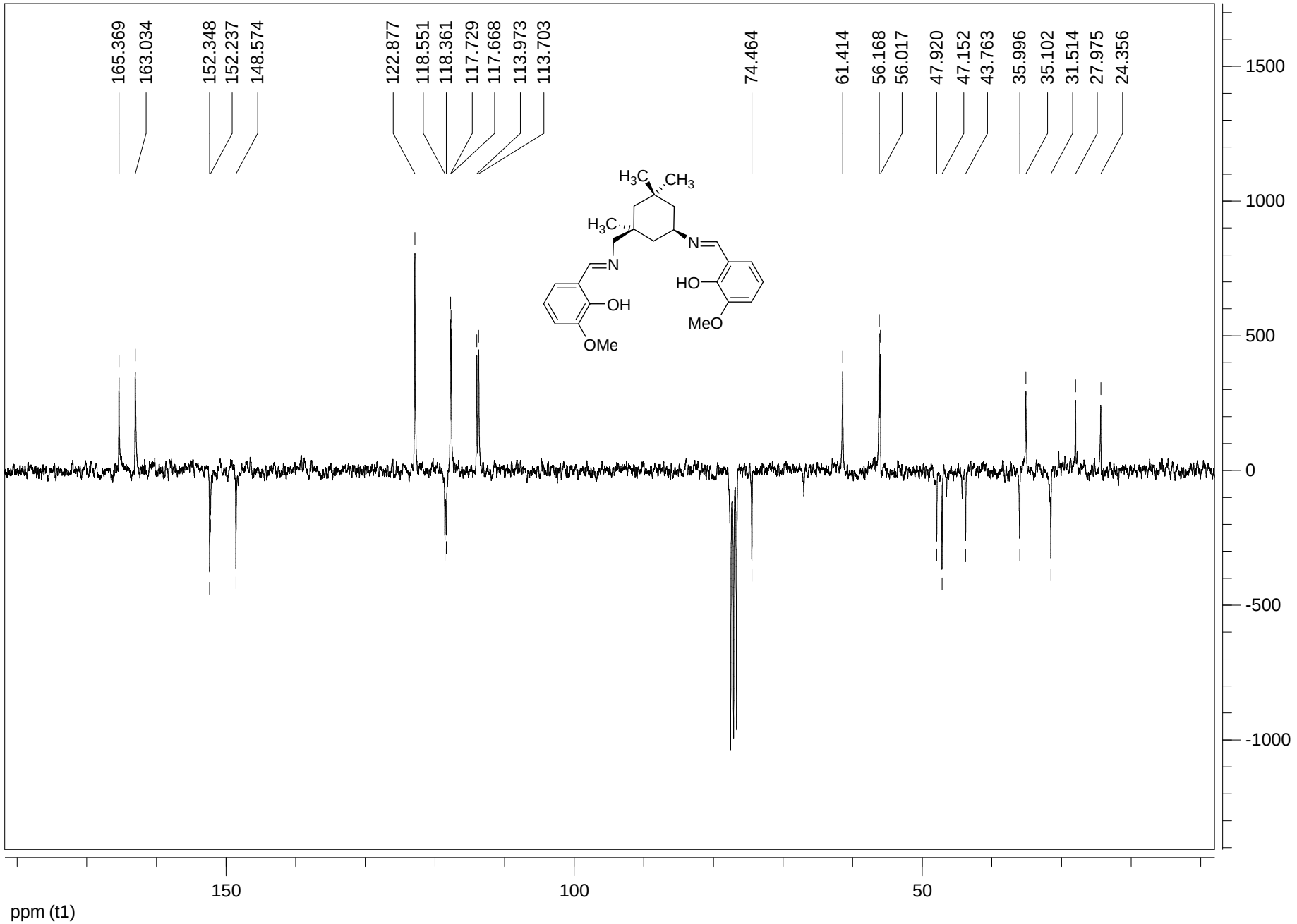

175.283
158.862
156.162
137.055
136.929
133.615
133.558
129.196
129.159
127.957
127.832
126.290
126.232
124.492
124.322
122.784
122.663
117.977
117.938
106.753
106.638
68.940
56.759
47.195
46.793
43.149
36.178
34.790
31.691
27.654
23.672
H3C
CH3
H3C
N
N
HO
OH
2000
1000
0
-1000
-2000
150
100
50
ppm (t1)

**10e**


H3C
CH3
H3C
N
N
HO
Br
Br
OH
MeO
OMe
10f
1500
1000
500
0
1.76
1.88
3.76
6.00
1.08
1.58
2.00
2.92
10.0
5.0
ppm (t1)


164.319
161.961
152.353
149.674
149.643
124.772
124.755
118.986
118.819
116.883
116.761
108.917
108.758
73.965
61.137
56.329
56.249
47.786
46.968
43.530
36.016
35.038
31.671
31.532
27.935
24.295
H3C
CH3
H3C
N
N
HO
Br
Br
OH
MeO
OMe
2000
1500
1000
500
0
-500
-1000
-1500
150
100
50
ppm (t1)


H3C
CH3
H3C
N
N
HO
OH
MeO
OMe
10g
700
600
500
400
300
200
100
0
-100
1.70
0.97
0.97
5.98
6.04
1.04
1.80
2.00
2.95
10.44
10.0
5.0
ppm (t1)


165.369
163.034
152.348
152.237
148.574
122.877
118.551
118.361
117.729
117.668
113.973
113.703
74.464
61.414
56.168
56.017
47.920
47.152
43.763
35.996
35.102
31.514
27.975
24.356
H3C
CH3
H3C
N
N
HO
OH
MeO
OMe
1500
1000
500
0
-500
-1000
150
100
50
ppm (t1)