Supporting material for

$NaIO_4$-Mediated Selective Oxidative Halogenation of Alkenes and Aromatics using Alkali Metal Halides

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General experimental procedure for bromohydrin of olefins:

To a stirred mixture of olefin (10 mmol), LiBr (12 mmol), and 30% aq. H_2SO_4 (0.5 ml, 10 mmol) in CH_3CN:H_2O (2:1, 15 ml) at 10-15^{0}C, $NaIO_4$ (25 mol%) was added portion wise. The reaction was monitored by TLC. After completion of the reaction, it was diluted with water and extracted with CH_2Cl_2 (25 ml x 3). The organic layers were washed with dilute solution of Na_2SO_3 and brine. It was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give crude products, which were purified by column chromatography packed with silica gel using petroleum ether and ethyl acetate (9:1) as eluent to afford the pure products.

General experimental procedure for Methoxybromination of olefins:

To a stirred mixture of olefin (10 mmol), LiBr (12 mmol), and 30% aq. H_2SO_4 (0.5 ml, 10 mmol) in MeOH:H_2O (3:1, 15 ml) at 10-15^{0}C, $NaIO_4$ (25 mol%) was added portion wise. The reaction was monitored by TLC. After completion of the reaction, it was diluted with water and extracted with CH_2Cl_2 (25 ml x 3). The organic layers were washed with dilute solution of Na_2SO_3 and brine. It was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give crude products, which were purified by column chromatography packed with silica gel using petroleum ether and ethyl acetate (9:1) as eluent to afford the pure products.

General experimental procedure for Dibromination of olefins:

To a stirred mixture of olefin (10 mmol), and, LiBr (12 mmol), in acetic acid (15 ml) and $NaIO_4$ (25 mol%) was added portion wise. The reaction was monitored by TLC. After completion of the reaction, it was diluted with water and extracted with CH_2Cl_2 (25 ml x 3). The organic layers were washed with dilute solution of $NaHCO_3$, Na_2SO_3 and

brine. It was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give crude products, which were purified by column chromatography packed with silica gel using petroleum ether and ethyl acetate (9:1) as eluent to afford the pure products.

1. NMR DATA of Bromohydrins:

•2-Bromo-1-phenylethanol

^{1}H-NMR (200 MHz, $CDCl_3$): δ 7.20-7.60 (5H, m), 4.90-5.00 (1H, dd, *J* = 8.10 Hz, 4.00 Hz), 3.55-3.70 (2H, m), 2.60 (1H, brs),

^{13}C-NMR (50 MHz, $CDCl_3$): δ 140.33, 128.42, 128.20, 125.85, 125.85, 125.63, 73.54, 39.62.

MS m/z (% rel. intensity): 200(M+, 4), 141 (1), 127 (1), 121 (10), 107 (100), 94 (25), 84 (60), 79 (75), 65 (18), 55 (14).

•2-Bromo-1-(4-bromophenyl)ethanol

^{1}H-NMR (200 MHz, $CDCl_3$): δ 7.48 (2H, d, J = 8 Hz), 7.35 (2H, d, J = 8 Hz), 4.86 (1H, (C) H (OH), dd, J = 8 Hz, J = 6 Hz), 3.44-3.50 (2H, (C) H_2 Br,m), 2.82 (1H, brs).

^{13}C-NMR (50 MHz, $CDCl_3$): δ 139.23, 13154, 127.54, 122.11, 72.85, 39.25.

MS m/z (% rel. intensity): 280 (30), 198 (5), 185 (100), 169 (15), 157 (70), 120 (10), 102 (20), 91 (50), 77 (100), 63 (40), 51 (100).

•2-Bromo-1-(4-ethoxyphenyl)ethanol.

^{1}H-NMR (200 MHz, $CDCl_3$): δ 7.31 (2H, d, J = 8.5 Hz), 6.91 (2H, d, J = 8.5 Hz), 4.81 (1H, (C) H (OH), dd, J = 8.9 Hz, 3.95 Hz), 3.83 (3H, s), 3.35 -3.52 (2H, (C) H_2 Br, m).

^{13}C-NMR (50 MHz, $CDCl_3$): δ 164.15,133.31, 126.99, 114.05, 73.73, 55.28, 15.43.

•2-Bromo-1-(4-methylphenyl)ethanol

^{1}H-NMR (200 MHz, $CDCl_3$): δ 7.25 (2H, d, J = 8.3 Hz), 7.17 (2H, d, J = 8.3 Hz), 4.86 (H, dd, J = 18.15 Hz, J = 3.7 Hz), 3.57-3.64 (2H, m), 2.55 (1H, brs), 2.34 (3H, s), ^{13}C-NMR (50 MHz, $CDCl_3$): δ 138.20, 137.72, 137.39, 128.98, 125.70, 125.52, 73.32, 39.43, 20.91.

•(±)-*trans*-2-Bromo-1-phenyl-1-hydroxypropane

^{1}H-NMR (200 MHz, $CDCl_3$): δ 7.34 – 7.35 (5H, m), 5.005 (H, d, J = 3.5 Hz), 4.425 (1H, quartet of doublets, J = 6.8 Hz, J = 3.5 Hz), 2.34 (1H, brs), 1.545 (3H, d, J = 6.4 Hz). ^{13}C-NMR (50 MHz, $CDCl_3$): δ 139.01, 127.54, 127.25, 125.70, 76.60, 55.09, 18.19.

•2-Bromo-1-(4-chloromethylphenyl)ethanol

^{1}H-NMR (200 MHz, $CDCl_3$): δ 7.32 – 7.65 (4H, m), 4.90 (1H, dd, J = 3.5 Hz, J = Hz), 4.58 (2H, s), 3.49 – 3.65 (2H, m), 2.58 (1H, brs).

^{13}C-NMR (50 MHz, $CDCl_3$): δ 140.52, 137.50, 128.76, 126.29, 73.21, 45.68, 39.69.

•(±)-*trans*-Methyl-2-bromo-3-hydroxy-3-phenylpropionate

^{1}H-NMR (200 MHz, $CDCl_3$): δ 7.4 (5H, m), 5.08 (1H, d, J = 8.15 Hz), 4.39 (12H, d, J = 8.15 Hz), 3.81 (3H, s), 2.97 (1H, brs).

^{13}C-NMR (50 MHz, $CDCl_3$): δ 140.52, 139.09, 128.68, 128.46, 126.95, 75.61, 53.00, 47.52.

•(±)-*trans*-Ethyl-2-bromo-3-hydroxy-3-(4-methoxyphenyl)propionate.

^{1}H-NMR (200 MHz, $CDCl_3$): δ 7.31 (2H, d, J = 8.15 Hz), 6.90 (2H, d, J = 8.15 Hz), 5.03 (1H, d, J = 8 Hz), 4.21 (1H, d, J= 8 Hz), 3.81 (3H, s), 2.64 (1H, brs), 1.29 (3H, t, J = 8.15 Hz).

^{13}C-NMR (50 MHz, $CDCl_3$): δ 169.12, 131.29, 128.02, 113.50, 74.32, 61.93, 54.91, 48.11, 13.52.

MS m/z (% rel. intensity): 304 (7), 259 (3), 223 (3), 206 (11), 161 (7), 137 (100), 121 (14), 109 (22), 94 (18), 77 (33), 64 (14).

•(±)-*trans*-2-Bromo-1-hydroxy –1-phenylpropanol

^{1}H-NMR (200 MHz, $CDCl_3$): δ 6.75 – 7.45 (5H, m), 4.25 – 4.44 (1H, m), 4.03 – 4.12 (2H, m), 3.50 – 3.67 (1H, m), 2.45 (1H, brs).

^{13}C-NMR (50 MHz, $CDCl_3$): δ 159.56, 138.28, 129.49, 121.48, 116.29, 114.64, 69.39, 69.24, 68.55, 63.99, 51.93, and 34.77.

MS m/z (% rel. intensity): 234 (2), 149 (3), 133 (30), 107 (99), 91 (20), 79 (100), 65 (10).

•(±)-*trans*-2-Bromo-1-hydroxy indane

^{1}H-NMR (200 MHz, DMSO-D_6): δ 7.23-7.45 (4H, m), 5.31 (1H, d, J = 6.15 Hz), 4.18 – 4.33 (1H, m), 3.53 – 3.64 (1H, m), 3.15 – 3.38 (1H, m), 2.49 (1H, brs).

^{13}C-NMR (50 MHz, DMSO-D_6): δ 206.50, 159.37, 141.91, 140.33, 139.16, 127.95, 126.70, 123.76, 123.54, 83.98, 82.33, 54.21, 41.86, 39.89.

•2,2-Dimethylcoumarin. Bromohydrin.

^{1}H-NMR (200 MHz, $CDCl_3$): δ 7.41 (1H, d, J = 10.17 Hz), 7.14 (1H, m), 6.91 (1H, m), 6.73 (1H, d, J = 10.17 Hz), 4.86 (1H, d, J = 10.15 Hz), 4.08 (1H, d, J = 10.15 Hz), 1.53 (3H, s), 1.34 (3H, s).

^{13}C-NMR (50 MHz, $CDCl_3$): δ 151.93, 129.70, 127.52, 122.30, 121.13, 116.93, 78.82, 70.28, 62.88, 28.77, 19.78.

•(±)-*trans*-2-Bromo-1-hydroxy cyclooctane.

^{1}H-NMR (200 MHz, $CDCl_3$): δ 4.37 – 4.75 (1H, m), 3.82 – 3.89 (1H, m), 1.98 – 2.19 (2H, m), 1.75 – 1.95 (8H, m), 1.49 –1.74 (2H, m).

^{13}C-NMR (50 MHz, $CDCl_3$): δ 70.79, 56.60, 34.47, 32.74, 32.23, 24.25, 21.57.

•Allyl bromide. Bromohydrin.

^{1}H-NMR (200 MHz, $CDCl_3$): δ 4.28 –4.56 (1H, m), 3.57 – 4.26 (4H, m), 2.46 (1H, brs).

^{13}C-NMR (50 MHz, $CDCl_3$): δ 69.98, 63.99, 53.18, 35.43, 31.79.

2. NMR-DATA of Methoxybromides:

• 2-Bromo-1-methoxy-1-phenylethane

^{1}H ($CDCl_3$). δ 7.37-7.32 (5H, m), 4.40-4.36 (1H, dd, 9 Hz, 3 Hz), 3.56-3.43 (2H, m), 3.30 (3H, s).

^{13}C ($CDCl_3$). δ 138.92, 128.33, 126.59, 83.26, 57.07, 36.07.

• 2-Bromo-1-methoxy-1-(p-chloromethylphenyl)ethane

^{1}H ($CDCl_3$). δ 7.43-7.30 (5H, m), 4.6 (3H, s), 4.42-4.38 (1H, dd, 9 Hz, 3.8 Hz), 3.52-3.46 (2H,m), 3.32 (3H,s).
^{13}C ($CDCl_3$). δ 139.23, 137.61, 128.70, 127.02, 82.83, 57.19, 45.63, 35.89.
MS (m/z, %RI). 264 (M^+, 5), 252 (10), 229 (60), 197 (50), 183 (20), 169 (100), 147 (70), 134 (75), 117 (85), 105 (90), 91 (75), 77 (90), 63 (85), 44 (85).

• 2-Bromo-1-methoxy-1-(4-bromophenyl)ethane

^{1}H ($CDCl_3$). δ 7.53-7.50 (2H, d, 9 Hz), 7.23-7.20 (2H, d, 9 Hz), 4.38-4.33 (1H, dd, 9Hz, 3.8 Hz), 3.51-3.44 (2H, m), 3.33 (3H, s).
^{13}C ($CDCl_3$). δ 138.65, 137.92, 131.57, 128.30, 82.40, 57.10, 35.58.
MS (m/z, %RI). 294 (M^+, 10), 263 (8), 212 (10), 199 (100), 184 (80), 169 (40), 157 (40), 134 (30), 120 (60), 102 (90), 77 (95), 63 (80), 51 (85).

• 2-Bromo-1-methoxy-1-methyl-1-phenylethane

^{1}H ($CDCl_3$). δ 7.39-7.32 (5H, m), 3.52-3.49 (1H, d, 9 Hz), 3.45-3.41 (1H, d, 12 Hz), 3.14 (3H, s), 1.71 (3H,s).
^{13}C ($CDCl_3$). δ 141.50, 128.18, 127.54, 126.14, 50.75, 42.70, 23.68, 21.70.
MS (m/z, %RI). 276 (M^+, 1), 149 (18), 135 (100), 117 (20), 103 (10), 91 (18), 77 (20), 43 (70).

• 2-Bromo-1-methoxy-1-(4-chlorophenyl)ethane

^{1}H ($CDCl_3$). δ 7.34 (4H, s), 3.59-3.56 (1H, d, 9 Hz), 3.14 (3H, s), 1.69 (3H, s).
^{13}C ($CDCl_3$). δ 140.39, 133.67, 128.55, 127.85, 51.00, 42.42.
MS (m/z, %RI). 314 (M^+, 2), 249 (30), 223 (25), 182 (30), 169 (95), 152 (72), 139 (70), 125 (75), 115 (89), 101 (95), 89 (80), 75 (85), 57 (90), 43 (100).

• 2-Bromo-1-methoxy-1-(4-methylphenyl)ethane

^{1}H ($CDCl_3$). δ 7.20 (2H, d, 8 Hz), 7.19 (2H, d, 8 Hz), 4.36-4.34 (1H, dd, 5 Hz, 4.5 Hz), 3.54-3.51 (1H, dd, 10 Hz, 5 Hz), 3.46-3.30 (1H, dd, 10 Hz, 5.6 Hz), 3.30 (3H, s), 2.36 (3H, s).
^{13}C ($CDCl_3$). δ 138.16, 135.96, 129.25, 126.59, 83.35, 57.01, 36.20, 21.06.
MS (m/z, %RI). 314 (M^+, 2), 228 (30), 197 (50), 148 (80), 135 (100), 117 (95), 105 (87), 91 (90), 77 (80), 65 (75), 51 (85).

• (±)-*trans*-2-Bromo-1-methoxy –1-phenylpropanol

^{1}H ($CDCl_3$). δ 7.38-7.35 (5H, m), 4.50-4.48 (1H, d, 6 Hz), 4.22-4.19 (1H, m), 4.02-3.88 (2H, dd, 12 Hz, 5.5 Hz), 3.26 (3H, s).
^{13}C ($CDCl_3$). δ 138.04, 128.45, 128.33, 127.49, 85.78, 64.42, 57.88, 57,42.
MS (m/z, %RI). 349 (M^+, 1), 184 (7), 164 (5), 147 (5), 134 (20), 121 (100), 103 (90), 91 (80), 77 (80), 65 (45), 51 (85).

• (±)-*trans*-Ethyl-2-bromo-3-methoxy-3-(4-methoxyphenyl)propionate.

^{1}H ($CDCl_3$). δ 7.30-7.28 (2H, d, 10 Hz), 6.92-6.91 (2H, d, 8 Hz), 4.51-4.49 (1H, d, 10 Hz), 4.32-4.27 (2H, q), 4.21-4.19 (1H, d, 10 Hz), 3.82 (3H,s), 3.20 (3H,s), 1.34-1.32 (3H, t)
^{13}C ($CDCl_3$). δ 168.8, 159.98, 129.16, 128.91, 113.75, 83.71, 61.86, 57.25, 55.15, 47.79, 13.89.
MS (m/z, %RI). 318 (M^+, 1), 271 (4), 205 (5), 177 (8), 152 (40), 151 (100), 135 (30), 135 (25), 121 (12), 108 (10), 89 (16), 77 (18), 63 (15), 44 (20).

• Indene. methoxybromide

^{1}H ($CDCl_3$). δ 7.41-7.22 (4H, m), 4.98-4.97 (1H, d, 4.8 Hz), 4.49-4.46 (1H, m), 3.73-3.66 (1H, dd, 12 Hz, 4.6 Hz), 3.58 (3H, s), 3.29-3.19 (1H, dd, 14 Hz, 6 Hz).

^{13}C ($CDCl_3$). δ 141.24, 140.33, 140.08, 139.90, 129.01, 127.05, 125.10, 124.61, 93.21, 91.62, 57.53, 50.72, 43.40, 41.54, 25.51.

3. NMR-DATA of Dibromides:

1,2-Dibromo-1-phenylethane

^{1}H (200 MHz, $CDCl_3$). δ 7.40 (5H, m), 5.12-5.20 (1H, dd, 6.0 Hz, 6.0 Hz), 3.98-4.15 (2H,m).

^{13}C (50 MHz, $CDCl_3$). δ 138.61, 129.09, 128.76, 127.62, 50.90, 35.02.

Ms (m/z, %RI). 264 (M+, 5), 185 (96), 183 (100), 77 (45), 63 (8)

• 1,2-Dibromo-1-(4-chloromethylphenyl)ethane

^{1}H ($CDCl_3$). δ 7.41 (4H, s), 5.1-5.2 (1H, dd, 6.0 Hz, 6.0 Hz), 4.51 (2H, s), 4.0-4.1 (2H, m).

^{13}C ($CDCl_3$). δ 138.72, 138.00, 128.98, 128.02, 50.02, 45.46, 34.69.

MS (m/z, %RI). 340 (M+, 2), 261 (30), 180 (100), 165 (25), 152 (10)

• 1,2-Dibromoindane.

^{1}H ($CDCl_3$). δ 7.3 (4H, s), 5.65 (1H, s), 4.8- 4.9 (1H, d, 7.0 Hz), 3.75-3.9 (1H, dd, 16.0 Hz, 7.0 Hz), 3.2-3.3 (1H, d, 7.0 Hz).

^{13}C ($CDCl_3$). δ 141.70, 138.25, 126.90, 125.71, 122.93, 80.97, 53.93, 39.06.

MS (m/z, %RI). 276 (M+, 2), 195 (40), 115 (100), 89 (15)

• 1,2-Dibromo-1-phenylpropane

^{1}H ($CDCl_3$). δ 7.38 (5H, m), 5.04-4.09 (1H, d, 10 Hz), 4.55-4.69 (1H, m), 2.05-2.08 (3H,d, 6 Hz).

^{13}C ($CDCl_3$). δ 138.10, 128.88, 127.54, 126.93, 52.49, 49.93, 21.45.

MS (m/z, %RI). 278 (M^+, 2), 199 (55), 171 (10), 118 (100), 91 (30).

• Methyl-2,3-dibromopropionate

^{1}H ($CDCl_3$). δ 7.4 (5H, m), 5.03-5.04 (1H, d, 12 Hz), 4.8- 4.9 (1H, d, 12 Hz), 3.8 (3H, s).

^{13}C ($CDCl_3$). δ 168.27, 137.47, 129.34, 128.83, 128.02, 53.36, 50.57, 46.64.

• 1,2-Dibromo-1-methyl-1-(-4-chlorophenyl)ethane

^{1}H ($CDCl_3$). δ 7.4-7.5 (2H, d, 4.5 Hz), 7.3-7.4 (2H, d, 4.5 Hz), 3.7 (2H, s), 1.65 (3H, s).

^{13}C ($CDCl_3$). δ 142.71, 133.37, 128.52, 126.41, 71.25, 45.78, 27.99.

MS (m/z, %RI). 232 (M^+-80, 35), 155 (100), 139 (20), 125 (15), 115 (70).

•1,2,3-Tribromopropane

^{1}H ($CDCl_3$). δ 4.41 (1H, m), 3.99-3.80 (4H, m),

^{13}C ($CDCl_3$). δ 48.26, 37.19, 34.84.

• 2,3-Dibromopropanamide

^{1}H ($CDCl_3$). δ 4.63-4.66 (1H, dd, 15 Hz, 6 Hz), 4.05-3.95 (1H, t, 15 Hz), 3.73-3.66 (1H, dd, 15 Hz, 6 Hz), 3.32 (2H, br).

^{13}C ($CDCl_3$). δ 168.20, 42.82, 29.99.

•2,3-Dibromo N,N-dimethylpropanamide

^{1}H ($CDCl_3$). δ

^{13}C ($CDCl_3$). δ 166.17, 38.88, 37.19, 36.20, 30.57.

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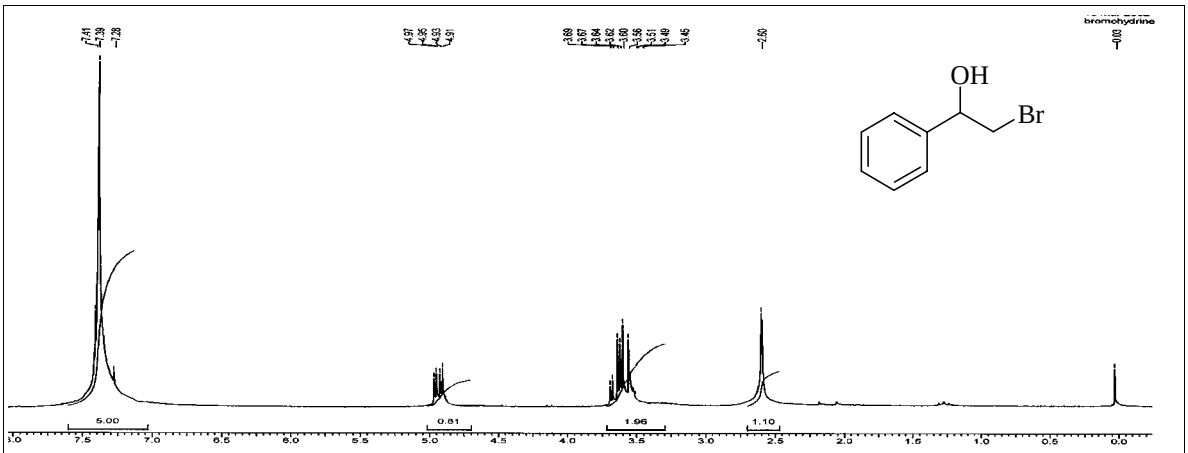
4. Spectral Data: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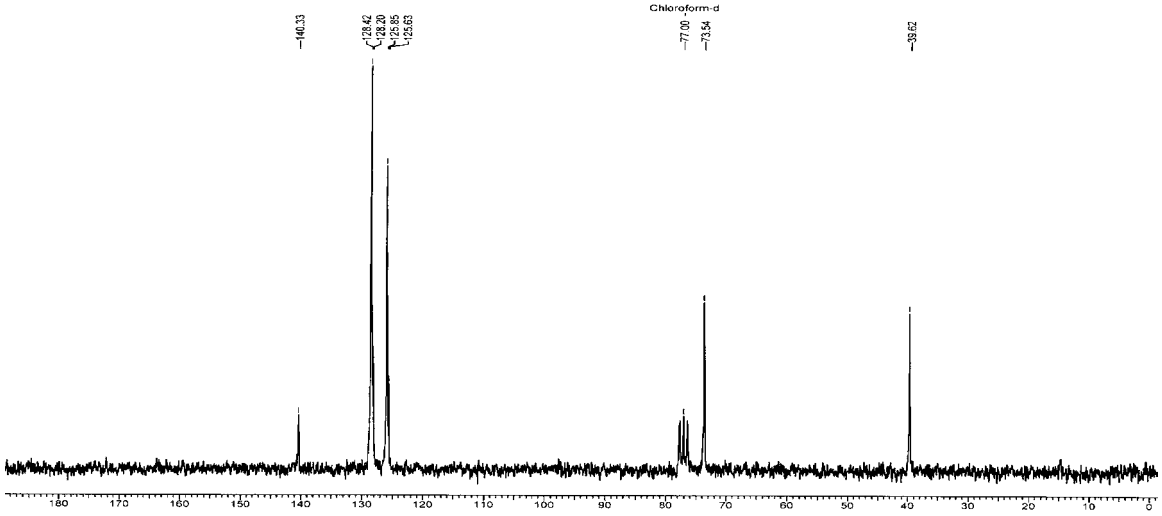
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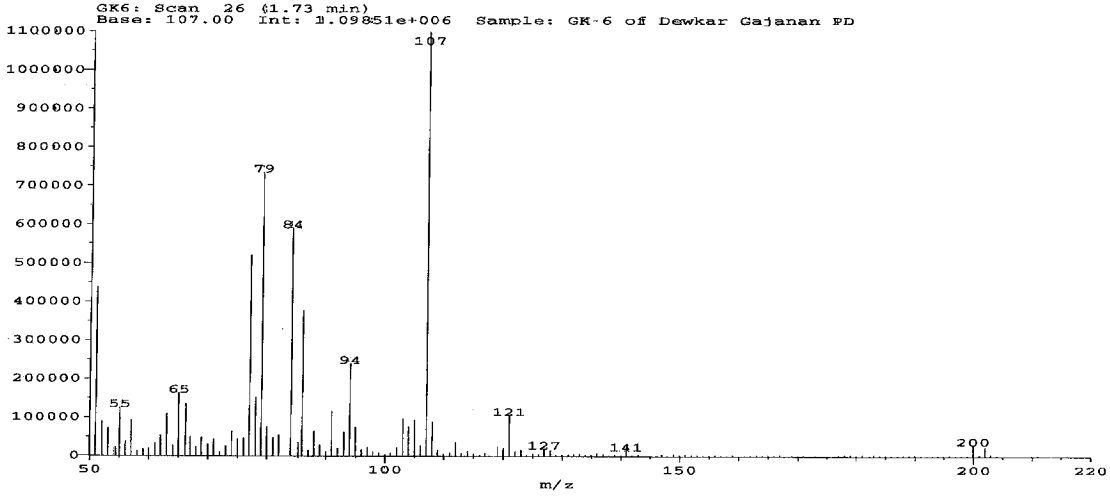
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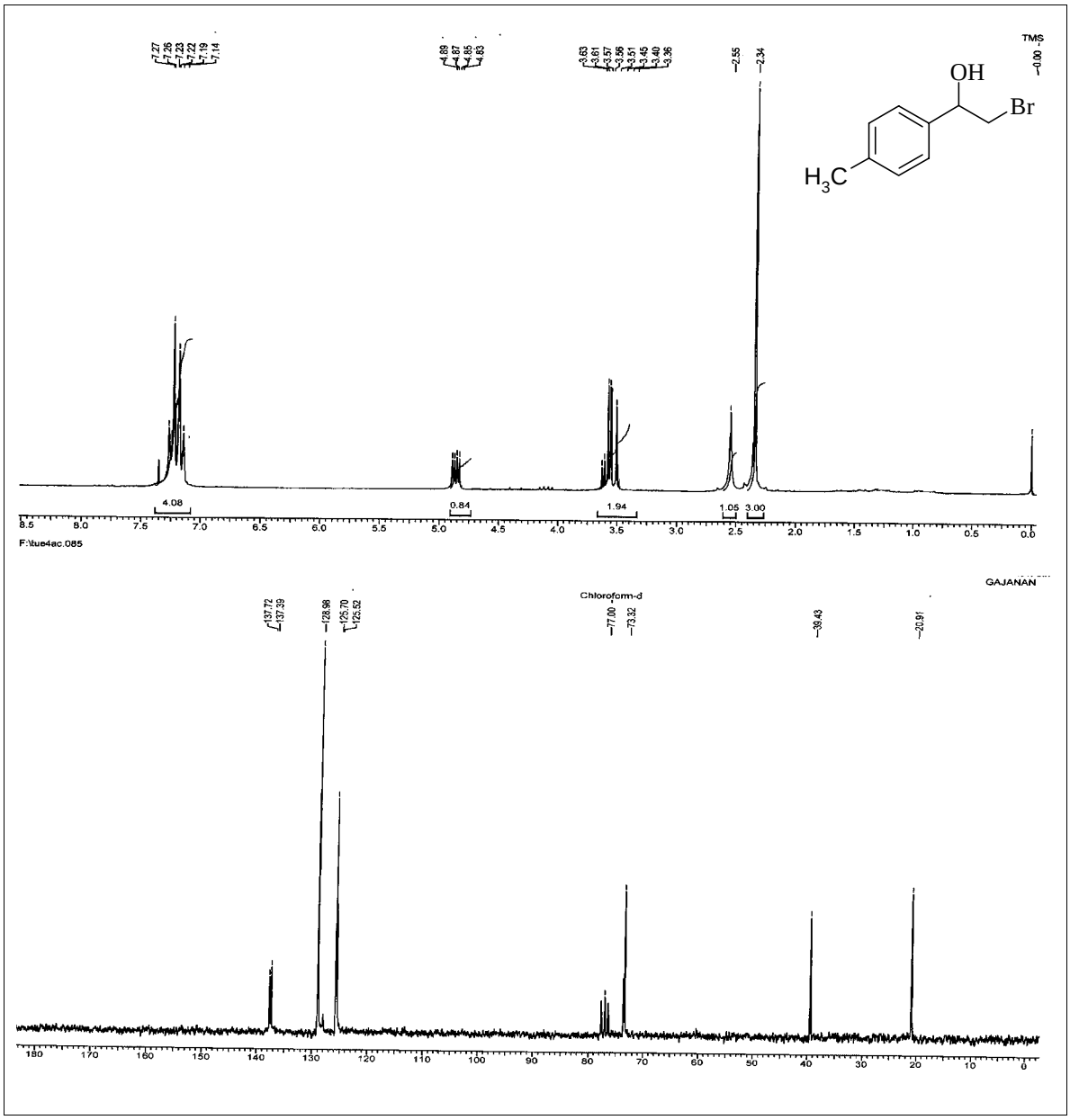
m/z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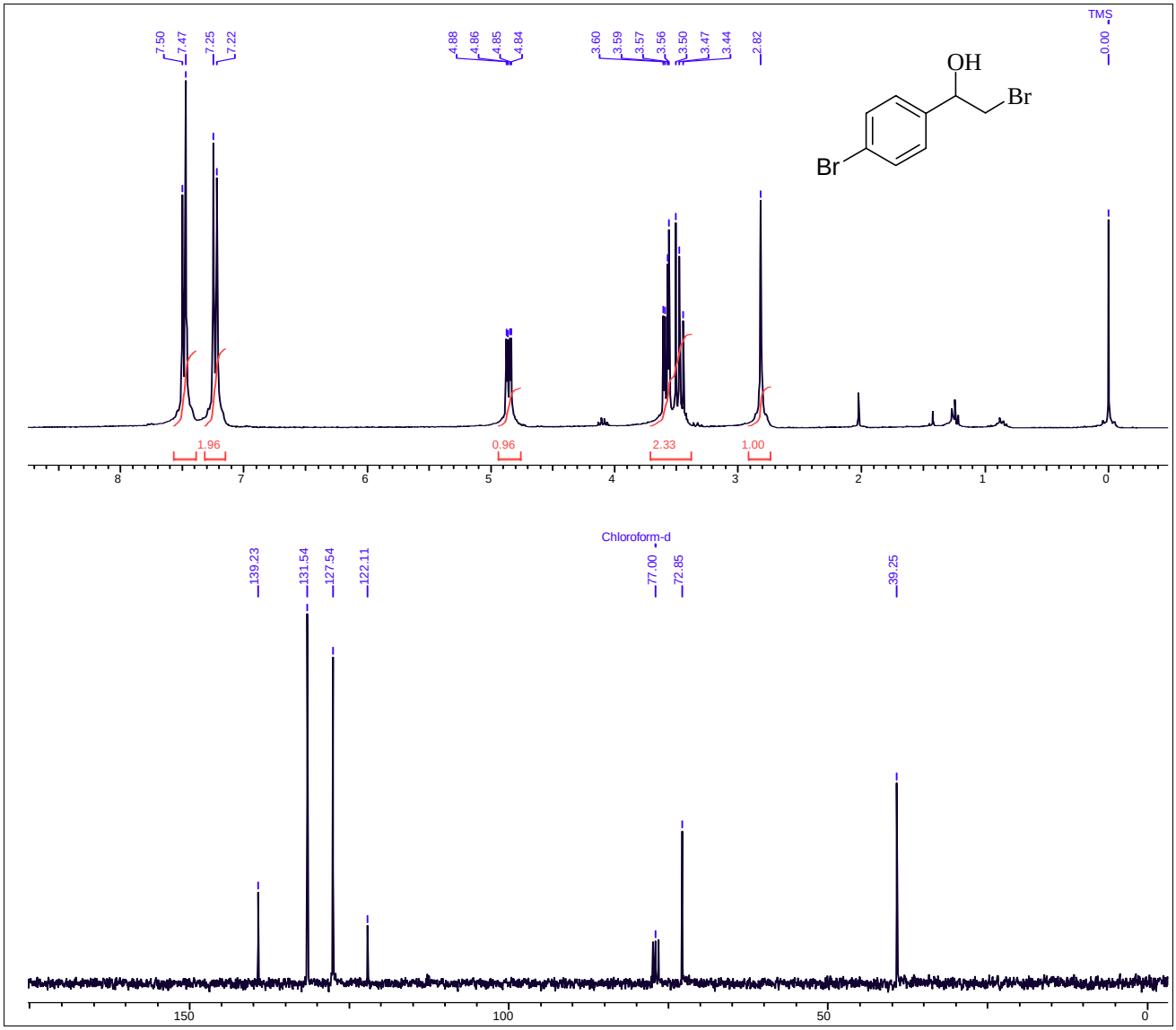
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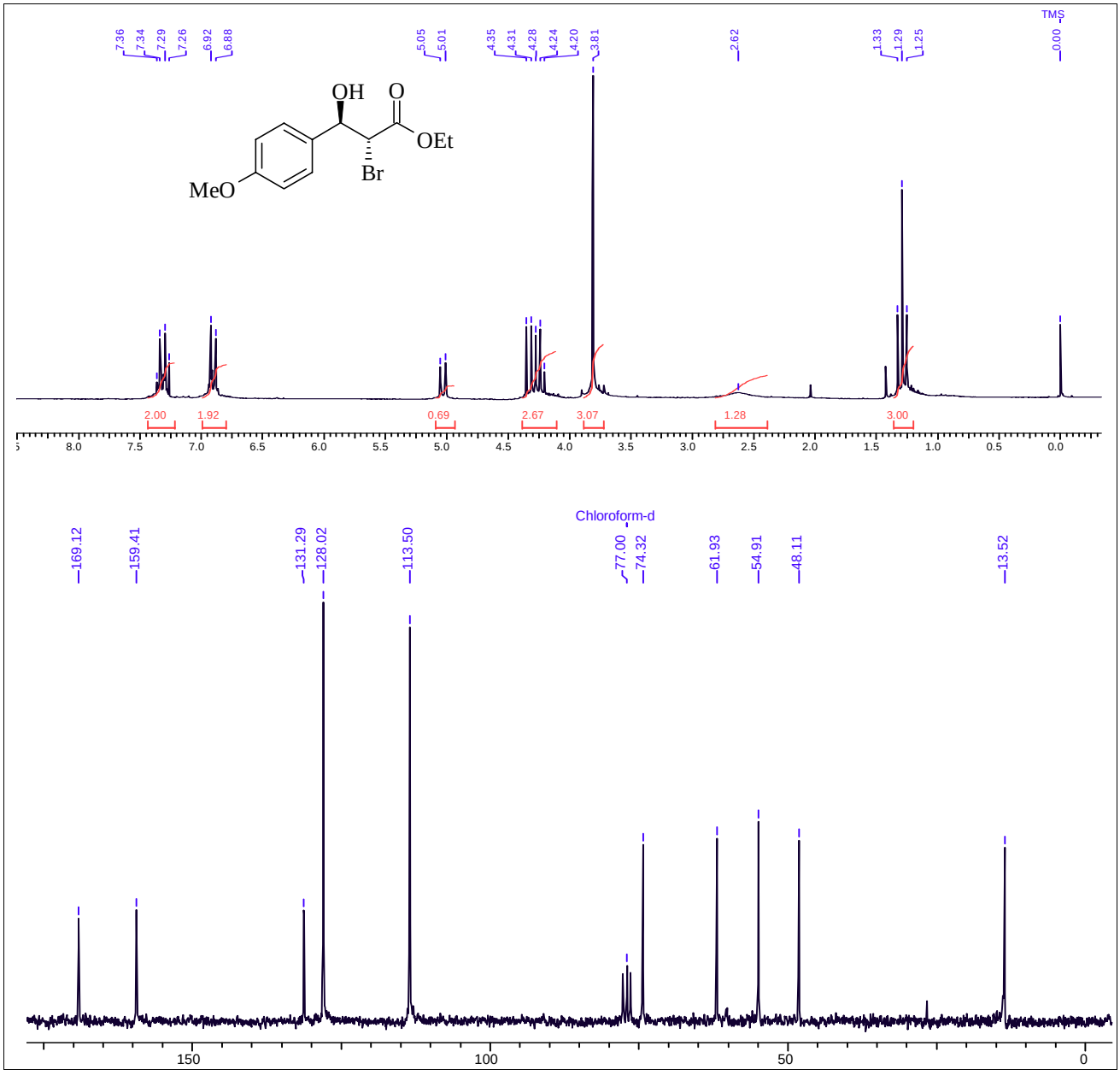
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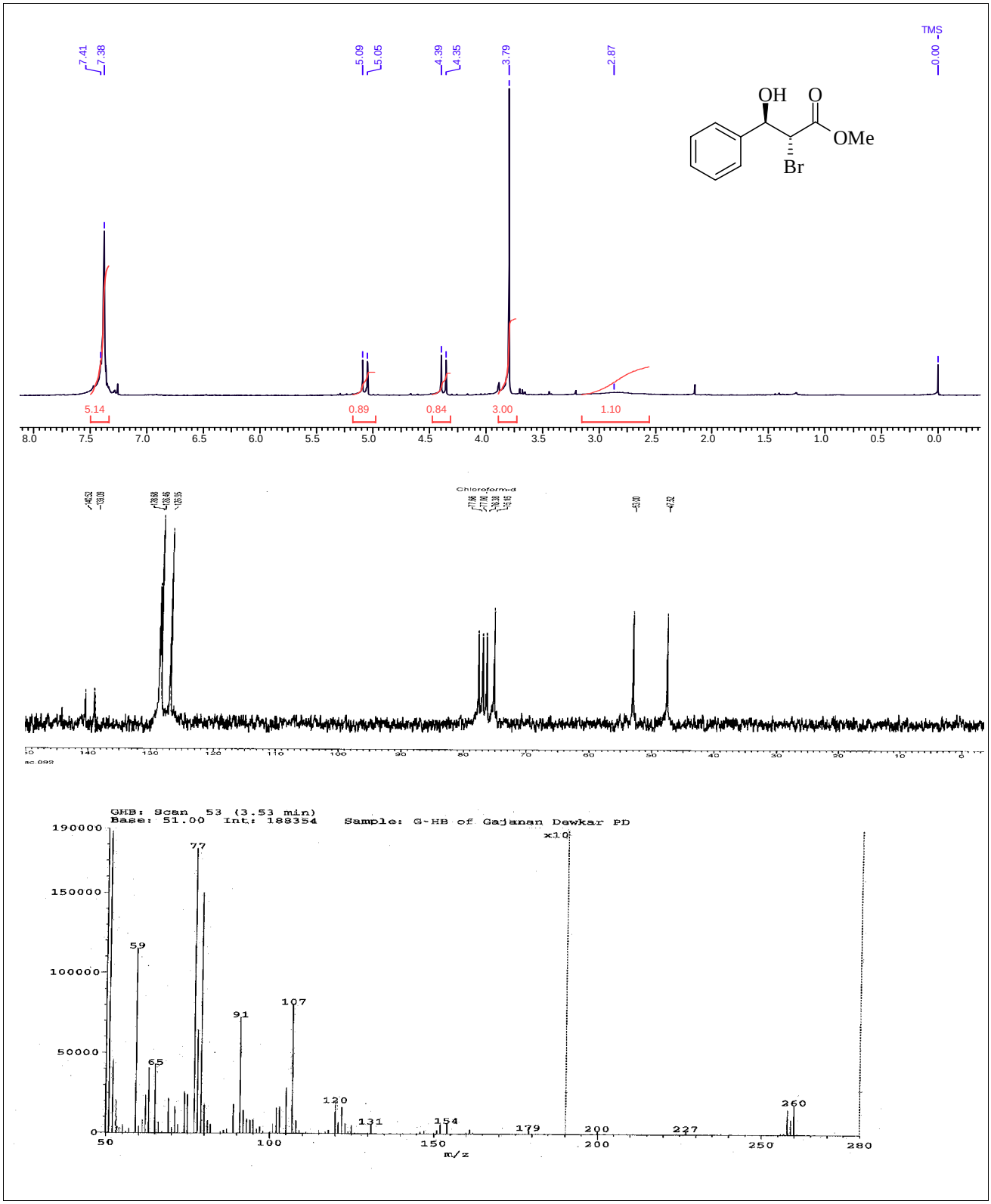
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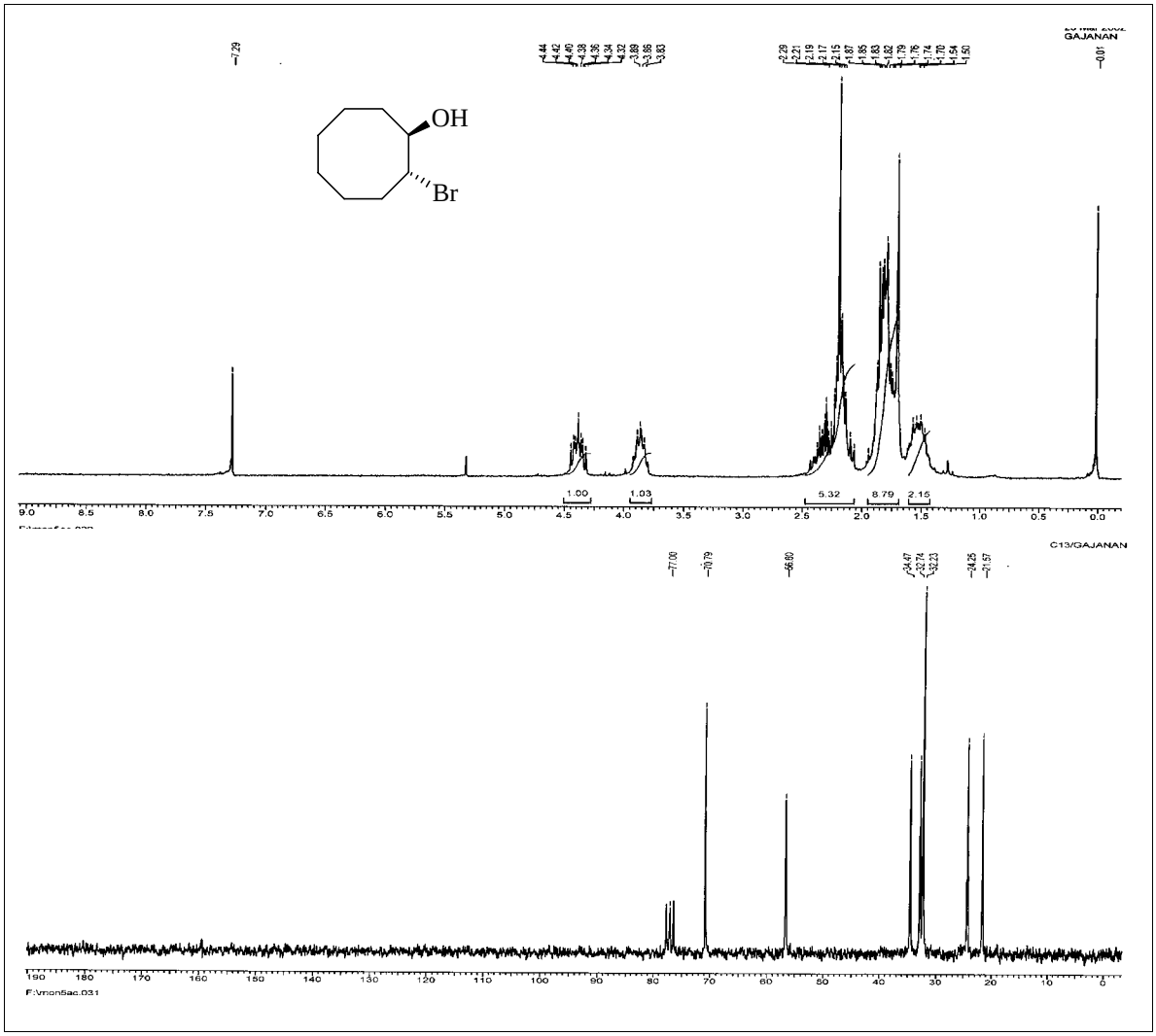
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1.87
1.85
1.83
1.82
1.79
1.76
1.74
1.70
1.54
1.50
0.01
1.00
1.03
5.32
8.79
2.15
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70.79
66.80
34.47
32.74
32.23
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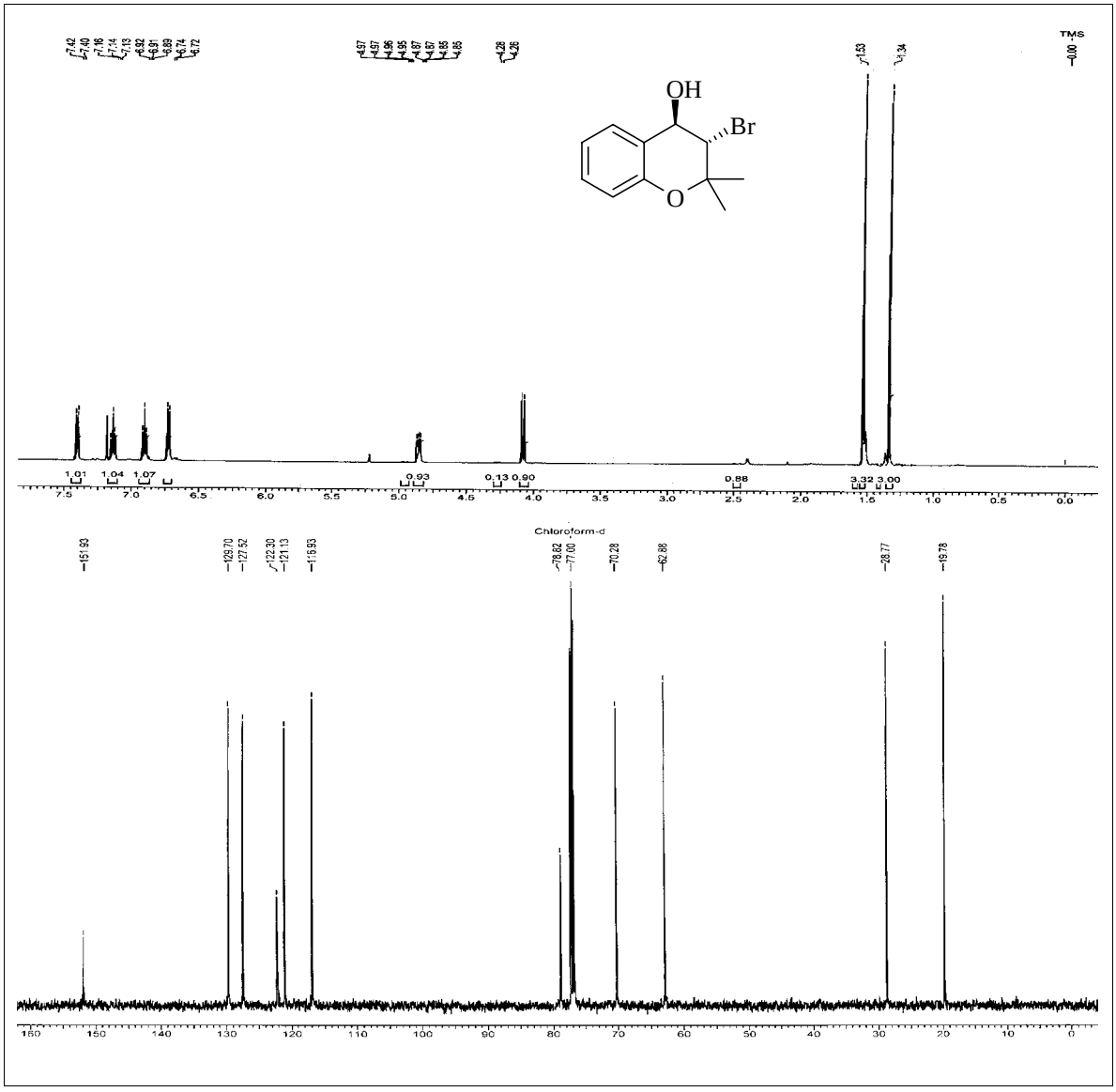
21.57
F:\mon5ac.031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Br
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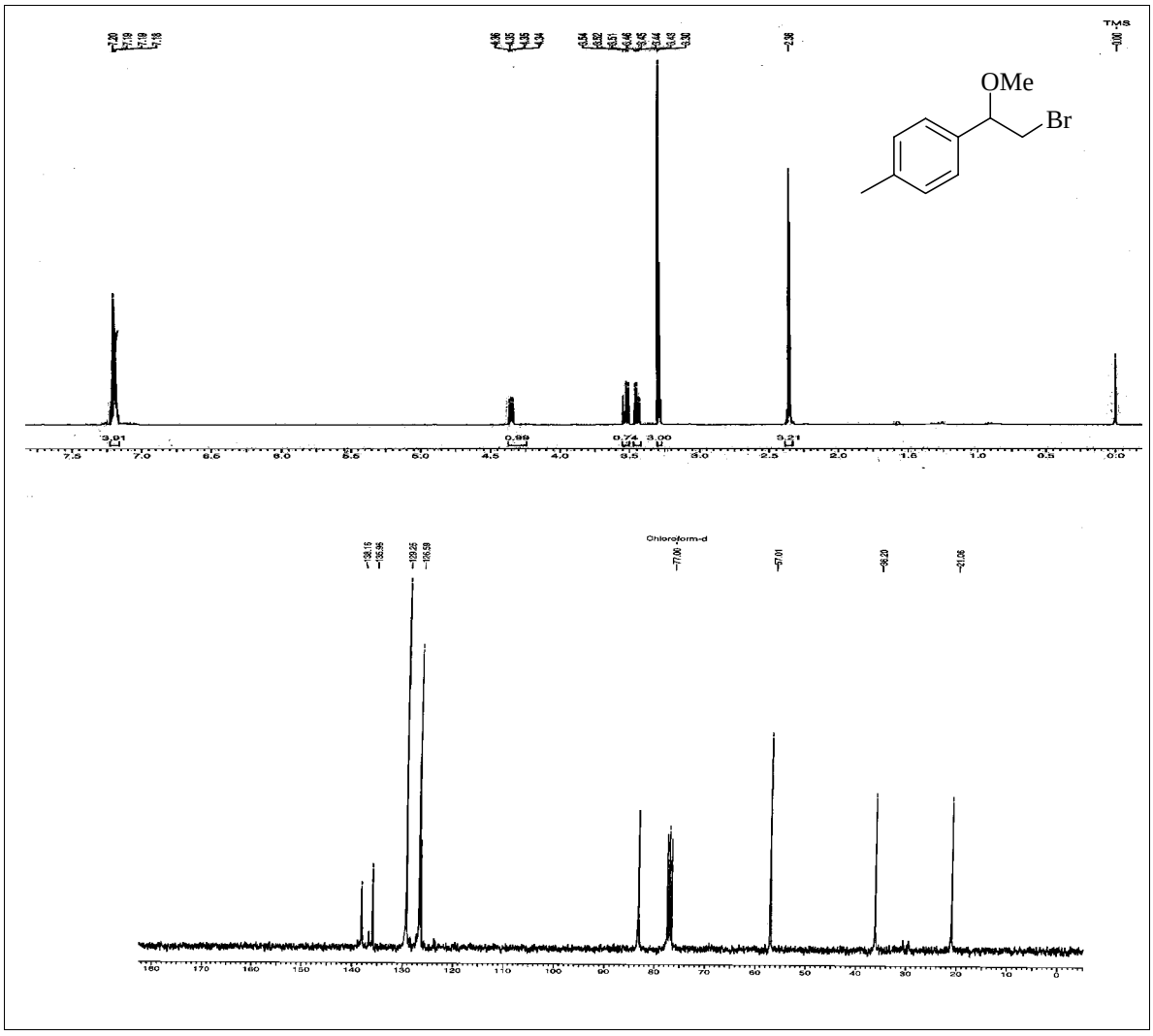
Chloroform-d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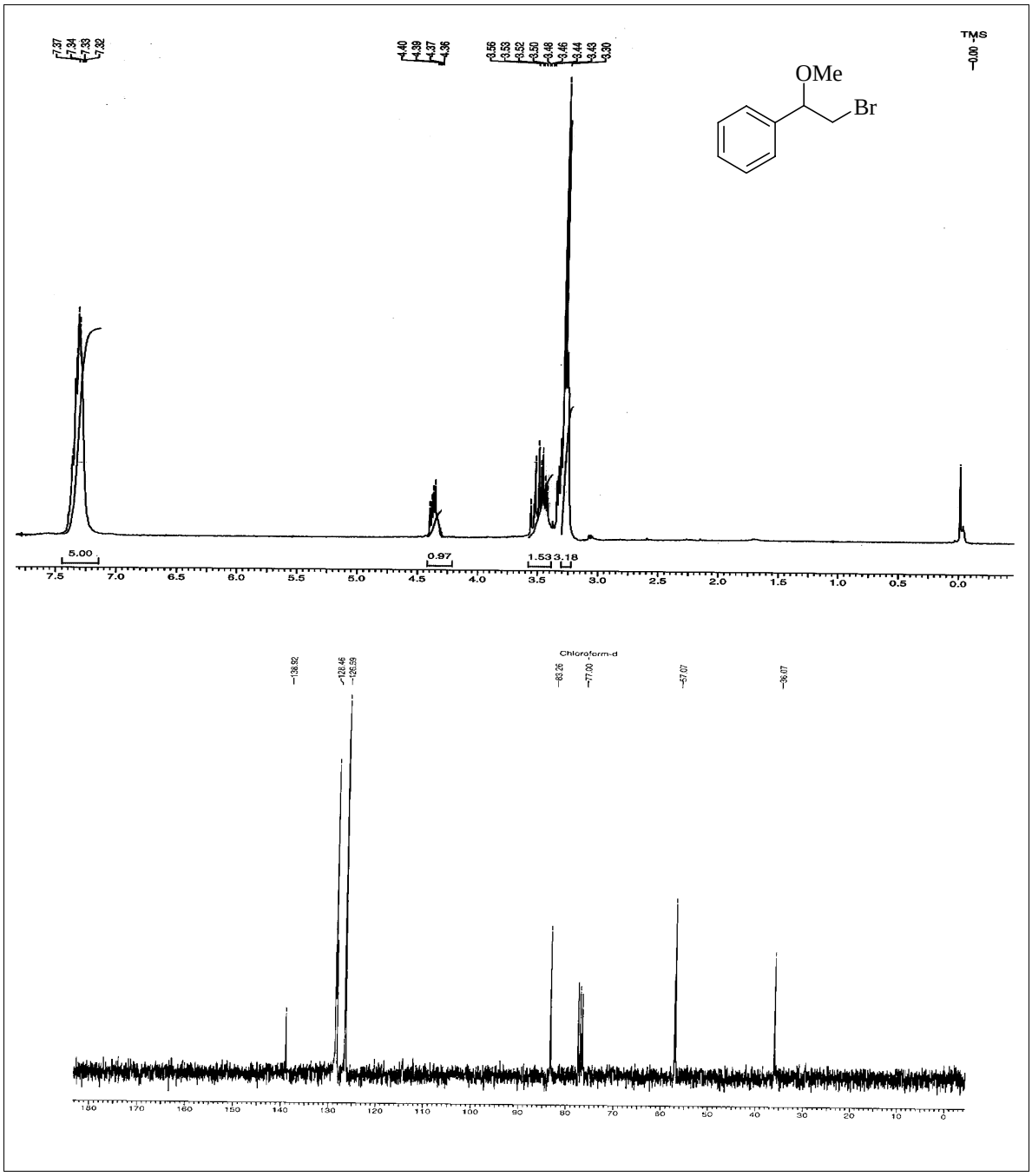
Br
Chloroform-d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4.36
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3.48
3.46
3.44
3.43
3.30
TMS
0.00
OMe
Br
5.00
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1.53
3.18
Chloroform-d
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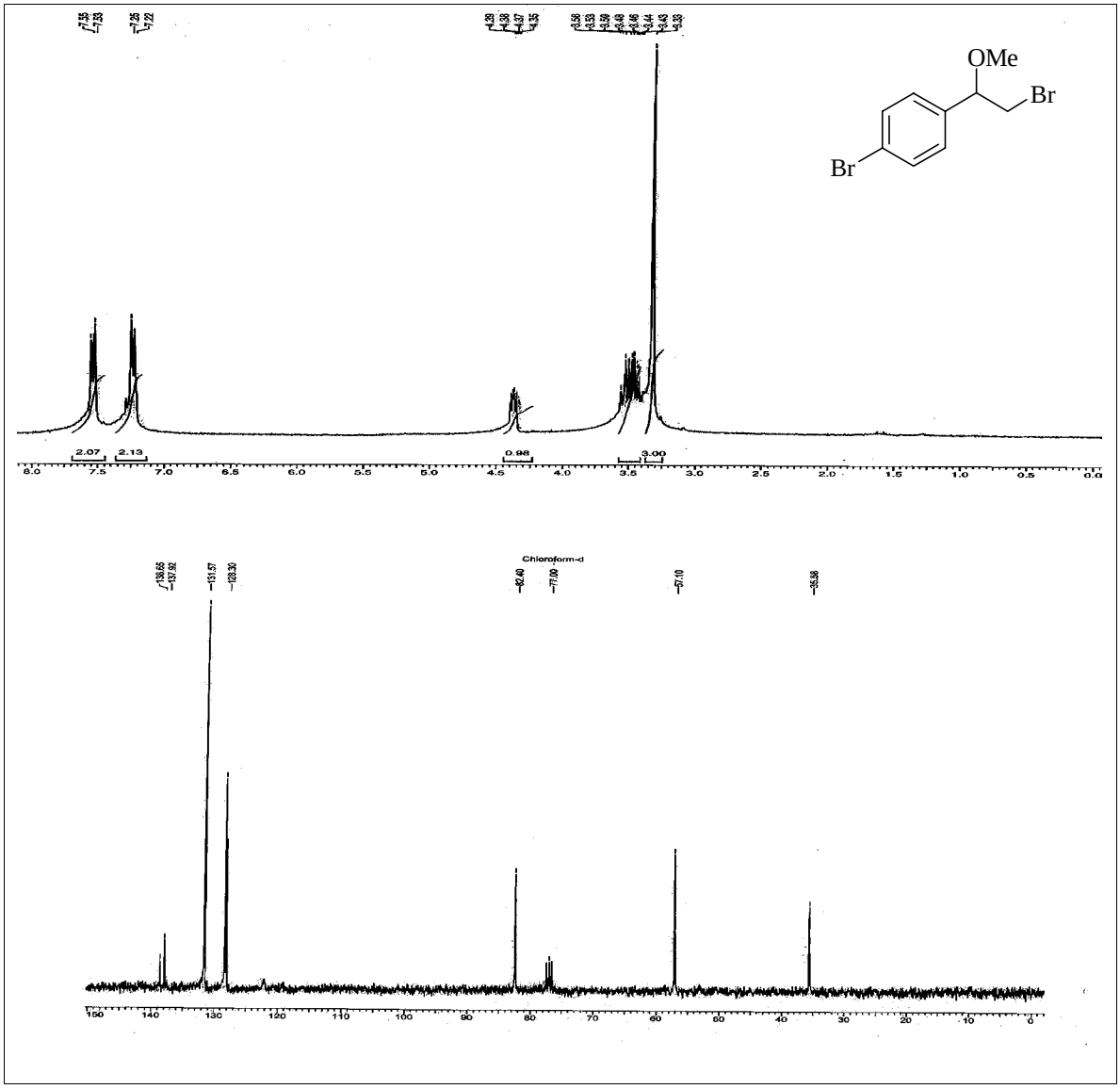
128.46
126.59
63.26
77.00
57.07
36.07


OMe
Br
Br
2.07
2.13
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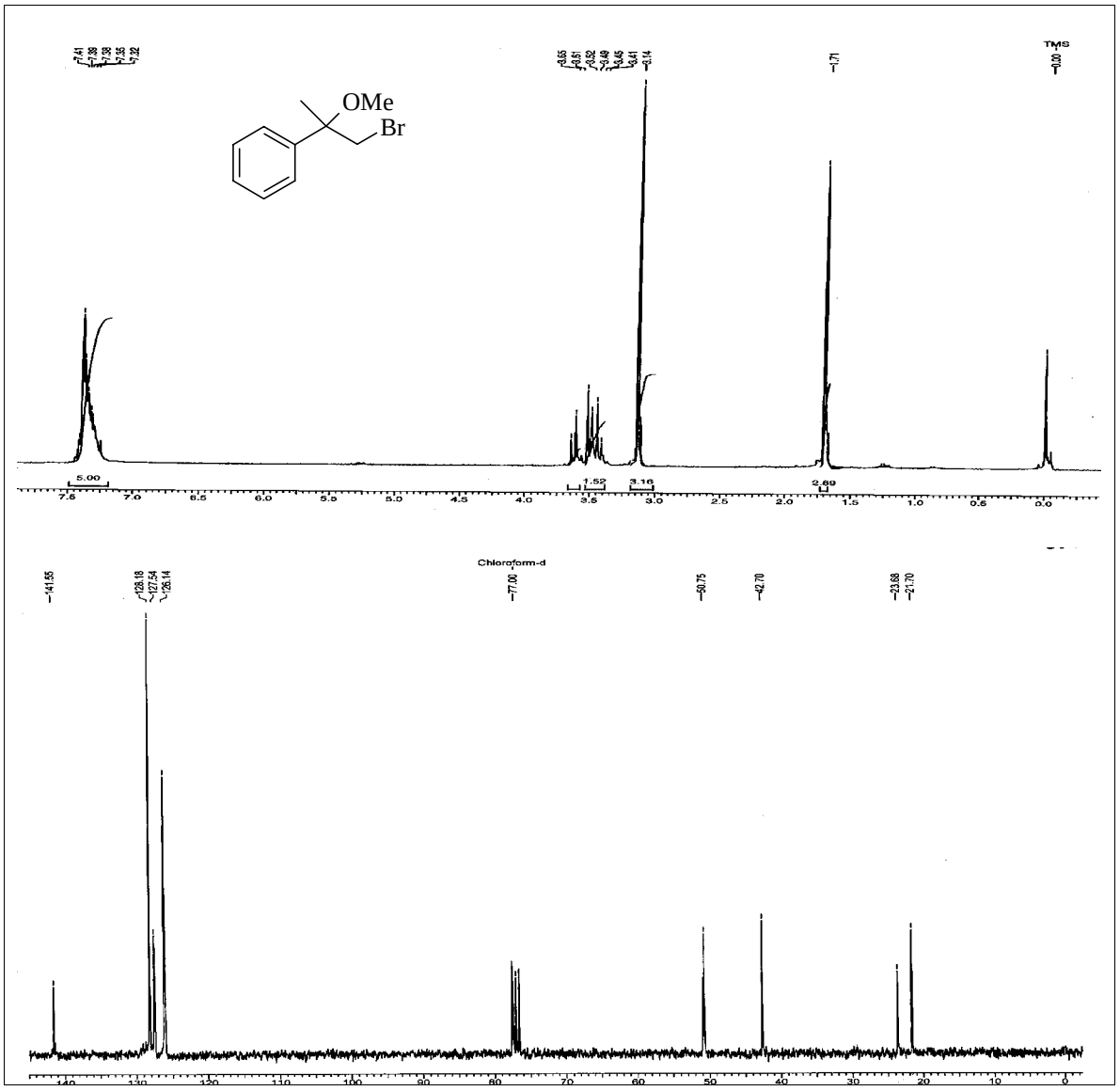
3.00
Chloroform-d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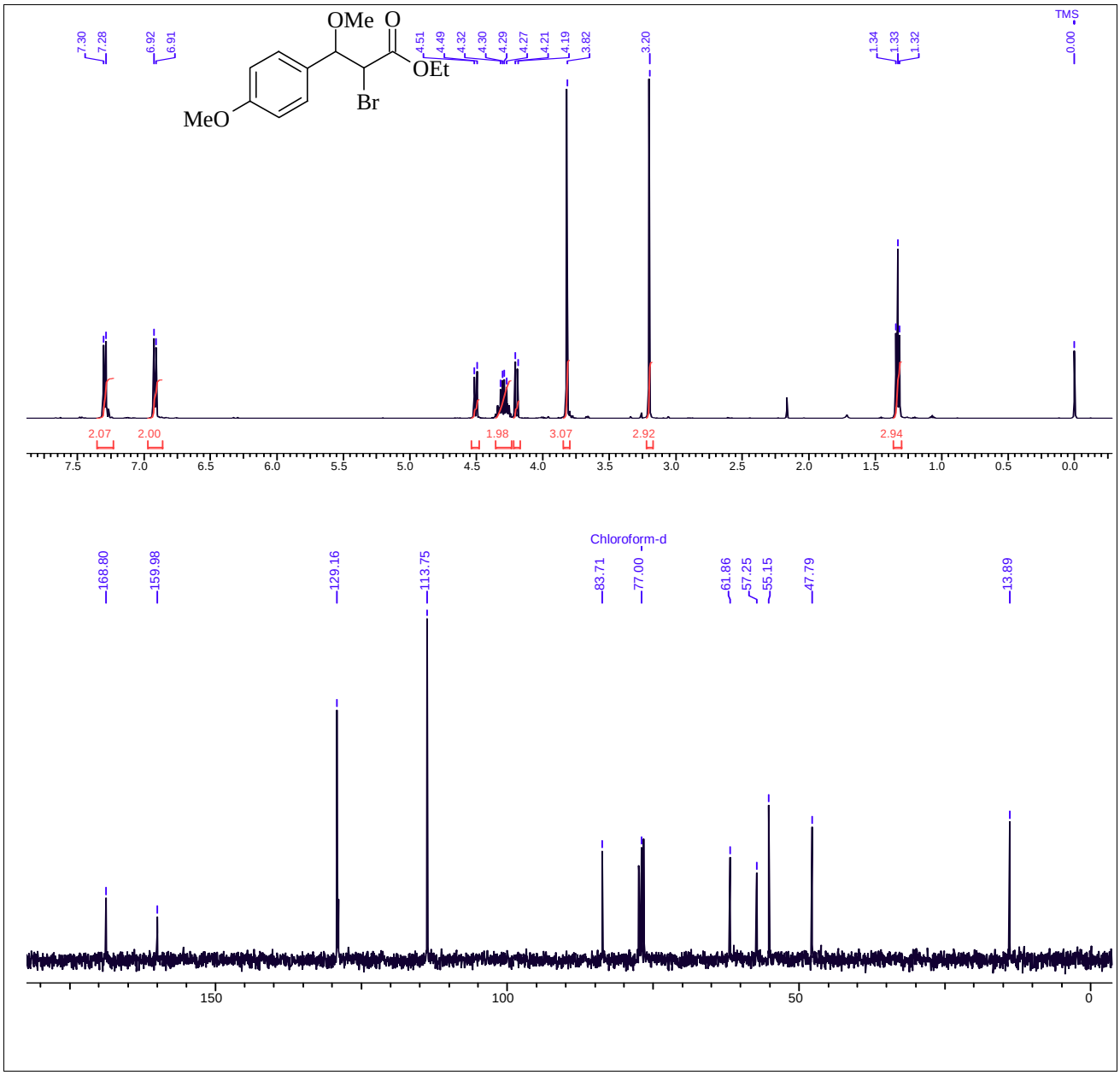
Br
TMS
Chloroform-d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O
OEt
Br
MeO
TMS
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7.28
6.92
6.91
4.51
4.49
4.32
4.30
4.29
4.27
4.21
4.19
3.82
3.20
1.34
1.33
1.32
0.00
2.07
2.00
1.98
3.07
2.92
2.94
Chloroform-d
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61.86
57.25
55.15
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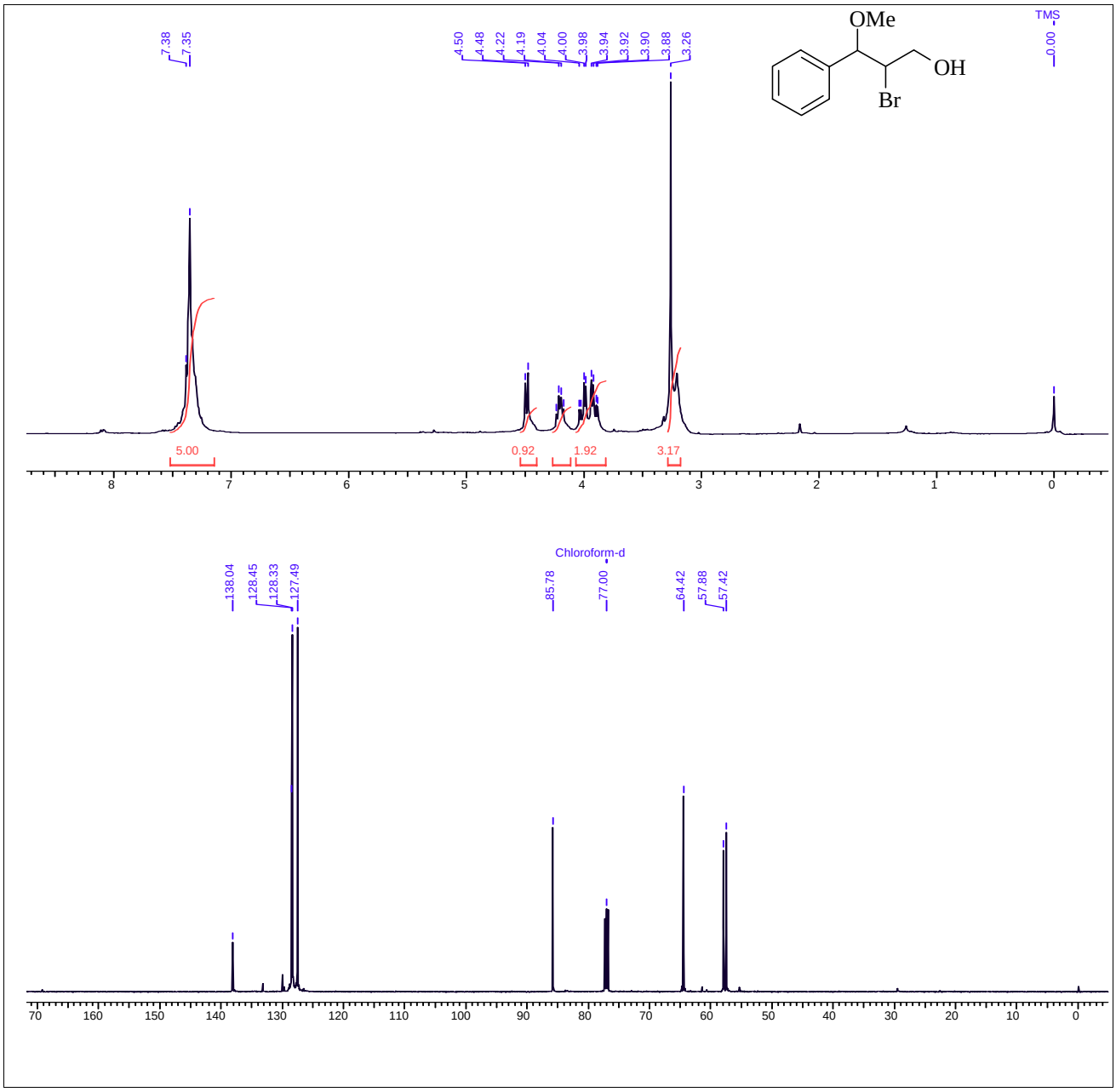
13.89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OH
Br
TMS
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4.50
4.48
4.22
4.19
4.04
4.00
3.98
3.94
3.92
3.90
3.88
3.26
5.00
0.92
1.92
3.17
8
7
6
5
4
3
2
1
0
Chloroform-d
138.04
128.45
128.33
127.49
85.78
77.00
64.42
57.88
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150
140
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70
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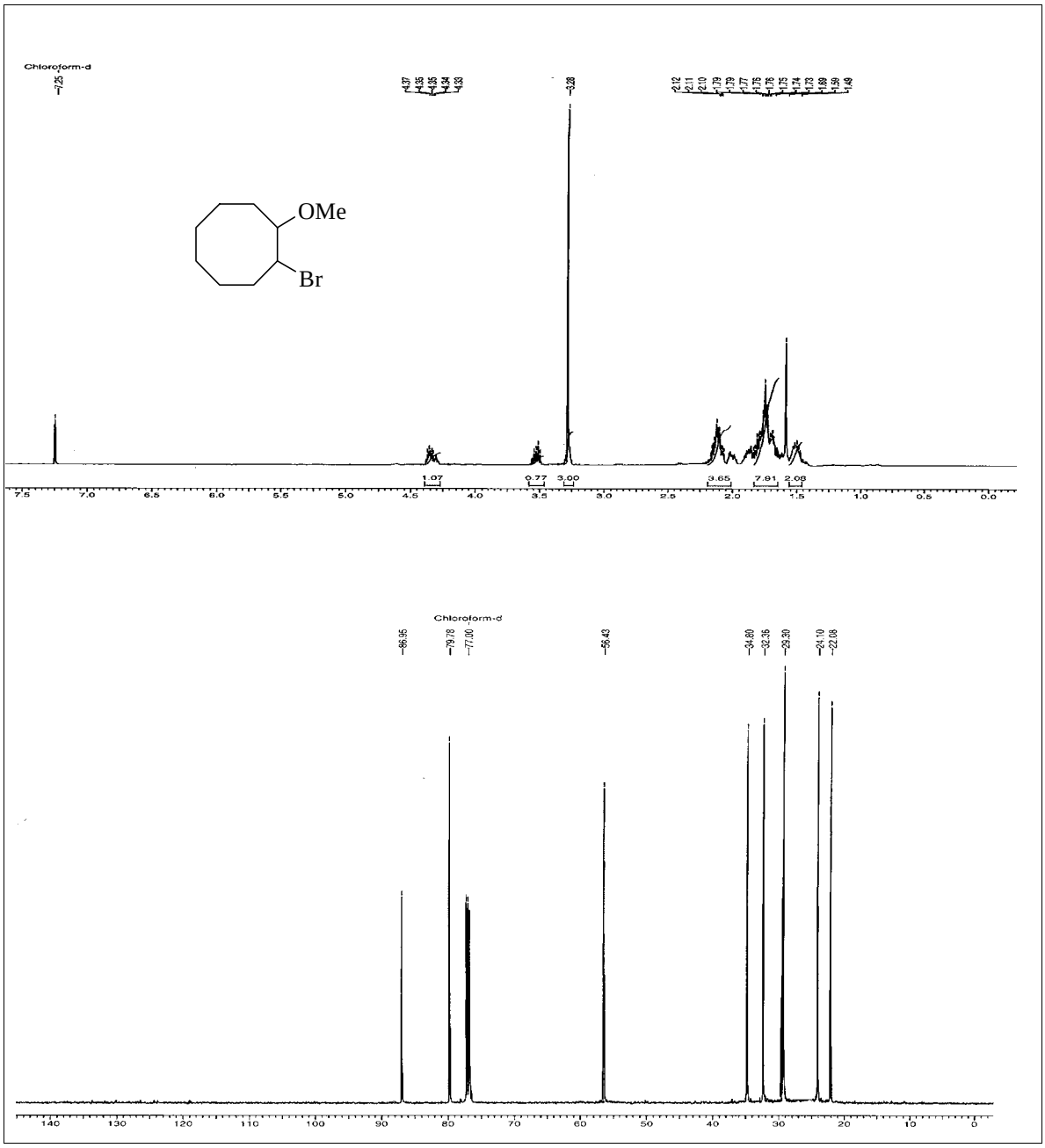
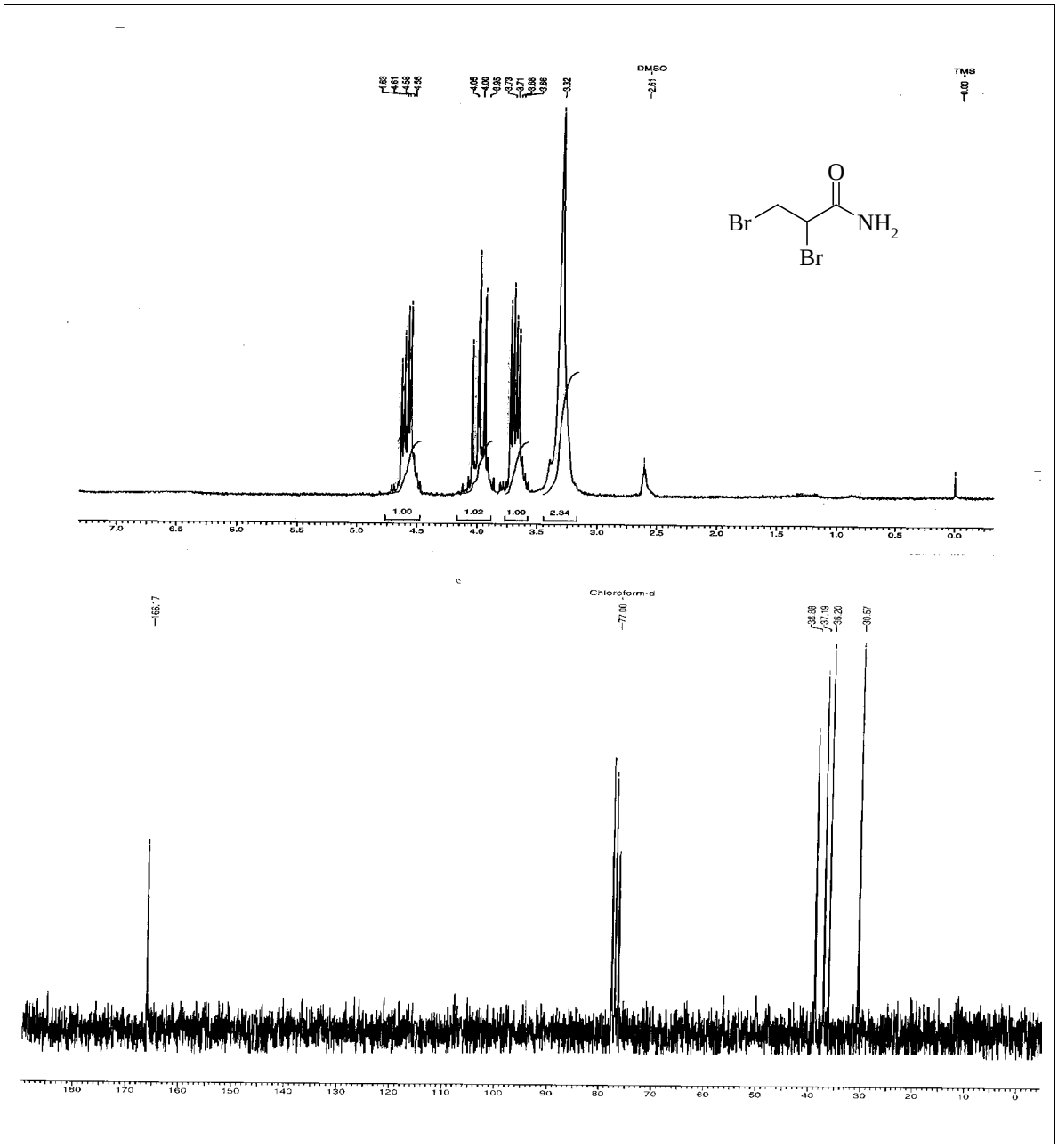

Chloroform-d
7.25
4.37
4.35
4.35
4.34
4.33
3.28
2.12
2.11
2.10
1.79
1.79
1.77
1.76
1.76
1.75
1.74
1.73
1.69
1.59
1.49
OMe
Br
1.07
0.77
3.00
3.65
7.91
2.08
Chloroform-d
66.95
79.78
77.00
56.43
34.80
32.35
29.39
24.10
22.08


DMSO
TMS
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4.58
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4.05
4.00
3.96
3.73
3.71
3.68
3.66
3.32
2.61
0.00
1.00
1.02
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2.34
O
Br
NH2
Br
Chloroform-d
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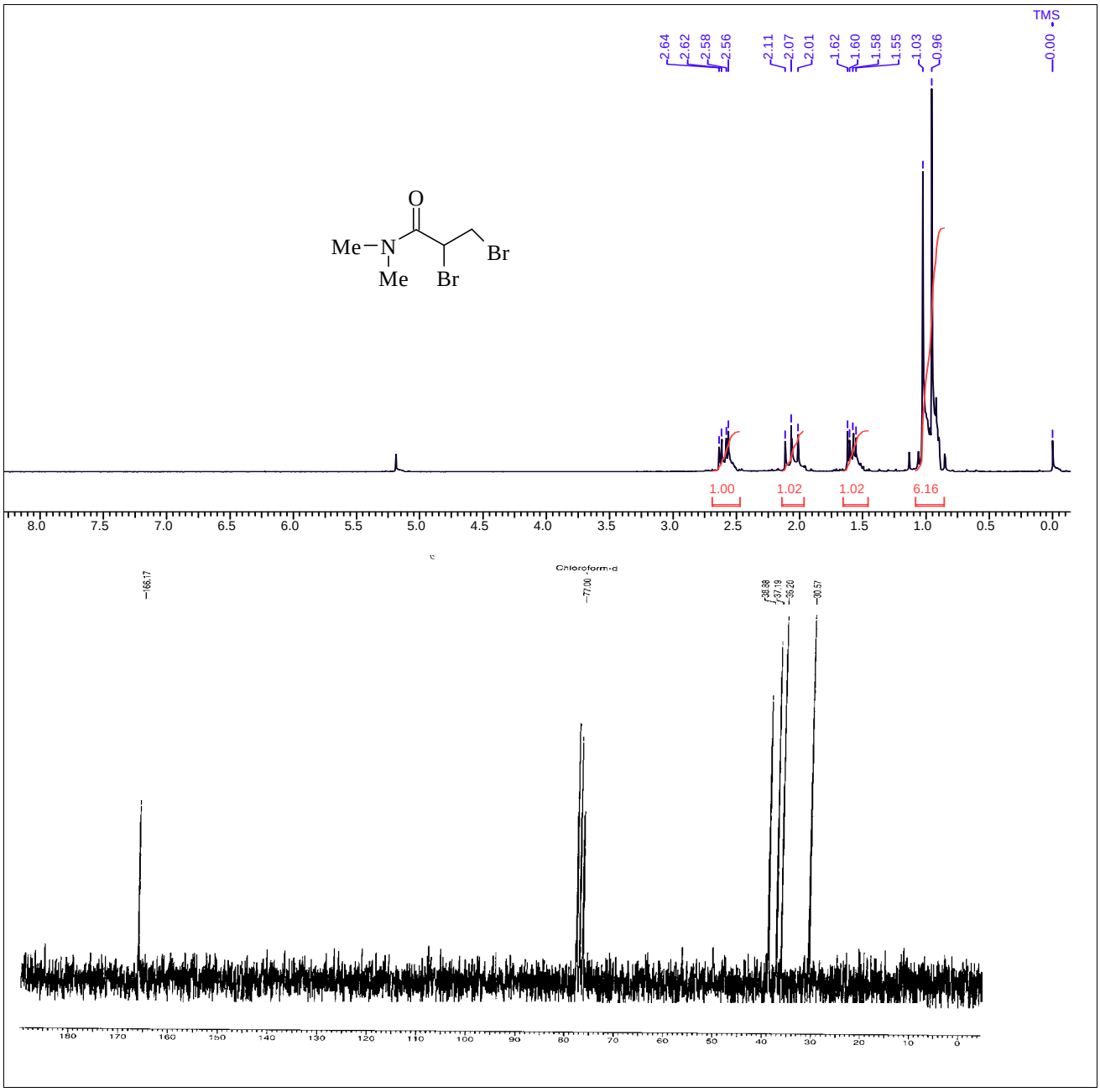
77.00
39.88
37.19
30.20
20.57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2.62
2.58
2.56
2.11
2.07
2.01
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1.60
1.58
1.55
1.03
0.96
0.00
O
Me
N
Me
Br
Br
1.00
1.02
1.02
6.16
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
166.17
Chloroform-d
77.00
39.38
37.19
35.20
30.57
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150
140
130
120
110
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70
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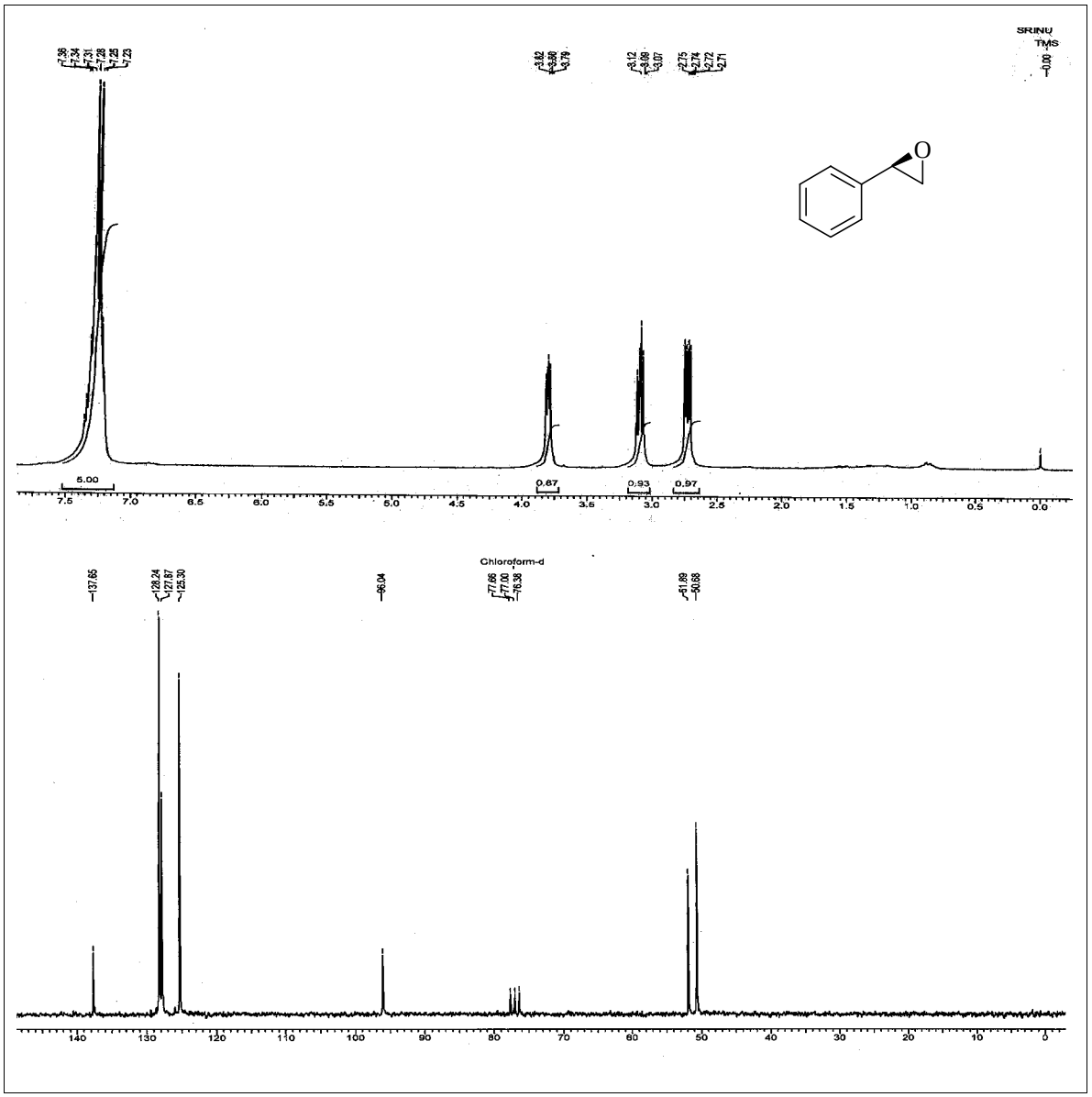
SRINU
TMS
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7.23
3.82
3.80
3.79
3.12
3.08
3.07
2.75
2.74
2.72
2.71
0.00
5.00
0.87
0.93
0.97
Chloroform-d
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128.24
127.87
125.30
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77.66
77.00
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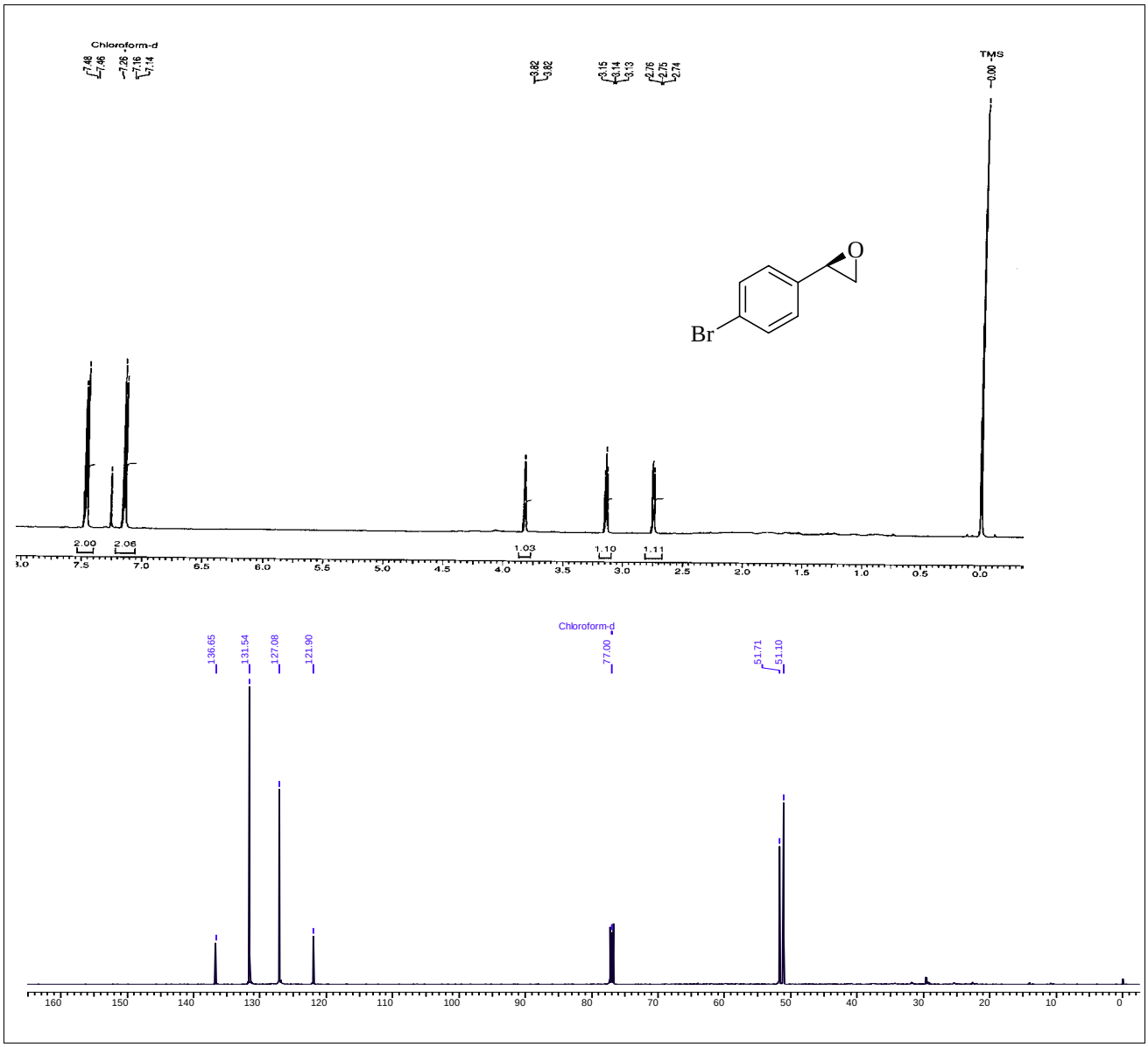
51.89
50.68


Chloroform-d
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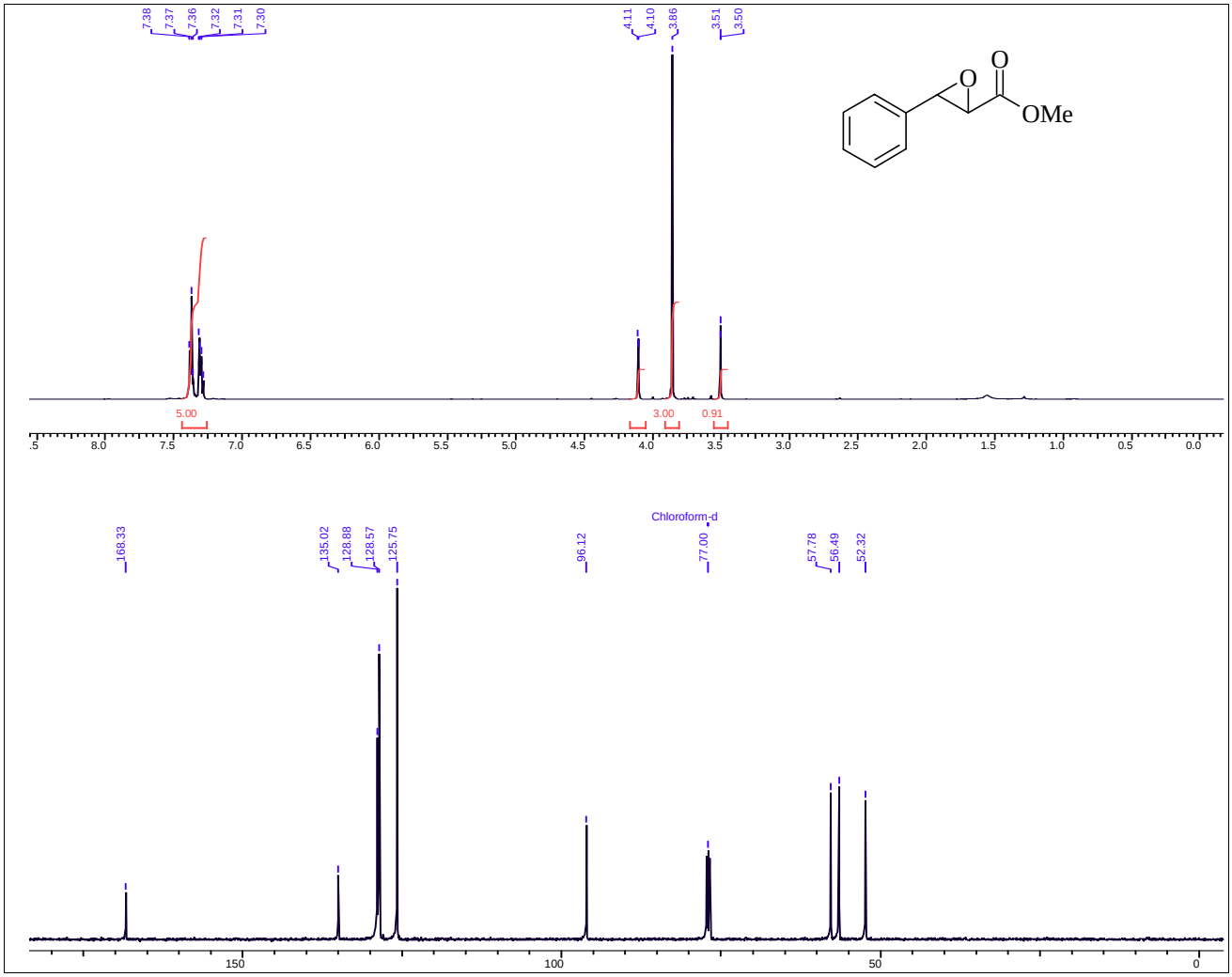
7.46
7.26
7.16
7.14
3.82
3.82
3.15
3.14
3.13
2.76
2.75
2.74
TMS
0.00
O
Br
2.00
2.06
1.03
1.10
1.11
Chloroform-d
136.65
131.54
127.08
121.90
77.00
51.71
51.10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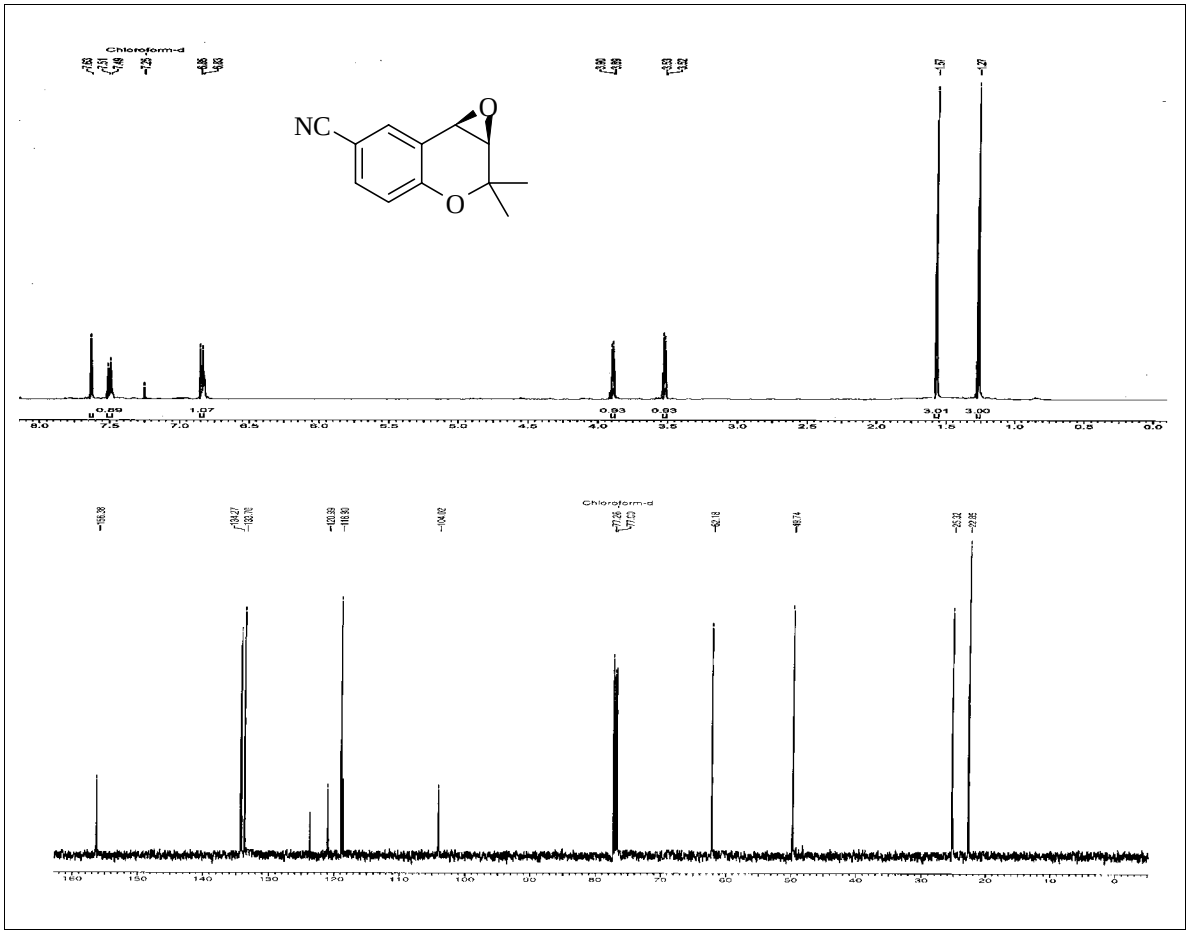
7.31
7.30
4.11
4.10
3.86
3.51
3.50
O
O
OMe
5.00
3.00
0.91
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
Chloroform-d
168.33
135.02
128.88
128.57
125.75
96.12
77.00
57.78
56.49
52.32
150
100
50
0


Chloroform-d
NC
O
O
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7.51
7.49
7.25
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4.56
4.55
3.53
3.52
1.57
1.27
0.89
1.07
0.93
0.93
3.01
3.00
Chloroform-d
156.38
134.27
132.76
120.39
119.90
104.62
77.26
77.01
76.77
62.18
48.74
25.32
23.85