Nickel-Catalyzed Kumada Cross-Coupling Reactions of Tertiary Alkylmagnesium Halides and Aryl Bromides/Triflates

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Supporting Information

General Reagent Information. BDH brand toluene was purchased from VWR. EMD brand Omnisolv THF (unstabilized) was also purchased from VWR. These solvents were transferred to separate 20L solvent-delivery kegs and vigorously purged with argon for 2 h. The solvents were further purified by passing them under argon pressure through two packed columns of neutral alumina (for THF and Et_2O) or through neutral alumina and copper (II) oxide (for toluene). *t*-BuMgCl (1M in THF) was purchased from Sigma-Aldrich. All NHC ligands were purchased form Strem. All nickel compounds with the exception of $NiCl_2(H_2O)_6$ (Fisher Scientific) were purchased from Strem. Grignard reagents were prepared from their corresponding *t*-alkyl chlorides or bromides using a literature procedure.[1] Molarities of Grignard reagents (typically between 0.8 M and 1.0 M) were determined using iodine titration.[2] Reagents and solvents were used as received unless otherwise noted. Flash chromatography was performed using Silicylcle silica gel (ultra pure grade).

General Analytical Information. All compounds were characterized by ^{1}H NMR and ^{13}C NMR spectroscopy. Copies of the ^{1}H and ^{13}C spectra for all new compounds can be found at the end of the Supporting Information. All previously unreported compounds were additionally characterized by high resolution MS. Nuclear Magnetic Resonance spectra were recorded on a Varian 300 or 500 MHz instrument. All ^{1}H NMR experiments are reported in δ units, parts per million (ppm), and were measured relative to the signals for residual chloroform (7.26 ppm). All ^{13}C NMR spectra are reported in ppm relative to deuterochloroform (77.23 ppm), and were obtained with ^{1}H decoupling. ^{19}F NMR spectra were recorded using $CFCl_3$ (0.00 ppm) as an internal reference. High resolution MS analyses were performed on an Agilent 6520 Q-TOF instrument. All GC analyses were performed on a Shimadzu GC-2010 gas chromatograph with an FID detector using a 25 m x 0.20 mm capillary column with cross-linked methyl siloxane as the stationary phase. All GC yields were calibrated using dodecane as an internal standard.

General Procedural Information.

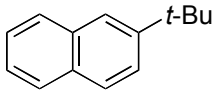
General procedure for the preparation of $NiCl_2•(H_2O)_n$

$NiCl_2•(H_2O)_6$ (2 g, 8.4 mmol) was finely ground using a mortar and pestle and transferred to a 50 mL round bottom flask containing a stirbar. The flask was place under high vacuum (0.5–1.0 torr) and immersed into a 100 ºC oil bath for 20 minutes. The contents of the flasks were vigorously mixed to ensure homogenous heating. The color of the nickel complex changed from bright green to yellow to yellow-orange as water was removed. The flask was removed from the oil bath and allowed to cool. Loss of water was determined by measuring mass lost during the heating process. In general, after 20 min, ca. 2 equiv water remained for each equivalent of $NiCl_2$. After loss of 4 equivalents of water, dehydration becomes slower. After having determined the extent of remaining hydration, the temperature of the oil bath was increased to 120 ºC. The flask was re-immersed in the heated oil bath for 5-10 min to generate $NiCl_2•(H_2O)_n$ where n = 1.4–1.7. $NiCl_2$ samples with different extents of hydration were achieved by sampling the heated reaction flask more frequently. In order to thoroughly dehydrate the $NiCl_2$, the temperature of the oil bath was increased to 150 ºC and the flask was heated for at least 2 h. In its anhydrous state, $NiCl_2$ is orange.

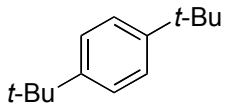
General procedure for the cross-coupling of aryl/vinyl halide/triflates and tertiary alkylmagnesium halides:

Because we are interested in developing methods of high operational simplicity as well as generality, we performed each of the reactions on the benchtop, using readily available disposable vials with screw-top septa. $NiCl_2•(H_2O)_{1.54}$ (16 mg, 0.1 mmol) and NHC ligand **18** (32 mg, 0.1 mmol) were weighed out on the benchtop in an oven-dried 10 mL screw top test tube with stir bar. The aryl bromide/triflate (1 mmol) was then added to the vial. The vial was sealed using a screw cap lined with a teflon septum. The reaction vial was evacuated and backfilled three times with argon using a needle attached to a vacuum manifold, and cooled to –10 ºC in a NaCl/ice slurry prior to the addition of the alkylmagnesium reagent. If the aryl bromide/triflate were a liquid, it was added via microsyringe after having backfilled the vial with argon. The tertiary alkylmagnesium halide (2.0 mmol) was then added via syringe under a positive pressure of argon. The vial was sealed with electrical tape and the reaction mixture was stirred for 90 minutes on the benchtop at –10 ºC with no additional argon pressure. The reaction mixture was quenched through the addition of ice chips, then poured into a separatory funnel containing saturated aqueous NH_4Cl (ca. 10 mL) and extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. The crude product was purified by column chromatography. Extent of isomerization can be easily determined via ^1H NMR spectroscopy by comparison of the intergral of the singlet from the *t*-butyl group (1.3-1.4 ppm, 9H) to the integral of the doublet from the benzylic

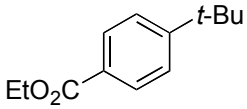
protons of the *i*-butyl group (2.4-2.5 ppm, 2H). The doublet from the methyls of the *i*-butyl group could additionally be used (ca. 0.8 ppm, 6H).



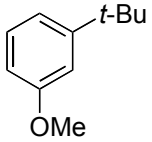
2-(tert-Butyl)naphthalene (Table 2, Row 1, Column 1). The general procedure was employed. A oily, white solid was isolated by column chromatography (96:4 Hex:Ether). This product consisted of 142 mg desired product (77% yield) and 15 mg reduction product. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.77-7.84 (m, 4H), 7.57 (dd, J = 8.4, 1.9 Hz, 1H), 7.39-7.47 (m, 2H), 1.43 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 133.6, 131.9, 128.2, 127.9, 127.6, 127.5, 126.0, 125.4, 125.0, 123.1, 35.0, 31.5 ppm.



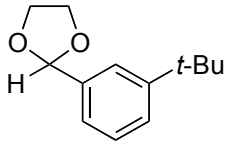
1,4-di-tert-Butylbenzene (Table 2, Row 1, Column 2). The general procedure was employed. A oily, white solid (147 mg, 77%) was isolated by column chromatography (98:2 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.34 (s, 4H), 1.34 (s, 18H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.2, 125.1, 34.5, 31.6 ppm.



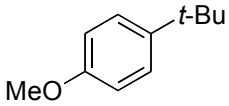
Ethyl 4-(tert-butyl)benzoate (Table 2, Row 1, Column 3). The general procedure was employed. A yellow liquid (167 mg, 81%) was isolated by column chromatography (80:20 Hex:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.98 (d, J = 8.6 Hz, 2H), 7.45 (d, J = 8.6 Hz, 2H), 4.36 (q, J = 7.2 Hz, 2H), 1.38 (t, J = 7.2 Hz, 3H), 1.34 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 166.7, 156.5, 129.5, 127.9, 125.4, 60.8, 35.1, 31.2, 14.5 ppm.



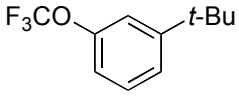
1-(tert-Butyl)-3-methoxybenzene (Table 2, Row 2, Column 1). The general procedure was employed. A colorless liquid (138 mg, 84%) was isolated by column chromatography (98:2 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.23 (t, J = 7.9 Hz, 1H), 6.99 (ddd, J = 7.8 Hz, 1.8 Hz, 0.9 Hz, 1H), 6.95 (m, 1H), 6.73 (ddd, J = 8.1 Hz, 2.6 Hz, 0.9 Hz, 1H), 3.81 (s, 3H), 1.31 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.6, 153.1, 129.1, 118.0, 112.2, 110.1, 55.2, 34.9, 31.5 ppm.



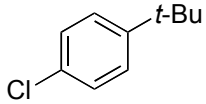
2-(3-(tert-butyl)phenyl)-1,3-dioxolane (Table 2, Row 2, Column 2). The general procedure was employed. The general procedure was employed. A colorless liquid (165 mg, 80%) was isolated by column chromatography (95:5 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.49 (m, 1H), 7.40 (dt, *J* = 7.2 Hz, 1.6 Hz, 1H), 7.31 (m, 2H), 5.81 (s, 1H), 4.10 (m, 4H), 1.33 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 151.4, 137.5, 128.3, 126.5, 123.7, 123.5, 104.3, 65.5, 34.9, 31.5 ppm. HRMS (ESI^+): $(M+H)^+$: 207.1380 (calc.); 207.1382 (found); diff (ppm) = 1.31.



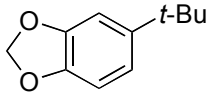
1-(tert-Butyl)-4-methoxybenzene (Table 2, Row 2, Column 3). The general procedure was employed. A colorless liquid (126 mg, 77%) was isolated by column chromatography (98:2 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.34 (d, *J* = 8.9 Hz, 2H), 6.88 (d, *J* = 8.9 Hz, 2H), 3.82 (s, 3H), 1.34 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.5, 143.5, 126.4, 113.5, 55.4, 34.2, 31.7 ppm.



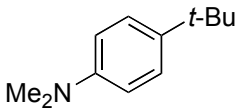
1-(tert-Butyl)-3-(trifluoromethoxy)benzene (Table 2, Row 3, Column 1). The general procedure was employed. A colorless liquid (153 mg, 70%) was isolated by column chromatography (98:2 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.32 (m, 2H), 7.22 (s, 1H), 7.05 (m, 1H), 1.33 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 153.9, 149.5, 129.4, 123.9, 118.4, 118.0, 35.1, 33.4 ppm. ^{19}F NMR (282 MHz, $CDCl_3$) δ: -58.2 ppm. HRMS ($APPI^+$): M^+: 218.0918 (calc.); 218.0915 (found); diff (ppm) = -1.49.



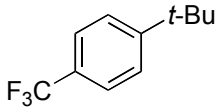
1-(tert-Butyl)-4-chlorobenzene (Table 2, Row 3, Column 2). The general procedure was employed. A pale yellow liquid (142 mg, 84%) was isolated by column chromatography (96:4 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.33 (d, *J* = 8.8 Hz, 2H), 7.27 (d, *J* = 8.8 Hz, 2H), 1.32 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 149.8, 131.3, 128.3, 126.9, 34.7, 31.5 ppm.



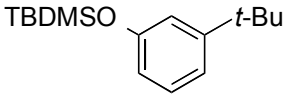
5-(tert-Butyl)benzo[d][1,3]dioxole (Table 2, Row 3, Column 3). The general procedure was employed. A purple liquid was isolated by column chromatography (90:10 Hex:EtOAc). This liquid consisted of 130 mg desired product (73% yield) and 17 mg reduction product. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.90 (d, J = 2.0 Hz, 1H), 6.82-6.84 (m, 2H), 5.92 (s, 2H), 1.28 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 147.6, 145.5, 145.3, 118.0, 107.8, 106.5, 100.9, 34.8, 31.8 ppm.



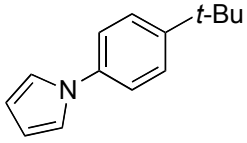
4-(tert-Butyl)-N,N-dimethylaniline (Table 2, Row 4, Column 1). The general procedure was employed. A pale yellow liquid was isolated by column chromatography (90:10 Hex:EtOAc). This liquid consisted of 135 mg desired product (76% yield) and 17 mg reduction product. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.39 (d, J = 8.8 Hz, 2H), 6.83 (d, J = 8.8 Hz, 2H), 3.02 (s, 6H), 1.42 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.7, 139.5, 126.0, 112.8, 41.2, 33.9, 31.7 ppm.



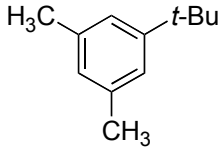
1-(tert-Butyl)-4-(trifluoromethyl)benzene (Table 2, Row 4, Column 2). The general procedure was employed. A colorless liquid (164 mg, 81%) was isolated by column chromatography (98:2 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.60 (d, J = 8.3 Hz, 2H), 7.54 (d, J = 8.3 Hz, 2H), 1.39 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 155.4, 128.1 (q, J = 32 Hz), 125.9, 125.2 (q, J = 4 Hz), 124.7 (q, J = 270 Hz), 35.2, 31.3 ppm.



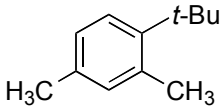
***tert*-butyl(3-(*tert*-butyl)phenoxy)dimethylsilane** (Table 2, Row 4, Column 3). The general procedure was employed. A colorless liquid was isolated by column chromatography (90:10 Hex:EtOAc). This liquid consisted of 177 mg desired product (67% yield) and 29 mg reduction product. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.20 (t, J = 7.9 Hz, 1H), 7.03 (ddd, J = 7.8 Hz, 1.9 Hz, 1.0 Hz, 1H), 6.93 (app. t, J = 2.1 Hz, 1H), 6.71 (ddd, J = 8.0 Hz, 2.4 Hz, 1.0 Hz, 1H), 1.36 (s, 9H), 1.06 (s, 9H), 0.27 (s, 6H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 155.6, 153.0, 129.0, 120.3, 118.4, 117.6, 117.2, 34.8, 31.6, 26.0, 18.5, 4.1 ppm.



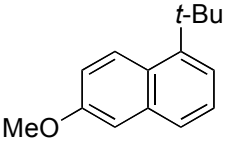
1-(4-(*tert*-butyl)phenyl)-1*H*-pyrrole (Table 2, Row 5, Column 1). The general procedure was employed. A pale yellow solid was isolated by column chromatography (98:2 Hex:Ether). Mp: 55-58 ºC. This solid consisted of 149 mg desired product (75% yield) and 29 mg reduction product. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.46 (d, J = 8.6 Hz, 2H), 7.34 (d, J = 8.6 Hz, 2H), 7.09 (t, J = 7.1 Hz, 2H), 6.36 (t, J = 7.1 Hz, 2H), 1.37 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.9, 129.7, 126.6, 120.5, 119.6, 110.2, 34.7, 31.6 ppm.



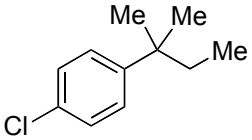
1-(tert-Butyl)-3,5-dimethylbenzene (Table 2, Row 6, Column 1). The general procedure was employed with the following modification: the reaction mixture was allowed to warm to rt and stir overnight (10h). A colorless liquid (138 mg, 85%) was isolated by column chromatography (98:2 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.21 (s, 2H), 7.02 (s, 1H), 2.52 (s, 6H), 1.51 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 151.3, 137.5, 127.3, 123.3, 34.6, 31.6, 21.7 ppm.



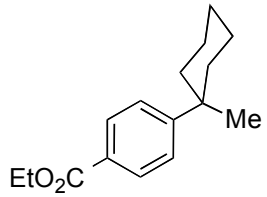
1-(tert-butyl)-2,4-dimethylbenzene (Table 2, Row 6, Column 2). The general procedure was employed. A colorless liquid (89 mg, 55%) was isolated by column chromatography (98:2 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.28 (d, J = 7.7 Hz, 1H), 6.96 (m, 2H), 2.53 (s, 3H), 2.29 (s, 3H), 1.41 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.2, 136.3, 135.4, 133.8, 126.5, 126.2, 42.4, 35.7, 31.2 ppm.



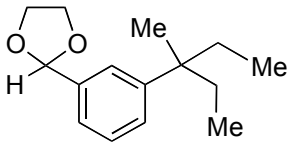
1-(tert-Butyl)-6-methoxynaphthalene (Table 2, Row 6, Column 3). The general procedure was employed. A white solid was isolated by column chromatography (96:4 Hex:Ether). Mp: 63-65 ºC. This white solid consisted of 184 mg desired product (86% yield) and 13 mg reduction product. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.70 (app. t, J = 8.0 Hz, 3H), 7.54 (dd, J = 8.6, 2.0 Hz, 1H), 7.11-7.14 (m, 2H), 3.92 (s, 3H), 1.41 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.5, 146.5, 132.8, 129.6, 129.0, 126.7, 125.5, 122.9, 118.7, 101.6, 55.5, 34.8, 31.5 ppm. HRMS (ESI^+): $(M+H)^+$: 215.1430 (calc.); 215.1432 (found); diff (ppm) = 0.70.



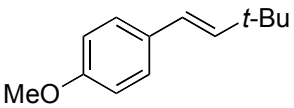
1-Chloro-4-(tert-pentyl)benzene (Table 3, Column 1). The general procedure was employed. A pale yellow liquid (133 mg, 73%) was isolated by column chromatography (99:1 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.29 (app. s, 4H), 1.66 (q, J = 7.3, 2H), 1.30 (s, 6H), 0.71 (t, J = 7.3, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 167.4, 153.5, 129.8, 127.9, 127.2, 52.1, 41.9, 31.1, 21.7, 12.3 ppm.



Ethyl 4-(1-methylcyclohexyl)benzoate (Table 3, Column 2). The general procedure was employed. A colorless liquid (187 mg, 76%) was isolated by column chromatography (90:10 Hex:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.99 (d, J = 8.7 Hz, 2H), 7.44 (d, J = 8.7 Hz, 2H), 4.37 (q, J = 7.1, 2H), 2.02 (m, 2H), 1.55-1.61 (m, 4H), 1.39-1.50 (m, 4H), 1.38 (t, J = 7.1, 3H), 1.19 (s, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 166.9, 155.6, 129.7, 127.7, 126.1, 60.9, 38.6, 37.5, 33.3, 26.5, 22.8, 14.6 ppm. HRMS (ESI^+): $(M+H)^+$: 247.1693 (calc.); 247.1696 (found); diff (ppm) = 1.22.



2-(3-(3-methylpentan-3-yl)phenyl)-1,3-dioxolane (Table 3, Column 3). The general procedure was employed with the following modification: the reaction mixture was allowed to warm to rt and stir overnight (10h). An oily, colorless solid (121 mg, 51%) was isolated by column chromatography (98:2 Hex:Ether). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.39 (s, 1H), 7.28-7.34 (m, 3H), 5.81 (s, 3H), 4.13-4.17 (m, 2H), 4.02-4.06 (m, 2H), 1.72-1.80 (m, 2H), 1.54-1.62 (m, 2H), 1.27 (s, 3H), 0.68 (t, J = 7.4 Hz, 6H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.0, 137.4, 128.1, 127.7, 125.0, 123.5, 104.4, 65.4, 41.5, 38.6, 35.3, 23.0, 8.9 ppm. HRMS (ESI^+): $(M+H)^+$: 235.1693 (calc.); 235.1691 (found); diff (ppm) = -0.65.

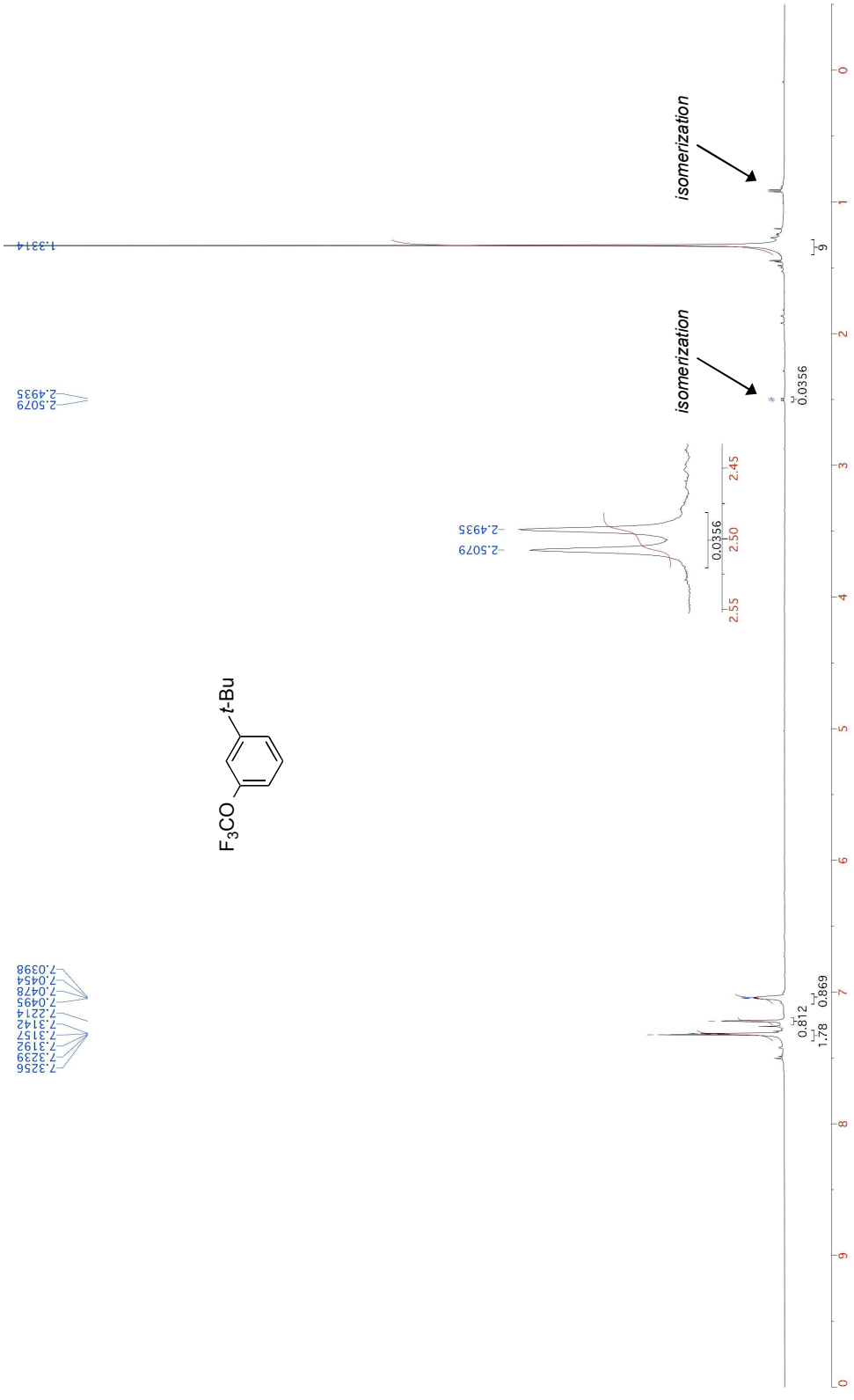


(E)-1-(3,3-dimethylbut-1-en-1-yl)-4-methoxybenzene (Figure 4). The general procedure was employed. A white solid (Cl: 158 mg, 83%; Br: 133 mg, 70%) was

isolated by column chromatography (98:2 Hex:Ether). Mp: 48-50 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.30 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.25 (d, *J* = 16 Hz, 1H), 6.12 (d, *J* = 16 Hz, 1H), 3.80 (s, 3H), 1.11 (s, 9H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.8, 140.0, 131.1, 127.3, 124.0, 114.1, 55.5, 33.4, 29.9 ppm.

References

1) Dragoli, D. R.; Burdett, M. T.; Ellman, J. A. *J. Am. Chem. Soc.* **2001**, *123*, 10127.
2) Krasovskiy, A.; Knochel, P. *Synthesis* **2006**, 5, 890.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isomerization
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7.3157
7.3142
7.2214
7.0495
7.0478
7.0454
7.0398
2.5079
2.4935
1.3314
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2.5079
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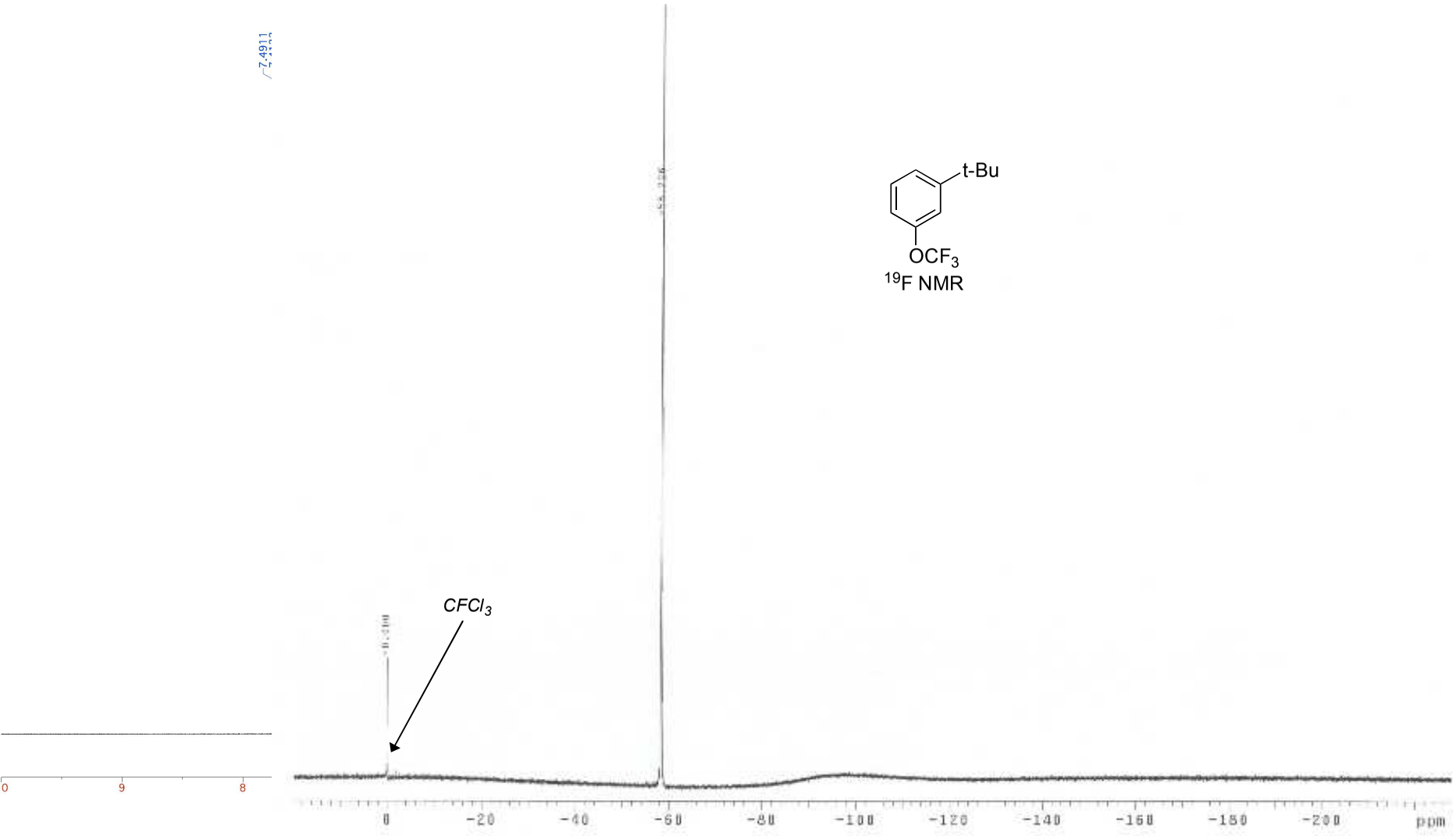
2.45
2.50
2.55
0.0356
0.812
1.78
0.869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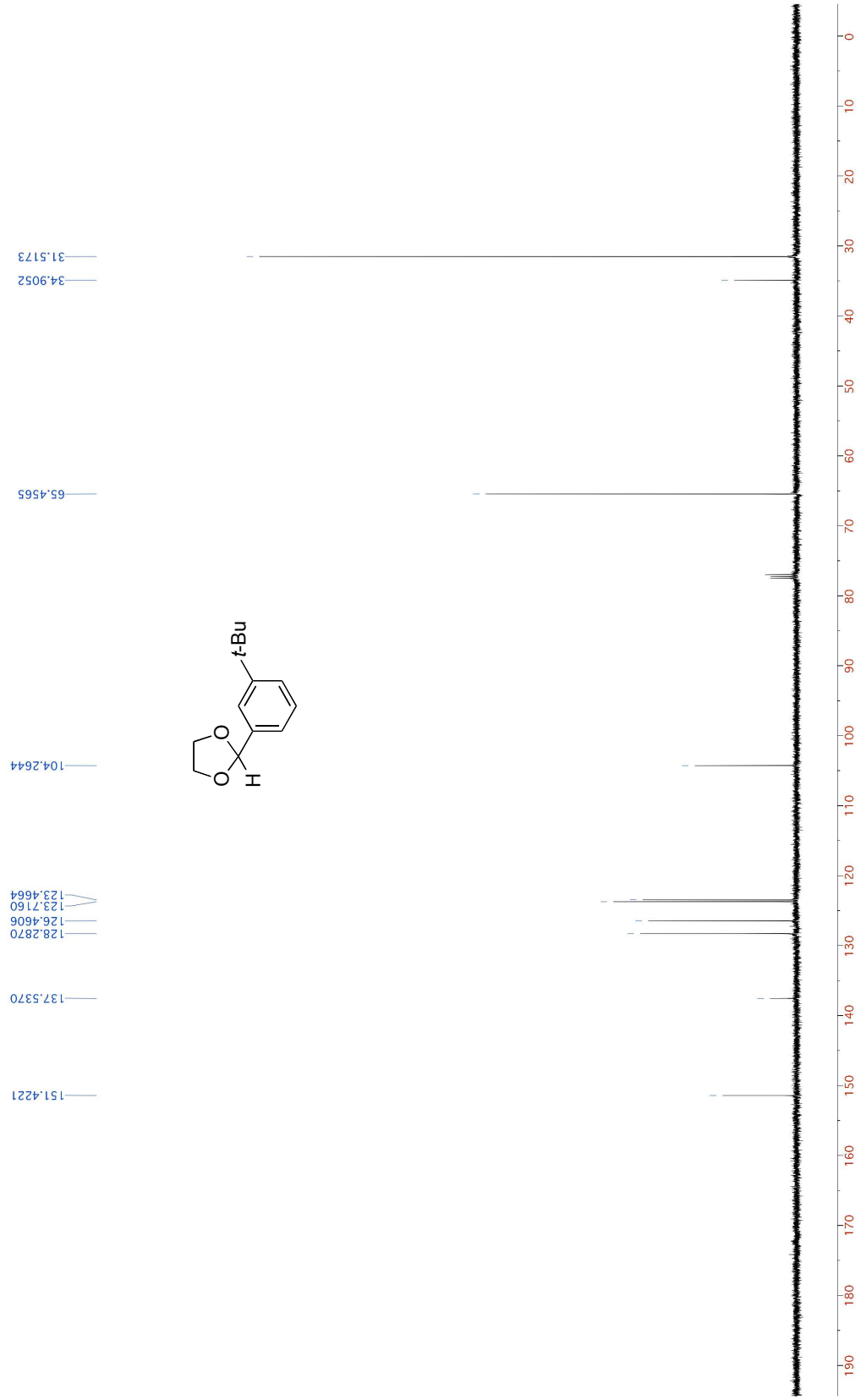
-60
-80
-100
-120
-140
-160
-180
-200
ppm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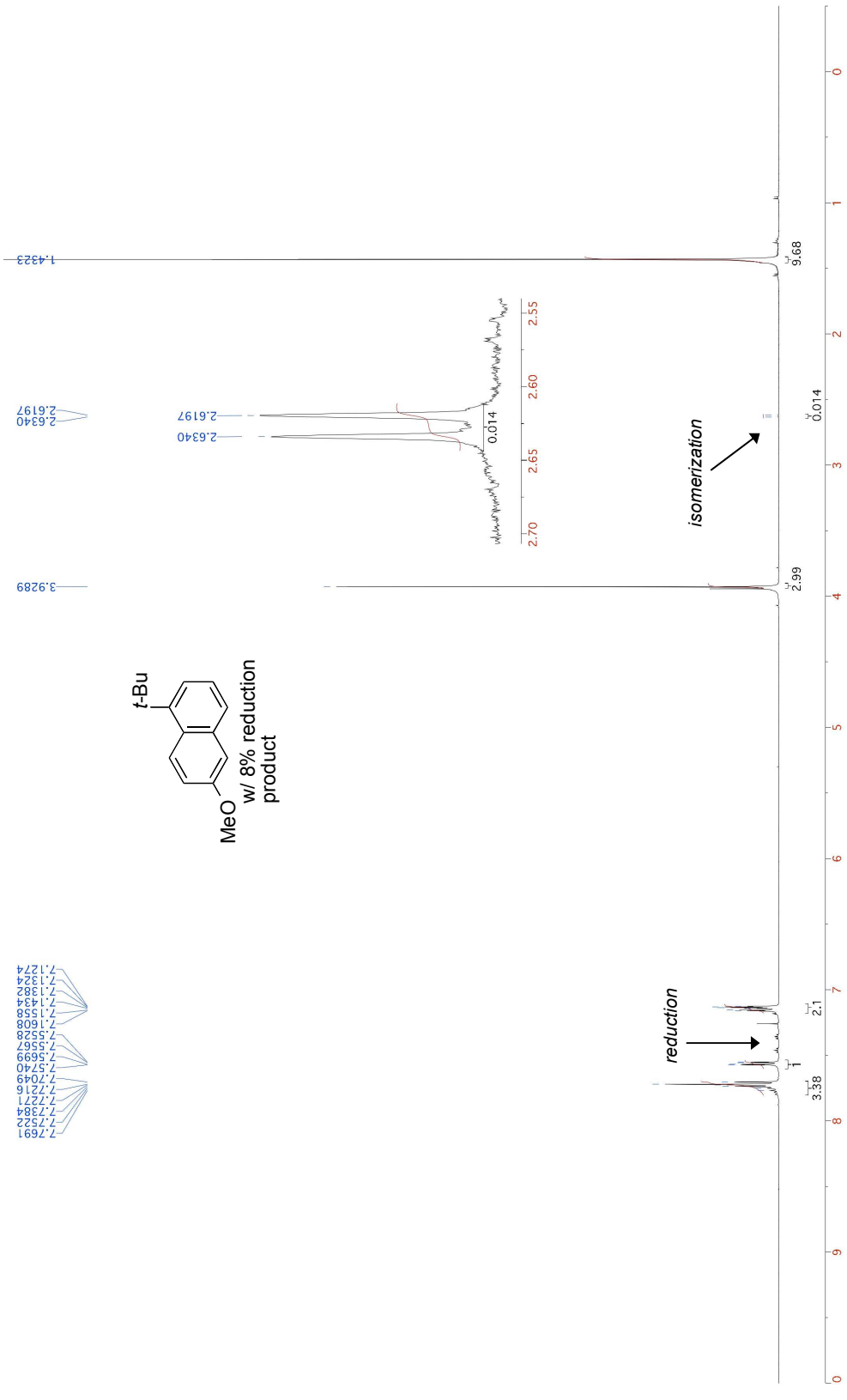
t-Bu
O
O
H
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190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7.5528
7.1608
7.1558
7.1434
7.1382
7.1324
7.1274
3.9289
2.6340
2.6197
1.4323
t-Bu
MeO
w/ 8% reduction product
reduction
isomerization
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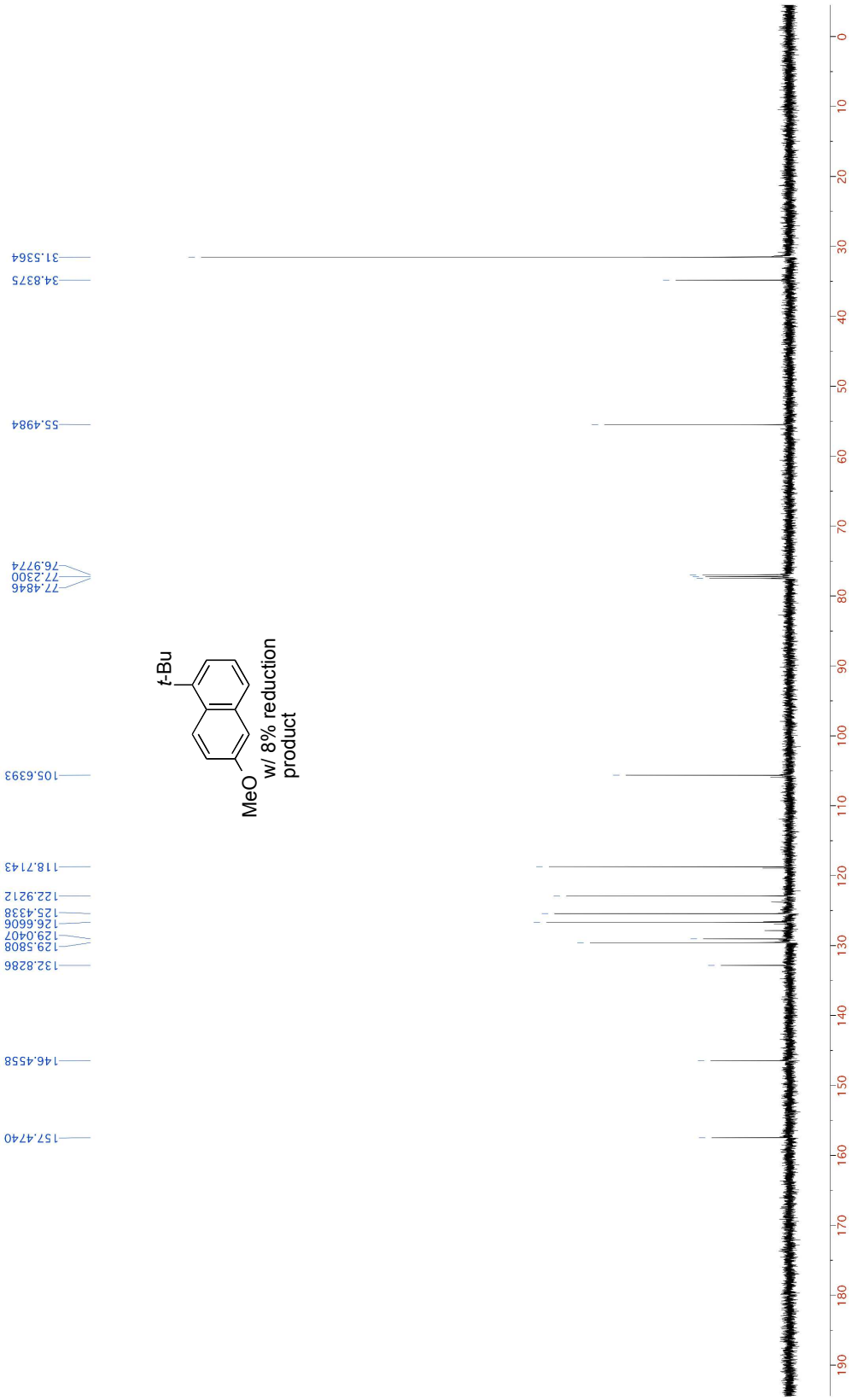
2.99
0.014
9.68
2.70
2.65
2.60
2.55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129.0407
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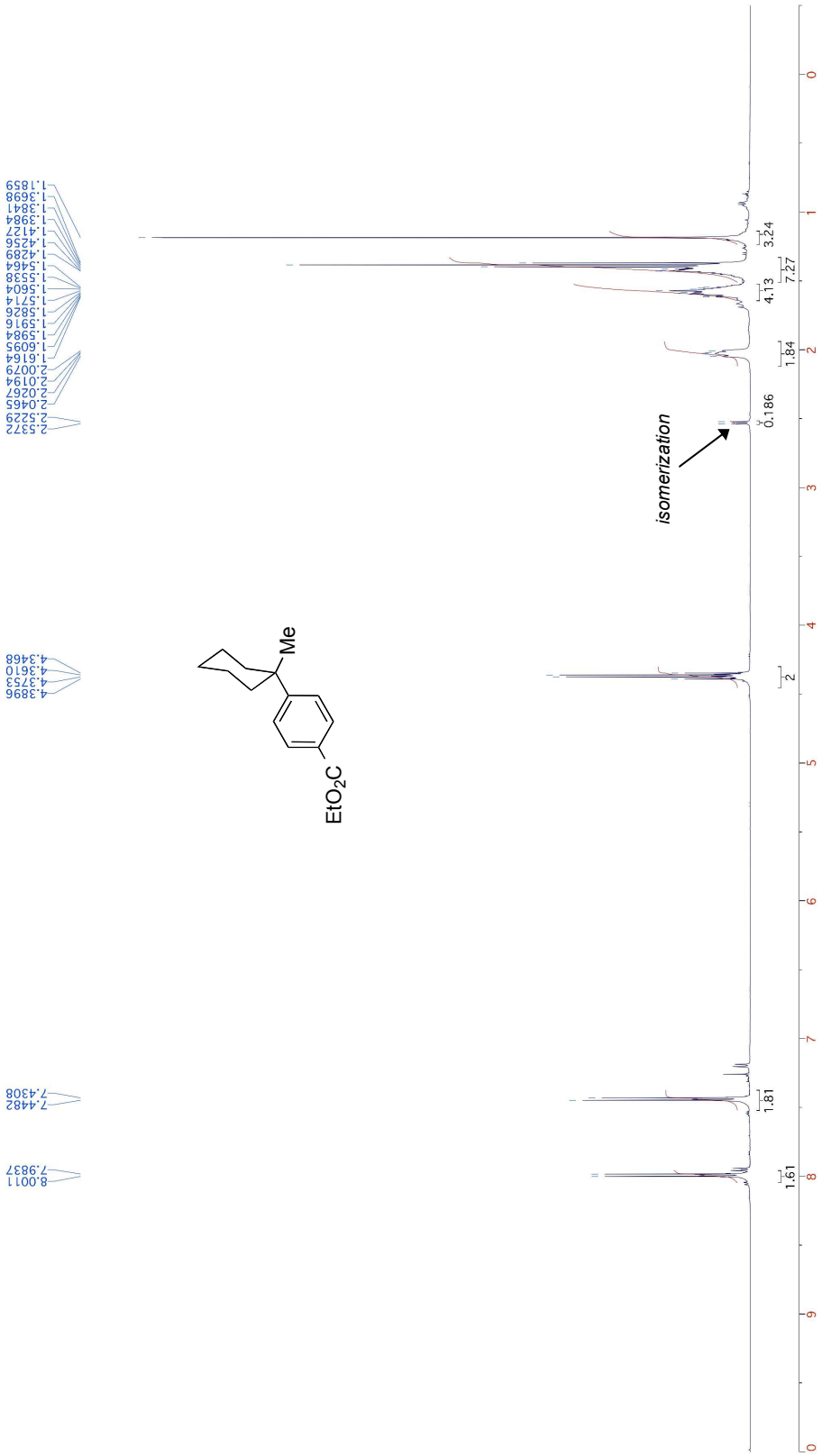
146.4558
157.4740
t-Bu
MeO
w/ 8% reduction product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1.5984
1.6095
1.6164
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4.3896
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7.4482
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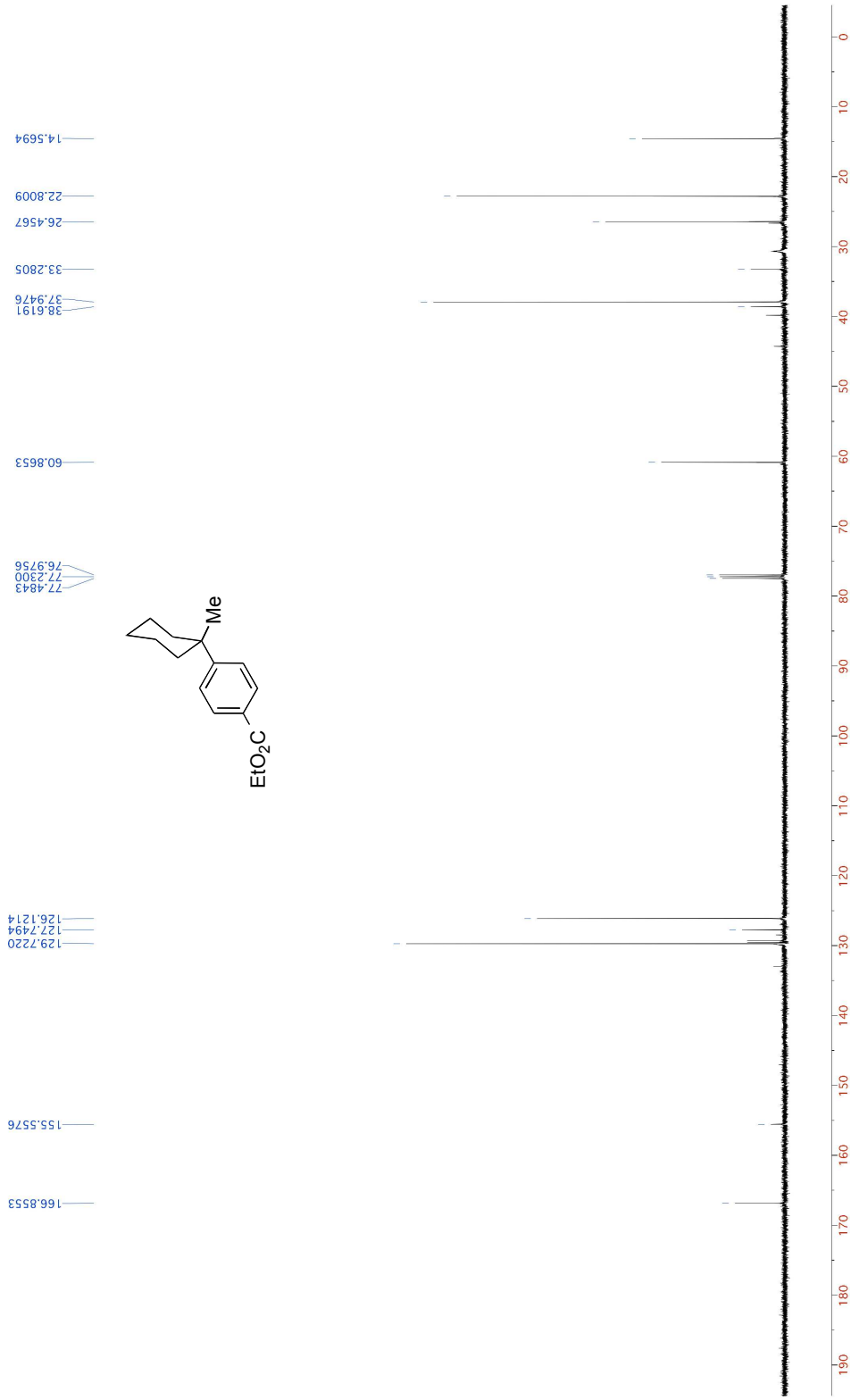
EtO2C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127.7494
129.7220
155.5576
166.8553
Me
EtO2C
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80
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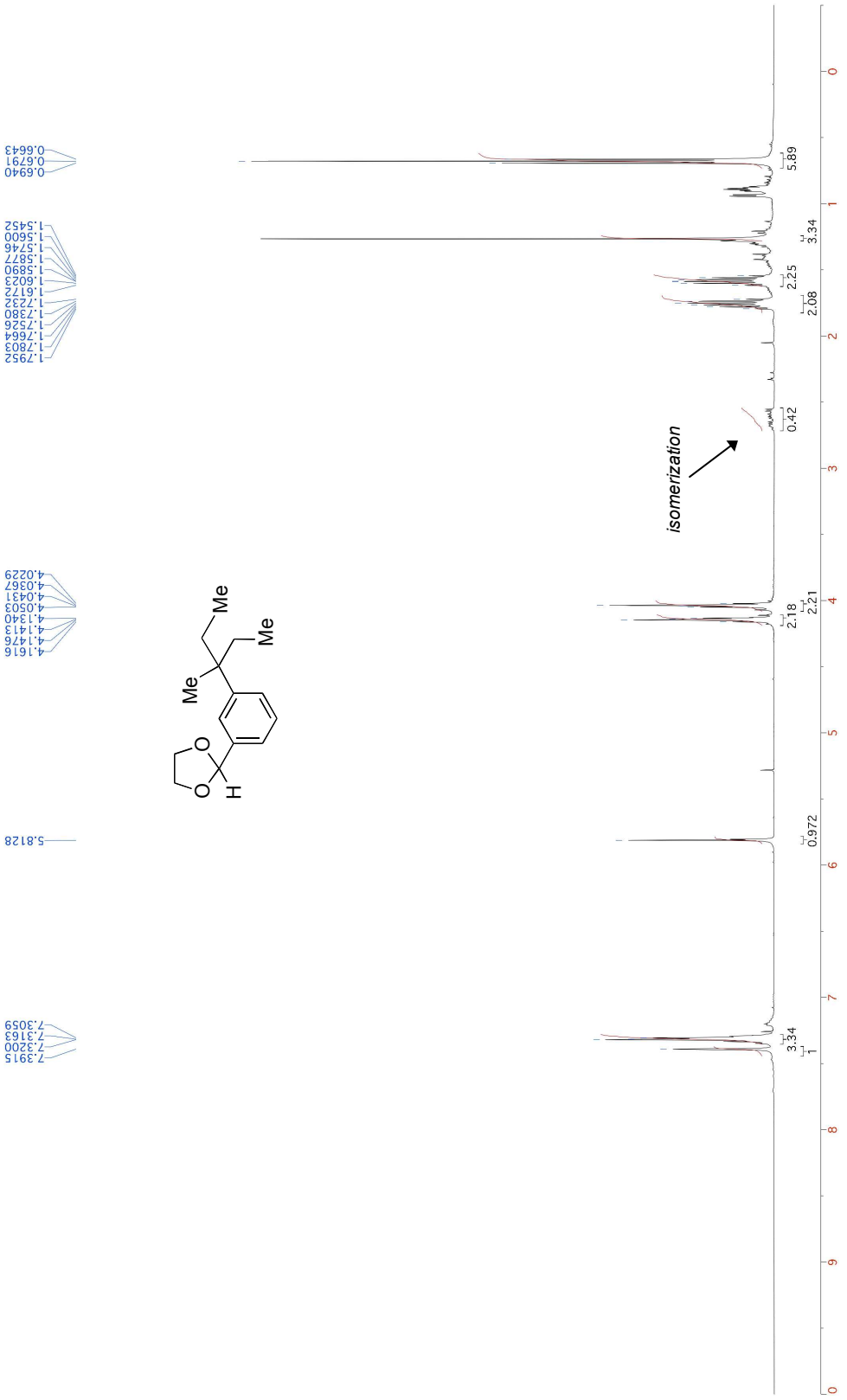
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Me
Me
O
O
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1.6023
1.6172
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1.7380
1.7526
1.7664
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1.7952
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4.0367
4.0431
4.0503
4.1340
4.1413
4.1476
4.1616
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7.3059
7.3163
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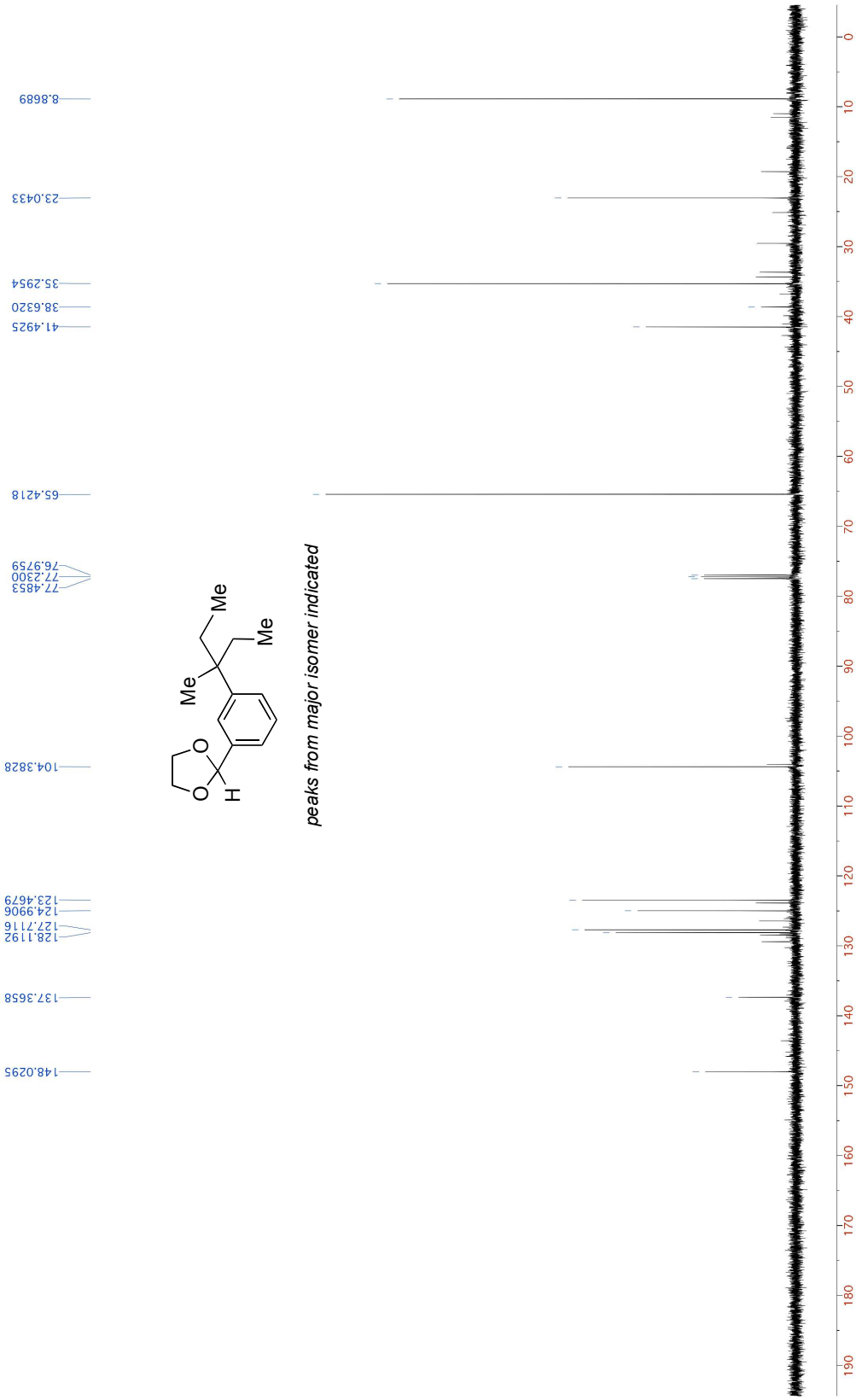
3.34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123.4679
124.9906
127.7116
128.1192
137.3658
148.0295
peaks from major isomer indicated
Me
Me
Me
O
O
H
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190