Supporting Information

Chemoselective Catalytic Dehydrogenative Cross Coupling of 2-Acylimidazoles: Mechanistic Investigations and Synthetic Scope

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1. General

All reactions were carried out using heat gun dried glassware under a positive pressure of dry argon unless otherwise noted. Catalytic reactions were run under argon atmosphere. Air- and moisture-sensitive liquids were transferred via a syringe and a stainless-steel needle. Reactions were magnetically stirred and monitored by thin layer chromatography using Merck Silica Gel 60 F254 plates. All work-up and purification procedures were carried out with reagent-grade solvents under ambient atmosphere. Flash chromatography was performed using silica gel 60N (spherical neutral, particle size 40–50μm) purchased from Kanto Chemical Co. Ltd.

2. Instrumentation

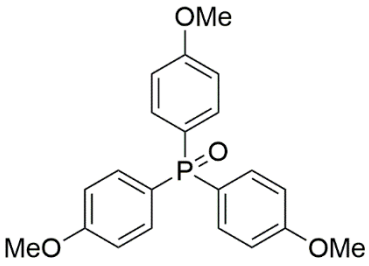
NMR was recorded on 500 MHz Bruker Advanced III. Chemical shifts for proton are reported in parts per million downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent ($CDCl_3$: δ 7.26 ppm). For ^{13}C NMR, chemical shifts were reported in the scale relative to NMR solvent ($CDCl_3$: δ 77.0 ppm) as an internal reference. NMR data are reported as follows: chemical shifts, multiplicity (s: singlet, d: doublet, dd: doublet of doublets, dt: doublet of triplets, dq: doublet of quarteds, t: triplet, td: triplet of doublets, tt: triplet of triplets, q: quartet, quin: quintet, sex: sextet, sep: septet, m: multiplet, br: broad signal), coupling constant (Hz), and integration. Infrared (IR) spectra were recorded on with Shimadzu IR Affinity-1S. High-resolution mass spectroscopy (HRMS) was obtained with Waters ACQUITY UPLC®–LCT-Premier™ XE system and Bruker MicrOTOF II. Ultraviolet and visible absorption spectrum was recorded on with Shimadzu UV-2600.

3. Materials

Toluene (dehydrated –super –) and acetonitrile (dehydrated –super –) were purchased from KANTO Chemical Co., Inc. and stored in a dry box. $FeCl_3$ (sublimed grade, ≥99.9% trace metals basis) and $FeCl_2$ (99.99% trace metals basis) were purchased from Aldrich and used as received and stored in a dry box. Chlorobenzene was dried over MS4A. DTBP was purchased from TCI and used as received. All other commercially available reagents, including alkylarenes, were used as received.

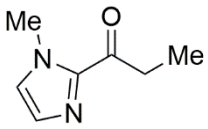
4. Substrate Syntheses and Characterization

Procedure for synthesis of tris(4-methoxyphenyl)phosphine oxide (L11)[1]



To a solution of tris(4-methoxyphenyl)phosphine (5.0 mmol, 1.0 equiv.) in THF (25 mL, 0.20 M) was added slowly 35% hydrogen peroxide in water (5.0 equiv.) at 0 °C. The cooling bath was removed and the reaction mixture was stirred 4 h at room temperature. After removal of solvent under reduced pressure, the resulting residue was purified by recrystallization to afford **L11**. : CAS Registry Number 803-17-8; (White solid, 84% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59-7.55 (m, 6H, Ar*H*), 6.96-6.94 (m, 6H, Ar*H*), 3.84 (s, 9H, OC*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.3 (d, *J* = 2.6 Hz), 133.9 (d, *J* = 11.1 Hz), 124.6 (d, *J* = 110.9 Hz), 114.0 (d, *J* = 13.1 Hz), 55.3.

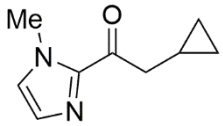
General procedure A for the synthesis of 2-acylimidazoles (1a-1h, 1m)[2-4]: To a solution of 1-methylimidazole (1.0 equiv.) in dry THF (0.70 M) under argon atmosphere was added slowly 2.6 M *n*-BuLi in *n*-hexane (1.1 equiv.) at –78 °C. The cooling bath was removed and the reaction mixture was stirred at room temperature. After 0.5 h, the mixture was again cooled to –78 °C and indicated ester or Weinreb amide was added. The cooling bath was removed and the reaction mixture was for indicated time at room temperature. After it was quenched by 1 M HCl aq, sat. $NaHCO_3$ aq was added to the resultant residue and extracted with CH_2Cl_2. After removal of solvent under reduced pressure, the residue was purified by silica gel flash chromatography to afford the desired 2-acylimidazole, which was used without further purification unless otherwise noted.



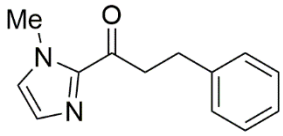
1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (1a): CAS Registry Number 138536-16-0; (Colorless liquid, 43% yield); Following the general procedure A using methyl propionate (3.0 equiv.) and stirring for 14 h, performing the distillation after silica gel flash chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 4.01 (s, 3H, NC*H*$_3$), 3.15 (q, *J* = 6.0 Hz, 2H, COC*H*$_2$), 1.20 (t, *J* = 6.0 Hz, 3H, CH_2C*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.9, 143.0, 128.9, 126.7, 36.2,

32.3, 8.1.

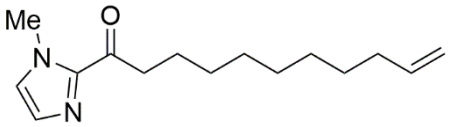
Procedure for the synthesis of 2-cyclopropyl-1-(1-methyl-1*H*-imidazol-2-yl)ethan-1-one (1b)[5,6]



To a solution of 1-methylimidazole (17 mmol, 1.0 equiv.) in dry THF (26 mL, 0.70 M) under argon atmosphere was added slowly 2.6 M *n*-BuLi in *n*-hexane (18 mmol, 1.1 equiv.) at –78°C. After 0.5 h, 2-cyclopropyl-*N*-methoxy-*N*-methylacetamide (18 mmol, 1.1 equiv.) was added and stirring for 17 h. After it was quenched by acetic acid, sat. $NaHCO_3$ aq was added to the resultant mixture and extracted with CH_2Cl_2. After removal of solvent under reduced pressure, the resulting residue was purified by silica gel flash chromatography to afford **1b**. : CAS Registry Number 1509675-93-7; (Colorless liquid, 38% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.12 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 4.02 (s, 3H, NCH_3), 3.01 (d, *J* = 7.0 Hz, 2H, COCH_2), 1.58-1.14 (m, 1H, CH(CH_2)$_2$), 0.58-0.54 (m, 2H, CH(CH_2)$_2$), 0.23-0.22 (m, 2H, CH(CH_2)$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.9, 143.1, 129.0, 126.9, 44.1, 36.2, 6.5, 4.3; IR (neat) 1670, 1460, 1402, 1381, 1287, 1223, 1155, 1016, 962, 935, 912, 831, 804, 768, 691 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_9H_{12}N_2O$ (M + H)$^+$ 165.1022, found 165.1050.

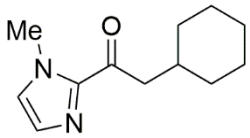


1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one (1c): CAS Registry Number 1179358-90-7; (Pale yellow liquid, 22% yield); Following the general procedure A using ethyl 3-phenylpropionate (1.3 equiv.) and stirring for 5 h, performing the distillation after silica gel flash chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29-7.26 (m, 4H, Ar*H*), 7.19-7.18 (m, 1H, Ar*H*), 7.12 (s, 1H, *imidazole*), 7.01 (s, 1H, *imidazole*), 4.00 (s, 3H, NCH_3), 3.48 (t, *J* = 7.5 Hz, 2H, COCH_2), 3.04 (t, *J* = 7.5 Hz, 2H, ArCH_2); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.1, 143.0, 141.1, 129.1, 128.5, 128.4, 126.9, 126.0, 40.4, 36.2, 30.0.



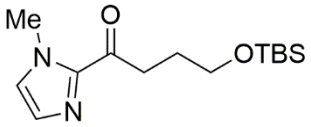
1-(1-methyl-1*H*-imidazol-2-yl)undec-10-en-1-one (1d): (Colorless liquid, 14% yield); Following the general procedure A using methyl 10-undecenoate (1.3 equiv.) and stirring for 5 h, performing the distillation after silica gel flash chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13 (s, 1H, *imidazole*),

7.01 (s, 1H, *imidazole*), 5.85-5.77 (m, 1H, C*H*=CH_2), 5.01-4.91 (m, 2H, CH=CH_2), 4.00 (s, 3H, NCH_3), 3.11 (t, *J* = 7.5 Hz, 2H, COCH_2), 2.05-2.01 (m, 2H, CH_2CH=CH_2), 1.73-1.67 (m, 2H, $COCH_2CH_2$), 1.38-1.29 (m, 10H, $(CH_2)_5CH_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.5, 143.2, 139.3, 128.9, 126.8, 114.1, 39.1, 36.2, 33.8, 29.4, 29.4, 29.3, 29.1, 28.9, 24.3; IR (neat) 2924, 2853, 2359, 2342, 1672, 1639, 1464, 1406, 1288, 1153, 993, 976, 912, 764, 696 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{15}H_{24}N_2O$ $(M + H)^+$ 249.1961, found 249.1967.



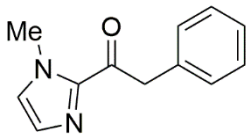
2-cyclohexyl-1-(1-methyl-1*H*-imidazol-2-yl)ethan-1-one (1e): (White solid, 26% yield); Following the general procedure A using methyl cyclohexylacetate (1.3 equiv.) and stirring for 22 h. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13 (s, 1H, *imidazole*), 7.01 (s, 1H, *imidazole*), 4.00 (s, 3H, NCH_3), 2.99 (d, *J* = 7.0 Hz, 2H, COCH_2), 2.03-1.95 (m, 1H, *cyclohexyl*), 1.75-1.62 (m, 5H, *cyclohexyl*), 1.33-1.24 (m, 2H, *cyclohexyl*), 1.21-1.14 (m, 1H, *cyclohexyl*), 1.09-1.01 (m, 2H, *cyclohexyl*); ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.1, 143.5, 128.9, 126.8, 46.5, 36.3, 34.5, 33.3, 26.3, 26.2; IR (neat) 3075, 2914, 2849, 1674, 1474, 1414, 1402, 993, 955, 920, 800, 754, 702, 691 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{12}H_{18}N_2O$ $(M + H)^+$ 207.1492, found 207.1509.

Procedure for the synthesis of 4-((*tert*-butyldimethylsilyl)oxy)-1-(1-methyl-1*H*-imidazol-2-yl)butan-1-one (1f)

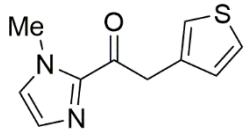


To a solution of 1-methylimidazole (0.10 mol, 1.0 equiv.) in dry THF (143 mL, 0.70 M) under argon atmosphere was added slowly 2.6 M *n*-BuLi in *n*-hexane (1.1 equiv.) at –78 °C. The cooling bath was removed and the reaction mixture was stirred at room temperature. After 0.5 h, the mixture was again cooled to –78°C and γ-butyrolactone (0.13 mol, 1.3 equiv.) was added. The cooling bath was removed and the reaction mixture was stirred for 3 h at room temperature. After it was quenched by 1 M HCl aq, sat. $NaHCO_3$ aq was added to the resultant mixture and extracted with CH_2Cl_2. After removal of solvent under reduced pressure, dry CH_2Cl_2 (26 mL, 0.45 M), TBSCl (15 mmol, 1.3 equiv.) and 1-methylimidazole (15 mmol, 1.3 equiv.) was added to the crude mixture (4-hydroxy-1-(1-methyl-1*H*-imidazol-2-yl)butan-1-one, 12 mmol, 1.0 equiv.) under argon atmosphere at 0 °C. The cooling bath was removed and the reaction mixture was stirred for 3 h at room

temperature. After it was quenched by sat. NH_4Cl aq. sat. $NaHCO_3$ aq was added to the resultant mixture and extracted with CH_2Cl_2. The residue was purified by distillation after silica gel flash chromatography to afford **1f**. : (Colorless liquid, 33% yield) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13 (s, 1H, *imidazole*), 7.01 (s, 1H, *imidazole*), 4.00 (s, 3H, NC*H*$_3$), 3.70 (t, *J* = 7.0 Hz, 2H, C*H*$_2$OTBS), 3.18 (t, *J* = 7.0 Hz, 2H, COC*H*$_2$), 1.94 (quin, *J* = 7.0 Hz, 2H, COCH$_2$C*H*$_2$), 0.87 (s, 9H, SiC(C*H*$_3$)$_3$), 0.30 (s, 6H, Si(C*H*$_3$)$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.0, 143.1, 128.9, 126.7, 62.5, 36.2, 35.5, 27.4, 25.9, 18.3, -5.3; IR (thin film, NaCl) 3021, 2955, 2931, 2859, 1674, 1416, 1254, 1215, 1103, 837, 772 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{26}N_2O_2Si$ $(M + H)^+$ 283.1836, found 283.1836.



1-(1-methyl-1*H*-imidazol-2-yl)-2-phenylethan-1-one (1g): (Brown solid, 62% yield); Following the general procedure A using ethyl phenylacetate (2.5 equiv.) and stirring for 3 h. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36-7.30 (m, 4H, Ar*H*), 7.24 (t, *J* = 7.0 Hz, 1H, Ar*H*), 7.19 (s, 1H, *imidazole*), 7.04 (s, 1H, *imidazole*) 4.43 (s, 2H, COC*H*$_2$), 3.97 (s, 3H, NC*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 190.1, 142.8, 134.6, 129.9, 129.3, 128.5, 127.4, 126.8,45.4, 36.2.; IR (neat) 1663, 1420, 1398, 1277, 1244, 1016, 907, 799, 789, 733, 702, 694, 669, 637, 615, 511 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{12}H_{12}N_2O$ $(M + H)^+$ 201.1022, found 201.1045.

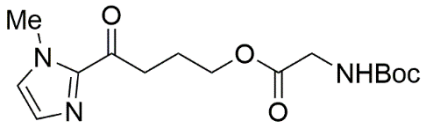


1-(1-methyl-1*H*-imidazol-2-yl)-2-(thiophen-3-yl)ethan-1-one (1h): CAS Registry Number 1519308-06-5; (Pale yellow solid, 26% yield); Following the general procedure A using *N*-methoxy-*N*-methyl-3-thiopheneacetamide (1.0 equiv.) and stirring for 14 h. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 5.0, 3.0 Hz, 1H, Ar*H*), 7.21 (m, 1H, Ar*H*), 7.19 (s, 1H, *imidazole*), 7.10 (dd, *J* = 5.0, 1.5 Hz, 1H, Ar*H*), 7.06 (s, 1H, *imidazole*), 4.47 (s, 2H, COC*H*$_2$), 3.99 (s, 3H, NC*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 189.5, 142.6, 134.1, 129.3, 129.0, 127.4, 125.4, 123.2, 39.9, 36.3.

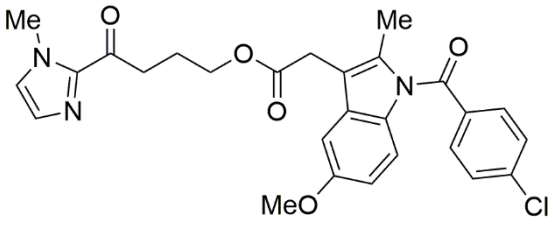
General procedure B for synthesis of 2-acylimidazoles (1i-1l, 1n)

To a solution of 1-methylimidazole (100 mmol, 1.0 equiv.) in dry THF (143 mL, 0.7 M) under argon atmosphere was added dropwise 2.6 M *n*-BuLi in *n*-hexane (1.1 equiv.) at –78 °C. The cooling bath was removed and the reaction mixture was stirred at room temperature. After 1 h, the mixture was

again cooled to –78°C and γ-butyrolactone (200 mmol, 2.0 equiv.) was added. The cooling bath was removed and the reaction mixture was stirred for 27 h at room temperature. After it was quenched by 1 M HCl aq, sat. $NaHCO_3$ aq was added to the resultant mixture and extracted with CH_2Cl_2. After removal of solvent under reduced pressure, the resulting residue was purified by silica gel flash chromatography to afford the intermediate (4-hydroxy-1-(1-methyl-1*H*-imidazol-2-yl)butan-1-one) in 43% yield as pale yellow solid. To a solution of 4-hydroxy-1-(1-methyl-1*H*-imidazol-2-yl)butan-1-one (1.0 equiv.) and 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (1.3 equiv.), DMAP (0.10 equiv.) in dry CH_2Cl_2 (0.20 M) under argon atmosphere was added triethylamine (1.4 equiv.) and indicated carboxylic acid. The reaction mixture was stirred at room temperature. To the resultant mixture was added sat. $NaHCO_3$ aq and extracted with CH_2Cl_2. After removal of solvent under reduced pressure, the residue was purified by silica gel flash chromatography to afford the desired 2-acylimidazole, which was used without further purification unless otherwise noted. The yield indicated bellow is condensation step.



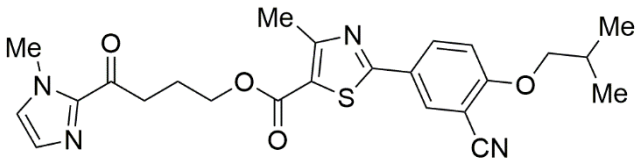
4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl (*tert*-butoxycarbonyl)glycinate (1i): (Yellow liquid, 96% yield); Following the general procedure B using *N*-(tert-Butoxycarbonyl)glycine (1.2 equiv.) and stirring for 21 h. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13 (s, 1H, *imidazole*), 7.04 (s, 1H, *imidazole*), 5.02 (br, 1H, N*H*), 4.24 (t, *J* = 6.5 Hz, 2H, OCH_2), 4.01 (s, 3H, NCH_3), 3.90 (d, *J* = 5.5 Hz, 2H, COCH_2NH), 3.23 (t, *J* = 7.0 Hz, 2H, COCH_2CH$_2$), 2.08 (quin, *J* = 7.0 Hz, 2H, COCH$_2$CH_2), 1.45 (s, 9H, COOC(CH_3)$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.7, 170.4, 155.7, 142.8, 129.1, 127.1, 79.9, 64.7, 42.4, 36.2, 35.2, 28.3, 23.0; IR (neat) 1748, 1713, 1674, 1514, 1408, 1366, 1288, 1250, 1157, 1053, 991, 914, 779 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{15}H_{23}N_3O_5$ $(M + H)^+$ 326.1710, found 326.1710.



4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl

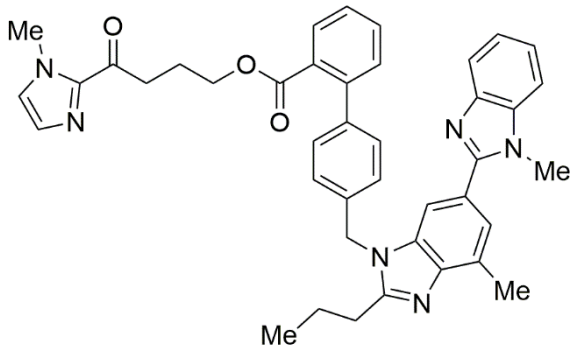
2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate (1j): (Yellow solid, 61% yield); Following the general procedure B using indometacin (1.3 equiv.) and stirring for 14 h. ^{1}H NMR (500

MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.5 Hz, 2H, Ar*H*), 7.47 (d, *J* = 8.5 Hz, 2H, Ar*H*), 7.10 (s, 1H, *imidazole*), 7.03 (s, 1H, *imidazole*), 6.96 (d, *J* = 2.5 Hz, 1H, Ar*H*), 6.87 (d, *J* = 9.0 Hz, 1H, Ar*H*), 6.65 (dd, *J* = 9.0 Hz, 2.5 Hz, 1H, Ar*H*), 4.19 (t, *J* = 7.0 Hz, 2H, OCH_2), 3.99 (s, 3H, NCH_3), 3.83 (s, 3H, OCH_3), 3.65 (s, 2H, ArCH_2), 3.19 (t, *J* = 7.0 Hz, 2H, COCH_2), 2.37 (s, 3H, ArCH_3), 2.06 (quin, *J* = 7.0 Hz, 2H, $COCH_2CH_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.7, 170.8, 168.3, 156.0, 142.8, 139.2, 135.9, 133.9. 131.2, 130.8, 130.6, 129.1, 129.1, 127.0, 115.0, 112.6, 111.8, 101.0, 64.4, 55.7, 36.2, 35.3, 30.3, 23.0, 13.4; IR (neat) 1732, 1672, 1589, 1476, 1456, 1437, 1402, 1356, 1314, 1288, 1260, 1219, 1163, 1142, 1111, 1088, 1067, 1034, 1013, 991, 926, 914, 831, 799, 773, 752, 739, 691, 667, 648, 631, 602, 592, 561, 546, 480, 432, 419 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{27}H_{26}ClN_3O_5$ $(M + H)^+$ 508.1634, found 508.1634.



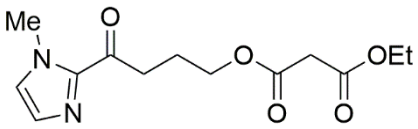
4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl

2-(3-cyano-4-isobutoxyphenyl)-4-methylthiazole-5-carboxylate (1k): (White solid, 76% yield); Following the general procedure B using febuxostat (1.0 equiv.) and stirring for 17 h. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.16 (d, *J* = 2.5 Hz, 1H, Ar*H*), 8.08 (dd, *J* = 9.0, 2.5 Hz, 1H, Ar*H*), 7.14 (s, 1H, *imidazole*), 7.04 (s, 1H, *imidazole*), 7.01 (d, *J* = 9.0 Hz, 1H, Ar*H*), 4.39 (t, *J* = 6.5 Hz, 2H, OCH_2CH_2), 4.00 (s, 3H, NCH_3), 3.90 (d, *J* = 6.5 Hz, 2H, OCH_2CH), 3.31 (t, *J* = 7.5 Hz, 2H, COCH_2), 2.75 (s, 3H, ArCH_3), 2.20 (m, 3H, $COCH_2CH_2$, $CH(CH_3)_2$), 1.09 (d, *J* = 7.0 Hz, 6H, $CH(CH_3)_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.6, 167.2, 162.5, 161.9, 161.3, 142.8, 132.5, 132.1, 129.1, 127.1, 126.0, 121.7, 115.4, 112.6, 102.9, 75.7, 64.7, 36.2, 35.3, 28.1, 23.1, 19.1, 17.5; IR (neat) 1682, 1603, 1431, 1414, 1350, 1329, 1300, 1292, 1275, 1132, 1111, 1043, 1013, 982, 914, 797 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{24}H_{26}N_4O_4S$ $(M + H)^+$ 467.1748, found 467.1748.

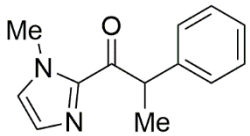


4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl

4'-((1,7'-dimethyl-2'-propyl-1*H*,3'*H*-[2,5'-bibenzo[*d*]imidazol]-3'-yl)methyl)-[1,1'-biphenyl]-2-carboxylate (1l): Following the general procedure B using telmisartan (1.0 equiv.) and stirring for 15 h. (White solid, 0.80g, 63% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81-7.77 (m, 2H, Ar*H*), 7.51-7.47 (m, 2H, Ar*H*), 7.44 (s, 1H, Ar*H*), 7.38 (dt, *J* = 7.5, 1.5 Hz, 1H, Ar*H*), 7.35-7.33 (m, 1H, Ar*H*), 7.29-7.24 (m, 5H, Ar*H*), 7.10 (s, 1H, *imidazole*), 7.08 (m, 2H, Ar*H*), 6.96 (s, 1H, *imidazole*), 5.46 (s, 2H, ArC*H*$_2$Ar), 4.11 (t, *J* = 6.5 Hz, 2H, OC*H*$_2$), 3.89 (s, 3H, NC*H*$_3$), 3.78 (s, 3H, NC*H*$_3$), 2.99-2,92 (m, 4H, COC*H*$_2$, ArC*H*$_2$CH$_2$), 2.76 (s, 3H, ArC*H*$_3$), 1.88 (sex, *J* = 7.5 Hz, 2H, ArCH$_2$C*H*$_2$), 1.80 (quin, *J* = 7.0 Hz, 2H, COCH$_2$C*H*$_2$), 1.05 (t, *J* = 7.5 Hz, 3H, ArCH$_2$CH$_2$C*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.8, 168.2, 156.5, 154.8, 143.2, 142.9, 142.7, 141.8, 141.3, 136.7, 135.1, 134.9, 131.3, 130.7, 130.7, 130.0, 129.5, 129.1, 129.0, 127.4, 127.0, 126.0, 123.9, 123.9, 122.4, 122.3, 119.6, 109.5, 109.0, 64.3, 47.1, 36.1, 35.3, 31.8, 29.9, 22.9, 22.0, 16.9, 14.1; IR (neat) 1719, 1670, 1404, 1319, 1281, 1244, 1126, 1086, 991, 914, 849, 743, 428, 405 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{41}H_{40}N_6O_3$ $(M + H)^+$ 665.3235, found 665.3210.



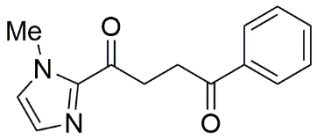
ethyl (4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl) malonate (1n): (Colorless liquid, 61% yield); Following the general procedure B using monoethyl malonate (1.3 equiv.) and stirring for 14 h. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13 (s, 1H, *imidazole*), 7.04 (s, 1H, *imidazole*), 4.25-4.18 (m, 4H, OC*H*$_2$CH$_3$, OC*H*$_2$CH$_2$), 4.01 (s, 3H, NC*H*$_3$), 3.36 (s, 2H, COC*H*$_2$CO), 3.23 (t, *J* = 7.5 Hz, 2H, COC*H*$_2$CH$_2$), 2.08 (quin, *J* = 7.5 Hz, 2H, COCH$_2$C*H*$_2$), 1.28 (t, *J* = 9.0 Hz, 3H, COCH$_2$C*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.7, 166.6, 166.5, 142.8, 129.1, 127.0, 64.8, 61.6, 41.6, 36.2, 35.2, 22.9, 14.1; IR (neat) 1744, 1726, 1672, 1464, 1408, 1368, 1331, 1288, 1267, 1182, 1150, 1098, 1030, 997, 970, 914, 779, 696, 685 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{13}H_{18}N_2O_5$ $(M + H)^+$ 283.1288, found 283.1288.



1-(1-methyl-1*H*-imidazol-2-yl)-2-phenylpropan-1-one (1m): (Brown solid, 67% yield); Following the general procedure A using (±)-methyl 2-phenylpropanoate (2.5 equiv.) and stirring for 3 h. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.0 Hz, 2H, Ar*H*), 7.29 (t, *J* = 8.0 Hz, 2H, Ar*H*), 7.20 (t, *J* = 8.0 Hz, 1H, Ar*H*), 7.14 (s, 1H, *imidazole*), 6.98 (s, 1H, *imidazole*) 5.28 (q, *J* = 7.0 Hz, 1H, COC*H*), 3.94(s, 3H, NC*H*$_3$), 1.54(d, *J* = 7.0 Hz, 3H,CHC*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.3, 142.6, 140.8, 129.2, 128.5, 128.3, 127.3, 126.8, 46.7, 36.2, 18.1; IR (neat) 1667, 1452, 1402, 1371, 1288, 1152, 995, 951, 910, 777, 752,

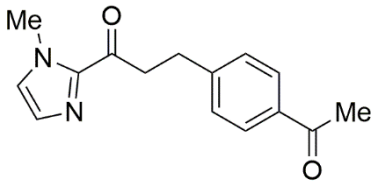
704, 687, 662, 561, 503 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{13}H_{14}N_2O$ $(M + H)^+$ 215.1179, found 215.1196.

Procedure for synthesis of 1-(1-methyl-1*H*-imidazol-2-yl)-4-phenylbutane-1,4-dione (1o)



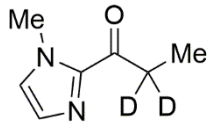
To a solution of 1-methylimidazole (36 mmol, 1.0 equiv.) in dry THF (50 mL, 0.70 M) under argon atmosphere was added slowly 2.6 M *n*-BuLi in *n*-hexane (1.1 equiv.) at –78°C. After 1 h, γ-phenyl-γ-butyrolactone (45 mmol, 1.3 equiv.) was added and stirring for 3 h. After it was quenched by 1 M HCl, sat. $NaHCO_3$ aq was added to the resultant mixture and extracted with CH_2Cl_2. After removal of solvent under reduced pressure, the resulting residue was purified by silica gel flash chromatography. To 4-hydroxy-1-(1-methyl-1*H*-imidazol-2-yl)-4-phenylbutan-1-one (1.2 mmol, 1.0 equiv.) in dry CH_2Cl_2 (23 ml, 0.05 M) under argon atmosphere was added Dess-Martin periodinane (1.3 mmol, 1.1 equiv.) at 0 °C. The cooling bath was removed and the reaction mixture was stirred for 5 h at room temperature. To the resultant mixture was added sat. $NaHCO_3$ aq and extracted with CH_2Cl_2. The residue was purified by recrystalization after silica gel flash chromatography to afford **1o**. : (Brown solid, 67% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.5 Hz, 2H, Ar*H*), 7.57 (t, *J* = 7.5, 1H, Ar*H*), 7.47 (t, *J* = 7.5, 2H, Ar*H*), 7.17 (s, 1H, *imidazole*), 7.03 (s, 1H, *imidazole*), 3.98 (s, 3H, NC*H*$_3$), 3.60 (t, *J* = 5.5, 2H, PhCOCH$_2$C*H*$_2$), 3.42 (t, *J* = 5.5, 2H, PhCOC*H*$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.4, 191.4, 142.9, 136.8, 133.1, 129.2, 128.6, 128.1, 126.8, 36.1, 33.1, 32.6; IR (neat) 1682, 1667, 1412, 1393, 1362, 1229, 989, 910, 791, 770, 729, 702, 692, 685, 513 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{14}N_2O_2$ $(M + H)^+$ 243.1128, found 243.1145.

Procedure for synthesis of 3-(4-acetylphenyl)-1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (1p)



To a solution of $AlCl_3$ (50 mmol, 3.3 equiv.) in dry CH_2Cl_2 (75 mL, 0.20 M) under argon atmosphere was added acetyl chloride (15 mmol, 1.0 equiv.) and 1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one (**1c**, 15 mmol, 1.0 equiv.) at 0 °C. The cooling bath was removed and the reaction mixture was stirred for 13 h at room temperature. After it was quenched by H_2O, sat. $NaHCO_3$ aq was added to the mixture and extracted with CH_2Cl_2. After

removal of solvent under reduced pressure, the resulting residue was purified by recrystallization after silica gel flash chromatography to afford **1p**. : (Brown solid, 1.7 g, 44% yield) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.5 Hz, 2H, Ar*H*), 7.36 (d, *J* = 8.5 Hz, 2H, Ar*H*), 7.13 (s, 1H, *imidazole*), 7.03 (s, 1H, *imidazole*), 4.00 (s, 3H, NCH_3), 3.51 (t, *J* = 7.5 Hz, 2H, COCH_2), 3.10 (t, *J* = 7.5 Hz, 2H, PhCH_2), 2.58 (s, 3H, COCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.9, 191.4, 146.9, 142.8, 135.2, 129.1, 128.7, 128.6, 127.1, 39.7, 36.2, 29.8, 26.6; IR (KBr) 3117, 1670, 1605, 1570, 1404, 1362, 1269, 1180, 1150, 1072, 980, 957, 914, 826, 787 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{15}H_{16}N_2O_2$ $(M + H)^+$ 257.1285, found 257.1285.



1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one-2,2-d_2 (1a(d^2))[7]: To a solution of TBD (5.6 mmol, 0.22 equiv.) in dry $CDCl_3$ (75 mL, 0.20 M) was added 1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (**1a**, 25 mmol, 1.0 equiv.) under argon atmosphere. The reaction mixture was stirred for 42 h at room temperature. After removal of solvent under reduced pressure, the resulting residue was purified by distillation after silica gel flash chromatography to afford **1a(d^2)**. : (colorless liquid, 81% yield, 96% deuterated); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 4.01 (s, 3H, NCH_3), 1.18 (s, 3H, CD_2CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.9, 142.9, 128.9, 126.8, 36.2, 31.8 (sex, *J* = 19.5 Hz), 8.0; IR (neat) 1668, 1458, 1400, 1290, 1261, 1152, 1024, 1005, 941, 910, 837, 768, 671 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_7H_8D_2N_2O$ $(M + H)^+$ 141.0991, found 141.0984.

5. General Procedure and Characterization of the Products

General procedure for catalytic oxidative cross coupling reaction about scope of alkylarenes: A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%) were added followed by the addition of alkylarene (0.20 M) and **1a** (30 µL, 1.0 equiv.). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product (**3aa-3ao, 3aa(*d*^{7})**).

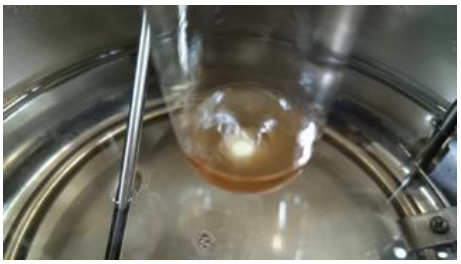
$FeCl_2$ + **L11**

$FeCl_2$ + **L11** + toluene (0.64 ml)

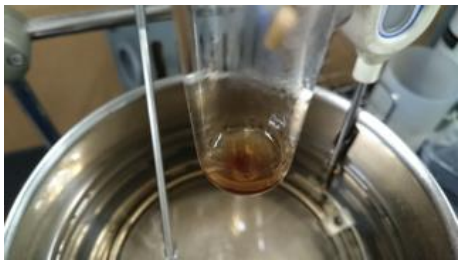
$FeCl_2$ + **L11** + toluene (0.64 ml) + **1a**

$FeCl_2$ + L11 + toluene (0.64 ml) + **1a** + DTBP + toluene (0.5 ml)

80 °C 1h

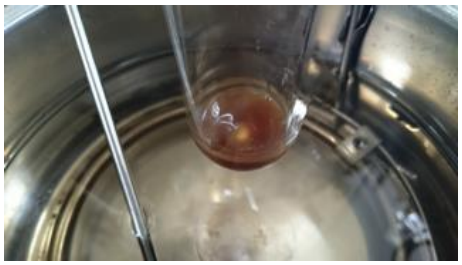


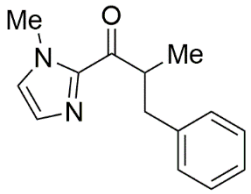
80 °C 2h



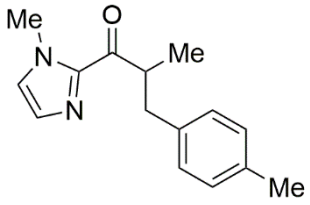
80 °C 2h

80 °C 10 h

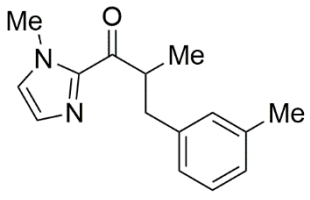




2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one (3aa): (Colorless liquid, *n*-Hexane/EtOAc = 9/1 to 4/1, 83% yield, 43.4 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25-7.22 (m, 4H, Ar*H*), 7.18-7.14 (m, 1H, Ar*H*), 7.13 (s, 1H, *imidazole*), 7.00 (s, 1H, *imidazole*), 4.24-4.17 (m, 1H, COC*H*), 3.97 (s, 3H, NCH_3), 3.15 (dd, *J* = 13.5, 6.5 Hz, 1H, ArCH_2), 2.68 (dd, *J* = 13.5, 8.5 Hz, 1H, ArCH_2), 1.19 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.2, 142.6, 139.9, 129.2, 129.0, 128.2, 127.0, 126.0, 43.0, 39.0, 36.2, 17.0; IR (neat) 1670, 1452, 1404, 1288, 1153, 970, 912, 772, 745, 698, 673, 662, 521, 484 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{16}N_2O$ $(M + H)^+$ 229.1335, found 226.1335.

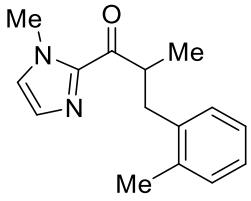


2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-(*p*-tolyl)propan-1-one (3ab): (White solid, *n*-Hexane/EtOAc = 9/1 to 4/1, 88% yield, 48.6 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (s, 1H, *imidazole*), 7.11 (d, *J* = 7.5 Hz, 2H, Ar*H*), 7.05 (d, *J* = 7.5 Hz, 2H, Ar*H*), 7.00 (s, 1H, *imidazole*), 4.21-4.14 (m, 1H, COC*H*), 3.97 (s, 3H, NCH_3), 3.11 (dd, *J* = 13.5, 6.5 Hz, 1H, ArCH_2), 2.64 (dd, *J* = 13.5, 8.0 Hz, 1H, ArCH_2), 2.29 (s, 3H, ArCH_3), 1.17 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.3, 142.6, 136.7, 135.4, 129.1, 129.0, 128.9, 127.0, 43.1, 38.5, 36.2, 21.0, 16.9; IR (neat) 1668, 1514, 1458, 1435, 1404, 1288, 1155, 970, 910, 887, 810, 777, 768, 673, 658, 550, 513, 482 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{15}H_{18}N_2O$ $(M + H)^+$ 243.1492, found 243.1492.

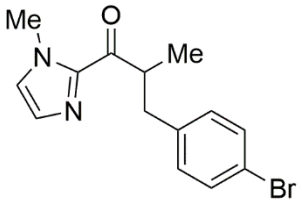


2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-(*m*-tolyl)propan-1-one (3ac): (Colorless liquid, *n*-Hexane/EtOAc = 9/1 to 4/1, 92% yield, 51.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.15-7.12 (m, 2H, *imidazole*, Ar*H*), 7.05-7.01 (m, 3H, *imidazole*, Ar*H*), 6.98 (d, *J* = 7.5 Hz, 1H, Ar*H*), 4.22-4.15 (m, 1H,

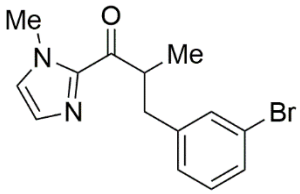
COC*H*), 3.98 (s, 3H, NC*H*$_3$), 3.11 (dd, *J* = 13.5, 6.5 Hz, 1H, ArC*H*$_2$), 2.63 (dd, *J* = 13.5, 8.5 Hz, 1H, ArC*H*$_2$), 2.30 (s, 3H, ArC*H*$_3$), 1.17 (d, *J* = 7.0 Hz, 3H, COCHC*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.3, 142.6, 139.8, 137.7, 130.0, 129.0, 128.1, 127.0, 126.8, 126.3, 43.0, 38.9, 36.2, 21.4, 16.9; IR (neat) 1670, 1458, 1404, 1375, 1288, 1153, 970, 912, 766, 741, 700, 662, 444 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{15}H_{18}N_2O$ $(M + H)^+$ 243.1492, found 243.1492.



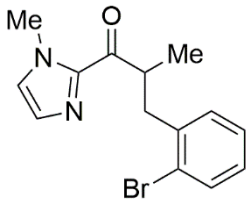
2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-(*o*-tolyl)propan-1-one (3ad): (Colorless liquid, *n*-Hexane/EtOAc = 9/1 to 4/1, 74% yield, 41.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.18-7.16 (m, 1H, Ar*H*), 7.12-7.10 (m, 2H, *imidazole*, Ar*H*), 7.08-7.06 (m, 2H, Ar*H*), 6.99(s, 1H, *imidazole*), 4.27-4.20 (m, 1H, COC*H*), 3.97 (s, 3H, NC*H*$_3$), 3.13 (dd, *J* = 14.0, 6.5 Hz, 1H, ArC*H*$_2$), 2.68 (dd, *J* = 14.0, 8.0 Hz, 1H, ArC*H*$_2$), 2.38 (s, 3H, ArC*H*$_3$), 1.20 (d, *J* = 7.0 Hz, 3H, COCHC*H*$_3$); IR (neat) 1670, 1491, 1458, 1404, 1288, 1275, 1153, 970, 912, 760, 741, 689, 662, 453 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{15}H_{18}N_2O$ $(M + H)^+$ 243.1492, found 243.1492.



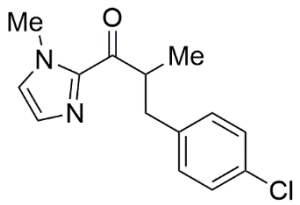
3-(4-bromophenyl)-2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (3ae): (White solid, *n*-Hexane/EtOAc = 9/1 to 4/1, 82% yield, 57.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.0 Hz, 2H, Ar*H*), 7.13 (s, 1H, *imidazole*), 7.11 (d, *J* = 8.0 Hz, 2H, Ar*H*), 7.01 (s, 1H, *imidazole*), 4.21-4.14 (m, 1H, COC*H*), 3.97 (s, 3H, NC*H*$_3$), 3.10 (dd, *J* = 13.5, 7.0 Hz, 1H, ArC*H*$_2$), 2.64 (dd, *J* = 13.5, 7.5 Hz, 1H, ArC*H*$_2$), 1.18 (d, *J* = 7.0 Hz, 3H, COCHC*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.7, 142.4, 138.9, 131.3, 130.9, 129.1, 127.2, 119.9, 42.9, 38.4, 36.2, 17.1; IR (neat) 1670, 1485, 1400, 1371, 1269, 1227, 1159, 1069, 1011, 968, 912, 810, 787, 756, 739, 687, 496 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{15}BrN_2O$ $(M + H)^+$ 307.0441, found 307.0432.



3-(3-bromophenyl)-2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (3af): (Pale yellow liquid, *n*-Hexane/EtOAc = 9/1 to 4/1, 78% yield, 54.3 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 1.5 Hz, 1H, Ar*H*), 7.29 (dt, *J* = 6.5, 1.5 Hz, 1H, Ar*H*), 7.17-7.10 (m, 3H, *imidazole*, Ar*H*), 7.02 (s, 1H, *imidazole*), 4.21-4.14 (m, 1H, COC*H*), 3.98 (s, 3H, NCH_3), 3.11 (dd, *J* = 13.5, 7.0 Hz, 1H, ArCH_2), 2.65 (dd, *J* = 13.5, 8.0 Hz, 1H, ArCH_2), 1.19 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.6, 142.4, 142.3, 132.2, 129.8, 129.2, 129.1, 127.9, 127.2, 122.3, 42.9, 38.6, 36.2, 17.0; IR (neat) 1670, 1566, 1470, 1460, 1404, 1288, 1153, 1070, 972, 912, 766, 696, 685, 667, 438 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{15}BrN_2O$ $(M + H)^+$ 307.0441, found 307.0443.

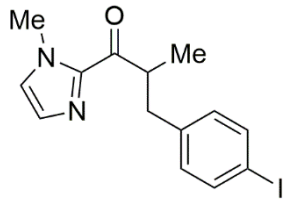


3-(2-bromophenyl)-2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (3ag): (White solid, *n*-Hexane/EtOAc = 9/1 to 4/1, 67% yield, 47.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 7.5, 1.0 Hz, 1H, Ar*H*), 7.27 (dd, *J* = 7.5, 1.5 Hz, 1H, Ar*H*), 7.18 (dt, *J* = 7.5, 1.0 Hz, 1H, Ar*H*), 7.12 (s, 1H, *imidazole*), 7.18 (dt, *J* = 7.5, 1.5 Hz, 1H, Ar*H*), 7.00 (s, 1H, *imidazole*), 4.35-4.28 (m, 1H, COC*H*), 3.98 (s, 3H, NCH_3), 3.23 (dd, *J* = 14.0, 7.0 Hz, 1H, ArCH_2), 2.91 (dd, *J* = 14.0, 7.0 Hz, 1H, ArCH_2), 1.25 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.8, 142.5, 139.4, 132.8, 131.2, 129.1, 127.8, 127.1, 127.1, 125.1, 41.6, 38.4, 36.2, 17.6; IR (neat) 1672, 1470, 1439, 1404, 1288, 1153, 1040, 972, 910, 772, 752, 723, 675, 658, 540, 451 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{15}BrN_2O$ $(M + H)^+$ 307.0441, found 307.0440.

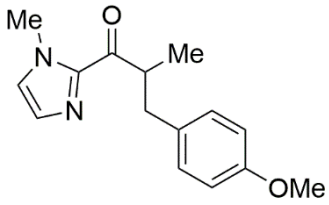


3-(4-chlorophenyl)-2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (3ah): (White solid,

n-Hexane/EtOAc = 9/1 to 4/1, 83% yield, 50.0 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 (d, *J* = 7.5 Hz, 2H, Ar*H*), 7.16 (d, *J* = 7.5 Hz, 2H, Ar*H*), 7.13 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 4.21-4.14 (m, 1H, COC*H*), 3.97 (s, 3H, NCH_3), 3.11 (dd, *J* = 13.5, 6.5 Hz, 1H, ArCH_2), 2.65 (dd, *J* = 13.5, 7.5 Hz, 1H, ArCH_2), 1.18 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.7, 142.4, 138.4, 131.8, 130.5, 129.1, 128.3, 127.2, 42.9, 38.3, 36.3, 17.1; IR (neat) 2359, 1670, 1458, 1400, 1371, 1269, 1159, 1088, 970, 912, 814, 787, 762, 689, 667, 500 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{15}ClN_2O$ $(M + H)^+$ 263.0946, found 263.0943.

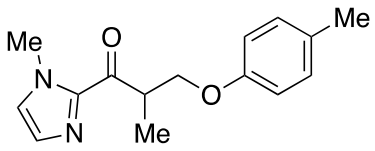


3-(4-iodophenyl)-2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (3ai): (Pale yellow solid, *n*-Hexane/EtOAc = 9/1 to 4/1, 78% yield, 63.3 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.5 Hz, 2H, Ar*H*), 7.14 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 6.99 (d, *J* = 8.5 Hz, 2H, Ar*H*), 4.21-4.13 (m, 1H, COC*H*), 3.97 (s, 3H, NCH_3), 3.09 (dd, *J* = 14.0, 7.0 Hz, 1H, ArCH_2), 2.62 (dd, *J* = 14.0, 8.0 Hz, 1H, ArCH_2), 1.18 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.7, 142.4, 139.6, 137.2, 131.3, 129.1, 127.2, 91.3, 42.8, 38.4, 36.3, 17.2; IR (neat) 1672, 1483, 1454, 1402, 1287, 1005, 972, 910, 889, 793, 775, 671, 664, 617, 519, 490 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{15}IN_2O$ $(M + H)^+$ 355.0302, found 355.0301.

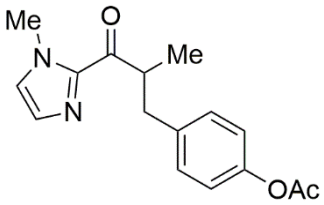


3-(4-methoxyphenyl)-2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (3aj): (Colorless liquid, *n*-Hexane/EtOAc = 9/1 to 4/1, Toluene/EtOAc = 1/0 to 9/1, 51% yield, 31.2 mg); byproduct (CH_2OAr) mixture ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.15-7.13 (m, 3H, *imidazole*, Ar*H*), 7.00 (s, 1H, *imidazole*), 6.79 (d, *J* = 8.5 Hz, 2H, Ar*H*), 4.19-4.12 (m, 1H, COC*H*), 3.97 (s, 3H, NCH_3), 3.77 (s, 3H, OCH_3), 3.08 (dd, *J* = 13.5, 6.5 Hz, 1H, ArCH_2), 2.62 (dd, *J* = 13.5, 8.0 Hz, 1H, ArCH_2), 1.17 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.3, 157.9, 142.6, 131.9, 130.1, 129.0, 127.0, 113.6, 55.2, 43.2, 38.2, 36.2, 16.9; IR (neat) 1670, 1611, 1510, 1462, 1402, 1244, 1177, 1153, 1034, 972, 912, 812, 557, 525 cm^{-1}; HRMS

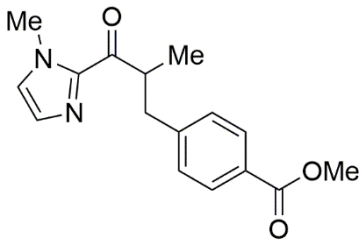
(ESI, H) *m/z* calc'd for $C_{15}H_{18}N_2O_2$ $(M + H)^+$ 259.1441, found 259.1450.



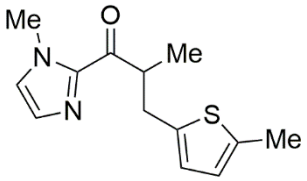
byproduct



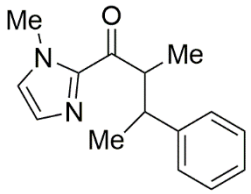
4-(2-methyl-3-(1-methyl-1*H*-imidazol-2-yl)-3-oxopropyl)phenyl acetate (3ak): (Orange solid, *n*-Hexane/EtOAc = 4/1 to 2/1, 40% yield, 26.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.5 Hz, 2H, Ar*H*), 7.13 (s, 1H, *imidazole*), 7.01 (s, 1H, *imidazole*), 6.96 (d, *J* = 8.5 Hz, 2H, Ar*H*), 4.23-4.16 (m, 1H, COC*H*), 3.97 (s, 3H, NCH_3), 3.14 (dd, *J* = 14.0, 7.0 Hz, 1H, ArCH_2), 2.67 (dd, *J* = 14.0, 8.0 Hz, 1H, ArCH_2), 2.27 (s, 3H, COCH_3), 1.20 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.9, 169.6, 148.9, 142.4, 137.5, 130.1, 129.1, 127.1, 121.2, 43.0, 38.3, 36.3, 21.2, 17.2; IR (neat) 1755, 1670, 1506, 1460, 1404, 1368, 1215, 1188, 1165, 1016, 972, 912, 773, 550, 511 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{16}H_{18}N_2O_3$ $(M + H)^+$ 287.1390, found 287.1390.



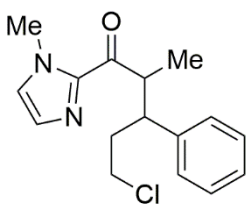
methyl 4-(2-methyl-3-(1-methyl-1*H*-imidazol-2-yl)-3-oxopropyl)benzoate (3al): (Pale yellow solid, *n*-Hexane/Et_2O = 1/1 to 1/2, 59% yield, 38.5 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.5 Hz, 2H, Ar*H*), 7.30 (d, *J* = 8.5 Hz, 2H, Ar*H*), 7.14 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 4.27-4.20 (m, 1H, COC*H*), 3.97 (s, 3H, NCH_3), 3.89 (s, 3H, OCH_3), 3.20 (dd, *J* = 13.5, 7.0 Hz, 1H, ArCH_2), 2.74 (dd, *J* = 13.5, 8.0 Hz, 1H, ArCH_2), 1.20 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.6, 167.2, 145.5, 142.4, 129.6, 129.2, 129.1, 128.0, 127.2, 52.0, 42.8, 39.0, 36.3, 17.2; IR (neat) 1715, 1670, 1611, 1458, 1433, 1404, 1273, 1179, 1107, 1020, 972, 912, 760, 704 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{16}H_{18}N_2O_3$ $(M + H)^+$ 287.1390, found 287.1406.



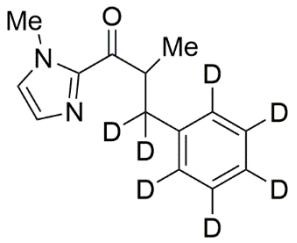
2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-(5-methylthiophen-2-yl)propan-1-one (3am): (Yellow liquid, *n*-Hexane/EtOAc = 9/1 to 4/1, 50% yield, 28.2 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.15 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 6.57 (d, *J* = 3.0 Hz, 1H, Ar*H*), 6.51-6.50 (m, 1H, Ar*H*) 4.20-4.13 (m, 1H, COC*H*), 3.99 (s, 3H, NCH_3), 3.25 (dd, *J* = 15.0, 7.0 Hz, 1H, ArCH_2), 2.88 (dd, *J* = 15.0, 7.0 Hz, 1H, ArCH_2), 2.39 (s, 3H, ArCH_3), 1.25 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.7, 142.5, 140.0, 137.9, 129.1, 127.1, 125.2, 124.6, 43.4, 36.3, 33.0, 17.5, 15.3; IR (neat) 1670, 1458, 1402, 1288, 1153, 970, 912, 795, 773, 665, 515, 490 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{13}H_{16}N_2OS$ $(M + H)^+$ 249.1056, found 249.1054.



2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylbutan-1-one (3an): (White solid, *n*-Hexane/EtOAc = 9/1 to 4/1, 74% yield, 40.7 mg, dr = 65/35); ^{1}H NMR (500 MHz, $CDCl_3$) (major) δ 7.32-7.16 (m, 5H, Ar*H*), 7.19 (s, 1H, *imidazole*), 7.07 (s, 1H, *imidazole*), 4.23-4.12 (m, 1H, COC*H*), 4.04 (s, 3H, NCH_3), 3.14-3.08 (m, 1H, ArC*H*), 1.21 (d, *J* = 6.5 Hz, 3H, COCHCH_3), 0.93 (d, *J* = 7.0 Hz, 3H, ArCHCH_3); (minor) δ 7.32-7.16 (m, 4H, Ar*H*), 7.09-7.06 (m, 2H, *imidazole*, Ar*H*), 6.89 (s, 1H, *imidazole*), 4.23-4.12 (m, 1H, COC*H*), 3.78 (s, 3H, NCH_3), 3.26-3.20 (m, 1H, ArC*H*), 1.29 (d, *J* = 7.5 Hz, 3H, COCHCH_3), 1.24 (d, *J* = 7.0 Hz, 3H, ArCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.2, 196.4, 145.7, 145.2, 143.3, 143.0, 129.1, 128.8, 128.4, 128.0, 127.8, 127.6, 127.4, 126.7, 126.3, 125.9, 47.5, 47.5, 43.2, 42.1, 36.4, 36.0, 21.2, 18.4, 17.0, 14.6; IR (neat) 1665, 1493, 1451, 1400, 1366, 1288, 1153, 964, 953, 910, 781, 766, 702, 691, 665, 546 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{15}H_{18}N_2O$ $(M + H)^+$ 243.1492, found 243.1502.

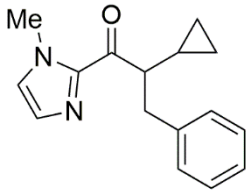


5-chloro-2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpentan-1-one (3ao): (Colorless liquid, *n*-Hexane/EtOAc = 9/1 to 4/1, Toluene/EtOAc = 14/1, Cyclohexane/EtOAc/DCM = 15/1/4, 60% yield (^{1}H NMR yield), dr = 75/25); ^{1}H NMR (500 MHz, $CDCl_3$) (major) δ 7.35-7.32 (m, 2H, Ar*H*), 7.28-7.23 (m, 3H, Ar*H*), 7.19 (s, 1H, *imidazole*), 7.09 (s, 1H, *imidazole*), 4.27-4.20 (m, 1H, COC*H*), 4.06 (s, 3H, NCH_3), 3.28-3.11 (m, 3H, ArC*H*, CH_2Cl), 2.09-1.95 (m, 2H, ArCHCH_2), 0.92 (d, *J* = 7.0 Hz, 3H, COCHCH_3); (minor) δ 7.21-7.16 (m, 4H, Ar*H*), 7.11-7.07 (m, 2H, *imidazole*, Ar*H*), 6.89 (s, 1H, *imidazole*), 4.32-4.26 (m, 1H, COC*H*), 3.73 (s, 3H, NCH_3), 3.40-3.36 (m, 1H, ArC*H*), 3.28-3.21 (m, 1H, CH_2Cl), 3.20-3.14 (m, 1H, CH_2Cl), 2.33-2.29 (m, 1H, ArCHCH_2), 2.09-2.03 (m, 1H, ArCHCH_2), 1.32 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) (major) δ 196.4, 143.0, 141.1, 129.3, 128.7, 128.5, 127.6, 126.9, 46.3, 46.1, 43.0, 37.7, 36.5, 17.1; (minor) δ 195.6, 142.9, 141.7, 128.8, 128.3, 128.2, 126.9, 126.5, 46.3, 45.5, 42.9, 36.0, 35.4, 15.6; IR (neat) (major + minor + byproduct) 1668, 1452, 1402, 1368, 1287, 1155, 966, 912, 897, 775, 702, 665, 577, 561; (minor + major) 1668, 1493, 1452, 1404, 1287, 1153, 1078, 966, 912, 895, 762, 700, 665, 556 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{16}H_{19}ClN_2O$ $(M + H)^+$ 291.1259, found (major + minor + byproduct) 291.1258; (minor + major) 291.1254.

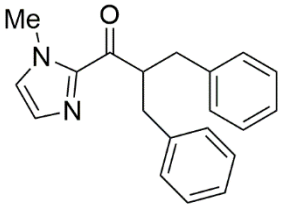


2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-(phenyl-d_5)propan-1-one-3,3-d_2 (3aa(d^7)): (Colorless liquid, *n*-Hexane/EtOAc = 2/1 (PTLC)); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (s, 1H, *imidazole*), 7.01 (s, 1H, *imidazole*), 4.19 (q, *J* = 7.0 Hz, 1H, COC*H*), 3.98 (s, 3H, NCH_3), 1.18 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.2, 142.5, 139.6, 129.0, 128.7 (t, *J* = 24.1 Hz), 127.7 (t, *J* = 24.1 Hz), 127.1, 125.5 (t, *J* = 24.4 Hz), 42.9, 38.1 (quin, *J* = 19.9 Hz), 36.3, 17.0; IR (neat) 2359, 2342, 1668, 1456, 1402, 1288, 968, 912, 772, 556, 538, 529, 446 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_9D_7N_2O$ $(M + H)^+$ 236.1775, found 236.1785.

General procedure for catalytic oxidative cross coupling reaction about scope of 2-acylimidazole: A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%) were added followed by the addition of toluene (0.20 M) and 2-acylimidazole (1.0 equiv.). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product (**3ba-3la, 3aa(*d*[1])**).

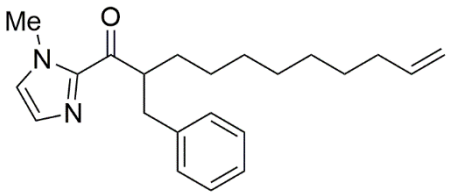


2-cyclopropyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one (3ba): (White solid, *n*-Hexane/EtOAc = 9/1 to 4/1, 79% yield, 39.5 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.20-7.19 (m, 4H, Ar*H*), 7.14-7.10 (m, 1H, Ar*H*), 7.09 (s, 1H, *imidazole*), 6.98 (s, 1H, *imidazole*), 3.96 (s, 3H, NCH_3), 3.49-3.44 (m, 1H, COC*H*), 3.21 (dd, *J* = 14.0, 8.5 Hz, 1H, ArCH_2), 2.99 (dd, *J* = 14.0, 6.5 Hz, 1H, ArCH_2), 1.04-0.97 (m, 1H, COCHC*H*), 0.52-0.47 (m, 1H, CH(CH_2)$_2$), 0.39-0.34 (m, 2H, CH(CH_2)$_2$), 0.13-0.08 (m, 1H, CH(CH_2)$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.3, 143.4, 139.8, 129.2, 129.0, 128.1, 127.1, 125.9, 52.7, 38.3, 36.2, 13.9, 4.7, 3.5; IR (neat) 1667, 1452, 1433, 1404, 1292, 1020, 955, 910, 826, 783, 748, 725, 704, 685, 658, 532, 494 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{16}H_{18}N_2O$ (M + H)$^+$ 255.1492, found 255.1489.

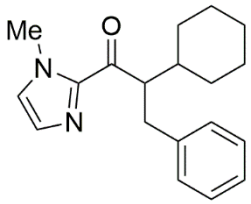


2-benzyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one (3ca): (White solid, *n*-Hexane/EtOAc = 9/1, 82% yield, 52.3 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23-7.18 (m, 8H, Ar*H*), 7.14-7.11 (m, 2H, Ar*H*), 7.08 (s, 1H, *imidazole*), 6.92 (s, 1H, *imidazole*), 4.60-4.54 (m, 1H, COC*H*), 3.88 (s, 3H, NCH_3), 3.13 (dd, *J* = 13.5, 7.5 Hz, 2H, COCH(CH_2Ar)$_2$), 2.77 (dd, *J* = 13.5, 6.5 Hz, 2H, COCH(CH_2Ar)$_2$); ^{13}C NMR

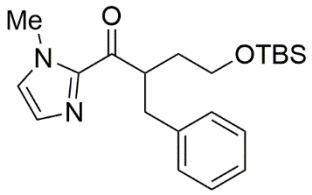
(125 MHz, $CDCl_3$) δ 194.8, 143.0. 139.6, 129.1, 129.1, 128.2, 126.9, 126.0, 50.0, 37.5, 36.1; IR (neat) 1670, 1445, 1406, 1350, 1150, 1013, 951, 907, 785, 754, 745, 700, 687, 565, 521, 496 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{20}H_{20}N_2O$ $(M + H)^+$ 305.1648, found 305.1644.



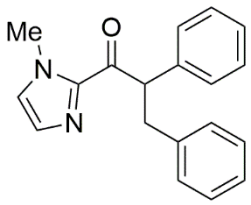
2-benzyl-1-(1-methyl-1*H*-imidazol-2-yl)undec-10-en-1-one (3da): (Colorless liquid, *n*-Hexane/EtOAc = 9/1, 60% yield, 39.9 mg); ^1H NMR (500 MHz, $CDCl_3$) δ 7.23-7.19 (m, 4H, Ar*H*), 7.14-7.12 (m, 1H, Ar*H*), 7.11 (s, 1H, *imidazole*), 6.97 (s, 1H, *imidazole*), 5.82-5.74 (m, 1H, C*H*=CH_2), 4.99-4.94 (m, 1H, CH=CH_2), 4.92-4.90 (m, 1H, CH=CH_2), 4.22-4.19 (m, 1H, COC*H*), 3.94 (s, 3H, NCH_3), 3.06 (dd, *J* = 13.5, 7.5 Hz, 1H, ArCH_2), 2.76 (dd, *J* = 13.5, 7.5 Hz, 1H, ArCH_2), 2.02-1.97 (m, 2H, CH_2CH=CH_2), 1.78-1.74 (m, 1H, COCHCH_2), 1.54-1.50 (m, 1H, COCHCH_2), 1.33-1.22 (m, 10H, $COCHCH_2(CH_2)_5$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.2, 143.3, 139.9, 139.2, 129.1, 129.0, 128.1, 127.0, 125.9, 114.1, 48.3, 37.9, 36.2, 33.8, 31.8, 29.7, 29.2, 29.0, 28.9, 27.3; IR (neat) 2924, 2853, 1670, 1454, 1406, 1288, 1153, 993, 945, 910, 768, 745, 698, 665 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{22}H_{30}N_2O$ $(M + H)^+$ 339.2431, found 339.2429.



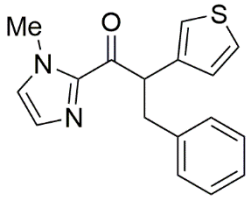
2-cyclohexyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one (3ea): (White solid, *n*-Hexane/EtOAc = 9/1, 68% yield, 40.5 mg); ^1H NMR (500 MHz, $CDCl_3$) δ 7.16-7.12 (m, 4H, Ar*H*), 7.07-7.03 (m, 2H, *imidazole*, Ar*H*), 6.89 (s, 1H, *imidazole*), 4.17-4.13 (m, 1H, COC*H*), 3.86 (s, 3H, NCH_3), 3.02-2.93 (m, 2H, ArCH_2), 1.88-1.59 (m, 6H, *cyclohexyl*), 1.27-1.06 (m, 5H, *cyclohexyl*); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.3, 144.1, 140.3, 129.0, 128.8, 128.0, 126.7, 125.7, 53.7, 41.0, 36.2, 34.7, 31.4, 30.2, 26.5, 26.5, 26.4; IR (neat) 2924, 1674, 1454, 1404, 1015, 945, 912, 905, 779, 750, 723, 700, 685, 675, 550, 523 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{19}H_{24}N_2O$ $(M + H)^+$ 297.1961, found 297.1961.



2-benzyl-4-((*tert*-butyldimethylsilyl)oxy)-1-(1-methyl-1*H*-imidazol-2-yl)butan-1-one (3fa): (White solid, *n*-Hexane/EtOAc = 19/1 to 9/1, 79% yield, 61.4 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24-7.20 (m, 4H, Ar*H*), 7.15-7.12 (m, 1H, Ar*H*), 7.11 (s, 1H, *imidazole*), 6.97 (s, 1H, *imidazole*), 4.28-4.22 (m, 1H, COC*H*), 3.94 (s, 3H, NCH_3), 3.58 (t, *J* = 7.0 Hz, 2H, OCH_2) 3.10 (dd, *J* = 13.5, 7.0 Hz, 1H, ArCH_2), 2.75 (dd, *J* = 13.5, 8.0 Hz, 1H, ArCH_2), 2.10-2.03 (m, 1H, $COCH_2CH_2$), 1.77-1.71 (m, 1H, $COCH_2CH_2$), 0.78 (s, 9H, SiC(CH_3)$_3$), -0.07 (s, 3H, Si(CH_3)$_2$), -0.12 (s, 3H, Si(CH_3)$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.4, 143.1, 139.6, 129.2, 129.0, 128.2, 126.8, 126.0, 61.7, 45.9, 38.3, 36.2, 34.3, 25.8, 18.2, -5.5; IR (neat) 1672, 1404, 1250, 1086, 1053, 995, 955, 914, 843, 773, 752, 725, 700, 662, 527 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{21}H_{32}N_2O_2Si$ $(M + H)^+$ 373.2306, found 373.2306.

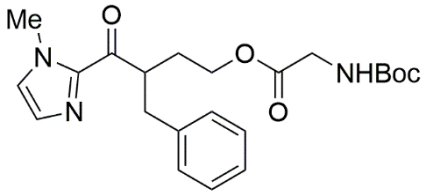


1-(1-methyl-1*H*-imidazol-2-yl)-2,3-diphenylpropan-1-one (3ga): (White solid, *n*-Hexane/EtOAc = 19/1 to 9/1, 49% yield, 28.6 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.5 Hz, 2H, Ar*H*), 7.28-7.25 (m, 2H, Ar*H*), 7.20-7.17 (m, 5H, Ar*H*), 7.12-7.11 (m, 1H, Ar*H*), 7.09 (s, 1H, *imidazole*), 6.92 (s, 1H, *imidazole*), 5.52-5.49 (m, 1H, COC*H*), 3.91 (s, 3H, NCH_3), 3.52 (dd, *J* = 14.0, 8.5 Hz, 1H, ArCH_2), 3.11 (dd, *J* = 14.0, 7.0 Hz, 1H, ArCH_2); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.8, 142.8, 139.6, 139.0, 129.3, 129.1, 128.8, 128.5, 128.2, 127.3, 127.0, 126.0, 54.2, 38.9, 36.2; IR (neat) 1667, 1452, 1398, 949, 910, 903, 791, 750, 739, 718, 696, 679, 665, 544, 521 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{19}H_{18}N_2O$ $(M + H)^+$ 291.1492, found 291.1510.

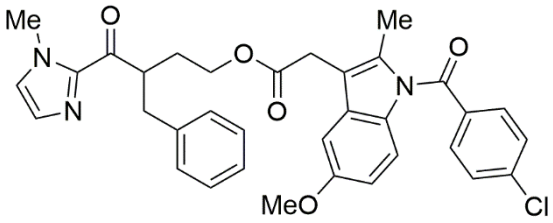


1-(1-methyl-1*H*-imidazol-2-yl)-3-phenyl-2-(thiophen-3-yl)propan-1-one (3ha): (Yellow solid,

n-Hexane/EtOAc = 9/1 to 4/1, 47% yield, 27.9 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22-7.11 (m, 9H, Ar*H*, *imidazole*), 6.97 (s, 1H, *imidazole*), 5.66-5.63 (m, 1H, COC*H*), 3.93 (s, 3H, NCH_3), 3.47 (dd, *J* = 13.5, 8.5 Hz, 1H, ArCH_2), 3.12 (dd, *J* = 13.5, 7.0 Hz, 1H, ArCH_2); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.4, 142.6, 139.4, 139.1, 129.3, 129.1, 128.2, 127.8, 127.4, 126.1, 125.4, 122.7, 49.6, 38.7, 36.3; IR (neat) 1670, 1454, 1400, 1377, 949, 905, 785, 766, 754, 727, 704, 694, 681, 606, 542, 521 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{17}H_{16}N_2OS$ $(M + H)^+$ 297.1056, found 297.1051.

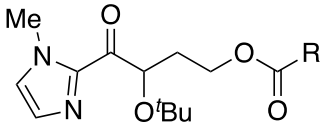


3-benzyl-4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl (*tert*-butoxycarbonyl)glycinate (3ia): (White liquid, *n*-Hexane/EtOAc = 4/1 to 2/1, 50% yield, 41.0 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26-7.20 (m, 4H, Ar*H*), 7.16 (tt, *J* = 7.0, 1.5 Hz, 1H, Ar*H*), 7.12 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 5.05 (br, 1H, N*H*), 4.34-4.29 (m, 1H, COC*H*), 4.15-4.07 (m, 2H, OCH_2), 3.97 (s, 3H, NCH_3), 3.81 (dd, *J* = 18.0, 5.5 Hz, 1H, COCH_2NH), 3.70 (dd, *J* = 18.0, 5.5 Hz, 1H, COCH_2NH), 3.12 (dd, *J* = 13.5, 6.5 Hz, 1H, ArCH_2), 2.75 (dd, *J* = 13.5, 8.0 Hz, 1H, ArCH_2), 2.19-2.11 (m, 1H, COCHCH_2), 1.90-1.83 (m, 1H, COCHCH_2), 1.44 (s, 9H, NHC(CH_3)$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.7, 170.1, 155.6, 142.8, 139.0, 129.3, 129.2, 128.4, 127.4, 126.3, 79.9, 63.5, 45.3, 42.3, 38.3, 36.3, 29.9, 28.3; IR (neat) 1749, 1711, 1668, 1508, 1454, 1406, 1366, 1285, 1250, 1194, 1155, 1055, 978, 912, 777, 731, 700 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{22}H_{29}N_3O_5$ $(M + H)^+$ 416.2180, found 416.2176.



3-benzyl-4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate (3ja): (Yellow solid, *n*-Hexane/Acetone = 9/1 to 4/1, 66% yield, 77.5 mg); byproduct (O^{t}Bu) mixture ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 6.5 Hz, 2H, Ar*H*), 7.44 (d, *J* = 6.5 Hz, 2H, Ar*H*), 7.22-7.19 (m, 2H, Ar*H*), 7.16-7.13 (m, 3H, Ar*H*), 7.08 (s, 1H, *imidazole*), 6.99 (s, 1H, *imidazole*), 6.93 (d, *J* = 2.5 Hz, 1H, Ar*H*), 6.84 (d, *J* = 9.0 Hz, 1H, Ar*H*), 6.65 (dd, *J* = 9.0, 2.5 Hz, 1H, Ar*H*), 4.32-4.27 (m, 1H, COC*H*), 4.08 (t, *J* = 6.5 Hz, 2H,

OC*H*$_2$), 3.95 (s, 3H, NC*H*$_3$), 3.81 (s, 3H, OC*H*$_3$), 3.54 (d, *J* = 1.5 Hz, 2H, COC*H*$_2$Ar), 3.10 (dd, *J* = 13.5, 6.5 Hz, 1H, PhC*H*$_2$), 2.72 (dd, *J* = 13.5, 7.5 Hz, 1H, PhC*H*$_2$), 2.32 (s, 3H, ArC*H*$_3$), 2.18-2.11 (m, 1H, COCHC*H*$_2$), 1.87-1.81 (m, 1H, COCHC*H*$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.7, 170.7, 168.3, 156.0, 142.8, 139.2, 139.0, 136.0, 133.9, 131.2, 130.7, 130.7, 129.3, 129.1, 129.1, 128.3, 127.3, 126.3, 115.0, 112.5, 111.8, 101.1, 63.2, 55.7, 45.3, 38.2, 36.3, 30.1, 29.9, 13.4; IR (neat) 1732, 1672, 1476, 1454, 1406, 1356, 1314, 1288, 1261, 1221, 1163, 1088, 1067, 1034, 1015, 910, 833, 754, 729, 700, 480 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{34}H_{32}ClN_3O_5$ (M + H)$^+$ 598.2103, found 598.2061.



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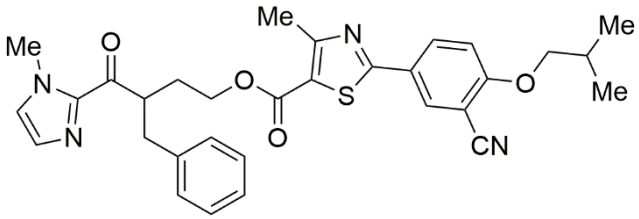
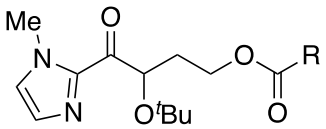
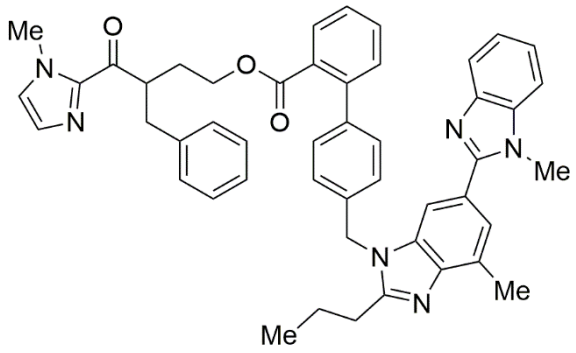


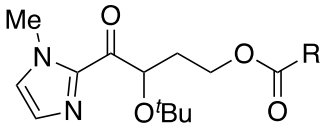
3-benzyl-4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl 2-(3-cyano-4-isobutoxyphenyl)-4-methylthiazole-5-carboxylate (3ka): (Pale yellow solid, *n*-Hexane/EtOAc = 4/1 to 2/1, 84% yield, 93.4 mg); byproduct (O^{t}Bu) mixture ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.12 (d, *J* = 2.5 Hz, 1H, Ar*H*), 8.07 (dd, *J* = 9.0, 2.5 Hz, 1H, Ar*H*), 7.27-7.23 (m, 4H, Ar*H*), 7.20-7.17 (m, 1H, Ar*H*), 7.13 (s, 1H, *imidazole*), 7.01 (d, *J* = 9.0 Hz, 1H, Ar*H*), 6.99 (s, 1H, *imidazole*), 4.41-4.35 (m, 1H, COC*H*), 4.32-4.28 (m, 1H, OC*H*$_2$CH$_2$), 4.26-4.21 (m, 1H, OC*H*$_2$CH$_2$), 3.91 (s, 3H, NC*H*$_3$), 3.90 (d, *J* = 6.5 Hz, 2H, OC*H*$_2$CH), 3.17 (dd, *J* = 13.5, 6.5 Hz, 1H, ArC*H*$_2$), 2.78 (dd, *J* = 13.5, 8.5 Hz, 1H, ArC*H*$_2$), 2.67 (s, 3H, ArC*H*$_3$), 2.35-2.27 (m, 1H, COCHC*H*$_2$), 2.21 (sep, *J* = 6.5 Hz, 1H, OCH$_2$C*H*) 1.98-1.91 (m, 1H, COCHC*H*$_2$), 1.10 (d, *J* = 6.5 Hz, 6H, CH(C*H*$_3$)$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.6, 167.1, 162.5, 161.7, 161.2, 142.8, 139.0, 132.5, 132.0, 129.4, 129.2, 128.4, 127.3, 126.4, 126.0, 121.7, 115.5, 112.6, 102.9, 75.7, 63.8, 45.7, 38.5, 36.2, 29.7, 28.2, 19.1, 19.1, 17.4; IR (neat) 1688, 1668, 1603, 1433, 1406, 1327, 1292, 1271, 1130, 1107, 1045, 1005, 991, 912, 827, 760, 737, 725, 698 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{31}H_{32}N_4O_4S$ (M + H)$^+$ 557.2217, found 557.2193.



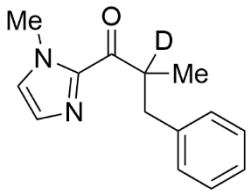
byproduct



3-benzyl-4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl 4'-((1,7'-dimethyl-2'-propyl-1*H*,3'*H*-[2,5'-bibenzo[*d*]imidazol]-3'-yl)methyl)-[1,1'-biphenyl]-2-carboxylate (3la): (White solid, *n*-Hexane/Acetone = 4/1 to 1/1, 63% yield, 47.4 mg); byproduct (O^{t}Bu) mixture ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81-7.79 (m, 1H, Ar*H*), 7.61 (dd, *J* = 7.5, 1.5 Hz, 1H, Ar*H*), 7.46-7.45 (m, 3H, Ar*H*), 7.36-7.13 (m, 12H, Ar*H*), 7.04-7.02 (m, 3H, *imidazole*, Ar*H*), 6.86 (s, 1H, *imidazole*), 5.42 (s, 2H, ArC*H*$_2$Ar), 4.29-4.23 (m, 1H, COC*H*), 4.11-4.06 (m, 1H, OC*H*$_2$), 4.01-3.97 (m, 1H, OC*H*$_2$), 3.77 (s, 3H, NC*H*$_3$), 3.71 (s, 3H, NC*H*$_3$), 3.04 (dd, *J* = 13.5, 6.5 Hz, 1H, PhC*H*$_2$), 2.94 (dd, *J* = 8.0, 6.5 Hz, 2H, ArC*H*$_2$CH$_2$), 2.77 (s, 3H, ArC*H*$_3$), 2.67 (dd, *J* = 13.5, 8.5 Hz, 1H, ArC*H*$_2$), 2.06-1.99 (m, 1H, COCHC*H*$_2$), 1.91-1.84 (m, 2H, ArCH$_2$C*H*$_2$CH$_3$), 1.76-1.70 (m, 1H, COCHC*H*$_2$), 1.06 (t, *J* = 7.5 Hz, 3H, CH$_2$C*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.7, 167.5, 156.5, 154.8, 143.2, 143.0, 142.8, 142.0, 141.0, 139.0, 136.7, 135.0, 134.7, 131.3, 130.7, 130.5, 129.9, 129.5, 129.2, 129.1, 129.1, 128.3, 127.2, 127.2, 126.3, 125.8, 123.9, 123.9, 122.5, 122.3, 119.6, 109.6, 109.0, 63.3, 47.1, 45.6, 38.2, 36.0, 31.9, 30.0, 29.9, 22.0, 16.9, 14.1; IR (neat) 1721, 1670, 1452, 1406, 1321, 1281, 1244, 1128, 1084, 1005, 974, 912, 760, 735, 700 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{48}H_{46}N_6O_3$ (M + H)$^+$ 755.3704, found 755.3572.



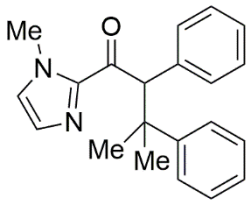
byproduct



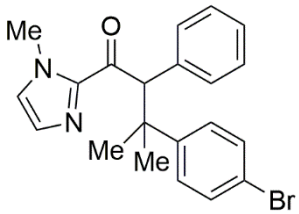
2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one-2-*d* (3aa(*d*^{1})): (Pale yellow liquid,

n-Hexane/EtOAc = 9/1 to 4/1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26-7.22 (m, 4H, Ar*H*), 7.18-7.14 (m, 1H, Ar*H*), 7.14 (s, 1H, *imidazole*), 7.01 (s, 1H, *imidazole*), 3.97 (s, 3H, NCH_3), 3.14 (d, *J* = 13.5 Hz, 1H, ArCH_2), 2.67 (d, *J* = 13.5 Hz, 1H, ArCH_2), 1.18 (s, 3H, COCDCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.2, 142.5, 139.9, 129.2, 129.0, 128.2, 127.1, 126.0, 42.6 (t, *J* = 20.4 Hz), 38.9, 36.3, 16.9; IR (neat) 1667, 1452, 1396, 1153, 991, 974, 914, 893, 770, 743, 698, 662, 515, 482 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{15}DN_2O$ $(M + H)^+$ 230.1398, found 230.1398.

Construction of All-Carbon Quaternary Center

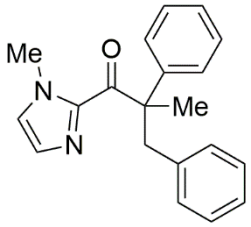


3-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-2,3-diphenylbutan-1-one (8): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%), 1-(1-methyl-1*H*-imidazol-2-yl)-2-phenylethan-1-one (**1g**) (1.0 equiv.) were added followed by the addition of cumene (0.20 M). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product (**8**). : (White solid, Toluene/EtOAc = 1/0 to 19/1, 34% yield, 21.8 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (dd, *J* = 8.0, 1.0 Hz, 2H, Ar*H*), 7.33 (dd, *J* = 8.0, 2.0 Hz, 2H, Ar*H*), 7.26-7.18 (m, 5H, Ar*H*), 7.10 (tt, *J* = 7.0, 1.0 Hz, 1H, Ar*H*), 7.02 (s, 1H, *imidazole*), 6.84 (s, 1H, *imidazole*), 5.70 (s, 1H, COC*H*), 3.80 (s, 3H, NCH_3), 1.56 (s, 3H, ArC(CH_3)$_2$), 1.41 (s, 3H, ArC(CH_3)$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.7, 147.8, 144.0, 135.8, 130.8, 128.7, 127.7, 127.6, 126.9, 126.9, 126.7, 125.8, 61.2, 42.0, 36.2, 27.6, 25.1; IR (neat) 1672, 1402, 1385, 1366, 1028, 914, 843, 804, 775, 756, 718, 694, 665, 613, 559 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{21}H_{22}N_2O$ (M + H)$^+$ 319.1805, found 319.1800.

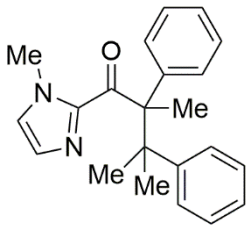


3-(4-bromophenyl)-3-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-2-phenylbutan-1-one (9): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%), 1-(1-methyl-1*H*-imidazol-2-yl)-2-phenylethan-1-one (**1g**) (1.0 equiv.) were added followed by the addition of 4-bromocumene (0.20 M). Then DTBP (3.3 equiv.) was added to the reaction mixture. The

mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product (**9**): (White solid, *n*-Hexane/EtOAc = 19/1 to 9/1, 56% yield, 44.8 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33-7.30 (m, 4H, Ar*H*), 7.26-7.22 (m, 5H, Ar*H*), 7.03 (s, 1H, *imidazole*), 6.87 (s, 1H, *imidazole*), 5.67 (s, 1H, COC*H*), 3.82 (s, 3H, NCH_3), 1.52 (s, 3H, ArC(CH_3)$_2$), 1.38 (s, 3H, ArC(CH_3)$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.2, 146.9, 143.7, 135.4, 130.8, 130.6, 128.8, 128.6, 127.8, 127.1, 127.1, 119.7, 60.9, 41.6, 36.3, 27.8, 25.1; IR (neat) 1672, 1402, 1383, 1096, 1026, 1005, 914, 845, 831, 824, 773, 739, 727, 718, 696, 642, 557, 542 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{21}H_{21}BrN_2O$ $(M + H)^+$ 397.0910, found 397.0909.

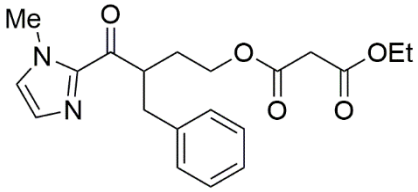


2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-2,3-diphenylpropan-1-one (10): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and 1-(1-methyl-1*H*-imidazol-2-yl)-2-phenylpropan-1-one (**1m**) (1.0 equiv.) were added followed by the addition of cumene (0.20 M). Then DTBP (3.3 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 120 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product (**10**). : (White solid, *n*-Hexane/EtOAc = 19/1 to 9/1, Toluene/EtOAc = 1/0 to 19/1, 41% yield, 24.9 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29-7.26 (m, 2H, Ar*H*), 7.21-7.17 (m, 3H, Ar*H*), 7.15-7.08 (m, 3H, Ar*H*), 6.96 (s, 1H, *imidazole*), 6.84 (s, 1H, *imidazole*), 6.68 (d, *J* = 6.5 Hz, 2H, Ar*H*), 3.93 (s, 3H, NCH_3), 3.85 (d, *J* = 13.5 Hz, 1H, ArCH_2), 3.29 (d, *J* = 13.5 Hz, 1H, ArCH_2), 1.72 (s, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.6, 144.3, 142.0, 137.8, 130.7, 128.6, 128.2, 127.6, 126.4, 126.3, 126.2, 125.5, 55.6, 45.0, 36.8, 23.3; IR (neat) 1657, 1452, 1443, 1387, 1373, 974, 899, 781, 773, 748, 741, 698, 669, 644, 563, 527 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{20}H_{20}N_2O$ $(M + H)^+$ 305.1648, found 305.1644.



2,3-dimethyl-1-(1-methyl-1*H*-imidazol-2-yl)-2,3-diphenylbutan-1-one (11): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and 1-(1-methyl-1*H*-imidazol-2-yl)-2-phenylpropan-1-one (**1m**) (1.0 equiv.) were added followed by the addition of cumene (0.20 M). Then DTBP (3.3 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 120 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product (**11**). : (White solid, *n*-Hexane/EtOAc = 9/1 to 4/1, *n*-Hexane/Et_2O = 4/1 to 2/1, 38 % yield, 25.3 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.16-7.10 (m, 6H, Ar*H*), 6.99 (br, 2H, Ar*H*), 6.82 (d, *J* = 7.0 Hz, 2H, Ar*H*), 6.77 (s, 1H, *imidazole*), 6.73 (s, 1H, *imidazole*), 3.93 (s, 3H, NCH_3), 2.03 (s, 3H, COCHCH_3), 1.69 (s, 3H, ArC(CH_3)$_2$), 1.56 (s, 3H, ArC(CH_3)$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.8, 146.7, 142.7, 141.5, 129.8, 129.2, 127.7, 126.7, 126.3, 126.2, 125.5, 124.8, 59.9, 45.4, 37.0, 25.8, 25.1, 21.0; IR (neat) 1668, 1443, 1381, 1369, 1287, 1234, 955, 908, 806, 773, 758, 700, 667, 658, 598 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{22}H_{24}N_2O$ $(M + H)^+$ 333.1961, found 333.1944.

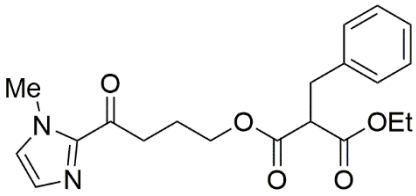
Chemoselective Benzylation



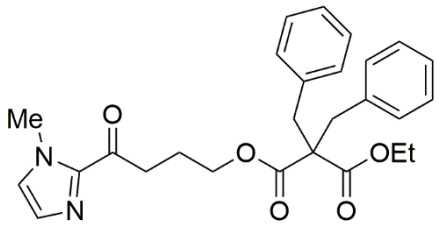
3-benzyl-4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl ethyl malonate (3na): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%) were added followed by the addition of toluene (0.20 M) and ethyl (4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl) malonate (1.0 equiv.). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product (**3na**). : (Colorless liquid, *n*-Hexane/EtOAc = 4/1 to 2/1, 53% yield, 39.4 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26-7.20 (m, 4H, Ar*H*), 7.16 (tt, *J* = 7.5, 2.0 Hz, 1H, Ar*H*), 7.13 (s, 1H, *imidazole*), 7.01 (s, 1H, *imidazole*), 4.33-4.27 (m, 1H, COC*H*), 4.16 (q, *J* = 7.0 Hz, 2H, OC*H*$_2$CH$_3$), 4.11 (dt, *J* = 7.0, 1.5 Hz, 2H, OC*H*$_2$CH$_2$), 3.97 (s, 3H, NC*H*$_3$), 3.24 (s, 2H, COC*H*$_2$CO), 3.13 (dd, *J* = 13.5, 6.5 Hz, 1H, ArC*H*$_2$), 2.76 (dd, *J* = 13.5, 8.0 Hz, 1H, ArC*H*$_2$), 2.19-2.11 (m, 1H, COCHC*H*$_2$), 1.90-1.84 (m, 1H, COCHC*H*$_2$), 1.25 (t, *J* = 7.0 Hz, 3H, OCH$_2$C*H*$_3$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.6, 166.5, 166.5, 142.8, 139.0, 129.3, 129.2, 128.3, 127.3, 126.3, 63.7, 61.5, 45.3, 41.5, 38.3, 36.3, 29.7, 14.0; IR (neat) 1748, 1728, 1668, 1454, 1406, 1368, 1329, 1267, 1184, 1148, 1030, 986, 912, 775, 745, 700 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{20}H_{24}N_2O_5$ $(M + H)^+$ 373.1758, found 373.1757.

Benzylation using KO^{t}Bu and Benzyl bromide

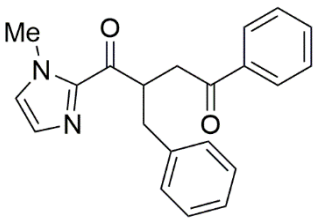
To a solution of ethyl (4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl) malonate (**1n**) (1.0 equiv.) in dry THF (0.20 M) under argon atmosphere was added potassium *tert*-butoxide (1.0 equiv.) in dry THF (0.20 M) at 0 °C. The cooling bath was removed and the reaction mixture was stirred at room temperature. After 2 h, the mixture was again cooled to 0 °C and benzyl bromide (1.7 equiv.) was added. The cooling bath was removed and the reaction mixture was stirred for 1 h at room temperature. After removal of solvent under reduced pressure, the residue was purified by silica gel flash chromatography to obtain **12** and **13**.



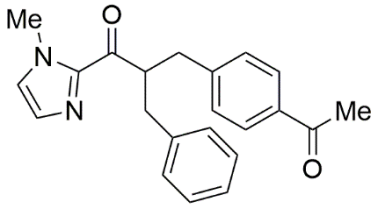
1-ethyl 3-(4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl) 2-benzylmalonate (12): (Colorless liquid, *n*-Hexane/EtOAc = 2/1 to 1/1, 55% yield, 20.3 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27-7.24 (m, 2H, Ar*H*), 7.20-7.16 (m, 3H, Ar*H*), 7.12 (s, 1H, *imidazole*), 7.03 (s, 1H, *imidazole*), 4.19 (t, *J* = 6.5 Hz, 2H, OCH_2CH_2), 4.14 (dq, *J* = 7.5, 2.0 Hz, 2H, OCH_2CH_3), 4.00 (s, 3H, NCH_3), 3.65 (t, *J* = 8.0 Hz, 1H, COC*H*CO), 3.20 (d, *J* = 8.0 Hz, 2H, $ArCH_2$), 3.15 (t, *J* = 7.0 Hz, 2H, $COCH_2$), 2.00 (m, 2H, $COCH_2CH_2$), 1.19 (t, *J* = 7.0 Hz, 3H, OCH_2CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ191.7, 168.9, 168.8, 142.8, 137.8, 129.1, 128.8, 128.5, 127.0, 126.7, 64.8, 61.6, 53.8, 36.2, 35.2, 34.7, 22.9, 14.0; IR (neat) 1728, 1674, 1454, 1408, 1368, 1335, 1288, 1225, 1150, 1082, 1057, 1028, 989, 914, 777, 752, 698 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{20}H_{24}N_2O_5$ $(M + H)^+$ 373.1758, found 373.1751.



1-ethyl 3-(4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutyl) 2,2-dibenzylmalonate (13): (Colorless liquid, *n*-Hexane/EtOAc = 2/1 to 1/1, 16% yield, 7.3 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27-7.24 (m, 4H, Ar*H*), 7.22-7.19 (m, 2H, Ar*H*), 7.18-7.16 (m, 4H, Ar*H*), 7.11 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 4.12-4.06 (m, 4H, OCH_2CH_2, OCH_2CH_3), 4.00 (s, 3H, NCH_3), 3.20 (s, 4H, $COC(CH_2Ar)_2$), 3.10 (t, *J* = 7.0 Hz, 2H, $COCH_2$), 1.95 (quin, *J* = 7.0 Hz, 2H, $COCH_2CH_2$), 1.14 (t, *J* = 7.0 Hz, 3H, OCH_2CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.7, 171.0, 170.9, 142.8, 136.3, 130.1, 129.1, 128.2, 127.0, 126.9, 64.7, 61.4, 60.3, 39.2, 36.2, 35.2, 22.8, 13.9; IR (neat) 1724, 1674, 1410, 1288, 1269, 1246, 1194, 1173, 1155, 1086, 1036, 989, 914, 768, 743, 698 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{27}H_{30}N_2O_5$ $(M + H)^+$ 463.2227, found 463.2228.



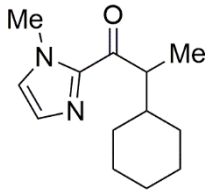
2-benzyl-1-(1-methyl-1*H*-imidazol-2-yl)-4-phenylbutane-1,4-dione (3oa): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%) were added followed by the addition of toluene (0.20 M) and 1-(1-methyl-1*H*-imidazol-2-yl)-4-phenylbutane-1,4-dione (1.0 equiv.). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product (**3oa**). : (Pale yellow solid, *n*-Hexane/EtOAc = 9/1 to 4/1, 51% yield, 33.7 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.0 Hz, 2H, Ar*H*), 7.52 (t, *J* = 7.5 Hz, 1H, Ar*H*), 7.40 (t, *J* = 7.5 Hz, 2H, Ar*H*), 7.33-7.26 (m, 4H, Ar*H*), 7.22-7.18 (m, 2H, *imidazole*, Ar*H*), 7.03 (s, 1H, *imidazole*), 4.75-4.69 (m, 1H, $PhCOCH_2CH$), 3.98 (s, 3H, NCH_3), 3.63 (dd, *J* = 13.0, 10.0 Hz, 1H, $COCH_2$), 3.34 (dd, *J* = 13.5, 5.0 Hz, 1H, $ArCH_2$), 3.07 (dd, *J* = 13.0, 9.0 Hz, 1H, $COCH_2$), 2.71 (dd, *J* = 13.5, 10.0 Hz, 1H, $ArCH_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.3, 194.2, 142.6, 139.0, 136.5, 133.0, 129.3, 129.2, 128.4, 128.4, 128.0, 126.9, 126.4, 44.3, 39.6, 37.9, 36.2; IR (neat) 1672, 1410, 1356, 1219, 997, 951, 910, 791, 770, 758, 743, 725, 698, 687, 669, 556, 492 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{21}H_{20}N_2O_2$ $(M + H)^+$ 333.1598, found 333.1598.



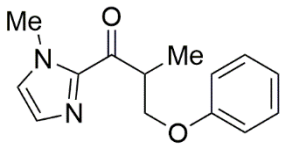
2-(4-acetylbenzyl)-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one (3pa): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%) were added followed by the addition of toluene (0.20 M) and 3-(4-acetylphenyl)-1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (1.0 equiv.). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product (**3pa**). : (White solid, Toluene/EtOAc = 1/0 to 4/1, 71% yield, 49.2 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.5 Hz, 2H, Ar*H*), 7.27 (d, *J* = 7.5 Hz, 2H, Ar*H*), 7.25-7.20 (m, 4H, Ar*H*), 7.15 (tt, *J* = 6.5, 1.5 Hz, 1H, Ar*H*), 7.08 (s,

1H, *imidazole*), 6.94 (s, 1H, *imidazole*), 4.63-4.57 (m, 1H, COC*H*), 3.89 (s, 3H, NCH_3), 3.20-3.14 (m, 2H, ArCH_2), 2.82 (dd, *J* = 13.5, 6.5 Hz, 1H, PhCH_2), 2.75 (dd, *J* = 13.5, 7.5 Hz, 1H, PhCH_2), 2.54 (s, 3H, COCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.9, 194.2, 145.6, 142.9, 139.2, 135.2, 129.3, 129.2, 129.1, 128.4, 128.3, 127.1, 126.3, 49.7, 37.9, 37.2, 36.1, 26.5; IR (neat) 1672, 1605, 1404, 1356, 1265, 945, 908, 772, 745, 698, 677, 598, 584, 573 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{22}H_{22}N_2O_2$ $(M + H)^+$ 347.1754, found 347.1754.

Dehydrogenative Cross Coupling using 2-Acylimidazole 1a

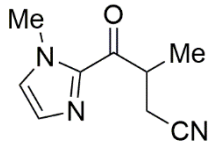


2-cyclohexyl-1-(1-methyl-1*H*-imidazol-2-yl)propan-1-one (15): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%) were added followed by the addition of cyclohexane (0.50 ml) and **1a** (30 µL, 1.0 equiv.), chlorobenzene (0.64 ml). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 100 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product. : (Colorless liquid, *n*-Hexane/EtOAc = 9/1 to 4/1, 29 % yield, 14.8 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (s, 1H, *imidazole*), 7.02 (s, 1H, *imidazole*), 4.00 (s, 3H, NC*H*$_3$), 3.76 (quin, *J* = 7.5 Hz, 1H, COC*H*), 1.79-1.71 (m, 3H, *cyclohexyl*), 1.67-1.59 (m, 3H, *cyclohexyl*), 1.27-0.98 (m, 8H, COCHC*H*$_3$, *cyclohexyl*); ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.8, 143.3, 128.9, 127.1, 46.4, 40.7, 36.4, 31.8, 29.4, 26.4, 26.4, 26.3, 14.0; IR (neat) 2922, 2851, 1667, 1449, 1402, 1287, 1153, 966, 914, 903, 891, 762, 692, 665 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{13}H_{20}N_2O$ $(M + H)^+$ 221.1648, found 221.1651.



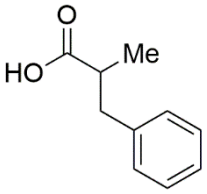
2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenoxypropan-1-one (17): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%) were added followed by the addition of anisole (0.20 M) and **1a** (30 µL, 1.0 equiv.). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 80 °C for 25 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product. **:** (Colorless liquid, *n*-Hexane/EtOAc = 9/1 to 4/1, 29% yield, 16.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24

(dd, *J* = 7.0, 1.5 Hz, 2H, Ar*H*), 7.17 (s, 1H, *imidazole*), 7.04 (s, 1H, *imidazole*), 6.93-6.88 (m, 3H, Ar*H*), 4.39-4.33 (m, 2H, ArCH_2), 4.12-4.09 (m, 1H, COC*H*), 4.00 (s, 3H, NCH_3), 1.33 (d, *J* = 6.5 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.9, 158.9, 142.6, 129.3, 129.2, 127.2, 120.7, 114.7, 69.7, 41.9, 36.2, 14.3; IR (neat) 1672, 1599, 1495, 1462, 1406, 1288, 1240, 1153, 1032, 974, 914, 752, 691, 509 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_{14}H_{16}N_2O_2$ $(M + H)^+$ 245.1285, found 245.1300.

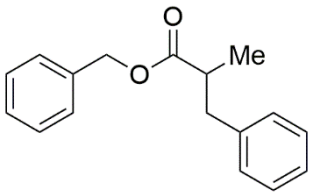


3-methyl-4-(1-methyl-1*H*-imidazol-2-yl)-4-oxobutanenitrile (19): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%) were added followed by the addition of acetonitrile (0.05 M) and **1a** (30 µL, 1.0 equiv.). Then dicumylperoxide (3.3 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 100 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product. : (Yellow liquid, *n*-Hexane/EtOAc = 9/1 to 2/1, 53% yield, 21.5 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.18 (s, 1H, *imidazole*), 6.98 (s, 1H, *imidazole*), 4.20-4.13 (m, 1H, COC*H*), 3.90 (s, 3H, NCH_3), 2.73 (dd, *J* = 17.0, 6.5 Hz, 1H, $COCH_2CH_2$), 2.62 (dd, *J* = 17.0, 7.5 Hz, 1H, $COCH_2CH_2$), 1.42 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.0, 141.4, 129.7, 127.7, 118.3, 38.6, 36.3, 20.4, 17.6; IR (neat) 1670, 1458, 1406, 1373, 1339, 1290, 1234, 1155, 1080, 978, 912, 885, 779, 691, 662, 623 cm^{-1}; HRMS (ESI, H) *m/z* calc'd for $C_9H_{11}N_3O$ $(M + H)^+$ 178.0975, found 178.0981.

6. Transformation of the Product[8]



2-methyl-3-phenylpropanoic acid (20): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, 2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one (**3aa**, 0.12 mmol, 1.0 equiv.) and CH_3CN (2.0 mL, 0.10 M), methyl trifluoromethanesulfonate (0.30 mmol, 1.5 equiv.) were added. After stirring for 2 h at room temperature, H_2O (0.44 ml, 203 equiv.) and DBU (1.2 mmol, 10 equiv.) were added and the reaction mixture was stirred for 1 h. After it was quenched by 1 M HCl aq, it was extracted with CH_2Cl_2. 2 N NaOH aq was added to the combined organic layers and extracted with CH_2Cl_2. 1 M HCl aq added to the combined organic layers and extracted again with CH_2Cl_2. After evaporation of the organic solvent under reduced pressure, product **20** was obtained. : CAS Registry Number 1009-67-2; (White solid, 75% yield, Extraction, 14.7 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30-7.27 (m, 2H, Ar*H*), 7.23-7.18 (m, 3H, Ar*H*), 3.08 (dd, *J* = 13.5, 6.5 Hz, 1H, ArCH_2), 2.81-2.74 (m, 1H, COC*H*), 2.68 (dd, *J* = 13.5, 8.0 Hz, 1H, ArCH_2), 1.18 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 181.4, 139.0, 129.0, 128.4, 126.4, 41.1, 39.3, 16.5.



benzyl 2-methyl-3-phenylpropanoate (21): A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, 2-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-3-phenylpropan-1-one (**3aa**, 0.12 mmol, 1.0 equiv.) and CH_3CN (2.0 mL, 0.10 M), methyl trifluoromethanesulfonate (0.30 mmol, 1.5 equiv.) were added. After stirring for 2 h at room temperature, BnOH (4.2 mmol, 35 equiv.) and DBU (1.2 mmol, 10 equiv.) were added and the reaction mixture was stirred for 1 h. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain product **21**. : CAS Registry Number 848613-18-3; (Colorless liquid, *n*-Hexane/Et_2O = 1/0 to 9/1, 91% yield, 27.7 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35-7.30 (m, 4H, Ar*H*),

7.27-7.24 (m, 3H, Ar*H*), 7.21-7.18 (m, 1H, Ar*H*), 7.15-7.13 (m, 2H, Ar*H*), 5.07 (s, 2H, OCH_2), 3.03 (dd, *J* = 13.5, 7.0 Hz, 1H, COCHCH_2), 2.84-2.77 (m, 1H, COC*H*), 2.68 (dd, *J* = 13.5, 7.5 Hz, 1H, COCHCH_2), 1.18 (d, *J* = 7.0 Hz, 3H, COCHCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.9, 139.3, 136.0, 129.0, 128.5, 128.4, 128.1, 128.1, 126.3, 66.1, 41.5, 39.7, 16.8.

7-1. UV – Vis Spectrum of Iron Catalyst[9]

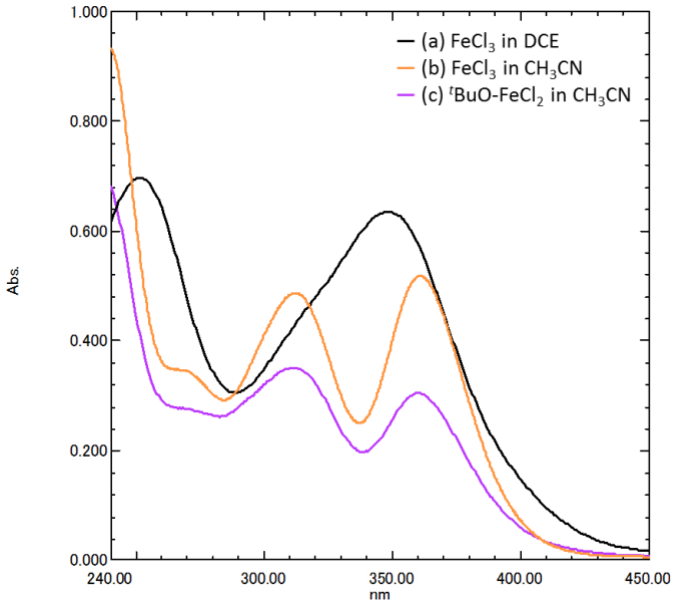


Figure S1. UV – Vis spectrum of Fe species

(a) $FeCl_3$ in DCE

Dichloroethane (1.0 mL) solution of $FeCl_3$ (0.020 mmol) was stirred for 0.5 h at 80°C under argon Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

(b) $FeCl_3$ in CH_3CN

Acetonitrile (1.0 mL) solution of $FeCl_3$ (0.020 mmol) was stirred for 0.5 h at room temperature under argon Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

(c) ^tBuO-$FeCl_2$ in CH_3CN

To a solution of $FeCl_2$ (1.0 equiv.) in PhCl (1.0 mL) was added DTBP (5.5 equiv.) at room temperature under argon atmosphere. The resulting mixture was stirred for 4 h at 120 °C. After removal of PhCl under vacuum, to the residue was added acetonitrile and stirred for 1 h at 80 °C. Then the mixture was subjected to the UV-Vis spectrum measurement (Figure S1).

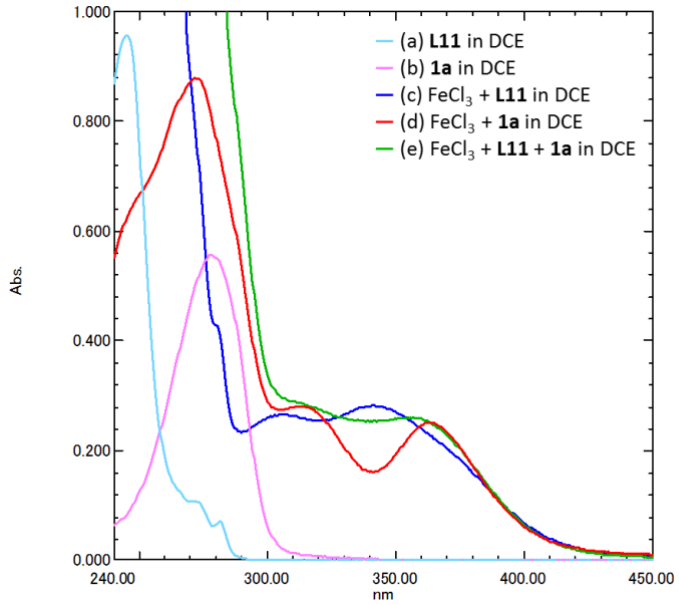


Figure S2. UV – Vis spectrum of $FeCl_3$ with Lewis bases (L11 and 1a)

(a) L11 in DCE

Dichloroethane (1.0 mL) solution of **L11** (0.020 mmol) was stirred for 0.5 h at 80°C under argon Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

(b) 1a in DCE

Dichloroethane (1.0 mL) solution of **1a** (0.020 mmol) was stirred for 0.5 h at 80 °C under argon Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

(c) $FeCl_3$ + L11 in DCE

Dichloroethane (1.0 mL) solution of $FeCl_3$ (0.020 mmol) and **L11** (0.020 mmol) was stirred for 3 h at room temperature under argon. Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

(d) $FeCl_3$ + 1a in DCE

Dichloroethane (1.0 mL) solution of $FeCl_3$ (0.020 mmol) and **1a** (0.020 mmol) was stirred for 3 h at room temperature under argon. Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

(e) $FeCl_3$ + L11 + 1a in DCE

Dichloroethane (1.0 mL) solution of $FeCl_3$ (0.020 mmol), **L11** (0.020 mmol) and **1a** (0.020 mmol) was stirred for 3 h at room temperature under argon. Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

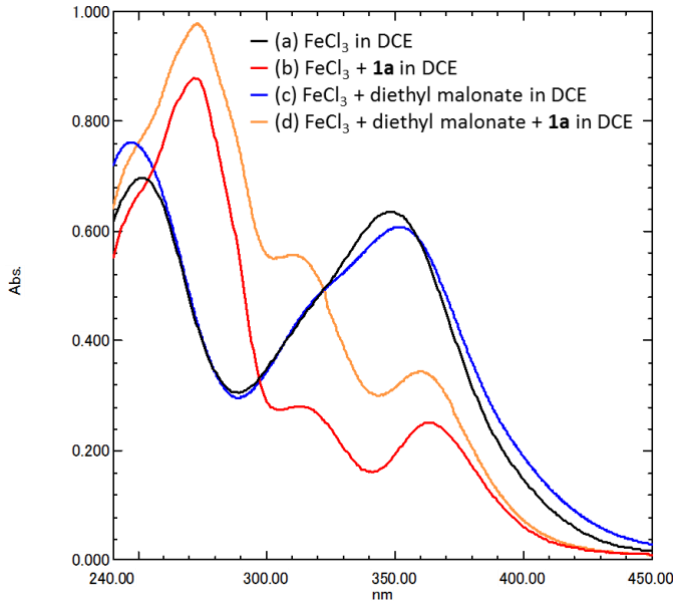


Figure S3. UV – Vis spectrum of $FeCl_3$ with Lewis bases (1a and diethylmalonate)

(a) $FeCl_3$ in DCE

Dichloroethane (1.0 mL) solution of $FeCl_3$ (0.020 mmol) was stirred for 0.5 h at 80°C under argon Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

(b) $FeCl_3$ + 1a in DCE

Dichloroethane (1.0 mL) solution of $FeCl_3$ (0.020 mmol) and **1a** (0.020 mmol) was stirred for 3 h at room temperature under argon. Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

(c) $FeCl_3$ + diethyl malonate in DCE

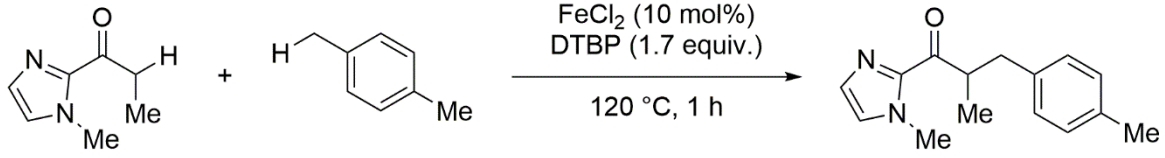
Dichloroethane (1.0 mL) solution of $FeCl_3$ (0.020 mmol), diethyl malonate (0.020 mmol) and **1a** (0.020 mmol) was stirred for 3 h at room temperature under argon. Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

(d) $FeCl_3$ + diethyl malonate + 1a in DCE

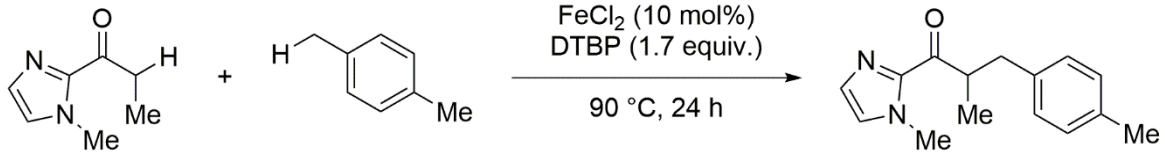
Dichloroethane (1.0 mL) solution of $FeCl_3$ (0.020 mmol) and diethyl malonate (0.020 mmol) was stirred for 3 h at room temperature under argon. Then the resultant mixture was subjected to the UV-Vis spectrum measurement.

7-2. Control Experiments

Effect of Lewis Bases

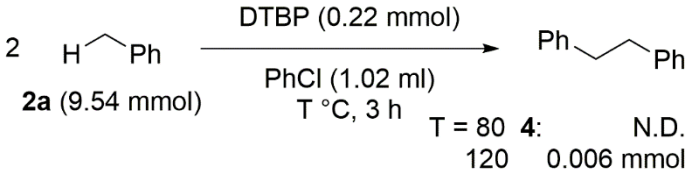


A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and **3aa** (0 or 1.0 equiv.) were added followed by the addition of toluene (0.20 M) and **1a** (30 µL, 1.0 equiv.). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 120 °C for 1 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was analyzed by ^{1}H-NMR to determine chemical yield using 1,2,4,5-tetramethyl benzene as an internal standard.

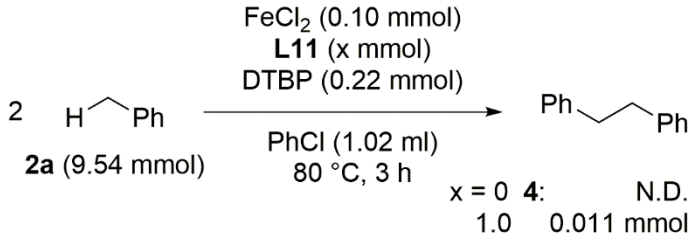


A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and **3aa** (10 mol%). **L11** (10 mol%) or none were added followed by the addition of toluene (0.20 M) and **1a** (30 µL, 1.0 equiv.). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 90 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was analyzed by ^{1}H-NMR to determine chemical yield using 1,2,4,5-tetramethyl benzene as an internal standard.

Effect of Lewis Bases in C–H Bond Cleavage

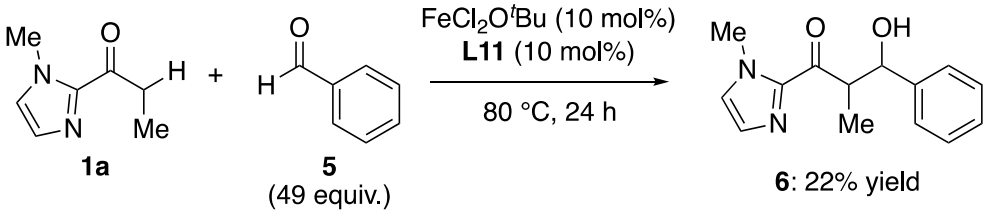


A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, toluene (1.02 ml) and PhCl (1.02 ml) were added followed by the addition of DTBP (0.22 mmol). The mixture was stirred under argon at 80 °C or 120 °C for 3 h and diluted with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was analyzed by ^{1}H-NMR to determine chemical yield using 1,2,4,5-tetramethylbenzene as an internal standard.

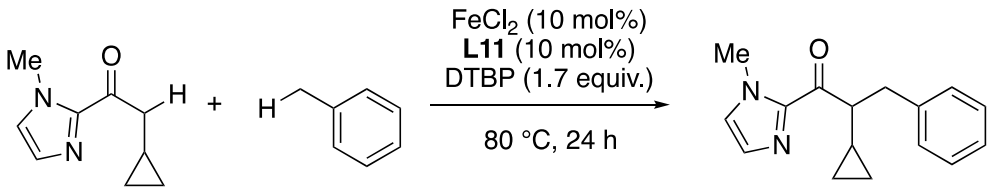


A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (0.10 mmol) and tris(4-methoxyphenyl)phosphine oxide (0 or 0.10 mmol) were added followed by the addition of toluene (1.02 ml) and PhCl (1.02 ml). Then DTBP (0.22 mmol) was added to the reaction mixture. The mixture was stirred under argon at 80 °C for 3 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was analyzed by ^{1}H-NMR to determine chemical yield using 1,2,4,5-tetramethylbenzene as an internal standard.

Detection of 2-Acylimidazole-derived Enolate

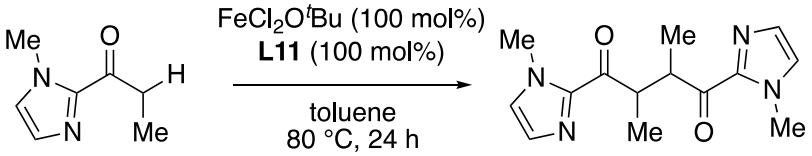


A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (0.023 mmol) and tris(4-methoxyphenyl)phosphine oxide (0.023 mmol) were added followed by the addition of benzene (1.14 ml) and DTBP (0.054 mmol). The mixture was stirred under argon at 80 °C for 2 h. After evaporation of the organic solvent under reduced pressure, benzaldehyde (1.14 ml) and **1a** (0.23 mmol) were added to the residue. The mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the volatiles under reduced pressure, the crude mixture was analyzed by ^{1}H-NMR to determine chemical yield.



1b **2a** **3ba**: 79% yield

A 20 mL sdhlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%) were added followed by the addition of toluene (0.20 M) and **1b** (1.0 equiv.). Then DTBP (1.7 equiv.) was added to the reaction mixture. The mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the organic solvent under reduced pressure, the resultant mixture was purified by silica gel flash chromatography to obtain cross coupling product.



1a **7**: Not detected

A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (0.23 mmol) and tris(4-methoxyphenyl)phosphine oxide (0.23 mmol) were added followed by the addition of benzene

(1.14 ml) and DTBP (0.11 mmol). The mixture was stirred under argon at 80 °C for 3 h. After evaporation of the organic solvent under reduced pressure, toluene (1.14 ml) and **1a** (0.23 mmol) were added to the residue. The mixture was stirred under argon at 80 °C for 24 h and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the volatiles under reduced pressure, the crude mixture was analyzed by ^{1}H-NMR.

7-3. Initial Kinetic Studies

Initial Rates of Varying Concentrations of Catalyst

A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (5.0, 7.5, 10, 12.5 mol%) and tris(4-methoxyphenyl)phosphine oxide (5.0, 7.5, 10, 12.5 mol%), 2-methoxynaphthalene (0.18 mmol) as an internal standard were added followed by the addition of toluene (0.20 M) and **1a** (50 µL, 0.53 mmol, 1.0 equiv.). The mixture was stirred under argon at 80 °C for 3 h to dissolve $FeCl_2$. Then DTBP (1.7 equiv.) was added to the reaction mixture. At the specified period, an aliquot of reaction mixture was extracted via a syringe filled by dry toluene with a stainless-steel needle from the test tube and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the volatiles under reduced pressure, the crude mixture was analyzed by ^{1}H NMR to determine chemical yield by the integration value of a peak at 2.68 ppm (**3aa**: ***H***-CHAr) and 3.93 ppm (2-methoxynaphthalene: ArO***CH$_3$***). The results were summarized in the Table S1 and Figures S4-S6.

Table S1. Initial Rates of Varying Concentrations of $FeCl_2$ and L11

Catalyst (mol%)	[Catalyst] (M)	ln([Catalyst])	*d*[**3aa**]/*dt* (M·min^{-1})	ln(*d*[**3aa**]/*dt*)
5	0.010	-4.61	0.000380	-7.88
7.5	0.015	-4.20	0.00100	-6.91
10	0.020	-3.91	0.00134	-6.62
12.5	0.025	-3.69	0.00245	-6.01

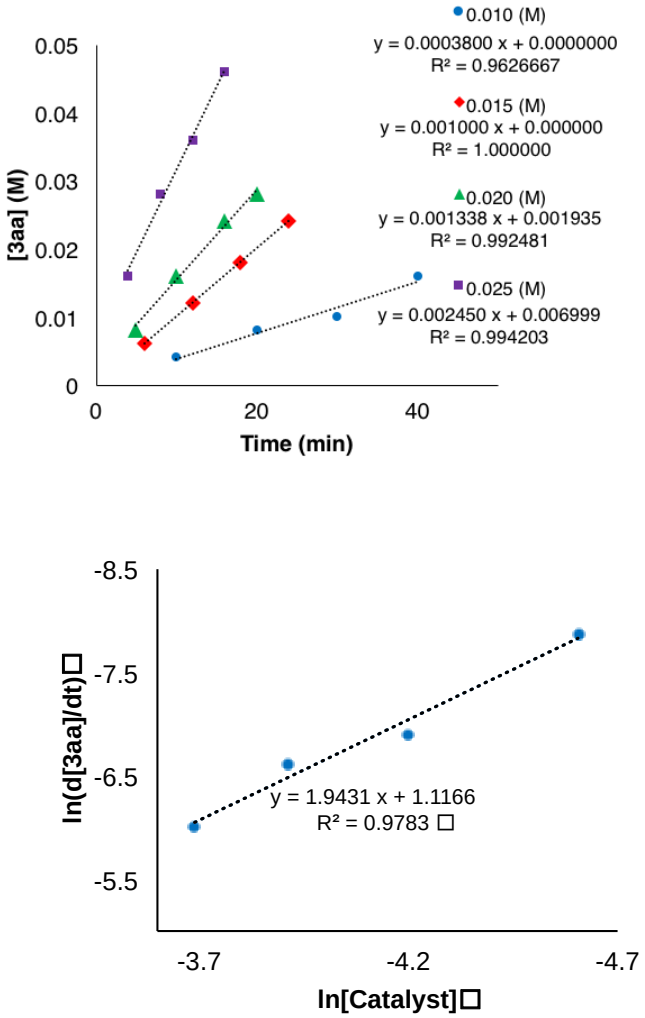


Figure S4. (a) Initial rate kinetic experiments for Catalyst. (b) Plot of ln (*d*[**3aa**]/*dt*) versus ln ([Catalyst]).

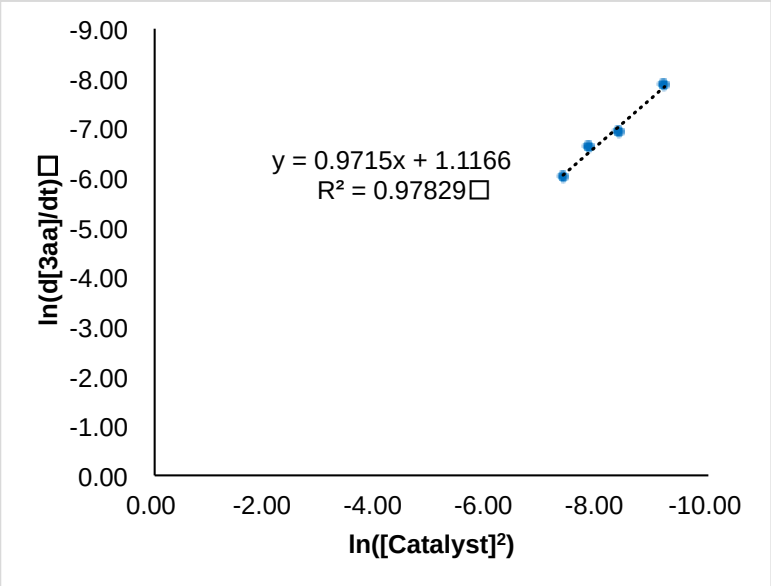


Figure S5. Plot of ln (*d*[**3aa**]/*dt*) versus ln ([Catalyst]^2).

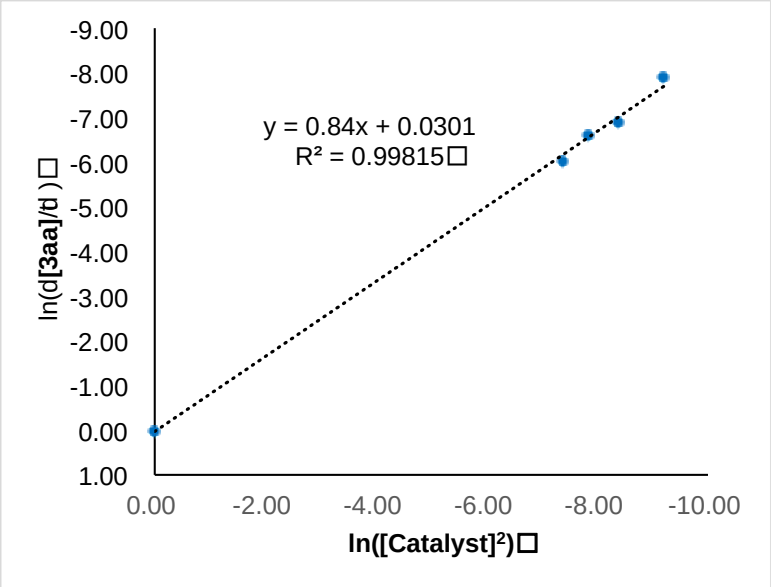


Figure S6. Plot of ln (*d*[**3aa**]/*dt* versus ln ([Catalyst]^2 that the fit passes through zero when [catalyst]=0).)

Initial Rates of Varying Concentrations of 2-Acylimidazole

A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (0.053 mmol) and tris(4-methoxyphenyl)phosphine oxide (0.053 mmol), 2-methoxynaphthalene (0.18 mmol) as an internal standard were added followed by the addition of toluene (2.66 ml) and **1a** (0.42 mmol, 0.53 mmol, 0.65 mmol, 0.76 mmol). The mixture was stirred under argon at 80 °C for 3 h to dissolve $FeCl_2$. Then DTBP (0.90 mmol) was added to the reaction mixture. At the specified period, an aliquot of reaction mixture was extracted via a syringe filled by dry toluene with a stainless-steel needle from the test tube and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the volatiles under reduced pressure, the crude mixture was analyzed by ^{1}H NMR to determine chemical yield by the integration value of a peak at 2.68 ppm (**3aa**: ***H***-CHAr) and 3.93 ppm (2-methoxynaphthalene: ArO***CH$_3$***). The results were summarized in the Table S2 and Figure S7.

Table S2. Initial Rates of Varying Concentrations of 2-Acylimidazole (1a)

1a (mmol)	[**1a**] (M)	ln[**1a**]	*d*[**3aa**]/*dt* (M·min^{-1})	ln(*d*[**3aa**]/*dt*)
0.42	0.16	-1.83	0.00185	-6.29
0.53	0.20	-1.61	0.00196	-6.23
0.65	0.24	-1.43	0.00194	-6.24
0.76	0.29	-1.24	0.00220	-6.12

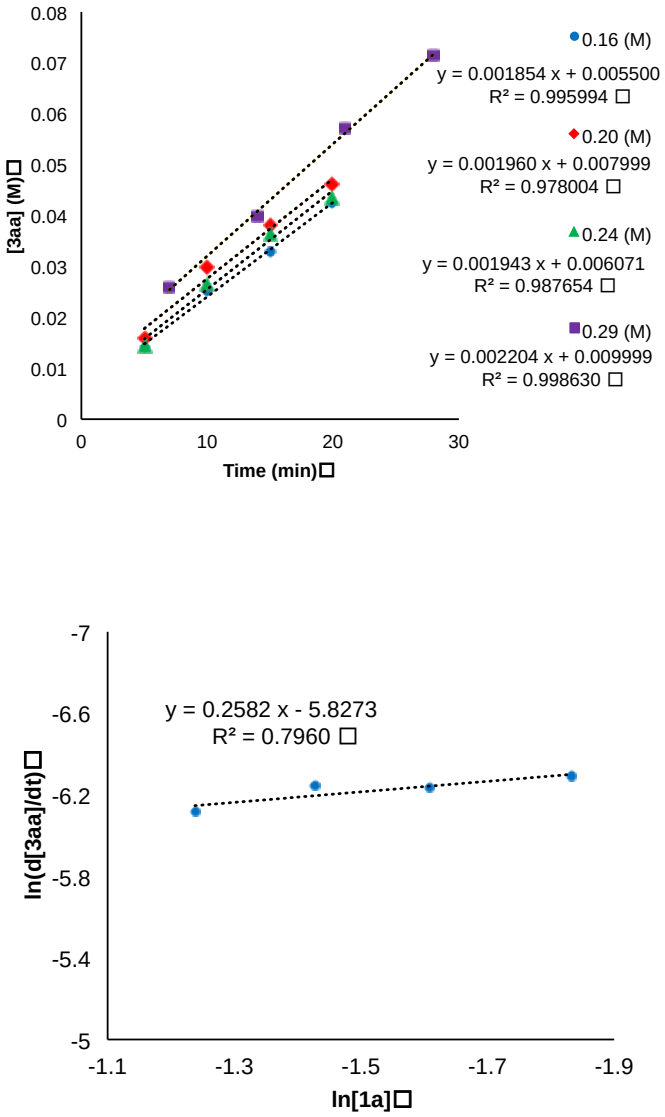


Figure S7. (a) Initial rate kinetic experiments for **1a**. (b) Plot of ln (*d*[**3aa**]/*dt*) versus ln ([**1a**]).

Initial Rates of Varying Concentrations of DTBP

A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%), 2-methoxynaphthalene (0.18 mmol) as internal standard were added followed by the addition of toluene (0.20 M) and **1a** (70 μL, 0.53 mmol, 1.0 equiv.). The mixture was stirred under argon at 80 °C for 3 h to dissolve $FeCl_2$. Then DTBP (1.7, 2.3, 2.8, 3.3 equiv.) was added to the reaction mixture. At the specified period, an aliquot of reaction mixture was extracted via a syringe filled by dry toluene with a stainless-steel needle from the test tube and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the volatiles under reduced pressure, the crude mixture was analyzed by ^{1}H NMR to determine chemical yield by the integration value of a peak at 2.68 ppm (**3aa**: ***H***-CHAr) and 3.93 ppm (2-methoxynaphthalene: ArO***CH$_3$***). The results were summarized in the Table S3 and Figure S8.

Table S3. Initial Rates of Varying Concentrations of DTBP

DTBP (equiv.)	[DTBP] (M)	ln[DTBP]	d[TM]/dt (M·min^{-1})	ln(d[TM]/dt)
1.7	0.34	-1.08	0.00134	-6.62
2.3	0.45	-0.80	0.00120	-6.73
2.8	0.56	-0.58	0.00135	-6.61
3.3	0.68	-0.39	0.00160	-6.44

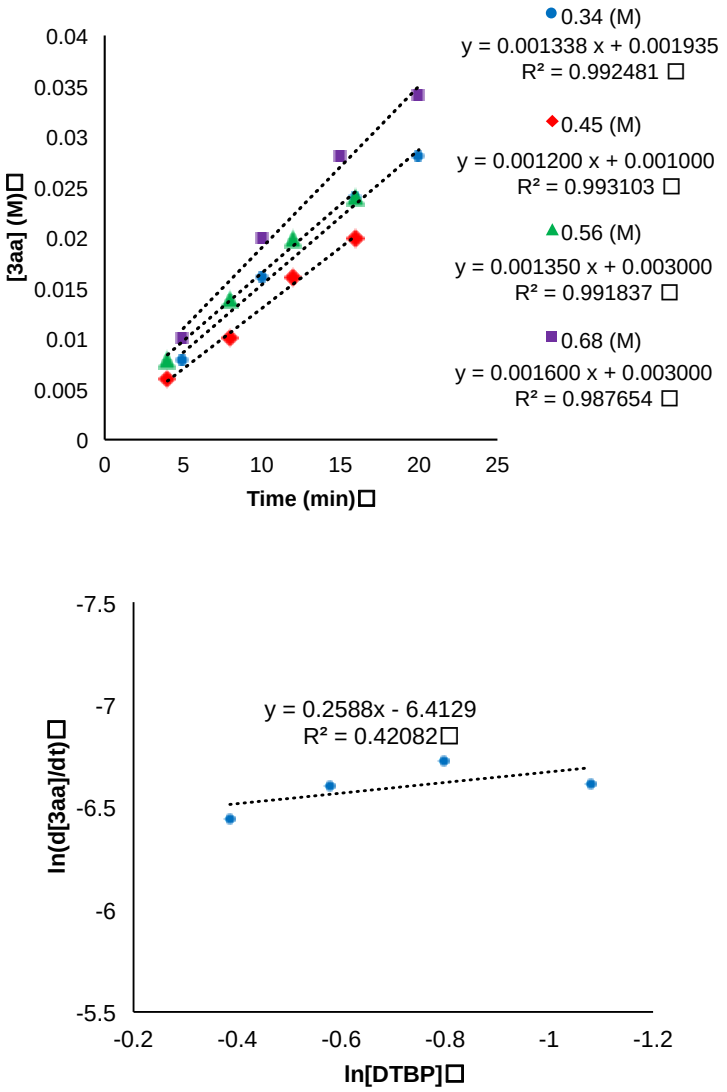


Figure S8. (a) Initial rate kinetic experiments for DTBP. (b) Plot of ln (*d*[**3aa**]/*dt*) versus ln ([DTBP]).

7-4. Kinetic Isotope Effect Experiments

Toluene/Toluene-*d*^{8}

A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%), 2-methoxynaphthalene (0.18 mmol) as internal standard were added followed by the addition of toluene or toluene-*d*^{8} (0.20 M) and 2-acylimidazole (70 µL, 0.53 mmol, 1.0 equiv.). The mixture was stirred under argon at 80 °C for 3 h to dissolve $FeCl_2$. Then DTBP (1.7 equiv.) was added to the reaction mixture. At the specified period, an aliquot of reaction mixture was extracted via a syringe filled by dry toluene with a stainless-steel needle from the test tube and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the volatiles under reduced pressure, the crude mixture was analyzed by ^{1}H NMR to determine chemical yield by the integration value of a peak at 4.19 ppm (**3aa**-*d*^{7}: CO***CH***) and 3.93 ppm (2-methoxynaphthalene: ArO***CH$_3$***). The results were summarized in the Figure S9.

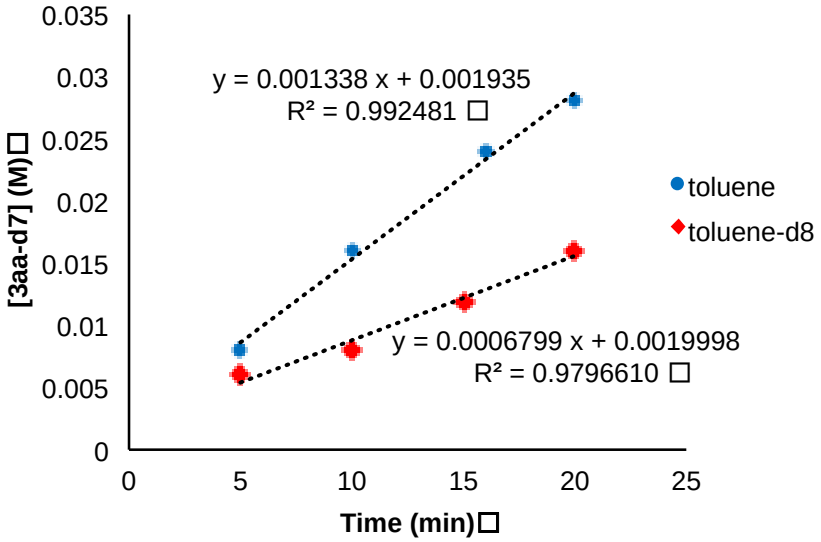


Figure S9. Kinetic Isotope Effect Experiments using toluene/toluene-*d*^{8}

2-Acylimidazole 1a/1a-*d*^{2}

A 20 mL schlenk flask equipped with a magnetic stirring bar and 3-way glass stopcock was flamed-dried under vacuum. After cooling down to room temperature, $FeCl_2$ (10 mol%) and tris(4-methoxyphenyl)phosphine oxide (10 mol%), 2-methoxynaphthalene (0.18 mmol) as internal standard were added followed by the addition of toluene (0.20 M) and **1a** or **1a**-*d*^{2} (70 µL, 1.0 equiv.). The mixture was stirred under argon at 80 °C for 3 h to dissolve $FeCl_2$. Then DTBP (1.7 equiv.) was added to the reaction mixture. At the specified period, an aliquot of reaction mixture was extracted via a syringe filled by dry toluene with a stainless-steel needle from the test tube and diluted with EtOAc. The diluted solution was filtered through silica short column and washed with EtOAc. After evaporation of the volatiles under reduced pressure, the crude mixture was analyzed by ^{1}H NMR to determine chemical yield by the integration value of a peak at 2.68 ppm (**3aa**-*d*^{1}: ***H***-CHAr) and 3.93 ppm (2-methoxynaphthalene: ArO***CH$_3$***). The results were summarized in Figure S10.

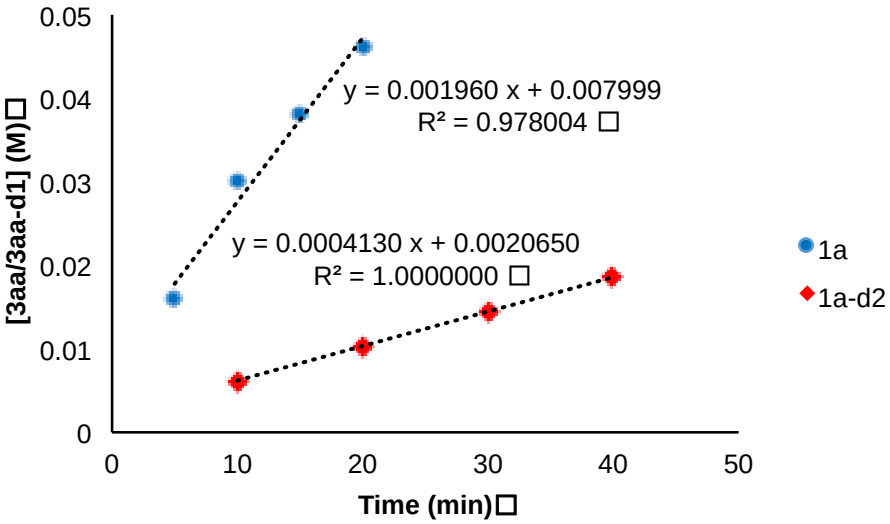


Figure S10. Kinetic Isotope Effect Experiments using **1a**/**1a-*d*^{2}**

7-5. ESI-mass Analysis

The premixed solution of $FeCl_3$ and **1a** was subjected to the ESI-mass analysis (Figure 11).

$FeCl_4^-$ species

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found: m/z = 197.8087, 195.8115, 199, 8054, 201.8022

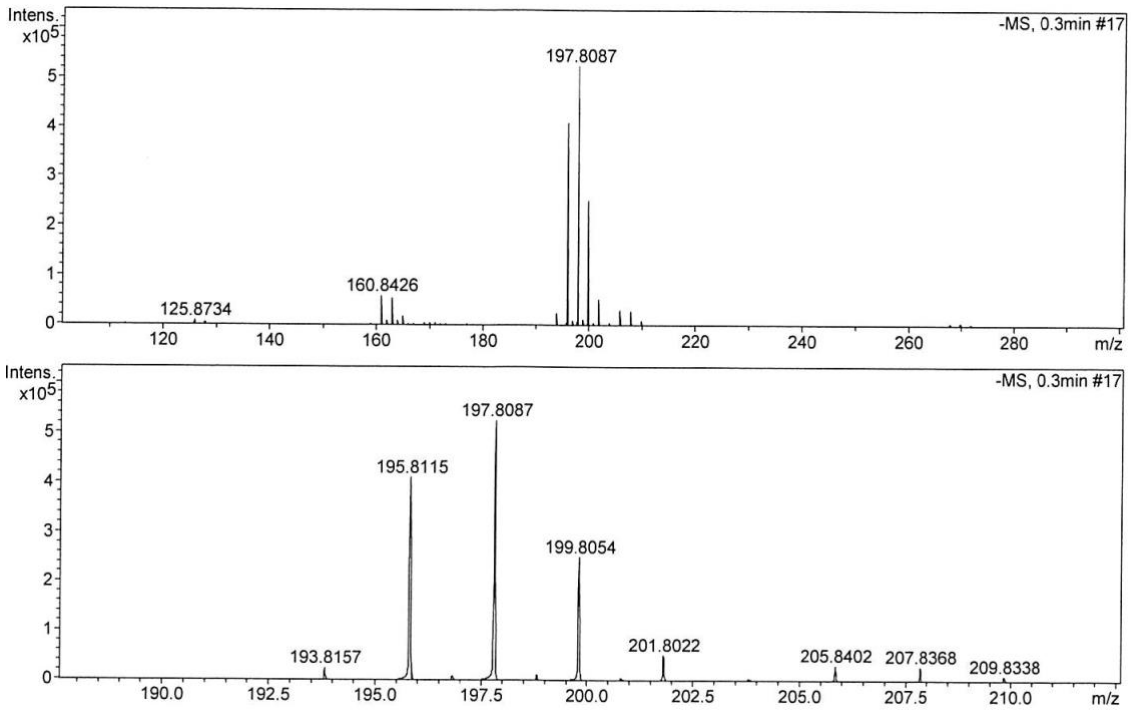
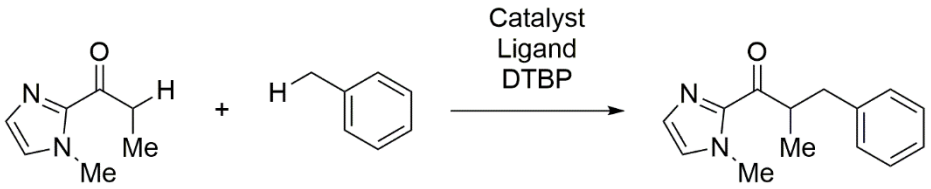


Figure S11. ESI-mass Spectrum of $FeCl_3$/**1a** (Negative Mode)

8. Full Data of Optimization Study

Table S4. Optimization of Reaction Conditions

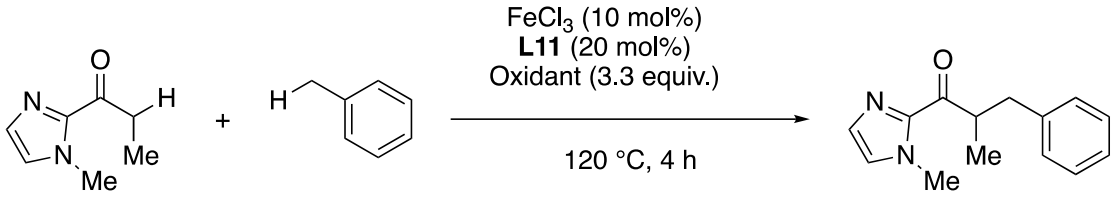


1a **2a** 47 equiv. (0.20 M) **3aa**

Entry	Catalyst (mol%)	Ligand (mol%)	DTBP (equiv.)	temp. (°C)	time (h)	yield (%)[a]
1	$FeCl_3$ (10)	**L7** (20)	3.3	120	4	74
2	$FeCl_2$ (10)	**L7** (20)	3.3	120	4	70
3	$FeBr_3$ (10)	**L7** (20)	3.3	120	4	54
4	$Fe(OAc)_2$ (10)	**L7** (20)	3.3	120	4	8
5	$Fe(acac)_3$ (10)	**L7** (20)	3.3	120	4	10
6	$Fe(acac)_2$ (10)	**L7** (20)	3.3	120	4	8
7	$Fe(OTf)_3$ (10)	**L7** (20)	3.3	120	4	40
8	$Fe(OTf)_2$ (10)	**L7** (20)	3.3	120	4	32
9	$CoCl_2$ (10)	**L7** (20)	3.3	120	4	4
10	$NiCl_2$ (10)	**L7** (20)	3.3	120	4	11
11	$CuCl_2$ (10)	**L7** (20)	3.3	120	4	N.D.
12	CuCl (10)	**L7** (20)	3.3	120	4	N.D.
13	$ZnCl_2$ (10)	**L7** (20)	3.3	120	4	3
14	$AlCl_3$ (10)	**L7** (20)	3.3	120	4	30
15	$FeCl_3$ (10)	**L7** (10)	1.7	80	8	79
16	$FeCl_3$ (5)	**L7** (10)	1.7	80	26	78
17	$FeCl_3$ (20)	**L7** (10)	1.7	80	8	66
18[b]	$FeCl_3$ (10)	**L7** (10)	1.7	80	8	82
19[c]	$FeCl_3$ (10)	**L7** (10)	1.7	80	8	55

[a] Yields were determined by ^{1}H-NMR analysis using 1,2,4,5-tetramethylbenzene as an internal standard. [b] Reaction concentration was 0.10 M. [c] Reaction concentration was 0.50 M.

Table S5. Evaluation of Oxidant



1a **2a** 47 equiv. 0.20 M **3aa**

Entry	Oxidant	Yield (%)
1	DTBP (Di-*tert*-butyl peroxide)	74
2	TBPB (tert-Butyl peroxybenzoate)	25
3	DCP (Dicumyl peroxide)	42
4	AIBN (2,2'-Azobis(isobutyronitrile))	0
5	TEMPO (2,2,6,6-Tetramethylpiperidine 1-oxyl free radical)	0
6	BQ (1,4-Benzoquinone)	0
7	DDQ (2,3-Dichloro-5,6-dicyano-1,4-benzoquinone)	0
8	O_2 (1 atm, balloon)	0

9. References

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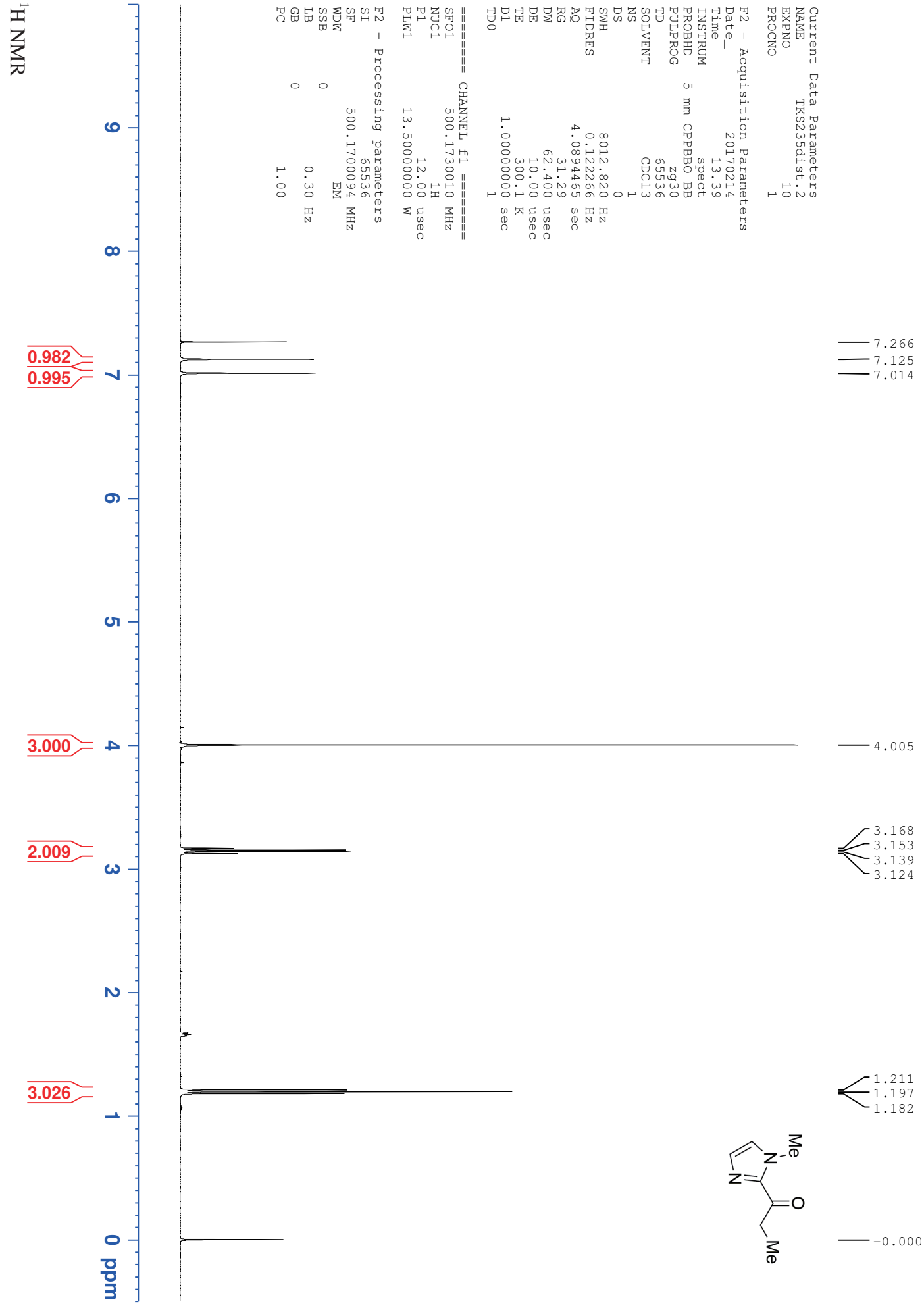
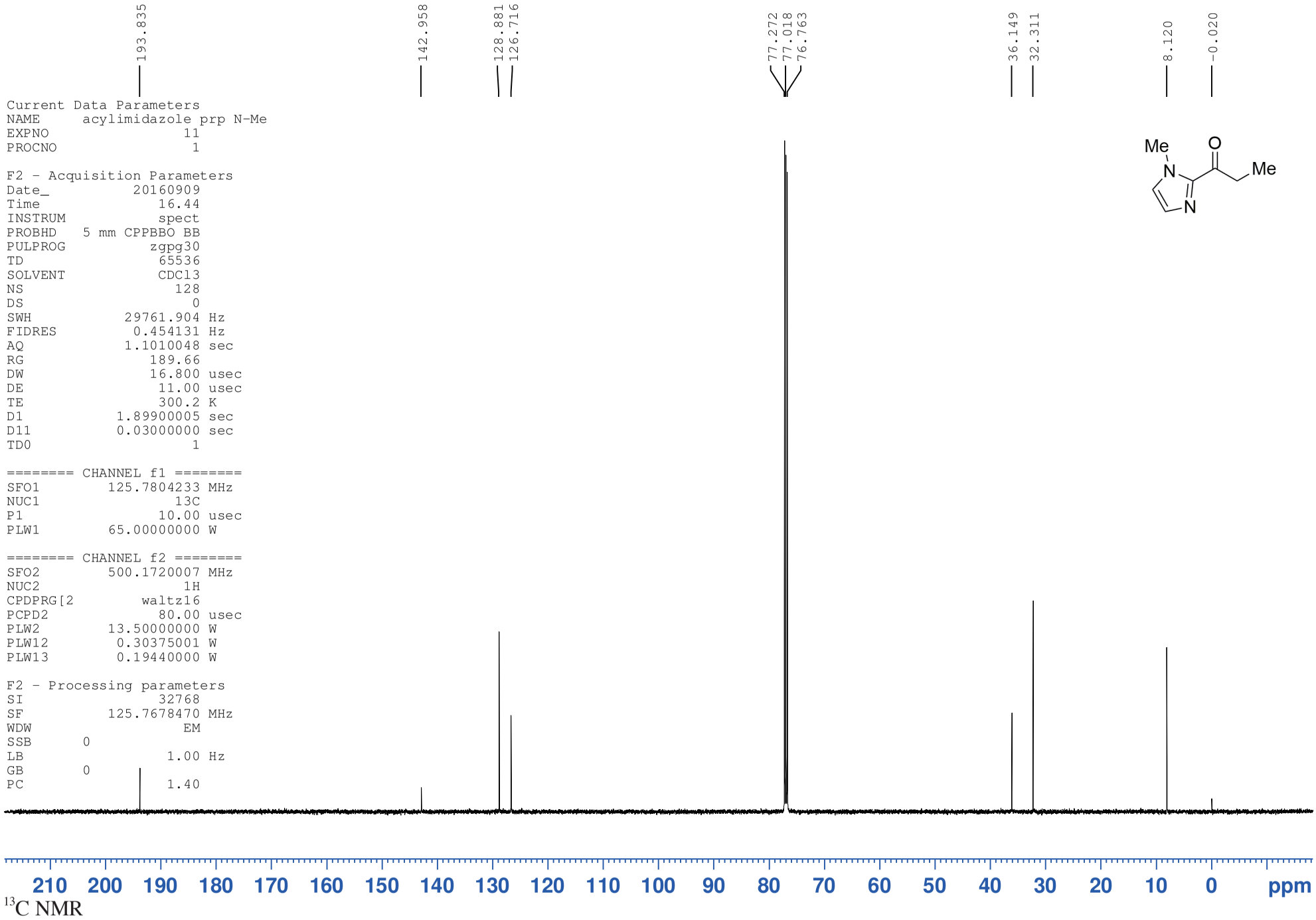
10. NMR Spectra of New Compounds

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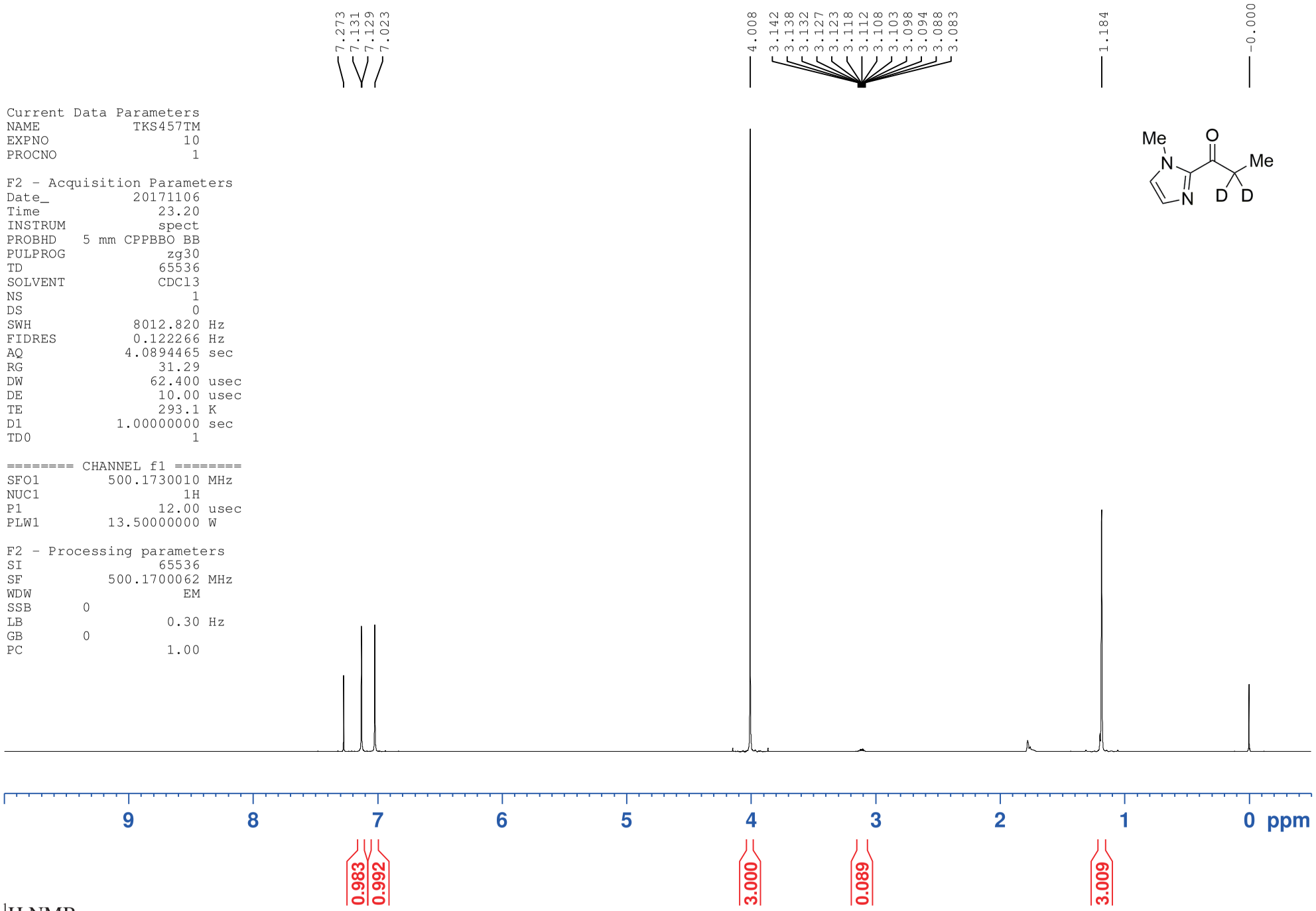
^{13}C NMR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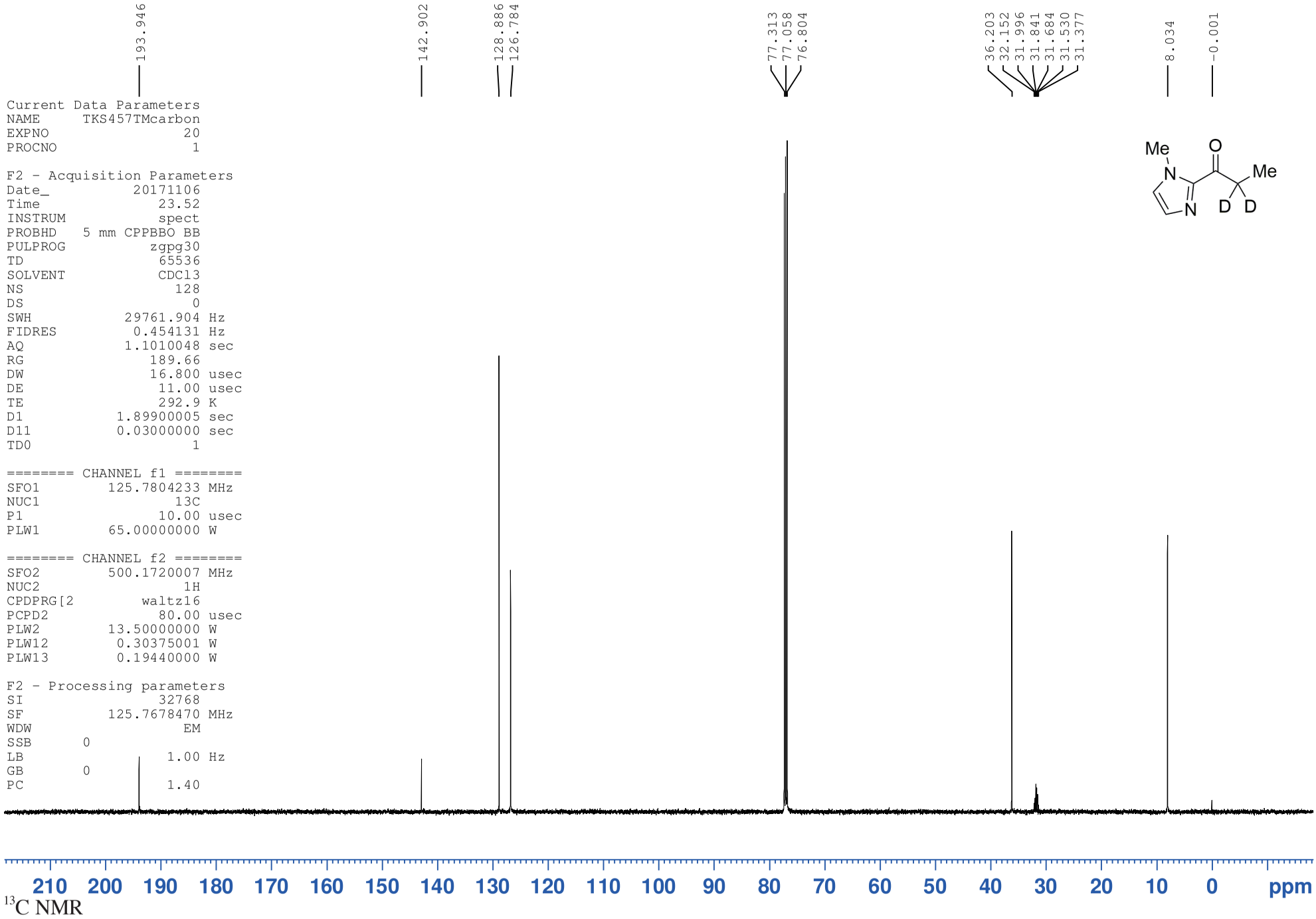
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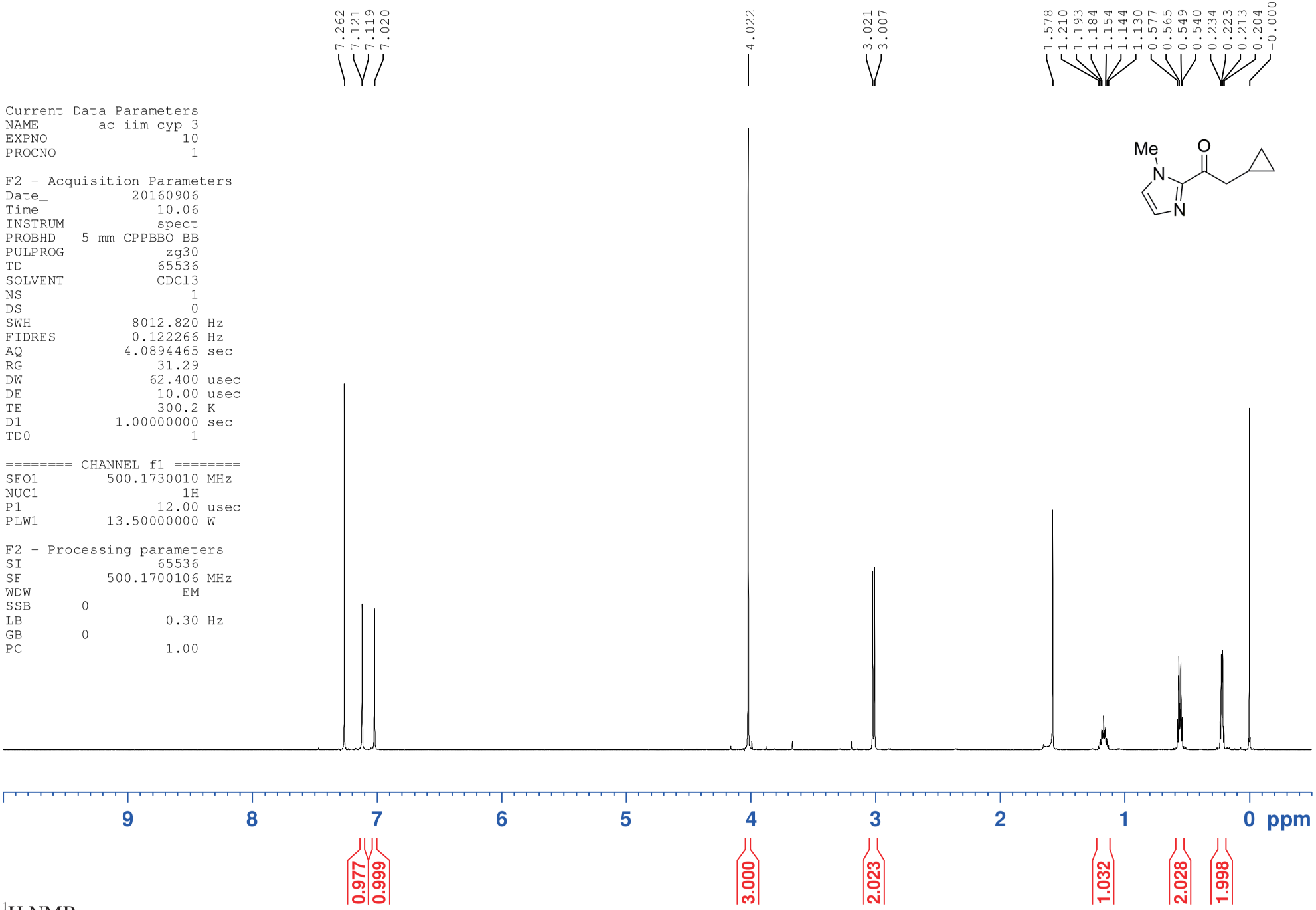
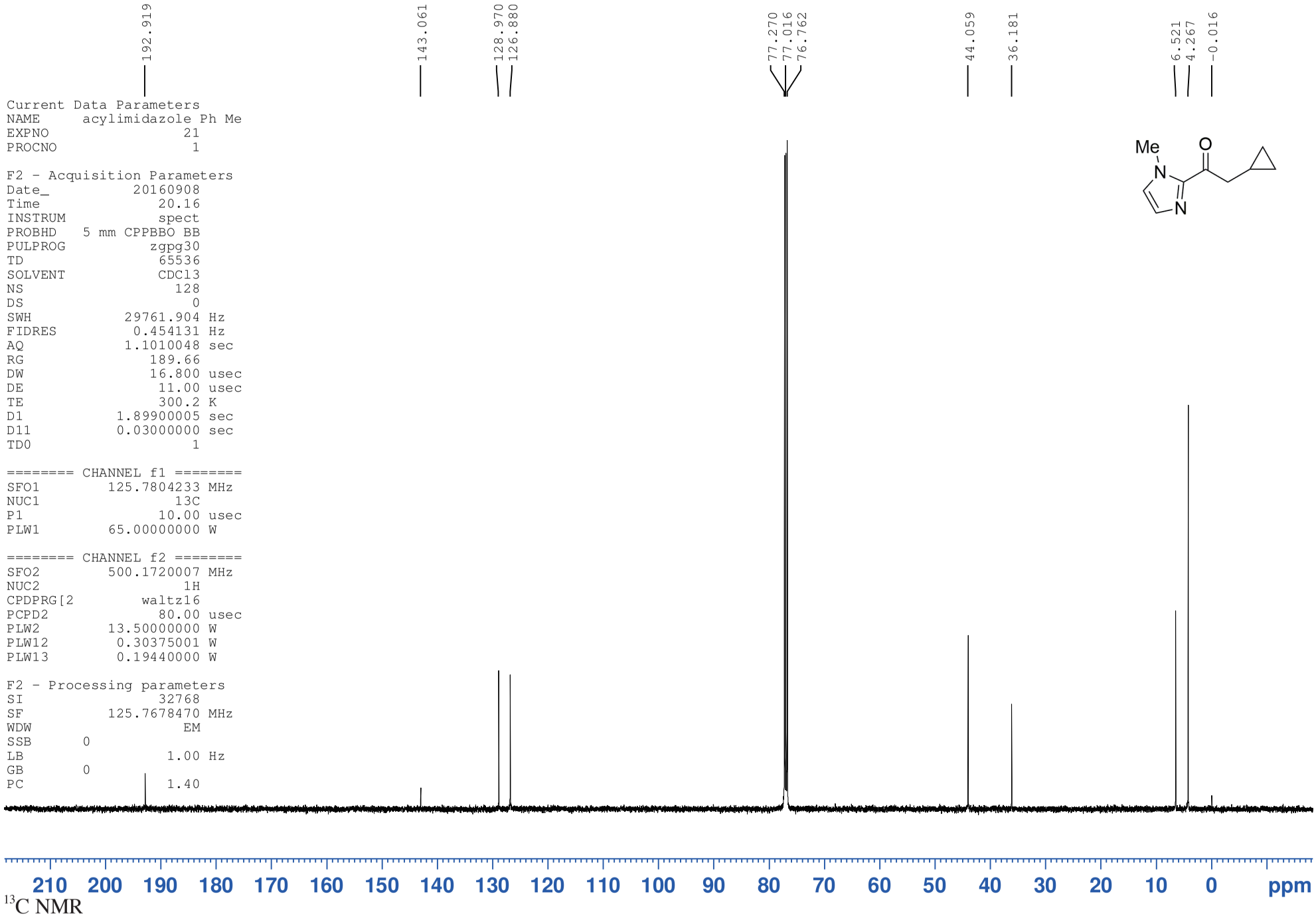
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

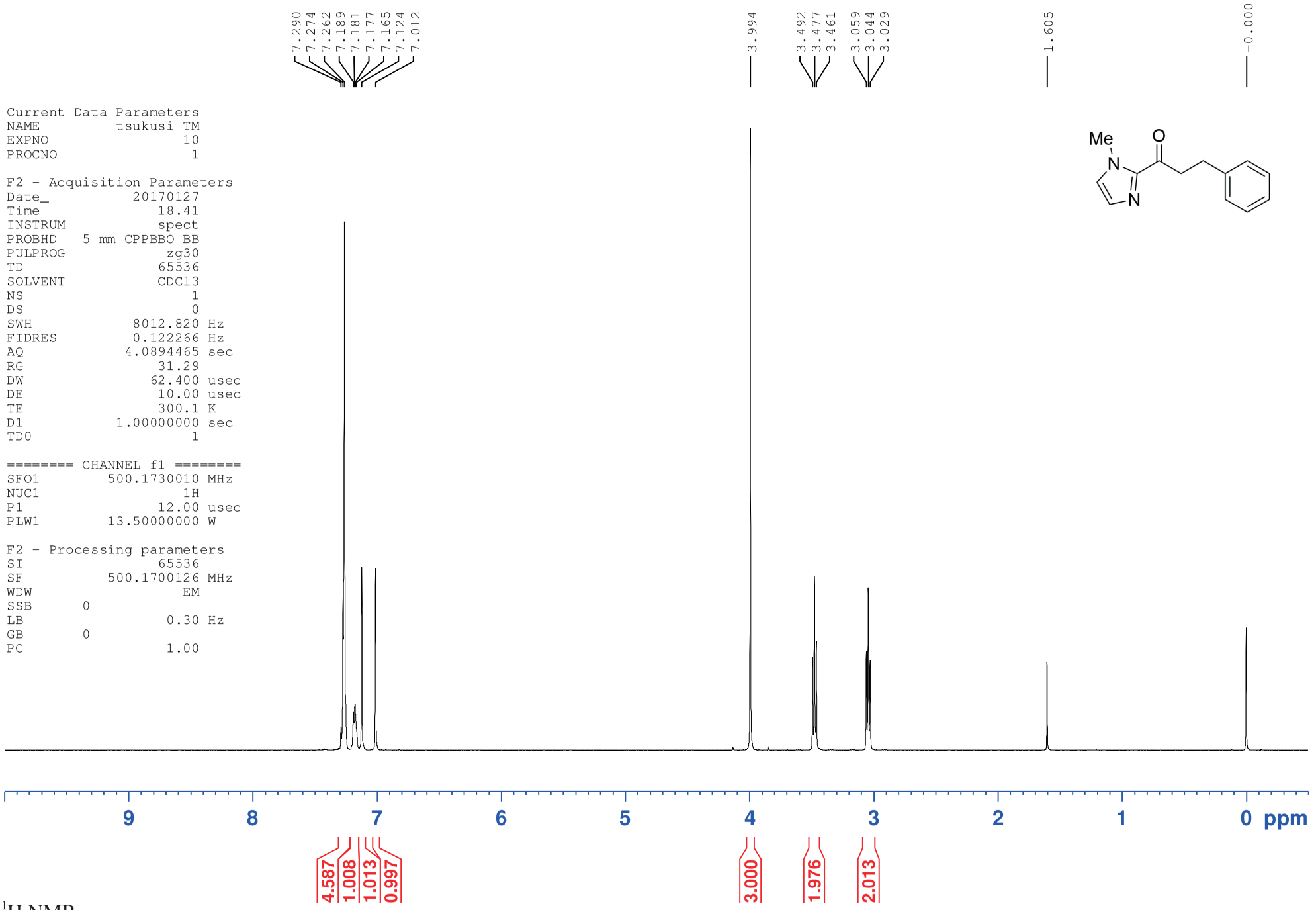
^{13}C NMR


7.262
7.121
7.119
7.020
4.022
3.021
3.007
1.578
1.210
1.193
1.184
1.154
1.144
1.130
0.577
0.565
0.549
0.540
0.234
0.223
0.213
0.204
-0.000
Me
N
O
N
Current Data Parameters
NAME ac iim cyp 3
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160906
Time 10.06
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
0.977
0.999
3.000
2.023
1.032
2.028
1.998

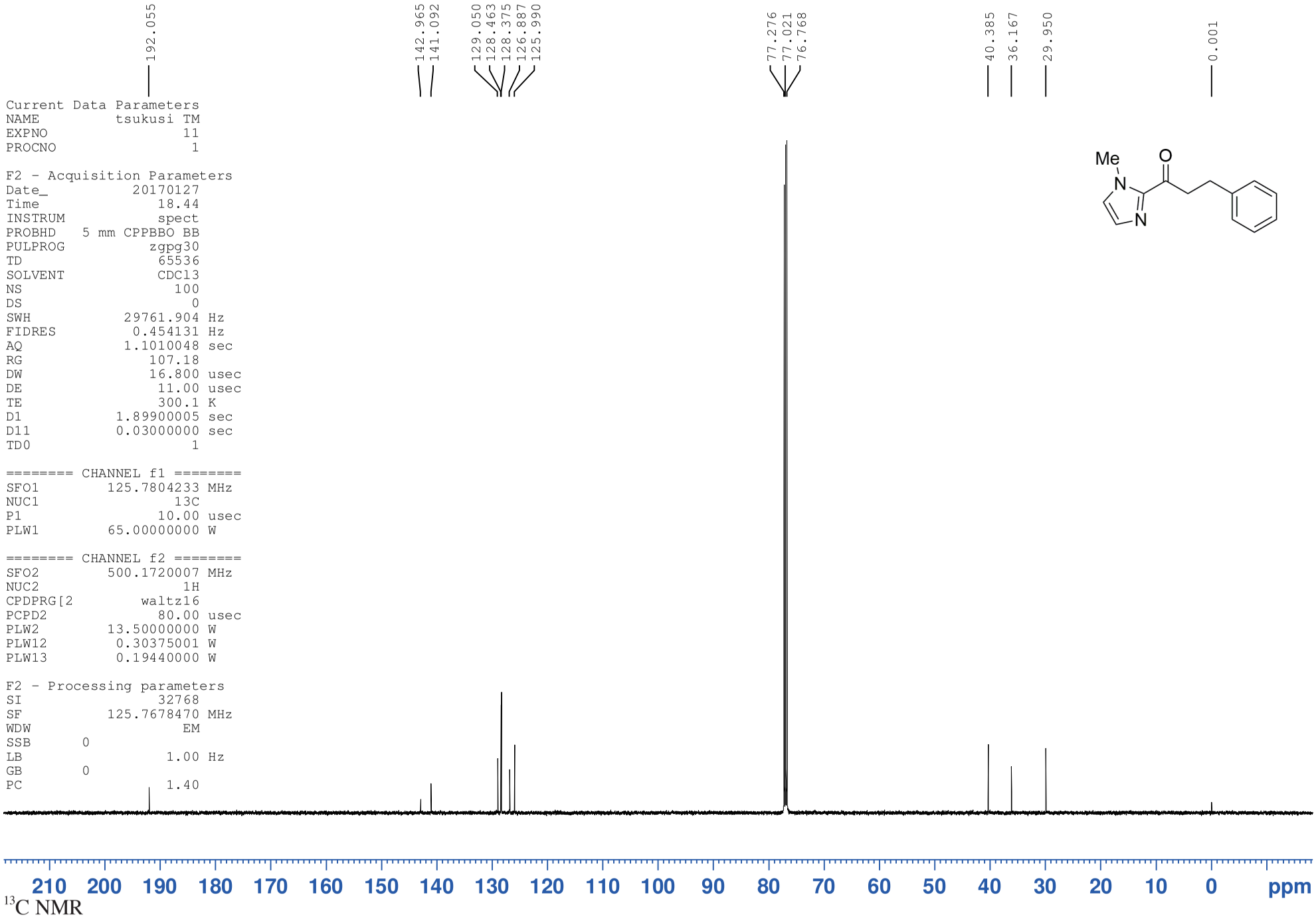
^{1}H NMR


192.919
143.061
128.970
126.880
77.270
77.016
76.762
44.059
36.181
6.521
4.267
-0.016
Me
N
O
N
Current Data Parameters
NAME acylimidazole Ph Me
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160908
Time 20.16
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

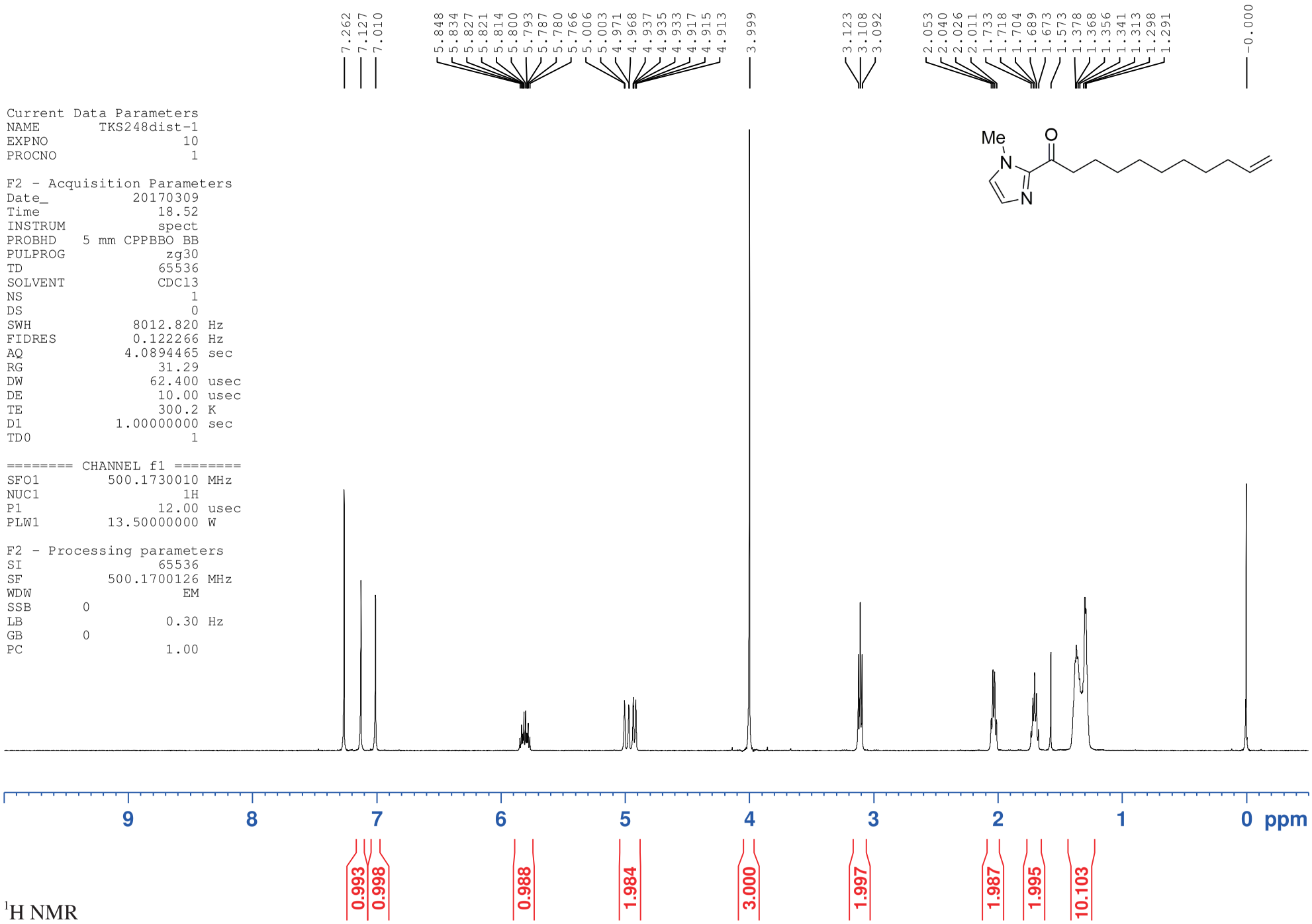
^{13}C NMR


7.290
7.274
7.262
7.189
7.181
7.177
7.165
7.124
7.012
3.994
3.492
3.477
3.461
3.059
3.044
3.029
1.605
-0.000
Current Data Parameters
NAME tsukusi TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170127
Time 18.41
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
9
8
7
6
5
4
3
2
1
0 ppm
4.587
1.008
1.013
0.997
3.000
1.976
2.013

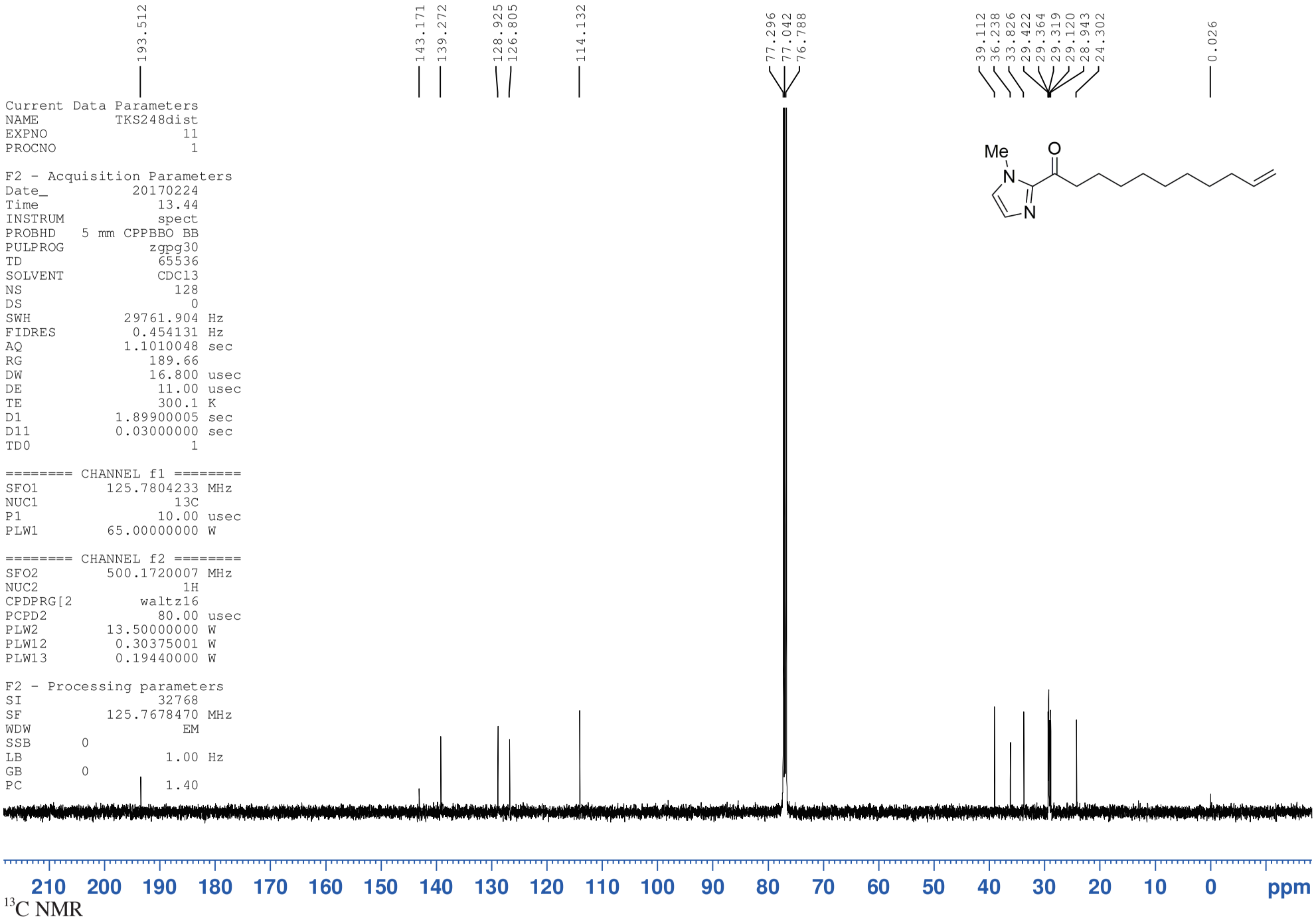
^{1}H NMR


192.055
142.965
141.092
129.050
128.463
128.375
126.887
125.990
77.276
77.021
76.768
40.385
36.167
29.950
0.001
Current Data Parameters
NAME tsukusi TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170127
Time 18.44
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

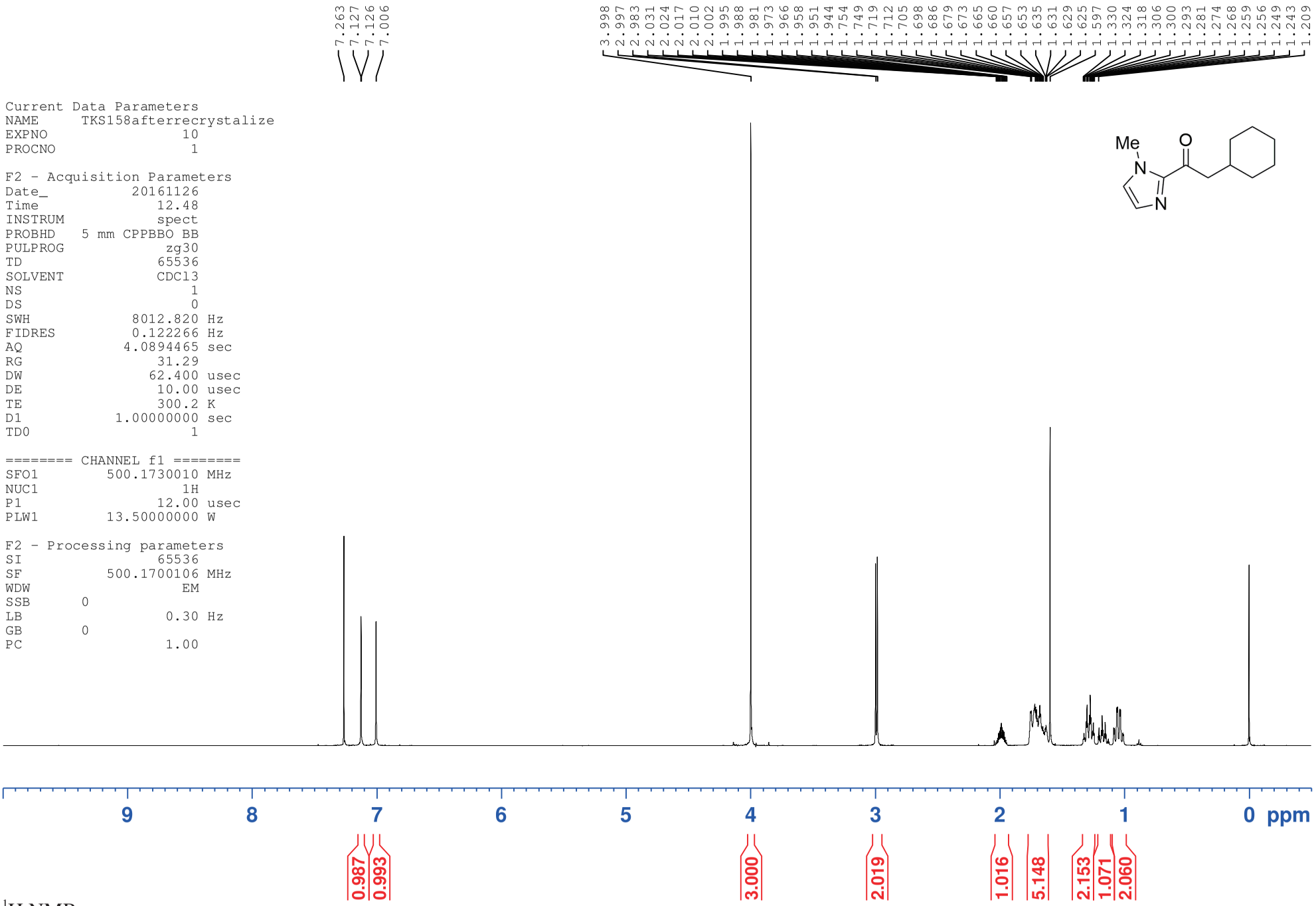
^{13}C NMR


7.262
7.127
7.010
5.848
5.834
5.827
5.821
5.814
5.800
5.793
5.787
5.780
5.766
5.006
5.003
4.971
4.968
4.937
4.935
4.933
4.917
4.915
4.913
3.999
3.123
3.108
3.092
2.053
2.040
2.026
2.011
1.733
1.718
1.704
1.689
1.673
1.573
1.378
1.368
1.356
1.341
1.313
1.298
1.291
-0.000
Current Data Parameters
NAME TKS248dist-1
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170309
Time 18.52
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
9
8
7
6
5
4
3
2
1
0 ppm
0.993
0.998
0.988
1.984
3.000
1.997
1.987
1.995
10.103

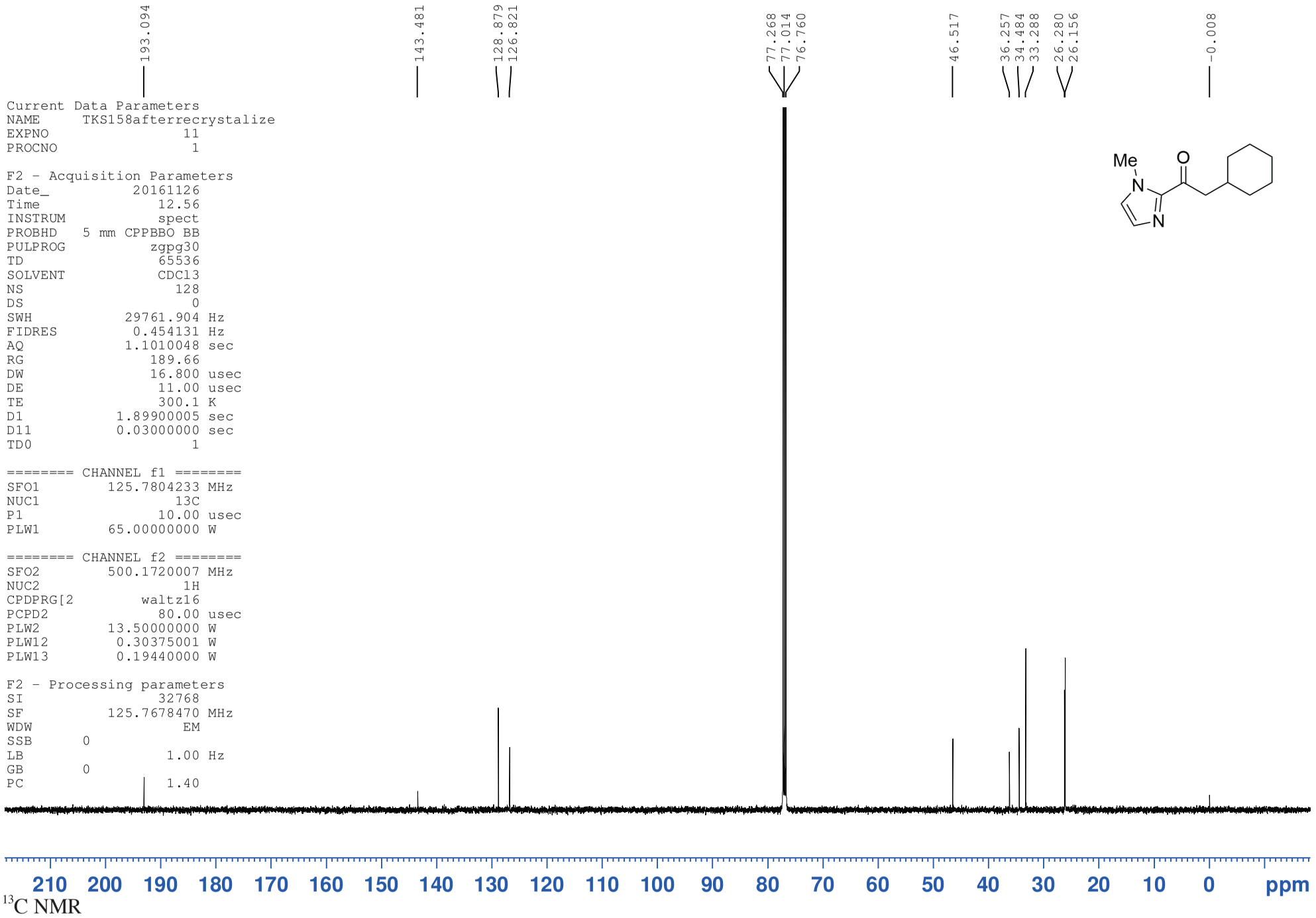
^{1}H NMR


193.512
143.171
139.272
128.925
126.805
114.132
77.296
77.042
76.788
39.112
36.238
33.826
29.422
29.364
29.319
29.120
28.943
24.302
0.026
Current Data Parameters
NAME TKS248dist
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170224
Time 13.44
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

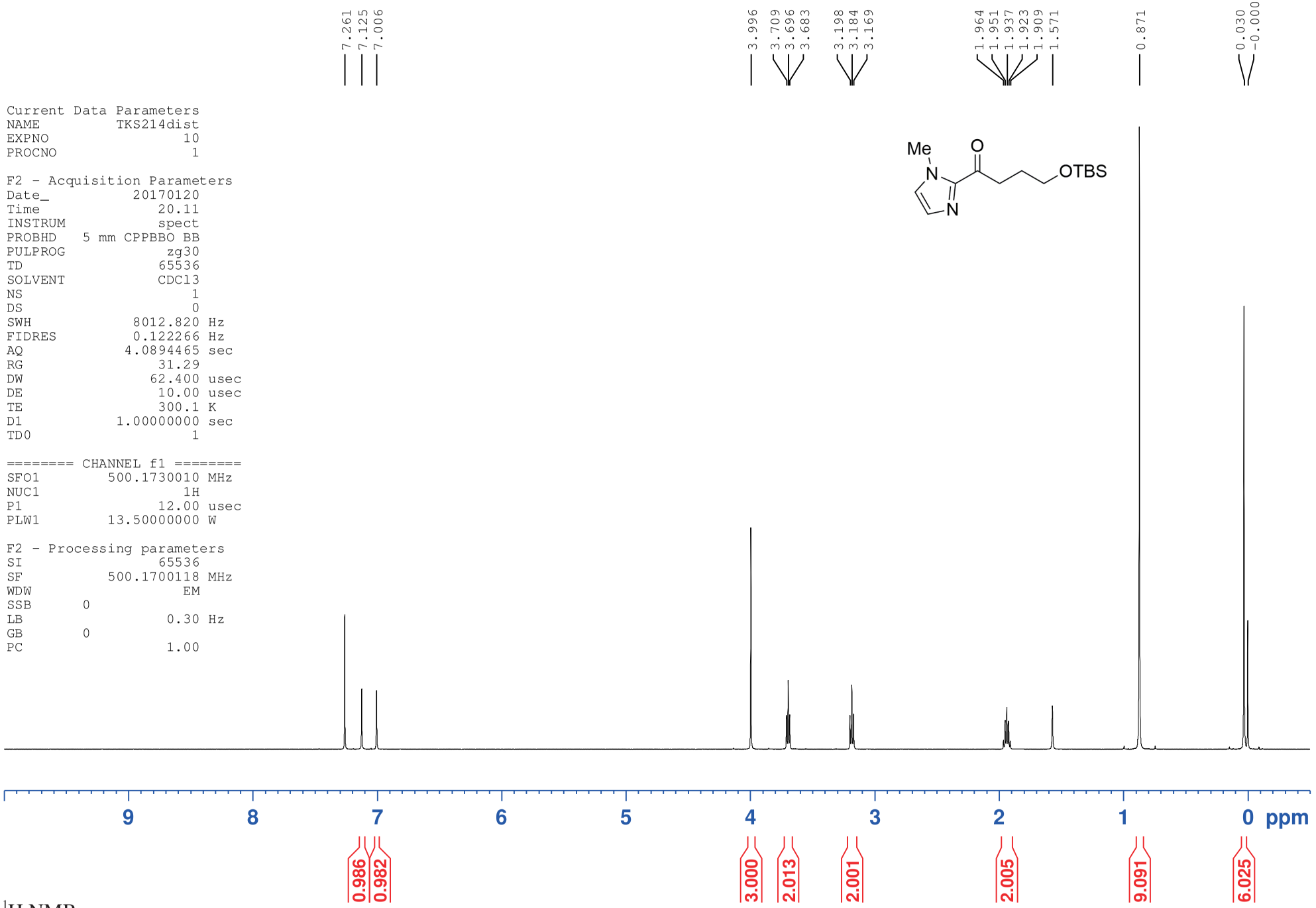
^{13}C NMR


7.263
7.127
7.126
7.006
3.998
2.997
2.983
2.031
2.024
2.017
2.010
2.002
1.995
1.988
1.981
1.973
1.966
1.958
1.951
1.944
1.754
1.749
1.719
1.712
1.705
1.698
1.686
1.679
1.673
1.665
1.660
1.657
1.653
1.635
1.631
1.629
1.625
1.597
1.330
1.324
1.318
1.306
1.300
1.293
1.281
1.274
1.268
1.259
1.256
1.249
1.243
1.209
Me
N
O
N
Current Data Parameters
NAME TKS158afterrecrystalize
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161126
Time 12.48
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
0.987
0.993
3.000
2.019
1.016
5.148
2.153
1.071
2.060

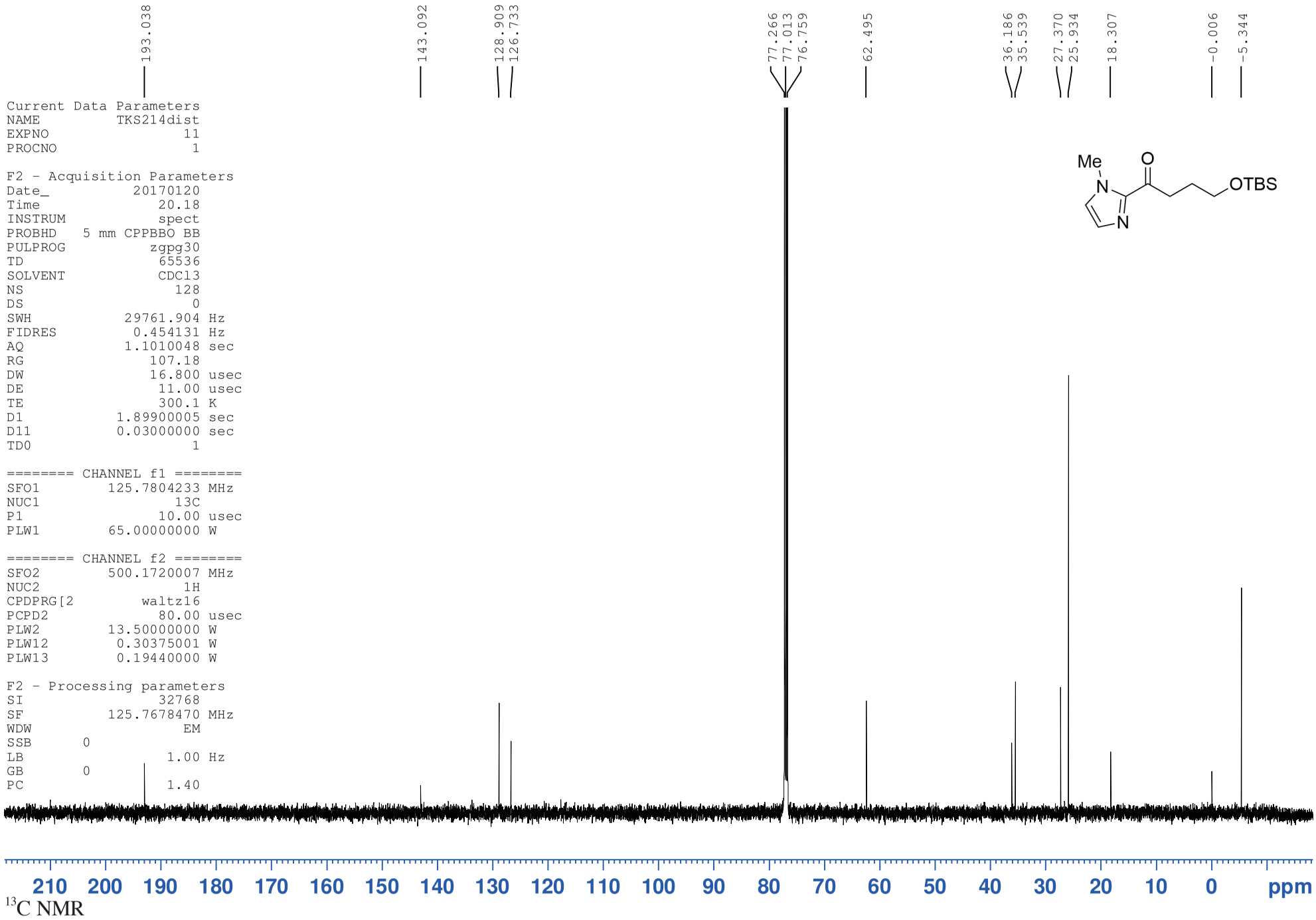
^{1}H NMR


193.094
143.481
128.879
126.821
77.268
77.014
76.760
46.517
36.257
34.484
33.288
26.280
26.156
-0.008
Current Data Parameters
NAME TKS158afterrecrystalize
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161126
Time 12.56
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

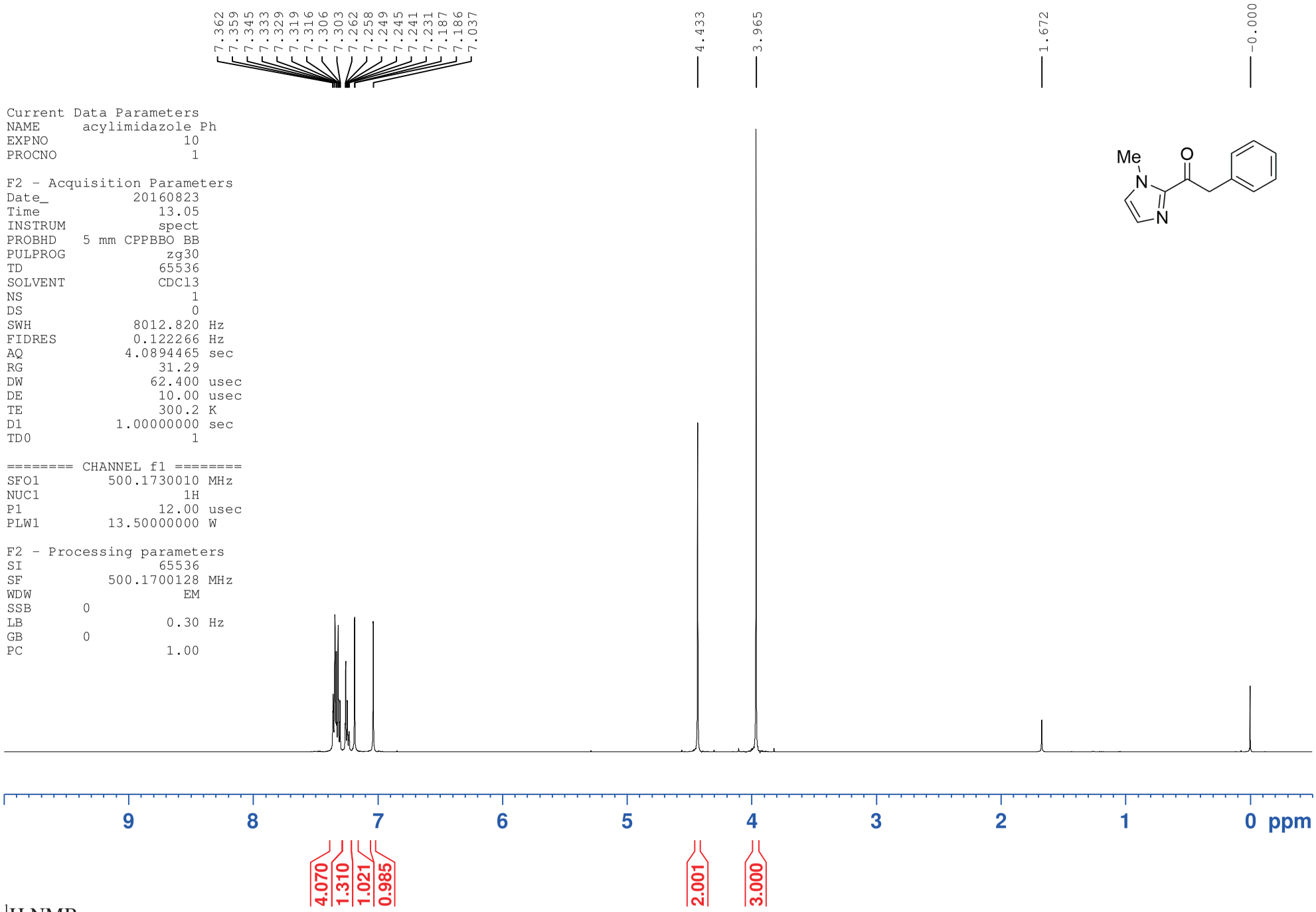
^{13}C NMR


7.261
7.125
7.006
3.996
3.709
3.696
3.683
3.198
3.184
3.169
1.964
1.951
1.937
1.923
1.909
1.571
0.871
0.030
-0.000
Me
N
N
O
OTBS
Current Data Parameters
NAME TKS214dist
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170120
Time 20.11
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
0.986
0.982
3.000
2.013
2.001
2.005
9.091
6.025

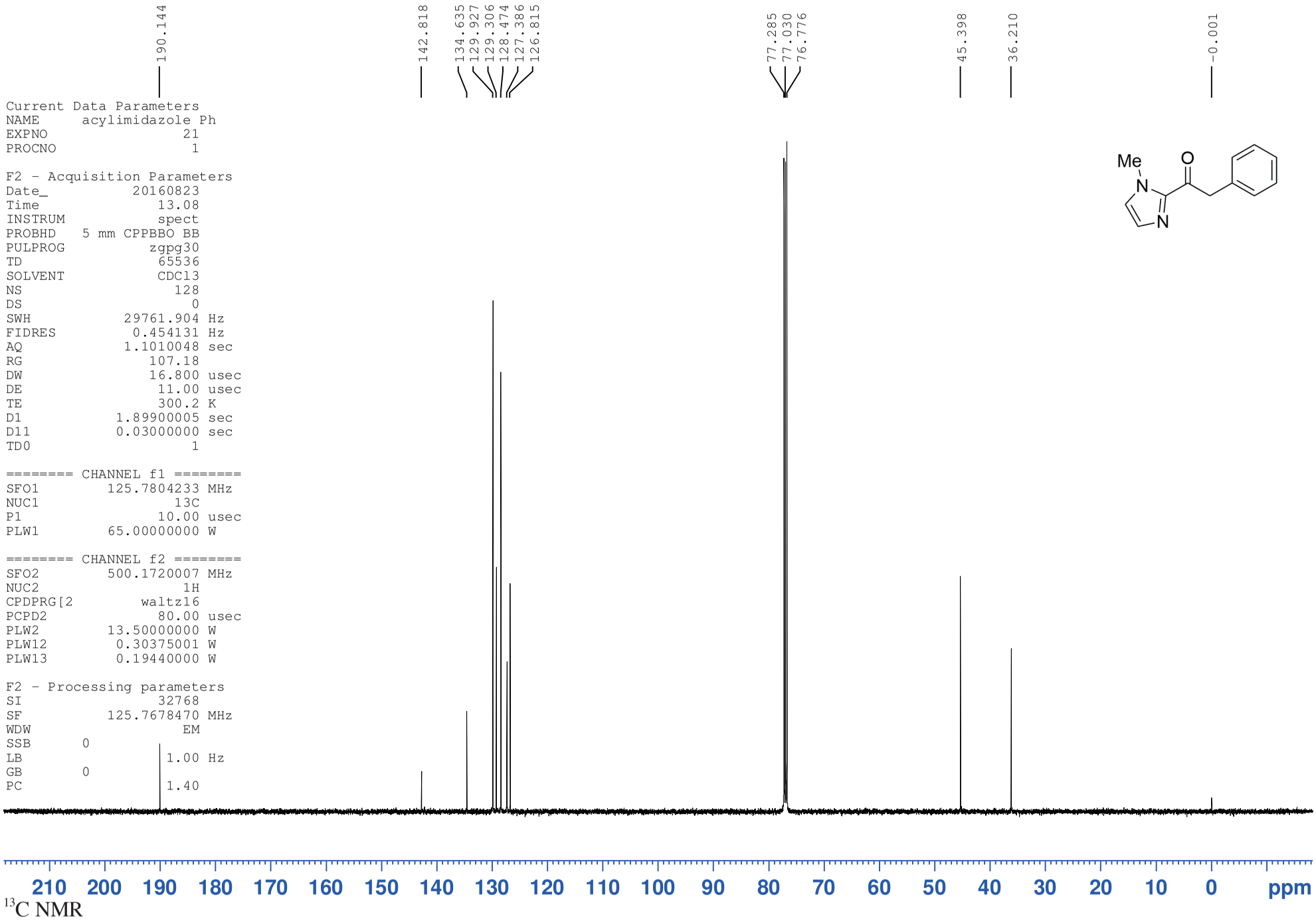
^{1}H NMR


193.038
143.092
128.909
126.733
77.266
77.013
76.759
62.495
36.186
35.539
27.370
25.934
18.307
-0.006
-5.344
Me
N
N
O
OTBS
Current Data Parameters
NAME TKS214dist
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170120
Time 20.18
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

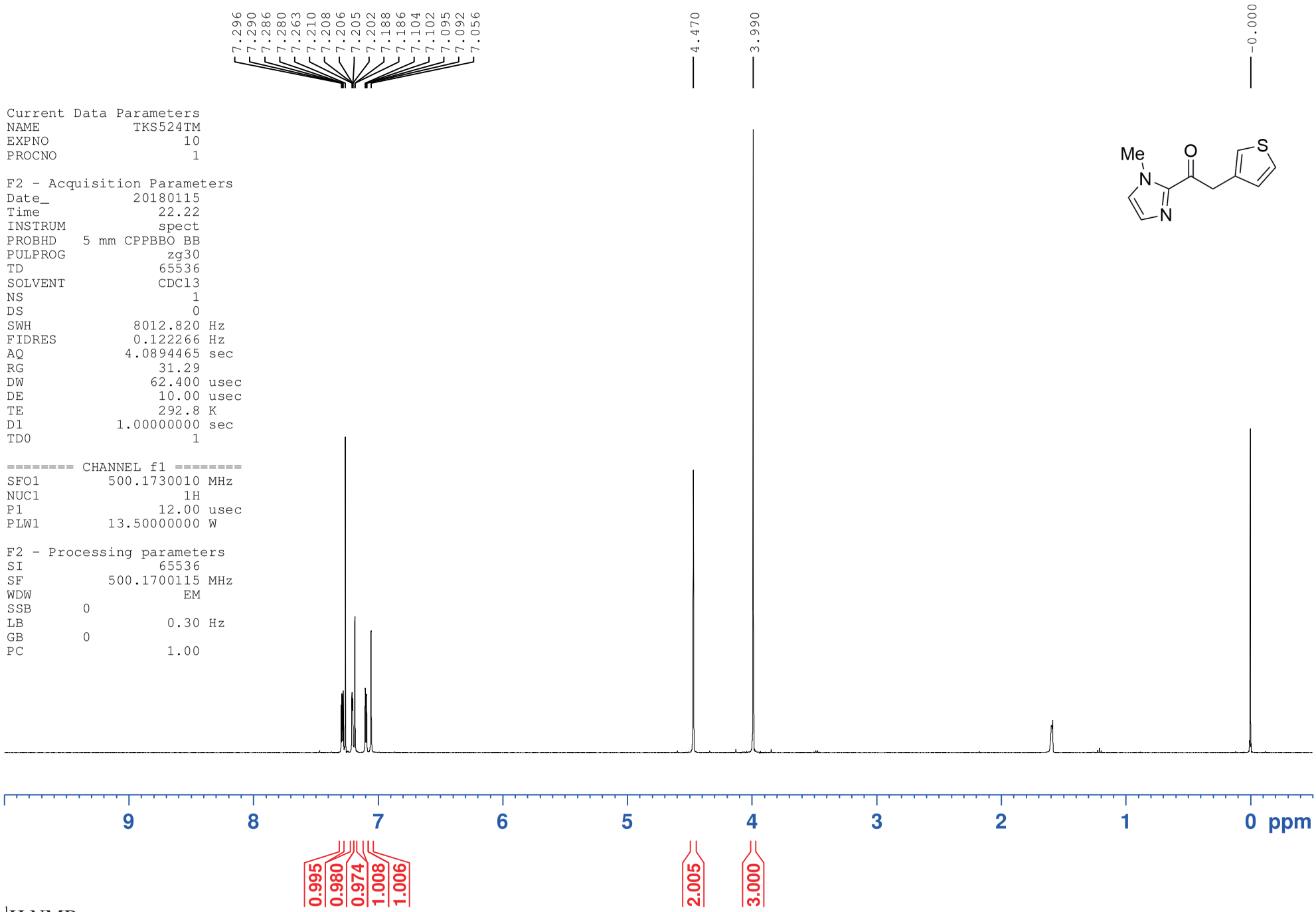
^{13}C NMR


7.362
7.359
7.345
7.333
7.329
7.319
7.316
7.306
7.303
7.262
7.258
7.249
7.245
7.241
7.231
7.187
7.186
7.037
4.433
3.965
1.672
-0.000
Current Data Parameters
NAME acylimidazole Ph
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160823
Time 13.05
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
9
8
7
6
5
4
3
2
1
0 ppm
4.070
1.310
1.021
0.985
2.001
3.000

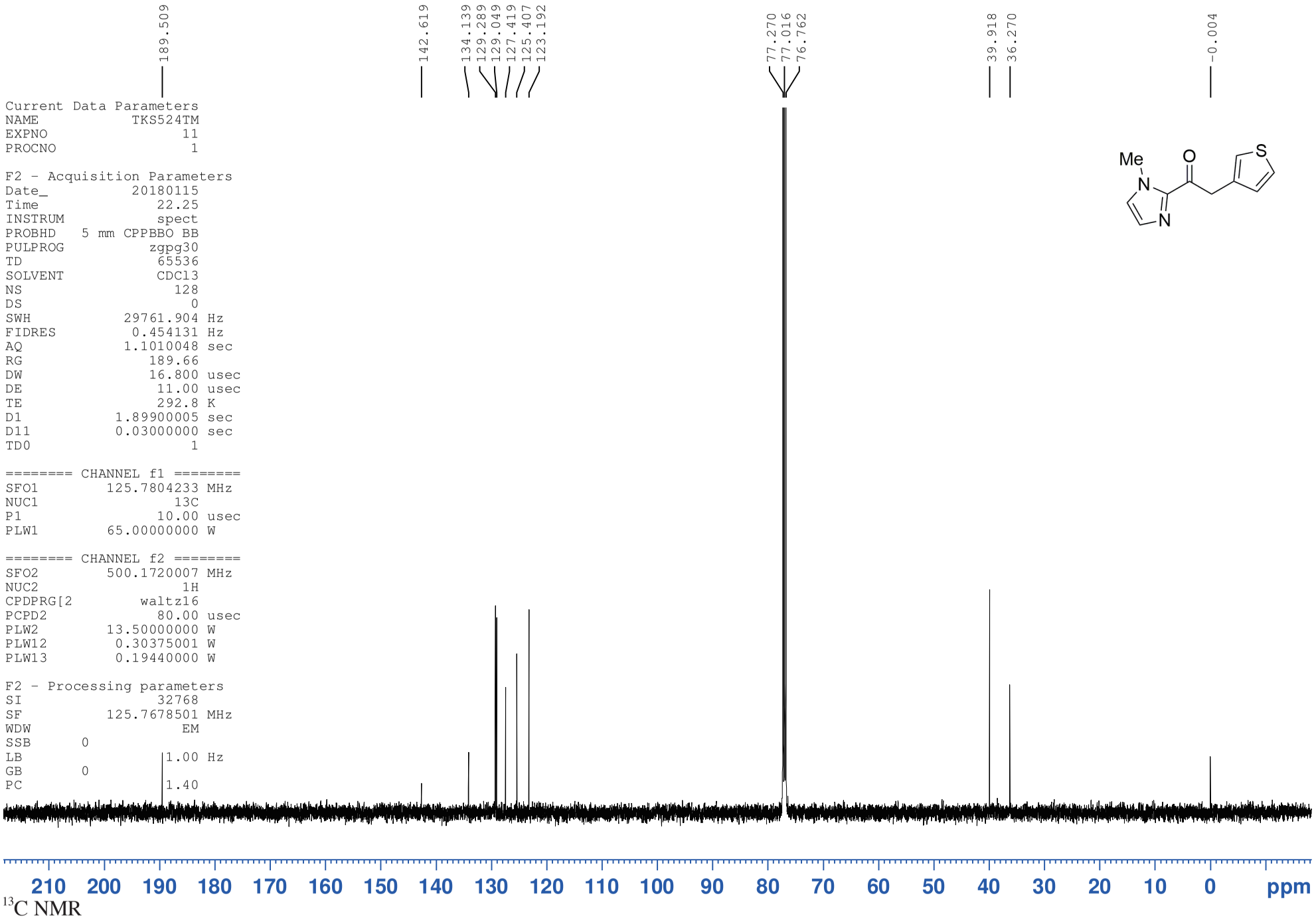
^{1}H NMR


190.144
142.818
134.635
129.927
129.306
128.474
127.386
126.815
77.285
77.030
76.776
45.398
36.210
-0.001
Current Data Parameters
NAME acylimidazole Ph
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160823
Time 13.08
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

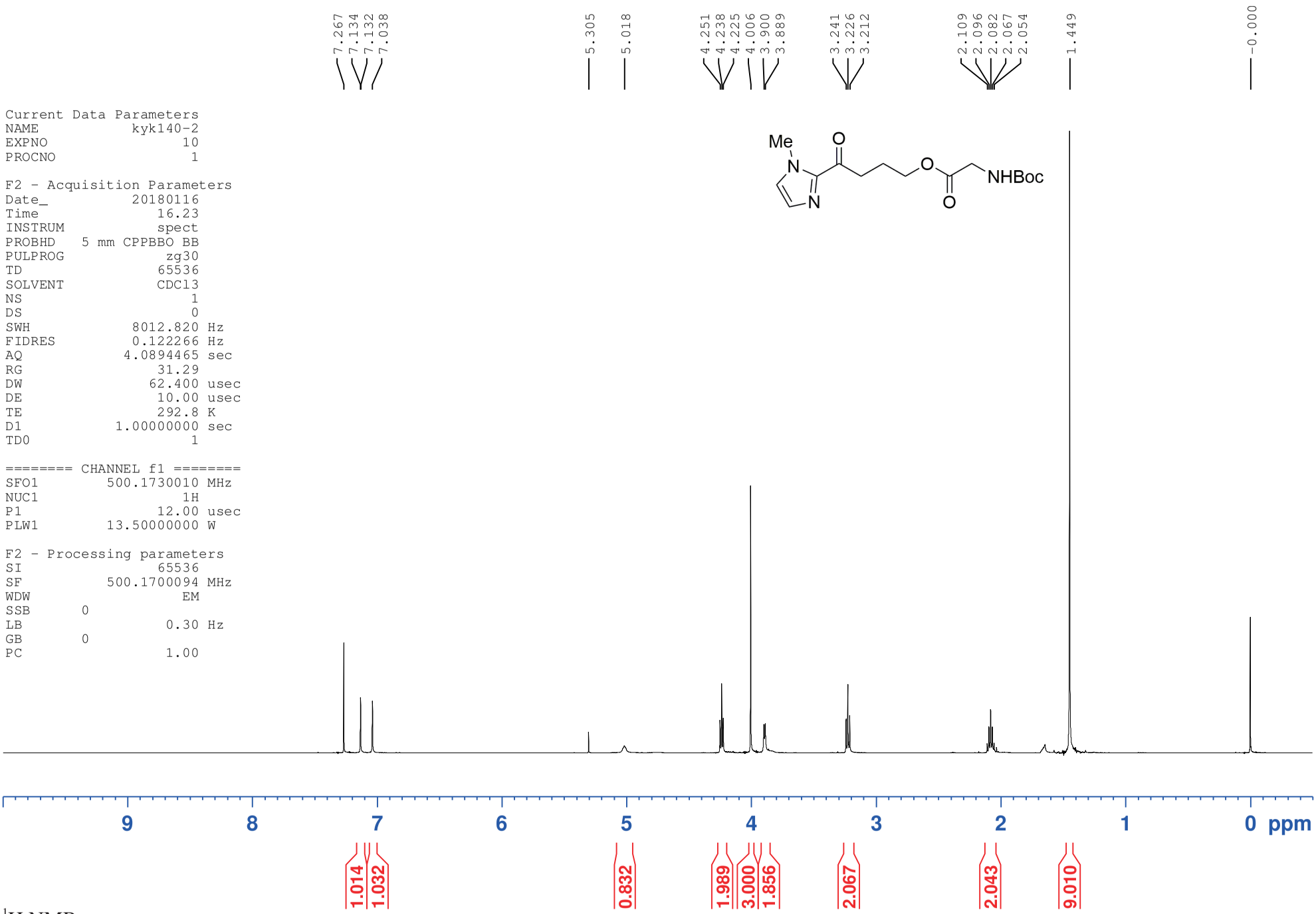
^{13}C NMR


7.296
7.290
7.286
7.280
7.263
7.210
7.208
7.206
7.205
7.202
7.188
7.186
7.104
7.102
7.095
7.092
7.056
4.470
3.990
-0.000
Current Data Parameters
NAME TKS524TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180115
Time 22.22
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
S
9
8
7
6
5
4
3
2
1
0 ppm
0.995
0.980
0.974
1.008
1.006
2.005
3.000

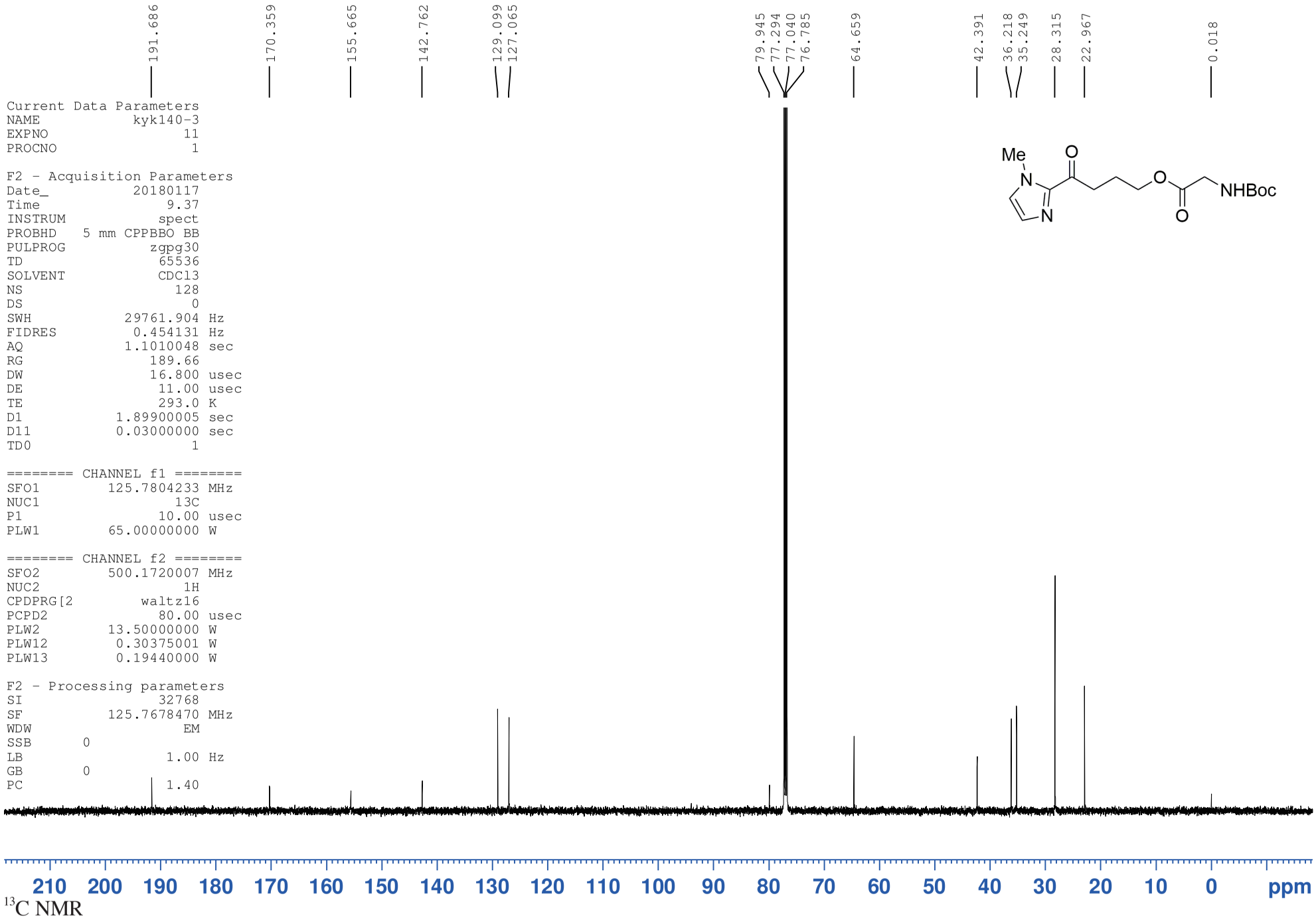
^{1}H NMR


189.509
142.619
134.139
129.289
129.049
127.419
125.407
123.192
77.270
77.016
76.762
39.918
36.270
-0.004
Current Data Parameters
NAME TKS524TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180115
Time 22.25
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.8 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678501 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
S
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

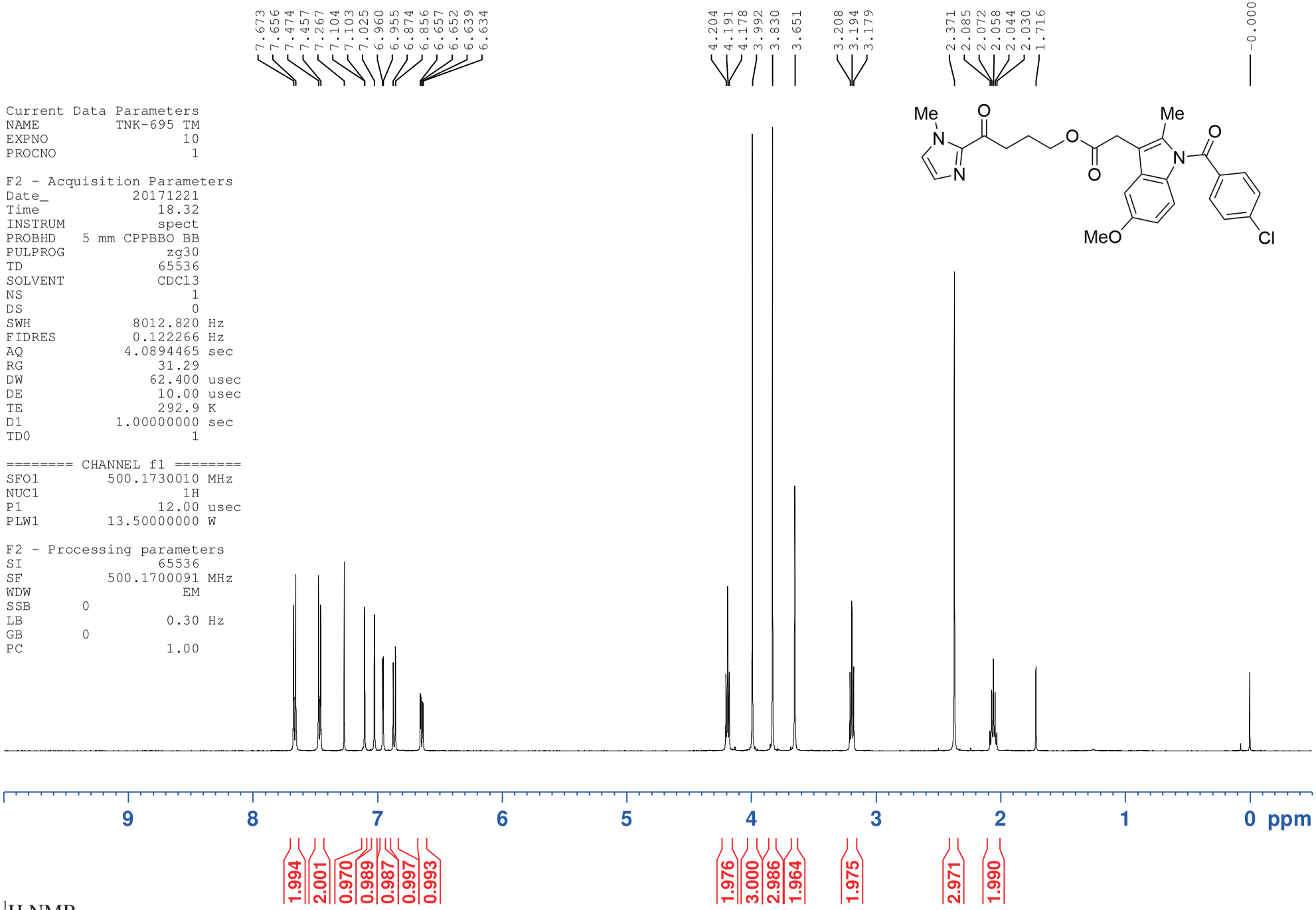
^{13}C NMR


7.267
7.134
7.132
7.038
5.305
5.018
4.251
4.238
4.225
4.006
3.900
3.889
3.241
3.226
3.212
2.109
2.096
2.082
2.067
2.054
1.449
-0.000
Current Data Parameters
NAME kyk140-2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180116
Time 16.23
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
O
O
NHBoc
9
8
7
6
5
4
3
2
1
0 ppm
1.014
1.032
0.832
1.989
3.000
1.856
2.067
2.043
9.010

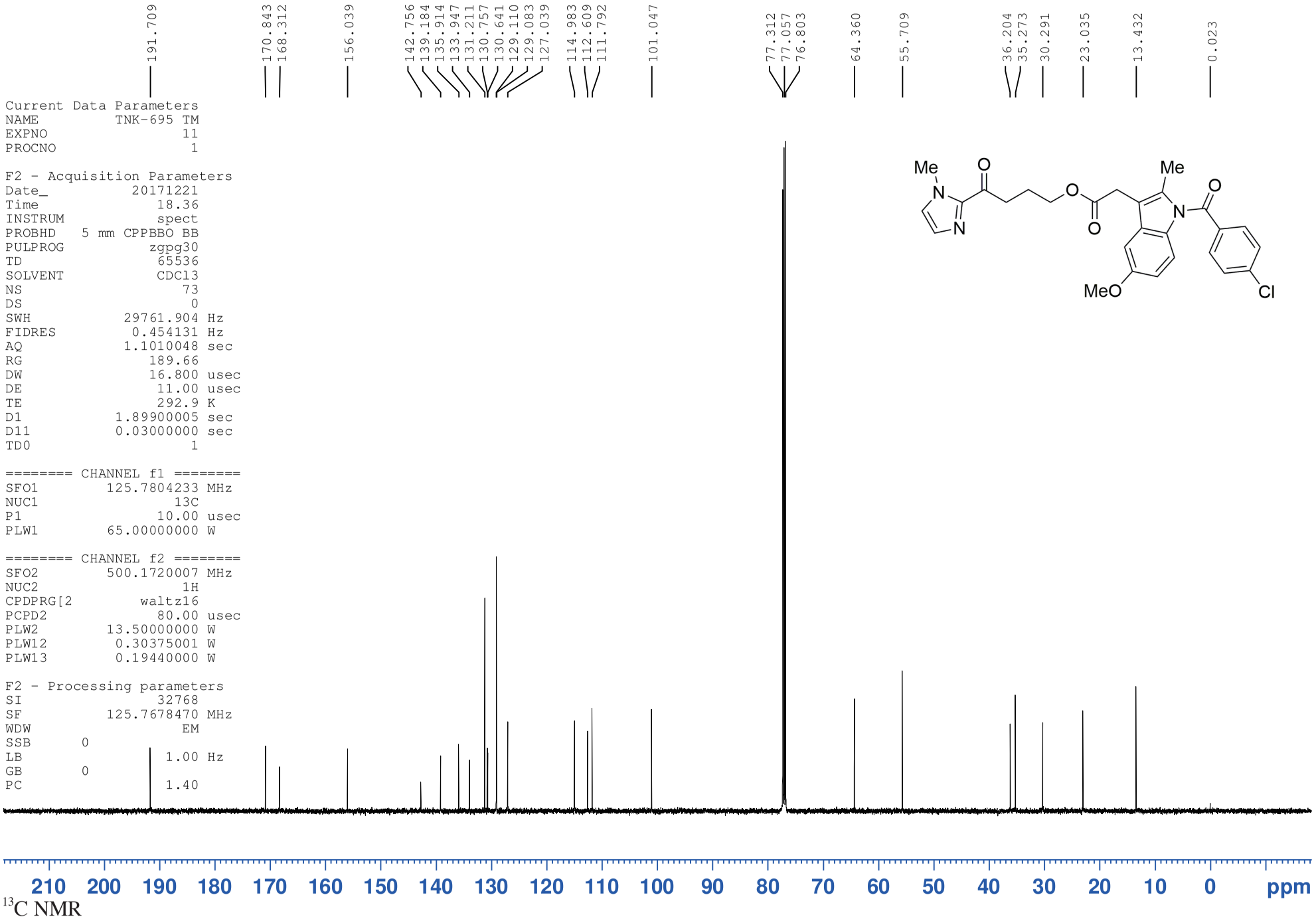
^{1}H NMR


191.686
170.359
155.665
142.762
129.099
127.065
79.945
77.294
77.040
76.785
64.659
42.391
36.218
35.249
28.315
22.967
0.018
Current Data Parameters
NAME kyk140-3
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180117
Time 9.37
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 293.0 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
O
O
NHBoc
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

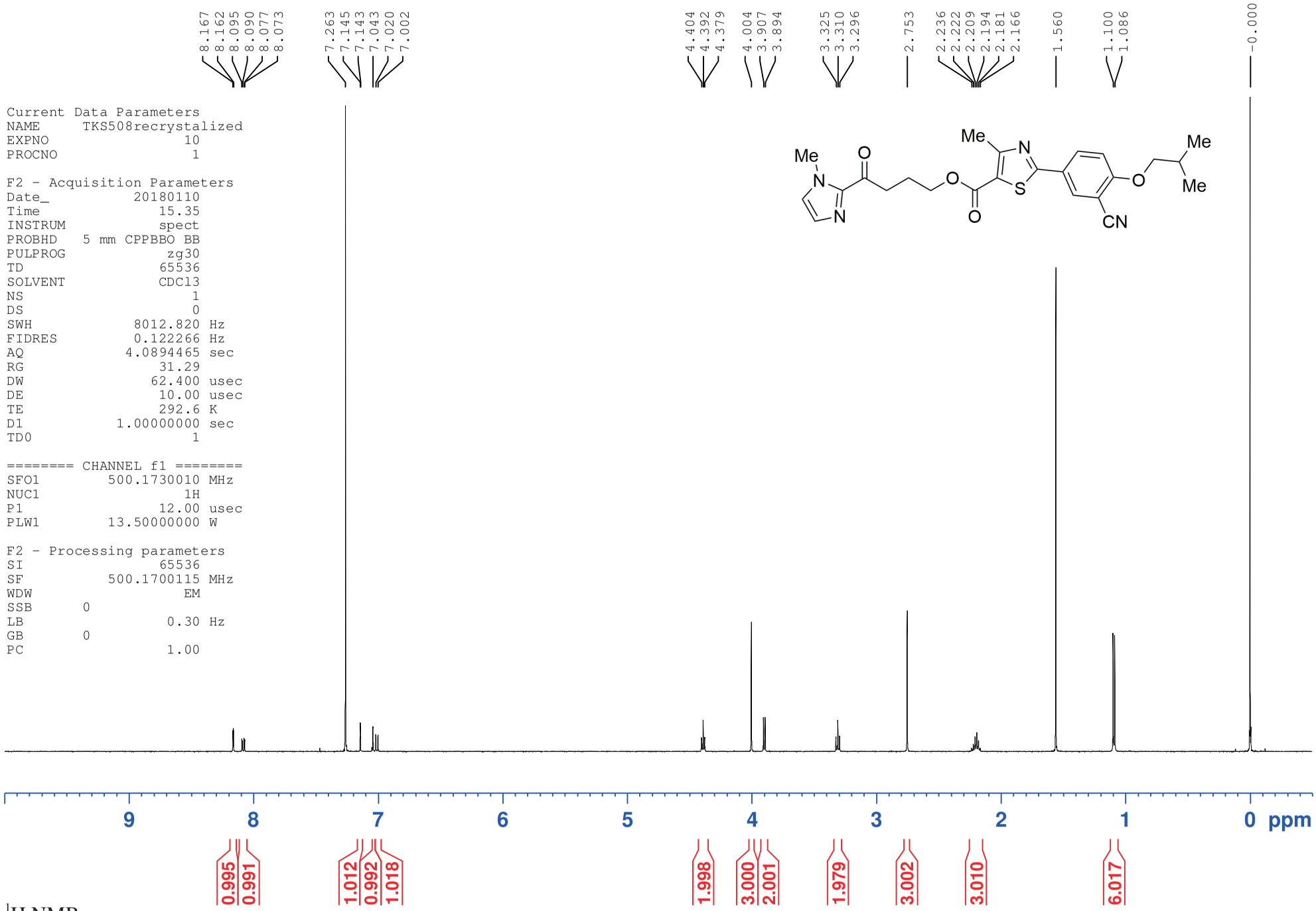
^{13}C NMR


7.673
7.656
7.474
7.457
7.267
7.104
7.103
7.025
6.960
6.955
6.874
6.856
6.657
6.652
6.639
6.634
4.204
4.191
4.178
3.992
3.830
3.651
3.208
3.194
3.179
2.371
2.085
2.072
2.058
2.044
2.030
1.716
0.000
Me
N
N
O
O
O
O
Me
N
MeO
Cl
Current Data Parameters
NAME TNK-695 TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171221
Time 18.32
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
1.994
2.001
0.970
0.989
0.987
0.997
0.993
1.976
3.000
2.986
1.964
1.975
2.971
1.990

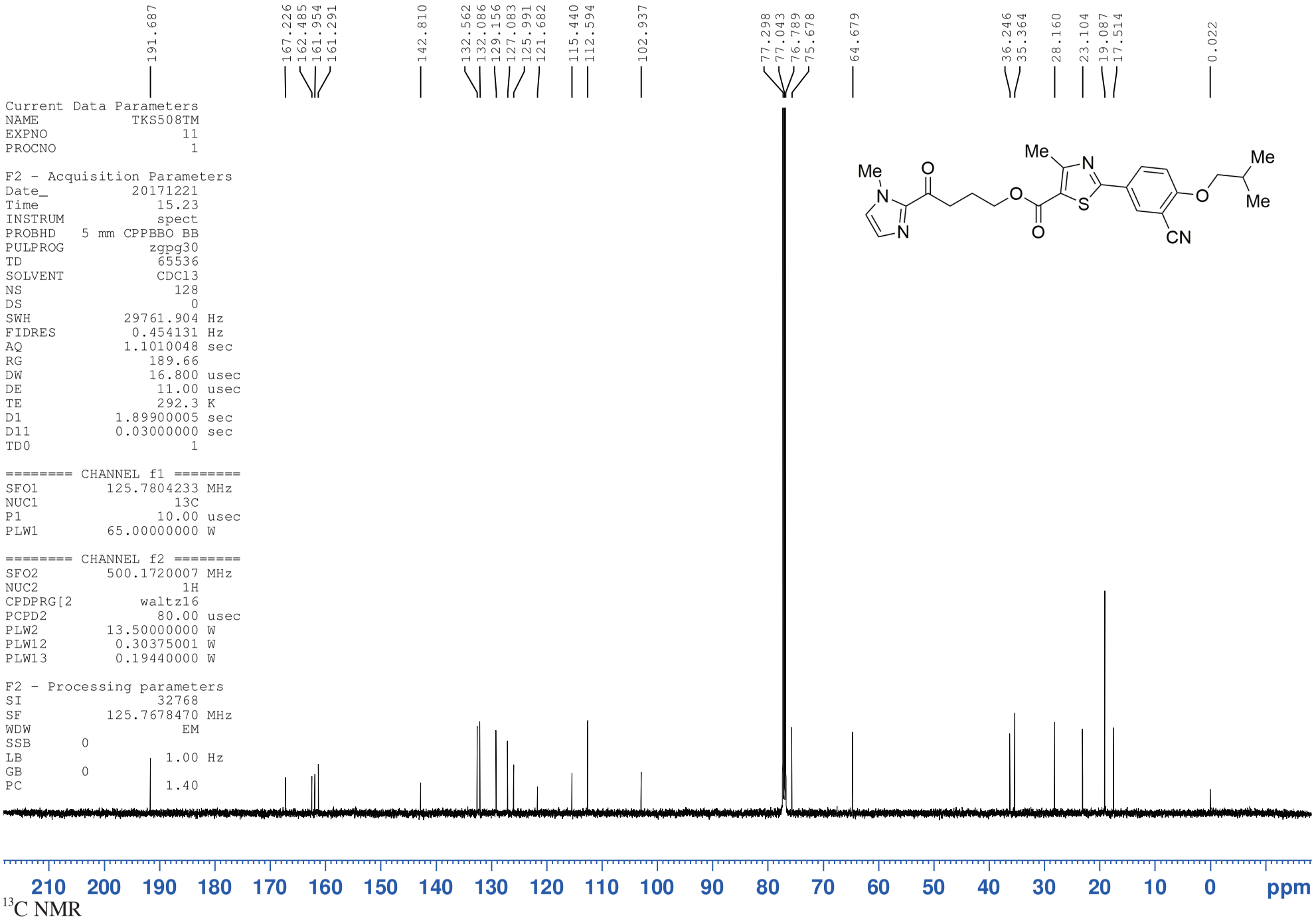
^{1}H NMR


191.709
170.843
168.312
156.039
142.756
139.184
135.914
133.947
131.211
130.757
130.641
129.110
129.083
127.039
114.983
112.609
111.792
101.047
77.312
77.057
76.803
64.360
55.709
36.204
35.273
30.291
23.035
13.432
0.023
Current Data Parameters
NAME TNK-695 TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171221
Time 18.36
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 73
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.9 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
O
Me
N
O
MeO
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

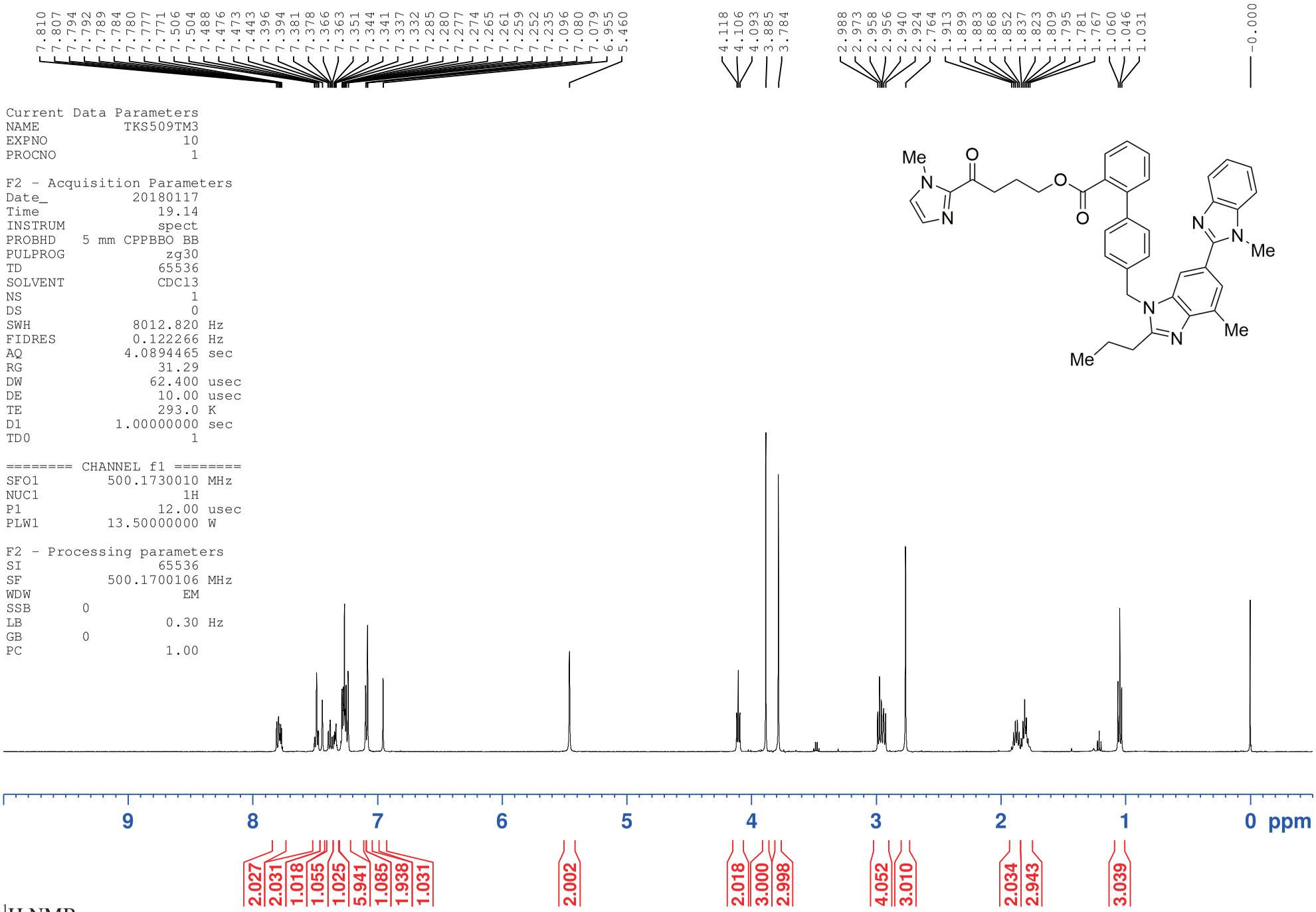
^{13}C NMR


8.167
8.162
8.095
8.090
8.077
8.073
7.263
7.145
7.143
7.043
7.020
7.002
4.404
4.392
4.379
4.004
3.907
3.894
3.325
3.310
3.296
2.753
2.236
2.222
2.209
2.194
2.181
2.166
1.560
1.100
1.086
-0.000
Current Data Parameters
NAME TKS508recrystalized
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180110
Time 15.35
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
O
O
Me
N
S
O
Me
CN
9
8
7
6
5
4
3
2
1
0 ppm
0.995
0.991
1.012
0.992
1.018
1.998
3.000
2.001
1.979
3.002
3.010
6.017

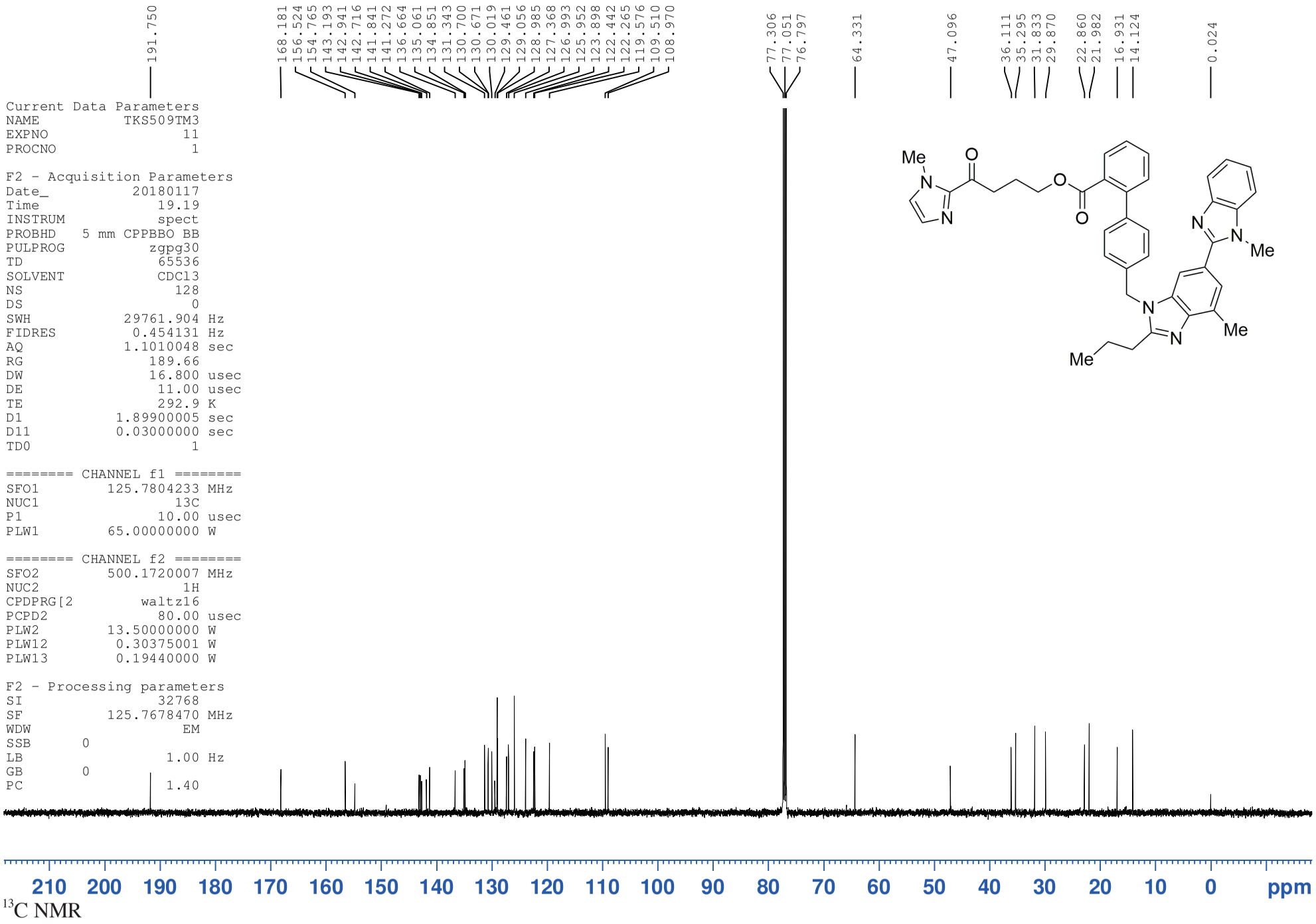
^{1}H NMR


191.687
167.226
162.485
161.954
161.291
142.810
132.562
132.086
129.156
127.083
125.991
121.682
115.440
112.594
102.937
77.298
77.043
76.789
75.678
64.679
36.246
35.364
28.160
23.104
19.087
17.514
0.022
Current Data Parameters
NAME TKS508TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171221
Time 15.23
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.3 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
N
Me
N
S
O
O
O
Me
Me
CN
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

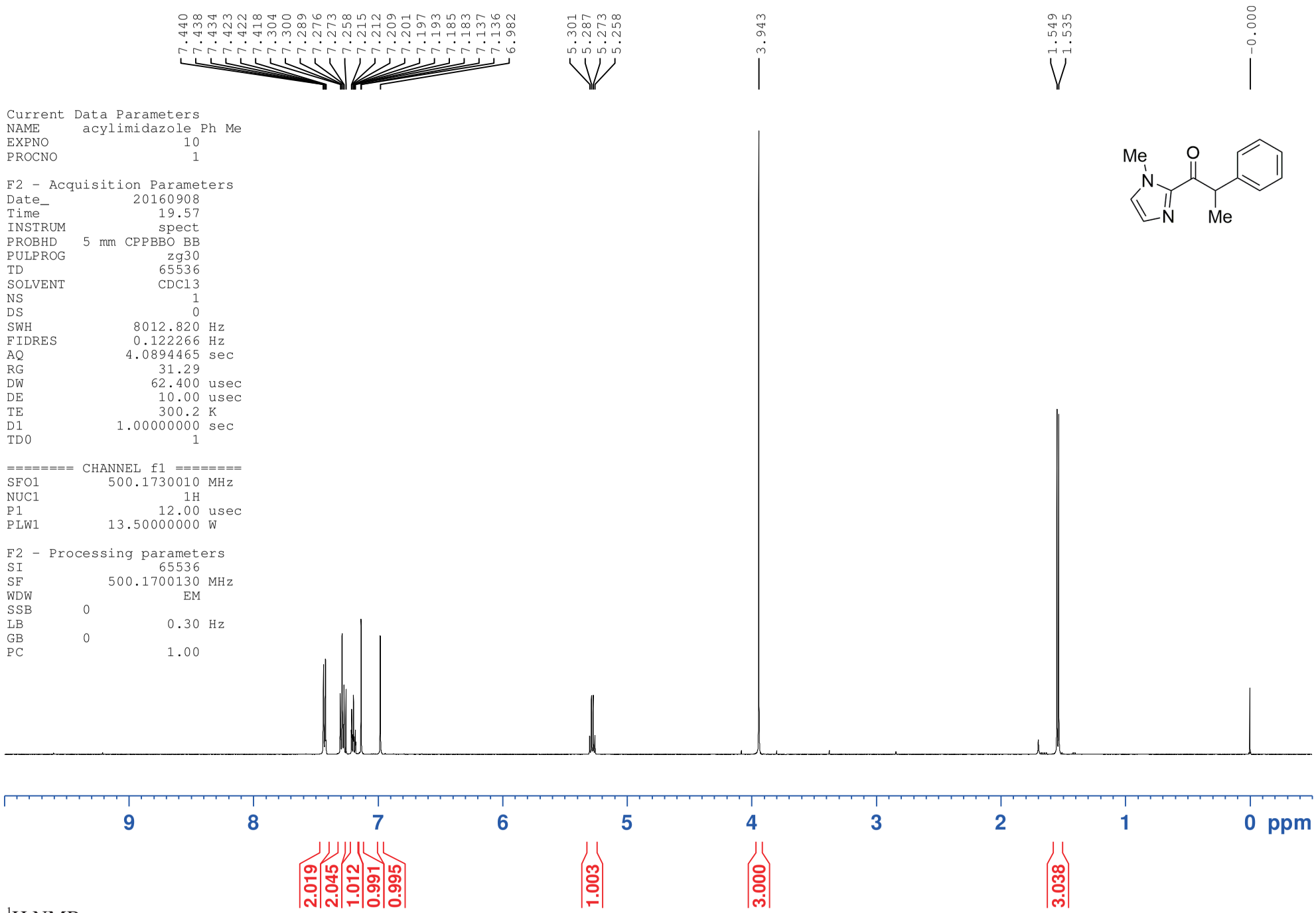
^{13}C NMR


7.810
7.807
7.794
7.792
7.789
7.784
7.780
7.777
7.771
7.506
7.504
7.488
7.476
7.473
7.443
7.396
7.394
7.381
7.378
7.366
7.363
7.351
7.344
7.341
7.337
7.332
7.285
7.280
7.277
7.274
7.265
7.261
7.259
7.252
7.235
7.096
7.080
7.079
6.955
5.460
4.118
4.106
4.093
3.885
3.784
2.988
2.973
2.958
2.956
2.940
2.924
2.764
1.913
1.899
1.883
1.868
1.852
1.837
1.823
1.809
1.795
1.781
1.767
1.060
1.046
1.031
-0.000
Current Data Parameters
NAME TKS509TM3
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180117
Time 19.14
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
O
O
N
N
Me
N
N
Me
Me
9
8
7
6
5
4
3
2
1
0 ppm
2.027
2.031
1.018
1.055
1.025
5.941
1.085
1.938
1.031
2.002
2.018
3.000
2.998
4.052
3.010
2.034
2.943
3.039

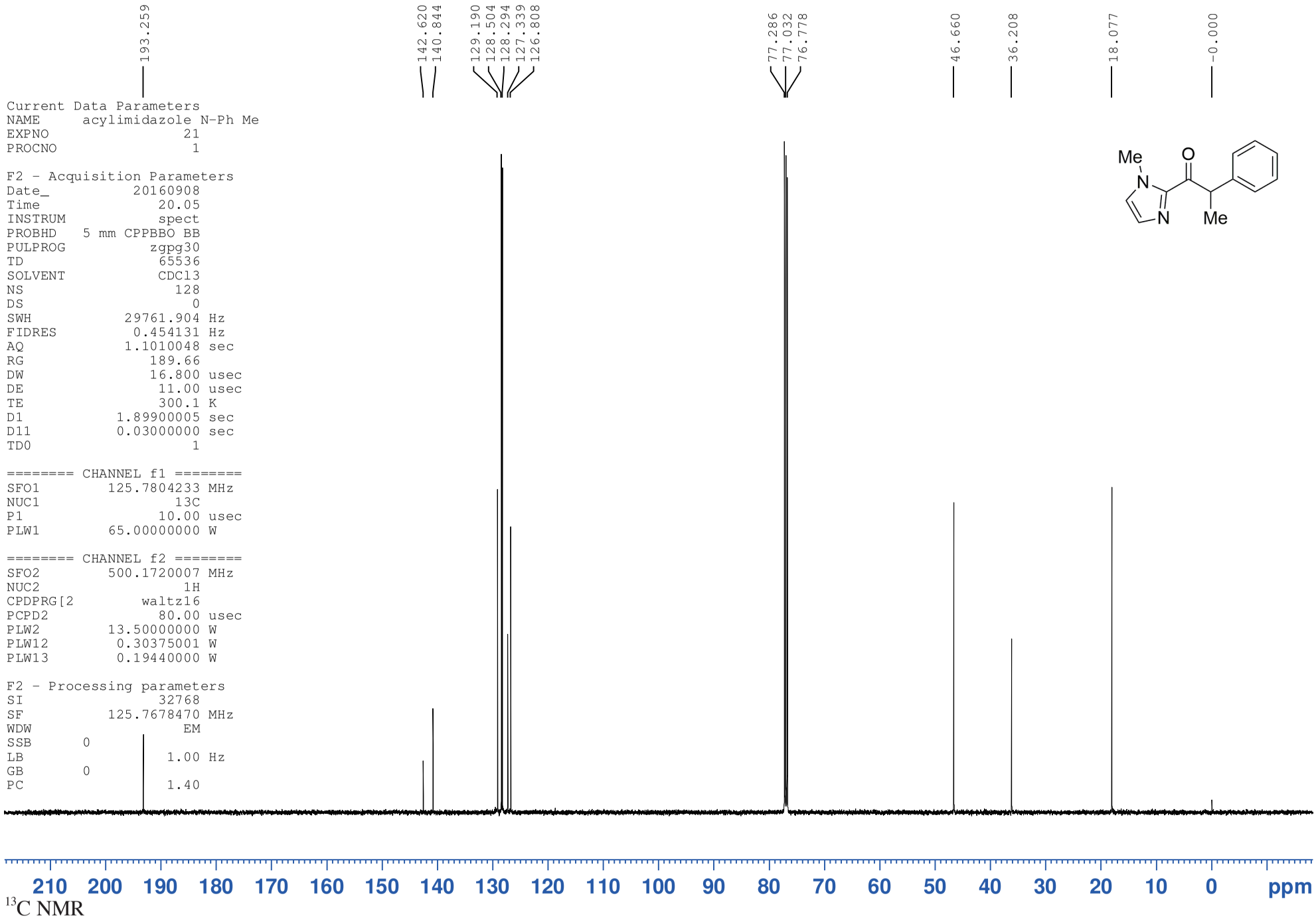
^{1}H NMR


191.750
168.181
156.524
154.765
143.193
142.941
142.716
141.841
141.272
136.664
135.061
134.851
131.343
130.700
130.671
130.019
129.461
129.056
128.985
127.368
126.993
125.952
123.898
122.442
122.265
119.576
109.510
108.970
77.306
77.051
76.797
64.331
47.096
36.111
35.295
31.833
29.870
22.860
21.982
16.931
14.124
0.024
Current Data Parameters
NAME TKS509TM3
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180117
Time 19.19
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.9 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
O
N
O
N
N
Me
N
Me
N
Me
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

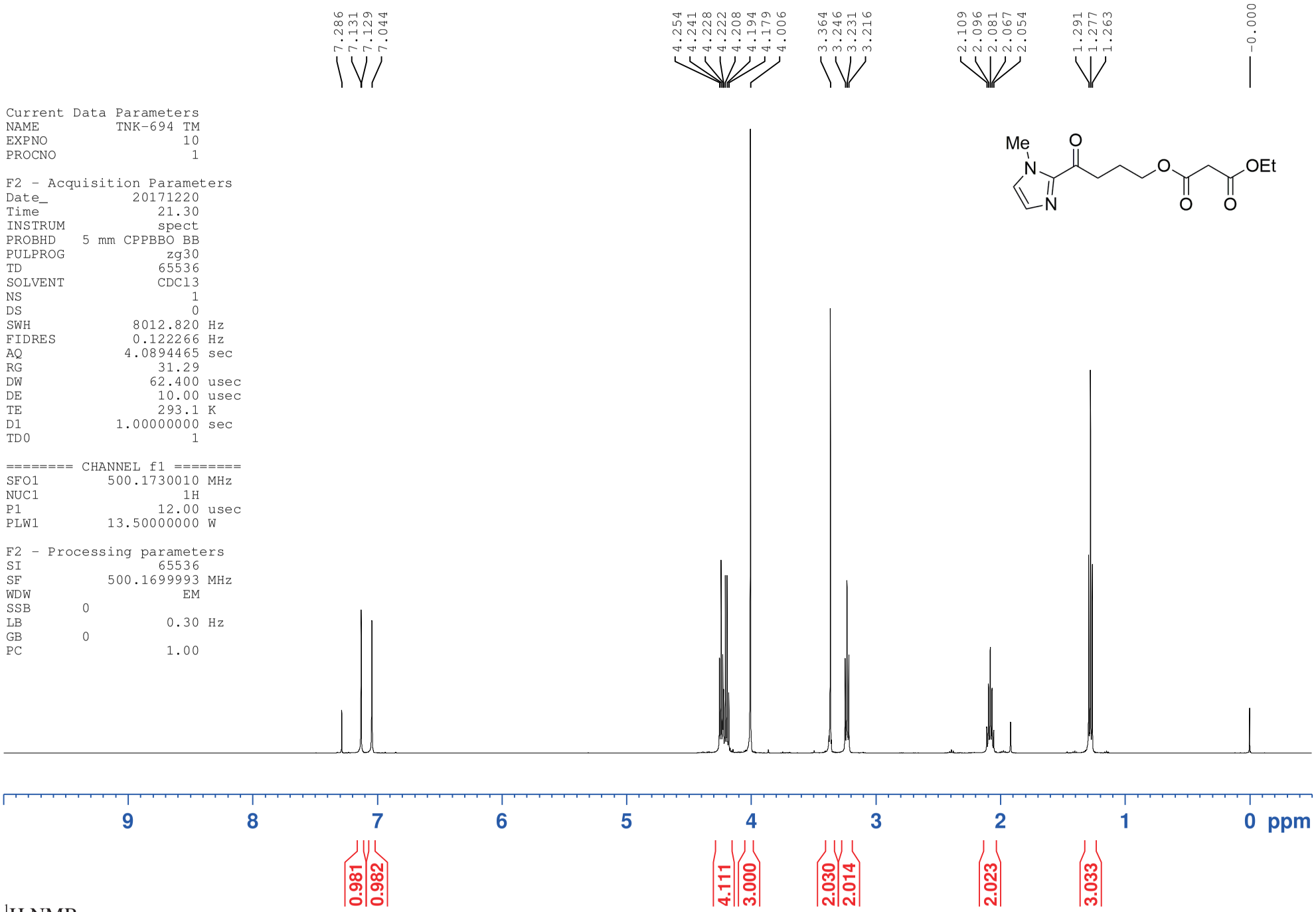
^{13}C NMR


7.440
7.438
7.434
7.423
7.422
7.418
7.304
7.300
7.289
7.276
7.273
7.258
7.215
7.212
7.209
7.201
7.197
7.193
7.185
7.183
7.137
7.136
6.982
5.301
5.287
5.273
5.258
3.943
1.549
1.535
0.000
Current Data Parameters
NAME acylimidazole Ph Me
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160908
Time 19.57
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
2.019
2.045
1.012
0.991
0.995
1.003
3.000
3.038

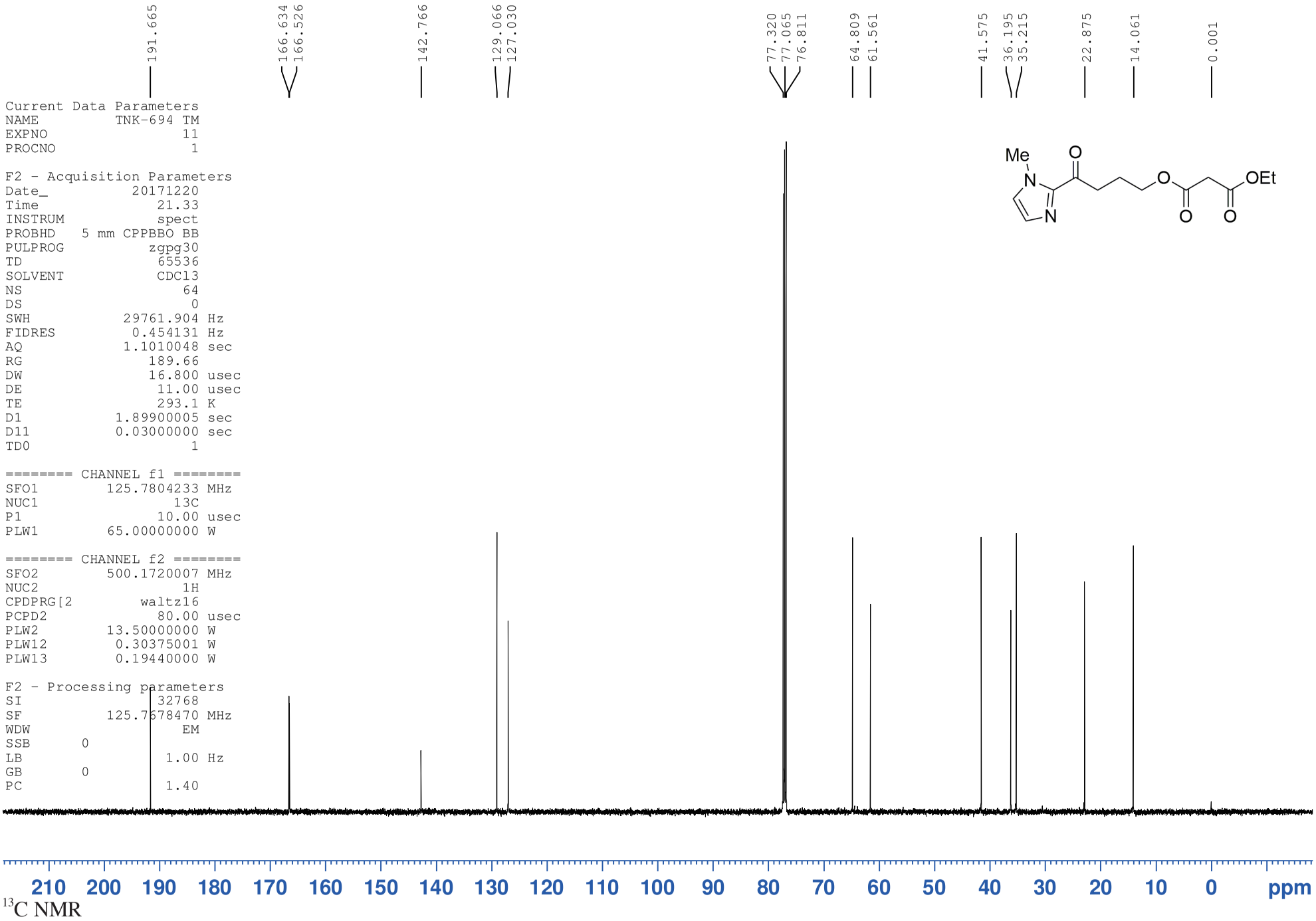
^{1}H NMR


193.259
142.620
140.844
129.190
128.504
128.294
127.339
126.808
77.286
77.032
76.778
46.660
36.208
18.077
-0.000
Current Data Parameters
NAME acylimidazole N-Ph Me
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160908
Time 20.05
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
N
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

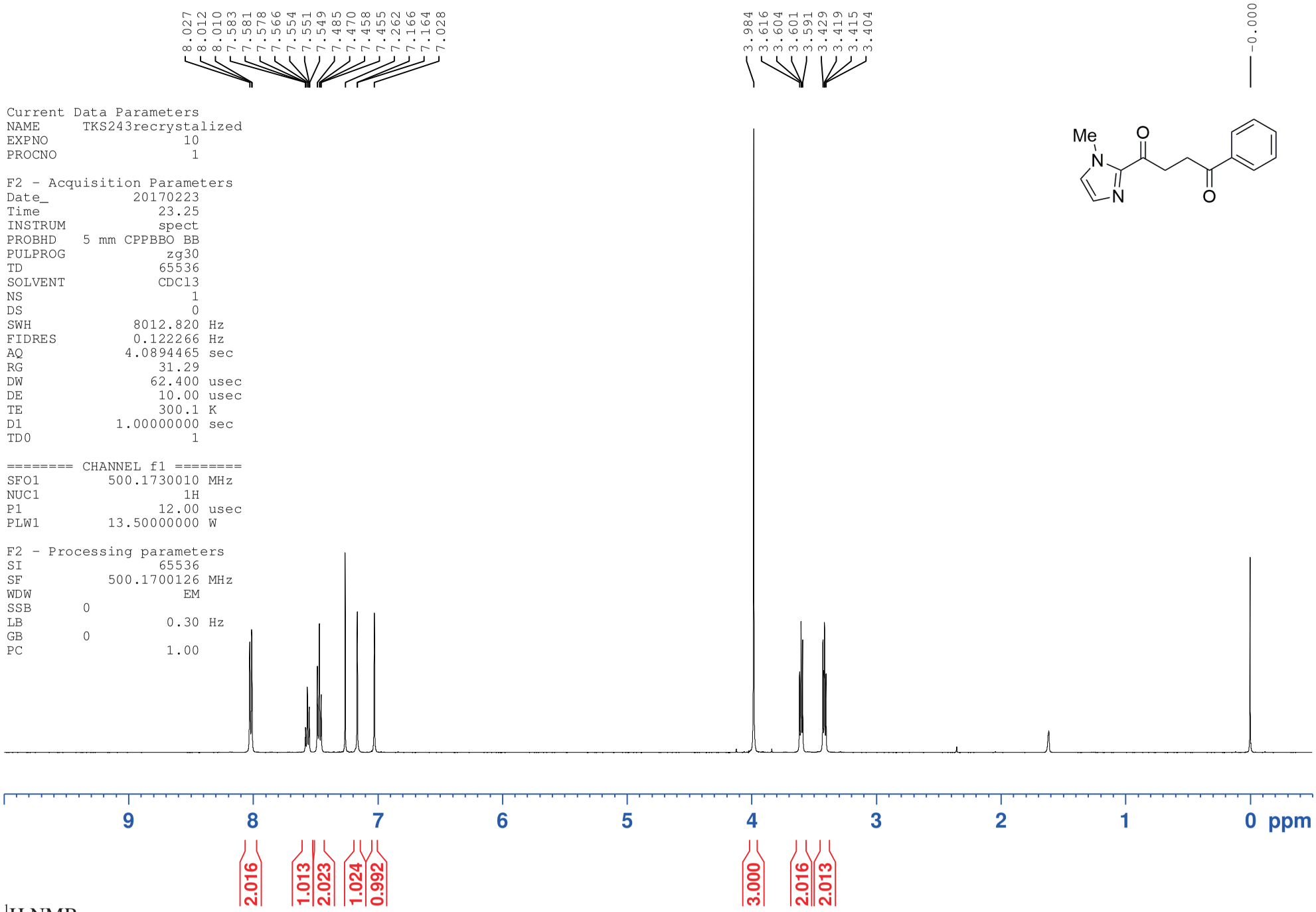
^{13}C NMR


7.286
7.131
7.129
7.044
4.254
4.241
4.228
4.222
4.208
4.194
4.179
4.006
3.364
3.246
3.231
3.216
2.109
2.096
2.081
2.067
2.054
1.291
1.277
1.263
-0.000
Current Data Parameters
NAME TNK-694 TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171220
Time 21.30
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 293.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1699993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
O
O
O
OEt
9
8
7
6
5
4
3
2
1
0 ppm
0.981
0.982
4.111
3.000
2.030
2.014
2.023
3.033

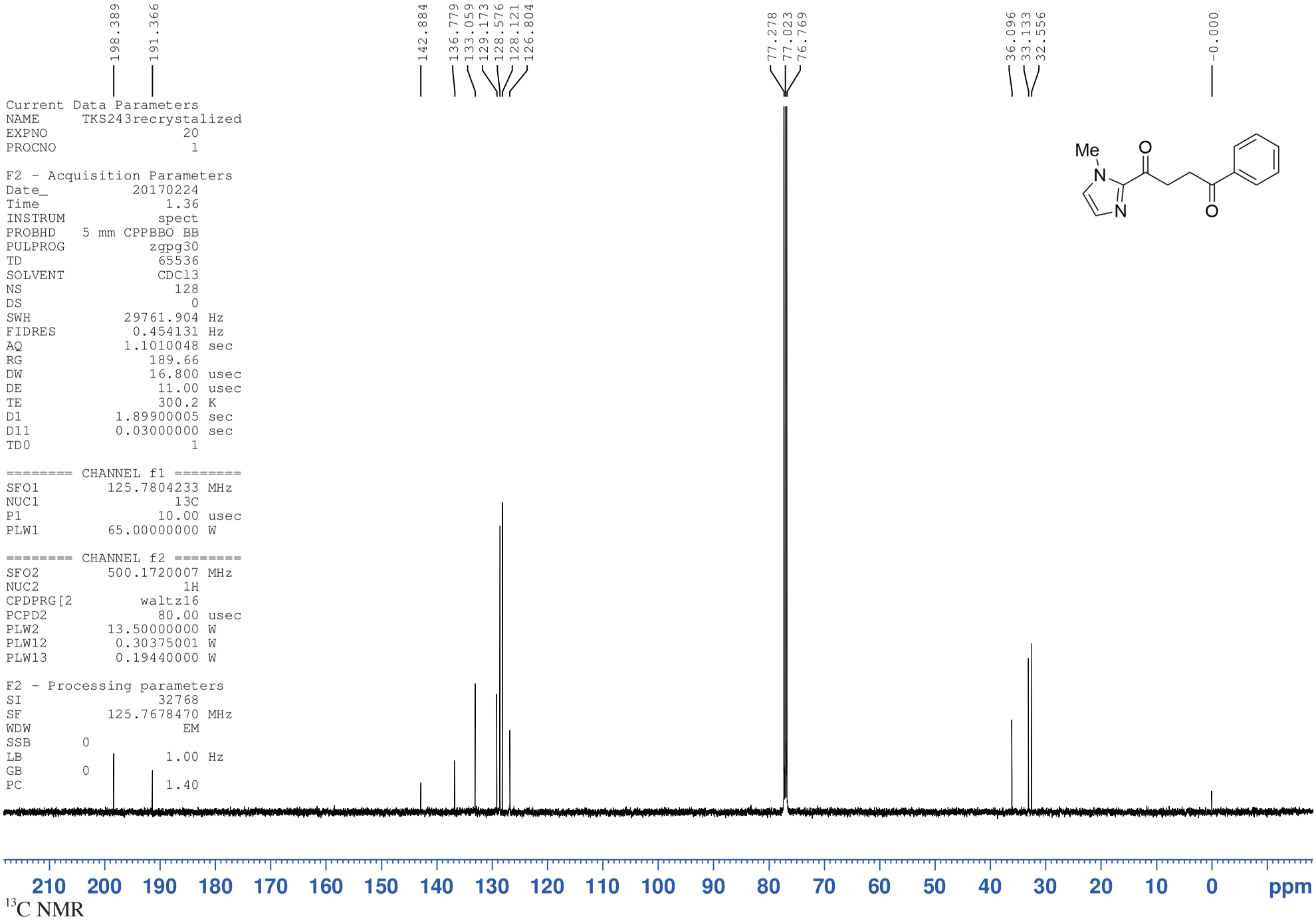
^{1}H NMR


191.665
166.634
166.526
142.766
129.066
127.030
77.320
77.065
76.811
64.809
61.561
41.575
36.195
35.215
22.875
14.061
0.001
Current Data Parameters
NAME TNK-694 TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171220
Time 21.33
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 293.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
O
O
OEt
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

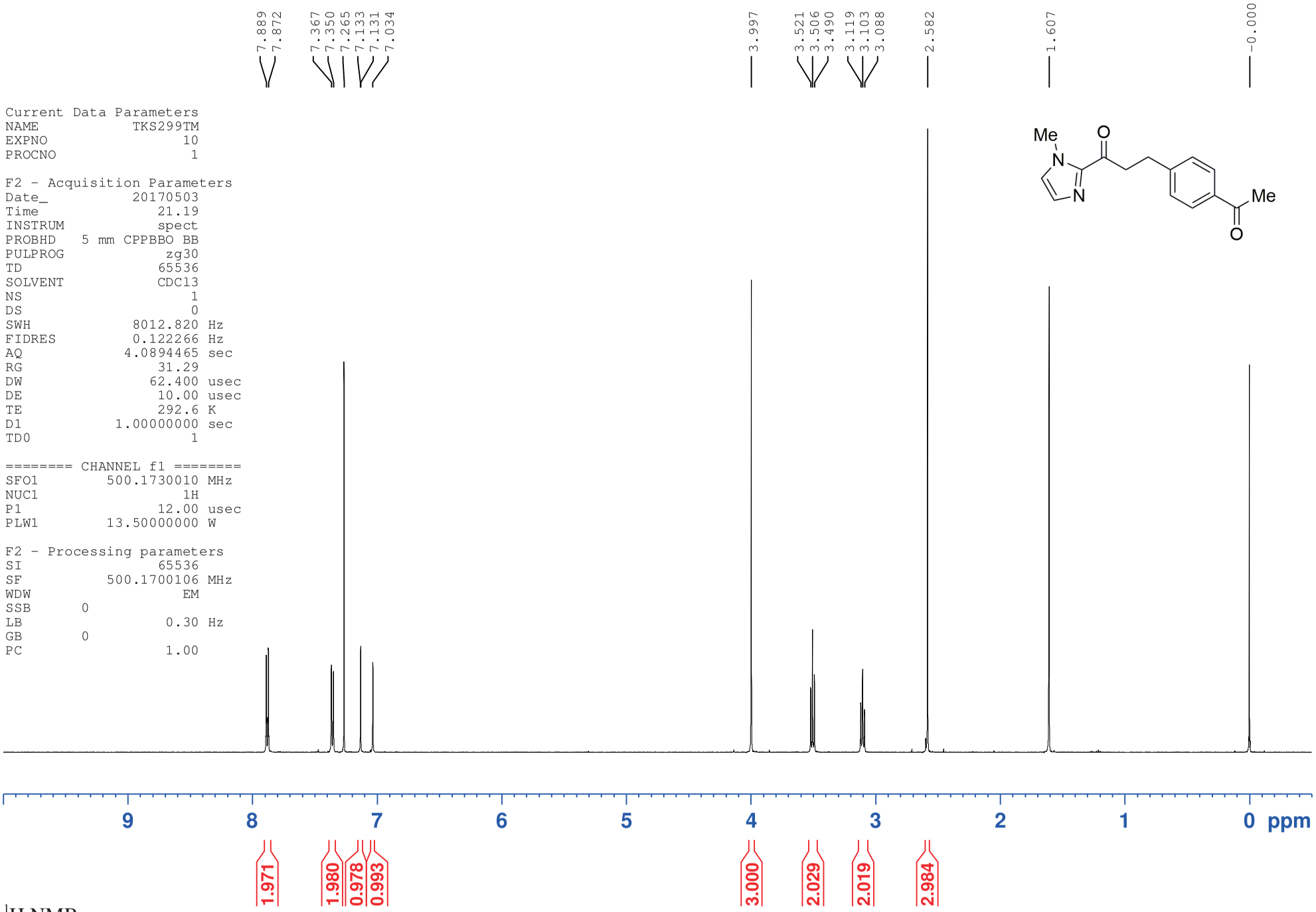
^{13}C NMR


8.027
8.012
8.010
7.583
7.581
7.578
7.566
7.554
7.551
7.549
7.485
7.470
7.458
7.455
7.262
7.166
7.164
7.028
3.984
3.616
3.604
3.601
3.591
3.429
3.419
3.415
3.404
-0.000
Current Data Parameters
NAME TKS243recrystalized
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170223
Time 23.25
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
O
9
8
7
6
5
4
3
2
1
0 ppm
2.016
1.013
2.023
1.024
0.992
3.000
2.016
2.013

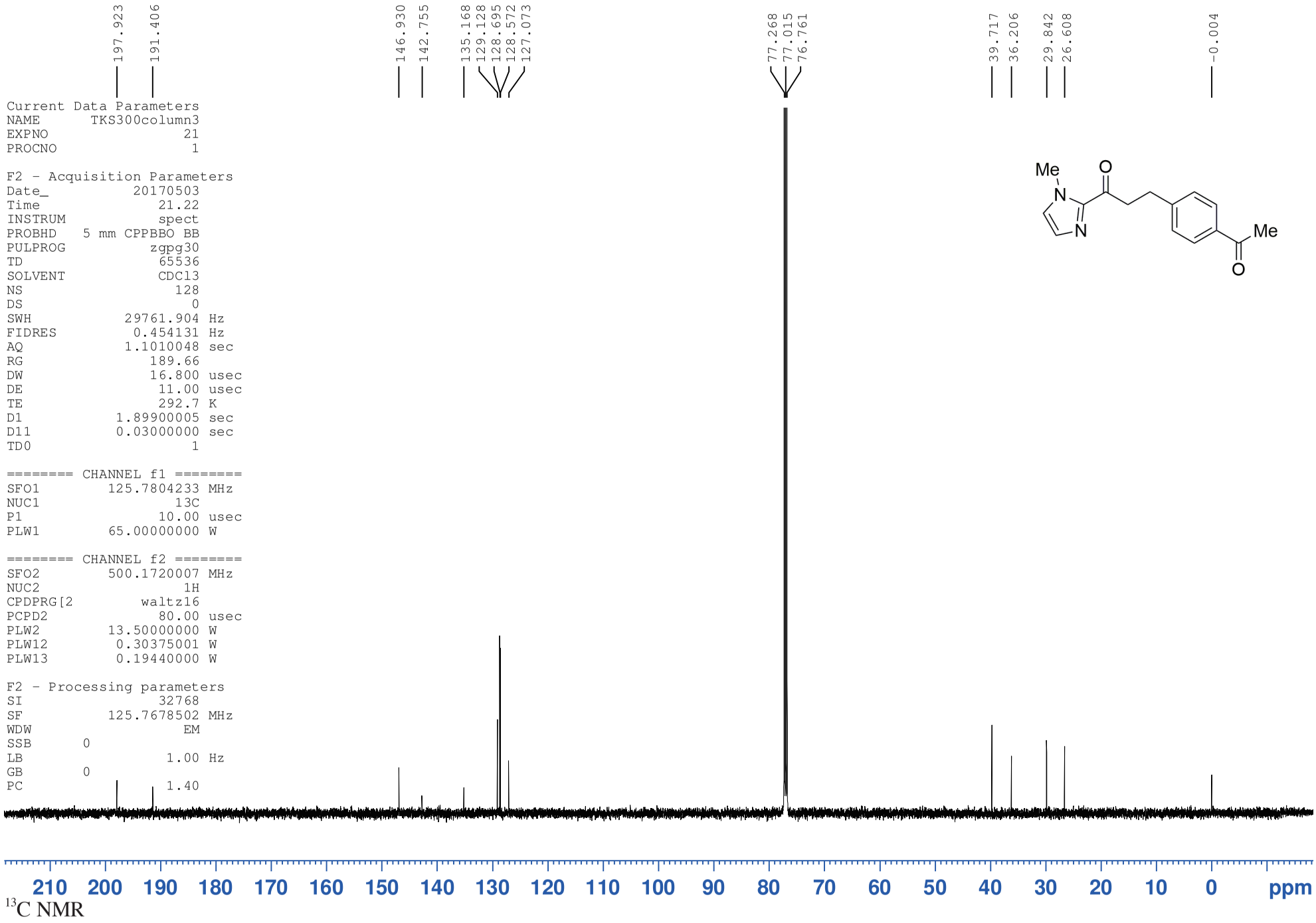
^{1}H NMR


198.389
191.366
142.884
136.779
133.059
129.173
128.576
128.121
126.804
77.278
77.023
76.769
36.096
33.133
32.556
-0.000
Me
N
N
O
O
Current Data Parameters
NAME TKS243recrystalized
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170224
Time 1.36
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

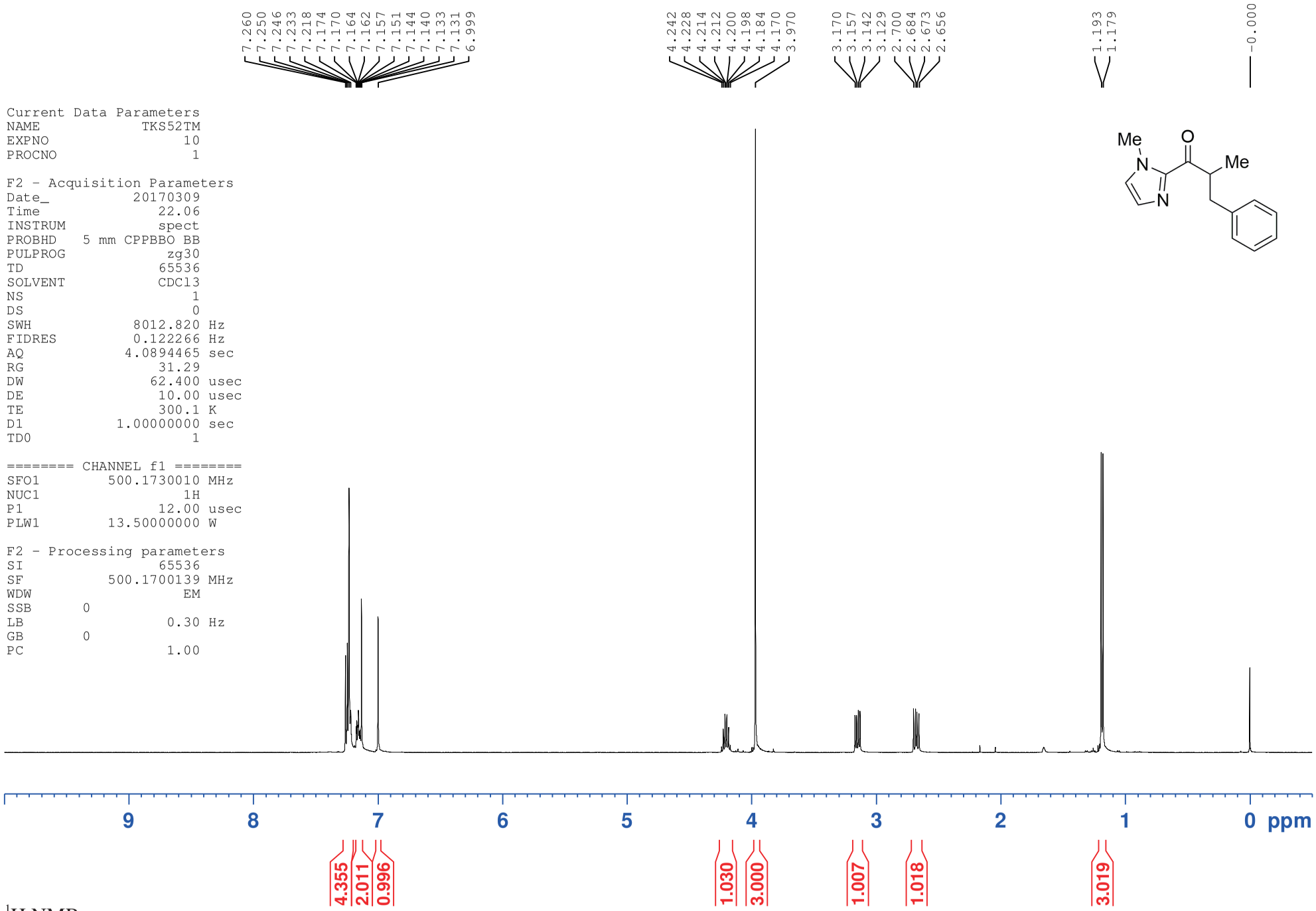
^{13}C NMR


7.889
7.872
7.367
7.350
7.265
7.133
7.131
7.034
3.997
3.521
3.506
3.490
3.119
3.103
3.088
2.582
1.607
-0.000
Current Data Parameters
NAME TKS299TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170503
Time 21.19
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
O
9
8
7
6
5
4
3
2
1
0 ppm
1.971
1.980
0.978
0.993
3.000
2.029
2.019
2.984

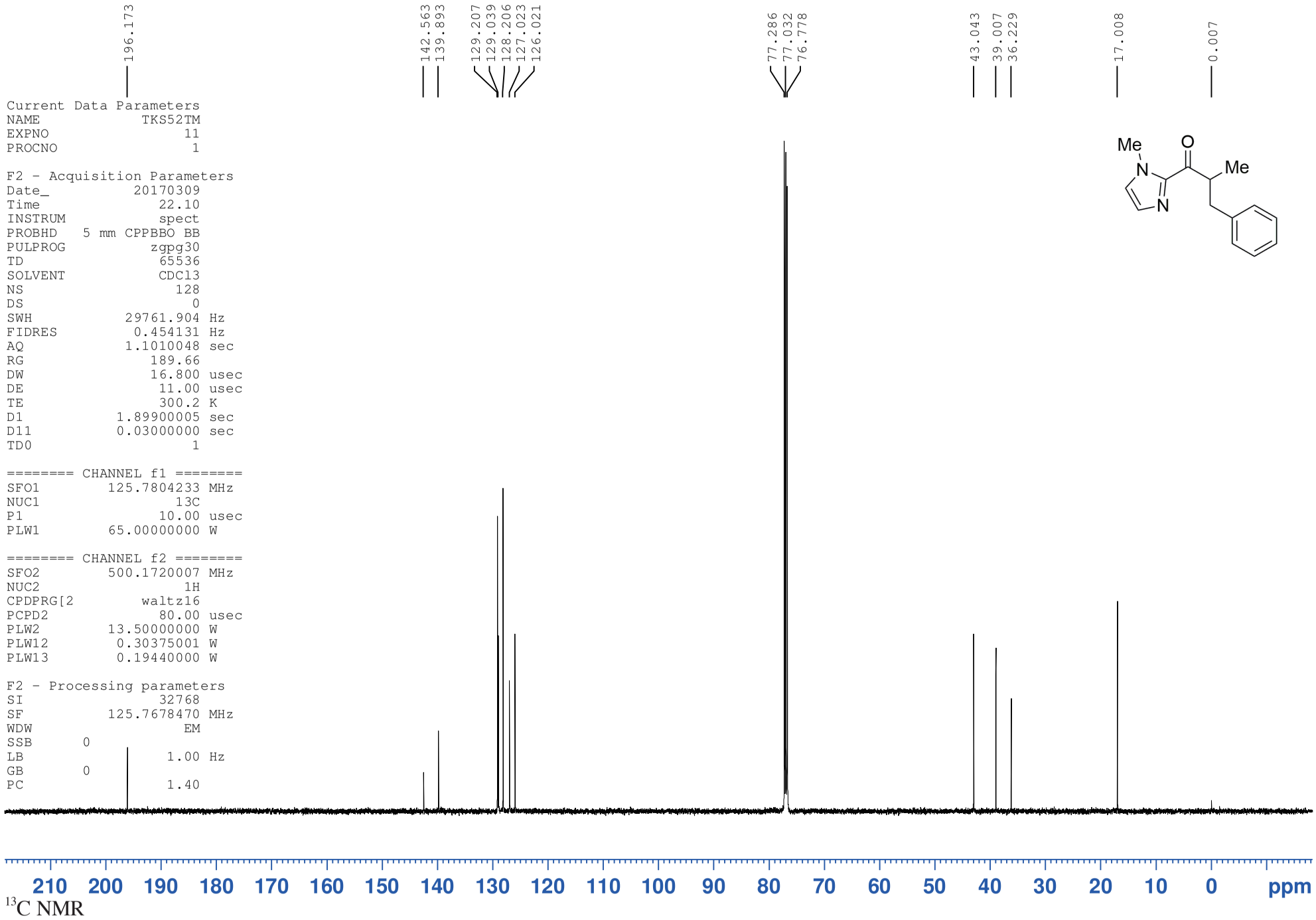
^{1}H NMR


197.923
191.406
146.930
142.755
135.168
129.128
128.695
128.572
127.073
77.268
77.015
76.761
39.717
36.206
29.842
26.608
-0.004
Current Data Parameters
NAME TKS300column3
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170503
Time 21.22
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.7 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678502 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

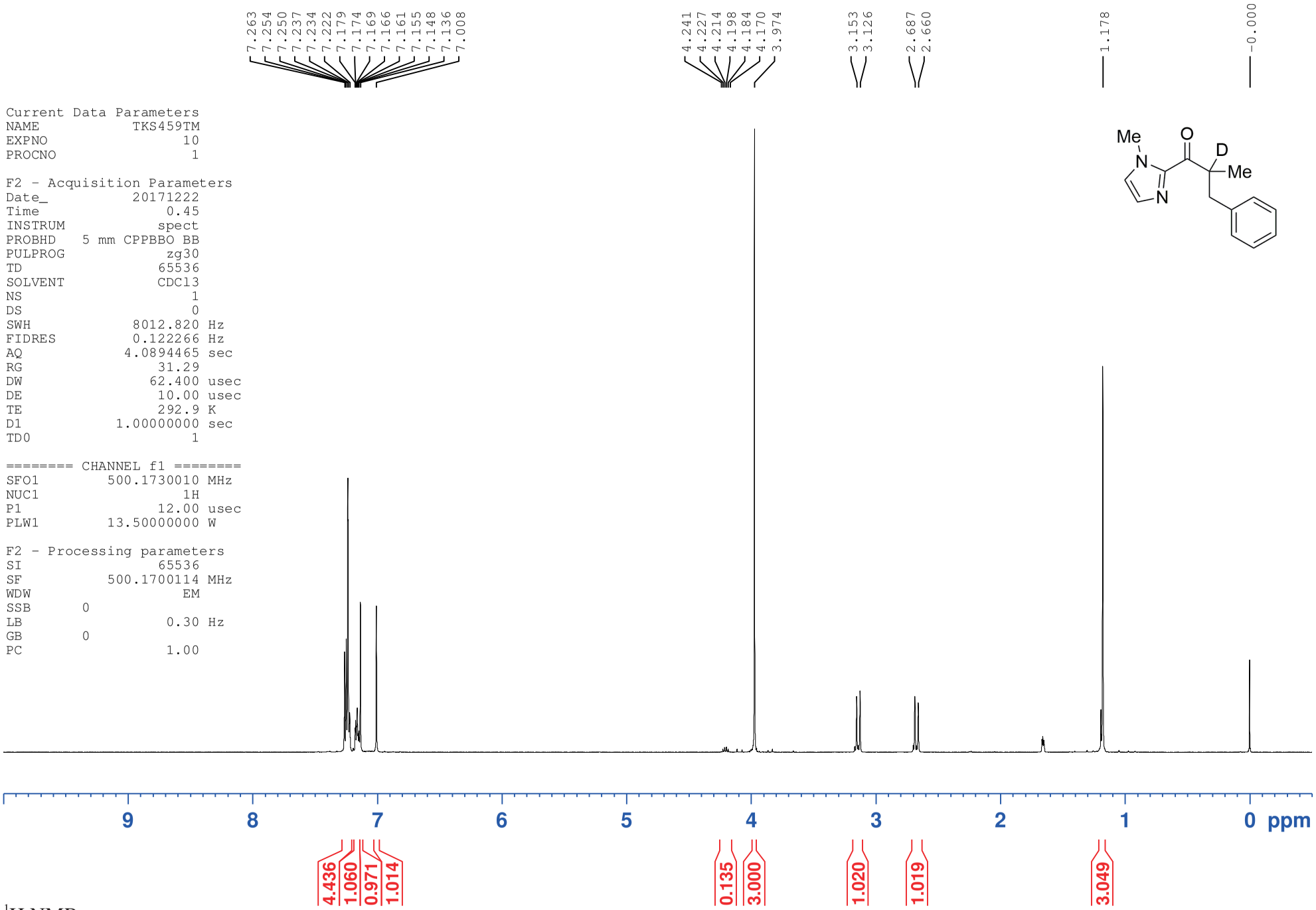
^{13}C NMR


7.260
7.250
7.246
7.233
7.218
7.174
7.170
7.164
7.162
7.157
7.151
7.144
7.140
7.133
7.131
6.999
4.242
4.228
4.214
4.212
4.200
4.198
4.184
4.170
3.970
3.170
3.157
3.142
3.129
2.700
2.684
2.673
2.656
1.193
1.179
-0.000
Me
N
N
O
Me
Current Data Parameters
NAME TKS52TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170309
Time 22.06
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
4.355
2.011
0.996
1.030
3.000
1.007
1.018
3.019

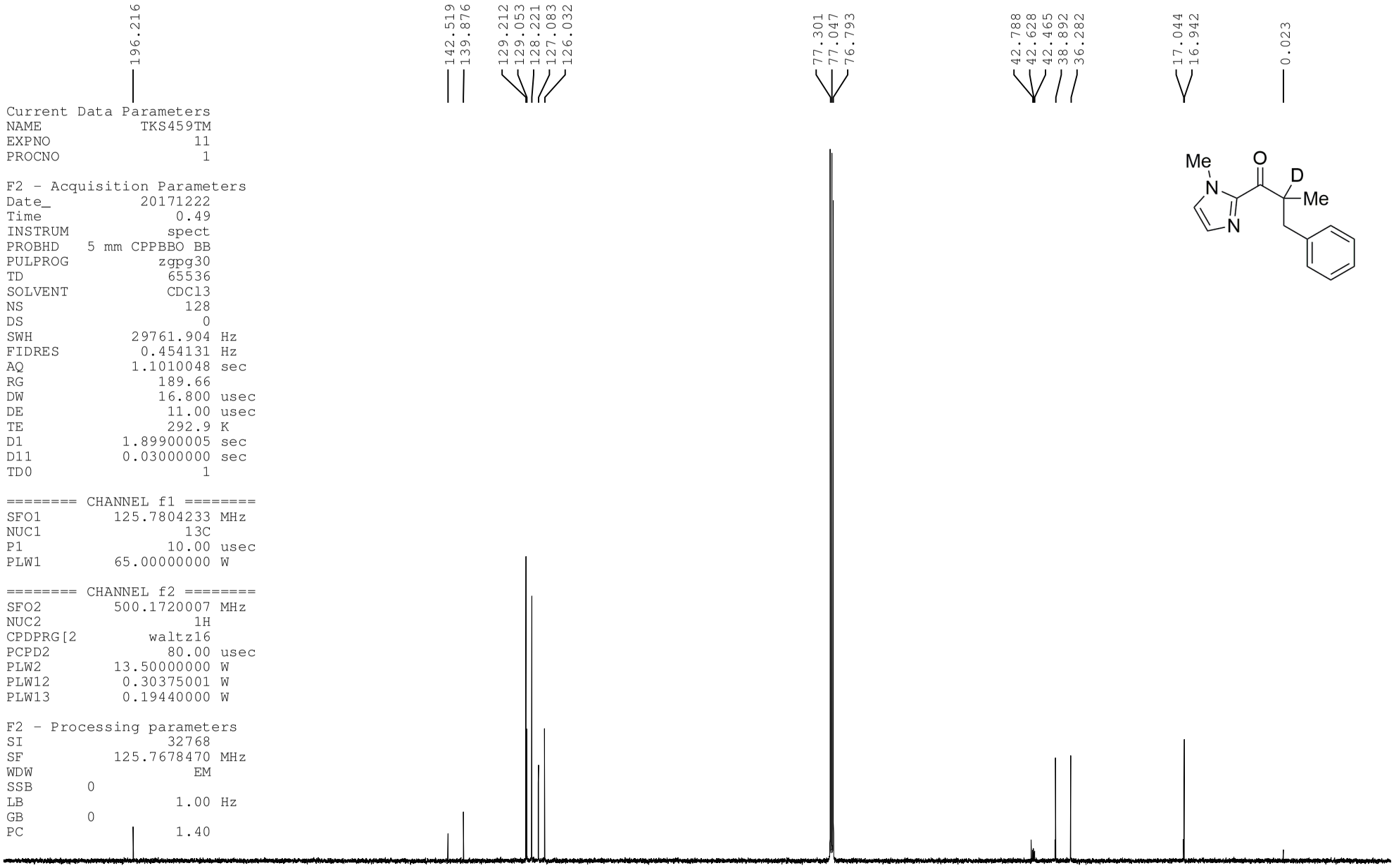
^{1}H NMR


196.173
142.563
139.893
129.207
129.039
128.206
127.023
126.021
77.286
77.032
76.778
43.043
39.007
36.229
17.008
0.007
Current Data Parameters
NAME TKS52TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170309
Time 22.10
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

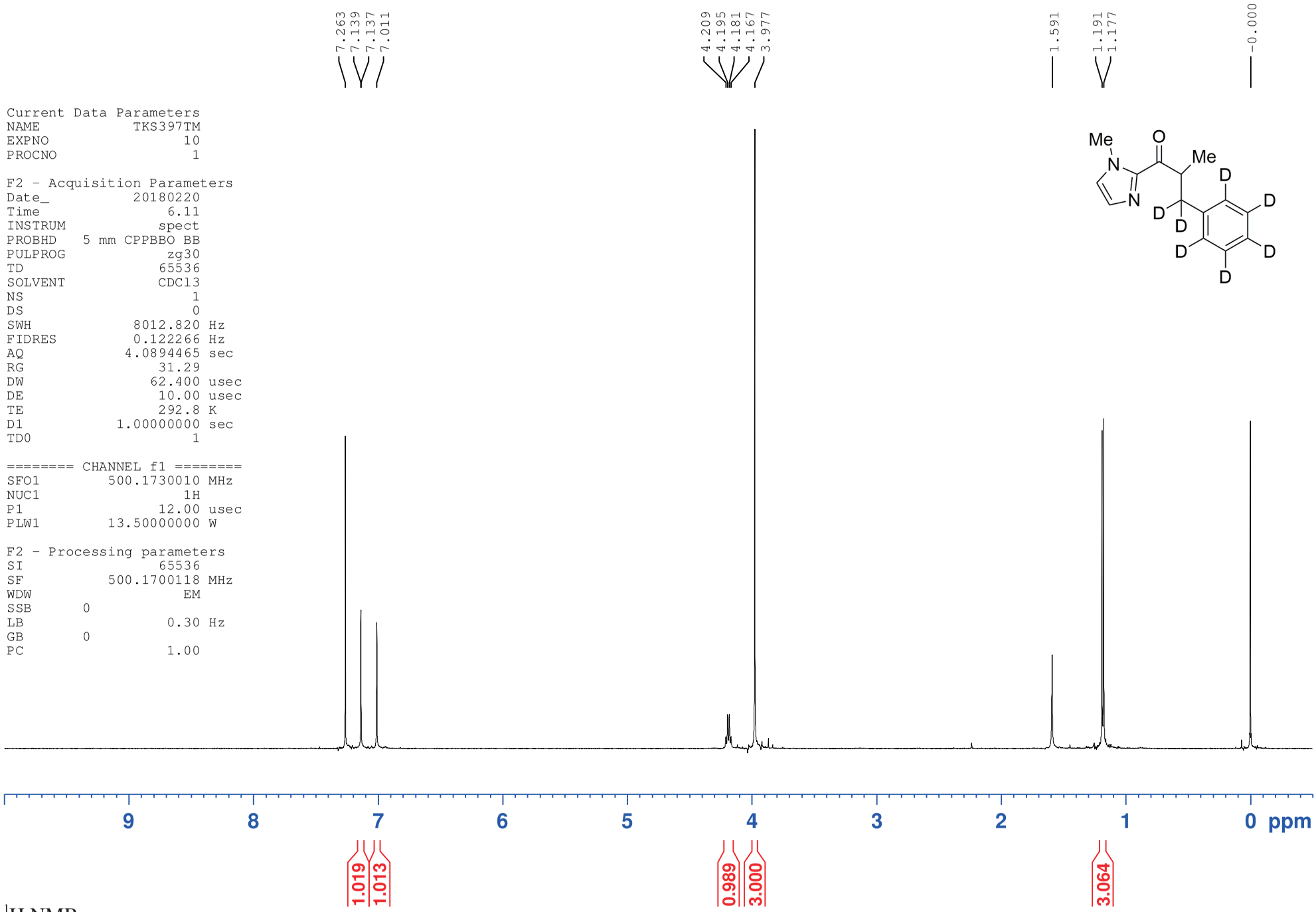
^{13}C NMR


7.263
7.254
7.250
7.237
7.234
7.222
7.179
7.174
7.169
7.166
7.161
7.155
7.148
7.136
7.008
4.241
4.227
4.214
4.198
4.184
4.170
3.974
3.153
3.126
2.687
2.660
1.178
-0.000
Current Data Parameters
NAME TKS459TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171222
Time 0.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
D
Me
9
8
7
6
5
4
3
2
1
0 ppm
4.436
1.060
0.971
1.014
0.135
3.000
1.020
1.019
3.049

^{1}H NMR


196.216
142.519
139.876
129.212
129.053
128.221
127.083
126.032
77.301
77.047
76.793
42.788
42.628
42.465
38.892
36.282
17.044
16.942
0.023
Current Data Parameters
NAME TKS459TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171222
Time 0.49
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.9 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
D
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR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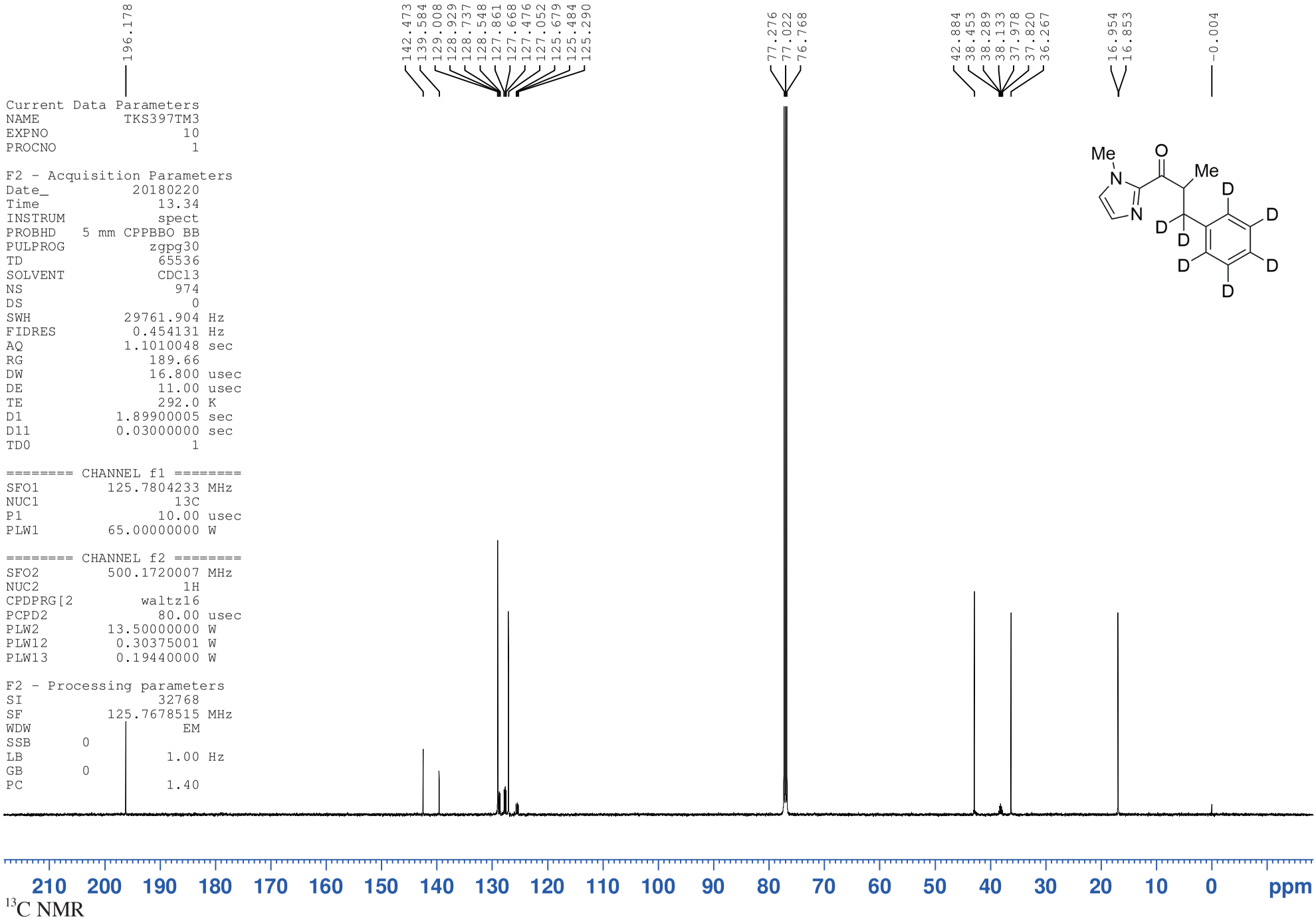
7.139
7.137
7.011
4.209
4.195
4.181
4.167
3.977
1.591
1.191
1.177
-0.000
Me
N
N
O
Me
D
D
D
D
D
D
D
Current Data Parameters
NAME TKS397TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180220
Time 6.11
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
1.019
1.013
0.989
3.000
3.064

^{1}H NMR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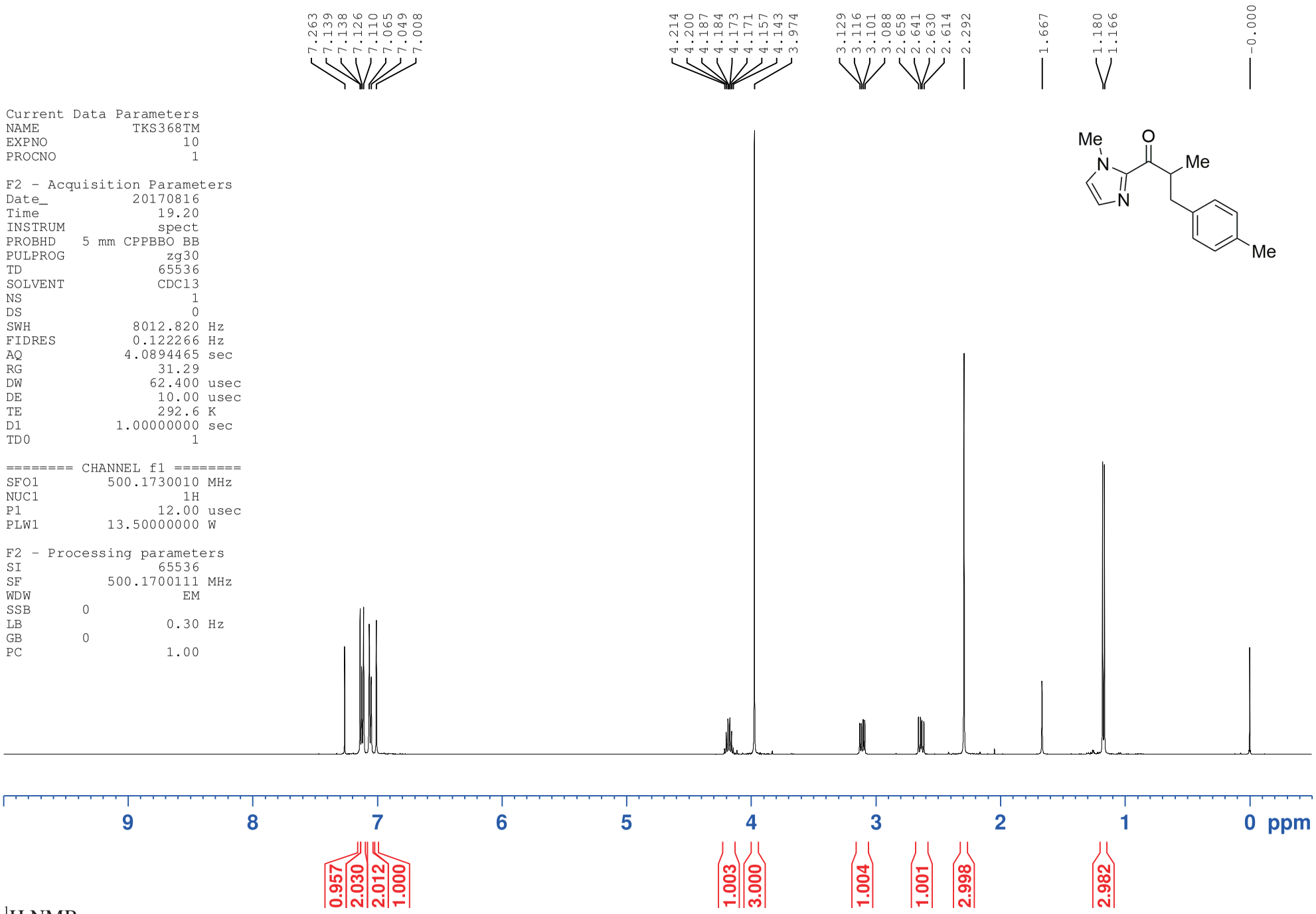
142.473
139.584
129.008
128.929
128.737
128.548
127.861
127.668
127.476
127.052
125.679
125.484
125.290
77.276
77.022
76.768
42.884
38.453
38.289
38.133
37.978
37.820
36.267
16.954
16.853
-0.004
Current Data Parameters
NAME TKS397TM3
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180220
Time 13.34
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 974
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.0 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678515 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
Me
N
D
D
D
D
D
D
D
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR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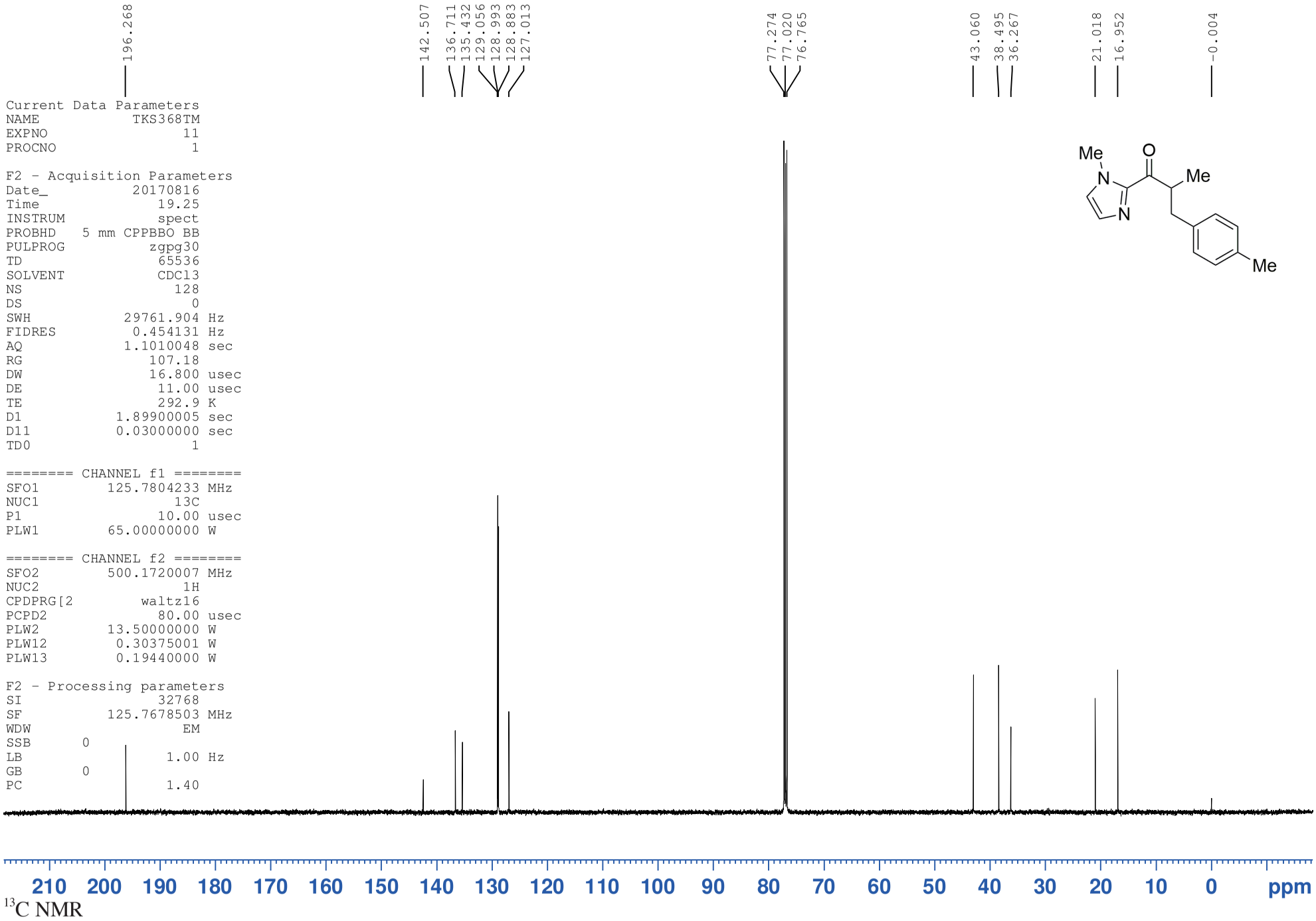
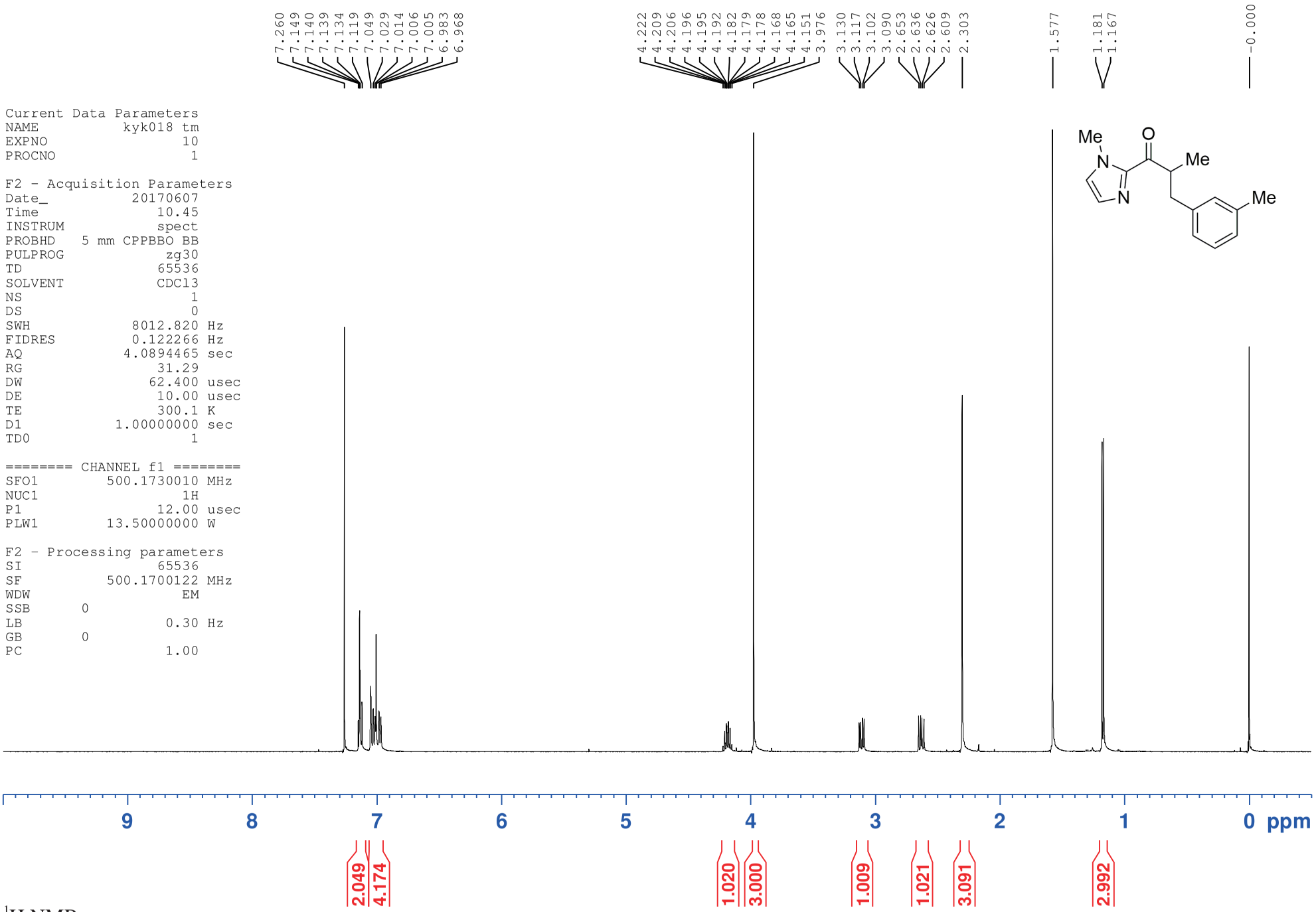
7.139
7.138
7.126
7.110
7.065
7.049
7.008
4.214
4.200
4.187
4.184
4.173
4.171
4.157
4.143
3.974
3.129
3.116
3.101
3.088
2.658
2.641
2.630
2.614
2.292
1.667
1.180
1.166
-0.000
Current Data Parameters
NAME TKS368TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170816
Time 19.20
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700111 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
Me
9
8
7
6
5
4
3
2
1
0 ppm
0.957
2.030
2.012
1.000
1.003
3.000
1.004
1.001
2.998
2.982

^{1}H NMR

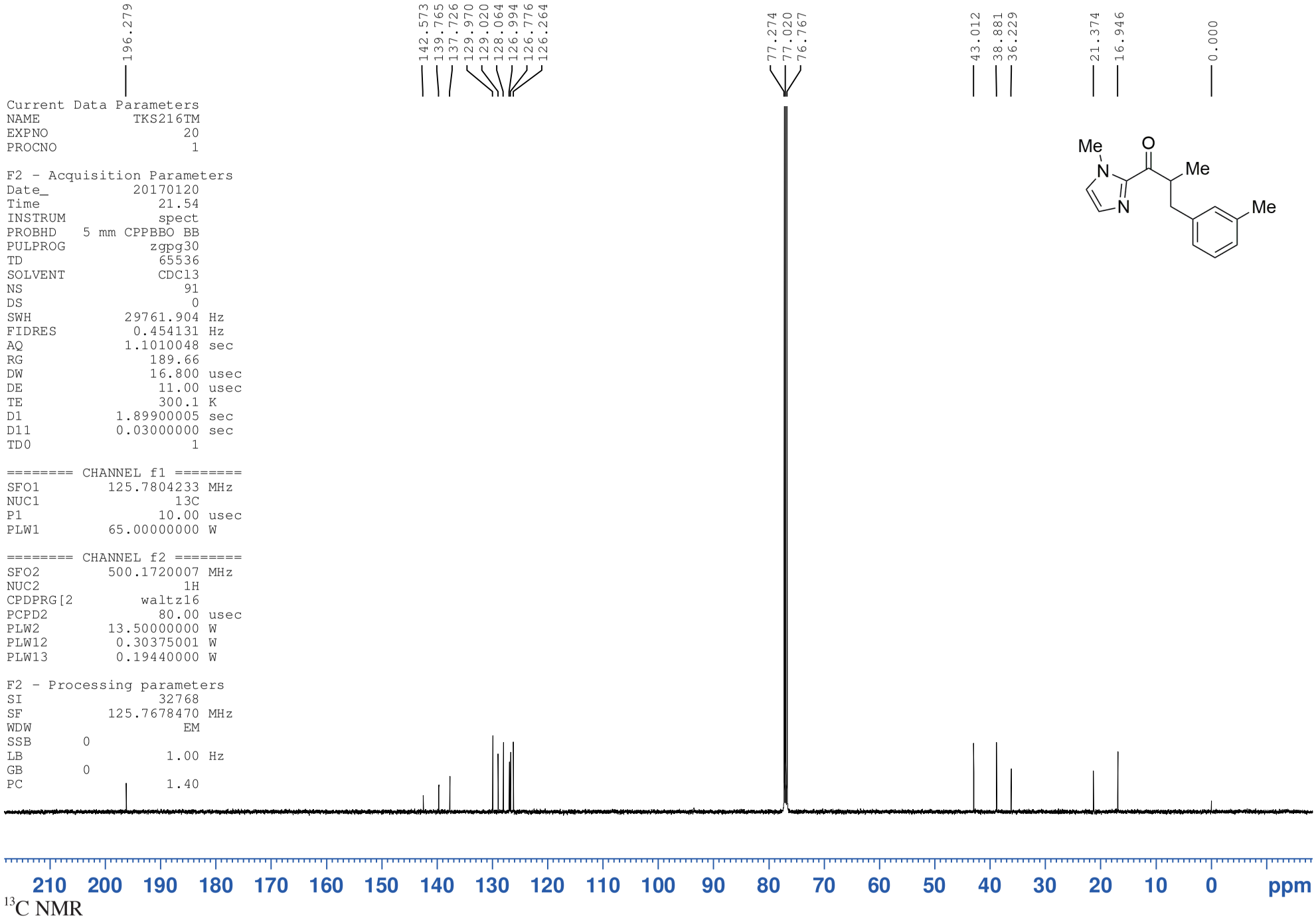

196.268
142.507
136.711
135.432
129.056
128.993
128.883
127.013
77.274
77.020
76.765
43.060
38.495
36.267
21.018
16.952
-0.004
Current Data Parameters
NAME TKS368TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170816
Time 19.25
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 292.9 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678503 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
Me
N
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

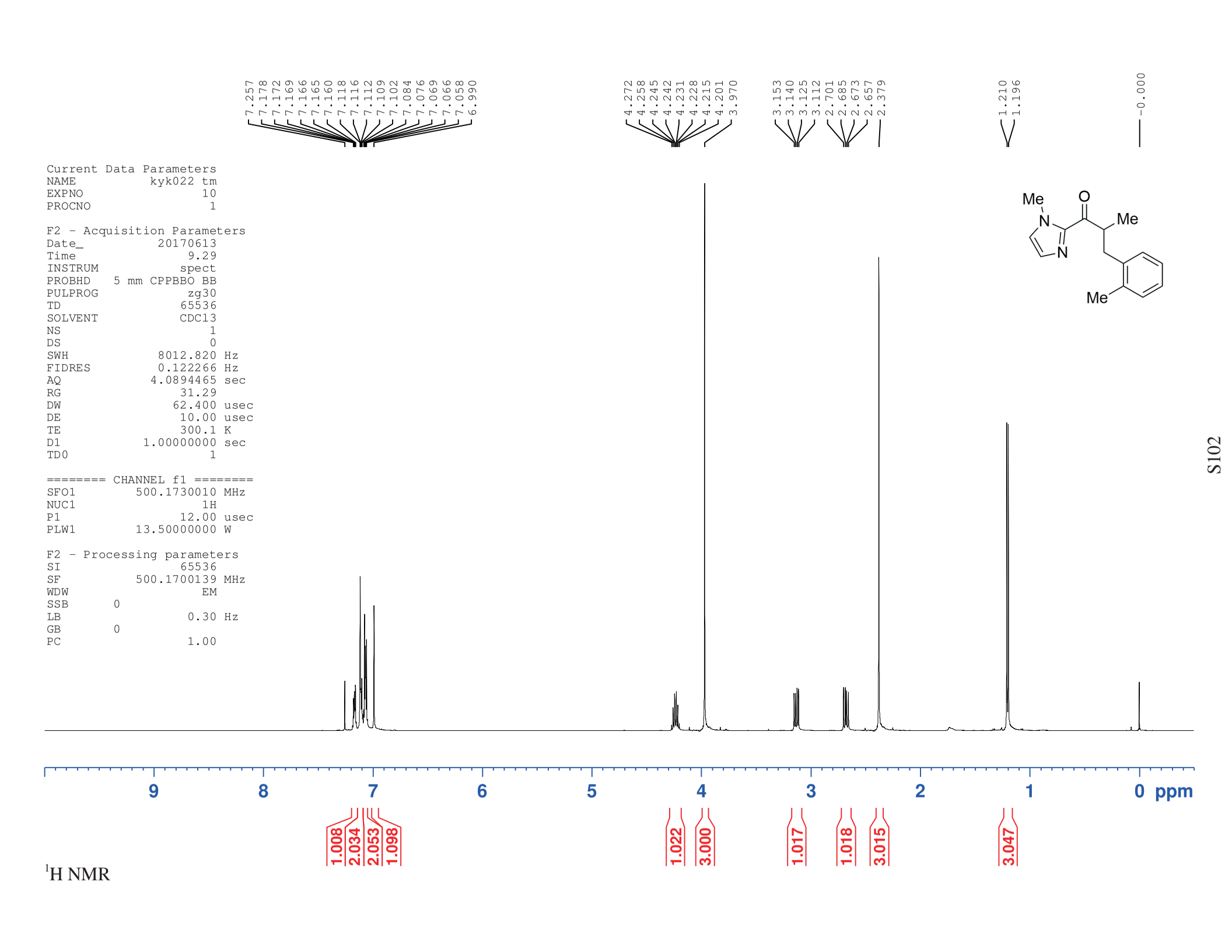

7.260
7.149
7.140
7.139
7.134
7.119
7.049
7.029
7.014
7.006
7.005
6.983
6.968
4.222
4.209
4.206
4.196
4.195
4.192
4.182
4.179
4.178
4.168
4.165
4.151
3.976
3.130
3.117
3.102
3.090
2.653
2.636
2.626
2.609
2.303
1.577
1.181
1.167
-0.000
Current Data Parameters
NAME kyk018 tm
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170607
Time 10.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
Me
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
2.049
4.174
1.020
3.000
1.009
1.021
3.091
2.992

S100

^{1}H NMR

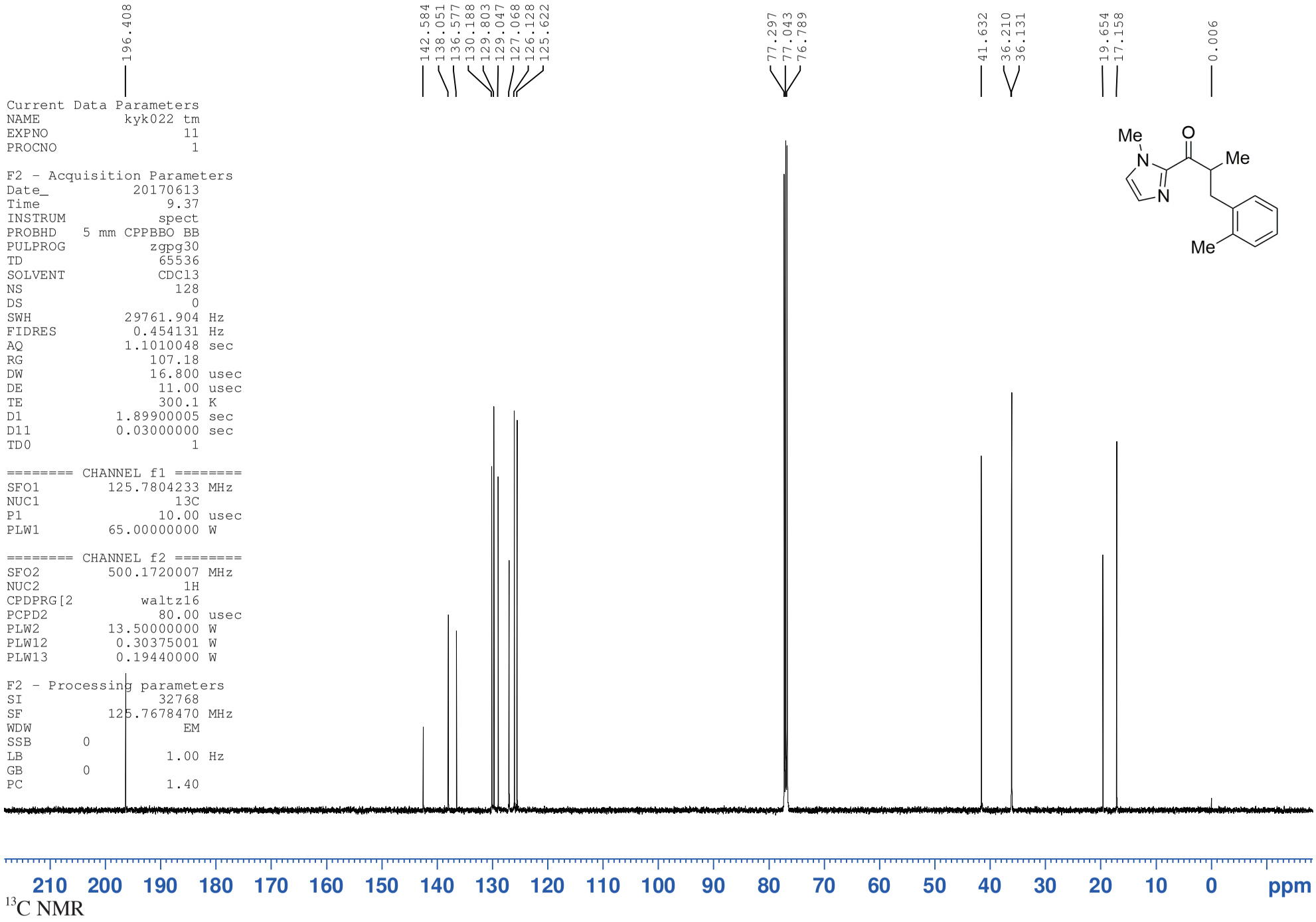

196.279
142.573
139.765
137.726
129.970
129.020
128.064
126.994
126.776
126.264
77.274
77.020
76.767
43.012
38.881
36.229
21.374
16.946
0.000
Current Data Parameters
NAME TKS216TM
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170120
Time 21.54
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 91
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

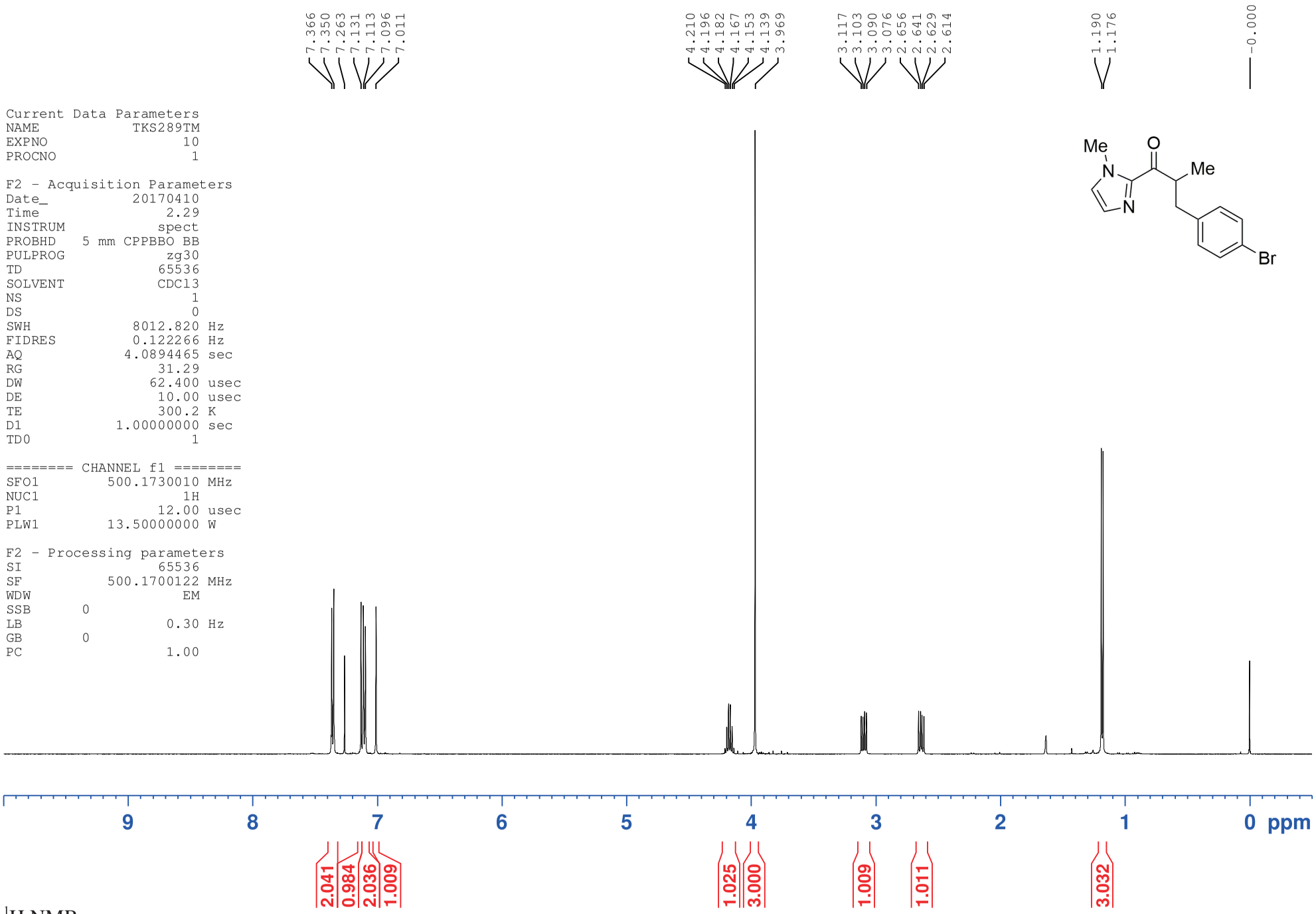

7.257
7.178
7.172
7.169
7.166
7.165
7.160
7.118
7.116
7.112
7.109
7.102
7.084
7.076
7.069
7.066
7.058
6.990
4.272
4.258
4.245
4.242
4.231
4.228
4.215
4.201
3.970
3.153
3.140
3.125
3.112
2.701
2.685
2.673
2.657
2.379
1.210
1.196
-0.000
Current Data Parameters
NAME kyk022 tm
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170613
Time 9.29
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
Me
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
1.008
2.034
2.053
1.098
1.022
3.000
1.017
1.018
3.015
3.047

S102

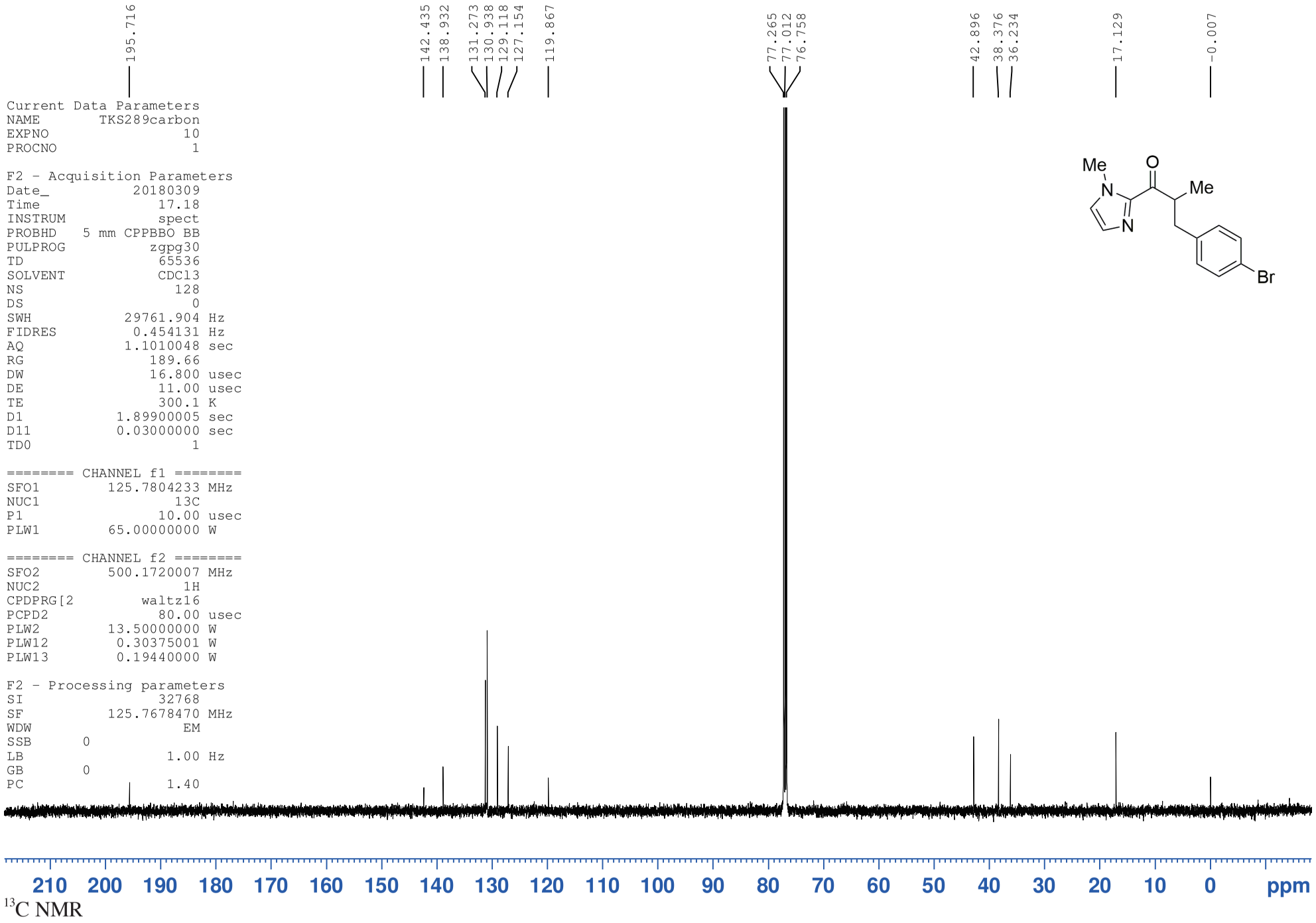
^{1}H NMR


196.408
142.584
138.051
136.577
130.188
129.803
129.047
127.068
126.128
125.622
77.297
77.043
76.789
41.632
36.210
36.131
19.654
17.158
0.006
Current Data Parameters
NAME kyk022 tm
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170613
Time 9.37
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

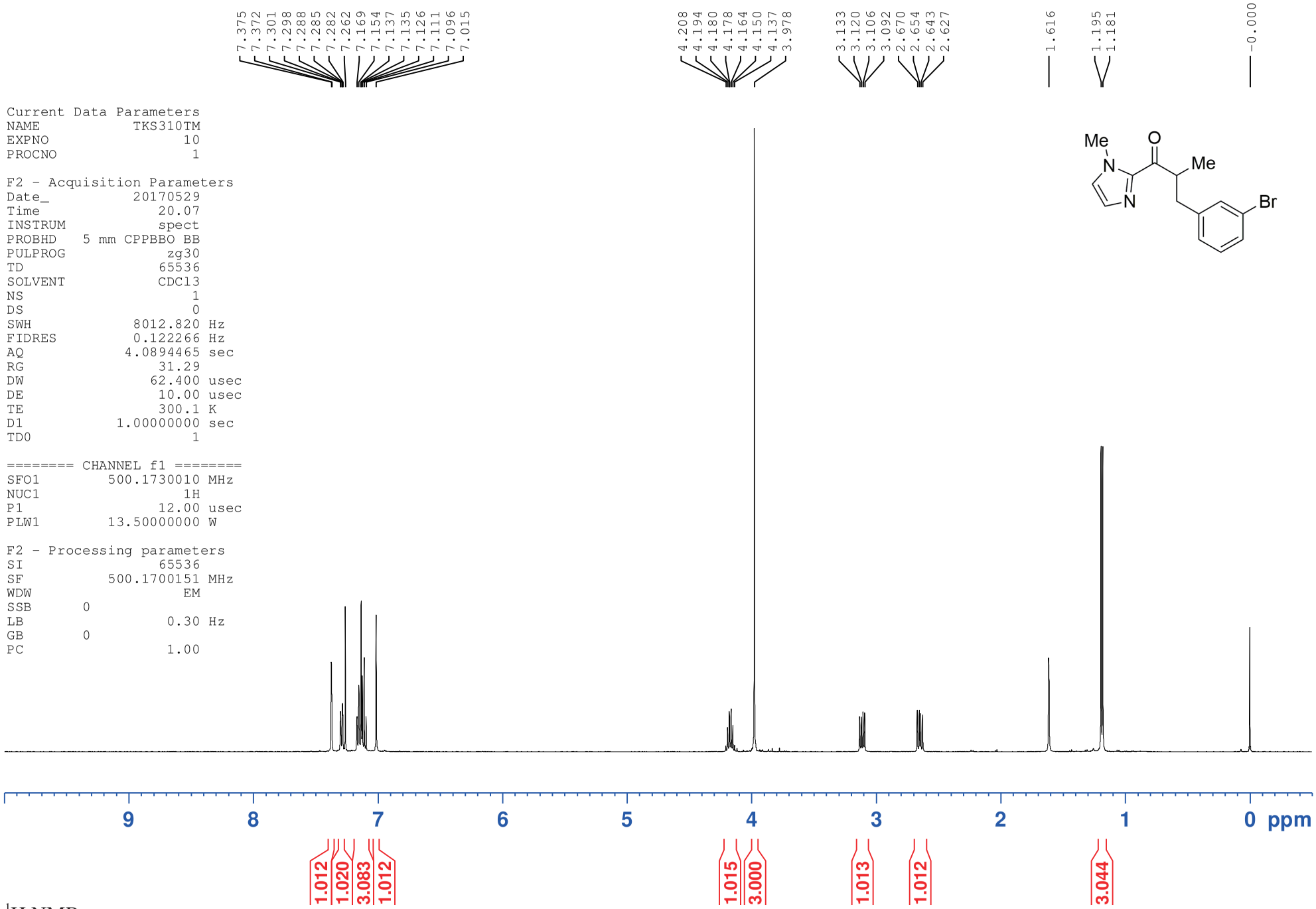
^{13}C NMR


7.366
7.350
7.263
7.131
7.113
7.096
7.011
4.210
4.196
4.182
4.167
4.153
4.139
3.969
3.117
3.103
3.090
3.076
2.656
2.641
2.629
2.614
1.190
1.176
-0.000
Current Data Parameters
NAME TKS289TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170410
Time 2.29
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
Br
9
8
7
6
5
4
3
2
1
0 ppm
2.041
0.984
2.036
1.009
1.025
3.000
1.009
1.011
3.032

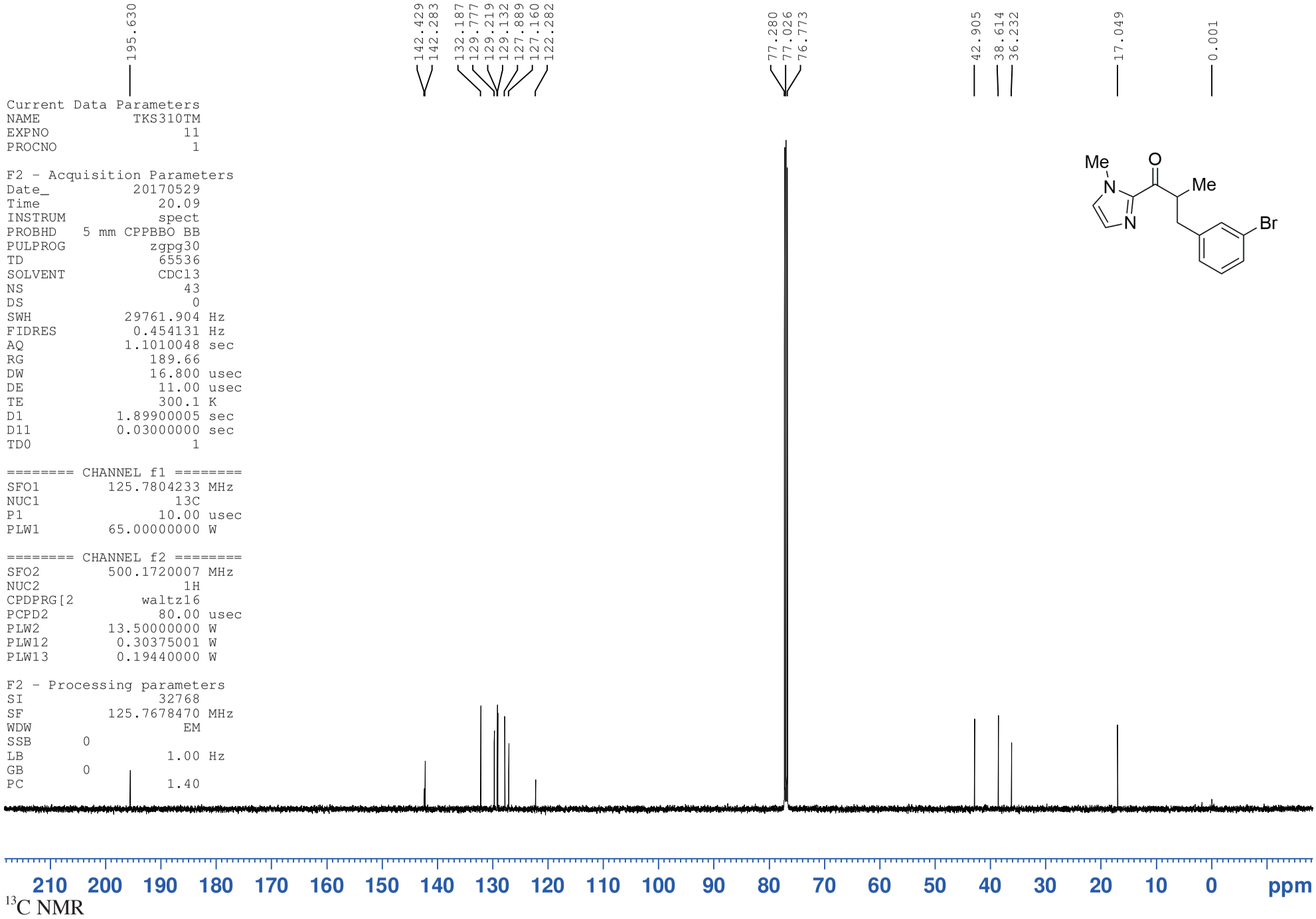
^{1}H NMR


195.716
142.435
138.932
131.273
130.938
129.118
127.154
119.867
77.265
77.012
76.758
42.896
38.376
36.234
17.129
-0.007
Current Data Parameters
NAME TKS289carbon
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180309
Time 17.18
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
Me
N
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

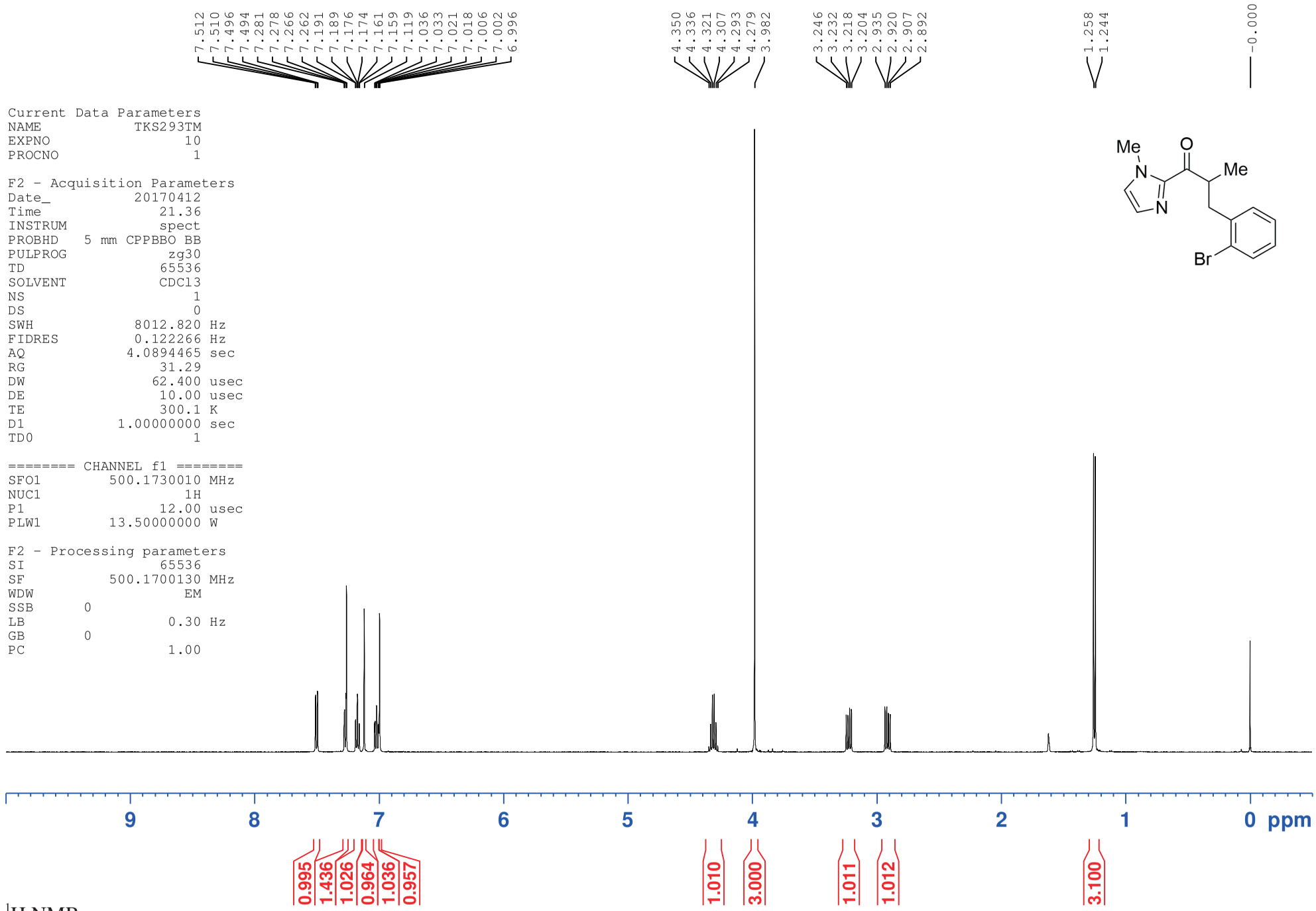
^{13}C NMR


7.375
7.372
7.301
7.298
7.288
7.285
7.282
7.262
7.169
7.154
7.137
7.135
7.126
7.111
7.096
7.015
4.208
4.194
4.180
4.178
4.164
4.150
4.137
3.978
3.133
3.120
3.106
3.092
2.670
2.654
2.643
2.627
1.616
1.195
1.181
-0.000
Current Data Parameters
NAME TKS310TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170529
Time 20.07
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
Me
N
Br
9
8
7
6
5
4
3
2
1
0 ppm
1.012
1.020
3.083
1.012
1.015
3.000
1.013
1.012
3.044

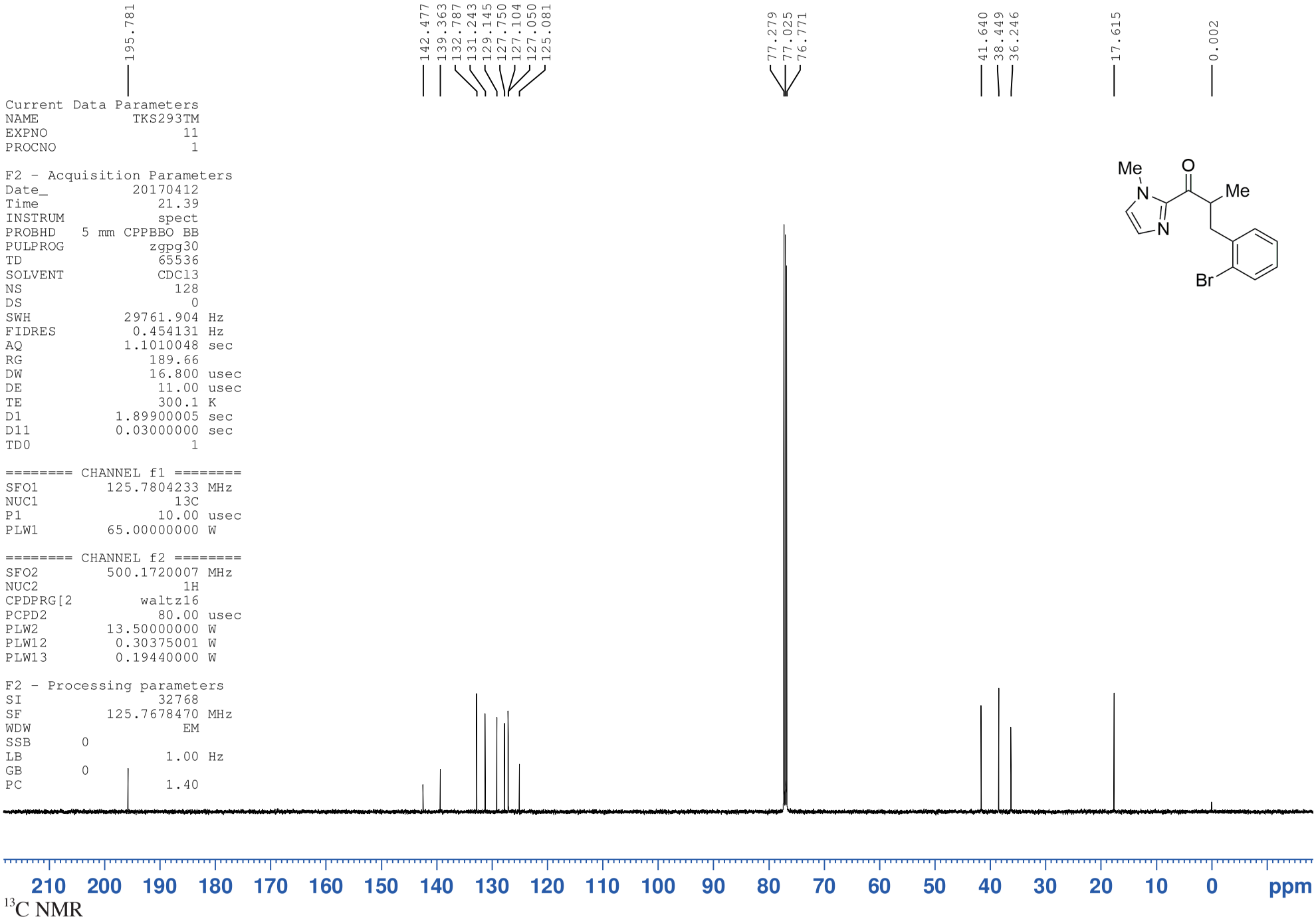
^{1}H NMR


195.630
142.429
142.283
132.187
129.777
129.219
129.132
127.889
127.160
122.282
77.280
77.026
76.773
42.905
38.614
36.232
17.049
0.001
Current Data Parameters
NAME TKS310TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170529
Time 20.09
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 43
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

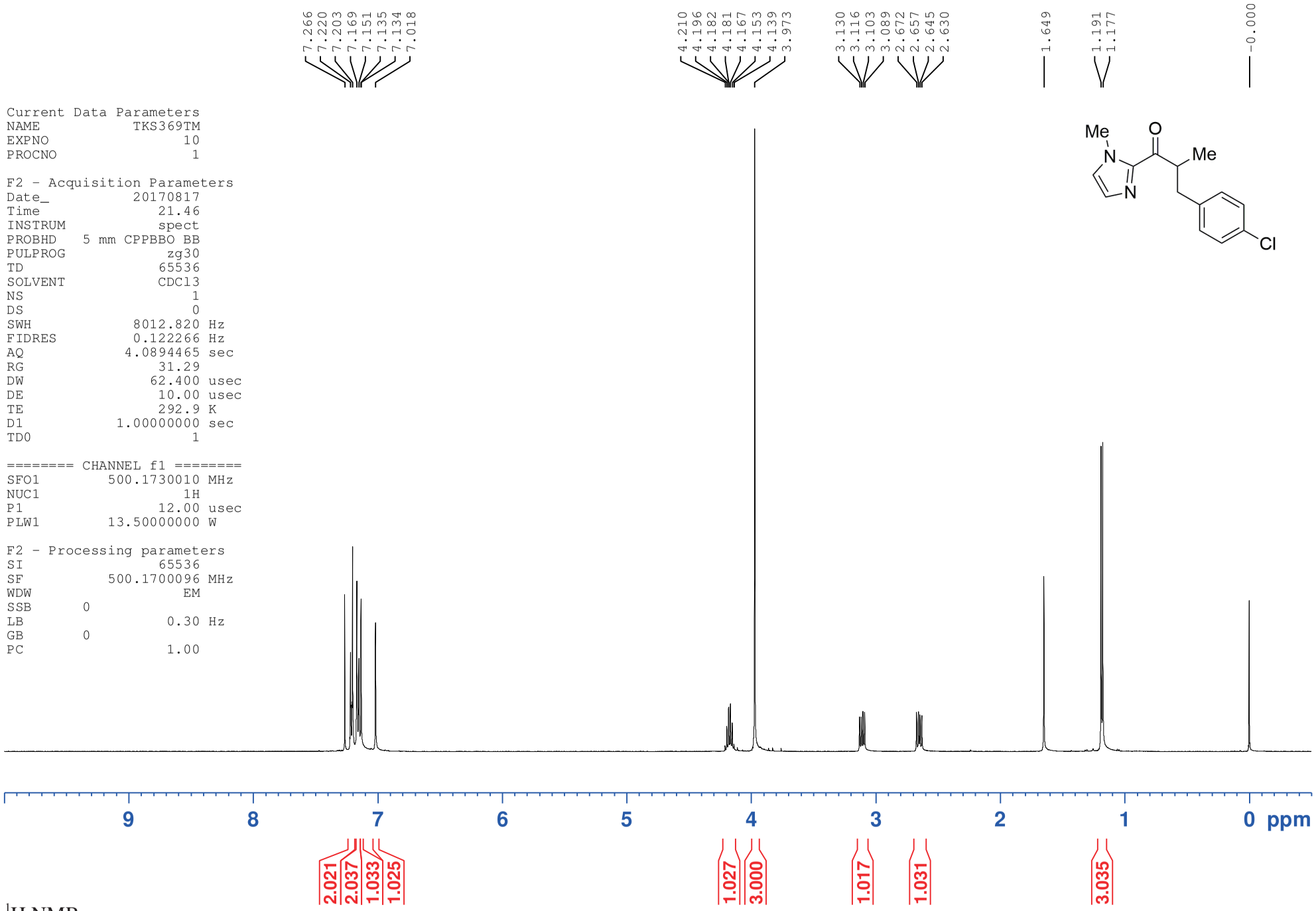
^{13}C NMR


7.512
7.510
7.496
7.494
7.281
7.278
7.266
7.262
7.191
7.189
7.176
7.174
7.161
7.159
7.119
7.036
7.033
7.021
7.018
7.006
7.002
6.996
4.350
4.336
4.321
4.307
4.293
4.279
3.982
3.246
3.232
3.218
3.204
2.935
2.920
2.907
2.892
1.258
1.244
-0.000
Current Data Parameters
NAME TKS293TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170412
Time 21.36
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
N
Me
N
Br
9
8
7
6
5
4
3
2
1
0 ppm
0.995
1.436
1.026
0.964
1.036
0.957
1.010
3.000
1.011
1.012
3.100

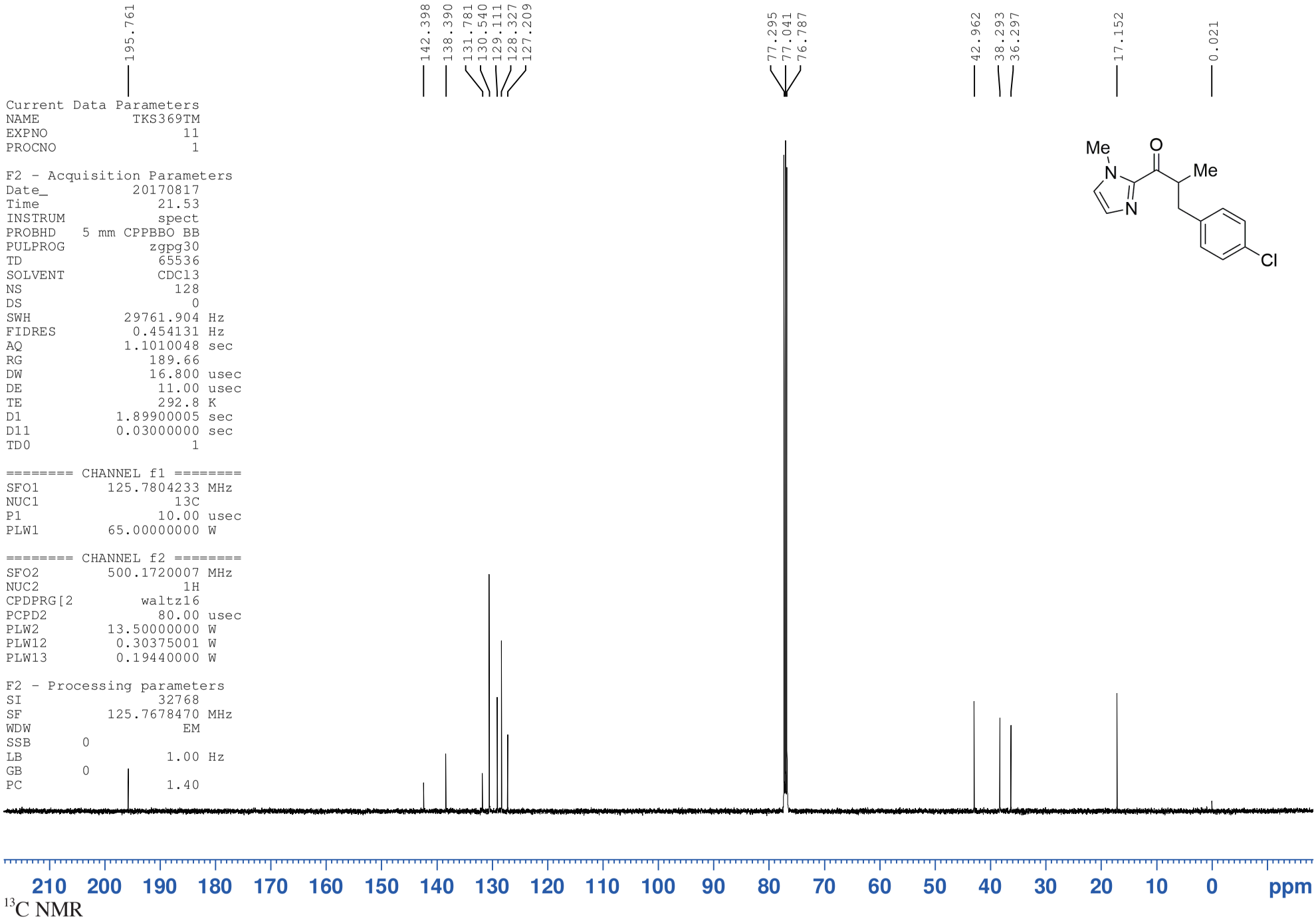
^{1}H NMR


195.781
142.477
139.363
132.787
131.243
129.145
127.750
127.104
127.050
125.081
77.279
77.025
76.771
41.640
38.449
36.246
17.615
0.002
Current Data Parameters
NAME TKS293TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170412
Time 21.39
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
O
N
Me
N
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

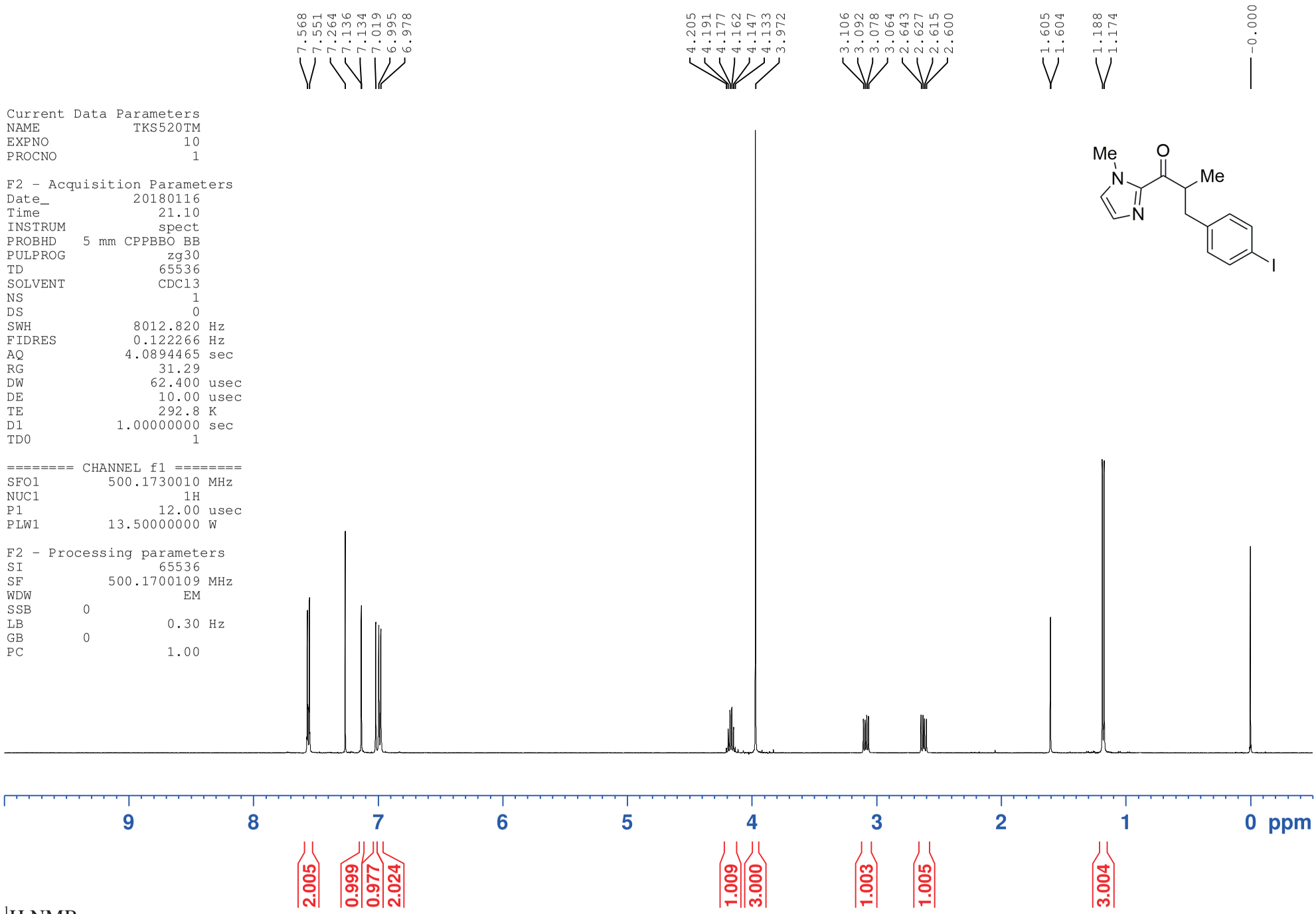
^{13}C NMR


7.266
7.220
7.203
7.169
7.151
7.135
7.134
7.018
4.210
4.196
4.182
4.181
4.167
4.153
4.139
3.973
3.130
3.116
3.103
3.089
2.672
2.657
2.645
2.630
1.649
1.191
1.177
0.000
Current Data Parameters
NAME TKS369TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170817
Time 21.46
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
Cl
9
8
7
6
5
4
3
2
1
0 ppm
2.021
2.037
1.033
1.025
1.027
3.000
1.017
1.031
3.035

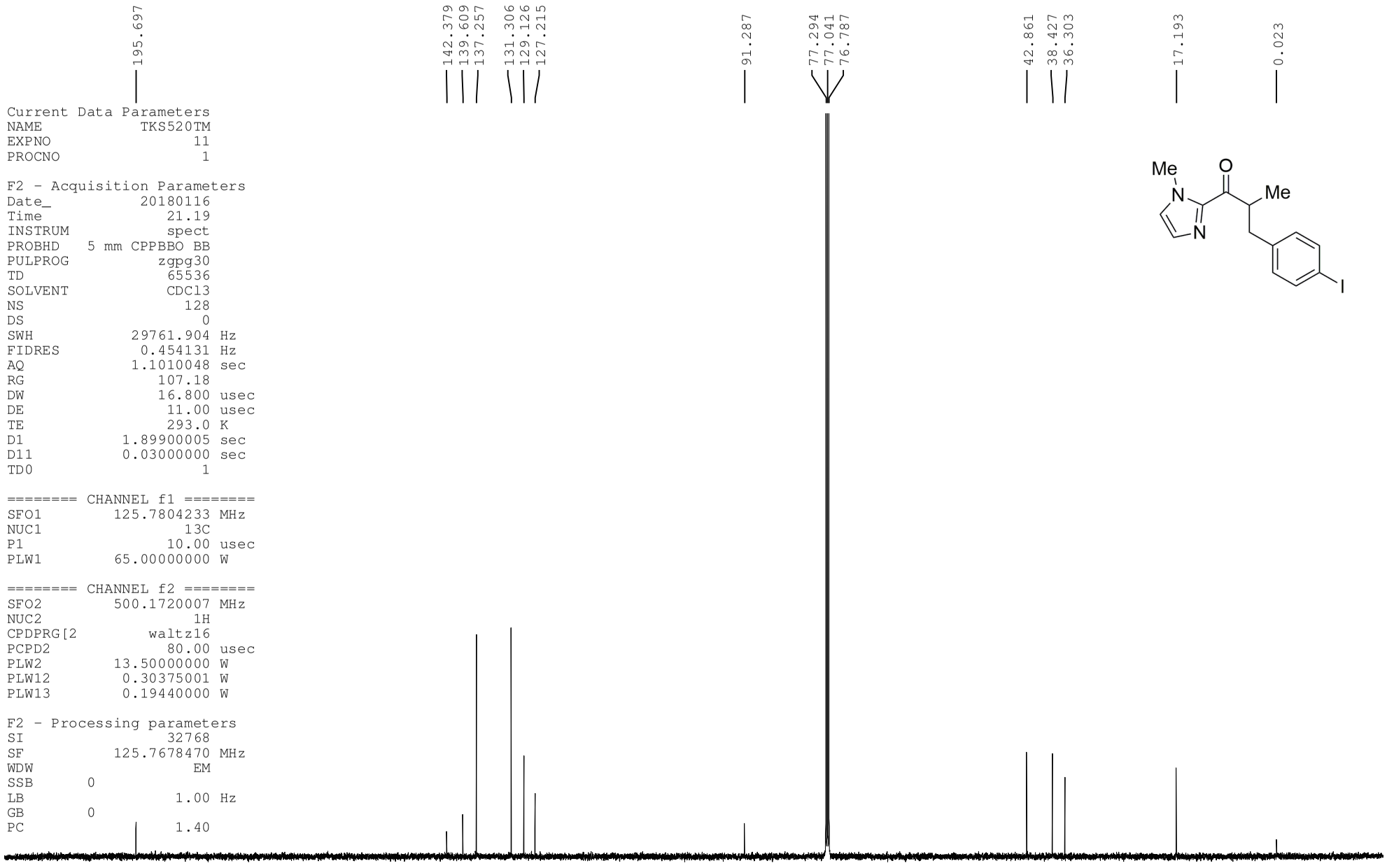
^{1}H NMR


195.761
142.398
138.390
131.781
130.540
129.111
128.327
127.209
77.295
77.041
76.787
42.962
38.293
36.297
17.152
0.021
Current Data Parameters
NAME TKS369TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170817
Time 21.53
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.8 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
Me
N
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

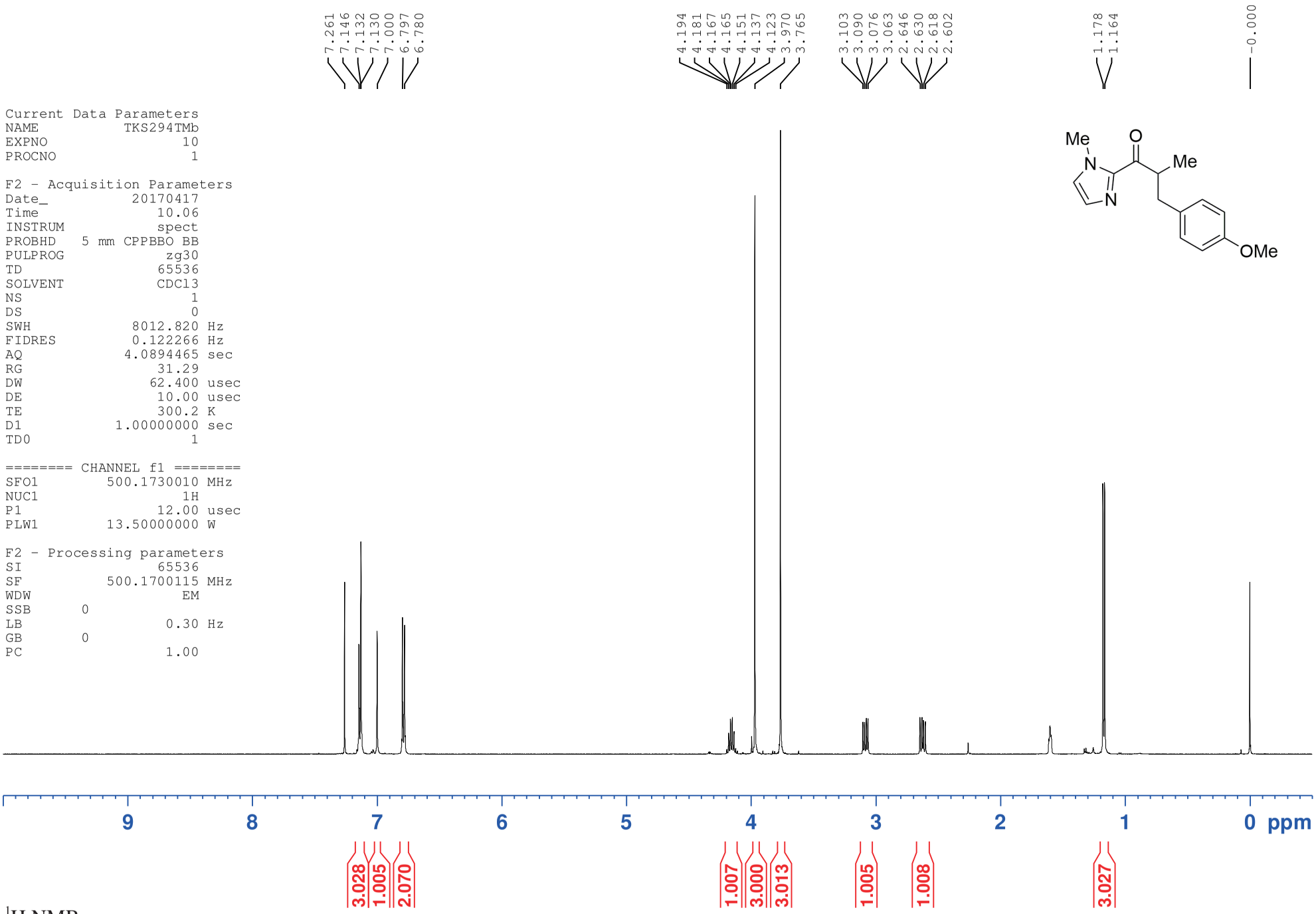
^{13}C NMR


7.568
7.551
7.264
7.136
7.134
7.019
6.995
6.978
4.205
4.191
4.177
4.162
4.147
4.133
3.972
3.106
3.092
3.078
3.064
2.643
2.627
2.615
2.600
1.605
1.604
1.188
1.174
-0.000
Current Data Parameters
NAME TKS520TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180116
Time 21.10
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700109 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
Me
N
I
9
8
7
6
5
4
3
2
1
0 ppm
2.005
0.999
0.977
2.024
1.009
3.000
1.003
1.005
3.004

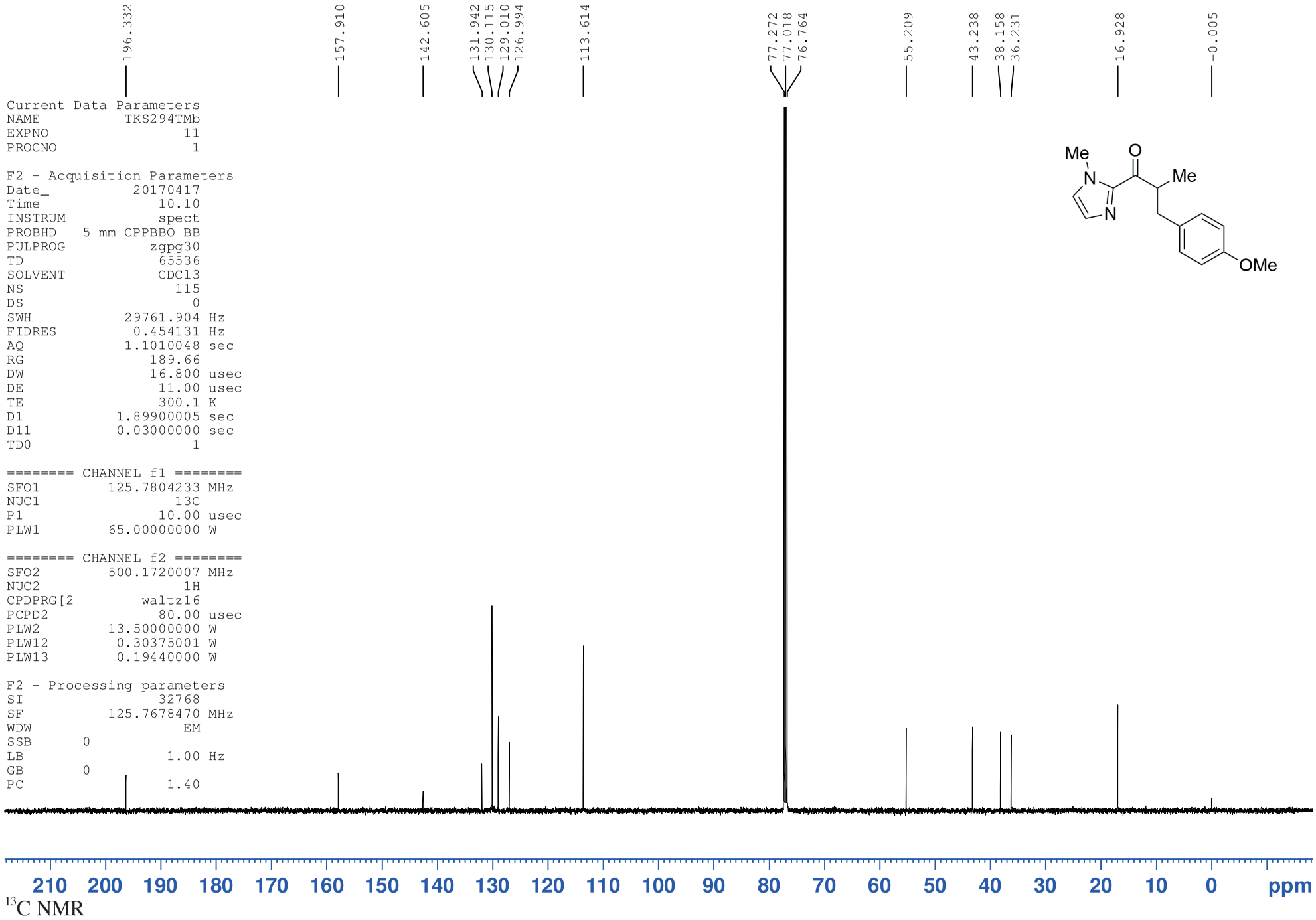
^{1}H NMR


195.697
142.379
139.609
137.257
131.306
129.126
127.215
91.287
77.294
77.041
76.787
42.861
38.427
36.303
17.193
0.023
Me
N
N
O
Me
I
Current Data Parameters
NAME TKS520TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180116
Time 21.19
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 293.0 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

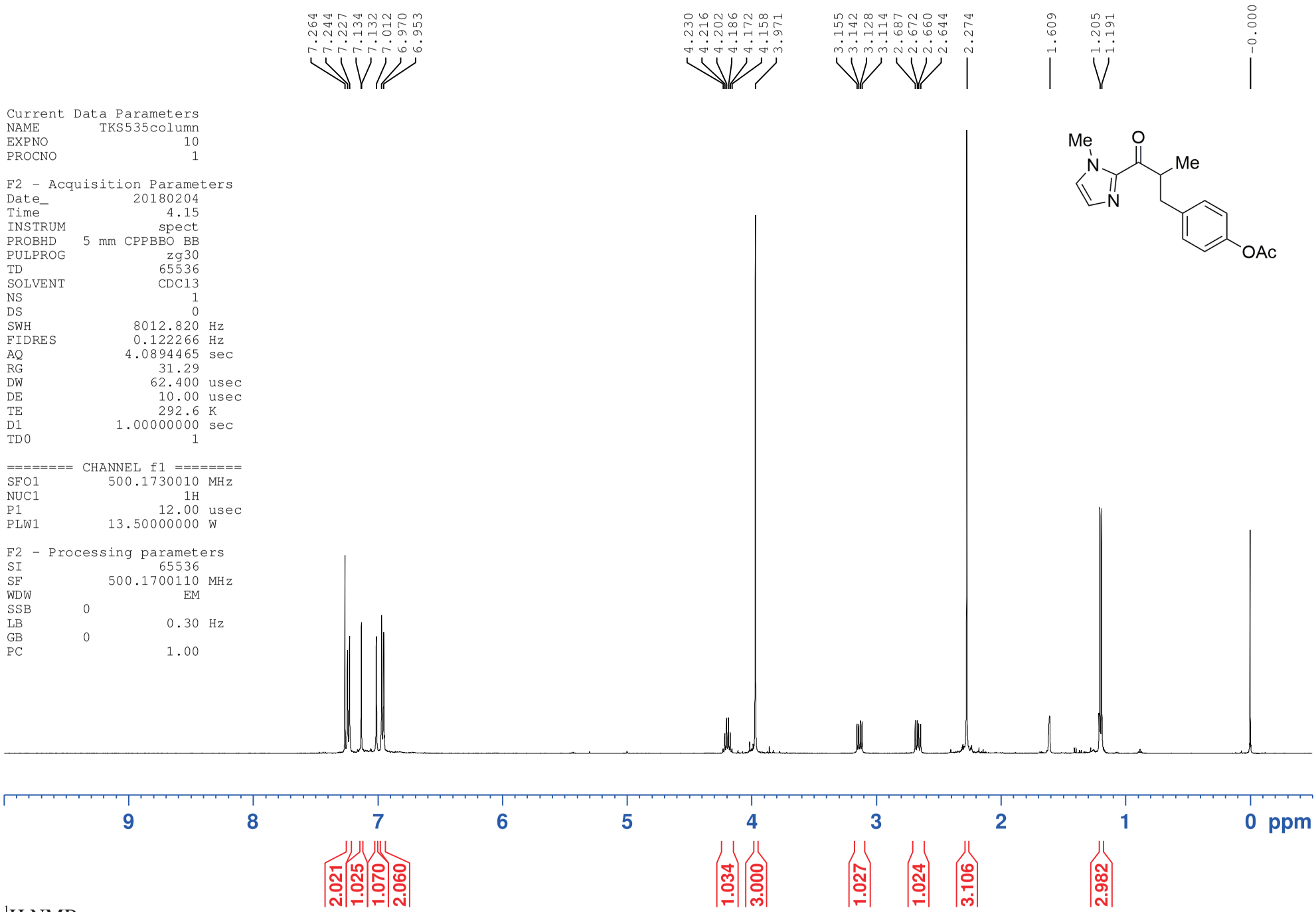
^{13}C NMR


7.261
7.146
7.132
7.130
7.000
6.797
6.780
4.194
4.181
4.167
4.165
4.151
4.137
4.123
3.970
3.765
3.103
3.090
3.076
3.063
2.646
2.630
2.618
2.602
1.178
1.164
-0.000
Current Data Parameters
NAME TKS294TMb
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170417
Time 10.06
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
OMe
9
8
7
6
5
4
3
2
1
0 ppm
3.028
1.005
2.070
1.007
3.000
3.013
1.005
1.008
3.027

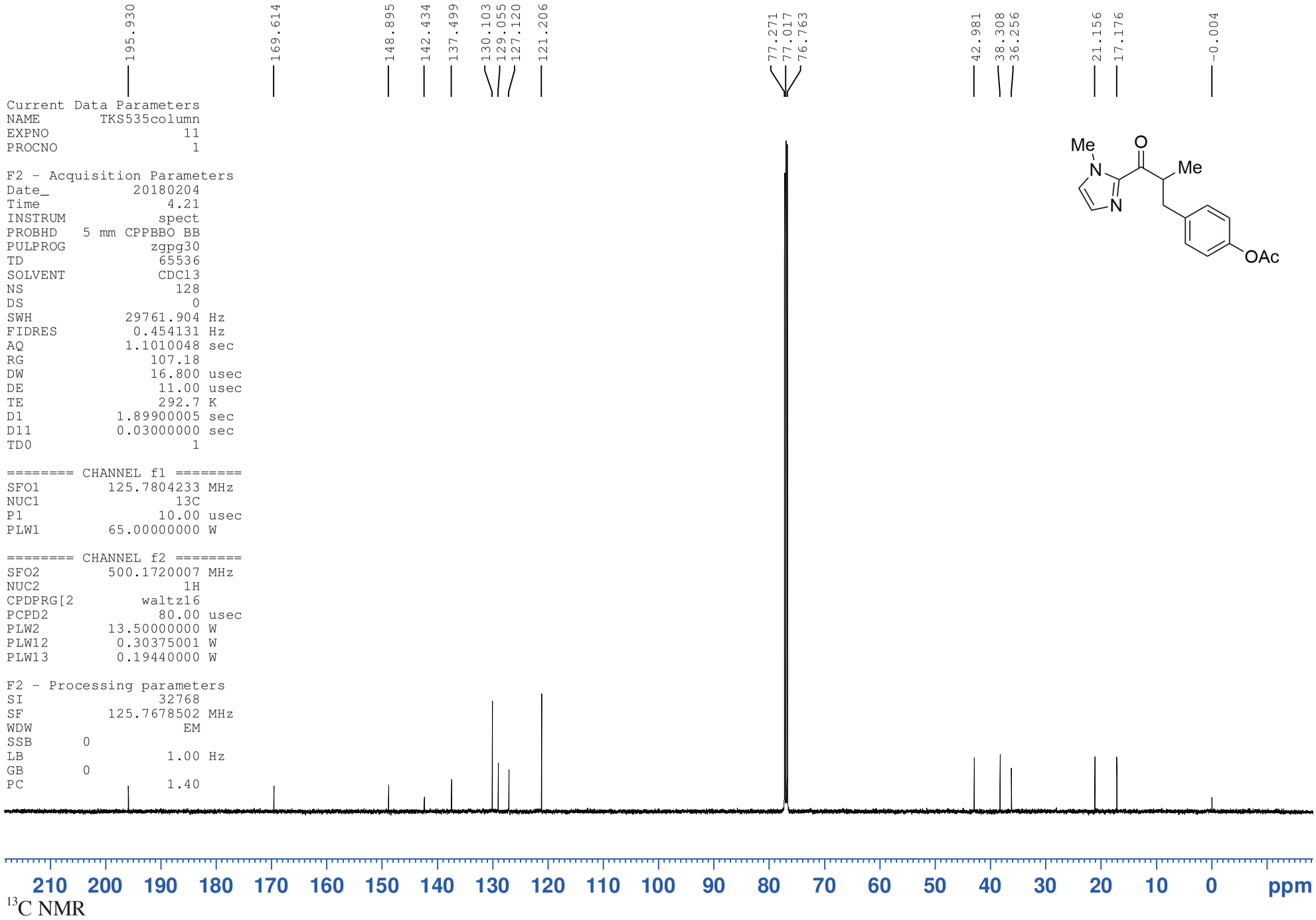
^{1}H NMR


196.332
157.910
142.605
131.942
130.115
129.010
126.994
113.614
77.272
77.018
76.764
55.209
43.238
38.158
36.231
16.928
-0.005
Current Data Parameters
NAME TKS294TMb
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170417
Time 10.10
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 115
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
Me
N
OMe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

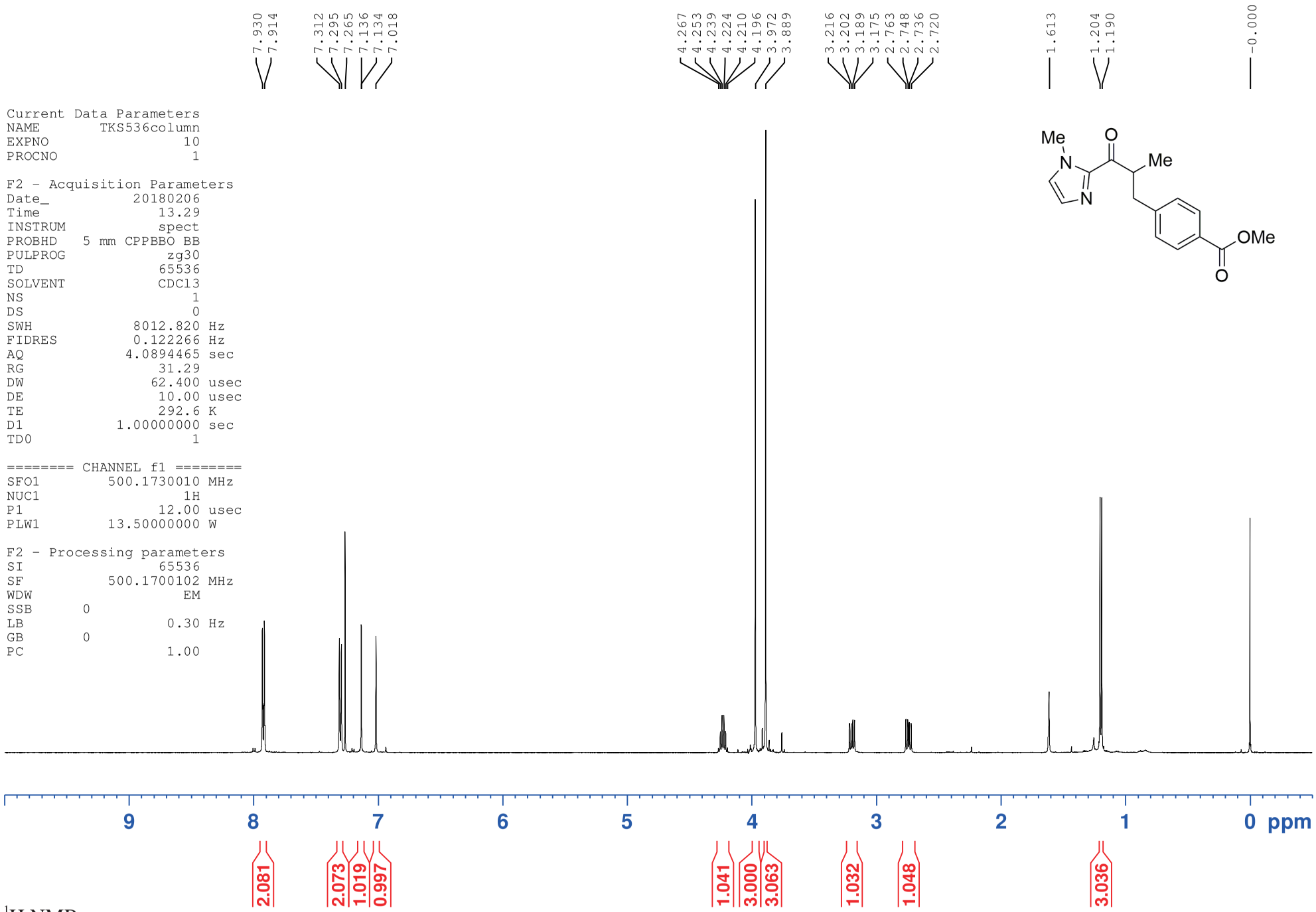
^{13}C NMR


7.264
7.244
7.227
7.134
7.132
7.012
6.970
6.953
4.230
4.216
4.202
4.186
4.172
4.158
3.971
3.155
3.142
3.128
3.114
2.687
2.672
2.660
2.644
2.274
1.609
1.205
1.191
-0.000
Current Data Parameters
NAME TKS535column
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180204
Time 4.15
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
OAc
9
8
7
6
5
4
3
2
1
0 ppm
2.021
1.025
1.070
2.060
1.034
3.000
1.027
1.024
3.106
2.982

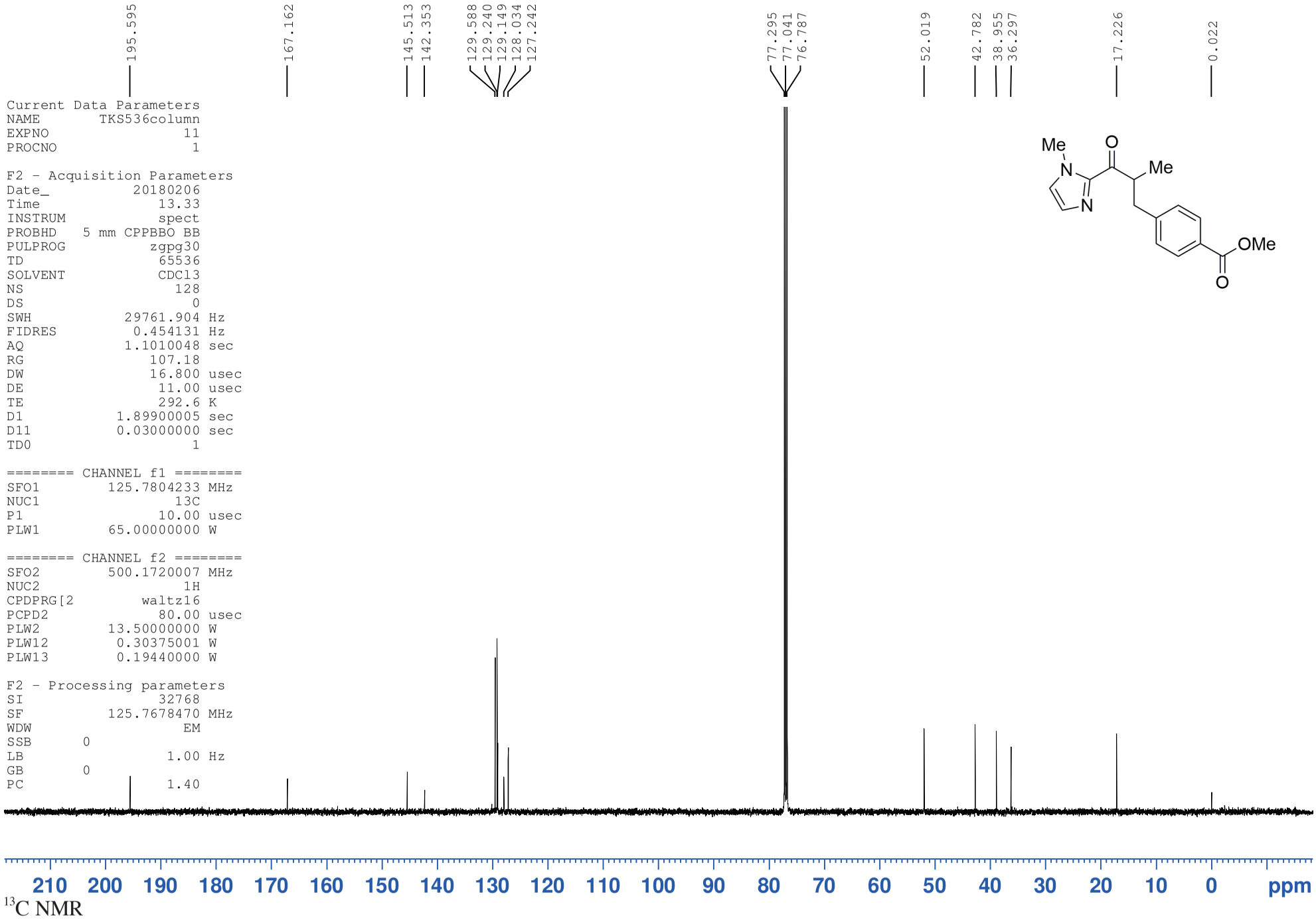
^{1}H NMR


195.930
169.614
148.895
142.434
137.499
130.103
129.055
127.120
121.206
77.271
77.017
76.763
42.981
38.308
36.256
21.156
17.176
-0.004
Current Data Parameters
NAME TKS535column
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180204
Time 4.21
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 292.7 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678502 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
OAc
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

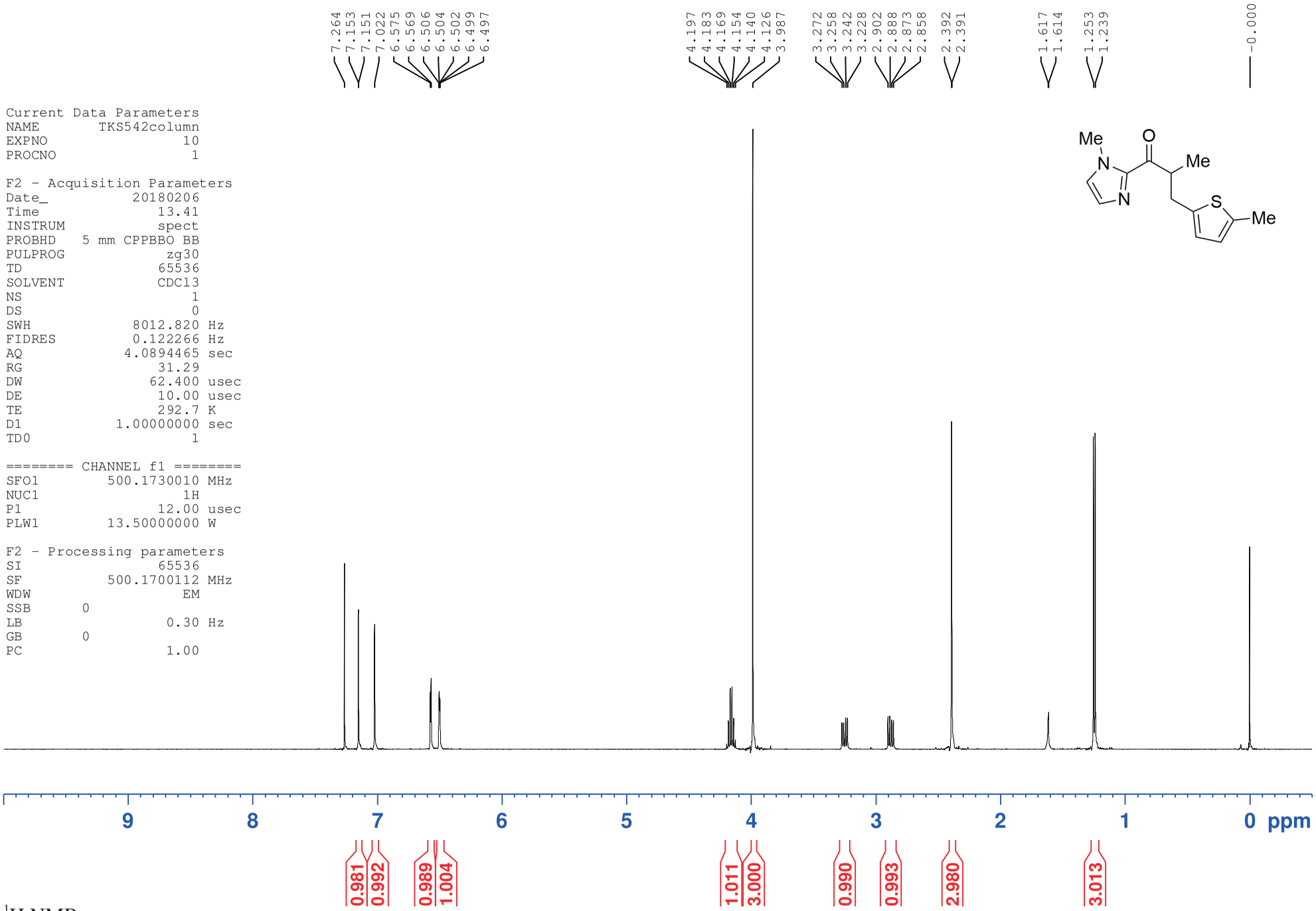
^{13}C NMR


7.930
7.914
7.312
7.295
7.265
7.136
7.134
7.018
4.267
4.253
4.239
4.224
4.210
4.196
3.972
3.889
3.216
3.202
3.189
3.175
2.763
2.748
2.736
2.720
1.613
1.204
1.190
-0.000
Current Data Parameters
NAME TKS536column
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180206
Time 13.29
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
OMe
O
9
8
7
6
5
4
3
2
1
0 ppm
2.081
2.073
1.019
0.997
1.041
3.000
3.063
1.032
1.048
3.036

^{1}H NMR

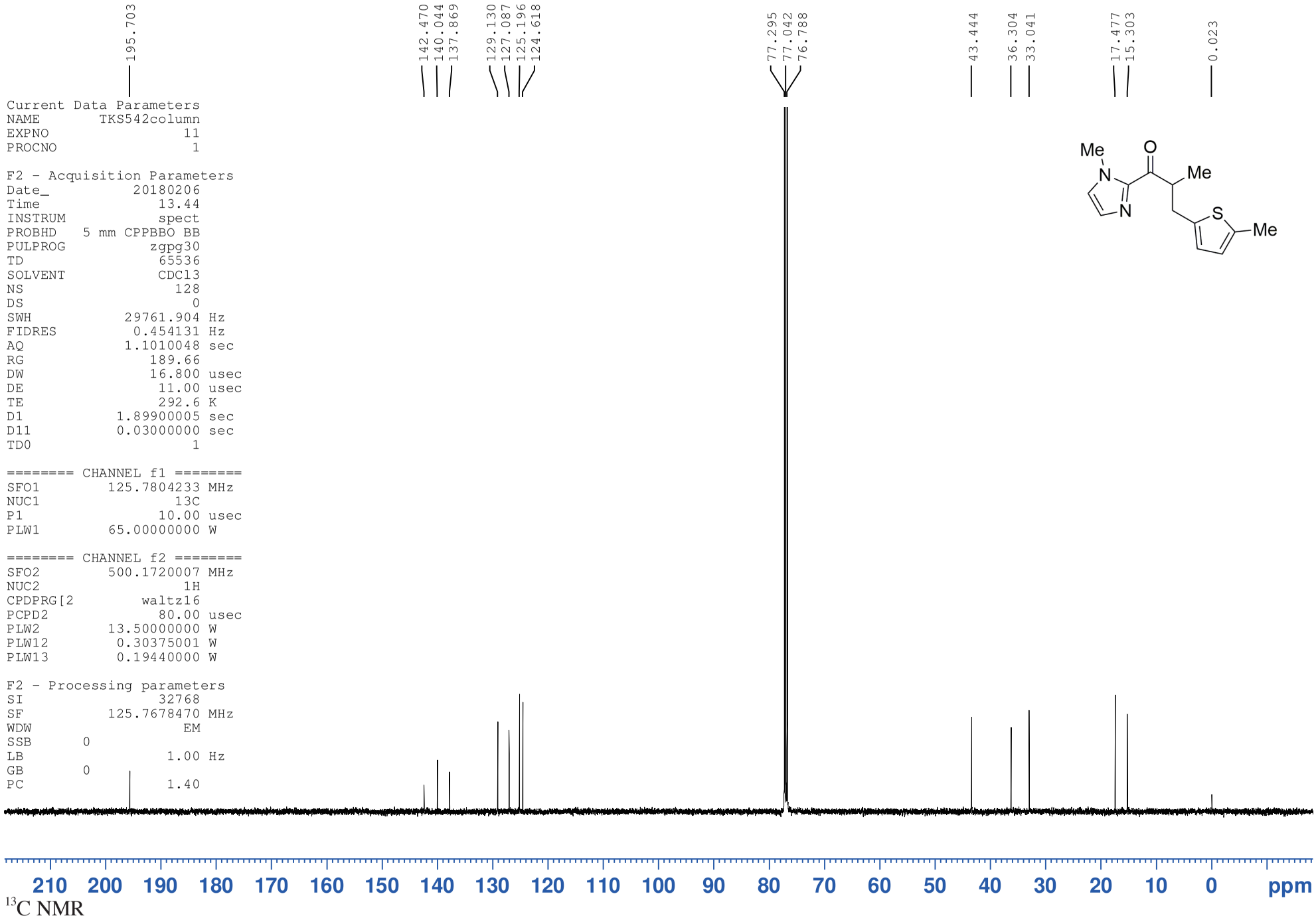

195.595
167.162
145.513
142.353
129.588
129.240
129.149
128.034
127.242
77.295
77.041
76.787
52.019
42.782
38.955
36.297
17.226
0.022
Current Data Parameters
NAME TKS536column
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180206
Time 13.33
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 292.6 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
OMe
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

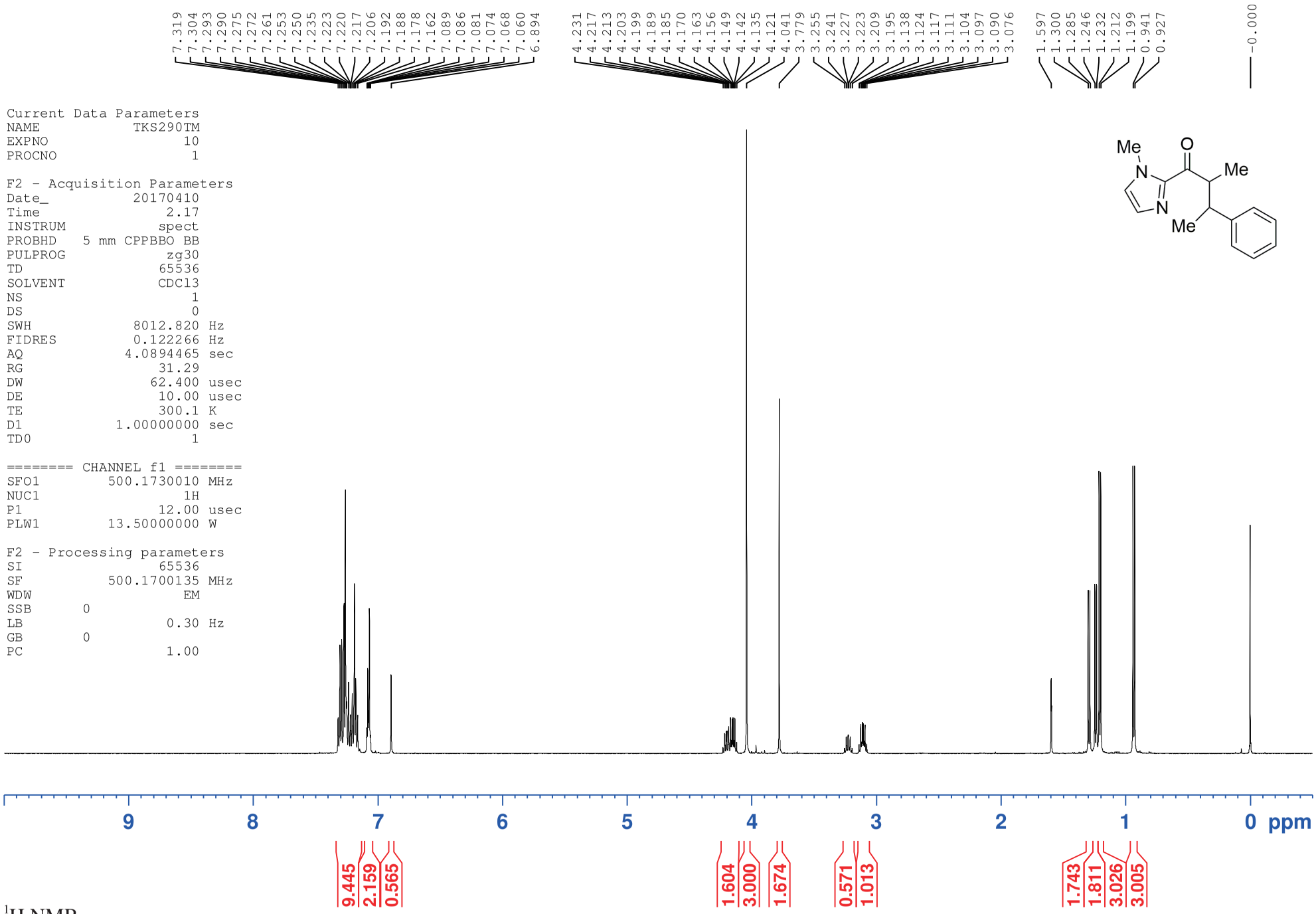

7.264
7.153
7.151
7.022
6.575
6.569
6.506
6.504
6.502
6.499
6.497
4.197
4.183
4.169
4.154
4.140
4.126
3.987
3.272
3.258
3.242
3.228
2.902
2.888
2.873
2.858
2.392
2.391
1.617
1.614
1.253
1.239
0.000
Current Data Parameters
NAME TKS542column
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180206
Time 13.41
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
Me
N
S
Me
9
8
7
6
5
4
3
2
1
0 ppm
0.981
0.992
0.989
1.004
1.011
3.000
0.990
0.993
2.980
3.013

S120

^{1}H NMR

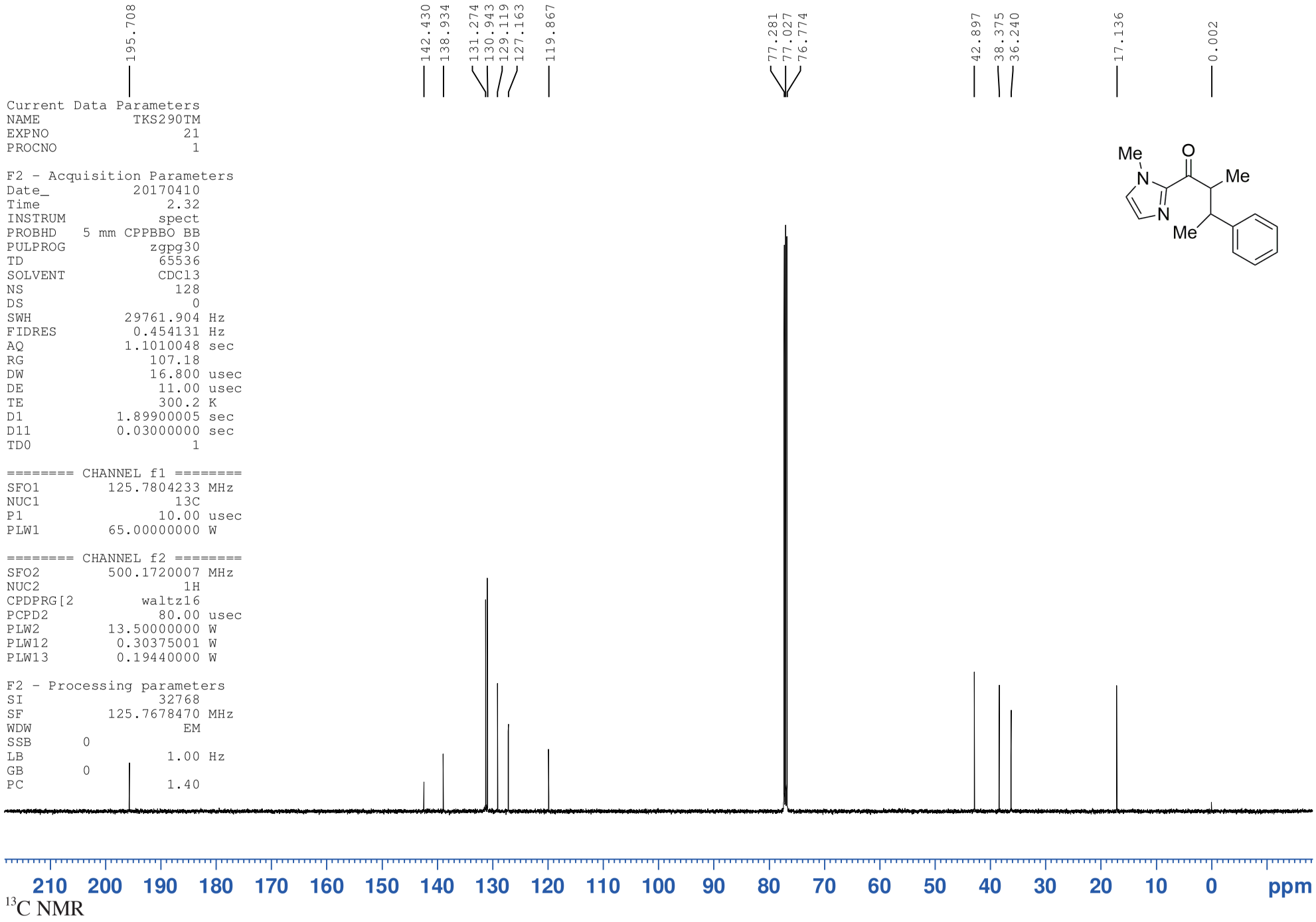

195.703
142.470
140.044
137.869
129.130
127.087
125.196
124.618
77.295
77.042
76.788
43.444
36.304
33.041
17.477
15.303
0.023
Current Data Parameters
NAME TKS542column
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180206
Time 13.44
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.6 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
O
N
Me
N
S
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

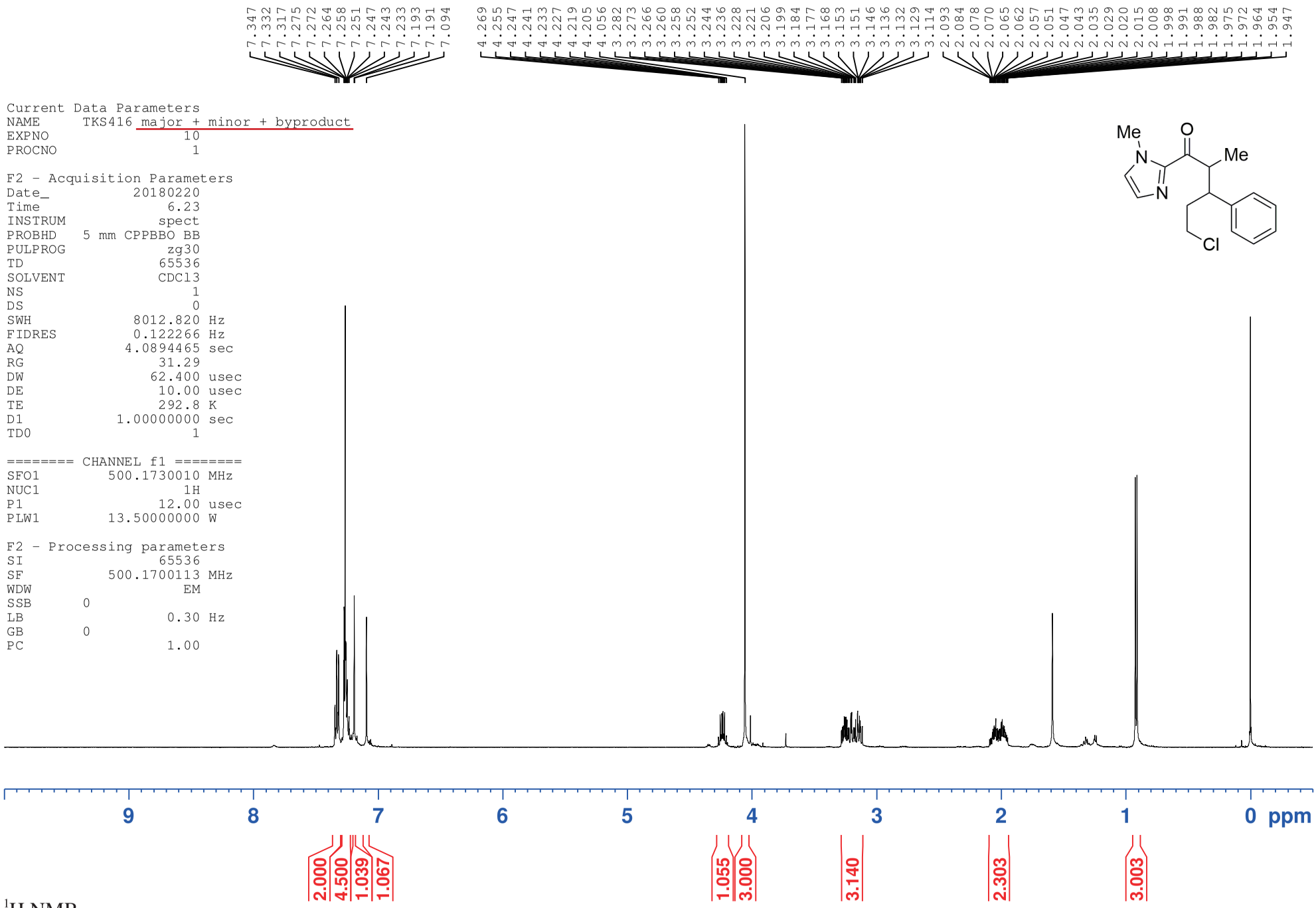

7.319
7.304
7.293
7.290
7.275
7.272
7.261
7.253
7.250
7.235
7.223
7.220
7.217
7.206
7.192
7.188
7.178
7.162
7.089
7.086
7.081
7.074
7.068
7.060
6.894
4.231
4.217
4.213
4.203
4.199
4.189
4.185
4.170
4.163
4.156
4.149
4.142
4.135
4.121
4.041
3.779
3.255
3.241
3.227
3.223
3.209
3.195
3.138
3.124
3.117
3.111
3.104
3.097
3.090
3.076
1.597
1.300
1.285
1.246
1.232
1.212
1.199
0.941
0.927
-0.000
Current Data Parameters
NAME TKS290TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170410
Time 2.17
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
N
Me
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
9.445
2.159
0.565
1.604
3.000
1.674
0.571
1.013
1.743
1.811
3.026
3.005

S122

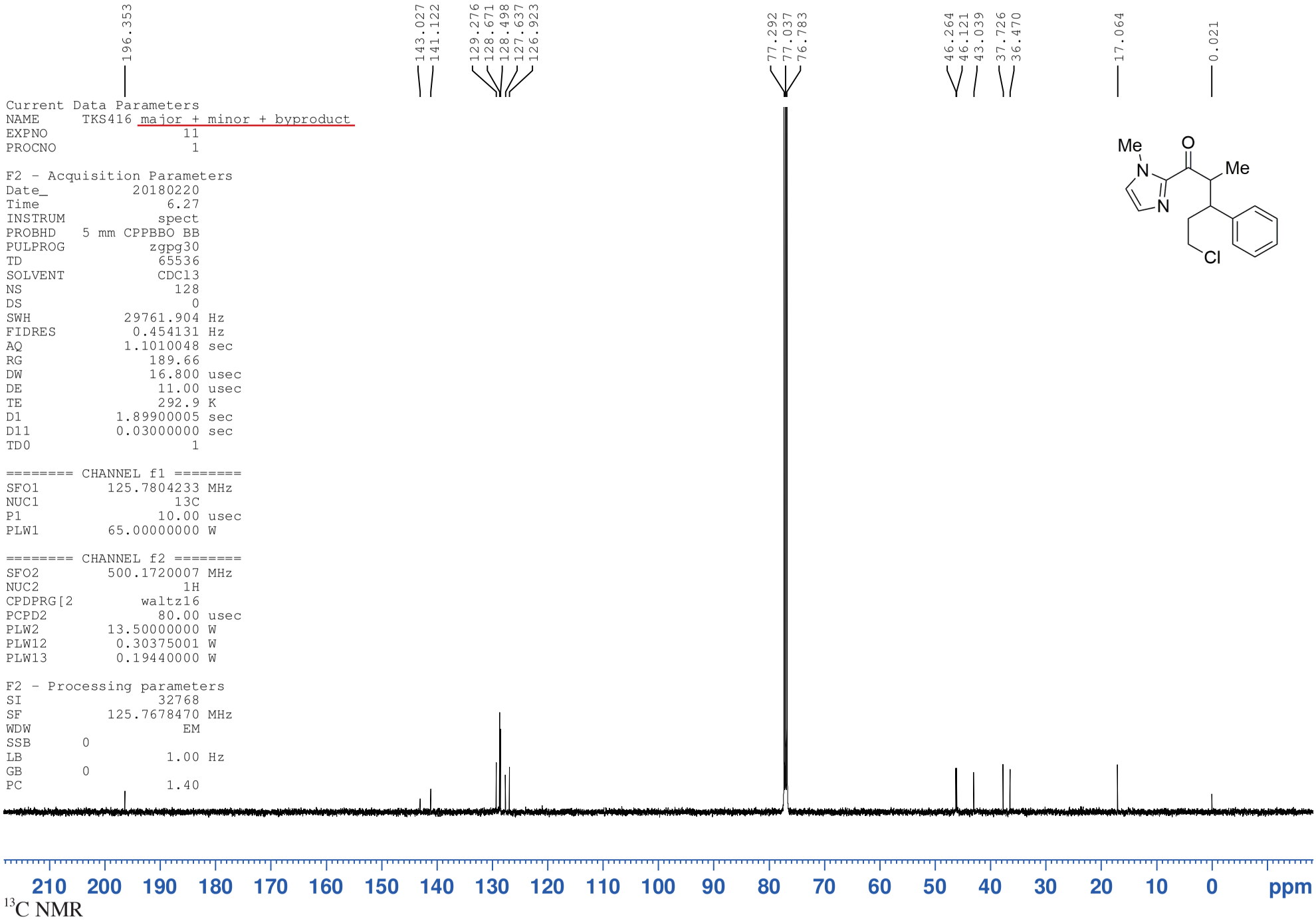
^{1}H NMR


195.708
142.430
138.934
131.274
130.943
129.119
127.163
119.867
77.281
77.027
76.774
42.897
38.375
36.240
17.136
0.002
Current Data Parameters
NAME TKS290TM
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170410
Time 2.32
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
Me
N
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

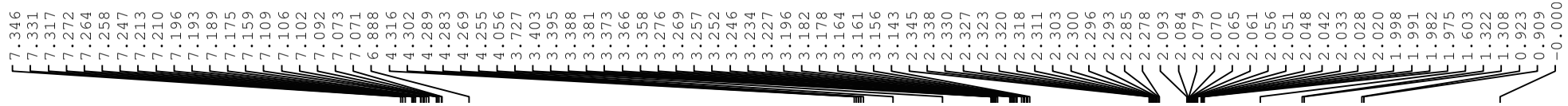
^{13}C NMR


7.347 7.332 7.317 7.275 7.272 7.264 7.258 7.251 7.247 7.243 7.233 7.193 7.191 7.094
4.269 4.255 4.247 4.241 4.233 4.227 4.219 4.205 4.056 3.282 3.273 3.266 3.260 3.258 3.252 3.244 3.236 3.228 3.221 3.206 3.199 3.184 3.177 3.168 3.153 3.151 3.146 3.136 3.132 3.129 3.114
2.093 2.084 2.078 2.070 2.065 2.062 2.057 2.051 2.047 2.043 2.035 2.029 2.020 2.015 2.008 1.998 1.991 1.988 1.982 1.975 1.972 1.964 1.954 1.947
Current Data Parameters
NAME TKS416 major + minor + byproduct
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180220
Time 6.23
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
Cl
9 8 7 6 5 4 3 2 1 0 ppm
2.000 4.500 1.039 1.067 1.055 3.000 3.140 2.303 3.003

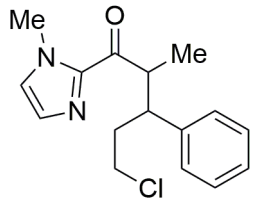
^{1}H NMR


196.353
143.027
141.122
129.276
128.671
128.498
127.637
126.923
77.292
77.037
76.783
46.264
46.121
43.039
37.726
36.470
17.064
0.021
Current Data Parameters
NAME TKS416 major + minor + byproduct
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180220
Time 6.27
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.9 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

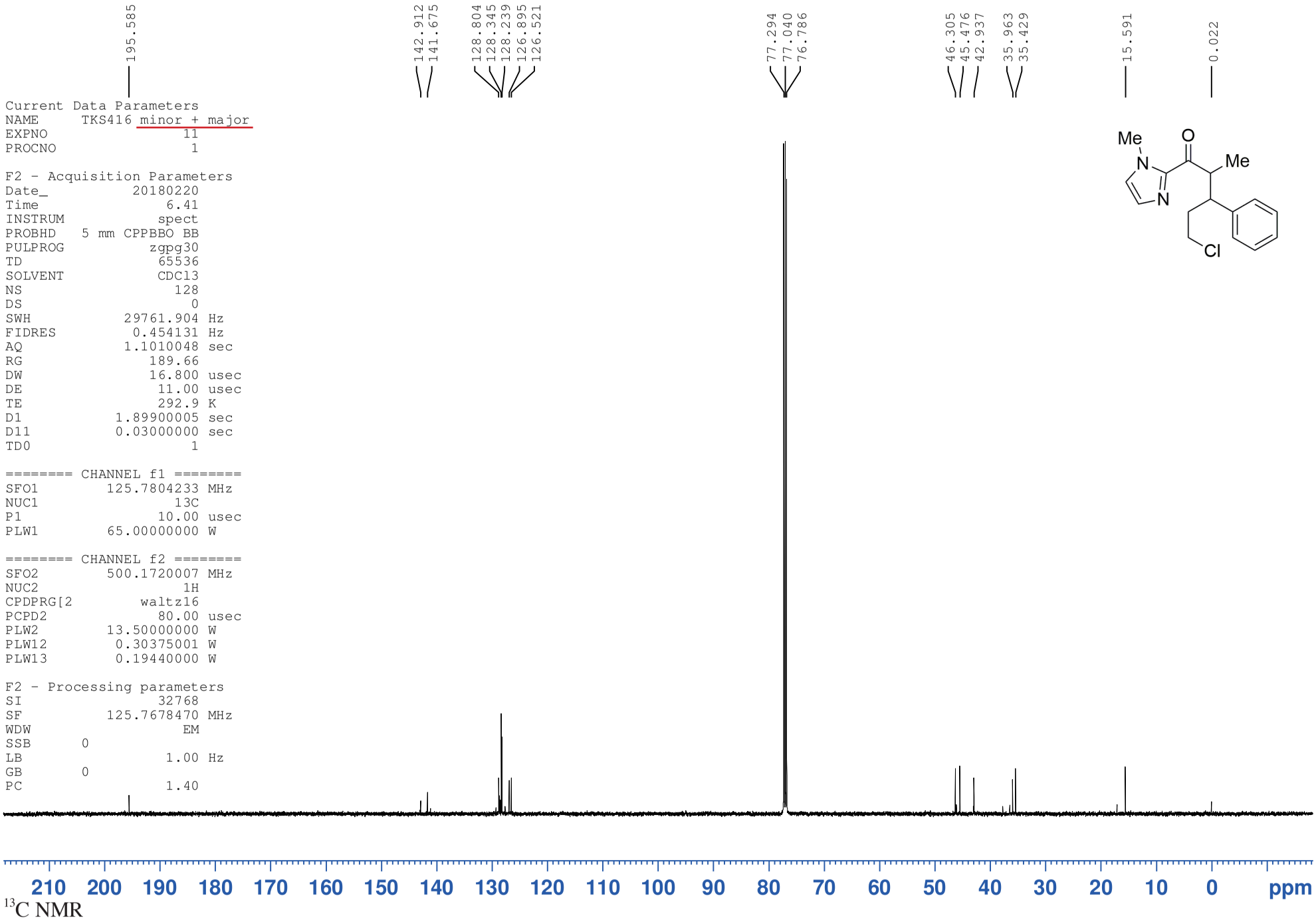
^{13}C NMR



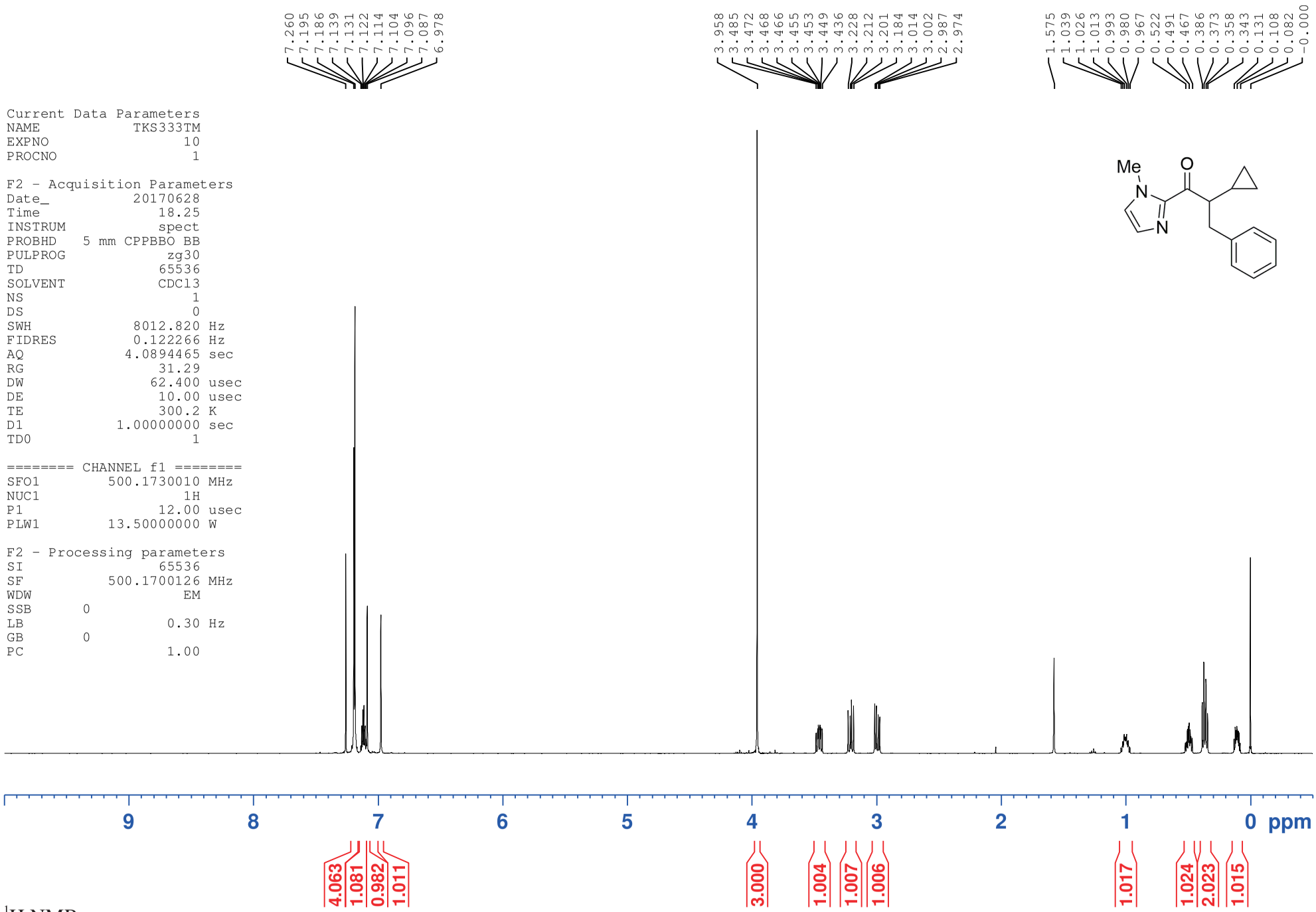

```
Current Data Parameters
NAME      TKS416 minor + major
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180220
Time                6.34
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     1
DS                     0
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  31.29
DW                 62.400 usec
DE                 10.00 usec
TE                 292.8 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1730010 MHz
NUC1                1H
P1                 12.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 65536
SF           500.1700110 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



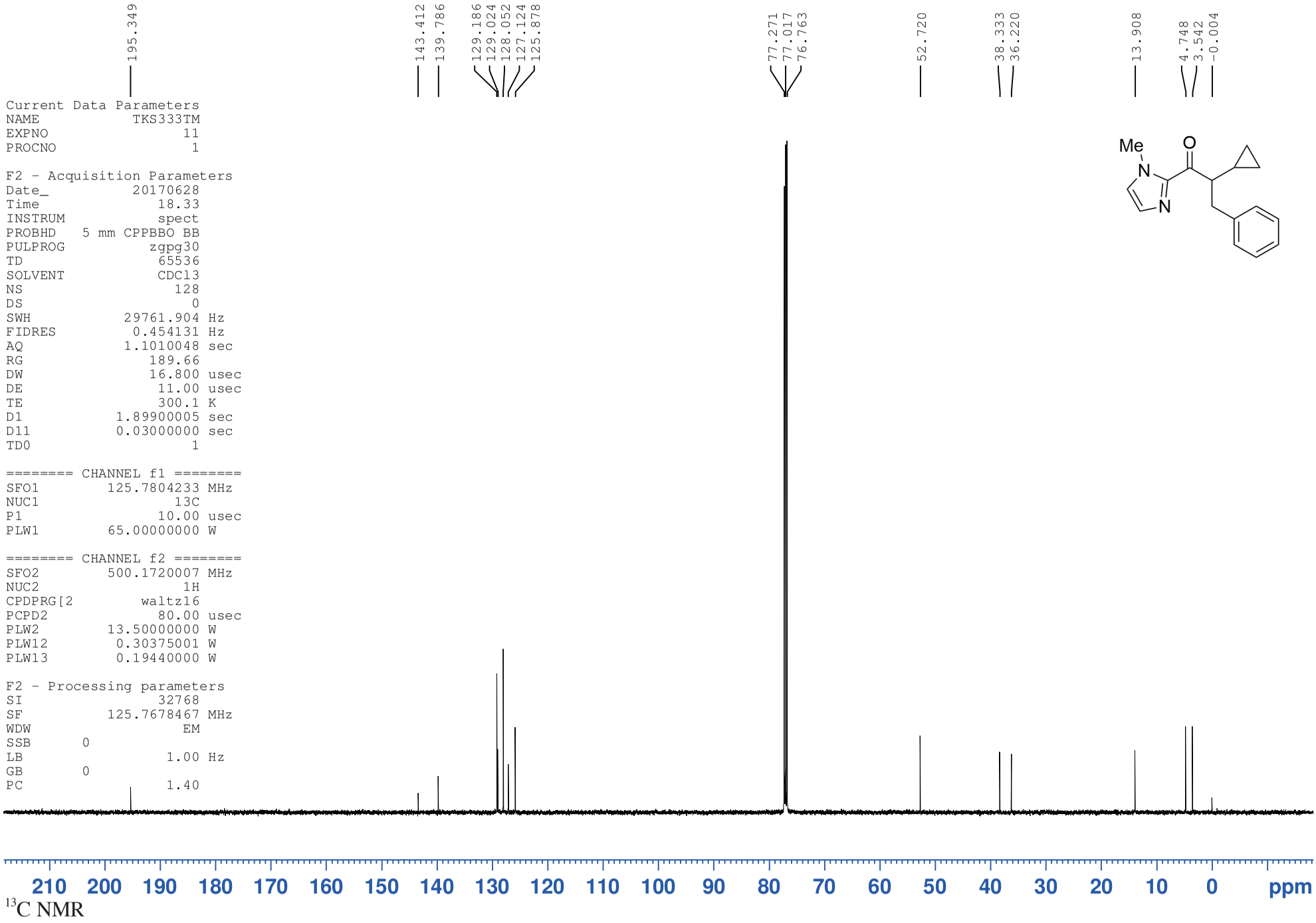
^{1}H NMR


195.585
142.912
141.675
128.804
128.345
128.239
126.895
126.521
77.294
77.040
76.786
46.305
45.476
42.937
35.963
35.429
15.591
0.022
Current Data Parameters
NAME TKS416 minor + major
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180220
Time 6.41
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.9 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
Me
N
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

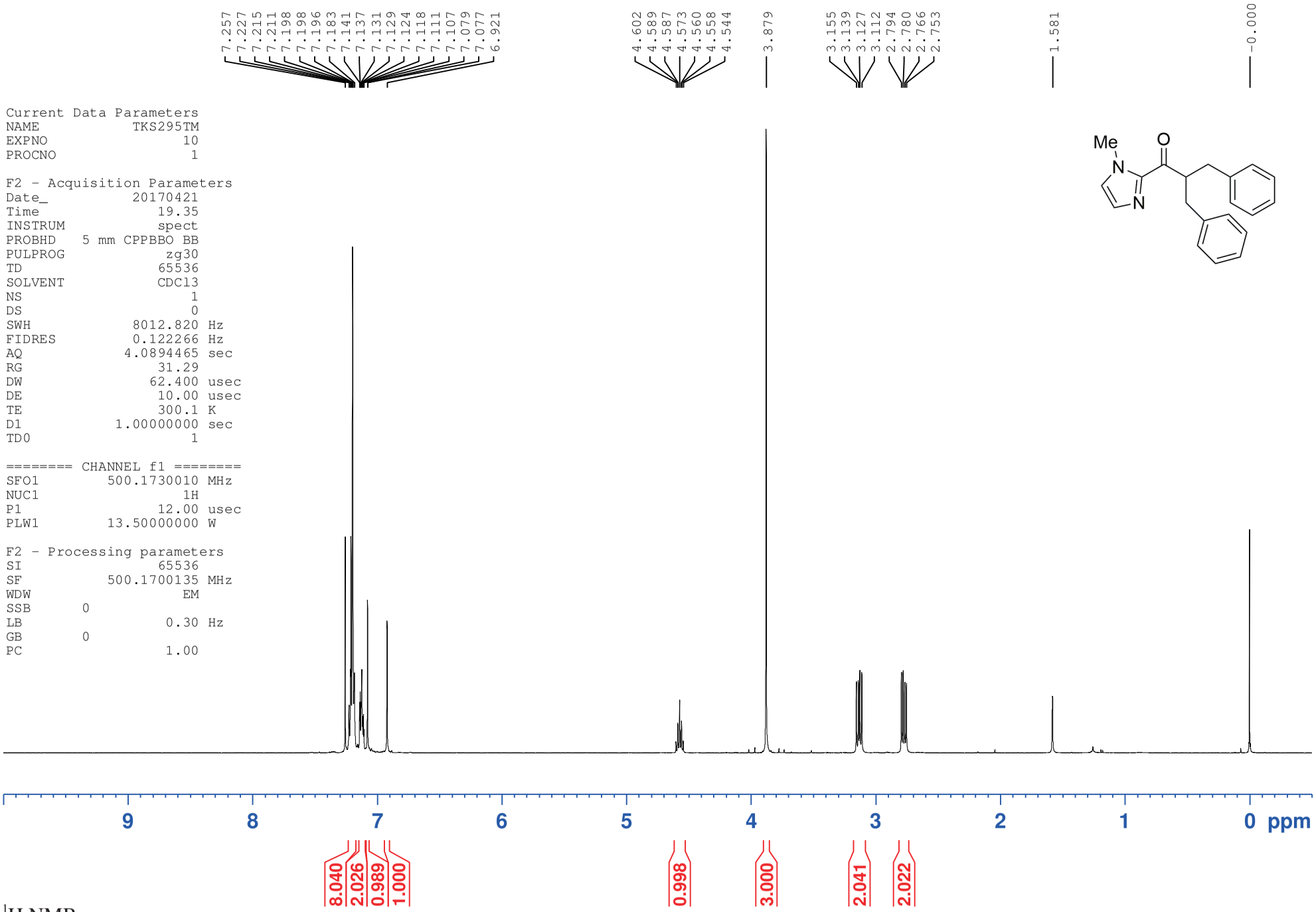
^{13}C NMR


7.260
7.195
7.186
7.139
7.131
7.122
7.114
7.104
7.096
7.087
6.978
3.958
3.485
3.472
3.468
3.466
3.455
3.453
3.449
3.436
3.228
3.212
3.201
3.184
3.014
3.002
2.987
2.974
1.575
1.039
1.026
1.013
0.993
0.980
0.967
0.522
0.491
0.467
0.386
0.373
0.358
0.343
0.131
0.108
0.082
-0.000
Me
N
N
O
Current Data Parameters
NAME TKS333TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170628
Time 18.25
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
4.063
1.081
0.982
1.011
3.000
1.004
1.007
1.006
1.017
1.024
2.023
1.015

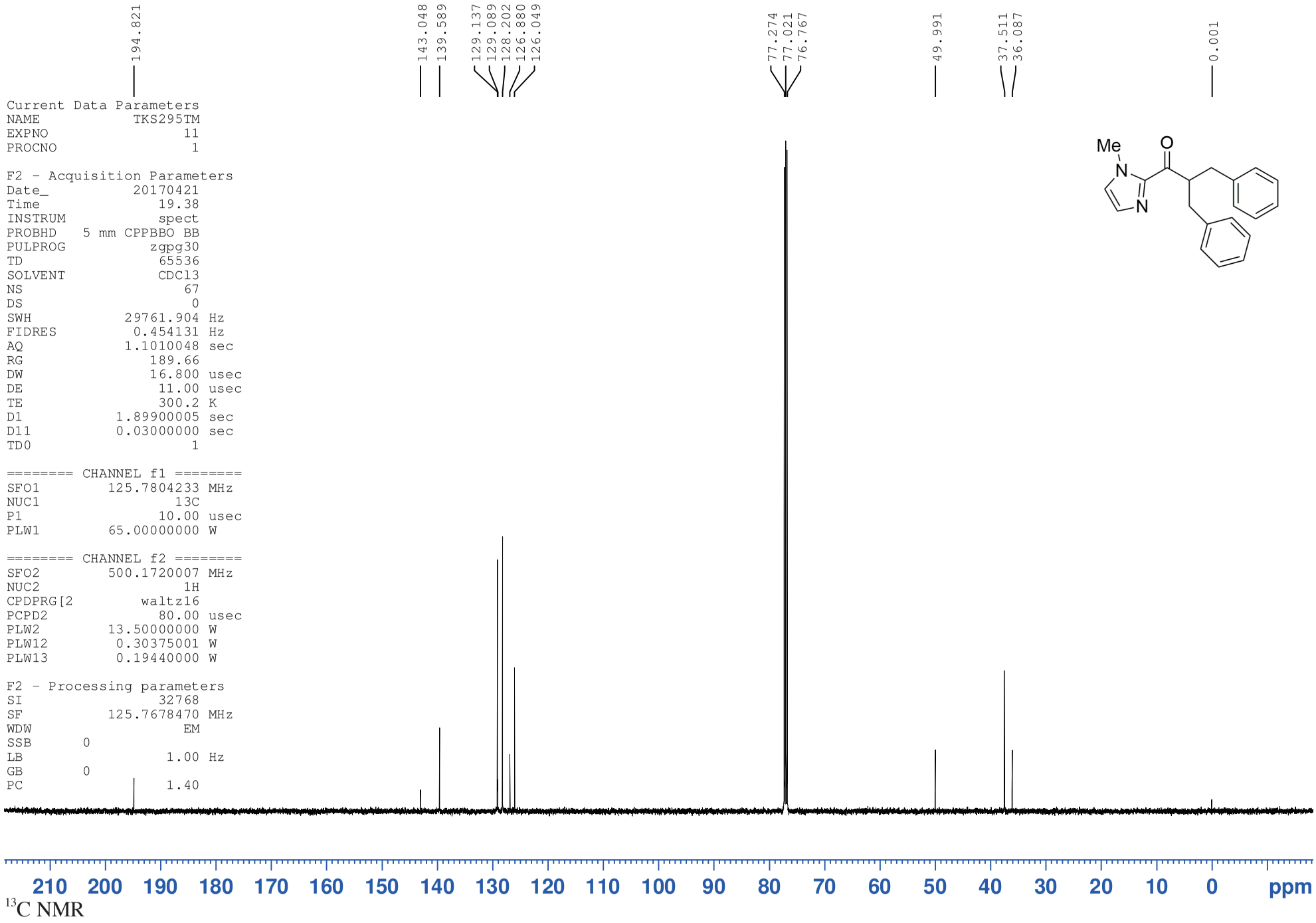
^{1}H NMR


195.349
143.412
139.786
129.186
129.024
128.052
127.124
125.878
77.271
77.017
76.763
52.720
38.333
36.220
13.908
4.748
3.542
-0.004
Me
N
N
O
Current Data Parameters
NAME TKS333TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170628
Time 18.33
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678467 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

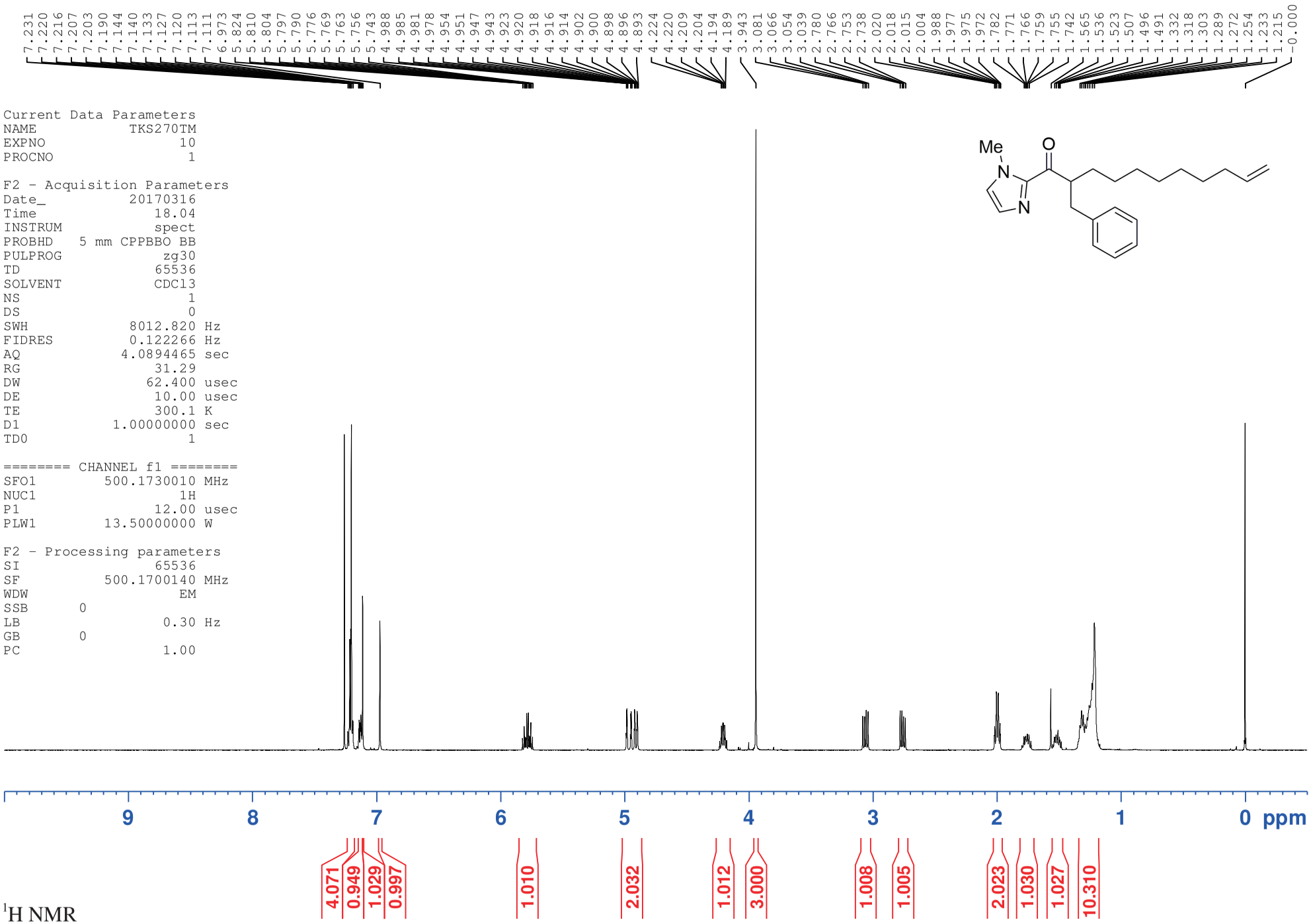
^{13}C NMR


7.257
7.227
7.215
7.211
7.198
7.198
7.196
7.183
7.141
7.137
7.131
7.129
7.124
7.118
7.111
7.107
7.079
7.077
6.921
4.602
4.589
4.587
4.573
4.560
4.558
4.544
3.879
3.155
3.139
3.127
3.112
2.794
2.780
2.766
2.753
1.581
0.000
Current Data Parameters
NAME TKS295TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170421
Time 19.35
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
9
8
7
6
5
4
3
2
1
0 ppm
8.040
2.026
0.989
1.000
0.998
3.000
2.041
2.022

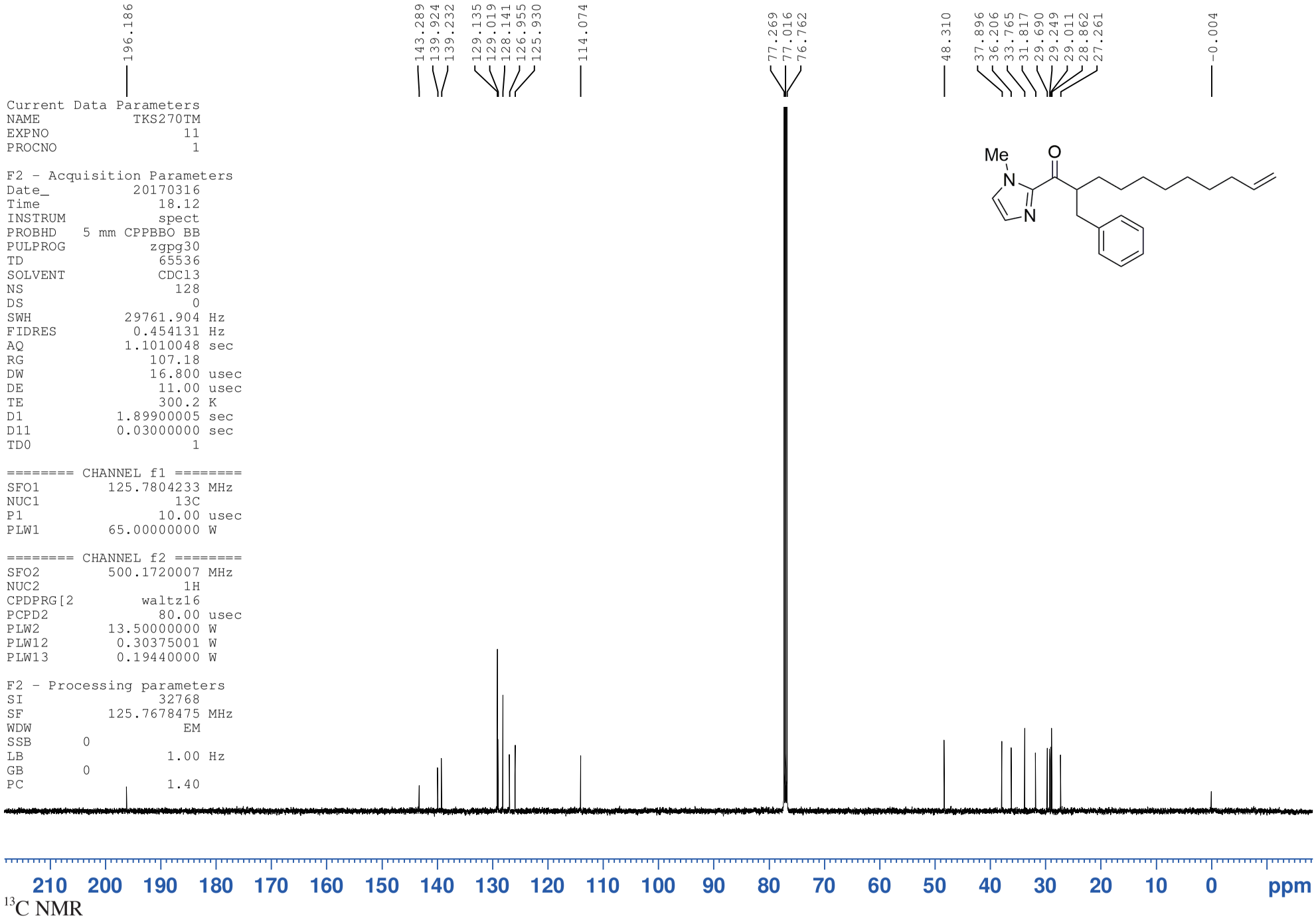
^{1}H NMR


194.821
143.048
139.589
129.137
129.089
128.202
126.880
126.049
77.274
77.021
76.767
49.991
37.511
36.087
0.001
Current Data Parameters
NAME TKS295TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170421
Time 19.38
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 67
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

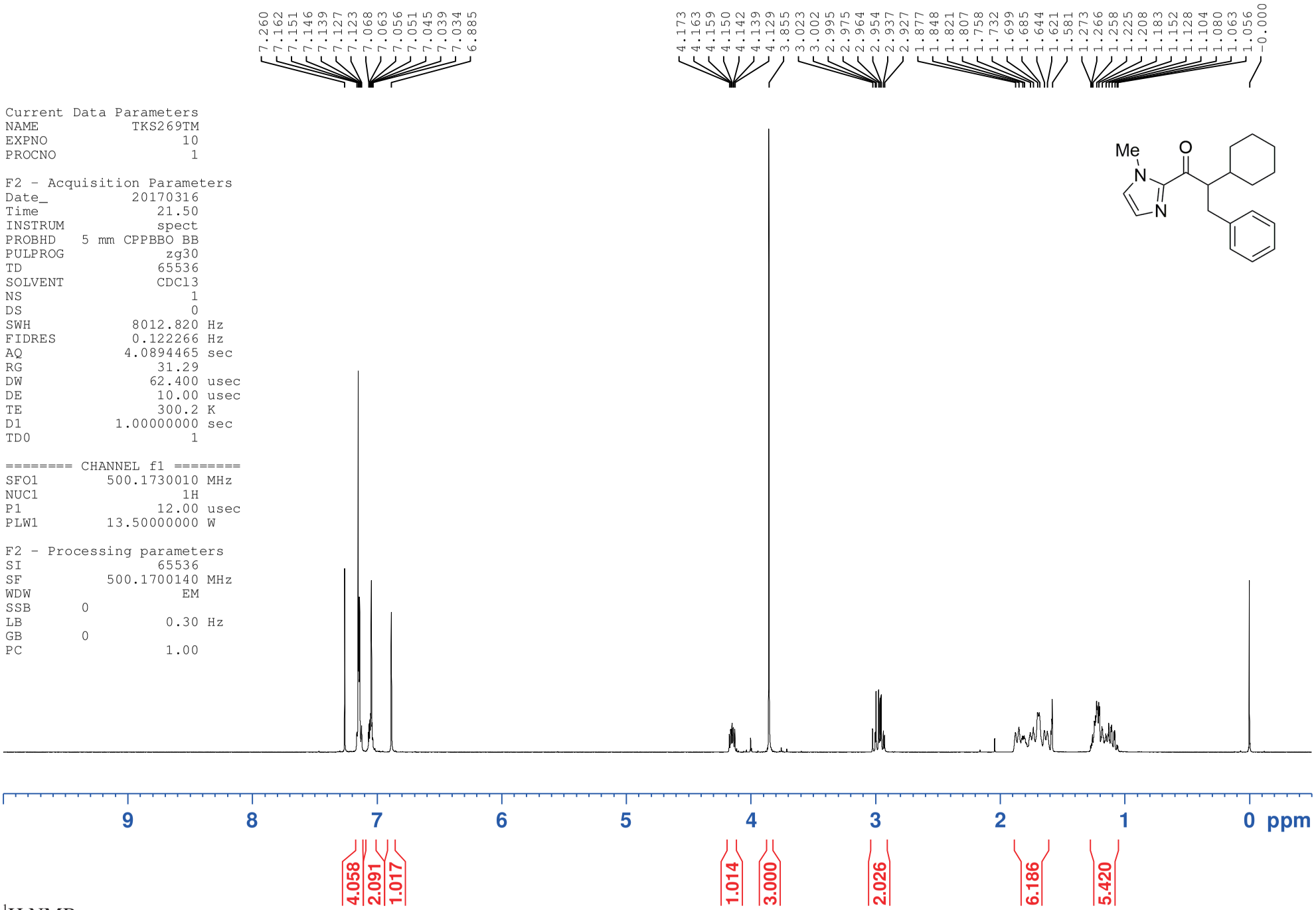
^{13}C NMR


7.231 7.220 7.216 7.207 7.203 7.190 7.144 7.140 7.133 7.127 7.120 7.113 7.111 6.973 5.824 5.810 5.804 5.797 5.790 5.776 5.769 5.763 5.756 5.743 4.988 4.985 4.981 4.978 4.954 4.951 4.947 4.943 4.923 4.920 4.918 4.916 4.914 4.902 4.900 4.898 4.896 4.893 4.224 4.220 4.209 4.204 4.194 4.189 3.943 3.081 3.066 3.054 3.039 2.780 2.766 2.753 2.738 2.020 2.018 2.015 2.004 1.988 1.977 1.975 1.972 1.782 1.771 1.766 1.759 1.755 1.742 1.565 1.536 1.523 1.507 1.496 1.491 1.332 1.318 1.303 1.289 1.272 1.254 1.233 1.215 -0.000
Current Data Parameters
NAME TKS270TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170316
Time 18.04
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
9 8 7 6 5 4 3 2 1 0 ppm
4.071 0.949 1.029 0.997 1.010 2.032 1.012 3.000 1.008 1.005 2.023 1.030 1.027 10.310

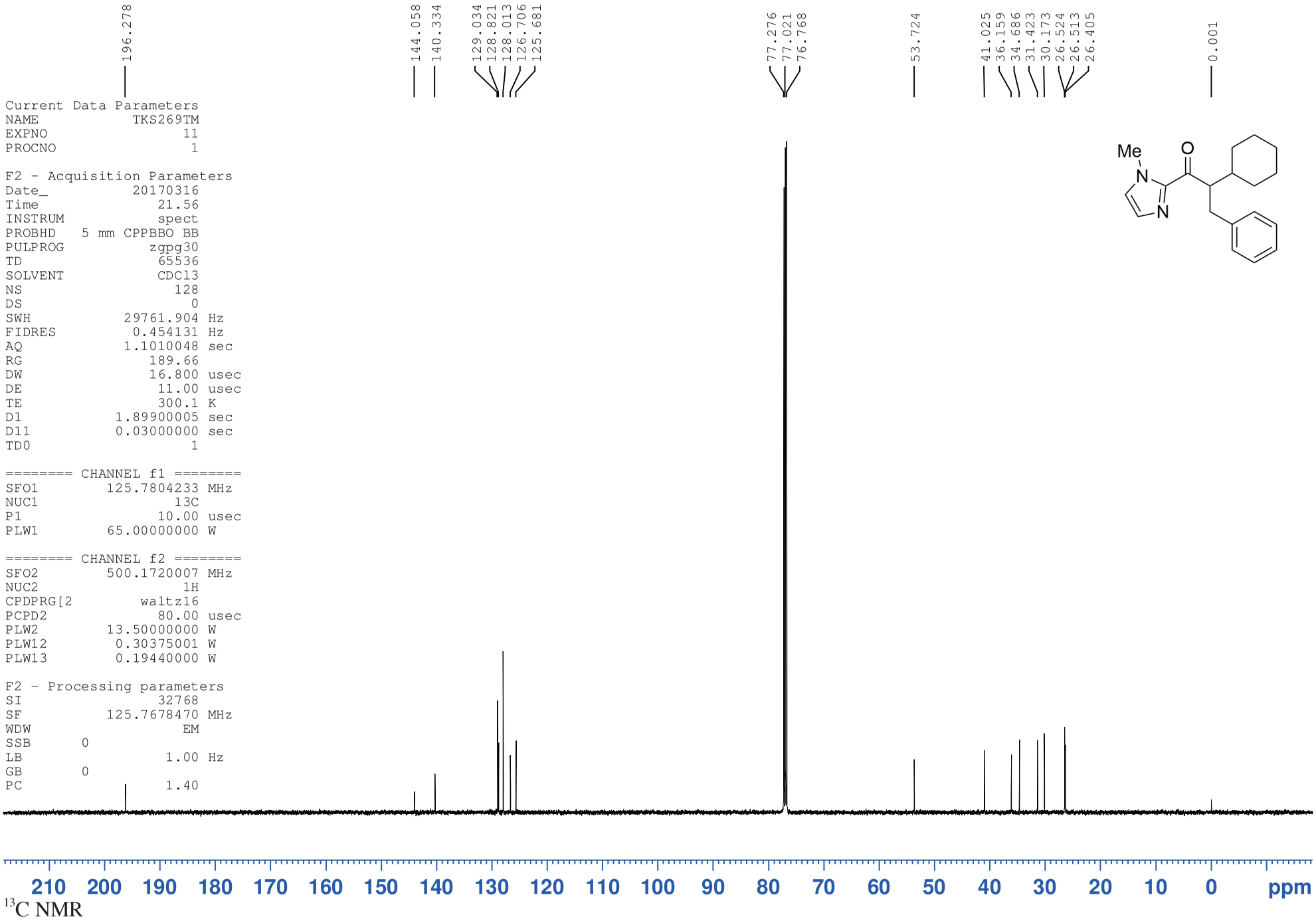
^{1}H NMR


196.186
143.289
139.924
139.232
129.135
129.019
128.141
126.955
125.930
114.074
77.269
77.016
76.762
48.310
37.896
36.206
33.765
31.817
29.690
29.249
29.011
28.862
27.261
-0.004
Me
N
N
O
Current Data Parameters
NAME TKS270TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170316
Time 18.12
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678475 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

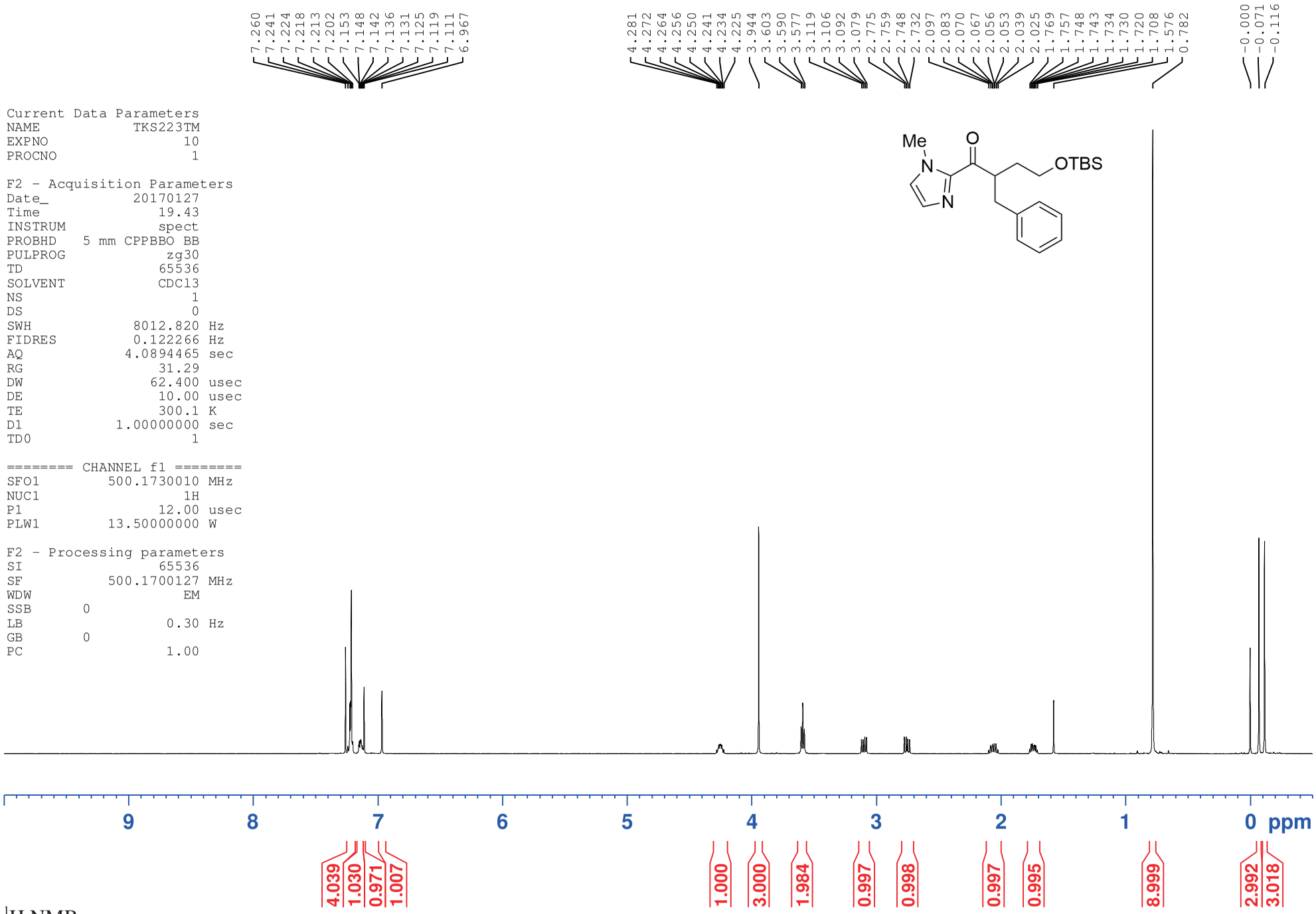
^{13}C NMR


7.260
7.162
7.151
7.146
7.139
7.127
7.123
7.068
7.063
7.056
7.051
7.045
7.039
7.034
6.885
4.173
4.163
4.159
4.150
4.142
4.139
4.129
3.855
3.023
3.002
2.995
2.975
2.964
2.954
2.937
2.927
1.877
1.848
1.821
1.807
1.758
1.732
1.699
1.685
1.644
1.621
1.581
1.273
1.266
1.258
1.225
1.208
1.183
1.152
1.128
1.104
1.080
1.063
1.056
0.000
Current Data Parameters
NAME TKS269TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170316
Time 21.50
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
9
8
7
6
5
4
3
2
1
0 ppm
4.058
2.091
1.017
1.014
3.000
2.026
6.186
5.420

^{1}H NMR


196.278
144.058
140.334
129.034
128.821
128.013
126.706
125.681
77.276
77.021
76.768
53.724
41.025
36.159
34.686
31.423
30.173
26.524
26.513
26.405
0.001
Me
N
N
O
Current Data Parameters
NAME TKS269TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170316
Time 21.56
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR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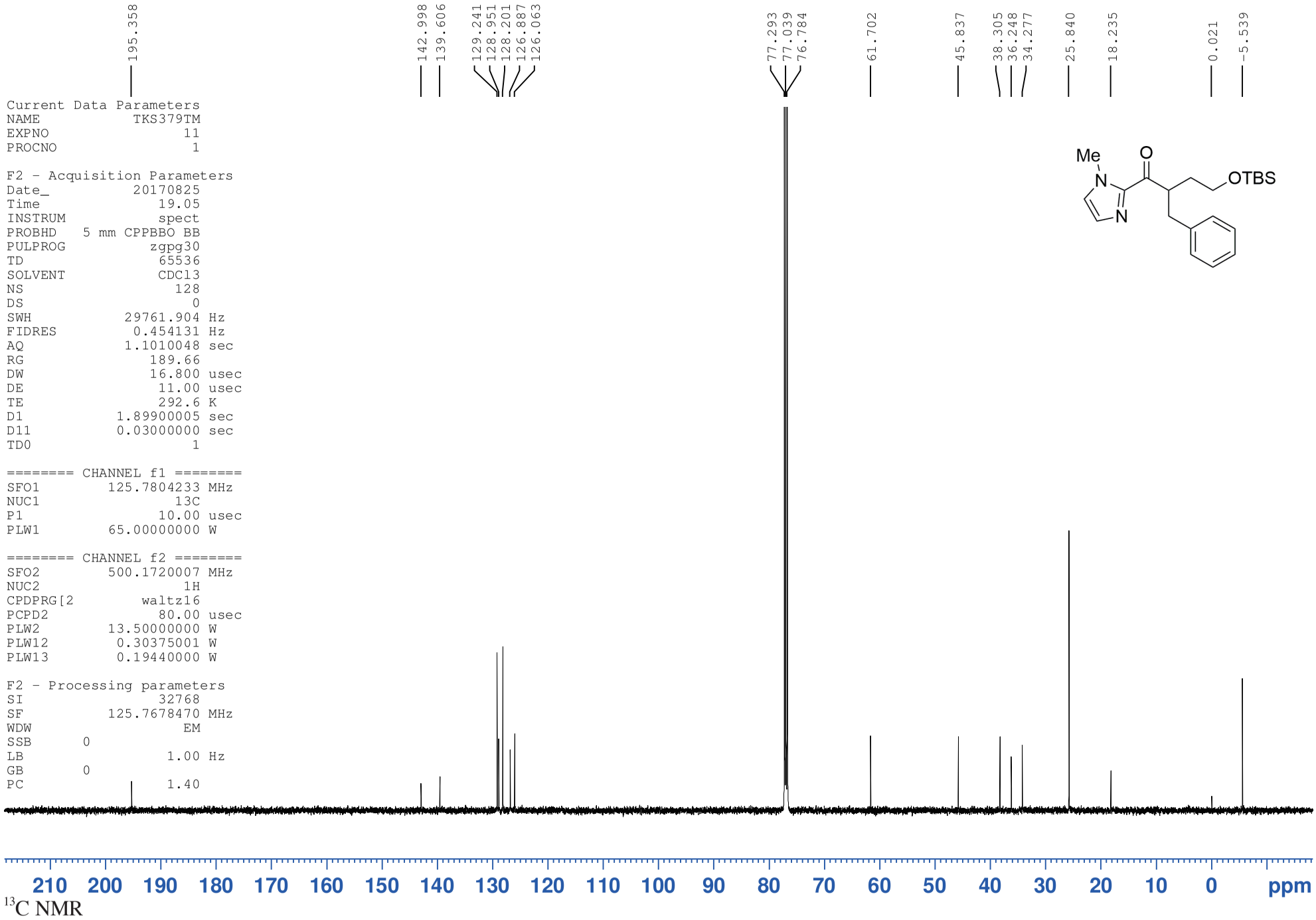
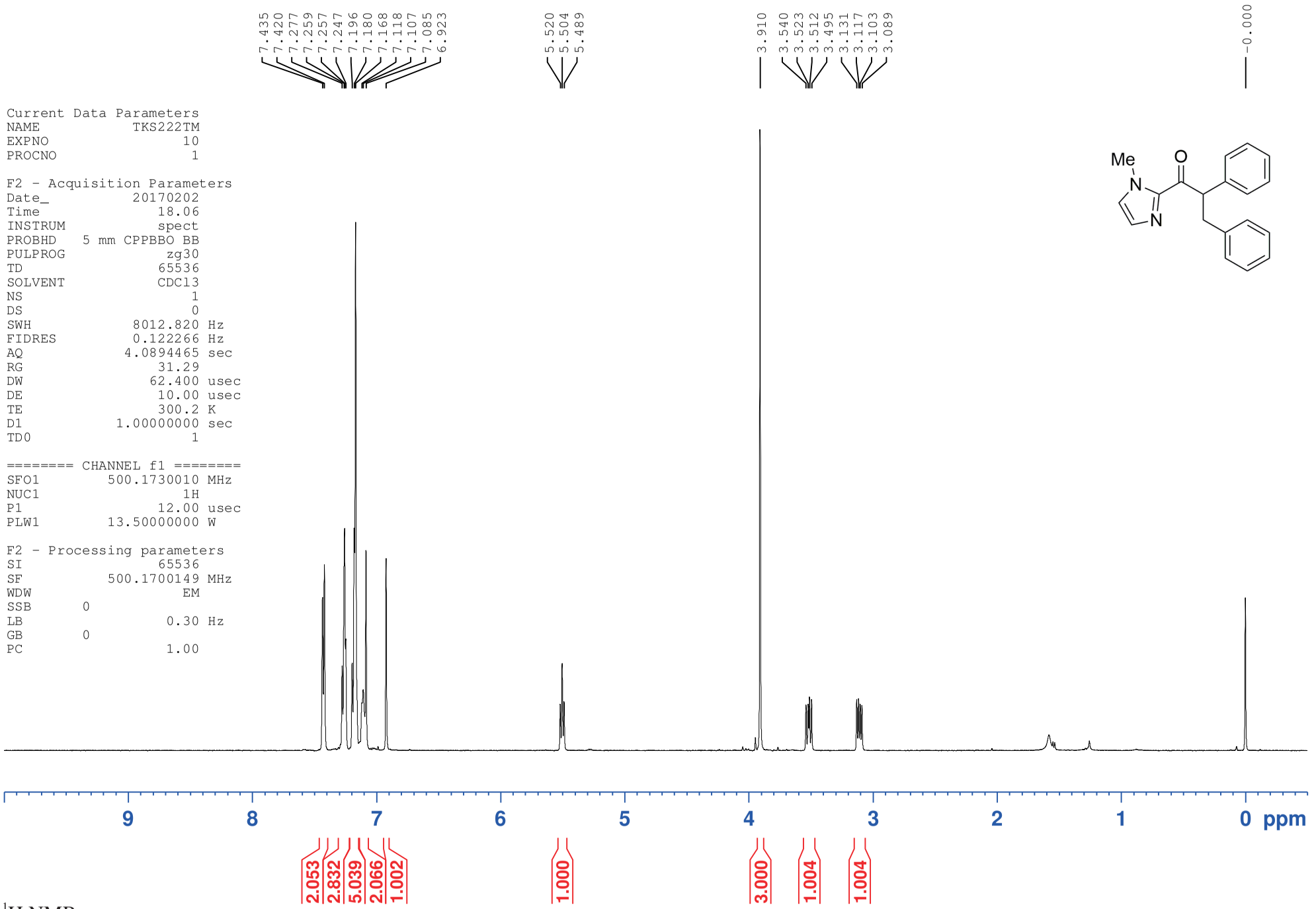
7.241
7.224
7.218
7.213
7.202
7.153
7.148
7.142
7.136
7.131
7.125
7.119
7.111
6.967
4.281
4.272
4.264
4.256
4.250
4.241
4.234
4.225
3.944
3.603
3.590
3.577
3.119
3.106
3.092
3.079
2.775
2.759
2.748
2.732
2.097
2.083
2.070
2.067
2.056
2.053
2.039
2.025
1.769
1.757
1.748
1.743
1.734
1.730
1.720
1.708
1.576
0.782
-0.000
-0.071
-0.116
Me
N
N
O
OTBS
Current Data Parameters
NAME TKS223TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170127
Time 19.43
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
4.039
1.030
0.971
1.007
1.000
3.000
1.984
0.997
0.998
0.997
0.995
8.999
2.992
3.018

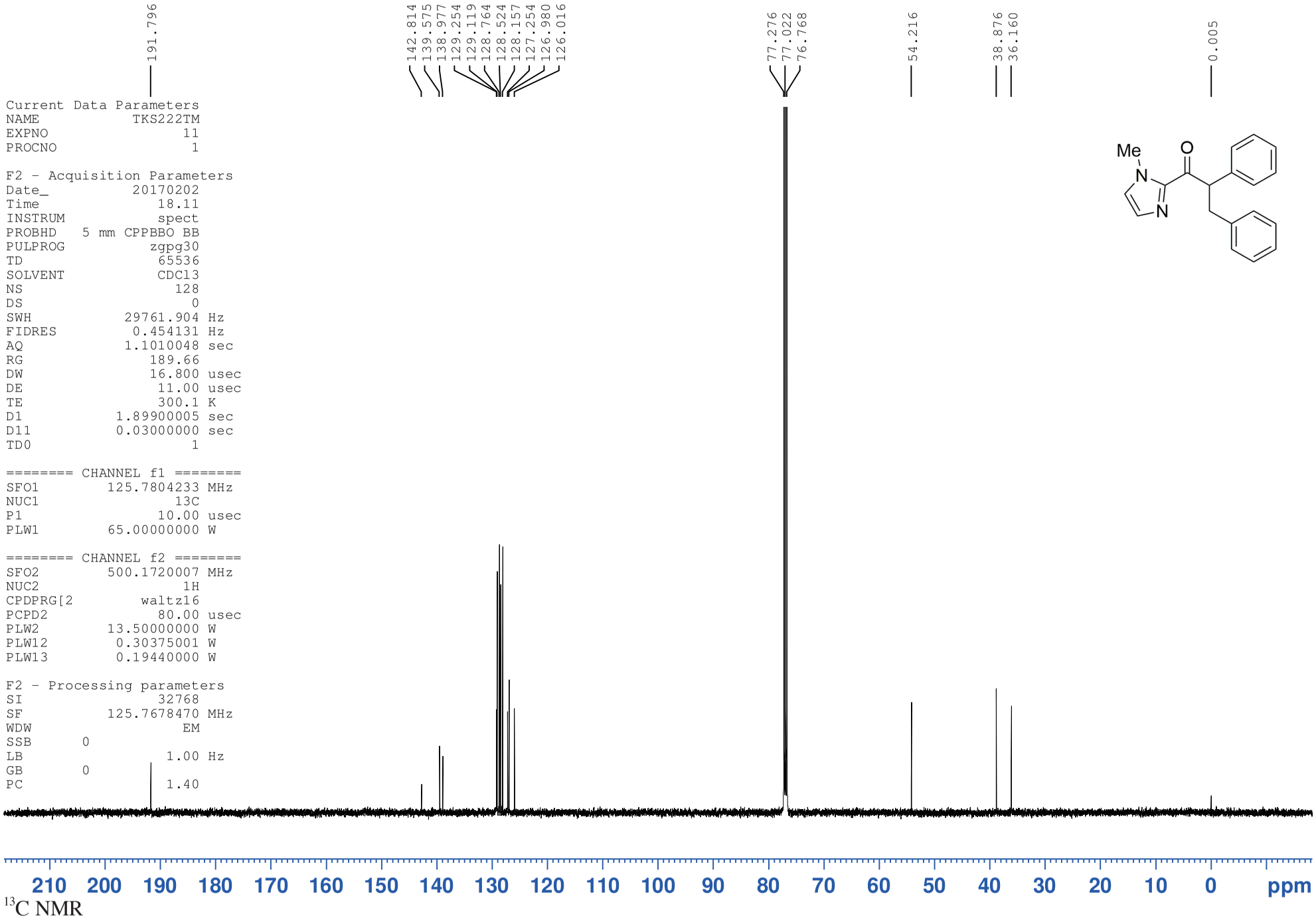
^{1}H NMR


195.358
142.998
139.606
129.241
128.951
128.201
126.887
126.063
77.293
77.039
76.784
61.702
45.837
38.305
36.248
34.277
25.840
18.235
0.021
-5.539
Me
N
N
O
OTBS
Current Data Parameters
NAME TKS379TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170825
Time 19.05
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.6 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

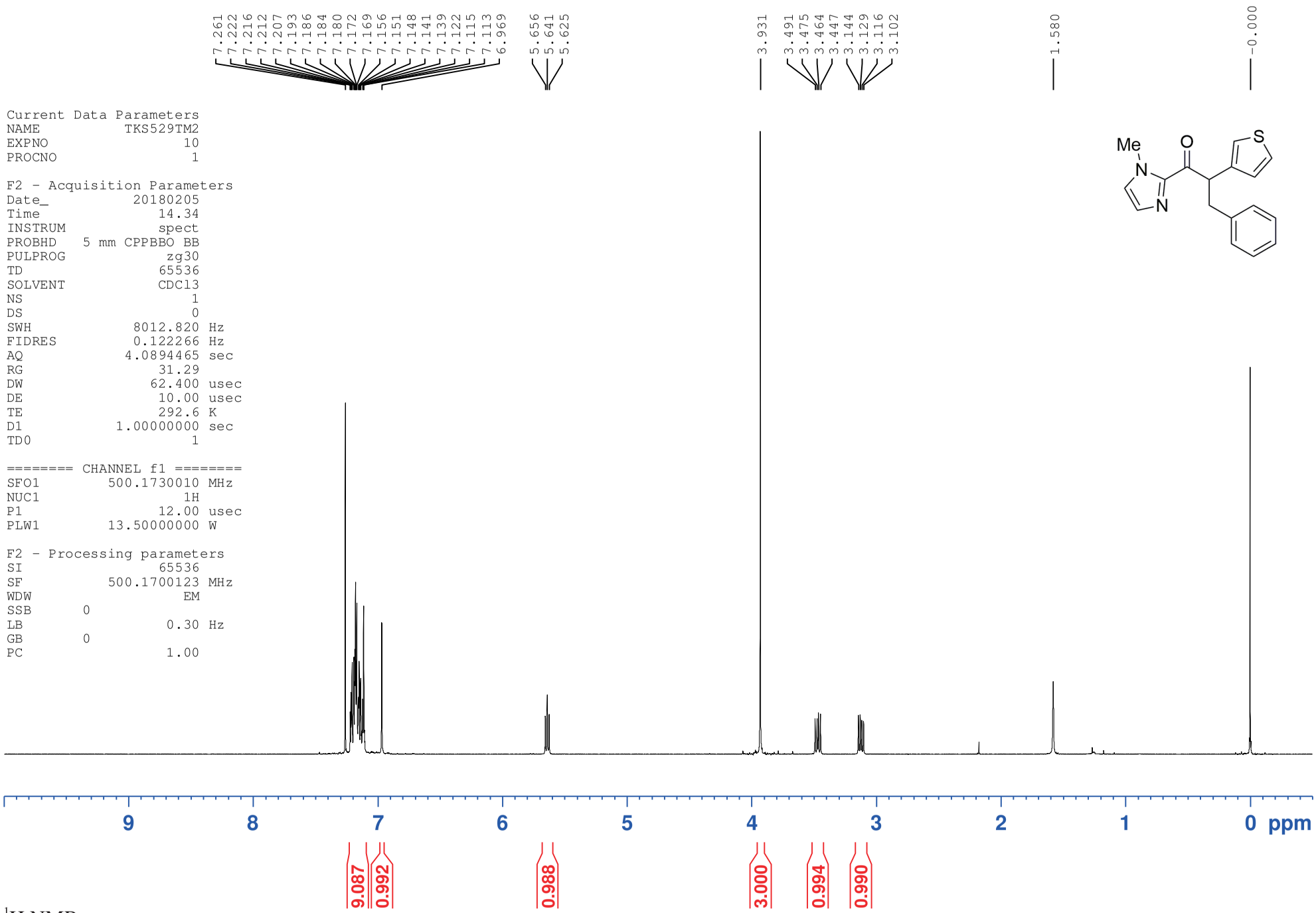
^{13}C NMR


7.435
7.420
7.277
7.259
7.257
7.247
7.196
7.180
7.168
7.118
7.107
7.085
6.923
5.520
5.504
5.489
3.910
3.540
3.523
3.512
3.495
3.131
3.117
3.103
3.089
-0.000
Current Data Parameters
NAME TKS222TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170202
Time 18.06
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700149 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
9
8
7
6
5
4
3
2
1
0 ppm
2.053
2.832
5.039
2.066
1.002
1.000
3.000
1.004
1.004

^{1}H NMR

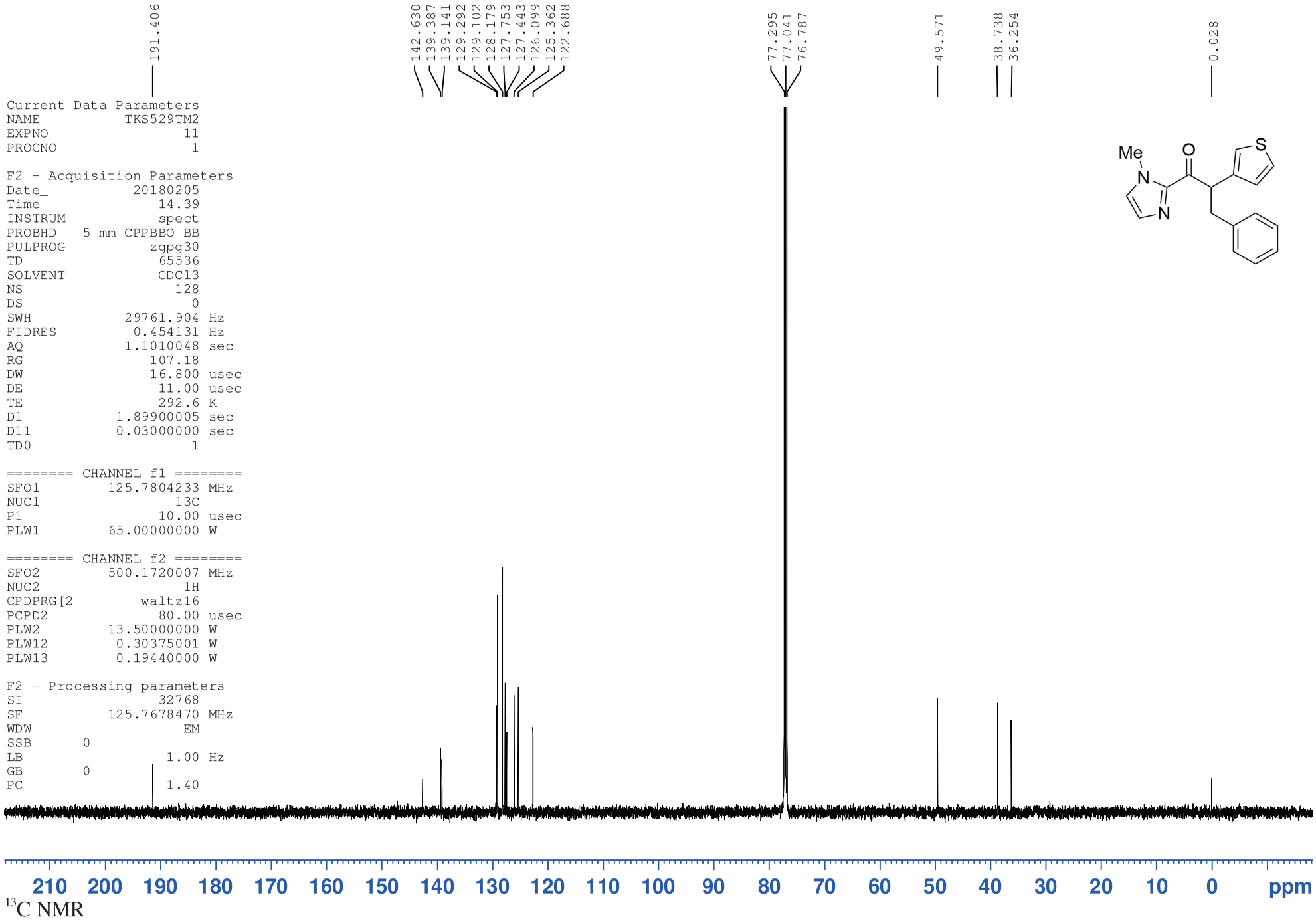

191.796
142.814
139.575
138.977
129.254
129.119
128.764
128.524
128.157
127.254
126.980
126.016
77.276
77.022
76.768
54.216
38.876
36.160
0.005
Me
N
N
O
Current Data Parameters
NAME TKS222TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170202
Time 18.11
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

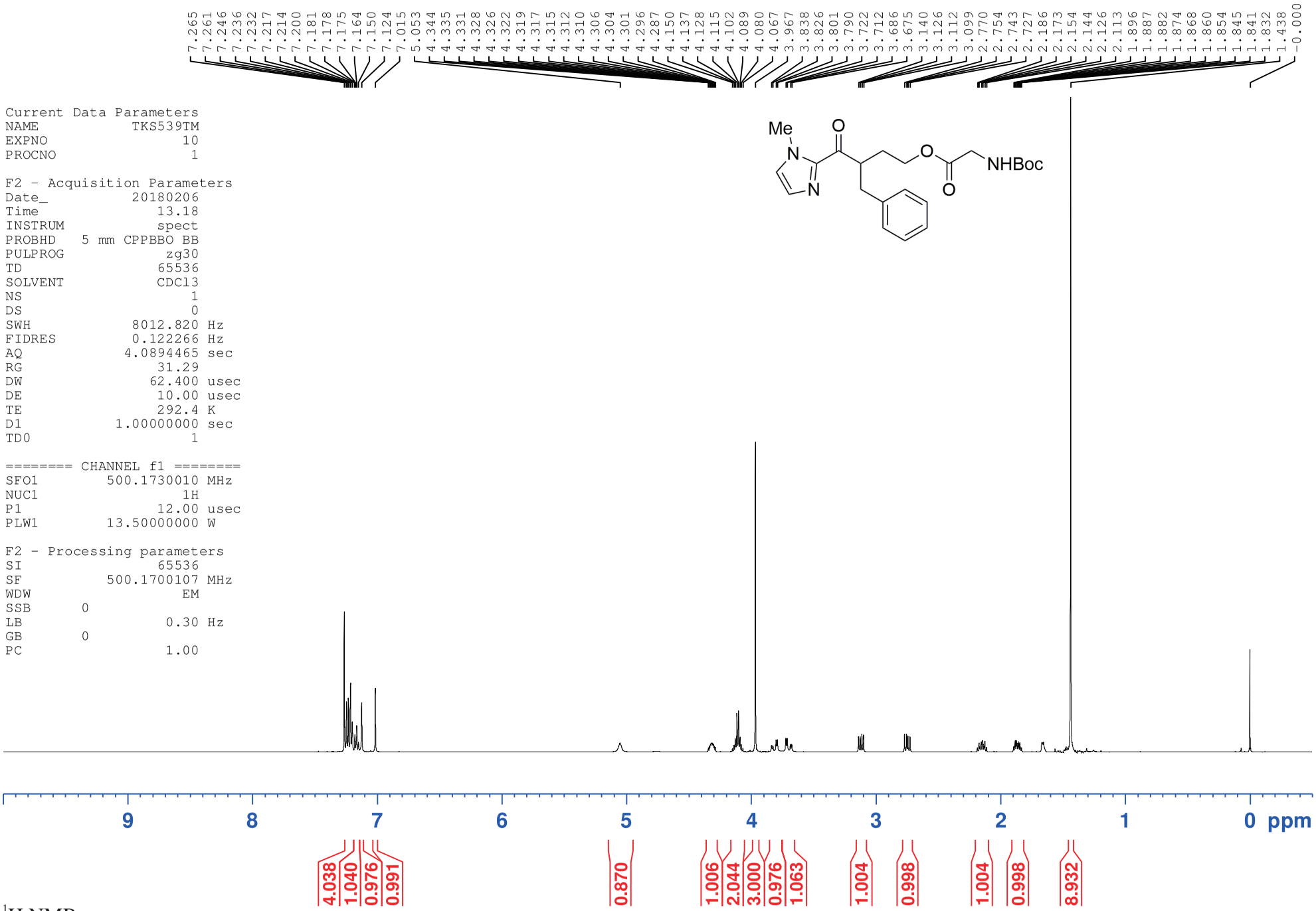

7.261
7.222
7.216
7.212
7.207
7.193
7.186
7.184
7.180
7.172
7.169
7.156
7.151
7.148
7.141
7.139
7.122
7.115
7.113
6.969
5.656
5.641
5.625
3.931
3.491
3.475
3.464
3.447
3.144
3.129
3.116
3.102
1.580
-0.000
Current Data Parameters
NAME TKS529TM2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180205
Time 14.34
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
S
9
8
7
6
5
4
3
2
1
0 ppm
9.087
0.992
0.988
3.000
0.994
0.990

S140

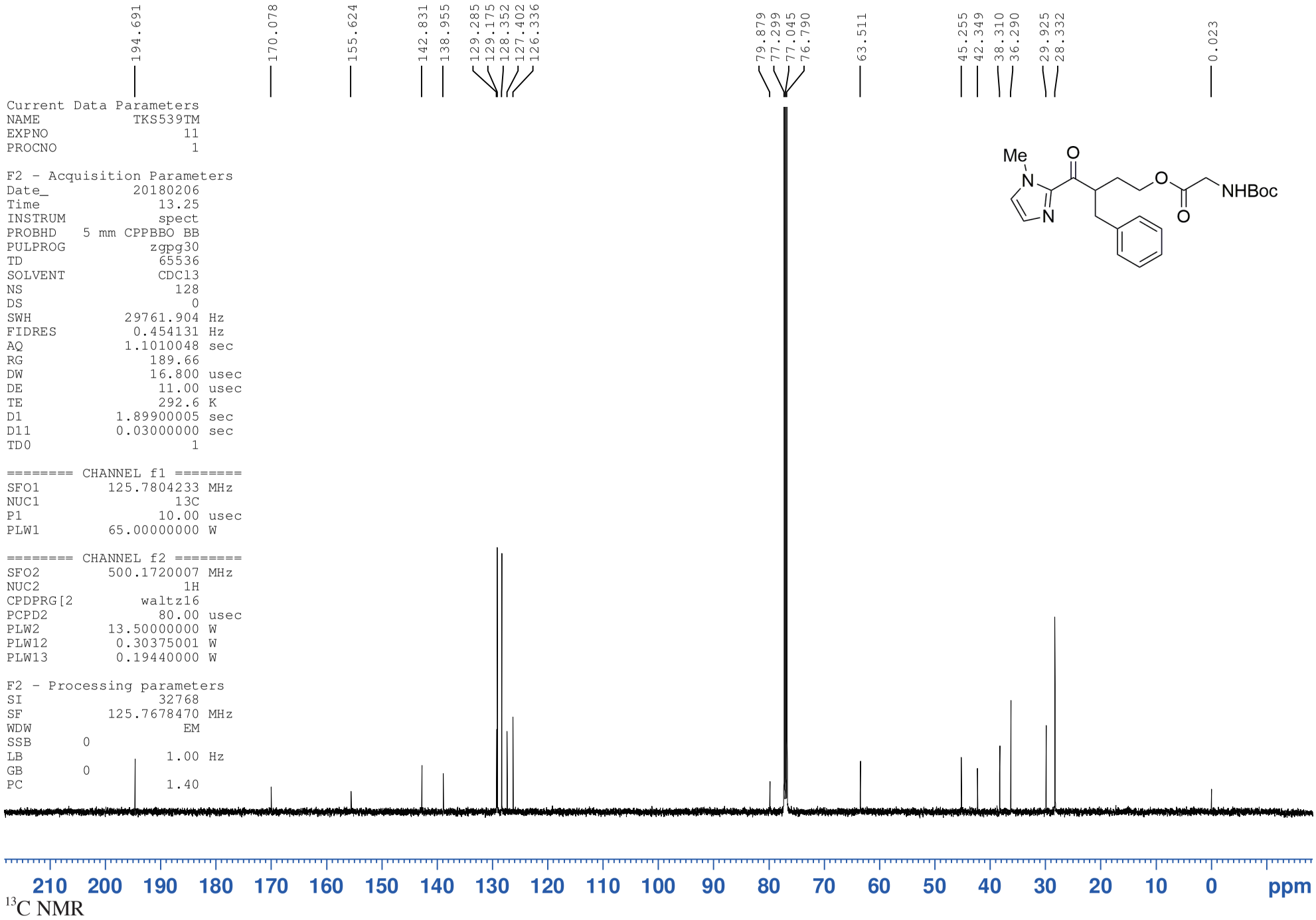
^{1}H NMR


191.406
142.630
139.387
139.141
129.292
129.102
128.179
127.753
127.443
126.099
125.362
122.688
77.295
77.041
76.787
49.571
38.738
36.254
0.028
Current Data Parameters
NAME TKS529TM2
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180205
Time 14.39
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 292.6 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
S
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

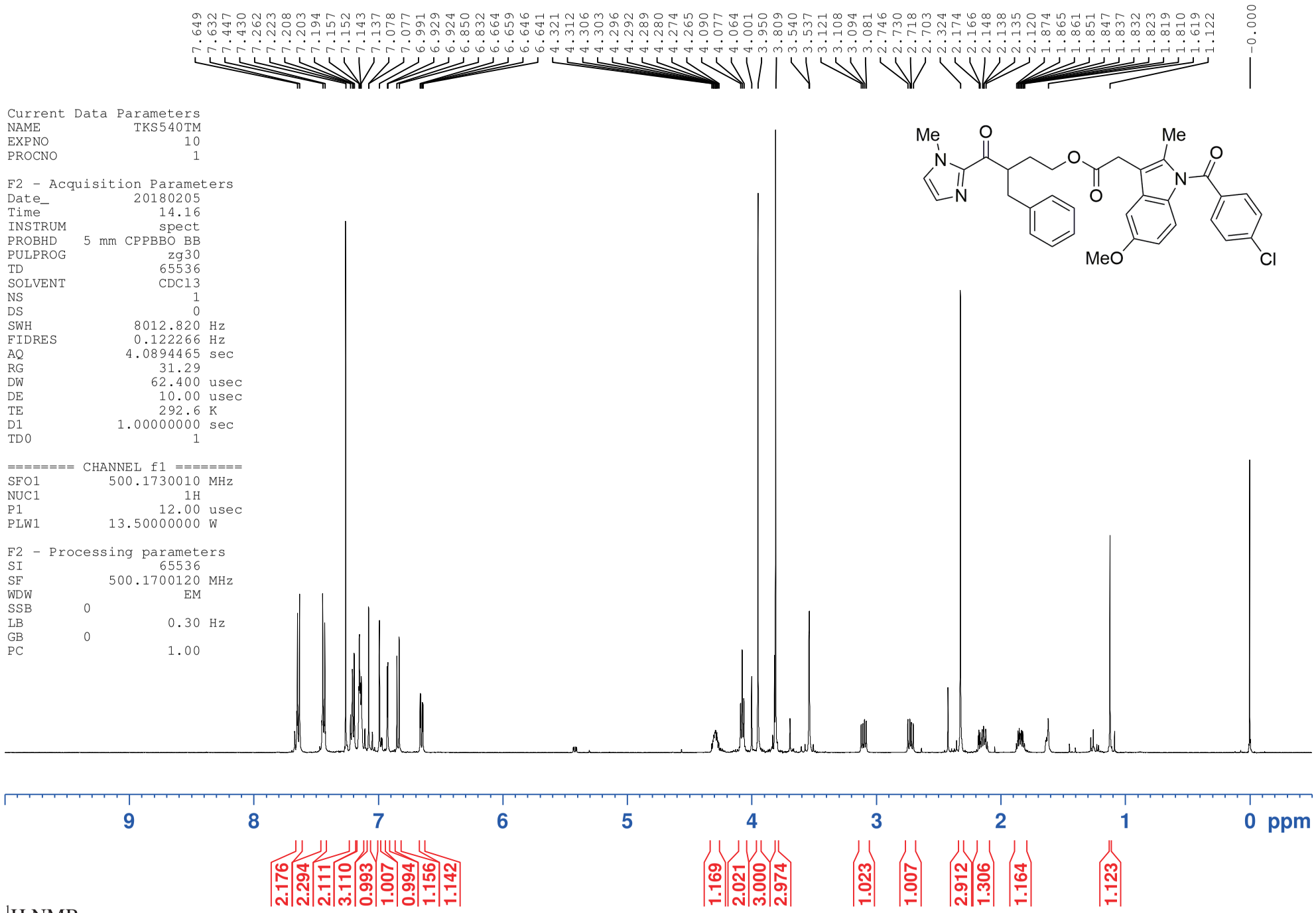
^{13}C NMR


7.265 7.261 7.246 7.236 7.232 7.217 7.214 7.200 7.181 7.178 7.175 7.164 7.150 7.124 7.015 5.053 4.344 4.335 4.331 4.328 4.326 4.322 4.319 4.317 4.315 4.312 4.310 4.306 4.304 4.301 4.296 4.287 4.150 4.137 4.128 4.115 4.102 4.089 4.080 4.067 3.967 3.838 3.826 3.801 3.790 3.722 3.712 3.686 3.675 3.140 3.126 3.112 3.099 2.770 2.754 2.743 2.727 2.186 2.173 2.154 2.144 2.126 2.113 1.896 1.887 1.882 1.874 1.868 1.860 1.854 1.845 1.841 1.832 1.438 -0.000
Me
N
N
O
O
O
NHBoc
Current Data Parameters
NAME TKS539TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180206
Time 13.18
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700107 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
4.038 1.040 0.976 0.991 0.870 1.006 2.044 3.000 0.976 1.063 1.004 0.998 1.004 0.998 8.932

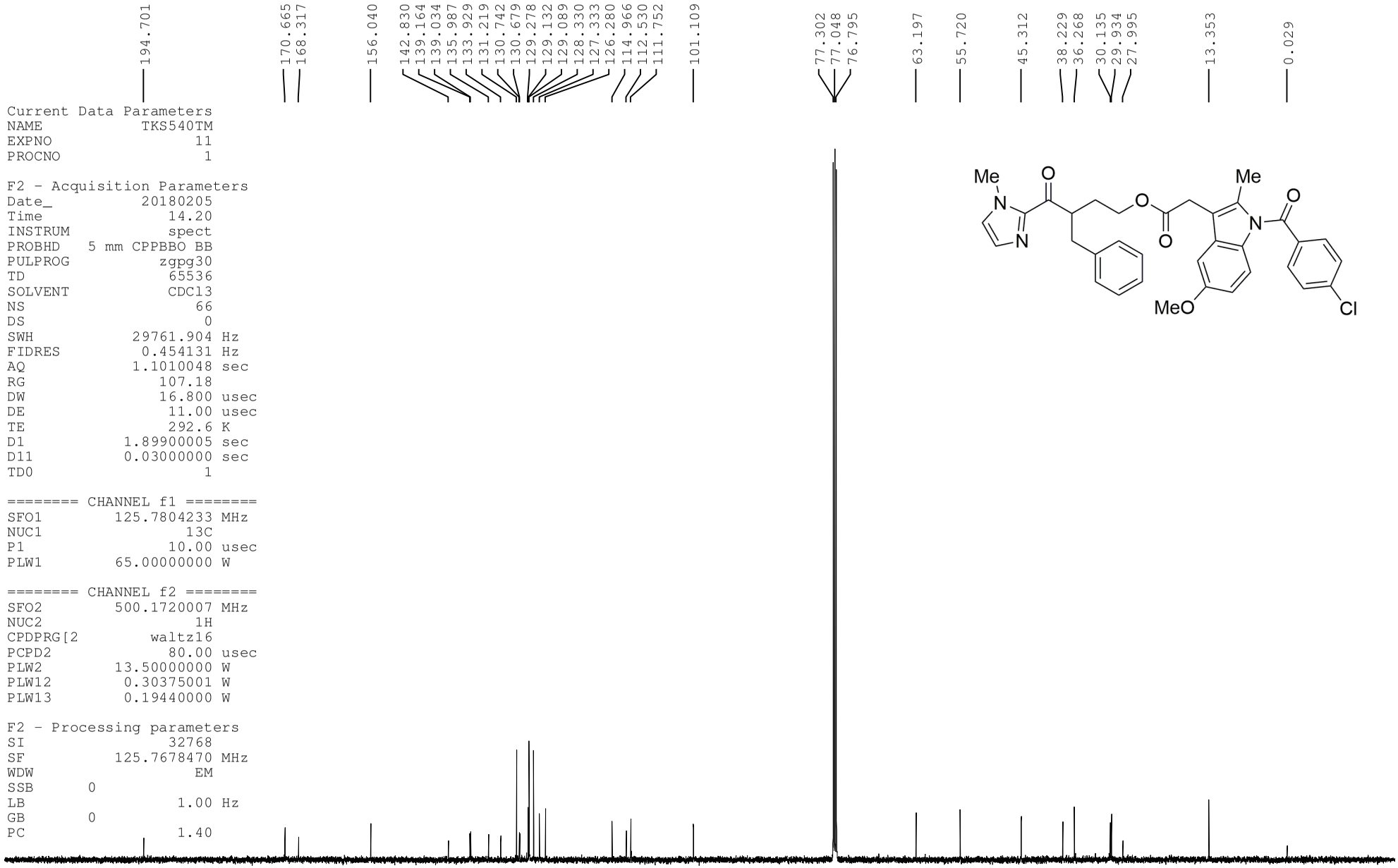
^{1}H NMR


194.691
170.078
155.624
142.831
138.955
129.285
129.175
128.352
127.402
126.336
79.879
77.299
77.045
76.790
63.511
45.255
42.349
38.310
36.290
29.925
28.332
0.023
Current Data Parameters
NAME TKS539TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180206
Time 13.25
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.6 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
O
NHBoc
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

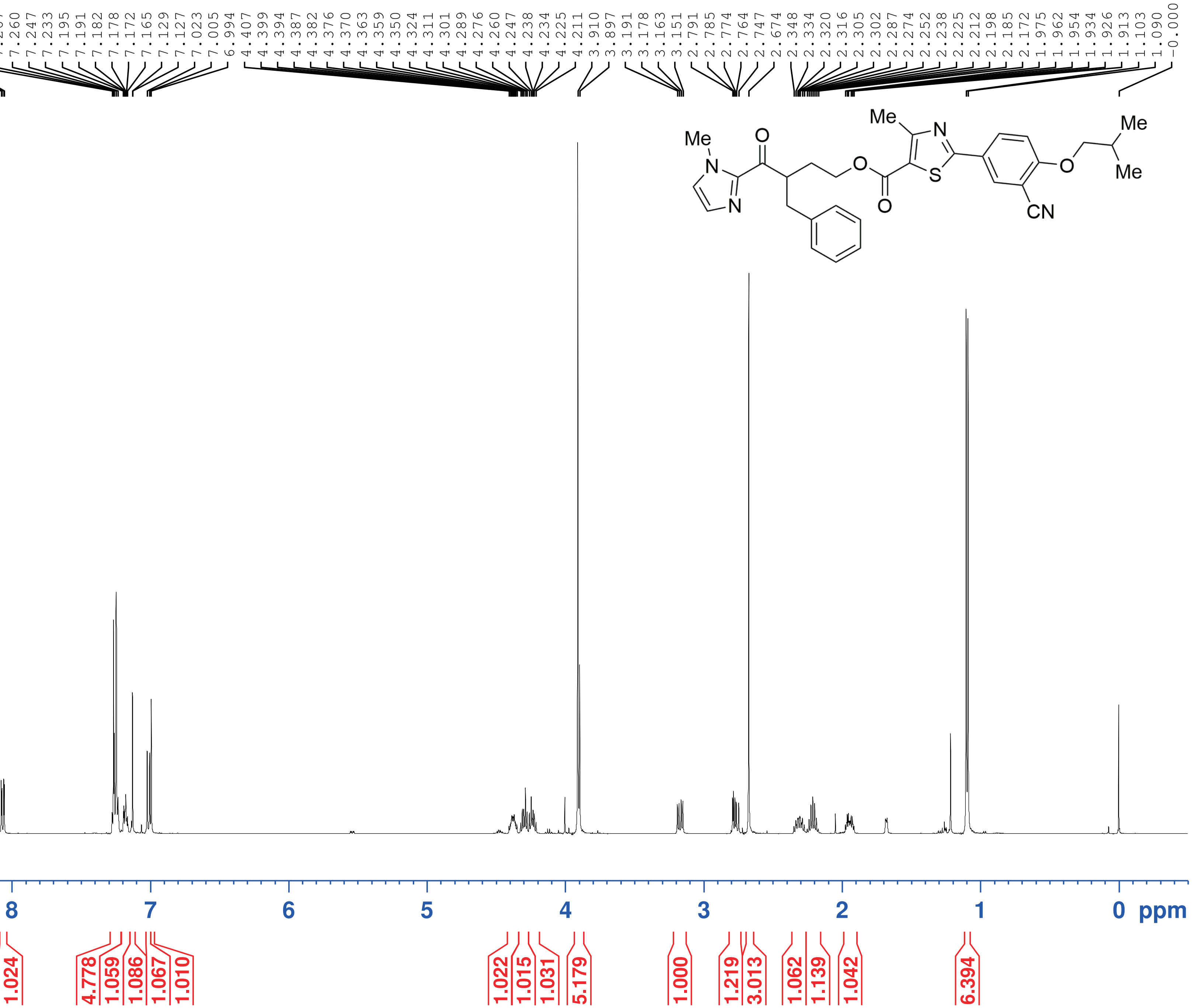

7.649 7.632 7.447 7.430 7.262 7.223 7.208 7.203 7.194 7.157 7.152 7.143 7.137 7.078 7.077 6.991 6.929 6.924 6.850 6.832 6.664 6.659 6.646 6.641 4.321 4.312 4.306 4.303 4.296 4.292 4.289 4.280 4.274 4.265 4.090 4.077 4.064 4.001 3.950 3.809 3.540 3.537 3.121 3.108 3.094 3.081 2.746 2.730 2.718 2.703 2.324 2.174 2.166 2.148 2.138 2.135 2.120 1.874 1.865 1.861 1.851 1.847 1.837 1.832 1.823 1.819 1.810 1.619 1.122 0.000
Current Data Parameters
NAME TKS540TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180205
Time 14.16
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
O
O
Me
N
O
MeO
Cl
9 8 7 6 5 4 3 2 1 0 ppm
2.176 2.294 2.111 3.110 0.993 1.007 0.994 1.156 1.142
1.169 2.021 3.000 2.974
1.023
1.007
2.912 1.306
1.164
1.123

^{1}H NMR

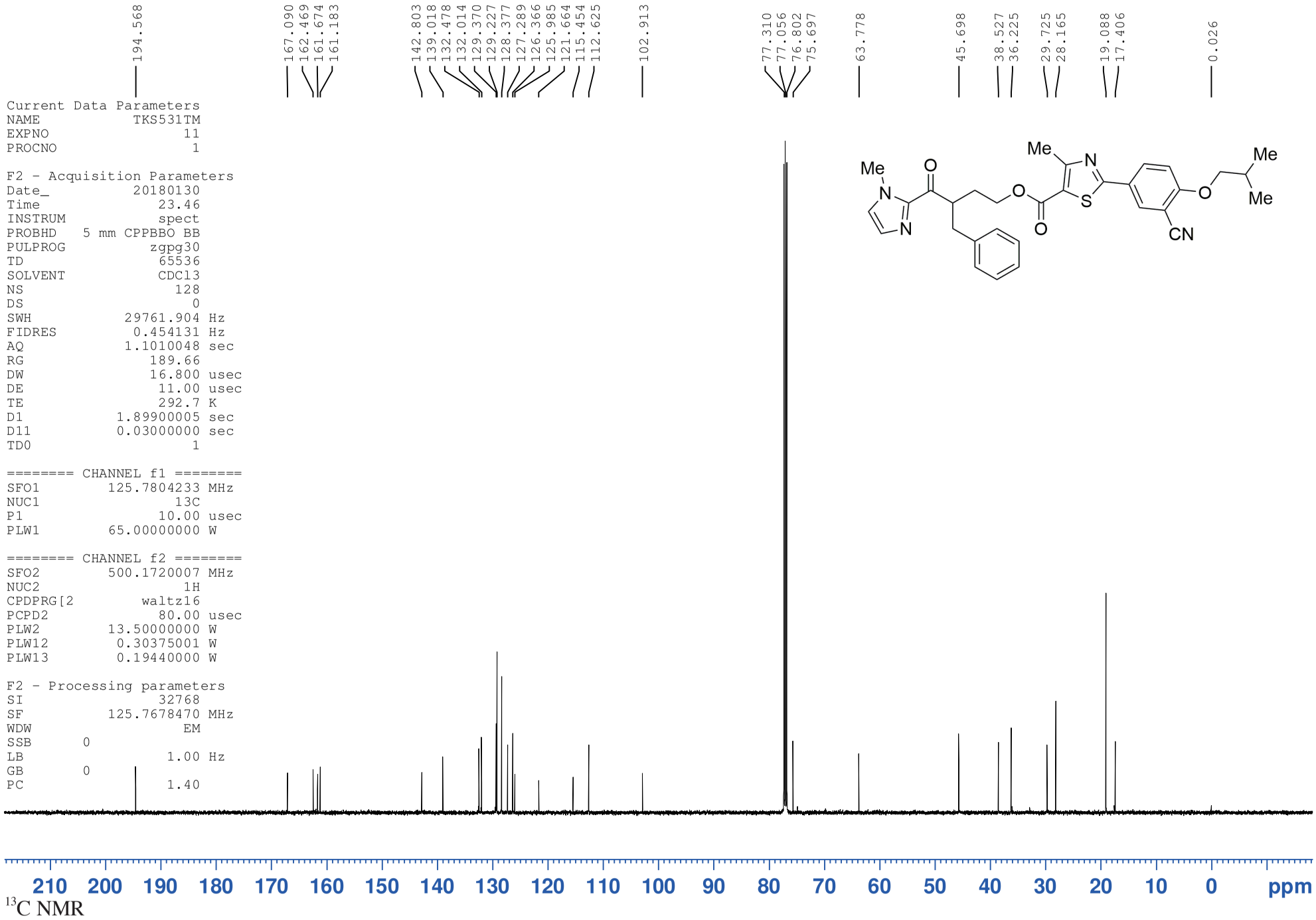

194.701
170.665
168.317
156.040
142.830
139.164
139.034
135.987
133.929
131.219
130.742
130.679
129.278
129.132
129.089
128.330
127.333
126.280
114.966
112.530
111.752
101.109
77.302
77.048
76.795
63.197
55.720
45.312
38.229
36.268
30.135
29.934
27.995
13.353
0.029
Current Data Parameters
NAME TKS540TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180205
Time 14.20
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 66
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 292.6 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
O
O
Me
N
MeO
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

Current Data Parameters	
NAME	TKS531TM
EXPNO	10
PROCNO	1
F2 - Acquisition Parameters	
Date_	20180130
Time	23.39
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	1
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894465 sec
RG	31.29
DW	62.400 usec
DE	10.00 usec
TE	292.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1730010 MHz
NUC1	1H
P1	12.00 usec
PLW1	13.50000000 W
F2 - Processing parameters	
SI	65536
SF	500.1700096 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



^{1}H NMR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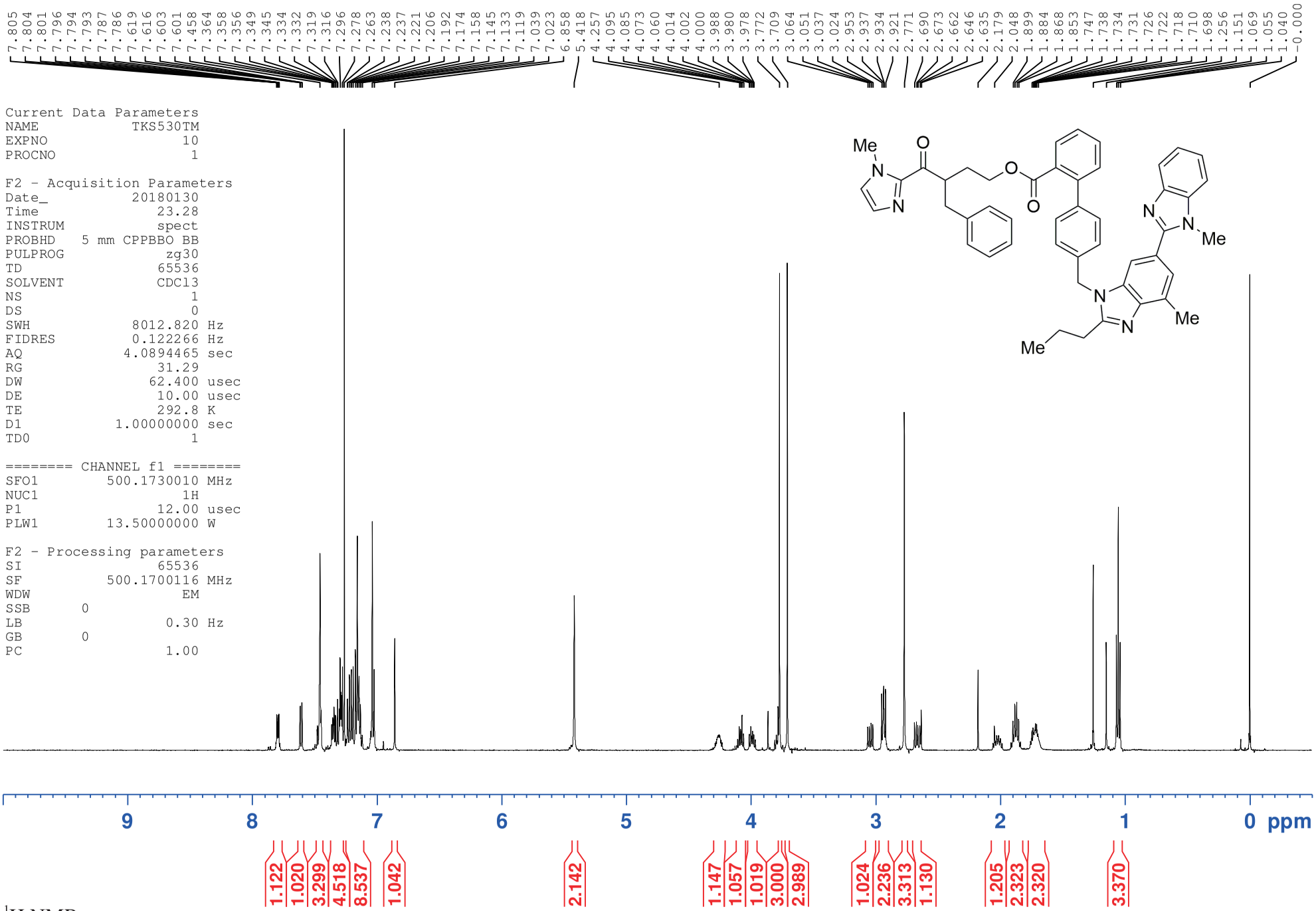
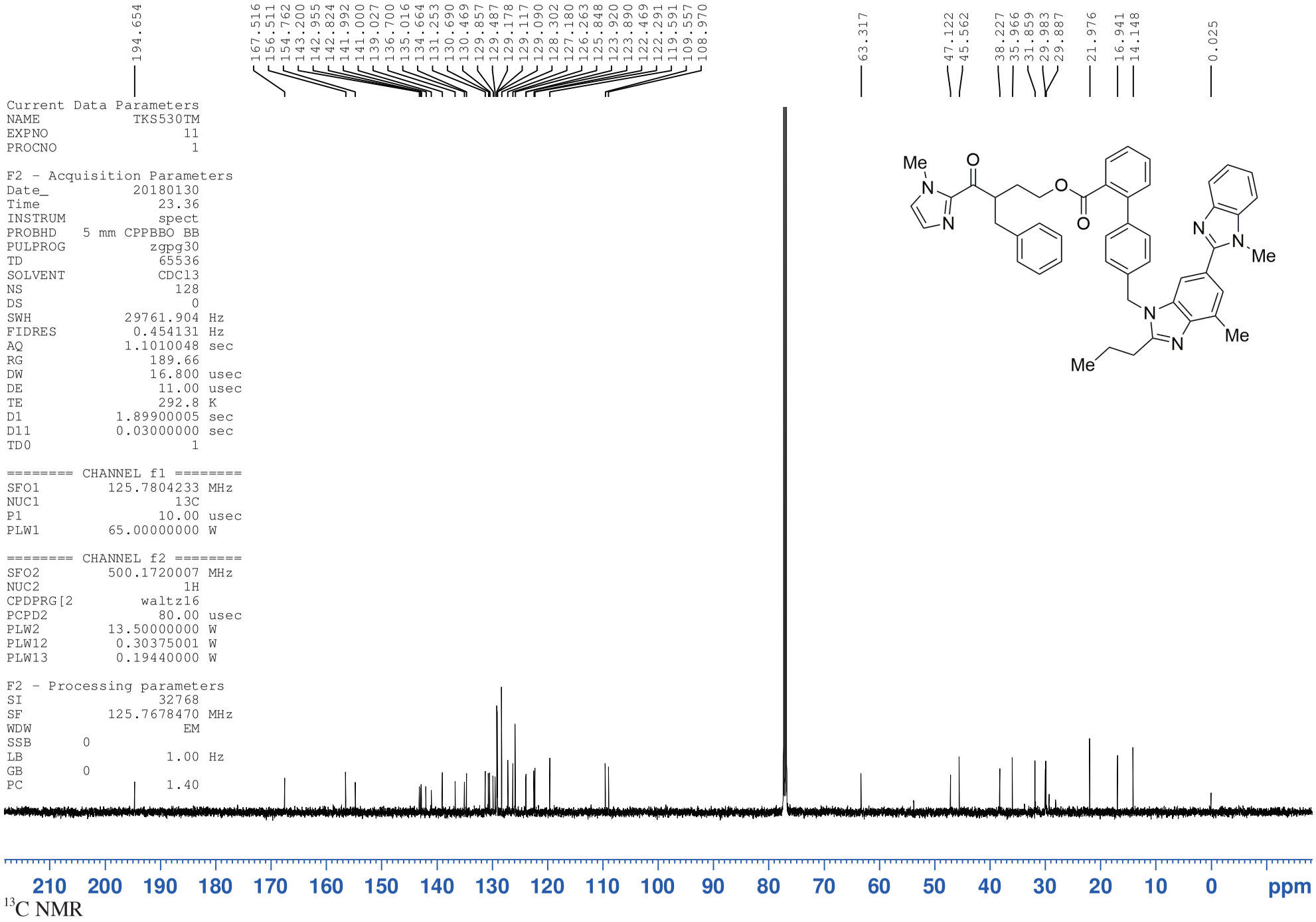
167.090
162.469
161.674
161.183
142.803
139.018
132.478
132.014
129.370
129.227
128.377
127.289
126.366
125.985
121.664
115.454
112.625
102.913
77.310
77.056
76.802
75.697
63.778
45.698
38.527
36.225
29.725
28.165
19.088
17.406
0.026
Current Data Parameters
NAME TKS531TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180130
Time 23.46
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.7 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
N
O
Me
N
S
O
Me
O
Me
CN
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR


7.805 7.804 7.801 7.796 7.794 7.793 7.787 7.786 7.619 7.616 7.603 7.601 7.458 7.364 7.358 7.356 7.349 7.345 7.334 7.332 7.319 7.316 7.296 7.278 7.263 7.238 7.237 7.221 7.206 7.192 7.174 7.158 7.145 7.133 7.119 7.039 7.023 6.858 5.418 4.257 4.095 4.085 4.073 4.060 4.014 4.002 4.000 3.988 3.980 3.978 3.772 3.709 3.064 3.051 3.037 3.024 2.953 2.937 2.934 2.921 2.771 2.690 2.673 2.662 2.646 2.635 2.179 2.048 1.899 1.884 1.868 1.853 1.747 1.738 1.734 1.731 1.726 1.722 1.718 1.710 1.698 1.256 1.151 1.069 1.055 1.040 0.000
Current Data Parameters
NAME TKS530TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180130
Time 23.28
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
O
O
N
N
N
Me
N
Me
Me
9 8 7 6 5 4 3 2 1 0 ppm
1.122 1.020 3.299 4.518 8.537 1.042 2.142 1.147 1.057 1.019 3.000 2.989 1.024 2.236 3.313 1.130 1.205 2.323 2.320 3.370

^{1}H NMR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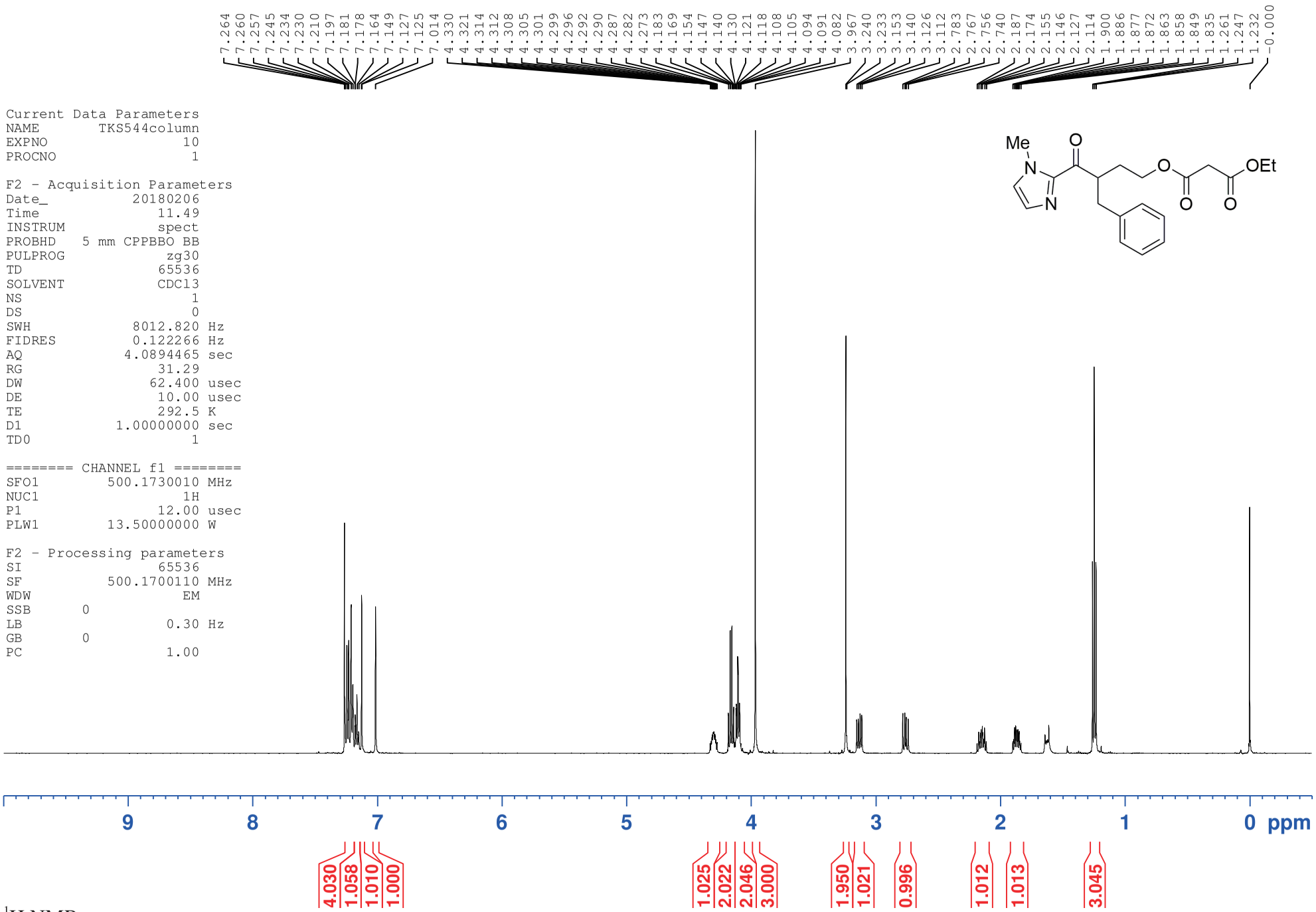
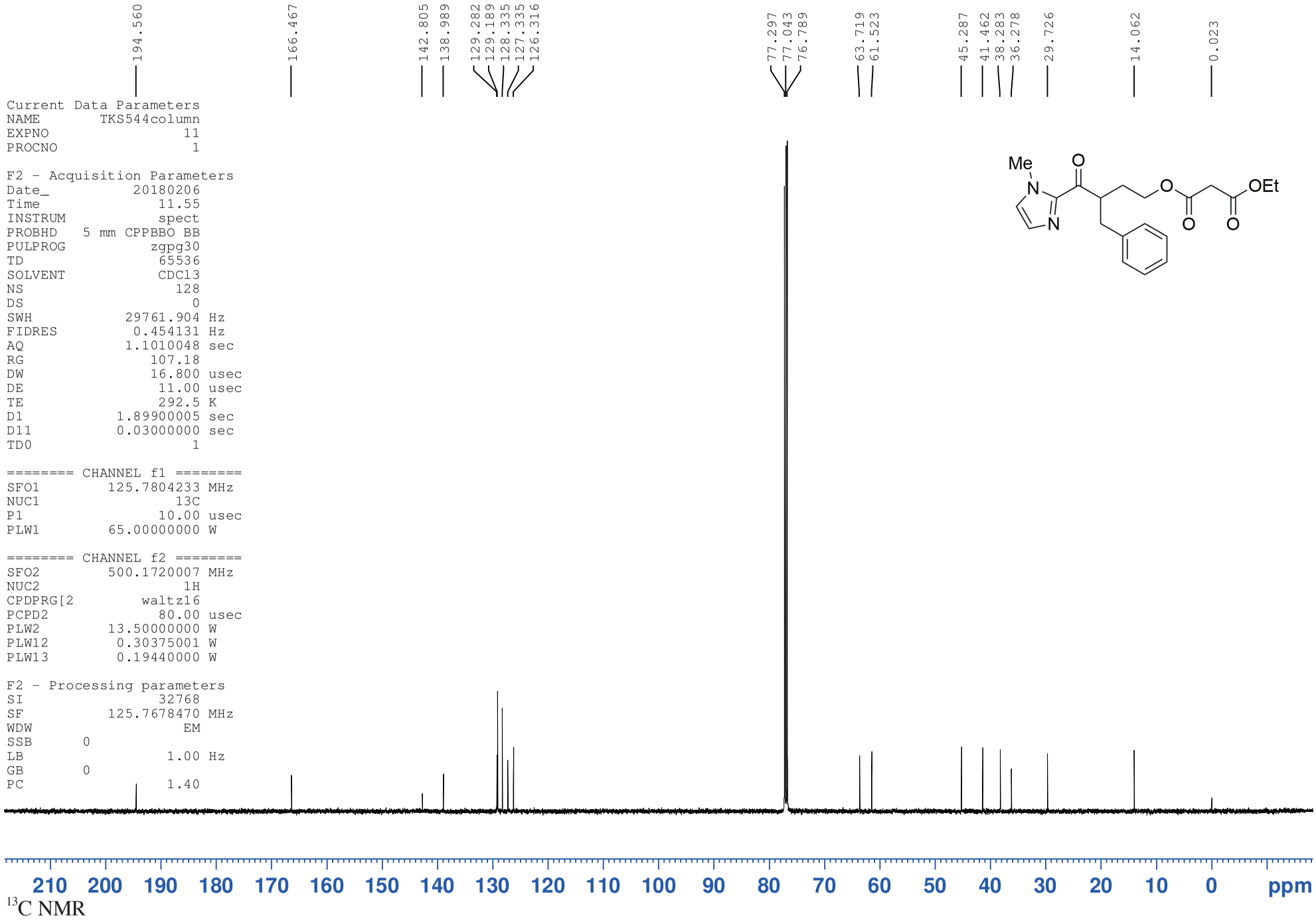
167.516
156.511
154.762
143.200
142.955
142.824
141.992
141.000
139.027
136.700
135.016
134.664
131.253
130.690
130.469
129.857
129.487
129.178
129.117
129.090
128.302
127.180
126.263
125.848
123.920
123.890
122.469
122.291
119.591
109.557
108.970
63.317
47.122
45.562
38.227
35.966
31.859
29.983
29.887
21.976
16.941
14.148
0.025
Current Data Parameters
NAME TKS530TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180130
Time 23.36
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.8 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
O
O
N
N
N
Me
N
N
Me
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

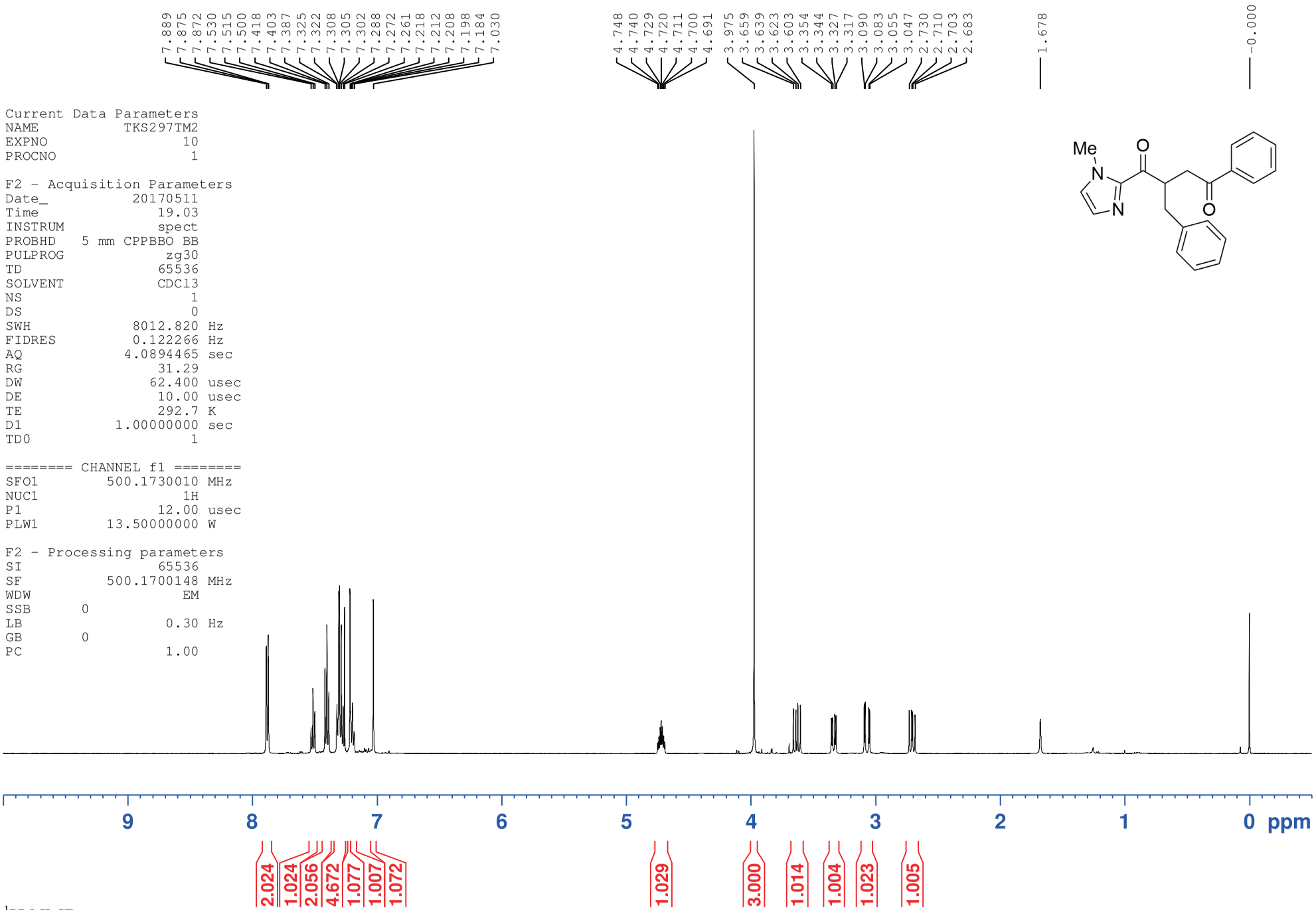
^{13}C NMR


7.264 7.260 7.257 7.245 7.234 7.230 7.210 7.197 7.181 7.178 7.164 7.149 7.127 7.125 7.014
4.330 4.321 4.314 4.312 4.308 4.305 4.301 4.299 4.296 4.292 4.290 4.287 4.282 4.273 4.183 4.169 4.154 4.147 4.140 4.130 4.121 4.118 4.108 4.105 4.094 4.091 4.082
3.967 3.240 3.233 3.153 3.140 3.126 3.112
2.783 2.767 2.756 2.740 2.187 2.174 2.155 2.146 2.127 2.114
1.900 1.886 1.877 1.872 1.863 1.858 1.849 1.835 1.261 1.247 1.232
-0.000
Current Data Parameters
NAME TKS544column
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180206
Time 11.49
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
O
O
O
OEt
9 8 7 6 5 4 3 2 1 0 ppm
4.030 1.058 1.010 1.000
1.025 2.022 2.046 3.000
1.950 1.021 0.996 1.012 1.013 3.045

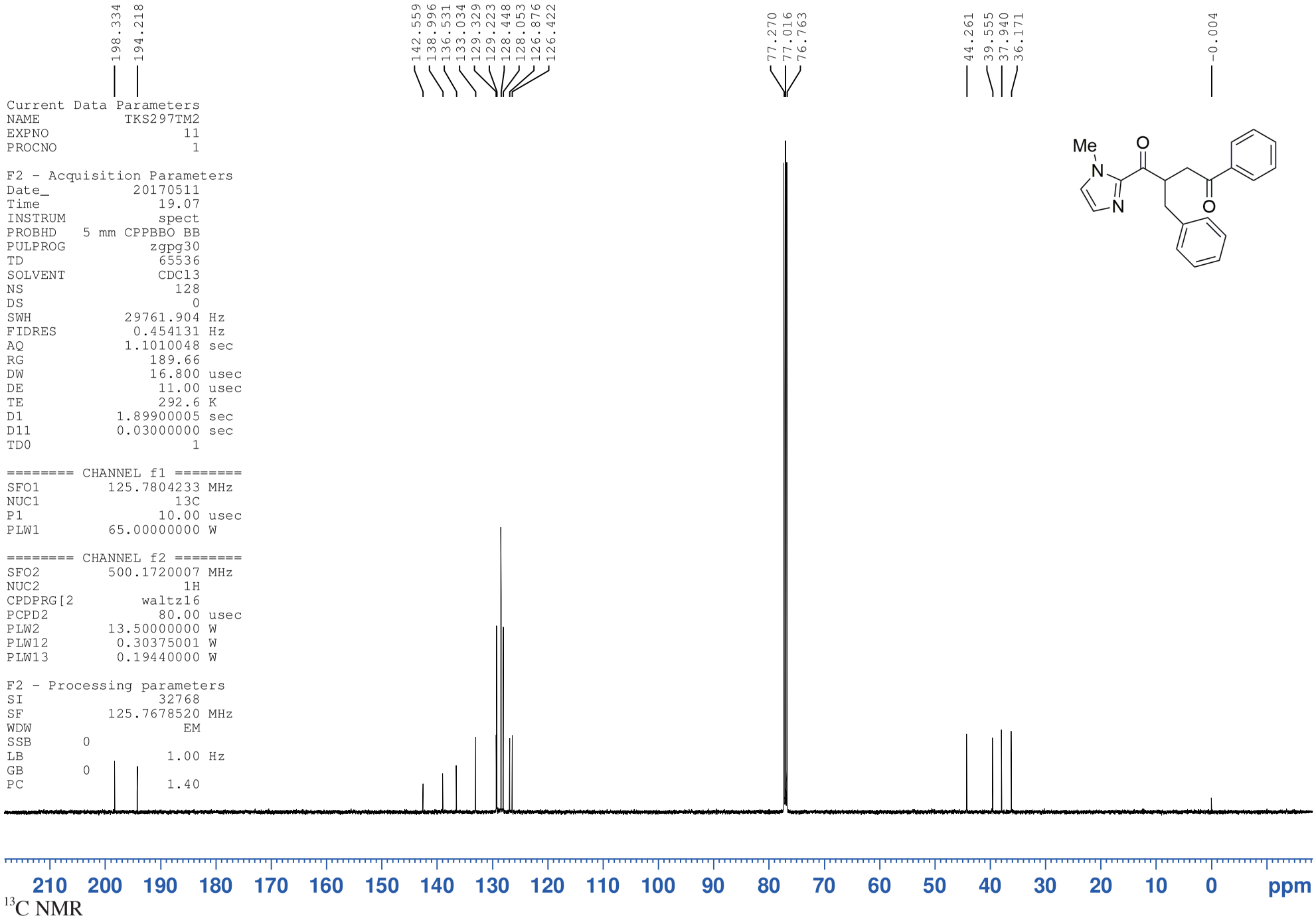
^{1}H NMR


194.560
166.467
142.805
138.989
129.282
129.189
128.335
127.335
126.316
77.297
77.043
76.789
63.719
61.523
45.287
41.462
38.283
36.278
29.726
14.062
0.023
Current Data Parameters
NAME TKS544column
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180206
Time 11.55
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 292.5 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
O
O
OEt
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

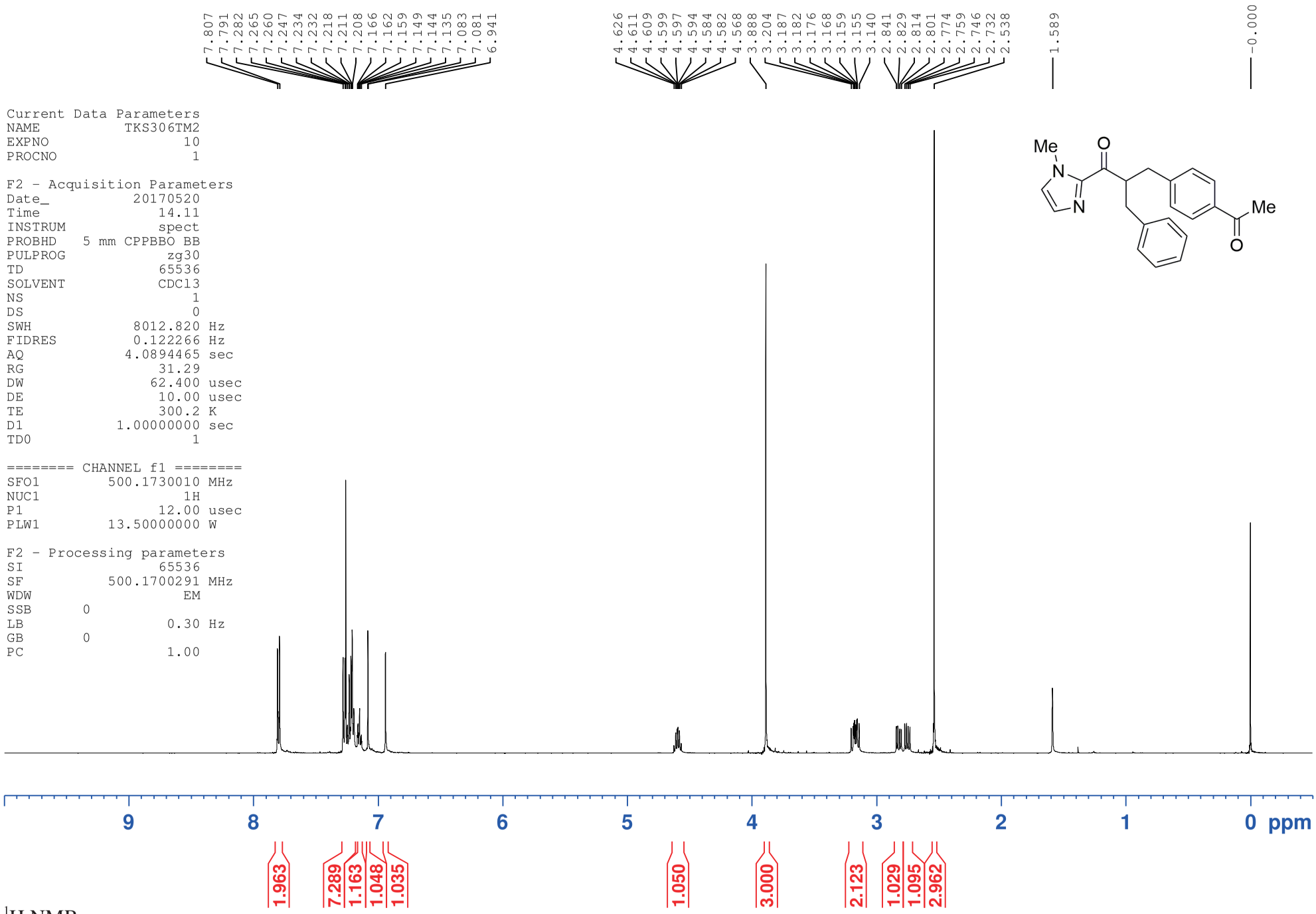
^{13}C NMR


7.889
7.875
7.872
7.530
7.515
7.500
7.418
7.403
7.387
7.325
7.322
7.308
7.305
7.302
7.288
7.272
7.261
7.218
7.212
7.208
7.198
7.184
7.030
4.748
4.740
4.729
4.720
4.711
4.700
4.691
3.975
3.659
3.639
3.623
3.603
3.354
3.344
3.327
3.317
3.090
3.083
3.055
3.047
2.730
2.710
2.703
2.683
1.678
-0.000
Current Data Parameters
NAME TKS297TM2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170511
Time 19.03
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700148 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
O
9
8
7
6
5
4
3
2
1
0 ppm
2.024
1.024
2.056
4.672
1.077
1.007
1.072
1.029
3.000
1.014
1.004
1.023
1.005

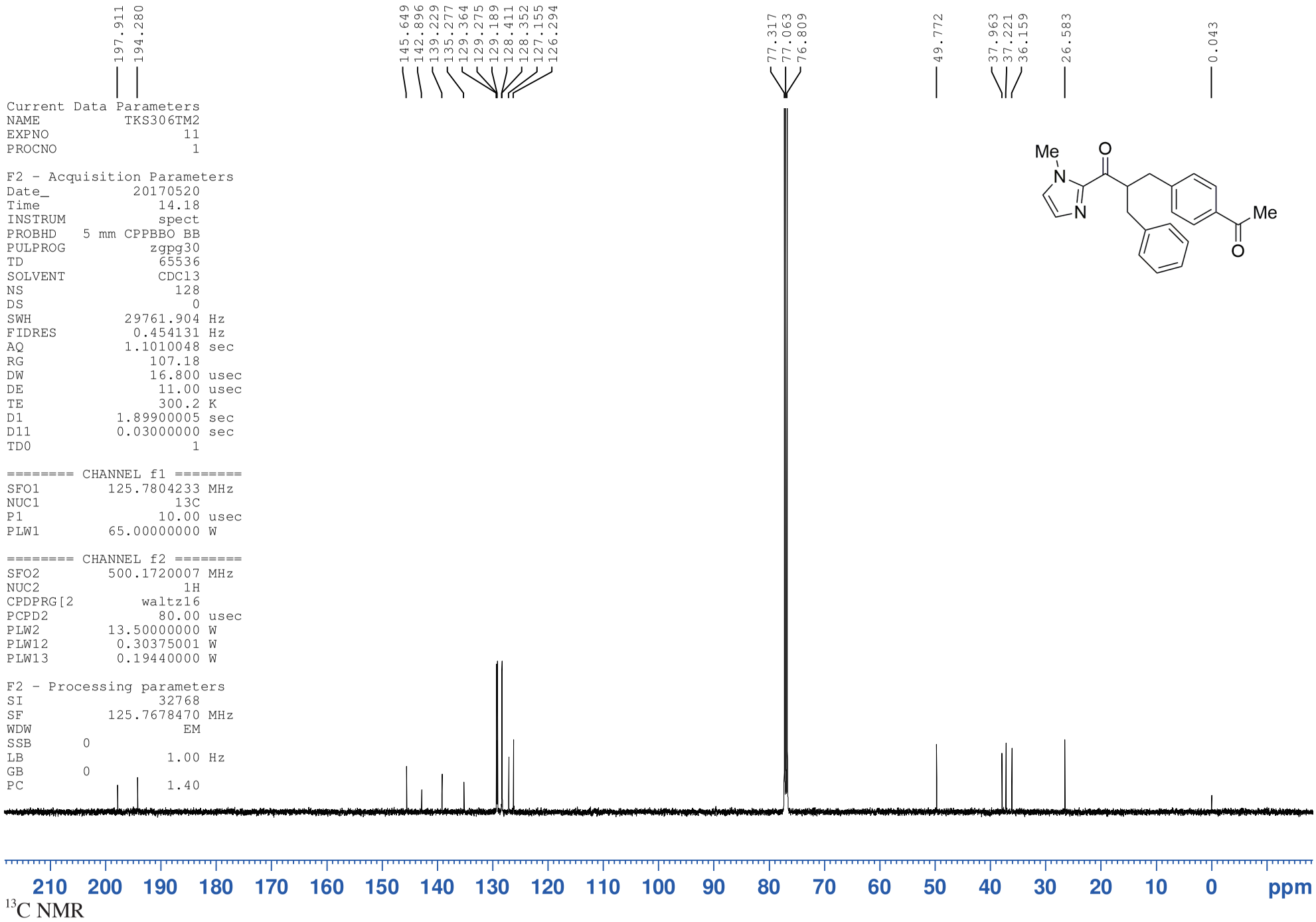
^{1}H NMR


198.334
194.218
142.559
138.996
136.531
133.034
129.329
129.223
128.448
128.053
126.876
126.422
77.270
77.016
76.763
44.261
39.555
37.940
36.171
-0.004
Current Data Parameters
NAME TKS297TM2
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170511
Time 19.07
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.6 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678520 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

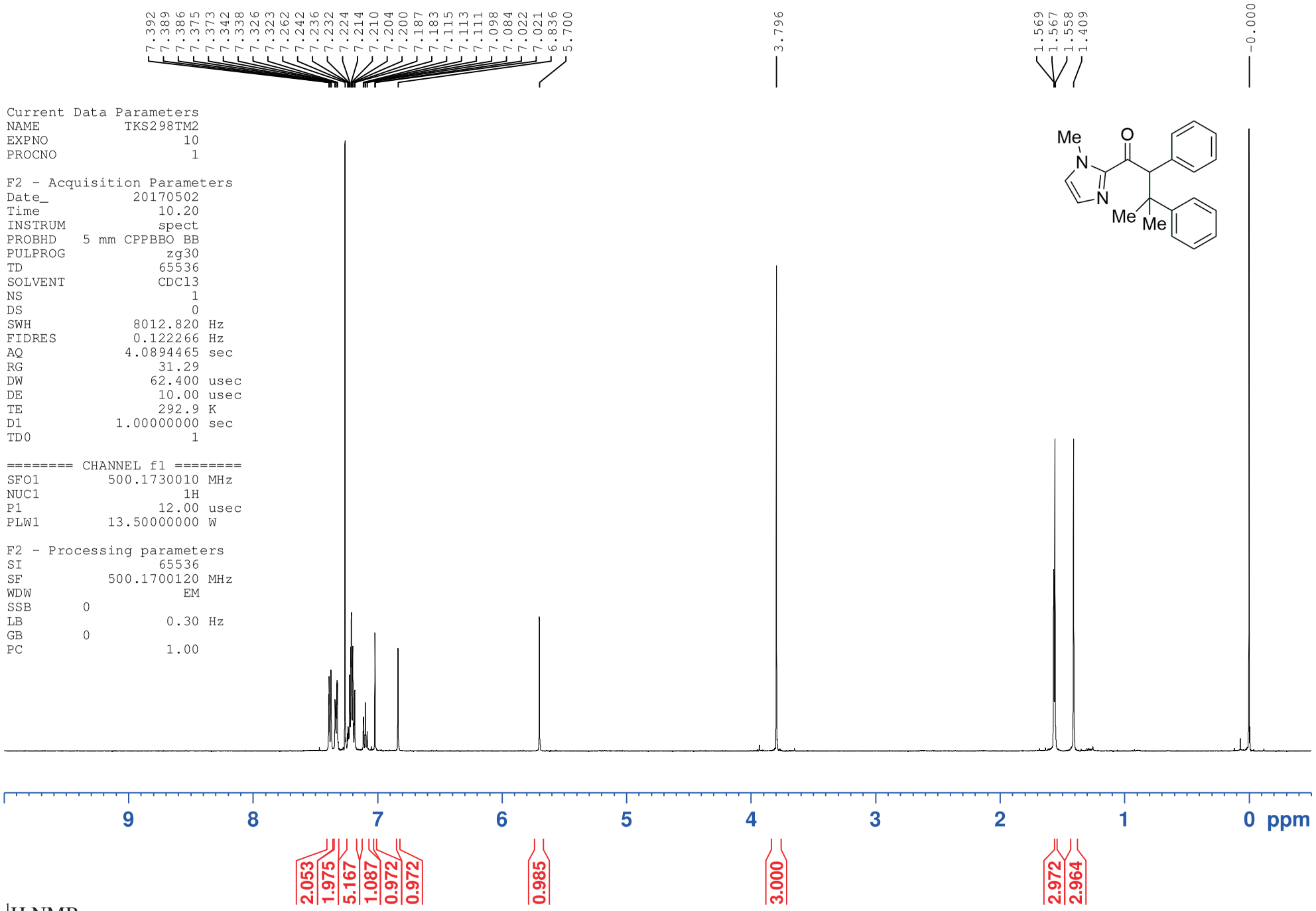
^{13}C NMR


7.807
7.791
7.282
7.265
7.260
7.247
7.234
7.232
7.218
7.211
7.208
7.166
7.162
7.159
7.149
7.144
7.135
7.083
7.081
6.941
4.626
4.611
4.609
4.599
4.597
4.594
4.584
4.582
4.568
3.888
3.204
3.187
3.182
3.176
3.168
3.159
3.155
3.140
2.841
2.829
2.814
2.801
2.774
2.759
2.746
2.732
2.538
1.589
-0.000
Current Data Parameters
NAME TKS306TM2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170520
Time 14.11
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700291 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
O
9
8
7
6
5
4
3
2
1
0 ppm
1.963
7.289
1.163
1.048
1.035
1.050
3.000
2.123
1.029
1.095
2.962

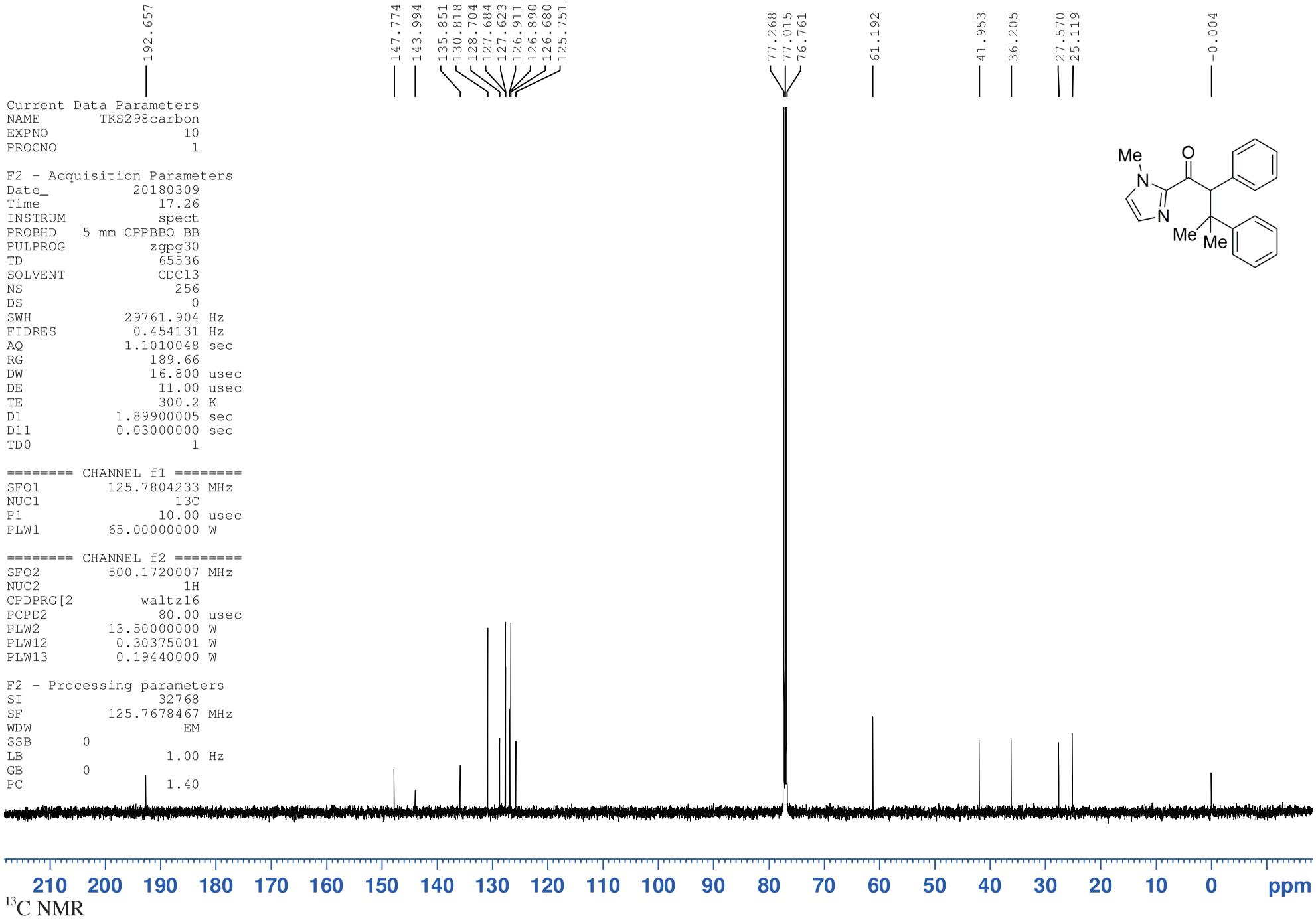
^{1}H NMR


197.911
194.280
145.649
142.896
139.229
135.277
129.364
129.275
129.189
128.411
128.352
127.155
126.294
77.317
77.063
76.809
49.772
37.963
37.221
36.159
26.583
0.043
Current Data Parameters
NAME TKS306TM2
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170520
Time 14.18
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

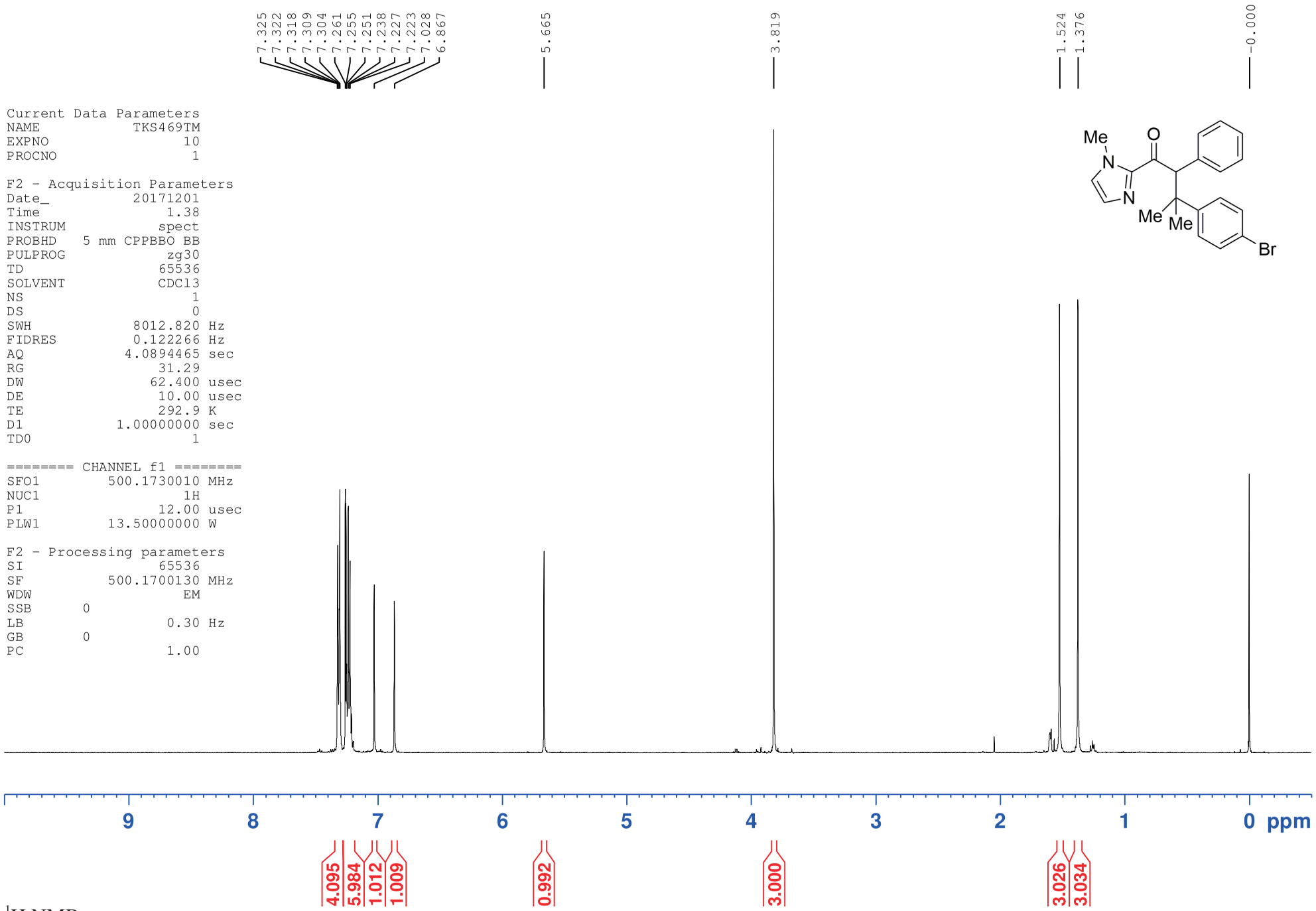
^{13}C NMR


7.392
7.389
7.386
7.375
7.373
7.342
7.338
7.326
7.323
7.262
7.242
7.236
7.232
7.224
7.214
7.210
7.204
7.200
7.187
7.183
7.115
7.113
7.111
7.098
7.084
7.022
7.021
6.836
5.700
3.796
1.569
1.567
1.558
1.409
0.000
Current Data Parameters
NAME TKS298TM2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170502
Time 10.20
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
Me
Me
9
8
7
6
5
4
3
2
1
0 ppm
2.053
1.975
5.167
1.087
0.972
0.972
0.985
3.000
2.972
2.964

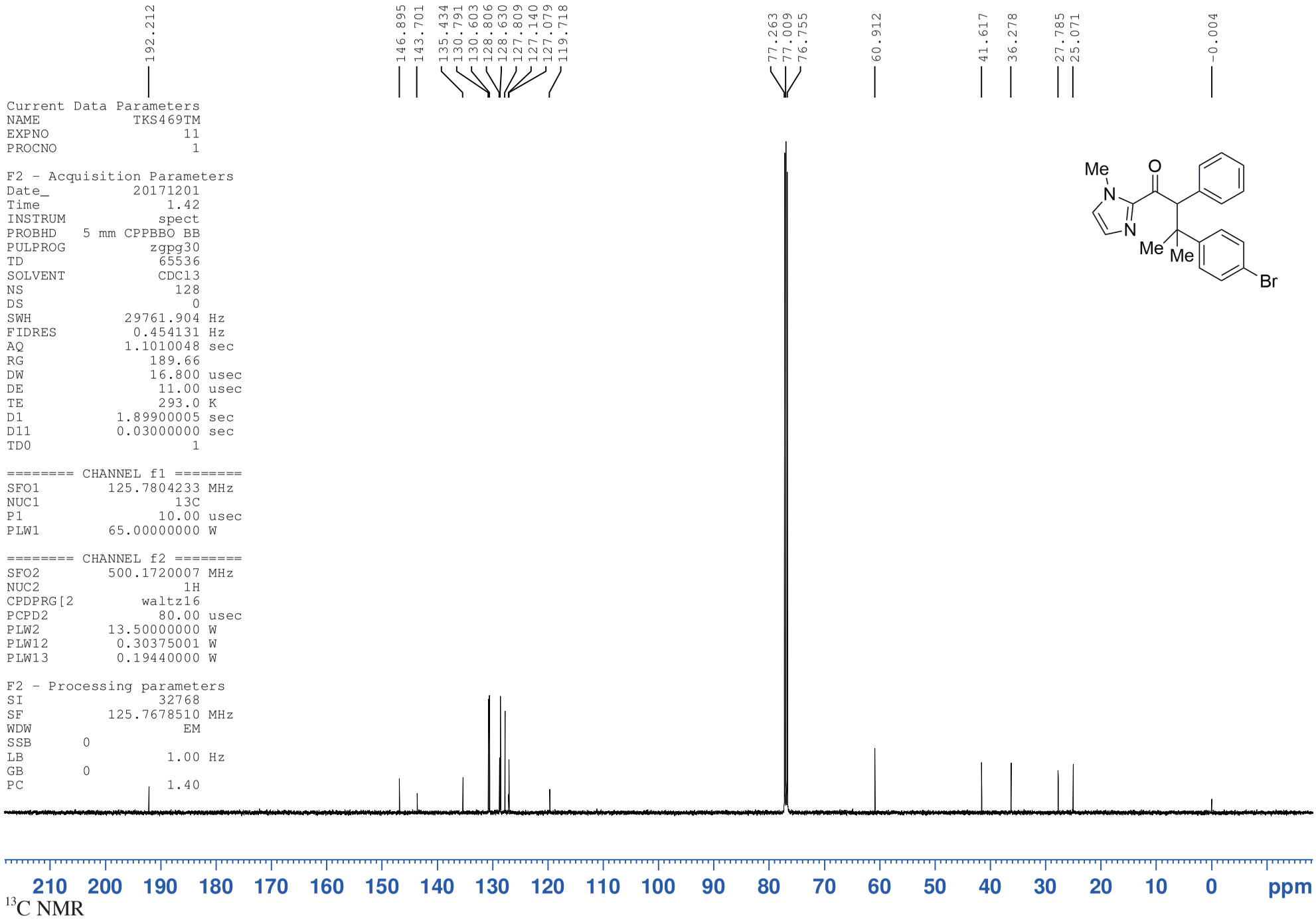
^{1}H NMR


192.657
147.774
143.994
135.851
130.818
128.704
127.684
127.623
126.911
126.890
126.680
125.751
77.268
77.015
76.761
61.192
41.953
36.205
27.570
25.119
-0.004
Current Data Parameters
NAME TKS298carbon
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180309
Time 17.26
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678467 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
N
Me
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

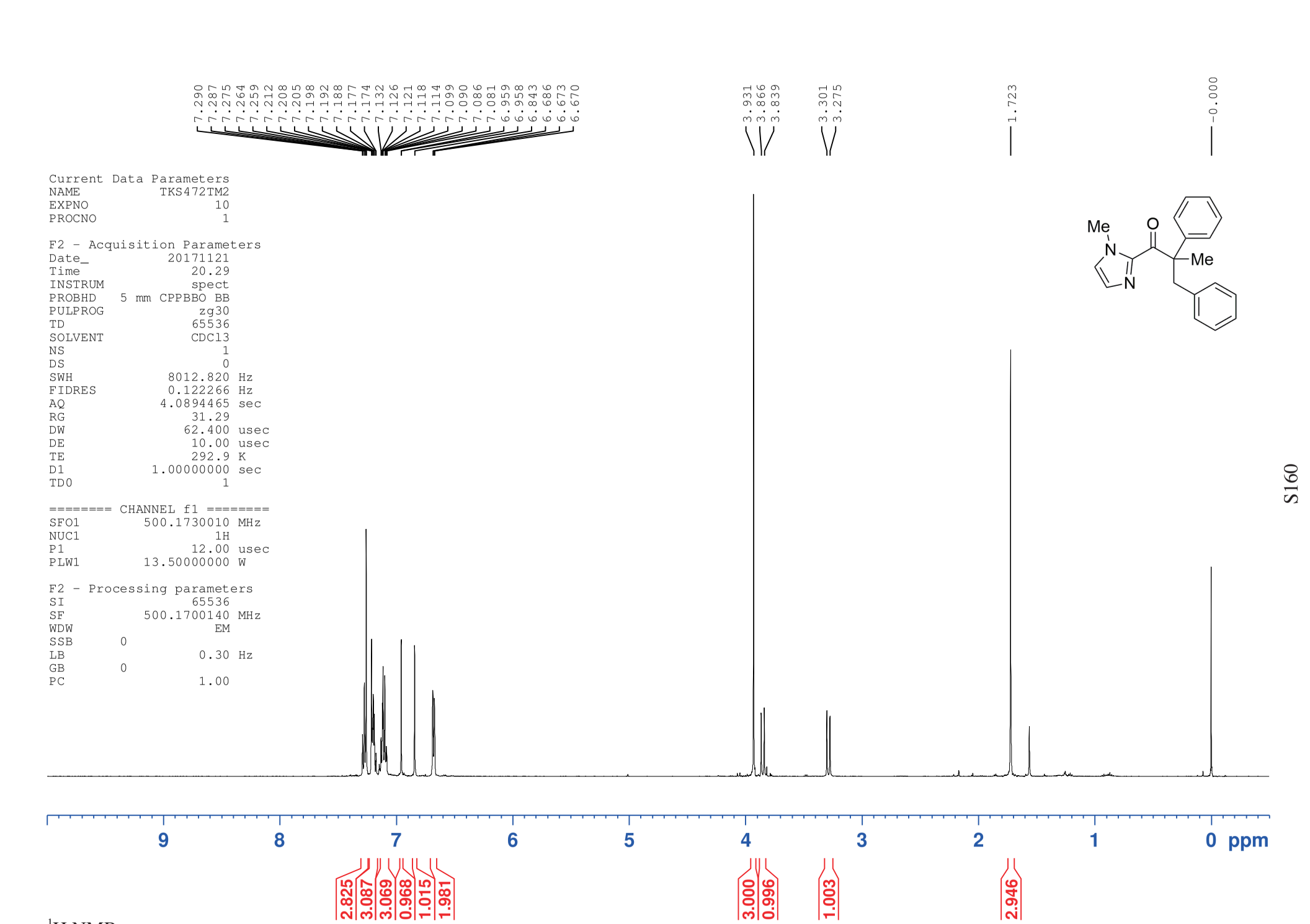
^{13}C NMR


7.325
7.322
7.318
7.309
7.304
7.261
7.255
7.251
7.238
7.227
7.223
7.028
6.867
5.665
3.819
1.524
1.376
0.000
Current Data Parameters
NAME TKS469TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171201
Time 1.38
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
Me
Br
9
8
7
6
5
4
3
2
1
0 ppm
4.095
5.984
1.012
1.009
0.992
3.000
3.026
3.034

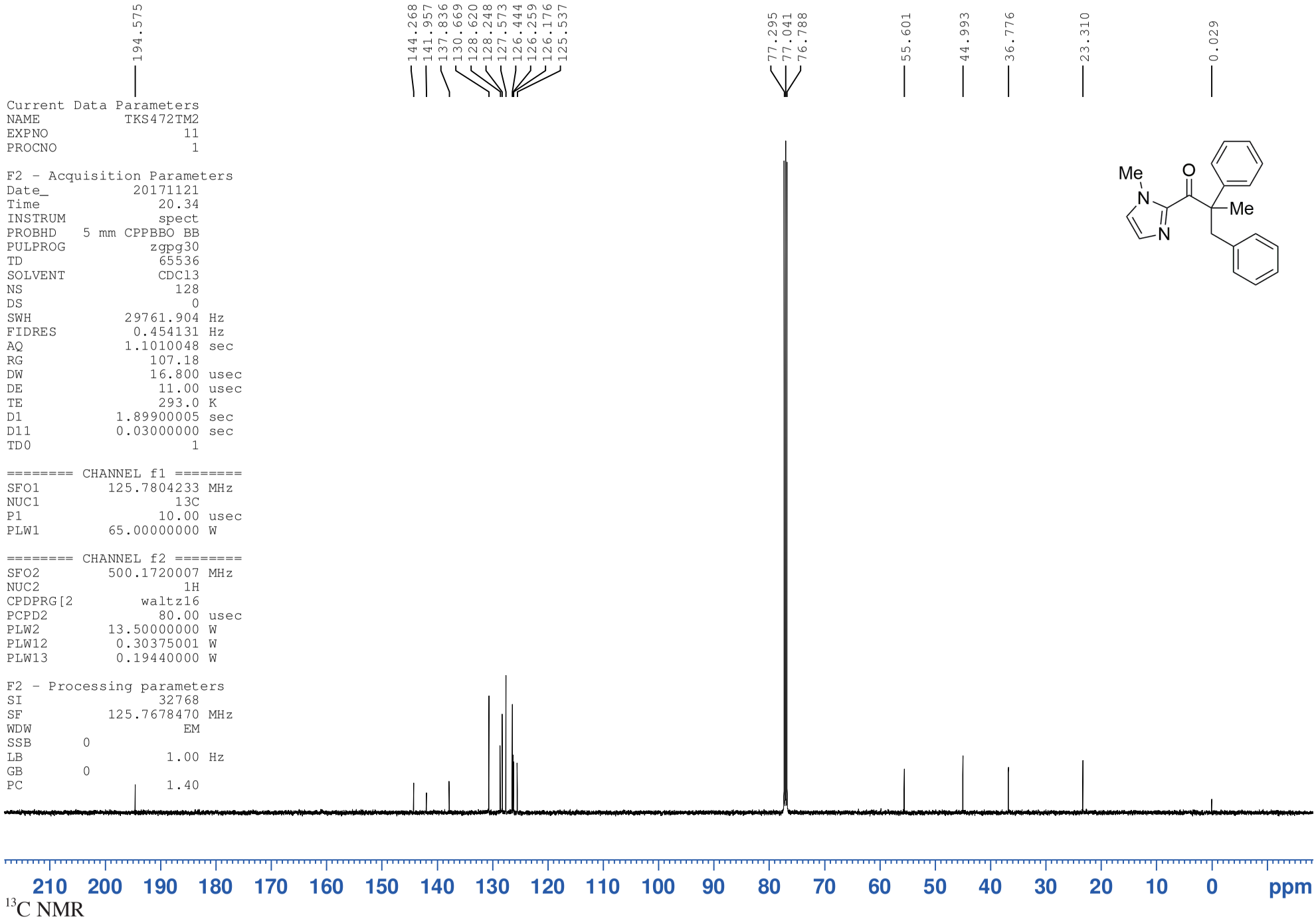
^{1}H NMR


192.212
146.895
143.701
135.434
130.791
130.603
128.806
128.630
127.809
127.140
127.079
119.718
77.263
77.009
76.755
60.912
41.617
36.278
27.785
25.071
-0.004
Current Data Parameters
NAME TKS469TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171201
Time 1.42
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 293.0 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
N
Me
Me
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

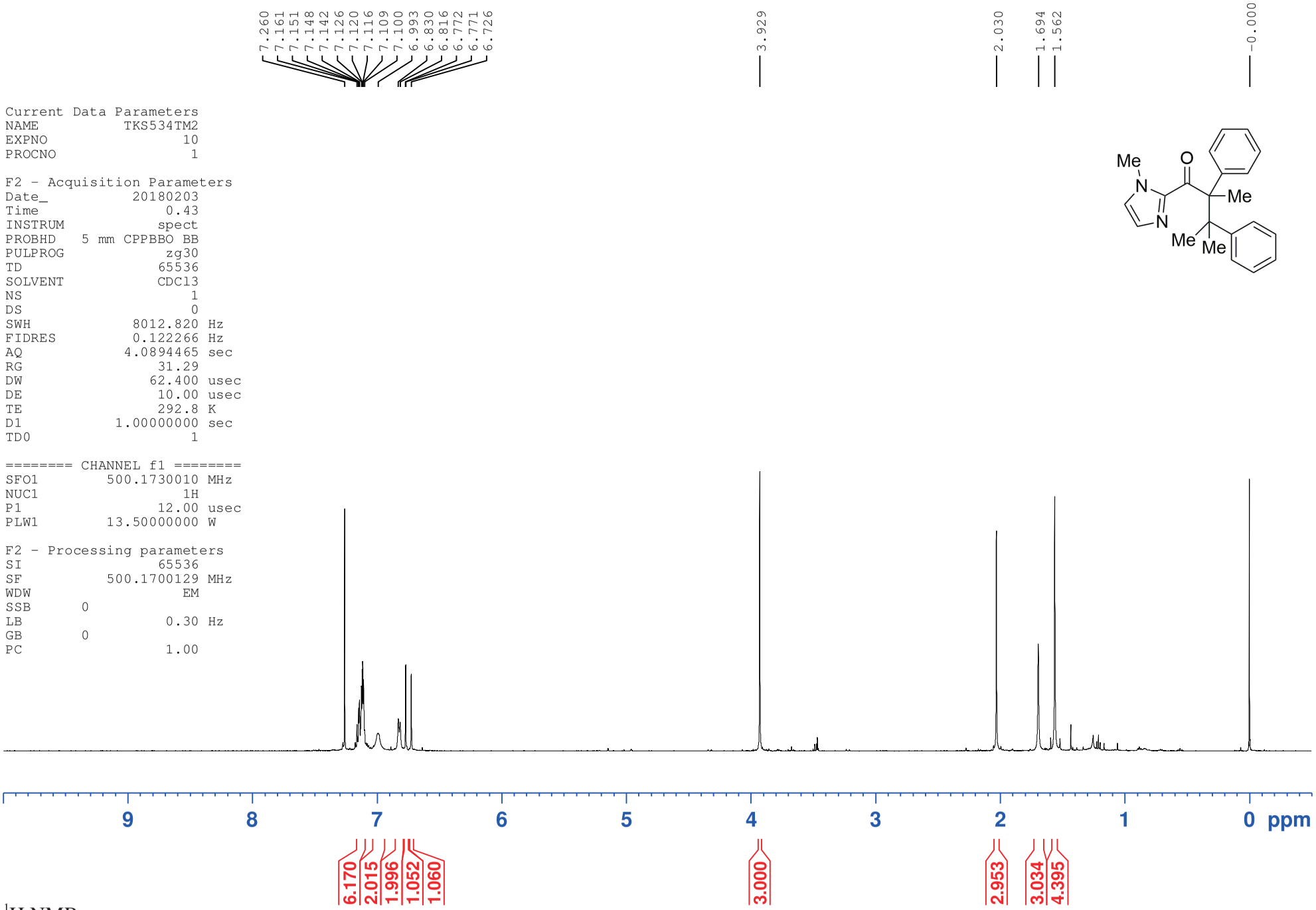
^{13}C NMR


7.290
7.287
7.275
7.264
7.259
7.212
7.208
7.205
7.198
7.192
7.188
7.177
7.174
7.132
7.126
7.121
7.118
7.114
7.099
7.090
7.086
7.081
6.959
6.958
6.843
6.686
6.673
6.670
3.931
3.866
3.839
3.301
3.275
1.723
-0.000
Current Data Parameters
NAME TKS472TM2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171121
Time 20.29
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
9
8
7
6
5
4
3
2
1
0 ppm
2.825
3.087
3.069
0.968
1.015
1.981
3.000
0.996
1.003
2.946

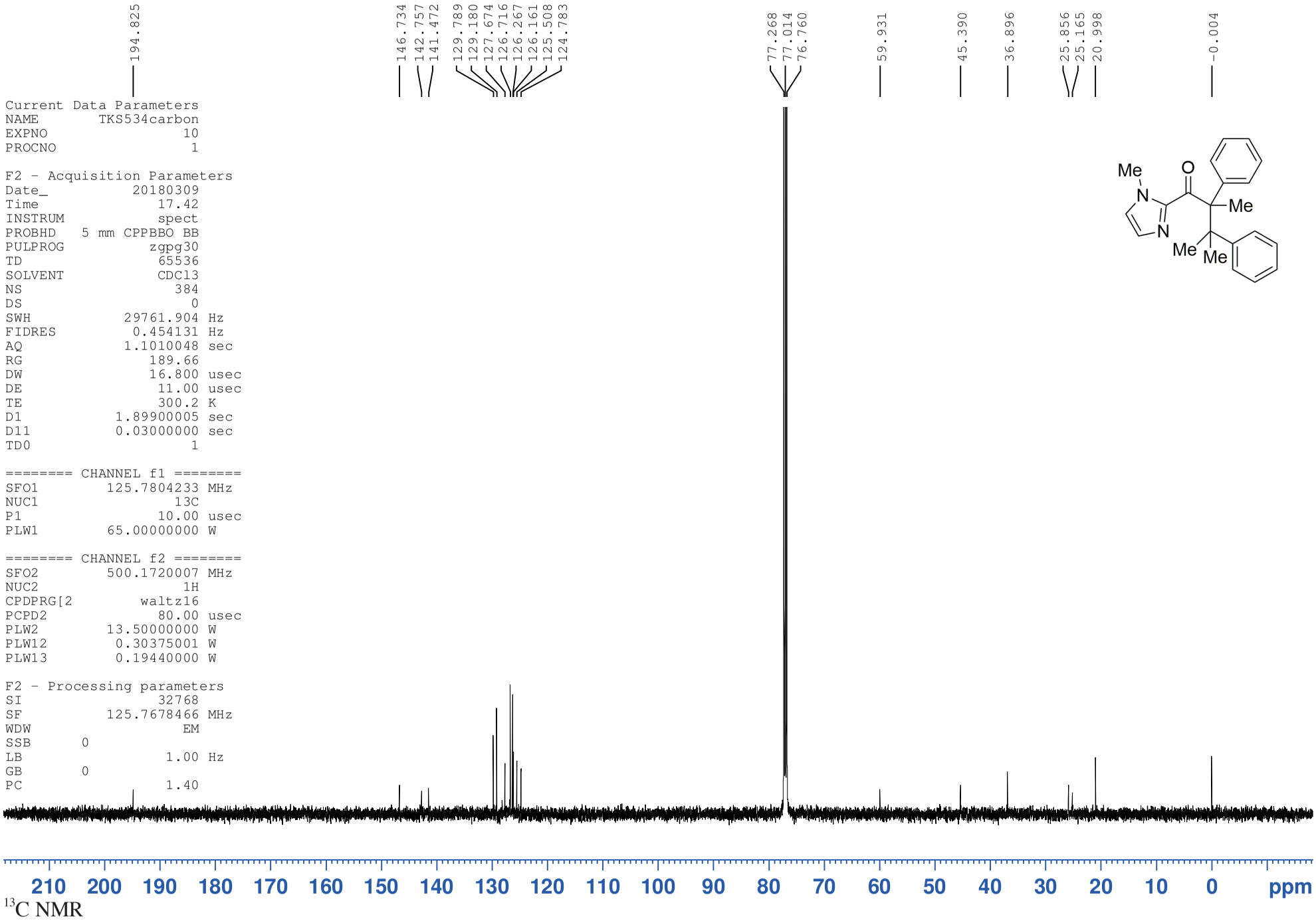
^{1}H NMR


194.575
144.268
141.957
137.836
130.669
128.620
128.248
127.573
126.444
126.259
126.176
125.537
77.295
77.041
76.788
55.601
44.993
36.776
23.310
0.029
Current Data Parameters
NAME TKS472TM2
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171121
Time 20.34
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 293.0 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

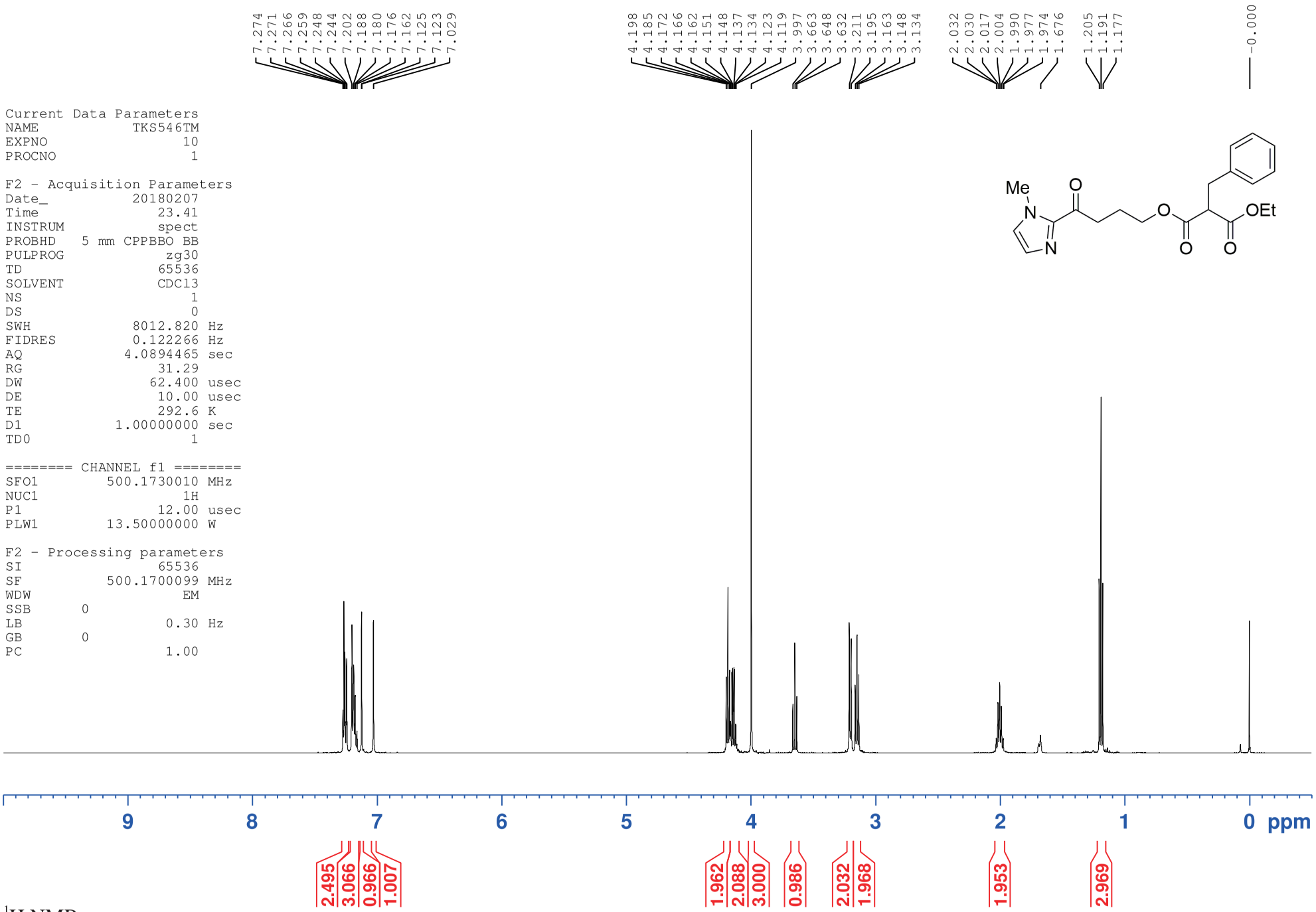
^{13}C NMR


7.260
7.161
7.151
7.148
7.142
7.126
7.120
7.116
7.109
7.100
6.993
6.830
6.816
6.772
6.771
6.726
3.929
2.030
1.694
1.562
-0.000
Current Data Parameters
NAME TKS534TM2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180203
Time 0.43
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
Me
Me
Me
9
8
7
6
5
4
3
2
1
0 ppm
6.170
2.015
1.996
1.052
1.060
3.000
2.953
3.034
4.395

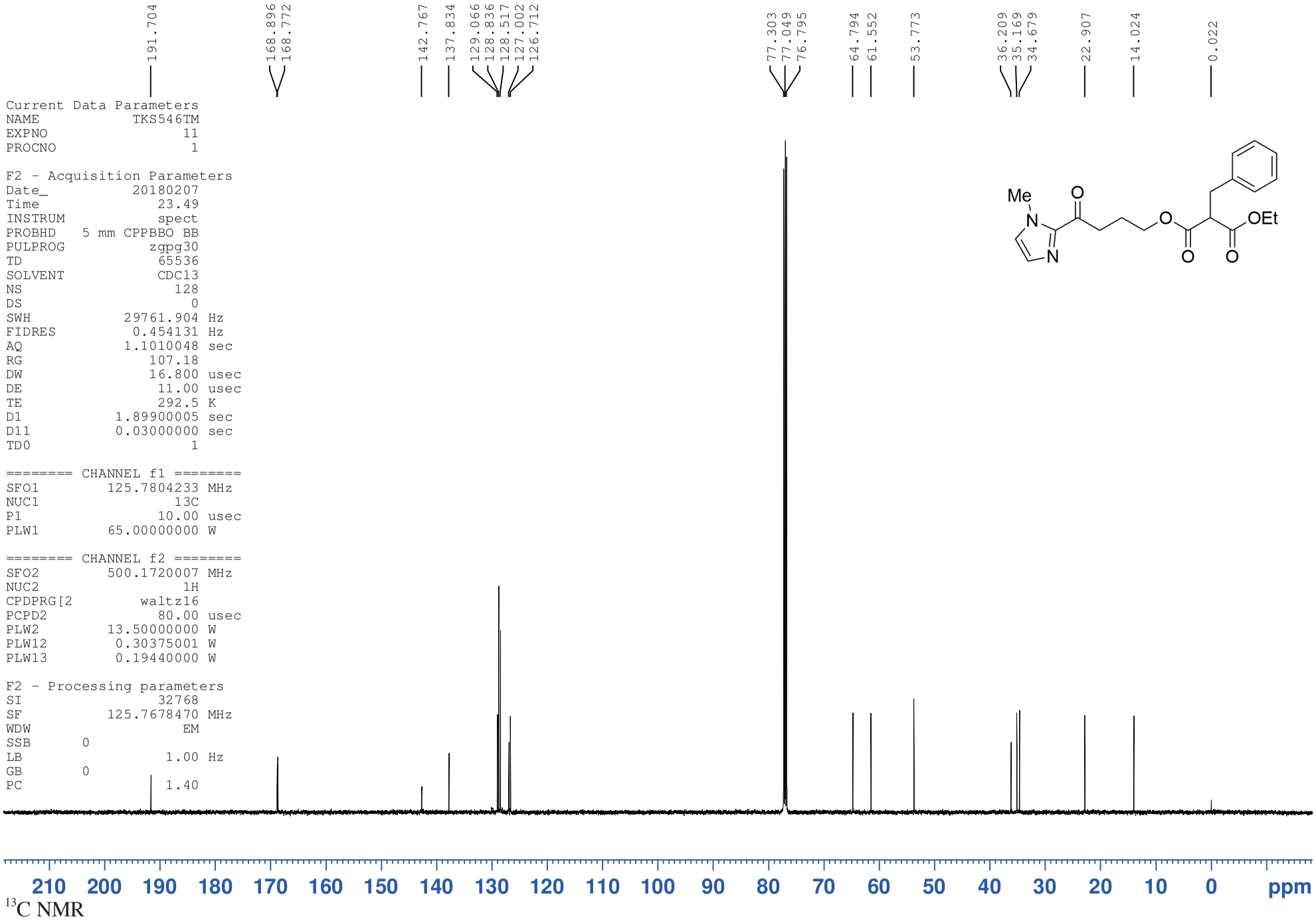
^{1}H NMR


194.825
146.734
142.757
141.472
129.789
129.180
127.674
126.716
126.267
126.161
125.508
124.783
77.268
77.014
76.760
59.931
45.390
36.896
25.856
25.165
20.998
-0.004
Current Data Parameters
NAME TKS534carbon
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180309
Time 17.42
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678466 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
N
Me
Me
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

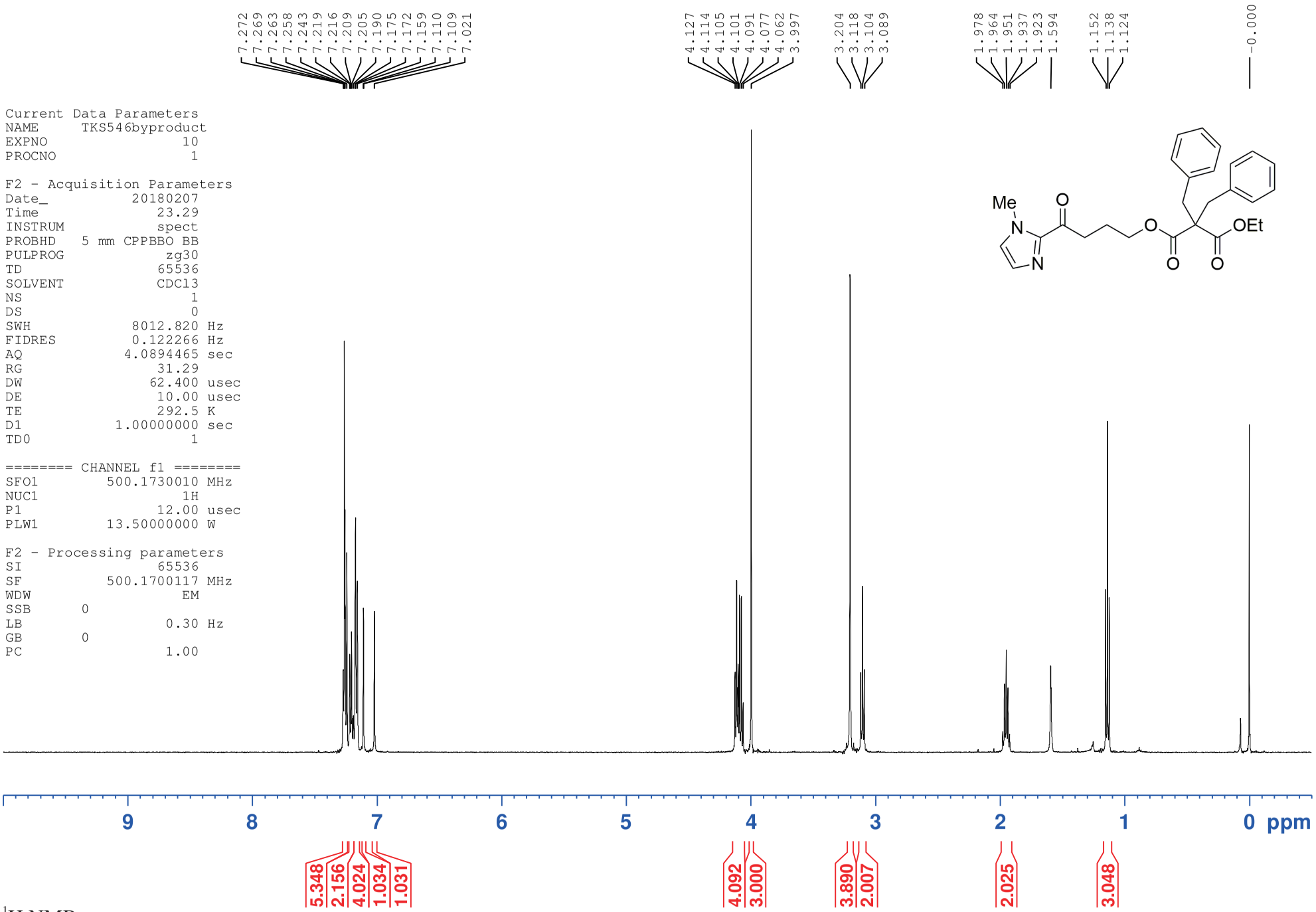
^{13}C NMR


7.274
7.271
7.266
7.259
7.248
7.244
7.202
7.188
7.180
7.176
7.162
7.125
7.123
7.029
4.198
4.185
4.172
4.166
4.162
4.151
4.148
4.137
4.134
4.123
4.119
3.997
3.663
3.648
3.632
3.211
3.195
3.163
3.148
3.134
2.032
2.030
2.017
2.004
1.990
1.977
1.974
1.676
1.205
1.191
1.177
0.000
Current Data Parameters
NAME TKS546TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180207
Time 23.41
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
O
O
O
OEt
9
8
7
6
5
4
3
2
1
0 ppm
2.495
3.066
0.966
1.007
1.962
2.088
3.000
0.986
2.032
1.968
1.953
2.969

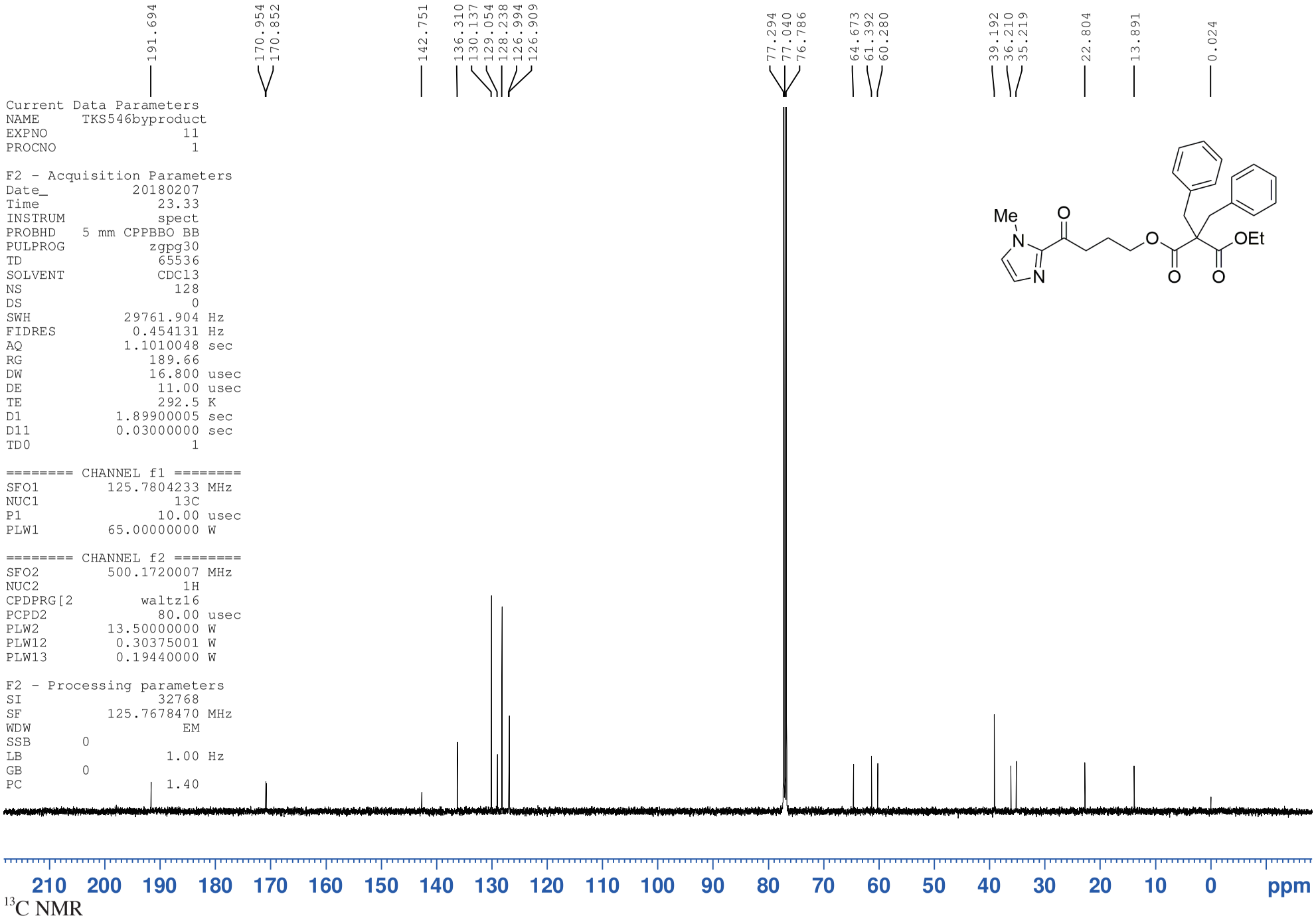
^{1}H NMR


191.704
168.896
168.772
142.767
137.834
129.066
128.836
128.517
127.002
126.712
77.303
77.049
76.795
64.794
61.552
53.773
36.209
35.169
34.679
22.907
14.024
0.022
Current Data Parameters
NAME TKS546TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180207
Time 23.49
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 292.5 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
O
O
OEt
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

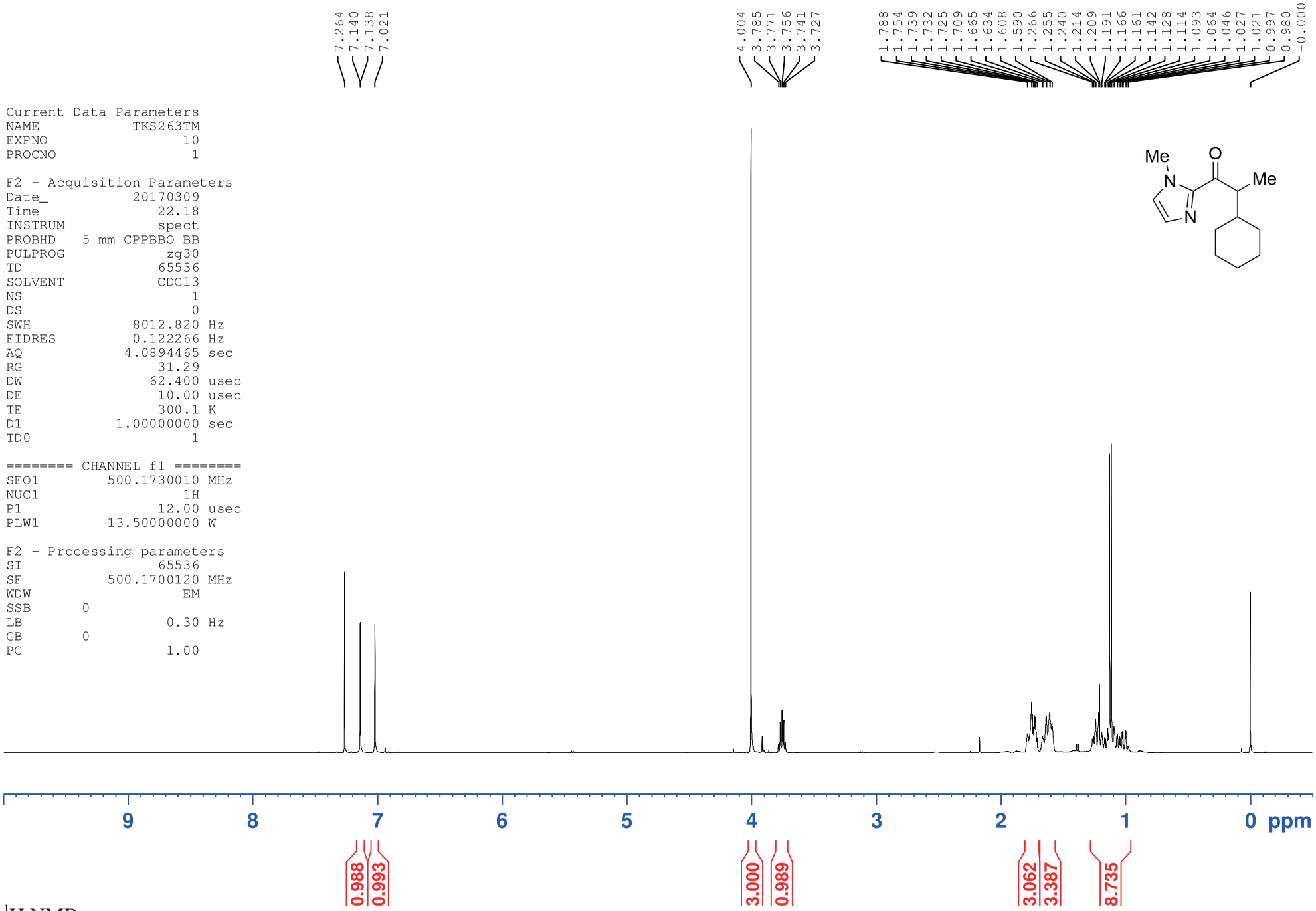
^{13}C NMR


7.272
7.269
7.263
7.258
7.243
7.219
7.216
7.209
7.205
7.190
7.175
7.172
7.159
7.110
7.109
7.021
4.127
4.114
4.105
4.101
4.091
4.077
4.062
3.997
3.204
3.118
3.104
3.089
1.978
1.964
1.951
1.937
1.923
1.594
1.152
1.138
1.124
-0.000
Current Data Parameters
NAME TKS546byproduct
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180207
Time 23.29
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
O
O
O
OEt
9
8
7
6
5
4
3
2
1
0 ppm
5.348
2.156
4.024
1.034
1.031
4.092
3.000
3.890
2.007
2.025
3.048

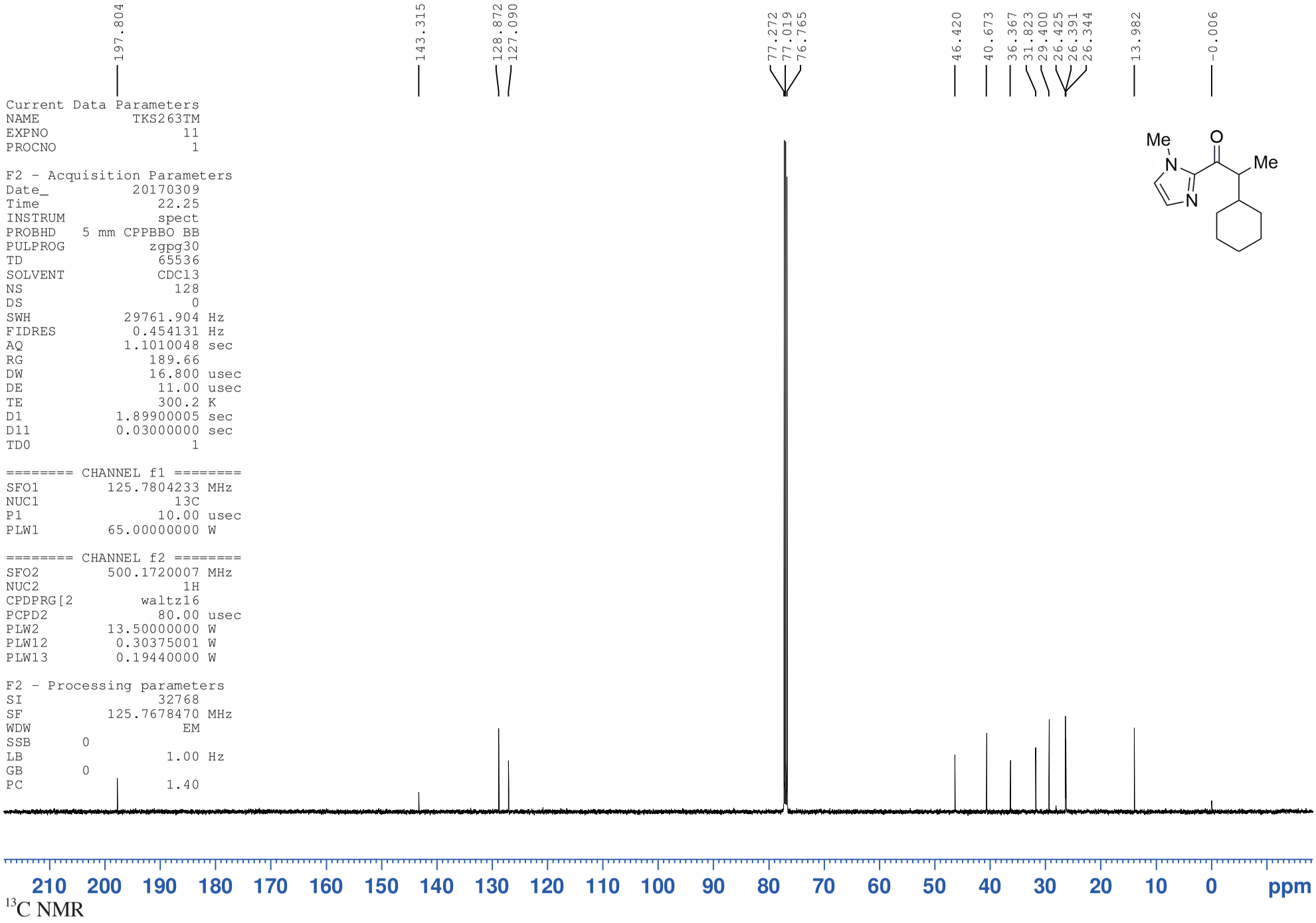
^{1}H NMR


191.694
170.954
170.852
142.751
136.310
130.137
129.054
128.238
126.994
126.909
77.294
77.040
76.786
64.673
61.392
60.280
39.192
36.210
35.219
22.804
13.891
0.024
Current Data Parameters
NAME TKS546byproduct
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180207
Time 23.33
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 292.5 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
O
O
O
OEt
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

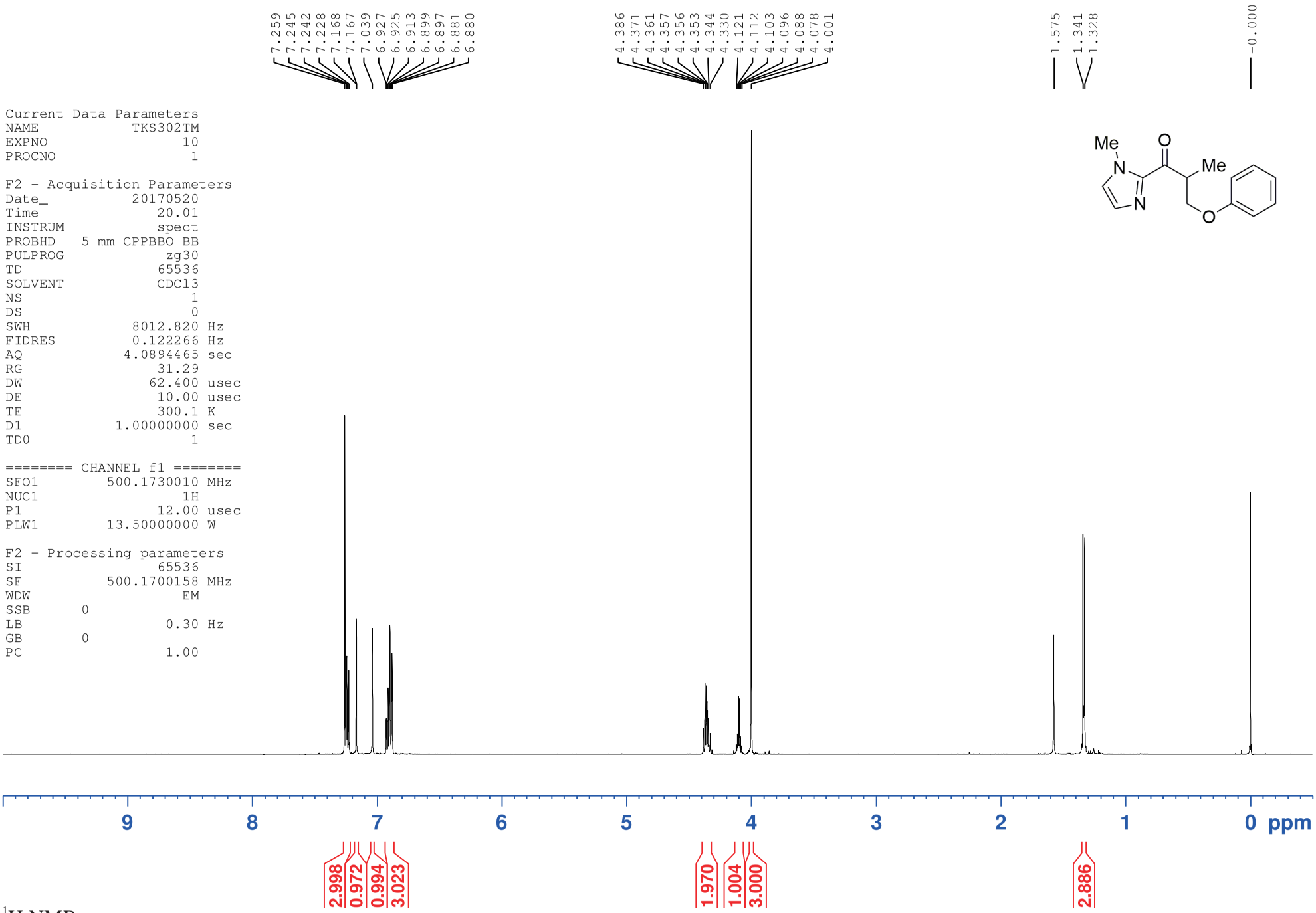
^{13}C NMR


7.264
7.140
7.138
7.021
4.004
3.785
3.771
3.756
3.741
3.727
1.788
1.754
1.739
1.732
1.725
1.709
1.665
1.634
1.608
1.590
1.266
1.255
1.240
1.214
1.209
1.191
1.166
1.161
1.142
1.128
1.114
1.093
1.064
1.046
1.027
1.021
0.997
0.980
-0.000
Current Data Parameters
NAME TKS263TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170309
Time 22.18
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
Me
N
9
8
7
6
5
4
3
2
1
0 ppm
0.988
0.993
3.000
0.989
3.062
3.387
8.735

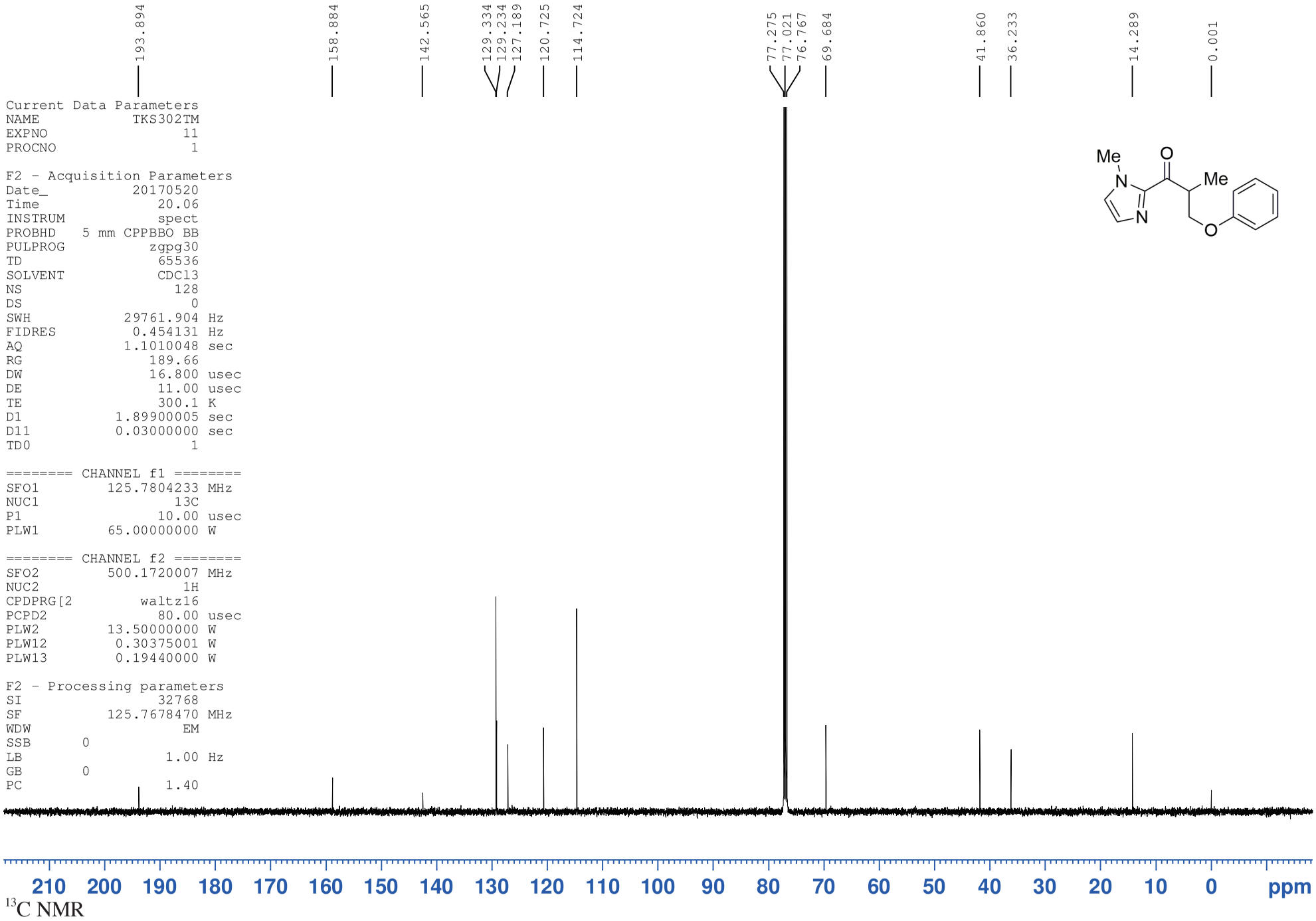
^{1}H NMR


197.804
143.315
128.872
127.090
77.272
77.019
76.765
46.420
40.673
36.367
31.823
29.400
26.425
26.391
26.344
13.982
-0.006
Current Data Parameters
NAME TKS263TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170309
Time 22.25
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

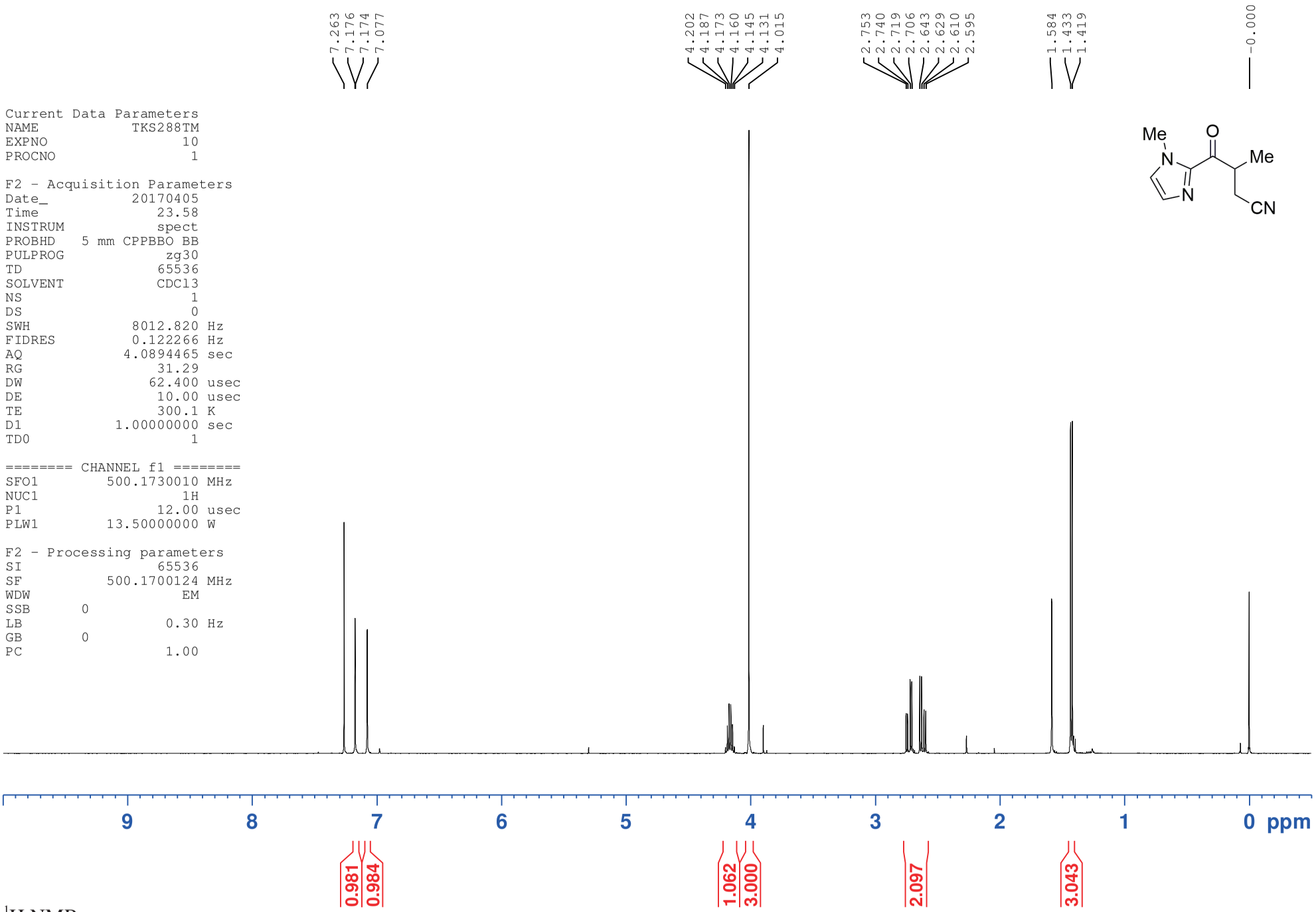
^{13}C NMR


7.259
7.245
7.242
7.228
7.168
7.167
7.039
6.927
6.925
6.913
6.899
6.897
6.881
6.880
4.386
4.371
4.361
4.357
4.356
4.353
4.344
4.330
4.121
4.112
4.103
4.096
4.088
4.078
4.001
1.575
1.341
1.328
0.000
Current Data Parameters
NAME TKS302TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170520
Time 20.01
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700158 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
O
9
8
7
6
5
4
3
2
1
0 ppm
2.998
0.972
0.994
3.023
1.970
1.004
3.000
2.886

^{1}H NMR

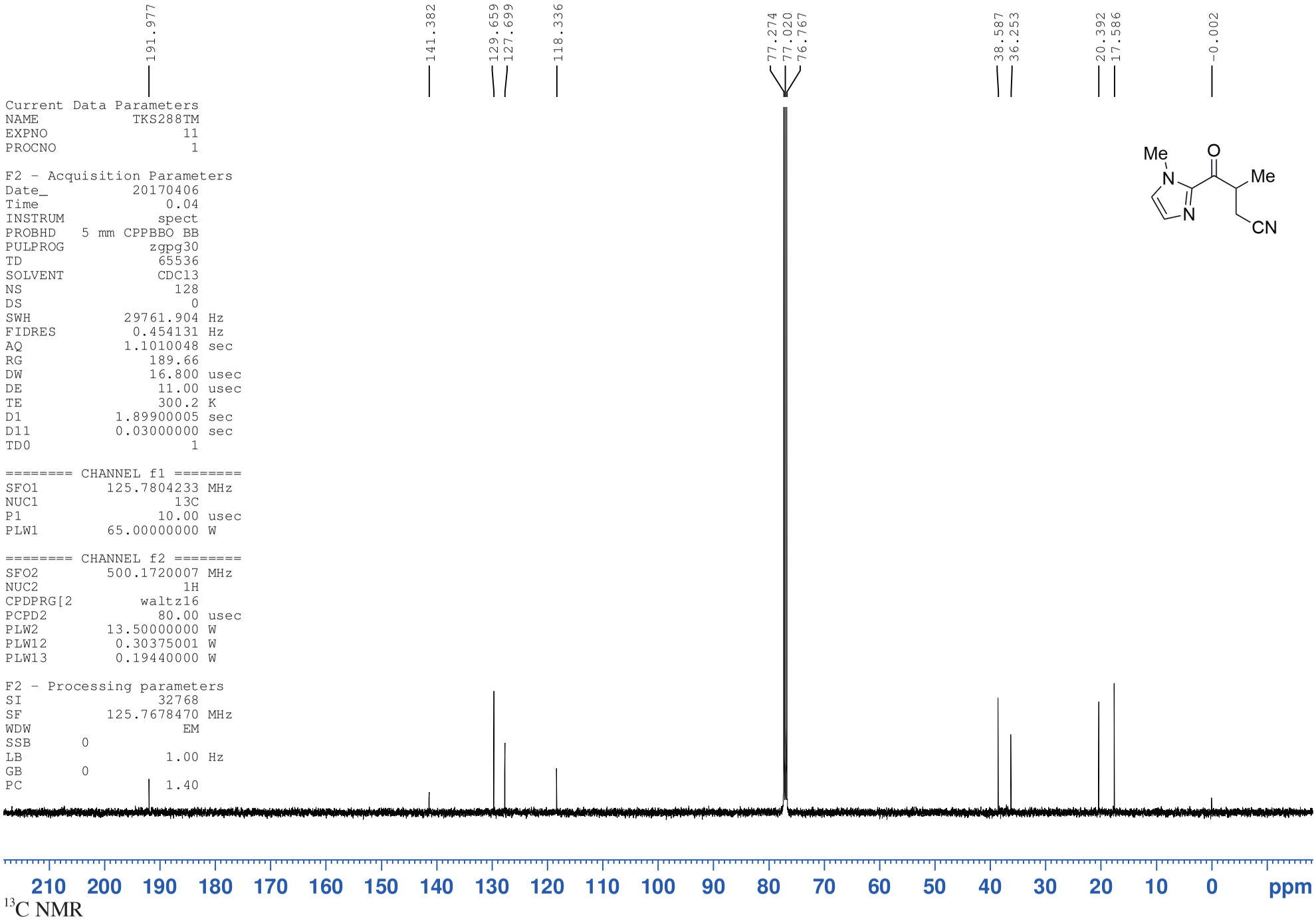

193.894
158.884
142.565
129.334
129.234
127.189
120.725
114.724
77.275
77.021
76.767
69.684
41.860
36.233
14.289
0.001
Current Data Parameters
NAME TKS302TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170520
Time 20.06
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
N
O
Me
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

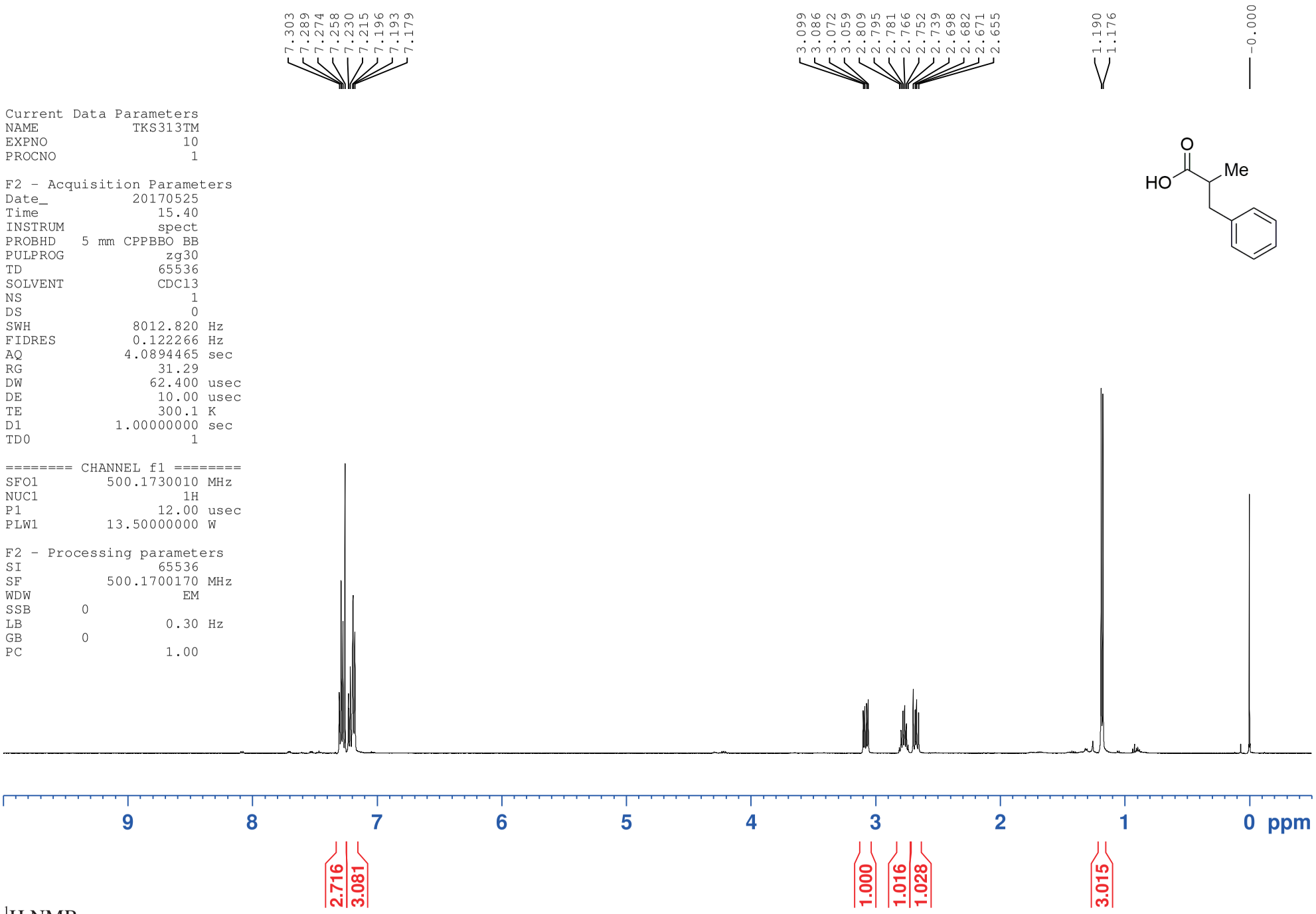

7.263
7.176
7.174
7.077
4.202
4.187
4.173
4.160
4.145
4.131
4.015
2.753
2.740
2.719
2.706
2.643
2.629
2.610
2.595
1.584
1.433
1.419
-0.000
Current Data Parameters
NAME TKS288TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170405
Time 23.58
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
N
O
Me
CN
9
8
7
6
5
4
3
2
1
0 ppm
0.981
0.984
1.062
3.000
2.097
3.043

S172

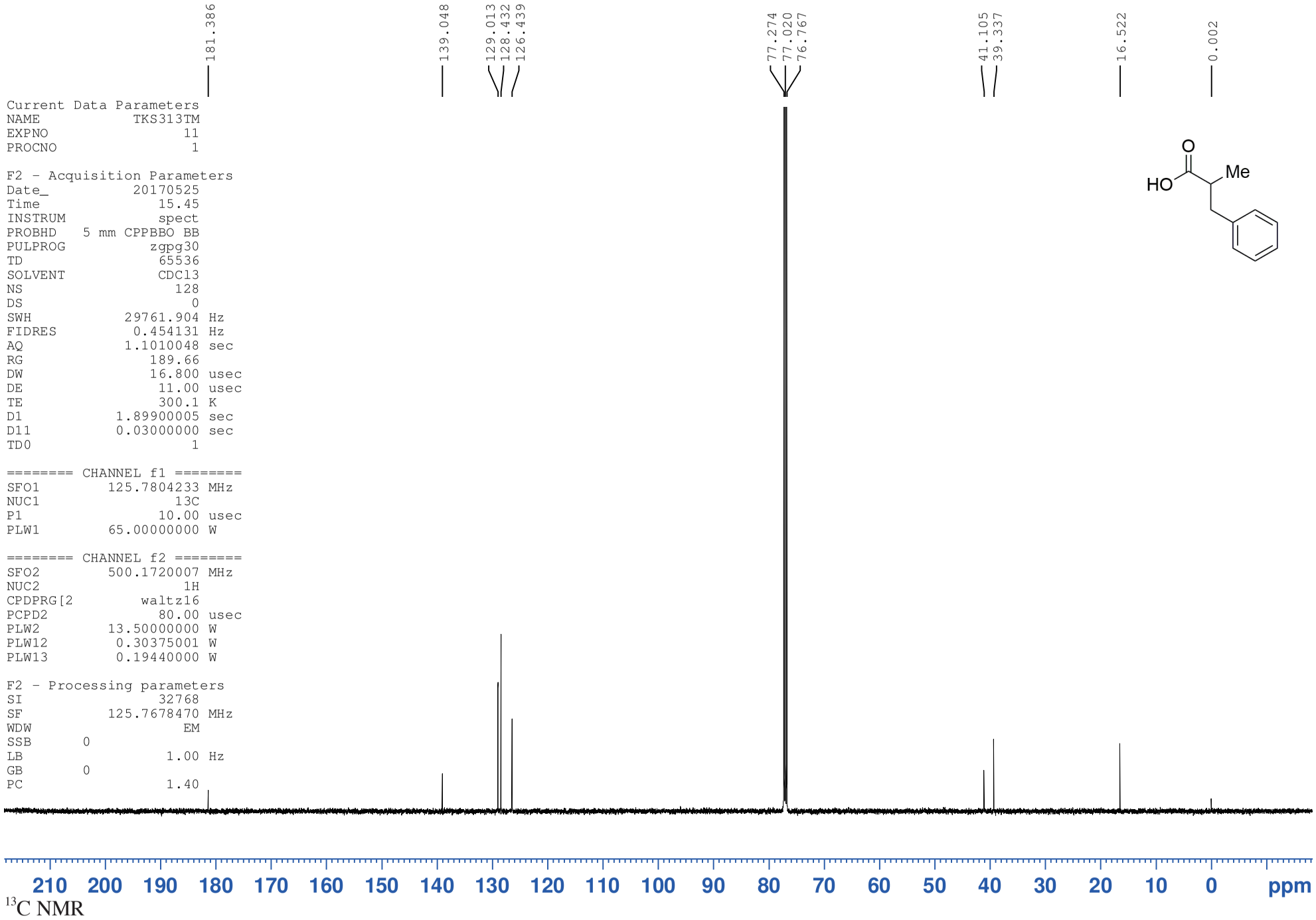
^{1}H NMR


191.977
141.382
129.659
127.699
118.336
77.274
77.020
76.767
38.587
36.253
20.392
17.586
-0.002
Current Data Parameters
NAME TKS288TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170406
Time 0.04
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.2 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
N
O
Me
N
CN
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

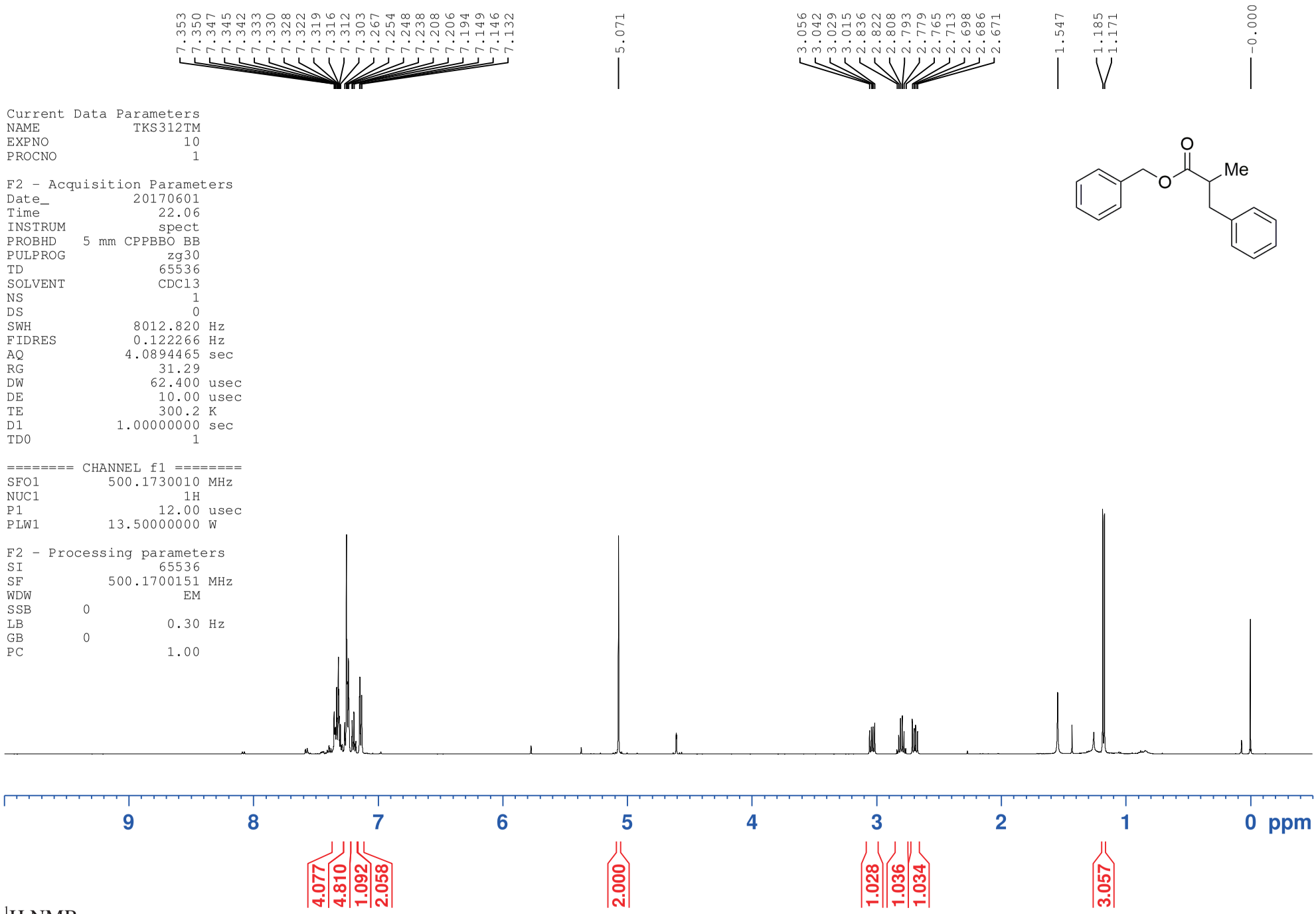
^{13}C NMR


7.303
7.289
7.274
7.258
7.230
7.215
7.196
7.193
7.179
3.099
3.086
3.072
3.059
2.809
2.795
2.781
2.766
2.752
2.739
2.698
2.682
2.671
2.655
1.190
1.176
-0.000
O
HO
Me
Current Data Parameters
NAME TKS313TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170525
Time 15.40
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700170 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
2.716
3.081
1.000
1.016
1.028
3.015

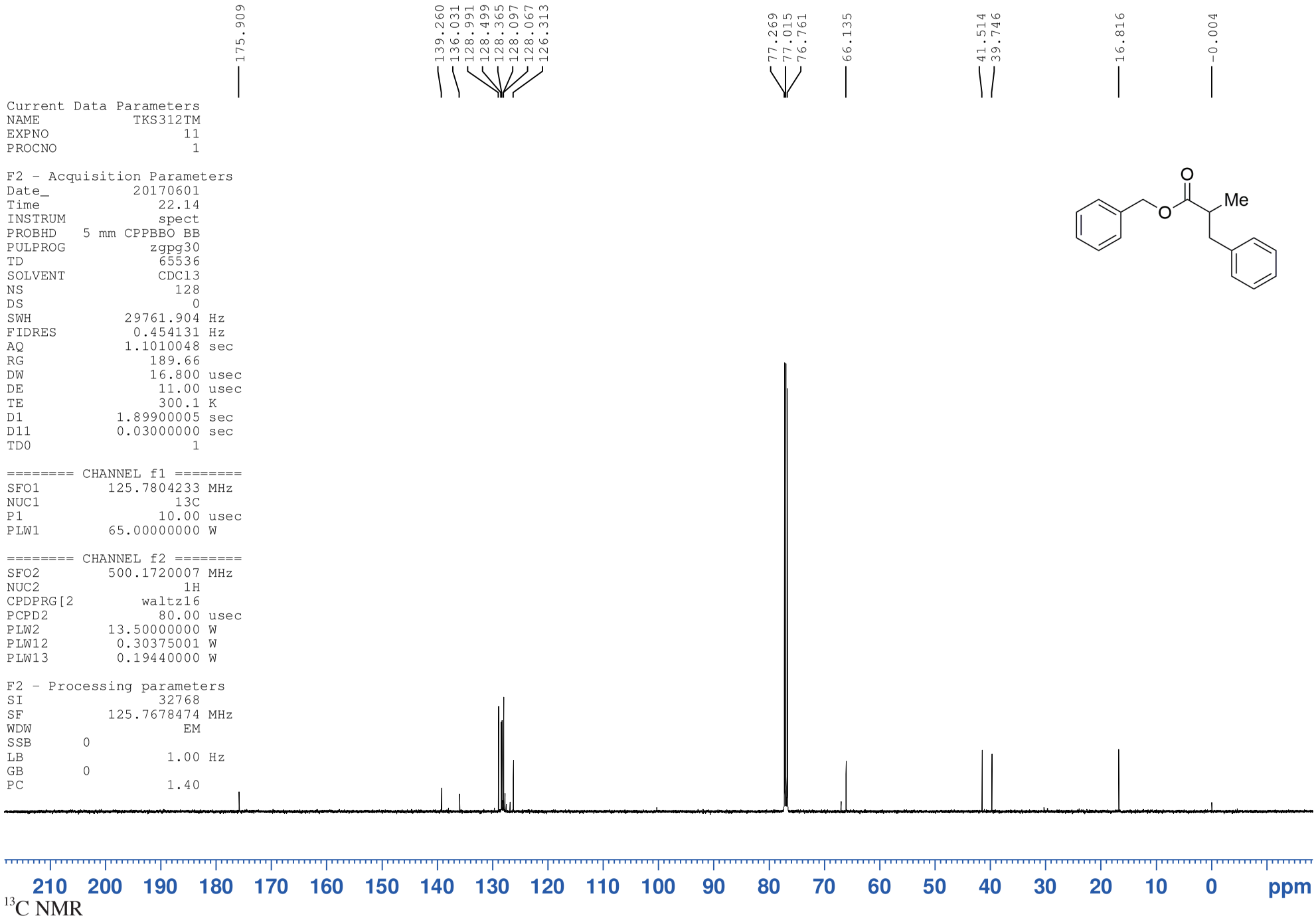
^{1}H NMR


181.386
139.048
129.013
128.432
126.439
77.274
77.020
76.767
41.105
39.337
16.522
0.002
Current Data Parameters
NAME TKS313TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170525
Time 15.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
HO
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

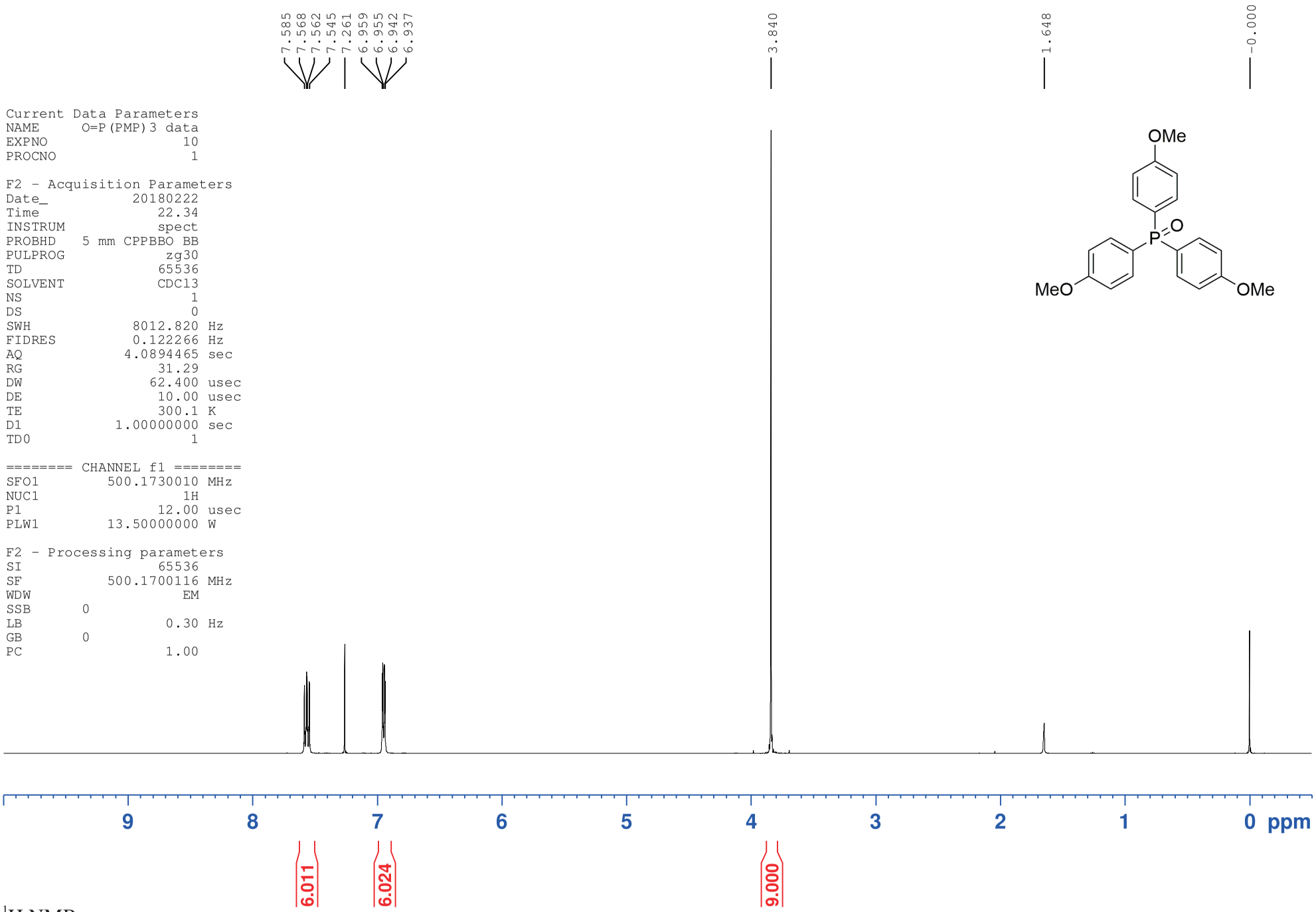
^{13}C NMR


7.353
7.350
7.347
7.345
7.342
7.333
7.330
7.328
7.322
7.319
7.316
7.312
7.303
7.267
7.254
7.248
7.238
7.208
7.206
7.194
7.149
7.146
7.132
5.071
3.056
3.042
3.029
3.015
2.836
2.822
2.808
2.793
2.779
2.765
2.713
2.698
2.686
2.671
1.547
1.185
1.171
0.000
Current Data Parameters
NAME TKS312TM
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170601
Time 22.06
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
Me
9
8
7
6
5
4
3
2
1
0 ppm
4.077
4.810
1.092
2.058
2.000
1.028
1.036
1.034
3.057

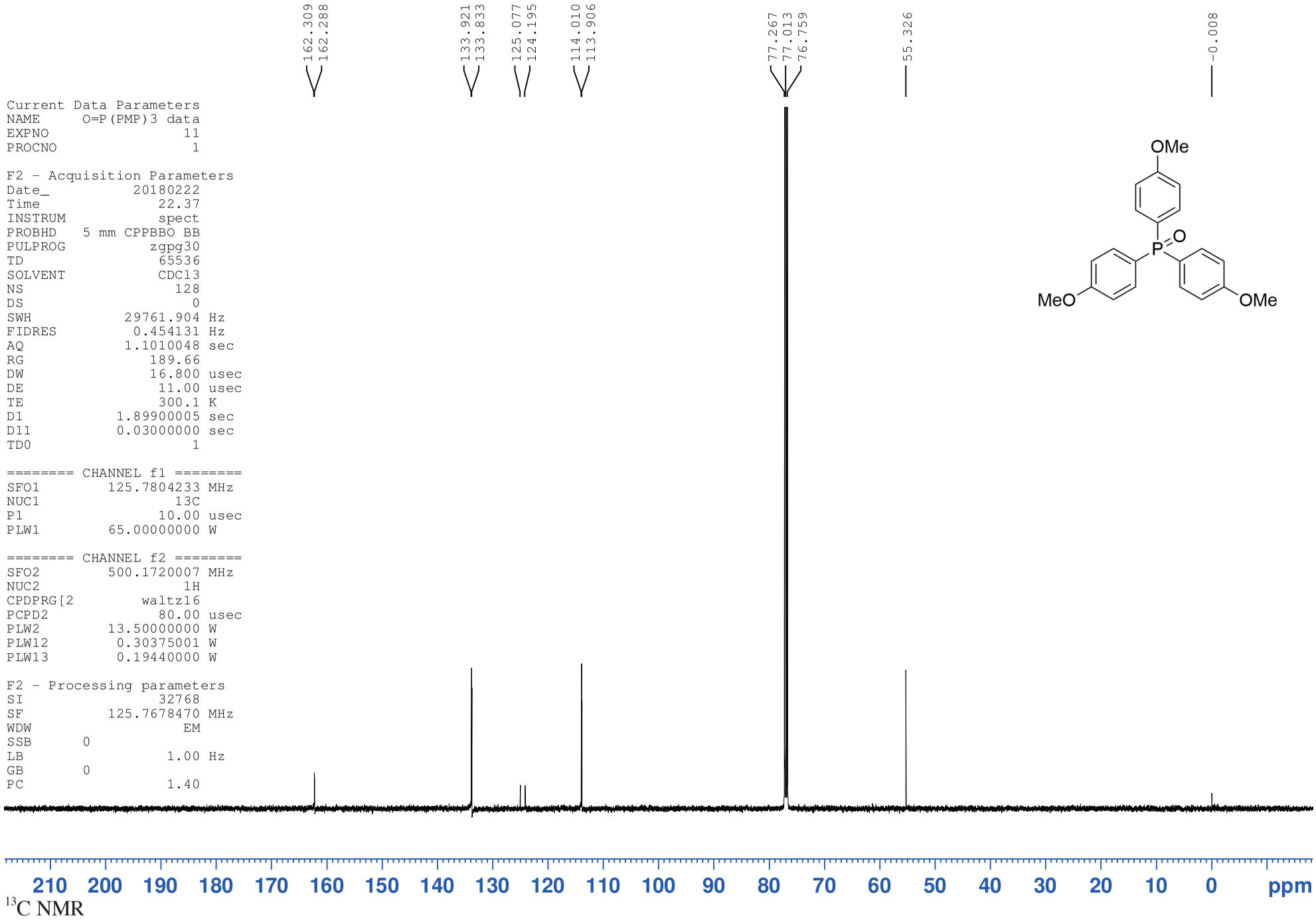
^{1}H NMR


175.909
139.260
136.031
128.991
128.499
128.365
128.097
128.067
126.313
77.269
77.015
76.761
66.135
41.514
39.746
16.816
-0.004
Current Data Parameters
NAME TKS312TM
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170601
Time 22.14
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678474 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR


7.585
7.568
7.562
7.545
7.261
6.959
6.955
6.942
6.937
3.840
1.648
-0.000
Current Data Parameters
NAME O=P(PMP)3 data
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180222
Time 22.34
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
O
P
MeO
OMe
9
8
7
6
5
4
3
2
1
0 ppm
6.011
6.024
9.000

^{1}H NMR


162.309
162.288
133.921
133.833
125.077
124.195
114.010
113.906
77.267
77.013
76.759
55.326
-0.008
Current Data Parameters
NAME O=P(PMP)3 data
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180222
Time 22.37
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.1 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804233 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OMe
O
P
MeO
OMe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

^{1}H NMR

^{13}C NMR