Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Modulating chitin synthesis in marine algae with iminosugars obtained by SmI_2 and $FeCl_3$-mediated diastereoselective carbonyl ene reaction

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[¶] both coauthors contributed equally to this work.

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1) General information

Moisture sensitive reactions were performed under nitrogen atmosphere using flame-dried glassware and standard Schlenk techniques. NMR-spectra were recorded on a Bruker Avance 300, Avance 400, Avance 500 or Avance 700. NMR-spectra data were reported as δ values in ppm relative to the NMR-solvent residue signals. Used solvents were specified for each compound. New compounds were fully characterized using 2D-NMR techniques for signal assignment. ^{1}H-NMR coupling constants (*J*) were reported in Hertz (Hz) and indicated with the following short tags for multiplicity: s (singlet); d (dublet); t (triplet); q (quartet); m (multiplet) and br (broad). Mass spectra were recorded on a Finnigan MAT MAT95 spectrometer (EI-conditions at 70 eV) or a Bruker Daltonics micro-TOF-Q spectrometer (ESI-conditions). IR spectra were recorded on a Bruker Alpha Platinum ATR with MKII Golden Gate Single Reflection Diamant ATR-system. HPLC was performed using a Shimadzu system containing LC-20AT pumps, a CBM-20A prominence communications bus module and a SPD-20A UV/Vis-detector. Separation was accomplished via achiral Orbit- or chiral Chiracel®-OD-column. Column chromatography was performed using Fluka silica (Type 60, diameter 40 – 70 µm). Solvents were distilled prior to use. Unless stated otherwise, all chemicals were used as received from the suppliers. Reaction progress was tracked using Macherey-Nagel TLC plates type 60 F 254 with fluorescence indicator. For non-UV-active compounds staining solutions were used and the plates were slightly heated.

Staining solutions for TLC:

Anisaldehyde-solution: 0.5 mL 4-methoxybenzaldehyde, 50 mL glacial acetic acid, 1.0 mL concentrated sulfuric acid, 300 mL ethanol.

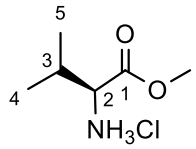
Ninhydrin-solution: 0.3 g ninhydrin in 95 mL acetone.

2) Synthetic procedures

GP1: Synthesis of amino acid methyl ester hydrochlorides from their respective D- or L-amino acid

The hydrochlorides were prepared according to the literature.[1] Thionyl chloride (8.7 mL, 14.3 g, 120 mmol) was added dropwise to a solution of the amino acid (40.0 mmol) in methanol (40 mL) at 0 °C. After stirring for 48 h at room temperature the solvent was evaporated. The products were isolated as colorless solids and used in further reactions without additional purifications.

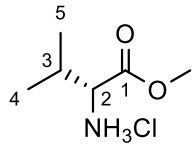
Methyl L-valinate hydrochloride (25a)



25a

According to GP1 **25a** was prepared from L-valine **24a** (1.76 g, 15.0 mmol) and thionyl chloride (3.3 mL, 5.35 g, 45.0 mmol) as a colorless solid (2.52 g, 15.0 mmol, quant.). $[\alpha]_D^{20}$ + 7 (*c* 1.0 in H_2O). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 0.94 (d, *J* = 6.9 Hz, 3H, 4-H/5-H), 0.99 (d, *J* = 6.9 Hz, 3H, 5-H/4-H), 2.13 – 2.29 (m, 1H, 3-H), 3.70 – 3.78 (m, 3H, OMe), 3.78 – 3.85 (m, 1H, 2-H), 8.42 – 9.00 (m, 3H, NH_3). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 17.5 (C-4/C-5), 18.4 (C-5/C-4), 29.3 (C-3), 52.5 (OMe), 57.2 (C-2), 169.2 (C-1). The spectral data are in accordance with previous reported literature.[2]

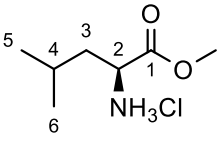
Methyl D-valinate hydrochloride (25b)



25b

According to GP1 **25b** was prepared from D-valine **24b** (4.67 g, 40.0 mmol) and thionyl chloride (8.7 mL, 14.3 g, 120 mmol) as a colorless solid (6.60 g, 39.4 mmol, 98 %). $[\alpha]_D^{20}$ - 17 (*c* 1.0 in H_2O). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 0.94 (d, *J* = 7.0 Hz, 3H, 4-H/5-H), 0.99 (d, *J* = 7.0 Hz, 3H, 5-H/4-H), 2.13 – 2.28 (m, 1H, 3-H), 3.74 (s, 3H, OMe), 3.78 – 3.87 (m, 1H, 2-H), 8.30 – 9.10 (m, 3H, NH_3). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 17.5 (C-4/C-5), 18.5 (C-5/C-4), 29.3 (C-3), 52.5 (OMe), 57.3 (C-2), 169.1 (C-1). The spectral data are in accordance with previous reported literature.[2]

Methyl L-leucine hydrochloride (25c)

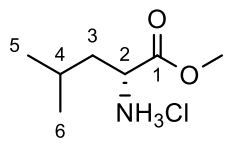


25c

According to GP1 **25c** was prepared from L-leucine **24c** (2.62 g, 20.0 mmol) and thionyl chloride (4.4 mL, 7.14 g, 60.0 mmol) as a colorless solid (3.59 g, 19.8 mmol, 99 %). $[\alpha]_D^{20}$ + 18 (*c* 1.0 in H_2O). ^{1}H-NMR (400 MHz, D_2O) δ 0.90 – 1.10 (m, 6H, 5-H, 6-H), 1.70 – 1.86 (m, 2H, 3-H_a, 4-H), 1.86 – 1.99 (m, 1H, 3-H_b), 3.86 – 3.93 (m, 3H, OMe), 4.15 – 4.25 (m, 1H, 2-H). ^{13}C-NMR (101 MHz, D_2O) δ 21.0 (C-5/C-6), 21.5 (C-6/C-5), 23.9 (C-4), 38.8 (C-3), 51.5 (C-

2), 53.5 (OMe), 171.3 (C-1). The spectral data are in accordance with previous reported literature.[3]

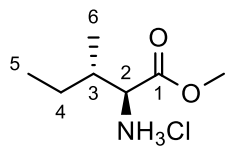
Methyl D-leucine hydrochloride (25d)



25d

According to GP1 **25d** was prepared from D-leucine **24d** (5.25 g, 40.0 mmol) and thionyl chloride (8.7 mL, 14.3 g, 120.0 mmol) as a colorless solid (6.80 g, 37.4 mmol, 94 %). $[\alpha]_D^{20}$ - 7 (*c* 1.0 in H_2O). ^{1}H-NMR (500 MHz, D_2O) δ 0.91 – 1.02 (m, 6H, 5-H, 6-H), 1.70 – 1.82 (m, 2H, 3-H_a, 4-H), 1.83 – 1.93 (m, 1H, 3-H_b), 3.86 (s, 3H, OMe), 4.12 – 4.22 (m, 1H, 2-H). ^{13}C-NMR (126 MHz, D_2O) δ 21.0 (C-5/C-6), 21.5 (C-6/C-5), 23.9 (C-4), 38.8 (C-3), 51.4 (C-2), 53.5 (OMe), 171.3 (C-1). The spectral data are in accordance with previous reported literature.[3]

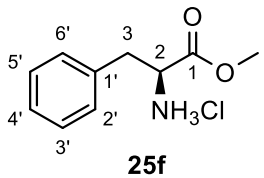
Methyl L-isoleucinate hydrochloride (25e)



25e

According to GP1 **25e** was prepared from L-isoleucine **24e** (5.25 g, 40.0 mmol) and thionyl chloride (8.7 mL, 14.3 g, 120 mmol) as a colorless solid (7.19 g, 39.6 mmol, 99 %). $[\alpha]_D^{20}$ + 15 (*c* 0.6 in $CHCl_3$). ^{1}H-NMR (400 MHz, D_2O) δ 0.98 (t, *J* = 7.4 Hz, 3H, 5-H), 1.05 (d, *J* = 7.1 Hz, 3H, 6-H), 1.30 – 1.46 (m, 1H, 4-H_a), 1.46 – 1.60 (m, 1H, 4-H_b), 2.02 – 2.20 (m, 1H, 3-H), 3.89 (s, 3H, OMe), 4.10 – 4.24 (m, 1H, 2-H). ^{13}C-NMR (101 MHz, D_2O) δ 10.8 (C-5), 14.1 (C-6), 24.8 (C-4), 36.0 (C-3), 53.3 (OMe), 57.3 (C-2), 170.3 (C-1). The spectral data are in accordance with previous reported literature.[4]

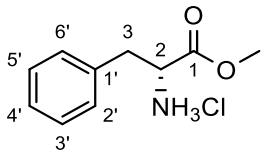
Methyl L-phenylalaninate hydrochloride (25f)



25f

According to GP1 **25f** was prepared from L-phenylalanine **24f** (6.60 g, 40.0 mmol) and thionyl chloride (8.7 mL, 14.3 g, 120 mmol) as a colorless solid (8.31 g, 38.5 mmol, 96 %). $[\alpha]_D^{20}$ + 13 (*c* 4.6 in $CHCl_3$). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 3.11 (dd, *J* = 13.9 Hz, 7.6 Hz, 1H, 3-H_a), 3.25 (dd, *J* = 13.9 Hz, 5.4 Hz, 1H, 3-H_b), 3.63 (s, 3H, OMe), 4.12 – 4.28 (m, 1H, 2-H), 7.18 – 7.40 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 8.87 (s, 3H, NH_3). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 35.8 (C-3), 52.4 (OMe), 53.3 (C-2), 127.2 (C-4'), 128.5 (C-2', C-6'), 129.4 (C-3', C-5'), 134.8 (C-1'), 169.3 (C-1). The spectral data are in accordance with previous reported literature.[5]

Methyl D-phenylalaninate hydrochloride (25g)



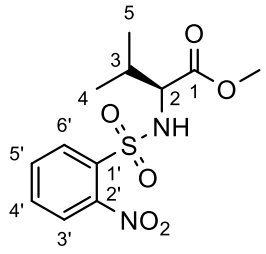
25g

According to GP1 **25g** was prepared from D-phenylalanine **24g** (6.61 g, 40.0 mmol) and thionyl chloride (8.7 mL, 14.3 g, 120 mmol) as a colorless solid (8.47 g, 39.3 mmol, 98 %). $[\alpha]_D^{20}$+ 64 (*c* 1.0 in H_2O). ^{1}H-NMR (500 MHz, DMSO-d_6) δ 3.09 (dd, *J* = 14.0 Hz, 7.4 Hz, 1H, 3-H_a), 3.18 (dd, *J* = 14.0 Hz, 5.4 Hz, 1H, 3-H_b), 3.66 (s, 3H, OMe), 4.21 – 4.30 (m, 1H, 2-H), 7.21 – 7.37 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 8.64 (s, 3H, NH_3). ^{13}C-NMR (126 MHz, DMSO-d_6) δ 35.9 (C-3), 52.6 (OMe), 53.2 (C-2), 127.3 (C-4'), 128.6 (C-2', C-6'), 129.4 (C-3', C-5'), 134.6 (C-1'), 169.4 (C-1). The spectral data are in accordance with previous reported literature.[5]

GP2: *N*-Protection of amino group using *o*-nitrobenzenesulfonyl chloride

The *N*-protected amino acid methyl esters were prepared according to the literature.[6] A round bottom flask was charged with a solution of the amino acid methyl ester hydrochloride (10.0 mmol) in dichloromethane (20 mL) under nitrogen atmosphere. Upon cooling to 0 °C solutions of triethylamine (3.5 mL, 25.0 mmol, 2.5 M in dichloromethane) and *o*-nitrobenzenesulfonyl chloride (2.88 g, 13.0 mmol, 2.6 M in dichloromethane) were added dropwise. After stirring for 20 h at room temperature the organic phase was washed with water (3 × 20 mL) and dried over magnesium sulfate. The solvents were evaporated and the crude product was purified using column chromatography. The products were isolated as oils or solids.

Methyl-((2-nitrophenyl)sulfonyl)-L-valinate (26a)

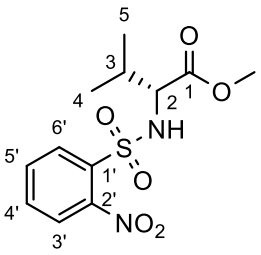


26a

According to GP2 **26a** was prepared from methyl L-valinate hydrochloride **25a** (1.68 g, 10.0 mmol), *o*-nitrobenzenesulfonyl chloride (2.88 g, 13.0 mmol) and triethylamine (3.5 mL, 2.53 g, 25.0 mmol) as a yellow oil (2.88 g, 9.11 mmol, 91 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (5 : 1 → 3 : 1). $[\alpha]_D^{20}$ - 194 (*c* 1.0 in $CHCl_3$). R_f = 0.34 (hexanes/EtOAc, 7 : 3, UV). FT-IR (ATR): $\tilde{\nu}$ = 3314 (w, br), 3098 (w), 2967 (w), 1739 (vs), 1594 (w), 1541 (vs), 1442 (m), 1427 (m), 1392 (w), 1357 (vs), 1301 (m), 1265 (m), 1209 (m), 1173 (vs), 1141 (s), 1126 (m), 1062 (w), 1017 (w), 996 (w), 965 (w), 917 (w), 879 (w), 854 (m), 784 (m), 760 (w), 742 (m), 701 (w), 655 (m), 598 (s), 567 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.94 (d, *J* = 6.8 Hz, 3H, 4-H/5-H), 1.01 (d, *J* = 6.8 Hz, 3H, 5-H/4-H), 2.16 (dqq, *J* = 6.8 Hz, 6.8 Hz, 5.2 Hz, 1H, 3-H), 3.44 (s, 3H, OMe), 4.02 (dd, *J* = 9.7 Hz, 5.2 Hz, 1H, 2-H), 6.03 (d, *J* = 9.7 Hz, 1H,

NH), 7.69 – 7.76 (m, 2H, 4'-H, 5'-H), 7.90 –7.96 (m, 1H, 3'-H), 8.03 – 8.09 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.5 (C-4/C-5), 19.0 (C-5/C-4), 31.6 (C-3), 52.2 (OMe), 62.2 (C-2), 125.6 (C-3'), 130.4 (C-6'), 132.8 (C-5'), 133.6 (C-4'), 134.2 (C-1'), 147.7 (C-2'), 171.1 (C-1). HRMS (ESI): calc (found) for $C_{12}H_{16}N_2O_6S$ ($[M+Na]^+$): 339.0621 (339.0630). The spectral data are in accordance with previous reported literature.[6]

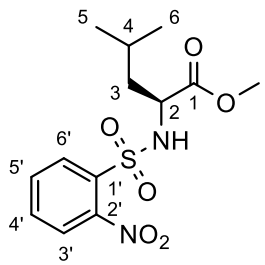
Methyl-((2-nitrophenyl)sulfonyl)-D-valinate (26b)



26b

According to GP2 **26b** was prepared from methyl D-valinate hydrochloride **25b** (1.68 g, 10.0 mmol), *o*-nitrobenzylsulfonyl chloride (2.88 g, 13.0 mmol) and triethylamine (3.5 mL, 2.53 g, 25.0 mmol) as a yellow oil (2.85 g, 9.00 mmol, 90 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (5 : 1 → 3 : 1). $[\alpha]_D^{20}$ + 205 (*c* 1.0 in $CHCl_3$). R_f = 0.21 (hexanes/EtOAc, 3 : 1, UV). FT-IR (ATR): $\tilde{\nu}$ = 3321 (w, br), 3099 (w), 2966 (w), 1737 (vs), 1594 (w), 1539 (vs), 1441 (s), 1425 (s), 1392 (w), 1354 (vs), 1300 (s), 1263 (s), 1208 (s), 1169 (vs), 1139 (s), 1125 (s), 1060 (m), 1016 (m), 996 (m), 964 (w), 914 (m), 878 (m), 854 (s), 783 (s), 759 (m), 731 (vs), 701 (m), 654 (s), 592 (vs), 563 (vs), 468 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.92 (d, *J* = 7.0 Hz, 3H, 4-H/5-H), 1.01 (d, *J* = 7.0 Hz, 3H, 5-H/4-H), 2.10 – 2.23 (m, 1H, 3-H), 3.44 (s, 3H, OMe), 4.01 (dd, *J* = 9.8 Hz, 5.3 Hz, 1H, 2-H), 6.04 (d, *J* = 9.8 Hz, 1H, NH), 7.69 – 7.77 (m, 2H, 4'-H, 5'-H), 7.90 – 7.97 (m, 1H, 3'-H), 8.03 – 8.09 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.5 (C-4/C-5), 19.0 (C-5/C-4), 31.6 (C-3), 52.2 (O-Me), 62.2 (C-2), 125.6 (C-3'), 130.4 (C-6'), 132.8 (C-5'), 133.6 (C-4'), 134.1 (C-1'), 147.7 (C-2'), 171.1 (C-1). HRMS (ESI): calc (found) for $C_{12}H_{16}N_2O_6S$ ($[M+Na]^+$): 339.0621 (339.0622). The spectral data are in accordance with previous reported literature.[7]

Methyl-((2-nitrophenyl)sulfonyl)-L-leucinate (26c)

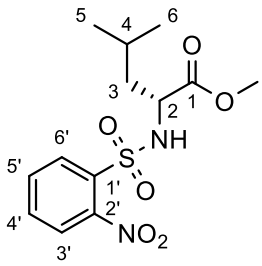


26c

According to GP2 **26c** was prepared from methyl L-leucine hydrochloride **25c** (2.73 g, 15.0 mmol), *o*-nitrobenzylsulfonyl chloride (4.32 g, 19.5 mmol) and triethylamine (5.2 mL, 3.80 g, 37.5 mmol) as a yellow oil (4.92 g, 14.9 mmol, 99 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). $[\alpha]_D^{20}$ - 183 (*c* 1.0 in $CHCl_3$). R_f = 0.64 (hexanes/EtOAc, 1 : 1, UV). FT-IR (ATR): $\tilde{\nu}$ = 3318 (w, br), 3100 (w), 2959 (m), 2873 (w), 1743 (s), 1594 (w), 1542 (vs), 1442 (m), 1423 (m), 1357 (vs), 1305 (m), 1275 (m), 1234 (m), 1205 (m), 1173 (vs), 1148 (s), 1126 (m), 1090 (w), 1060 (w), 1021 (w), 980 (w), 900 (w), 854 (w), 821 (w), 785 (w), 742 (m), 701

(w), 655 (m), 597 (m), 575 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.95 (d, *J* = 6.6 Hz, 6H, 5-H, 6-H), 1.57 – 1.64 (m, 2H, 3-H), 1.79 – 1.91 (m, 1H, 4-H), 3.42 (s, 3H, OMe), 4.18 – 4.27 (m, 1H, 2-H), 5.95 (d, *J* = 9.9 Hz, 1H, N*H*), 7.70 – 7.76 (m, 2H, 4'-H, 5'-H), 7.92 – 7.96 (m, 1H, 3'-H), 8.05 – 8.09 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 21.3 (C-5/C-6), 22.7 (C-6/C-5), 24.3 (C-4), 42.0 (C-3), 52.2 (OMe), 55.4 (C-2), 125.6 (C-3'), 130.4 (C-6'), 132.9 (C-5'), 133.6 (C-4'), 134.1 (C-1'), 147.6 (C-2'), 172.0 (C-1). HRMS (ESI): calc (found) for $C_{13}H_{18}N_2O_6S$ ($[M+Na]^+$): 353.0778 (353.0788). The spectral data are in accordance with previous reported literature.[8]

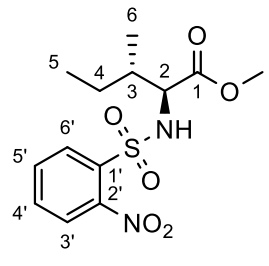
Methyl-((2-nitrophenyl)sulfonyl)-D-leucinate (26d)



26d

According to GP2 **26d** was prepared from methyl D-leucine hydrochloride **25d** (2.73 g, 15.0 mmol) , *o*-nitrobenzylsulfonyl chloride (4.32 g, 19.5 mmol) and triethylamine (5.2 mL, 3.80 g, 37.5 mmol) as a yellow oil (2.90 g, 8.78 mmol, 59 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1). $[\alpha]_D^{20}$ + 188 (*c* 1.0 in $CHCl_3$). R_f = 0.63 (hexanes/EtOAc, 1 : 1, UV). FT-IR (ATR): $\tilde{\nu}$ = 3321 (w, br), 3100 (w), 2958 (m), 2872 (w), 1740 (s), 1594 (w), 1539 (vs), 1441 (m), 1422 (s), 1351 (vs), 1303 (m), 1273 (m), 1234 (m), 1204 (m), 1169 (vs), 1125 (s), 1089 (m), 1060 (m), 1020 (m), 981 (w), 901 (m), 854 (m), 819 (w), 784 (s), 740 (vs), 732 (s), 701 (m), 655 (s), 594 (vs), 574 (vs), 486 (w), 465 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.90 – 0.97 (m, 6H, 5-H, 6-H), 1.57 – 1.62 (m, 2H, 3-H), 1.78 – 1.88 (m, 1H, 4-H), 3.40 (s, 3H, OMe), 4.15 – 4.26 (m, 1H, 2-H), 5.96 (d, *J* = 9.6 Hz, 1H, N*H*), 7.69 – 7.77 (m, 2H, 4'-H, 5'-H), 7.89 – 7.95 (m, 1H, 3'-H), 8.03 – 8.09 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 21.3 (C-5/C-6), 22.7 (C-6/C-5), 24.4 (C-4), 42.0 (C-3), 52.2 (OMe), 55.4 (C-2), 125.6 (C-3'), 130.5 (C-6'), 132.9 (C-5'), 133.7 (C-4'), 134.1 (C-1'), 147.7 (C-2'), 172.0 (C-1). HRMS (ESI): calc (found) for $C_{13}H_{18}N_2O_6S$ ($[M+NH_4]^+$): 348.1224 (348.1225). The spectral data are in accordance with previous reported literature.[8]

Methyl-((2-nitrophenyl)sulfonyl)-L-isoleucinate (26e)

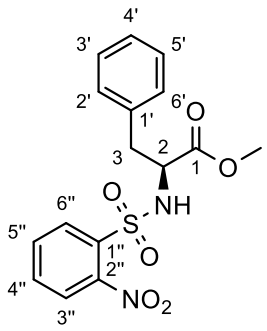


26e

According to GP2 **26e** was prepared from methyl L-isoleucinate hydrochloride **25e** (1.82 g, 10.0 mmol), *o*-nitrobenzylsulfonyl chloride (2.88 g, 13.0 mmol) and triethylamine (3.5 mL, 2.53 g, 25.0 mmol) as a red oil (2.64 g, 8.00 mmol, 80 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (5 : 1 → 4 : 1 → 3 : 1). $[\alpha]_D^{20}$ - 84 (*c* 2.4 in $CHCl_3$). R_f = 0.26 (hexanes/EtOAc,

3 : 1, UV). FT-IR (ATR): $\tilde{\nu}$ = 3325 (w, br), 3098 (w), 2965 (m), 2932 (w), 2879 (w), 1738 (vs), 1594 (w), 1540 (vs), 1442 (s), 1426 (s), 1355 (vs), 1299 (m), 1259 (m), 1207 (m), 1171 (vs), 1140 (s), 1125 (s), 1061 (m), 1014 (w), 988 (w), 880 (w), 854 (m), 784 (m), 742 (s), 702 (w), 655 (m), 593 (s), 569 (m), 467 (w) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.91 (t, *J* = 7.4 Hz, 3H, 5-H), 0.96 (d, *J* = 6.9 Hz, 3H, 6-H), 1.16 – 1.32 (m, 1H, 4-H_a), 1.40 – 1.54 (m, 1H, 4-H_b), 1.85 – 1.98 (m, 1H, 3-H), 3.42 (s, 3H, OMe), 4.06 (dd, *J* = 9.7 Hz, 5.4 Hz, 1H, 2-H), 6.04 (d, *J* = 9.7 Hz, 1H, NH), 7.68 – 7.76 (m, 2H, 4'-H, 5'-H), 7.88 – 7.96 (m, 1H, 3'-H), 8.02 – 8.10 (m, 1H, 6'-H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 11.2 (C-5), 15.5 (C-6), 24.7 (C-4), 38.2 (C-3), 52.1 (OMe), 61.4 (C-2), 125.6 (C-3'), 130.5 (C-6'), 132.8 (C-5'), 133.6 (C-4'), 134.2 (C-1'), 147.7 (C-2'), 171.1 (C-1). HRMS (ESI): calc (found) for $C_{13}H_{18}N_2O_6S$ ($[M+Na]^+$): 353.0778 (353.0778). The spectral data are in accordance with previous reported literature.[9]

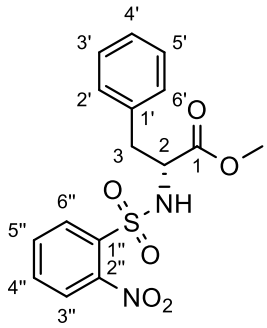
Methyl-((2-nitrophenyl)sulfonyl)-L-phenylalaninate (26f)



26f

According to GP2 **26f** was prepared from methyl L-phenylalaninate hydrochloride **25f** (2.16 g, 10.0 mmol), *o*-nitrobenzylsulfonyl chloride (2.88 g, 13.0 mmol) and triethylamine (3.5 mL, 2.53 g, 25.0 mmol) as a yellow solid (2.22 g, 6.10 mmol, 61 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (5 : 1 → 4 : 1 → 3 : 1 → 2 : 1). mp 88 °C. $[\alpha]_D^{20}$ - 38 (*c* 2.8 in $CHCl_3$). R_f = 0.18 (hexanes/EtOAc, 3 : 1, UV). FT-IR (ATR): $\tilde{\nu}$ = 3323 (w, br), 3097 (w), 3028 (w), 2954 (w), 1743 (s), 1594 (w), 1540 (vs), 1497 (w), 1440 (m), 1423 (m), 1359 (vs), 1302 (m), 1219 (m), 1169 (vs), 1105 (m), 1060 (w), 1032 (w), 962 (w), 903 (w), 854 (m), 784 (w), 740 (m), 702 (m), 655 (w), 599 (m), 575 (m), 538 (w), 494 (w) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 3.08 (dd, *J* = 13.9 Hz, 6.9 Hz, 1H, 3-H_a), 3.16 (dd, *J* = 13.9 Hz, 5.7 Hz, 1H, 3-H_b), 3.52 (s, 3H, OMe), 4.40 – 4.55 (m, 1H, 2-H), 5.98 (d, *J* = 8.7 Hz, 1H, NH), 7.07 – 7.13 (m, 2H, 2'-H, 6'-H), 7.15 – 7.25 (m, 3H, 3'-H, 4'-H, 5'-H), 7.61 – 7.73 (m, 2H, 4"-H, 5"-H), 7.80 – 7.90 (m, 1H, 3"-H), 7.92 – 8.02 (m, 1H, 6"-H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 39.2 (C-3), 52.4 (OMe), 57.8 (C-2), 125.6 (C-3"), 127.4 (C-4'), 128.7 (C-3', C-5'), 129.3 (C-2', C-6'), 130.3 (C-6"), 132.9 (C-5"), 133.4 (C-4"), 134.3 (C-1"), 134.8 (C-1'), 147.5 (C-2"), 170.8 (C-1). HRMS (ESI): calc (found) for $C_{16}H_{16}N_2O_6S$ ($[M+Na]^+$): 387.0621 (387.0622). The spectral data are in accordance with previous reported literature.[6]

Methyl-((2-nitrophenyl)sulfonyl)-D-phenylalaninate (26g)

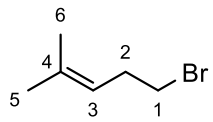


26g

According to GP2 **26g** was prepared from methyl D-phenylalaninate hydrochloride **25g** (3.24 g, 15.0 mmol), *o*-nitrobenzylsulfonyl chloride (4.32 g, 19.5 mmol) and triethylamine (5.2 mL, 3.80 g, 37.5 mmol) as a yellow solid (4.51 g, 12.4 mmol, 83 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1 → 1 : 1). mp 80 °C. $[\alpha]_D^{20}$ + 67 (*c* 1.0 in $CHCl_3$). FT-IR (ATR): $\tilde{\nu}$ = 3328 (w), 3103 (w), 3031 (w), 2956 (w), 2939 (w), 1738 (vs), 1594 (w), 1540 (vs), 1497 (m), 1450 (m), 1419 (s), 1346 (vs), 1301 (vs), 1253 (s), 1217 (s), 1200 (s), 1185 (s), 1167 (vs), 1124 (s), 1100 (vs), 1056 (m), 1033 (m), 1013 (m), 977 (m), 946 (m), 935 (m), 854 (s), 814 (m), 777 (s), 763 (m), 748 (s), 736 (vs), 704 (vs), 670 (m), 651 (s), 609 (m), 588 (vs), 555 (vs), 530 (s), 487 (m), 471 (m) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 3.08 (dd, *J* = 13.9 Hz, 6.9 Hz, 1H, 3-H_a), 3.15 (dd, *J* = 13.9 Hz, 5.7 Hz, 1H, 3-H_b), 3.52 (s, 3H, OMe), 4.46 (ddd, *J* = 8.7 Hz, 6.9 Hz, 5.7 Hz, 1H, 2-H), 5.98 (d, *J* = 8.7 Hz, 1H, NH), 7.08 – 7.13 (m, 2H, 2'-H, 6'-H), 7.17 – 7.24 (m, 3H, 3'-H, 4'-H, 5'-H), 7.64 – 7.70 (m, 2H, 4"-H, 5"-H), 7.83 – 7.88 (m, 1H, 3"-H), 7.94 – 8.00 (m, 1H, 6"-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 39.2 (C-3), 52.5 (OMe), 57.8 (C-2), 125.6 (C-3"), 127.5 (C-4'), 128.7 (C-3', C-5'), 129.3 (C-2', C-6'), 130.3 (C-6"), 132.9 (C-5"), 133.4 (C-4"), 134.2 (C-1"), 134.8 (C-1'), 147.5 (C-2"), 170.8 (C-1).). HRMS (ESI): calc (found) for $C_{16}H_{16}N_2O_6S$ ($[M+Na]^+$): 387.0621 (387.0623). The spectral data are in accordance with previous reported literature.[6]

5-Bromo-2-methylpent-2-ene (S1)

The reaction was performed according to the literature.[10] A round bottom flask was charged with a solution of methyl magnesium bromide (26.0 mL, 78.0 mmol, 3 M in Et_2O) under nitrogen atmosphere. Upon cooling to 0 °C a solution of methyl cyclopropyl ketone (5.9 mL, 5.05 g, 60.0 mmol) in diethyl ether (8 mL) was added dropwise and the resulting mixture was stirred for 30 min at 0 °C. Afterwards a mixture of concentrated sulfuric acid and water (36 mL, 1 : 2) was added slowly under ice-cooling, keeping the temperature under 7 °C. Stirring was continued for another 30 min at 0 °C. The organic phase was separated and the aqueous phase was extracted with diethyl ether (2 × 25 mL). The organic phases were washed with sodium hydrogen sulfite solution (5 %), saturated sodium bicarbonate solution and brine (each 1 × 45 mL). They were dried over magnesium sulfate and the solvents were evaporated. The product was obtained as a colorless oil (9.32 g, 57.2 mmol, 95 %) without further purification.



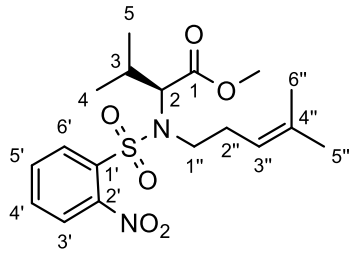
S1

^{1}H-NMR (300 MHz, $CDCl_3$) δ 1.63 (d, *J* = 1.4 Hz, 3H, 5-H/6-H), 1.69 – 1.76 (q, *J* = 1.4 Hz, 3H, 6-H/5-H), 2.56 (dt, *J* = 7.3 Hz, 7.3 Hz, 2H, 2-H), 3.34 (t, *J* = 7.3 Hz, 2H, 1-H), 5.13 (tdq, *J* = 7.3 Hz, 2.9 Hz, 1.4 Hz, 1H, 3-H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 17.9 (C-2), 25.7 (C-1), 31.8 (C-5), 32.9 (C-6), 121.0 (C-4), 135.0 (C-3). The spectral data are in accordance with previous reported literature.[10]

GP3: *N*-Alkylation of amino acid methyl esters

The *N*-alkylated amino acid methyl esters were prepared according to the literature.[6] A round bottom flask was charged with a solution of the amino acid methyl ester (8.00 mmol) in dimethylformamide (60 mL) under nitrogen atmosphere. Cesium carbonate (3.91 g, 12.0 mmol) and 5-bromo-2-methylpent-2-ene (1.96 g, 12.0 mmol) were added and the mixture was stirred for 20 h at 60 °C. After cooling to room temperature diethyl ether (120 mL) was added. The organic phase was washed with water (2 × 60 mL) and brine (2 × 40 mL) and dried over sodium sulfate. The solvents were evaporated and the crude product was purified by column chromatography. The products were isolated as oils.

Methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)- L-valinate (27a)

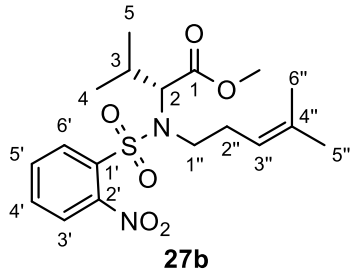


27a

According to GP3 **27a** was prepared from methyl-((2-nitrophenyl)sulfonyl)-L-valinate **26a** (2.83 g, 8.94 mmol), cesium carbonate (4.37 g, 13.4 mmol) and 5-bromo-2-methylpent-2-ene **S1** (2.19 g, 13.4 mmol) as a yellow oil (1.56 g, 3.92 mmol, 44 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). Reisolation of starting material **26a** was possible (0.55 g, 1.74 mmol, 19 %). $[\alpha]_D^{20}$ - 70 (*c* 1.0 in $CHCl_3$). R_f = 0.61 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3096 (w), 2968 (m), 2933 (w), 2916 (w), 2878 (w), 1740 (s), 1590 (w), 1544 (vs), 1469 (m), 1372 (s), 1352 (s), 1291 (m), 1249 (w), 1204 (m), 1165 (s), 1146 (s), 1126 (s), 1067 (m), 1009 (m), 973 (m), 891 (w), 852 (m), 817 (w), 776 (m), 749 (m), 653 (w), 585 (s), 565 (m), 525 (w) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.97 (d, *J* = 6.7 Hz, 3H, 4-H/5-H), 1.02 (d, *J* = 6.7 Hz, 3H, 5-H/4-H), 1.63 (s, 3H, 5''-H/6''-H), 1.69 (s, 3H, 6''-H/5''-H), 2.10 – 2.36 (m, 2H, 3-H, 2''-H_a), 2.37 – 2.56 (m, 1H, 2''-H_b), 3.24 – 3.47 (m, 2H, 1''-H), 3.54 (s, 3H, OMe), 4.15 (d, *J* = 10.3 Hz, 1H, 2-H), 5.01 – 5.07 (m, 1H, 3''-H), 7.52 – 7.61 (m, 1H, 3'-H), 7.61 – 7.74 (m, 2H, 4'-H, 5'-H), 7.90 – 8.10 (m, 1H, 6'-H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 17.8 (C-5''/C-6''), 19.6 (C-4, C-5), 25.7 (C-6''/ C-5''), 28.8 (C-3), 29.7 (C-2''), 45.8 (C-1''), 51.7 (OMe), 65.9 (C-2), 119.8 (C-3''),

123.8 (C-3'), 130.8 (C-6'), 131.2 (C-5'), 133.2 (C-1'), 133.5 (C-4'), 134.8 (C-4''), 148.4 (C-2'), 170.7 (C-1). HRMS (ESI): calc (found) for $C_{18}H_{26}N_2O_6S$ ($[M+Na]^+$): 421.1404 (421.1406).

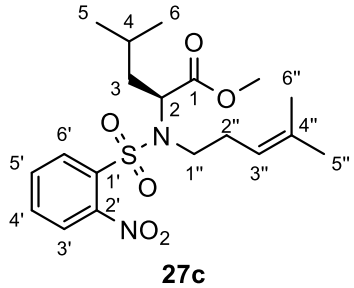
Methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-D-valinate (27b)



27b

According to GP3 **27b** was prepared from methyl-((2-nitrophenyl)sulfonyl)-D-valinate **26b** (2.76 g, 8.73 mmol), cesium carbonate (4.27 g, 13.1 mmol) and 5-bromo-2-methylpent-2-ene **S1** (2.14 g, 13.1 mmol) as a yellow oil (2.01 g, 5.05 mmol, 58 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). Starting material **26b** was reisolated (0.49 g, 1.54 mmol, 18 %). $[\alpha]_D^{20}$ + 58 (*c* 1.0 in $CHCl_3$). R_f = 0.44 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3100 (w), 2969 (m), 2933 (w), 2878 (w), 1741 (s), 1590 (w), 1546 (vs), 1438 (m), 1373 (s), 1352 (s), 1291 (m), 1204 (m), 1165 (s), 1146 (s), 1126 (m), 1068 (m), 1009 (m), 973 (m), 891 (w), 776 (m), 749 (m), 652 (w), 585 (s), 565 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.97 (d, *J* = 6.5 Hz, 3H, 4-H/5-H), 1.02 (d, *J* = 6.5 Hz, 3H, 5-H/4-H), 1.63 (s, 3H, 5''-H/6''-H), 1.69 (s, 3H, 6''-H/5''-H), 2.15 – 2.24 (m, 1H, 3-H), 2.24 – 2.34 (m, 1H, 2''-H_a), 2.39 – 2.51 (m, 1H, 2''-H_b), 3.28 – 3.43 (m, 2H, 1''-H), 3.54 (s, 3H, OMe), 4.15 (d, *J* = 10.2 Hz, 1H, 2-H), 5.01 – 5.09 (m, 1H, 3''-H), 7.54 –7.60 (m, 1H, 3'-H), 7.63 – 7.73 (m, 2H, 4'-H, 5'-H), 7.98 – 8.04 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.8 (C-5''/C-6''), 19.6 (C-4, C-5), 25.7 (C-6''/C-5''), 28.8 (C-3), 29.7 (C-2''), 45.8 (C-1''), 51.8 (OMe), 65.9 (C-2), 119.8 (C-3''), 123.8 (C-3'), 130.8 (C-6'), 131.2 (C-5'), 133.2 (C-1'), 133.4 (C-4'), 134.9 (C-4''), 148.3 (C-2'), 170.7 (C-1). HRMS (ESI): calc (found) for $C_{18}H_{26}N_2O_6S$ ($[M+Na]^+$): 421.1404 (421.1399).

Methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-L-leucinate (27c)

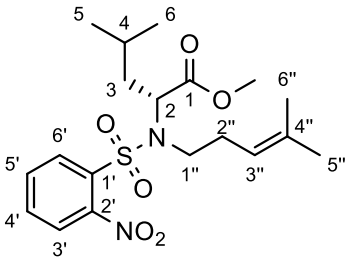


27c

According to GP3 **27c** was prepared from methyl-((2-nitrophenyl)sulfonyl)-L-leucinate **26c** (4.85 g, 14.7 mmol), cesium carbonate (7.18 g, 22.0 mmol) and 5-bromo-2-methyl-pent-2-ene **S1** (3.59 g, 22.0 mmol) as a yellow oil (4.43 g, 10.7 mmol, 73 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). Starting material **26c** was reisolated (0.19 g, 0.58 mmol, 4 %). $[\alpha]_D^{20}$ + 13 (*c* 1.0 in $CHCl_3$). R_f = 0.58 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 2957 (m), 2871 (w), 1743 (s), 1591 (w), 1545 (vs), 1438 (m), 1373 (s), 1354 (s), 1253 (m), 1205 (m), 1154 (s), 1127 (m), 1068 (w), 1005 (w), 964 (w), 947 (w), 906 (w), 852 (w), 775 (m), 755 (m), 733 (m), 652 (w),

587 (m), 567 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 0.98 (d, *J* = 6.3 Hz, 3H, 5-H/6-H), 1.00 (d, *J* = 6.3 Hz, 3H, 6-H/5-H), 1.61 – 1.72 (m, 1H, 3-H_a), 1.65 (s, 3H, 5"-H/6"-H), 1.70 (s, 3H, 6"-H/5"-H), 1.72 – 1.90 (m, 2H, 3-H_b, 4-H), 2.18 – 2.28 (m, 1H, 2"-H_a), 2.50 – 2.61 (m, 1H, 2"-H_b), 3.00 (ddd, *J* = 15.4 Hz, 12.0 Hz, 5.3 Hz, 1H, 1"-H_a), 3.39 (ddd, *J* = 15.4 Hz, 12.0 Hz, 4.8 Hz, 1H, 1"-H_b), 3.50 (s, 3H, OMe), 4.69 (dd, *J* = 10.4 Hz, 4.5 Hz, 1H, 2-H), 5.02 – 5.09 (m, 1H, 3"-H), 7.53 – 7.58 (m, 1H, 3'-H), 7.65 – 7.71 (m, 2H, 4'-H/5'-H), 8.00 – 8.07 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$): δ 17.9 (C-5"/C-6"), 21.2 (C-5/C-6), 23.0 (C-6/C-5), 24.4 (C-4), 25.7 (C-6"/C-5"), 30.3 (C-2"), 39.3 (C-3), 46.6 (C-1), 52.1 (OMe), 59.1 (C-2), 119.8 (C-3"), 123.9 (C-3'), 131.0 (C-6'), 131.2 (C-5'), 132.8 (C-1'), 133.3 (C-4'), 134.8 (C-4"), 148.2 (C-2'), 171.7 (C-1). HRMS (ESI): calc (found) for $C_{19}H_{28}N_2O_6S$ ($[M+Na]^+$): 435.1560 (435.1557).

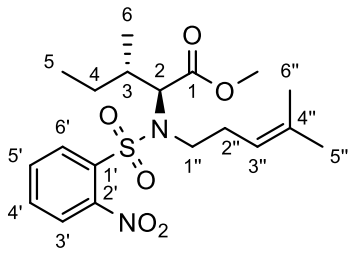
Methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-D-leucinate (27d)



27d

According to GP3 **27d** was prepared from methyl-((2-nitrophenyl)sulfonyl)-D-leucinate **26d** (2.64 g, 8.00 mmol), cesium carbonate (3.91 g, 12.0 mmol) and 5-bromo-2-methyl-pent-2-ene **S1** (1.96 g, 12.0 mmol) as a yellow oil (2.02 g, 4.88 mmol, 61 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). $[\alpha]_D^{20}$ - 21 (*c* 1.0 in $CHCl_3$). R_f = 0.56 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 2956 (m), 2872 (w), 1741 (s), 1590 (w), 1543 (vs), 1438 (m), 1372 (vs), 1352 (vs), 1293 (w), 1252 (m), 1204 (m), 1152 (vs), 1126 (s), 1067 (m), 1004 (m), 963 (m), 946 (m), 906 (w), 852 (m), 838 (w), 775 (m), 754 (s), 732 (s), 652 (m), 586 (vs), 566 (s) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.98 (d, *J* = 6.3 Hz, 3H, 5-H/6-H), 1.00 (d, *J* = 6.3 Hz, 3H, 6-H/5-H), 1.61 – 1.72 (m, 1H, 3-H_a), 1.65 (s, 3H, 5"-H/6"-H), 1.69 (s, 3H, 6"-H/ 5"-H), 1.74 – 1.89 (m, 2H, 3-H_b, 4-H), 2.16 – 2.30 (m, 1H, 2"-H_a), 2.50 – 2.61 (m, 1H, 2"-H_b), 3.01 (ddd, *J* = 15.4 Hz, 12.0 Hz, 5.3 Hz, 1H, 1"-H_a), 3.39 (ddd, *J* = 15.4 Hz, 12.0 Hz, 4.8 Hz, 1H, 1"-H_b), 3.50 (s, 3H, OMe), 4.69 (dd, *J* = 10.4 Hz, 4.5 Hz, 1H, 2-H), 5.02 – 5.09 (m, 1H, 3"-H), 7.52 – 7.58 (m, 1H, 3'-H), 7.64 – 7.72 (m, 2H, 4'-H, 5'-H), 7.99 – 8.07 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.9 (C-5"/C-6"), 21.2 (C-5/C-6), 23.0 (C-6/C-5), 24.4 (C-4), 25.7 (C-6"/C-5"), 30.3 (C-2"), 39.3 (C-3), 46.6 (C-1), 52.1 (OMe), 59.1 (C-2), 119.8 (C-3"), 123.9 (C-3'), 131.0 (C-6'), 131.2 (C-5'), 132.8 (C-1'), 133.4 (C-4'), 134.8 (C-4"), 148.2 (C-2'), 171.7 (C-1). HRMS (ESI): calc (found) for $C_{19}H_{28}N_2O_6S$ ($[M+Na]^+$): 435.1560 (435.1561).

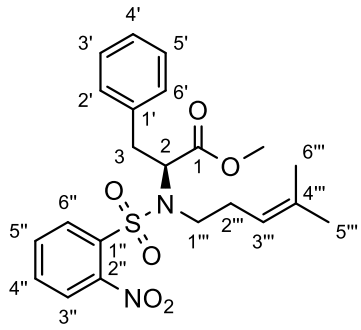
Methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-L-isoleucinate (27e)



27e

According to GP3 **27e** was prepared from methyl-((2-nitrophenyl)sulfonyl)-L-isoleucinate **26e** (0.28 g, 0.84 mmol), cesium carbonate (0.41 g, 1.26 mmol) and 5-bromo-2-methyl-pent-2-ene **S1** (0.21 g, 1.26 mmol) as a yellow oil (0.26 g, 0.63 mmol, 75 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). $[\alpha]_D^{20}$ - 70 (*c* 1.0 in $CHCl_3$). R_f = 0.32 (hexanes/EtOAc, 5 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 2967 (m), 2933 (w), 2878 (w), 1739 (s), 1590 (w), 1544 (vs), 1437 (m), 1372 (s), 1351 (vs), 1290 (w), 1257 (m), 1199 (m), 1168 (s), 1145 (vs), 1125 (s), 1067 (m), 996 (m), 960 (m), 912 (m), 889 (m), 851 (s), 775 (s), 731 (vs), 652 (m), 584 (vs), 567 (vs), 524 (w), 455 (w) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.86 – 0.95 (m, 6H, 5-H, 6-H), 1.04 – 1.19 (m, 1H, 4-H_a), 1.62 (s, 3H, 5"-H/6"-H), 1.68 (s, 3H, 6"-H/5"-H), 1.70 – 1.80 (m, 1H, 4-H_b), 1.85 – 1.98 (m, 1H, 3-H), 2.14 – 2.32 (m, 1H, 2"-H_a), 2.40 – 2.54 (m, 1H, 2"-H_b), 3.27 – 3.45 (m, 2H, 1"-H), 3.49 (s, 3H, OMe), 4.20 (d, *J* = 10.1 Hz, 1H, 2-H), 5.04 (t, *J* = 7.3 Hz, 1H, 3"-H), 7.50 – 7.58 (m, 1H, 3'-H), 7.61 – 7.72 (m, 2H, 4'-H, 5'-H), 7.92 – 8.04 (m, 1H, 6'-H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 11.0 (C-5), 15.6 (C-6), 17.8 (C-5"), 25.4 (C-4), 25.7 (C-6"), 30.0 (C-2"), 35.2 (C-3), 45.8 (C-1"), 51.6 (OMe), 64.8 (C-2), 119.8 (C-3"), 123.8 (C-3'), 130.8 (C-6'), 131.2 (C-5'), 133.0 (C-1'), 133.5 (C-4'), 134.7 (C-4"), 148.3 (C-2'), 170.7 (C-1). HRMS (ESI): calc (found) for $C_{19}H_{28}N_2O_6S$ ($[M+Na]^+$): 435.1560 (435.1565).

Methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-L-phenylalaninate (27f)

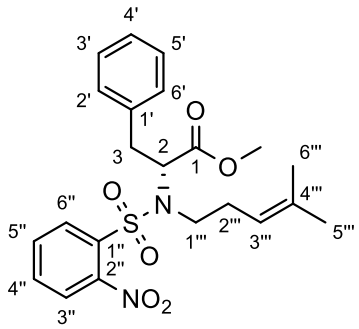


27f

According to GP3 **27f** was prepared from methyl-((2-nitrophenyl)sulfonyl)-L-phenylalaninate **26f** (2.17 g, 5.94 mmol), cesium carbonate (2.90 g, 8.91 mmol) and 5-bromo-2-methylpent-2-ene **S1** (1.45 g, 8.91 mmol) as a yellow oil (1.42 g, 3.18 mmol, 53 %). Starting material **26f** was reisolated (0.08 g, 0.23 mmol, 4 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1 → 2 : 1). $[\alpha]_D^{20}$ - 24 (*c* 1.0 in $CHCl_3$). R_f = 0.24 (hexanes/EtOAc, 4 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3030 (w), 2954 (w), 2925 (w), 1742 (s), 1589 (w), 1543 (vs), 1497 (w), 1454 (m), 1438 (m), 1373 (s), 1352 (s), 1205 (m), 1164 (vs), 1127 (s), 1090 (m), 1070 (m), 1011 (m), 950 (w), 852 (m), 776 (m), 749 (s), 700 (m), 652 (w), 588 (m), 577 (m), 543 (w) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.62 (s, 3H, 5'''-H/6'''-H), 1.69 (s, 3H,

6'''-H/5''-H), 2.18 – 2.41 (m, 2H, 2'''-H), 3.04 (dd, *J* = 14.2 Hz, 7.2 Hz, 1H, 3-H_a), 3.16 – 3.30 (ddd, *J* = 15.2 Hz, 11.3 Hz, 5.5 Hz, 1H, 1'''-H_a), 3.34 – 3.48 (m, 2H, 1'''-H_b, 3-H_b), 3.55 (s, 3H, OMe), 4.90 (t, *J* = 7.2 Hz, 1H, 2-H), 5.00 – 5.08 (m, 1H, 3'''-H), 7.18 – 7.33 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 7.54 – 7.62 (m, 2H, 3''-H, 5''-H), 7.62 – 7.70 (m, 1H, 4''-H), 7.84 – 7.90 (m, 1H, 6''-H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 17.9 (C-5'''/C-6'''), 25.7 (C-6'''/C-5'''), 29.4 (C-2'''), 36.8 (C-3), 46.2 (C-1'''), 52.3 (OMe), 61.3 (C-2), 119.7 (C-3'''), 124.0 (C-5''), 127.0 (C-4'), 128.6 (C-3', C-5'), 129.2 (C-2', C-6'), 130.9 (C-6''), 131.4 (C-3''), 133.4 (C-1'', C-4''), 134.9 (C-4'''), 136.4 (C-1'), 148.3 (C-2''), 170.7 (C-1). HRMS (ESI): calc (found) for $C_{22}H_{26}N_2O_6S$ ($[M+Na]^+$): 469.1404 (469.1403).

Methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-D-phenylalaninate (27g)



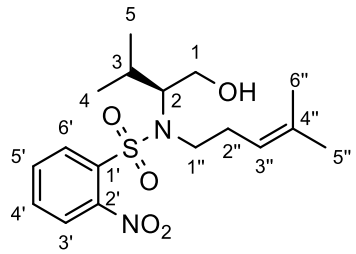
27g

According to GP3 **27g** was prepared from methyl-((2-nitrophenyl)sulfonyl)-D-phenylalaninate **26g** (4.37 g, 12.0 mmol), cesium carbonate (5.86 g, 18.0 mmol) and 5-bromo-2-methylpent-2-ene **S1** (2.94 g, 18.0 mmol) as a yellow oil (3.03 g, 3.18 mmol, 56 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1). $[\alpha]_D^{20}$ + 21 (*c* 1.0 in $CHCl_3$). R_f = 0.58 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3029 (w), 2953 (w), 1741 (s), 1589 (w), 1542 (vs), 1497 (w), 1454 (m), 1438 (m), 1373 (s), 1350 (vs), 1205 (m), 1163 (vs), 1126 (s), 1090 (m), 1069 (m), 1008 (m), 949 (m), 895 (w), 852 (m), 838 (w), 775 (m), 748 (vs), 700 (s), 652 (m), 588 (s), 576 (s) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.61 (s, 3H, 5'''-H/6'''-H), 1.68 (s, 3H, 6'''-H/5'''-H), 2.19 – 2.38 (m, 2H, 2'''-H), 3.04 (dd, *J* = 14.2 Hz, 7.2 Hz, 1H, 3-H_a), 3.21 (ddd, *J* = 15.2 Hz, 11.3 Hz, 5.5 Hz, 1H, 1'''-H_a), 3.34 – 3.45 (m, 2H, 1'''-H_b, 3-H_b), 3.54 (s, 3H, OMe), 4.89 (t, *J* = 7.2 Hz, 1H, 2-H), 4.99 – 5.07 (m, 1H, 3'''-H), 7.18 – 7.30 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 7.54 – 7.61 (m, 2H, 3''-H, 5''-H), 7.62 – 7.68 (m, 1H, 4''-H), 7.82 – 7.87 (m, 1H, 6''-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.9 (C-5'''/C-6'''), 25.7 (C-6'''/C-5'''), 29.4 (C-2'''), 36.8 (C-3), 46.2 (C-1'''), 52.3 (OMe), 61.3 (C-2), 119.7 (C-3'''), 124.0 (C-5''), 127.0 (C-4'), 128.6 (C-3', C-5'), 129.2 (C-2', C-6'), 130.8 (C-6''), 131.5 (C-3''), 133.3 (C-1''), 133.4 (C-4''), 135.0 (C-4'''), 136.4 (C-1'), 148.2 (C-2''), 170.7 (C-1). HRMS (ESI): calc (found) for $C_{22}H_{26}N_2O_6S$ ($[M+Na]^+$): 469.1404 (469.1403).

GP4: Reduction via lithium aluminium hydride

The amino alcohols were prepared according to the literature.[6] A round bottom flask was charged with a solution of the methyl ester (4.00 mmol) in tetrahydrofuran (18 mL) under nitrogen atmosphere. Upon cooling to 0 °C a solution of lithium aluminium hydride (0.17 g, 4.40 mmol, 0.5 M in THF) was added dropwise. The rection mixture was stirred for 30 min at 0 °C. Afterwards the reaction was quenched with water (1.0 mL) and dichloromethane (35 mL) was added. The organic phase was washed with hydrochloric acid (1 × 15 mL, 1 M in Wasser) and brine (2 × 15 mL) and dried over magnesium sulfate. The solvents were evaporated and the crude product was purified by column chromatography. The products were isolated as oils.

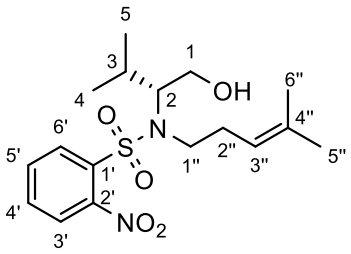
(*S*)-*N*-(1-Hydroxy-3-methylbutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene sulfonamide (28a)



28a

According to GP4 **28a** was prepared from methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-L-valinate **27a** (0.98 g, 2.45 mmol) and lithium aluminium hydride (0.10 g, 2.70 mmol) as a yellow oil (0.66 g, 1.77 mmol, 72 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). $[\alpha]_D^{20}$ + 59 (*c* 1.0 in $CHCl_3$). R_f = 0.35 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3556 (w), 3097 (w), 2968 (m), 2929 (m), 2879 (w), 1590 (w), 1544 (vs), 1469 (w), 1440 (w), 1373 (s), 1341 (s), 1295 (w), 1249 (w), 1202 (w), 1160 (s), 1125 (m), 1089 (w), 1065 (m), 1000 (w), 956 (w), 923 (w), 852 (w), 774 (w), 745 (w), 653 (w), 594 (m), 528 (w) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.80 (d, *J* = 6.6 Hz, 3H, 4-H/5-H), 0.96 (d, *J* = 6.6 Hz, 3H, 5-H/4-H), 1.65 (s, 3H, 5"-H/6"-H), 1.70 (s, 3H, 6"-H/5"-H), 1.73 – 1.87 (m, 1H, 3-H), 1.87 – 2.29 (m, 1H, OH), 2.32 – 2.56 (m, 2H, 2"-H), 3.12 (ddd, *J* = 15.2 Hz, 11.4 Hz, 5.4 Hz, 1H, 1"-H_a), 3.34 (ddd, *J* = 15.2 Hz, 11.4 Hz, 5.4 Hz, 1H, 1"-H_b), 3.51 – 3.66 (m, 2H, 1-H_a, 2-H), 3.81 – 3.96 (m, 1H, 1-H_b), 5.07 (t, *J* = 7.3 Hz, 1H, 3"-H), 7.51 – 7.60 (m, 1H, 3'-H), 7.62 – 7.72 (m, 2H, 4'-H, 5'-H), 8.03 – 8.14 (m, 1H, 6'-H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 17.9 (C-5"/C-6"), 20.2 (C-4/C-5), 20.3 (C-5/C-4), 25.7 (C-6"/C-5"), 28.9 (C-3), 30.1 (C-2"), 44.4 (C-1"), 62.1 (C-1), 66.1 (C-2), 120.0 (C-3"), 123.8 (C-3'), 131.3 (C-6'), 131.4 (C-5'), 133.3 (C-4'), 134.0 (C-1'), 134.9 (C-4"), 147.9 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_5S$ ($[M+Na]^+$): 393.1455 (393.1465). The spectral data are in accordance with previous reported literature.[6]

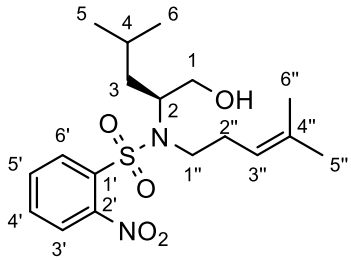
(*R*)-*N*-(1-Hydroxy-3-methylbutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide (28b)



28b

According to GP4 **28b** was prepared from methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-D-valinate **27b** (2.00 g, 5.02 mmol) and lithium aluminium hydride (0.21 g, 5.52 mmol) as a yellow oil (1.33 g, 3.60 mmol, 72 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). $[\alpha]_D^{20}$ - 65 (*c* 1.0 in $CHCl_3$). R_f = 0.30 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3551 (w), 2967 (w), 2254 (w), 1543 (s), 1470 (w), 1440 (w), 1372 (m), 1340 (m), 1202 (w), 1156 (m), 1125 (m), 1088 (w), 1064 (m), 999 (w), 956 (w), 907 (vs), 852 (m), 771 (w), 726 (vs), 650 (m), 592 (s), 450 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.78 (d, *J* = 6.5 Hz, 3H, 4-H/5-H), 0.95 (d, *J* = 6.5 Hz, 3H, 5-H/4-H), 1.64 (s, 3H, 5"-H/6"-H), 1.69 (s, 3H, 6"-H/5"-H), 1.74 – 1.88 (m, 1H, 3-H), 2.25 (s, br, 1H, OH), 2.33 – 2.54 (m, 2H, 2"-H), 3.12 (ddd, *J* = 15.1 Hz, 11.5 Hz, 5.4 Hz, 1H, 1"-H_a), 3.34 (ddd, *J* = 15.1 Hz, 11.5 Hz, 5.4 Hz, 1H, 1"-H_b), 3.52 – 3.66 (m, 2H, 1-H_a, 2-H), 3.82 – 3.93 (m, 1H, 1-H_b), 5.03 – 5.11 (m, 1H, 3"-H), 7.52 – 7.58 (m, 1H, 3'-H), 7.64 – 7.71 (m, 2H, 4'-H, 5'-H), 8.04 – 8.12 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.9 (C-5"/C-6"), 20.2 (C-4/C-5), 20.3 (C-5/C-4), 25.7 (C-6"/C-5"), 28.8 (C-3), 30.1 (C-2"), 44.4 (C-1"), 62.1 (C-1), 66.0 (C-2), 120.0 (C-3"), 123.8 (C-3'), 131.2 (C-6'), 131.5 (C-5'), 133.4 (C-4'), 133.8 (C-1'), 134.8 (C-4"), 147.8 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_5S$ ($[M+Na]^+$): 393.1455 (393.1459).

(*S*)-*N*-(1-Hydroxy-4-methylpentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide (28c)

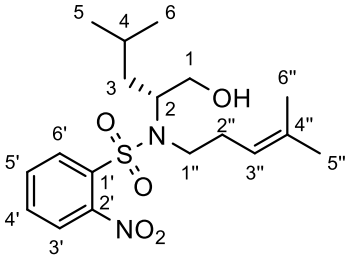


28c

According to GP4 **28c** was prepared from methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-L-leucinate **27c** (2.42 g, 5.87 mmol) and lithium aluminium hydride (0.25 g, 6.46 mmol) as a yellow oil (1.69 g, 4.39 mmol, 75 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1 → 1 : 1). $[\alpha]_D^{20}$ + 15 (*c* 1.0 in $CHCl_3$). R_f = 0.24 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3543 (w), 2958 (m), 1545 (vs), 1468 (w), 1440 (w), 1372 (s), 1341 (m), 1202 (w), 1158 (s), 1125 (m), 1064 (w), 949 (w), 852 (w), 745 (w), 653 (w), 590 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.82 (d, *J* = 6.6 Hz, 3H, 5-H/6-H), 0.84 (d, *J* = 6.6 Hz, 3H, 6-H/5-H), 1.30 (ddd, *J* = 14.1 Hz, 7.1 Hz,

7.1 Hz, 1H, 3-H_a), 1.39 (ddd, *J* = 14.1 Hz, 7.1 Hz, 7.1 Hz, 1H, 3-H_b), 1.55 – 1.63 (m, 1H, 4-H), 1.64 (s, 3H, 5"-H/6"-H), 1.71 (s, 3H, 6"-H/5"-H), 2.13 (s, br, 1H, OH), 2.28 – 2.37 (m, 1H, 2"-H_a), 2.39 – 2.50 (m ,1H, 2"-H_b), 3.22 (t, *J* = 8.3 Hz, 2H, 1"-H), 3.52 – 3.63 (m, 2H, 1-H), 3.84 – 3.92 (m, 1H, 2-H), 5.05 – 5.12 (m, 1H, 3"-H), 7.57 – 7.63 (m, 1H, 3'-H), 7.65 – 7.72 (m, 2H, 4'-H, 5'-H), 8.08 – 8.14 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.9 (C-5"/C-6"), 22.5 (C-5, C-6), 24.6 (C-4), 25.7 (C-6"/C-5"), 30.6 (C-8), 39.3 (C-3), 43.8 (C-1"), 58.3 (C-2), 63.3 (C-1), 119.9 (C-3"), 124.1 (C-3'), 131.6 (C-5', C-6'), 133.5 (C-4'), 133.7 (C-1'), 134.9 (C-4"), 147.9 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{28}N_2O_5S$ ($[M+Na]^+$): 407.1611 (407.1618).

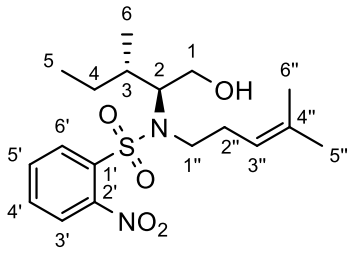
(*R*)-*N*-(1-Hydroxy-4-methylpentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide (28d)



28d

According to GP4 **28d** was prepared from methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-D-leucinate **27d** (2.02 g, 4.88 mmol) and lithium aluminium hydride (0.20 g, 5.37 mmol) as a yellow oil (1.26 g, 3.25 mmol, 67 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1 → 1 : 1). $[\alpha]_D^{20}$ - 18 (*c* 1.0 in $CHCl_3$). R_f = 0.60 (hexanes/EtOAc, 1 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3552 (w), 2958 (m), 2929 (m), 2873 (w), 1545 (vs), 1467 (w), 1373 (s), 1342 (s), 1201 (w), 1159 (s), 1125 (m), 1063 (w), 949 (w), 852 (w), 777 (w), 745 (w), 653 (w), 591 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.82 (d, *J* = 6.6 Hz, 3H, 5-H/6-H), 0.84 (d, *J* = 6.6 Hz, 3H, 6-H/5-H), 1.30 (ddd, *J* = 14.1 Hz, 7.1 Hz, 7.1 Hz, 1H, 3-H_a), 1.37 (ddd, *J* = 14.1 Hz, 7.1 Hz, 7.1 Hz, 1H, 3-H_b), 1.53 – 1.62 (m, 1H, 4-H), 1.64 (s, 3H, 5"-H/6"-H), 1.70 (s, 3H, 6"-H/5"-H), 2.27 –2.38 (m, 1H, 2"-H_a), 2.39 – 2.50 (m ,1H, 2"-H_b), 3.22 (t, *J* = 8.3 Hz, 2H, 1"-H), 3.51 – 3.63 (m, 2H, 1-H), 3.83 – 3.92 (m, 1H, 2-H), 5.03 – 5.12 (m, 1H, 3"-H), 7.55 – 7.63 (m, 1H, 3'-H), 7.63 – 7.74 (m, 2H, 4'-H, 5'-H), 8.04 – 8.18 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.9 (C-5"/C-6"), 22.5 (C-5, C-6), 24.7 (C-4), 25.7 (C-6"/C-5"), 30.6 (C-8), 39.3 (C-3), 43.9 (C-1"), 58.3 (C-2), 63.3 (C-1), 119.9 (C-3"), 124.1 (C-3'), 131.6 (C-5', C-6'), 133.5 (C-4'), 133.7 (C-1'), 134.9 (C-4"), 148.0 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{28}N_2O_5S$ ($[M+H]^+$): 385.1792 (385.1795).

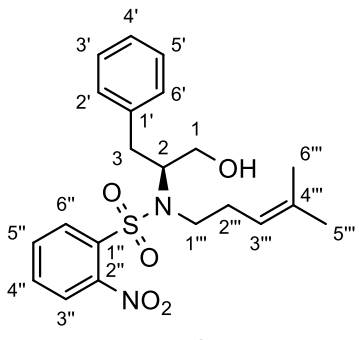
***N*-((2*S*,3*S*)-1-Hydroxy-3-methylpentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide (28e)**



28e

According to GP4 **28e** was prepared from methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-L-leucinate **27e** (1.96 g, 4.75 mmol) and lithium aluminium hydride (0.20 g, 5.22 mmol) as a yellow oil (0.76 g, 1.97 mmol, 42 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). $[\alpha]_D^{20}$ + 42 (*c* 2.0 in $CHCl_3$). R_f = 0.23 (hexanes/EtOAc, 4 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3544 (w, br), 2966 (m), 2931 (m), 2877 (m), 1591 (w), 1543 (vs), 1457 (m), 1440 (m), 1372 (s), 1340 (s), 1202 (w), 1155 (s), 1125 (s), 1080 (m), 1066 (m), 995 (m), 959 (m), 913 (w), 852 (m), 773 (m), 744 (m), 653 (m), 580 (s), 451 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.80 (t, *J* = 6.9 Hz, 3H, 5-H), 0.82 – 0.89 (m, 1H, 4-H_a), 0.91 (d, *J* = 6.6 Hz, 3H, 6-H), 1.46 – 1.63 (m, 2H, 3-H, 4-H_b), 1.65 (s, 3H, 5"-H/6"-H), 1.70 (s, 3H, 6"-H/5"-H), 1.90 – 2.13 (m, 1H, OH), 2.26 – 2.40 (m, 1H, 2"-H_a), 2.44 – 2.58 (m, 1H, 2"-H_b), 3.17 (ddd, *J* = 15.1 Hz, 11.5 Hz, 5.4 Hz, 1H, 1"-H_a), 3.25 (ddd, *J* = 15.1 Hz, 11.5 Hz, 5.4 Hz, 1H, 1"-H_b), 3.57 – 3.66 (m, 2H, 1-H_a, 2-H), 3.81 – 3.88 (m, 1H, 1-H_b), 5.07 (t, *J* = 7.2 Hz, 1H, 3"-H), 7.53 – 7.56 (m, 1H, 3'-H), 7.64 – 7.69 (m, 2H, 4'-H, 5'-H), 8.05 – 8.09 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 11.2 (C-5), 15.9 (C-6), 17.9 (C-5"/C-6"), 25.7 (C-4/(C-6"/C-5")), 25.8 ((C-5"/C-6")/C-4), 30.1 (C-2"), 35.6 (C-3), 44.5 (C-1"), 62.1 (C-1), 64.9 (C-2), 120.0 (C-3"), 123.8 (C-3'), 131.3 (C-6'), 131.5 (C-5'), 133.3 (C-4'), 133.9 (C-1'), 134.9 (C-4"), 147.8 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{28}N_2O_5S$ ($[M+Na]^+$): 407.1611 (407.1618).

(*S*)-*N*-(1-Hydroxy-3-phenylpropan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide (28f)

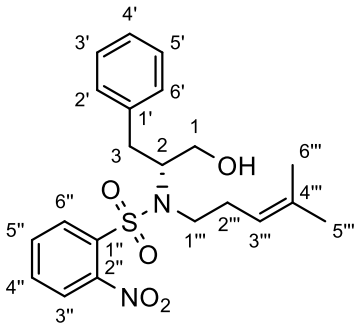


28f

According to GP4 **28f** was prepared from methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-L-phenylalaninate **27f** (5.37 g, 12.0 mmol) and lithium aluminium hydride (0.50 g, 13.2 mmol) as a yellow oil (2.85 g, 6.80 mmol, 57 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1 → 1 : 1). $[\alpha]_D^{20}$ + 6 (*c* 1.0 in $CHCl_3$). R_f = 0.37 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3538 (w), 2923 (w), 1543 (vs),1496 (w), 1455 (m), 1372 (s), 1339 (s), 1160 (s), 1126 (m), 1034 (m), 983 (m), 939 (m), 853 (m), 743 (m), 701 (m),

653 (w), 579 (m) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.65 (s, 3H, 5'''-H/6'''-H), 1.71 (s, 3H, 6'''-H/5'''-H), 1.94 – 2.16 (s, br, 1H, OH), 2.41 (q, *J* = 7.9 Hz, 2H, 2'''-H), 2.78 (dd, *J* = 13.6 Hz, 9.2 Hz, 1H, 3-H_a), 2.94 (dd, *J* = 13.6 Hz, 5.6 Hz, 1H, 3-H_b), 3.26 – 3.47 (m, 2H, 1'''-H), 3.59 – 3.70 (m, 2H, 1-H), 4.05 – 4.16 (m, 1H, 2-H), 5.05 – 5.16 (m, 1H, 3'''-H), 7.05 – 7.12 (m, 2H, 2'-H, 6'-H), 7.12 – 7.23 (m, 3H, 3'-H, 4'-H, 5'-H), 7.53 – 7.68 (m, 3H, 3''-H, 4''-H, 5''-H), 7.91 – 8.01 (m, 1H, 6''-H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 17.9 (C-5'''/C-6'''), 25.7 (C-6'''/C-5'''), 30.4 (C-2'''), 37.2 (C-3), 44.4 (C-1'''), 61.9 (C-2), 62.3 (C-1), 119.9 (C-3'''), 124.2 (C-5''), 126.8 (C-4'), 128.6 (C-3', C-5'), 129.0 (C-2', C-6'), 131.3 (C-6''), 131.7 (C-3''), 133.5 (C-4''), 133.6 (C-1''), 135.0 (C-4'''), 137.4 (C-1'), 147.9 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_5S$ ($[M+Na]^+$): 441.1455 (441.1456).

(*R*)-*N*-(1-Hydroxy-3-phenylpropan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide (28g)



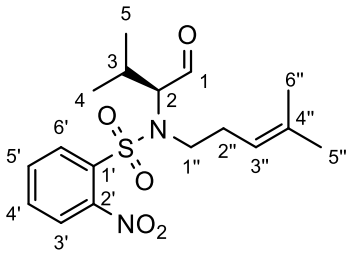
28g

According to GP4 **28g** was prepared from methyl-*N*-(4-methylpent-3-en-1-yl)-*N*-((2-nitrophenyl)sulfonyl)-D-phenyl-alaninate **27g** (3.03 g, 6.77 mmol) and lithium aluminium hydride (0.28 g, 7.45 mmol) as a yellow oil (2.14 g, 5.11 mmol, 75 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1 → 1 : 1). $[\alpha]_D^{20}$ - 7 (*c* 1.0 in $CHCl_3$). R_f = 0.37 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3557 (w), 2923 (w), 1543 (vs),1496 (w), 1454 (m), 1372 (s), 1340 (s), 1160 (s), 1125 (m), 1033 (w), 982 (w), 939 (w), 853 (w), 742 (m), 701 (m), 653 (w), 578 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.65 (s, 3H, 5'''-H/6'''-H), 1.72 (s, 3H, 6'''-H/5'''-H), 2.42 (q, *J* = 7.9 Hz, 2H, 2'''-H), 2.78 (dd, *J* = 13.6 Hz, 9.2 Hz, 1H, 3-H_a), 2.95 (dd, *J* = 13.6 Hz, 5.6 Hz, 1H, 3-H_b), 3.29 – 3.44 (m, 2H, 1'''-H), 3.60 – 3.70 (m, 2H, 1-H), 4.05 – 4.15 (m, 1H, 2-H), 5.07 – 5.15 (m, 1H, 3'''-H), 7.07 – 7.12 (m, 2H, 2'-H, 6'-H), 7.12 – 7.23 (m, 3H, 3'-H, 4'-H, 5'-H), 7.55 – 7.69 (m, 3H, 3''-H, 4''-H, 5''-H), 7.96 – 8.02 (m, 1H, 6''-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 18.0 (C-5'''/C-6'''), 25.7 (C-6'''/C-5'''), 30.4 (C-2'''), 37.2 (C-3), 44.3 (C-1'''), 61.9 (C-2), 62.2 (C-1), 119.9 (C-3'''), 124.3 (C-5''), 126.8 (C-4'), 128.6 (C-3', C-5'), 129.0 (C-2', C-6'), 131.4 (C-6''), 131.7 (C-3''), 133.5 (C-4''), 133.7 (C-1''), 135.1 (C-4'''), 137.4 (C-1'), 148.0 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_5S$ ($[M+Na]^+$): 441.1455 (441.1458).

GP5: Oxidation with Dess-Martin-Periodinane (DMP)

The aldehydes or ketones were prepared according to the literature.[6] A round bottom flask was charged with a solution of the primary or secondary alcohol (3.00 mmol) in dichloromethane (50 mL). DMP (3.18 g, 7.50 mmol) was added at room temperature and the mixture was stirred for 40 min at room temperature. Diethyl ether (130 mL) and a mixture (25 mL, 1 : 1) of saturated solutions of sodium thiosulfate and sodium bicarbonate were added and stirring continued for 15 min at room temperature until a clear solution was obtained. The phases were separated and the aqueous phase was extracted with diethyl ether (1 × 45 mL). The organic phases were washed with saturated sodium bicarbonate solution, water and brine (each 2 × 45 mL) and dried over magnesium sulfate. The solvents were evaporated and the crude product was purified by column chromatography. The products were isolated as oils.

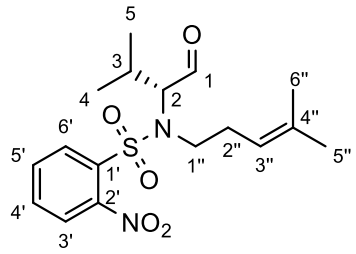
(*S*)-*N*-(3-Methyl-1-oxobutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide (29a)



29a

According to GP5 **29a** was prepared from (*S*)-*N*-(1-Hydroxy-3-methylbutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide **28a** (0.62 g, 1.68 mmol) and DMP (1.78 g, 4.20 mmol) as a yellow oil (0.41 g, 1.10 mmol, 65 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1). $[\alpha]_D^{20}$ + 37 (*c* 1.0 in $CHCl_3$). R_f = 0.73 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3095 (w), 2968 (m), 2932 (w), 2877 (w), 2729 (w), 1732 (m), 1590 (w), 1544 (vs), 1470 (w), 1440 (w), 1372 (s), 1348 (s), 1297 (w), 1249 (w), 1203 (w), 1162 (s), 1126 (m), 1104 (w), 1070 (w), 1002 (w), 975 (w), 960 (w), 852 (m), 778 (m), 745 (m), 676 (w), 653 (w), 588 (m) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.97 (d, *J* = 6.6 Hz, 3H, 4-H/5-H), 1.16 (d, *J* = 6.6 Hz, 3H, 5-H/4-H), 1.60 (s, 3H, 5"-H/6"-H), 1.67 (s, 3H, 6"-H/5"-H), 2.12 – 2.31 (m, 2H, 3-H, 2"-H_a), 2.31 – 2.44 (m, 1H, 2"-H_b), 3.16 (ddd, *J* = 15.2 Hz, 11.2 Hz, 5.4 Hz, 1H, 1"-H_a), 3.27 (ddd, *J* = 15.2 Hz, 11.2 Hz, 5.7 Hz, 1H, 1"-H_b), 4.20 (d, *J* = 10.1 Hz, 1H, 2-H), 4.98 (t, *J* = 7.0 Hz, 1H, 3"-H), 7.57 – 7.63 (m, 1H, 3'-H), 7.66 – 7.74 (m, 2H, 4'-H, 5'-H), 8.06 – 8.12 (m, 1H, 6'-H), 9.73 (s, 1H, 1-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 17.8 (C-5"/C-6"), 20.1 (C-4/C-5), 20.2 (C-5/C-4), 25.7 (C-6"/C-5"), 27.3 (C-3), 29.6 (C-2"), 46.7 (C-1"), 71.5 (C-2), 119.3 (C-3"), 124.2 (C-3'), 131.1 (C-6'), 131.7 (C-5'), 133.5 (C-1'), 133.6 (C-4'), 135.2 (C-4"), 147.9 (C-2'), 199.0 (C-1). HRMS (ESI): calc (found) for $C_{17}H_{24}N_2O_5S$ ($[M+H]^+$): 369.1479 (369.1479). The spectral data are in accordance with previous reported literature.[6]

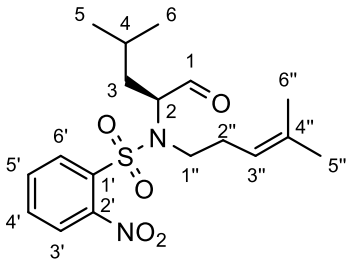
(*R*)-*N*-(3-Methyl-1-oxobutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide (29b)



29b

According to GP5 **29b** was prepared from (*R*)-*N*-(1-hydroxy-3-methylbutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide **28b** (0.55 g, 1.48 mmol) and DMP (1.57 g, 3.70 mmol) as a yellow oil (0.41 g, 1.12 mmol, 76 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1). $[\alpha]_D^{20}$ - 36 (*c* 1.0 in $CHCl_3$). R_f = 0.48 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3560 (w), 3092 (w), 2969 (m), 2929 (w), 2880 (w), 2858 (w), 1733 (m), 1591 (w), 1544 (vs), 1470 (w), 1440 (w), 1373 (s), 1346 (m), 1161 (s), 1126 (m), 1072 (w), 976 (w), 914 (w), 852 (w), 776 (w), 752 (m), 672 (w), 653 (w), 580 (m), 563 (w) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 0.96 (d, *J* = 6.6 Hz, 3H, 4-H/5-H), 1.15 (d, *J* = 6.6 Hz, 3H, 5-H/4-H), 1.59 (s, 3H, 5"-H/6"-H), 1.66 (s, 3H, 6"-H/5"-H), 2.18 (dqq, *J* = 10.1 Hz, 6.6 Hz, 6.6 Hz, 1H, 3-H), 2.20 – 2.29 (m, 1H, 2"-H_a), 2.32 – 2.43 (m, 1H, 2"-H_b), 3.16 (ddd, *J* = 15.2 Hz, 11.6 Hz, 5.4 Hz, 1H, 1"-H_a), 3.26 (ddd, *J* = 15.2 Hz, 11.6 Hz, 5.4 Hz, 1H, 1"-H_b), 4.20 (d, *J* = 10.1 Hz, 1H, 2-H), 4.97 (t, *J* = 7.3 Hz, 1H, 3"-H), 7.58 – 7.63 (m, 1H, 6'-H), 7.67 – 7.73 (m, 2H, 4'-H, 5'-H), 8.06 – 8.11 (m, 1H, 3'-H), 9.73 (s, 1H, 1-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 17.8 (C-5"/C-6"), 20.1 (C-4/C-5), 20.2 (C-5/C-4), 25.7 (C-6"/C-5"), 27.3 (C-3), 29.6 (C-2"), 46.7 (C-1"), 71.5 (C-2), 119.4 (C-3"), 124.2 (C-6'), 131.1 (C-3'), 131.7 (C-4'/C-5'), 133.5 (C-1'), 133.7 (C-5'/C-4'), 135.2 (C-4"), 147.9 (C-2'), 199.0 (C-1). HRMS (ESI): calc (found) for $C_{17}H_{24}N_2O_5S$ ($[M+Na]^+$): 391.1298 (391.1299).

(*S*)-*N*-(4-Methyl-1-oxopentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide (29c)

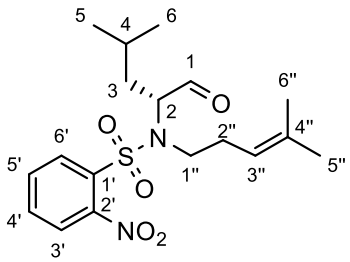


29c

According to GP5 **29c** was prepared from (*S*)-*N*-(1-hydroxy-4-methylpentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitro-benzenesulfonamide **28c** (0.18 g, 0.48 mmol) and DMP (0.41 g, 0.96 mmol) as a yellow oil (0.12 g, 0.32 mmol, 67 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1). $[\alpha]_D^{20}$ + 30 (*c* 1.0 in $CHCl_3$). R_f = 0.63 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 2960 (m), 1736 (m), 1545 (vs), 1440 (w), 1372 (s), 1161 (s), 1126 (m), 1069 (w), 948 (w), 852 (w), 777 (m), 746 (m), 653 (w), 588 (m), 573 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.90 (d, *J* = 5.8 Hz,

3H, 5-H/6-H), 0.95 (d, J = 5.8 Hz, 3H, 6-H/5-H), 1.47 – 1.56 (m, 1H, 3-H_a), 1.61 (s, 3H, 5''-H/6''-H), 1.67 (s, 3H, 6''-H/5''-H), 1.72 – 1.82 (m, 2H, 3-H_b, 4-H), 2.14 – 2.24 (m, 1H, 2''-H_a), 2.35 – 2.45 (m, 1H, 2''-H_b), 3.09 – 3.18 (m, 1H, 1''-H_a), 3.25 – 3.34 (m, 1H, 1''-H_b), 4.53 (dd, J = 10.0 Hz, 4.0 Hz, 1H, 2-H), 4.98 – 5.04 (m, 1H, 3''-H), 7.60 – 7.64 (m, 1H, 3'-H), 7.68 – 7.75 (m, 2H, 4'-H, 5'-H), 8.06 – 8.12 (m, 1H, 6'-H), 9.56 (s, 1H, 1-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.9 (C-5/C-6), 21.3 (C-5''/C-6''), 23.1 (C-6''/C-5''), 24.3 (C-4), 25.7 (C-6/C-5), 29.8 (C-2''), 35.7 (C-3), 46.3 (C-1), 64.7 (C-2), 119.3 (C-3''), 124.3 (C-3'), 131.2 (C-6'), 131.7 (C-5'), 133.1 (C-1'), 133.7 (C-4'), 135.3 (C-4''), 148.1 (C-2'), 199.9 (C-1). HRMS (ESI): calc (found) for $C_{18}H_{26}N_2O_5S$ ($[M+Na]^+$): 405.1455 (405.1455).

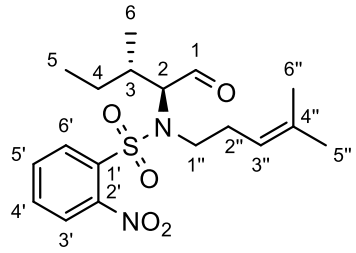
(*R*)-*N*-(4-Methyl-1-oxopentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide (29d)



29d

According to GP5 **29d** was prepared from (*R*)-*N*-(1-hydroxy-4-methylpentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide **28d** (1.01 g, 2.64 mmol) and DMP (2.24 g, 5.28 mmol) as a yellow oil (0.79 g, 2.07 mmol, 78 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1). $[\alpha]_D^{20}$ - 15 (*c* 1.0 in $CHCl_3$). R_f = 0.62 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 2958 (m), 2871 (w), 1735 (m),1675 (w), 1589 (w), 1545 (vs), 1467 (m), 1440 (w), 1372 (s), 1347 (s), 1161 (vs), 1126 (m), 1069 (m), 949 (m), 852 (m), 776 (m), 746 (m), 653 (w), 587 (m), 575 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.90 (d, J = 5.8 Hz, 3H, 5-H/6-H), 0.95 (d, J = 5.8 Hz, 3H, 6-H/5-H), 1.47 – 1.56 (m, 1H, 3-H_a), 1.61 (s, 3H, 5''-H/6''-H), 1.68 (s, 3H, 6''-H/5''-H), 1.72 – 1.82 (m, 2H, 3-H_b, 4-H), 2.14 – 2.23 (m, 1H, 2''-H_a), 2.35 – 2.45 (m, 1H, 2''-H_b), 3.13 (ddd, J = 15.1 Hz, 11.0 Hz, 5.7 Hz, 1H, 1''-H_a), 3.29 (ddd, J = 15.1 Hz, 11.0 Hz, 5.1 Hz, 1H, 1''-H_b), 4.53 (dd, J = 10.0 Hz, 4.0 Hz, 1H, 2-H), 4.97 – 5.04 (m, 1H, 3''-H), 7.58 – 7.64 (m, 1H, 3'-H), 7.67 – 7.75 (m, 2H, 4'-H, 5'-H), 8.06 – 8.12 (m, 1H, 6'-H), 9.56 (s, 1H, 1-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.9 (C-5/C-6), 21.3 (C-5''/C-6''), 23.0 (C-6''/C-5''), 24.3 (C-4), 25.7 (C-6/C-5), 29.8 (C-2), 35.7 (C-3), 46.3 (C-1), 64.7 (C-2), 119.3 (C-3''), 124.3 (C-3'), 131.2 (C-6'), 131.7 (C-5'), 133.1 (C-1'), 133.7 (C-4'), 135.3 (C-4''), 148.1 (C-2'), 199.9 (C-1). HRMS (ESI): calc (found) for $C_{18}H_{26}N_2O_5S$ ($[M+Na]^+$): 405.1455 (405.1461).

***N*-((2*S*,3*S*)-3-Methyl-1-oxopentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide (29e)**



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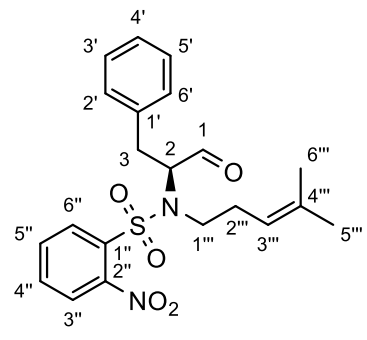
According to GP5 **29e** was prepared from *N*-((2*S*,3*S*)-1-hydroxy-3-methylpentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitro-benzenesulfonamide **28e** (0.76 g, 1.98 mmol) and DMP (2.10 g, 4.95 mmol) as a yellow oil (0.72 g, 1.88 mmol, 95 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (5 : 1 → 4 : 1 → 3 : 1). $[\alpha]_D^{20}$ + 23 (*c* 2.6 in $CHCl_3$). R_f = 0.40 (hexanes/EtOAc, 4 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 2966 (m), 2928 (m), 2878 (w), 1731 (m), 1673 (w), 1590 (w), 1545 (vs), 1462 (m), 1440 (w), 1373 (s), 1349 (s), 1203 (w), 1161 (s), 1126 (m), 1069 (w), 1007 (w), 962 (w), 852 (w), 777 (m), 746 (m), 691 (w), 653 (w), 586 (m), 456 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.91 (t, *J* = 7.4 Hz, 3H, 5-H), 1.09 – 1.16 (m, 1H, 4-H_a), 1.13 (d, *J* = 6.7 Hz, 3H, 6-H), 1.62 (s, 3H, 5"-H/6"-H), 1.64 – 1.70 (m, 1H, 4-H_b), 1.68 (s, 3H, 6"-H/5"-H), 1.85 – 1.97 (m, 1H, 3-H), 2.16 – 2.26 (m, 1H, 2"-H_a), 2.38 – 2.50 (m, 1H, 2"-H_b), 3.17 (ddd, *J* = 15.1 Hz, 11.5 Hz, 5.7 Hz, 1H, 1"-H_a), 3.28 (ddd, *J* = 15.1 Hz, 11.5 Hz, 5.7 Hz, 1H, 1"-H_b), 4.30 (d, *J* = 9.8 Hz, 1H, 2-H), 4.96 – 5.03 (m, 1H, 3"-H), 7.54 – 7.64 (m, 1H, 3'-H), 7.65 – 7.75 (m, 2H, 4'-H, 5'-H), 8.01 – 8.12 (m, 1H, 6'-H), 9.72 (s, 1H, 1-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 10.7 (C-5), 16.1 (C-6), 17.9 (C-5"/C-6"), 25.7 (C-6"/C-5"), 26.0 (C-4), 29.8 (C-2"), 34.0 (C-3), 46.9 (C-1"), 70.6 (C-2), 119.4 (C-3"), 124.2 (C-3'), 131.1 (C-6'), 131.7 (C-1'), 133.2 (C-5'), 133.7 (C-4'), 135.2 (C-4"), 147.9 (C-2'), 199.2 (C-1). HRMS (ESI): calc (found) for $C_{18}H_{26}N_2O_5S$ ($[M+Na]^+$): 405.1455 (405.1448).

(*S*)-*N*-(4-Methylpent-3-en-1-yl)-2-nitro-*N*-(1-oxo-3-phenylpropan-2-yl)benzene-sulfonamide (29f)

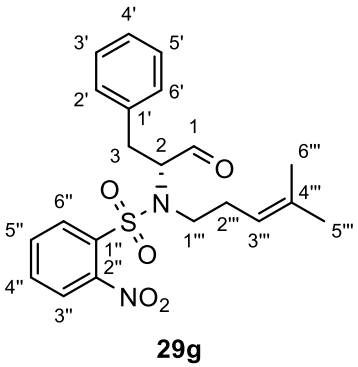


29f

According to GP5 **29f** was prepared from (*S*)-*N*-(1-hydroxy-3-phenylpropan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitro-benzenesulfonamide **28f** (0.45 g, 1.08 mmol) and DMP (1.15 g, 2.70 mmol) as a yellow oil (0.44 g, 1.07 mmol, 99 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (4 : 1 → 2 : 1). $[\alpha]_D^{20}$ - 20 (*c* 3.9 in $CHCl_3$). R_f = 0.44 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3029 (w), 2915 (w), 1735 (m), 1590 (w), 1542 (vs), 1498 (w), 1454 (w), 1439 (w), 1371 (s), 1346 (s), 1254 (w), 1204 (w), 1162 (s), 1126 (m), 1096 (w), 1076

(w), 1031 (w), 961 (m), 912 (w), 852 (m), 740 (s), 700 (m), 653 (w), 576 (m), 541 (w), 494 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.60 (s, 3H, 5'''-H/6'''-H), 1.68 (s, 3H, 6'''-H/5'''-H), 2.19 – 2.30 (m, 2H, 2'''-H), 2.89 (dd, *J* = 14.4 Hz, 8.4 Hz, 1H, 3-H_a), 3.26 – 3.35 (m, 1H, 1'''-H_a), 3.35 – 3.45 (m, 2H, 1'''-H_b, 3-H_b), 4.73 (dd, *J* = 8.4 Hz, 6.4 Hz, 1H, 2-H), 4.99 – 5.05 (m, 1H, 3'''-H), 7.10 – 7.20 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 7.51 – 7.60 (m, 2H, 3"-H, 5"-H), 7.62 – 7.68 (m, 1H, 4"-H), 7.78 – 7.84 (m, 1H, 6"-H), 9.70 (s, 1H, 1-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.9 (C-5'''/C-6'''), 25.7 (C-6'''/C-5'''), 29.3 (C-2'''), 33.0 (C-3), 46.0 (C-1'''), 67.1 (C-2), 119.1 (C-3'''), 124.4 (C-3"), 126.9 (C-4'), 128.6 (C-3', C-5'), 129.0 (C-2', C-6'), 130.9 (C-6"), 131.8 (C-5"), 133.3 (C-1"), 133.6 (C-4'''), 135.6 (C-4"), 136.2 (C-1'), 147.8 (C-2"), 199.3 (C-1). HRMS (ESI): calc (found) for $C_{21}H_{24}N_2O_5S$ ($[M+H]^+$): 417.1479 (417.1480).

(*R*)-*N*-(4-Methylpent-3-en-1-yl)-2-nitro-*N*-(1-oxo-3-phenylpropan-2-yl)benzene-sulfonamide (29g)



29g

According to GP5 **29g** was prepared from (*R*)-*N*-(1-hydroxy-3-phenylpropan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzene-sulfonamide **28g** (1.34 g, 3.20 mmol) and DMP (2.71 g, 6.40 mmol) as a yellow oil (1.03 g, 2.46 mmol, 77 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (4 : 1 → 2 : 1). $[\alpha]_D^{20}$ + 35 (*c* 1.0 in $CHCl_3$). R_f = 0.42 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3066 (w), 3029 (w), 2926 (w), 1736 (m), 1673 (w), 1590 (w), 1542 (vs), 1497 (w), 1455 (m), 1440 (m), 1371 (s), 1345 (s), 1203 (w), 1161 (vs), 1126 (s), 1094 (m), 1030 (w), 959 (m), 852 (m), 741 (s), 700 (m), 653 (w), 579 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.60 (s, 3H, 5'''-H/6'''-H), 1.68 (s, 3H, 6'''-H/5'''-H), 2.19 – 2.29 (m, 2H, 2'''-H), 2.88 (dd, *J* = 14.4 Hz, 8.4 Hz, 1H, 3-H_a), 3.26 – 3.35 (ddd, *J* = 14.4 Hz, 8.6 Hz, 7.1 Hz, 1H, 1'''-H_a), 3.35 – 3.45 (m, 2H, 1'''-H_b, 3-H_b), 4.73 (dd, *J* = 8.4 Hz, 6.4 Hz, 1H, 2-H), 4.98 – 5.07 (m, 1H, 3'''-H), 7.10 – 7.20 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 7.52 – 7.60 (m, 2H, 3"-H, 5"-H), 7.62 – 7.68 (m, 1H, 4"-H), 7.78 – 7.83 (m, 1H, 6"-H), 9.70 (s, 1H, 1-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.9 (C-5'''/C-6'''), 25.7 (C-6'''/C-5'''), 29.3 (C-2'''), 33.1 (C-3), 46.0 (C-1'''), 67.1 (C-2), 119.1 (C-3'''), 124.4 (C-3"), 127.0 (C-4'), 128.6 (C-3', C-5'), 129.0 (C-2', C-6'), 131.0 (C-6"), 131.8 (C-5"), 133.3 (C-1"), 133.6 (C-4'''), 135.6 (C-4"), 136.2 (C-1'), 148.6 (C-2"), 199.4 (C-1). HRMS (ESI): calc (found) for $C_{21}H_{24}N_2O_5S$ ($[M+H]^+$): 417.1479 (417.1481).

GP6: Cyclisation of amino aldehydes

Method A: Using Iron(III)chloride

The reaction was performed according to the literature.[11] A round bottom flask was charged with a solution of the amino aldehyde (1.50 mmol) in dichloromethane (50 mL) under nitrogen atmosphere. Upon cooling to 0 °C a solution of iron(III)chloride (3.0 mL, 3.00 mmol, 1 M in diethyl ether) was added dropwise. The mixture was stirred for 24 h at room temperature. Afterwards sodium hydroxide solution (50 mL, 2 M in water) was added and stirring continued for 5 min. The phases were separated and the aqueous phase was extracted with dichloromethane (3 × 75 mL). The organic phases were dried over magnesium sulfate and the solvents were evaporated. The crude product was purified by column chromatography or HPLC.

Method B: Using SmI_2

The reaction was performed according to the literature.[12] A round bottom flask was charged with a solution of the amino aldehyde (1.92 mmol) in dichloromethane (25 mL) under nitrogen atmosphere. A solution of samarium diiodide (21 mL, 2.10 mmol, 0.1 M in tetrahydrofuran) was added dropwise at room temperature and stirred for 24 h at room temperature. The solvents were evaporated and the crude product was purified by column chromatography.

Method C: Using $MeAlCl_2$

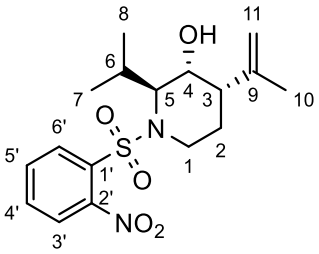
The reaction was performed according to the literature.[13] A round bottom flask was charged with a solution of the amino aldehyde (1.96 mmol) in chloroform (75 mL) under nitrogen atmosphere. A solution of methyl aluminium dichloride (2.0 mL, 2.00 mmol, 1.0 M in hexane) was added dropwise and the reaction was stirred for 24 h at reflux temperature. After cooling to room temperature the reaction was quenched with water (25 mL) and extracted with dichloromethane (2 × 40 mL). The organic phases were washed with brine (20 mL), dried over magnesium sulfate and the solvents were evaporated. The crude product was purified by column chromatography.

(2*S*,3*R*,4*S*)-2-Isopropyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-ol ((*R*,*S*)-**30a) and (2*S*,3*R*,4*R*)-4-(2-Hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)-sulfonyl)-piperidin-3-ol (**(*R*,*R*)-**31a)**

According to GP6A (*R*,*S*)-**30a** was prepared from (*S*)-*N*-(3-methyl-1-oxobutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide **29a** (0.72 g, 1.95 mmol) and iron(III)-chloride solution (3.9 mL, 3.90 mmol, 1 M in Et_2O) as a colorless solid (0.47 g, 1.28 mmol, 66 %). Diol (*R*,*R*)-**31a** was isolated as well as a colorless solid (0.18 g, 0.46 mmol, 24 %).

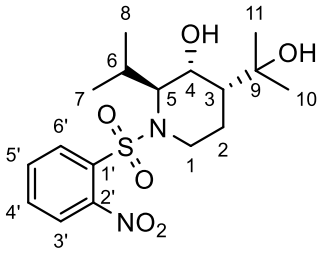
Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1 → 1 : 1 → 1 : 2).

Alkene (*R*,*S*)-**30a** was also prepared according to GP6B from (*S*)-*N*-(3-methyl-1-oxobutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide **29a** (0.71 g, 1.92 mmol) and samarium diiodide solution (21.0 mL, 2.10 mmol, 1 M in THF) as a colorless solid (0.32 g, 0.87 mmol, 45 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1).



(*R*,*S*)-**30a**

(*R*,*S*)-**30a**, mp 138 °C. $[\alpha]_D^{20}$ + 112 (*c* 1.0 in $CHCl_3$). R_f = 0.75 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3547 (w), 3088 (w), 2966 (m), 2932 (w), 2877 (w), 1736 (w), 1646 (w), 1590 (w), 1542 (vs), 1470 (w), 1440 (w), 1373 (s), 1339 (s), 1280 (w), 1207 (w), 1158 (s), 1126 (m), 1096 (w), 1065 (m), 1046 (w), 976 (m), 914 (m), 853 (w), 824 (w), 777 (w), 752 (s), 735 (m), 670 (w), 651 (w), 623 (w), 580 (s), 562 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.86 (d, *J* = 6.7 Hz, 3H, 7-H/8-H), 1.03 (d, *J* = 6.7 Hz, 3H, 8-H/7-H), 1.38 – 1.45 (m, 1H, 2-H_a), 1.77 (s, 3H, 10-H), 1.84 (dddd, *J* = 13.1 Hz, 13.1 Hz, 13.1 Hz, 4.7 Hz, 1H, 2-H_b), 1.98 (s, br, 1H, OH), 2.00 – 2.11 (m, 1H, 6-H), 2.26 (d, *J* = 12.7 Hz, 1H, 3-H), 3.10 (dt, *J* = 13.9 Hz, 2.6 Hz, 1H, 1-H_a), 3.69 (d, *J* = 10.7 Hz, 1H, 5-H), 4.02 – 4.12 (m, 2H, 1-H_b, 4-H), 4.68 (s, 1H, 11-H_a), 4.90 – 4.97 (m, 1H, 11-H_b), 7.50 – 7.56 (m, 1H, 3'-H), 7.58 – 7.67 (m, 2H, 4'-H, 5'-H), 8.10 – 8.17 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 19.9 (C-7/C-8), 20.4 (C-8/C-7), 22.0 (C-10), 22.6 (C-2), 26.5 (C-6), 41.4 (C-1), 41.6 (C-3), 65.2 (C-4), 66.0 (C-5), 112.2 (C-11), 123.5 (C-3'), 131.2 (C-6'), 131.3 (C-5'), 133.1 (C-4'), 134.8 (C-1'), 145.1 (C-9), 148.0 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{24}N_2O_5S$ ($[M+Na]^+$): 391.1298 (391.1306); calc (found) for $C_{17}H_{24}N_2O_5S$ ($[M+H]^+$): 369.1479 (369.1476).



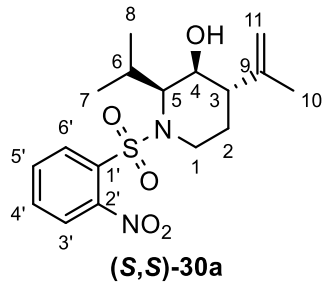
(*R*,*R*)-**31a**

(*R*,*R*)-**31a**, mp 185 °C. $[\alpha]_D^{20}$ + 148 (*c* 1.0 in $CHCl_3$). R_f = 0.44 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3362 (w, br), 2969 (m), 1543 (vs), 1470 (m), 1440 (w), 1371 (s), 1340 (s), 1278 (m), 1216 (w), 1157 (vs), 1125 (s), 1092 (m), 1064 (m), 1048 (m), 988 (w), 970 (s), 911 (m), 853 (m), 806 (w), 751 (s), 733 (s), 686 (w), 653 (m), 637 (w), 580 (s), 562 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.77 (d, *J* = 6.7 Hz, 3H, 1-H), 0.98 (d, *J* = 6.7 Hz, 3H, 2-H), 1.17 (s, 3H, 10-H/11-H), 1.32 (s, 3H, 11-H/10-H), 1.43 – 1.49 (m, 1H, 3-H), 1.53 – 1.60 (m, 1H, 2-H_a), 1.84 – 2.00 (m, 2H, 2-H_b, 6-H), 3.06 (s, br, 2H, 2 × OH), 3.11 (dt, *J* = 3.0 Hz,

14.1 Hz, 1H, 1-H_a), 3.52 (d, *J* = 10.7 Hz, 1H, 5-H), 4.09 – 4.17 (m, 1H, 1-H_b), 4.36 – 4.43 (m, 1H, 4-H), 7.53 – 7.58 (m, 1H, 3'-H), 7.63 – 7.69 (m, 2H, 4'-H, 5'-H), 8.07 – 8.13 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 19.4 (C-2), 19.8 (C-7/C-8), 20.3 (C-8/C-7), 26.4 (C-6), 28.0 (C-10/C-11), 28.6 (C-11/C-10), 41.8 (C-1), 42.0 (C-3), 66.4 (C-4), 66.8 (C-5), 72.1 (C-9), 123.7 (C-3'), 130.9 (C-6'), 131.6 (C-5'), 133.2 (C-4'), 134.7 (C-1'), 147.7 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_6S$ ($[M+H]^+$): 387.1584 (387.1582).

(2*S*,3*S*,4*S*)-2-Isopropyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-ol ((*S*,*S*)-**30a)**

According to GP6C (*S*,*S*)-**30a** was prepared from (*S*)-*N*-(3-methyl-1-oxobutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide **29a** (0.72 g, 1.96 mmol) and methyl aluminium dichloride solution (2.0 mL, 1.96 mmol, 1 M in hexane) as a colorless solid (0.16 g, 0.44 mmol, 23 %). Alkene (*R*,*S*)-**30a** was isolated as well as colorless solid (0.09 g, 0.23 mmol, 12 %). Purification was accomplished by HPLC (OD, *n*-heptane/isopropanol (95 : 5), 15 mL / min, R_t ((*S*,*S*)-**30a**) = 32.5 min, R_t ((*R*,*S*)-**30a**) = 40 min).

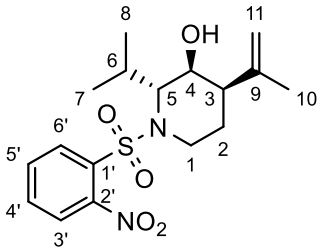


(*S*,*S*)-30a

(*S*,*S*)-**30a**, mp 43 °C. $[\alpha]_D^{20}$ + 115 (*c* 1.0 in $CHCl_3$). R_f = 0.77 (hexanes/EtOAc, 1 : 1, anisaldehyde solution), FT-IR (ATR): $\tilde{\nu}$ = 3544 (w), 3077 (w), 2962 (w), 1646 (w), 1591 (w), 1541 (vs), 1471 (w), 1440 (m), 1369 (s), 1333 (s), 1295 (m), 1256 (m), 1200 (w), 1157 (vs), 1125 (s), 1082 (s), 1001 (s), 966 (m), 901 (s), 852 (m), 799 (m), 778 (m), 763 (s), 729 (s), 653 (m), 598 (vs), 573 (s) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.64 (d, *J* = 6.7 Hz, 3H, 7-H/8-H), 1.13 (d, *J* = 6.7 Hz, 3H, 8-H/7-H), 1.58 (dddd, *J* = 13.0 Hz, 13.0 Hz, 13.0 Hz, 5.0 Hz, 1H, 2-H_a), 1.63 (s, 3H, 10-H), 1.63 – 1.73 (m, 1H, 2-H_b), 1.86 (s, 1H, OH), 2.15 (dqq, *J* = 9.5 Hz, 6.7 Hz, 6.7 Hz, 1H, 6-H), 2.45 (ddd, *J* = 13.0 Hz, 10.8 Hz, 3.9 Hz, 1H, 3-H), 2.94 – 3.05 (m, 1H, 1-H_a), 3.80 (dd, *J* = 9.5 Hz, 5.2 Hz, 1H, 5-H), 3.85 (dd, *J* = 10.8 Hz, 5.2 Hz, 1H, 4-H), 3.90 – 4.00 (m, 1H, 1-H_b), 4.87 (s, 1H, 11-H_a), 4.94 (s, 1H, 11-H_b), 7.59 – 7.65 (m, 1H, 3'-H), 7.65 – 7.72 (m, 2H, 4'-H, 5'-H), 8.04 – 8.10 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 18.8 (C-10), 20.4 (C-7/C-8), 22.3 (C-8/C-7), 25.7 (C-6), 30.3 (C-2), 41.4 (C-1), 46.4 (C-3), 62.7 (C-5), 71.7 (C-4), 114.5 (C-11), 124.2 (C-3'), 130.8 (C-6'), 131.7 (C-5'), 133.3 (C-4'), 134.6 (C-1'), 144.6 (C-9), 147.5 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{24}N_2O_5S$ ($[M+H]^+$): 369.1479 (369.1481).

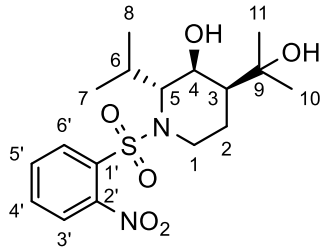
(2*R*,3*S*,4*R*)-2-Isopropyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-ol ((*S*,*R*)-**30b) and (2*R*,3*S*,4*S*)-4-(2-Hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)-sulfonyl)-piperidin-3-ol (**(*S*,*S*)-**31b)**

According to GP6A (*S*,*R*)-**30b** was prepared from (*R*)-*N*-(3-methyl-1-oxobutan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide **29b** (0.41 g, 1.11 mmol) and iron(III)-chloride solution (2.2 mL, 2.22 mmol, 1 M in diethyl ether) as a colorless solid (0.28 g, 0.76 mmol, 68 %). Diol (*S*,*S*)-**31b** was isolated as well as a colorless solid (0.05 g, 0.13 mmol, 12 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1 → 2 : 1 → 1 : 1).



(*S*,*R*)-**30b**

(*S*,*R*)-**30b**, mp 146 °C. $[\alpha]_D^{20}$ – 110 (*c* 1.0 in $CHCl_3$). R_f = 0.46 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3549 (w), 3091 (w), 2966 (m), 2931 (w), 2876 (w), 1646 (w), 1590 (w), 1542 (vs), 1470 (m), 1439 (m), 1372 (s), 1338 (s), 1294 (m), 1280 (m), 1207 (w), 1157 (vs), 1125 (s), 1095 (m), 1065 (m), 1045 (m), 975 (s), 913 (s), 852 (m), 776 (m), 752 (s), 733 (s), 670 (m), 650 (w), 623 (w), 579 (vs), 562 (s) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.86 (d, *J* = 6.5 Hz, 3H, 7-H/8-H), 1.03 (d, *J* = 6.5 Hz, 3H, 8-H/7-H), 1.41 (d, *J* = 13.1 Hz, 1H, 2-H_a), 1.77 (s, 3H, 10-H), 1.83 (ddt, *J* = 13.1 Hz, 13.1 Hz, 4.7 Hz, 1H, 2-H_b), 1.99 (s, br, 1H, OH), 2.05 (dqq, *J* = 10.8 Hz, 6.5 Hz, 6.5 Hz, 1H, 6-H), 2.26 (d, *J* = 12.5 Hz, 1H, 3-H), 3.10 (t, *J* = 13.7 Hz, 1H, 1-H_a), 3.69 (d, *J* = 10.8 Hz, 1H, 5-H), 4.02 – 4.12 (m, 2H, 1-H_b, 4-H), 4.68 (s, 1H, 11-H_a), 4.93 (s, 1H, 11-H_b), 7.50 – 7.56 (m, 1H, 3'-H), 7.58 – 7.67 (m, 2H, 4'-H, 5'-H), 8.10 – 8.17 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 19.9 (C-7/C-8), 20.4 (C-8/C-7), 21.9 (C-10), 22.6 (C-2), 26.5 (C-6), 41.4 (C-1), 41.6 (C-3), 65.2 (C-4), 66.0 (C-5), 112.2 (C-11), 123.5 (C-3'), 131.2 (C-6'), 131.3 (C-5'), 133.1 (C-4'), 134.7 (C-1'), 145.1 (C-9), 147.9 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{24}N_2O_5S$ ($[M+Na]^+$): 391.1298 (391.1299).



(*S*,*S*)-**31b**

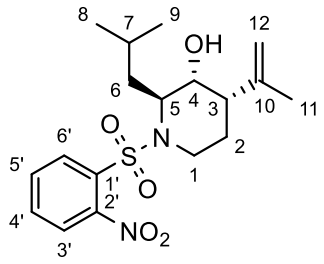
(*S*,*S*)-**31b**, mp 180 °C. $[\alpha]_D^{20}$ - 135 (*c* 1.0 in $CHCl_3$). R_f = 0.22 (hexanes/EtOAc, 1 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3365 (w, br), 2970 (m), 2939 (m), 2877 (w), 1732 (w), 1591 (w), 1543 (vs), 1470 (m), 1440 (w), 1372 (s), 1341 (s), 1278 (m), 1216 (w), 1158 (s), 1126 (m), 1092 (w), 1065 (m), 1048 (w), 970 (m), 911 (m), 853 (w), 806 (w), 776 (w), 751 (m), 734 (s), 685 (w), 653 (w), 637 (w), 581 (s), 562 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.77 (d, *J* = 6.8 Hz, 3H, 1-H), 0.98 (d, *J* = 6.8 Hz, 3H, 2-H), 1.17 (s, 3H, 10-H/11-H), 1.32 (s, 3H, 11-H/10-H), 1.46

(ddd, *J* = 13.3 Hz, 3.7 Hz, 2.1 Hz, 1H, 3-H), 1.58 – 1.62 (m, 1H, 2-H_a), 1.89 (ddt, *J* = 13.3 Hz, 13.3 Hz, 5.0 Hz, 1H, 2-H_b), 1.95 (dqq, *J* = 10.8 Hz, 6.8 Hz, 6.8 Hz, 1H, 6-H), 3.01 (s, br, 2H, 2 × OH), 3.11 (ddd, *J* = 13.3 Hz, 13.3 Hz, 3.0 Hz, 1H, 1-H_a), 3.52 (d, *J* = 10.8 Hz, 1H, 5-H), 4.09 – 4.16 (m, 1H, 1-H_b), 4.36 – 4.42 (m, 1H, 4-H), 7.53 – 7.58 (m, 1H, 3'-H), 7.63 – 7.69 (m, 2H, 4'-H/5'-H), 8.07 – 8.13 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 19.4 (C-2), 19.8 (C-7/C-8), 20.3 (C-8/C-7), 26.4 (C-6), 28.0 (C-10/C-11), 28.6 (C-11/C-10), 41.7 (C-1), 42.0 (C-3), 66.4 (C-4), 66.8 (C-5), 72.1 (C-9), 123.7 (C-3'), 130.9 (C-6'), 131.6 (C-5'), 133.2 (C-4'), 134.7 (C-1'), 147.7 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_6S$ ($[M+Na]^+$): 409.1404 (409.1407).

(2*S*,3*R*,4*S*)-2-Isobutyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-ol ((*R*,*S*)-30c), (2*S*,3*S*,4*S*)-2-Isobutyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-ol ((*S*,*S*)-30c), (2*S*,3*S*,4*R*)-4-(2-Hydroxypropan-2-yl)-2-isobutyl-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol ((*S*,*R*)-31c) and (2*S*,3*R*,4*R*)-4-(2-Hydroxypropan-2-yl)-2-isobutyl-1-((2-nitrophenyl)-sulfonyl)-piperidin-3-ol ((*R*,*R*)-31c)

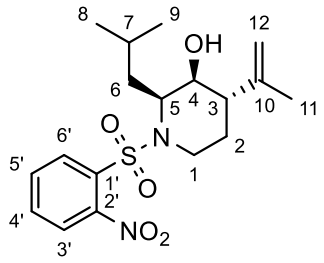
According to GP6A (*R*,*S*)-**30c** was prepared from (*S*)-*N*-(4-methyl-1-oxopentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide **29c** (1.08 g, 2.82 mmol) and iron(III)-chloride solution (5.6 mL, 5.64 mmol, 1 M in diethyl ether) as a colorless solid (63.0 mg, 0.17 mmol, 52 %). Alkene (*S*,*S*)-**30c** was isolated as well as a colorless solid (4.00 mg, 0.01 mmol, 3 %). Purification was accomplished by HPLC (Orbit, hexane/isopropanol (97 : 3), 12 mL / min, R_t ((*R*,*S*)-**30c**) = 19 min, R_t ((*S*,*S*)-**30c**) = 21 min). In addition diols (*S*,*R*)-**31c** and (*R*,*R*)-**31c** were isolated as colorless solids ((*S*,*R*)-**31c** 3.00 mg, 7.49 µmol, 2 %; (*R*,*R*)-**31c** 12.0 mg, 0.03 mmol, 9 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (90 : 10), 15 mL / min, R_t ((*S*,*R*)-**31c**) = 19 min, R_t ((*R*,*R*)-**31c**) = 21 min). Mixtures of alkenes and diols were separated beforehand via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1 → 2 : 1 → 1 : 1).

Alkene (*R*,*S*)-**30c** was also prepared according to GP6B from (*S*)-*N*-(4-methyl-1-oxopentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide **29c** (0.63 g, 1.65 mmol) and samarium diiodide solution (18.2 mL, 1.82 mmol, 0.1 M in THF) as a colorless solid (0.33 g, 0.87 mmol, 53 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1).



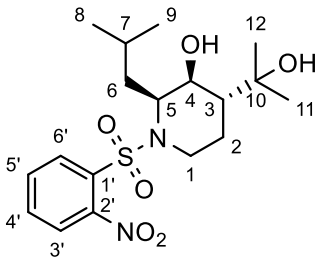
(*R*,*S*)-30c

(*R*,*S*)-**30c**, mp 141 °C. $[\alpha]_D^{20}$ + 71 (*c* 0.5 in $CHCl_3$). R_f = 0.49 (hexanes/ EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3542 (w), 3089 (w), 2956 (m), 2923 (m), 2853 (m), 1725 (w), 1646 (w), 1608 (w), 1591 (w), 1541 (vs), 1467 (m), 1439 (m), 1371 (s), 1340 (s), 1272 (m), 1206 (w), 1160 (vs), 1130 (s), 1067 (m), 1049 (w), 991 (m), 940 (m), 928 (m), 852 (m), 829 (w), 777 (m), 747 (m), 732 (m), 671 (m), 615 (m), 578 (s), 451 (w), 418 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.92 (d, *J* = 6.6 Hz, 3H, 8-H/9-H), 0.93 (d, *J* = 6.6 Hz, 3H, 9-H/8-H), 1.38 – 1.47 (m, 2H, 2-H_a, 6-H_a), 1.57 (ddd, *J* = 13.8 Hz, 7.0 Hz, 7.0 Hz, 1H, 6-H_b), 1.64 (ddqq, *J* = 7.0 Hz, 7.0 Hz, 6.6 Hz, 6.6 Hz, 1H, 7-H), 1.77 (s, 3H, 11-H), 1.88 (ddt, *J* = 13.4 Hz, 12.9 Hz, 4.5 Hz, 1H, 2-H_b), 2.28 (d, *J* = 12.9 Hz, 1H, 3-H), 3.20 (ddd, *J* = 13.6 Hz, 13.4 Hz, 2.5 Hz, 1H, 1-H_a), 3.76 – 3.79 (m, 1H, 4-H), 3.92 – 4.00 (m, 1H, 1-H_b), 4.11 – 4.16 (m, 1H, 5-H), 4.71 (s, 1H, 12-H_a), 4.95 (s, 1H, 12-H_b), 7.56 – 7.61 (m, 1H, 3'-H), 7.63 – 7.68 (m, 2H, 4'-H, 5'-H), 8.13 – 8.18 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 22.0 (C-11), 22.7 (C-8, C-9), 22.7 (C-2), 24.9 (C-7), 38.1 (C-6), 41.0 (C-1), 41.2 (C-3), 57.8 (C-5), 67.3 (C-4), 112.4 (C-12), 123.9 (C-3'), 131.3 (C-6'), 131.4 (C-4'/C-5'), 133.2 (C-5'/C-4'), 134.3 (C-1'), 145.0 (C-10), 147.9 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{26}N_2O_5S$ ($[M+Na]^+$): 405.1455 (405.1459).



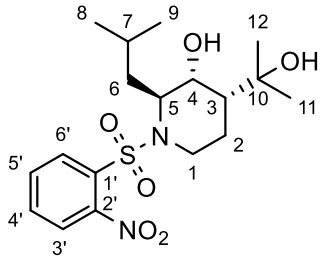
(*S*,*S*)-30c

(*S*,*S*)-**30c**, mp 37 °C. $[\alpha]_D^{20}$ + 50 (*c* 0.2 in $CHCl_3$). R_f = 0.49 (hexanes/ EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 35475 (w), 3077 (w), 2954 (m), 2925 (m), 2870 (w), 1646 (w), 1590 (w), 1543 (vs), 1467 (m), 1439 (m), 1369 (s), 1334 (s), 1294 (m), 1263 (m), 1160 (vs), 1127 (s), 1080 (s), 1060 (m), 1016 (m), 966 (m), 941 (m), 906 (s), 852 (m), 828 (w), 812 (w), 778 (m), 747 (s), 732 (s), 702 (w), 652 (m), 594 (vs), 567 (s), 447 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.72 (d, *J* = 6.4 Hz, 3H, 8-H/9-H), 0.85 (d, *J* = 6.4 Hz, 3H, 9-H/8-H), 1.40 – 1.59 (m, 4H, 2-H_a, 6-H, 7-H), 1.59 – 1.65 (m, 1H, 2-H_b), 1.61 (s, 3H, 11-H), 1.81 (d, *J* = 2.3 Hz, 1H, OH), 2.32 (dt, *J* = 11.7 Hz, 4.2 Hz, 1H, 3-H), 3.07 – 3.19 (m, 1H, 1-H_a), 3.71 (ddd, *J* = 10.6 Hz, 5.3 Hz, 2.3 Hz, 1H, 4-H), 3.84 – 3.92 (m, 1H, 1-H_b), 4.16 – 4.23 (m, 1H, 5-H), 4.85 (s, 1H, 12-H_a), 4.92 (s, 1H, 12-H_b), 7.62 – 7.67 (m, 1H, 3'-H), 7.67 – 7.73 (m, 2H, 4'-H, 5'-H), 8.09 – 8.15 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 18.7 (C-11), 21.4 (C-8/C-9), 23.9 (C-9/C-8), 24.2 (C-7), 29.7 (C-2), 32.8 (C-6), 40.0 (C-1), 45.9 (C-3), 55.6 (C-5), 69.1 (C-4), 114.3 (C-12), 124.3 (C-3'), 131.0 (C-6'), 131.7 (C-5'), 133.5 (C-4'), 134.3 (C-1'), 144.5 (C-10), 147.8 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{26}N_2O_5S$ ($[M+Na]^+$): 405.1455 (405.1456).



(*S*,*R*)-31c

(*S*,*R*)-**31c**, mp 195 °C. $[\alpha]_D^{20}$ + 63 (*c* 0.1 in $CHCl_3$). R_f = 0.11 (hexanes/ EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3323 (w, br), 2958 (m), 2930 (m), 2871 (m), 1545 (vs), 1468 (m), 1439 (m), 1370 (s), 1341 (s), 1279 (m), 1216 (w), 1157 (s), 1126 (s), 1078 (m), 1059 (m), 1015 (m), 964 (m), 943 (w), 900 (m), 853 (m), 778 (m), 748 (m), 732 (s), 652 (w), 595 (vs), 567 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.74 (d, *J* = 6.6 Hz, 3H, 8-H/9-H), 0.85 (d, *J* = 6.6 Hz, 3H, 9-H/8-H), 1.12 (s, 3H, 11-H/12-H), 1.10 – 1.20 (m, 1H, 2-H_a), 1.20 (s, 3H, 12-H/11-H), 1.40 – 1.50 (m, 1H, 7-H), 1.52 – 1.64 (m, 3H, 2-H_b, 6-H), 1.75 – 1.84 (m, 1H, 3-H), 2.60 (s, br, 1H, OH), 3.06 – 3.18 (m, 1H, 1-H_a), 3.83 – 3.89 (m, 1H, 1-H_b), 3.90 (dd, *J* = 11.0 Hz, 5.3 Hz, 1H, 4-H), 4.05 (ddd, *J* = 8.3 Hz, 5.3 Hz, 5.3 Hz, 1H, 5-H), 4.24 (s, br, 1H, OH), 7.60 – 7.66 (m, 1H, 3'-H), 7.66 – 7.72 (m, 2H, 4'-H, 5'-H), 8.07 – 8.15 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 21.4 (C-8/C-9), 23.1 (C-11/C-12), 23.9 (C-9/C-8), 24.3 (C-7), 27.3 (C-2), 30.0 (C-12/C-11), 33.0 (C-6), 40.1 (C-1), 45.0 (C-3), 56.1 (C-5), 71.8 (C-4), 74.9 (C-10), 124.3 (C-3'), 131.0 (C-6'), 131.7 (C-5'), 133.4 (C-4'), 134.4 (C-1'), 147.8 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{28}N_2O_6S$ ($[M+Na]^+$): 423.1560 (423.1563).



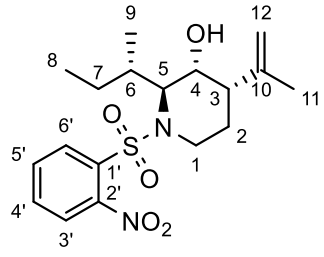
(*R*,*R*)-31c

(*R*,*R*)-**31c**, mp 54 °C. $[\alpha]_D^{20}$ + 210 (*c* 0.1 in $CHCl_3$). R_f = 0.11 (hexanes/ EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3373 (w, br), 2960 (m), 2925 (m), 2872 (m), 1544 (vs), 1469 (m), 1372 (s), 1341 (s), 1261 (m), 1159 (s), 1126 (s), 1094 (m), 1067 (m), 946 (m), 852 (m), 801 (m), 746 (m), 684 (w), 652 (m), 580 (s) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.86 (d, *J* = 6.6 Hz, 3H, 8-H/9-H), 0.89 (d, *J* = 6.6 Hz, 3H, 9-H/8-H), 1.18 (s, 3H, 11-H/12-H), 1.23 – 1.32 (m, 1H, 6-H_a), 1.30 (s, 3H, 12-H/11-H), 1.42 – 1.62 (m, 4H, 2-H_a, 3-H, 6-H_b, 7-H), 1.90 (dddd, *J* = 13.4 Hz, 13.4 Hz, 13.4 Hz, 4.9 Hz, 1H, 2-H_b), 2.79 (s, br, 1H, OH), 3.15 – 3.24 (m, 1H, 1-H_a), 3.23 (s, br, 1H, OH), 3.97 – 4.04 (m, 2H, 1-H_b, 5-H), 4.07 – 4.12 (m, 1H, 4-H), 7.55 – 7.61 (m, 1H, 3'-H), 7.63 – 7.70 (m, 2H, 4'-H, 5'-H), 8.10 – 8.17 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 19.4 (C-2), 22.5 (C-8/C-9), 22.8 (C-9/C-8), 24.8 (C-7), 28.1 (C-11/C-12), 28.6 (C-12/C-11), 37.9 (C-6), 41.1 (C-1), 41.8 (C-3), 58.7 (C-5), 68.8 (C-4), 72.3 (C-10), 123.9 (C-3'), 131.0 (C-6'), 131.7 (C-5'), 133.3 (C-4'), 134.3 (C-1'), 147.8 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{28}N_2O_6S$ ($[M+Na]^+$): 423.1560 (423.1563).

(2*S*,3*R*,4*S*)-2-((*S*)-*sec*-Butyl)-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol ((*R*,*S*)-30e) and (2*S*,3*R*,4*R*)-2-((*S*)-*sec*-Butyl)-4-(2-hydroxypropan-2-yl)-1-((2-nitrophenyl)-sulfonyl)piperidin-3-ol ((*R*,*R*)-31e)

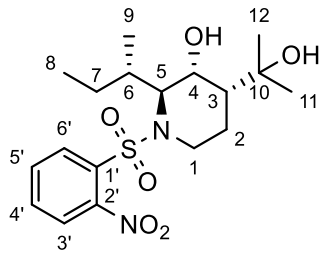
According to GP6A (*R*,*S*)-**30e** was prepared from *N*-((2*S*,3*S*)-3-methyl-1-oxopentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide **29e** (0.72 mg, 1.87 mmol) and iron(III)chloride solution (3.74 mL, 3.74 mmol, 1 M in diethyl ether) as a colorless solid (0.42 g, 1.09 mmol, 58 %). Diol (*R*,*R*)-**31e** was isolated as well as a colorless solid (79.0 mg, 0.20 mmol, 11 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1 → 1 : 1 → 1 : 2).

Alkene (*R*,*S*)-**30e** was also prepared according to GP6B from *N*-((2*S*,3*S*)-3-methyl-1-oxopentan-2-yl)-*N*-(4-methylpent-3-en-1-yl)-2-nitrobenzenesulfonamide **29e** (0.13 g, 0.34 mmol) and samarium diiodide solution (3.7 mL, 0.37 mmol, 0.1 M in THF) as a colorless solid (0.07 g, 0.19 mmol, 57 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1).



(*R*,*S*)-30e

(*R*,*S*)-**30e**, mp 124 °C. $[\alpha]_D^{20}$ + 24 (*c* 0.9 in $CHCl_3$). R_f = 0.37 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3551 (w, br), 3089 (w), 2965 (m), 2930 (m), 2877 (w), 1646 (w), 1590 (w), 1542 (vs), 1464 (m), 1440 (m), 1372 (s), 1338 (s), 1280 (m), 1206 (w), 1159 (s), 1126 (m), 1098 (w), 1065 (m), 1047 (w), 988 (m), 974 (m), 952 (m), 907 (m), 853 (m), 824 (w), 776 (m), 751 (s), 734 (m), 671 (m), 651 (w), 622 (w), 580 (s), 531 (w) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.80 (t, *J* = 7.1 Hz, 3H, 8-H), 0.84 – 0.93 (m, 1H, 7-H_a), 0.94 (d, *J* = 6.6 Hz, 3H, 9-H), 1.30 – 1.40 (m, 1H, 2-H_a), 1.47 – 1.60 (m, 1H, 7-H_b), 1.72 (s, 3H, 11-H), 1.72 – 1.84 (m, 2H, 2-H_b, 3-H), 1.93 (d, *J* = 2.8 Hz, 1H, OH), 2.18 – 2.30 (m, 1H, 6-H), 2.99 – 3.13 (m, 1H, 1-H_a), 3.70 – 3.78 (m, 1H, 4-H), 3.95 – 4.07 (m, 2H, 1-H_b, 5-H), 4.62 (s, 1H, 12-H_a), 4.87 (s, 1H, 12-H_b), 7.43 – 7.52 (m, 1H, 3'-H), 7.55 – 7.65 (m, 2H, 4'-H, 5'-H), 8.05 – 8.15 (m, 1H, 6'-H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 11.3 (C-8), 16.2 (C-9), 21.9 (C-11), 22.4 (C-2), 25.2 (C-7), 32.7 (C-3), 41.2 (C-6), 41.6 (C-1), 65.0 (C-4), 65.3 (C-5), 112.1 (C-12), 123.4 (C-3'), 131.2 (C-5'), 131.3 (C-6'), 133.2 (C-4'), 134.4 (C-1'), 145.1 (C-10), 148.0 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{26}N_2O_5S$ ($[M+Na]^+$): 405.1455 (405.1463).



(*R*,*R*)-**31e**

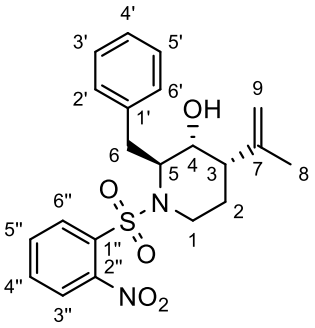
(*R*,*R*)-**31e**, mp 166 °C. $[\alpha]_D^{20}$ + 13 (*c* 1.0 in $CHCl_3$). R_f = 0.08 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3362 (w, br), 2966 (m), 2930 (m), 2877 (m), 1732 (w), 1591 (w), 1543 (vs), 1464 (m), 1440 (m), 1372 (s), 1341 (s), 1277 (m), 1241 (w), 1213 (w), 1158 (s), 1126 (s), 1094 (w), 1065 (m), 958 (m), 908 (m), 852 (m), 806 (w), 776 (m), 751 (m), 734 (m), 686 (w), 653 (w), 637 (w), 581 (s) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 0.76 – 0.80 (m, 3H, 8-H), 0.80 – 0.85 (m, 1H, 7-H_a), 0.92 (d, *J* = 6.5 Hz, 3H, 9-H), 1.15 (s, 3H, 11-H/12-H), 1.30 (s, 3H, 12-H/11-H), 1.41 – 1.47 (m, 1H, 3-H), 1.47 – 1.55 (m, 2H, 2-H_a, 7-H_b), 1.60 – 1.70 (m, 1H, 6-H), 1.81 – 1.90 (dddd, *J* = 13.3 Hz, 13.3 Hz, 13.3 Hz, 5.0 Hz, 1H, 2-H_b), 2.93 (s, br, 2H, 2xOH), 3.04 – 3.12 (m, 1H, 1-H_a), 3.59 (d, *J* = 10.9 Hz, 1H, 5-H), 4.05 – 4.13 (m, 1H, 1-H_b), 4.38 – 4.41 (m, 1H, 4-H), 7.50 – 7.55 (m, 1H, 3'-H), 7.60 – 7.67 (m, 2H, 4'-H, 5'-H), 8.06 – 8.12 (m, 1H, 6'-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 11.2 (C-8), 16.1 (C-9), 19.2 (C-2), 25.1 (C-7), 28.0 (C-11/C-12), 28.6 (C-12/C-11), 32.5 (C-6), 41.8 (C-1),41.8 (C-3), 65.7 (C-5), 66.5 (C-4), 72.2 (C-10), 123.5 (C-3'), 130.9 (C-6'), 131.5 (C-5'), 133.2 (C-4'), 134.6 (C-1'), 147.8 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{28}N_2O_6S$ ($[M+Na]^+$): 423.1560 (423.1560).

(2*S*,3*R*,4*S*)-2-Benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-ol ((*R*,*S*)-**30f), (2*S*,3*S*,4*S*)-2-Benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-ol (**(*S*,*S*)-**30f) and (2*S*,3*R*,4*R*)-2-Benzyl-4-(2-hydroxypropan-2-yl)-1-((2-nitrophenyl)-sulfonyl)-piperidin-3-ol (**(*R*,*R*)-**31f)**

According to GP6A (*R*,*S*)-**30f** was prepared from (*S*)-*N*-(4-methylpent-3-en-1-yl)-2-nitro-*N*-(1-oxo-3-phenylpropan-2-yl)benzenesulfonamide **29f** (0.44 g, 1.06 mmol) and iron(III)chloride solution (2.1 mL, 2.12 mmol, 1 M in diethyl ether) as a colorless solid (0.10 g, 0.24 mmol, 23 %). Alkene (*S*,*S*)-**30f** and diol (*R*,*R*)-**31f** were isolated as colorless solids as well ((*S*,*S*)-**30f** 0.12 g, 0.30 mmol, 28 % and (*R*,*R*)-**31f** 6.00 mg, 0.01 mmol, 3 %). Diol (*R*,*R*)-**31f** was isolated via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1 → 1 : 1 → 1 : 2). Purification of the mixture of alkenes was accomplished by HPLC (OD, *n*-hexane/isopropanol (90 : 10), 15 mL / min, R_t ((*R*,*S*)-**30f**) = 44 min, R_t ((*S*,*S*)-**30f**) = 51 min).

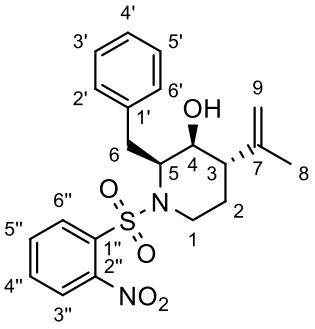
Alkene (*R*,*S*)-**30f** was also prepared according to GP6B from (*S*)-*N*-(4-methylpent-3-en-1-yl)-2-nitro-*N*-(1-oxo-3-phenylpropan-2-yl)benzenesulfonamide **29f** (0.32 g, 0.78 mmol) and samarium diiodide solution (8.6 mL, 0.86 mmol, 0.1 M in THF) as a colorless solid (0.21 g, 0.51 mmol, 65 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (3 : 1).

Alkenes (*R*,*S*)-**30f** and (*S*,*S*)-**30f** were also prepared according to GP6C from (*S*)-*N*-(4-methylpent-3-en-1-yl)-2-nitro-*N*-(1-oxo-3-phenylpropan-2-yl)benzenesulfonamide **29f** (0.17 g, 0.40 mmol) and methyl aluminium dichloride solution (0.4 mL, 0.40 mmol, 1 M in hexane) as a brown oil (crude yield: 0.18 g, 0.42 mmol, quant, ratio: 21 % (*R*,*S*)-**30f**, 79 % (*S*,*S*)-**30f**). The mixture wasn´t purified. The ratio was taken from ^{1}H-NMR spectrum.



(*R*,*S*)-**30f**

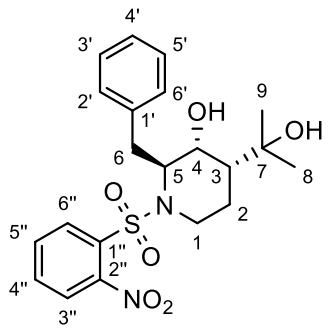
(*R*,*S*)-**30f**, mp 110 °C. $[\alpha]_D^{20}$ + 196 (*c* 1.0 in $CHCl_3$). R_f = 0.27 (hexanes/ EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3545 (w, br), 3087 (w), 3028 (w), 2946 (w), 2854 (w), 1646 (w), 1541 (vs), 1497 (w), 1440 (w), 1369 (s), 1331 (m), 1159 (s), 1126 (m), 1079 (m), 1016 (w), 954 (m), 906 (w), 853 (w), 777 (w), 756 (m), 741 (m), 700 (m), 652 (w), 593 (s), 576 (m), 505 (w) cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.70 – 1.82 (m, 2H, 2-H), 1.75 (s, 3H, 8-H), 2.05 (s, br, 1H, OH), 2.47 (ddd, *J* = 10.9 Hz, 10.9 Hz, 5.2 Hz, 1H, 3-H), 2.78 (dd, *J* = 14.1 Hz, 11.4 Hz, 1H, 6-H$_a$), 3.12 (dd, *J* = 14.1 Hz, 3.1 Hz, 1H, 6-H$_b$), 3.37 (ddd, *J* = 14.9 Hz, 11.9 Hz, 3.9 Hz, 1H, 1-H$_a$), 3.95 (dd, *J* = 10.9 Hz, 5.5 Hz, 1H, 4-H), 4.01 – 4.08 (m, 1H, 1-H$_b$), 4.39 (ddd, *J* = 11.4 Hz, 5.5 Hz, 3.1 Hz, 1H, 5-H), 4.96 (s, 1H, 9-H$_a$), 5.01 (s, 1H, 9-H$_b$), 6.83 – 6.92 (m, 3H, 3'-H, 4'-H, 5'-H), 6.96 – 7.04 (m, 2H, 2'-H, 6'-H), 7.24 – 7.30 (m, 1H, 5''-H), 7.37 – 7.42 (m, 1H, 6''-H), 7.43 – 7.50 (m, 2H, 3''-H, 4''-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 18.8 (C-8), 29.8 (C-6), 30.7 (C-2), 40.5 (C-1), 46.4 (C-3), 60.6 (C-5), 69.8 (C-4), 114.6 (C-9), 124.2 (C-3''), 126.3 (C-4'), 128.0 (C-3', C-5'), 129.2 (C-2', C-6'), 130.7 (C-6''), 131.9 (C-5''), 132.6 (C-4''), 134.1 (C-1''), 138.4 (C-1'), 144.4 (C-7), 147.0 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{24}N_2O_5S$ ([M+Na]$^+$): 439.1298 (439.1297).



(*S*,*S*)-**30f**

(*S*,*S*)-**30f**, mp 54 °C. $[\alpha]_D^{20}$ + 58 (*c* 0.38 in $CHCl_3$). R_f = 0.27 (hexanes/ EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3546 (w, br), 3087 (w), 3026 (w), 2923 (m), 2854 (w), 1646 (w), 1591 (w), 1540 (vs), 1495 (w), 1455 (m), 1440 (m), 1371 (s), 1336 (s), 1272 (m), 1159 (vs), 1126 (s), 1094 (w), 1072 (m), 989 (s), 974 (m), 949 (m), 930 (m), 908 (s), 852 (m), 823 (w), 778 (m), 758 (m), 730 (vs), 702 (s), 672 (w), 660 (m), 650 (m), 611 (m), 573 (s), 546 (m), 522 (m), 461 (w), 414 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.50 – 1.57 (m, 1H, 2-H$_a$), 1.73 (s, 3H, 8-H), 1.78 (s, br, 1H, OH), 2.00 (ddt, *J* = 13.2 Hz, 13.2 Hz, 4.8 Hz, 1H, 2-H$_b$), 2.42 – 2.50 (m, 1H, 3-H), 2.96 (dd, *J* = 13.6 Hz, 9.7 Hz, 1H, 6-H$_a$), 3.05 (dd, *J* = 13.6 Hz, 6.2 Hz, 1H, 6-H$_b$), 3.28 – 3.37 (m, 1H, 1-H$_a$), 3.72 (s, 1H, 4-H), 3.98 – 4.06 (m, 1H, 1-H$_b$), 4.27 – 4.34

(m, 1H, 5-H), 4.75 (s, 1H, 9-H_a), 4.92 – 4.99 (s, 1H, 9-H_b), 7.13 – 7.20 (m, 3H, 2'-H, 4'-H, 6'-H), 7.20 – 7.26 (m, 2H, 3'-H, 5'-H), 7.53 – 7.66 (m, 3H, 3''-H, 4''-H, 5''-H), 7.95 – 7.99 (m, 1H, 6''-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 22.0 (C-8), 22.9 (C-2), 35.9 (C-6), 41.2 (C-3), 41.4 (C-1), 61.0 (C-5), 65.3 (C-4), 112.5 (C-9), 124.1 (C-3''), 126.9 (C-4'), 128.7 ((C-2', C-6')/(C-3', C-5')), 129.0 ((C-3', C-5')/(C-2', C-6')), 131.1 (C-6''), 131.7 (C-5''), 133.1 (C-4''), 134.1 (C-1''), 137.3 (C-1'), 144.8 (C-7), 147.7 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{24}N_2O_5S$ ($[M+Na]^+$): 439.1298 (439.1297).



(*R*,*R*)-31f

(*R*,*R*)-**31f**, mp 73 °C. $[\alpha]_D^{20}$ + 118 (*c* 2.5 in $CHCl_3$). R_f = 0.51 (hexanes/ EtOAc, 1 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3346 (w, br), 3090 (w), 3027 (w), 2969 (w), 2929 (w), 1736 (w), 1592 (w), 1541 (vs), 1496 (w), 1455 (m), 1440 (m), 1369 (s), 1337 (s), 1272 (m), 1225 (w), 1158 (vs), 1126 (s), 1092 (m), 1072 (m), 1046 (m), 989 (m), 956 (m), 937 (m), 910 (s), 866 (w), 852 (m), 800 (w), 779 (m), 757 (m), 731 (vs), 701 (s), 684 (m), 652 (m), 621 (m), 576 (s), 537 (m) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 1.23 (s, 3H, 8-H/9-H), 1.27 (s, 3H, 9-H/8-H), 1.67 – 1.73 (m, 2H, 2-H_a, 3-H), 1.98 – 2.08 (m, 1H, 2-H_b), 2.75 (s, br, 1H, OH), 2.85 (dd, *J* = 13.6 Hz, 9.2 Hz, 1H, 6-H_a), 2.98 (dd, *J* = 13.6 Hz, 6.5 Hz, 1H, 6-H_b), 3.07 (d, *J* = 3.9 Hz, 1H, OH), 3.31 – 3.38 (m, 1H, 1-H_a), 4.04 – 4.10 (m, 2H, 1-H_b, 4-H), 4.16 – 4.20 (m, 1H, 5-H), 7.09 – 7.21 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 7.55 – 7.65 (m, 3H, 3''-H, 4''-H, 5''-H), 7.94 – 7.98 (m, 1H, 6''-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 19.8 (C-2), 28.2 (C-8/C-9), 28.6 (C-9/C-8), 35.7 (C-6), 41.5 (C-1), 41.9 (C-3), 62.2 (C-4), 66.9 (C-5), 72.3 (C-7), 124.3 (C-3''), 127.0 (C-4'), 128.7 (C-3', C-5'), 129.0 (C-2', C-6'), 131.1 (C-6''), 131.9 (C-5''), 133.3 (C-4''), 134.0 (C-1''), 137.1 (C-1'). The signal of quatary C-2'' wasn´t observed due to low intensity. HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_6S$ ($[M+Na]^+$): 457.1404 (457.1396).

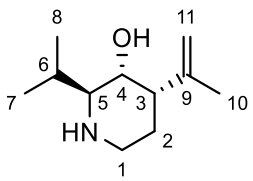
GP7: Deprotection of the amino group

Method A: The reaction was performed according to the literature.[14] A round bottom flask was charged with a solution of the *N*-protected derivative (1.00 mmol) in dimethylformamide (13.0 mL) under nitrogen atmosphere. Potassium carbonate (0.28 g, 2.00 mmol) and thiophenol (0.11 mL, 0.12 g, 1.10 mmol) were added and the mixture was stirred for 20 h at room temperature. Afterwards methyl-*tert*-butyl ether (15 mL) was added and the organic phase was extracted with hydrochloric acid (2 × 12 mL, 1 M in water). The aqueous phase was washed with MTBE (3 × 12 mL) and neutralized with sodium hydroxide solution (2 M). The solvents were evaporated and the crude product was taken up in dichloromethane (5 mL) and filtered

through a short cotton pad to remove remaining salts. The filtrate was evaporated and then purified by column chromatography. The product was once again taken up in dichloromethane (5 mL) and filtered over a short cotton pad to remove eluted silica. The filtrate was evaporated to yield the unprotected product.

Method B: The reaction was performed according to the literature.[15] A round bottom flask was charged with a solution of the *N*-protected derivative (0.13 mmol) in dimethylformamide (2.0 mL) under nitrogen atmosphere. Upon cooling to 0 °C diisopropylethylamine (88.0 µL, 67.0 mg, 0.52 mmol) and thiophenol (67.0 mL, 72.0 mg, 0.65 mmol) were added and the mixture was stirred for 7 d at room temperature till TLC showed a complete conversion of the starting material. Afterwards methyl-*tert*-butyl ether (9 mL) was added and the organic phase was extracted with hydrochloric acid (2 × 1 mL, 1 M in water). The aqueous phase was washed with MTBE (3 × 3 mL) and neutralized with sodium hydroxide solution (2 M). The solvents were evaporated and the crude product was taken up in dichloromethane (5 mL) and filtered through a short cotton pad to remove remaining salts. The filtrate was evaporated and then purified by column chromatography. The product was once again taken up in dichloromethane (5 mL) and filtered over a short cotton pad to remove eluted silica. The filtrate was evaporated to yield the unprotected product.

(2*S*,3*R*,4*S*)-2-Isopropyl-4-(prop-1-en-2-yl)piperidin-3-ol ((*R*,*S*)-32a)

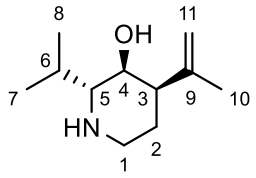


(*R*,*S*)-32a

According to GP7A (*R*,*S*)-**32a** was prepared from (2*S*,3*R*,4*S*)-2-isopropyl-1-((2-nitrophenyl)-sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*R*,*S*)-**30a** (0.13 g, 0.34 mmol), potassium carbonate (0.09 g, 0.68 mmol) and thiophenol (0.04 g, 0.37 mmol) as a colorless oil (0.05 g, 0.25 mmol, 74 %). Purification was accomplished via column chromatography on SiO_2 with dichloromethane/methanol (10 : 1 → 5 : 1). $[\alpha]_D^{20}$ + 45 (*c* 1.0 in $CHCl_3$). R_f = 0.75 (dichloromethane/methanol, 5 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3335 (m, br), 3086 (m), 2958 (vs), 2870 (s), 1645 (m), 1469 (s), 1384 (m), 1304 (m), 1259 (m), 1201 (m), 1147 (m), 1119 (m), 1089 (m), 1065 (m), 988 (s), 946 (w), 886 (s), 807 (w), 772 (m), 624 (w), 522 (w), 485 (w), 420 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.94 (d, *J* = 6.7 Hz, 3H, 7-H/8-H), 1.00 (d, *J* = 6.7 Hz, 3H, 8-H/7-H), 1.38 – 1.47 (m, 1H, 2-H_a), 1.81 (s, 3H, 10-H), 1.84 (dddd, *J* = 12.6 Hz, 12.6 Hz, 12.6 Hz, 5.6 Hz, 1H, 2-H_b), 2.00 – 2.14 (m, 1H, 6-H), 2.19 (d, *J* = 12.6 Hz, 1H, 3H), 2.36 (s, br, 2H, OH, NH), 2.48 (dd, *J* = 10.7 Hz, 2.5 Hz, 1H, 5-H), 2.71 – 2.84 (m, 2H, 1-H), 3.93 (s, 1H, 4-H), 4.78 (s, 1H, 11-H_a), 4.92 – 4.98 (m, 1H, 11-H_b). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 19.7 (C-7/C-8), 20.1 (C-8/C-7), 22.1 (C-10), 24.1 (C-2), 24.8

(C-6), 39.3 (C-1), 41.8 (C-3), 64.8 (C-5), 66.0 (C-4), 111.3 (C-11), 146.6 (C-9). HRMS (ESI): calc (found) for $C_{11}H_{21}NO$ ($[M+H]^+$): 184.1696 (184.1692).

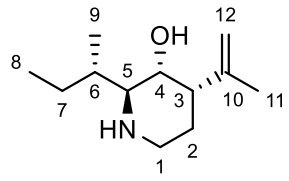
(2*R*,3*S*,4*R*)-2-Isopropyl-4-(prop-1-en-2-yl)piperidin-3-ol ((*S*,*R*)-32b)



(*S*,*R*)-32b

According to GP7A (*S*,*R*)-**32b** was prepared from (2*R*,3*S*,4*R*)-2-Isopropyl-1-((2-nitrophenyl)-sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*S*,*R*)-**30b** (52.0 mg, 141 µmol), potassium carbonate (39.0 mg, 282 µmol) and thiophenol (17.0 mg, 155 µmol) as a colorless oil (25.0 mg, 136 µmol, 96 %). Purification was accomplished via column chromatography on SiO_2 with dichloromethane/methanol (10 : 1 → 5 : 1). $[\alpha]_D^{20}$ - 14 (*c* 1.0 in $CHCl_3$). R_f = 0.16 (dichloromethane/methanol, 10 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3328 (br, s), 2966 (vs), 2875 (s), 2502 (w), 1733 (m), 1646 (m), 1586 (vs), 1440 (vs), 1396 (s), 1375 (s), 1307 (s), 1265 (m), 1221 (m), 1197 (m), 1168 (m), 1143 (m), 1082 (m), 1063 (m), 1043 (m), 1016 (m), 989 (vs), 946 (w), 887 (s), 848 (w), 834 (m), 718 (w), 625 (m), 579 (m), 529 (s) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.04 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 1.24 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.59 – 1.68 (m, 1H, 2-H_a), 1.78 (s, 3H, 10-H), 2.05 (dqq, *J* = 10.8 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 2.18 – 2.28 (m, 1H, 3-H), 2.36 (dddd, *J* = 13.2 Hz, 13.2 Hz, 13.2 Hz, 5.0 Hz, 1H, 2-H_b), 2.91 (ddd, *J* = 13.2 Hz, 13.2 Hz, 3.5 Hz, 1H, 1-H_a), 3.30 – 3.42 (m, 1H, 5-H), 3.45 – 3.59 (m, 1H, 1-H_b), 4.13 – 4.24 (m, 1H, 4-H), 4.79 (s, 1H, 11-H_a), 4.92 (s, 1H, 11-H_b). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 19.5 (C-7/C-8), 20.6 (C-8/C-7), 20.8 (C-2), 21.8 (C-10), 25.1 (C-6), 39.1 (C-1), 39.7 (C-3), 65.2 (C-5), 65.3 (C-4), 112.3 (C-11), 144.4 (C-9). HRMS (ESI): calc (found) for $C_{11}H_{21}NO$ ($[M+H]^+$): 184.1696 (184.1696).

(2*S*,3*R*,4*S*)-2-((*S*)-*sec*-Butyl)-4-(prop-1-en-2-yl)piperidin-3-ol ((*R*,*S*)-32e)

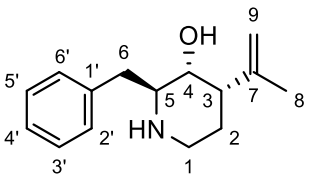


(*R*,*S*)-32e

According to GP7A (*R*,*S*)-**32e** was prepared from (2*S*,3*R*,4*S*)-2-((*S*)-*sec*-butyl)-1-((2-nitrophenyl)-sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*R*,*S*)-**30e** (65.0 g, 0.17 mmol), potassium carbonate (47.0 mg, 0.34 mmol) and thiophenol (21.0 mg, 0.19 mmol) as a colorless solid (21.0 mg, 0.11 mmol, 62 %). Purification was accomplished via column chromatography on SiO_2 with dichloromethane/methanol (10 : 1 → 5 : 1). mp 55 °C. $[\alpha]_D^{20}$ + 11 (*c* 0.46 in $CHCl_3$). R_f = 0.50 (dichloromethane/methanol, 5 : 1, ninhydrin solution). FT-IR (ATR): $\tilde{\nu}$ = 3296 (br, w), 2966 (s), 2935 (s), 2878 (m), 1732 (w), 1647 (w), 1580 (m), 1454 (m), 1377 (m), 1307 (m), 1261 (m), 1218 (w), 1193 (w), 1141 (w), 1084 (m), 1067 (m), 1044 (m), 1018 (m), 985 (s), 957 (w), 802 (m), 728 (vs), 643 (m), 624 (m), 552 (w), 519 (w), 491 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.93 (t, *J* = 7.2 Hz, 3H, 8-H),

1.00 (d, *J* = 6.7 Hz, 3H, 9-H), 1.31 – 1.43 (m, 1H, 7-H_a), 1.59 – 1.69 (m, 1H, 2-H_a), 1.80 (s, 3H, 11-H), 1.82 – 1.95 (m, 2H, 6-H, 7-H_b), 2.21 – 2.29 (m, 1H, 3-H), 2.35 (dddd, *J* = 13.4 Hz, 13.4 Hz, 13.4 Hz, 4.7 Hz, 1H, 2-H_b), 2.93 (ddd, *J* = 13.4 Hz, 13.4 Hz, 3.3 Hz, 1H, 1-H_a), 3.35 –3.43 (m, 1H, 5-H), 3.43 – 3.52 (m, 1H, 1-H_b), 4.13 – 4.23 (m, 1H, 4-H), 4.81 (s, 1H, 12-H_a), 4.95 (s, 1H, 12-H_b). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 10.2 (C-8), 15.1 (C-9), 20.9 (C-2), 21.9 (C-11), 25.4 (C-7), 30.8 (C-6), 39.2 (C-1), 39.9 (C-3), 63.0 (C-5), 65.5 (C-4), 112.4 (C-12), 144.4 (C-10). HRMS (ESI): calc (found) for $C_{12}H_{23}NO$ ($[M+H]^+$): 198.1852 (198.1850).

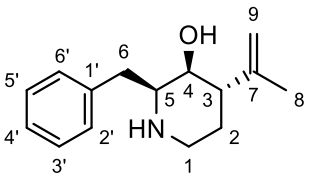
(2*S*,3*R*,4*S*)-2-Benzyl-4-(prop-1-en-2-yl)piperidin-3-ol ((*R*,*S*)-32f)



(***R*,*S*)-32f**

According to GP7A (*R*,*S*)-**32f** was prepared from (2*S*,3*R*,4*S*)-2-benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*R*,*S*)-**30f** (32.0 mg, 0.08 mmol), potassium carbonate (22.0 mg, 0.16 mmol) and thiophenol (8.0 µL, 9.00 mg, 0.08 mmol) as a colorless solid (19.0 mg, 0.08 mmol, quant). Purification was accomplished via column chromatography on SiO_2 with dichloromethane/methanol (10 : 1 → 5 : 1). mp 147 °C. $[\alpha]_D^{20}$ - 25 (*c* 0.2 in $CHCl_3$). R_f = 0.19 (dichloromethane /methanol, 10 : 1, ninhydrin solution). FT-IR (ATR): $\tilde{\nu}$ = 3311 (w, br), 2932 (m), 2854 (m), 2476 (w), 1647 (w), 1580 (m), 1496 (m), 1454 (s), 1376 (m), 1312 (m), 1246 (m), 1179 (m), 1099 (m), 1057 (m), 1019 (m), 993 (m), 959 (w), 908 (s), 832 (w), 729 (vs), 645 (m), 625 (m), 585 (m), 543 (m), 489 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.61 – 1.70 (m, 1H, 2-H_a), 1.71 (s, 3H, 8-H), 2.23 – 2.35 (m, 1H, 2-H_b), 2.42 – 2.53 (m, 1H, 3-H), 2.88 – 2.98 (m, 1H, 6-H_a), 3.09 – 3.20 (m, 1H, 1-H_a), 3.37 – 3.50 (m, 2H, 1-H_b, 6-H), 3.76 – 3.84 (m, 1H, 4-H), 3.95 – 4.05 (m, 1H, 5-H), 4.77 (s, 1H, 9-H_a), 4.95 (s, 1H, 9-H_b), 7.22 – 7.34 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 21.1 (C-2), 21.8 (C-8), 33.6 (C-6), 39.1 (C-1), 39.8 (C-3), 59.3 (C-5), 64.6 (C-4), 112.6 (C-9), 127.3 (C-4'), 129.0 (C-3', C-5'), 129.1 (C-2', C-6'), 135.8 (C-1'), 144.2 (C-7). HRMS (ESI): calc (found) for $C_{15}H_{21}NO$ ($[M+H]^+$): 232.1696 (232.1703).

(2*S*,3*S*,4*S*)-2-Benzyl-4-(prop-1-en-2-yl)piperidin-3-ol ((*S*,*S*)-32f)

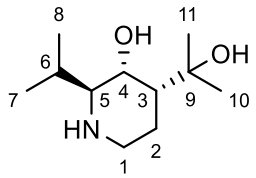


(***S*,*S*)-32f**

According to GP7A (*S*,*S*)-**32f** was prepared from (2*S*,3*S*,4*S*)-2-benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*S*,*S*)-**30f** (58.0 mg, 0.14 mmol), potassium carbonate (39.0 mg, 0.28 mmol) and thiophenol (16.0 µL, 17.0 mg, 0.15 mmol) as a colorless solid (27.0 mg, 0.12 mmol, 86 %). Purification was accomplished via column chromatography on SiO_2 with dichloromethane/methanol (10 : 1 → 5 : 1). mp 213 °C. $[\alpha]_D^{20}$ - 32 (*c* 1.0 in $CHCl_3$). R_f = 0.20 (dichloromethane/methanol, 10 : 1,

ninhydrin solution). FT-IR (ATR): $\tilde{\nu}$ = 3324 (br, s), 3083 (s), 3061 (s), 3027 (s), 2939 (vs), 2849 (s), 2815 (vs), 2713 (s), 2570 (m), 2553 (m), 2486 (m), 1725 (w), 1642 (m), 1604 (m), 1583 (m), 1495 (m), 1445 (vs), 1409 (s), 1381 (s), 1315 (m), 1266 (m), 1199 (m), 1114 (m), 1092 (m), 1075 (m), 1053 (s), 1032 (s), 1018 (s), 1004 (s), 980 (m), 954 (m), 896 (s), 781 (w), 746 (s), 702 (vs), 637 (m), 586 (m), 543 (m), 526 (m), 467 (m), 419 (w) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 1.51 (s, 3H, 8-H), 1.90 – 1.98 (m, 1H, 2-H_a), 2.41 –2.51 (m, 1H, 2-H_b), 2.54 – 2.60 (m, 1H, 3-H), 3.14 (ddd, *J* = 12.8 Hz, 12.8 Hz, 3.4 Hz, 1H, 1-H_a), 3.20 (dd, *J* = 13.0 Hz, 10.8 Hz, 1H, 6-H_a), 3.27 (dd, *J* = 13.0 Hz, 4.9 Hz, 1H, 6-H_b), 3.32 – 3.40 (m, 2H, 1-H_b, 5-H), 3.95 – 4.01 (m, 1H, 4-H), 4.59 (s, 1H, 9-H_a), 4.91 (s, 1H, 9-H_b), 7.20 – 7.25 (m, 1H, 4'-H), 7.27 – 7.31 (m, 2H, 3'-H, 5'-H), 7.31 – 7.35 (m, 2H, 2'-H, 6'-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 20.0 (C-2), 22.0 (C-8), 34.5 (C-6), 41.1 (C-1), 43.4 (C-3), 57.5 (C-5), 64.4 (C-4), 113.1 (C-9), 127.2 (C-4'), 128.9 (C-3', C-5'), 129.5 (C-2', C-6'), 135.5 (C-1'), 142.1 (C-7). HRMS (ESI): calc (found) for $C_{15}H_{21}NO$ ($[M+H]^+$): 232.1696 (232.1692).

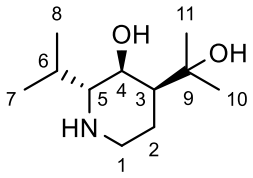
(2*S*,3*R*,4*R*)-4-(2-Hydroxypropan-2-yl)-2-isopropylpiperidin-3-ol ((*R*,*R*)-33a)



(*R*,*R*)-33a

According to GP7B (*R*,*R*)-**33a** was prepared from (2*S*,3*R*,4*R*)-4-(2-hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)-sulfonyl)piperidin-3-ol (*R*,*R*)-**31a** (89 mg, 0.23 mmol), diisopropylethylamine (59 mg, 0.46 mmol) and thiophenol (63 mg, 0.58 mmol) as a colorless solid (22 mg, 0.11 mmol, 48 %). Purification was accomplished via column chromatography on SiO_2 with dichloromethane/methanol (5 : 1). mp 71 °C. $[\alpha]_D^{20}$ + 6 (*c* 1.0 in $CHCl_3$). R_f = 0.26 (dichloromethane/methanol, 5 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3288 (br, s), 2970 (vs), 1738 (w), 1585 (s), 1465 (m), 1396 (m), 1377 (m), 1278 (w), 1171 (m), 1097 (w), 1063 (w), 989 (m), 912 (w), 849 (w), 808 (w), 730 (m), 644 (w), 572 (w), 525 (w), 426 (w), 415 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.01 (d, *J* = 6.4 Hz, 3H, 7-H/8-H), 1.21 (s, 3H, 10-H/11-H), 1.24 (d, *J* = 6.4 Hz, 3H, 8-H/7-H), 1.34 (s, 3H, 11-H/10-H), 1.42 – 1.50 (m, 1H, 3-H), 1.76 (d, *J* = 13.8 Hz, 1H, 2-H_a), 1.96 (dqq, *J* = 10.8 Hz, 6.4 Hz, 6.4 Hz, 1H, 6-H), 2.39 (dddd, *J* =13.5 Hz, 13.5 Hz, 13.5 Hz, 4.5 Hz, 1H, 2-H_b), 2.90 (ddd, *J* = 13.5 Hz, 13.3 Hz, 3.3 Hz, 1H, 1-H_a), 3.31 (d, *J* = 10.8 Hz, 1H, 5-H), 3.49 – 3.59 (m, 1H, 1-H_b), 4.43 (s, br, 1H, OH), 4.49 (s, 1H, 4-H), 5.73 (s, br, 1H, OH). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.6 (C-2), 19.5 (C-7/C-8), 20.6 (C-8/C-7), 24.8 (C-6), 27.9 (C-10/C-11), 28.4 (C-11/C-10), 38.9 (C-1), 40.5 (C-3), 65.0 (C-5), 65.5 (C-4), 72.2 (C-9). HRMS (ESI): calc (found) for $C_{11}H_{23}NO_2$ ($[M+H]^+$): 202.1802 (202.1801).

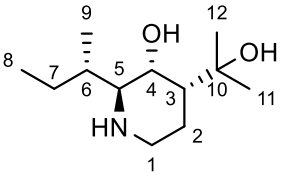
(2*R*,3*S*,4*S*)-4-(2-Hydroxypropan-2-yl)-2-isopropylpiperidin-3-ol ((*S*,*S*)-33b)



(*S*,*S*)-33b

According to GP7B (*S*,*S*)-**33b** was prepared from (2*R*,3*S*,4*S*)-4-(2-hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)-sulfonyl)-piperidin-3-ol (*S*,*S*)-**31b** (50.0 mg, 0.13 mmol), diisopropylethylamine (88.0 µL, 67.0 mg, 0.52 mmol) and thiophenol (67.0 µL, 72.0 mg, 0.65 mmol) as a colorless solid (15.0 mg, 0.07 mmol, 54 %). Purification was accomplished via column chromatography on SiO_2 with dichloromethane/methanol (10 : 1). mp 75 °C. $[\alpha]_D^{20}$ - 27 (*c* 0.33 in $CHCl_3$). R_f = 0.49 (dichloromethane/methanol, 5 : 1, ninhydrin solution). FT-IR (ATR): $\tilde{\nu}$ = 3321 (br, s), 2968 (vs), 1585 (s), 1464 (s), 1395 (s), 1377 (s), 1309 (w), 1263 (m), 1169 (s), 1097 (m), 1076 (m), 1064 (m), 1007 (m), 988 (s), 911 (s), 849 (w), 834 (w), 807 (m), 729 (vs), 643 (s), 577 (m), 524 (s), 455 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.03 (d, *J* = 6.4 Hz, 3H, 7-H/8-H), 1.23 (s, 3H, 10-H/11-H), 1.20 – 1.26 (d, *J* = 6.4 Hz, 3H, 8-H/7-H), 1.36 (s, 3H, 11-H/10-H), 1.43 – 1.51 (m, 1H, 3-H), 1.72 – 1.82 (m, 1H, 2-H_a), 1.97 (dqq, *J* = 11.0 Hz, 6.4 Hz, 6.4 Hz, 1H, 6-H), 2.34 – 2.50 (m, 1H, 2-H_b), 2.83 – 2.98 (m, 1H, 1-H_a), 3.38 (d, *J* = 11.0 Hz, 1H, 5-H), 3.54 – 3.64 (m, 1H, 1-H_b), 4.52 (s, 1H, 4-H), 8.34 (s, 1H, NH/OH), 8.80 (s, 1H, OH/NH). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 17.6 (C-2), 19.5 (C-7/C-8), 20.6 (C-8/C-7), 24.8 (C-6), 27.9 (C-10/C-11), 28.4 (C-11/C-10), 38.9 (C-1), 40.5 (C-3), 64.9 (C-5), 65.6 (C-4), 72.3 (C-9). HRMS (ESI): calc (found) for $C_{11}H_{23}NO_2$ ($[M+H]^+$): 202.1802 (202.1802).

(2*S*,3*R*,4*R*)-2-((*S*)-*sec*-Butyl)-4-(2-hydroxypropan-2-yl)piperidin-3-ol ((*R*,*R*)-33e)

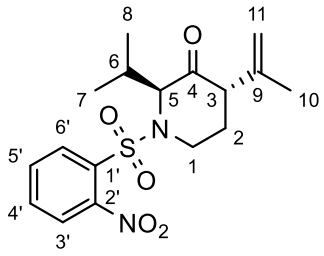


(*R*,*R*)-33e

According to GP7B (*R*,*R*)-**33e** was prepared from (2*S*,3*R*,4*R*)-2-((*S*)-*sec*-butyl)-4-(2-hydroxypropan-2-yl)-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol (*R*,*R*)-**31e** (47.0 mg, 117 µmol), diisopropylethylamine (80 µL, 60 mg, 468 mmol) and thiophenol (59 µL, 64 mg, 585 mmol) as a colorless solid (16.0 mg, 74.0 µmol, 63 %). Purification was accomplished via column chromatography on SiO_2 with dichloromethane/methanol (10 : 1 → 5 : 1). mp 173 °C. $[\alpha]_D^{20}$ - 12 (*c* 0.5 in $CHCl_3$). R_f = 0.35 (dichloromethane/methanol, 5 : 1, ninhydrin solution). FT-IR (ATR): $\tilde{\nu}$ = 3337 (s, br), 2969 (vs), 2880 (s), 1588 (s), 1462 (s), 1382 (vs), 1283 (m), 1197 (m), 1164 (s), 1098 (m), 1066 (m), 983 (s), 953 (w), 912 (m), 838 (w), 809 (m), 785 (w), 730 (s), 643 (m), 557 (m), 499 (m) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 0.94 (t, *J* = 7.4 Hz, 3H, 8-H), 0.97 (d, *J* = 6.7 Hz, 3H, 9-H), 1.22 (s, 3H, 11-H/12-H), 1.34 (s, 3H, 12-H/11-H), 1.34 – 1.40 (m, 1H, 7-H_a), 1.45 – 1.50 (m, 1H, 3-H), 1.72 – 1.82 (m, 2H, 2-H_a, 6-H), 1.82 – 1.91 (m, 1H, 7-H_b), 2.37 (dddd, *J* = 13.8 Hz, 13.8 Hz, 13.8 Hz, 4.7 Hz, 1H, 2-H_b),

2.85 – 2.97 (m, 1H, 1-H_a), 3.39 (d, *J* = 11.0 Hz, 1H, 5-H), 3.49 – 3.57 (m, 1H, 1-H_b), 4.45 – 4.55 (m, 1H, 4-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 10.2 (C-8), 15.0 (C-9), 17.6 (C-2), 25.3 (C-7), 27.9 (C-11/C-12), 28.3 (C-12/C-11), 30.3 (C-6), 38.9 (C-1), 40.4 (C-3), 63.0 (C-5), 65.5 (C-4), 72.2 (C-10). HRMS (ESI): calc (found) for $C_{12}H_{25}NO_2$ ($[M+H]^+$): 216.1958 (216.1968).

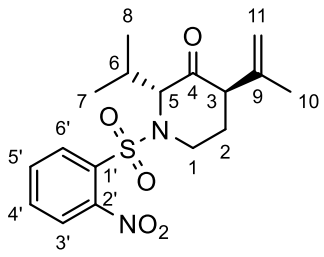
(2*S*,4*S*)-2-Isopropyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-one ((*S*)-S2a)



(*S*)-S2a

According to GP5 (*S*)-**S2a** was prepared from (2*S*,3*R*,4*S*)-2-isopropyl-4-(prop-1-en-2-yl)-piperidin-3-ol (*R*,*S*)-**30a** (0.11 g, 0.30 mmol) and DMP (0.32 g, 0.75 mmol) as a colorless solid (0.118 g, 0.32 mmol, quant) without further purification. mp 112 °C. $[\alpha]_D^{20}$ + 30 (*c* 1.0 in $CHCl_3$). R_f = 0.50 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3089 (w), 2967 (m), 2929 (w), 2874 (w), 1716 (s), 1650 (w), 1590 (w), 1543 (vs), 1467 (m), 1440 (m), 1369 (vs), 1302 (m), 1251 (w), 1195 (w), 1161 (vs), 1126 (m), 1091 (m), 1061 (m), 1040 (w), 999 (m), 981 (s), 905 (s), 852 (m), 778 (s), 761 (m), 739 (m), 729 (m), 675 (w), 655 (m), 585 (s), 535 (w), 466 (w) cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.89 (d, *J* = 6.7 Hz, 3H, 7-H/8-H), 0.95 (d, *J* = 6.7 Hz, 3H, 8-H/7-H), 1.54 (s, 3H, 10-H), 1.83 – 2.00 (m, 2H, 2-H), 2.22 (dqq, *J* = 10.8 Hz, 6.7 Hz, 6.7 Hz, 1H, 6-H), 3.17 (dd, *J* = 12.5 Hz, 6.4 Hz, 1H, 3-H), 3.52 (ddd, *J* = 15.3 Hz, 12.3 Hz, 3.4 Hz, 1H, 1-H_a), 3.88 (d, *J* = 10.8 Hz, 1H, 5-H), 4.11 – 4.19 (m, 1H, 1-H_b), 4.63 (s, 1H, 11-H_a), 4.90 (s, 1H, 11-H_b), 7.56 – 7.64 (m, 1H, 3'-H), 7.64 – 7.73 (m, 2H, 4'-H, 5'-H), 7.95 – 8.02 (m, 1H, 6'-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 18.6 (C-7/C-8), 19.4 (C-8/C-7), 20.2 (C-10), 28.0 (C-6), 31.4 (C-2), 40.7 (C-1), 53.6 (C-3), 71.8 (C-5), 114.4 (C-11), 124.0 (C-3'), 130.9 (C-6'), 131.9 (C-5'), 133.5 (C-1'), 133.8 (C-4'), 141.3 (C-9), 147.8 (C-2'), 204.4 (C-4). HRMS (ESI): calc (found) for $C_{17}H_{22}N_2O_5S$ ($[M+H]^+$): 367.1322 (367.1324).

(2*R*,4*R*)-2-Isopropyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-one ((*R*)-S2b)

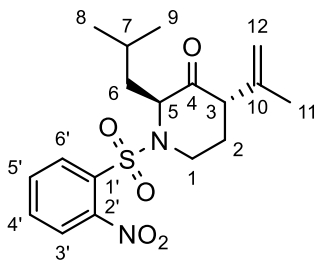


(*R*)-S2b

According to GP5 (*R*)-**S2b** was prepared from (2*R*,3*S*,4*R*)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*S*,*R*)-**30b** (0.21 g, 0.56 mmol) and DMP (0.59 g, 1.40 mmol) as a colorless solid (0.17 g, 0.47 mmol, 84 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1). mp 124 °C. $[\alpha]_D^{20}$ - 15 (*c* 1.0 in $CHCl_3$). R_f = 0.51 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3091 (w),

2927 (m), 2934 (w), 1717 (s), 1650 (w), 1590 (w), 1544 (vs), 1467 (m), 1440 (w), 1371 (vs), 1302 (w), 1195 (w), 1163 (vs), 1126 (m), 1091 (w), 1061 (w), 1040 (w), 981 (m), 905 (m), 852 (w), 778 (m), 761 (m), 740 (m), 728 (m), 675 (w), 655 (w), 585 (s) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.89 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.94 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.53 (s, 3H, 10-H), 1.89 (dddd, *J* = 12.6 Hz, 12.6 Hz, 12.6 Hz, 4.7 Hz, 1H, 2-H_a), 1.91 – 1.99 (m, 1H, 2-H_b), 2.22 (dqq, *J* = 10.7 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 3.16 (dd, *J* = 12.6 Hz, 6.4 Hz, 1H, 3-H), 3.52 (ddd, *J* = 15.2 Hz, 12.6 Hz, 3.3 Hz, 1H, 1-H_a), 3.87 (d, *J* = 10.7 Hz, 1H, 5-H), 4.11 – 4.19 (m, 1H, 1-H_b), 4.62 (s, 1H, 11-H_a), 4.90 (s, 1H, 11-H_b), 7.57 – 7.63 (m, 1H, 3'-H), 7.64 – 7.73 (m, 2H, 4'-H, 5'-H), 7.94 –8.02 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 18.6 (C-7/C-8), 19.4 (C-8/C-7), 20.2 (C-10), 28.0 (C-6), 31.5 (C-2), 40.8 (C-1), 53.6 (C-3), 71.8 (C-5), 114.4 (C-11), 124.0 (C-3'), 131.0 (C-6'), 131.9 (C-5'), 133.5 (C-1'), 133.8 (C-4'), 141.2 (C-9), 147.8 (C-2'), 204.4 (C-4). HRMS (ESI): calc (found) for $C_{17}H_{22}N_2O_5S$ ($[M+Na]^+$): 389.1142 (389.1143).

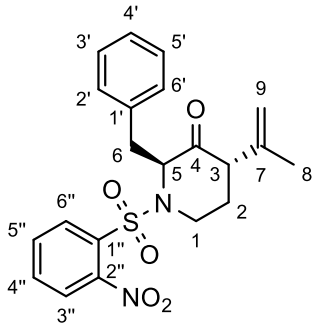
(2*S*,4*S*)-2-Isobutyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-one ((*S*)-S2c)



(*S*)-S2c

According to GP5 (*S*)-**S2c** was prepared from (2*S*,4*S*)-2-isobutyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*R*,*S*)-**30c** (60.0 mg, 0.16 mmol) and DMP (0.17 mg, 0.39 mmol) as a colorless solid (49.0 mg, 0.13 mmol, 82 %). Purification was accomplished via column chromatography on SiO_2 with hexanes/EtOAc (2 : 1). mp 136 °C. $[\alpha]_D^{20}$ + 38 (*c* 1.0 in $CHCl_3$). R_f = 0.32 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3091 (w), 2958 (m), 2929 (w), 2871 (w), 1716 (s), 1650 (w), 1590 (w), 1542 (vs), 1468 (m), 1440 (m), 1369 (vs), 1297 (m), 1194 (w), 1160 (vs), 1126 (s), 1098 (m), 1061 (m), 1019 (m), 998 (w), 968 (s), 911 (s), 852 (m), 836 (w), 777 (m), 749 (s), 733 (s), 654 (m), 621 (w), 587 (vs), 573 (vs), 538 (w), 477 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.87 (d, *J* = 6.1 Hz, 3H, 8-H/9-H), 0.94 (d, *J* = 6.1 Hz, 3H, 9-H/8-H), 1.55 (s, 3H, 11-H), 1.56 – 1.64 (m, 2H, 6-H_a, 7-H), 1.66 – 1.76 (m, 1H, 6-H_b), 1.90 –1.98 (m, 2H, 2-H), 3.18 – 3.24 (m, 1H, 3-H), 3.50 – 3.62 (m, 1H, 1-H_a), 4.03 – 4.11 (m, 1H, 1-H_b), 4.34 – 4.41 (m, 1H, 5-H), 4.67 (s, 1H, 12-H_a), 4.91 (s, 1H, 12-H_b), 7.62 – 7.66 (m, 1H, 3-H'), 7.67 – 7.74 (m, 2H, 4-H', 5-H'), 8.03 – 8.06 (m, 1H, 6-H'). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 20.0 (C-11), 22.1 (C-8/C-9), 22.5 (C-9/C-8), 24.4 (C-7), 30.5 (C-2), 39.4 (C-6), 40.3 (C-1), 53.1 (C-3), 63.8 (C-5), 114.8 (C-12), 124.3 (C-3'), 131.1 (C-6'), 132.0 (C-5'), 133.4 (C-1'), 133.9 (C-4'), 141.4 (C-10), 147.9 (C-2'), 204.7 (C-4). HRMS (ESI): calc (found) for $C_{18}H_{24}N_2O_5S$ ($[M+H]^+$): 381.1479 (381.1474).

(2*S*,4*S*)-2-Benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-one ((*S*)-S2f)



(*S*)-S2f

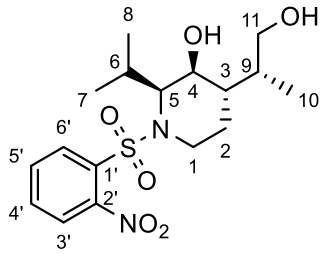
According to GP5 (***S*)-S2f** was prepared from a mixture of (2*S*,3*R*,4*S*)-2-benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-ol (*R*,*S*)-**30f** and (2*S*,3*S*,4*S*)-2-benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-ol (*S*,*S*)-**30f** (0.10 g, 0.24 mmol) and DMP (0.25 g, 0.60 mmol) as a colorless solid (0.10 g, 0.24 mmol, quant). mp 178 °C. $[\alpha]_D^{20}$ + 108 (*c* 1.0 in $CHCl_3$). R_f = 0.24 (hexanes/EtOAc, 3 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3087 (w), 3028 (w), 2930 (w), 2865 (w), 1717 (s), 1650 (w), 1590 (w), 1541 (vs), 1496 (w), 1455 (m), 1440 (m), 1368 (s), 1303 (m), 1251 (w), 1161 (vs), 1126 (s), 1100 (m), 1061 (m), 1030 (w), 1012 (w), 967 (s), 909 (m), 852 (m), 756 (s), 734 (s), 700 (s), 654 (m), 596 (s), 573 (s), 506 (w), 465 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.64 (s, 3H, 8-H), 1.98 (ddt, *J* = 13.4 Hz, 6.1 Hz, 2.7 Hz, 1H, 2-H_a), 2.08 (ddt, *J* = 13.4 Hz, 12.6 Hz, 4.5 Hz, 1H, 2-H_b), 3.05 (dd, *J* = 13.9 Hz, 6.2 Hz, 1H, 6-H_a), 3.15 (dd, *J* = 13.9 Hz, 8.8 Hz, 1H, 6-H_b), 3.19 (dd, *J* = 12.6 Hz, 6.1 Hz, 1H, 3-H), 3.57 (ddd, *J* = 14.6 Hz, 12.5 Hz, 2.6 Hz, 1H, 1-H_a), 4.09 – 4.16 (m, 1H, 1-H_b), 4.52 – 4.58 (m, 1H, 5-H), 4.73 (s, 1H, 9-H_a), 4.90 – 4.96 (m, 1H, 9-H_b), 7.04 – 7.16 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 7.50 – 7.56 (m, 1H, 5''-H), 7.56 – 7.65 (m, 2H, 3''-H, 4''-H), 7.76 – 7.82 (m, 1H, 6''-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 20.1 (C-8), 30.8 (C-2), 37.3 (C-6), 41.1 (C-1), 54.4 (C-3), 66.8 (C-5), 114.8 (C-9), 124.5 (C-3''), 127.1 (C-4'), 128.5 ((C-2', C-6')/(C-3', C-5')), 129.1 ((C-3',C-5')/(C-6',C-2')), 131.1 (C-6''), 132.0 (C-5''), 133.3 (C-1''), 133.6 (C-4''), 135.4 (C-1'), 141.4 (C-7), 147.4 (C-2''), 203.8 (C-4). HRMS (ESI): calc (found) for $C_{21}H_{22}N_2O_5S$ ($[M+NH_4]^+$): 432.1588 (432.1588).

GP8: Conversion to diols with terminal alcohol

The reaction was performed according to the literature.[16] A round bottom flask was charged with a solution of the alkene (0.15 mmol) in tetrahydrofuran (2 mL) under nitrogen atmosphere. Upon cooling to 0 °C a solution of borane (1 M in THF) was added dropwise and the mixture was stirred for 20 h at room temperature. Potassium hydroxide solution (200 µL, 0.60 mmol, 3 M in water) and hydrogen peroxide (2.0 mL, 0.60 mmol, 30 %) were added and stirring was continued for 10 min. Dichloromethane (17 mL) was added and the phases were separated. The aqueous phase was extracted with dichloromethane (3 × 8 mL) and the organic phases were dried over sodium sulfate. The crude product was purified by HPLC.

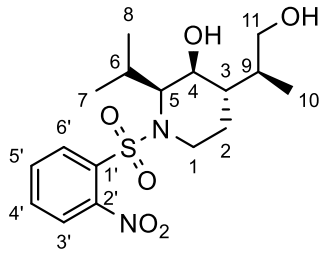
(2*S*,3*S*,4*S*)-4-((*R*)-1-Hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol ((*S*,*S*,*R*)-**34a) and (2*S*,3*S*,4*S*)-4-((*S*)-1-Hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol (**(*S*,*S*,*S*)-**34a)**

According to GP8 (*S*,*S*,*R*)-**34a** was prepared from (2*S*,4*S*)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)piperidin-3-one (*S*)-**S2a** (33.0 mg, 90.0 µmol), borane-THF-solution (0.12 mL, 0.12 mmol, 1 M in THF), hydrogen peroxide (1.2 mL, 0.36 mmol, 30 %) and potassium hydroxide (20.0 mg, 0.36 mmol) as a colorless solid (6.00 mg, 16.0 µmol, 17 %). Diol (*S*,*S*,*S*)-**34a** was isolated as well as a colorless solid (8.00 mg, 21.0 µmol, 23 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (95 : 5), 15 mL / min, R_t ((*S*,*S*,*R*)-**34a**) = 56.5 min, R_t ((*S*,*S*,*S*)-**34a**) = 93 min).



(*S*,*S*,*R*)-34a

(*S*,*S*,*R*)-**34a**, mp 58 °C. $[\alpha]_D^{20}$ + 96 (*c* 0.39 in $CHCl_3$). R_f = 0.34 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3277 (w, br), 2961 (m), 2930 (m), 2877 (m), 1590 (w), 1542 (vs), 1470 (m), 1440 (w), 1370 (s), 1344 (s), 1264 (w), 1204 (w), 1159 (vs), 1125 (m), 1079 (m), 1061 (m), 1017 (m), 993 (m), 953 (w), 911 (m), 884 (m), 852 (m), 778 (m), 764 (m), 730 (vs), 653 (m), 599 (vs), 571 (s), 530 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.63 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.87 (d, *J* = 7.3 Hz, 3H, 10-H), 1.13 (d, *J* = 6.6 Hz, 3H, 8-H, 7-H), 1.41 (dddd, *J* = 13.0 Hz, 13.0 Hz, 13.0 Hz, 5.1 Hz 1H, 2-H_a), 1.52 – 1.63 (m, 1H, 2-H_b), 1.87 – 2.00 (m, 2H, 3-H, 9-H), 2.16 (dqq, *J* = 9.8 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 2.98 (ddd, *J* = 13.0 Hz, 13.0 Hz, 2.8 Hz, 1H, 1-H_a), 3.54 (dd, *J* = 10.6 Hz, 2.8 Hz, 1H, 11-H_a), 3.63 (dd, *J* = 10.6 Hz, 6.3 Hz, 1H, 11-H_b), 3.71 (dd, *J* = 9.8 Hz, 4.9 Hz, 1H, 5-H), 3.83 (dd, *J* = 10.9 Hz, 4.9 Hz, 1H, 4-H), 3.88 – 3.97 (m, 1H, 1-H_b), 7.59 – 7.65 (m, 1H, 3'-H), 7.65 – 7.72 (m, 2H, 4'-H, 5'-H), 8.04 – 8.12 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 12.2 (C-10), 20.5 (C-7/C-8), 22.1 (C-8/C-7), 25.7 (C-6), 28.8 (C-2), 37.0 (C-3), 40.4 (C-9), 41.4 (C-1), 63.7 (C-5), 66.1 (C-11), 72.4 (C-4), 124.1 (C-3'), 130.8 (C-6'), 131.8 (C-5'), 133.3 (C-4'), 134.6 (C-1'), 147.4 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_6S$ ($[M+Na]^+$): 409.1404 (409.1404).



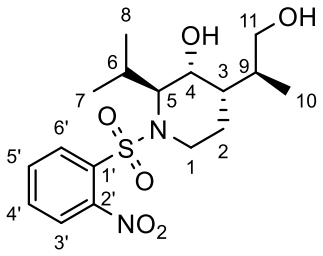
(*S*,*S*,*S*)-34a

(*S*,*S*,*S*)-**34a**, mp 165 °C. $[\alpha]_D^{20}$ + 130 (*c* 0.24 in $CHCl_3$). R_f = 0.25 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3366 (w, br), 2961 (m), 2878 (m), 1590 (w), 1544 (vs), 1471 (m), 1440 (w), 1372 (s), 1345 (s), 1267 (m), 1160 (s), 1125 (m), 1080 (m), 1024 (m), 985 (m), 946 (w), 912 (m), 891 (w), 852 (w), 764 (m), 736 (m), 653 (m), 600 (vs), 571 (m) cm^{-1}. ^{1}H-NMR (500 MHz,

$CDCl_3$) δ 0.66 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.77 (d, *J* = 6.9 Hz, 3H, 10-H), 1.14 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.29 (dddd, *J* = 13.0 Hz, 13.0 Hz, 13.0 Hz, 5.2 Hz, 1H, 2-H_a), 1.54 – 1.62 (m, 1H, 2-H_b), 1.95 – 2.13 (m, 4H, 3-H, 9-H, 2xOH), 2.19 (dqq, *J* = 9.8 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 2.98 (ddd, *J* = 13.0 Hz, 13.0 Hz, 2.9 Hz, 1H, 1-H_a), 3.47 (dd, *J* = 10.5 Hz, 7.8 Hz, 1H, 11-H_a), 3.60 (dd, *J* = 10.5 Hz, 5.2 Hz, 1H, 11-H_b), 3.70 (dd, *J* = 9.8 Hz, 4.9 Hz, 1H, 5-H), 3.85 (dd, *J* = 11.1 Hz, 4.9 Hz, 1H, 4-H), 3.87 – 3.95 (m, 1H, 1-H_b), 7.58 – 7.64 (m, 1H, 3'-H), 7.64 – 7.73 (m, 2H, 4'-H, 5'-H), 8.02 – 8.10 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 11.9 (C-10), 20.5 (C-7/C-8), 22.3 (C-8/C-7), 25.7 (C-6), 26.1 (C-2), 34.8 (C-9), 37.3 (C-3), 41.4 (C-1), 63.8 (C-5), 66.0 (C-11), 73.0 (C-4), 124.1 (C-3'), 130.7 (C-6'), 131.7 (C-5'), 133.2 (C-4'), 134.7 (C-1'), 147.5 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_6S$ ($[M+Na]^+$): 409.1404 (409.1406).

(2*S*,3*R*,4*S*)-4-((*S*)-1-Hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol ((*R*,*S*,*S*)-**34a und (2*S*,3*R*,4*S*)-4-((*R*)-1-Hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol (**(*R*,*S*,*R*)-**34a)**

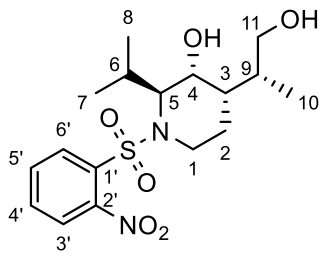
According to GP8 (*R*,*S*,*S*)-**34a** was prepared from (2*S*,3*R*,4*S*)-2-isopropyl-1-((2-nitrophenyl)-sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*R*,*S*)-**30a** (55.0 mg, 150 µmol), borane-THF-solution (0.2 mL, 0.20 mmol, 1 M in THF), hydrogen peroxide (2.0 mL, 0.60 mmol, 30 %) and potassium hydroxide (34.0 mg, 0.60 mmol) as a colorless solid (18.0 mg, 47.0 µmol, 31 %). Diol (*R*,*S*,*R*)-**34a** was isolated as well as a colorless solid (6.00 mg, 16.0 µmol, 11 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (95 : 5), 15 mL / min, R_t ((*R*,*S*,*S*)-**34a**) = 122 min, R_t ((*R*,*S*,*R*)-**34a**) = 134 min).



(*R*,*S*,*S*)-**34a**

(*R*,*S*,*S*)-**34a**, mp 138 °C. $[\alpha]_D^{20}$ + 154 (*c* 1.0 in $CHCl_3$). R_f = 0.24 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3539 (w), 3355 (w), 2964 (m), 2929 (m), 2877 (m), 1542 (vs), 1470 (m), 1439 (w), 1372 (s), 1335 (s), 1281 (w), 1207 (w), 1158 (s), 1125 (m), 1066 (m), 1044 (m), 970 (s), 913 (s), 853 (m), 778 (w), 751 (s), 734 (s), 685 (w), 663 (w), 651 (w), 620 (w), 578 (s), 560 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.72 (d, *J* = 6.8 Hz, 3H, 7-H/8-H), 0.93 – 1.00 (m, 6H, 8-H/7-H, 10-H), 1.36 – 1.45 (m, 1H, 2-H_a), 1.54 – 1.61 (m, 1H, 3-H), 1.61 – 1.69 (m, 1H, 9-H), 1.69 (dddd, *J* = 12.7 Hz, 12.7 Hz, 12.7 Hz, 4.9 Hz, 1H, 2-H_b), 2.01 (dqq, *J* = 11.0 Hz, 6.8 Hz, 6.8 Hz, 1H, 6-H), 2.84 (br, s, 2H, 2xOH), 3.10 (ddd, *J* = 15.3 Hz, 12.7 Hz, 2.7 Hz, 1H, 1-H_a), 3.50 (dd, *J* = 11.2 Hz, 5.2 Hz, 1H, 11-H_a), 3.55 (d, *J* = 11.0 Hz, 1H, 5-H), 3.65 (dd, *J* = 11.2 Hz, 3.0 Hz, 1H, 11-H_b), 4.00 – 4.08 (m, 1H, 1-H_b), 4.12 – 4.18 (m, 1H, 4-H), 7.52 – 7.59 (m, 1H, 3'-H), 7.61 – 7.70 (m, 2H, 4'-H, 5'-H), 8.08 – 8.16 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 15.4

(C-10), 19.9 (C-7/C-8), 20.2 (C-8/C-7), 24.3 (C-2), 26.7 (C-6), 37.3 (C-9), 38.3 (C-3), 42.2 (C-1), 64.6 (C-11), 64.8 (C-4), 66.9 (C-5), 123.8 (C-3'), 131.0 (C-6'), 131.6 (C-5'), 133.3 (C-4'), 134.7 (C-1'), 147.7 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_6S$ ($[M+H]^+$): 387.1584 (387.1586).

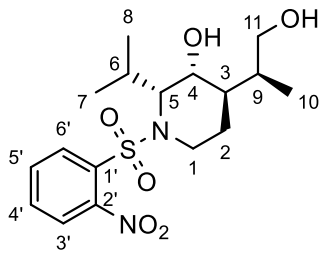


(*R*,*S*,*R*)-**34a**

(*R*,*S*,*R*)-**34a**, mp 41 °C. $[\alpha]_D^{20}$ + 129 (*c* 1.0 in $CHCl_3$). R_f = 0.24 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3547 (w), 3345 (w), 2963 (m), 2927 (m), 2876 (m), 1543 (vs), 1469 (m), 1440 (w), 1372 (s), 1336 (s), 1281 (w), 1159 (s), 1125 (m), 1076 (m), 1046 (m), 995 (w), 967 (m), 912 (m), 853 (w), 778 (w), 751 (m), 734 (m), 652 (w), 581 (m), 560 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.71 (d, *J* = 6.8 Hz, 3H, 7-H/8-H), 0.96 (d, *J* = 6.8 Hz, 3H, 8-H/7-H), 0.97 (d, *J* = 6.6 Hz, 3H, 10-H), 1.43 – 1.54 (m, 1H, 2-H_a), 1.63 (dddd, *J* = 12.7 Hz, 12.7 Hz, 12.7 Hz, 4.4 Hz, 1H, 2-H_b), 1.62 – 1.70 (m, 1H, 3-H), 1.70 – 1.80 (m, 1H, 9-H), 2.02 (dqq, *J* = 10.7 Hz, 6.8 Hz, 6.8 Hz, 1H, 6-H), 2.94 (s, br, 2H, 2xOH), 3.05 – 3.18 (m, 1H, 1-H_a), 3.45 (dd, *J* = 11.1 Hz, 4.2 Hz, 1H, 11-H_a), 3.49 (dd, *J* = 11.1 Hz, 6.4 Hz, 1H, 11-H_b), 3.53 (d, *J* = 10.7 Hz, 1H, 5-H), 4.01 (s, 1H, 4-H), 4.04 – 4.13 (m, 1H, 1-H_b), 7.53 – 7.60 (m, 1H, 3'-H), 7.60 – 7.70 (m, 2H, 4'-H, 5'-H), 8.05 – 8.15 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 16.3 (C-10), 19.8 (C-7/C-8), 20.3 (C-8/C-7), 21.4 (C-2), 26.7 (C-6), 37.6 (C-9), 38.1 (C-3), 42.3 (C-1), 64.5 (C-11), 67.1 (C-5), 67.1 (C-4), 123.8 (C-3'), 130.9 (C-6'), 131.6 (C-5'), 133.3 (C-4'), 134.7 (C-1'), 147.7 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_6S$ ($[M+H]^+$): 387.1584 (387.1583).

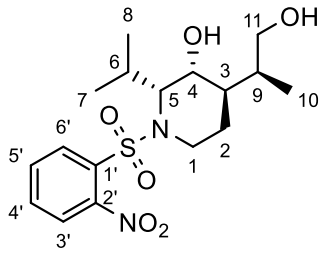
(2*R*,3*R*,4*S*)-4-((*S*)-1-Hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol ((*R*,*R*,*S*)-**34b), (2*R*,3*R*,4*R*)-4-((*R*)-1-Hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol (**(*R*,*R*,*R*)-**34b) and (2*R*,3*S*,4*R*)-4-((*S*)-1-Hydroxypropan-2-yl)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol (**(*S*,*R*,*S*)-**34b)**

According to GP8 (*R*,*R*,*S*)-**34b** was prepared from (2*R*,4*R*)-2-isopropyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-one (*R*)-**S2b** (0.15 g, 0.40 mmol), borane solution (0.54 mL, 0.54 mmol, 1 M in THF), potassium hydroxide (91 mg, 1.62 mmol) and hydrogen peroxide (5.4 mL, 1.62 mmol, 30 %) as a colorless oil (59.0 mg, 0.15 mmol, 38 %). Diol (*R*,*R*,*R*)-**34b** was isolated as a colorless solid (56.0 mg, 0.15 mmol, 36 %) and Diol (*S*,*R*,*S*)-**34b** was isolated as a colorless oil (12.0 mg, 31 µmol, 8 %). Purification was accomplished by HPLC (Orbit, hexanes/EtOAc (40 : 60), 15 mL / min, R_t ((*R*,*R*,*S*)-**34b**) = 26 min, R_t ((*R*,*R*,*R*)-**34b**) = 32 min, R_t ((*S*,*R*,*S*)-**34b**) = 39.5 min).



(*R*,*R*,*S*)-34b

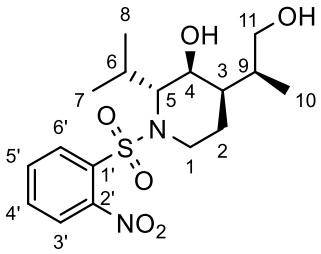
(*R*,*R*,*S*)-**34b**, $[\alpha]_D^{20}$ - 119 (*c* 1.0 in $CHCl_3$). R_f = 0.38 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3276 (br, w), 2961 (m), 2877 (m), 1591 (w), 1542 (vs), 1470 (m), 1440 (w), 1370 (s), 1343 (s), 1262 (m), 1204 (w), 1158 (s), 1125 (m), 1079 (s), 1060 (m), 1017 (m), 992 (s), 953 (m), 910 (s), 884 (m), 852 (m), 778 (m), 764 (s), 728 (vs), 653 (m), 598 (vs), 570 (s), 530 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.62 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.86 (d, *J* = 7.2 Hz, 3H, 10-H), 1.12 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.40 (dddd, *J* = 13.0 Hz, 13.0 Hz, 13.0 Hz, 5.2 Hz, 1H, 2-H_a), 1.52 – 1.61 (m, 1H, 2-H_b), 1.85 (br, s, 1H, OH), 1.88 – 1.97 (m, 2H, 3-H, 9-H), 2.15 (dqq, *J* = 9.8 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 2.98 (ddd, *J* = 14.6 Hz, 13.0 Hz, 3.1 Hz, 1H, 1-H_a), 3.53 (dd, *J* = 10.6 Hz, 3.2 Hz, 1H, 11-H_a), 3.61 (dd, *J* = 10.6 Hz, 6.0 Hz, 1H, 11-H_b), 3.70 (dd, *J* = 9.8 Hz, 5.0 Hz, 1H, 5-H), 3.82 (dd, *J* = 11.1 Hz, 5.0 Hz, 1H, 4-H), 3.86 – 3.95 (m, 1H, 1-H_b), 7.58 – 7.64 (m, 1H, 3'-H), 7.64 – 7.71 (m, 2H, 4'-H, 5'-H), 8.03 – 8.10 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 12.2 (C-10), 20.5 (C-7/C-8), 22.1 (C-8/C-7), 25.7 (C-6), 28.8 (C-2), 37.1 (C-3), 40.4 (C-9), 41.4 (C-1), 63.7 (C-5), 66.1 (C-11), 72.3 (C-4), 124.1 (C-3'), 130.8 (C-6'), 131.8 (C-5'), 133.3 (C-4'), 134.6 (C-1'), 147.4 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_6S$ ($[M+H]^+$): 387.1584 (387.1585).



(*R*,*R*,*R*)-34b

(*R*,*R*,*R*)-**34b**, mp 167 °C. $[\alpha]_D^{20}$ - 107 (*c* 1.0 in $CHCl_3$). R_f = 0.27 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3366 (br, w), 2961 (m), 2878 (m), 1590 (w), 1543 (vs), 1471 (w), 1440 (w), 1372 (s), 1345 (s), 1268 (w), 1159 (s), 1125 (m), 1080 (m), 1024 (m), 1002 (m), 985 (m), 947 (w), 912 (m), 891 (w), 853 (w), 764 (m), 736 (m), 653 (m), 600 (vs), 571 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.65 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.76 (d, *J* = 7.0 Hz, 3H, 10-H), 1.13 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.28 (dddd, *J* = 13.1 Hz, 13.1 Hz, 13.1 Hz, 5.0 Hz, 1H, 2-H_a), 1.53 – 1.60 (m, 1H, 2-H_b), 1.64 (s, 1H, OH), 1.95 – 2.11 (m, 2H, 3-H, 9-H), 2.18 (dqq, *J* = 9.9 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 2.98 (ddd, *J* = 14.5 Hz, 13.1 Hz, 3.0 Hz, 1H, 1-H_a), 3.47 (dd, *J* = 10.5 Hz, 7.7 Hz, 1H, 11-H_a), 3.59 (dd, *J* = 10.5 Hz, 5.3 Hz, 1H, 11-H_b), 3.69 (dd, *J* = 9.9 Hz, 5.0 Hz, 1H, 5-H), 3.84 (dd, *J* = 11.1 Hz, 5.0 Hz, 1H, 4-H), 3.87 – 3.95 (m, 1H, 1-H_b), 7.57 – 7.64 (m, 1H, 3'-H), 7.64 – 7.71 (m, 2H, 4'-H, 5'-H), 8.00 – 8.10 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 11.9 (C-10), 20.5 (C-7/C-8), 22.3 (C-8/C-7), 25.7 (C-2), 26.1 (C-6), 34.8 (C-9), 37.3 (C-3), 41.4 (C-1), 63.9 (C-5), 66.0 (C-11), 73.0 (C-4), 124.1 (C-3'), 130.7 (C-6'),

131.7 (C-5'), 133.2 (C-4'), 134.1 (C-1'), 147.5 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_6S$ ($[M+H]^+$): 387.1584 (387.1589).

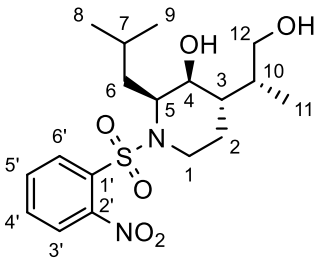


(*S*,*R*,*S*)-34b

(*S*,*R*,*S*)-**34b**, $[\alpha]_D^{20}$ - 129 (*c* 0.62 in $CHCl_3$). R_f = 0.22 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3335 (br, w), 2961 (m), 2927 (m), 2876 (m), 1591 (w), 1542 (vs), 1468 (m), 1439 (w), 1371 (s), 1334 (s), 1281 (m), 1211 (w), 1157 (s), 1124 (s), 1075 (m), 1063 (m), 1045 (m), 994 (m), 966 (s), 910 (vs), 852 (m), 810 (w), 777 (m), 751 (s), 731 (vs), 687 (w), 651 (m), 628 (w), 580 (vs), 560 (s) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.70 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.95 (d, *J* = 7.0 Hz, 3H, 10-H), 0.96 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.40 – 1.52 (m, 1H, 2-H_a), 1.62 (dddd, *J* = 12.6 Hz, 12.6 Hz, 12.6 Hz, 4.5 Hz, 1H, 2-H_b), 1.62 – 1.70 (m, 1H, 3-H), 1.70 – 1.80 (m, 1H, 9-H), 2.02 (dqq, *J* = 10.8 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 2.90 – 3.30 (m, 2H, 1-H_a, OH), 3.44 (dd, *J* = 11.1 Hz, 4.4 Hz, 1H, 11-H_a), 3.47 (dd, *J* = 11.1 Hz, 6.6 Hz, 1H, 11-H_b), 3.51 – 3.57 (m, 1H, 5-H), 4.00 (s, 1H, 4-H), 4.02 – 4.10 (m, 1H, 1-H_b), 7.53 – 7.59 (m, 1H, 3'-H), 7.62 – 7.70 (m, 2H, 4'-H, 5'-H), 8.06 – 8.15 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 16.3 (C-10), 19.8 (C-7/C-8), 20.3 (C-8/C-7), 21.3 (C-2), 26.7 (C-6), 37.6 (C-9), 38.2 (C-3), 42.3 (C-1), 64.4 (C-11), 67.0 (C-5), 67.1 (C-4), 123.8 (C-3'), 130.9 (C-6'), 131.6 (C-5'), 133.3 (C-4'), 134.7 (C-1'), 147.7 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_6S$ ($[M+H]^+$): 387.1584 (387.1583).

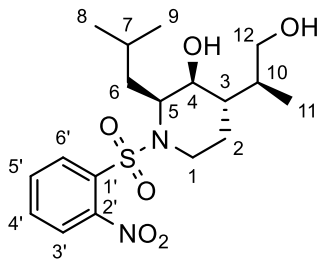
(2*S*,3*S*,4*S*)-4-((*R*)-1-Hydroxypropan-2-yl)-2-isobutyl-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol ((*S*,*S*,*R*)-**34c) and (2*S*,3*S*,4*S*)-4-((*S*)-1-Hydroxypropan-2-yl)-2-isobutyl-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol (**(*S*,*S*,*S*)-**34c)**

According to GP8 (*S*,*S*,*R*)-**34c** was prepared from (2*S*,4*S*)-2-isobutyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-one (*S*)-**S2c** (43.0 mg, 0.11 mmol), borane-THF-solution (0.15 mL, 1 M in THF, 0.15 mmol), potassium hydroxide (25.0 mg, 0.44 mmol) and hydrogen peroxide (1.46 mL, 30 % in water, 0.44 mmol) as a colorless solid (13.0 mg, 0.03 mmol, 20 %). Diol (*S*,*S*,*S*)-**34c** was isolated as well as a colorless solid (9.00 mg, 0.02 mmol, 29 %). Purification was accomplished by HPLC (Orbit, hexane/isopropanol (90 : 10), 15 mL / min, R_t ((*S*,*S*,*R*)-**34c**) = 35 min, R_t ((*S*,*S*,*S*)-**34c**) = 43 min).



(*S*,*S*,*R*)-34c

(*S*,*S*,*R*)-**34c**, mp 48 °C. $[\alpha]_D^{20}$ + 138 (*c* 0.1 in $CHCl_3$). R_f = 0.26 (hexanes/EtOAc, 1 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 2956 (m), 2924 (m), 2872 (m), 1543 (vs), 1467 (m), 1439 (w), 1371 (s), 1339 (s), 1268 (m), 1210 (w), 1161 (s), 1125 (m), 1067 (m), 1038 (m), 996 (m), 939 (m), 926 (m), 852 (m), 779 (w), 747 (m), 733 (m), 680 (w), 652 (w), 619 (w), 577 (m), 526 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.85 (d, *J* = 6.2 Hz, 3H, 8-H/9-H), 0.88 (d, *J* = 6.2 Hz, 3H, 9-H/8-H), 0.96 (d, *J* = 7.1 Hz, 3H, 11-H), 1.35 – 1.43 (m, 1H, 6-H_a), 1.46 – 1.58 (m, 3H, 2-H_a, 6-H_b, 7-H), 1.60 – 1.69 (m, 2H, 2-H_b, 3-H), 1.70 – 1.80 (m, 1H, 10-H), 3.13 –3.23 (m, 1H, 1-H_a), 3.47 (dd, *J* = 11.1 Hz, 4.4 Hz, 1H, 12-H_a), 3.50 (dd, *J* = 11.1 Hz, 6.3 Hz, 1H, 12-H_b), 3.70 – 3.75 (m, 1H, 4-H), 3.92 – 4.03 (m, 2H, 1-H_b, 5-H), 7.57 – 7.64 (m, 1H, 3'-H), 7.64 – 7.71 (m, 2H, 4'-H, 5'-H), 8.10 – 8.16 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 16.3 (C-11), 21.2 (C-2), 22.6 (C-8/C-9), 22.7 (C-9/C-8), 24.9 (C-3), 37.5 (C-10), 37.8 (C-7), 38.0 (C-6), 41.5 (C-1), 58.8 (C-5), 64.5 (C-12), 69.3 (C-4), 124.1 (C-3'), 131.1 (C-6'), 131.7 (C-5'), 133.4 (C-4'), 134.1 (C-1'), 147.8 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{24}N_2O_5S$ ($[M+H]^+$): 401.1741 (401.1742).

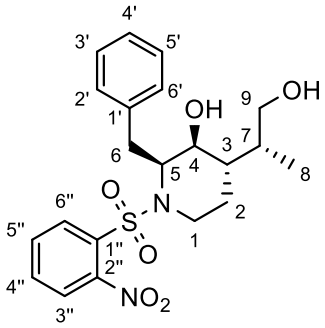


(*S*,*S*,*S*)-34c

(*S*,*S*,*S*)-**34c**, mp 172 °C. $[\alpha]_D^{20}$ + 14 (*c* 0.2 in $CHCl_3$). R_f = 0.21 (hexanes/EtOAc, 1 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3346 (m, br), 2956 (s), 2926 (s), 1545 (vs), 1467 (m), 1439 (m), 1372 (s), 1269 (m), 1163 (s), 1125 (m), 1079 (m), 1038 (m), 998 (w), 942 (m), 852 (w), 780 (w), 748 (m), 731 (m), 652 (w), 595 (s), 568 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.74 (d, *J* = 5.9 Hz, 3H, 8-H/9-H), 0.76 (d, *J* = 7.1 Hz, 3H, 11-H), 0.85 (d, *J* = 5.9 Hz, 3H, 9-H/8-H), 1.17 – 1.29 (m, 1H, 2-H_a), 1.39 – 1.50 (m, 2H, 6-H_a, 7-H), 1.52 – 1.61 (m, 2H, 2-H_b, 6-H_b), 1.80 – 1.89 (m, 1H, 3-H), 1.92 – 2.01 (m, 1H, 10-H), 3.05 – 3.15 (m, 1H, 1-H_a), 3.47 (dd, *J* = 10.4 Hz, 7.8 Hz, 1H, 12-H_a), 3.60 (dd, *J* = 10.4 Hz, 5.3 Hz, 1H, 12-H_b), 3.72 (dd, *J* = 11.0 Hz, 5.4 Hz, 1H, 4-H), 3.79 – 3.86 (m, 1H, 1-H_b), 4.05 – 4.13 (m, 1H, 5-H), 7.60 – 7.66 (m, 1H, 3'-H), 7.66 – 7.73 (m, 2H, 4'-H, 5'-H), 8.07 – 8.13 (m, 1H, 6'-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 12.1 (C-11), 21.4 (C-8/C-9), 24.0 (C-9/C-8), 24.2 (C-7), 25.8 (C-2), 32.8 (C-6), 35.2 (C-10), 37.3 (C-3), 40.1 (C-1), 56.5 (C-5), 66.1 (C-12), 70.4 (C-4), 124.3 (C-3'), 131.0 (C-6'), 131.7 (C-5'), 133.4 (C-4'), 134.4 (C-1'), 147.8 (C-2'). HRMS (ESI): calc (found) for $C_{18}H_{24}N_2O_5S$ ($[M+Na]^+$): 423.1560 (423.1562).

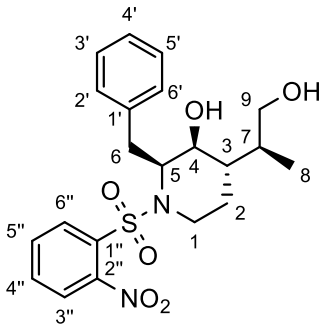
(2*S*,3*S*,4*S*)-2-Benzyl-4-((*R*)-1-hydroxypropan-2-yl)-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol ((*S*,*S*,*R*)-**34f), (2*S*,3*R*,4*S*)-2-Benzyl-4-((*S*)-1-hydroxypropan-2-yl)-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol (**(*R*,*S*,*S*)-**34f), (2*S*,3*S*,4*S*)-2-Benzyl-4-((*S*)-1-hydroxypropan-2-yl)-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol (**(*S*,*S*,*S*)-**34f) and (2*S*,3*R*,4*S*)-2-Benzyl-4-((*R*)-1-hydroxypropan-2-yl)-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol (**(*R*,*S*,*R*)-**34f)**

According to GP8 (*S*,*S*,*R*)-**34f** was prepared from (2*S*,4*S*)-2-benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-one (*S*)-**S2f** (0.16 g, 0.39 mmol), borane-solution (0.52 mL, 0.52 mmol, 1 M in THF), potassium hydroxide (88.0 mg, 1.56 mmol) and hydrogen peroxide (5.2 mL, 53.0 mg, 1.56 mmol, 30 %) as a colorless solid (35.0 mg, 31.0 µmol, 21 %). Diols (*R*,*S*,*S*)-**34f**, (*S*,*S*,*S*)-**34f** and (*R*,*S*,*R*)-**34f** were isolated as well as colorless solids ((*R*,*S*,*S*)-**34f**, 5.00 mg, 12.0 µmol, 3 %; (*S*,*S*,*S*)-**34f**, 26.0 mg, 60.0 µmol, 15 %; (*R*,*S*,*R*)-**34f,** 17.0 mg, 39.0 µmol, 10 %). Purification was accomplished by HPLC (Orbit, hexanes/EtOAc (40 : 60), 12 mL / min, R_t ((*S*,*S*,*R*)-**34f**) = 27 min, R_t ((*R*,*S*,*S*)-**34f**) = 30.5 min and Orbit, hexanes/isopropanol (90 : 10), 15 mL / min, R_t ((*S*,*S*,*S*)-**34f**) = 22 min, R_t ((*R*,*S*,*R*)-**34f**) = 45 min).



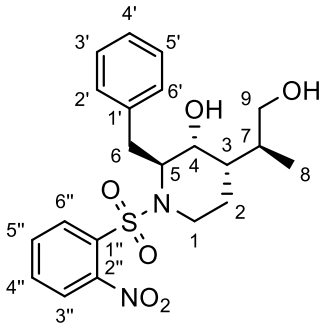
(*S*,*S*,*R*)-34f

(*S*,*S*,*R*)-**34f**, mp 60 °C. $[\alpha]_D^{20}$ + 158 (*c* 1.0 in $CHCl_3$). R_f = 0.35 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3279 (br, w), 3029 (w), 2937 (w), 2880 (w), 1592 (w), 1539 (vs), 1497 (w), 1455 (m), 1440 (m), 1366 (s), 1336 (s), 1263 (w), 1199 (w), 1157 (s), 1126 (s), 1067 (m), 1009 (m), 953 (s), 909 (s), 870 (m), 852 (m), 834 (w), 777 (w), 755 (s), 729 (vs), 699 (vs), 650 (m), 590 (vs), 570 (s), 503 (m), 462 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.99 (d, *J* = 7.3 Hz, 3H, 8-H), 1.59 (dddd, *J* = 12.9 Hz, 12.9 Hz, 12.9 Hz, 4.9 Hz, 1H, 2-H_a), 1.64 – 1.71 (m, 1H, 2-H_b), 1.73 (s, br, 1H, OH), 1.90 – 2.02 (m, 2H, 3-H, 7-H), 2.78 (dd, *J* = 14.3 Hz, 11.5 Hz, 1H, 6-H_a), 3.12 (dd, *J* = 14.3 Hz, 3.1 Hz, 1H, 6-H_b), 3.29 – 3.38 (m, 1H, 1-H_a), 3.63 (dd, *J* = 10.5 Hz, 3.1 Hz, 1H, 9-H_a), 3.74 (dd, *J* = 10.5 Hz, 5.8 Hz, 1H, 9-H_b), 3.92 (dd, *J* = 11.0 Hz, 5.5 Hz, 1H, 4-H), 3.96 – 4.03 (m, 1H, 1-H_b), 4.30 (ddd, *J* = 11.5 Hz, 5.5 Hz, 3.1 Hz, 1H, 5-H), 6.82 – 6.92 (m, 3H, 3'-H, 4'-H, 5'-H), 6.96 – 7.03 (m, 2H, 2'-H, 6'-H), 7.21 – 7.28 (m, 1H, 5"-H), 7.31 – 7.38 (m, 1H, 6"-H), 7.40 – 7.50 (m, 2H, 3"-H, 4"-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 12.1 (C-8), 29.7 (C-2), 29.9 (C-6), 37.4 (C-7), 40.7 (C-1, C-3), 61.5 (C-5), 66.4 (C-9), 70.3 (C-4), 124.1 (C-3"), 126.2 (C-4'), 128.0 (C-3', C-5'), 129.2 (C-2', C-6'), 130.6 (C-6"), 131.9 (C-5"), 132.6 (C-4"), 134.1 (C-1"), 138.7 (C-1'), 147.0 (C-2"). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_6S$ ($[M+Na]^+$): 457.1404 (457.1404).



(*S*,*S*,*S*)-34f

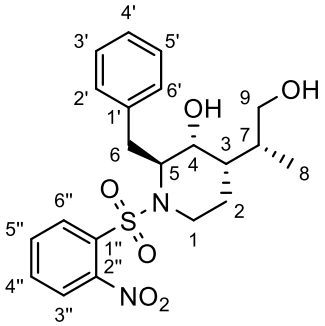
(*S*,*S*,*S*)-**34f**, mp 183 °C. $[\alpha]_D^{20}$ + 150 (*c* 1.0 in $CHCl_3$). R_f = 0.29 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3530 (w), 3365 (br, w), 3091 (w), 3027 (w), 2957 (m), 2930 (m), 2881 (w), 1591 (w), 1541 (vs), 1497 (w), 1454 (m), 1441 (m), 1371 (s), 1336 (s), 1268 (w), 1159 (vs), 1126 (m), 1074 (m), 1032 (m), 955 (s), 911 (m), 881 (w), 852 (m), 834 (w), 779 (w), 756 (s), 735 (s), 700 (m), 651 (w), 593 (s), 568 (m), 500 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.91 (d, *J* = 6.9 Hz, 3H, 8-H), 1.45 (dddd, *J* = 13.0 Hz, 13.0 Hz, 13.0 Hz, 4.9 Hz, 1H, 2-H_a), 1.56 (s, 1H, OH), 1.70 – 1.77 (m, 1H, 2-H_b), 1.95 – 2.10 (m, 2H, 3-H, 7-H), 2.81 (dd, *J* = 14.2 Hz, 11.3 Hz, 1H, 6-H_a), 3.11 (dd, *J* = 14.2 Hz, 3.3 Hz, 1H, 6-H_b), 3.28 – 3.37 (m, 1H, 1-H_a), 3.56 (dd, *J* = 10.5 Hz, 7.8 Hz, 1H, 9-H_a), 3.70 (dd, *J* = 10.5 Hz, 4.8 Hz, 1H, 9-H_b), 3.94 (dd, *J* = 10.9 Hz, 5.4 Hz, 1H, 4-H), 3.96 – 4.02 (m, 1H, 1-H_b), 4.29 (ddd, *J* = 11.3 Hz, 5.4 Hz, 3.3 Hz, 1H, 5-H), 6.84 – 6.94 (m, 3H, 3'-H, 4'-H, 5'-H), 6.98 – 7.04 (m, 2H, 2'-H, 6'-H), 7.26 – 7.31 (m, 1H, 5"-H), 7.35 – 7.41 (m, 1H, 6"-H), 7.42 – 7.50 (m, 2H, 3"-H, 4"-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 12.5 (C-8), 27.1 (C-2), 30.1 (C-6), 35.6 (C-7), 38.0 (C-3), 40.6 (C-1), 61.6 (C-5), 66.1 (C-9), 71.4 (C-4), 124.2 (C-3"), 126.3 (C-4'), 128.1 (C-3', C-5'), 129.2 (C-2', C-6'), 130.6 (C-6"), 131.8 (C-5"), 132.7 (C-4"), 134.1 (C-1"), 138.5 (C-1'), 147.2 (C-2"). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_6S$ ($[M+H]^+$): 435.1584 (435.1584).



(*R*,*S*,*S*)-34f

(*R*,*S*,*S*)-**34f**, mp 53 °C. $[\alpha]_D^{20}$ + 90 (*c* 0.26 in $CHCl_3$). R_f = 0.33 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3326 (br, w), 3087 (w), 3028 (w), 2957 (m), 2927 (m), 1731 (w), 1591 (w), 1542 (vs), 1495 (w), 1455 (m), 1440 (m), 1372 (s), 1338 (s), 1273 (w), 1160 (s), 1126 (m), 1066 (m), 1032 (m), 991 (m), 966 (w), 946 (m), 930 (m), 852 (m), 780 (w), 758 (m), 745 (m), 702 (m), 680 (w), 653 (w), 572 (m), 543 (w), 516 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.00 (d, *J* = 6.9 Hz, 3H, 8-H), 1.52 – 1.57 (m, 1H, 2-H_a),1.58 (s, br, 1H, OH), 1.66 – 1.75 (m, 1H, 7-H), 1.76 – 1.89 (m, 2H, 2-H_b, 3-H), 2.24 (s, br, 1H, OH), 2.90 (dd, *J* = 13.5 Hz, 9.2 Hz, 1H, 6-H_a), 2.99 (dd, *J* = 13.5 Hz, 6.6 Hz, 1H, 6-H_b), 3.28 – 3.37 (m, 1H, 1-H_a), 3.49 (dd, *J* = 11.0 Hz, 5.1 Hz, 1H, 9-H_a), 3.63 (dd, *J* = 11.0 Hz, 3.0 Hz, 1H, 9-H_b), 3.80 – 3.86 (m, 1H, 4-H), 3.96 – 4.04 (m, 1H, 1-H_b), 4.17 – 4.24 (m, 1H, 5-H), 7.09 – 7.20 (m, 5H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 7.54 – 7.65 (m, 3H, 3"-H, 4"-H, 5"-H), 7.91 – 7.97 (m, 1H, 6"-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 15.3 (C-8), 24.3 (C-2), 35.8 (C-6), 37.1 (C-7), 37.9 (C-3), 41.9 (C-1), 62.2 (C-5), 64.6 (C-9), 65.5 (C-4), 124.3 (C-3"), 126.9 (C-

4'), 128.6 (C-2', C-6'), 129.1 (C-3', C-5'), 131.1 (C-6''), 131.9 (C-5''), 133.3 (C-4''), 134.0 (C-1''), 137.3 (C-1'), 147.6 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_6S$ ($[M+Na]^+$): 457.1404 (457.1399).



(*R*,*S*,*R*)-**34f**

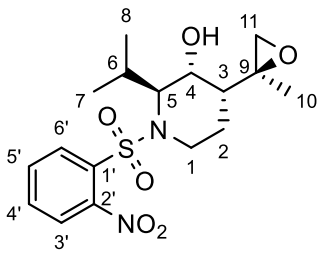
(*R*,*S*,*R*)-**34f**, mp 57 °C. $[\alpha]_D^{20}$ + 60 (*c* 1.0 in $CHCl_3$). R_f = 0.26 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3540 (w), 3304 (br, w), 3090 (w), 3027 (w), 2958 (m), 2928 (m), 2879 (w), 1591 (w), 1541 (vs), 1495 (w), 1455 (m), 1440 (w), 1371 (s), 1335 (s), 1272 (w), 1159 (vs), 1126 (m), 1066 (m), 1034 (m), 994 (m), 963 (m), 945 (m), 929 (m), 911 (m), 878 (w), 852 (m), 778 (w), 758 (m), 732 (vs), 701 (m), 680 (m), 651 (w), 616 (w), 574 (s), 543 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.95 (d, *J* = 7.0 Hz, 3H, 8-H), 1.52 – 1.64 (m, 1H, 2-H_a), 1.70 – 1.82 (m, 2H, 2-H_b, 7-H), 1.82 – 1.90 (m, 1H, 3-H), 2.89 (dd, *J* = 13.5 Hz, 8.5 Hz, 1H, 6-H_a), 2.98 (dd, *J* = 13.5 Hz, 6.8 Hz, 1H, 6-H_b), 3.26 – 3.36 (m, 1H, 1-H_a), 3.50 (dd, *J* = 11.0 Hz, 4.4 Hz, 1H, 9-H_a), 3.53 (dd, *J* = 11.0 Hz, 6.3 Hz, 1H, 9-H_b), 3.68 – 3.74 (m, 1H, 4-H), 3.96 – 4.06 (m, 1H, 1-H_b), 4.16 – 4.26 (m, 1H, 5-H), 7.07 – 7.13 (m, 3H, 2'-H, 4'-H, 6'-H), 7.13 – 7.20 (m, 2H, 3'-H, 5'-H), 7.50 – 7.56 (m, 1H, 5''-H), 7.56 – 7.64 (m, 2H, 3''-H, 4''-H), 7.83 – 7.91 (m, 1H, 6''-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 16.2 (C-8), 21.7 (C-2), 35.7 (C-6), 37.3 (C-7), 37.8 (C-3), 41.8 (C-1), 62.3 (C-5), 64.5 (C-9), 67.5 (C-4), 124.3 (C-3''), 126.9 (C-4'), 128.6 (C-2', C-6'), 129.0 (C-3', C-5'), 131.0 (C-6''), 131.9 (C-5''), 133.3 (C-4''), 133.9 (C-1''), 137.3 (C-1'), 147.6 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_6S$ ($[M+H]^+$): 435.1584 (435.1585).

GP9: Conversion to epoxides

The reaction was performed according to the literature.[17] A round bottom flask was charged with a solution of the alkene (0.27 mmol) in a mixture of isopropanol and water (20 mL, 4 : 1). *meta*-Chloroperbenzoic acid (71.0 mg, 0.41 mmol) was added and the mixture was stirred for 24 h at room temperature. Isopropanol was evaporated and the remaining mixture was extracted with ethyl acetate (2 × 60 mL). The organic phases were washed with saturated sodium thiosulfate solution (2 × 60 mL), sodium hydroxide solution (2 × 60 mL, 1 M) and brine (1 × 60 mL). They were dried over sodium sulfate and the solvents were evaporated. The crude product was purified by HPLC.

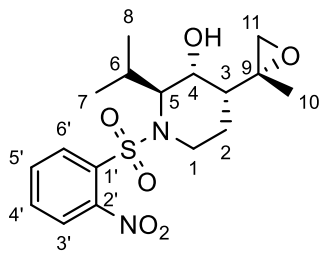
(2*S*,3*R*,4*R*)-2-Isopropyl-4-((*S*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol ((*R*,*R*,*S*)-**35a) and (2*S*,3*R*,4*R*)-2-Isopropyl-4-((*R*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol (**(*R*,*R*,*R*)-**35a)**

According to GP9 (*R*,*R*,*S*)-**35a** was prepared from (2*S*,3*R*,4*S*)-2-isopropyl-1-((2-nitrophenyl)-sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*R*,*S*)-**30a** (0.10 mg, 0.27 mmol) and *m*-chloro-perbenzoic acid (71.0 mg, 0.41 mmol) as a yellow oil (42.0 mg, 0.11 mmol, 41 %). Epoxide (*R*,*R*,*R*)-**35a** was isolated as well as a yellow oil (55.0 mg, 0.14 mmol, 52 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (90 : 10), 15 mL / min, R_t ((*R*,*R*,*S*)-**35**) = 21.5 min, R_t ((*R*,*R*,*R*)-**35a**) = 24.5 min.



(*R*,*R*,*S*)-35a

(*R*,*R*,*S*)-**35a**, $[\alpha]_D^{20}$ + 108 (*c* 1.0 in $CHCl_3$). R_f = 0.29 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3532 (br, w), 2966 (m), 2939 (m), 1541 (vs), 1470 (m), 1440 (m), 1372 (s), 1337 (s), 1281 (m), 1258 (m), 1158 (vs), 1125 (s), 1081 (m), 1062 (m), 1046 (m), 969 (s), 909 (s), 852 (m), 778 (m), 751 (s), 731 (vs), 680 (m), 651 (m), 620 (m), 577 (vs), 559 (s) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 0.78 (d, *J* = 6.7 Hz, 3H, 7-H/8-H), 0.98 (d, *J* = 6.7 Hz, 3H, 8-H/7-H), 1.35 (s, 3H, 10-H) 1.38 – 1.42 (m, 1H, 2-H_a), 1.68 – 1.75 (m, 1H, 3-H), 1.74 (dddd, *J* = 12.7 Hz, 12.7 Hz, 12.7 Hz, 4.6 Hz, 1H, 2-H_b), 1.94 (dqq, *J* = 10.8 Hz, 6.7 Hz, 6.7 Hz, 1H, 6-H), 2.52 (d, *J* = 4.4 Hz, 1H, 11-H_a), 2.71 (d, *J* = 4.4 Hz, 1H, 11-H_b), 2.74 (d, *J* = 3.3 Hz, 1H, OH), 3.02 – 3.10 (m, 1H, 1-H_a), 3.57 (d, *J* = 10.8 Hz, 1H, 5-H), 4.04 – 4.09 (m, 1H, 1-H_b), 4.17 – 4.21 (m, 1H, 4-H), 7.53 – 7.57 (m, 1H, 3'-H), 7.61 – 7.69 (m, 2H, 4'-H, 5'-H), 8.08 – 8.13 (m, 1H, 6'-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 19.9 (C-7/C-8), 20.2 (C-8/C-7), 20.6 (C-10), 21.1 (C-2), 26.4 (C-6), 39.4 (C-3), 41.4 (C-1), 51.9 (C-11), 59.0 (C-9), 66.2 (C-4), 66.8 (C-5), 123.6 (C-3'), 131.3 (C-5'), 131.4 (C-6'), 133.2 (C-4'), 134.7 (C-1'), 147.9 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{24}N_2O_6S$ ($[M+NH_4]^+$): 402.1693 (402.1695).



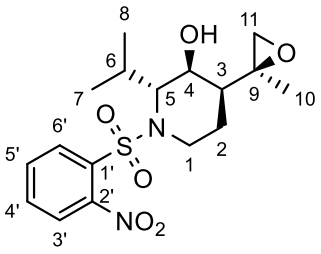
(*R*,*R*,*R*)-35a

(*R*,*R*,*R*)-**35a**, $[\alpha]_D^{20}$ + 94 (*c* 1.0 in $CHCl_3$). R_f = 0.23 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3529 (br, w), 2968 (m), 2933 (m), 2876 (w), 1542 (vs), 1470 (w), 1449 (w), 1372 (s), 1340 (s), 1278 (m), 1159 (vs), 1125 (m), 1079 (m), 1063 (m), 1046 (m), 971 (s), 912 (s), 852 (m), 778 (m), 749 (m), 732 (s), 680 (m), 651 (w), 614 (m), 577 (s), 559 (m) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 0.78 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.98 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.34 (s, 3H, 10-H), 1.47 – 1.53 (m, 1H, 2-H_a), 1.64 (dddd, *J* = 13.2 Hz, 13.2 Hz,

13.2 Hz, 4.7 Hz, 1H, 2-H_b), 1.86 – 1.91 (m, 1H, 3-H), 1.94 (dqq, *J* = 10.8 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 2.57 (d, *J* = 4.3 Hz, 1H, 11-H_a), 2.82 (d, *J* = 2.6 Hz, 1H, OH), 2.85 (d, *J* = 4.3 Hz, 1H, 11-H_b), 3.05 (ddd, *J* = 13.2 Hz, 13.2 Hz, 2.5 Hz, 1H, 1-H_a), 3.55 (d, *J* = 10.8 Hz, 1H, 5-H), 4.03 – 4.10 (m, 2H, 1-H_b, 4-H), 7.50 – 7.57 (m, 1H, 3'-H), 7.60 – 7.70 (m, 2H, 4'-H, 5'-H), 8.06 – 8.12 (m, 1H, 6'-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 19.8 (C-7/C-8), 20.3 (C-8/C-7), 20.4 (C-2), 20.7 (C-10), 26.4 (C-6), 38.5 (C-3), 41.6 (C-1), 52.0 (C-11), 58.7 (C-9), 65.6 (C-4), 66.7 (C-5), 123.6 (C-3'), 131.3 (C-5'), 131.3 (C-6'), 133.2 (C-4'), 134.7 (C-1'), 147.9 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{24}N_2O_6S$ ($[M+NH_4]^+$): 402.1693 (402.1693).

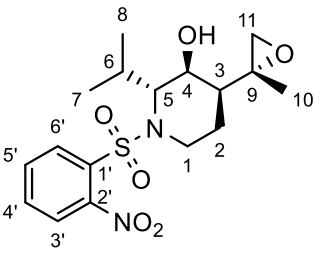
(2*R*,3*S*,4*S*)-2-Isopropyl-4-((*S*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol ((*S*,*S*,*S*)-**35b) and (2*R*,3*S*,4*S*)-2-Isopropyl-4-((*R*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol (**(*S*,*S*,*R*)-**35b)**

According to GP9 (*S*,*S*,*S*)-**35b** was prepared from (2*R*,3*S*,4*R*)-2-isopropyl-1-((2-nitrophenyl)-sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*S*,*R*)-**30b** (0.22 g, 0.60 mmol) and *m*-chloroperbenzoic acid (0.16 g, 0.90 mmol) as a colorless solid (0.11 g, 0.28 mmol, 47 %). Epoxide (*S*,*S*,*R*)-**35b** was isolated as well as colorless solid (51.0 mg, 0.14 mmol, 23 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (95 : 5), 15 mL / min, R_t ((*S*,*S*,*S*)-**35b**) = 38 min, R_t ((*S*,*S*,*R*)-**35b**) = 47.5 min).



(*S*,*S*,*S*)-35b

(*S*,*S*,*S*)-**35b**, mp 120 °C. $[\alpha]_D^{20}$ - 103 (*c* 1.0 in $CHCl_3$). R_f = 0.16 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3524 (br, w), 3097 (w), 2968 (m), 2933 (w), 1591 (w), 1543 (vs), 1470 (w), 1440 (w), 1373 (s), 1339 (s), 1281 (w), 1206 (w), 1160 (s), 1126 (m), 1082 (m), 1062 (m), 1046 (w), 970 (m), 911 (m), 852 (m), 816 (w), 779 (w), 751 (m), 733 (m), 680 (w), 652 (w), 620 (w), 578 (s), 560 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.78 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.98 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.35 (s, 3H, 10-H), 1.36 – 1.43 (m, 1H, 2-H_a), 1.67 – 1.75 (m, 1H, 3-H), 1.75 (dddd, *J* = 13.0 Hz, 13.0 Hz, 13.0 Hz, 4.9 Hz, 1H, 2-H_b), 1.94 (dqq, *J* = 11.2 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 2.53 (d, *J* = 4.5 Hz, 1H, 11-H_a), 2.71 (d, *J* = 4.5 Hz, 1H, 11-H_b), 2.74 (d, *J* = 3.1 Hz, 1H, OH), 2.99 – 3.13 (m, 1H, 1-H_a), 3.53 – 3.61 (m, 1H, 5-H), 4.02 – 4.10 (m, 1H, 1-H_b), 4.17 – 4.24 (m, 1H, 4-H), 7.51 – 7.58 (m, 1H, 3'-H), 7.60 – 7.69 (m, 2H, 4'-H, 5'-H), 8.06 – 8.14 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 19.9 (C-7/C-8), 20.2 (C-8/C-7), 20.6 (C-10), 21.1 (C-2), 26.4 (C-6), 39.4 (C-3), 41.5 (C-1), 51.9 (C-11), 59.0 (C-9), 66.2 (C-4), 66.8 (C-5), 123.7 (C-3'), 131.3 (C-6'), 131.4 (C-5'), 133.2 (C-4'), 134.7 (C-1'), 147.9 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{24}N_2O_6S$ ($[M+Na]^+$): 407.1247 (407.1249).

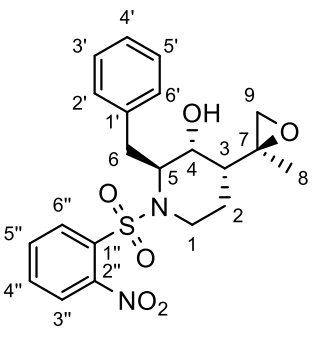


(***S*,*S*,*R*)-35b**

(*S*,*S*,*R*)-**35b**, mp 49 °C. $[\alpha]_D^{20}$ - 81 (*c* 1.0 in $CHCl_3$). R_f = 0.14 (hexanes/EtOAc, 2 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3527 (br, w), 2967 (m), 2934 (w), 2877 (w), 1591 (w), 1542 (vs), 1470 (w), 1440 (w), 1372 (s), 1339 (s), 1277 (m), 1210 (w), 1159 (vs), 1125 (m), 1079 (m), 1063 (m), 1046 (m), 971 (s), 911 (s), 852 (m), 816 (w), 778 (m), 749 (s), 731 (s), 680 (m), 651 (m), 613 (m), 577 (s), 559 (s) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.78 (d, *J* = 6.5 Hz, 3H, 7-H/8-H), 0.98 (d, *J* = 6.5 Hz, 3H, 8-H/7-H), 1.34 (s, 3H, 10-H), 1.45 – 1.53 (m, 1H, 2-H_a), 1.64 (dddd, *J* = 13.1 Hz, 13.1 Hz, 13.1 Hz, 5.7 Hz, 1H, 2-H_b), 1.85 – 1.92 (m, 1H, 3-H), 1.94 (dqq, *J* = 10.9 Hz, 6.5 Hz, 6.5 Hz, 1H, 6-H), 2.56 (d, *J* = 4.1 Hz, 1H, 11-H_a), 2.83 (d, *J* = 3.1 Hz, 1H, OH), 2.85 (d, *J* = 4.1 Hz, 1H, 11-H_b), 3.05 (ddd, *J* = 15.4 Hz, 13.1 Hz, 2.8 Hz, 1H, 1-H_a), 3.52 – 3.59 (m, 1H, 5-H), 4.03 – 4.10 (m, 2H, 1-H_b, 4-H), 7.50 – 7.57 (m, 1H, 3'-H), 7.59 – 7.67 (m, 2H, 4'-H, 5'-H), 8.06 – 8.15 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 19.8 (C-7/C-8), 20.4 (C-8/C-7), 20.4 (C-10), 20.7 (C-2), 26.4 (C-6), 38.5 (C-3), 41.6 (C-1), 52.0 (C-11), 58.7 (C-9), 65.6 (C-4), 66.7 (C-5), 123.6 (C-3'), 131.3 (C-6'), 131.3 (C-5'), 133.2 (C-4'), 134.6 (C-1'), 147.9 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{24}N_2O_6S$ ($[M+NH_4]^+$): 402.1693 (402.1697).

(2*S*,3*R*,4*R*)-2-Benzyl-4-((*S*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol ((*R*,*R*,*S*)-35f) and (2*S*,3*R*,4*R*)-2-Benzyl-4-((*R*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)sulfonyl)-piperidin-3-ol ((*R*,*R*,*R*)-35f)

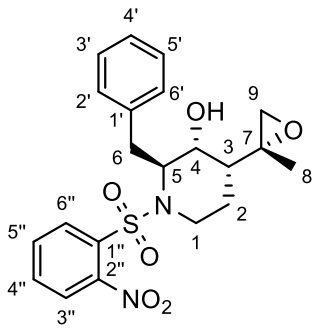
According to GP9 (*R*,*R*,*S*)-**35f** was prepared from (2*S*,3*R*,4*S*)-2-benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*R*,*S*)-**30f** (0.21 g, 0.51 mmol) and *m*-chlorperbenzoic acid (0.13 mg, 0.77 mmol) as a colorless solid (0.13 g, 0.29 mmol, 58 %). Epoxide (*R*,*R*,*R*)-**35f** was also isolated as a colorless solid (0.09 g, 0.20 mmol, 39 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (90 : 10), 15 mL / min, R_t ((*R*,*R*,*S*)-**35f**) = 21 min, R_t ((*R*,*R*,*R*)-**35f**) = 24 min.



(***R*,*R*,*S*)-35f**

(*R*,*R*,*S*)-**35f**, mp 173 °C. $[\alpha]_D^{20}$ + 40 (*c* 0.1 in $CHCl_3$). R_f = 0.57 (hexanes/EtOAc, 1 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3546 (w), 2957 (m), 2925 (s), 2854 (m), 1726 (m), 1543 (vs), 1495 (w), 1455 (m), 1374 (s), 1339 (s), 1269 (m), 1162 (s), 1127 (m), 1078 (m), 990 (w), 965 (w), 852 (w), 745 (m), 732 (m), 702 (m), 678 (w), 608 (w), 571 (m) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 1.33 (s, 3H, 8-H), 1.51 – 1.54 (m, 1H, 2-H_a), 1.86 –1.95 (m, 2H, 2-H_b, 3-H), 2.54 (dd, *J* = 4.3 Hz, 1H, 9-H_a), 2.56 (d, *J* = 4.4 Hz, 1H, OH), 2.71 (d, *J* = 4.3 Hz, 1H, 9-H_b), 2.85

(dd, *J* = 13.7 Hz, 9.7 Hz, 1H, 6-H_a), 3.01 (dd, *J* = 13.7 Hz, 6.3 Hz, 1H, 6-H_b), 3.27 – 3.34 (m, 1H, 1-H_a), 3.85 – 3.88 (m, 1H, 4-H), 4.01 – 4.05 (m, 1H, 1-H_b), 4.18 – 4.23 (m, 1H, 5-H), 7.12 – 7.18 (m, 3H, 2'-H, 4'-H, 6'-H), 7.19 – 7.23 (m, 2H, 3'-H, 5'-H), 7.55 – 7.59 (m, 1H, 5''-H), 7.60 – 7.65 (m, 2H, 3''-H, 4''-H), 7.95 – 7.99 (m, 1H, 6''-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 20.5 (C-8), 21.3 (C-2), 35.6 (C-6), 39.4 (C-3), 41.2 (C-1), 51.9 (C-9), 59.0 (C-7), 61.0 (C-5), 66.7 (C-4), 124.3 (C-3''), 127.0 (C-4'), 128.8 (C-3', C-5'), 128.9 (C-2', C-6'), 131.2 (C-6''), 131.8 (C-5''), 133.3 (C-4''), 134.0 (C-1''), 137.0 (C-1'), 147.7 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{24}N_2O_6S$ ([M+Na]$^+$): 455.1247 (455.1248).



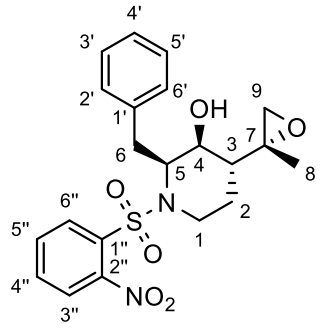
(*R*,*R*,*R*)-35f

(*R*,*R*,*R*)-**35f**, mp 120 °C. $[\alpha]_D^{20}$ + 30 (*c* 0.1 in $CHCl_3$). R_f = 0.51 (hexanes/EtOAc, 1 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3465 (w), 3026 (w), 2926 (m), 2855 (w), 1722 (w), 1592 (w), 1542 (vs), 1495 (w), 1455 (m), 1372 (s), 1338 (s), 1271 (m), 1161 (vs), 1127 (m), 1078 (m), 1066 (m), 989 (m), 968 (m), 947 (m), 928 (m), 909 (m), 874 (w), 852 (m), 781 (w), 761 (m), 733 (s), 702 (m), 679 (m), 651 (w), 607 (m), 588 (m), 572 (m), 546 (w), 518 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.35 (s, 3H, 8-H), 1.58 – 1.65 (m, 1H, 2-H_a), 1.77 (dddd, *J* = 13.3 Hz, 4.7 Hz, 1H, 2-H_b), 2.08 – 2.14 (m, 1H, 3-H), 2.56 (d, *J* = 4.3 Hz, 1H, 9-H_a), 2.60 (d, *J* = 4.3 Hz, 1H, OH), 2.83 (d, *J* = 4.3 Hz, 9-H_b), 2.87 (dd, *J* = 13.8 Hz, 9.2 Hz, 1H, 6-H_a), 2.99 (dd, *J* = 13.8 Hz, 6.5 Hz, 1H, 6-H_b), 3.25 – 3.34 (m, 1H, 1-H_a), 3.76 – 3.82 (m, 1H, 4-H), 3.99 – 4.07 (m, 1H, 1-H_b), 4.16 – 4.23 (m, 1H, 5-H), 7.10 – 7.17 (m, 3H, 2'-H, 4'-H, 6'-H), 7.17 – 7.24 (m, 2H, 3'-H, 5'-H), 7.52 – 7.58 (m, 1H, 5''-H), 7.58 – 7.66 (m, 2H, 3''-H, 4''-H), 7.90 – 7.95 (m, 1H, 6''-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 20.5 (C-8), 20.8 (C-2), 35.7 (C-6), 38.5 (C-3), 41.4 (C-1), 51.8 (C-9), 58.3 (C-7), 62.0 (C-5), 66.1 (C-4), 124.2 (C-3''), 127.0 (C-4'), 128.7 (C-3', C-5'), 129.0 (C-2', C-6'), 131.1 (C-6''), 131.8 (C-5''), 133.3 (C-4''), 134.0 (C-1''), 137.1 (C-1'), 147.8 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{24}N_2O_6S$ ([M+H]$^+$): 433.1428 (433.1438).

(2*S*,3*S*,4*R*)-2-Benzyl-4-((*R*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol ((*S*,*R*,*R*)-**35f) and (2*S*,3*S*,4*R*)-2-Benzyl-4-((*S*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)-sulfonyl)piperidin-3-ol (**(*S*,*R*,*S*)-**35f)**

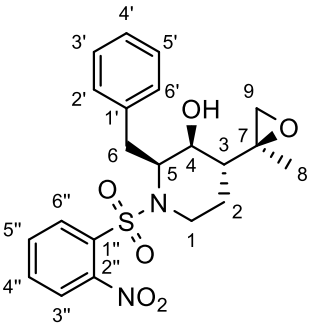
According to GP9 (*S*,*R*,*R*)-**35f** was prepared from (2*S*,3*S*,4*S*)-2-benzyl-1-((2-nitrophenyl)sulfonyl)-4-(prop-1-en-2-yl)-piperidin-3-ol (*S*,*S*)-**30f** (55.0 mg, 0.13 mmol) and *m*-chloroperbenzoic acid (34.0 mg, 0.20 mmol) as a colorless solid (25.0 mg, 58.0 mmol, 44 %). Epoxide (*S*,*R*,*S*)-**35f** was isolated as well as a colorless solid (20.0 mg, 46.0 mmol, 35 %).

Purification was accomplished by HPLC (Orbit, hexanes/EtOAc (60 : 40), 15 mL / min, R_t ((*S*,*R*,*R*)-**35f**) = 24.5 min, R_t ((*S*,*R*,*S*)-**35f**) = 27 min.



(*S*,*R*,*R*)-35f

(*S*,*R*,*R*)-**35f**, mp 58 °C. $[\alpha]_D^{20}$ = + 203 (*c* = 1.0 in $CHCl_3$). R_f = 0.43 (hexanes/EtOAc, 1 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3448 (br, w), 3026 (w), 2928 (w), 1592 (w), 1541 (vs), 1497 (w), 1454 (w), 1370 (s), 1333 (s), 1297 (m), 1256 (w), 1205 (w), 1159 (s), 1126 (m), 1086 (m), 1058 (m), 1013 (m), 957 (m), 943 (m), 911 (w), 853 (m), 832 (w), 811 (w), 779 (w), 756 (s), 740 (s), 700 (m), 652 (w), 592 (s), 573 (m), 512 (w) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 1.42 (s, 3H, 8-H), 1.52 (dddd, *J* = 13.3 Hz, 13.3 Hz, 13.3 Hz, 4.9 Hz, 1H, 2-H_a), 1.93 – 1.99 (m, 1H, 2-H_b), 2.08 (ddd, *J* = 13.3 Hz, 11.0 Hz, 4.0 Hz, 1H, 3-H), 2.71 (d, *J* = 3.7 Hz, 1H, 9-H_a), 2.72 (dd, *J* = 14.4 Hz, 11.7 Hz, 1H, 6-H_a), 3.05 (d, *J* = 3.7 Hz, 1H, 9-H_b), 3.13 (dd, *J* = 14.4 Hz, 3.2 Hz, 1H, 6-H_b), 3.36 (ddd, *J* = 14.6 Hz, 13.3 Hz, 2.8 Hz, 1H, 1-H_a), 3.73 (s, 1H, OH), 3.75 (dd, *J* = 11.0 Hz, 5.7 Hz, 1H, 4-H), 4.02 – 4.08 (m, 1H, 1-H_b), 4.24 (ddd, *J* = 11.7 Hz, 5.7 Hz, 3.2 Hz, 1H, 5-H), 6.82 – 6.90 (m, 3H, 3'-H, 4'-H, 5'-H), 6.95 – 7.03 (m, 2H, 2'-H, 6'-H), 7.24 – 7.29 (m, 1H, 5"-H), 7.35 – 7.40 (m, 1H, 4"-H), 7.42 – 7.50 (m, 2H, 3"-H, 6"-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 20.9 (C-8), 29.0 (C-2), 29.8 (C-6), 40.1 (C-1), 41.3 (C-3), 51.7 (C-9), 60.1 (C-7), 60.4 (C-5), 70.0 (C-4), 124.3 (C-3"), 126.3 (C-4'), 128.0 (C-3', C-5'), 129.2 (C-2', C-6'), 130.7 (C-4"), 131.9 (C-5"), 132.7 (C-6"), 133.9 (C-1"), 138.3 (C-1'), 147.0 (C-2"). HRMS (ESI): calc (found) for $C_{21}H_{24}N_2O_6S$ ($[M+Na]^+$): 455.1247 (455.1244).



(*S*,*R*,*S*)-35f

(*S*,*R*,*S*)-**35f**, mp 142 °C. $[\alpha]_D^{20}$ + 220 (*c* 1.0 in $CHCl_3$). R_f = 0.31 (hexanes/EtOAc, 1 : 1, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3481 (br, w), 3089 (w), 3028 (w), 2925 (w), 1591 (w), 1541 (vs), 1497 (w), 1441 (w), 1370 (s), 1333 (s), 1294 (w), 1275 (w), 1250 (w), 1159 (s), 1126 (m), 1090 (m), 1073 (m), 1013 (w), 957 (m), 924 (m), 91 (m), 866 (w), 852 (m), 833 (w), 807 (w), 779 (w), 756 (s), 740 (s), 700 (m), 651 (w), 592 (s), 575 (m), 539 (w), 501 (w), 443 (w) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 1.36 (s, 3H, 8-H), 1.46 (ddd, *J* = 13.1 Hz, 11.2 Hz, 3.9 Hz, 1H, 3-H), 1.65 (dddd, *J* = 13.1 Hz, 13.1 Hz, 13.1 Hz, 4.9 Hz, 1H, 2-H_a), 1.77 – 1.83 (m, 1H, 2-H_b), 2.60 (d, *J* = 4.4 Hz, 1H, 9-H_a), 2.65 (dd, *J* = 14.1 Hz, 11.5 Hz, 1H, 6-H_a), 2.66 (d, *J* = 4.4 Hz, 1H, 9-H_b), 3.12 (dd, *J* = 14.1 Hz, 3.0 Hz, 1H, 6-H_b), 3.12 (s, br, 1H, OH), 3.29 – 3.36 (m, 1H, 1-H_a), 4.02 – 4.10 (m, 1H, 1-H_b), 4.19 (ddd, *J* = 11.2 Hz, 5.5 Hz, 2.3 Hz, 1H, 4-H), 4.32 (ddd, *J* = 11.5 Hz, 5.5 Hz, 3.0 Hz, 1H, 5-H), 6.81 – 6.89 (m, 3H, 3'-H, 4'-H, 5'-H), 6.94 – 7.00 (m,

2H, 2'-H, 6'-H), 7.25 – 7.30 (m, 1H, 5''-H), 7.37 – 7.42 (m, 1H, 4''-H), 7.42 – 7.49 (m, 2H, 3''-H, 6''-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 16.3 (C-8), 28.8 (C-2), 29.6 (C-6), 40.0 (C-1), 44.2 (C-3), 52.6 (C-9), 58.5 (C-7), 60.8 (C-5), 71.1 (C-4), 124.3 (C-3''), 126.3 (C-4'), 128.0 (C-3', C-5'), 129.2 (C-2', C-6'), 130.8 (C-4''), 132.0 (C-5''), 132.7 (C-6''), 134.0 (C-1''), 138.2 (C-1'), 146.9 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{24}N_2O_6S$ ($[M+Na]^+$): 455.1247 (455.1245).

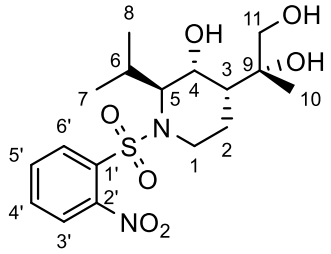
GP10: Conversion to triols

The reaction was performed according to the literature.[17] A round bottom flask was charged with a solution of the alkene (0.09 mmol) in a mixture of tetrahydrofuran and water (8 mL, 4 : 1) under nitrogen atmosphere. Upon cooling to 0 °C perchloric acid solution (1.25 mL, 7 %) was added and the mixture was stirred for 24 h at room temperature. Afterwards the mixture was neutralized using sodium hydroxide solution (1 M) and extracted with ethyl acetate (3 × 14 mL). The organic phases were washed with brine (2 × 9 mL) and dried over sodium sulfate. The solvents were evaporated and the crude product was purified by HPLC.

(*R*)-2-((2*S*,3*R*,4*R*)-3-Hydroxy-2-isopropyl-1-((2-nitrophenyl)sulfonyl)piperidin-4-yl)-propane-1,2-diol ((*R*,*R*,*R*)-36a) und (*S*)-2-((2*S*,3*R*,4*R*)-3-Hydroxy-2-isopropyl-1-((2-nitrophenyl)-sulfonyl)piperidin-4-yl)propane-1,2-diol ((*R*,*R*,*S*)-36a)

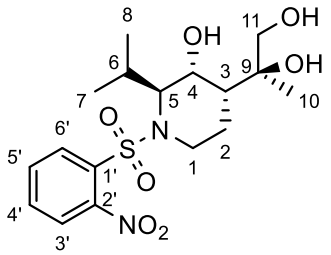
According to GP10 (*R*,*R*,*R*)-**36a** was prepared from (2*S*,3*R*,4*R*)-2-isopropyl-4-((*S*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)-sulfonyl)piperidin-3-ol (*R*,*R*,*S*)-**35a** (33.0 mg, 90.0 µmol) and perchloric acid (1.3 mL, 1.54 mmol, 7 % in water) as a colorless solid (1.00 mg, 2.48 µmol, 3 %). Triol (*R*,*R*,*S*)-**36a** was isolated as well as a colorless solid (15.0 mg, 37.0 µmol, 41 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (85 : 15), 15 mL / min, R_t ((*R*,*R*,*R*)-**36a**) = 47 min, R_t ((*R*,*R*,*S*)-**36a**) = 51 min).

Both triols were also obtained according to GP9 starting from (2*S*,3*R*,4*R*)-2-isopropyl-4-((*R*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)-sulfonyl)piperidin-3-ol (*R*,*R*,*R*)-**35a** (49.0 mg, 0.13 mmol) and perchloric acid (1.8 mL, 2.22 mmol, 7 % in water): Triol (*R*,*R*,*R*)-**36a** (11.0 mg, 27.0 µmol, 21 %), triol (*R*,*R*,*S*)-**36a** (2.00 mg, 4.97 µmol, 4 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (85 : 15), 15 mL / min).



(*R*,*R*,*R*)-36a

(*R*,*R*,*R*)-**36a**, mp 50 °C. $[\alpha]_D^{20}$ + 141 (*c* 1.0 in $CHCl_3$). R_f = 0.12 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3382 (br, w), 2967 (m), 2933 (m), 1542 (vs), 1469 (m), 1440 (m), 1371 (s), 1332 (s), 1279 (m), 1158 (vs), 1125 (s), 1085 (m), 1064 (s), 1045 (s), 997 (w), 970 (s), 911 (s), 852 (m), 806 (w), 776 (m), 751 (s), 732 (vs), 688 (w), 652 (m), 580 (s), 560 (m) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 0.65 (d, *J* = 6.7 Hz, 3H, 7-H/8-H), 0.97 (d, *J* = 6.7 Hz, 3H, 8-H/7-H), 1.22 (s, 3H, 10-H), 1.45 – 1.50 (m, 1H, 2-H_a), 1.62 (s, br, 1H, OH), 1.67 – 1.72 (m, 1H, 3-H), 1.82 (dddd, *J* = 13.0 Hz, 13.0 Hz, 13.0 Hz, 4.9 Hz, 1H, 2-H_b), 1.99 (dqq, *J* = 10.8 Hz, 6.7 Hz, 6.7 Hz, 1H, 6-H), 3.08 – 3.15 (m, 1H, 1-H_a), 3.26 (d, *J* = 11.5 Hz, 1H, 11-H_a), 3.36 (s, br, 1H, OH), 3.51 (d, *J* = 10.8 Hz, 1H, 5-H), 3.55 (d, *J* = 11.5 Hz, 1H, 11-H_b), 4.08 – 4.15 (m, 1H, 1-H_b), 4.37 (s, 1H, 4-H), 7.56 – 7.63 (m, 1H, 3'-H), 7.64 – 7.73 (m, 2H, 4'-H, 5'-H), 8.04 – 8.12 (m, 1H, 6'-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 19.7 (C-7/C-8), 20.1 (C-8/C-7), 20.9 (C-2), 24.8 (C-10), 26.7 (C-6), 41.9 (C-1), 42.1 (C-3), 65.2 (C-4), 66.5 (C-11), 67.0 (C-5), 73.0 (C-9), 124.0 (C-3'), 130.8 (C-6'), 131.8 (C-5'), 133.5 (C-4'), 134.5 (C-1') 147.6 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_7S$ ($[M+H]^+$): 403.1533 (403.1543).

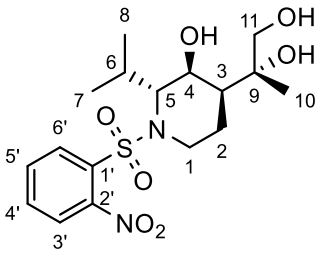


(*R*,*R*,*S*)-36a

(*R*,*R*,*S*)-**36a**, mp 50 °C. $[\alpha]_D^{20}$ = + 132 (*c* = 1.0 in $CHCl_3$). R_f = 0.09 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3382 (br, w), 2967 (m), 2929 (m), 1542 (vs), 1466 (m), 1440 (m), 1371 (s), 1334 (s), 1277 (m), 1156 (s), 1124 (s), 1092 (m), 1063 (m), 1047 (s), 995 (w), 973 (s), 910 (vs), 853 (m), 807 (w), 776 (m), 751 (s), 731 (vs), 685 (m), 652 (m), 579 (s), 560 (s) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 0.69 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.97 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.17 (s, 3H, 10-H), 1.52 – 1.59 (m, 1H, 2-H_a), 1.71 – 1.78 (m, 1H, 3-H), 1.92 (dddd, *J* = 13.4 Hz, 13.4 Hz, 13.4 Hz, 4.9 Hz, 1H, 2-H_b), 1.98 (dqq, *J* = 10.7 Hz, 10.7 Hz, 6.6 Hz, 1H, 6-H), 2.07 (s, br, 1H, OH), 3.08 – 3.18 (m, 1H, 1-H_a), 3.34 (s, br, 2H, 2xOH), 3.47 (d, *J* = 10.7 Hz, 1H, 5-H), 3.57 (s, 2H, 11-H), 4.11 – 4.18 (m, 1H, 1-H_b), 4.40 (s, 1H, 4-H), 7.53 – 7.62 (m, 1H, 3'-H), 7.62 – 7.73 (m, 2H, 4'-H, 5'-H), 8.03 – 8.10 (m, 1H, 6'-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 19.5 (C-2), 19.7 (C-7/C-8), 20.2 (C-8/C-7), 23.1 (C-10), 26.5 (C-6), 38.9 (C-3), 42.0 (C-1), 66.2 (C-4), 66.8 (C-5), 68.8 (C-11), 74.0 (C-9), 123.9 (C-3'), 130.7 (C-6'), 131.7 (C-5'), 133.4 (C-4'), 134.6 (C-1'), 147.6 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_7S$ ($[M+H]^+$): 403.1533 (403.1535).

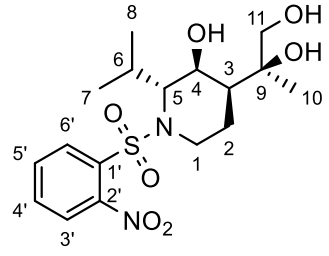
(*R*)-2-((2*R*,3*S*,4*S*)-3-Hydroxy-2-isopropyl-1-((2-nitrophenyl)sulfonyl)piperidin-4-yl)-propan-1,2-diol ((*S*,*S*,*R*)-36b) and (*S*)-2-((2*R*,3*S*,4*S*)-3-Hydroxy-2-isopropyl-1-((2-nitrophenyl)sulfonyl)piperidin-4-yl)propan-1,2-diol ((*S*,*S*,*S*)-36b)

According to GP10 (*S*,*S*,*R*)-**36b** was prepared from (2*R*,3*S*,4*S*)-2-isopropyl-4-((*S*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)sulfonyl)piperidin-3-ol (*S*,*S*,*S*)-**35b** (0.11 g, 0.28 mmol) and perchloric acid (4.0 mL, 4.93 mmol, 7 % in water) as a colorless solid (7.00 mg, 17.4 µmol, 6 %). Triol (*S*,*S*,*S*)-**36b** was isolated as well as a colorless solid (0.05 g, 124 µmol, 44 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (90 : 10), 15 mL / min, R_t ((*S*,*S*,*R*)-**36b**) = 109 min, R_t ((*S*,*S*,*S*)-**36b**) = 113 min).



(*S*,*S*,*R*)-**36b**

(*S*,*S*,*R*)-**36b**, mp 51 °C. $[\alpha]_D^{20}$ - 121 (*c* 0.4 in $CHCl_3$). R_f = 0.09 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3372 (br, w), 2967 (m), 2932 (m), 1591 (w), 1543 (vs), 1469 (m), 1440 (m), 1372 (s), 1334 (s), 1279 (m), 1159 (s), 1125 (s), 1085 (m), 1064 (m), 1046 (s), 997 (w), 971 (s), 912 (s), 853 (m), 806 (w), 777 (w), 751 (s), 733 (vs), 688 (w), 652 (m), 581 (s), 560 (m) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.65 (d, *J* = 6.6 Hz, 3H, 7-H/8-H), 0.97 (d, *J* = 6.6 Hz, 3H, 8-H/7-H), 1.23 (s, 3H, 10-H), 1.45 – 1.51 (m, 1H, 2-H_a), 1.67 – 1.73 (m, 1H, 3-H), 1.82 (dddd, *J* = 13.2 Hz, 13.2 Hz, 13.2 Hz, 4.6 Hz, 1H, 2-H_b), 1.99 (dqq, *J* = 11.2 Hz, 6.6 Hz, 6.6 Hz, 1H, 6-H), 3.07 – 3.16 (m, 1H, 1-H_a), 3.26 (d, *J* = 11.3 Hz, 1H, 11-H_a), 3.36 (s, br, 1H, OH), 3.48 – 3.53 (m, 1H, 5-H), 3.56 (d, *J* = 11.3 Hz, 1H, 11-H_b), 4.07 – 4.16 (m, 1H, 1-H_b), 4.34 – 4.40 (m, 1H, 4-H), 7.57 – 7.62 (m, 1H, 3'-H), 7.65 – 7.71 (m, 2H, 4'-H, 5'H), 8.04 – 8.11 (m, 1H, 6'-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 19.7 (C-7/C-8), 20.1 (C-8/C-7), 20.9 (C-2), 24.8 (C-10), 26.7 (C-6), 41.9 (C-1), 42.1 (C-3), 65.2 (C-4), 66.5 (C-11), 67.0 (C-5), 73.0 (C-9), 124.0 (C-3'), 130.8 (C-6'), 131.8 (C-5'), 133.5 (C-4'), 134.5 (C-1'), 147.6 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_7S$ ($[M+H]^+$): 403.1533 (403.1535).



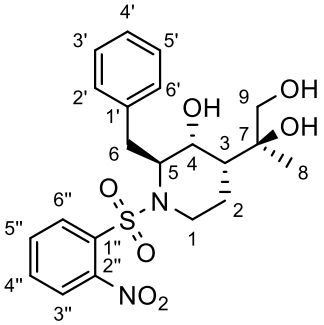
(*S*,*S*,*S*)-**36b**

(*S*,*S*,*S*)-**36b**, mp 51 °C, $[\alpha]_D^{20}$ - 107 (*c* 0.5 in $CHCl_3$). R_f = 0.06 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3388 (br, m), 2967 (m), 2935 (m), 2877 (w), 1591 (w), 1542 (vs), 1469 (m), 1440 (m), 1372 (s), 1335 (s), 1278 (m), 1157 (s), 1124 (s), 1092 (m), 1063 (m), 1047 (s), 973 (s), 912 (s), 853 (m), 807 (w), 777 (w), 751 (s), 733 (vs), 686 (w), 652 (m), 580 (s), 560 (m) cm^{-1}. ^{1}H-NMR (700 MHz, $CDCl_3$) δ 0.68 (d, *J* = 6.7 Hz, 3H, 7-H/8-H), 0.96 (d, *J* = 6.7 Hz, 3H, 8-H/7-H), 1.16 (s, 3H, 10-H), 1.52 – 1.62 (m, 1H, 2-H_a), 1.71 – 1.78 (m, 1H, 3-H), 1.91 (dddd,

J = 13.2 Hz, 13.2 Hz, 13.2 Hz, 4.8 Hz, 1H, 2-H_b), 1.97 (dqq, *J* = 10.8 Hz, 6.7 Hz, 6.7 Hz, 1H, 6-H), 2.22 (s, br, 1H, OH), 3.08 – 3.18 (m, 1H, 1-H_a), 3.34 – 3.44 (m, 2H, 2xOH), 3.44 – 3.50 (m, 1H, 5-H), 3.56 (s, 2H, 11-H), 4.08 – 4.18 (m, 1H, 1-H_b), 4.39 (s, 1H, 4-H), 7.52 – 7.62 (m, 1H, 3'-H), 7.62 – 7.71 (m, 2H, 4'-H, 5'-H), 8.04 – 8.15 (m, 1H, 6'-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 19.5 (C-2), 19.8 (C-7/C-8), 20.1 (C-8/C-7), 23.1 (C-10), 26.5 (C-6), 38.9 (C-3), 42.0 (C-1), 66.2 (C-4), 66.8 (C-5), 68.7 (C-11), 74.1 (C-9), 123.9 (C-3'), 130.8 (C-6'), 131.8 (C-5'), 133.4 (C-4'), 134.6 (C-1'), 147.6 (C-2'). HRMS (ESI): calc (found) for $C_{17}H_{26}N_2O_7S$ ($[M+H]^+$): 403.1533 (403.1537).

(*S*)-2-((2*S*,3*R*,4*R*)-2-Benzyl-3-hydroxy-1-((2-nitrophenyl)sulfonyl)piperidin-4-yl)-propan-1,2-diol ((*R*,*R*,*S*)-**36f) and (*R*)-2-((2*S*,3*R*,4*R*)-2-Benzyl-3-hydroxy-1-((2-nitrophenyl)-sulfonyl)piperidin-4-yl)propan-1,2-diol (**(*R*,*R*,*R*)-**36f)**

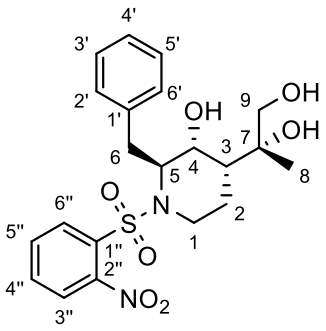
According to GP10 (*R*,*R*,*S*)-**36f** was prepared from a mixture of (2*S*,3*R*,4*R*)-2-benzyl-4-((*S*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)-sulfonyl)-piperidin-3-ol (*R*,*R*,*S*)-**35f** and (2*S*,3*R*,4*R*)-2-benzyl-4-((*R*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)-sulfonyl)-piperidin-3-ol (*R*,*R*,*R*)-**35f** (0.15 g, 0.36 mmol) and perchloric acid (5.0 mL, 6.17 mmol, 7 % in water) as a colorless solid (64.0 mg, 0.06 mmol, 39 %). Triol (*R*,*R*,*R*)-**36f** was isolated as well as a colorless solid (64.0 mg, 0.06 mmol, 39 %). Purification was accomplished by HPLC (OD, *n*-hexane/ isopropanol (90 : 10), 15 mL / min, R_t ((*R*,*R*,*S*)-**36f**) = 120 min, R_t ((*R*,*R*,*R*)-**36f**) = 136 min.



(*R*,*R*,*S*)-**36f**

(*R*,*R*,*S*)-**36f**, mp 60 °C. $[\alpha]_D^{20}$ + 30 (*c* 0.2 in $CHCl_3$). R_f = 0.24 (hexanes/EtOAc, 1 : 2, anisaldehyde solution), FT-IR (ATR): $\tilde{\nu}$ = 3364 (w, br), 2922 (vs), 2852 (s), 1736 (w), 1543 (s), 1465 (m), 1426 (w), 1373 (m), 1338 (m), 1161 (m), 1125 (m), 1045 (w), 995 (w), 932 (w), 852 (w), 732 (w), 652 (w), 573 (w), 418 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.23 (s, 3H, 8-H), 1.60 – 1.66 (m, 1H, 2-H_a), 1.87 – 2.01 (m, 2H, 2-H_b, 3-H), 2.86 (dd, *J* = 13.8 Hz, 8.4 Hz, 1H, 6-H_a), 2.97 (dd, *J* = 13.8 Hz, 7.2 Hz, 1H, 6-H_b), 3.12 – 3.29 (m, br, 1H, OH), 3.32 (d, *J* = 11.2 Hz, 1H, 9-H_a), 3.33 – 3.41 (m, 1H, 1-H_a), 3.61 (d, *J* = 11.2 Hz, 1H, 9-H_b), 4.04 – 4.12 (m, 2H, 1-H_b, 4-H), 4.16 – 4.21 (m, 1H, 5-H), 7.06 – 7.11 (m, 3H, 2'-H, 4'-H, 6'-H), 7.11 – 7.16 (m, 2H, 3'-H, 5'-H), 7.53 – 7.58 (m, 1H, 5''-H), 7.59 – 7.65 (m, 2H, 3''-H, 4''-H), 7.85 – 7.90 (m, 1H, 6''-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 21.0 (C-2), 24.6 (C-8), 35.6 (C-6), 41.6 (C-3), 41.7 (C-1), 62.6 (C-5), 66.2 (C-4), 66.7 (C-9), 73.1 (C-7), 124.5 (C-3''), 127.0 (C-4'), 128.6 (C-3', C-5'), 129.0 (C-2', C-6'), 131.0 (C-6''), 132.0 (C-5''),

133.4 (C-4"), 133.8 (C-1"), 136.9 (C-1'), 147.5 (C-2"). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_7S$ ([M+Na]$^+$): 473.1353 (473.1352).

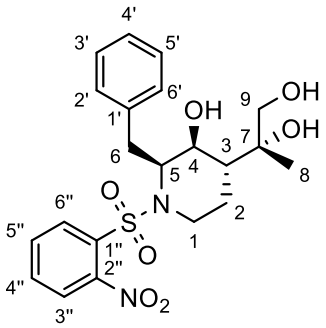


(*R*,*R*,*R*)-36f

(*R*,*R*,*R*)-**36f**, mp 71 °C. $[\alpha]_D^{20}$ + 70 (*c* 0.1 in $CHCl_3$). R_f = 0.22 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3364 (m, br), 2924 (vs), 2853 (s), 1733 (w), 1542 (vs), 1496 (w), 1455 (m), 1372 (s), 1337 (s), 1277 (m), 1160 (s), 1126 (s), 1089 (m), 1047 (m), 975 (m), 934 (m), 853 (w), 733 (m), 703 (m), 652 (w), 576 (m), 422 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.23 (s, 3H, 8-H), 1.66 – 1.73 (m, 1H, 2-H$_a$), 1.90 – 1.98 (m, 1H, 3-H), 2.05 (dddd, *J* = 13.2 Hz, 13.2 Hz, 13.2 Hz, 4.9 Hz, 1H, 2-H$_b$), 2.86 (dd, *J* = 13.7 Hz, 9.9 Hz, 1H, 6-H$_a$), 2.97 (dd, *J* = 13.7 Hz, 6.6 Hz, 1H, 6-H$_b$), 3.05 (s, br, 1H, OH), 3.18 (s, br, 1H, OH), 3.33 – 3.42 (m, 1H, 1-H$_a$), 3.49 – 3.55 (m, 2H, 9-H), 4.04 – 4.12 (m, 2H, 1-H$_b$, 4-H), 4.12 – 4.18 (m, 1H, 5-H), 7.08 – 7.13 (m, 3H, 2'-H, 4'-H, 6'-H), 7.14 – 7.19 (m, 2H, 3'-H, 5'-H), 7.54 – 7.66 (m, 3H, 3"-H, 4"-H, 5"-H), 7.89 – 7.95 (m, 1H, 6"-H). ^{13}C-NMR (176 MHz, $CDCl_3$) δ 19.8 (C-2), 23.2 (C-8), 35.6 (C-6), 38.9 (C-3), 41.7 (C-1), 62.3 (C-5), 66.8 (C-4), 68.6 (C-9), 74.1 (C-7), 124.4 (C-3"), 127.0 (C-4'), 128.6 (C-3', C-5'), 129.0 (C-2', C-6'), 131.0 (C-6"), 132.0 (C-5"), 133.4 (C-4"), 133.9 (C-1"), 136.9 (C-1'), 147.7 (C-2"). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_7S$ ([M+Na]$^+$): 473.1353 (473.1354).

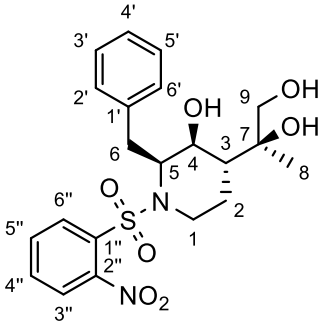
(*R*)-2-((2*S*,3*S*,4*R*)-2-Benzyl-3-hydroxy-1-((2-nitrophenyl)sulfonyl)piperidin-4-yl)-propane-1,2-diol ((*S*,*R*,*R*)-**36f) and (*S*)-2-((2*S*,3*S*,4*R*)-2-Benzyl-3-hydroxy-1-((2-nitrophenyl)sulfonyl)piperidin-4-yl)-propane-1,2-diol (**(*S*,*R*,*S*)-**36f)**

According to GP10 (*S*,*R*,*R*)-**36f** was prepared from (2*S*,3*S*,4*R*)-2-benzyl-4-((*S*)-2-methyloxiran-2-yl)-1-((2-nitrophenyl)-sulfonyl)piperidin-3-ol (*S*,*R*,*R*)-**35f** (65.0 mg, 0.15 mmol) and perchloric acid (2.0 mL, 2.47 mmol, 7 % in water) as a colorless solid (16.0 mg, 0.04 mmol, 24 %). Triol (*S*,*R*,*S*)-**36f** was isolated as well as a colorless solid (7.00 mg, 0.02 mmol, 11 %). Purification was accomplished by HPLC (Orbit, hexanes/isopropanol (90 : 10), 15 mL / min, R_t ((*S*,*R*,*R*)-**36f**) = 43 min, R_t ((*S*,*R*,*S*)-**36f**) = 51 min.



(*S*,*R*,*R*)-**36f**

(*S*,*R*,*R*)-**36f**, mp 107 °C. $[\alpha]_D^{20}$ + 246 (*c* 0.1 in $CHCl_3$). R_f = 0.27 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 2929 (m), 1543 (vs), 1468 (m), 1455 (m), 1440 (m), 1374 (vs), 1340 (vs), 1303 (m), 1281 (s), 1260 (m), 1157 (vs), 1125 (s), 1082 (vs), 1048 (vs), 1017 (m), 982 (w), 956 (s), 941 (m), 909 (s), 871 (s), 853 (m), 793 (m), 778 (m), 757 (vs), 730 (vs), 699 (s), 651 (m), 590 (vs), 569 (s), 558 (m), 541 (m), 508 (w), 492 (w), 460 (w), 421 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.21 (s, 3H, 8-H), 1.35 (dddd, *J* = 12.8 Hz, 12.8 Hz, 12.8 Hz, 4.8 Hz, 1H, 2-H_a), 1.62 (s, br, 2H, 2xOH), 1.70 – 1.80 (m, 1H, 2-H_b), 2.19 –2.28 (m, 1H, 3-H), 2.81 (dd, *J* = 14.1 Hz, 11.3 Hz, 1H, 6-H_a), 3.19 (dd, *J* = 14.1 Hz, 2.8 Hz, 1H, 6-H_b), 3.32 – 3.41 (m, 1H, 1-H_a), 3.42 (d, *J* = 11.1 Hz, 1H, 9-H_a), 3.59 (d, *J* = 11.1 Hz, 1H, 9-H_b), 3.94 – 4.02 (m, 1H, 1-H_b), 4.21 (dd, *J* = 10.9 Hz, 5.4 Hz, 1H, 4-H), 4.24 – 4.30 (m, 1H, 5-H), 6.83 – 6.93 (m, 3H, 3'-H, 4'-H, 5'-H), 6.97 – 7.03 (m, 2H, 2'-H, 6'-H), 7.24 – 7.30 (m, 1H, 5''-H), 7.33 – 7.40 (m, 1H, 6''-H), 7.42 – 7.50 (m, 2H, 3''-H, 4''-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 18.7 (C-8), 27.7 (C-2), 30.0 (C-6), 40.2 (C-3), 40.3 (C-1), 61.2 (C-5), 68.3 (C-9), 71.9 (C-4), 76.4 (C-7), 124.2 (C-3''), 126.3 (C-4'), 128.1 (C-3', C-5'), 129.2 (C-2', C-6'), 130.6 (C-6''), 131.9 (C-5''), 132.8 (C-4''), 134.0 (C-1''), 138.5 (C-1'), 147.1 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_7S$ ($[M+H]^+$): 451.1533 (451.1533).



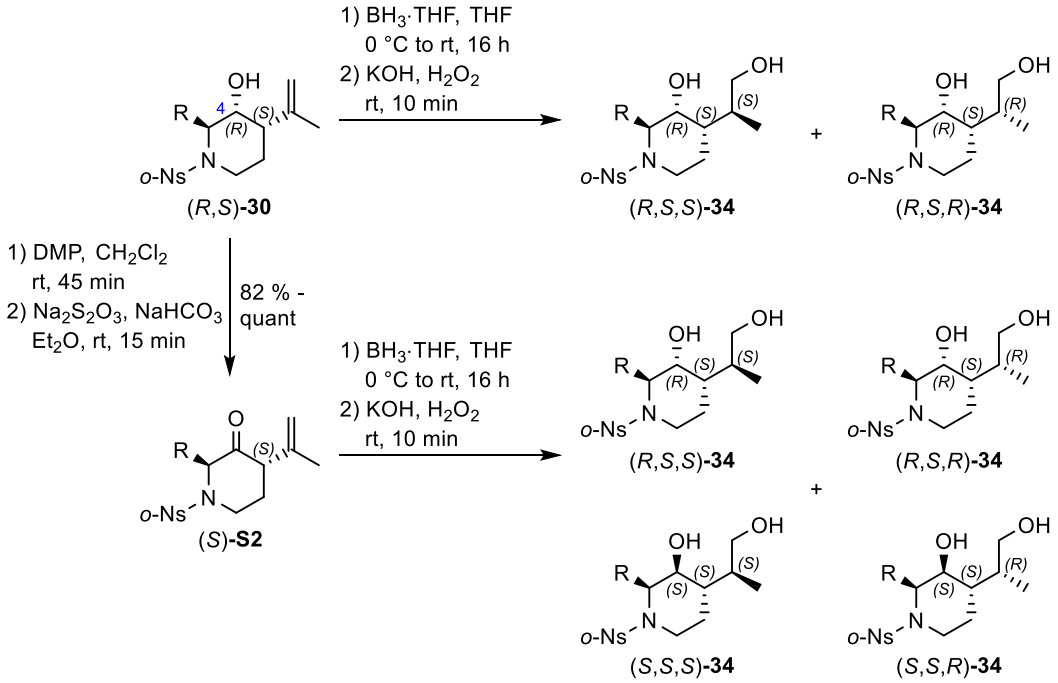
(*S*,*R*,*S*)-**36f**

(*S*,*R*,*S*)-**36f**, mp 160 °C. $[\alpha]_D^{20}$ + 270 (*c* 0.1 in $CHCl_3$). R_f = 0.19 (hexanes/EtOAc, 1 : 2, anisaldehyde solution). FT-IR (ATR): $\tilde{\nu}$ = 3029 (w), 2935 (m), 1541 (vs), 1497 (w), 1454 (m), 1440 (m), 1370 (s), 1335 (s), 1282 (m), 1159 (s), 1124 (s), 1097 (m), 1069 (m), 1014 (w), 957 (m), 912 (w), 865 (m), 853 (m), 778 (w), 756 (s), 739 (m), 700 (m), 652 (w), 591 (s), 570 (m), 508 (w) cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 1.26 (s, 3H, 8-H), 1.42 (dddd, *J* = 13.1 Hz, 13.1 Hz, 13.1 Hz, 4.7 Hz, 1H, 2-H_a), 1.80 – 1.89 (m, 1H, 2-H_b), 2.06 – 2.15 (m, 1H, 3-H), 2.78 (dd, *J* = 14.2 Hz, 11.2 Hz, 1H, 6-H_a), 3.17 (dd, *J* = 14.2 Hz, 2.3 Hz, 1H, 6-H_b), 3.29 – 3.40 (m, 1H, 1-H_a), 3.57 (d, *J* = 10.4 Hz, 1H, 9-H_a), 3.86 (d, *J* = 10.4 Hz, 1H, 9-H_b), 3.98 – 4.06 (m, 1H, 1-H_b), 4.15 – 4.27 (m, 2H, 4-H, 5-H), 6.83 – 6.93 (m, 3H, 3'-H, 4'-H, 5'-H), 6.97 – 7.03 (m, 2H, 2'-H, 6'-H), 7.24 – 7.30 (m, 1H, 5''-H), 7.35 – 7.40 (m, 1H, 6''-H), 7.42 – 7.52 (m, 2H, 3''-H, 4''-H). ^{13}C-NMR (126 MHz, $CDCl_3$) δ 24.2 (C-8), 27.8 (C-2), 29.8 (C-6), 40.7 (C-1), 44.1 (C-3), 61.5 (C-5), 66.2 (C-9), 72.3 (C-4), 76.1 (C-7), 124.2 (C-3''), 126.3 (C-4'), 128.1 (C-3', C-5'), 129.2 (C-2', C-6'), 130.7 (C-6''), 132.0 (C-5''), 132.8 (C-4''), 133.9 (C-1''), 138.5 (C-1'), 147.0 (C-2''). HRMS (ESI): calc (found) for $C_{21}H_{26}N_2O_7S$ ($[M+Na]^+$): 473.1353 (473.1359).

3) Functionalizations of hydroxypiperidines

In addition to the previous efforts a series of functionalizations of hydroxypiperidines was examined (Table S1). We aimed towards iminosugar analogues with different substitution patterns and stereoinformation to create a variety of compounds for the following biological examinations.

Table S1: Functionalization of olefin to diols 34



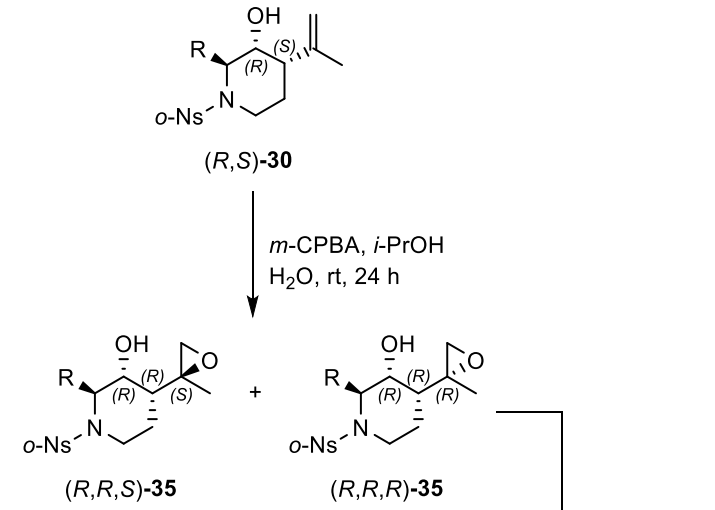
entry		substrate	R	yield [a] (*R*,*S*,*X*)-**34** [%]	dr [b]	yield [c] (*S*,*S*,*X*)-**34** [%]	dr [d]
1	L-Val	(*R*,*S*)-**30a**	*i*-Pr	42	74 : 26	-	-
2	L-Val	(*S*)-**S2a**	*i*-Pr	-	-	40	57 : 73
3	D-Val [e]	(*R*)-**S2b**	*i*-Pr	8	1 : 99<	74	49 : 51
4	L-Leu	(*S*)-**S2c**	*i*-Bu	-		49	59 : 41
5	L-Phe	(*S*)-**S2f**	Bn	13	23 : 77	36	42 : 58

[a] Combined yields of (*R*,*S*,*S*)-**34** and (*R*,*S*,*R*)-**34**; [b] Diastereomeric ratio of (*R*,*S*,*S*)-**34** and (*R*,*S*,*R*)-**34**; [c] Combined yields of (*S*,*S*,*S*)-**34** and (*S*,*S*,*R*)-**34**; [d] Diastereomeric ratio of (*S*,*S*,*S*)-**34** and (*S*,*S*,*R*)-**34**; [e] Starting from D-valine, all stereocenters are inverted. [d,e] Diastereomeric ratios were determined by HPLC and/or ^{1}H-NMR. In case of dr >99 : 1 no traces of the minor diastereomer could be detected by ^{1}H-NMR.

Conversion of L-Val alkene (*R*,*S*)-**30a** to diol (*R*,*S*,*X*)-**34a** was achieved by hydroboration using borane-THF solution. Two diastereomers (*R*,*S*,*S*)-**34a** and (*R*,*S*,*R*)-**34a** (entry 1) were isolated in a combined yield of 42 % with a diastereomeric ration of 74 : 26 after separation via HPLC. Since NOESY-data of our compounds showed no major differences due to the free rotation of the *exo*cyclic group, the newly formed stereocenter was assigned by comparison to similar structures in the literature.[18]

In an attempt to invert the stereoinformation at C-4 of these diols, the alkenes **30** were first converted to their corresponding ketones (*S*)-**S2** by DMP-oxidation according to the literature[6,16] in good yields (82 % - quant). This gave the additional benefit of using mixtures of alkenes (*R*,*S*)-**30** and (*S*,*S*)-**30**, which differed at the C-4, without the necessity of prior separation of the alkenes by HPLC. Employing the ketones (*S*)-**S2** in borane-mediated hydroboration under the same conditions as before, led to diols (*S*,*S*,*S*)-**34** and (*S*,*S*,*R*)-**34** with inverted stereochemistry at C-4 compared to the first attempt (entry 1). For L-valine no additional diols (*R*,*S*)-**34a** were observed with this method. Instead the diols (*S*,*S*,*S*)-**34a** and (*S*,*S*,*R*)-**34a** were isolated with the desired inversion with a combined yield of 40 % (dr 57 : 43) (entry 2). For D-valine diols (*R*,*R*,*R*)-**34b** and (*R*,*R*,*S*)-**34b** (all stereocenters are inverted) were isolated with a combined yield of 74 % (dr 49 : 51) (entry 3). In addition diol (*S*,*R*,*S*)-**34b** was isolated with 8 % yield as a single diastereomer. L-leucine ketone (*S*)-**S2c** gave (*S*,*S*,*S*)-**34c** and (*S*,*S*,*R*)-**34c** with a combined yield of 49 % (dr 59 : 41) (entry 4). Turning to L-phenylalanine all four diastereomers could be observed and isolated. The diols (*R*,*S*,*S*)-**34f** and (*R*,*S*,*R*)-**34f** were isolated with a combined yield of 13 % (dr 23 : 77) and the diols (*S*,*S*,*S*)-**34f** and (*S*,*S*,*R*)-**34f** were isolated with a combined yield of 36 % (dr 42 : 58) (entry 5). While being isolated mostly as solids, none of these diols could be recrystallized from different solvents to obtain crystal structures for assignment of the newly created *exo*cyclic stereocenter. Hence comparison to the literature[18] as described above was necessary.

To increase the polarity of the products we addressed next the synthesis of the corresponding triols **36** (Scheme S1). Therefore alkenes **30** were converted to their corresponding epoxides **35** by using *m*-CPBA. The epoxides were isolated as mixtures of diastereomers (*R*,*R*,*S*)-**35** and (*R*,*R*,*R*)-**35** and were separated for analytical purposes. The newly formed stereocenter couldn´t be assigned from the measured NOESY-spectra and therefore assignment was done by comparison of the epoxides **35** to similar compounds from the literature[19] and correlation with the triols **36**. The L-valine based epoxides (*R*,*R*,*S*)-**35a** and (*R*,*R*,*R*)-**35a** were isolated with a combined yield of 93 % (dr 44 : 56) (entry 1), while D-valine gave the epoxides (*S*,*S*,*R*)-**35b** and (*S*,*S*,*S*)-**35b** with a combined yield of 70 % (dr 33 : 67) (entry 2). For L-phenylalanine both diastereomers, (*R*,*S*)-**30f** as well as (*S*,*S*)- **30f**, could successfully be converted (entries 3,4). First, the epoxides (*R*,*R*,*S*)-**35f** and (*R*,*R*,*R*)-**35f** were isolated with a combined yield of 97 % (dr 60 : 40) (entry 3) and in a following experiment the epoxides (*S*,*R*,*S*)-**35f** and (*S*,*R*,*R*)-**35f** were isolated with a combined yield of 79 % (44 : 56) (entry 4).



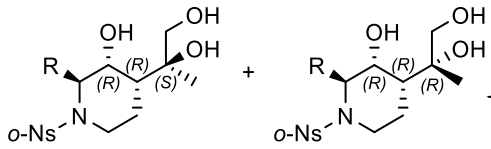
(*R*,*S*)-**30**

m-CPBA, *i*-PrOH
H_2O, rt, 24 h

(*R*,*R*,*S*)-**35** (*R*,*R*,*R*)-**35**

entry		R	substrate	yield **35** [%]	dr
1	L-Val	*i*-Pr	(*R*,*S*)-**30a**	93	44 : 56
2	D-Val [a]	*i*-Pr	(*S*,*R*)-**30b**	70	33 : 67
3	L-Phe	Bn	(*R*,*S*)-**30f**	97	60 : 40
4	L-Phe	Bn	(*S*,*S*)-**30f**	79	44 : 56 [c]

$HClO_4$, THF
H_2O, rt, 24 h



(*R*,*R*,*S*)-**36** (*R*,*R*,*R*)-**36**

entry		R	substrate	yield **36** [%]	dr
5	L-Val	*i*-Pr	(*R*,*R*,*S*)-**35a**	44	93 : 7
6	L-Val	*i*-Pr	(*R*,*R*,*R*)-**35a**	25	16 : 84
7	D-Val [a]	*i*-Pr	(*S*,*S*,*S*)-**35b**	50	12 : 88
8	L-Phe	Bn	(*R*,*R*,*X*)-**35f** [b]	78	50 : 50
9	L-Phe	Bn	(*S*,*R*,*R*)-**35f**	35	31 : 69 [c]

Scheme S1 [a] All stereocenters are inverted for D-valine; [b] A mixture of (*R*,*R*,*S*)-**35f** and (*R*,*R*,*R*)-**35f** was used; [c] dr of (*S*,*R*,*S*)- and (*S*,*R*,*R*)-derivatives. [d] Diastereomeric ratios were determined by HPLC and/or ^{1}H-NMR.

Subsequently, the epoxide groups were opened with perchloric acid to yield the corresponding triols as mixtures of diastereomers differing at the newly formed stereocenter (Scheme 7). The assignment of the new stereocenter was done by comparison to the literature[20] for the same reasons as described above. Also, no crystal structures could be obtained for both species. After separation of the epoxides *(R,R,S)*-**35a** and *(R,R,R)*-**35a**, the single diastereomer (*R*,*R*,*S*)-**35a** was converted to triols (*R*,*R*,*S*)-**36a** and (*R*,*R*,*R*)-**36a** with a combined yield of 44 % (dr 93 : 7) (entry 5), while epoxide (*R*,*R*,*R*)-**35a** gave the corresponding triols with a combined yield of 25 % (dr 16 : 84) (entry 6). D-Valine epoxide (*S*,*S*,*S*)-**35b** gave the corresponding triols (*S*,*S*,*R*)-**36b** and (*S*,*S*,*S*)-**36b** in a combined yield of 50 % (dr 12 : 88) (entry 7). For

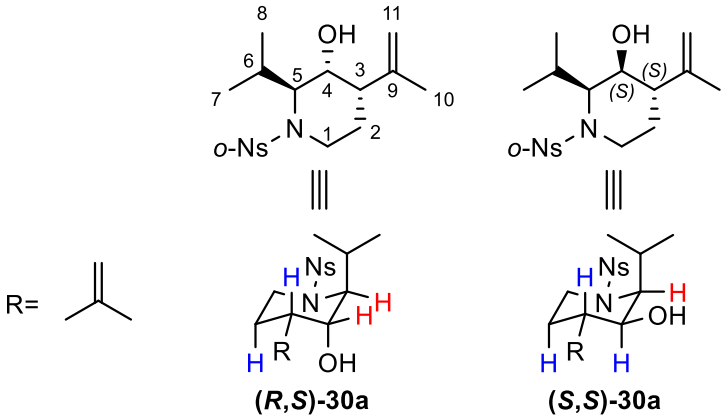
L-phenylalanine a mixture of both epoxides (*R*,*R*,*S*)-**35f** and (*R*,*R*,*R*)-**35f** was used, which gave the triols (*R*,*R*,*S*)-**36f** and (*R*,*R*,*R*)-**36f** in a combined yield of 78 % (dr 50 : 50) (entry 8). Switching to epoxide (*S*,*R*,*R*)-**35f** of L-phenylalanine gave the corresponding triols (*S*,*R*,*S*)-**36f** and (*S*,*R*,*R*)-**36f** in a combined yield of 35 % (dr 31 : 69) (entry 9). Using a single diastereomer of the epoxides gave one of their corresponding triols as major product, which resemble the orientation of the starting material. Mixtures of epoxides could be used as well and gave both diastereomers in the same amount.

4) Stereochemical elucidation of the cyclic hydroxypiperidines

All endocyclic stereocenters of the piperidine ring system could be assigned by analyzing the coupling constants between attached hydrogens using standard Karplus relations. The resulting structures were then verified by comparison to NOESY spectra.

One example for this approach is given in Table S2 by comparison of L-valine derivatives (*R*,*S*)-**30a** and (*S*,*S*)-**30a**. This method was applied for all further compounds of this work in an analogous procedure.

Table S2: Comparison of ^{3}J-coupling constants for stereo elucidation of L-valine derivatives (*R*,*S*)-**30a** and (*S*,*S*)-**30a**. *Equatorial* hydrogens were marked in red, *axial* hydrogens in blue.



Entry	Coupling constant	(*R*,*S*)-**30a**	(*S*,*S*)-**30a**
(1)	$^{3}J_{2H(ax)-3H}$	12.7 Hz	13.0 Hz
(2)	$^{3}J_{3H-4H}$	<5 Hz [a]	10.8 Hz
(3)	$^{3}J_{4H-5H}$	<5 Hz [a]	5.2 Hz

[a] Coupling constants taken from multiplet.

The Karplus relation was used to distinguish between *axial* and *equatorial* hydrogens in the six-membered ring system (Table S2). The stereocenter at C-5 was given by L-valine as the starting material. Since alkene (*R*,*S*)-**30a** showed a coupling constant of 12.7 Hz between one hydrogen of C-2 and C-3 (entry 1), these two hydrogens were in an *axial*-*axial*-correlation. This information led to an *axial* position of the isopropyl side chain at C-5. Both the coupling constants between hydrogens at C-3 and C-4 as well as between hydrogens at C-4 and C-5 were taken from multiplets and showed values of <5 Hz respectively (entries 2 and 3). Coming from an *axial* position of the hydrogen at C-3, this led to both hydrogens at C-4 and C-5 being in an *equatorial* position. A similar approach for alkene (*S*,*S*)-**30a** gave again an *axial* position for

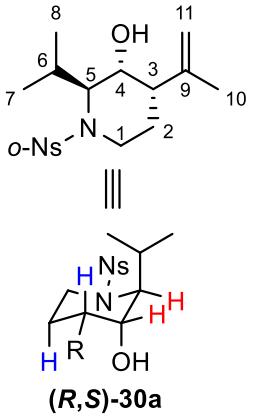
the hydrogen at C-3 because of a coupling constant of 13.0 Hz between one of the hydrogens at C-2 and C-3 (entry 1). This time however the coupling constant between the hydrogens at C-3 and C-4 showed a value of 10.8 Hz (entry 2), which indicated an *axial-axial* relation. Therefore, the hydrogen at C-4 was assigned to an *axial* position. The coupling constant for the hydrogens at C-4 and C-5 showed a value of 5.2 Hz (entry 3), which was possible for the expected *axial-equatorial* correlation. The resulting structures were compared to NOESY data for the investigated compounds (Table S3). This confirmed the previously assigned structures.

The newly formed exocyclic stereocenter of the functionalized hydroxy piperidines was assigned by comparison to similar structures from the literature (see Table S4-S8, only selected examples were shown). This method was applied for all further compounds of this work in an analogous procedure.

One example is given in Table S4 by comparison of the L-valine derivatives (*R*,*S*,*X*)-**34a** and the L-phenylalanine derivatives (*R*,*S*,*X*)-**34f** with compound **S3** from the literature.[21] The signals from the ^{1}H and ^{13}C NMR-spectra for the exocyclic group containing the newly formed stereocenter were analyzed. The chemical shift values for the 8-H of **S3** were more like the values obtained for the first diols (entry 1). Also, the values for the chemical shifts and coupling constants of 9-H_a and 9-H_b were a better fit with these compounds (entries 2 and 3). The values for 10-H (entry 4) were fitting this assignment as well, while the comparison of ^{13}C NMR signals gave no clear relation to one of these compounds (entries 5 – 7).. However because of the mentioned similarities in the ^{1}H NMR data the first diols were assigned as (*R*,*S*,*S*)-**34a** and (*R*,*S*,*S*)-**34f**, while the second diols were assigned as (*R*,*S*,*R*)-**34a** and (*R*,*S*,*R*)-**34f**. Similar analysis led to assignment of further compounds derived from other amino acids.

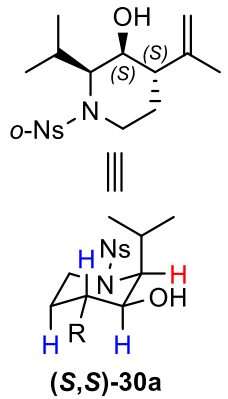
Table S3: Comparison of NOESY-signals for L-valine derivatives (*R*,*S*)-**30a** and (*S*,*S*)-**30a**.Color coding: red: no signal observed, green: signal visible, yellow: no information due to overlapping signals, orange: diagonal.

	1-H_{ax}	1-H_{eq}	2-H_{ax}	2-H_{eq}	3-H	4-H	5-H	6-H	7/8-H	8/7-H	10-H	11-H_a	11-H_b
1-H_{ax}													
1-H_{eq}													
2-H_{ax}													
2-H_{eq}													
3-H													
4-H													
5-H													
6-H													
7/8-H													
8/7-H													
10-H													
11-H_a													
11-H_b													



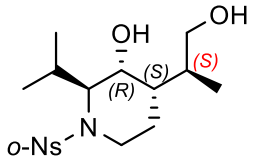
(*R*,*S*)-30a

	1-H_{ax}	1-H_{eq}	2-H_{ax}	2-H_{eq}	3-H	4-H	5-H	6-H	7/8-H	8/7-H	10-H	11-H_a	11-H_b
1-H_{ax}													
1-H_{eq}													
2-H_{ax}													
2-H_{eq}													
3-H													
4-H													
5-H													
6-H													
7/8-H													
8/7-H													
10-H													
11-H_a													
11-H_b													

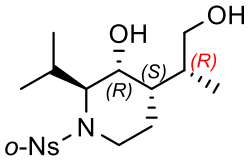


(*S*,*S*)-30a

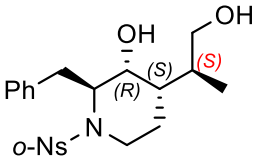
Table S4: Comparison of NMR data with the literature known compound **S3**[21] for the assignment of the newly formed stereocenter of diols (*R*,*S*,*X*)-**34a,f**.



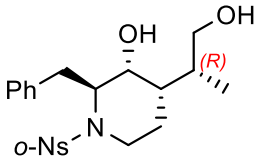
(*R*,*S*,*S*)-**34a**



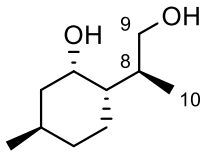
(*R*,*S*,*R*)-**34a**



(*R*,*S*,*S*)-**34f**



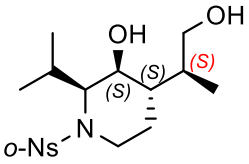
(*R*,*S*,*R*)-**34f**



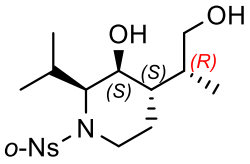
S3

Entry		(*R*,*S*,*S*)-**34a**	(*R*,*S*,*R*)-**34a**	(*R*,*S*,*S*)-**34f**	(*R*,*S*,*R*)-**34f**	**S3**
(1)	8-H	1.61-1.69 (m)	1.70-1.80 (m)	1.66-1.75 (m)	1.70-1.82 (m)	1.62 (m)
(2)	9-H_a	3.50 (dd, *J* = 11.2, 5.2 Hz)	3.45 (dd, *J* = 11.1, 4.2 Hz)	3.49 (dd, *J* = 11.0, 5.1 Hz)	3.50 (dd, *J* = 11.0, 4.4 Hz)	3.56 (dd, *J* = 11.0, 6.0 Hz)
(3)	9-H_b	3.65 (dd, *J* = 11.2, 3.0 Hz)	3.49 (dd, *J* = 11.1, 6.4 Hz)	3.63 (dd, *J* = 11.0, 3.0 Hz)	3.53 (dd, *J* = 11.0, 6.3 Hz)	3.68 (dd, *J* = 11.0, 3.0 Hz)
(4)	10-H	0.93-1.00 (m)	0.97 (d, *J* = 10.6 Hz)	1.00 (d, *J* = 6.9 Hz)	0.95 (d, *J* = 7.0 Hz)	1.00 (m, *J* = 7.0 Hz)
(5)	C-8	37.3	37.6	37.1	37.3	38.1
(6)	C-9	64.6	64.5	64.6	64.5	65.0
(7)	C-10	15.4	16.3	15.3	16.2	15.9

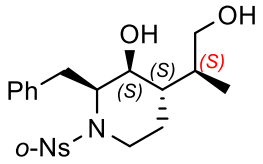
Table S5: Comparison of NMR data with the literature known compound **S4**[18] for the assignment of the newly formed stereocenter of diols (*S*,*S*,*X*)-**34a,f**.



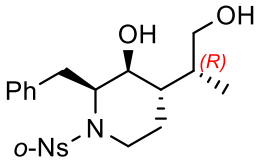
(*S*,*S*,*S*)-**34a**



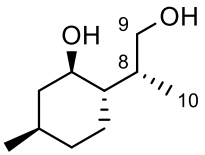
(*S*,*S*,*R*)-**34a**



(*S*,*S*,*S*)-**34f**



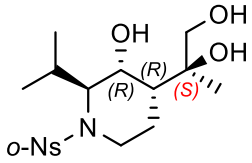
(*S*,*S*,*R*)-**34f**



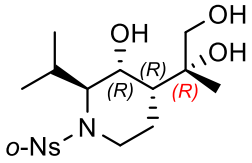
S4

Entry		(*S*,*S*,*S*)-**34a**	(*S*,*S*,*R*)-**34a**	(*S*,*S*,*S*)-**34f**	(*S*,*S*,*R*)-**34f**	**S4**
(1)	8-H	1.95-2.13 (m)	1.87-2.00 (m)	1.95-2.10 (m)	1.90-2.02 (m)	1.80-1.88 (m)
(2)	9-H_a	3.47 (dd, *J* = 10.5, 7.8 Hz)	3.54 (dd, *J* = 10.6, 2.8 Hz)	3.56 (dd, *J* = 10.5, 7.8 Hz)	3.63 (dd, *J* = 10.5, 3.1 Hz)	3.56 (dd, *J* = 10.7, 3.4 Hz)
(3)	9-H_b	3.60 (dd, *J* = 10.5, 5.2 Hz)	3.63 (dd, *J* = 10.6, 6.3 Hz)	3.70 (dd, *J* = 10.5, 4.8 Hz)	3.74 (dd, *J* = 10.5, 5.8 Hz)	3.65 (dd, *J* = 10.7, 5.5 Hz)
(4)	10-H	0.77 (d, *J* = 6.9 Hz)	0.87 (d, *J* = 7.3 Hz)	0.91 (d, *J* = 6.9 Hz)	0.99 (d, *J* = 7.3 Hz)	-
(5)	C-8	34.8	40.4	35.6	37.4	-
(6)	C-9	66.0	66.1	66.1	66.4	67.2
(7)	C-10	11.9	12.2			11.9

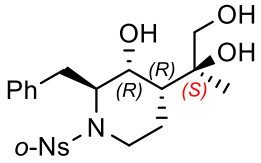
Table S6: Comparison of NMR data with the literature known compounds **S5** and **S6**[17] for the assignment of the newly formed stereocenter of triols (*R*,*R*,*X*)-**36a,f**.



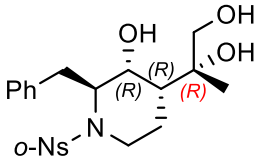
(*R*,*R*,*S*)-**36a**



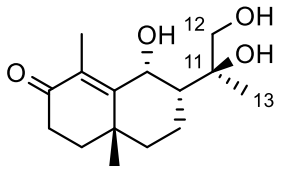
(*R*,*R*,*R*)-**36a**



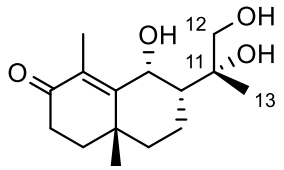
(*R*,*R*,*S*)-**36f**



(*R*,*R*,*R*)-**36f**



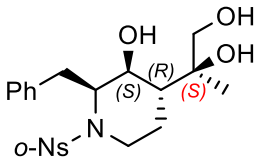
S5



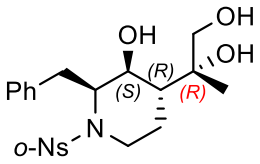
S6

Entry		(*R*,*R*,*S*)-**36a**	(*R*,*R*,*R*)-**36a**	(*R*,*R*,*S*)-**36f**	(*R*,*R*,*R*)-**36f**	**S5**	**S6**
(1)	12-H_a	3.26 (d, *J* = 11.5 Hz)	3.57 (s),	3.32 (d, *J* = 11.2 Hz)	3.49-3.55 (m),	3.34 (d, *J* = 11.2 Hz)	3.62 (d, *J* = 11.9 Hz)
(2)	12-H_b	3.55 (d, *J* = 11.5 Hz)	-	3.61 (d, *J* = 11.2 Hz)	-	3.73 (d, *J* = 11.2 Hz)	3.78 (d, *J* = 11.9 Hz)
(3)	13-H	1.22 (s)	1.17 (s)	1.23 (s)	1.23 (s)	1.34 (s)	1.23 (s)
(4)	C-11	73.0	74.0	73.1	74.1	74.8	75.7
(5)	C-12	66.5	68.8	66.7	68.6	66.2	69.2
(6)	C-13	24.8	23.1	24.6	23.2	24.4	22.8

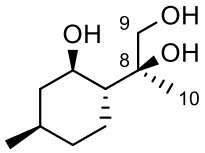
Table S7: Comparison of NMR data with the literature known compounds **S7** and **S8**[20] for the assignment of the newly formed stereocenter of triols (*S*,*R*,*X*)-**36a,f**.



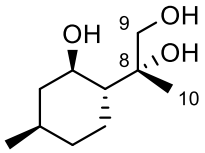
(*S*,*R*,*S*)-**36f**



(*S*,*R*,*R*)-**36f**



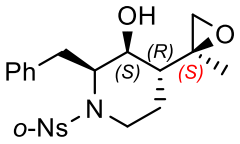
S7



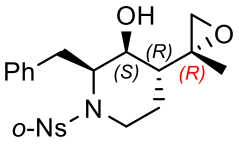
S8

Entry		(*S*,*R*,*S*)-**36f**	(*S*,*R*,*R*)-**36f**	**S7**	**S8**
(1)	9-H_a	3.57 (d, *J* = 10.4 Hz)	3.42 (d, *J* = 11.1 Hz)	3.34 (dd, *J* = 10.3, 5.5 Hz)	3.16 (dd, *J* = 11.2, 5.1 Hz)
(2)	9-H_b	3.86 (d, *J* = 10.4 Hz)	3.59 (d, *J* = 11.1 Hz)	3.41 (dd, *J* = 10.8, 6.2 Hz)	3.26 (dd, *J* = 11.2, 6.9 Hz)
(3)	10-H	1.26 (s)	1.21 (s)	1.01 (s)	0.97 (s)
(4)	C-8	76.1	76.4	74.8	75.7
(5)	C-9	66.2	68.3	67.6	68.1
(6)	C-10	24.2	18.7	22.1	19.8

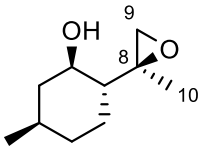
Table S8: Comparison of NMR data with the literature known compounds **S9** and **S10**[19] for the assignment of the newly formed stereocenter of epoxides (*S*,*R*,*X*)-**35f**.



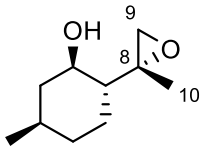
(*S*,*R*,*S*)-**35f**



(*S*,*R*,*R*)-**35f**



S9



S10

Eintrag		(*S*,*R*,*S*)-**35f**	(*S*,*R*,R)-**35f**	**S9**	**S10**
(1)	9-H_a	2.60 (d, *J* = 11.1 Hz)	2.71 (d, *J* = 10.4 Hz)	2.54 (dd, *J* = 11.2, 5.1 Hz)	2.66 (dd, *J* = 10.3, 5.5 Hz)
(2)	9-H_b	2.66 (d, *J* = 11.1 Hz)	3.05 (d, *J* = 10.4 Hz)	2.59 (dd, *J* = 11.2, 6.9 Hz)	2.92 (dd, *J* = 10.8, 6.2 Hz)
(3)	10-H	1.36 (s)	1.42 (s)	1.31 (s)	1.37 (s)
(4)	C-8	58.5	60.1	59.1	60.2
(5)	C-9	52.6	51.7	51.2	49.0
(6)	C-10	16.3	20.9	16.9	20.8

5) Chitin fiber synthesis assay with *Thalassiosira rotula*

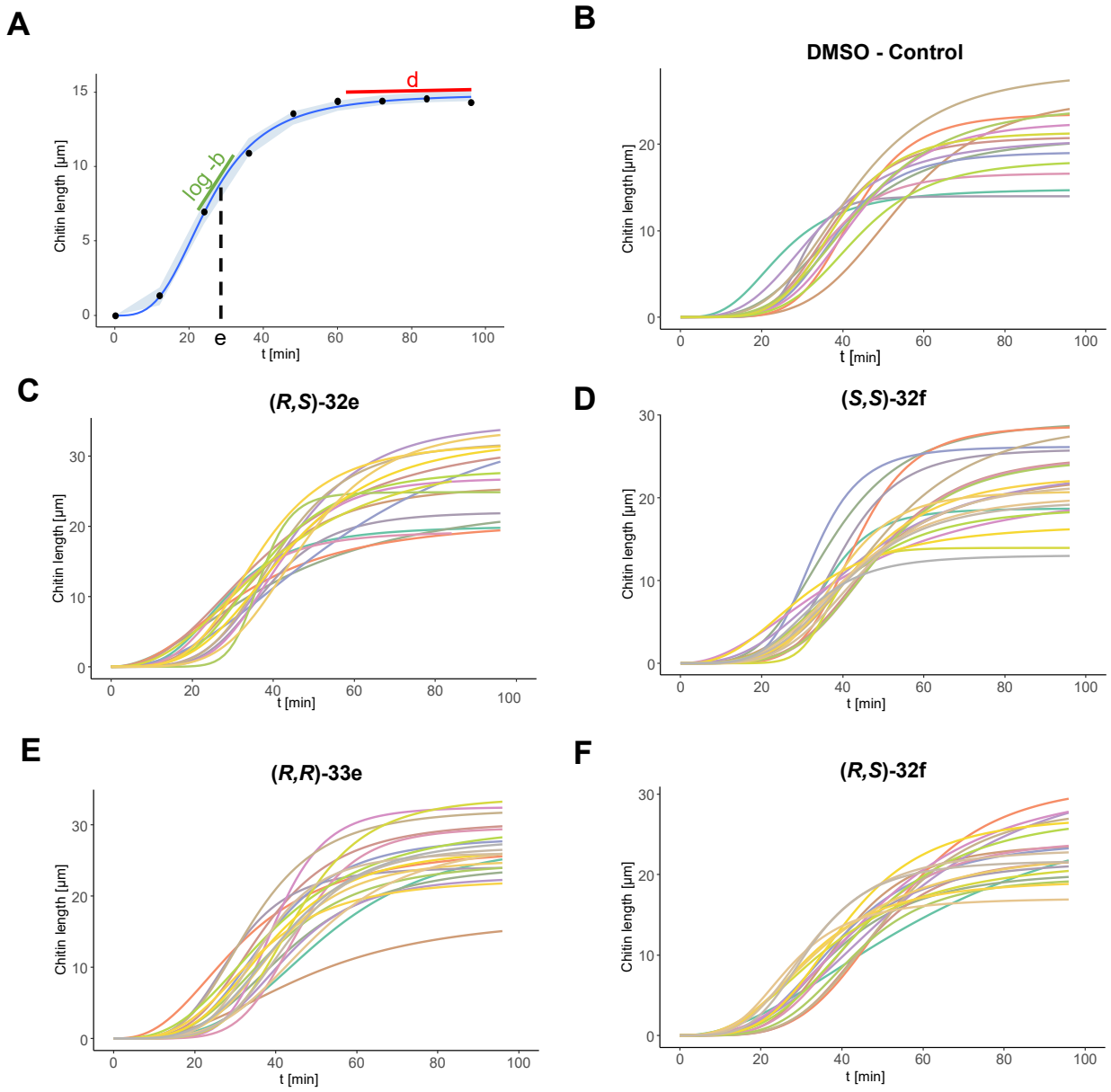


Figure S1: Nonlinear regression model fit of the single cell measurements. **A**: Interpretation of the calculated parameters. *e* is the halfway point (time where half the maximum length of the chitin is synthesized), *b* is the Hill slope (relative slope at the halfway point), *d* is the achieved length of the synthesized chitin fiber. Black dots are original chitin length measurements of cell 1 from the DMSO control (see Supplementary File 1 - Thalassiosira Measurements). Blue line represents the calculated fit. Light-blue ribbon signifies the 95% confidence interval calculated with the *predict* function in R[22]. **B**-**F**: Three-parameter logistic fit of the single chitin measurements for each individual treatment. **B**: (*S*,*S*)-32f, **C**: (*R*,*S*)-33e, **D**: (*R*,*S*)-33f, and **E**: (*R*,*R*)-33e). For each treatment condition the cells were supplemented with 100 µM (final concentration) iminosugar and were observed for 100 min. All fits were calculated with the *drm* function (LL.3) of the drc package (version 3.0-1) in R[23].

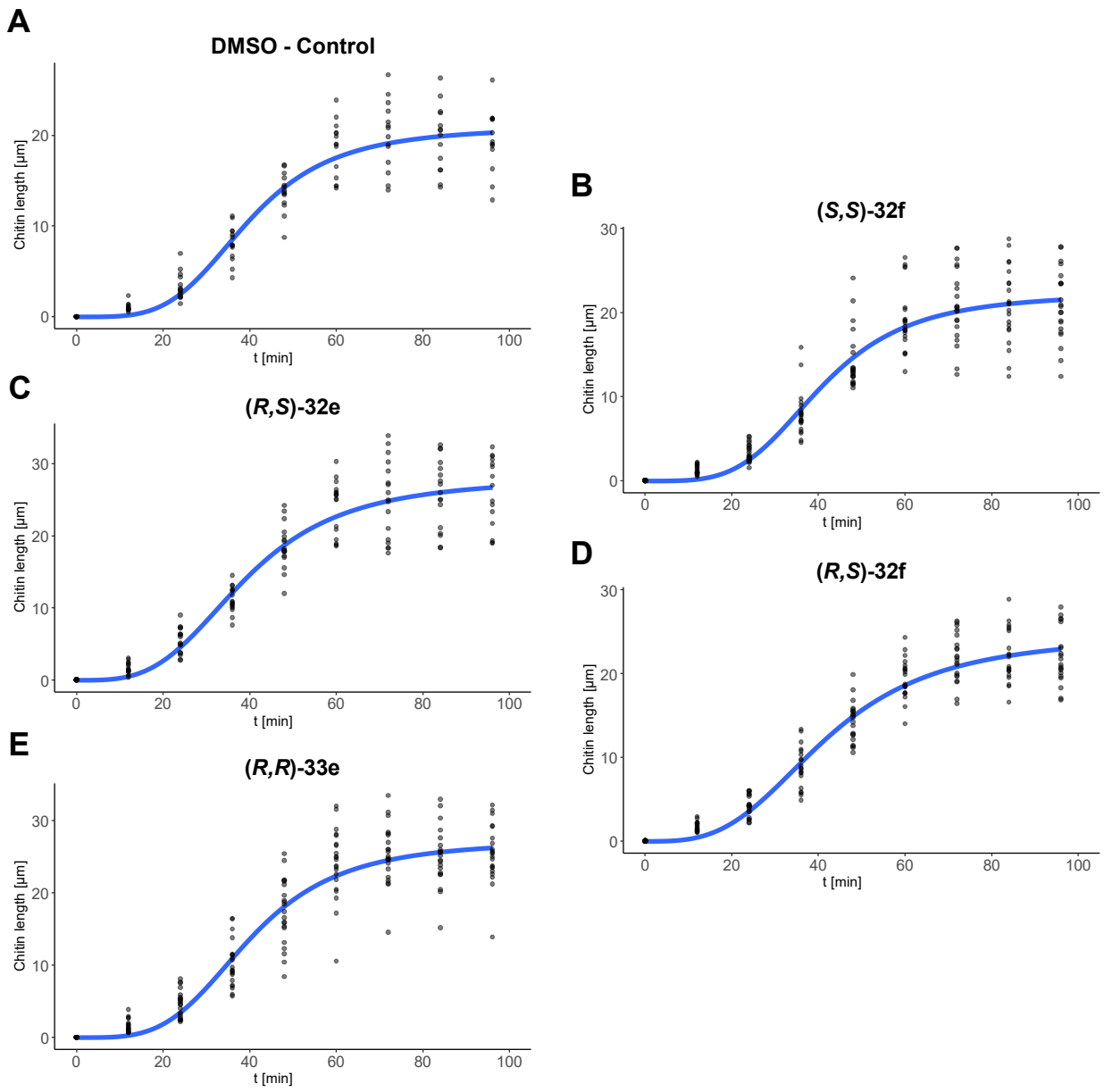


Figure S2: Average of the three-parameter logistic fit of the iminosugar-treated cells (blue line). The original measured datapoints are represented by black dots. In each dataset the cells were treated with 100 µM iminosugar (final concentration) and were observed for 100 min. **A**: Control with DMSO, **B**: (*S*,*S*)-32f, **C**: (*R*,*S*)-32e, **D**: (*R*,*S*)-32f, and **E**: (*R*,*R*)-33e. All fits were calculated with the *drm* function (LL.3) of the drc package (version 3.0-1) in the R environment[23].

6) Literature

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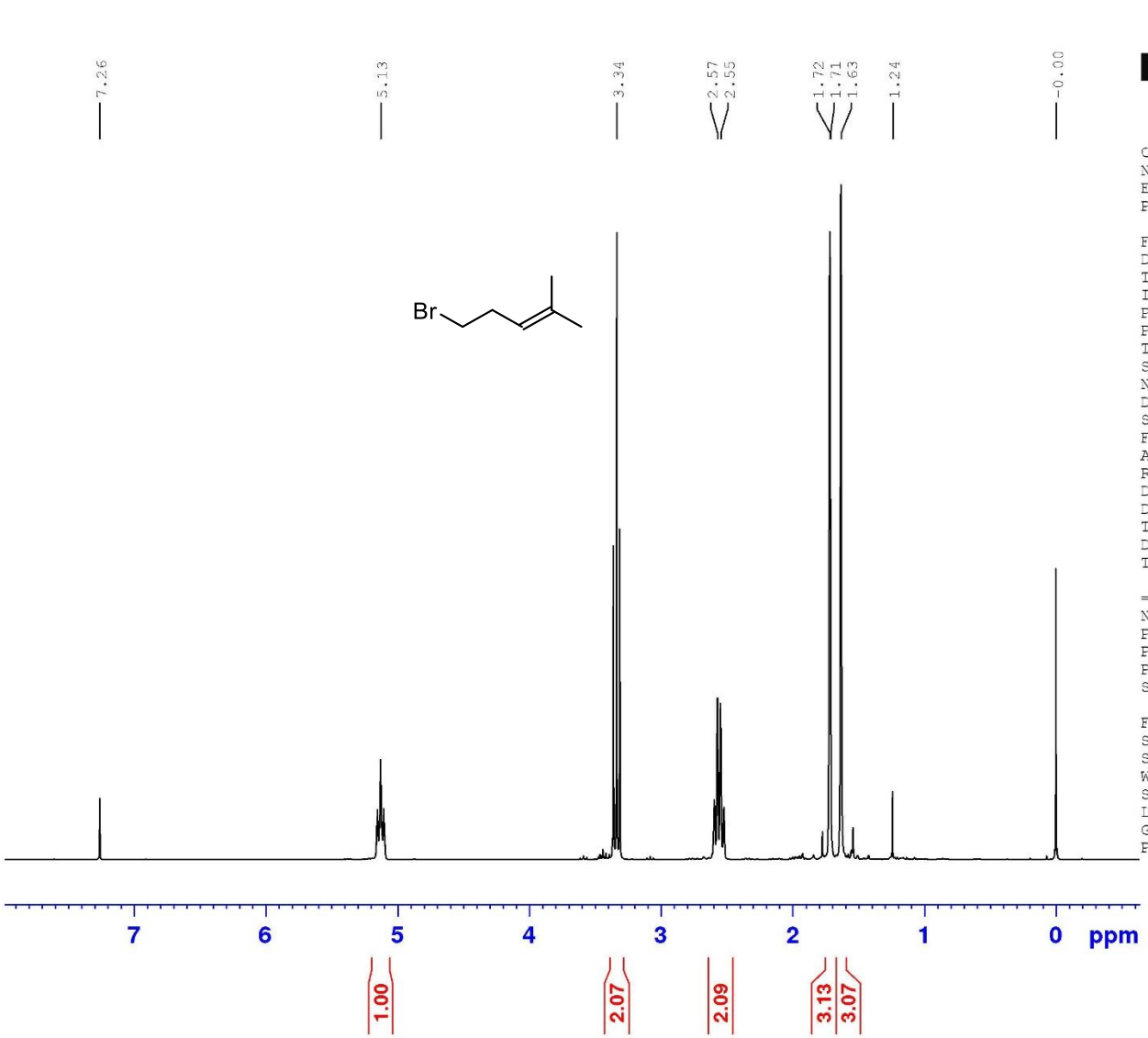
7) NMR-spectra

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RG                   203
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DE                 8.00 usec
TE                298.1 K
D1           1.00000000 sec
TD0                    1

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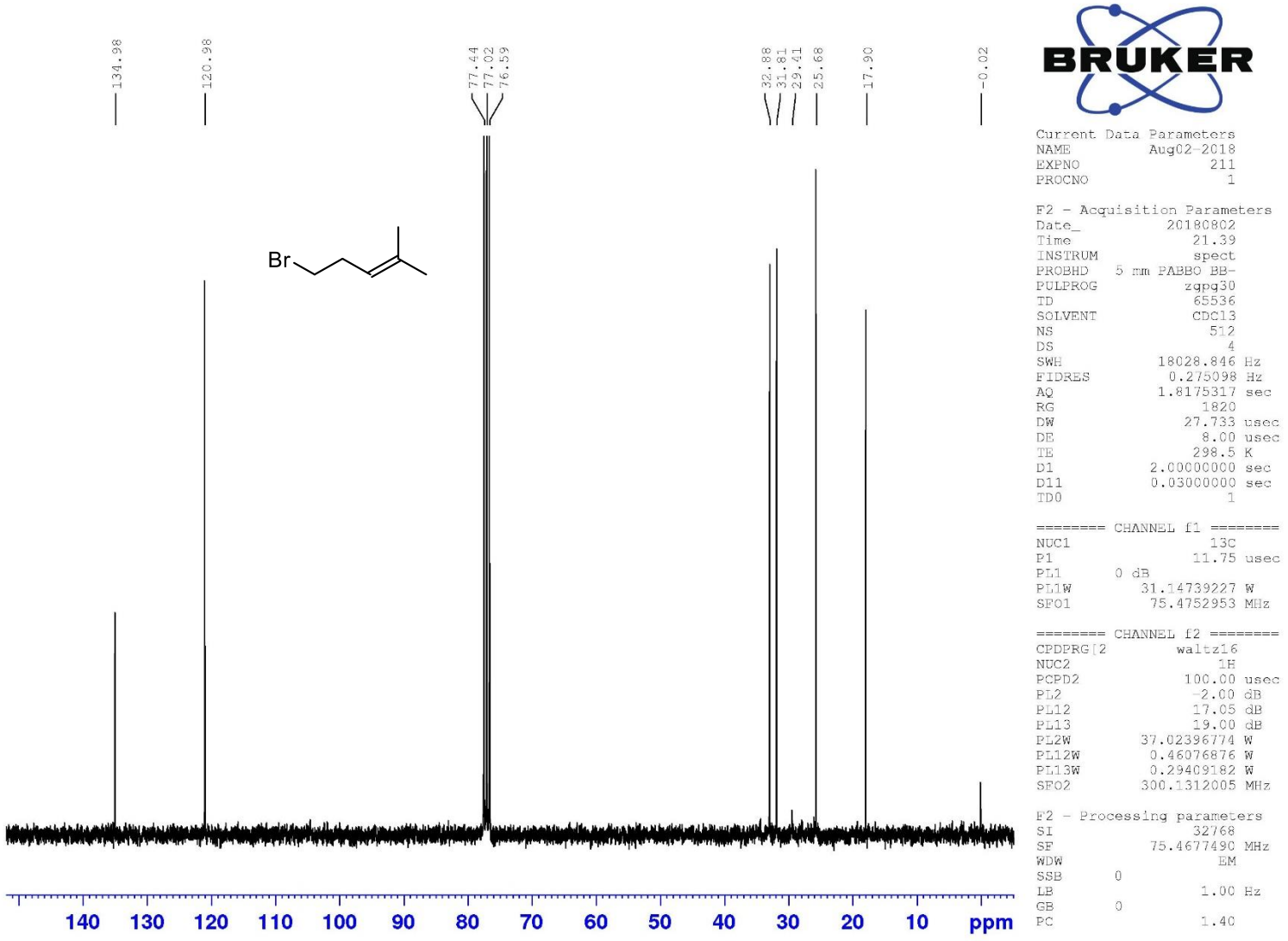
^{1}H-NMR **S1**

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DS                    4
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AQ            1.8175317 sec
RG                 1820
DW               27.733 usec
DE                8.00 usec
TE                298.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

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NUC1                13C
P1                11.75 usec
PL1       0 dB
PL1W        31.14739227 W
SFO1         75.4752953 MHz

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NUC2                1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.05 dB
PL13               19.00 dB
PL2W        37.02396774 W
PL12W        0.46076876 W
PL13W        0.29409182 W
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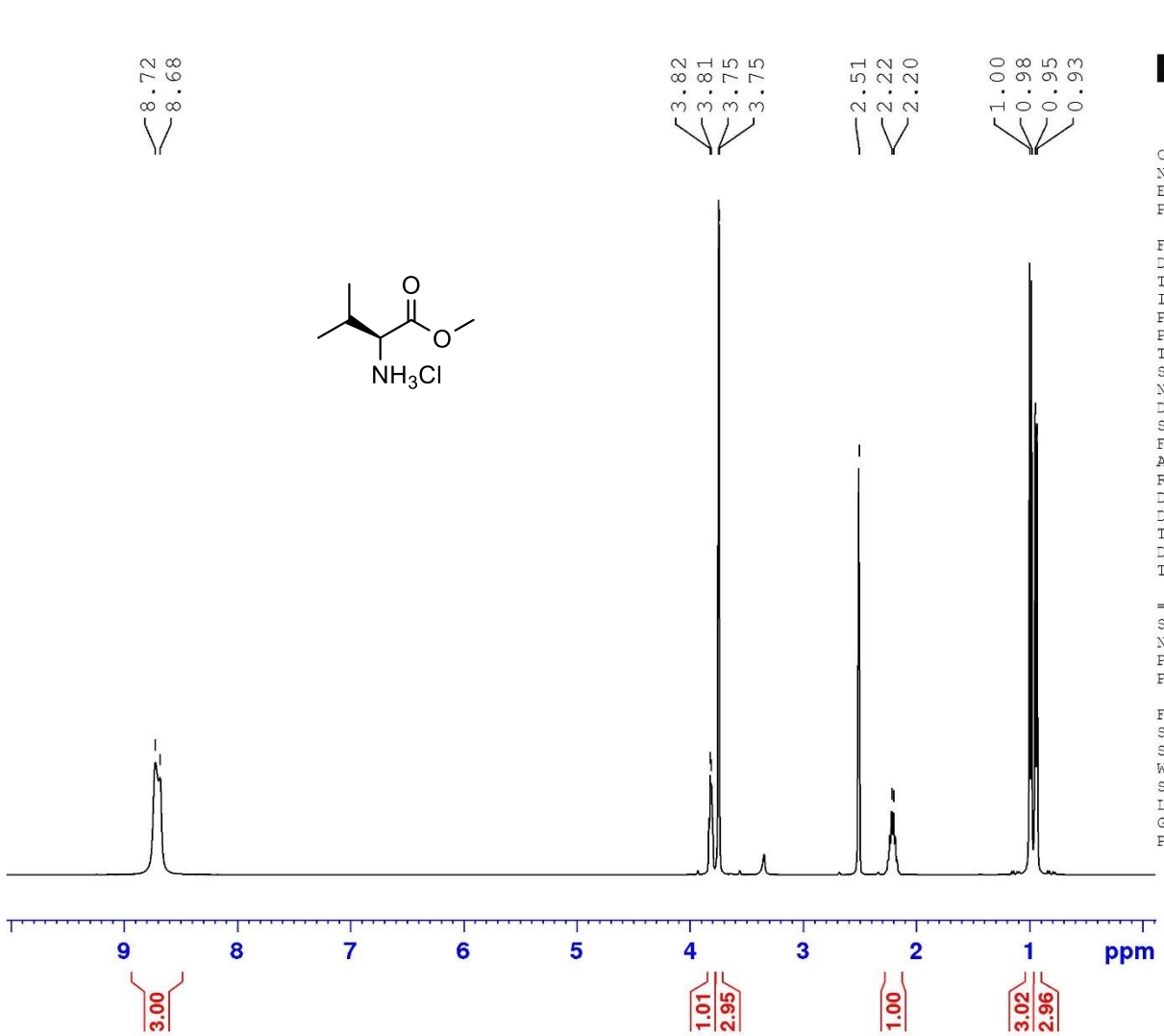


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TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 114.36
DW                 62.400 usec
DE                   6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                    1

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NUC1                   1H
P1                  13.70 usec
PLW1         12.00000000 W

F2 - Processing parameters
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WDW                   EM
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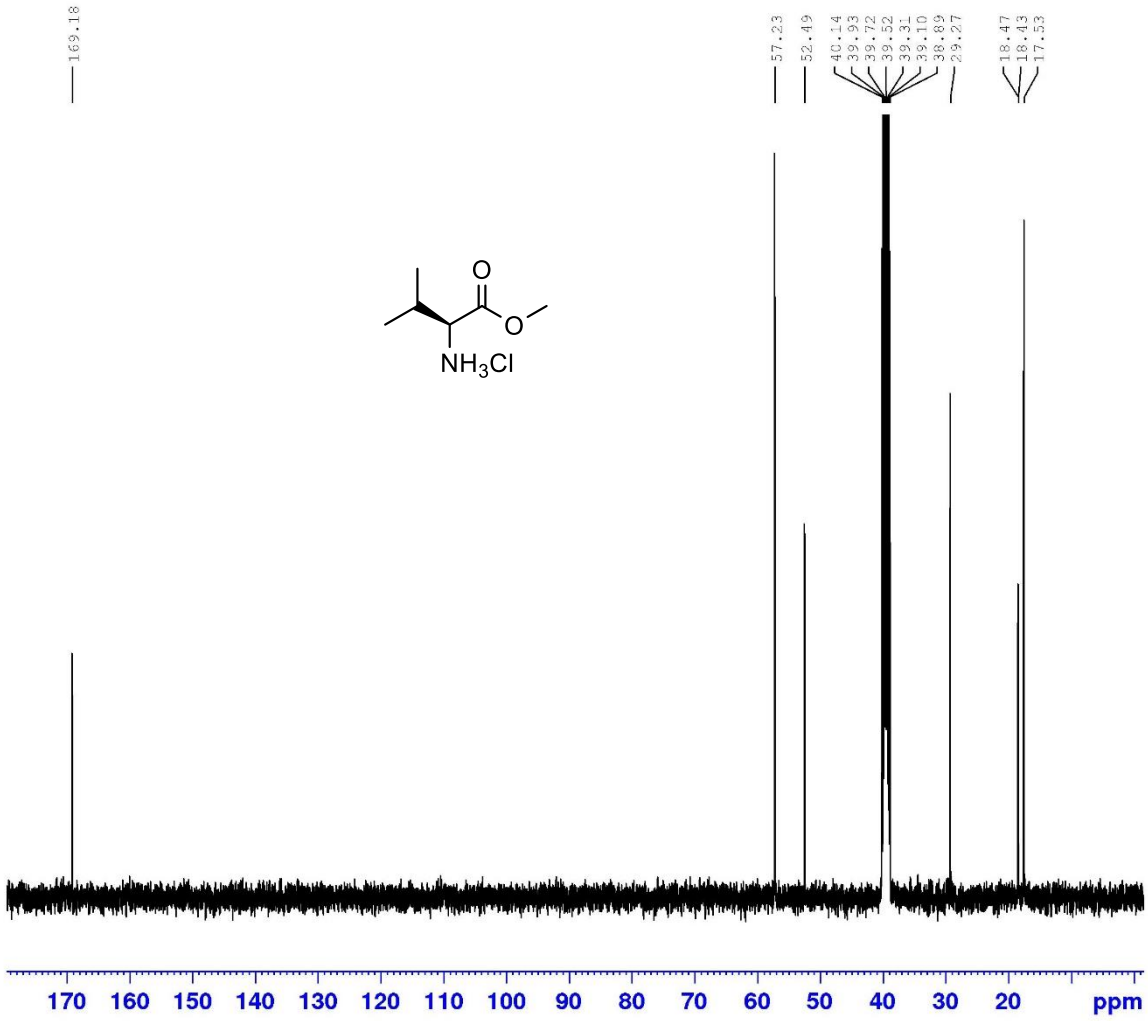
^{1}H-NMR **25a**

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FIDRES          0.366798 Hz
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RG                205.35
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

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SFO1         100.6152851 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1016004 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.27805999 W
PLW13         0.22522999 W

F2 - Processing parameters
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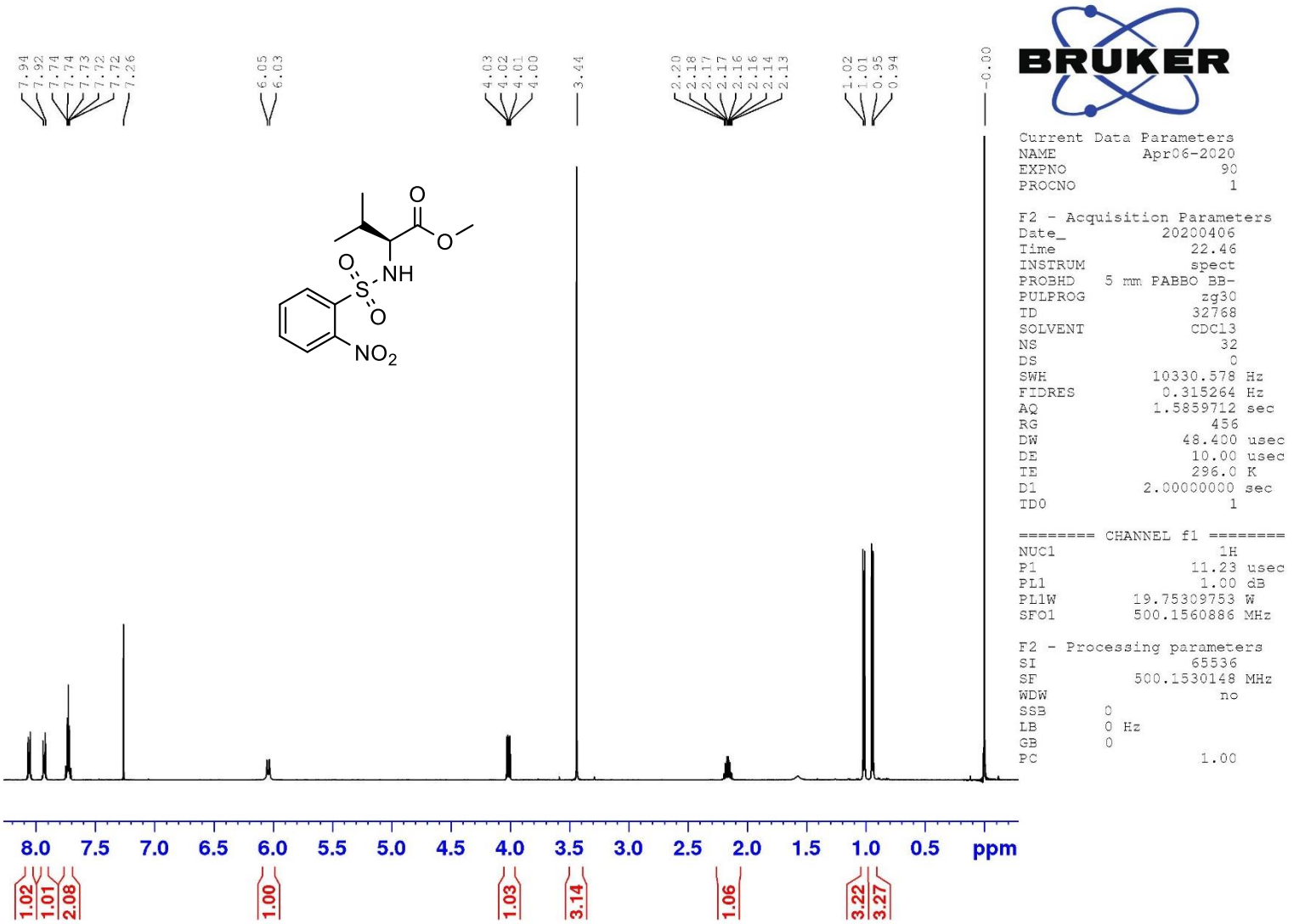
^{13}C-NMR **25a**



BRUKER

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FIDRES         0.315264 Hz
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RG                   456
DW                48.400 usec
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TE                296.0 K
D1            2.00000000 sec
ID0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
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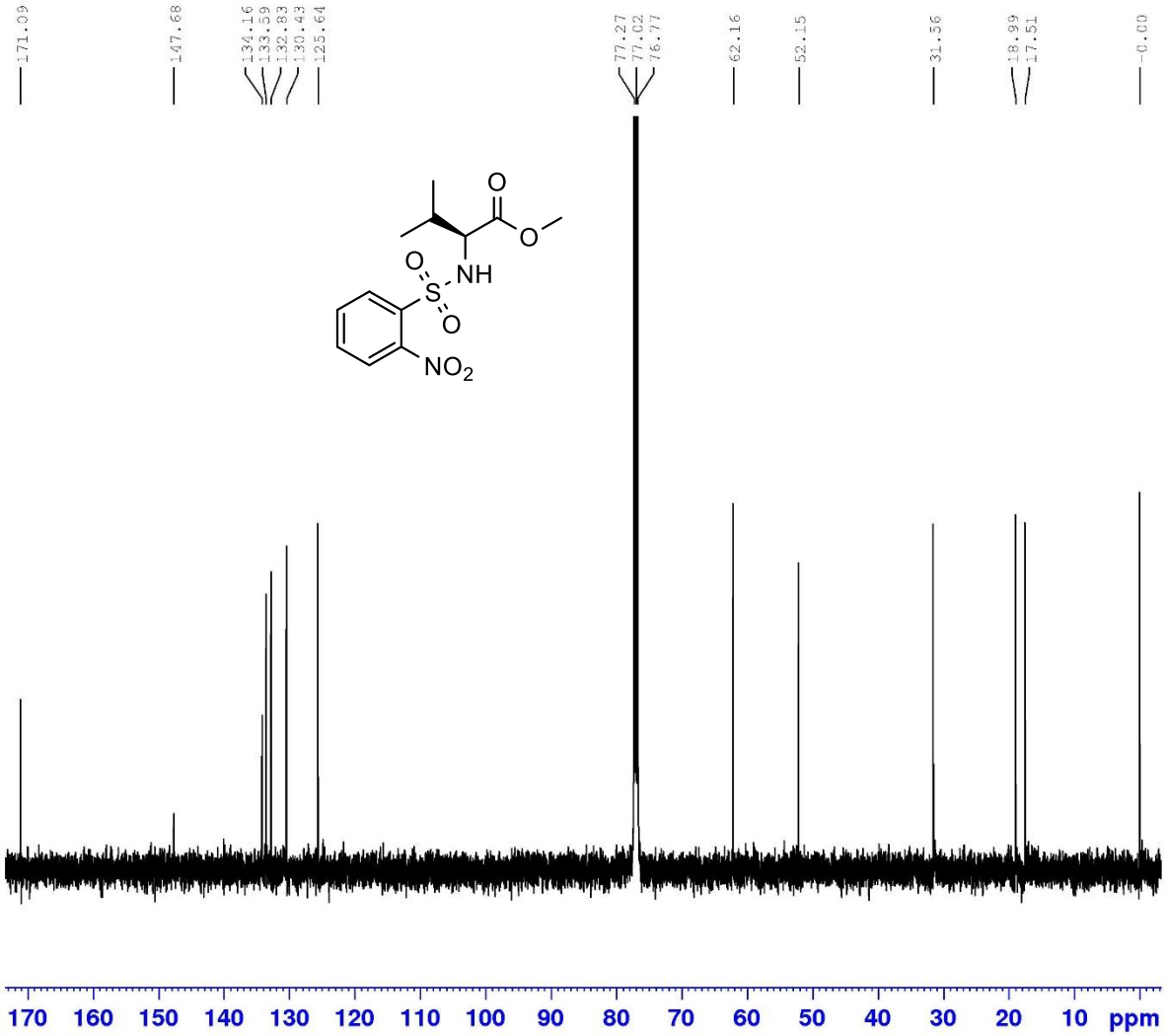
^{1}H-NMR **26a**



BRUKER

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FIDRES         0.501934 Hz
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RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
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P1                  10.20 usec
PL1                  1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
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PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
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SFO2        500.1550006 MHz

F2 - Processing parameters
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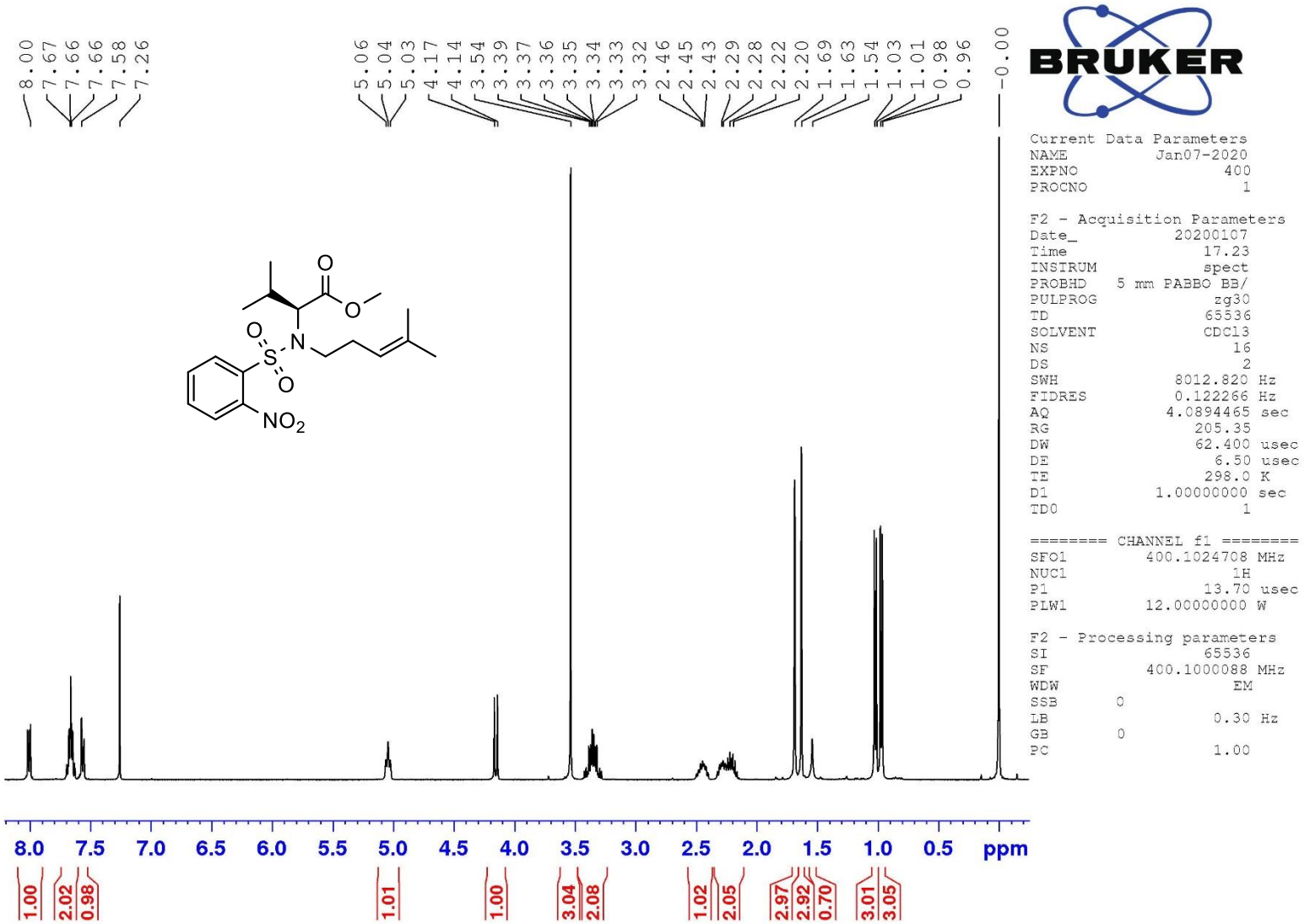
^{13}C-NMR **26a**



BRUKER

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TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
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FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                205.35
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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NUC1                   1H
P1                 13.70 usec
PLW1         12.00000000 W

F2 - Processing parameters
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WDW                   EM
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LB                 0.30 Hz
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PC                 1.00
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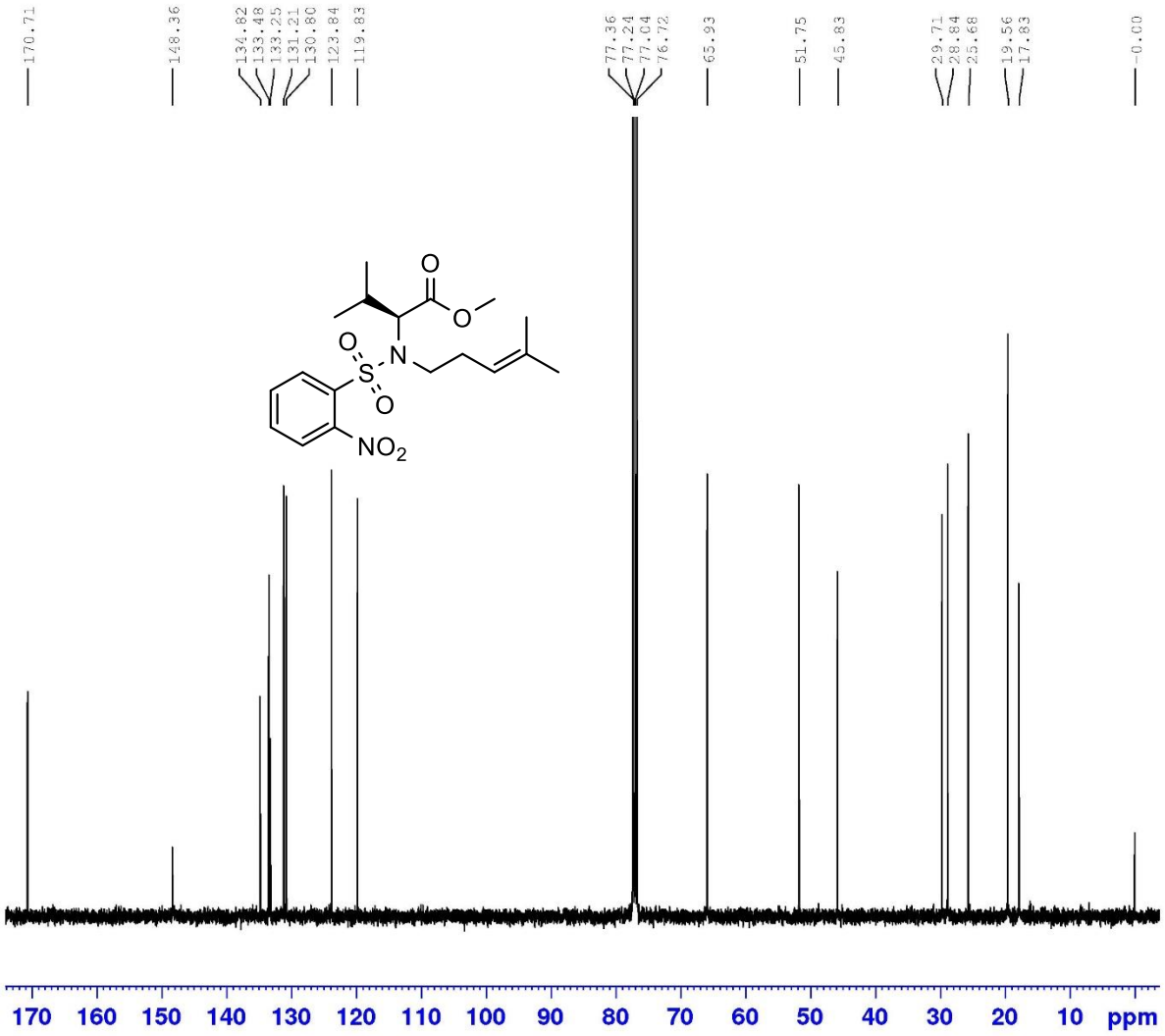
^{1}H-NMR **27a**



BRUKER

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PROCNO 1

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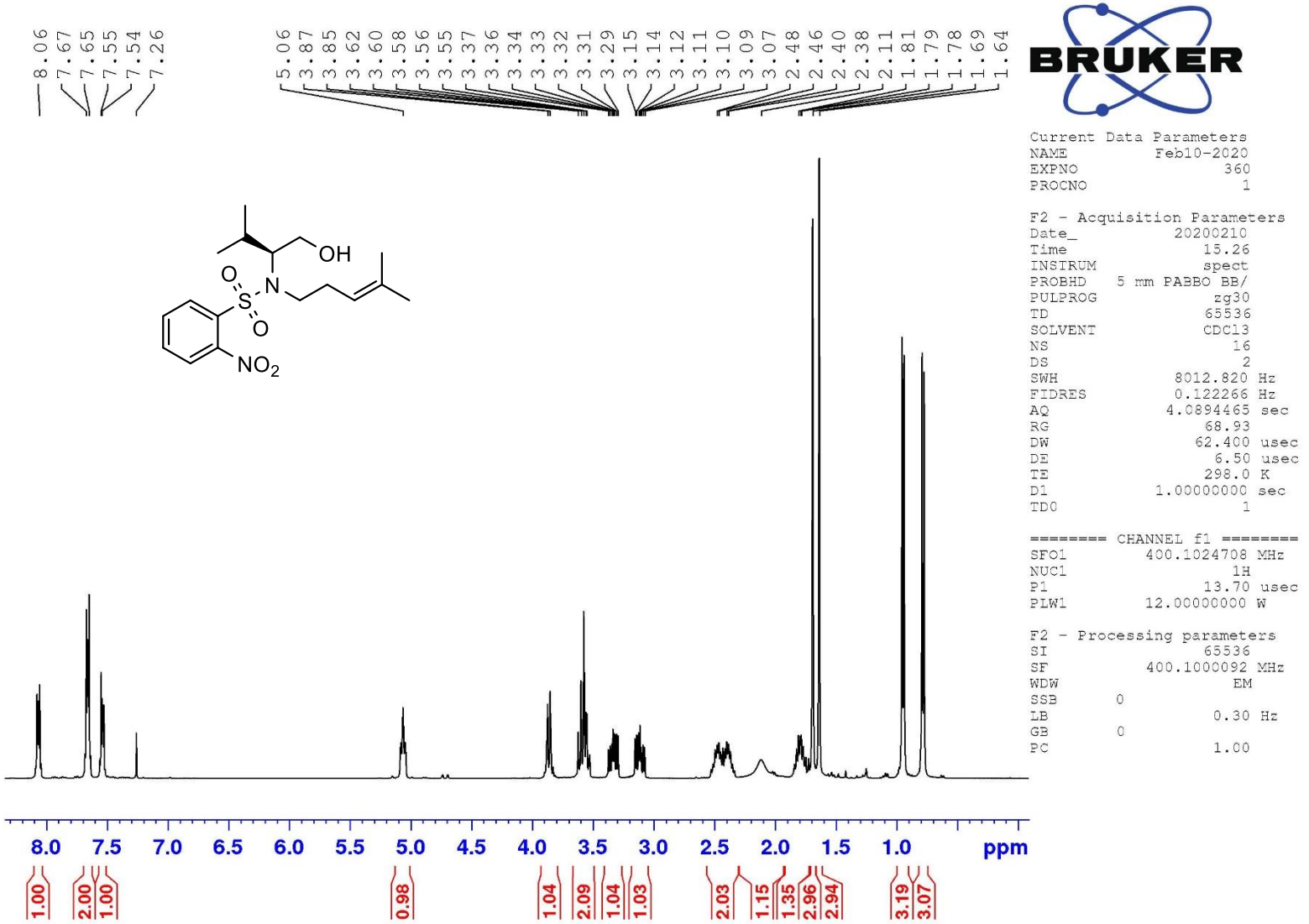
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 205.35
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6152851 MHz
NUC1 13C
P1 10.00 usec
PLW1 48.00000000 W

======== CHANNEL f2 ========
SFO2 400.1016004 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.27805999 W
PLW13 0.22522999 W

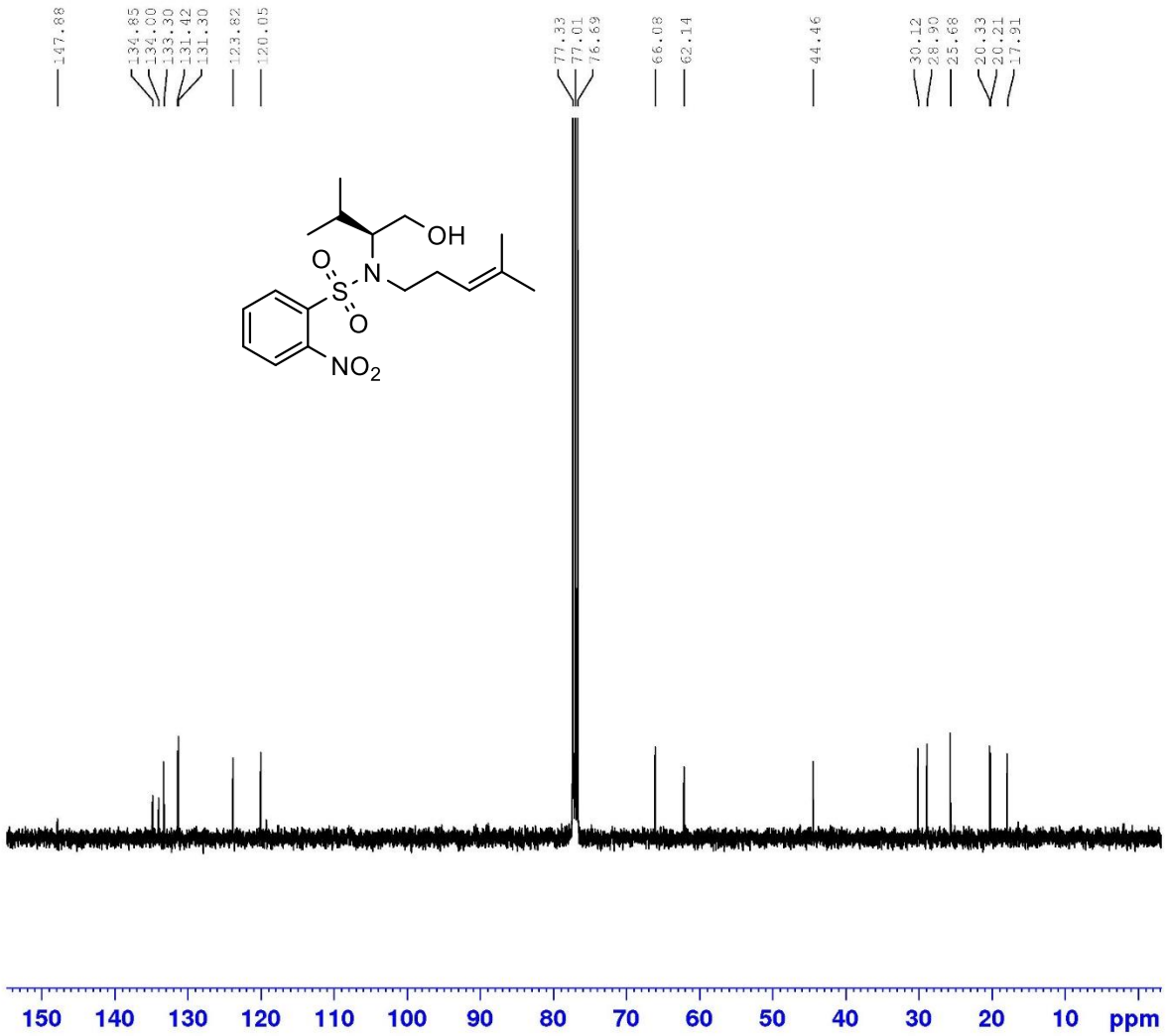
F2 - Processing parameters
SI 32768
SF 100.6052239 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{13}C-NMR **27a**



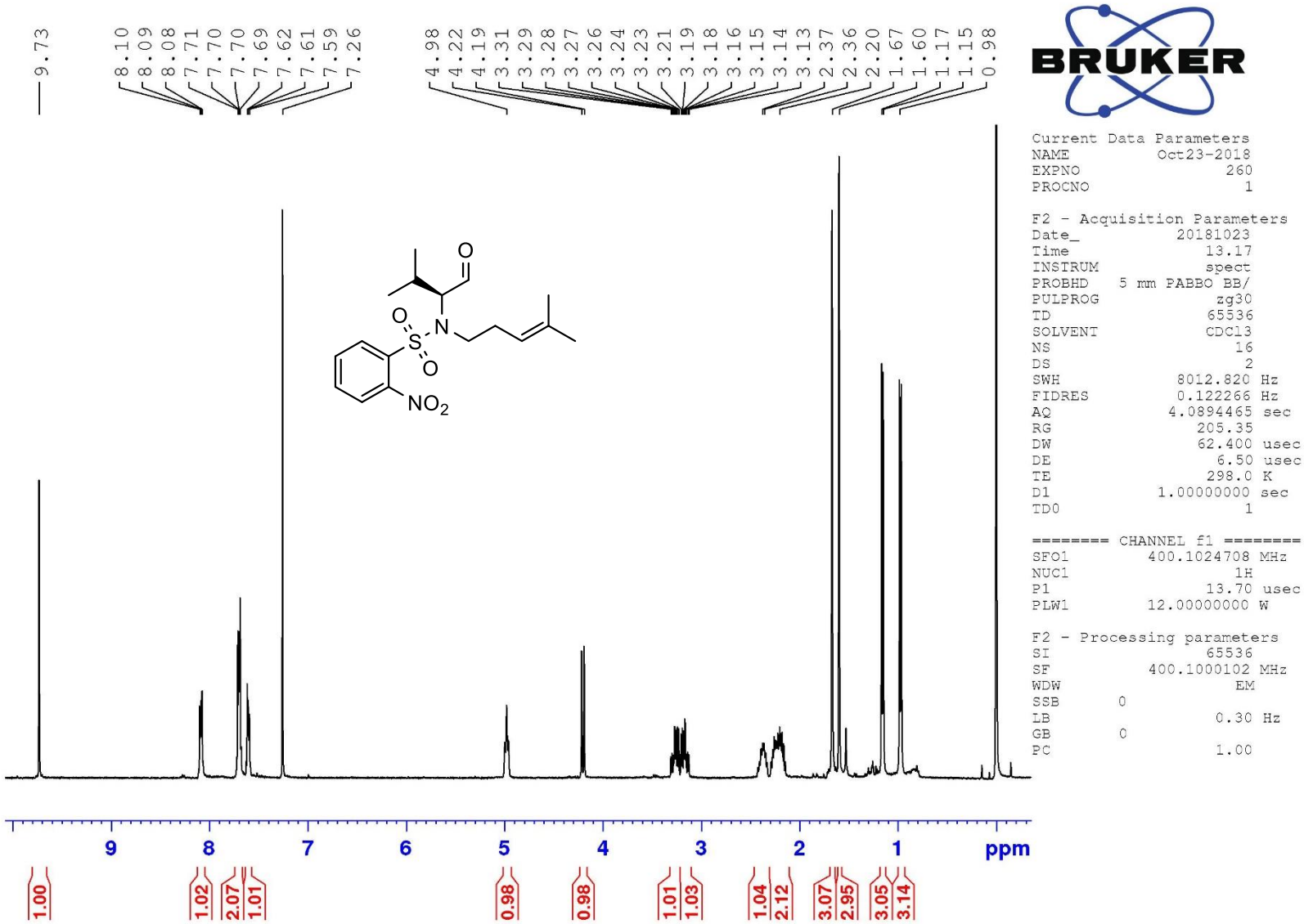

```
Current Data Parameters
NAME          Feb10-2020
EXPNO                360
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200210
Time               15.26
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 68.93
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1024708 MHz
NUC1                  1H
P1                 13.70 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1000092 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR **28a**

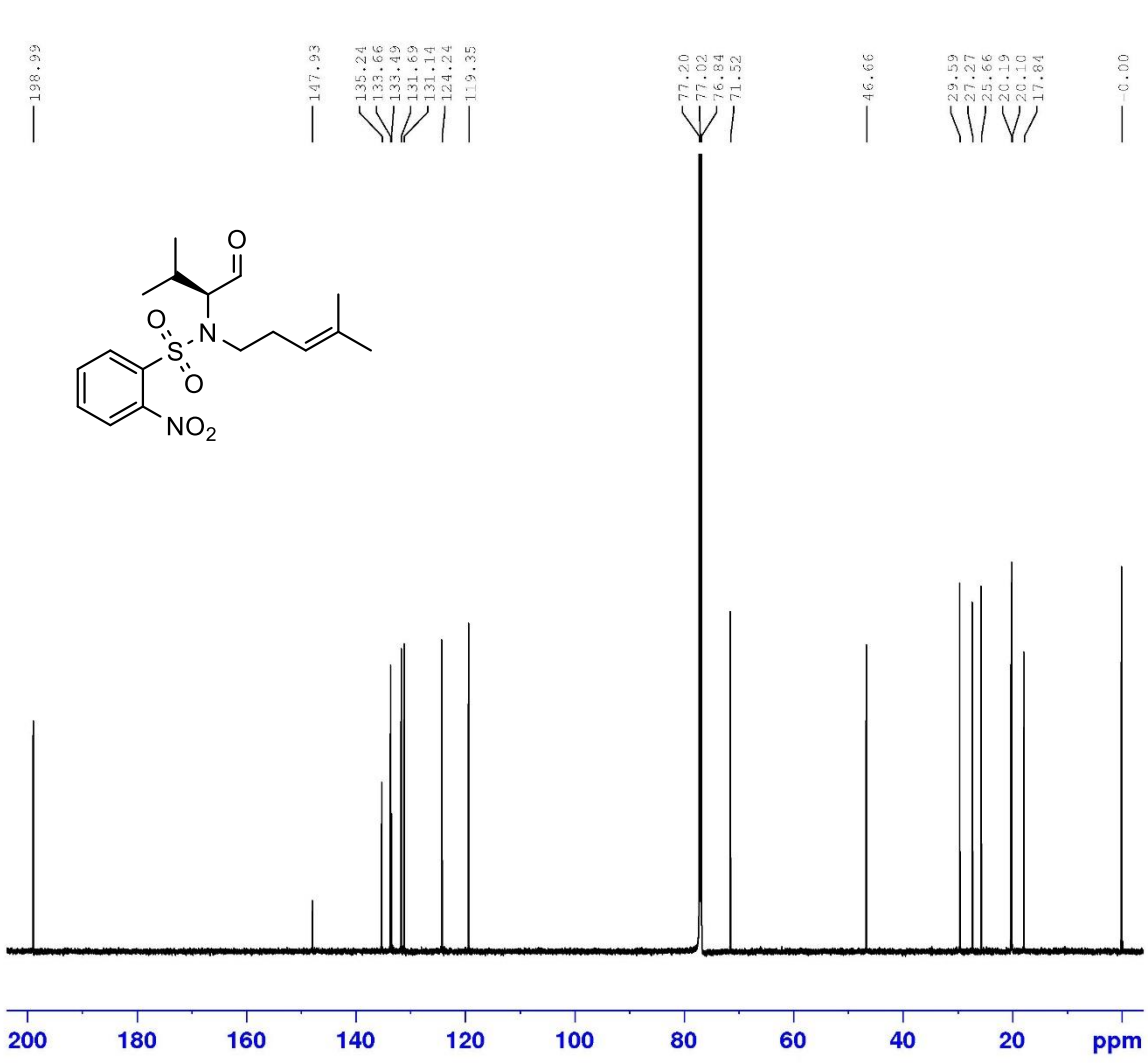



```
Current Data Parameters
NAME          Jan20-2020
EXPNO                731
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200120
Time               22.51
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               205.35
DW               20.800 usec
DE                6.50 usec
TE               298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6152851 MHz
NUC1               13C
P1               10.00 usec
PLW1        48.00000000 W

======== CHANNEL f2 ========
SFO2        400.1016004 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2        12.00000000 W
PLW12        0.27805999 W
PLW13        0.22522999 W

F2 - Processing parameters
SI                32768
SF          100.6052250 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C-NMR **28a**

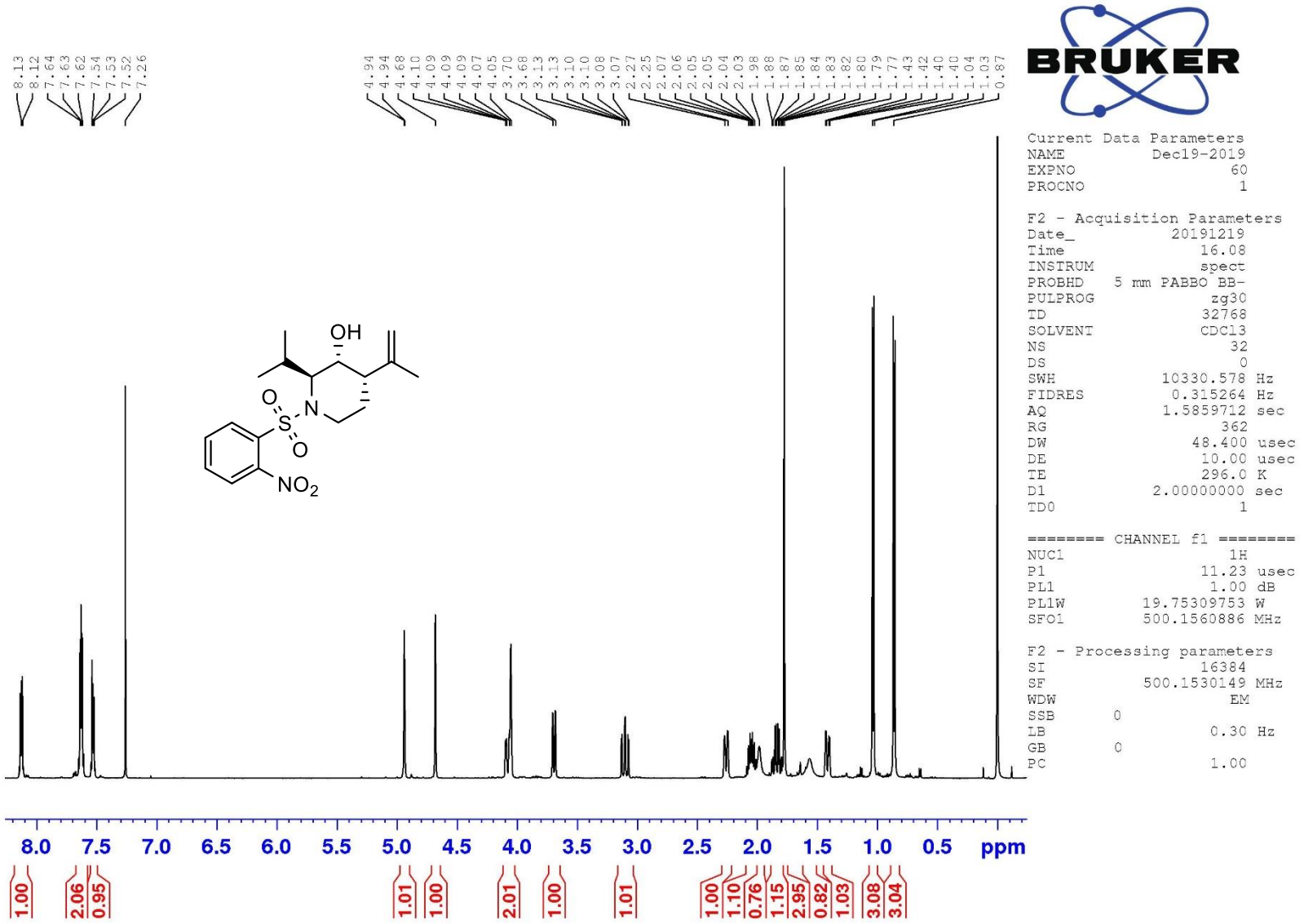

9.73
8.10
8.09
8.08
7.71
7.70
7.70
7.69
7.62
7.61
7.59
7.26
4.98
4.22
4.19
3.31
3.29
3.28
3.27
3.26
3.24
3.23
3.21
3.19
3.18
3.16
3.15
3.14
3.13
2.37
2.36
2.20
1.67
1.60
1.17
1.15
0.98
BRUKER
Current Data Parameters
NAME Oct23-2018
EXPNO 260
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181023
Time 13.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 205.35
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1024708 MHz
NUC1 1H
P1 13.70 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1000102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
S
N
O
NO2
9
8
7
6
5
4
3
2
1
ppm
1.00
1.02
2.07
1.01
0.98
0.98
1.01
1.03
1.04
2.12
3.07
2.95
3.05
3.14

^{1}H-NMR **29a**




```
Current Data Parameters
NAME          Oct25-2018
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181025
Time               13.07
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1               13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056634 MHz
WDW                EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

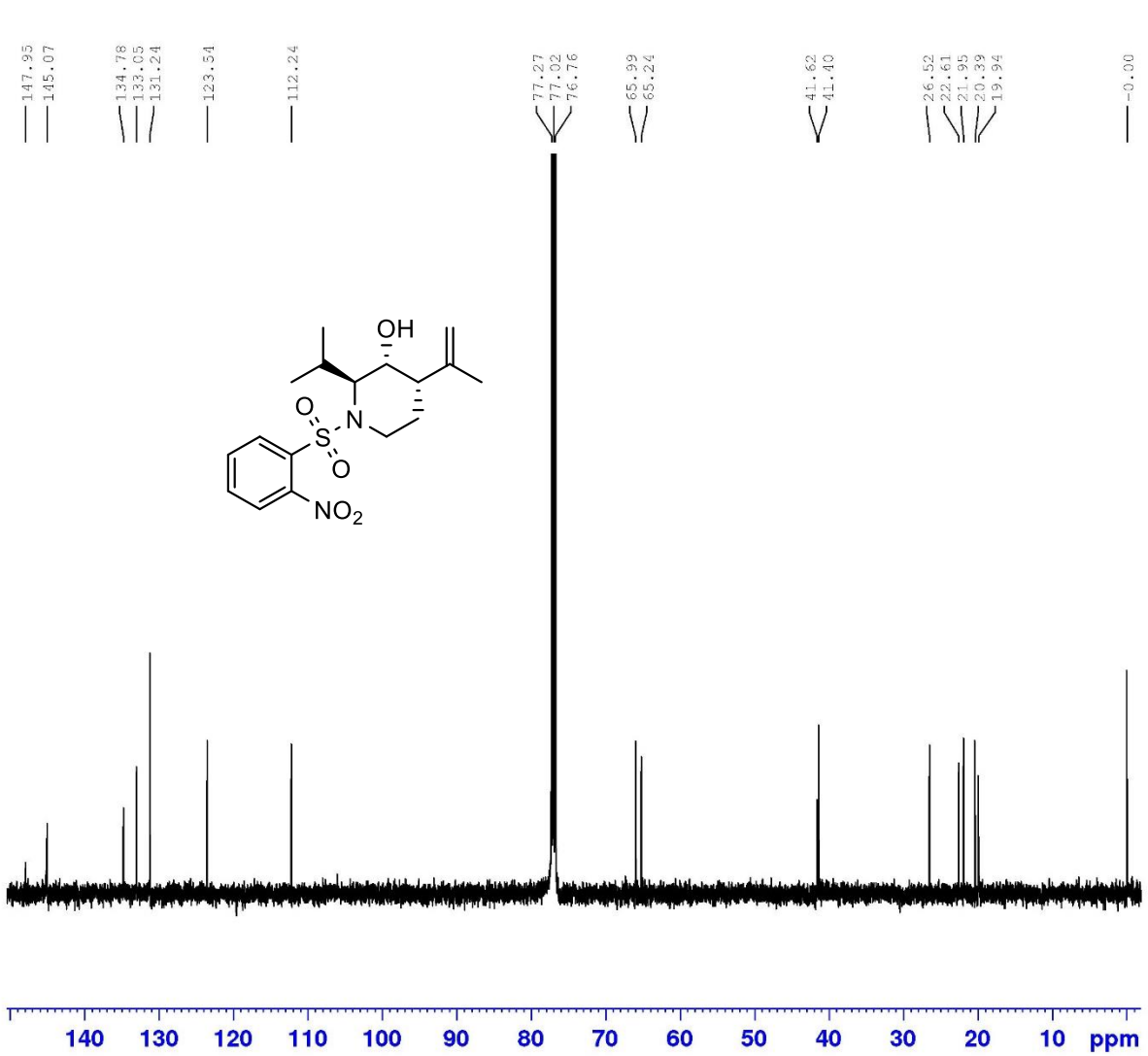
^{13}C-NMR **29a**



BRUKER

```
Current Data Parameters
NAME          Dec19-2019
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20191219
Time               16.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES           0.315264 Hz
AQ             1.5859712 sec
RG                   362
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530149 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

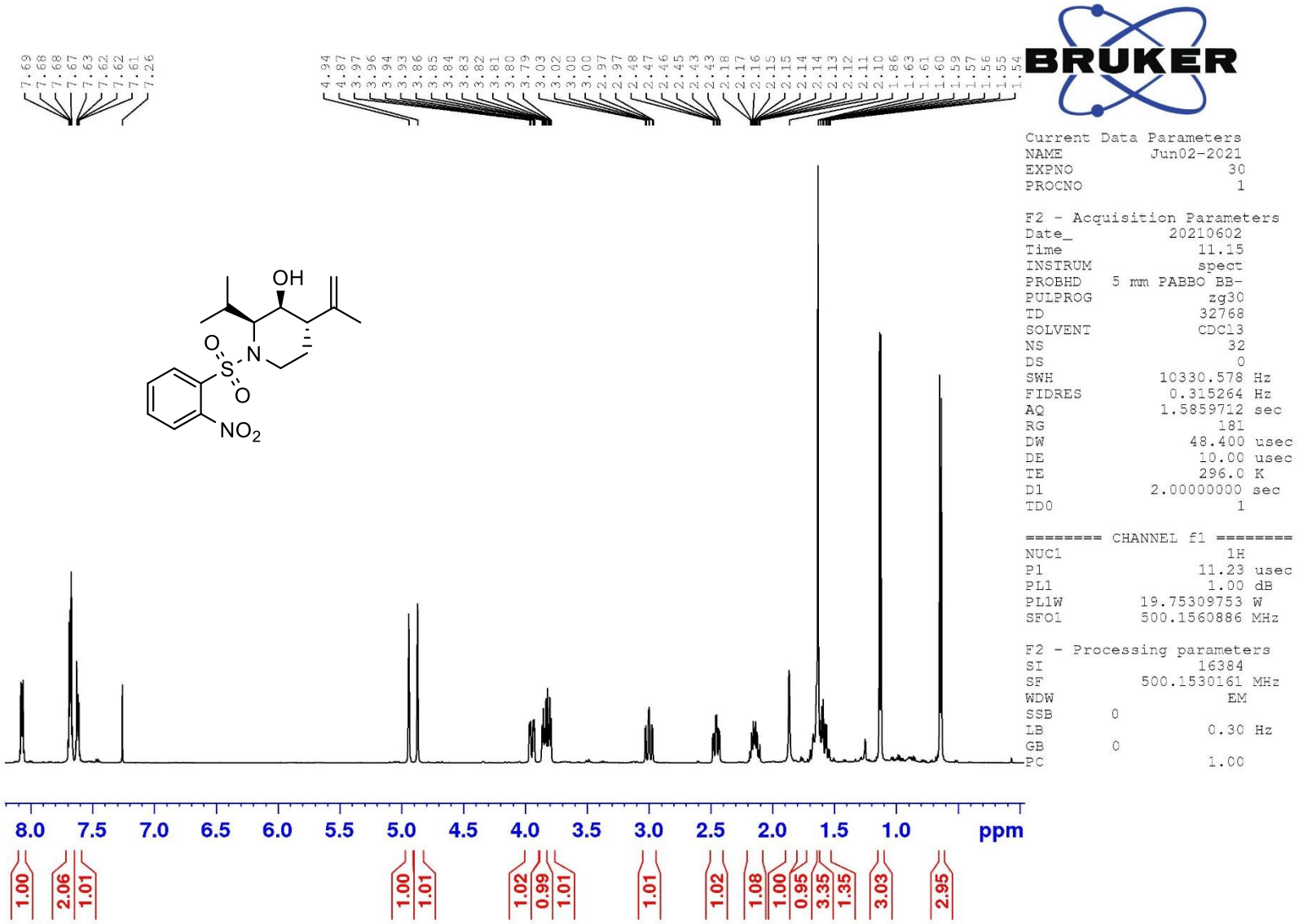
^{1}H-NMR (*R*,*S*)-**30a**



BRUKER

```
Current Data Parameters
NAME          Dec19-2019
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20191219
Time               17.01
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2300
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF         125.7635738 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

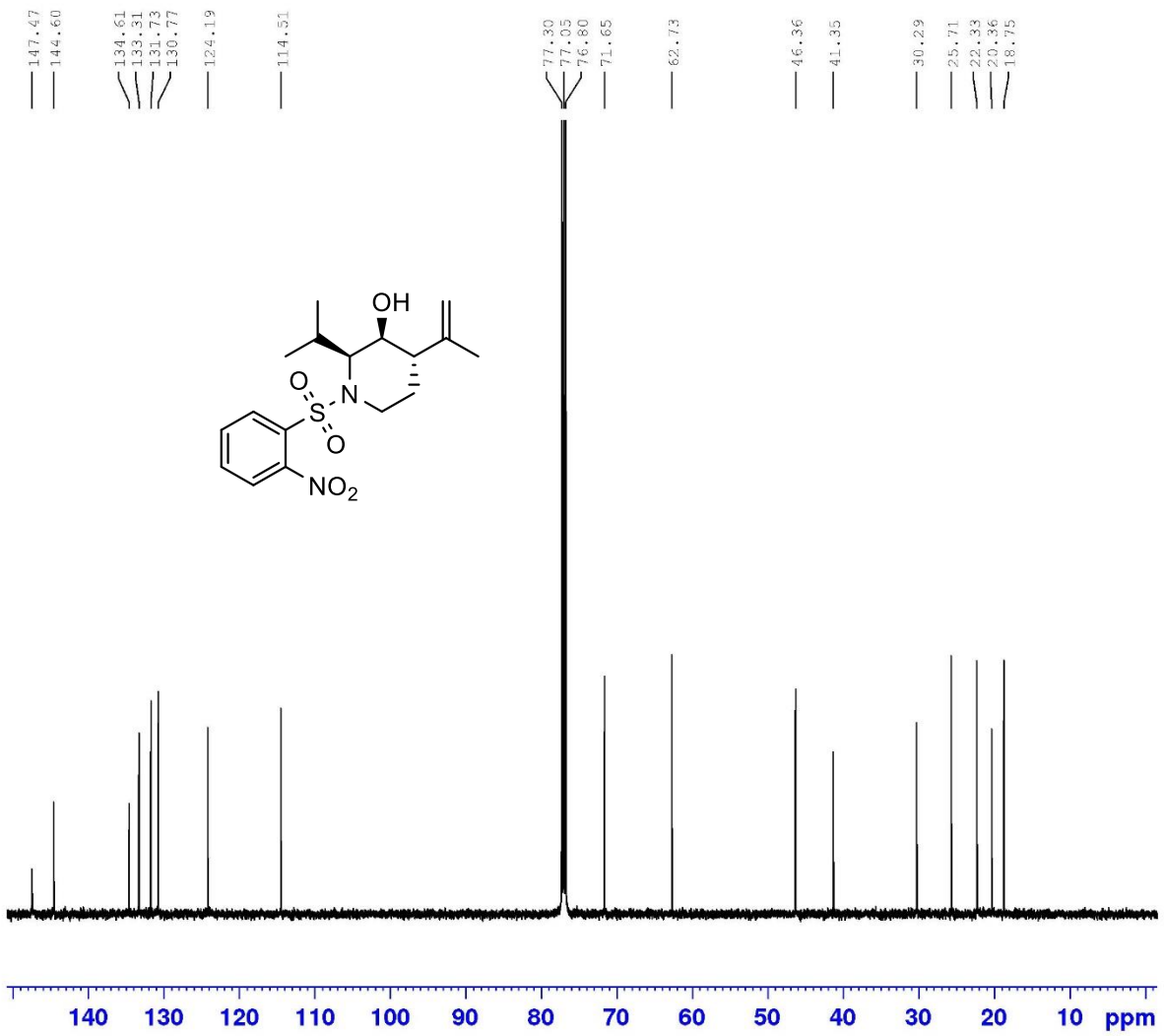
^{13}C-NMR (*R*,*S*)-**30a**



BRUKER

```
Current Data Parameters
NAME          Jun02-2021
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210602
Time               11.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   181
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530161 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

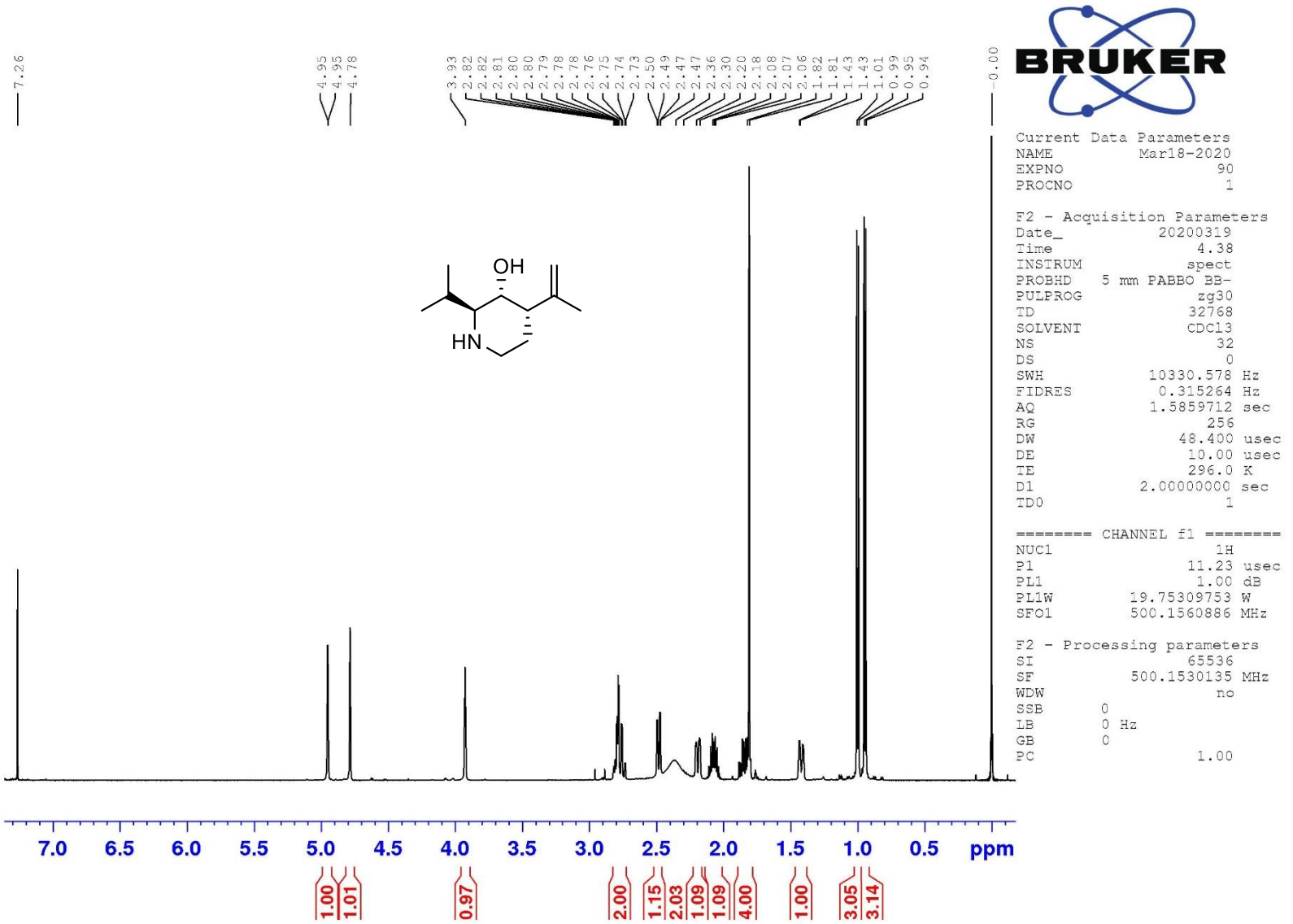
^{1}H-NMR (*S*,*S*)-**30a**



BRUKER

```
Current Data Parameters
NAME                  Jun02-2021
EXPNO                         31
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20210602
Time                       12.09
INSTRUM                    spect
PROBHD    5 mm PABBO BB-
PULPROG                  zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          1024
DS                             4
SWH                    32894.738 Hz
FIDRES                 0.501934 Hz
AQ                     0.9961472 sec
RG                          2580
DW                        15.200 usec
DE                         10.00 usec
TE                         296.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                        10.20 usec
PL1                        1.50 dB
PL1W                51.74793243 W
SFO1               125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2                 waltz16
NUC2                         1H
PCPD2                    100.00 usec
PL2                        1.00 dB
PL12                      19.99 dB
PL13                      21.00 dB
PL2W                19.75309753 W
PL12W                0.24925002 W
PL13W                0.19753097 W
SFO2               500.1550006 MHz

F2 - Processing parameters
SI                         32768
SF                 125.7635720 MHz
WDW                           EM
SSB          0
LB                         1.00 Hz
GB           0
PC                         1.40
```

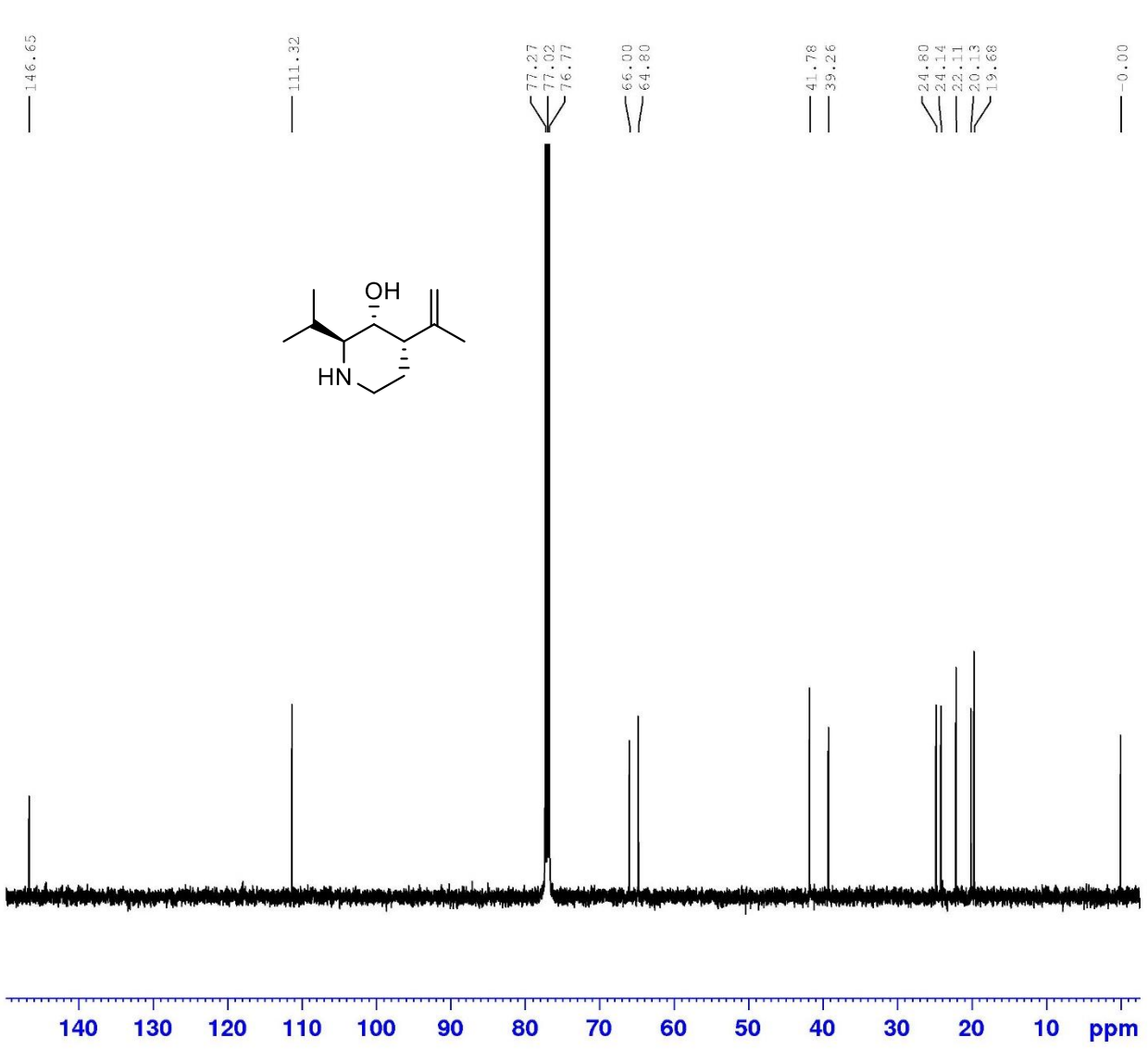
^{13}C-NMR (*S*,*S*)-**30a**



BRUKER

```
Current Data Parameters
NAME          Mar18-2020
EXPNO                 90
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200319
Time                4.38
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   256
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 65536
SF           500.1530135 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

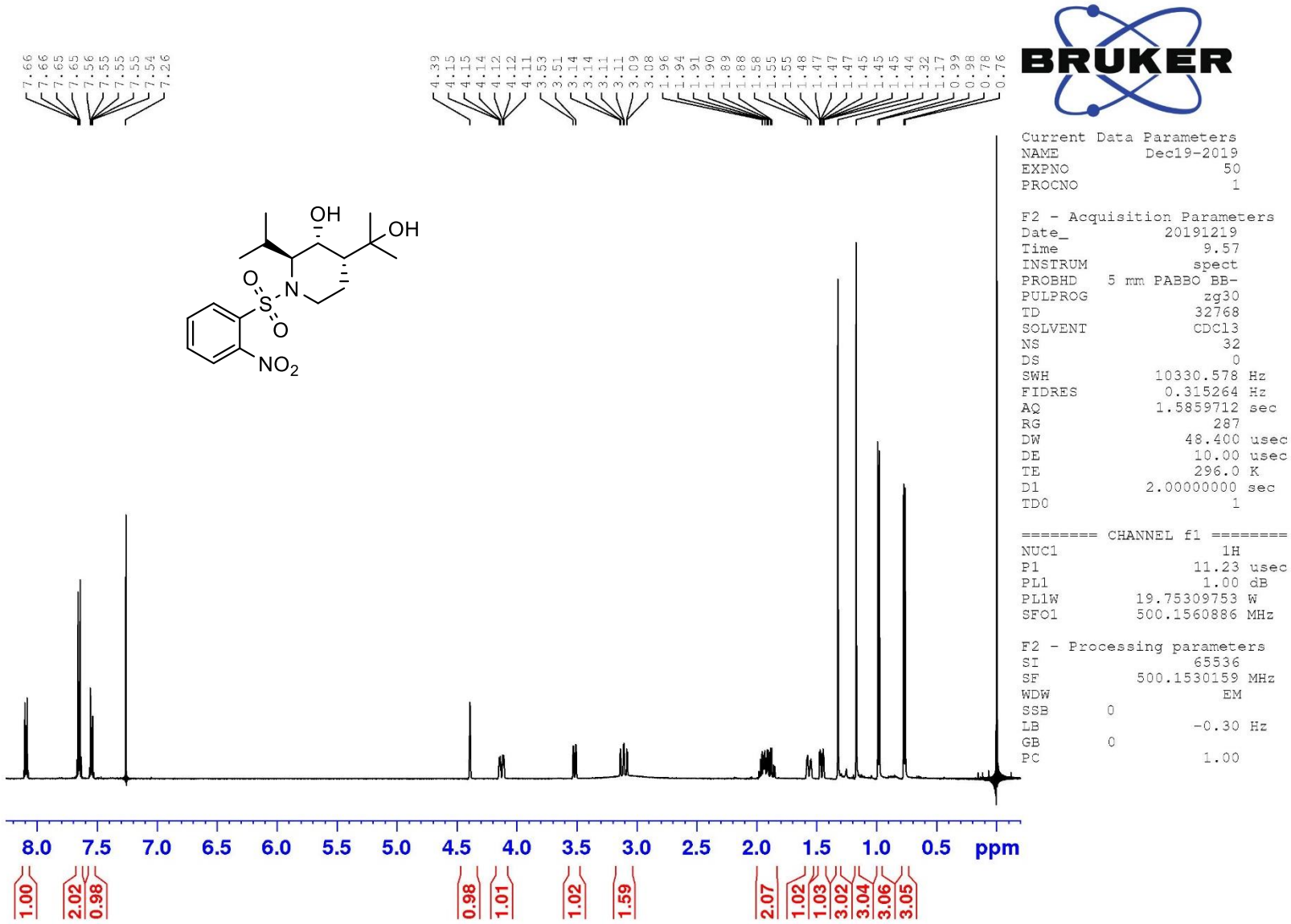
^{1}H-NMR (*R*,*S*)-**32a**



BRUKER

```
Current Data Parameters
NAME          Mar18-2020
EXPNO                 91
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200319
Time                5.31
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635736 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

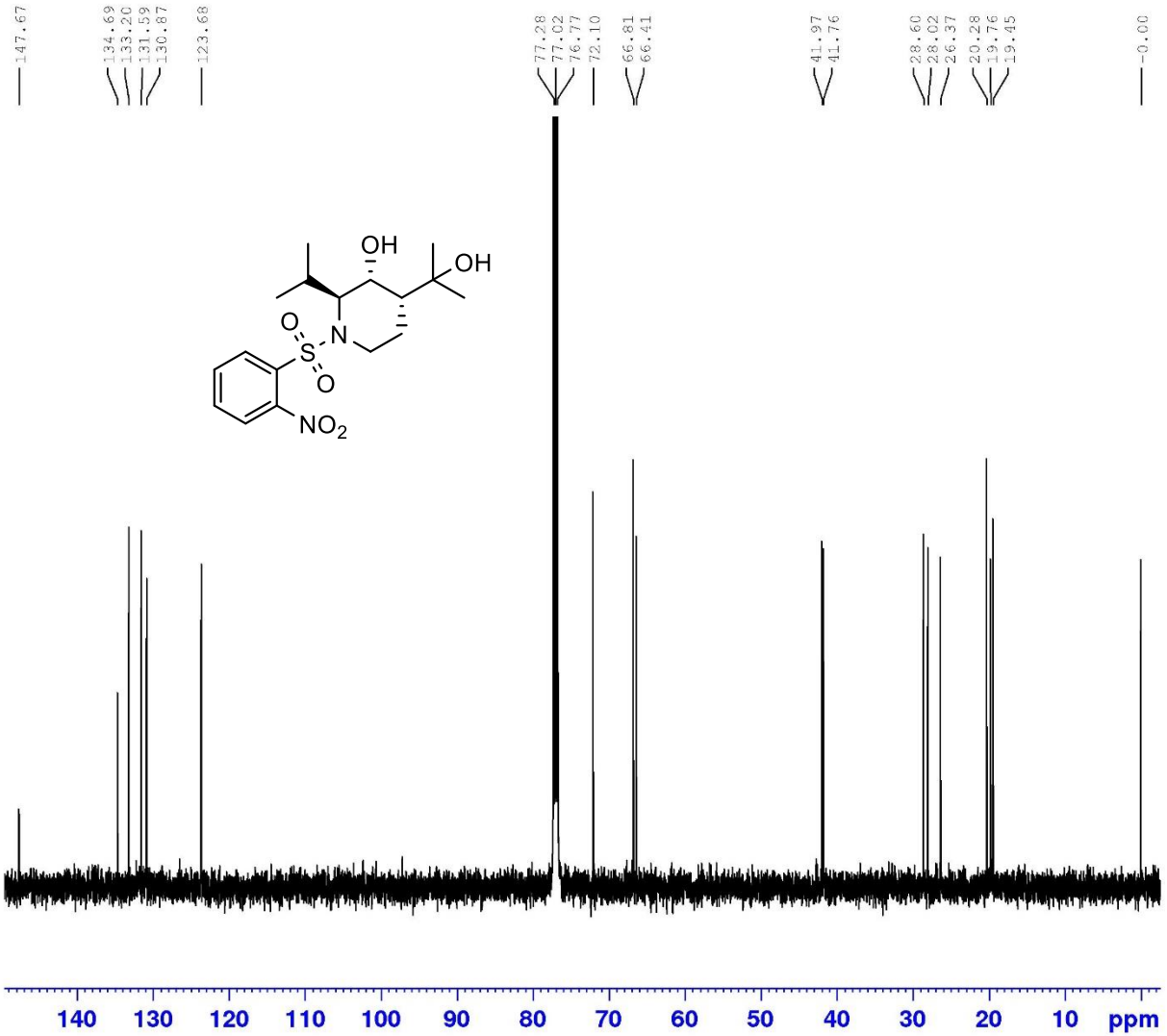
^{13}C-NMR (*R*,*S*)-**32a**

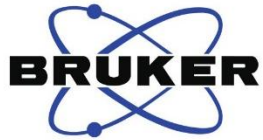


BRUKER

```
Current Data Parameters
NAME          Dec19-2019
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20191219
Time                9.57
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   287
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 65536
SF          500.1530159 MHz
WDW                   EM
SSB       0
LB                -0.30 Hz
GB        0
PC                 1.00
```

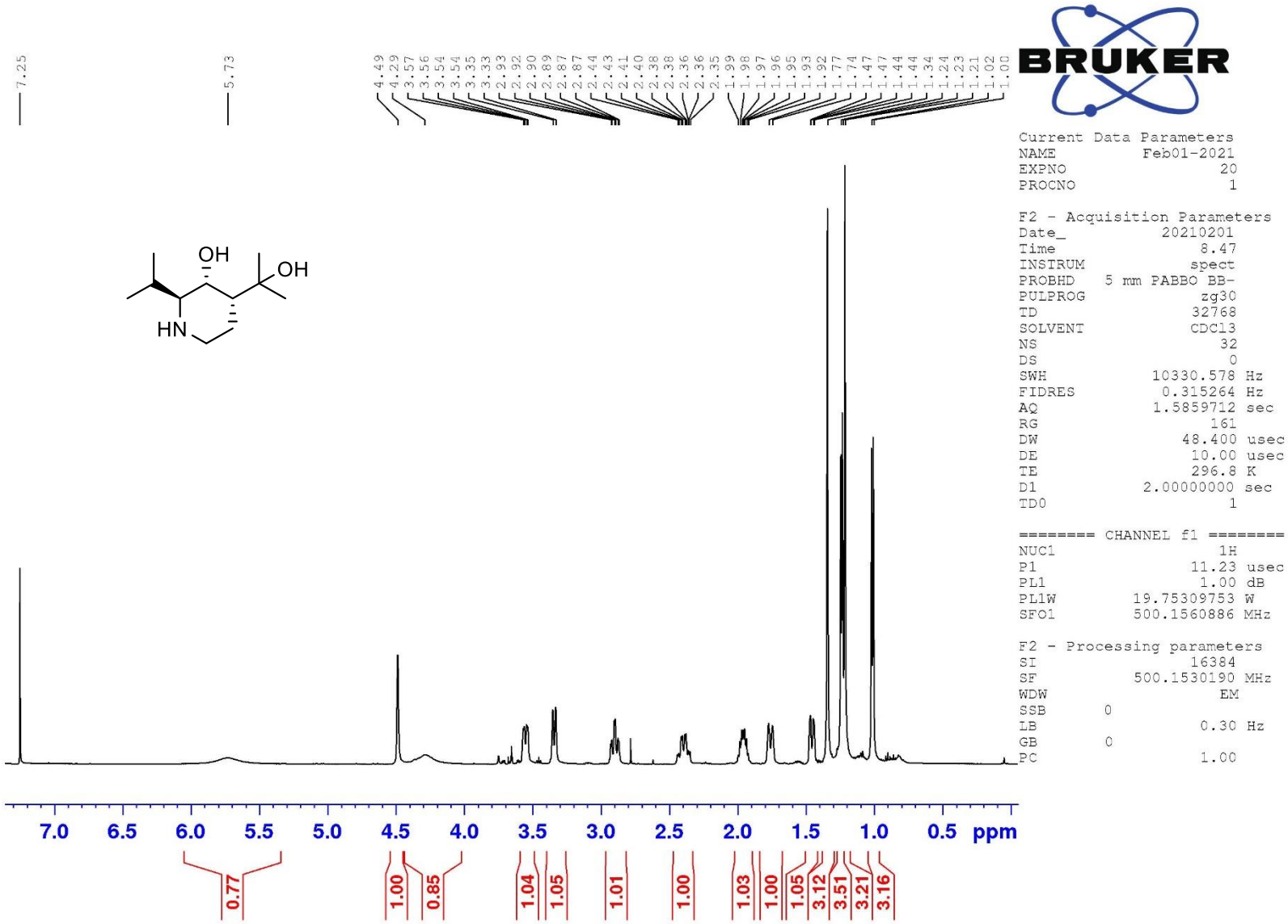
^{1}H-NMR (*R*,*R*)-**31a**





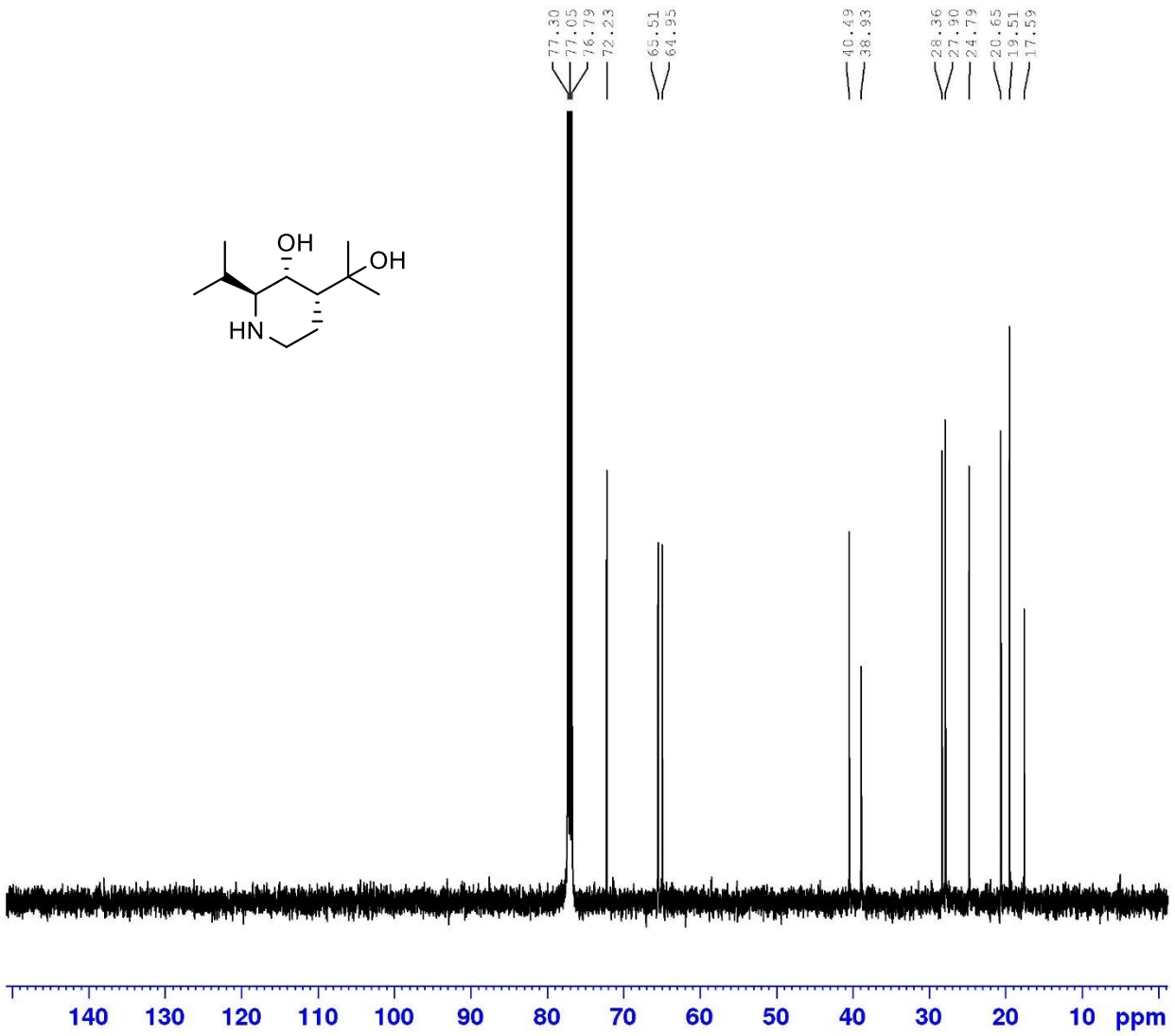

```
Current Data Parameters
NAME          Dec19-2019
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20191219
Time               10.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF         125.7635739 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*R*,*R*)-**31a**



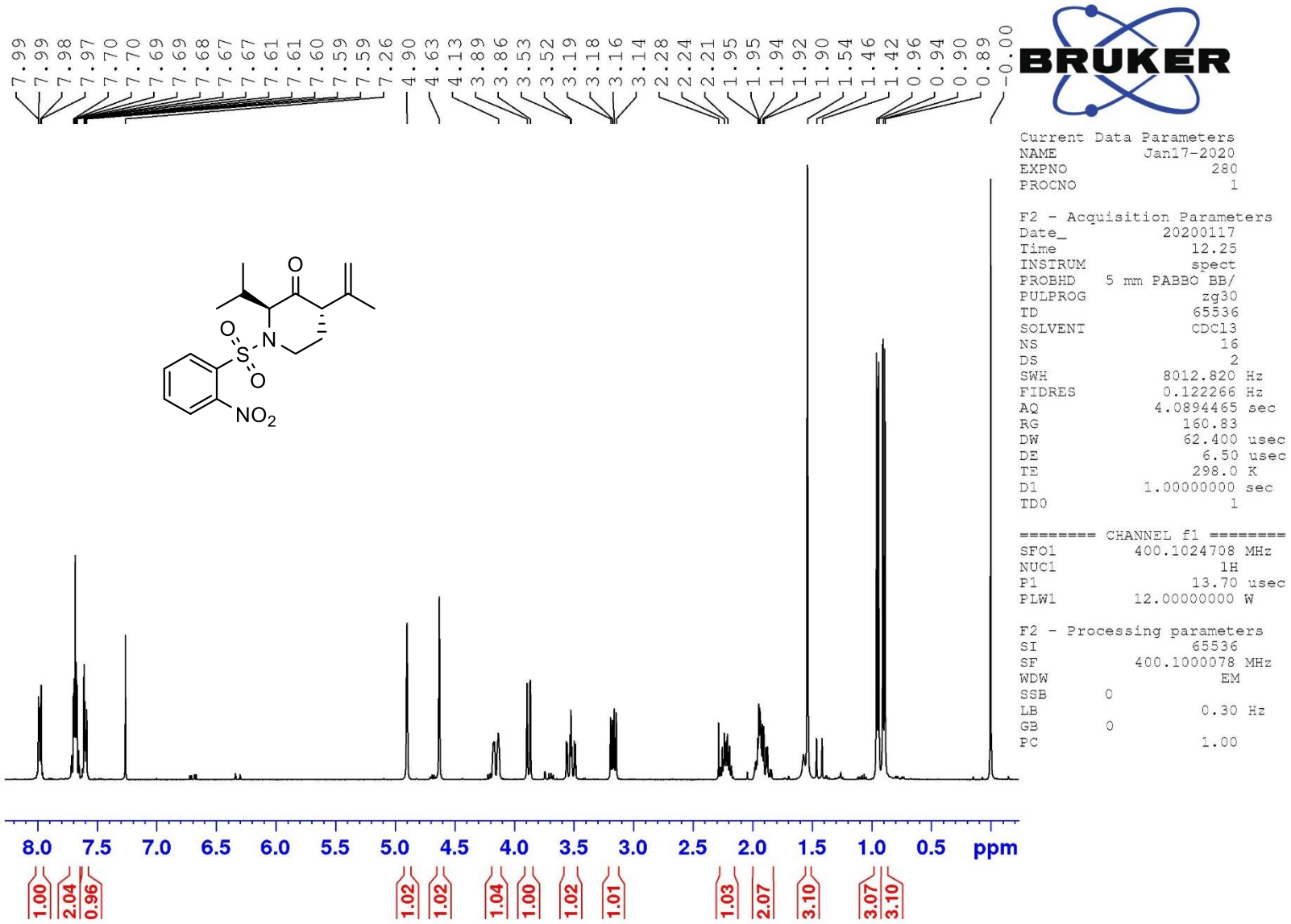

```
Current Data Parameters
NAME          Feb01-2021
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210201
Time                8.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   161
DW                48.400 usec
DE                10.00 usec
TE                296.8 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530190 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*R*,*R*)-**33a**

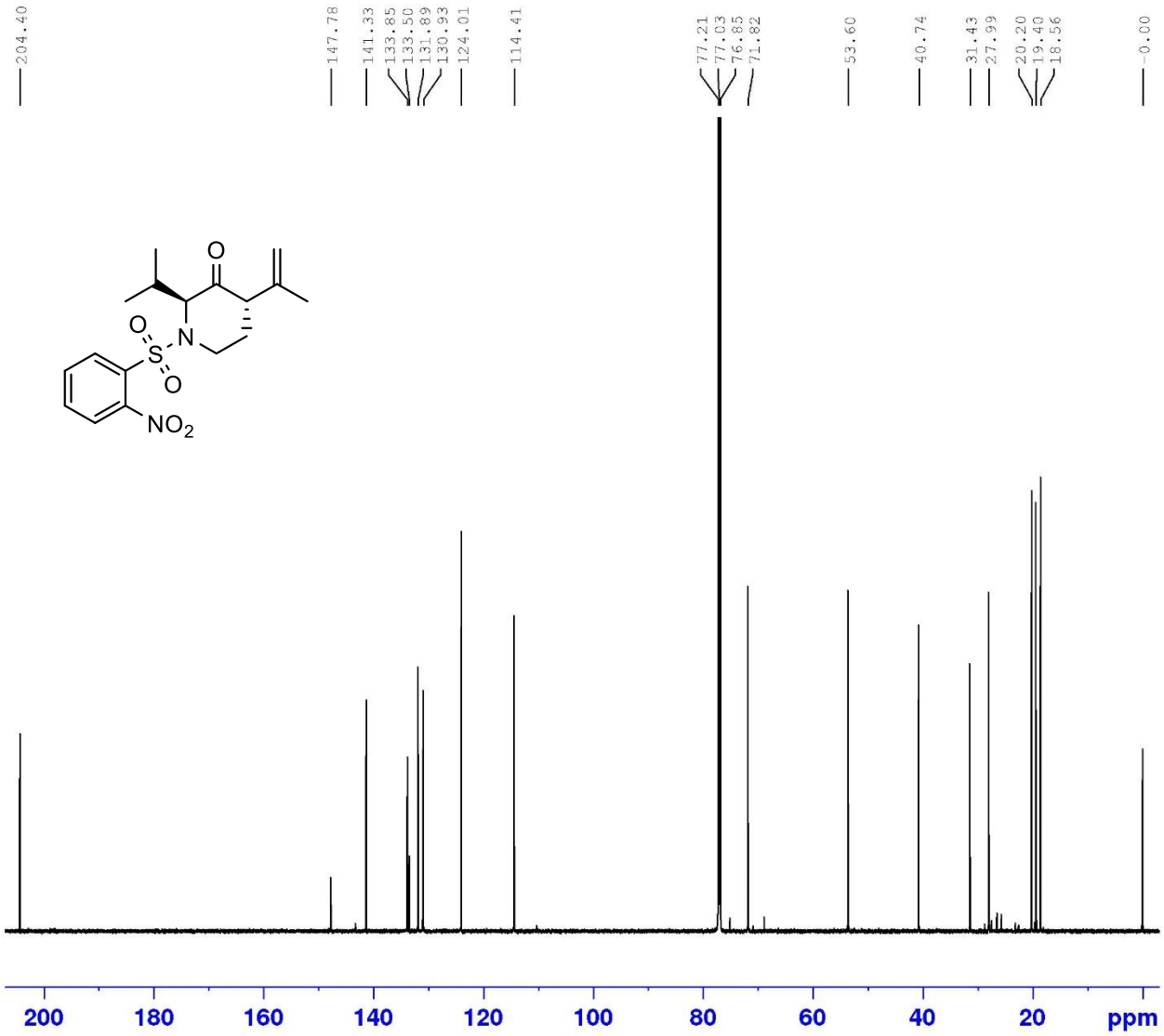



```
Current Data Parameters
NAME          Feb01-2021
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210201
Time                9.41
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*R*,*R*)-**31a**


7.99 7.99 7.98 7.97 7.70 7.70 7.69 7.69 7.68 7.67 7.67 7.61 7.61 7.60 7.59 7.59 7.26 4.90 4.63 4.13 3.89 3.86 3.53 3.52 3.19 3.18 3.16 3.14 2.28 2.24 2.21 1.95 1.95 1.94 1.92 1.90 1.54 1.46 1.42 0.96 0.94 0.90 0.89 -0.00
BRUKER
Current Data Parameters
NAME Jan17-2020
EXPNO 280
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200117
Time 12.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 160.83
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1024708 MHz
NUC1 1H
P1 13.70 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1000078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
S
N
O
NO2
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00 2.04 0.96 1.02 1.02 1.04 1.00 1.02 1.01 1.03 2.07 3.10 3.07 3.10

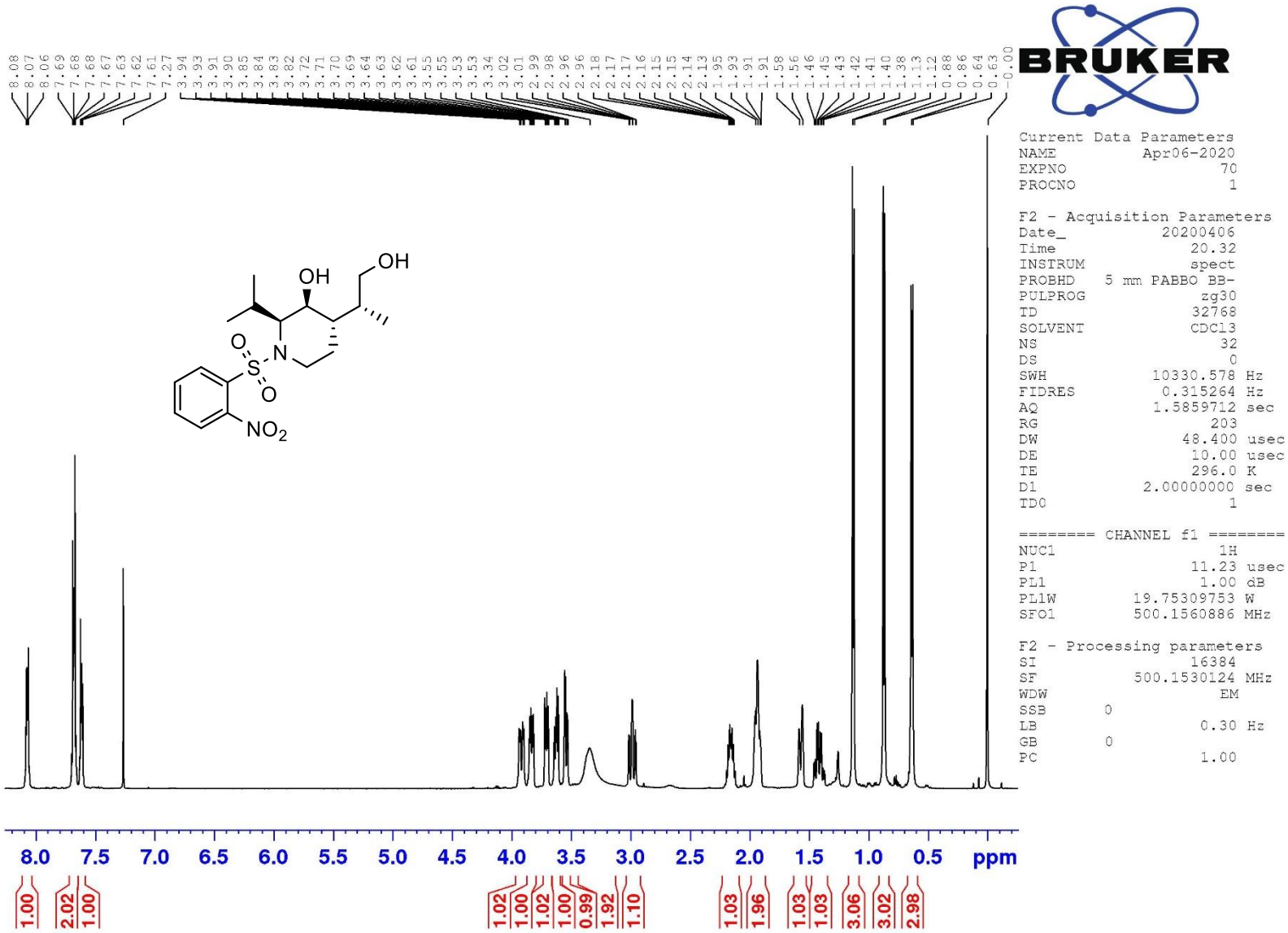
^{1}H-NMR (*S*)-**S2a**



BRUKER

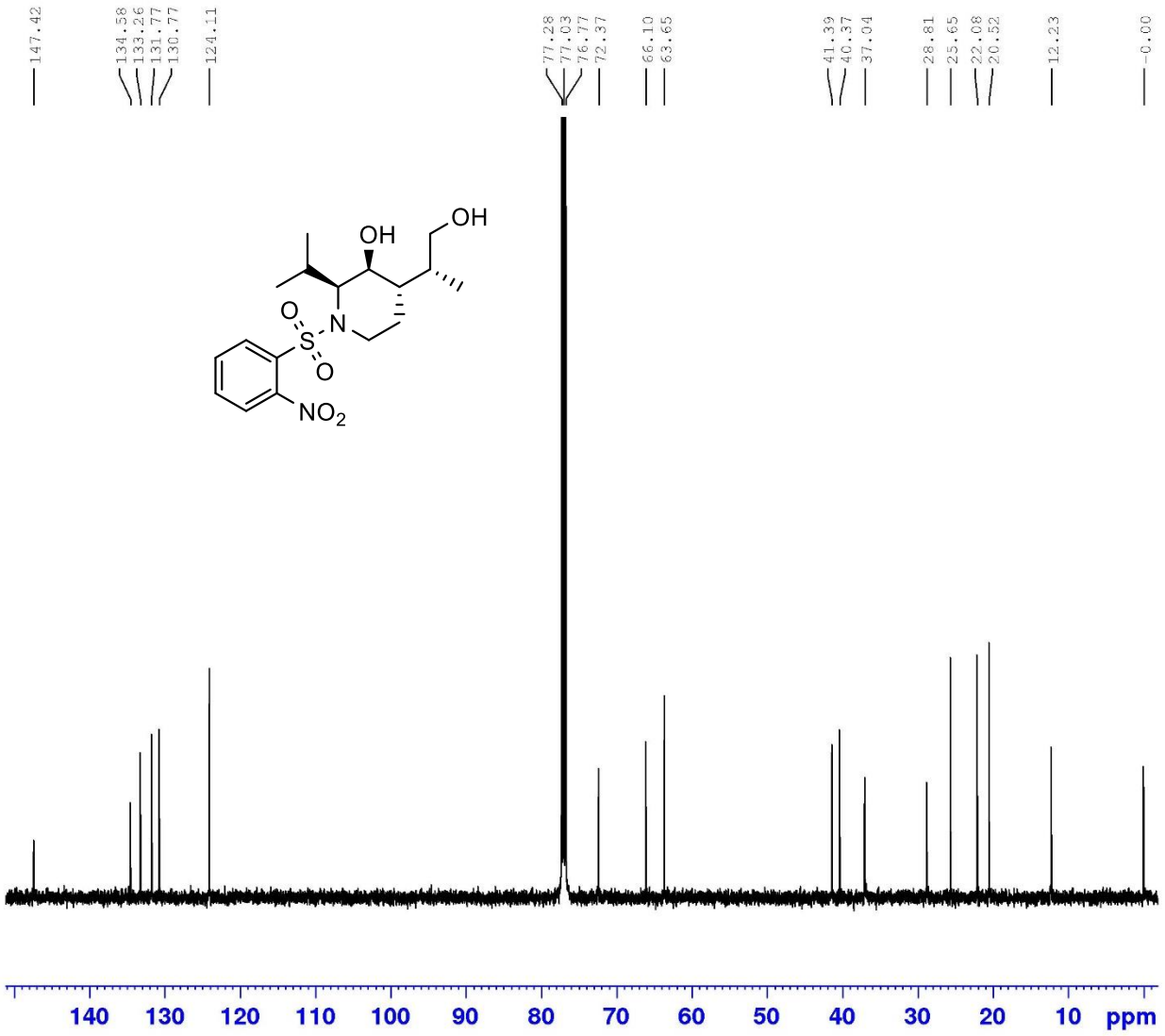
```
Current Data Parameters
NAME          Apr23-2020
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200423
Time               18.22
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG                182.53
DW                12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2           9.89999962 W
PLW12          0.15564001 W
PLW13          0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056637 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*S*)-**S2a**




```
Current Data Parameters
NAME          Apr06-2020
EXPNO                 70
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200406
Time               20.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   203
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530124 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*S*,*R*)-**34a**



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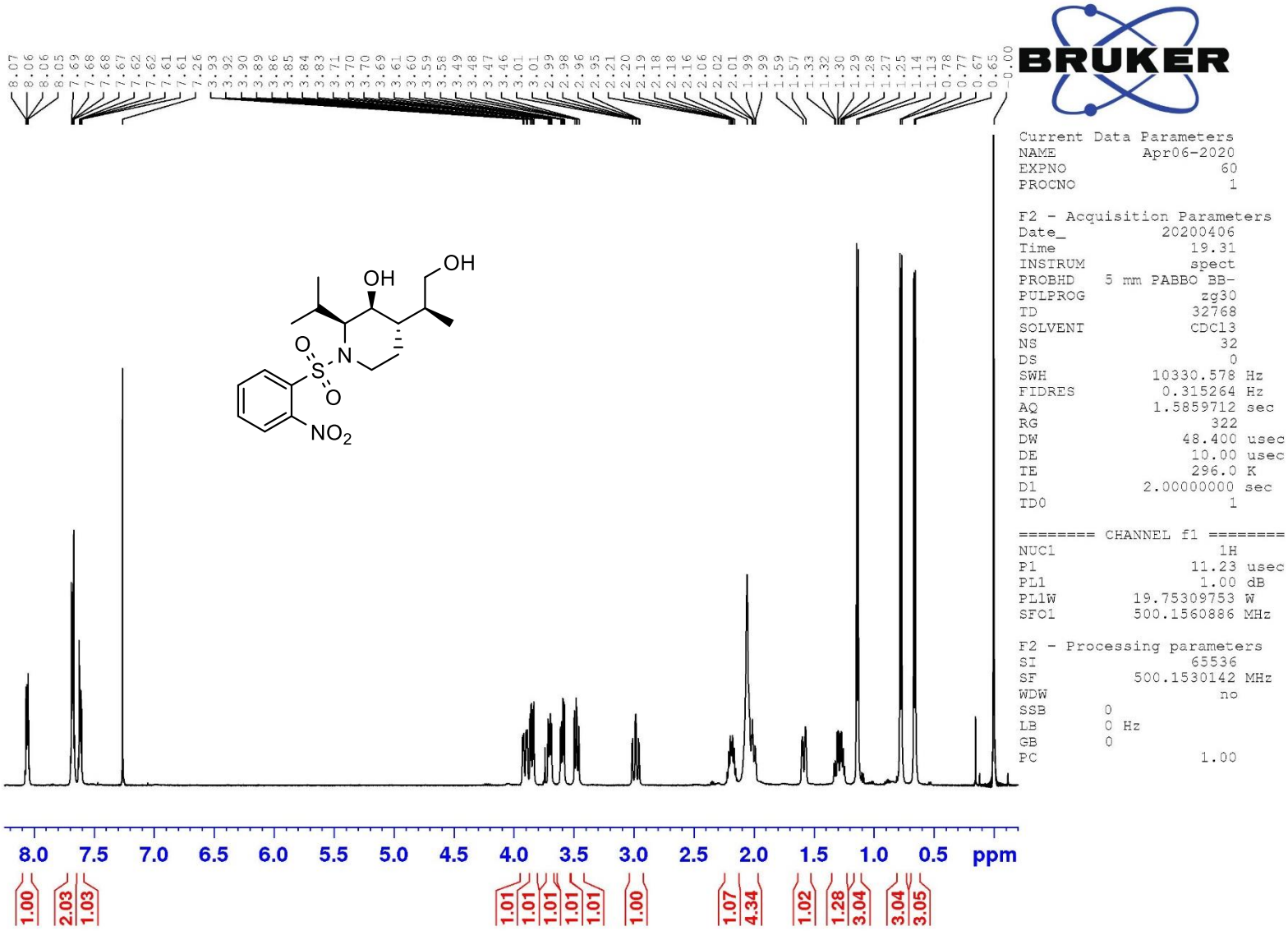
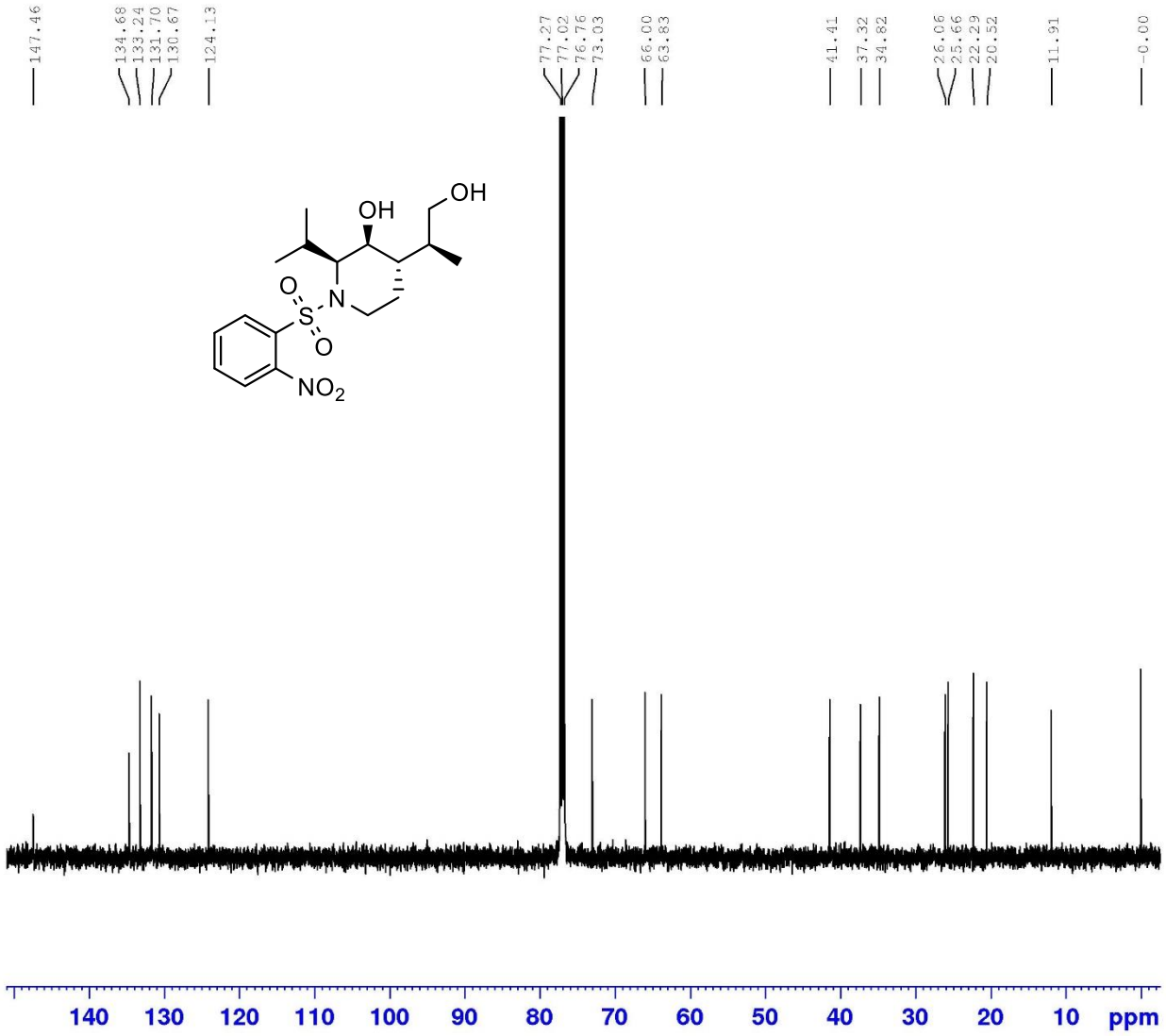
```
Current Data Parameters
NAME          Apr06-2020
EXPNO                 71
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200406
Time               21.25
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635745 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*S*,*S*,*R*)-**34a**




```
Current Data Parameters
NAME          Apr06-2020
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200406
Time               19.31
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 65536
SF          500.1530142 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

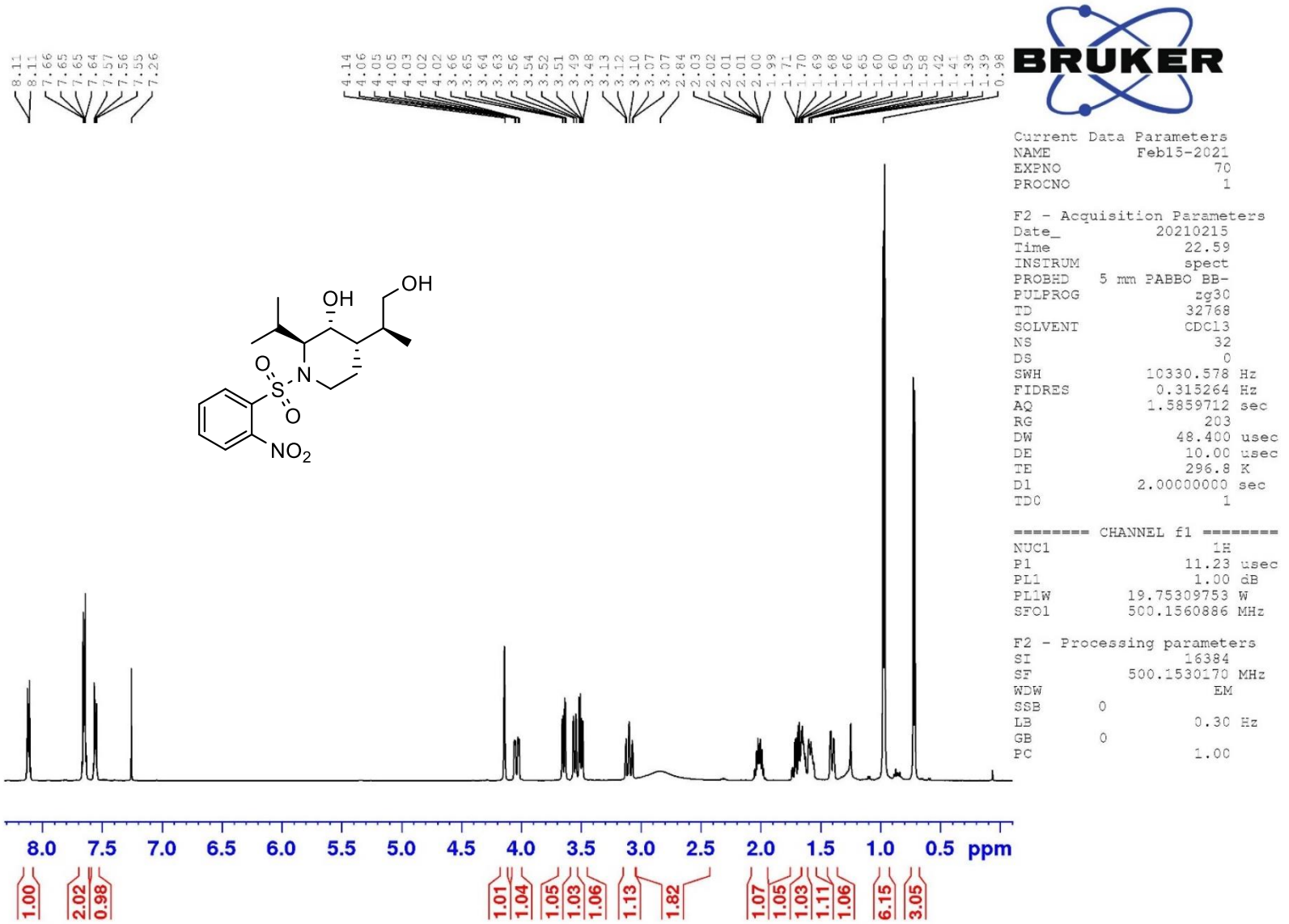
^{1}H-NMR (*S*,*S*,*S*)-**34a**



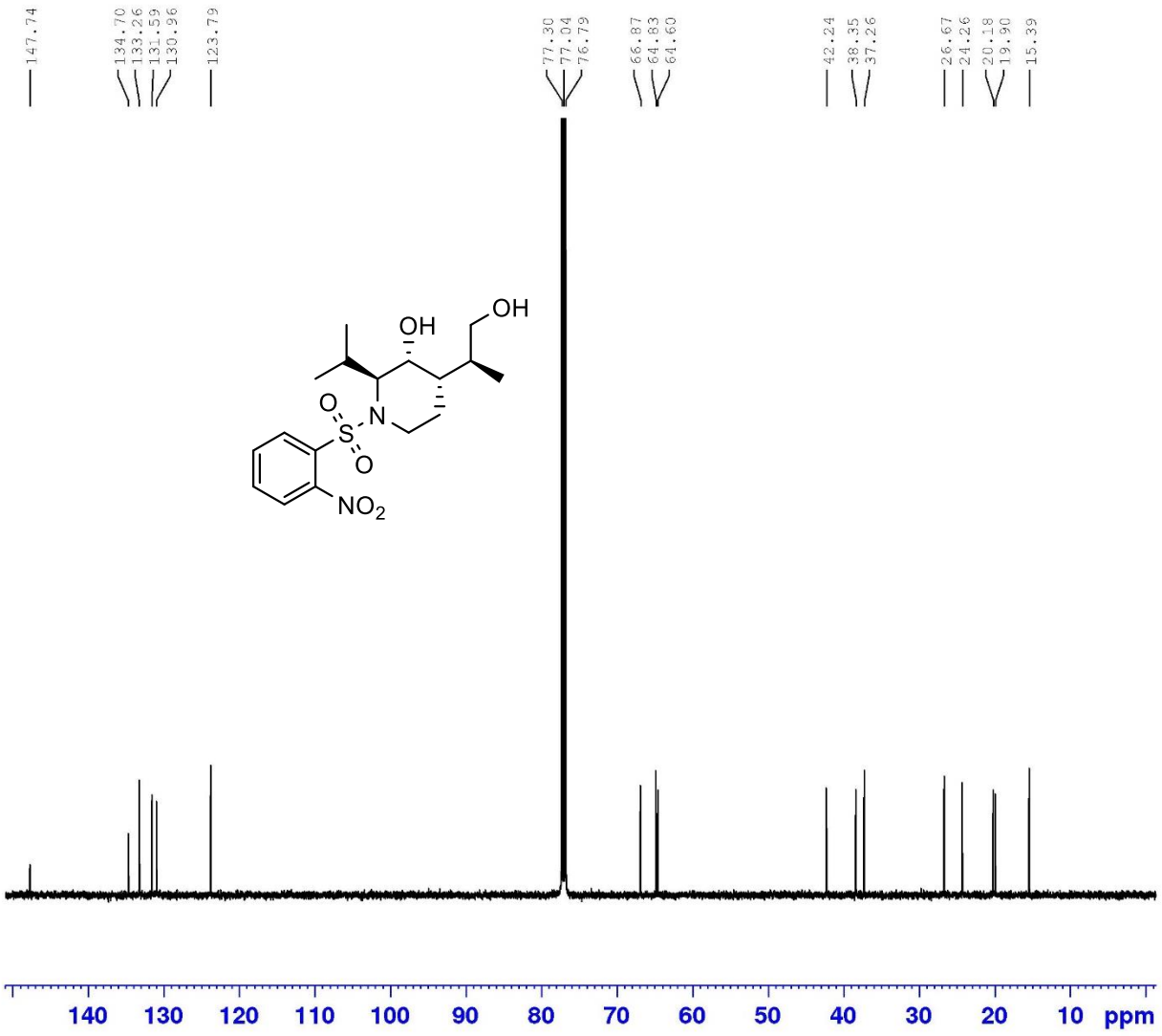
BRUKER

```
Current Data Parameters
NAME          Apr06-2020
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200406
Time               20.24
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635744 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*S*,*S*,*S*)-**34a**



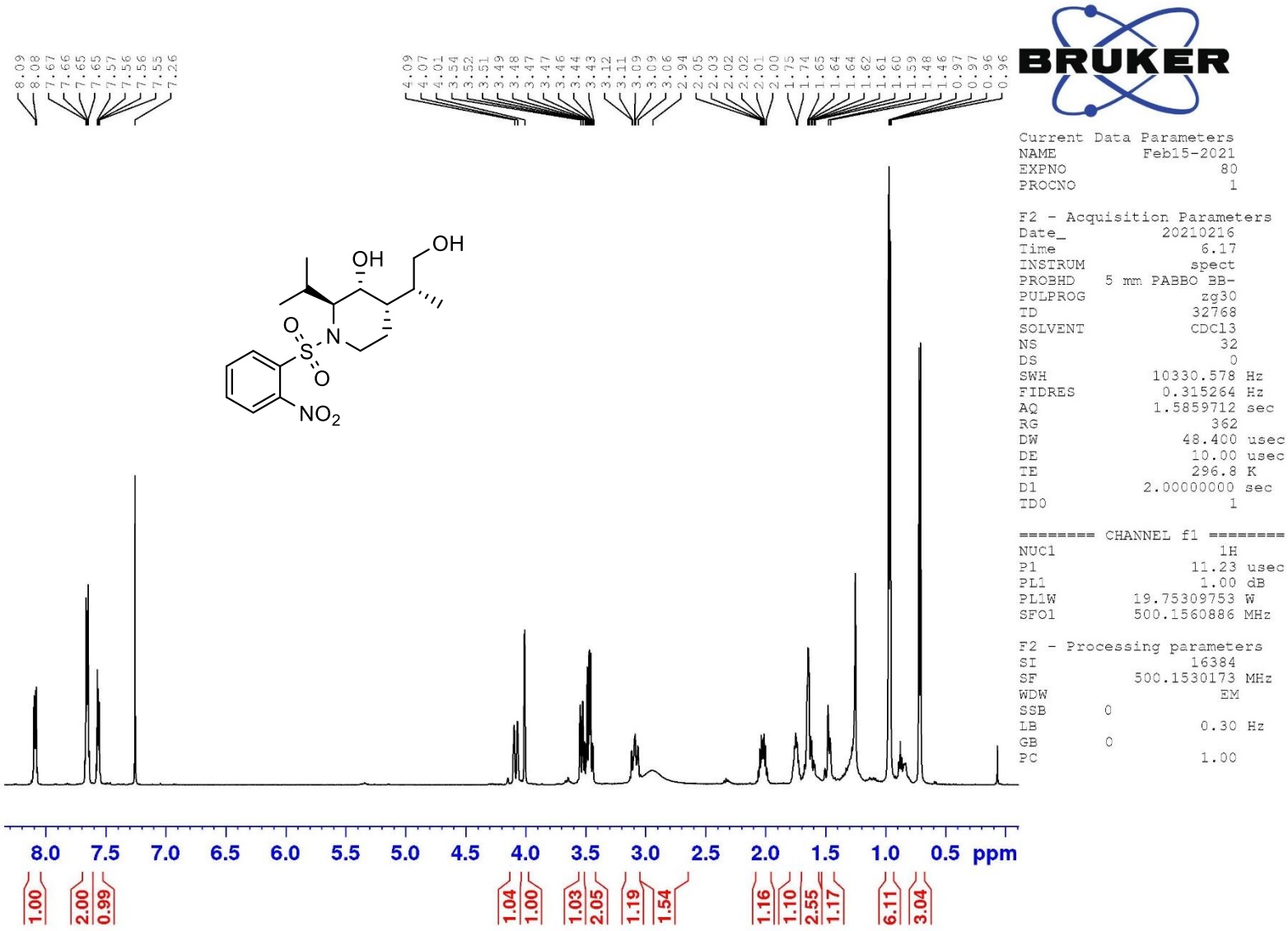
^{1}H-NMR (*R*,*S*,*S*)-**34a**



BRUKER

```
Current Data Parameters
NAME          Feb15-2021
EXPNO                 71
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210215
Time               23.52
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW                15.200 usec
DE                10.00 usec
TE                296.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.20 usec
PL1                  1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

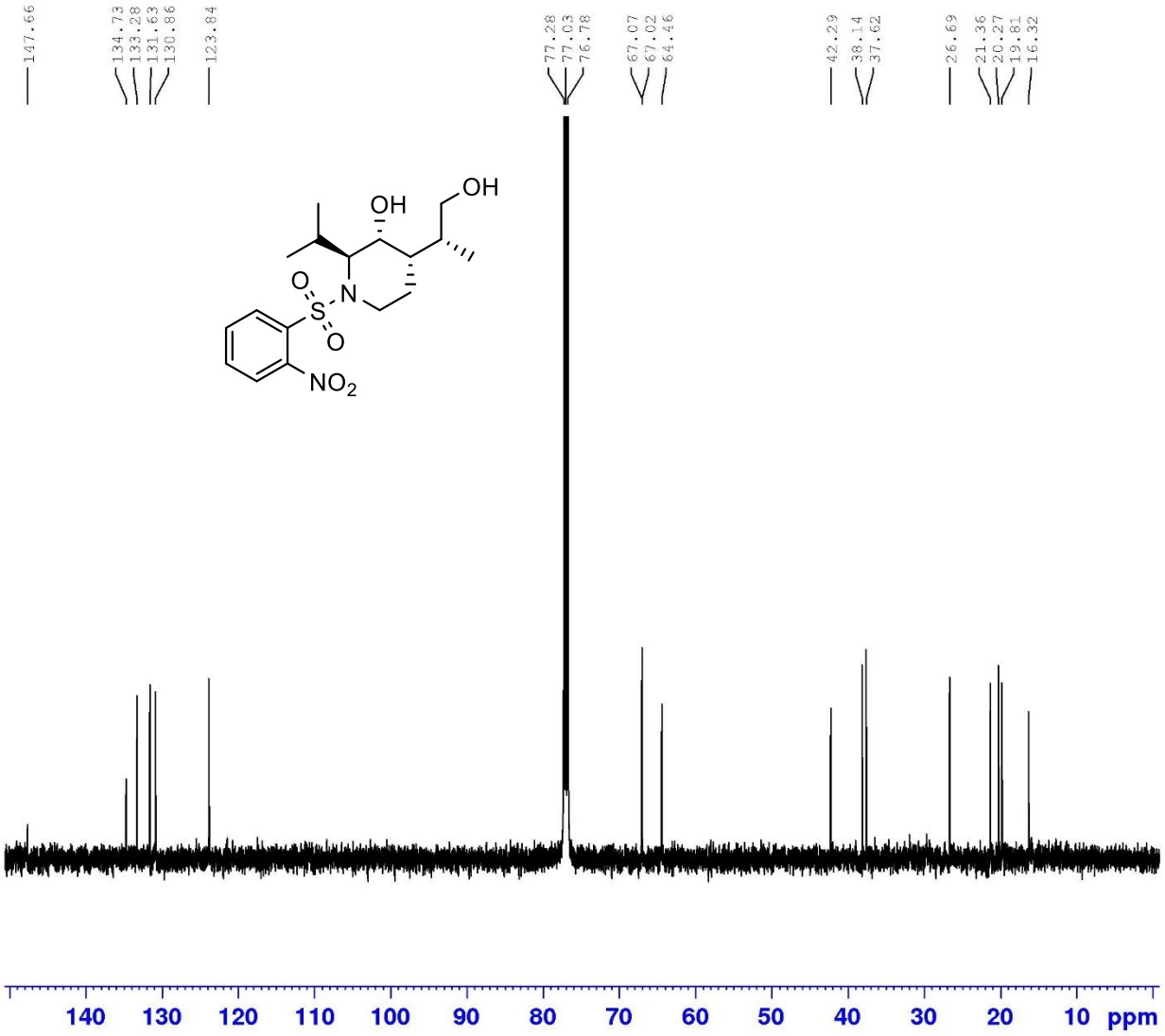
^{13}C-NMR (*R*,*S*,*S*)-**34a**



BRUKER

```
Current Data Parameters
NAME          Feb15-2021
EXPNO                 80
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210216
Time                6.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   362
DW                48.400 usec
DE                10.00 usec
TE                296.8 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530173 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

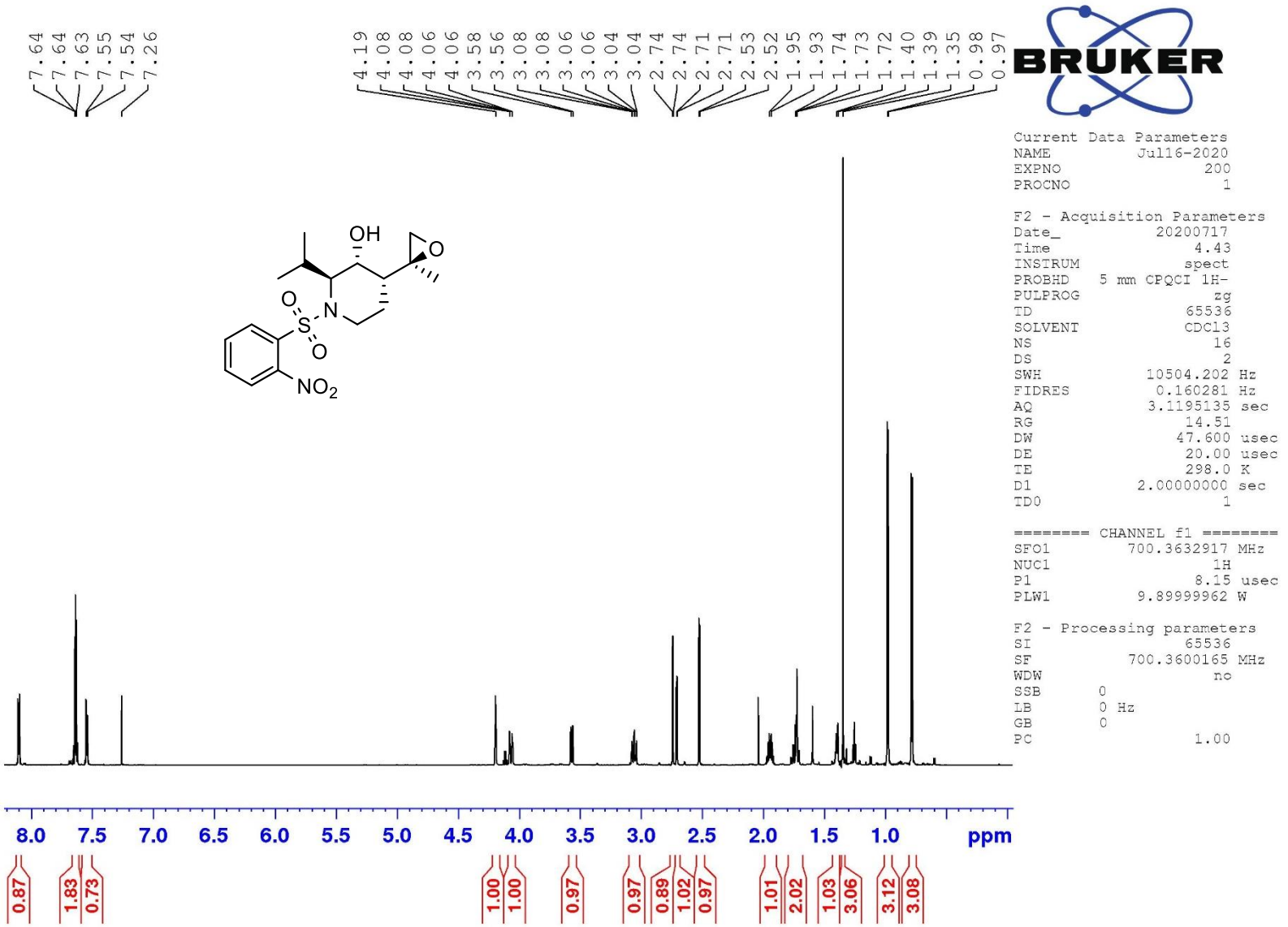
^{1}H-NMR (*R*,*S*,*R*)-**34a**



BRUKER

```
Current Data Parameters
NAME          Feb15-2021
EXPNO                 81
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210216
Time                7.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.20 usec
PL1                  1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

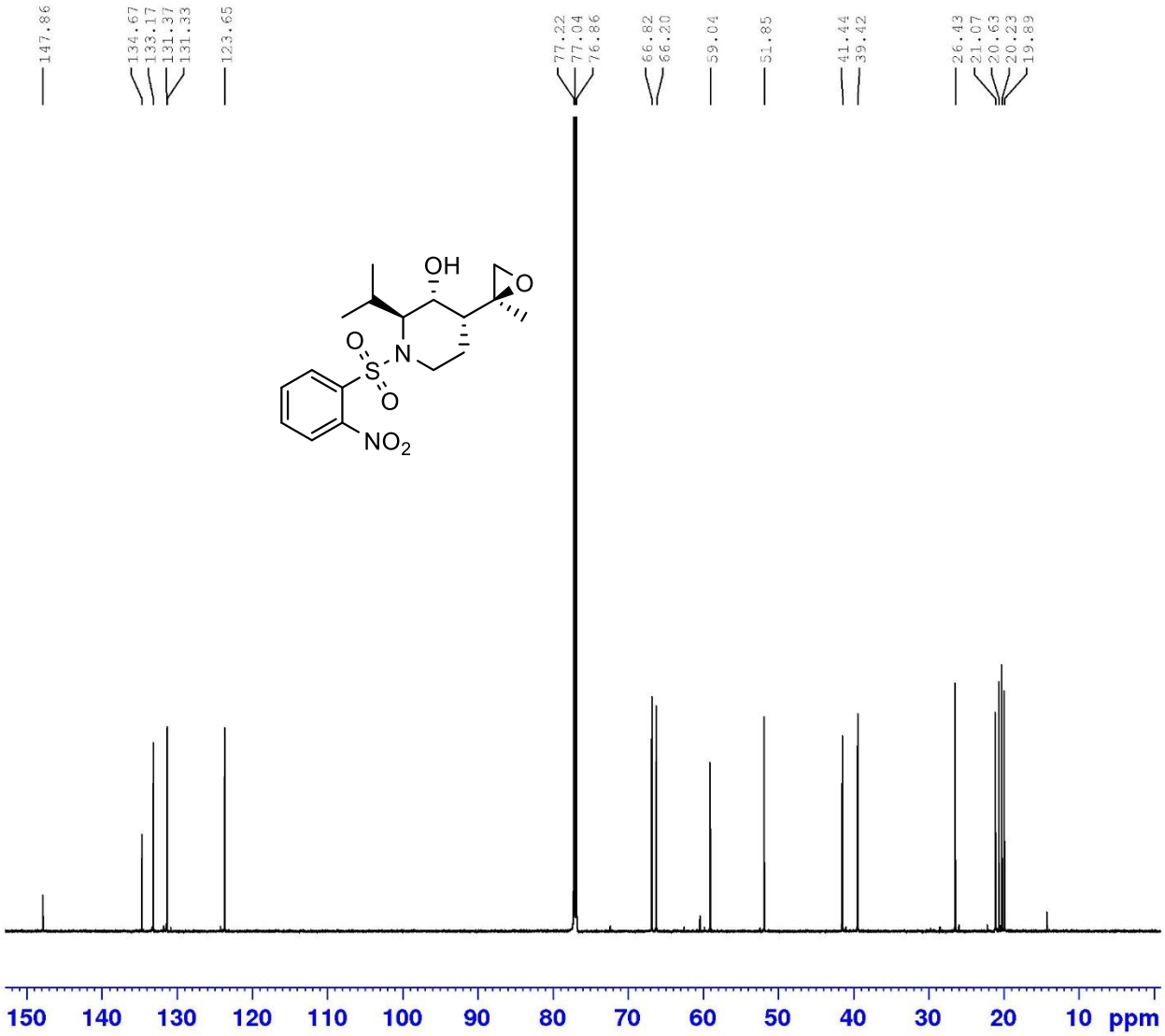
^{13}C-NMR (*R*,*S*,*R*)-**34a**



BRUKER

```
Current Data Parameters
NAME          Jul16-2020
EXPNO                200
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200717
Time                4.43
INSTRUM            spect
PROBHD    5 mm CPQCI 1H-
PULPROG                zg
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10504.202 Hz
FIDRES          0.160281 Hz
AQ             3.1195135 sec
RG                 14.51
DW                47.600 usec
DE                20.00 usec
TE                298.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         700.3632917 MHz
NUC1                  1H
P1                 8.15 usec
PLW1          9.89999962 W

F2 - Processing parameters
SI                 65536
SF           700.3600165 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

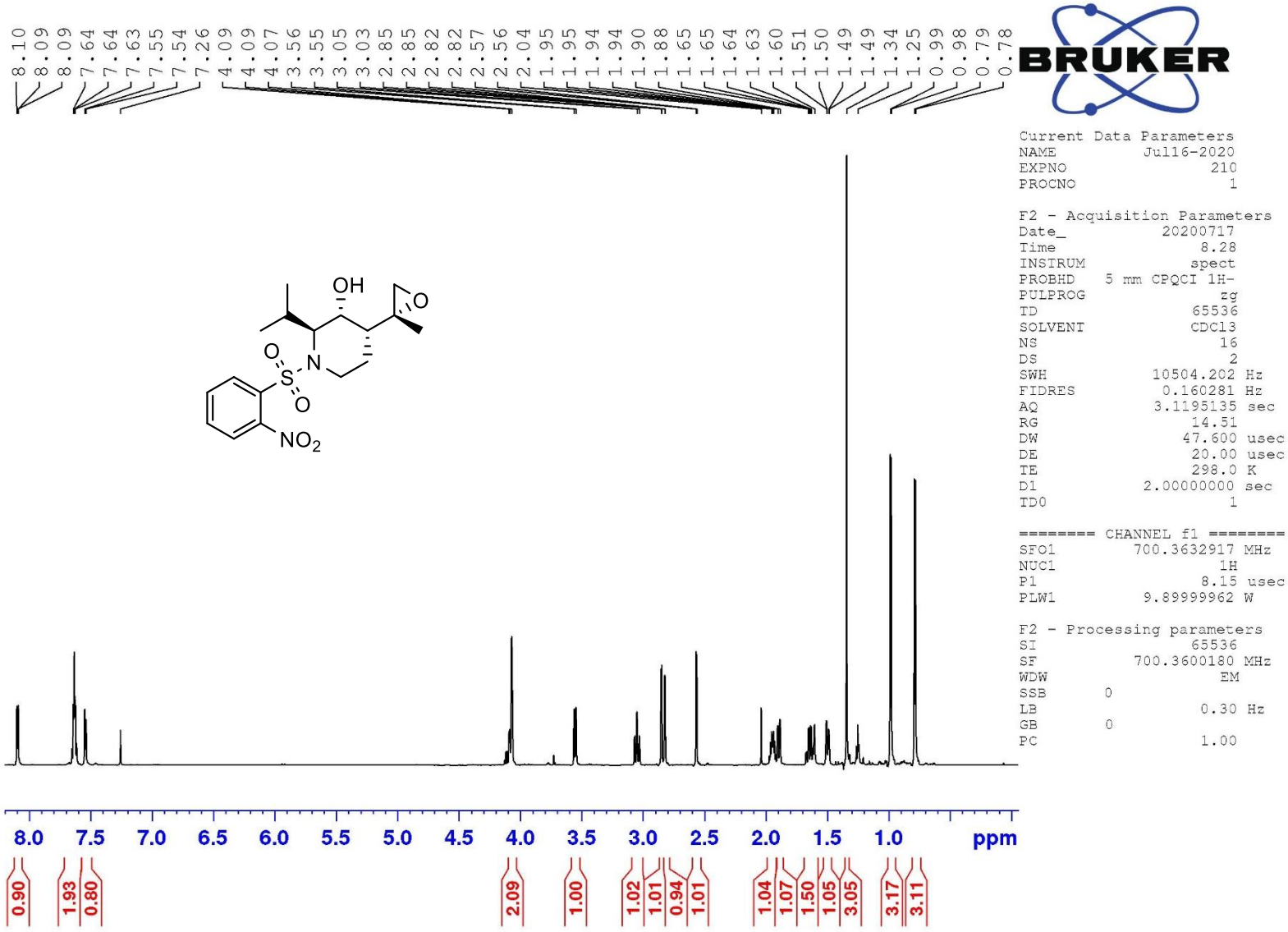
^{1}H-NMR (*R*,*R*,*S*)-**35a**



BRUKER

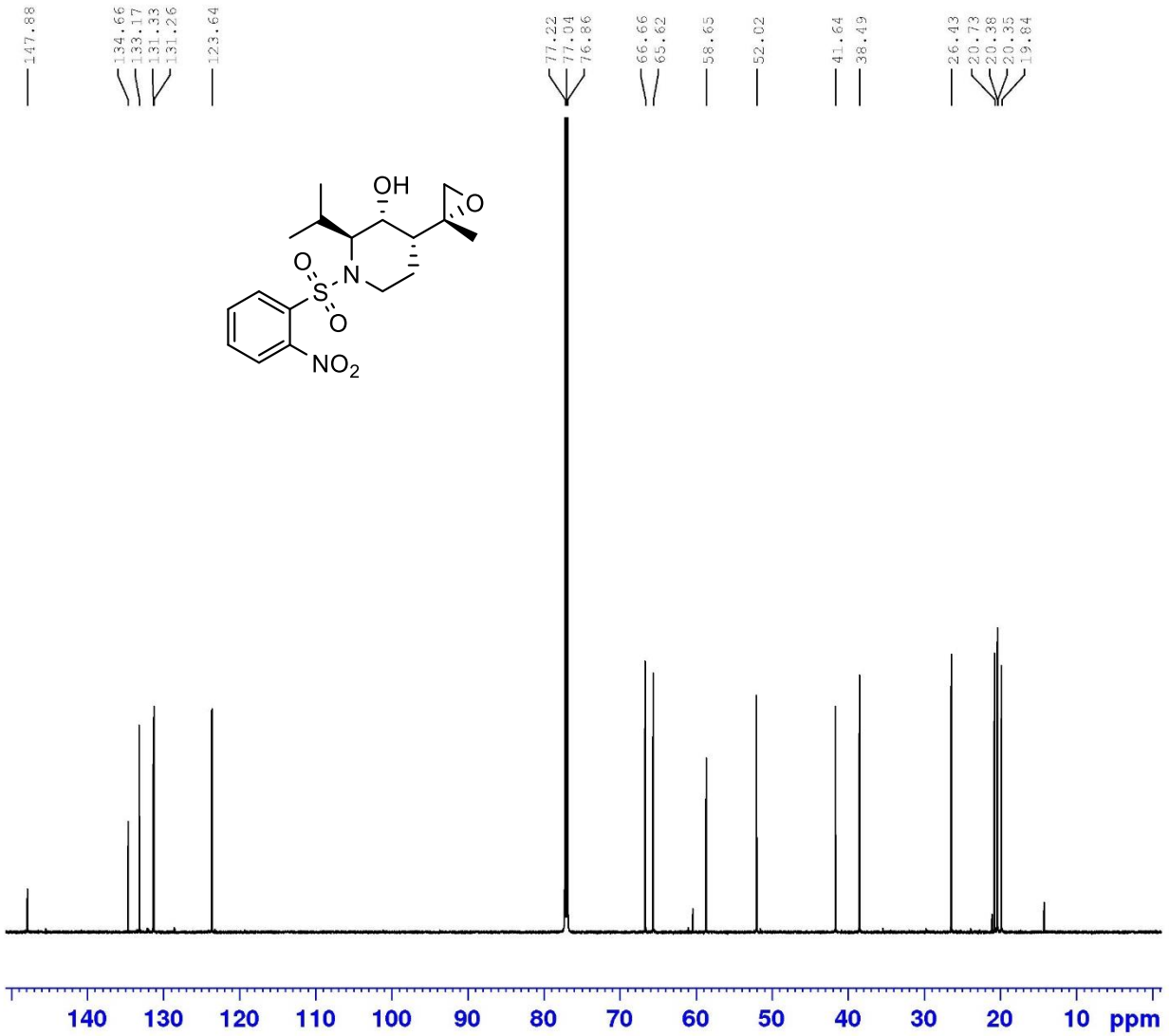
```
Current Data Parameters
NAME          Jul16-2020
EXPNO                201
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200717
Time                5.10
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
ID0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C-NMR (*R*,*R*,*S*)-**35a**




```
Current Data Parameters
NAME          Jul16-2020
EXPNO                210
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200717
Time                8.28
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG               zg
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10504.202 Hz
FIDRES          0.160281 Hz
AQ             3.1195135 sec
RG                 14.51
DW                47.600 usec
DE                20.00 usec
TE                298.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         700.3632917 MHz
NUC1                  1H
P1                 8.15 usec
PLW1          9.89999962 W

F2 - Processing parameters
SI                 65536
SF           700.3600180 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

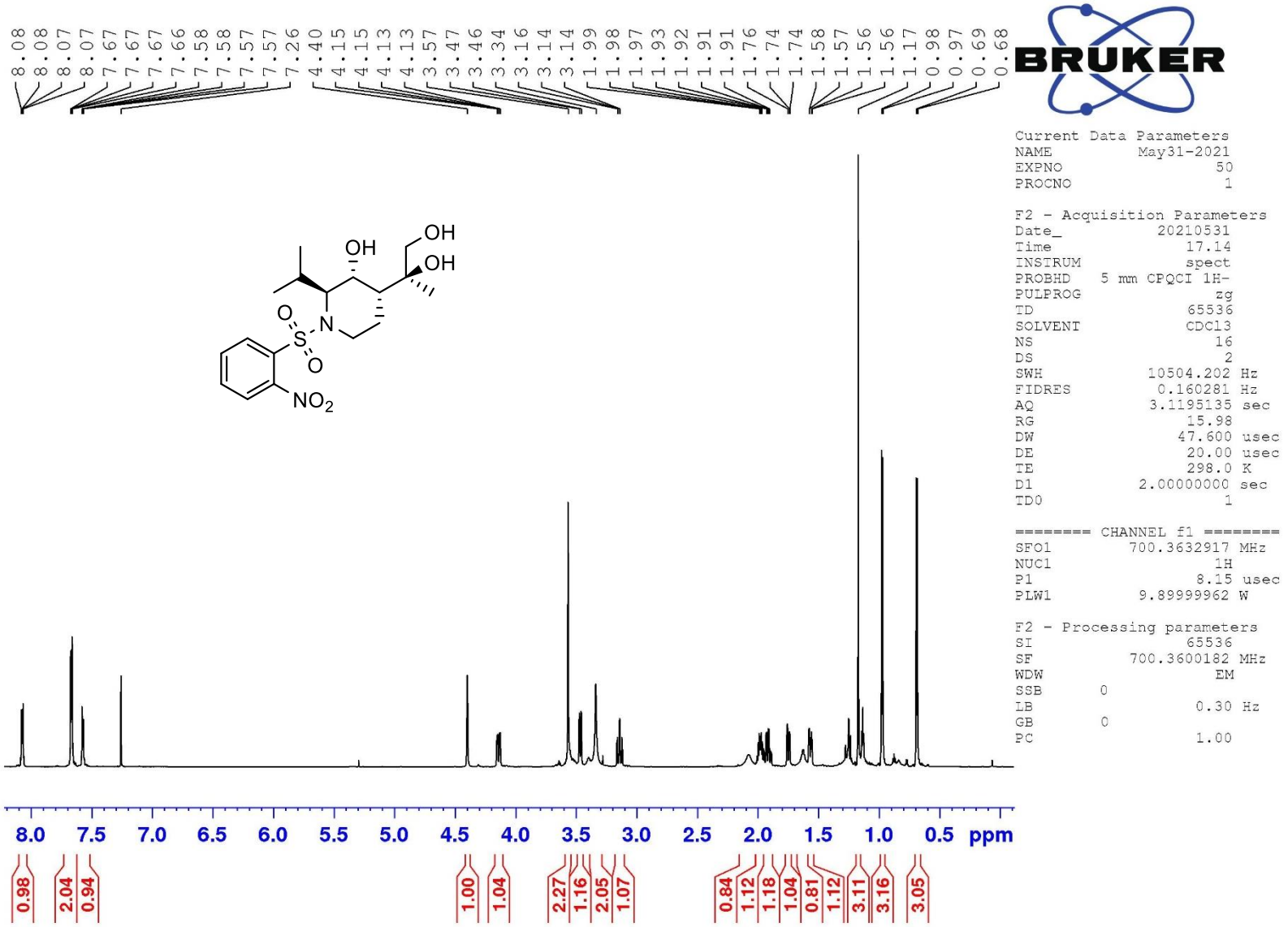
^{1}H-NMR (*R*,*R*,*R*)-**35a**



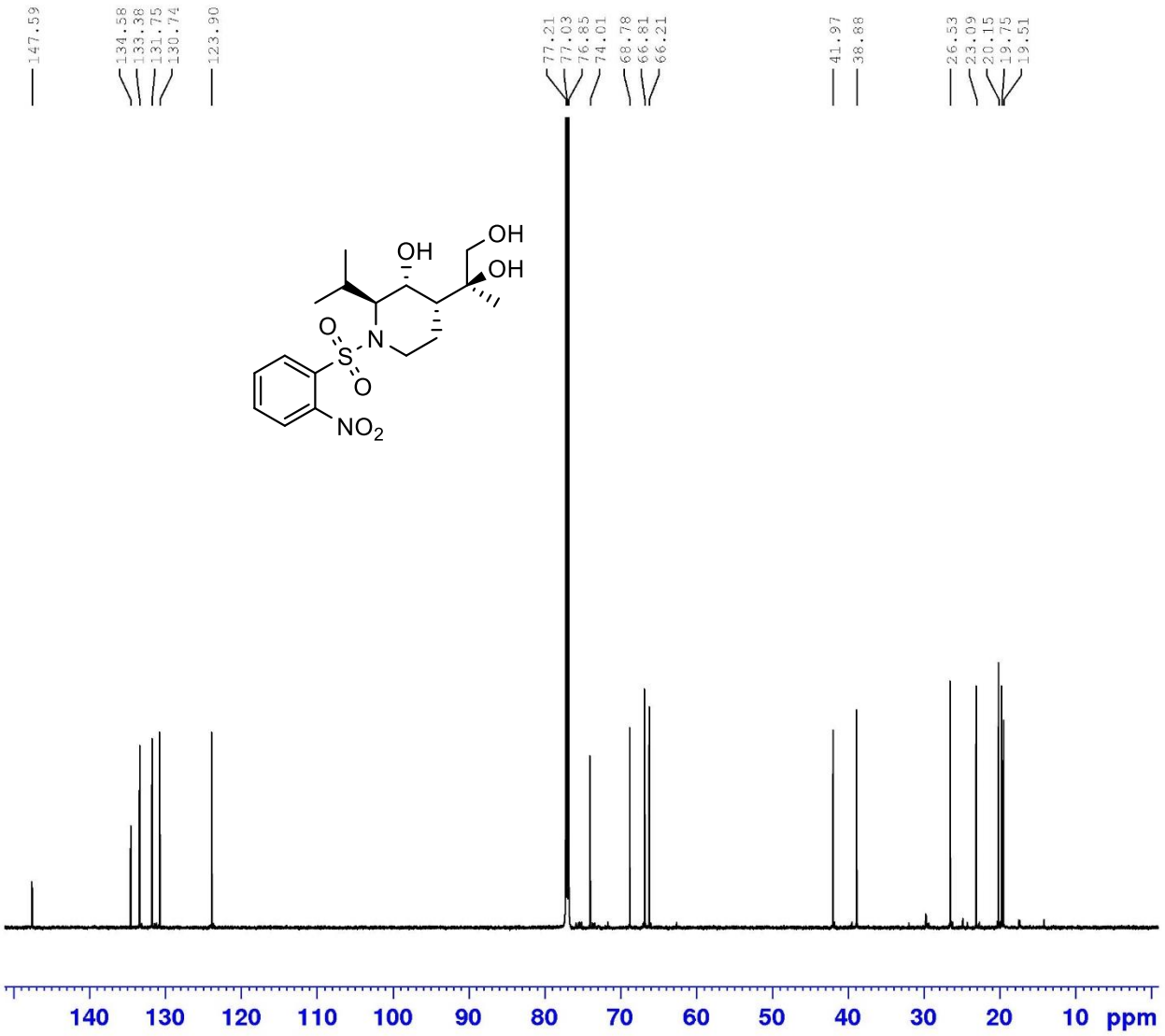
BRUKER

```
Current Data Parameters
NAME          Jul16-2020
EXPNO                211
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200717
Time                8.55
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         176.1232717 MHz
NUC1                 13C
P1                 12.00 usec
PLW1        105.00000000 W

======== CHANNEL f2 ========
SFO2         700.3628014 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                 131072
SF           176.1056620 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*R*,*R*,*R*)-**35a**

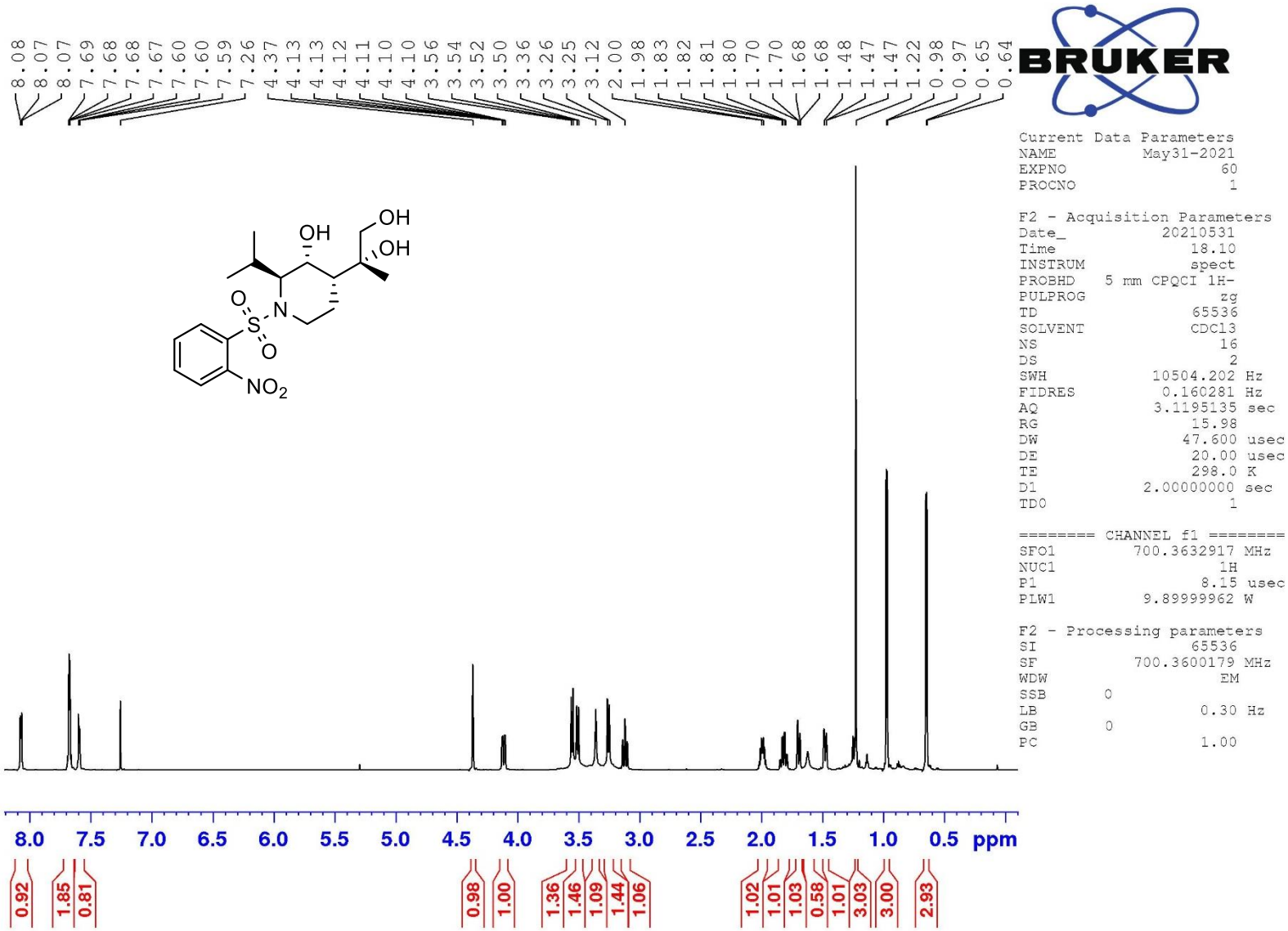


^{1}H-NMR (*R*,*R*,*S*)-**36a**

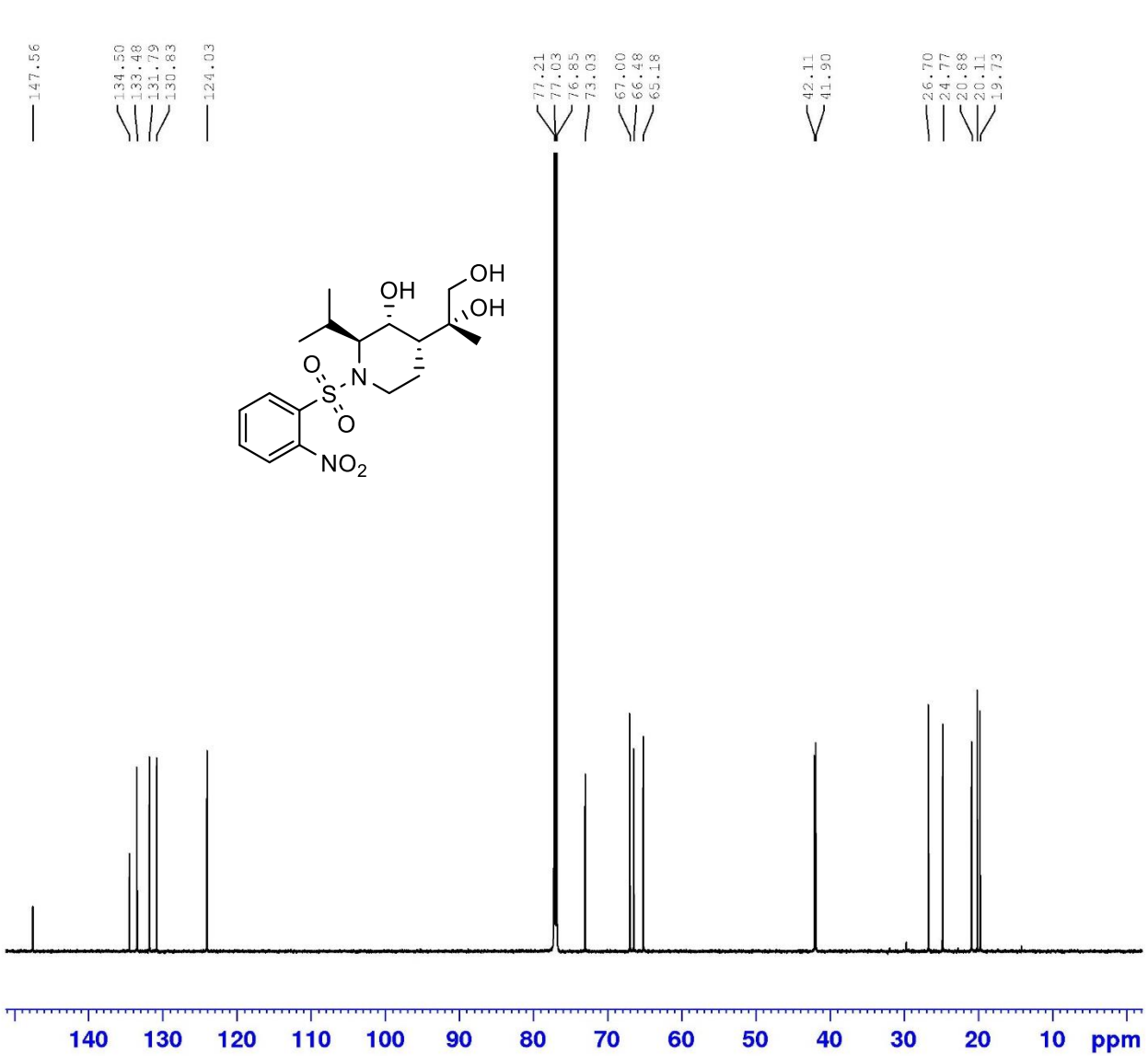


^{13}C-NMR (*R*,*R*,*S*)-**36a**

```
Current Data Parameters
NAME          May31-2021
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210531
Time               18.04
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE               18.00 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2           9.89999962 W
PLW12          0.15564001 W
PLW13          0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

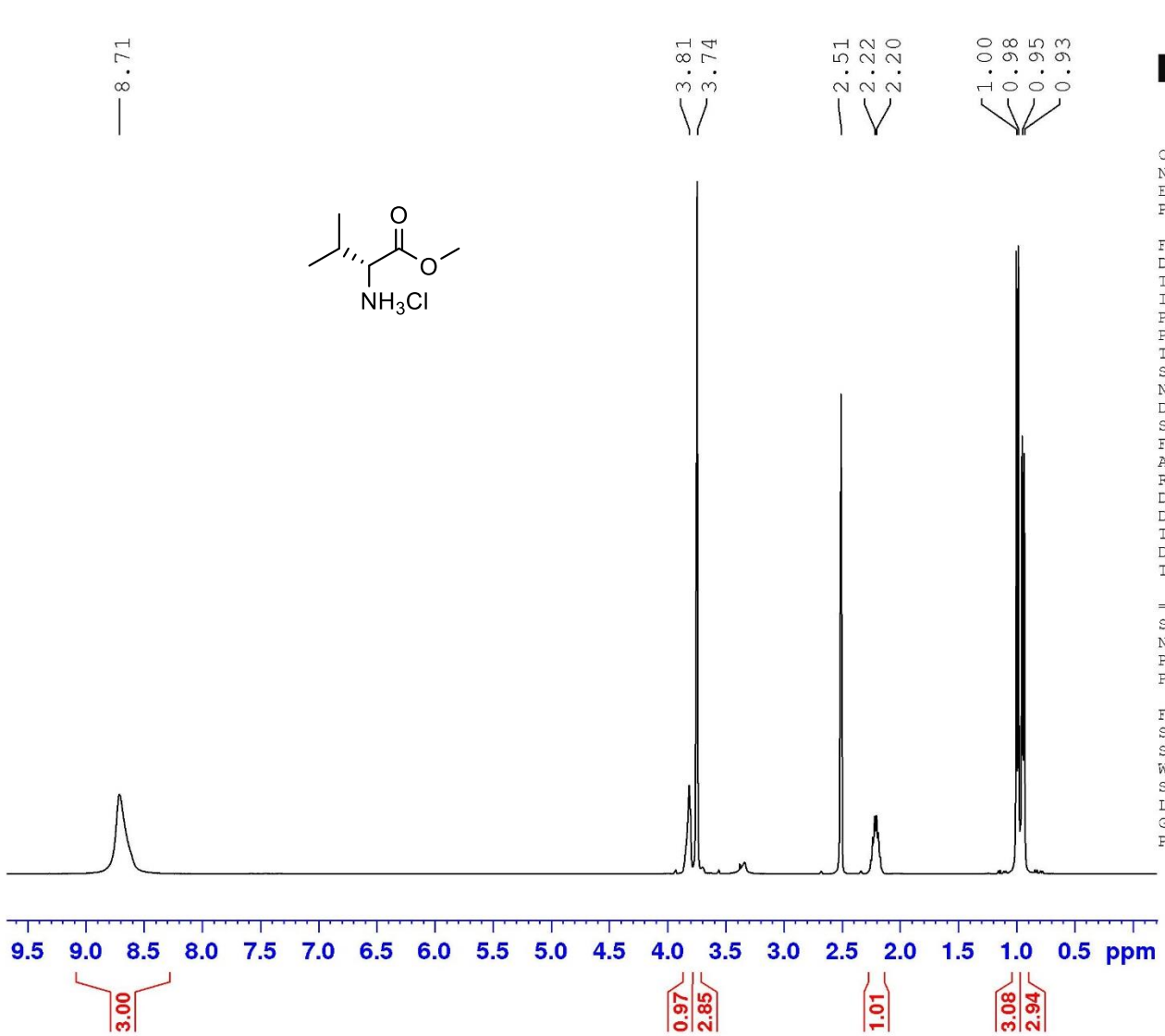


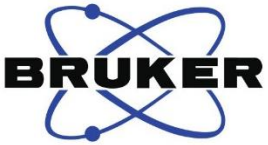
^{1}H-NMR (*R*,*R*,*R*)-**36a**



^{13}C-NMR (*R*,*R*,*R*)-**36a**

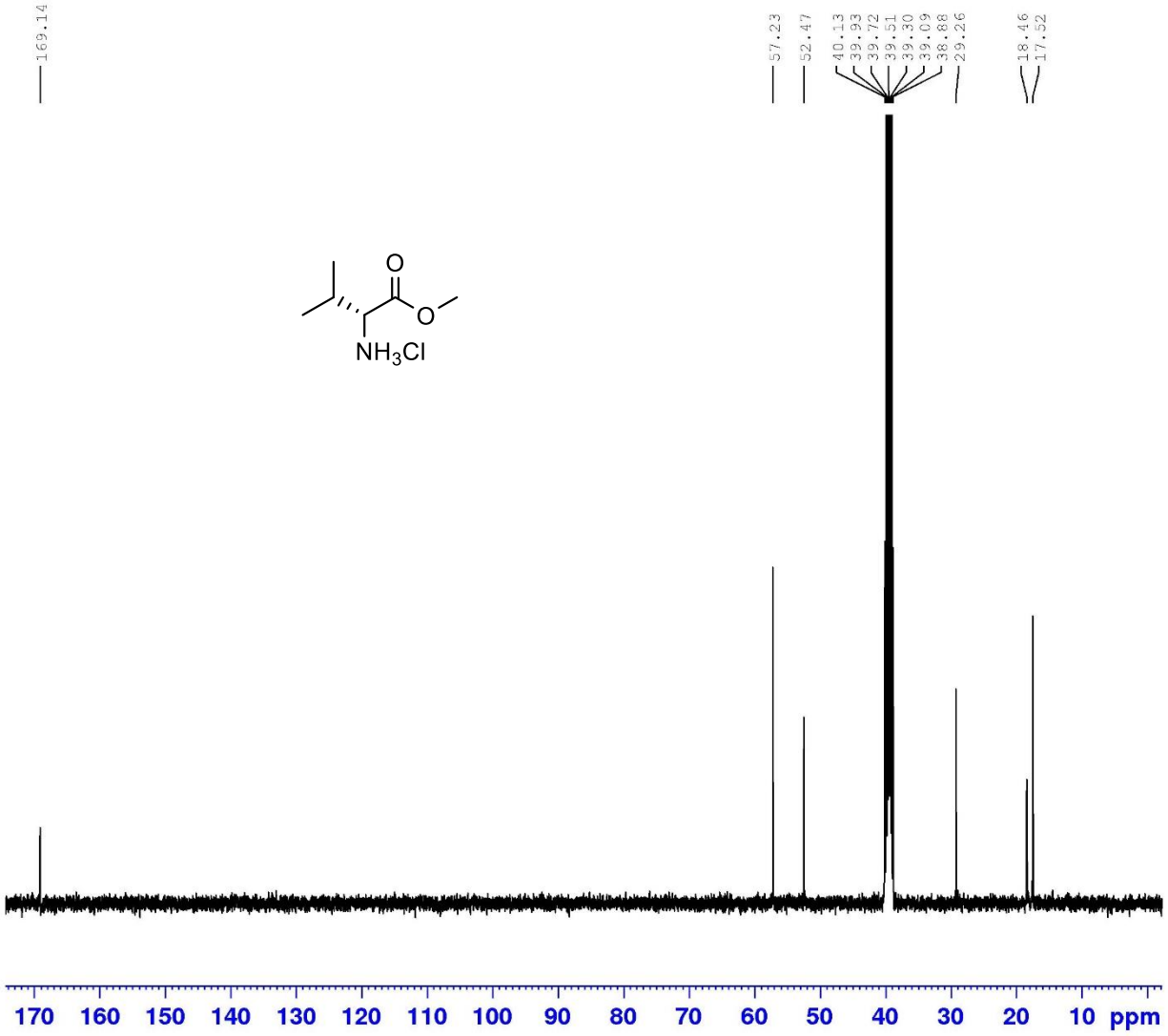
```
Current Data Parameters
NAME          May31-2021
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210531
Time               19.00
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2           9.89999962 W
PLW12          0.15564001 W
PLW13          0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

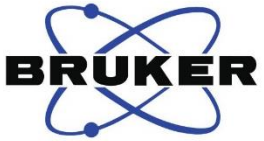





```
Current Data Parameters
NAME          Mar05-2020
EXPNO                400
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200306
Time                3.10
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 124.07
DW                 62.400 usec
DE                  6.50 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1024708 MHz
NUC1                   1H
P1                  13.70 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1000000 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

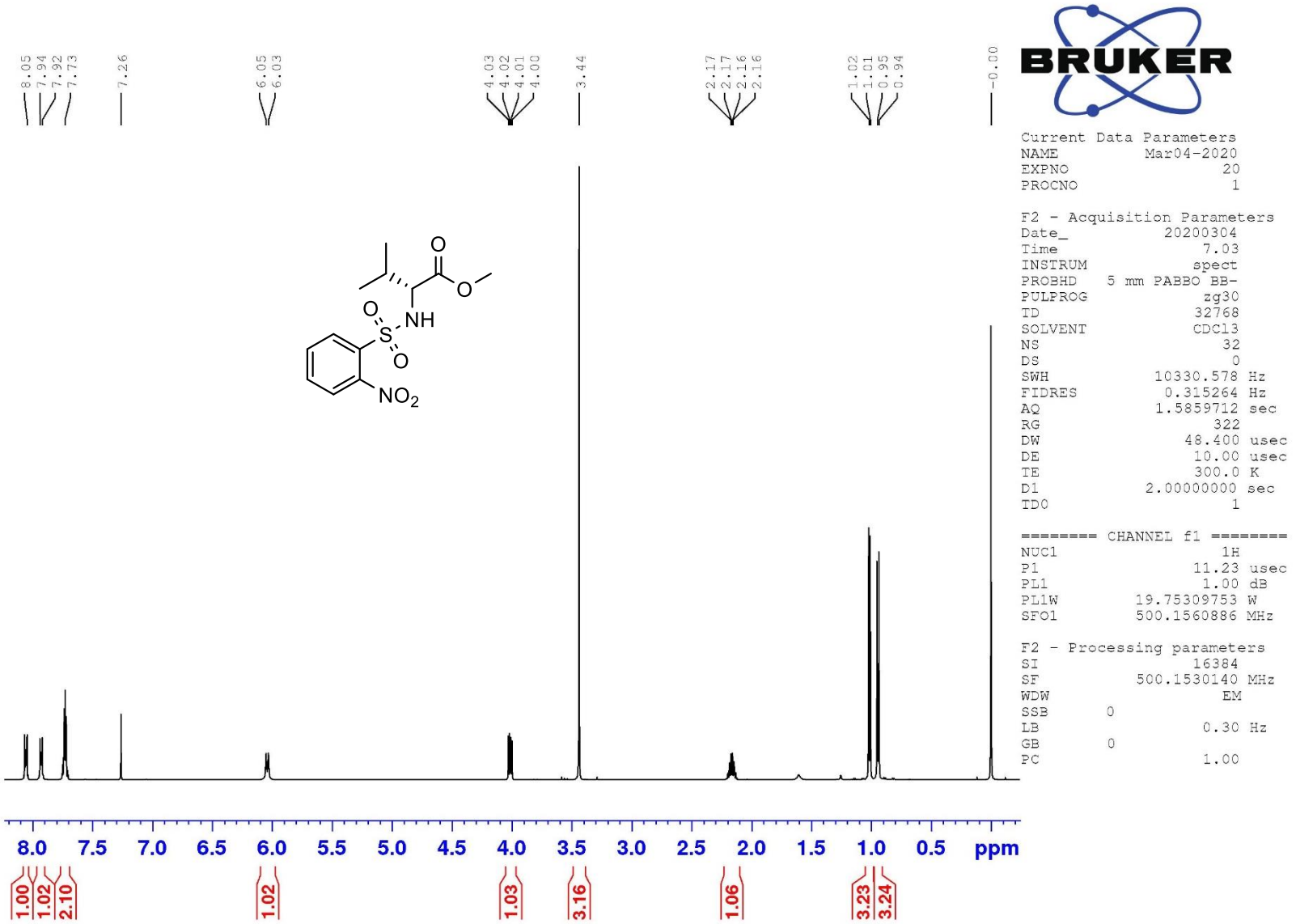
^{1}H-NMR **25b**






```
Current Data Parameters
NAME          Mar05-2020
EXPNO                401
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200306
Time                3.40
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                    512
DS                      4
SWH             24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                 205.35
DW                 20.800 usec
DE                  6.50 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6152851 MHz
NUC1                  13C
P1                  10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1016004 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2         12.00000000 W
PLW12         0.27805999 W
PLW13         0.22522999 W

F2 - Processing parameters
SI                 32768
SF           100.6052755 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

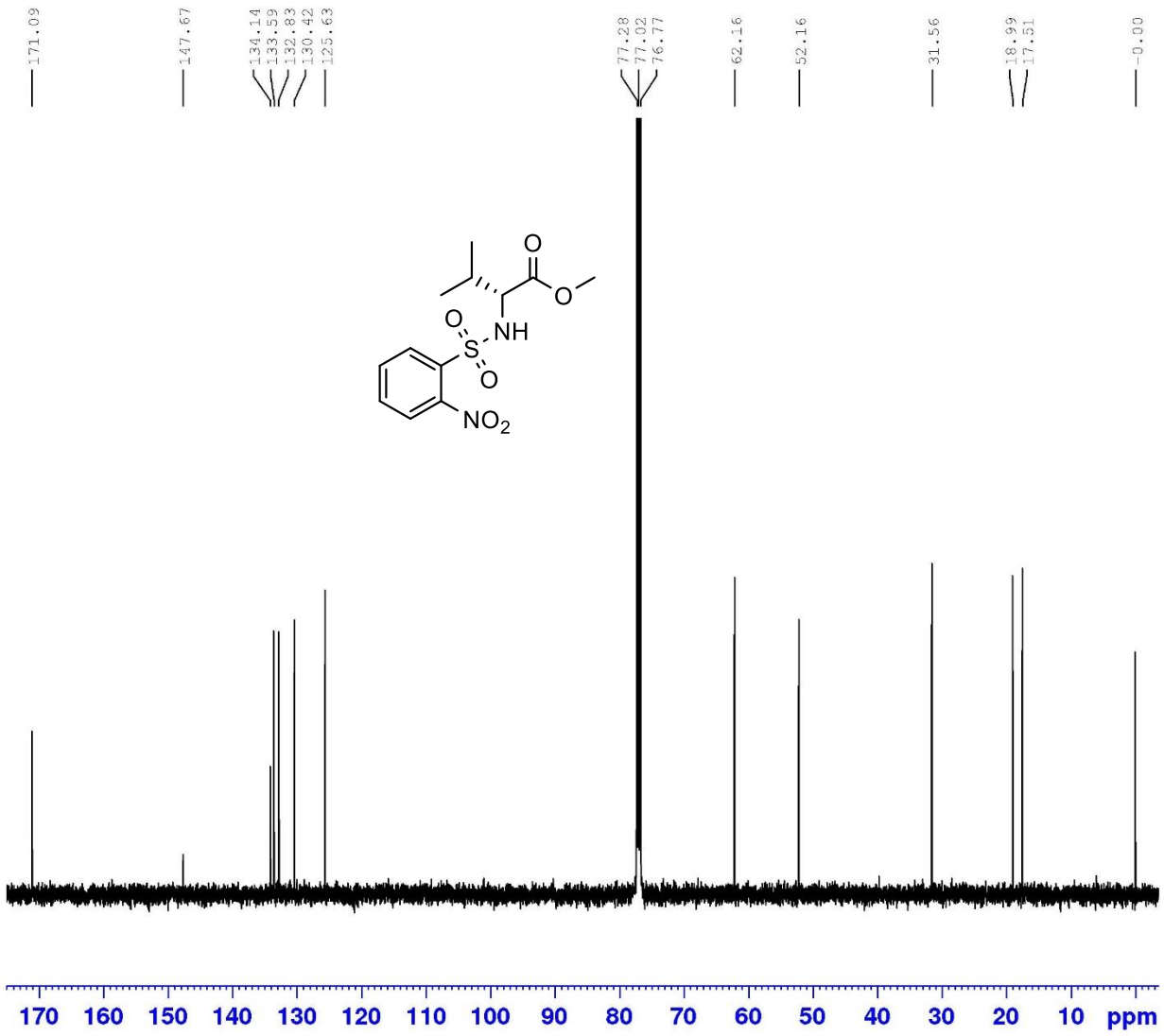
^{13}C-NMR **25b**



BRUKER

```
Current Data Parameters
NAME          Mar04-2020
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200304
Time                7.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530140 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

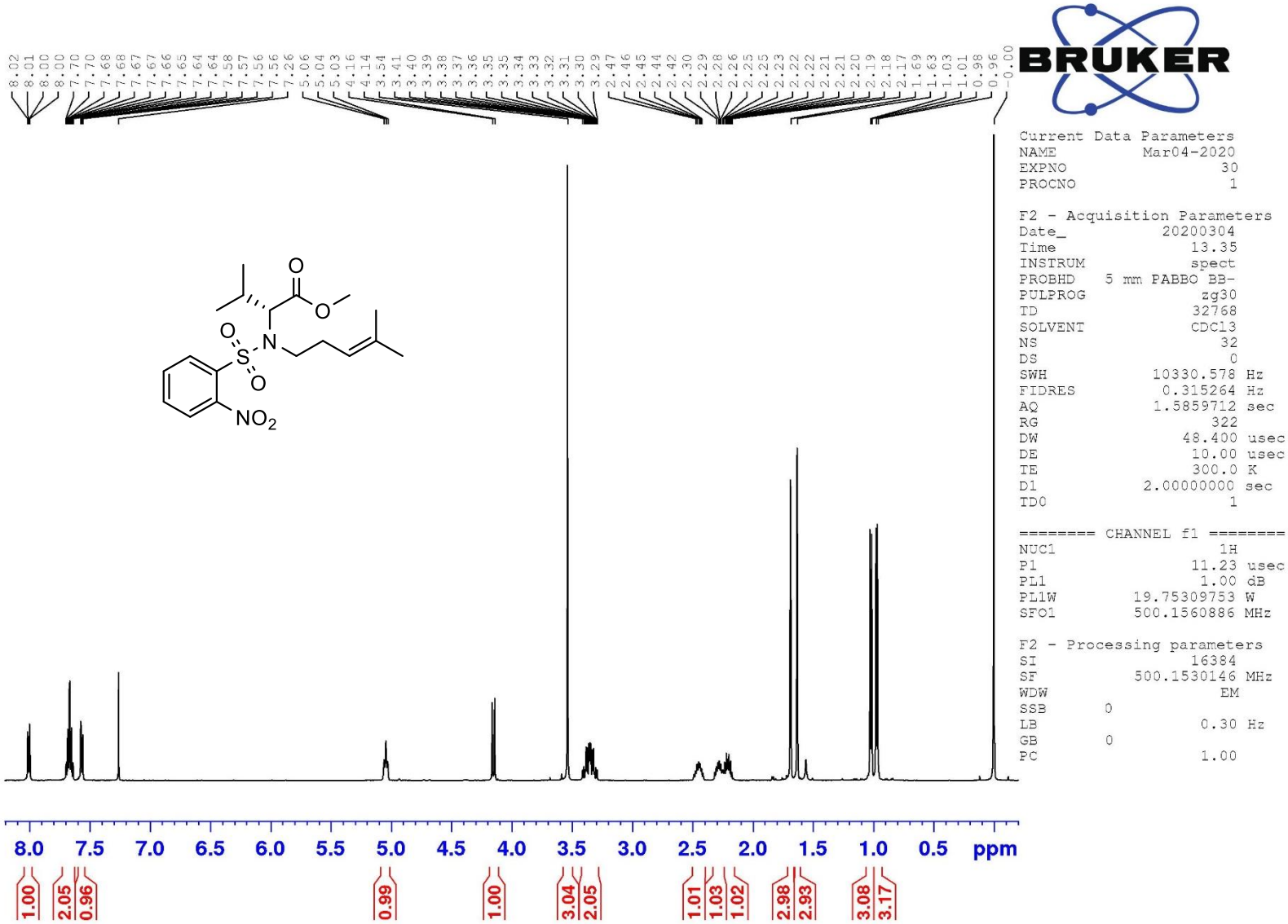
^{1}H-NMR **26b**



BRUKER

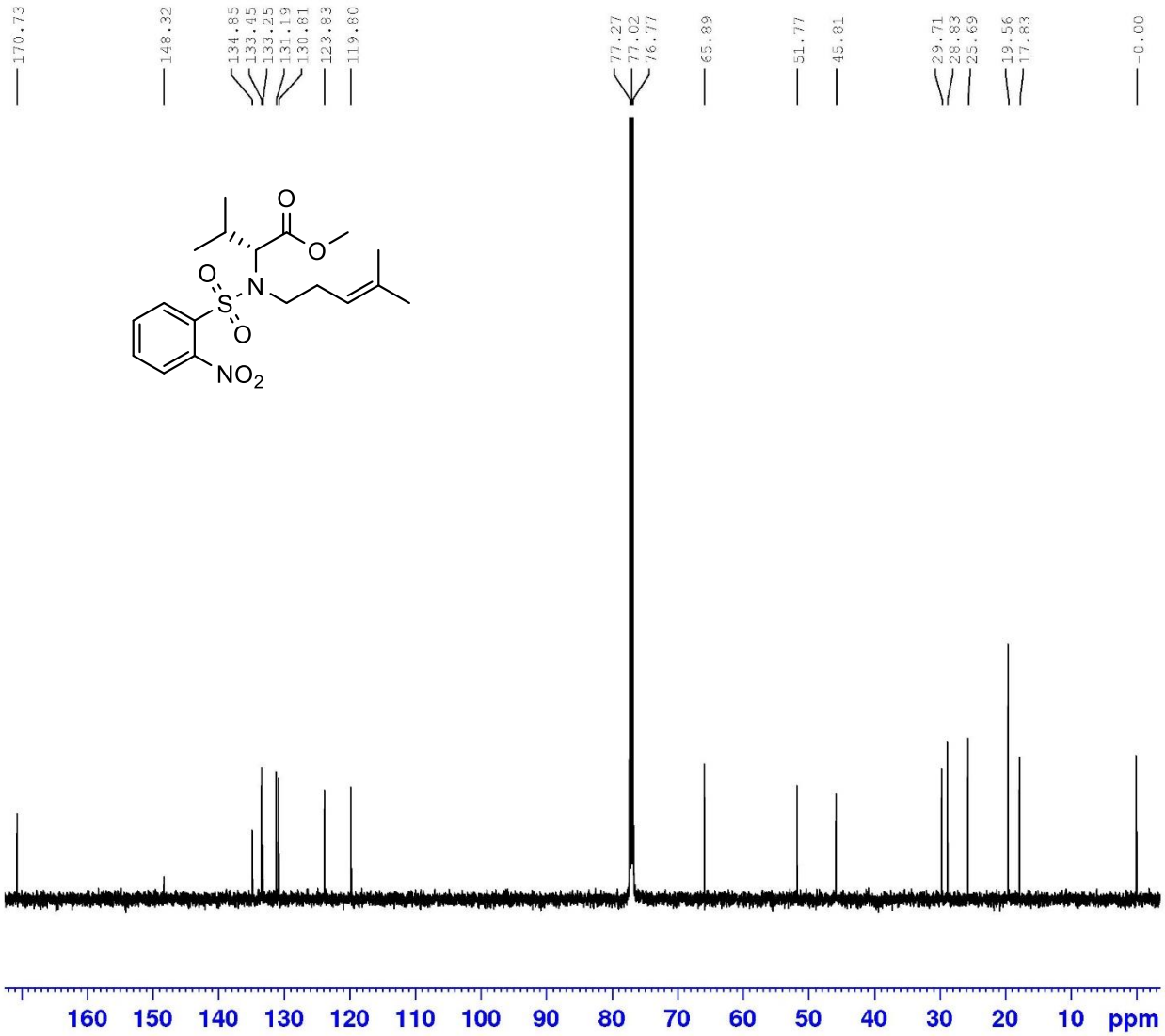
```
Current Data Parameters
NAME          Mar04-2020
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200304
Time                7.57
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2300
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.20 usec
PL1                  1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635739 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **26b**




```
Current Data Parameters
NAME          Mar04-2020
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200304
Time               13.35
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530146 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

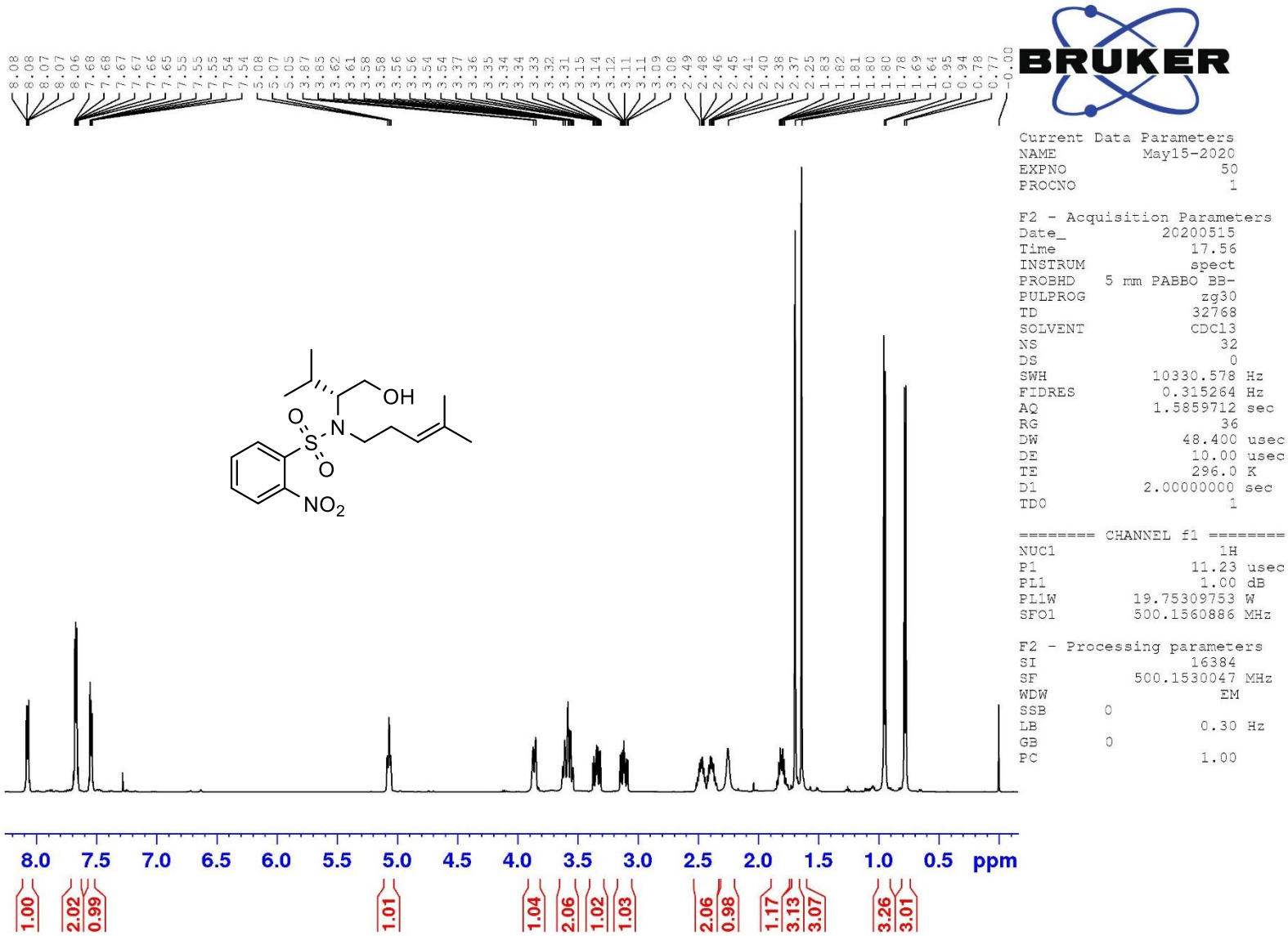
^{1}H-NMR **27b**



BRUKER

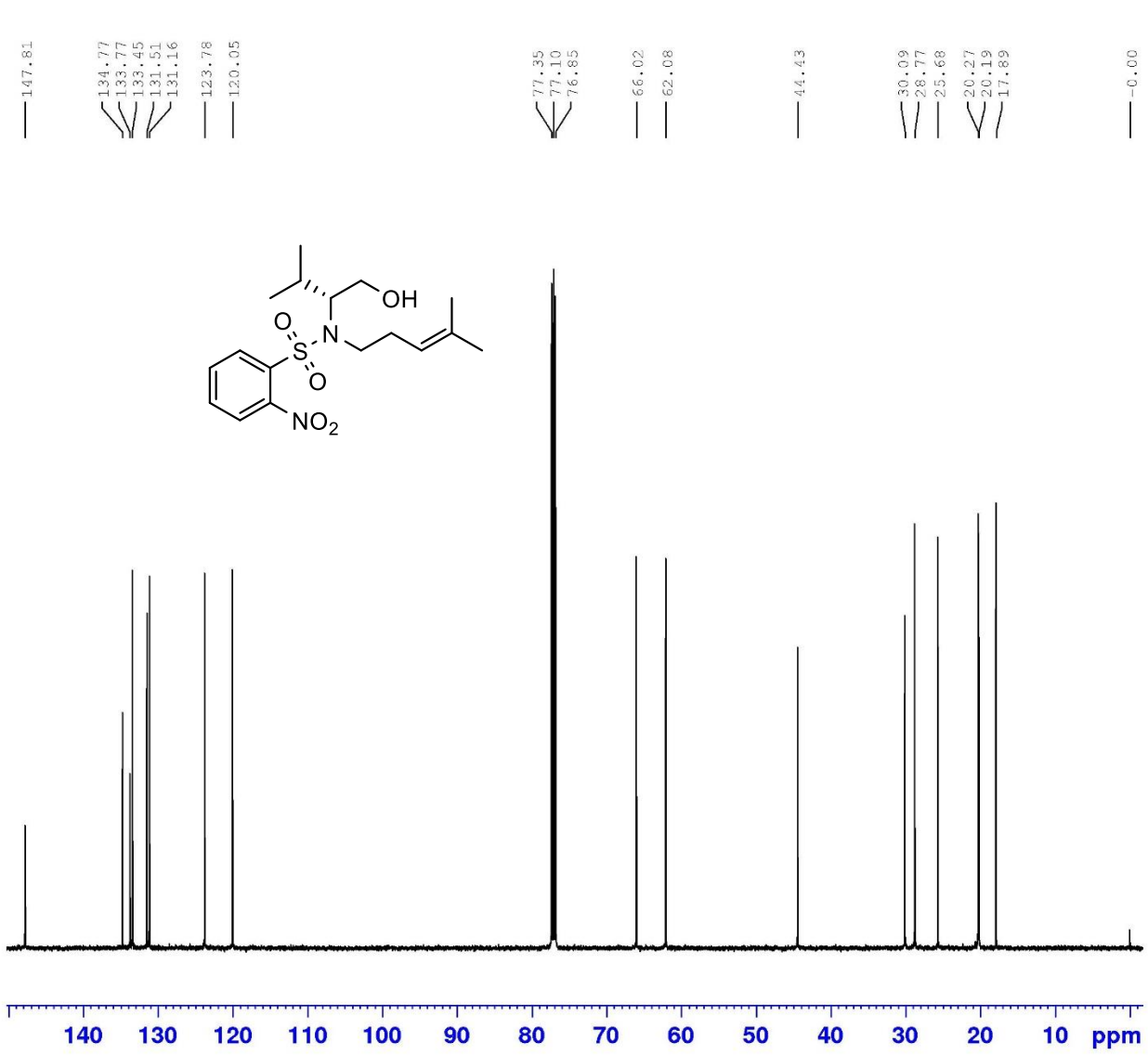
```
Current Data Parameters
NAME          Mar04-2020
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200304
Time               14.28
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                  2300
DW                15.200 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.20 usec
PL1                  1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                   1.00 dB
PL12                 19.99 dB
PL13                 21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635740 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **27b**




```
Current Data Parameters
NAME          May15-2020
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200515
Time               17.56
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                    36
DW               48.400 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530047 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

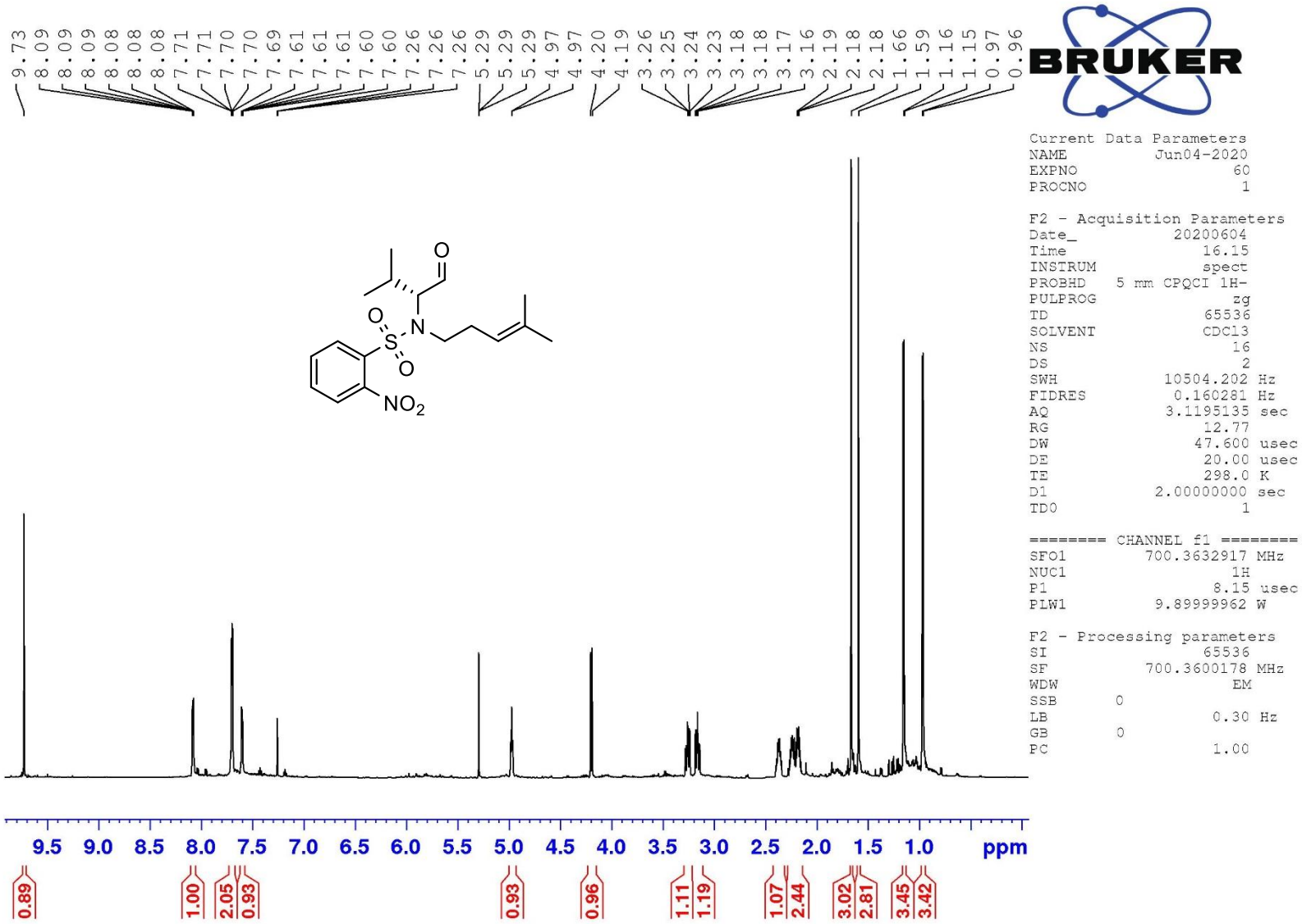
^{1}H-NMR **28b**



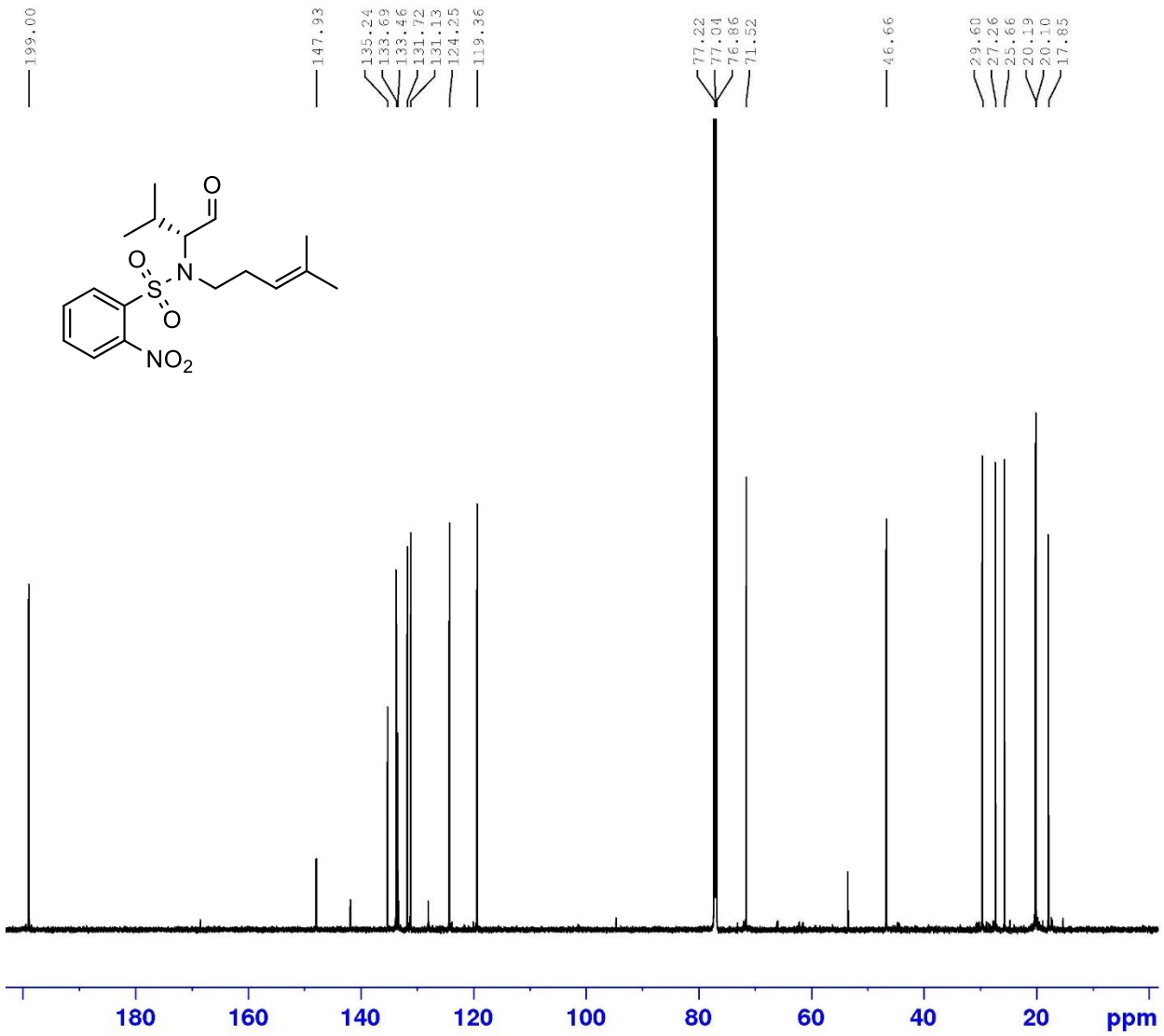
BRUKER

```
Current Data Parameters
NAME          May15-2020
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200515
Time               18.49
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES        0.501934 Hz
AQ            0.9961472 sec
RG                 2300
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635743 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **28b**

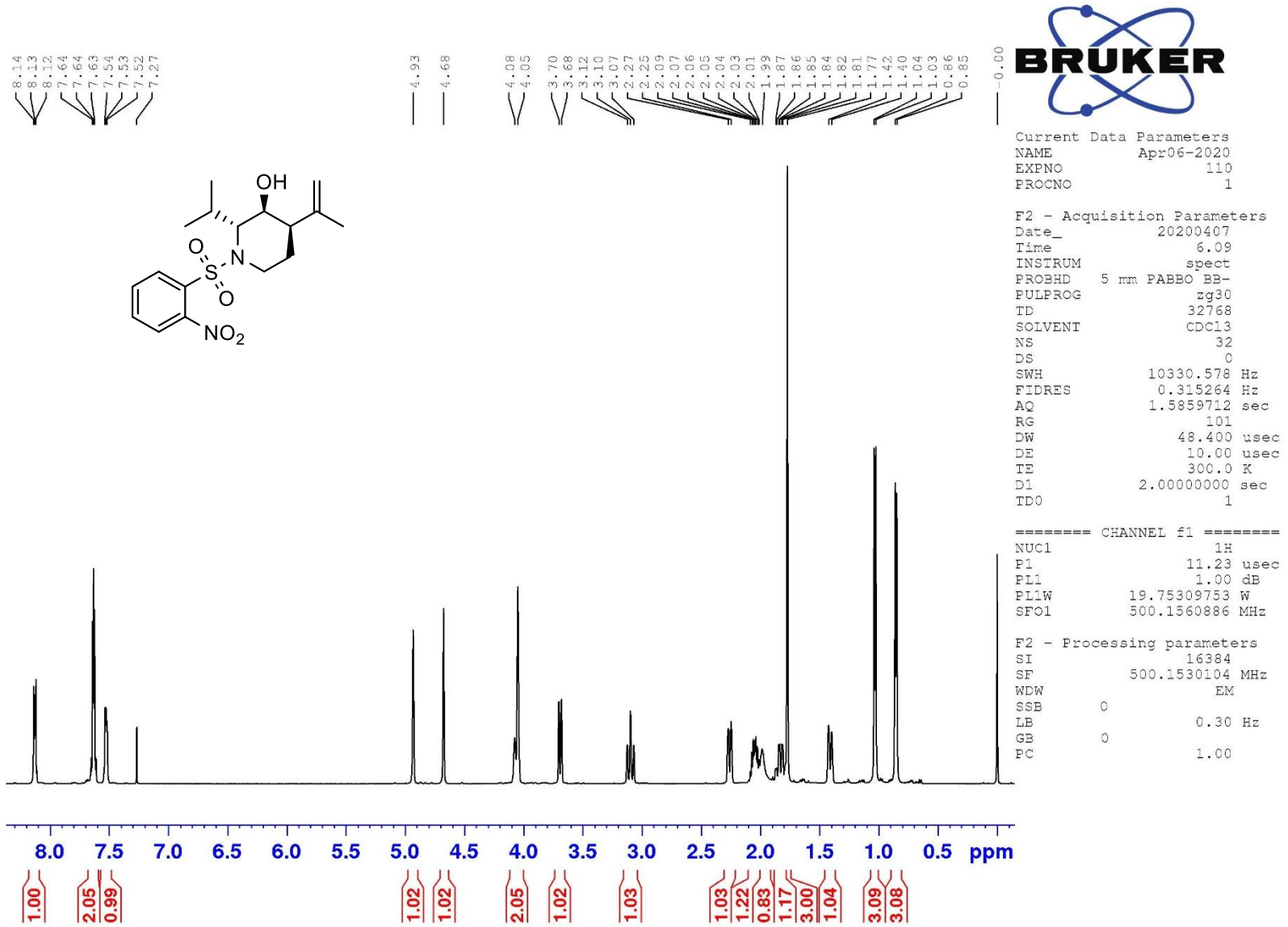

9.73 8.09 8.09 8.09 8.08 8.08 8.08 7.71 7.71 7.70 7.70 7.69 7.61 7.61 7.61 7.60 7.60 7.26 7.26 7.26 5.29 5.29 5.29 4.97 4.97 4.20 4.19 3.26 3.25 3.24 3.23 3.18 3.18 3.17 3.16 2.19 2.18 2.18 1.66 1.59 1.16 1.15 0.97 0.96
BRUKER
Current Data Parameters
NAME Jun04-2020
EXPNO 60
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200604
Time 16.15
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10504.202 Hz
FIDRES 0.160281 Hz
AQ 3.1195135 sec
RG 12.77
DW 47.600 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 700.3632917 MHz
NUC1 1H
P1 8.15 usec
PLW1 9.89999962 W
F2 - Processing parameters
SI 65536
SF 700.3600178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
S
N
O
NO2
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.89 1.00 2.05 0.93 0.93 0.96 1.11 1.19 1.07 2.44 3.02 2.81 3.45 3.42

^{1}H-NMR **29b**



^{13}C-NMR **29b**

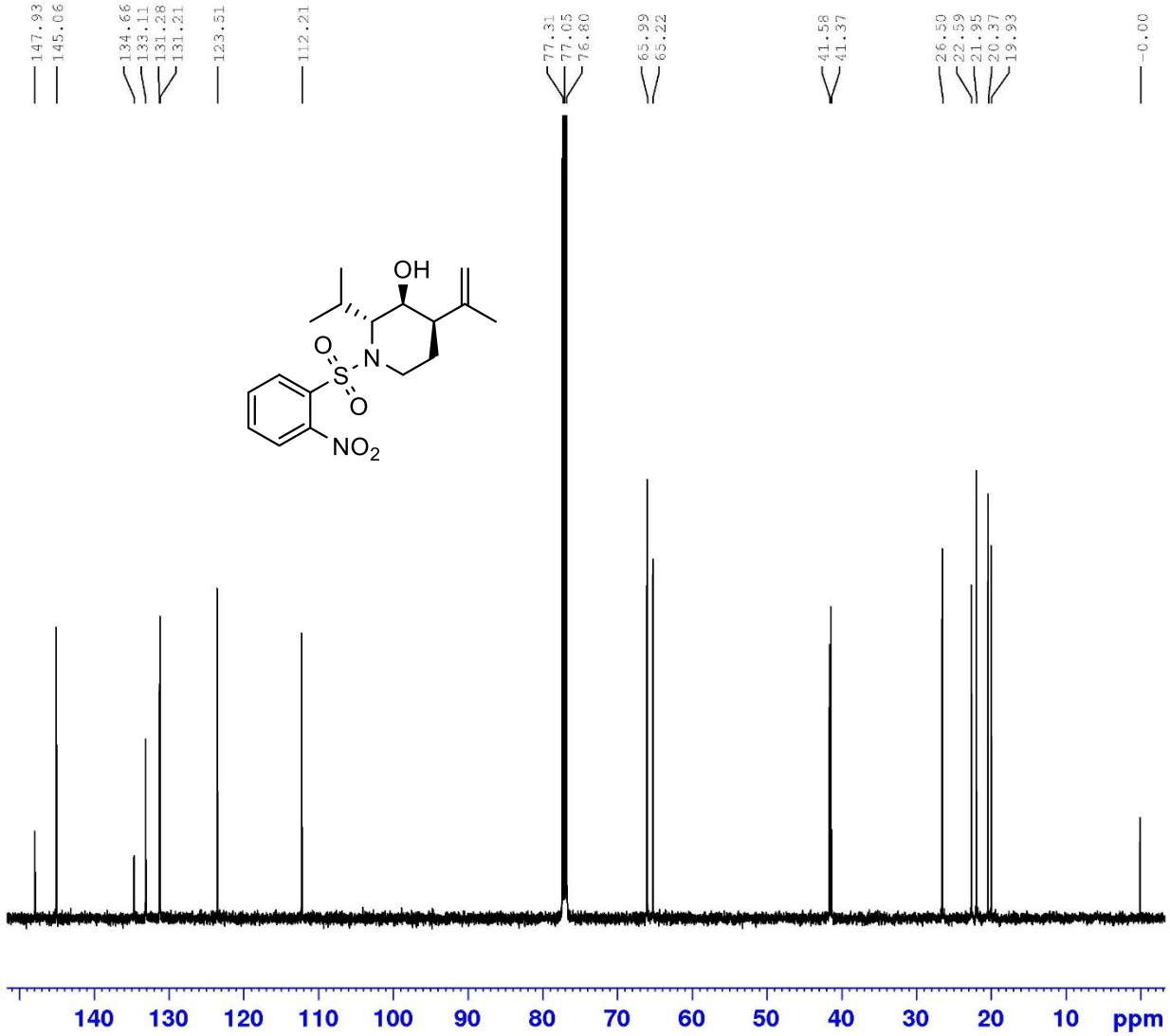
```
Current Data Parameters
NAME          Jun04-2020
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200604
Time               16.28
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

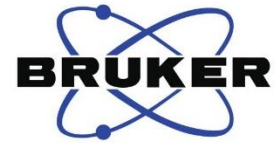


BRUKER

```
Current Data Parameters
NAME          Apr06-2020
EXPNO                110
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200407
Time                6.09
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   101
DW               48.400 usec
DE               10.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530104 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

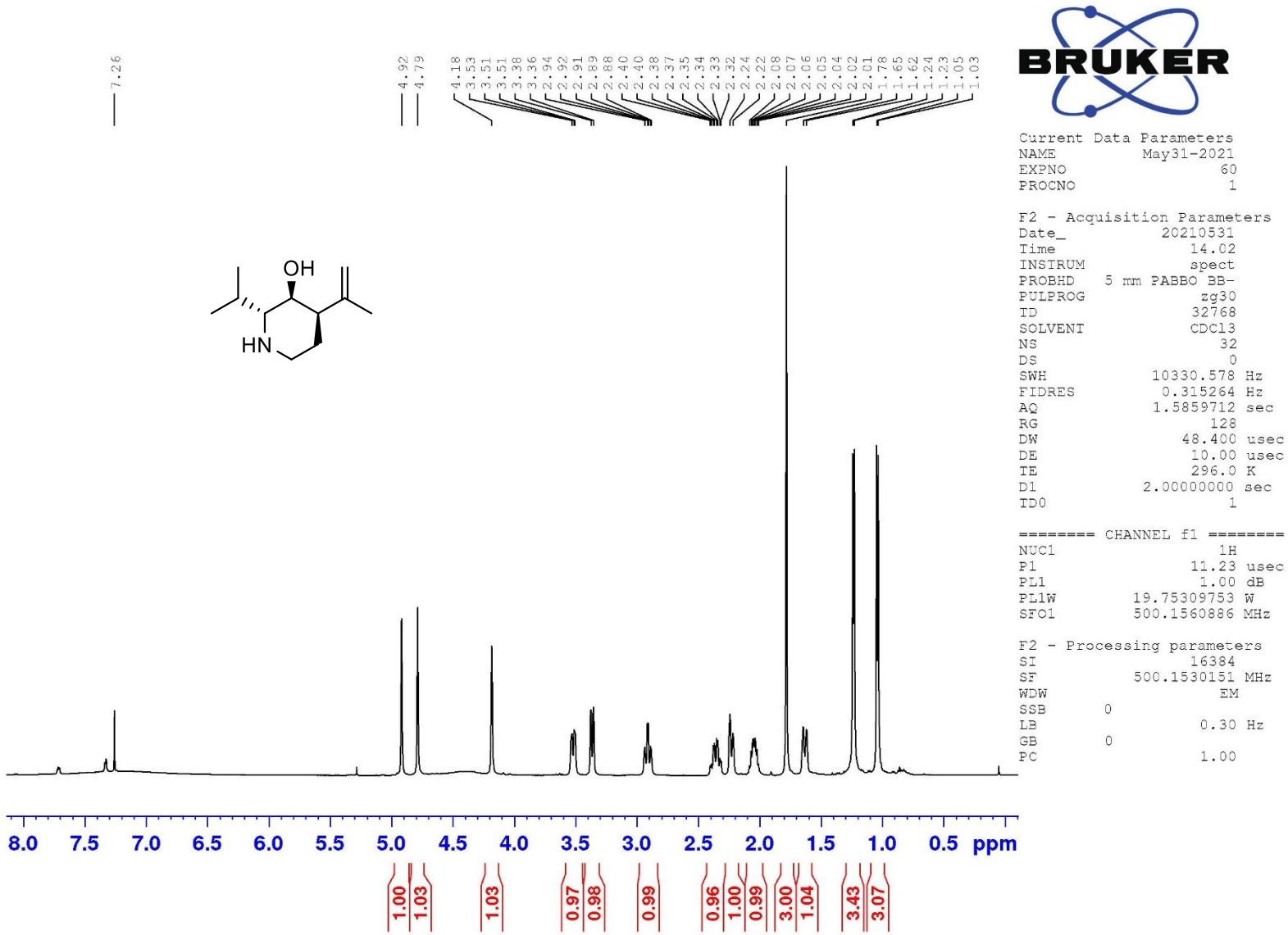
^{1}H-NMR (*S*,*R*)-**30b**






```
Current Data Parameters
NAME          Apr06-2020
EXPNO                111
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200407
Time                7.02
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635748 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

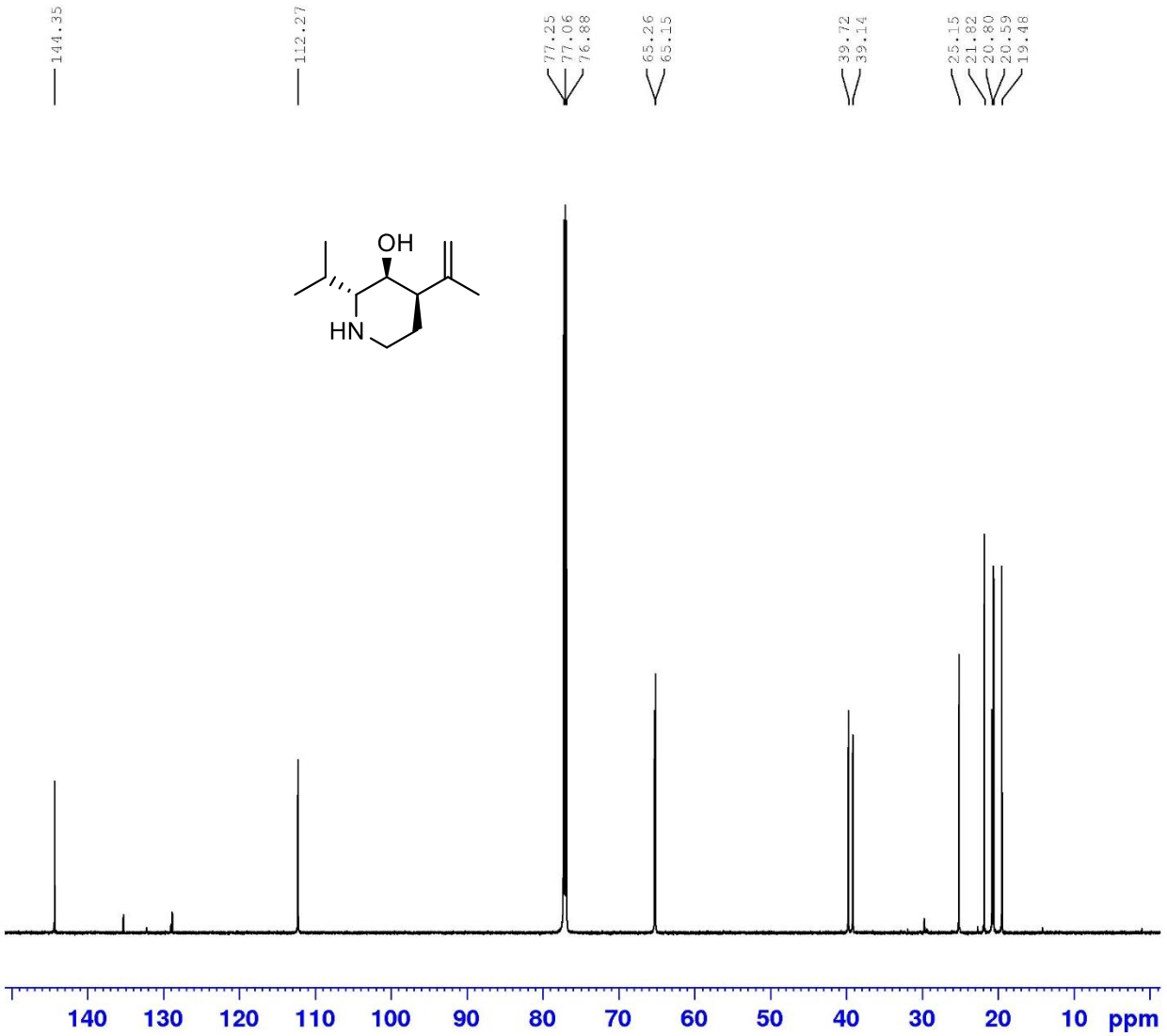
^{13}C-NMR (*S*,*R*)-**30b**

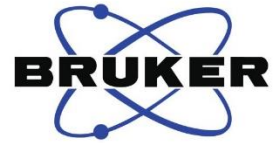


BRUKER

```
Current Data Parameters
NAME          May31-2021
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210531
Time               14.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   128
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530151 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

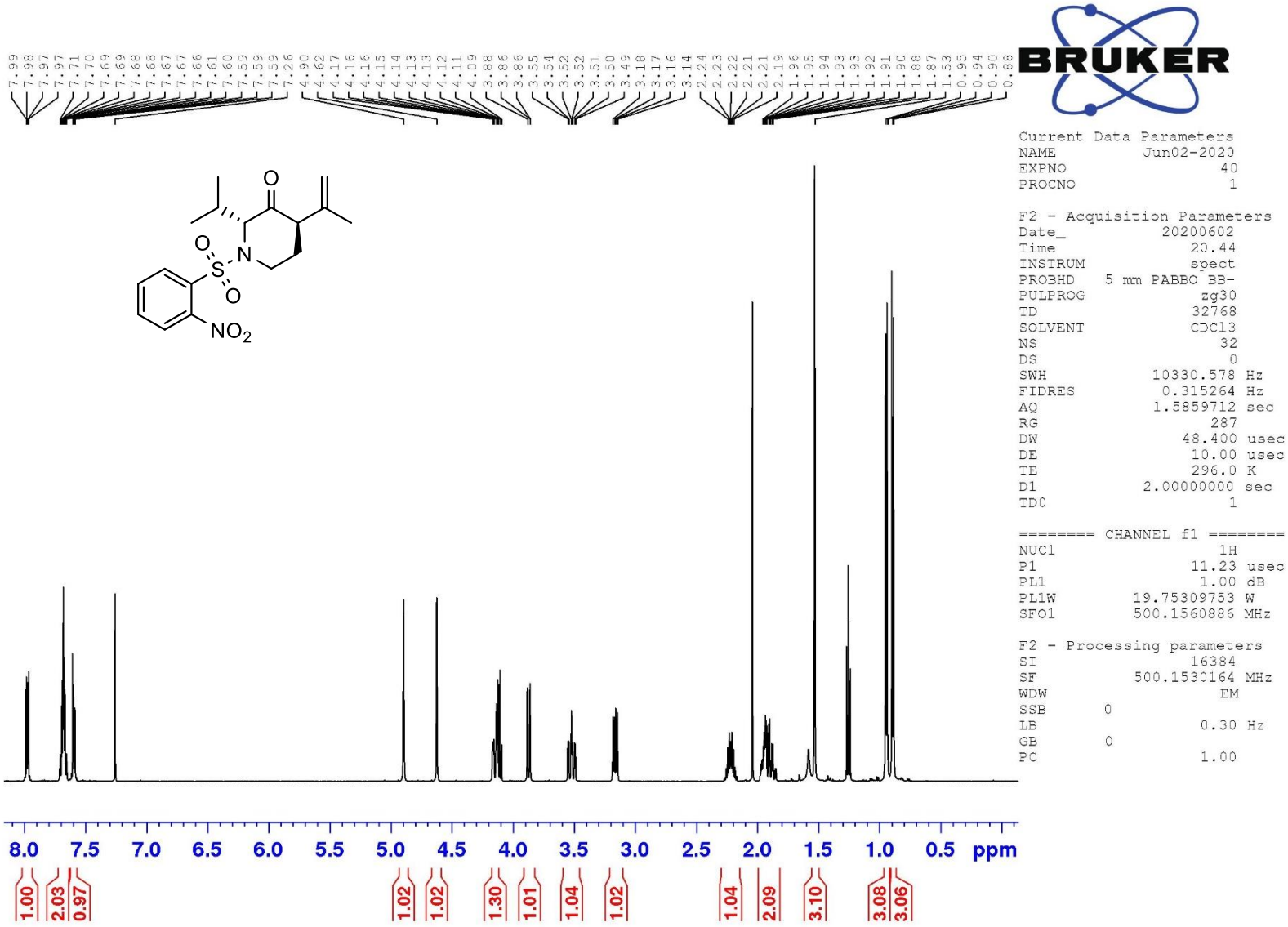
^{1}H-NMR (*S*,*R*)-**31b**



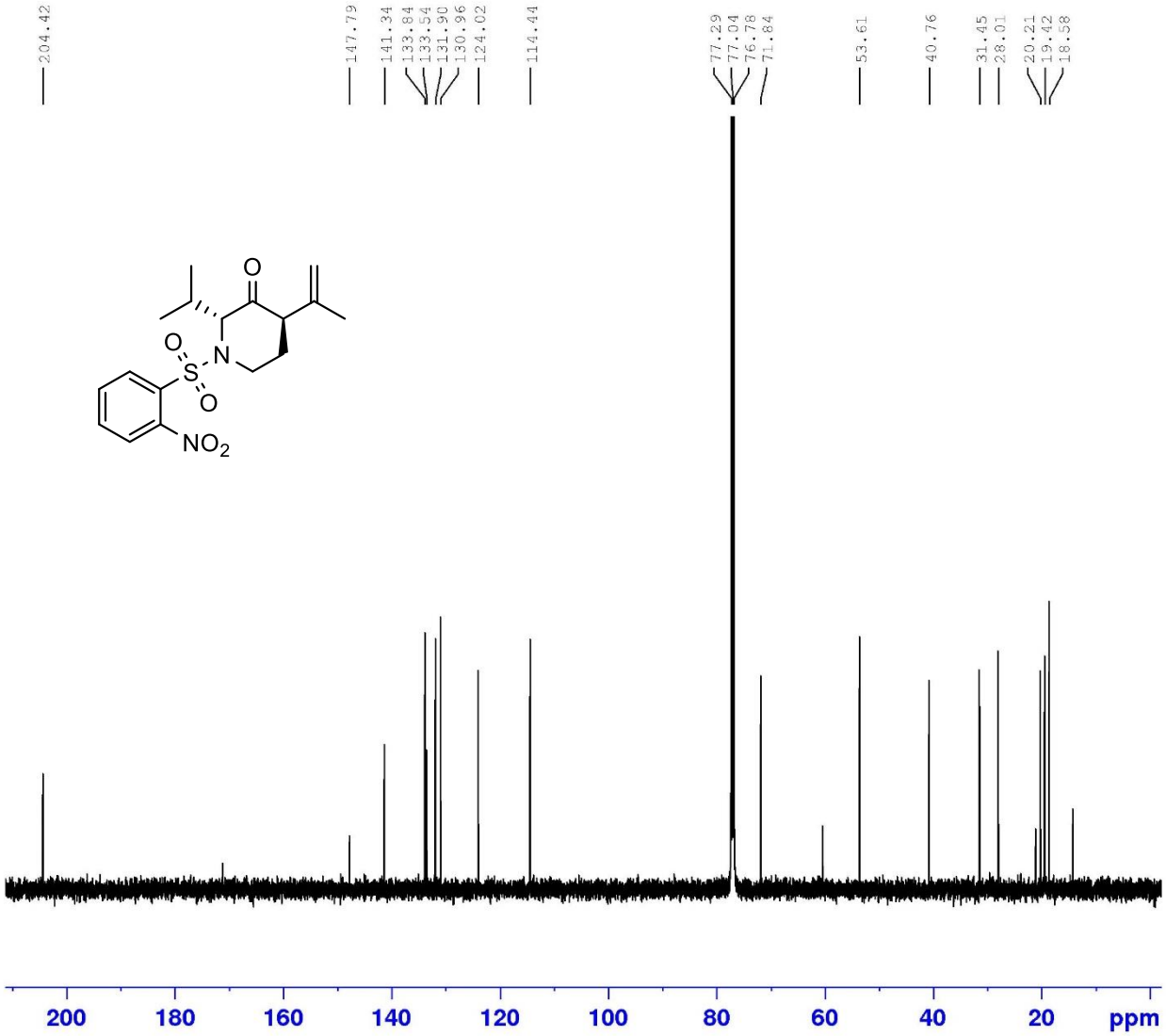



```
Current Data Parameters
NAME          May31-2021
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210531
Time               10.41
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2           9.89999962 W
PLW12          0.15564001 W
PLW13          0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C-NMR (*S*,*R*)-**31b**


7.99 7.98 7.97 7.97 7.71 7.70 7.69 7.69 7.68 7.68 7.67 7.67 7.66 7.61 7.60 7.59 7.59 7.59 7.26
4.90 4.62 4.17 4.16 4.16 4.15 4.14 4.13 4.13 4.12 4.11 4.09 3.88 3.86 3.86 3.55 3.54 3.52 3.52 3.51 3.50 3.49 3.18 3.17 3.16 3.14
2.24 2.23 2.22 2.21 2.21 2.19 1.96 1.95 1.94 1.93 1.93 1.92 1.91 1.90 1.88 1.87 1.53 0.95 0.94 0.90 0.88
BRUKER
Current Data Parameters
NAME Jun02-2020
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200602
Time 20.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5859712 sec
RG 287
DW 48.400 usec
DE 10.00 usec
TE 296.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.23 usec
PL1 1.00 dB
PL1W 19.75309753 W
SFO1 500.1560886 MHz
F2 - Processing parameters
SI 16384
SF 500.1530164 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
S
O
O
NO2
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00 2.03 0.97 1.02 1.02 1.30 1.01 1.04 1.02 1.04 2.09 3.10 3.08 3.06

^{1}H-NMR (*R*)-**S2b**



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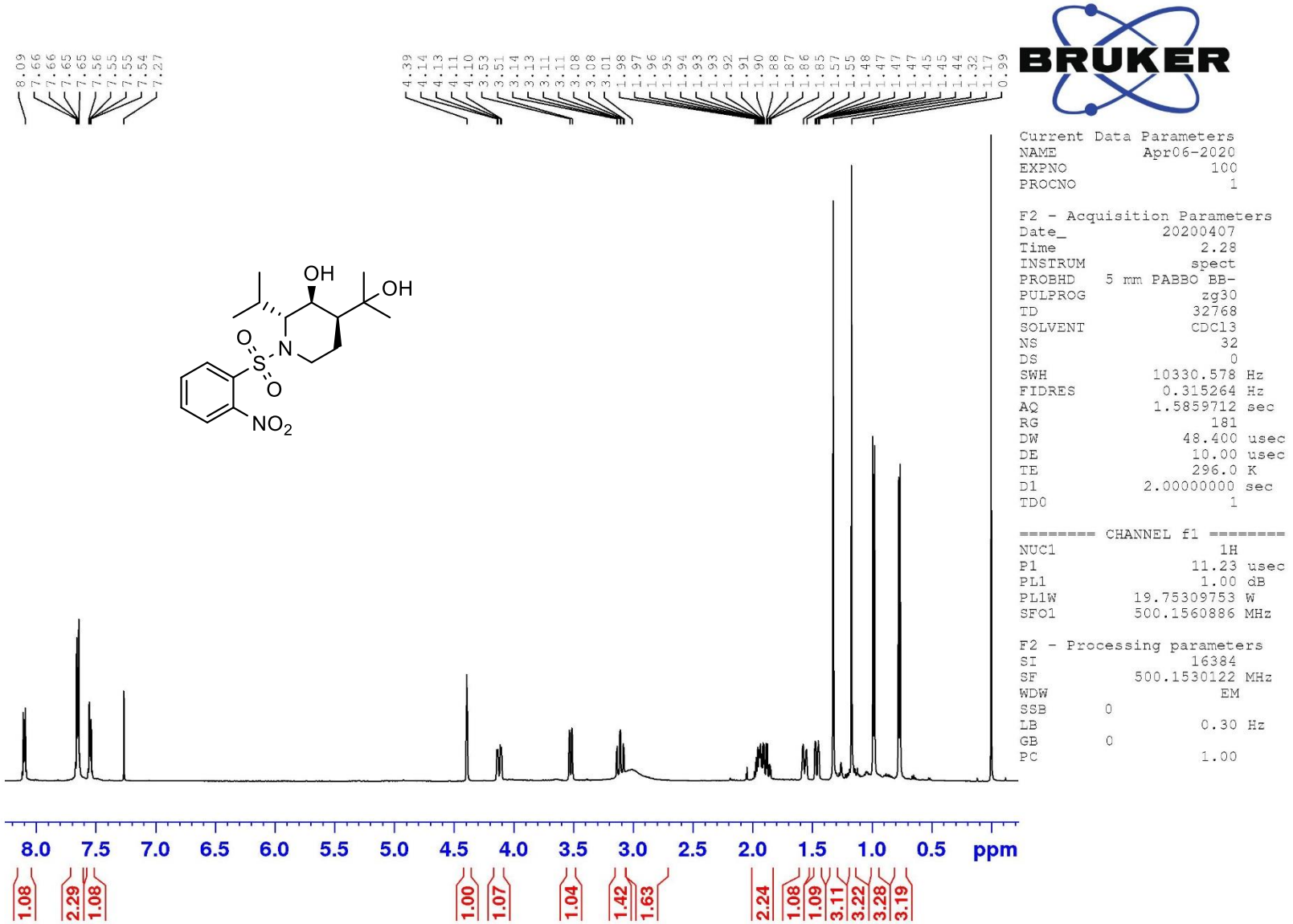
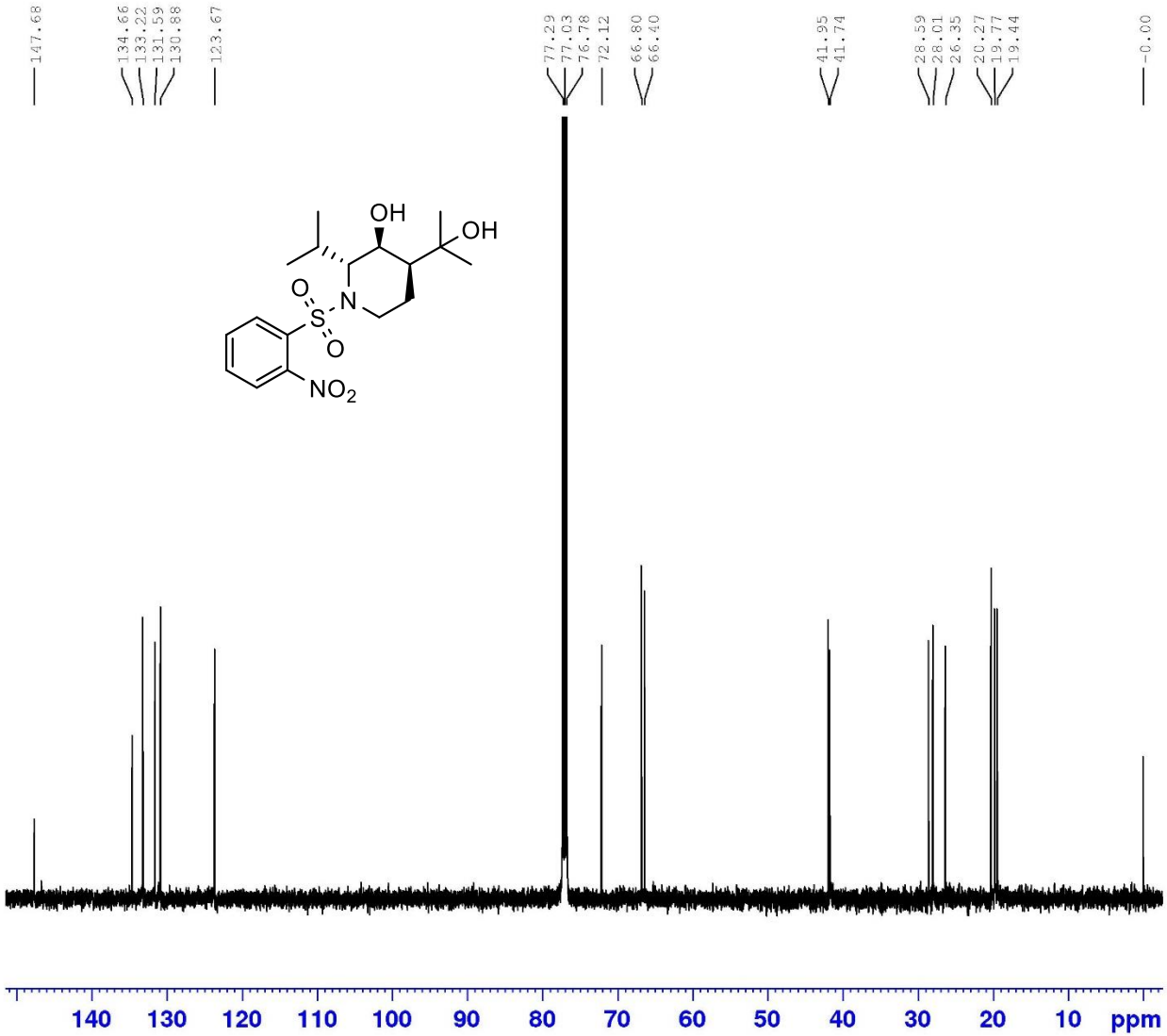
```
Current Data Parameters
NAME          Jun02-2020
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200602
Time               21.37
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*R*)-**S2b**




```
Current Data Parameters
NAME          Apr06-2020
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200407
Time                2.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   181
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*S*)-**33b**



Current Data Parameters
NAME Apr06-2020
EXPNO 101
PROCNO 1

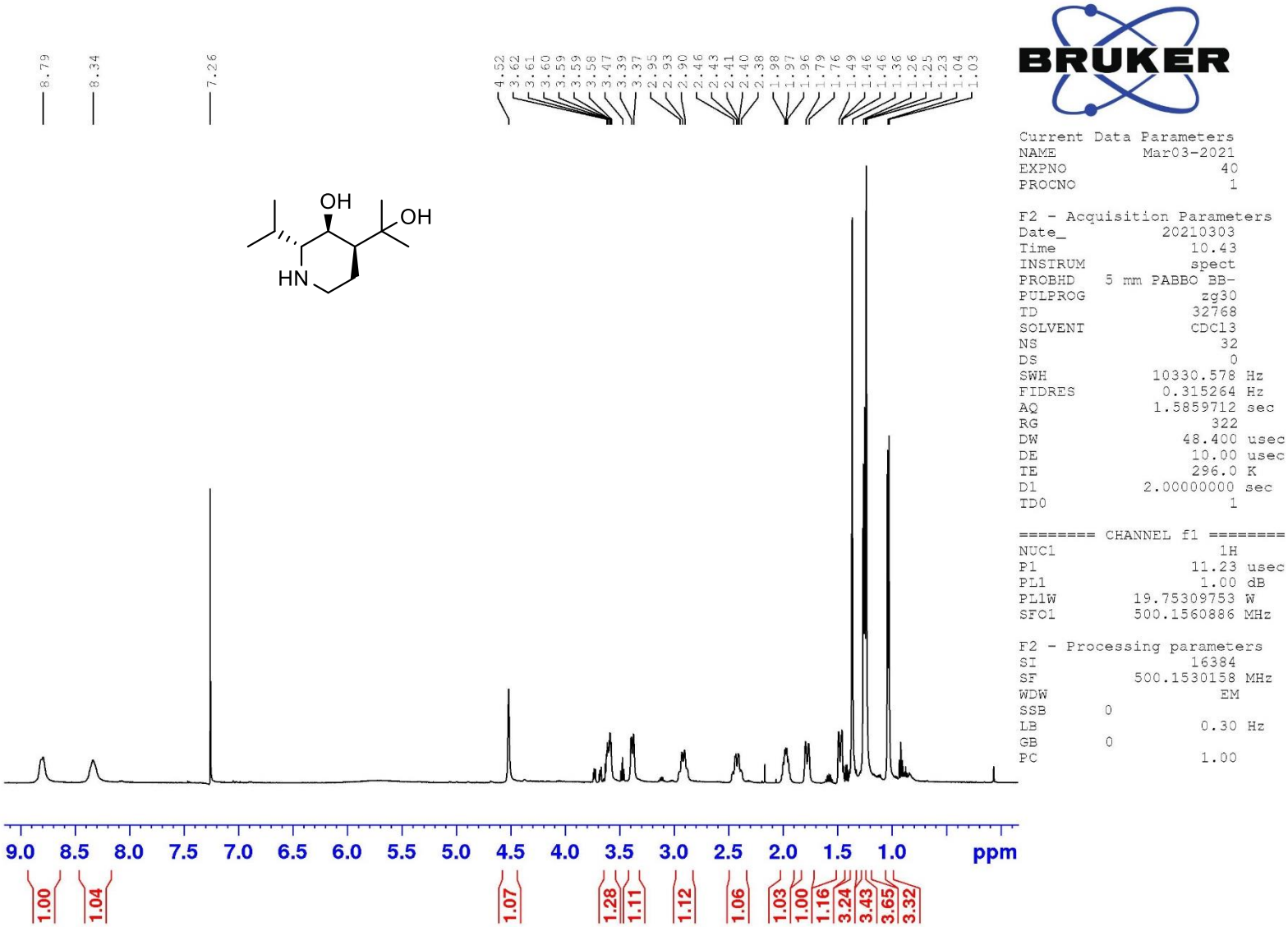
F2 - Acquisition Parameters
Date_ 20200407
Time 3.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 32894.738 Hz
FIDRES 0.501934 Hz
AQ 0.9961472 sec
RG 2580
DW 15.200 usec
DE 10.00 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.20 usec
PL1 1.50 dB
PL1W 51.74793243 W
SFO1 125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 19.99 dB
PL13 21.00 dB
PL2W 19.75309753 W
PL12W 0.24925002 W
PL13W 0.19753097 W
SFO2 500.1550006 MHz

F2 - Processing parameters
SI 32768
SF 125.7635742 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{13}C-NMR (*S*,*S*)-**33b**



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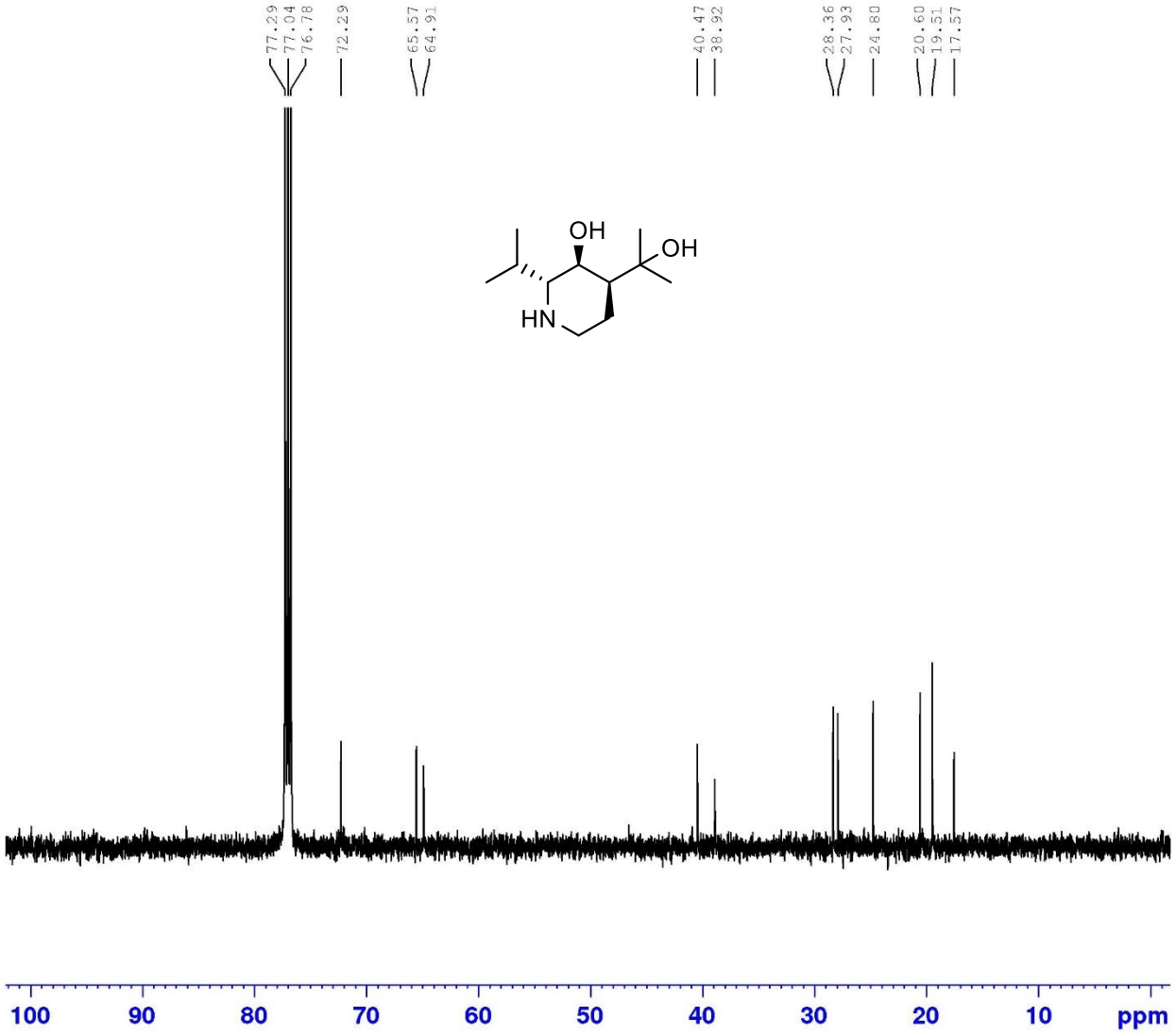
```
Current Data Parameters
NAME          Mar03-2021
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210303
Time               10.43
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530158 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

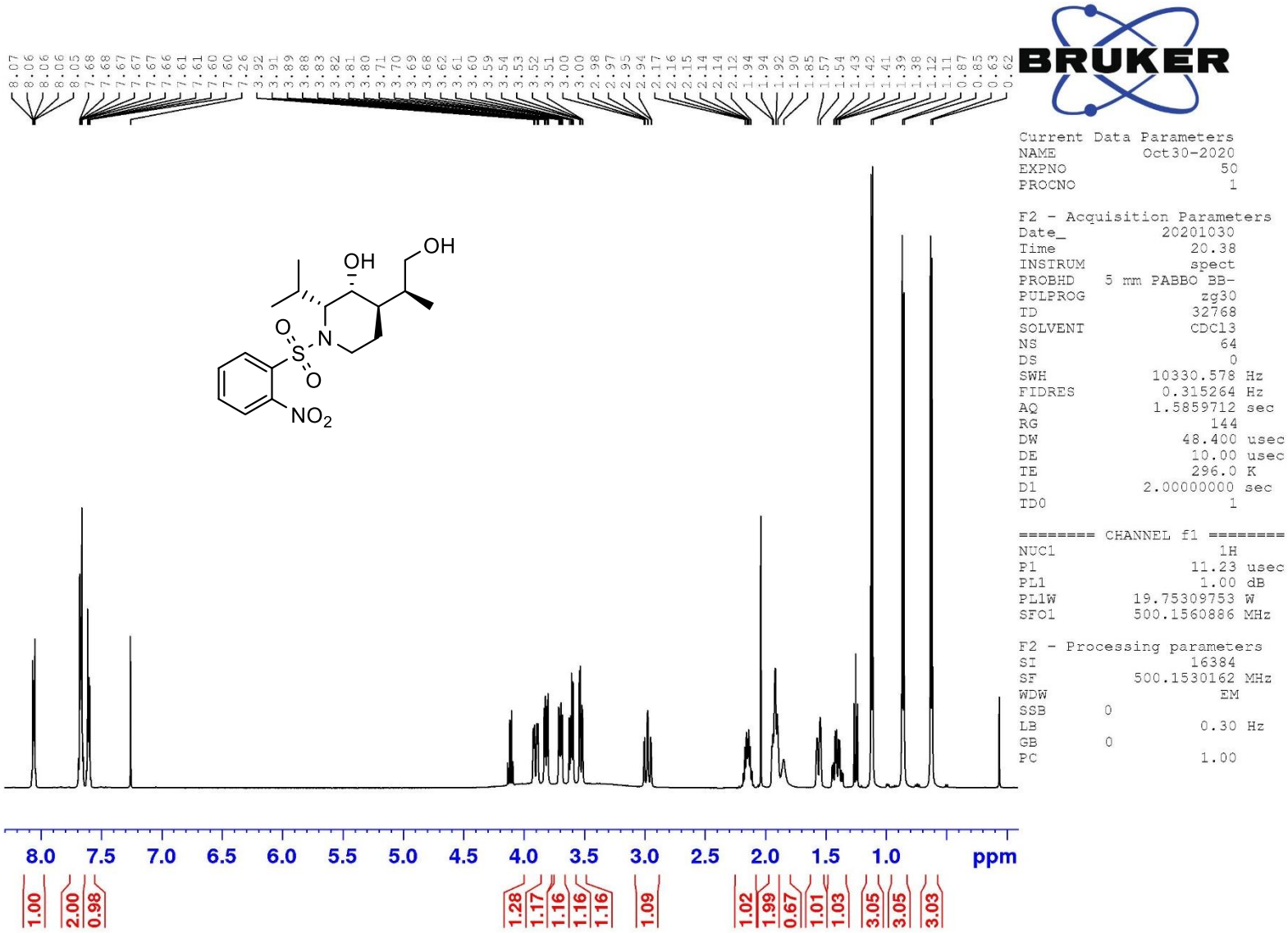
^{1}H-NMR (*S*,*S*)-**33b**



BRUKER

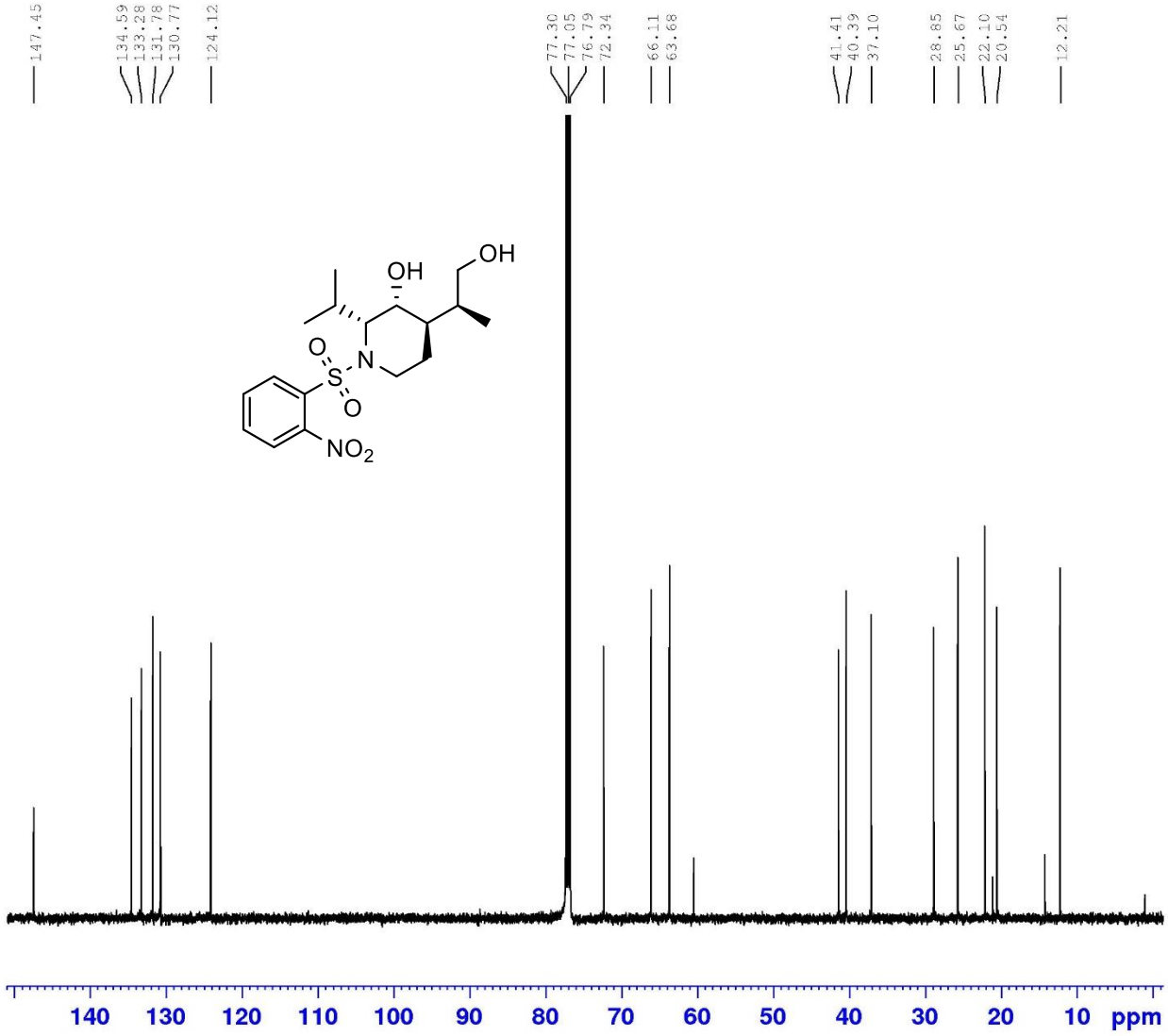
```
Current Data Parameters
NAME          Mar03-2021
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210303
Time              11.37
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*S*,*S*)-**33b**



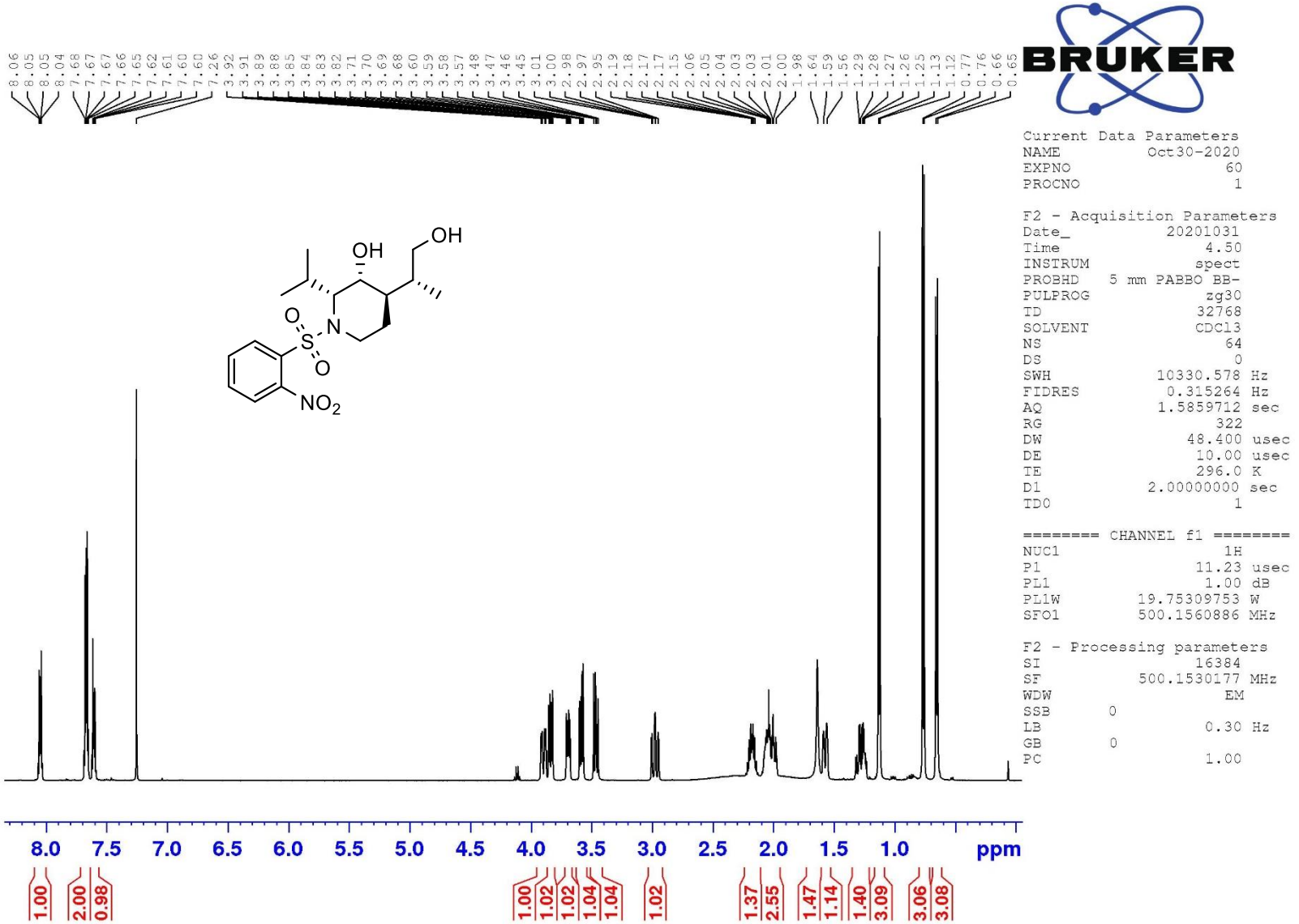

```
Current Data Parameters
NAME          Oct30-2020
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201030
Time               20.38
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   144
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530162 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*R*,*R*,*S*)-**34b**



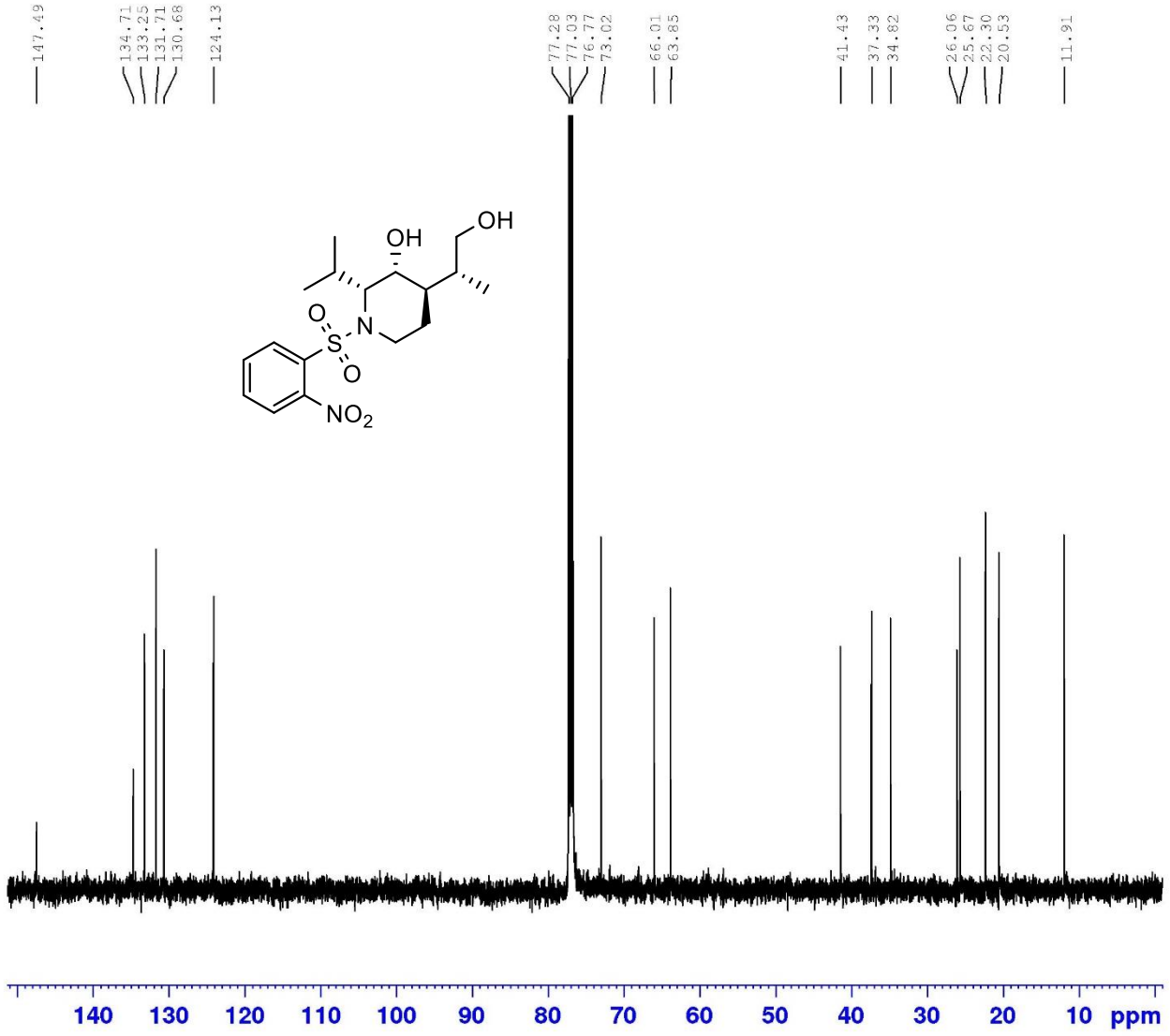
^{13}C-NMR (*R*,*R*,*S*)-**34b**

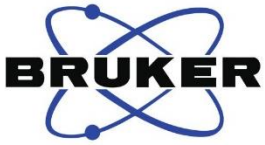
```
Current Data Parameters
NAME          Oct30-2020
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201030
Time               22.24
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           32894.738 Hz
FIDRES        0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```




```
Current Data Parameters
NAME          Oct30-2020
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201031
Time                4.50
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530177 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

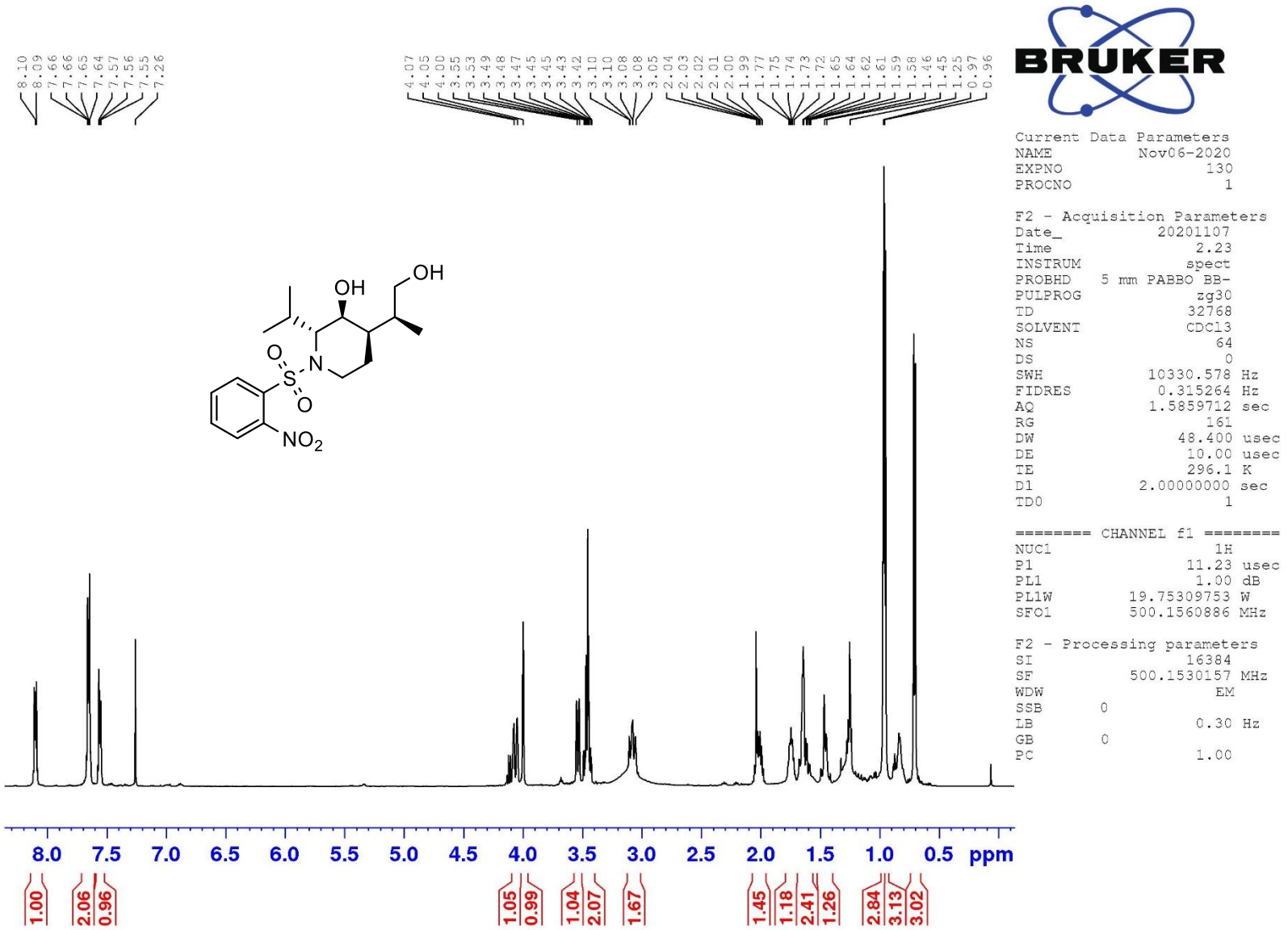
^{1}H-NMR (*R*,*R*,*R*)-**34b**





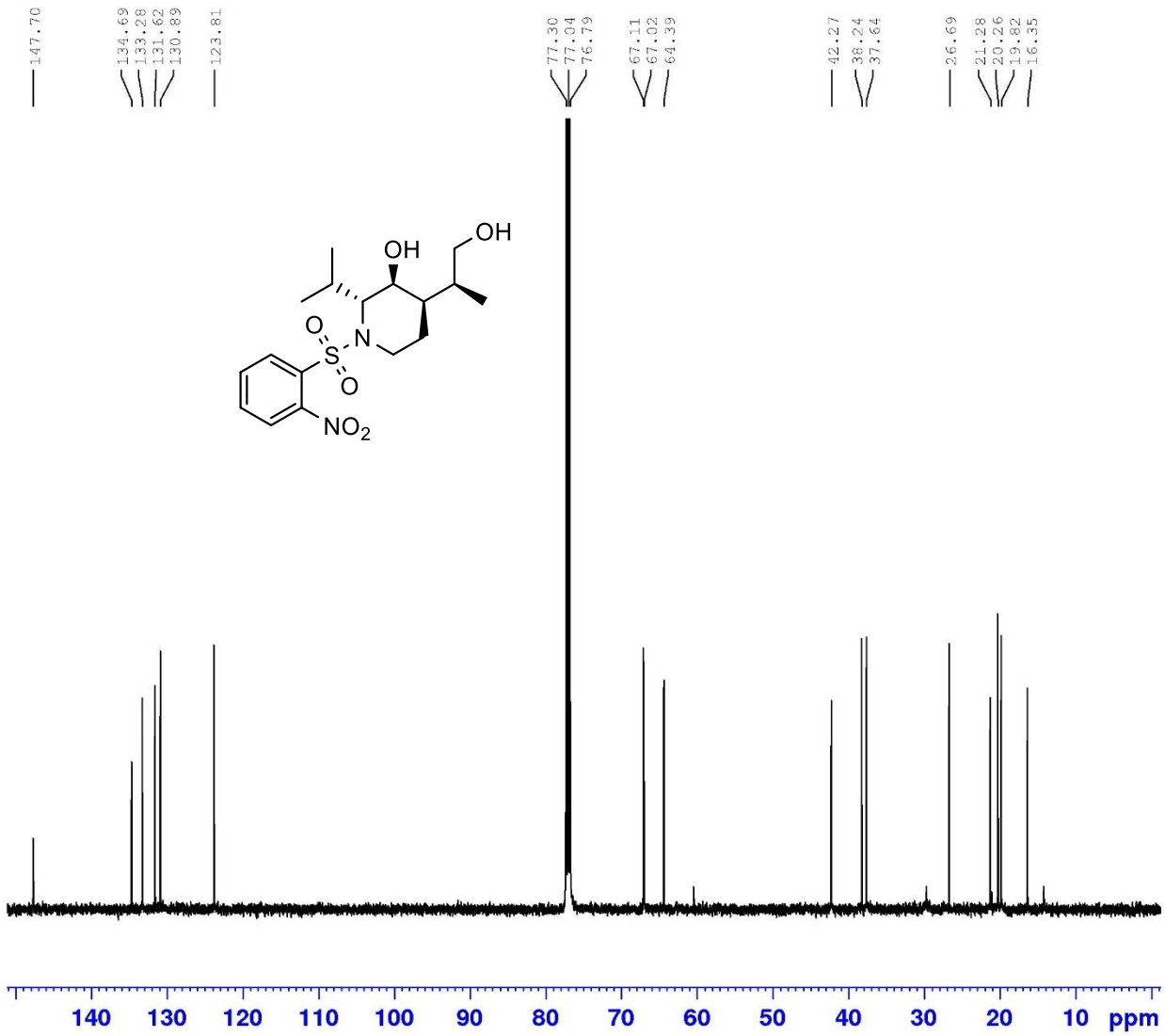

```
Current Data Parameters
NAME          Oct30-2020
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201031
Time                6.35
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                  32768
SF           125.7635720 MHz
WDW                    EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

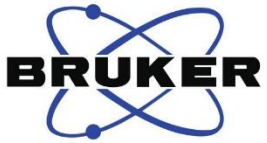
^{13}C-NMR (*R*,*R*,*R*)-**34b**




```
Current Data Parameters
NAME          Nov06-2020
EXPNO                130
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201107
Time                2.23
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     64
DS                      0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                    161
DW                48.400 usec
DE                10.00 usec
TE                296.1 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530157 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

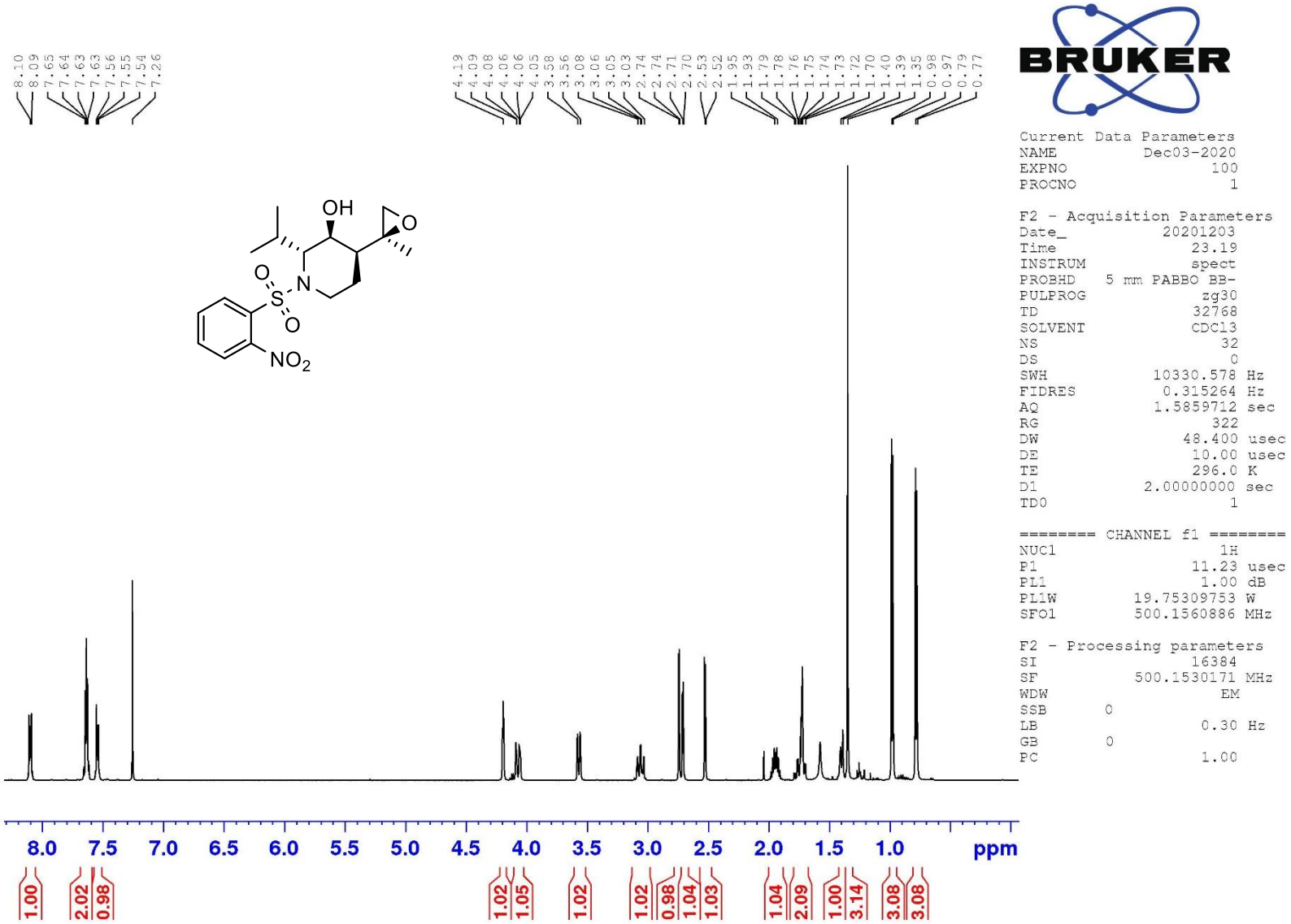
^{1}H-NMR (*S*,*R*,*S*)-**34b**





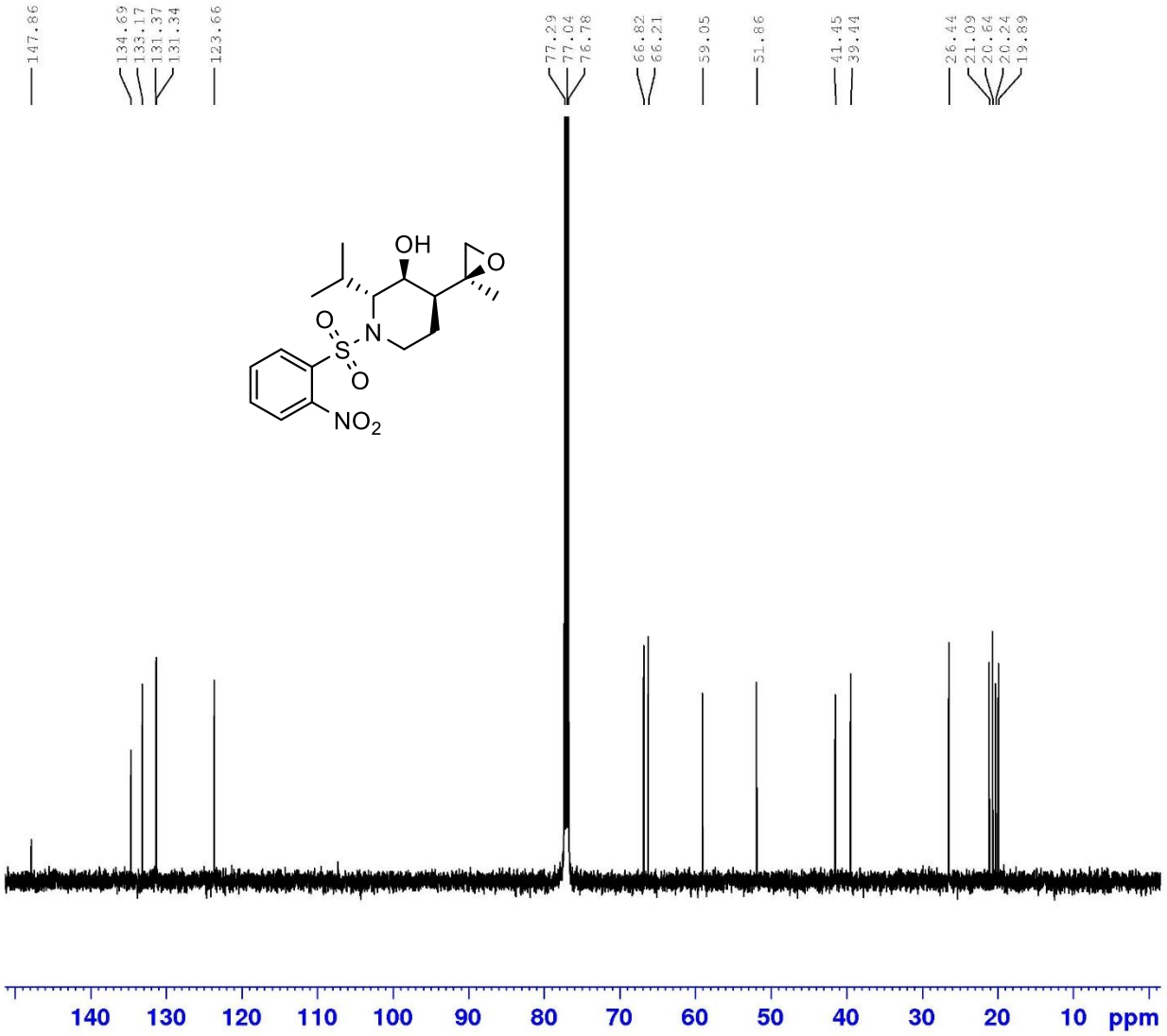

```
Current Data Parameters
NAME          Nov06-2020
EXPNO                131
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201107
Time                4.10
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                297.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                32768
SF         125.7635720 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C-NMR (*S*,*R*,*S*)-**34b**




```
Current Data Parameters
NAME          Dec03-2020
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201203
Time               23.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530171 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

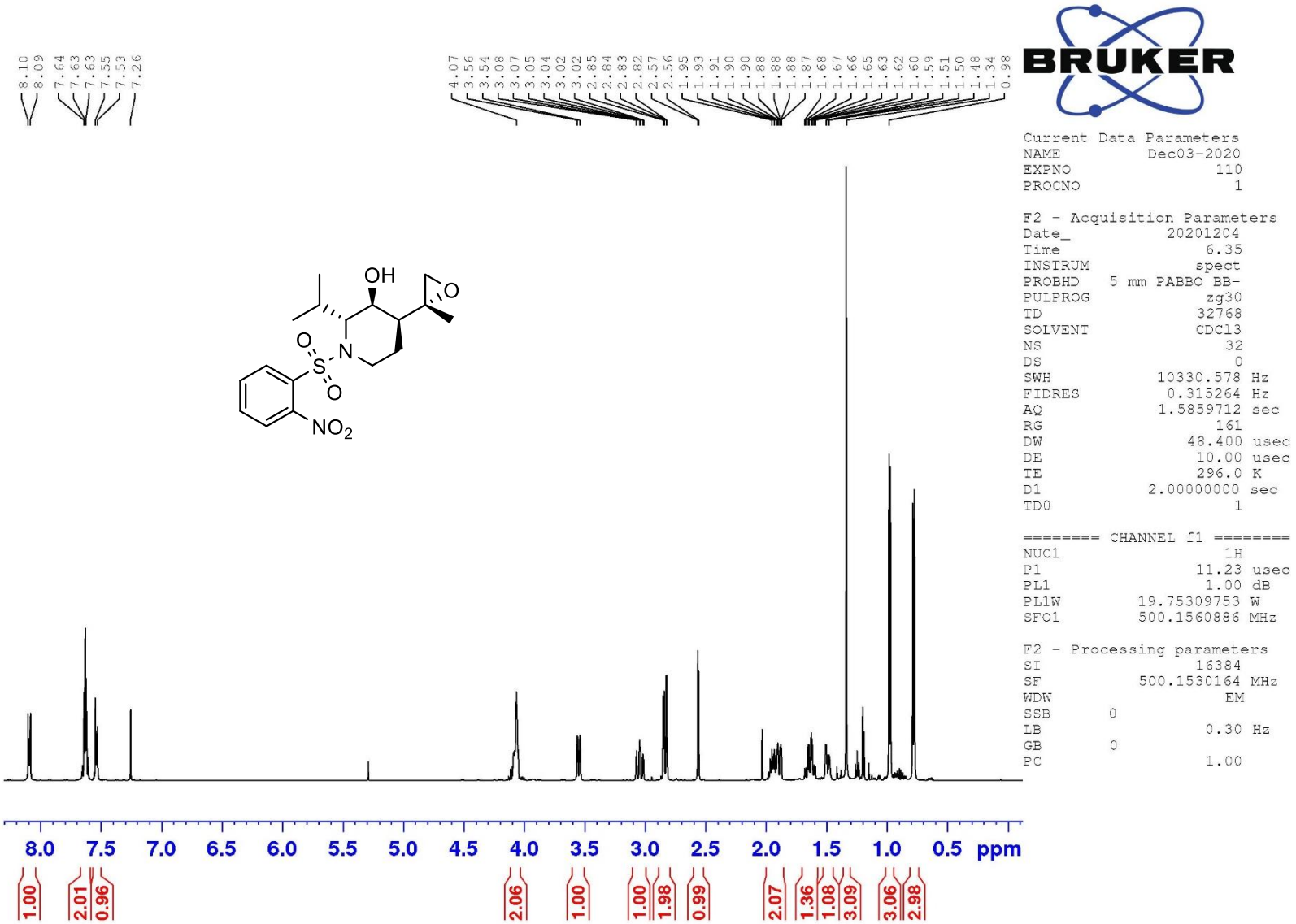
^{1}H-NMR (*S*,*S*,*S*)-**35b**



BRUKER

```
Current Data Parameters
NAME          Dec03-2020
EXPNO                101
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201204
Time                0.12
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W        51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

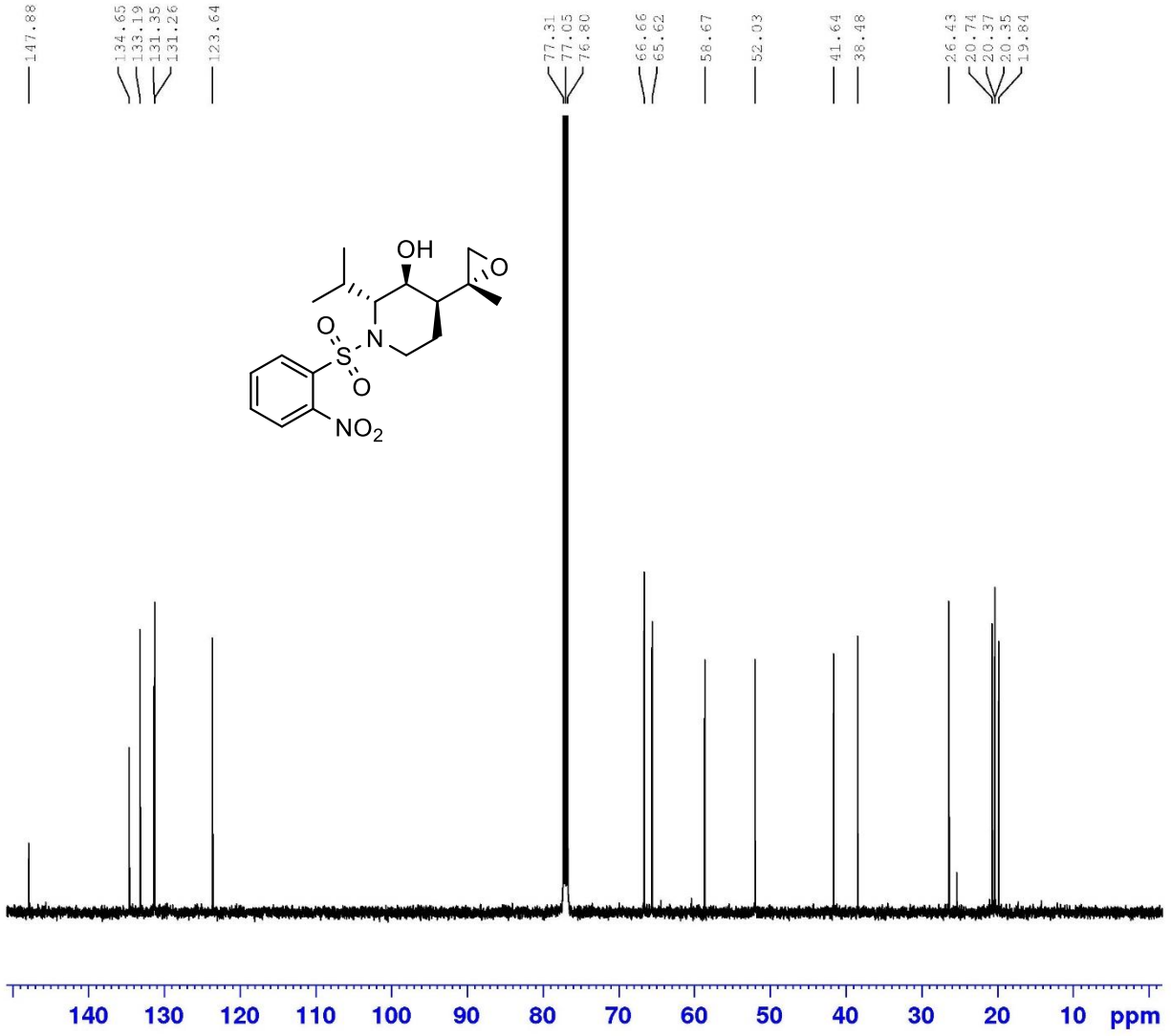
^{13}C-NMR (*S*,*S*,*S*)-**35b**



BRUKER

```
Current Data Parameters
NAME          Dec03-2020
EXPNO                110
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201204
Time                6.35
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   161
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530164 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

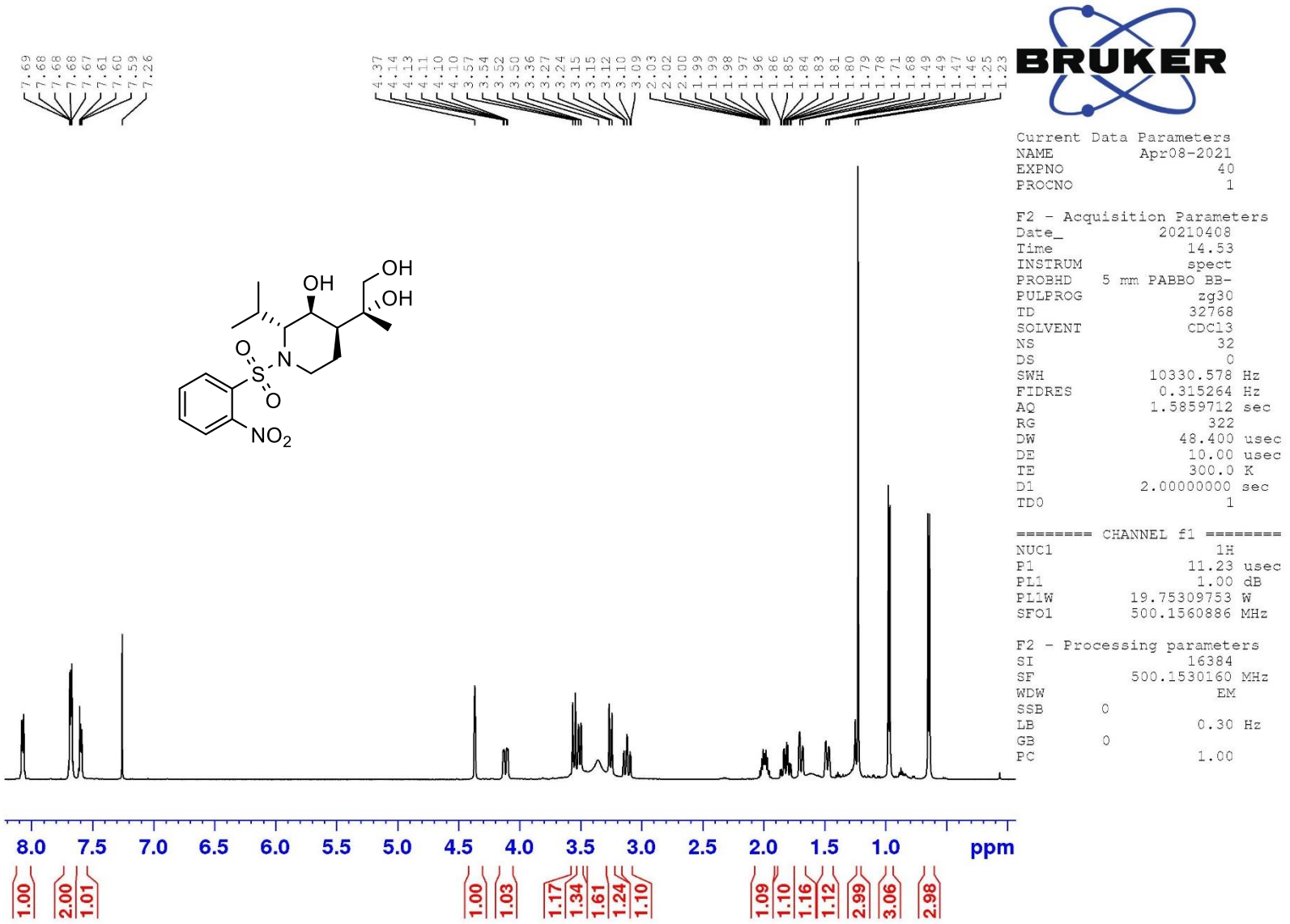
^{1}H-NMR (*S*,*S*,*R*)-**35b**



BRUKER

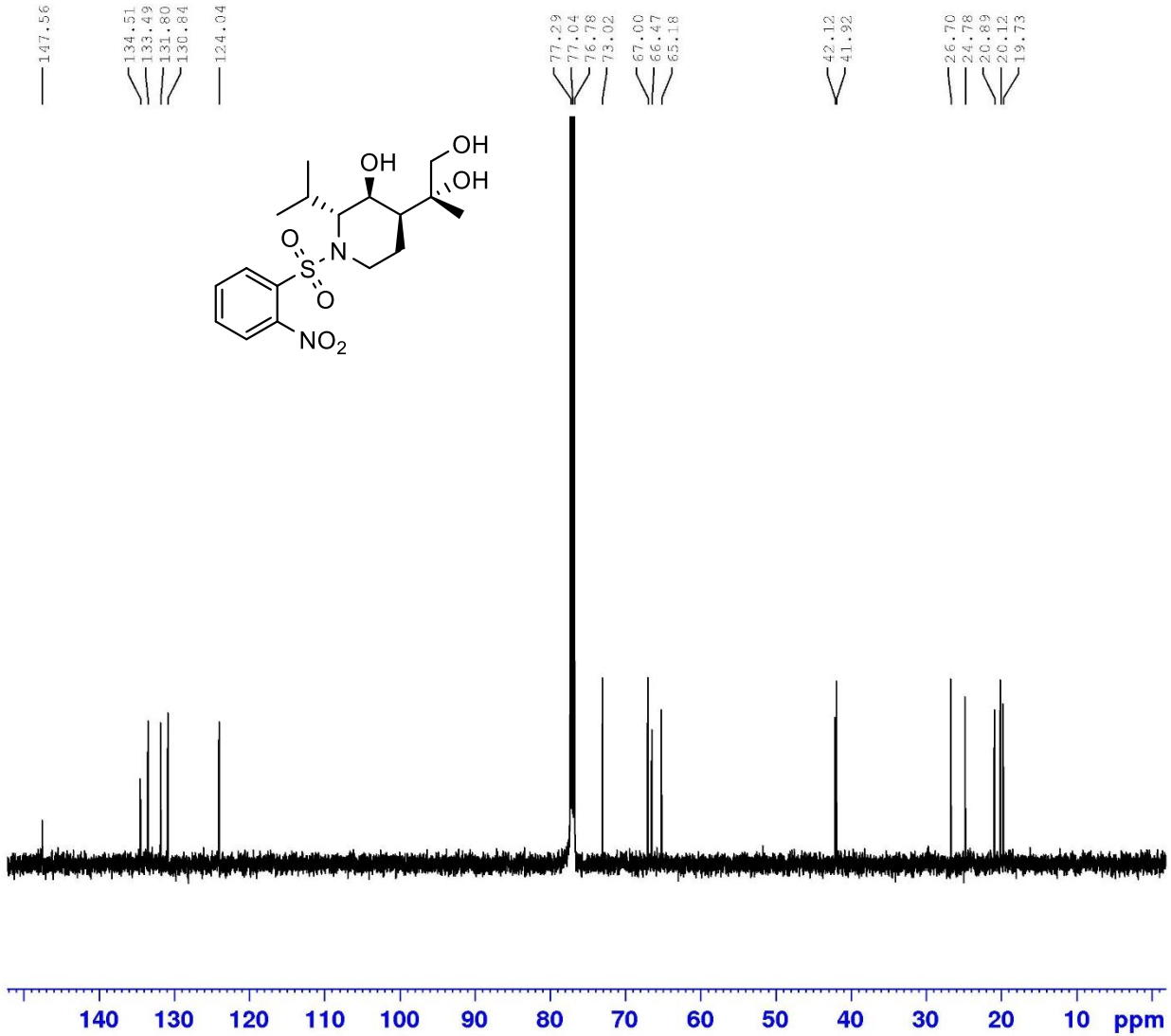
```
Current Data Parameters
NAME          Dec03-2020
EXPNO                111
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201204
Time                7.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W        51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                  32768
SF          125.7635720 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

^{13}C-NMR (*S*,*S*,*R*)-**35b**




```
Current Data Parameters
NAME          Apr08-2021
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210408
Time               14.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530160 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*S*,*R*)-**36b**



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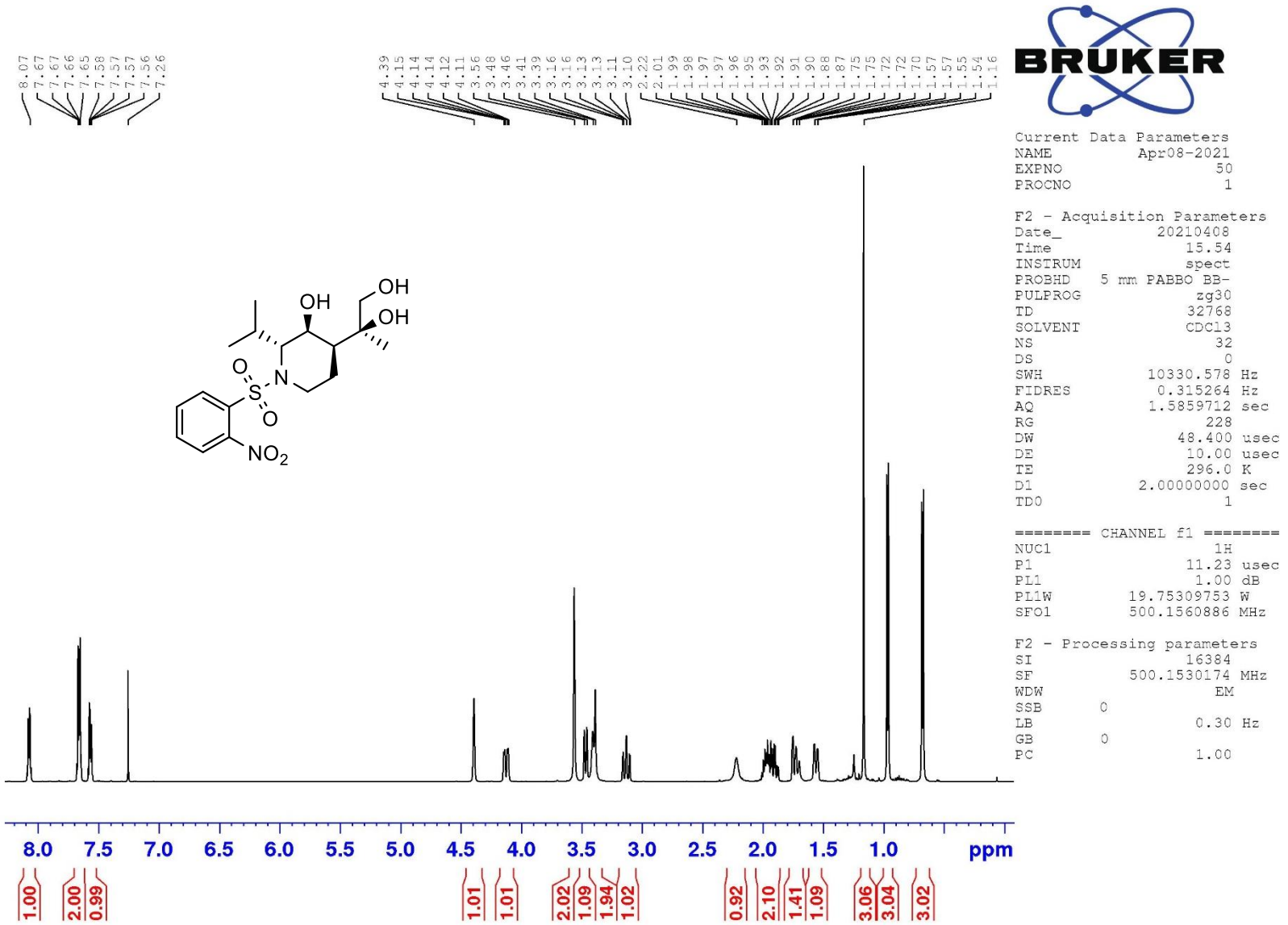
```
Current Data Parameters
NAME          Apr08-2021
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210408
Time               15.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*S*,*S*,*R*)-**36b**



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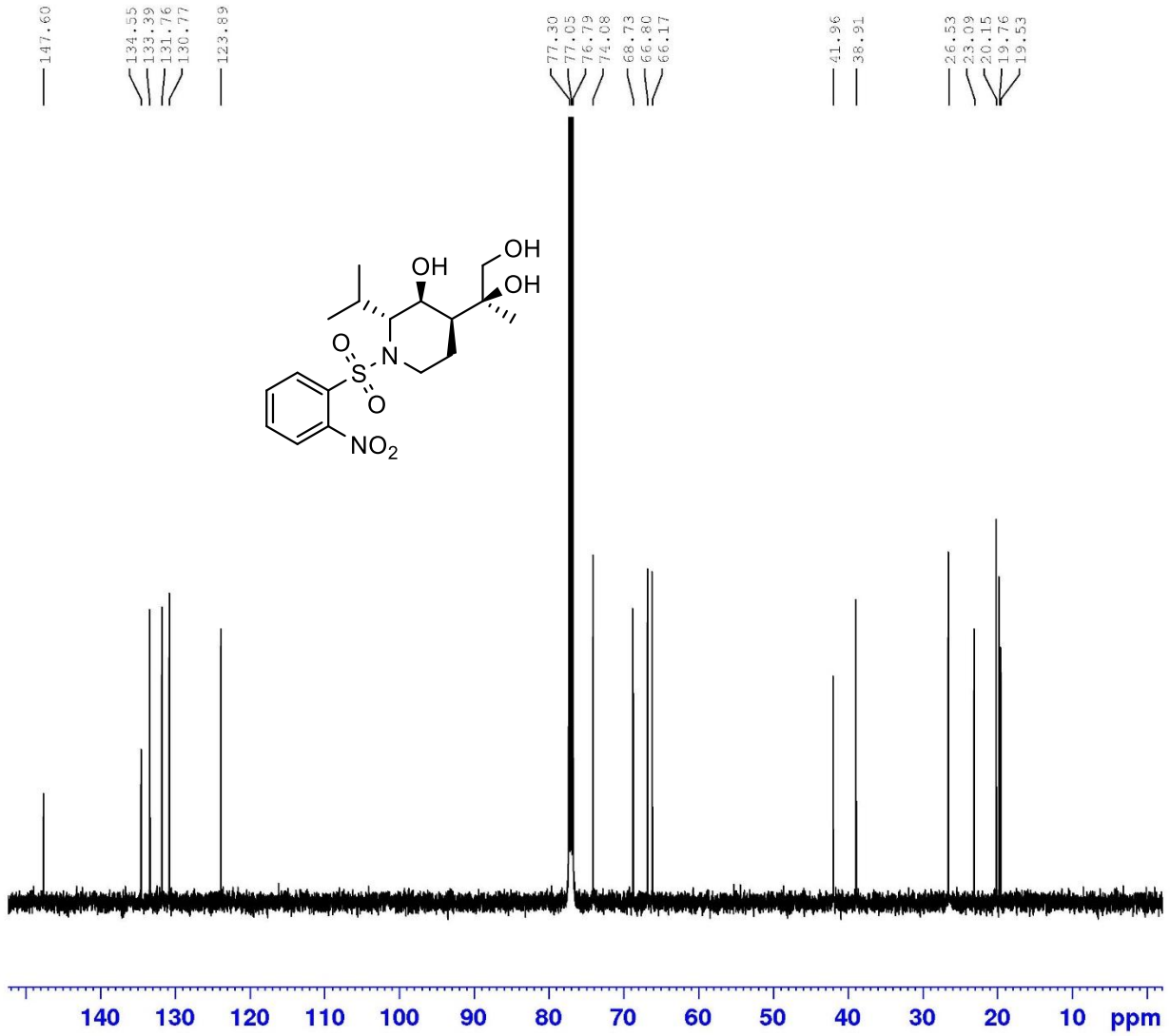
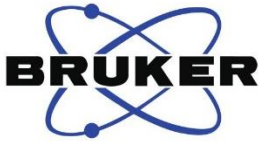
```
Current Data Parameters
NAME          Apr08-2021
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210408
Time               15.54
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   228
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530174 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

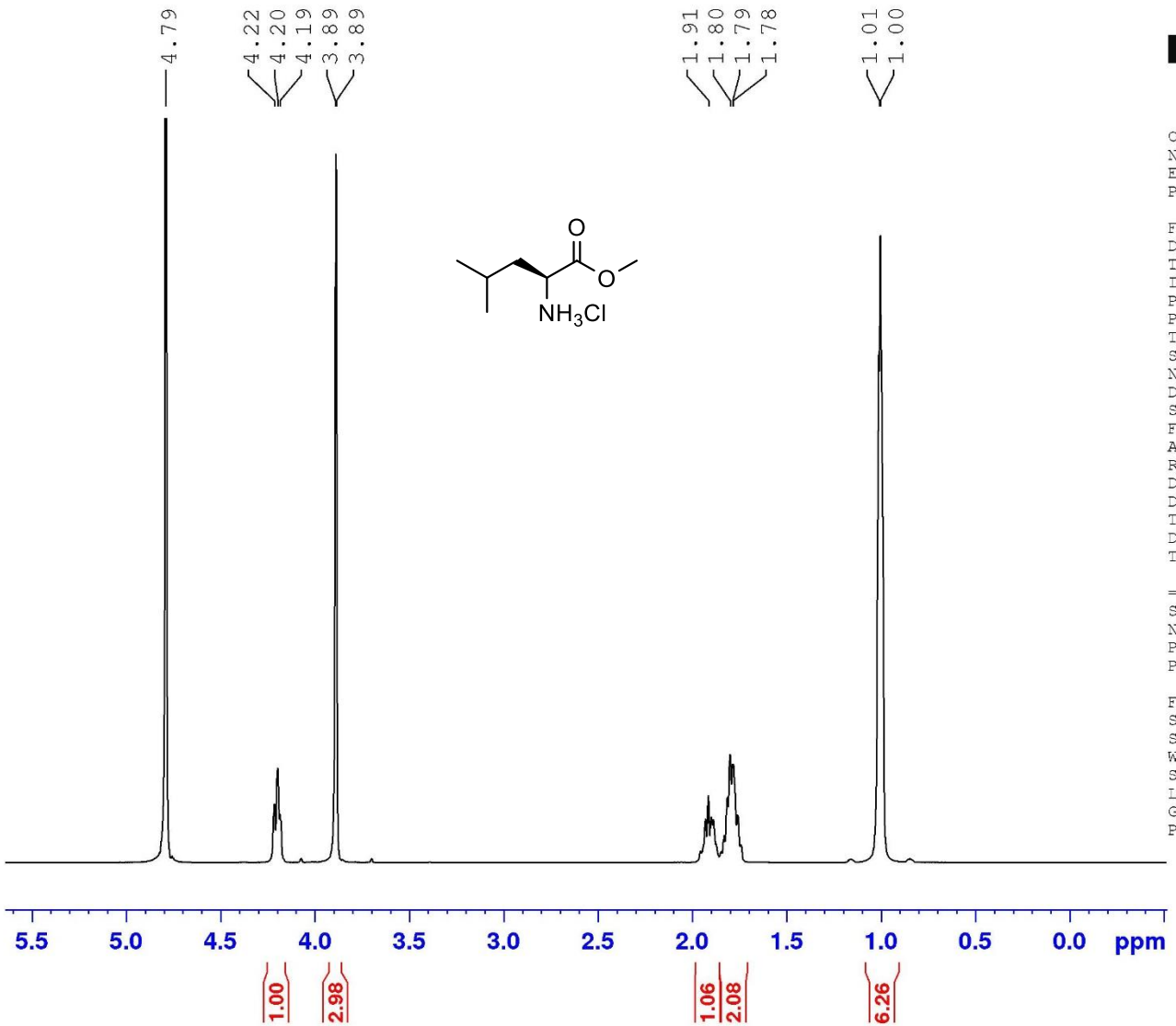
^{1}H-NMR (*S*,*S*,*S*)-**36b**





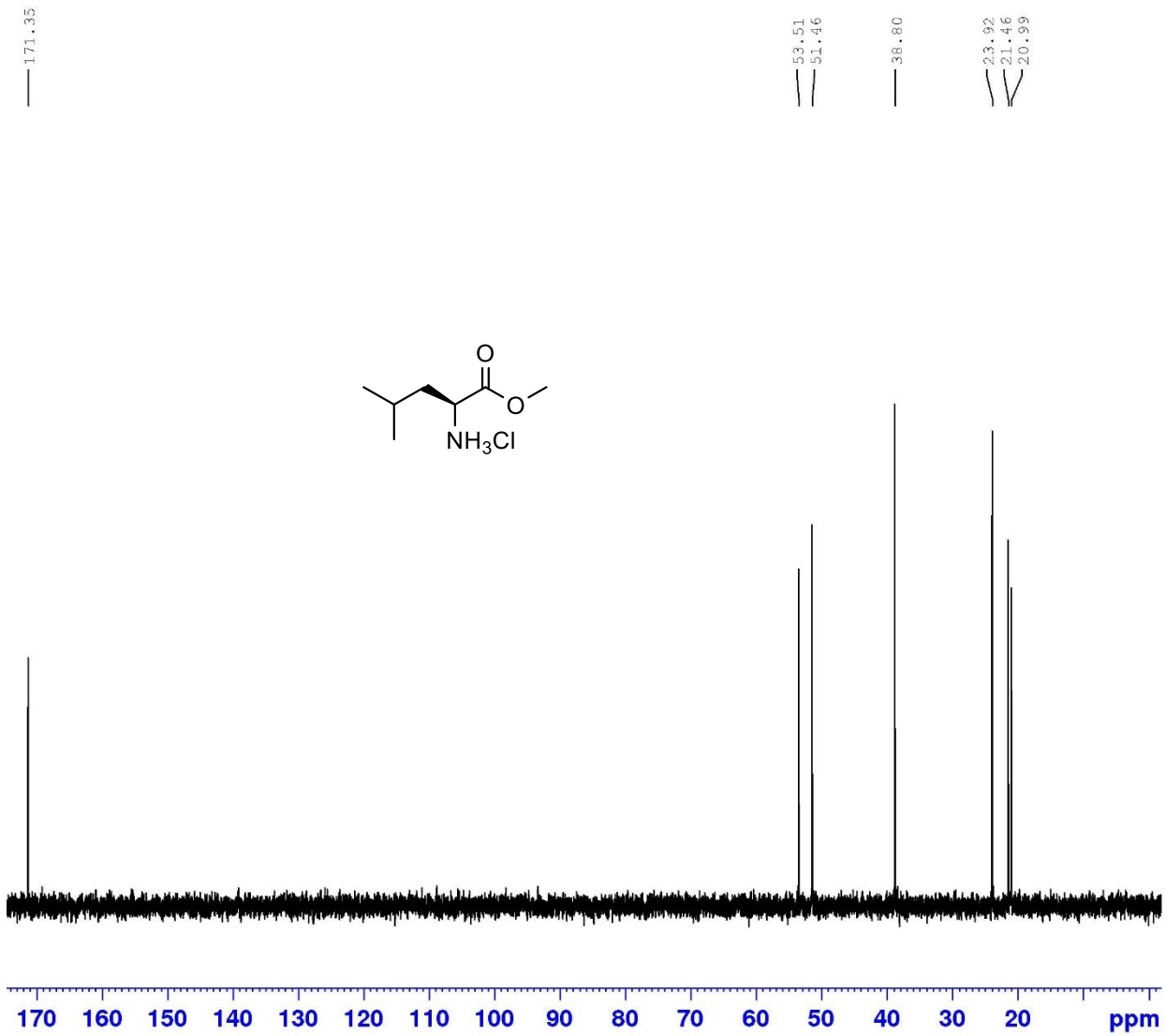

```
Current Data Parameters
NAME          Apr08-2021
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210408
Time               16.48
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF         125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*S*,*S*,*S*)-**36b**




```
Current Data Parameters
NAME          Mar05-2020
EXPNO                360
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200306
Time                0.19
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT              D2O
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 124.07
DW                 62.400 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1024708 MHz
NUC1                   1H
P1                  13.70 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.0999646 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

^{1}H-NMR **25c**



^{13}C-NMR **25c**

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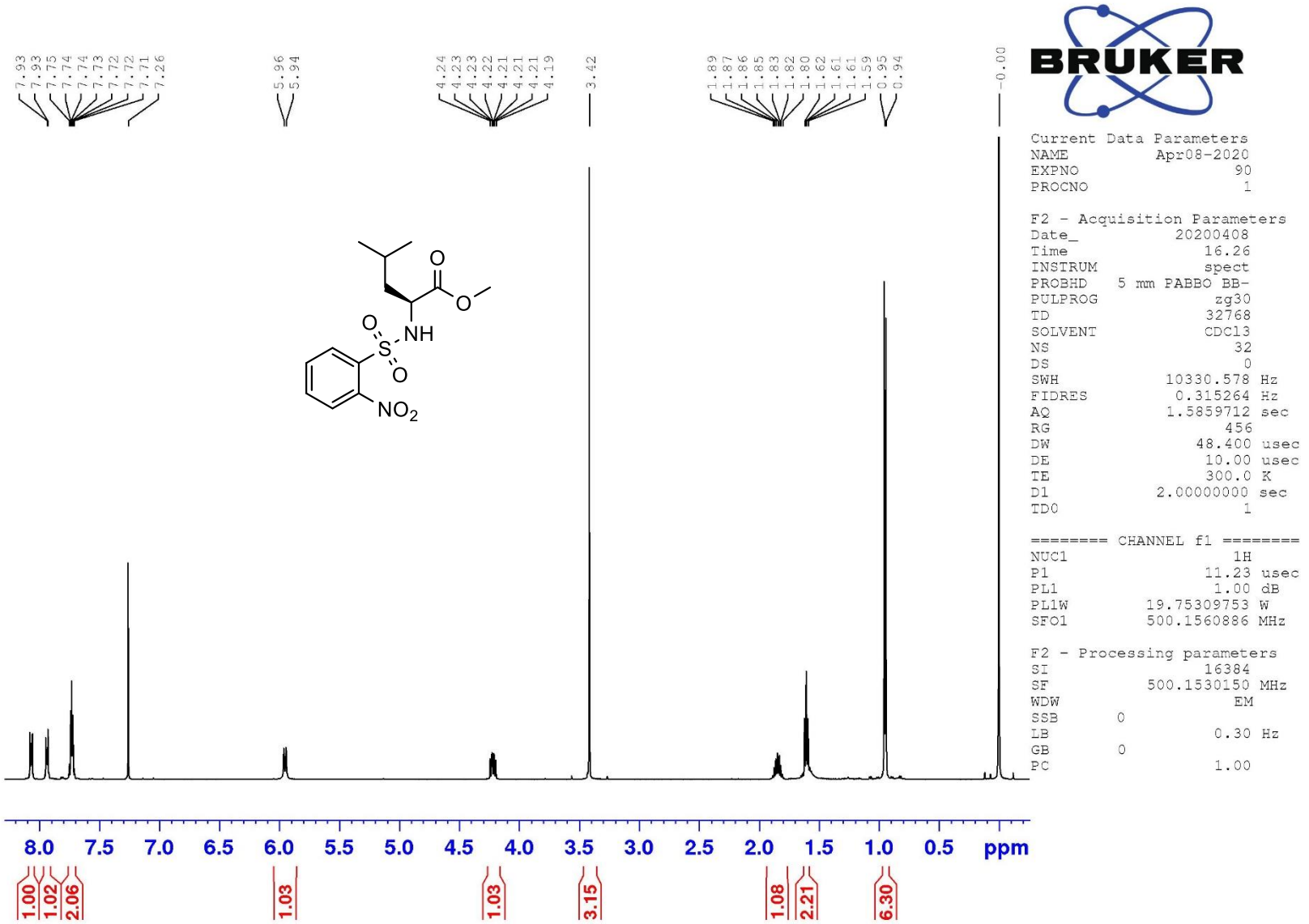
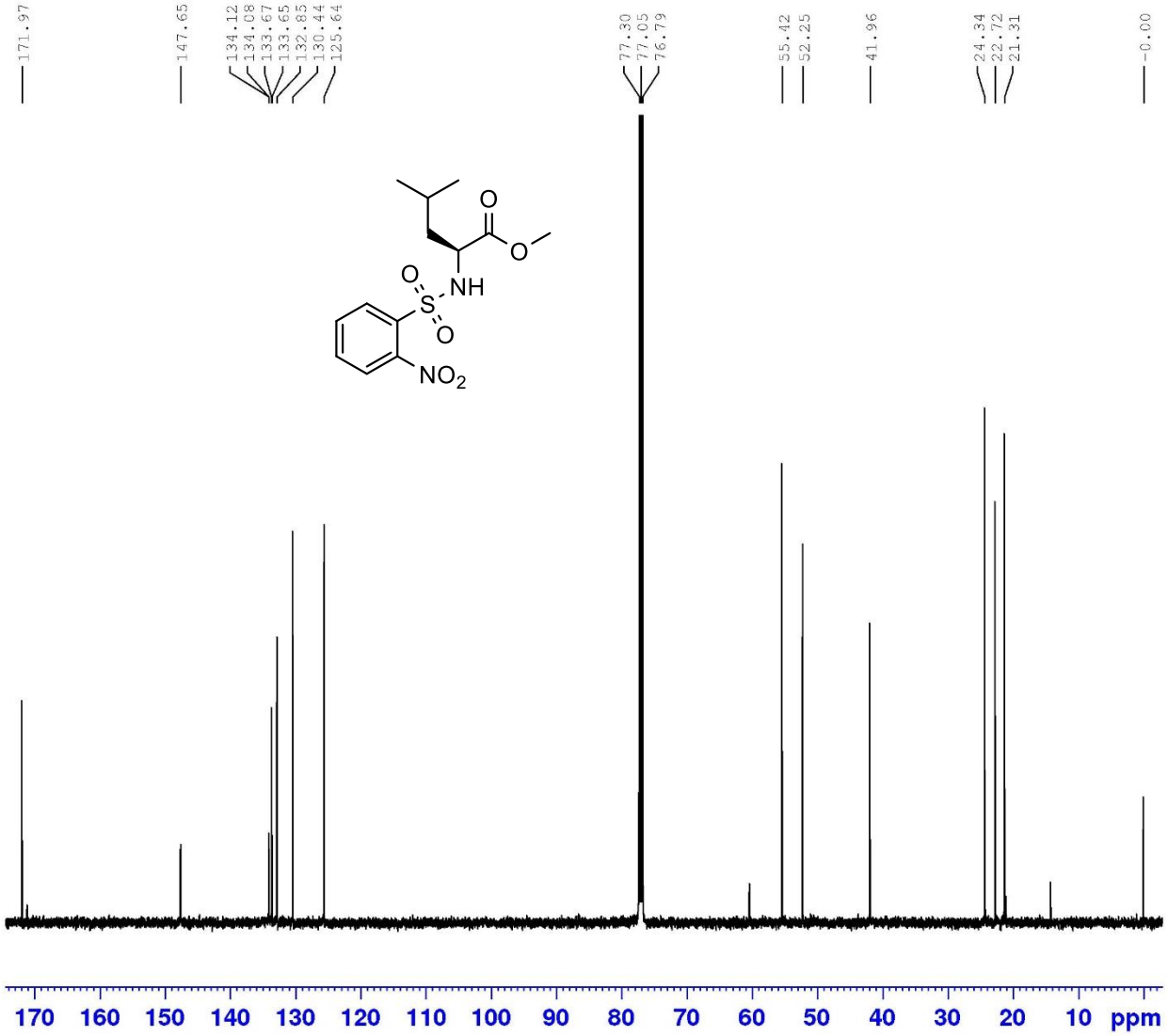
```
Current Data Parameters
NAME          Mar05-2020
EXPNO                361
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200306
Time                0.49
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT             D2O
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               205.35
DW               20.800 usec
DE                6.50 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6152851 MHz
NUC1                13C
P1                10.00 usec
PLW1        48.00000000 W

======== CHANNEL f2 ========
SFO2        400.1016004 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.27805999 W
PLW13        0.22522999 W

F2 - Processing parameters
SI                32768
SF          100.6052250 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



BRUKER

```
Current Data Parameters
NAME          Apr08-2020
EXPNO                 90
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200408
Time               16.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   456
DW                48.400 usec
DE                10.00 usec
TE                300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530150 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

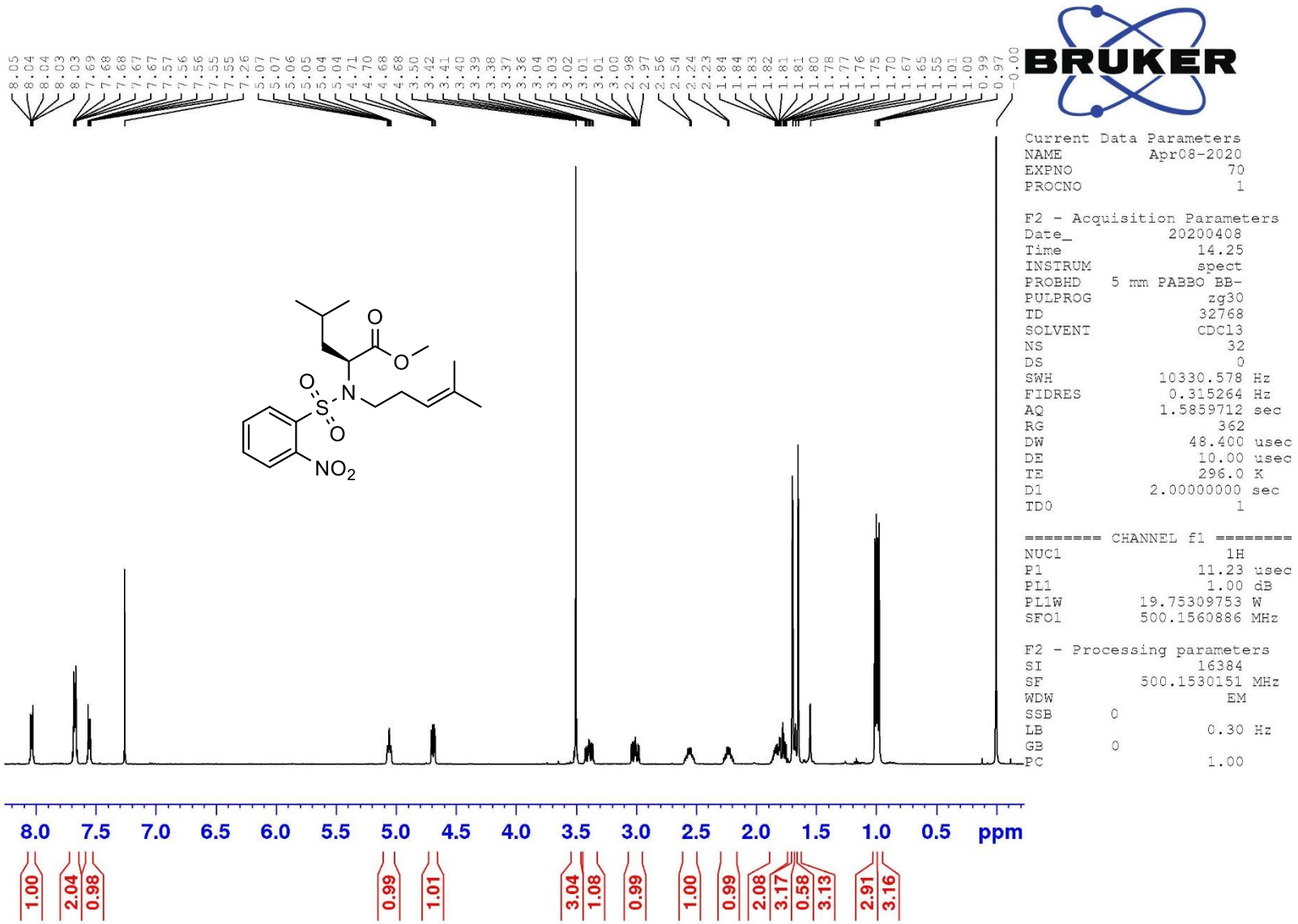
^{1}H-NMR **26c**



BRUKER

```
Current Data Parameters
NAME          Apr23-2020
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200424
Time                8.33
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                10.20 usec
PL1                1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635737 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

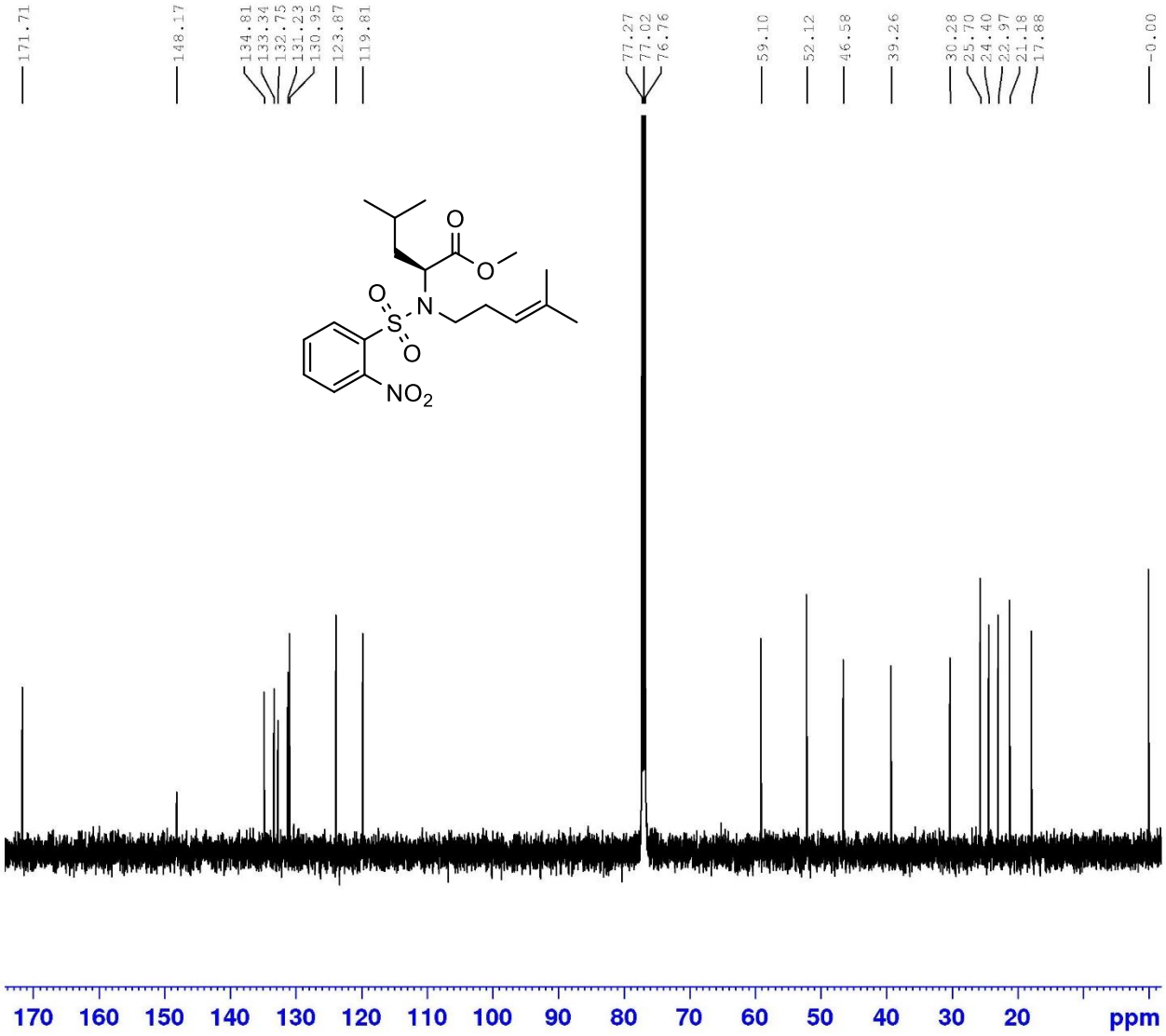
^{13}C-NMR **26c**



BRUKER

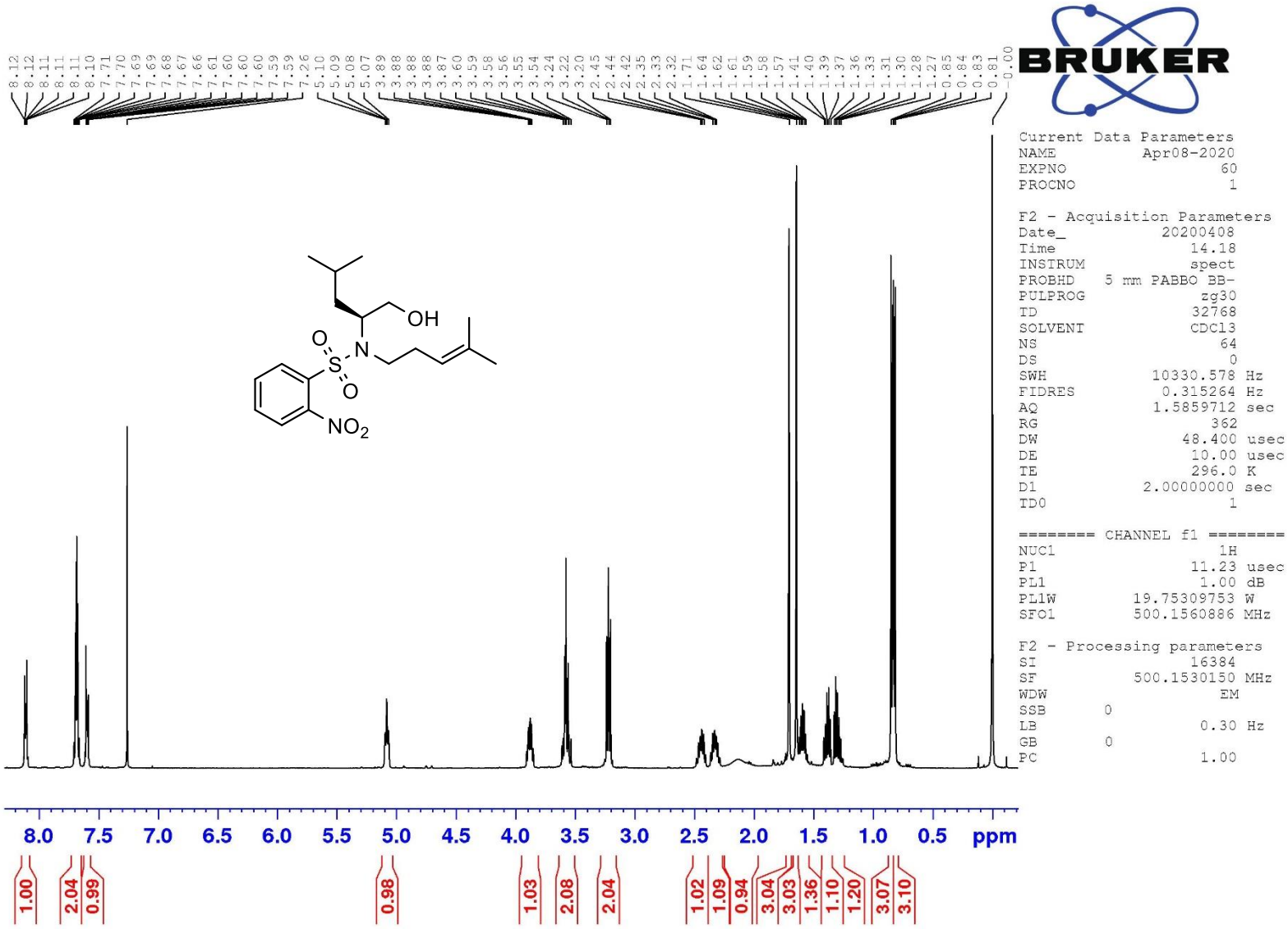
```
Current Data Parameters
NAME          Apr08-2020
EXPNO                 70
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200408
Time               14.25
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   362
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530151 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR **27c**



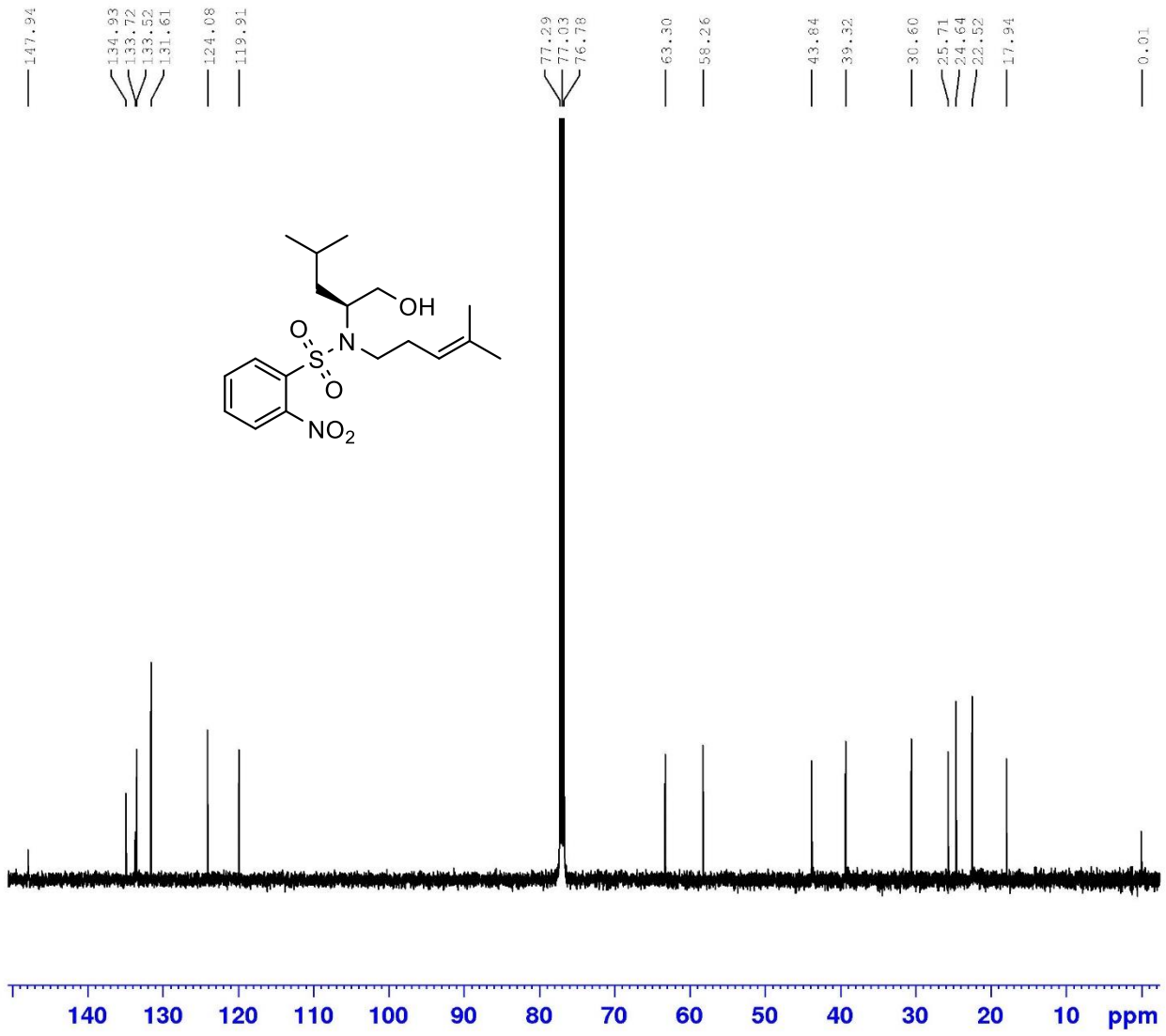

```
Current Data Parameters
NAME          Apr08-2020
EXPNO                 71
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200408
Time               15.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635740 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **27c**



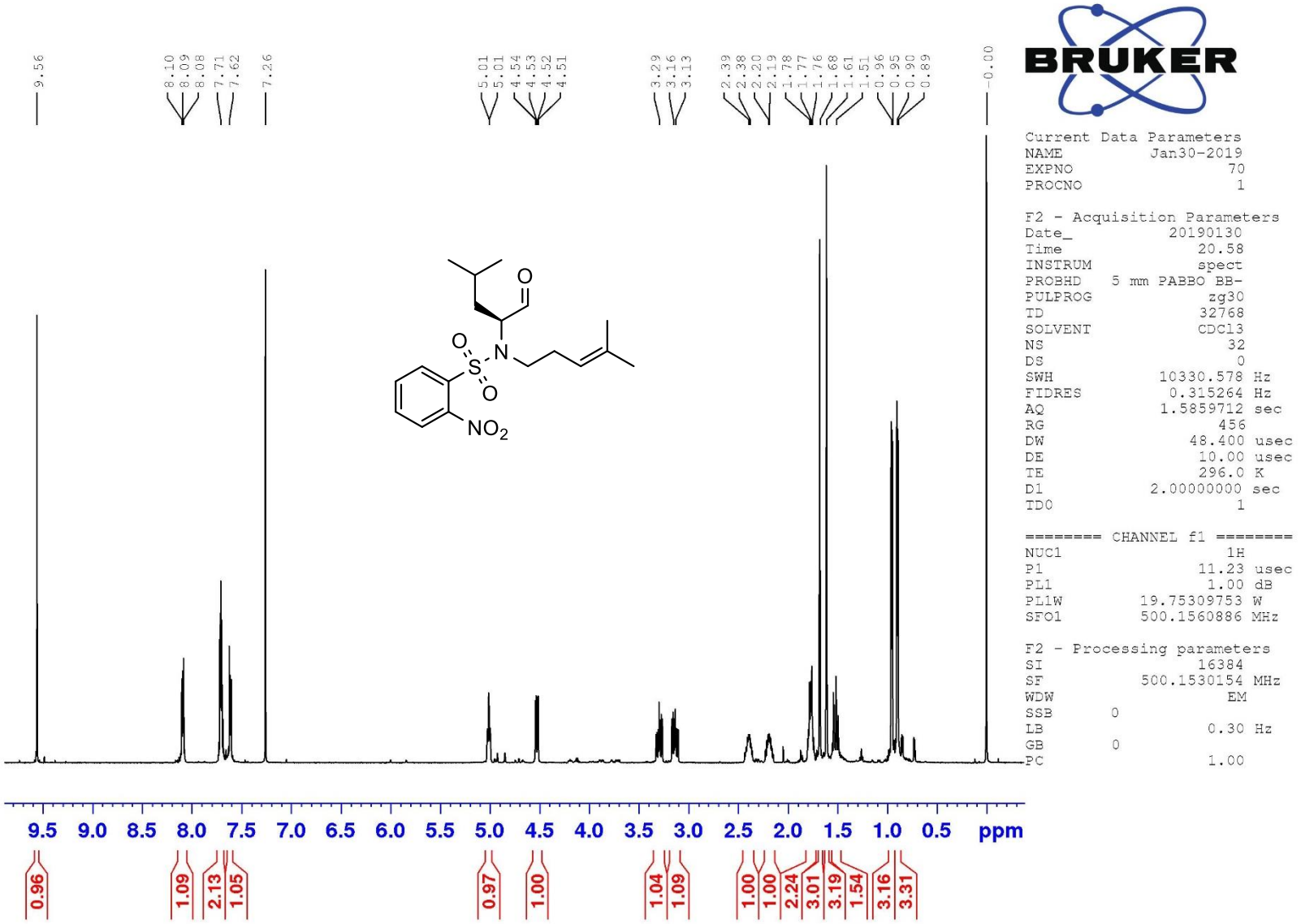

```
Current Data Parameters
NAME          Apr08-2020
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200408
Time               14.18
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   362
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530150 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR **28c**




```
Current Data Parameters
NAME          Jan28-2019
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190128
Time               19.04
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2890
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

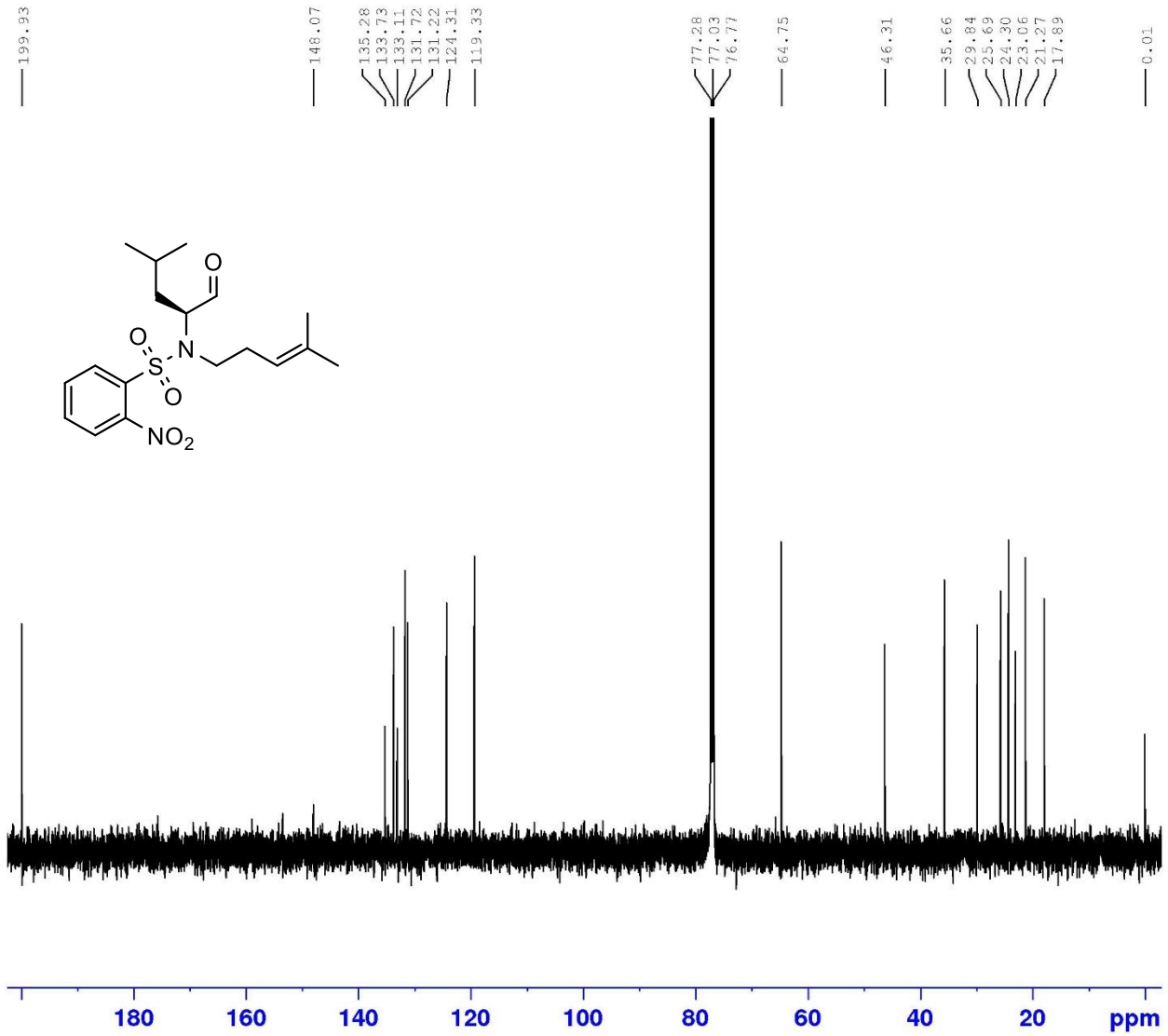
^{13}C-NMR **28c**

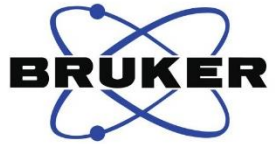


BRUKER

```
Current Data Parameters
NAME          Jan30-2019
EXPNO                 70
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190130
Time               20.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   456
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530154 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

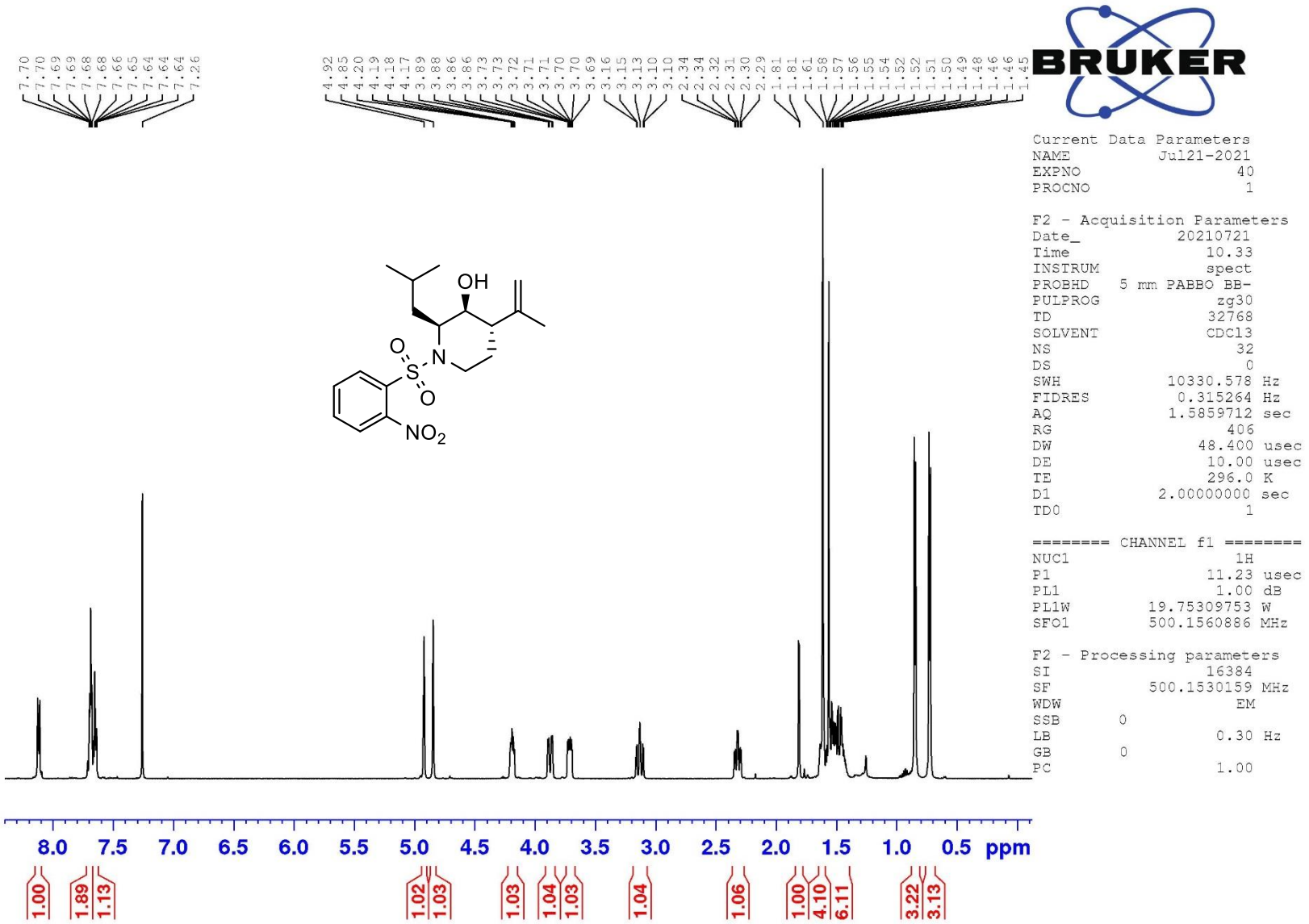
^{1}H-NMR **29c**





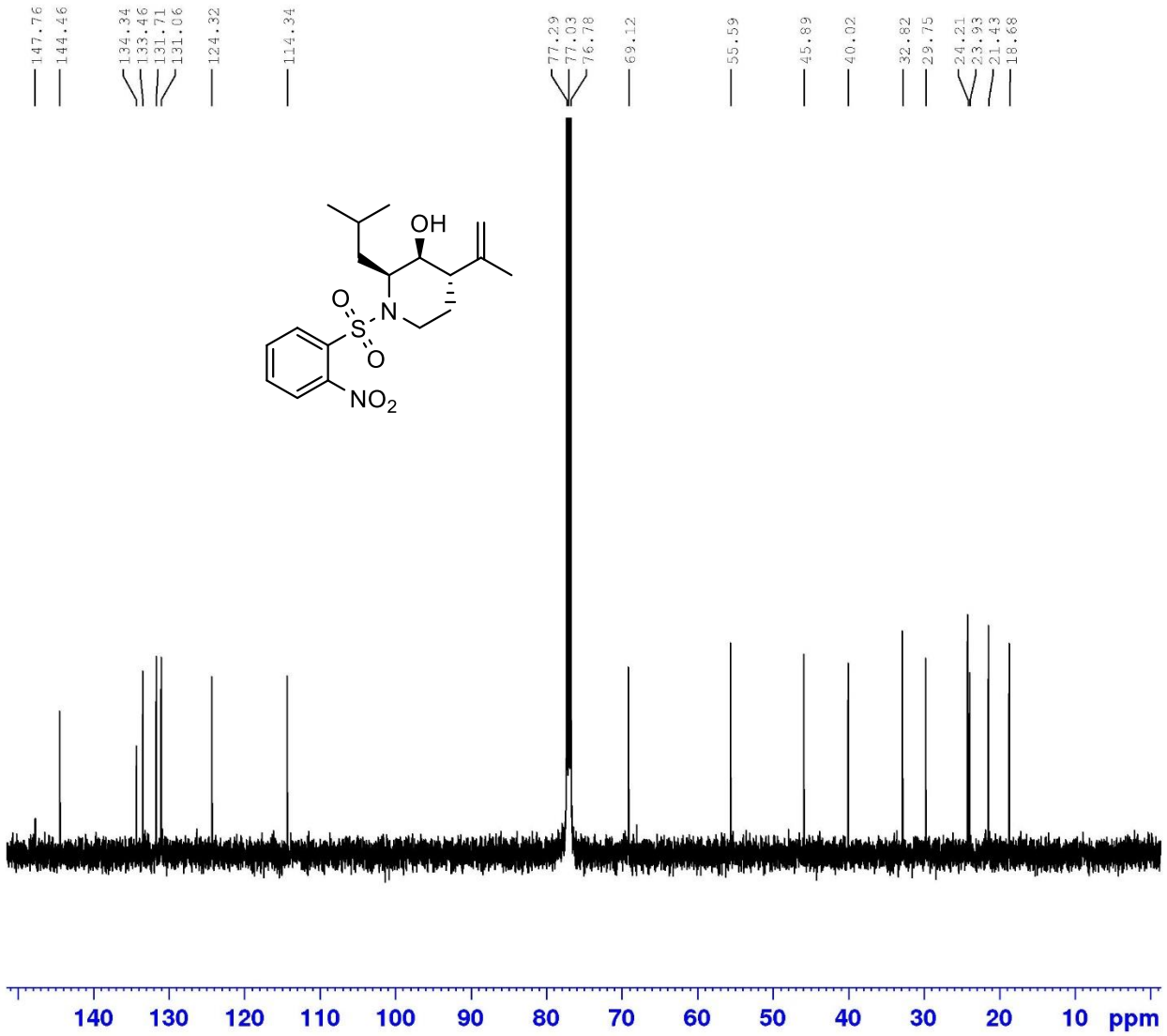
```
Current Data Parameters
NAME          Jan30-2019
EXPNO                 71
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190130
Time               21.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2890
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **29c**



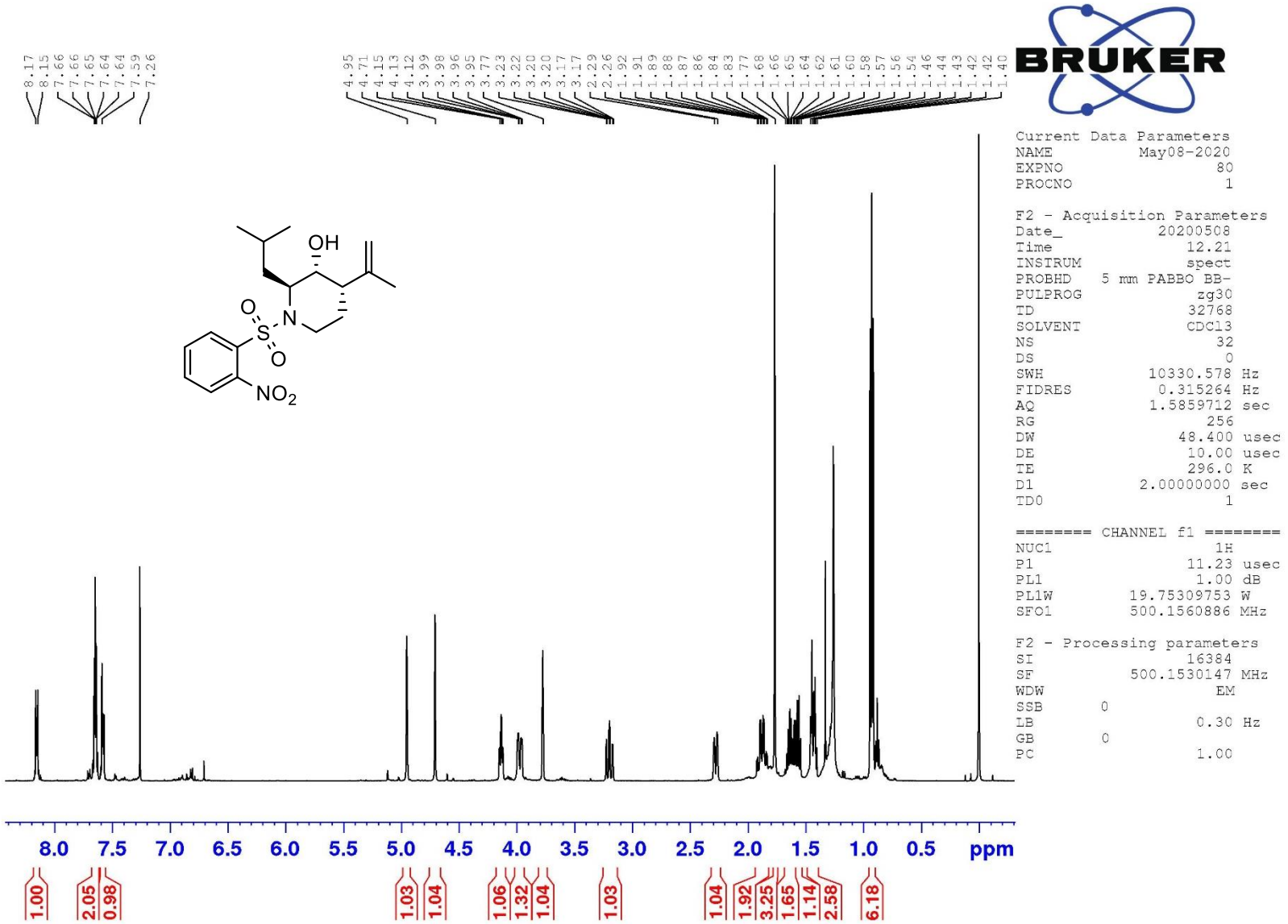

```
Current Data Parameters
NAME          Jul21-2021
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210721
Time               10.33
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   406
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530159 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*S*)-**30c**



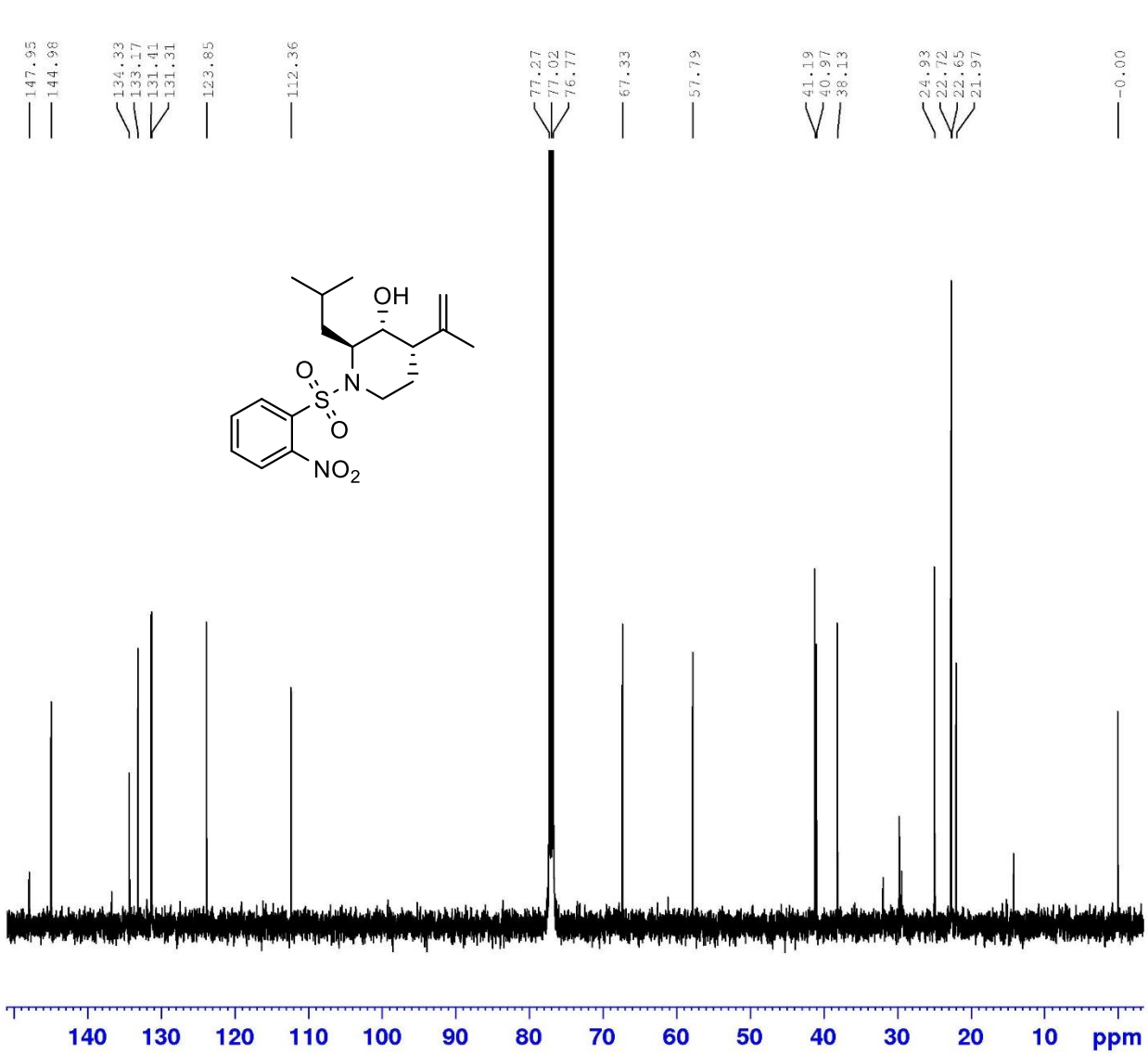

```
Current Data Parameters
NAME          Jul21-2021
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210721
Time               11.27
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                32768
SF          125.7635720 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

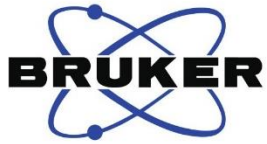
^{13}C-NMR (*S*,*S*)-**30c**




```
Current Data Parameters
NAME          May08-2020
EXPNO                 80
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200508
Time               12.21
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   256
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530147 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

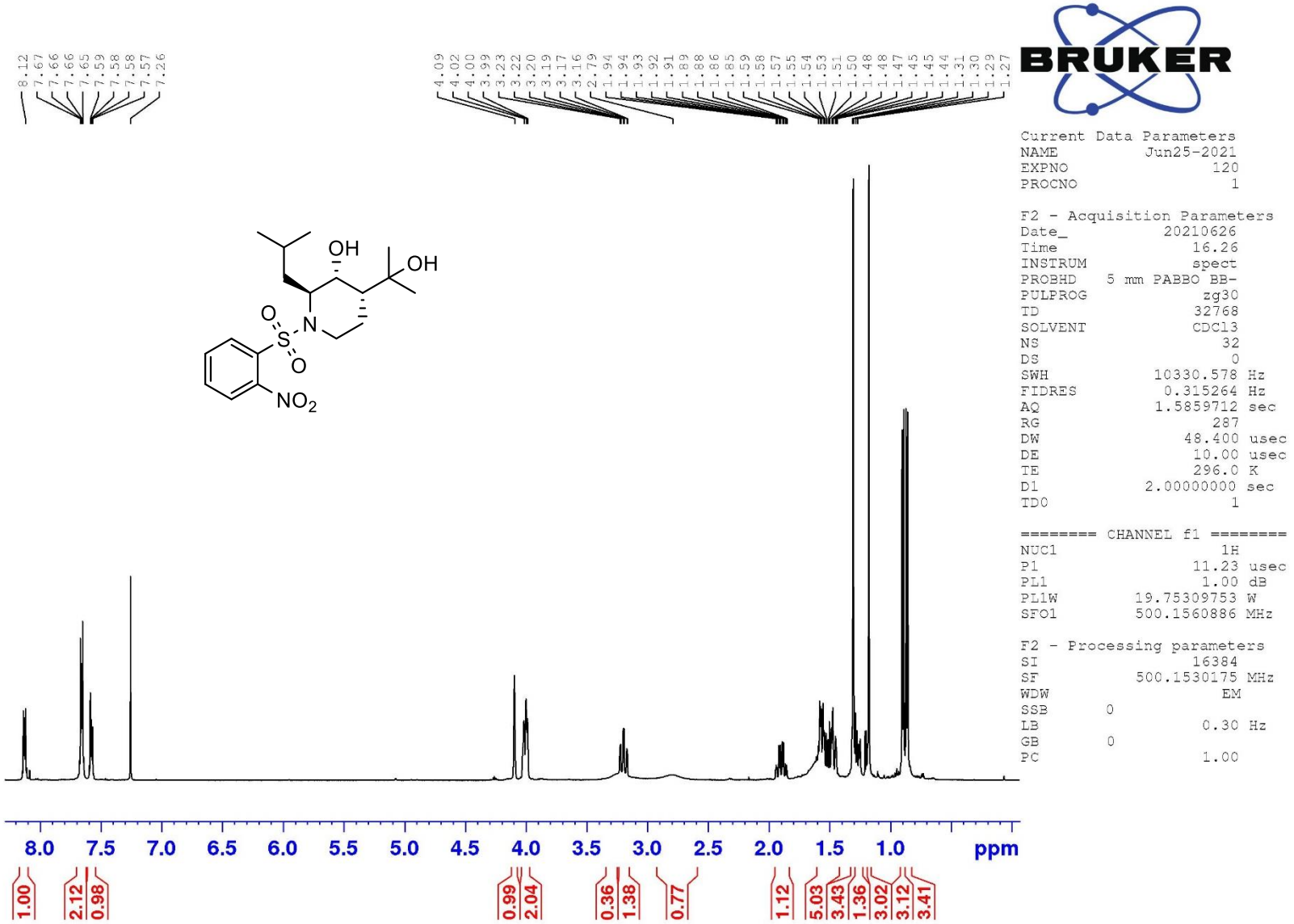
^{1}H-NMR (*R*,*S*)-**30c**





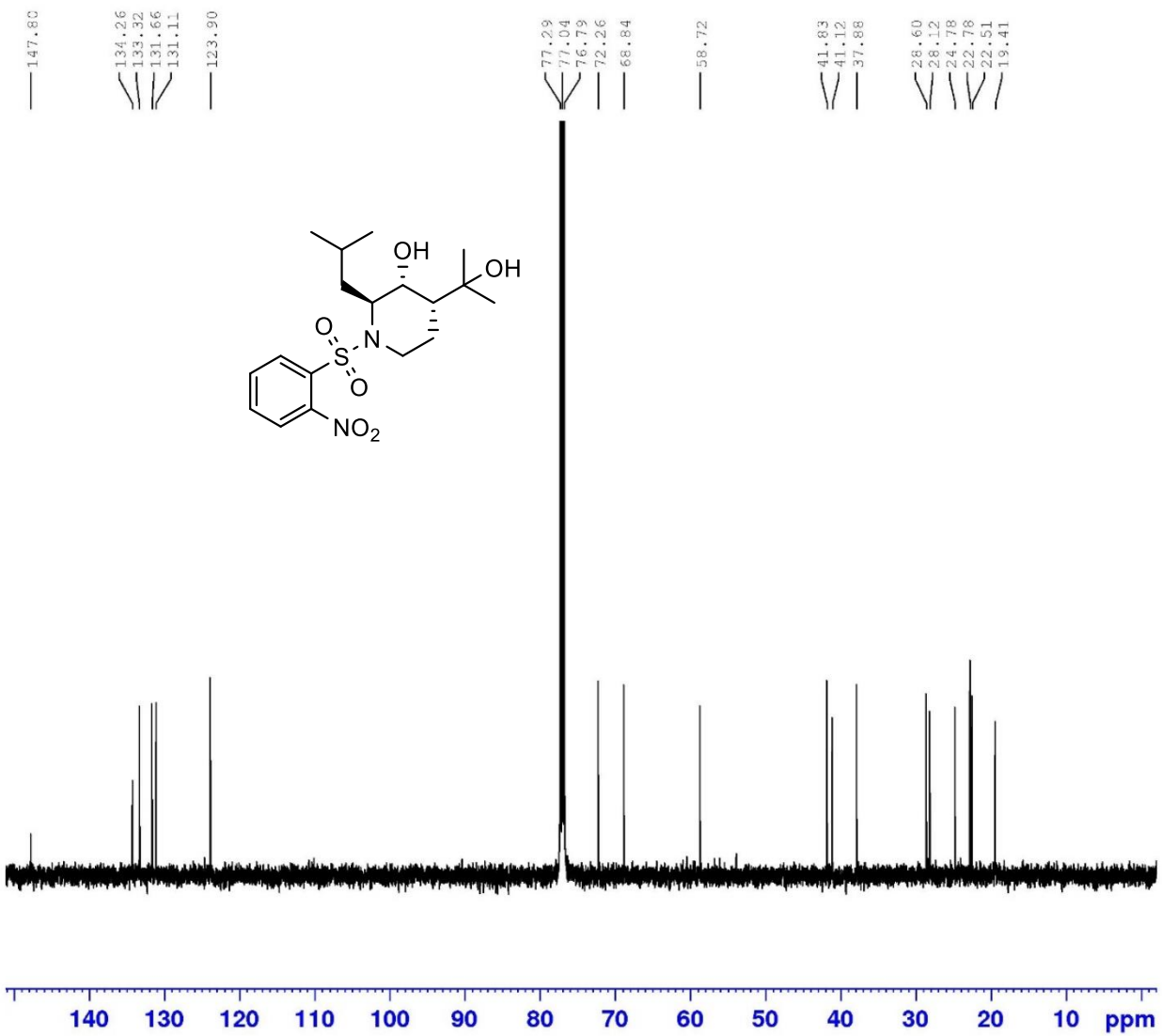

```
Current Data Parameters
NAME          May08-2020
EXPNO                 81
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200508
Time               13.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2300
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635742 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*R*,*S*)-**30c**




```
Current Data Parameters
NAME          Jun25-2021
EXPNO                120
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210626
Time               16.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   287
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530175 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*R*,*R*)-**31c**



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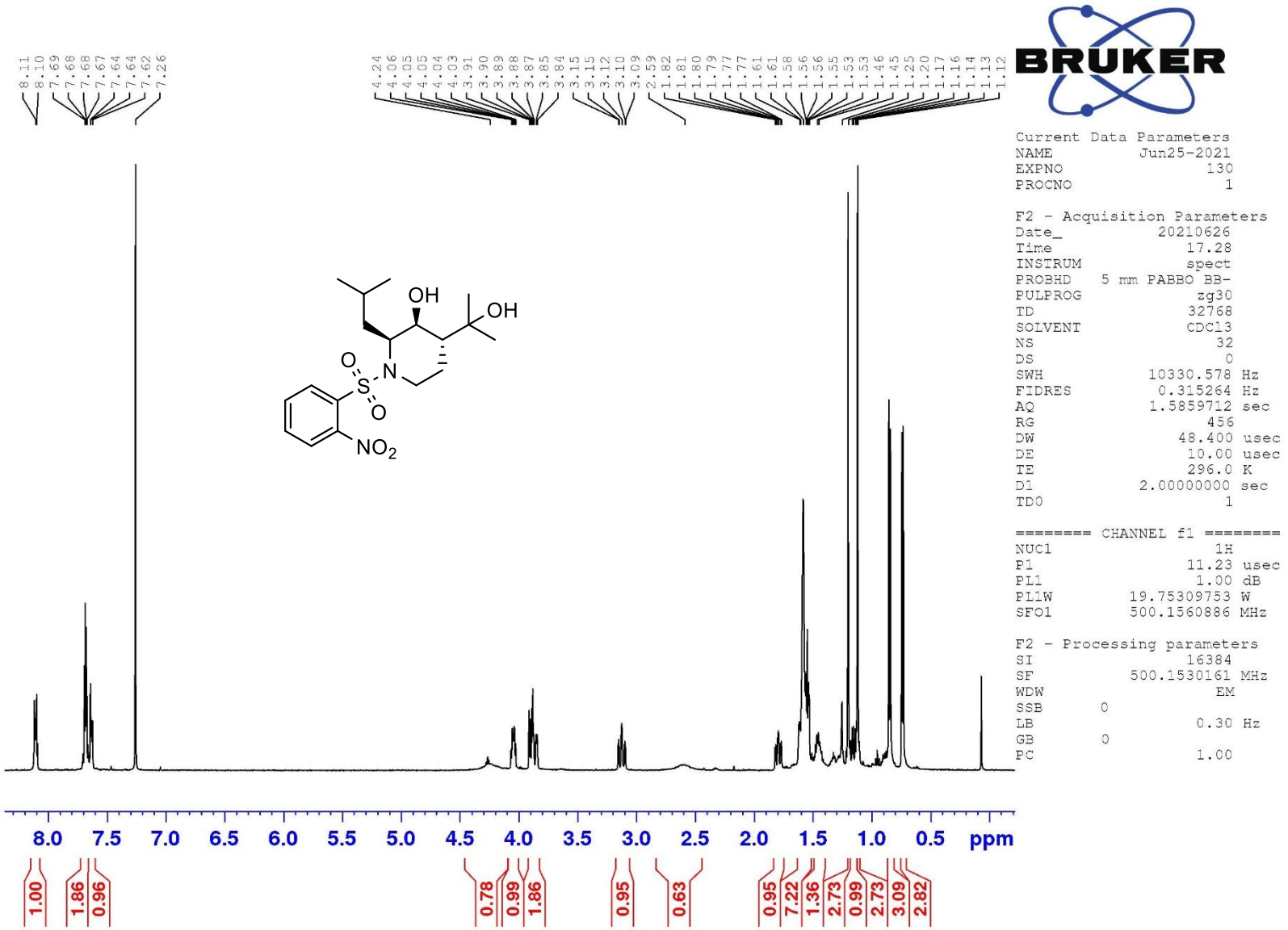
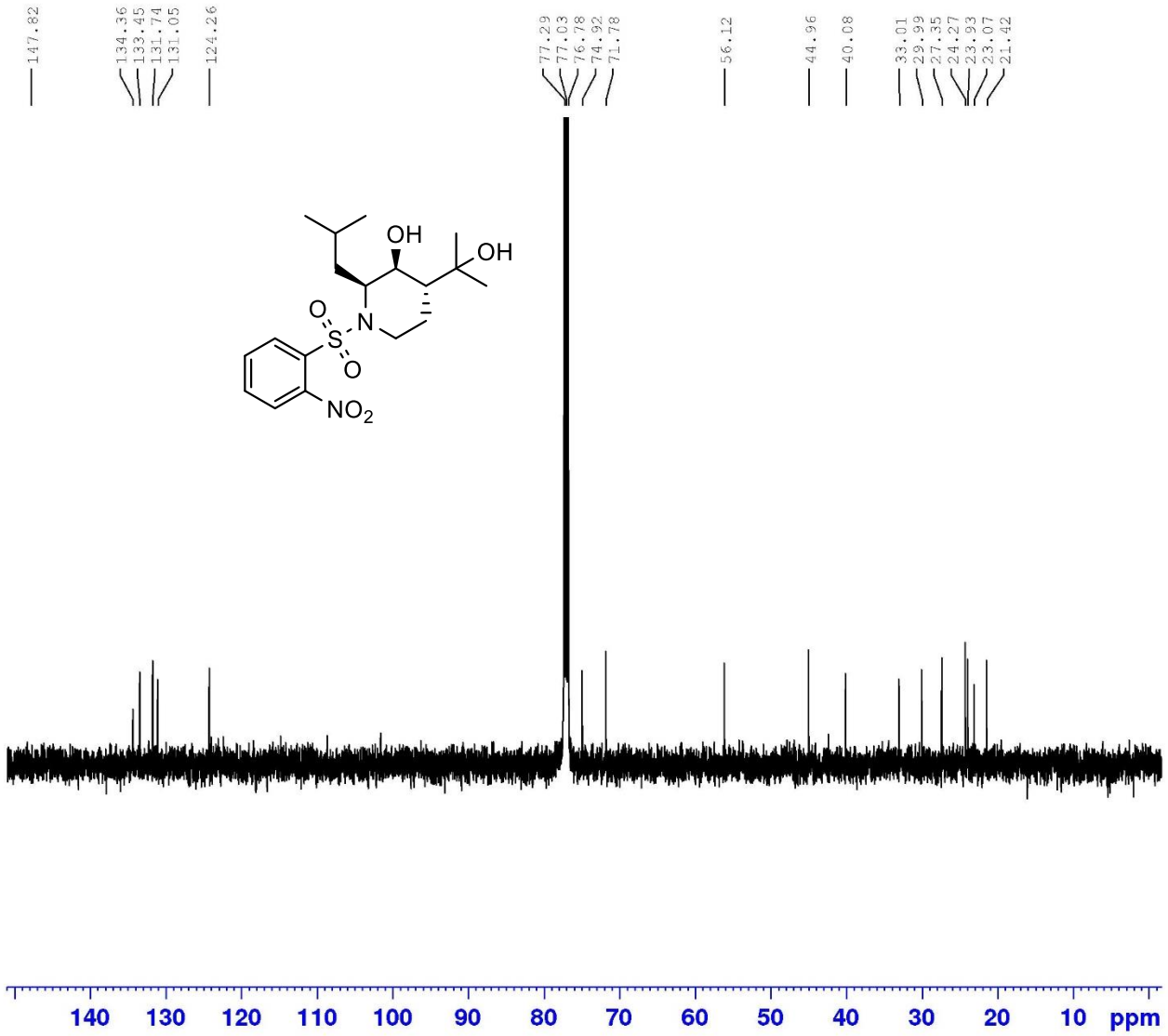
```
Current Data Parameters
NAME          Jun25-2021
EXPNO                121
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210626
Time               17.20
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

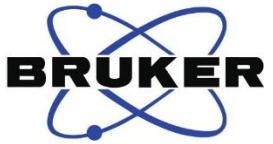
^{13}C-NMR (*R*,*R*)-**31c**




```
Current Data Parameters
NAME          Jun25-2021
EXPNO                130
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210626
Time               17.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   456
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530161 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

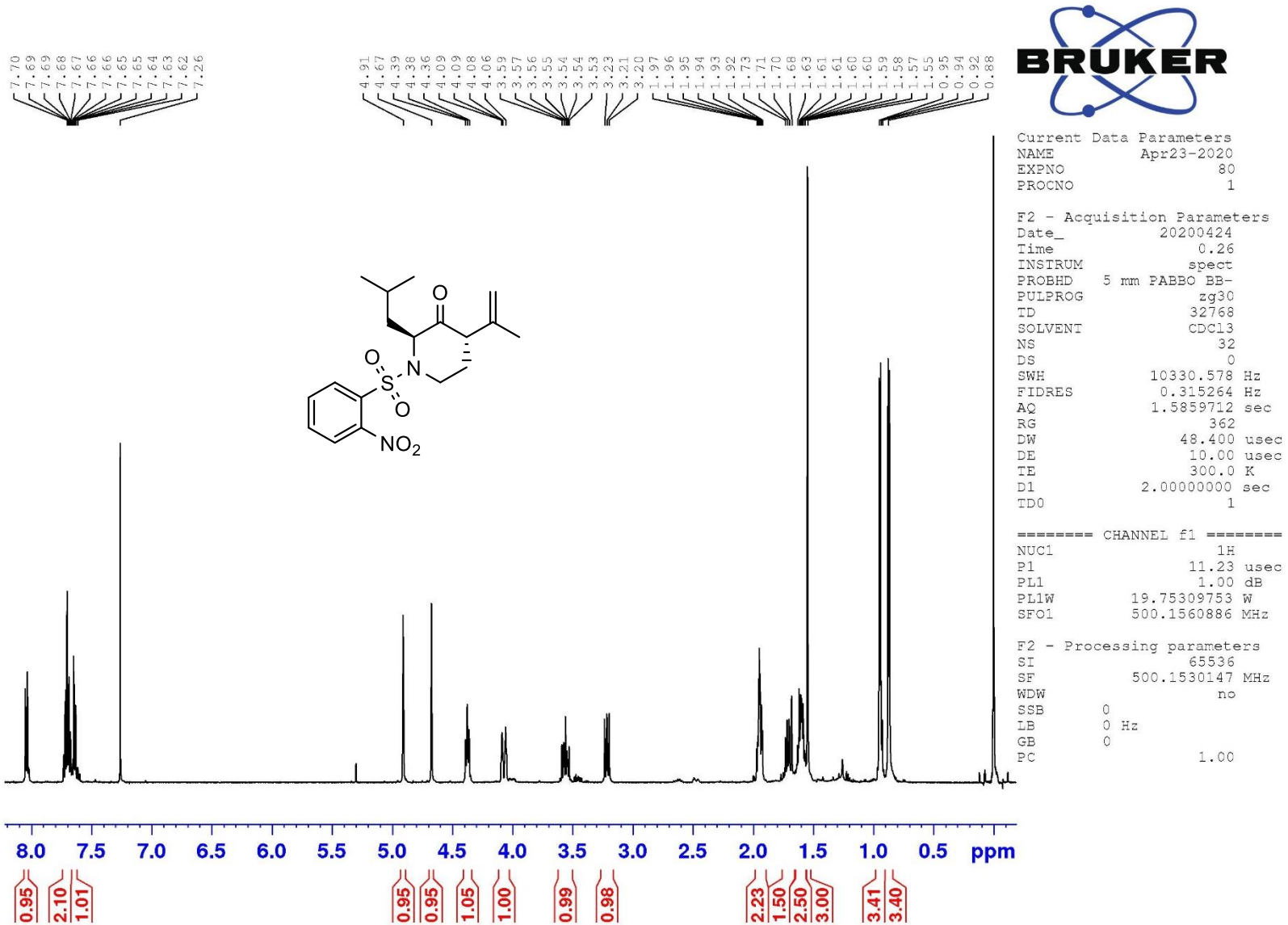
^{1}H-NMR (*S*,*R*)-**31c**






```
Current Data Parameters
NAME          Jun25-2021
EXPNO                131
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210626
Time               18.21
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*S*,*R*)-**31c**



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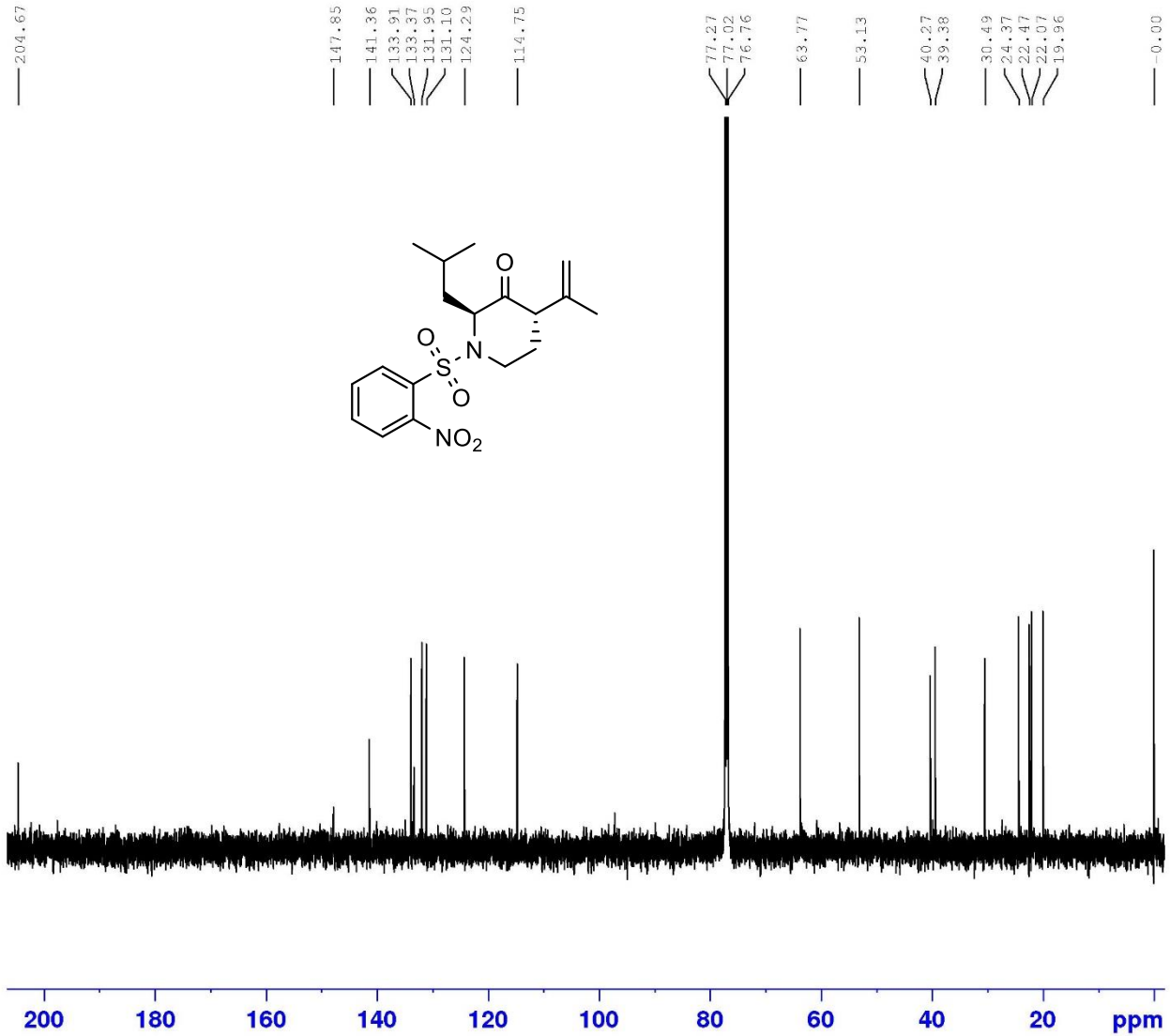
```
Current Data Parameters
NAME          Apr23-2020
EXPNO                 80
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200424
Time                0.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   362
DW                48.400 usec
DE                10.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 65536
SF          500.1530147 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*)-**S2c**



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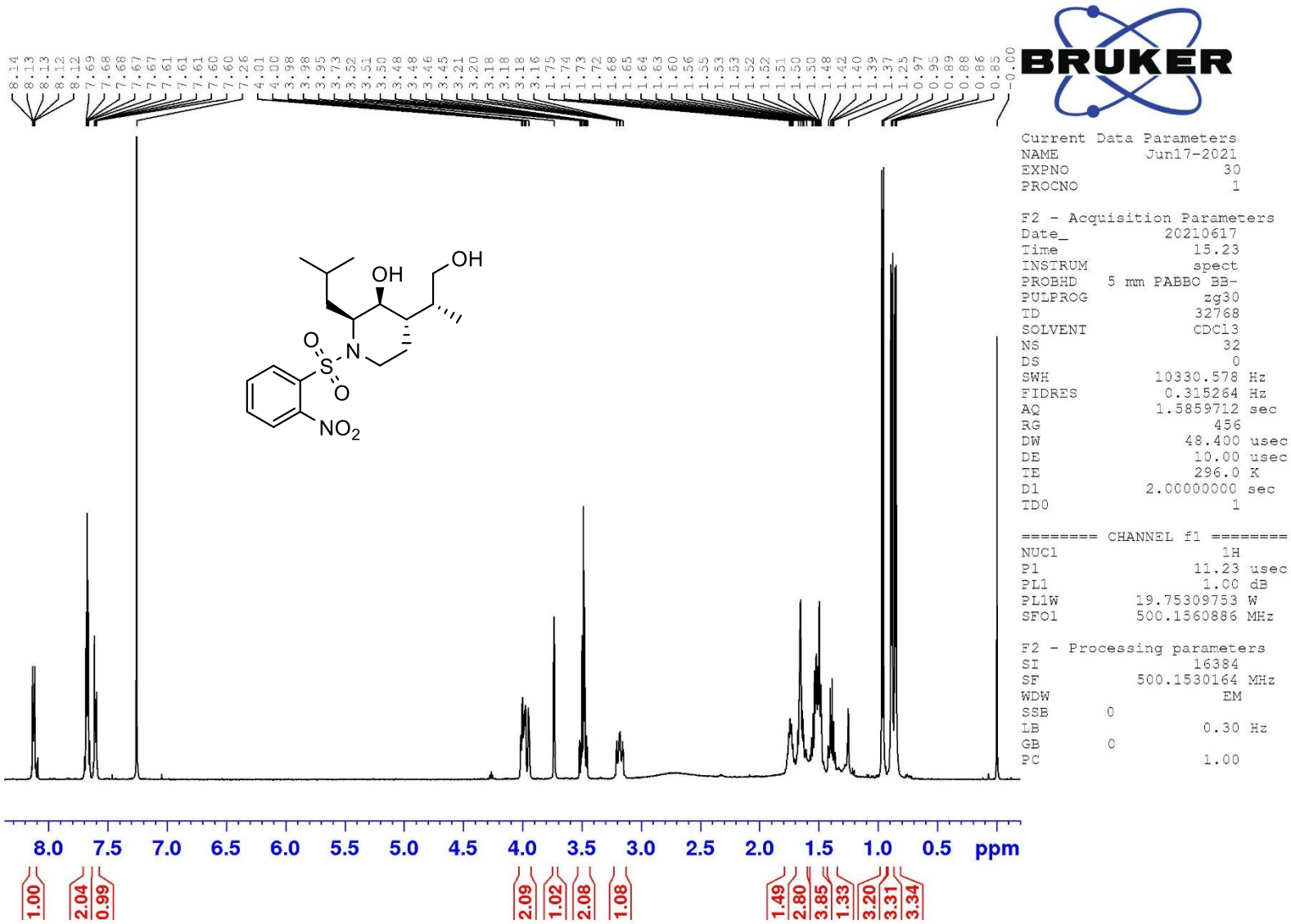
```
Current Data Parameters
NAME                 Apr23-2020
EXPNO                        81
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200424
Time                       1.20
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                32894.738 Hz
FIDRES              0.501934 Hz
AQ                  0.9961472 sec
RG                        2580
DW                      15.200 usec
DE                      10.00 usec
TE                      296.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                      10.20 usec
PL1                      1.50 dB
PL1W              51.74793243 W
SFO1             125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2               waltz16
NUC2                       1H
PCPD2                  100.00 usec
PL2                      1.00 dB
PL12                    19.99 dB
PL13                    21.00 dB
PL2W              19.75309753 W
PL12W              0.24925002 W
PL13W              0.19753097 W
SFO2             500.1550006 MHz

F2 - Processing parameters
SI                       32768
SF                125.7635738 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```

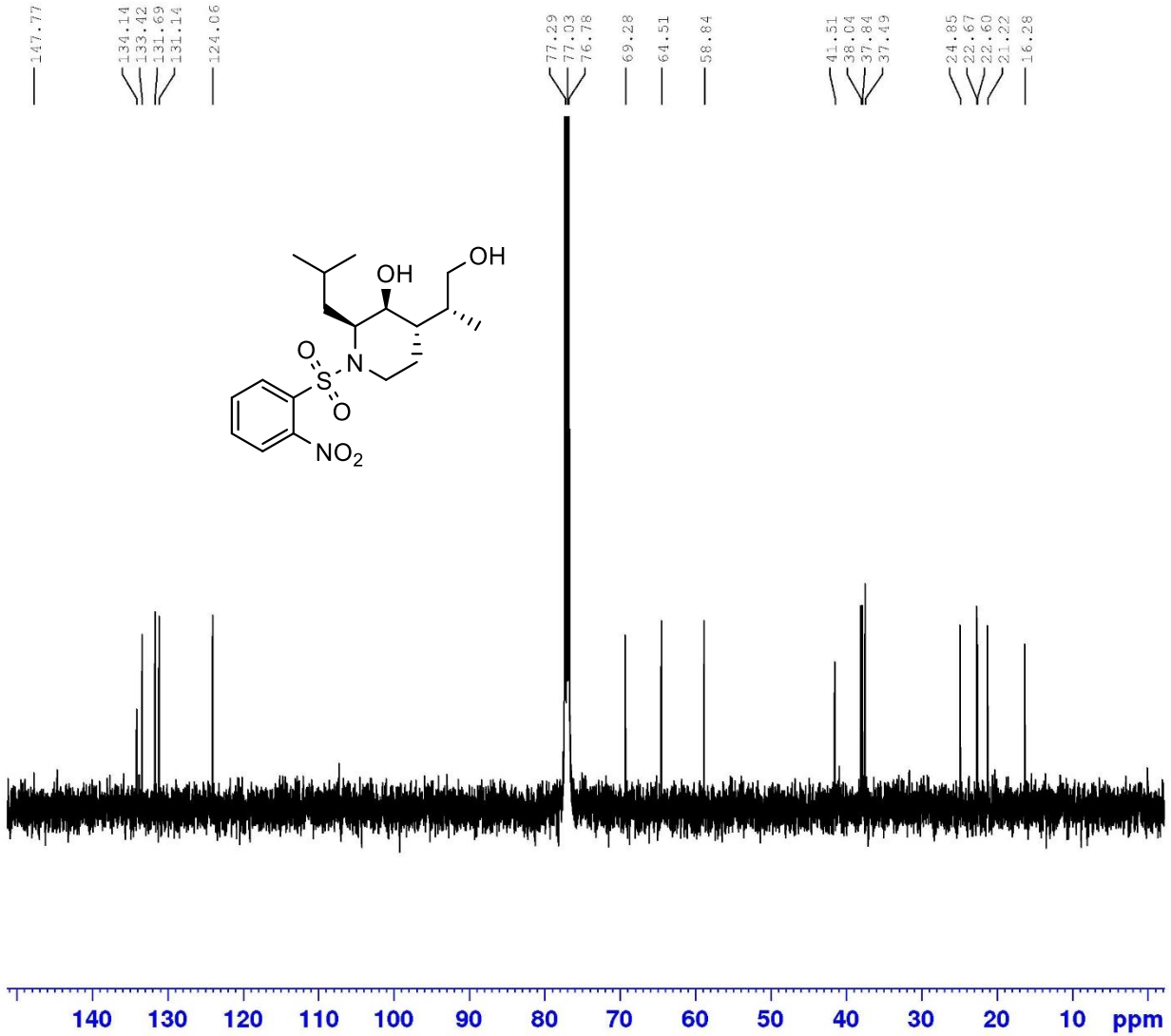
^{13}C-NMR (*S*)-**S2c**



BRUKER

```
Current Data Parameters
NAME          Jun17-2021
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210617
Time               15.23
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   456
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530164 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*S*,*R*)-**34c**



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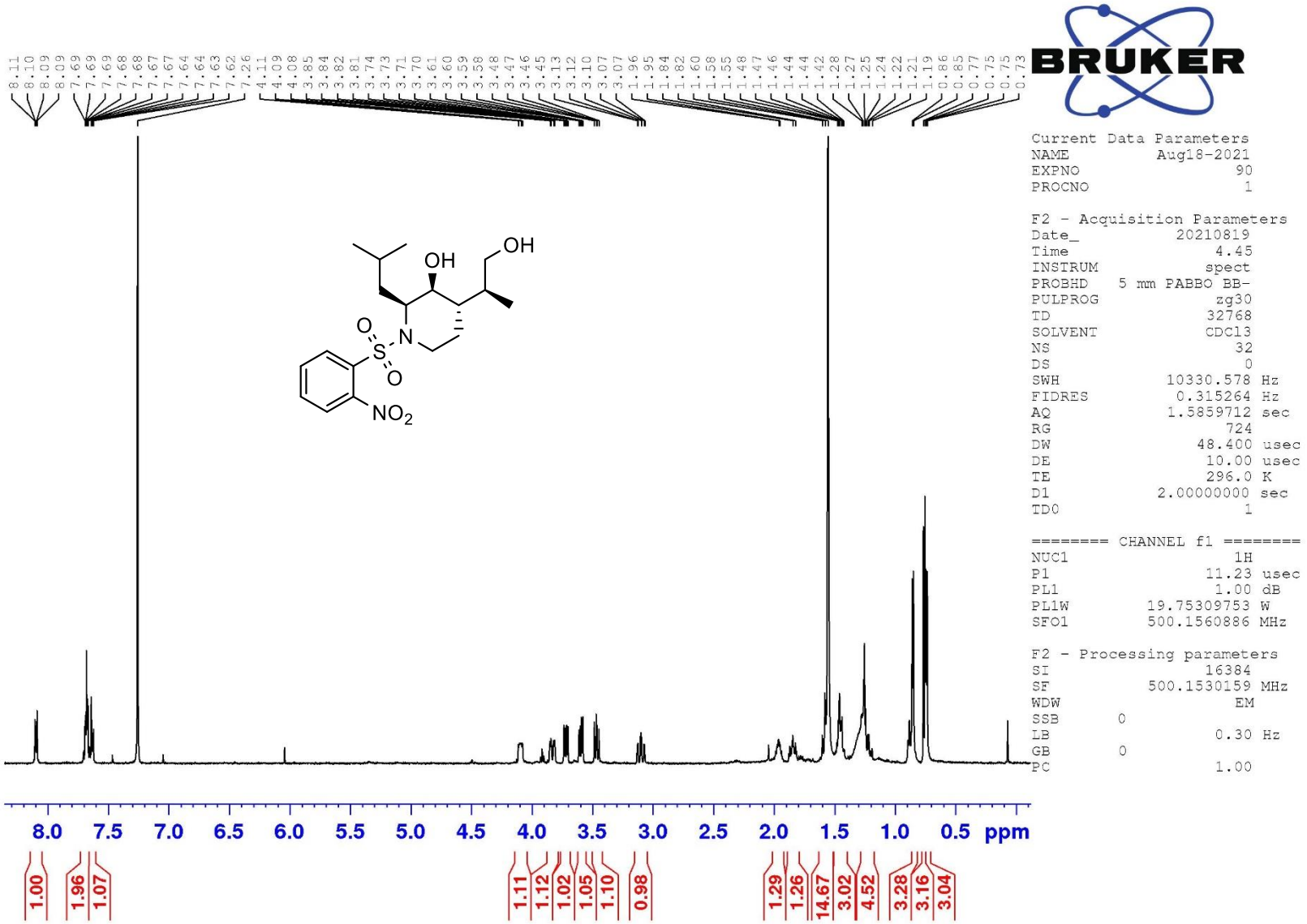
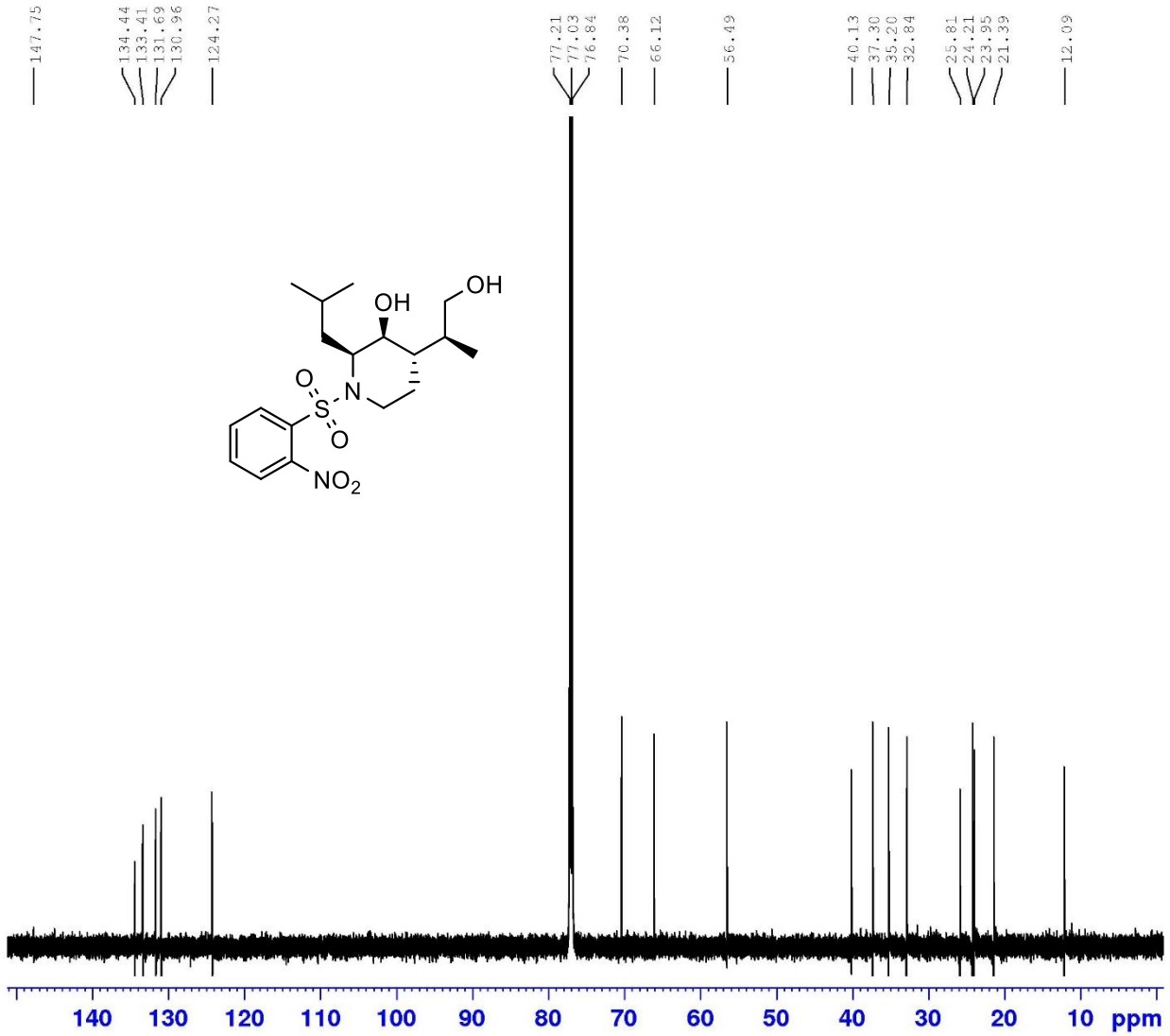
```
Current Data Parameters
NAME          Jun17-2021
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210617
Time               16.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2300
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*S*,*S*,*R*)-**34c**




```
Current Data Parameters
NAME          Aug18-2021
EXPNO                 90
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210819
Time                4.45
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   724
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530159 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*S*,*S*)-**34c**



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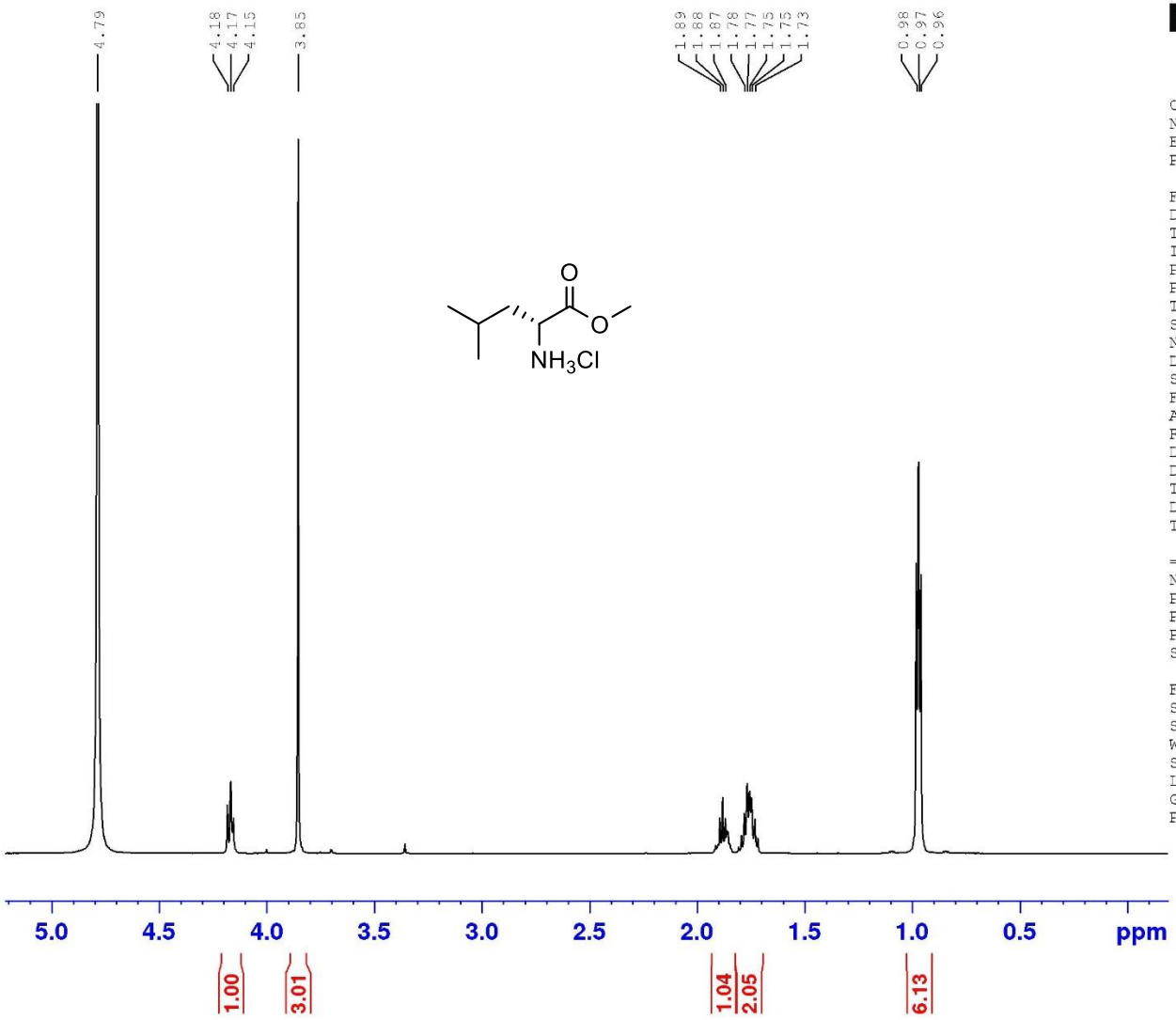
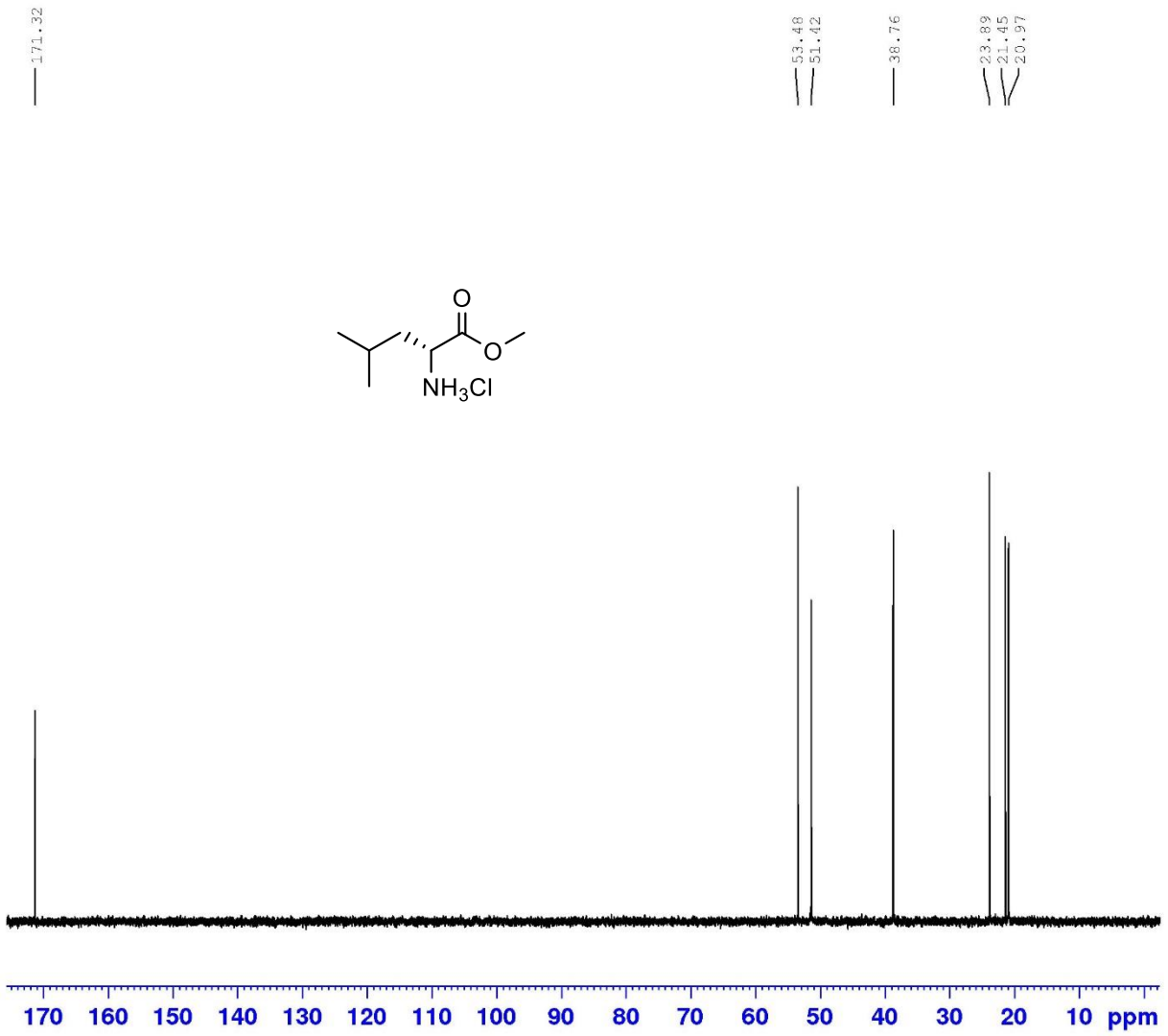
```
Current Data Parameters
NAME          Jun23-2021
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210623
Time               11.54
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2           9.89999962 W
PLW12          0.15564001 W
PLW13          0.07837200 W

F2 - Processing parameters
SI                131072
SF           176.1056620 MHz
WDW                 no
SSB       0
LB        0 Hz
GB        0
PC                 1.40
```

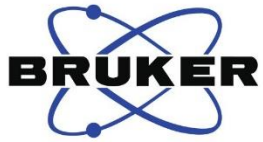
^{13}C-NMR (*S*,*S*,*S*)-**34c**




```
Current Data Parameters
NAME          Oct13-2020
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201013
Time               13.40
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT              D2O
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                  90.5
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1529587 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

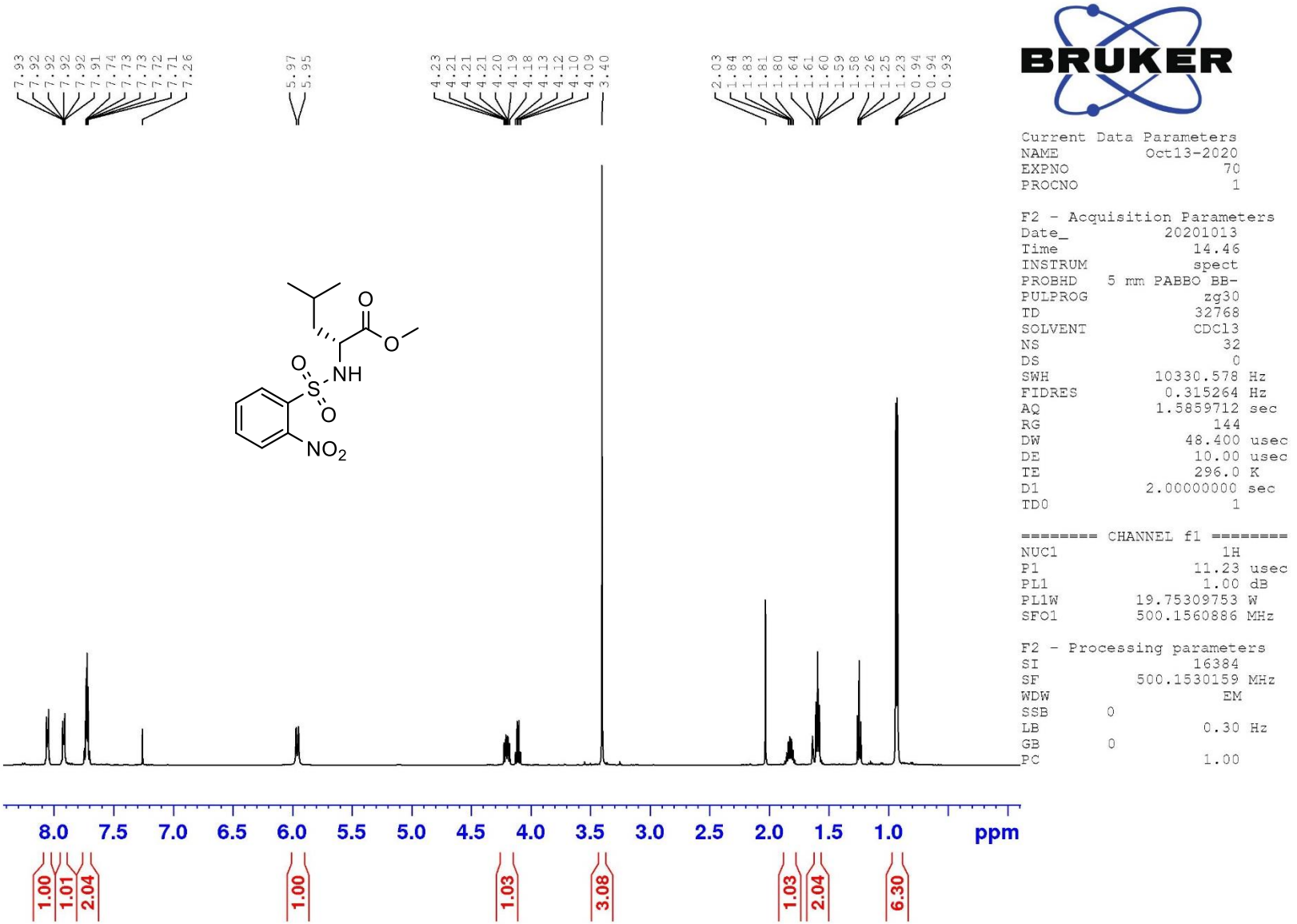
^{1}H-NMR **25d**






```
Current Data Parameters
NAME          Oct13-2020
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201013
Time               14.34
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             D2O
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **25d**



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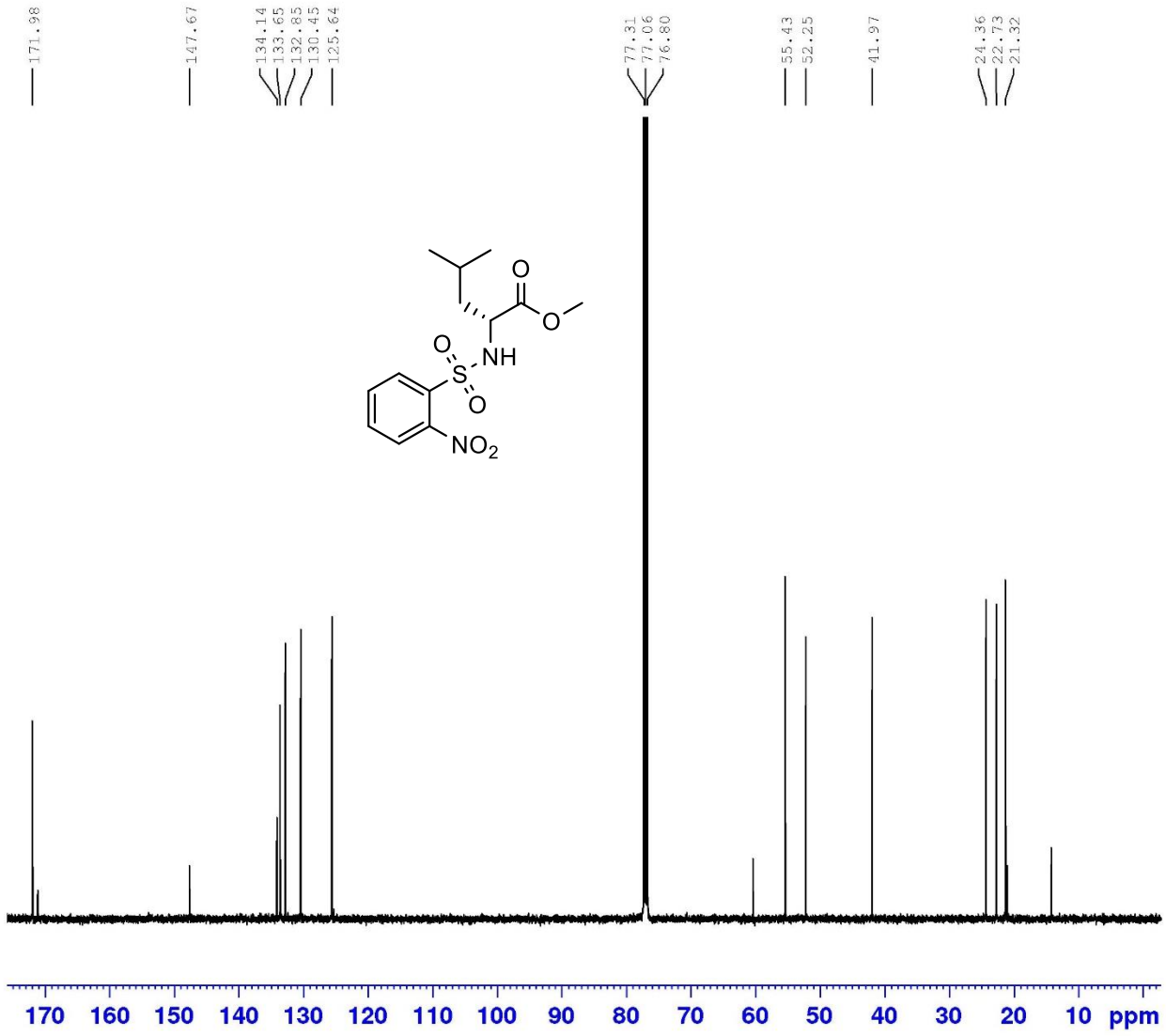
```
Current Data Parameters
NAME          Oct13-2020
EXPNO                 70
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201013
Time               14.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   144
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530159 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

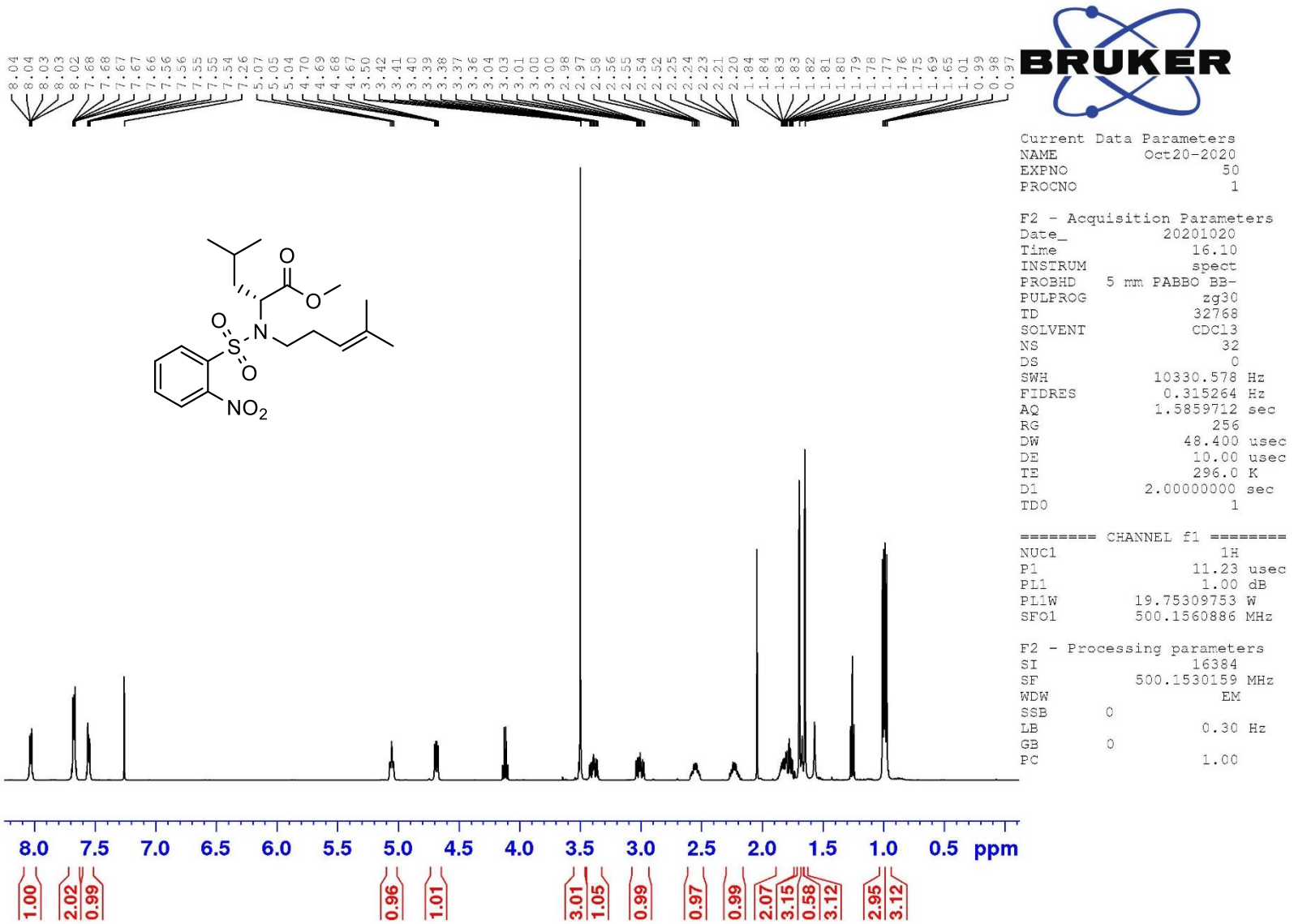
^{1}H-NMR **26d**



BRUKER

```
Current Data Parameters
NAME          Oct13-2020
EXPNO                 71
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201013
Time               15.40
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

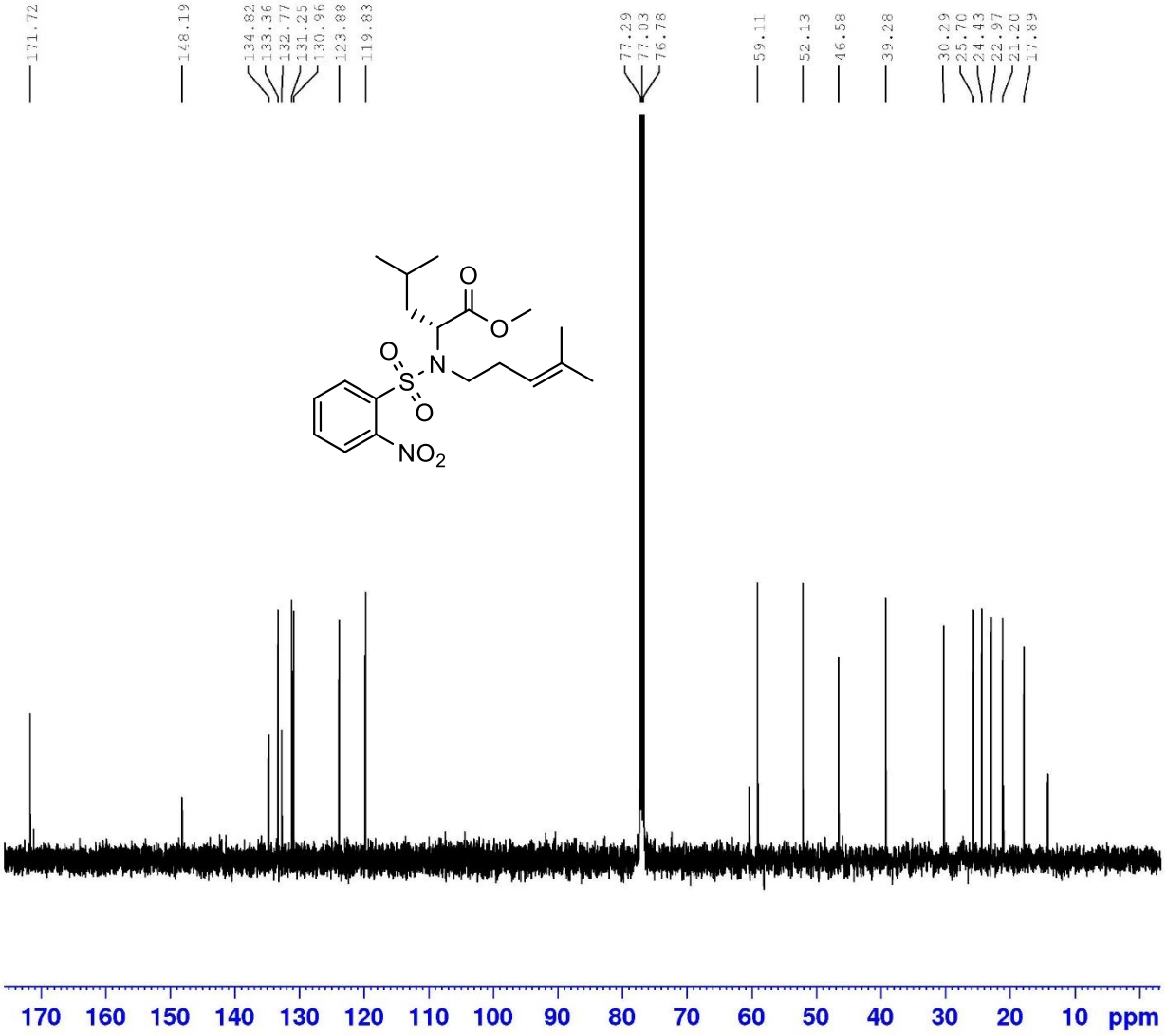
^{13}C-NMR **26d**



BRUKER

```
Current Data Parameters
NAME          Oct20-2020
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201020
Time               16.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   256
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530159 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

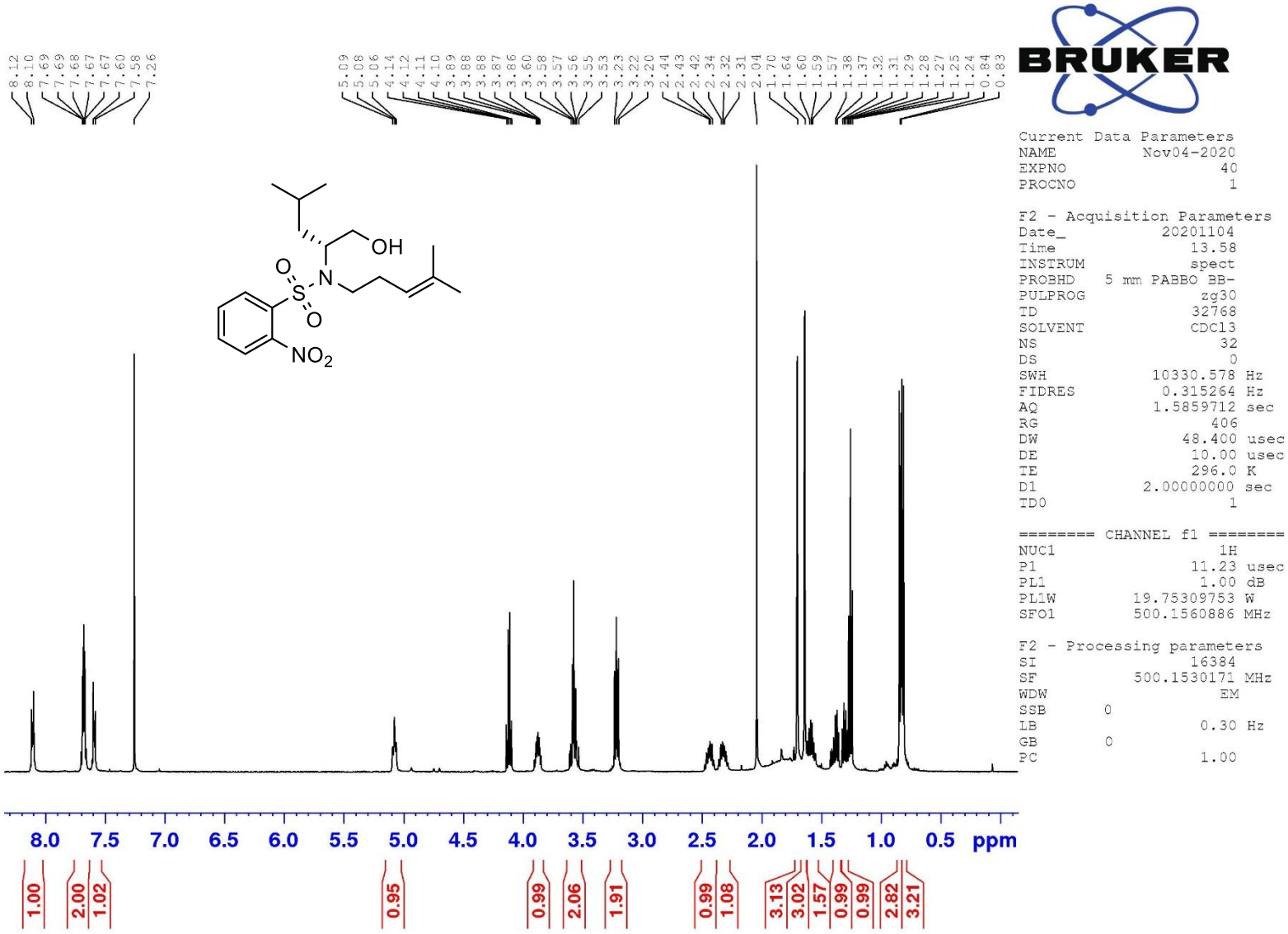
^{1}H-NMR **27d**



BRUKER

```
Current Data Parameters
NAME          Oct20-2020
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201020
Time               17.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES        0.501934 Hz
AQ            0.9961472 sec
RG                 2300
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.20 usec
PL1                  1.50 dB
PL1W        51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

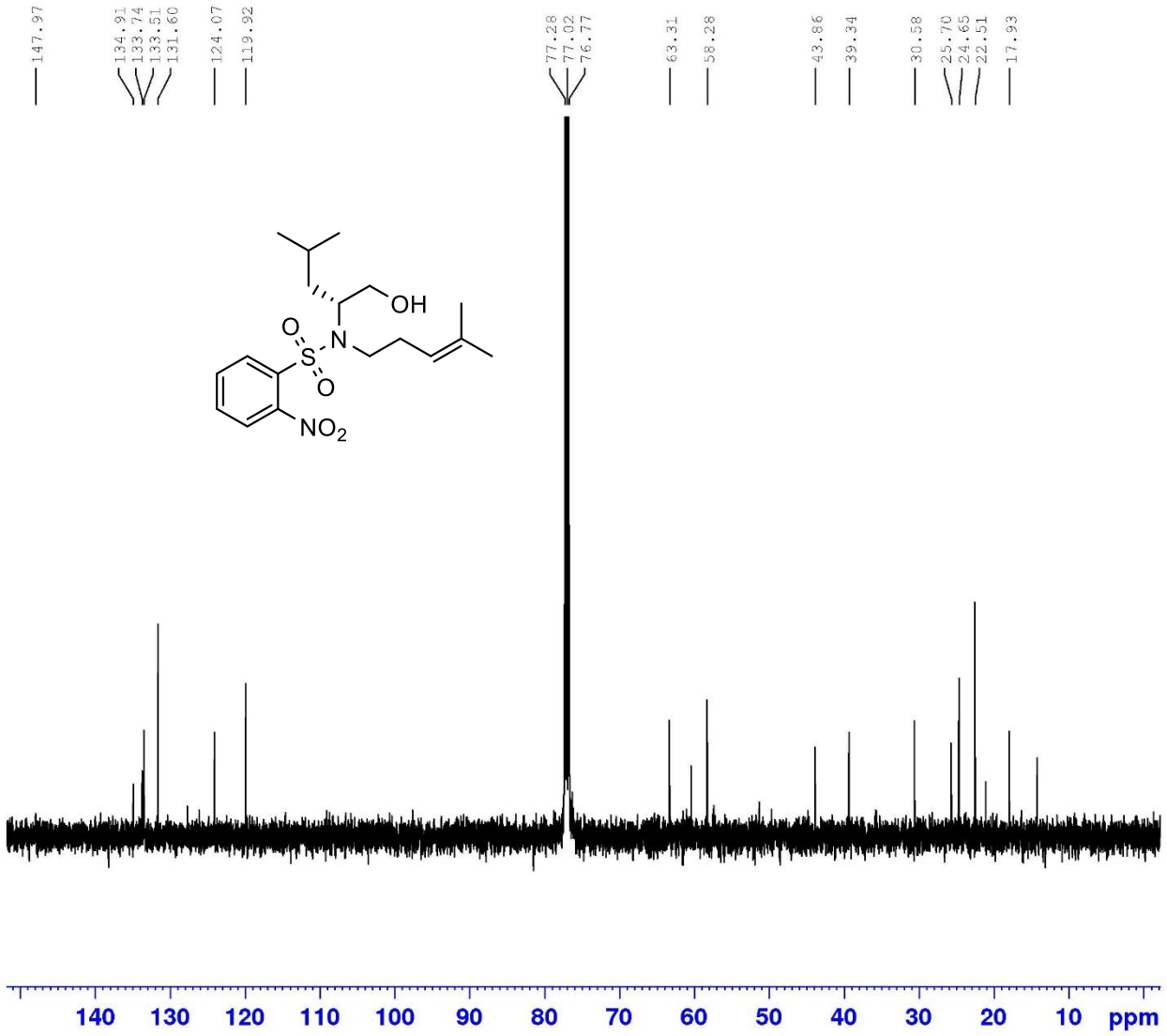
^{13}C-NMR **27d**



BRUKER

```
Current Data Parameters
NAME          Nov04-2020
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201104
Time               13.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   406
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530171 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

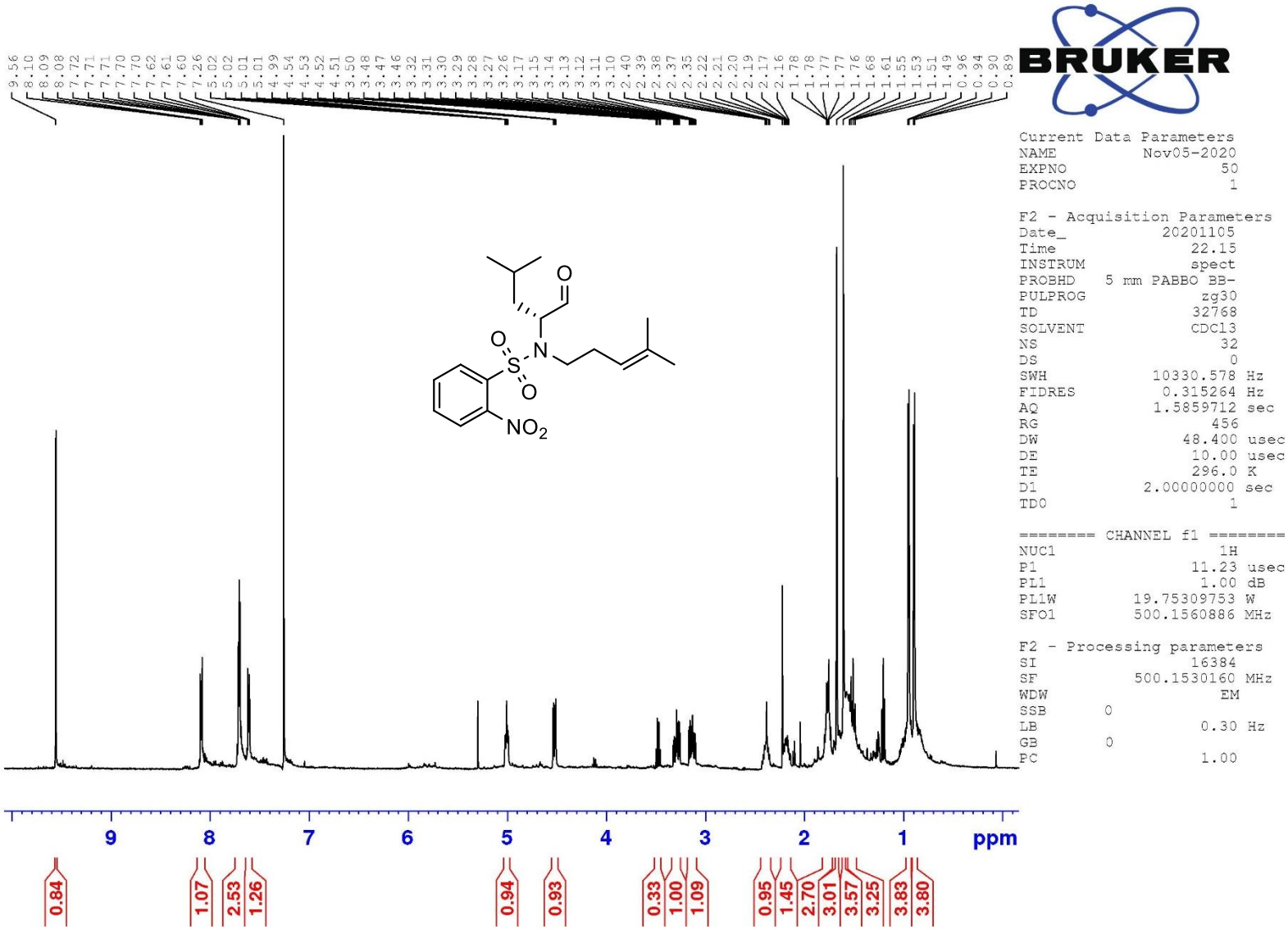
^{1}H-NMR **28d**



BRUKER

```
Current Data Parameters
NAME          Nov04-2020
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201104
Time               14.52
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW                15.200 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

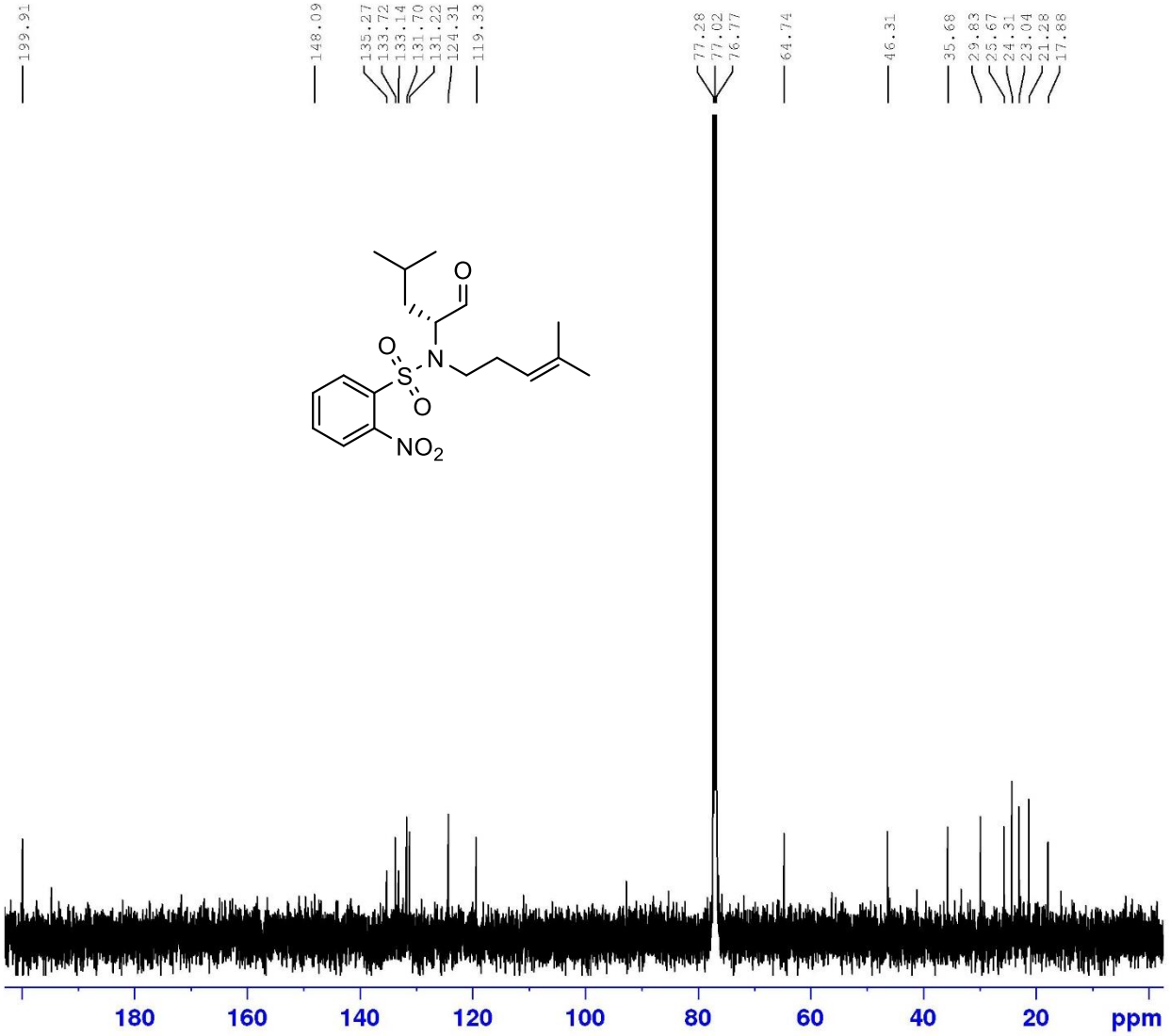
^{13}C-NMR **28d**



BRUKER

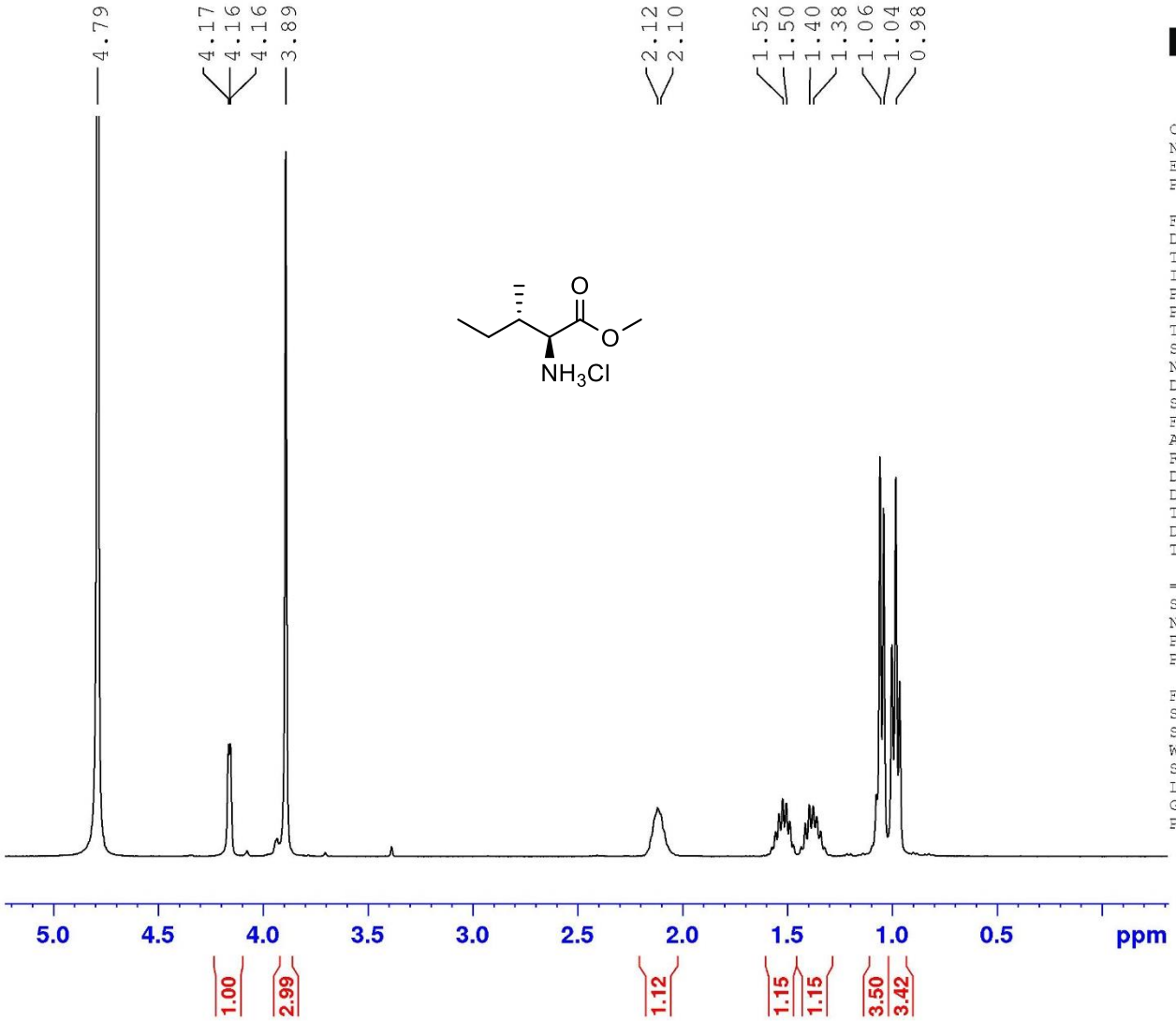
```
Current Data Parameters
NAME          Nov05-2020
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201105
Time               22.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   456
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530160 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR **29d**



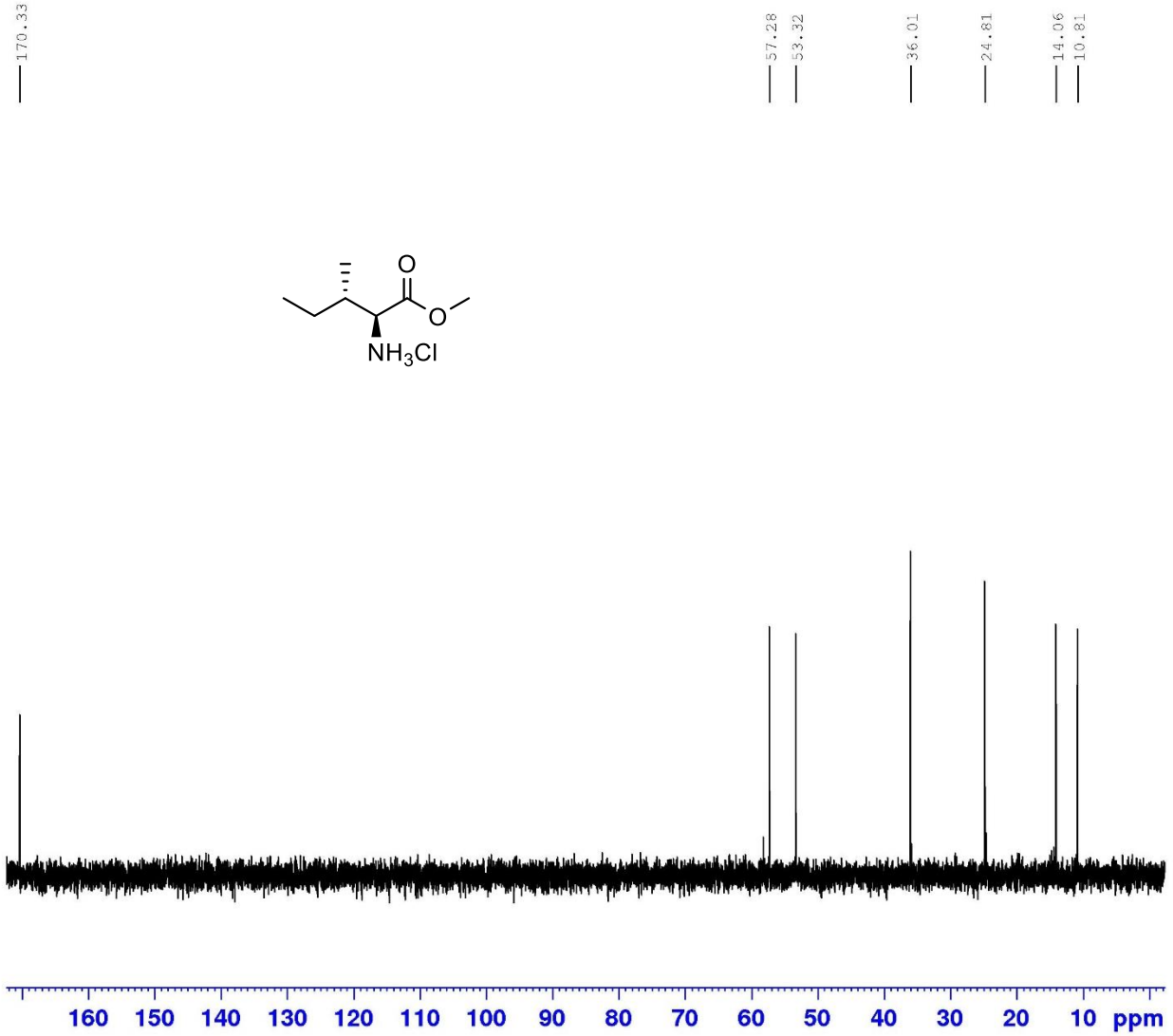

```
Current Data Parameters
NAME          Nov05-2020
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201105
Time               23.09
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                32768
SF         125.7635720 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **29d**




```
Current Data Parameters
NAME          Mar05-2020
EXPNO                370
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200306
Time                0.54
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT              D2O
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 160.83
DW                 62.400 usec
DE                  6.50 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1024708 MHz
NUC1                   1H
P1                  13.70 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.0999650 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

^{1}H-NMR **25e**



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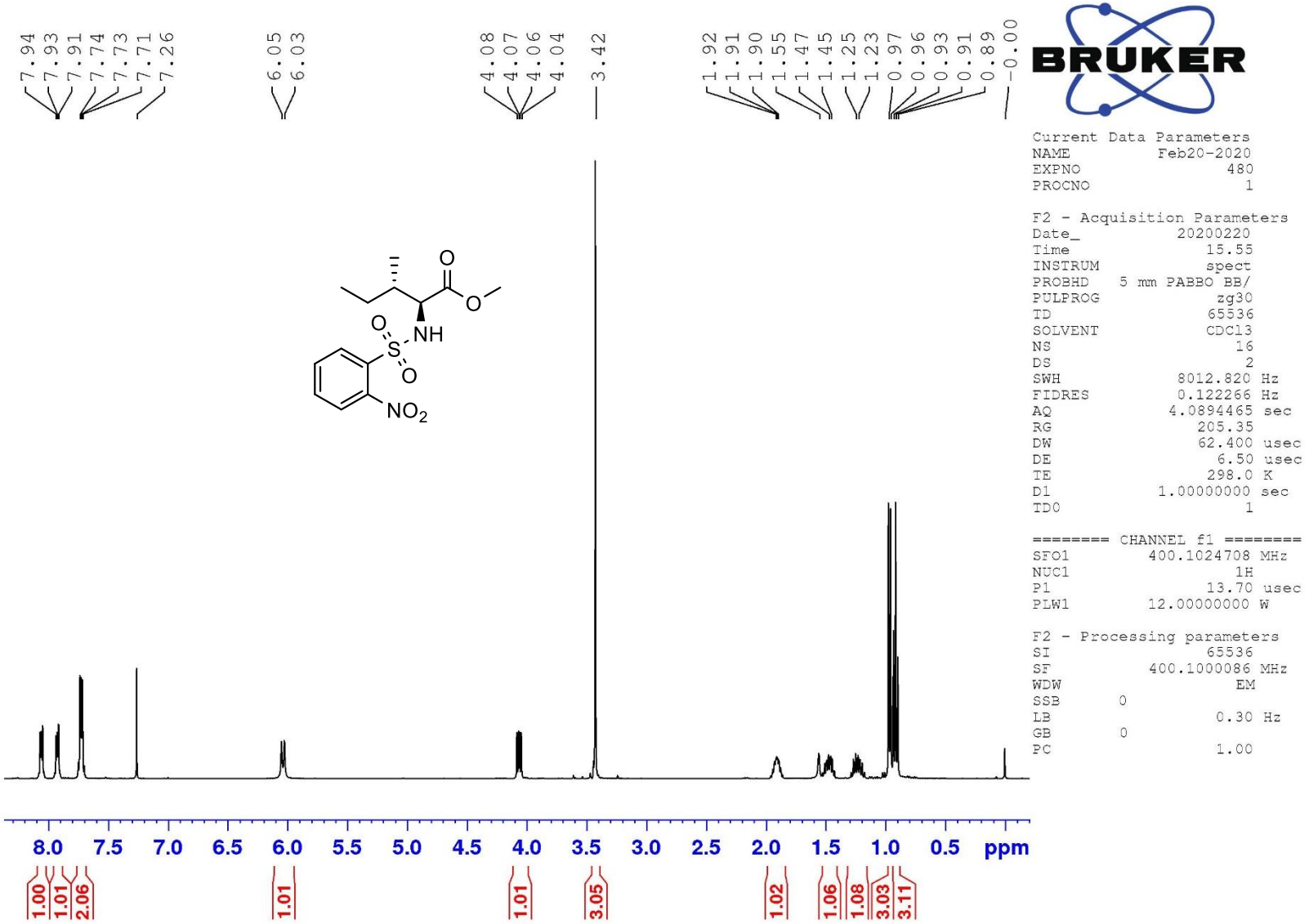
```
Current Data Parameters
NAME          Mar05-2020
EXPNO                371
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200306
Time                1.24
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT             D2O
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               205.35
DW               20.800 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6152851 MHz
NUC1                 13C
P1                10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1016004 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.27805999 W
PLW13         0.22522999 W

F2 - Processing parameters
SI                 32768
SF           100.6052250 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

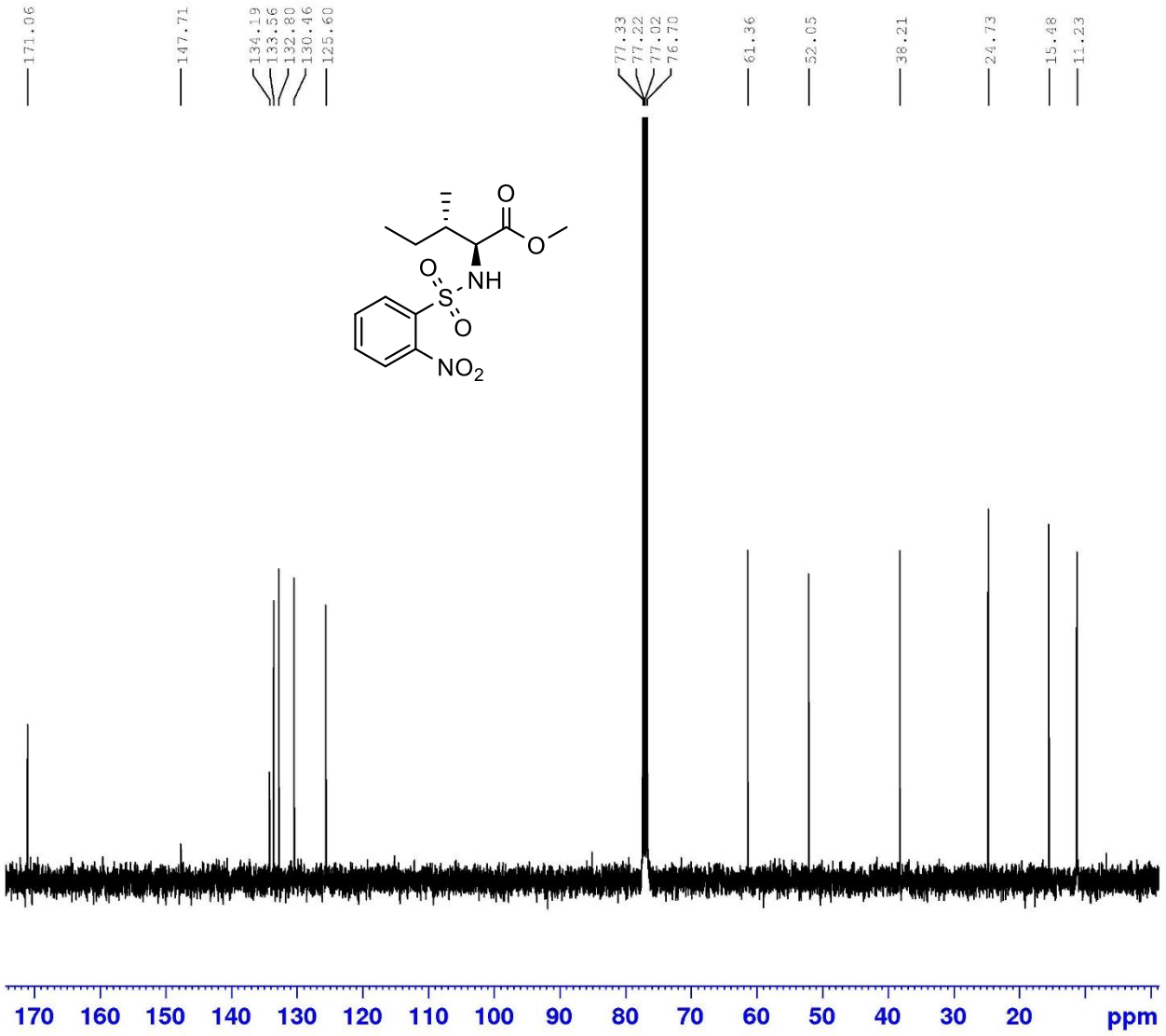
^{13}C-NMR **25e**



BRUKER

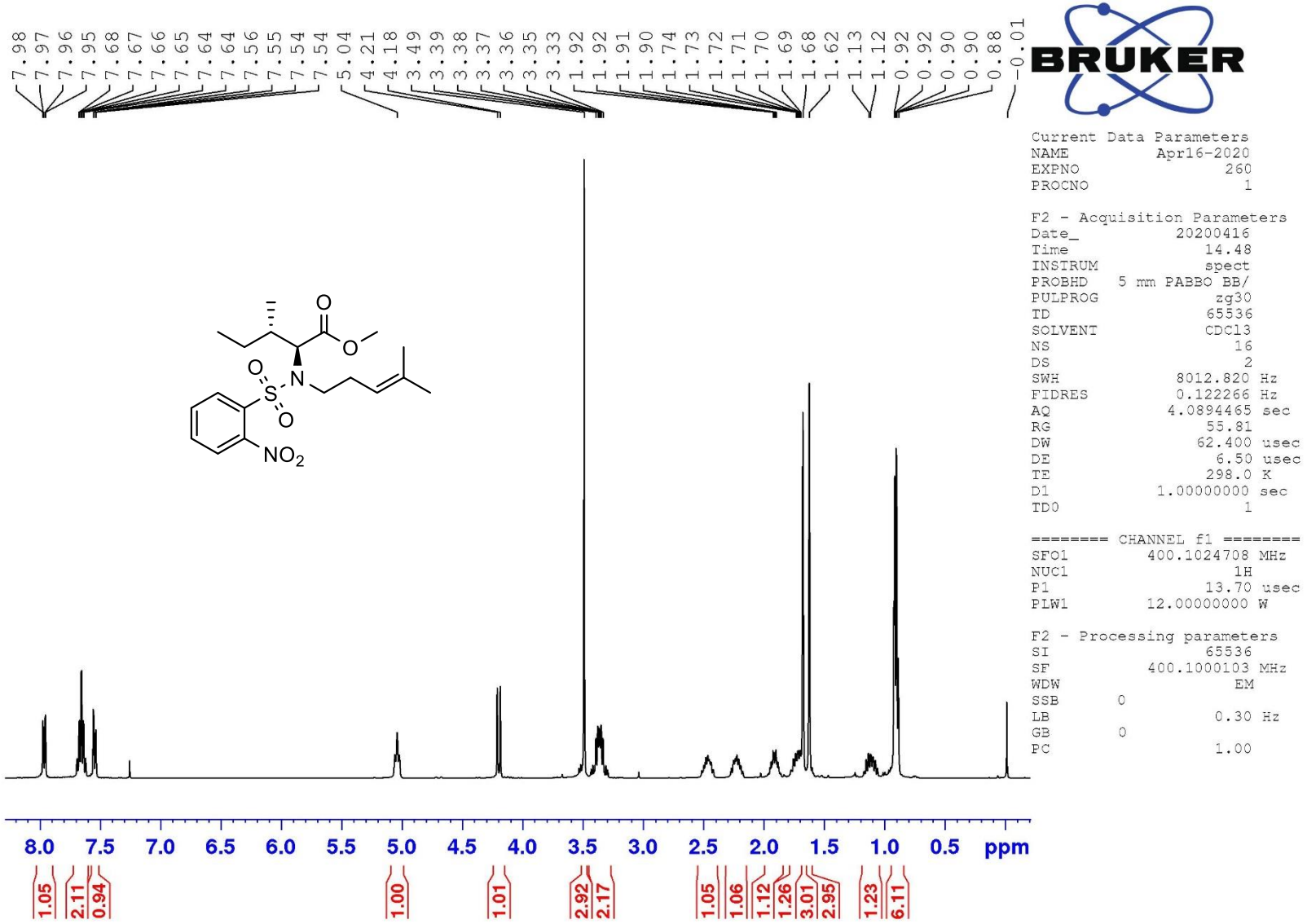
```
Current Data Parameters
NAME          Feb20-2020
EXPNO                480
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200220
Time               15.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 205.35
DW                 62.400 usec
DE                  6.50 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1024708 MHz
NUC1                   1H
P1                 13.70 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1000086 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR **26e**

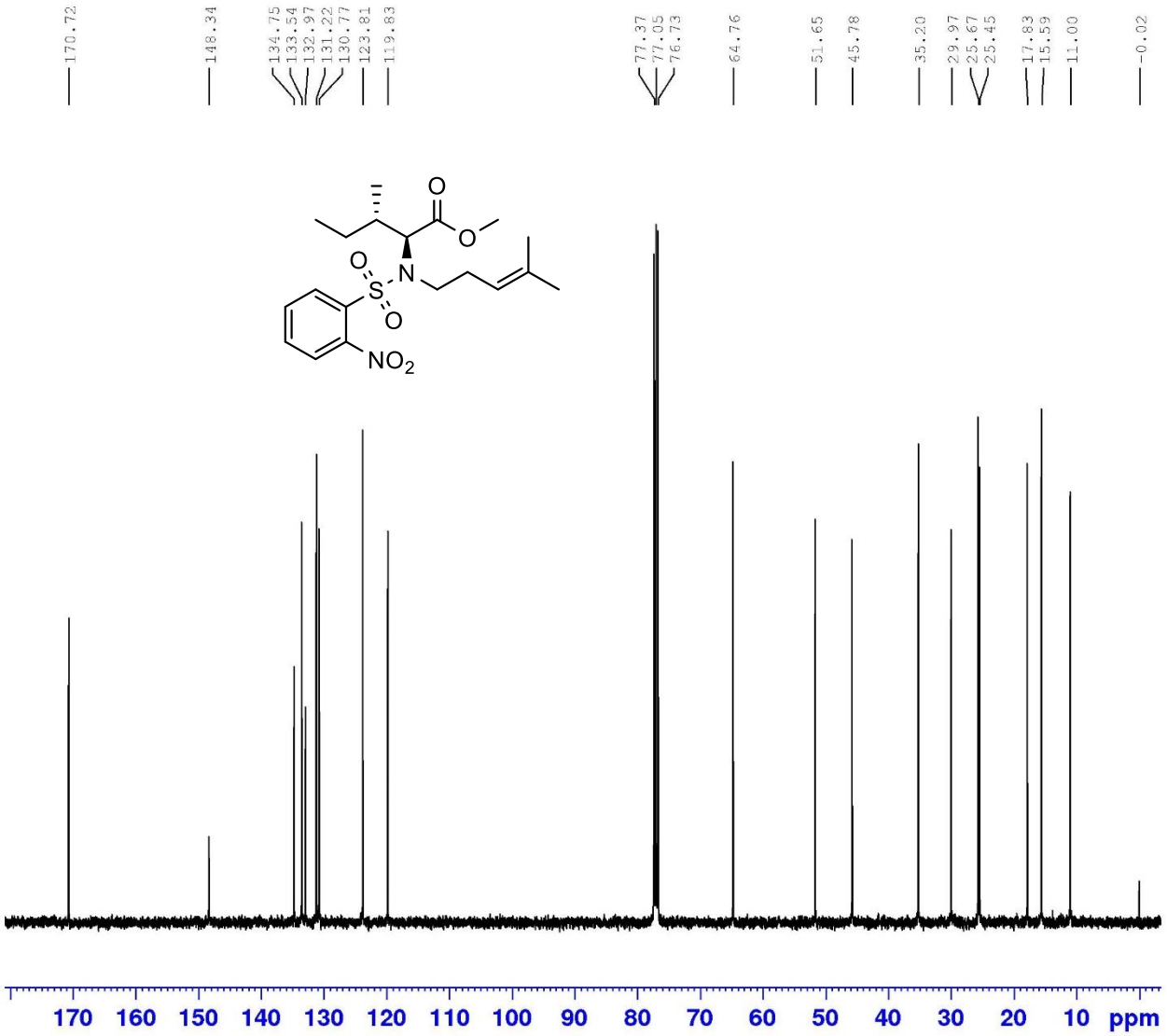



```
Current Data Parameters
NAME          Feb20-2020
EXPNO                481
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200220
Time               23.58
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               205.35
DW               20.800 usec
DE                6.50 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6152851 MHz
NUC1                13C
P1                10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2        400.1016004 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.27805999 W
PLW13         0.22522999 W

F2 - Processing parameters
SI                32768
SF          100.6052250 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C-NMR **26e**

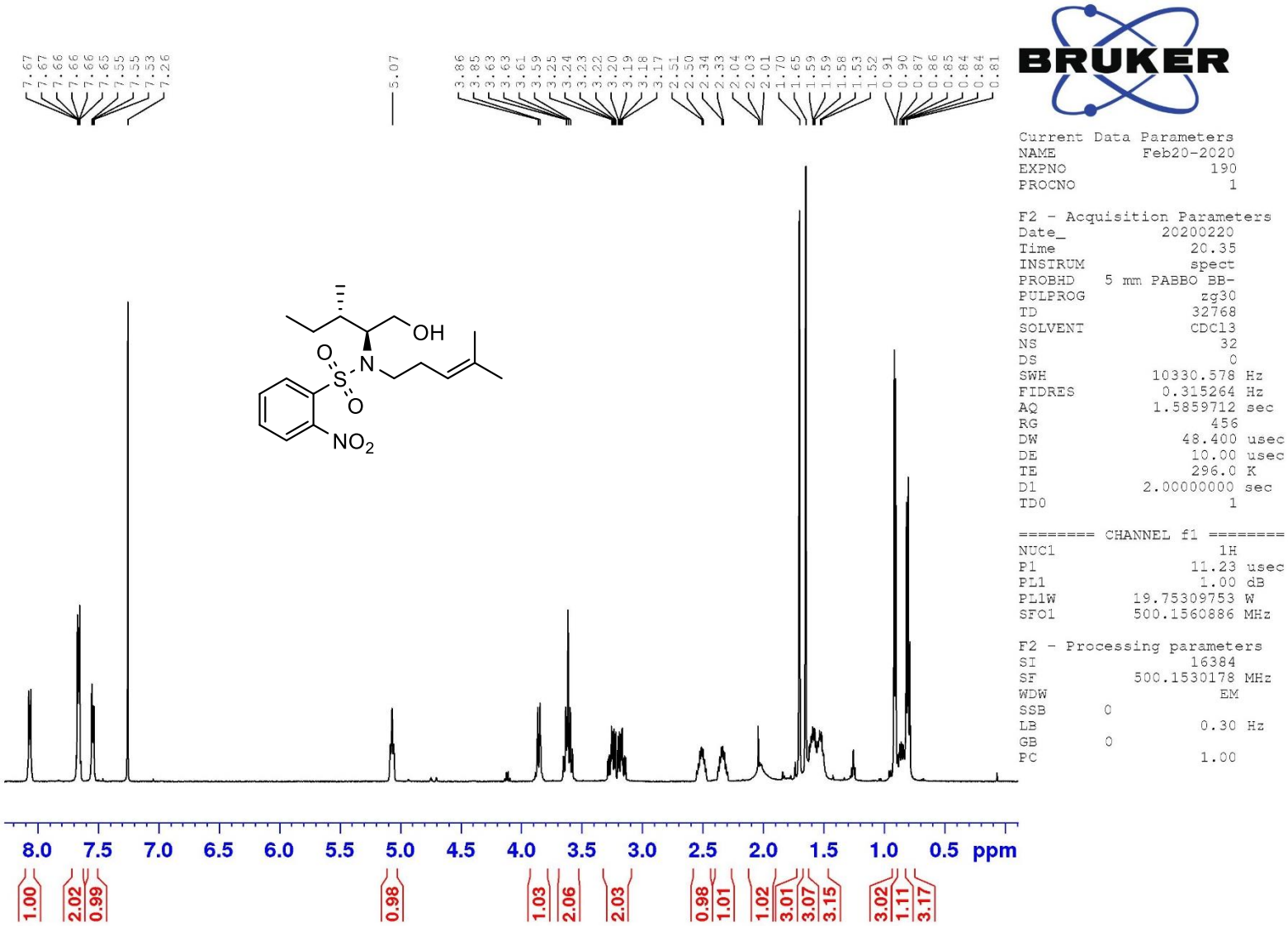

7.98 7.97 7.96 7.95 7.68 7.67 7.66 7.65 7.64 7.64 7.56 7.55 7.54 7.54 5.04 4.21 4.18 3.49 3.39 3.38 3.37 3.36 3.35 3.33 1.92 1.92 1.91 1.90 1.74 1.73 1.72 1.71 1.70 1.69 1.68 1.62 1.13 1.12 0.92 0.92 0.90 0.90 0.88 -0.01
BRUKER
Current Data Parameters
NAME Apr16-2020
EXPNO 260
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200416
Time 14.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 55.81
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1024708 MHz
NUC1 1H
P1 13.70 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1000103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
N
S
O
O
NO2
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.05 2.11 0.94 1.00 1.01 2.92 2.17 1.05 1.06 1.12 1.26 3.01 2.95 1.23 6.11

^{1}H-NMR **27e**




```
Current Data Parameters
NAME          Apr16-2020
EXPNO                261
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200417
Time                1.11
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                205.35
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6152851 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2        400.1016004 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.27805999 W
PLW13         0.22522999 W

F2 - Processing parameters
SI                 32768
SF          100.6052250 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

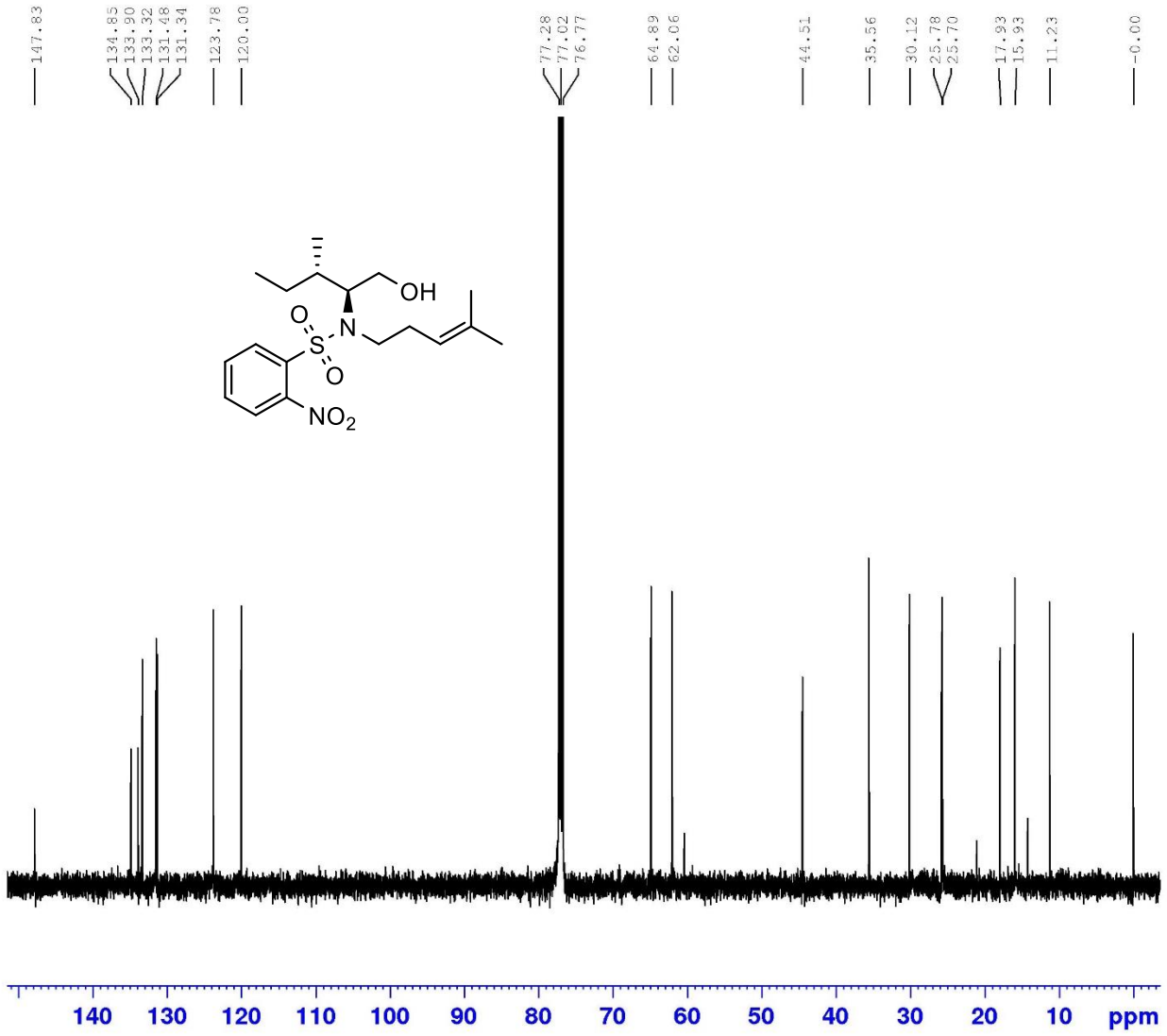
^{13}C-NMR **27e**

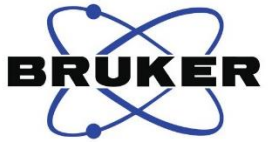


BRUKER

```
Current Data Parameters
NAME          Feb20-2020
EXPNO                190
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200220
Time               20.35
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   456
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530178 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

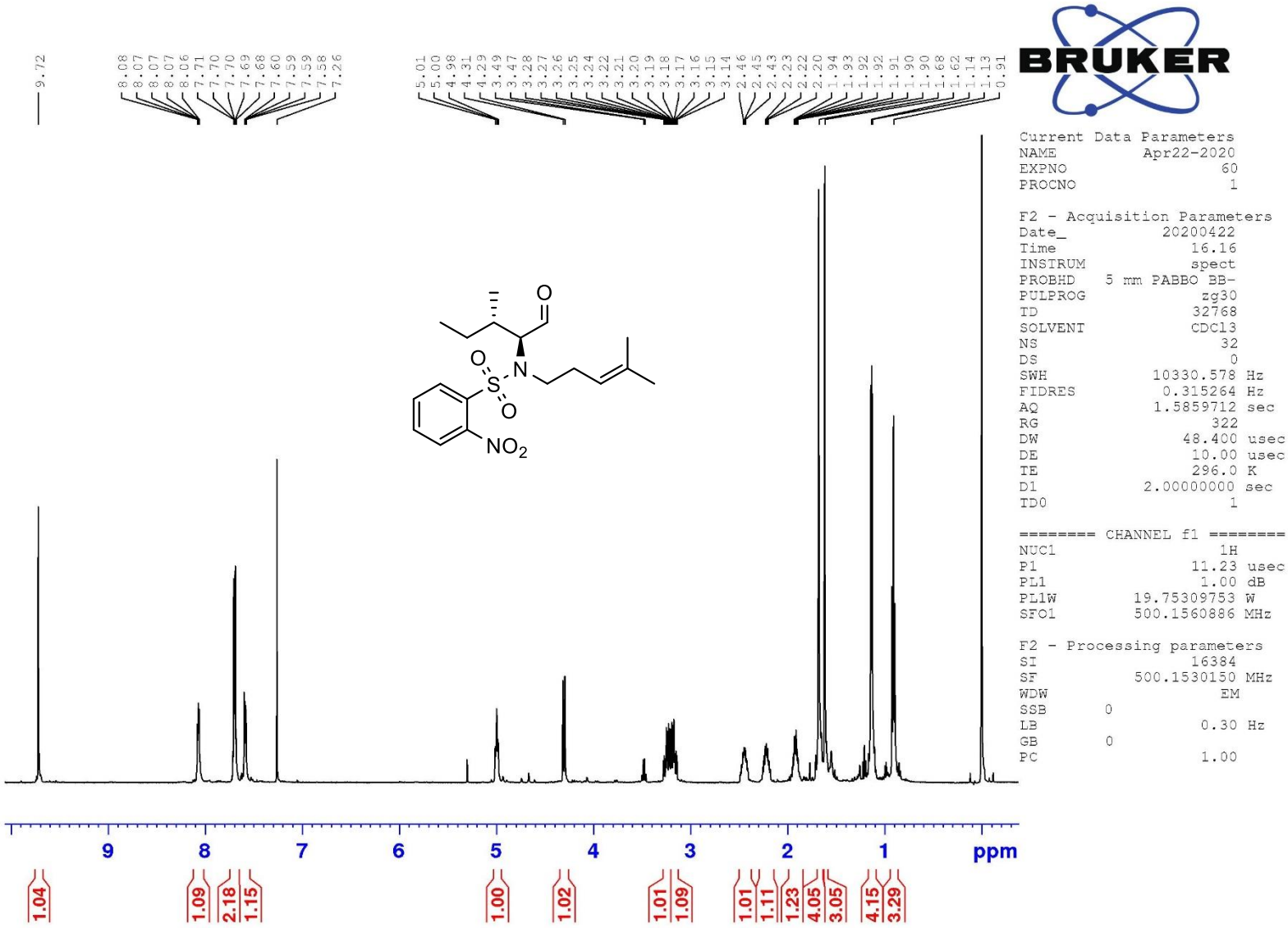
^{1}H-NMR **28e**





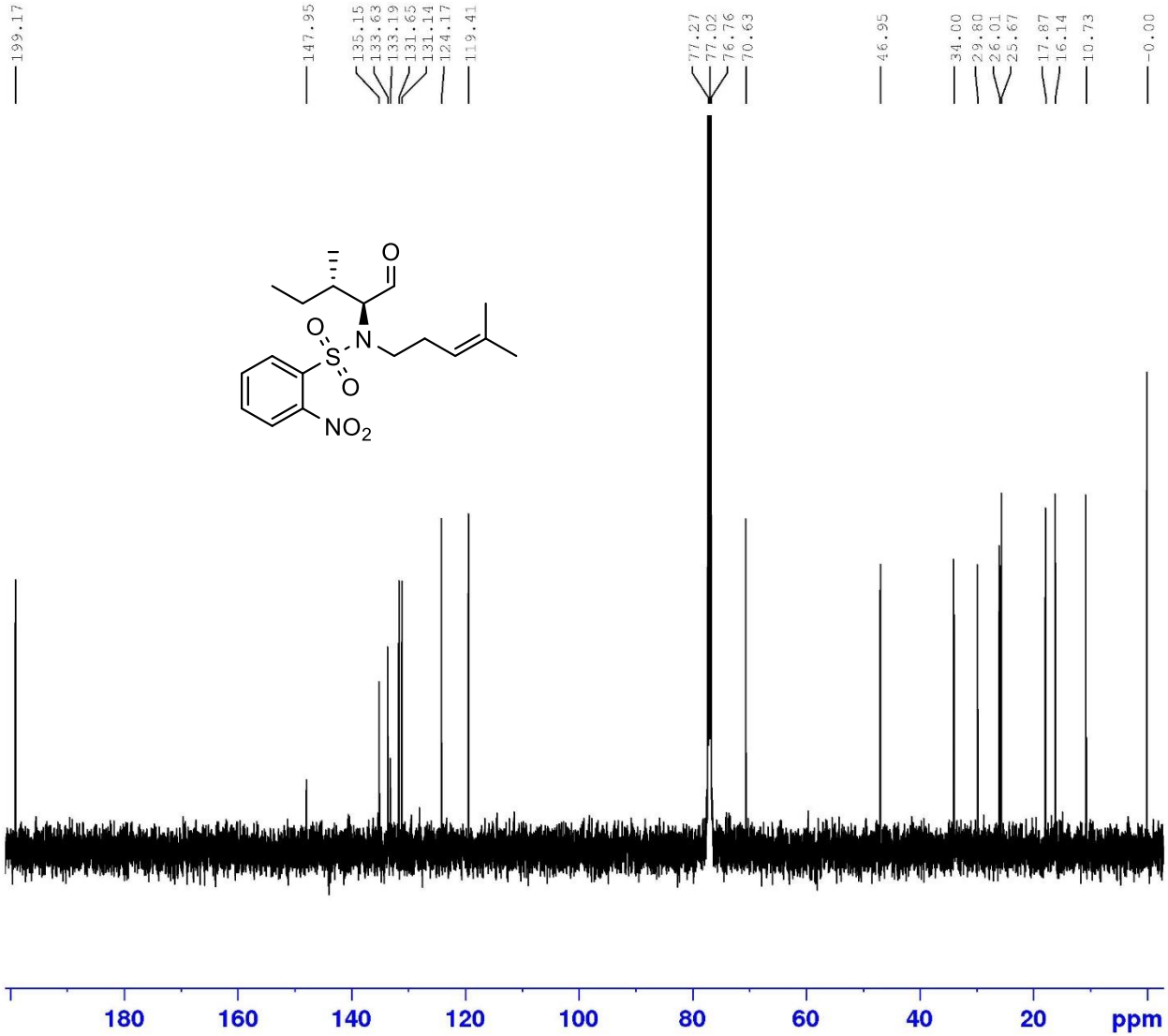

```
Current Data Parameters
NAME          Apr08-2020
EXPNO                 80
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200408
Time               16.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635739 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **28e**




```
Current Data Parameters
NAME          Apr22-2020
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200422
Time               16.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530150 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

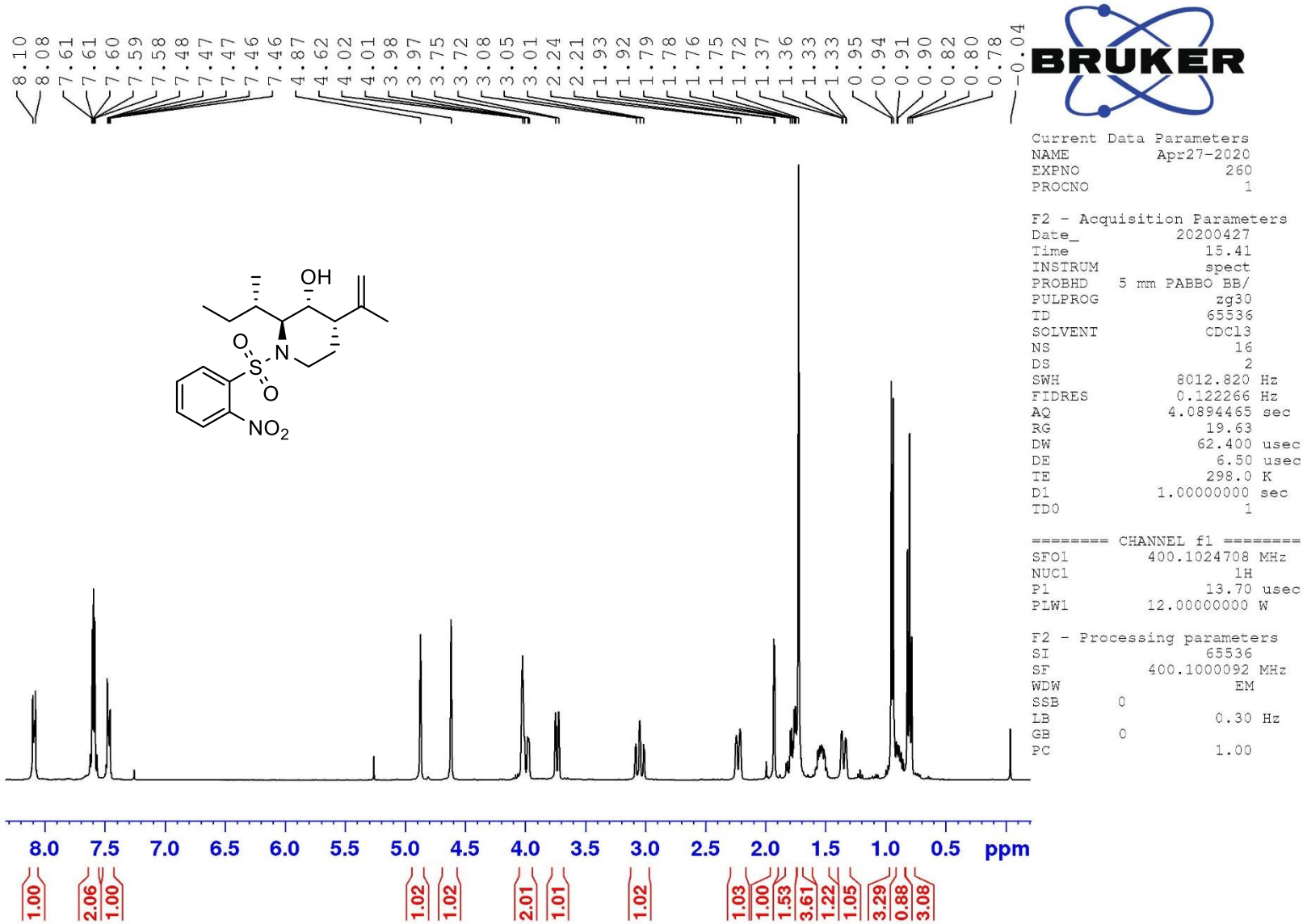
^{1}H-NMR **29e**



BRUKER

```
Current Data Parameters
NAME          Apr22-2020
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200422
Time               17.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635739 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

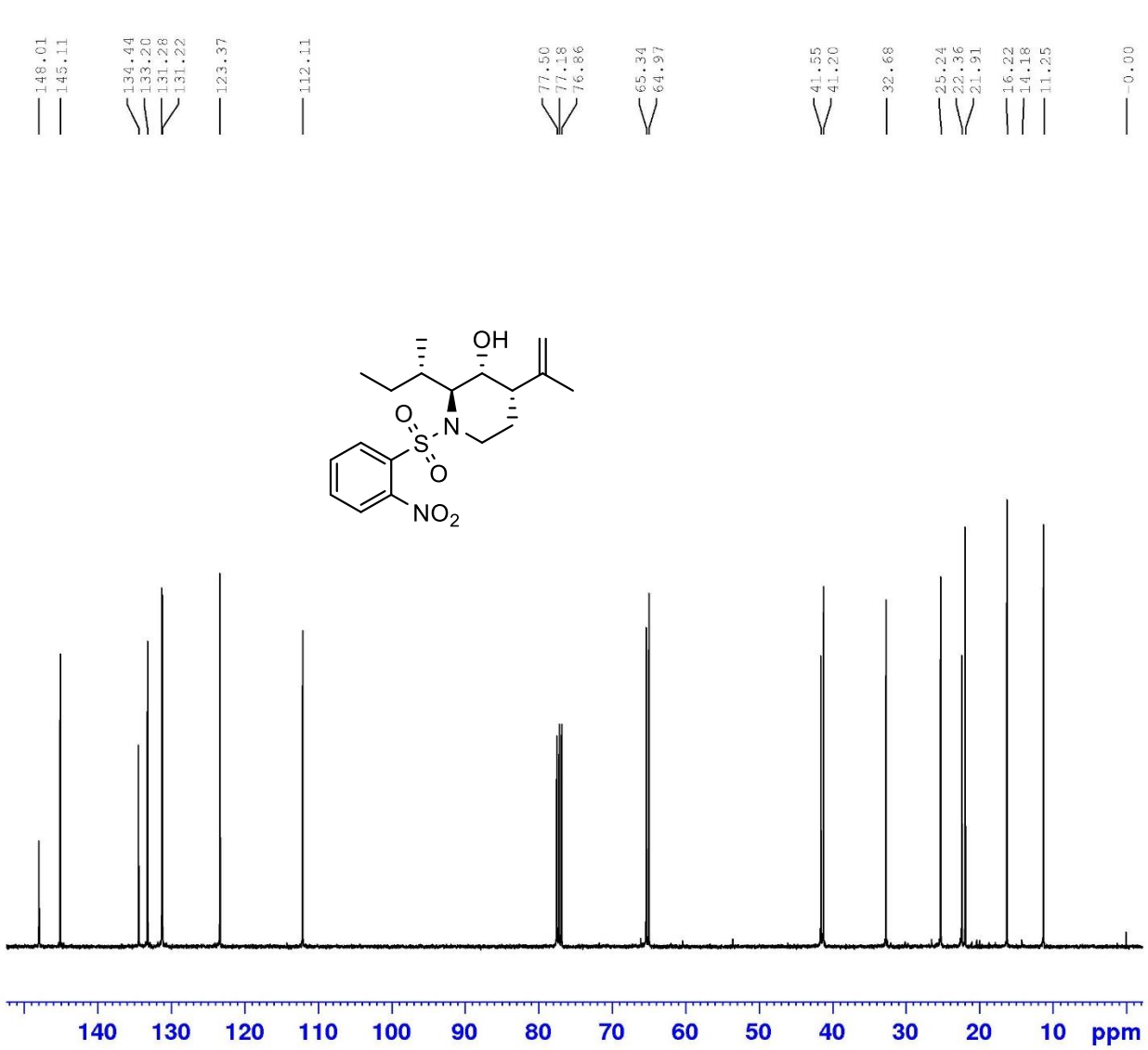
^{13}C-NMR **29e**



BRUKER

```
Current Data Parameters
NAME          Apr27-2020
EXPNO                260
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200427
Time               15.41
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 19.63
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1024708 MHz
NUC1                   1H
P1                 13.70 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1000092 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

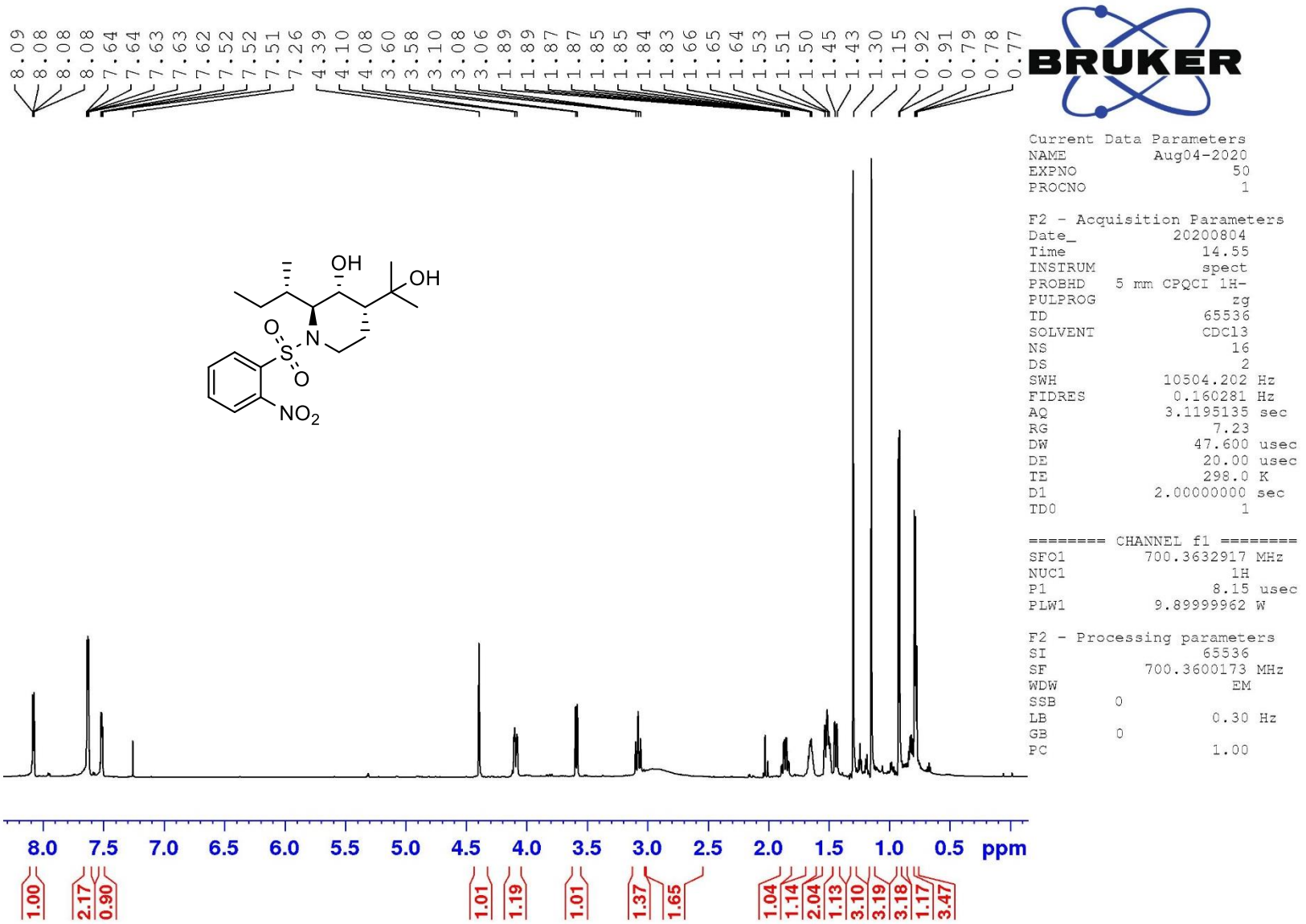
^{1}H-NMR (*R*,*S*)-**30e**



BRUKER

```
Current Data Parameters
NAME          Apr27-2020
EXPNO                261
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200427
Time               21.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                205.35
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6152851 MHz
NUC1                13C
P1                10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2        400.1016004 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.27805999 W
PLW13         0.22522999 W

F2 - Processing parameters
SI                 32768
SF          100.6052251 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

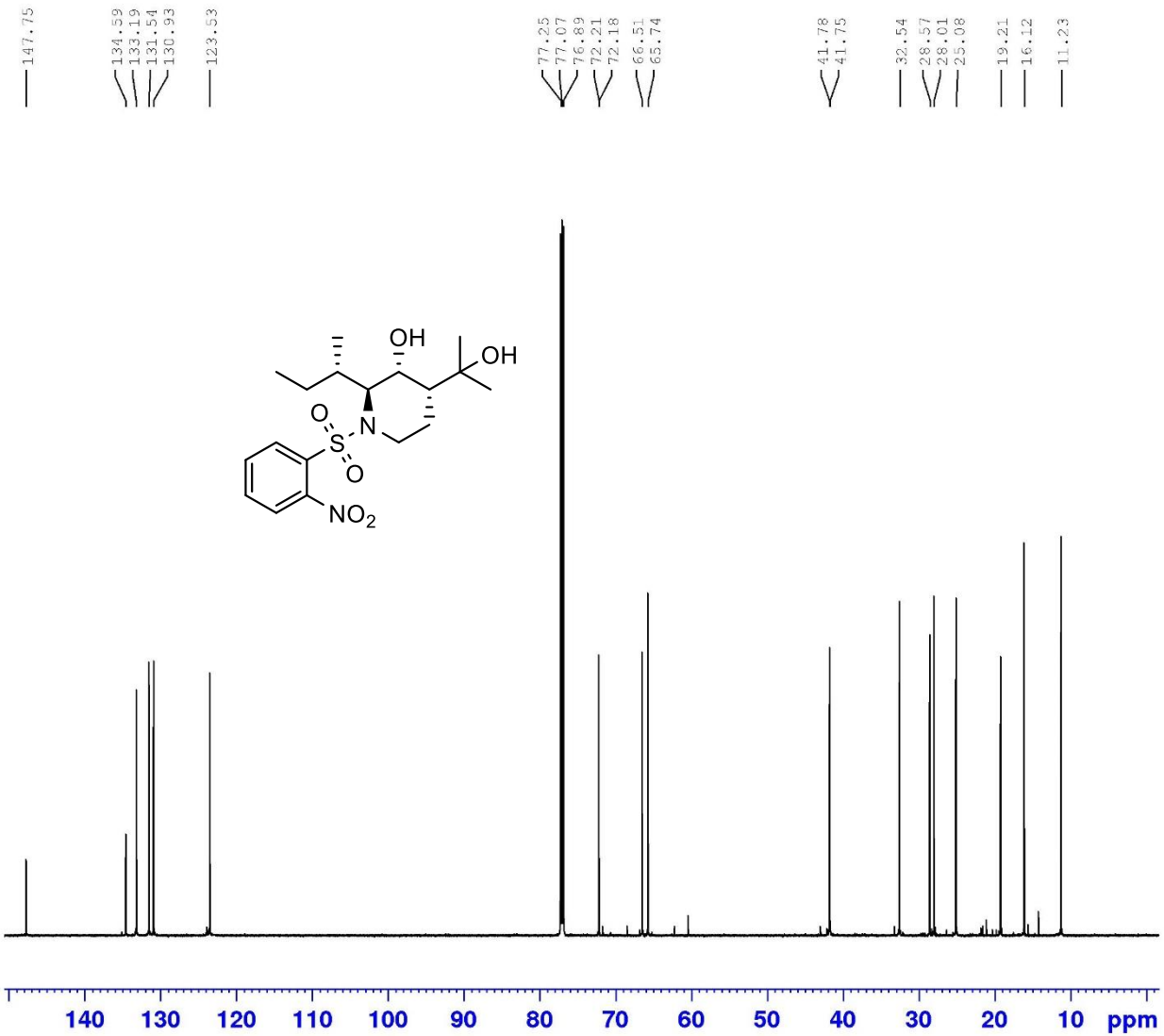
^{13}C-NMR (*R*,*S*)-**30e**

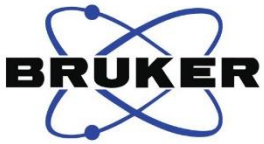


BRUKER

```
Current Data Parameters
NAME          Aug04-2020
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200804
Time               14.55
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG                zg
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10504.202 Hz
FIDRES          0.160281 Hz
AQ             3.1195135 sec
RG                  7.23
DW                47.600 usec
DE                20.00 usec
TE                298.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         700.3632917 MHz
NUC1                   1H
P1                  8.15 usec
PLW1          9.89999962 W

F2 - Processing parameters
SI                 65536
SF           700.3600173 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

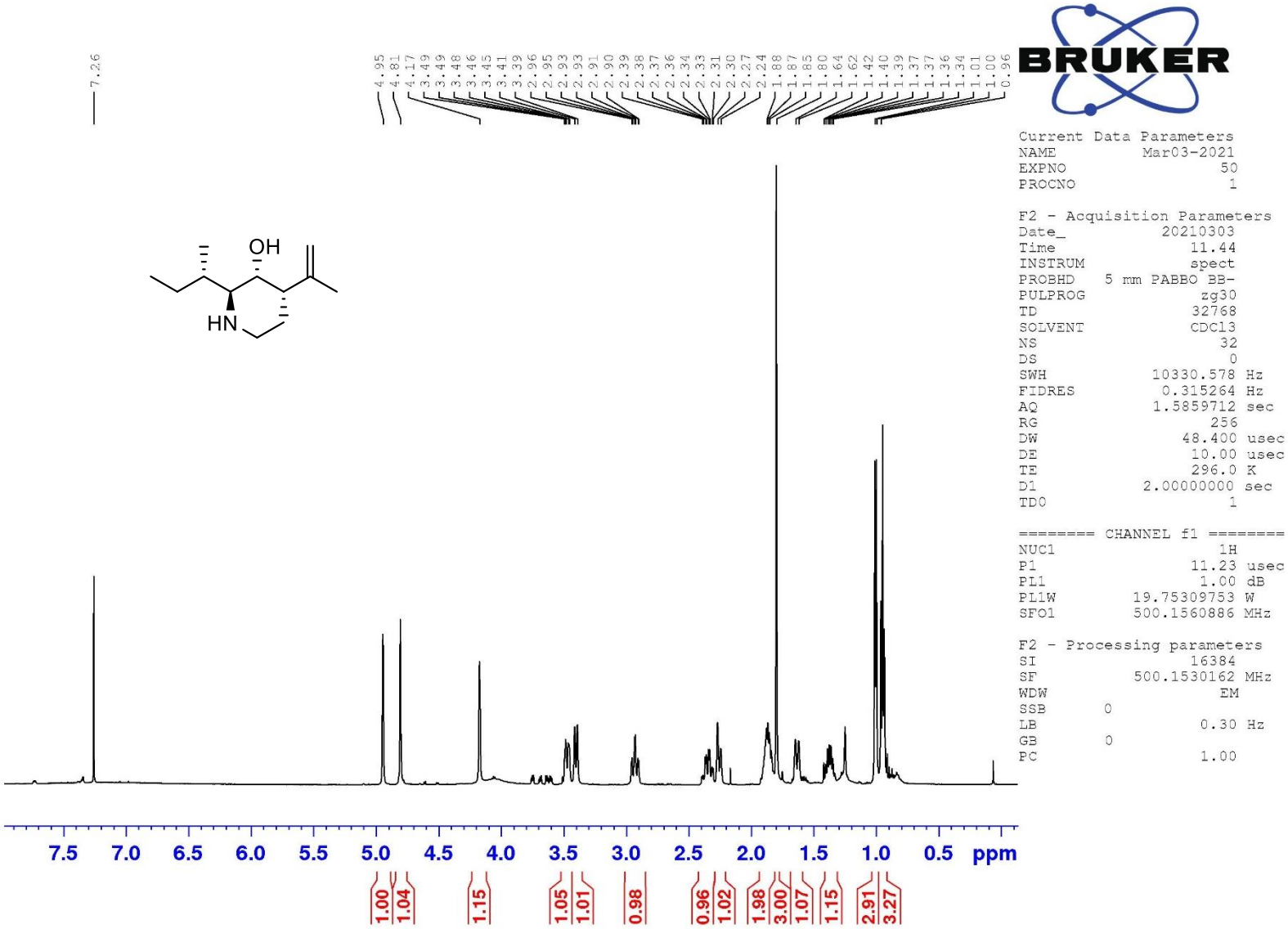
^{1}H-NMR (*R*,*R*)-**31e**





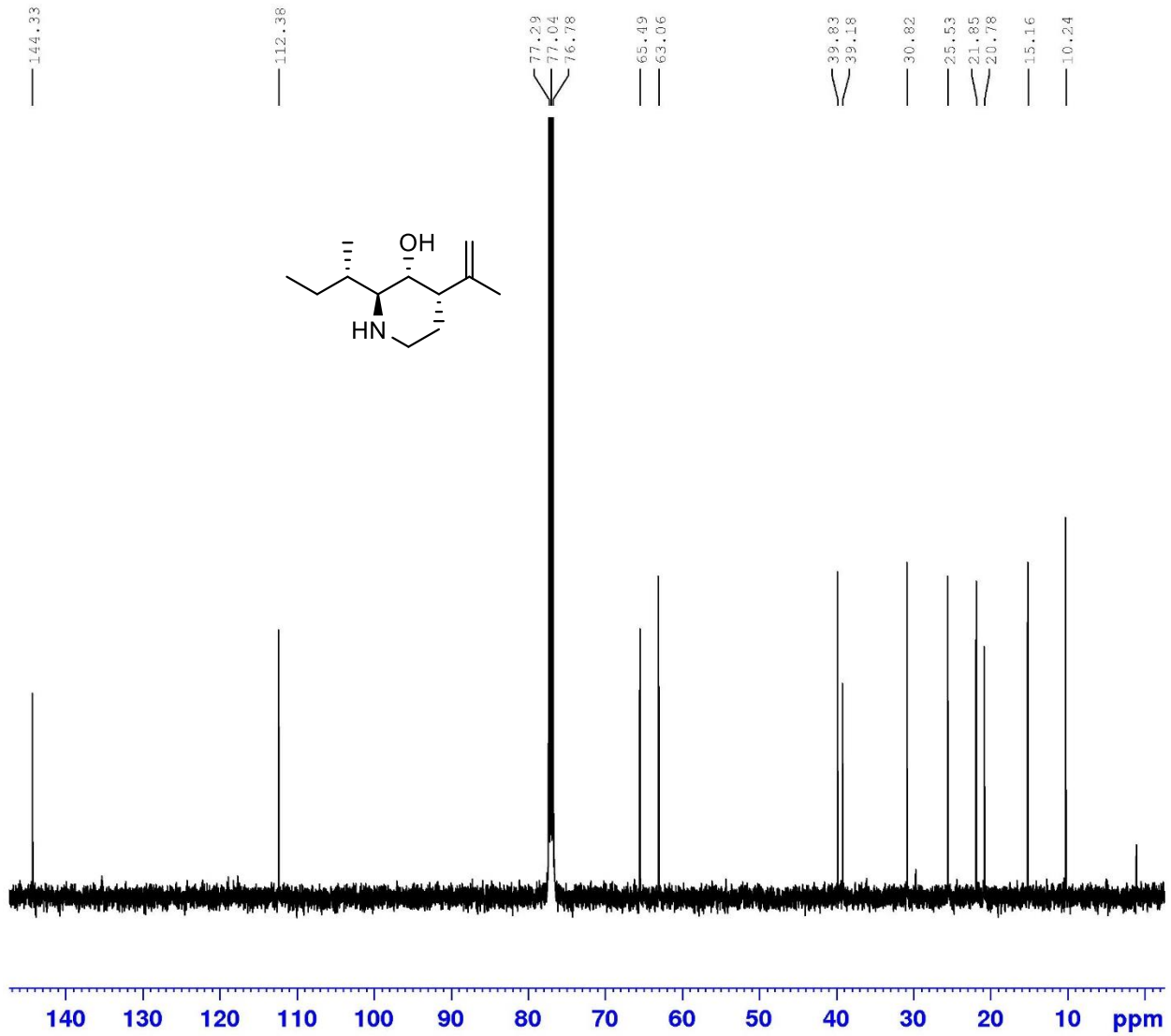

```
Current Data Parameters
NAME          Aug04-2020
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200804
Time               15.21
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C-NMR (*R*,*R*)-**31e**




```
Current Data Parameters
NAME          Mar03-2021
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210303
Time               11.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   256
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530162 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*R*,*S*)-**32e**



^{13}C-NMR (*R*,*S*)-**32e**

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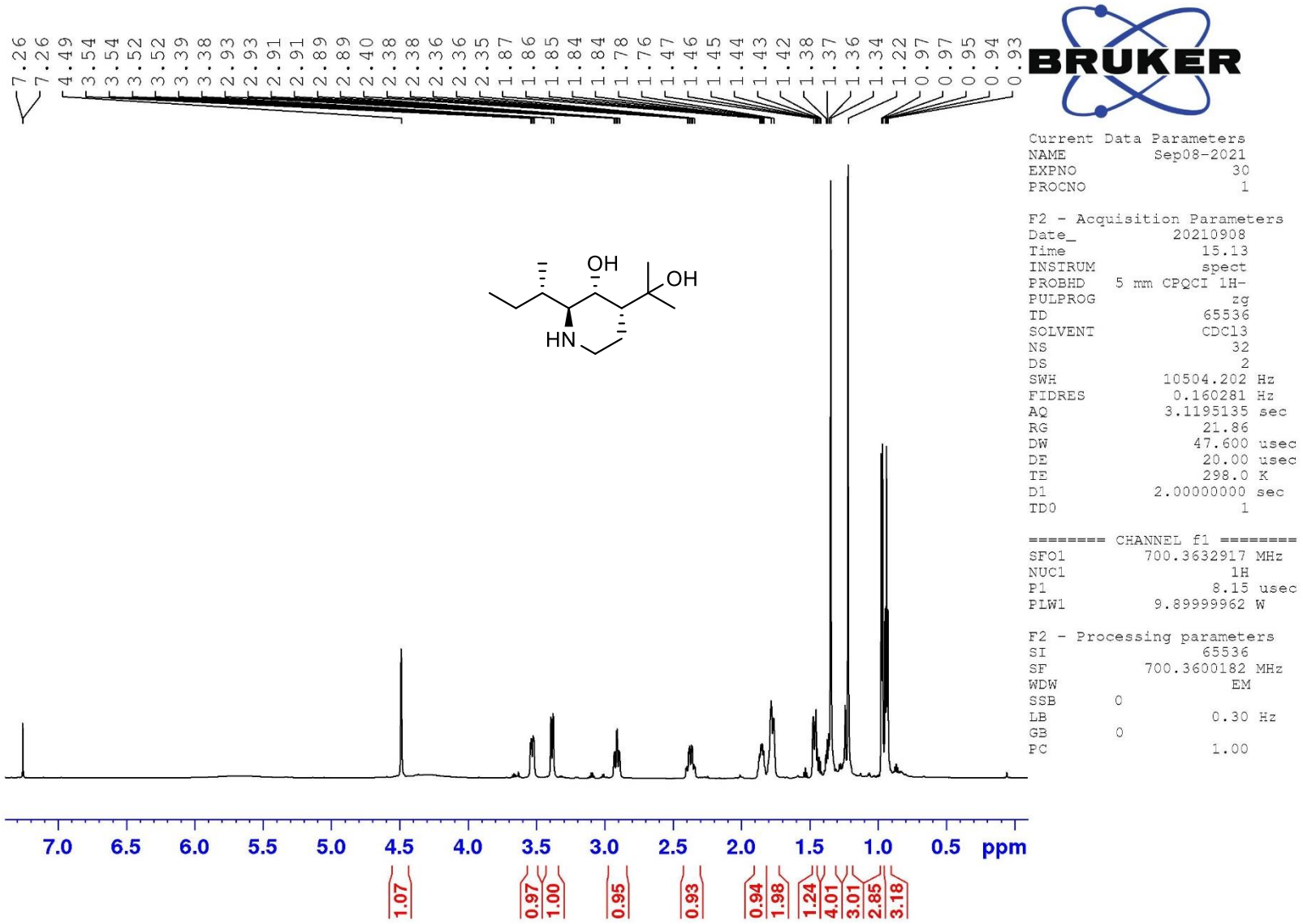
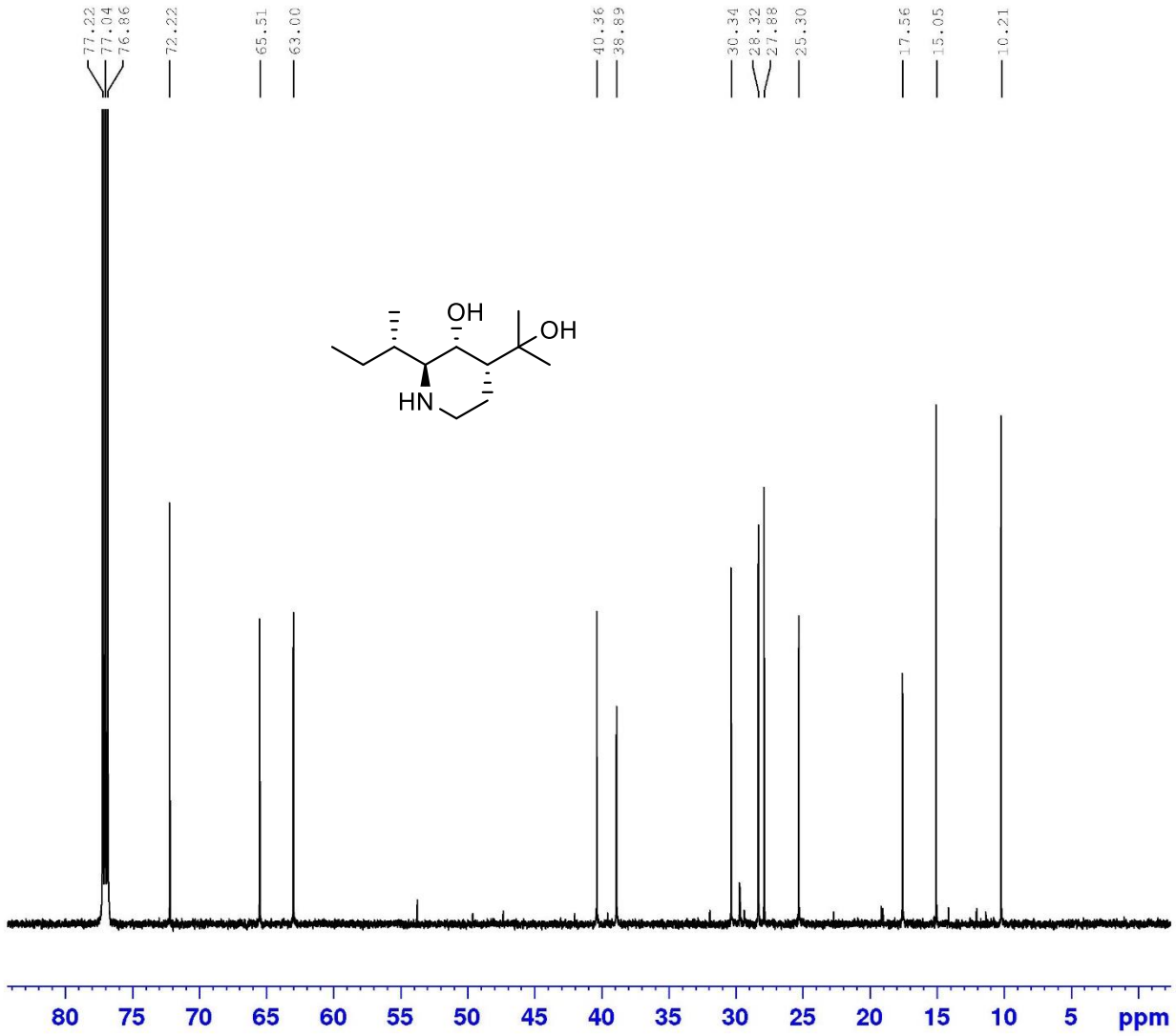
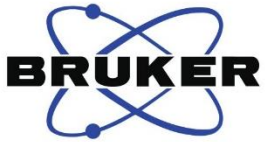
```
Current Data Parameters
NAME          Jan26-2021
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210126
Time               17.21
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES        0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME          Sep08-2021
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210908
Time               15.13
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG               zg
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    2
SWH           10504.202 Hz
FIDRES         0.160281 Hz
AQ            3.1195135 sec
RG                21.86
DW               47.600 usec
DE               20.00 usec
TE               298.0 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        700.3632917 MHz
NUC1                  1H
P1                 8.15 usec
PLW1          9.89999962 W

F2 - Processing parameters
SI                65536
SF          700.3600182 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

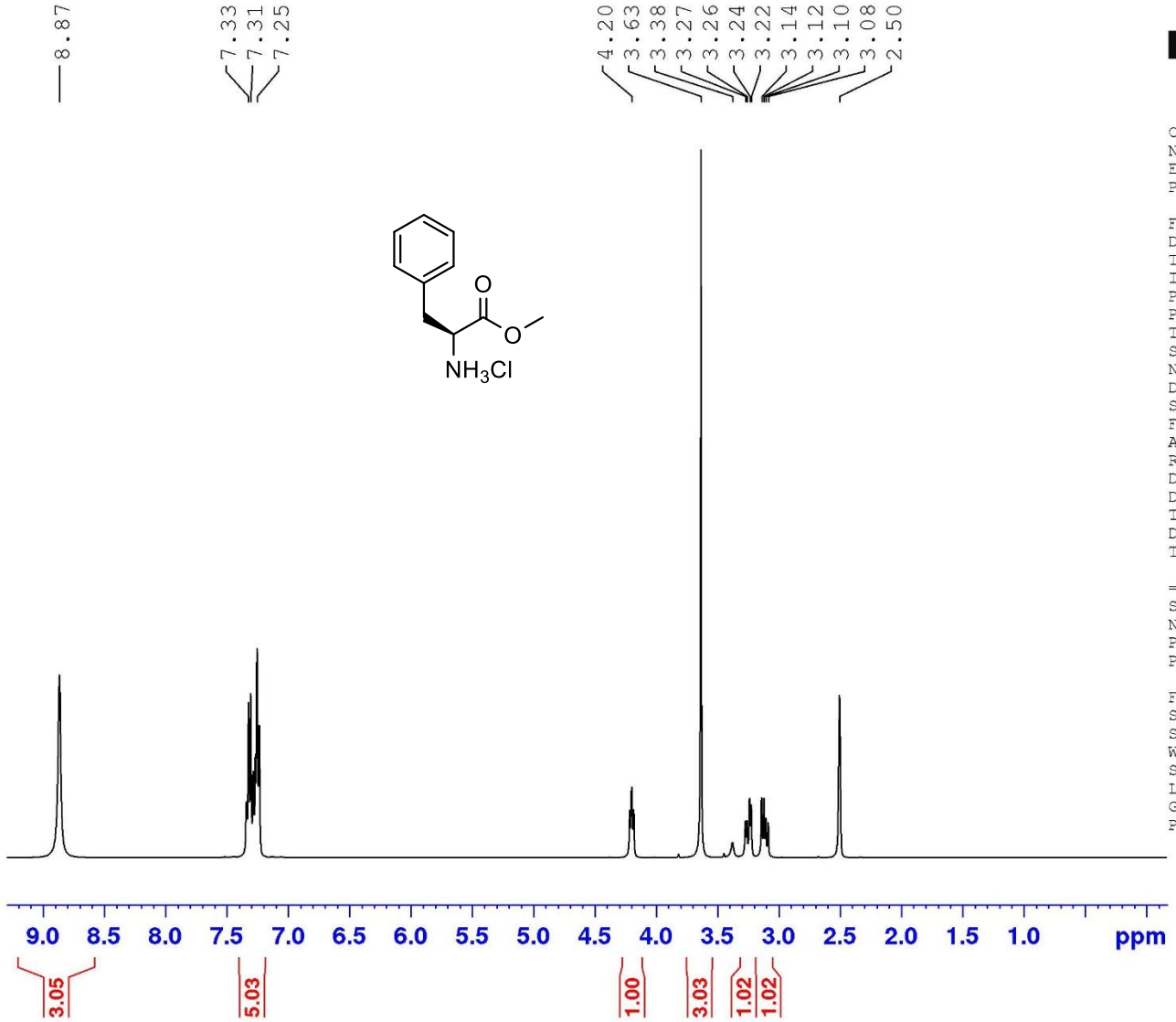
^{1}H-NMR (*R*,*R*)-**33e**





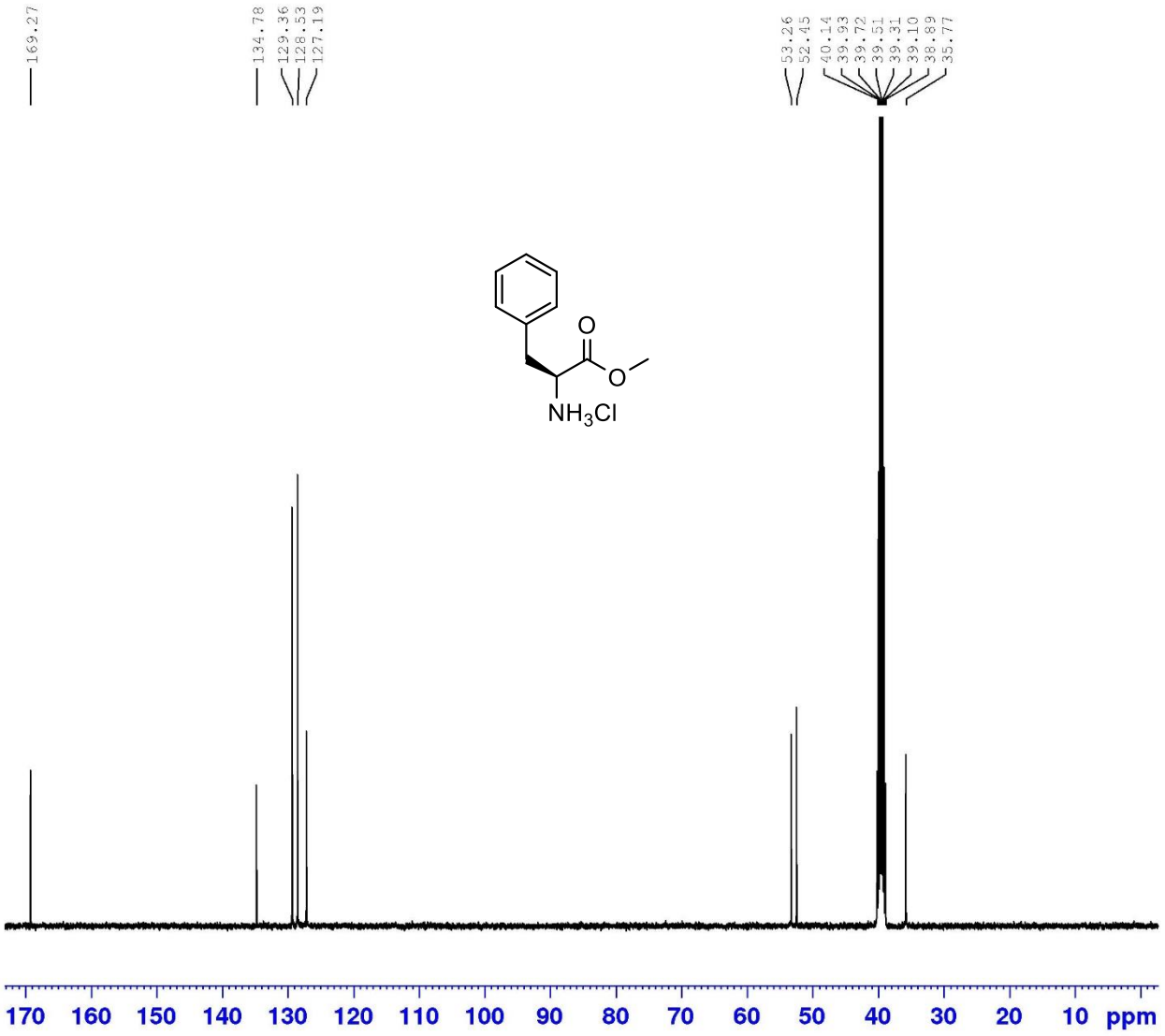

```
Current Data Parameters
NAME          Sep08-2021
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210908
Time               16.54
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ             0.8017003 sec
RG                182.53
DW                12.267 usec
DE                18.00 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         176.1232717 MHz
NUC1                  13C
P1                 12.00 usec
PLW1        105.00000000 W

======== CHANNEL f2 ========
SFO2         700.3628014 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF           176.1056620 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

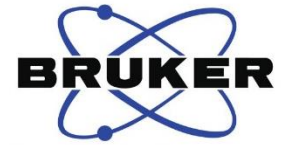
^{13}C-NMR (*R*,*R*)-**33e**




```
Current Data Parameters
NAME          Mar10-2020
EXPNO                350
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200310
Time               13.46
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 90.96
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1024708 MHz
NUC1                   1H
P1                 13.70 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1000000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

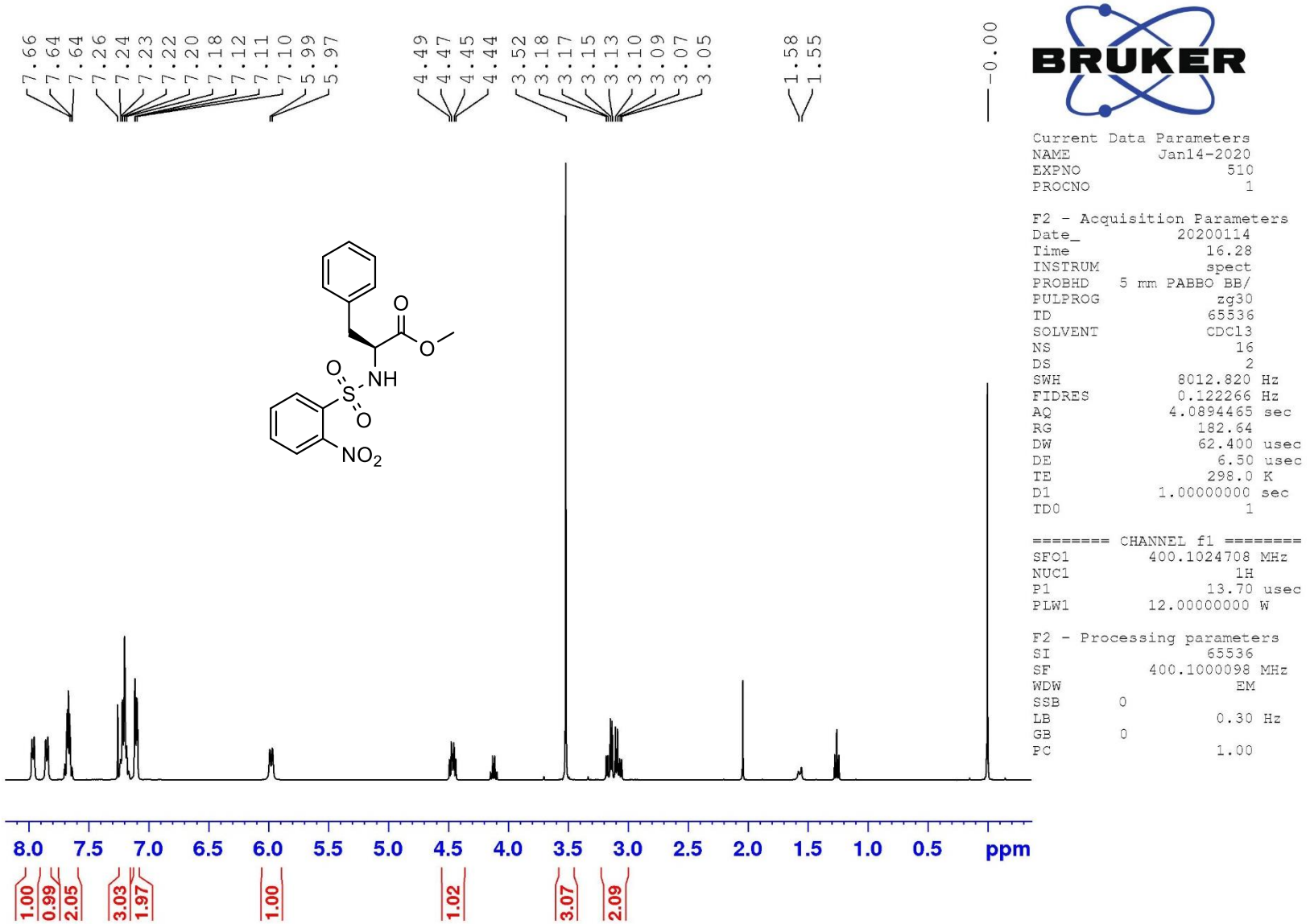
^{1}H-NMR **25f**





```
Current Data Parameters
NAME          Mar10-2020
EXPNO                351
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200311
Time                2.07
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                   512
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                205.35
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6152851 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1016004 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.27805999 W
PLW13         0.22522999 W

F2 - Processing parameters
SI                 32768
SF           100.6052749 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **25f**



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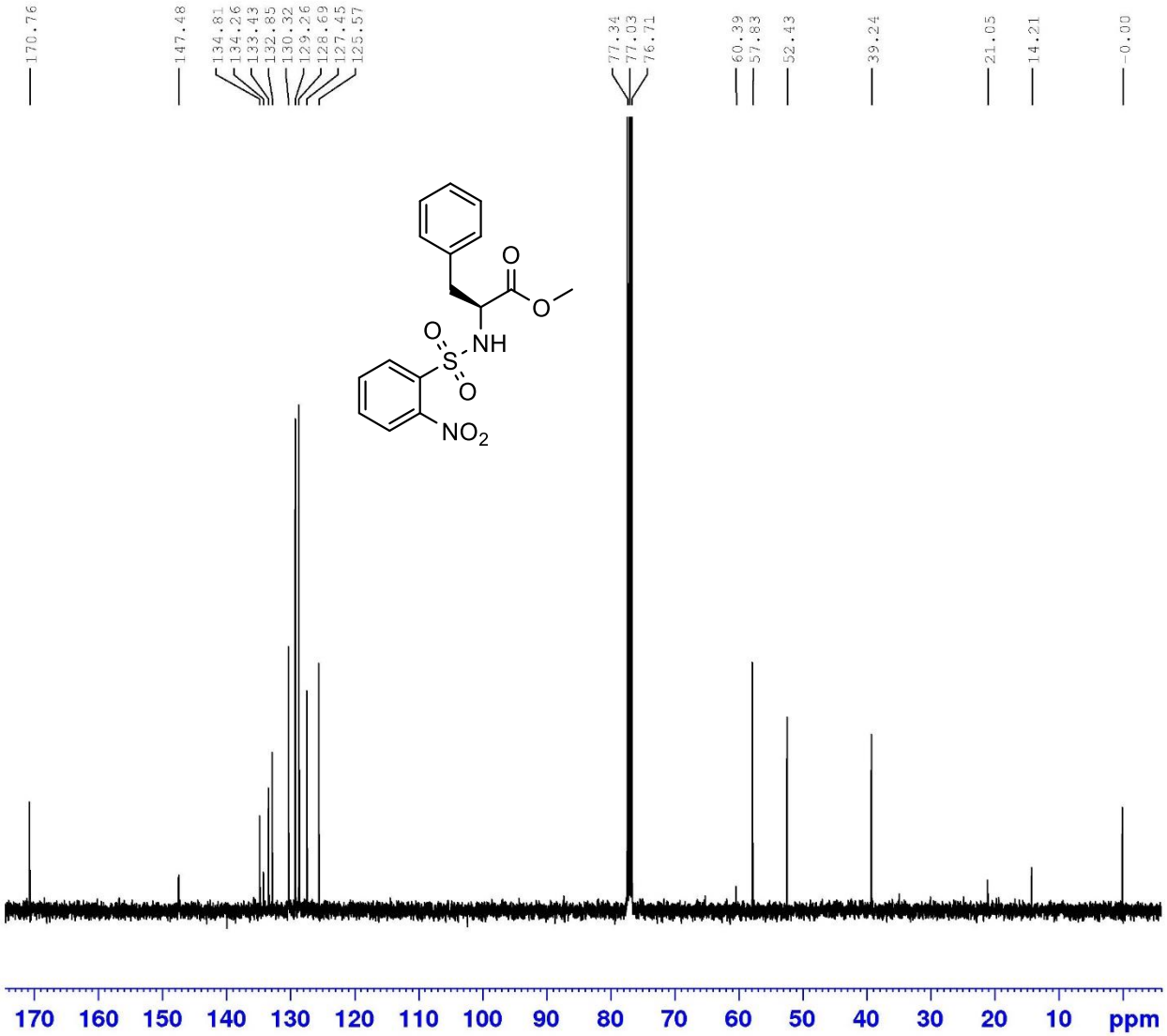
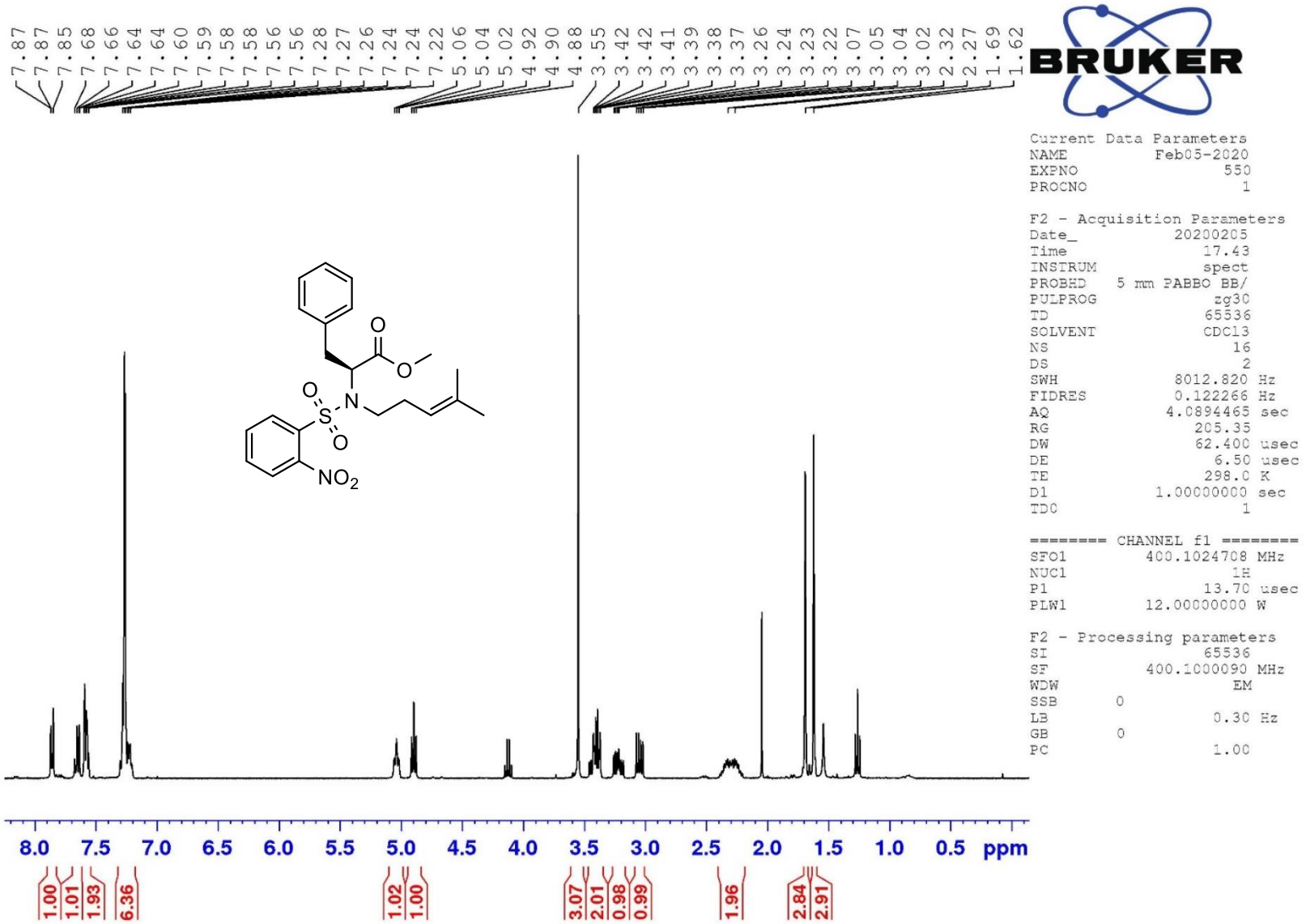
```
Current Data Parameters
NAME          Jan14-2020
EXPNO                510
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200114
Time               16.28
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 182.64
DW                 62.400 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1024708 MHz
NUC1                   1H
P1                 13.70 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1000098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR **26f**

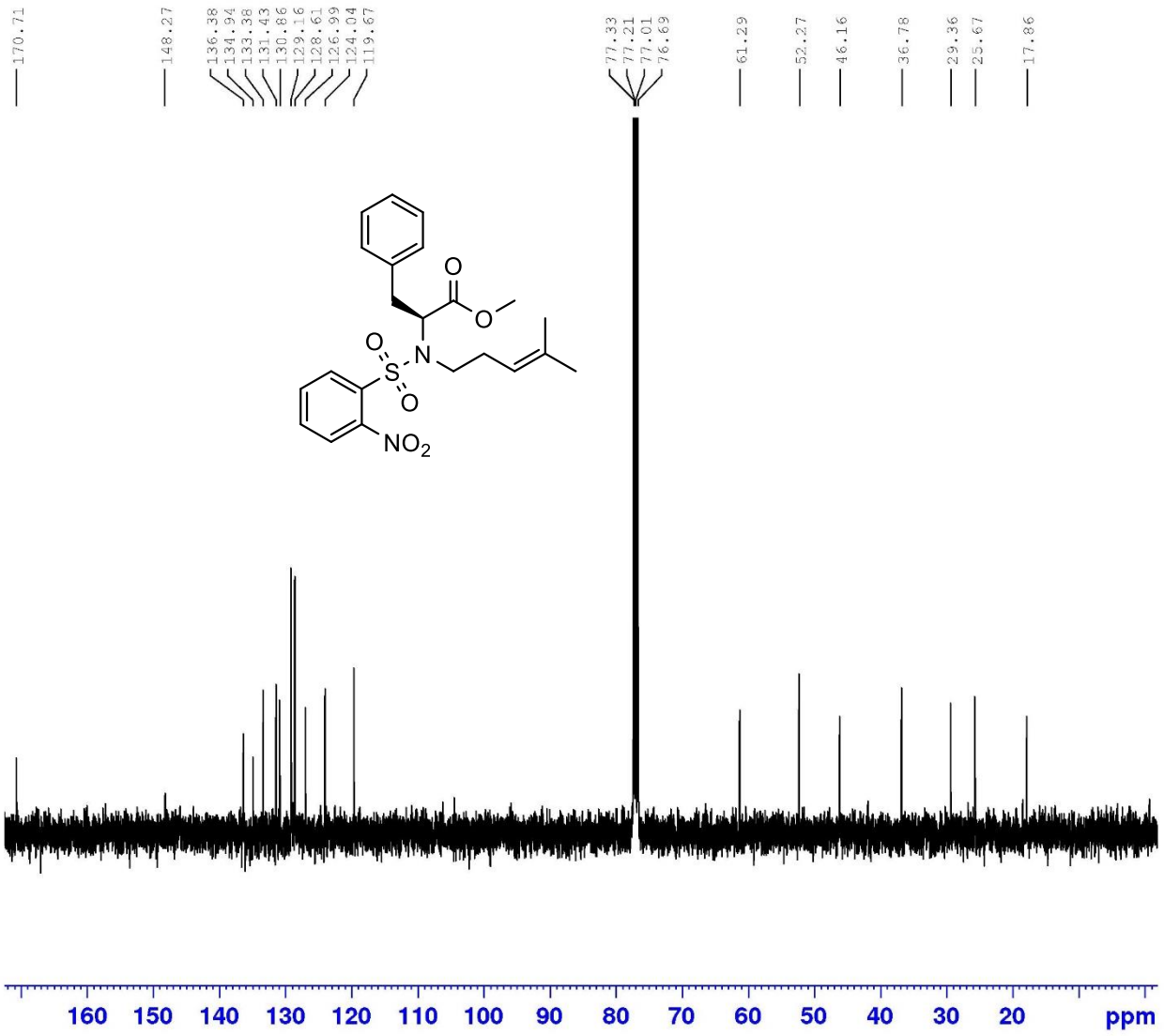


^{13}C-NMR **26f**

```
Current Data Parameters
NAME          Jan14-2020
EXPNO                511
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200114
Time               22.14
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                205.35
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6152851 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1016004 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.27805999 W
PLW13         0.22522999 W

F2 - Processing parameters
SI                 32768
SF           100.6052247 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```


7.87
7.87
7.85
7.68
7.66
7.64
7.64
7.60
7.59
7.58
7.58
7.56
7.56
7.28
7.27
7.26
7.24
7.24
7.22
5.06
5.04
5.02
4.92
4.90
4.88
3.55
3.42
3.42
3.41
3.39
3.38
3.37
3.26
3.24
3.23
3.22
3.07
3.05
3.04
3.02
2.32
2.27
1.69
1.62
BRUKER
Current Data Parameters
NAME Feb05-2020
EXPNO 550
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200205
Time 17.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 205.35
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1024708 MHz
NUC1 1H
P1 13.70 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1000090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
N
O
S
O
NO2
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.01
1.93
6.36
1.02
1.00
3.07
2.01
0.98
0.99
1.96
2.84
2.91

^{1}H-NMR **27f**



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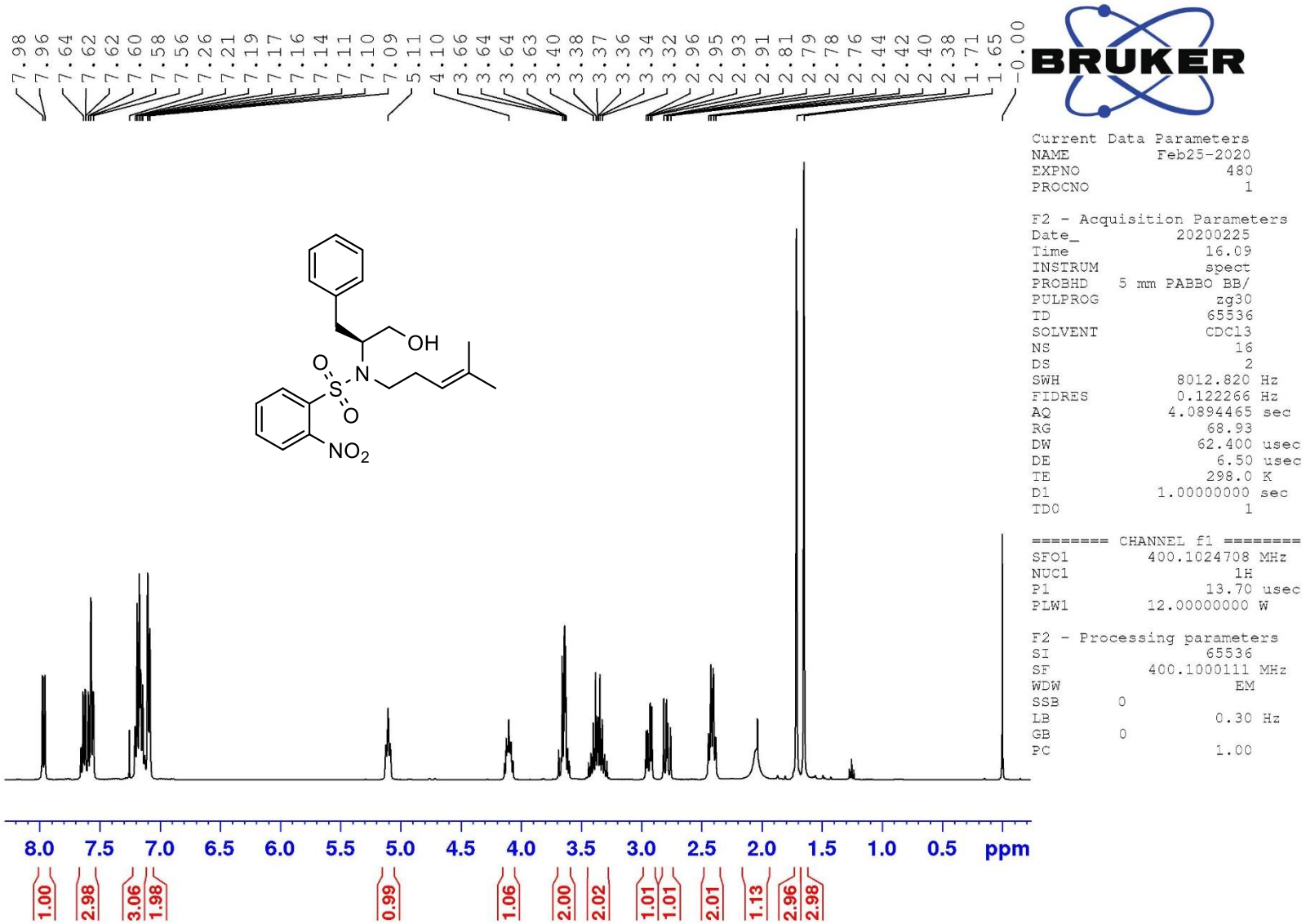
```
Current Data Parameters
NAME          Feb05-2020
EXPNO                551
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200205
Time               20.34
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               205.35
DW               20.800 usec
DE                6.50 usec
TE               298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6152851 MHz
NUC1                13C
P1                10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2        400.1016004 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.27805999 W
PLW13         0.22522999 W

F2 - Processing parameters
SI                 32768
SF          100.6052250 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

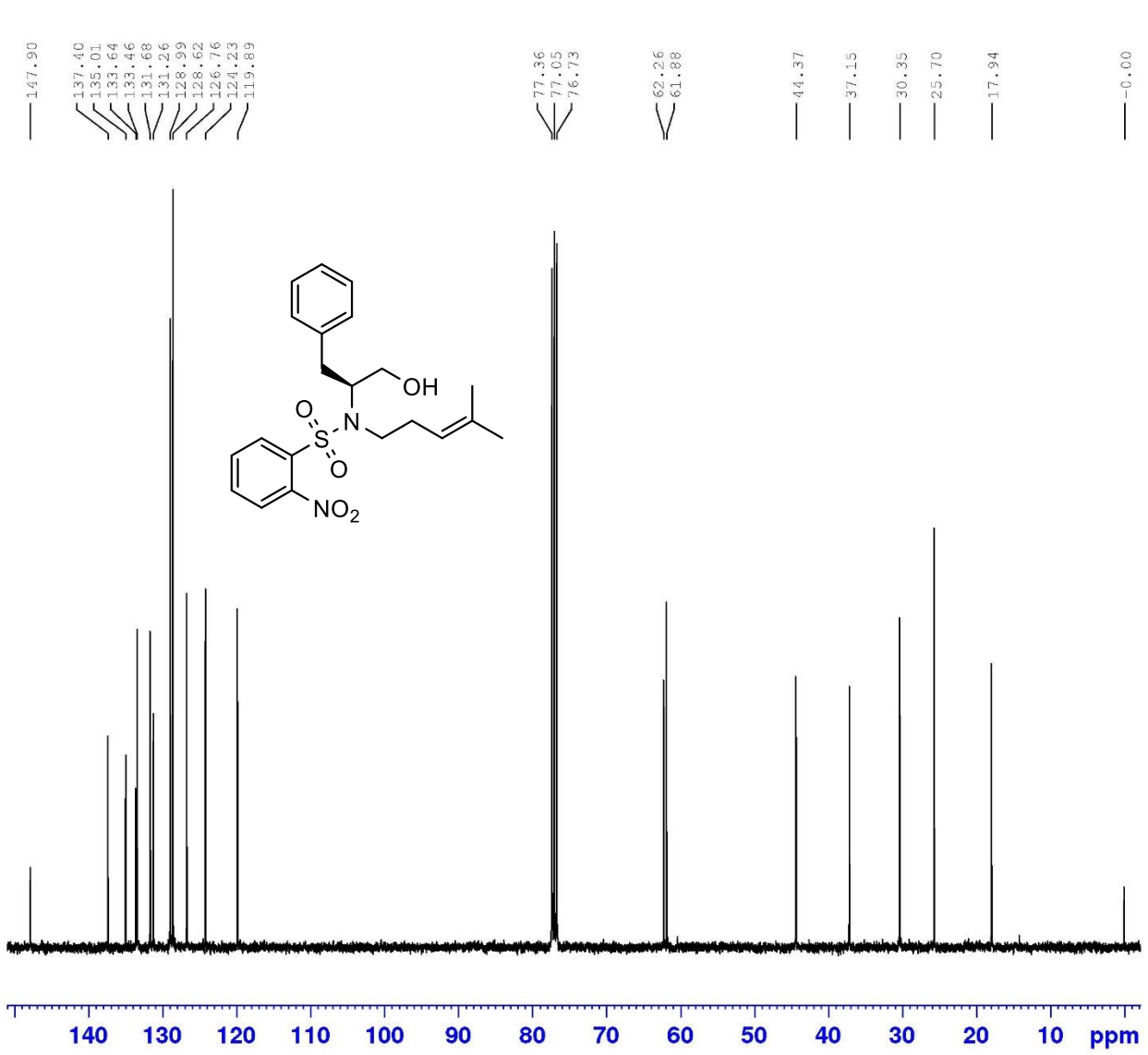
^{13}C-NMR **27f**



BRUKER

```
Current Data Parameters
NAME          Feb25-2020
EXPNO                480
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200225
Time               16.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 68.93
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1024708 MHz
NUC1                   1H
P1                 13.70 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1000111 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR **28f**



Current Data Parameters
NAME Feb25-2020
EXPNO 481
PROCNO 1

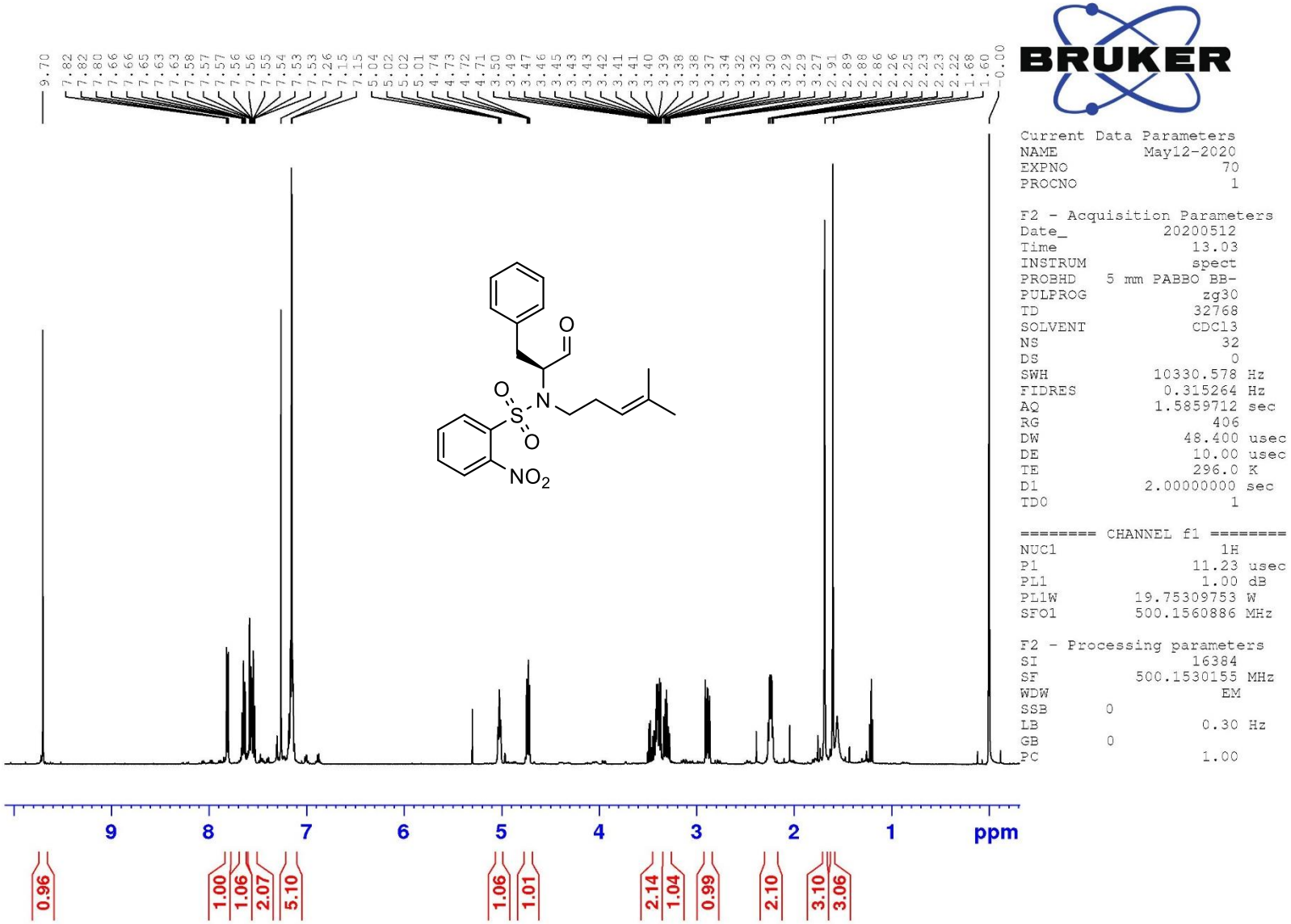
F2 - Acquisition Parameters
Date_ 20200226
Time 1.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 205.35
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
ID0 1

======== CHANNEL f1 ========
SFO1 100.6152851 MHz
NUC1 13C
P1 10.00 usec
PLW1 48.00000000 W

======== CHANNEL f2 ========
SFO2 400.1016004 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.27805999 W
PLW13 0.22522999 W

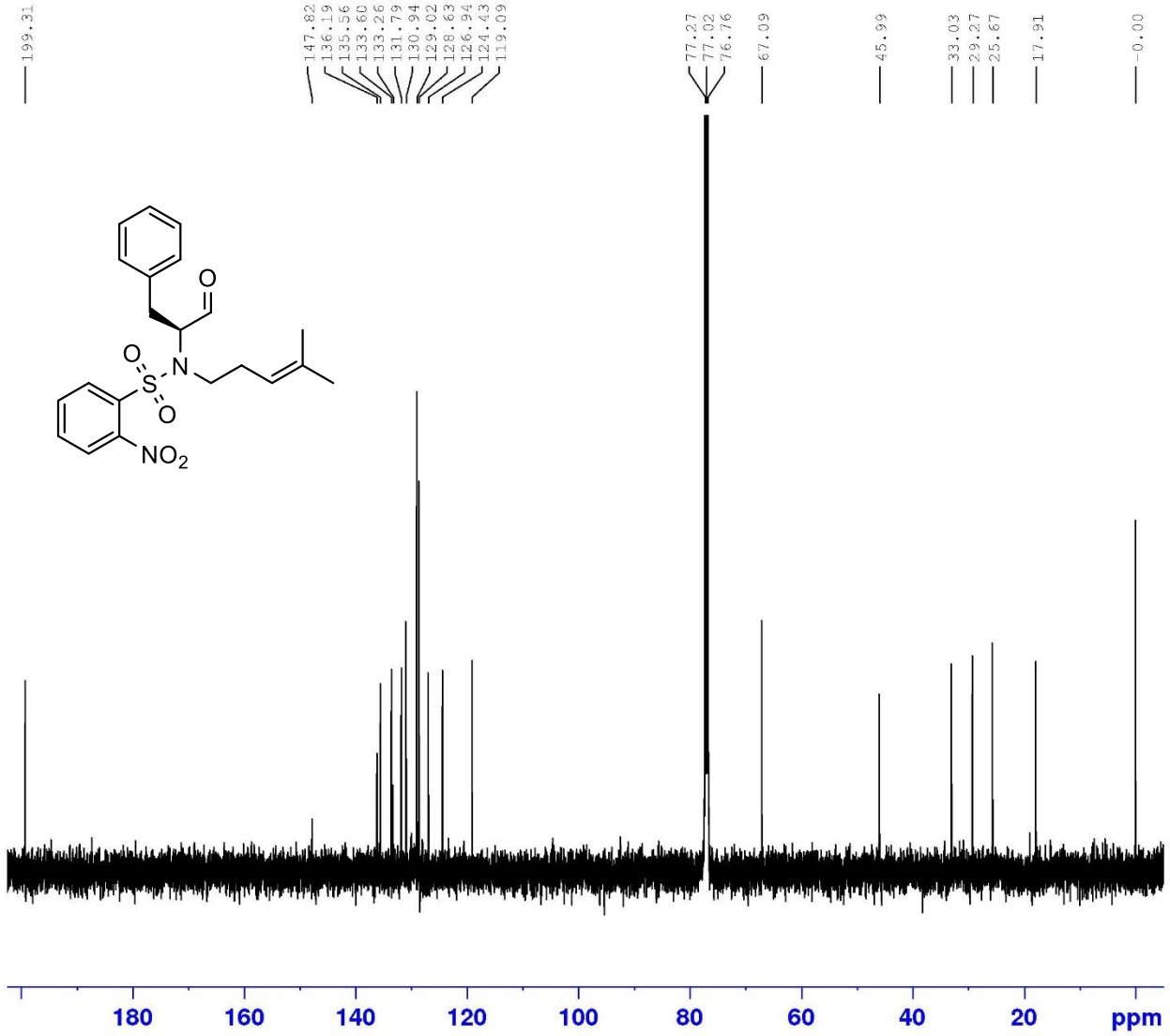
F2 - Processing parameters
SI 32768
SF 100.6052260 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

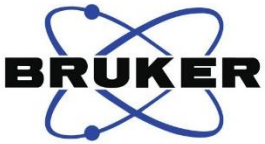
^{13}C-NMR **28f**




```
Current Data Parameters
NAME          May12-2020
EXPNO                 70
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200512
Time               13.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   406
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530155 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

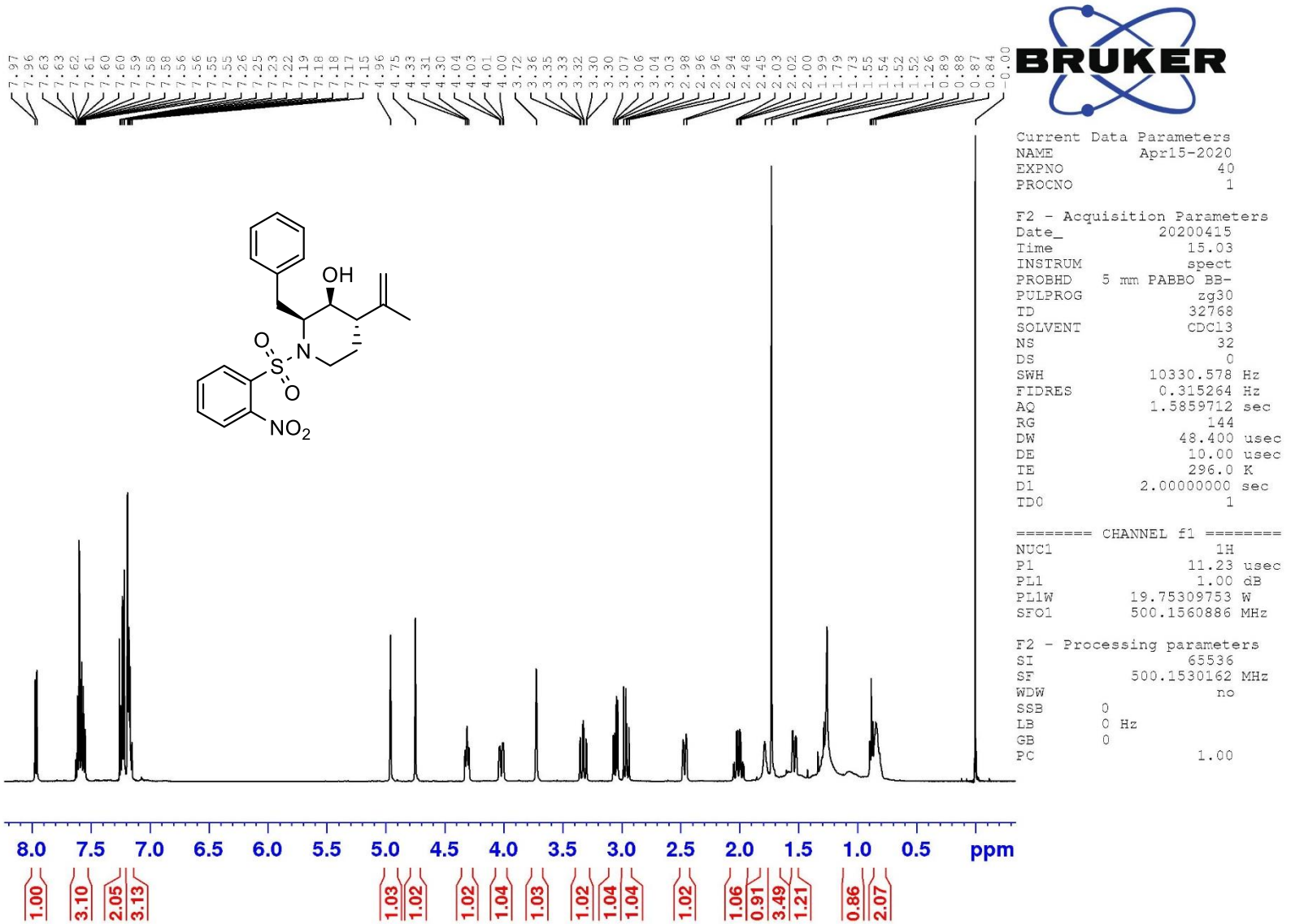
^{1}H-NMR **29f**





```
Current Data Parameters
NAME          May12-2020
EXPNO                 71
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200512
Time               13.12
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635740 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **29f**



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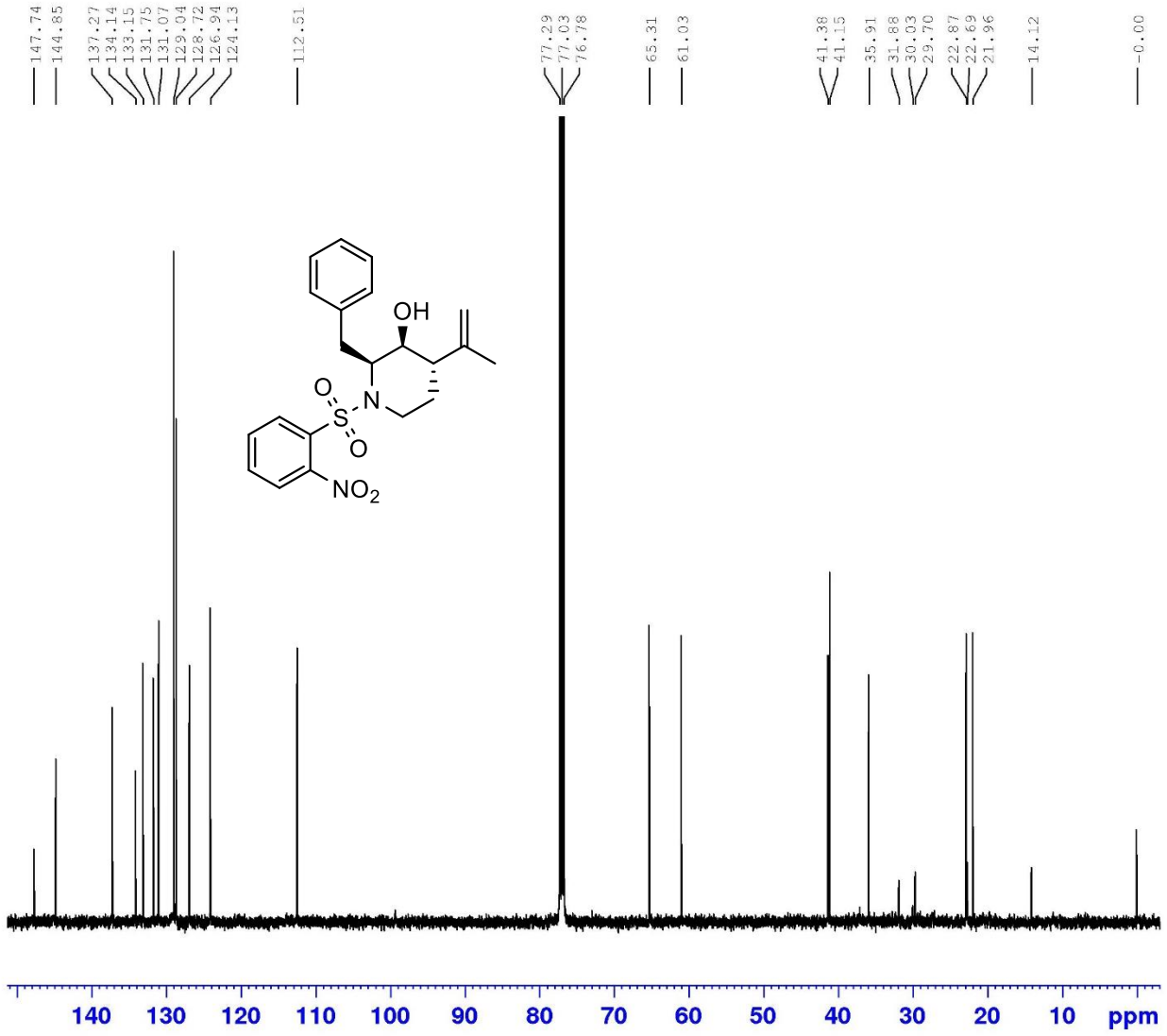
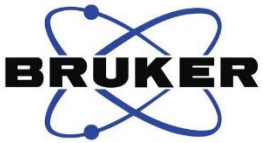
```
Current Data Parameters
NAME          Apr15-2020
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200415
Time               15.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   144
DW               48.400 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 65536
SF          500.1530162 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

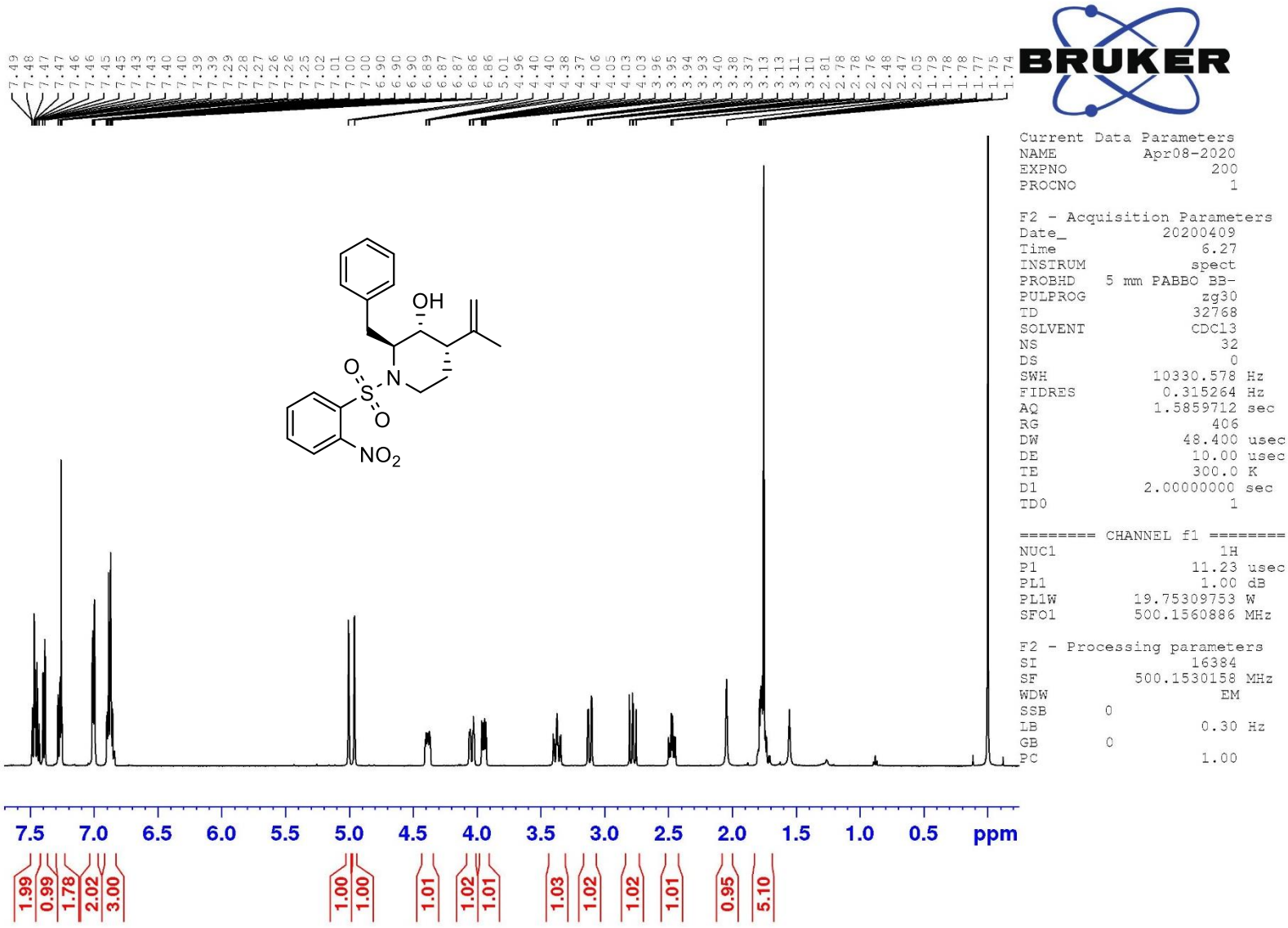
^{1}H-NMR (*S*,*S*)-**30f**






```
Current Data Parameters
NAME          Apr15-2020
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200415
Time               15.57
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                  2580
DW                15.200 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635756 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

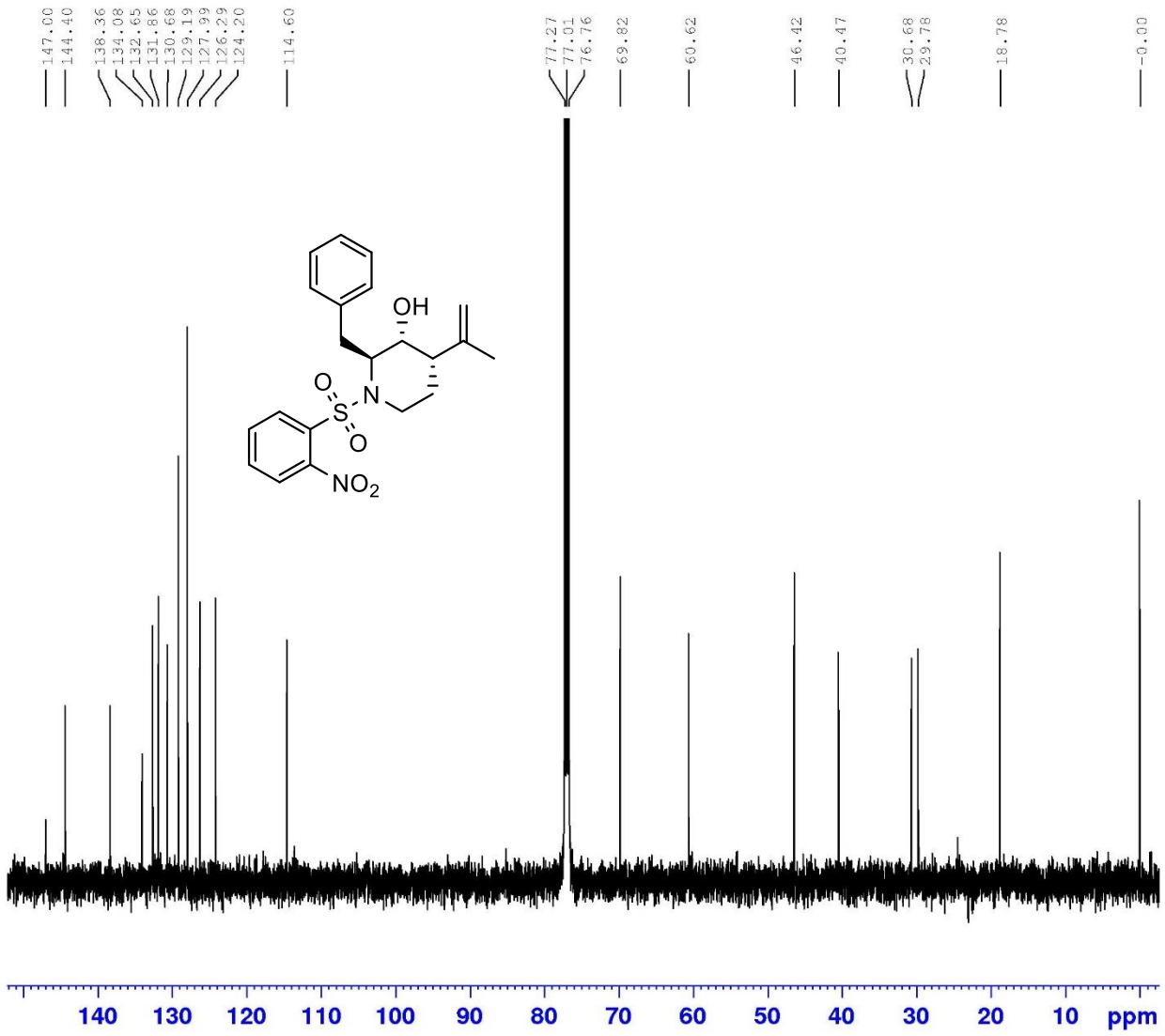
^{13}C-NMR (*S*,*S*)-**30f**

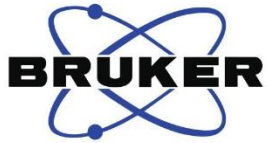


BRUKER

```
Current Data Parameters
NAME          Apr08-2020
EXPNO                200
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200409
Time                6.27
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   406
DW                48.400 usec
DE                10.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530158 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

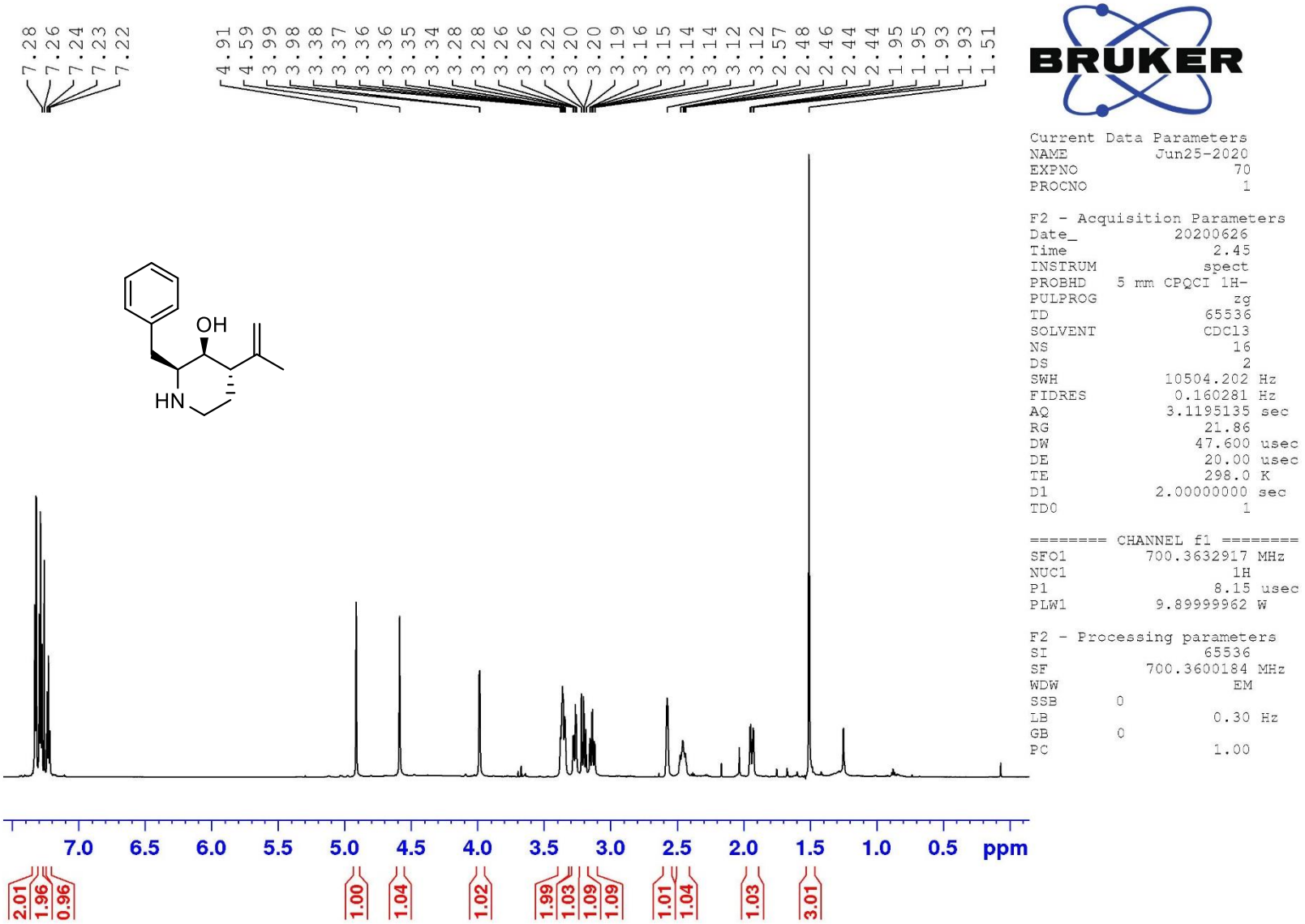
^{1}H-NMR (*R*,*S*)-**30f**






```
Current Data Parameters
NAME          Apr08-2020
EXPNO                201
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200409
Time                7.20
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635746 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

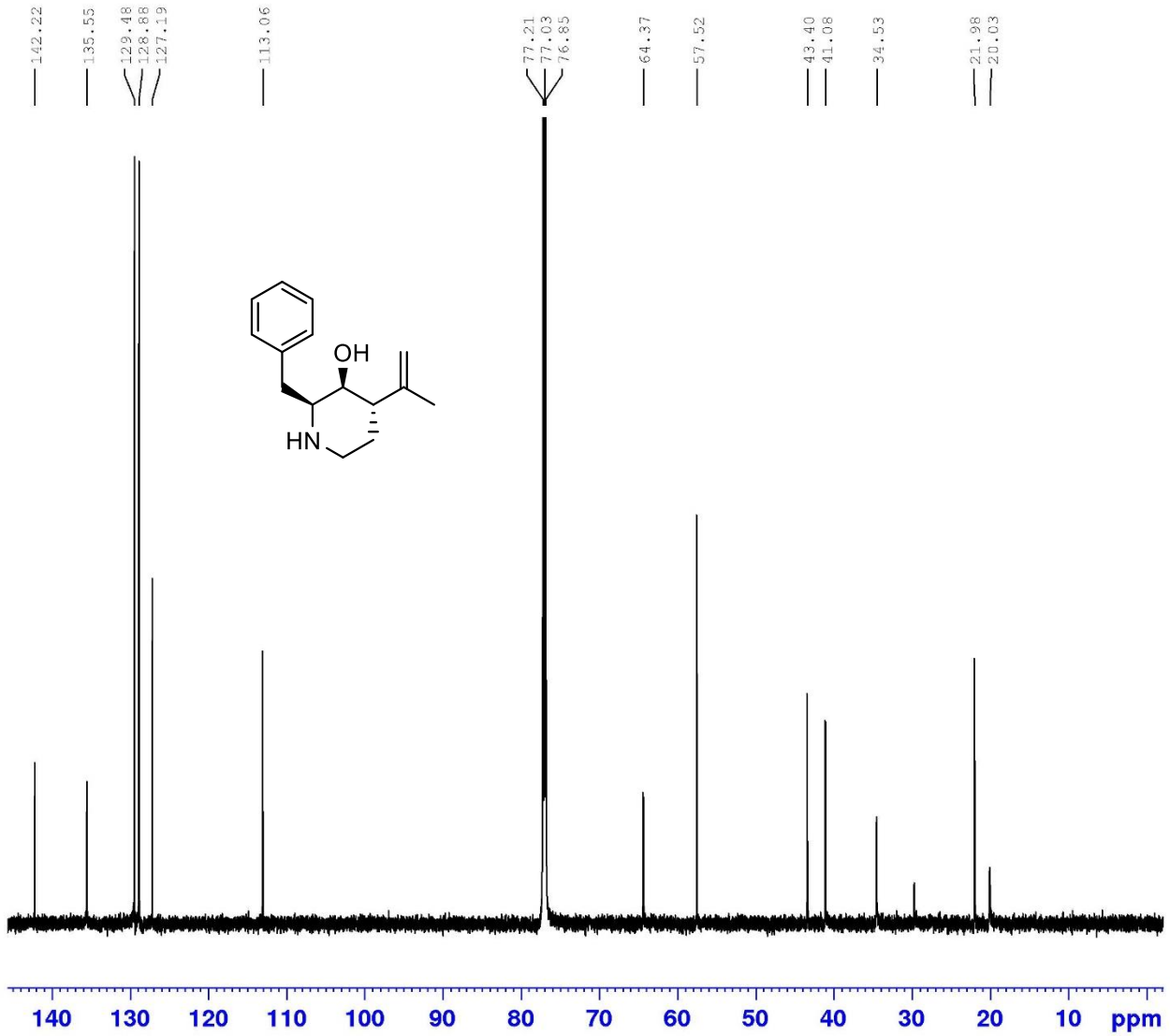
^{13}C-NMR (*R*,*S*)-**30f**



BRUKER

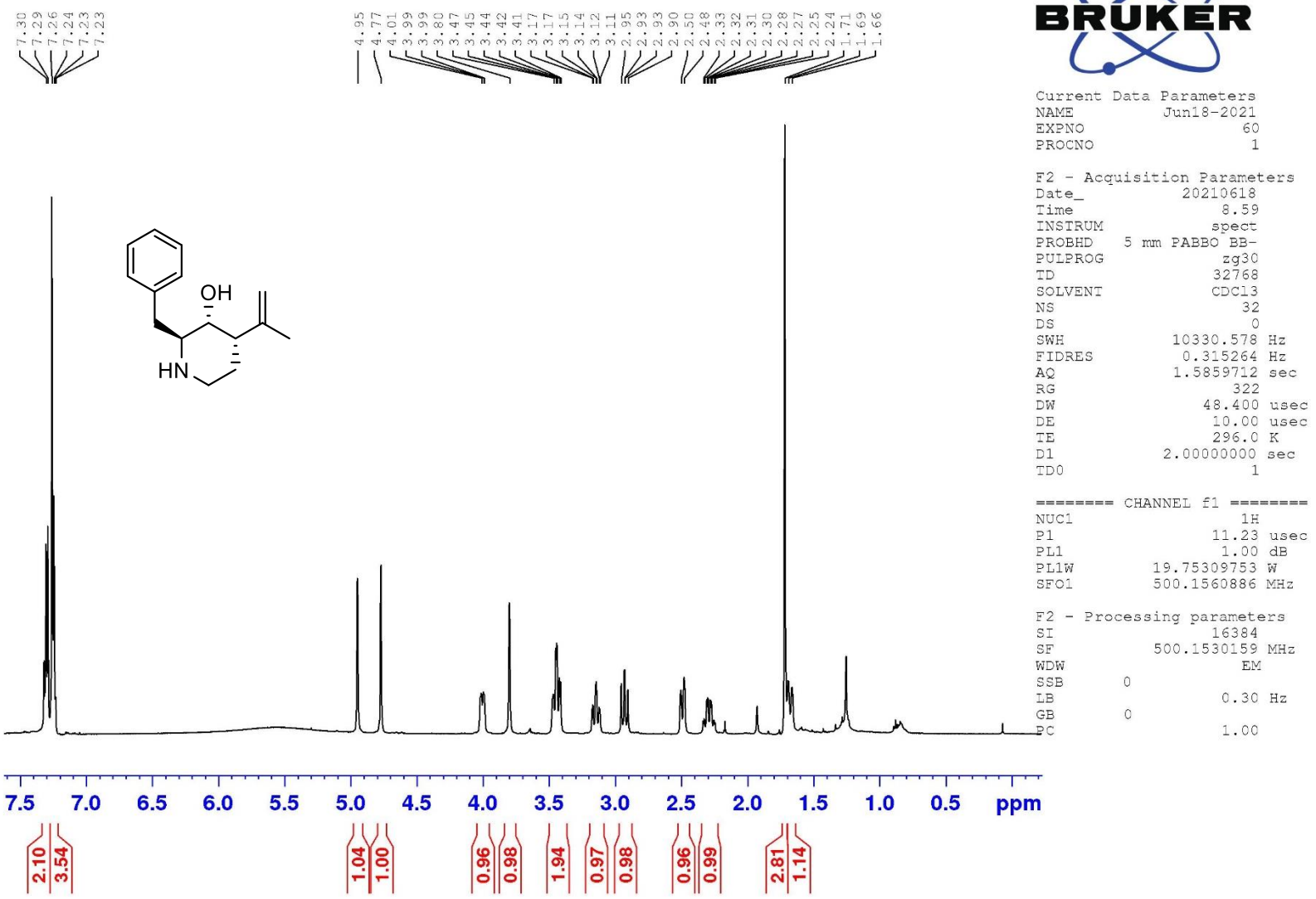
```
Current Data Parameters
NAME          Jun25-2020
EXPNO                 70
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200626
Time                2.45
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG               zg
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10504.202 Hz
FIDRES          0.160281 Hz
AQ             3.1195135 sec
RG                 21.86
DW                47.600 usec
DE                20.00 usec
TE                298.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         700.3632917 MHz
NUC1                  1H
P1                 8.15 usec
PLW1          9.89999962 W

F2 - Processing parameters
SI                 65536
SF            700.3600184 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*S*)-**32f**




```
Current Data Parameters
NAME          Jun25-2020
EXPNO                 71
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200626
Time                3.10
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG           zgpg30
TD                 65356
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            40760.871 Hz
FIDRES          0.623675 Hz
AQ              0.8017003 sec
RG                 182.53
DW                 12.267 usec
DE                 18.00 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
ID0                    1

======== CHANNEL f1 ========
SFO1         176.1232717 MHz
NUC1                 13C
P1                 12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2         700.3628014 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                 131072
SF           176.1056620 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

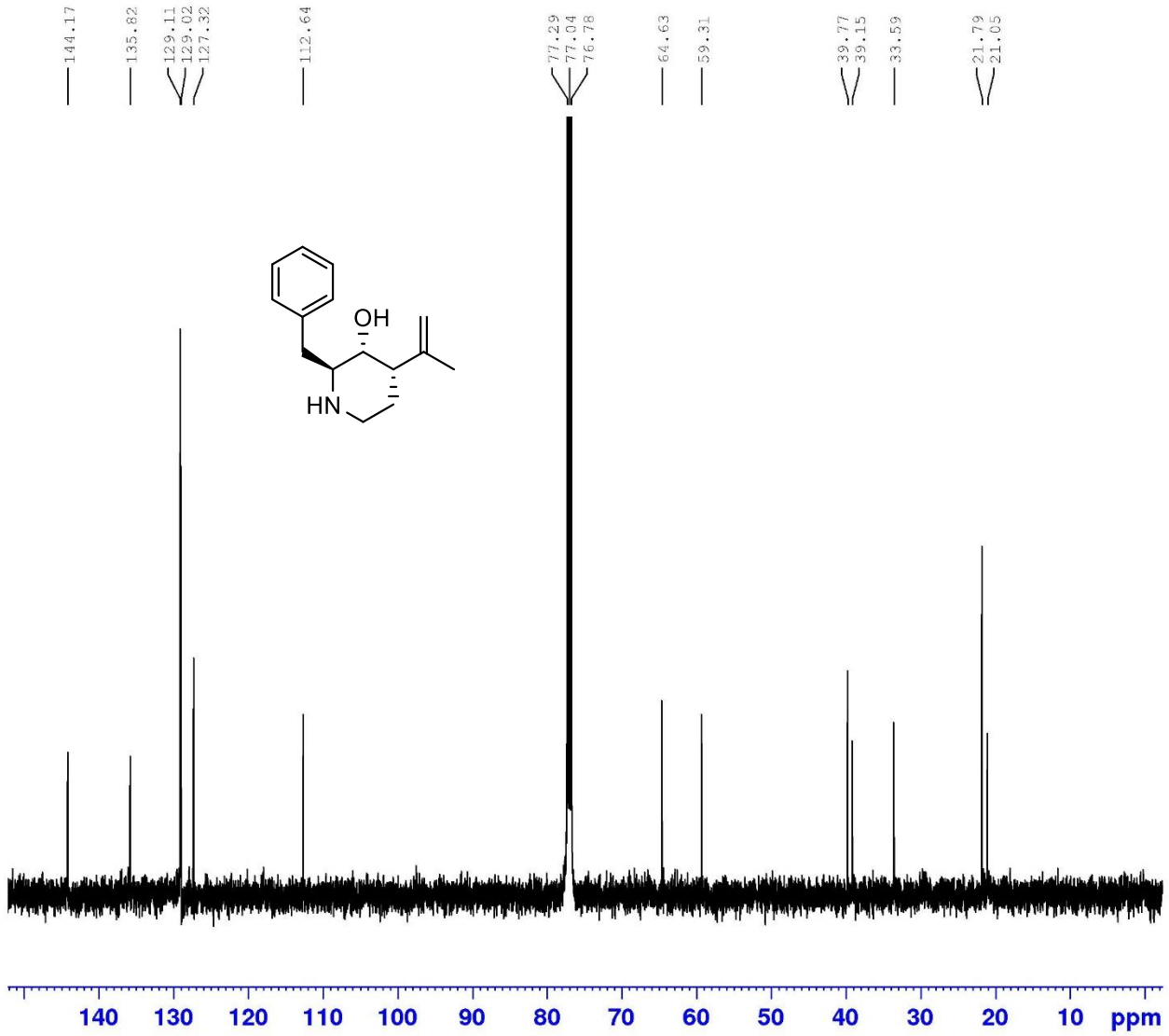
^{13}C-NMR (*S*,*S*)-**32f**



BRUKER

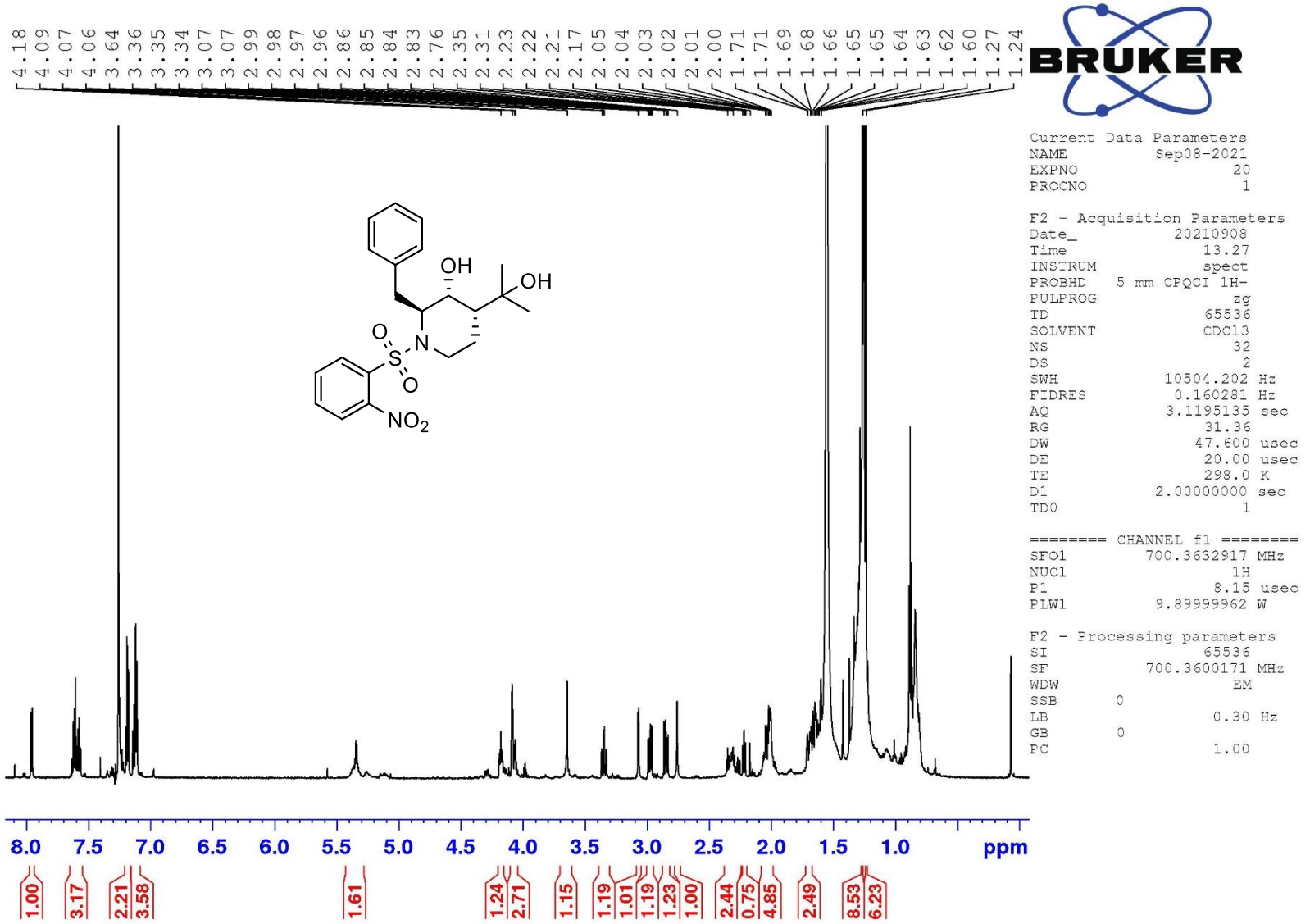
```
Current Data Parameters
NAME          Jun18-2021
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210618
Time                8.59
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530159 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*R*,*S*)-**32f**




```
Current Data Parameters
NAME          Jun18-2021
EXPNO                 61
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210618
Time               9.54
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          32894.738 Hz
FIDRES        0.501934 Hz
AQ           0.9961472 sec
RG                2580
DW              15.200 usec
DE              10.00 usec
TE              296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF         125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

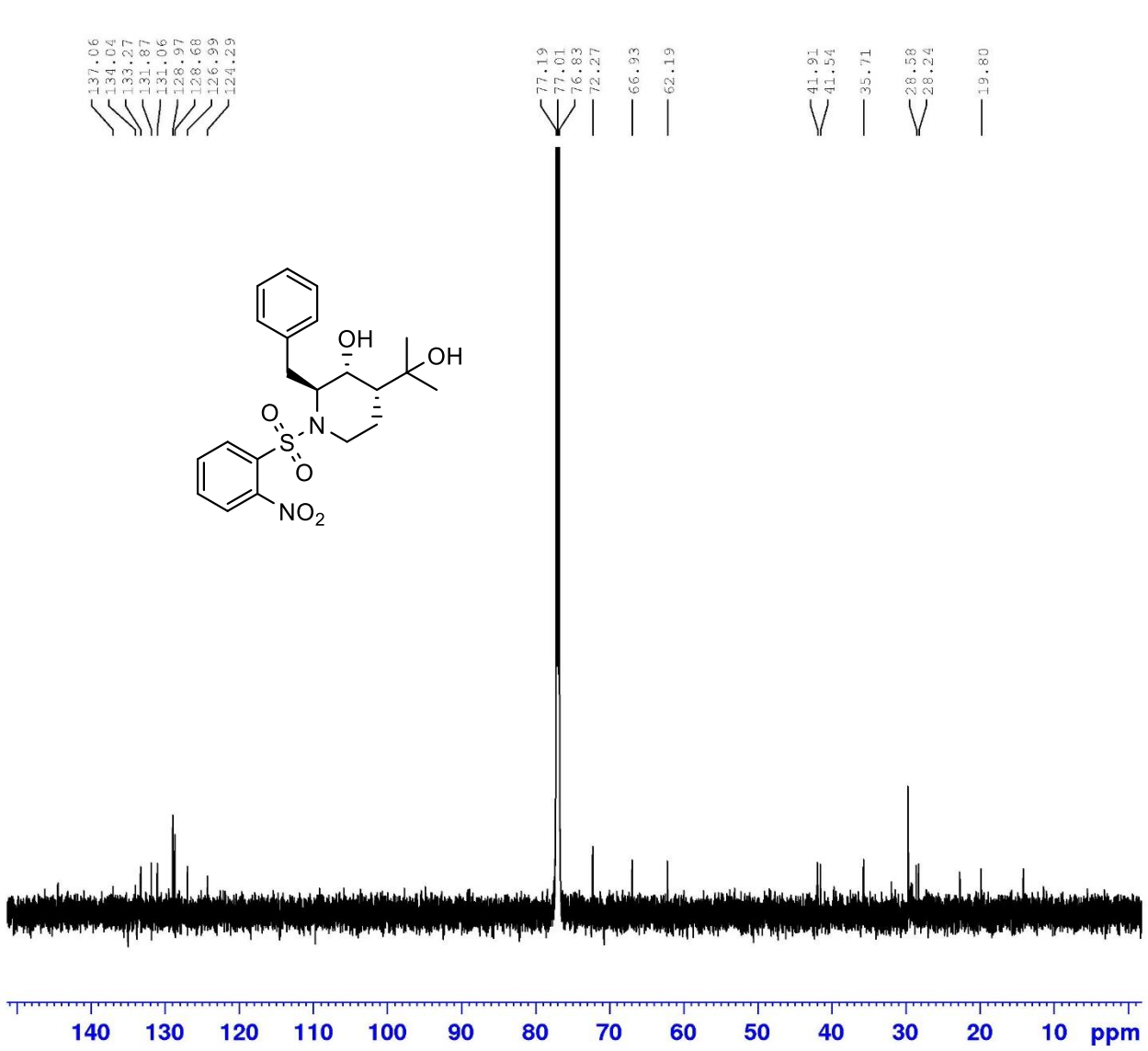
^{13}C-NMR (*R*,*S*)-**32f**



BRUKER

```
Current Data Parameters
NAME          Sep08-2021
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210908
Time               13.27
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG                zg
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH            10504.202 Hz
FIDRES          0.160281 Hz
AQ             3.1195135 sec
RG                 31.36
DW                47.600 usec
DE                20.00 usec
TE                298.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         700.3632917 MHz
NUC1                   1H
P1                  8.15 usec
PLW1          9.89999962 W

F2 - Processing parameters
SI                 65536
SF           700.3600171 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

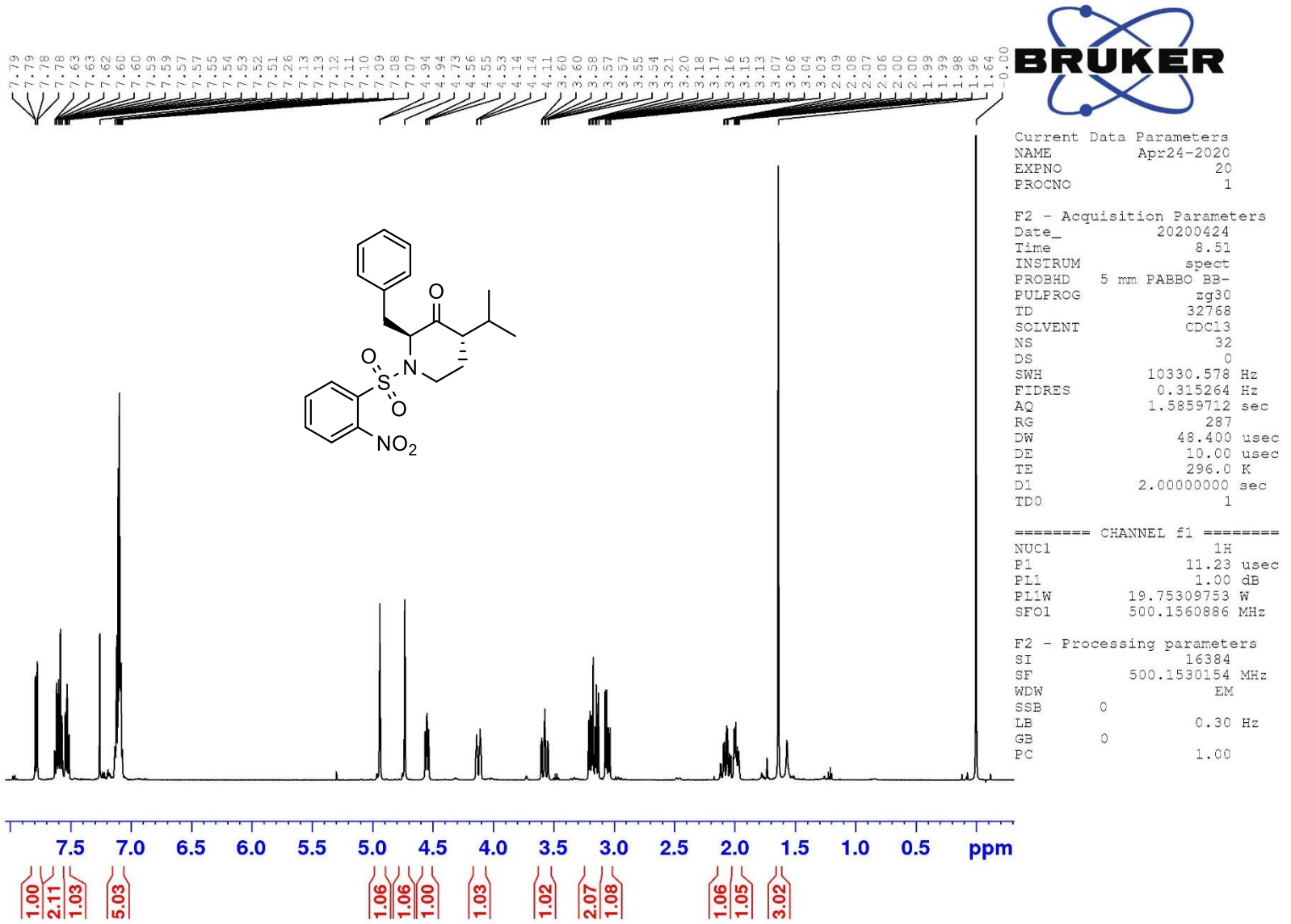
^{1}H-NMR (*R*,*R*)-**31f**



BRUKER

```
Current Data Parameters
NAME          Sep08-2021
EXPNO                 22
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210909
Time                3.26
INSTRUM            spect
PROBHD    5 mm CPQCI 1H-
PULPROG           zgpg30
TD                 65356
SOLVENT            CDCl3
NS                  6144
DS                     4
SWH            40760.871 Hz
FIDRES          0.623675 Hz
AQ              0.8017003 sec
RG                 182.53
DW                 12.267 usec
DE                 18.00 usec
TE                 298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         176.1232717 MHz
NUC1                  13C
P1                 12.00 usec
PLW1        105.00000000 W

======== CHANNEL f2 ========
SFO2         700.3628014 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              65.00 usec
PLW2           9.89999962 W
PLW12          0.15564001 W
PLW13          0.07837200 W

F2 - Processing parameters
SI                  131072
SF           176.1056620 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

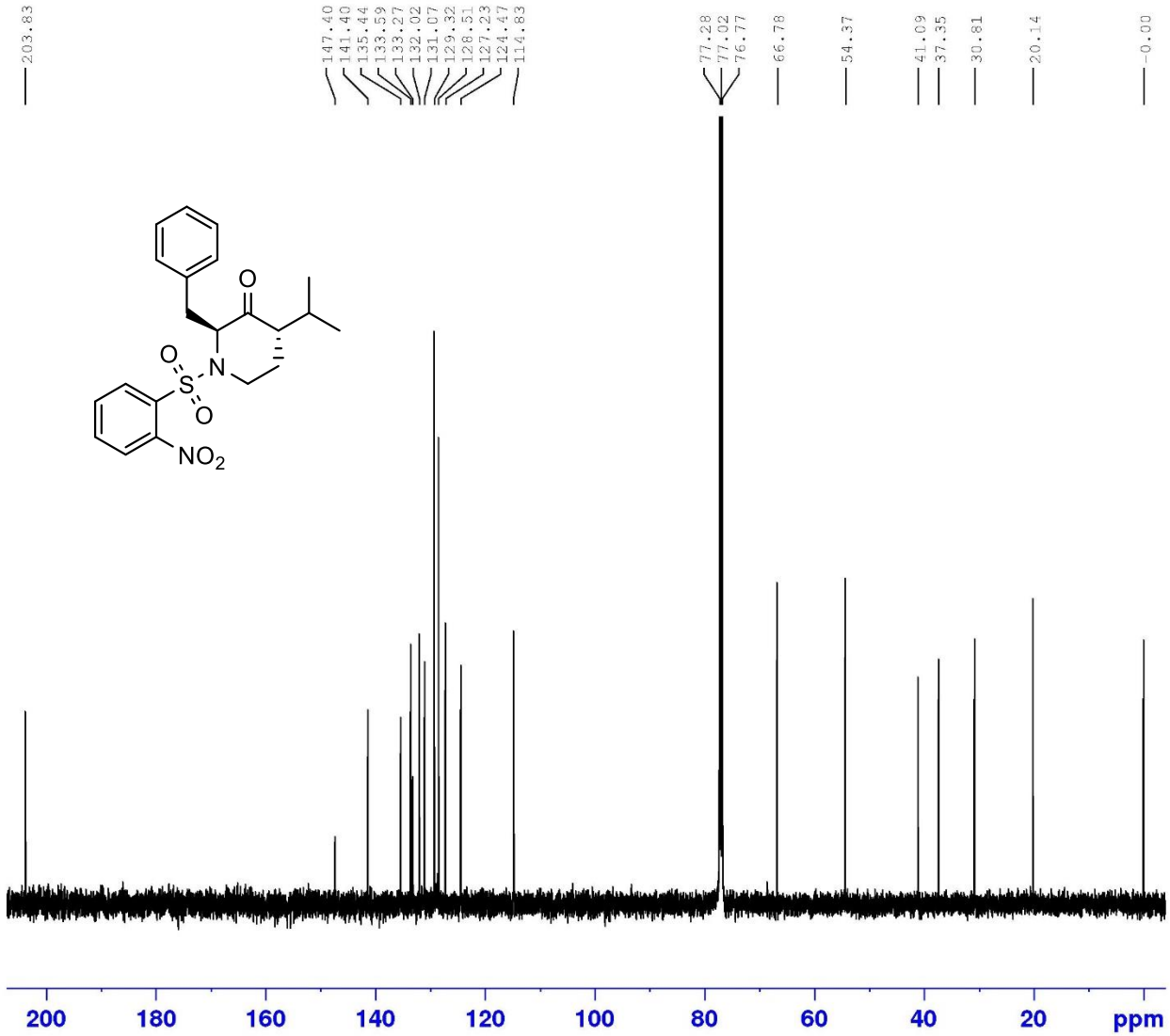
^{13}C-NMR (*R*,*R*)-**31f**



BRUKER

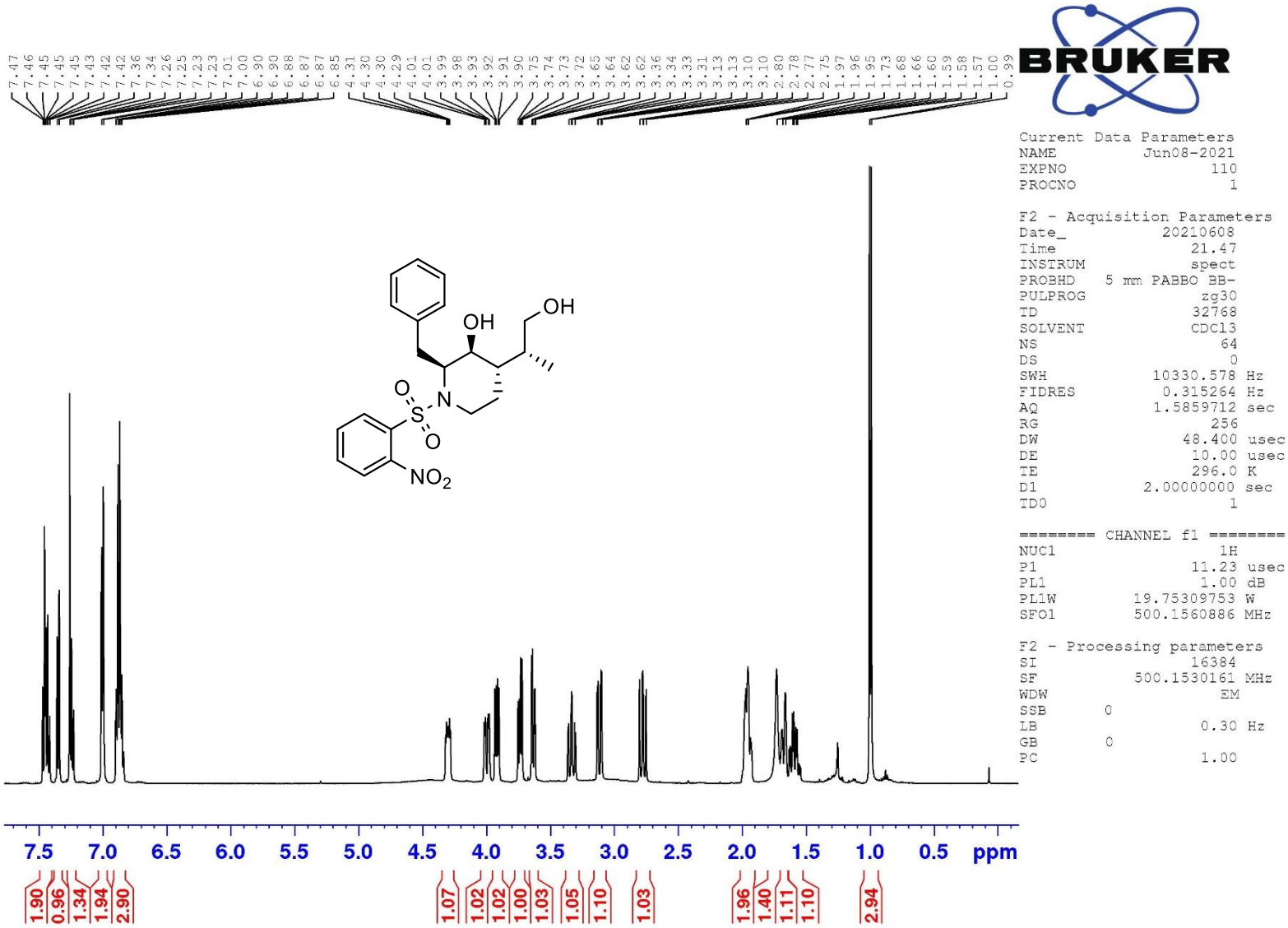
```
Current Data Parameters
NAME          Apr24-2020
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200424
Time                8.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   287
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530154 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*)-**S2f**




```
Current Data Parameters
NAME          Apr24-2020
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200424
Time                9.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2300
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635748 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

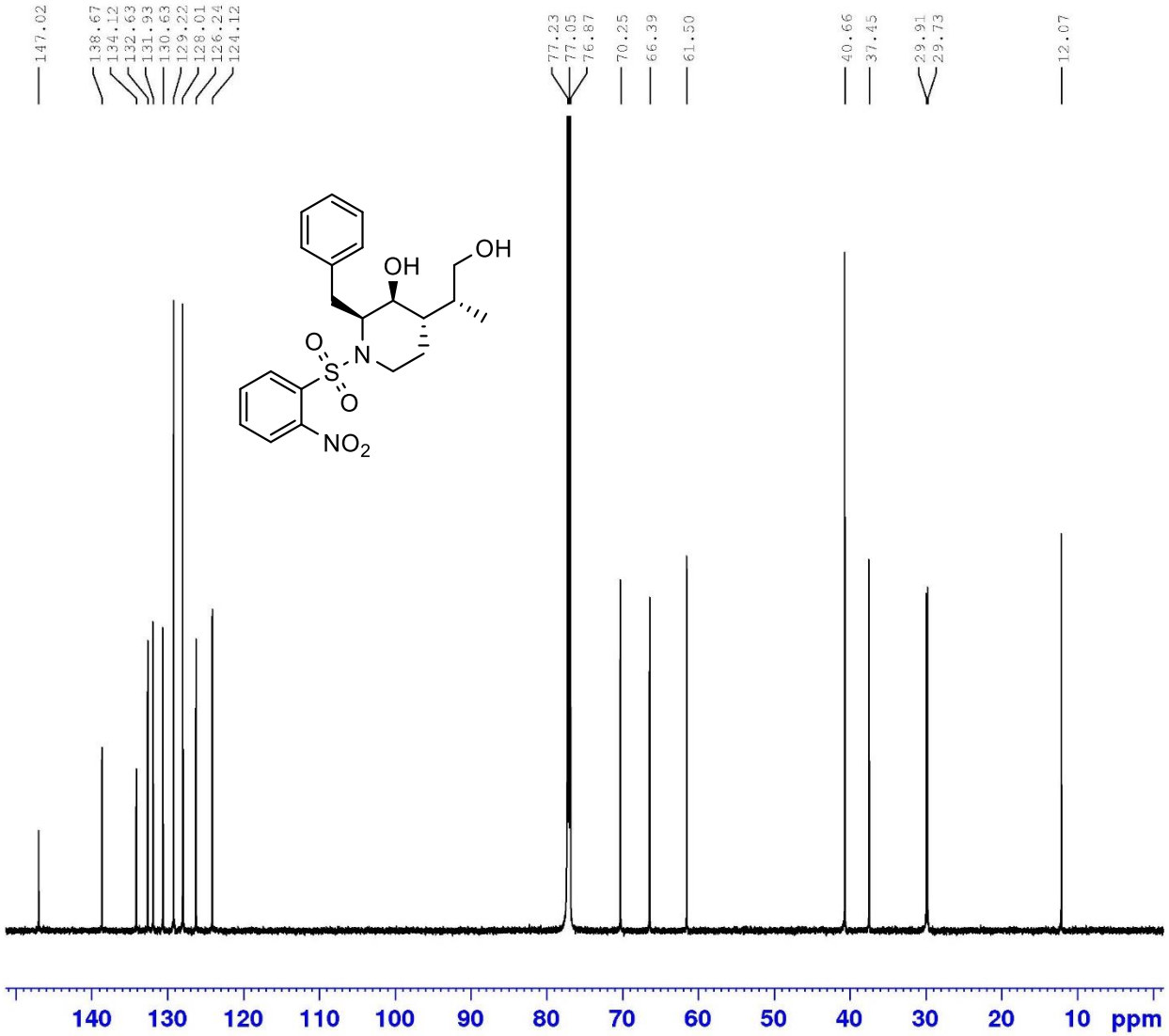
^{13}C-NMR (*S*)-**S2f**



BRUKER

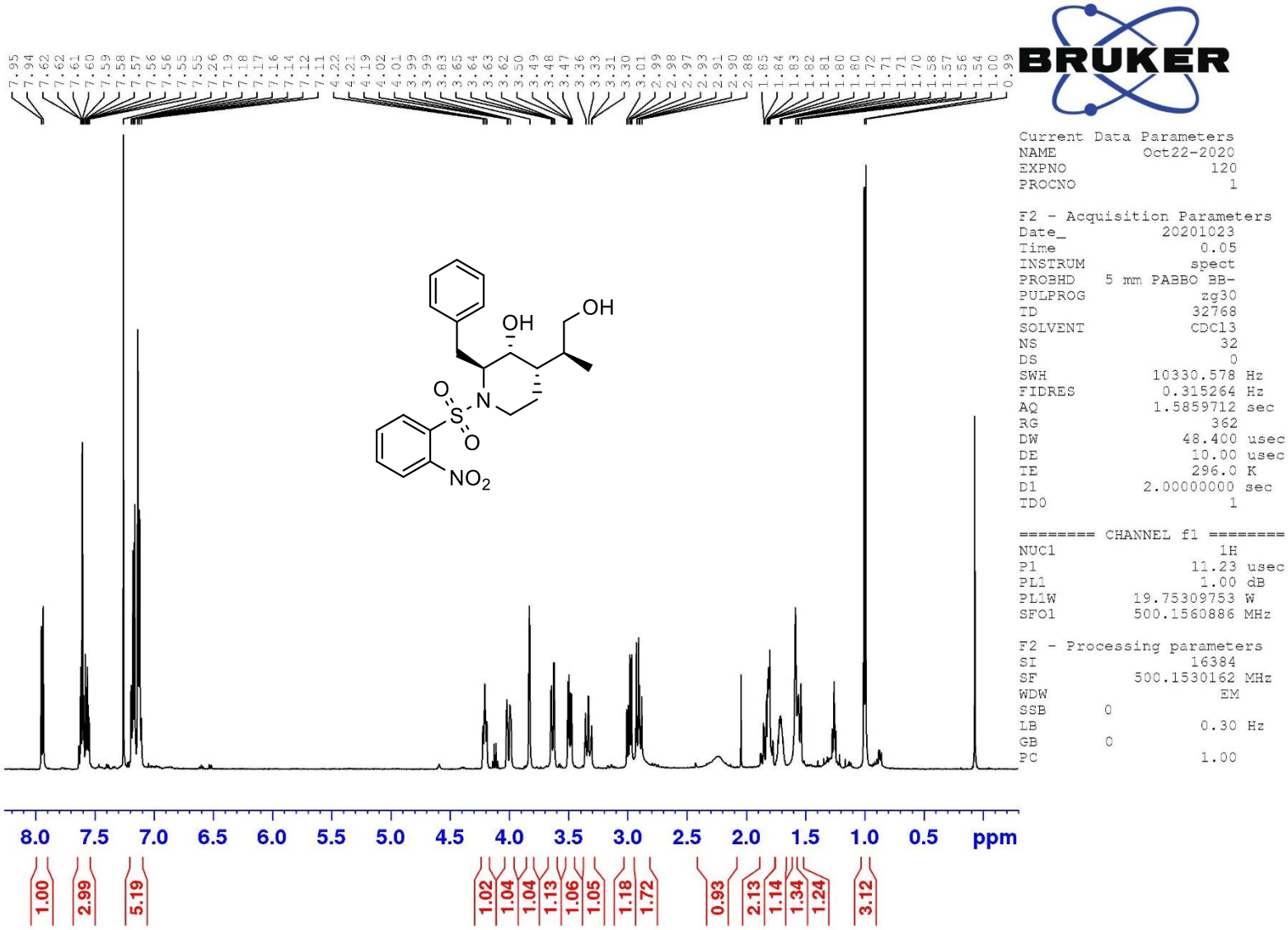
```
Current Data Parameters
NAME          Jun08-2021
EXPNO                110
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210608
Time               21.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   256
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530161 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*S*,*R*)-**34f**



^{13}C-NMR (*S*,*S*,*R*)-**34f**

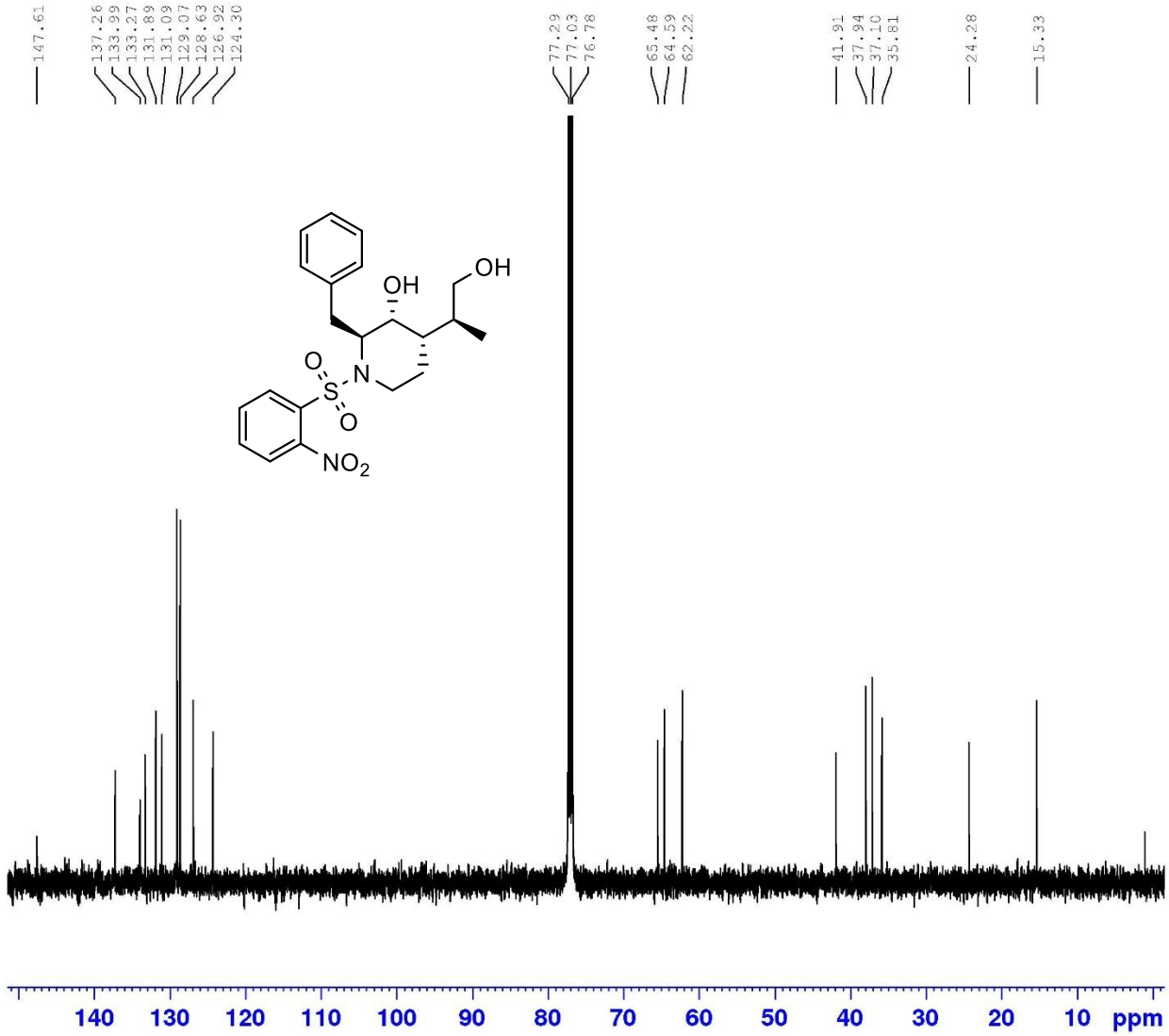
```
Current Data Parameters
NAME          Jun04-2021
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210604
Time               10.28
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



BRUKER

```
Current Data Parameters
NAME          Oct22-2020
EXPNO                120
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201023
Time                0.05
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   362
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530162 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

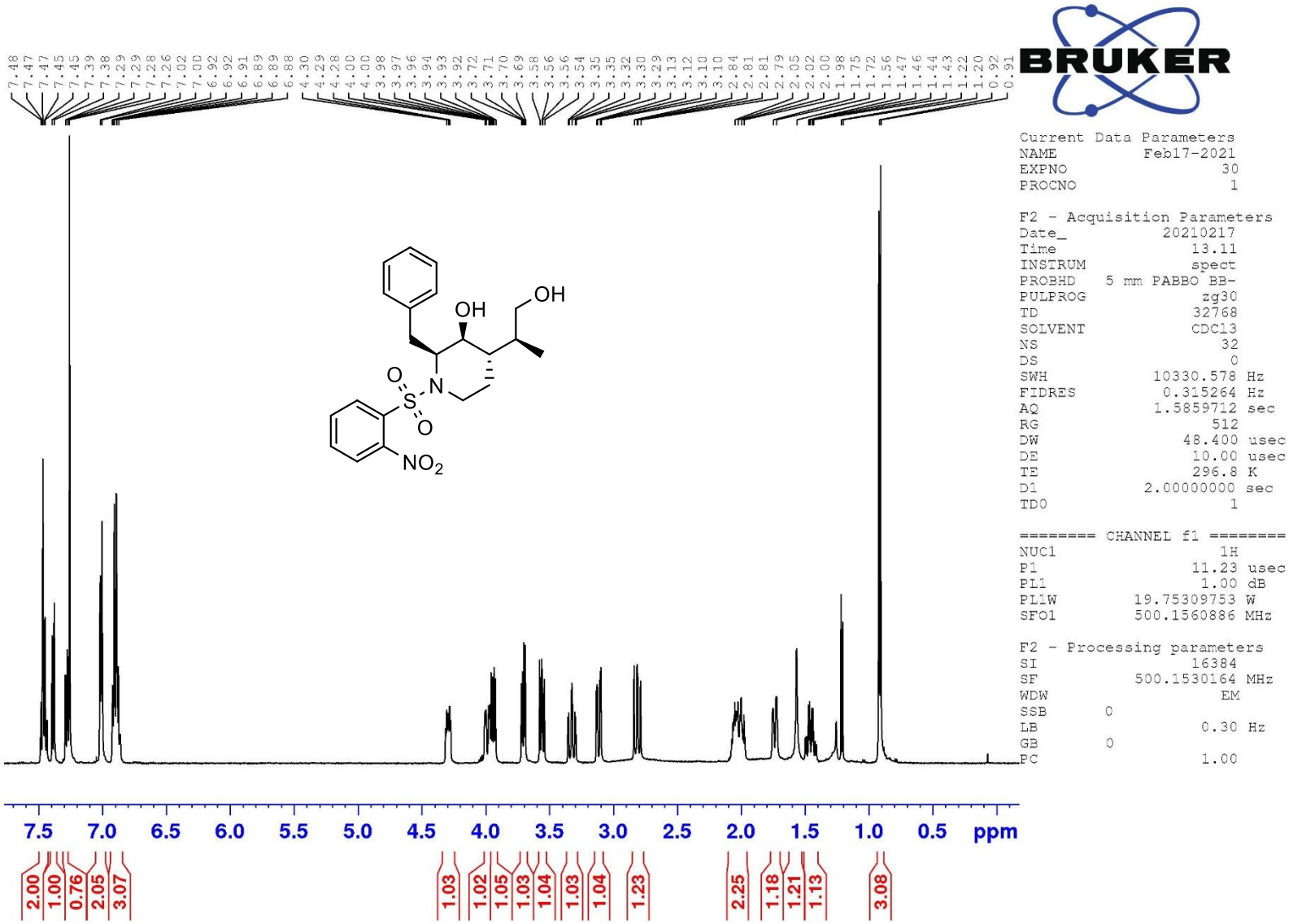
^{1}H-NMR (*R*,*S*,*S*)-**34f**



^{13}C-NMR (*R*,*S*,*S*)-**34f**

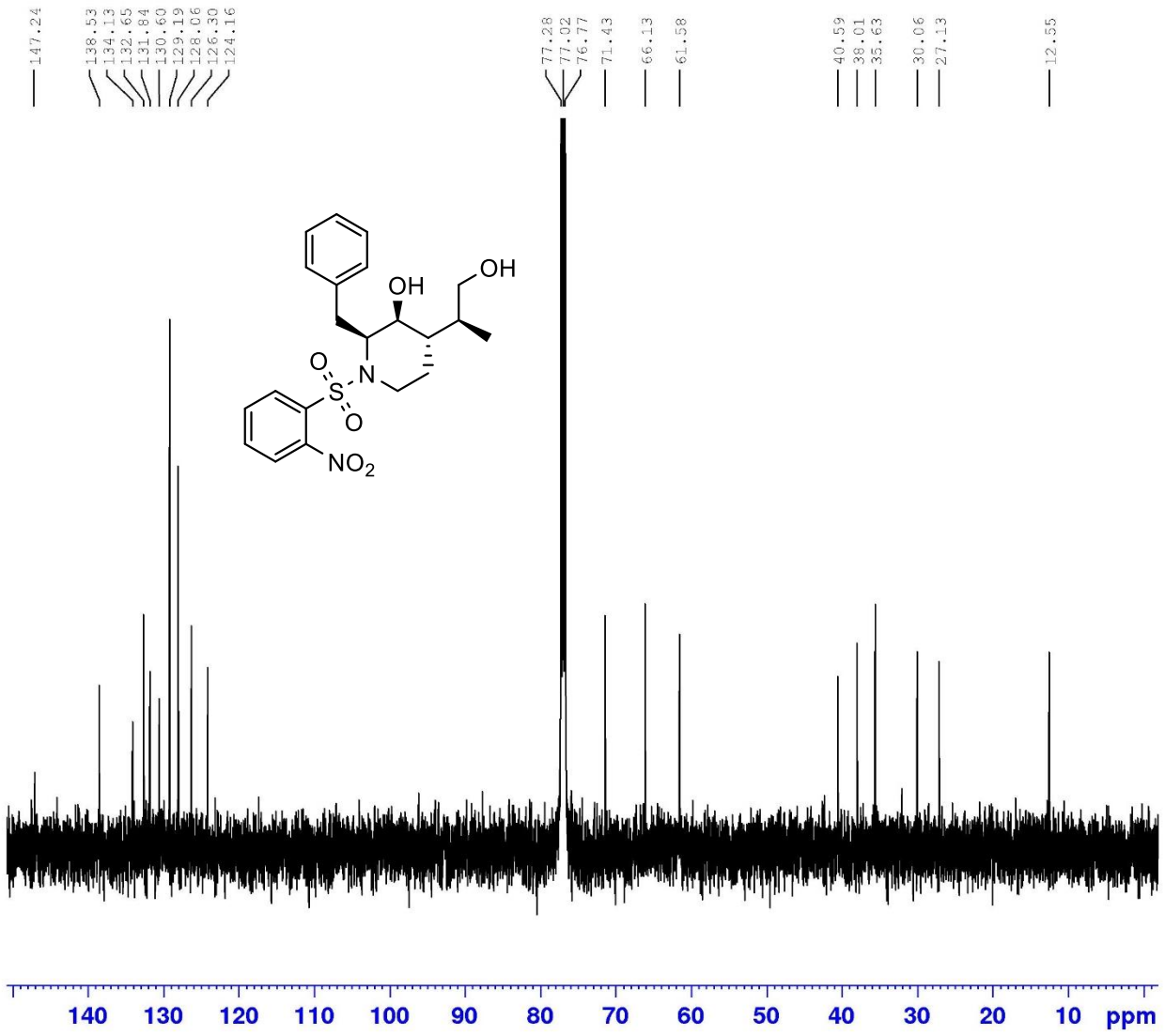
BRUKER

```
Current Data Parameters
NAME          Oct22-2020
EXPNO                121
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201023
Time                0.59
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF         125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME          Feb17-2021
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210217
Time               13.11
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   512
DW                48.400 usec
DE                10.00 usec
TE                296.8 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530164 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

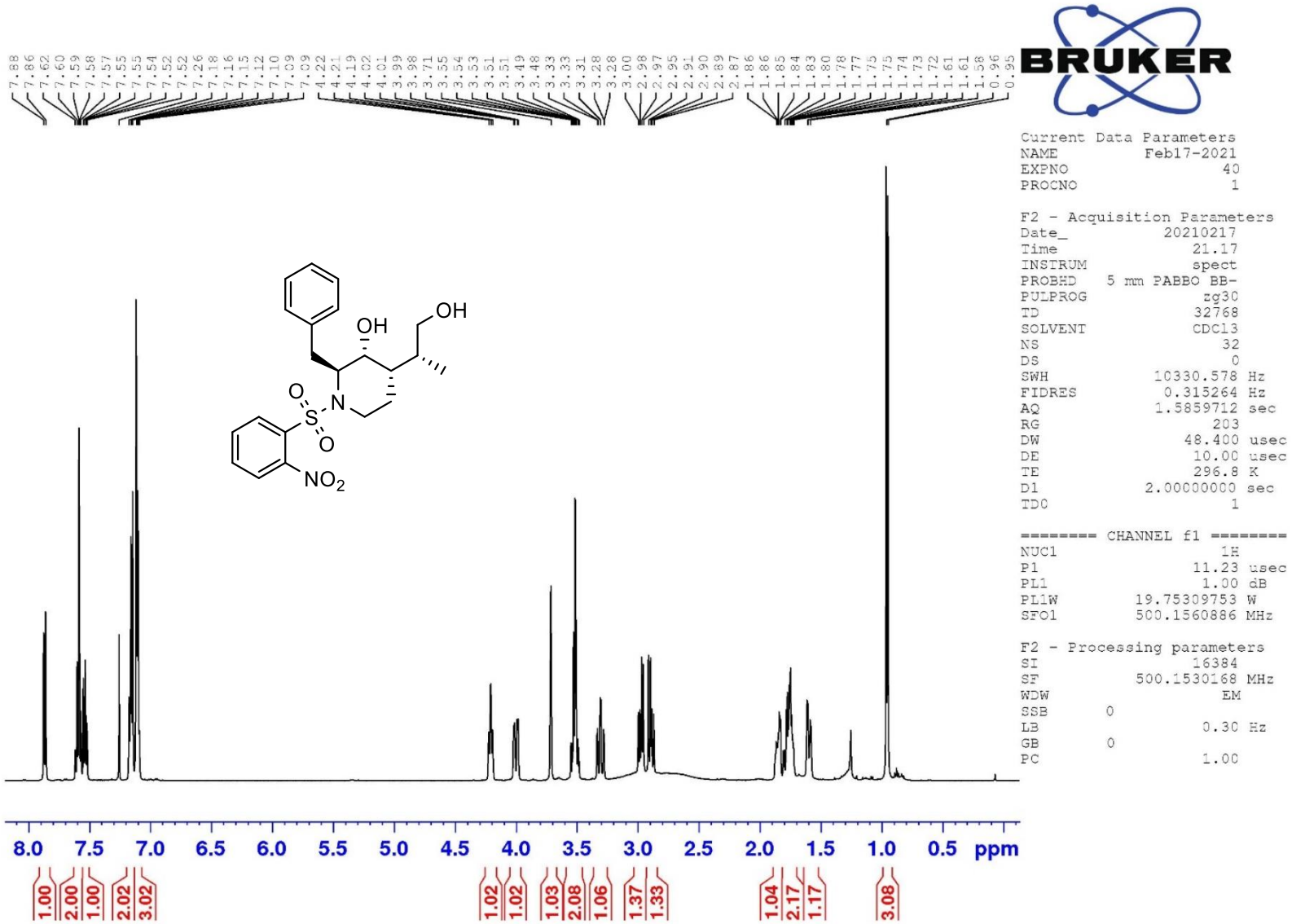
^{1}H-NMR (*S*,*S*,*S*)-**34f**



BRUKER

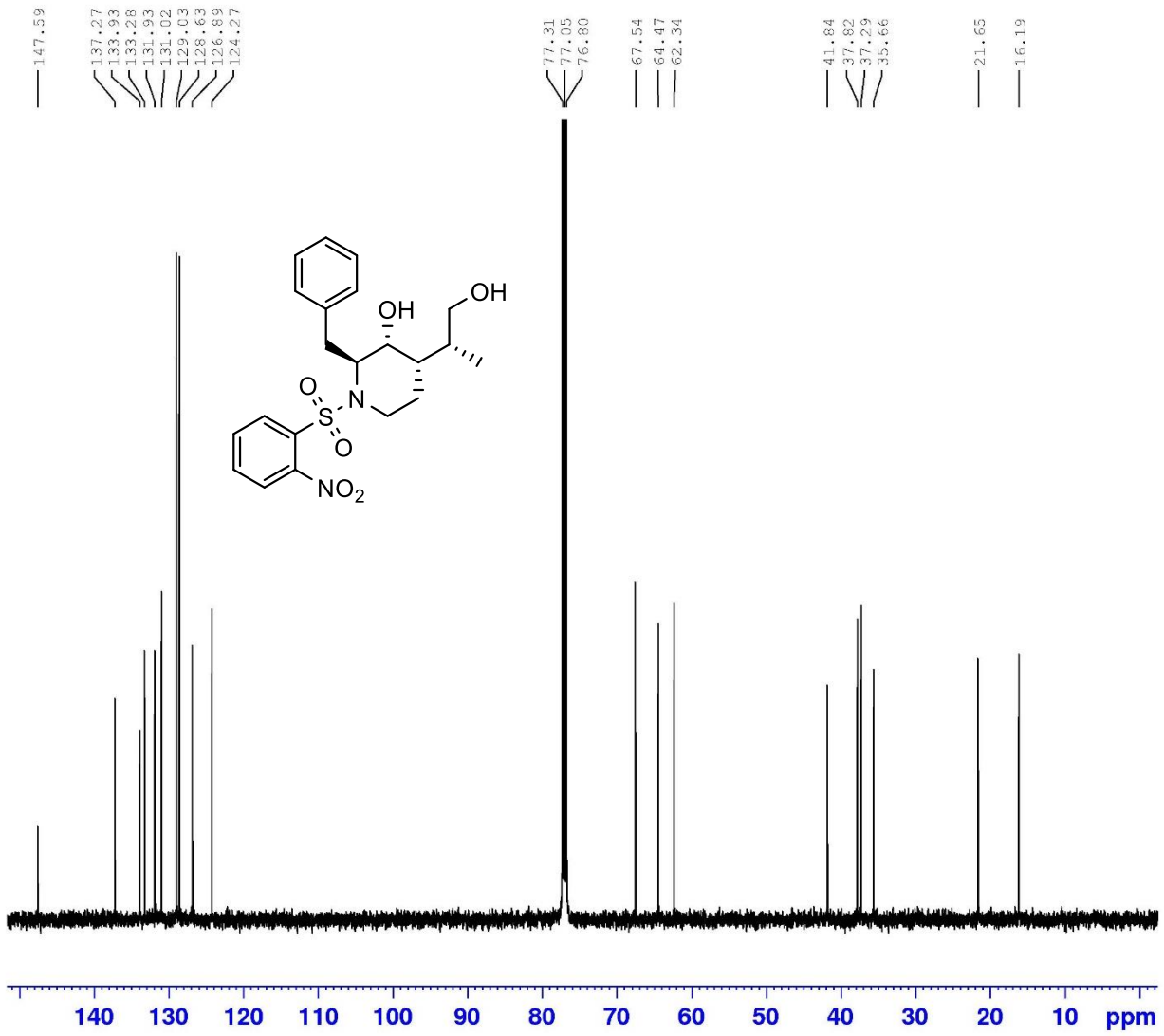
```
Current Data Parameters
NAME          Feb17-2021
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210217
Time               14.42
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

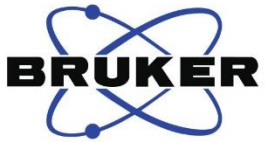
^{13}C-NMR (*S*,*S*,*S*)-**34f**




```
Current Data Parameters
NAME          Feb17-2021
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210217
Time              21.17
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                  203
DW                48.400 usec
DE                10.00 usec
TE                296.8 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                16384
SF          500.1530168 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

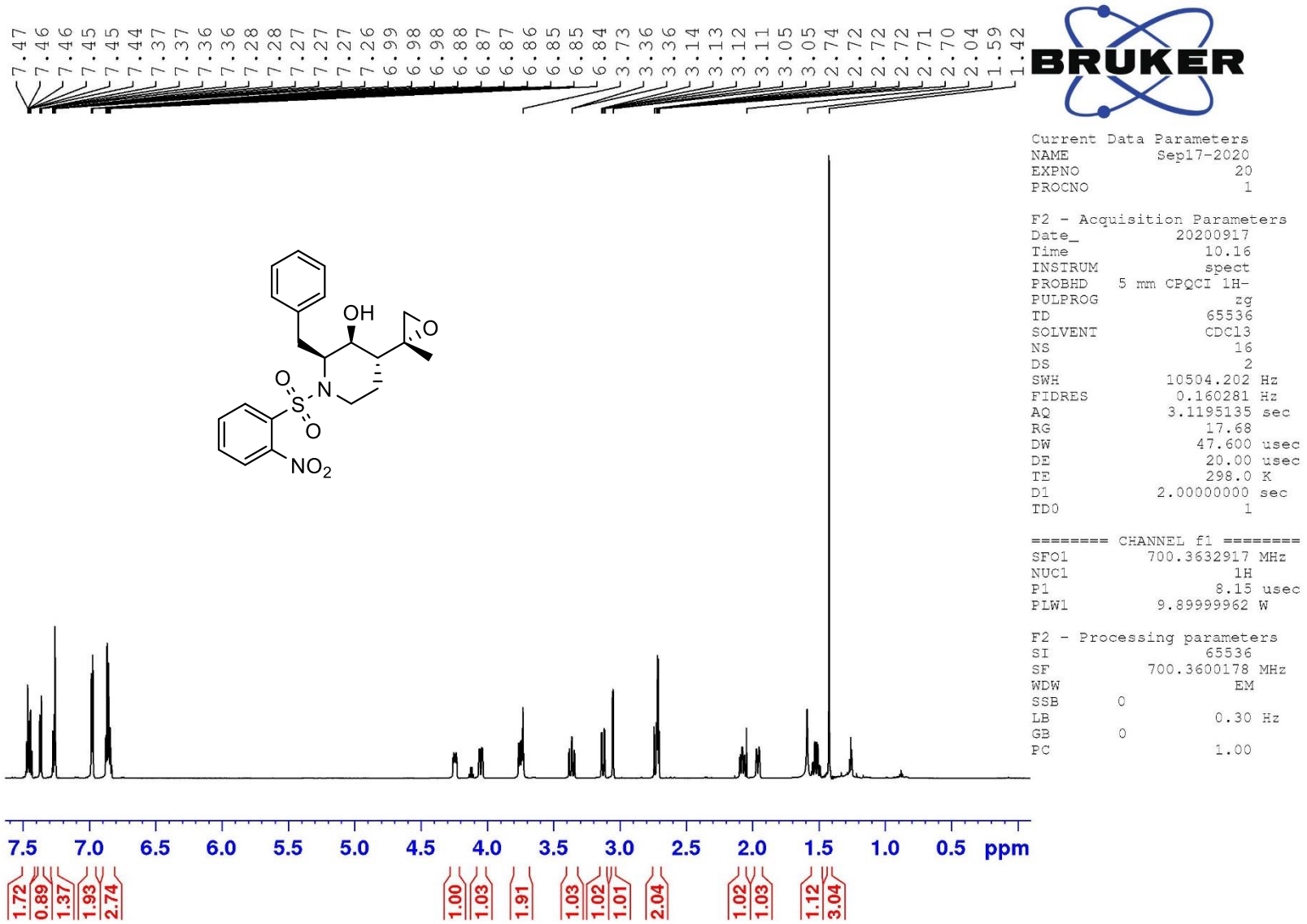
^{1}H-NMR (*R*,*S*,*R*)-**34f**





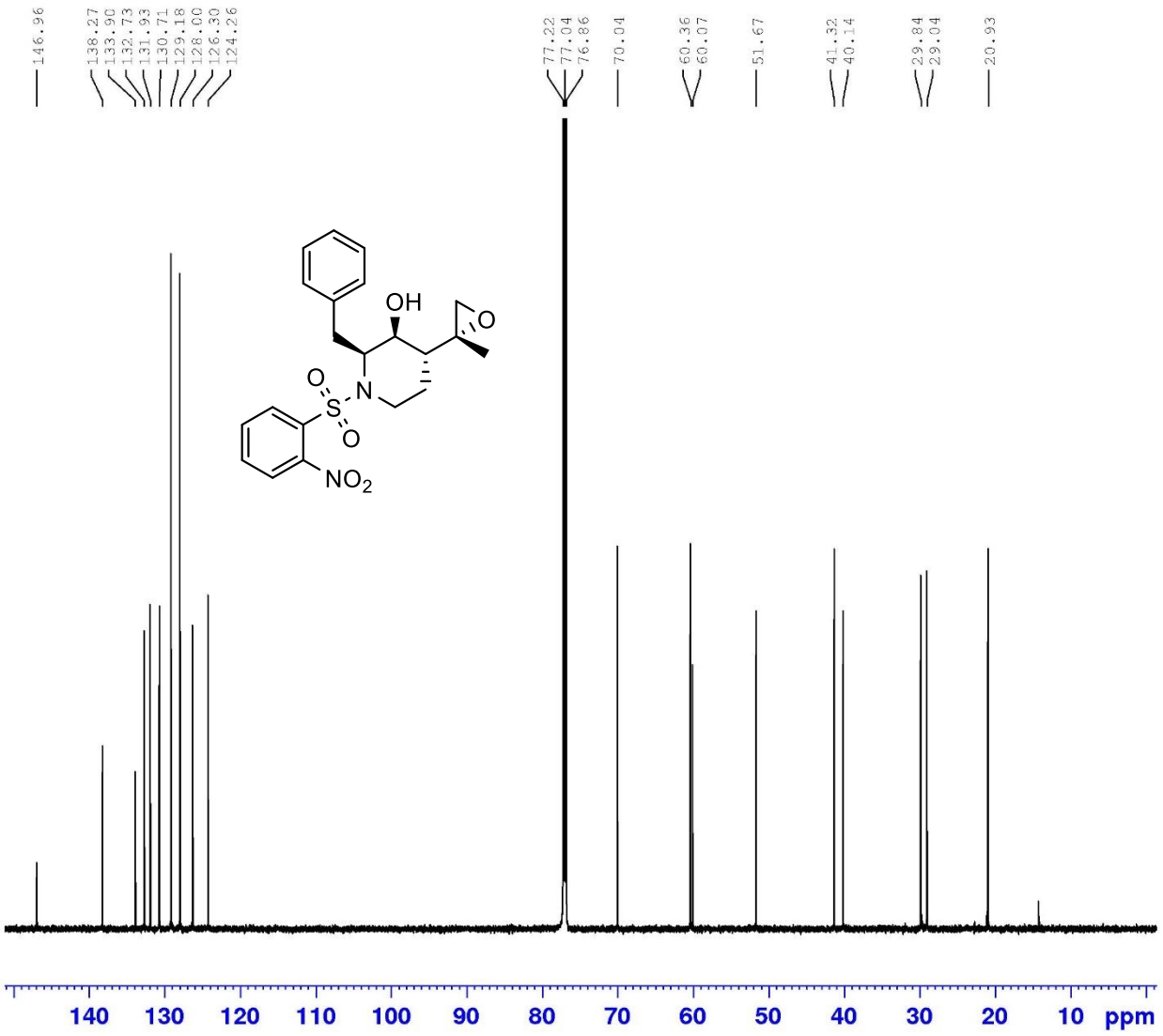

```
Current Data Parameters
NAME          Feb17-2021
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210217
Time               22.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.20 usec
PL1                  1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*R*,*S*,*R*)-**34f**



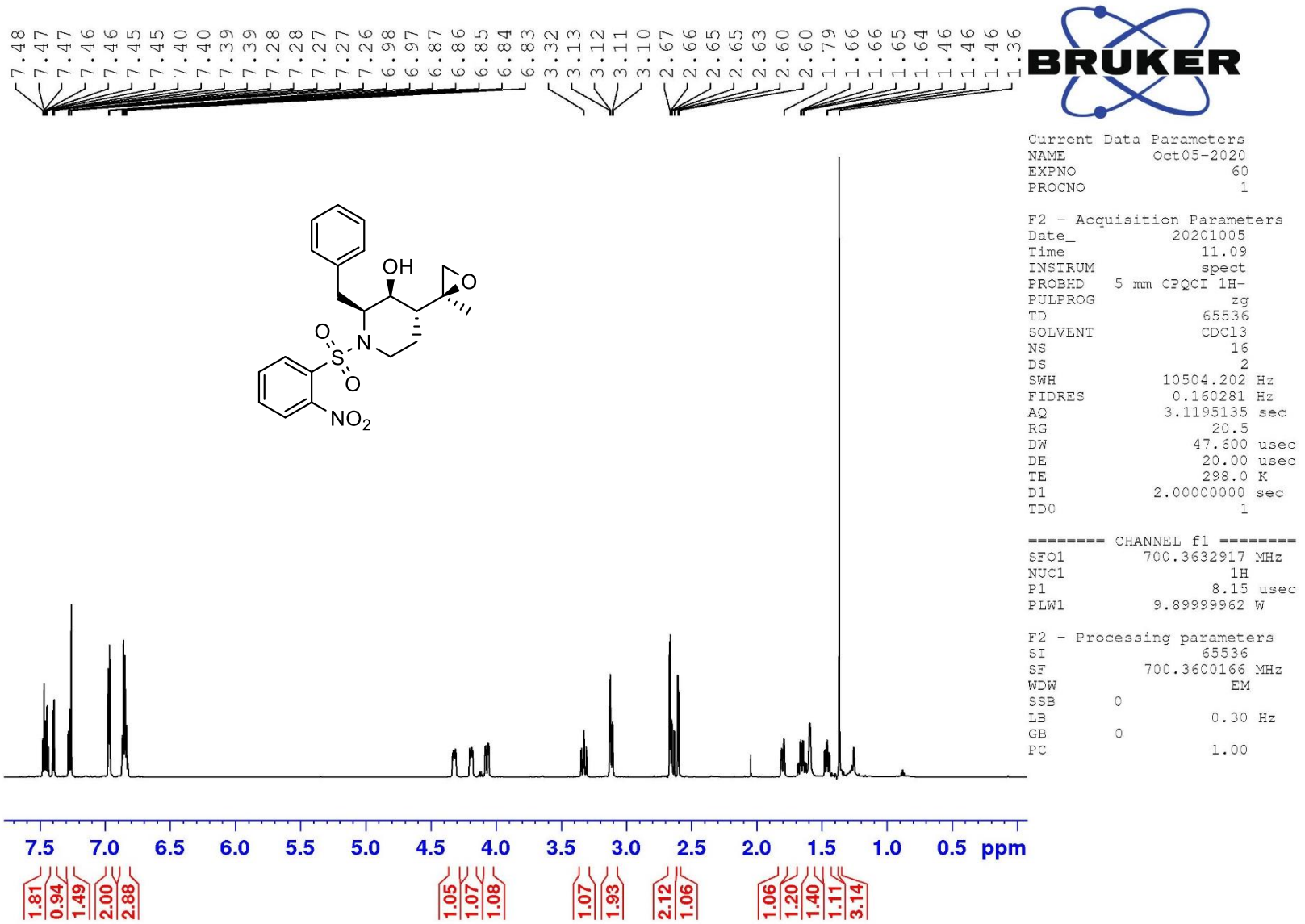

```
Current Data Parameters
NAME          Sep17-2020
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200917
Time               10.16
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG               zg
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10504.202 Hz
FIDRES         0.160281 Hz
AQ            3.1195135 sec
RG                 17.68
DW                47.600 usec
DE                20.00 usec
TE                298.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         700.3632917 MHz
NUC1                  1H
P1                 8.15 usec
PLW1          9.89999962 W

F2 - Processing parameters
SI                 65536
SF           700.3600178 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*R*,*R*)-**35f**



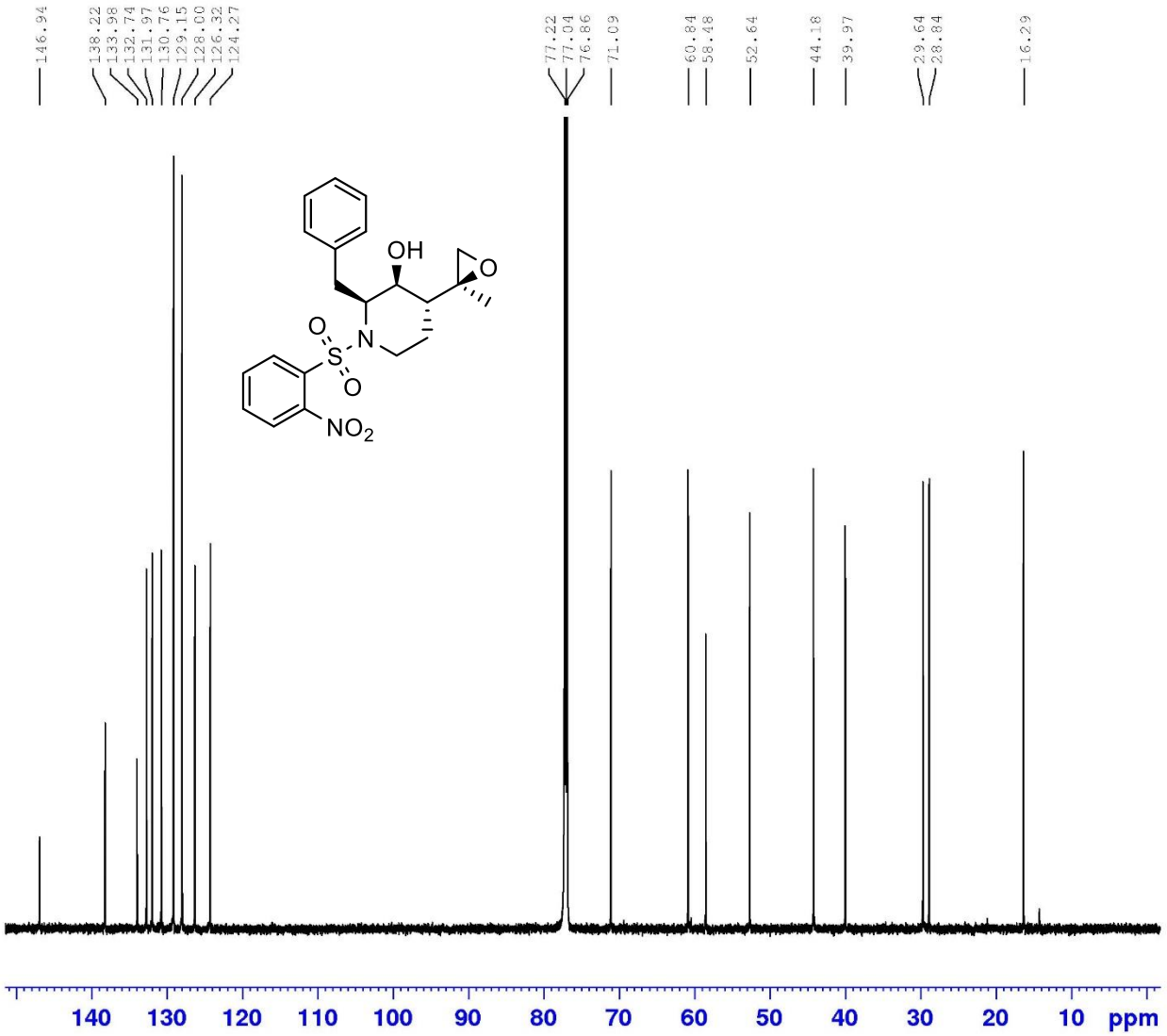
^{13}C-NMR (*S*,*R*,*R*)-**35f**

```
Current Data Parameters
NAME          Sep17-2020
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200917
Time               10.41
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



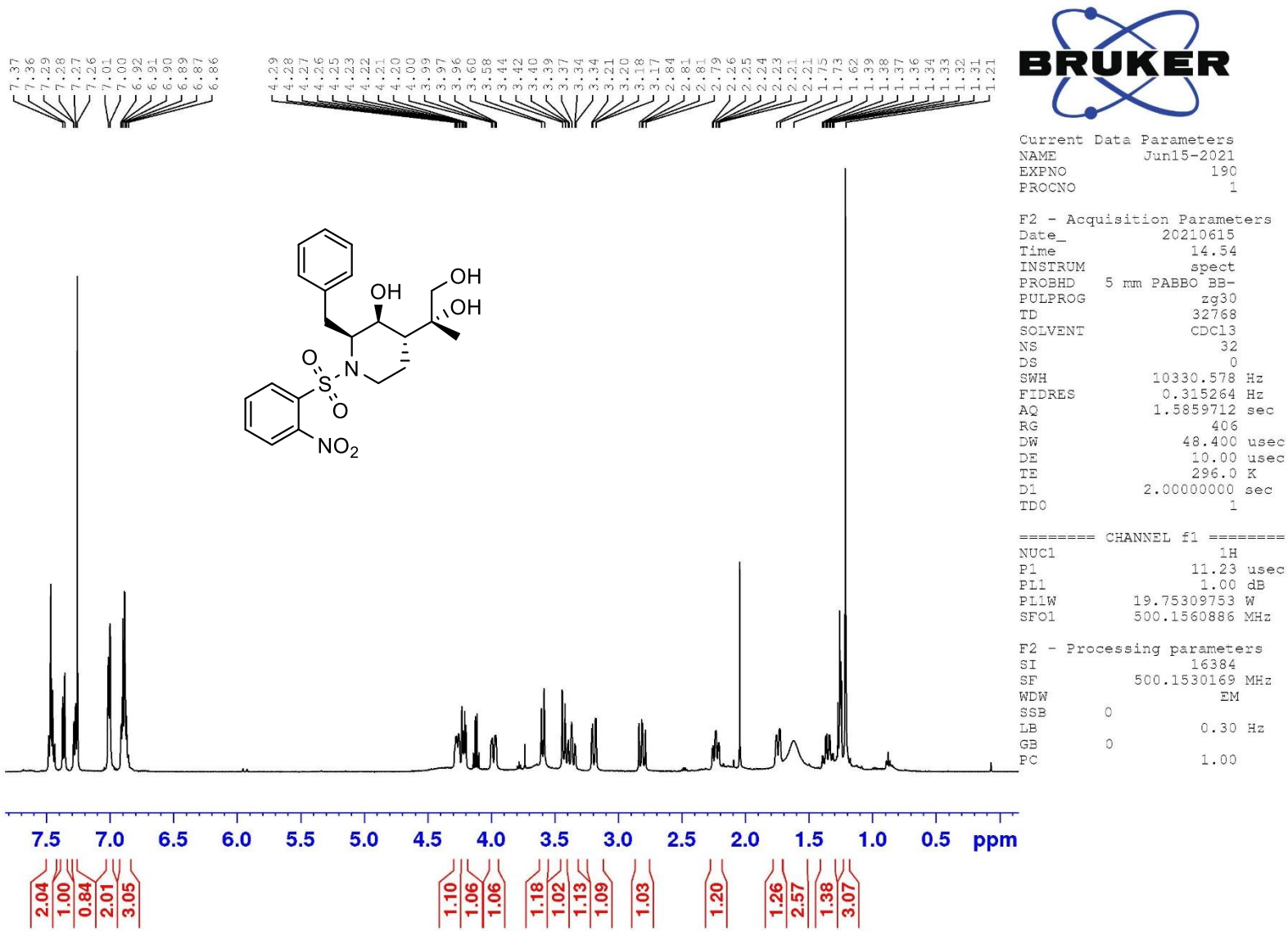

```
Current Data Parameters
NAME          Oct05-2020
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201005
Time               11.09
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG                zg
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10504.202 Hz
FIDRES          0.160281 Hz
AQ             3.1195135 sec
RG                  20.5
DW                47.600 usec
DE                20.00 usec
TE                298.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         700.3632917 MHz
NUC1                  1H
P1                 8.15 usec
PLW1          9.89999962 W

F2 - Processing parameters
SI                 65536
SF           700.3600166 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*R*,*S*)-**35f**



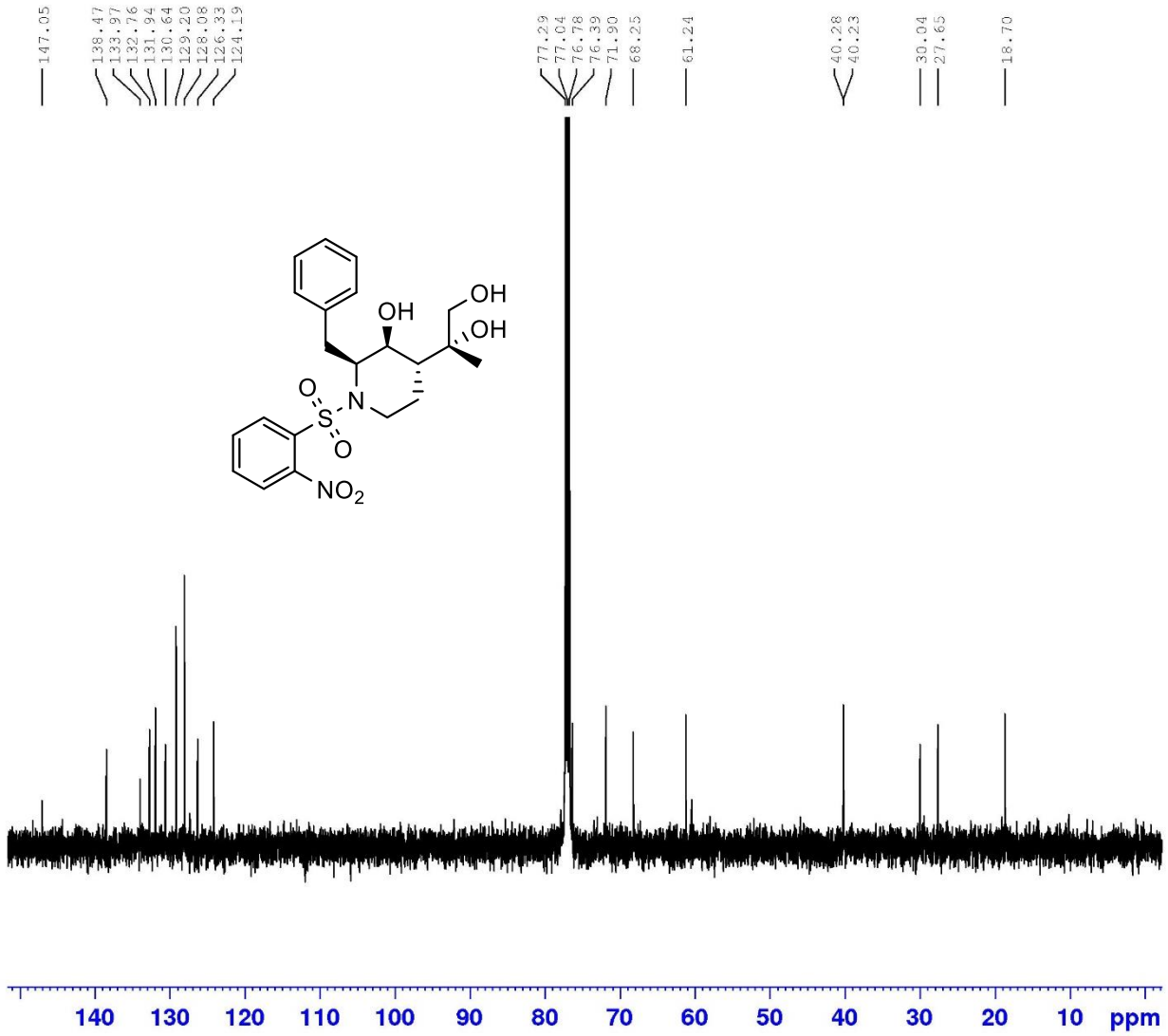
^{13}C-NMR (*S*,*R*,*S*)-**35f**

```
Current Data Parameters
NAME          Sep17-2020
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200917
Time               14.19
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF           176.1056620 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME          Jun15-2021
EXPNO                190
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210615
Time               14.54
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   406
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530169 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*S*,*R*,*R*)-**36f**



Current Data Parameters
NAME Jun15-2021
EXPNO 191
PROCNO 1

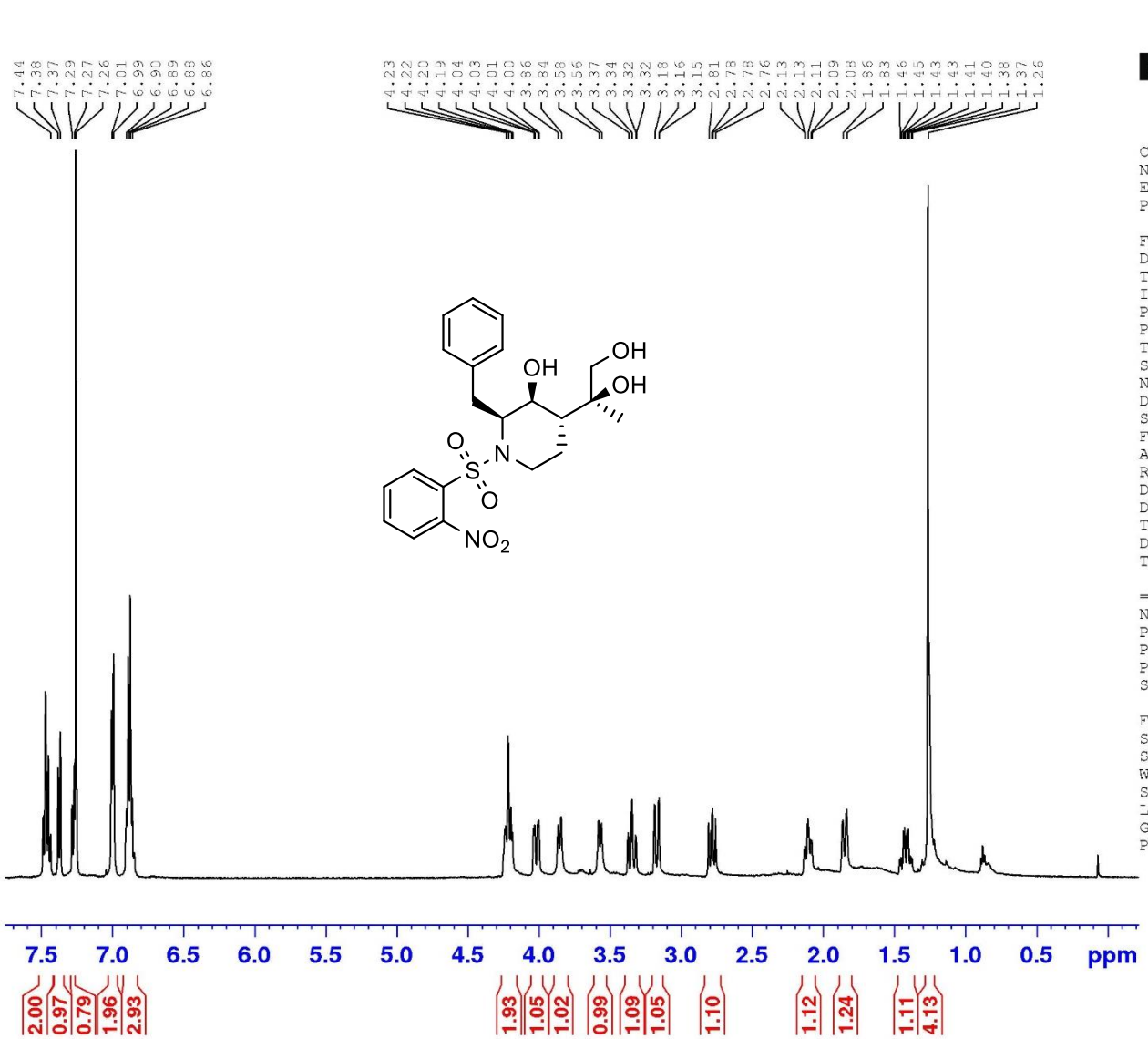
F2 - Acquisition Parameters
Date_ 20210615
Time 15.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 32894.738 Hz
FIDRES 0.501934 Hz
AQ 0.9961472 sec
RG 2580
DW 15.200 usec
DE 10.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.20 usec
PL1 1.50 dB
PL1W 51.74793243 W
SFO1 125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 19.99 dB
PL13 21.00 dB
PL2W 19.75309753 W
PL12W 0.24925002 W
PL13W 0.19753097 W
SFO2 500.1550006 MHz

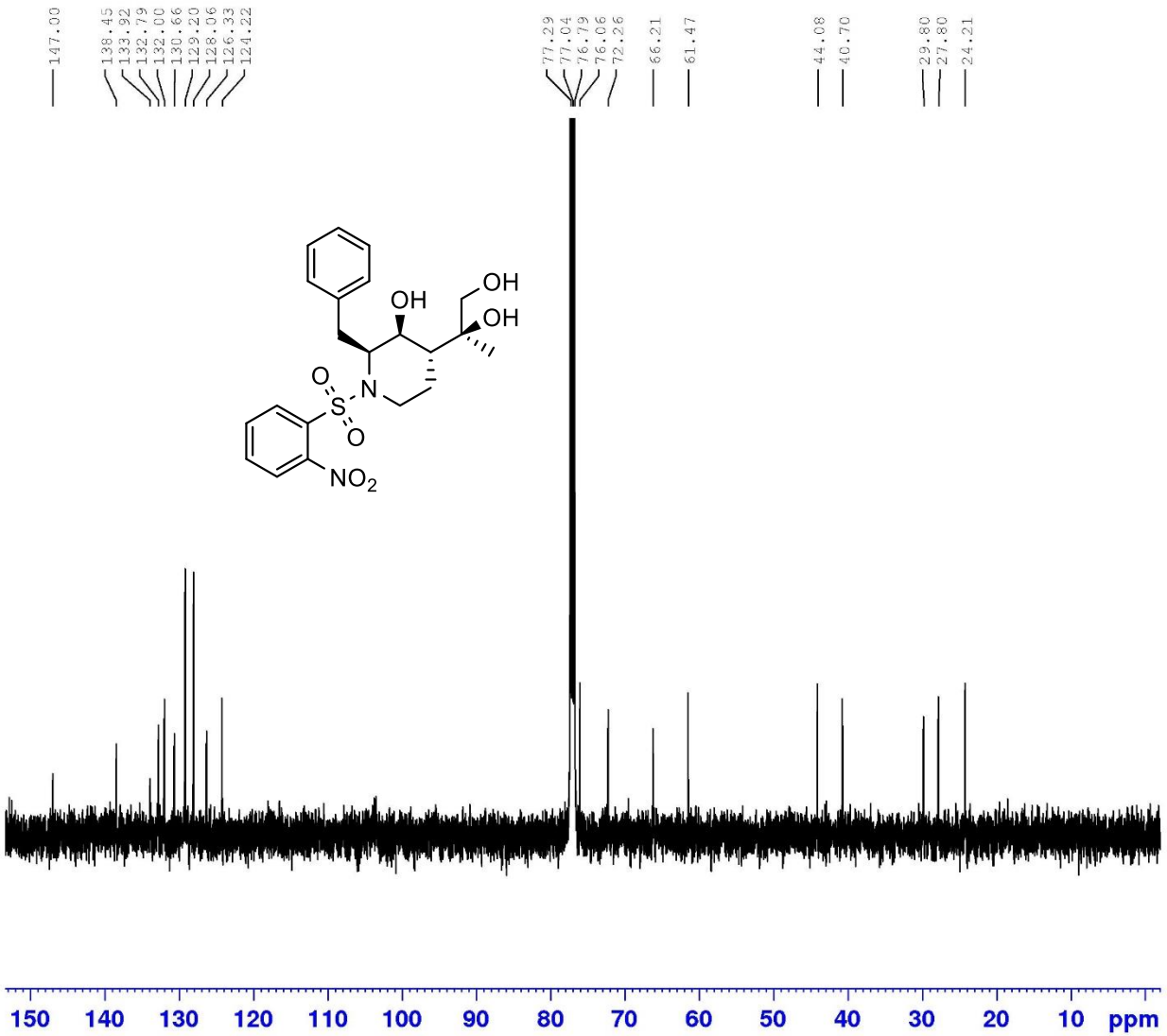
F2 - Processing parameters
SI 32768
SF 125.7635720 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{13}C-NMR (*S*,*R*,*R*)-**36f**




```
Current Data Parameters
NAME          Jul06-2021
EXPNO                 70
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210706
Time               11.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   512
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530169 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

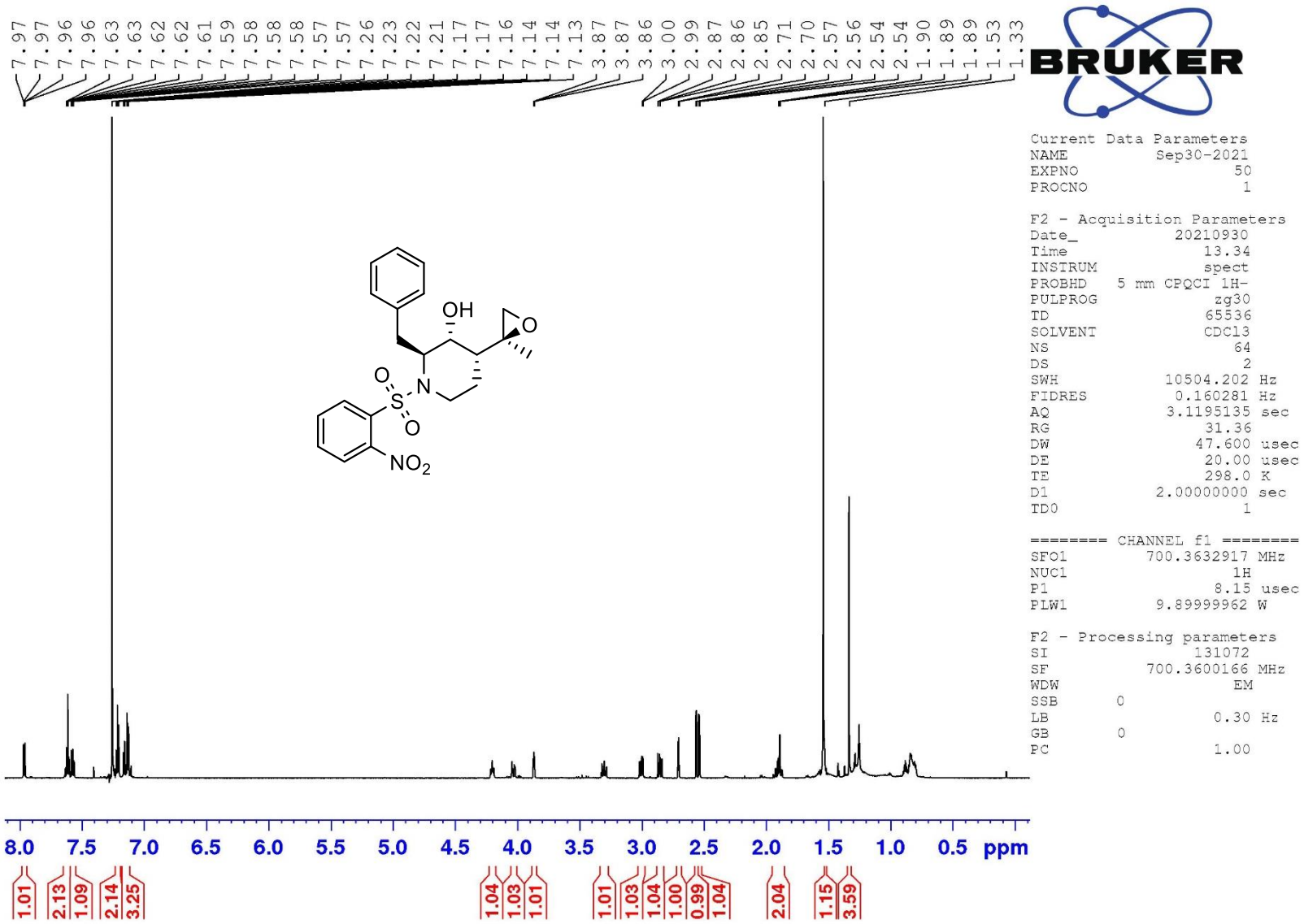
^{1}H-NMR (*S*,*R*,*S*)-**36f**



^{13}C-NMR (*S*,*R*,*S*)-**36f**

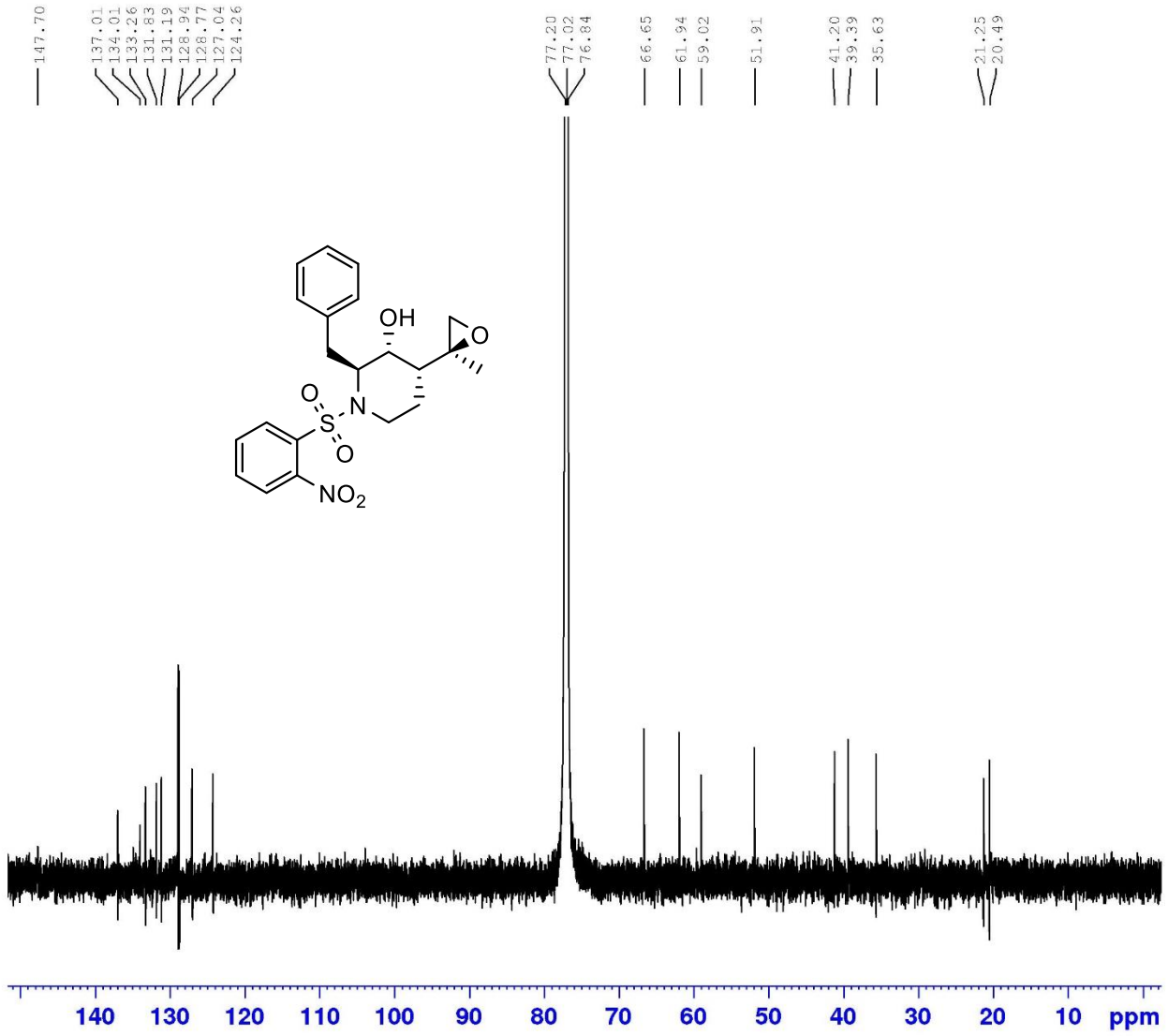
BRUKER

```
Current Data Parameters
NAME          Jun15-2021
EXPNO                201
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210615
Time               23.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



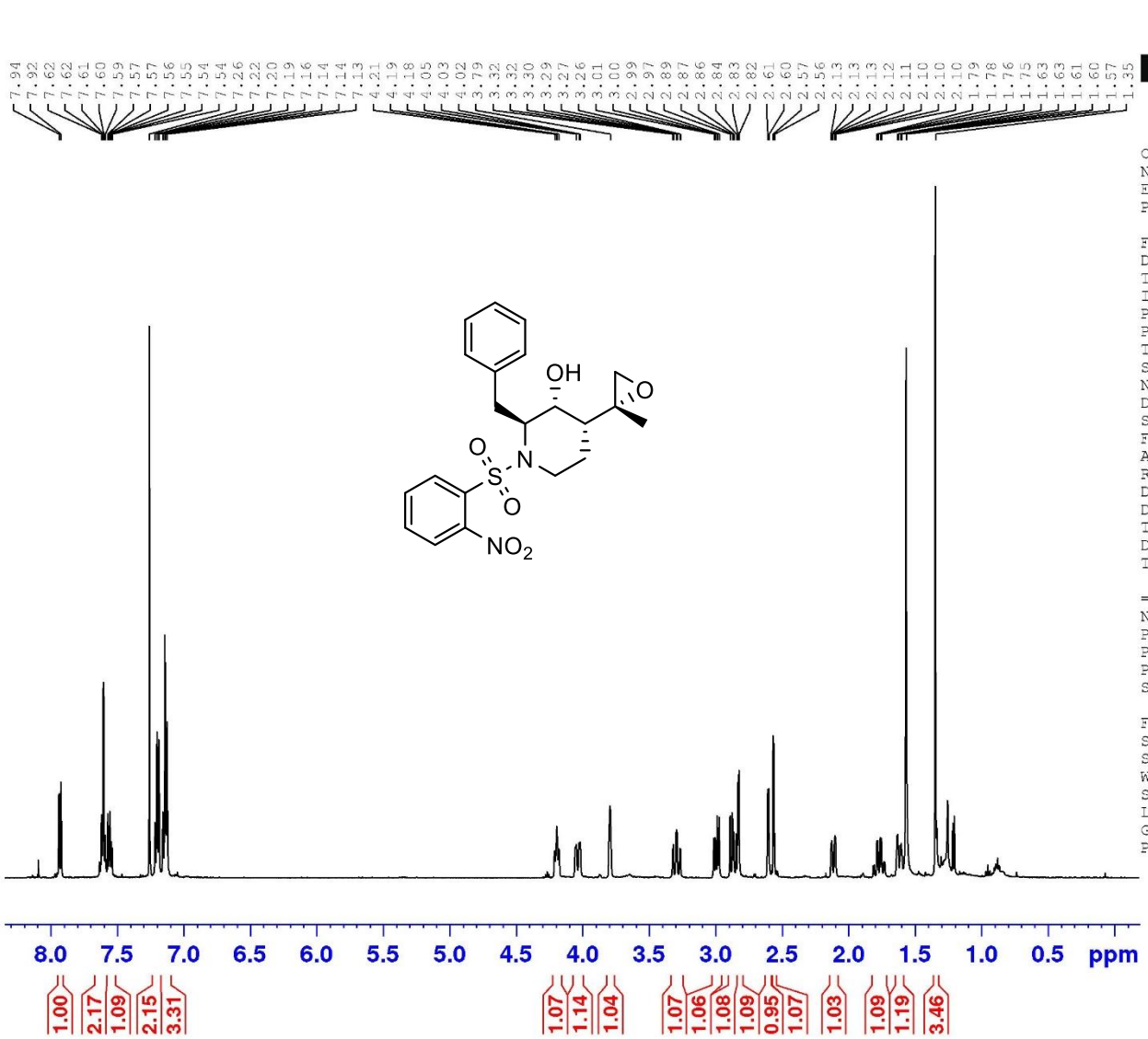

```
Current Data Parameters
NAME          Sep30-2021
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210930
Time               13.34
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    64
DS                     2
SWH            10504.202 Hz
FIDRES          0.160281 Hz
AQ             3.1195135 sec
RG                 31.36
DW                47.600 usec
DE                20.00 usec
TE                298.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         700.3632917 MHz
NUC1                   1H
P1                  8.15 usec
PLW1          9.89999962 W

F2 - Processing parameters
SI                131072
SF           700.3600166 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*R*,*R*,*S*)-**35f**



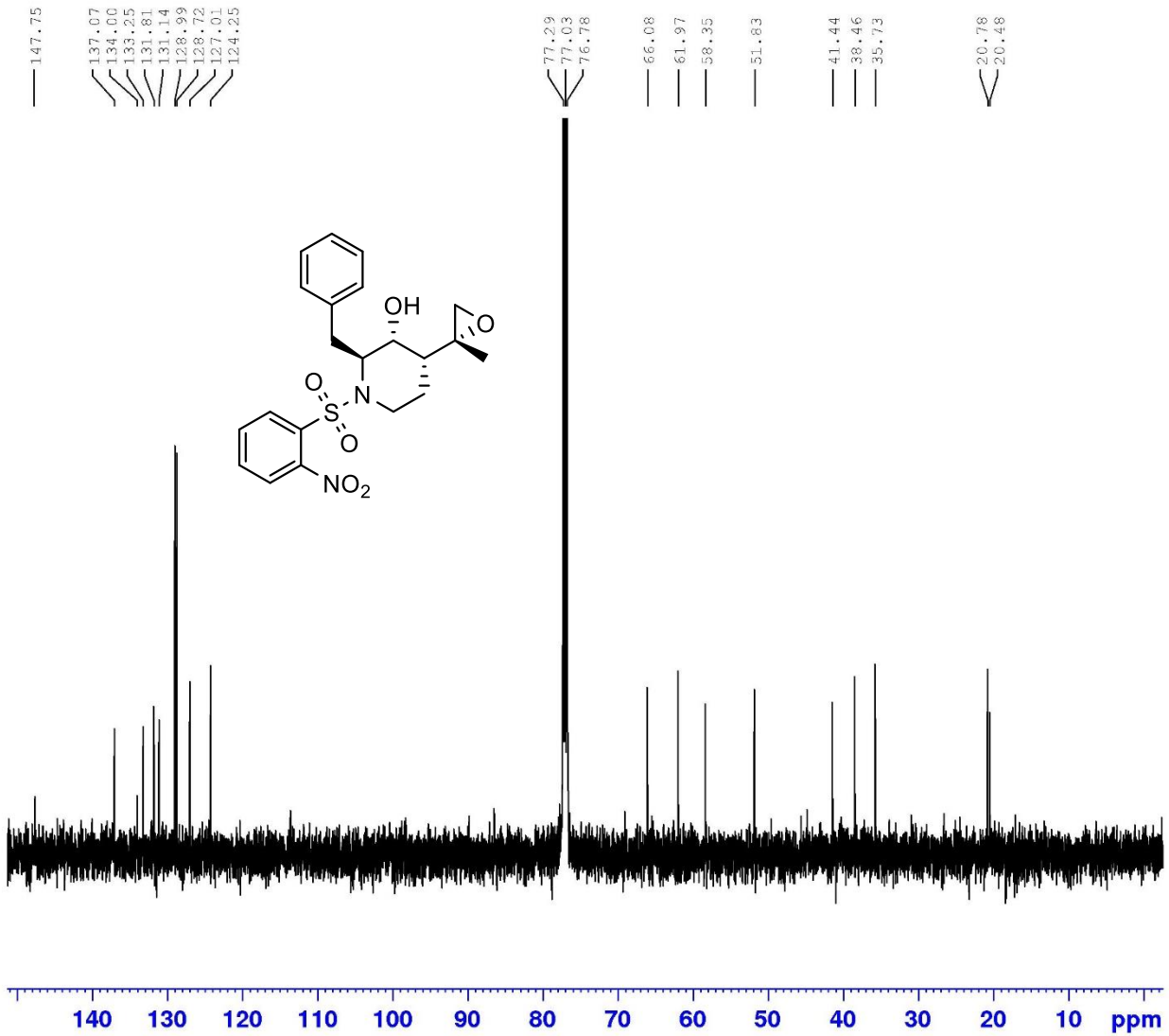
^{13}C-NMR (*R*,*R*,*S*)-**35f**

```
Current Data Parameters
NAME          Sep02-2021
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210902
Time               13.19
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG                182.53
DW                12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1               13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME          Jul14-2021
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210714
Time               11.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   512
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530160 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*R*,*R*,*R*)-**35f**



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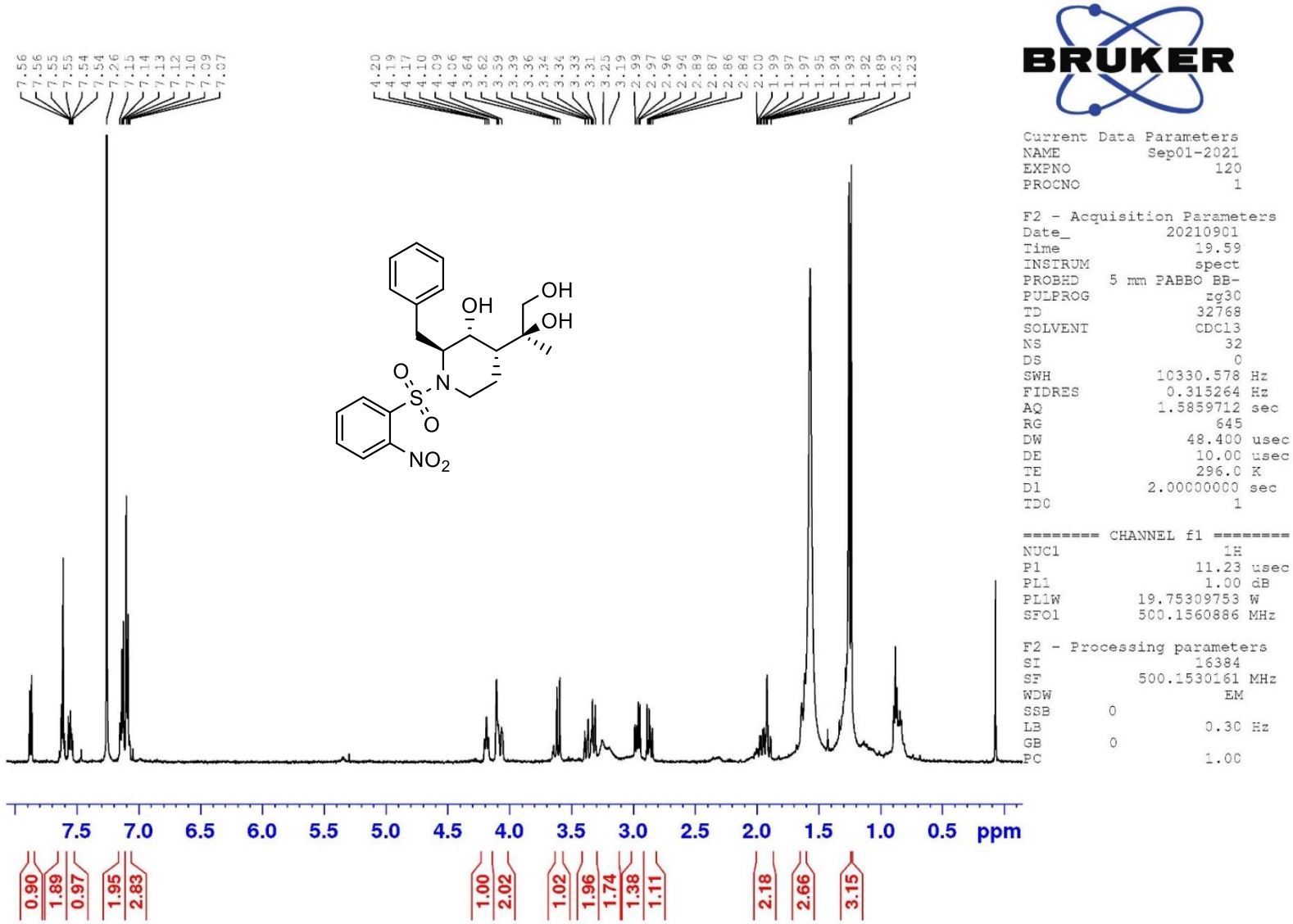
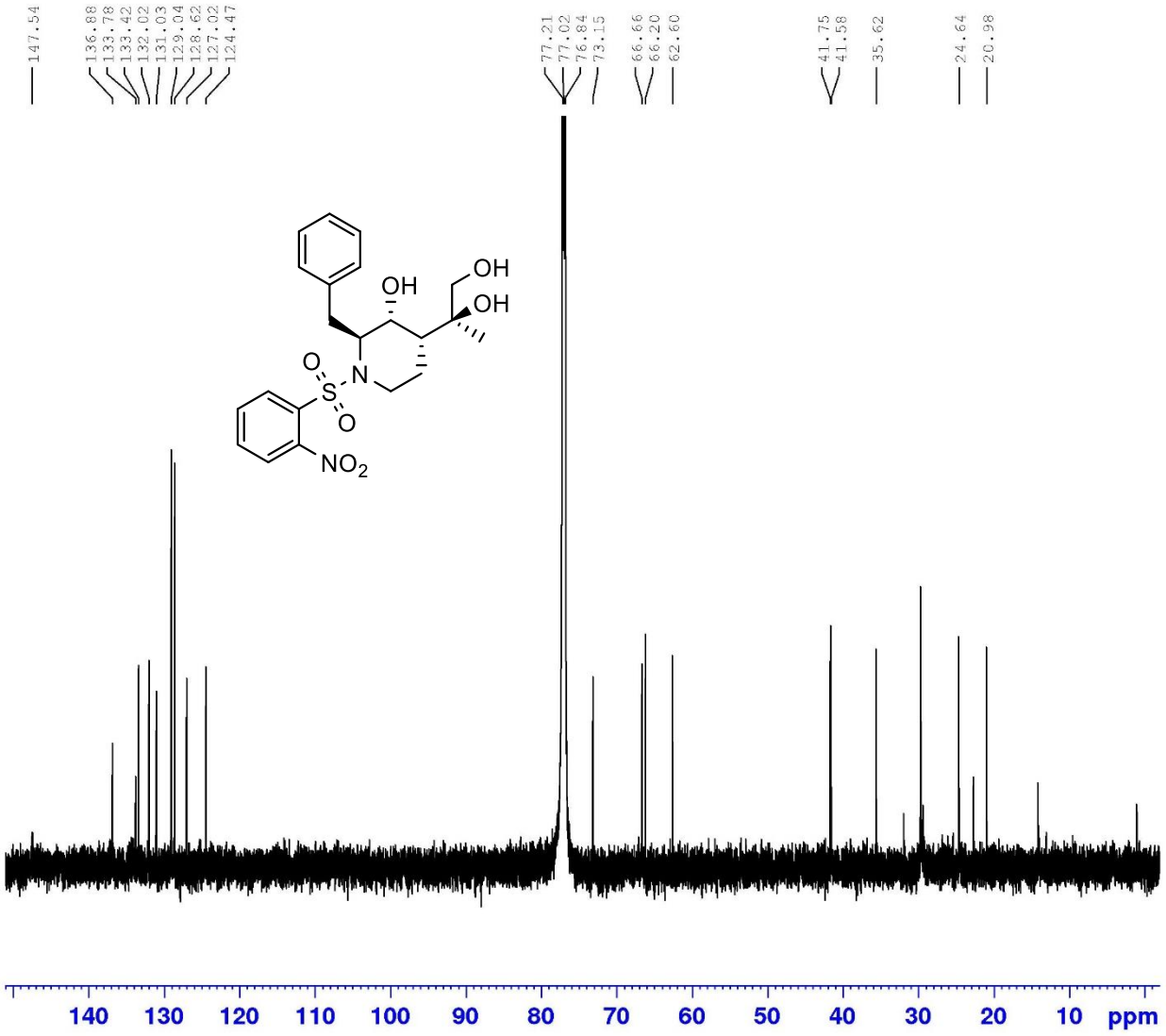
```
Current Data Parameters
NAME          Jul14-2021
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210714
Time               12.22
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                32768
SF          125.7635720 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR (*R*,*R*,*R*)-**35f**



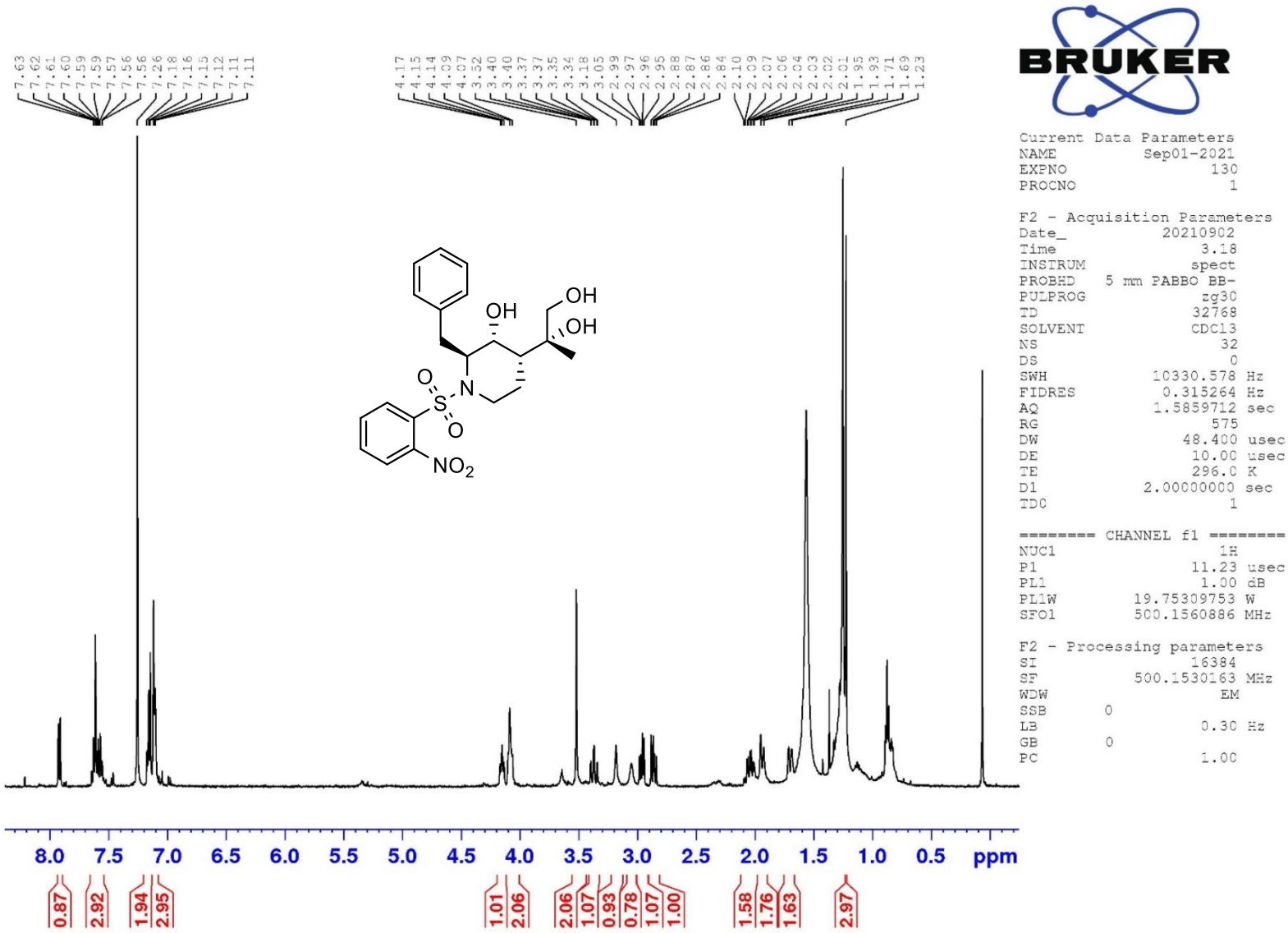

```
Current Data Parameters
NAME          Sep01-2021
EXPNO                120
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210901
Time               19.59
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   645
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530161 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (*R*,*R*,*S*)-**36f**



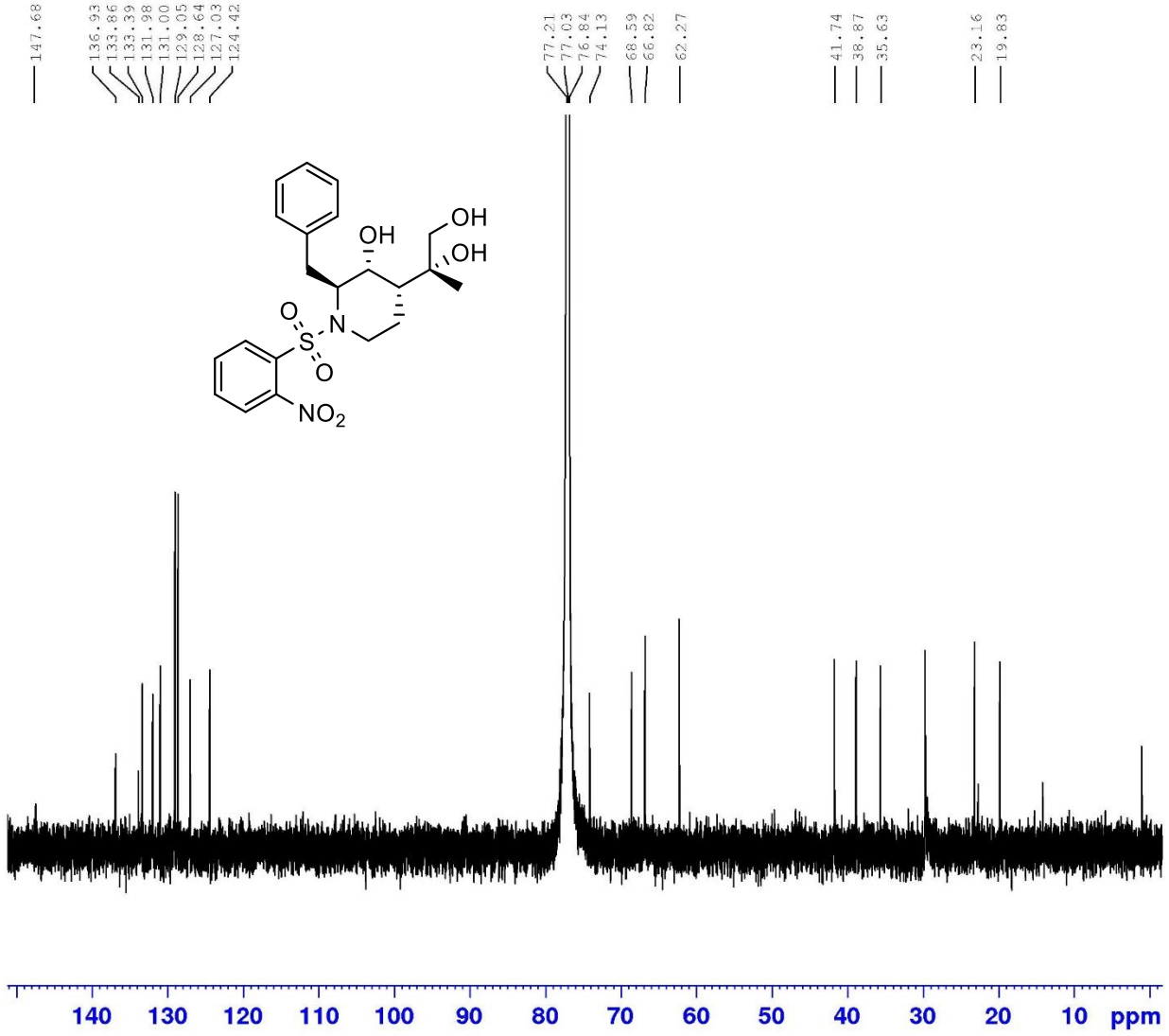
^{13}C-NMR (*R*,*R*,*S*)-**36f**

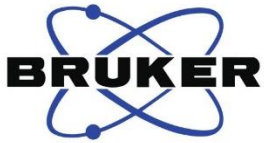
```
Current Data Parameters
NAME          Sep03-2021
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210903
Time               13.52
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE               18.00 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1               13C
P1               12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI               131072
SF          176.1056620 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```




```
Current Data Parameters
NAME          Sep01-2021
EXPNO                130
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210902
Time                3.18
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   575
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530163 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

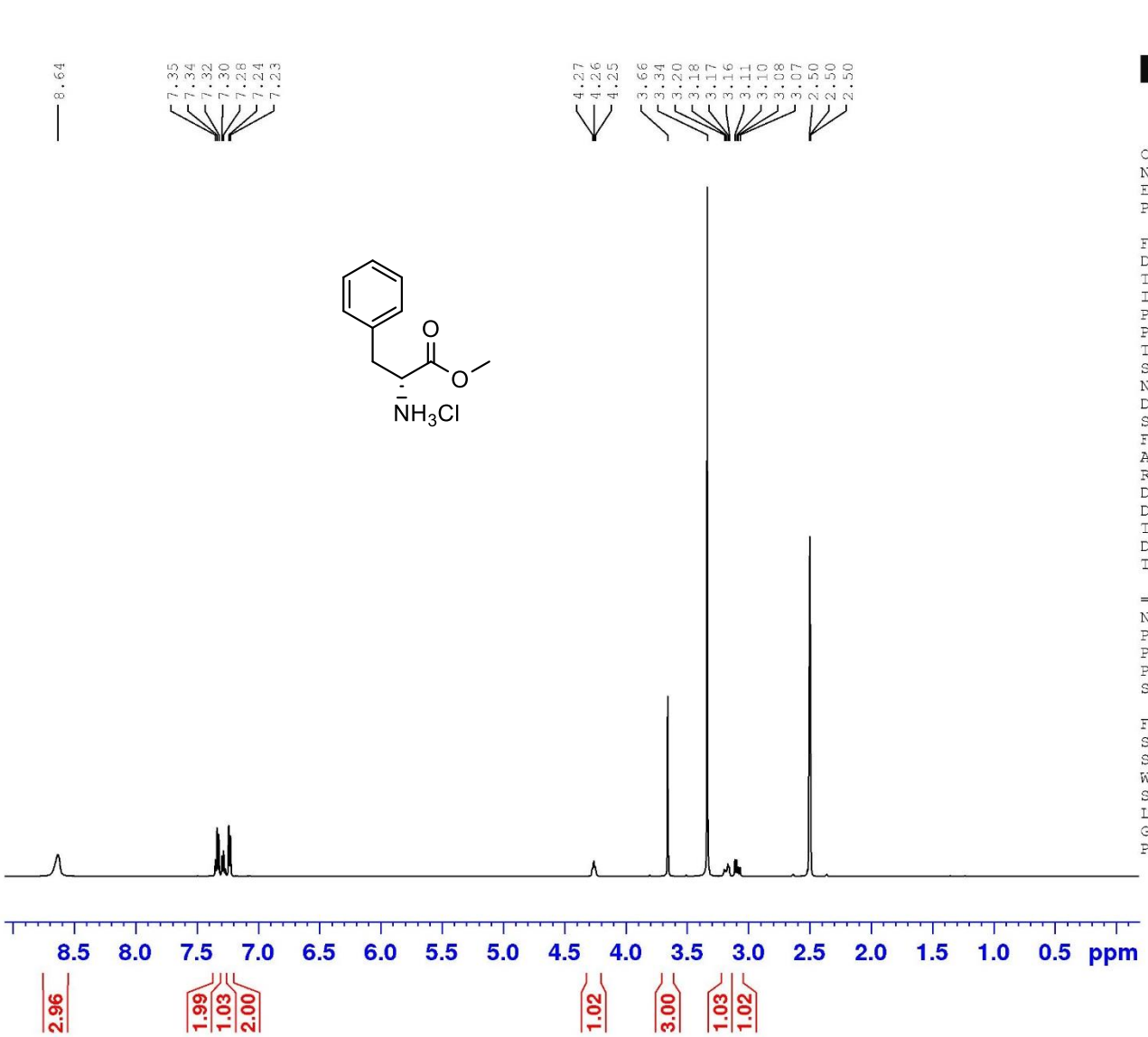
^{1}H-NMR (*R*,*R*,*R*)-**36f**





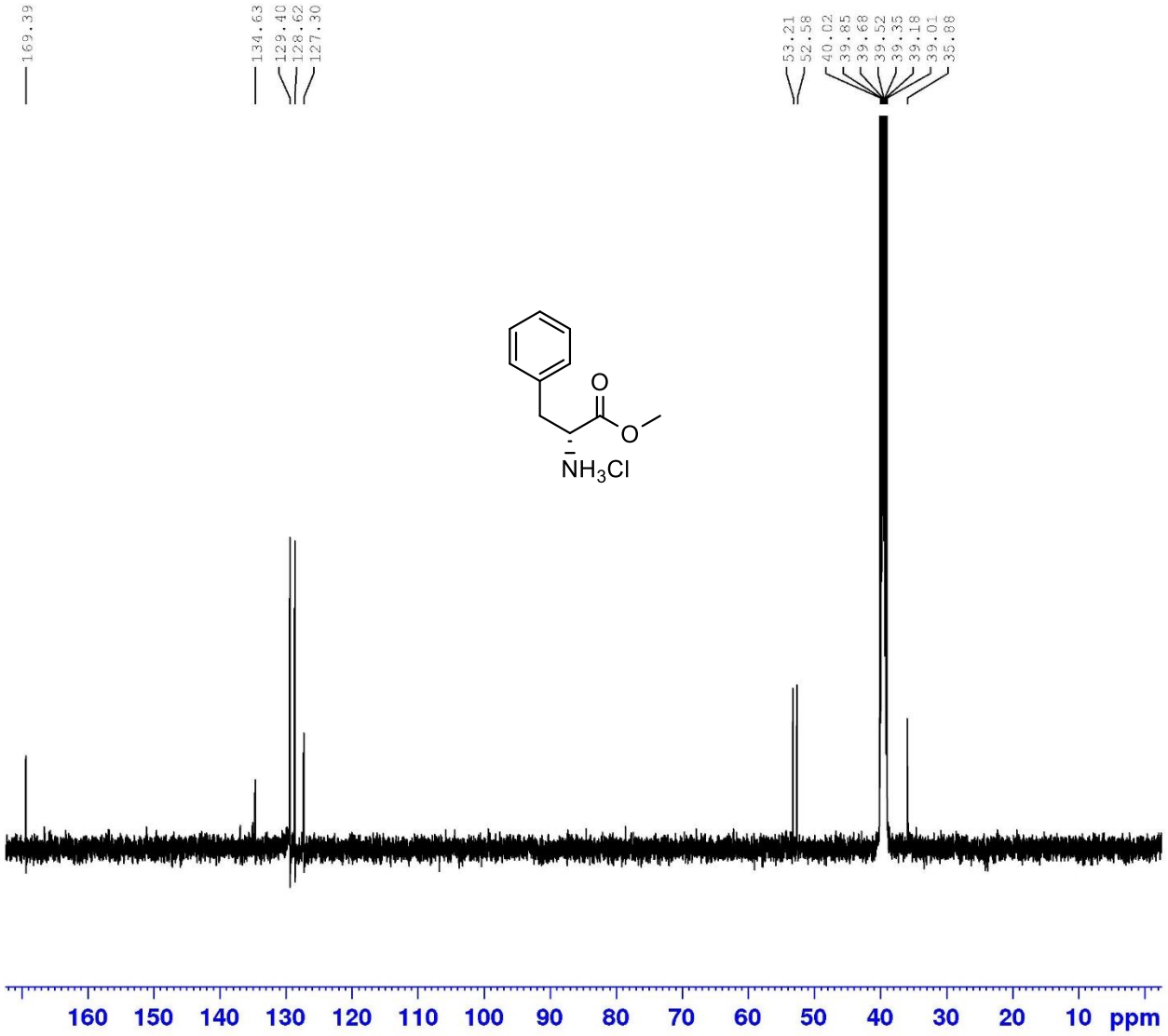

```
Current Data Parameters
NAME          Sep03-2021
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210903
Time               12.08
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG                182.53
DW                12.267 usec
DE                 18.00 usec
TE                 298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                  13C
P1                 12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              65.00 usec
PLW2          9.89999962 W
PLW12         0.15564001 W
PLW13         0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

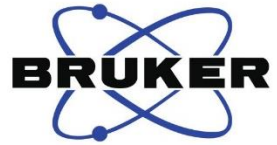
^{13}C-NMR (*R*,*R*,*R*)-**36f**




```
Current Data Parameters
NAME          Oct13-2020
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201013
Time               12.38
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   322
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530073 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

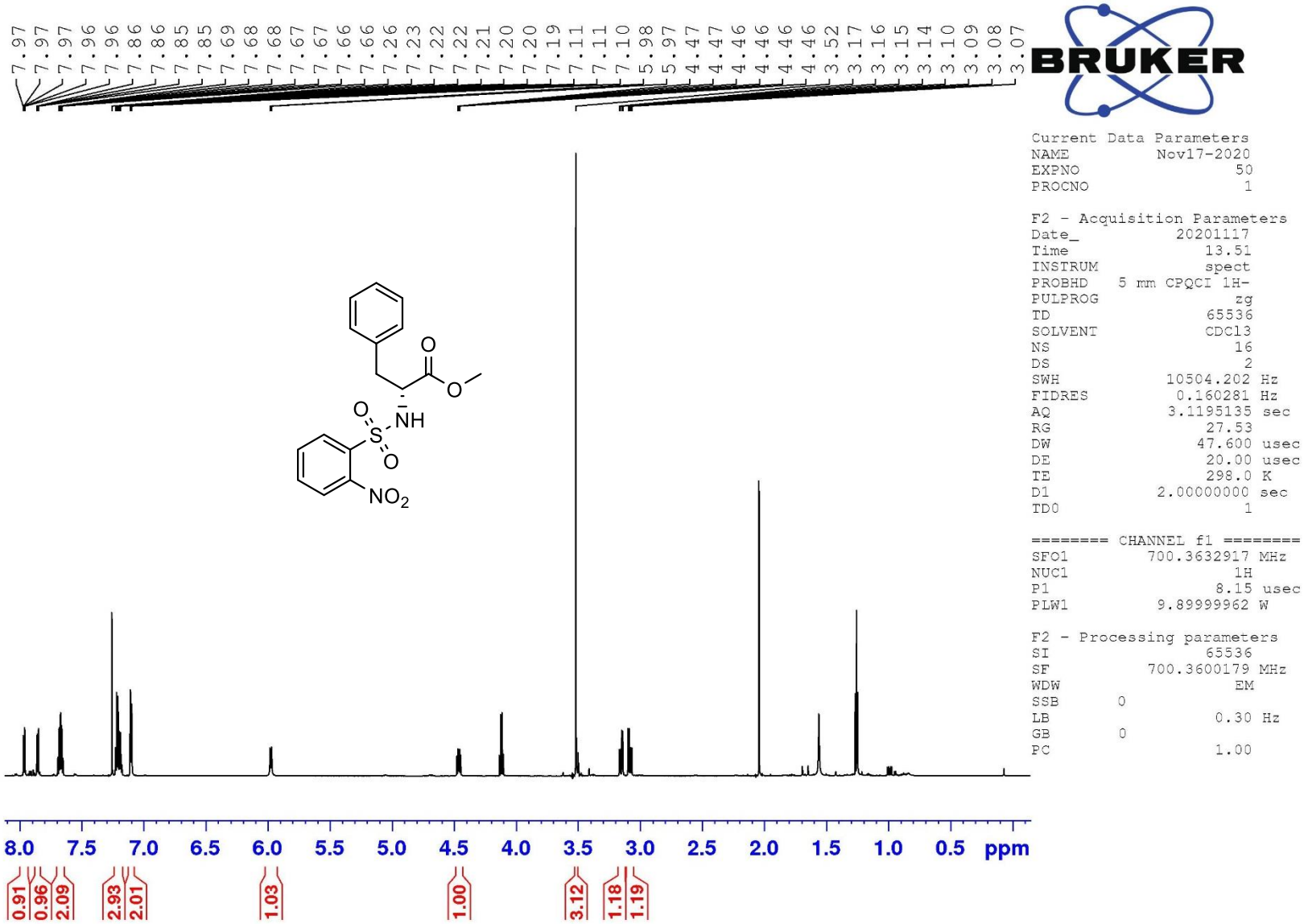
^{1}H-NMR **25g**



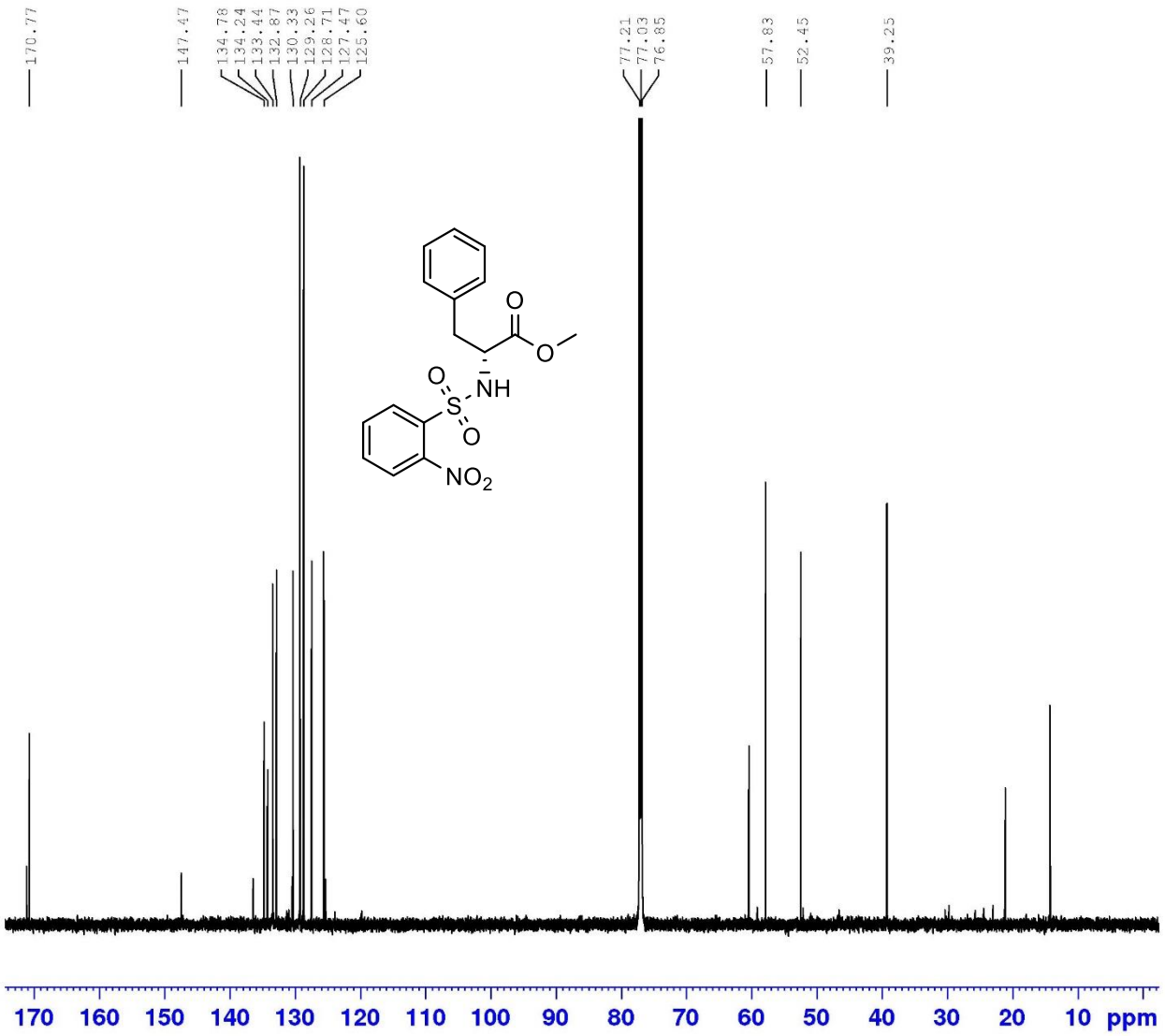



```
Current Data Parameters
NAME          Oct13-2020
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201013
Time               13.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  1024
DS                     4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961472 sec
RG                  2580
DW                15.200 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.20 usec
PL1                  1.50 dB
PL1W         51.74793243 W
SFO1        125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7636319 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

^{13}C-NMR **25g**

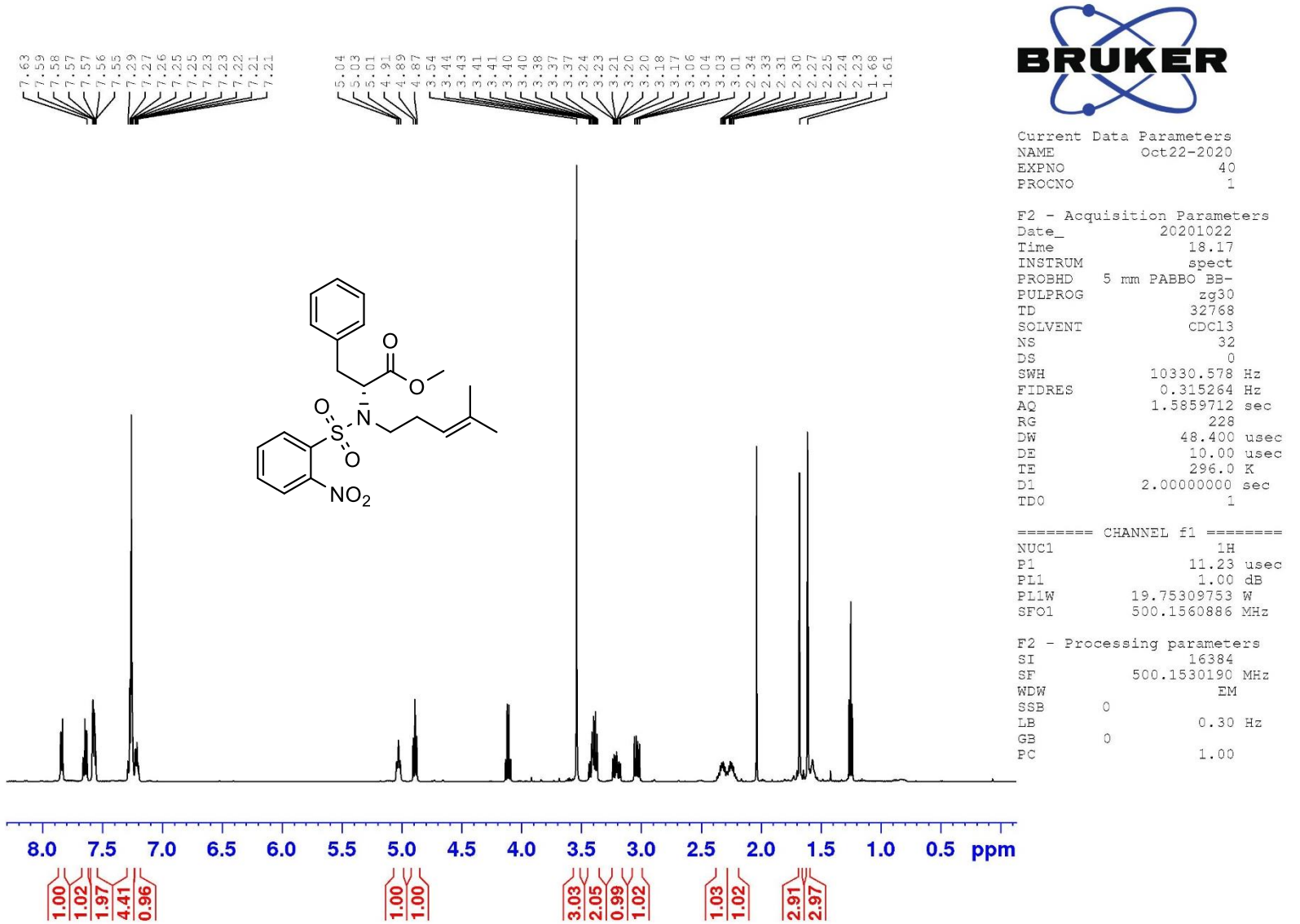

7.97 7.97 7.97 7.96 7.96 7.86 7.86 7.85 7.85 7.69 7.68 7.68 7.67 7.67 7.66 7.66 7.26 7.23 7.22 7.22 7.21 7.20 7.20 7.19 7.11 7.11 7.10 5.98 5.97 4.47 4.47 4.46 4.46 4.46 4.46 3.52 3.17 3.16 3.15 3.14 3.10 3.09 3.08 3.07
BRUKER
Current Data Parameters
NAME Nov17-2020
EXPNO 50
PROCNO 1
F2 - Acquisition Parameters
Date_ 20201117
Time 13.51
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10504.202 Hz
FIDRES 0.160281 Hz
AQ 3.1195135 sec
RG 27.53
DW 47.600 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 700.3632917 MHz
NUC1 1H
P1 8.15 usec
PLW1 9.89999962 W
F2 - Processing parameters
SI 65536
SF 700.3600179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
NH
S
O
O
NO2
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.91 0.96 2.09 2.93 2.01 1.03 1.00 3.12 1.18 1.19

^{1}H-NMR **26g**



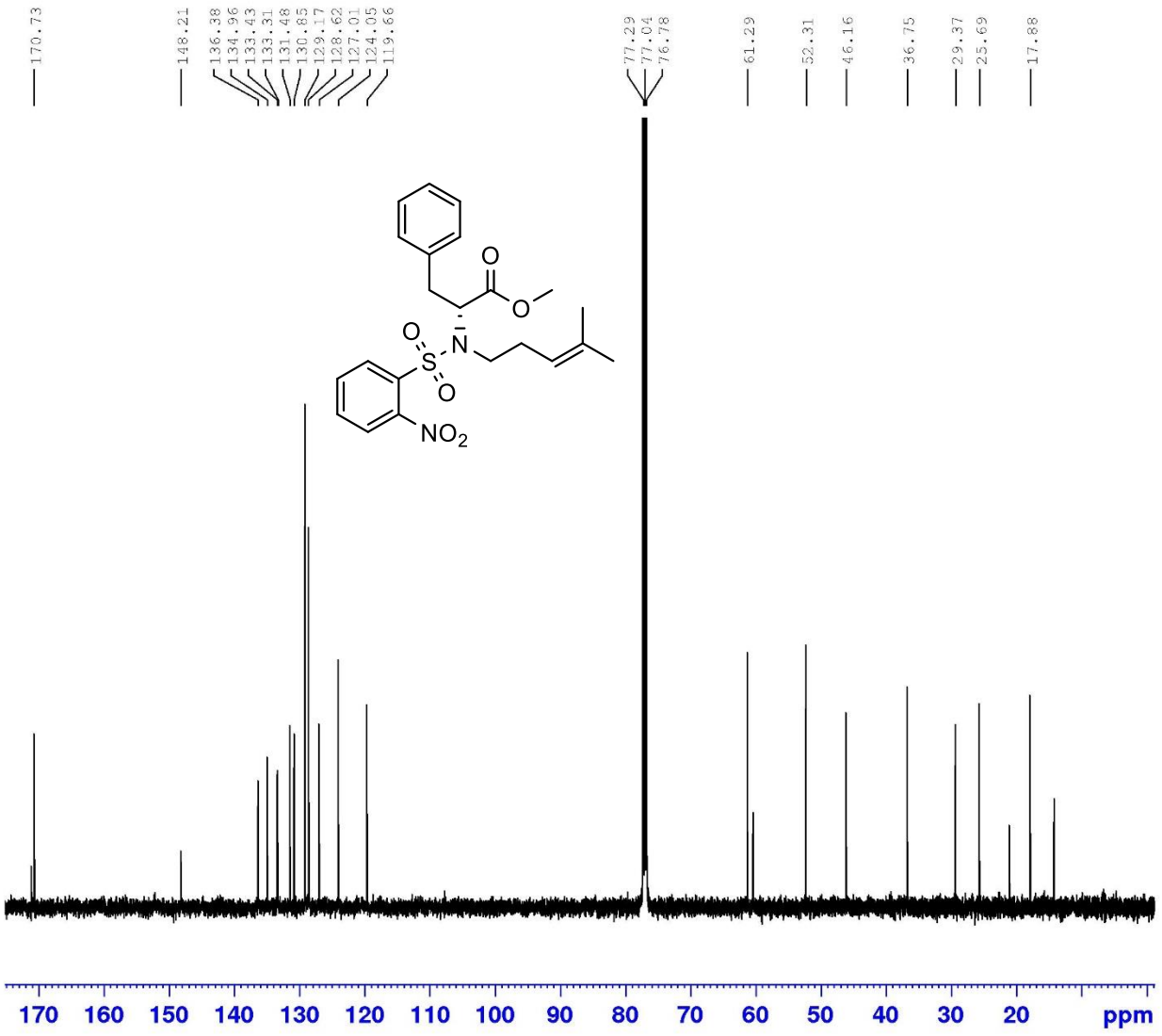
^{13}C-NMR **26g**


```
Current Data Parameters
NAME          Nov17-2020
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201117
Time               14.16
INSTRUM            spect
PROBHD   5 mm CPQCI 1H-
PULPROG          zgpg30
TD                65356
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           40760.871 Hz
FIDRES         0.623675 Hz
AQ            0.8017003 sec
RG               182.53
DW               12.267 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.1232717 MHz
NUC1                13C
P1                12.00 usec
PLW1       105.00000000 W

======== CHANNEL f2 ========
SFO2        700.3628014 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2           9.89999962 W
PLW12          0.15564001 W
PLW13          0.07837200 W

F2 - Processing parameters
SI                131072
SF          176.1056620 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```




```
Current Data Parameters
NAME          Oct22-2020
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201022
Time               18.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ             1.5859712 sec
RG                   228
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530190 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR **27g**



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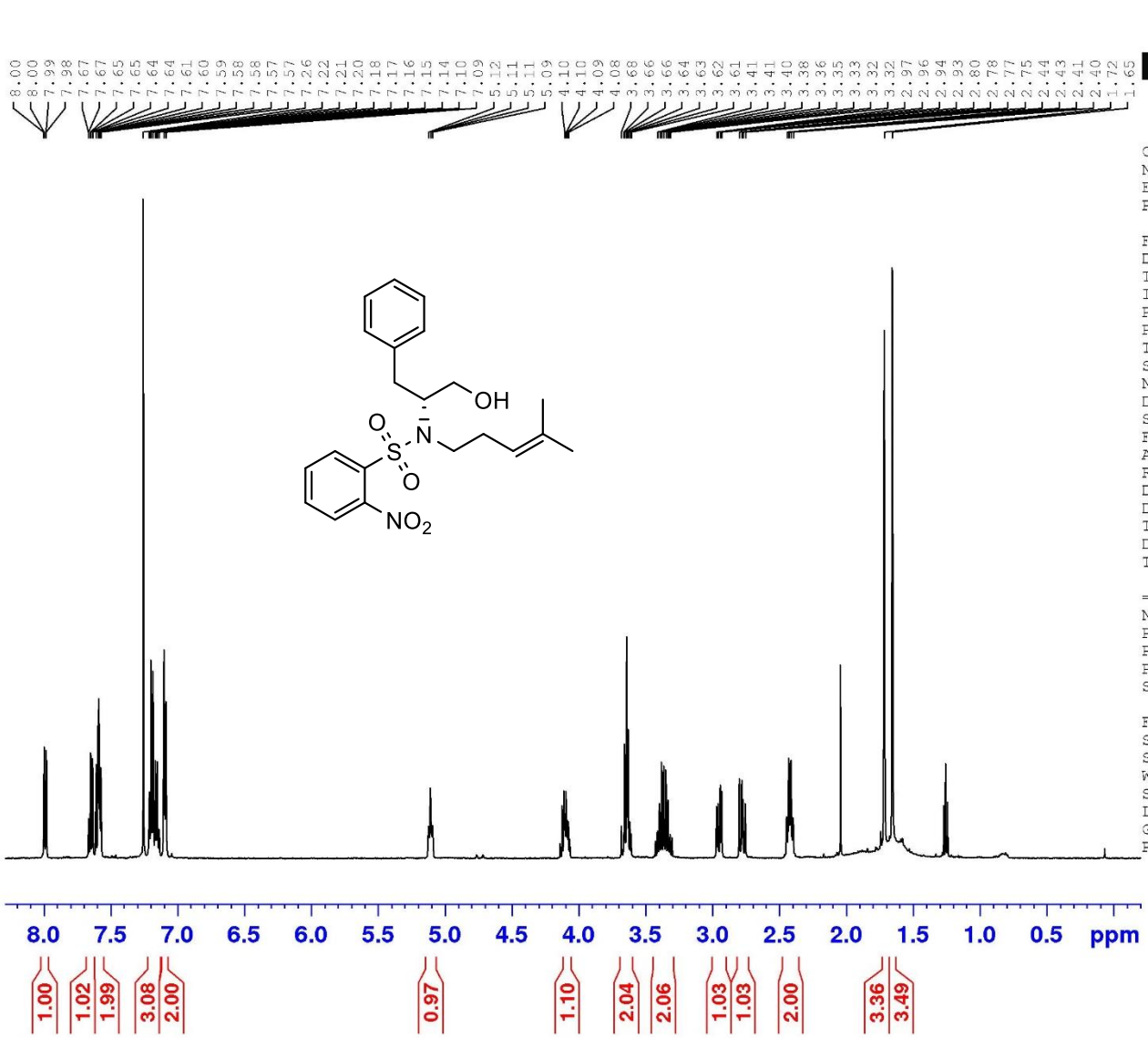
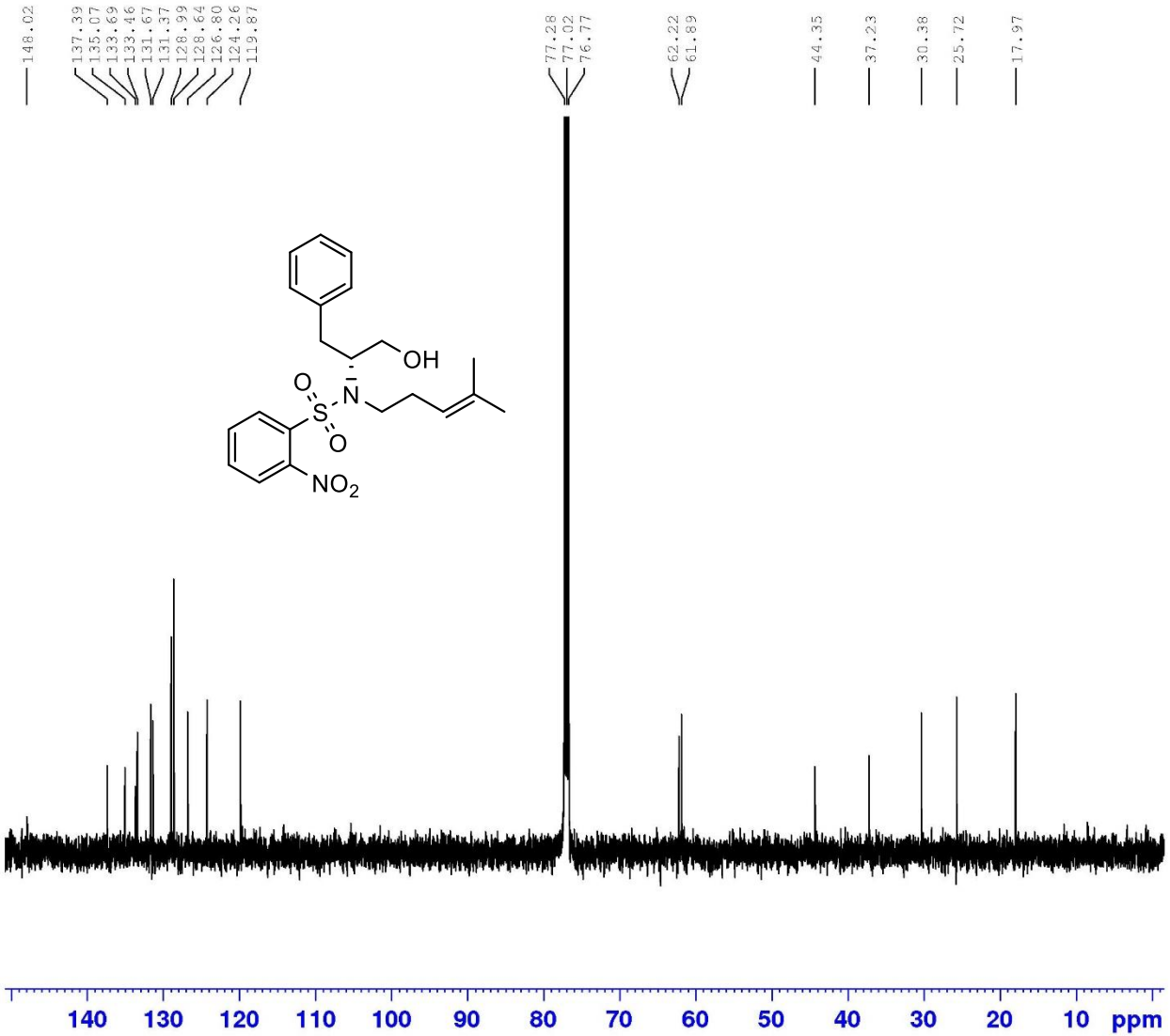
```
Current Data Parameters
NAME          Oct22-2020
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201022
Time               19.11
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES        0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.20 usec
PL1                  1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                  1.00 dB
PL12                19.99 dB
PL13                21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

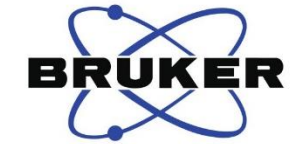
^{13}C-NMR **27g**




```
Current Data Parameters
NAME          Nov05-2020
EXPNO                 40
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201105
Time               18.31
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH           10330.578 Hz
FIDRES         0.315264 Hz
AQ            1.5859712 sec
RG                   512
DW               48.400 usec
DE               10.00 usec
TE                296.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.23 usec
PL1                1.00 dB
PL1W        19.75309753 W
SFO1        500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF          500.1530172 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

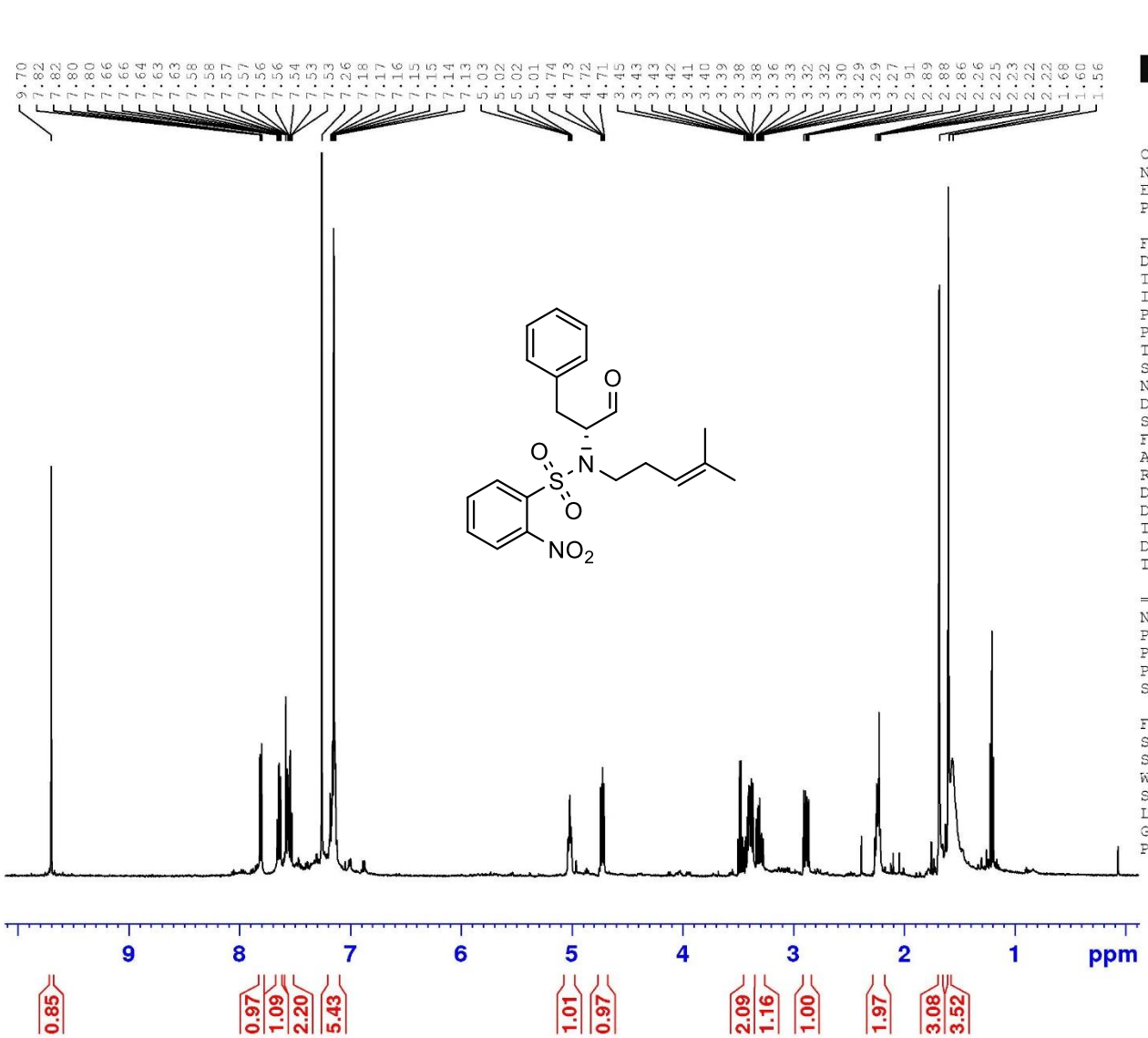
^{1}H-NMR **28g**






```
Current Data Parameters
NAME          Nov05-2020
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201105
Time               19.25
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1       125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2       500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF         125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

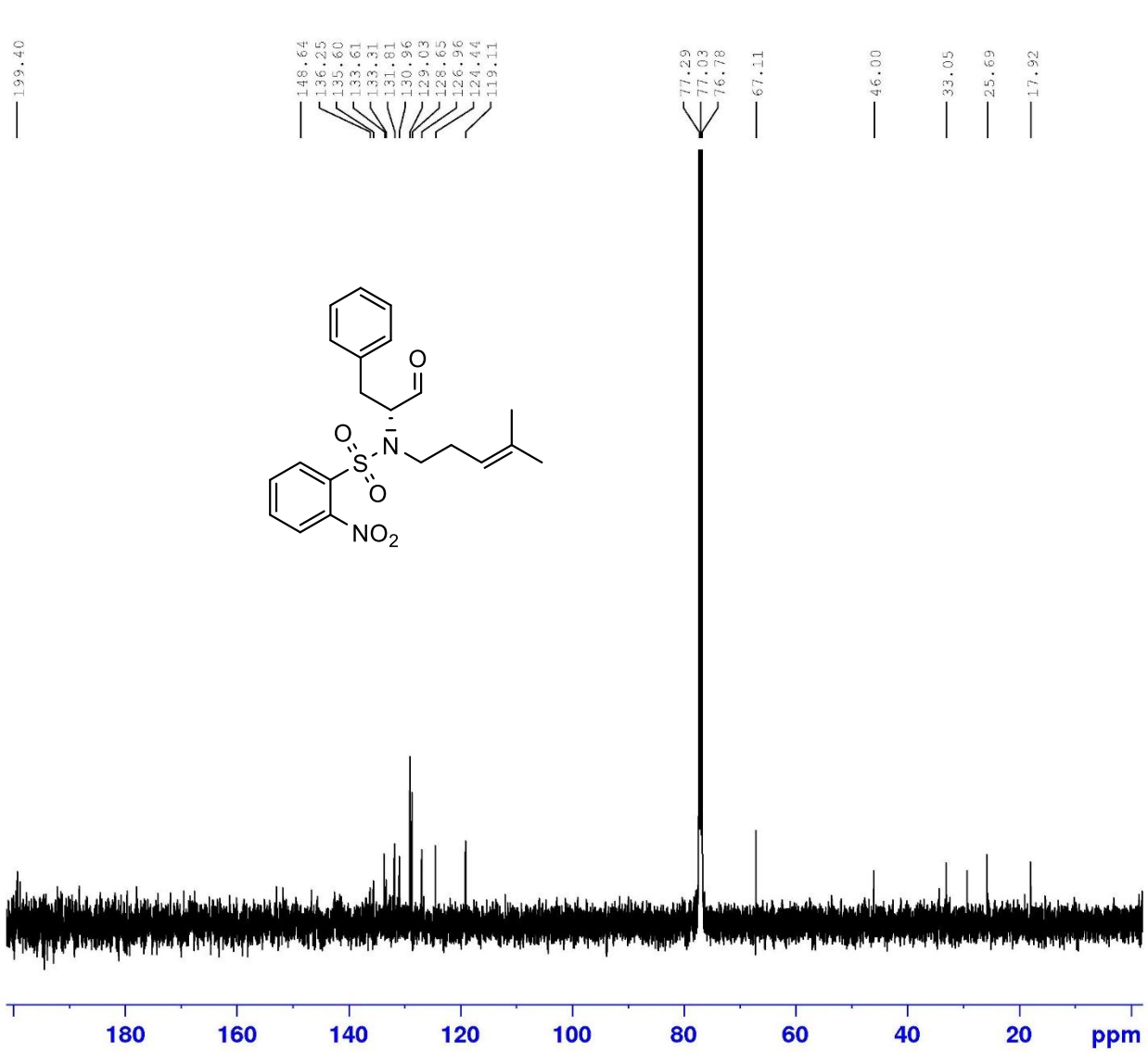
^{13}C-NMR **28g**



BRUKER

```
Current Data Parameters
NAME          Nov09-2020
EXPNO                120
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201109
Time               15.33
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5859712 sec
RG                   512
DW                48.400 usec
DE                10.00 usec
TE                296.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.23 usec
PL1                 1.00 dB
PL1W         19.75309753 W
SFO1         500.1560886 MHz

F2 - Processing parameters
SI                 16384
SF           500.1530160 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR **29g**



BRUKER

```
Current Data Parameters
NAME          Nov09-2020
EXPNO                121
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201109
Time               16.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES         0.501934 Hz
AQ            0.9961472 sec
RG                 2580
DW               15.200 usec
DE               10.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                10.20 usec
PL1                1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C-NMR **29g**