Electronic Supplementary Material (ESI) for Green Chemistry.

Supplementary Information for

Metal-free radical-mediated alkylfunctionalization of ethylene and low-boiling alkenes

Xu Zhang[1], Xinxin Wu[1], Yasu Chen,[2] and Chen Zhu[1,2]*

[1] Key Laboratory of Organic Synthesis of Jiangsu Province, College of Chemistry, Chemical Engineering and Materials Science, Soochow University, 199 Ren-Ai Road, Suzhou, Jiangsu 215123, People's Republic of China

[2] Frontiers Science Center for Transformative Molecules and Shanghai Key Laboratory for Molecular Engineering of Chiral Drugs, Shanghai Jiao Tong University, 800 Dongchuan Road, Shanghai 200240, People's Republic of China

*Corresponding authors: chzhu@sjtu.edu.cn (C.Z.)

Contents

1. General experimental details

Unless otherwise noted, chemicals and anhydrous solvents were purchased from commercial suppliers and used without further purification. DMF and DMSO was distilled from NaH under reduced pressure, and THF was distilled from sodium. The LED PR160 series (ν_{max} = 390 nm, 40 W) were used as light sources for reaction optimization. The product mixtures were analyzed by thin layer chromatography using TLC silica gel plates (MerckSchuchardt) with fluorescent indicator (λ = 254 nm). The purification of the products was performed by flash column chromatography using silica gel 60 (63-200 μm) from SANPONT. Infrared (FT-IR) spectra were recorded on a BRUKER VERTEX 70, ν_{max} in cm^{-1}. ^{1}H-NMR spectra were recorded on a BRUKER AVANCE III HD (400 MHz) spectrometer. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$: δ 7.26). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quadruplet, br = broad, m = multiplet), coupling constants (Hz) and integration. ^{13}C-NMR spectra were recorded on a BRUKER AVANCE III HD (100 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$: δ 77.16). ^{19}F-NMR spectra were recorded on a BRUKER AVANCE III HD (376 MHz) spectrometer. Mass spectra were measured with an Agilent Technologies 6120 Quadrupole LC/MS. High resolution mass spectrometry (HRMS) were measured with a GCT Premier™ and BRUKER micrOTF-Q III. Melting points were measured using INESA WRR and values are uncorrected. Quantum yield was measured by using Cary 5000 UV-Vis-NIR Spectrophotometer.

2. Reaction parameters survey

2.1 Reaction setups

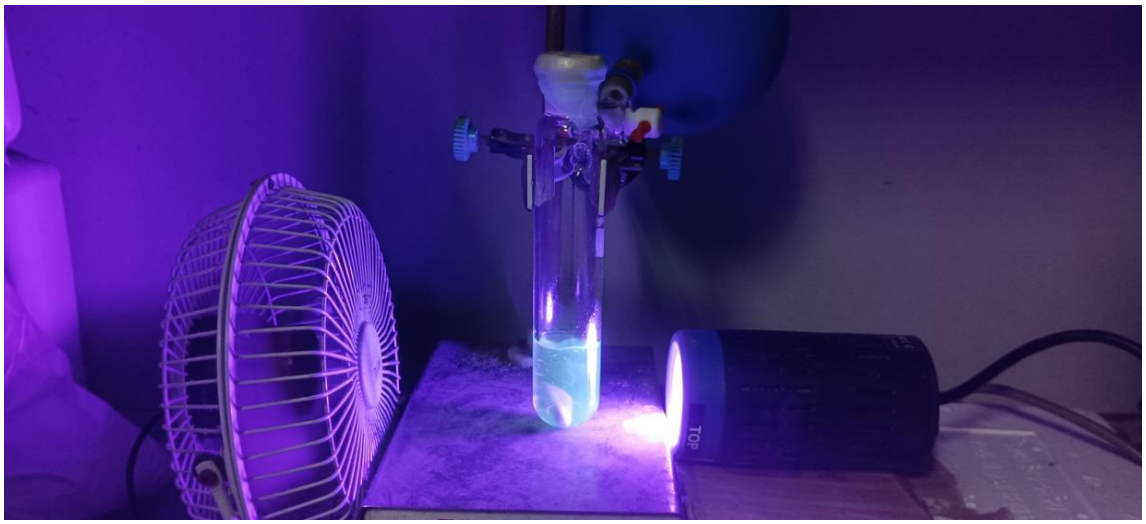
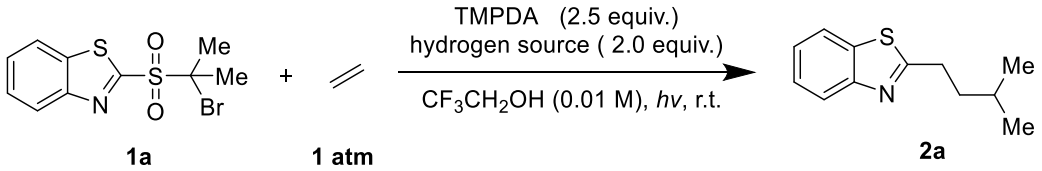


Figure S1. Reaction setups. 0.2 mmol-scale reactor charged with an ethylene balloon (1 atm)

2.2 Reaction parameters survey

Table S1. Evaluation of hydrogen source [a]

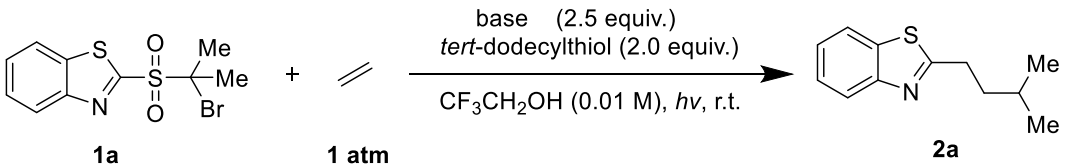


1a **1 atm** **2a**

entry	hydrogen source	yield (%) [b]
1	1,4-dihydropyridine	0
2	PhSH	25
3	triisopropylsilanethiol	30
4	*tert*-dodecylthiol	73
5	Cyclohexa-1,4-diene	trace

[a] Reaction condition: **1a** (0.2 mmol, 1.0 equiv.), hydrogen source (0.4 mmol, 2.0 equiv.) and TMPDA (0.5 mmol, 2.5 equiv.) in CF_3CH_2OH (20 mL) were irradiated with 40 W 390 nm LED light at r.t. for 6 h under ethylene (1 atm). [b]Yields of isolated products.

Table S2. Evaluation of bases [a]

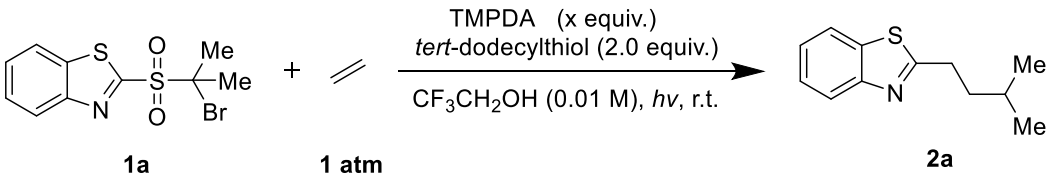


1a **1 atm** **2a**

entry	base	yield (%) [b]
1	2,6-lutidine	trace
2	TMEDA	52
3	Et_3N	54
4	TMPDA	73

[a] Reaction condition: **1a** (0.2 mmol, 1.0 equiv.), *tert*-dodecylthiol (0.4 mmol, 2.0 equiv.), and base (0.5 mmol, 2.5 equiv.) in CF_3CH_2OH (20 mL) were irradiated with 40 W 390 nm LED light at r.t. for 6 h under ethylene (1 atm). [b]Yields of isolated products.

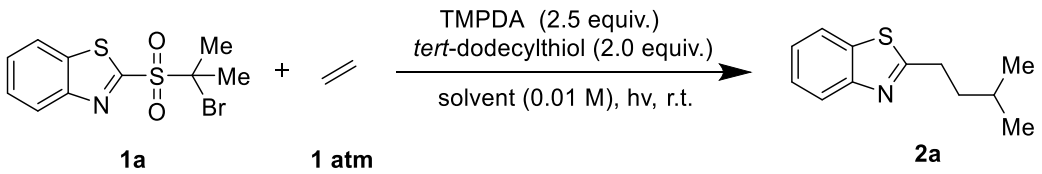
Table S3. Evaluation of the amount of base [a]



entry	equiv.	yield (%) [b]
1	3.0	69
2	2.5	73
3	2.0	71
4	1.5	68
5	1.0	61

[a] Reaction condition: **1a** (0.2 mmol, 1.0 equiv.), *tert*-dodecylthiol (0.4 mmol, 2.0 equiv.), and TMPDA (x equiv.) in CF_3CH_2OH (20 mL) were irradiated with 40 W 390 nm LED light at r.t. for 6 h under ethylene (1 atm). [b]Yields of isolated products.

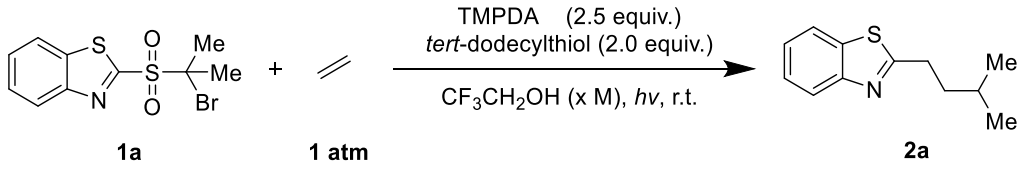
Table S4. Evaluation of solvents [a]



entry	solvent	yield (%) [b]
1	CF_3CH_2OH	73
2	MeOH	36
3	CH_3CN	43
4	DMSO	33
5	DMF	45
6	CCl_4	0
7	DCM	10
8	DCE	trace

[a] Reaction condition: **1a** (0.2 mmol, 1.0 equiv.), *tert*-dodecylthiol (0.4 mmol, 2.0 equiv.), and TMPDA (0.5 mmol, 2.5 equiv.) in solvent (20 mL) were irradiated with 40 W 390 nm LED light at r.t. for 6 h under ethylene (1 atm). [b]Yields of isolated products.

Table S5. Evaluation of concentration [a]



1a **1 atm** **2a**

entry	solvent	yield (%) [b]
1	CF_3CH_2OH (0.1 M)	45
2	CF_3CH_2OH (0.05 M)	56
3	CF_3CH_2OH (0.025 M)	60
4	CF_3CH_2OH (0.01 M)	73

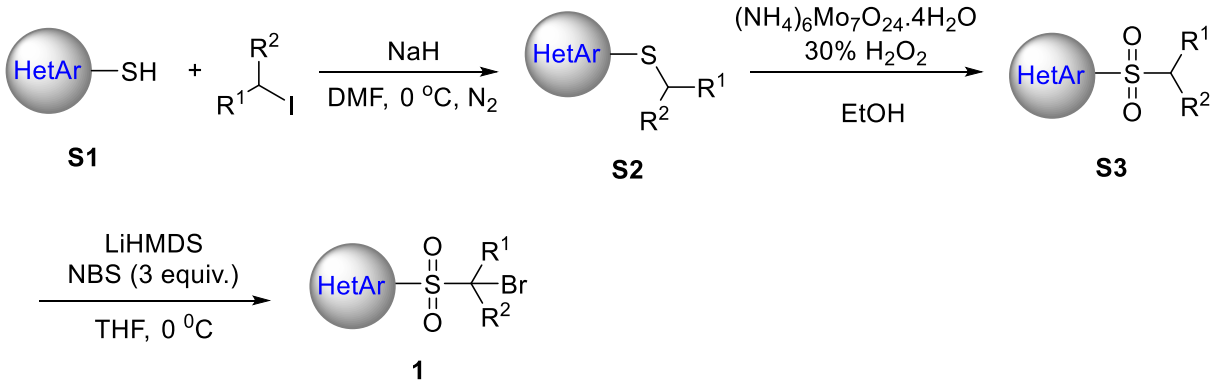
[a] Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), *tert*-dodecylthiol (0.4 mmol, 2.0 equiv.), and TMPDA (0.5 mmol, 2.5 equiv.) in CF_3CH_2OH were irradiated with 40 W 390 nm LED light at r.t. for 6 h under ethylene (1 atm). [b]Yields of isolated products.

3. Preparation of starting materials

The bifunctional reagents were synthesized according to the following procedures:

3.1 Synthesis of bifunctional heteroaryl-alkylating reagents 1

Method A



S1 **S2** **S3** **1**

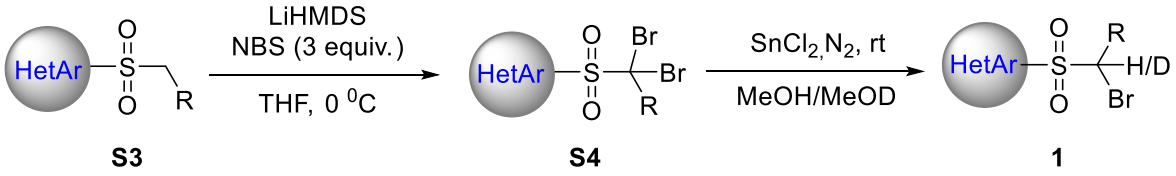
Figure S2. Synthetic approaches for bifunctional reagents 1b-1j and 1u

First Step: A dry flask charged with **S1** (5 mmol) and NaH (1.18 eq, 5.9 mmol) was evacuated and backfilled with N_2 for 3 times. DMF (18 mL) was added at 0 °C, and the mixture was stirred for 30 min. Then alkyl iodide (1.18 eq, 5.9 mmol) was added dropwise with syringe at 0 °C, and the mixture was stirred at rt for 12 h. After the reaction was complete, the mixture was quenched with H_2O at 0 °C. The mixture was extracted with Et_2O for 3 times. Then the organic phase was combined and dried over anhydrous $MgSO_4$. The solvent was removed under vacuum to afford the crude product **S2** which was used in next step without further purification.

Second step: A solution of **S2** in EtOH (18 mL) was treated with $(NH_4)_6Mo_7O_{24}•4H_2O$ (0.1 equiv., 0.5 mmol) followed by aq. H_2O_2 (7 equiv., 30 wt.%, 35 mmol) and stirred for 75 h at rt. After the reaction was complete, the mixture was quenched with saturated aq. $Na_2S_2O_3$ for 1 h. After filtration, the residue was washed with ethyl acetate. The filtrate was extracted with ethyl acetate for 3 times. Then the organic phase was combined and dried over anhydrous $MgSO_4$. The solvent was removed under vacuum and the residue was purified by flash column chromatography on silica gel to provide **S3**.

Third Step: Sulfone **S3** (5 mmol, 1.0 equiv.) and NBS (15 mmol, 3.0 equiv.) were loaded in a dry Schlenk tube. The flask was evacuated and backfilled with N_2 for 3 times. Then THF (20 mL) was added with syringe. The mixture was cooled to 0 °C. LiHMDS (1.0 M in THF, 15 mmol, 15 mL, 3.0 equiv.) was added with syringe in 15 min. The mixture was stirred at 0 °C for 5 h. After the reaction was complete, the mixture was quenched by saturated aq. NH_4Cl. The aqueous layer was extracted with Et_2O for 3 times. Then the organic phase was combined and dried over anhydrous $MgSO_4$. The solvent was removed under vacuum and the residue was purified by flash column chromatography on silica gel to provide bifunctional reagent **1**.

Method B



S3 **S4** **1**

Figure S3. Synthetic approaches for bifunctional reagents 1m-1q

First Step: Sulfone **S3** (5 mmol, 1.0 equiv.) and NBS (15 mmol, 3.0 equiv.) were added to a dry Schlenk tube. The flask was evacuated and backfilled with N_2 for 3 times. Then THF (20 mL) was added with syringe. The mixture was cooled to 0 °C. LiHMDS (1.0 M in THF, 15 mmol, 15 mL, 3.0 equiv.) was added with syringe in 15 min. The mixture was stirred at 0 °C for 5 h. After the reaction was complete, the mixture was quenched by saturated aq. NH_4Cl. The aqueous layer was extracted with Et_2O for 3 times. Then the organic phase was combined and dried over anhydrous $MgSO_4$. The solvent was removed under vacuum and the residue was purified by flash column chromatography on silica gel to provide **S4**.

Second Step: A dry flask was evacuated and backfilled with N_2 for 3 times. **S4** (0.52 mmol) and $SnCl_2$ (0.78 mmol) followed by MeOH/MeOD (1.5 mL) were added at rt. The mixture was stirred for 4 h. The solvent was removed under vacuum and the residue was purified by flash column chromatography on silica gel to afford bifunctional reagent **1 .**

Method C

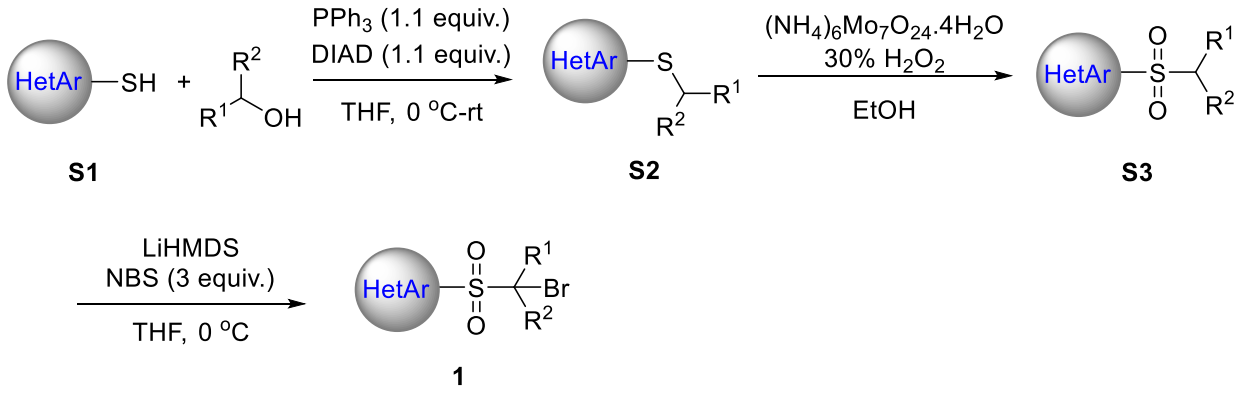
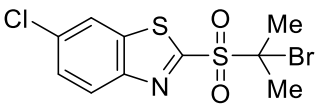


Figure S4. Synthetic approaches for bifunctional reagents 1k, 1s, 1v and 1w

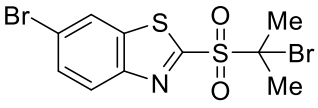
First Step: A flask was charged with alcohol (10 mmol, 1.0 equiv.), triphenyl phosphine (11 mmol, 1.1 equiv.) and **S1** (11 mmol, 1.1 equiv.). The flask was evacuated and backfilled with N_2. Anhydrous THF (0.2 M) was added, and the mixture was cooled to 0 ℃. DIAD (11 mmol, 1.1 equiv.) was added dropwise, and the reaction mixture was warmed to room temperature and stir overnight. The mixture was concentrated and purified by flash column chromatography on silica gel to afford thioether **S2**.

The following second and third steps are same as ***Method A***.

Bifunctional reagent **1a**, **1g**, **1h**, **1l**, **1r, 1t and 1x** are known compounds, which are prepared according to the reported procedures.[1] Bifunctional reagent **1b-1f**, **1i-1k**, **1m-1q, 1s, 1u-1w** are new compounds, which are synthesized by the above methods.

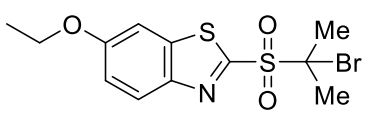


1b was prepared by **Method A**, white solid, m.p. 134-135 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 9.2 Hz, 1H), 8.02 (d, *J* = 2.0 Hz, 1H), 7.61 (dd, *J* = 8.8, 2.8 Hz, 1H), 2.26 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 151.2, 138.8, 134.8, 128.9, 126.6, 121.7, 74.1, 28.2. FT-IR: ν (cm^{-1}) 2928, 2343, 1820, 1636, 1473, 1234. HRMS [EI] calcd for $C_{10}H_9BrClNO_2S_2$ $[M]^+$ 352.8947, found 352.8951.

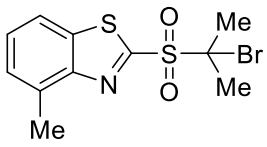


1c was prepared by **Method A**, white solid, m.p. 123-124 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 2.0 Hz, 1H), 8.12 (d, *J* = 8.8 Hz, 1H), 7.76 (dd, *J* =

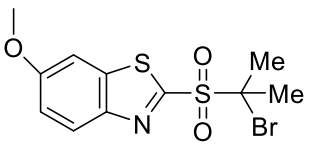
8.8, 2.0 Hz, 1H), 2.27 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 151.5, 139.2, 131.5, 126.9, 124.7, 122.7, 74.1, 28.2. FT-IR: ν (cm^{-1}) 2988, 2341, 1558, 1457, 1395, 1318. HRMS [EI] calcd for $C_{10}H_9Br_2NO_2S_2$ $[M]^+$ 396.8441, found 396.8450.



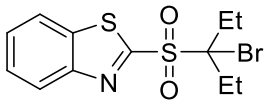
1d was prepared by **Method A**, white solid, m.p. 121-122 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 9.2 Hz, 1H), 7.37 (d, *J* = 2.4 Hz, 1H), 7.22 (dd, *J* = 9.2, 2.4 Hz, 1H), 4.13 (q, *J* = 6.8 Hz, 2H), 2.24 (s, 6H), 1.48 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 157.8, 147.3, 139.9, 126.5, 118.8, 103.6, 74.4, 64.4, 28.3, 14.7. FT-IR: ν (cm^{-1}) 2988, 2901, 2342, 1653, 1648, 1375. HRMS [ESI] calcd for $C_{12}H_{14}BrNNaO_3S_2$ $[M+Na]^+$ 385.9491, found 385.9485.



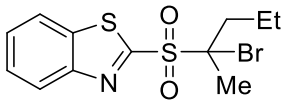
1e was prepared by **Method A**, yellow solid, m.p. 127-128 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.4 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.45-7.41 (m, 1H), 2.81 (s, 3H), 2.28 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.4, 152.6, 137.6, 136.2, 128.4, 127.9, 119.4, 74.3, 28.3, 18.3. FT-IR: ν (cm^{-1}) 2360, 2341, 1829, 1772, 1671, 1250. HRMS [ESI] calcd for $C_{11}H_{12}BrNNaO_2S_2$ $[M+Na]^+$ 355.9385, found 355.9382.



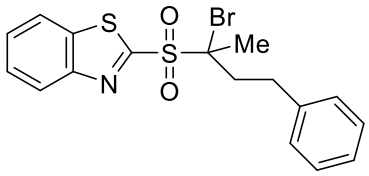
1f was prepared by **Method A**, yellow solid, m.p. 145-146 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.8 Hz, 1H), 7.40 (d, *J* = 2.8 Hz, 1H), 7.26-7.20 (m, 1H), 3.93 (s, 3H), 2.25 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 158.0, 147.4, 139.9, 126.5, 118.5, 103.0, 74.3, 56.0, 28.3. FT-IR: ν (cm^{-1}) 3236, 2963, 2471, 1478, 1364, 1230. HRMS [ESI] calcd for $C_{11}H_{12}BrNNaO_3S_2$ $[M+Na]^+$ 371.9334, found 371.9340.



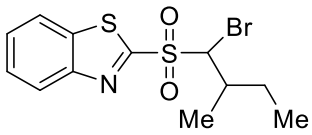
1i was prepared by **Method A**, yellow solid, m.p. 132- 133 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30-8.22 (m, 1H), 8.05-7.99 (m, 1H), 7.69-7.57 (m, 2H), 2.65-2.53 (m, 2H), 2.51-2.40 (m, 2H), 1.22 (t, *J* = 7.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 152.6, 137.7, 128.2, 127.6, 125.9, 122.1, 87.3, 29.7, 10.2. FT-IR: ν (cm^{-1}) 3446, 2987, 2360, 1636, 1362, 1318. HRMS [ESI] calcd for $C_{12}H_{14}BrNNaO_2S_2$ $[M+Na]^+$ 369.9542, found 369.9540.



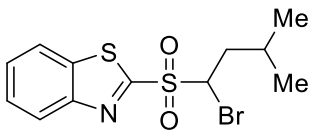
1j was prepared by **Method A**, yellow solid, m.p. 130- 131 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31-8.24 (m, 1H), 8.06-8.01 (m, 1H), 7.70-7.58 (m, 2H), 2.45-2.34 (m, 1H), 2.32-2.22 (m, 1H), 2.21 (s, 3H), 1.78-1.54 (m, 2H), 1.03 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2, 152.8, 137.8, 128.3, 127.7, 125.9, 122.1, 80.7, 39.8, 25.2, 18.7, 13.9. FT-IR: ν (cm^{-1}) 2966, 2934, 1724, 1462, 1326, 1311. HRMS [EI] calcd for $C_{12}H_{14}BrNO_2S_2$ $[M]^+$ 346.9649, found 369.9650.



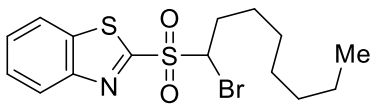
1k was prepared by **Method C**, white solid, m.p. 155-156 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30-8.25 (m, 1H), 8.06-8.01 (m, 1H), 7.70-7.58 (m, 2H), 7.34-7.28 (m, 2H), 7.25-7.20 (m, 3H), 3.10-2.98 (m, 1H), 2.97-2.84 (m, 1H), 2.80-2.69 (m, 1H), 2.66-2.56 (m, 1H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0, 152.8, 139.7, 137.8, 128.7, 128.5, 128.3, 127.7, 126.6, 125.9, 122.1, 80.1, 39.9, 31.7, 25.5. FT-IR: ν (cm^{-1}) 2989, 2932, 1463, 1328, 1298, 1250. HRMS [EI] calcd for $C_{17}H_{16}BrNO_2S_2$ $[M]^+$ 408.9806, found 408.9812.



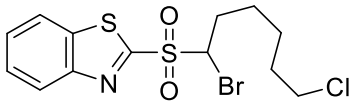
1m was prepared by **Method B**, yellow solid, *d.r.*=1:1, m.p. 120-121 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27-8.21 (m, 1H, two isomers), 8.03 (d, *J* = 7.6 Hz, 1H, two isomers), 7.70-7.58 (m, 2H, two isomers), 5.50 (d, *J* = 2.0 Hz, 0.5H, one isomer), 5.42 (d, *J* = 2.4 Hz, 0.5H one isomer), 2.65-2.48 (m, 1H, two isomers), 2.12-2.00 (m, 0.5H, one isomer), 1.55-1.49 (m, 0.5H, one isomer), 1.48-1.36 (m, 1H, two isomers), 1.25 (d, *J* = 6.4 Hz, 1.5H, one isomer), 1.19 (d, *J* = 6.8 Hz, 1.5H, one isomer), 1.04-0.94 (m, 3H, two isomers); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4 & 164.2 (two isomers), 152.7 (overlap, two isomers), 137.4 & 137.4 (two isomers), 128.3 (overlap, two isomers), 127.8 (overlap, two isomers), 125.7 & 125.6 (two isomers), 122.3 (overlap, two isomers), 70.6 & 69.7 (two isomers), 35.5 & 34.6 (two isomers), 28.6 & 25.3 (two isomers), 17.6 & 15.7 (two isomers), 11.4 & 11.3 (two isomers). FT-IR: ν (cm^{-1}), 2960, 2924, 1722, 1530, 1350, 1322. HRMS [EI] calcd for $C_{12}H_{14}BrNO_2S_2$ $[M]^+$ 346.9649, found 369.9655.



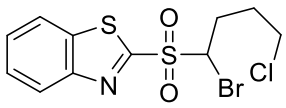
1n was prepared by **Method B,** white solid, m.p. 122-124 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29-8.23 (m, 1H), 8.06-8.01 (m, 1H), 7.70-7.58 (m, 2H), 5.35-5.28 (m, 1H), 2.27-2.13 (m, 2H), 2.05-1.93 (m, 1H), 1.05 (d, *J* = 6.8 Hz, 3H), 0.94 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0, 152.7, 137.3, 128.3, 127.8, 125.7, 122.3, 63.0, 38.3, 26.0, 23.1, 20.2. FT-IR: ν (cm^{-1}) 2961, 2341, 1716, 1436, 1318, 1251. HRMS [ESI] calcd for $C_{12}H_{14}BrNO_2S_2$ $[M]^+$ 346.9649, found 346.9652.



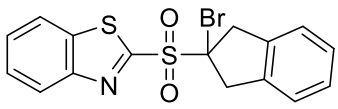
1o was prepared by **Method B,** white solid, m.p. 108-109 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27-8.22 (m, 1H), 8.05-8.01 (m, 1H), 7.70-7.58 (m, 2H), 5.29 (dd, *J* = 10.8, 2.8 Hz, 1H), 2.57-2.45 (m, 1H), 2.25-2.10 (m, 1H), 1.80-1.62 (m, 1H), 1.57-1.45 (m, 1H), 1.40-1.20 (m, 8H), 0.93-0.82 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0, 152.7, 137.3, 128.3, 127.8, 125.7, 122.3, 64.3, 31.6, 30.3, 28.8, 28.4, 26.8, 22.6, 14.1. FT-IR: ν (cm^{-1}) 2988, 2901, 1671, 1418, 1339, 1250. HRMS [ESI] calcd for $C_{15}H_{20}BrNNaO_2S_2$ $[M+Na]^+$ 412.0011, found 412.0011.



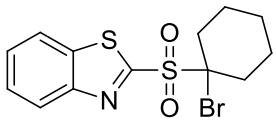
1p was prepared by **Method B**, white solid, m.p. 101-102 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26-8.20 (m, 1H), 8.06-8.00 (m, 1H), 7.70-7.58 (m, 2H), 5.29 (dd, *J* = 10.8, 3.2 Hz, 1H), 3.57-3.49 (m, 2H), 2.60-2.45 (m, 1H), 2.30-2.10 (m, 1H), 1.86-1.65 (m, 3H), 1.62-1.42 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.8, 152.7, 137.3, 128.4, 127.9, 125.7, 122.4, 63.9, 44.7, 32.0, 30.4, 26.2, 25.8. FT-IR: ν (cm^{-1}) 2990, 2359, 1645, 1556, 1432, 1311. HRMS [EI] calcd for $C_{13}H_{15}BrClNO_2S_2$ $[M]^+$ 394.9416, found 394.9401.



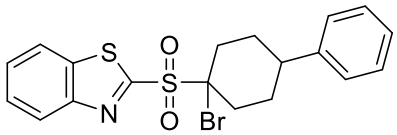
1q was prepared by **Method B**, white solid, m.p. 101-102 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28-8.23 (m, 1H)，8.07-8.02 (m, 1H), 7.70-7.60 (m, 2H), 5.34 (dd, *J* = 10.4, 3.6 Hz, 1H), 3.62 (t, *J* = 6.4 Hz, 2H), 2.80-2.67 (m, 1H), 2.44-2.32 (m, 1H), 2.30-2.18 (m, 1H), 2.10-1.97 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6, 152.7, 137.3, 128.4, 127.9, 125.7, 122.3, 63.1, 43.3, 29.7, 28.4. FT-IR: ν (cm^{-1}) 2988, 2361, 1653, 1473, 1395, 1318. HRMS [ESI] calcd for $C_{11}H_{11}BrClNNaO_2S_2$ $[M+Na]^+$ 389.8995, found 389.8989.



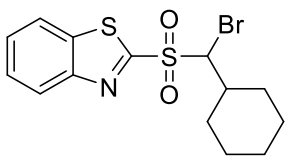
1s was prepared by **Method C**, white solid, m.p. 148-149 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35-8.27 (m, 1H), 8.09-8.03 (m, 1H), 7.75-7.60 (m, 2H), 7.34-7.22 (m, 4H), 4.51 (d, *J* = 17.6 Hz, 2H), 3.73 (d, *J* = 17.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 152.9, 137.6, 137.4, 128.4, 127.8, 127.8, 125.9, 124.7, 122.2, 80.5, 46.3. FT-IR: ν (cm^{-1}) 2998, 2361, 1554, 1415, 1315, 1228. HRMS [ESI] calcd for $C_{16}H_{12}BrNNaO_2S_2$ $[M+Na]^+$ 415.9385, found 415.9384.



1u was prepared by **Method A**, yellow solid, m.p. 132- 133 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29-8.24 (m, 1H), 8.05-8.00 (m, 1H), 7.68-7.57 (m, 2H), 2.51-2.42 (m, 2H), 2.37-2.26 (m, 2H), 1.92-1.82 (m, 2H), 1.81-1.67 (m, 3H), 1.34-1.20 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2, 152.7, 137.7, 128.2, 127.6, 125.8, 122.1, 84.4, 33.4, 24.3, 22.0. FT-IR: ν (cm^{-1}) 3503, 2987, 2360, 1558, 1418, 1339. HRMS [ESI] calcd for $C_{13}H_{14}BrNNaO_2S_2$ $[M+Na]^+$ 381.9542, found 381.9548.



1v was prepared by **Method C**, white solid, m.p. 128-129 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25-8.20 (m, 1H), 8.02-7.97 (m, 1H), 7.67-7.55 (m, 2H), 7.30-7.22 (m, 4H), 7.18-7.10 (m, 1H), 3.04-2.88 (m, 3H), 2.50-2.36 (m, 4H), 2.14-1.98 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9, 152.6, 143.6, 137.6, 128.5, 128.2, 127.6, 127.0, 126.3, 125.8, 122.1, 81.5, 38.5, 33.8, 28.9. FT-IR: ν (cm^{-1}) 2925, 2866, 1722, 1457, 1330, 1249. HRMS [EI] calcd for $C_{19}H_{18}BrNO_2S_2$ $[M]^+$ 434.9962, found 434.9953.



1w was prepared by **Method C**, white solid, m.p. 168-169 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26-8.20 (m, 1H), 8.06-8.01 (m, 1H), 7.69-7.59 (m, 2H), 5.37 (d, *J* = 2.4 Hz, 1H), 2.60- 2.45 (m, 1H), 2.25-2.10 (m, 1H), 1.92-1.77 (m, 2H), 1.76-1.65 (m, 2H), 1.56-1.32 (m, 4H), 1.25-1.11 (m, 1H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.3, 152.6, 137.4, 128.2, 127.8, 125.6, 122.3, 70.3, 37.8, 31.5, 28.6, 26.0, 25.5, 25.3. FT-IR: ν (cm^{-1}) 2988, 2341, 1508, 1419, 1318, 1250. HRMS [ESI] calcd for $C_{14}H_{16}BrNO_2S_2$ $[M]^+$ 372.9806, found 372.9815.

3.2 Synthesis of bifunctional oximino-alkylating reagent 1y

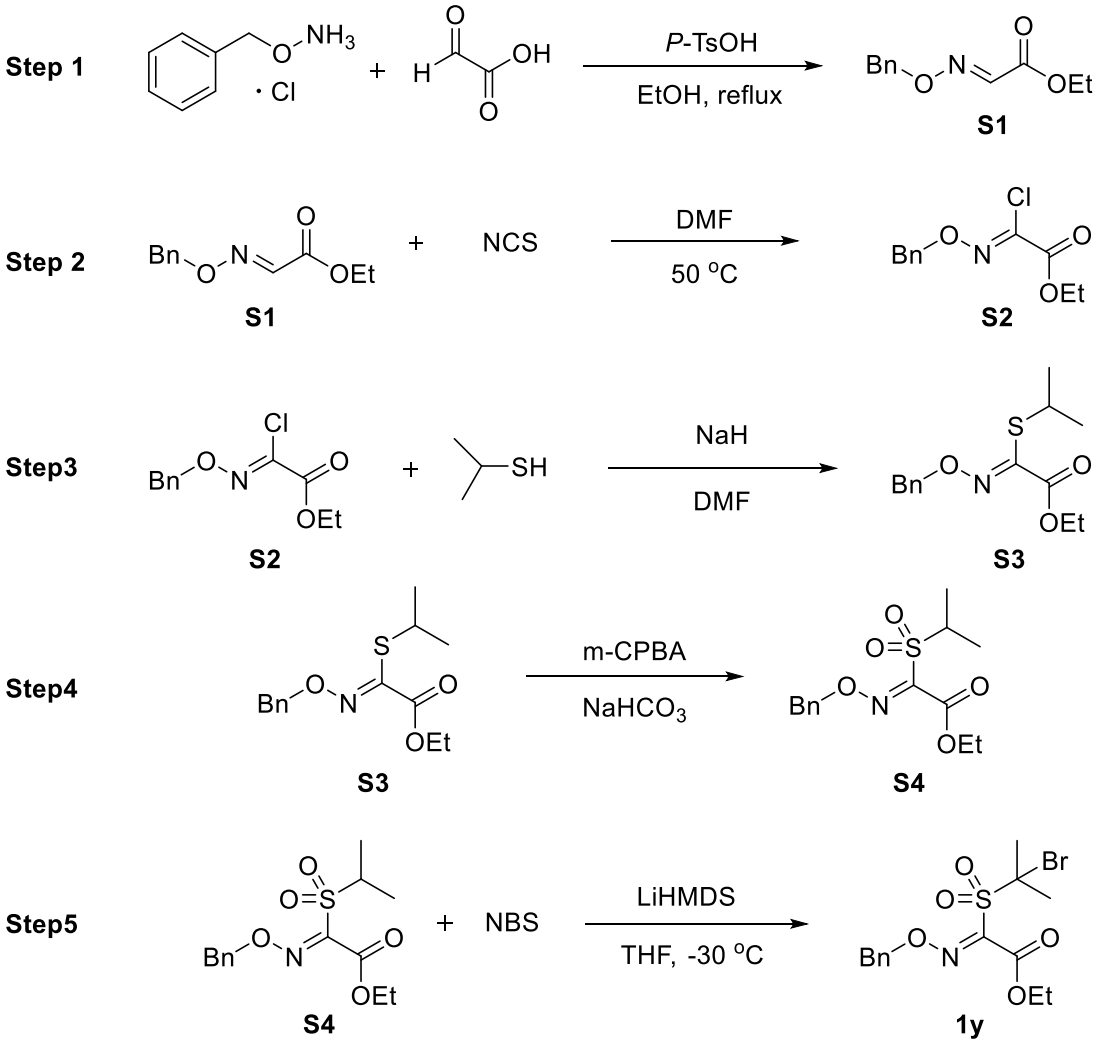


Figure S5. Synthetic approaches for bifunctional reagent 1y

Step 1: Glyoxylic acid (100 mmol, 1 equiv.) was dissolved in ethanol. O-Benzylhydroxylamine hydrochloride (1 equiv.) and *p*-TsOH (0.1 equiv.) were then added. The mixture was heated at reflux for 5 h. A Dean-Stark apparatus was then installed above the flask and ethanol was progressively distilled off. After an hour, most of

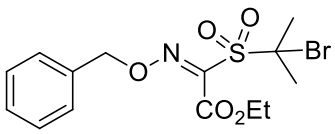
the ethanol was removed and heating was stopped. The crude mixture was diluted with DCM. The organic phase was successively washed with saturated $NaHCO_3$ and the resulting aqueous phase was back-extracted with DCM. The combined organic extracts were washed with saturated aqueous NaCl, dried over $MgSO_4$, filtered and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel to provide **S1**.

Step 2: A mixture of **S1** (60 mmol, 1.0 equiv.) and *N*-chlorosuccinimide (3.0 equiv.) in DMF was stirred at 50 °C for 2 days. The mixture was diluted with Et_2O, washed with brine, dried over $MgSO_4$, filtered and concentrated in vacuo. Crude **S2** was afforded and directly used in the next step without further purification.

Step 3: Isopropyl thiol (30mmol, 1.0 equiv.) was added to a slurry of NaH (1.0 equiv) in DMF at 0 °C. After being stirred at 0 °C for 30 min, **S2** (1.2 equiv.) was added to the reaction mixture at 0 °C. The resulting reaction mixture was stirred at room temperature for 5 h, treated with NH_4Cl (aq), diluted with Et_2O, washed with brine, dried over MgSO4, filtered and concentrated in vacuo. Crude **S3** was afforded and directly used in the next step without further purification.

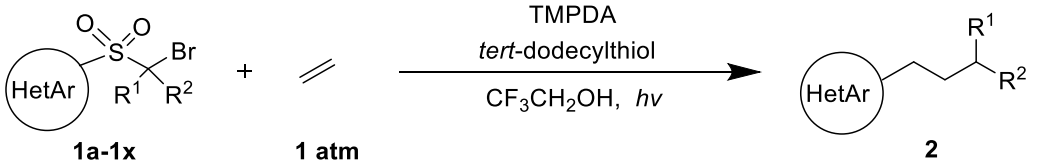
Step 4: A solution of **S3** (20 mmol, 1.0 equiv.) in CH_2Cl_2 was treated with $NaHCO_3$ (3.0 equiv.) and *m*-CPBA (3.0 equiv.) at 0 °C. After being stirred at room temperature for 6 h, the reaction mixture was diluted with CH_2Cl_2, washed with 10% $Na_2S_2O_3$ (aq), $NaHCO_3$ (aq), and brine, dried over $MgSO_4$, and concentrated. The residue was purified by flash column chromatography on silica gel to provide **S4**.

Step 5: A solution of **S4** (15 mmol, 1.0 equiv.) and NBS (1.2 equiv.) in dry THF was stired at -30 °C, and then LiHMDS (1.5 equiv.) was added dropwise into the mixture under N_2. The resulting reaction mixture was stirred at -30 °C for 4 h, treated with NH_4Cl (aq), diluted with ethyl acetate, washed with brine, dried over $MgSO_4$ and concentrated. The residue was purified by flash column chromatography on silica gel to give bifunctional reagent **1y**.



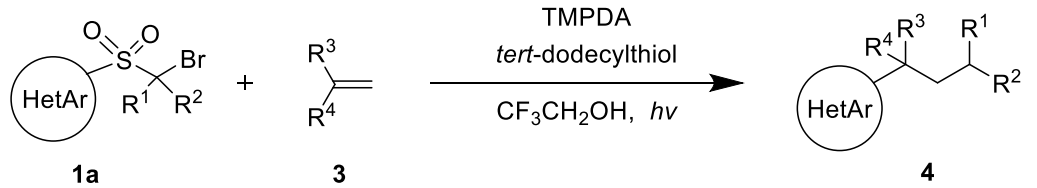
1y: yellow solid, m.p. 29-30 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.34 (m, 5H), 5.41 (s, 2H), 4.37 (q, *J* = 7.2 Hz, 2H), 2.06 (s, 6H), 1.37 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 142.4, 134.5, 129.0, 128.7, 128.7, 80.7, 76.4, 63.6, 28.6, 13.9. FT-IR: ν (cm^{-1}) 3035, 2995, 2967, 2940, 2901, 1965, 1733, 1565, 1497, 1469. HRMS [ESI] calcd for $C_{14}H_{18}O_5BrNSNa$ $[M+Na]^+$ 413.9981, found 413.9983.

4. General procedures for radical alkylfunctionalization of alkenes



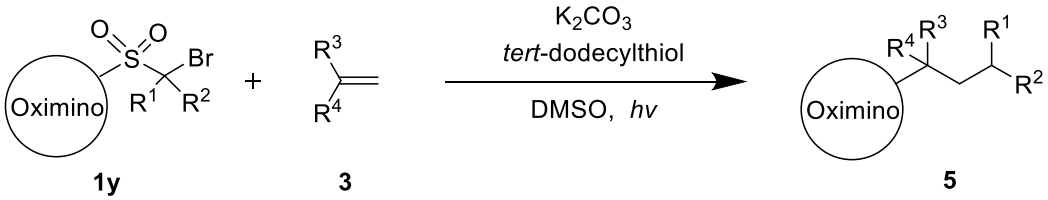
Procedure A

1 (0.2 mmol, 1.0 equiv.) was loaded in a Schlenk tube charged with a balloon, which was subjected to evacuation/flushing with ethylene for three times. Then TMPDA (0.5 mmol, 2.5 equiv.), *tert*-dodecylthiol (0.4 mmol, 2.0 equiv.) and CF_3CH_2OH (20 mL) were added to the mixture via syringe. The reaction was irradiated with 40 W 390 nm LED light under gaseous ethylene (1 atm) and stirred at r.t. until the starting material had been consumed as determined by TLC. The mixture was extracted with ethyl acetate. The combined organic extracts were washed by brine, dried over Na_2SO_4, filtered, concentrated, and purified by flash column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether) to give the corresponding product **2**.



Procedure B

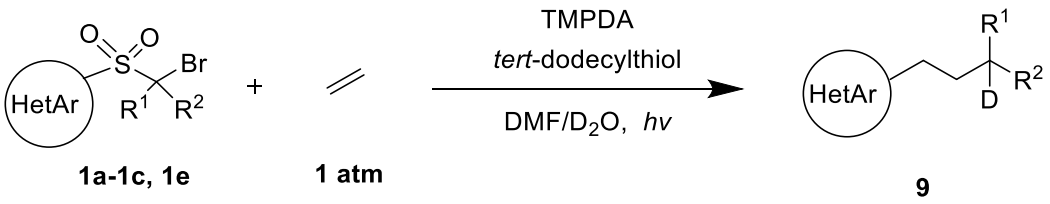
1 (0.2 mmol, 1.0 equiv.) were loaded in a flask, which was subjected to evacuation/flushing with N_2 for three times. Then alkene **3** (0.6 mmol, 3.0 equiv.), TMPDA (0.5 mmol, 2.5 equiv.), *tert*-dodecylthiol (0.4 mmol, 2.0 equiv.) and CF_3CH_2OH (8 mL) were added to the mixture via syringe, which was irradiated with 40 W 390 nm LED light and stirred at r.t. until the starting material had been consumed as determined by TLC. The mixture was extracted with ethyl acetate. The combined organic extracts were washed by brine, dried over Na_2SO_4, filtered, concentrated, and purified by flash column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether) to give the corresponding product **4.**



Procedure C

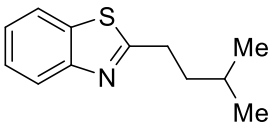
1 (0.2 mmol, 1.0 equiv.) were loaded in a flask, which was subjected to evacuation/flushing with N_2 for three times. Then alkene **3** (0.6 mmol, 3.0 equiv.), k_2CO_3 (0.5 mmol, 2.5 equiv.), *tert*-dodecylthiol (0.6 mmol, 3.0

equiv.) and DMSO (8 mL) were added to the mixture via syringe, which was irradiated with 40 W 390 nm LED light and stirred at r.t. until the starting material had been consumed as determined by TLC. The mixture was extracted with ethyl acetate. The combined organic extracts were washed by brine, dried over Na_2SO_4, filtered, concentrated, and purified by flash column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether) to give the corresponding product **5**.

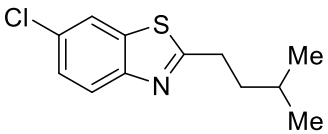


Procedure D

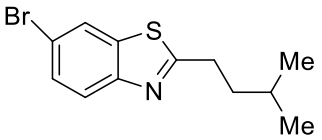
1 (0.2 mmol, 1.0 equiv.) was loaded in a Schlenk tube charged with a balloon, which was subjected to evacuation/flushing with ethylene for three times. Then TMPDA (0.5 mmol, 2.5 equiv.), *tert*-dodecylthiol (0.4 mmol, 2.0 equiv.), DMF (18 mL) and D_2O (2 mL) were added to the mixture via syringe. The reaction was irradiated with 40 W 390 nm LED light under gaseous ethylene (1 atm) and stirred at r.t. until the starting material had been consumed as determined by TLC. The mixture was extracted with ethyl acetate. The combined organic extracts were washed by brine, dried over Na_2SO_4, filtered, concentrated, and purified by flash column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether) to give the corresponding product **9.**



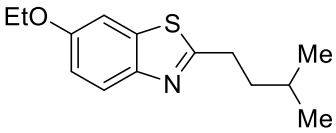
2a was prepared by **Procedure A:** 30.0 mg, 73%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.46-7.41 (m, 1H), 7.36-7.30 (m, 1H), 3.15-3.09 (m, 2H), 1.82-1.65 (m, 3H), 0.98 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 153.3, 135.1, 125.9, 124.6, 122.5, 121.5, 38.6, 32.4, 27.8, 22.4. FT-IR: ν (cm^{-1}) 2964, 2921, 1654, 1383, 1362, 1240. HRMS [ESI] calcd for $C_{12}H_{16}NS$ $[M+H]^+$ 206.0998, found 206.0999.



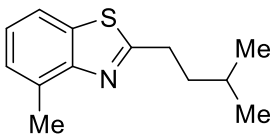
2b was prepared by **Procedure A**: 33.6 mg, 70%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.8 Hz, 1H), 7.80 (d, *J* = 2.0 Hz, 1H), 7.39 (dd, *J* = 8.8, 2.0 Hz, 1H), 3.12- 3.07 (m, 2H), 1.90-1.65 (m, 3H), 0.97 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 151.8, 136.3, 130.5, 126.6, 123.2, 121.1, 38.5, 32.4, 27.8, 22.3. FT-IR: ν (cm^{-1}) 2956, 2869, 2360, 1467, 1300, 1235. HRMS [ESI] calcd for $C_{12}H_{15}ClNS$ $[M+H]^+$ 240.0608, found 240.0604.



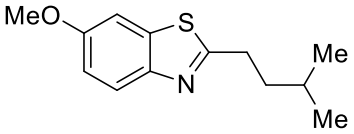
2c was prepared by **Procedure A**: 37.5 mg, 66%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 2.0 Hz, 1H), 7.80 (d, *J* = 8.8 Hz, 1H), 7.54 (dd, *J* = 8.8, 2.0 Hz, 1H), 3.12-3.07 (m, 2H), 1.80-1.62 (m, 3H), 0.98 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2, 152.1, 136.8, 129.3, 124.0, 123.6, 118.1, 38.5, 32.4, 27.8, 22.3. FT-IR: ν (cm^{-1}) 2955, 2360, 1587, 1435, 1320, 1235. HRMS [EI] calcd for $C_{12}H_{14}BrNS$ $[M]^+$ 283.0025, found 283.0026.



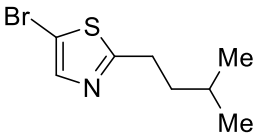
2d was prepared by **Procedure A**: 32.4 mg, 65%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.8 Hz, 1H), 7.29 (d, *J* = 2.4 Hz, 1H), 7.03 (dd, *J* = 8.8, 2.4 Hz, 1H), 4.09 (q, *J* = 6.8 Hz, 2H), 3.10-3.04 (m, 2H), 1.79-1.62 (m, 3H), 1.47-1.42 (m, 3H), 0.97 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 156.6, 147.7, 136.3, 122.8, 115.4, 105.0, 64.1, 38.6, 32.3, 27.8, 22.3, 14.8. FT-IR: ν (cm^{-1}) 2956, 2340, 1560, 1430, 1278, 1230. HRMS [EI] calcd for $C_{14}H_{19}NOS$ $[M]^+$ 249.1182, found 249.1183.



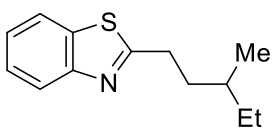
2e was prepared by **Procedure A**: 33.4 mg, 76%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71-7.66 (m, 1H), 7.29-7.22 (m, 2H), 3.19-3.13 (m, 2H), 2.76 (s, 3H), 1.83-1.70 (m, 3H), 1.01 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 152.6, 135.0, 132.4, 126.4, 124.4, 118.9, 38.9, 32.6, 27.9, 22.4, 18.6. FT-IR: ν (cm^{-1}) 2955, 2360, 1577, 1402, 1263, 1238. HRMS [ESI] calcd for $C_{13}H_{18}NS$ $[M+H]^+$ 220.1154, found 220.1159.



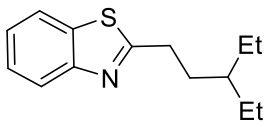
2f was prepared by **Procedure A**: 31.1 mg, 66%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.8 Hz, 1H), 7.29 (d, *J* = 2.4 Hz, 1H), 7.04 (dd, *J* = 8.8, 2.8 Hz, 1H), 3.86 (s, 3H), 3.10-3.04 (m, 2H), 1.79-1.64 (m, 3H), 0.97 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 157.3, 147.7, 136.3, 122.9, 114.9, 104.2, 55.8, 38.6, 32.3, 27.8, 22.4. FT-IR: ν (cm^{-1}) 2955, 2360, 1984, 1385, 1314, 1226. HRMS [ESI] calcd for $C_{13}H_{18}NOS$ $[M+H]^+$ 236.1104, found 236.1094.



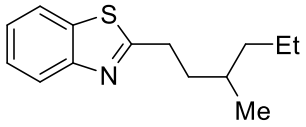
2g was prepared by **Procedure A**: 38.9 mg, 83%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (s, 1H), 3.00-2.92 (m, 2H), 1.70-1.60 (m, 3H), 0.94 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.4, 143.3, 107.0, 38.6, 31.9, 27.6, 22.3. FT-IR: ν (cm^{-1}) 2956, 2341, 1557, 1466, 1385, 1214. HRMS [ESI] calcd for $C_{11}H_{14}NS$ $[M+H]^+$ 192.0841, found 192.0844.



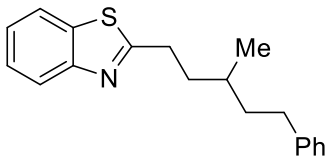
2h was prepared by **Procedure A**: 28.5 mg, 65%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 1H), 7.85-7.81 (m, 1H), 7.47-7.41 (m, 1H), 7.37-7.31 (m, 1H), 3.20-3.03 (m, 2H), 1.97-1.85 (m, 1H), 1.76-1.64 (m, 1H), 1.54-1.38 (m, 2H), 1.28-1.18 (m, 1H), 0.96 (d, *J* = 6.4 Hz, 3H), 0.90 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 153.3, 135.1, 125.9, 124.6, 122.5, 121.5, 36.4, 34.1, 32.2, 29.2, 18.9, 11.3. FT-IR: ν (cm^{-1}) 2959, 2872, 2360, 1794, 1311, 1243. HRMS [ESI] calcd for $C_{13}H_{18}NS$ $[M+H]^+$ 220.1154, found 220.1159.



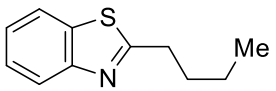
2i was prepared by **Procedure A**: 21.9 mg, 47%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 3.14-3.07 (m, 2H), 1.89-1.80 (m, 2H), 1.44-1.24 (m, 5H), 0.89 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 153.3, 135.1, 125.9, 124.6, 122.5, 121.5, 40.0, 32.8, 31.9, 25.1, 10.8. FT-IR: ν (cm^{-1}) 2958, 2360, 1520, 1400, 1321, 1242. HRMS [ESI] calcd for $C_{14}H_{20}NS$ $[M+H]^+$ 234.1311, found 234.1310.



2j was prepared by **Procedure A**: 34.1 mg, 73%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.47-7.41 (m, 1H), 7.37-7.31 (m, 1H), 3.24-3.04 (m, 2H), 1.96-1.85 (m, 1H), 1.77-1.51 (m, 1H), 1.40-1.12 (m, 5H), 0.96 (d, *J* = 6.4 Hz, 3H), 0.92-0.85 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 153.3, 135.1, 125.9, 124.6, 122.5, 121.5, 39.0, 36.8, 32.3, 32.1, 20.0, 19.4, 14.3. FT-IR: ν (cm^{-1}) 2956, 2361, 1520, 1378, 1278, 1243. HRMS [ESI] calcd for $C_{14}H_{20}NS$ $[M+H]^+$ 234.1311, found 234.1313.

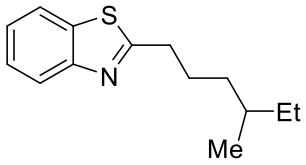


2k was prepared by **Procedure A**: 24.8 mg, 42%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 1H), 7.86-7.81 (m, 1H), 7.48-7.42 (m, 1H), 7.37-7.32 (m, 1H), 7.29-7.23 (m, 2H), 7.20-7.13 (m, 3H), 3.23-3.03 (m, 2H), 2.74-2.54 (m, 2H), 2.05-1.91 (m, 1H), 1.82-1.68 (m, 2H), 1.65-1.45 (m, 2H), 1.04 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 153.3, 142.7, 135.1, 128.3, 128.3, 125.9, 125.7, 124.6, 122.5, 121.5, 38.6, 36.7, 33.3, 32.2, 32.0, 19.3. FT-IR: ν (cm^{-1}) 2958, 2361, 1560, 1408, 1340 1209. HRMS [EI] calcd for $C_{19}H_{21}NS$ $[M]^+$ 295.1395, found.295.1393.

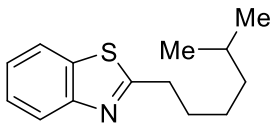


2l was prepared by **Procedure A**: 32.6 mg, 85%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR

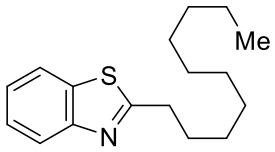
(400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 1H), 7.94-7.90 (m, 1H), 7.46-7.40 (m, 1H), 7.36-7.30 (m, 1H), 3.11 (t, *J* = 7.6 Hz, 2H), 1.94-1.90 (m, 2H), 1.52-1.41 (m, 2H), 0.97 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 153.3, 135.1, 125.9, 124.6, 122.5, 121.5, 34.1, 31.8, 22.3, 13.8. FT-IR: ν (cm^{-1}) 3064, 2957, 2871, 1456, 1311, 1243. HRMS [ESI] calcd for $C_{11}H_{14}NS$ $[M+H]^+$ 192.0841, found 192.0844.



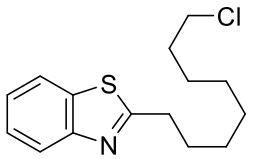
2m was prepared by **Procedure A**: 37.3 mg, 80%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^1H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 8.4 Hz, 1H), 7.49-7.41 (m, 1H), 7.37-7.30 (m, 1H), 3.17-3.01 (m, 2H), 1.99-1.77 (m, 2H), 1.50-1.10 (m, 5H), 0.90-0.81 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 153.3, 135.1, 125.8, 124.6, 122.5, 121.5, 36.1, 34.7, 34.2, 29.3, 27.4, 19.1, 11.4. FT-IR: ν (cm^{-1}) 2958, 2872, 2360, 1456, 1278, 1215. HRMS [ESI] calcd for $C_{14}H_{20}NS$ $[M+H]^+$ 234.1311, found 234.1313.



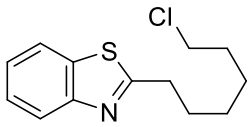
2n was prepared by **Procedure A**: 37.3 mg, 80%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^1H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.0 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.47-7.41 (m, 1H), 7.36-7.31 (m, 1H), 3.11 (t, *J* = 7.6 Hz, 2H), 1.91-1.77 (m, 2H), 1.61-1.39 (m, 2H), 1.30-1.20 (m, 3H), 0.87 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 153.3, 135.1, 125.8, 124.6, 122.5, 121.5, 38.6, 34.4, 30.0, 27.8, 27.0, 22.6. FT-IR: ν (cm^{-1}) 2952, 2866, 2360, 1558, 1384, 1279. HRMS [ESI] calcd for $C_{14}H_{20}NS$ $[M+H]^+$ 234.1311, found 234.1308.



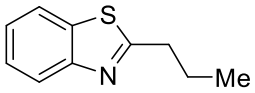
2o was prepared by **Procedure A**: 28.8 mg, 55%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^1H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 1H), 7.86-7.82 (m, 1H), 7.47-7.42 (m, 1H), 7.37-7.31 (m, 1H), 3.11 (t, *J* = 7.6 Hz, 2H), 1.92-1.83 (m, 2H), 1.50-1.20 (m, 14H), 0.92-0.84 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 153.3, 135.2, 125.9, 124.6, 122.5, 121.5, 34.4, 31.9, 29.7, 29.7, 29.6, 29.5, 29.3, 29.2, 22.7, 14.1. FT-IR: ν (cm^{-1}) 2955, 2853, 2360, 1521, 1377, 1242. HRMS [EI] calcd for $C_{16}H_{23}NS$ $[M]^+$ 261.1546, found 261.1547.



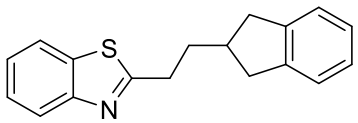
2p was prepared by **Procedure A**: 36.1 mg, 64%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^1H NMR (400 MHz, $CDCl_3$) δ 7.99-7.94 (m, 1H), 7.85-7.81 (m, 1H), 7.47-7.41 (m, 1H), 7.37-7.31 (m, 1H), 3.51 (t, *J* = 6.8 Hz, 2H), 3.11 (t, *J* = 7.6 Hz , 2H), 1.92-1.83 (m, 2H), 1.80-1.71 (m, 2H), 1.49-1.24 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.3, 153.3, 135.1, 125.9, 124.6, 122.5, 121.5, 45.1, 34.3, 32.6, 29.6, 29.1, 29.0, 28.7, 26.8. FT-IR: ν (cm^{-1}) 2926, 2854, 2360, 1595, 1456, 1278. HRMS [ESI] calcd for $C_{15}H_{21}ClNS$ $[M+H]^+$ 282.1078, found 282.1078.



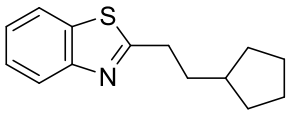
2q was prepared by **Procedure A**: 40.1 mg, 79%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.47-7.42 (m, 1H), 7.37-7.32 (m, 1H), 3.53 (t, *J* = 6.4 Hz, 2H), 3.12 (t, *J* = 7.6 Hz, 2H), 1.96-1.85 (m, 2H), 1.83-1.73 (m, 2H), 1.57-1.41 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.0, 153.3, 135.1, 125.9, 124.7, 122.5, 121.5, 45.0, 34.2, 32.4, 29.5, 28.4, 26.6. FT-IR: ν (cm^{-1}) 2932, 2857, 2360, 1595, 1456, 1242. HRMS [ESI] calcd for $C_{13}H_{17}ClNS$ $[M+H]^+$ 254.0765, found 254.0761.



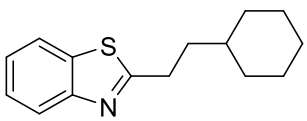
2r was prepared by **Procedure A**: 26.3 mg, 74%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.49-7.43 (m, 1H), 7.38-7.32 (m, 1H), 3.11 (t, *J* = 7.2 Hz, 2H), 1.99-1.88 (m, 2H), 1.09 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 153.3, 135.2, 125.9, 124.6, 122.5, 121.5, 36.3, 23.1, 13.7. FT-IR: ν (cm^{-1}) 2961, 2929, 1519, 1311, 1279, 1243. HRMS [ESI] calcd for $C_{10}H_{12}NS$ $[M+H]^+$ 178.0685, found 178.0684.



2s was prepared by **Procedure A**: 30.2 mg, 54%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.49-7.42 (m, 1H), 7.39-7.31 (m, 1H), 7.23-7.17 (m, 2H), 7.16-7.11 (m, 2H), 3.25-3.18 (m, 2H), 3.13 (dd, *J* = 15.2, 8.0 Hz, 2H), 2.70 (dd, *J* = 15.6, 8.4 Hz, 2H), 2.64-2.51 (m, 1H), 2.12 (dd, *J* = 15.6, 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 153.3, 143.1, 135.1, 126.2, 125.9, 124.7, 124.4, 122.5, 121.5, 39.7, 39.1, 35.5, 33.2. FT-IR: ν (cm^{-1}) 2931, 2360, 1518, 1430, 1359, 1243. HRMS [ESI] calcd for $C_{18}H_{18}NS$ $[M+H]^+$ 280.1154, found 280.1155.

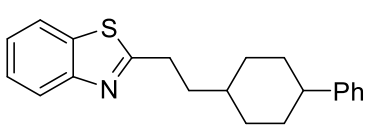


2t was prepared by **Procedure A**: 24.1 mg, 52%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.47-7.41 (m, 1H), 7.36-7.30 (m, 1H), 3.15-3.09 (m, 2H), 1.94-1.79 (m, 5H), 1.69-1.48 (m, 4H), 1.30-1.10 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 153.3, 135.1, 125.9, 124.6, 122.5, 121.5, 39.5, 35.7, 33.5, 32.2, 25.3. FT-IR: ν (cm^{-1}) 3063, 2972, 2360, 1595, 1311, 1243. HRMS [ESI] calcd for $C_{14}H_{18}NS$ $[M+H]^+$ 232.1154, found 232.1152.

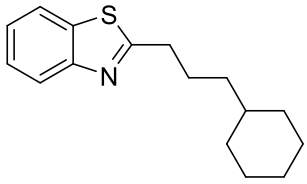


2u was prepared by **Procedure A**: 23.6 mg, 48%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H

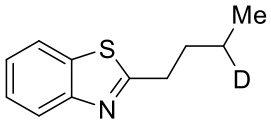
NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 1H), 7.83 (d, *J* = 7.6 Hz, 1H), 7.47-7.41 (m, 1H), 7.37-7.31 (m, 1H), 3.12 (t, *J* = 8.0 Hz, 2H), 1.85-1.60 (m, 7H), 1.45-1.10 (m, 4H), 1.03-0.75 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.8, 153.3, 135.1, 125.8, 124.6, 122.5, 121.5, 37.3, 37.2, 33.1, 31.9, 26.5, 26.2. FT-IR: ν (cm^{-1}) 2920, 2849, 2340, 1558, 1312, 1242. HRMS [ESI] calcd for $C_{15}H_{20}S$ $[M+H]^+$ 246.1311, found 246.1316.



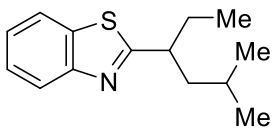
2v was prepared by **Procedure A**: 40.5 mg, 63%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^1H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.49-7.43 (m, 1H), 7.39-7.33 (m, 1H), 7.32-7.25 (m, 2H), 7.24-7.14 (m, 3H), 3.18 (t, *J* = 8.0 Hz, 2H), 2.56-2.44 (m, 1H), 2.04-1.80 (m, 6H), 1.55-1.41 (m, 3H), 1.30-1.10 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 153.3, 147.5, 135.1, 128.3, 126.8, 125.9, 125.9, 124.6, 122.5, 121.5, 44.4, 37.1, 36.9, 34.1, 33.3, 32.0. FT-IR: ν (cm^{-1}) 2928, 2933, 1647, 1543, 1330, 1240. HRMS [EI] calcd for $C_{21}H_{23}NS$ $[M]^+$ 321.1551, found 321.1557.



2w was prepared by **Procedure A**: 30.1 mg, 58%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^1H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 1H), 7.83 (d, *J* = 7.6 Hz, 1H), 7.47-7.41 (m, 1H), 7.37-7.31 (m, 1H), 3.09 (t, *J* = 8.0 Hz, 2H), 1.94-1.83 (m, 2H), 1.77-1.60 (m, 5H), 1.40-1.05 (m, 5H), 0.96-0.82 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 153.3, 135.2, 125.8, 124.6, 122.5, 121.5, 37.4, 36.9, 34.7, 33.3, 27.2, 26.6, 26.3. FT-IR: ν (cm^{-1}) 2929, 2360,1560, 1421, 1338, 1230. HRMS [EI] calcd for $C_{16}H_{21}NS$ $[M]^+$ 259.1395, found 259.1396.

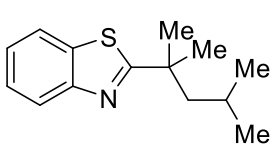


2x was prepared by **Procedure A**: 31.6 mg, 82% yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^1H NMR (400 MHz, $CDCl_3$) δ 7.99-7.94 (m, 1H), 7.85-7.81 (m, 1H), 7.47-7.41 (m, 1H), 7.36-7.30 (m, 1H), 3.11 (t, *J* = 7.6 Hz, 2H), 1.91-1.82 (m, 2H), 1.52-1.41 (m, 1H), 1.00-0.94 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 153.3, 135.2, 125.9, 124.6, 122.5, 121.5, 34.0, 31.7, 21.9 (t, $J_{C\text{-}D}$ = 19.3 Hz), 13.7. FT-IR: ν (cm^{-1}) 3063, 2954, 2360, 1519, 1378, 1276. HRMS [ESI] calcd for $C_{11}H_{13}DNS$ $[M+H]^+$ 193.0904, found 193.0900.

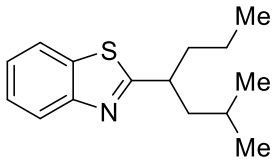


4a was prepared by **Procedure B**: 43.5 mg, 93%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^1H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.4 Hz, 1H), 7.87-7.83 (m, 1H), 7.47-7.41 (m, 1H), 7.37-7.31 (m, 1H), 3.24-3.12 (m, 1H), 1.87-1.71 (m, 3H), 1.63-1.49 (m, 2H), 0.96-0.85 (m, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.2, 153.0, 134.7, 125.7, 124.5, 122.6, 121.5, 45.4, 44.9, 29.9, 25.7, 23.2, 22.0, 11.9.

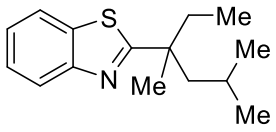
FT-IR: ν (cm^{-1}) 2957, 2870, 1437, 1366, 1254, 1239. HRMS [ESI] calcd for $C_{14}H_{20}NS$ $[M+H]^+$ 234.1311, found 234.1309.



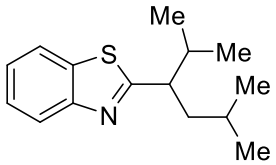
4b was prepared by **Procedure B**: 26.6 mg, 57%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/40). ^1H NMR (400 MHz, $CDCl_3$) δ 8.02-7.97 (m, 1H), 7.87-7.83 (m, 1H), 7.47-7.41 (m, 1H), 7.36-7.30 (m, 1H), 1.80 (d, *J* = 5.6 Hz, 2H), 1.75-1.65 (m, 1H), 1.51 (s, 6H), 0.80 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.7, 153.2, 135.0, 125.7, 124.5, 122.7, 121.4, 52.9, 41.7, 29.3, 25.1, 24.5. FT-IR: ν (cm^{-1}) 2957, 2360, 1437, 1311, 1285, 1244. HRMS [ESI] calcd for $C_{14}H_{20}NS$ $[M+H]^+$ 234.1311, found 234. 1314.



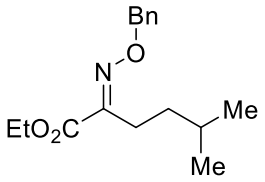
4c was prepared by **Procedure B**: 40.6 mg, 82%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^1H NMR (400 MHz, $CDCl_3$) δ 8.01-7.96 (m, 1H), 7.87-7.82 (m, 1H), 7.47-7.41 (m, 1H), 7.37-7.30 (m, 1H), 3.33-3.23 (m, 1H), 1.81-1.68 (m, 3H), 1.62-1.47 (m, 2H), 1.40-1.20 (m, 2H), 0.94 (d, *J* = 6.0 Hz, 3H), 0.92-0.85 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.4, 153.0, 134.7, 125.7, 124.5, 122.6, 121.5, 45.8, 43.1, 39.2, 25.7, 23.2, 22.0, 20.5, 14.0. FT-IR: ν (cm^{-1}) 2955, 2869, 2360, 1437, 1310, 1247. HRMS [ESI] calcd for $C_{15}H_{22}NS$ $[M+H]^+$ 248.1467, found 248.1470.



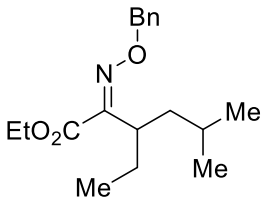
4d was prepared by **Procedure B**: 22.3 mg, 45%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^1H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.47-7.40 (m, 1H), 7.36-7.30 (m, 1H), 2.00-1.83 (m, 2H), 1.82-1.65 (m, 3H), 1.50 (s, 3H), 0.87 (d, *J* = 6.4 Hz, 3H), 0.81 (t, *J* = 7.6 Hz, 3H), 0.69 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.0, 153.1, 135.0, 125.6, 124.4, 122.7, 121.4, 51.7, 45.2, 36.5, 25.0, 24.8, 24.2, 23.6, 8.6. FT-IR: ν (cm^{-1}) 2958, 2360, 1435, 1320, 1245, 1240. HRMS [EI] calcd for $C_{15}H_{21}NS$ $[M]^+$ 247.1395, found 247.1391.



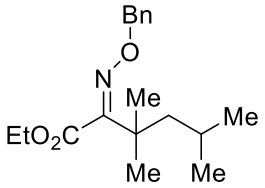
4e was prepared by **Procedure B**: 32.7 mg, 66%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/40). ^1H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.48-7.41 (m, 1H), 7.37-7.31 (m, 1H), 3.10-2.96 (m, 1H), 2.06-1.94 (m, 1H), 1.85-1.74 (m, 1H), 1.70-1.56 (m, 1H), 1.49-1.35 (m, 1H), 1.04 (d, *J* = 6.8 Hz, 3H), 0.91 (d, *J* = 6.4 Hz, 3H), 0.90 (d, *J* = 6.8 Hz, 3H), 0.86 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.1, 153.0, 134.7, 125.7, 124.5, 122.6, 121.5, 49.9, 42.5, 34.0, 25.9, 23.8, 21.4, 20.6, 20.6. FT-IR: ν (cm^{-1}) 2960, 2360, 1438, 1410, 1340, 1238. HRMS [ESI] calcd for $C_{15}H_{22}NS$ $[M+H]^+$ 248.1467, found 248.1469.



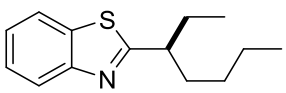
5a was prepared by **Procedure C**: 42.2 mg, 76%, Z/E ratio 1:5, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). **Major isomer**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.28 (m, 5H), 5.30 (s, 2H), 4.33 (q, *J* = 7.2 Hz, 2H), 2.70-2.40 (m, 2H), 1.60-1.48 (m, 1H), 1.45-1.25 (m, 5H), 0.90 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7, 153.8, 136.8, 128.4, 128.1, 128.1, 61.7, 34.8, 31.4, 30.2, 28.2, 23.9, 22.3, 14.2. FT-IR: ν (cm^{-1}) 2959, 2924, 1718, 1496, 1314, 1258. HRMS [ESI] calcd for $C_{16}H_{23}NO_3Na$ $[M+Na]^+$ 300.1570, found.300.1567.



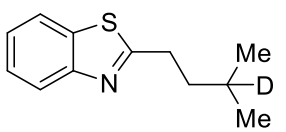
5b was prepared by **Procedure C**: 52.5 mg, 86%, Z/E ratio 1:5, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). **Major isomer**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.29 (m, 5H), 5.24 (s, 2H), 4.29 (q, *J* = 7.2 Hz, 2H), 3.40-3.26 (m, 1H), 1.79-1.65 (m, 2H), 1.60-1.41 (m, 3H), 1.34 (t, *J* = 7.2 Hz, 3H), 0.86-0.80 (m, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.6, 155.8, 136.8, 128.4, 128.3, 128.1, 76.7, 61.4, 41.2, 37.4, 26.5, 25.6, 23.4, 22.1, 14.2, 12.3. FT-IR: ν (cm^{-1}) 2956, 2923, 1717, 1558, 1313, 1241. HRMS [EI] calcd for $C_{18}H_{27}NO_3$ $[M]^+$ 305.1985, found.305.1986.



5c was prepared by **Procedure C**: 43.9 mg, 72%, Z/E ratio 1:5, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). **Major isomer**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.27 (m, 5H), 5.10 (s, 2H), 4.30 (q, *J* = 7.2 Hz, 2H), 1.70-1.59 (m, 1H), 1.44-1.37 (m, 2H), 1.36-1.27 (m, 3H), 1.14 (s, 6H), 0.85 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 159.5, 137.8, 128.2, 127.8, 127.5, 76.0, 61.0, 48.8, 38.9, 26.4, 24.7, 24.6, 14.2. FT-IR: ν (cm^{-1}) 2956, 2924, 1698, 1455, 1384, 1277. HRMS [ESI] calcd for $C_{18}H_{27}NO_3Na$ $[M+Na]^+$ 328.1883, found.328.1881.



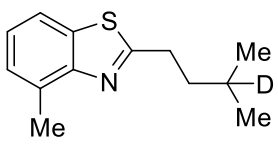
6 was prepared by **Procedure B**: 41.1 mg, 88%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.48-7.41 (m, 1H), 7.37-7.31 (m, 1H), 3.16-3.02 (m, 1H), 1.90-1.70 (m, 4H), 1.40-1.10 (m, 6H), 0.96-0.82 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.1, 152.0, 133.7, 124.7, 123.5, 121.6, 120.5, 46.1, 34.8, 28.5, 28.4, 21.6, 12.9, 10.9. FT-IR: ν (cm^{-1}) 2957, 2928, 1714, 1595, 1378, 1278. HRMS [EI] calcd for $C_{12}H_{19}NS$ $[M]^+$ 233.1238, found 233.1240.



(85% D)

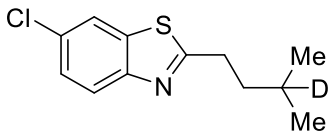
9a was prepared by **Procedure D:** 23.1 mg, 56%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 1H), 7.83 (dd, *J* = 8.0, 0.4 Hz, 1H), 7.48-7.41 (m, 1H), 7.37-7.31 (m, 1H), 3.16-3.09 (m, 2H), 1.77 (t, *J* = 8.0 Hz, 2.15H, **0.85 D**), 0.97 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 153.3, 135.1, 125.9, 124.6, 122.5, 121.5, 38.5, 32.4,

27.3 (t, $J_{C\text{-}D}$ = 19.0 Hz), 22.2. FT-IR: ν (cm^{-1}) 2956, 2930, 1556, 1440, 1285, 1240. HRMS [ESI] calcd for $C_{12}H_{15}DNS$ $[M+H]^+$ 207.1061, found 207.1065.



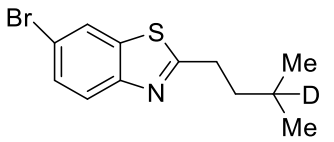
(80% D)

9b was prepared by **Procedure D:** 28.6 mg, 65%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74-7.66 (m, 1H), 7.30-7.22 (m, 2H), 3.20-3.12 (m, 2H), 2.76 (s, 3H), 1.79 (t, *J* = 8.0 Hz, 2.20 H, **0.80 D**), 1.00 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 152.6, 135.0, 132.4, 126.4, 124.4, 118.9, 38.8, 32.5, 27.4 (t, $J_{C\text{-}D}$ = 19.3 Hz), 22.3, 18.6. FT-IR: ν (cm^{-1}) 2956, 2934, 1536, 1330, 1242. HRMS [ESI] calcd for $C_{13}H_{17}DNS$ $[M+H]^+$ 221.1217, found 221.1210.



(85% D)

9c was prepared by **Procedure D**: 20.7mg, 43%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate /petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.8 Hz, 1H), 7.80 (d, *J* = 2.0 Hz, 1H), 7.39 (dd, *J* = 8.8, 2.0 Hz, 1H), 3.13- 3.06 (m, 2H), 1.76 (t, *J* = 8.0 Hz, 2.15 H, **0.85 D**), 0.97 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2, 151.8, 136.3, 130.5, 126.6, 123.2, 121.1, 38.4, 32.3, 27.3 (t, $J_{C\text{-}D}$ = 19.2 Hz), 22.2. FT-IR: ν 2956, 2925, 1592, 1385, 1266, 1231. (cm^{-1}) HRMS [ESI] calcd for $C_{12}H_{14}ClDNS$ $[M+H]^+$ 241.0671, found 241.0674.

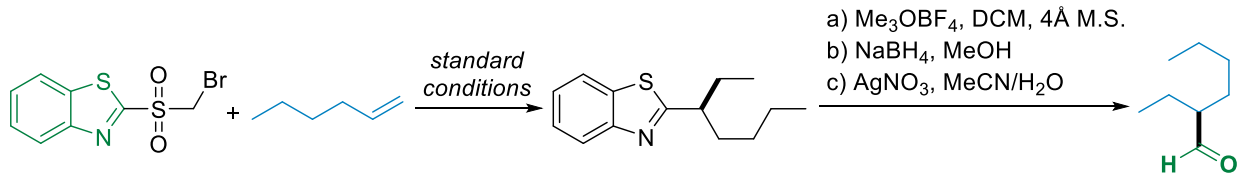


(90% D)

9d was prepared by **Procedure D**: 30.8 mg, 54%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate /petroleum ether = 1/150). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 2.0 Hz, 1H), 7.80 (d, *J* = 8.8 Hz, 1H), 7.54 (dd, *J* = 8.8, 2.0 Hz, 1H), 3.13-3.05 (m, 2H), 1.78 (t, *J* = 7.6 Hz, 2.10 H, **0.90 D**), 0.99 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 152.1, 136.8, 129.3, 124.0, 123.6, 118.1, 38.4, 32.3, 27.3 (t, $J_{C\text{-}D}$ = 19.2 Hz), 22.2. FT-IR: ν (cm^{-1}) 2956, 2925, 2140, 1518, 1385, 1267. HRMS [EI] calcd for $C_{12}H_{13}BrDNS$ $[M]^+$ 284.0088, found 284.0089.

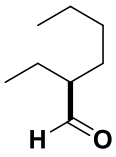
5.Transformation of compound 6

5.1 Hydrolysis of compound 6



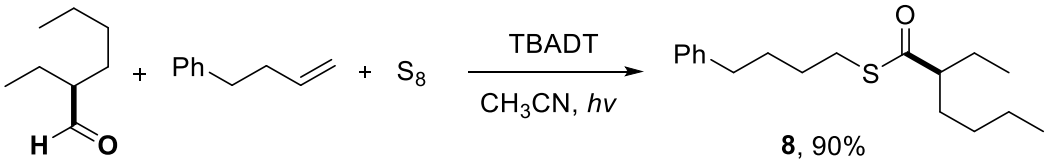
1r **6**, 88%,12 h **7**, 66%

A mixture of **6** (0.5 mmol), activated 4 Å molecular sieves powders (750 mg), and anhydrous DCM (5 mL) were stirred at r.t. for 10 min, and then Me_3OBF_4 (1.25 mmol) was added. After stirred at r.t. for 2 h, another batch of Me_3OBF_4 (1.25 mmol) was added to the suspension, which was continued to react until starting material was fully consumed as determined by TLC. The reaction was concentrated without filtering off the molecular sieves to give the crude N-methylbenzothiazolium salt. The residue was re-dissolve in MeOH (1.0 mL), which was then cooled to 0 ℃ and added $NaBH_4$ (0.75 mmol). Another batch of $NaBH_4$ (0.5 mmol) was added to the reaction until the starting material had been consumed as determined by TLC. The mixture was diluted with acetone, filtered through a pad of Celite, and concentrated to give the crude benzothiazolines. To a vigorously stirred solution of the crude benzothiazolines in CH_2Cl_2 (1.5 mL) and CH_3CN (7.5 mL) were added H_2O (1.0 mL) followed by $AgNO_3$ (1.5 mmol). The mixture was stirred at r.t. (monitored by TLC), and then diluted with 1 M phosphate buffer at pH = 7 (0.1 mL). After stirred for 15 min, the reaction mixture was diluted with 1 M phosphate buffer at pH = 7 (5.0 mL) and partially concentrated to remove CH_3CN. The suspension was extracted with EtOAc, and the combined organic layers were dried over Na_2SO_4, filtered through a pad of Celite, and concentrated. The residue was purified by flash column chromatography on silica gel to afford product **7**.

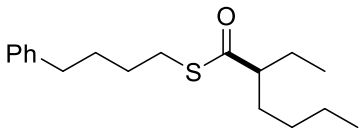


7: 46.9 mg, 66%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate /petroleum ether = 1/80). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.57 (d, *J* = 3.6 Hz, 1H), 2.22-2.12 (m, 1H), 1.74-1.40 (m, 4H), 1.36-1.20 (m, 4H), 0.95-0.87 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.7, 53.4, 29.2, 28.2, 22.7, 21.8, 13.9, 11.4. FT-IR: ν (cm^{-1}) 2931, 2861, 1725, 1460, 1381, 1228. HRMS [EI] calcd for $C_9H_{18}O$ $[M]^+$ 142.1358, found 142.1358.

5.2 Synthesis of thioester 8



To an 4 mL vial equipped with a magnetic stir bar was added the corresponding sulfur powder (0.1 mmol, 1.0 equiv.), TBADT (2 mol%) and acetonitrile (1 mL). The resulting mixture was cooled to 0 °C using an ice-water bath, and bubbled with an argon balloon for 10 min. Alkene (0.1 mmol, 1.0 equiv.) and aldehyde 7 (0.15 mmol, 1.5 equiv.) was then added. Then the reactor was placed at a 40 W 390 nm LED, stirred and irradiated at room temperature for 24 h under argon atmosphere. The reaction mixture was quenched by removing from the light source. The solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel to give the corresponding product **8**.



8: 26.3 mg, 90%, yellow oil. Purification by flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/100). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.22 (m, 2H), 7.20-7.12 (m, 3H), 2.89 (t, *J* = 7.2 Hz, 2H), 2.62 (t, *J* = 7.2 Hz, 2H), 2.50-2.40 (m, 1H), 1.75-1.57 (m, 6H), 1.53-1.39 (m, 2H), 1.35-1.20 (m, 4H), 0.93-0.84 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.6, 142.2, 128.4, 128.3, 125.8, 56.2, 35.4, 32.4, 30.5, 29.5, 29.3, 28.4, 26.1, 22.7, 14.0, 11.8. FT-IR: ν (cm^{-1}) 2959, 1683, 1496, 1401, 1380, 1261. HRMS [EI] calcd for $C_{18}H_{28}OS$ $[M]^+$ 292.1861, found 292.1863.

6. Mechanistic Studies

6.1 Cyclic voltammograms of TMPDA

All voltammograms were taken at room temperature using a saturated calomel (SCE) reference electrode, a mesh platinum (Pt) counter electrode, and a glassy carbon working electrode. The conditions of the experiments were the following: an acetonitrile solution of 100 mM tetrabutylammonium hexafluorophosphate (NBu4PF6) and 3 mM TMPDA, a scan rate of 0.1 V/s, and a negative initial scan direction. The reported potentials were averages over segments, and were taken at half-height of the cathodic peaks (Ep/2) of TMPDA, since all reductions were nonreversible. To convert the potentials from SCE to Fc/Fc+ reference, 380 mV were subtracted from the measured values. The positive peaks on the return sweep of most substrates were thought to signify an ECE-type mechanism.

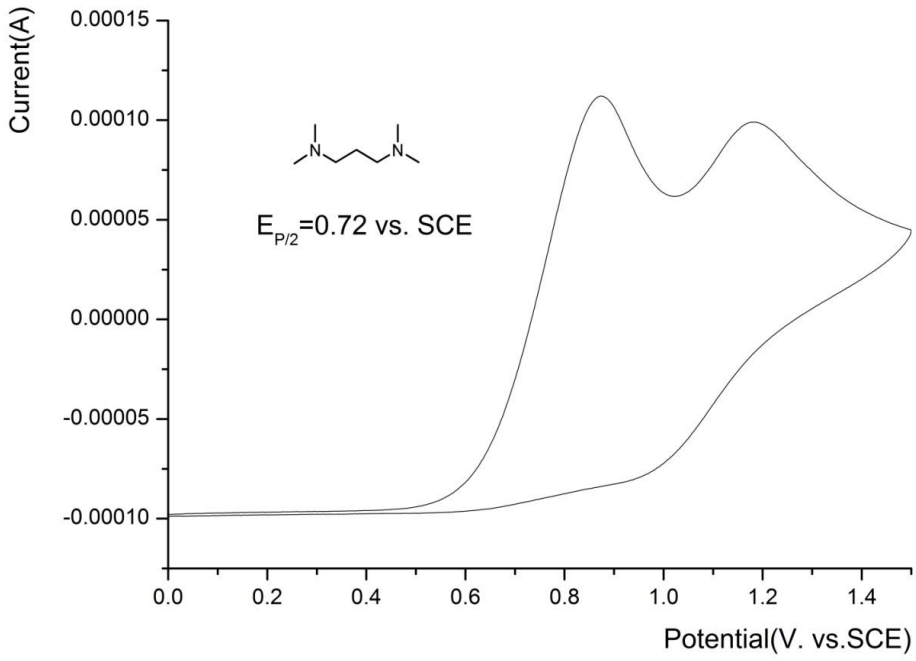
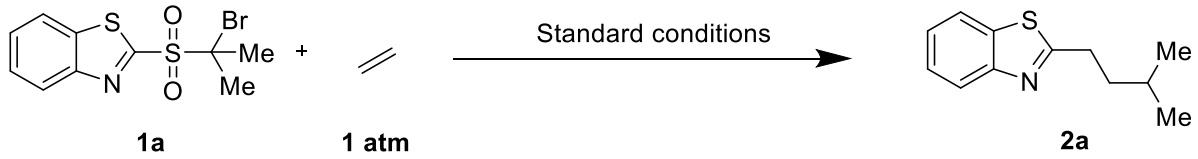
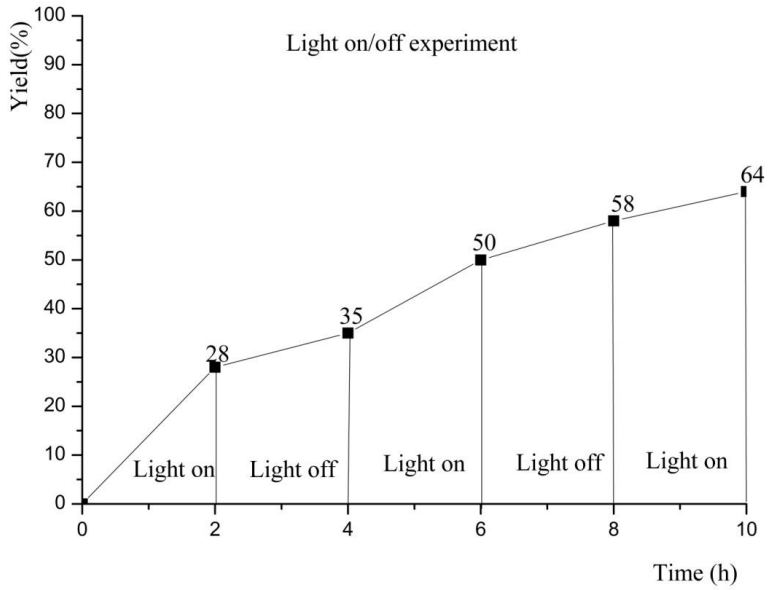


Fig. S6 Cyclic voltammogram of TMPDA in CH_3CN

6.2 Light on/off experiments



1a **1 atm** **2a**



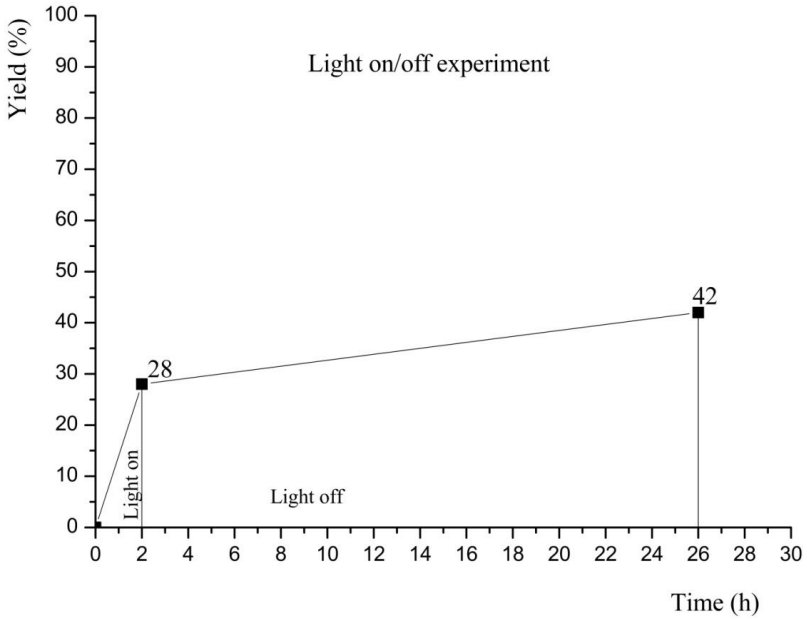


Figure S7. Time profile of the light on/off experiments

6.3 Quantum yield measurements

Determination of the light intensity at 390 nm

According to the procedure of Yoon [2] the photon flux of the LED (λmax = 390 nm) was determined, by standard ferrioxalate actinometry. A 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate hydrate (0.737 g) in H_2SO_4 (10 mL of a 0.05 M solution). A buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (5.0 mg) and sodium acetate (1.13 g) in H_2SO_4 (5.0 mL of a 0.5 M solution). Both solutions were stored in the dark. To determine the photon flux of the LED, the ferrioxalate solution (3.0 mL) was placed in a cuvette and irradiated for 90 seconds at λmax = 390 nm. After irradiation, the phenanthroline solution (0.525 mL) was added to the cuvette and the mixture was allowed to stir in the dark for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. The same procedure was repeated two more times. A nonirradiated sample was also prepared and the absorbance at 510 nm was measured. The average of the absorption of the irradiated and non-irradiated samples was determined and used to calculate the generated amount of Fe(II) according to the Lambert-Beer law (equation 1),

$$\textbf{mol } Fe^{2+} = (V \times \Delta A)/ (l \times \varepsilon) \qquad (1)$$

$$\textbf{mol } Fe^{2+} = [3.525 \times 10^{-3}\ L \times (1.815\text{-}0.327)]/ (1\ cm \times 11100\ L\ mol^{-1}cm^{-1}) = 4.725 \times 10^{-7}\ mol$$

V is the total volume (0.003525 L) of the solution after addition of phenanthroline, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, l is the path length (1.00 cm), and ε is the molar absorptivity of the ferrioxalate actinometer at 510 nm (11,100 Lmol-1cm-1).[3] The photon flux can be calculated according (equation 2)

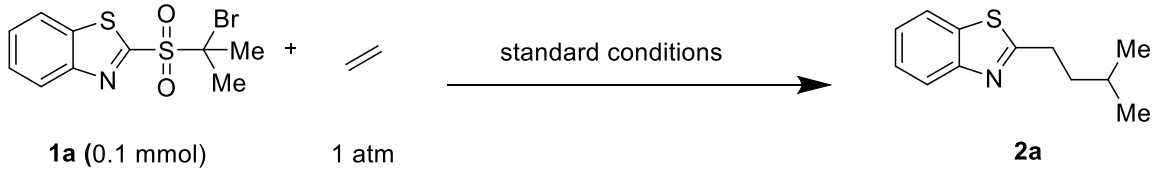
$$\textbf{photo flux} = \text{mol } Fe^{2+}/ (\Phi \times t \times f) \qquad (2)$$

$$\textbf{photo flux} = 4.725 \times 10^{-7} / (1.13 \times 90 \times 1) = 4.651 \times 10^{-9}\ \text{einstein s}^{-1}$$

Where Φ is the quantum yield for the ferrioxalate actinometer (1.13 at λ = 390 nm) [4] is the irradiation time (90 s), and f is the fraction of light absorbed at 390 nm by the ferrioxalate actinometer. This value is calculated using equation 3 where $A_{390\ nm}$ is the absorbance of the ferrioxalate solution at 390 nm. An absorption spectrum gave an $A_{390\ nm}$ value of >3, indicating that the fraction of absorbed light (f) is 1 (f > 0.999)..

$$f = 1 - 10^{-A_{390\ nm}} \qquad (3)$$

Determination of the reaction quantum yield



1a (0.1 mmol) 1 atm **2a**

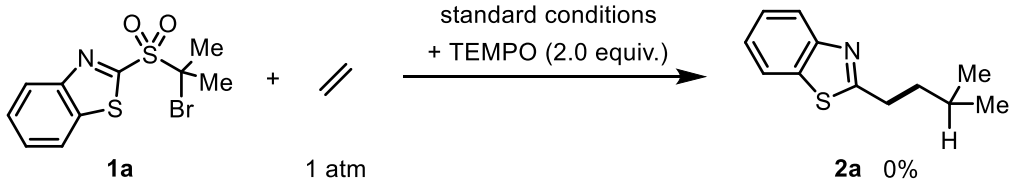
The reaction mixture was stirred and irradiated by blue LED (λmax = 390 nm) for 1800 s. The yield of product was determined by ^{1}H NMR analysis using 1,3,5-Trimethoxybenzene as an internal standard. The yield of **3a** was determined to be 23.0% (0.023×10^{-3} mol of **3a**). The reaction quantum yield (Φ) was determined using equation 4 where the photon flux is 4.651×10^{-9} einsteins s^{-1} (determined by actinometry as described above), t is the reaction time (1800 s) and f is the fraction of incident light absorbed by the catalyst, determined using (equation 3).

$$\textbf{Quantum Yield} = \text{moles of product formed}/ (\text{flux} \times f \times t) \quad (4)$$

$$= 0.023 \times 10^{-3} /(4.651 \times 10^{-9} \times 1 \times 1800) = 2.7$$

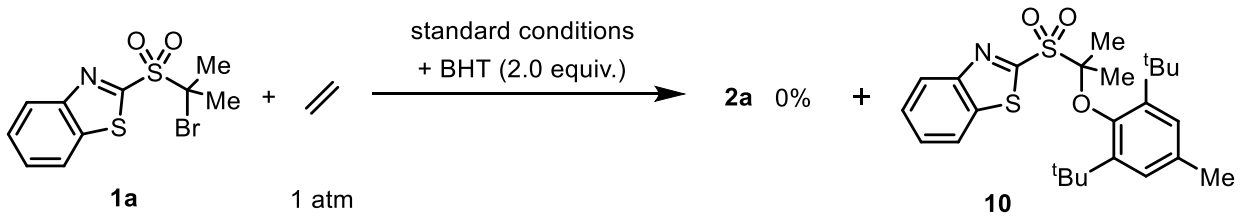
6.4 Radical trapping experiment

6.4.1 TEMPO was used as a radical scavenger



1a (0.2mmol, 1.0 equiv.) and TEMPO (0.4 mmol, 2.0 equiv.) were loaded were loaded in a Schlenk tube with a balloon, which was subjected to evacuation/flushing with ethylene for three times and aerated with ethylene gas in the balloon. Then, TMPDA (0.5mmol, 2.5 equiv.), *tert*-dodecylthiol (0.4 mmol, 2.0 equiv.), and CF_3CH_2OH (20 mL) were added to the mixture via syringe, which was irradiated with 40 W 390 nm LED light and stirred at r.t. for 6 h.. The mixture was extracted with ethyl acetate. The combined organic extracts were washed by brine, dried over Na_2SO_4, filtered, concentrated.

6.4.2 BHT was used as a radical scavenger



1a (0.2mmol, 1.0equiv.) and BHT (0.4 mmol, 2.0equiv.) were loaded were loaded in a Schlenk tube with a balloon, which was subjected to evacuation/flushing with ethylene for three times and aerated with ethylene gas in the balloon. Then, TMPDA (0.5mmol, 2.5equiv.), *tert*-dodecylthiol (0.4 mmol, 2.0equiv.), and CF_3CH_2OH (20 mL) were added to the mixture via syringe, which was irradiated with 40 W 390 nm LED light

and stirred at r.t. for 6h.. The mixture was extracted with ethyl acetate. The combined organic extracts were washed by brine, dried over Na_2SO_4, filtered, concentrated.

The crude product was subjected to HR-MS measurements. MS calcd for $C_{24}H_{31}NO_3S_2$ (**10**) $[M]^+$ 459.1902, found 459.1921.

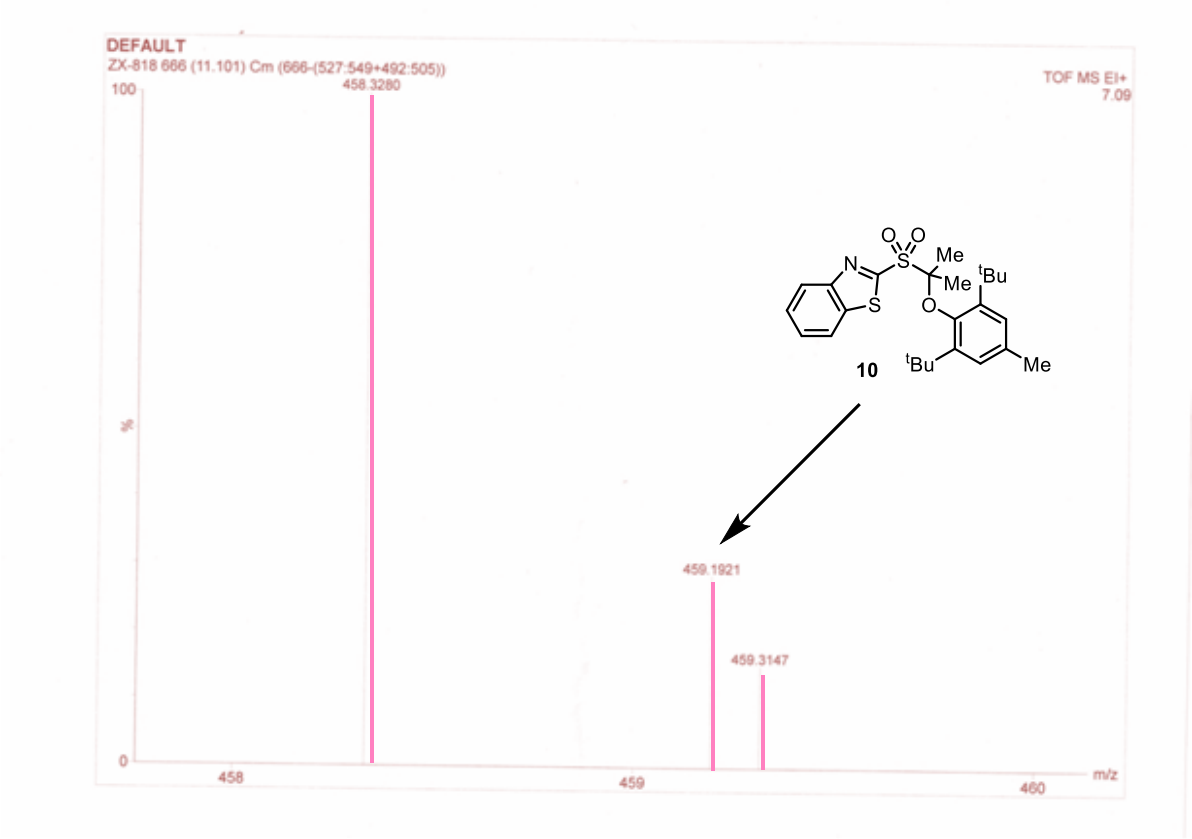
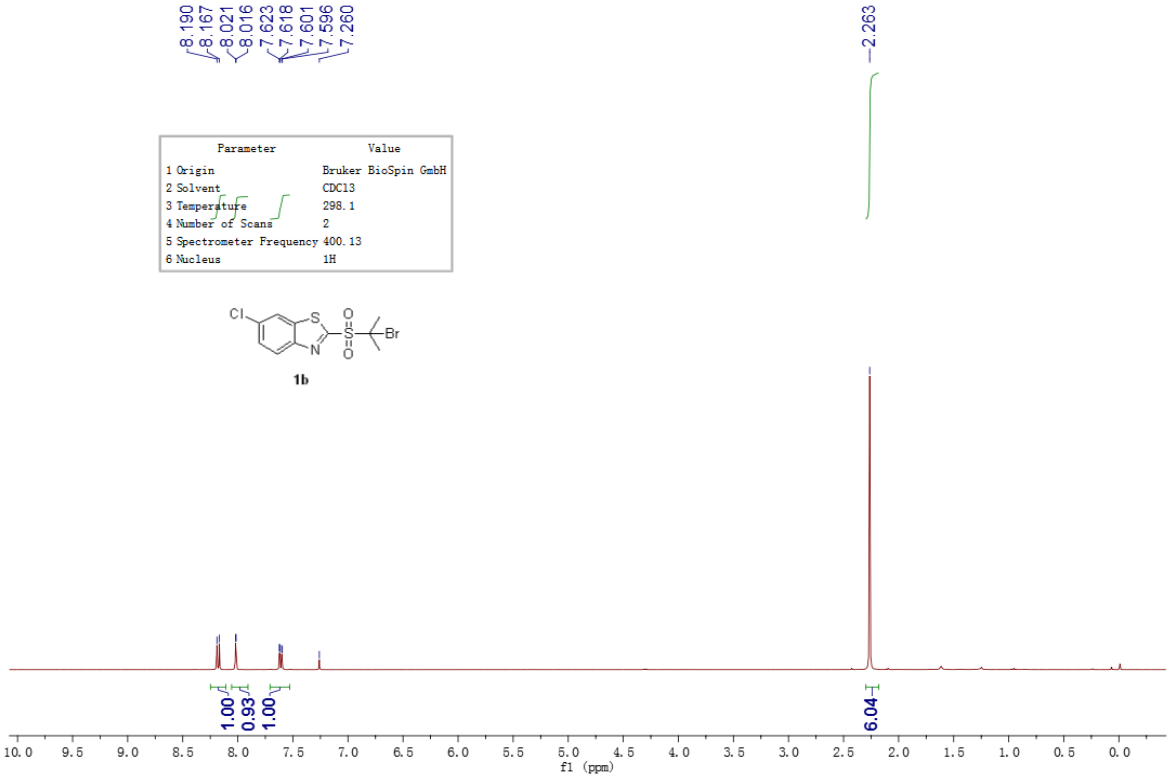
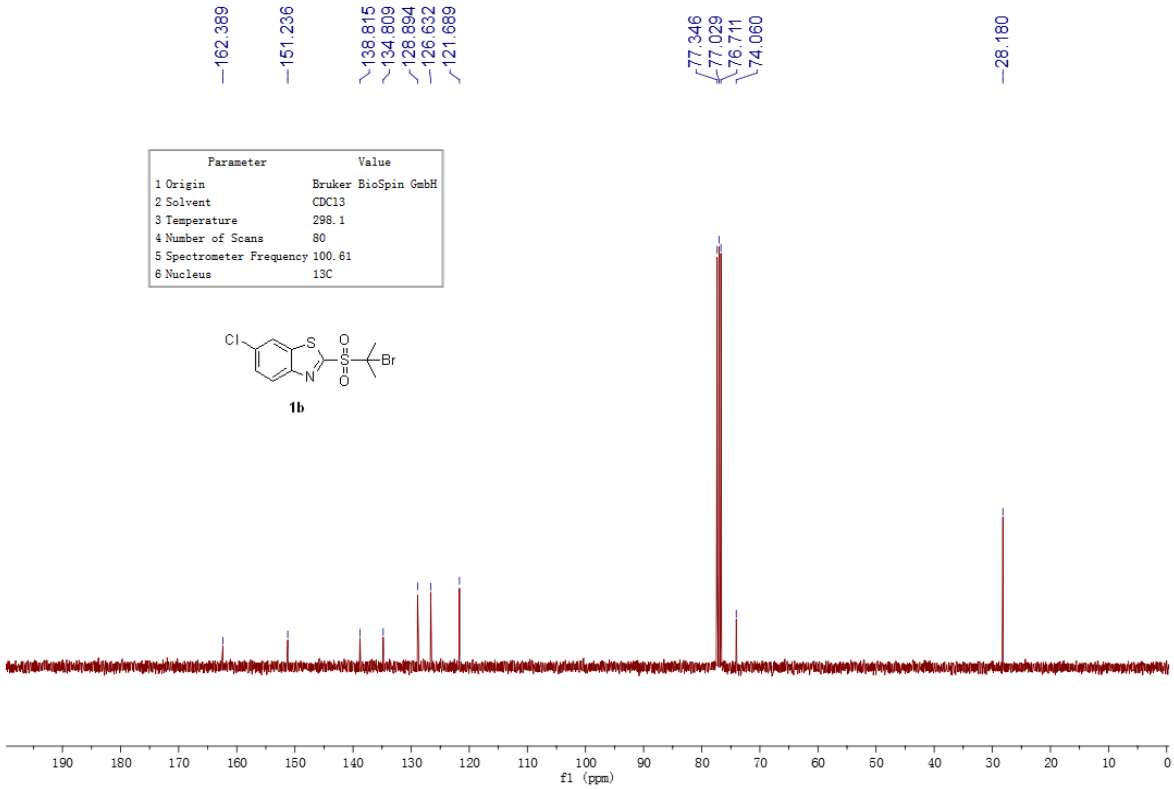
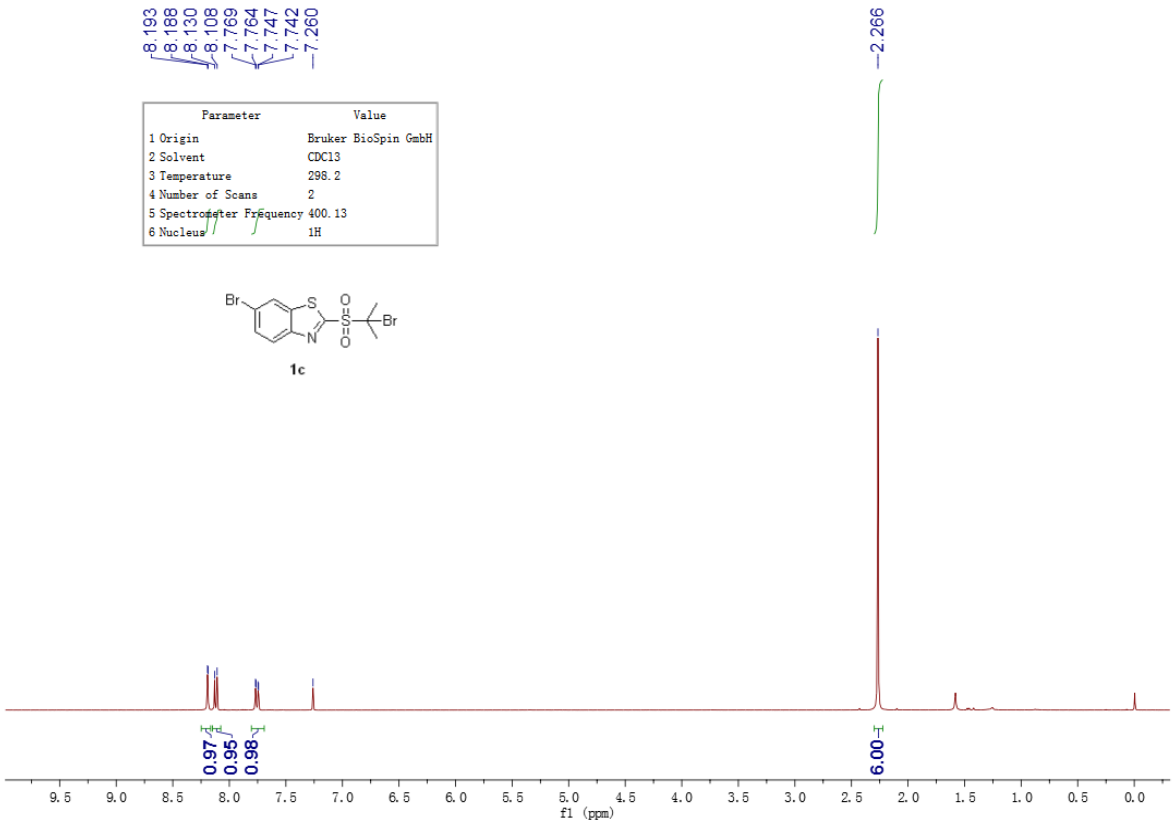


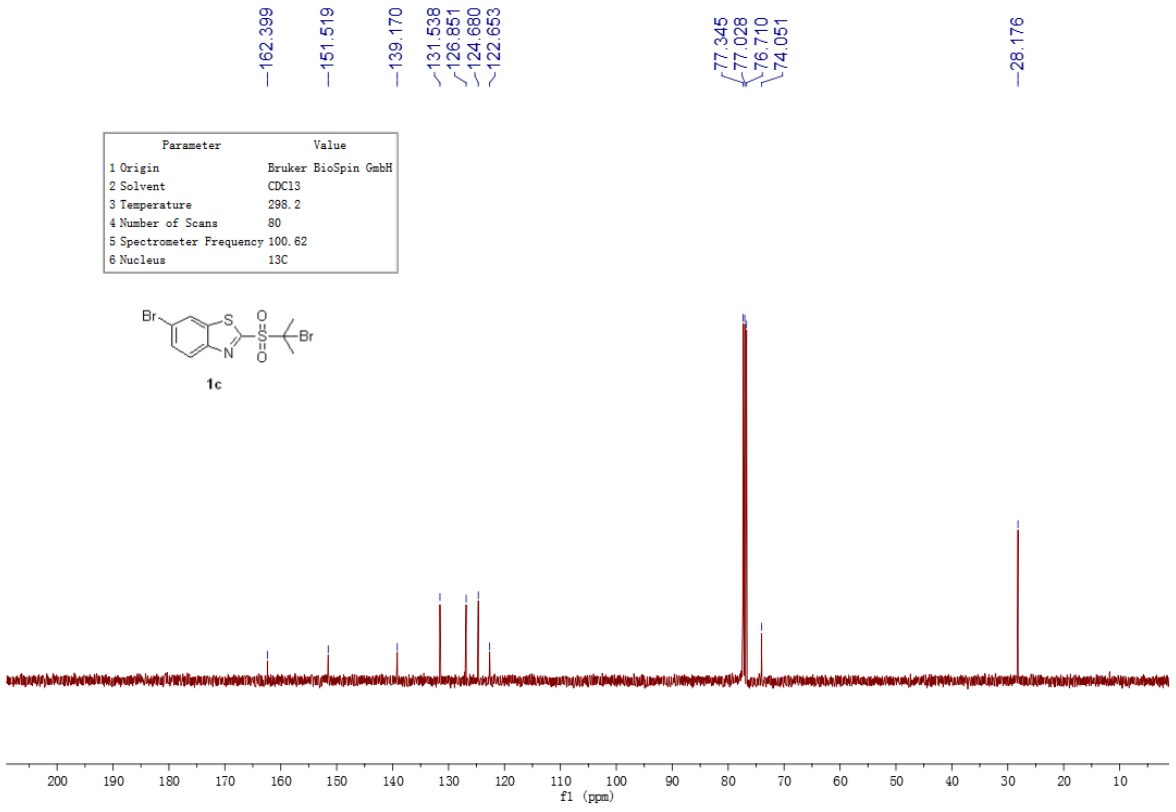
Figure S8. HRMS analysis of trapping adduct **10**

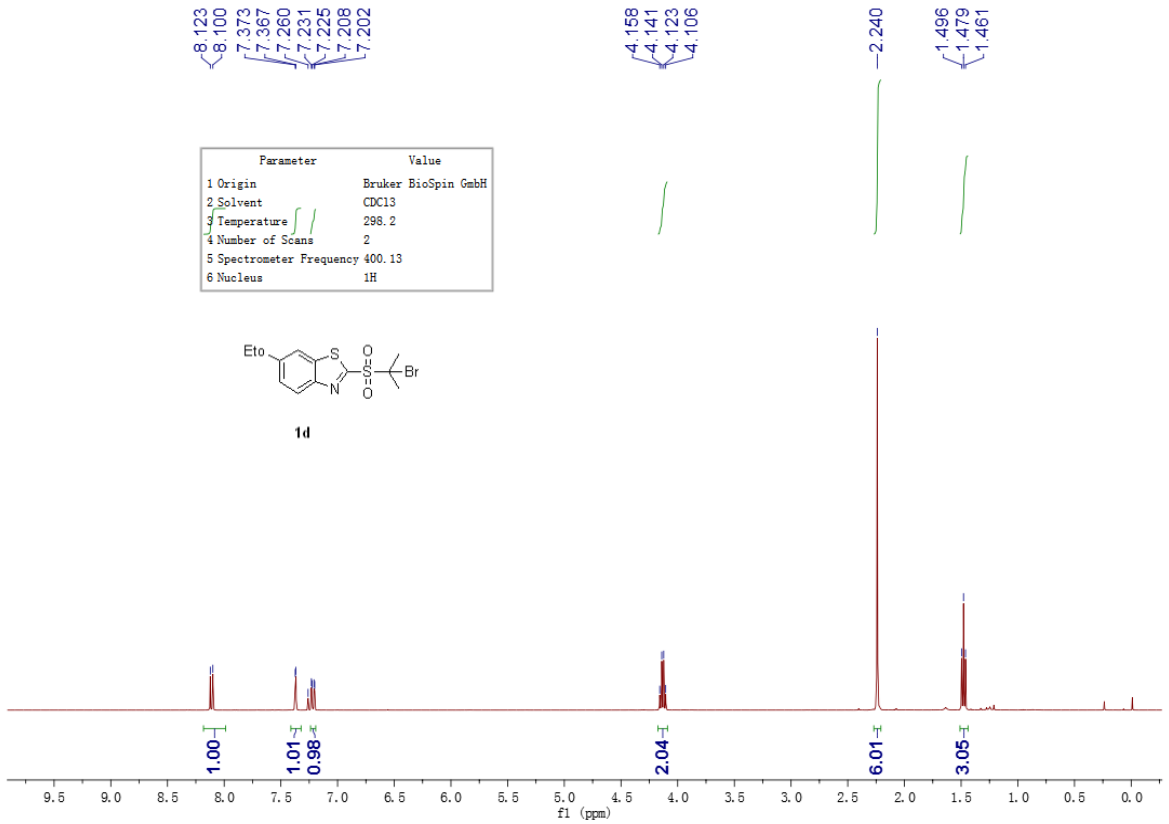
7. NMR spectra

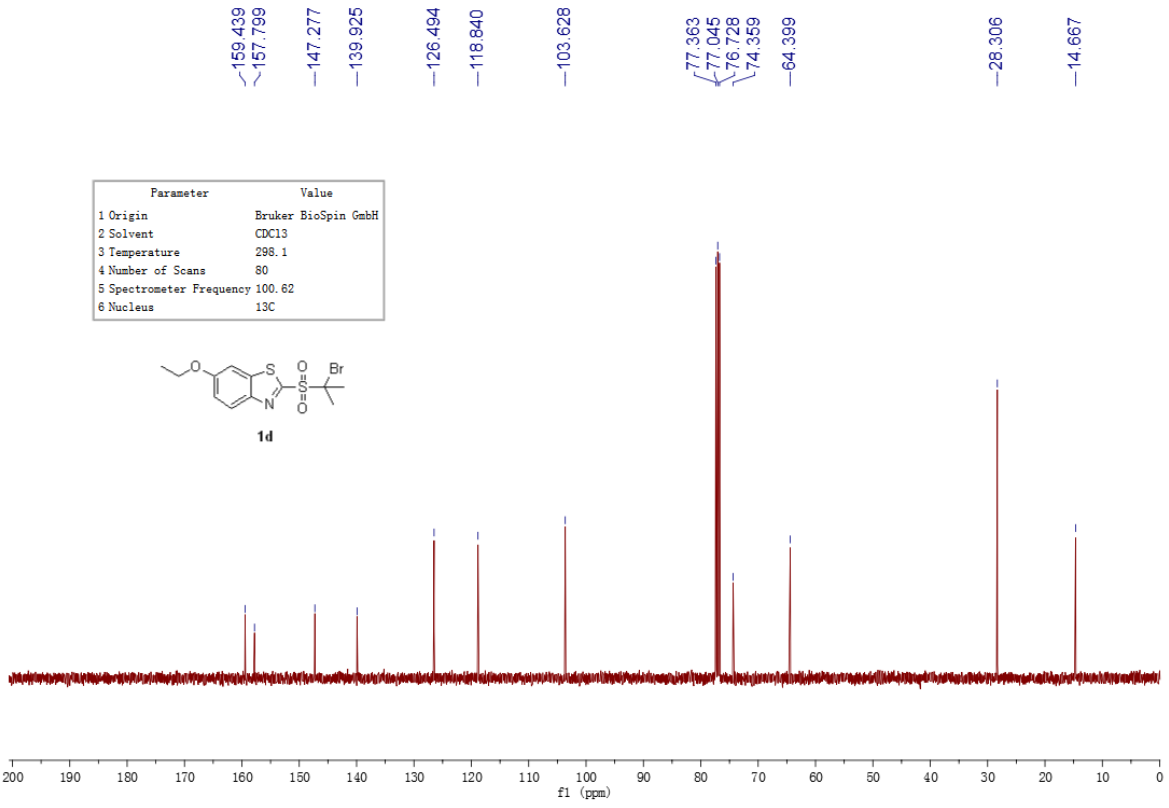


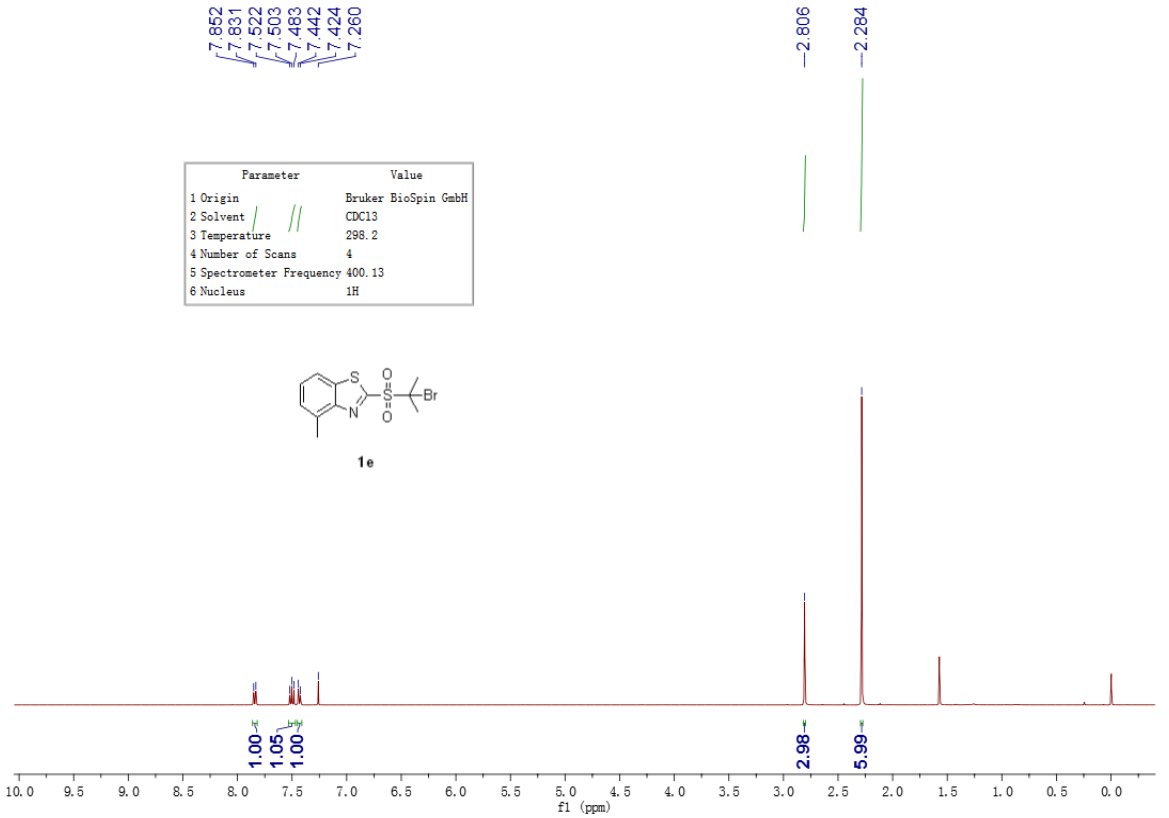


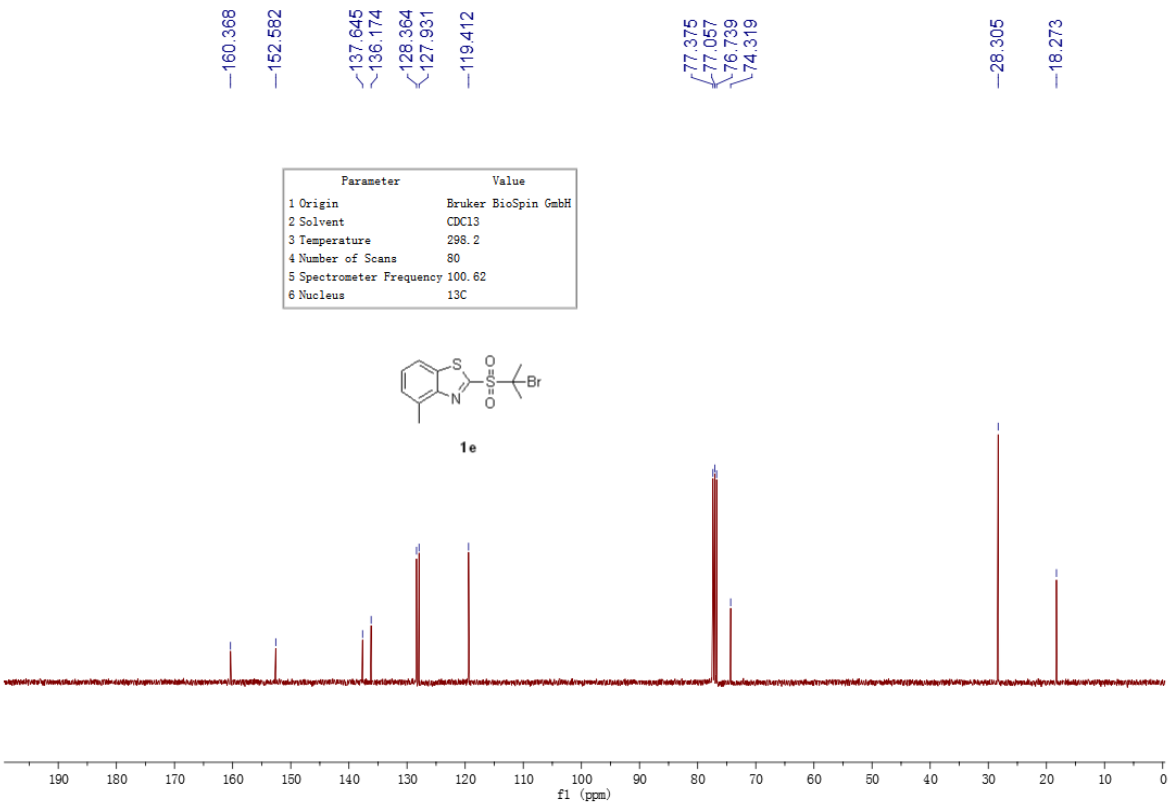

8.193
8.188
8.130
8.108
7.769
7.764
7.747
7.742
7.260
2.266
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Br
S
N
O
O
Br
1c
0.97
0.95
0.98
6.00
f1 (ppm)

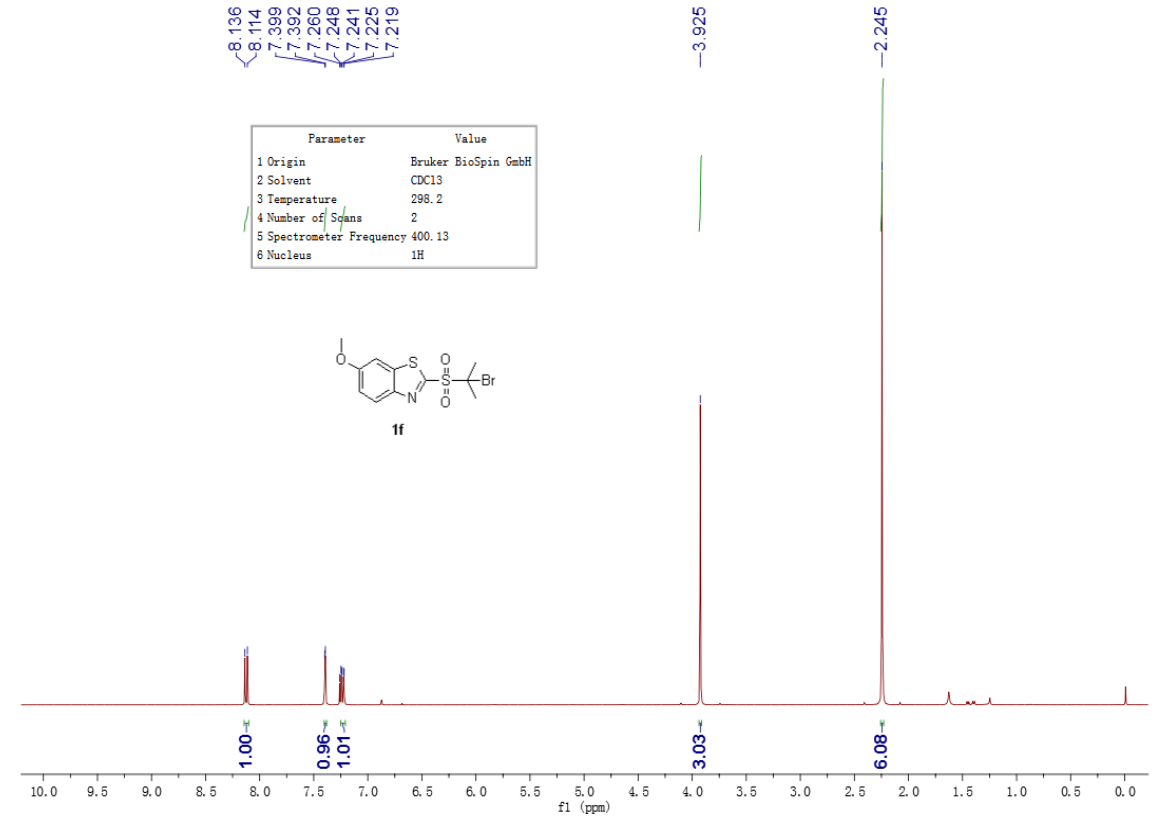

162.399
151.519
139.170
131.538
126.851
124.680
122.653
77.345
77.028
76.710
74.051
28.176
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Br
S
N
O
O
Br
1c
f1 (ppm)

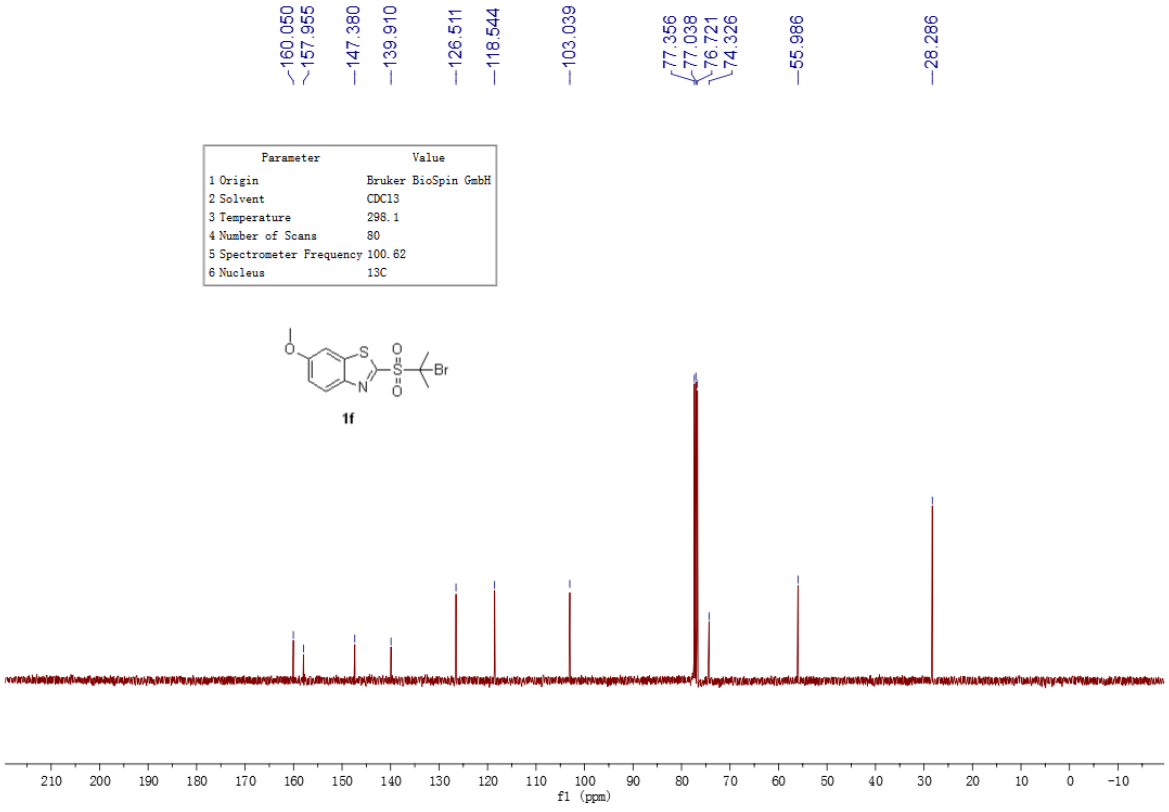

8.123
8.100
7.373
7.367
7.260
7.231
7.225
7.208
7.202
4.158
4.141
4.123
4.106
2.240
1.496
1.479
1.461
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Eto
Br
1d
1.00
1.01
0.98
2.04
6.01
3.05
f1 (ppm)

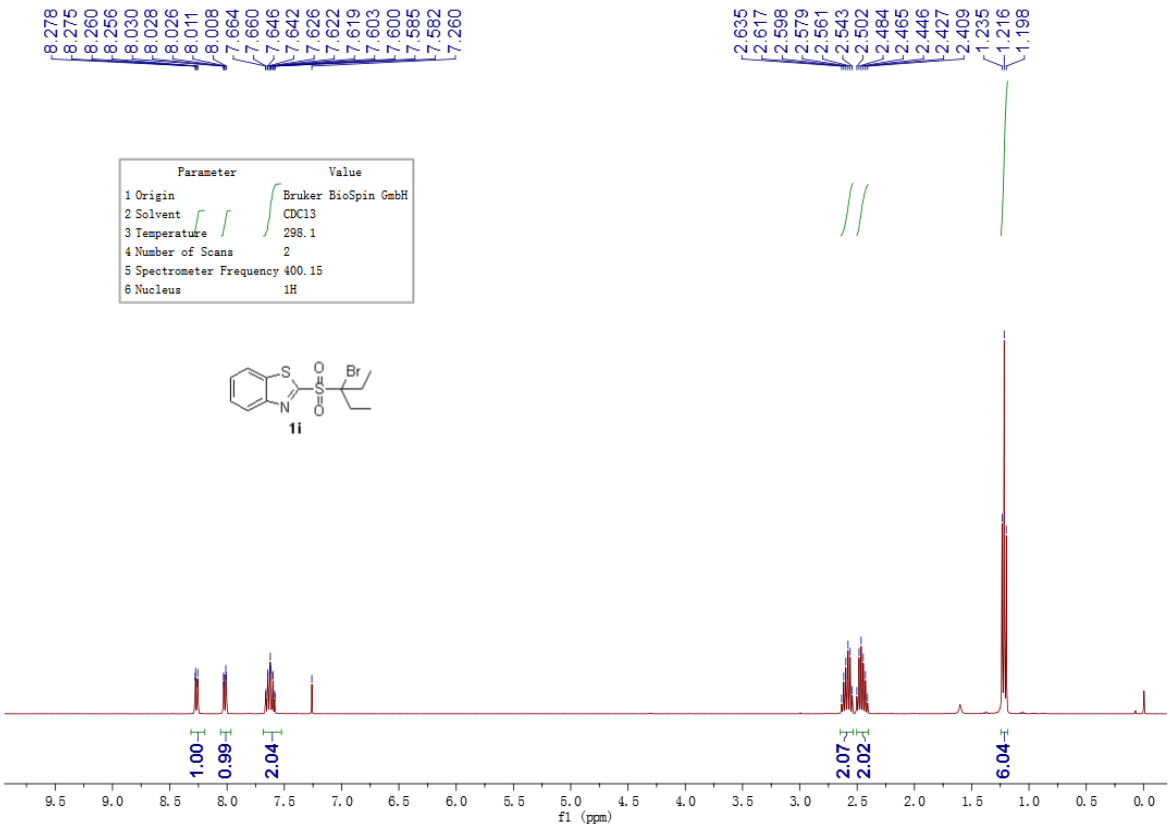

159.439
157.799
147.277
139.925
126.494
118.840
103.628
77.363
77.045
76.728
74.359
64.399
28.306
14.667
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Br
1d
f1 (ppm)

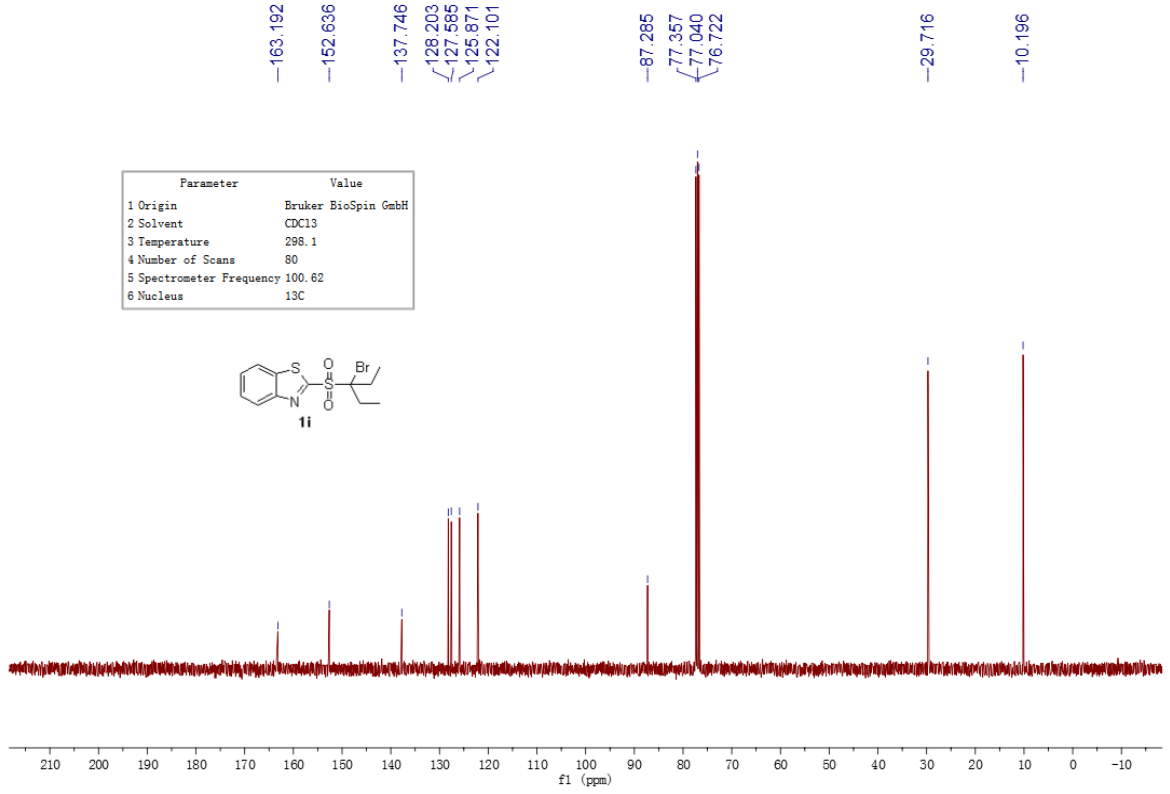

7.852
7.831
7.522
7.503
7.483
7.442
7.424
7.260
2.806
2.284
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
1e
1.00
1.05
1.00
2.98
5.99
f1 (ppm)

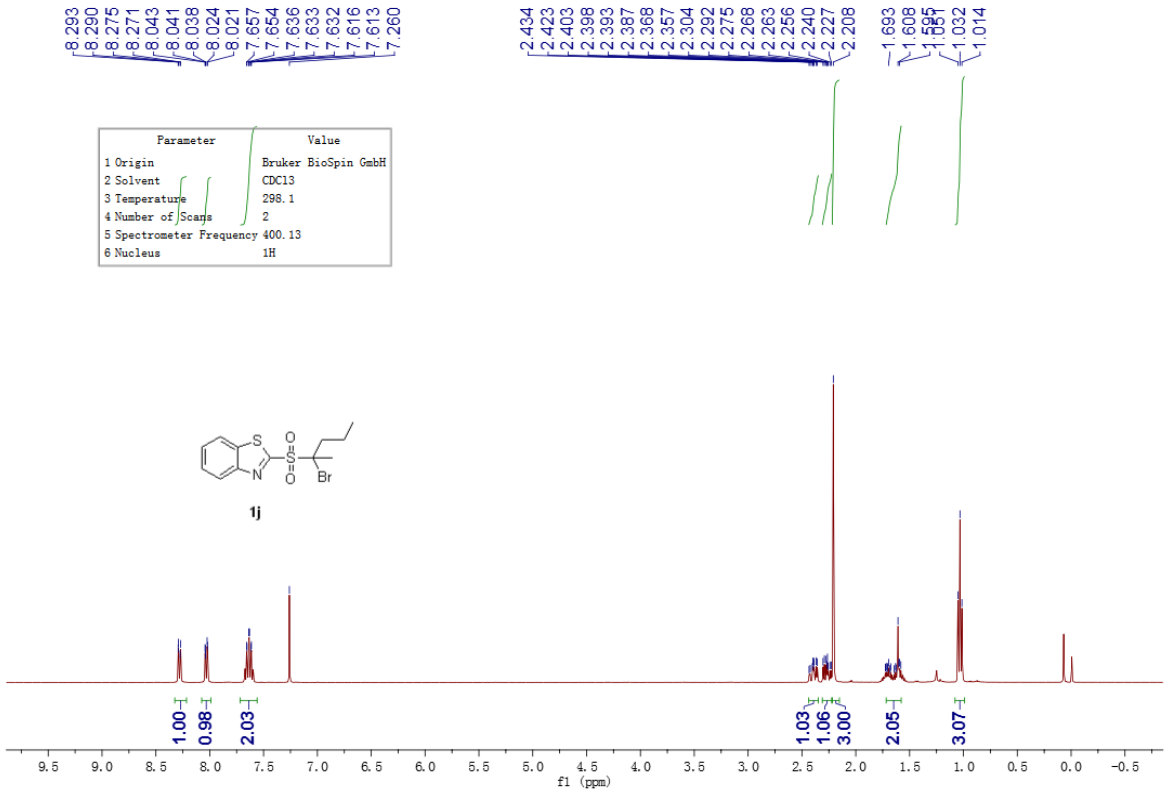

160.368
152.582
137.645
136.174
128.364
127.931
119.412
77.375
77.057
76.739
74.319
28.305
18.273
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1e
f1 (ppm)

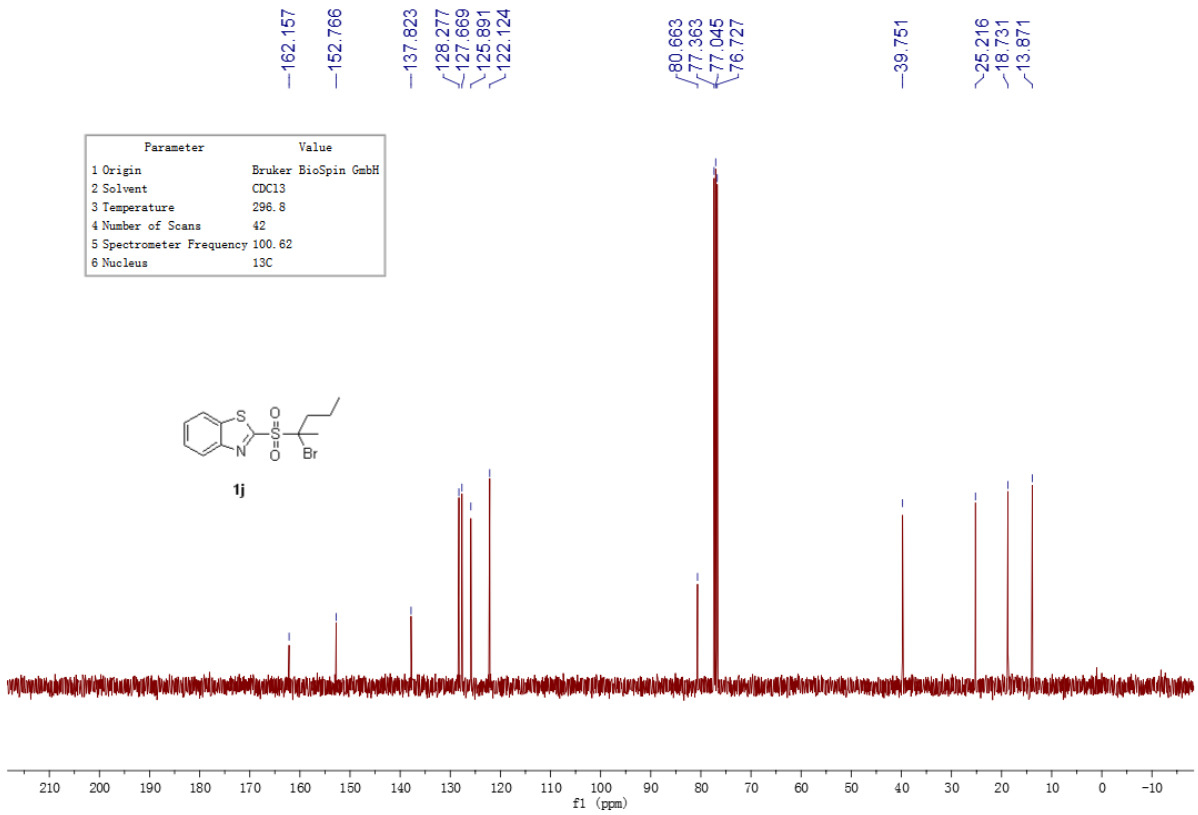

8.136
8.114
7.399
7.392
7.260
7.248
7.241
7.225
7.219
3.925
2.245
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
1f
1.00
0.96
1.01
3.03
6.08
f1 (ppm)

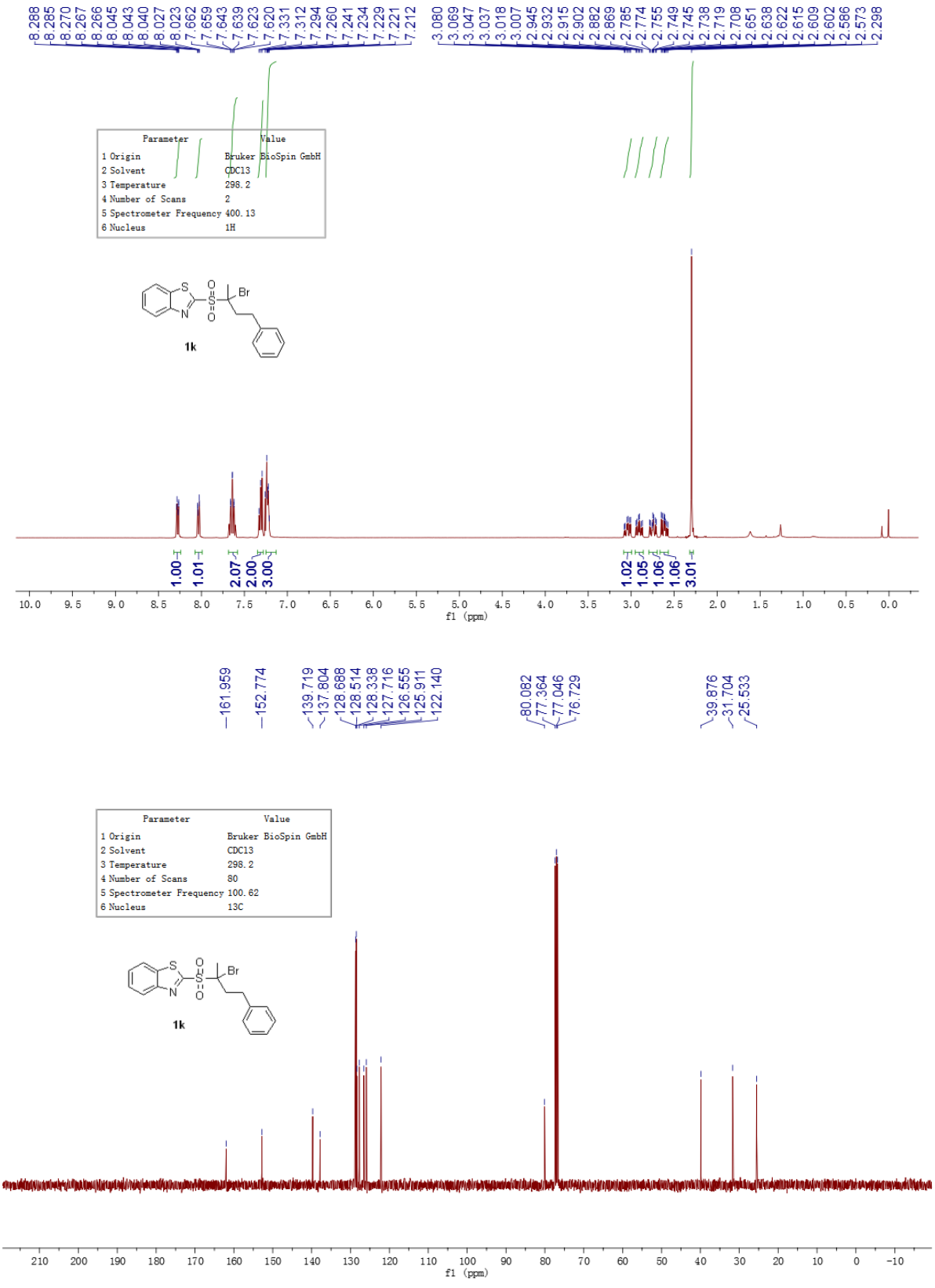

160.050
157.955
147.380
139.910
126.511
118.544
103.039
77.356
77.038
76.721
74.326
55.986
28.286
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1f
f1 (ppm)

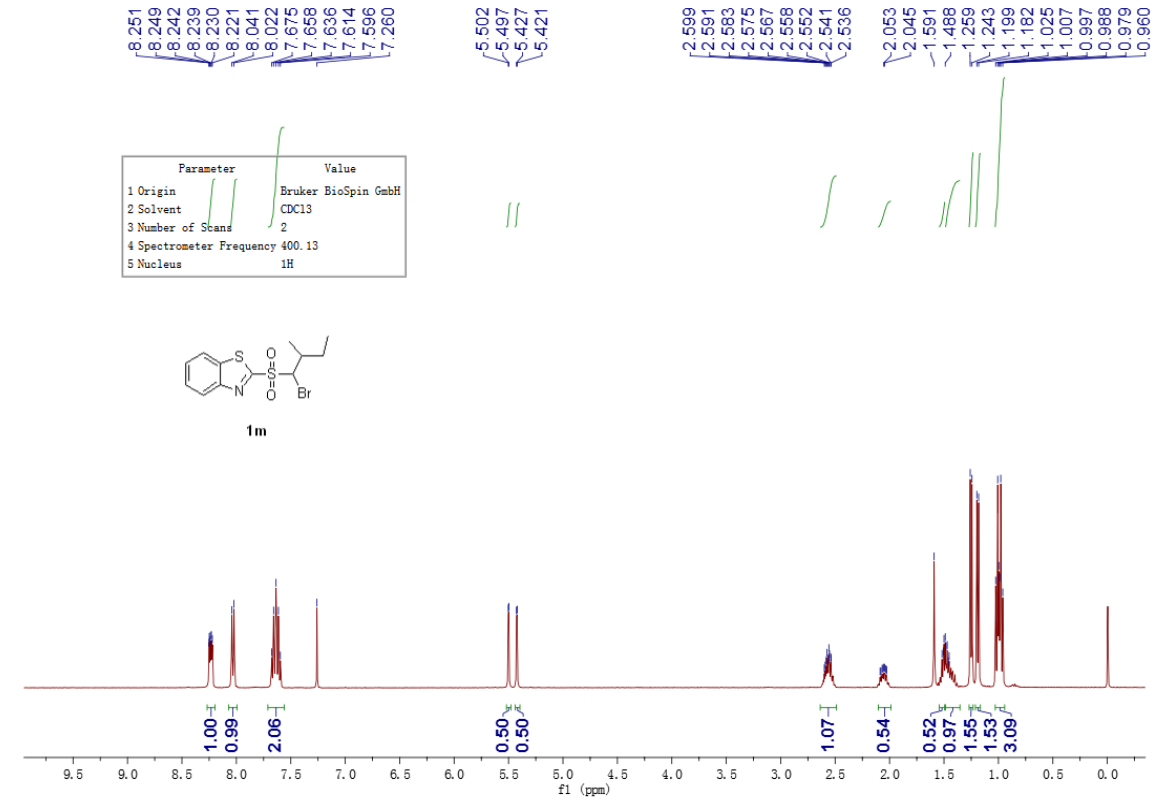

8.278
8.275
8.260
8.256
8.030
8.028
8.026
8.011
8.008
7.664
7.660
7.646
7.642
7.626
7.622
7.619
7.603
7.600
7.585
7.582
7.260
2.635
2.617
2.598
2.579
2.561
2.543
2.502
2.484
2.465
2.446
2.427
2.409
1.235
1.216
1.198
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
1i
1.00
0.99
2.04
2.07
2.02
6.04
f1 (ppm)

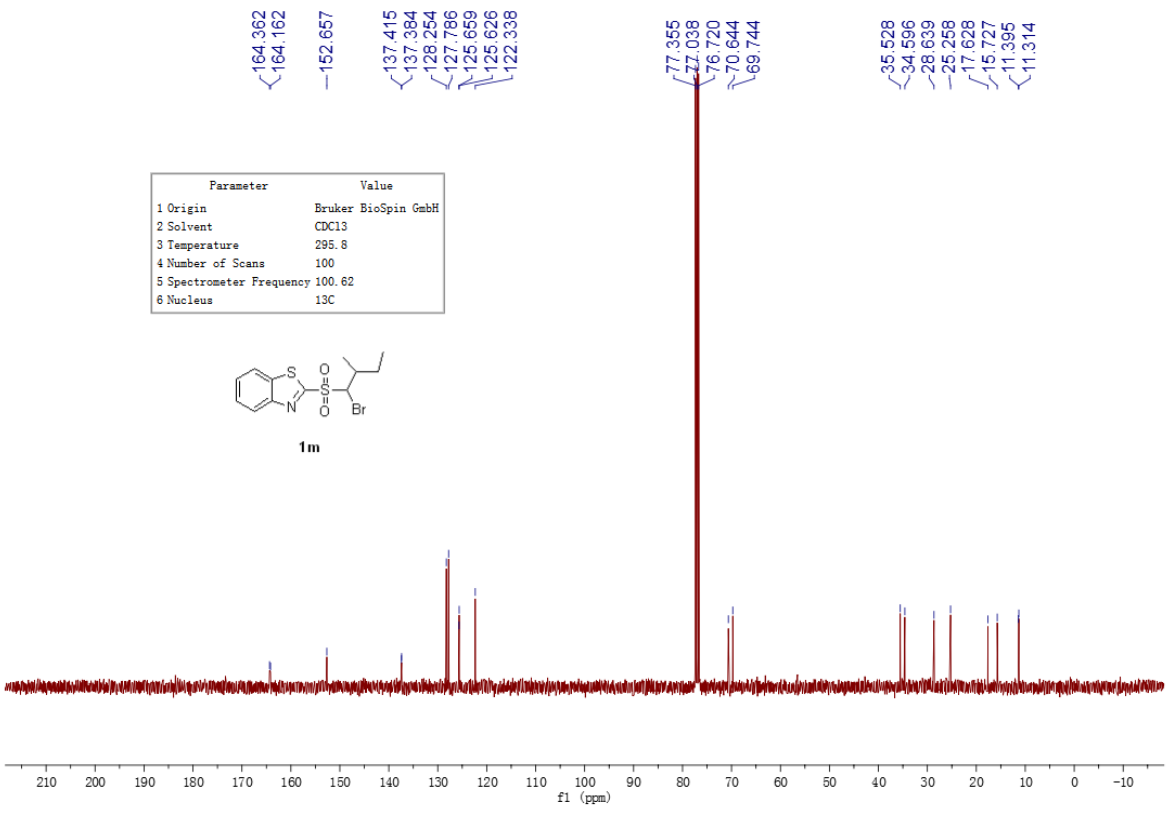

163.192
152.636
137.746
128.203
127.585
125.871
122.101
87.285
77.357
77.040
76.722
29.716
10.196
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1i
f1 (ppm)

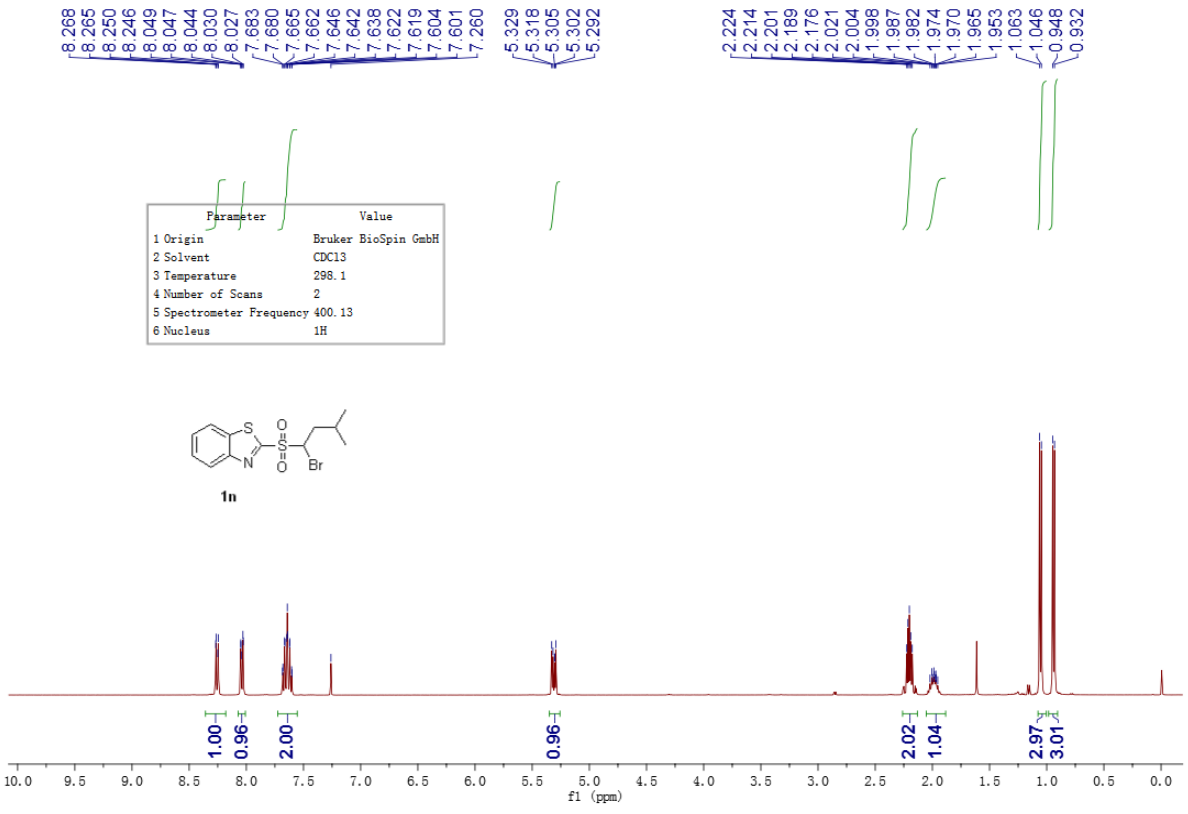

8.293
8.290
8.275
8.271
8.043
8.041
8.038
8.024
8.021
7.657
7.654
7.636
7.633
7.632
7.616
7.613
7.260
2.434
2.423
2.403
2.398
2.393
2.387
2.368
2.357
2.304
2.292
2.275
2.268
2.263
2.256
2.240
2.227
2.208
1.693
1.608
1.595
1.551
1.032
1.014
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
1j
1.00
0.98
2.03
1.03
1.06
3.00
2.05
3.07
f1 (ppm)

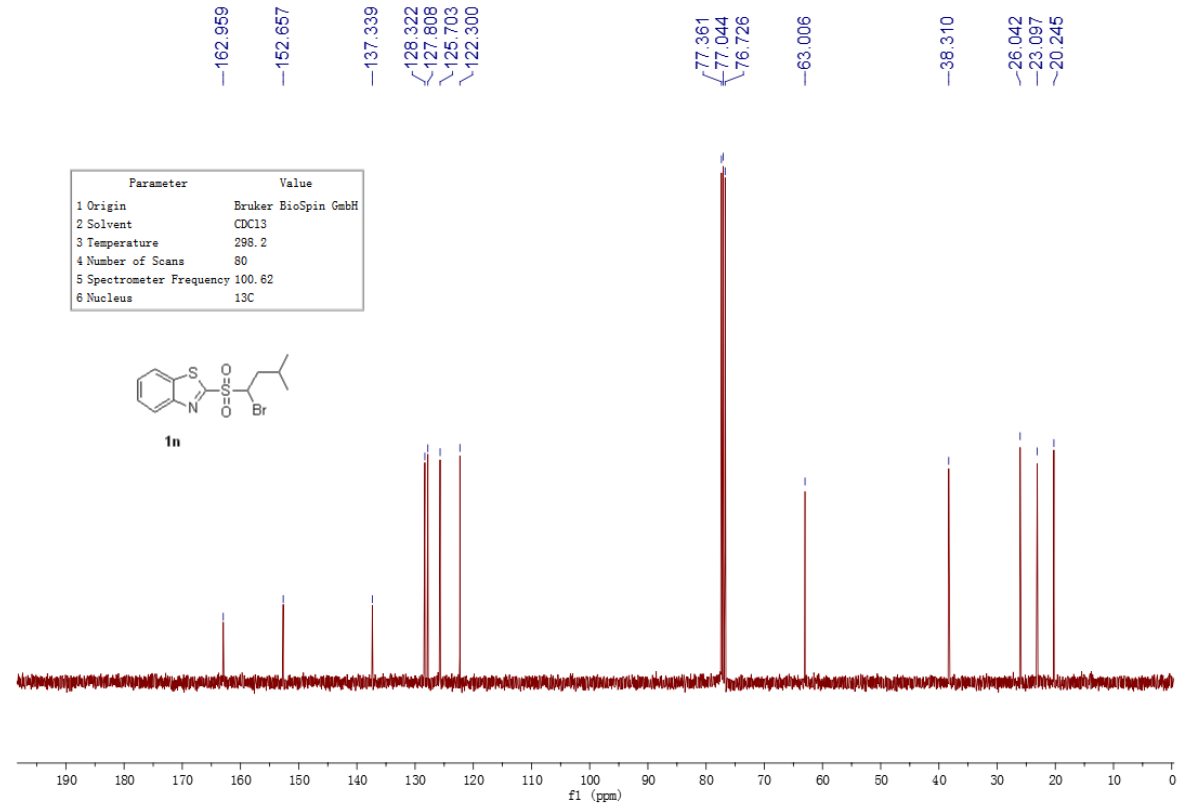

162.157
152.766
137.823
128.277
127.669
125.891
122.124
80.663
77.363
77.045
76.727
39.751
25.216
18.731
13.871
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.8
4 Number of Scans 42
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1j
f1 (ppm)

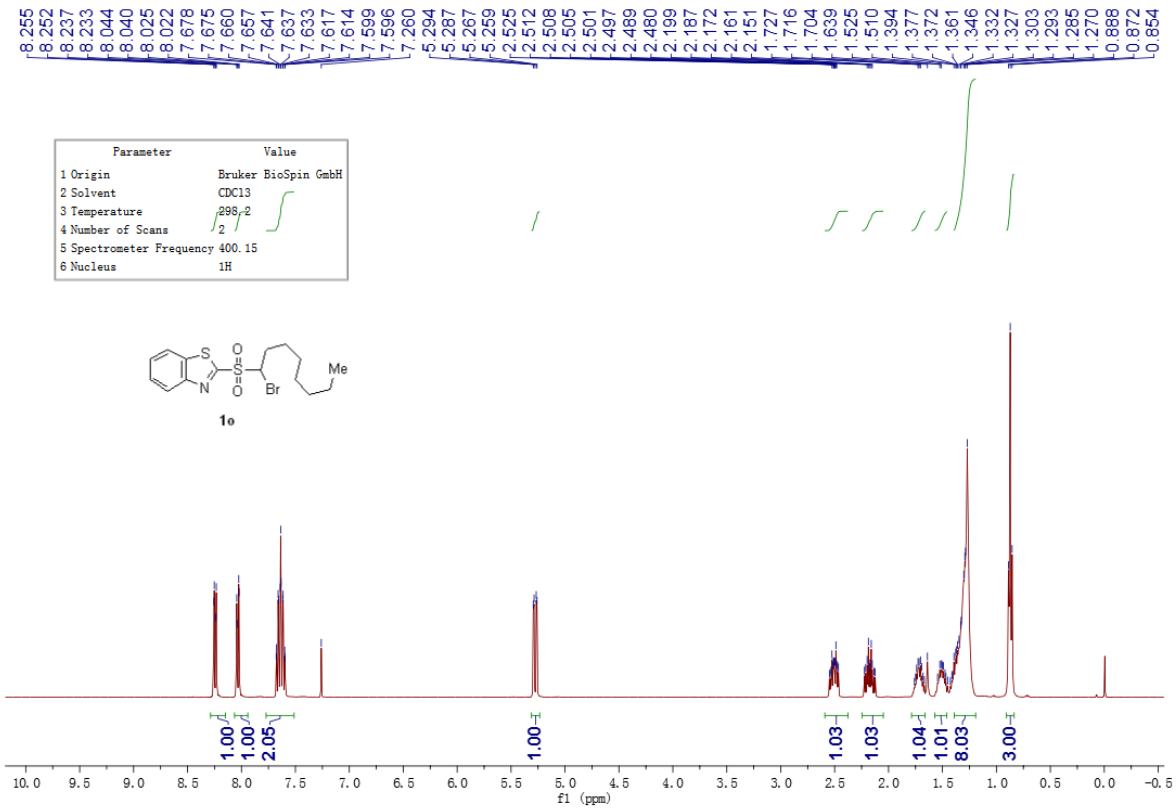

8.288
8.285
8.270
8.267
8.266
8.045
8.043
8.040
8.027
8.023
7.662
7.659
7.643
7.639
7.623
7.620
7.331
7.312
7.294
7.260
7.241
7.234
7.229
7.221
7.212
3.080
3.069
3.047
3.037
3.018
3.007
2.945
2.932
2.915
2.902
2.882
2.869
2.785
2.774
2.755
2.749
2.745
2.738
2.719
2.708
2.651
2.638
2.622
2.615
2.609
2.602
2.586
2.573
2.298
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
1k
1.00
1.01
2.07
2.00
3.00
1.02
1.05
1.06
1.06
3.01
f1 (ppm)
161.959
152.774
139.719
137.804
128.688
128.514
128.338
127.716
126.555
125.911
122.140
80.082
77.364
77.046
76.729
39.876
31.704
25.533
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1k
f1 (ppm)

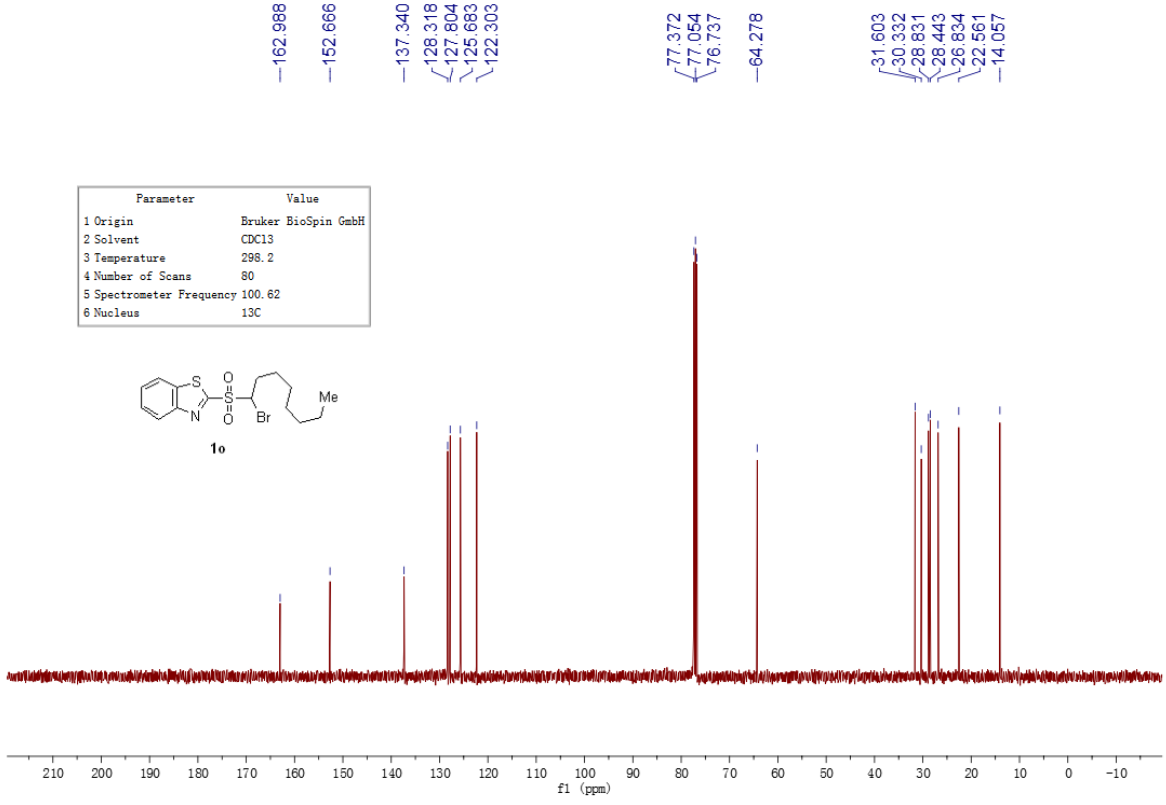

8.251
8.249
8.242
8.239
8.230
8.221
8.041
8.022
7.675
7.658
7.636
7.614
7.596
7.260
5.502
5.497
5.427
5.421
2.599
2.591
2.583
2.575
2.567
2.558
2.552
2.541
2.536
2.053
2.045
1.591
1.488
1.259
1.243
1.199
1.182
1.025
1.007
0.997
0.988
0.979
0.960
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Number of Scans 2
4 Spectrometer Frequency 400.13
5 Nucleus 1H
1m
1.00
0.99
2.06
0.50
0.50
1.07
0.54
0.52
0.97
1.55
1.53
3.09
f1 (ppm)

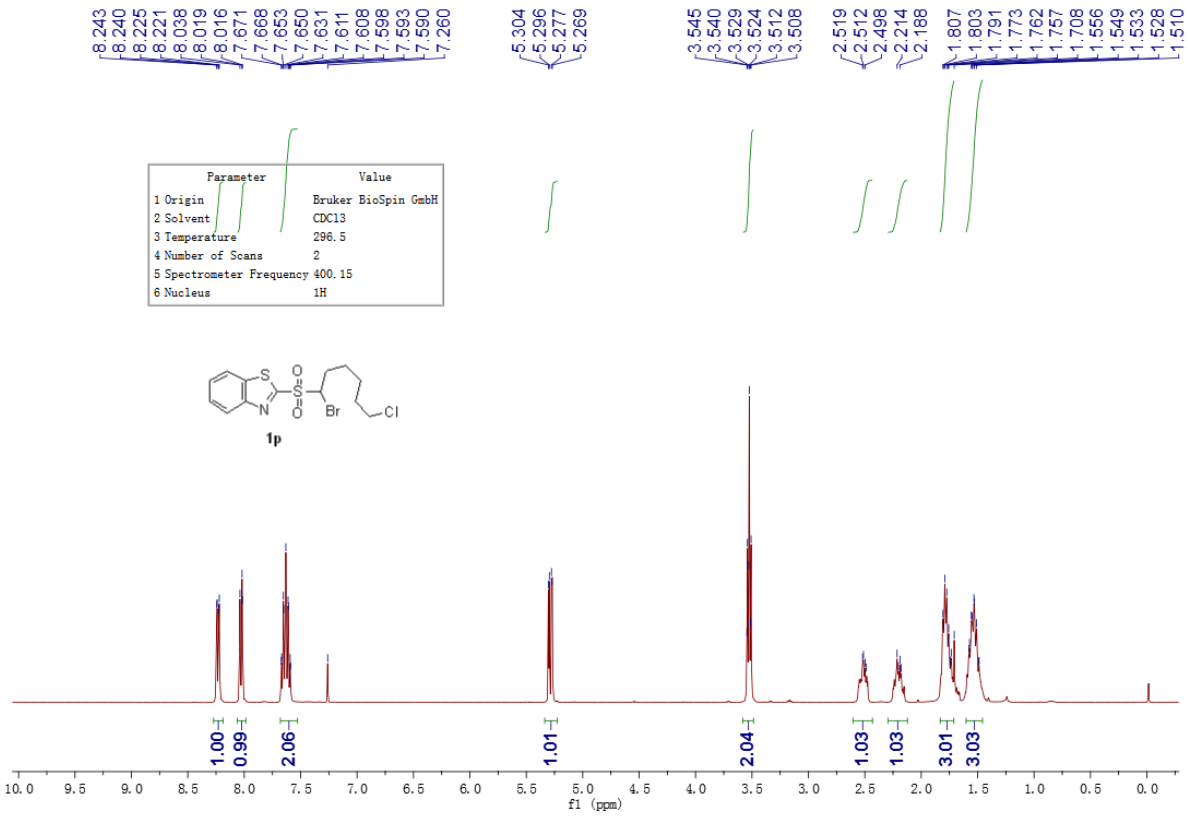

164.362
164.162
152.657
137.415
137.384
128.254
127.786
125.659
125.626
122.338
77.355
77.038
76.720
70.644
69.744
35.528
34.596
28.639
25.258
17.628
15.727
11.395
11.314
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.8
4 Number of Scans 100
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1m
f1 (ppm)

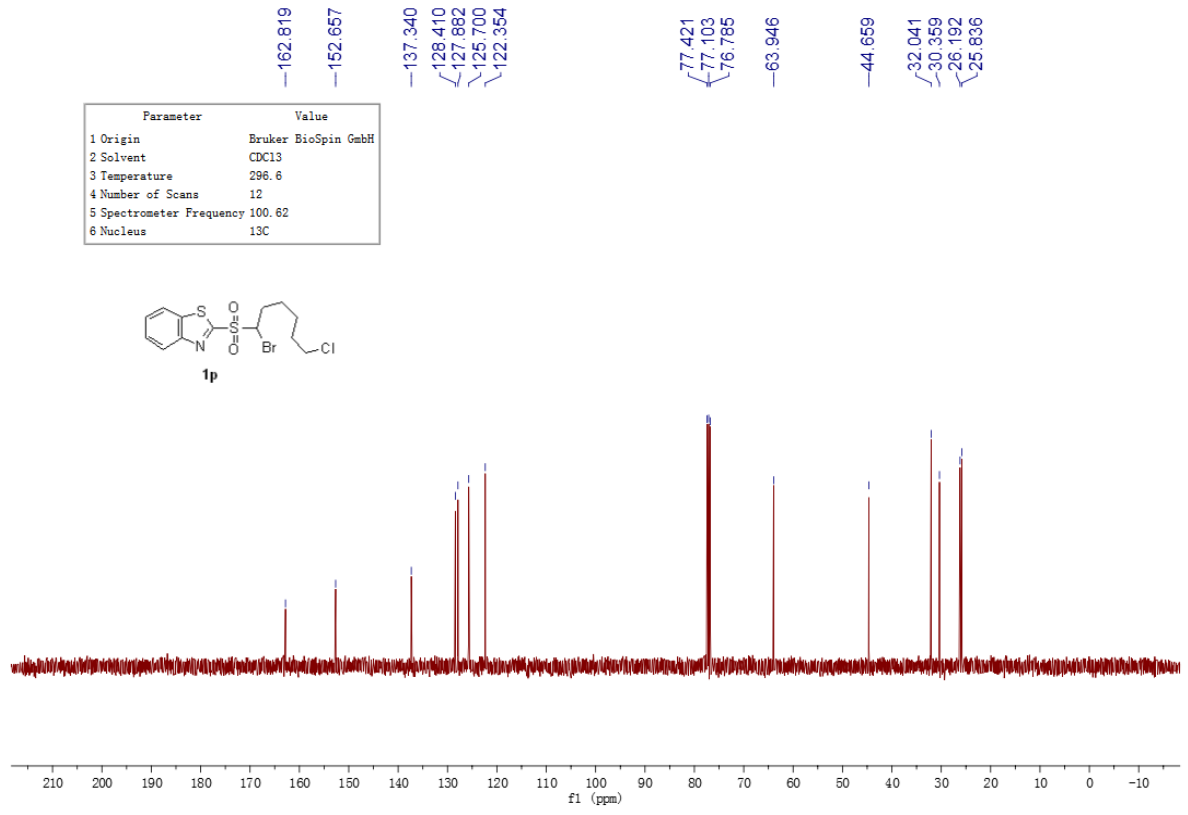

8.268
8.265
8.250
8.246
8.049
8.047
8.044
8.030
8.027
7.683
7.680
7.665
7.662
7.646
7.642
7.638
7.622
7.619
7.604
7.601
7.260
5.329
5.318
5.305
5.302
5.292
2.224
2.214
2.201
2.189
2.176
2.021
2.004
1.998
1.987
1.982
1.974
1.970
1.965
1.953
1.063
1.046
0.948
0.932
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
1n
1.00
0.96
2.00
0.96
2.02
1.04
2.97
3.01
f1 (ppm)

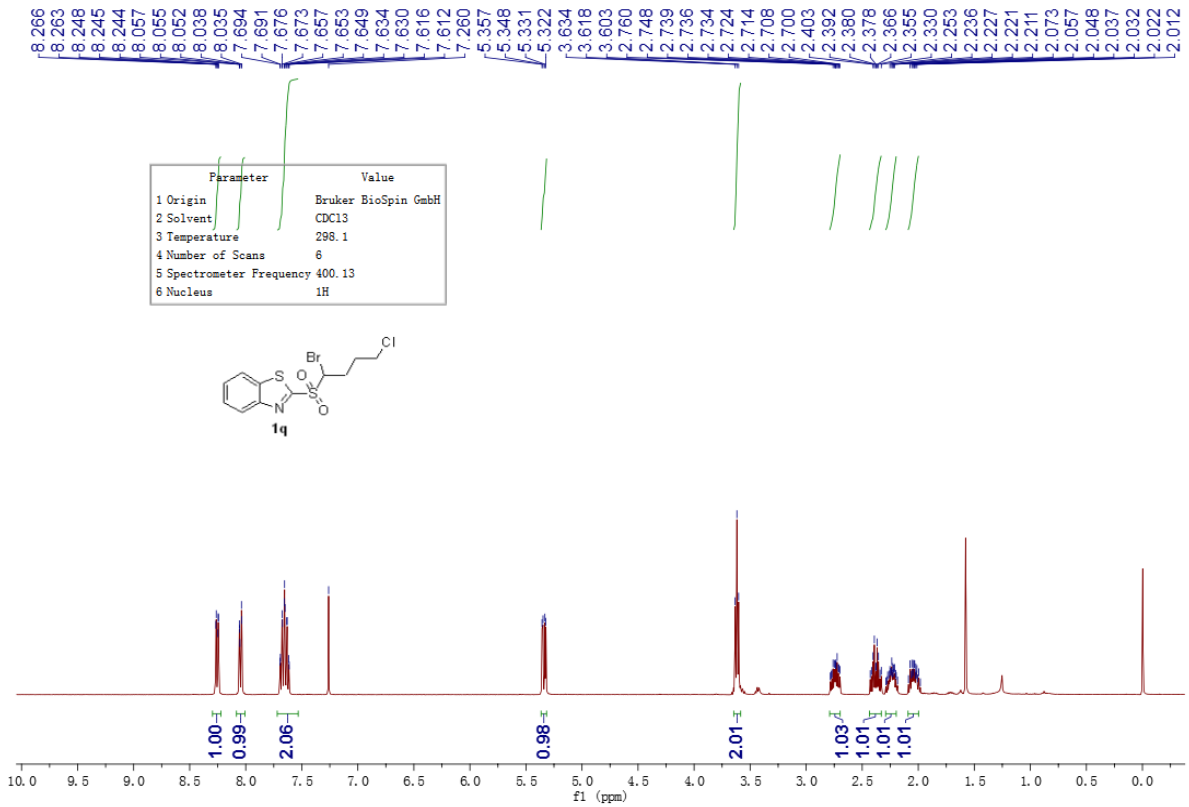

162.959
152.657
137.339
128.322
127.808
125.703
122.300
77.361
77.044
76.726
63.006
38.310
26.042
23.097
20.245
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1n
f1 (ppm)

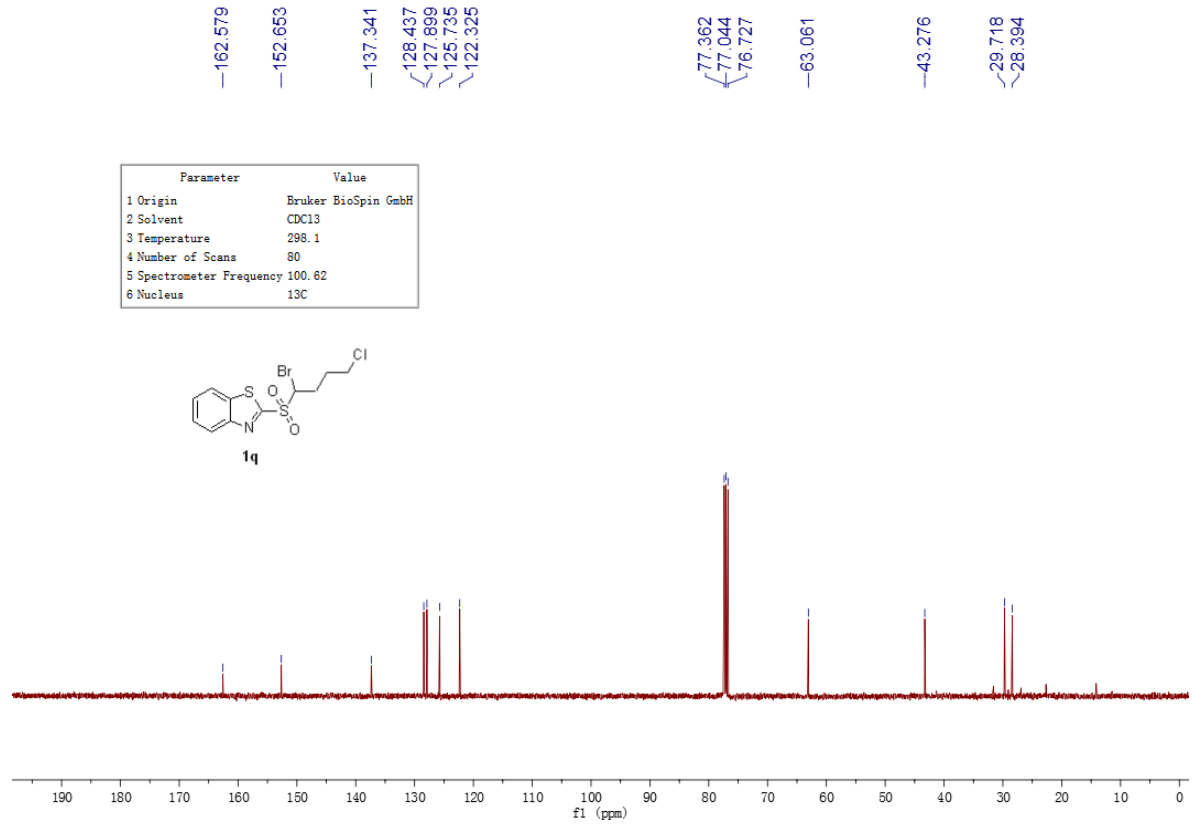

8.255
8.252
8.237
8.233
8.044
8.040
8.025
8.022
7.678
7.675
7.660
7.657
7.641
7.637
7.633
7.617
7.614
7.599
7.596
7.260
5.294
5.287
5.267
5.259
2.525
2.512
2.508
2.505
2.501
2.497
2.489
2.480
2.199
2.187
2.172
2.161
2.151
1.727
1.716
1.704
1.639
1.525
1.510
1.394
1.377
1.372
1.361
1.346
1.332
1.327
1.303
1.293
1.285
1.270
0.888
0.872
0.854
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
Me
Br
1o
1.00
1.00
2.05
1.00
1.03
1.03
1.04
1.01
8.03
3.00
f1 (ppm)

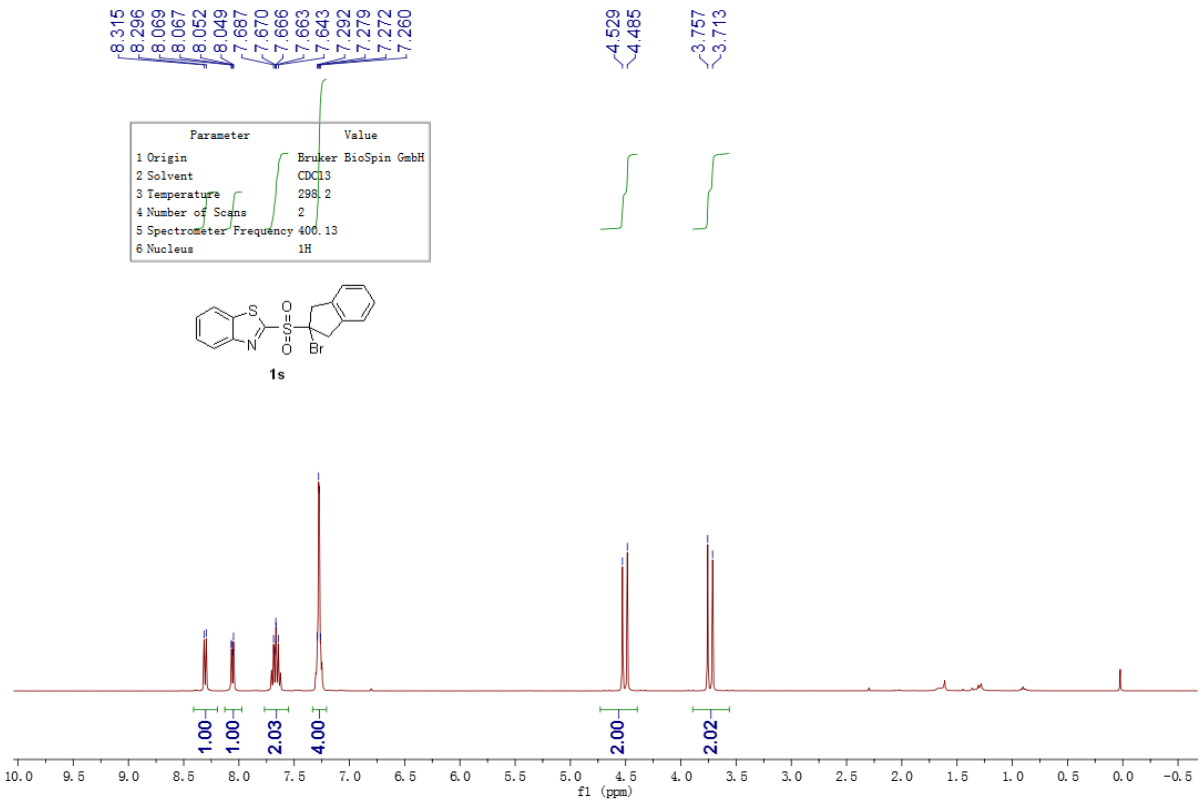

162.988
152.666
137.340
128.318
127.804
125.683
122.303
77.372
77.054
76.737
64.278
31.603
30.332
28.831
28.443
26.834
22.561
14.057
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Me
Br
1o
f1 (ppm)

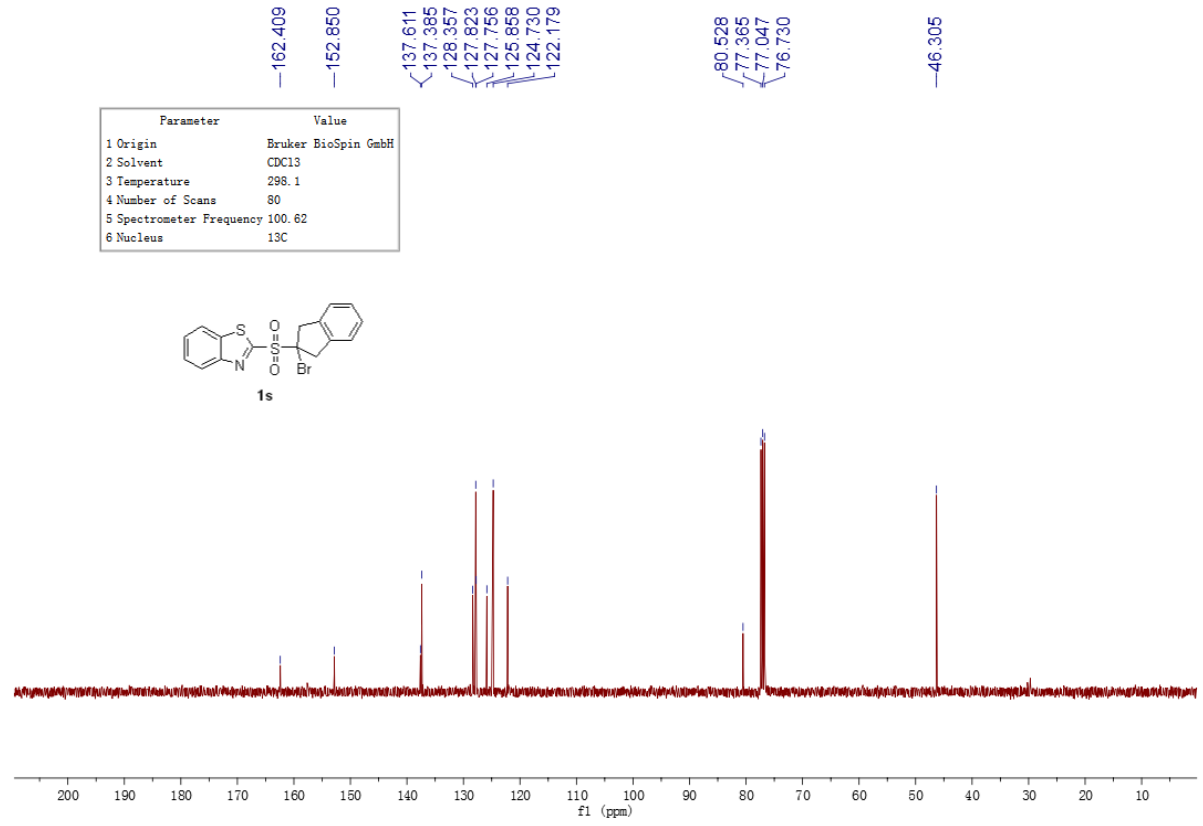

8.243
8.240
8.225
8.221
8.038
8.019
8.016
7.671
7.668
7.653
7.650
7.631
7.611
7.608
7.598
7.593
7.590
7.260
5.304
5.296
5.277
5.269
3.545
3.540
3.529
3.524
3.512
3.508
2.519
2.512
2.498
2.214
2.188
1.807
1.803
1.791
1.773
1.762
1.757
1.708
1.556
1.549
1.533
1.528
1.510
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.5
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
1p
1.00
0.99
2.06
1.01
2.04
1.03
1.03
3.01
3.03
f1 (ppm)

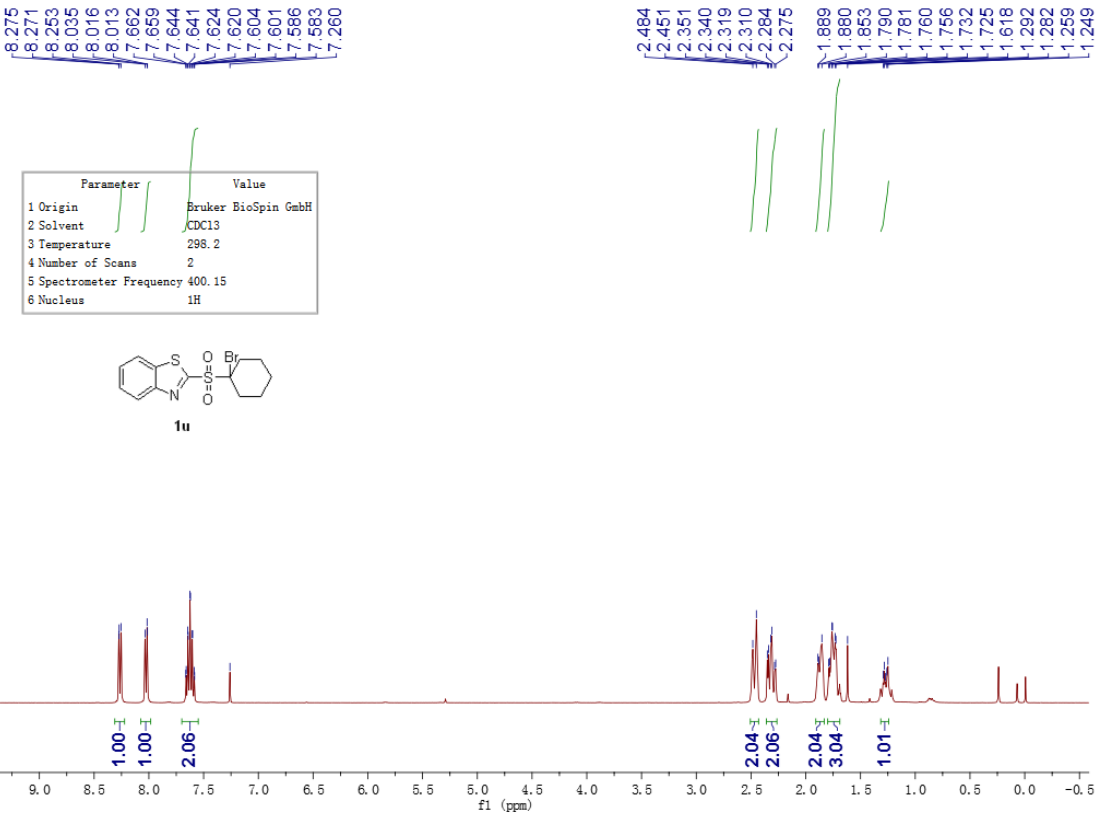

162.819
152.657
137.340
128.410
127.882
125.700
122.354
77.421
77.103
76.785
63.946
44.659
32.041
30.359
26.192
25.836
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.6
4 Number of Scans 12
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1p
f1 (ppm)

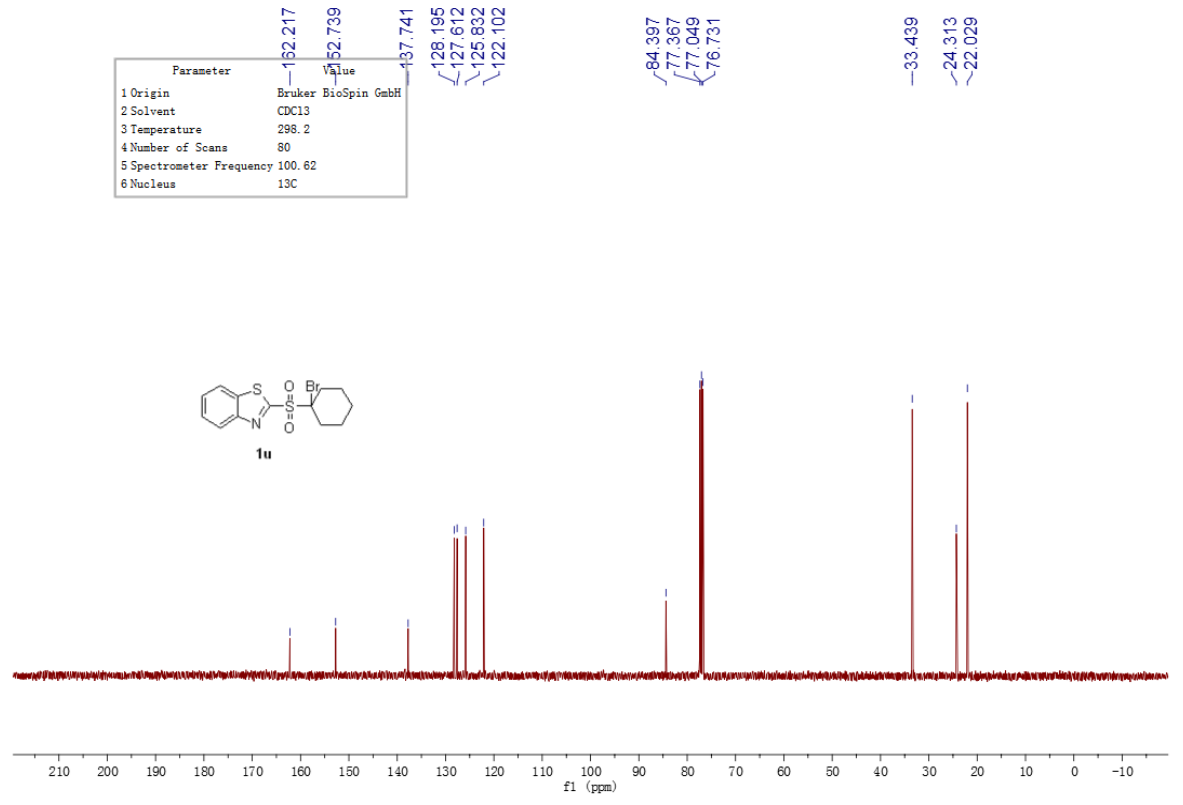

8.266
8.263
8.248
8.245
8.244
8.057
8.055
8.052
8.038
8.035
7.694
7.691
7.676
7.673
7.657
7.653
7.649
7.634
7.630
7.616
7.612
7.260
5.357
5.348
5.331
5.322
3.634
3.618
3.603
2.760
2.748
2.739
2.736
2.734
2.724
2.714
2.708
2.700
2.403
2.392
2.380
2.378
2.366
2.355
2.330
2.253
2.236
2.227
2.221
2.211
2.073
2.057
2.048
2.037
2.032
2.022
2.012
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 6
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Br
Cl
S
O
N
O
O
1q
1.00
0.99
2.06
0.98
2.01
1.03
1.01
1.01
1.01
10.0 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 0.5 0.0
f1 (ppm)

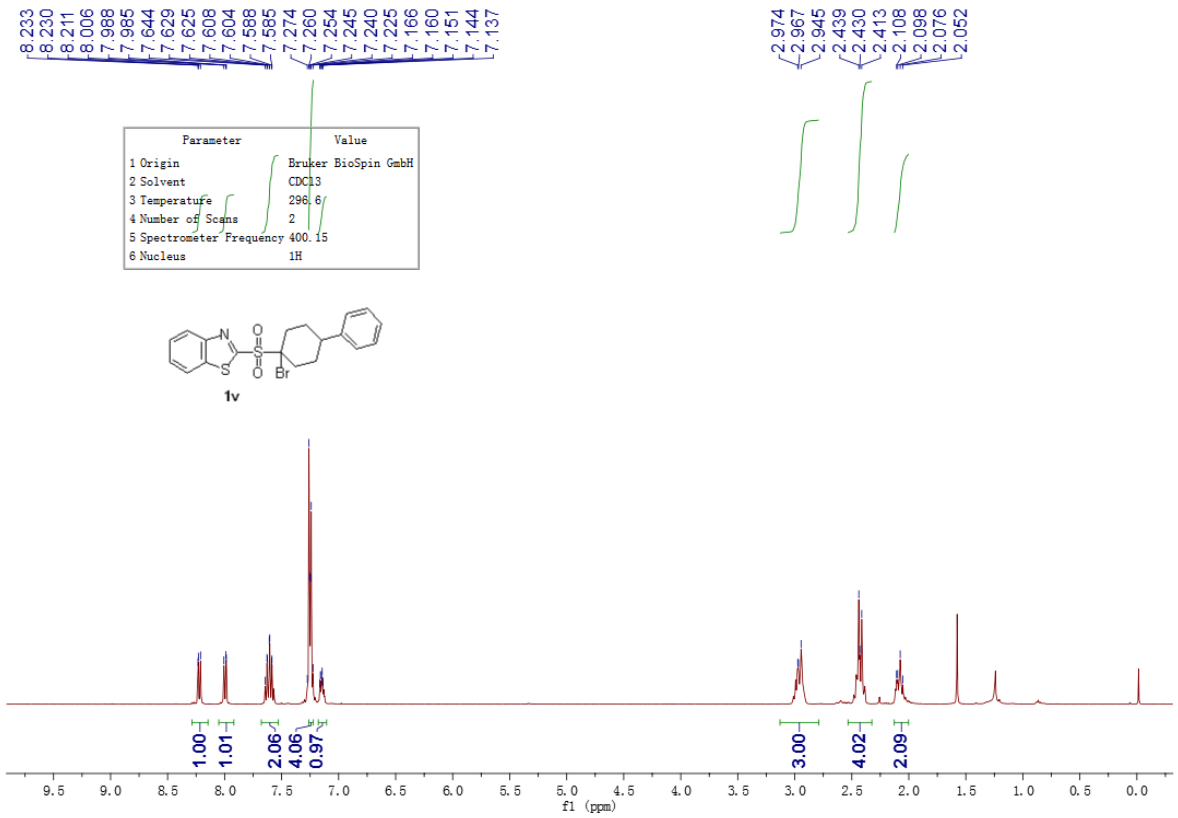

162.579
152.653
137.341
128.437
127.899
125.735
122.325
77.362
77.044
76.727
63.061
43.276
29.718
28.394
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Br
Cl
S
O
N
O
O
1q
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

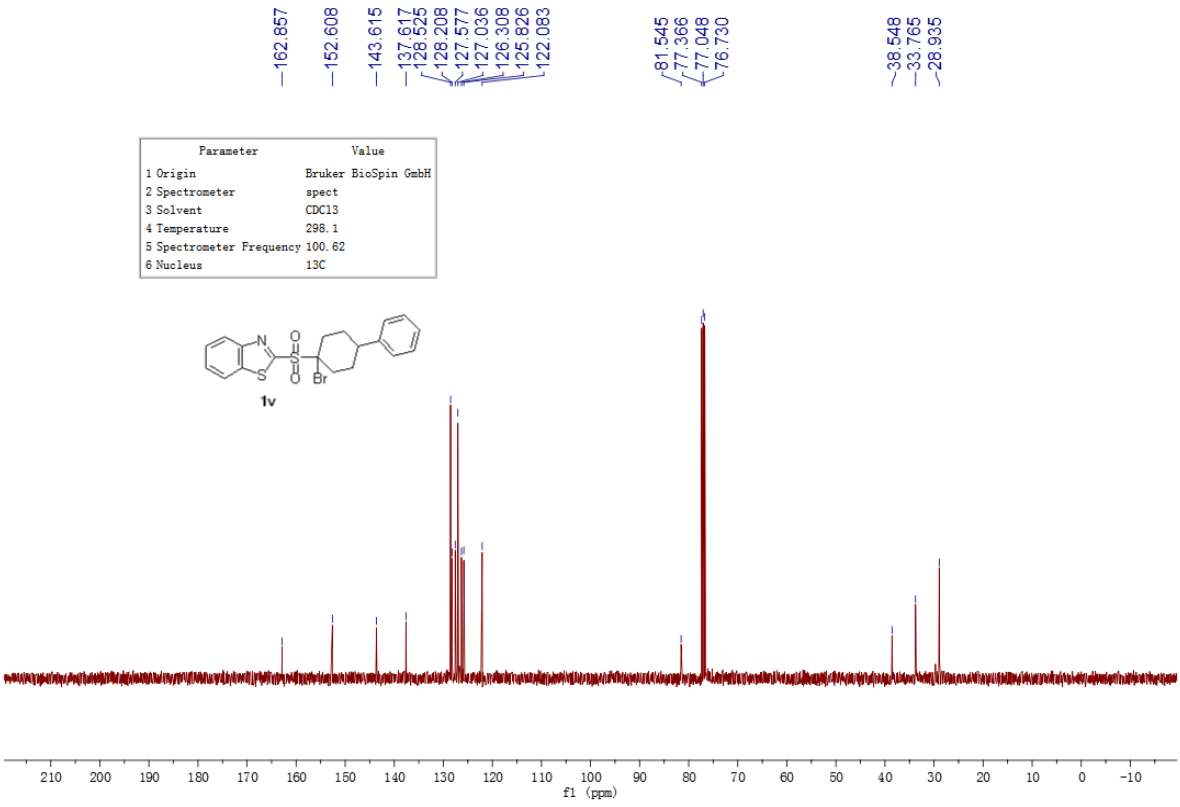

8.315
8.296
8.069
8.067
8.052
8.049
7.687
7.670
7.666
7.663
7.643
7.292
7.279
7.272
7.260
4.529
4.485
3.757
3.713
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
1s
1.00
1.00
2.03
4.00
2.00
2.02
f1 (ppm)

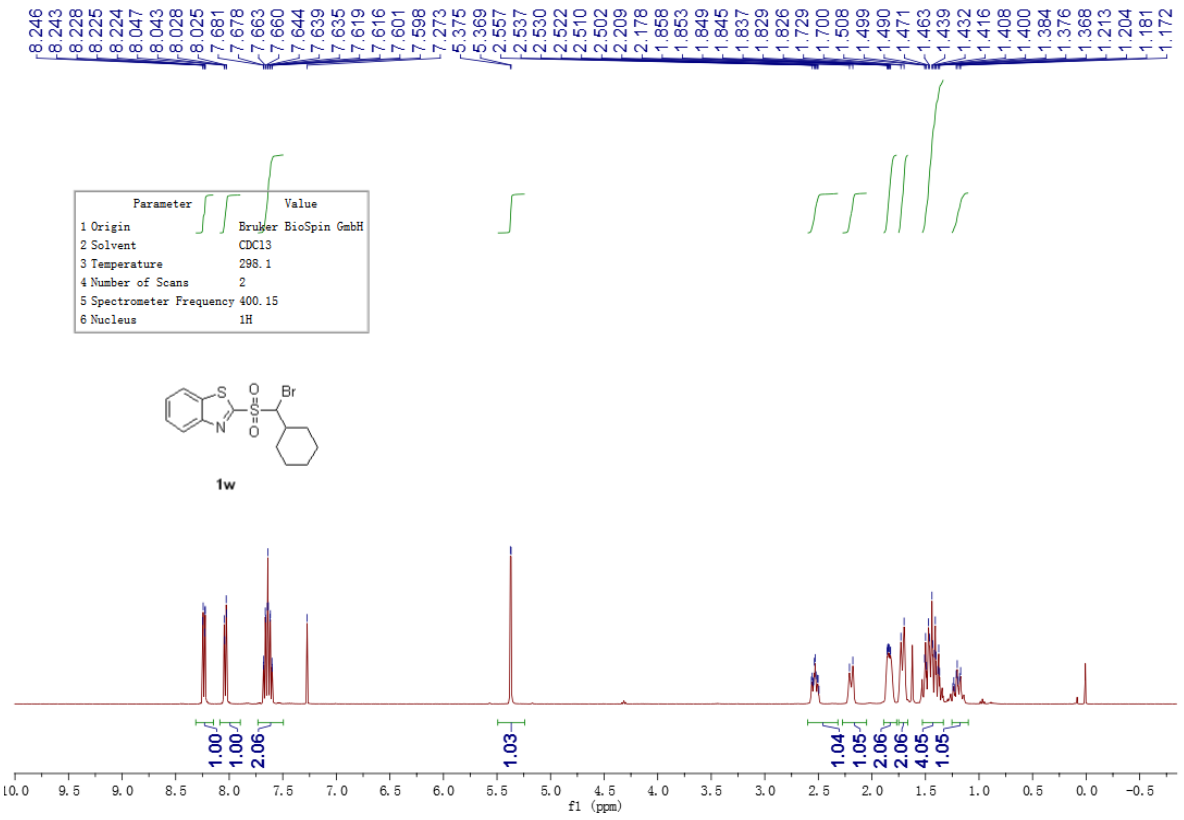

162.409
152.850
137.611
137.385
128.357
127.823
127.756
125.858
124.730
122.179
80.528
77.365
77.047
76.730
46.305
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1s
f1 (ppm)

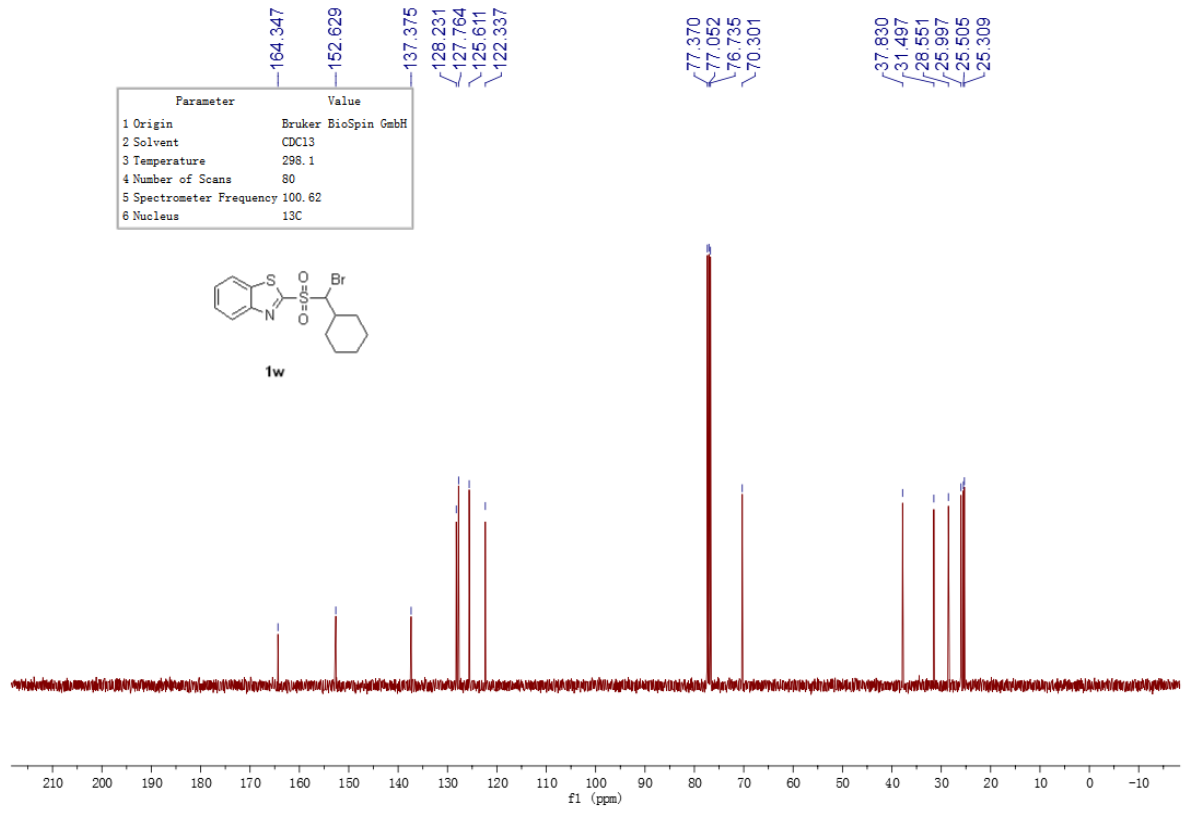

8.275
8.271
8.253
8.035
8.016
8.013
7.662
7.659
7.644
7.641
7.624
7.620
7.604
7.601
7.586
7.583
7.260
2.484
2.451
2.351
2.340
2.319
2.310
2.284
2.275
1.889
1.880
1.853
1.790
1.781
1.760
1.756
1.732
1.725
1.618
1.292
1.282
1.259
1.249
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
1u
1.00
1.00
2.06
2.04
2.06
2.04
3.04
1.01
f1 (ppm)

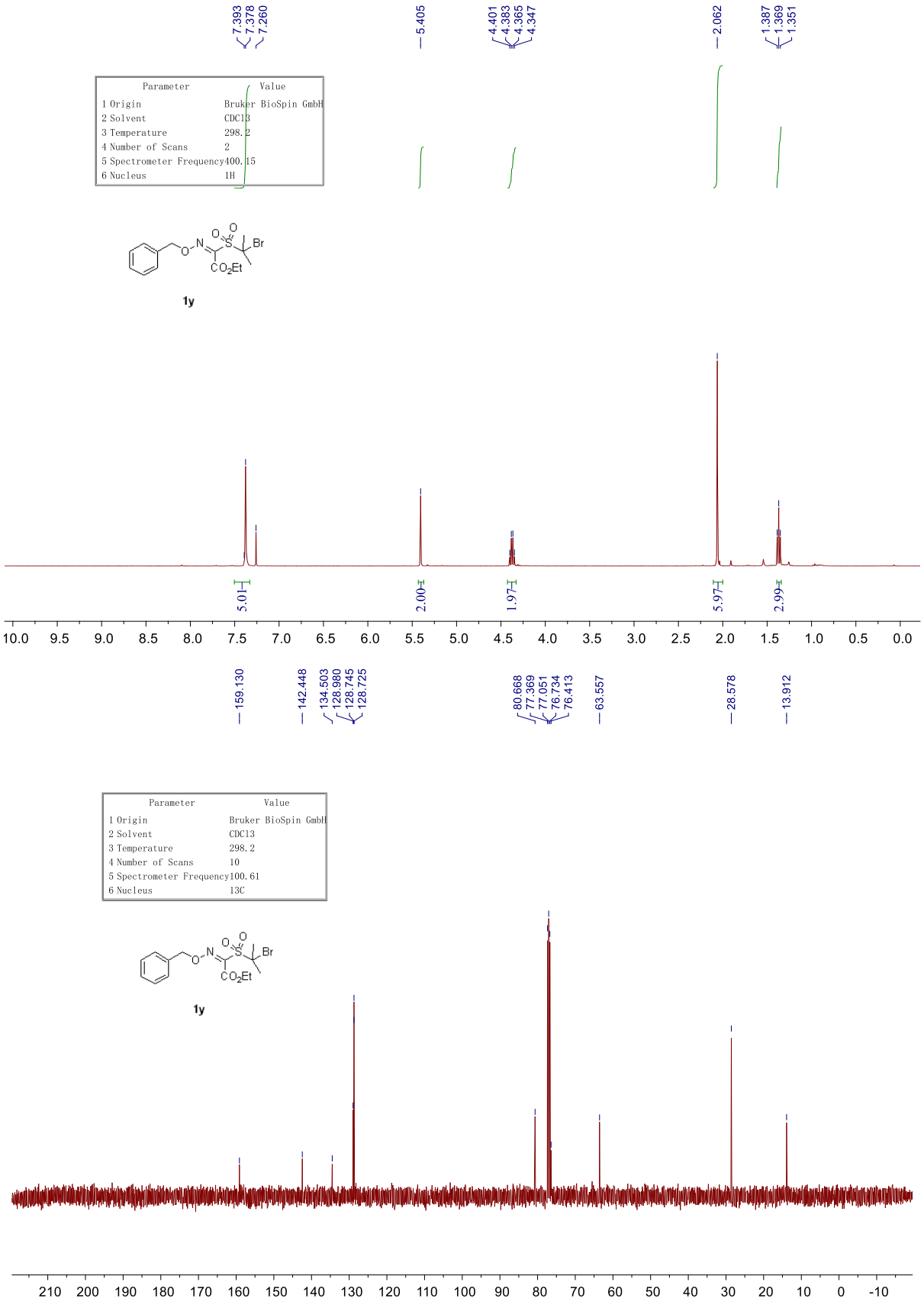

152.217
152.739
137.741
128.195
127.612
125.832
122.102
84.397
77.367
77.049
76.731
33.439
24.313
22.029
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1u
f1 (ppm)

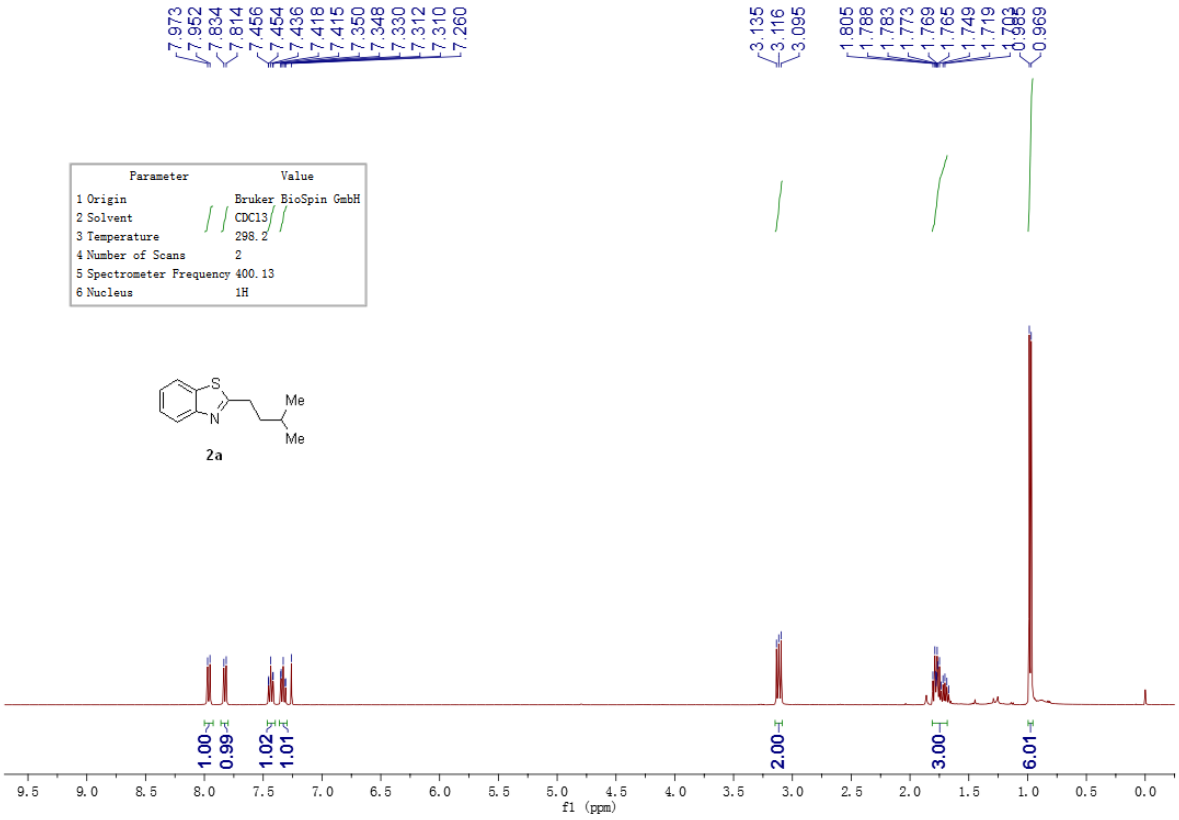

8.233
8.230
8.211
8.006
7.988
7.985
7.644
7.629
7.625
7.608
7.604
7.588
7.585
7.274
7.260
7.254
7.245
7.240
7.225
7.166
7.160
7.151
7.144
7.137
2.974
2.967
2.945
2.439
2.430
2.413
2.108
2.098
2.076
2.052
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.6
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
1v
1.00
1.01
2.06
4.06
0.97
3.00
4.02
2.09
f1 (ppm)

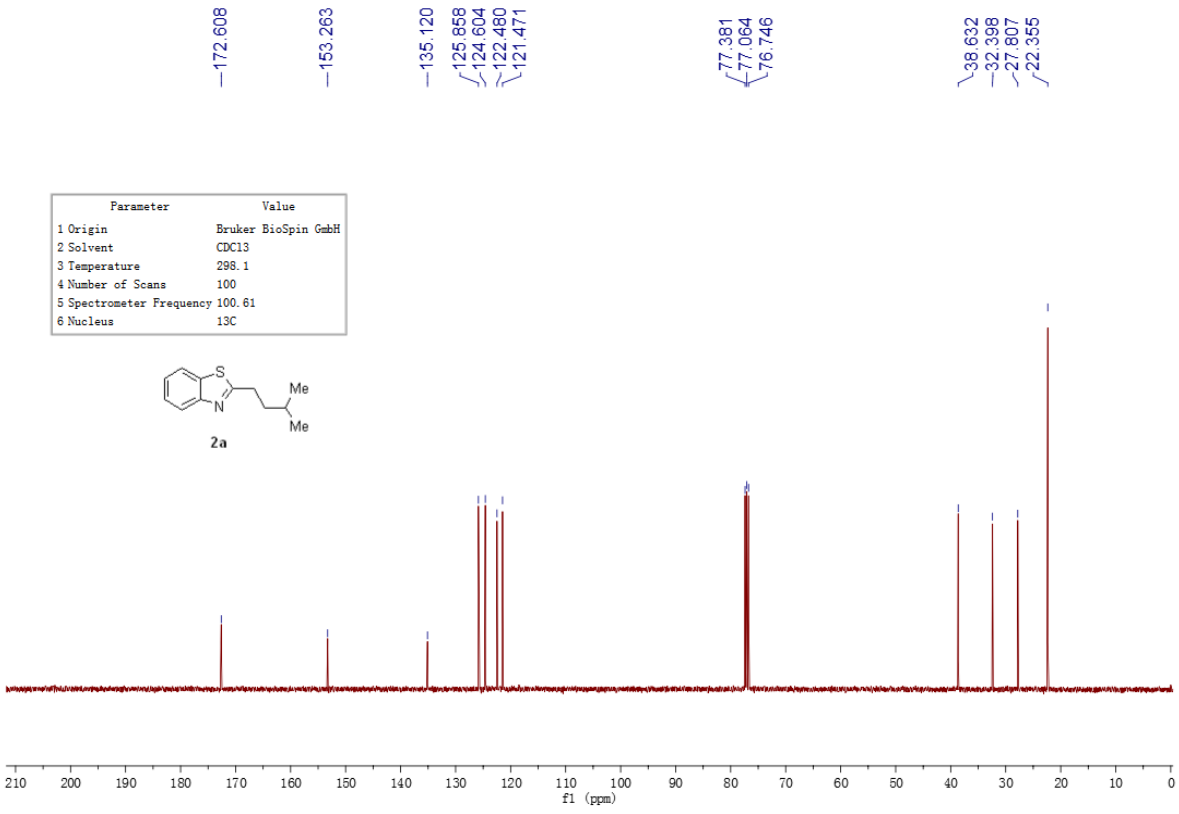

162.857
152.608
143.615
137.617
128.525
128.208
127.577
127.036
126.308
125.826
122.083
81.545
77.366
77.048
76.730
38.548
33.765
28.935
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Spectrometer spect
3 Solvent CDCl3
4 Temperature 298.1
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1v
f1 (ppm)

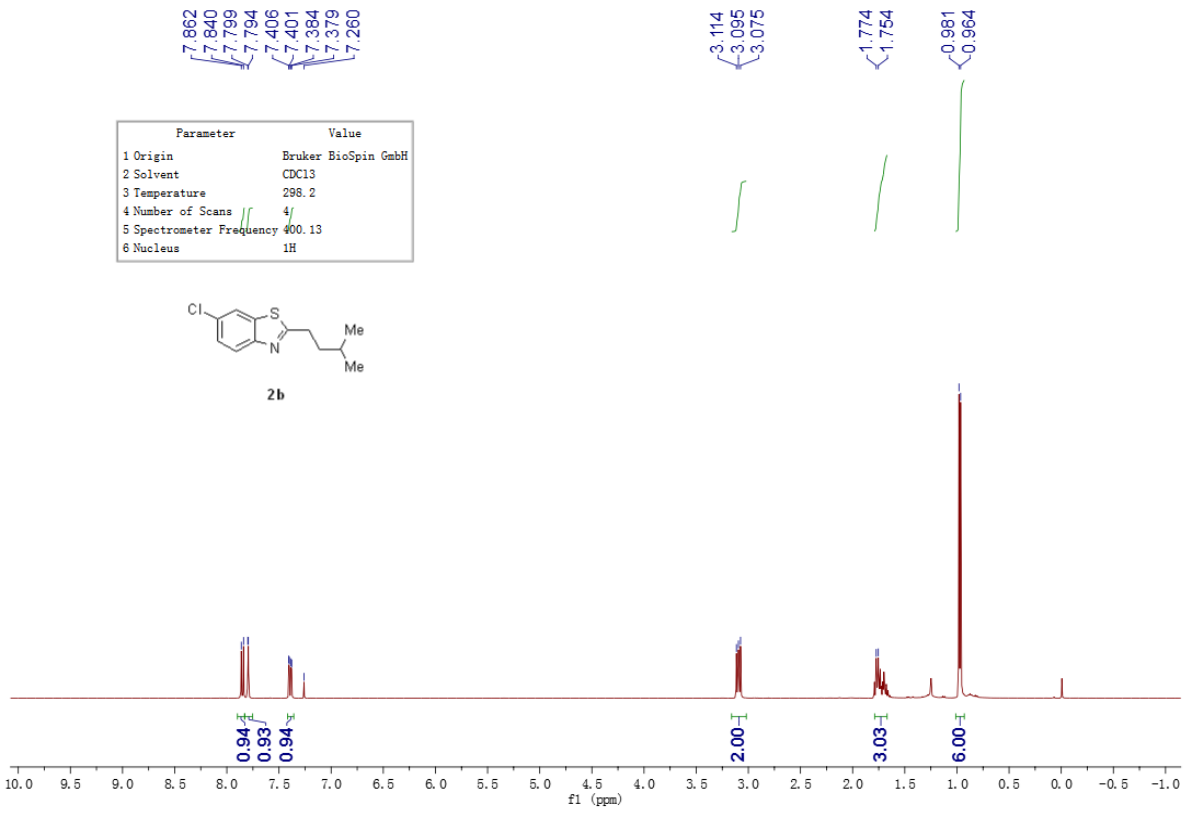

8.246
8.243
8.228
8.225
8.224
8.047
8.043
8.028
8.025
7.681
7.678
7.663
7.660
7.644
7.639
7.635
7.619
7.616
7.601
7.598
7.273
5.375
5.369
2.557
2.537
2.530
2.522
2.510
2.502
2.209
2.178
1.858
1.853
1.849
1.845
1.837
1.829
1.826
1.729
1.700
1.508
1.499
1.490
1.471
1.463
1.439
1.432
1.416
1.408
1.400
1.384
1.376
1.368
1.213
1.204
1.181
1.172
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
1w
1.00
1.00
2.06
1.03
1.04
1.05
2.06
2.06
4.05
1.05
f1 (ppm)

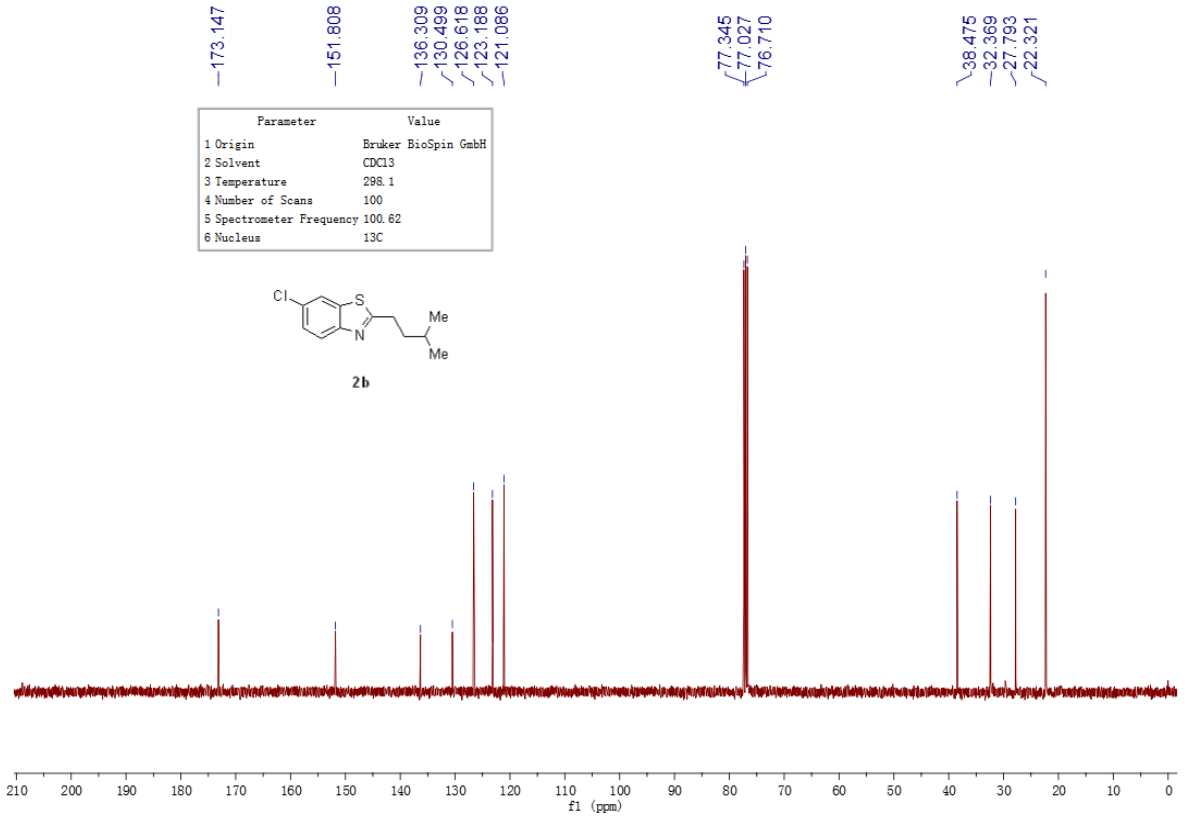

164.347
152.629
137.375
128.231
127.764
125.611
122.337
77.370
77.052
76.735
70.301
37.830
31.497
28.551
25.997
25.505
25.309
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
1w
f1 (ppm)

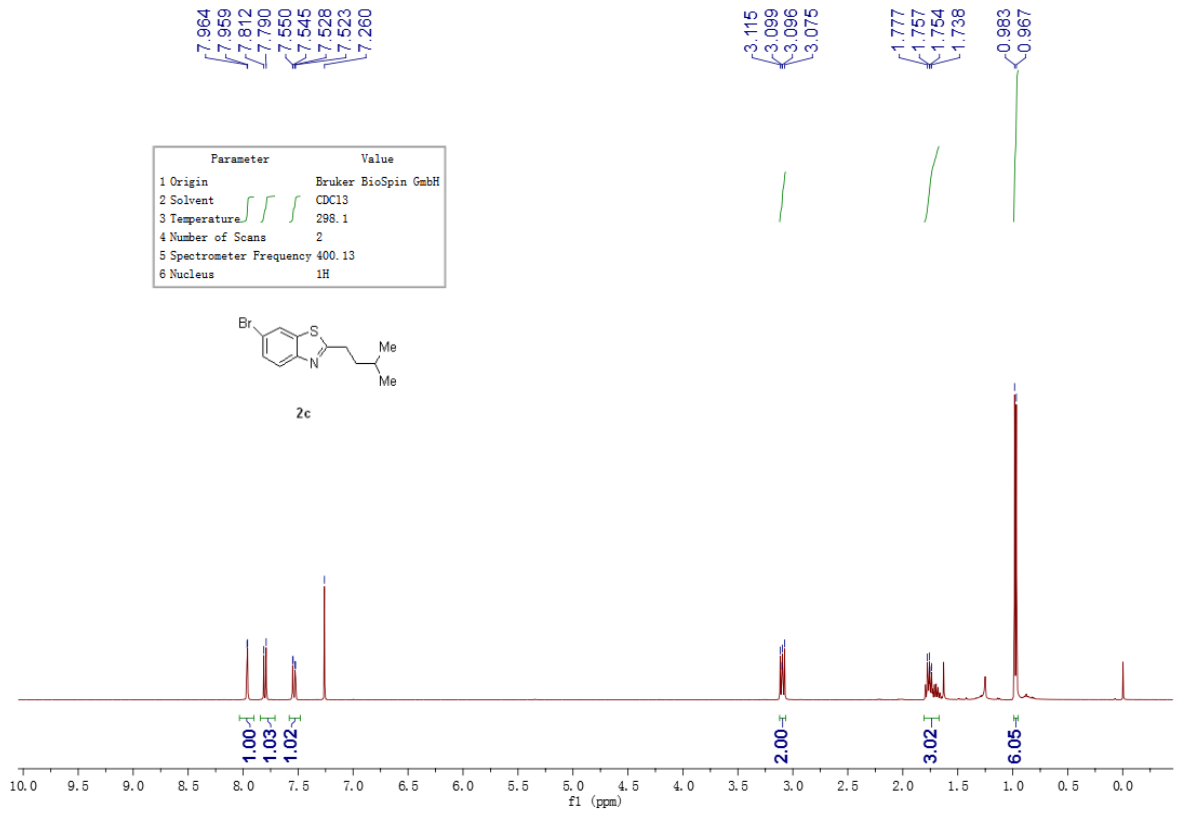

7.393
7.378
7.260
5.405
4.401
4.383
4.365
4.347
2.062
1.387
1.369
1.351
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
1y
5.01
2.00
1.97
5.97
2.99
159.130
142.448
134.503
128.980
128.745
128.725
80.668
77.369
77.051
76.734
76.413
63.557
28.578
13.912
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 10
5 Spectrometer Frequency 100.61
6 Nucleus 13C
1y

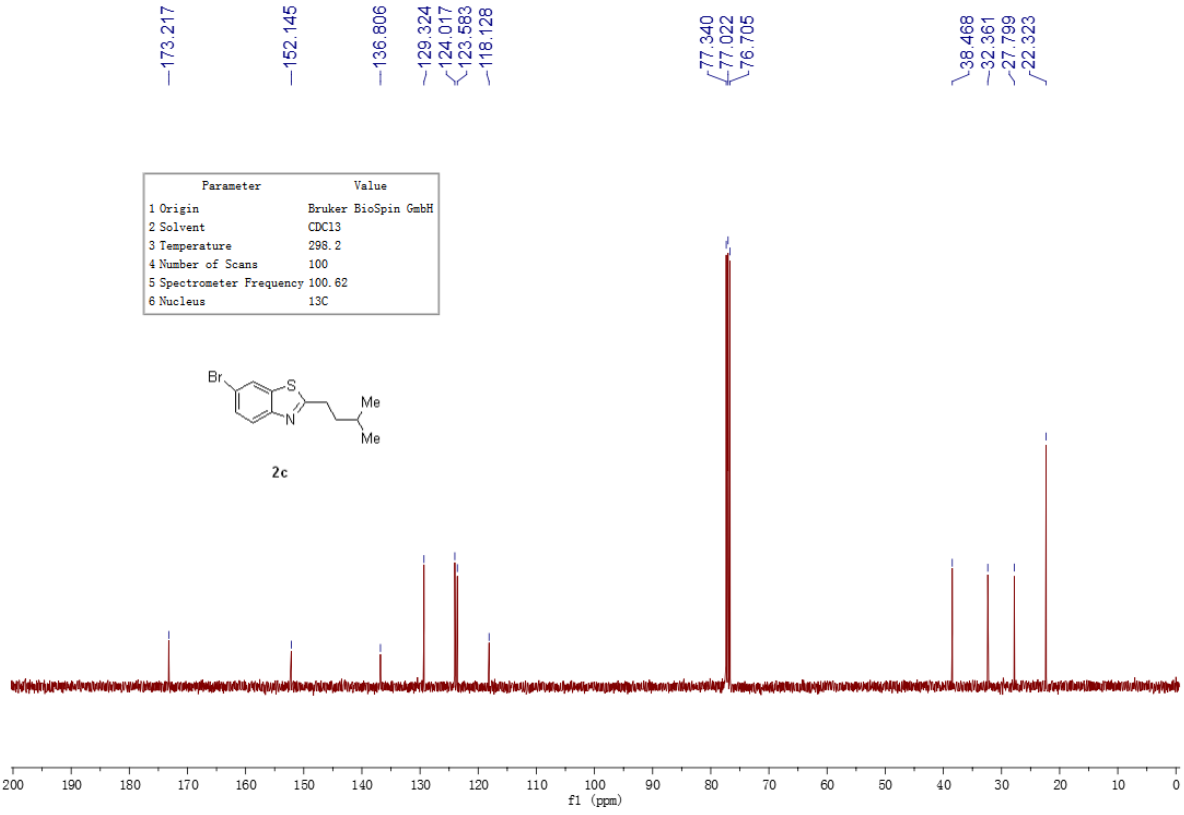

7.973
7.952
7.834
7.814
7.456
7.454
7.436
7.418
7.415
7.350
7.348
7.330
7.312
7.310
7.260
3.135
3.116
3.095
1.805
1.788
1.783
1.773
1.769
1.765
1.749
1.719
0.985
0.969
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
S
N
Me
Me
2a
1.00
0.99
1.02
1.01
2.00
3.00
6.01
f1 (ppm)

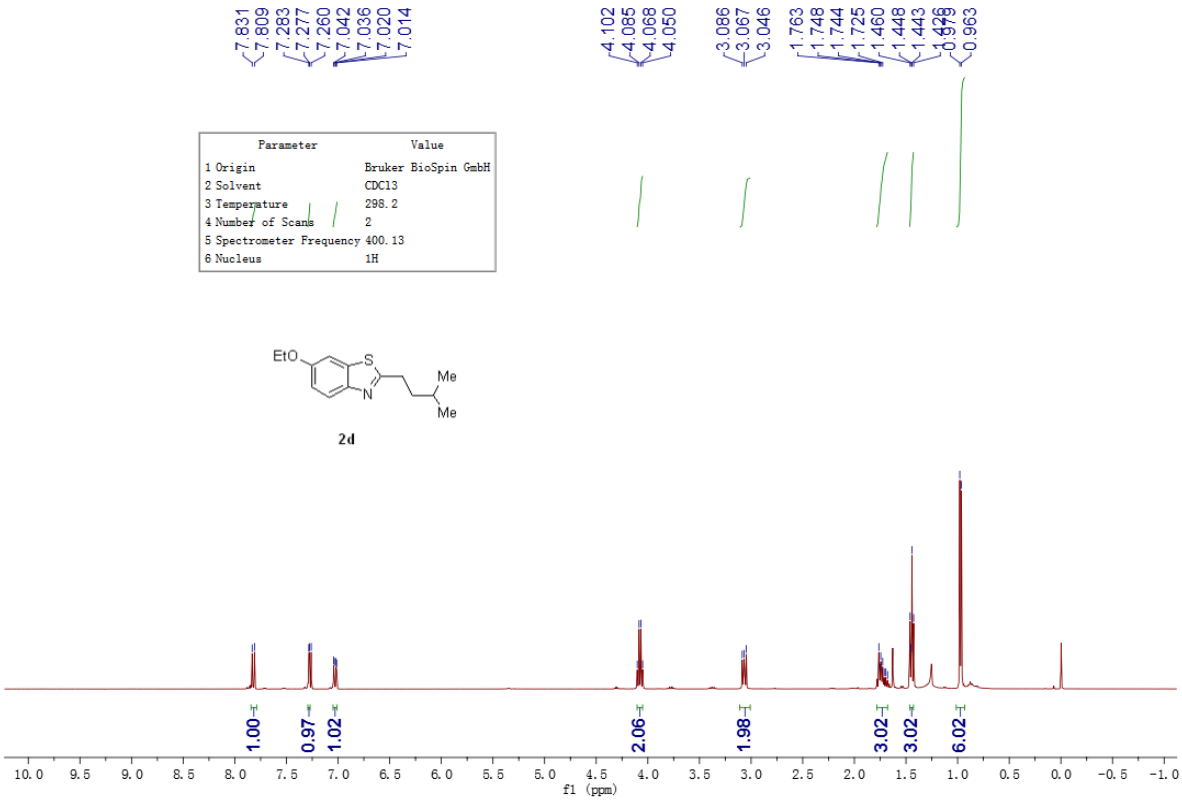

172.608
153.263
135.120
125.858
124.604
122.480
121.471
77.381
77.064
76.746
38.632
32.398
27.807
22.355
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 100
5 Spectrometer Frequency 100.61
6 Nucleus 13C
S
N
Me
Me
2a
f1 (ppm)

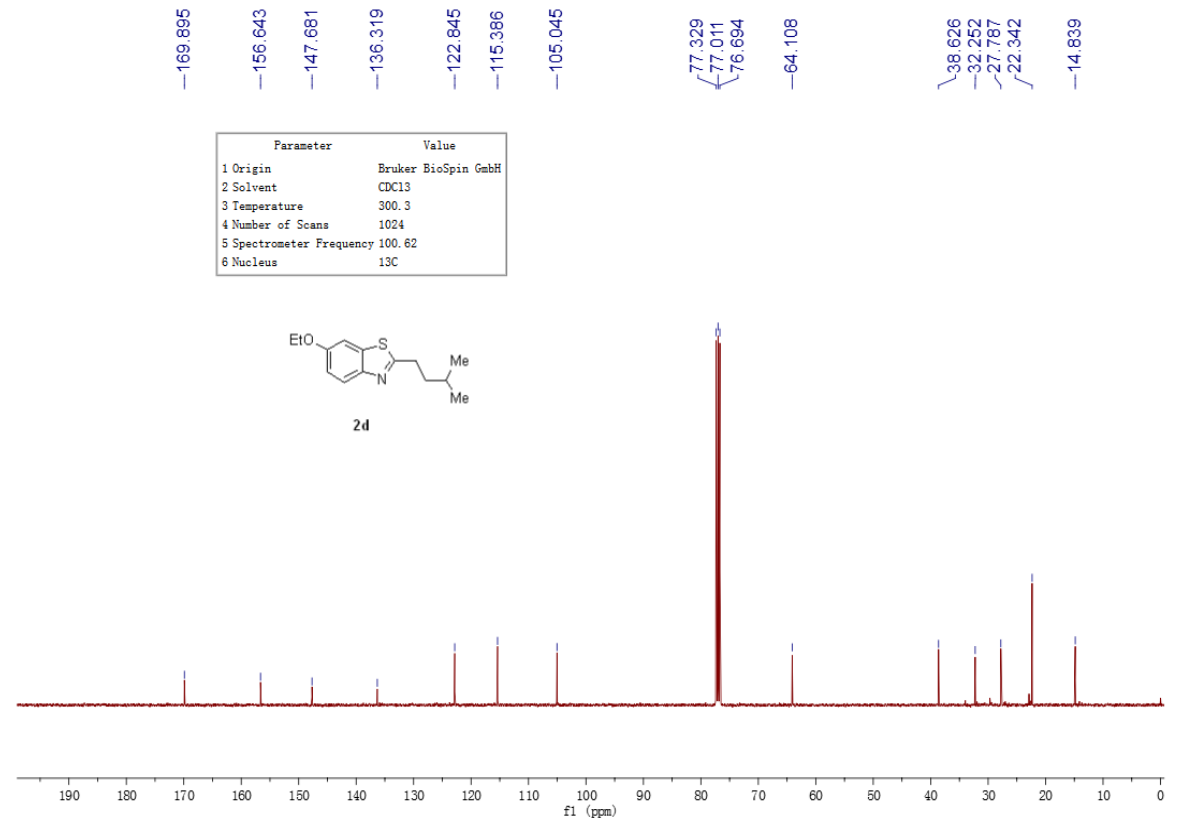

7.862
7.840
7.799
7.794
7.406
7.401
7.384
7.379
7.260
3.114
3.095
3.075
1.774
1.754
0.981
0.964
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Cl
S
N
Me
Me
2b
0.94
0.93
0.94
2.00
3.03
6.00
f1 (ppm)

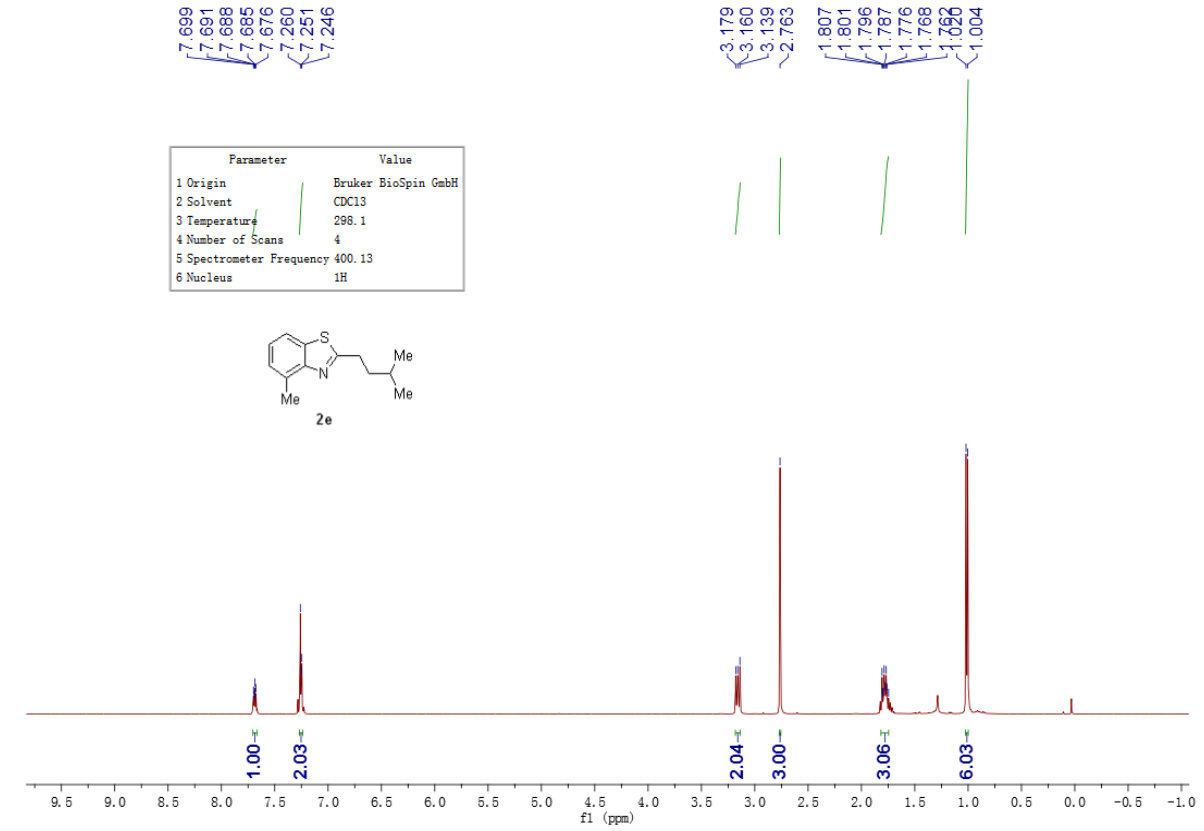

173.147
151.808
136.309
130.499
126.618
123.188
121.086
77.345
77.027
76.710
38.475
32.369
27.793
22.321
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 100
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Cl
S
N
Me
Me
2b
f1 (ppm)

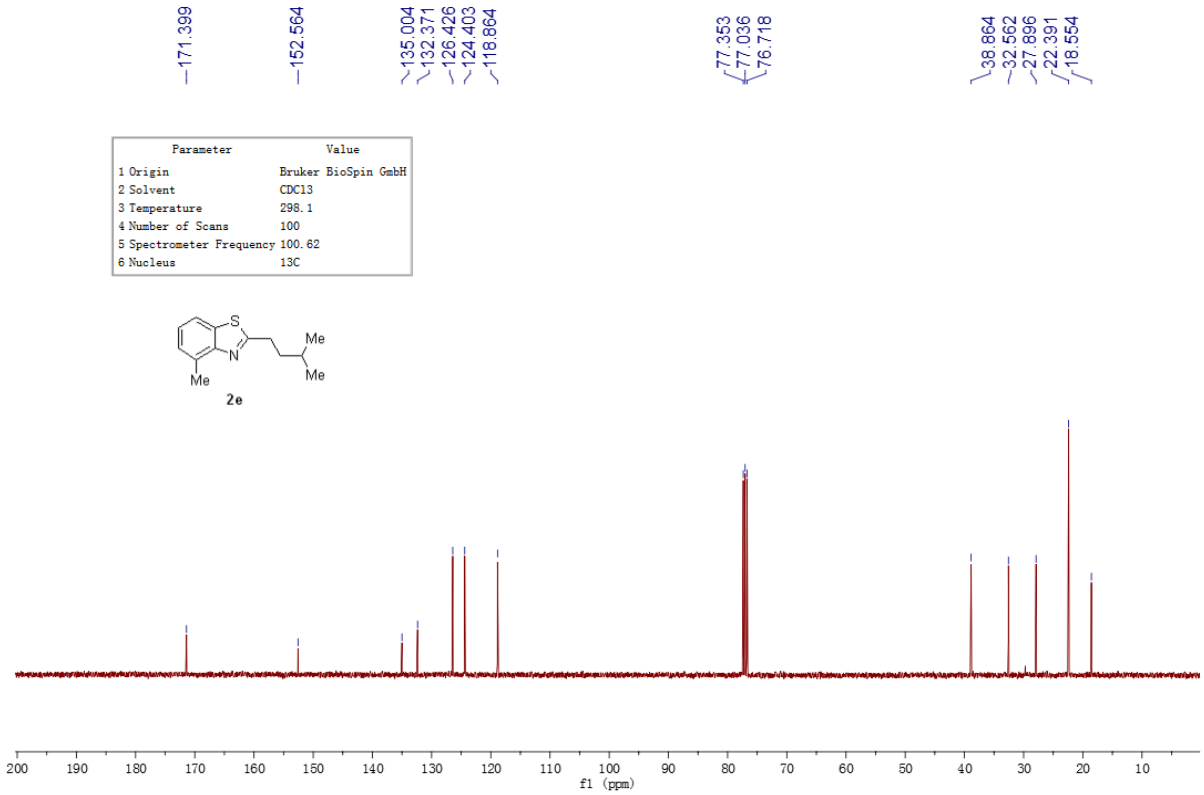

7.964
7.959
7.812
7.790
7.550
7.545
7.528
7.523
7.260
3.115
3.099
3.096
3.075
1.777
1.757
1.754
1.738
0.983
0.967
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Br
S
N
Me
Me
2c
1.00
1.03
1.02
2.00
3.02
6.05
f1 (ppm)

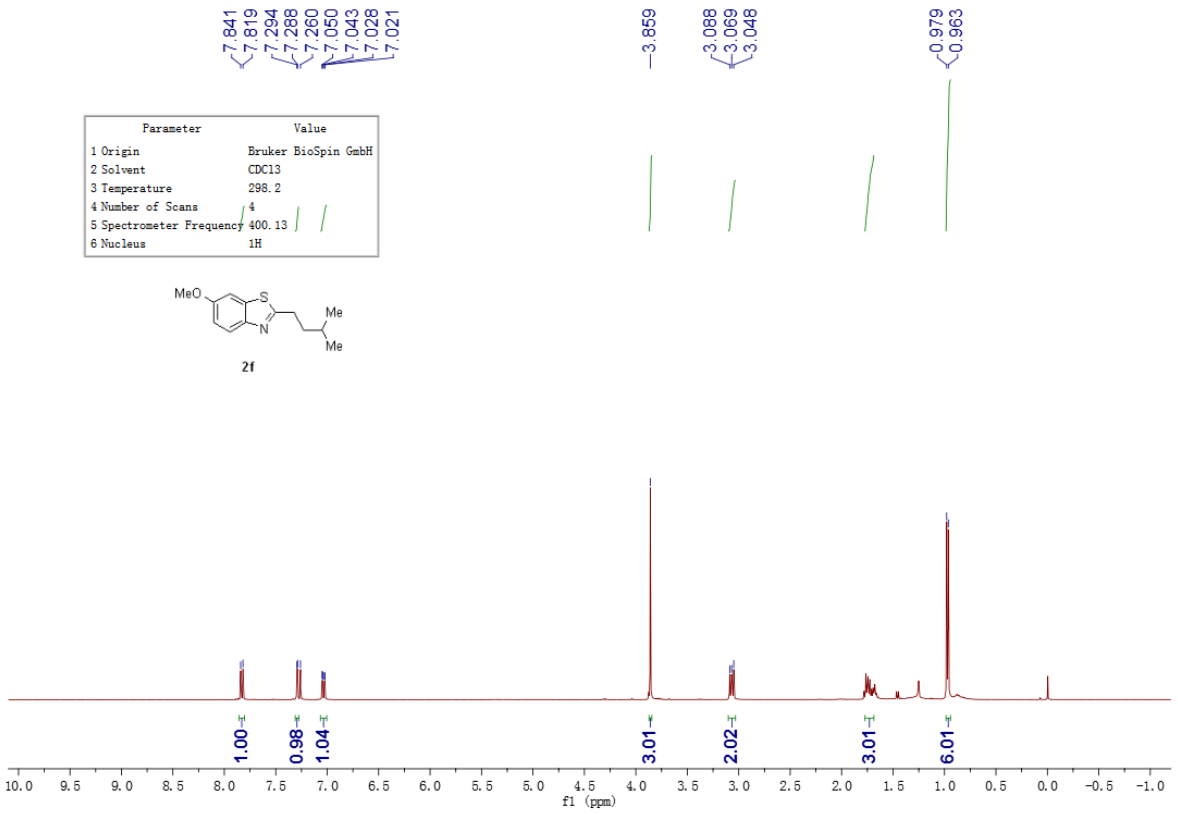

173.217
152.145
136.806
129.324
124.017
123.583
118.128
77.340
77.022
76.705
38.468
32.361
27.799
22.323
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 100
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Br
S
N
Me
Me
2c
f1 (ppm)

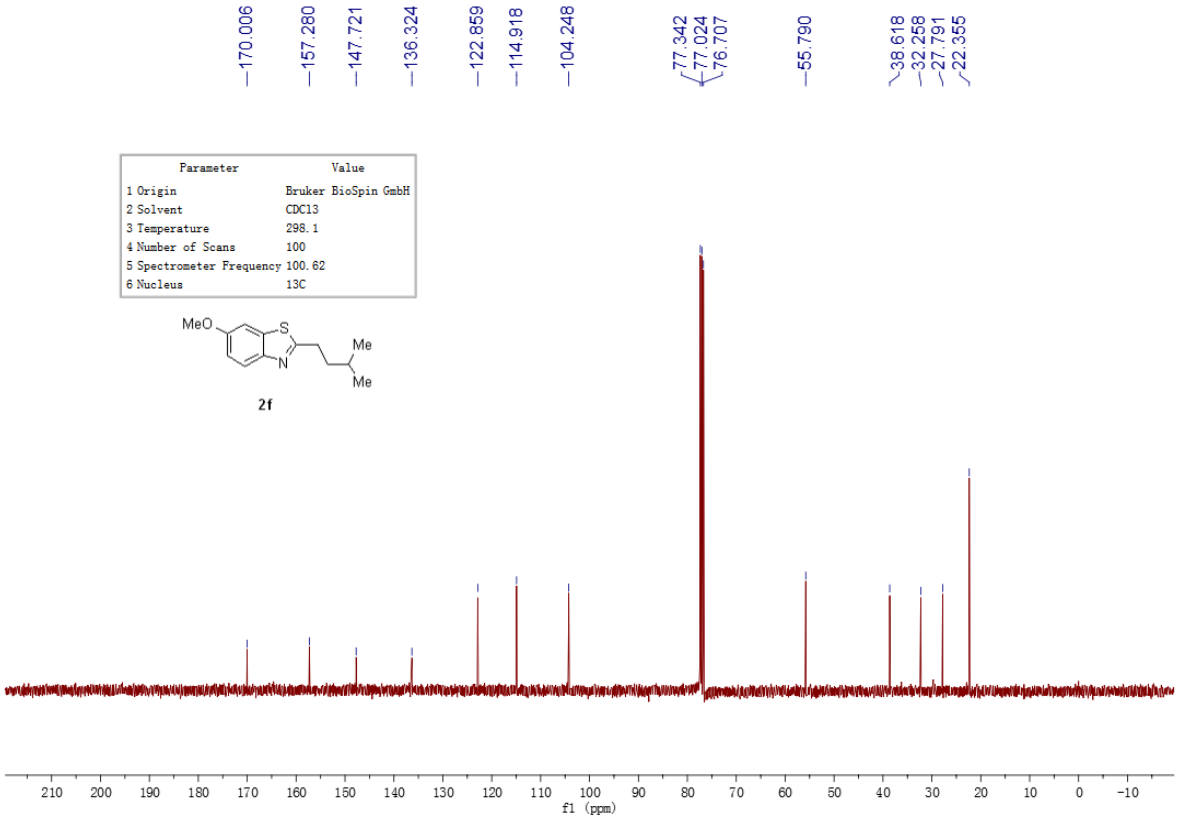

7.831
7.809
7.283
7.277
7.260
7.042
7.036
7.020
7.014
4.102
4.085
4.068
4.050
3.086
3.067
3.046
1.763
1.748
1.744
1.725
1.460
1.448
1.443
0.963
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
EtO
S
N
Me
Me
2d
1.00
0.97
1.02
2.06
1.98
3.02
3.02
6.02
f1 (ppm)

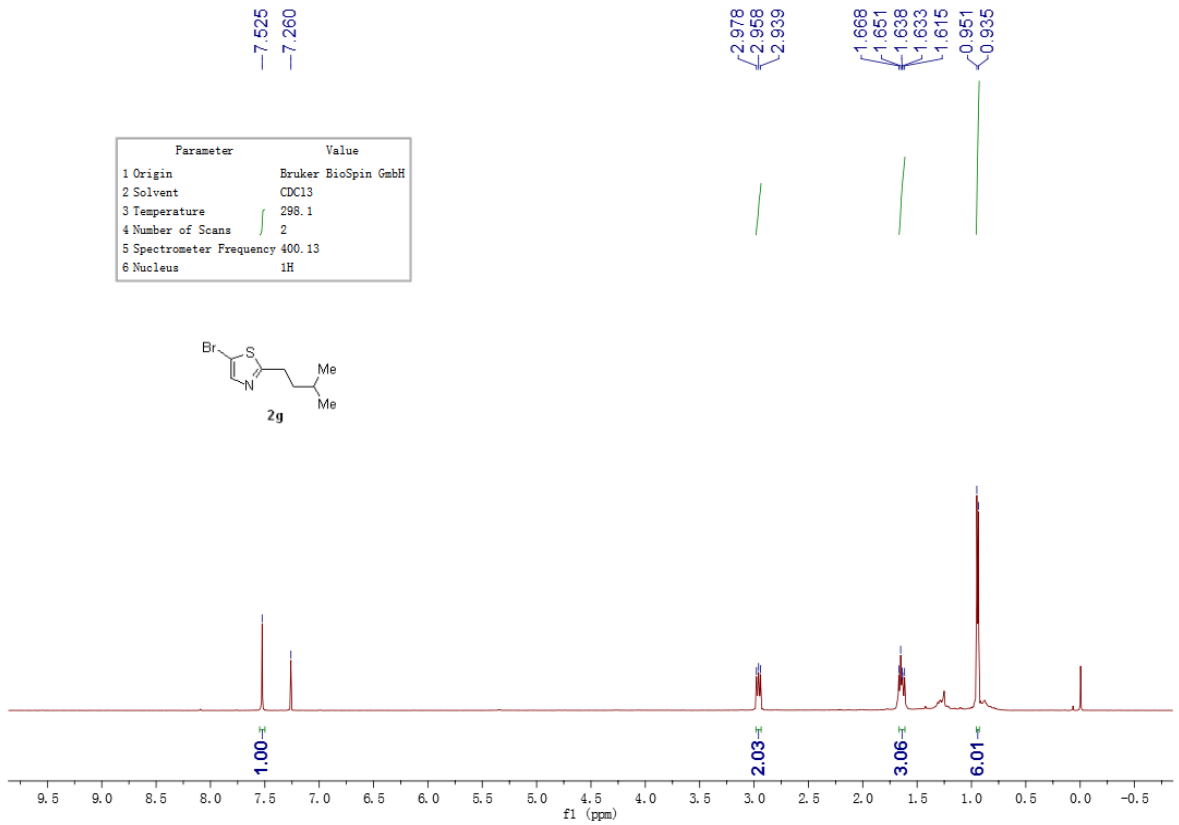

169.895
156.643
147.681
136.319
122.845
115.386
105.045
77.329
77.011
76.694
64.108
38.626
32.252
27.787
22.342
14.839
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.3
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
EtO
S
N
Me
Me
2d
f1 (ppm)

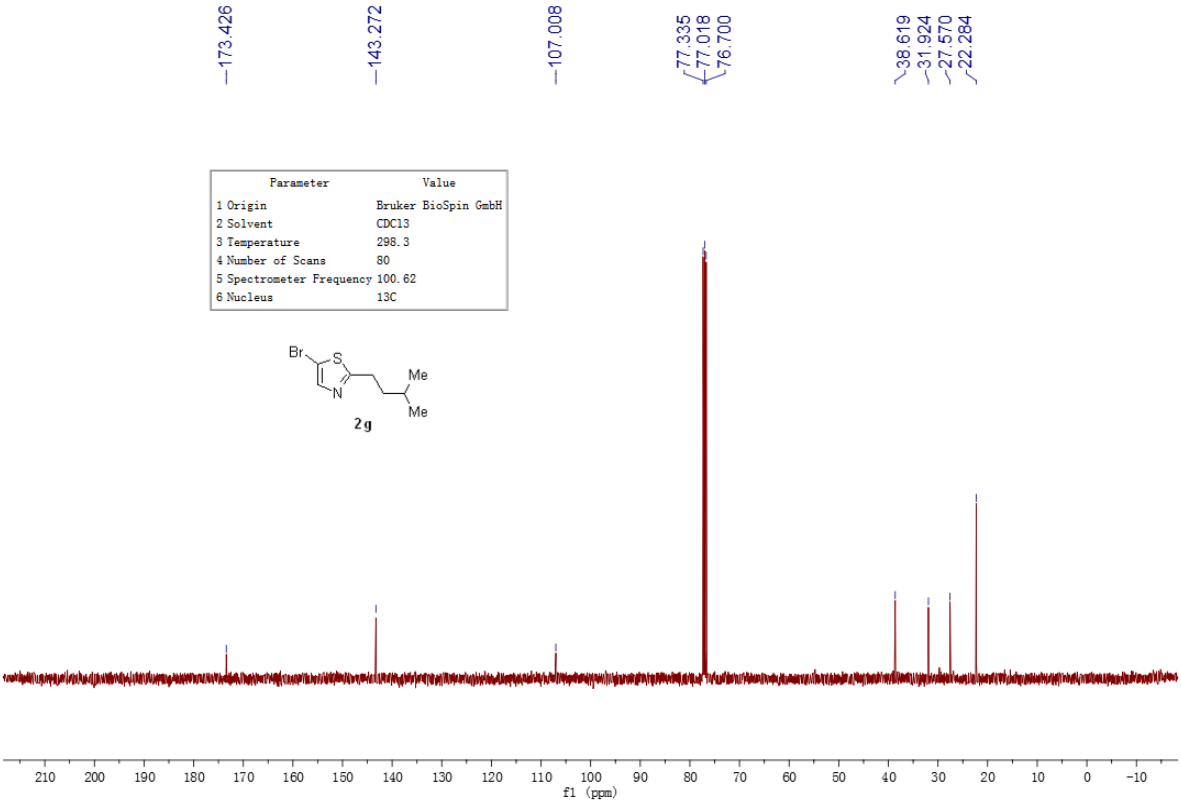

7.699
7.691
7.688
7.685
7.676
7.260
7.251
7.246
3.179
3.160
3.139
2.763
1.807
1.801
1.796
1.787
1.776
1.768
1.020
1.004
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
S
N
Me
Me
Me
2e
1.00
2.03
2.04
3.00
3.06
6.03
f1 (ppm)

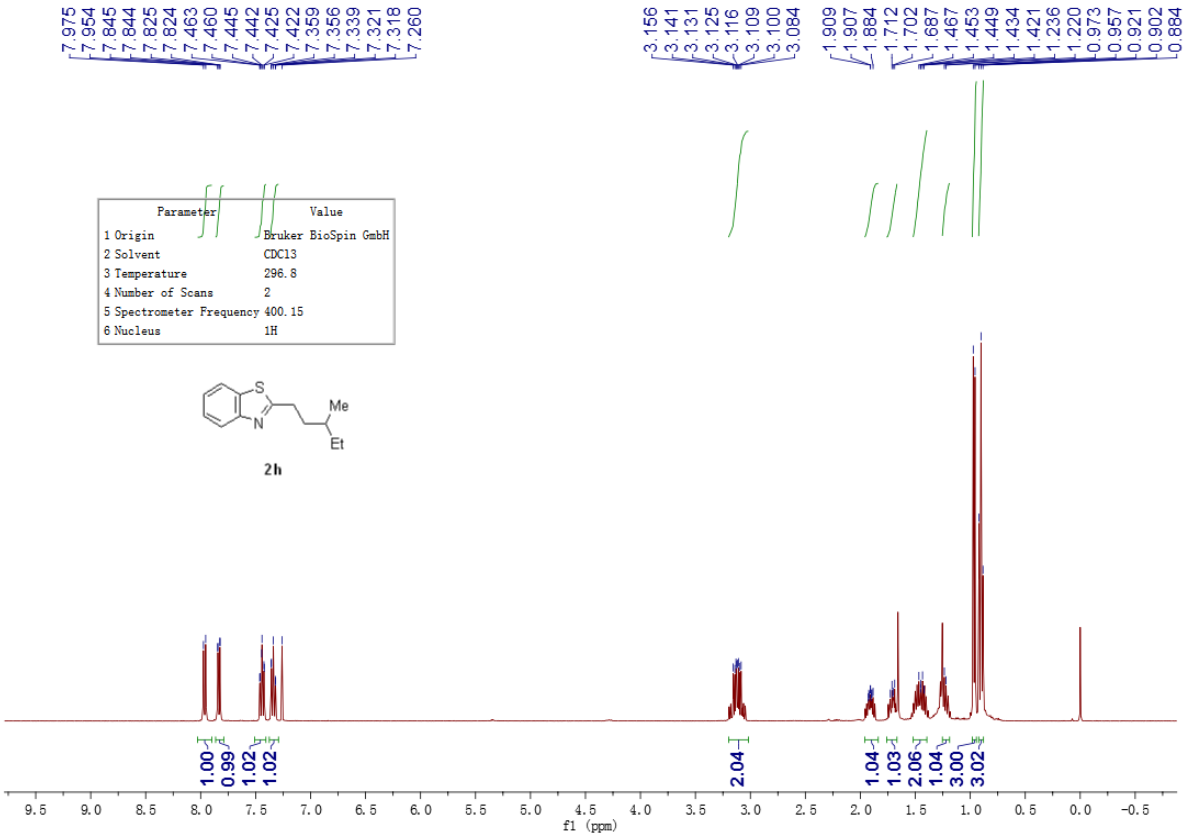

171.399
152.564
135.004
132.371
126.426
124.403
118.864
77.353
77.036
76.718
38.864
32.562
27.896
22.391
18.554
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 100
5 Spectrometer Frequency 100.62
6 Nucleus 13C
S
N
Me
Me
Me
2e
f1 (ppm)

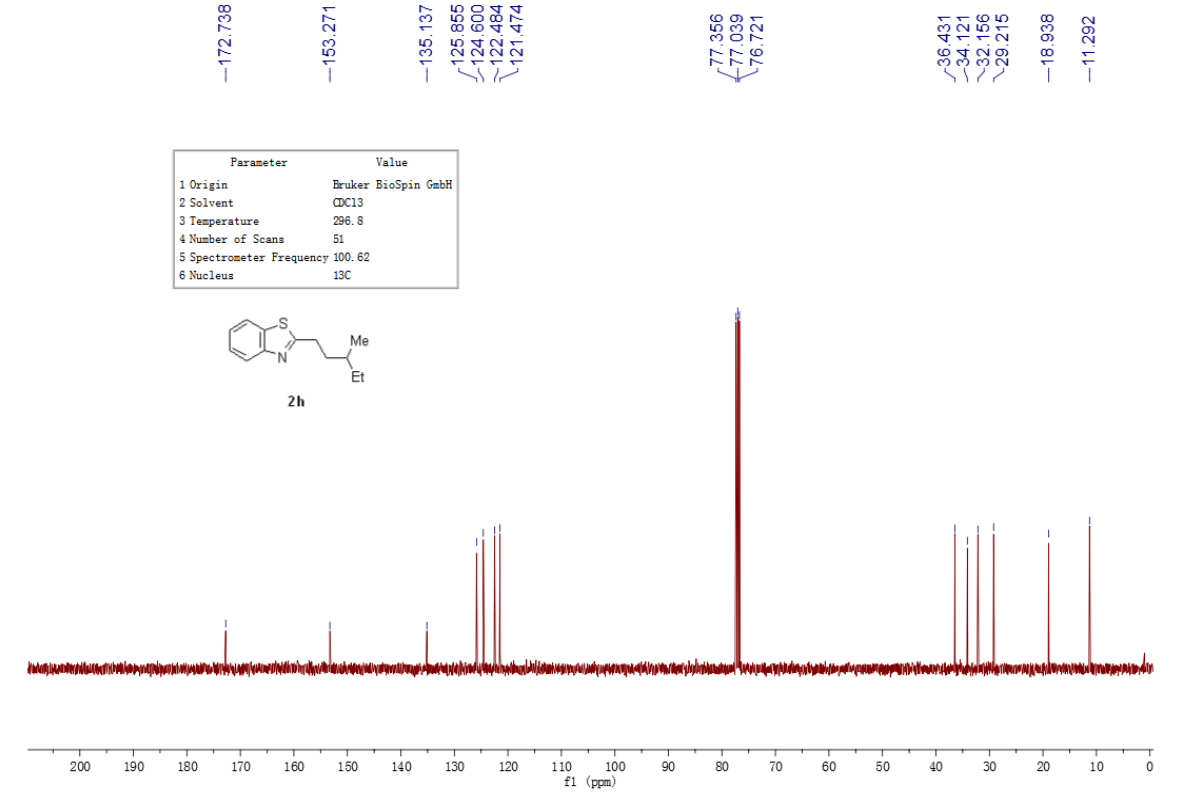

7.841
7.819
7.294
7.288
7.260
7.050
7.043
7.028
7.021
3.859
3.088
3.069
3.048
0.979
0.963
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
MeO
S
N
Me
Me
2f
1.00
0.98
1.04
3.01
2.02
3.01
6.01
f1 (ppm)

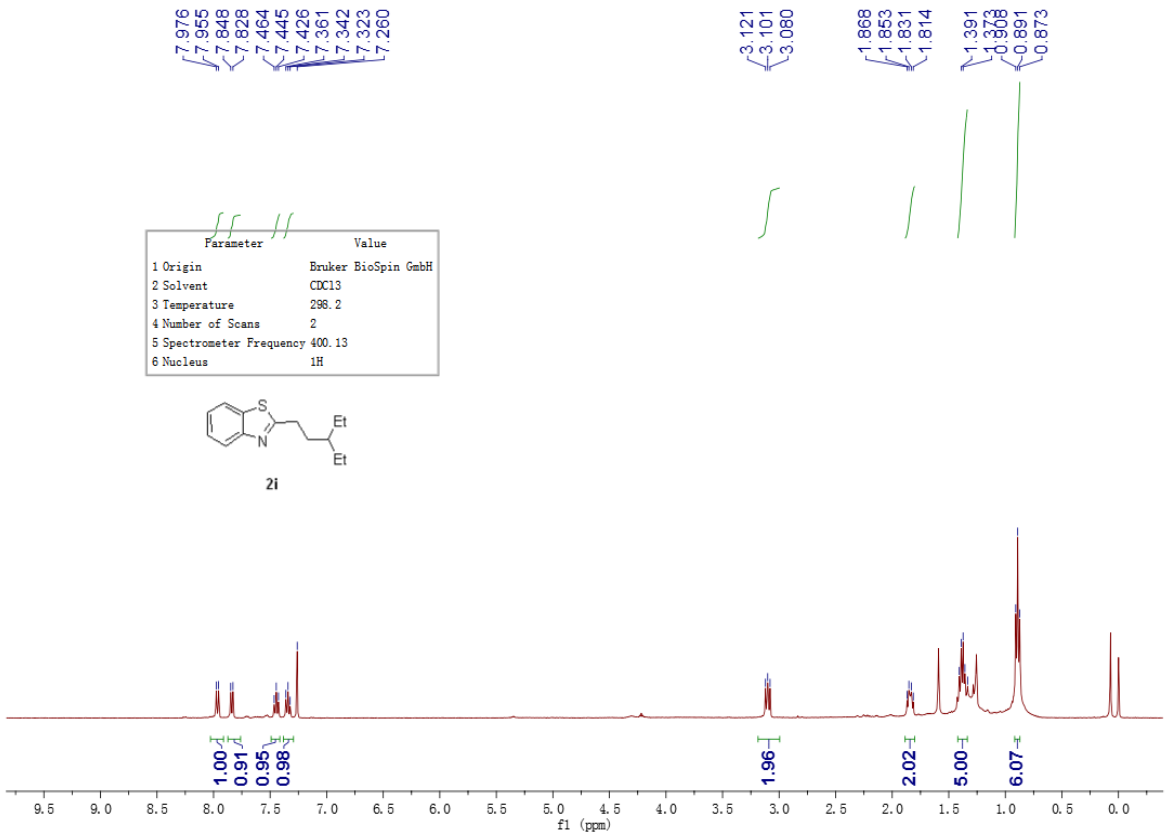

170.006
157.280
147.721
136.324
122.859
114.918
104.248
77.342
77.024
76.707
55.790
38.618
32.258
27.791
22.355
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 100
5 Spectrometer Frequency 100.62
6 Nucleus 13C
MeO
S
N
Me
Me
2f
f1 (ppm)

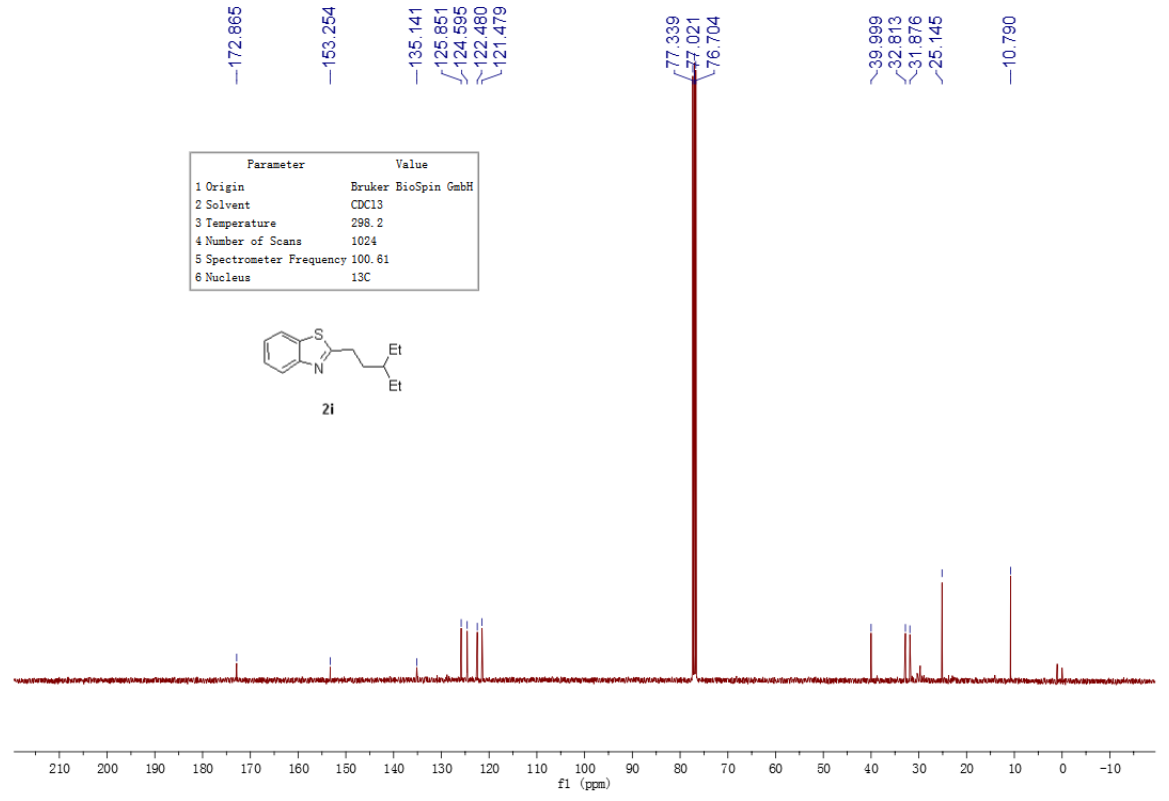

7.525
7.260
2.978
2.958
2.939
1.668
1.651
1.638
1.633
1.615
0.951
0.935
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Br
S
N
Me
Me
2g
1.00
2.03
3.06
6.01
f1 (ppm)

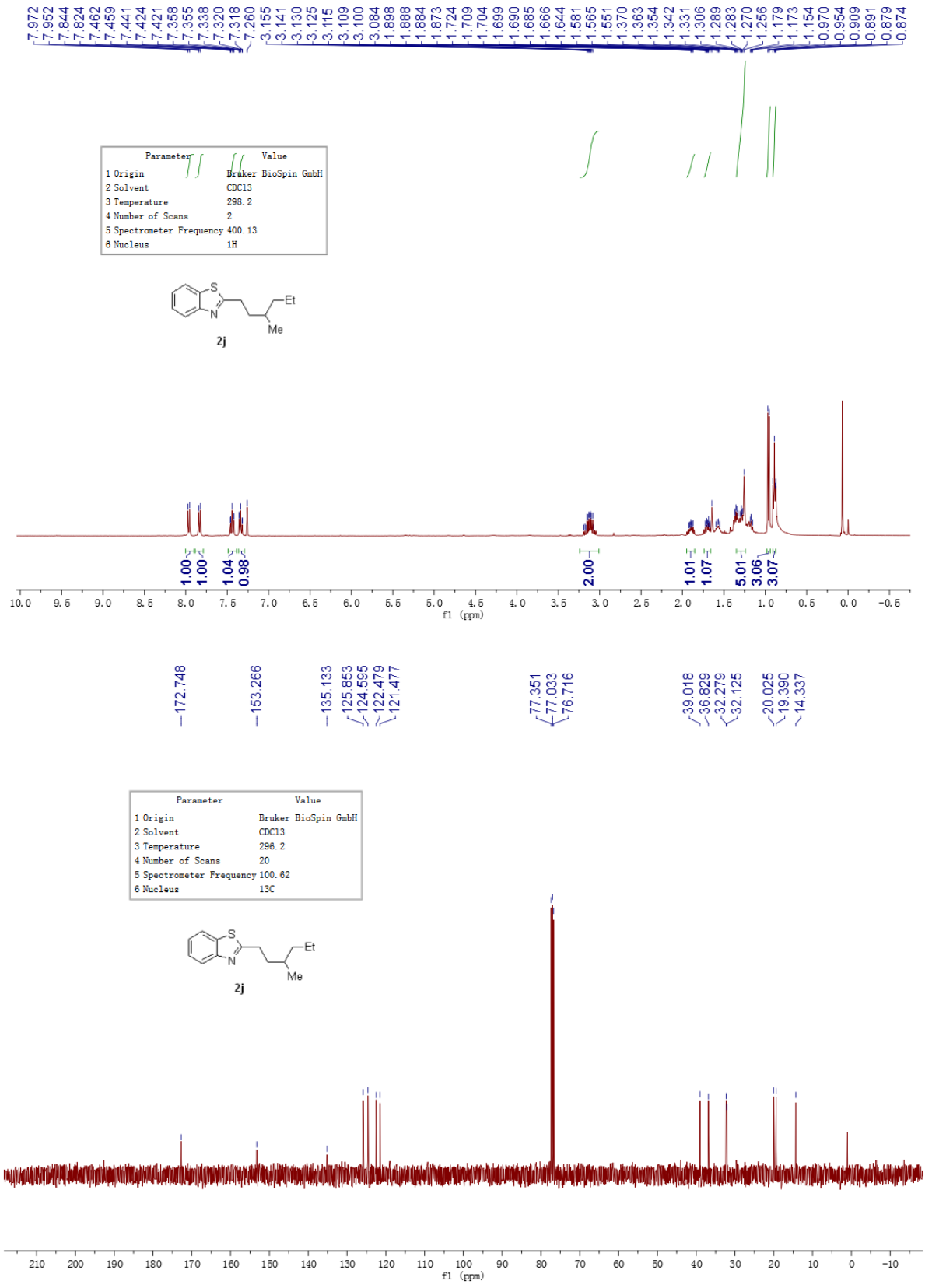

173.426
143.272
107.008
77.335
77.018
76.700
38.619
31.924
27.570
22.284
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.3
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Br
S
N
Me
Me
2g
f1 (ppm)

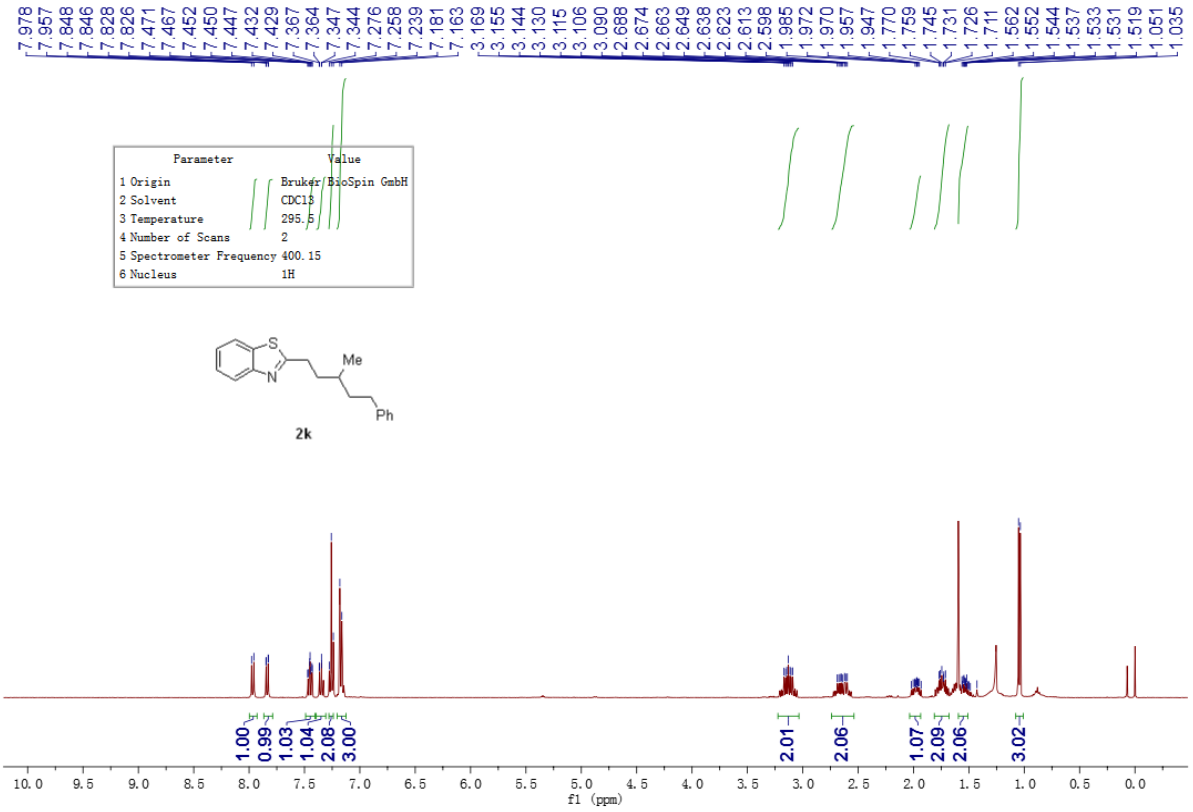

7.975
7.954
7.845
7.844
7.825
7.824
7.463
7.460
7.445
7.442
7.425
7.422
7.359
7.356
7.339
7.321
7.318
7.260
3.156
3.141
3.131
3.125
3.116
3.109
3.100
3.084
1.909
1.907
1.884
1.712
1.702
1.687
1.467
1.453
1.449
1.434
1.421
1.236
1.220
0.973
0.957
0.921
0.902
0.884
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.8
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
S
N
Me
Et
2h
1.00
0.99
1.02
1.02
2.04
1.04
1.03
2.06
1.04
3.00
3.02
f1 (ppm)

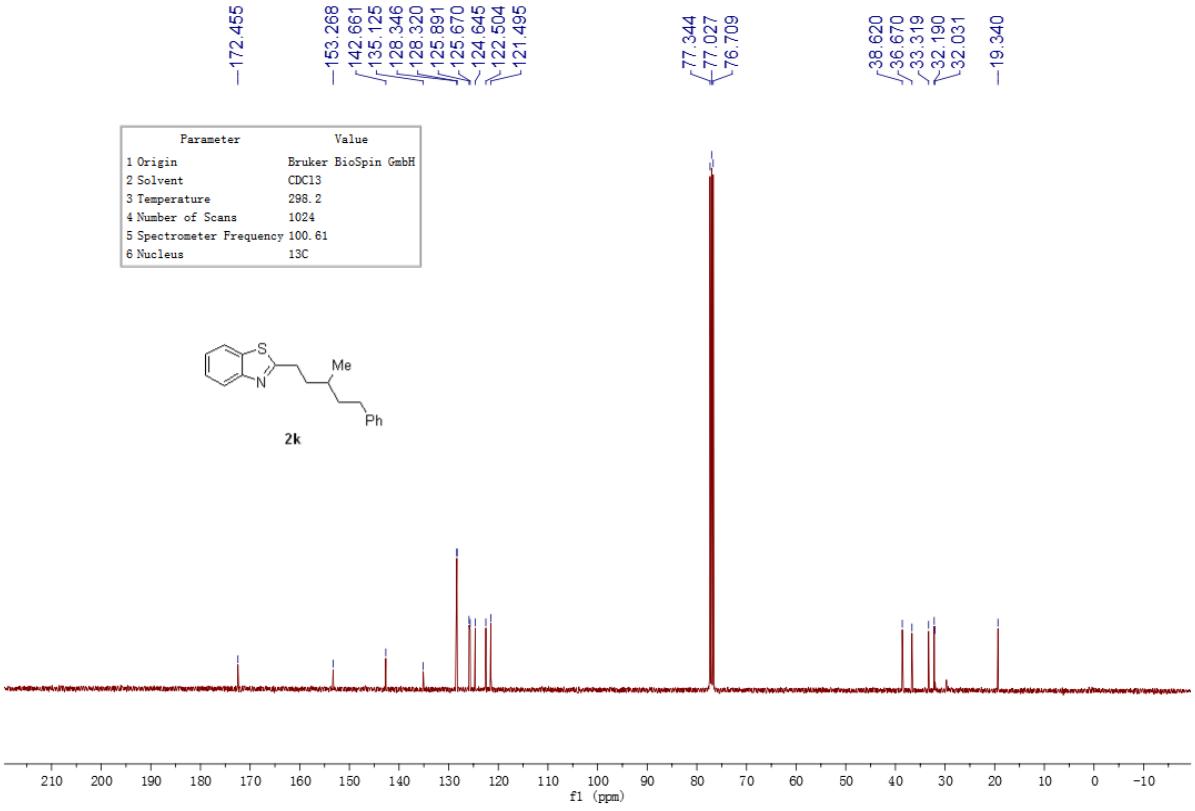

172.738
153.271
135.137
125.855
124.600
122.484
121.474
77.356
77.039
76.721
36.431
34.121
32.156
29.215
18.938
11.292
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.8
4 Number of Scans 51
5 Spectrometer Frequency 100.62
6 Nucleus 13C
S
N
Me
Et
2h
f1 (ppm)

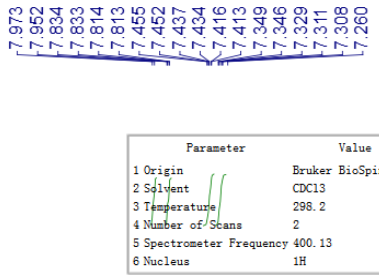

7.976
7.955
7.848
7.828
7.464
7.445
7.426
7.361
7.342
7.323
7.260
3.121
3.101
3.080
1.868
1.853
1.831
1.814
1.391
0.908
0.891
0.873
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
S
N
Et
Et
2i
1.00
0.91
0.95
0.98
1.96
2.02
5.00
6.07
f1 (ppm)


172.865
153.254
135.141
125.851
124.595
122.480
121.479
77.339
77.021
76.704
39.999
32.813
31.876
25.145
10.790
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
S
N
Et
Et
2i
f1 (ppm)

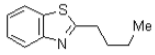

7.972
7.952
7.844
7.824
7.462
7.459
7.441
7.424
7.421
7.358
7.355
7.338
7.320
7.318
7.260
3.155
3.141
3.130
3.125
3.115
3.109
3.100
3.084
1.898
1.888
1.884
1.873
1.724
1.709
1.704
1.699
1.690
1.685
1.666
1.644
1.581
1.565
1.551
1.370
1.363
1.354
1.342
1.331
1.306
1.289
1.283
1.270
1.256
1.179
1.173
1.154
0.970
0.954
0.909
0.891
0.879
0.874
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
S
N
Et
Me
2j
1.00
1.00
1.04
0.98
2.00
1.01
1.07
5.01
3.06
3.07
f1 (ppm)
172.748
153.266
135.133
125.853
124.595
122.479
121.477
77.351
77.033
76.716
39.018
36.829
32.279
32.125
20.025
19.390
14.337
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.2
4 Number of Scans 20
5 Spectrometer Frequency 100.62
6 Nucleus 13C
S
N
Et
Me
2j
f1 (ppm)


7.978
7.957
7.848
7.846
7.828
7.826
7.471
7.467
7.452
7.450
7.447
7.432
7.429
7.367
7.364
7.347
7.344
7.276
7.258
7.239
7.181
7.163
3.169
3.155
3.144
3.130
3.115
3.106
3.090
2.688
2.674
2.663
2.649
2.638
2.623
2.613
2.598
1.985
1.972
1.970
1.957
1.947
1.770
1.759
1.745
1.731
1.726
1.711
1.562
1.552
1.544
1.537
1.533
1.531
1.519
1.051
1.035
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.5
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
S
N
Me
Ph
2k
1.00
0.99
1.03
1.04
2.08
3.00
2.01
2.06
1.07
2.09
2.06
3.02
10.0 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 0.5 0.0
f1 (ppm)

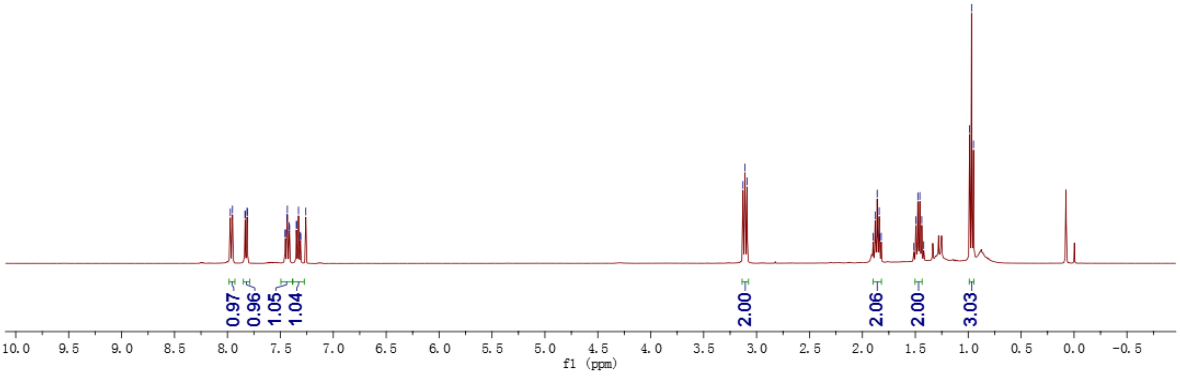

172.455
153.268
142.661
135.125
128.346
128.320
125.891
125.670
124.645
122.504
121.495
77.344
77.027
76.709
38.620
36.670
33.319
32.190
32.031
19.340
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
S
N
Me
Ph
2k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



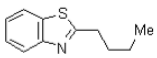
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H



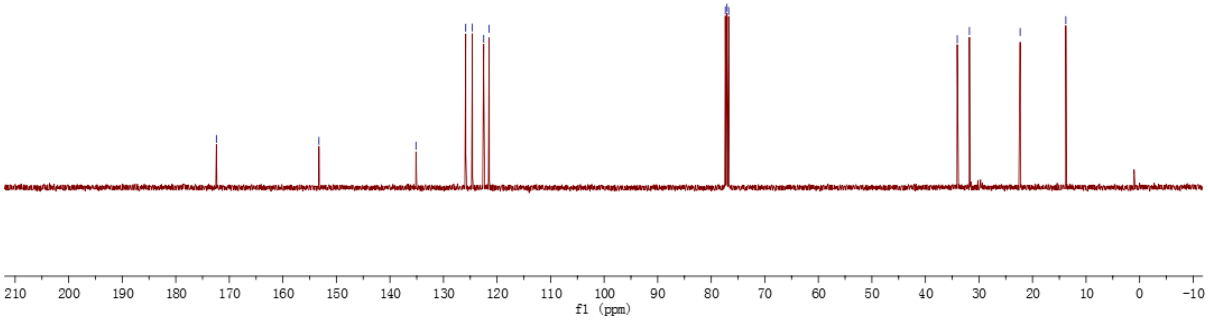
2l

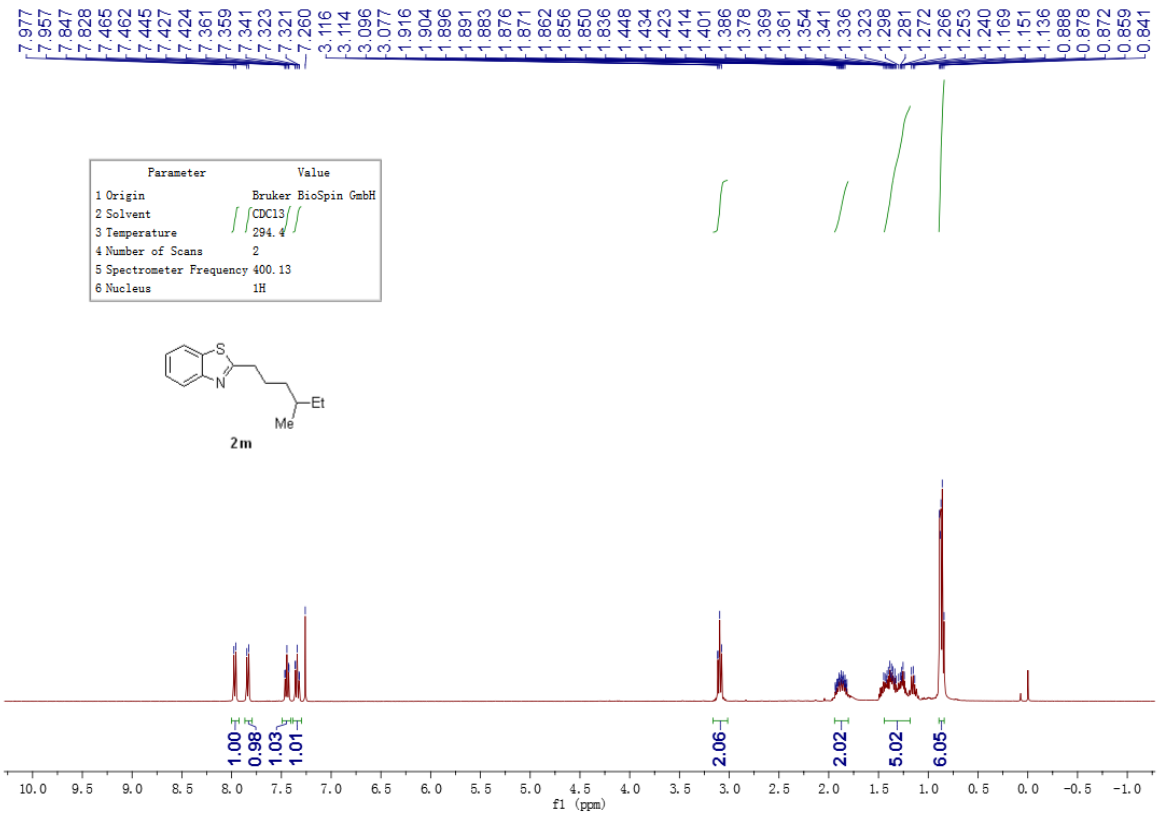


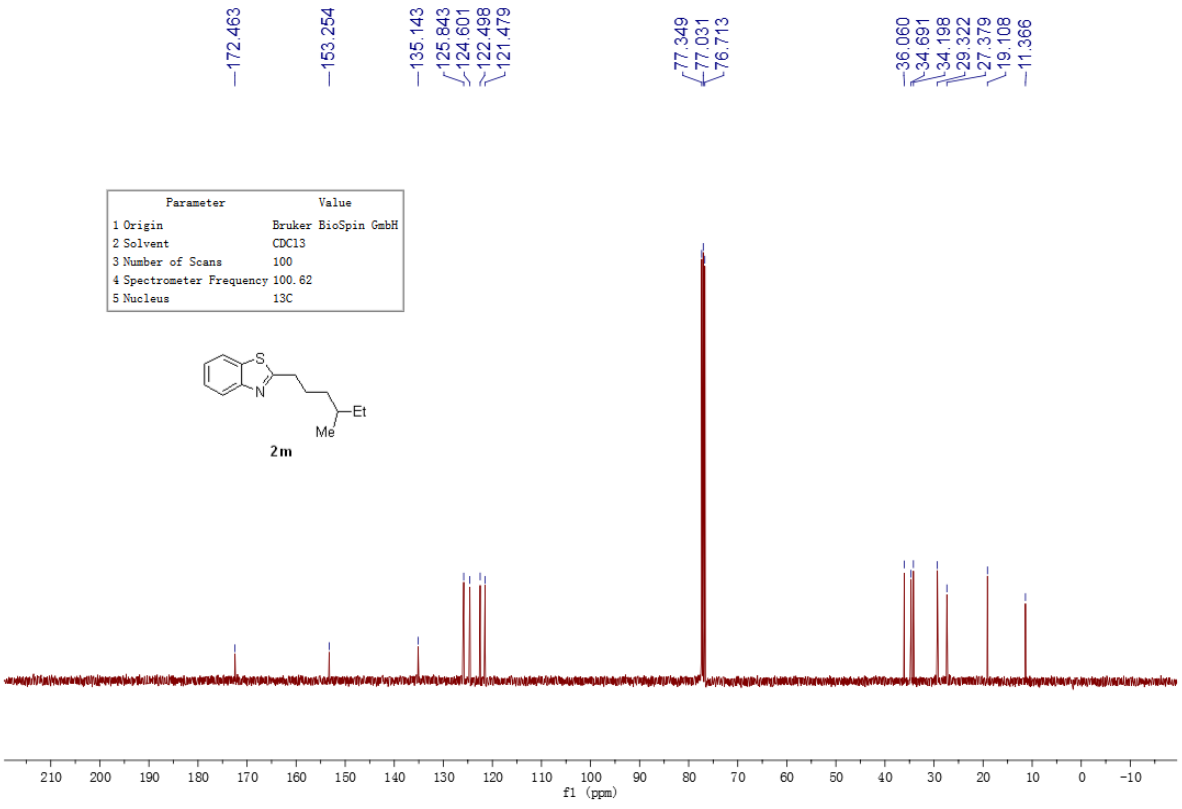
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	100
5 Spectrometer Frequency	100.61
6 Nucleus	13C

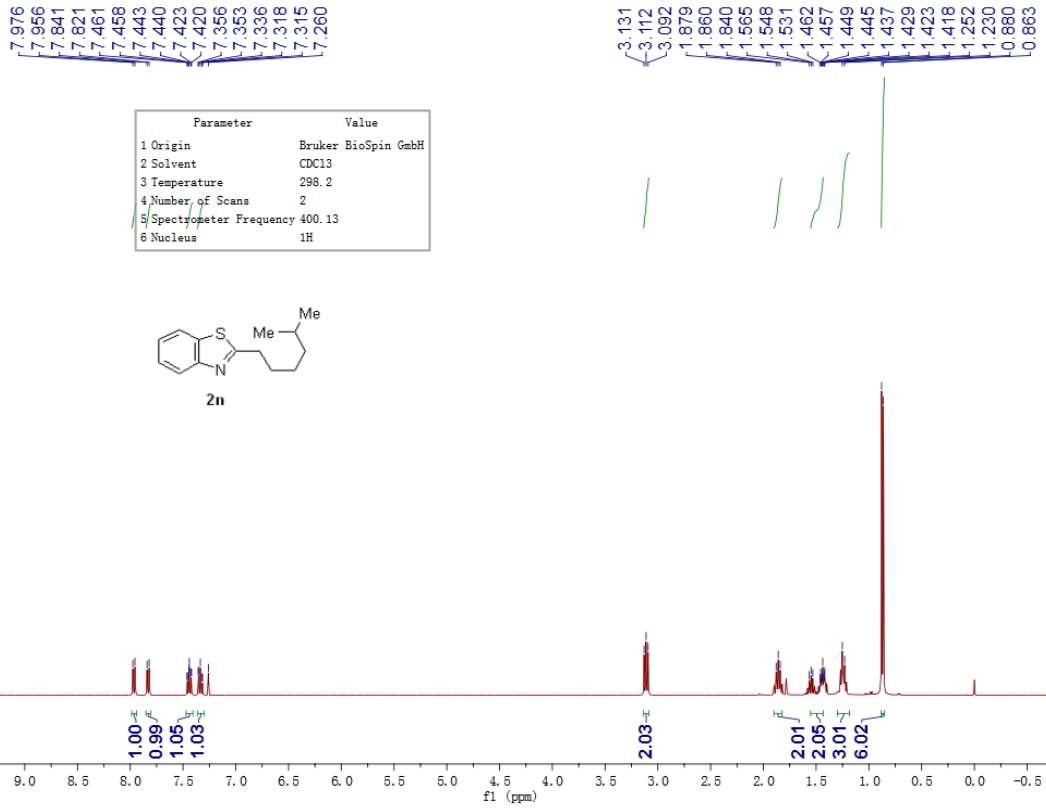


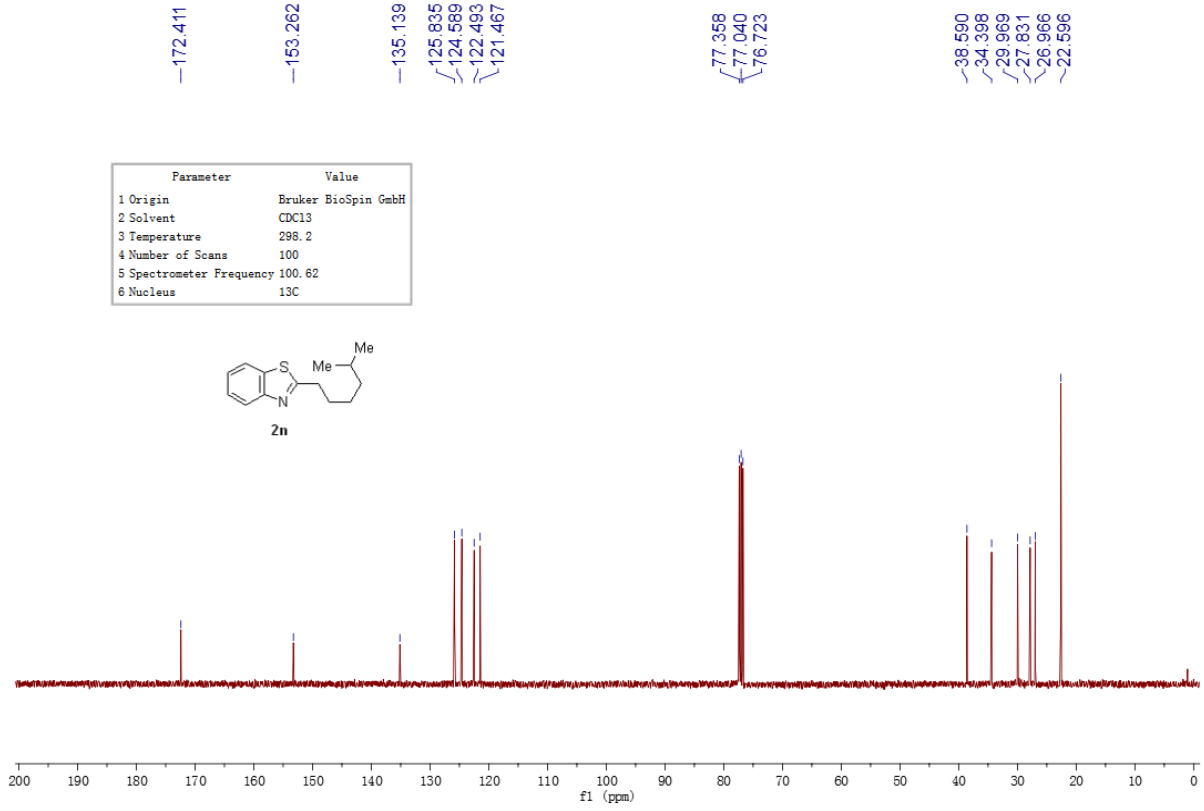
2l

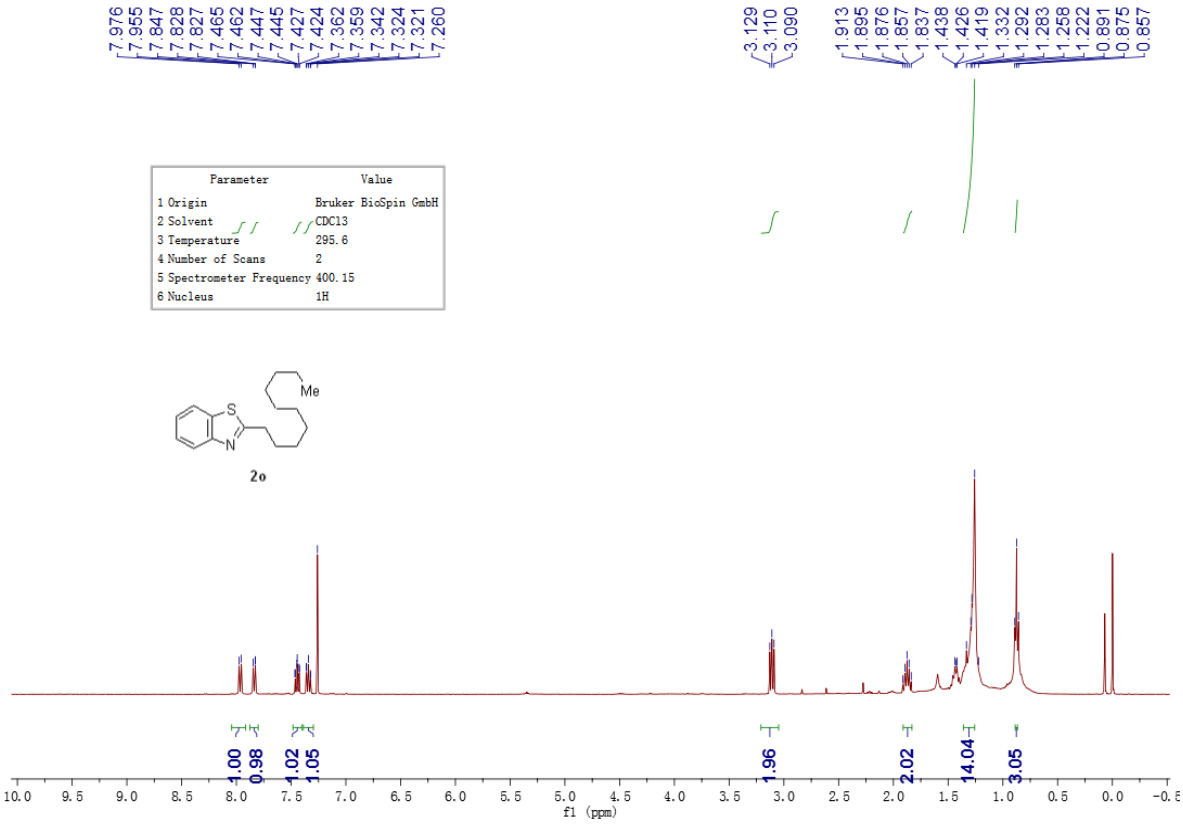


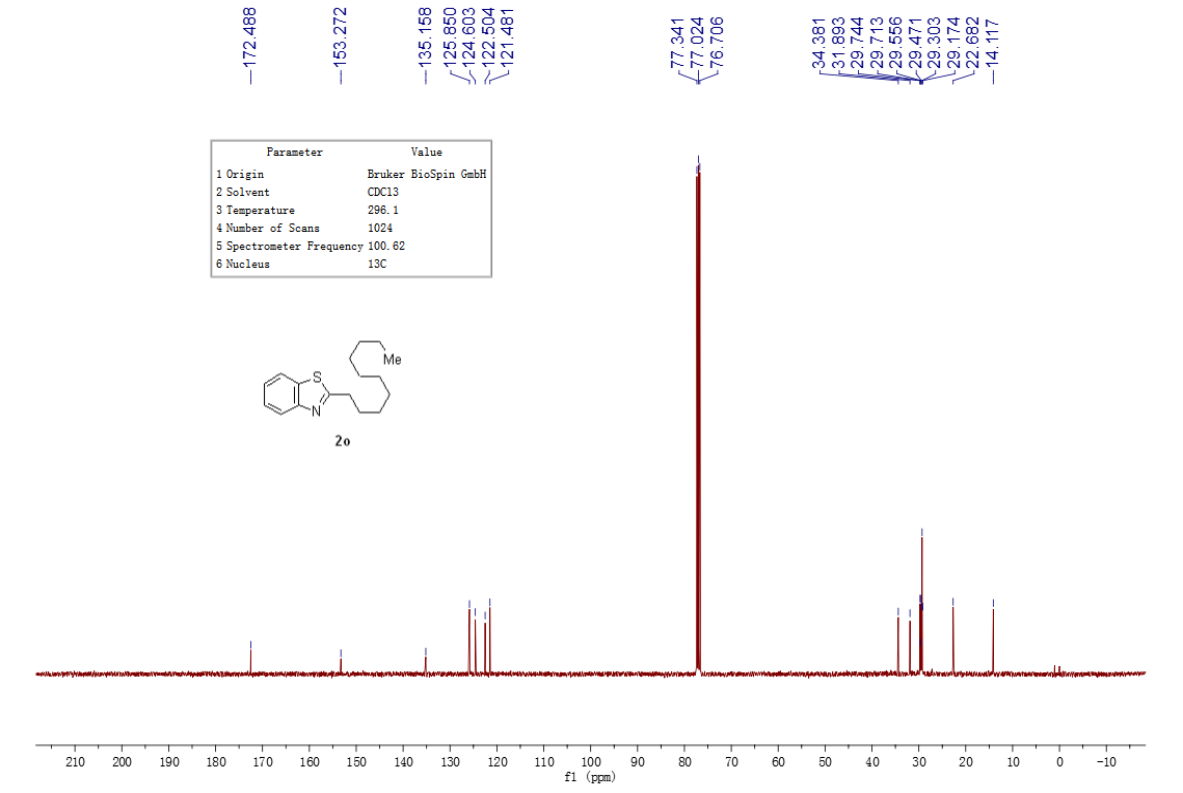

7.977
7.957
7.847
7.828
7.465
7.462
7.445
7.427
7.424
7.361
7.359
7.341
7.323
7.321
7.260
3.116
3.114
3.096
3.077
1.916
1.904
1.896
1.891
1.883
1.876
1.871
1.862
1.856
1.850
1.836
1.448
1.434
1.423
1.414
1.401
1.386
1.378
1.369
1.361
1.354
1.341
1.336
1.323
1.298
1.281
1.272
1.266
1.253
1.240
1.169
1.151
1.136
0.888
0.878
0.872
0.859
0.841
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 294.4
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
S
N
Et
Me
2m
1.00
0.98
1.03
1.01
2.06
2.02
5.02
6.05
f1 (ppm)

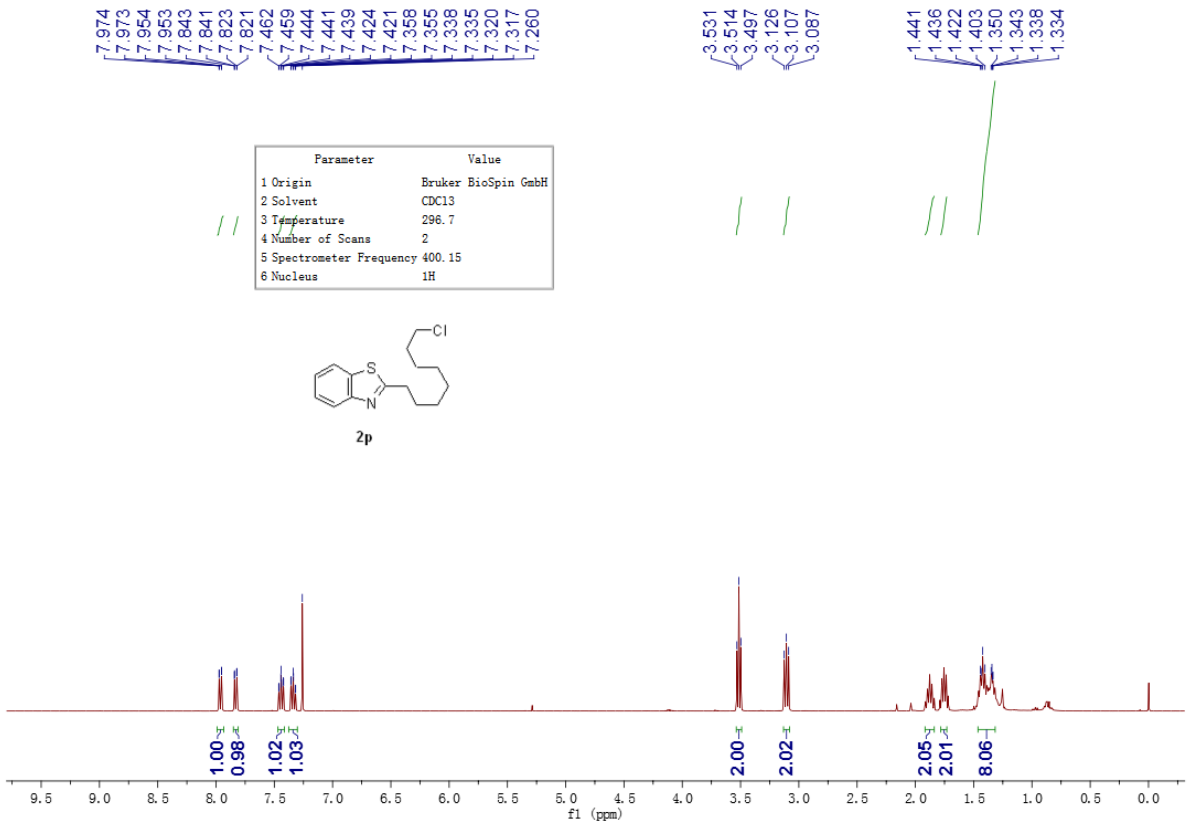

172.463
153.254
135.143
125.843
124.601
122.498
121.479
77.349
77.031
76.713
36.060
34.691
34.198
29.322
27.379
19.108
11.366
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Number of Scans 100
4 Spectrometer Frequency 100.62
5 Nucleus 13C
S
N
Et
Me
2m
f1 (ppm)

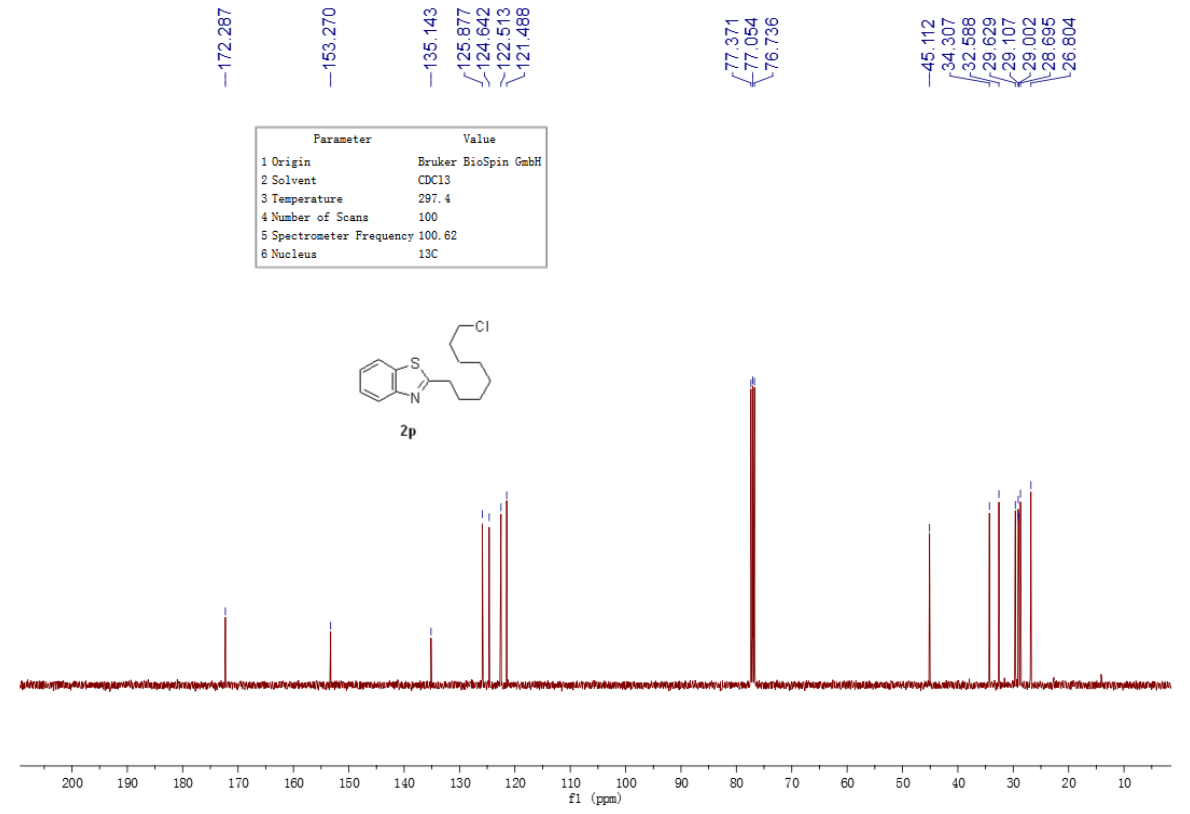

7.976
7.956
7.841
7.821
7.461
7.458
7.443
7.440
7.423
7.420
7.356
7.353
7.336
7.318
7.315
7.260
3.131
3.112
3.092
1.879
1.860
1.840
1.565
1.548
1.531
1.462
1.457
1.449
1.445
1.437
1.429
1.423
1.418
1.252
1.230
0.880
0.863
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Me
Me
S
N
2n
1.00
0.99
1.05
1.03
2.03
2.01
2.05
3.01
6.02
f1 (ppm)

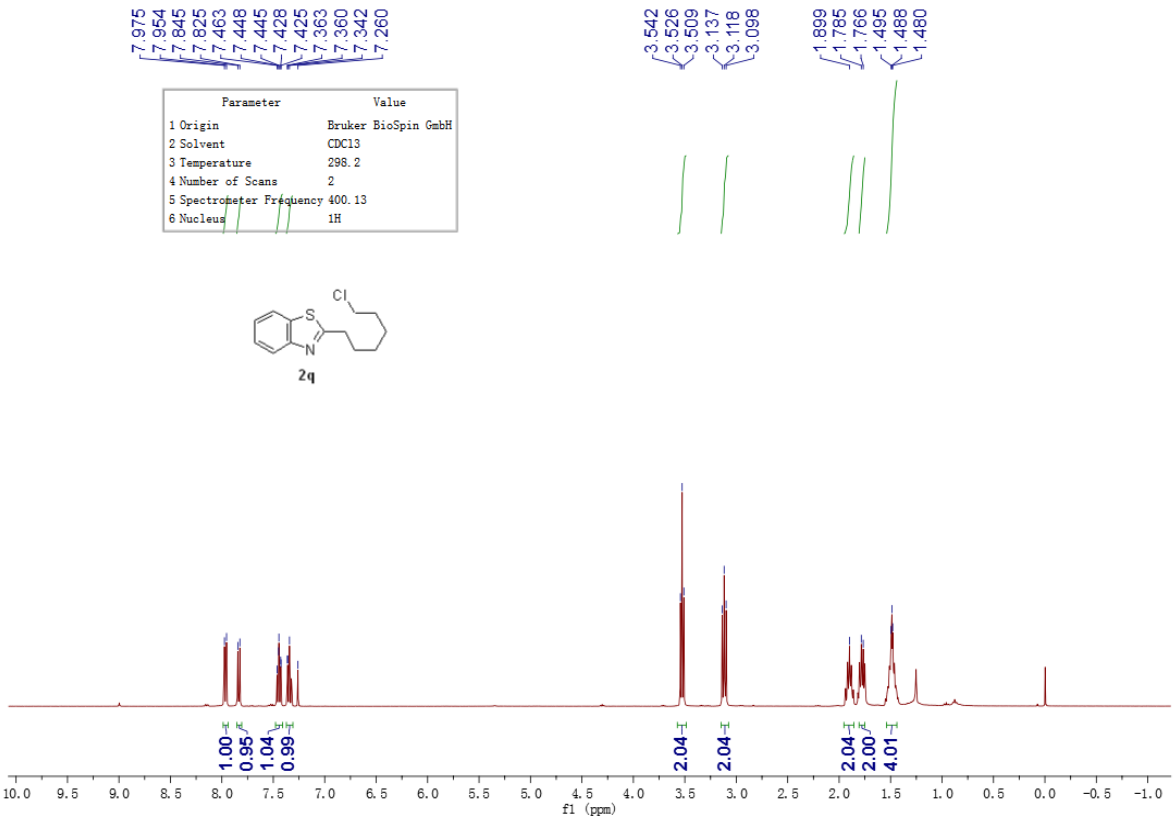

172.411
153.262
135.139
125.835
124.589
122.493
121.467
77.358
77.040
76.723
38.590
34.398
29.969
27.831
26.966
22.596
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 100
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Me
Me
S
N
2n
f1 (ppm)

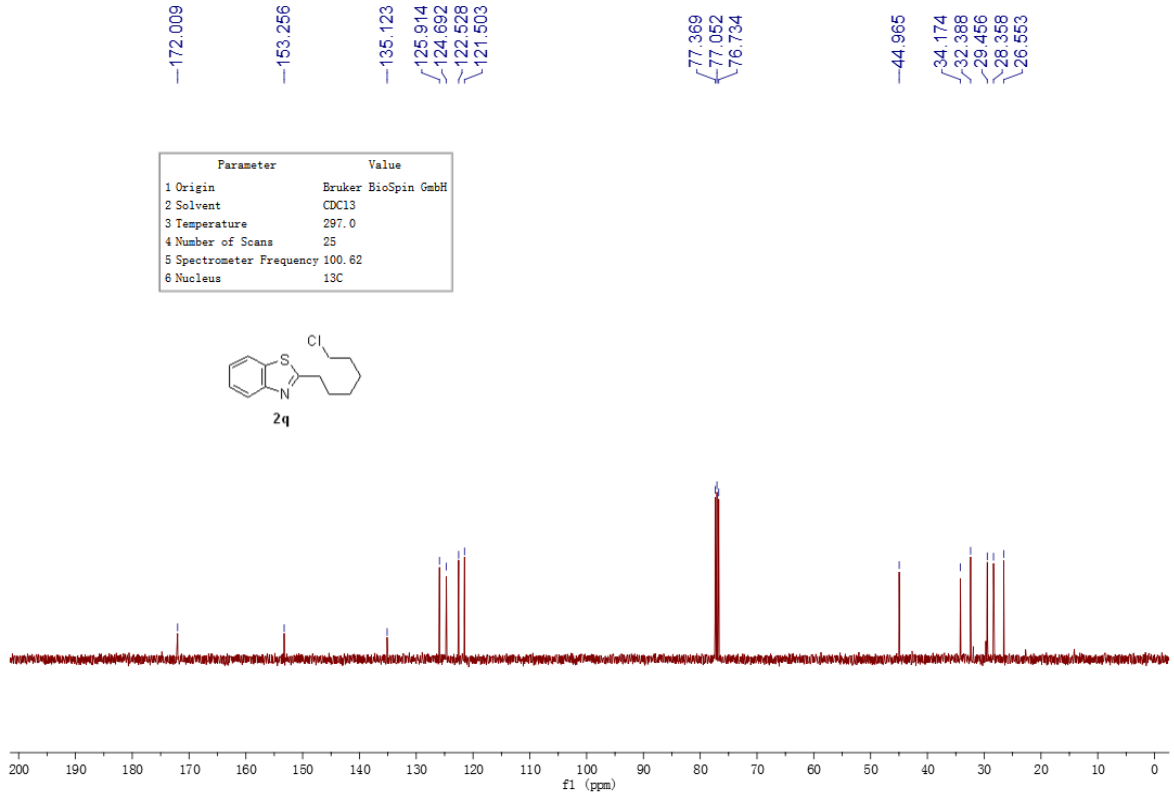

7.976
7.955
7.847
7.828
7.827
7.465
7.462
7.447
7.445
7.427
7.424
7.362
7.359
7.342
7.324
7.321
7.260
3.129
3.110
3.090
1.913
1.895
1.876
1.857
1.837
1.438
1.426
1.419
1.332
1.292
1.283
1.258
1.222
0.891
0.875
0.857
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.6
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
Me
S
N
2o
1.00
0.98
1.02
1.05
1.96
2.02
14.04
3.05
f1 (ppm)

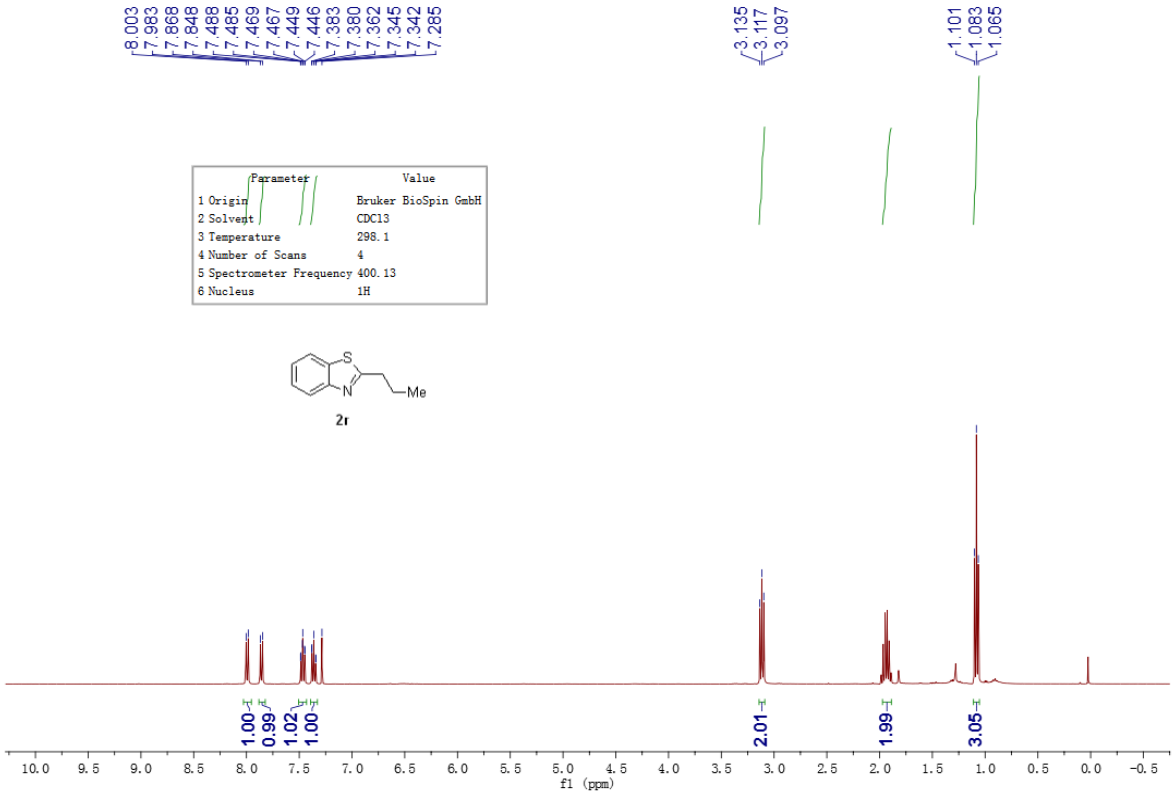

172.488
153.272
135.158
125.850
124.603
122.504
121.481
77.341
77.024
76.706
34.381
31.893
29.744
29.713
29.556
29.471
29.303
29.174
22.682
14.117
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.1
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Me
S
N
2o
f1 (ppm)

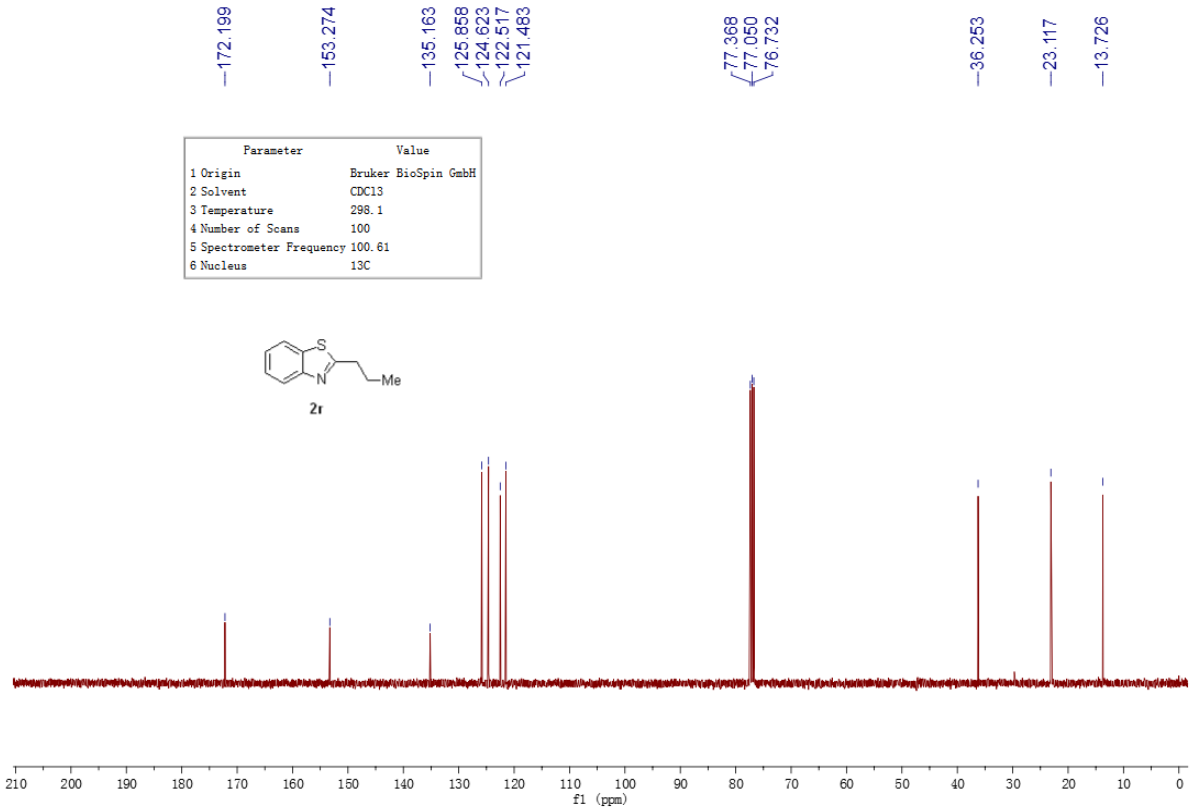

7.974
7.973
7.954
7.953
7.843
7.841
7.823
7.821
7.462
7.459
7.444
7.441
7.439
7.424
7.421
7.358
7.355
7.338
7.335
7.320
7.317
7.260
3.531
3.514
3.497
3.126
3.107
3.087
1.441
1.436
1.422
1.403
1.350
1.343
1.338
1.334
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.7
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
Cl
S
N
2p
1.00
0.98
1.02
1.03
2.00
2.02
2.05
2.01
8.06
f1 (ppm)

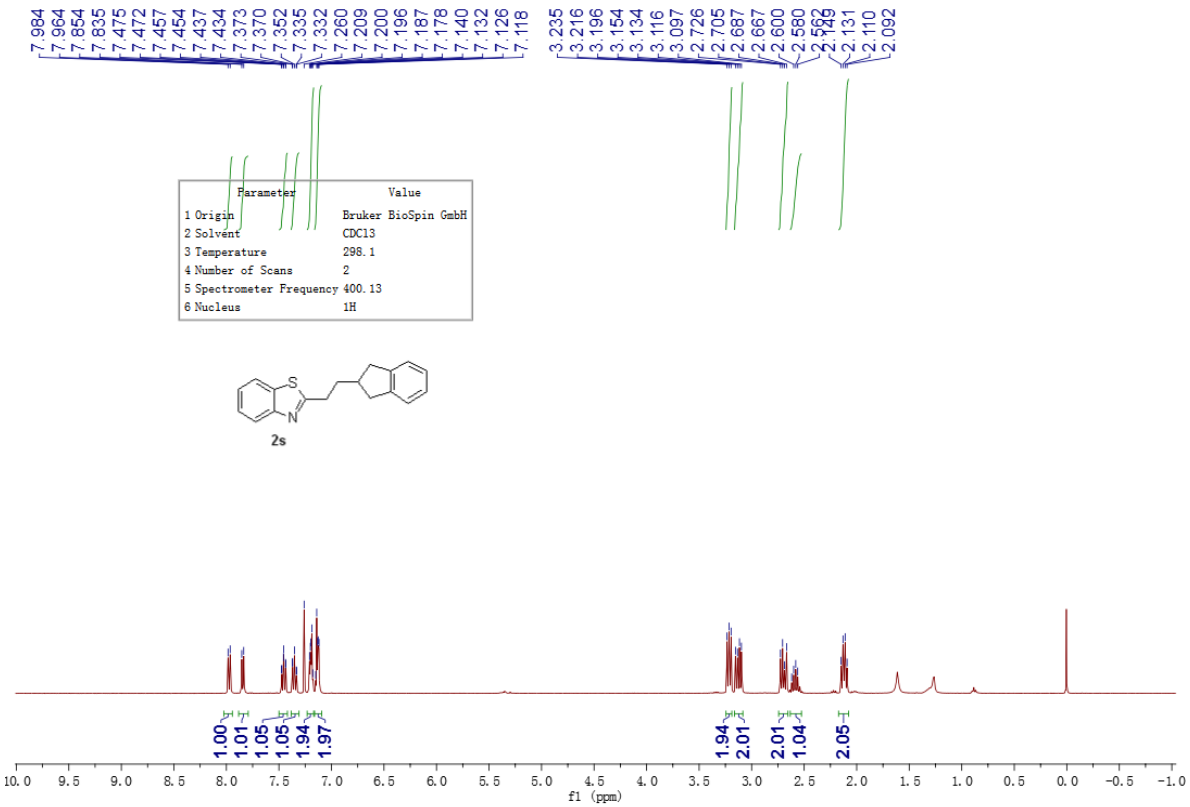

172.287
153.270
135.143
125.877
124.642
122.513
121.488
77.371
77.054
76.736
45.112
34.307
32.588
29.629
29.107
29.002
28.695
26.804
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 297.4
4 Number of Scans 100
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Cl
S
N
2p
f1 (ppm)

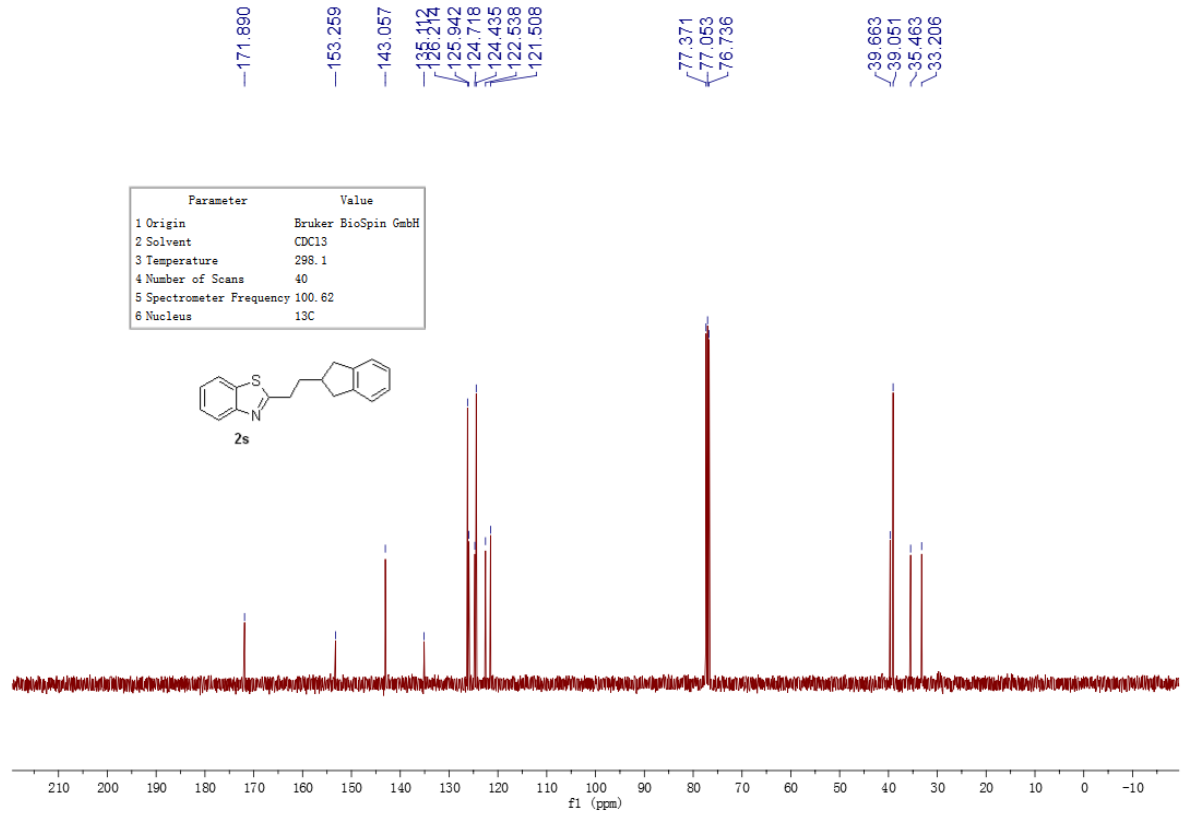

7.975
7.954
7.845
7.825
7.463
7.448
7.445
7.428
7.425
7.363
7.360
7.342
7.260
3.542
3.526
3.509
3.137
3.118
3.098
1.899
1.785
1.766
1.495
1.488
1.480
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Cl
S
N
2q
1.00
0.95
1.04
0.99
2.04
2.04
2.04
2.00
4.01
f1 (ppm)

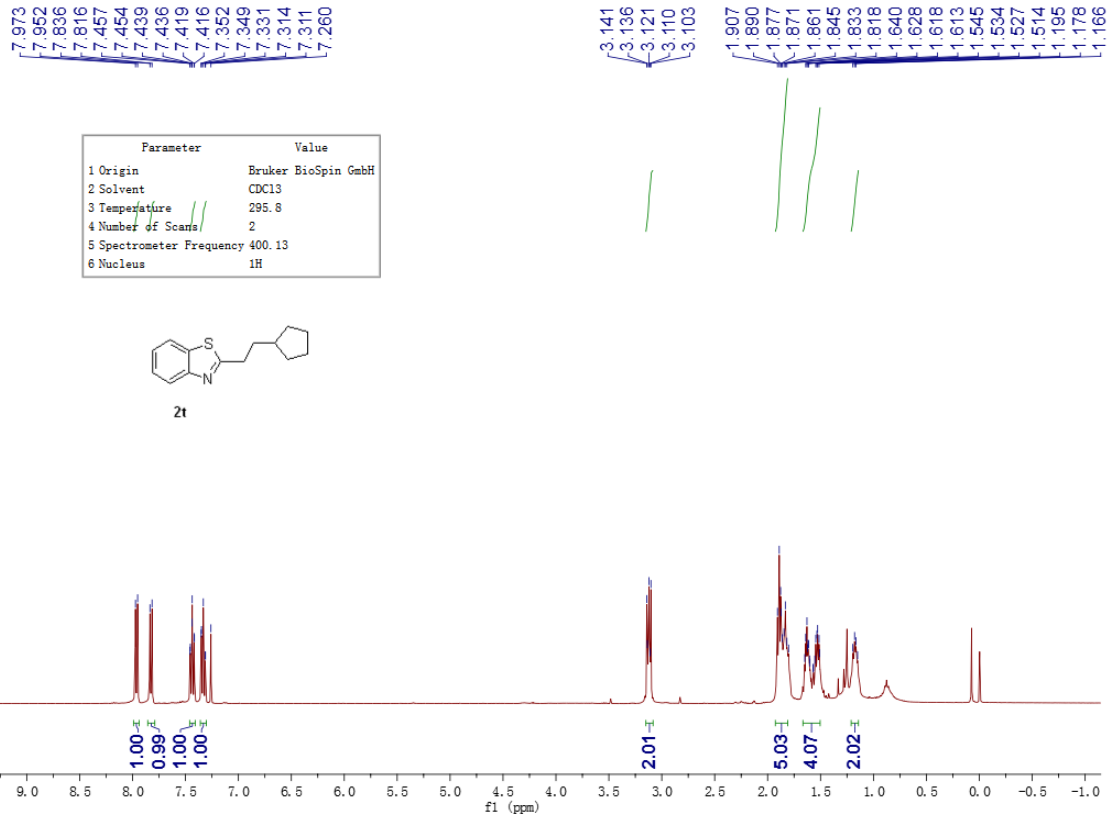

172.009
153.256
135.123
125.914
124.692
122.528
121.503
77.369
77.052
76.734
44.965
34.174
32.388
29.456
28.358
26.553
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 297.0
4 Number of Scans 25
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Cl
S
N
2q
f1 (ppm)

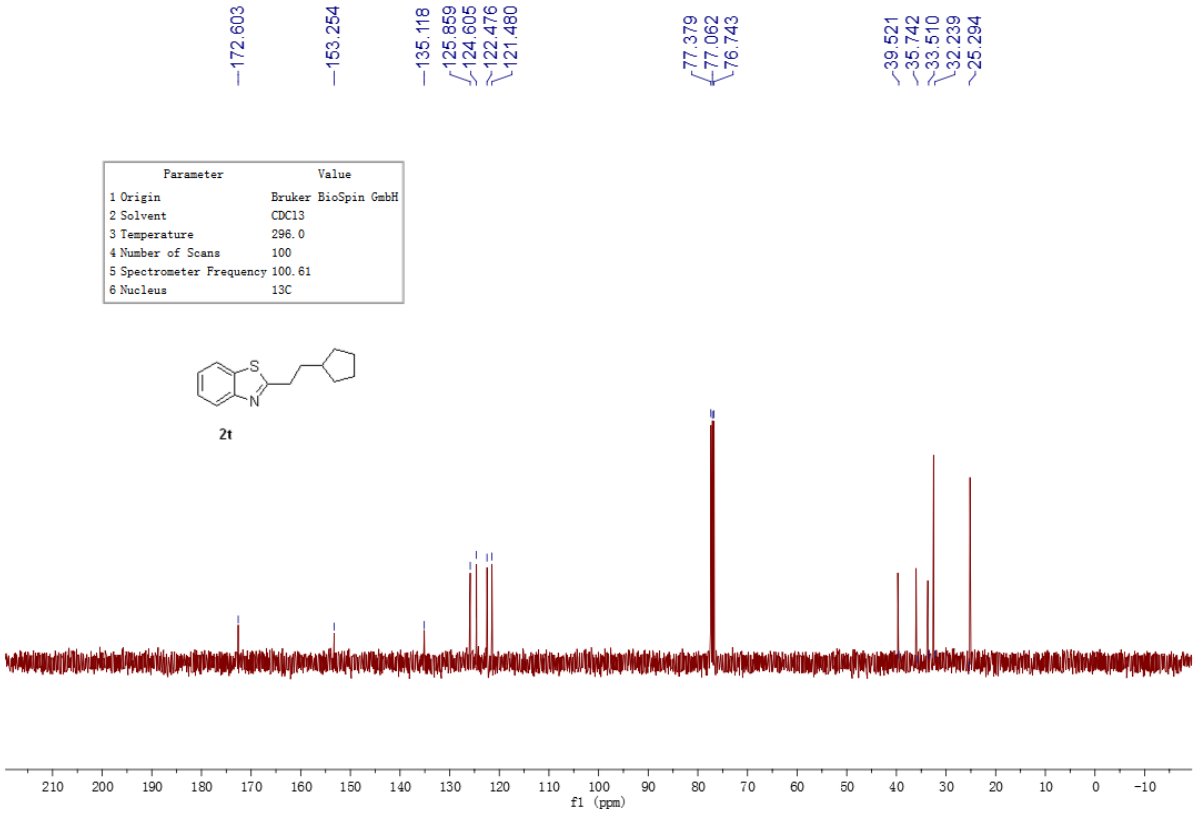

8.003
7.983
7.868
7.848
7.488
7.485
7.469
7.467
7.449
7.446
7.383
7.380
7.362
7.345
7.342
7.285
3.135
3.117
3.097
1.101
1.083
1.065
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
S
N
Me
2r
1.00
0.99
1.02
1.00
2.01
1.99
3.05
f1 (ppm)

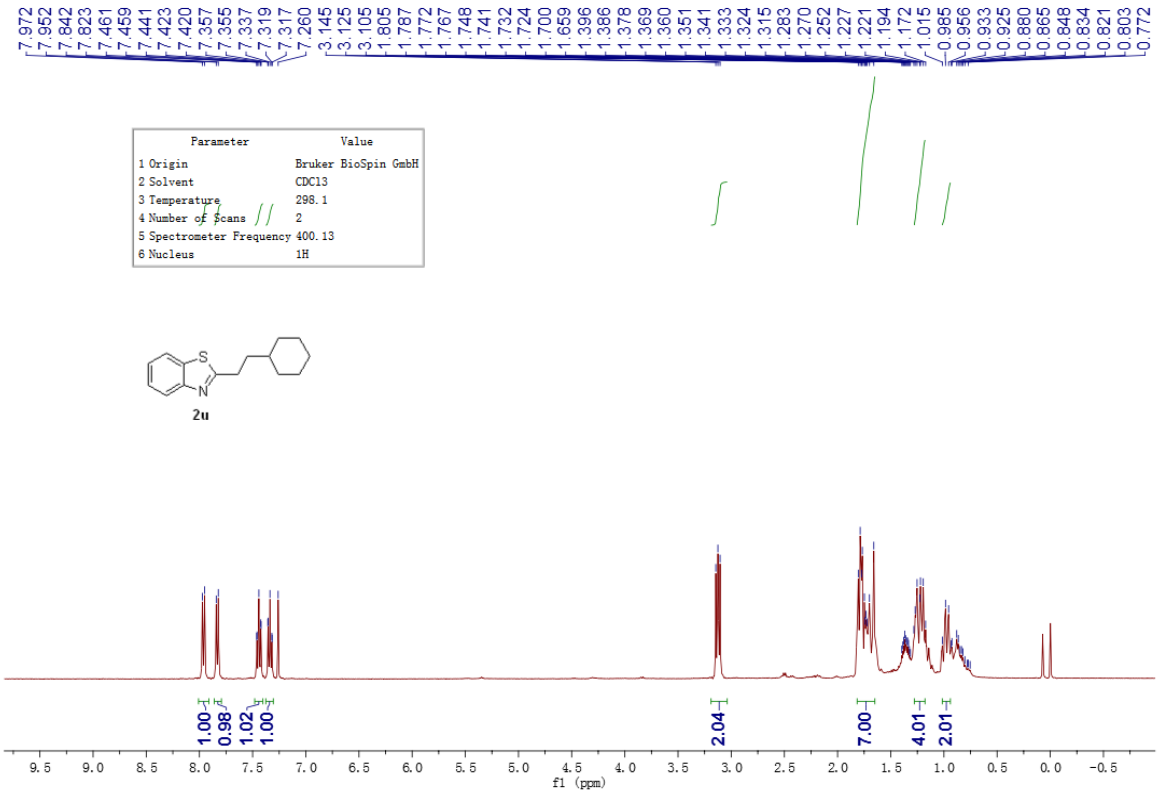

172.199
153.274
135.163
125.858
124.623
122.517
121.483
77.368
77.050
76.732
36.253
23.117
13.726
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 100
5 Spectrometer Frequency 100.61
6 Nucleus 13C
S
N
Me
2r
f1 (ppm)

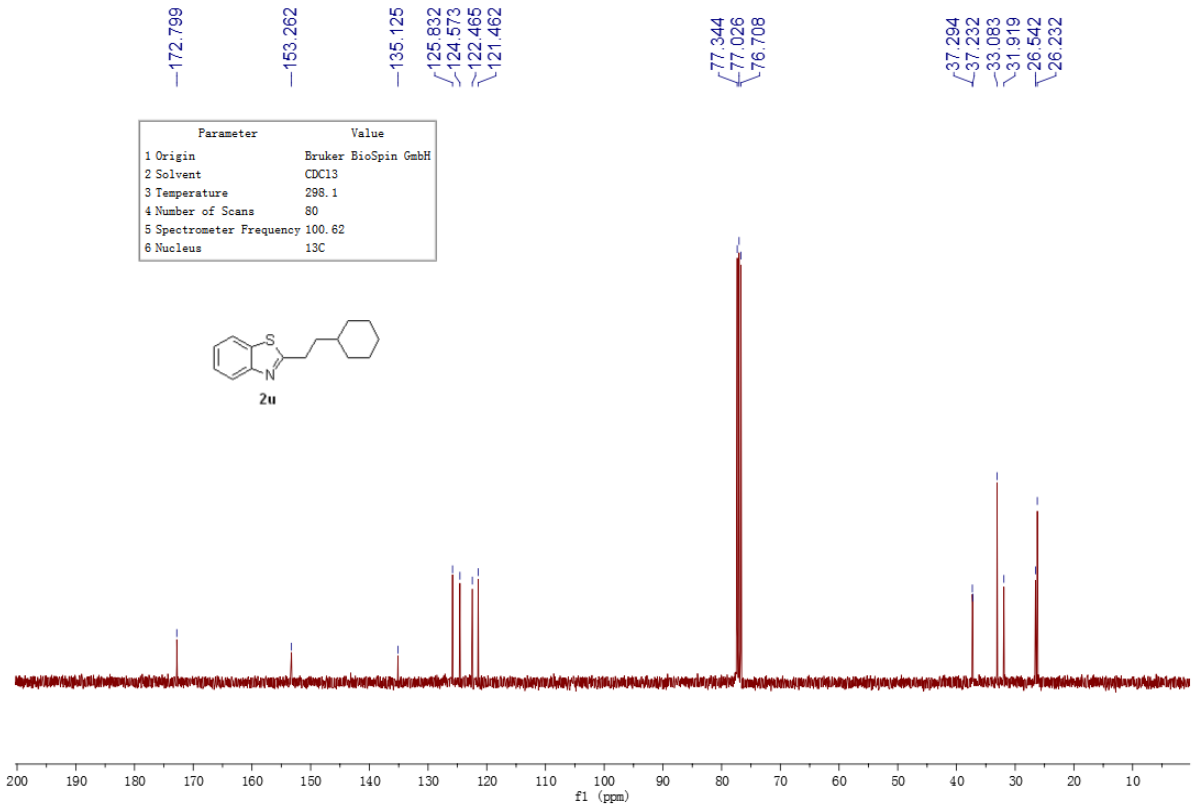

7.984
7.964
7.854
7.835
7.475
7.472
7.457
7.454
7.437
7.434
7.373
7.370
7.352
7.335
7.332
7.260
7.209
7.200
7.196
7.187
7.178
7.140
7.132
7.126
7.118
3.235
3.216
3.196
3.154
3.134
3.116
3.097
2.726
2.705
2.687
2.667
2.600
2.580
2.562
2.549
2.131
2.110
2.092
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
2s
1.00
1.01
1.05
1.05
1.94
1.97
1.94
2.01
2.01
1.04
2.05
f1 (ppm)

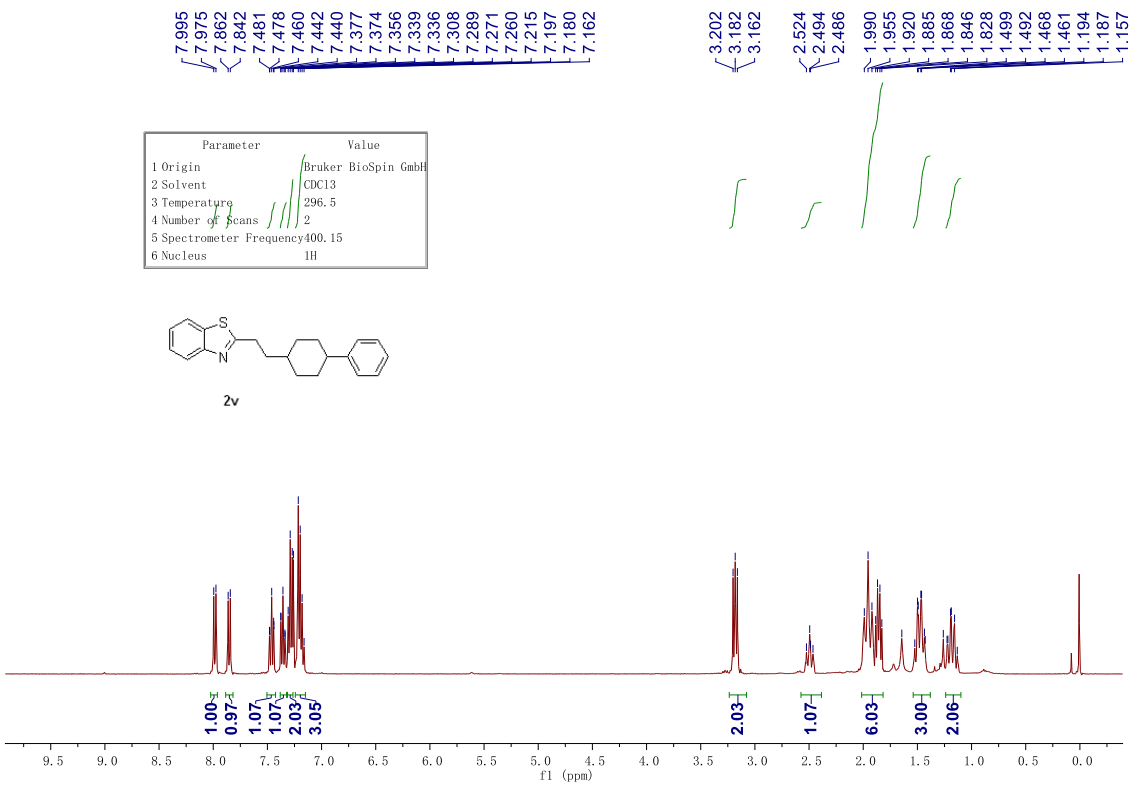

171.890
153.259
143.057
135.112
126.214
125.942
124.718
124.435
122.538
121.508
77.371
77.053
76.736
39.663
39.051
35.463
33.206
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 40
5 Spectrometer Frequency 100.62
6 Nucleus 13C
2s
f1 (ppm)

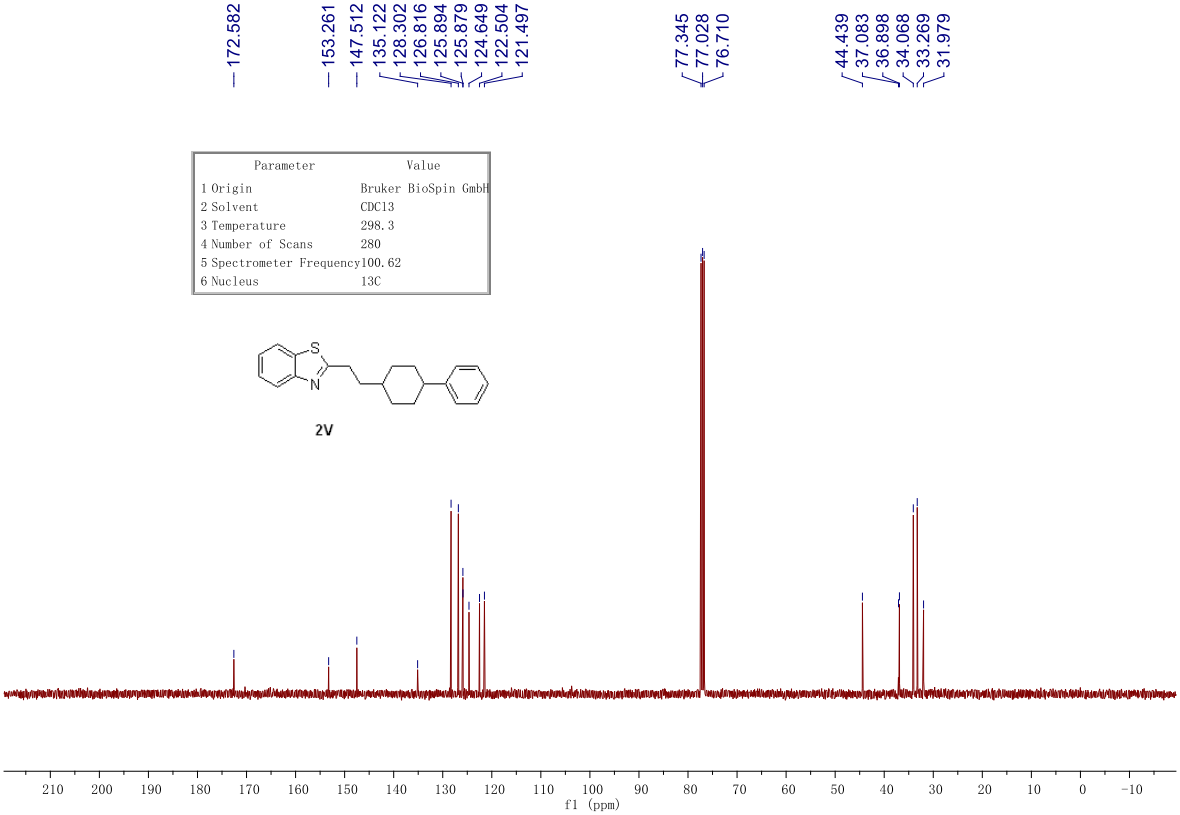

7.973
7.952
7.836
7.816
7.457
7.454
7.439
7.436
7.419
7.416
7.352
7.349
7.331
7.314
7.311
7.260
3.141
3.136
3.121
3.110
3.103
1.907
1.890
1.877
1.871
1.861
1.845
1.833
1.818
1.640
1.628
1.618
1.613
1.545
1.534
1.527
1.514
1.195
1.178
1.166
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.8
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
2t
1.00
0.99
1.00
1.00
2.01
5.03
4.07
2.02
f1 (ppm)

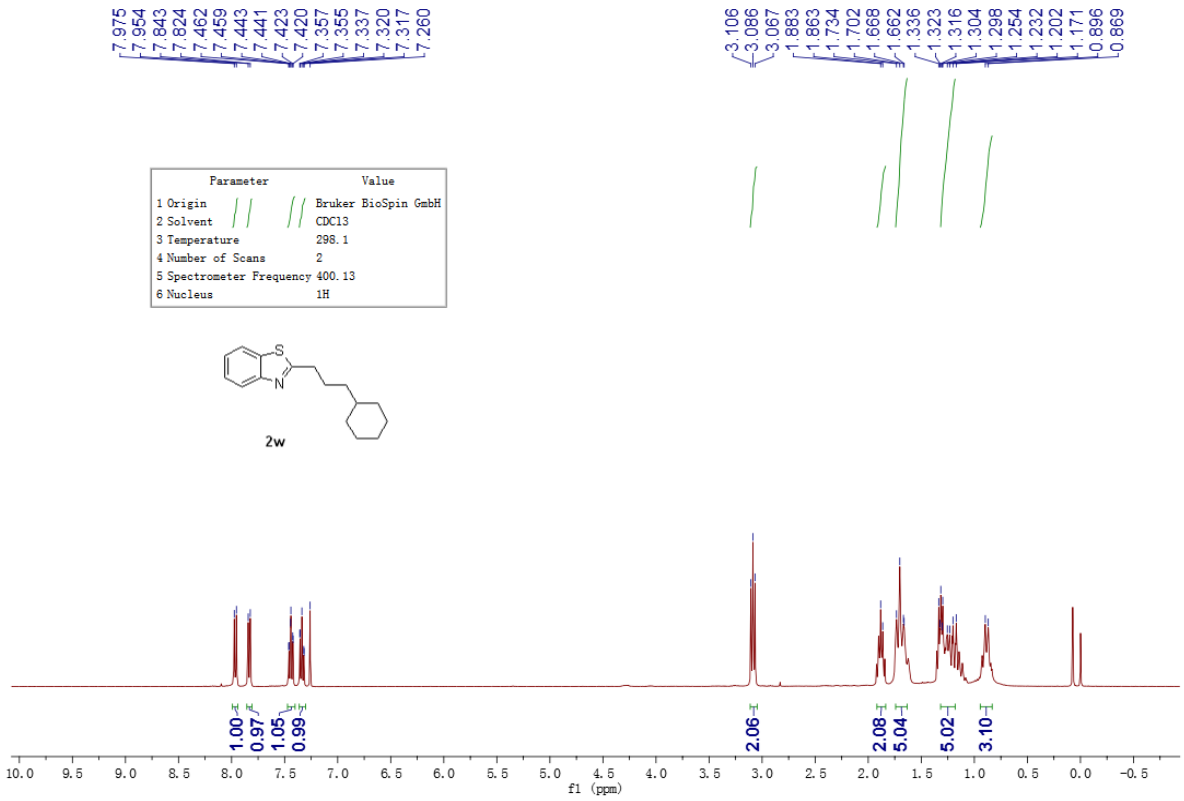

172.603
153.254
135.118
125.859
124.605
122.476
121.480
77.379
77.062
76.743
39.521
35.742
33.510
32.239
25.294
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.0
4 Number of Scans 100
5 Spectrometer Frequency 100.61
6 Nucleus 13C
2t
f1 (ppm)

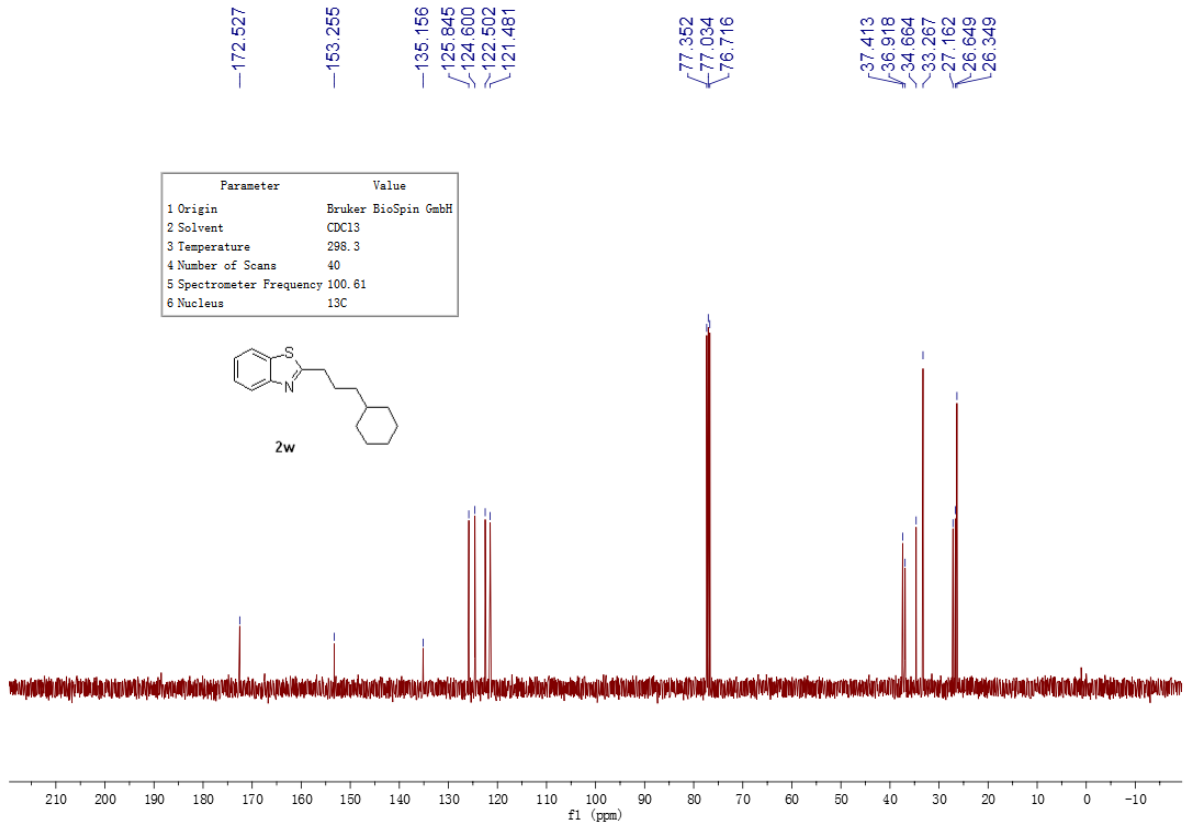

7.972
7.952
7.842
7.823
7.461
7.459
7.441
7.423
7.420
7.357
7.355
7.337
7.319
7.317
7.260
3.145
3.125
3.105
1.805
1.787
1.772
1.767
1.748
1.741
1.732
1.724
1.700
1.659
1.396
1.386
1.378
1.369
1.360
1.351
1.341
1.333
1.324
1.315
1.283
1.270
1.252
1.227
1.221
1.194
1.172
1.015
0.985
0.956
0.933
0.925
0.880
0.865
0.848
0.834
0.821
0.803
0.772
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
2u
1.00
0.98
1.02
1.00
2.04
7.00
4.01
2.01
f1 (ppm)

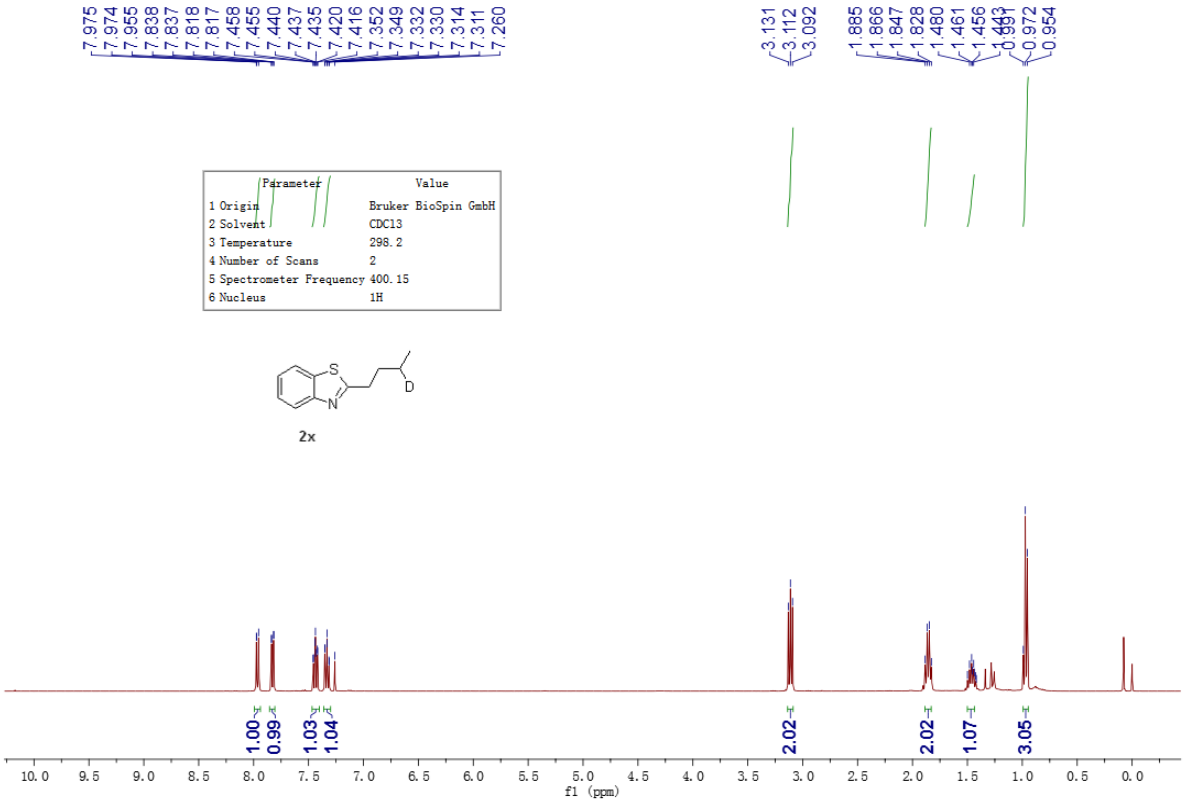

172.799
153.262
135.125
125.832
124.573
122.465
121.462
77.344
77.026
76.708
37.294
37.232
33.083
31.919
26.542
26.232
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
2u
f1 (ppm)

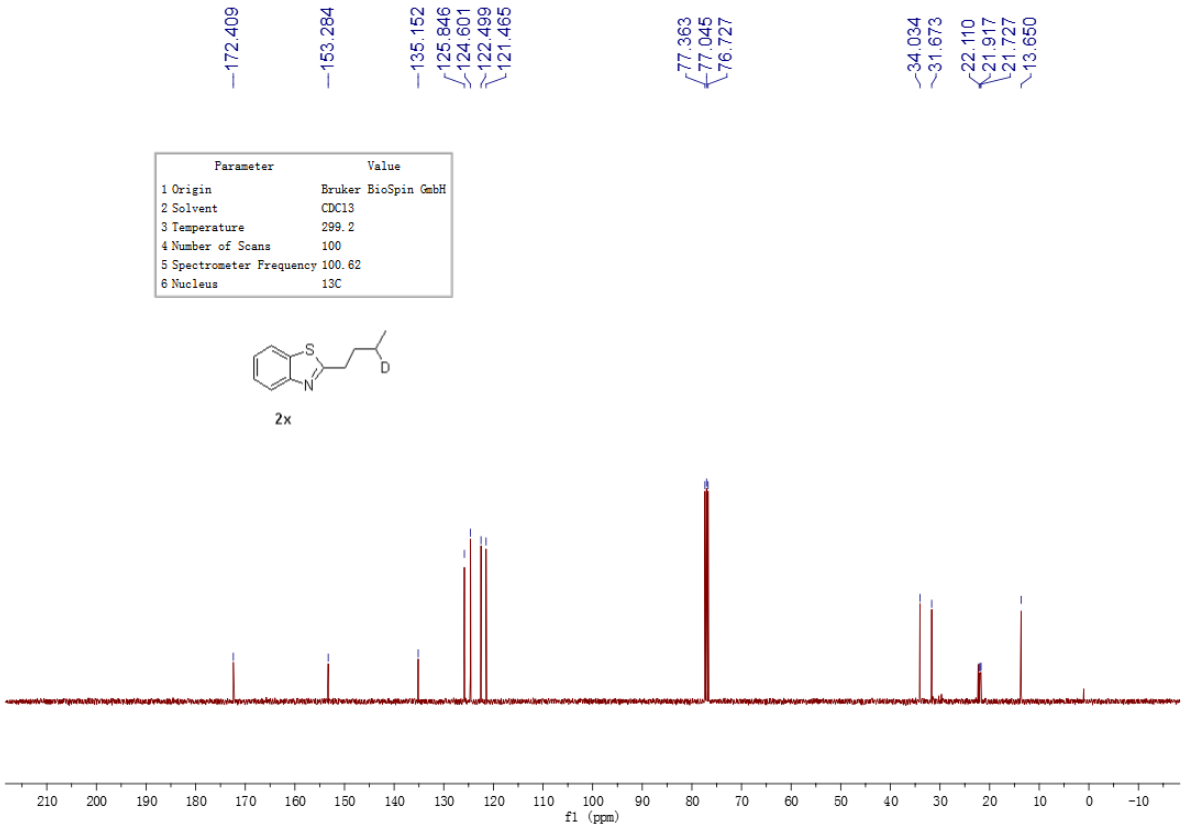

7.995
7.975
7.862
7.842
7.481
7.478
7.460
7.442
7.440
7.377
7.374
7.356
7.339
7.336
7.308
7.289
7.271
7.260
7.215
7.197
7.180
7.162
3.202
3.182
3.162
2.524
2.494
2.486
1.990
1.955
1.920
1.885
1.868
1.846
1.828
1.499
1.492
1.468
1.461
1.194
1.187
1.157
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.5
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
2v
1.00
0.97
1.07
1.07
2.03
3.05
2.03
1.07
6.03
3.00
2.06
f1 (ppm)

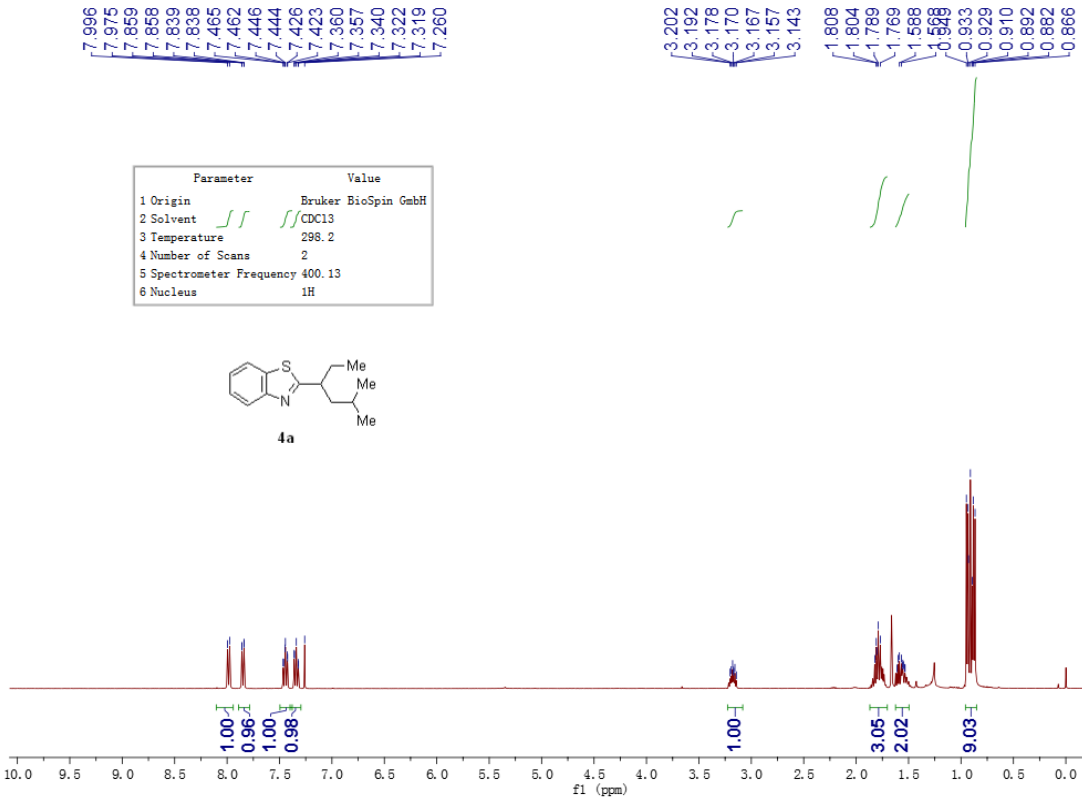

172.582
153.261
147.512
135.122
128.302
126.816
125.894
125.879
124.649
122.504
121.497
77.345
77.028
76.710
44.439
37.083
36.898
34.068
33.269
31.979
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.3
4 Number of Scans 280
5 Spectrometer Frequency 100.62
6 Nucleus 13C
2V
f1 (ppm)

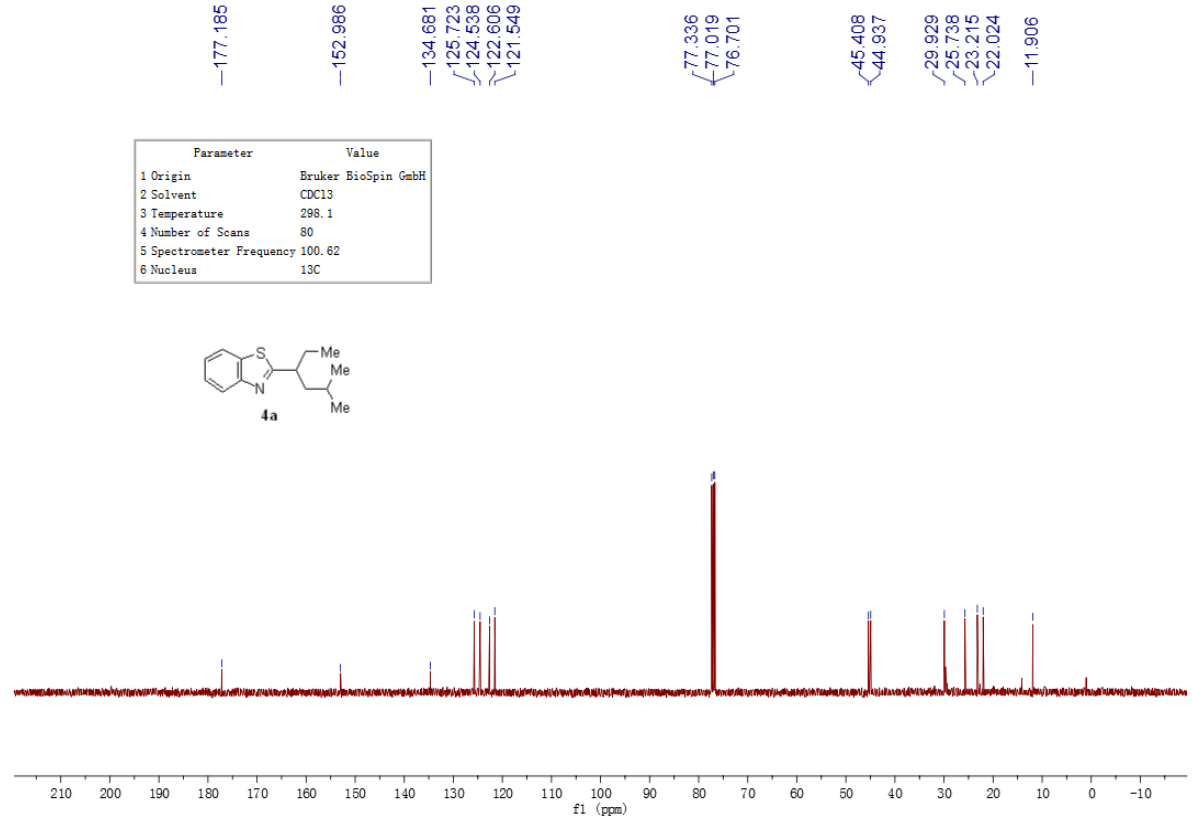

7.975
7.954
7.843
7.824
7.462
7.459
7.443
7.441
7.423
7.420
7.357
7.355
7.337
7.320
7.317
7.260
3.106
3.086
3.067
1.883
1.863
1.734
1.702
1.668
1.662
1.336
1.323
1.316
1.304
1.298
1.254
1.232
1.202
1.171
0.896
0.869
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
2w
1.00
0.97
1.05
0.99
2.06
2.08
5.04
5.02
3.10
f1 (ppm)

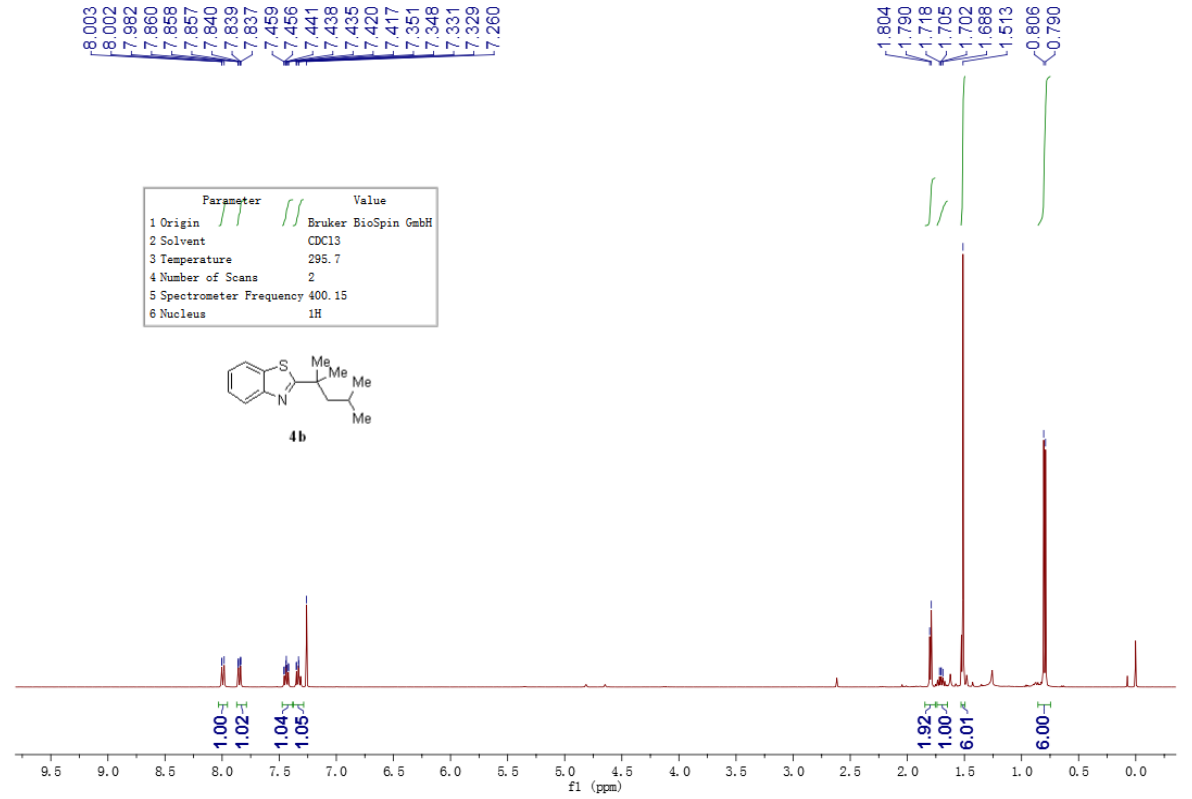

172.527
153.255
135.156
125.845
124.600
122.502
121.481
77.352
77.034
76.716
37.413
36.918
34.664
33.267
27.162
26.649
26.349
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.3
4 Number of Scans 40
5 Spectrometer Frequency 100.61
6 Nucleus 13C
2w
f1 (ppm)

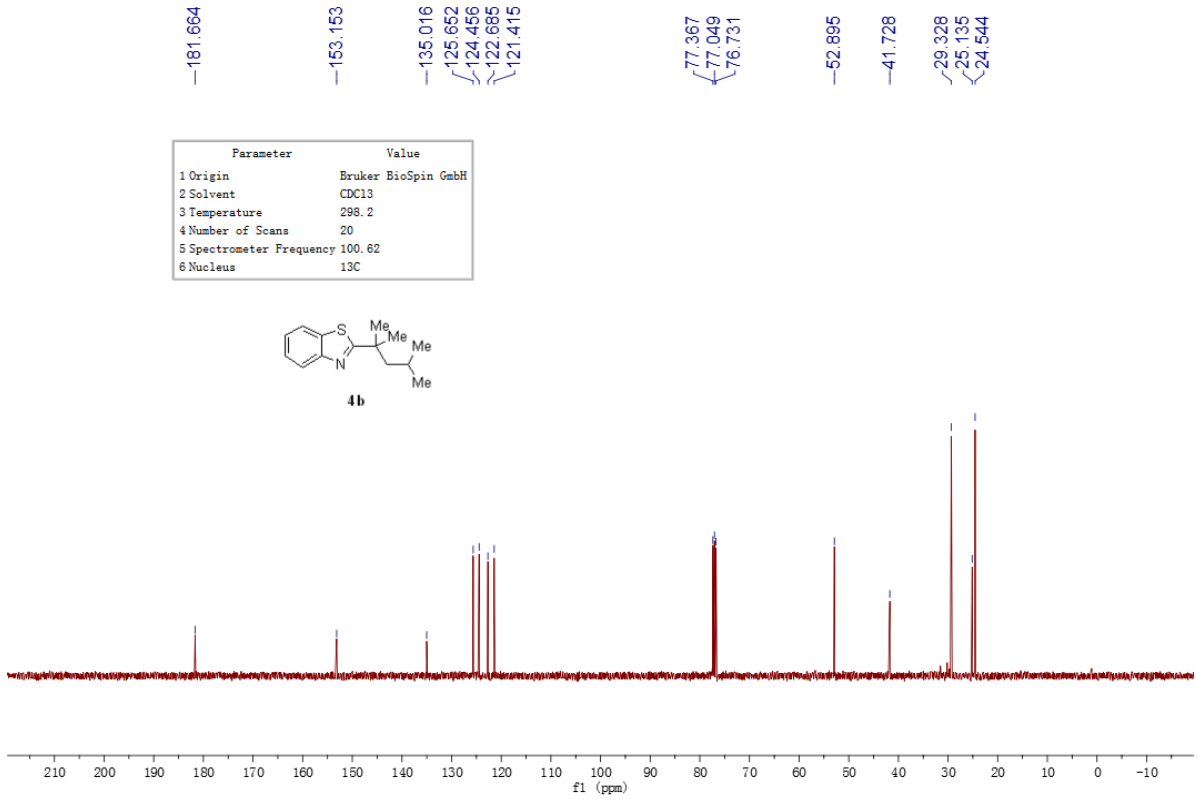

7.975
7.974
7.955
7.838
7.837
7.818
7.817
7.458
7.455
7.440
7.437
7.435
7.420
7.416
7.352
7.349
7.332
7.330
7.314
7.311
7.260
3.131
3.112
3.092
1.885
1.866
1.847
1.828
1.480
1.461
1.456
0.991
0.972
0.954
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
2x
1.00
0.99
1.03
1.04
2.02
2.02
1.07
3.05
f1 (ppm)

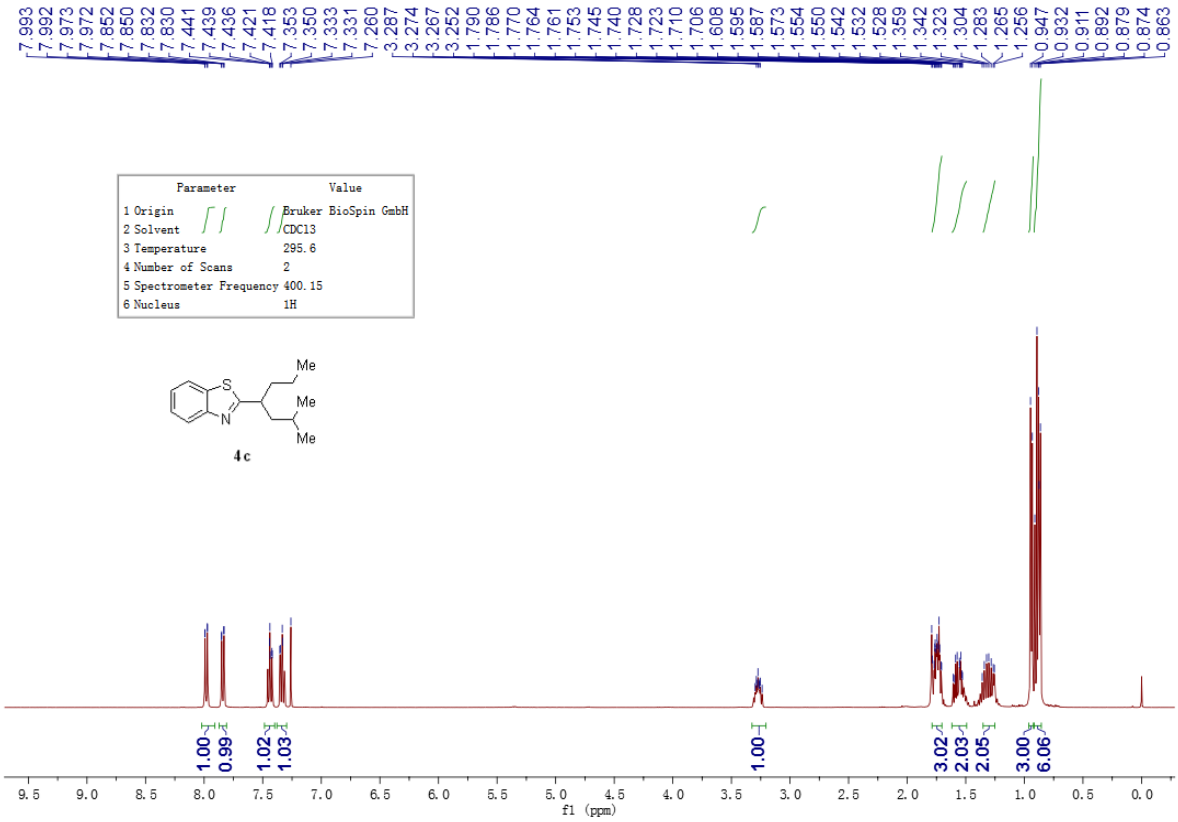

172.409
153.284
135.152
125.846
124.601
122.499
121.465
77.363
77.045
76.727
34.034
31.673
22.110
21.917
21.727
13.650
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 299.2
4 Number of Scans 100
5 Spectrometer Frequency 100.62
6 Nucleus 13C
2x
f1 (ppm)

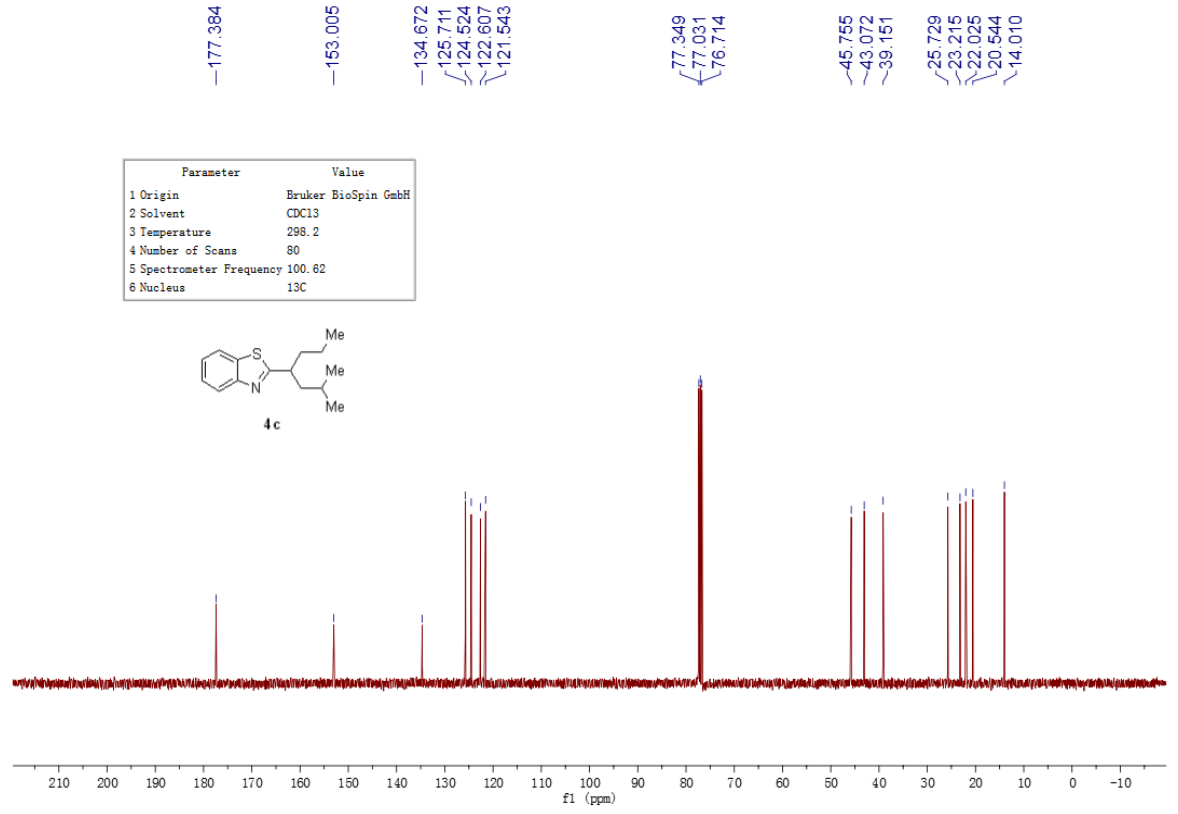

7.996
7.975
7.859
7.858
7.839
7.838
7.465
7.462
7.446
7.444
7.426
7.423
7.360
7.357
7.340
7.322
7.319
7.260
3.202
3.192
3.178
3.170
3.167
3.157
3.143
1.808
1.804
1.789
1.769
1.588
0.933
0.929
0.910
0.892
0.882
0.866
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Me
Me
Me
4a
1.00
0.96
1.00
0.98
1.00
3.05
2.02
9.03
f1 (ppm)

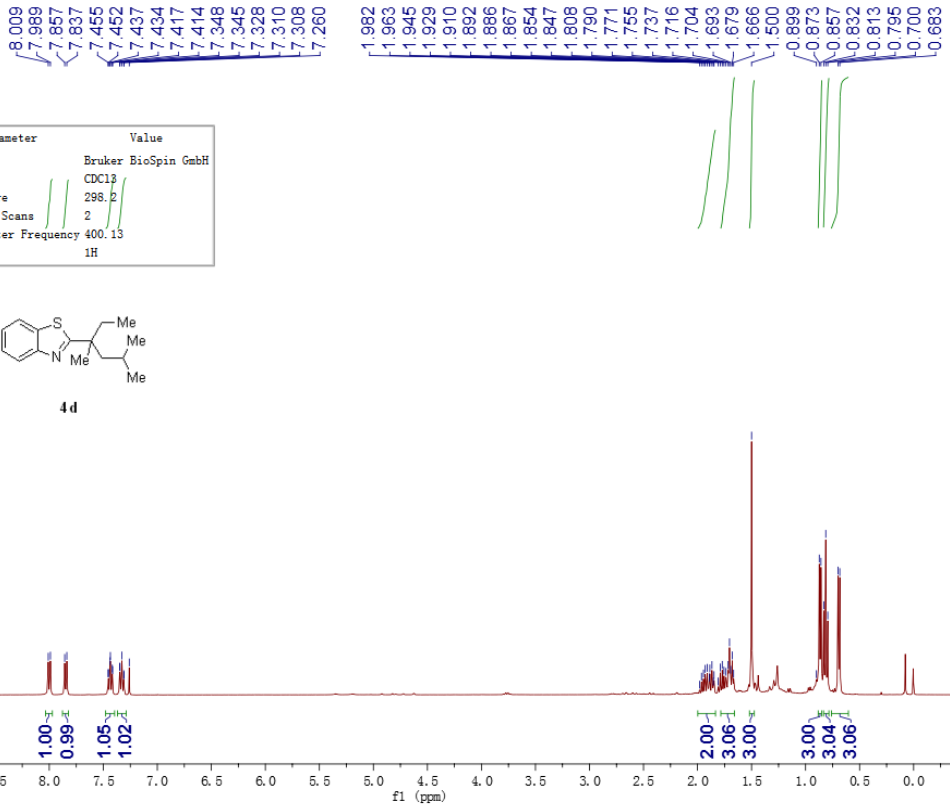

177.185
152.986
134.681
125.723
124.538
122.606
121.549
77.336
77.019
76.701
45.408
44.937
29.929
25.738
23.215
22.024
11.906
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Me
Me
Me
4a
f1 (ppm)

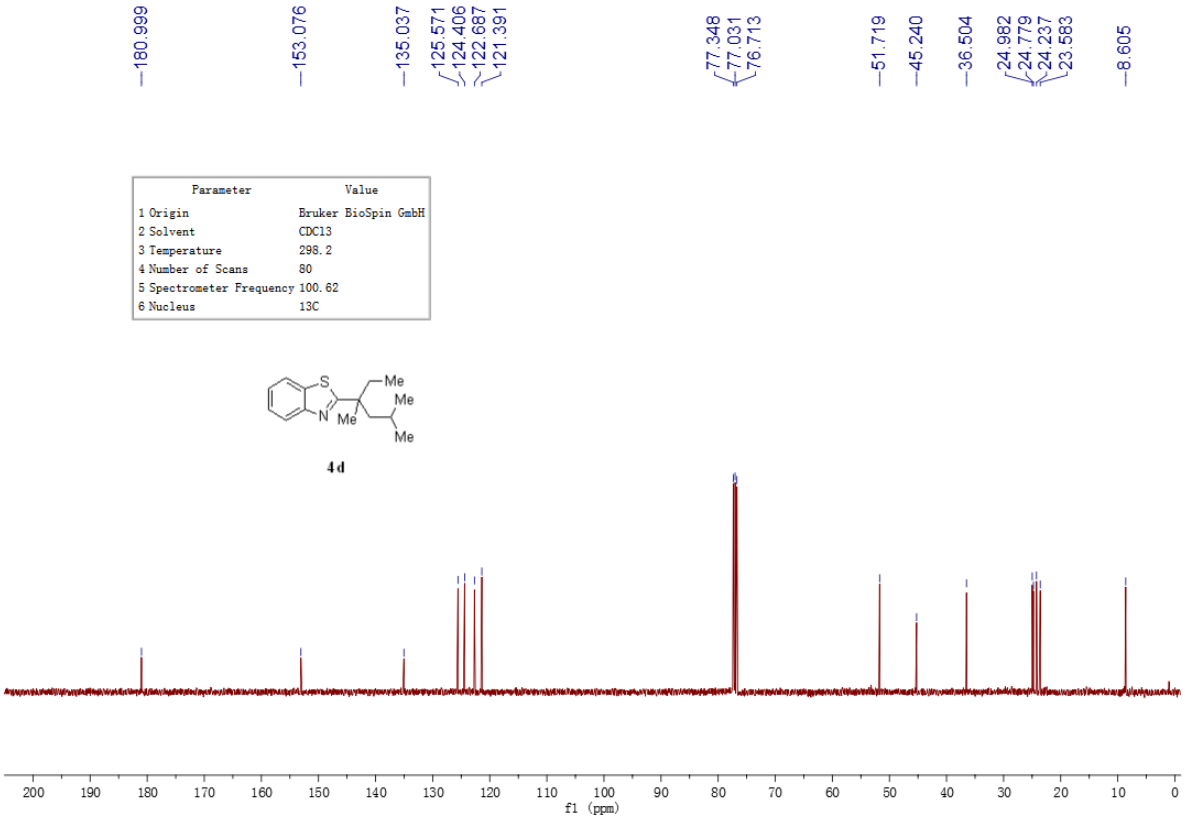

8.003
8.002
7.982
7.860
7.858
7.857
7.840
7.839
7.837
7.459
7.456
7.441
7.438
7.435
7.420
7.417
7.351
7.348
7.331
7.329
7.260
1.804
1.790
1.718
1.705
1.702
1.688
1.513
0.806
0.790
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.7
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
4b
1.00
1.02
1.04
1.05
1.92
1.00
6.01
6.00
f1 (ppm)

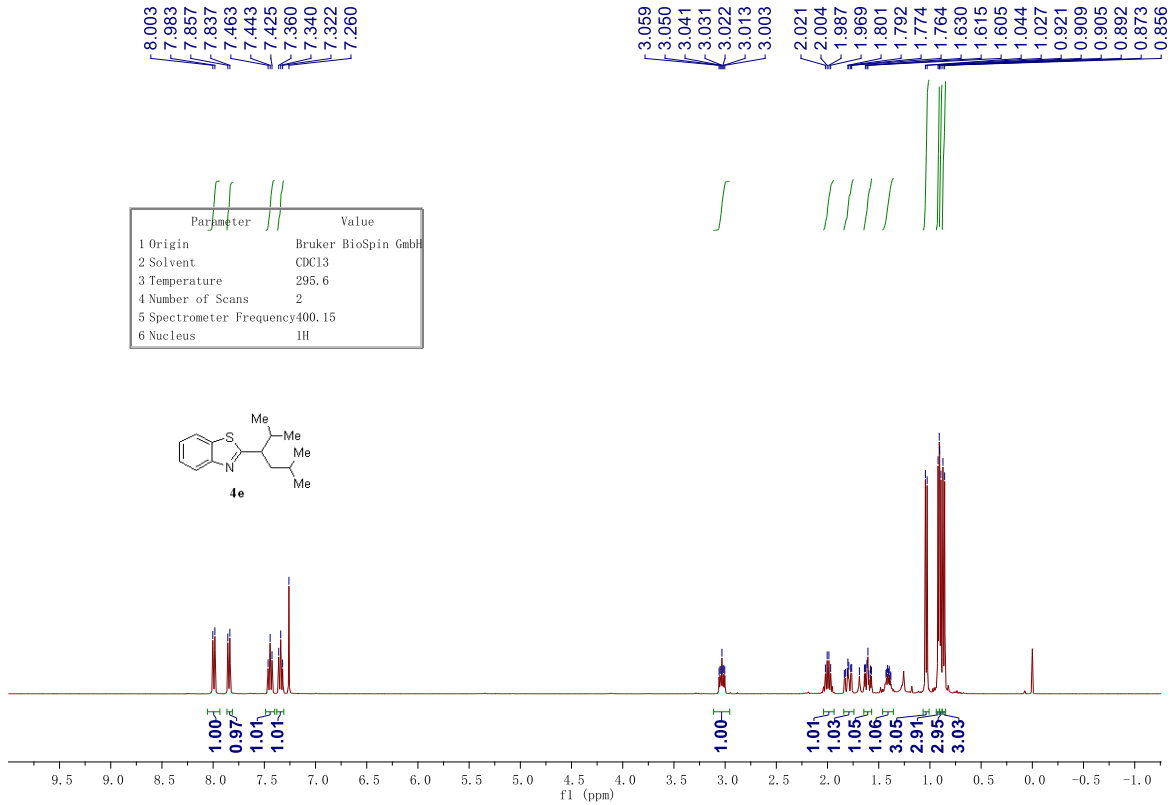

181.664
153.153
135.016
125.652
124.456
122.685
121.415
77.367
77.049
76.731
52.895
41.728
29.328
25.135
24.544
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 20
5 Spectrometer Frequency 100.62
6 Nucleus 13C
4b
f1 (ppm)

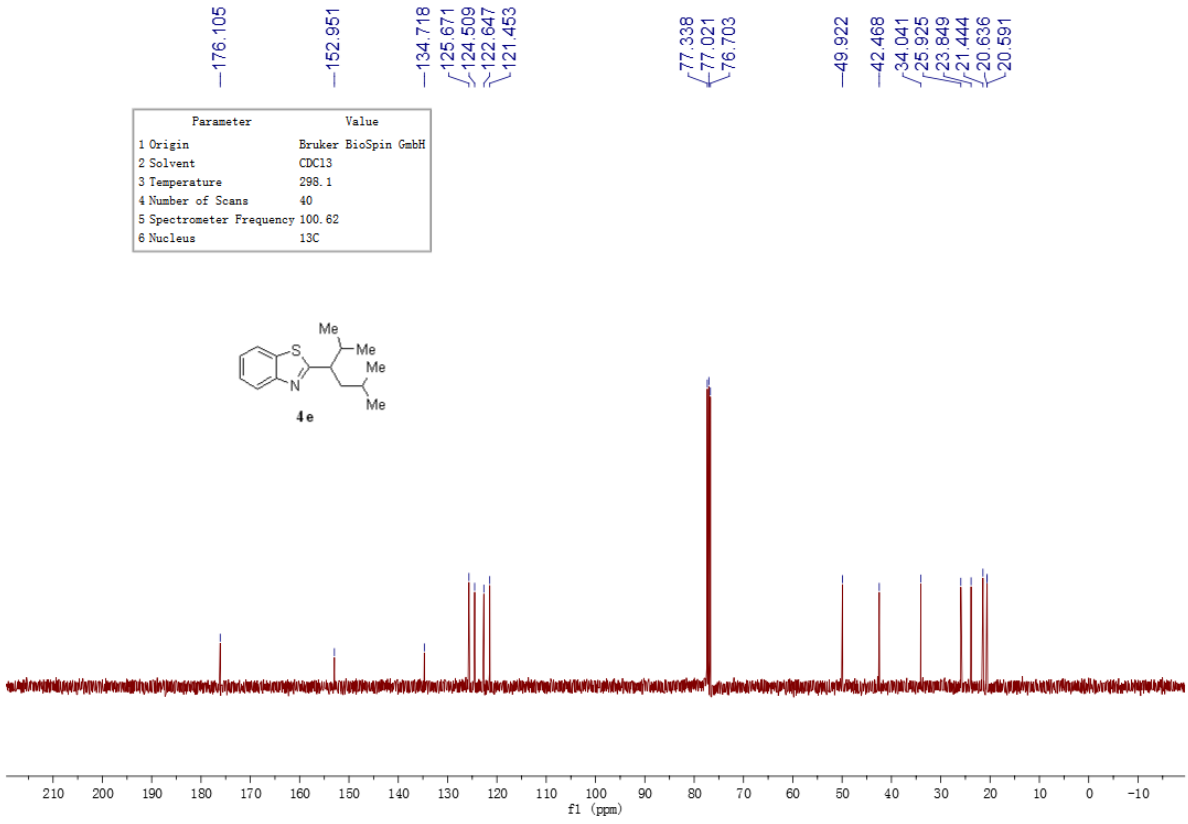

7.993
7.992
7.973
7.972
7.852
7.850
7.832
7.830
7.441
7.439
7.436
7.421
7.418
7.353
7.350
7.333
7.331
7.260
3.287
3.274
3.267
3.252
1.790
1.786
1.770
1.764
1.761
1.753
1.745
1.740
1.728
1.723
1.710
1.706
1.608
1.595
1.587
1.573
1.554
1.550
1.542
1.532
1.528
1.359
1.342
1.323
1.304
1.283
1.265
1.256
0.947
0.932
0.911
0.892
0.879
0.874
0.863
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.6
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
Me
Me
Me
4c
1.00
0.99
1.02
1.03
1.00
3.02
2.03
2.05
3.00
6.06
f1 (ppm)

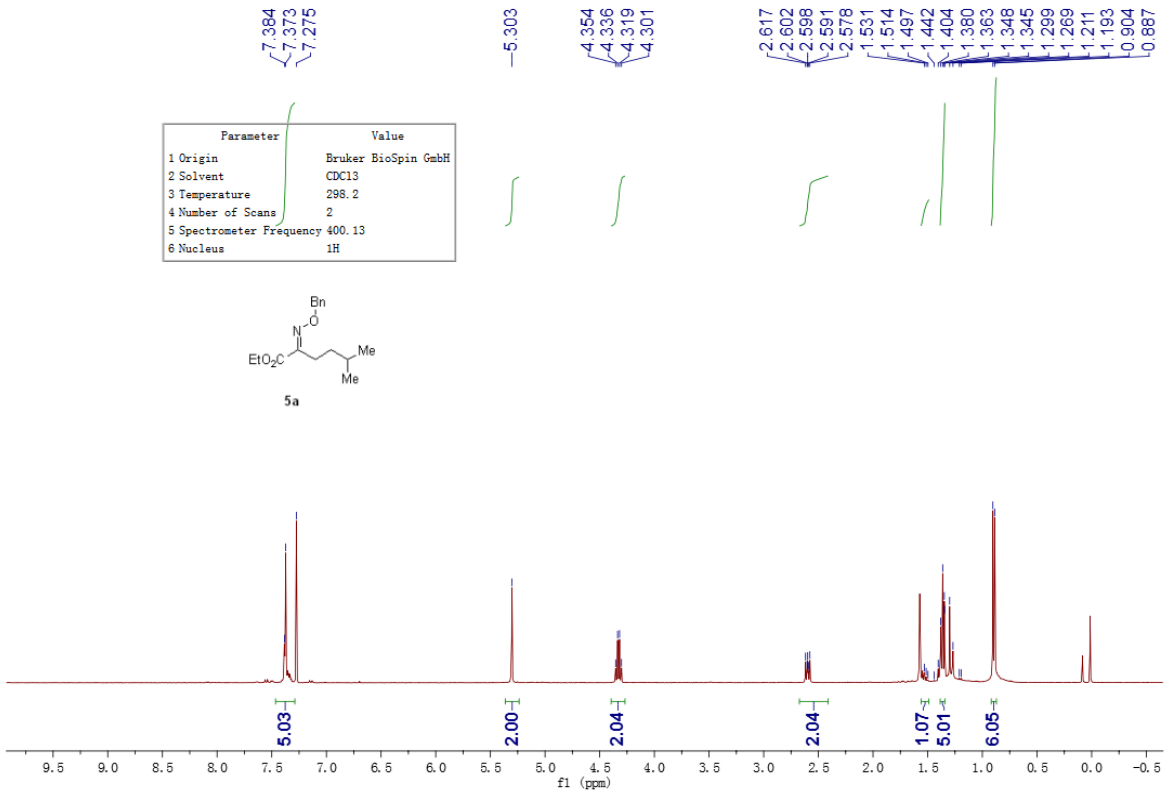

177.384
153.005
134.672
125.711
124.524
122.607
121.543
77.349
77.031
76.714
45.755
43.072
39.151
25.729
23.215
22.025
20.544
14.010
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Me
Me
Me
4c
f1 (ppm)

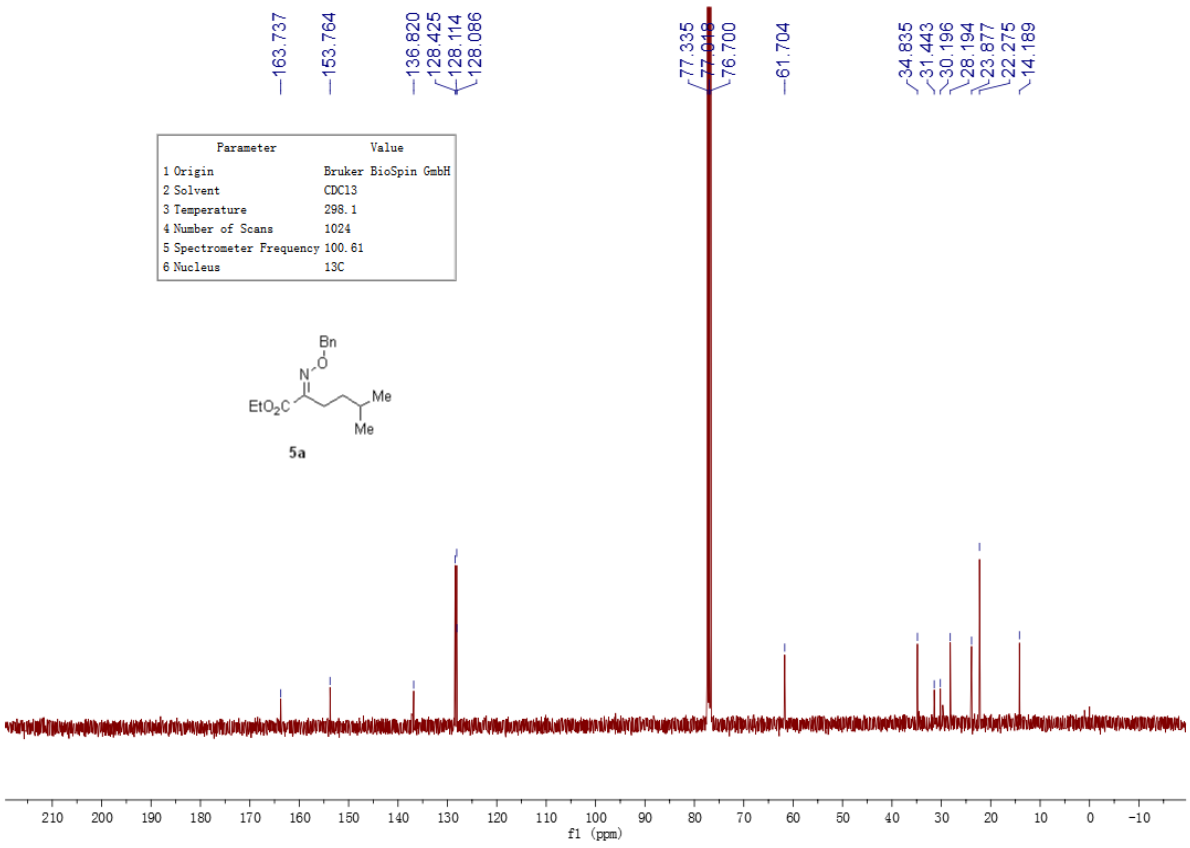

8.009
7.989
7.857
7.837
7.455
7.452
7.437
7.434
7.417
7.414
7.348
7.345
7.328
7.310
7.308
7.260
1.982
1.963
1.945
1.929
1.910
1.892
1.886
1.867
1.854
1.847
1.808
1.790
1.771
1.755
1.737
1.716
1.704
1.693
1.679
1.666
1.500
0.899
0.873
0.857
0.832
0.813
0.795
0.700
0.683
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Me
Me
Me
Me
S
N
4d
1.00
0.99
1.05
1.02
2.00
3.06
3.00
3.00
3.04
3.06
f1 (ppm)

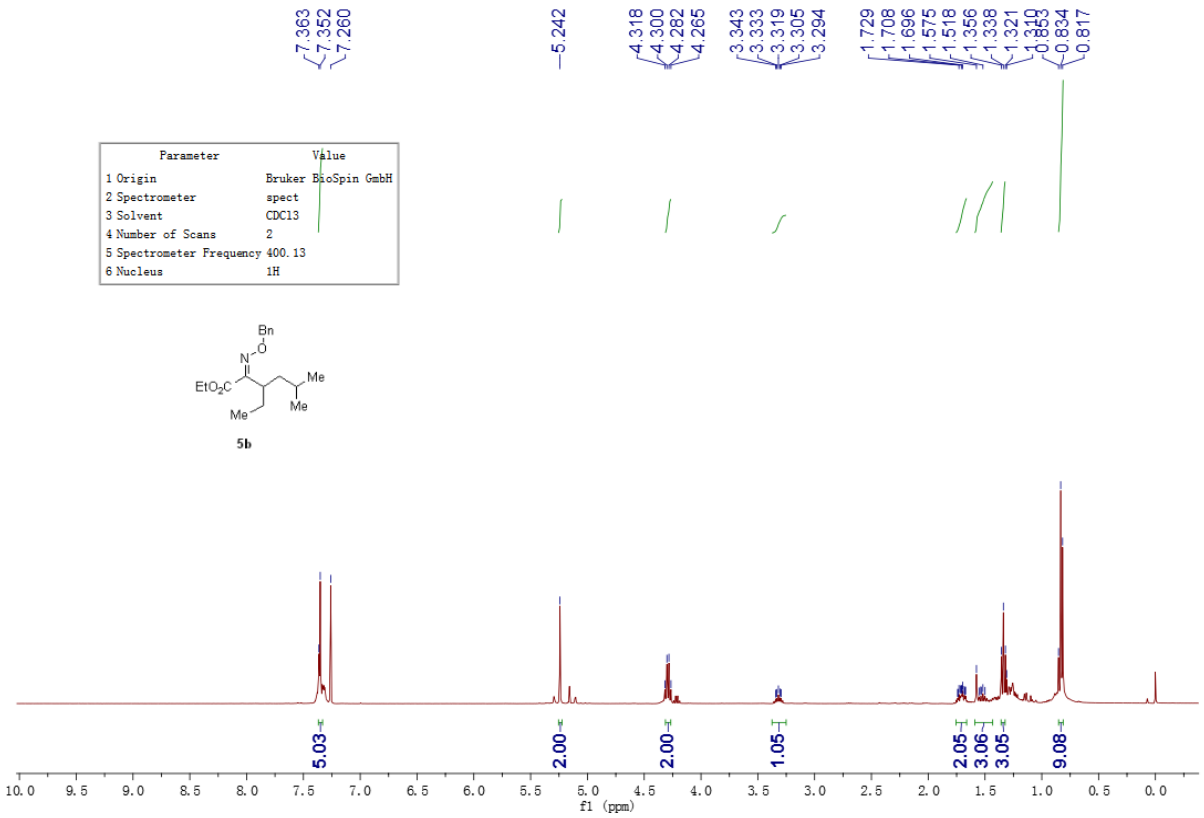

180.999
153.076
135.037
125.571
124.406
122.687
121.391
77.348
77.031
76.713
51.719
45.240
36.504
24.982
24.779
24.237
23.583
8.605
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Me
Me
Me
Me
S
N
4d
f1 (ppm)

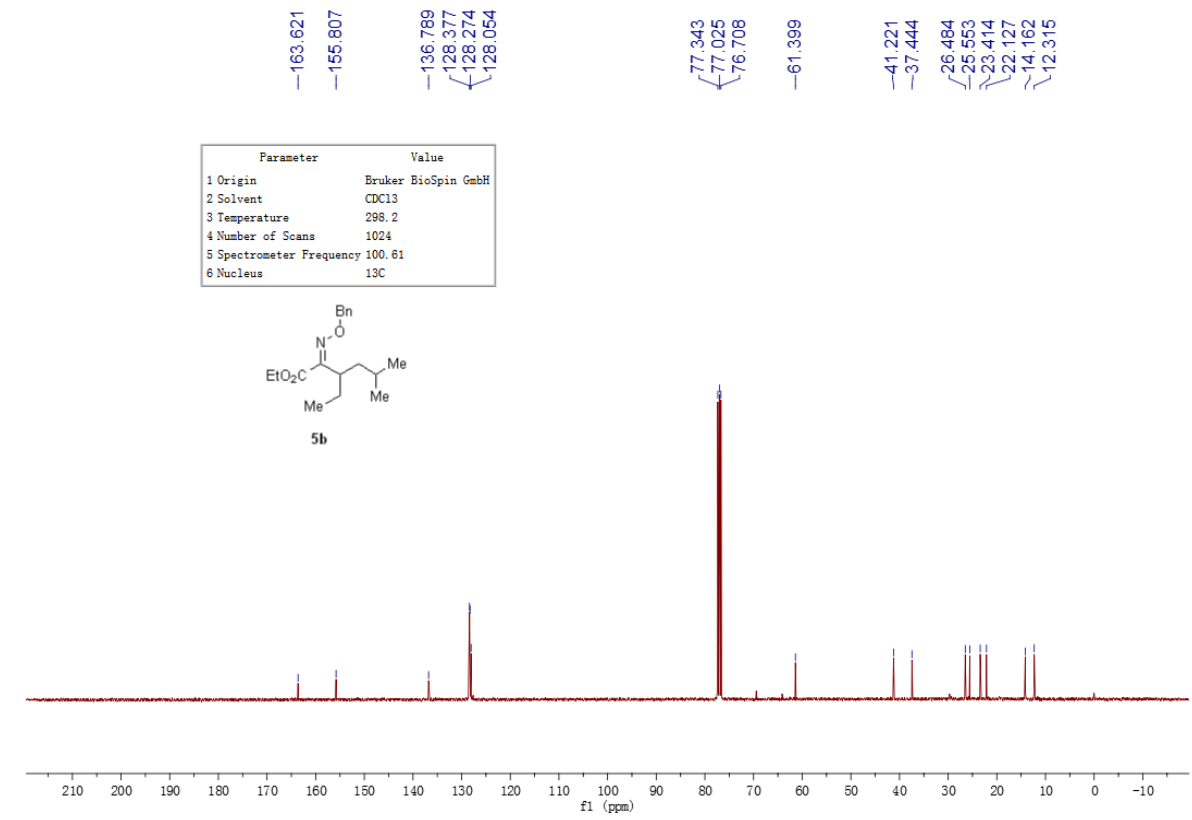

8.003
7.983
7.857
7.837
7.463
7.443
7.425
7.360
7.340
7.322
7.260
3.059
3.050
3.041
3.031
3.022
3.013
3.003
2.021
2.004
1.987
1.969
1.801
1.792
1.774
1.764
1.630
1.615
1.605
1.044
1.027
0.921
0.909
0.905
0.892
0.873
0.856
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.6
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
Me
Me
Me
Me
S
N
4e
1.00
0.97
1.01
1.01
1.00
1.01
1.03
1.05
1.06
3.05
2.91
2.95
3.03
f1 (ppm)

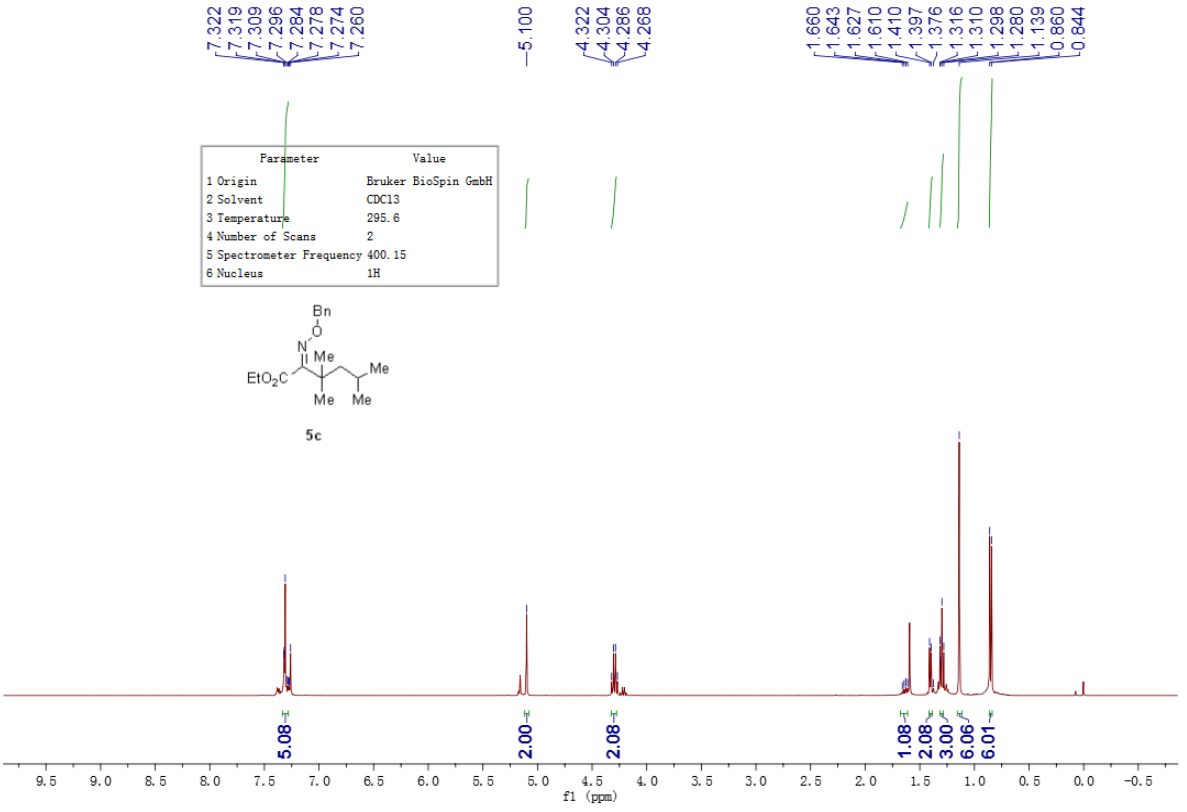

176.105
152.951
134.718
125.671
124.509
122.647
121.453
77.338
77.021
76.703
49.922
42.468
34.041
25.925
23.849
21.444
20.636
20.591
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 40
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Me
Me
Me
Me
S
N
4e
f1 (ppm)

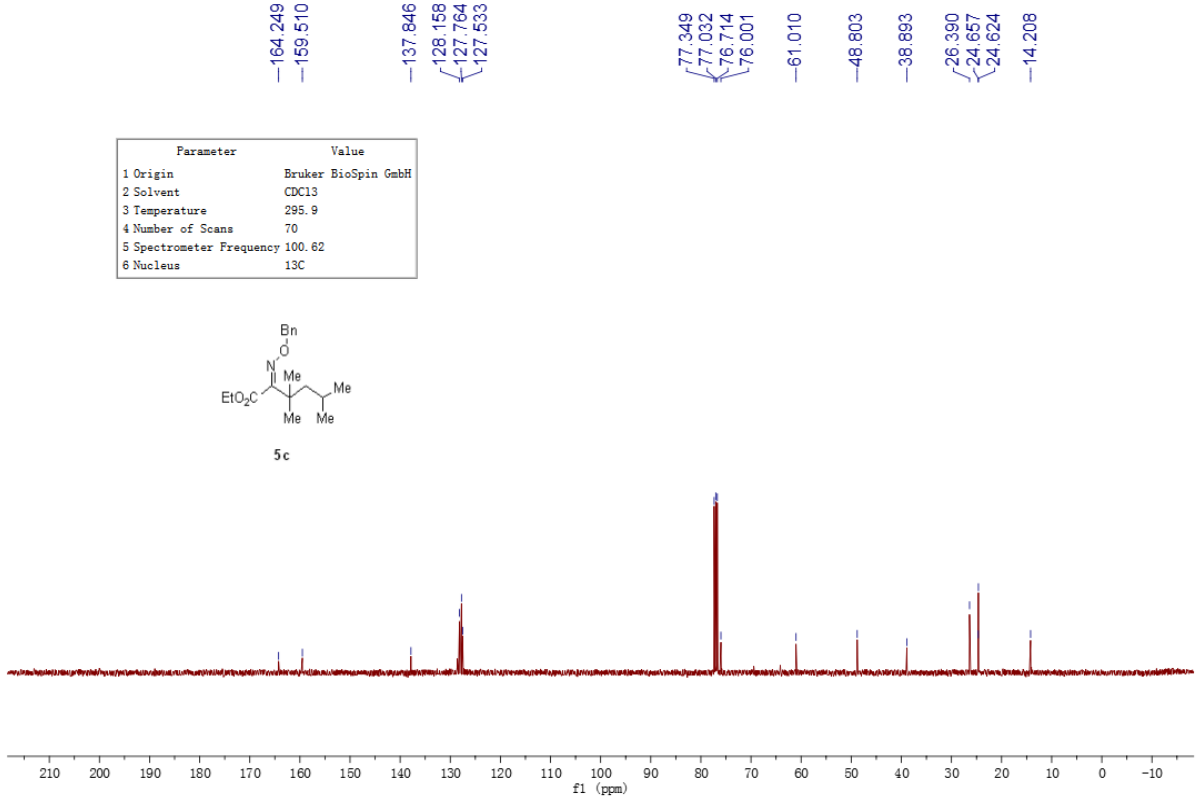

7.384
7.373
7.275
5.303
4.354
4.336
4.319
4.301
2.617
2.602
2.598
2.591
2.578
1.531
1.514
1.497
1.442
1.404
1.380
1.363
1.348
1.345
1.299
1.269
1.211
1.193
0.904
0.887
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Bn
N
O
EtO2C
Me
Me
5a
5.03
2.00
2.04
2.04
1.07
5.01
6.05
f1 (ppm)

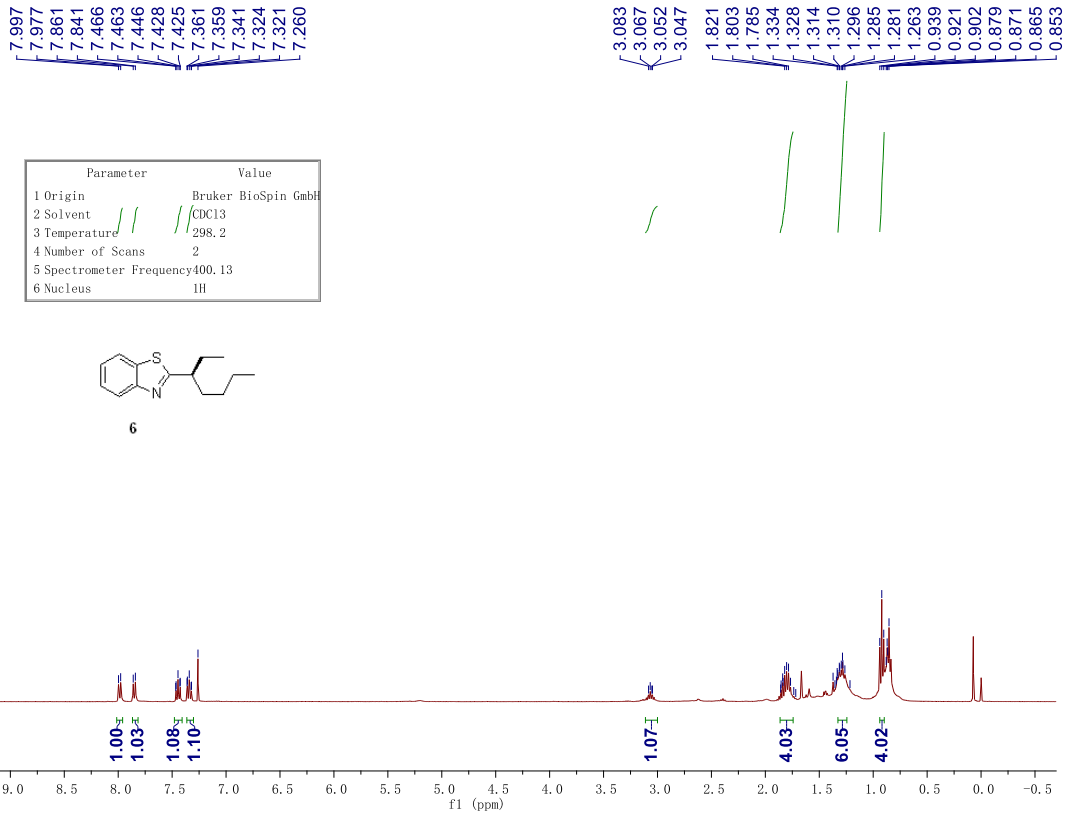

163.737
153.764
136.820
128.425
128.114
128.086
77.335
77.049
76.700
61.704
34.835
31.443
30.196
28.194
23.877
22.275
14.189
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Bn
N
O
EtO2C
Me
Me
5a
f1 (ppm)

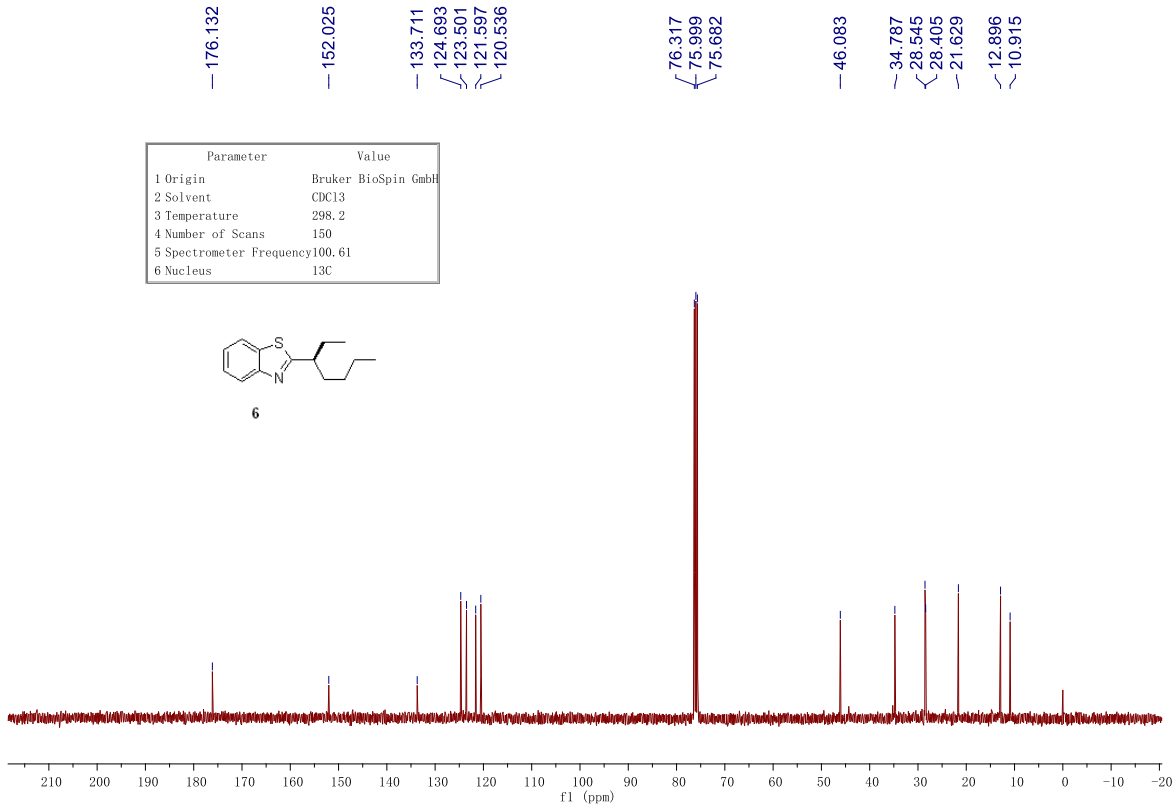

7.363
7.352
7.260
5.242
4.318
4.300
4.282
4.265
3.343
3.333
3.319
3.305
3.294
1.729
1.708
1.696
1.575
1.518
1.356
1.338
1.321
0.853
0.834
0.817
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Spectrometer spect
3 Solvent CDCl3
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Bn
N
O
EtO2C
Me
Me
Me
5b
5.03
2.00
2.00
1.05
2.05
3.06
3.05
9.08
f1 (ppm)

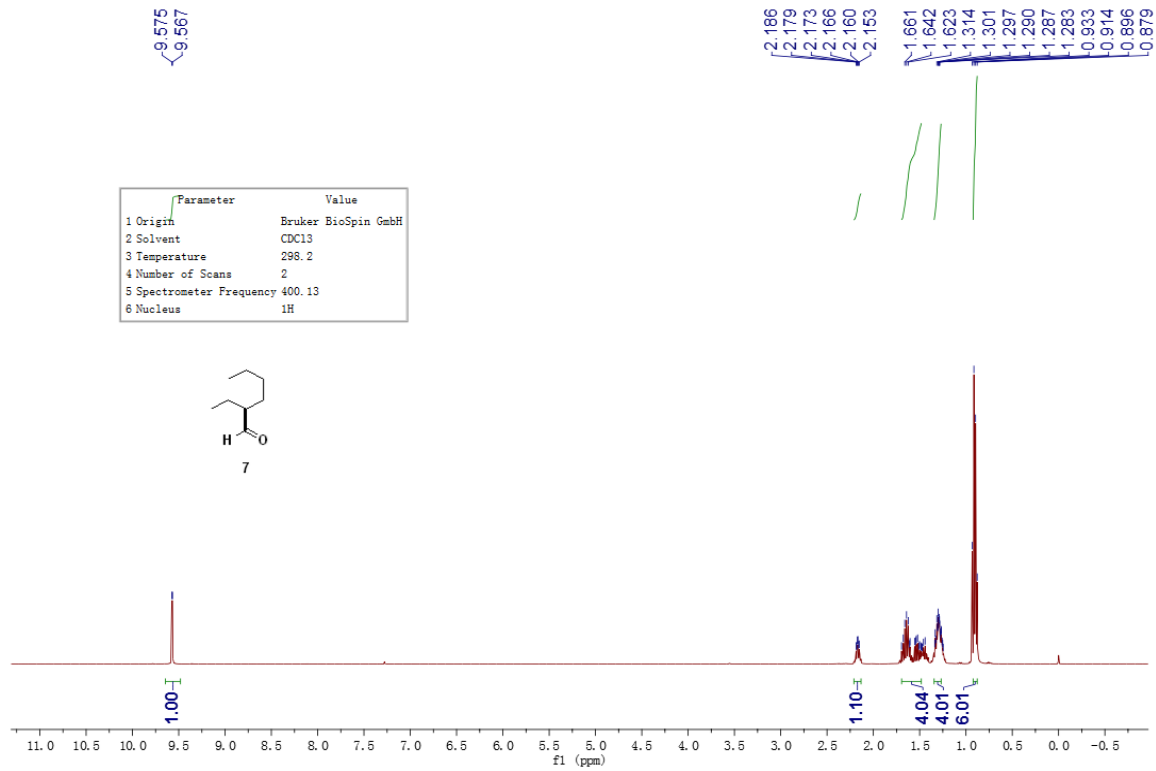

163.621
155.807
136.789
128.377
128.274
128.054
77.343
77.025
76.708
61.399
41.221
37.444
26.484
25.553
23.414
22.127
14.162
12.315
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Bn
N
O
EtO2C
Me
Me
Me
5b
f1 (ppm)

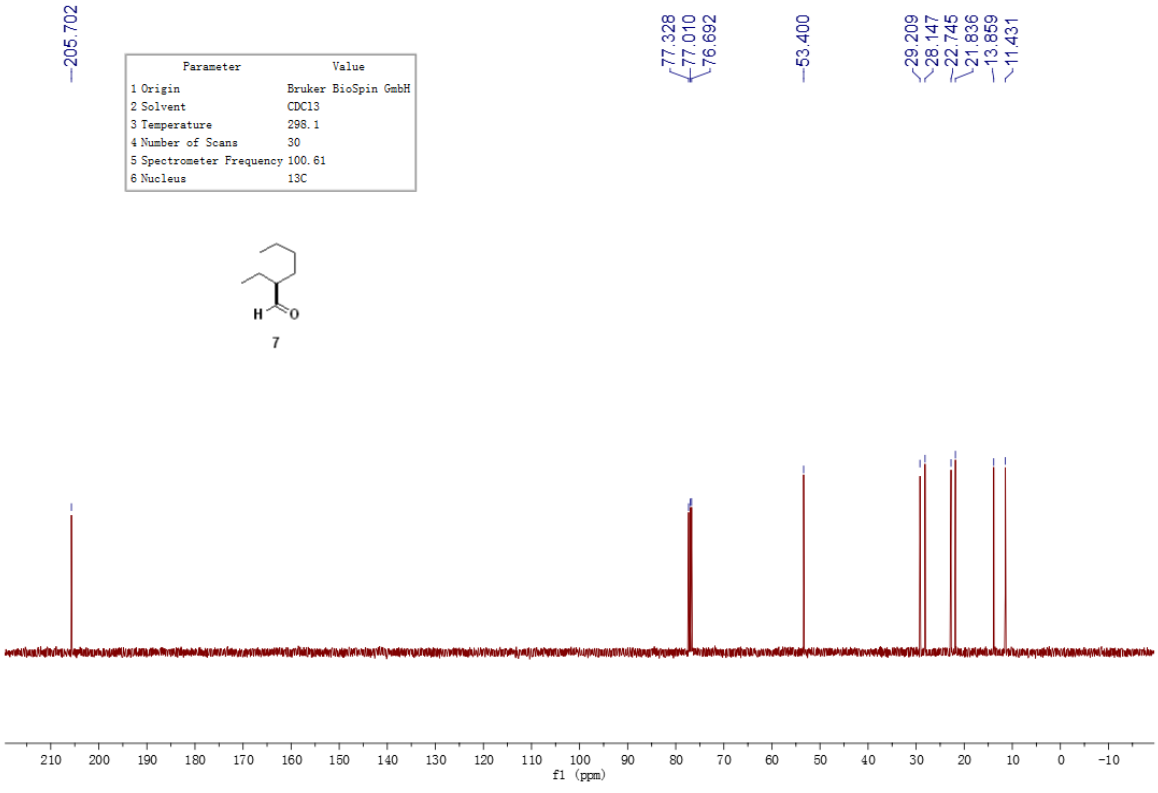

7.322
7.319
7.309
7.296
7.284
7.278
7.274
7.260
5.100
4.322
4.304
4.286
4.268
1.660
1.643
1.627
1.610
1.410
1.397
1.376
1.316
1.310
1.298
1.280
1.139
0.860
0.844
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.6
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
Bn
O
N
Me
EtO2C
Me
Me
Me
5c
5.08
2.00
2.08
1.08
2.08
3.00
6.06
6.01
f1 (ppm)

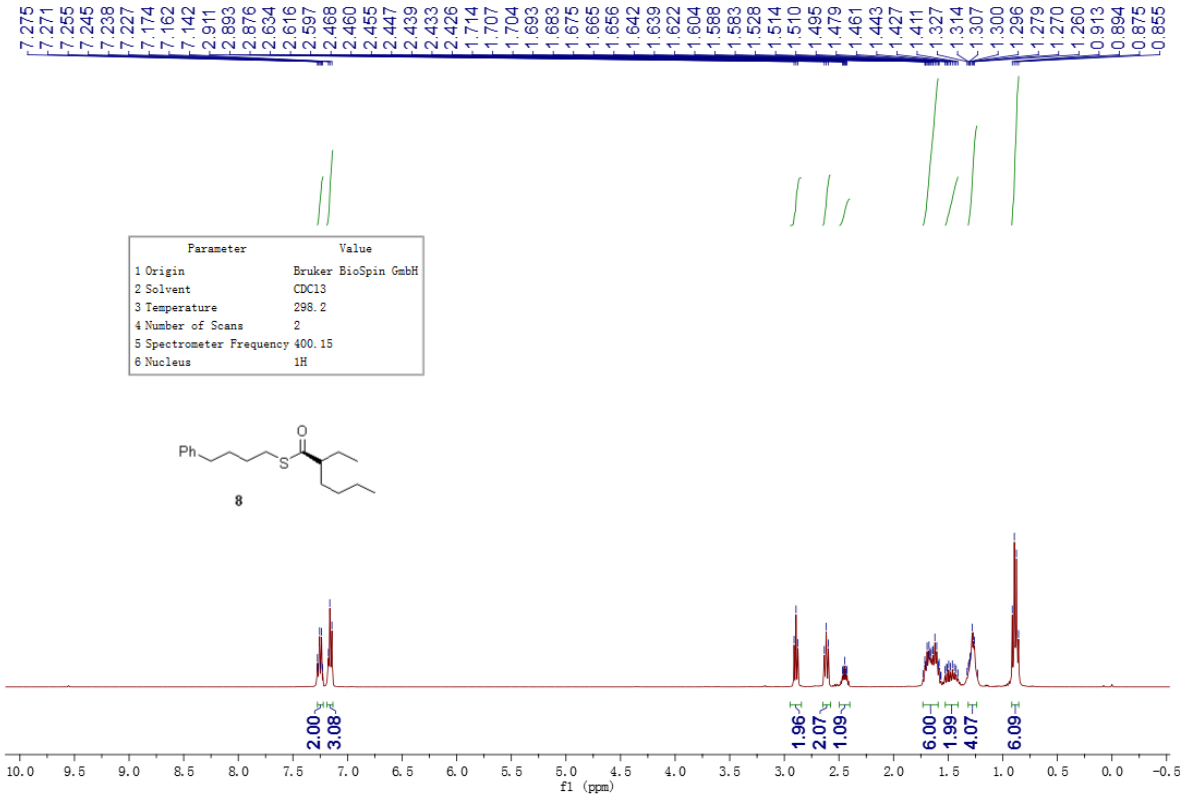

164.249
159.510
137.846
128.158
127.764
127.533
77.349
77.032
76.714
76.001
61.010
48.803
38.893
26.390
24.657
24.624
14.208
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.9
4 Number of Scans 70
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Bn
O
N
Me
EtO2C
Me
Me
Me
5c
f1 (ppm)

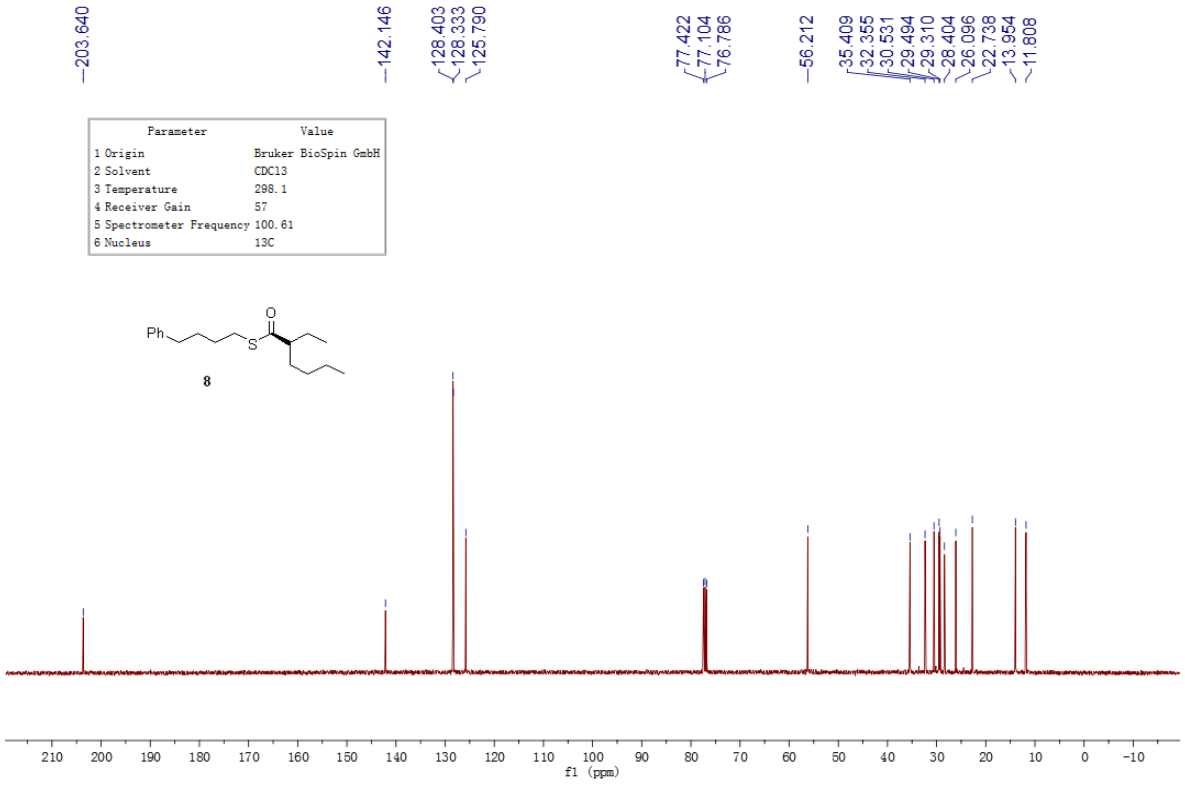

7.997
7.977
7.861
7.841
7.466
7.463
7.446
7.428
7.425
7.361
7.359
7.341
7.324
7.321
7.260
3.083
3.067
3.052
3.047
1.821
1.803
1.785
1.334
1.328
1.314
1.310
1.296
1.285
1.281
1.263
0.939
0.921
0.902
0.879
0.871
0.865
0.853
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
6
1.00
1.03
1.08
1.10
1.07
4.03
6.05
4.02
f1 (ppm)

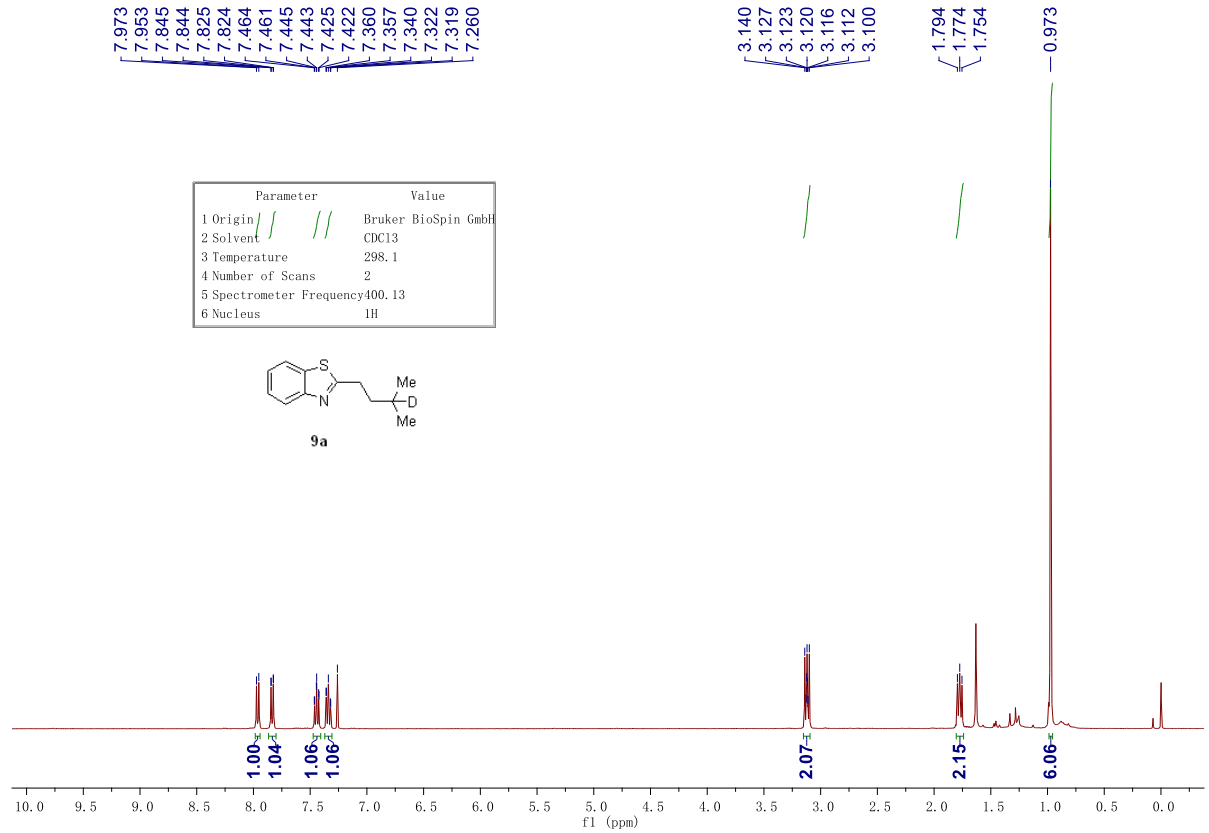

176.132
152.025
133.711
124.693
123.501
121.597
120.536
76.317
75.999
75.682
46.083
34.787
28.545
28.405
21.629
12.896
10.915
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 150
5 Spectrometer Frequency 100.61
6 Nucleus 13C
6
f1 (ppm)

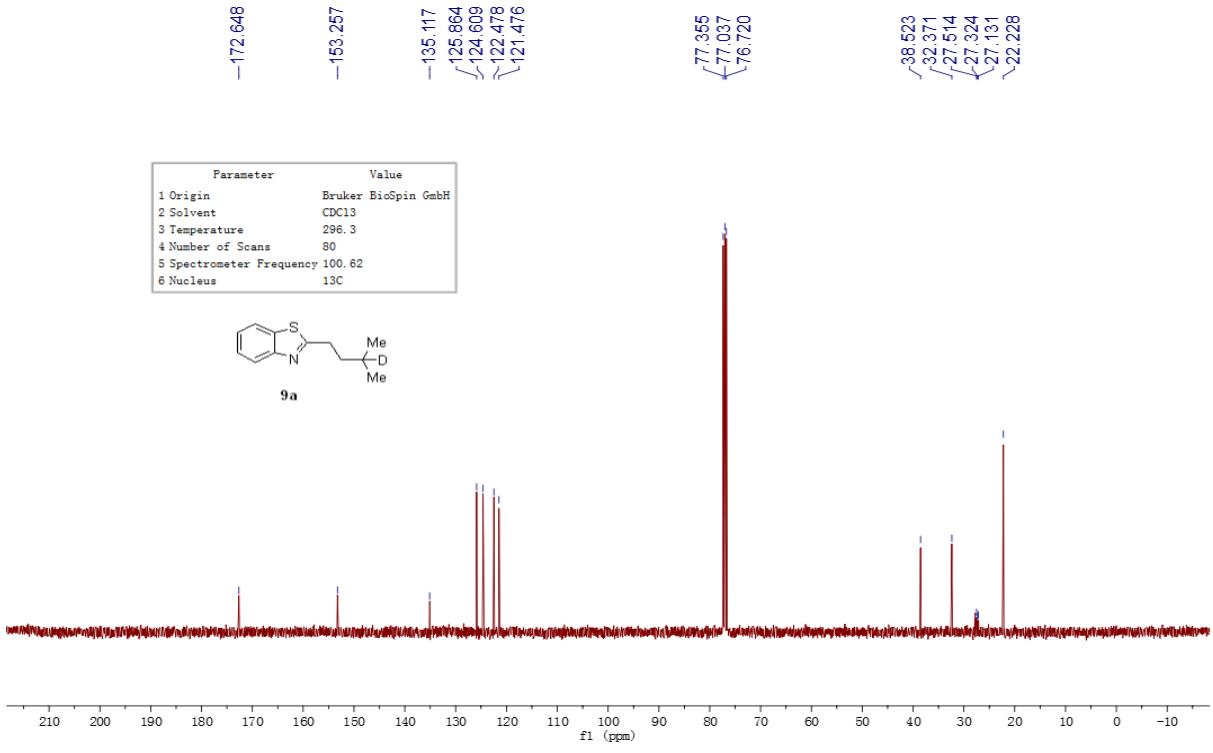

9.575
9.567
2.186
2.179
2.173
2.166
2.160
2.153
1.661
1.642
1.623
1.314
1.301
1.297
1.290
1.287
1.283
0.933
0.914
0.896
0.879
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
H
O
7
1.00
1.10
4.04
4.01
6.01
f1 (ppm)

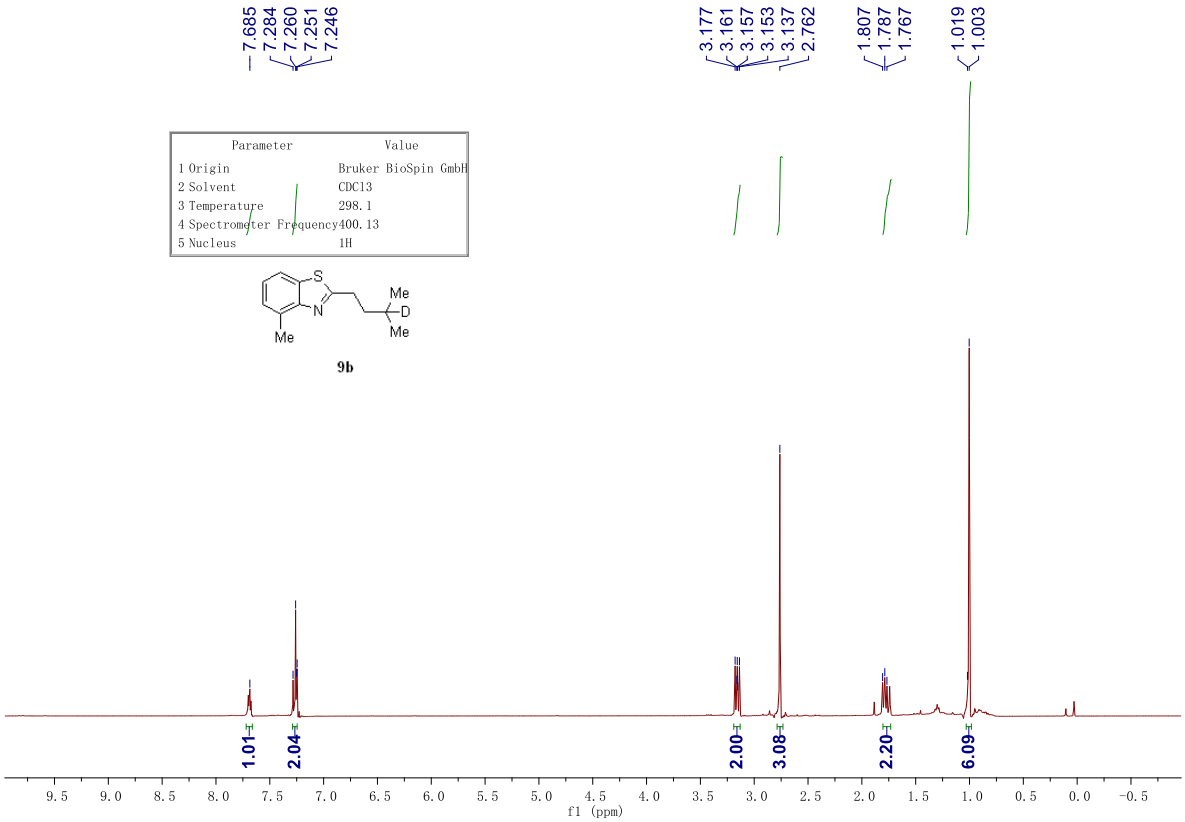

205.702
77.328
77.010
76.692
53.400
29.209
28.147
22.745
21.836
13.859
11.431
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 30
5 Spectrometer Frequency 100.61
6 Nucleus 13C
H
O
7
f1 (ppm)

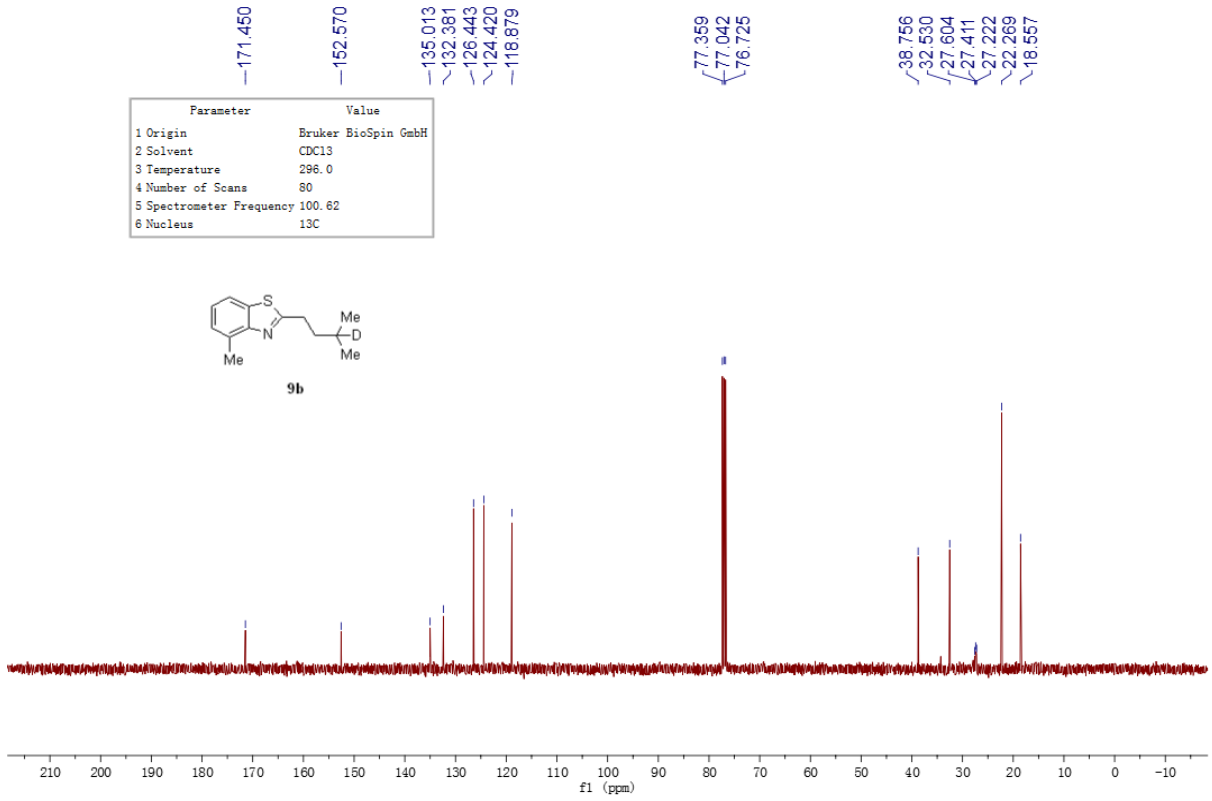

7.275
7.271
7.255
7.245
7.238
7.227
7.174
7.162
7.142
2.911
2.893
2.876
2.634
2.616
2.597
2.468
2.460
2.455
2.447
2.439
2.433
2.426
1.714
1.707
1.704
1.693
1.683
1.675
1.665
1.656
1.642
1.639
1.622
1.604
1.588
1.583
1.528
1.514
1.510
1.495
1.479
1.461
1.443
1.427
1.411
1.327
1.314
1.307
1.300
1.296
1.279
1.270
1.260
0.913
0.894
0.875
0.855
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
Ph
S
O
8
2.00
3.08
1.96
2.07
1.09
6.00
1.99
4.07
6.09
f1 (ppm)

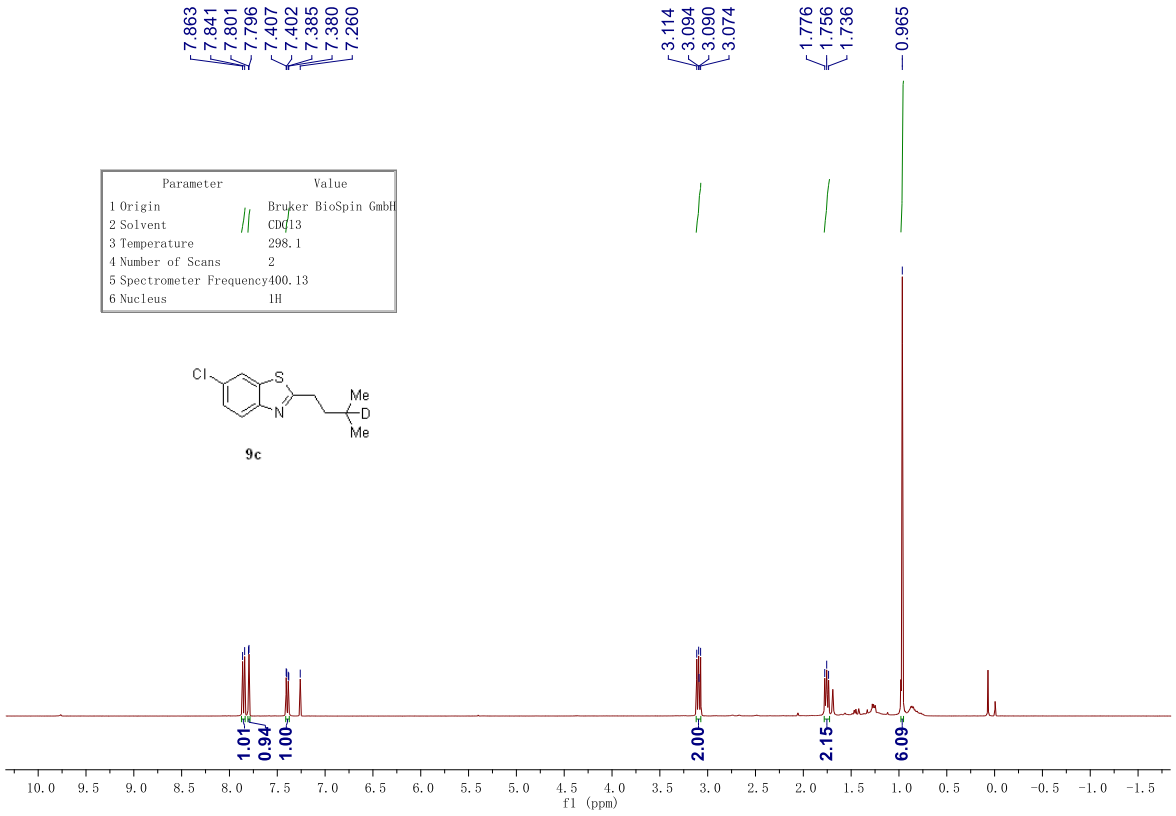

203.640
142.146
128.403
128.333
125.790
77.422
77.104
76.786
56.212
35.409
32.355
30.531
29.494
29.310
28.404
26.096
22.738
13.954
11.808
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Receiver Gain 57
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Ph
S
O
8
f1 (ppm)

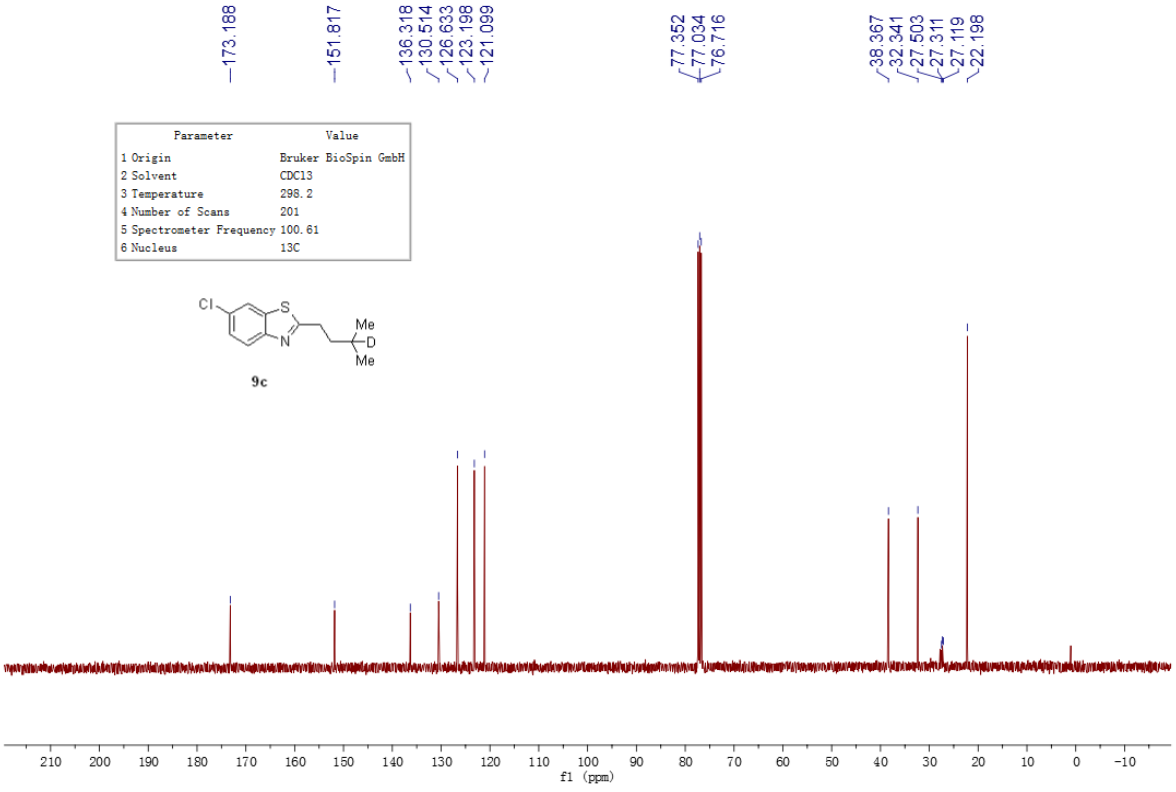

7.973
7.953
7.845
7.844
7.825
7.824
7.464
7.461
7.445
7.443
7.425
7.422
7.360
7.357
7.340
7.322
7.319
7.260
3.140
3.127
3.123
3.120
3.116
3.112
3.100
1.794
1.774
1.754
0.973
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
S
N
Me
D
Me
9a
1.00
1.04
1.06
1.06
2.07
2.15
6.06
10.0 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 0.5 0.0
f1 (ppm)

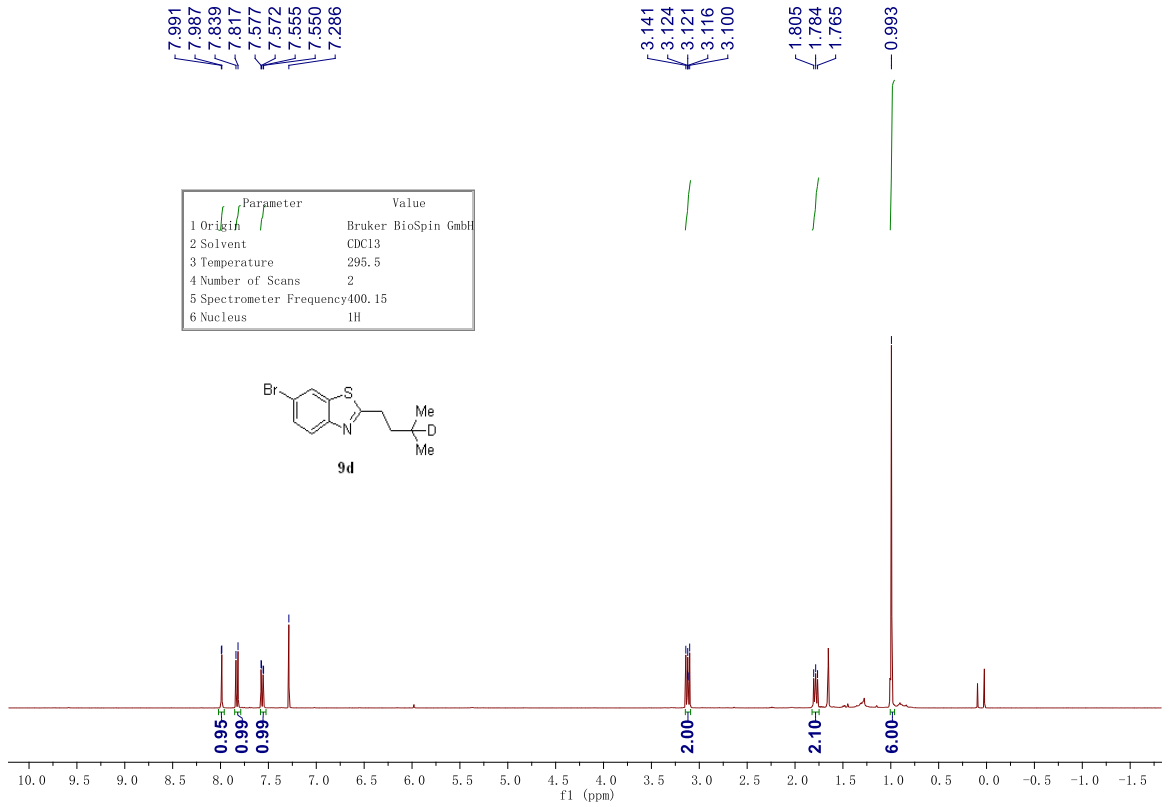

172.648
153.257
135.117
125.864
124.609
122.478
121.476
77.355
77.037
76.720
38.523
32.371
27.514
27.324
27.131
22.228
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.3
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
S
N
Me
D
Me
9a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

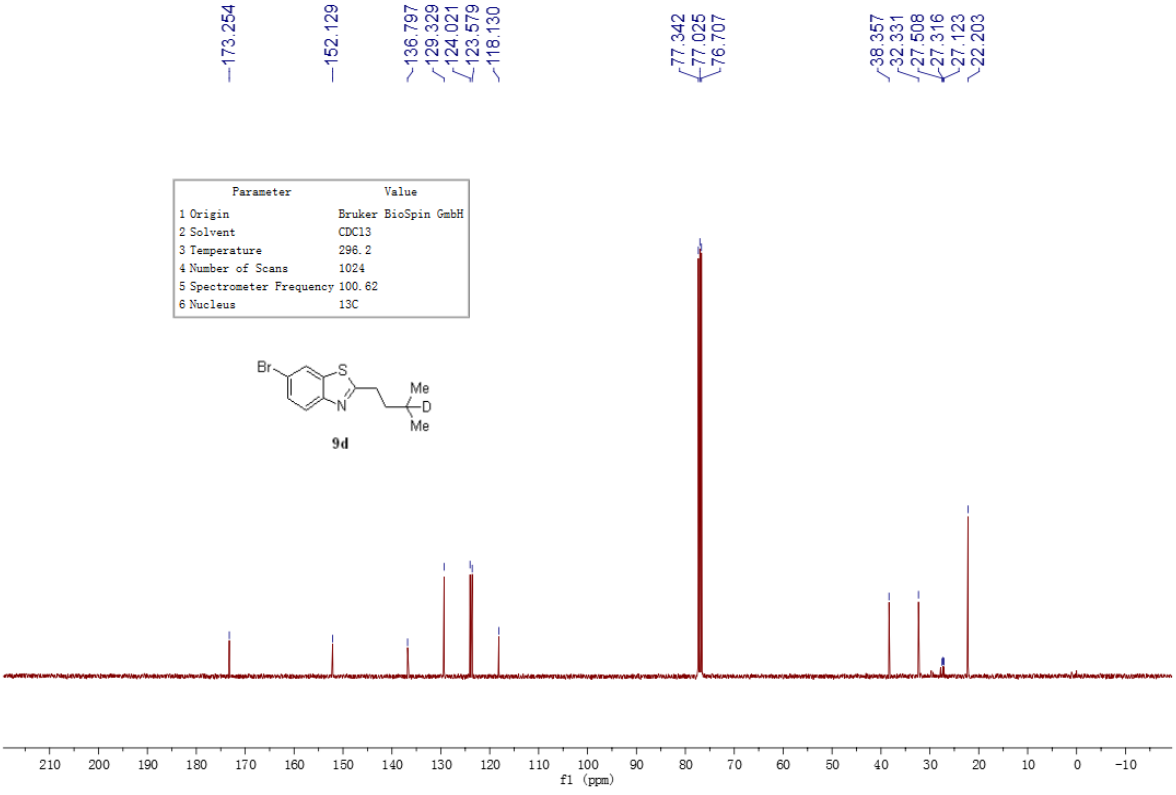

7.685
7.284
7.260
7.251
7.246
3.177
3.161
3.157
3.153
3.137
2.762
1.807
1.787
1.767
1.019
1.003
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Spectrometer Frequency 400.13
5 Nucleus 1H
S
N
Me
Me
D
Me
Me
9b
1.01
2.04
2.00
3.08
2.20
6.09
f1 (ppm)


171.450
152.570
135.013
132.381
126.443
124.420
118.879
77.359
77.042
76.725
38.756
32.530
27.604
27.411
27.222
22.269
18.557
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.0
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
S
N
Me
Me
D
Me
Me
9b
f1 (ppm)


7.863
7.841
7.801
7.796
7.407
7.402
7.385
7.380
7.260
3.114
3.094
3.090
3.074
1.776
1.756
1.736
0.965
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Cl
S
N
Me
D
Me
9c
1.01
0.94
1.00
2.00
2.15
6.09
f1 (ppm)


173.188
151.817
136.318
130.514
126.633
123.198
121.099
77.352
77.034
76.716
38.367
32.341
27.503
27.311
27.119
22.198
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 201
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Cl
S
N
Me
D
Me
9c
f1 (ppm)


7.991
7.987
7.839
7.817
7.577
7.572
7.555
7.550
7.286
3.141
3.124
3.121
3.116
3.100
1.805
1.784
1.765
0.993
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 295.5
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
Br
S
N
Me
D
Me
9d
0.95
0.99
0.99
2.00
2.10
6.00
f1 (ppm)


173.254
152.129
136.797
129.329
124.021
123.579
118.130
77.342
77.025
76.707
38.357
32.331
27.508
27.316
27.123
22.203
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 296.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Br
S
N
Me
D
Me
9d
f1 (ppm)

References:

1. J. Liu, S. Wu, J. Yu, C. Lu, Z. Wu, X. Wu, X.-S. Xue and C. Zhu, *Angew. Chem. Int. Ed.*, 2020, **59**, 8195-8202.
2. M. A. Cismesia and T. P. Yoon, *Chem. Sci.*, 2015, **6**, 5426–5434.
3. I. P. Pozdnyakov, O. V. Kel, V. F. Plyusnin, V. P. Grivin and N. M. Bazhin, *J. Phys. Chem. A*, 2008, **112**, 8316–8322.
4. C. G. Hatchard and C. A. Parker, *Proc. R. Soc. London. Ser. A. Math. Phys. Sci.* 1956, *235*, 518–536.