Design and synthesis of 3,3'-triazolyl biisoquinoline *N,N'*-dioxides via Hiyama coupling of 4-trimethylsilyl-1,2,3-triazoles

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Supporting Information

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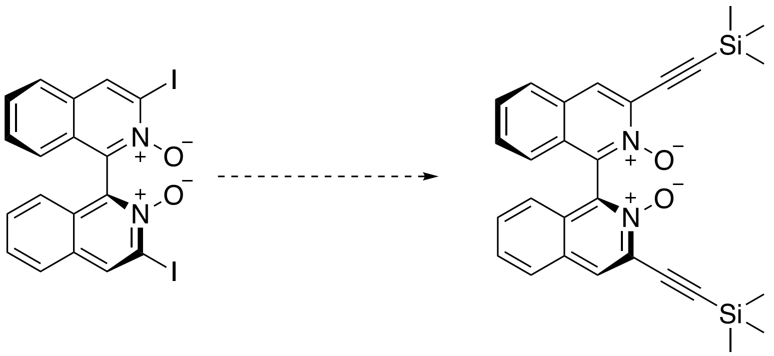
1. General Information.

All reactions were carried out in oven- or flame-dried glassware under an atmosphere of dry argon or nitrogen unless otherwise noted. Except as otherwise indicated, all reactions were magnetically stirred and monitored by analytical thin-layer chromatography using SiliCycle® Inc. pre-coated silica gel plates with F_{254} indicator. Visualization was accomplished by UV light (254 nm), with combination of potassium permanganate, *p*-anisaldehyde, and/or cerium molybdate solution as an indicator. Flash column chromatography was performed according to the method of Still [1] using silica gel 60 (mesh 230-400) supplied by SiliCycle® Inc or aluminum oxide (basic, 50-200μm, 60A). Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise stated. Commercial grade reagents and solvents were purchased from Sigma-Aldrich, Alfa-Aesar, Acros, Fisher, TCI, and VWR, and were used as received without further purification except as indicated below. THF was freshly distilled over sodium/benzophenone under an atmosphere of dry nitrogen prior to use. CH_2Cl_2 and CH_3CN were freshly distilled over CaH_2 under an atmosphere of dry nitrogen prior to use. All ^1H NMR and ^{13}C NMR spectra were obtained using a Bruker 400 Ultrashield or an Oxford AS400 Spectrometer (^1H 400 MHz, ^{13}C 100 MHz) at ambient temperature in $CDCl_3$ purchased from Cambridge Isotope Laboratories, Inc. Chemical shifts in ^1H NMR spectra are reported in parts per million (ppm) respective to tetramethylsilane (δ 0.00 ppm) unless otherwise noted. The proton spectra are reported as follows δ (multiplicity, coupling constant *J*, number of protons). Multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), and br (broad). Chemical shifts in ^{13}C NMR spectra are reported in ppm respective to $CDCl_3$ (δ 77.0 ppm). All ^{13}C NMR spectra were recorded with complete proton decoupling. Infrared (IR) spectra were recorded using a Nicolet iS5 FT-IR instrument. HRMS data were obtained at USF Mass Spec and Peptide Core Facility in Department of Chemistry at University of South Florida, and the Chouinard research group Department of Biomedical and Chemical Engineering and Sciences, Florida Institute of Technology. Optical rotations were measured using a Jasco P2000 Polarimeter at 589 nm and were reported as $[\alpha]_D^{T\,°C}$, where C is reported in g/mL. Chiral HPLC analysis was performed on Varian Polaris HPLC system with a diode array detector using analytical chiral columns (250 x 4.6 mm, L x I.D.) purchased from CHIRAL TECHNOLOGIES, INC. (CHIRALCEL® OD-H). Compounds that are not numbered in the manuscript are labeled as **S1**, **S2**, etc.

2. Experimental Procedures.

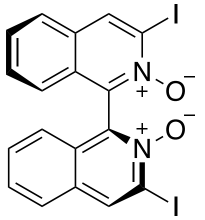
Representative Attempted Sonogashira Reaction.[2]



(*S*)-3,3’-Diiodo-1,1’-biisoquinoline *N*, *N*’-dioxide (50 mg, 0.093 mmol), CuI (4.2 mg, 0.022 mmol), and $PdCl_2(PPh_3)$ (10.4 mg, 0.015 mmol) were charged in a round bottom flask with a stir egg, then vacuumed and back-filled with argon five times. Freshly distilled Et_3N (1 mL) and THF (1 mL) were added into the reaction mixture followed by adding trimethylsilylacetylene (38 µL, 0.28 mmol), reaction was stirred at rt overnight. The reaction mixture was diluted in CH_2Cl_2, filtered through celite and concentrated *in vacuo*.

The TLC and ^1H NMR analysis of the crude reaction mixture indicated that all starting materials used completely decomposed.

Synthesis of (*S*)-3,3’-diiodo-1,1’-biisoquinoline *N, N’*-dioxide (4):



Following our previously reported procedure,[3] to a solution of LDA in THF (0.22 M, 2.0 mL) in a 25 mL round-bottom flask at -78 °C was added a solution of (*S*)-1,1’-biisoquinoline *N,N’*-dioxide (50 mg, 0.173 mmol) in THF (7 mL) dropwise by a canula. The reaction mixture was stirred for 4 hours at -78 °C, treated dropwise with a solution of iodine (264 mg, 1.04 mmol) in THF (2 mL) by a canula at -78 °C, and stirred for an additional 16 hours at -78 °C. The reaction was warmed to rt and quenched with 10% sodium thiosulfate solution (50 mL). The aqueous layer was back-extracted with CH_2Cl_2 (25 mL x 2). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The resulting crude material was purified by flash chromatography on basic aluminum oxide with 1% MeOH in CH_2Cl_2 to afford the title compound as a yellow solid (64 mg, 68%).

^1H NMR (400 MHz, $CDCl_3$) δ 8.56 (s, 2H), 7.80 (d, *J* = 8.0 Hz, 2H), 7.57 (dd, *J* = 8.0, 8.0 Hz, 2H), 7.46 (dd, *J* = 8.4, 8.4 Hz, 2H), 7.05 (d, *J* = 8.4 Hz, 2H)

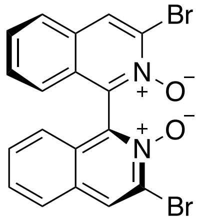
^{13}C NMR (100 MHz, $CDCl_3$) δ 137.1, 136.3, 130.4, 129.3,129.3, 128.8, 126.1, 123.3, 108.6

IR (thin film): 1345, 1220, 749, 538, 529 cm $^{-1}$

HRMS (ESI): Exact mass calculated for $C_{18}H_{11}I_2N_2O_2^+$ $[M+H]^+$ expected: 540.8905, found: 540.8900

$[\alpha]_D^{25}$ = +21.9 (c = 0.001, CH_2Cl_2)

Synthesis of (*S*)-3,3'-dibromo-1,1'-biisoquinoline *N, N'*-dioxide (5):



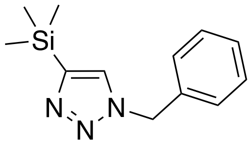
The title compound was prepared accordingly to our previously reported procedure.[3]

Synthesis of 4-TMS triazoles.

Caution: Organic azides have the potential to decompose violently upon input of external energy. While we experienced no explosions handling or producing the azides used for this work, safety precautions including adequate shielding are strongly encouraged. All organic azides used in the following procedures were synthesized according to literature procedures without modification.[4]

CuAAC Representative Procedure A: The protocol reported by Jeong and Ryu was used without further optimization. [5]

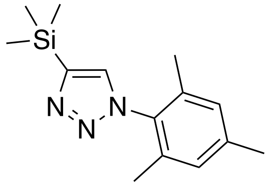
1-Benzyl-4-(trimethylsilyl)-1*H*-1,2,3-triazole (6a)



Benzyl azide (1.278 g, 9.6 mmol), trimethylsilylacetylene (1.33 mL, 9.6 mmol), $CuSO_4 \cdot 5H_2O$ (120 mg, 0.48 mmol), and sodium ascorbate (190 mg, 0.96 mmol) were suspended in a mixed solution of tert-butyl alcohol/water (0.25M, 1:1). The mixture was stirred at room temperature under an atmosphere of nitrogen for 24 hours. The mixture was poured into 50 mL water and extracted with CH_2Cl_2 (50 mL x 2). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The crude residue was purified by flash column chromatography using 20% EtOAc in hexanes as eluent to afford the product as an off-white solid (1.9 g, 86%). All spectral data were identical to the literature values. [5]

CuAAC Representative Procedure B:[6]

1-Mesityl-4-(trimethylsilyl)-1*H*-1,2,3-triazole (6b)



Caution: This procedure involves overheating of a THF solution about 25 °C above its boiling point (65-67 °C) in a tightly closed reaction vessel, thus it is suggested that the reaction be conducted behind a blast shield in an efficient fume hood.

Mesityl azide (6.4 g, 40 mmol), trimethylsilylacetylene (11.1 mL, 80 mmol), CuI (1.52 g, 8 mmol), and *N*,*N*-diisopropylethylamine (27.8 mL, 160 mmol) were suspended in THF

(1.0 M) degassed by purging with argon for 30 mins in a round bottom flask with a stir egg. The flask was sealed with a septum that was then tied up with a piece of copper wire. The reaction mixture was stirred in an oil bath at 90 °C for 17 hours. The reaction mixture was washed with 150 mL saturated aqueous ammonium chloride and extracted with EtOAc (100 mL x 2). The combined organic extracts were washed with water and brine, dried over Na_2SO_4, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography using 5% EtOAc in hexanes as eluent. The resulting product was further purified by precipitating the title compound as a white solid from hexanes (4.83 g, 47%).

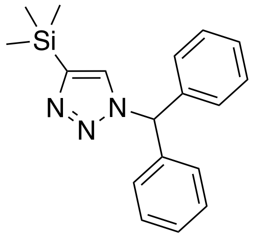
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (s, 1H), 6.98 (s, 2H), 2.35 (s, 3H) 1.93 (s, 6H), 0.39 (s, 9H)

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.2, 139.6, 135.1, 133.5, 130.6, 128.9, 21.0, 17.2, –1.1

IR (thin film): 3123, 2953, 1609, 1493, 1442, 1249, 1133, 835 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{14}H_{22}N_3Si^+$ [M+H]$^+$ expected: 260.1578, found: 260.1591.

1-(Diphenylmethyl)-4-(trimethylsilyl)-1*H*-1,2,3-triazole (6c)



The representative procedure B was followed with trimethylsilylacetylene (17.6 mL, 127.5 mmol), the corresponding azide (13.34 g, 63.8 mmol) and Et_3N instead of *N*,*N*-diisopropylethylamine. The reaction was stirred at 60°C for 3 days. The resulting crude material was purified by flash column chromatography using 10% EtOAc in hexanes then 20% EtOAc in hexanes to give the product as a white solid (5.47 g, 28% yield).

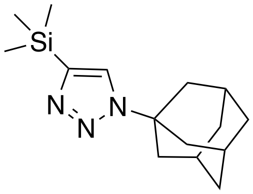
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.32 (m, 6H), 7.16 (s, 1H), 7.12-7.08 (m, 4H), 0.30 (s, 9H)

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.3, 138.5, 128.8, 128.7, 128.4, 128.1, 67.5, -1.1

IR (thin film): 2958, 1450, 1406, 1245, 1109, 836 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{18}H_{22}N_3Si^+$ $[M+H]^+$ expected: 308.1578, found: 308.1583.

1-(1-Adamantyl)-4-(trimethylsilyl)-1*H*-1,2,3-triazole (6d)



The representative procedure B was followed with trimethylsilylacetylene (11.1 mL, 80 mmol), and the corresponding azide (7.09 g, 40 mmol). The reaction mixture was stirred in an oil bath at 90 °C for 17 hours. The crude was purified by flash column chromatography using 10% EtOAc in hexanes as eluent to give product as a white solid (8.29 g, 75%).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.57 (s, 1H), 2.26 (s, 9H), 1.80 (s, 6H), 0.32 (s, 9H)

^{13}C NMR (100 MHz, $CDCl_3$) δ 145.0, 124.9, 59.0, 43.1, 36.0, 29.5, -1.0

IR (thin film): 3087, 2910, 2851, 1487, 1454, 1241, 1058, 833 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{15}H_{25}N_3NaSi^+$ $[M+Na]^+$ expected: 298.1710, found: 298.1730.

Regarding the Choice of 4-Silyl-1,2,3-triazoles.

For the following reasons, we thought it would be beneficial if we could utilize readily available 4-trimethylsilyl-5-unsubstituted-1,2,3-triazoles for Hiyama coupling.

To the best of our knowledge, there is only one Chinese patent (Preparation method of silane-modified polyether resin for MS sealant. CN110776632) with respect to the cycloaddition of ethynyltrialcoxysilanes and azides. Furthermore, ethynyltrimethoxysilane is only commercially available from two companies in China (Chemieliva Pharmaceutical Co., Ltd.) and Hong Kong (Hong Kong Chemhere Co., Ltd.). As such, we decided not to begin the project by this route because the information available to us is very limited.

The most common method for the preparation of silanols and silanol surrogates involves the reaction of a silicon electrophile with an organometallic reagent (lithium or magnesium) generated from a corresponding halide.[7] The direct synthesis of 4-halo-5-unsubstituted-1,2,3-triazoles (i.e., cycloaddition of azides with bromoethyne or iodoethyne) remains elusive[8] and thus they are commonly synthesized from 4-trimethylsilyl-5-unsubstituted-1,2,3-triazoles and *N*-halosuccinimides.[9] Likewise, the direct preparation of 4-magnesio (or lithio)-5-unsubstituted-1,2,3-triazoles (i.e., cycloaddition of azides with ethynylmagnesium halide or lithium acetylide) remains elusive, too.[10] The direct lithiation of 1-substituted-1,2,3-triazoles with ^{n}BuLi or LiTMP strongly favors 5-lithiation over 4-lithiation.[11]

Evaluation of Reaction Conditions for Hiyama Cross-coupling.

Fluoride Sources: We evaluated KF, CsF, DAST (diethylaminosulfurtrifluoride), TASF (tris(dimethylamino)sulfonium difluorotrimethylsilicate), and TBAF·4tBuOH[12] under otherwise identical reaction conditions for TBAF·3H_2O. TASF gave 8% yield but all others produced trace amount.

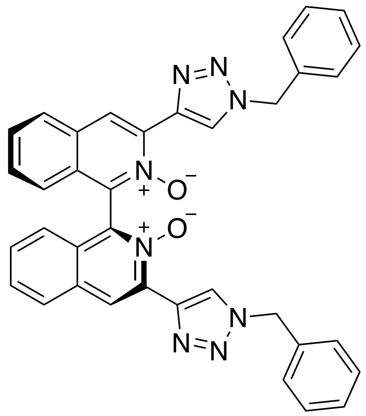
Metal Additives: We evaluated $AgBF_4$, silver(II) oxide, CuI, CuCl, and copper(II) oxide under otherwise identical reaction conditions for Ag_2O. Silver(II) oxide gave 23% yield but all others produced trace amount.

Solvents: We evaluated 1,4-dioxane, DME, toluene, CH_2Cl_2, Et_3N, and diisopropylamine but all were less effective than THF.

Reducing Sources for $PdCl_2$(dppp): We expected to observe the formation of 1,1'-dibenzyl-4,4'-bi-1*H*-1,2,3-triazole if a cross-coupling nucleophile generated from **6a** reduced a Pd(II) pre-catalyst. However, we did not find 1,1'-dibenzyl-4,4'-bi-1*H*-1,2,3-triazole in the crude reaction mixture. As such, we set up a reaction without electrophile **5** and found that the amount of 1,1'-dibenzyl-4,4'-bi-1*H*-1,2,3-triazole formed corresponded to only 30% of Pd(II) used. In reference to the fluoride-promoted R_3P-mediated reduction method reported,[13] we tested a Hiyama coupling reaction with an extra equivalent of dppp, and **3a** formed in only 27% yield compared to 41% under the standard conditions (vide infra). Pd(0)-dppp complex generated according to the reported method[14] provided mono-coupled product **S1** as a major product.

Hiyama cross-coupling of (*S*)-3,3'-dibromo-1,1'-biisoquinoline *N*, *N'*-dioxide with 4-TMS triazoles.

(*S*)-3,3'-Bis-(1-benzyl-1*H*-1,2,3-triazole-4-yl)-1,1'-biisoquinoline *N, N'*-dioxide (3a)



Hiyama Cross-coupling Representative Procedure: THF was degassed by bubbling argon for 30 mins. (*S*)-3,3'-Dibromo-1,1'-biisoquinoline *N*, *N'*-dioxide prepared accordingly to our published procedure[3] (500 mg, 1.12 mmol), the corresponding 4-TMS triazole (1.24 g, 5.38 mmol), and $PdCl_2$(dppp) (264 mg, 0.448 mmol), were charged in a flame-dried round bottom flask with a stir egg. Ag_2O (1.25 g, 5.38 mmol) was weighed in the dark and added to the flask. The reaction flask was covered with foil and vacuumed and back-filled with argon five times. THF (44 mL, 0.025 M) was added, followed by a solution of TBAF·$3H_2O$ (1.70 g, 5.38 mmol) in THF (5.4 mL, 1M). The reaction was stirred at 40 °C for overnight. The reaction was cooled to room temperature then poured into 112 mL of saturated $NaHCO_3$ solution and stirred for 15 mins. The reaction was then filtered through celite, extracted with CH_2Cl_2 (100 mL x2), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The resulting crude was purified by flash column chromatography on silica gel with 1% MeOH in CH_2Cl_2. The resulting product was further purified by precipitating the title compound as an off-white solid from a mixture of CH_2Cl_2 and toluene (309 mg, 46%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.16 (s, 2H), 8.83 (s, 2H), 8.02 (d, *J* = 8.0 Hz, 2H), 7.58 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.41 (dd, *J* = 7.6, 7.6 Hz, 2H), 7.34-7.25 (m, 10H), 7.03 (d, *J* = 8.4 Hz, 2H), 5.55 (d, *J* = 14.8 Hz, 2H), 5.51 (d, *J* = 14.8 Hz, 2H);

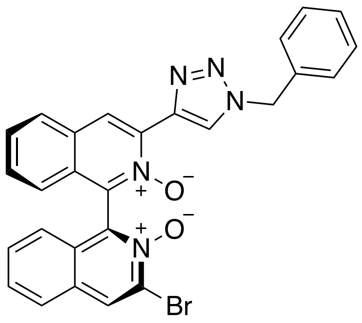
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 138.9, 138.4, 133.9, 130.0, 129.1, 129.0, 128.9, 128.8, 128.4, 127.9, 127.8, 126.6, 123.0, 122.5, 54.3;

IR (thin film): 3056, 2923, 1454, 1434, 1314, 1224, 749 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{36}H_{27}N_8O_2^+$ $[M+H]^+$ expected: 603.2251, found: 603.2252.

$[\alpha]_D^{25}$ = +61.56 (c = 0.0025, CH_2Cl_2)

(*S*)-3-(1-Benzyl-1*H*-1,2,3-triazole-4-yl)-3’-bromo-1,1’-biisoquinoline *N, N’*-dioxide (S1)



We combined fractions of the reaction mixtures containing trace amount of the title compound, which were produced during the reaction optimization study, and purified it by prep TLC with 5% MeOH in CH_2Cl_2 to obtain 9 mg of the title compound as an off-white solid for characterization purposes.

^1H NMR (400 MHz, $CDCl_3$) δ 9.14 (s,1H), 8.83 (s,1H), 8.34 (s,1H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.83 (d, *J* = 8.4 Hz, 1H), 7.62-7.55 (m, 2H), 7.46-7.42 (m, 2H), 7.34-7.28 (m, 5H), 7.08-7.03 (m, 2H), 5.58 (d, *J* = 14.8 Hz, 1H), 5.54 (d, *J* = 14.8 Hz, 1H)

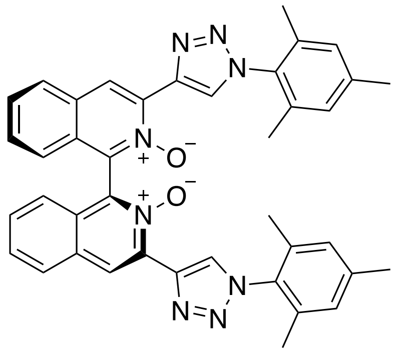
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.2, 138.9, 138.8, 138.0, 134.0, 131.2, 130.3, 129.9, 129.1, 129.1, 129.0, 128.9, 128.9, 128.6, 128.4, 127.9, 126.6, 126.4, 123.4, 123.1, 122.7, 55.4 (six signals overlap to give three signals).

IR (thin film): 3056, 2923, 1454, 1434, 1314, 1224, 1137, 749, 650 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{27}H_{19}BrN_5O_2^+$ $[M+H]^+$ expected: 524.0717, found 524.0714.

$[\alpha]_D^{25}$ = +18.2 (c = 0.001, CH_2Cl_2)

(*S*)-3,3'-Bis-(1-mesityl-1*H*-1,2,3-triazole-4-yl)-1,1'-biisoquinoline *N, N'*-dioxide (3b)



The representative procedure was followed with (*S*)-3,3'-dibromo-1,1'-biisoquinoline *N*, *N*'-dioxide (446 mg, 1.00 mmol) and the corresponding 4-TMS triazole (1.245 g, 4.8 mmol). The resulting crude material was purified by flash column chromatography on silica gel with 26% EtOAc in hexanes to afford the title compound as an off-white solid (353 mg, 54%).

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.30 (s, 2H), 9.00 (s, 2H), 8.08 (d, *J* = 8.2 Hz, 2H), 7.63 (dd, *J* = 7.6, 7.6 Hz, 2H), 7.48 (dd, *J* = 8.0, 8.0 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 6.97 (s, 4H), 2.33 (s, 6H), 1.99 (s, 12H)

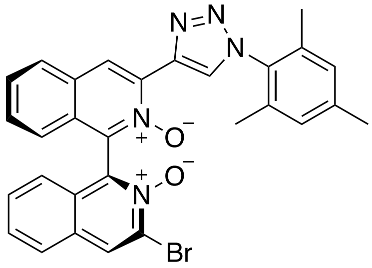
^{13}C NMR (100 MHz, CDCl$_3$) δ 140.1, 139.1, 138.8, 138.6, 135.0, 133.1, 130.0, 129.1, 129.1, 129.0, 128.7, 128.1, 127.9, 123.2, 122.7, 21.1, 17.4

IR (thin film): 2921, 2851, 1610, 1491, 1459, 1313, 1226, 1038, 728 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{40}H_{35}N_8O_2^+$ $[M+H]^+$ expected: 659.2877, found: 659.2889.

$[\alpha]_D^{25}$ = +73.3 (c = 0.001, CH_2Cl_2)

(*S*)-3-(1-Mesityl-1*H*-1,2,3-triazole-4-yl)-3'-bromo-1,1'-biisoquinoline *N, N'*-dioxide (S2)



THF was degassed by bubbling nitrogen for 15 mins. (*S*)-3,3'-Dibromo-1,1'-biisoquinoline *N*, *N*'-dioxide (223 mg, 0.5 mmol), the corresponding 4-TMS triazole (623 mg, 2.4 mmol), PdCl$_2$(dppp) (118 mg, 0.2 mmol), and silver(II) oxide (556 mg, 4.49 mmol) were charged in a flame-dried round bottom flask with a stir egg. The reaction flask was covered with foil and vacuumed and back-filled with argon five times. THF (20 mL, 0.025M) was added, followed by a solution of TBAF·3H$_2$O (757 mg, 2.4 mmol) in THF (2.4 mL, 1M). The reaction was stirred at 40 °C for 2 hours. The reaction was cooled to room temperature then poured into 50 mL of saturated $NaHCO_3$ solution and stirred for 15 mins. The reaction was then filtered through celite, extracted with CH_2Cl_2 (50 mL x2), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The resulting crude mixture was purified by flash column chromatography on silica gel with 45% EtOAc in hexanes to afford the title compound as a pale brown solid (53 mg, 19%). When Hiyama representative procedure is used (vide supra), this compound remains only in trace quantities.

^1H NMR (400 MHz, $CDCl_3$) δ 9.26 (s, 1H), 8.97 (s,1H), 8.36 (s,1H), 8.06 (d, *J* = 8.2, 1H), 7.85 (d, *J* = 8.2 Hz, 1H), 7.64-7.56 (m, 2H), 7.49-7.44 (m, 2H), 7.13 (d, *J* = 8.5 Hz, 1H), 7.08 (d, *J* = 8.5 Hz, 1H), 6.86 (s, 2H), 2.34 (s, 3H), 2.00 (s, 1H)

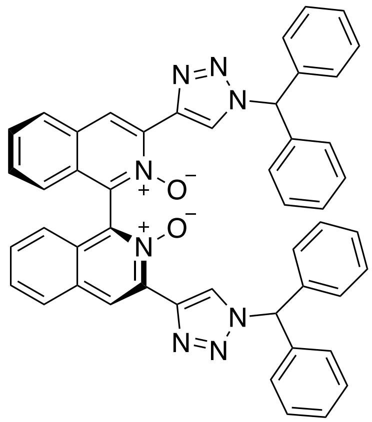
^{13}C NMR (100 MHz, $CDCl_3$) δ 140.0, 139.0, 138.9, 138.7, 138.0, 135.0, 133.1, 131.1, 130.2, 129.9, 129.1, 128.9, 128.9, 128.9, 128.6, 127.9, 127.8, 126.4, 123.4, 123.0, 122.8, 21.1, 17.3 (six signals overlap to give three signals).

IR (thin film): 3055, 1490, 1412, 1317, 1225, 1140, 734, 621 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{30}H_{26}BrN_5O_2^+$ $[M+H]^+$ expected: 552.1030, found: 552.1037.

$[\alpha]_D^{25}$ = +39.4 (c = 0.002, CH_2Cl_2)

(*S*)-3,3'-Bis-(1-diphenylmethyl-1*H*-1,2,3-triazole-4-yl)-1,1'-biisoquinoline *N, N'*-dioxide (3c)



The representative procedure was followed with (*S*)-3,3'-dibromo-1,1'-biisoquinoline *N*, *N*'-dioxide (446 mg, 1.00 mmol) and the corresponding 4-TMS triazole (1.476 g, 4.8 mmol). The resulting crude material was purified by flash column chromatography on silica gel with 28% EtOAc in hexanes to afford the title compound as a light orange solid (234 mg, 31%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.20 (s, 2H), 8.80 (s, 2H), 8.02 (d, *J* = 8.2 Hz, 2H), 7.58 (dd, *J* = 7.4, 7.4 Hz, 2H), 7.41 (dd, *J* = 7.7, 7.7 Hz, 2H), 7.34-7.20 (m, 12H), 7.19 (s, 2H), 7.13-7.11 (m, 8H), 7.01 (d, *J* = 8.4 Hz, 2H)

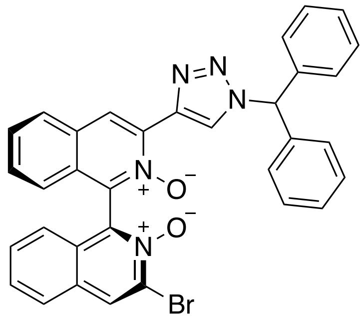
^{13}C NMR (100 MHz, $CDCl_3$) δ139.1, 138.8, 138.4, 137.7, 137.6, 130.0, 129.1, 128.9, 128.9, 128.6, 128.5, 128.2, 128.1, 128.0, 127.9, 126.6, 123.1, 122.8, 68.5 (two signals overlap to give one signal).

IR (thin film): 3028, 1491, 1453, 1314, 1226, 1143, 749 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{48}H_{35}N_8O_2^+$ $[M+H]^+$ expected: 755.2877, found: 755.2889.

$[\alpha]_D^{25}$ = +65.4 (c = 0.001, CH_2Cl_2)

(*S*)-3-(1-Diphenylmethyl-1*H*-1,2,3-triazole-4-yl)-3'-bromo-1,1'-biisoquinoline *N, N'*-dioxide (S3)



THF was degassed by bubbling nitrogen for 15 mins. (*S*)-3,3'-Dibromo-1,1'-biisoquinoline *N*, *N*'-dioxide (50 mg, 0.112 mmol), the corresponding 4-TMS triazole (165 mg, 0.537 mmol), $PdCl_2$(dppp) (27 mg, 0.046 mmol), and silver(II) oxide (125 g, 1.0 mmol) were charged in a flame-dried round bottom flask with a stir egg. The reaction flask was covered with foil and vacuumed and back-filled with argon five times. THF (4 mL, 0.025M) was added, followed by a solution of TBAF·$3H_2O$ (170 mg, 0.539 mmol) in THF (0.5 mL, 1M). The reaction was stirred at 40 °C for 2 hours. The reaction was cooled to room temperature then poured into 50 mL of saturated $NaHCO_3$ solution and stirred for 15 mins. The reaction was then filtered through celite, extracted with CH_2Cl_2 (50 mL x2), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The resulting crude was purified by flash column chromatography on silica gel with 45% EtOAc in hexanes to afford the title compound as a pale pink solid (27 mg, 40%). When Hiyama representative procedure is used, this compound remains only in trace quantities (vide supra).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.19 (s, 1H), 8.79 (s, 1H), 8.33 (s, 1H), 8.03 (d, *J* = 8.2 Hz, 1H), 7.83 (d, *J* = 8.2 Hz, 1H), 7.65-7.52 (m, 2H), 7.49-7.38 (m, 2H), 7.34-7.30 (m, 6H), 7.20 (s,1H), 7.16-7.11 (m, 4H), 7.07 (d, *J* = 8.4 Hz, 1H), 7.01 (d, *J* = 8.4 Hz, 1H)

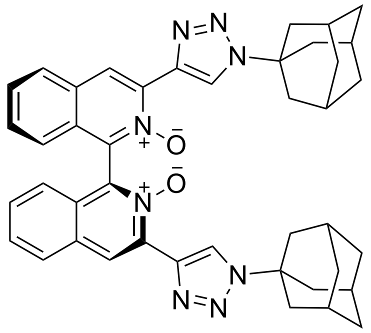
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 138.9, 138.8, 137.9, 137.7, 137.6,.131.2, 130.3, 129.9, 129.1, 129.0, 128.9, 128.8, 128.6, 128.6, 128.3, 128.1, 127.9, 127.9, 126.6, 126.4, 123.4, 123.0, 122.9, 68.5

IR (thin film): 3059, 1490, 1455, 1317, 1234, 1139, 735, 702 cm^{-1}

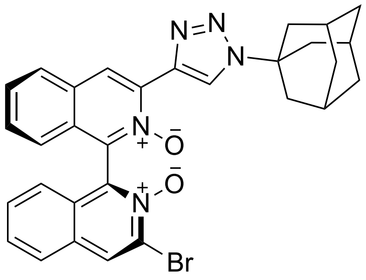
HRMS (ESI): Exact mass calculated for $C_{33}H_{23}BrN_5O_2^+$ [M+H]$^+$ expected: 600.1030, found: 600.1039.

$[\alpha]_D^{25}$ = +24.0 (c = 0.001, CH_2Cl_2)

(*S*)-3,3'-Bis-[1-(1-adamantyl)-1*H*-1,2,3-triazole-4-yl]-1,1'-biisoquinoline *N, N'*-dioxide (3d) and (*S*)-3-[1-(1-adamantyl)-1*H*-1,2,3-triazole-4-yl]-3'-bromo-1,1'-biisoquinoline *N, N'*-dioxide (S4)



&



The representative procedure was followed with (*S*)-3,3'-dibromo-1,1'-biisoquinoline *N*, *N*'-dioxide (446 mg, 1.00 mmol) and the corresponding 4-TMS triazole (1.322 g, 4.8 mmol). The resulting crude material was purified by flash column chromatography on silica gel with 32% EtOAc in hexanes to afford **3d** compound as off-white solid (162 mg, 23%), and then with 45% EtOAc in hexanes to afford **S4** as off-white solid (106 mg, 19%).

(*S*)-3,3'-Bis-[1-(1-adamantyl)-1*H*-1,2,3-triazole-4-yl]-1,1'-biisoquinoline *N, N'*-dioxide (3d)

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.19 (s, 2H), 9.02 (s, 2H), 8.05 (d, *J* = 8.2 Hz, 2H), 7.60 (dd, *J* = 7.5, 7.5 Hz, 2H), 7.43 (dd, *J* = 7.5, 7.5 Hz, 2H), 7.07 (d, *J* = 8.5 Hz, 2H), 2.26 (br s, 6H), 2.24 (br s, 3H), 1.80-1.72 (br m, 6H)

^{13}C NMR (100 MHz, $CDCl_3$) δ 139.5, 138.6, 138.1, 129.8, 129.2, 128.9, 127.9, 127.9, 123.7, 123.2, 122.4, 60.0, 42.9, 35.9, 29.5

IR (thin film): 2905, 2850, 1452, 1417, 1311, 1223, 1208, 1073, 748 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{42}H_{43}N_8O_2^+$ [M+H]$^+$ expected: 691.3503, found: 691.3515.

$[\alpha]_D^{25}$ = +68.24 (c = 0.0025, CH_2Cl_2)

(*S*)-3-[1-(1-Adamantyl)-1*H*-1,2,3-triazole-4-yl]-3'-bromo-1,1'-biisoquinoline *N, N'*-dioxide (S4)

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.15 (s,1H), 8.98 (s,1H), 8.36 (s, 1H), 8.03 (d, *J* = 8.2 Hz, 1H), 7.85 (d, *J* = 8.2 Hz, 1H), 7.62-7.56 (m, 2H), 7.48-7.41 (m, 2H), 7.11-7.04 (m, 2H), 2.27 (br s, 6H), 2.25 (br s, 3H), 1.81-1.73 (br m, 6H)

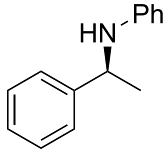
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4, 139.1, 138.0, 138.0, 131.2, 130.2, 129.7, 129.1, 129.1, 128.9, 128.8, 128.7, 127.8, 127.8, 126.4, 123.6, 123.5, 123.0, 122.5, 60.0, 42.9, 35.9, 29.5 (two signals overlap to give one signal).

IR (thin film): 2913, 2850, 1453, 1420, 1314, 1222, 1138, 749, 625 cm^{-1}

HRMS (ESI): Exact mass calculated for $C_{30}H_{27}BrN_5O_2^+$ [M+H]$^+$ expected: 568.1343, found: 568.1345

$[\alpha]_D^{25}$ = +37.1 (c = 0.001, CH_2Cl_2)

(*S*)-*N*-[1-(Phenyl)-ethyl]aniline (8):



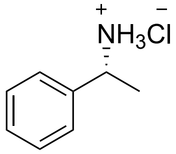
Representative Procedure for the Reduction of *N*-(1-Phenylethylidene)benzene-amine (7): Ketimine **7** was synthesized according to the literature procedure.[15] In a flame-dried test tube with stir egg, the ketimine (49 mg, 0.25 mmol) and catalyst **3c** (19 mg, 0.025 mmol) were dissolved in 1 mL of a 3:1 mixture of MeCN:CH_2Cl_2 and cooled to -50 °C. A solution of trichlorosilane in CH_2Cl_2 (131 µL, 2.86 M) was slowly added to the reaction mixture at -50 °C. The reaction was warmed to -40 °C and stirred 16 hours at that temperature. The reaction was poured into 5 mL of saturated $NaHCO_3$ solution cooled to 0°C and stirred for 20 min. at rt. It was then filtered through celite and extracted twice with 2 mL of CH_2Cl_2. The combined organic layers were dried over Na_2SO_4, filtered, and condensed *in vacuo*. ^{1}H NMR yield was determined using 0.5 mmol of 1,1,2,2-tetracholoroethane as an internal standard. A fraction of the crude reaction mixture was purified by prep TLC for characterization purposes. All spectral data were consistent with the literature values.[16]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.6 Hz, 2H), 7.32 (dd, *J* =7.2, 8.0 Hz, 2H), 7.22 (t, *J* = 7.2 Hz, 1H), 7.09 (dd, *J* = 7.6, 7.6 Hz, 2H), 6.64 (t, *J* = 7.6 Hz, 1H), 6.51 (d, *J* = 7.6 Hz, 2H), 4.48 (q, *J* = 6.8 Hz, 1H), 4.03 (br, 1H), 1.52 (d, *J* = 6.8 Hz, 3H)

ee = 56 %; $[\alpha]^{20}_{D}$ = +16.0 (c = 0.001, CH_2Cl_2); The enantiomeric excess and the absolute stereochemistry were determined by HPLC analysis:[16] t_R (major) 17.85 min; t_R

(minor) 21.67 min, (Daicel Chiralcel® OD-H with an OD-H guard column, hexane/2-propanol = 98:2, 0.5mL/min).

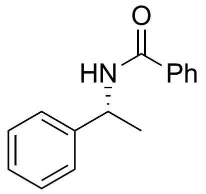
(*R*)-1-Phenylethanaminium Chloride (S5):



Representative Procedure for the Reduction of 1-Phenylethaniminium Chloride (9): Ketimine salt **9** was synthesized according to literature procedure.[17] A flame-dried Schlenk tube was charged with the ketimine salt (156 mg, 1.0 mmol) and a stir bar in a dry box, then taken out of a dry box. To this, catalyst **3a** (60 mg, 0.1 mmol) and freshly distilled CH_2Cl_2 (4 mL, 0.25 M) were added under nitrogen atmosphere. The resulting suspension was cooled to -60 °C with stirring, and then treated with a solution of trichlorosilane in CH_2Cl_2 (528 μL, 2.86 M). The reaction mixture was stirred at -40 °C for 16 hours. The reaction was quenched with 4 mL of 3N NaOH and stirred for 20 min, making sure that the pH of the aqueous layer was ≥12. The aqueous layer was extracted 5 times with 2 mL CH_2Cl_2. The organic layers were combined and dried over Na_2SO_4, filtered, and condensed *in vacuo* to give 1-phenyl-*N*-(1-phenylethylidene)-ethanamine.[18] The amount of 1-phenyl-*N*-(1-phenylethylidene)-ethanamine in the crude reaction mixture was determined by ^{1}H NMR using 0.5 mmol 1,1,2,2-tetracholoroethane as an internal standard (23% NMR yield). 1-Phenyl-*N*-(1-phenylethylidene)-ethanamine in the crude reaction mixture was hydrolyzed on silica during the flash column chromatography (100% CH_2Cl_2 to 5% methanol in CH_2Cl_2) to afford phenylethylamine. To prevent product loss, 500 μL of 1 M HCl in ether were added before being condensed *in vacuo* (1.0 mmHg), affording the title compound as a white solid (32 mg, 20% yield). All spectral data were consistent with the reported values.[17]

^{1}H NMR (400 MHz, $CDCl_3$) 8.71 (br s, 3H), 7.48-7.34 (m, 5H), 4.36 (d, *J* = 8.0 Hz, 1H), 1.66 (d, *J* = 4.0 Hz, 3H).

(*R*)-*N*-(1-Phenylethyl)benzamide (S6):



Procedure for the Benzoylation of 1-Phenylethanaminium Chloride: (*R*)-1-Phenylethanaminium chloride was converted to the title compound according to the literature procedure[19] for HPLC analysis. A solution of triethylamine in THF (115 μL, 0.6 M) was added to (*R*)-1-phenylethanaminium chloride (6 mg, 0.04 mmol). To this, a solution of benzoyl chloride in THF (115 μL, 0.4 M) was added and the resulting reaction mixture was stirred 1 hour at rt. The reaction was quenched with 4 mL of saturated NH_4Cl solution and extracted with 2 mL of CH_2Cl_2. A fraction of the crude reaction mixture was purified by prep TLC for characterization purposes. All spectral data were consistent with the literature values.[20]

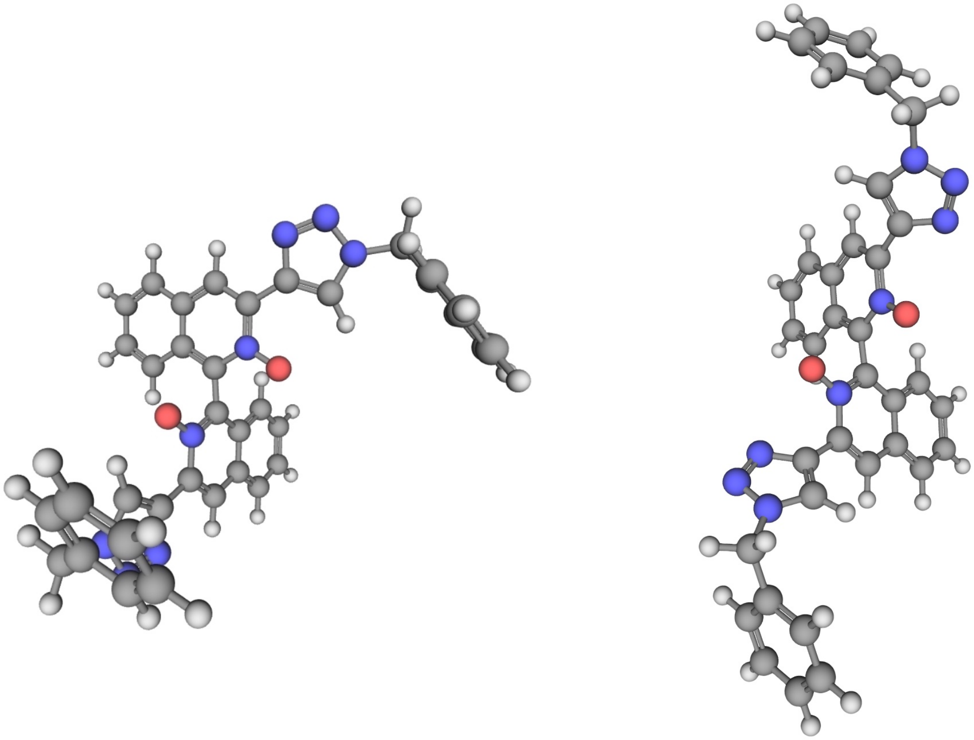
^{1}H NMR (400 MHz, $CDCl_3$) 7.78-7.76 (m, 2H), 7.52-7.48 (m, 1H), 7.45-7.35 (m, 6H), 7.30-7.25 (m, 1H), 6.32 (br s, 1H), 5.34 (q, *J* = 7.2 Hz), 1.62 (d, *J* = 7.2 Hz, 3H).

ee = 54 %; $[\alpha]^{20}_{D}$ = +4.8 (c = 0.001, CH_2Cl_2); The enantiomeric excess and the absolute stereochemistry were determined by HPLC analysis:[20] t_R (major) 27.53 min;

(minor) 39.36 min, (Daicel Chiralcel® OD-H with an OD-H guard column, hexane/2-propanol = 90:10, 0.5mL/min).

3. Computational Procedures.

All calculations reported here were performed with DFT methods, using the Q-Chem 4 quantum chemistry code.[21] These preliminary optimizations were all conducted using the PBEh-3c composite procedure,[22] with solvent effects included using the C-PCM method with the dielectric constant of CH_2Cl_2 ($\varepsilon = 9.08$).[23] Several geometry optimization calculations have been performed for each isomer to locate its minima on the potential energy surface. Molecular symmetry has been turned off for these calculations due to the necessity of the C-PCM method. As such, the C2 symmetry expected from the X-Ray data is not completely verified in the optimized structures, but respected approximately. The initial structures for the geometry optimizations have been obtained via a molecular mechanics based conformational search algorithm, developed in-house. Given the fact that the molecules in this project and the nature of their interaction are well within the limits of recent validation studies of the method that we used, we expect geometries to be converged within 0.05 Å accuracy,[24] and energies to be converged within 2 kcal/mol accuracy.[25] The structures of the minima for the complex and the free Lewis bases are also provided in cartesian coordinates (xyz) as additional supplementary text files.



Anti-**3a** *Syn*-**3a**

Figure S1. *Anti*-**3a** and Syn-**3a**. 3-Deminsional structures of the free Lewis base (**3a**) calculated with PBEh-3c/PCM(DCM).

4. Crystallographic Experimental Section.

Representative Procedure for Crystallographic Analysis:

3,3'-Bis-(1-benzyl-1*H*-1,2,3-triazole-4-yl)-1,1'-biisoquinoline *N, N'*-dioxide (3a)

The data crystal of **3a** was glued onto the end of a thin glass fiber. X-ray intensity data were measured with a Bruker SMART APEX2 CCD-based diffractometer using Mo Kα radiation (λ = 0.71073 Å).[26] The raw data frames were integrated with the SAINT+ program by using a narrow-frame integration algorithm.[26] Corrections for Lorentz and polarization effects were also applied with SAINT+. An empirical absorption correction based on the multiple measurement of equivalent reflections was applied using the program SADABS. The structure was solved by a combination of direct methods and difference Fourier syntheses, and refined by full-matrix least-squares on F^2, by using the SHELXTL software package.[27] All non-hydrogen atoms were refined with anisotropic displacement parameters unless otherwise stated. Hydrogen atoms were placed in geometrically idealized positions and included as standard riding atoms during the least-squares refinements. Crystal data, data collection parameters, and results of the analyses are listed in Table S1.

Yellow single crystals of **3a** suitable for x-ray diffraction analyses obtained by slow diffusion of Et_2O into a solution of **3a** in CH_2Cl_2 crystallized in the orthorhombic crystal system. The systematic absences in the intensity data were consistent with the unique space group *Fdd*2. With Z =8, the molecule has crystallographic 2-fold symmetry.

Table S1. Crystallographic Data for 3,3’-Bis-(1-benzyl-1*H*-1,2,3-triazole-4-yl)-1,1’-biisoquinoline *N, N’*-dioxide (3a)

	3a
Empirical formula	$C_{36}H_{26}N_8O_2$
Formula weight	602.65
Crystal system	Orthorhombic
Lattice parameters	
a (Å)	13.0942(5)
b (Å)	36.6120(14)
c (Å)	12.3660(5)
α (°)	90
β (°)	90
γ (°)	90
V (Å^3)	5928.3(4)
Space group	*Fdd*2 (# 43)
Z value	8
ρ_{calc} (g / cm^3)	1.350
μ (Mo Kα) (mm^{-1})	0.088
Temperature (K)	273
$2\Theta_{max}$ (°)	58.0
No. Obs. ($I > 2\sigma(I)$)	3785
No. Parameters	208
Goodness of fit	1.047
Max. shift in cycle	0.000
Residuals*:R1; wR2	0.0387; 0.1028
Absorption Correction, Max/min	Multi-scan 0.7013/0.7466
Largest peak in Final Diff. Map (e$^-$ / Å^3)	0.299

*$R = \Sigma_{hkl}(||F_{obs}|-|F_{calc}||)/\Sigma_{hkl}|F_{obs}|$; $R_w = [\Sigma_{hkl}w(|F_{obs}|-|F_{calc}|)^2/\Sigma_{hkl}wF_{obs}^2]^{1/2}$, $w = 1/\sigma^2(F_{obs})$; GOF = $[\Sigma_{hkl}w(|F_{obs}|-|F_{calc}|)^2/(n_{data} - n_{vari})]^{1/2}$.

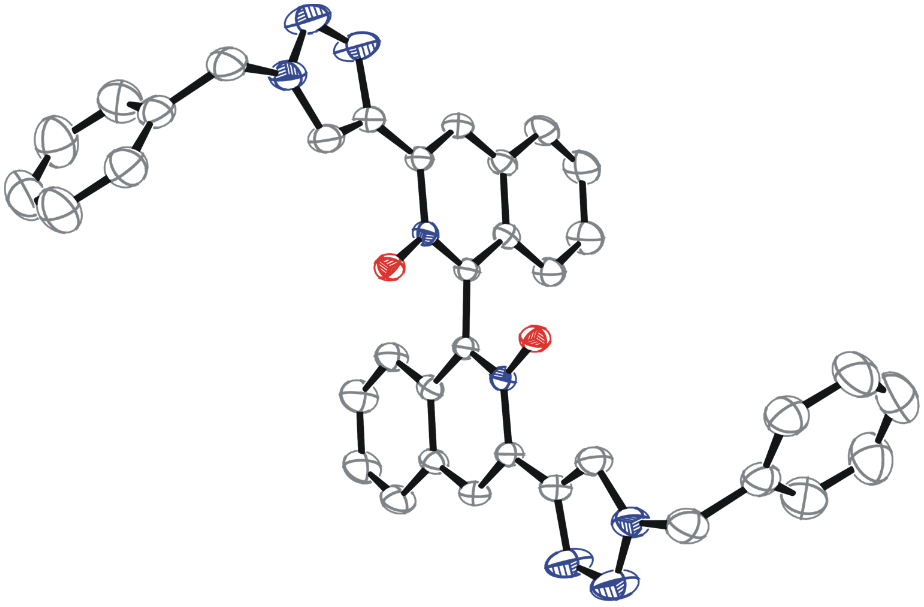


Figure S2. An ORTEP of the molecular structure of 3,3'-Bis-(1-benzyl-1*H*-1,2,3-triazole-4-yl)-1,1'-biisoquinoline *N, N'*-dioxide (**3a**) showing 40 % thermal ellipsoid probability.

1-(1-Adamantyl)-4-(trimethylsilyl)-1*H*-1,2,3-triazole (6d)

CuAAC of trimethylsilylacetylene with aryl or alkyl azides are generally thought to be exclusively regioselective, providing 1-aryl- (or alkyl)-4-TMS-1*H*-1,2,3-triazoles but not its 1,5-regioisomer. However, in many studies in the literature, 1,4-isomers are simply assigned using the assumption that trimethylsilylacetylene lead to 1,4-isomers under Cu catalysis. As such, we thought it would be prudent to secure the structure of the second example of the triazoles used in this study as provided below.

The representative procedure for crystallographic analysis was followed. Crystal data, data collection parameters, and results of the analyses are listed in Table S2. Colorless single crystals of **6d** suitable for x-ray diffraction analyses crystallized in the monoclinic crystal system by cooling a solution of **6d** in a 1:1 mixture of CH_2Cl_2 and toluene in the freezer (ca. –20 °C). The systematic absences in the intensity data were consistent with the space groups *Cc* and *C*2/*c*. The latter was chosen and confirmed by the successful refinement of the structure.

Table S2. Crystallographic Data for 1-(1-adamantyl)-4-(trimethylsilyl)-1*H*-1,2,3-triazole (6d)

	6d
Empirical formula	$C_{15}H_{25}N_3Si$
Formula weight	275.47
Crystal system	Monoclinic
Lattice parameters	
a (Å)	21.221(2)
b (Å)	13.6917(15)
c (Å)	11.2089(12)
β (°)	106.723(1)
V (Å^3)	3119.0(6)
Space group	*C*2/*c* (# 15)
Z value	8
ρ_{calc} (g / cm^3)	1.173
μ (Mo Kα) (mm^{-1})	0.143
Temperature (K)	273
$2\Theta_{max}$ (°)	51.0
No. Obs. (I > 2σ(I))	2353
No. Parameters	175
Goodness of fit	1.041
Max. shift in cycle	0.001
Residuals*:R1; wR2	0.0394; 0.1035
Absorption Correction, Max/min	Multi-scan 0.7457/0.6170
Largest peak in Final Diff. Map (e$^-$ / Å^3)	0.329

*$R = \Sigma_{hkl}(||F_{obs}|-|F_{calc}||)/\Sigma_{hkl}|F_{obs}|$; $R_w = [\Sigma_{hkl}w(|F_{obs}|-|F_{calc}|)^2/\Sigma_{hkl}wF_{obs}^2]^{1/2}$, $w = 1/\sigma^2(F_{obs})$; GOF = $[\Sigma_{hkl}w(|F_{obs}|-|F_{calc}|)^2/(n_{data} - n_{vari})]^{1/2}$.

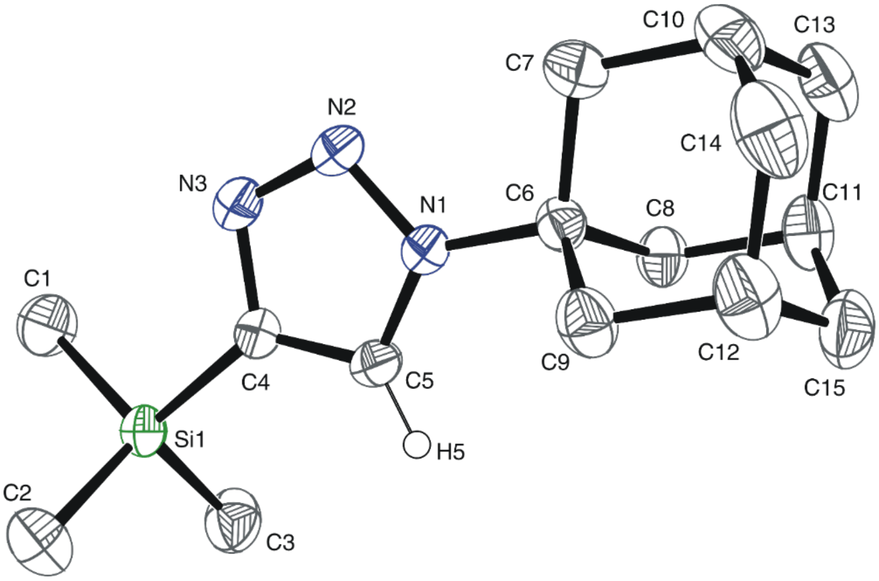


Figure S3. An ORTEP of the molecular structure of 1-(1-adamantyl)-4-(trimethylsilyl)-1*H*-1,2,3-triazole (**6d**) showing 40 % thermal ellipsoid probability.

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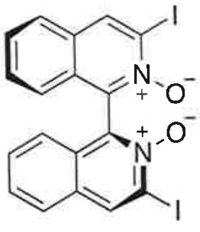
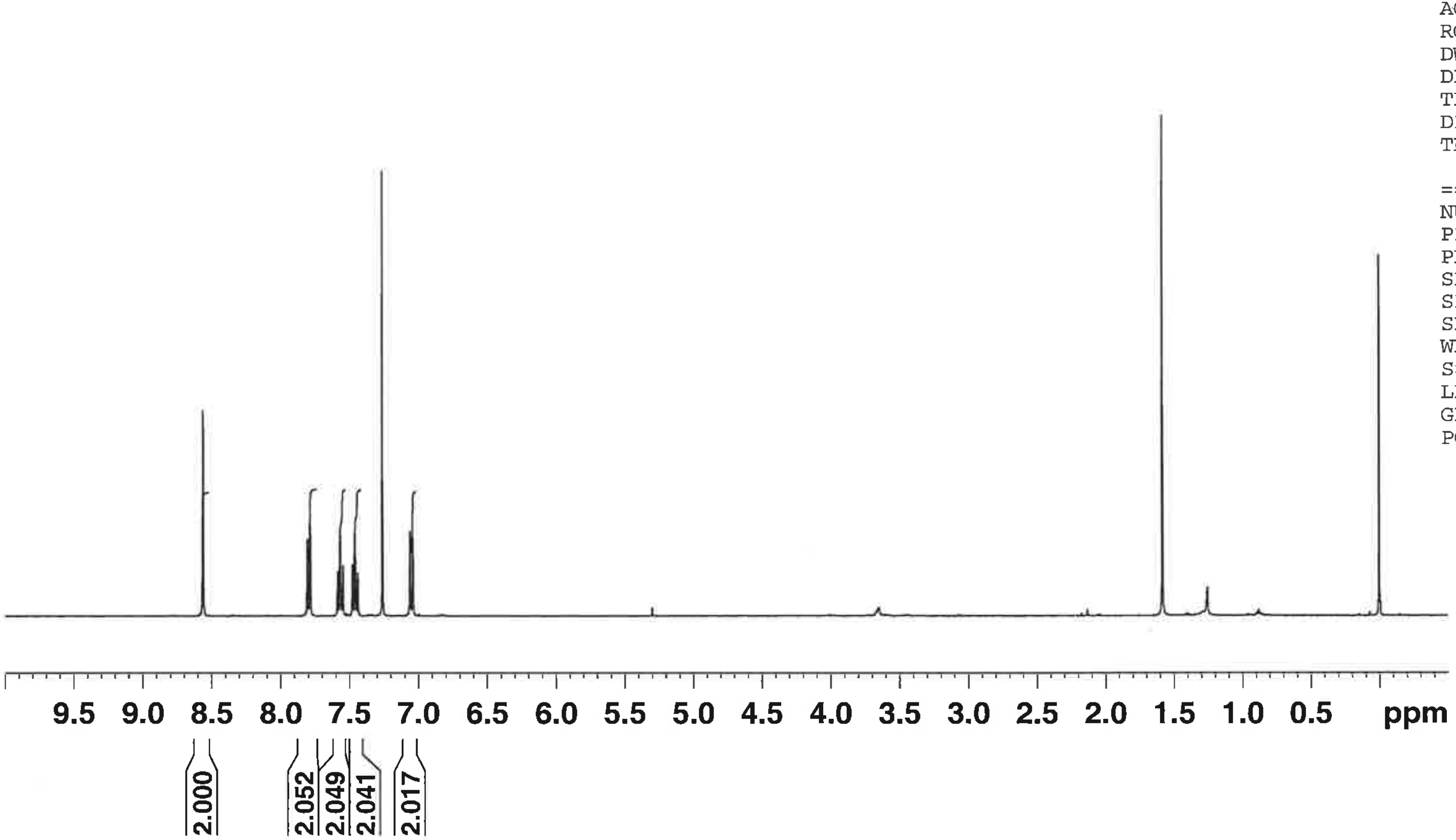
BIQNO-I

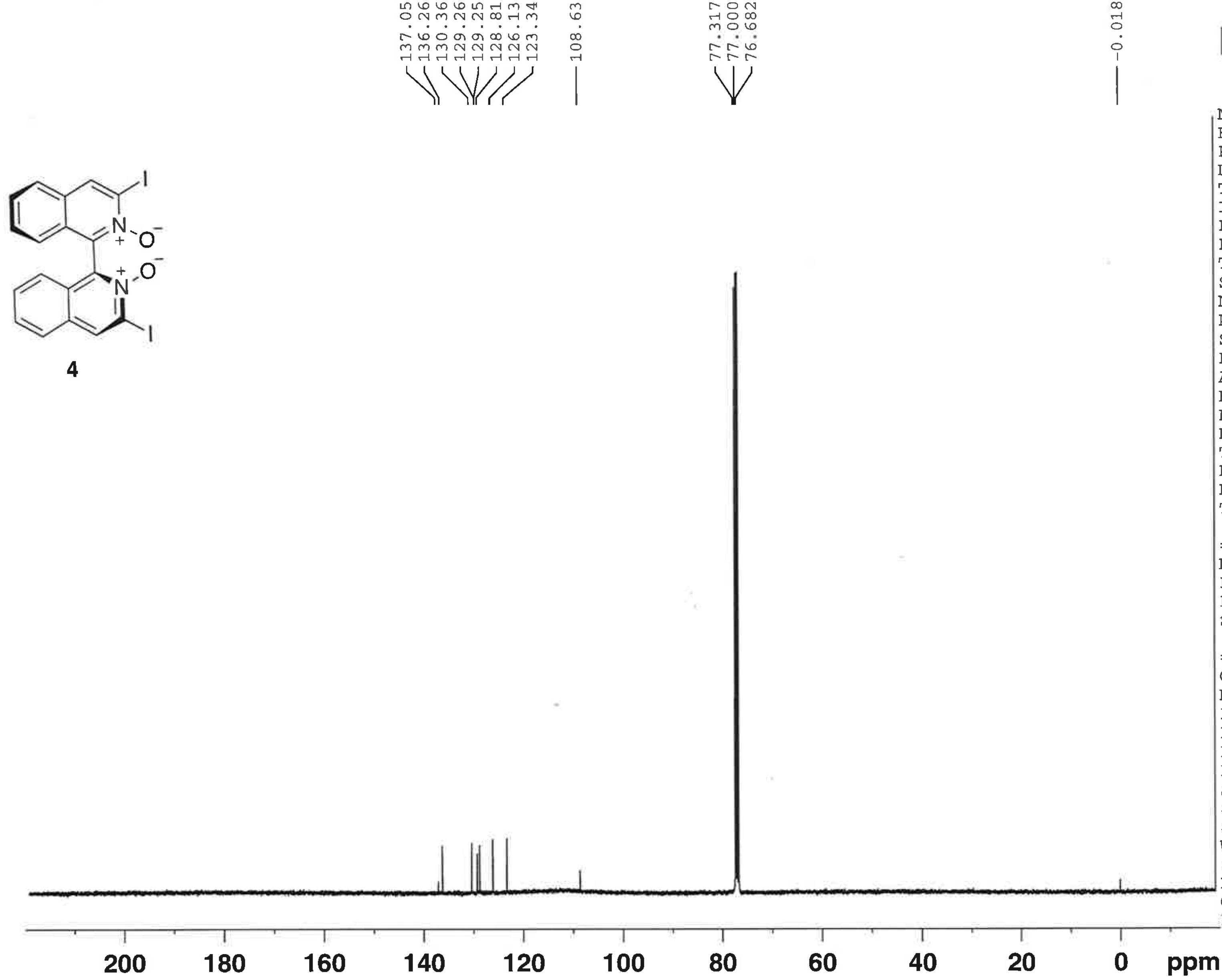


4



```
NAME          Aug23-2019
EXPNO                  9
PROCNO                 1
Date_           20190823
Time               17.43
INSTRUM            spect
PROBHD   5 mm TBI 1H/31
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                1149.4
DW                60.400 usec
DE                  6.50 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.75 usec
PL1                 3.00 dB
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300087 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

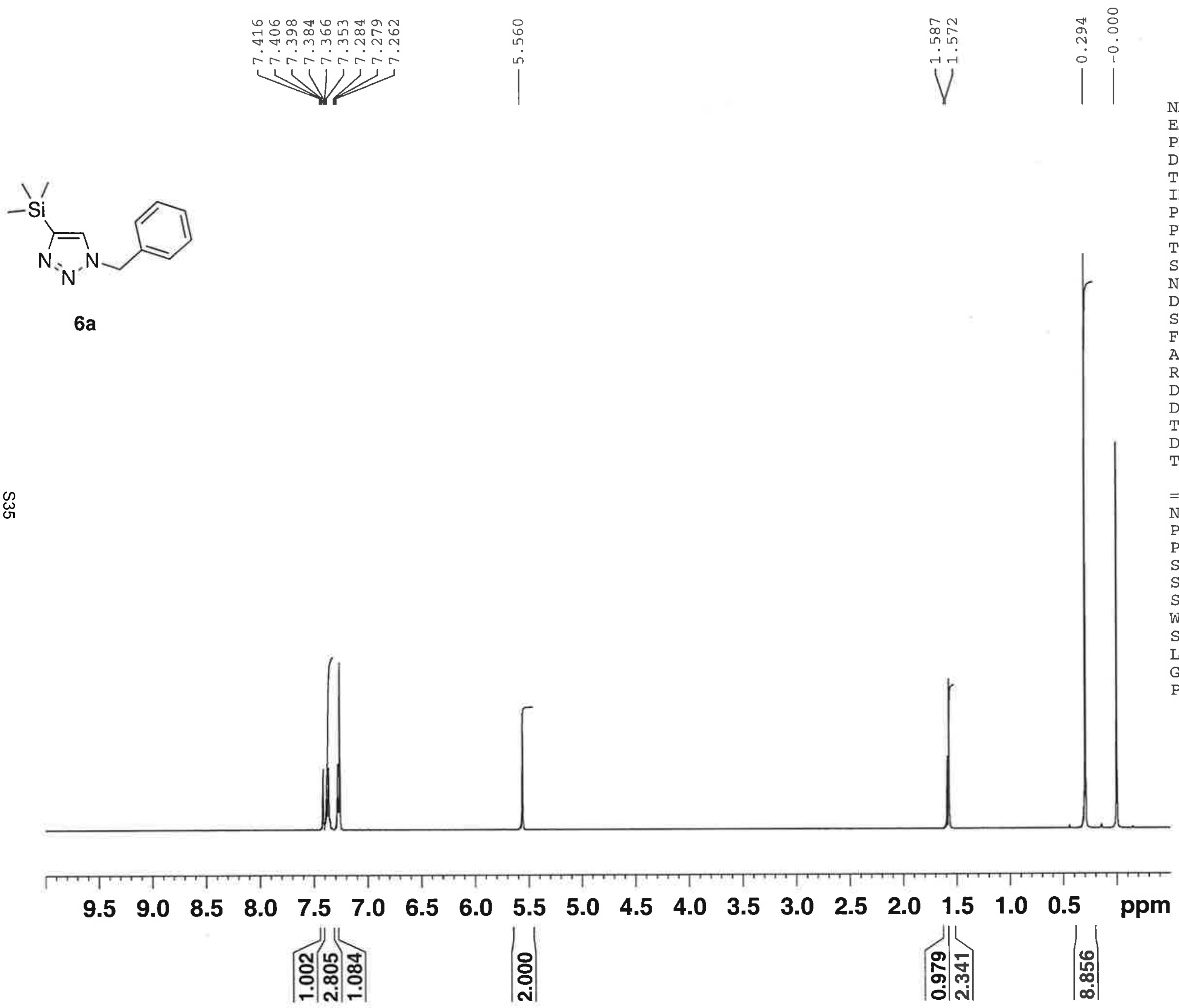
S-BIQNO-I



4

```
NAME           Aug23-2019
EXPNO                  11
PROCNO                  1
Date_            20190824
Time                 8.48
INSTRUM             spect
PROBHD   5 mm TBI 1H/31
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  10000
DS                      4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                 7298.2
DW                 20.850 usec
DE                   6.50 usec
TE                  300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  21.00 usec
PL1                 -4.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -2.00 dB
PL12                14.49 dB
PL13                14.49 dB
SFO2          400.1316005 MHz
SI                  32768
SF            100.6127702 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

bn tms triazole

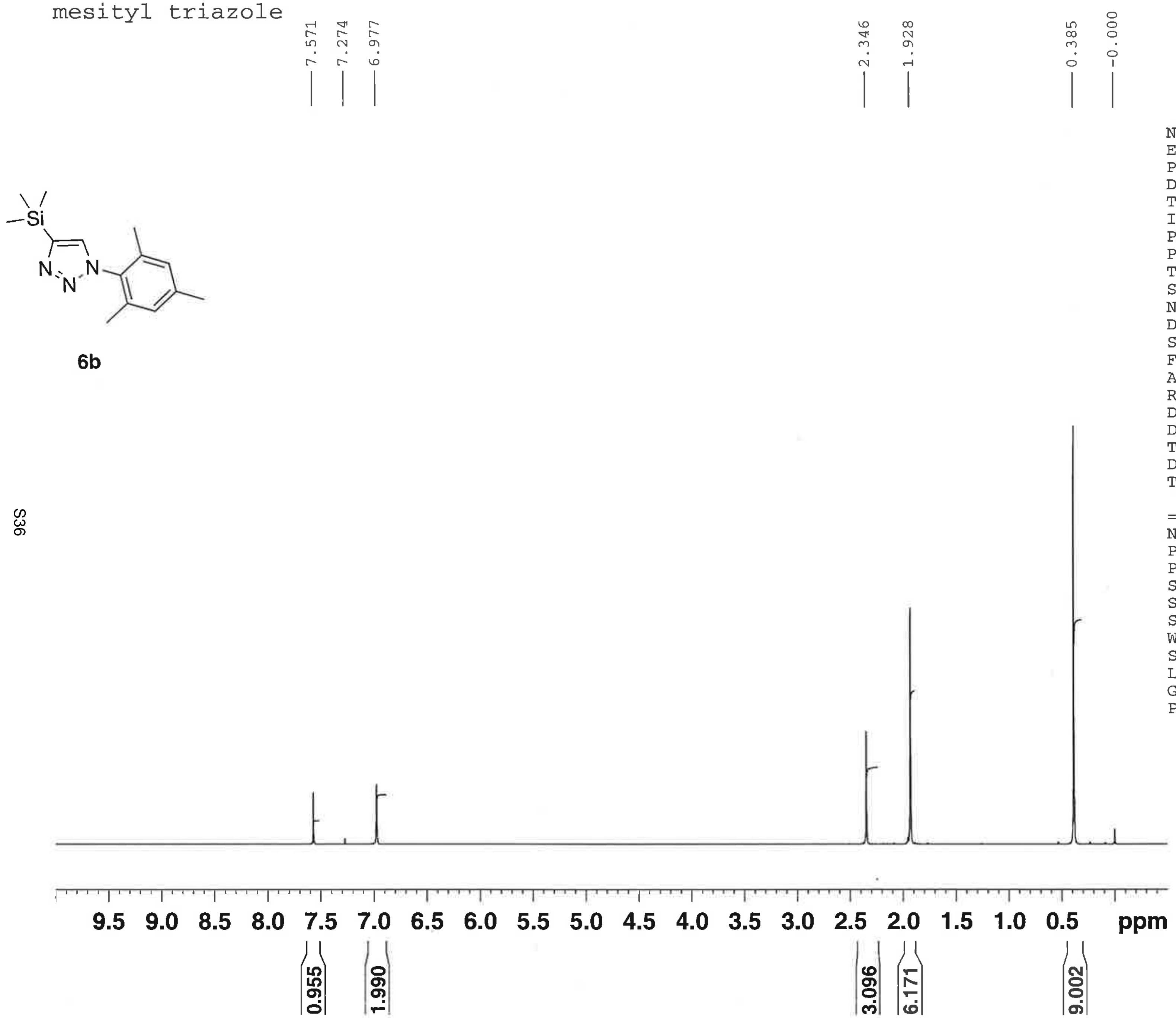


NAME	Jan25-2021
EXPNO	30
PROCNO	1
Date_	20210125
Time	14.16
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	456.1
DW	60.400 usec
DE	6.50 usec
TE	295.5 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	11.60 usec
PL1	3.00 dB
SFO1	400.1324710 MHz
SI	32768
SF	400.1300089 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

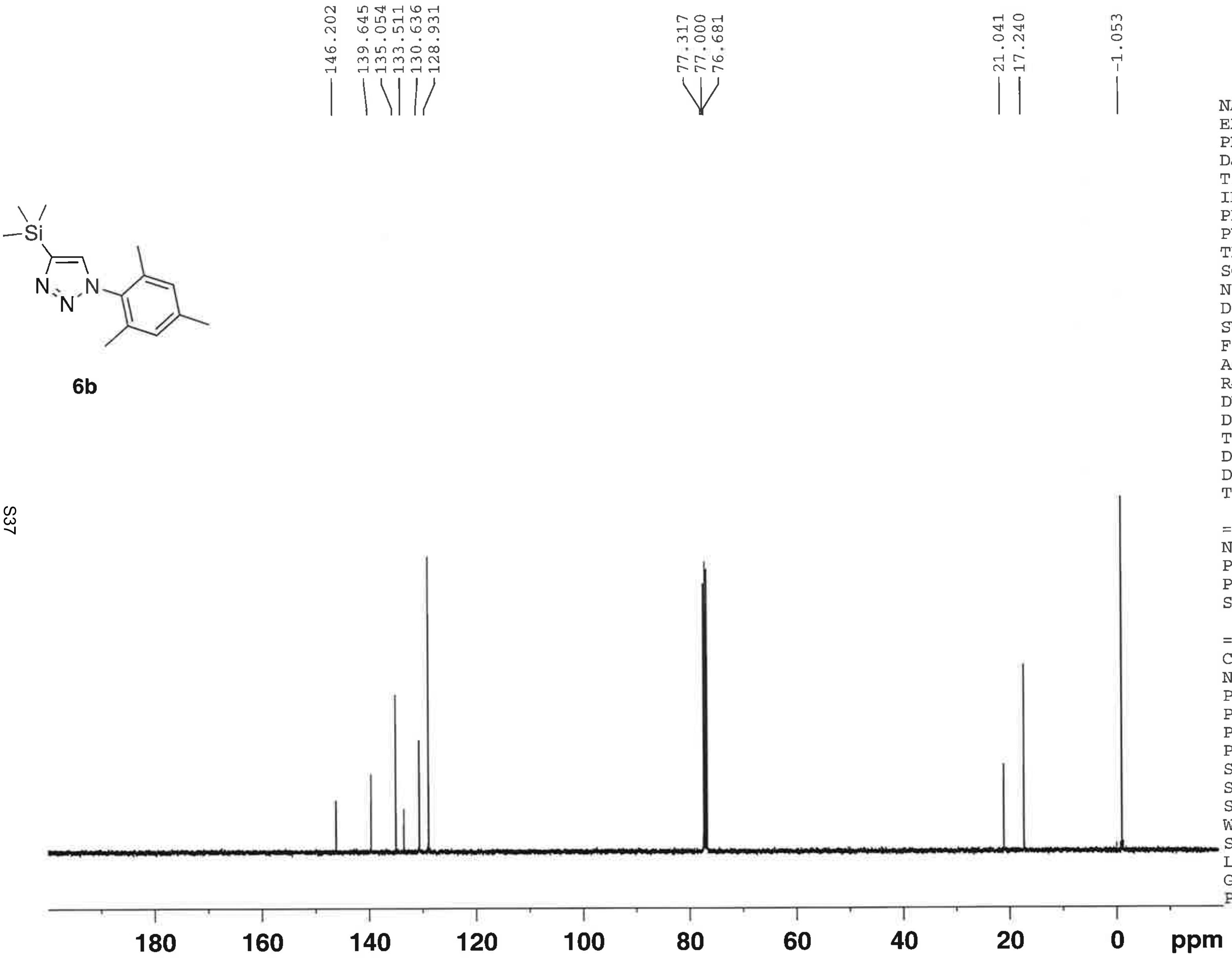
mesityl triazole



6b

```
NAME           Jan19-2021
EXPNO                  30
PROCNO                  1
Date_            20210119
Time                13.49
INSTRUM             spect
PROBHD      5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                   90.5
DW                 60.400 usec
DE                   6.50 usec
TE                  293.7 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  11.60 usec
PL1                  3.00 dB
SFO1          400.1324710 MHz
SI                  32768
SF            400.1300038 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

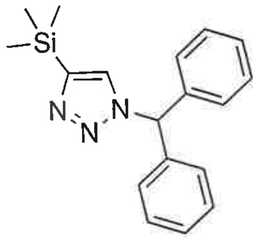
mesityl triazole



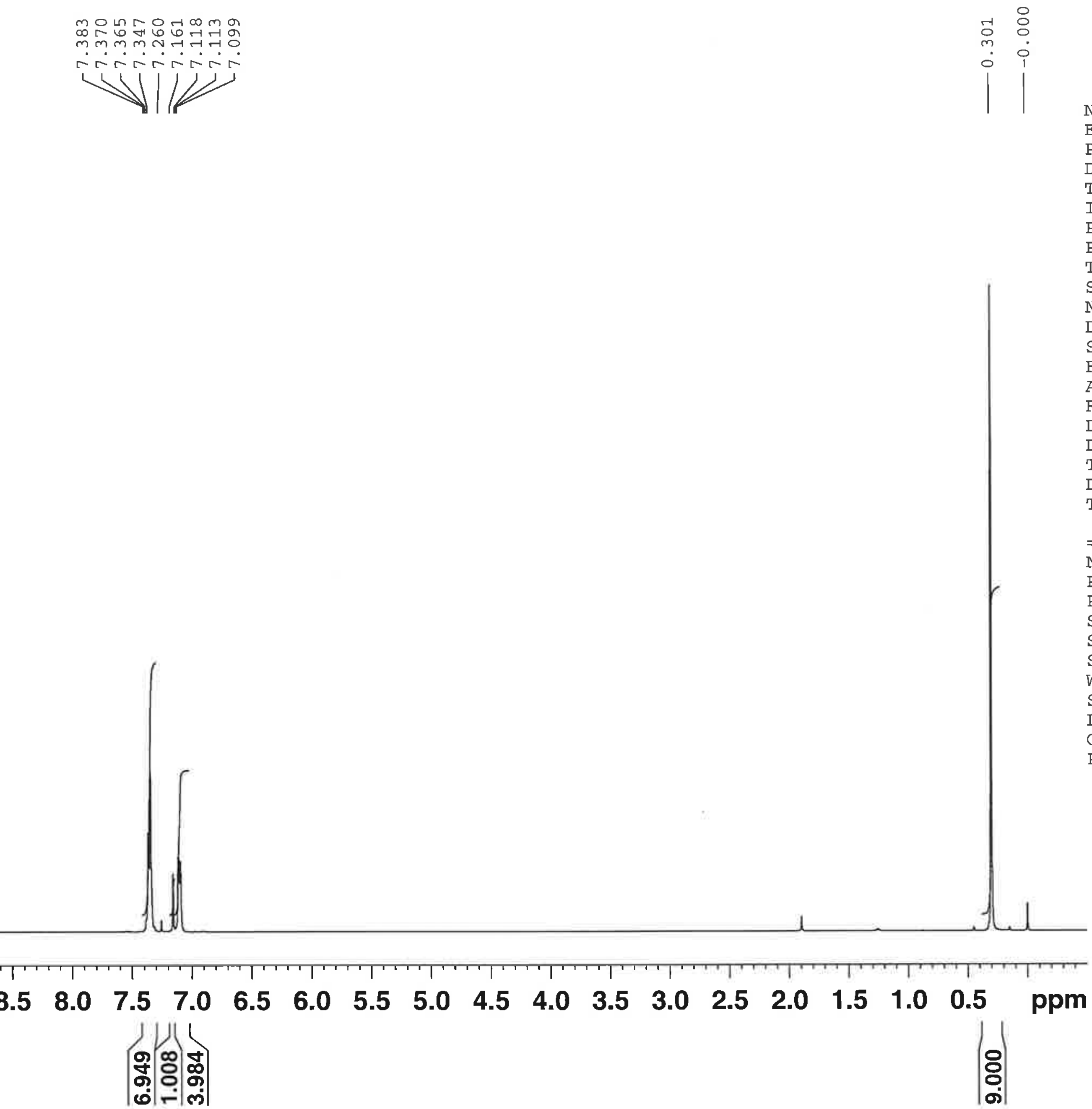
6b

```
NAME             Jan19-2022
EXPNO                    31
PROCNO                    1
Date_              20210119
Time                  14.57
INSTRUM               spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               23980.814 Hz
FIDRES             0.365918 Hz
AQ                1.3664756 sec
RG                    32768
DW                   20.850 usec
DE                     6.50 usec
TE                    298.8 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    14.75 usec
PL1                    0.00 dB
SFO1            100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                   -2.00 dB
PL12                  12.54 dB
PL13                  15.00 dB
SFO2            400.1316005 MHz
SI                    32768
SF              100.6127732 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

diphenyl triazole



6c

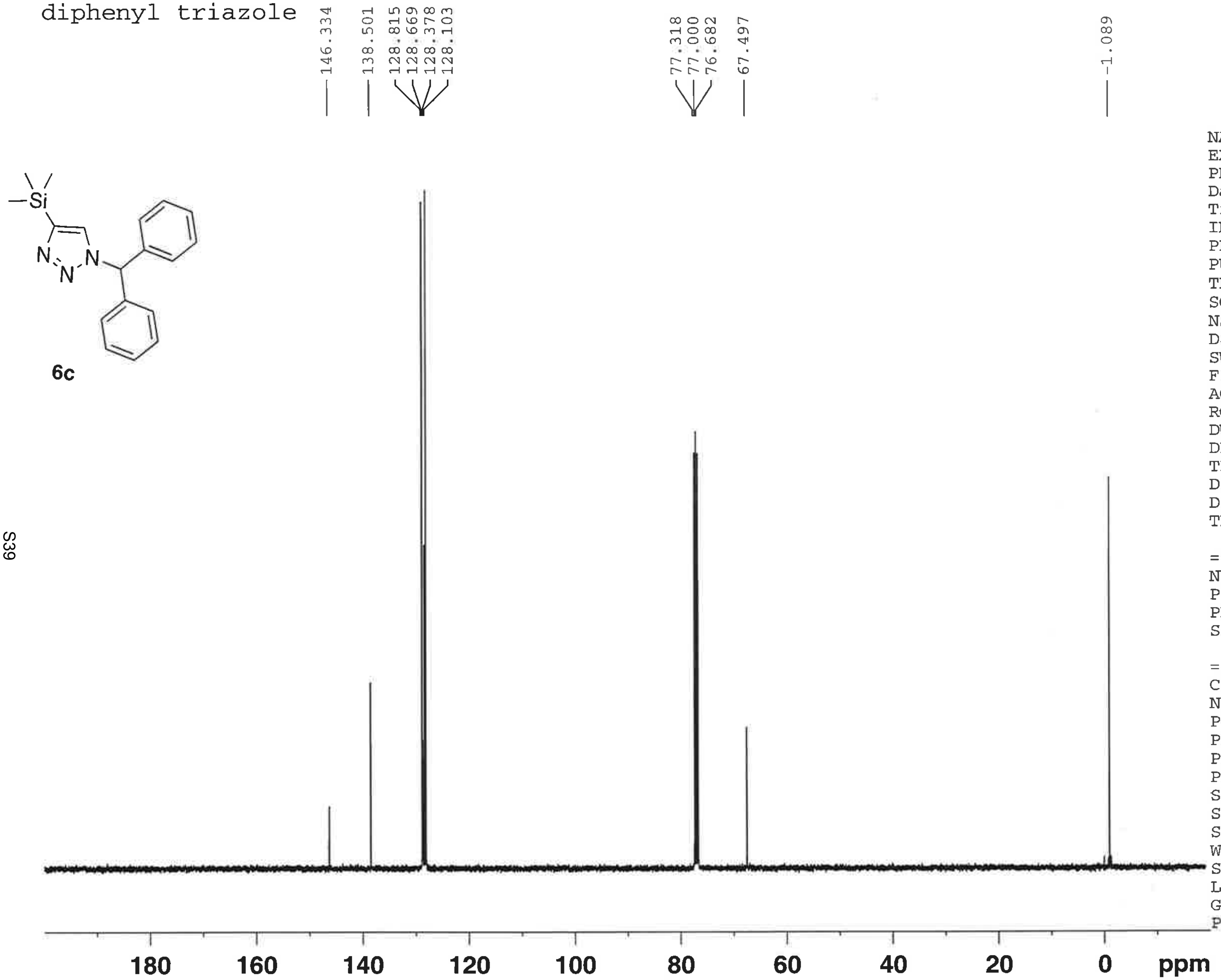


NAME	Jan11-2021
EXPNO	20
PROCNO	1
Date_	20210111
Time	13.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	114
DW	60.400 usec
DE	6.50 usec
TE	293.3 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	11.60 usec
PL1	3.00 dB
SFO1	400.1324710 MHz
SI	32768
SF	400.1300099 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

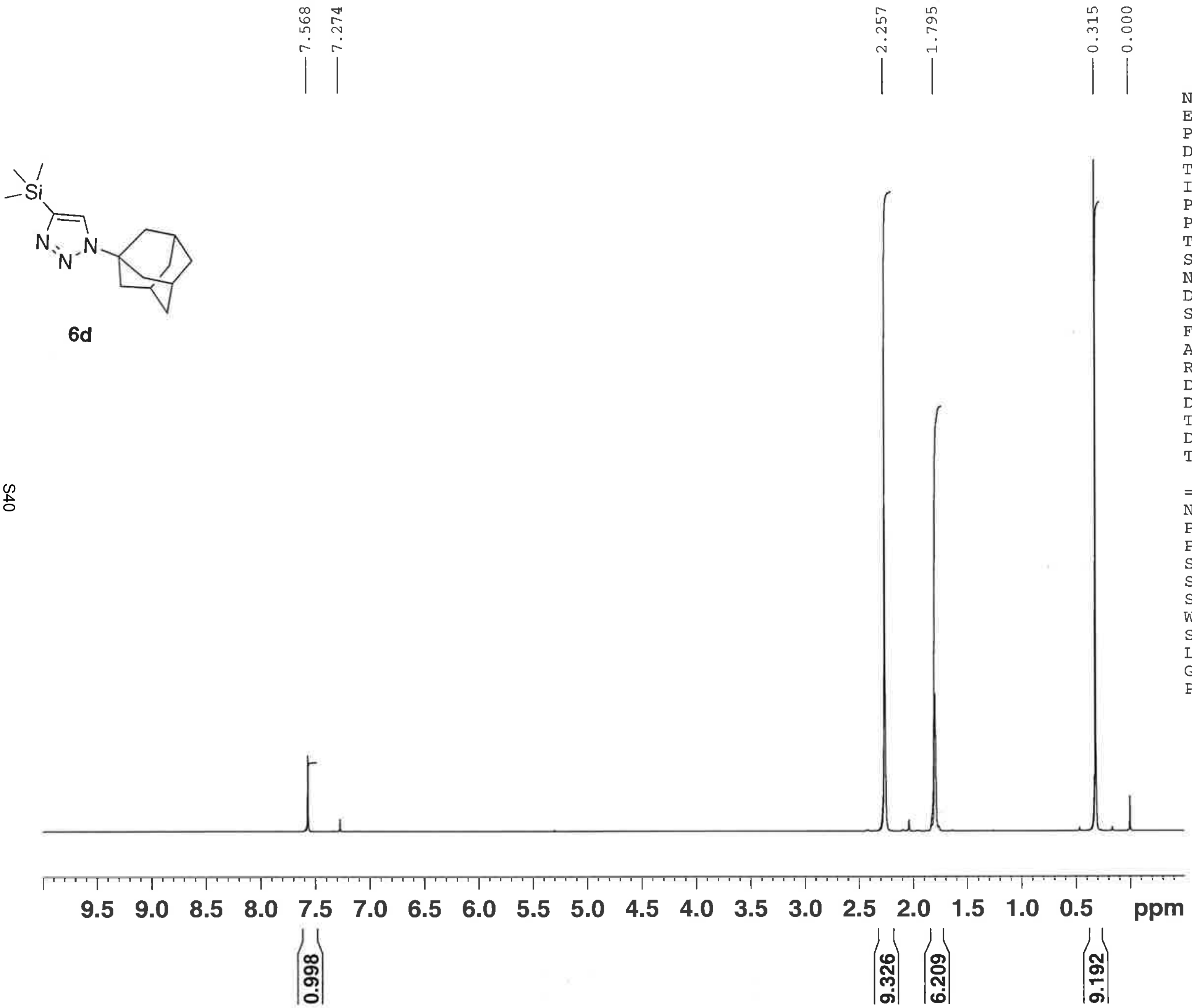
diphenyl triazole



6c

```
NAME          Jan11-2021
EXPNO                 21
PROCNO                 1
Date_           20210111
Time               15.25
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                 32768
DW                20.850 usec
DE                  6.50 usec
TE                 298.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 14.75 usec
PL1                 0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               12.54 dB
PL13               15.00 dB
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127733 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

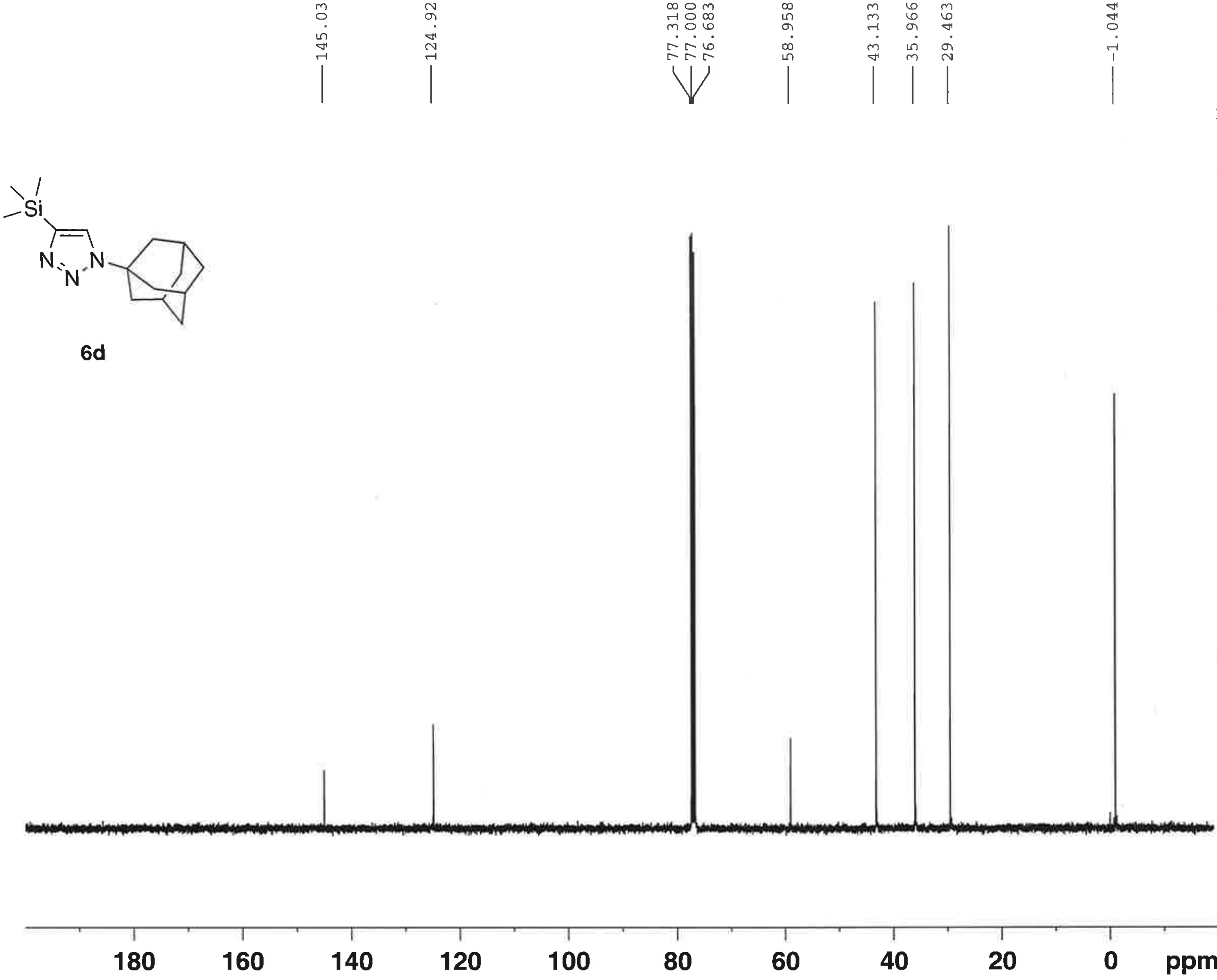
adamantane triazole



6d

```
NAME          Jan15-2021
EXPNO                 20
PROCNO                 1
Date_           20210115
Time               10.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                  90.5
DW                60.400 usec
DE                  6.50 usec
TE                 293.4 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.60 usec
PL1                 3.00 dB
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300039 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

adamantane triazole



6d

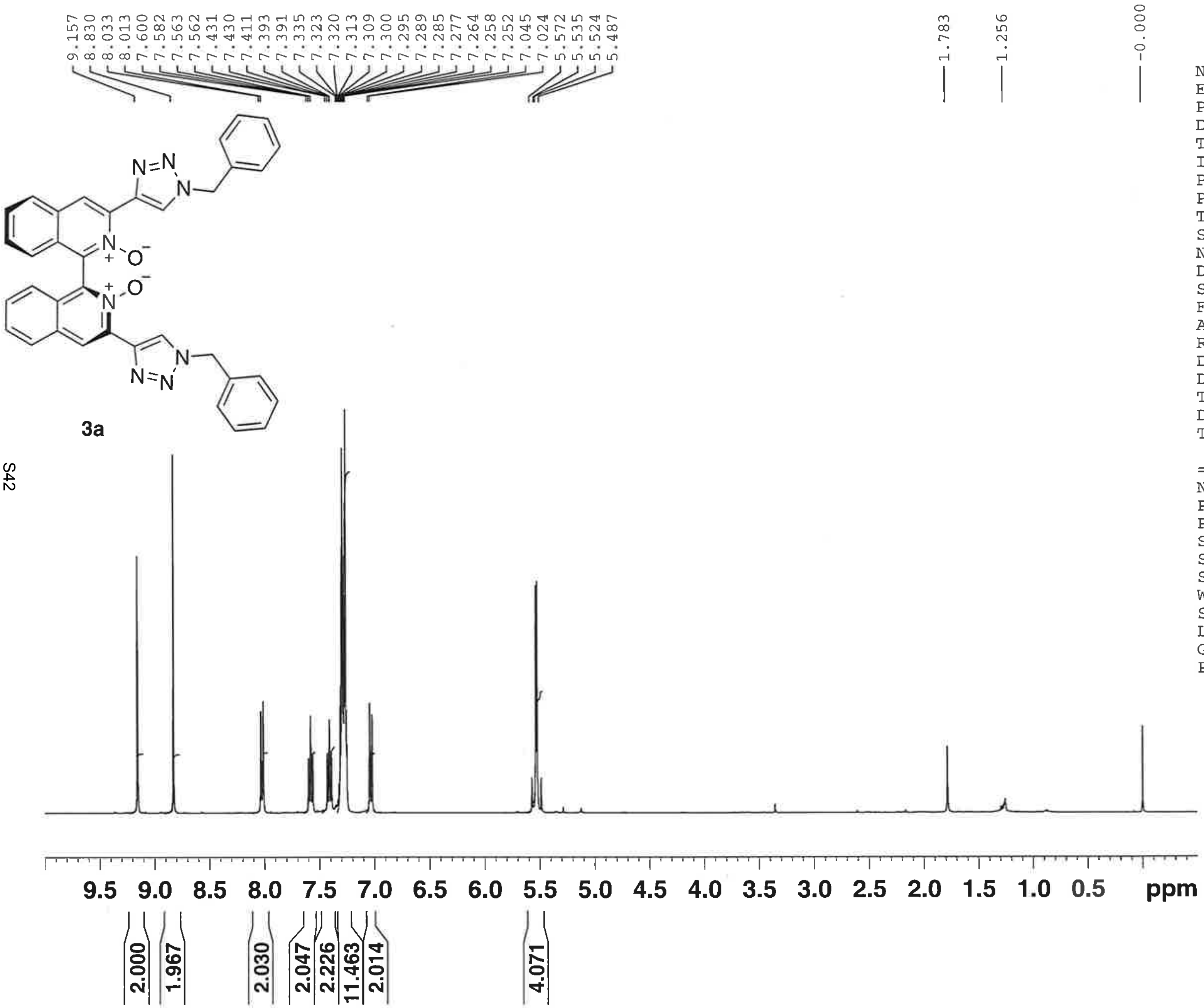
NAME	Jan15-2021
EXPNO	21
PROCNO	1
Date_	20210115
Time	11.56
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	32768
DW	20.850 usec
DE	6.50 usec
TE	298.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	14.75 usec
PL1	0.00 dB
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

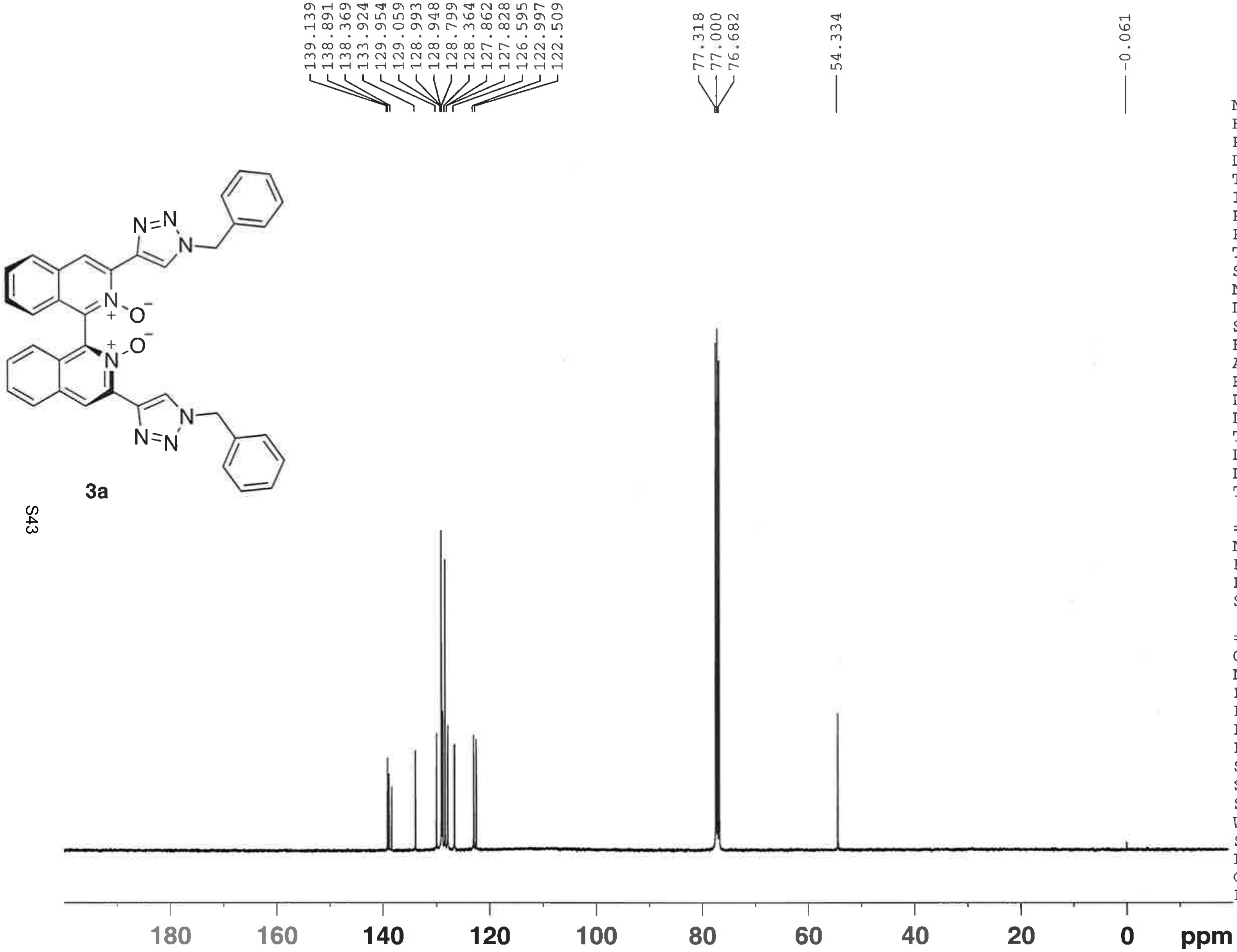
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	12.54 dB
PL13	15.00 dB
SFO2	400.1316005 MHz
SI	32768
SF	100.6127720 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



3a

```
NAME          Nov04-2019
EXPNO                  6
PROCNO                 1
Date_           20191104
Time               17.02
INSTRUM            spect
PROBHD   5 mm TBI 1H/31
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                   181
DW                60.400 usec
DE                  6.50 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.75 usec
PL1                 3.00 dB
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300076 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

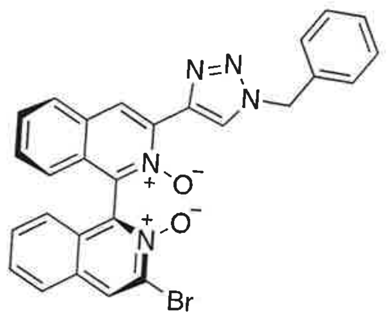
s-BIQNO-triazole-Bn



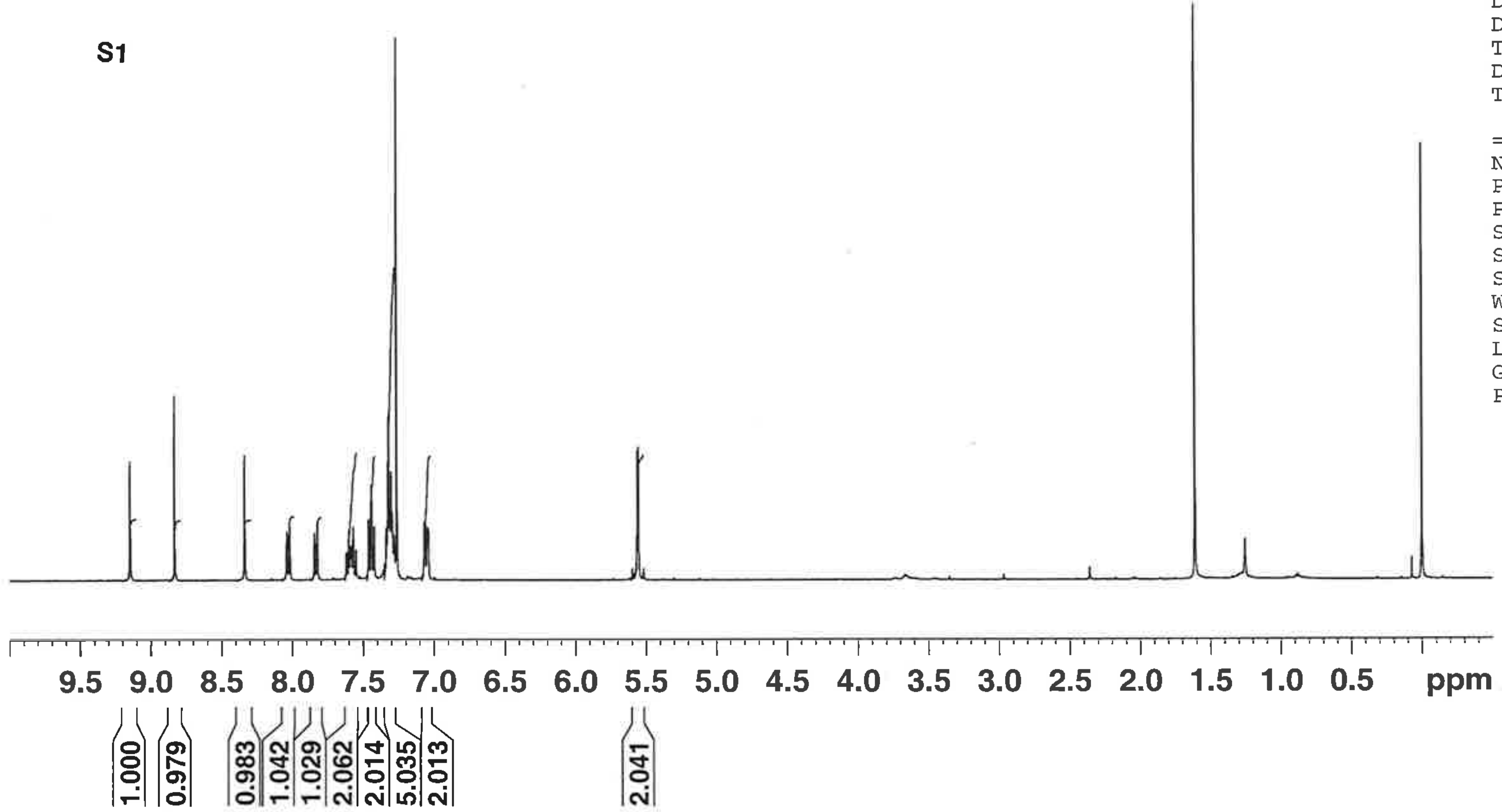
3a

```
NAME           Nov04-2019
EXPNO                   8
PROCNO                  1
Date_            20191105
Time                 6.35
INSTRUM             spect
PROBHD   5 mm TBI 1H/31
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 10000
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                 16384
DW                20.850 usec
DE                  6.50 usec
TE                 300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 21.00 usec
PL1                -4.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               14.49 dB
PL13               14.49 dB
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127785 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

mono-coupling P



S1

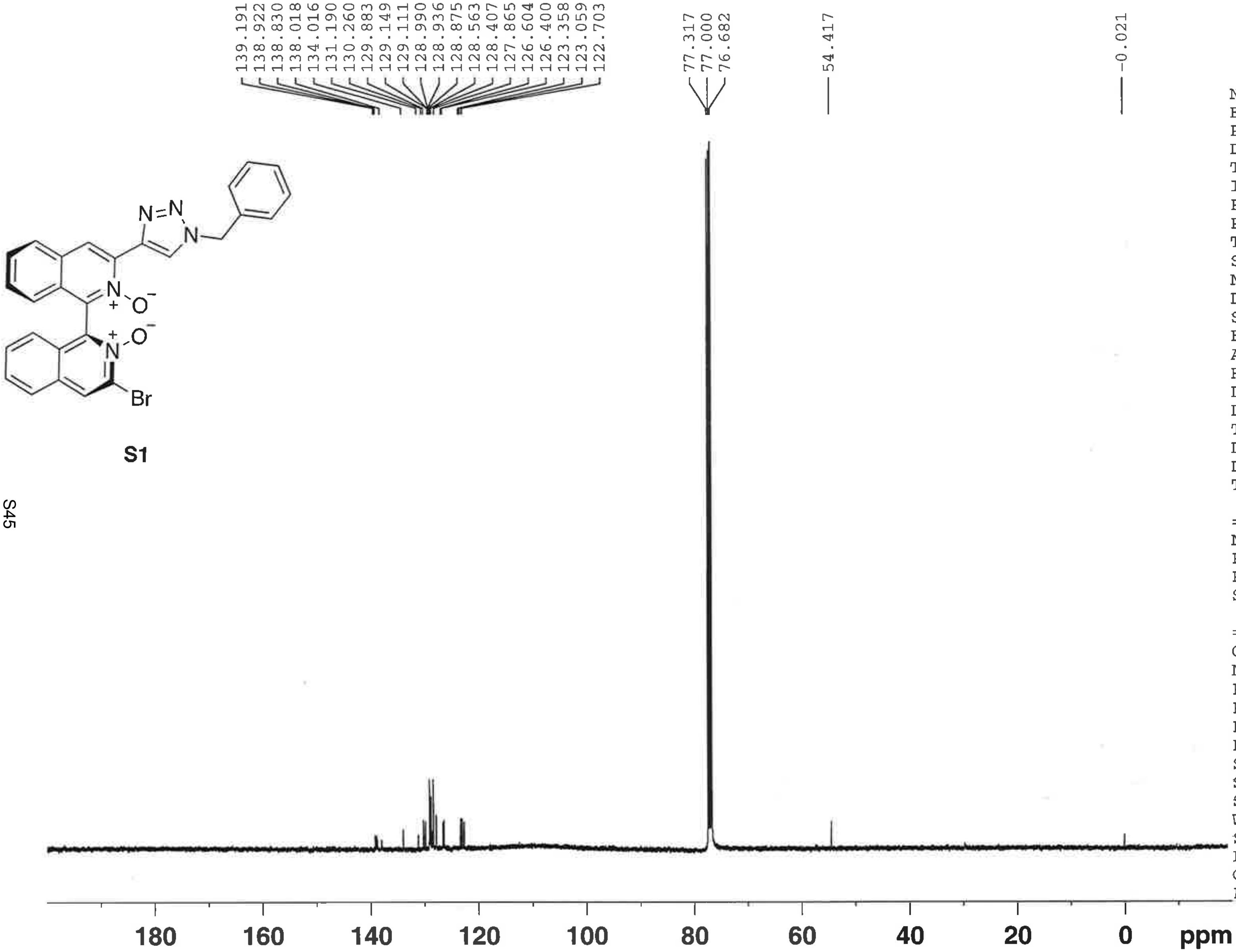


NAME	Aug28-2019
EXPNO	2
PROCNO	1
Date_	20190828
Time	16.10
INSTRUM	spect
PROBHD	5 mm TBI 1H/31
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	574.7
DW	60.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

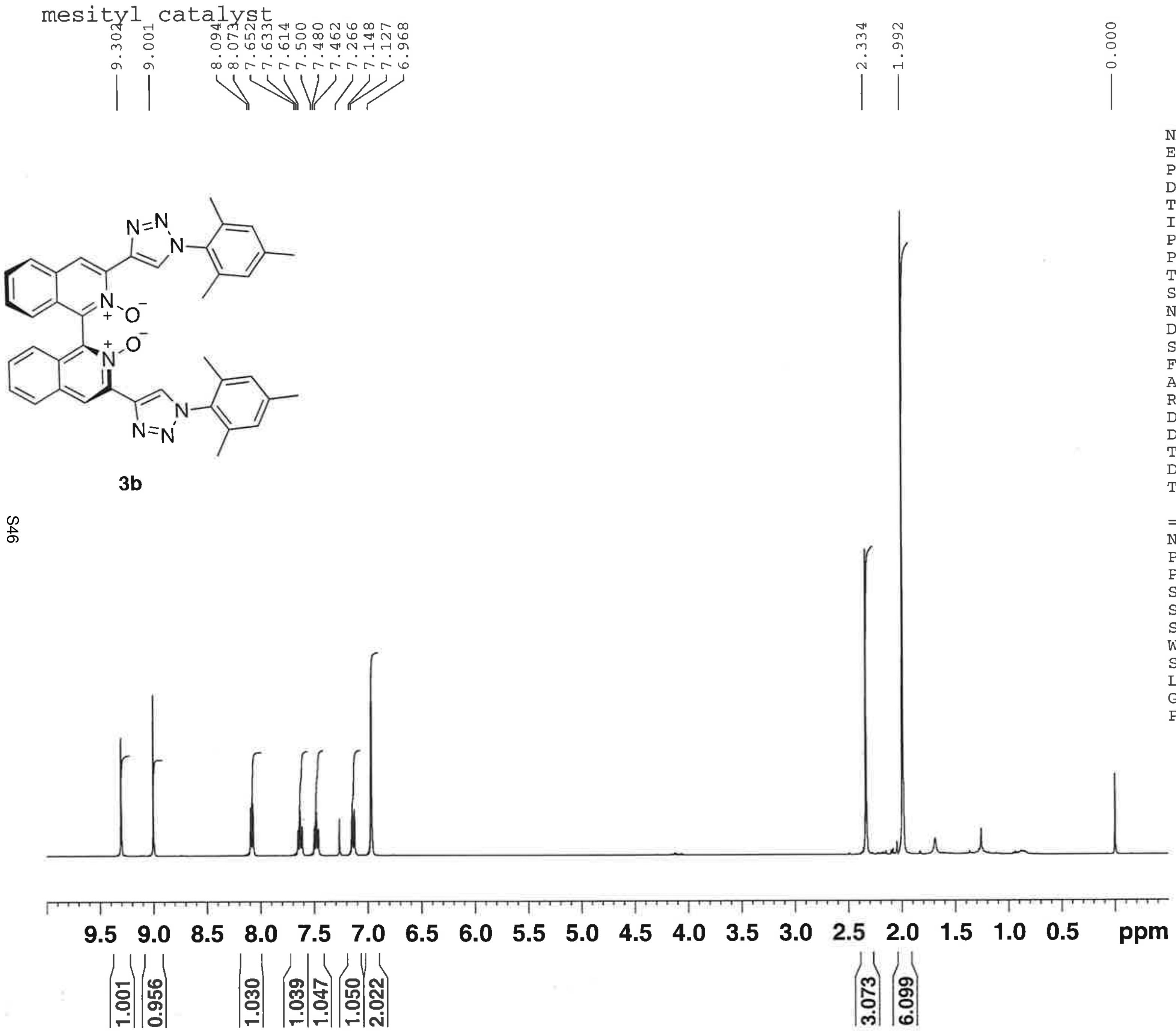
NUC1	1H
P1	11.75 usec
PL1	3.00 dB
SFO1	400.1324710 MHz
SI	32768
SF	400.1300082 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

mono-coupling product



S1

```
NAME           Aug28-2019
EXPNO                   6
PROCNO                  1
Date_            20190829
Time                 6.17
INSTRUM             spect
PROBHD     5 mm TBI 1H/31
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  12000
DS                      4
SWH             23980.814 Hz
FIDRES           0.365918 Hz
AQ              1.3664756 sec
RG                20642.5
DW                 20.850 usec
DE                   6.50 usec
TE                  300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  21.00 usec
PL1                 -4.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -2.00 dB
PL12                14.49 dB
PL13                14.49 dB
SFO2          400.1316005 MHz
SI                  32768
SF            100.6127716 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



3b

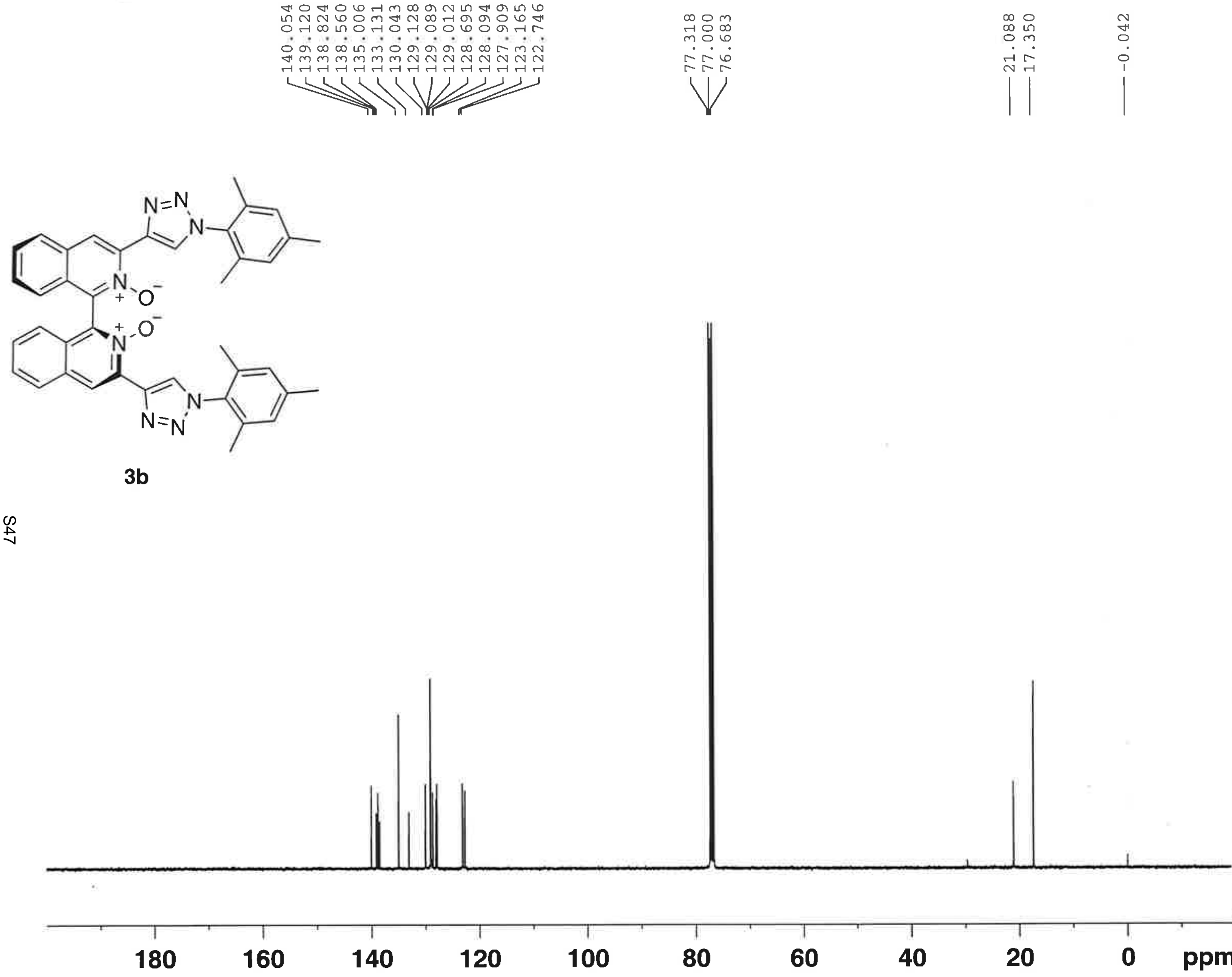
NAME	Jan18-2023
EXPNO	20
PROCNO	1
Date_	20210118
Time	18.06
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	128
DW	60.400 usec
DE	6.50 usec
TE	294.6 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	11.60 usec
PL1	3.00 dB
SFO1	400.1324710 MHz
SI	32768
SF	400.1300072 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

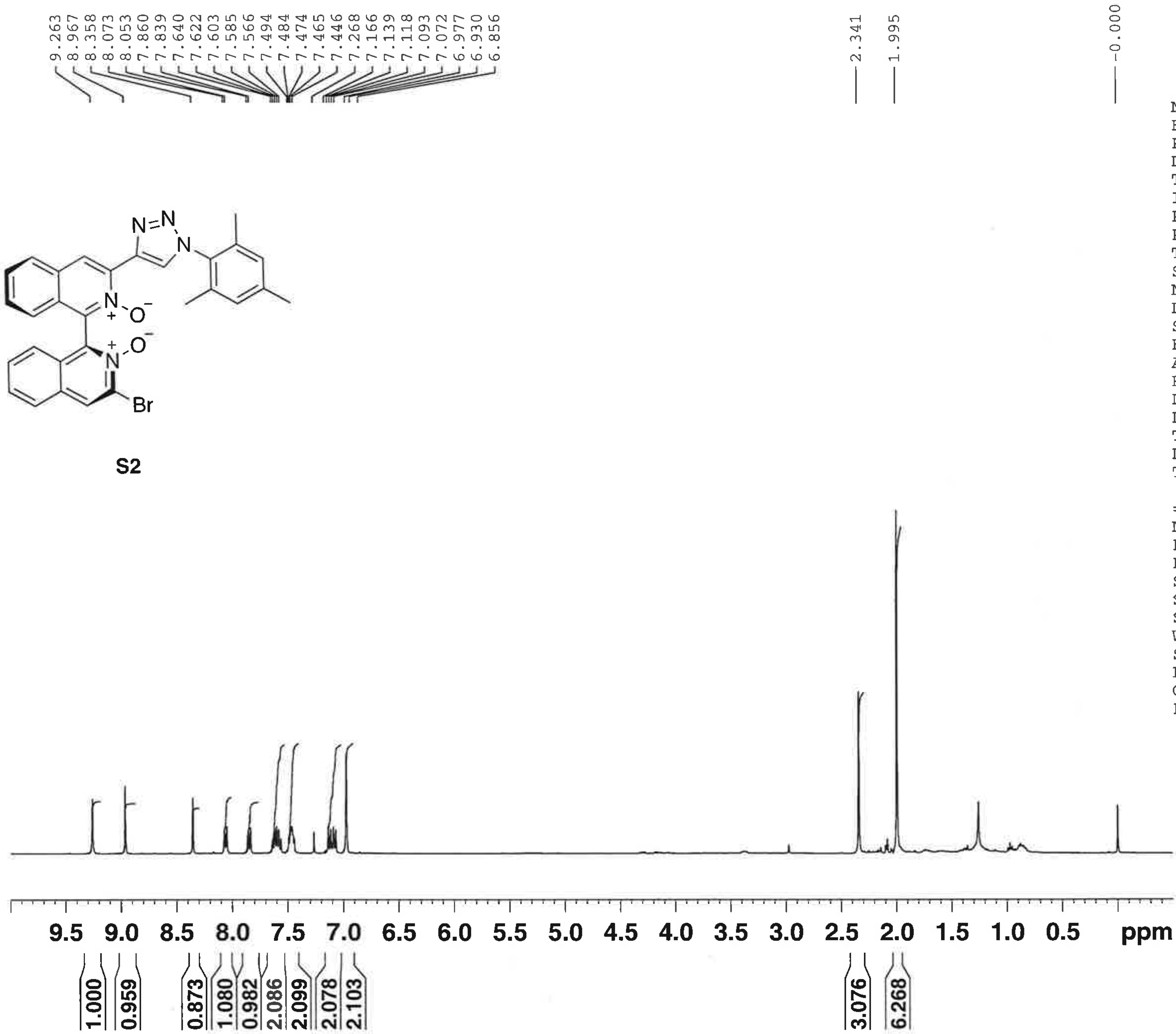
mesityl catalyst

3b



```
NAME             Jan18-2023
EXPNO                    21
PROCNO                    1
Date_              20210119
Time                   3.40
INSTRUM               spect
PROBHD      5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                    10000
DS                        4
SWH              23980.814 Hz
FIDRES            0.365918 Hz
AQ               1.3664756 sec
RG                    32768
DW                   20.850 usec
DE                     6.50 usec
TE                    299.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    14.75 usec
PL1                    0.00 dB
SFO1            100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                   -2.00 dB
PL12                  12.54 dB
PL13                  15.00 dB
SFO2            400.1316005 MHz
SI                    32768
SF              100.6127722 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

mes monocouple



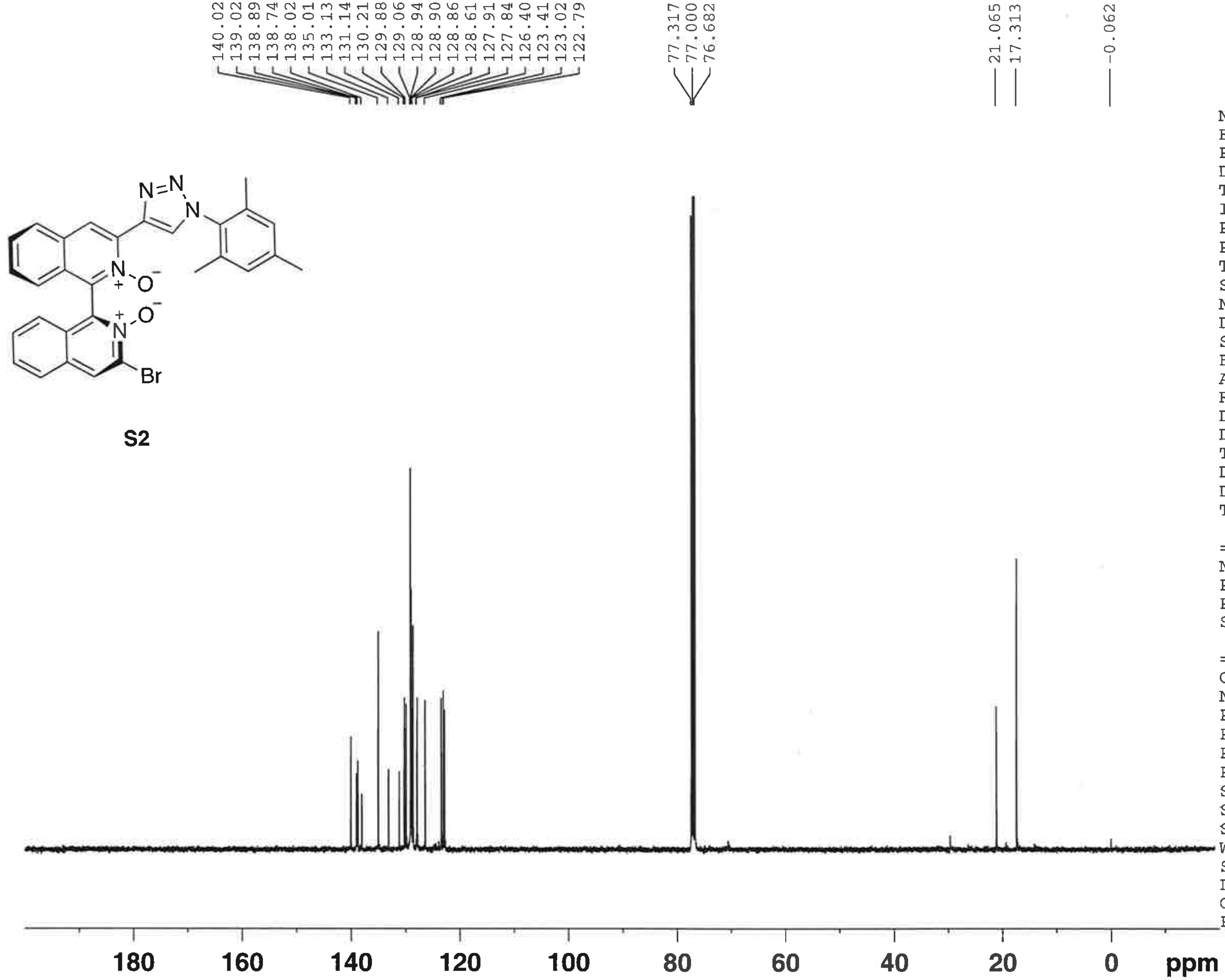
S2

NAME	Feb05-2021
EXPNO	40
PROCNO	1
Date_	20210205
Time	15.47
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	114
DW	60.400 usec
DE	6.50 usec
TE	293.4 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	11.60 usec
PL1	3.00 dB
SFO1	400.1324710 MHz
SI	32768
SF	400.1300066 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

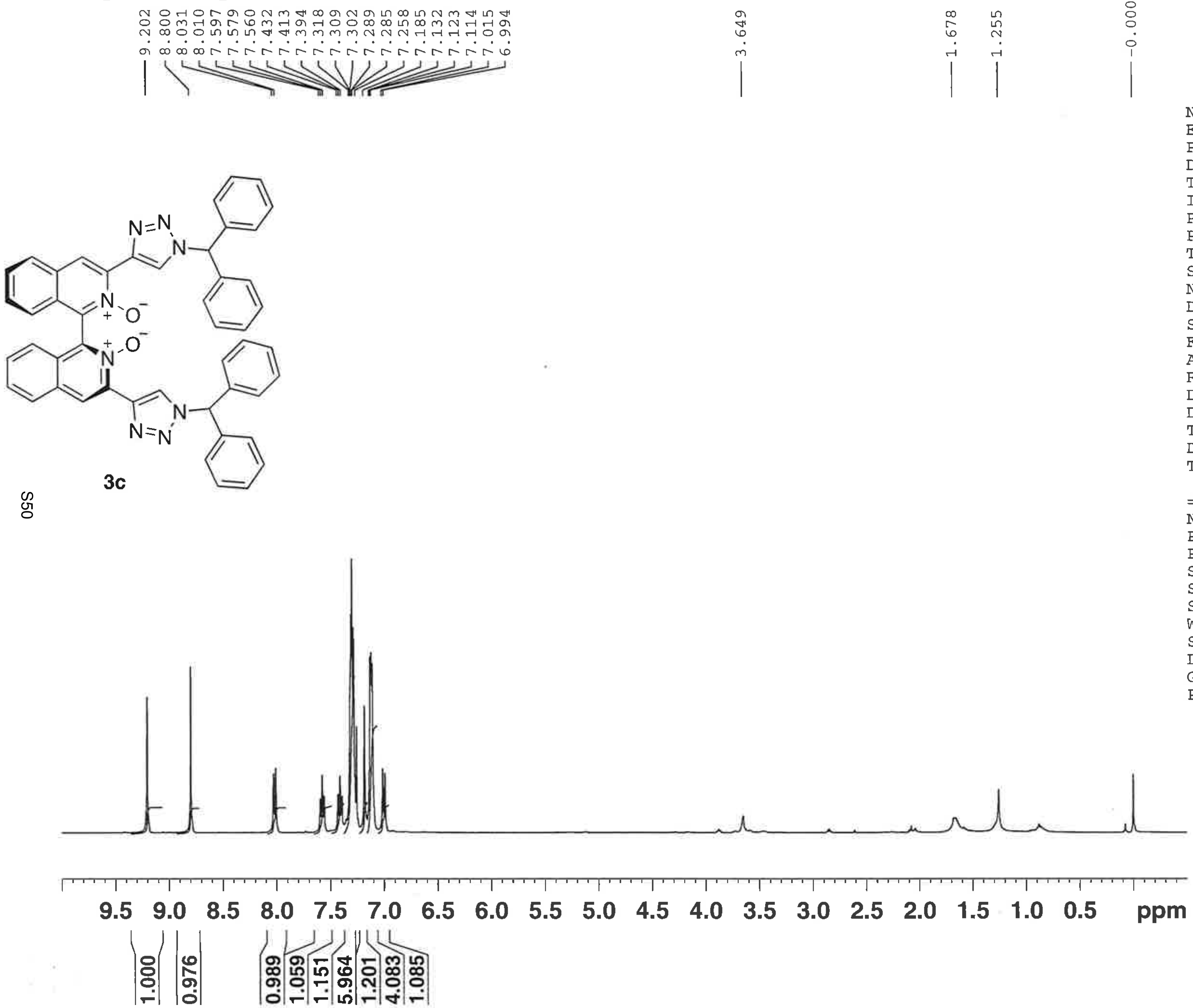
Mes-mono



S2

```
NAME            Feb12-2021
EXPNO                   31
PROCNO                   1
Date_             20210213
Time                  6.45
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  10000
DS                      4
SWH             23980.814 Hz
FIDRES           0.365918 Hz
AQ              1.3664756 sec
RG                  32768
DW                 20.850 usec
DE                   6.50 usec
TE                  300.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  14.75 usec
PL1                  0.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -2.00 dB
PL12                12.54 dB
PL13                15.00 dB
SFO2          400.1316005 MHz
SI                  32768
SF            100.6127746 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

di-ph-dicouple

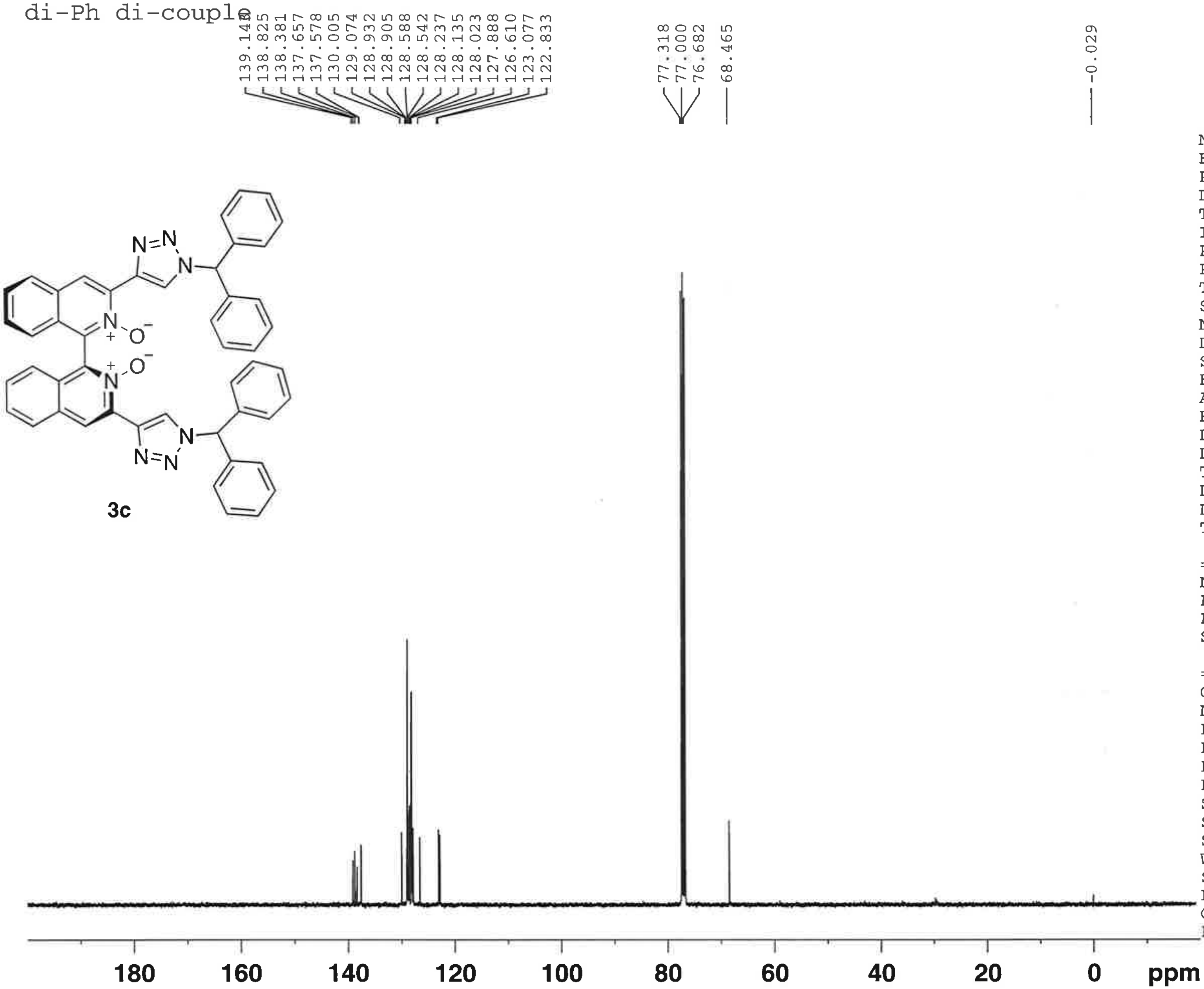


NAME	Feb18-2021
EXPNO	10
PROCNO	1
Date_	20210218
Time	16.54
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	181
DW	60.400 usec
DE	6.50 usec
TE	295.0 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	11.60 usec
PL1	3.00 dB
SFO1	400.1324710 MHz
SI	32768
SF	400.1300103 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

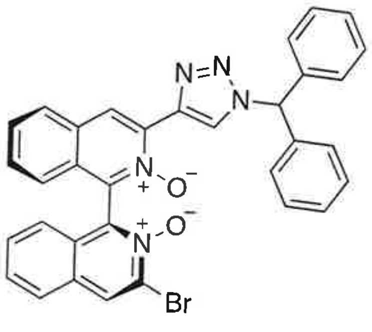
di-Ph di-couple



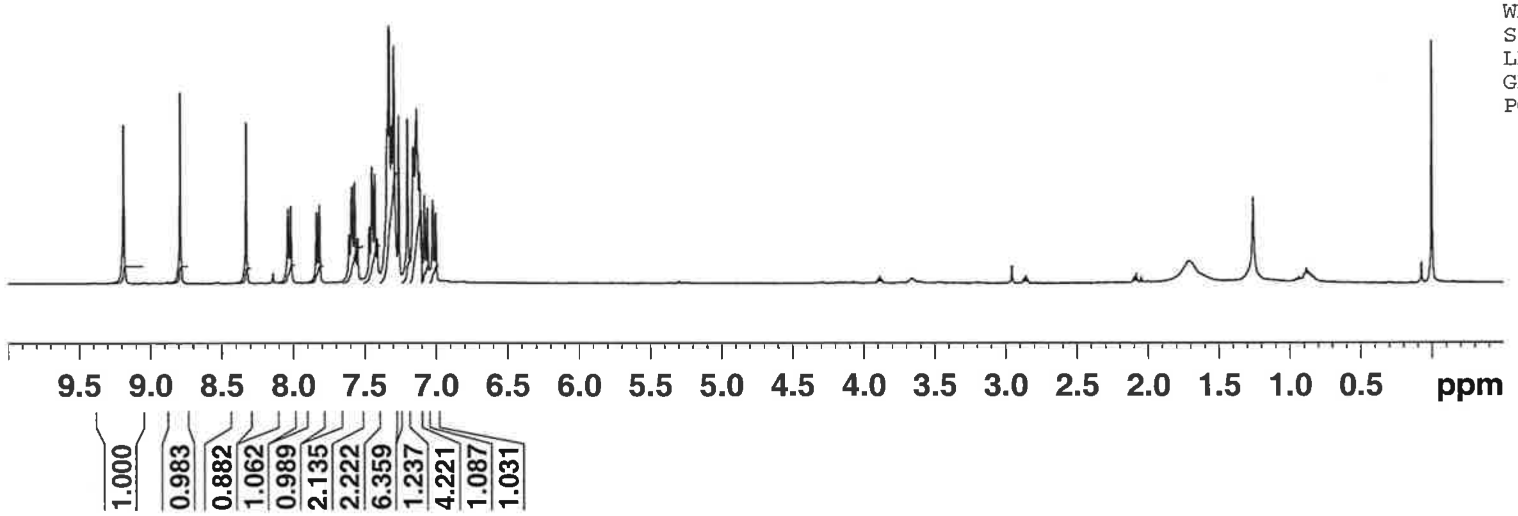
3c

```
NAME          Feb07-2021
EXPNO                 31
PROCNO                 1
Date_           20210207
Time               21.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 10000
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                 32768
DW                20.850 usec
DE                  6.50 usec
TE                 300.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 14.75 usec
PL1                 0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               12.54 dB
PL13               15.00 dB
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127712 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

di-Ph mono



S3

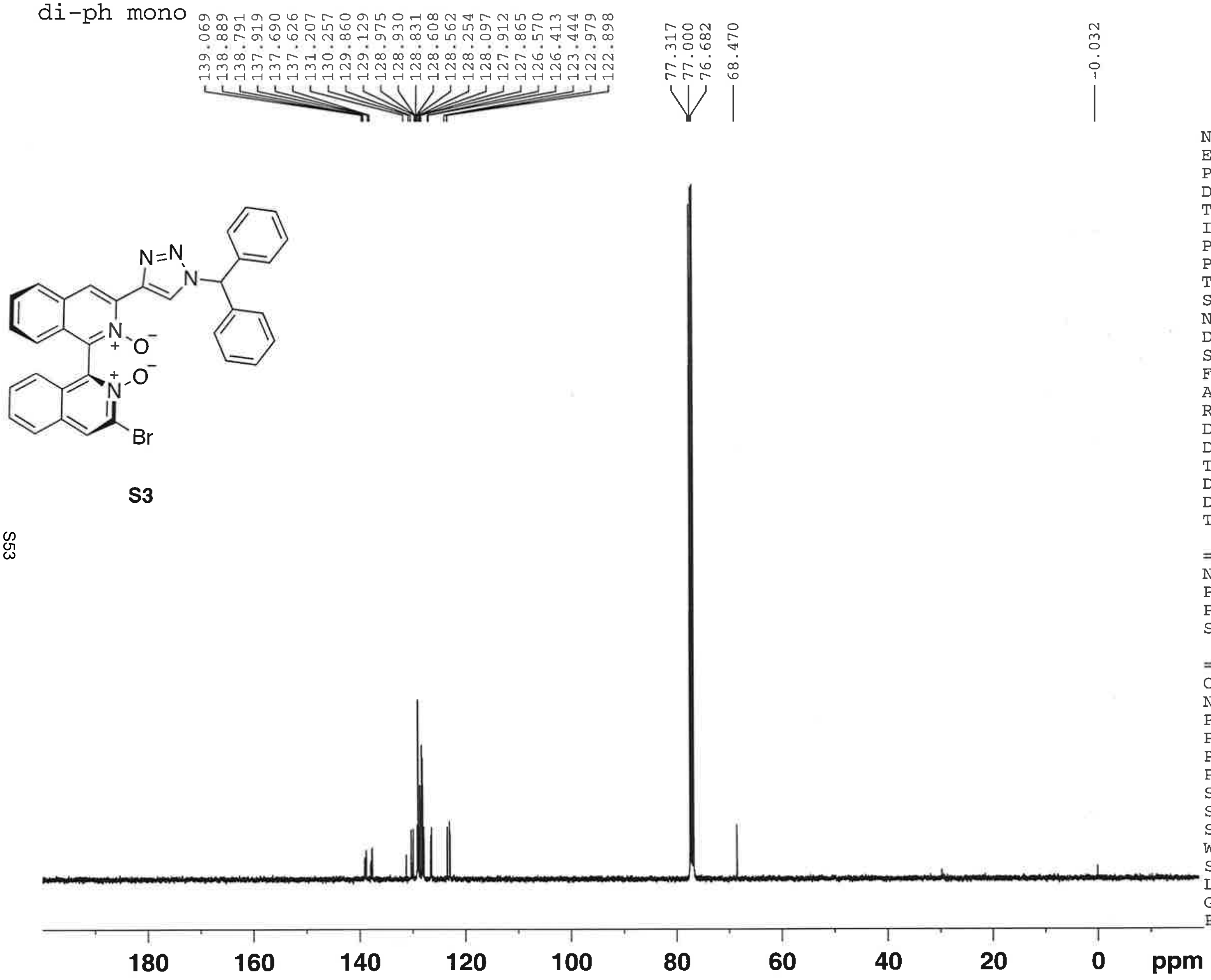


NAME	Mar11-2021
EXPNO	50
PROCNO	1
Date_	20210311
Time	13.36
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	256
DW	60.400 usec
DE	6.50 usec
TE	294.6 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	11.60 usec
PL1	3.00 dB
SFO1	400.1324710 MHz
SI	32768
SF	400.1300088 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

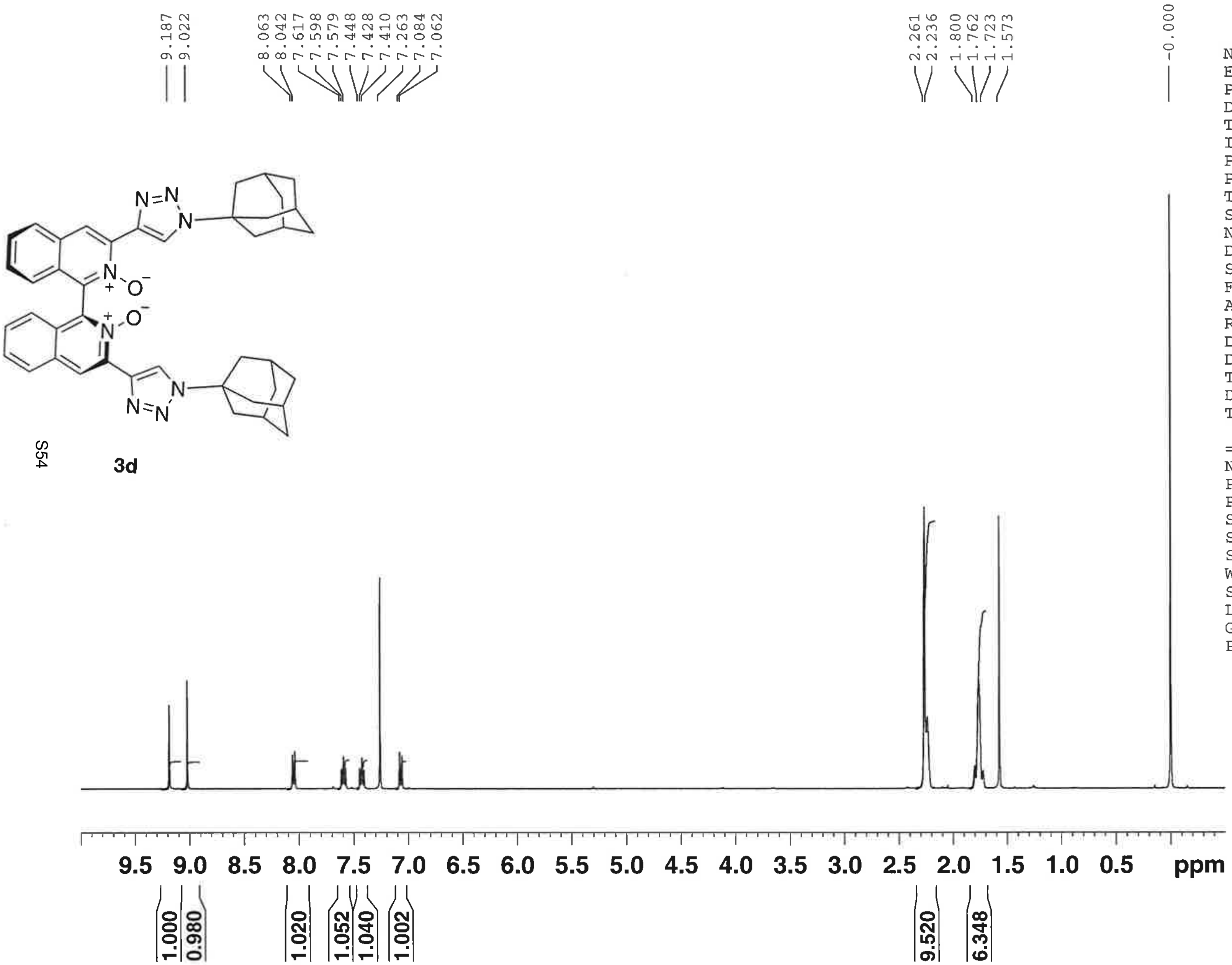
di-ph mono



S3

```
NAME             Mar11-2021
EXPNO                    91
PROCNO                    1
Date_              20210312
Time                   7.57
INSTRUM               spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                    12000
DS                        4
SWH               23980.814 Hz
FIDRES             0.365918 Hz
AQ                1.3664756 sec
RG                    32768
DW                   20.850 usec
DE                     6.50 usec
TE                    300.7 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    14.75 usec
PL1                    0.00 dB
SFO1            100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                   -2.00 dB
PL12                  12.54 dB
PL13                  15.00 dB
SFO2            400.1316005 MHz
SI                    32768
SF              100.6127709 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

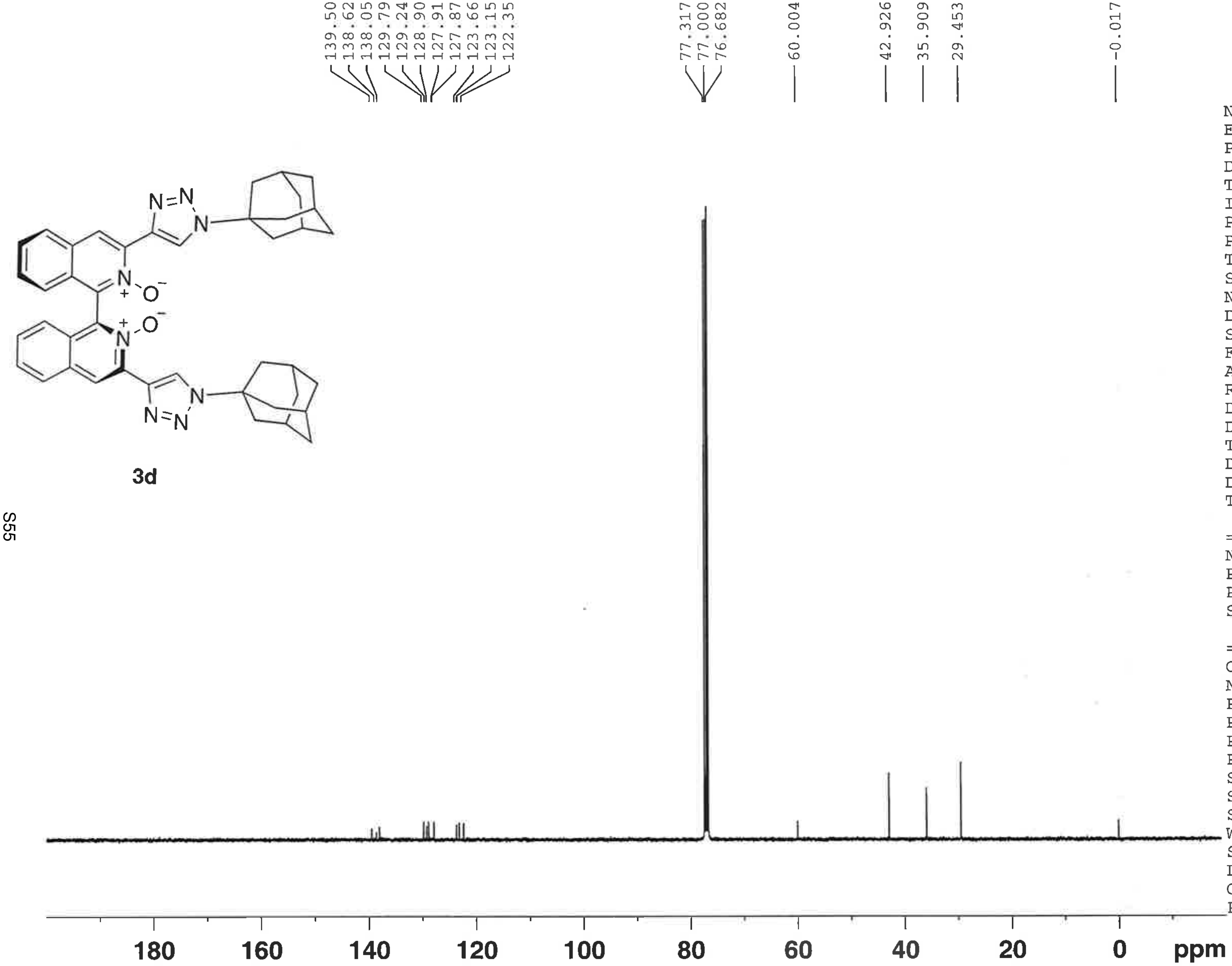
ad di



3d

```
NAME           Feb02-2021
EXPNO                  40
PROCNO                  1
Date_            20210202
Time                11.39
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                    362
DW                 60.400 usec
DE                   6.50 usec
TE                  294.4 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  11.60 usec
PL1                  3.00 dB
SFO1          400.1324710 MHz
SI                  32768
SF            400.1300085 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

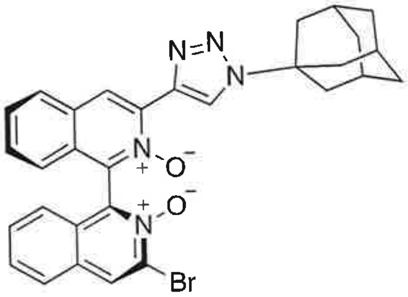
ad di couple



3d

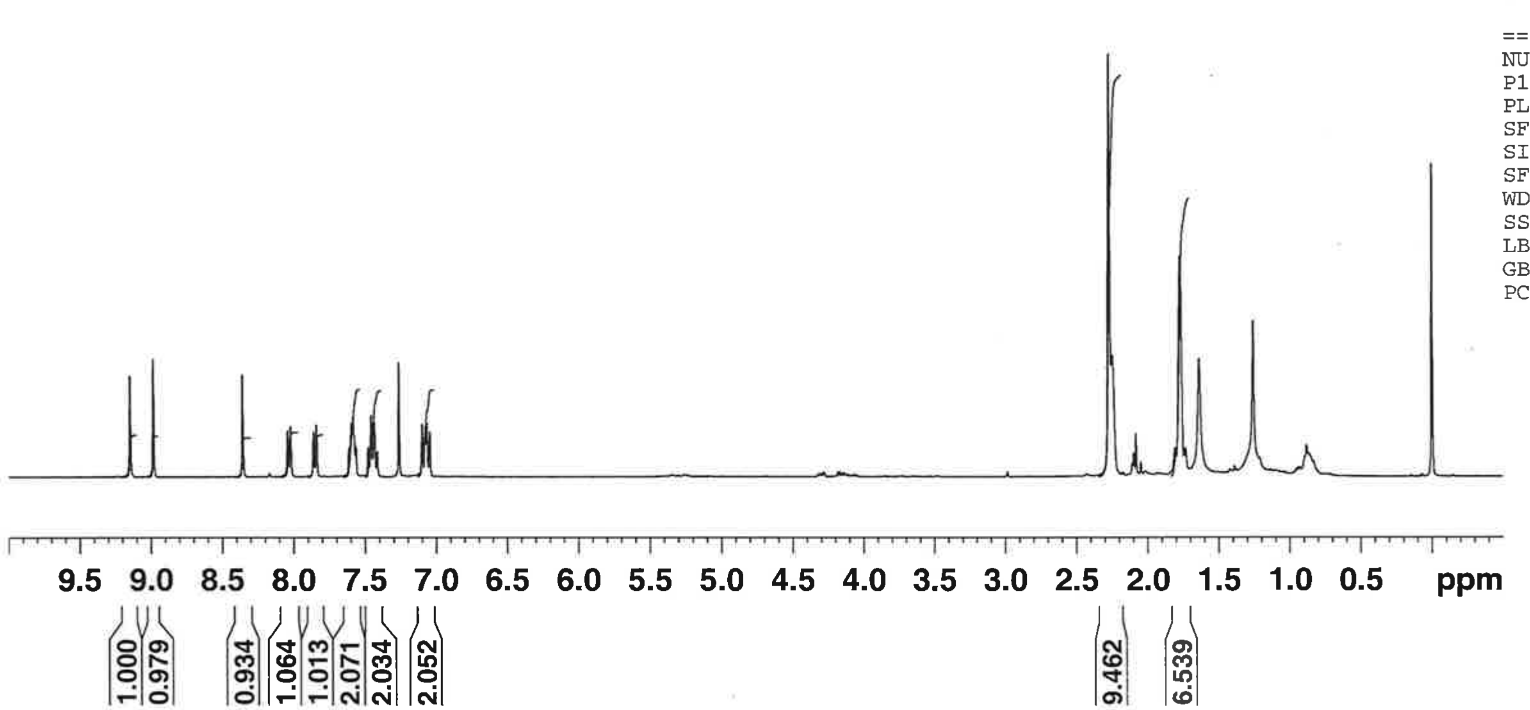
```
NAME          Feb01-2021
EXPNO                 81
PROCNO                 1
Date_           20210202
Time                7.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 12000
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                 32768
DW                20.850 usec
DE                  6.50 usec
TE                 300.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 14.75 usec
PL1                 0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               12.54 dB
PL13               15.00 dB
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127680 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

ad mono couple

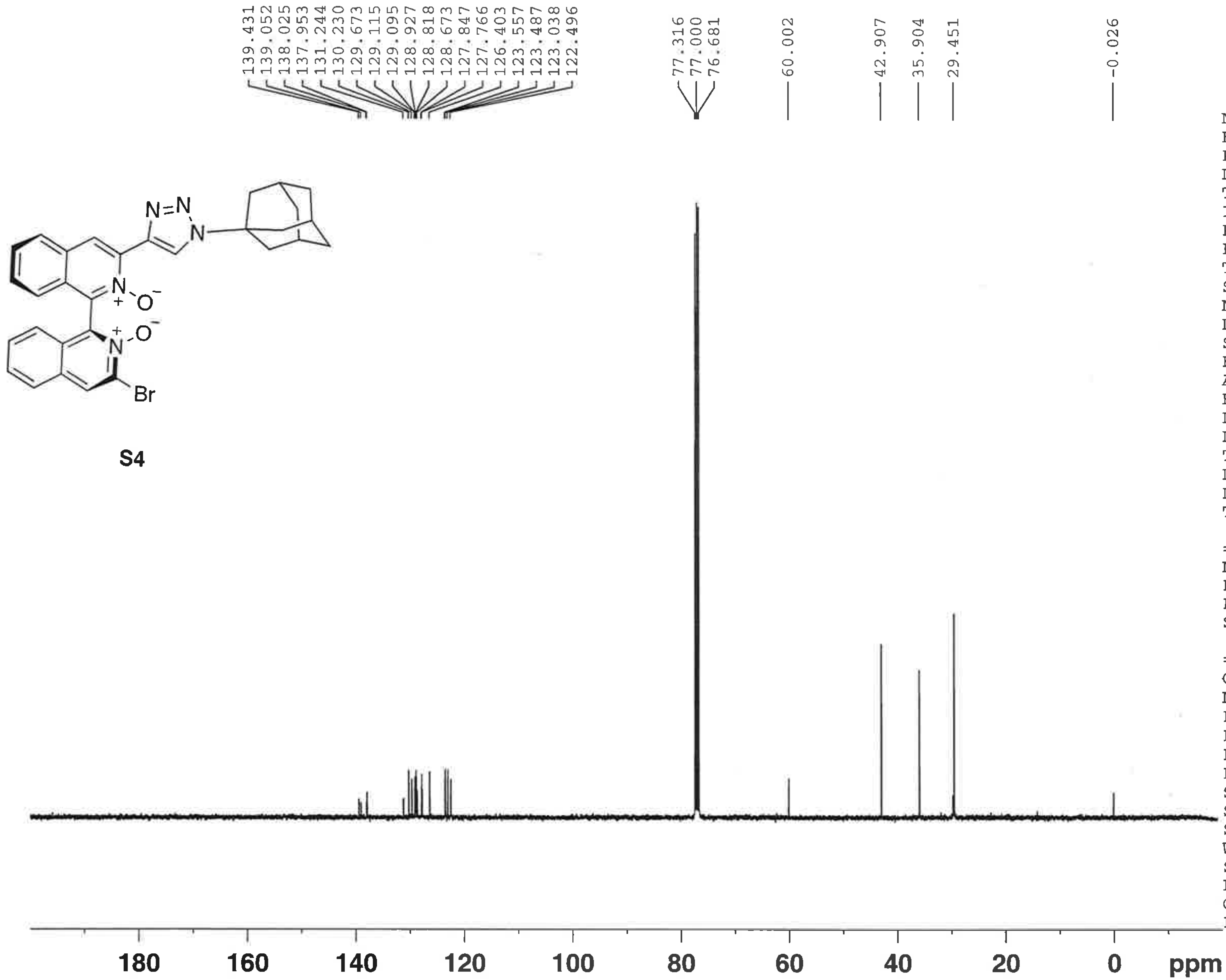


S4

```
NAME           Jan27-2021
EXPNO                  20
PROCNO                  1
Date_            20210127
Time                12.13
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                    256
DW                 60.400 usec
DE                   6.50 usec
TE                  295.7 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  11.60 usec
PL1                  3.00 dB
SFO1          400.1324710 MHz
SI                  32768
SF            400.1300079 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

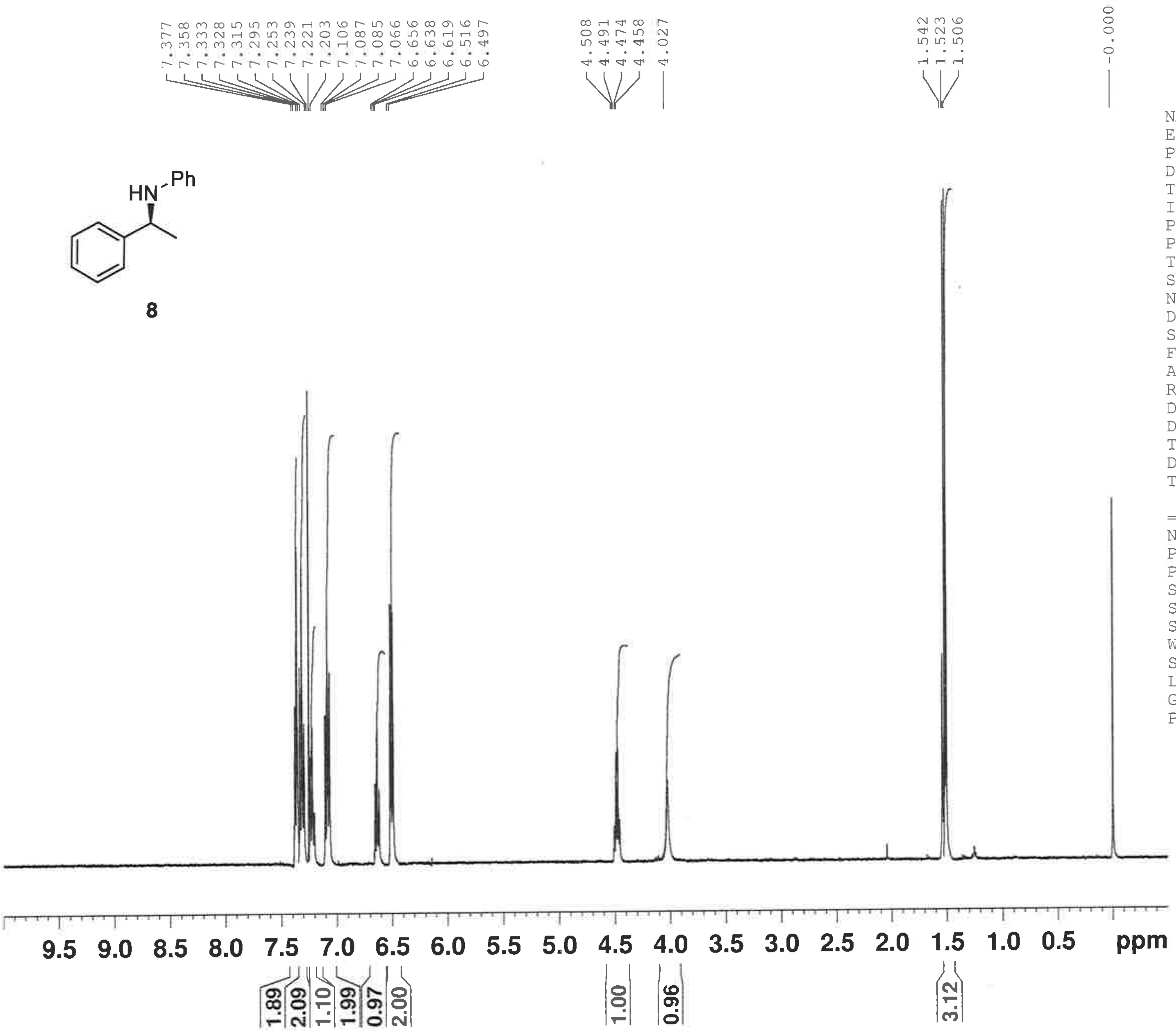


mono ad triazole biqno



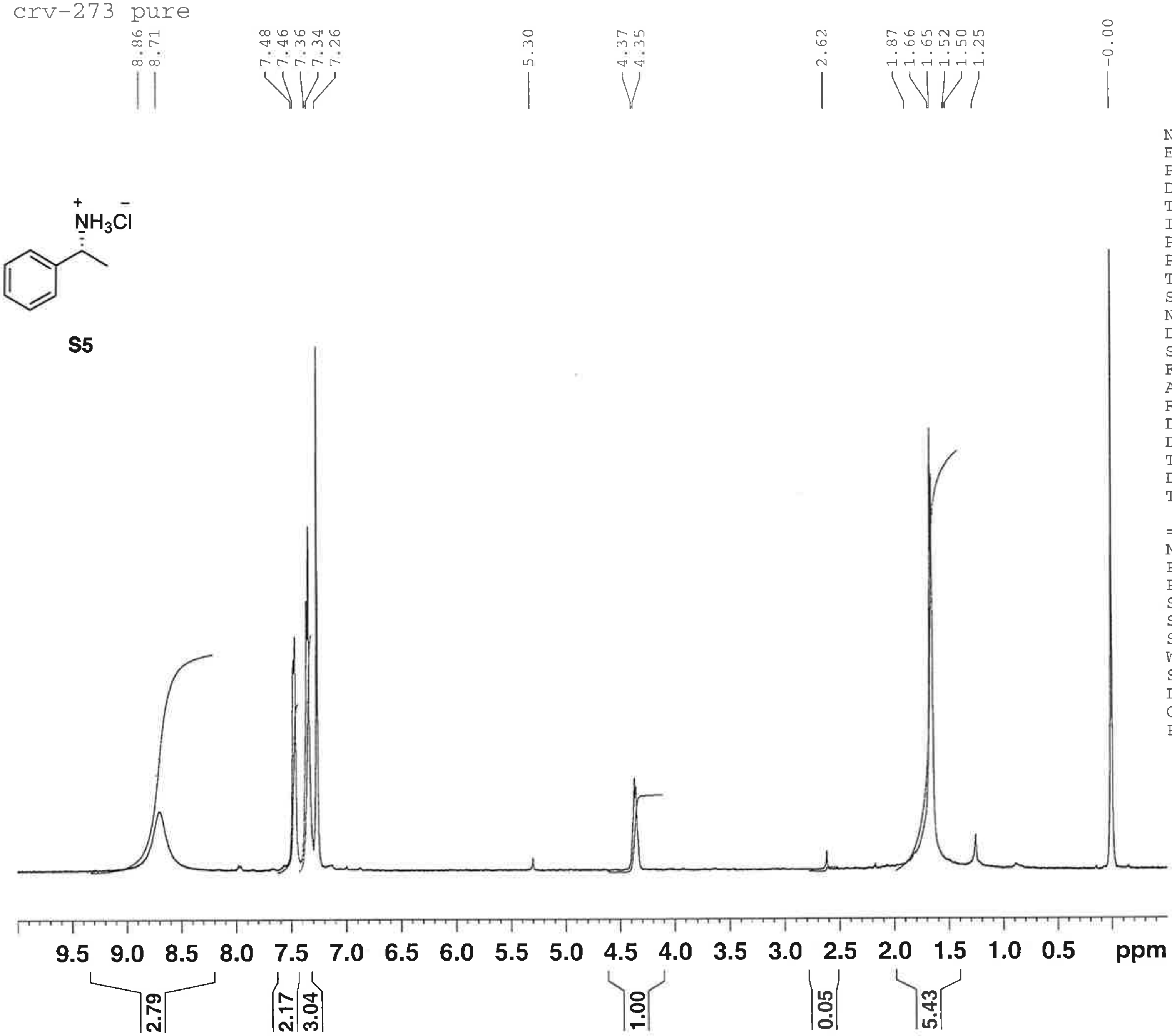
S4

```
NAME          Jan27-2021
EXPNO                130
PROCNO                 1
Date_           20210128
Time                6.57
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 12000
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                 32768
DW                20.850 usec
DE                  6.50 usec
TE                 300.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 14.75 usec
PL1                 0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               12.54 dB
PL13               15.00 dB
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127689 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



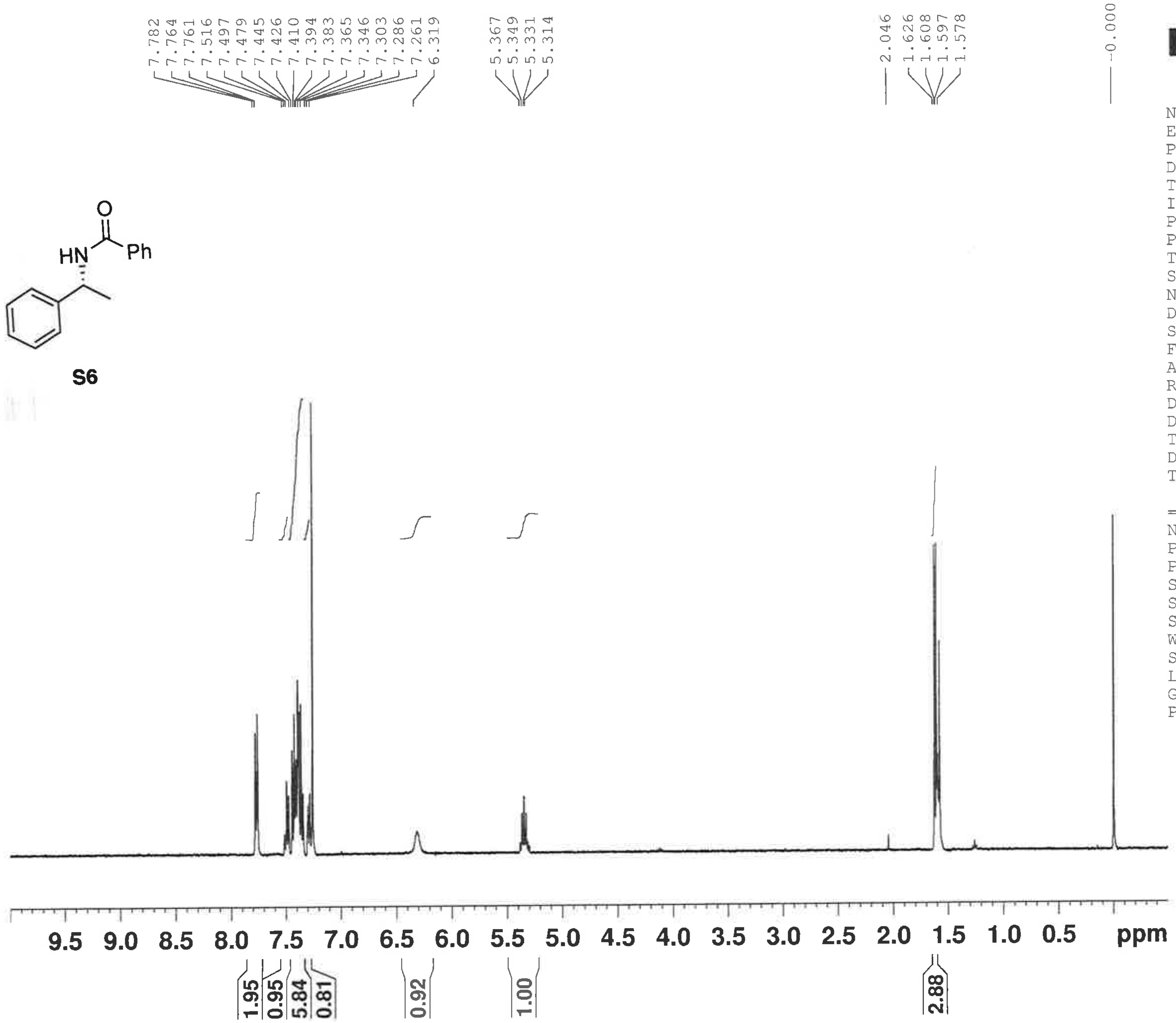
8

```
NAME       Aug04-2021-clr
EXPNO                   2
PROCNO                  1
Date_            20210804
Time                14.46
INSTRUM             spect
PROBHD   5 mm TBI 1H/31
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                14596.5
DW                 60.400 usec
DE                   6.50 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 =======
NUC1                   1H
P1                  18.75 usec
PL1                  0.50 dB
SFO1          400.1324710 MHz
SI                  32768
SF            400.1300122 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



S5

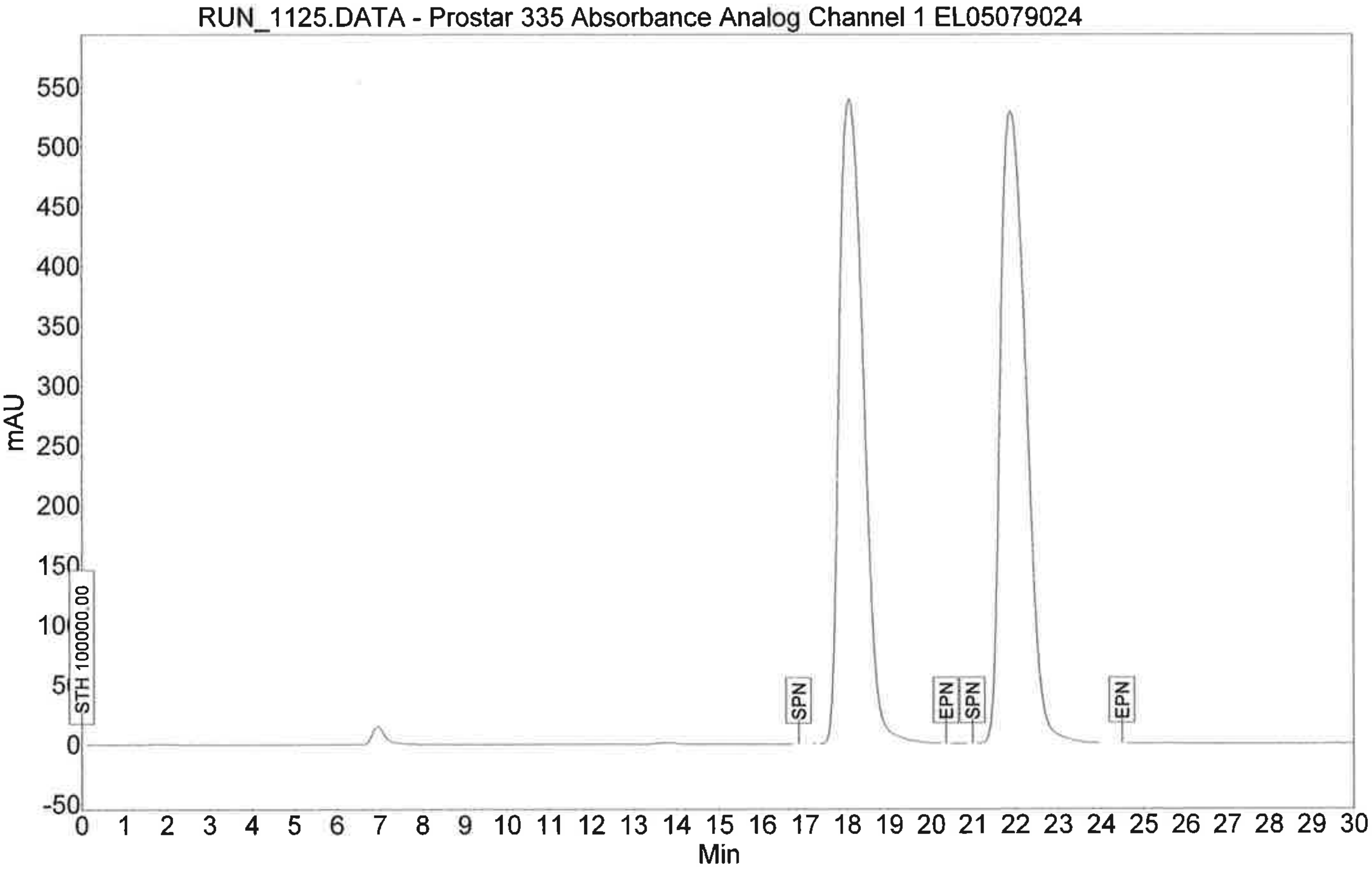
```
NAME          Oct09-2020
EXPNO                 20
PROCNO                 1
Date_           20201009
Time                8.59
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                   362
DW                60.400 usec
DE                  6.50 usec
TE                 295.8 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.60 usec
PL1                 3.00 dB
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300087 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



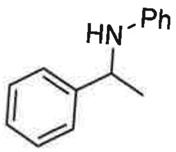
S6

BRUKER

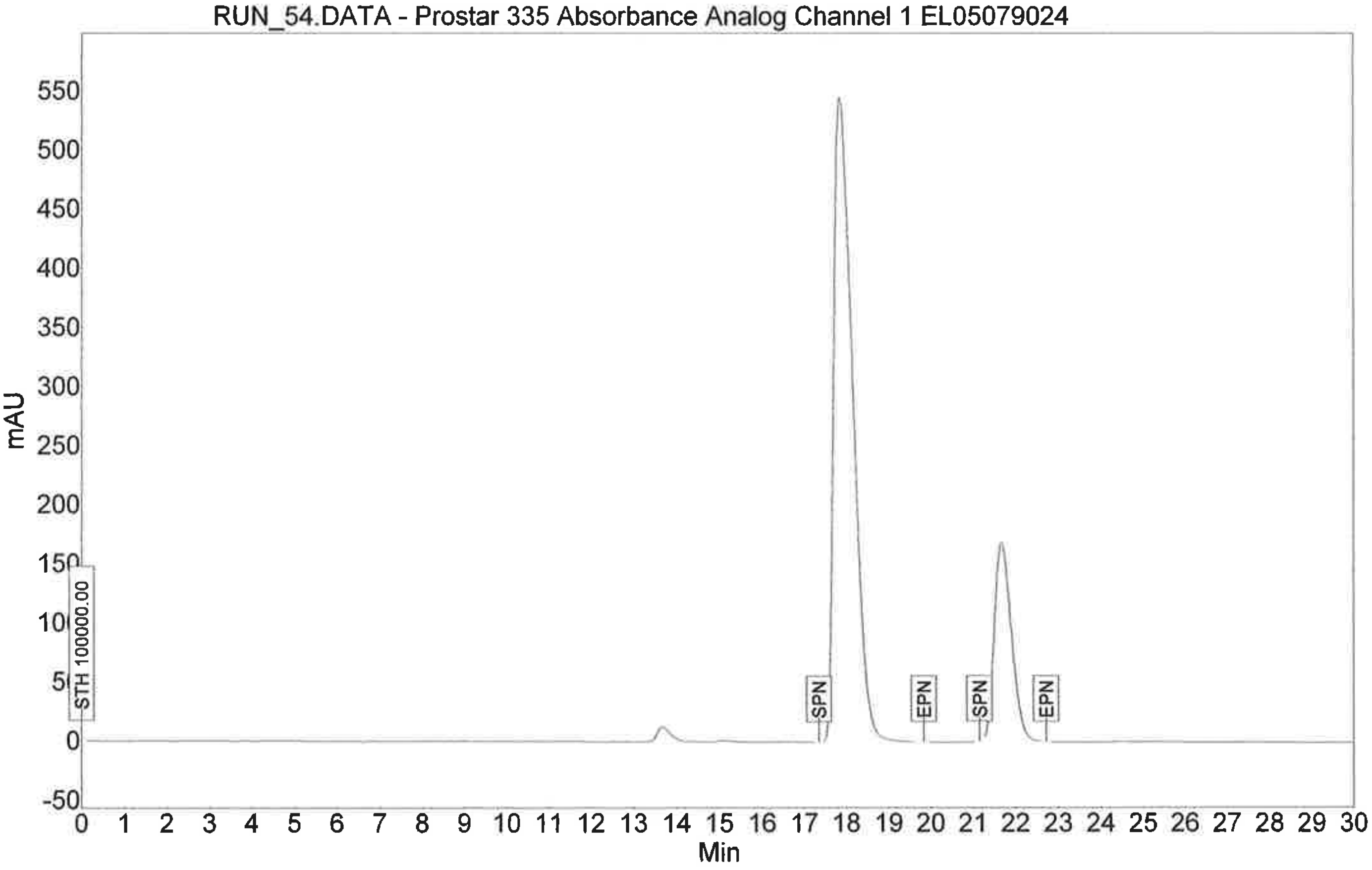
```
NAME           Aug05-2021
EXPNO                   3
PROCNO                  1
Date_            20210805
Time                15.59
INSTRUM             spect
PROBHD     5 mm TBI 1H/31
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                20642.5
DW                 60.400 usec
DE                   6.50 usec
TE                  300.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  18.75 usec
PL1                  0.50 dB
SFO1          400.1324710 MHz
SI                  32768
SF            400.1300092 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



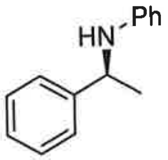
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	18.09	49.60	539.9	363.2	49.602
2	UNKNOWN	21.91	50.40	529.4	369.0	50.398
Total			100.00	1069.4	732.2	100.000



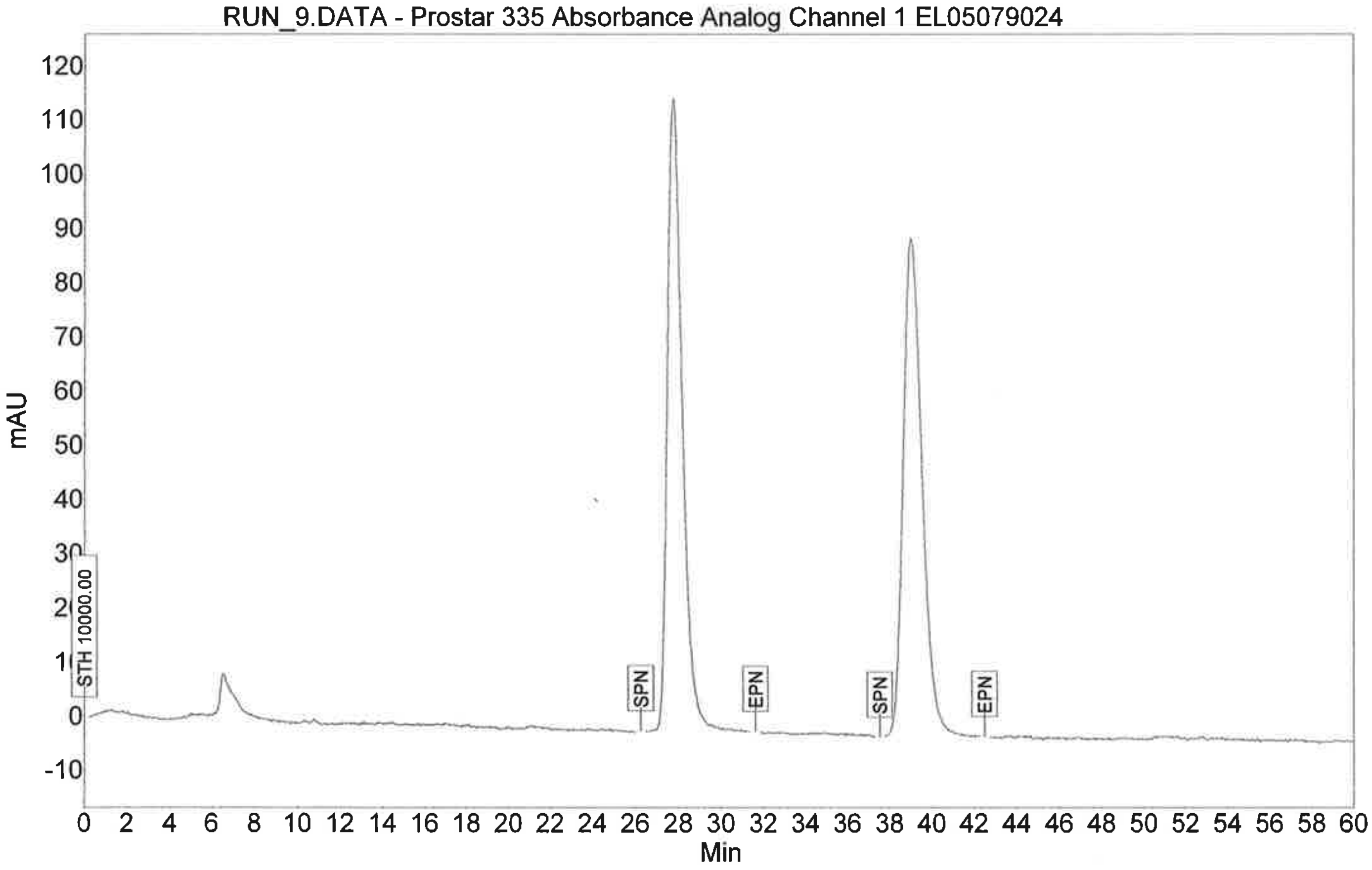
8



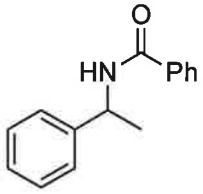
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	17.85	78.01	545.6	283.0	78.014
2	UNKNOWN	21.67	21.99	168.7	79.8	21.986
Total			100.00	714.3	362.8	100.000



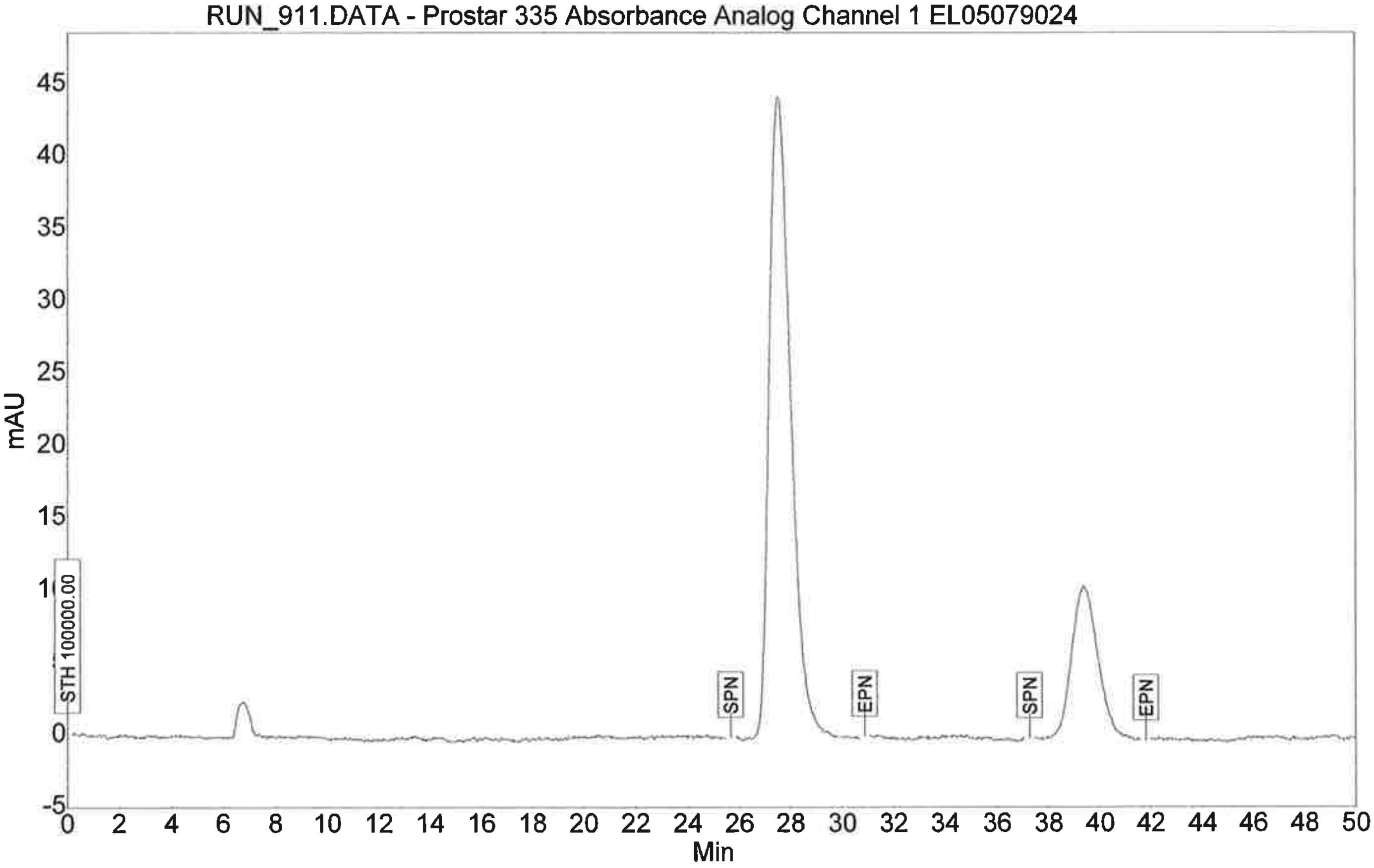
8



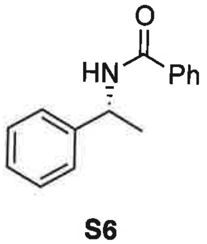
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	27.77	49.81	116.8	94.5	49.806
2	UNKNOWN	39.03	50.19	92.1	95.3	50.194
Total			100.00	208.8	189.8	100.000



S6



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	27.53	76.98	44.3	43.3	76.981
2	UNKNOWN	39.36	23.02	10.7	13.0	23.019
Total			100.00	55.0	56.3	100.000



S6