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

Supporting Information

A formal [4+2] annulation of diamines and prop-2-ynyl sulfonium salts: for the synthesis of tetrahydroquinoxalines

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Table of Contents

Part I Experimental part

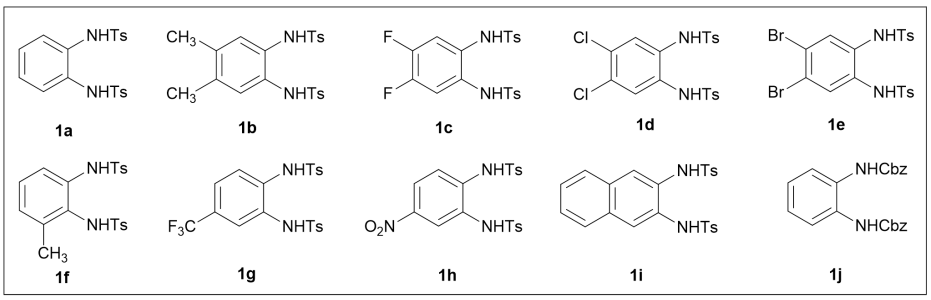
1. General information

Unless otherwise indicated, commercial reagents and solvents were used as received without further purification. Anhydrous THF and toluene were distilled from sodium and benzophenone. Anhydrous CH_2Cl_2 was distilled from CaH_2. Column chromatography was performed on silica gel 200~300 mesh. All ^1H NMR (500 and 600MHz), ^{13}C NMR (100 and 125 MHz) spectra were recorded in $CDCl_3$, with tetramethylsilane as an internal standard and reported in parts per million (ppm, δ). ^1H NMR Spectroscopy splitting patterns were designated as singlet (s), doublet (d), triplet (t), quartet (q). Splitting patterns that could not be interpreted or easily visualized were designated as multiplet (m).

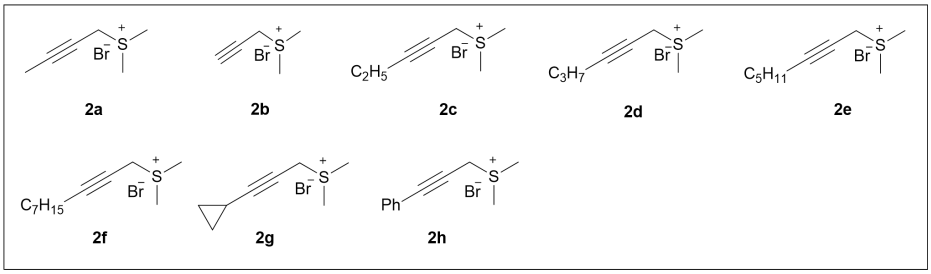
2. The Preparation of Substrates.

2.1. The substrates investigated

o-phenylendiamine derivatives：

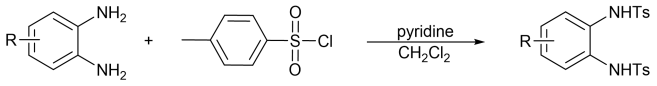


Prop-2-ynyl sulfonium salts：

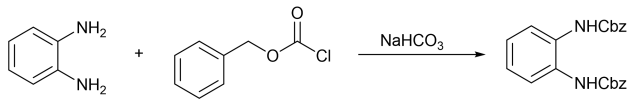


o-phenylendiamine derivatives **1a-1j**[1-2] and prop-2-ynyl sulfonium salts **2a-2b**,[3] **2g-2h**[4-5] are known compounds. Prop-2-ynyl sulfonium salts **2c-2f** were prepared according to the reported literature procedures.

2.2. The preparation of diamines



The *o*-phenylendiamines (5.0 mmol, 1 equiv) was dissolved in CH_2Cl_2 (15 mL), then to this mixture was added a solution of 4-toluene sulfonyl chloride (2.09 g, 2.2 equiv) and pyridine (1.2 mL, 3 equiv) in CH_2Cl_2 (20 mL). The mixture was heated at reflux overnight. The organic layer is washed three times successively by dilute hydrochloric acid, saturated $NaHCO_3$ and saturated NaCl. The organic layer was dried over Na_2SO_4, filtered, concentrated under reduced pressure and the residue was purified by chromatography on silica gel (petroleum ether/ethylacetate, typically 4/1) to give the desired product **1a**-**1i**.

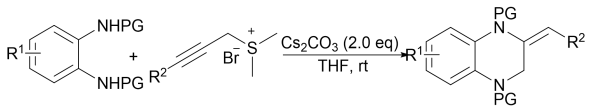


The o-phenylenediamine (0.54g, 5.0 mmol, 1 equiv), $NaHCO_3$ (0.84 g, 10 mmol, 2 equiv) and ethyl acetate/water (30mL/15mL) was added in a 100ml reaction flask. The mixture was stirred for 10 minutes, benzyl chloroformate (1.70 g, 10 mmol, 2 equiv) was added, After stirring for overnight, the organic layer was washed with saturated NaCl and dried over anhydrous Na_2SO_4. Then it was filtered and concentrated under reduced pressure. Purification by chromatography on silica gel (petroleum ether/EtOAc = 5/1) gave the desired product **1j** (1.5g, 80%yield).

2.3. The preparation of prop-2-ynyl sulfonium salts

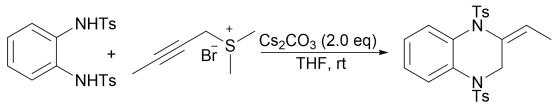
1-Bromopropl-2-yne (10 mmol, 1.0 equiv) is added in a 25ml reaction flask, then dimethyl sulfide (2.2g, 3.0equiv) and acetone (1mL) was added to this reaction flask.The mixture was stirred at room temperature for 24 h. The mixture was filtered by filling funnel to give the desired product **2a**-**2h**.

3. General procedure for the preparation of tetrahydroquinoxalines 3.



To an oven-dried 25 mL Schlenk tube equipped with a stir bar was charged with protected *o*-phenylendiamines **1** (0.2 mmol), 3-alkyl- or 3-aryl-substituted prop-2-ynyl sulfonium salts **2** (0.3 mmol) and anhydrous Cs_2CO_3 (130.4 mg, 0.40 mmol). To this mixture was added freshly distilled THF (2 mL). After stirring for overnight, the reaction mixture was concentrated and the residue was purified by chromatography on silica gel (petroleum ether/ethylacetate, typically 4/1) to give the desired product **3**.

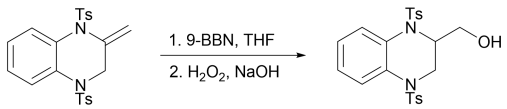
4 Procedure for the preparation of gram-scale reaction of 3aa



1a (2.08g, 5.0mmol) **2a** **3aa** (2.02, 86% yield)

To an oven-dried 100 mL Schlenk tube equipped with a stir bar was charged with sulfonyl protected *o*-phenylendiamine **1a** (5 mmol, 2.08g), but-2-ynyl sulfonium salt **2a** (7.5 mmol, 1.46g) and anhydrous Cs_2CO_3 (3.26 g, 10 mmol). To this mixture was added freshly distilled THF (40 mL). After stirring for overnight, the reaction mixture was concentrated and the residue was purified by chromatography on silica gel (petroleum ether/ethylacetate = 4/1) to give the desired product **3aa** (2.02g, 86%yield).

5. Synthetic transformation

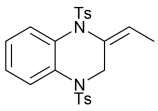


3ab **4**

To a solution of **3ab** (90.8 mg, 0.20 mmol) in 2 mL THF was added 9-BBN (0.6 mL, 0.3 mmol, 0.5 mol/L in THF) dropwise at 0℃. The mixture was stirred at room temperature until the complete consumption of **3ab**. Then NaOH (30 mg) and 0.1 mL H_2O_2 was added and quenched with $Na_2S_2O_3$ after 1h. The mixture was extracted with CH_2Cl_2. The organic layer was washed with brine, dried over $MgSO_4$, filtered and

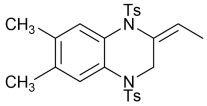
concentrated and purified by chromatography on silica gel (petroleum ether/ethyl acetate = 2/1) to afford the desired product **4**.

6. Characterization data



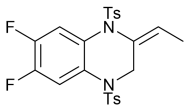
3aa

(E)-2-ethylidene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3aa**, white solid, mp 123-124 °C, 88.9 mg, 95% yield; R_f = 0.4 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79-7.77 (m, 1H), 7.70-7.68 (m, 1H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.20-7.15 (m, 4H), 7.12 (d, *J* = 8.0 Hz, 2H), 5.89 (q, *J* = 14.5 Hz, *J* = 7.0 Hz, 1H), 3.82 (s, 2H), 2.40 (s, 3H), 2.35 (s, 3H), 1.73 (d, *J* = 7.5 Hz, 3H);^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 144.1, 135.5, 134.6, 129.8, 129.5, 129.4, 127.6, 127.1, 126.7, 125.8, 125.6, 125.1, 122.8, 42.6, 21.6, 21.5, 13.4; HRMS (ESI) calcd for $C_{24}H_{25}N_2O_4S_2[M+H]^+$: 469.1250, found 469.1246.



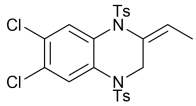
3ba

(E)-2-ethylidene-6,7-dimethyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ba**, white solid, mp 118-119°C, 89.3 mg, 90% yield; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (s, 2H), 7.46 (s, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.5 Hz, 2H), 5.84 (q, *J* = 14.5 Hz, *J* = 7.0 Hz, 1H), 3.76 (s, 2H), 2.40 (s, 3H), 2.35 (s, 3H), 2.26 (s, 3H), 2.24 (s, 3H), 1.68 (d, *J* = 7.5 Hz, 3H);^{13}C NMR (125 MHz, $CDCl_3$) δ 144.4, 143.9, 135.6, 134.8, 134.5, 133.8, 129.7, 129.3, 127.6, 127.1, 127.0, 126.3, 126.2, 123.5, 42.7, 21.6, 21.5, 19.6, 19.5, 13.4; HRMS (ESI) calcd for $C_{26}H_{29}N_2O_4S_2[M+H]^+$: 497.1563, found 497.1555.



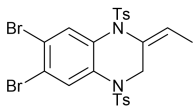
3ca

(*E*)-2-ethylidene-6,7-difluoro-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ca**, white solid, mp 133-134°C; 87.7 mg, 87% yield; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.67 (s, 1H), 7.35 (d, *J* = 5.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 6.6 Hz, 2H), 7.16 (d, *J* = 7.8 Hz, 2H), 5.90 (d, *J* = 9.6 Hz, 1H), 3.70 (s, 2H), 2.42 (s, 3H), 2.37 (s, 3H), 1.73 (d, *J* = 4.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 148.1 (d, *J* = 13.5 Hz), 147.8 (d, *J* = 13.5 Hz), 146.5 (d, *J* = 13.5 Hz), 146.2 (d, *J* = 13.5 Hz), 145.1, 144.6, 134.8, 134.0, 130.0, 129.6, 127.9, 127.6, 127.1, 125.3, 114.0 (d, *J* = 22.5 Hz), 111.8 (d, *J* = 22.5 Hz), 41.5, 21.6, 13.5; ^{19}F NMR (565 MHz, $CDCl_3$) δ -138.2--138.3(m, 2F); HRMS (ESI) calcd for $C_{24}H_{23}F_2N_2O_4S_2[M+H]^+$: 505.1062, found 505.1054.



3da

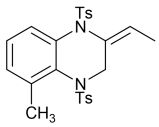
(E)-2-ethylidene-6,7-dichloro-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3da**, white solid, mp 135-136°C; 91.4 mg, 85%; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.90 (s, 1H), 7.37 (d, *J* = 4.8 Hz, 2H), 7.31 (d, *J* = 4.8 Hz, 2H), 7.24 (d, *J* = 5.4 Hz, 2H), 7.17 (d, *J* = 5.4 Hz, 2H), 5.91 (d, *J* = 4.2 Hz, 1H), 3.72 (s, 2H), 2.42 (s, 3H), 2.38 (s, 3H), 1.73 (d, *J* = 8.4 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 145.1, 144.7, 134.8, 134.0, 130.0, 129.6, 129.2, 128.4, 128.3, 128.1, 127.7, 127.1, 126.2, 125.3, 123.7, 41.6, 21.7, 21.6, 13.5; HRMS (ESI) calcd for $C_{24}H_{23}Cl_2N_2O_4S_2[M+H]^+$: 537.0471, found 537.0466.



3ea

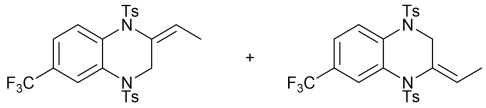
(*E*)-2-ethylidene-6,7-dibromo-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ea**, white solid, mp 161-162 °C; 110.6 mg, 88% yield; R_f = 0.4 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (s, 1H), 8.05 (s, 1H), 7.37 (*J* = 8.5 Hz, 2H), 7.31 (d, *J* = 8.5 Hz, 2H), 7 .24 (d, *J* = 8.5 Hz, 2H), 7 .17 (d, *J* = 8.5 Hz, 2H), 5.91 (q, *J* = 14.5 Hz, *J* = 6.0 Hz, 1H), 3.72 (s, 2H), 2.43 (s, 3H), 2.38 (s, 3H), 1.73 (d, *J* = 7.5 Hz,

3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.2, 144.8, 134.8, 134.1, 130.0, 129.7, 129.2, 128.9, 127.7, 126.7, 125.3, 121.0, 120.1, 41.6, 21.7, 21.6, 13.6; HRMS (ESI) calcd for $C_{24}H_{22}Br_2N_2O_4S_2[M+H]^+$: 624.9461, found 624.9452.



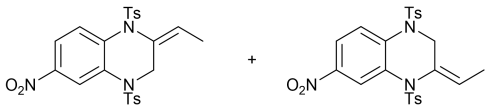
3fa

(*E*)-2-ethylidene-5-methyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3fa**, white solid, r.r >20:1 on the crude reaction mixture, mp 121-122 °C; 86.1 mg, 89%; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.5 Hz, 2H), 7.64 (d, *J* = 8.5 Hz, 2H), 7.32-7.28 (m, 4H), 7.21 (d, *J* = 7.0 Hz, 1H), 7.07-7.01 (m, 2H), 5.96-5.90 (q, *J* = 17.0 Hz, *J* = 9.5 Hz, 1H), 4.66 (d, *J* = 14.5 Hz, 1H), 3.93 (d, *J* = 14.5 Hz, 1H), 2.47 (s, 3H), 2.45 (s, 3H), 2.39 (s, 3H), 1.64 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.2, 145.1, 144.1, 143.8, 142.4, 141.4, 138.2, 133.2, 131.3, 130.9, 129.7, 129.5, 129.4, 129.0, 126.1, 125.4, 124.9, 66.1, 21.6, 21.5, 16.6, 13.1; HRMS (ESI) calcd for $C_{25}H_{27}N_2O_4S_2[M+H]^+$: 483.1407, found 483.1408.



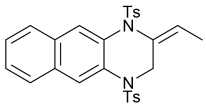
3ga **3ga***

(*E*)-2-ethylidene-1,4-ditosyl-6-(trifluoromethyl)-1,2,3,4-tetrahydroquinoxaline **3ga**, and (*E*)-2-ethylidene-1,4-ditosyl-7-(trifluoromethyl)-1,2,3,4-tetrahydroquinoxaline **3ga***. A mixture of two isomers, r.r = 4:1on the crude reaction mixture; white solid, mp 146-148 °C; 89.2 mg, 83%. R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.84 (d, *J* = 9.0 Hz, 1H), 7.38-7.32 (m, 5H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 5.98-5.93 (m, 1H), 3.87 (s, 2H), 2.42 (s, 3H), 2.37 (s, 3H), 1.74 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.0, 144.7, 135.1, 134.3, 132.2, 130.0, 129.7, 129.6, 129.0, 127.8, 127.7, 127.6, 127.2, 127.1, 125.8, 122.8 (d, *J* = 3.75 Hz), 122.4, 122.3 (d, *J* = 3.75 Hz), 42.4, 41.7, 21.7, 21.6, 13.5; ^{19}F NMR (565 MHz, $CDCl_3$) δ -62.3, -62.4; HRMS (ESI) calcd for $C_{25}H_{24}F_3N_2O_4S_2[M+H]^+$: 537.1130, found 537.1130.



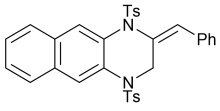
3ha **3ha***

(*E*)-2-ethylidene-6-nitro-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ha** and (*E*)-2-ethylidene-7-nitro-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ha***. A mixture of two isomers, r.r = 10:1 on the crude reaction mixture; white solid, mp 149-151 °C; 82.3 mg, 80%. R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 8.0 Hz, 1H), 7.97 (q, *J* = 9.0 Hz, *J* = 2.5 Hz, 1H), 7.90 (d, *J* = 9.5 Hz, 1H), 7.42 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H), 7.25 (d, *J* = 9.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 5.98 (q, *J* = 14.5 Hz, *J* = 7.5 Hz, 1H), 3.92 (s, 2H), 2.43 (s, 3H), 2.38 (s, 3H), 1.77 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.3, 145.1, 143.6, 134.8, 134.7, 134.1, 130.1, 129.9, 128.7, 128.6, 127.8, 127.1, 125.3, 121.7, 121.1, 120.4, 42.4, 21.7, 21.6, 13.6; HRMS (ESI) calcd for $C_{24}H_{24}N_3O_6S_2[M+H]^+$: 514.1107, found 514.1107.



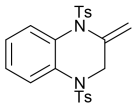
3ia

(*E*)-2-ethylidene-1,4-ditosyl-1,2,3,4-tetrahydrobenzo[g]quinoxaline **3ia**, white solid, mp 167-168 °C; 86.1 mg, 83% yield; R_f = 0.3 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.17 (s, 1H), 8.04 (s, 1H), 7.84-7.81 (m, 1H), 7.76-7.74 (m, 1H), 7.50-7.44 (m, 2H), 7.40-7.37 (m, 4H), 7.18 (d, *J* = 10.0 Hz, 2H), 7.13 (d, *J* = 10.0 Hz, 2H), 5.98 (q, *J* = 18.5 Hz, *J* = 9.0 Hz, 1H), 4.05 (s, 2H), 2.40 (s, 3H), 2.34 (s, 3H), 1.75 (d, *J* = 9.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.5, 144.2, 135.7, 135.2, 131.2, 130.5, 129.9, 129.6, 128.7, 128.5, 127.8, 127.5, 127.1, 126.4, 126.3, 126.0, 123.9, 119.9, 43.9, 21.6, 21.5, 13.4; HRMS (ESI) calcd for $C_{28}H_{26}N_2O_4S_2Na[M+Na]^+$: 541.1226, found 541.1221.



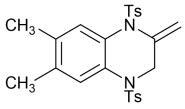
3ih

(*E*)-2-benzylidene-1,4-ditosyl-1,2,3,4-tetrahydrobenzo[g]quinoxaline **3ig**, white solid, mp 156-157 °C; 91.7 mg, 79%; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (s, 1H), 8.07 (s, 1H), 7.87-7.85 (m, 1H), 7.78-7.76 (m, 1H), 7.49-7.41 (m, 6H), 7.38-7.34 (m, 1H), 7.24-7.20 (m, 4H), 7.15 (d, *J* = 7.5 Hz, 2H), 7.06 (d, *J* = 8.0 Hz, 2H), 6.96 (s, 1H), 4.31 (s, 2H), 2.42 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.8, 144.1, 135.5, 135.2, 134.2, 131.3, 130.5, 130.4, 130.0, 129.6, 129.1, 128.8, 128.5, 128.4, 128.4, 128.3, 127.9, 127.8, 127.6, 127.2, 126.5, 126.1, 124.1, 119.9, 44.7, 21.7, 21.6; HRMS (ESI) calcd for $C_{33}H_{29}N_2O_4S_2[M+H]^+$: 581.15697, found 581.1569.



3ab

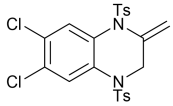
2-methylene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ab**, white solid, mp 110-111 °C; 80.1 mg, 88% yield; R_f = 0.4 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (q, *J* = 7.5 Hz, *J* = 2.0 Hz, 1H), 7.67 (q, *J* = 7.5 Hz, *J* = 2.0 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.21 - 7.18 (m, 3H), 7.17-7.14 (m, 3H), 5.33 (s, 1H), 4.95 (s, 1H), 3.78 (s, 2H), 2.39 (s, 3H), 2.36 (s, 3H);^{13}C NMR (125 MHz, $CDCl_3$) δ 144.8, 144.2, 135.4, 134.4, 134.3, 129.8, 129.7, 129.6, 129.5, 127.6, 127.3, 125.7, 125.4, 125.2, 123.4, 112.7, 48.0, 21.6, 21.5; HRMS (ESI) calcd for $C_{23}H_{23}N_2O_4S_2[M+H]^+$: 455.1094, found 455.1086.



3bb

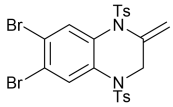
6,7-dimethyl-2-methylene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3bb**, white solid, mp 109-110 °C; 83.9 mg, 87% yield; R_f = 0.4 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (s, 1H), 7.34 (s, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 5.26 (s, 1H), 4.87 (s, 1H), 3.70 (s, 2H), 2.39 (s, 3H), 2.36 (s, 3H), 2.27 (s, 3H), 2.25 (s, 3H);^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 144.0, 135.6, 134.6, 134.5, 134.4, 134.2, 129.7, 129.5, 127.6, 127.4, 127.3, 126.9, 125.7, 124.1, 112.3, 48.1, 21.6, 19.6; HRMS (ESI) calcd

for $C_{25}H_{27}N_2O_4S_2[M+H]^+$: 483.1407, found 483.1404.



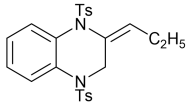
3db

6,7-dichloro-2-methylene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3db**, white solid, mp 110-111°C; 88.6 mg, 85% yield; R_f = 0.4 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.89 (s, 1H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 5.34 (s, 1H), 5.00 (s, 1H), 3.63 (s, 2H), 2.42 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.4, 144.8, 134.7, 133.7, 132.5, 130.0, 129.8, 129.1, 129.0, 128.5, 128.0, 127.7, 127.4, 125.6, 124.6, 115.0, 47.0, 21.7, 21.6; HRMS (ESI) calcd for $C_{23}H_{21}Cl_2N_2O_4S_2[M+H]^+$: 523.0314, found 523.0314.



3eb

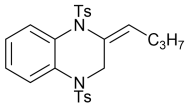
6,7-dibromo-2-methylene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline, white solid, mp 114-115 °C; 104.8 mg, 86%. R_f = 0.4 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.16 (s, 1H), 8.03 (s, 1H), 7.40 (d, *J* = 8.5 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 5.34 (s, 1H), 4.99 (s, 1H), 3.63 (s, 2H), 2.42 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.4, 144.9, 134.7, 133.7, 132.4, 130.0, 129.8, 129.1, 128.6, 127.7, 127.5, 120.8, 120.7, 115.0, 47.0, 21.7, 21.6; HRMS (ESI) calcd for $C_{23}H_{21}Br_2N_2O_4S_2[M+H]^+$: 610.9304, found 610.9287.



3ac

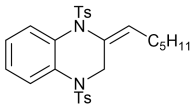
(*E*)-2-propylidene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ac**, white solid, mp 116-117 °C; 88.9 mg, 92% yield; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79-7.77 (m, 1H), 7.70-7.68 (m, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.16-7.12 (m, 4H), 5.87 (t, *J* = 16.0

Hz, J = 8.0 Hz, 1H), 3.84 (s, 2H), 2.40 (s, 3H), 2.35 (s, 3H), 2.14-2.08 (m, 2H), 1.05 (t, J = 15.0 Hz, J = 7.5 Hz, 3H);^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 144.1, 135.5, 134.7, 133.4, 129.8, 129.7, 129.5, 129.4, 127.7, 127.1, 125.9, 125.7, 125.0, 122.5, 43.0, 21.6, 21.5, 21.2, 13.7; HRMS (ESI) calcd for $C_{25}H_{27}N_2O_4S_2[M+H]^+$: 483.1407, found 483.1400.



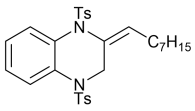
3ad

(*E*)-2-butylidene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ad**, white solid, mp 121-122 °C; 88.5 mg, 89% yield; R_f = 0.6 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78-7.77 (m, 1H), 7.69-7.67 (m, 1H), 7.35 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 7.19 (d, J = 8.0 Hz, 2H), 7.16-7.12 (m, 4H), 5.87 (t, J = 15.5 Hz, J = 7.5 Hz, 1H), 3.87 (s, 2H), 2.40 (s, 3H), 2.35 (s, 3H), 2.06 (q, J = 15.0 Hz, J = 7.5 Hz, 2H), 1.50-1.43 (m, 2H), 0.99 (t, J = 15.0 Hz, J = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 144.1, 135.5, 134.8, 132.0, 129.8, 129.7, 129.5, 127.6, 127.1, 126.2, 125.8, 124.9, 122.4, 43.1, 29.9, 22.5, 21.6, 21.5, 13.8; HRMS (ESI) calcd for $C_{26}H_{29}N_2O_4S_2[M+H]^+$: 497.1563, found 497.1558.



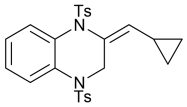
3ae

(*E*)-2-hexylidene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ae**, white solid, mp 144-145 °C; 94.6 mg, 90% yield; R_f = 0.6 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1H), 7.69-7.67 (m, 1H), 7.35 (d, J = 8.5 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 7.19 (d, J = 8.0 Hz, 2H), 7.16-7.12 (m, 4H), 5.87 (t, J = 15.5 Hz, J = 7.5 Hz, 1H), 3.86 (s, 2H), 2.40 (s, 3H), 2.35 (s, 3H), 2.07 (q, J = 15.0 Hz, J = 7.5 Hz, 2H), 1.44-1.41 (m, 2H), 1.37-1.33 (m, 4H), 0.94 (t, J = 13.0 Hz, J = 6.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 144.1, 135.5, 134.8, 132.2, 129.8, 129.7, 129.5, 127.6, 127.1, 126.0, 125.9, 124.9, 122.4, 43.1, 31.4, 28.8, 27.8, 22.5, 21.6, 21.5, 14.1; HRMS (ESI) calcd for $C_{28}H_{33}N_2O_4S_2[M+H]^+$: 525.1876, found 525.1873.



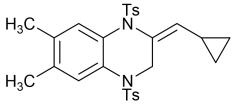
3af

(*E*)-2-octylidene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3af**, white solid, mp 126-127 °C; 100.7 mg, 91% yield; R_f = 0.6 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1H), 7.69-7.67 (m, 1H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.16-7.12 (m, 4H), 5.87 (t, *J* = 15.5 Hz, *J* = 7.5 Hz, 1H), 3.86 (s, 2H), 2.40 (s, 3H), 2.35 (s, 3H), 2.09-2.04 (m, 2H), 1.43-1.40 (m, 2H), 1.34-1.31 (m, 8H), 0.91 (t, *J* = 13.5 Hz, *J* = 6.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 144.1, 135.5, 134.8, 132.2, 129.8, 129.7, 129.5, 127.6, 127.1, 126.0, 125.9, 124.9, 122.4, 43.1, 31.8, 29.2, 29.1, 27.9, 22.7, 21.6, 21.5, 14.1; HRMS (ESI) calcd for $C_{30}H_{37}N_2O_4S_2[M+H]^+$: 553.2189, found 553.2181.



3ag

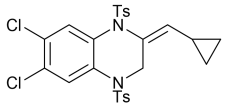
3-cyclopropylidene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ag**, white solid, mp 126-127 °C; 84.9 mg, 88% yield; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81-7.77 (m, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H), 7.20-7.15 (m, 4H), 7.08 (d, *J* = 8.0 Hz, 2H), 5.17 (d, *J* = 10.5 Hz, 1H), 3.84 (s, 2H), 2.40 (s, 3H), 2.33 (s, 3H), 1.54-1.51 (m, 1H), 0.97-0.93 (m, 2H), 0.53-0.50 (m, 2H);^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 144.0, 137.0, 135.1, 134.4, 129.7, 129.4, 129.3, 128.7, 127.7, 127.4, 125.5, 125.3, 125.2, 123.4, 123.1, 42.5, 21.6, 21.5, 10.3, 7.7; HRMS (ESI) calcd for $C_{26}H_{27}N_2O_4S_2[M+H]^+$: 495.1407, found 495.1401.



3bg

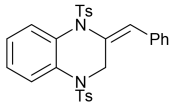
(*E*)-2-(cyclopropylmethylene)-6,7-dimethyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3bg**, white solid, mp 151-152 °C; 90.8, 87% yield; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 11.5 Hz, 2H), 7.35 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 8.5 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 5.12

(d, J = 5.5 Hz, 1H), 3.76 (s, 2H), 2.40 (s, 3H), 2.34 (s, 3H), 2.27 (s, 3H), 2.25 (s, 3H), 1.52-1.47 (m, 1H), 0.95-0.91 (m, 2H), 0.50-0.47 (m, 2H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.4, 143.8, 136.8, 135.2, 134.4, 134.1, 133.9, 129.7, 129.2, 127.8, 127.4, 126.9, 126.1, 125.8, 124.0, 123.4, 42.5, 21.6, 19.6, 10.3, 7.7; HRMS (ESI) calcd for $C_{28}H_{31}N_2O_4S_2[M+H]^+$: 523.1704, found 523.1704.



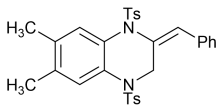
3dg

(*E*)-6,7-dichloro-2-(cyclopropylmethylene)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3dg**, white solid, mp 163-164 °C; 94.3 mg, 84% yield; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (d, J = 4.0 Hz, 2H), 7.42-7.37 (m, 4H), 7.28 (d, J = 4.5 Hz, 2H), 7.17 (d, J = 8.0 Hz, 2H), 5.22 (d, J = 11.0 Hz, 1H), 3.78 (s, 2H), 2.45 (s, 3H), 2.39 (s, 3H), 1.58-1.53 (m,1H), 1.01-0.99 (m, 2H), 0.57-0.56 (m, 2H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.2, 144.6, 138.2, 134.6, 133.9, 130.0, 129.6, 128.5, 128.3, 127.8, 127.5, 126.0, 124.1, 121.9, 41.8, 21.7, 21.6, 10.5, 7.8; HRMS (ESI) calcd for $C_{26}H_{25}Cl_2N_2O_4S_2[M+H]^+$: 563.0627, found 563.0615.



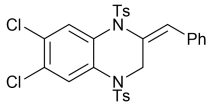
3ah

(*E*)-2-benzylidene-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3ah**, white solid, mp 158-159 °C; 90.3 mg, 85% yield. R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (q, J = 7.5 Hz, J = 2.0 Hz, 1H), 7.73 (q, J = 7.5 Hz, J = 2.5 Hz, 1H), 7.44-7.41 (m, 4H), 7.36 (t, J = 15.0 Hz, J = 7.5 Hz, 1H), 7.25-7.24 (m, 2H), 7.22-7.19 (m, 2H), 7.12 (d, J = 7.0 Hz, 2H), 7.06-7.02 (m, 4H), 6.87 (s, 1H), 4.09 (s, 2H), 2.44 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.9, 143.9, 135.2, 134.6, 134.1, 130.9, 129.9, 129.5, 129.3, 129.2, 128.8, 128.4, 127.8, 127.1, 127.0, 125.9, 125.7, 125.1, 122.9, 43.1, 21.7, 21.6; HRMS (ESI) calcd for $C_{29}H_{27}N_2O_4S_2[M+H]^+$: 531.1407, found 531.1401.



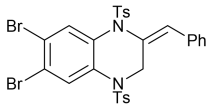
3bh

(*E*)-2-benzylidene-6,7-dimethyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3bh**, white solid, mp 162-163 °C; 93.6 mg, 84% yield; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (s, 1H), 7.49 (s, 1H), 7.43-7.40 (m, 4H), 7.37-7.35 (m, 1H), 7.26-7.24 (m, 2H), 7.08 (d, *J* = 7.0 Hz, 2H), 7.04 (s, 4H), 6.82 (s, 1H), 4.02 (s, 2H), 2.44 (s, 3H), 2.35 (s, 3H), 2.30 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.8, 143.7, 135.2, 134.7, 134.6, 134.2, 133.8, 130.6, 129.9, 129.4, 129.2, 128.7, 128.3, 127.8, 127.4, 127.1, 126.9, 126.6, 126.3, 43.0, 21.7, 21.6, 19.7, 19.6; HRMS (ESI) calcd for $C_{31}H_{31}N_2O_4S_2[M+H]^+$: 559.1725, found 559.1724.



3dh

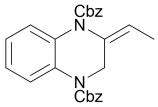
(*E*)-2-benzylidene-6,7-dichloro-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3dh**, white solid, mp 180-181 °C; 96.9 mg, 81% yield; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.97 (s, 1H), 7.47-7.39 (m, 5H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 7.0 Hz, 2H), 7.04 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 2H), 6.87 (s, 1H), 3.94 (s, 2H), 2.47 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.5, 144.5, 134.3, 133.9, 133.6, 132.4, 130.1, 129.7, 129.3, 129.2, 128.9, 128.8, 128.6, 128.0, 127.9, 127.7, 127.1, 126.2, 125.0, 124.0, 41.6, 21.8, 21.7; HRMS (ESI) calcd for $C_{29}H_{25}Cl_2N_2O_4S_2[M+H]^+$: 599.0633, found 599.0629.



3eh

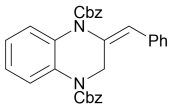
(*E*)-2-benzylidene-6,7-dibromo-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline **3eh**, white solid, mp 174-175 °C; 109.5 mg, 80% yield; R_f = 0.5 (petroleum ether / EtOAc = 4:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (s, 1H), 8.11 (s, 1H), 7.46-7.40 (m, 5H), 7.30 (d, *J* = 8.5 Hz, 2H), 7.08 (d, *J* = 7.5 Hz, 2H), 7.04 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 8.5 Hz, 2H), 6.87 (s, 1H), 3.94 (s, 2H), 2.47 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$)

δ 145.5, 144.5, 134.3, 133.9, 133.6, 132.3, 130.1, 129.7, 129.2, 128.9, 128.8, 128.5, 128.3, 127.9, 127.1, 126.9, 125.0, 121.0, 120.2, 41.6, 21.8, 21.6; HRMS (ESI) calcd for $C_{29}H_{25}Br_2N_2O_4S_2[M+H]^+$: 686.9622, found 686.9622.



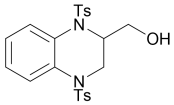
3ja

dibenzyl (*E*)-2-ethylidene-2,3-dihydroquinoxaline-1,4-dicarboxylate, oil, 48.9 mg, 57% yield; R_f = 0.6 (petroleum ether / EtOAc = 5:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40-7.31 (m, 12H), 7.14-7.07 (m, 2H), 5.76-5.71 (m, 1H), 5.25 (s, 2H), 5.20 (s, 2H), 4.61 (s, 2H), 1.69 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.0, 153.5, 136.1, 135.9, 132.9, 128.6, 128.5, 128.3, 128.2, 128.1, 128.0, 127.8, 125.4, 124.7, 124.5, 68.0, 67.8, 48.1, 12.9; HRMS (ESI) calcd for $C_{26}H_{24}N_2O_4Na[M+Na]^+$: 451.1633, found 451.1634.



3jh

dibenzyl (*E*)-2-benzylidene-2,3-dihydroquinoxaline-1,4-dicarboxylate, oil, 49.7 mg, 51% yield; R_f = 0.6 (petroleum ether / EtOAc = 5:1); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.48-7.46 (m, 1H), 7.36-7.31 (m, 12H), 7.27-7.25 (m, 2H), 7.20 (d, *J* = 7.8 Hz, 2H), 7.14-7.12 (m, 2H), 6.78 (s, 1H), 5.26 (s, 2H), 5.24 (s, 2H), 4.87 (s, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.4, 135.9, 134.9, 132.7, 128.7, 128.6, 128.5, 128.3, 128.2, 128.1, 127.9, 125.6, 124.7, 68.0; HRMS (ESI) calcd for $C_{31}H_{27}N_2O_4[M+H]^+$: 491.1971, found 491.1968.



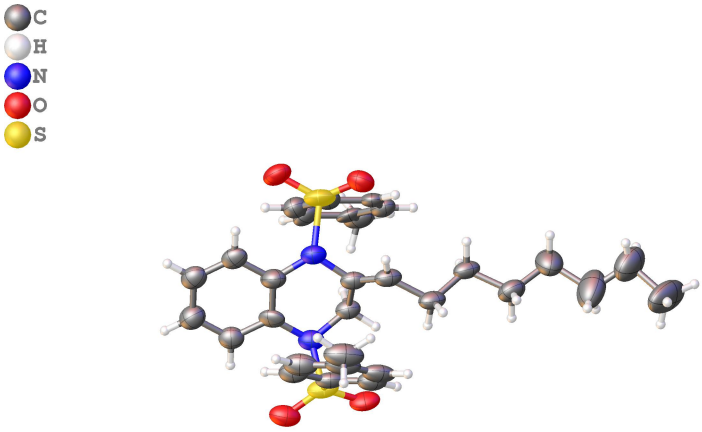
4

1,4-ditosyl-1,2,3,4-tetrahydroquinoxalin-2-yl)methanol **4**, oil; 57.7 mg, 61% yield; R_f = 0.4 (petroleum ether / EtOAc = 3:1); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.8 Hz, 2H), 7.56 (d, *J* = 7.8 Hz, 1H), 7.42-7.38 (m, 4H), 7.17 (d, *J* = 7.8 Hz, 2H), 7.00-6.98 (m, 1H), 6.71-6.69 (m, 1H), 6.53 (d, *J* = 7.8 Hz, 1H), 4.14-4.11 (m, 1H),

3.53 (m,1H), 3.25-3.22 (m, 1H), 3.14-3.11 (m,1H), 3.05-3.01 (m, 1H), 2.50 (s, 3H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 144.0, 136.4, 136.2, 135.9, 130.2, 129.8, 127.9, 127.1, 117.9, 115.2, 58.9, 48.3, 43.2, 21.8, 21.6; HRMS (ESI) calcd for $C_{23}H_{24}N_2O_5S_2[M+Na]^+$: 495.1024, found 495.0816.

7. X-Ray crystallographic data of 3af.

The relative configuration 3af was determined by X-ray single crystal diffraction analysis. CCDC 2208884 (3af) contain the supplementary crystallographic data for this paper. X-Ray Crystallography Data for 3af (CCDC 2208884): A colorless crystal suitable for X-ray crystallography was obtained from a petroleum ether/dichloromethane solution at room temperature under air.



3af

Bond precision: = 0.0000 A Wavelength=1.54184

Cell: a=9.8087(3) b=10.6541(4) c=14.2276(4)

alpha=75.029(3) beta=87.646(2) gamma=77.962(3)

Temperature: 100 K

	Calculated	Reported
Volume	1404.64(8)	1404.64(8)
Space group	P -1	P -1
Hall group	-P 1	-P 1

Moiety formula	$C_{30}H_{36}N_2O_4S_2$	1($C_{30}H_{36}N_2O_4S_2$)
Sum formula	$C_{30}H_{36}N_2O_4S_2$	$C_{30}H_{36}N_2O_4S_2$
Mr	552.73	552.73
Dx,g cm-3	1.307	1.307
Z	2	2
Mu (mm-1)	2.025	2.025
F000	588.0	588.0
F000’	590.87	
h,k,lmax	12,13,17	12,13,17
Nref	5737	5635
Tmin,Tmax	0.747,0.904	0.699,1.000
Tmin’	0.567	

Correction method= # Reported T Limits: Tmin=0.699 Tmax=1.000

AbsCorr = MULTI-SCAN

Data completeness= 0.982 Theta(max)= 74.503

R(reflections) = 0.0734(4916) wR2(reflections)= 0.1943(5635)

S = 1.105 Npar= 620

8.References

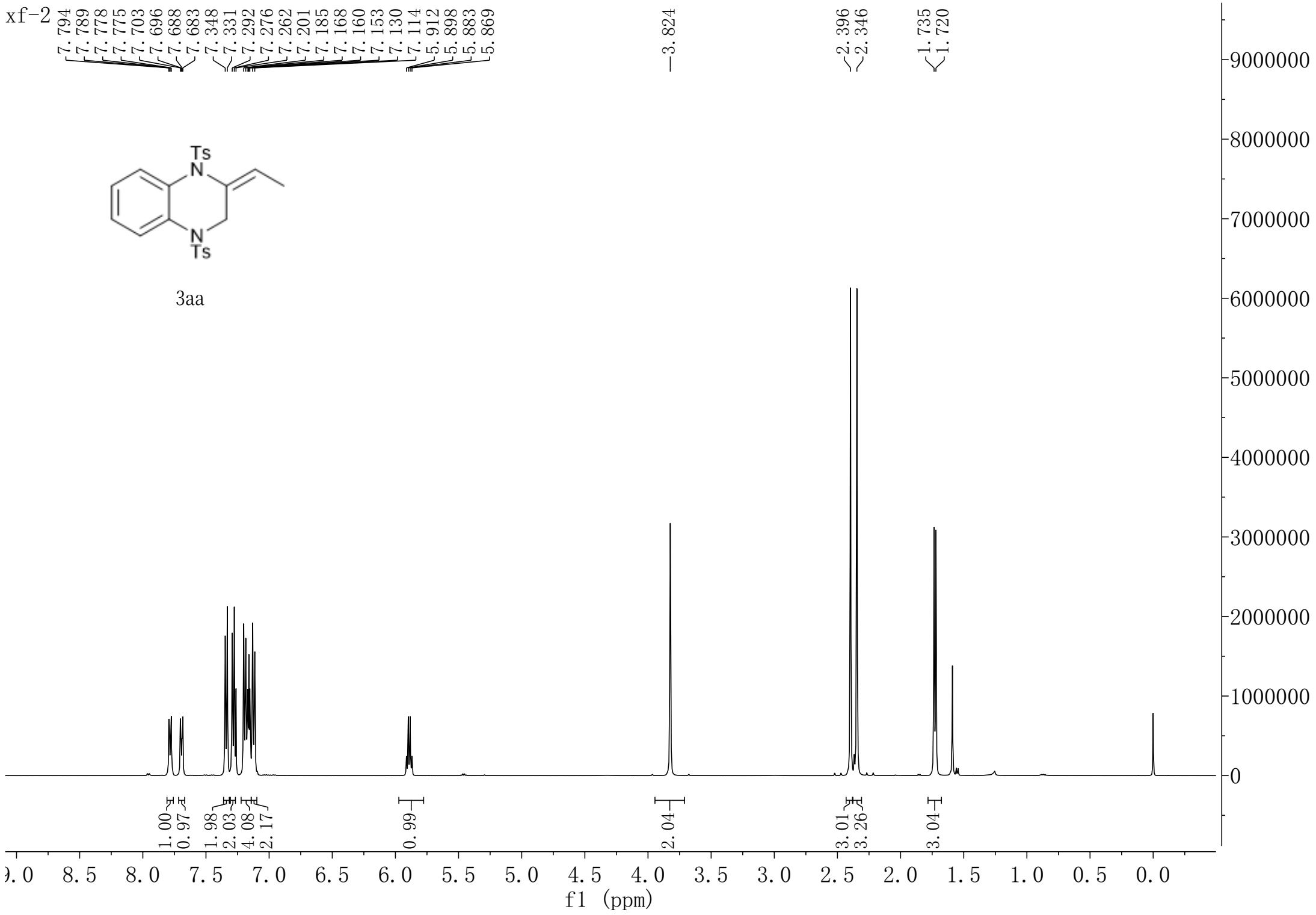
[1] D. Wang, H. Yu, S. Sun, and F. Zhong, *Org. Lett.*, **2020**, *22*, 2425-2430.

[2] Z. Wu, K. Wen, J. Zhang and W. Zhang, *Org. Lett.,* **2017**, *19*, 2813-2816.

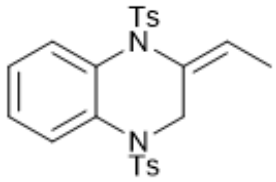
[3] B. Deng, C. B. Rao, R. Zhang, J. Li, Y. Liang, Y. Zhao, M. Gao and D. W. Dong, *Adv. Synth. Catal.,* **2019**, *19*, 4549-4557.

[4] Y.-M. Zhou, Y. Chen, Y. Huang, *Org. Lett.*, **2020**, *22*, 5941-5946.

[5] Y.-M. Zhou, N, Li, W. Cai, Y. Huang, *Org. Lett.*, **2021**, *23*, 8755-8760.


xf-2
7.794
7.789
7.778
7.775
7.703
7.696
7.688
7.683
7.348
7.331
7.292
7.276
7.262
7.201
7.185
7.168
7.160
7.153
7.130
7.114
5.912
5.898
5.883
5.869
3.824
2.396
2.346
1.735
1.720
Ts
N
N
Ts
3aa
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
1.00
0.97
1.98
2.03
4.08
2.17
0.99
2.04
3.01
3.26
3.04
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)

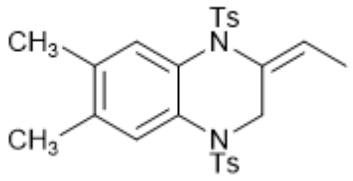
xf-2


Ts
N
N
Ts

3aa

144.6
144.1
135.5
134.6
129.8
129.5
129.4
127.6
127.1
126.7
125.8
125.6
125.1
122.8
42.6
21.6
21.5
13.4
18000000
17000000
16000000
15000000
14000000
13000000
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
-1000000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

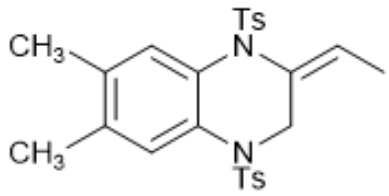
xf-5



3ba

f1 (ppm)

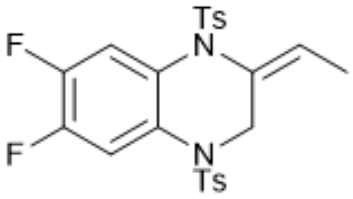
xf-5


Ts
N
CH3
CH3
N
Ts

3ba

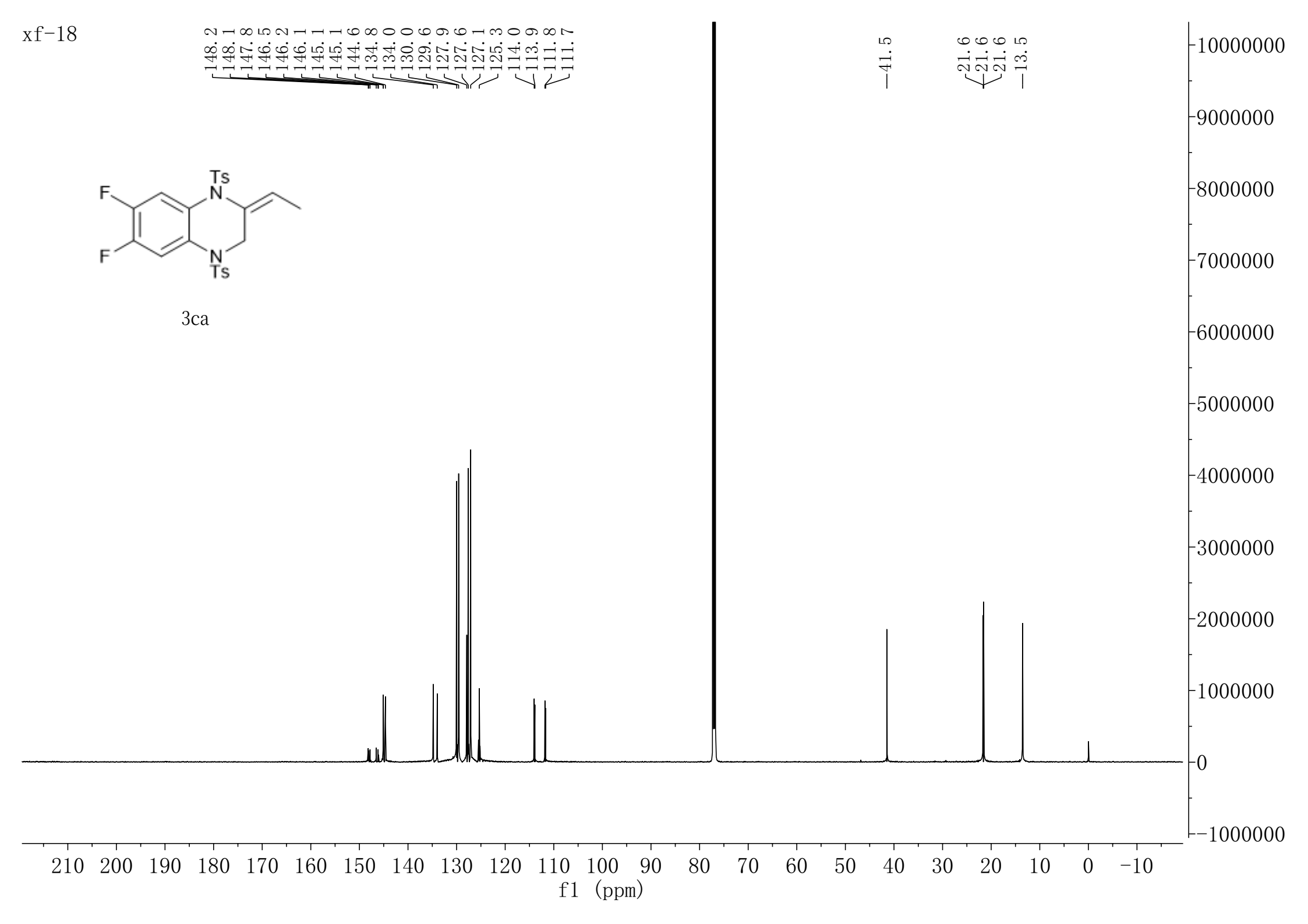
144.4
143.9
135.6
134.8
134.5
133.8
129.7
129.3
127.6
127.1
127.1
127.0
127.0
126.3
126.2
123.5
42.7
21.6
21.5
19.6
19.5
13.4
15000000
14000000
13000000
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
-1000000
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

xf-18

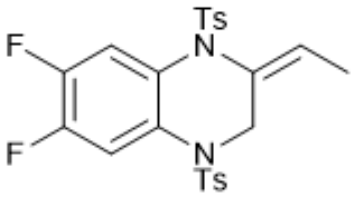

Ts
N
F
F
N
Ts

3ca

7.740
7.665
7.351
7.342
7.293
7.279
7.262
7.235
7.224
7.163
7.150
5.904
5.888
3.701
2.418
2.371
1.738
1.730
1.00
0.99
2.07
2.64
2.62
2.05
0.98
2.08
3.09
3.46
3.09
15000000
14000000
13000000
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
-1000000
0.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

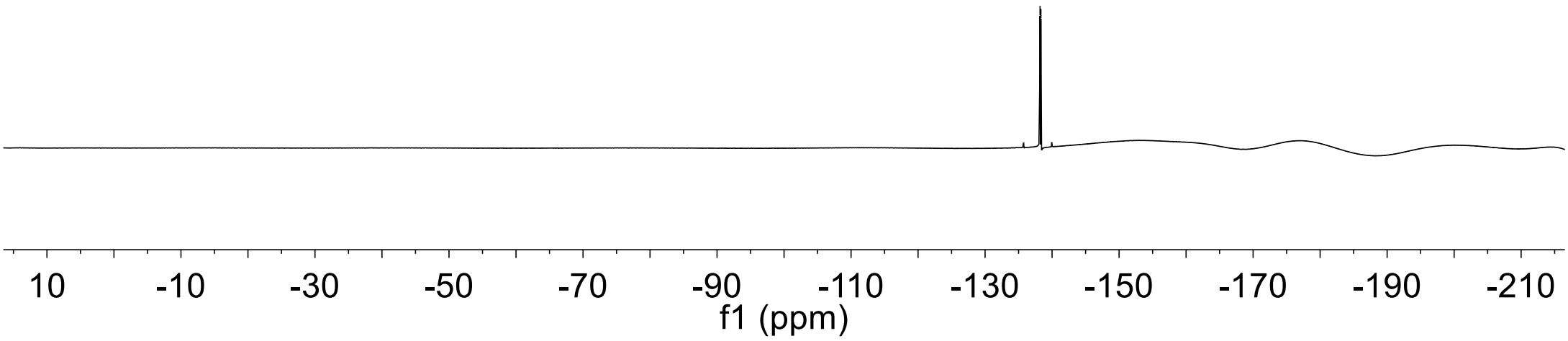

xf-18
148.2
148.1
147.8
146.5
146.2
146.1
145.1
145.1
144.6
134.8
134.0
130.0
129.6
127.9
127.6
127.1
125.3
114.0
113.9
111.8
111.7
41.5
21.6
21.6
21.6
13.5
Ts
N
F
F
N
Ts
3ca
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
-1000000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

xf-18

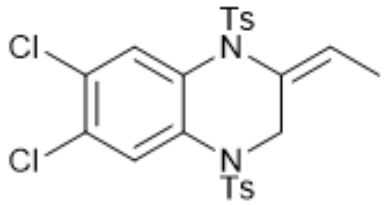


3ca

-138.20
-138.33



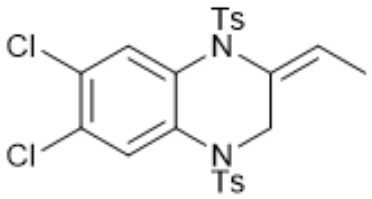
xf-19


Ts
N
Cl
Cl
N
Ts

3da

7.952
7.902
7.370
7.362
7.312
7.304
7.263
7.240
7.231
7.173
7.165
5.915
5.908
3.719
2.424
2.377
1.739
1.725
1.568
1.00
1.01
2.18
2.20
2.99
2.29
1.07
2.26
3.36
3.33
3.40
22000000
20000000
18000000
16000000
14000000
12000000
10000000
8000000
6000000
4000000
2000000
0
-2000000
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
-0.5
f1 (ppm)

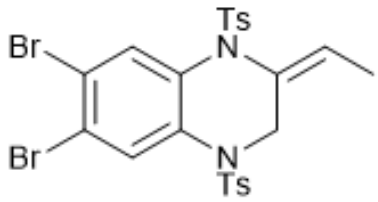
xf-19


Ts
N
Cl
Cl
N
Ts

3da

145.1
144.7
134.8
134.0
130.0
130.0
129.6
129.2
128.4
128.3
128.3
128.1
127.7
127.1
126.2
125.3
123.7
41.6
21.7
21.6
13.5
3.5E+07
3.0E+07
2.5E+07
2.0E+07
1.5E+07
1.0E+07
5.0E+06
0.0E+00
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

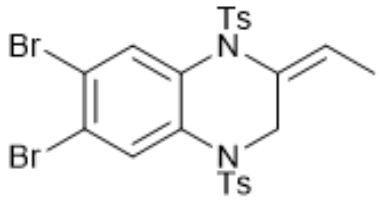
xf-17



3ea

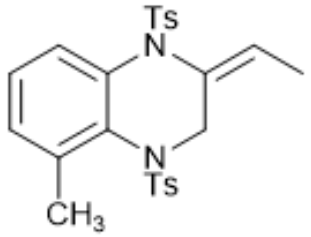
f1 (ppm)

xf-17



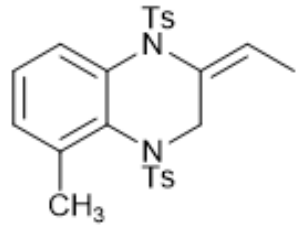
3ea

xf-7



3fa r.r >20:1 on the crude reaction mixture

xf-7


Ts
N
N
Ts
CH3

3fa

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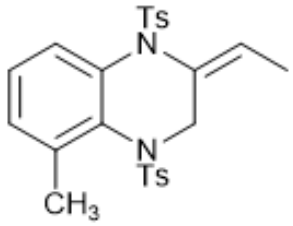
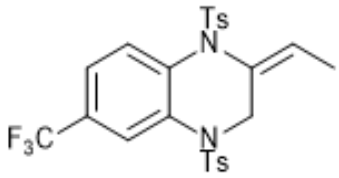
7.689
7.648
7.631
7.318
7.302
7.295
7.279
7.219
7.205
7.072
7.056
7.041
7.022
7.008
5.957
5.938
5.923
5.904
4.672
4.643
3.944
3.915
2.467
2.445
2.388
1.645
1.630
2.04
1.99
4.06
1.07
2.06
1.00
1.05
1.04
3.02
3.06
3.26
3.49
f1 (ppm)

xf-7



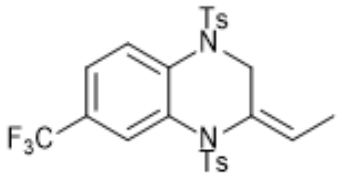
3fa

xf-39



3ga

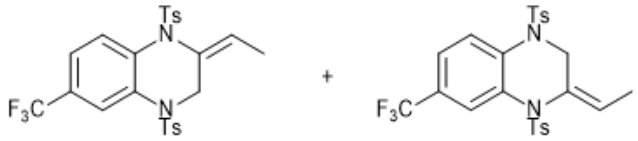
+



3ga*

r.r = 4:1 on the crude reaction mixture

xf-39


Ts
N
F3C
N
Ts

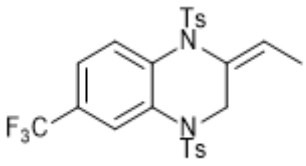
3ga

Ts
N
F3C
N
Ts

3ga*

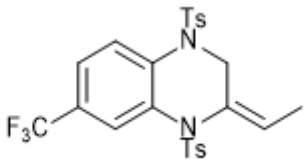
8.071
7.848
7.830
7.379
7.363
7.340
7.324
7.232
7.216
7.175
7.159
5.976
5.972
5.961
5.947
5.932
3.868
2.417
2.371
1.751
1.737
0.77
0.20
0.21
0.81
4.79
0.46
2.04
1.62
0.42
1.00
1.69
0.40
3.04
2.42
0.67
3.09
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
9.5 9.0 8.5 8.0 7.5 7.0 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 -0.5
f1 (ppm)

xf-39



3ga

+



3ga*

145.0
144.7
135.1
134.3
132.2
130.0
130.0
129.7
129.6
129.0
127.8
127.7
127.6
127.2
127.1
125.8
122.8
122.8
122.4
122.3
122.3

42.4
41.7

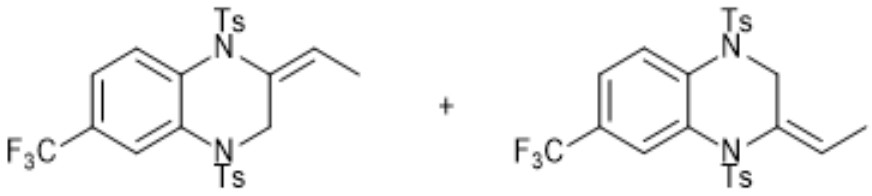
21.7
21.6

13.5

1300000
1200000
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
−100000

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

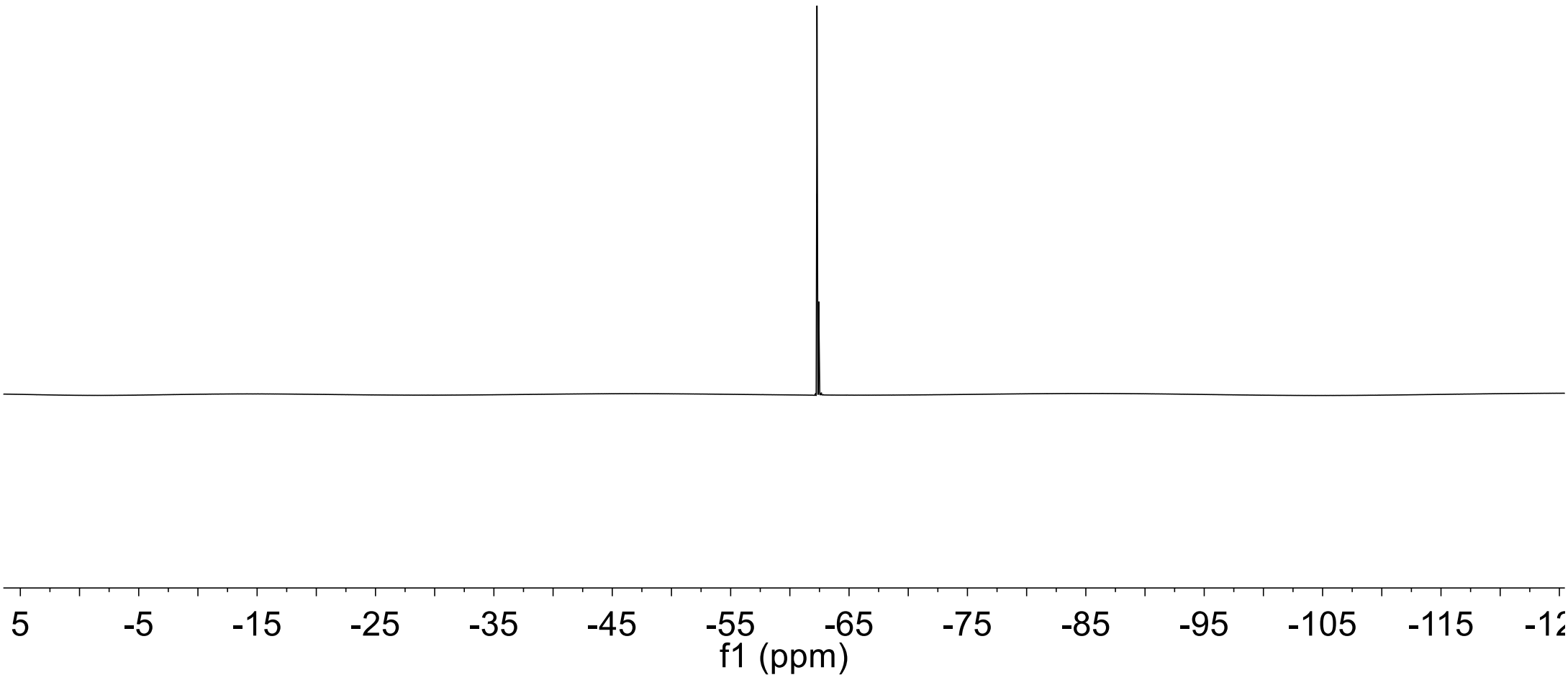


3ga

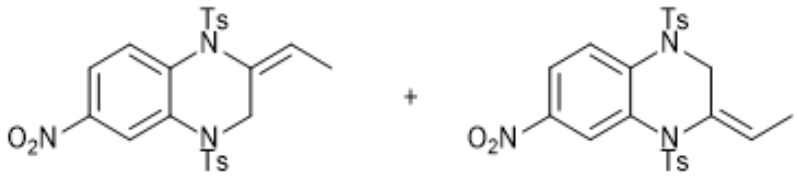
+

3ga*

-62.29
-62.44



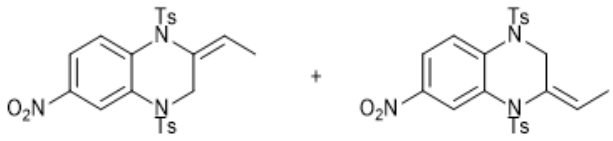
xf-40



3ha 3ha*

3ha:3ha*(r.r)= 10:1 on the crude reaction mixture

XF-40

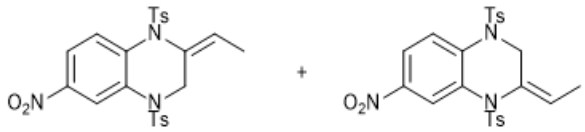


3ha

3ha*

f1 (ppm)

xf-40



3ha

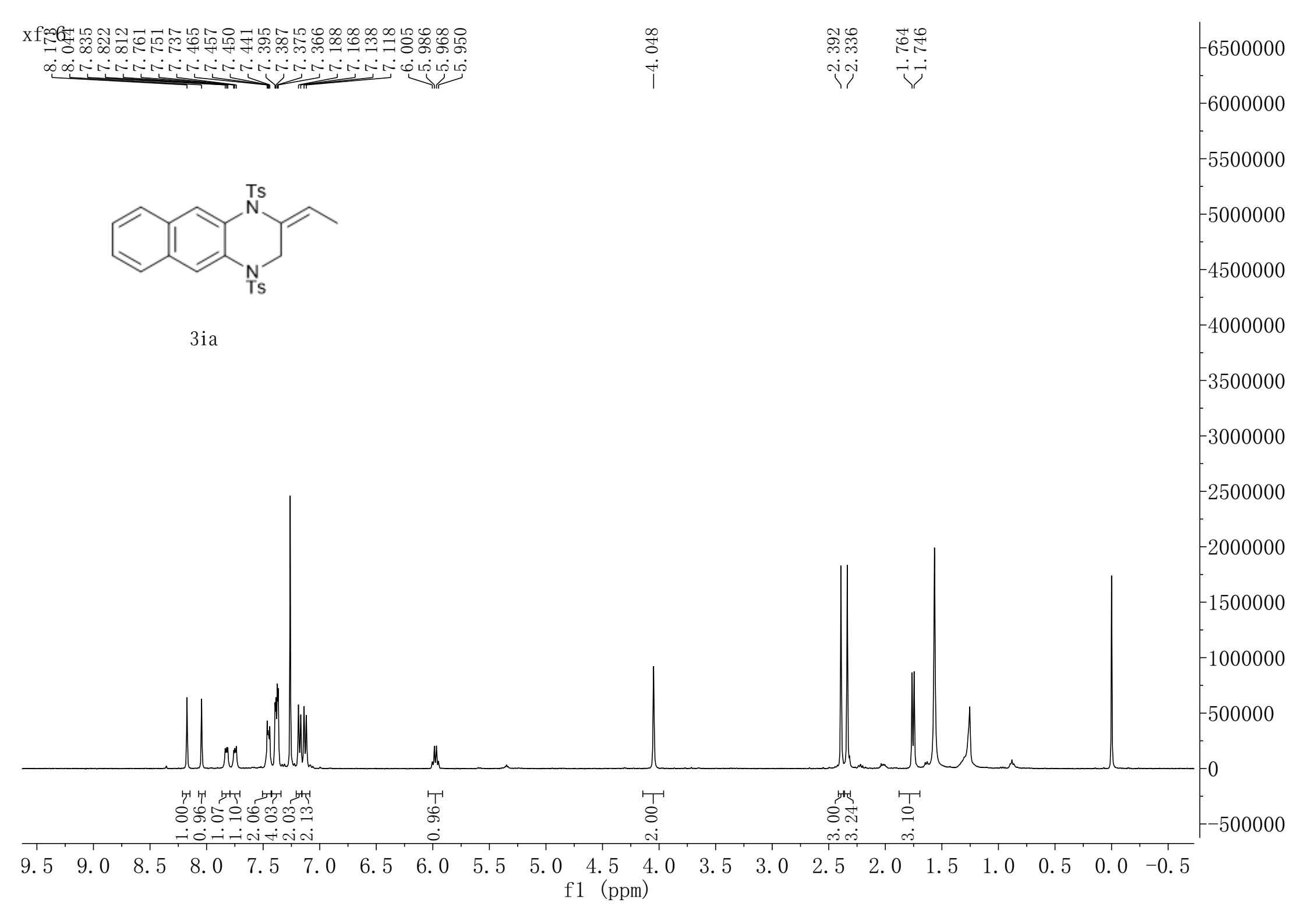
3ha*

145.3
145.1
143.6
134.8
134.7
134.1
130.1
129.9
128.7
128.6
127.8
127.1
125.3
121.7
121.1
120.4
42.4
21.7
21.6
13.6

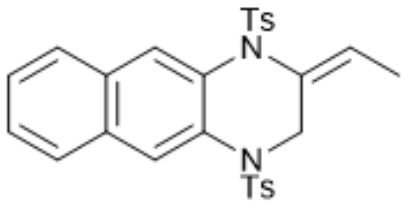
1400000
1300000
1200000
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
-100000

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

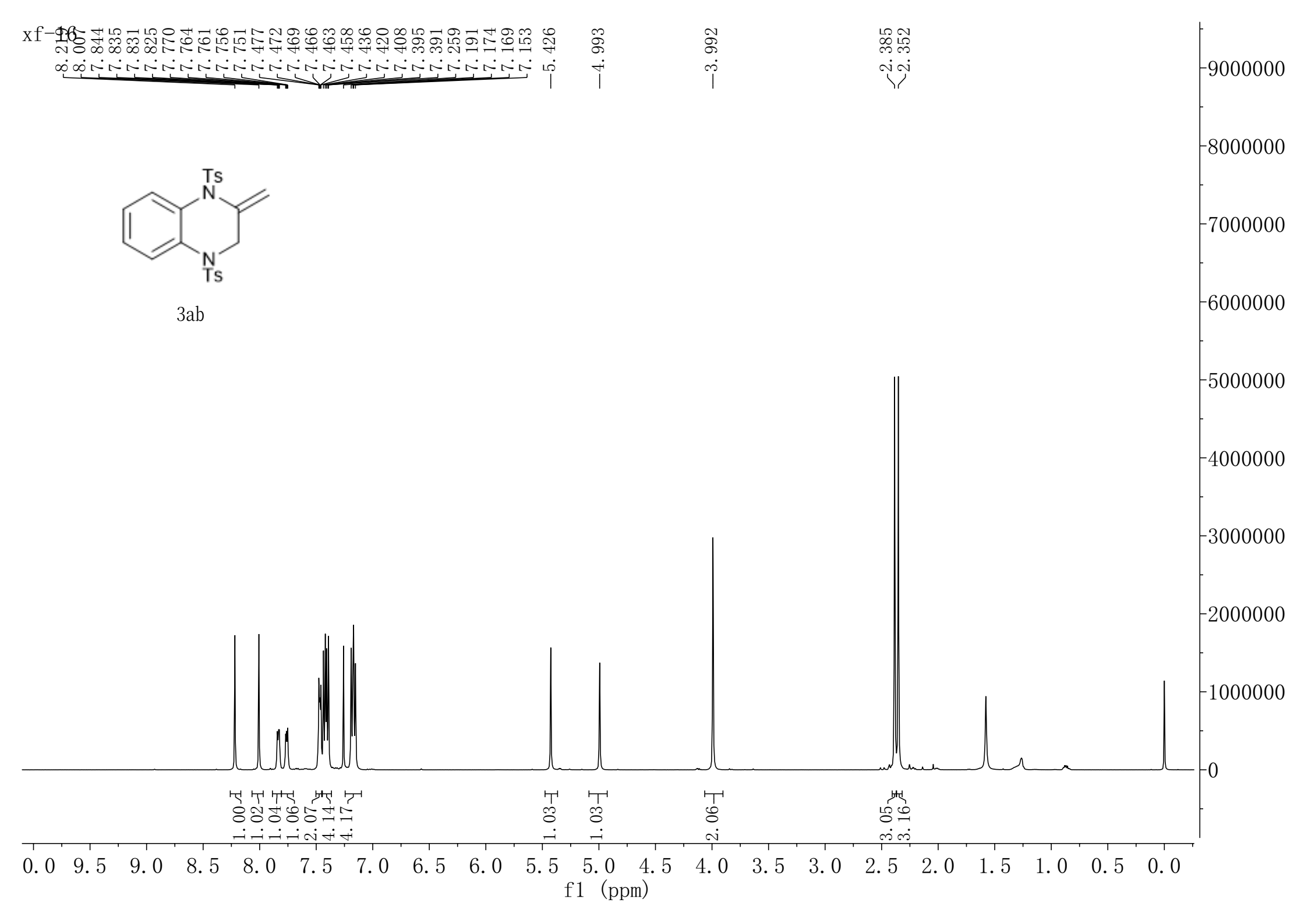

xf-6
8.173
8.044
7.835
7.822
7.812
7.761
7.751
7.737
7.465
7.457
7.450
7.441
7.395
7.387
7.375
7.366
7.188
7.168
7.138
7.118
6.005
5.986
5.968
5.950
4.048
2.392
2.336
1.764
1.746
Ts
N
N
Ts
3ia
1.00
0.96
1.07
1.10
2.06
4.03
2.03
2.13
0.96
2.00
3.00
3.24
3.10
f1 (ppm)

xf-6

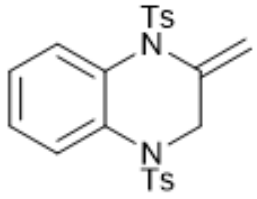

Ts
N
N
Ts

3ia

144.5
144.2
135.7
135.2
131.2
130.5
129.9
129.6
128.7
128.5
127.8
127.5
127.5
127.1
126.4
126.3
126.0
123.9
119.9
43.9
21.6
21.5
13.4
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
-100000
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


xf-16
8.215
8.007
7.844
7.835
7.831
7.825
7.770
7.764
7.761
7.756
7.751
7.477
7.472
7.469
7.466
7.463
7.458
7.436
7.420
7.408
7.395
7.391
7.259
7.191
7.174
7.169
7.153
5.426
4.993
3.992
2.385
2.352
Ts
N
N
Ts
3ab
1.00
1.02
1.04
1.06
2.07
4.14
4.17
1.03
1.03
2.06
3.05
3.16
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
0.0
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)

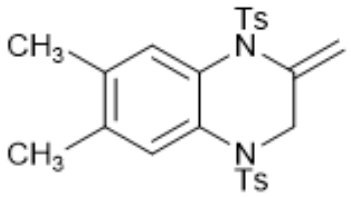
xf-1


Ts
N
N
Ts

3ab

144.8
144.2
135.4
134.4
134.3
129.8
129.7
129.6
129.5
127.6
127.3
125.7
125.4
125.2
123.4
112.7
48.0
21.6
21.5
20000000
19000000
18000000
17000000
16000000
15000000
14000000
13000000
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
−1000000
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)

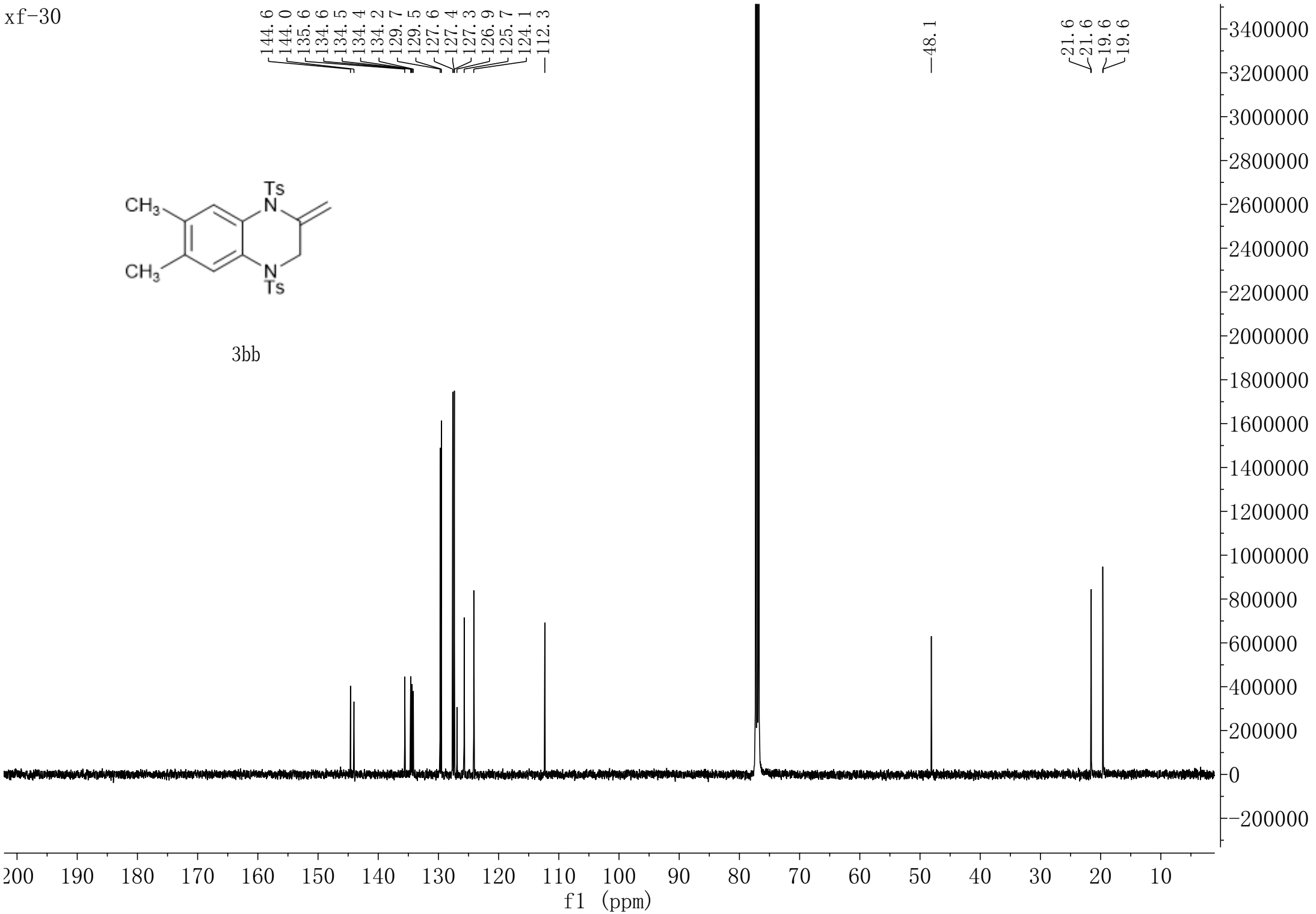
xf-30


Ts
N
CH3
CH3
N
Ts

3bb

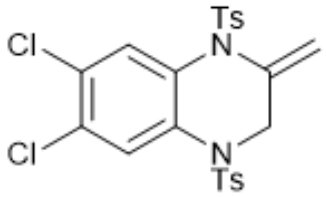
7.593
7.430
7.391
7.375
7.331
7.314
7.262
7.208
7.192
7.158
7.142
5.264
4.871
3.707
2.394
2.362
2.271
2.245
1.569
1.04
1.01
1.95
1.93
1.99
1.91
1.00
1.00
2.01
3.04
2.83
2.95
2.89
f1 (ppm)

xf-30


144.6
144.0
135.6
134.6
134.5
134.4
134.2
129.7
129.5
127.6
127.4
127.3
126.9
125.7
124.1
112.3
48.1
21.6
21.6
19.6
19.6
Ts
N
CH3
CH3
N
Ts
3400000
3200000
3000000
2800000
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
-200000
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

3bb

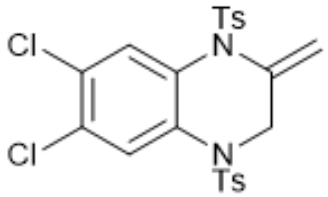
xf-31


Ts
N
Cl
Cl
N
Ts

3db

8.028
7.888
7.403
7.387
7.340
7.324
7.262
7.248
7.232
7.196
7.180
5.338
4.995
3.634
2.416
2.381
1.573
1.00
0.99
2.11
2.10
2.60
2.19
1.05
1.06
2.15
3.19
3.03
2.8E+07
2.6E+07
2.4E+07
2.2E+07
2.0E+07
1.8E+07
1.6E+07
1.4E+07
1.2E+07
1.0E+07
8.0E+06
6.0E+06
4.0E+06
2.0E+06
0.0E+00
-2.0E+06
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

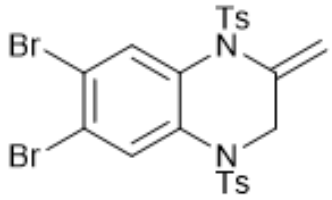
xf-31


Ts
N
Cl
Cl
N
Ts

3db

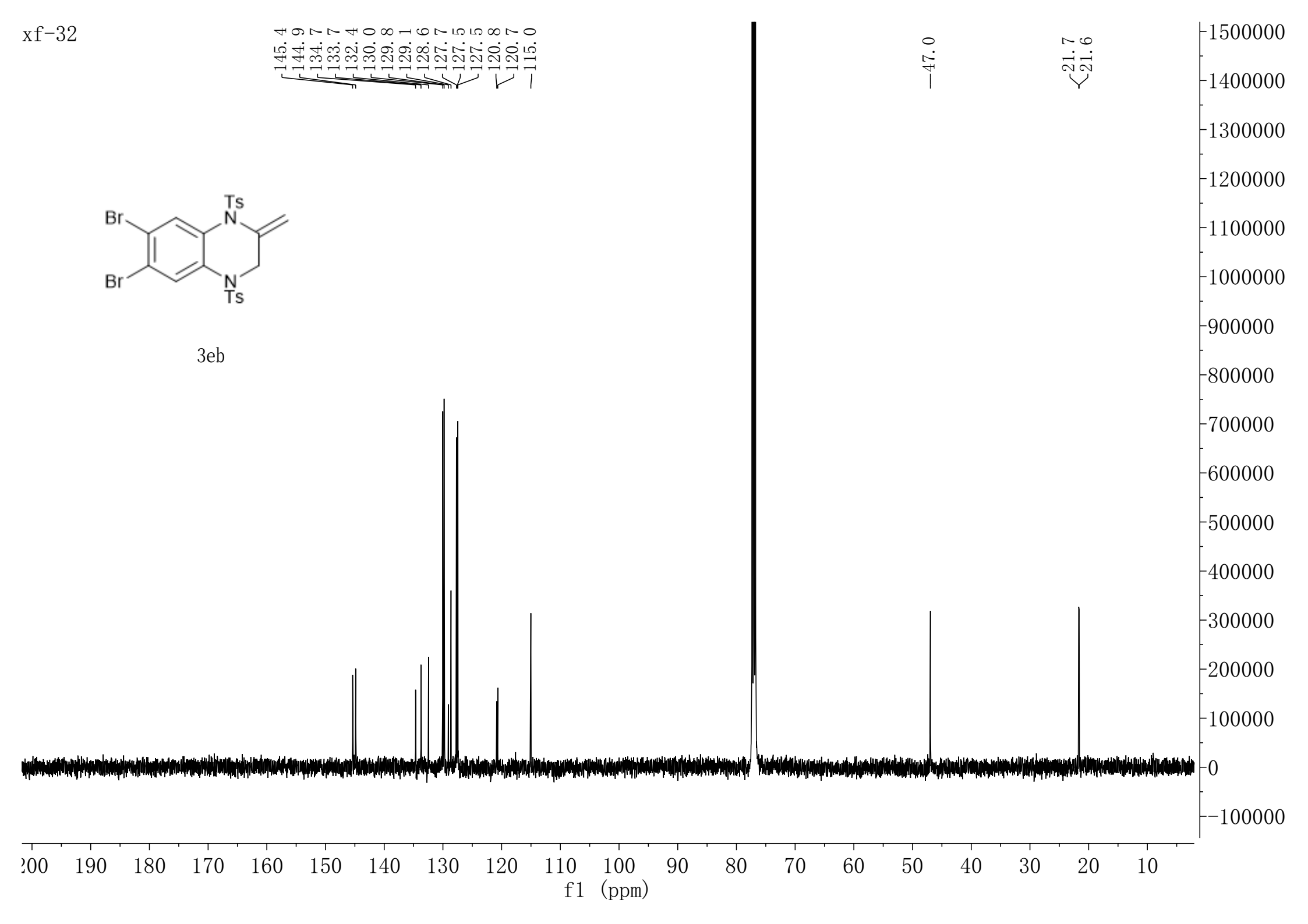
145.4
144.8
134.7
133.7
132.5
130.0
129.8
129.1
129.0
128.5
128.0
127.7
127.4
125.6
124.6
115.0
47.0
21.7
21.6
3000000
2800000
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
-200000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

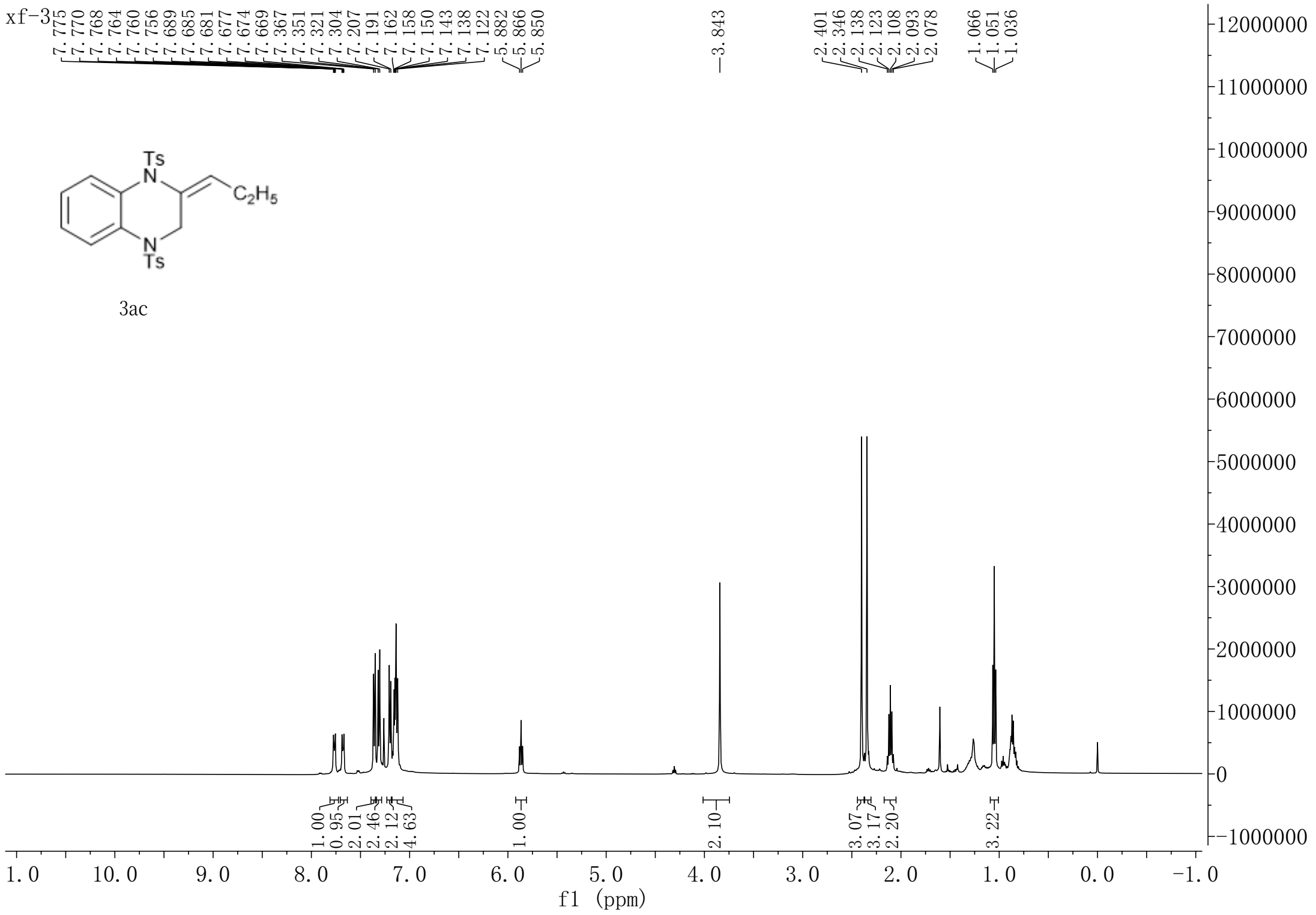
xf-32


Ts
N
Br
Br
N
Ts

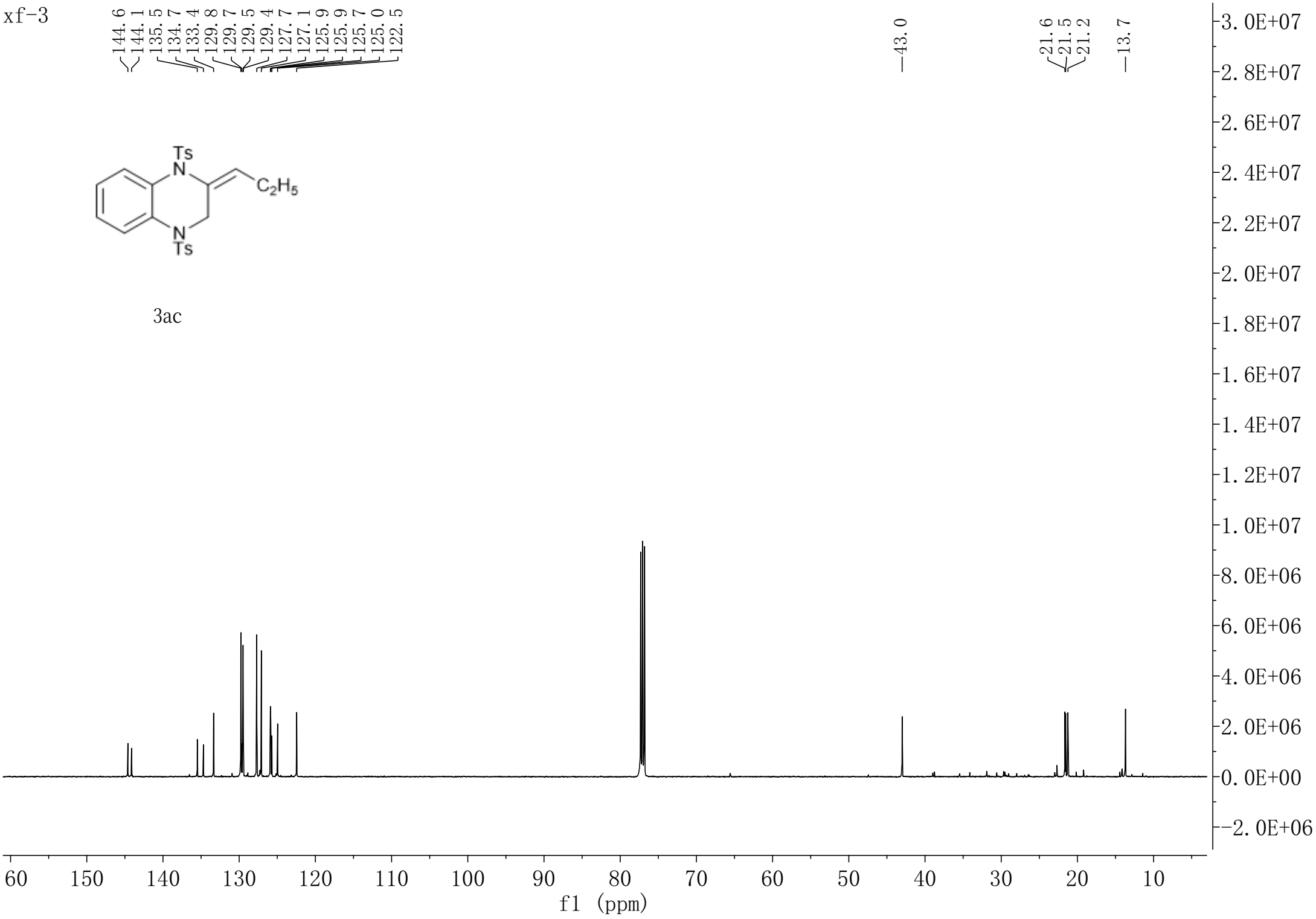
3eb

8.164
8.031
7.408
7.391
7.344
7.328
7.262
7.252
7.236
7.201
7.185
5.337
4.992
3.630
2.419
2.384
1.562
1.00
0.94
2.01
1.86
2.45
1.81
1.09
1.08
2.13
3.11
2.81
f1 (ppm)

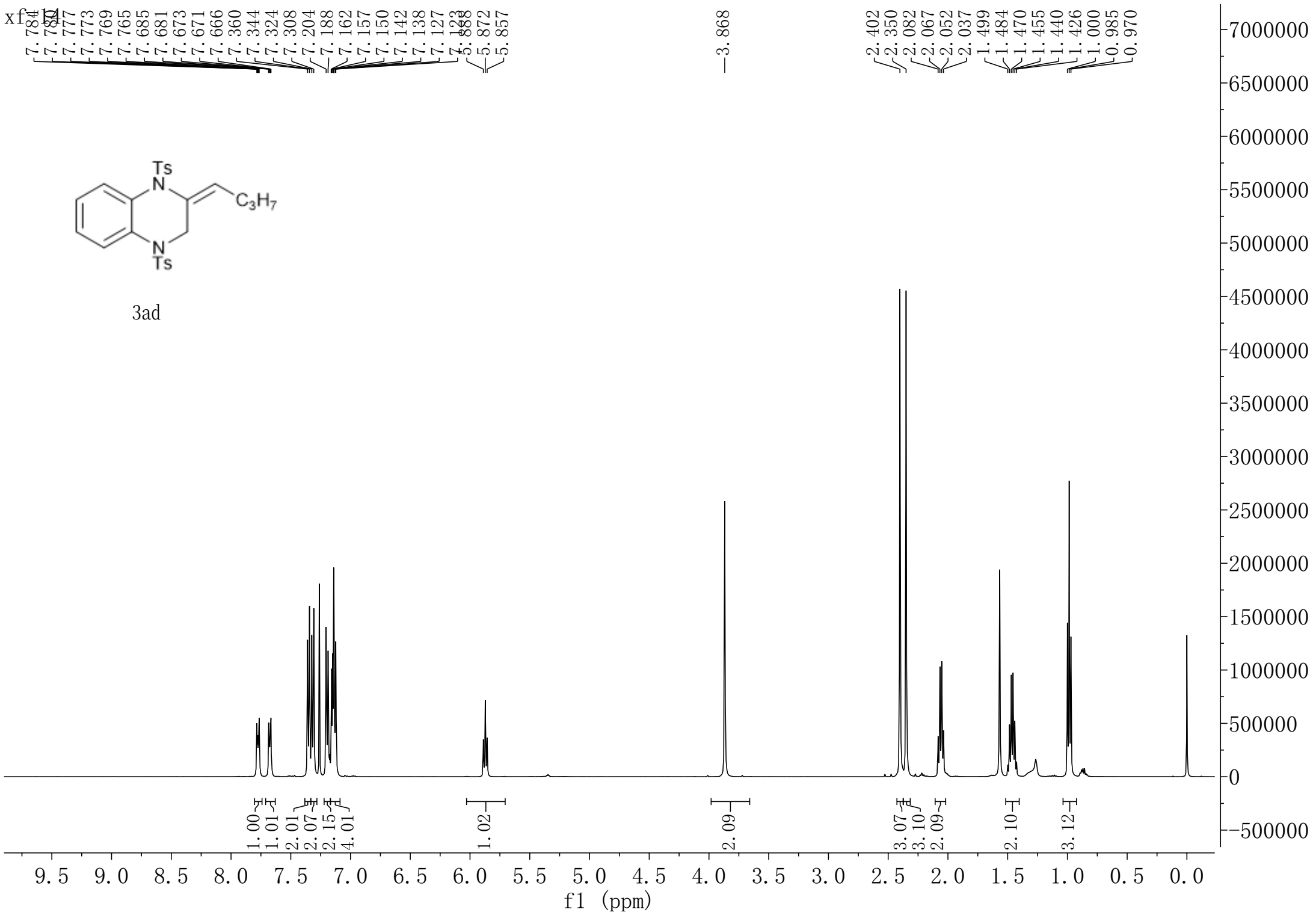

xf-32
145.4
144.9
134.7
133.7
132.4
130.0
129.8
129.1
128.6
127.7
127.5
127.5
120.8
120.7
115.0
47.0
21.7
21.6
Br
Br
N
Ts
N
Ts
3eb
1500000
1400000
1300000
1200000
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
-100000
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


xf-3
7.775
7.770
7.768
7.764
7.760
7.756
7.689
7.685
7.681
7.677
7.674
7.669
7.367
7.351
7.321
7.304
7.207
7.191
7.162
7.158
7.150
7.143
7.138
7.122
5.882
5.866
5.850
3.843
2.401
2.346
2.138
2.123
2.108
2.093
2.078
1.066
1.051
1.036
Ts
N
C2H5
N
Ts
3ac
1.00
0.95
2.01
2.46
2.12
4.63
1.00
2.10
3.07
3.17
2.20
3.22
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
-1000000
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

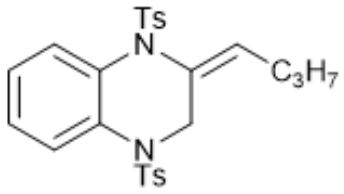
xf-3


144.6
144.1
135.5
134.7
133.4
129.8
129.7
129.5
129.4
127.7
127.1
125.9
125.9
125.7
125.0
122.5
43.0
21.6
21.5
21.2
13.7
Ts
N
C2H5
N
Ts
3.0E+07
2.8E+07
2.6E+07
2.4E+07
2.2E+07
2.0E+07
1.8E+07
1.6E+07
1.4E+07
1.2E+07
1.0E+07
8.0E+06
6.0E+06
4.0E+06
2.0E+06
0.0E+00
-2.0E+06
60
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

3ac

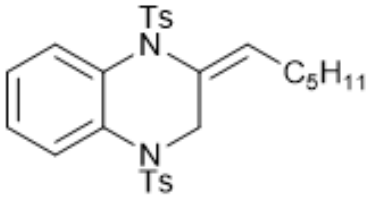

xf2-1H
7.784
7.782
7.777
7.773
7.769
7.765
7.685
7.681
7.673
7.671
7.666
7.360
7.344
7.324
7.308
7.204
7.188
7.162
7.157
7.150
7.142
7.138
7.127
7.123
5.872
5.857
3.868
2.402
2.350
2.082
2.067
2.052
2.037
1.499
1.484
1.470
1.455
1.440
1.426
1.000
0.985
0.970
Ts
N
C3H7
N
Ts
3ad
1.00
1.01
2.01
2.07
2.15
4.01
1.02
2.09
3.07
3.10
2.09
2.10
3.12
f1 (ppm)

1h. 22. 1. 1r
XF-14



3ad

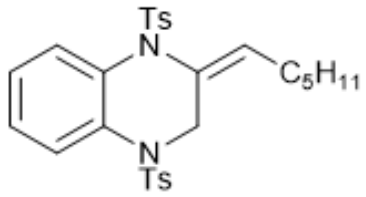
xf-1-3



3ae

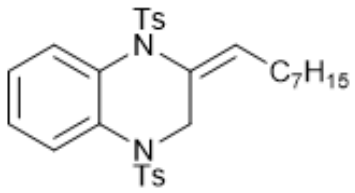
f1 (ppm)

1h.24.1.1r
XF-15



3ae

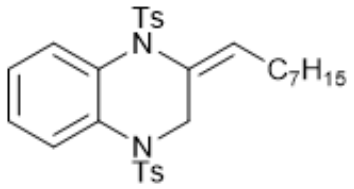
xf-10


Ts
N
C7H15
N
Ts

3af

7.783
7.778
7.771
7.763
7.689
7.681
7.674
7.669
7.358
7.342
7.322
7.305
7.201
7.185
7.157
7.149
7.142
7.137
7.120
5.881
5.865
5.850
3.857
2.403
2.350
2.089
2.074
2.060
2.044
1.430
1.426
1.415
1.401
1.340
1.328
1.324
1.316
1.309
1.305
0.927
0.914
0.900
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
1.00
0.97
1.91
2.13
2.10
3.83
0.98
2.00
3.01
2.97
1.99
2.09
8.43
3.05
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

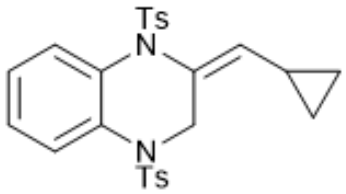
xf-10


Ts
N
C7H15
N
Ts

3af

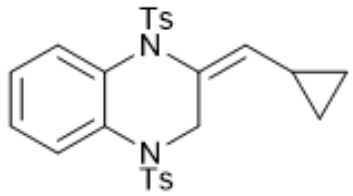
144.6
144.1
135.5
134.8
132.2
129.8
129.7
129.5
127.6
127.1
126.0
125.9
124.9
122.4
43.1
31.8
29.2
29.2
29.1
27.9
22.7
21.6
21.5
14.1
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
−1000000
)0 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

xf-4



3ag

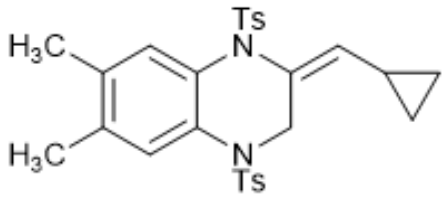
xf-4


Ts
N
N
Ts

3ag

144.6
144.0
137.0
135.1
134.4
129.7
129.4
129.3
128.7
127.7
127.4
125.5
125.3
125.2
123.4
123.1
42.5
21.6
21.5
10.3
7.7
3.2E+07
3.0E+07
2.8E+07
2.6E+07
2.4E+07
2.2E+07
2.0E+07
1.8E+07
1.6E+07
1.4E+07
1.2E+07
1.0E+07
8.0E+06
6.0E+06
4.0E+06
2.0E+06
0.0E+00
-2.0E+06
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

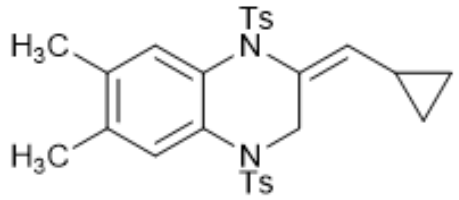
xf-25


Ts
N
H3C
H3C
N
Ts

3bg

7.567
7.546
7.356
7.339
7.299
7.282
7.206
7.190
7.097
7.081
5.131
5.109
3.763
2.401
2.339
2.265
2.254
1.529
1.504
1.489
1.474
1.457
0.945
0.936
0.932
0.920
0.907
0.500
0.491
0.479
0.470
2.00
2.05
2.03
2.03
2.03
0.99
2.05
3.25
3.09
2.95
3.34
1.07
2.22
2.08
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)
15000000
14000000
13000000
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
-1000000

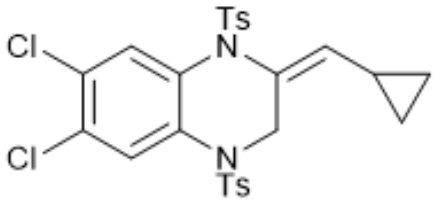
XF-25


Ts
N
H3C
H3C
N
Ts

3bg

144.4
143.8
136.8
135.2
134.4
134.1
133.9
129.7
129.2
127.8
127.4
126.9
126.1
125.8
124.0
123.4
42.5
21.6
21.6
19.6
10.3
7.7
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

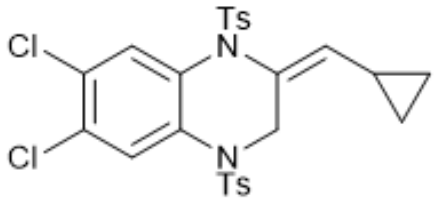
xf-23


Ts
N
Cl
Cl
N
Ts

3dg

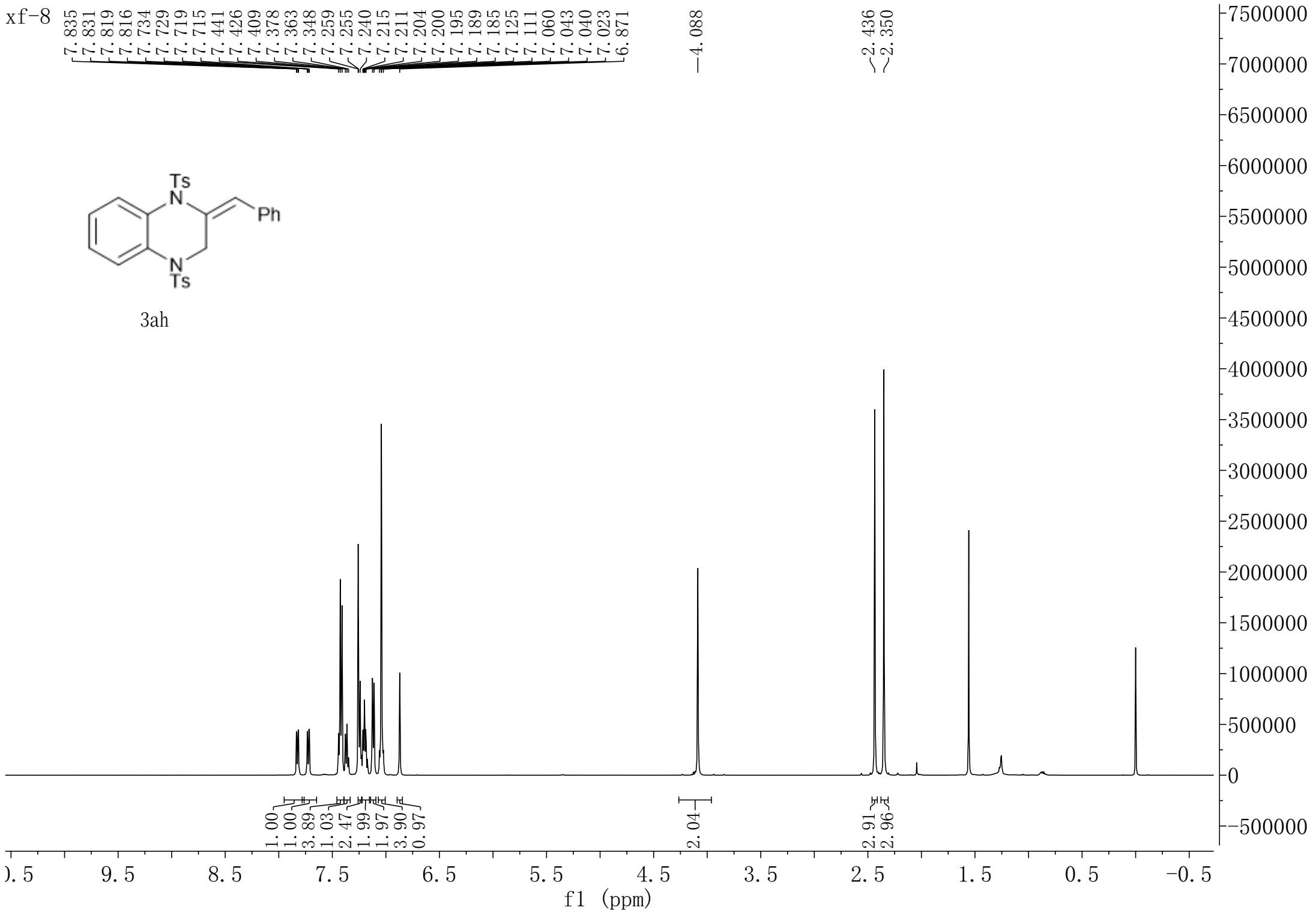
145.2
144.6
138.2
134.6
133.9
130.0
129.6
128.9
128.5
128.3
127.9
127.8
127.5
126.0
124.1
121.9
41.8
21.7
21.6
10.5
7.8
6500000
6000000
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
-500000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

xf-23

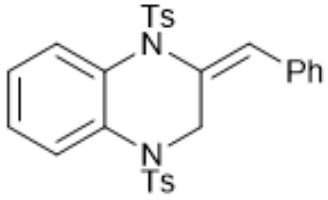


3dg

f1 (ppm)


xf-8
7.835
7.831
7.819
7.816
7.734
7.729
7.719
7.715
7.441
7.426
7.409
7.378
7.363
7.348
7.259
7.255
7.240
7.215
7.211
7.204
7.200
7.195
7.189
7.185
7.125
7.111
7.060
7.043
7.040
7.023
6.871
4.088
2.436
2.350
Ts
N
Ph
N
Ts
3ah
1.00
1.00
3.89
1.03
2.47
1.99
1.97
3.90
0.97
2.04
2.91
2.96
7500000
7000000
6500000
6000000
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
-500000
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

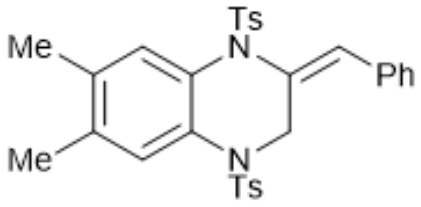
xf-8


Ts
N
Ph
N
Ts

3ah

144.9
143.9
135.2
134.6
134.1
130.9
129.9
129.5
129.3
129.2
128.8
128.4
127.8
127.1
127.0
125.9
125.7
125.1
122.9
43.1
21.7
21.6
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

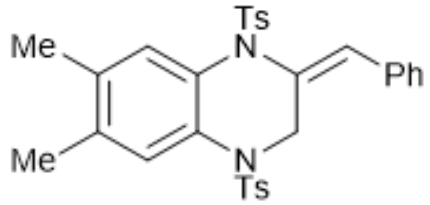
xf-26


Ts
N
Me
Ph
Me
N
Ts

3bh

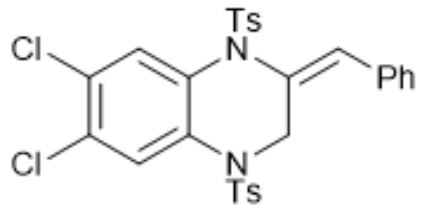
7.586
7.490
7.432
7.415
7.397
7.365
7.350
7.335
7.260
7.240
7.089
7.074
6.822
4.015
2.436
2.354
2.295
2.259
1.00
1.07
3.91
1.05
2.66
2.04
1.00
2.06
3.03
2.99
2.94
2.98
f1 (ppm)

xf-26



3bh

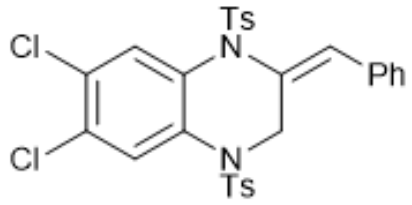
xf-28


Ts
N
Cl
Ph
Cl
N
Ts

3dh

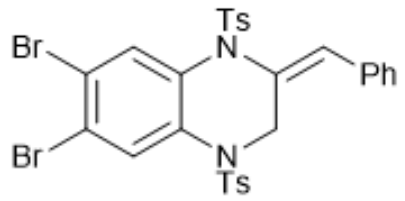
8.009
7.969
7.467
7.464
7.460
7.450
7.446
7.434
7.429
7.417
7.414
7.411
7.404
7.400
7.394
7.385
7.305
7.289
7.093
7.079
7.050
7.034
7.003
6.987
6.869
3.943
2.466
2.371
1.00
1.02
5.45
2.19
2.17
2.25
2.10
1.12
2.20
3.32
3.41
14000000
13000000
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
-1000000
5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

xf-28



3dh

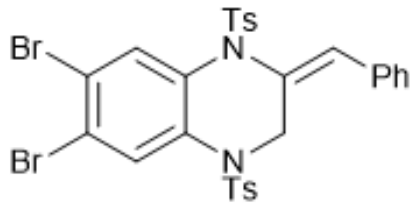
xf-29


Ts
N
Br
Ph
Br
N
Ts

3eh

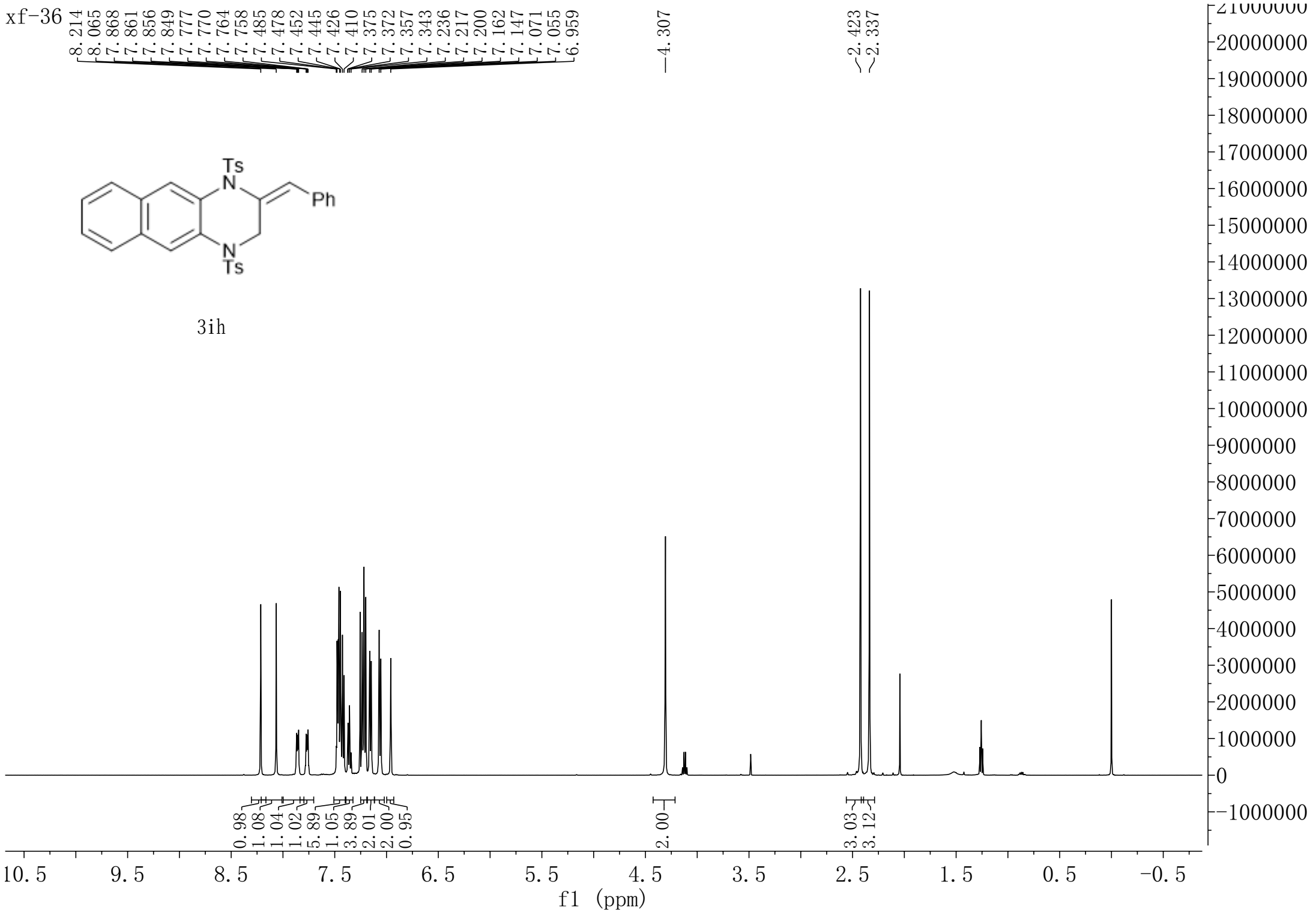
8.142
8.112
7.448
7.433
7.412
7.398
7.384
7.307
7.290
7.089
7.074
7.053
7.037
7.009
6.992
6.867
3.938
2.467
2.371
1.00
1.03
5.88
2.29
2.24
2.82
2.44
1.12
2.23
3.61
3.64
13000000
12000000
11000000
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
-1000000
9.0
8.5
8.0
7.5
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
-0.5
f1 (ppm)

xf-29

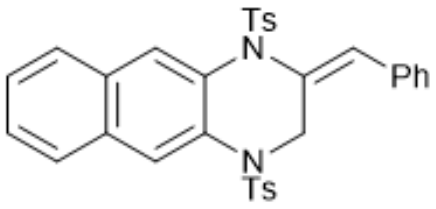

Ts
N
Br
Ph
Br
N
Ts

3eh

145.5
144.5
134.3
133.9
133.6
132.3
130.1
129.7
129.2
129.2
128.9
128.8
128.5
128.3
127.9
127.1
126.9
125.0
121.0
120.2
41.6
21.8
21.6
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
-200000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


xf-36
8.214
8.065
7.868
7.861
7.856
7.849
7.777
7.770
7.764
7.758
7.485
7.478
7.452
7.445
7.426
7.410
7.375
7.372
7.357
7.343
7.236
7.217
7.200
7.162
7.147
7.071
7.055
6.959
4.307
2.423
2.337
Ts
N
Ph
N
Ts
3ih
0.98
1.08
1.04
1.02
5.89
1.05
3.89
2.01
2.00
0.95
2.00
3.03
3.12
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

XF-36



3ih

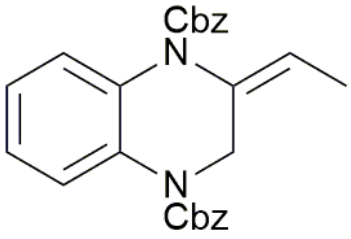
144.8
144.1
135.5
135.2
134.2
131.3
130.5
130.4
130.0
129.6
129.1
128.8
128.5
128.4
128.4
128.3
127.9
127.7
127.6
127.1
126.5
126.1
124.1
119.9
44.7
21.7
21.6

4000000
3800000
3600000
3400000
3200000
3000000
2800000
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
-200000
-400000

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

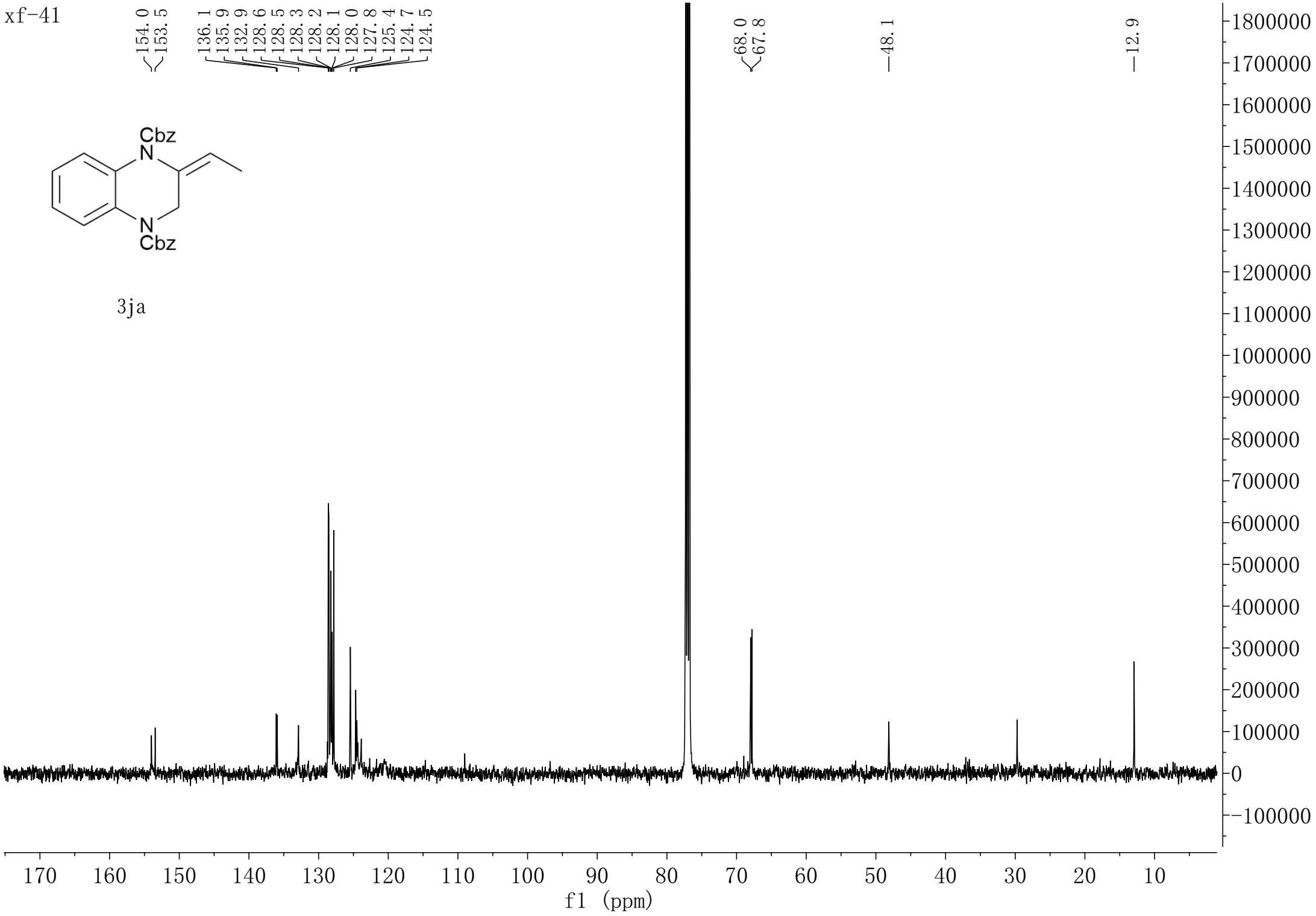
f1 (ppm)

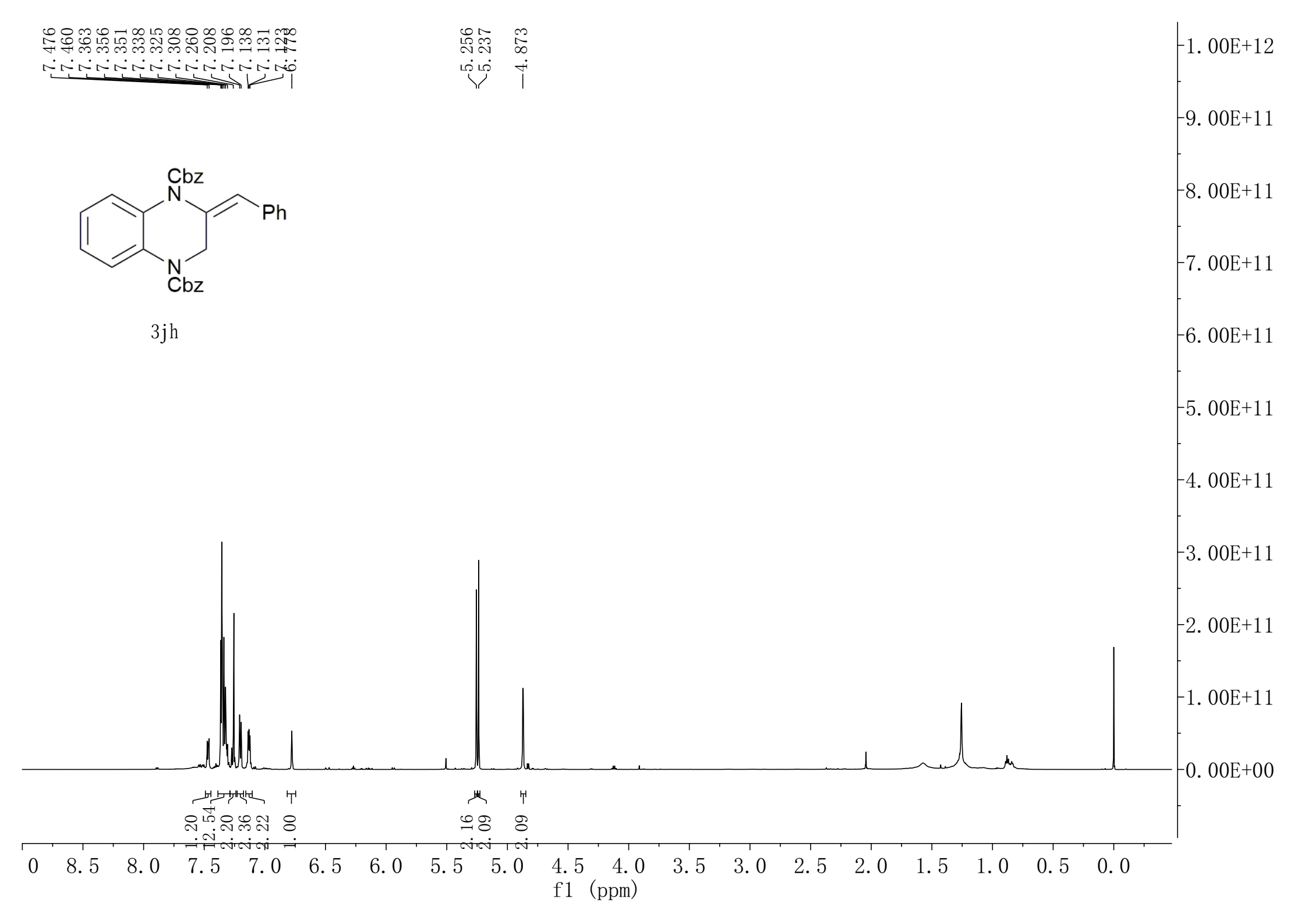
xf-41

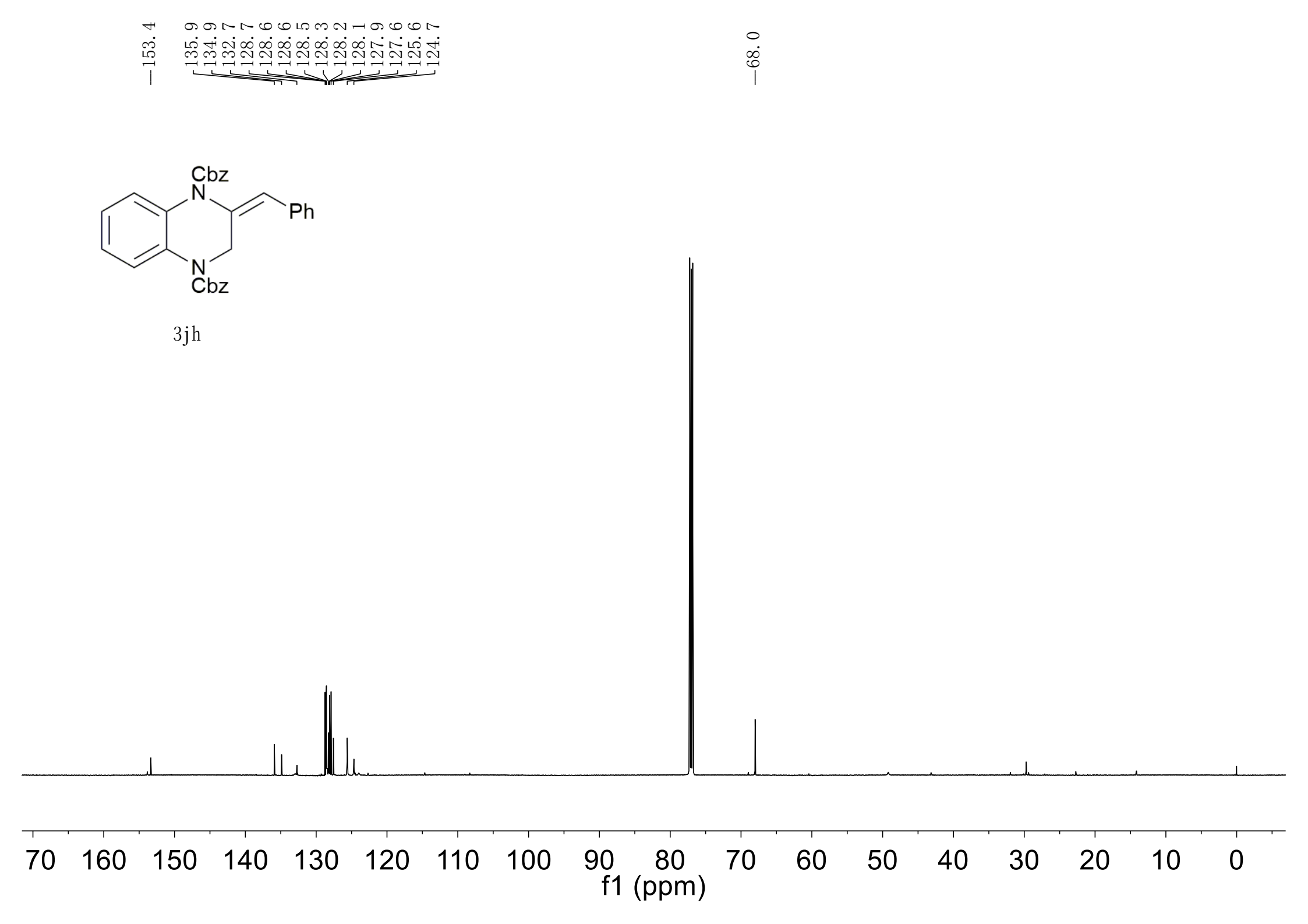

Cbz
N
N
Cbz

3ja

7.398
7.385
7.372
7.346
7.334
7.323
7.306
7.138
7.123
7.102
7.086
7.071
5.756
5.742
5.728
5.713
5.247
5.202
4.615
1.699
1.684
12.04
2.60
0.93
2.17
2.00
1.97
3.42
f1 (ppm)


xf-41
154.0
153.5
136.1
135.9
132.9
128.6
128.5
128.3
128.2
128.1
128.0
127.8
125.4
124.7
124.5
68.0
67.8
48.1
12.9
Cbz
N
N
Cbz
3ja
1800000
1700000
1600000
1500000
1400000
1300000
1200000
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
-100000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

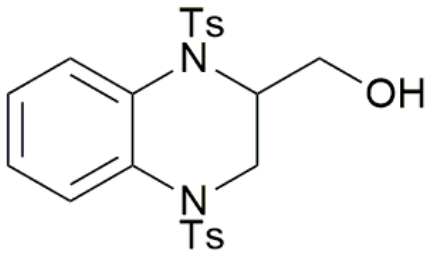

7.476
7.460
7.363
7.356
7.351
7.338
7.325
7.308
7.260
7.208
7.196
7.138
7.131
7.123
6.478
5.256
5.237
4.873
Cbz
N
Ph
N
Cbz
3jh
1.20
12.54
2.20
2.36
2.22
1.00
2.16
2.09
2.09
1.00E+12
9.00E+11
8.00E+11
7.00E+11
6.00E+11
5.00E+11
4.00E+11
3.00E+11
2.00E+11
1.00E+11
0.00E+00
0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


153.4
135.9
134.9
132.7
128.7
128.6
128.6
128.5
128.3
128.2
128.1
127.9
127.6
125.6
124.7
68.0
Cbz
N
Ph
N
Cbz
3jh
70
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

xf-B1

7.737
7.724
7.568
7.555
7.421
7.407
7.395
7.381
7.176
7.163
7.001
6.989
6.976
6.714
6.701
6.688
6.537
6.524

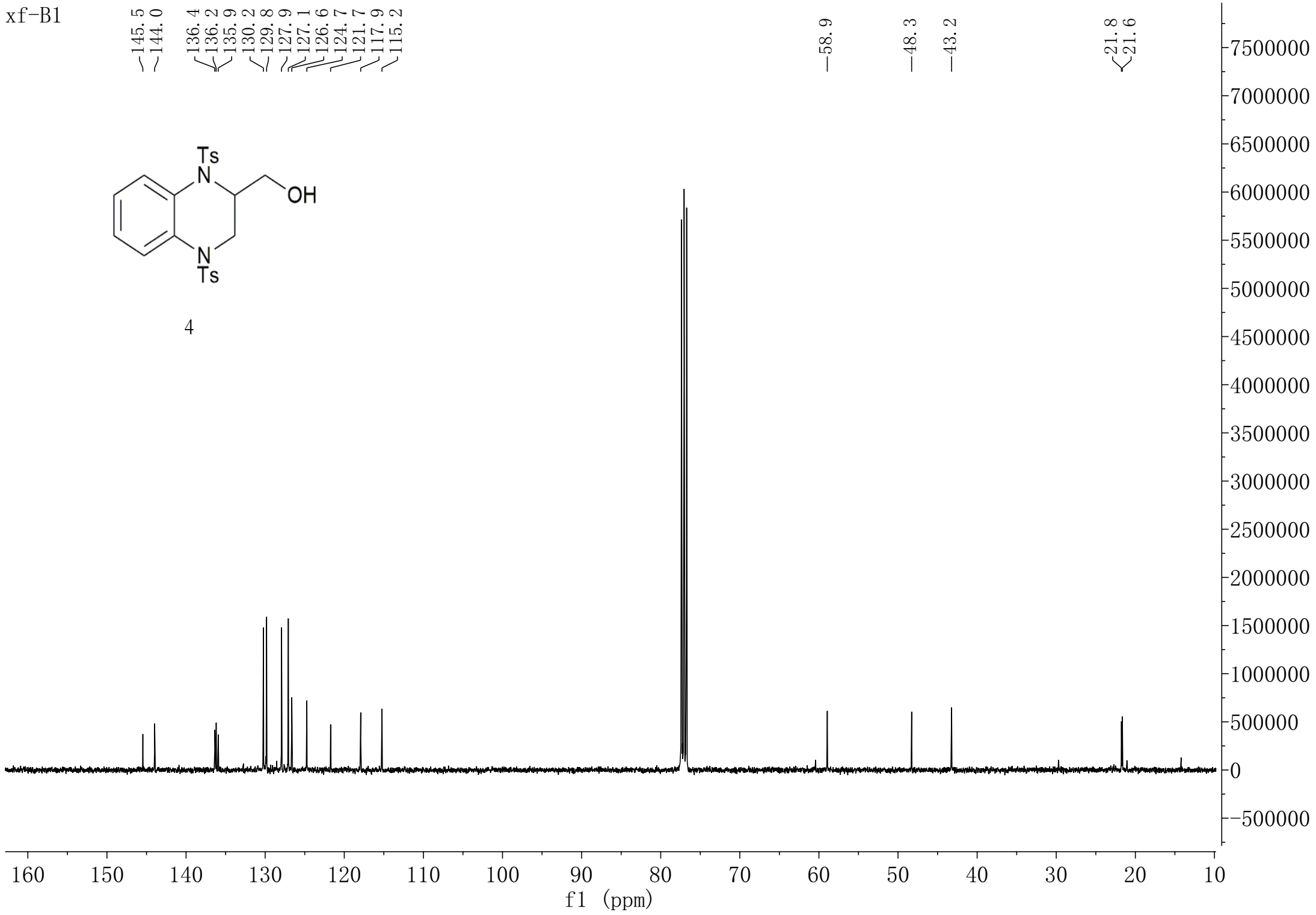
4.143
4.127
4.120
4.114
3.530
3.510
3.498
3.490
3.254
3.239
3.231
3.216
3.139
3.116
3.113
3.052
3.036
3.029
3.013
2.499
2.408



4

1.93
0.96
3.79
1.94
0.97
0.96
0.99
1.32
1.11
1.07
1.02
1.01
3.00
3.20

f1 (ppm)


xf-B1
145.5
144.0
136.4
136.2
135.9
130.2
129.8
127.9
127.1
126.6
124.7
121.7
117.9
115.2
58.9
48.3
43.2
21.8
21.6
Ts
N
OH
N
Ts
4
7500000
7000000
6500000
6000000
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
-500000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)