Asymmetric sequential Au(I)/chiral tertiary amine catalysis: an enone-formation/cyanosilylation sequence to optically active 3-alkenyloxindoles from diazooxindoles

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Supporting Information

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General: Reactions were monitored by thin layer chromatography using UV light to visualize the course of reaction. Purification of reaction products was carried out by flash chromatography on silica gel. Chemical yields refer to pure isolated substances. The $[\alpha]_D$ was recorded using PolAAr 3005 High Accuracy Polarimeter. Infrared (IR) spectra were obtained using a Bruker tensor 27 infrared spectrometer. ^{1}H, ^{13}C, and ^{19}F NMR spectra were obtained using a Bruker DPX-300, 400 or 500 spectrometer. Chemical shifts are reported in ppm from $CDCl_3$, $(CD_3)_2CO$ or DMSO-d_6 with the solvent resonance as the internal standard. The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad.

Anhydrous CH_2Cl_2 was prepared by first distillation over P_2O_5 and then from CaH_2. AgOTf and 2,5-dimethyl furan **2a** were purchased from TCI Company. 2,5-Diethylfuran was purchased from Beijing Donghualituo Technology Development Co. Ltd. Ph_3PAuCl was synthesized according to the literature method.[1] 3-Diazooxindoles **1**,[2] *p*-methoxyphenyldiazoacetate **5**[3] and chiral thiourea catalyst **C4**[4] were synthesized according to the literature procedures. TMSCN was purchased from Energy Chemical and was distilled before use.

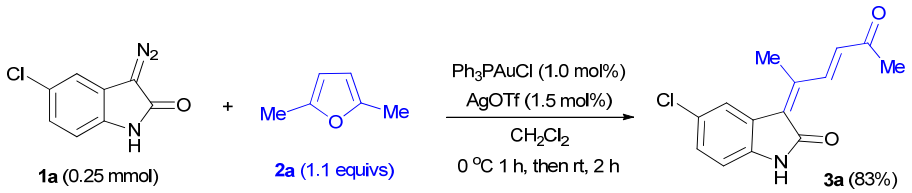
[1] T. N. Hooper, C. P. Butts, M. Green, M. F. Haddow, J. E. McGrady and C. A. Russell, *Chem. Eur. J.,* 2009, **15**, 12196.

[2] R. Augusti and C. Kascheres, *J. Org. Chem.,* 1993, **58**, 7079.

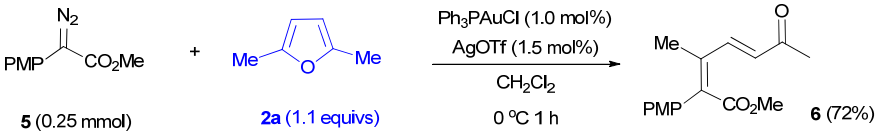
[3] C. C. Jing, D. Xing, Y. Qian, T.-D. Shi, Y. Zhao and W. H. Hu, *Angew. Chem., Int. Ed.*, 2013, **52**, 9289.

[4] a) A. Berkessel, S. Mukherjee, T. N. Müller, F. Cleemann, K. Roland, M. Brandenburg, J.-M. Neudörfl and J. Lex, *Org. Biomol. Chem.,* 2006, **4**, 4319; b) G. Tárkányi, P. Király, S. Varga, B. Vakulya and T. Soós, *Chem. Eur. J.*, 2008, **14**, 6078; c) B. Vakulya, S. Varga and T. Soós, *J. Org. Chem.,* 2008, **73**, 3475; d) A. Peschiulli, C. Quigley, S. Tallon, Y. K. Gun'ko and S. J. Connon, *J. Org. Chem*., 2008, **73**, 6409; e) B. Vakulya, S. Varga, A. Csámpai and T. Soós, *Org. Lett.,* 2005, **7**, 1967.

1)The Au(I)-catalyzed enone formation from diazo reagents 1a or 5 and 2,5-dimethyl furan 2a.



Under an atmosphere of nitrogen, to a Schlenk tube were added Ph_3PAuCl (1.2 mg, 0.0025 mmol) and AgOTf (1.0 mg, 0.0038 mmol), followed by anhydrous CH_2Cl_2 (2.5 mL). The resulting mixture was stirred at room temperature for 15-20 minutes, and then cooled down to 0 ^oC, followed by the successive addition of 2,5-dimethylfuran **2a** (0.28 mmol) and diazooxindole **1a** (0.25 mmol, 48.4 mg) in one portion. The reaction was kept stirring at -0 ^oC for 1.0 h, and then at rt for 2 h. Column chromatographic purification using CH_2Cl_2/acetone (from 100:1 to 20:1) as the eluent gave product **3a** as red solid (m.p. 131-133 ^oC). ^1H NMR (400 MHz, DMSO-d_6): δ 10.81 (s, 1H), 9.23 (d, J = 16.4 Hz, 1H), 7.63 (d, J = 2.0 Hz, 1H), 7.30 (d, J = 2.0 Hz, 1H), 6.85 (d, J = 8.0 Hz, 1H), 6.75 (d, J = 2.0 Hz, 1H), 2.42 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 198.54, 167.72, 146.40, 140.91, 139.26, 133.44, 129.35, 127.43, 125.45, 125.17, 124.62, 110.84, 27.83, 16.58; IR (ATR): 3085, 2853, 1674, 1647, 1603, 1251, 1308, 1080; HRMS (ESI): Exact mass calcd for $C_{14}H_{13}NO_2{}^{35}Cl$ $[M+H]^+$: 262.0629, Found: 262.0630.



PMP = *p*-methoxyphenyl

The procedure was similar to that of previous experiment expect that the diazo **5** was added using syringe pump within one hour. The product **6** was obtained as green oil. ^1H NMR (300 MHz, $CDCl_3$): δ 7.76 (ABd, J = 16.0 Hz, 1H), 7.14 (ABd, J = 8.5 Hz, 2H), 6.88 (ABd, J = 9.0 Hz, 2H), 6.31 (ABd, J = 16.0 Hz, 1H), 3.80 (s, 3H), 3.76 (s, 3H), 2.32 (s, 3H), 1.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.73, 168.60, 159.38, 141.87, 139.33, 136.94, 130.35, 130.17, 128.43, 113.74, 55.15, 52.28, 27.09, 15.94; IR (ATR): 3003, 2952, 2838, 1723, 1593, 1510, 1360, 1248, 1204; GC-MS: 274 (M^+,3), 215 (100), 172 (13), 128 (10), 231 (7), 157 (6), 243 (3), 200 (3); HRMS (EI): Exact mass calcd for $C_{16}H_{18}O_4$ $[M]^+$: 274.1205, Found: 274.1201.

2) The assignment of the configuration of enone 6 by NMR analysis

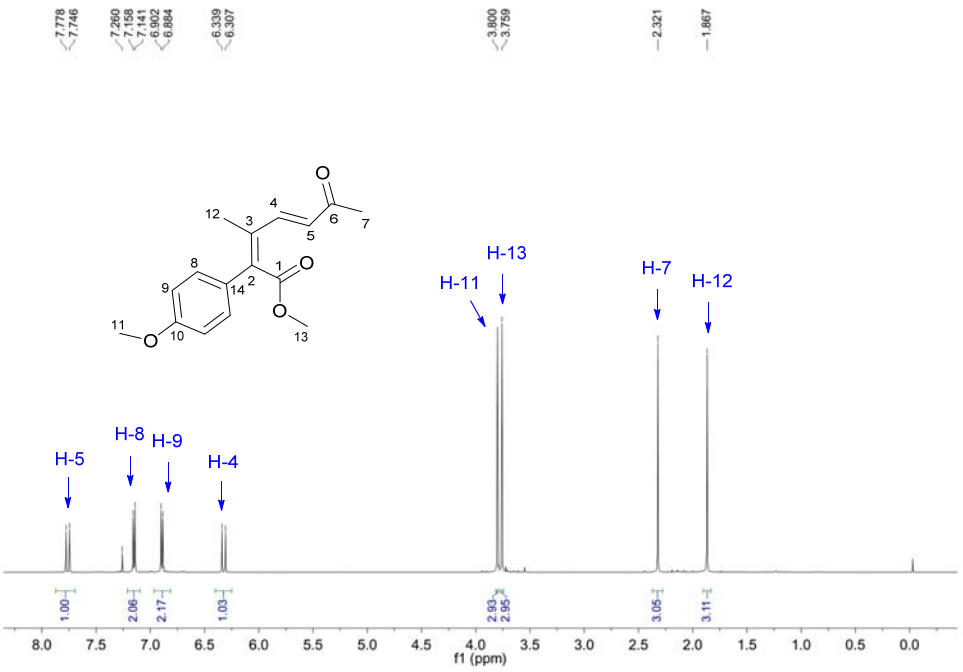


Figure 1. ^{1}H NMR spectrum of **6** ($CDCl_3$, 500M HZ)

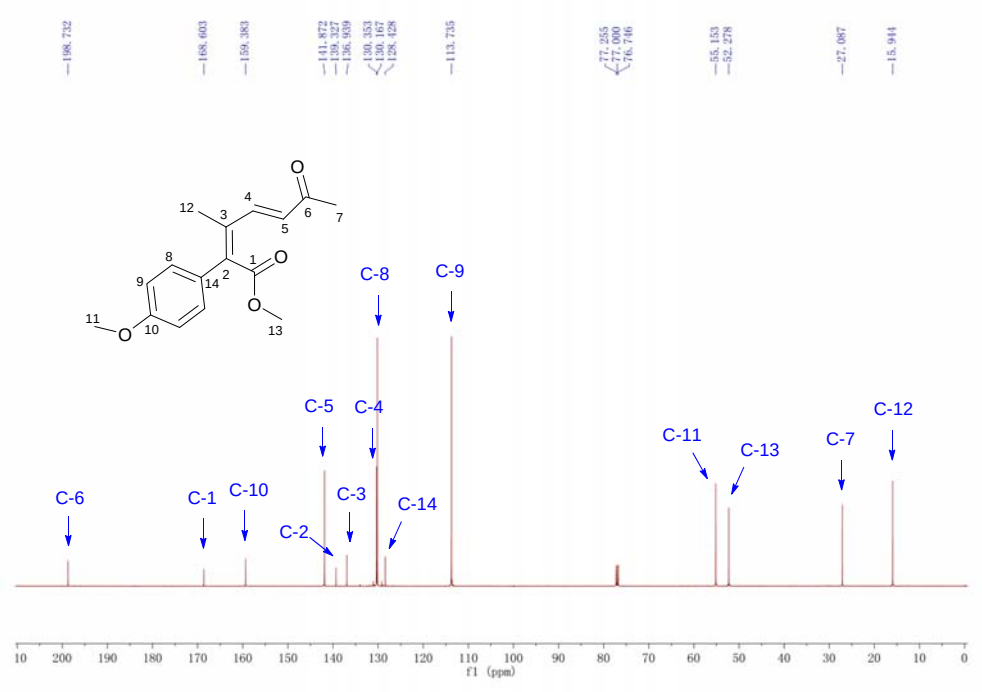


Figure 2. ^{13}C NMR spectrum of **6** ($CDCl_3$, 500M HZ)

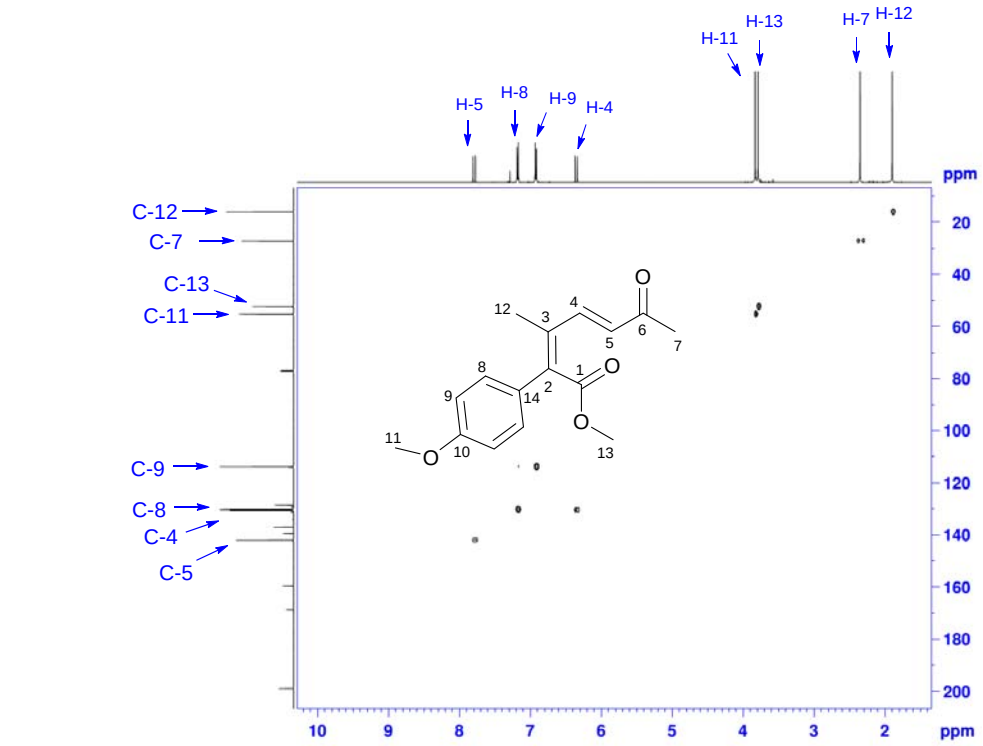


Figure 3. HSQC spectrum of **6** ($CDCl_3$, 500M HZ)

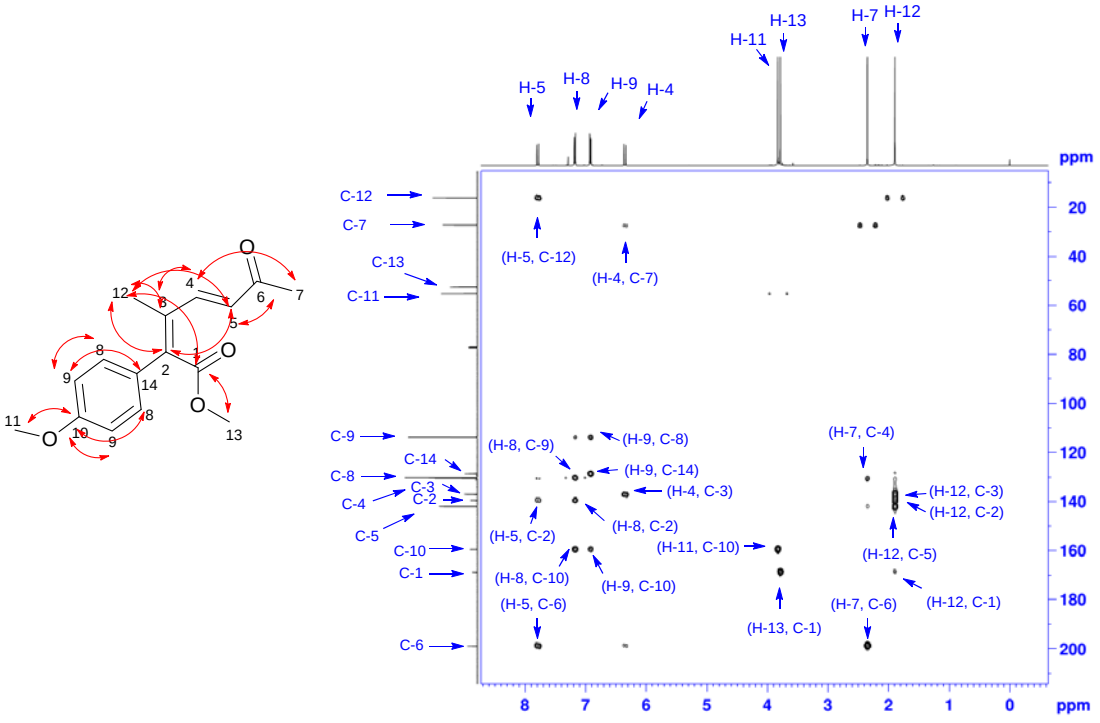


Figure 4. HMBC spectrum of **6** ($CDCl_3$, 500M HZ)

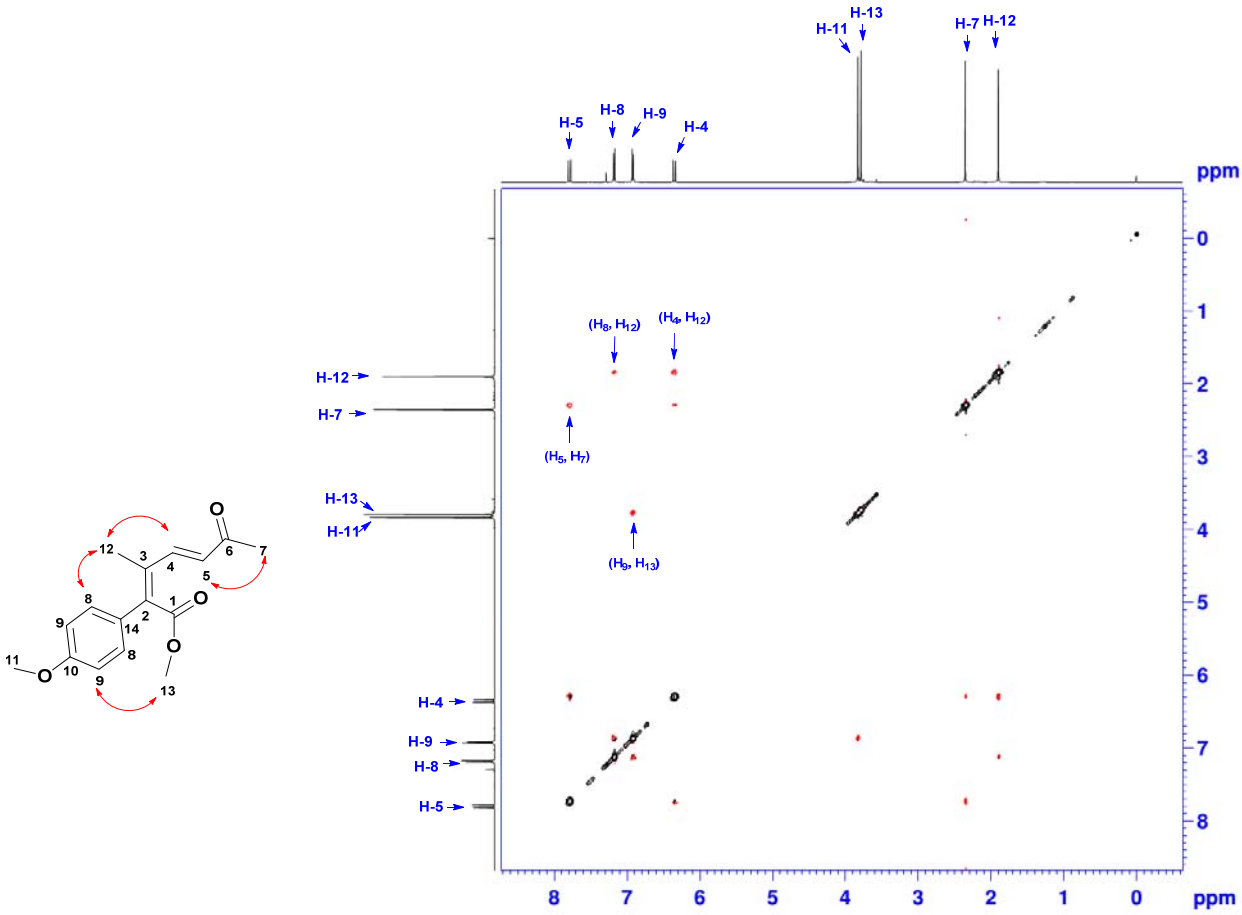
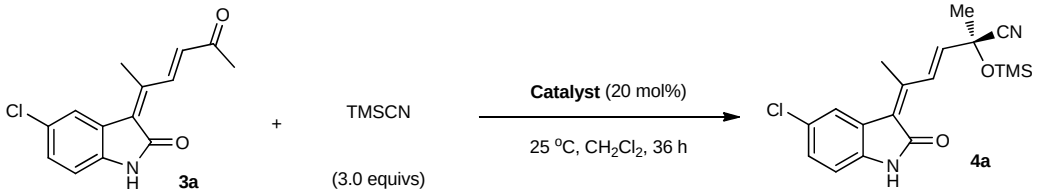


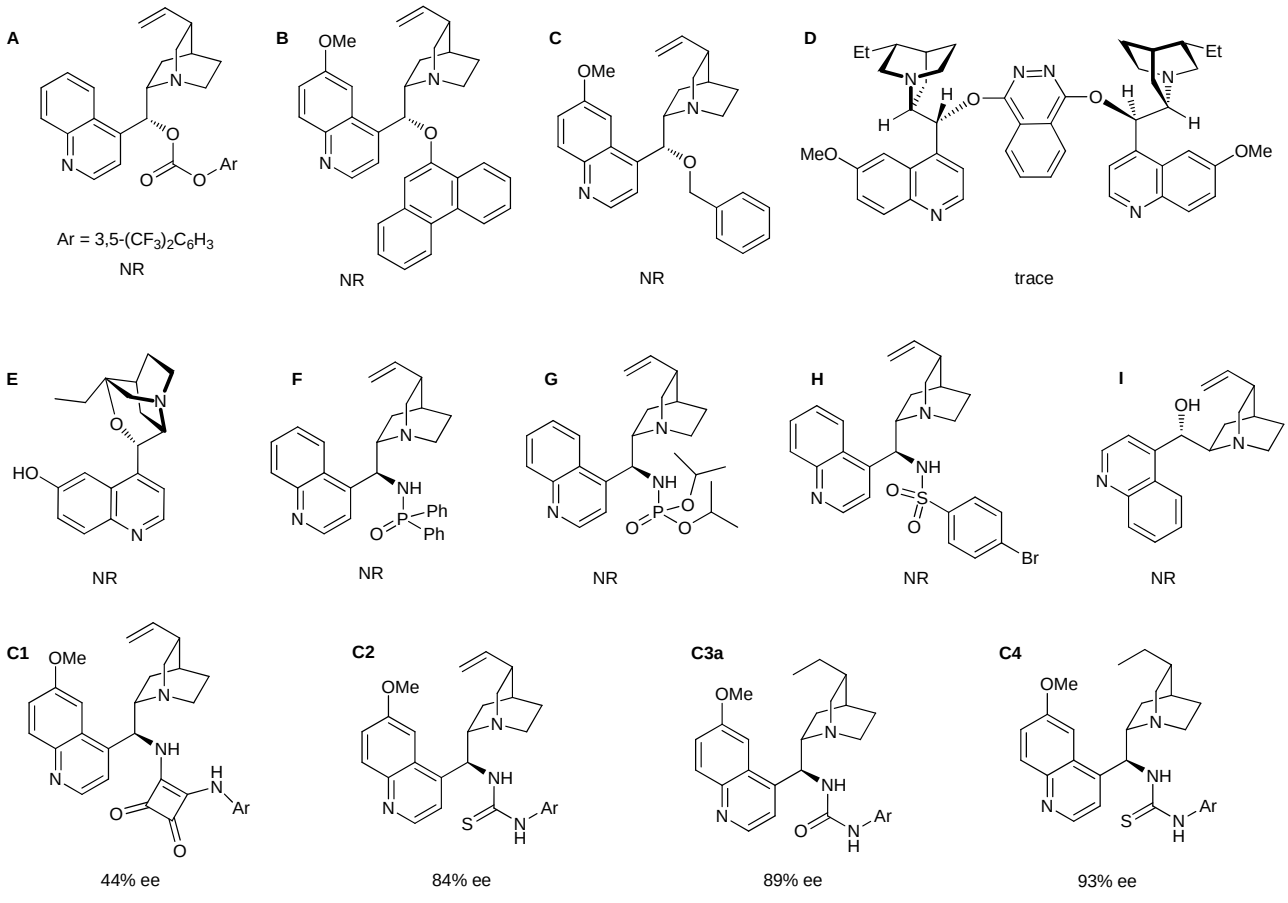
Figure 5. Key NOESY correlations of **6** ($CDCl_3$, 500M HZ)

3) Condition optimization for the asymmetric cyanosilylation reaction.

The asymmetric cyanosilylation reaction of enone **3a** using TMSCN was studied by using a variety of cinchona alkaloid derivatives, see Table S1. For a quick optimization of a suitable catalyst, all the reactions were run in CH_2Cl_2 at 25 ^oC using 3.0 equivs of TMSCN, and took a sample for preparative TLC separation to get some product for HPLC analysis after 36 h, no matter the reaction was completed or not. So the conversion was not detected.

Table S1. The optimization of chiral catalysts for asymmetric cyanosilylation reaction[a,b]

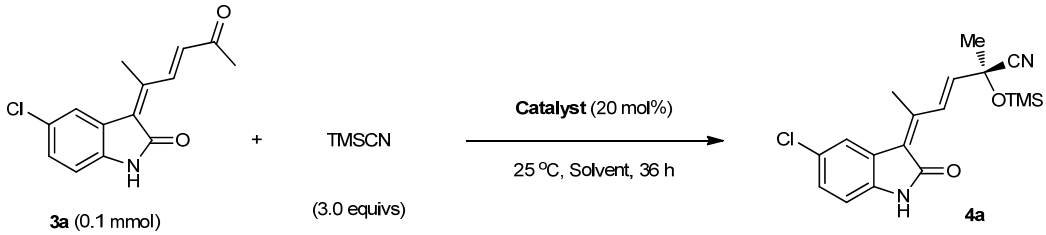




a) 0.05 mmol scale in 0.5 mL of CH_2Cl_2; b) Ee value was determined by HPLC analysis.

After screening of a variety of chiral tertiary amine based Lewis bases, cinchona alkaloid derived bifunctional thiourea **C4** proved to be a promising catalyst, which could achieve 93% ee for the desired product **4a**. A similar urea catalyst **C3a** afforded the product **4a** in slightly lower 89% ee, but it could catalyze the reaction in a faster rate. So we use **C3a** for the optimization of reaction solvent, and CH_2Cl_2 still proved to be the best choice, allowing the reaction to finish within 36 h to give afford product **4a** in 69% yield with 90% ee (entry 1 vs entries 2-6, Table S2). On the other hand, when the corresponding chiral thiourea **C4** was used as the catalyst, the reaction could finish with 60 h, giving the desired product **4a** in 68% yield with 93% ee (entry 7).

Table S2. Solvent effects for the asymmetric cyanosilylation reaction

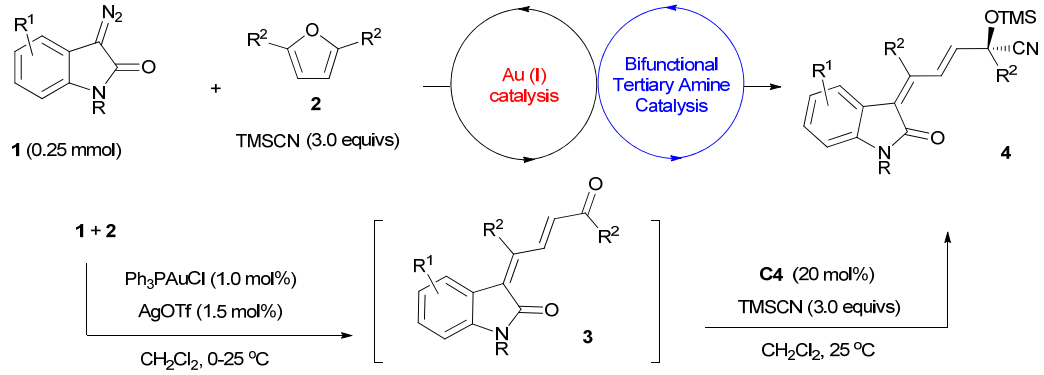


Entry	Catalyst	Solvent	Yield (%)[a]	Ee (%)[b]
1	**C3a**	CH_2Cl_2	69	90
2	**C3a**	CH_3CN	68	-35
3	**C3a**	THF	trace	ND
4	**C3a**	PhMe	27	71
5	**C3a**	acetone	NR	ND
6	**C3a**	Et_2O	NR	ND
7	**C4**	CH_2Cl_2	68[c]	93

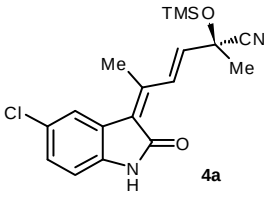
a) Isolated yield; b) Determined by HPLC analysis; c) 60 h.

The above results suggested that the best solvent for the **C4** catalyzed asymmetric cyanosilylation was CH_2Cl_2. We further tried using CH_2Cl_2 as the solvent for the Au(I) catalyzed reaction of 5-chlorodiazooxindole **1a** with 2,5-dimethylfuran **2a**. It was found that under the catalysis of 1.0 mol% of $Ph_3PAuOTf$, **1a** worked well with 2,5-dimethylfuran **2a** (1.1 equivs), and the reaction could finish within 2 hours (1 h at 0 ^oC and 1 h at 25 ^oC), to afford oxindole based enone **3a** in 83% yield. Therefore, a convenient one-pot cyclopropanation/ring-opening/asymmetric cyanosilylation was developed, without changing the reaction solvent.

4) General procedure for the one-pot enone-formation/cyanosilylation sequence.



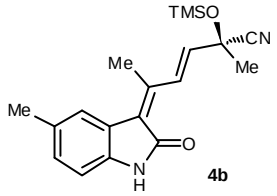
Under nitrogen atmosphere, to a Schlenk tube were added Ph_3PAuCl (1.2 mg, 0.0025 mmol) and AgOTf (1.0 mg, 0.00375 mmol), followed by anhydrous CH_2Cl_2 (2.5 mL). The resulting mixture was stirred at room temperature for 15-20 minutes. The reaction was cooled down to 0 ^oC, followed by the addition of substituted furan **2** (0.28 mmol) and diazooxindole **1** (0.25 mmol). The reaction was kept stirring at 0 ^oC for about 1 h, and then was warmed to 25 ^oC for 1-8 hours. Then chiral catalyst **C4** (29.8 mg, 0.05 mmol) and TMSCN (94.0 uL, 0.75 mmol) were added sequentially. The resulting mixture was stirring at 25 ^oC about 1.5 to 6.5 days till almost full conversion of oxindole-enone **3** by TLC analysis. The mixture was carefully concentrated under reduced pressure. The residue was subjected to column chromatography for purification, using acetone/CH_2Cl_2 (from 0:1 to 1:100, v:v) or petroleum ether/CH_2Cl_2 (from 2:1 to 0:1, v:v) as the eluent.



4a

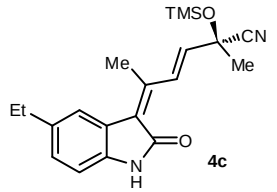
The total reaction time was 7 days. Product **4a** was obtained in 59% yield as yellow solid. $[\alpha]^{25}_D$ = -29.0 (c = 0.65, CH_2Cl_2), m.p. 147-149 ^oC; HPLC analysis (Chiralpak AD-H, ^{i}PrOH/hexane = 10/90, 1.0 mL/min, 230 nm; t_r (major) = 7.79 min, t_r (minor) = 9.21 min) gave the isomeric composition of the product: 94% ee; ^1H NMR (400 MHz, acetone-d_6): δ 9.61 (s, 1H), 8.95 (d, J = 16.0 Hz, 1H), 7.64 (d, J = 1.6 Hz, 1H), 7.24 (d, J = 2.0 Hz, 1H), 6.93 (d, J = 8.4 Hz, 1H), 6.58 (d, J = 16.0 Hz, 1H), 2.51 (s, 3H), 1.78 (s, 3H), 0.28 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 168.82, 147.92, 141.40, 139.21, 129.91, 129.39, 126.98, 126.51, 125.78, 125.55, 121.40, 111.44, 70.78, 30.90, 17.39, 1.49; IR (ATR): 3159, 2960, 1684, 1584, 1449, 1254, 1208, 1129; HRMS (ESI): Exact mass calcd for $C_{18}H_{21}N_2O_2NaSi^{35}Cl$

$[M+Na]^+$: 383.0959, Found: 383.0964.



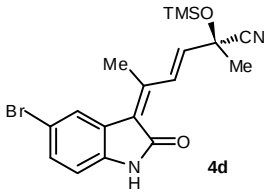
4b

The total reaction time was 3 days. Product **4b** was obtained in 77% yield as yellow solid. $[\alpha]^{25}_D$ = -13.3 (c = 0.54, CH_2Cl_2), m.p. 203-205 ^oC; HPLC analysis (Chiralpak AD-H, ^{i}PrOH/hexane = 10/90, 1.0 mL/min, 230 nm; t_r (major) = 9.06 min, t_r (minor) = 11.58 min) gave the isomeric composition of the product: 96% ee; ^1H NMR (400 MHz, acetone-d_6): δ 9.38 (s, 1H), 8.99 (d, *J* = 16.0 Hz, 1H), 7.52 (s, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.47 (d, *J* = 16.0 Hz, 1H), 2.48 (s, 3H), 2.33 (s, 3H), 1.77 (s, 3H), 0.27 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 169.23, 145.33, 140.52, 137.69, 131.27, 130.30, 130.26, 126.79, 126.65, 125.09, 121.48, 109.99, 70.78, 30.90, 21.36, 17.20, 1.46; IR (ATR): 3160, 2997, 1681, 1621, 1584, 1453, 1320, 1252; HRMS (ESI): Exact mass calcd for $C_{19}H_{28}N_3O_2Si$ $[M+NH_4]^+$: 385.1945, Found: 385.1953.



4c

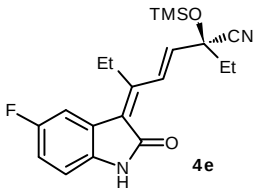
The total reaction time was 3 days. Product **20f** was obtained in 70% yield as yellow solid. $[\alpha]^{25}_D$ = -17.4 (c = 0.41, CH_2Cl_2), m.p. 178-180 ^oC; HPLC analysis (Chiralpak AD-H, ^{i}PrOH/hexane = 5/95, 1.0 mL/min, 230 nm; t_r (major) = 16.04 min, t_r (minor) = 23.28 min) gave the isomeric composition of the product: 90% ee; ^1H NMR (400 MHz, acetone-d_6): δ 9.39 (s, 1H), 8.99 (d, *J* = 16.0 Hz, 1H), 7.54 (s, 1H), 7.09 (d, *J* = 8.0 Hz, 1H), 6.83 (d, *J* = 7.6 Hz, 1H), 6.47 (d, *J* = 16.0 Hz, 1H), 2.64 (q, *J* = 7.6 Hz, 2H), 2.49 (s, 3H), 1.77 (s, 3H), 1.22 (t, *J* = 7.6 Hz, 3H), 0.27 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 169.27, 145.29, 140.74, 138.10, 137.66, 130.31, 129.21, 126.72, 125.78, 125.10, 121.48, 110.08, 70.79, 30.90, 17.23, 16.66, 1.47; IR (ATR): 3062, 2932, 1623, 1583, 1457, 1375, 1339, 1209; HRMS (ESI): Exact mass calcd for $C_{20}H_{30}N_3O_2Si$ $[M+NH_4]^+$: 372.2102, Found: 372.2108.



4d

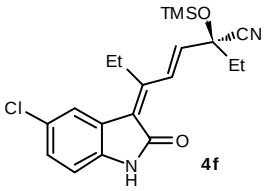
The total reaction time was 5 days. Product **4d** was obtained in 40% yield as yellow solid. $[\alpha]^{25}_D$ = -77.4 (c = 0.28, CH_2Cl_2), m.p. 206-208 ^oC; HPLC analysis (Chiralpak AD-H, ^{i}PrOH/hexane = 5/95, 1.0 mL/min, 230 nm; t_r (major) = 13.53 min, t_r (minor) = 16.87 min) gave the isomeric composition of the product: 92% ee; ^1H NMR (400 MHz, acetone-d_6): δ 9.63 (s, 1H), 8.95 (d, *J* = 15.6 Hz, 1H), 7.78 (s, 1H), 7.40 (d, *J* =

8.0 Hz, 1H), 6.90 (d, J = 8.4 Hz, 1H), 6.58 (d, J = 15.6 Hz, 1H), 2.51 (s, 3H), 1.78 (s, 3H), 0.28 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 168.61, 147.91, 141.73, 139.19, 132.24, 129.83, 128.46, 126.93, 125.36, 121.33, 114.21, 111.90, 70.71, 30.84, 17.34, 1.43; IR (ATR): 3084, 1684, 1613, 1564, 1444, 1372, 1308, 1208; HRMS (ESI): Exact mass calcd for $C_{18}H_{25}N_3O_2Si^{79}Br$ $[M+NH_4]^+$: 422.0894, Found: 422.0898.



4e

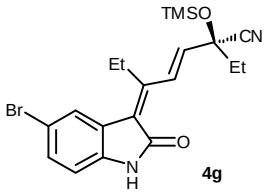
The total reaction time was 3 days. Product **4e** was obtained in 72% yield as yellow solid. $[\alpha]^{25}_D$ = -37.3 (c = 0.60, CH_2Cl_2), m.p. 179-181 ^oC; HPLC analysis (Chiralpak AD-H, ^{i}PrOH/hexane = 5/95, 1.0 mL/min, 230 nm; t_r (major) = 10.16 min, t_r (minor) = 13.33 min) gave the isomeric composition of the product: 96% ee; ^{1}H NMR (400 MHz, acetone-d_6): δ 9.53 (s, 1H), 8.90 (d, J = 16.4 Hz, 1H), 7.36 (dd, J = 10.0, 2.4 Hz, 1H), 7.03 (td, J = 8.4, 2.4 Hz, 1H), 6.94-6.91 (m, 1H), 6.53 (d, J = 16.0 Hz, 1H), 2.95 (q, J = 7.6 Hz, 2H), 1.97 (q, J = 7.6 Hz, 2H), 1.30 (t, J = 7.6 Hz, 3H), 1.07 (t, J = 7.6 Hz, 3H), 0.28 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 169.38, 159.30 (d, J_{F-C} = 233.0 Hz), 153.30, 139.03, 137.67, 129.48, 125.04 (d, J_{F-C} = 3.0 Hz), 124.83 (d, J_{F-C} = 8.0 Hz), 120.50, 115.98 (d, J_{F-C} = 24.0 Hz), 112.61 (d, J_{F-C} = 26.0 Hz), 110.85 (d, J_{F-C} = 9.0 Hz), 75.13, 36.95, 22.41, 13.10, 8.67, 1.36; ^{19}F NMR (376 MHz, acetone-d_6): δ -123.18; IR (ATR): 3175, 2967, 2933, 1686, 1632, 1491, 1323, 1306, 1276, 1197; HRMS (ESI): Exact mass calcd for $C_{20}H_{29}N_3O_2FSi$ $[M+NH_4]^+$: 390.2008, Found: 390.2014.



4f

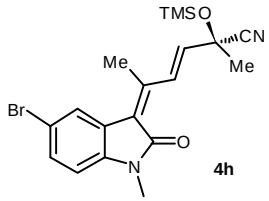
The total reaction time was 3 days. Product **4f** was obtained in 69% yield as yellow solid. $[\alpha]^{25}_D$ = -19.2 (c = 0.55, CH_2Cl_2), m.p. 173-175 ^oC; HPLC analysis (Chiralpak AD-H, ^{i}PrOH/hexane = 10/90, 1.0 mL/min, 230 nm; t_r (major) = 5.91 min, t_r (minor) = 7.22 min) gave the isomeric composition of the product: 94% ee; ^{1}H NMR (400 MHz, acetone-d_6): δ 9.64 (s, 1H), 8.88 (d, J = 16.0 Hz, 1H), 7.56 (d, J = 2.0 Hz, 1H), 7.27 (dd, J = 8.0, 1.6 Hz, 1H), 6.95 (d, J = 8.4 Hz, 1H), 6.54 (d, J = 16.0 Hz, 1H), 2.96 (q, 7.6 Hz, 2H), 1.97 (q, J = 7.6 Hz, 2H), 1.30 (t, J = 7.6 Hz, 3H), 1.07 (t, J = 7.6 Hz, 3H), 0.28 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 169.06, 153.60, 141.48, 137.90, 129.47, 129.45, 127.12, 125.45, 125.14, 124.48, 120.50, 111.55, 75.13, 36.96, 23.56, 13.10, 8.67, 1.36; IR (ATR): 3172, 2967, 2873, 1686, 1578, 1419, 1302, 1224, 1127; HRMS (ESI): Exact mass calcd for $C_{20}H_{29}N_3O_2Si^{35}Cl$ $[M+NH_4]^+$: 406.1712, Found:

406.1717.



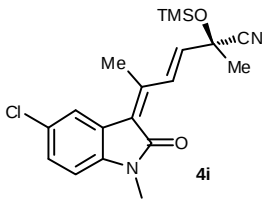
4g

The total reaction time was 3 days. Product **4g** was obtained in 61% yield as yellow solid. $[\alpha]^{25}_{D}$ = -48.9 (c = 0.45, CH_2Cl_2), m.p. 178-180 ^{o}C; HPLC analysis (Chiralpak AD-H, ^{i}PrOH/hexane = 5/95, 1.0 mL/min, 230 nm; t_r (major) = 9.48 min, t_r (minor) = 12.11 min) gave the isomeric composition of the product: 93% ee; ^{1}H NMR (400 MHz, acetone-d_6): δ 9.67 (s, 1H), 8.88 (d, *J* = 16.0 Hz, 1H), 7.68 (d, *J* = 1.6 Hz, 1H), 7.40 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.91 (d, *J* = 8.4 Hz, 1H), 6.53 (d, *J* = 16.0 Hz, 1H), 2.94 (q, *J* = 7.6 Hz, 2H), 1.97 (q, *J* = 7.6 Hz, 2H), 1.30 (t, *J* = 7.6 Hz, 3H), 1.07 (t, *J* = 7.6 Hz, 3H), 0.28 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 168.91, 153.61, 141.81, 137.90, 132.35, 129.39, 127.85, 125.90, 124.31, 120.47, 114.39, 112.05, 75.10, 36.93, 23.56, 13.08, 8.67, 1.36; IR (ATR): 3162, 2971, 1683, 1612, 1482, 1328, 1298, 1251, 1129; HRMS (ESI): Exact mass calcd for $C_{20}H_{29}N_3O_2Si^{79}Br$ $[M+NH_4]^+$: 450.1207, Found: 450.1211.



4h

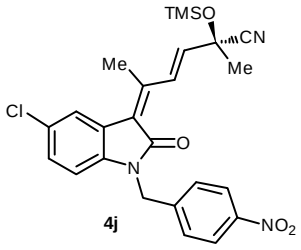
The total reaction time was 1.5 days. Product **4h** was obtained in 87% yield as yellow solid. $[\alpha]^{25}_{D}$ = -4.4 (c = 0.54, CH_2Cl_2), m.p. 152-154 ^{o}C; HPLC analysis (Chiralpak IE, ^{i}PrOH/hexane = 5/95, 1.0 mL/min, 230 nm; t_r (major) = 27.25 min, t_r (minor) = 23.69 min) gave the isomeric composition of the product: 95% ee; ^{1}H NMR (400 MHz, acetone-d_6): δ 8.99 (d, *J* = 15.6 Hz, 1H), 7.79 (d, *J* = 1.6 Hz, 1H), 7.48 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.95 (d, *J* = 8.4 Hz, 1H), 6.59 (d, *J* = 16.0 Hz, 1H), 3.22 (s, 3H), 2.52 (s, 3H), 1.79 (s, 3H), 0.28 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6) : δ 167.05, 148.00, 143.31, 139.38, 132.17, 129.84, 128.07, 125.95, 124.66, 121.29, 114.59, 110.45, 70.72, 30.82, 25.95, 17.35, 1.44; IR (ATR): 3435, 2959, 1692, 1606, 1483, 1253, 1214, 1106; HRMS (ESI): Exact mass calcd for $C_{19}H_{27}N_3O_2Si^{79}Br$ $[M+NH_4]^+$: 436.1050, Found: 436.1053.



4i

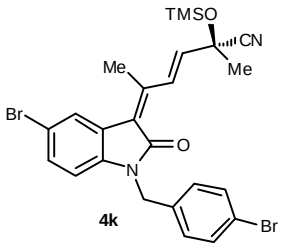
The total reaction time was 2 days. Product **4i** was obtained in 80% yield as yellow solid. $[\alpha]^{25}_{D}$ = -3.6 (c = 0.49, CH_2Cl_2), m.p. 134-136 ^{o}C; HPLC analysis (Chiralpak IE, ^{i}PrOH/hexane = 10/90, 1.0 mL/min, 230 nm; t_r (major) = 15.39 min, t_r (minor) = 13.76 min) gave the isomeric composition of the product: 94% ee; ^{1}H NMR (400 MHz, acetone-d_6): δ 8.99 (d, *J* = 16.0 Hz, 1H), 7.66 (d, *J* = 1.6 Hz, 1H), 7.34 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.98 (d, *J* = 8.0 Hz, 1H), 6.60 (d, *J* = 15.6 Hz, 1H), 3.21 (s, 3H), 2.52 (s, 3H), 1.78 (s,

3H), 0.28 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 167.11, 147.89, 142.86, 139.29, 129.87, 129.19, 127.22, 125.45, 125.33, 124.74, 121.29, 109.85, 70.71, 30.82, 25.97, 17.33, 1.45; IR (ATR): 3435, 2961, 1692, 1608, 1485, 1254, 1210, 1141, 1109; HRMS (ESI): Exact mass calcd for $C_{19}H_{27}N_3O_2Si^{35}Cl$ $[M+NH_4]^+$: 392.1556, Found: 392.1560.



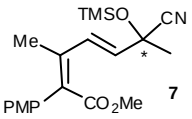
4j

The total reaction time was 3 days. Product **4j** was obtained in 82% yield as yellow solid. $[\alpha]^{25}_D$ = -1.4 (c = 0.43, CH_2Cl_2), m.p. 50-52 ^oC; HPLC analysis (Chiralpak IE, ^{i}PrOH/hexane = 15/85, 1.0 mL/min, 230 nm; t_r (major) = 27.01 min, t_r (minor) = 24.89 min) gave the isomeric composition of the product: 92% ee; ^{1}H NMR (400 MHz, acetone-d_6): δ 9.01 (d, *J* = 16.0 Hz, 1H), 8.22 (ABd, *J* = 8.8 Hz, 2H), 7.74 (d, *J* = 2.0 Hz, 1H), 7.63 (ABd, *J* = 8.8 Hz, 2H), 7.27 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.93 (d, *J* = 8.4 Hz, 1H), 6.67 (d, *J* = 15.6 Hz, 1H), 5.19 (s, 2H), 2.59 (s, 3H), 1.80 (s, 3H), 0.29 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 167.41, 149.28, 148.36, 145.21, 141.44, 140.08, 129.76, 129.25, 129.19, 127.84, 125.83, 125.77, 124.64, 124.35, 121.26, 110.60, 70.76, 43.12, 30.79, 17.58, 1.45; IR (ATR): 3435, 2960, 1692, 1607, 1525, 1344, 1254, 1109; HRMS (ESI): Exact mass calcd for $C_{25}H_{30}N_4O_4Si^{35}Cl$ $[M+NH_4]^+$: 513.1719, Found: 513.1719.



4k

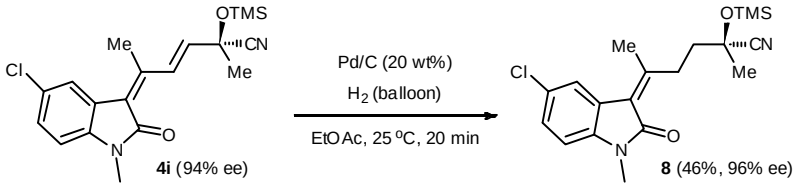
The total reaction time was 2 days. Product **4k** was obtained in 79% yield as yellow oil. $[\alpha]^{25}_D$ = +1.9 (c = 0.38, CH_2Cl_2); HPLC analysis (Chiralpak IE, ^{i}PrOH/hexane = 10/90, 1.0 mL/min, 230 nm; t_r (major) = 12.70 min, t_r (minor) = 11.94 min) gave the isomeric composition of the product: 94% ee; ^{1}H NMR (400 MHz, acetone-d_6): δ 9.01 (d, *J* = 15.6 Hz, 1H), 7.84 (d, *J* = 1.6 Hz, 1H), 7.51 (ABd, *J* = 8.4 Hz, 2H), 7.39 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.31 (ABd, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 1H), 6.65 (d, *J* = 16.0 Hz, 1H), 5.00 (s, 2H), 2.56 (s, 3H), 1.80 (s, 3H), 0.29 (s, 9H); ^{13}C NMR (100 MHz, acetone-d_6): δ 167.21, 148.98, 141.96, 139.87, 136.86, 132.55, 132.09, 130.26, 129.80, 128.35, 126.18, 124.32, 121.70, 121.26, 114.99, 111.16, 70.73, 42.93, 30.79, 17.55, 1.46; IR (ATR): 3466, 2958, 1692, 1604, 1475, 1342, 1254, 1186; HRMS (ESI): Exact mass calcd for $C_{25}H_{30}N_3O_2Si^{79}Br_2$ $[M+NH_4]^+$: 590.0469, Found: 590.0467.



7

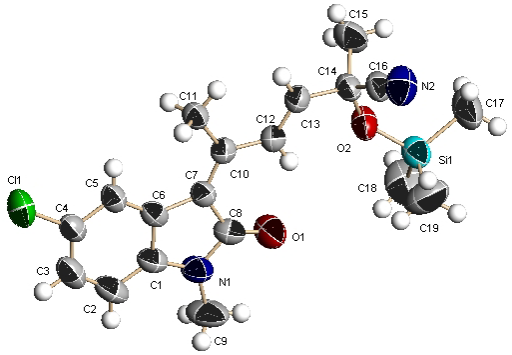
The total reaction time was 4 days. Product **7** was obtained in 75% yield as yellow oil. $[\alpha]^{25}_{D}$ = -4.1 (c = 1.10, CH_2Cl_2); HPLC analysis (Chiralpak IE, ^{i}PrOH/hexane = 3/97, 1.0 mL/min, 230 nm; t_r (minor) = 7.13 min, t_r (major) = 7.45 min) gave the isomeric composition of the product: 46% ee; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.26-7.16 (m, 3H), 6.92-6.89 (m, 2H), 5.92-5.86 (m, 1H), 3.82 (s, 3H), 3.77 (s, 3H), 1.85 (s, 3H), 1.72 (s, 3H), 0.26 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.01, 159.09, 136.38, 135.23, 132.88, 130.32, 130.19, 128.86, 120.52, 113.68, 69.61, 55.19, 52.13, 30.64, 16.17, 1.27; IR (ATR): 2956, 2841, 1723, 1609, 1151, 1463, 1424, 1304, 1253, 1106; HRMS (ESI): Exact mass calcd for $C_{20}H_{27}NO_4SiNa$ $[M+Na]^+$: 396.1607, Found: 396.1607.

5) The synthesis of product 8



To a stirred solution of **4i** (40 mg, 94% ee) in EtOAc (2.0 mL) was added Pd/C catalyst (10% Pd, 8.0 mg), and back-filled with hydrogen by a H_2 balloon. The resulting mixture was stirred at 25 ^{o}C till almost full consumption of **4i** by TLC analysis (about 20 min). Then the reaction mixture was directly subjected to column chromatography using petroleum ether/CH_2Cl_2 (from 1:2 to 0:1) as the eluent, affording 18.3 mg product **8** as yellow oil. $[\alpha]^{25}_{D}$ = -12.1 (c = 0.92, CH_2Cl_2); HPLC analysis (Chiralpak OD, ^{i}PrOH/hexane = 10/90, 1.0 mL/min, 230 nm; t_r (major) = 5.61 min, t_r (minor) = 6.27 min) gave the isomeric composition of the product: 96% ee; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 (d, J = 2.0 Hz, 1H), 7.23 (dd, J_1 = 8.4 Hz, J_2 = 2.0 Hz, 1H), 6.73 (d, J = 8.4 Hz, 1H), 3.36-3.29 (m, 1H), 3.22 (s, 3H), 3.20-3.15 (m, 1H), 2.38 (s, 3H), 2.00-1.95 (m, 2H), 1.69 (s, 3H), 0.25 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.76, 157.84, 140.77, 127.51, 126.97, 124.57, 123.77, 122.79, 121.92, 108.33, 69.45, 40.79, 30.94, 28.26, 25.78, 23.54, 1.30; IR (ATR): 2954, 1693, 1806, 1484, 1368, 1335, 1252, 1181, 1104, 1039; HRMS (ESI): Exact mass calcd for $C_{19}H_{25}N_2O_2SiNa^{35}Cl$ $[M+Na]^+$: 399.1272 , Found: 399.1256.

6) Single-Crystal X-ray Crystallography of 4i[5]



Data intensity of **4i** was collected using a Bruker SMART APEX II (Mo radiation). The X-ray condition of was 50 kV × 30 mA. Data collection and reduction were done by using the Bruker ApexII software package. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for non-H atoms using SHELX-97. Hydrogen atoms were added at their geometrically idea positions and refined isotropically. Crystal data for **4i**: $C_{19}H_{23}ClN_2O_2Si$, M = 374.93, T = 293(2) K, λ = 0.71073 Å, Orthorhombic, space group P 21 21 2, a = 16.4159(14) Å, b = 36.026(3) Å, c = 7.0919(6) Å, V = 4194.2(6) $Å^3$, z = 8, d_{calc} = 1.188 Mg/m^3, 24607 reflections measured, 7821 [R(int) = 0.0655], R_1 = 0.1189, wR_2 = 0.1407 ($I > 2\sigma(I)$, final R_1 = 0.0549, wR_2 = 0.1240, GOF = 0.900, and 463 parameters.

Table 1. Crystal data and structure refinement for cd214559.

Identification code	cd214559	
Empirical formula	$C_{19}H_{23}ClN_2O_2Si$	
Formula weight	374.93	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 2	
Unit cell dimensions	a = 16.4159(14) Å	a= 90°.
	b = 36.026(3) Å	b= 90°.
	c = 7.0919(6) Å	g = 90°.
Volume	4194.2(6) Å3	
Z	8	

[5] Supplementary crystallographic data have been deposited at the Cambridge Crystallographic Data Center. (CCDC1024592).

Density (calculated)	1.188 Mg/m^3
Absorption coefficient	0.253 mm^{-1}
F(000)	1584
Crystal size	0.211 x 0.154 x 0.112 mm^3
Theta range for data collection	1.678 to 25.500°.
Index ranges	-19<=h<=19, -43<=k<=40, -8<=l<=8
Reflections collected	24607
Independent reflections	7821 [R(int) = 0.0655]
Completeness to theta = 25.242°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7457 and 0.6323
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7821 / 0 / 463
Goodness-of-fit on F^2	0.900
Final R indices [I>2sigma(I)]	R_1 = 0.0549, wR_2 = 0.1240
R indices (all data)	R_1 = 0.1189, wR_2 = 0.1407
Absolute structure parameter	0.09(6)
Extinction coefficient	n/a
Largest diff. peak and hole	0.268 and -0.178 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for cd214559. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Si(1)	8937(1)	8638(1)	9194(3)	78(1)
Si(2)	7844(1)	82(1)	4547(3)	93(1)
Cl(1)	2139(1)	8130(1)	10534(3)	103(1)
Cl(2)	4390(1)	2775(1)	5566(3)	122(1)
N(1)	4950(3)	9122(1)	9892(7)	75(1)
N(2)	8564(3)	7681(2)	7086(9)	98(2)
N(3)	4506(3)	1159(2)	5003(7)	90(2)

N(4)	8997(5)	631(2)	8345(11)	142(3)
O(1)	6303(2)	9005(1)	9884(7)	100(2)
O(2)	8258(2)	8374(1)	10245(6)	80(1)
O(3)	5590(3)	758(1)	4914(8)	112(2)
O(4)	8010(2)	526(1)	4142(6)	87(1)
C(1)	4219(3)	8922(1)	10020(8)	65(1)
C(2)	3421(4)	9055(2)	10008(8)	81(2)
C(3)	2789(3)	8805(2)	10170(8)	81(2)
C(4)	2955(3)	8435(2)	10357(8)	66(1)
C(5)	3741(3)	8296(1)	10375(7)	61(1)
C(6)	4390(3)	8543(1)	10218(6)	54(1)
C(7)	5277(2)	8503(1)	10169(7)	56(1)
C(8)	5606(3)	8893(1)	9981(8)	68(2)
C(9)	4993(4)	9522(1)	9741(11)	114(2)
C(10)	5748(3)	8191(1)	10265(6)	51(1)
C(11)	5371(3)	7815(1)	10420(8)	59(1)
C(12)	6632(3)	8222(1)	10193(7)	59(1)
C(13)	7158(3)	7949(1)	10244(7)	59(1)
C(14)	8065(3)	7997(1)	10201(8)	62(1)
C(15)	8460(4)	7794(2)	11870(9)	98(2)
C(16)	8348(3)	7818(2)	8421(10)	69(2)
C(17)	8861(5)	9075(2)	10508(14)	144(3)
C(18)	8659(5)	8706(2)	6709(11)	140(3)
C(19)	9956(3)	8427(2)	9345(12)	120(3)
C(20)	4372(3)	1538(2)	5151(8)	80(2)
C(21)	3645(3)	1730(2)	5214(9)	94(2)
C(22)	3664(4)	2106(3)	5343(9)	103(2)
C(23)	4391(4)	2293(2)	5384(8)	82(2)
C(24)	5131(3)	2103(2)	5368(7)	70(1)
C(25)	5137(3)	1719(2)	5252(7)	66(1)
C(26)	5779(3)	1435(1)	5164(7)	63(1)
C(27)	5322(4)	1070(2)	5009(9)	85(2)
C(28)	3865(4)	876(2)	4883(12)	138(3)
C(29)	6597(3)	1470(1)	5199(7)	60(1)
C(30)	6980(3)	1847(1)	5422(9)	73(2)
C(31)	7126(3)	1148(1)	5013(7)	66(1)

C(32)	7922(3)	1146(1)	5287(9)	74(2)
C(33)	8451(3)	809(2)	4982(9)	72(2)
C(34)	9180(4)	902(2)	3756(11)	113(2)
C(35)	8764(4)	701(2)	6884(12)	92(2)
C(36)	8796(5)	-177(2)	4759(15)	174(4)
C(37)	7223(6)	38(3)	6715(11)	166(4)
C(38)	7255(5)	-62(2)	2503(11)	132(3)

Table 3. Bond lengths [Å] and angles [°] for cd214559.

Si(1)-O(2)	1.646(4)
Si(1)-C(17)	1.832(6)
Si(1)-C(18)	1.837(8)
Si(1)-C(19)	1.840(6)
Si(2)-O(4)	1.647(4)
Si(2)-C(38)	1.817(7)
Si(2)-C(36)	1.826(7)
Si(2)-C(37)	1.851(8)
Cl(1)-C(4)	1.737(5)
Cl(2)-C(23)	1.742(6)
N(1)-C(8)	1.358(6)
N(1)-C(1)	1.402(6)
N(1)-C(9)	1.450(6)
N(2)-C(16)	1.124(7)
N(3)-C(27)	1.378(7)
N(3)-C(20)	1.386(7)
N(3)-C(28)	1.467(7)
N(4)-C(35)	1.133(9)
O(1)-C(8)	1.216(6)
O(2)-C(14)	1.395(5)
O(3)-C(27)	1.210(7)
O(4)-C(33)	1.385(6)
C(1)-C(2)	1.393(7)
C(1)-C(6)	1.401(6)
C(2)-C(3)	1.378(7)
C(2)-H(2)	0.9300

C(3)-C(4)	1.367(7)
C(3)-H(3)	0.9300
C(4)-C(5)	1.385(6)
C(5)-C(6)	1.392(6)
C(5)-H(5)	0.9300
C(6)-C(7)	1.465(6)
C(7)-C(10)	1.367(6)
C(7)-C(8)	1.511(7)
C(9)-H(9A)	0.9600
C(9)-H(9B)	0.9600
C(9)-H(9C)	0.9600
C(10)-C(12)	1.456(6)
C(10)-C(11)	1.493(6)
C(11)-H(11A)	0.9600
C(11)-H(11B)	0.9600
C(11)-H(11C)	0.9600
C(12)-C(13)	1.309(6)
C(12)-H(12)	0.9300
C(13)-C(14)	1.499(6)
C(13)-H(13)	0.9300
C(14)-C(16)	1.492(8)
C(14)-C(15)	1.534(7)
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(17)-H(17A)	0.9600
C(17)-H(17B)	0.9600
C(17)-H(17C)	0.9600
C(18)-H(18A)	0.9600
C(18)-H(18B)	0.9600
C(18)-H(18C)	0.9600
C(19)-H(19A)	0.9600
C(19)-H(19B)	0.9600
C(19)-H(19C)	0.9600
C(20)-C(21)	1.381(8)
C(20)-C(25)	1.417(7)

C(21)-C(22)	1.357(8)
C(21)-H(21)	0.9300
C(22)-C(23)	1.371(8)
C(22)-H(22)	0.9300
C(23)-C(24)	1.394(7)
C(24)-C(25)	1.387(7)
C(24)-H(24)	0.9300
C(25)-C(26)	1.471(7)
C(26)-C(29)	1.351(6)
C(26)-C(27)	1.516(8)
C(28)-H(28A)	0.9600
C(28)-H(28B)	0.9600
C(28)-H(28C)	0.9600
C(29)-C(31)	1.456(6)
C(29)-C(30)	1.503(6)
C(30)-H(30A)	0.9600
C(30)-H(30B)	0.9600
C(30)-H(30C)	0.9600
C(31)-C(32)	1.321(7)
C(31)-H(31)	0.9300
C(32)-C(33)	1.508(7)
C(32)-H(32)	0.9300
C(33)-C(35)	1.495(10)
C(33)-C(34)	1.518(8)
C(34)-H(34A)	0.9600
C(34)-H(34B)	0.9600
C(34)-H(34C)	0.9600
C(36)-H(36A)	0.9600
C(36)-H(36B)	0.9600
C(36)-H(36C)	0.9600
C(37)-H(37A)	0.9600
C(37)-H(37B)	0.9600
C(37)-H(37C)	0.9600
C(38)-H(38A)	0.9600
C(38)-H(38B)	0.9600
C(38)-H(38C)	0.9600

O(2)-Si(1)-C(17)	102.7(3)
O(2)-Si(1)-C(18)	110.0(3)
C(17)-Si(1)-C(18)	110.9(4)
O(2)-Si(1)-C(19)	110.5(3)
C(17)-Si(1)-C(19)	112.8(4)
C(18)-Si(1)-C(19)	109.7(4)
O(4)-Si(2)-C(38)	103.0(3)
O(4)-Si(2)-C(36)	111.5(3)
C(38)-Si(2)-C(36)	112.1(4)
O(4)-Si(2)-C(37)	108.6(4)
C(38)-Si(2)-C(37)	110.2(4)
C(36)-Si(2)-C(37)	111.1(5)
C(8)-N(1)-C(1)	111.4(4)
C(8)-N(1)-C(9)	124.7(4)
C(1)-N(1)-C(9)	123.8(4)
C(27)-N(3)-C(20)	112.5(5)
C(27)-N(3)-C(28)	122.4(6)
C(20)-N(3)-C(28)	125.1(6)
C(14)-O(2)-Si(1)	135.0(3)
C(33)-O(4)-Si(2)	136.6(4)
C(2)-C(1)-C(6)	121.5(5)
C(2)-C(1)-N(1)	128.9(5)
C(6)-C(1)-N(1)	109.5(4)
C(3)-C(2)-C(1)	118.9(5)
C(3)-C(2)-H(2)	120.6
C(1)-C(2)-H(2)	120.6
C(4)-C(3)-C(2)	119.6(5)
C(4)-C(3)-H(3)	120.2
C(2)-C(3)-H(3)	120.2
C(3)-C(4)-C(5)	122.6(5)
C(3)-C(4)-Cl(1)	118.1(4)
C(5)-C(4)-Cl(1)	119.3(4)
C(4)-C(5)-C(6)	118.7(4)
C(4)-C(5)-H(5)	120.6
C(6)-C(5)-H(5)	120.6

C(5)-C(6)-C(1)	118.6(4)
C(5)-C(6)-C(7)	134.4(4)
C(1)-C(6)-C(7)	107.1(4)
C(10)-C(7)-C(6)	130.0(4)
C(10)-C(7)-C(8)	124.6(4)
C(6)-C(7)-C(8)	105.4(4)
O(1)-C(8)-N(1)	122.8(5)
O(1)-C(8)-C(7)	130.6(4)
N(1)-C(8)-C(7)	106.6(4)
N(1)-C(9)-H(9A)	109.5
N(1)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
N(1)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(7)-C(10)-C(12)	119.8(4)
C(7)-C(10)-C(11)	121.1(4)
C(12)-C(10)-C(11)	119.1(4)
C(10)-C(11)-H(11A)	109.5
C(10)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(10)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(13)-C(12)-C(10)	126.6(4)
C(13)-C(12)-H(12)	116.7
C(10)-C(12)-H(12)	116.7
C(12)-C(13)-C(14)	124.6(4)
C(12)-C(13)-H(13)	117.7
C(14)-C(13)-H(13)	117.7
O(2)-C(14)-C(16)	111.7(4)
O(2)-C(14)-C(13)	109.6(4)
C(16)-C(14)-C(13)	106.1(4)
O(2)-C(14)-C(15)	110.4(5)
C(16)-C(14)-C(15)	108.4(4)
C(13)-C(14)-C(15)	110.5(5)

C(14)-C(15)-H(15A)	109.5
C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
N(2)-C(16)-C(14)	179.5(7)
Si(1)-C(17)-H(17A)	109.5
Si(1)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
Si(1)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
Si(1)-C(18)-H(18A)	109.5
Si(1)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
Si(1)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
Si(1)-C(19)-H(19A)	109.5
Si(1)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
Si(1)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(21)-C(20)-N(3)	129.4(6)
C(21)-C(20)-C(25)	122.2(6)
N(3)-C(20)-C(25)	108.5(5)
C(22)-C(21)-C(20)	118.9(6)
C(22)-C(21)-H(21)	120.6
C(20)-C(21)-H(21)	120.6
C(21)-C(22)-C(23)	120.8(6)
C(21)-C(22)-H(22)	119.6
C(23)-C(22)-H(22)	119.6
C(22)-C(23)-C(24)	121.1(6)
C(22)-C(23)-Cl(2)	119.4(5)

C(24)-C(23)-Cl(2)	119.5(5)
C(25)-C(24)-C(23)	119.7(5)
C(25)-C(24)-H(24)	120.1
C(23)-C(24)-H(24)	120.1
C(24)-C(25)-C(20)	117.2(5)
C(24)-C(25)-C(26)	134.6(4)
C(20)-C(25)-C(26)	108.2(5)
C(29)-C(26)-C(25)	130.3(5)
C(29)-C(26)-C(27)	125.1(5)
C(25)-C(26)-C(27)	104.6(5)
O(3)-C(27)-N(3)	124.7(6)
O(3)-C(27)-C(26)	129.1(6)
N(3)-C(27)-C(26)	106.2(6)
N(3)-C(28)-H(28A)	109.5
N(3)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
N(3)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(26)-C(29)-C(31)	121.0(4)
C(26)-C(29)-C(30)	120.2(4)
C(31)-C(29)-C(30)	118.7(4)
C(29)-C(30)-H(30A)	109.5
C(29)-C(30)-H(30B)	109.5
H(30A)-C(30)-H(30B)	109.5
C(29)-C(30)-H(30C)	109.5
H(30A)-C(30)-H(30C)	109.5
H(30B)-C(30)-H(30C)	109.5
C(32)-C(31)-C(29)	125.4(5)
C(32)-C(31)-H(31)	117.3
C(29)-C(31)-H(31)	117.3
C(31)-C(32)-C(33)	123.5(5)
C(31)-C(32)-H(32)	118.2
C(33)-C(32)-H(32)	118.2
O(4)-C(33)-C(35)	112.1(5)
O(4)-C(33)-C(32)	110.8(4)

C(35)-C(33)-C(32)	106.1(5)
O(4)-C(33)-C(34)	109.1(5)
C(35)-C(33)-C(34)	107.6(5)
C(32)-C(33)-C(34)	111.0(5)
C(33)-C(34)-H(34A)	109.5
C(33)-C(34)-H(34B)	109.5
H(34A)-C(34)-H(34B)	109.5
C(33)-C(34)-H(34C)	109.5
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
N(4)-C(35)-C(33)	177.8(8)
Si(2)-C(36)-H(36A)	109.5
Si(2)-C(36)-H(36B)	109.5
H(36A)-C(36)-H(36B)	109.5
Si(2)-C(36)-H(36C)	109.5
H(36A)-C(36)-H(36C)	109.5
H(36B)-C(36)-H(36C)	109.5
Si(2)-C(37)-H(37A)	109.5
Si(2)-C(37)-H(37B)	109.5
H(37A)-C(37)-H(37B)	109.5
Si(2)-C(37)-H(37C)	109.5
H(37A)-C(37)-H(37C)	109.5
H(37B)-C(37)-H(37C)	109.5
Si(2)-C(38)-H(38A)	109.5
Si(2)-C(38)-H(38B)	109.5
H(38A)-C(38)-H(38B)	109.5
Si(2)-C(38)-H(38C)	109.5
H(38A)-C(38)-H(38C)	109.5
H(38B)-C(38)-H(38C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for cd214559. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}

Si(1)	61(1)	66(1)	108(1)	-8(1)	-6(1)	-7(1)
Si(2)	115(1)	70(1)	95(1)	-8(1)	-3(1)	4(1)
Cl(1)	56(1)	127(1)	126(1)	2(1)	1(1)	-2(1)
Cl(2)	101(1)	132(2)	132(2)	-11(1)	-3(1)	50(1)
N(1)	76(3)	53(3)	95(4)	3(3)	-2(3)	9(2)
N(2)	95(4)	93(4)	107(5)	-30(3)	22(4)	-4(3)
N(3)	72(3)	117(4)	79(4)	15(3)	0(3)	-35(3)
N(4)	167(7)	129(5)	130(6)	-15(5)	-60(5)	18(4)
O(1)	70(2)	63(2)	167(5)	1(3)	-7(3)	-13(2)
O(2)	60(2)	69(2)	111(3)	-17(2)	14(2)	-5(2)
O(3)	111(3)	84(3)	141(5)	0(3)	-3(3)	-36(3)
O(4)	101(3)	61(2)	98(3)	-12(2)	-14(3)	-1(2)
C(1)	73(3)	61(3)	62(4)	2(3)	-1(3)	18(3)
C(2)	91(4)	78(4)	75(5)	-3(3)	-7(4)	35(3)
C(3)	59(3)	110(5)	73(4)	0(4)	-6(3)	21(3)
C(4)	59(3)	85(4)	55(3)	3(3)	0(3)	16(3)
C(5)	51(3)	78(3)	55(3)	-2(3)	-3(3)	8(3)
C(6)	56(3)	68(3)	39(3)	-2(3)	-6(3)	12(2)
C(7)	51(3)	60(3)	56(3)	-3(3)	-4(3)	3(2)
C(8)	74(4)	58(3)	72(4)	2(3)	-6(4)	3(3)
C(9)	115(4)	53(4)	173(7)	17(4)	-20(6)	7(3)
C(10)	57(3)	62(3)	35(3)	-3(2)	-1(2)	0(2)
C(11)	52(3)	57(3)	68(3)	-2(3)	-1(3)	0(2)
C(12)	55(3)	62(3)	59(4)	1(3)	5(3)	0(2)
C(13)	53(3)	62(3)	62(4)	2(3)	6(3)	-3(2)
C(14)	51(3)	57(3)	80(4)	-1(3)	0(3)	2(2)
C(15)	79(5)	129(6)	85(5)	20(4)	-16(4)	21(4)
C(16)	59(3)	61(4)	85(5)	-5(3)	11(3)	5(3)
C(17)	137(6)	82(5)	211(10)	-52(6)	-1(7)	-13(4)
C(18)	165(8)	127(6)	129(7)	47(5)	-21(6)	-31(5)
C(19)	57(3)	116(5)	188(8)	-6(6)	0(5)	-8(3)
C(20)	74(4)	120(5)	45(4)	9(4)	-6(3)	-19(4)
C(21)	48(3)	156(6)	79(5)	6(5)	-3(3)	-10(4)
C(22)	73(4)	169(7)	67(4)	12(5)	-2(4)	20(5)
C(23)	73(4)	121(5)	51(4)	4(3)	0(4)	8(4)

C(24)	58(3)	104(5)	50(3)	-5(3)	-6(3)	2(3)
C(25)	55(3)	104(5)	39(3)	-3(3)	5(3)	-15(3)
C(26)	62(3)	78(3)	48(3)	6(3)	0(3)	-13(3)
C(27)	92(5)	97(5)	65(4)	9(4)	6(4)	-25(4)
C(28)	106(5)	152(6)	154(8)	11(6)	-12(6)	-73(5)
C(29)	63(3)	70(3)	48(3)	1(3)	4(3)	-7(3)
C(30)	59(3)	67(3)	93(4)	0(3)	-6(3)	0(2)
C(31)	72(4)	59(3)	67(4)	-3(3)	6(3)	-7(3)
C(32)	76(4)	60(3)	87(4)	-1(3)	-1(4)	-7(3)
C(33)	75(4)	76(4)	66(4)	-1(3)	-5(4)	3(3)
C(34)	86(5)	122(6)	130(7)	6(5)	13(4)	18(4)
C(35)	93(5)	75(4)	109(6)	-11(4)	-9(5)	7(4)
C(36)	175(7)	107(6)	239(12)	-34(7)	-45(9)	54(5)
C(37)	206(10)	188(9)	102(6)	1(6)	50(6)	-57(8)
C(38)	185(8)	95(5)	115(6)	-21(5)	-21(6)	-30(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for cd214559.

	x	y	z	U(eq)
H(2)	3318	9308	9893	98
H(3)	2252	8888	10151	97
H(5)	3835	8043	10489	73
H(9A)	4774	9633	10863	170
H(9B)	4683	9603	8668	170
H(9C)	5551	9597	9590	170
H(11A)	5097	7793	11611	88
H(11B)	5788	7629	10333	88
H(11C)	4986	7781	9416	88
H(12)	6845	8461	10102	71
H(13)	6954	7709	10313	71
H(15A)	9042	7803	11742	147
H(15B)	8283	7541	11887	147
H(15C)	8304	7913	13026	147
H(17A)	8332	9181	10315	215

H(17B)	9271	9244	10071	215
H(17C)	8940	9027	11828	215
H(18A)	8656	8470	6075	211
H(18B)	9047	8867	6118	211
H(18C)	8126	8815	6635	211
H(19A)	10099	8392	10645	180
H(19B)	10347	8588	8756	180
H(19C)	9952	8191	8713	180
H(21)	3151	1605	5168	113
H(22)	3178	2238	5405	123
H(24)	5619	2234	5434	84
H(28A)	4110	636	4746	206
H(28B)	3524	926	3813	206
H(28C)	3542	882	6011	206
H(30A)	6843	1999	4356	110
H(30B)	7561	1821	5500	110
H(30C)	6781	1961	6554	110
H(31)	6883	925	4672	79
H(32)	8170	1364	5689	89
H(34A)	9510	684	3594	169
H(34B)	9497	1092	4351	169
H(34C)	8996	987	2547	169
H(36A)	9149	-113	3729	260
H(36B)	8684	-438	4731	260
H(36C)	9057	-114	5929	260
H(37A)	7365	233	7579	248
H(37B)	7325	-198	7292	248
H(37C)	6656	57	6398	248
H(38A)	6704	24	2632	198
H(38B)	7259	-328	2414	198
H(38C)	7492	43	1384	198

Table 6. Torsion angles [°] for cd214559.

C(17)-Si(1)-O(2)-C(14)	-168.5(5)
C(18)-Si(1)-O(2)-C(14)	73.3(6)

C(19)-Si(1)-O(2)-C(14)	-48.0(6)
C(38)-Si(2)-O(4)-C(33)	-174.6(6)
C(36)-Si(2)-O(4)-C(33)	-54.3(7)
C(37)-Si(2)-O(4)-C(33)	68.5(6)
C(8)-N(1)-C(1)-C(2)	179.1(6)
C(9)-N(1)-C(1)-C(2)	0.8(9)
C(8)-N(1)-C(1)-C(6)	0.5(6)
C(9)-N(1)-C(1)-C(6)	-177.8(5)
C(6)-C(1)-C(2)-C(3)	-0.9(9)
N(1)-C(1)-C(2)-C(3)	-179.4(5)
C(1)-C(2)-C(3)-C(4)	0.7(9)
C(2)-C(3)-C(4)-C(5)	-0.7(9)
C(2)-C(3)-C(4)-Cl(1)	-179.4(5)
C(3)-C(4)-C(5)-C(6)	0.8(8)
Cl(1)-C(4)-C(5)-C(6)	179.5(4)
C(4)-C(5)-C(6)-C(1)	-1.0(7)
C(4)-C(5)-C(6)-C(7)	-179.5(5)
C(2)-C(1)-C(6)-C(5)	1.1(8)
N(1)-C(1)-C(6)-C(5)	179.8(5)
C(2)-C(1)-C(6)-C(7)	179.9(5)
N(1)-C(1)-C(6)-C(7)	-1.3(6)
C(5)-C(6)-C(7)-C(10)	0.2(10)
C(1)-C(6)-C(7)-C(10)	-178.4(5)
C(5)-C(6)-C(7)-C(8)	-179.8(5)
C(1)-C(6)-C(7)-C(8)	1.6(5)
C(1)-N(1)-C(8)-O(1)	179.4(6)
C(9)-N(1)-C(8)-O(1)	-2.3(9)
C(1)-N(1)-C(8)-C(7)	0.6(6)
C(9)-N(1)-C(8)-C(7)	178.8(5)
C(10)-C(7)-C(8)-O(1)	0.0(10)
C(6)-C(7)-C(8)-O(1)	179.9(6)
C(10)-C(7)-C(8)-N(1)	178.7(5)
C(6)-C(7)-C(8)-N(1)	-1.3(6)
C(6)-C(7)-C(10)-C(12)	-179.8(5)
C(8)-C(7)-C(10)-C(12)	0.2(8)
C(6)-C(7)-C(10)-C(11)	1.0(8)

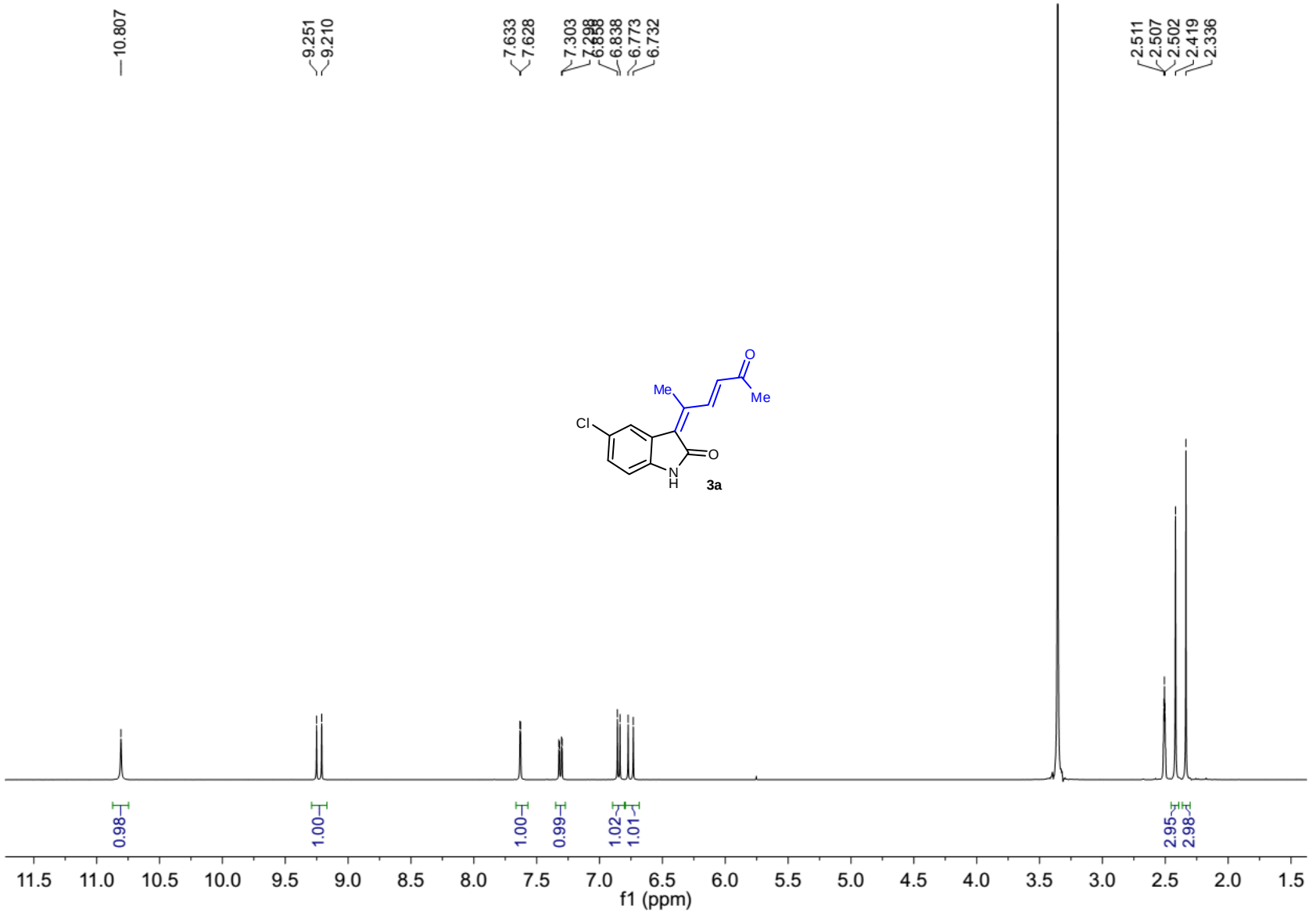
C(8)-C(7)-C(10)-C(11)	-179.0(5)
C(7)-C(10)-C(12)-C(13)	-179.0(5)
C(11)-C(10)-C(12)-C(13)	0.3(8)
C(10)-C(12)-C(13)-C(14)	-178.9(5)
Si(1)-O(2)-C(14)-C(16)	-22.8(7)
Si(1)-O(2)-C(14)-C(13)	-140.2(4)
Si(1)-O(2)-C(14)-C(15)	97.9(6)
C(12)-C(13)-C(14)-O(2)	4.5(8)
C(12)-C(13)-C(14)-C(16)	-116.2(6)
C(12)-C(13)-C(14)-C(15)	126.5(6)
C(27)-N(3)-C(20)-C(21)	-179.2(6)
C(28)-N(3)-C(20)-C(21)	-0.1(10)
C(27)-N(3)-C(20)-C(25)	0.4(7)
C(28)-N(3)-C(20)-C(25)	179.5(6)
N(3)-C(20)-C(21)-C(22)	-179.3(6)
C(25)-C(20)-C(21)-C(22)	1.2(9)
C(20)-C(21)-C(22)-C(23)	0.9(10)
C(21)-C(22)-C(23)-C(24)	-2.5(10)
C(21)-C(22)-C(23)-Cl(2)	-179.6(5)
C(22)-C(23)-C(24)-C(25)	1.9(8)
Cl(2)-C(23)-C(24)-C(25)	179.0(4)
C(23)-C(24)-C(25)-C(20)	0.1(8)
C(23)-C(24)-C(25)-C(26)	178.5(5)
C(21)-C(20)-C(25)-C(24)	-1.6(8)
N(3)-C(20)-C(25)-C(24)	178.7(5)
C(21)-C(20)-C(25)-C(26)	179.5(5)
N(3)-C(20)-C(25)-C(26)	-0.1(6)
C(24)-C(25)-C(26)-C(29)	1.1(10)
C(20)-C(25)-C(26)-C(29)	179.6(5)
C(24)-C(25)-C(26)-C(27)	-178.7(6)
C(20)-C(25)-C(26)-C(27)	-0.2(6)
C(20)-N(3)-C(27)-O(3)	179.2(6)
C(28)-N(3)-C(27)-O(3)	0.1(11)
C(20)-N(3)-C(27)-C(26)	-0.5(7)
C(28)-N(3)-C(27)-C(26)	-179.6(5)
C(29)-C(26)-C(27)-O(3)	0.9(11)

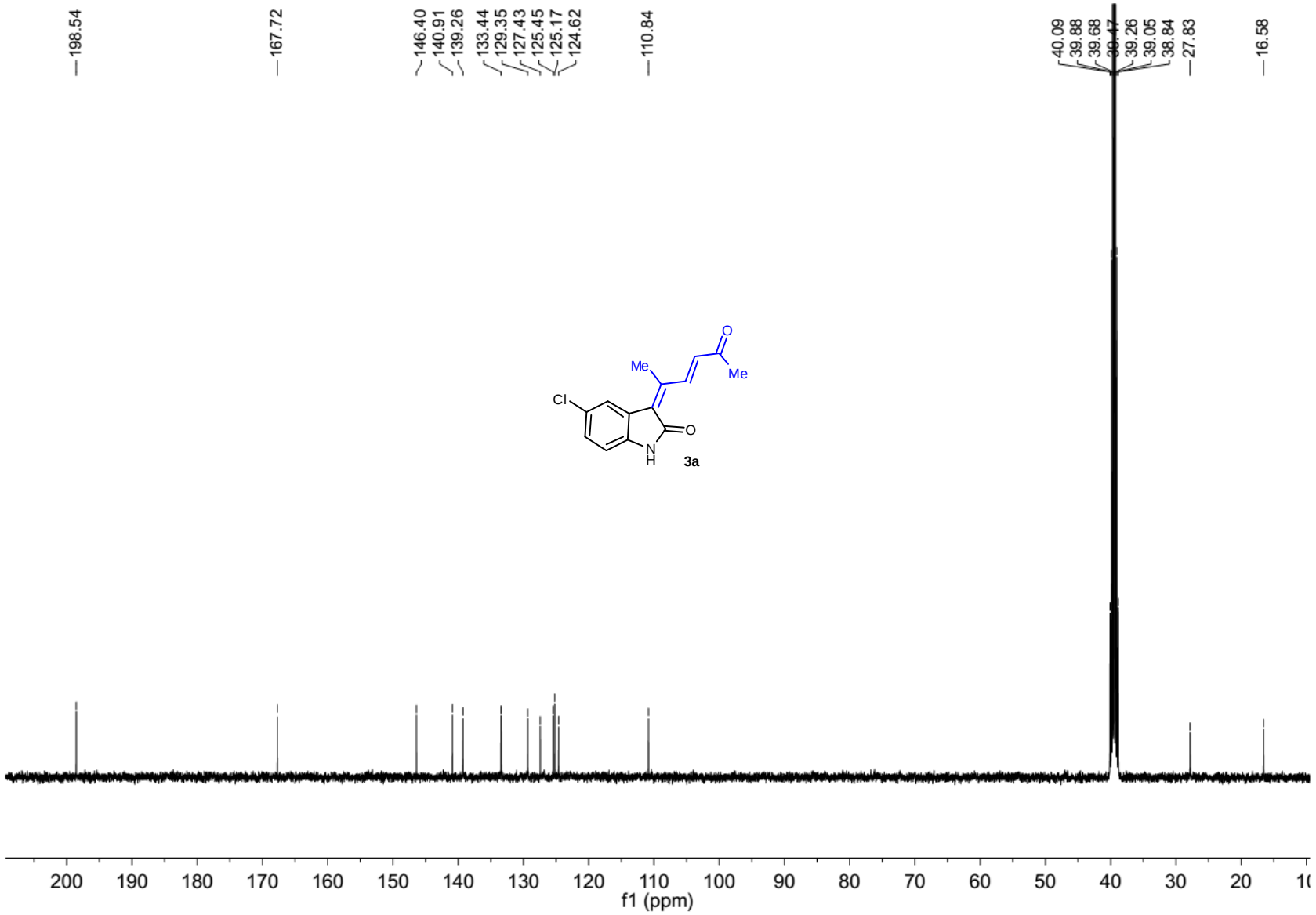
C(25)-C(26)-C(27)-O(3)	-179.3(7)
C(29)-C(26)-C(27)-N(3)	-179.4(5)
C(25)-C(26)-C(27)-N(3)	0.4(6)
C(25)-C(26)-C(29)-C(31)	-177.4(5)
C(27)-C(26)-C(29)-C(31)	2.4(9)
C(25)-C(26)-C(29)-C(30)	2.3(9)
C(27)-C(26)-C(29)-C(30)	-178.0(5)
C(26)-C(29)-C(31)-C(32)	-170.1(6)
C(30)-C(29)-C(31)-C(32)	10.2(8)
C(29)-C(31)-C(32)-C(33)	-176.8(5)
Si(2)-O(4)-C(33)-C(35)	-10.5(8)
Si(2)-O(4)-C(33)-C(32)	-128.9(5)
Si(2)-O(4)-C(33)-C(34)	108.6(6)
C(31)-C(32)-C(33)-O(4)	8.3(8)
C(31)-C(32)-C(33)-C(35)	-113.6(7)
C(31)-C(32)-C(33)-C(34)	129.7(7)

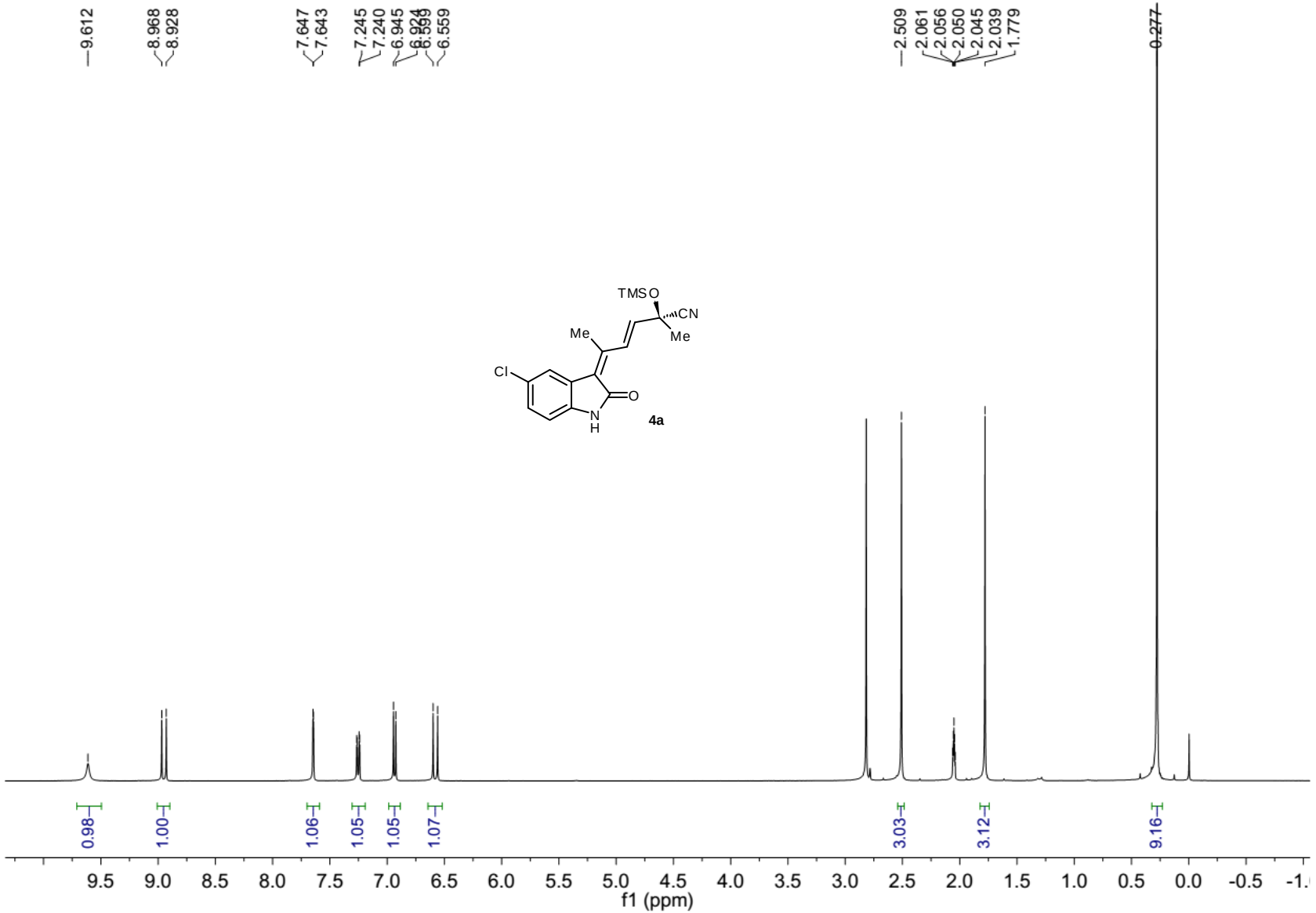
Symmetry transformations used to generate equivalent atoms:

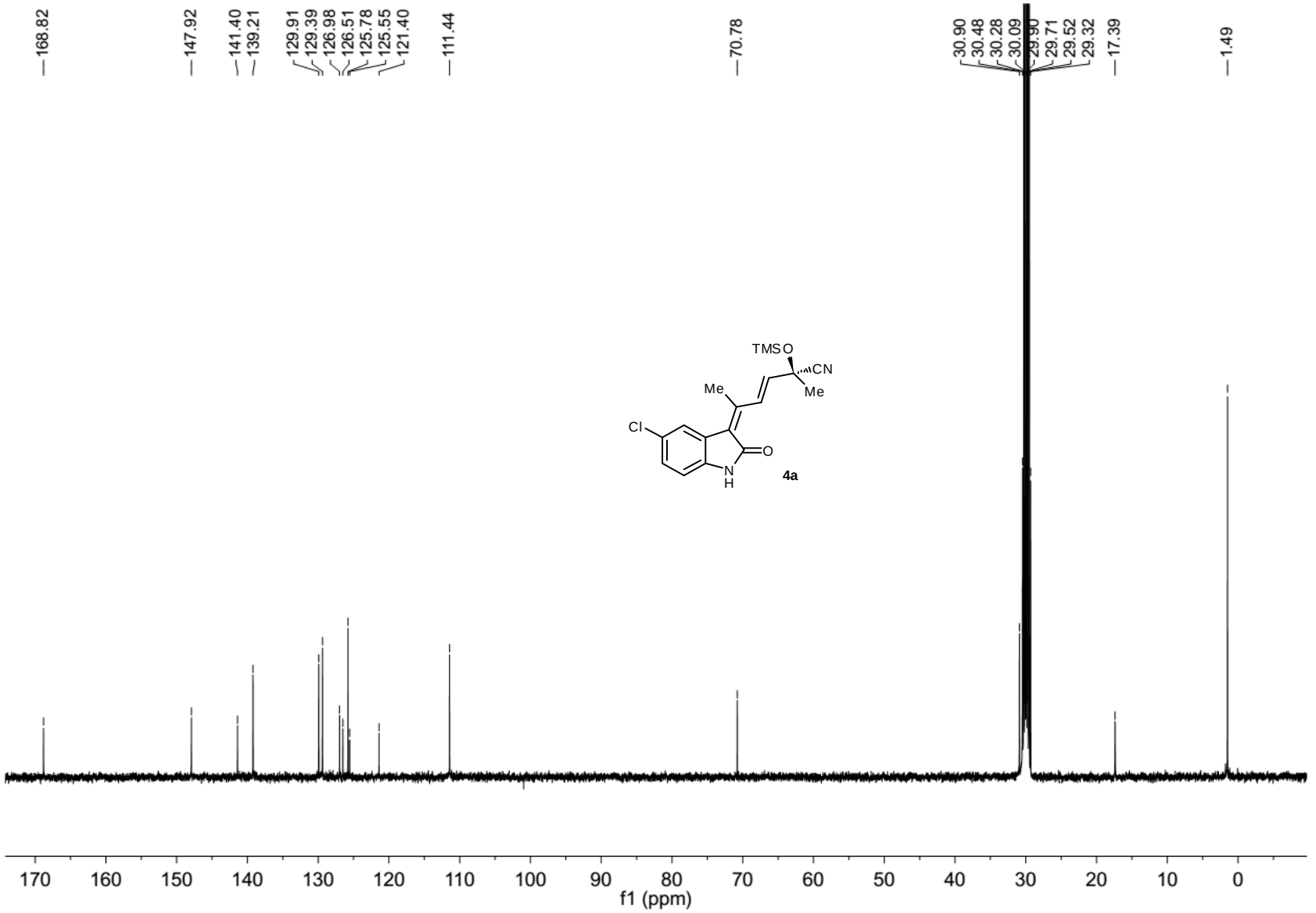
Table 7. Hydrogen bonds for cd214559 [Å and °].

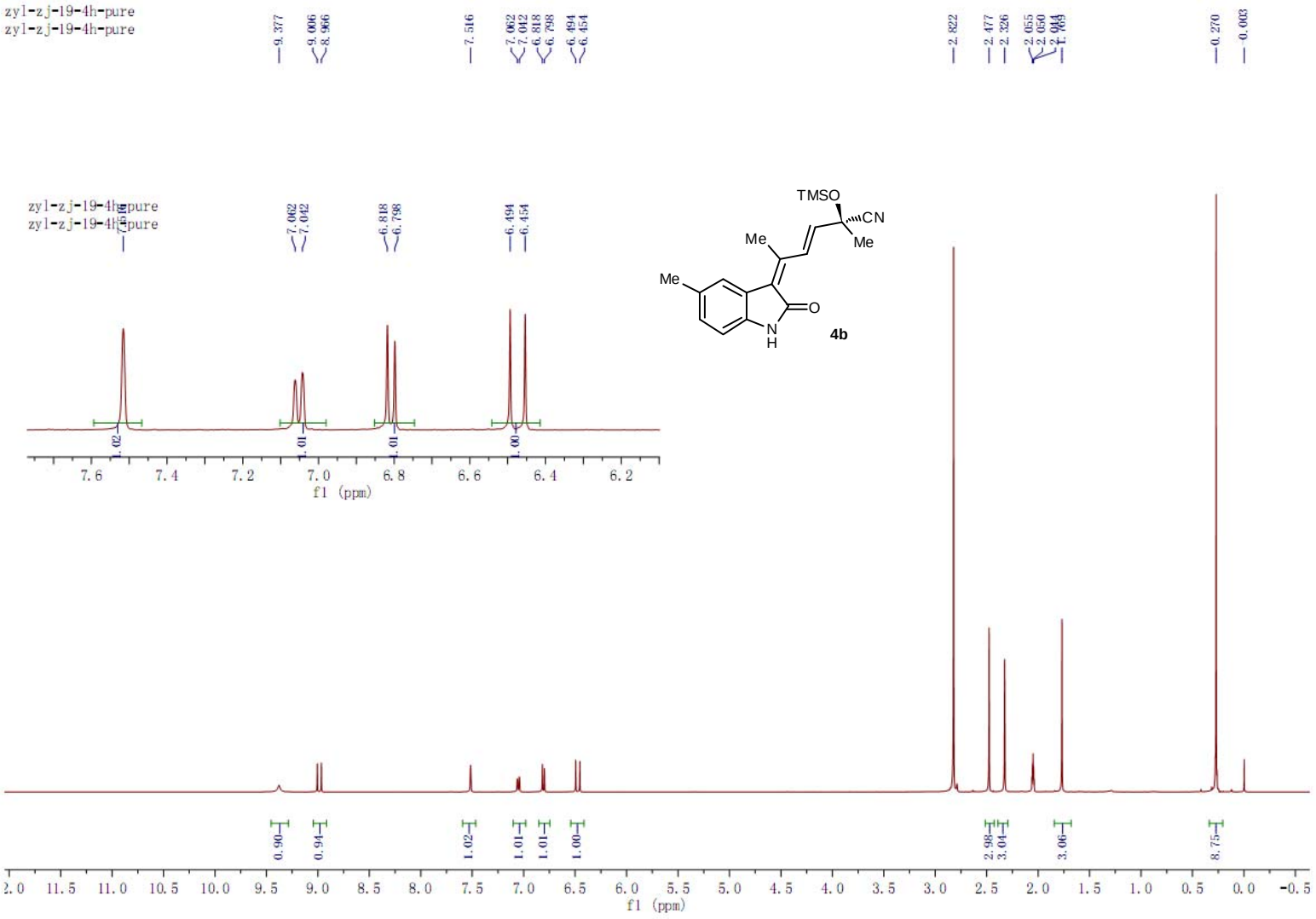
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

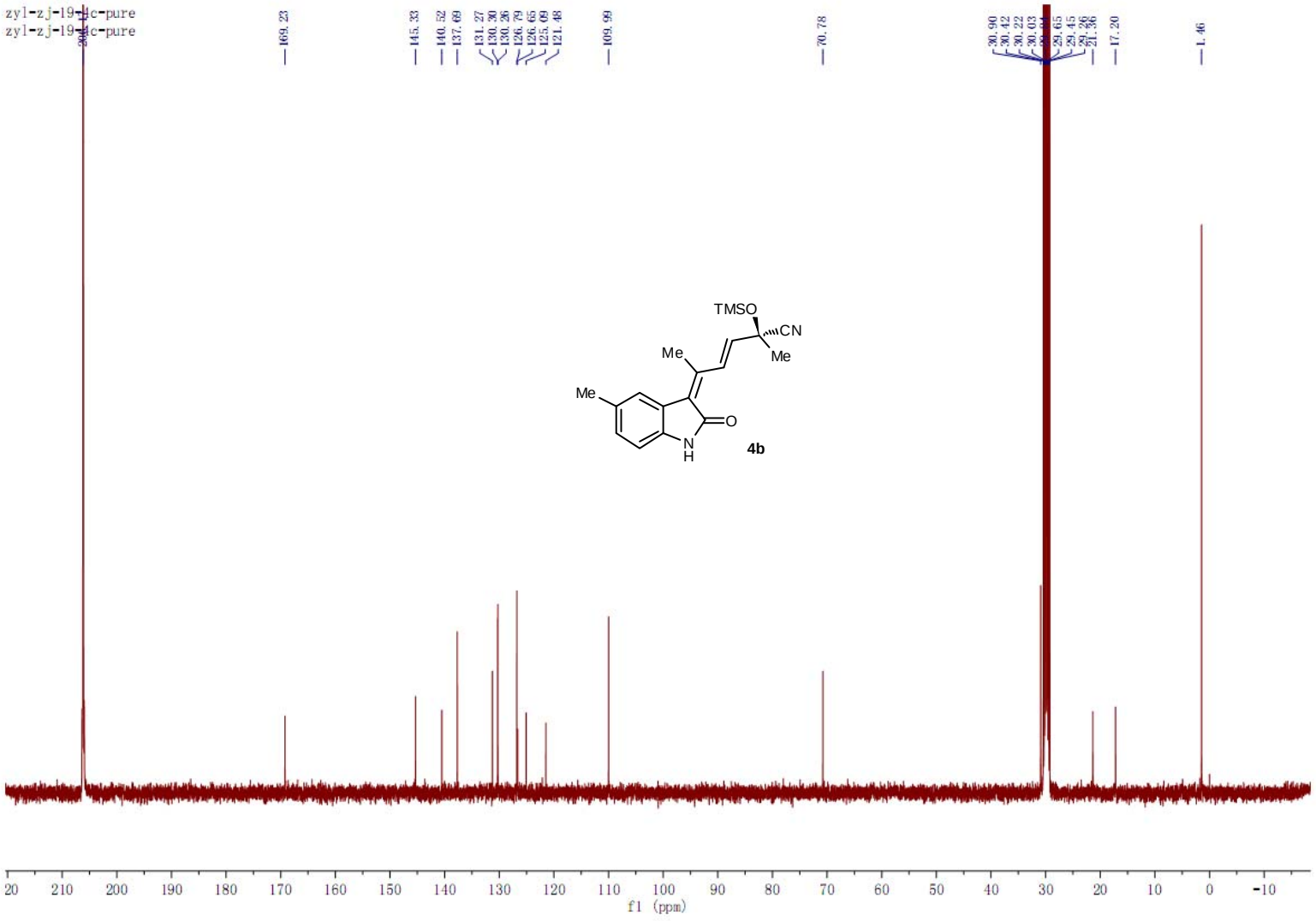

10.807
9.251
9.210
7.633
7.628
7.303
6.838
6.773
6.732
2.511
2.507
2.502
2.419
2.336
Me
Me
O
Cl
O
N
H
3a
0.98
1.00
1.00
0.99
1.02
1.01
2.95
2.98
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
f1 (ppm)

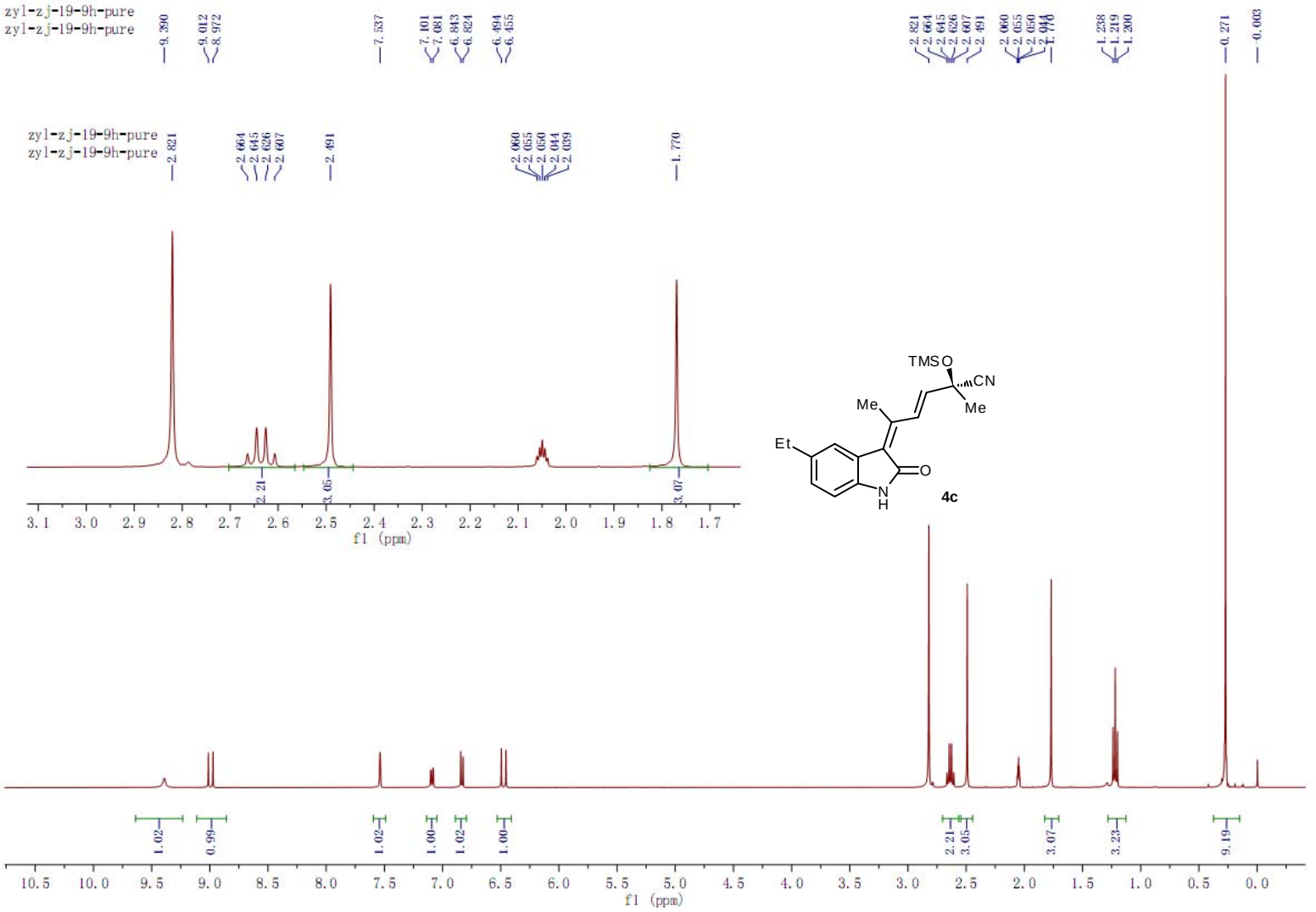

198.54
167.72
146.40
140.91
139.26
133.44
129.35
127.43
125.45
125.17
124.62
110.84
40.09
39.88
39.68
39.47
39.26
39.05
38.84
27.83
16.58
Me
Me
O
Cl
O
N
H
3a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

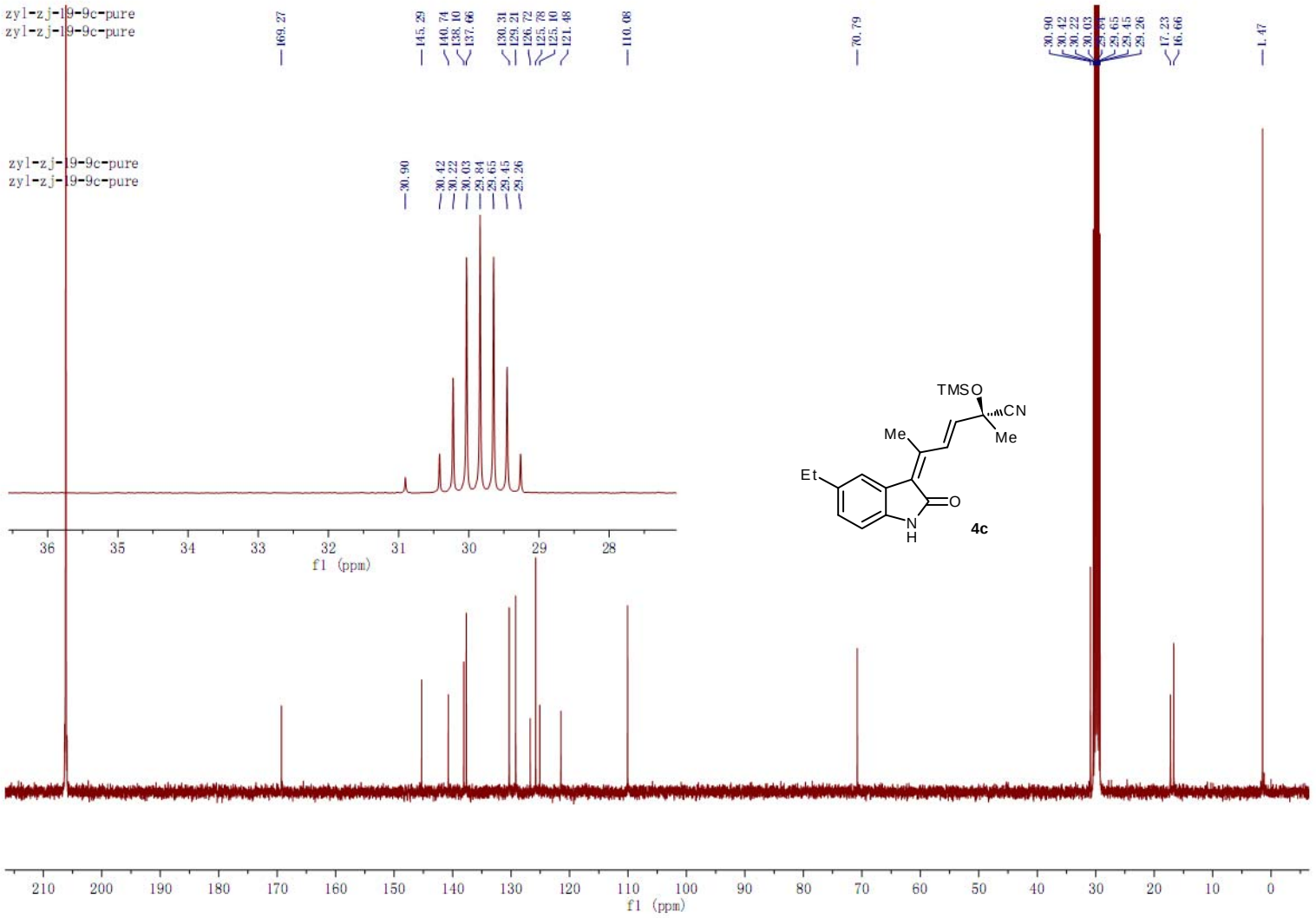

9.612
8.968
8.928
7.647
7.643
7.245
7.240
6.945
6.824
6.559
2.509
2.061
2.056
2.050
2.045
2.039
1.779
0.277
TMSO
CN
Me
Me
Cl
O
N
H
4a
0.98
1.00
1.06
1.05
1.05
1.07
3.03
3.12
9.16
f1 (ppm)

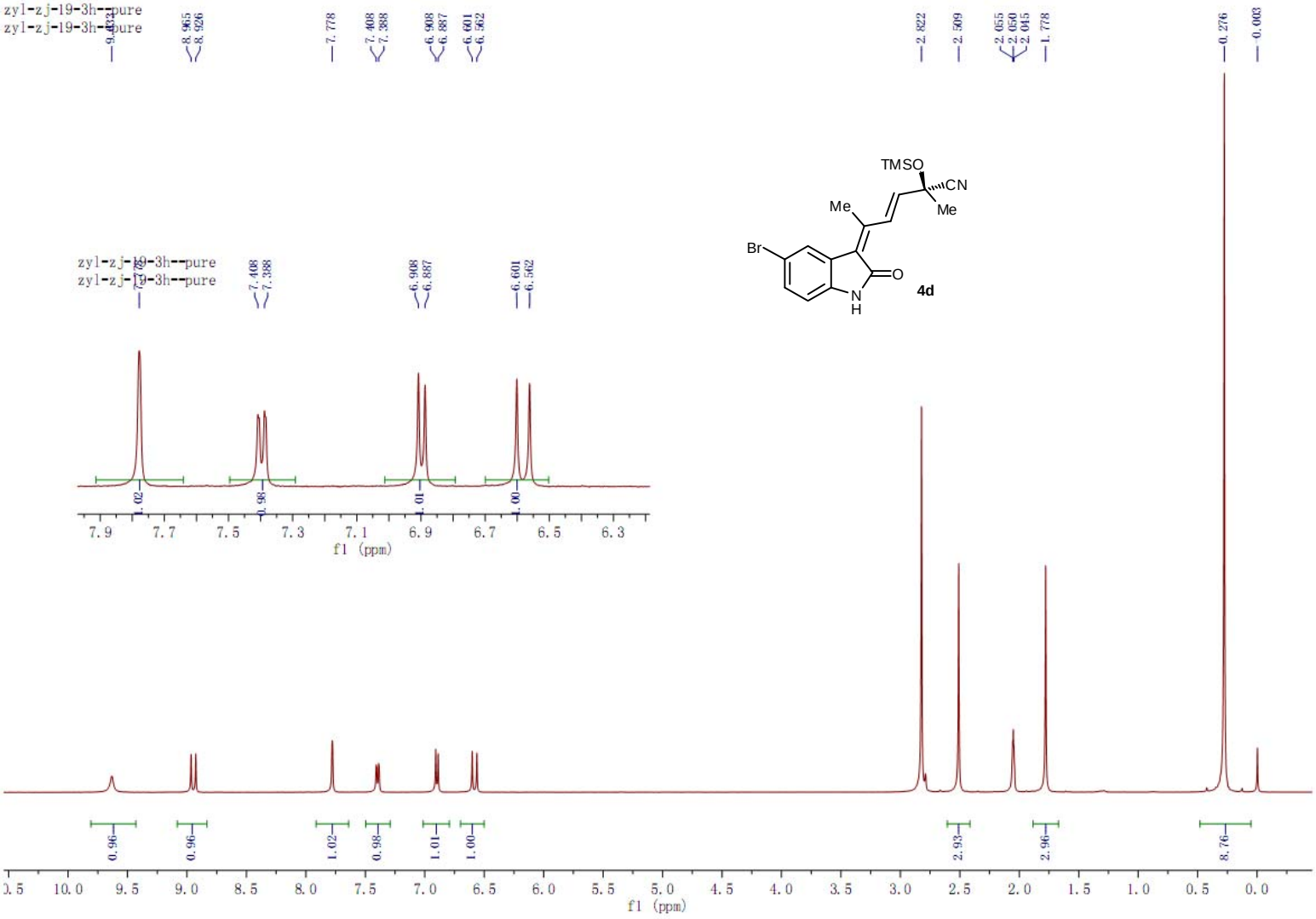

168.82
147.92
141.40
139.21
129.91
129.39
126.98
126.51
125.78
125.55
121.40
111.44
70.78
30.90
30.48
30.28
30.09
29.90
29.71
29.52
29.32
17.39
1.49
TMSO
CN
Me
Me
Cl
O
N
H
4a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

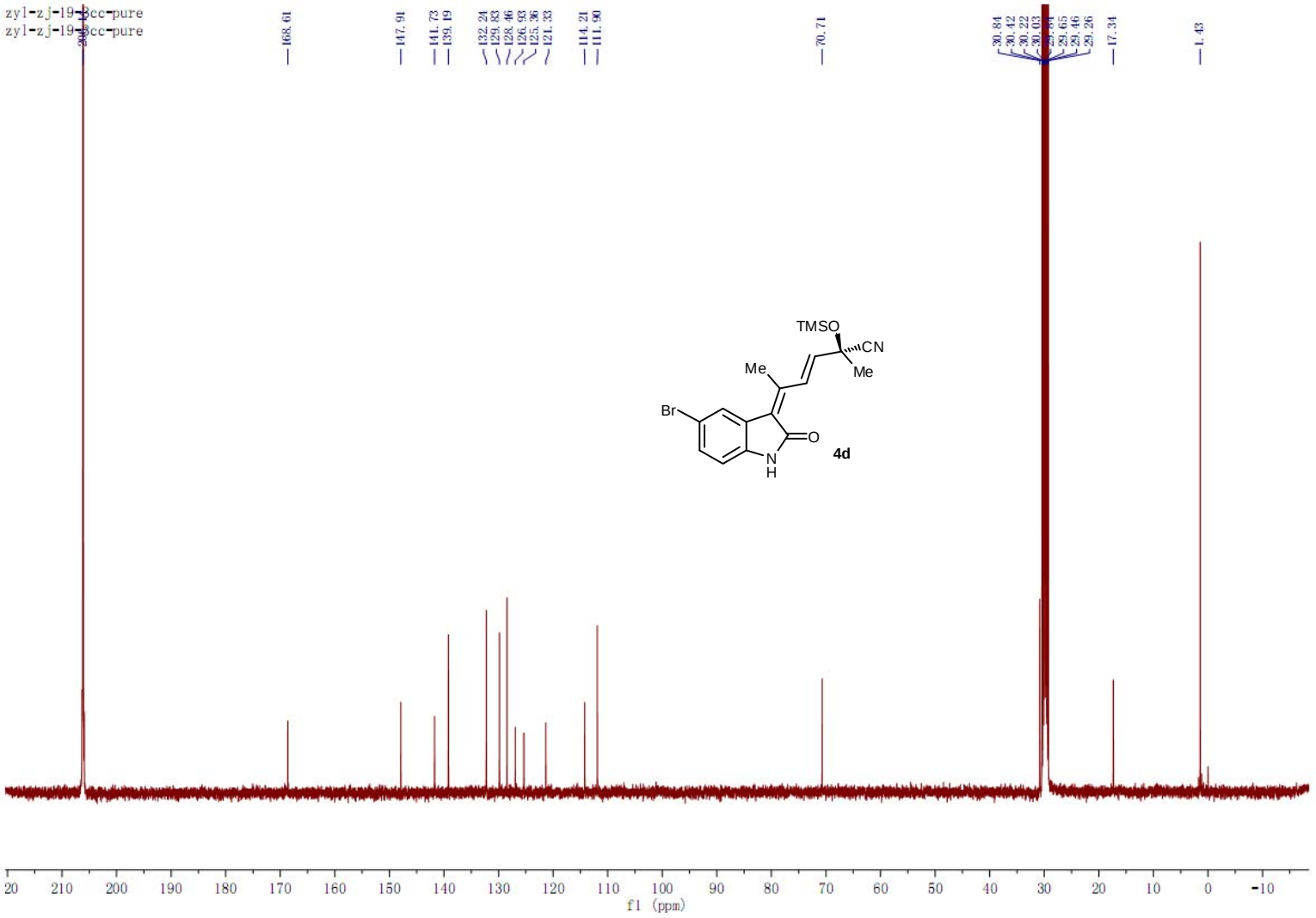

zyl-zj-19-4h-pure
zyl-zj-19-4h-pure
9.377
9.006
8.966
7.516
7.062
7.042
6.818
6.798
6.494
6.454
2.822
2.477
2.326
2.055
2.050
1.969
0.270
0.003
TMSO
CN
Me
Me
Me
O
N
H
4b
1.02
1.01
1.01
1.00
f1 (ppm)
0.90
0.94
1.02
1.01
1.01
1.00
2.98
3.04
3.06
8.75
f1 (ppm)

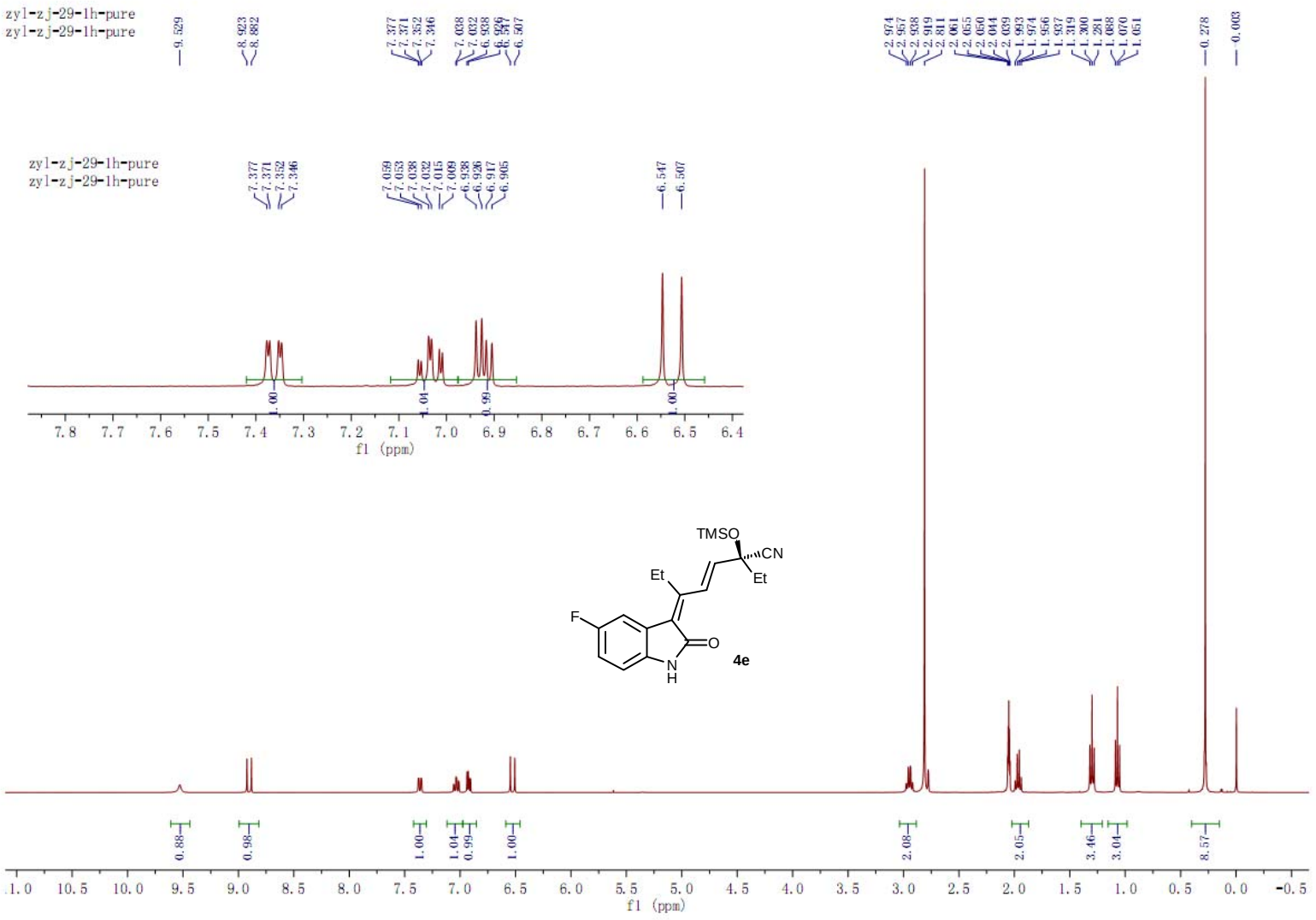

zyl-zj-19-1c-pure
zyl-zj-19-1c-pure
169.23
145.33
140.52
137.69
131.27
130.30
130.26
126.79
126.65
125.09
121.48
109.99
70.78
30.90
30.42
30.22
30.03
29.65
29.45
29.26
21.36
17.20
1.46
TMSO
CN
Me
Me
Me
O
N
H
4b
f1 (ppm)


zyl-zj-19-9h-pure
zyl-zj-19-9h-pure
9.390
9.012
8.972
7.537
7.101
7.081
6.843
6.824
6.494
6.455
2.821
2.664
2.645
2.626
2.607
2.491
2.060
2.055
2.050
2.044
2.039
1.770
1.238
1.219
1.200
0.271
0.003
TMS O
CN
Me
Me
Et
O
N
H
4c
f1 (ppm)

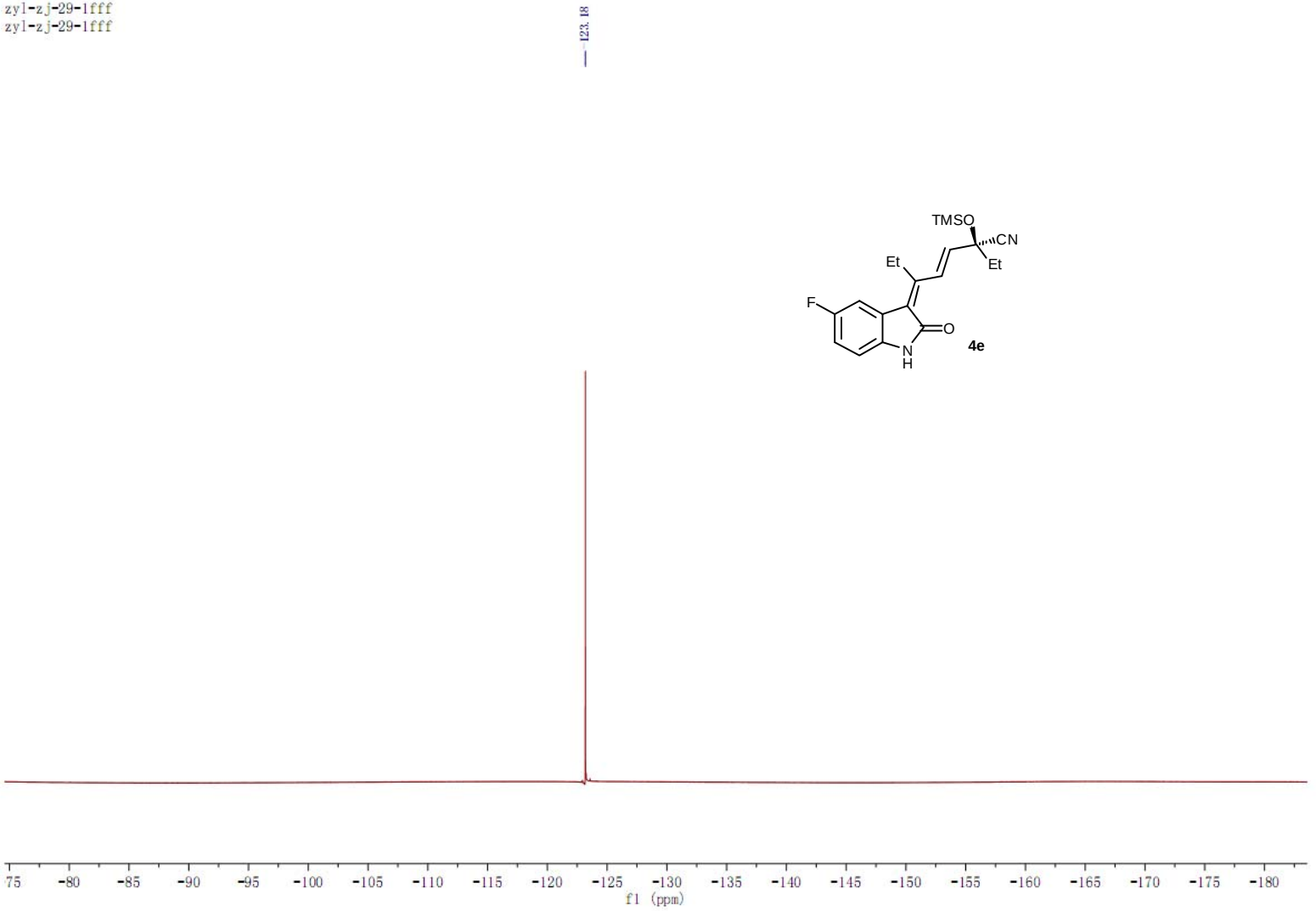

zyl-zj-19-9c-pure
zyl-zj-19-9c-pure
169.27
145.29
140.74
138.10
137.66
130.31
129.21
126.72
125.78
125.10
121.48
110.08
70.79
30.90
30.42
30.22
30.03
29.84
29.65
29.45
29.26
17.23
16.66
1.47
TMSO
CN
Me
Me
Et
O
N
H
4c
f1 (ppm)
f1 (ppm)

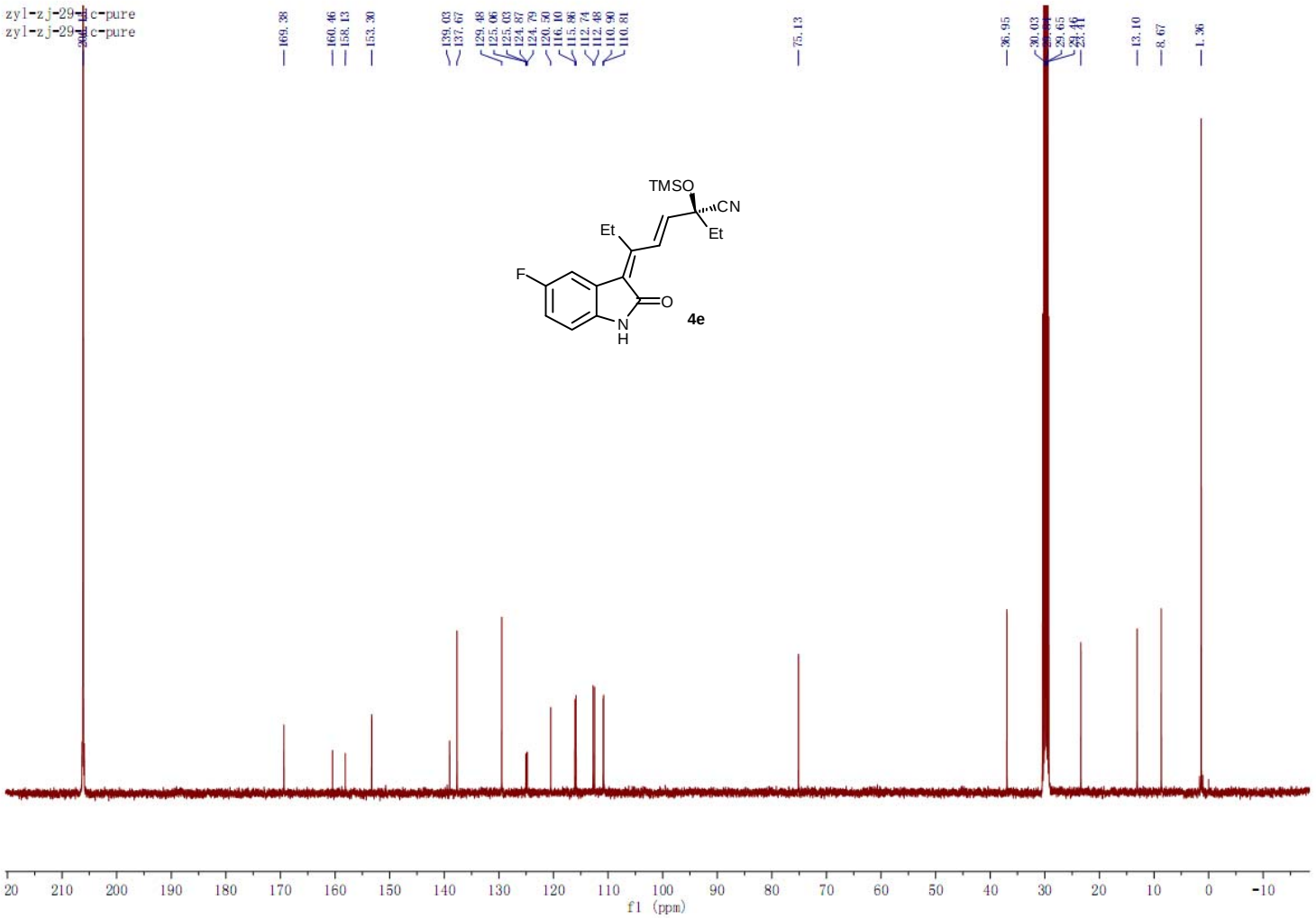

zyl-zj-19-3h-pure
zyl-zj-19-3h-pure
9.432
8.965
8.926
7.778
7.408
7.388
6.908
6.887
6.601
6.562
2.822
2.509
2.055
2.050
2.045
1.778
0.276
0.003
TMSO
CN
Me
Me
Br
O
N
H
4d
zyl-zj-19-3h--pure
zyl-zj-19-3h--pure
7.408
7.388
6.908
6.887
6.601
6.562
1.02
0.98
1.01
1.00
7.9
7.7
7.5
7.3
7.1
6.9
6.7
6.5
6.3
f1 (ppm)
0.96
0.96
1.02
0.98
1.01
1.00
2.93
2.96
8.76
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

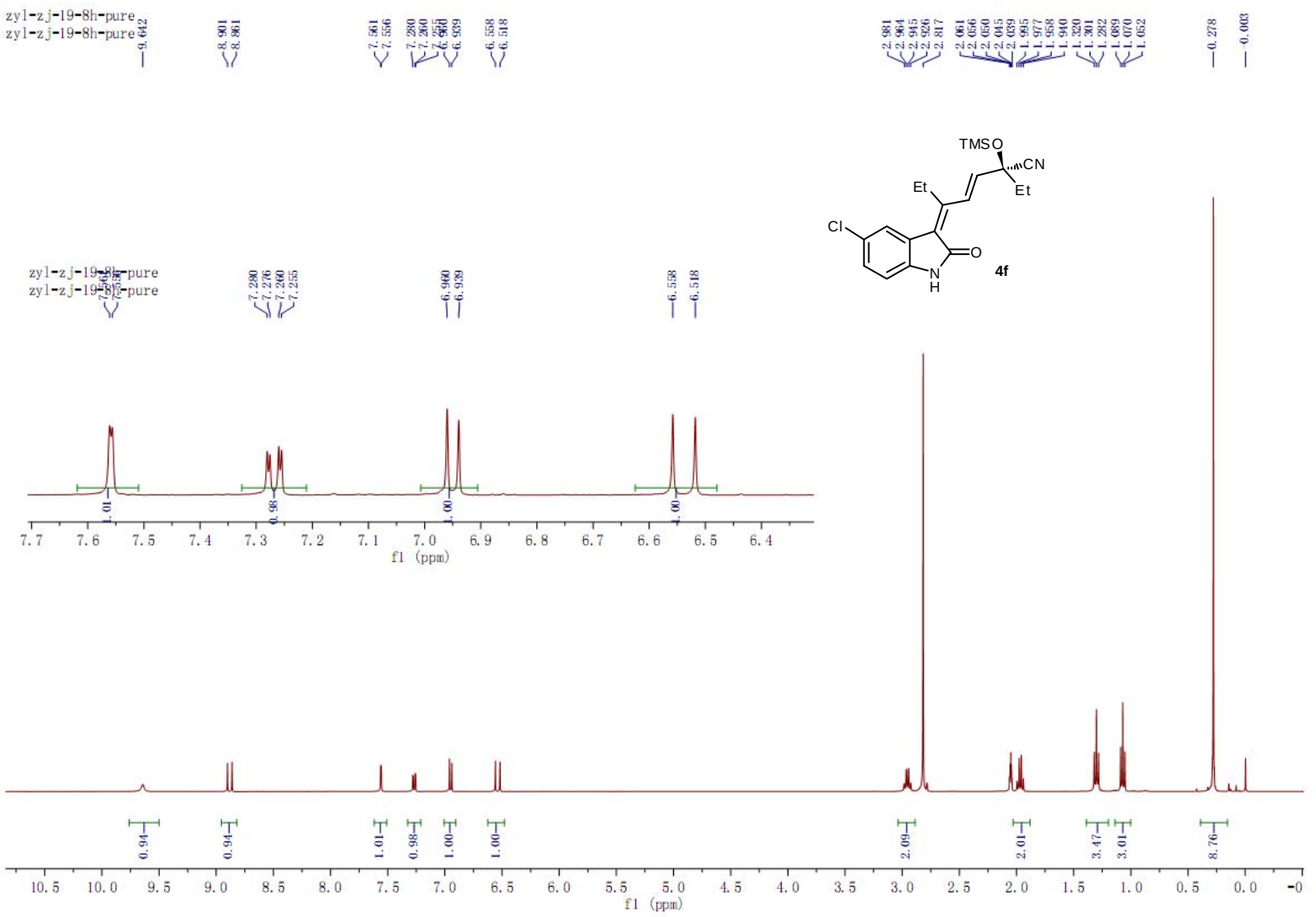

zyl-zj-19-3cc-pure
zyl-zj-19-3cc-pure
168.61
147.91
141.73
139.19
132.24
129.83
128.46
126.93
125.36
121.33
114.21
111.90
70.71
30.84
30.42
30.22
30.03
29.65
29.46
29.26
17.34
1.43
TMSO
CN
Me
Me
Br
O
N
H
4d
f1 (ppm)

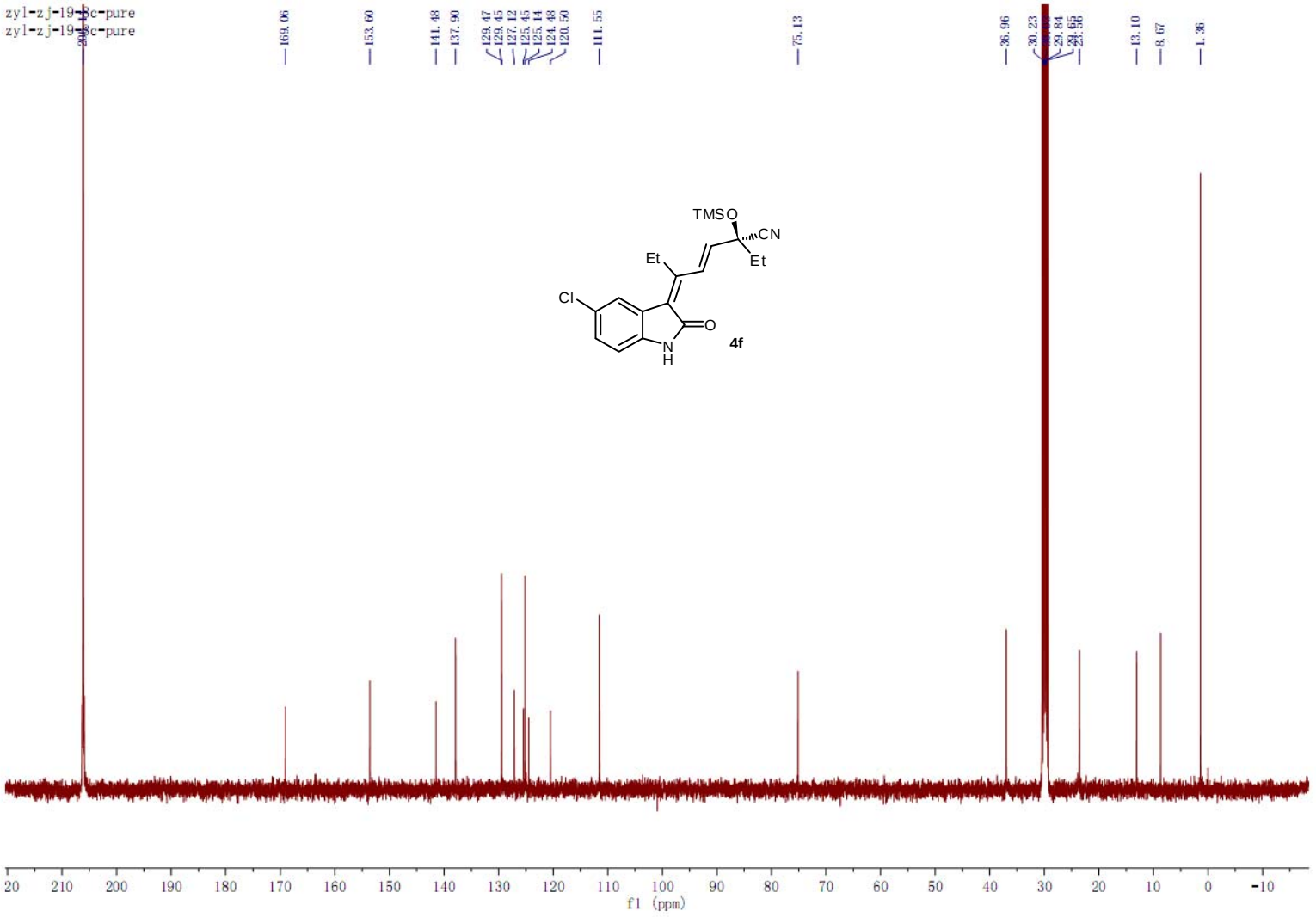

zyl-zj-29-1h-pure
zyl-zj-29-1h-pure
9.529
8.923
8.882
7.377
7.371
7.352
7.346
7.038
7.032
6.938
6.926
6.547
6.507
2.974
2.957
2.938
2.919
2.811
2.061
2.055
2.050
2.044
2.039
1.993
1.974
1.956
1.937
1.319
1.300
1.291
1.088
1.070
1.051
0.278
0.003
TMSO
CN
Et
Et
F
O
N
H
4e
f1 (ppm)

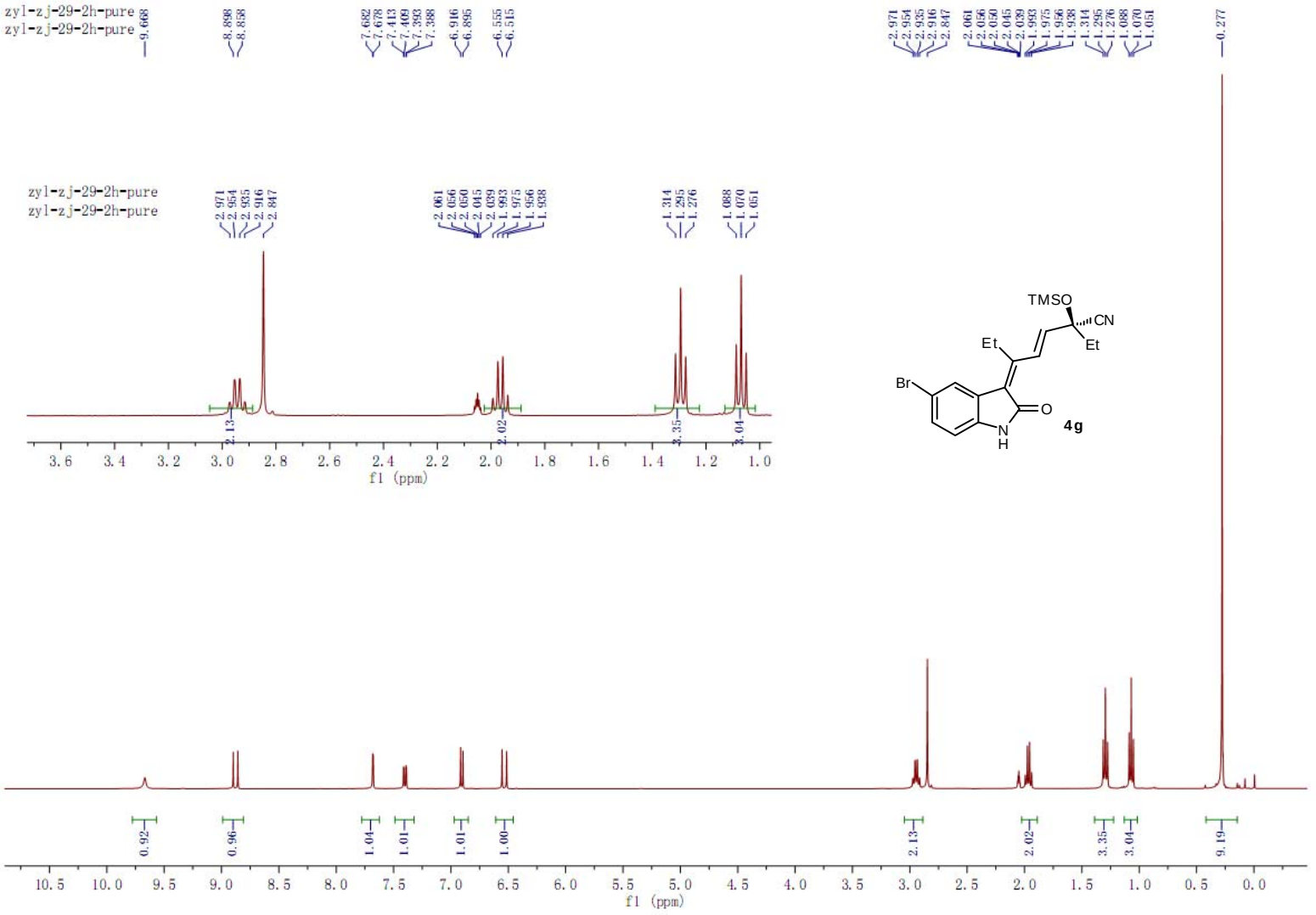
zyl-zj-29-1fff
zyl-zj-29-1fff

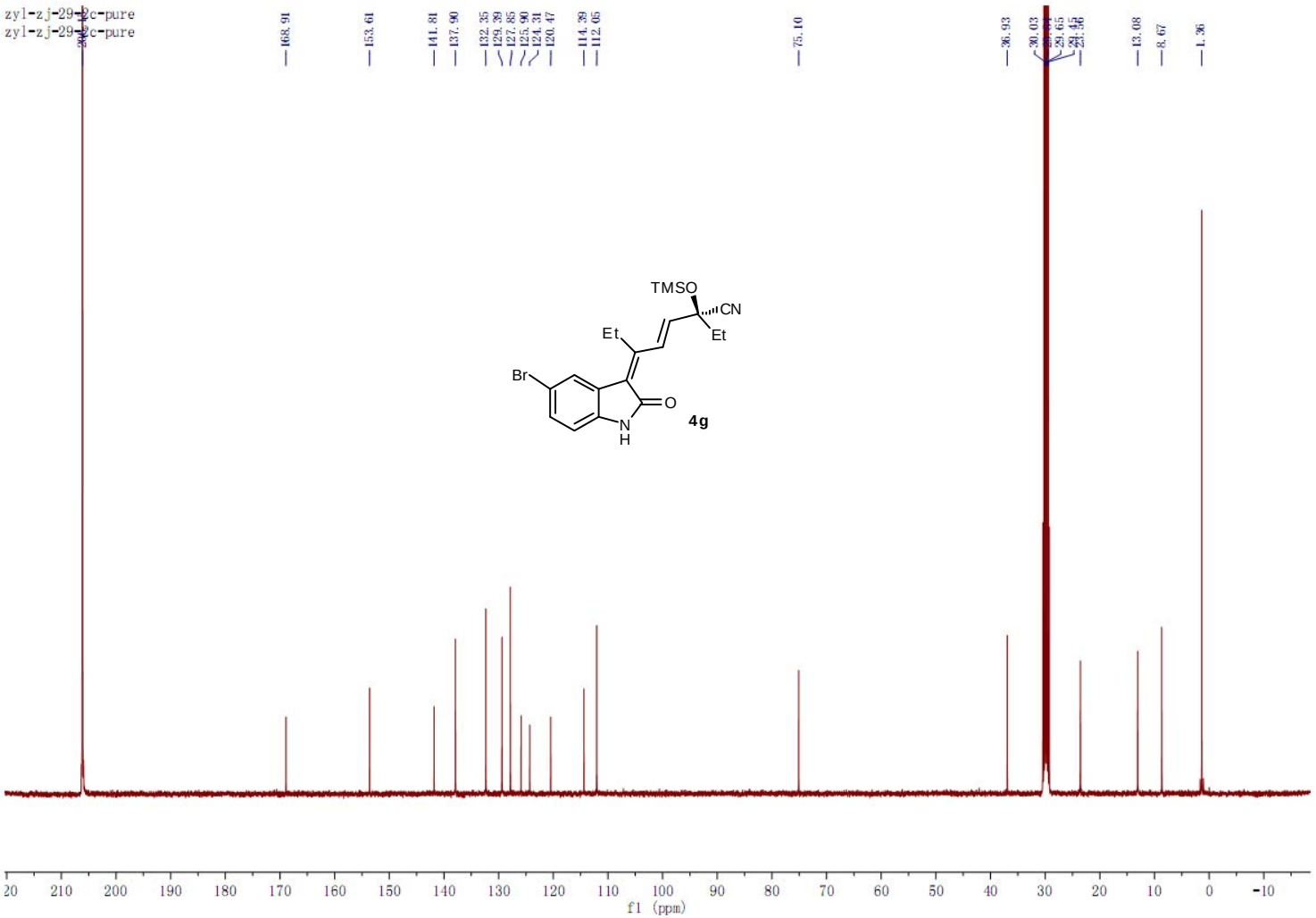


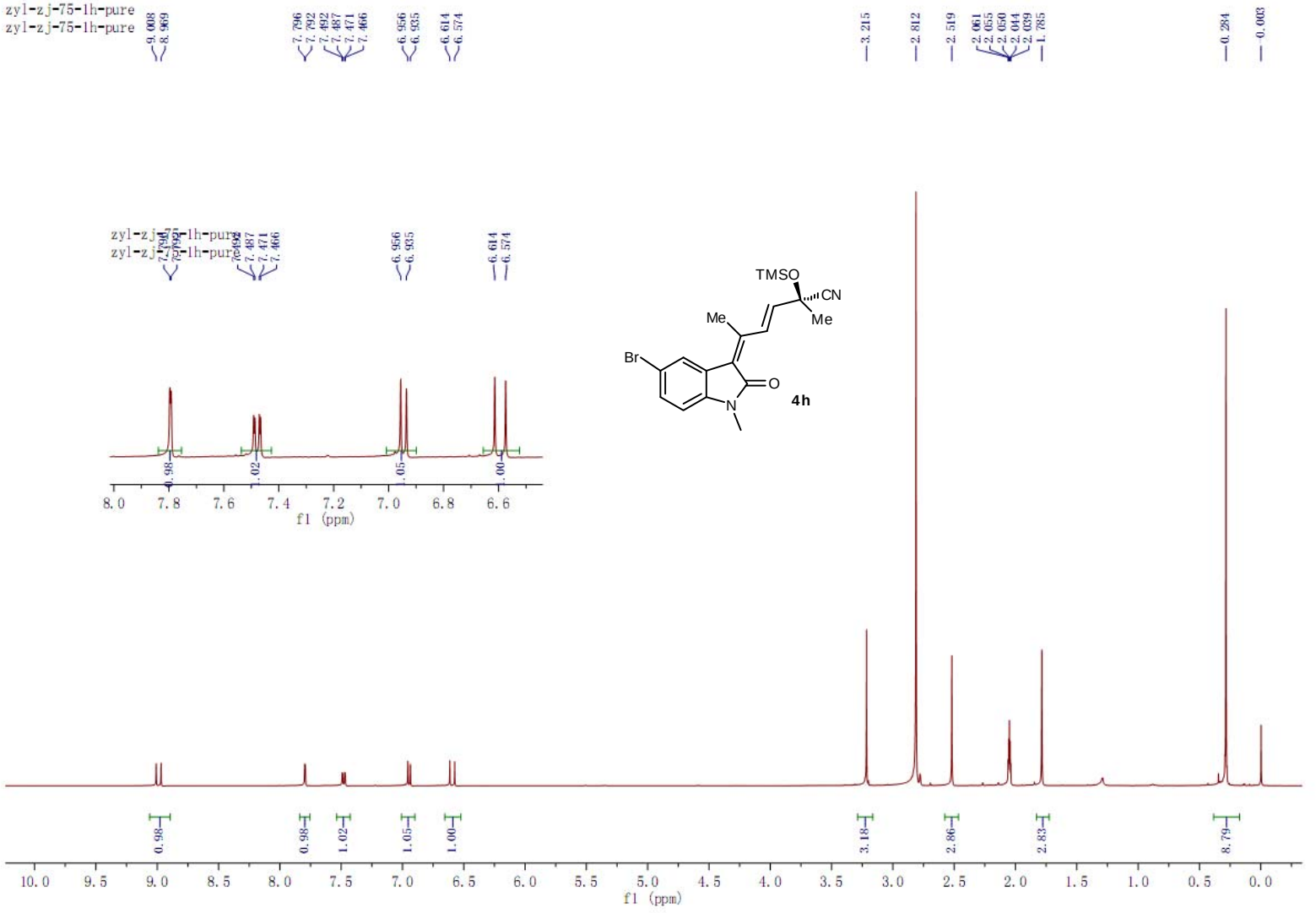

zyl-zj-29-1c-pure
zyl-zj-29-1c-pure
169.38
160.46
158.13
153.30
139.03
137.67
129.48
125.06
125.03
124.87
124.79
120.50
116.10
115.86
112.74
112.48
110.90
110.81
75.13
26.95
30.03
29.65
13.10
8.67
1.36
TMSO
CN
Et
Et
F
O
N
H
4e
f1 (ppm)

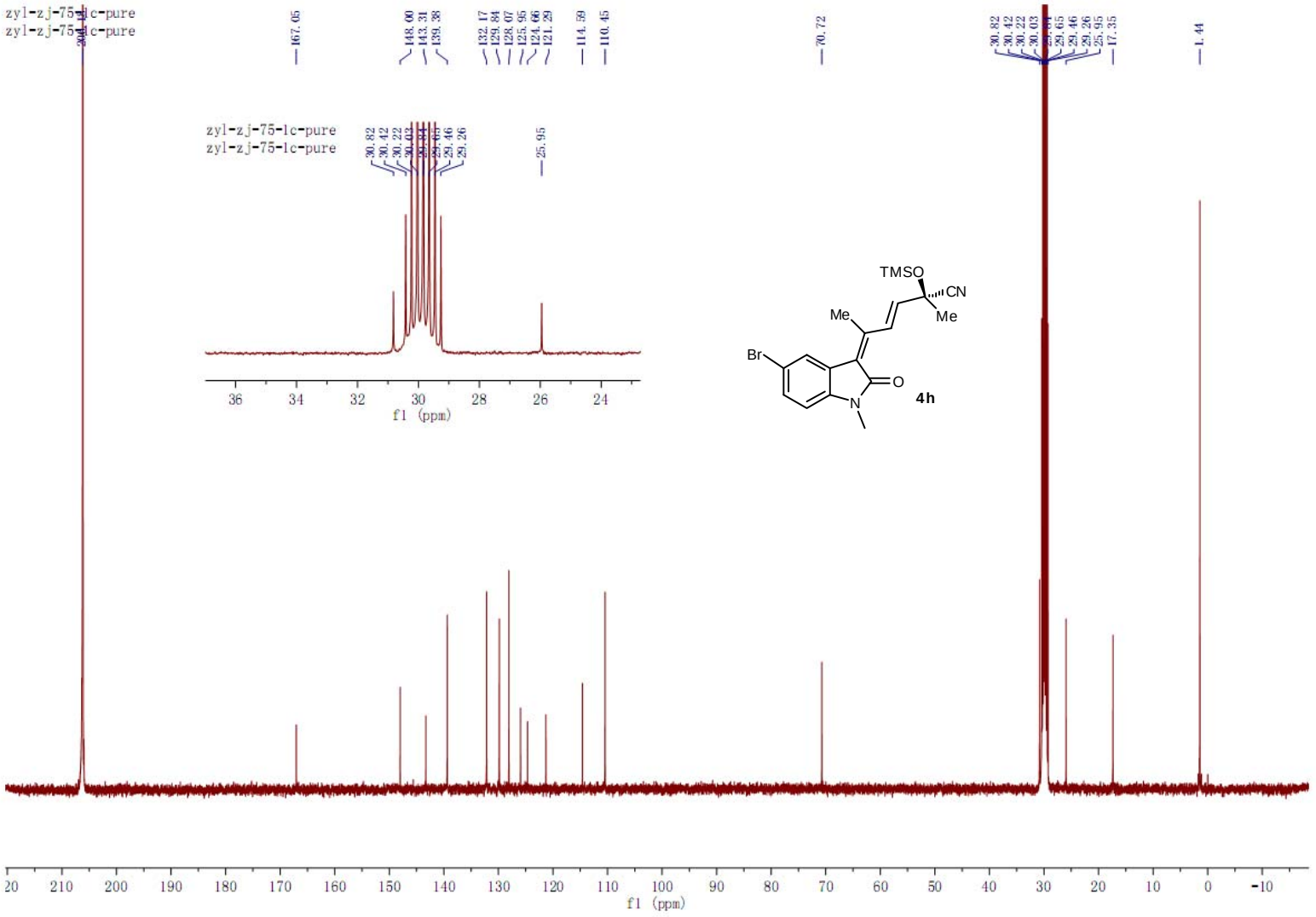

zyl-zj-19-8h-pure
zyl-zj-19-8h-pure
9.642
8.901
8.861
7.561
7.556
7.280
7.260
7.255
6.960
6.939
6.558
6.518
2.981
2.964
2.945
2.926
2.817
2.061
2.056
2.050
2.045
2.039
1.995
1.977
1.958
1.940
1.320
1.301
1.282
1.089
1.070
1.052
0.278
0.003
TMSO
CN
Et
Et
Cl
O
N
H
4f
f1 (ppm)
f1 (ppm)

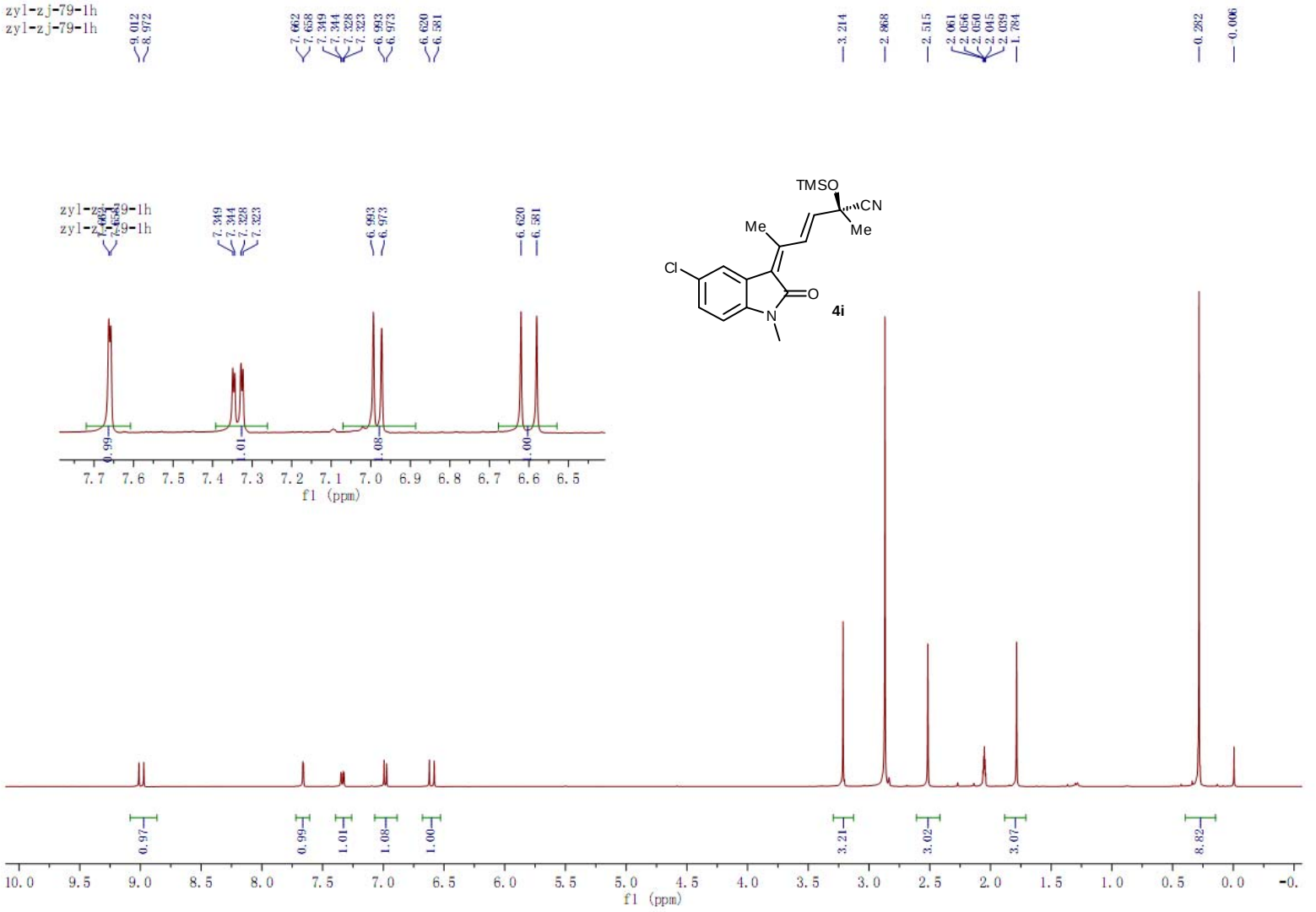

zyl-zj-19-3c-pure
zyl-zj-19-3c-pure
169.06
153.60
141.48
137.90
129.47
129.45
127.12
125.45
125.14
124.48
120.50
111.55
75.13
36.96
30.23
29.84
29.65
23.56
13.10
8.67
1.36
TMSO
CN
Et
Et
Cl
O
N
H
4f
f1 (ppm)

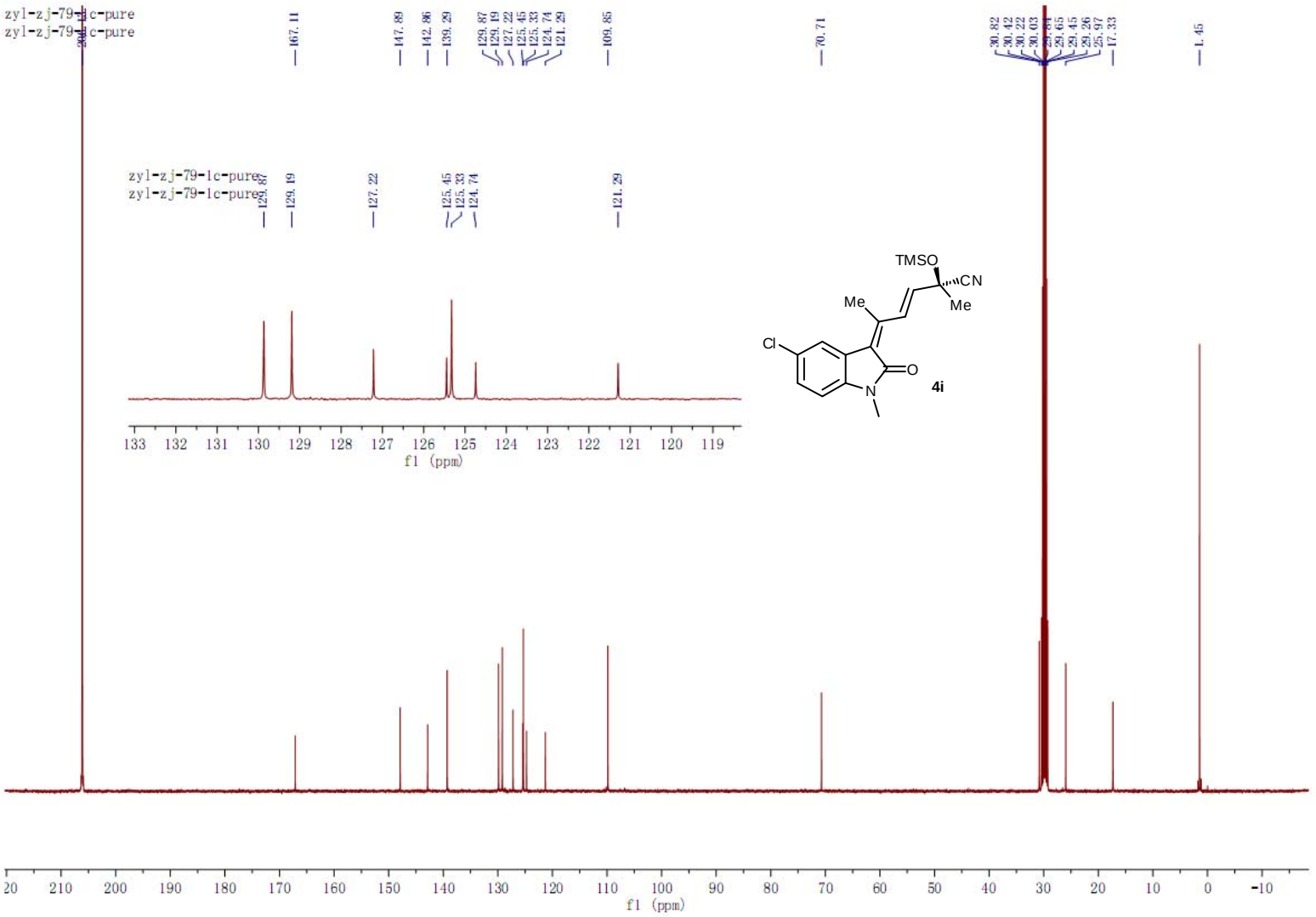

zyl-zj-29-2h-pure
zyl-zj-29-2h-pure
9.668
8.898
8.858
7.682
7.678
7.413
7.409
7.393
7.388
6.916
6.895
6.555
6.515
2.971
2.954
2.935
2.916
2.847
2.061
2.056
2.050
2.045
2.039
1.993
1.975
1.956
1.938
1.314
1.295
1.276
1.088
1.070
1.051
0.277
TMSO
CN
Et
Et
Br
O
N
H
4g
f1 (ppm)

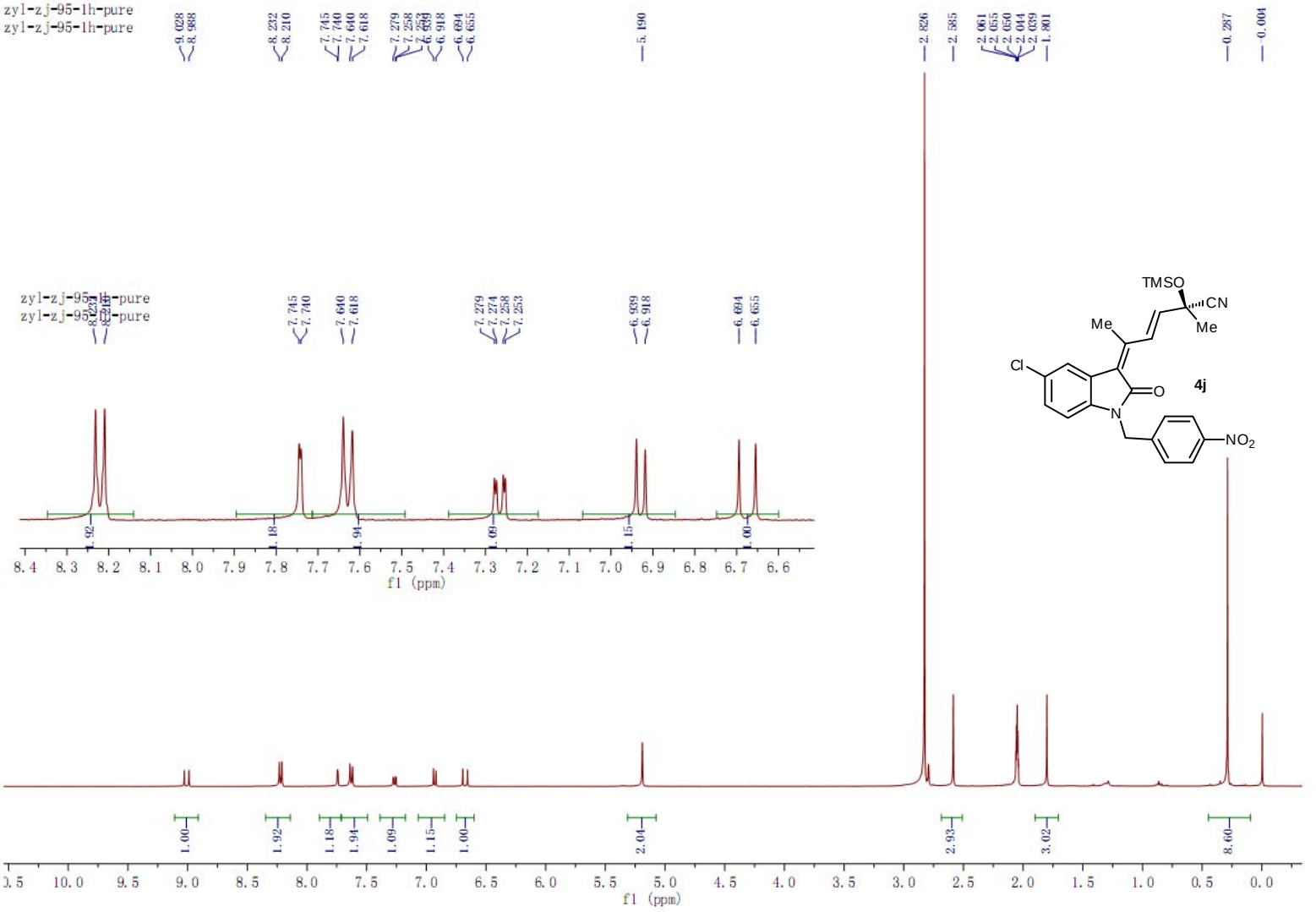

zyl-zj-29-2c-pure
zyl-zj-29-2c-pure
168.91
153.61
141.81
137.90
132.35
129.39
127.85
125.90
124.31
120.47
114.39
112.05
75.10
36.93
30.03
29.65
23.36
13.08
8.67
1.36
TMSO
CN
Et
Et
Br
O
N
H
4g
f1 (ppm)

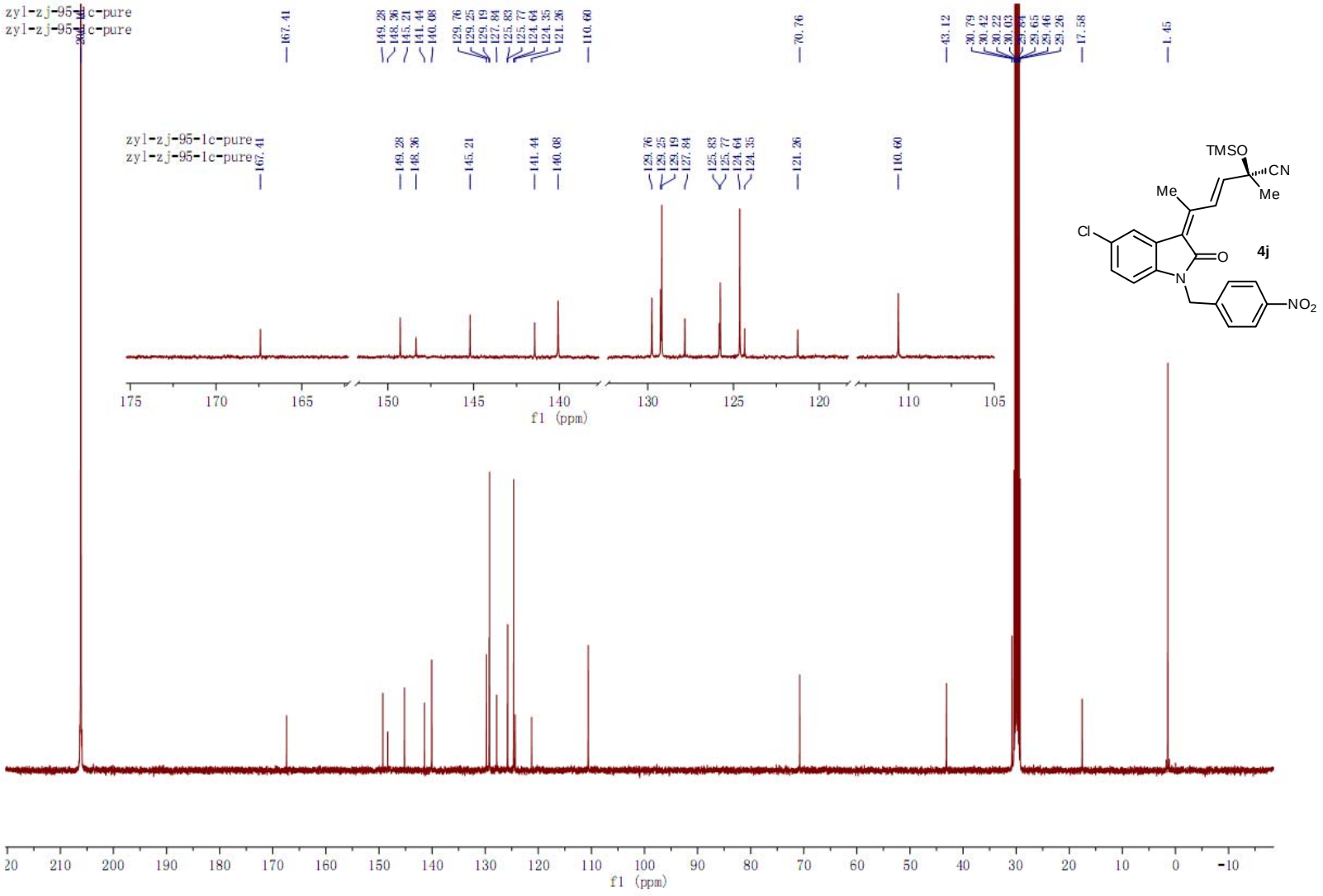

zyl-zj-75-1h-pure
zyl-zj-75-1h-pure
9.008
8.969
7.796
7.792
7.492
7.487
7.471
7.466
6.956
6.935
6.614
6.574
3.215
2.812
2.519
2.061
2.055
2.050
2.044
2.039
1.785
0.284
0.003
TMSO
CN
Me
Me
Br
O
N
4h
0.98
0.98
1.02
1.05
1.00
3.18
2.86
2.83
8.79
f1 (ppm)

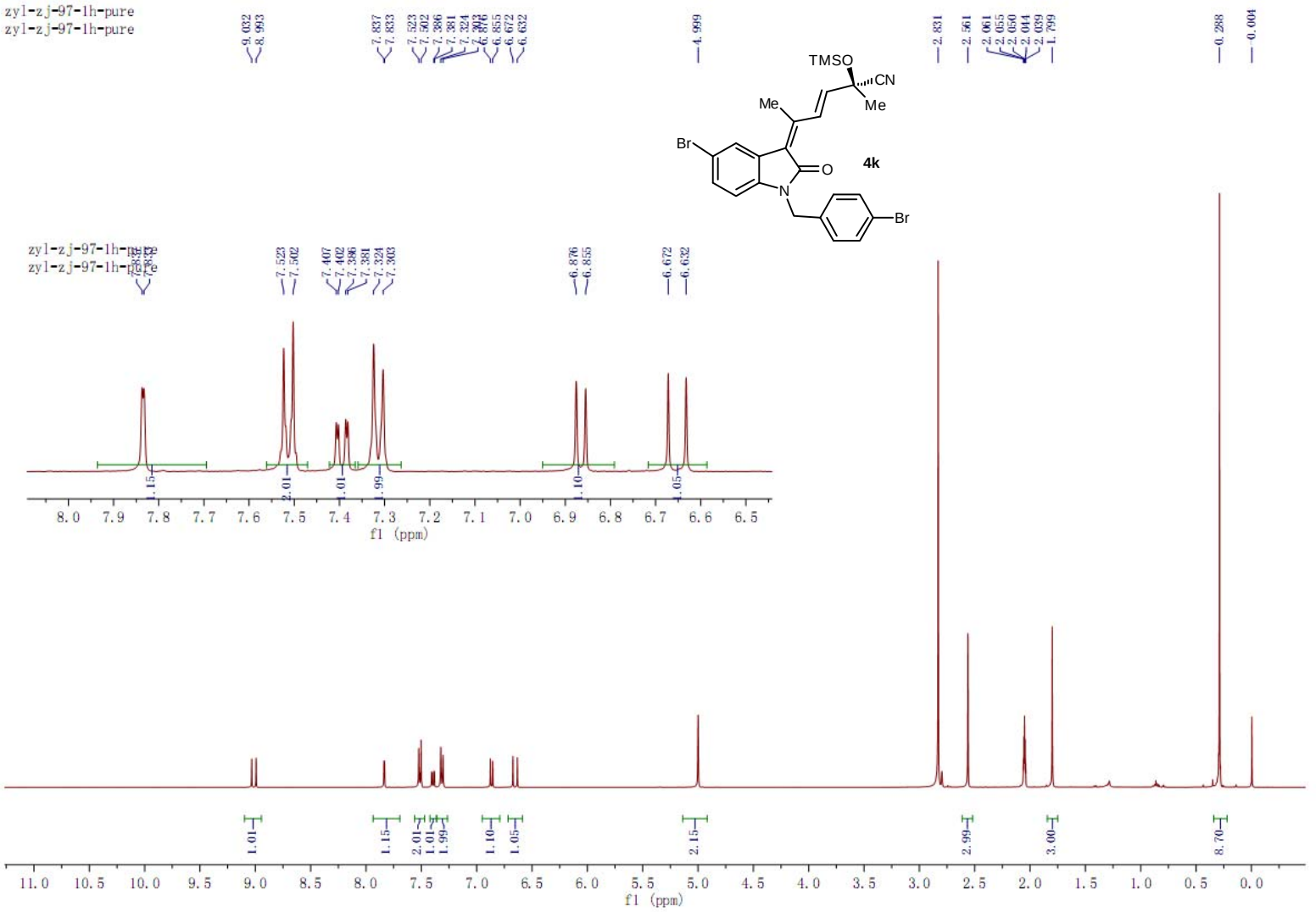

zyl-zj-75-1c-pure
zyl-zj-75-1c-pure
167.05
148.60
143.31
139.38
132.17
129.84
128.07
125.95
124.66
121.29
114.59
110.45
70.72
30.82
30.42
30.22
30.03
29.65
29.46
29.26
25.95
17.35
1.44
zyl-zj-75-1c-pure
zyl-zj-75-1c-pure
25.95
f1 (ppm)
TMSO
CN
Me
Me
Br
O
N
4h
f1 (ppm)

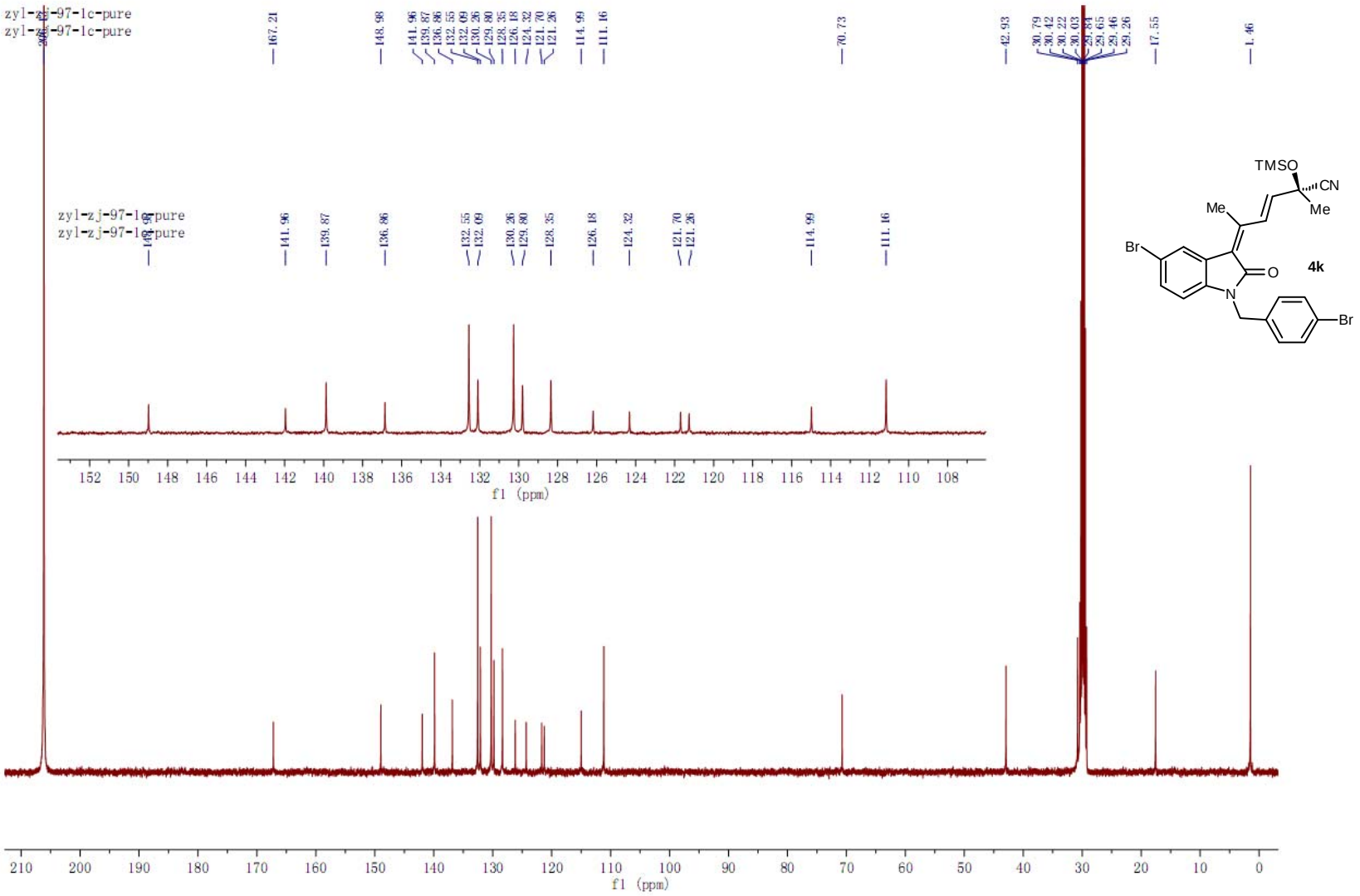

zyl-zj-79-1h
zyl-zj-79-1h
9.012
8.972
7.662
7.658
7.349
7.344
7.328
7.323
6.993
6.973
6.620
6.581
3.214
2.868
2.515
2.061
2.056
2.050
2.045
2.039
1.784
0.282
0.006
TMSO
CN
Me
Me
Cl
O
N
4i
f1 (ppm)
0.97
0.99
1.01
1.08
1.00
3.21
3.02
3.07
8.82

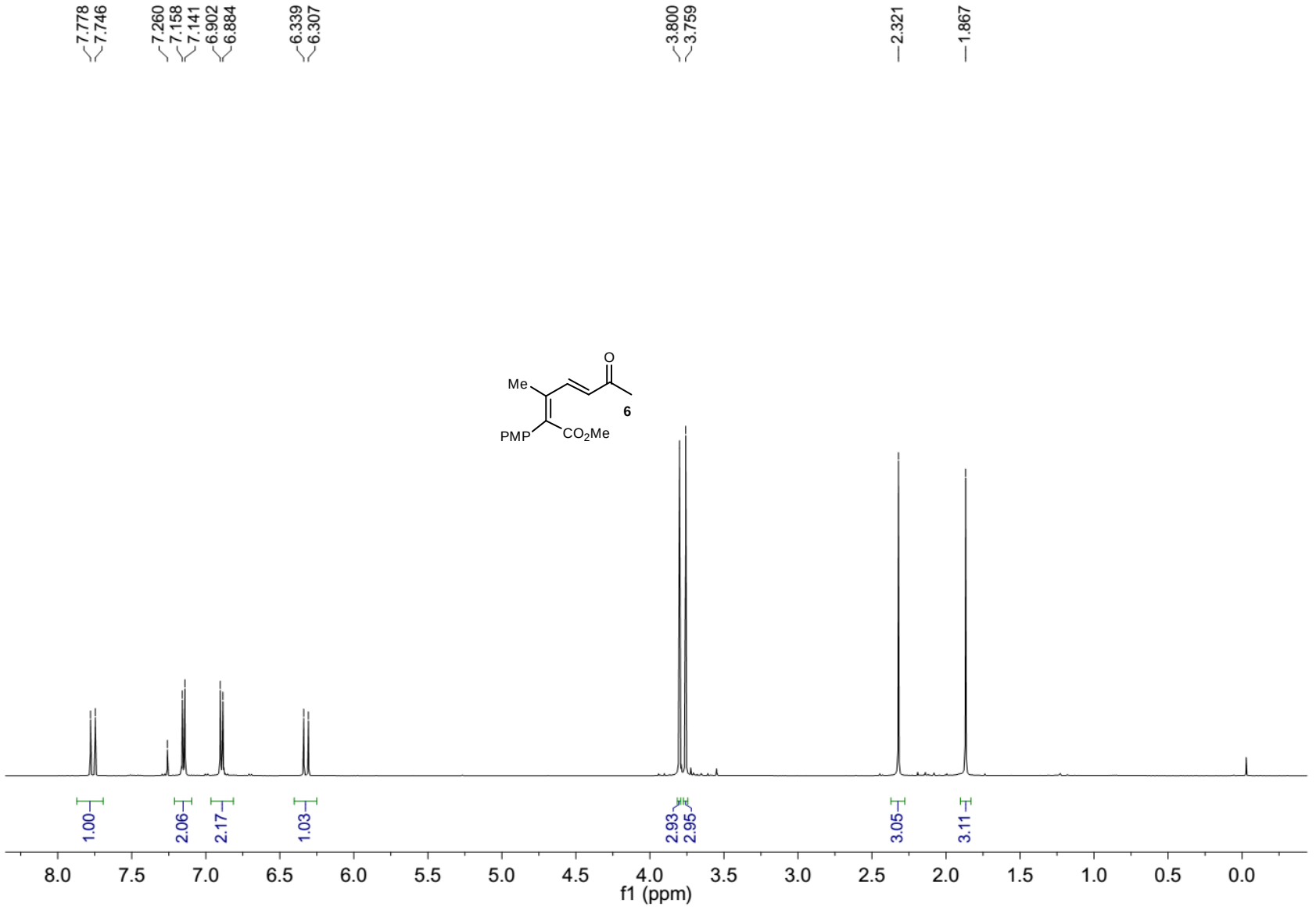

zyl-zj-79-1c-pure
zyl-zj-79-1c-pure
167.11
147.89
142.86
139.29
129.87
129.19
127.22
125.45
125.33
124.74
121.29
109.85
70.71
30.82
30.42
30.22
30.03
29.65
29.45
29.26
25.97
17.33
1.45
zyl-zj-79-1c-pure
zyl-zj-79-1c-pure
129.87
129.19
127.22
125.45
125.33
124.74
121.29
133 132 131 130 129 128 127 126 125 124 123 122 121 120 119
f1 (ppm)
TMSO
CN
Me
Me
Cl
O
N
4i
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

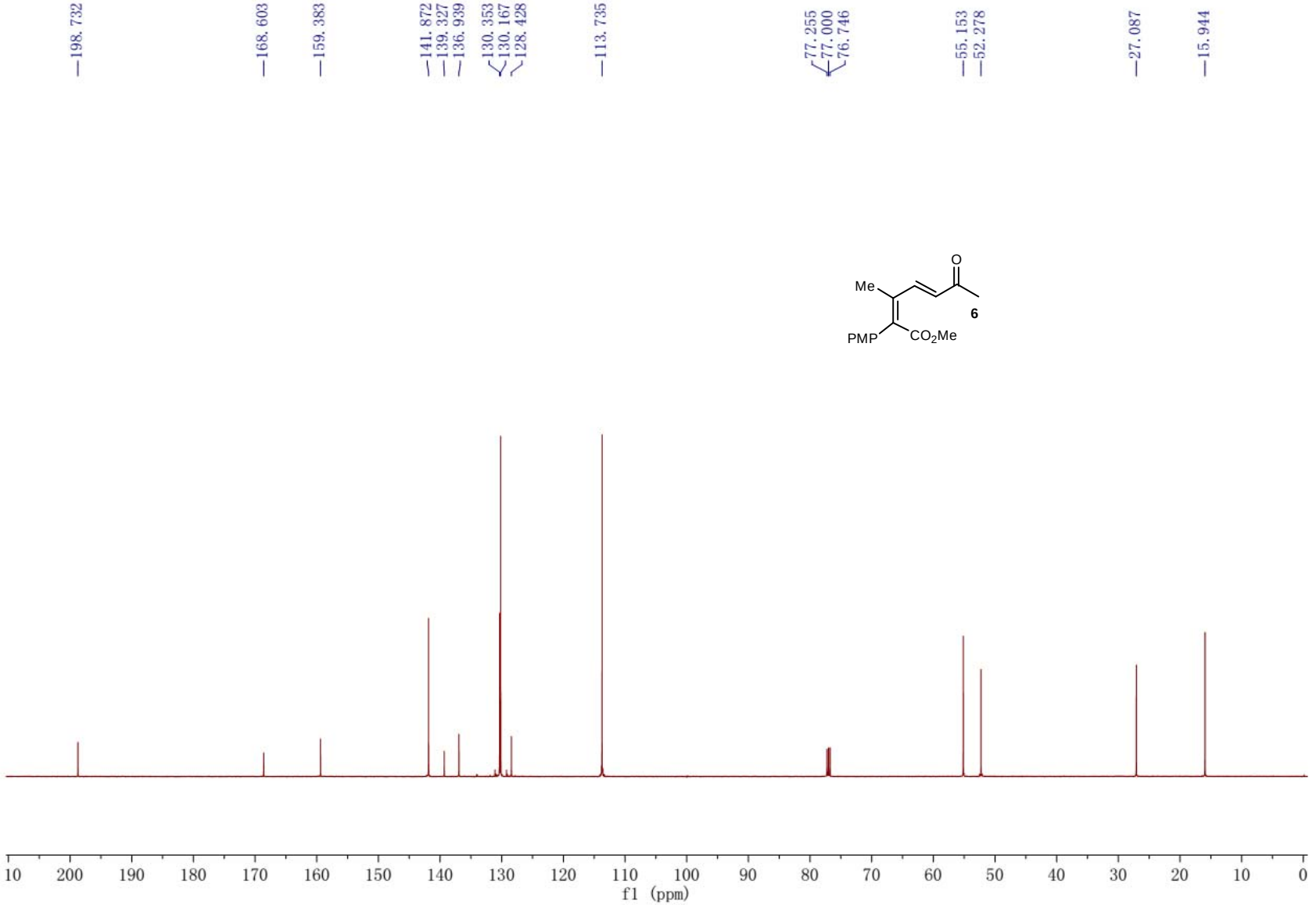

zyl-zj-95-1h-pure
zyl-zj-95-1h-pure
9.028
8.988
8.232
8.210
7.745
7.740
7.640
7.618
7.279
7.274
7.258
7.253
6.939
6.918
6.694
6.655
5.190
2.826
2.585
2.061
2.055
2.050
2.044
2.039
1.801
0.287
0.004
TMSO
CN
Me
Me
Cl
O
N
NO2
4j
f1 (ppm)

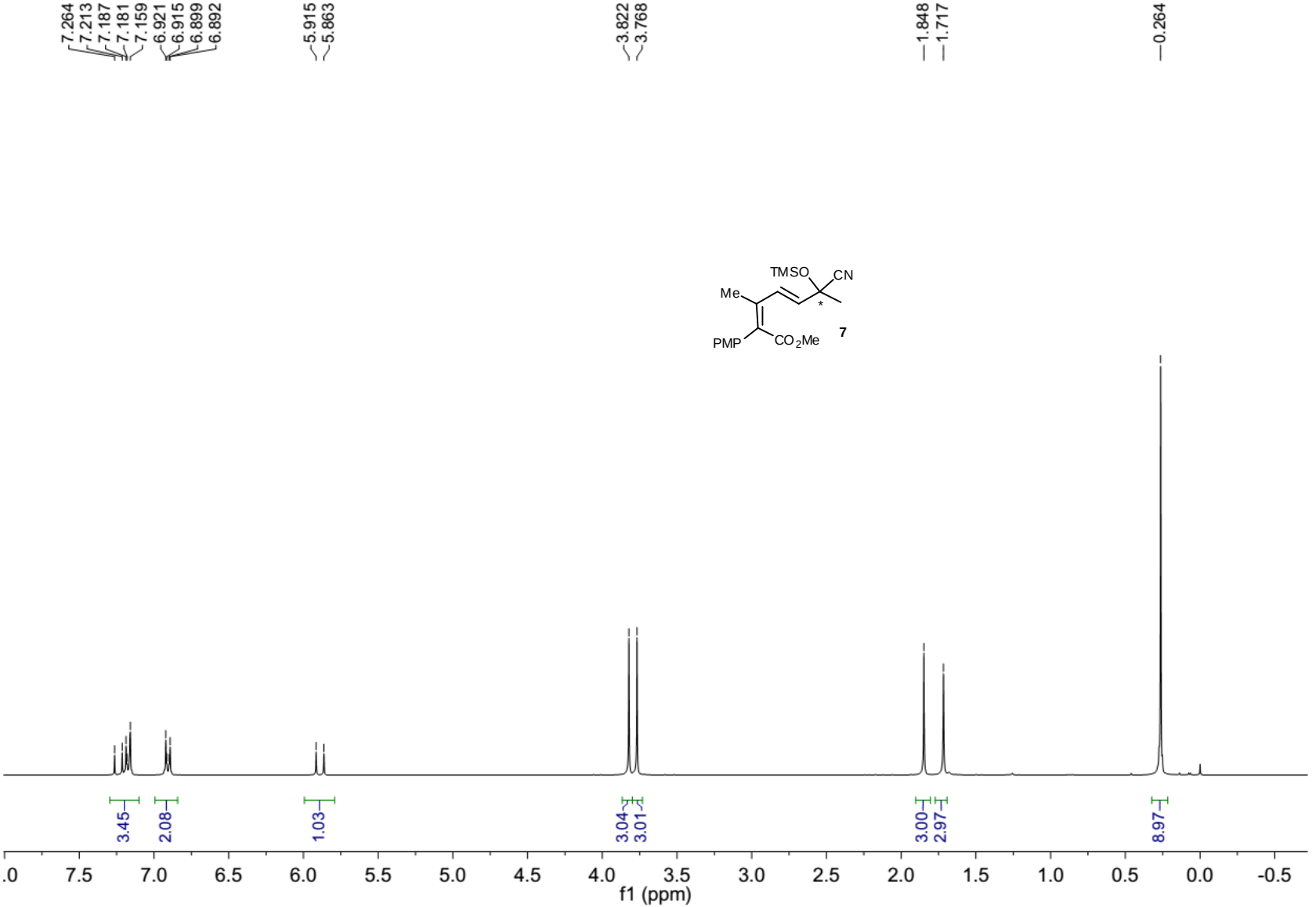

zyl-zj-95-1c-pure
zyl-zj-95-1c-pure
167.41
149.28
148.36
145.21
141.44
140.08
129.76
129.25
129.19
127.84
125.83
125.77
124.64
124.35
121.26
110.60
70.76
43.12
30.79
30.42
30.22
30.03
29.84
29.65
29.46
29.26
17.58
1.45
TMSO
CN
Me
Me
Cl
O
N
NO2
4j
f1 (ppm)


zyl-zj-97-1h-pure
zyl-zj-97-1h-pure
TMSO
CN
Me
Me
Br
O
N
Br
4k
f1 (ppm)
f1 (ppm)


zyl-zj-97-1c-pure
zyl-zj-97-1c-pure
167.21
148.98
141.96
139.87
136.86
132.55
132.09
130.26
129.90
128.35
126.18
124.32
121.70
121.26
114.99
111.16
70.73
42.93
30.79
30.42
30.22
30.03
29.84
29.65
29.46
29.26
17.55
1.46
TMSO
CN
Me
Me
Br
O
N
Br
4k
f1 (ppm)


7.778
7.746
7.260
7.158
7.141
6.902
6.884
6.339
6.307
3.800
3.759
2.321
1.867
Me
O
PMP
CO2Me
6
1.00
2.06
2.17
1.03
2.93
2.95
3.05
3.11
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)


198.732
168.603
159.383
141.872
139.327
136.939
130.353
130.167
128.428
113.735
77.255
77.000
76.746
55.153
52.278
27.087
15.944
Me
O
PMP
CO2Me
6
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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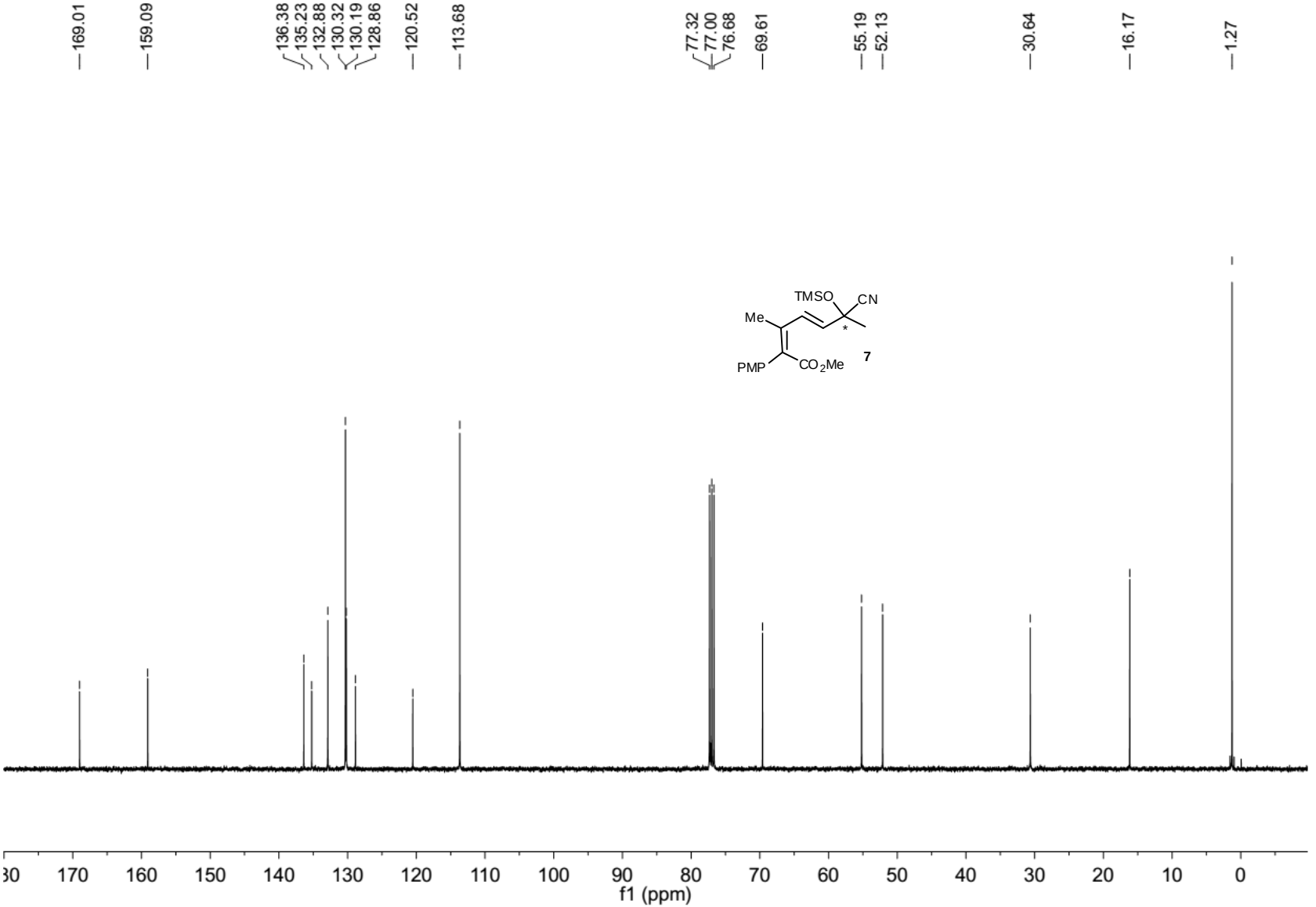
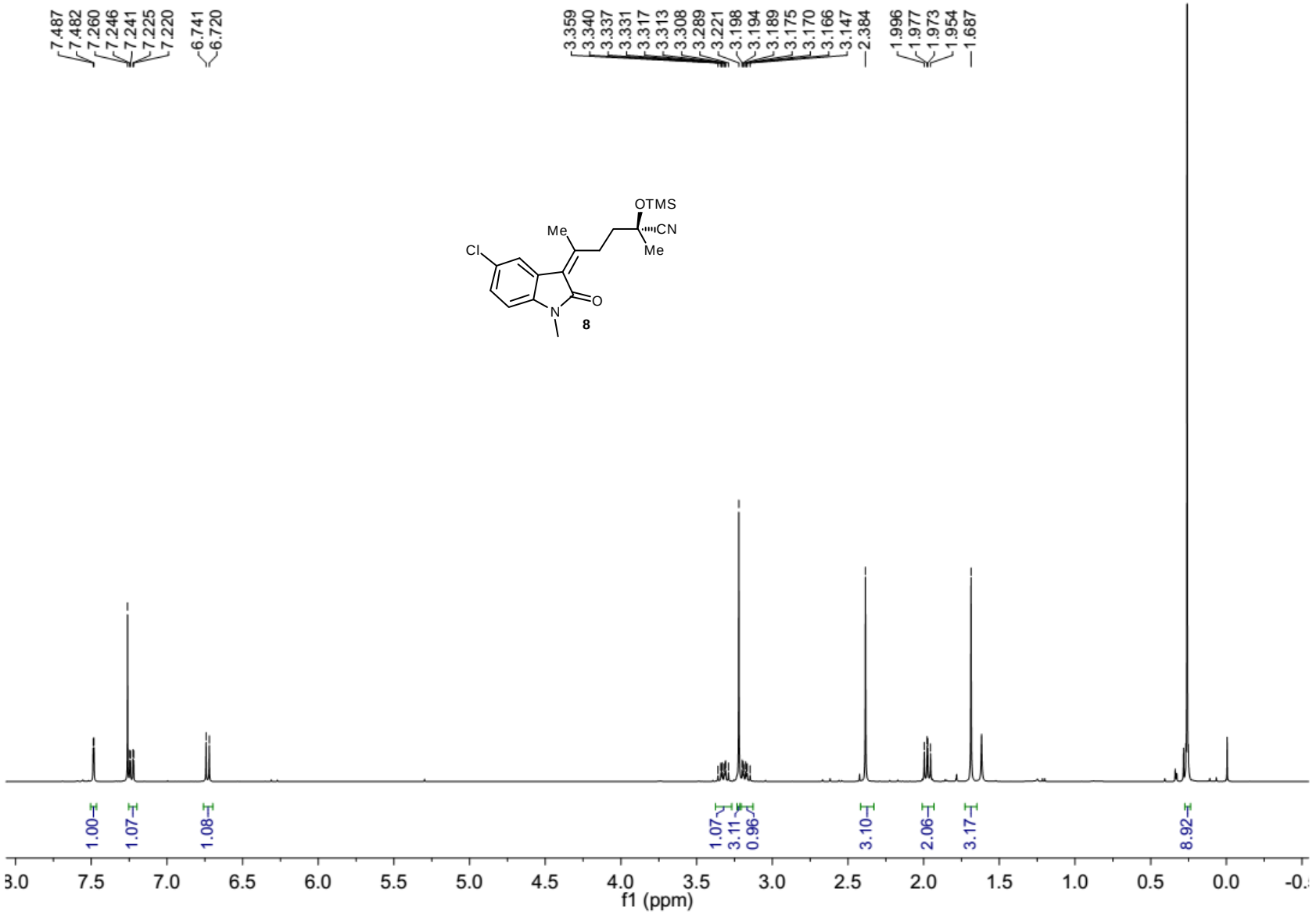
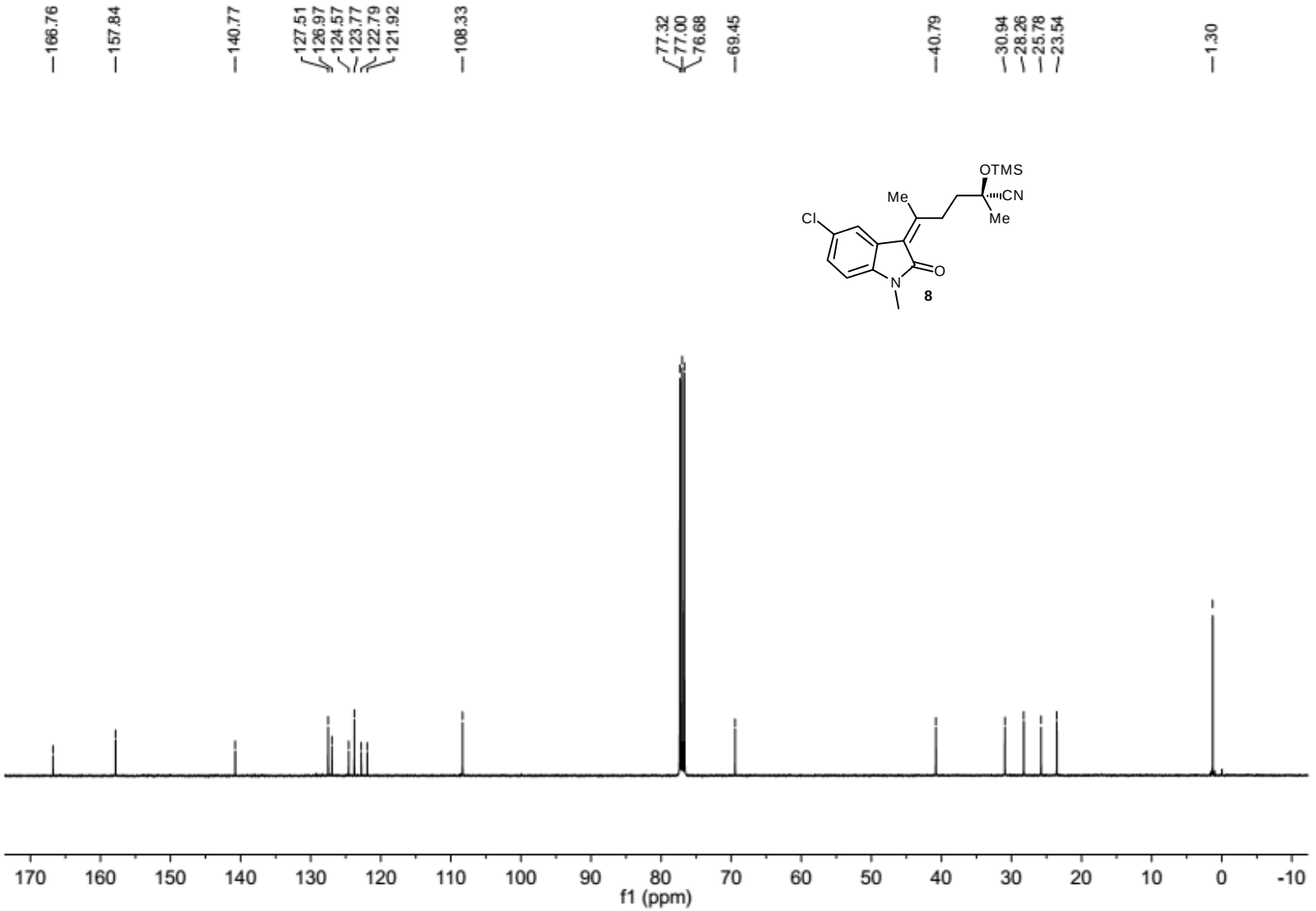
7.213
7.187
7.181
7.159
6.921
6.915
6.899
6.892
5.915
5.863
3.822
3.768
1.848
1.717
0.264
TMSO
CN
Me
*
PMP
CO2Me
7
3.45
2.08
1.03
3.04
3.01
3.00
2.97
8.97
f1 (ppm)


169.01
159.09
136.38
135.23
132.88
130.32
130.19
128.86
120.52
113.68
77.32
77.00
76.68
69.61
55.19
52.13
30.64
16.17
1.27
TMSO
CN
Me
*
PMP
CO2Me
7
f1 (ppm)


7.487
7.482
7.260
7.246
7.241
7.225
7.220
6.741
6.720
3.359
3.340
3.337
3.331
3.317
3.313
3.308
3.289
3.221
3.198
3.194
3.189
3.175
3.170
3.166
3.147
2.384
1.996
1.977
1.973
1.954
1.687
OTMS
Me
CN
Me
Cl
O
N
8
1.00
1.07
1.08
1.07
3.11
0.96
3.10
2.06
3.17
8.92
f1 (ppm)

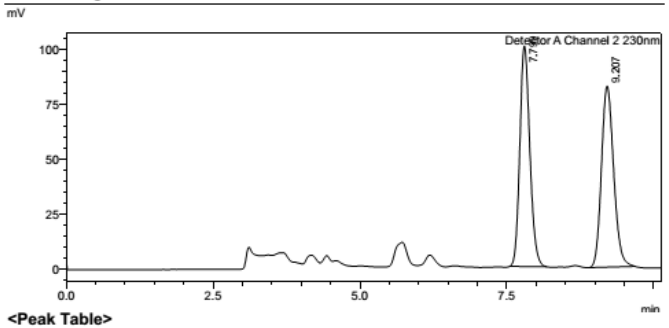

166.76
157.84
140.77
127.51
126.97
124.57
123.77
122.79
121.92
108.33
77.32
77.00
76.68
69.45
40.79
30.94
28.26
25.78
23.54
1.30
OTMS
Me
CN
Me
Cl
O
N
8
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

Analysis Report

<Sample Information>

Sample Name	: CZY-CR-87-RAC-AD-90-10		
Sample ID	:		
Data Filename	: CZY-CR-87-RAC-AD-90-10-需要.lcd		
Method Filename	: WAC-93-FANFA.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2015/12/26 22:37:04	Acquired by	: System Administrator
Date Processed	: 2015/12/26 22:48:18	Processed by	: System Administrator

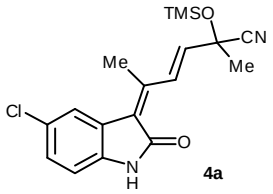
<Chromatogram>



<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.
1	7.799	1190151	100311	49.980
2	9.207	1191102	82366	50.020
Total		2381253	182678	



4a

D:\Data\caozhongyan\czy-cr\CZY-CR-87-RAC-AD-90-10-需要.lcd

Analysis Report

<Sample Information>

Sample Name	: CZY-CR-87-as-AD-90-10		
Sample ID	:		
Data Filename	: CZY-CR-87-as-AD-90-10-RE3.lcd		
Method Filename	: WAC-93-FANFA.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2015/12/26 22:56:21	Acquired by	: System Administrator
Date Processed	: 2015/12/26 23:07:23	Processed by	: System Administrator

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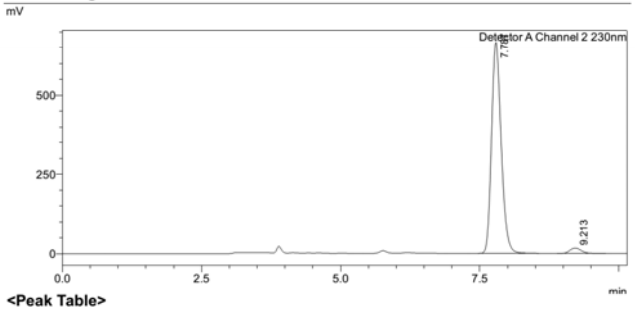
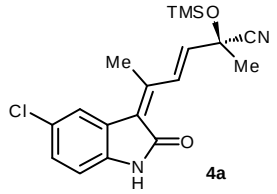


<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.
1	7.787	8072347	666375	96.989
2	9.213	250622	17303	3.011
Total		8322968	683678	



4a

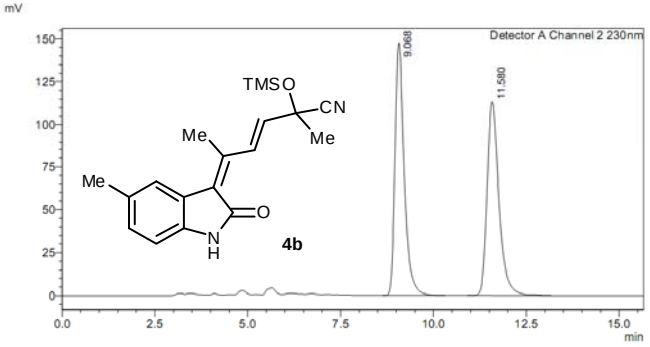
D:\Data\caozhongyan\czy-cr\CZY-CR-87-as-AD-90-10-RE3.lcd

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-27-4-RAC-ADH-90-10-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-27-4-RAC-ADH-90-10-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-22 18:43:52	Acquired by	: System Administrator
Date Processed	: 2014-6-23 11:16:24	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.068	2544783	147671	50.299			
2	11.580	2514492	113262	49.701		S	
Total		5059275	260934				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-19-4-ASY-ADH-90-10-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-19-4-ASY-ADH-90-10-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-22 19:11:38	Acquired by	: System Administrator
Date Processed	: 2014-6-22 19:26:48	Processed by	: System Administrator

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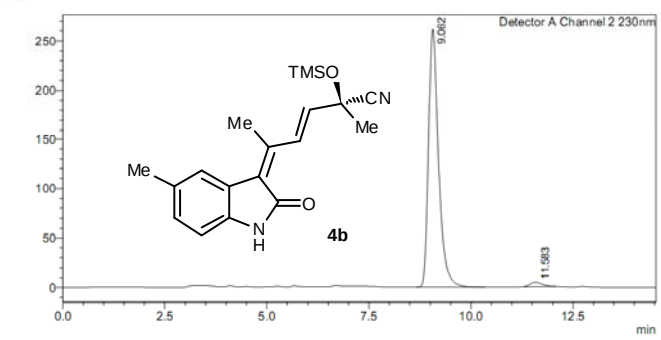


<Peak Table>

Detector A Channel 2 230nm

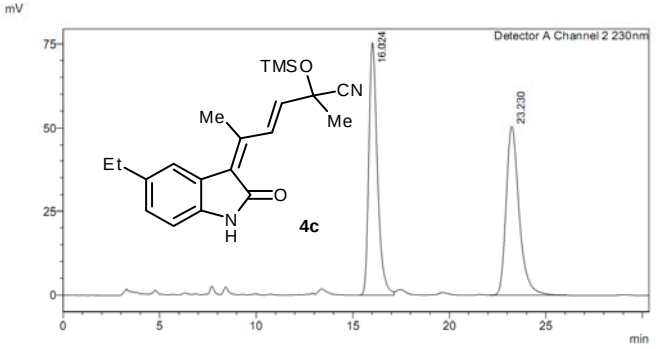
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.062	4490391	261347	97.999			
2	11.583	91701	4486	2.001		M	
Total		4582093	265834				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-19-9-RAC-ADH-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-19-9-RAC-ADH-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-24 10:18:01	Acquired by	: System Administrator
Date Processed	: 2014-6-24 11:27:43	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	16.024	2321436	75211	49.635		M	
2	23.230	2355568	50399	50.365		S	
Total		4677005	125610				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-19-9-ASY-ADH-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-19-9-ASY-ADH-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-24 11:02:15	Acquired by	: System Administrator
Date Processed	: 2014-6-24 11:29:39	Processed by	: System Administrator

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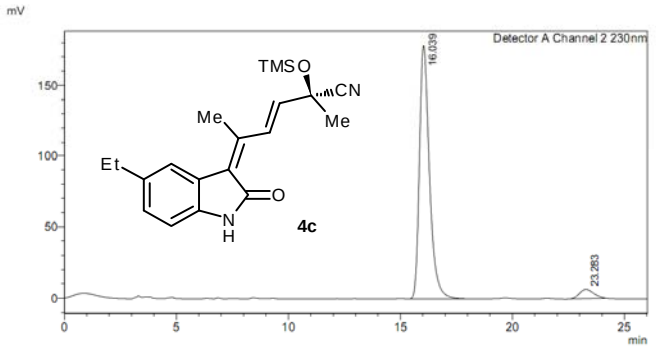


<Peak Table>

Detector A Channel 2 230nm

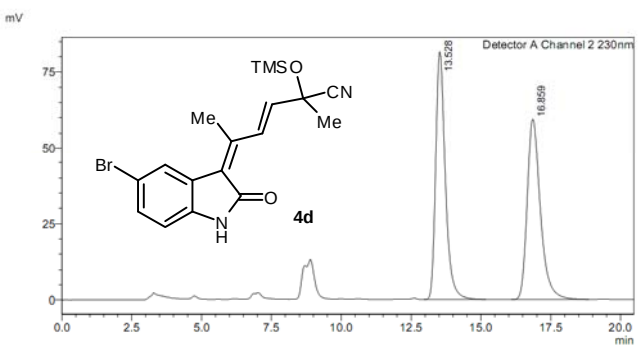
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	16.039	5560595	178572	95.027			
2	23.283	291016	6596	4.973		M	
Total		5851611	185169				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-19-3-RAC-ADH-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-19-3-RAC-ADH-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-23 18:01:28	Acquired by	: System Administrator
Date Processed	: 2014-6-23 18:22:25	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

Detector A Channel 2 230nm

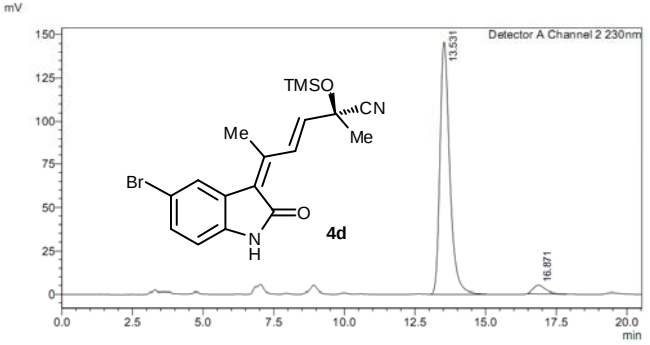
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.528	1955409	81499	50.042			
2	16.859	1952158	59297	49.958		S	
Total		3907568	140796				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-19-3-ASY-ADH-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-19-3-ASY-ADH-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-23 18:51:26	Acquired by	: System Administrator
Date Processed	: 2014-6-23 19:12:24	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

Detector A Channel 2 230nm

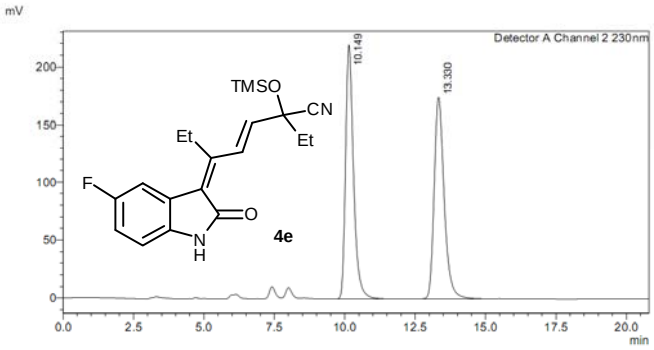
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.531	3502669	145644	96.085		M	
2	16.871	142724	4749	3.915		M	
Total		3645392	150393				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-27-8-RAC-ADH-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-27-8-RAC-ADH-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-28 15:12:05	Acquired by	: System Administrator
Date Processed	: 2014-6-28 15:44:53	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

Detector A Channel 2 230nm

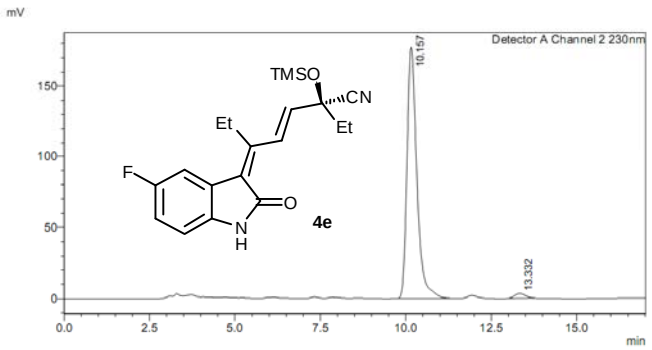
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.149	4321611	219344	49.953			
2	13.330	4329744	174463	50.047			
Total		8651355	393806				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-29-1-ASY-ADH-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-29-1-ASY-ADH-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-28 15:41:59	Acquired by	: System Administrator
Date Processed	: 2014-6-28 16:00:07	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.157	3555705	177616	98.057		M	
2	13.332	70466	3289	1.943		M	
Total		3626170	180905				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-19-8--RAC-ADH-90-10-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-19-8--RAC-ADH-90-10-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-23 10:46:27	Acquired by	: System Administrator
Date Processed	: 2014-6-23 10:57:17	Processed by	: System Administrator

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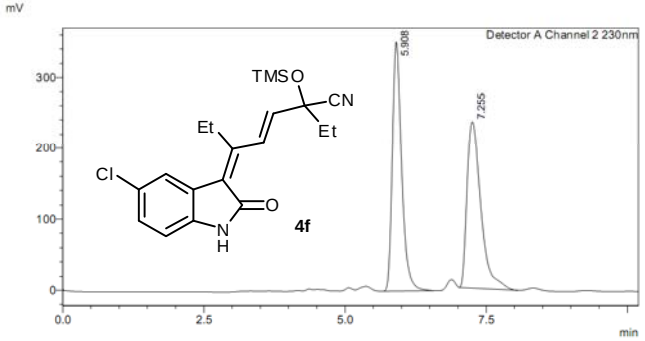

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.908	4027160	350787	50.196			
2	7.255	3995708	233859	49.804			
Total		8022868	584646				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-19-8--ASY-ADH-90-10-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-19-8--ASY-ADH-90-10-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-23 10:32:41	Acquired by	: System Administrator
Date Processed	: 2014-6-23 10:43:20	Processed by	: System Administrator

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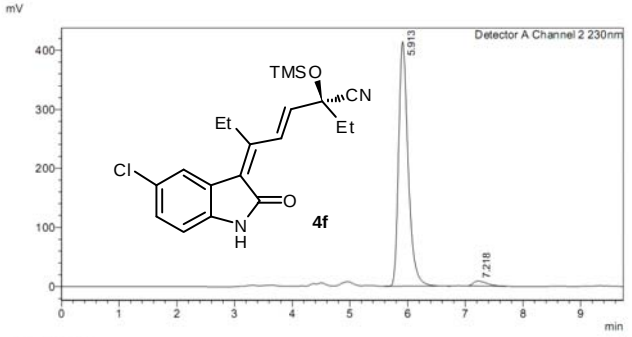

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.913	4761103	413779	97.144		M	
2	7.218	139953	8416	2.856		M	
Total		4901055	422196				

SHIMADZU LabSolutions

Analysis Report

<Sample Information>

Sample Name	: ZYL-ZJ-27-9-RAC-ADH-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-27-9-RAC-ADH-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-28 16:06:48	Acquired by	: System Administrator
Date Processed	: 2014-6-28 16:26:30	Processed by	: System Administrator

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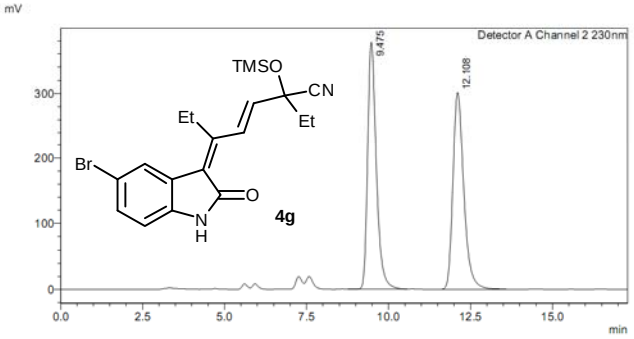

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.475	6938523	377380	49.894			
2	12.108	6967943	301550	50.106			
Total		13906465	678930				

SHIMADZU LabSolutions

Analysis Report

<Sample Information>

Sample Name	: ZYL-ZJ-29-2--ASY-ADH-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-29-2--ASY-ADH-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-6-28 17:02:09	Acquired by	: System Administrator
Date Processed	: 2014-6-28 17:19:53	Processed by	: System Administrator

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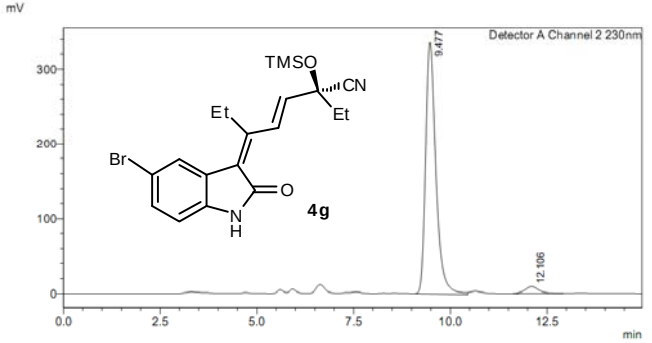

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.477	6244229	335963	96.424		M	
2	12.106	231546	9264	3.576		M	
Total		6475775	345228				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-75-RAC-IE-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-75-RAC-IE-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-8-25 16:10:56	Acquired by	: System Administrator
Date Processed	: 2014-8-25 17:00:32	Processed by	: System Administrator

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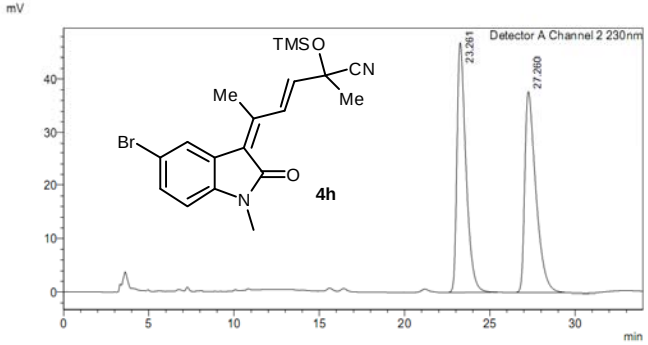

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	23.261	1733766	46964	50.130		S	
2	27.260	1724799	37836	49.870		S	
Total		3458565	84800				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-75-ASY---IE-95-5-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-75-ASY---IE-95-5-205-230-1-.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-8-25 18:11:10	Acquired by	: System Administrator
Date Processed	: 2014-8-25 18:51:54	Processed by	: System Administrator

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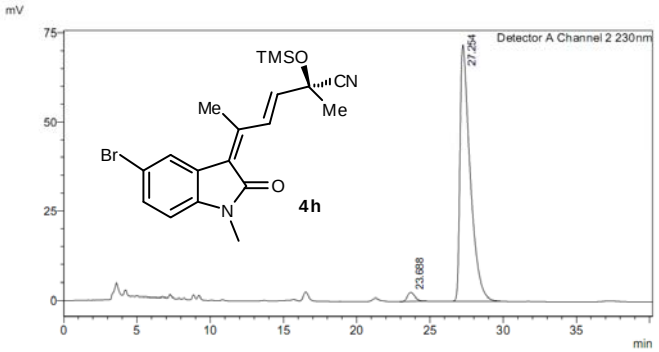

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	23.688	88061	2448	2.482			
2	27.254	3459372	71643	97.518		V	
Total		3547433	74091				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-79-RAC-IE-90-10-205-230-1		
Sample ID	:		
Data Filename	: ZYL-ZJ-79-RAC-IE-90-10-205-230-1.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-8-27 16:38:25	Acquired by	: System Administrator
Date Processed	: 2014-8-27 17:02:54	Processed by	: System Administrator

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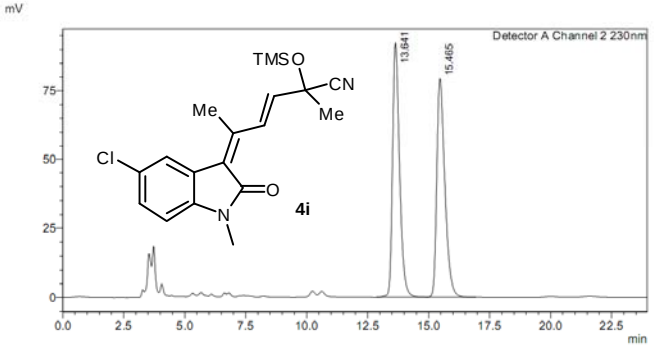

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.641	1845979	92153	50.220			
2	15.465	1829776	79304	49.780		SV	
Total		3675755	171458				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-79-ASY-IE-90-10-205-230-1		
Sample ID	:		
Data Filename	: ZYL-ZJ-79-ASY-IE-90-10-205-230-1.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-8-27 16:19:04	Acquired by	: System Administrator
Date Processed	: 2014-8-27 17:03:47	Processed by	: System Administrator

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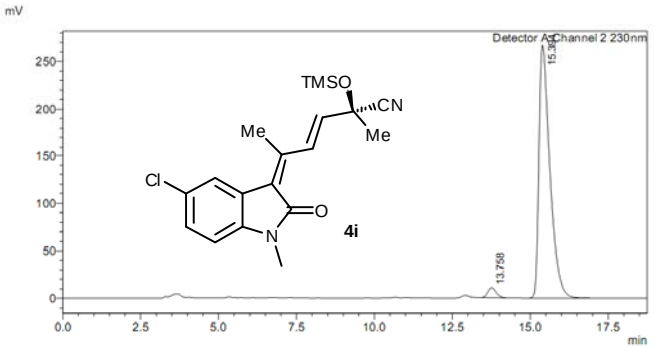

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.758	196312	10636	2.922		M	
2	15.394	6522235	266913	97.078		S	
Total		6718547	277549				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-95--RAC-IE-85-15-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-95--RAC-IE-85-15-205-230-1-4.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-9-1 22:12:39	Acquired by	: System Administrator
Date Processed	: 2014-9-1 22:47:37	Processed by	: System Administrator

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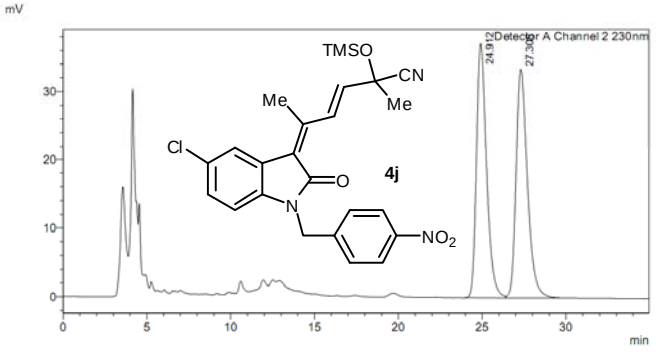

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.912	1559108	37154	49.854			
2	27.305	1568224	33433	50.146		SV	
Total		3127332	70587				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-95-ASY-IE-85-15-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-95-ASY-IE-85-15-205-230-1-3.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-9-1 21:36:26	Acquired by	: System Administrator
Date Processed	: 2014-9-1 22:11:15	Processed by	: System Administrator

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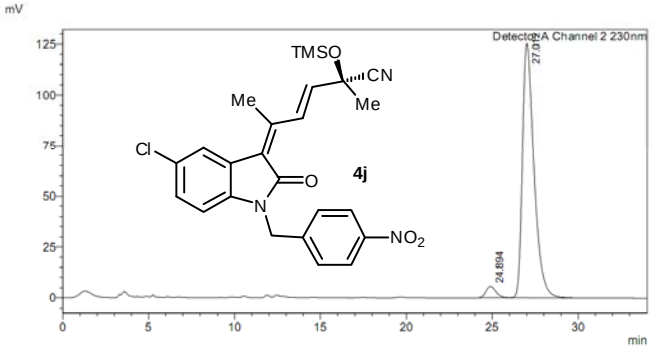

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.894	230621	5769	3.784		M	
2	27.012	5864489	125199	96.216		S	
Total		6095110	130968				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-97-RAC-IE-90-10-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-97-RAC-IE-90-10-205-230-1-5.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-9-2 9:08:13	Acquired by	: System Administrator
Date Processed	: 2014-9-2 9:30:26	Processed by	: System Administrator

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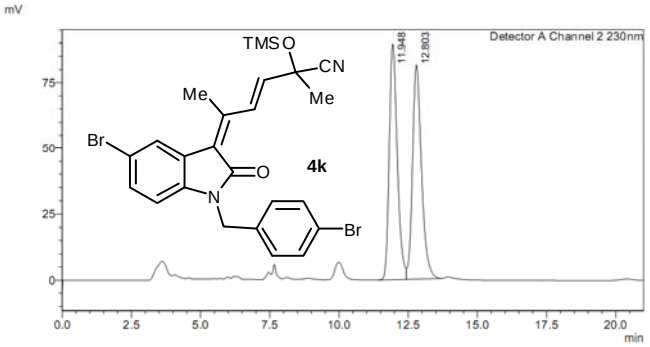

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.948	1806720	89446	49.672			
2	12.803	1830604	81087	50.328		V	
Total		3637324	170534				

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ZYL-ZJ-97-ASY-IE-90-10-205-230-1-		
Sample ID	:		
Data Filename	: ZYL-ZJ-97-ASY-IE-90-10-205-230-1-6.lcd		
Method Filename	: ZYL-A.lcm		
Batch Filename	:		
Vial #	: 0-0	Sample Type	: Unknown
Injection Volume	: 20 uL		
Date Acquired	: 2014-9-2 9:31:52	Acquired by	: System Administrator
Date Processed	: 2014-9-2 9:49:19	Processed by	: System Administrator

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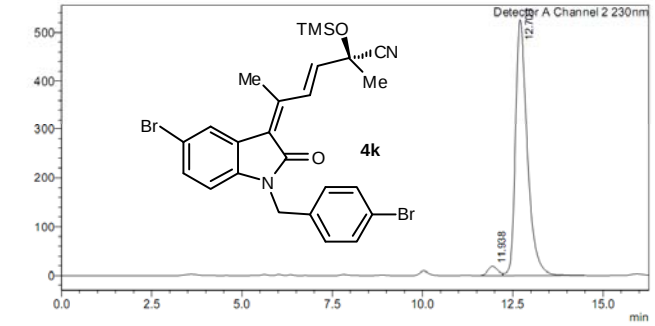


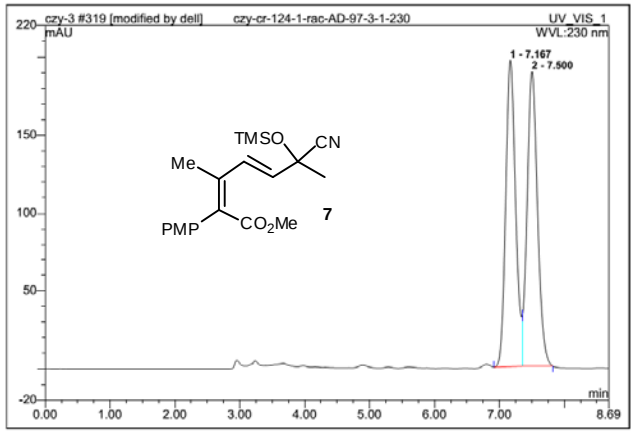
<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.938	352284	18543	2.860		M	
2	12.703	11964055	525704	97.140		V M	
Total		12316339	544246				

319 czy-cr-124-1-rac-AD-97-3-1-230

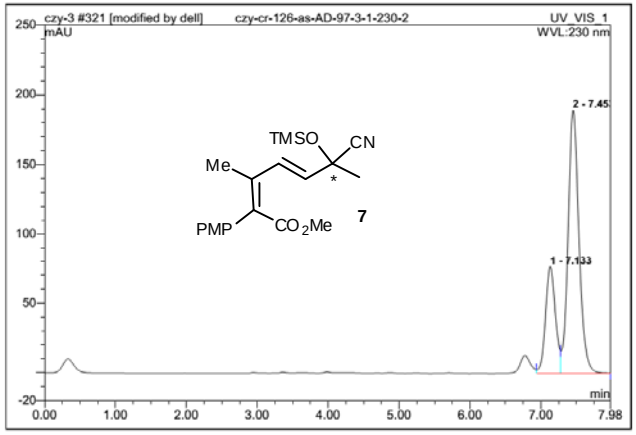
Sample Name:	**czy-cr-124-1-rac-AD-97-3-1-230**	*Injection Volume:*	**20.0**
Vial Number:	**426**	*Channel:*	**UV_VIS_1**
Sample Type:	**standard**	*Wavelength:*	**230**
Control Program:	**czy**	*Bandwidth:*	**n.a.**
Quantif. Method:	**czy**	*Dilution Factor:*	**1.0000**
Recording Time:	**2015-12-22 14:09**	*Sample Weight:*	**1.0000**
Run Time (min):	**8.69**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.17	n.a.	196.429	34.658	49.67	n.a.	BM *
2	7.50	n.a.	188.856	35.122	50.33	n.a.	MB*
Total:			385.285	69.779	100.00	0.000	

321 czy-cr-126-as-AD-97-3-1-230-2

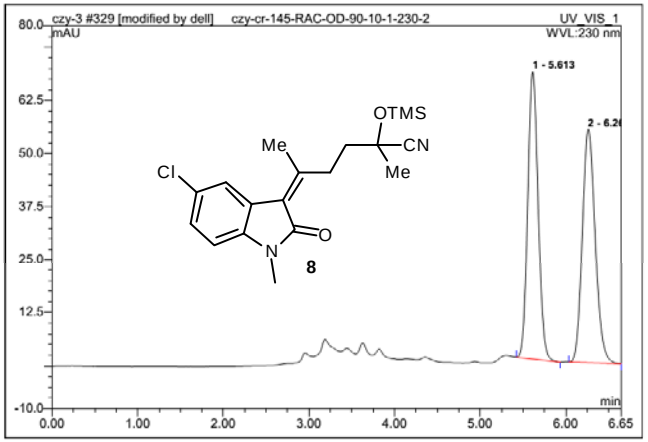
Sample Name:	**czy-cr-126-as-AD-97-3-1-230-2**	*Injection Volume:*	**20.0**
Vial Number:	**428**	*Channel:*	**UV_VIS_1**
Sample Type:	**standard**	*Wavelength:*	**230**
Control Program:	**czy**	*Bandwidth:*	**n.a.**
Quantif. Method:	**czy**	*Dilution Factor:*	**1.0000**
Recording Time:	**2015-12-22 14:31**	*Sample Weight:*	**1.0000**
Run Time (min):	**7.98**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.13	n.a.	76.480	12.417	26.97	n.a.	M *
2	7.45	n.a.	189.564	33.620	73.03	n.a.	MB*
Total:			266.044	46.038	100.00	0.000	

329 czy-cr-145-RAC-OD-90-10-1-230-2

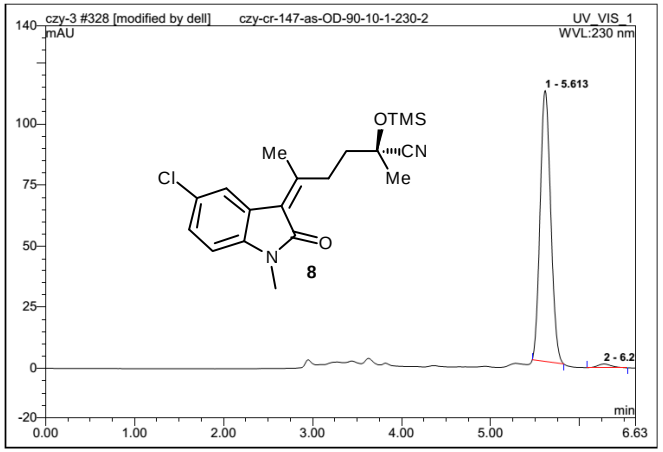
Sample Name:	**czy-cr-145-RAC-OD-90-10-1-230-2**	*Injection Volume:*	**20.0**
Vial Number:	**436**	*Channel:*	**UV_VIS_1**
Sample Type:	**standard**	*Wavelength:*	**230**
Control Program:	**czy**	*Bandwidth:*	**n.a.**
Quantif. Method:	**czy**	*Dilution Factor:*	**1.0000**
Recording Time:	**2016-1-6 20:03**	*Sample Weight:*	**1.0000**
Run Time (min):	**6.65**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	5.61	n.a.	67.616	9.482	49.81	n.a.	BMB*
2	6.26	n.a.	54.853	9.556	50.19	n.a.	BMB*
Total:			122.469	19.038	100.00	0.000	

328 czy-cr-147-as-OD-90-10-1-230-2

Sample Name:	**czy-cr-147-as-OD-90-10-1-230-2**	*Injection Volume:*	**20.0**
Vial Number:	**435**	*Channel:*	**UV_VIS_1**
Sample Type:	**standard**	*Wavelength:*	**230**
Control Program:	**czy**	*Bandwidth:*	**n.a.**
Quantif. Method:	**czy**	*Dilution Factor:*	**1.0000**
Recording Time:	**2016-1-6 19:52**	*Sample Weight:*	**1.0000**
Run Time (min):	**6.63**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	5.61	n.a.	110.841	15.284	98.07	n.a.	BMB*
2	6.27	n.a.	1.578	0.300	1.93	n.a.	MB*
Total:			112.419	15.584	100.00	0.000	