Electronic Supplementary Material (ESI) for Chemical Communications

Catalytic [2 + 2] and [3 + 2] cycloaddition reactions of allenonates with cyclic ketimines

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Part I General Information

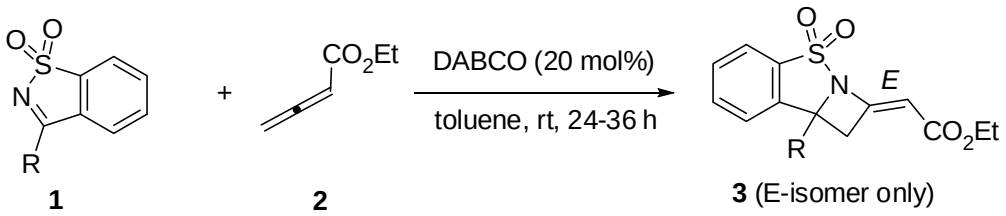
Unless otherwise indicated, all reactions were carried out under N_2 atmosphere in oven-dried glassware with magnetic stirring. Anhydrous THF and toluene were distilled from sodium and benzophenone. Anhydrous CH_2Cl_2 was distilled from CaH_2. Column chromatograph was performed on silica gel 200~300 mesh. All ^1H NMR (300 MHz), ^{13}C NMR (75 MHz) spectra were recorded on a Bruker-DMX 300 spectrometer 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). Splitting patterns that could not be interpreted or easily visualized were designated as multiplet (m) or broad (br). Infrared spectra were recorded on a JASCO FT/IR-480 spectrophotometer and reported as wave number (cm^{-1}).

Part II Experimental part

1. Materials

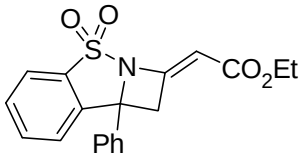
Both cyclic ketimines[1] and ethyl 2,3-butadienoate[2] were prepared according to literature methods.

2. DABCO-catalyzed [2 + 2] cycloaddition (Table 2).



1 **2** **3** (E-isomer only)

To a stirred solution of cyclic ketimines (0.2 mmol) and DABCO (4.5 mg, 0.04 mmol) in toluene (2 mL) was added ethyl 2,3-butadienoate (48.8 mg, 0.4 mmol). The reaction mixture was stirred for 24-36 h at room temperature until the full consumption of the cyclic ketimines. The reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc, typically 10:1-5:1) to furnish the corresponding [2+2] cycloadduct **3**.

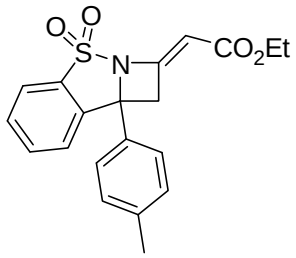


3a

(*E*)-ethyl 2-(4,4-dioxido-8b-phenyl-1H-azeto[1,2-b]benzo[d]isothiazol-2-(8bH)-ylidene)acetate

Reaction time: 24 h; Yield: 61 mg, 86%; white solid; mp 154 ^{o}C; R_f = 0.25 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.77 (d, *J* = 7.7 Hz, 1H), 7.71-7.51 (m, 4H), 7.49-7.29 (m, 4H), 5.94 (t, *J* = 2.3 Hz, 1H), 4.11 (q, *J* = 7.2 Hz, 2H), 4.04 (dd, *J* = 17.1, 2.3 Hz, 1H), 3.76 (dd, *J* = 17.1, 2.3 Hz, 1H), 1.23 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 166.1, 155.9, 143.0, 139.1, 136.2,

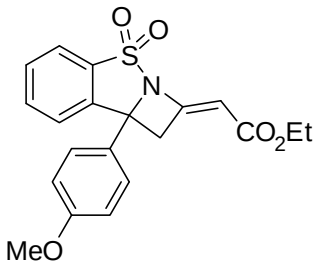
134.2, 130.1, 129.0, 128.6, 125.3, 124.9, 122.1, 104.8, 76.9, 60.1, 43.2, 14.3. **IR (KBr):** 1713, 1671, 1449, 1336, 1181, 1111, 763, 573, 526. **HRMS (EI)** calcd for $C_{19}H_{17}NO_4S$ $[M]^+$ 355.0878, found 355.0883.



3b

(*E*)-ethyl 2-(4,4-dioxido-8b-(p-tolyl)-1H-azeto[1,2-b]benzo[d]isothiazol-2(8bH)-ylidene)acetate

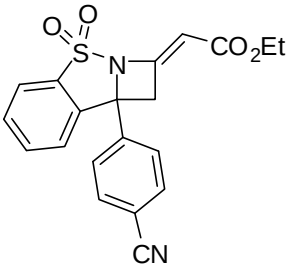
Reaction time: 36 h; Yield: 59 mg, 80%; white solid; mp 140 ^oC; R_f = 0.3 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.77 (d, *J* = 7.8 Hz, 1H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.1 Hz, 3H), 7.21 (d, *J* = 8.0 Hz, 2H), 5.94 (d, *J* = 2.1 Hz, 1H), 4.11 (q, *J* = 7.1 Hz, 2H), 4.03 (dd, *J* = 17.1, 2.1 Hz, 1H), 3.73 (dd, *J* = 17.1, 2.1 Hz, 1H), 2.34 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 166.2, 156.0, 143.2, 138.6, 136.3, 136.0, 134.2, 130.0, 129.6, 125.3, 124.9, 122.1, 104.7, 76.9, 60.1, 43.0, 21.1, 14.3. **IR (KBr):** 1713, 1671, 1336, 1182, 1108, 1042, 821, 760, 584, 526. **HRMS (EI)** calcd for $C_{20}H_{19}NO_4S$ $[M]^+$ 369.1035, found 369.1039.



3c

(*E*)-ethyl 2-(8b-(4-methoxyphenyl)-4,4-dioxido-1H-azeto[1,2-b]benzo[d]isothiazol -2(8bH)-ylidene)acetate

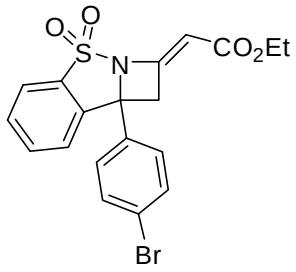
Reaction time: 36 h; Yield: 35 mg, 45%; white solid; mp 140 ^{o}C; R_f = 0.33 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.79 (d, *J* = 7.8 Hz, 1H), 7.65-7.56 (m, 2H), 7.47 (d, *J* = 8.8 Hz, 2H), 7.41 (d, *J* = 7.7 Hz, 1H), 6.92 (d, *J* = 8.8 Hz, 2H), 5.93(t, *J* = 2.4 HZ, 1H), 4.11 (q, *J* = 7.2 Hz, 2H), 4.04 (dd, *J* = 17.2, 2.4 Hz, 1H), 3.80 (s, 3H), 3.70 (dd, *J* = 17.1, 2.3 Hz, 1H), 1.24 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 166.3, 159.9, 156.0, 143.4, 136.5, 134.2, 130.9, 130.0, 127.1, 125.0, 122.2, 114.4, 104.7, 76.9, 60.1, 55.4, 43.0, 14.3. **IR (KBr):** 1713, 1670, 1514, 1336, 1254, 1180, 1108, 833, 530. **HRMS (EI)** calcd for $C_{20}H_{19}NO_5S$ $[M]^+$ 385.0948, found 385.0988.



3d

(*E*)-ethyl 2-(8b-(4-cyanophenyl)-4,4-dioxido-1H-azeto[1,2-b]benzo[d]isothiazol-2(8bH)-ylidene)acetate

Reaction time: 24 h; Yield: 53 mg, 70%; white solid; mp 146 ^{o}C; R_f = 0.2 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.82 (d, *J* = 7.6 Hz, 1H), 7.78-7.58 (m, 6H), 7.46 (d, *J* = 7.7 Hz, 1H), 5.96 (t, *J* = 2.2 Hz, 1H), 4.12 (q, *J* = 7.1 Hz, 2H), 3.99 (dd, *J* = 17.0, 2.2 Hz, 1H), 3.81 (dd, *J* = 17.0, 2.3 Hz, 1H), 1.24 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 164.9, 153.8, 143.1, 140.6, 135.3, 133.6, 131.9, 129.6, 125.2, 123.6, 121.5, 117.1, 111.8, 104.3, 74.9, 59.3, 42.2, 13.2. **IR (KBr):** 2230, 1713, 1672, 1353, 1336, 1182, 1108, 843, 761, 578. **HRMS (EI)** calcd for $C_{20}H_{16}N_2O_4S$ $[M]^+$ 380.0831, found 380.0836.

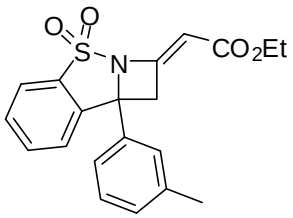


3e

(*E*)-ethyl 2-(8b-(4-bromophenyl)-4,4-dioxido-1H-azeto[1,2-b]benzo[d]isothiazol-2(8bH)-ylidene)acetate

Reaction time: 24 h; Yield: 81 mg, 93%; white solid; mp 140 ^{o}C; R_f = 0.3 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.79 (d, *J* = 7.5 Hz, 1H), 7.67 (dd, *J* = 10.7, 4.2 Hz, 1H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.54 (d, *J* = 8.6 Hz, 2H), 7.49-7.39 (m, 3H), 5.94 (t, *J* = 2.1 Hz, 1H), 4.11 (q, *J* = 7.1 Hz,. 2H), 3.99 (dd, *J* = 17.0, 2.2 Hz, 1H), 3.75 (dd, *J* = 17.0, 2.2 Hz, 1H), 1.24 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 166.0, 155.4, 142.4, 138.1, 136.3, 134.4, 132.2, 130.3, 127.1, 124.7, 123.0, 122.3, 105.0, 76.3, 60.2, 43.1, 14.3. **IR (KBr):** 1713, 1675, 1336,

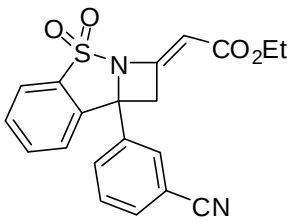
1181, 1108, 994, 835, 759, 575. **HRMS (EI)** calcd for $C_{19}H_{16}BrNO_4S$ $[M]^+$ 432.9983, 434.9963, found 432.9987, 434.9970.



3f

(*E*)-ethyl 2-(4,4-dioxido-8b-(m-tolyl)-1H-azeto[1,2-b]benzo[d]isothiazol-2(8bH)-ylidene)acetate

Reaction time: 36 h; Yield: 65 mg, 88%; white solid; mp 189 ^oC; R_f = 0.33 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.79 (d, *J* = 7.8 Hz, 1H), 7.65 (td, *J* = 7.6, 1.1 Hz, 1H), 7.57 (dd, *J* = 11.2, 3.8 Hz, 1H), 7.45 (d, *J* = 7.7 Hz, 1H), 7.37-7.30 (m, 3H), 7.16 (d, *J* = 7.3 Hz, 1H), 5.93 (t, *J* = 2.3 Hz, 1H), 4.11 (q, *J* = 7.1 Hz, 2H), 4.03 (dd, *J* = 17.1, 2.3 Hz, 1H), 3.74 (dd, *J* = 17.1, 2.4 Hz, 1H), 2.37 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 166.2, 156.0, 143.2, 139.1, 138.9, 136.3, 134.2, 130.1, 129.5, 128.9, 126.0, 124.9, 122.4, 122.2, 104.8, 77.0, 60.1, 43.3, 21.6, 14.3. **IR (KBr):** 1713, 1671, 1336, 1182, 1112, 1039, 843, 761, 575. **HRMS (EI)** calcd for $C_{20}H_{19}NO_4S$ $[M]^+$ 369.1035, found 369.1040.

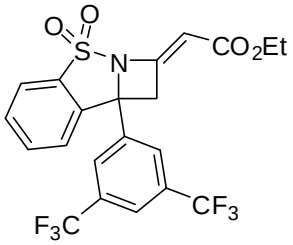


3g

(*E*)-ethyl 2-(8b-(3-cyanophenyl)-4,4-dioxido-1H-azeto[1,2-b]benzo[d]isothiazol-

2(8bH)-ylidene)acetate

Reaction time: 24 h; Yield: 61 mg, 80%; white solid; mp 167 ^{o}C; R_f = 0.2 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, CDCl$_3$):** δ 7.90-7.87 (m, 2H), 7.83 (d, *J* = 7.7 Hz, 1H), 7.77-7.53 (m, 4H), 7.48 (d, *J* = 7.8 Hz, 1H), 5.96 (t, *J* = 2.3 Hz, 1H), 4.12 (q, *J* = 7.1 Hz, 1H), 4.00 (dd, *J* = 17.0, 2.3 Hz, 1H), 3.81 (dd, *J* = 17.0, 2.3 Hz, 1H), 1.24 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, CDCl$_3$):** δ 165.9, 154.80, 141.6, 140.8, 136.3, 134.6, 132.3, 130.6, 130.1, 129.9, 128.86, 124.7, 122.4, 118.1, 113.3, 105.3, 75.5, 60.3, 43.3, 14.2. **IR (KBr):** 2231, 1713, 1671, 1353, 1333, 1183, 1112, 761, 729. **HRMS (EI)** calcd for $C_{20}H_{16}N_2O_4S$ $[M]^+$ 380.0831, found 380.0836.

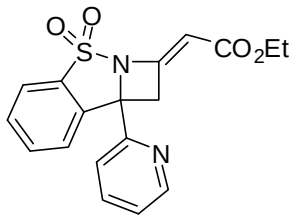


3h

(*E*)-ethyl 2-(8b-(3,5-bis(trifluoromethyl)phenyl)-4,4-dioxido-1H-azeto [1,2-b]benzo[d]isothiazol-2(8bH)-ylidene)acetate

Reaction time: 24 h; Yield: 93 mg, 95%; white solid; mp 136 ^{o}C; R_f = 0.33 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, CDCl$_3$):** δ 8.11 (s, 2H), 7.91-7.83 (m, 2H), 7.75 (t, *J* = 7.3 Hz, 1H), 7.66 (t, *J* = 7.5 Hz, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 5.99 (s, 1H), 4.13 (q, *J* = 7.1 Hz, 2H), 3.95 (dd, *J* = 45.2, 17.0 Hz, 1H), 1.25 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, CDCl$_3$):** δ 165.8, 154.4, 142.0, 141.1, 136.5, 134.8, 134.0, 132.7 (q, $J_{C\text{-}F}$ = 37.5 Hz), 130.9, 125.7, 124.6, 122.9 (q, $J_{C\text{-}F}$ = 271.5 Hz),

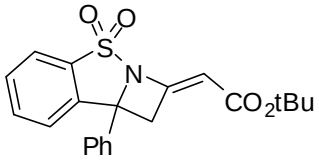
122.6, 105.5, 75.2, 60.4, 43.6, 14.2. **IR (KBr):** 1714, 1674, 1337, 1280, 1183, 1135, 901, 705, 681, 572. **HRMS (EI)** calcd for $C_{21}H_{15}F_6NO_4S$ $[M]^+$ 491.0626, found 491.0633.



3i

(*E*)-ethyl 2-(4,4-dioxido-8b-(pyridin-2-yl)-1H-azeto[1,2-b]benzo[d]isothiazol-2(8bH)-ylidene)acetate

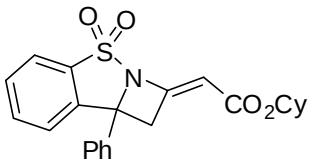
Reaction time: 24 h; Yield: 68 mg, 96%; orange solid; mp 132 ^oC; R_f = 0.2 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 8.66 (d, *J* = 4.8 Hz, 1H), 7.88 (d, *J* = 7.7 Hz, 1H), 7.81-7.52 (m, 5H), 7.37-7.16 (m, 1H), 5.96 (t, *J* = 2.1 Hz, 1H), 4.22-4.00 (m, 3H), 3.75 (dd, *J* = 17.5, 2.2 Hz, 1H), 1.23 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 165.9, 157.7, 156.2, 149.6, 141.6, 137.3, 136.2, 134.2, 130.3, 125.9, 123.2, 121.8, 120.2, 105.0, 76.0, 60.1, 42.9, 14.3. **IR (KBr):** 1714, 1671, 1448, 1353, 1336, 1182, 1112, 1093, 842, 753, 574, 527. **HRMS (EI)** calcd for $C_{18}H_{16}N_2O_4S$ $[M]^+$ 356.0831, found 356.0835.



3j

(*E*)-tert-butyl 2-(4,4-dioxido-8b-phenyl-1H-azeto[1,2-b]benzo[d]isothiazol-2(8bH)-ylidene)acetate

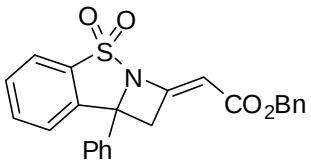
Reaction time: 1 d; Yield: 73 mg, 94%; white solid; mp 160 ^{o}C; R_f = 0.5 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.77 (d, *J* = 7.7 Hz, 1H), 7.69-7.50 (m, 4H), 7.49-7.31 (m, 4H), 5.85 (t, *J* = 2.3 Hz, 1H), 4.01 (dd, *J* = 17.0, 2.3 Hz, 1H), 3.73 (dd, *J* = 17.0, 2.3 Hz, 1H), 1.42 (s, 9H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 165.6, 154.8, 143.2, 139.3, 136.3, 134.3, 130.1, 129.1, 128.7, 125.4, 124.9, 122.2, 106.7, 80.6, 77.0, 43.3, 28.3. **IR (KBr):** 1700, 1670, 1381, 1270, 728, 573. **HRMS (EI)** calcd for $C_{21}H_{21}NO_4S$ $[M]^+$ 383.1191, found 383.1196.



3k

(*E*)-cyclohexyl 2-(4,4-dioxido-8b-phenyl-1H-azeto[1,2-b]benzo[d] isothiazol-2(8bH)-ylidene)acetate

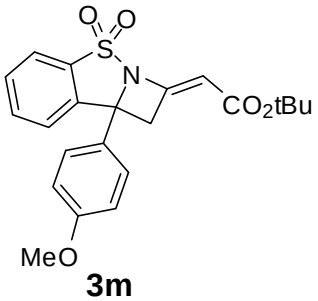
Reaction time: 1 d; Yield: 80 mg, 98%; white solid; mp 130 ^{o}C; R_f = 0.5 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.78 (d, *J* = 7.8 Hz, 1H), 7.68-7.51 (m, 4H), 7.49-7.31 (m, 4H), 5.93 (t, *J* = 2.3 Hz, 1H), 4.76-4.72 (m, 1H), 4.03 (dd, *J* = 17.0, 2.3 Hz, 1H), 3.75 (dd, *J* = 17.0, 2.3 Hz, 1H), 1.90-1.62 (m, 4H), 1.52-1.20 (m, 6H). ^{13}C **NMR (75 MHz, $CDCl_3$):** δ 165.7, 155.6, 143.1, 139.2, 136.3, 134.3, 130.2, 129.1, 128.7, 125.4, 124.9, 122.2, 105.5, 76.9, 72.6, 43.4, 31.8, 31.8, 25.4, 23.8. **IR (KBr):** 1852, 1687, 1351, 1226, 768, 503. **HRMS (EI)** calcd for $C_{23}H_{23}NO_4S$ $[M]^+$ 409.1348, found 409.1352.



3l

(*E*)-benzyl 2-(4,4-dioxido-8b-phenyl-1H-azeto[1,2-b]benzo[d] isothiazol-2(8bH)-ylidene)acetate

Reaction time: 1 d; Yield: 76 mg, 91%; white solid; mp 146 ^{o}C; R_f = 0.4 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.74 (d, *J* = 7.8 Hz, 1H), 7.66-7.47 (m, 4H), 7.46-7.35 (m, 4H), 7.36-7.25 (m, 5H), 6.00 (t, *J* = 2.2 Hz, 1H), 5.09 (s, 2H), 4.02 (dd, *J* = 17.1, 2.3 Hz, 1H), 3.74 (dd, *J* = 17.1, 2.3 Hz, 1H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 166.0, 156.6, 143.0, 139.1, 136.3, 136.0, 134.3, 130.2, 129.1, 128.7, 128.6, 128.3, 125.4, 124.9, 122.3, 104.4, 76.9, 66.0, 43.3. **IR (KBr):** 1705, 1670, 1344, 1108, 700, 572, 571. **HRMS (EI)** calcd for $C_{24}H_{19}NO_4S$ $[M]^+$ 417.1035, found 417.1039.

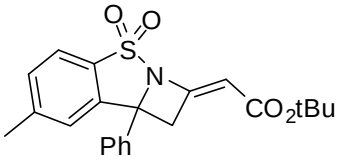


3m

(*E*)-tert-butyl 2-(8b-(4-methoxyphenyl)-4,4-dioxido-1H-azeto[1,2-b]benzo [d]isothiazol-2(8bH)-ylidene)acetate

Reaction time: 5 d; Yield: 51 mg, 61%; white solid; mp 191 ^{o}C; R_f = 0.4 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.79 (d, *J* = 7.9

Hz, 1H), 7.72-7.51 (m, 2H), 7.50-7.36 (m, 3H), 7.02-6.81 (m, 2H), 5.84 (t, *J* = 2.3 Hz, 1H), 4.01 (dd, *J* = 17.0, 2.3 Hz, 1H), 3.80 (s, 3H), 3.67 (dd, *J* = 17.0, 2.3 Hz, 1H), 1.43 (s, 9H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 165.7, 160.0, 155.0, 143.6, 136.6, 134.3, 131.2, 130.1, 127.2, 125.0, 122.3, 114.4, 106.7, 80.6, 77.0, 55.5, 43.0, 28.4. **IR (KBr):** 1707, 1670, 1343, 1132, 762, 553. **HRMS (EI)** calcd for $C_{22}H_{23}NO_5S$ $[M]^+$ 413.1297, found 413.1303.

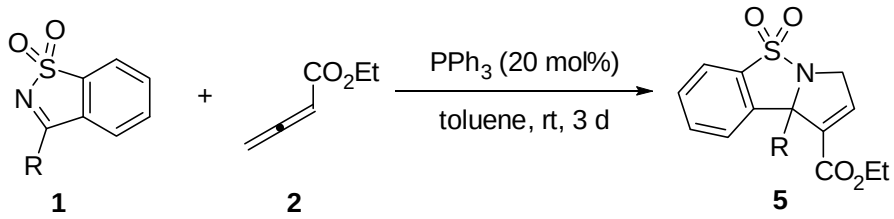


3a'

(*E*)-tert-butyl 2-(7-methyl-4,4-dioxido-8b-phenyl-1H-azeto[1,2-b]benzo[d] isothiazol-2(8bH)-ylidene)acetate

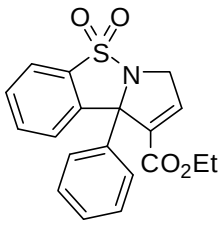
Reaction time: 1 d; Yield: 70 mg, 89%; white solid; mp 157 ^{o}C; R_f = 0.6 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.65 (d, *J* = 8.0 Hz, 1H), 7.61-7.55 (m, 2H), 7.47-7.29 (m, 4H), 7.20 (s, 1H), 5.84 (t, *J* = 2.3 Hz, 1H), 3.99 (dd, *J* = 17.0, 2.3 Hz, 1H), 3.72 (dd, *J* = 17.0, 2.3 Hz, 1H), 2.41 (s, 3H), 1.42 (s, 9H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 165.6, 155.1, 145.6, 143.6, 139.5, 133.8, 131.1, 129.0, 128.6, 125.4, 125.0, 121.9, 106.6, 80.5, 76.8, 43.2, 28.3, 21.9. **IR (KBr):** 1707, 1670, 1343, 1132, 762, 553. **HRMS (EI)** calcd for $C_{22}H_{23}NO_4S$ $[M]^+$ 397.1348, found 397.1353.

3. PPh_3-catalyzed [3 + 2] cycloaddition (Table 3).



1 **2** **5**

To a stirred solution of cyclic ketimines (0.2 mmol) and PPh_3 (10.5 mg, 0.04 mmol) in toluene (2 mL) was added ethyl 2,3-butadienoate (48.8 mg, 0.4 mmol). The reaction mixture was stirred for 3 d at room temperature until the full consumption of the cyclic ketimines. The reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleumether/EtOAc as the eluent, typically 10:1-5:1) to furnish the corresponding [3+2] cycloadduct **5**.

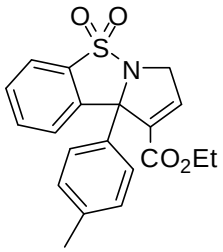


5a

ethyl 9b-phenyl-3,9b-dihydrobenzo[d]pyrrolo[1,2-b]isothiazole-1-carboxylate 5,5-dioxide

Reaction time: 3 d; Yield: 75 mg, 75%; white solid; mp 156 ^{o}C; R_f = 0.4 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 8.00 (dd, *J* = 6.9, 1.6 Hz, 1H), 7.86-7.80 (m, 1H), 7.61 (m, 2H), 7.30 (dt, *J* = 4.5, 2.4 Hz, 3H), 7.25-7.18 (m, 2H), 7.10 (t, *J* = 2.2 Hz, 1H), 4.79 (dd, *J* = 18.4, 2.5 Hz, 1H), 4.26 (dd, *J* = 15.8, 2.6 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75**

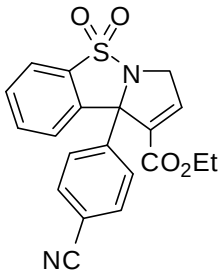
MHz, $CDCl_3$): δ 162.2, 141.0, 140.8, 140.1, 136.0, 135.8, 133.1, 130.0, 128.6, 128.4, 127.9, 126.8, 121.4, 82.8, 61.3, 52.6, 14.1. **IR (KBr):** 1717, 1315, 1175, 1315, 1175, 1112, 762, 697, 572. **HRMS (EI)** calcd for $C_{19}H_{17}NO_4S$ $[M]^+$ 355.0878, found 355.0883.



5b

ethyl 9b-(p-tolyl)-3,9b-dihydrobenzo[d]pyrrolo[1,2-b]isothiazole-1-carboxylate 5,5-dioxide

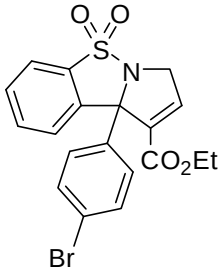
Reaction time: 3 d; Yield: 40 mg, 54%; white solid; mp 128 ^oC; R_f = 0.2 (petroleum ether/ethyl acetate = 5:1); **^1H NMR (300 MHz, $CDCl_3$):** δ 8.01-7.98 (m, 1H), 7.82 (dd, *J* = 6.8, 1.9 Hz, 1H), 7.68-7.53 (m, 2H), 7.14-7.05 (m, 5H), 4.78 (dd, *J* = 18.4, 2.5 Hz, 1H), 4.33-4.09 (m, 3H), 2.31 (s, 3H), 1.27 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 162.2, 140.8, 140.3, 138.3, 138.0, 136.0, 135.8, 133.0, 129.9, 129.3, 127.9, 126.7, 121.3, 82.7, 61.2, 52.5, 21.1, 14.1. **IR (KBr):** 1716, 1511, 1317, 1175, 1108, 923, 755, 571, 523. **HRMS (EI)** $C_{20}H_{19}NO_4S$ $[M]^+$ 369.1035, found 369.1039.



5d

ethyl 9b-(4-cyanophenyl)-3,9b-dihydrobenzo[d]pyrrolo[1,2-b] isothiazole-1-carboxylate 5,5-dioxide

Reaction time: 3 d; Yield: 56 mg, 74%; white solid; mp 132 ^{o}C; R_f = 0.2 (petroleum ether/ethyl acetate = 5:1); **^{1}H NMR (300 MHz, CDCl$_3$):** δ 8.00-7.97 (m, 1H), 7.86-7.83 (m, 1H), 7.70-7.59 (m, 4H), 7.39 (d, *J* = 8.5 Hz, 2H), 7.12 (t, *J* = 2.0 Hz, 1H), 4.81 (dd, *J* = 18.5, 2.3 Hz, 1H), 4.35 (dd, *J* = 18.5, 1.8 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 1.27 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, CDCl$_3$):** δ 162.0, 146.0, 141.1, 139.1, 136.1, 135.1, 133.5, 132.3, 130.5, 127.8, 127.5, 121.7, 118.4, 112.3, 81.8, 61.6, 53.7, 14.1. **IR (KBr):** 2229, 1720, 1450, 1243, 1177, 1112, 926, 765, 574. **HRMS (EI)** calcd for $C_{20}H_16N_2O_4S$ $[M]^+$ 380.0831, found 380.0835.

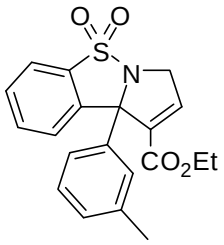


5e

ethyl 9b-(4-bromophenyl)-3,9b-dihydrobenzo[d]pyrrolo[1,2-b]isothiazole-1-carboxylate 5,5-dioxide

Reaction time: 3 d; Yield: 65 mg, 75%; white solid; mp 93 ^{o}C; R_f = 0.4 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, CDCl$_3$):** δ 8.00-7.97 (m,

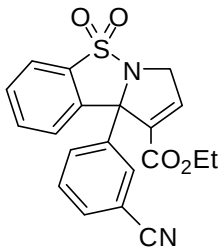
1H), 7.83-7.78 (m, 1H), 7.62 (pd, *J* = 7.3, 1.2 Hz, 2H), 7.44-7.36 (m, 2H), 7.13-7.08 (m, 3H), 4.77 (dd, *J* = 18.4, 2.4 Hz, 1H), 4.28 (dd, *J* = 18.4, 1.9 Hz, 1H), 4.21 (q, *J* = 7.2 Hz, 2H), 1.27 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 162.0, 140.9, 140.0, 139.7, 136.0, 135.4, 133.3, 131.6, 130.2, 128.6, 127.6, 122.7, 121.5, 82.1, 61.4, 53.1, 14.1. **IR (KBr):** 1717, 1325, 1175, 1114, 923, 758, 572. **HRMS (EI)** calcd for $C_{19}H_{16}BrNO_4S$ $[M]^+$ 432.9983, 432.9963, found 432.9987, 432.9969.



5f

ethyl 9b-(m-tolyl)-3,9b-dihydrobenzo[d]pyrrolo[1,2-b]isothiazole-1-carboxylate 5,5-dioxide

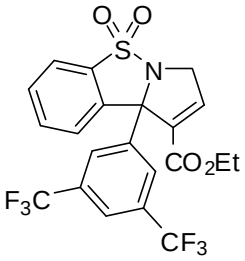
Reaction time: 3 d; Yield: 49 mg, 66%; white solid; mp 180 °C; R_f = 0.3 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 8.00–7.95 (m, 1H), 7.87–7.79 (m, 1H), 7.61 (pd, *J* = 7.3, 1.4 Hz, 2H), 7.24–7.15 (m, 1H), 7.10 (dd, *J* = 4.3, 2.0 Hz, 2H), 7.04–6.93 (m, 2H), 4.78 (dd, *J* = 18.4, 2.5 Hz, 1H), 4.32–4.23 (m, 1H), 4.23–4.12 (m, 2H), 2.29 (s, 3H), 1.26 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 162.2, 140.9, 140.7, 140.1, 138.2, 135.9, 133.0, 129.9, 129.3, 128.4, 128.0, 127.4, 123.9, 121.3, 82.8, 61.2, 52.5, 21.6, 14.1. **IR (KBr):** 1716, 1522, 1316, 1176, 1111, 925, 760, 572. **HRMS (EI)** calcd for $C_{20}H_{19}NO_4S$ $[M]^+$ 369.1035, found 369.1040.



5g

ethyl 9b-(3-cyanophenyl)-3,9b-dihydrobenzo[d]pyrrolo[1,2-b] isothiazole -1-carboxylate 5,5-dioxide

Reaction time: 3 d; Yield: 59 mg, 77%; white solid; mp 137 ^{o}C; R_f = 0.3 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, CDCl$_3$):** δ 8.00-7.97 (m, 1H), 7.90-7.81 (m, 1H), 7.76-7.55 (m, 4H), 7.53-7.38 (m, 2H), 7.12 (t, *J* = 2.0 Hz, 1H), 4.80 (dd, *J* = 18.5, 2.3 Hz, 1H), 4.36 (dd, *J* = 18.5, 1.8 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 1.28 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, CDCl$_3$):** δ 161.9, 142.6, 141.1, 139.1, 136.2, 135.1, 133.6, 132.0, 131.4, 130.7, 130.5, 129.4, 127.4, 121.8, 118.6, 112.7, 81.56, 61.6, 53.7, 14.1. **IR (KBr):** 2230, 1716, 1330, 1176, 1025, 797, 644, 570. **HRMS (EI)** $C_{20}H_16N_2O_4S$ $[M]^+$ 380.0831, found 380.0835.

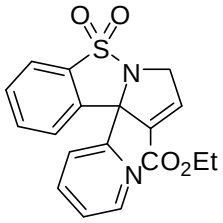


5h

ethyl 9b-(3,5-bis(trifluoromethyl)phenyl)-3,9b-dihydrobenzo[d]pyrrolo [1,2-b]isothiazole-1-carboxylate 5,5-dioxide

Reaction time: 3 d; Yield: 61 mg, 62%; white solid; mp 126 ^{o}C; R_f = 0.5 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, CDCl$_3$):** δ 8.03 (d, *J* = 7.5 Hz, 1H), 7.85 (dd, *J* = 8.0, 7.3 Hz, 2H), 7.77 (s, 2H), 7.75–7.62 (m, 2H), 7.14 (s, 1H),

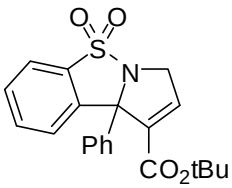
4.82 (dd, J = 18.5, 2.2 Hz, 1H), 4.43 (dd, J = 18.5, 1.5 Hz, 1H), 4.22 (q, J = 7.1 Hz, 2H), 1.26 (t, J = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, CDCl$_3$):** δ 161.9, 143.7, 141.3, 138.8, 136.2, 135.1, 133.7, 131.8 (q, $J_{C\text{-}F}$ = 33.1 Hz), 130.7, 127.3, 127.2, 124.9 (q, $J_{C\text{-}F}$ = 270 Hz), 122.0, 121.3, 81.3, 61.7, 54.0, 14.0. **IR (KBr):** 1720, 1372, 1279, 1175, 1134, 841, 763, 569. **HRMS (EI)** calcd for $C_{21}H_{15}F_6NO_4S$ $[M]^+$ 491.0626, found 491.0632.



5i

ethyl 9b-(pyridin-2-yl)-3,9b-dihydrobenzo[d]pyrrolo[1,2-b]isothiazole-1-carboxylate 5,5-dioxide

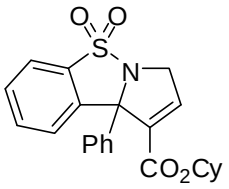
Reaction time: 3 d; Yield: 56 mg, 79%; yellow solid; mp 195 ^{o}C; R_f = 0.2 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, CDCl$_3$):** δ 8.56 (d, J = 4.1 Hz, 1H), 8.05 (dd, J = 6.5, 2.1 Hz, 1H), 7.84 (dd, J = 6.3, 2.4 Hz, 1H), 7.69-7.53 (m, 3H), 7.25-7.16 (m, 1H), 7.09-7.01 (m, 2H), 4.84 (dd, J = 18.1, 2.4 Hz, 1H), 4.32 (dd, J = 18.0, 1.8 Hz, 1H), 4.22 (q, J = 7.1 Hz, 2H), 1.27 (t, J = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, CDCl$_3$):** δ 162.3, 159.5, 149.0, 139.5, 139.5, 137.1, 135.9, 135.7, 133.2, 130.1, 128.0, 123.2, 121.3, 121.2, 83.8, 61.1, 53.5, 14.1. **IR (KBr):** 1714, 1671, 1448, 1353, 1336, 1182, 1112, 1093, 842, 753, 574, 527. **HRMS (EI)** calcd for $C_{18}H_{16}N_2O_4S$ $[M]^+$ 355.0753, found 355.0757.



5j

tert-butyl 9b-phenyl-3,9b-dihydrobenzo[d]pyrrolo[1,2-b]isothiazole-1-carboxylate 5,5-dioxide

Reaction time: 2 d; Yield: 56 mg, 73%; yellow solid; mp 158 ^{o}C; R_f = 0.6 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.98 (d, *J* = 7.7 Hz, 1H), 7.82 (d, *J* = 7.2 Hz, 1H), 7.60 (dq, *J* = 14.3, 7.1 Hz, 2H), 7.44-7.25 (m, 3H), 7.22-7.12 (m, 2H), 6.98 (s, 1H), 4.73 (dd, *J* = 18.2, 2.3 Hz, 1H), 4.24 (dd, *J* = 18.2, 1.4 Hz, 1H), 1.42 (s, 9H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 161.5, 141.2, 140.5, 140.0, 137.1, 136.2, 133.1, 130.0, 128.5, 128.3, 127.7, 126.8, 121.4, 82.7, 82.4, 52.9, 28.1. **IR (KBr):** 1712, 1310, 1175, 764, 611. **HRMS (EI)** calcd for $C_{21}H_{21}NO_4S$ $[M]^+$ 383.1191, found 383.1195.

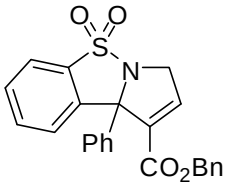


5k

cyclohexyl 9b-phenyl-3,9b-dihydrobenzo[d]pyrrolo[1,2-b] isothiazole-1-carboxylate 5,5-dioxide

Reaction time: 2 d; Yield: 59 mg, 72%; yellow solid; mp 140 ^{o}C; R_f = 0.5 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 8.00 (dd, *J* = 6.9, 1.4 Hz, 1H), 7.83 (dd, *J* = 6.8, 1.9 Hz, 1H), 7.65-7.55 (m, 2H), 7.38-7.27 (m, 3H),

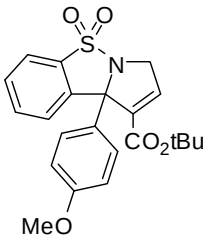
7.22-7.19 (m, 2H), 7.09 (t, J = 2.1 Hz, 1H), 4.97-4.66 (m, 2H), 4.27 (dd, J = 18.3, 1.8 Hz, 1H), 1.95-1.62 (m, 4H), 1.58-1.11 (m, 6H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 161.7, 141.1, 140.8, 140.3, 136.3, 136.1, 133.1, 130.0, 128.6, 128.5, 127.9, 126.9, 121.5, 82.8, 74.0, 52.8, 31.6, 31.5, 25.3, 23.8, 23.7. **IR (KBr):** 1713, 1318, 1240, 1176, 761, 611. **HRMS (EI)** calcd for $C_{23}H_{23}NO_4S$ $[M]^+$ 409.1348, found 409.1354.



5l

benzyl 9b-phenyl-3,9b-dihydrobenzo[d]pyrrolo[1,2-b]isothiazole-1-carboxylate 5,5-dioxide

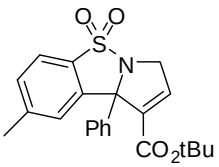
Reaction time: 2 d; Yield: 63 mg, 76%; yellow solid; mp 143 °C; R_f = 0.7 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.93 (dd, J = 6.1, 2.8 Hz, 1H), 7.84-7.76 (m, 1H), 7.56 (dd, J = 5.8, 3.2 Hz, 2H), 7.39-7.17 (m, 10H), 7.13 (t, J = 2.1 Hz, 1H), 5.17 (s, 2H), 4.77 (dd, J = 18.5, 2.5 Hz, 1H), 4.24 (dd, J = 18.5, 1.8 Hz, 1H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 162.0, 141.7, 140.8, 140.0, 136.1, 135.6, 135.2, 133.2, 130.1, 128.7, 128.7, 128.6, 128.5, 128.3, 127.9, 126.9, 121.4, 82.8, 67.0, 52.8. **IR (KBr):** 1716, 1315, 1175, 752, 697. **HRMS (EI)** calcd for $C_{24}H_{19}NO_4S$ $[M]^+$ 417.1035, found 417.1040.



5m

tert-butyl 9b-(4-methoxyphenyl)-3,9b-dihydrobenzo[d]pyrrolo[1,2-b] isothiazole-1-carboxylate 5,5-dioxide

Reaction time: 5 d; Yield: 56 mg, 67%; yellow solid; mp 155 ^{o}C; R_f = 0.5 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, CDCl$_3$):** δ 8.05-7.93 (m, 1H), 7.89-7.80 (m, 1H), 7.71-7.52 (m, 2H), 7.19-7.06 (m, 2H), 6.96 (t, *J* = 2.1 Hz, 1H), 6.89-6.71 (m, 2H), 4.72 (dd, *J* = 18.1, 2.4 Hz, 1H), 4.24 (dd, *J* = 18.2, 1.8 Hz, 1H), 3.77 (s, 3H), 1.44 (s, 9H). ^{13}C **NMR (75 MHz, CDCl$_3$):** δ 161.6, 159.6, 140.9, 139.9, 137.2, 136.3, 133.5, 133.1, 129.9, 128.2, 127.8, 121.5, 113.9, 82.6, 82.4, 55.4, 52.7, 28.2. **IR (KBr):** 1713, 1253, 1174, 756, 612. **HRMS (EI)** calcd for $C_{22}H_{23}NO_5S$ $[M]^+$ 413.1297, found 413.1302.



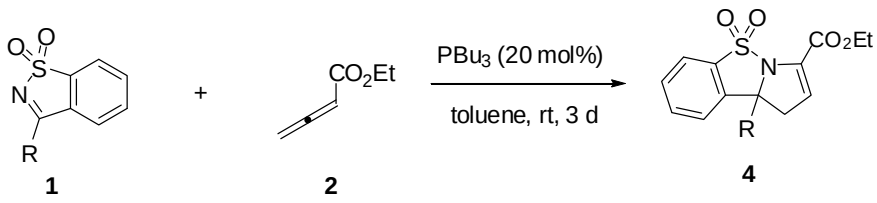
5a'

tert-butyl 8-methyl-9b-phenyl-3,9b-dihydrobenzo[d]pyrrolo[1,2-b] isothiazole-1-carboxylate 5,5-dioxide

Reaction time: 2 d; Yield: 63 mg, 80%; white solid; mp 163 ^{o}C; R_f = 0.6 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, CDCl$_3$):** δ 7.76 (s, 1H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.34-7.24 (m, 3H), 7.22-7.13 (m,

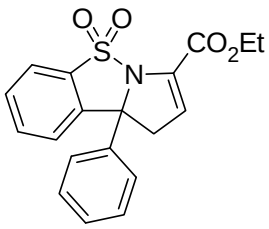
2H), 6.98 (t, *J* = 1.9 Hz, 1H), 4.72 (dd, *J* = 18.2, 2.4 Hz, 1H), 4.23 (dd, *J* = 18.2, 1.7 Hz, 1H), 2.43 (s, 3H), 1.42 (s, 9H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 161.6, 144.1, 141.4, 140.9, 140.0, 137.2, 133.7, 130.9, 128.5, 128.3, 127.8, 126.9, 121.2, 82.6, 82.3, 52.9, 28.1, 22.0. **IR (KBr):** 1709, 1308, 1105, 834, 604. **HRMS (EI)** calcd for $C_{22}H_{23}NO_4S$ $[M]^+$ 397.1348, found 397.1353.

4. PBu_3-catalyzed [3 + 2] cycloaddition (Scheme 1).



1 **2** **4**

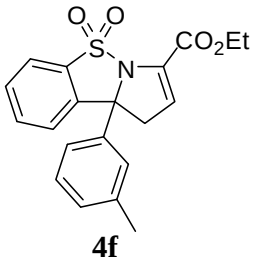
To a stirred solution of cyclic ketimines (0.2 mmol) and PBu_3 (8.1 mg, 0.04 mmol) in toluene (2 mL) was added ethyl 2,3-butadienoate (48.8 mg, 0.4 mmol). The reaction mixture was stirred for 3 d at room temperature until the full consumption of the cyclic ketimines. The reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleumether/EtOAc, typically 20:1-5:1) to furnish the corresponding [3+2] cycloadduct **4**.



4a

ethyl 9b-phenyl-1,9b-dihydrobenzo[d]pyrrolo[1,2-b]isothiazole-3-carboxylate 5,5-dioxide

Reaction time: 3 d; **3a**:**4a**:**5a** = 9:82:9. Yield of pure **4a**: 37 mg, 52%; white solid; mp 165 ^{o}C; R_f = 0.3 (petroleum ether/ethyl acetate = 3:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.74 (d, *J* = 7.8 Hz, 1H), 7.69 (dd, *J* = 5.5, 3.6 Hz, 2H), 7.61 (td, *J* = 7.6, 1.2 Hz, 1H), 7.54-7.43 (m, 2H), 7.42-7.35 (m, 2H), 7.34-7.27 (m, 1H), 6.34 (dd, *J* = 3.2, 2.7 Hz, 1H), 4.47-4.27 (m, 2H), 3.56 (ddd, *J* = 20.9, 18.3, 3.0 Hz, 2H), 1.39 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 160.7, 143.4, 142.4, 136.2, 134.5, 134.0, 129.9, 129.0, 128.4, 125.5, 124.4, 124.2, 122.5, 79.1, 61.8, 46.0, 14.1. **IR (KBr):** 1729, 1330, 1178, 739, 571. **HRMS (EI)** calcd for $C_{19}H_{17}NO_4S$ $[M]^+$ 355.0878, found 355.0882.



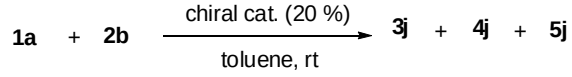
4f

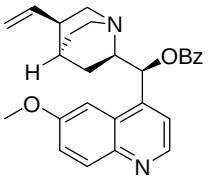
ethyl 9b-(m-tolyl)-1,9b-dihydrobenzo[d]pyrrolo[1,2-b]isothiazole-3-carboxylate 5,5-dioxide

Reaction time: 3 d; **3f:4f:5f** = 3:88:9. Yield of **4f**: 43 mg, 58%; white solid; mp 148 ^{o}C; R_f = 0.3 (petroleum ether/ethyl acetate = 10:1); **^{1}H NMR (300 MHz, $CDCl_3$):** δ 7.73 (d, *J* = 7.8 Hz, 1H), 7.60 (dd, *J* = 11.0, 4.0 Hz, 1H), 7.55-7.41 (m, 4H), 7.26 (t, *J* = 7.6 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 6.34 (t, *J* = 2.8 Hz, 1H), 4.51-4.25 (m, 2H), 3.54 (ddd, *J* = 20.8, 18.4, 2.9 Hz, 2H), 2.35 (s, 3H), 1.38 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 160.7, 143.3, 142.4, 138.8, 136.1, 134.4, 134.0, 129.8,

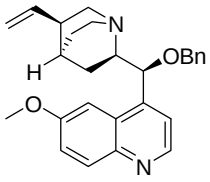
129.2, 128.9, 126.1, 124.4, 124.4, 122.5, 122.4, 79.1, 61.7, 46.0, 21.7, 14.1. **IR (KBr):** 1729, 1634, 1605, 1449, 1371, 1179, 1137, 759, 652, 569. **HRMS (EI)** calcd for $C_{20}H_{19}NO_4S$ $[M]^+$ 369.1035, found 369.1039.

5. Chiral catalysts for the reactions (Table S1).

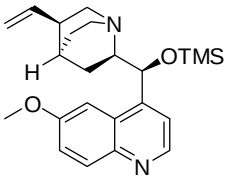




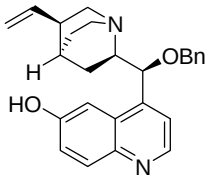
6a, trace



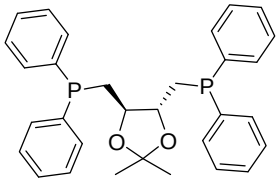
6b, trace



6c, trace

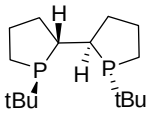


6d, trace



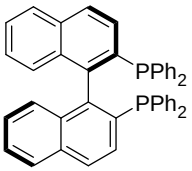
7a (+)-DIOP

5j, 68% yield[a], 0% ee[b]
(**3j**:**4j**:**5j** = 0:9:91)[c]



7b (+)-Tangphos

5j, 45% yield[a] , 0% ee[b]
(**3j**:**4j**:**5j** = 0:37:63)[c]

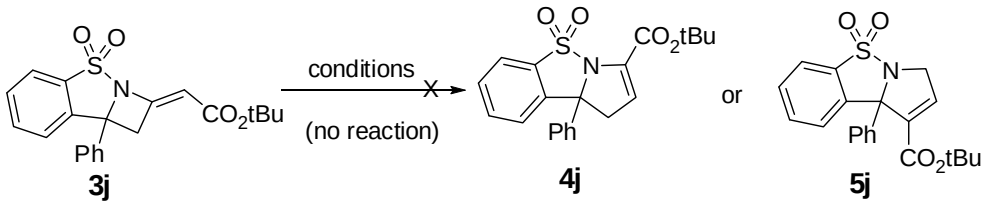


7c (*R*)-BINAP

5j, 14% yield[a] , 0% ee[b]
(**3j**:**4j**:**5j** = 0:0:100)[c]

[a] Isolated yield of pure **5j**.
[b] Determined by chiral HPLC for **5j**.
[c] Determined by the ^{1}H NMR (300 MHz) of the reaction mixture.

6. Control experiments for expansion of cycloadduct 3j to 4j/5j

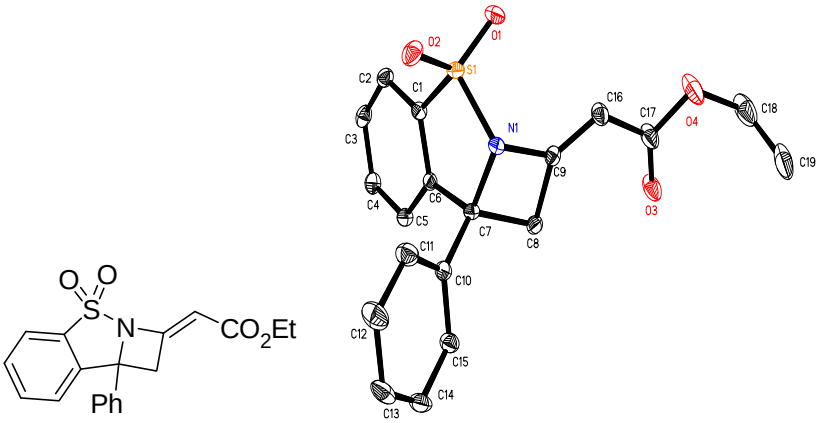


3j **4j** **5j**

conditions A: toluene, rt, 3d, no reaction detected
conditions B: **DABCO (20 mol%)**, toluene, rt, 3d, no reaction detected
conditions C: **PPh_3 (20 mol%)**, toluene, rt, 3d, no reaction detected

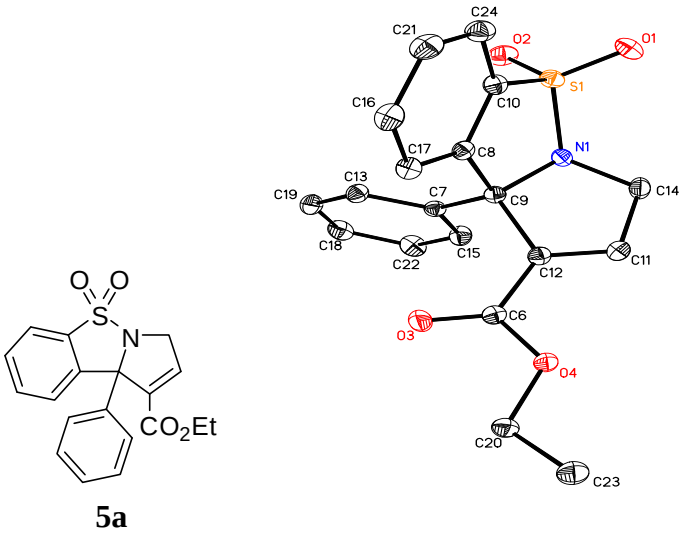
7. X-Ray structures of cycloadduct 3a and 5a (Fig. S1 and S2)

The crystal suitable for X-ray analysis was prepared by slow evaporation of the solvent of the solution of **3a** and **5a** in chloroform at room temperature (Figure S1 and S2).



3a

Fig. S1 X-ray structures of cycloaddition products **3a**



5a

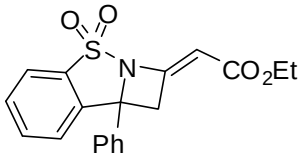
Fig. S2 X-ray structures of cycloaddition products **5a**.

8. References

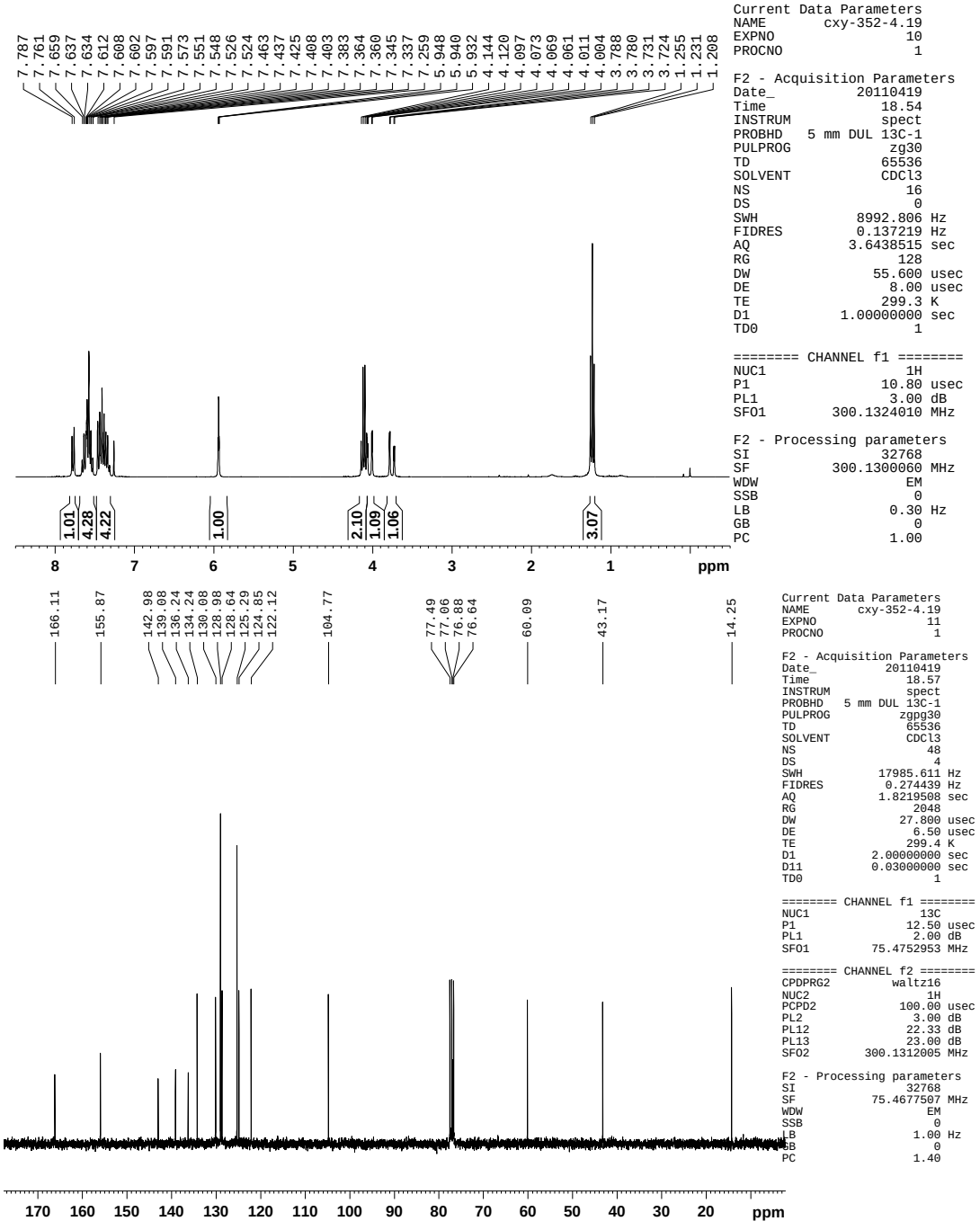
1. F. A. Davis, J. C. Towson, D. B. Vashi, R. ThimmaReddy, J. P. McCauley, M. E. Harakal and D. J. Gosciniak, *J. Org. Chem.*, 1990, **55**, 1254;

2. (a) Y.-L. Shi and M. Shi, *Org. Lett.*, 2005, **7**, 3057; (b) G. S. Creech and O. Kwon, *Org. Lett.*, 2008, **10**, 429;

Part III NMR Spectra



3a

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DS                    0
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FIDRES         0.137219 Hz
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RG                  128
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DE                 8.00 usec
TE                299.3 K
D1           1.00000000 sec
TD0                   1

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PL1                3.00 dB
SFO1        300.1324010 MHz

F2 - Processing parameters
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P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

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PL2                3.00 dB
PL12               22.33 dB
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SFO2        300.1312005 MHz

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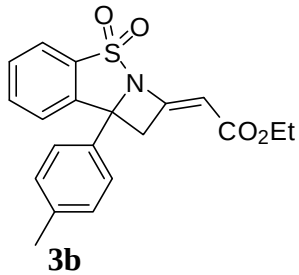
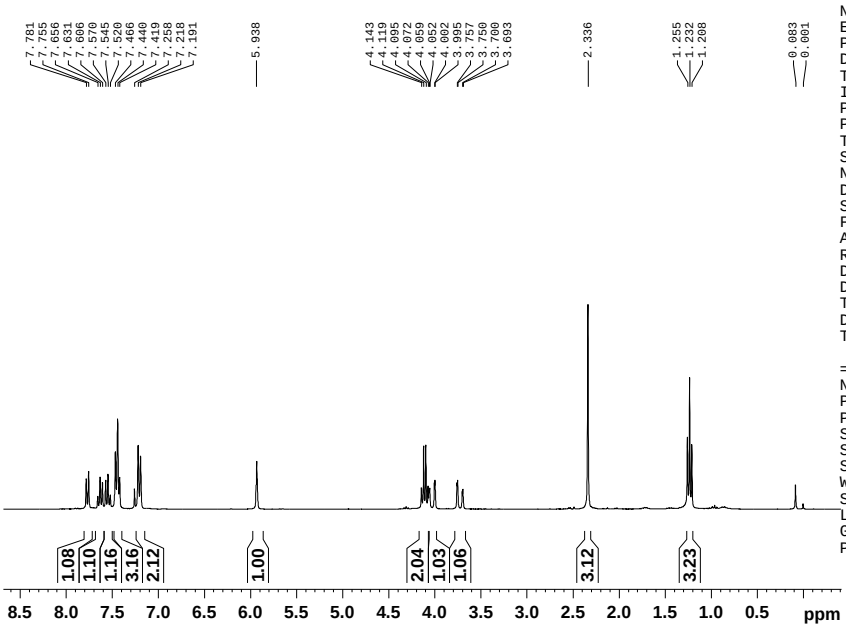


3b

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SOLVENT            CDCl3
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DS                     0
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FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   128
DW                55.600 usec
DE                 8.00 usec
TE                296.7 K
D1             1.00000000 sec
TD0                    1

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P1                10.80 usec
PL1                3.00 dB
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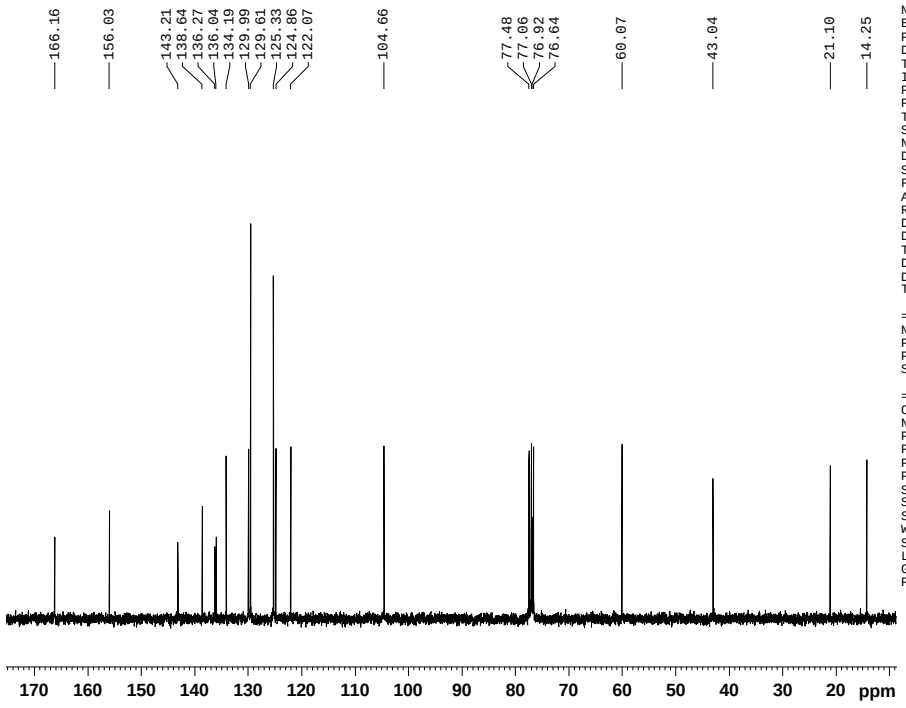


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TD                 65536
SOLVENT            CDCl3
NS                    60
DS                     4
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FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 574.7
DW                27.800 usec
DE                 6.50 usec
TE                296.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

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P1                12.50 usec
PL1                2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
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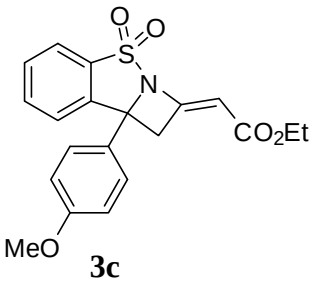
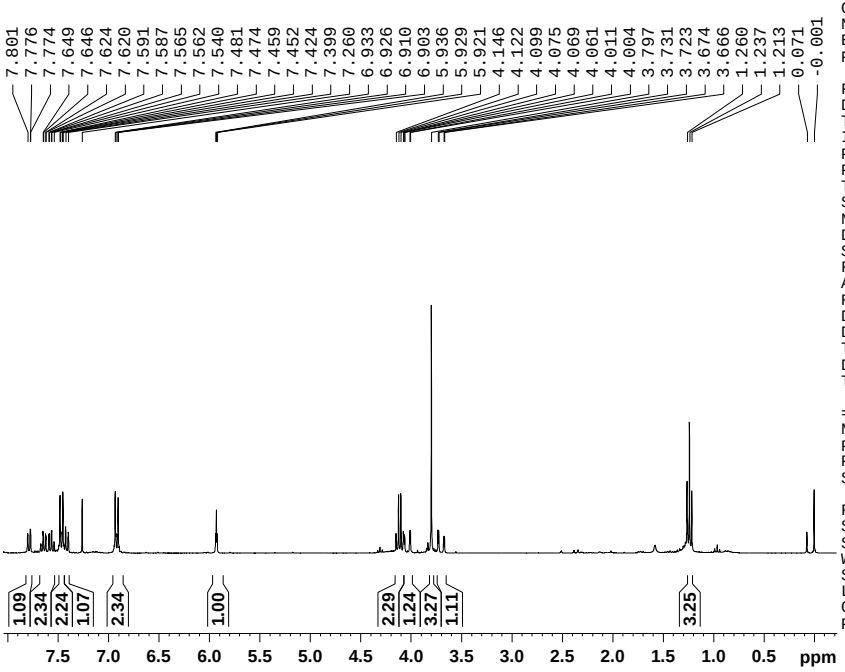


3c

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TD                65536
SOLVENT           CDCl3
NS                   11
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SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ             3.6438515 sec
RG                  362
DW               55.600 usec
DE                8.00 usec
TE                299.9 K
D1           1.00000000 sec
TD0                   1

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PL1                3.00 dB
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F2 - Processing parameters
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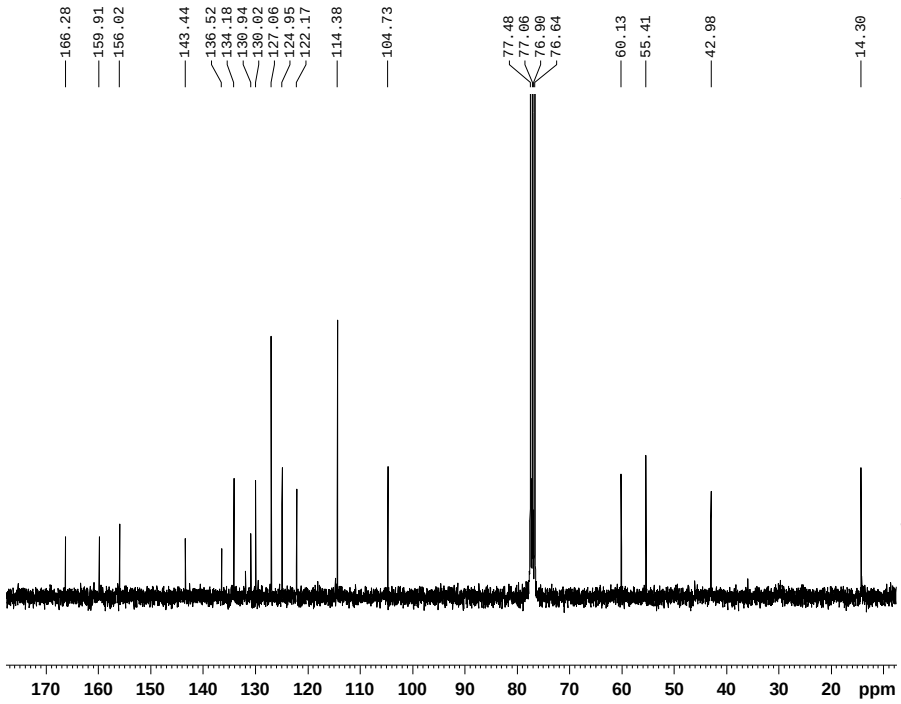


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TD                65536
SOLVENT           CDCl3
NS                  651
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                812.7
DW               27.800 usec
DE                6.50 usec
TE                300.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
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P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
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NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
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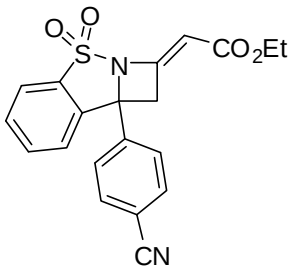
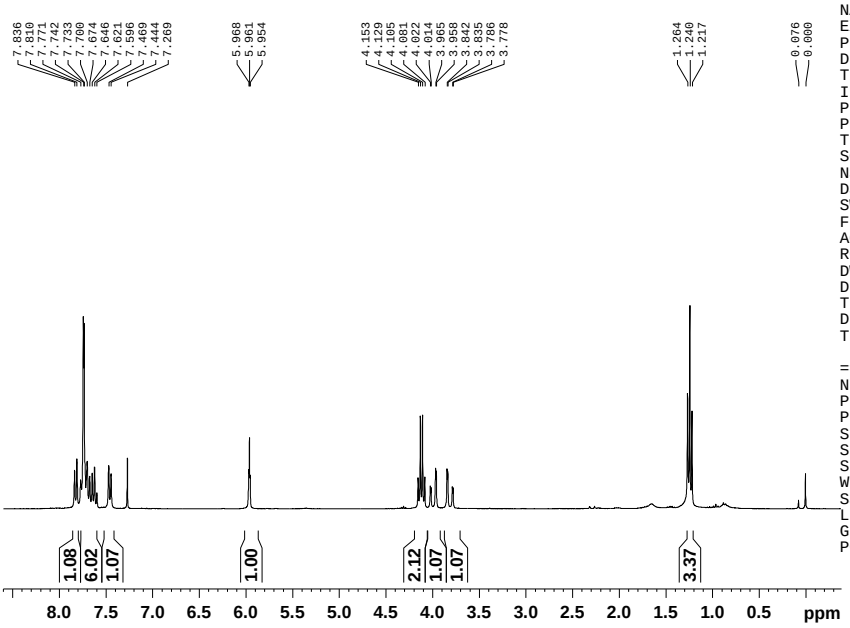


3d

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FIDRES           0.137219 Hz
AQ               3.6438515 sec
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TE                 299.7 K
D1              1.00000000 sec
TD0                     1

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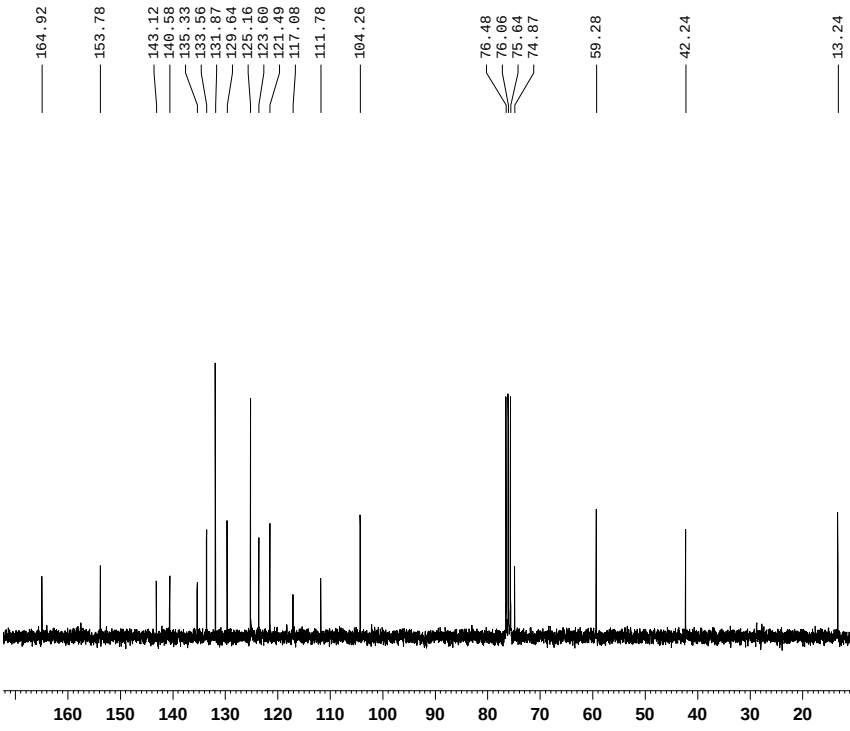


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TD                  65536
SOLVENT             CDCl3
NS                     62
DS                      4
SWH              17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                  1625.5
DW                 27.800 usec
DE                  6.50 usec
TE                 299.8 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                  12.50 usec
PL1                  2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                  3.00 dB
PL12                22.33 dB
PL13                23.00 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
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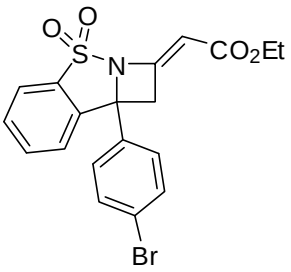
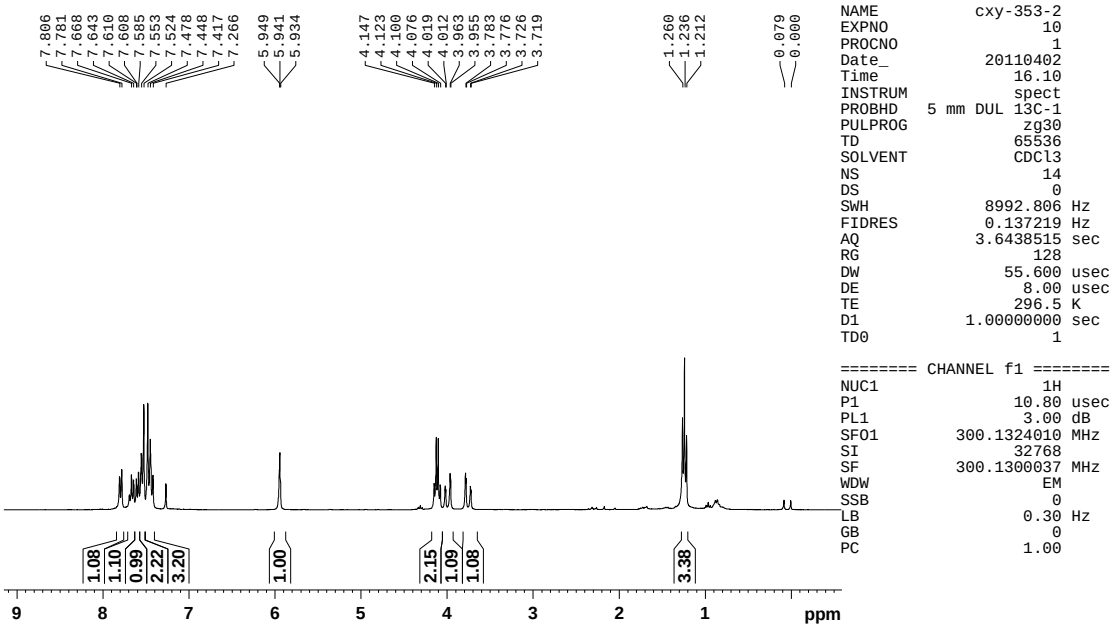
SF            75.4678238 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

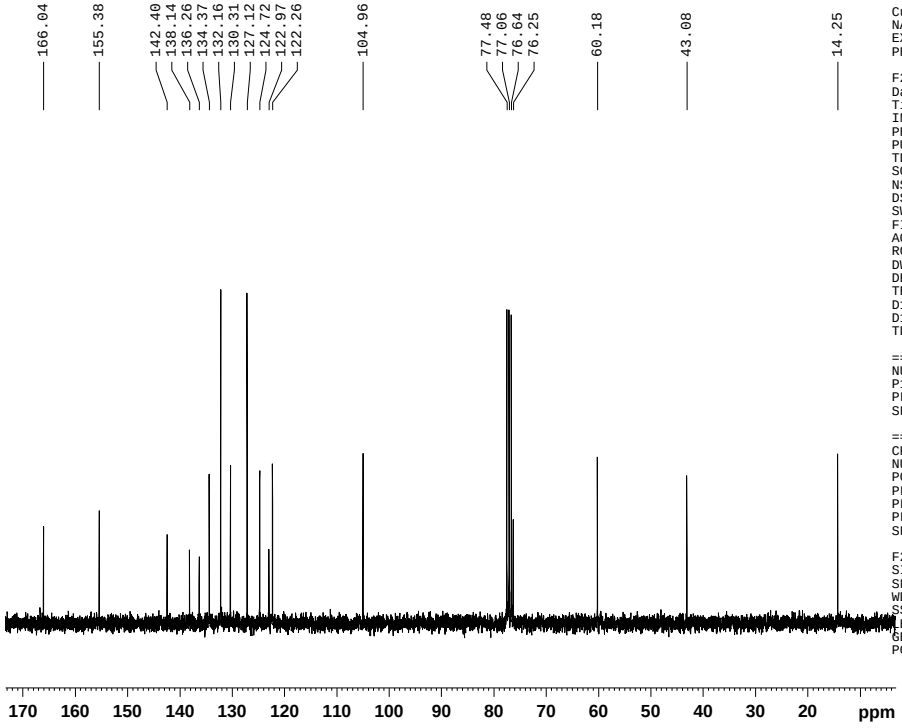


3e




```
NAME            cxy-353-2
EXPNO                  10
PROCNO                  1
Date_            20110402
Time                16.10
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     14
DS                      0
SWH              8992.806 Hz
FIDRES           0.137219 Hz
AQ               3.6438515 sec
RG                    128
DW                 55.600 usec
DE                  8.00 usec
TE                 296.5 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.80 usec
PL1                  3.00 dB
SFO1          300.1324010 MHz
SI                  32768
SF            300.1300037 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



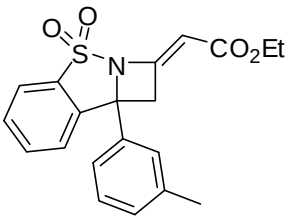

```
Current Data Parameters
NAME            cxy-353-2
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110402
Time                16.15
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     43
DS                      4
SWH             17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                    512
DW                 27.800 usec
DE                  6.50 usec
TE                 296.7 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

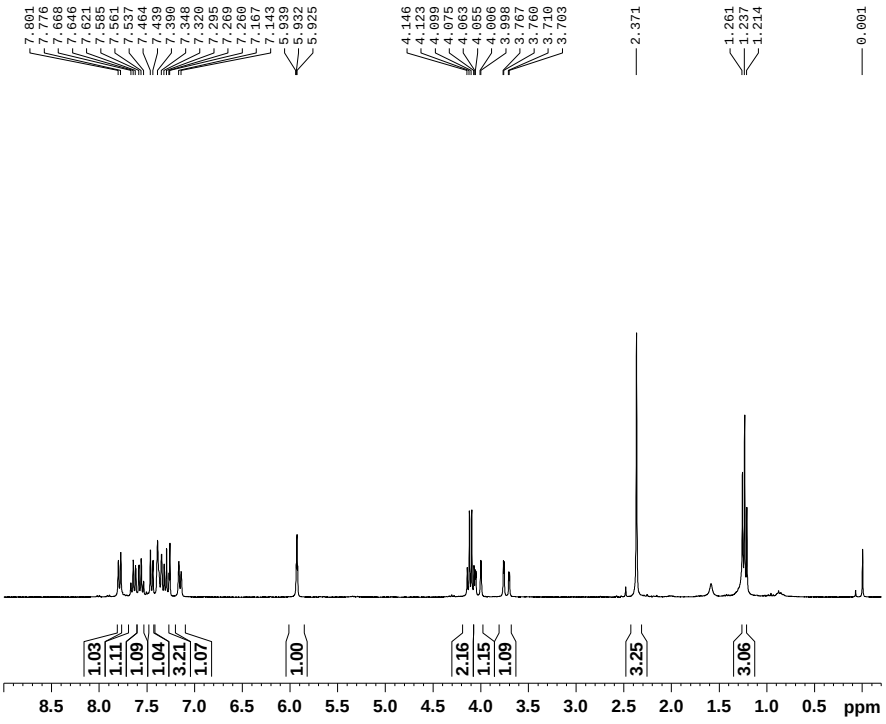
======== CHANNEL f1 ========
NUC1                  13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                  3.00 dB
PL12                22.33 dB
PL13                23.00 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                  32768
SF            75.4677498 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

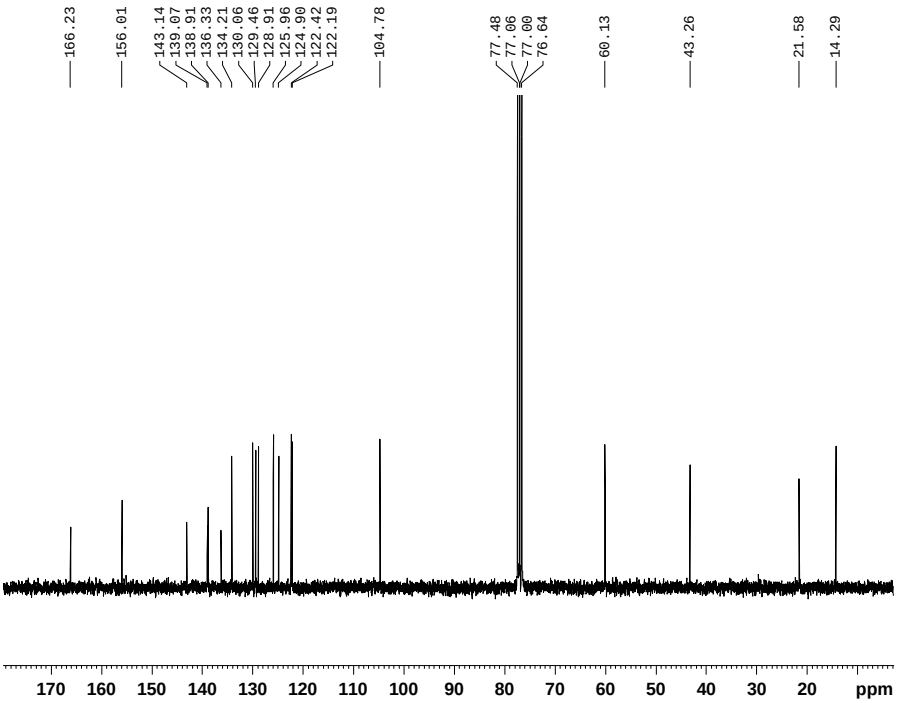


3f




```
NAME          cxy-366-6.16
EXPNO                   10
PROCNO                   1
Date_             20110616
Time                 16.00
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      4
DS                      0
SWH               8992.806 Hz
FIDRES           0.137219 Hz
AQ               3.6438515 sec
RG                    256
DW                 55.600 usec
DE                  8.00 usec
TE                 299.6 K
D1              1.00000000 sec
TD0                     1

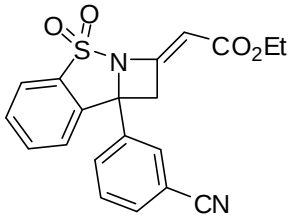
======== CHANNEL f1 ========
NUC1                   1H
P1                  10.80 usec
PL1                  3.00 dB
SFO1          300.1324010 MHz
SI                   32768
SF            300.1300060 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



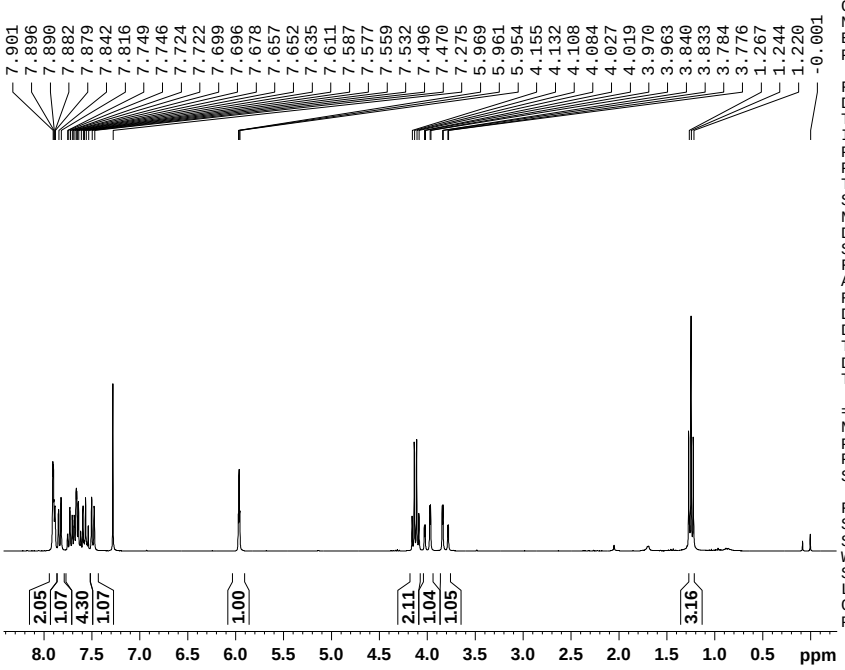

```
NAME          cxy-366-6.17
EXPNO                   10
PROCNO                   1
Date_             20110617
Time                 16.42
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    304
DS                      4
SWH              17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                    512
DW                 27.800 usec
DE                  6.50 usec
TE                 299.7 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                  12.50 usec
PL1                  2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                  3.00 dB
PL12                22.33 dB
PL13                23.00 dB
SFO2          300.1312005 MHz
SI                   32768
SF            75.4677462 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



3g

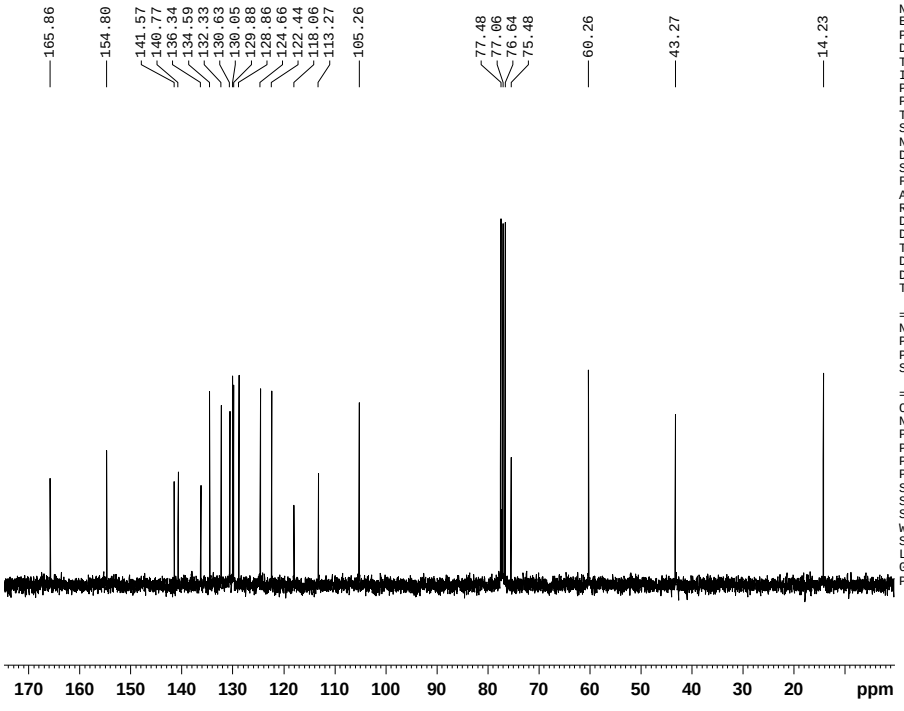



```
Current Data Parameters
NAME         cxy-367-3.3
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110303
Time               16.05
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    14
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   128
DW                55.600 usec
DE                 8.00 usec
TE                296.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.80 usec
PL1                3.00 dB
SFO1        300.1324010 MHz

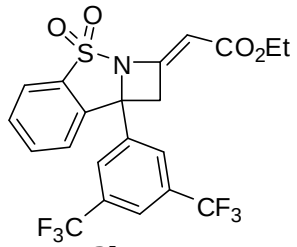
F2 - Processing parameters
SI                 32768
SF          300.1300014 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



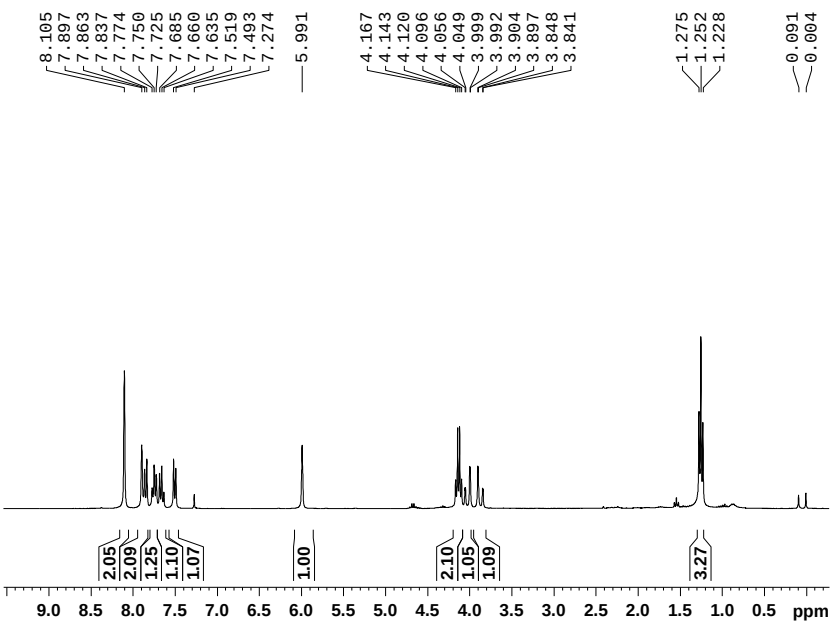

```
NAME       cxy-367-3.4-c
EXPNO                 10
PROCNO                 1
Date_           20110304
Time               16.16
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    52
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 13004
DW                27.800 usec
DE                 8.00 usec
TE                295.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2        300.1312005 MHz
SI                 32768
SF           75.4677508 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

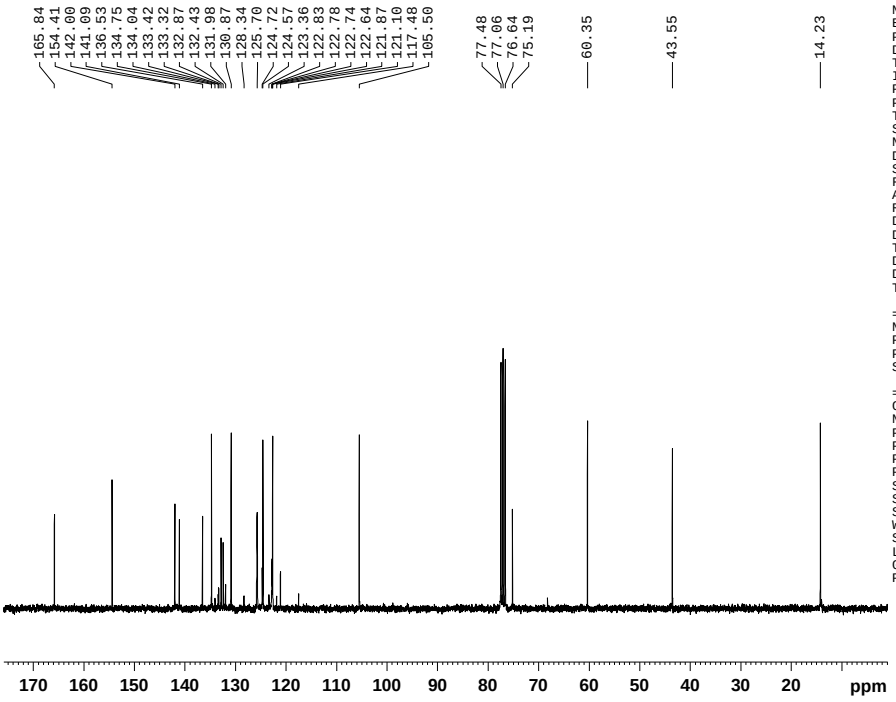


3h




```
NAME          cxy-360-6.13
EXPNO                   10
PROCNO                   1
Date_             20110613
Time                19.05
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8992.806 Hz
FIDRES           0.137219 Hz
AQ               3.6438515 sec
RG                    128
DW                 55.600 usec
DE                  8.00 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                     1

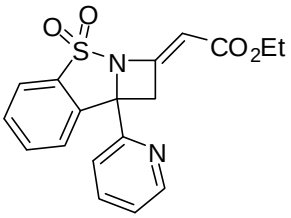
======== CHANNEL f1 ========
NUC1                   1H
P1                 10.80 usec
PL1                 3.00 dB
SFO1          300.1324010 MHz
SI                  32768
SF            300.1300018 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```



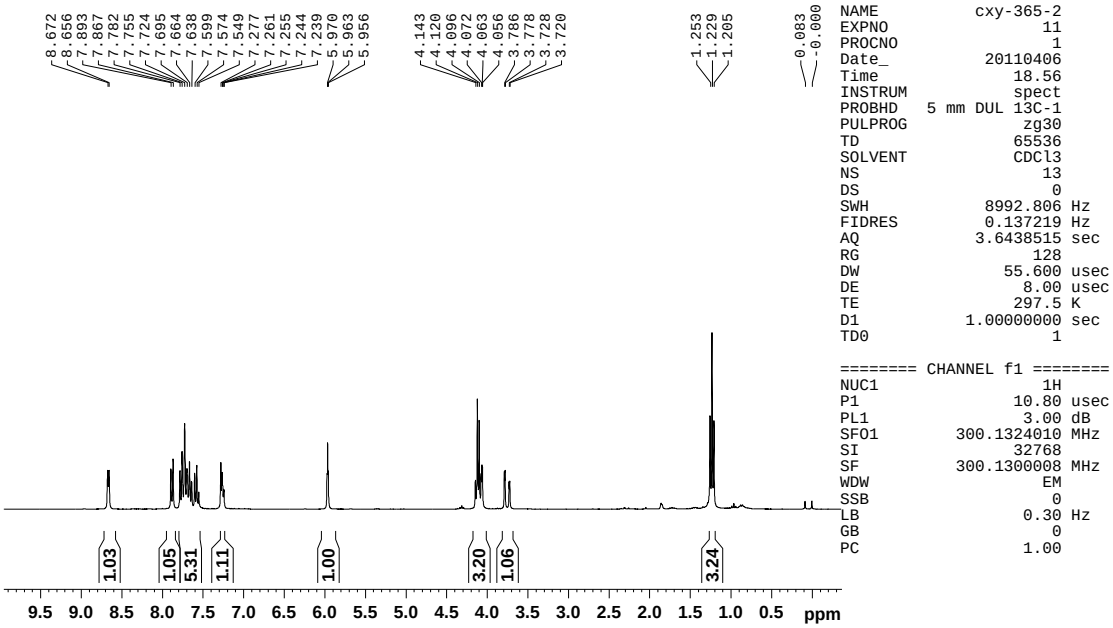

```
NAME      cxy-360-6-13-c
EXPNO                 10
PROCNO                 1
Date_           20110614
Time                8.16
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  276
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 sec
RG                574.7
DW               27.800 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677461 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

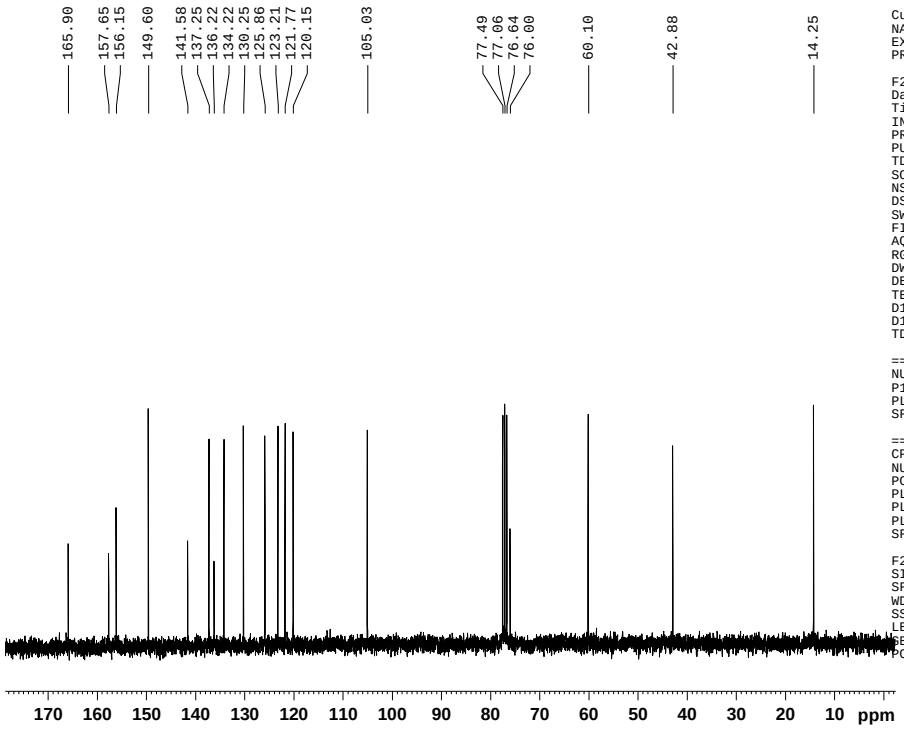


3i




```
NAME            cxy-365-2
EXPNO                  11
PROCNO                  1
Date_            20110406
Time                18.56
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     13
DS                      0
SWH              8992.806 Hz
FIDRES           0.137219 Hz
AQ               3.6438515 sec
RG                    128
DW                 55.600 usec
DE                  8.00 usec
TE                 297.5 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.80 usec
PL1                 3.00 dB
SFO1          300.1324010 MHz
SI                  32768
SF            300.1300008 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```



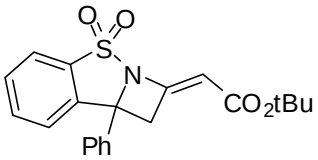

```
Current Data Parameters
NAME            cxy-365-2
EXPNO                  12
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110406
Time                18.58
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     37
DS                      4
SWH             17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                    512
DW                 27.800 usec
DE                  6.50 usec
TE                 297.8 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

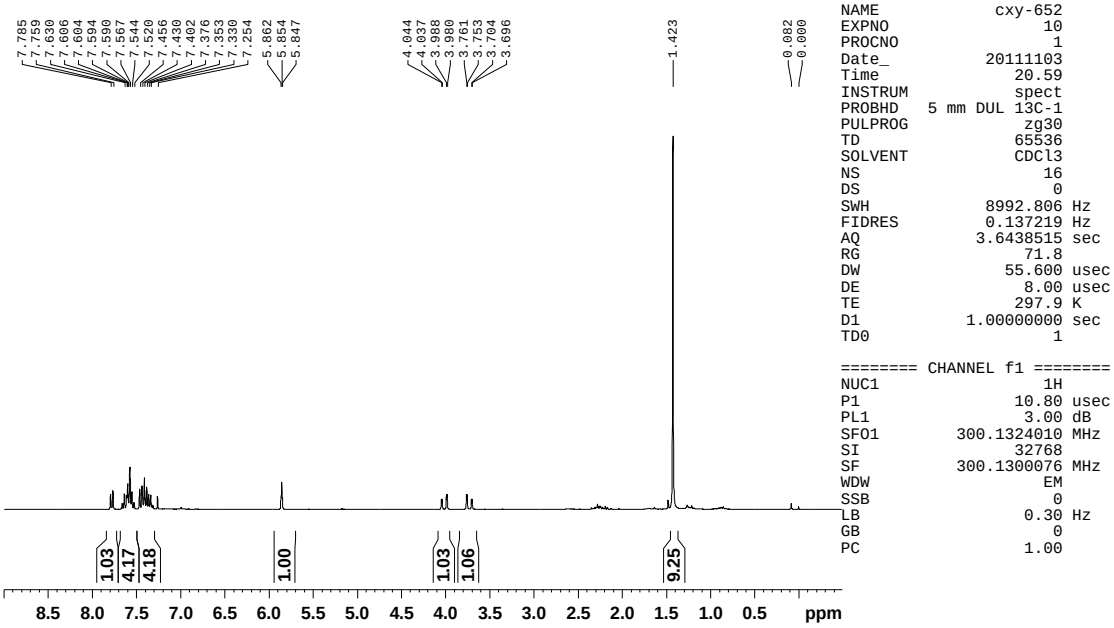
======== CHANNEL f1 ========
NUC1                  13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                 3.00 dB
PL12               22.33 dB
PL13               23.00 dB
SFO2          300.1312005 MHz

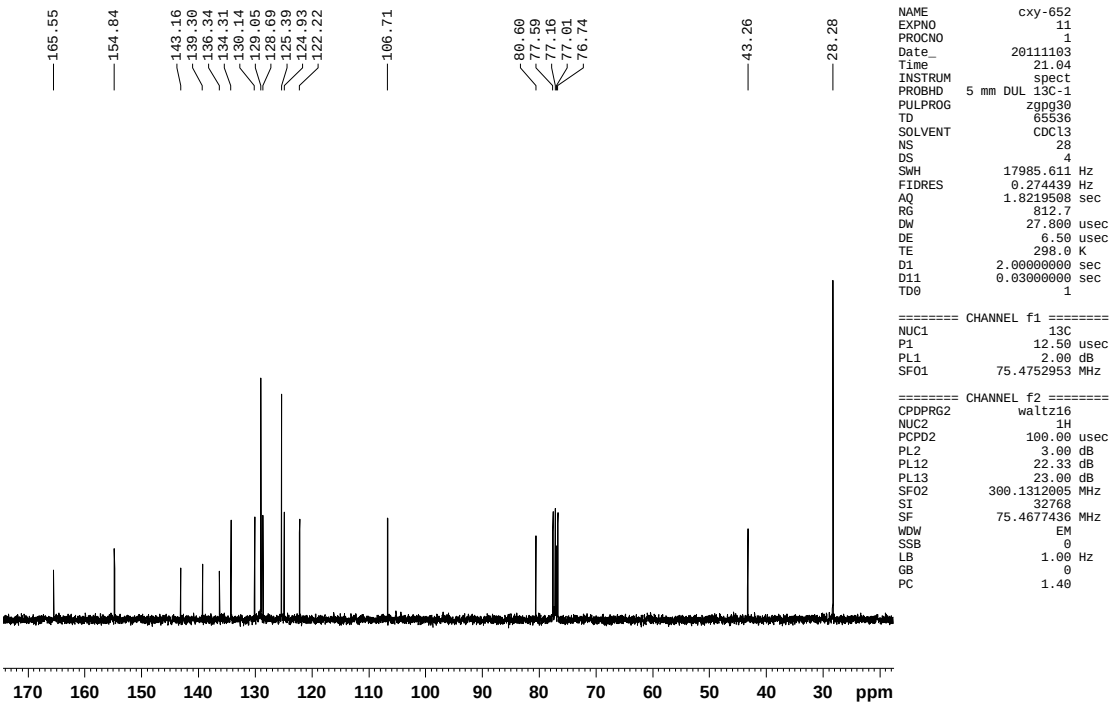
F2 - Processing parameters
SI                  32768
SF            75.4677500 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```



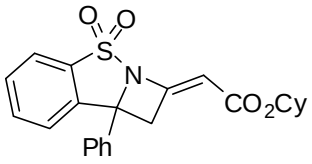
3j



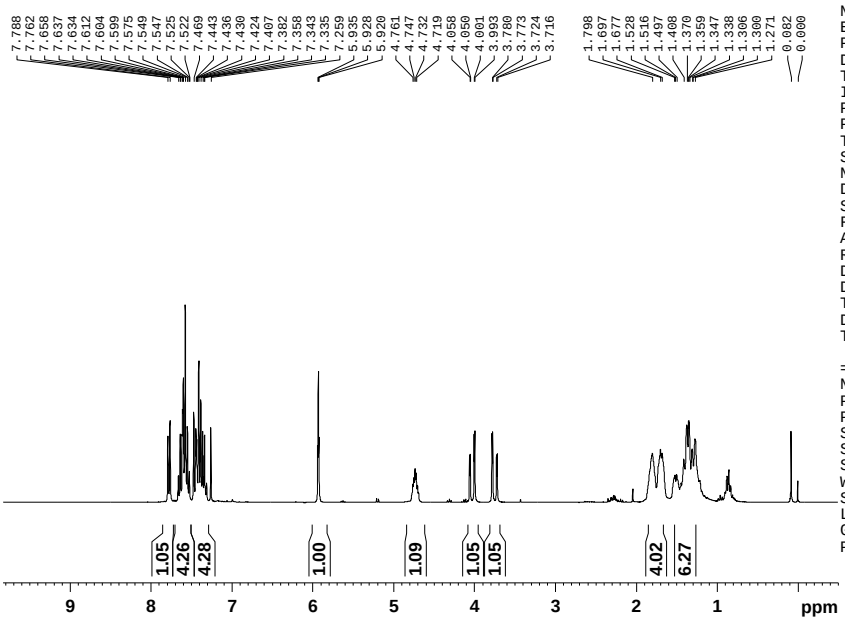
NAME	cxy-652
EXPNO	10
PROCNO	1
Date_	20111103
Time	20.59
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8992.806 Hz
FIDRES	0.137219 Hz
AQ	3.6438515 sec
RG	71.8
DW	55.600 usec
DE	8.00 usec
TE	297.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	10.80 usec
PL1	3.00 dB
SFO1	300.1324010 MHz
SI	32768
SF	300.1300076 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	cxy-652
EXPNO	11
PROCNO	1
Date_	20111103
Time	21.04
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	28
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	812.7
DW	27.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.50 usec
PL1	2.00 dB
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	3.00 dB
PL12	22.33 dB
PL13	23.00 dB
SFO2	300.1312005 MHz
SI	32768
SF	75.4677436 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

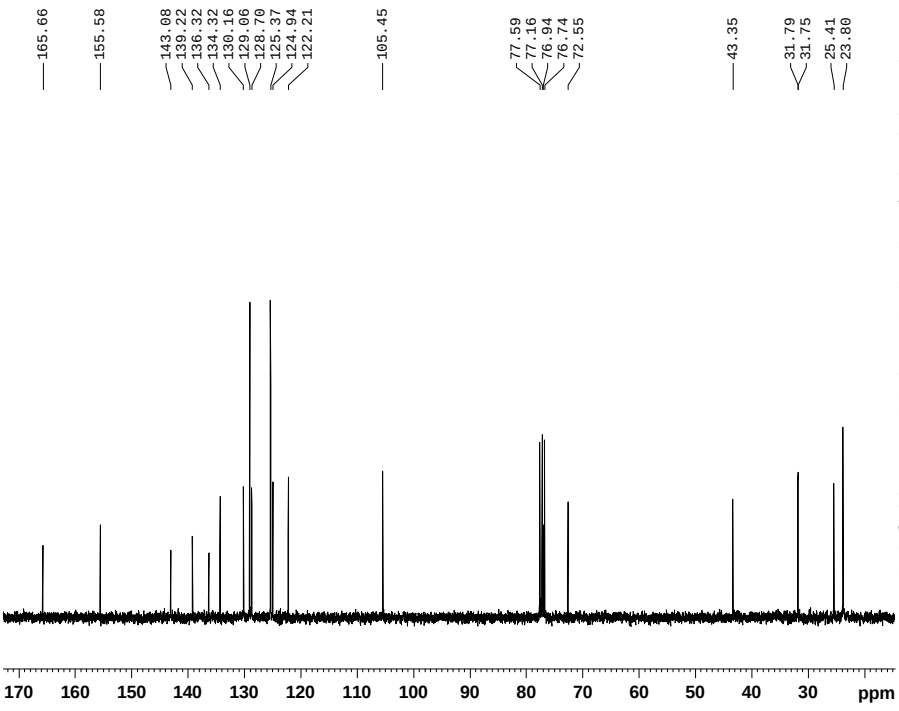


3k




```
NAME          cxy-668-a
EXPNO                10
PROCNO                1
Date_          20111117
Time              16.35
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   12
DS                    0
SWH            8992.806 Hz
FIDRES        0.137219 Hz
AQ           3.6438515 sec
RG                   64
DW              55.600 usec
DE               8.00 usec
TE               296.5 K
D1          1.00000000 sec
TD0                   1

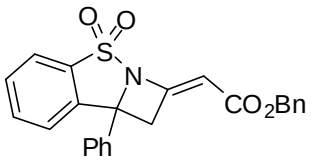
======== CHANNEL f1 ========
NUC1                 1H
P1                10.80 usec
PL1                3.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300062 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



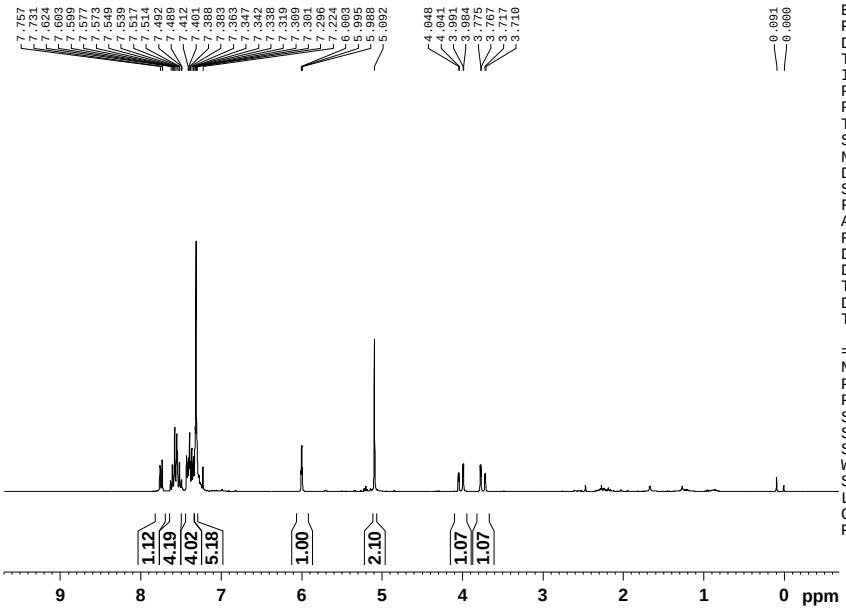

```
NAME          cxy-668-a
EXPNO                11
PROCNO                1
Date_          20111117
Time              16.38
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   31
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                322.5
DW               27.800 usec
DE                6.50 usec
TE                296.9 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1               12.50 usec
PL1               2.00 dB
SFO1         75.4752953 MHz

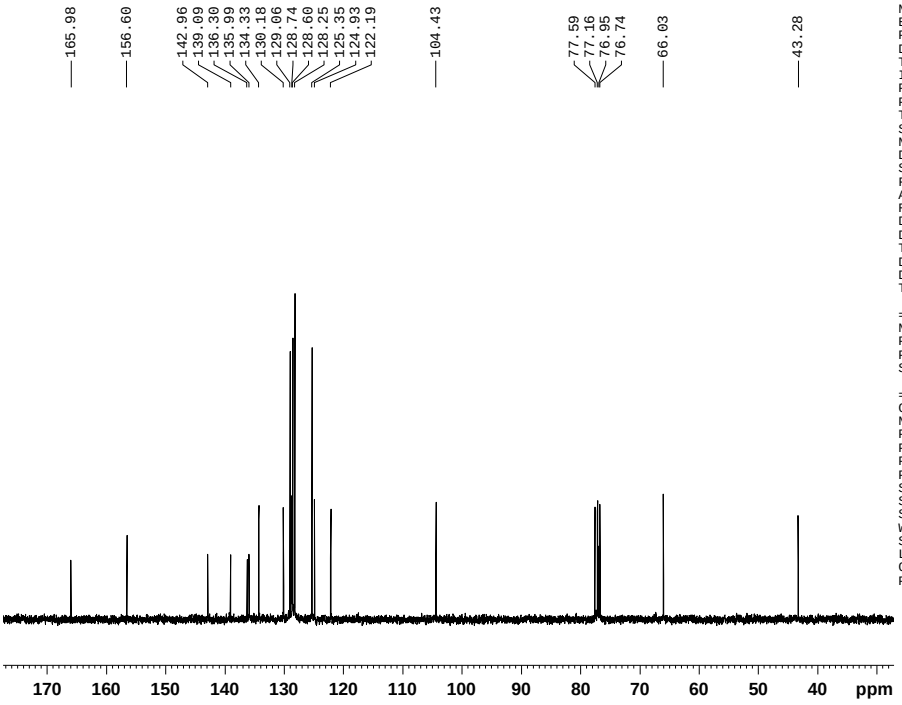
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2           100.00 usec
PL2               3.00 dB
PL12             22.33 dB
PL13             23.00 dB
SFO2        300.1312005 MHz
SI                32768
SF          75.4677445 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



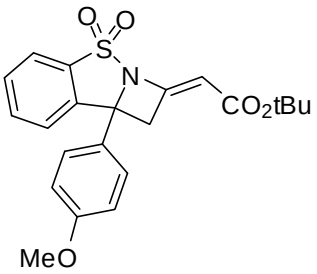
3l



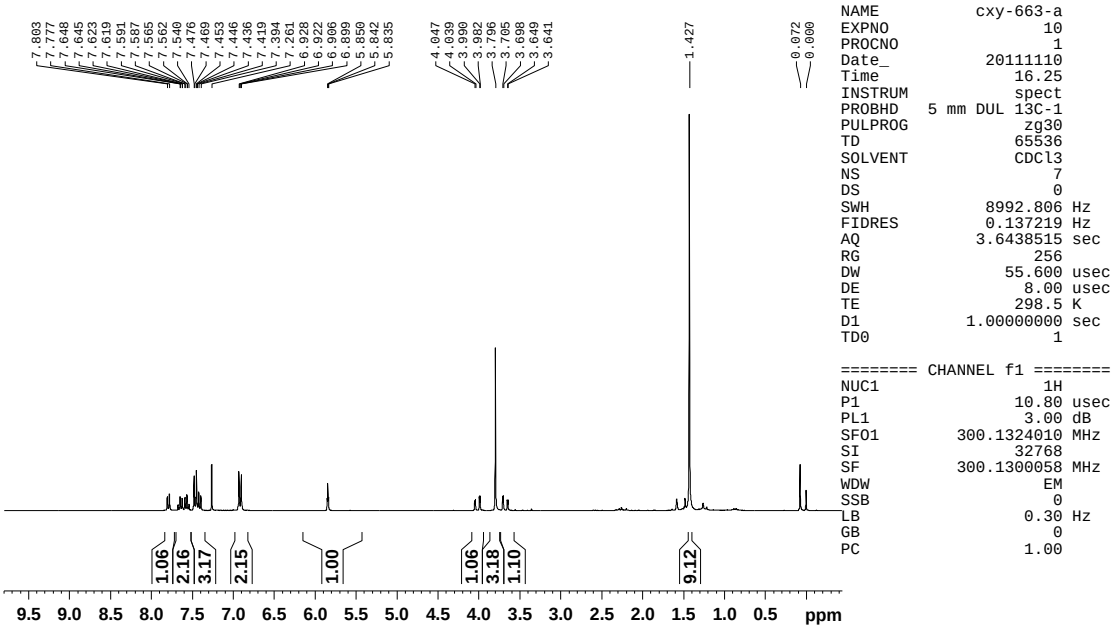
NAME	cxy-651
EXPNO	10
PROCNO	1
Date_	20111103
Time	16.31
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	10
DS	0
SWH	8992.806 Hz
FIDRES	0.137219 Hz
AQ	3.6438515 sec
RG	71.8
DW	55.600 usec
DE	8.00 usec
TE	298.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	10.80 usec
PL1	3.00 dB
SFO1	300.1324010 MHz
SI	32768
SF	300.1300166 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	cxy-651
EXPNO	11
PROCNO	1
Date_	20111103
Time	16.34
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	41
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	1625.5
DW	27.800 usec
DE	6.50 usec
TE	299.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.50 usec
PL1	2.00 dB
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	3.00 dB
PL12	22.33 dB
PL13	23.00 dB
SFO2	300.1312005 MHz
SI	32768
SF	75.4677472 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

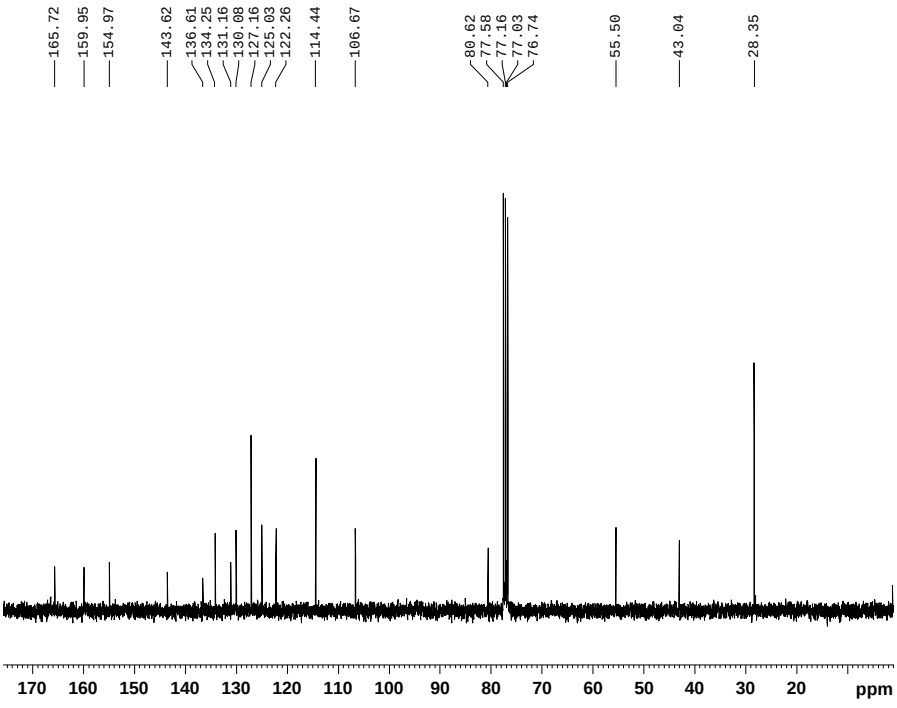


3m




```
NAME           cxy-663-a
EXPNO                 10
PROCNO                 1
Date_           20111110
Time               16.25
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     7
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   256
DW                55.600 usec
DE                 8.00 usec
TE                298.5 K
D1             1.00000000 sec
TD0                    1

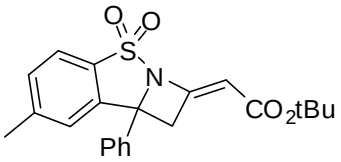
======== CHANNEL f1 ========
NUC1                  1H
P1                10.80 usec
PL1                3.00 dB
SFO1         300.1324010 MHz
SI                 32768
SF           300.1300058 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



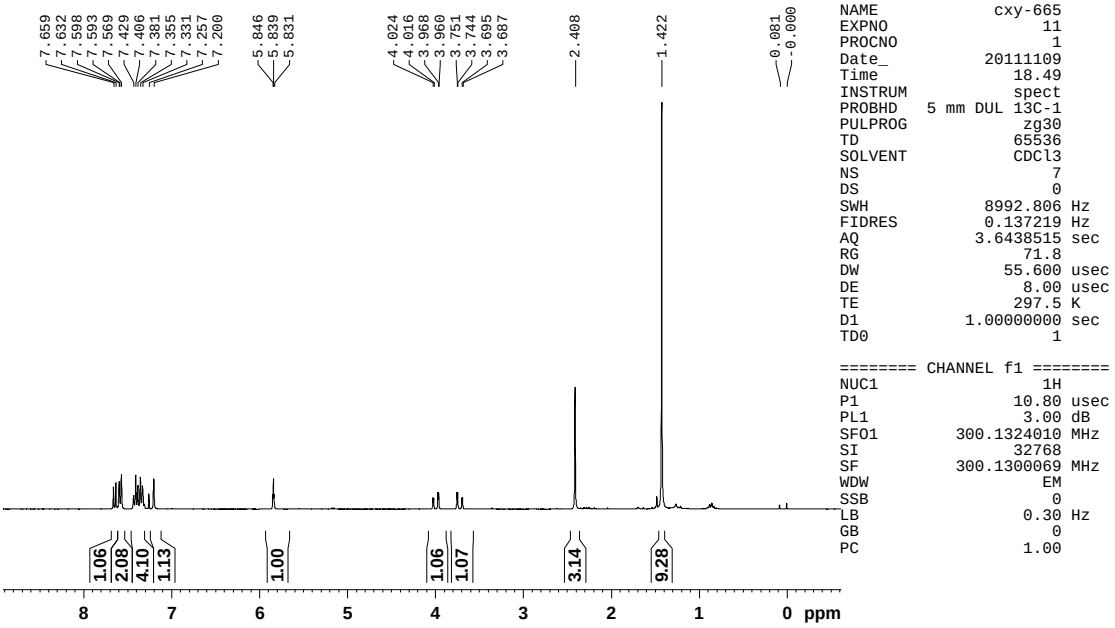

```
NAME           cxy-663-a
EXPNO                 11
PROCNO                 1
Date_           20111110
Time               16.27
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   118
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 322.5
DW                27.800 usec
DE                 6.50 usec
TE                298.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677383 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

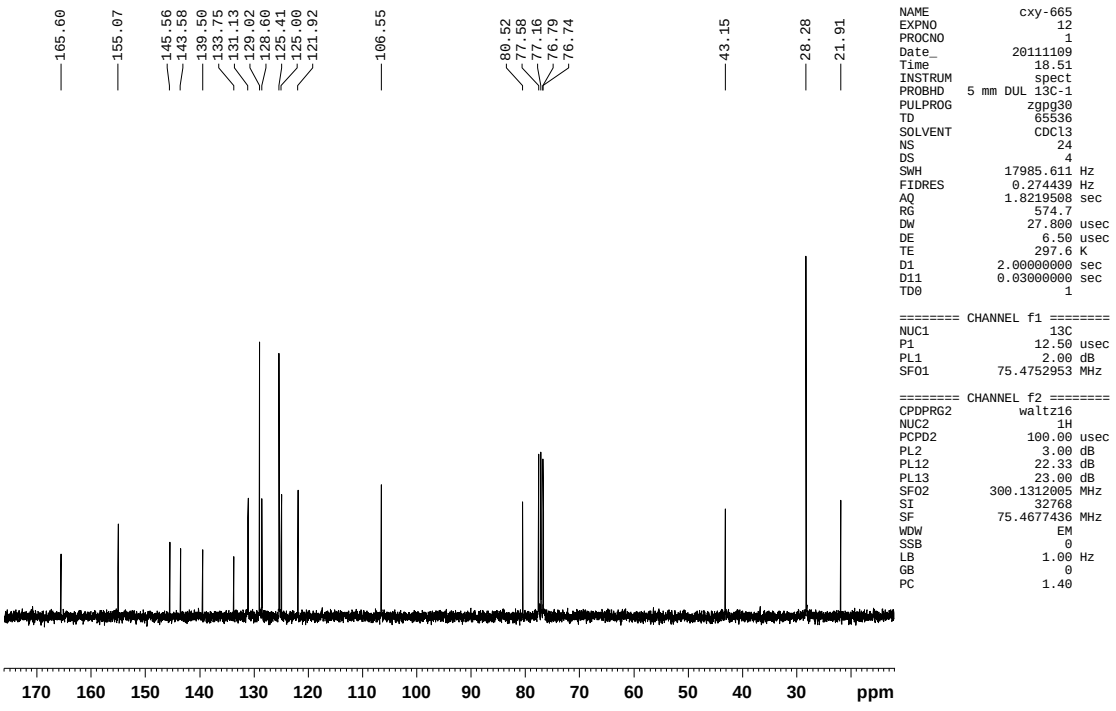


3a'




```
NAME            cxy-665
EXPNO                11
PROCNO                1
Date_          20111109
Time              18.49
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    7
DS                    0
SWH            8992.806 Hz
FIDRES        0.137219 Hz
AQ            3.6438515 sec
RG                 71.8
DW               55.600 usec
DE                8.00 usec
TE               297.5 K
D1           1.00000000 sec
TD0                   1

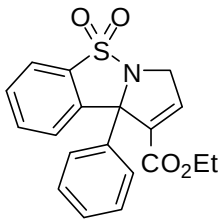
======== CHANNEL f1 ========
NUC1                 1H
P1                10.80 usec
PL1                3.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300069 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



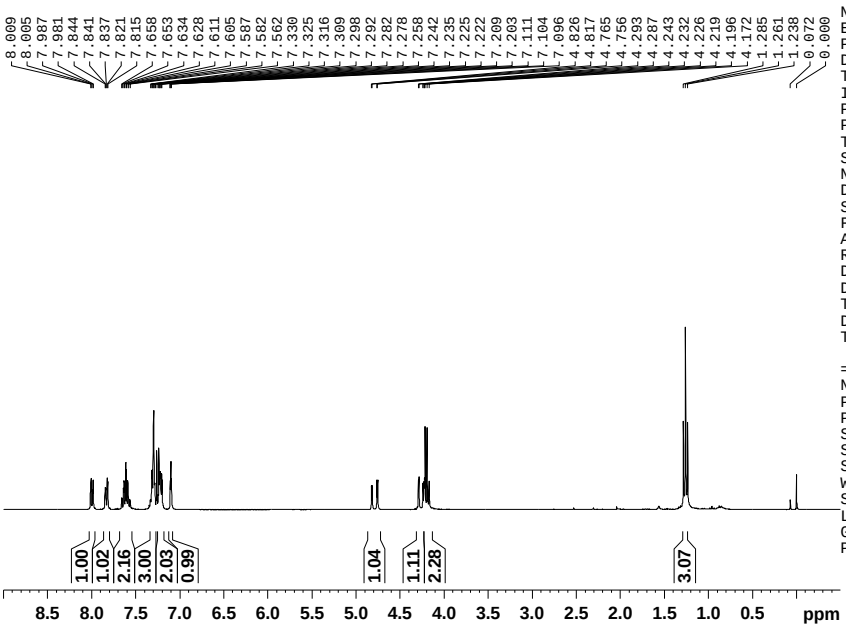

```
NAME            cxy-665
EXPNO                12
PROCNO                1
Date_          20111109
Time              18.51
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   24
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                574.7
DW               27.800 usec
DE                6.50 usec
TE               297.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                32768
SF           75.4677436 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

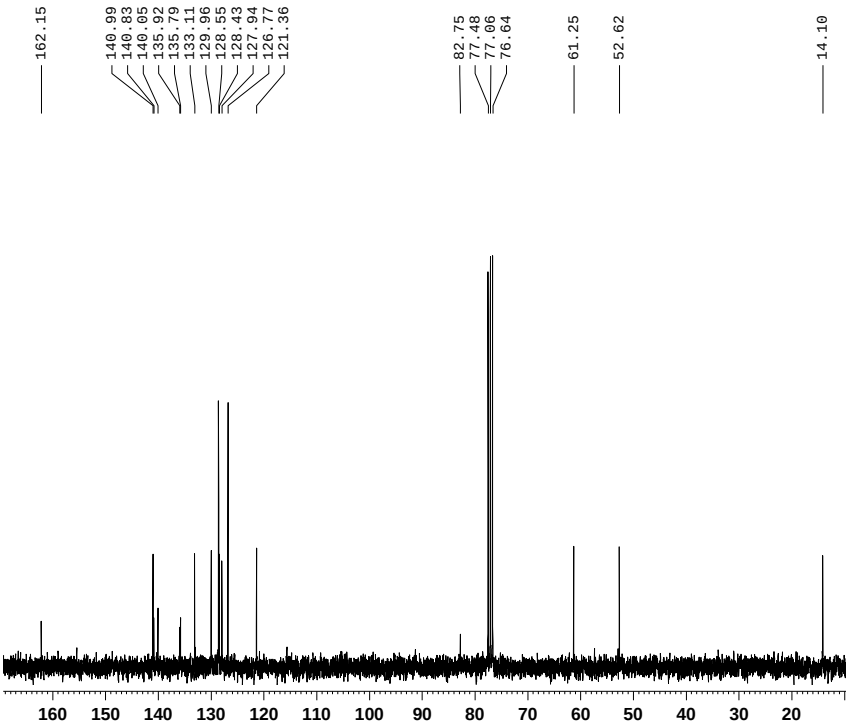


5a




```
NAME              cxy-349
EXPNO                  10
PROCNO                  1
Date_            20110224
Time                16.23
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      0
SWH           8992.806 Hz
FIDRES        0.137219 Hz
AQ            3.6438515 sec
RG                    256
DW               55.600 usec
DE                8.00 usec
TE                296.7 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.80 usec
PL1                 3.00 dB
SFO1         300.1324010 MHz
SI                  32768
SF           300.1300065 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```



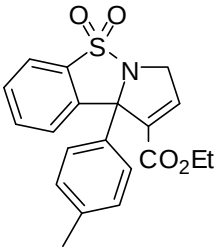

```
Current Data Parameters
NAME             cxy-349-c
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20110225
Time                 16.50
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     48
DS                      4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                   4096
DW                27.800 usec
DE                 8.00 usec
TE                 296.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

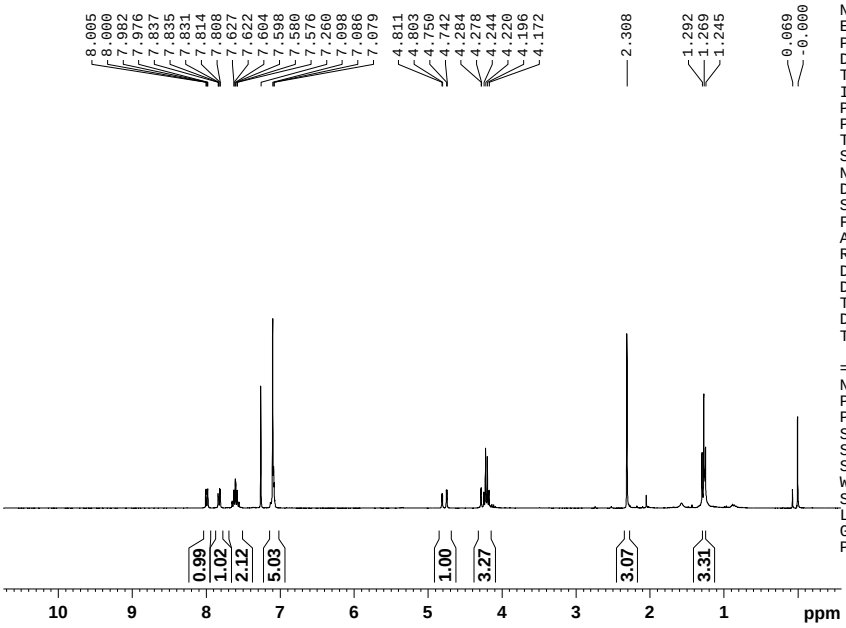
======== CHANNEL f1 ========
NUC1                   13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                 3.00 dB
PL12               22.33 dB
PL13               23.00 dB
SFO2          300.1312005 MHz

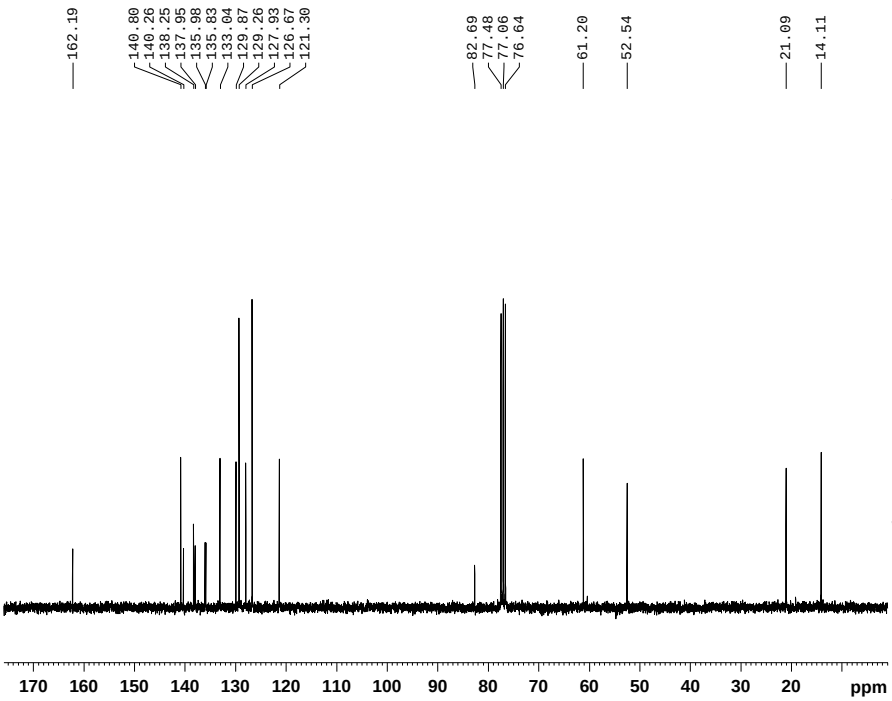
F2 - Processing parameters
SI                  32768
SF            75.4677478 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.40
```



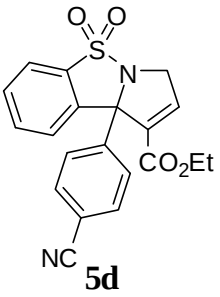
5b



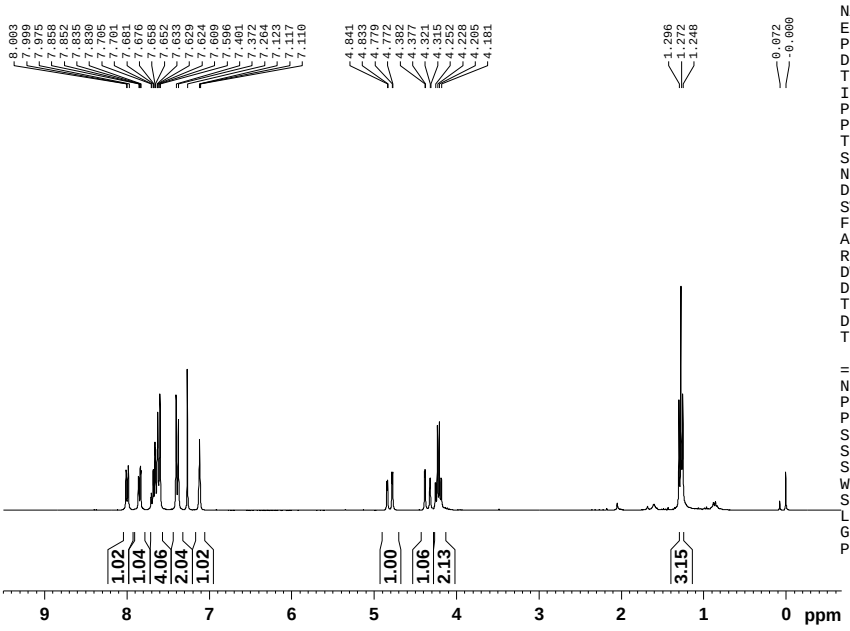
NAME	lrc-5-1
EXPNO	10
PROCNO	1
Date_	20110304
Time	16.23
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8992.806 Hz
FIDRES	0.137219 Hz
AQ	3.6438515 sec
RG	574.7
DW	55.600 usec
DE	8.00 usec
TE	295.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	10.80 usec
PL1	3.00 dB
SFO1	300.1324010 MHz
SI	32768
SF	300.1300061 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	lrc-5-1-c
EXPNO	10
PROCNO	1
Date_	20110415
Time	16.46
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	234
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	2048
DW	27.800 usec
DE	6.50 usec
TE	300.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.50 usec
PL1	2.00 dB
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	3.00 dB
PL12	22.33 dB
PL13	23.00 dB
SFO2	300.1312005 MHz
SI	32768
SF	75.4677474 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

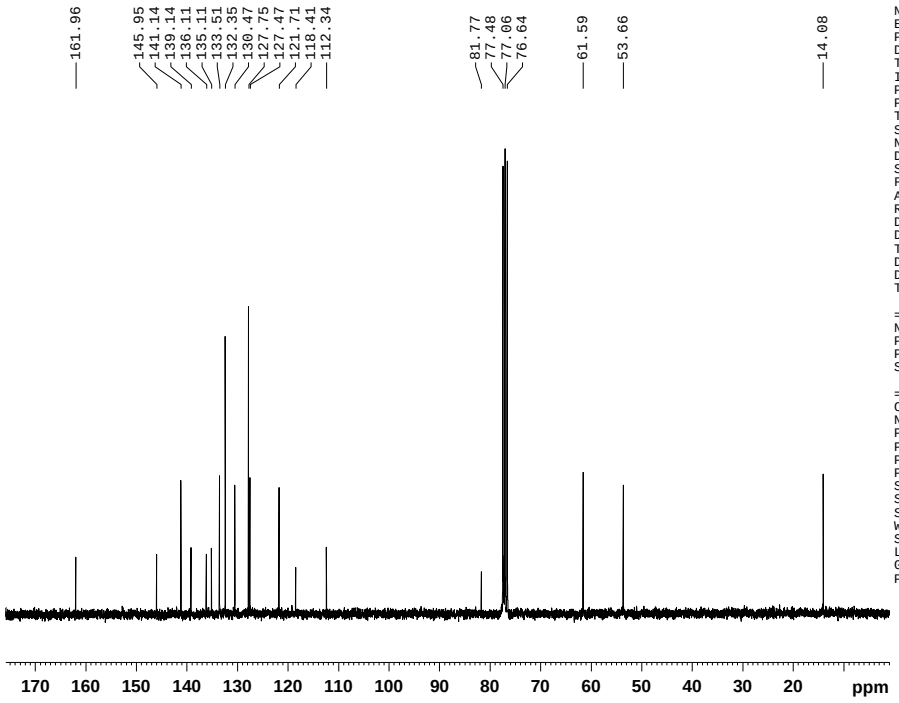


5d




```
NAME                lrc-4
EXPNO                  21
PROCNO                  1
Date_            20110302
Time                18.35
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8992.806 Hz
FIDRES           0.137219 Hz
AQ               3.6438515 sec
RG                    256
DW                 55.600 usec
DE                  8.00 usec
TE                 295.7 K
D1              1.00000000 sec
TD0                     1

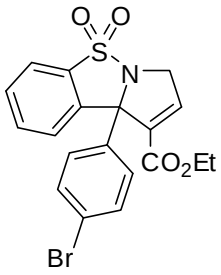
======== CHANNEL f1 ========
NUC1                   1H
P1                 10.80 usec
PL1                 3.00 dB
SFO1         300.1324010 MHz
SI                  32768
SF           300.1300046 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```



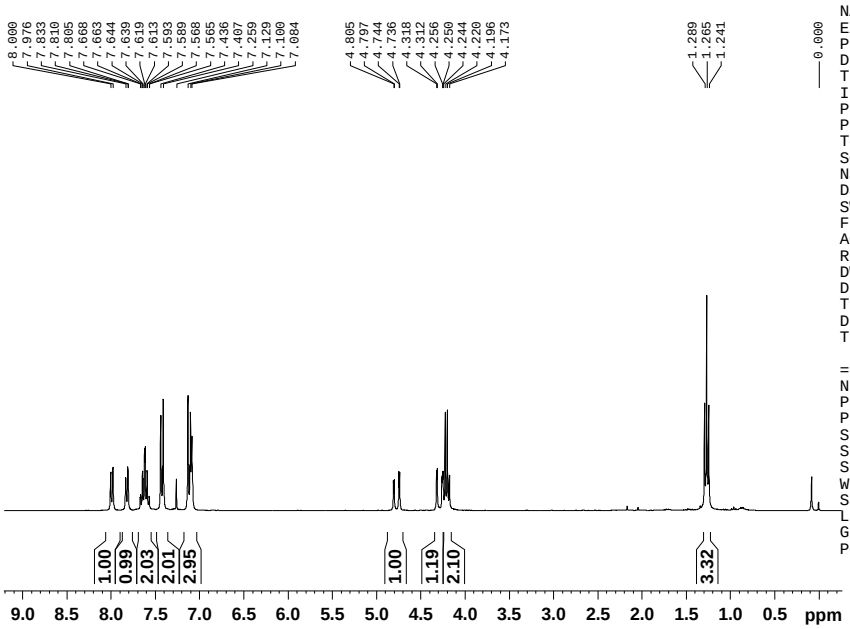

```
NAME             lrc-4-c
EXPNO                 10
PROCNO                 1
Date_           20110304
Time               20.50
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  409
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG               11585.2
DW                27.800 usec
DE                 8.00 usec
TE                295.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2        300.1312005 MHz
SI                32768
SF           75.4677483 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

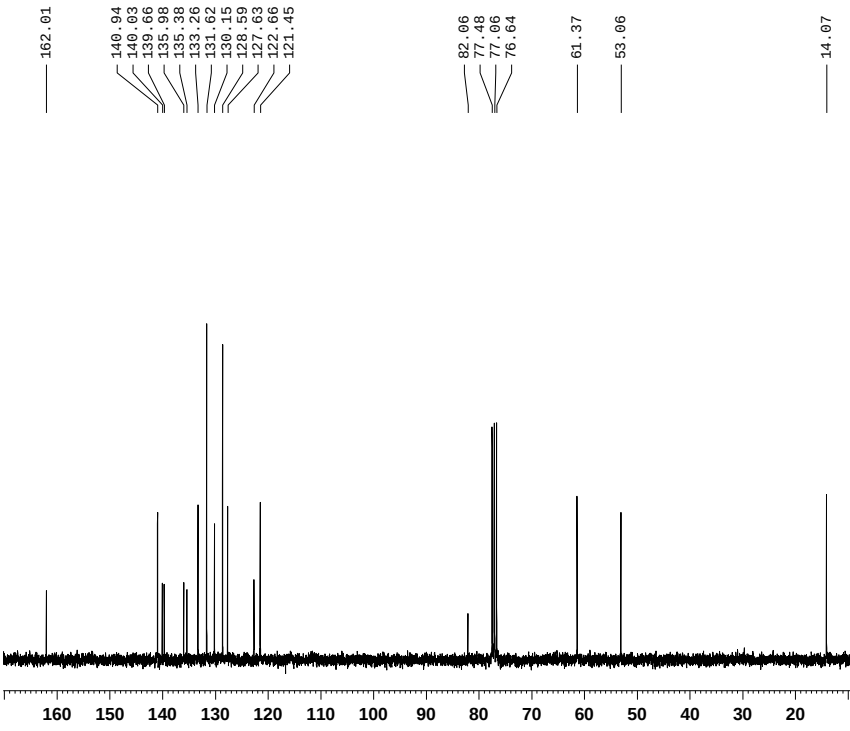


5e




```
NAME           cxy-353-3
EXPNO                 10
PROCNO                 1
Date_           20110413
Time                8.12
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   128
DW                55.600 usec
DE                 8.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.80 usec
PL1                3.00 dB
SFO1         300.1324010 MHz
SI                 32768
SF           300.1300061 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



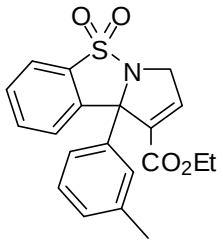

```
Current Data Parameters
NAME           cxy-353-3
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110413
Time                8.14
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    51
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 812.7
DW                27.800 usec
DE                 6.50 usec
TE                298.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

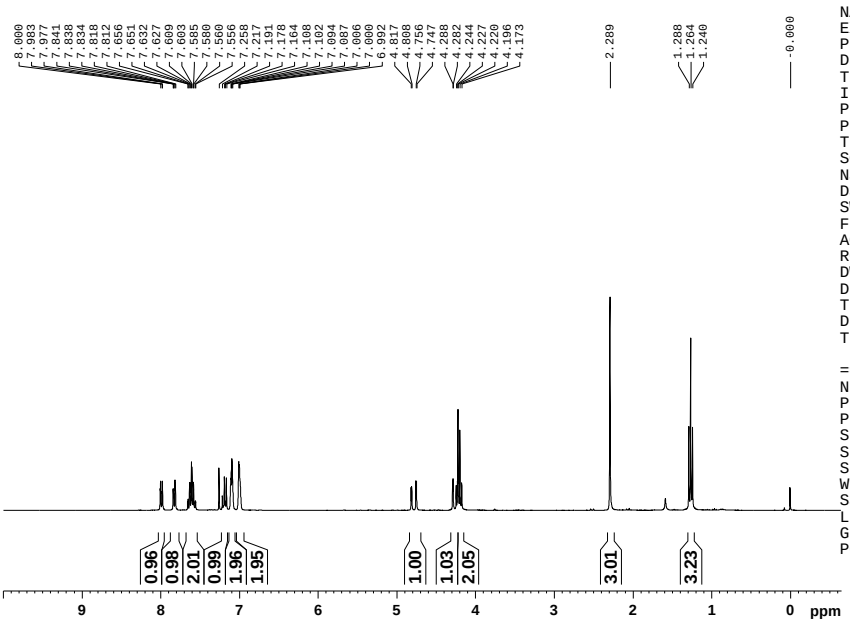
======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF           75.4677507 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

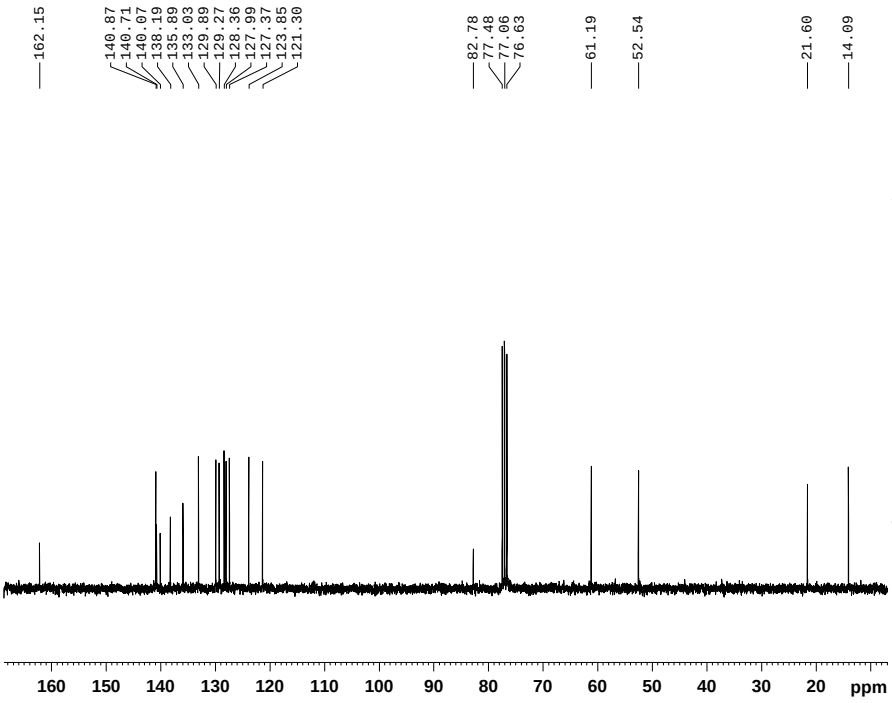


5f




```
NAME          cxy-lrc-5-2
EXPNO                  10
PROCNO                  1
Date_            20110401
Time                16.19
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                 228.1
DW                55.600 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                     1

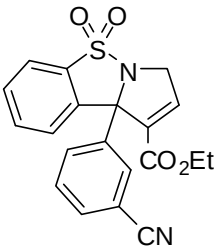
======== CHANNEL f1 ========
NUC1                   1H
P1                 10.80 usec
PL1                 3.00 dB
SFO1          300.1324010 MHz
SI                  32768
SF            300.1300067 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```



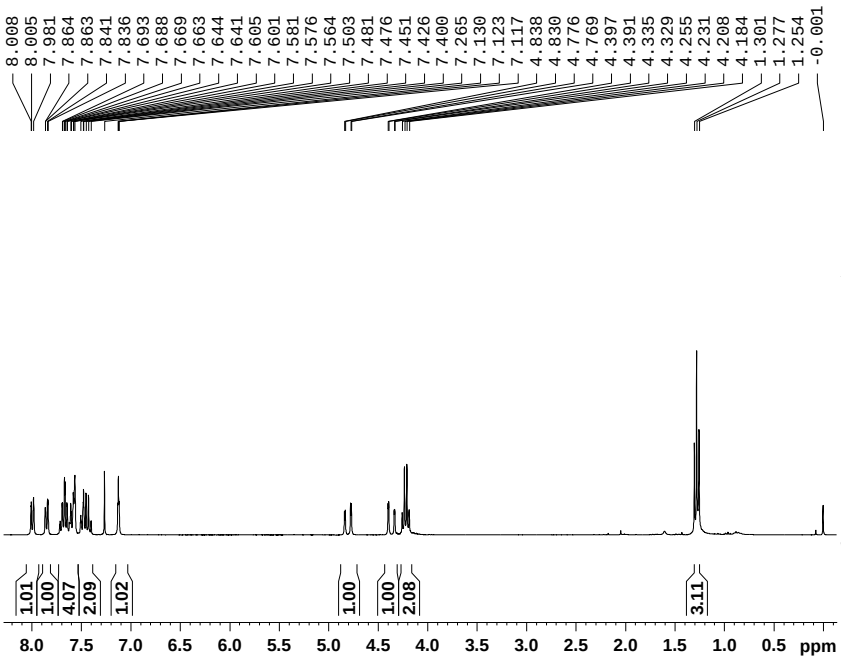

```
NAME      cxy-lrc-5-2-4.20
EXPNO                   11
PROCNO                   1
Date_             20110420
Time                 8.20
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    103
DS                      4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                1290.2
DW                27.800 usec
DE                 6.50 usec
TE                299.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                12.50 usec
PL1                2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2          300.1312005 MHz
SI                  32768
SF            75.4677488 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.40
```



5g

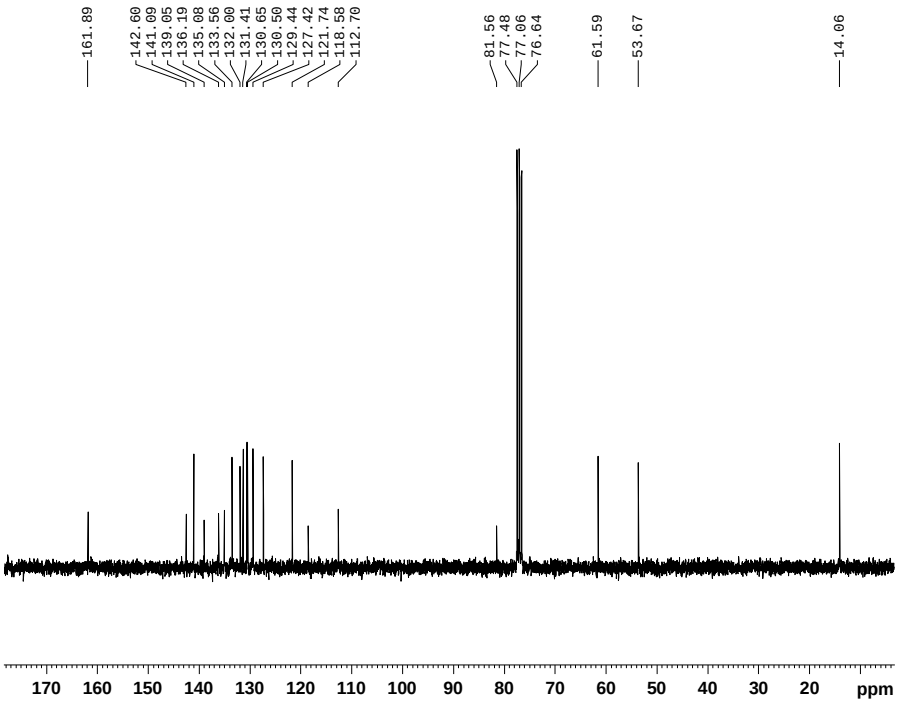



```
Current Data Parameters
NAME         cxy-370-3.4
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110304
Time               15.53
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    11
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   256
DW                55.600 usec
DE                 8.00 usec
TE                295.6 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.80 usec
PL1                 3.00 dB
SFO1         300.1324010 MHz

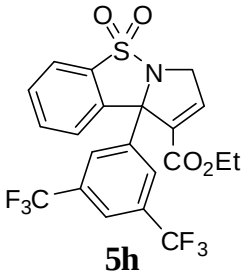
F2 - Processing parameters
SI                 32768
SF          300.1300044 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



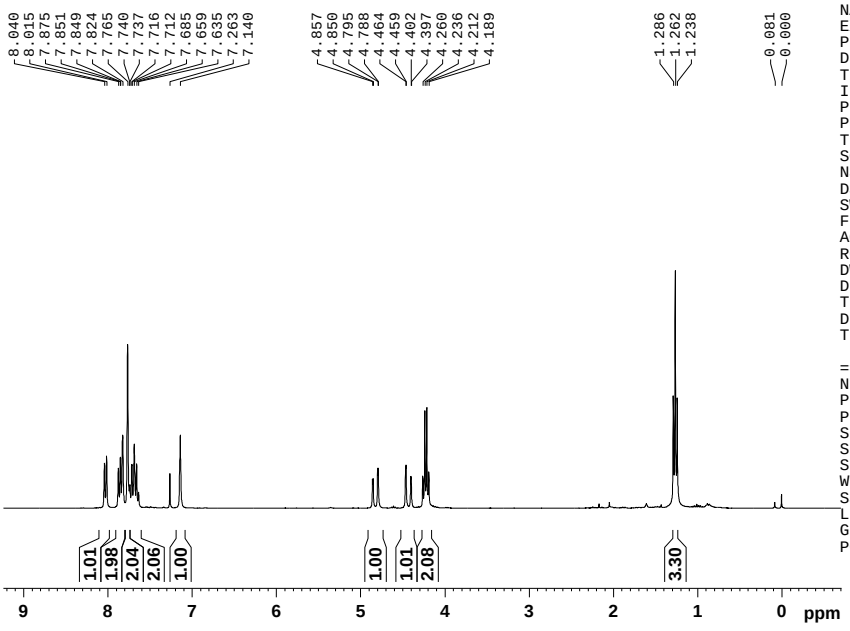

```
NAME       cxy-370-3.4-c
EXPNO                 10
PROCNO                 1
Date_           20110304
Time               15.55
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    79
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                9195.2
DW                27.800 usec
DE                 8.00 usec
TE                295.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 3.00 dB
PL12               22.33 dB
PL13               23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677488 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

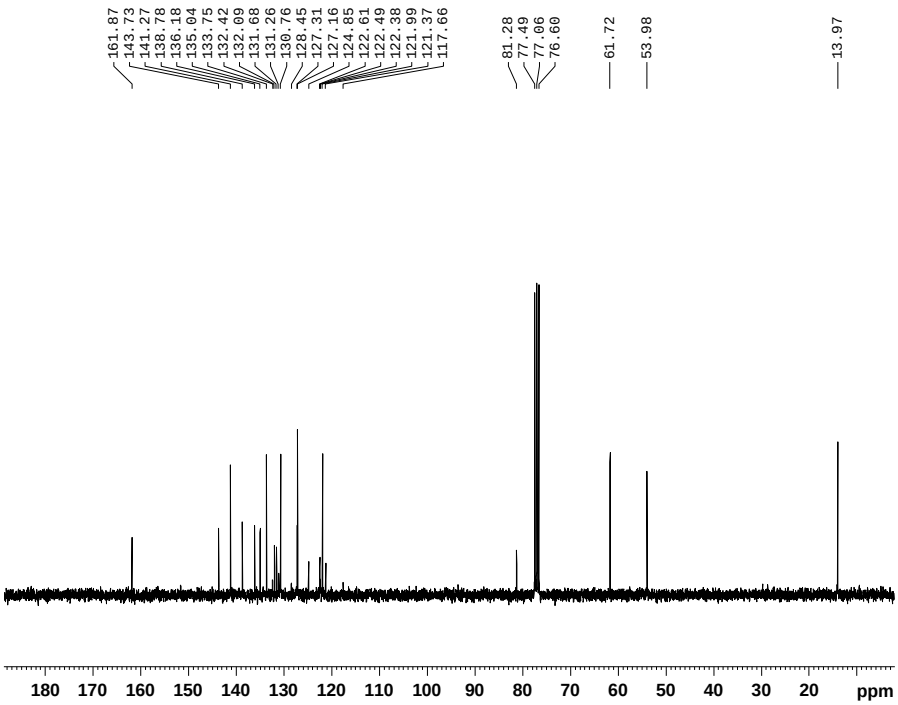


5h




```
NAME          cxy-371-3.4
EXPNO                  11
PROCNO                  1
Date_            20110304
Time                16.05
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     11
DS                      0
SWH              8992.806 Hz
FIDRES           0.137219 Hz
AQ               3.6438515 sec
RG                    128
DW                 55.600 usec
DE                  8.00 usec
TE                 295.7 K
D1              1.00000000 sec
TD0                     1

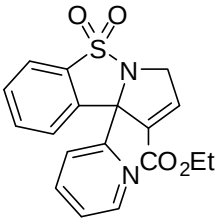
======== CHANNEL f1 ========
NUC1                   1H
P1                 10.80 usec
PL1                 3.00 dB
SFO1          300.1324010 MHz
SI                  32768
SF            300.1300050 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```



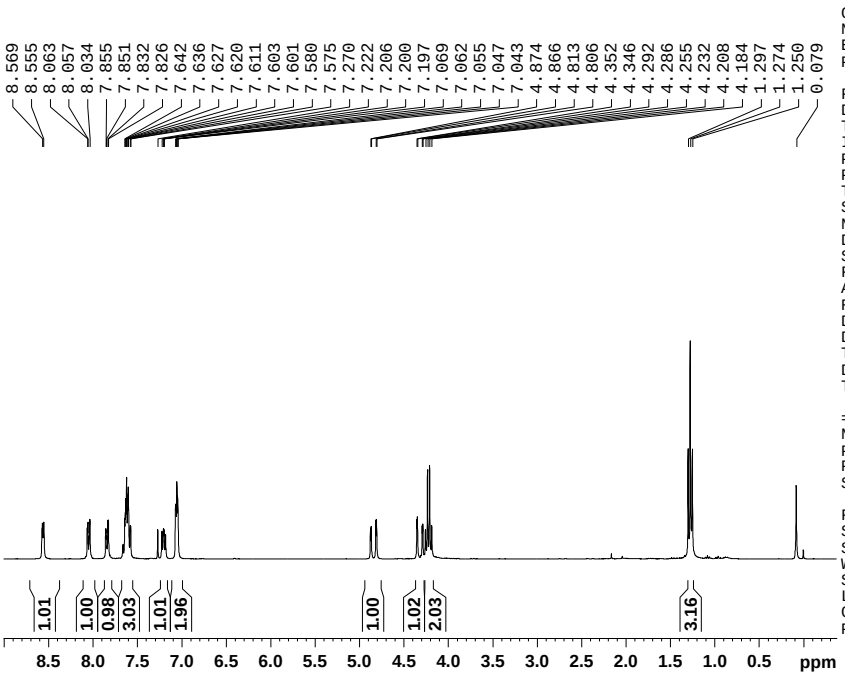

```
NAME       cxy-371-3.4-c
EXPNO                 10
PROCNO                 1
Date_           20110304
Time               16.07
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                   73
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG               11585.2
DW                27.800 usec
DE                 8.00 usec
TE                295.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2          300.1312005 MHz
SI                 32768
SF            75.4677478 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```



5i

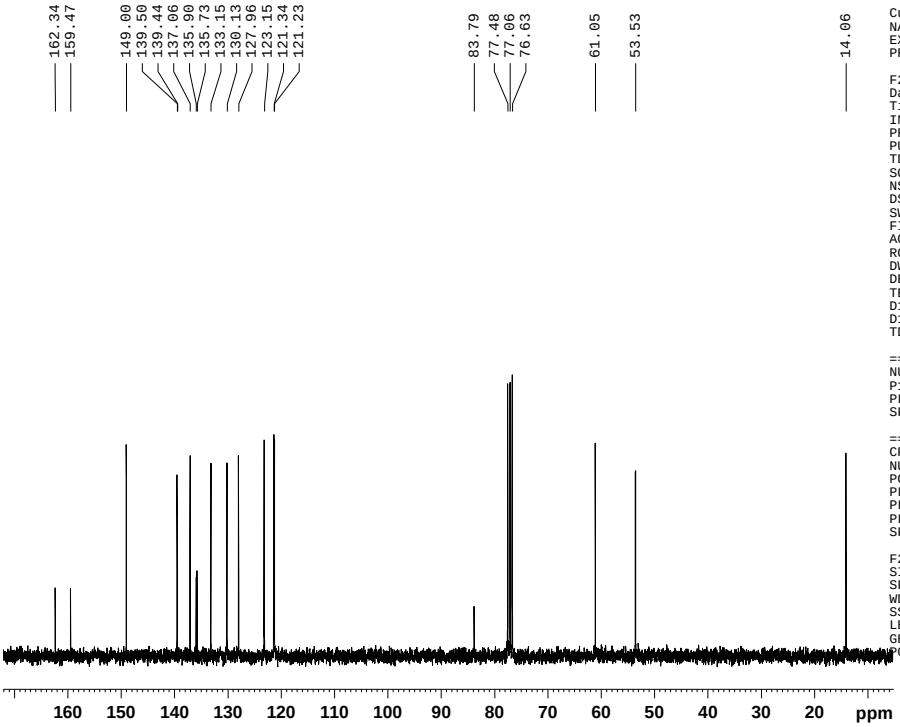



```
Current Data Parameters
NAME          cxy-365-3
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20110413
Time               8.20
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    9
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ             3.6438515 sec
RG                  128
DW                55.600 usec
DE                 8.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.80 usec
PL1                3.00 dB
SFO1        300.1324010 MHz

F2 - Processing parameters
SI                32768
SF          300.1300029 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



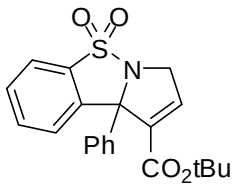

```
Current Data Parameters
NAME          cxy-365-3
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20110413
Time               8.23
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   62
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                574.7
DW               27.800 usec
DE                6.50 usec
TE                298.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

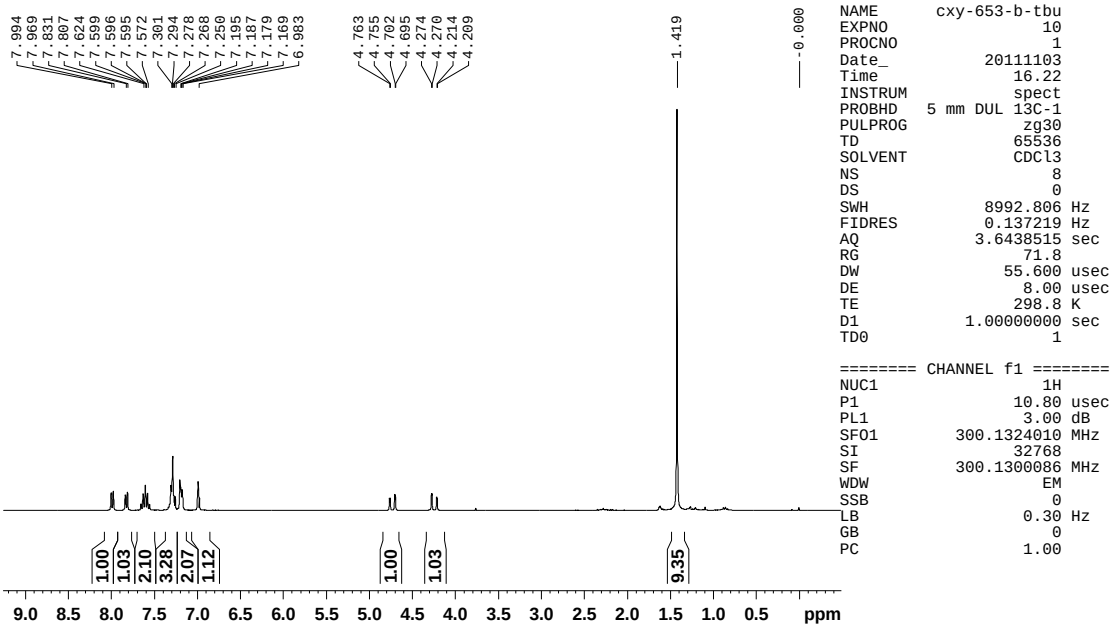
======== CHANNEL f1 ========
NUC1                13C
P1               12.50 usec
PL1                2.00 dB
SFO1        75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2           100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2       300.1312005 MHz

F2 - Processing parameters
SI                32768
SF          75.4677509 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

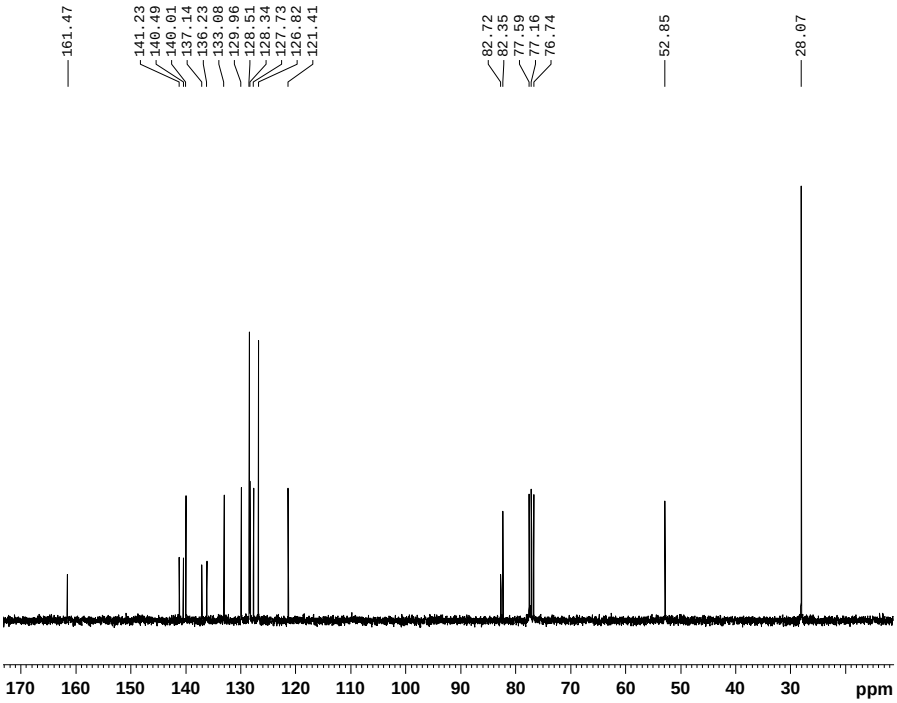


5j




```
NAME          cxy-653-b-tbu
EXPNO                    10
PROCNO                    1
Date_              20111103
Time                  16.22
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8992.806 Hz
FIDRES            0.137219 Hz
AQ                3.6438515 sec
RG                     71.8
DW                  55.600 usec
DE                   8.00 usec
TE                  298.8 K
D1                1.00000000 sec
TD0                       1

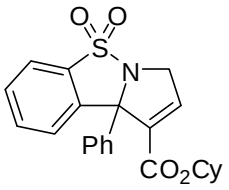
======== CHANNEL f1 ========
NUC1                     1H
P1                   10.80 usec
PL1                   3.00 dB
SFO1            300.1324010 MHz
SI                    32768
SF              300.1300086 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                    1.00
```



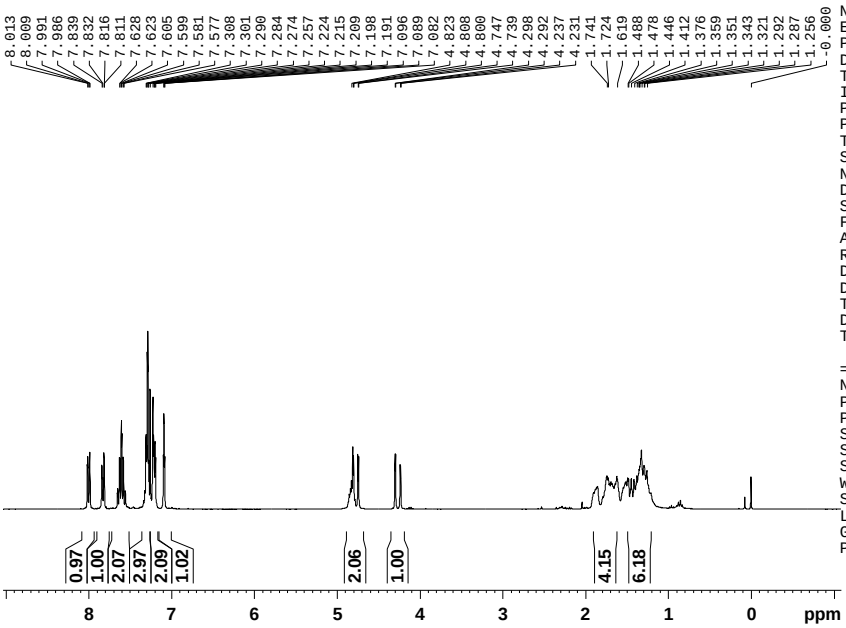

```
NAME          cxy-653-b-tbu
EXPNO                    11
PROCNO                    1
Date_              20111103
Time                  16.26
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       46
DS                        4
SWH               17985.611 Hz
FIDRES            0.274439 Hz
AQ                1.8219508 sec
RG                    574.7
DW                  27.800 usec
DE                   6.50 usec
TE                  299.1 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   12.50 usec
PL1                   2.00 dB
SFO1            75.4752953 MHz

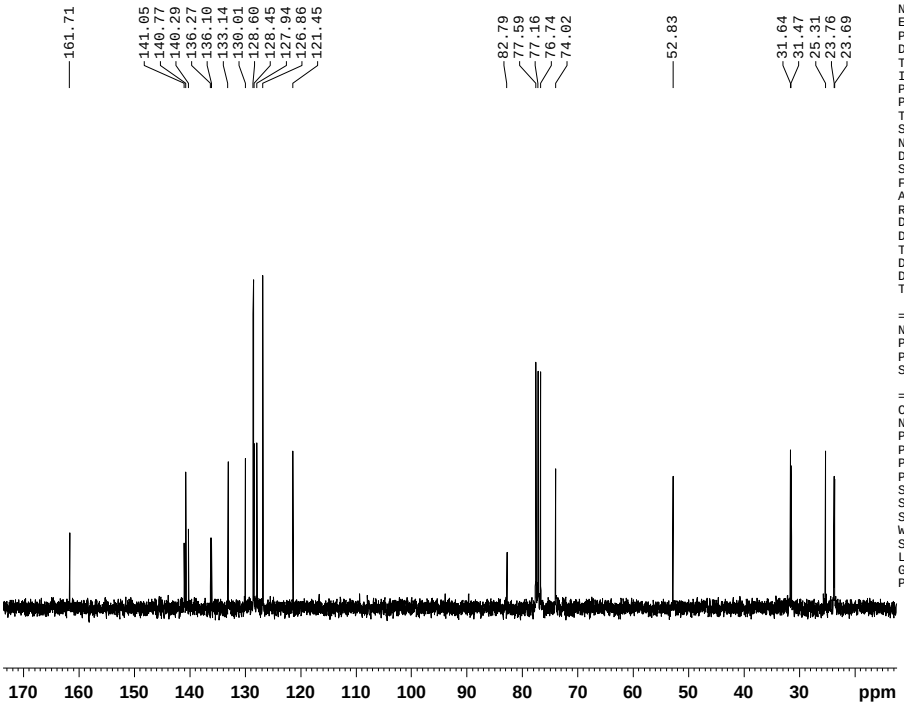
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2               100.00 usec
PL2                   3.00 dB
PL12                 22.33 dB
PL13                 23.00 dB
SFO2            300.1312005 MHz
SI                    32768
SF              75.4677445 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```



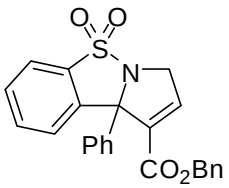
5k



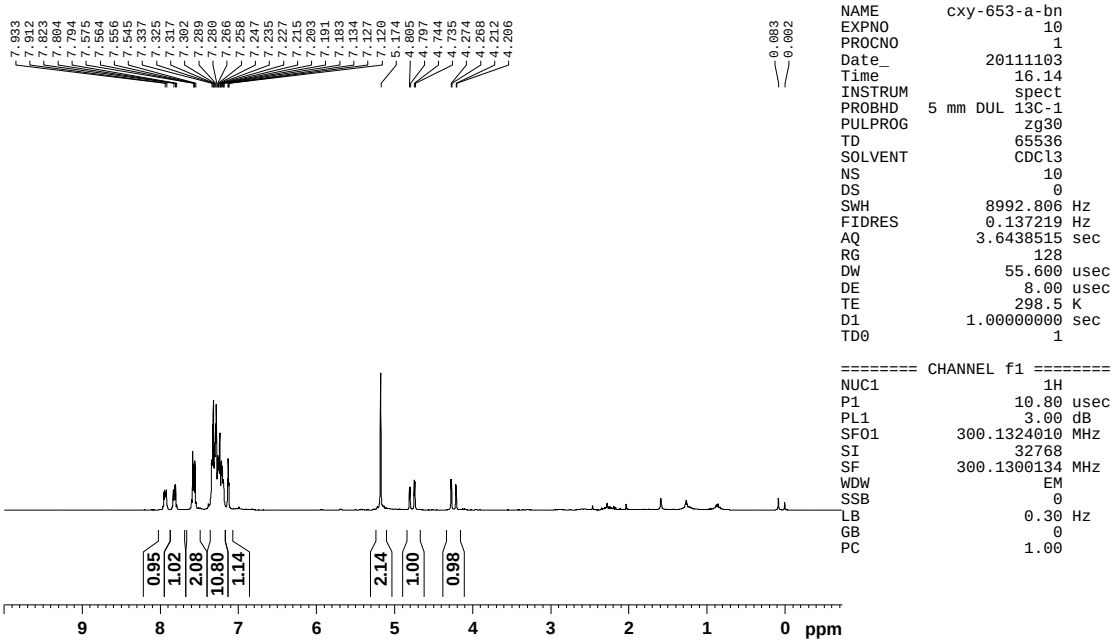
NAME	cxy-668-b
EXPNO	10
PROCNO	1
Date_	20111117
Time	16.44
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	11
DS	0
SWH	8992.806 Hz
FIDRES	0.137219 Hz
AQ	3.6438515 sec
RG	128
DW	55.600 usec
DE	8.00 usec
TE	296.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	10.80 usec
PL1	3.00 dB
SFO1	300.1324010 MHz
SI	32768
SF	300.1300069 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	cxy-668-b
EXPNO	11
PROCNO	1
Date_	20111117
Time	16.47
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	63
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	456.1
DW	27.800 usec
DE	6.50 usec
TE	297.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.50 usec
PL1	2.00 dB
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	3.00 dB
PL12	22.33 dB
PL13	23.00 dB
SFO2	300.1312005 MHz
SI	32768
SF	75.4677420 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

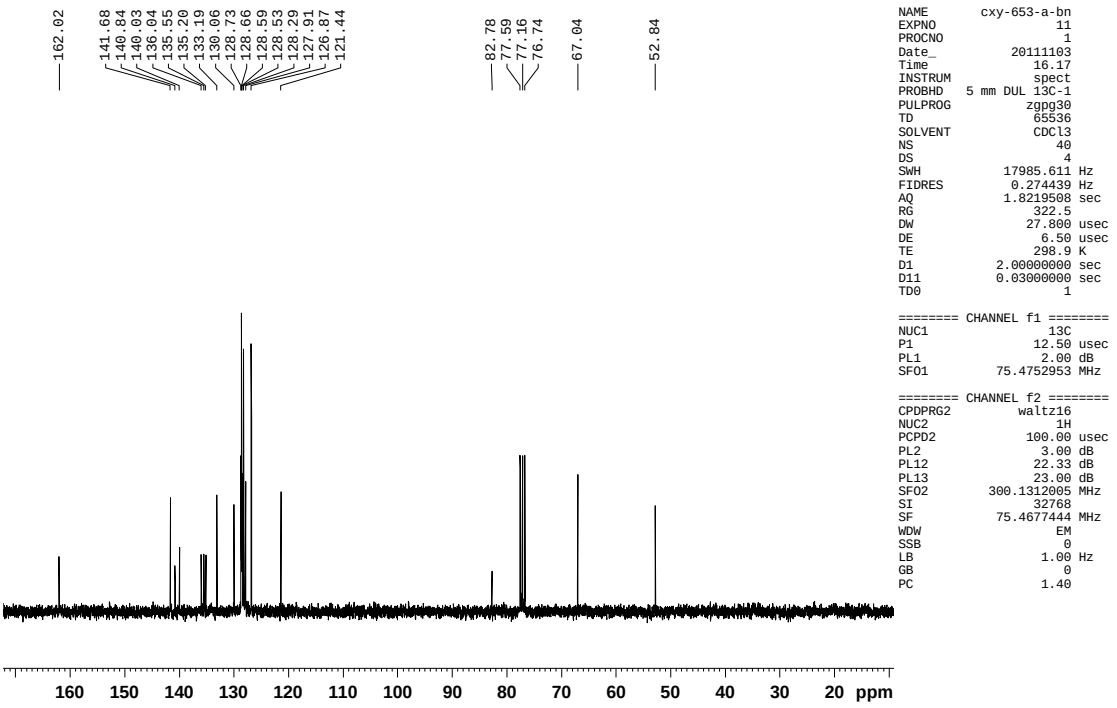


5l




```
NAME          cxy-653-a-bn
EXPNO                   10
PROCNO                   1
Date_             20111103
Time                 16.14
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      10
DS                       0
SWH               8992.806 Hz
FIDRES            0.137219 Hz
AQ                3.6438515 sec
RG                     128
DW                  55.600 usec
DE                   8.00 usec
TE                  298.5 K
D1                1.00000000 sec
TD0                      1

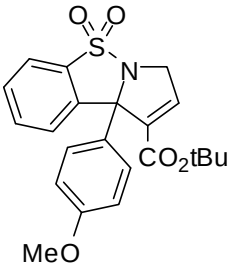
======== CHANNEL f1 ========
NUC1                    1H
P1                   10.80 usec
PL1                   3.00 dB
SFO1           300.1324010 MHz
SI                   32768
SF             300.1300134 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



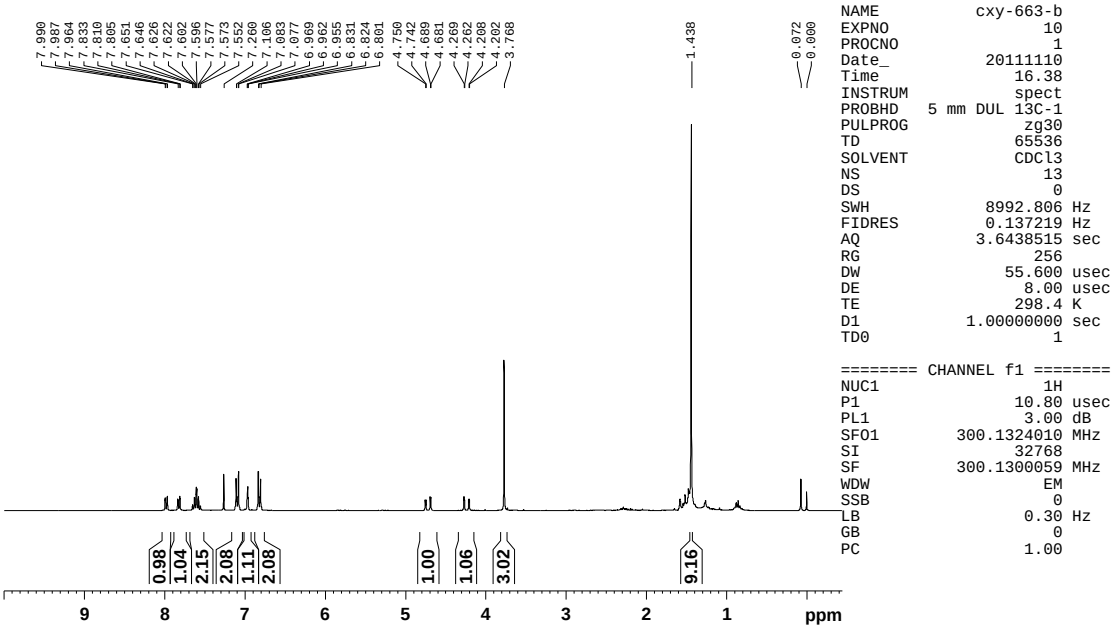

```
NAME          cxy-653-a-bn
EXPNO                   11
PROCNO                   1
Date_             20111103
Time                 16.17
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      40
DS                       4
SWH              17985.611 Hz
FIDRES            0.274439 Hz
AQ                1.8219508 sec
RG                   322.5
DW                  27.800 usec
DE                   6.50 usec
TE                  298.9 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.50 usec
PL1                   2.00 dB
SFO1           75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                   3.00 dB
PL12                 22.33 dB
PL13                 23.00 dB
SFO2           300.1312005 MHz
SI                   32768
SF             75.4677444 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

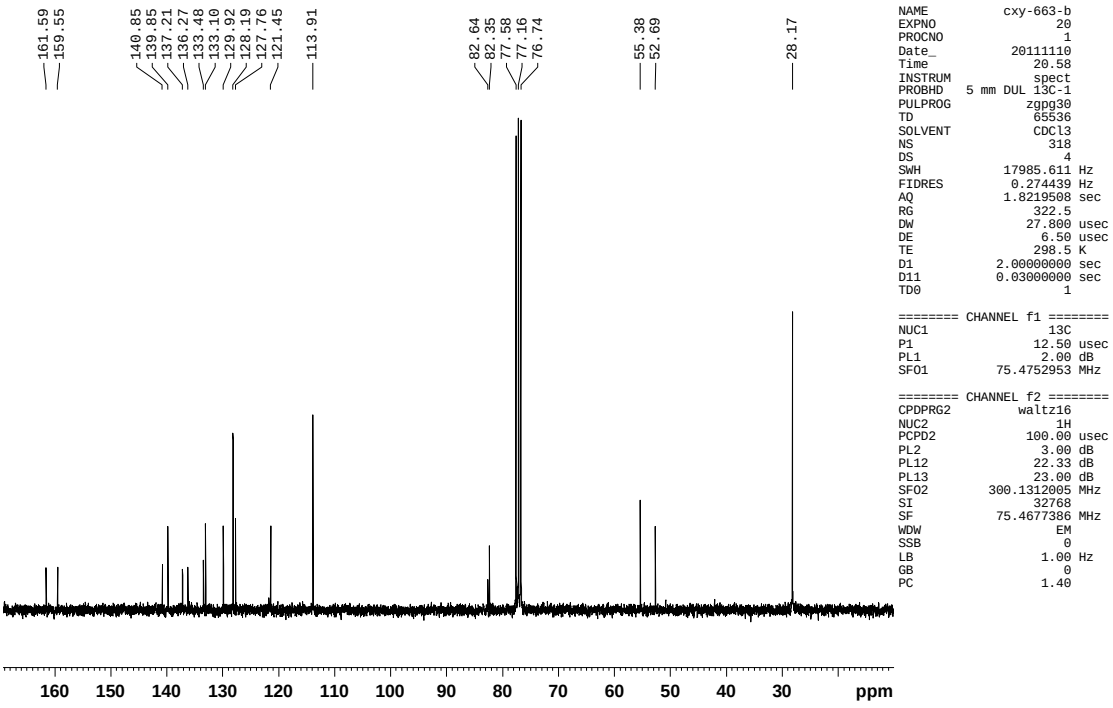


5m




```
NAME          cxy-663-b
EXPNO                10
PROCNO                1
Date_          20111110
Time              16.38
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   13
DS                    0
SWH            8992.806 Hz
FIDRES        0.137219 Hz
AQ            3.6438515 sec
RG                  256
DW               55.600 usec
DE                8.00 usec
TE               298.4 K
D1           1.00000000 sec
TD0                   1

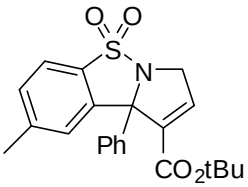
======== CHANNEL f1 ========
NUC1                 1H
P1                10.80 usec
PL1                3.00 dB
SFO1        300.1324010 MHz
SI                 32768
SF          300.1300059 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



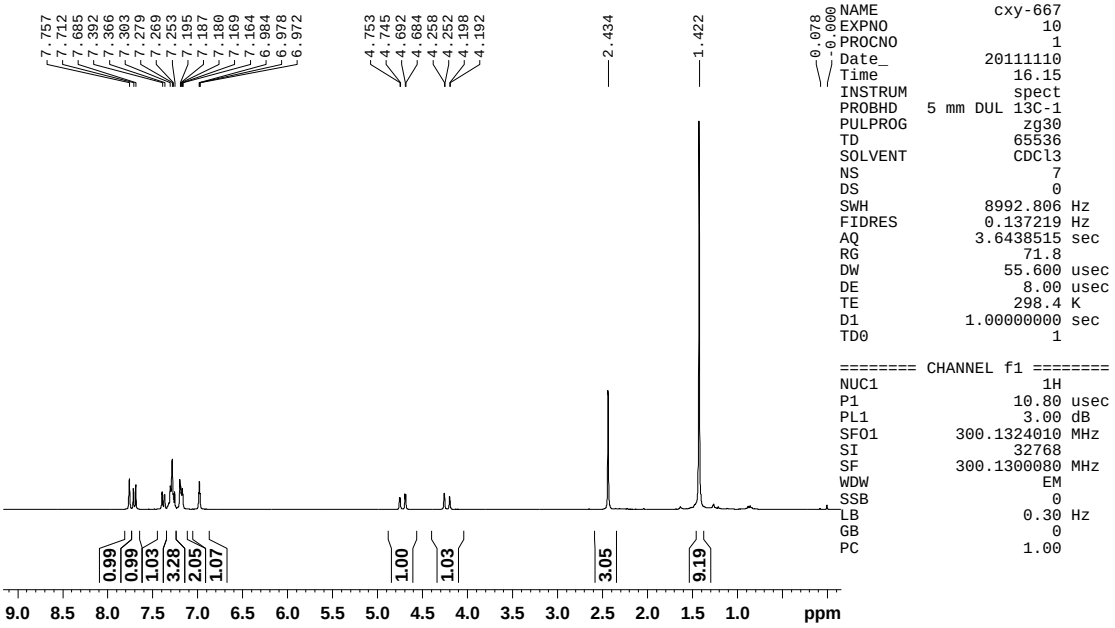

```
NAME          cxy-663-b
EXPNO                20
PROCNO                1
Date_          20111110
Time              20.58
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  318
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                322.5
DW               27.800 usec
DE                6.50 usec
TE               298.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2           100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677386 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

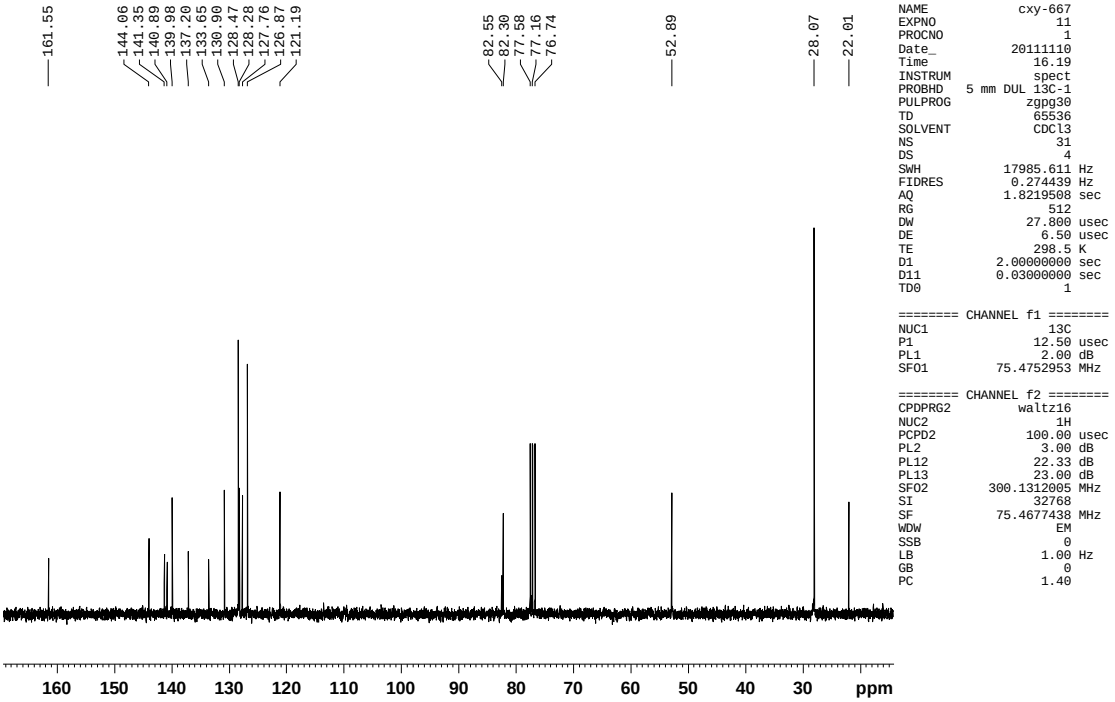


5a'




```
NAME              cxy-667
EXPNO                  10
PROCNO                  1
Date_            20111110
Time                16.15
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      7
DS                      0
SWH              8992.806 Hz
FIDRES           0.137219 Hz
AQ               3.6438515 sec
RG                   71.8
DW                 55.600 usec
DE                  8.00 usec
TE                 298.4 K
D1              1.00000000 sec
TD0                     1

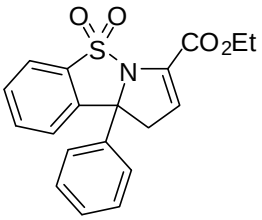
======== CHANNEL f1 ========
NUC1                   1H
P1                 10.80 usec
PL1                 3.00 dB
SFO1         300.1324010 MHz
SI                  32768
SF           300.1300080 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```




```
NAME              cxy-667
EXPNO                  11
PROCNO                  1
Date_            20111110
Time                16.19
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     31
DS                      4
SWH             17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                    512
DW                 27.800 usec
DE                  6.50 usec
TE                 298.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                 3.00 dB
PL12               22.33 dB
PL13               23.00 dB
SFO2         300.1312005 MHz
SI                  32768
SF           75.4677438 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.40
```


O
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S
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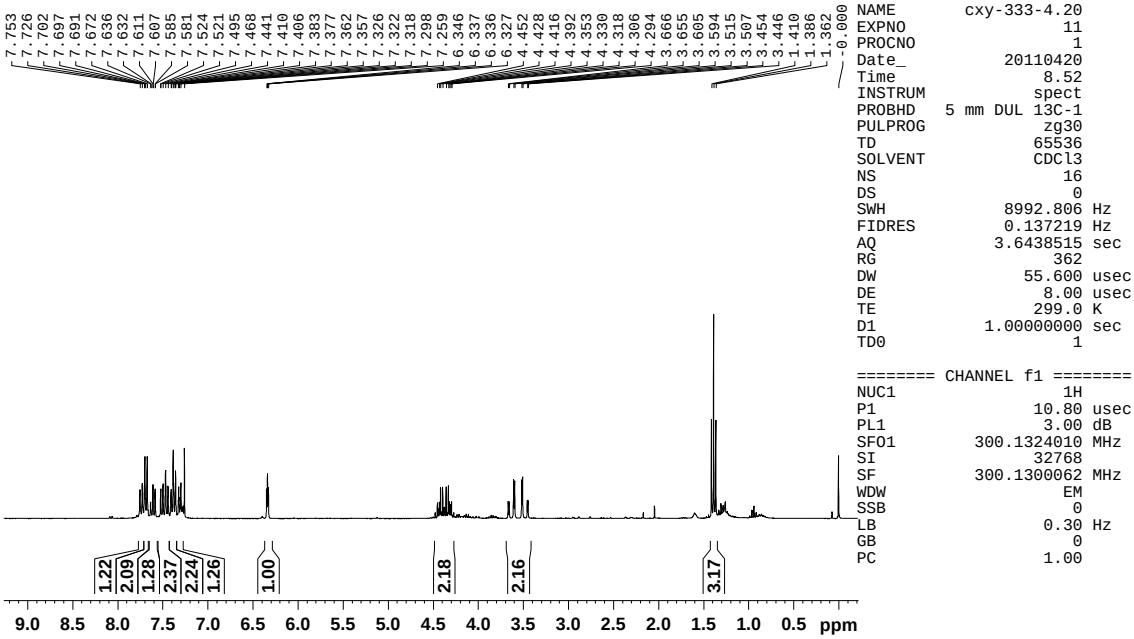
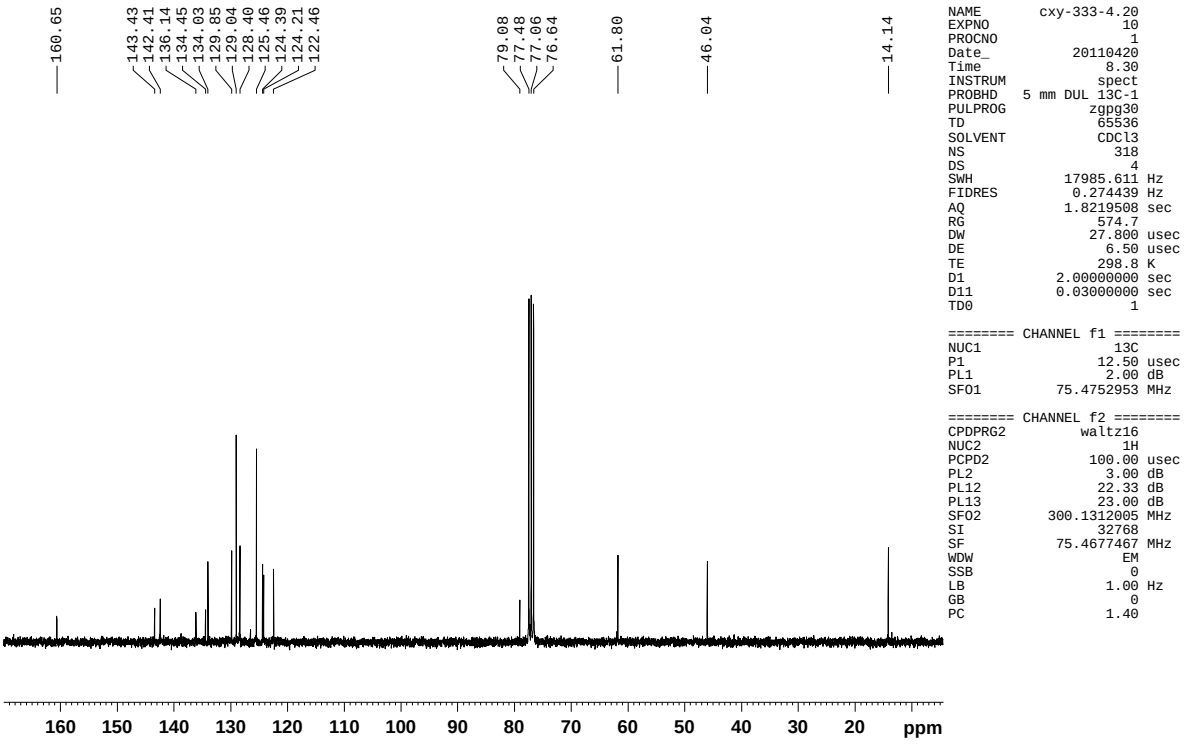
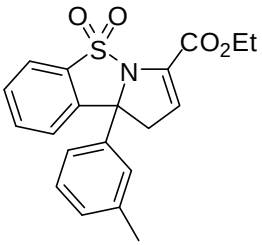
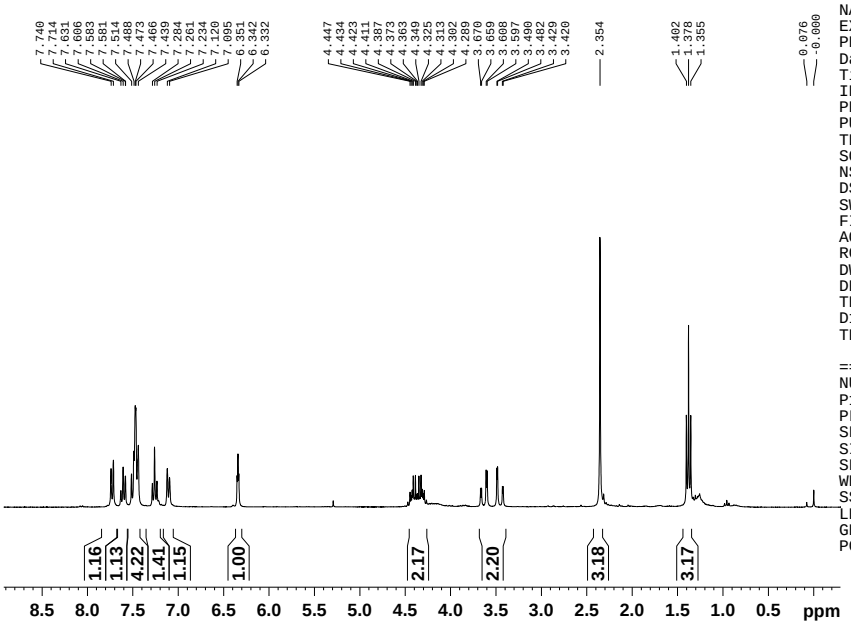
4a


7.753
7.726
7.702
7.697
7.691
7.672
7.636
7.632
7.611
7.607
7.585
7.581
7.524
7.521
7.495
7.468
7.441
7.410
7.406
7.383
7.377
7.362
7.357
7.326
7.322
7.318
7.298
7.259
6.346
6.337
6.336
6.327
4.452
4.428
4.416
4.392
4.353
4.330
4.318
4.306
4.294
3.666
3.655
3.605
3.594
3.515
3.507
3.454
3.446
1.410
1.386
1.362
-0.000
1.22
2.09
1.28
2.37
2.24
1.26
1.00
2.18
2.16
3.17
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 ppm
NAME cxy-333-4.20
EXPNO 11
PROCNO 1
Date_ 20110420
Time 8.52
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 362
DW 55.600 usec
DE 8.00 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


160.65
143.43
142.41
136.14
134.45
134.03
129.85
129.04
128.40
125.46
124.39
124.21
122.46
79.08
77.48
77.06
76.64
61.80
46.04
14.14
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
NAME cxy-333-4.20
EXPNO 10
PROCNO 1
Date_ 20110420
Time 8.30
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 318
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 574.7
DW 27.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677467 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

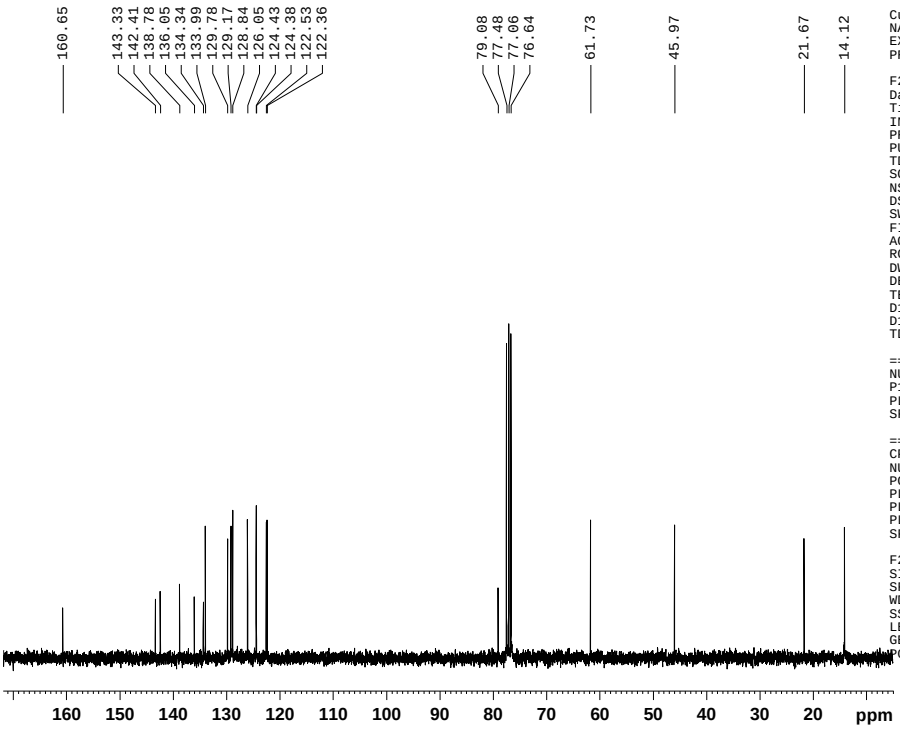


4f




```
NAME          cxy-372-3.8
EXPNO                  10
PROCNO                  1
Date_           20110308
Time                8.27
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   128
DW                55.600 usec
DE                 8.00 usec
TE                290.6 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.80 usec
PL1                3.00 dB
SFO1         300.1324010 MHz
SI                 32768
SF           300.1300049 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```




```
Current Data Parameters
NAME          cxy-372-c
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20110304
Time              20.42
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    71
DS                     4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                 13004
DW                27.800 usec
DE                 8.00 usec
TE                295.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
PL13              23.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF           75.4677500 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```