Supporting Information

An effective and general method for highly regioselective synthesis of 1-phenylpyrazoles from β -enaminoketoesters, tandem Blaise-acylation adducts

Young Ok Ko, ^a Yu Sung Chun, ^a Cho-Long Park, ^a Youngmee Kim, ^a Hyunik Shin, ^b*
Sungho Ahn, ^c Jongki Hong, ^c Sang-gi Lee^a*

^aDepartment of Chemistry and Nano Science (BK21), Ewha Womans University, Seoul 120-750, Korea. ^bChemical Development Division, LG Life Sciences, Ltd./R&D, Daejeon 305-380, Korea. ^cCollege of Pharmacy, Kyung Hee University, Seoul 130-701, Korea

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General. All reactions and manipulations were performed in a nitrogen atmosphere using standard Schelenk techniques. The reaction solvents were distilled prior to use (THF: distilled from sodium benzophenone ketyl). All purchased reagents were used without further purification. Anhydrous solvents were transferred by oven-dried syringe. Flasks were flames dried under a stream of nitrogen. The NMR spectra were recorded at 250 MHz (¹H)/62.5 MHz (¹³C). The chemical shifts were relative to TMS (as an internal reference) for ¹H NMR.

General Procedures for the Synthesis of β-Enaminoketoesters 1. To a stirred suspension of commercial zinc dust (Aldrich, $10 \mu m$, 0.65 g, 10 mmol) in THF (2.5 mL) was added 5 mol% of methansulfonic acid and the mixture was heated at reflux for 10 min. To the mixture was added benzonitrile (0.52 mL, 5 mmol) in one portion followed by addition of ethyl bromoacetate (0.83 mL, 7.5 mmol) over 1h by using syringe pump while maintaining the reflux temperature. After 1h stirring, the reaction mixture was cooled to $0 \, ^{\circ}$ C and added 1.0 equivalent of n-BuLi (2 m in cyclohexane, 2.5 m, 5 mmol) and acetic anhydride (0.62 m, 6.5 mmol) in sequence. The reaction temperature was allowed to room temperature and stirred for 3 m h. The reaction was quenched by addition of sat'd NH₄Cl (aq.) and extracted with ethyl acetate. The organic layer was washed with water, brine, and dried over anhydrous MgSO₄. After evaporation of solvent, the residue was purified by column chromatography on silica gel (eluent: n-henxane: EtOAc = 1:1) to afford the product n 1a (0.96 m, n 2 mmol, n3 mmol).

1a: Yield : 82 %, ¹H NMR (250 MHz, CDCl₃) δ 0.71 (t, J = 7.2 Hz, 3H), 2.38 (s, 3H), 3.76 (q, J = 7.2 Hz, 2H), 5.49 (bs, 1H), 7.36 ~ 7.45 (m, 5H), 11.00 (bs, 1H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 13.3, 29.4, 60.0, 104.1, 126.6, 128.6, 130.0, 138.4, 166.9, 169.8, 197.1 ppm; HRMS (ESI) Cal. for (M⁺+H):C₁₃H₁₆NO₃:234.1130. Found: 234.1121.

1b: Yield: 81 % (E/Z = 1:1), ¹H NMR (250 MHz, CDCl₃) δ [0.68 (t, J = 7.2 Hz) + 0.77 (t, J = 7.1 Hz)] (3H), [2.13 (s) + 2.33 (s)] (3H), [3.77 (q, J = 7.1 Hz) + 3.89 (q, J = 7.1 Hz)] (2H), [5.80 (bs) + 6.35 (bs)] (1H), 7.29 \sim 7.74 (m, 10H), [9.05 (bs) + 10.71 (bs)] (1H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 13.2, 13.6, 21.6, 22.5, 59.1, 60.0, 99.0, 102.0, 126.4, 127.9, 128.0, 128.3, 129.8, 131.5, 141.7, 143.4, 165.1, 168.1, 169.3, 170.0, 195.0, 196.3 ppm; HRMS (ESI) Cal. for (M⁺+H):C₁₃H₁₆NO₃:234.1130. Found: 234.1144.

1c: Yield: 94 %, ¹H NMR (250 MHz, CDCl₃)
$$\delta$$
 0.70 (t, J = 7.2 Hz, 3H), 0.95 (d, J = 6.6 Hz, 6H), 2.11 ~ 2.22 (m, 1H), 2.58 (d, J = 7.0 Hz, 2H), 3.73 (q, J = 7.2 Hz, 2H), 5.58 (bs, 1H), 7.35 ~ 7.47 (m, 5H),

11.39 (bs, 1H) ppm; 13 C NMR (62.5 MHz, CDCl₃) δ 13.3, 22.8, 25.5, 49.4, 60.1, 104.6, 126.6, 128.6, 130.0, 138.4, 166.4, 169.9, 199.1 ppm; HRMS (ESI) Cal. for (M⁺+H):C₁₆H₂₂NO₃:276.1600. Found: 276.1586.

1d: Yield : 90 % (E/Z = 1:1), ¹H NMR (250 MHz, CDCl₃)
$$\delta$$
 [0.68 (t, J = 7.1 Hz) + 0.75 (t, J = 7.1 Hz)] (3H), [0.91 (d, J = 6.6 Hz) + 0.99 (d, J = 6.6 Hz)] (6H), [1.80 \sim 1.96 (m) + 1.98 \sim

2.11 (m)] (1H), [2.36 (d, J = 7.5 Hz) + 2.56 (d, J = 7.3 Hz)] (2H), [3.75 (q, J = 7.1 Hz) + 3.89 (q, J = 7.1 Hz)] (2H), [5.64 (bs) + 6.14 (bs,)] (1H), 7.34 ~ 7.76 (m, 5H), [9.14 (bs) + 10.81 (bs)] (1H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 13.2, 13.6, 22.3, 22.4, 28.1, 28.5, 42.8, 43.6, 59.1, 60.0, 99.0, 102.2, 126.4, 127.9, 128.0, 128.3, 128.4, 129.8, 130.0, 131.5, 141.9, 143.4, 167.7, 169.5, 170.0, 170.6, 195.2, 196.3 ppm; HRMS (ESI) Cal. for (M⁺+H):C₁₆H₂₂NO₃:276.1600. Found: 276.1618.

NH₂ O
$$1e$$
:
NH₂ O CF_3 + CF_3 (q, ...

1e: Yield: 84 % (E/Z = 1.23/1), 1 H NMR (250 MHz, CDCl₃) δ [0.62 (t, J = 7.1 Hz) + 0.92 (t, J = 7.1 Hz)] (3H), [3.60 (q, J = 7.1Hz) + 4.03 (q, J = 7.1 Hz)]

(2H), [5.45 (bs) + 5.94 (bs)] (1H), 7.26-7.86 (m, 9H), [9.15 (bs) + 10.78 (bs)] (1H) ppm; ^{13}C NMR (62.5 MHz, CDCl₃) δ 13.1, 13.8, 59.7, 60.3, 99.7, 103.6, 124.89, 124.92, 124.95, 124.98, 126.7, 126.8, 127.3, 128.6, 128.7, 128.9, 130.2, 130.3, 136.8, 137.1, 144.0, 145.8, 166.0, 168.2, 168.5, 169.0, 193.1, 194.2 ppm; HRMS (ESI) Cal. for $(\text{M}^+\text{+H}):C_{19}\text{H}_{17}\text{F}_3\text{NO}_3:364.1161$. Found: 364.1157.

1f: Yield : 89 % (E/Z = 2/1), ¹H NMR (250 MHz, CDCl₃) δ [0.61 (t, J = 7.1 Hz) + 0.92 (t, J = 7.1 Hz)] (3H), [3.60 (q, J = 7.1 Hz) + 4.03 (q, J = 7.1 Hz)]

(2H), [5.38 (bs) + 5.81 (bs)] (1H), 7.19-7.73 (m, 9H), [9.02 (bs) + 10.6 (bs)] (1H) ppm; 13 C NMR (62.5 MHz, CDCl₃) δ 13.1, 13.8, 59.7, 60.3, 100,9, 103.7, 125.5, 126.0, 126.5, 127.5, 128.0, 128.1, 128.7, 130.4, 131.4, 132.0, 132.1, 140.4, 140.6, 140.7, 142.2, 163.1, 165.5, 168.6, 168.9, 194.9, 195.0 ppm HRMS (ESI) Cal. for (M⁺+H):C₁₉H₁₇F₃NO₃:364.1161. Found: 364.1164.

1g: Yield : 82 %, ¹H NMR (250 MHz, CDCl₃) δ 1.09 (t,
$$J = 7.4$$
 Hz, 3H), 1.33 (t, $J = 7.1$ Hz, 3H), 2.20 (s, 3H), 2.60 (q, $J = 7.4$ Hz, 2H), 4.23 (q, $J = 7.1$ Hz, 2H), 6.34 (bs, 1H), 11.1 (bs, 1H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 9.6, 14.1, 23.0, 34.3, 60.2, 102.9, 166.6, 169.9, 200.0 ppm;

HRMS (ESI) Cal. for (M⁺+H):C₉H₁₆NO₃:186.1130. Found: 186.1139.

1h: Yield : 86 % (E/Z = 1:1), ¹H NMR (250 MHz, CDCl₃) δ [1.21 (t,
$$J$$
 = 7.5 Hz) + 1.22 (t, J = 7.5 Hz)] (3H), [1.32 (t, J = 7.1 Hz) + 1.33 (t, J = 7.1 Hz)] (3H), [2.27 (s) + 2.28 (s)] (3H), 2.53 (q, J = 7.5 Hz, 2H), [4.23 (q, J = 7.1 Hz) + 4.24 (q, J = 7.1 Hz)] (2H), [5.65 (bs) + 5.67 (bs)] (1H), 11.2 (bs, 1H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 12.4, 14.2, 28.9, 29.9, 60.2, 104.3, 169.7, 171.3, 196.9 ppm; HRMS (ESI) Cal. for (M⁺+H):C₉H₁₆NO₃:186.1130. Found: 186.1129.

1i: Yield: 83 %, ¹H NMR (250 MHz, CDCl₃) δ 1.33 (t, J = 7.1 Hz, 3H), 2.24 (s, 3H), 2.30 (s, 3H), 4.23 (q, J = 7.1 Hz, 2H), 6.20 (bs, 1H), 11.18 (bs, 1H) ppm, ¹³C NMR (62.5 MHz, CDCl₃) δ 14.3, 23.4, 30.1, 60.1, 103.2, 167.3, 169.7, 197.0 ppm; HRMS (ESI) Cal. for (M⁺+H):C₈H₁₄NO₃: 172.0974. Found: 172.0979.

NH₂ O **1j**: Yield : 68 %, ¹H NMR (250 MHz, CDCl₃) δ 1.33 (t, J = 7.1 Hz, 3H), 2.23 (s, 3H), 4.23 (q, J = 7.1 Hz, 2H), 5.99 (bs, 1H), 11.20 (bs, 1H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 14.2, 23.5, 60.1, 103.3, 167.2, 169.6, 197.2 ppm; HRMS (ESI) Cal. for (M⁺+H):C₈H₁₁D₃NO₃: 175.1159. Found: 175.1172.

1k: Yield: 70 %, ¹H NMR (250 MHz, CDCl₃) δ 1.33 (t, J = 7.1 Hz, 3H), 2.29 (s, 3H), 4.23 (q, J = 7.1 Hz, 2H), 6.82 (bs, 1H), 11.15 (bs, 1H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 14.1, 30.0, 60.0, 102.9, 167.7, 169.7, 196.8 ppm; HRMS (ESI) Cal. for (M⁺+H):C₈H₁₁D₃NO₃: 175.1159. Found: 175.1156.

General Procedures for the Synthesis of Pyrazoles 2. To a solution of 1a (165 mg, 0.5 mmol) in absolute ethanol (1 mL) was added phenyl hydrazine (0.3 mL, 2.5 mmol) and *p*-toluenesulfonic acid (5 mg, 5 mol%) and stirred at room temperature for 1 h. The reaction mixture was refluxed for 1 h, and cooled to room temperature. After evaporation of solvent, the residue was dissolved in ethyl acetate, and organic layer was washed subsequently with sat'd NaHCO₃(aq), 1N HCl(aq), and brine, and then dried with anhydrous MgSO₄. After comcentration under reduced pressure, the residue was purified by column chromatography on silica gel to afford the product 2a (139 mg, 0.45 mmol, 91 %).

2a: Yield : 91 %, ¹H NMR (250 MHz, CDCl₃)
$$\delta$$
 1.22 (t, J = 7.1 Hz, 3H), 2.59 (s, 3H), 4.24 (q, J = 7.1 Hz, 2H), 7.38 \sim 7.51 (m, 8H), 7.65 \sim 7.69 (m, 2H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 12.8, 14.1, 60.0, 110.6,

125.8, 127.6, 128.2, 128.7, 129.2, 129.5, 133.1, 138.8, 144.8, 153.6, 164.2 ppm; HRMS (ESI) Cal. for (M^++H) : $C_{19}H_{19}N_2O_2$: 307.1447. Found: 307.1457.

2b: Yield: 88 %, ¹H NMR (250 MHz, CDCl₃) δ 1.13 (t, J = 7.1 Hz, 3H), 2.60 (s, 3H), 4.15 (q, J = 7.1 Hz, 2H), $7.15 \sim 7.34$ (m, 10H) ppm; 13 C NMR (62.5 MHz, CDCl₃) δ 14.0, 14.3, 59.8, 111.9, 125.3, 127.6, 127.9, 128.7, 128.8, 129.9, 130.4, 139.2, 146.3, 151.8, 163.8 ppm; HRMS (ESI) Cal. for (M^++H) : $C_{19}H_{19}N_2O_2$: 307.1447. Found: 307.1457.

2c: Yield : 87 %, ¹H NMR (250 MHz, CDCl₃) δ 0.78 (d, J = 6.7 Hz, 6H), 1.21 (t, J = 7.0 Hz, 3H), 1.89 ~ 1.83 (m, 1H), 2.92 (d, J = 7.3 Hz, 2H), 4.23 (q, J = 7.1 Hz, 2H), $7.36 \sim 7.49$ (m, 8H), $7.65 \sim 7.69$ (m, 2H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 13.9, 22.2, 28.9, 33.8, 59.9, 110.2, 126.7, 127.6, 128.1, 128.8, 129.2, 129.4, 133.1, 139.1, 148.5,

153.5, 164.2 ppm; HRMS (ESI) Cal. for $(M^++H):C_{22}H_{25}N_2O_2:349.1916$. Found: 349.1909.

 $(M^++H):C_{22}H_{25}N_2O_2:349.1916$. Found: 349.1923.

2d: Yield: 85 %, ¹H NMR (250 MHz, CDCl₃) δ 1.03 (d, J = 6.6 Hz, 6H), 1.12 (t, J = 7.1 Hz, 3H), 2.10 ~ 2.20 (m, 1H), 2.89 (d, J = 7.1Hz, 2H), 4.14 (q, J = 7.1 Hz, 2H), $7.17 \sim 7.33$ (m, 10H) ppm; 13 C NMR (62.5 MHz, CDCl₃) δ 13.9, 22.6, 28.5, 36.9, 59.8, 111.7, 125.3, 127.5, 127.8, 128.7, 130.1, 130.4, 139.2, 146.3, 154.8, 163.9 ppm; HRMS (ESI) Cal. for

2e: Yield : 83 %; ¹H NMR (250 MHz, CDCl₃) δ 0.97 (t, J = 7.1Hz, 3H), 4.08 (q, J = 7.1 Hz, 2H), $7.22 \sim 7.48$ (m, 10H), 7.60 (d, $J = 8.1 \text{ Hz}, 2\text{H}), 7.75 \sim 7.79 \text{ (m, 2H) ppm;}^{13}\text{C NMR (62.5 MHz,}$ CDCl₃) δ 13.6, 60.4, 112.5, 124.9, 125.1, 125.5, 127.9, 128.3,

128.6, 129.1, 129.3, 131.0, 132.4, 133.3, 138.7, 144.8, 153.6, 163.4 ppm; HRMS (ESI) Cal. for (M^++H) : $C_{25}H_{20}F_3N_2O_2$: 437.1477. Found: 437.1455.

2f: Yield : 88 %, ¹H NMR (250 MHz, CDCl₃) δ 0.96 (t, J = 7.1Hz, 3H), 4.08 (q, J = 7.1 Hz, 2H), $7.25 \sim 7.39$ (m, 10H), 7.70 (d. $J = 8.2 \text{ Hz}, 2\text{H}, 7.93 \text{ (d, } J = 8.0 \text{ Hz}, 2\text{H) ppm;}^{13}\text{C NMR (62.5)}$ MHz, CDCl₃) δ 13.6, 60.4, 112.2, 124.7, 124.8, 124.9, 125.4,

128.1, 129.0, 129.2, 129.4, 129.6, 130.4, 136.4, 139.0, 146.7, 152.0, 162.9 ppm; HRMS (ESI) Cal. for (M^++H) : $C_{25}H_{20}F_3N_2O_2$: 437.1477. Found: 437.1498.

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2g: Yield : 91 %, ¹H NMR (250 MHz, CDCl₃) δ 1.17 (t, J = 7.4 Hz, 3H), 1.39 (t, J = 7.1 Hz, 3H), 2.50 (s, 3H), 2.90 (q, J = 7.4 Hz, 2H), 4.34 (q, J = 7.1 Hz, 2H), 7.36 \sim 7.50 (m, 5H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 13.7, 14.3, 19.1, 59.7, 109.7, 126.1, 128.7, 129.2, 138.9, 150.4, 151.5, 164.3 ppm; HRMS (ESI) Cal. for (M⁺+H):C₁₅H₁₉N₂O₂: 259.1447. Found: 259.1435.

2h: Yield : 90 %, ¹H NMR (250 MHz, CDCl₃) δ 1.29 (t, J = 7.6 Hz, 3H), 1.39 (t, J = 7.1 Hz, 3H), 2.52 (s, 3H), 2.94 (q, J = 7.1 Hz, 2H), 4.34 (q, J = 7.1 Hz, 2H), 7.27 ~ 7.50 (m, 5H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 12.6, 13.5, 14.3, 21.7, 59.6, 110.0, 125.6, 128.3, 129.1, 138.8, 144.4, 156.6, 164.3 ppm; HRMS (ESI) Cal. for (M⁺+H):C₁₅H₁₉N₂O₂:259.1447. Found: 259.1438.

2i: Yield : 97 %, ¹H NMR (250 MHz, CDCl₃) δ 1.38 (t, J = 7.1 Hz, 3H), 2.50 (s, 3H), 2.52 (s, 3H), 4.33 (q, J = 7.1 Hz, 2H), 7.37 \sim 7.49 (m, 5H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 12.6, 14.3, 14.4, 59.7, 110.8, 125.6, 128.4, 129.2, 138.8, 144.5, 151.3, 164.6 ppm; HRMS (ESI) Cal. for (M⁺+H):C₁₄H₁₇N₂O₂: 245.1290. Found: 245.1311.

2j: Yield: 79 %, ¹H NMR (250 MHz, CDCl₃) δ 1.38 (t, J = 7.1 Hz, 3H), 2.50 (s, 3H), 4.33 (q, J = 7.1 Hz, 2H), 7.27 \sim 7.49 (m, 5H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 14.36, 14.43, 59.7, 110.9, 125.6, 128.4, 129.2, 138.8, 144.4, 151.5, 164.6 ppm; HRMS (ESI) Cal. for (M⁺+H):C₁₄H₁₄D₃N₂O₂: 248.1475. Found: 248.1493.

2k: Yield: 78 %, ¹H NMR (250 MHz, CDCl₃) δ 1.38 (t, J = 7.1 Hz, 3H), 2.52 (s, 3H), 4.33 (q, J = 7.1 Hz, 2H), 7.38 \sim 7.51 (m, 5H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 12.6, 14.4, 59.7, 110.8, 125.6, 128.4, 129.2, 138.7, 144.4, 151.3, 164.5 ppm; HRMS (ESI) Cal. for (M⁺+H):C₁₄H₁₄D₃N₂O₂: 248.1475. Found: 248.1485.

3: Yield : 64 %, ¹H NMR (250 MHz, CDCl₃) δ 0.70 (t, J = 7.2 Hz, 3H), 2.35 (s, 3H), 3.67 (q, J = 7.2 Hz, 2H), 4.23 (d, J = 6.2 Hz), 7.10 \sim 7.13 (m, 2H), 7.20 \sim 7.40 (m, 8H), 12.57 (bs, 1H) ppm; ¹³C NMR (62.5 MHz, CDCl₃) δ 13.38, 29.30, 48.44, 59.93, 104.75, 126.91, 127.30, 127.58, 128.40, 128.75, 129.31, 134.10, 137.37, 167.97, 169.71, 196.36 ppm.

Table S1-1. Crystal data for 2a (CCDS 708928)

Table S1-1. Crystal data for 2a (CCDS 700928)			
Empirical formula	C19 H18 N2 O2		
Formula weight	306.35		
Temperature	170(2) K		
Wavelength	0.71073 Å		
Crystal system	Monoclinic		
Space group	P2(1)/c		
Unit cell dimensions	a = 8.2775(12) Å	α= 90.00°	
	b = 15.616(2) Å	β= 98.896(3)°	
	c = 12.3892(18) Å	$\gamma = 90.00^{\circ}$	
Volume	$1582.2(4) \text{ Å}^3$		
Z	4		
Density (calculated)	1.286 Mg/m^3		
Absorption coefficient	0.084 mm ⁻¹		
F(000)	648		
Crystal size	$0.13 \times 0.08 \times 0.08 \text{ mm}^3$		
Theta range for data collection	2.11 to 26.00 .		
Index ranges	$-9 \le h \le 10, -19 \le k \le 19, -12 \le l \le 15$		
Reflections collected	8739		
Independent reflections	3094 [R(int) = 0.0571]		
Completeness to theta $= 26.00$	99.5 %		
Absorption correction	None		
Refinement method	Full-matrix least-squares on F	72	
Data / restraints / parameters	3094 / 0 / 211		
Goodness-of-fit on F ²	0.885		
Final R indices [I>2sigma(I)]	R1 = 0.0421, $wR2 = 0.0862$		
R indices (all data)	R1 = 0.0793, $wR2 = 0.0943$		
Extinction coefficient	0.0065(10)		
Largest diff. peak and hole	0.176 and -0.157 e.Å-3		

Table S2. Crystal data for 2b (CCDS 708929).

Empirical formula C19 H18 N2 O2

Formula weight 306.35

Temperature 293(2) K

Wavelength 0.71073 Å

Crystal system Monoclinic

Space group P2(1)/n

Unit cell dimensions a = 10.468(8) Å $\alpha = 90.00^{\circ}$

b = 15.676(12) Å β = 103.79° c = 10.468(8) Å γ = 90.00°

Volume $1668(2) \text{ Å}^3$

Z 4

Density (calculated) 1.220 Mg/m³
Absorption coefficient 0.080 mm⁻¹

F(000) 648

Crystal size $0.15 \times 0.10 \times 0.08 \text{ mm}^3$

Theta range for data collection 2.39 to 25.99.

Index ranges $-12 \le h \le 12, -17 \le k \le 19, -9 \le l \le 12$

Reflections collected 9153

Independent reflections 3264 [R(int) = 0.1063]

Completeness to theta = 25.99 99.9 %
Absorption correction None

Refinement method Full-matrix least-squares on F²

Data / restraints / parameters 3264 / 0 / 211

Goodness-of-fit on F² 0.681

Final R indices [I>2sigma(I)] R1 = 0.0587, wR2 = 0.1324 R indices (all data) R1 = 0.1489, wR2 = 0.1464

Extinction coefficient 0.017(2)

Largest diff. peak and hole 0.233 and -0.188 e.Å⁻³

Table S3. Crystal data for 2e(CCDS 708930).

Table 83. Crystal data for 2e(C	CDS 708930).		
Empirical formula	C25 H19 F3 N2 O2		
Formula weight	formula weight 436.42		
Temperature 170(2) K			
Wavelength	0.71073 Å		
Crystal system	Monoclinic		
Space group	P2(1)/c		
Unit cell dimensions	a = 12.182(2) Å	α= 90.00°	
	b = 19.321(4) Å	β= 98.535(4)°	
	c = 9.0766(17) Å	$\gamma = 90.00^{\circ}$	
Volume	$2112.7(7) \text{ Å}^3$		
Z	4		
Density (calculated)	1.372 Mg/m^3		
Absorption coefficient	0.106 mm ⁻¹		
F(000)	904		
Crystal size	$0.15 \times 0.08 \times 0.08 \text{ mm}^3$	0.15 x 0.08 x 0.08 mm ³	
Theta range for data collection	1.69 to 26.00 .	1.69 to 26.00 .	
Index ranges	$-15 \le h \le 15, -23 \le k \le 21, -11 \le l \le 10$		
Reflections collected	effections collected 11696		
Independent reflections	ections $4139 [R(int) = 0.0557]$		
Completeness to theta = 26.00	99.7 %		
Absorption correction	None		
Refinement method	Full-matrix least-square	es on F ²	
Data / restraints / parameters	parameters 4139 / 0 / 290		
Goodness-of-fit on F^2 0.676			
Final R indices [I>2sigma(I)]	R1 = 0.0341, $wR2 = 0.0$	R1 = 0.0341, $wR2 = 0.0551$	
R indices (all data) $R1 = 0.0749$, $wR2 = 0.0586$		0586	
Largest diff. peak and hole 0.159 and -0.167 e.Å ⁻³			

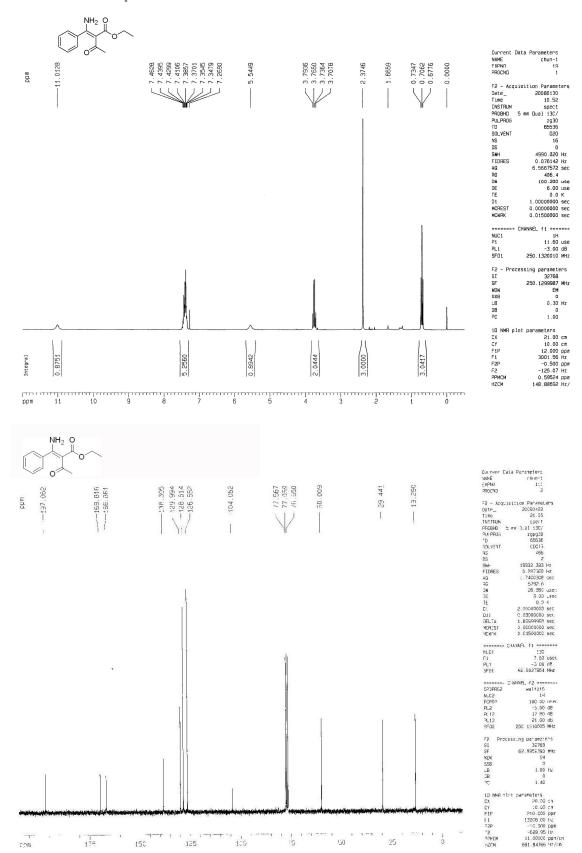
Table S4. Crystal data for 2f (CCDS 708931).

Largest diff. peak and hole

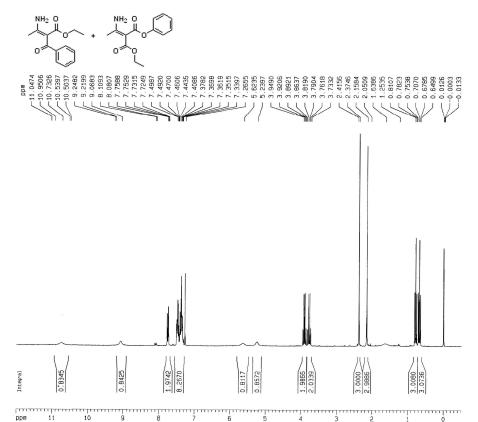
Table S4. Crystal data for 2f (C	CCDS 708931).		
Empirical formula	C25 H19 F3 N2 O2		
Formula weight	436.42	436.42	
Temperature	170(2) K		
Wavelength	0.71073 Å		
Crystal system	Triclinic		
Space group	P-1		
Unit cell dimensions	a = 9.801(2) Å	α = 78.875(5)°	
	b = 9.982(2) Å	β = 88.801(5)°	
	c = 12.377(3) Å	$\gamma = 64.005(4)^{\circ}$	
Volume	$1065.2(4) \text{ Å}^3$		
Z	2		
Density (calculated)	$1.361~\mathrm{Mg/m^3}$		
Absorption coefficient	0.105 mm ⁻¹		
F(000)	452		
Crystal size	$0.10 \times 0.08 \times 0.05 \text{ mm}^3$	$0.10 \times 0.08 \times 0.05 \text{ mm}^3$	
Theta range for data collection	1.68 to 26.00 .	1.68 to 26.00 .	
Index ranges	$\text{-}12 \leq h \leq 12, \text{-}12 \leq k \leq$	$-12 \le h \le 12, -12 \le k \le 11, -10 \le l \le 15$	
Reflections collected	5963		
Independent reflections	4052 [R(int) = 0.2207]	4052 [R(int) = 0.2207]	
Completeness to theta = 26.00	96.8 %	96.8 %	
Absorption correction	None		
Refinement method	Full-matrix least-square	es on F ²	
Data / restraints / parameters	4052 / 0 / 291	4052 / 0 / 291	
Goodness-of-fit on F ²	Goodness-of-fit on F^2 0.714		
Final R indices [I>2sigma(I)]	R1 = 0.0684, $wR2 = 0.1$	R1 = 0.0684, $wR2 = 0.1151$	
R indices (all data)	R1 = 0.2089, $wR2 = 0.1$	R1 = 0.2089, $wR2 = 0.1613$	
Extinction coefficient	0.0067(14)	0.0067(14)	

 $0.345 \text{ and } -0.398 \text{ e.Å}^{-3}$

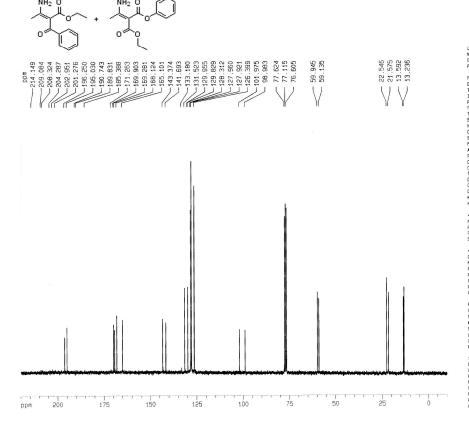
 ^{1}H and ^{13}C NMR spectra of 1a



^{1}H and ^{13}C NMR spectra of 1b





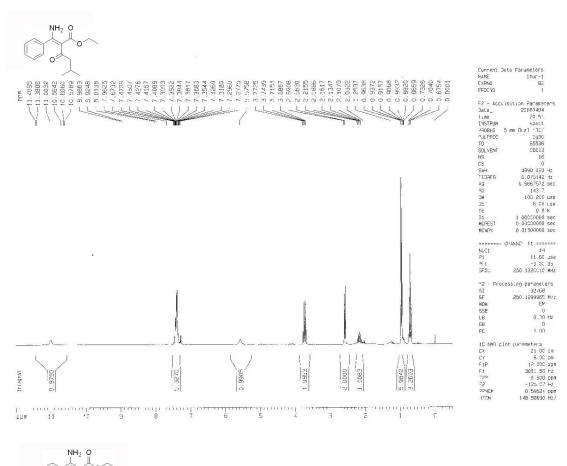




 ^{1}H and ^{13}C NMR spectra of 1c

169.895

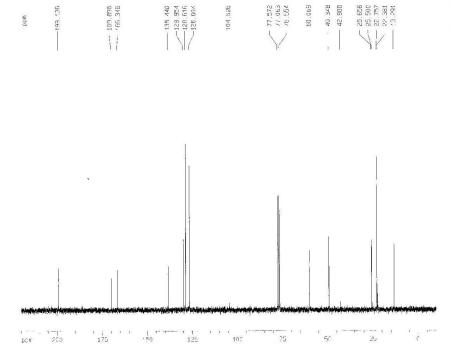
mdd



49.34B 42.90B

12 22 23 25 25

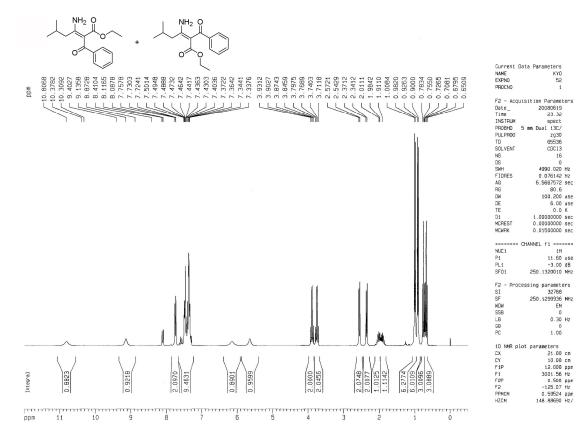
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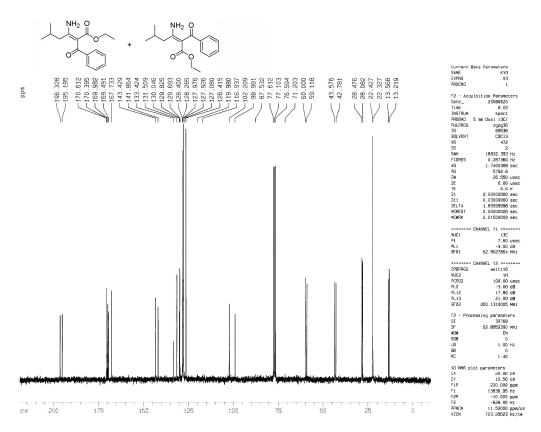


104.52E

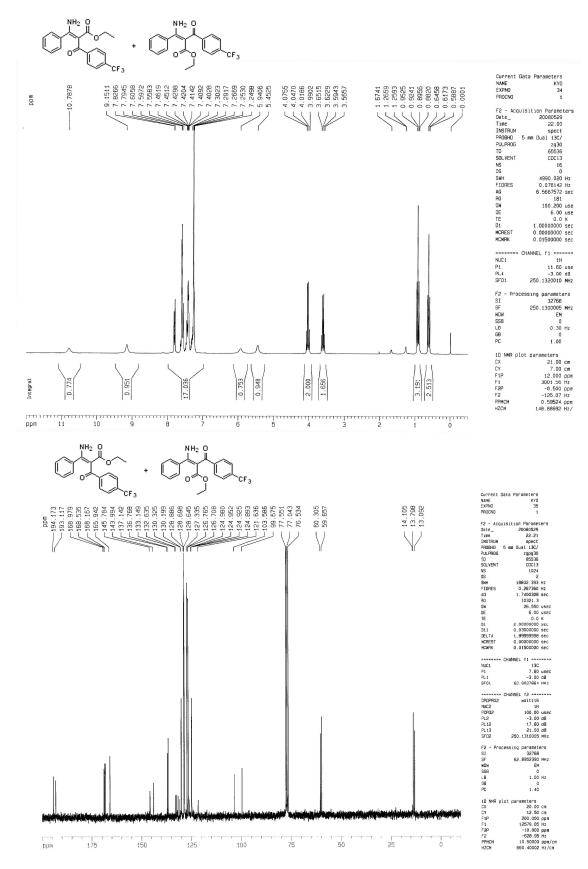


 ^{1}H and ^{13}C NMR spectra of $\mathbf{1d}$

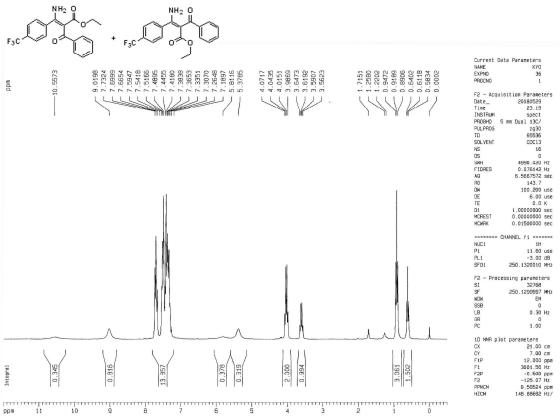


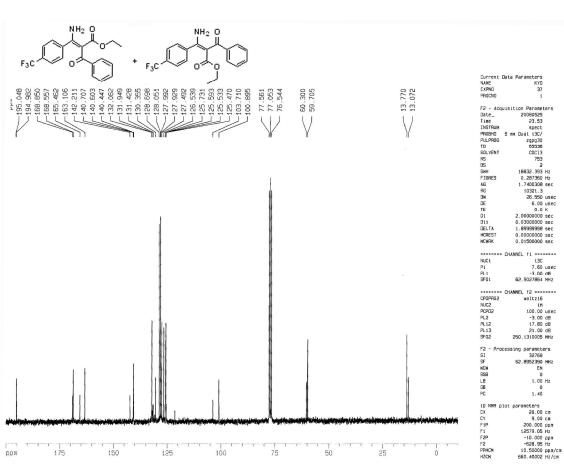


 ^{1}H and ^{13}C NMR spectra of 1e

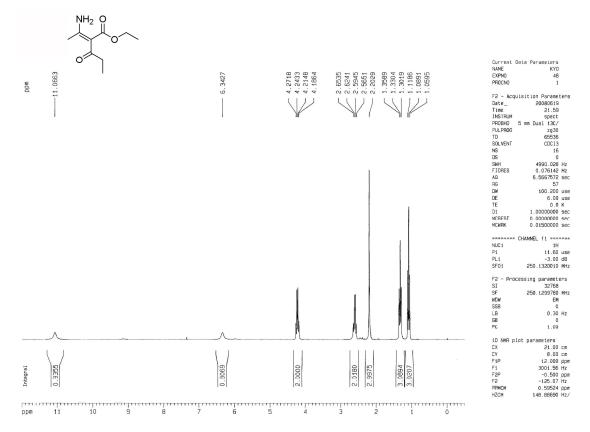


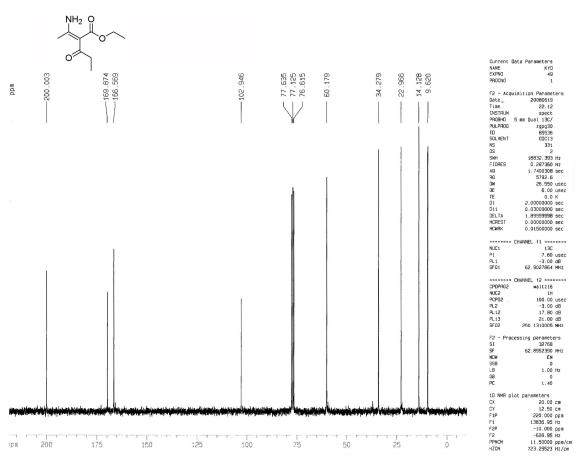
 ^{1}H and ^{13}C NMR spectra of 1f



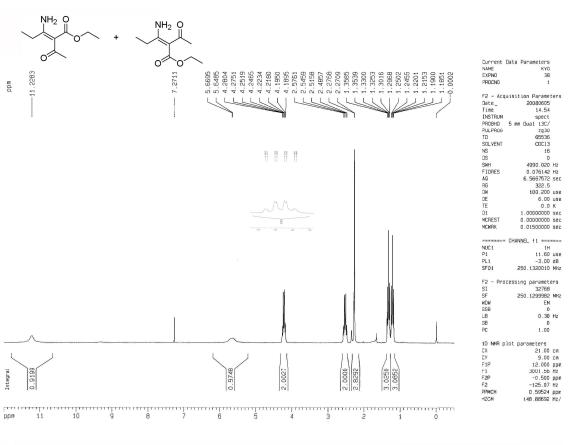


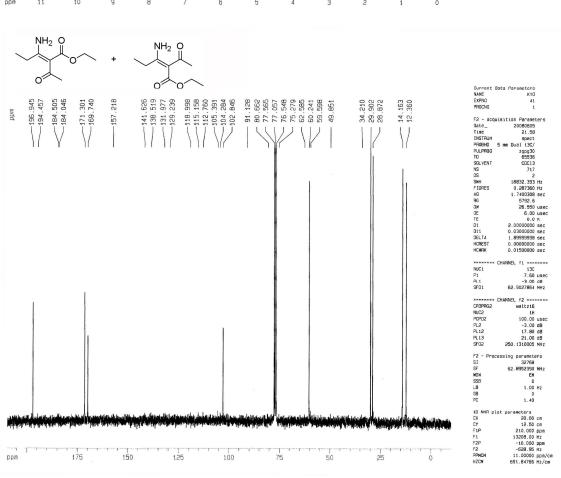
 ^{1}H and ^{13}C NMR spectra of 1g



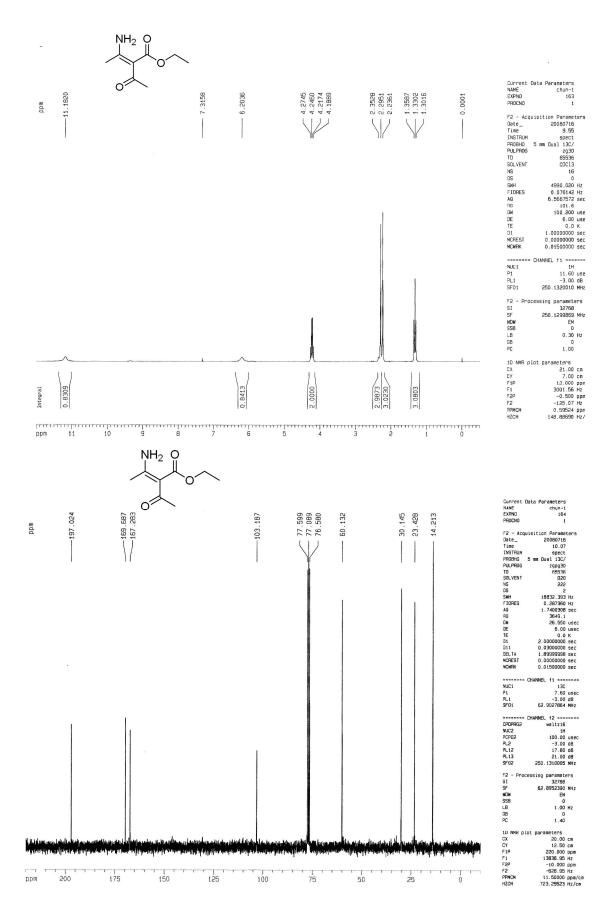


 ^{1}H and ^{13}C NMR spectra of 1h





 ^{1}H and ^{13}C NMR spectra of 1i



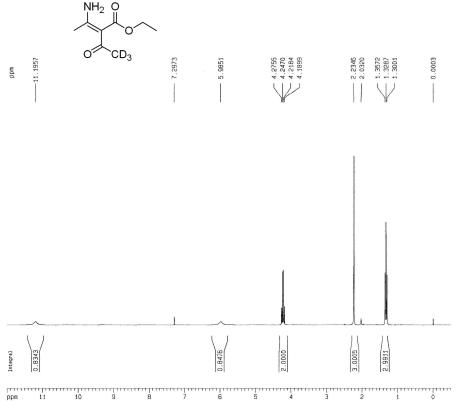
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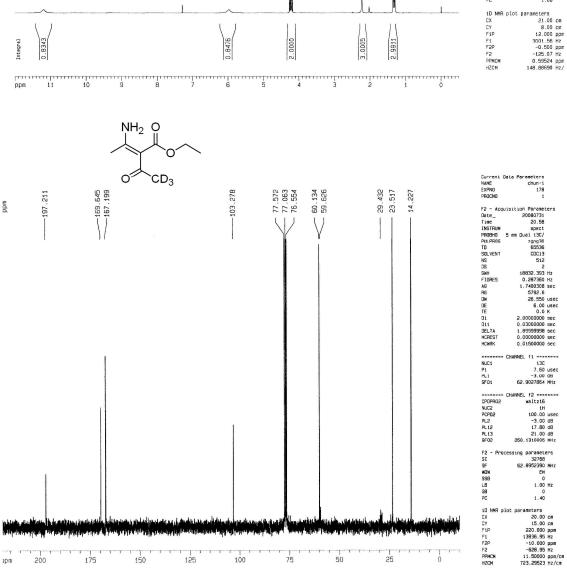
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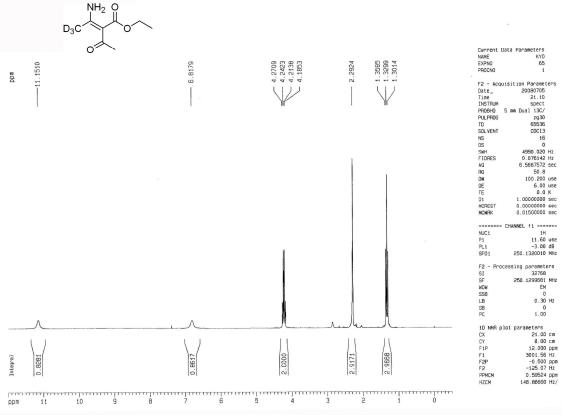
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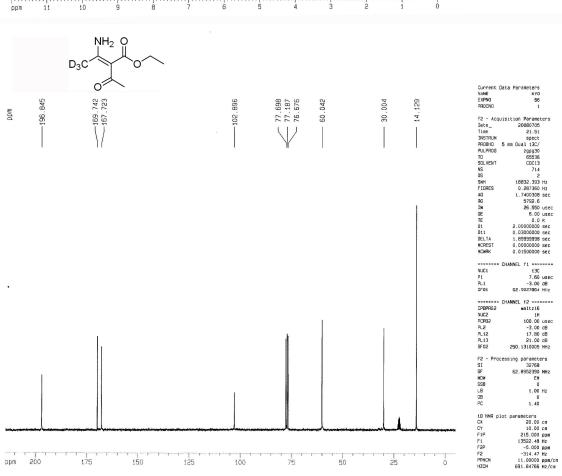
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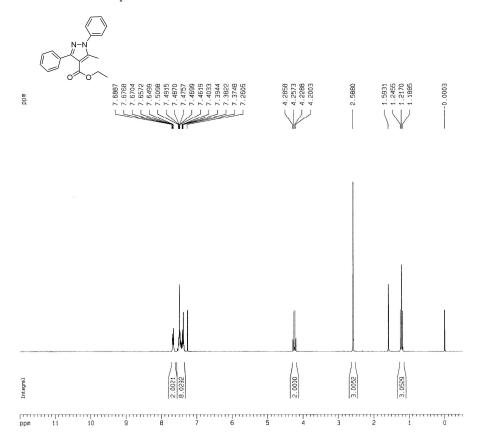


 ^{1}H and ^{13}C NMR spectra of 1k

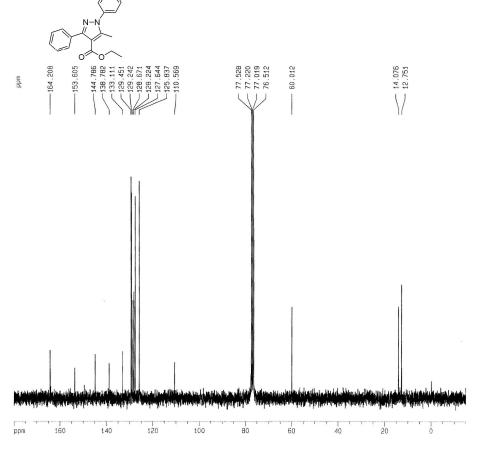




 ^{1}H and ^{13}C NMR spectra of $\mathbf{2a}$

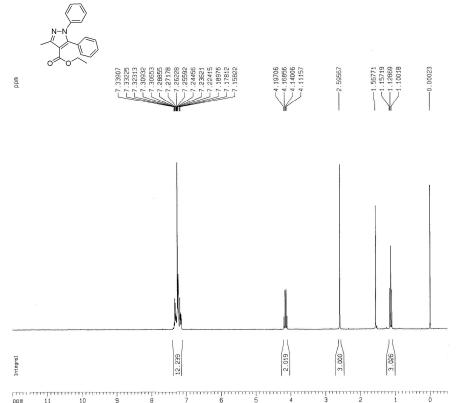




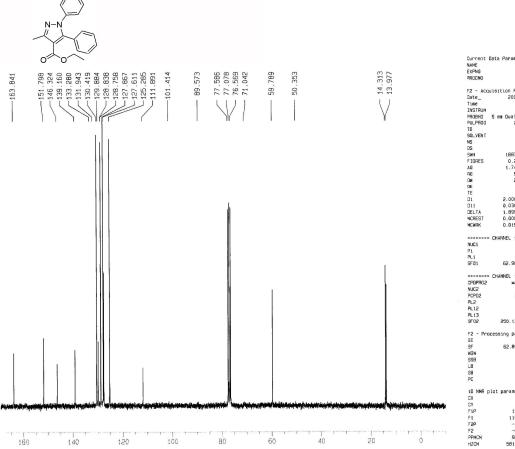


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EMPMI 201 PROCHOM			
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TID	PROBHO		
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PI			
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FROI 62.9027864 WH;			
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PL2 -3.00 dB PL12 17.80 dB PL13 21.000 dB PL13 27.0130005 NH2 F2 - Processing parameters SI 32768 FS 62.8952390 NH2 MDN EM DLB 0.00 H2 BB 0.00 H2 BC 2.000 Cm CV 10.00 Cm CV 10.00 Cm F1 11321.14 H2 F2P -494.3.43 H2 PMCM 9,75000 ppm/c F2P -494.3.43 H2	NUC2	1H	
PL12 17.80 dB PL13 21.00 dB PL13 27.00 dB PL	PCPD2	100.00	usec
PL13 25.00 dB sFPC2 250.1310005 Metz FFC2 - Processing parameters SI 32766 SF 62.9952390 Met	PL2	~3.00	dΒ
PL13 25.00 dB sFPC2 250.1310005 Metz FFC2 - Processing parameters SI 32766 SF 62.9952390 Met	PL12		
FIG. 250.1310005 Metz F2 - Processing parameters S2766 SF 62.9952390 Metz MOM EM SSS 0 BB 1.00 Hz BB 1.00 Hz BC 1.40 CO CC 20.00 Cm CV 20.00 Cm CV 10.00 Cm F1 180.000 ppm F1 11321.14 Hz F2P - 15.000 ppm F2P - 75000 ppm F2P - 75000 ppm F2P - 75000 ppm F3000	PL13		
SI 32766 SF 62.892390 MH2 MDM SSS 0 LB 1.00 H2 BB 0 CC 1.40 LB 0 CC 1.40 LD NMR plot parameters CX 20.00 cm CY 10.00 cm F1F 180.000 pm F1F 11321.14 H2 F2P -15.000 pm F2P -49.43.43 H2 FPMCM 9,75000 pm/F2	SF02		
SI 32766 SF 62.892390 MH2 MDM SSS 0 LB 1.00 H2 BB 0 CC 1.40 LB 0 CC 1.40 LD NMR plot parameters CX 20.00 cm CY 10.00 cm F1F 180.000 pm F1F 11321.14 H2 F2P -15.000 pm F2P -49.43.43 H2 FPMCM 9,75000 pm/F2	F2 - Pro	cessino paramet	ers
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MOM EM SS9 0 LB 1.00 H2 BB 0.00 H2 BC 1.40 L40 L10 NMR plot parameters TO XNR 20.00 cm F1 100.000 pm F1 11321.14 H2 F2P -15.000 pm F2P -15.000 pm F2P -49.3.43 H2 PMPOK 9.75000 pm/F2	SF		
SSS 0 1B 1.00 Hz BB 0.00 Hz BB 1.00 Hz BB 1.	NBH		
LB 1.00 H2 M8 0 PC 1.40 L40 L40 L40 L40 L40 L40 L40 L40 L40 L			
08 0 PC 1.40 III NNA plot parameters CX 20.00 cm CY 10.00 cm FIF 188.000 ppm FI 11321.14 Hz FZP -15.000 ppm FZ -943.43 Hz 9.75000 ppm/CP 9.75000 ppm/CP 9.75000 ppm/CP 9.75000 ppm/CP			H2
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10 NNA plot parameters CX 20.00 cm CY 10.00 cm FIP 180.000 ppm F1 11321.14 Hz F2P -15.000 ppm F2 -943.43 Hz PPMCN 9.75000 ppm/pm			
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CY 10.00 cm F1P 180.000 ppm F1 11321.14 Hz F2P -15.000 ppm F2 -943.43 Hz PPMCN 9.75000 ppm/c			
FIP 180.000 ppm F1 11321.14 Hz F2P -15.000 ppm F2 -943.43 Hz PPMCN 9.75000 ppm/c			
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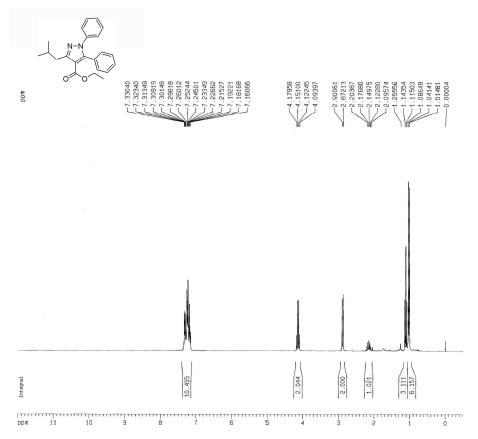
 ^{1}H and ^{13}C NMR spectra of 2b



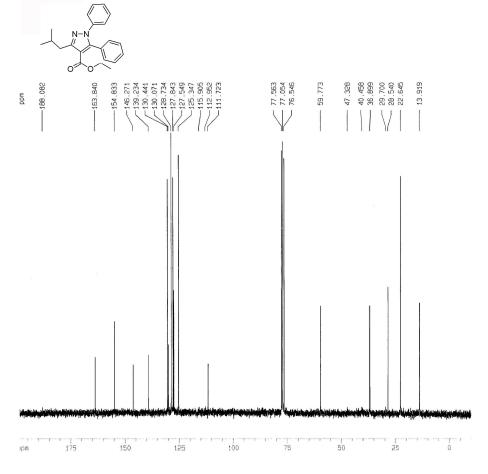
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 $^{1}\mbox{H}$ and $^{13}\mbox{C}$ NMR spectra of 2c

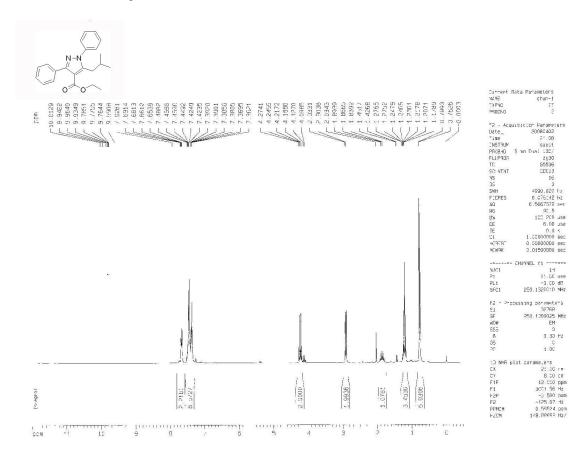


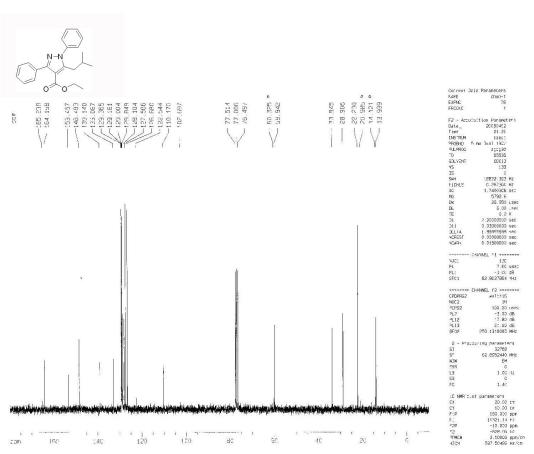
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SI 32768 SF 250.1300008 NDW EM SS9 0.08 GB 0.30 GB 0.0 PC 1.00 LD NMR plot parameters CX 21.00 CY 8.00 F1 12.000 F1 3001.56 F2P -0.500	Hz cm ppm Hz ppm Hz ppm



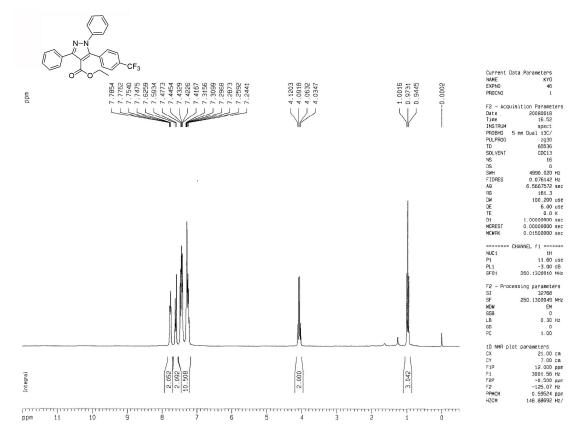
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MDM	EN EN	
SSB	0	
LB	1.00	H7
GB	0	
PC	1.40	
10 NNO -1		
CX NMH DI	ot parameters 20.00	C#
CY	12.50	
FIP	200.000	
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F2P	~10.000	
F2	-628.95	
PPMCN	10.50000	
HZCM	660.40002	

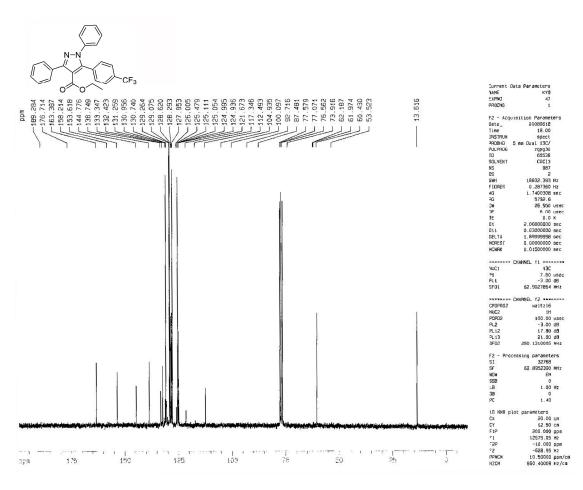
 ^{1}H and ^{13}C NMR spectra of $\mathbf{2d}$



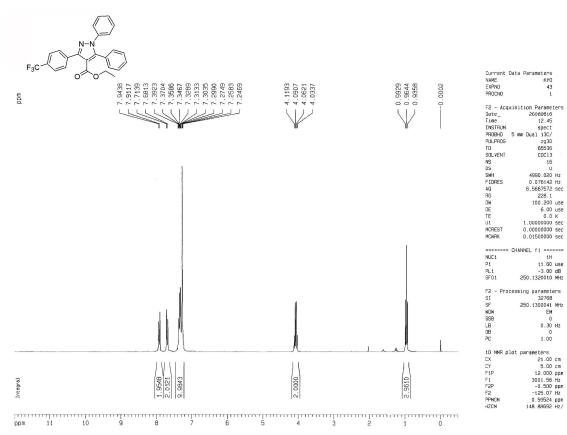


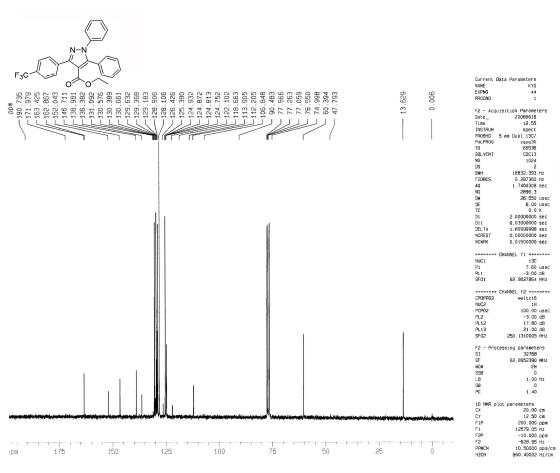
 ^{1}H and ^{13}C NMR spectra of 2e



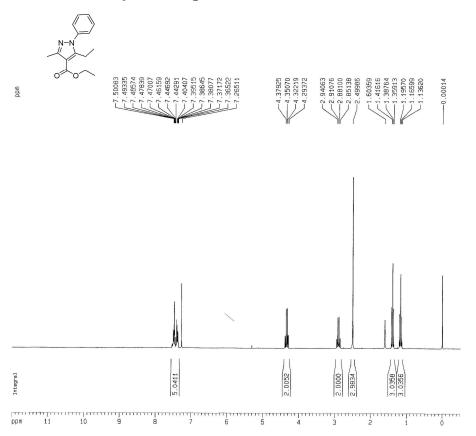


 ^{1}H and ^{13}C NMR spectra of 2f

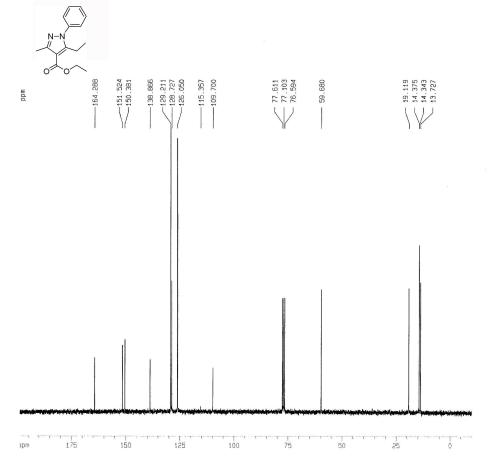






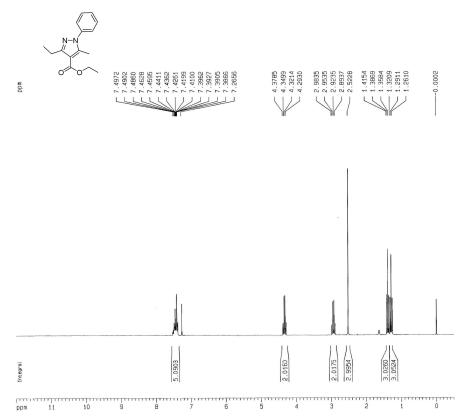


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EXPNO	75	
PROCNO	/5	
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F2 - Acqu	isition Parame	ters
Date_	20080909	
Time	3.16	
INSTRUM	spect	
PROBED	5 mm Dual 13C/	
PULPROG	zg30	
TD	65536	
SOLVENT	CDC13	
NS	16	
DS	0	
SWH	4990.020	Hz
FIDRES	0.076142	
AQ	6.5667572	sec
RG	812.7	
DW	100.200	
DE	6.00	
TE	0.0	K
D1	1.00000000	
MCREST	0.00000000	
MCWRK	0.01500000	
	CHANNEL f1 ====	
NUC 1	1H	
P1	11.60	
PL1	-3.00	
SEO1	250.1320010	
5. 01	EGO. ISEOUTO	11112
	essing paramete	ers
SI	32768	
SF	250.1299997	
MOM	EM	
SSB	0	
LB	0.30	HZ
GB	0	
PC	1.00	
1D NMR pl	ot parameters	
CX	21.00	Cm
CY	8.00	cm
FiP	12.000	ppm
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F2	-125.07	
РРМСМ	0.59524	ppm
HZCM	148.88692	

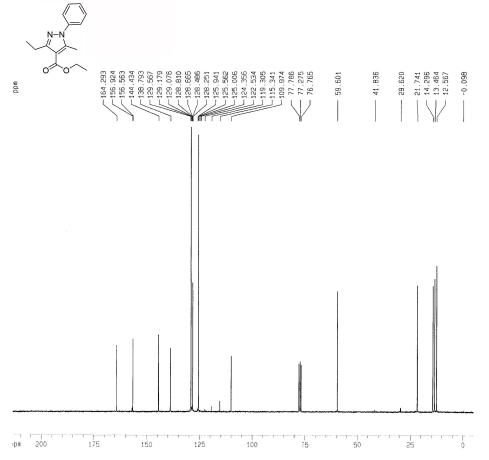


Current	Data Parameters	
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PROCNO	1	
F2 - AC	quisition Parame	tere
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Time	21.57	
INSTRUM		
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TD	65536	
SOLVENT	CDC13	
NS	407	
OS SWH	18832.393	
FIGRES	0.287360	
AR	1.7400308	
AG .	5792.6	
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011	0.03000000	sec
DELTA	1.89999998	
NCREST	0.00000000	
NCWRK	0.01500000	sec
	CHANNEL f1 ===:	
NUC1	130	
Pi		usec
Pl 1	-3.00	
SF01	62.9027864	MHZ
	CHANNEL f2 ===:	
CPOPRG2		
NUC2	18	
PCP02	100.00	usec
PL2	-3.00	
PL 12	17.80	
PL13	21.00	dB
SF02	250.1310005	NHZ
F2 - Pro	cessing paramete	ers
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MDM	02.0932390 EN	MITZ
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CX NNR C	lot parameters 20.00	
CY	12.50	
FIP	200.000	
F1	12579.05	
F2P	-10,000	
F2	-628.95	
PPNCM	10.50000	
HZCH	660.40002	Hz/cm

 ^{1}H and ^{13}C NMR spectra of 2h

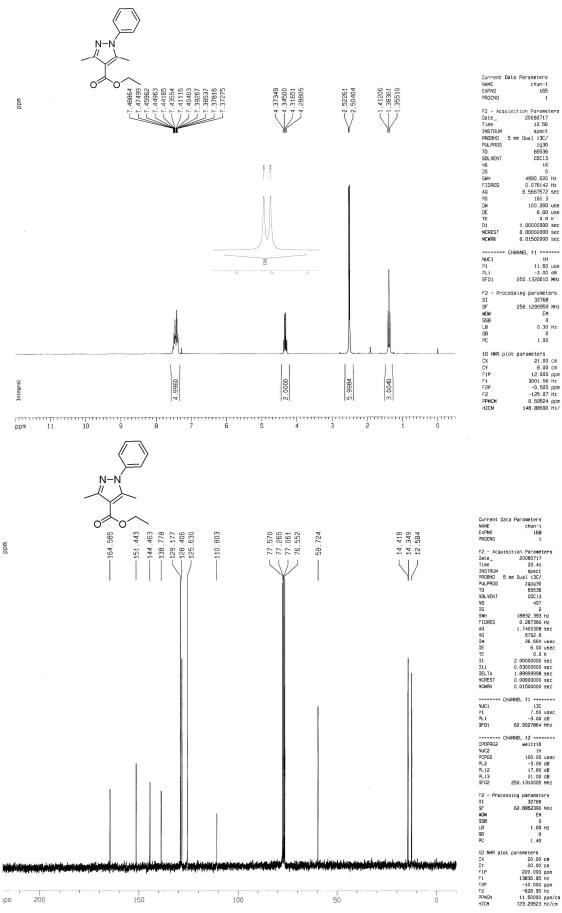




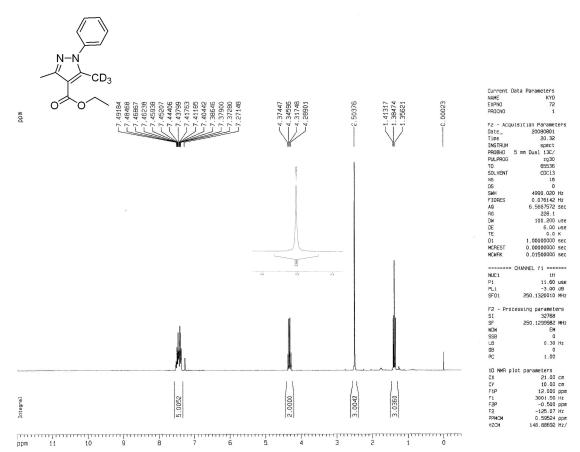


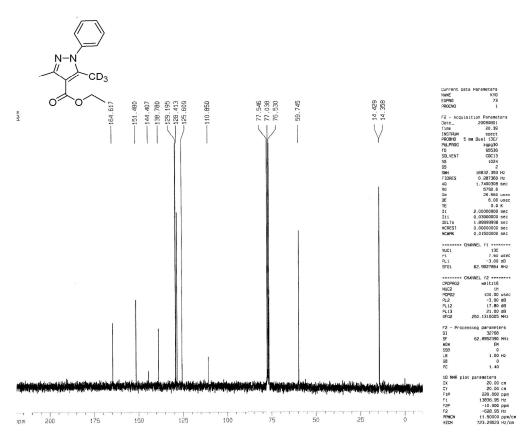




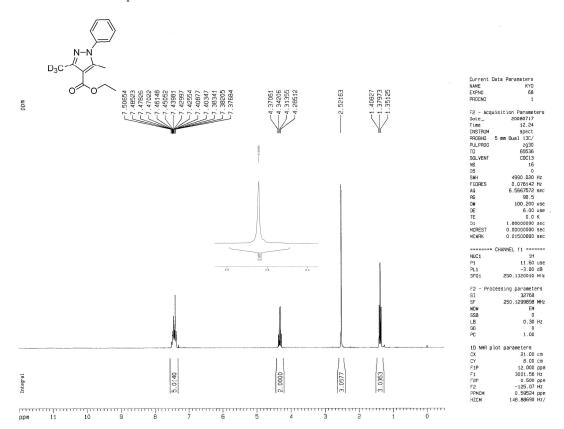


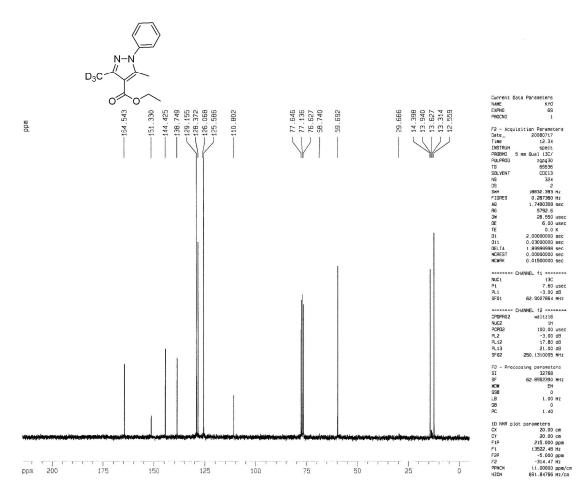
 ^{1}H and ^{13}C NMR spectra of 2j





 ^{1}H and ^{13}C NMR spectra of 2k





 $^{1}\mbox{H}$ and $^{13}\mbox{C}$ NMR spectra of 3

