Palladium-Catalyzed One-Pot Synthesis of 2-Alkyl-2-aryl Cyanoacetates

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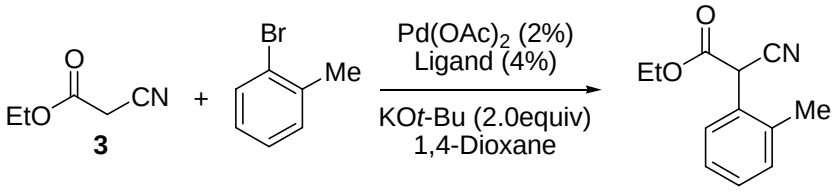
Supporting Information

Table of contents:

General information:

^{1}H-NMR and ^{13}C-NMR spectra were recorded on a 400 MHz spectrometer using $CDCl_3$ as the deuterated solvent. The chemical shifts (δ) are reported in parts per million (ppm) relative to trace amount of tetramethylsilane (0.00 ppm for 1H-NMR and 13C-NMR). The coupling constants (J) are reported in Hertz (Hz). HPLC is preformed on a commercial HPLC using a C18 column. High-resolution mass spectra were recorded on a commercial Fourier-Transform Mass Spectrometer (FTMS) using ES ionization modes (ESI). IR spectra were recorded on a Fourier-Transform Infrared Spectrometer. Yields refer to isolated material judged to be ≥95% pure by 1H NMR spectroscopy following silica gel chromatography. All the chemicals were used as received unless otherwise stated. The purifications were performed by flash chromatography using silica gel F-254 (230-500 mesh particle size).

Selected Screening Results for Arylation of 3 with 2-Bromotoluene:

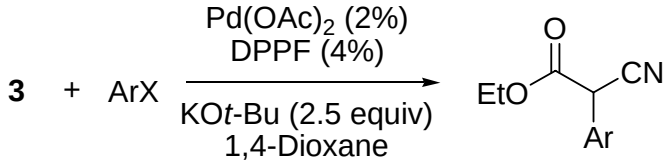


Ligand	RuPhos	Q-phos	P(*t*-Bu)$_3$	PCy$_3$	Josiphos	IPr/HCl	DPPF	BINAP
GC Conversion	<50%	<50%	84%	92%	44%	60%	100%	95%

Reaction conditions: 90^{o}C, 2h.

General Procedure for Arylation of 3:

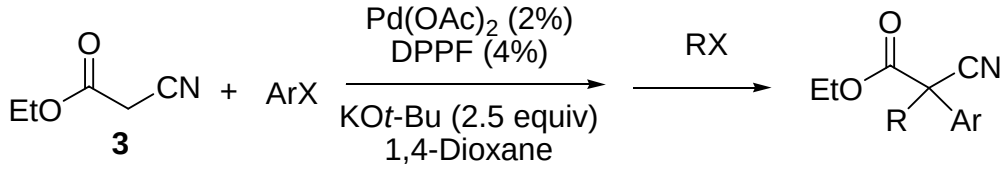
Scheme S1



3 (106 µL, 113mg, 1.0 mmol) and aryl halide (1.2 mmol) were added sequentially to a suspension of KO*t*-Bu (280 mg, 2.5mmol) in 1,4-dioxane (3 mL), resulting in a white suspension. A prepared solution of $Pd(OAc)_2$ (4.5 mg, 0.02 mmol) and DPPF (23 mg, 0.04 mmol) in 1,4-dioxane (1 mL) was then added. The resulting mixture was heated at 70^{o}C for 1-4h, when GC analysis of reaction mixture indicating the complete consumption of **3**. The reaction was cooled to room temperature, and AcOH (1N, 2mL) and EtOAc (8mL) were added sequentially. Organic layer was dried, concentrated and chromatographed to give isolated product (Scheme S1).

General Procedure for One-Pot Arylation/Alkylation of 3:

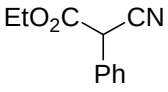
Scheme S2



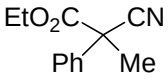
3 (106 µL, 113mg, 1.0 mmol) and aryl halide (1.2 mmol) were added sequentially to a suspension of KO*t*-Bu (280 mg, 2.5mmol) in 1,4-dioxane (3 mL), resulting in a white suspension. A prepared solution of $Pd(OAc)_2$ (4.5 mg, 0.02 mmol) and DPPF (23 mg, 0.04 mmol) in 1,4-dioxane (1 mL) was then added. The resulting mixture was heated at 70^{o}C for 1-4h, when analysis of reaction mixture indicating the complete consumption of **3**. The reaction was cooled to room temperature, and alkyl halide (1.2 mmol) was added. The resulting mixture was

stirred at 25°C-70°C for 1-12h, and GC or HPLC analysis indicated complete consumption of arylated cyanonitrile. The resulting reaction mixture was worked up via AcOH (1N)/DCM extraction or hexanes dilution/filtration. The crude product was purified via column chromatographed to give isolated product (Scheme S2).

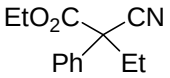
Characterization of Compounds:



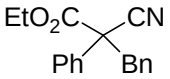
Ethyl 2-phenylcyanoacetate (Table 1, entry 1):[1] Ethyl 2-phenyl cyanoacetate was isolated as a colorless oil (171mg, 90%) from the reaction of **3** with bromobenzene employing the standard procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37-7.46 (m, 5H), 4.74 (s, 1H), 4.18-4.24 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.1, 130.1, 129.3, 129.2, 127.9, 115.8, 63.2, 43.7, 13.9. IR (film, cm^{-1}): 1734, 1234, 1197, 1026, 911, 730.



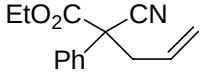
Ethyl 2-methyl-2-phenylcyanoacetate (Table 1, entry 2): [2] Ethyl 2-methyl-2-phenyl-cyanoacetate was isolated as a colorless oil (179 mg, 88%) from the reaction of **3** and bromobenzene followed by trapping with MeI at room temperature for 1h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.54 (m, 2 H), 7.34-7.42 (m, 3 H), 4.18-4.27 (m, 2 H), 1.94 (s, 3 H), 1.23 (t, *J* = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.9, 135.9, 129.2, 128.8, 125.7, 119.5, 63.2, 48.3, 24.9, 13.8. IR (film, cm^{-1}): 1743, 1448, 1238, 1101, 697.



Ethyl 2-ethyl-2-phenylcyanoacetate (Table 1, entry 3):[3] Ethyl 2-ethyl-2-phenylcyanoacetate was isolated as a colorless oil (173 mg, 80%) from the reaction of **3** and bromobenzene followed by trapping with EtI at 70°C for 4h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, *J* = 7.2 Hz, 2 H), 7.33-7.41 (m, 3 H), 4.16-4.29 (m, 2 H), 2.43 (qd, *J* = 7.6, 14.8 Hz, 1 H), 2.16 (qd, *J* = 7.6, 14.8 Hz, 1 H), 1.23 (t, *J* = 7.2 Hz, 3 H), 1.06 (t, *J* = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.6, 134.5, 129.1, 128.8, 126.1, 118.3, 63.1, 55.1, 31.6, 13.8, 9.8. IR (film, cm^{-1}): 1742, 1230, 910, 728.

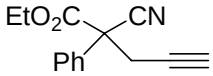


Ethyl 2-benzyl-2-phenylcyanoacetate (Table 1, entry 4): Ethyl 2-benzyl-2-phenylcyanoacetate was isolated as a colorless oil (220 mg, 79%) from the reaction of **3** and bromobenzene followed by trapping with benzyl bromide at room temperature for 1h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.55 (m, 2 H), 7.32-7.39 (m, 3 H), 7.21-7.23 (m, 3 H), 7.14-7.16 (m, 2 H), 4.11-4.22 (m, 2 H), 3.69 (d, *J* = 14.0 Hz, 1 H), 3.31 (d, *J* = 13.2 Hz, 1 H), 1.15 (t, *J* = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.3, 134.5, 134.2, 130.4, 129.1, 129.0, 128.3, 127.8, 126.4, 118.0, 63.3, 55.8, 44.2, 13.8. HRMS calcd (found) for $C_{18}H_{18}NO_2$ (MH^+): 280.1338 (280.1324). IR (film, cm^{-1}): 1741, 1497, 1221, 723, 697.

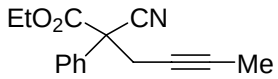


Ethyl 2-allyl-2-phenylcyanoacetate (Table 1, entry 5):[4] Ethyl 2-allyl-2-phenylcyanoacetate was isolated as a colorless oil (169 mg, 74%) from the reaction of **3** and bromobenzene followed

by trapping with allylbromide at room temperature for 2h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54-7.56 (m, 2 H), 7.34-7.42 (m, 3 H), 5.69-5.80 (m, 1 H), 5.26 (d, *J* = 17.2 Hz, 1 H), 5.21 (d, *J* = 10.2 Hz, 1 H), 4.16-4.29 (m, 2 H), 3.12 (dd, *J* = 7.2, 14.0 Hz, 1 H), 2.85 (dd, *J* = 6.8, 14.0 Hz, 1 H), 1.23 (t, *J* = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.4, 134.4, 130.1, 129.4, 129.2, 126.4, 121.4, 128.2, 63.5, 54.4, 42.5, 14.1. . HRMS calcd (found) for $C_{14}H_{16}NO_2$ (MH^+): 230.1181 (230.1181). IR (film, cm^{-1}): 1741, 1450, 1224, 695.

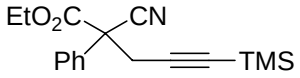


Ethyl 2-propargyl-2-phenylcyanoacetate (Table 1, entry 6): Ethyl 2-benzyl-2-propargyl-cyanoacetate was isolated as a colorless oil (136 mg, 60%) from the reaction of **3** and bromobenzene followed by trapping with propargyl bromide (1.2 equiv) and NaH (1.0 equiv) at room temperature for 4h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.45 (m, 2 H), 7.26-7.33 (m, 3 H), 4.09-4.23 (m, 2 H), 3.16 (dd, *J* = 2.4, 16.8 Hz, 1 H), 2.89 (dd, *J* = 2.4, 16.8 Hz, 1 H),2.04 (t, *J* = 2.6 Hz, 1 H), 1.14 (t, *J* = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7, 133.6, 129.6, 129.6, 126.3, 117.8, 77.1, 73.2, 64.0, 53.9, 29.2, 14.1. HRMS calcd (found) for $C_{14}H_{14}NO_2$ (MH^+): 228.1025 (228.1025). IR (film, cm^{-1}): 1745, 1233, 905, 726.

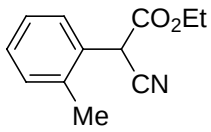


Ethyl 2-(2-butynyl)-2-phenylcyanoacetate (Table 1, entry 7): Ethyl 2-benzyl-2-propargyl-cyanoacetate was isolated as a colorless oil (159 mg, 66%) from the reaction of **3** and bromobenzene followed by trapping with 2-bromobutyne at 55°C for 10h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53-7.55 (m, 2 H), 7.36-7.44 (m, 3 H), 4.20-4.34 (m, 2 H), 3.23 (qd, *J* = 2.4, 16.4

Hz, 1 H), 2.92 (qd, *J* = 2.4, 16.4 Hz, 1 H), 1.76 (t, *J* = 2.4 Hz, 3 H), 1.23 (t, *J* = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$):[1] δ 166.7, 133.7, 129,1, 126.0, 118.0, 80.5, 72.3, 63,4, 54.2, 29.4, 13.8, 3.5. HRMS calcd (found) for $C_{15}H_{15}NO_2Na$ (MNa^+): 264.1000 (264.0986). IR (film, cm^{-1}): 1744, 1450, 1226, 912, 730.



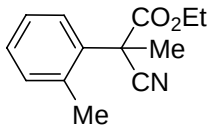
Ethyl 2-(3-trimethylsilyl-2-propynyl)-2-phenylcyanoacetate (Table 1, entry 8): Ethyl 2-(3-trimethylsilyl-2-propynyl)-2-phenylcyanoacetate was isolated as a colorless oil (269 mg, 90%) from the reaction of **3** and bromobenzene followed by trapping with 1-bromo-3-trimethylsilylpropyne at 55°C for 12h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.44 (m, 2 H), 7.26-7.31 (m, 3 H), 4.12-4.22 (m, 2 H), 3.14 (d, *J* = 16.4 Hz, 1 H), 2.89 (d, *J* = 16.8 Hz, 1 H), 1.16 (t, *J* = 7.2 Hz, 3 H), 0.00 (s, 9 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.8, 133,8, 129.5, 129.4, 126.4, 117.8, 99.7, 90.3, 63.8, 54.2, 30.7, 14.2, 0.02. HRMS calcd (found) for $C_{15}H_{21}N3O_2Si$ (MH^+): 300.1420 (300.1405). IR (film, cm^{-1}): 1743, 1227, 1025, 845.



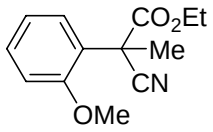
Ethyl 2-(2-tolyl)-cyanoacetate (Table 1, entry 9):[5] Ethyl 2-(2-tolyl)-cyanoacetate was isolated as a colorless oil (182 mg, 90%) from the reaction of **3** and 2-bromotoluene. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.46 (m, 1 H), 7.21-7.29 (m, 3 H), 4.89 (s, 1 H), 4.19-4.27 (m, 2 H), 2.39 (s, 3

[1] One carbon is missing in aromatic regions in our assignment presumably due to overlapping.

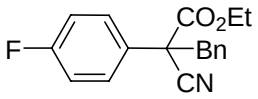
H), 1.26 (t, J = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.1, 136.3, 131.3, 129.4, 129.3, 128.7, 127.0, 116.0, 63.2, 41.1, 19.4, 13.9. IR (film, cm^{-1}): 1743, 1250, 1206, 910, 729.



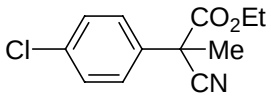
Ethyl 2-methyl-2-(2-tolyl)-cyanoacetate (Table 1, entry 10): Ethyl 2-methyl-2-(2-tolyl)-cyanoacetate was isolated as a colorless oil (182 mg, 84%) from the reaction of **3** and 2-bromotoluene followed by trapping with MeI at room temperature for 2h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38-7.40 (m, 1 H), 7.19-7.27 (m, 3 H), 4.23-4.28 (m, 2 H), 2.39 (s, 3 H), 2.03 (s, 3 H), 1.26 (t, J = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.2, 136.6, 133.5, 132.2, 129.0, 126.6, 126.0, 119.4, 63.2, 46.9, 23.9, 20.0, 12.9. HRMS calcd (found) for $C_{13}H_{16}NO_2(MH^+)$: 218.1181 (218.1178).



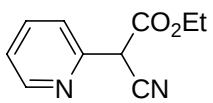
Ethyl 2-methyl-2-(2-methoxyphenyl)-cyanoacetate (Table 1, entry 11): Ethyl 2-methyl-2-(2-methoxyphenyl)-cyanoacetate was isolated as a colorless oil (184 mg, 79%) from the reaction of **3** and 2-bromoanisole followed by trapping with MeI at room temperature for 2h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (dd, J = 1.2, 7.6 Hz, 1 H), 7.33-7.37 (m, 1 H), 7.01 (d, J = 7.6 Hz, 1 H), 6.92 (d, J = 8.0 Hz, 1 H), 4.18-4.27 (m, 2 H), 3.81 (s, 3 H), 1.94 (s, 3 H), 1.24 (t, J = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.0, 156.4, 130.3, 126.7, 124.9, 121.0, 119.6, 111.4, 62.6, 55.5, 45.1, 22.7, 14.0. HRMS calcd (found) for $C_{13}H_{16}NO_3$ (MH^+): 234.1130 (234.1115). IR (film, cm^{-1}): 1744, 1255, 907, 727.



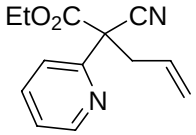
Ethyl 2-benzyl-2-(4-fluorophenyl)-cyanoacetate (Table 1, entry 12): Ethyl 2-benzyl-2-(4-fluorophenyl)-cyanoacetate was isolated as a colorless oil (228 mg, 77%) from the reaction of **3** and 1-bromo-4-fluorobenzene followed by trapping with benzylbromide at room temperature for 2h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50-7.54 (m, 2 H), 7.23-7.25 (m, 3 H), 7.13-1.15 (m, 2 H), 7.06 (t, J = 8.4 Hz, 2 H), 4.14-4.24 (m, 2 H), 3.67 (d, J = 14.0 Hz, 1 H), 3.30 (d, J = 13.6 Hz, 1 H), 1.18 (t, J = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.3, 162.9 (d, $J_{C\text{-}F}$ = 248 Hz), 133.9, 130.4, 130.2 (d, $J_{C\text{-}F}$ = 3.4 Hz), 128.4 (d, $J_{C\text{-}F}$ = 7.8 Hz), 128.1 (d, $J_{C\text{-}F}$ = 50.9 Hz), 117.9, 116.2, 115.9, 63.4, 55.2, 44.3, 13.8. HRMS calcd (found) for $C_{18}H_{17}FNO_2$ (MH^+): 298.1243 (298.1220). IR (film, cm^{-1}): 1742, 1510, 1238, 906, 728.



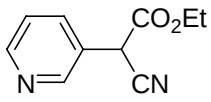
Ethyl 2-methyl-2-(4-chlorophenyl)-cyanoacetate (Table 1, entry 13):[2] Ethyl 2-methyl-2-(4-chlorophenyl)-cyanoacetate was isolated as a colorless oil (171 mg, 72%) from the reaction of **3** and 1-bromo-4-chlorobenzene followed by trapping with MeI at room temperature for 1h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 (d, J = 8.8 Hz, 2 H), 7.39 (d, J = 8.4 Hz, 2 H), 4.20-4.29 (m, 2 H), 1.94 (s, 3 H), 1.26 (t, J = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5, 134.0, 133.2, 128.2, 126.2, 118.1, 62.4, 46.8, 23.8, 12.7.



Ethyl 2-(2-pyridinyl)-cyanoacetate (Table 1, entry 14):[5] Ethyl 2-(2-pyridinyl)-cyanoacetate was isolated as a yellow solid (162 mg, 85%) from the reaction of **3** and 2-bromopyridine. ^{1}H NMR (400 MHz, $CDCl_3$): δ 14.01 (s, 1H), 7.65 (d, *J* = 6.0 Hz, 1H), 7.59 (t, *J* = 8.0 Hz, 1H), 7.30 (d, *J* = 9.2 Hz, 1 H), 6.67 (t, *J* = 6.4 Hz, 1H), 4.24 (q, *J* = 7.2 Hz, 2 H), 1.33 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.5, 155.6, 139.7, 134.0, 120.4, 119.3, 112.5, 62.6, 60.2, 14.6. IR (film, cm^{-1}): 1744, 1229, 907, 727.

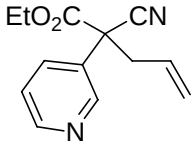


2-Allyl-2-(2-pyridinyl)-cyanoacetate (Table 1, entry 15): 2-Allyl-2-(2-pyridinyl)-cyanoacetate as a pale yellow oil (191 mg, 83%) from the reaction of **3** and bromobenzene followed by trapping with allylbromide at room temperature for 4h. ^{1}H NMR (400 MHz, $CDCl_3$): 8.62 (d, *J* = 4.8 Hz, 1 H), 7.78 (t, *J* = 7.6 Hz, 1 H), 7.62 (d, *J* = 8.4 Hz, 1 H), 7.30-7.33 (m, 1 H), 5.73-5.83 (m, 1 H), 5.19-5.26 (m, 2 H), 4.25-4.30 (m, 2 H), 3.17 (dd, *J* = 7.2, 14.0 Hz, 1 H), 3.04 (dd, *J* = 7.2, 14.0 Hz, 1 H), 1.25 (t, *J* = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.7, 152.2, 148.3, 136.0, 129.3, 122.1, 120.1, 119.6, 116.4, 61.8, 54.6, 39.3, 12.4. HRMS calcd (found) for $C_{13}H_{15}N_2O_2$ (MH^+): 231.1134 (231.1194). IR (film, cm^{-1}): 1741, 1462, 1239, 910, 730.

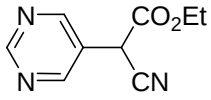


Ethyl 2-(3-pyridinyl)cyanoacetate (Table 1, entry 16):[6] Ethyl 2-(3-pyridinyl) cyanoacetate was isolated as a yellow solid (162 mg, 85%) from the reaction of **3** and 3-bromopyridine employing the standard procedure. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.71 (s, 1H), 8.65 (d, *J* = 3.6 Hz, 1H),

7.86 (d, J = 7.6 Hz, 1H), 7.40 (dd, J = 4.8, 8.0 Hz, 1H), 4.90 (s, 1H), 4.24-4.29 (m, 2 H), 1.29 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.4, 148.5, 147.2, 133.7, 124.6, 122.2, 113.1, 61.8, 39.4, 12.0. IR (film, cm^{-1}): 2169, 1746, 1543, 1262, 730.

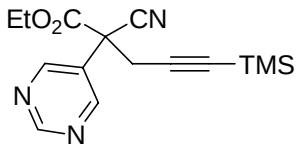


Ethyl 2-allyl-2-(3-pyridinyl)cyanoacetate (Table 1, entry 17): Ethyl 2-allyl-2-(3-pyridinyl) cyanoacetate was isolated as a pale yellow oil (191mg, 83%) from the reaction of **3** and 3-bromopyridine followed by trapping with allylbromide at room temperature for 4h. ^{1}H NMR (400 MHz, $CDCl_3$): 8.86 (s, 1 H), 8.65 (d, J = 4.4 Hz, 1 H), 7.92 (d, J = 8.0 Hz, 1 H), 7.40 (dd, J = 4.4, 8.0 Hz, 1 H), 5.71-5.81 (m, 1 H), 5.24-5.30 (m, 2 H), 4.22-4.34 (m, 2 H), 3.18 (dd, J = 7.2, 14.0 Hz, 1 H), 2.91 (dd, J = 7.2, 14.0 Hz, 1 H), 1.28 (t, J = 7.6 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.9, 147.6, 145.1, 131.7, 127.6, 127.5, 121.2, 119.4, 114.4, 61.2, 49.8, 39.6, 11.3. HRMS calcd (found) for $C_{13}H_{15}N_2O_2$ (MH^+): 231.1134 (231.1237). IR (film, cm^{-1}): 1744, 1222, 730.



Ethyl 2-(5-pyrimidinyl)-cyanoacetate (Table 1, entry 18): [7] Ethyl 2-(5-pyrimidinyl)-cyanoacetate was isolated as yellow solids (162mg, 85%) from the reaction of **3** and 3-bromopyridine followed by trapping with allylbromide at room temperature for 4h. ^{1}H NMR (400 MHz, $CDCl_3$): 9.28 (s, 1 H), 8.91 (s, 1 H), 4.95 (s, 1 H), 4.32 (q, J = 7.2 Hz, 2 H), 1.33 (t, J

= 7.6 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.4, 159.0, 156.2, 124.9, 113.8, 64.2, 39.1, 13.8. IR (film, cm^{-1}): 1745, 1414, 1252, 1229, 724.



Ethyl 2-(3-trimethylsilyl-2-propynyl)-2-(5-pyrimidinyl)-cyanoacetate (Table 1, entry 19): Ethyl 2-(3-trimethylsilyl-2-propynyl)-2-(5-pyrimidinyl)-cyanoacetate was isolated as a pale yellow oil (243mg, 81%) from the reaction of **3** and 3-bromopyridine followed by trapping with 1-bromo-3-trimethylsilylpropyne at 70°C for 4h. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.17 (s, 1 H), 8.85 (s, 2 H), 4.12-4.29 (m, 2 H), 3.18 (d, *J* = 16.8 Hz, 1 H), 3.36 (d, *J* = 16.8 Hz, 1 H), 1.23 (t, *J* = 7.2 Hz, 3 H), 0.00 (s, 9 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.6, 159.6, 155.6, 128.2, 115.9, 97.9, 92.6, 64.9, 50.5, 30.7, 14.3, 0.03. HRMS calcd (found) for $C_{15}H_{20}N_3O_2Si$ (MH^+): 302.1324 (302.1422). IR (film, cm^{-1}): 1749, 1419, 1234, 840, 725.

Reference:

[1]. Abdelaziz, A. S.; Dedenus, C. R. *J. Chem. Soc. Perkin. Trans. 1* **1993**, 293-298.

[2]. Pivsa-Art, S.; Fukui, Y.; Miura, M.; Nomura, M. *Bull. Chem. Soc. Jpn.* **1996**, *69*, 2039-2042.

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[5]. You, J.; Verkade, J. G. *Angew. Chem., Int. Ed. Engl.* **2003**, *42*, 5051-5053.

[6]. Sakamoto, T.; Kato, E.; Kondo, Y.; Yamanaka, H. *Chem. Pharm. Bull.* **1988**, *36*, 1664-1668.

[7]. Sakakibara, T.; Kume, T.; Shimohara, K.; Fujishima, H.; Hara, S.; Shimoda, T. *Heterocycles* **1990**, *31*, 459-472.

Acquisition Time (sec)	5.1120
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Date	28 Sep 2007 20:05:38
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Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2394.9553
Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

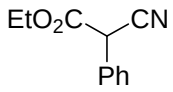

Table 1, entry 1

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Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12079.7080
Sweep Width (Hz)	28408.22
Temperature (degree C)	27.000

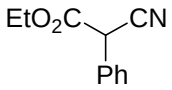

Table 1, entry 1

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Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
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Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

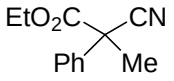

Table 1, entry 2

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Frequency (MHz)	100.65
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12079.7080
Sweep Width (Hz)	28408.22
Temperature (degree C)	26.900

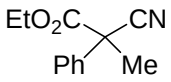

Table 1, entry 2

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Frequency (MHz)	400.23
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
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Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

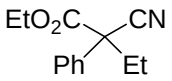

Table 1, entry 3

Acquisition Time (sec)	1.1535
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Frequency (MHz)	100.65
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12079.7080
Sweep Width (Hz)	28408.22
Temperature (degree C)	26.900

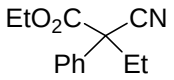

Table 1, entry 3

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Nucleus	1H
Number of Transients	32
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2373.8752
Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

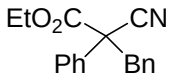

Table 1, entry 4

Acquisition Time (sec)	1.1535
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Frequency (MHz)	100.65
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12079.7080
Sweep Width (Hz)	28408.22
Temperature (degree C)	27.000

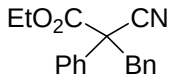

Table 1, entry 4

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Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	16384
Owner	smallmolecules
Points Count	16384
SW(cyclical) (Hz)	8223.68
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2465.9604
Sweep Width (Hz)	8223.18
Temperature (degree C)	27.000

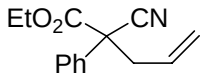

Table 1, entry 5

Acquisition Time (sec)	1.1535
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Nucleus	13C
Number of Transients	2048
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
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Sweep Width (Hz)	28408.22
Temperature (degree C)	24.900

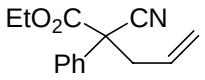

Table 1, entry 5

Acquisition Time (sec)	5.1120
Comment	xiangw
Date	11 Oct 2007 21:51:00
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Frequency (MHz)	400.23
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2355.0376
Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

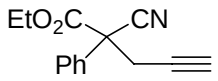

Table 1, entry 6

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Frequency (MHz)	100.65
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
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Sweep Width (Hz)	28408.22
Temperature (degree C)	26.900

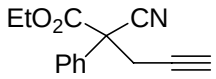

Table 1, entry 6

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Nucleus	1H
Number of Transients	32
Origin	Bruker BioSpin GmbH
Original Points Count	16384
Owner	smallmolecules
Points Count	16384
SW(cyclical) (Hz)	8223.68
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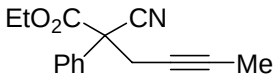

Table 1, entry 7

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Number of Transients	512
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Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12079.7080
Sweep Width (Hz)	28408.22
Temperature (degree C)	26.900

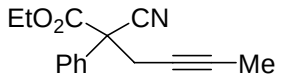

Table 1, entry 7

Acquisition Time (sec)	5.1120
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Frequency (MHz)	400.23
Nucleus	1H
Number of Transients	32
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2359.2029
Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

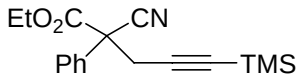

Table 1, entry 8

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Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12110.2158
Sweep Width (Hz)	28408.22
Temperature (degree C)	26.900

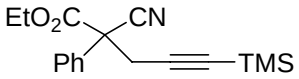

Table 1, entry 8

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Number of Transients	16
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Original Points Count	16384
Owner	smallmolecules
Points Count	16384
SW(cyclical) (Hz)	8223.68
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2458.1777
Sweep Width (Hz)	8223.18
Temperature (degree C)	27.000

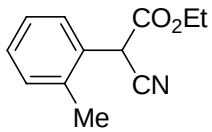

Table 1, entry 9

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Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12077.6240
Sweep Width (Hz)	28408.22
Temperature (degree C)	27.000

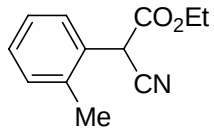

Table 1, entry 9

Acquisition Time (sec)	5.1120
Comment	xiangw
Date	29 Sep 2007 00:23:32
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-76-1_10.DX
Frequency (MHz)	400.23
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2388.3918
Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

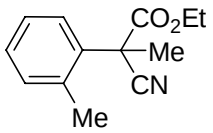

Table 1, entry 10

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	29 Sep 2007 02:41:00
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-76-1_11.DX
Frequency (MHz)	100.65
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12079.7080
Sweep Width (Hz)	28408.22
Temperature (degree C)	26.900

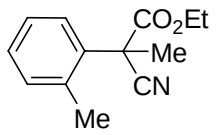

Table 1, entry 10

Acquisition Time (sec)	5.1120
Comment	xiangw
Date	08 Oct 2007 23:00:21
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-80-2_10.DX
Frequency (MHz)	400.23
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2399.5662
Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

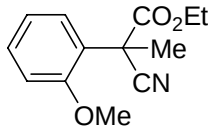

Table 1, entry 11

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	09 Oct 2007 00:42:19
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-80-2_11.DX
Frequency (MHz)	100.65
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12079.7080
Sweep Width (Hz)	28408.22
Temperature (degree C)	26.900

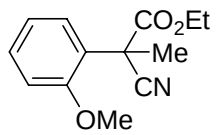

Table 1, entry 11

Acquisition Time (sec)	5.1120
Comment	xiangw
Date	11 Oct 2007 23:20:56
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-80-6_10.DX
Frequency (MHz)	400.23
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2383.6926
Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

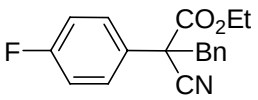

Table 1, entry 12

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	11 Oct 2007 23:48:09
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-80-6_11.DX
Frequency (MHz)	100.65
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12079.7080
Sweep Width (Hz)	28408.22
Temperature (degree C)	27.000

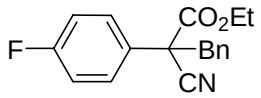

Table 1, entry 12

Acquisition Time (sec)	1.9924
Comment	xiangw
Date	28 Mar 2007 22:35:03
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\76065-70-1_12.DX
Frequency (MHz)	400.16
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	16384
Owner	smallmolecules
Points Count	16384
SW(cyclical) (Hz)	8223.68
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2466.3169
Sweep Width (Hz)	8223.18
Temperature (degree C)	27.000

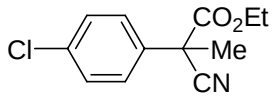

Table 1, entry 13

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	29 Mar 2007 03:25:24
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\76065-70-1_13.DX
Frequency (MHz)	100.63
Nucleus	13C
Number of Transients	2048
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	11967.1104
Sweep Width (Hz)	28408.22
Temperature (degree C)	26.800

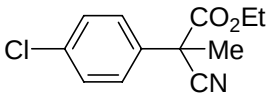

Table 1, entry 13

Acquisition Time (sec)	1.9924
Comment	xiangw
Date	15 Oct 2007 17:39:25
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-85-1_10.DX
Frequency (MHz)	400.16
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	16384
Owner	smallmolecules
Points Count	16384
SW(cyclical) (Hz)	8223.68
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2473.9233
Sweep Width (Hz)	8223.18
Temperature (degree C)	27.000

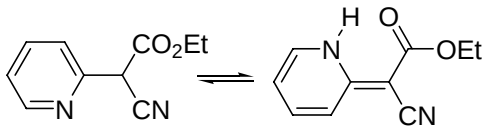

Table 1, entry 14

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	15 Oct 2007 21:19:39
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-80-5_10.DX
Frequency (MHz)	100.63
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12077.6240
Sweep Width (Hz)	28408.22
Temperature (degree C)	27.000

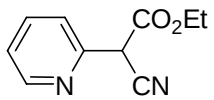

Table 1, entry 14

Acquisition Time (sec)	1.9924
Comment	xiangw
Date	21 Jun 2007 18:55:37
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-30-1_10.DX
Frequency (MHz)	400.16
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	16384
Owner	smallmolecules
Points Count	16384
SW(cyclical) (Hz)	8223.68
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2481.5908
Sweep Width (Hz)	8223.18
Temperature (degree C)	27.000

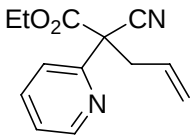

Table 1, entry 15

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	23 Jun 2007 06:04:56
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-30-1_11.DX
Frequency (MHz)	100.63
Nucleus	13C
Number of Transients	2048
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	11929.0908
Sweep Width (Hz)	28408.22
Temperature (degree C)	24.900

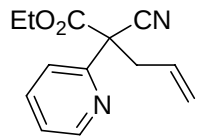

Table 1, entry 15

Acquisition Time (sec)	1.9924
Comment	xiangw
Date	13 Jul 2007 22:06:21
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-42-1_10.DX
Frequency (MHz)	400.16
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	16384
Owner	smallmolecules
Points Count	16384
SW(cyclical) (Hz)	8223.68
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2502.5874
Sweep Width (Hz)	8223.18
Temperature (degree C)	27.000

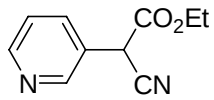

Table 1, entry 16

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	14 Jul 2007 06:03:55
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-42-1_11.DX
Frequency (MHz)	100.63
Nucleus	13C
Number of Transients	2048
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	11889.4307
Sweep Width (Hz)	28408.22
Temperature (degree C)	24.900

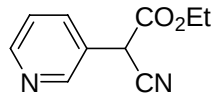

Table 1, entry 16

Acquisition Time (sec)	1.9924
Comment	xiangw
Date	16 Jul 2007 21:27:24
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-42-2_10.DX
Frequency (MHz)	400.16
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	16384
Owner	smallmolecules
Points Count	16384
SW(cyclical) (Hz)	8223.68
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2513.5522
Sweep Width (Hz)	8223.18
Temperature (degree C)	27.000

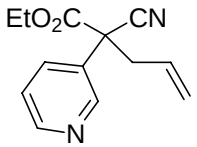

Table 1, entry 17

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	17 Jul 2007 03:25:32
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-42-2_11.DX
Frequency (MHz)	100.63
Nucleus	13C
Number of Transients	2048
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	11831.4971
Sweep Width (Hz)	28408.22
Temperature (degree C)	24.900

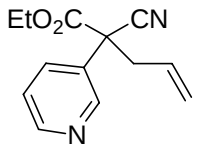

Table 1, entry 17

Acquisition Time (sec)	5.1120
Comment	xiangw
Date	04 Oct 2007 17:47:05
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-77-20_11.DX
Frequency (MHz)	400.23
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2433.3938
Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

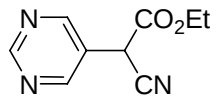

Table 1, entry 18

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	04 Oct 2007 18:14:19
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-77-20_12.DX
Frequency (MHz)	100.65
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12079.7080
Sweep Width (Hz)	28408.22
Temperature (degree C)	26.900

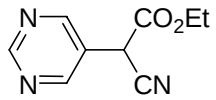

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Table 1, entry 18

Acquisition Time (sec)	5.1120
Comment	xiangw
Date	04 Oct 2007 22:39:28
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-77-21_11.DX
Frequency (MHz)	400.23
Nucleus	1H
Number of Transients	16
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	6410.26
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2374.3757
Sweep Width (Hz)	6410.06
Temperature (degree C)	27.000

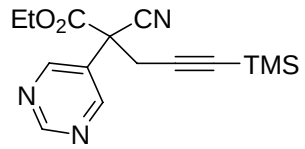

Table 1, entry 19

Acquisition Time (sec)	1.1535
Comment	xiangw
Date	04 Oct 2007 23:36:25
File Name	\\SCONE\NMR-ARCHIVE\JCAMP\XIANGW\2007\84693-77-21_12.DX
Frequency (MHz)	100.65
Nucleus	13C
Number of Transients	512
Origin	Bruker BioSpin GmbH
Original Points Count	32768
Owner	smallmolecules
Points Count	32768
SW(cyclical) (Hz)	28409.09
Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12127.6670
Sweep Width (Hz)	28408.22
Temperature (degree C)	27.000

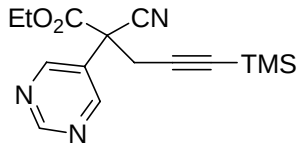

Table 1, entry 19