Supplementary Material (ESI) for Chemical Communications

Enantioselective Michael Addition of Malononitrile to Chalcone Catalyzed by Simple Quinine-Al(OiPr)3 Complex: a Simple Method for the Synthesis of Chiral 4-(H)-Pyran Derivative

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(A) General

^{1}H NMR spectra were recorded on commercial instruments (400 MHz). Chemical shifts are recorded in ppm relative to tetramethylsilane and with the solvent resonance as the internal standard. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, br = broad), coupling constants (Hz), integration. ^{13}C NMR data were collected on commercial instruments (100 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard. Enantiomer excesses were determined by chiral HPLC analysis on Daicel Chiralcel AS-H/ IA/IB in comparison with the authentic racemates. Optical rotations were reported as follows: $[\alpha]_D^{20}$ (c: g/100 mL, in solvent, λ= 589 nm). HRMS was recorded on a commercial apparatus (ESI Source). All the solvents were purified by usual methods before use.

(B) General procedure for chiral catalyst preparation

All of the alkaloids and metal reagents are commercially available.

(C) General procedure for the synthesis of enones (2a-2v)

The aldehyde (10 mmol) was added gradually to a solution of NaOH (0.5 g) in H_2O (10.0 mL) and ketone (10 mmol) in ethanol (15 mL) at 0 ℃. The mixture was then allowed to warm to room temperature and stirred for overnight. At the end of this period, $KHSO_4$ (1 N) solution was added to the flask until PH ≈ 6, followed by extraction with ether. The combined organic layers were dried over Na_2SO_4 and concentrated to give a solid which was purified by flash column chromatography using EtOAc–hexanes at last. [1] All spectroscopic data of the enones were identical to those reported in the literature. [2]

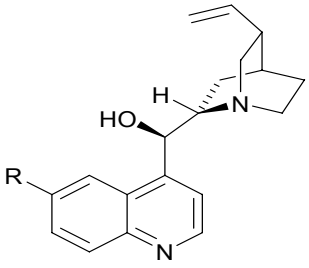
(D) General Procedure for the Asymmetric Catalytic Michael Reaction (3a-3v)

$Al(O^iPr)_3$ (0.01 mmol) was added to a dry reaction tube containing a suspension of quinine (3.2 mg, 0.01 mmol), chalcone (20.8 mg, 0.1 mmol) and 0.06 mL dry toluene. The mixture was stirred at 25 ^{o}C for 1 h to give a solution under nitrogen atmosphere. Then malononitrile (6.9 mg, 0.105 mmol) dissolved in toluene (0.4 mL) (if required, heated the toluene solvent) was added at 0 ^{o}C. After being stirred for 80 h, the reaction mixture was directly purified by column chromatography on silica gel eluted (ethyl acetate: petroleum ether = 1:5) to afford the corresponding compound.

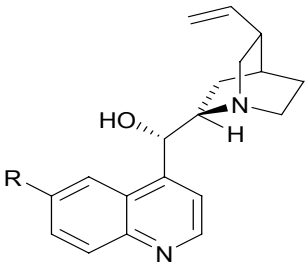
(E) General procedure for the synthesis of 4-(H)-pyran derivative

2-(3-Oxo-1,3-diphenylpropyl)malononitrile (0.274 mg, 1 mmol) was added in dry ethanol (5 mL), then piperidine (1.0 mL) was added. The resulting solution was stirred at 30 ^{o}C for 48 h. Following, the solvent was removed. The reaction mixture was directly purified by column chromatography on silica gel eluted (CH_2Cl_2: petroleum ether = 1:2) to afford the corresponding compound. [3]

(F) Optimization of conditions

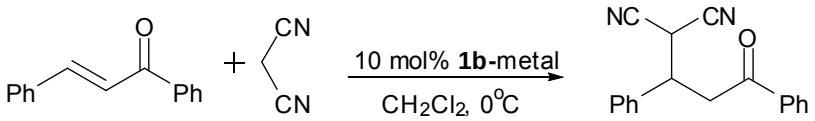


1a: R = H
1b: R = OMe



1c: R = H
1d: R = OMe

Screening of metals

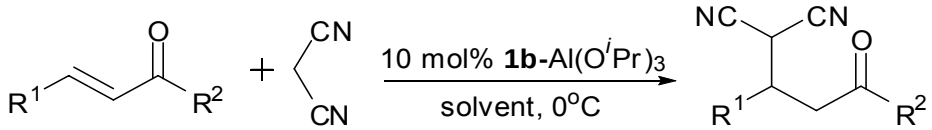


2a **3a**

Entry[a]	Metal	Yield[b] (%)	Ee[c] (%)
1	$Al(O^iPr)_3$	75	77
2	$AlEt_3$	80	39
3	$AlEt_2Cl$	35	18
4	$AlCl_3$	N. D.	
5	$Al_2(SO_4)_3$	N. D.	
6	$Ti(O^iPr)_4$	73	70
7	$Ni(acac)_2$	68	74
8	$Ni(ClO_4)_2{\cdot}6H_2O$	35	22
9	$Fe(acac)_3$	70	74
10	$Co(acac)_2$	54	59

[a] Unless noted otherwise, the reaction was carried out with **1b** (0.01 mmol), metal (0.01 mmol), **2a** (0.1 mmol) and malononitrile (0.105 mmol) in CH_2Cl_2 (1.0 mL) at 0 °C under nitrogen for 80 h. [b] Isolated yield. [c] Determined by chiral HPLC.

Screening of solvent

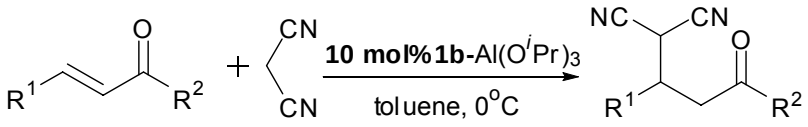


2a~v **3a~v**

Entrya	Catalyst	Solvent	Yieldb (%)	Eec (%)
1	1b + Al(OiPr)3	CH2Cl2	75	77
2	1b + Al(OiPr)3	Et2O	66	29
3	1b + Al(OiPr)3	CHCl3	41	51
4	1b + Al(OiPr)3	THF	55	15
5	1b + Al(OiPr)3	MeOH	69	race
6	1b + Al(OiPr)3	cyclohexane	43	19
7	1b + Al(OiPr)3	toluene	92	90
8	1b + Al(OiPr)3	o-xylene	90	89

[a] Unless noted otherwise, the reaction was carried out with **1b** (0.01 mmol), metal (0.01 mmol), **2a** (0.1 mmol) and malononitrile (0.105 mmol) in solvent (1.0 mL) at 0 °C under nitrogen for 80 h. [b] Isolated yield. [c] Determined by chiral HPLC.

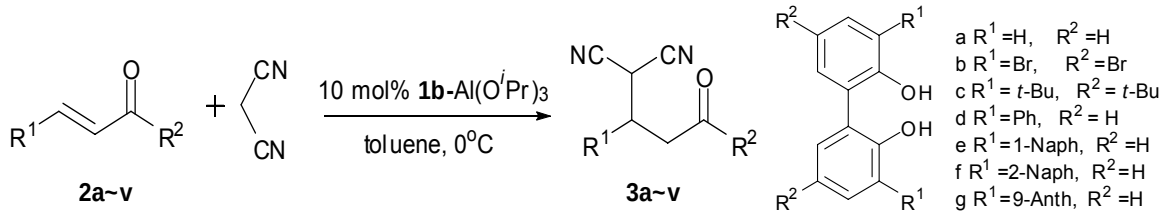
Optimization of the ratio between ligand and metal



Entry[a]	Ligand/metal	Yield[b] (%)	Ee[c] (%)
1	2:1	90	83
2	1:2	93	87
3	1:0	51	80
4	1:1	92	90

[a] Unless noted otherwise, the reaction was carried out with **2a** (0.1 mmol) under nitrogen at 25℃ for 1 h, then malononitrile (0.105 mmol) in toluene (1.0 mL) at 0 °C for 80 h.. [b] Isolated yield. [d] Determined by chiral HPLC.

Optimization of additive

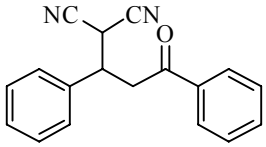


Entry[a]	Additive	Yield[b] (%)	Ee[c] (%)
1	a	71	85
2	b	67	86
3	c	74	85
4	d	73	86
5	f	77	86
6	g	76	85
7	h	70	85
8	R-binol	70	72
9	S-binol	73	-62
10	*o*-dihydroxybenzene	81	86
11	*m*-dihydroxybenzene	80	84
12	*p*-dihydroxybenzene	78	84
13	triethylanmine	92	77
14	3 Å MS	85	88
15	4 Å MS	87	88
16	5 Å MS	87	88

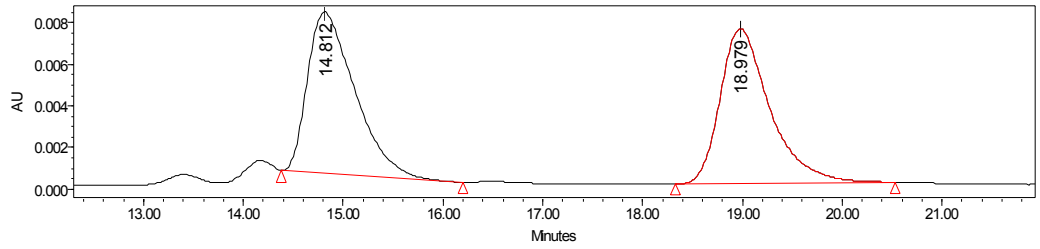
[a] Unless noted otherwise, the reaction was carried out with **1b** (0.01 mmol), $Al(O^iPr)_3$ (0.01 mmol), **2a** (0.1 mmol) and additive (entries 1-13, 0.01 mmol, entries 14-16, 10 mg) and malononitrile (0.105 mmol) in toluene (1.0 mL) at 0 °C under nitrogen for 80 h. [b] Isolated yield. [c] Determined by chiral HPLC

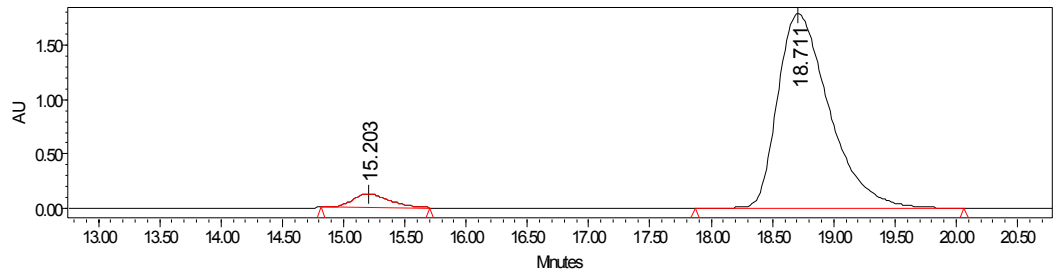
(G) The analytical and spectral characterization data of reaction products

2-(3-Oxo-1, 3-diphenylpropyl)malononitrile (3a)

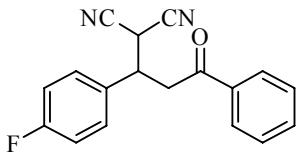


white solid; 92% yield, 90% ee; $[\alpha]_D^{20}$ = -12.59 (*c* 0.270 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 18.711 min (major) and 15.203 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.69 (2H, m, O=C-CH_2), 3.96 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.4 Hz, Ar-CH), 4.61 (1H, d, *J* = 5.2 Hz, NC-CH), 7.39-7.83 (10H, m, Ar-H) ppm.



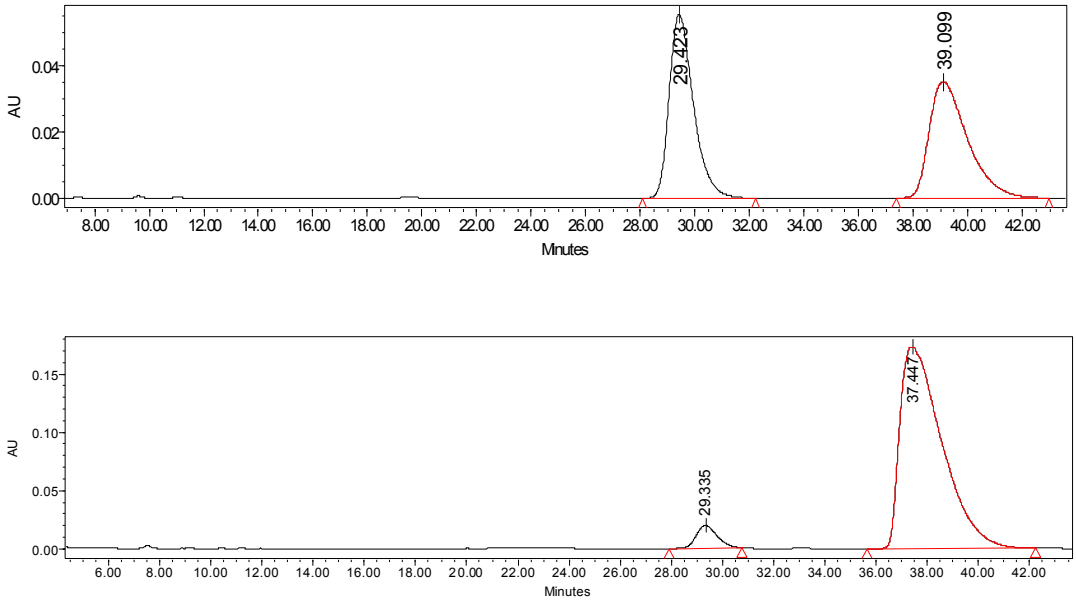


2-(1-(4-Fluorophenyl)-3-Oxo-3-phenylpropyl)malononitrile (3b)

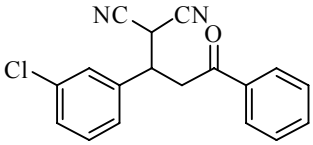


white solid; 94% yield, 89% ee; $[\alpha]_D^{20}$ = -5.56 (*c* 0.288 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AS-H, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 37.447 min (major) and 29.335 min (minor);

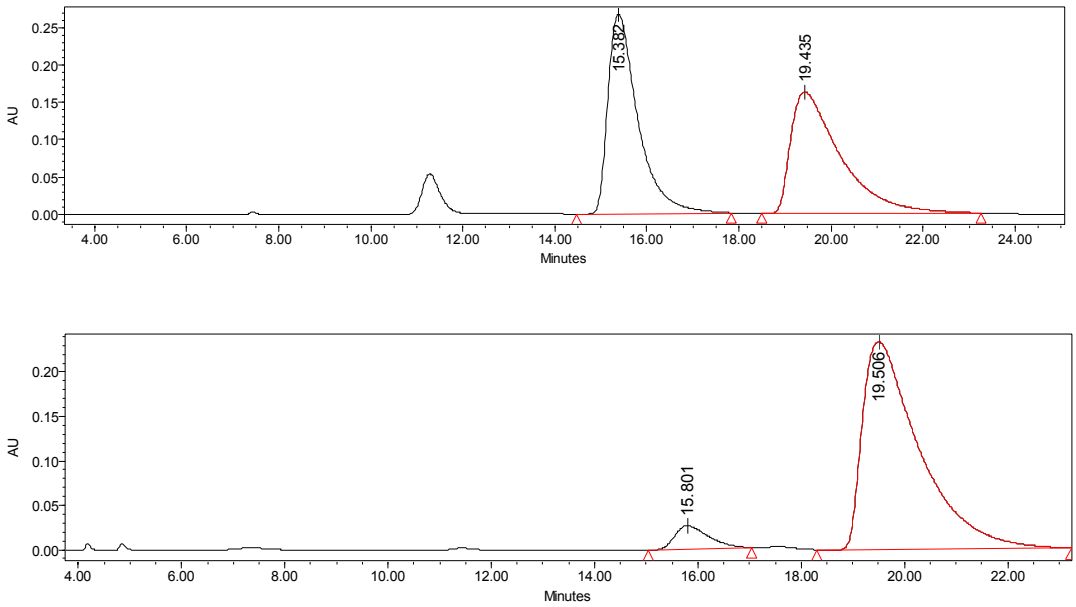
^{1}H NMR (400 MHz, $CDCl_3$), δ 3.66 (2H, m, O=C-CH_2), 3.96 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.4 Hz, Ar-CH), 5.34 (1H, d, J = 4.8 Hz, NC-CH), 7.11-7.96 (9H, m, Ar-H) ppm.



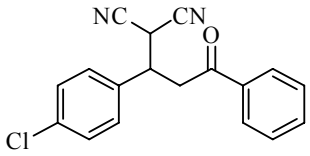
2-(1-(3-Chlorophenyl)-3-Oxo-3-phenylpropyl)malononitrile (3c)



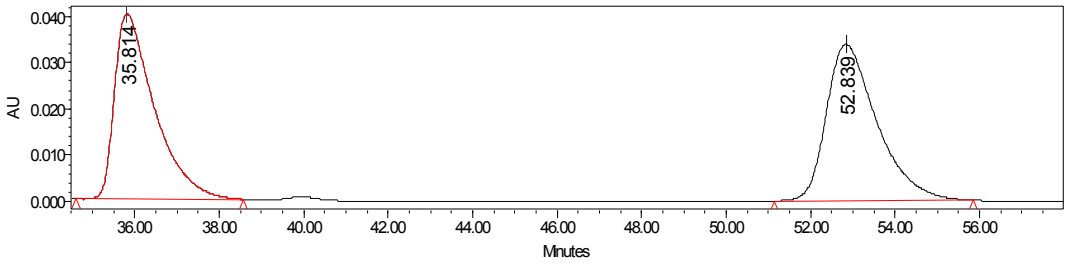
white solid; 92% yield, 87% ee; $[\alpha]_D^{20}$ = -13.48 (*c* 0.178 in CH_2Cl_2). HPLC DAICEL CHIRALCEL AS-H, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 19.506 min (major) and 15.801 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.66 (2H, m, O=C-CH_2), 3.94 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.4 Hz, Ar-CH), 4.68 (1H, d, J = 4.8 Hz, Ar-CH), 7.35-7.98 (9H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.5, 39.9, 40.8, 111.4, 111.5, 126.3, 128.1, 128.2, 128.9, 129.5, 130.6, 134.3, 135.2, 135.6, 138.4, 196.2 ppm; HRMS (ESI-TOF) calcd for $C_{18}H_{13}ClN_2O$ ([M-H^+]) = 307.0643, Found 307.0638.

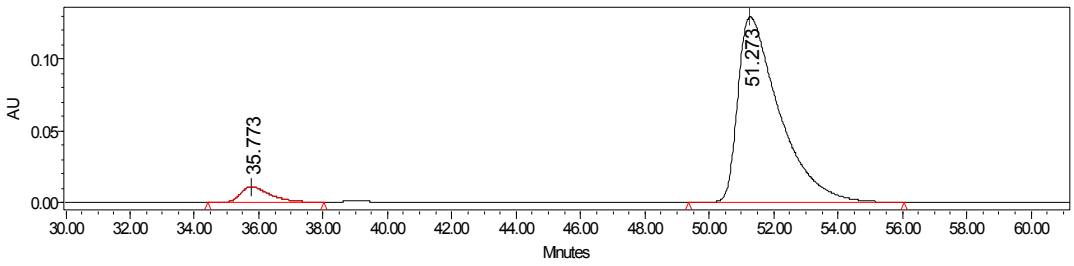


2-(1-(4-Chlorophenyl)-3-Oxo-3-phenylpropyl)malononitrile (3d)

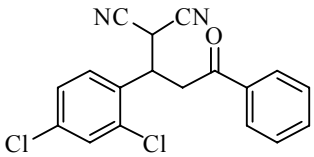


white solid; 89% yield, 89% ee; $[\alpha]_D^{20}$ = -5.15 (*c* 0.194 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 5/95, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 51.273 min (major) and 35.773 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.65 (2H, m, O=C-CH_2), 3.95 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.8 Hz, Ar-CH), 4.31 (1H, d, *J* = 5.2 Hz, NC-CH), 7.38-7.97 (9H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.1, 36.7, 39.2, 111.1, 111.5, 128.13, 128.19, 128.8, 129.0, 130.4, 132.7, 134.3, 134.9, 135.5, 195.6 ppm; HRMS (ESI-TOF) calcd for $C_{18}H_{13}ClN_2O$ ([M-H$^+$]) = 307.0643, Found 307.0641.

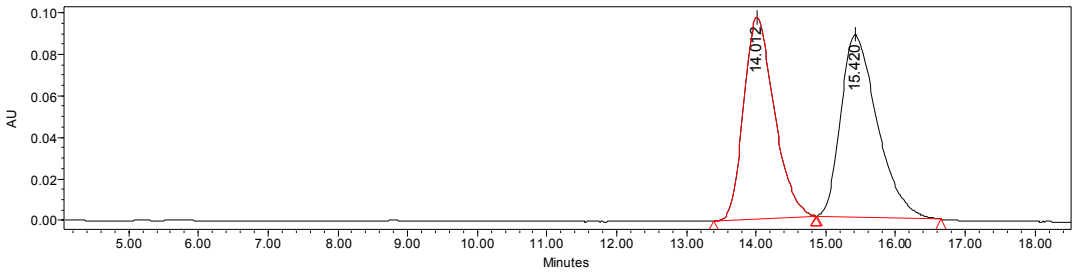


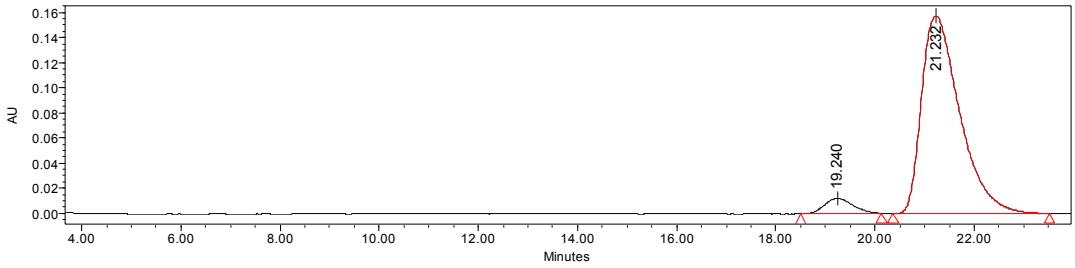


2-(1-(2, 4-Dichlorophenyl)-3-Oxo-3-phenylpropyl)malononitrile (3e)

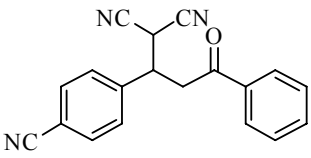


white solid; 90% yield, 89% ee; $[\alpha]_D^{20}$ = 10.34 (*c* 0.232 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AS-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 21.232 min (major) and 19.240 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.70 (2H, m, O=C-CH_2), 4.63 (2H, m, Ar-CH and NC-CH), 7.31-7.969 (8H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.1, 36.7, 39.2, 111.1, 111.5, 128.13, 128.19, 128.8, 129.0, 130.4, 132.3, 134.9, 135.5, 195.6 ppm; HRMS (ESI-TOF) calcd for $C_{18}H_{12}Cl_2N_2O$ ([M-H$^+$]) = 341.0254, Found 341.0256.

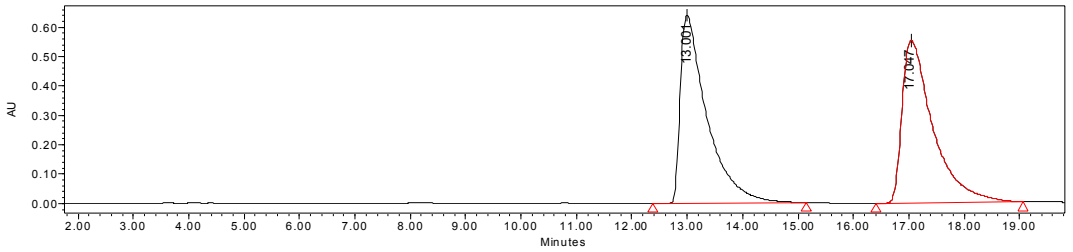


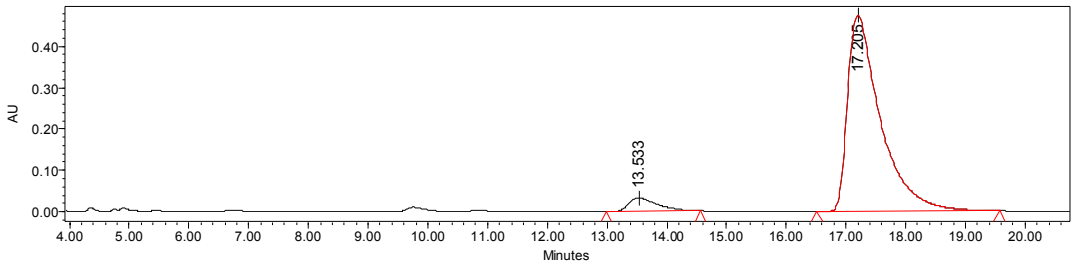


2-(1-(4-Cyanophenyl)-3-Oxo-3-phenylpropyl)malononitrile (3f)

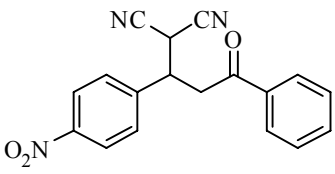


white solid; 93% yield, 89% ee; $[\alpha]_D^{20}$ = -5.88 (*c* 0.170 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 17.205 min (major) and 13.533 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.68 (2H, m, O=C-CH_2), 4.03 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.4 Hz, Ar-CH), 4.66 (1H, d, *J* = 5.2 Hz, NC-CH), 7.49-7.97 (9H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.3, 39.6, 41.1, 111.1, 111.3, 113.3, 118.0, 128.1, 129.04, 129.05, 133.1, 134.5, 135.4, 141.4, 195.8 ppm; HRMS (ESI-TOF) calcd for $C_{19}H_{13}N_3O$ ([M-H$^+$]) = 298.0986, Found 298.0980.

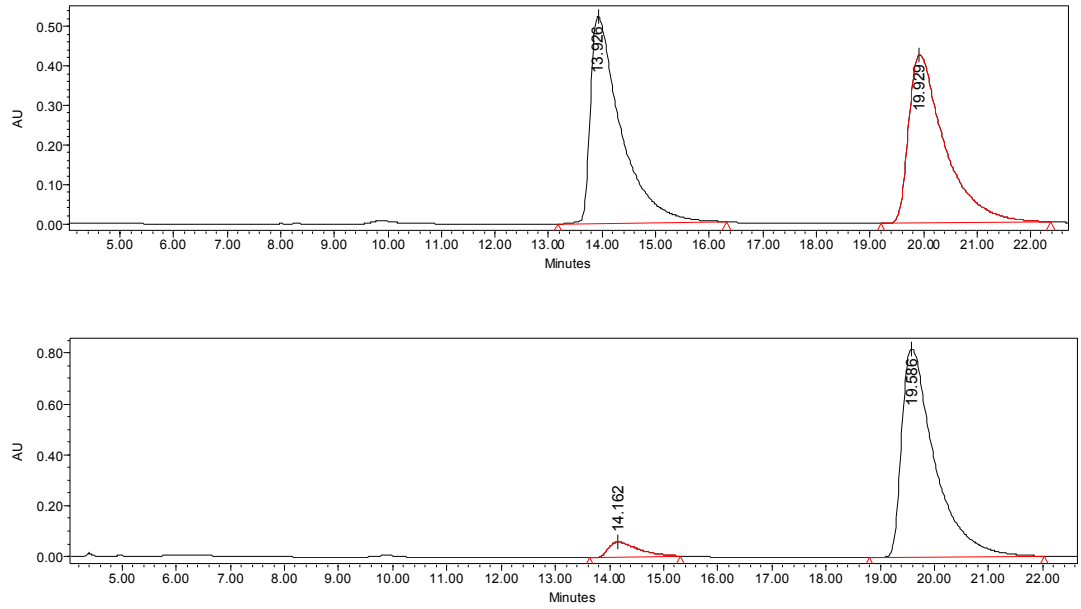




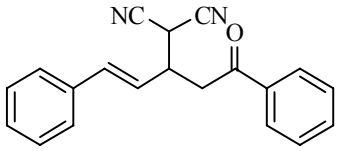
2-(1-(4-Nitrophenyl)-3-Oxo-3-phenylpropyl)malononitrile (3g)



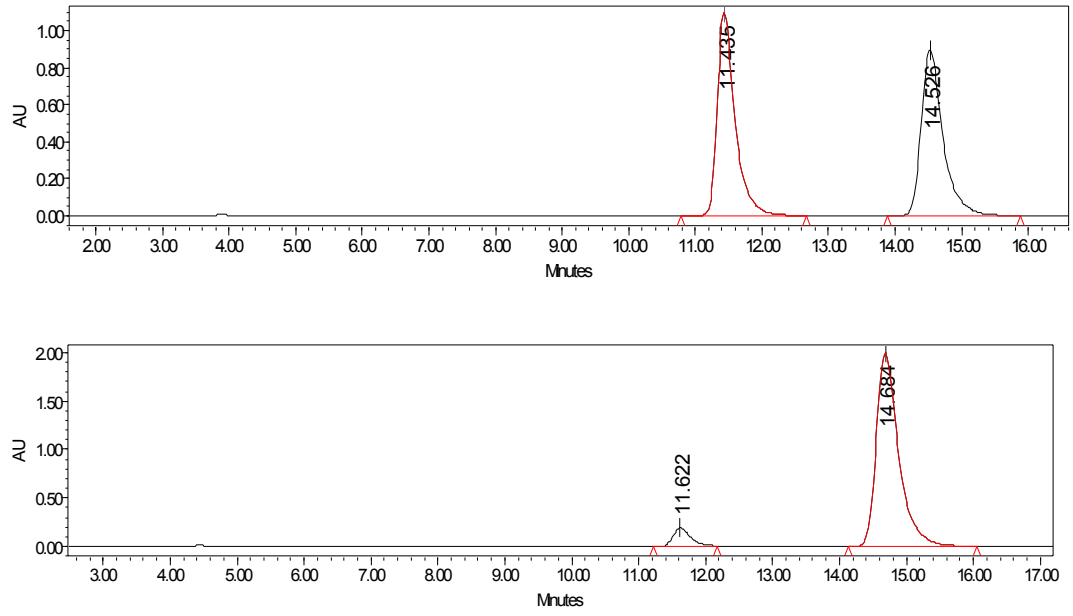
white solid; 95% yield, 89% ee; $[\alpha]_D^{20}$ = -7.14 (*c* 0.042 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, λ = 210 nm, retention time: 19.586 min (major) and 14.162 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.71 (2H, m, O=C-CH_2), 4.10 (1H, dt, J_1 = 5.6 Hz, J_2 = 8.4 Hz, Ar-CH), 4.69 (1H, d, *J* = 4.8 Hz, NC-CH), 7.50-7.96 (9H, m, Ar-H) ppm.



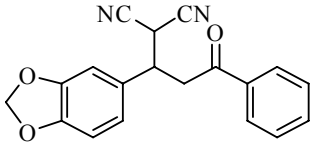
2-(*Z*-1-styrene -3-Oxo-1,3-phenylpropyl)malononitrile (3i)



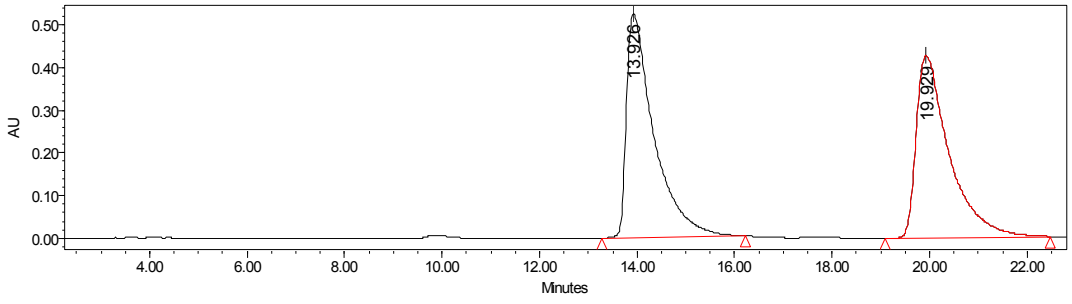
colorless liquid; 94% yield, 87% ee; $[\alpha]_D^{20}$ = -0.65 (*c* 0.154 in CH_2Cl_2). HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 15/85, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 14.684 min (major) and 11.622 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.45 (2H, m, OC=CH_2), 3.57 (1H, m, C=CH-C*H*), 4.60 (1H, d, *J*= 4.4 Hz, OC=CH_2), 6.26 (1H, dd, J_1 = 9.2 Hz, J_2 = 15.6 Hz, Ar-CH=C*H*), 6.81 (1H, d, *J* = 15.6 Hz, Ar-C*H*=CH), 7.30-8.00 (10H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.5, 39.6, 40.0, 111.6, 112.0, 123.3, 126.8, 127.5, 128.6, 128.7, 128.9, 129.0, 134.2, 135.3, 135.8, 136.3, 196.7 ppm; HRMS (ESI-TOF) calcd for $C_{20}H_{16}N_2O$ ([M-H$^+$]) = 299.1190, Found 299.1186.

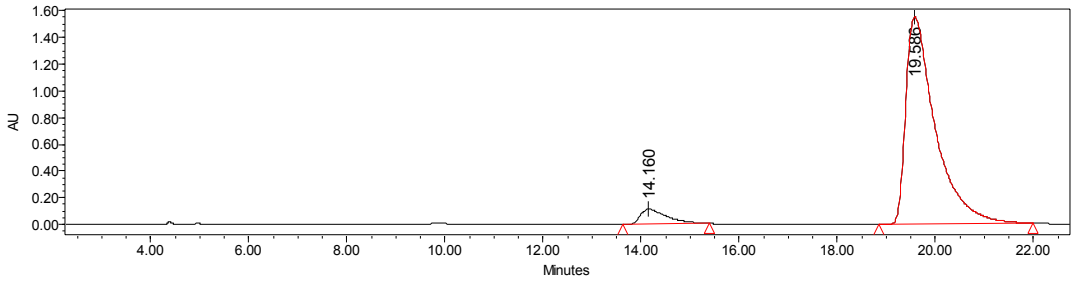


2-(1-(3, 4-Methylenedioxyphenyl)-3-Oxo-3-phenylpropyl)malononitrile (3h)

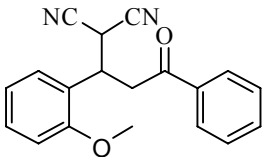


white solid; 91% yield, 88% ee; $[\alpha]_D^{20}$ = -11.20 (*c* 0.116 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 19.586 min (major) and 14.160 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.65 (2H, m, O=C-CH_2), 3.90 (1H, dt, J_1 = 4.8 Hz, J_2 = 8.4 Hz, Ar-CH), 4.62 (1H, d, *J* = 5.2 Hz, NC-CH), 6.02 (2H, s, O-CH_2-O), 6.85-8.00 (8H, m, Ar-H) ppm.

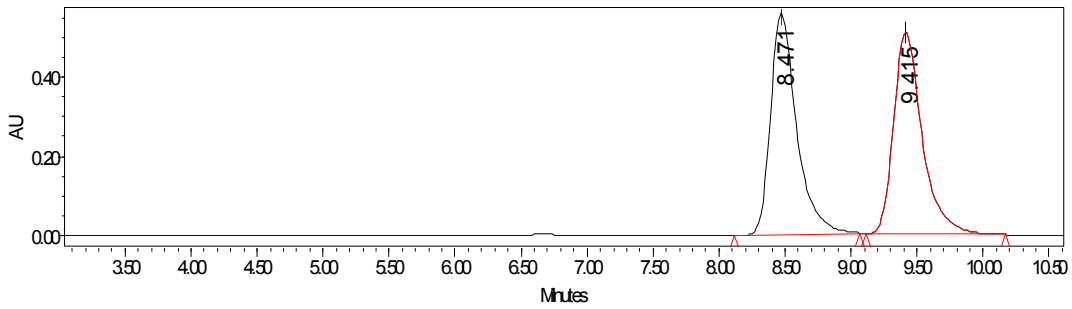


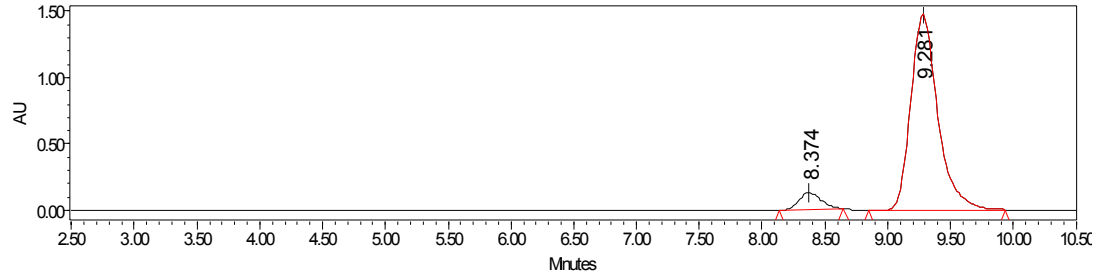


2-(1-(2-Methoxyphenyl)-3-Oxo-3-phenylpropyl)malononitrile (3j)

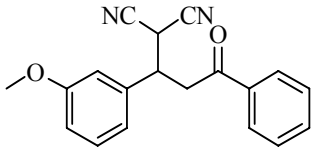


white solid; 89% yield, 87% ee; $[\alpha]_D^{20}$ = 24.22 (*c* 0.194 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane= 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 9.281 min (major) and 8.374 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.72 (2H, m, O=C-CH_2), 3.92 (3H, s, -OCH_3), 4.47 (1H, dd, J_1 = 6.8 Hz, J_2 = 13.6 Hz, Ar-CH), 4.69 (1H, d, *J* = 6.4 Hz, NC-CH), 6.95-7.99 (9H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.1, 36.0, 39.1, 55.4, 111.1, 112.1, 112.3, 121.2, 124.5, 128.0, 128.7, 128.8, 130.1, 133.8, 136.0, 156.8, 196.5 ppm; HRMS (ESI-TOF) calcd for $C_{19}H_{16}N_2O_2$ ([M-H^+]) = 303.1139, Found 303.1150.

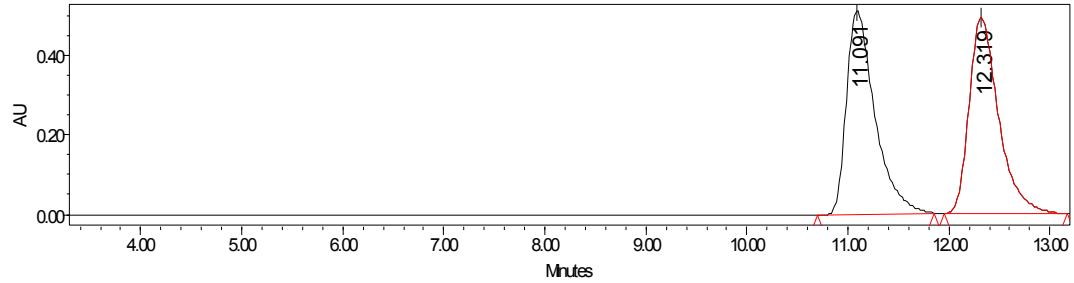


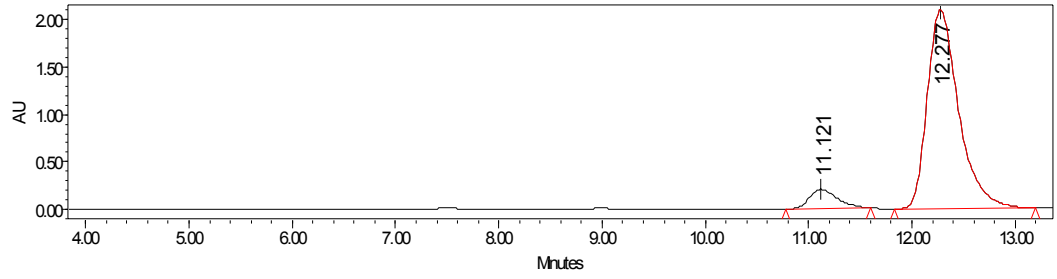


2-(1-(3-Methoxyphenyl)-3-Oxo-3-phenylpropyl)malononitrile (3k)

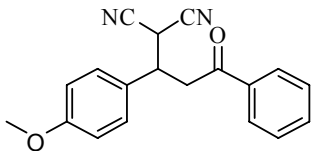


white solid; 84% yield, 84% ee; $[\alpha]_D^{20}$ = -8.33 (*c* 0.144 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 12.277 min (major) and 11.121 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.69 (2H, m, O=C-CH_2), 3.85 (3H, s, -OCH_3), 3.94 (1H, dt, J_1 = 5.6 Hz, J_2 = 8.0 Hz, Ar-CH), 4.66 (1H, d, J = 5.2 Hz, NC-CH), 6.94-7.98 (9H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.7, 40.1, 41.1, 55.3, 111.7, 111.8, 113.9, 114.3, 120.0, 128.1, 128.9, 130.4, 134.1, 135.7, 138.0, 160.1, 196.6 ppm; HRMS (ESI-TOF) calcd for $C_{19}H_{16}N_2O_2$ ([M-H^+]) = 303.1139, Found 303.1142.

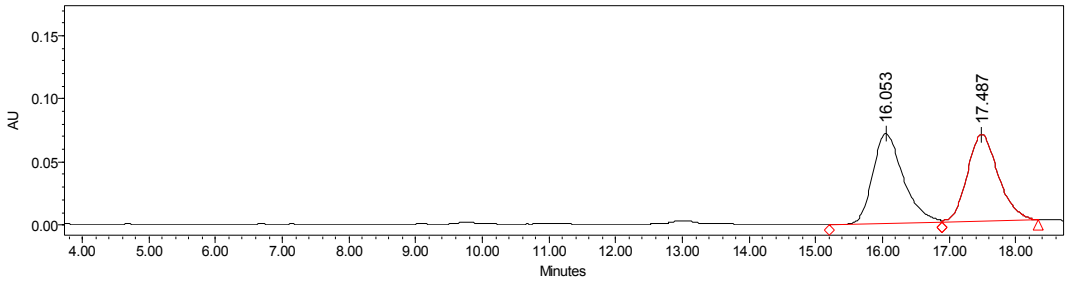


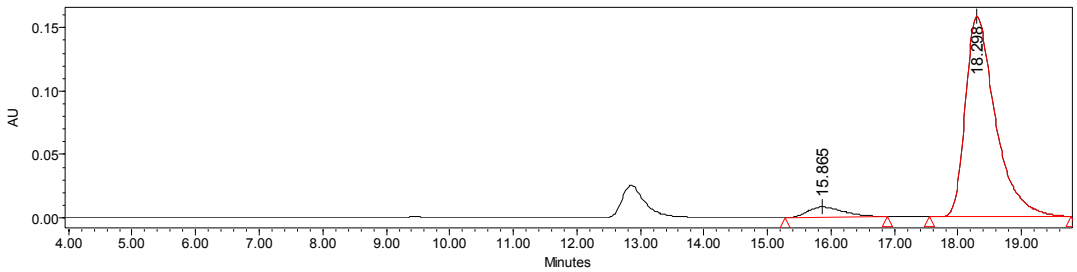


2-(1-(4-Methoxyphenyl)-3-Oxo-3-phenylpropyl)malononitrile (3l)

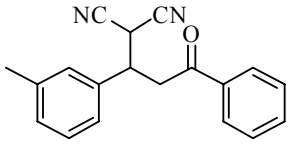


white solid; 89% yield, 88% ee; $[\alpha]_D^{20}$ = -0.48 (*c* 0.208 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 18.298 min (major) and 15.865 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.68 (2H, m, OC=CH_2), 3.82(3H, s, -OCH_3) 3.92 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.4 Hz, Ar-CH), 4.61 (1H, d, J = 5.2 Hz, NC-CH), 6.93-7.98 (9H, m, Ar-H) ppm.

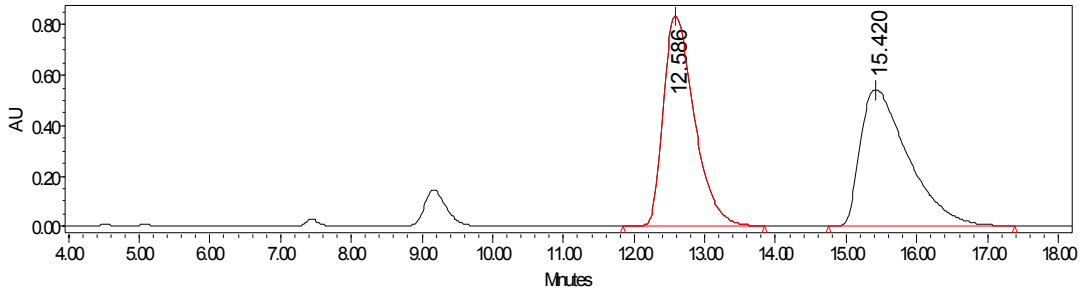


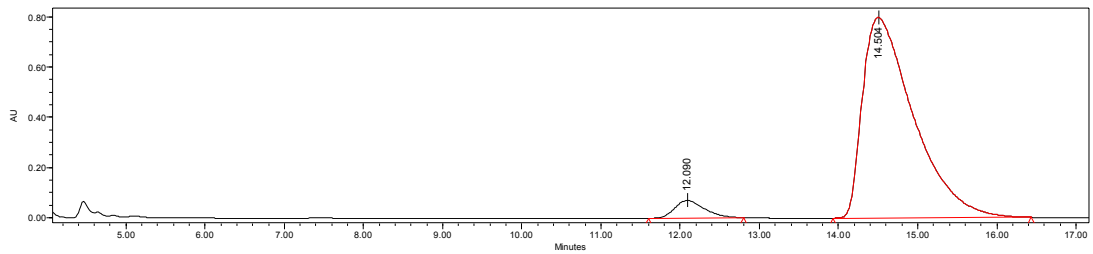


2-(1-(3-Methylphenyl)-3-Oxo-3-phenylpropyl)malononitrile (3m)

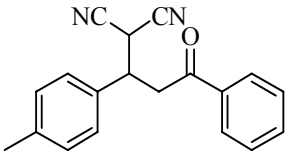


white solid; 84% yield, 90% ee; $[\alpha]_D^{20}$ = -20.83 (*c* 0.240 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AS-H, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 210 nm, retention time: 14.504 min (major) and 12.050 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.41 (3H, s, -CH_3), 3.69 (2H, m, O=C-CH_2), 3.94 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.4 Hz, Ar-CH), 4.66 (1H, d, J = 5.2 Hz, NC-CH), 7.22-8.01 (9H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 28.7, 40.1, 41.1, 111.7, 111.9, 124.9, 128.1, 128.6, 128.9, 129.2, 129.5, 134.1, 135.8, 136.5, 139.1, 196.7 ppm; HRMS (ESI-TOF) calcd for $C_{19}H_{16}N_2O$ ([M-H$^+$]) = 287.1190, Found 287.1191.

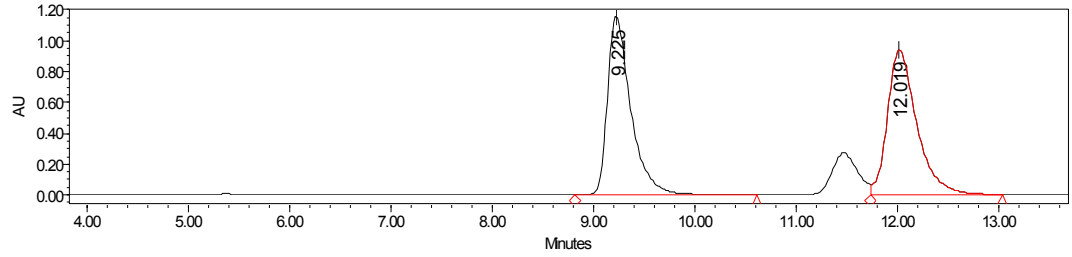


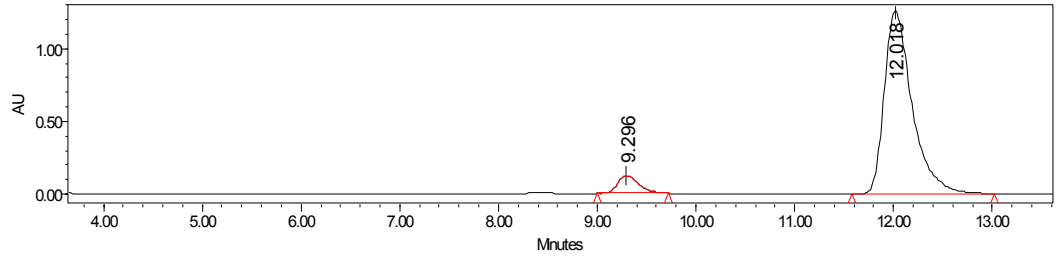


2-(1-(4-Methylphenyl)-3-Oxo-3-phenylpropyl)malononitrile (3n)

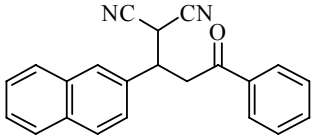


white solid; 84% yield, 87% ee; $[\alpha]_D^{20}$ = -2.41 (*c* 0.166 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ= 210 nm, retention time: 12.018 min (major) and 9.296 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.39 (3H, s, -CH_3), 3.68 (2H, m, O=C-CH_2), 3.95 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.4 Hz, Ar-CH), 4.65 (1H, d, *J* = 5.2 Hz, NC-CH), 7.25-8.00 (9H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.2, 28.9, 40.2, 40.9, 111.8, 111.9, 127.9, 128.1, 128.1, 128.9, 130.0, 133.5, 134.1, 135.8, 139.1, 139.1, 196.8 ppm; HRMS (ESI-TOF) calcd for $C_{19}H_{16}N_2O$ ([M+Na^+]) = 311.1155, Found 311.1165

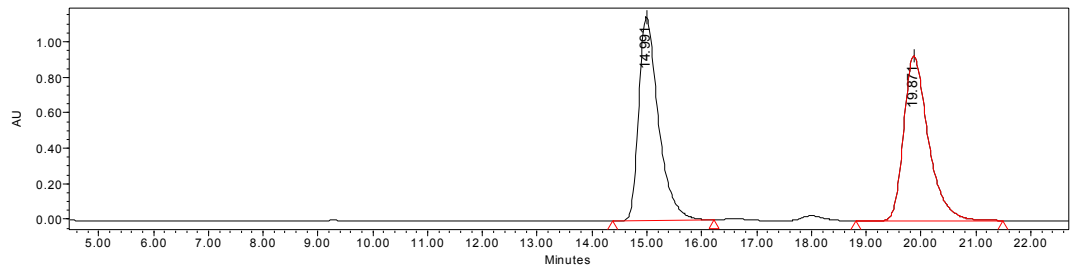


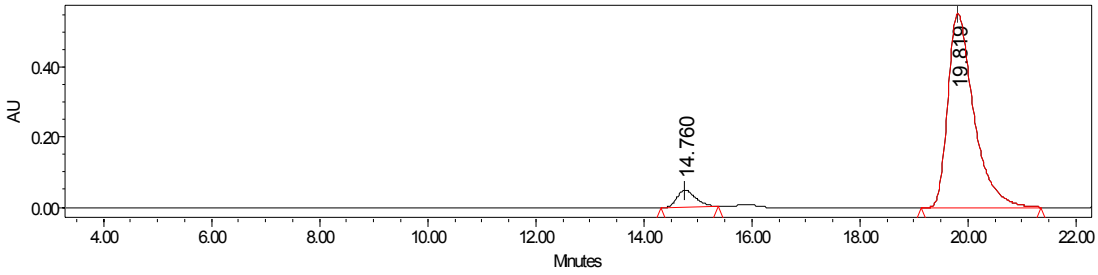


2-(1-(2-naphthyl)-3-Oxo-3-phenylpropyl)malononitrile (3o)

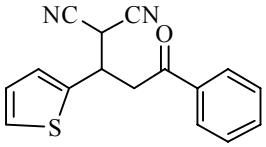


white solid; 86% yield, 88% ee; $[\alpha]_D^{20}$ = -4.55 (*c* 0.198 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 15/85, flow rate = 1.0 mL/min, λ = 210 nm, retention time: 19.819 min (major) and 14.760 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.72 (2H, m, O=C-CH_2), 4.07 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.4 Hz, Ar-CH), 4.66 (1H, d, *J* = 5.2 Hz, NC-CH), 7.41-7.93 (12H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.8, 40.3, 41.3, 111.79, 111.9, 125.2, 126.83, 126.87, 127.5, 127.7, 128.15, 128.19, 129.3, 133.4, 133.9, 134.1, 135.8, 196.6 ppm; HRMS (ESI-TOF) calcd for $C_{22}H_{16}N_2O$ ([M-H^+]) = 323.1190, Found 323.1176.

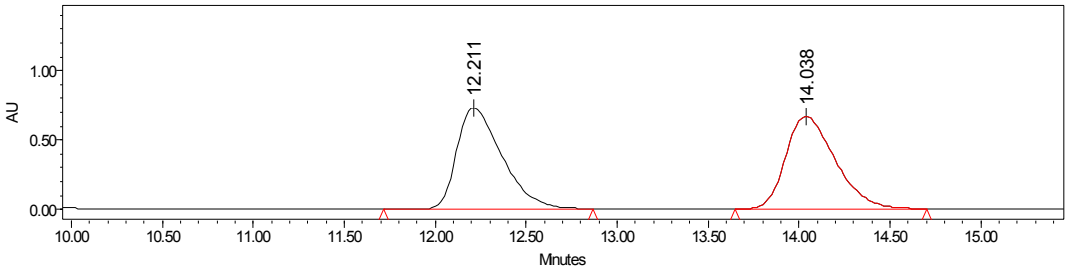


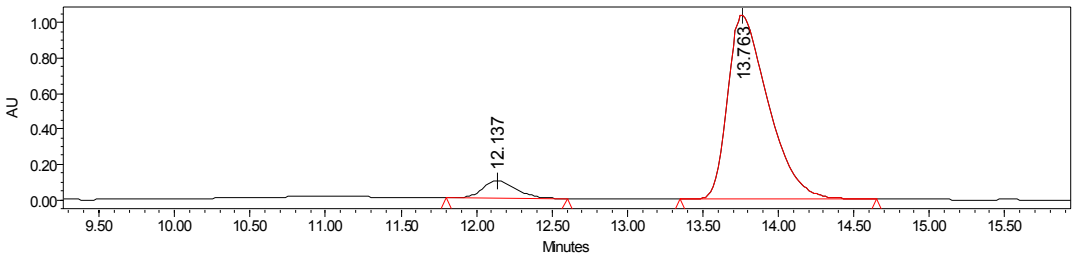


2-(1-(2-thienyl)-3-Oxo-3-phenylpropyl)malononitrile (3p)

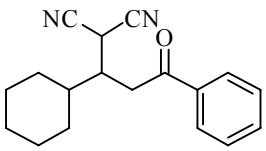


white solid; 83% yield, 86% ee; $[\alpha]_D^{20}$ = -1.47 (*c* 0.068 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 13.763 min (major) and 12.137 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.73 (2H, m, O=C-CH_2), 4.32 (1H, dt, J_1 = 4.8 Hz, J_2 = 8.4 Hz, Ar-CH), 4.71 (1H, d, *J* = 4.8 Hz, NC-CH), 7.03-8.01 (8H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 29.5, 37.2, 41.4, 111.4, 111.7, 126.0, 127.1, 127.4, 128.1, 128.9, 134.3, 135.6, 138.5, 196.2 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{12}N_2OS$ ([M-H^+]) = 279.0597, Found 279.0600.

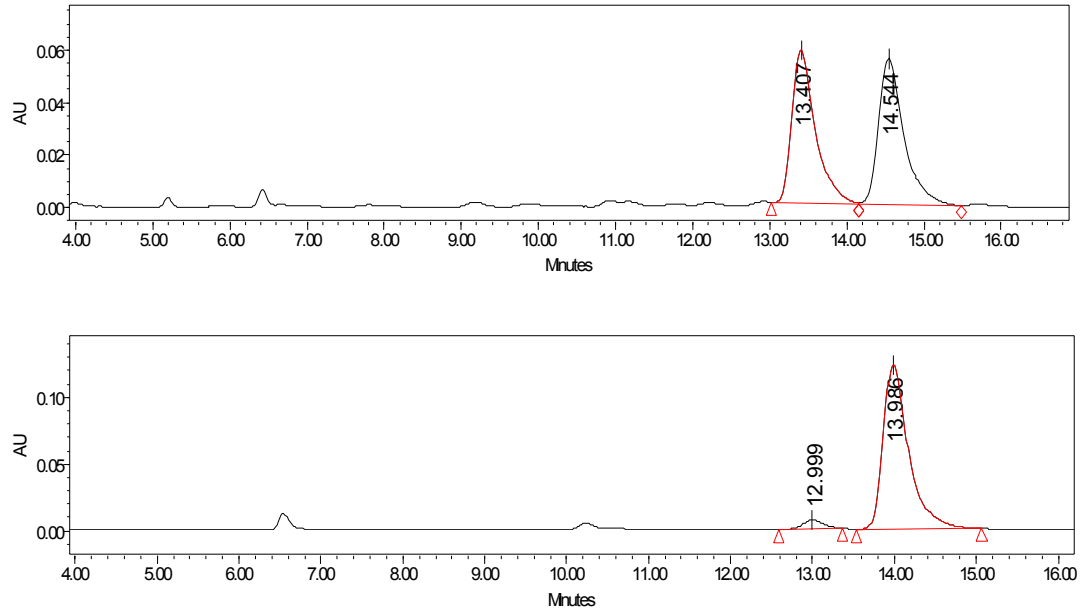




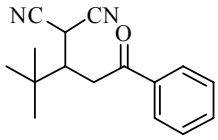
2-(1-cyclohexyl-3-Oxo-3-phenylpropyl)malononitrile (3q)



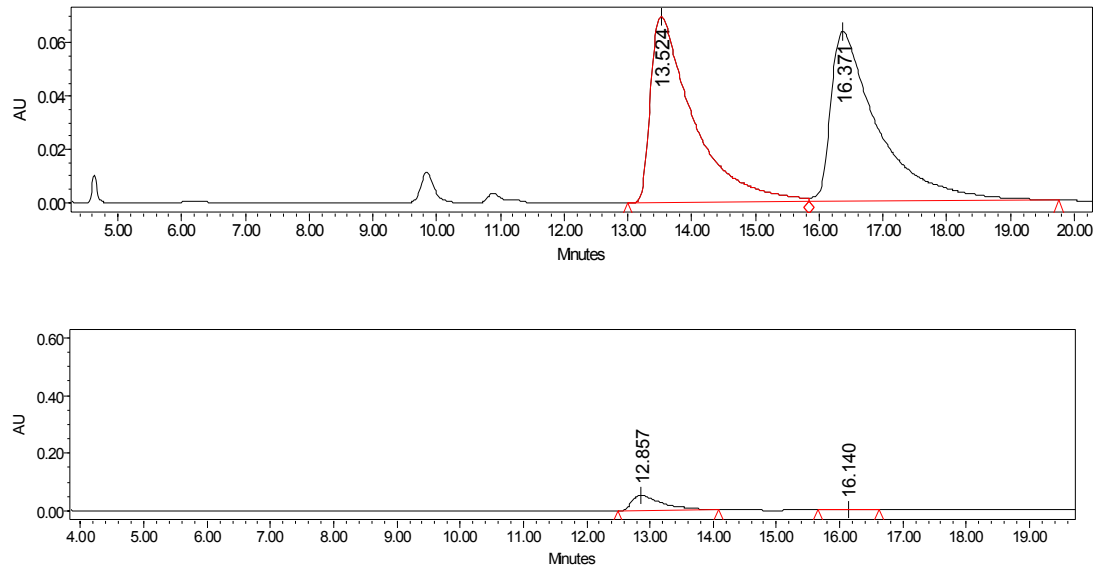
colorless liquid; 80% yield, 92% ee; $[\alpha]_D^{20}$ = 37.21 (*c* 0.086 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 5/95, flow rate = 1.0 mL/min, λ = 210 nm, retention time: 13.986 min (major) and 12.999 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.26 (5H, m, cyclohexyl-H), 1.79 (6H, m, cyclohexyl-H), 2.73 (1H, m, cyclohexyl-CH), 3.17 (1H, dd, J_1 = 5.2 Hz, J_2 = 12.0 Hz, O=C-CH), 3.36 (1H, dd, J_1 = 3.2 Hz, J_2 = 12.4 Hz, OC=CH), 4.37 (1H, d, *J* = 3.2 Hz, NC-CH), 7.49-7.99 (5H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3, 25.9., 26.1, 26.2, 29.3, 31.2, 36.9, 40.0, 40.2, 112.4, 112.5, 128.1, 128.9, 134.0, 136.0, 197.2 ppm; HRMS (ESI-TOF) calcd for $C_{18}H_{20}N_2O$ ([M+Na^+]) = 303.1468, Found 303.1468.



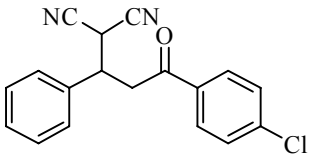
3-(2, 2-Methyl-5-Oxo-5-phenylpentyl)malononitrile (3r)



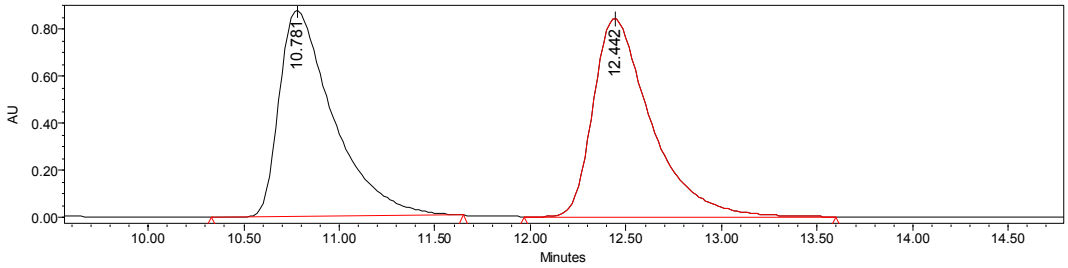
colorless liquid; 72% yield, 93% ee; $[\alpha]_D^{20}$ = 38.10 (*c* 0.042 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 5/95, flow rate = 1.0 mL/min, λ = 210 nm, retention time: 12.857 min (major) and 16.140 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.10 (9H, s, $(CH_3)_3C$), 2.93 (1H, m, $(CH_3)_3C$-C*H*), 3.19 (1H, dd, J_1 = 4.8 Hz, J_2 = 12.4 Hz, OC=CH), 3.41 (1H, dd, J_1 = 3.2 Hz, J_2 = 12.4 Hz, O=C-CH), 4.09 (1H, d, J = 1.6 Hz, NC-CH), 7.50-8.02 (5H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.0, 27.7, 34.3, 37.2, 44.4, 113.00, 113.04, 128.2, 128.9, 133.9, 136.1, 196.7 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{18}N_2O$ ([M+Na^+]) = 277.1311, Found 277.1319.

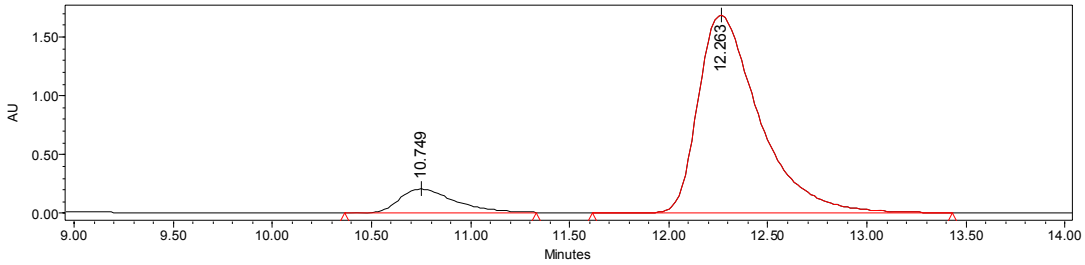


2-(3-(4-Chlorophenyl)-3-Oxo-3-phenylpropyl)malononitrile (3s)

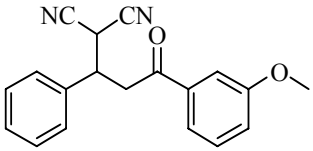


white solid; 88% yield, 80% ee; $[\alpha]_D^{20}$ = 7.87 (*c* 0.254 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 12.263 min (major) and 10.749 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.64 (2H, m, OC=CH_2), 3.95 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.0 Hz, Ar-CH), 4.62 (1H, d, *J* = 5.2 Hz, NC-CH), 7.40-7.92 (9H, m, Ar-H) ppm.

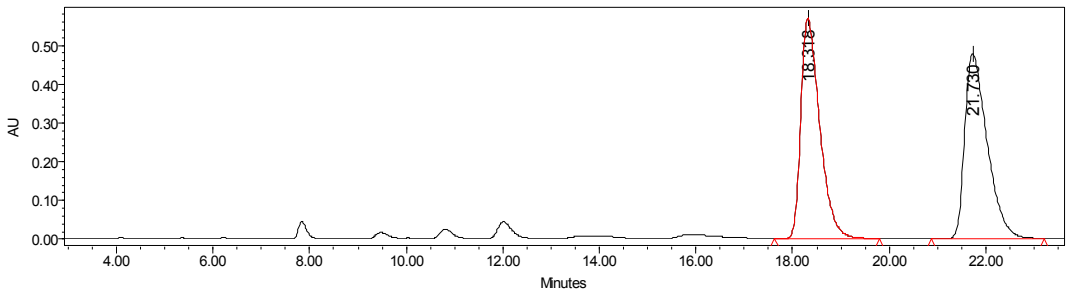


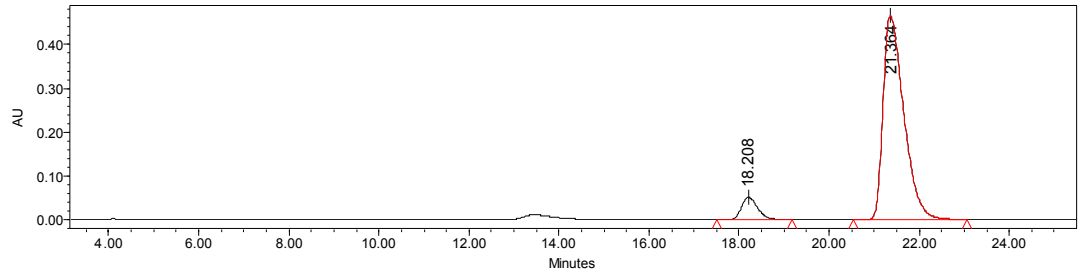


2-(3-(3-Methoxyphenyl)-3-Oxo-3-phenylpropyl)malononitrile (3t)

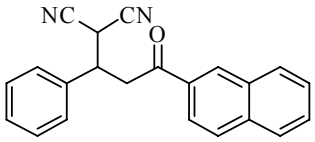


white solid; 84% yield, 84% ee; $[\alpha]_D^{20}$ = -4.17 (*c* 0.120 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 21.364 min (major) and 18.208 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.64 (2H, m, O=C-CH_2), 3.91 (3H, s, -OCH_3), 3.96 (1H, dd, J_1 = 5.2 Hz, J_2 = 9.6 Hz, Ar-H), 4.72 (1H, d, *J* = 5.2 Hz, NC-CH), 6.96-7.98 (9H, m, Ar-H) ppm. HRMS (ESI-TOF) calcd for $C_{19}H_{16}N_2O_2$ ([M-H^+]) = 303.1139, Found 303.1132.

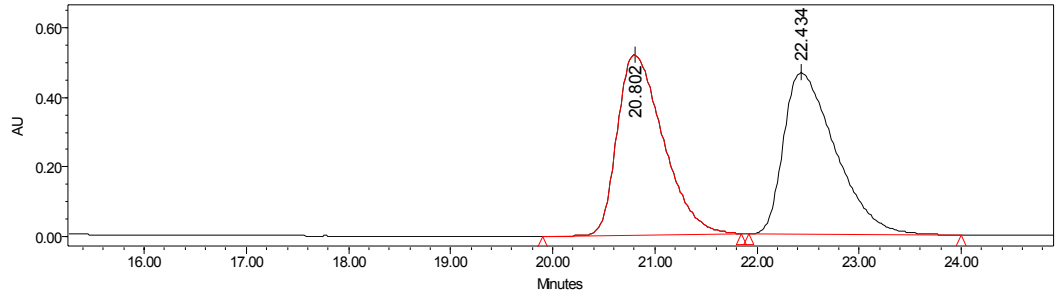


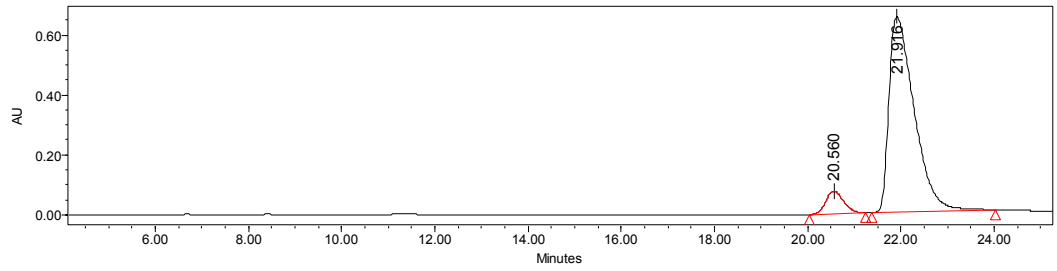


2-(3-(2-naphthyl)-3-Oxo-3-phenylpropyl)malononitrile (3u)

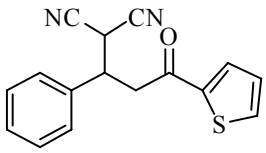


white solid; 97% yield, 85% ee; $[\alpha]_D^{20}$ = 26.70 (*c* 0.206 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 21.916 min (major) and 20.560 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.87 (2H, m, O=C-CH_2), 4.04 (1H, dt, J_1 = 5.2 Hz, J_2 = 8.0 Hz, Ar-CH), 4.72 (1H, d, *J* = 5.2 Hz, NC-CH), 7.43-8.52 (12H, m, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.8, 40.1, 41.3, 111.7, 111.9, 123.3, 127.2, 127.8, 128.0, 128.9, 129.1, 129.2, 129.3, 129.7, 130.1, 132.3, 133.1, 136.0, 136.6, 196.5 ppm; HRMS (ESI-TOF) calcd for $C_{22}H_{16}N_2O$ ([M-H^+]) = 323.1190, Found 323.1183.

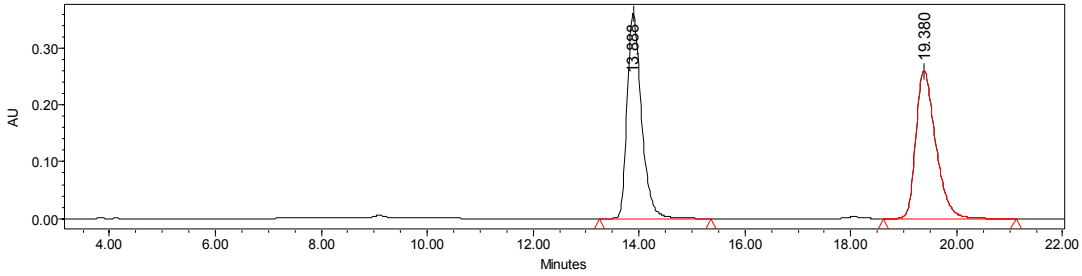


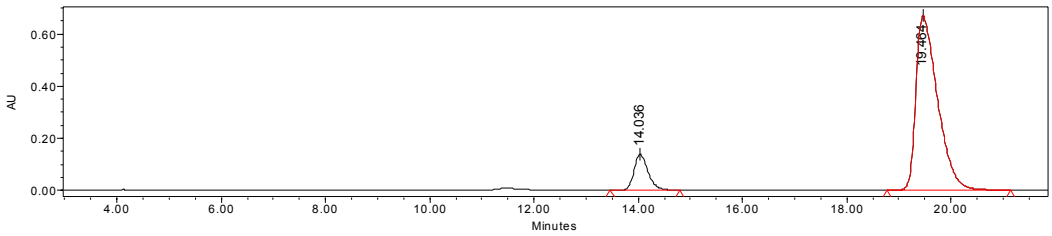


2-(3-(2-thienyl)-3-Oxo-3-phenylpropyl)malononitrile (3v)

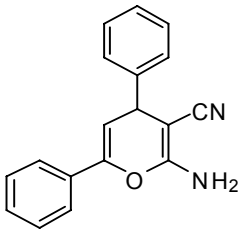


white solid; 92% yield, 77% ee; $[\alpha]_D^{20}$ = -1.80 (*c* 0.278 in CH_2Cl_2); HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 19.464 min (major) and 14.036 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.64 (2H, m, O=C-CH_2), 3.95 (1H, dt, J_1= 5.2 Hz, J_2= 8.4 Hz, Ar-CH), 4.67 (1H, d, *J* = 5.2 Hz, NC-CH), 7.18-7.80 (8H, m, Ar-H) ppm.



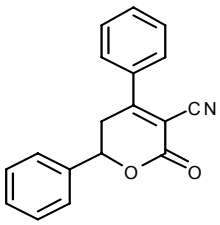


2-amino-4,6-diphenyl-4H-pyran-3-carbonitrile (4a)



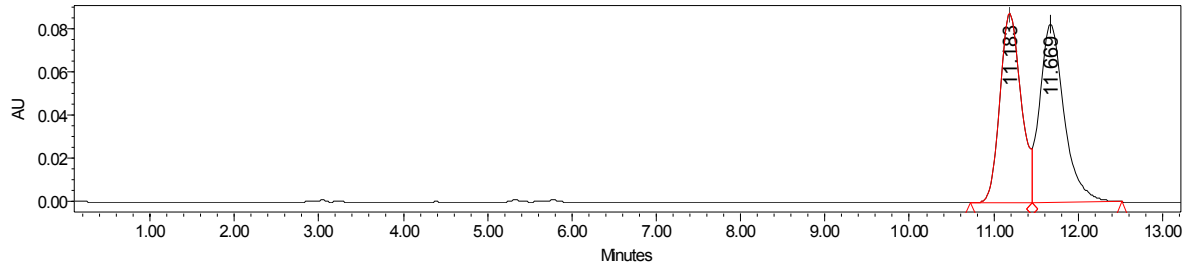
white solid; 51% yield; IR 3478.18, 3377.45 (-NH_2), 3226.99 (C=C), 2215.84 (-CN), 1635.64 (-O-*C*=*C*H); HRMS (ESI-TOF) calcd for $C_{18}H_{14}N_2O$ ([M-H^+]) = 273.1033, Found 273.1028.

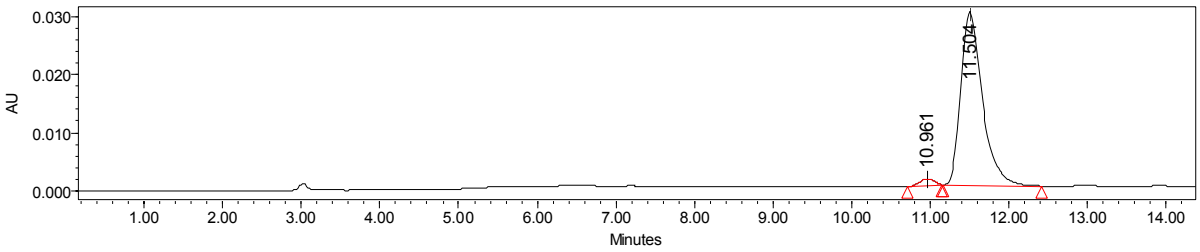
2-oxo-4,6-diphenyl-3,4-dihydro-2H-pyran-3-carbonitrile (5e)



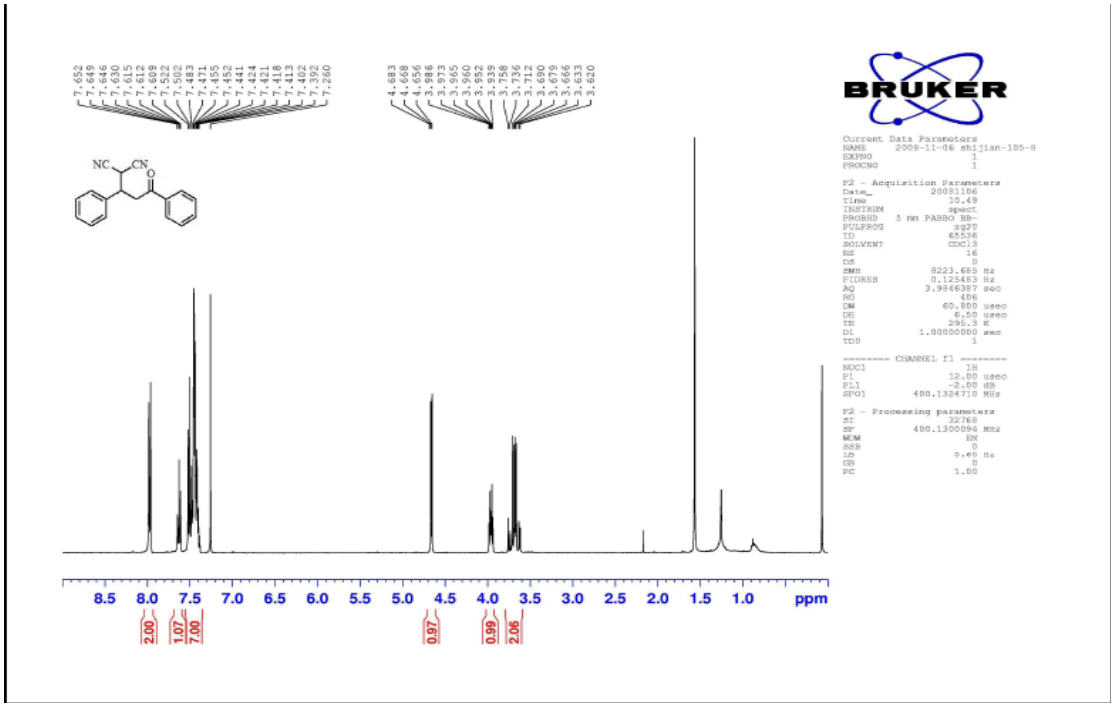
2-amino-4,6-diphenyl-4(H)-pyran-3-carbonitrile (0.274 mg, 1 mmol) was added in 99% ethanol (5 mL), then triethylamine (0.5 mL) was added. The resulting solution was stirred at room temperature for 48 h. Following, the solvent was removed. The reaction mixture was directly purified by column chromatography on silica gel eluted (CH_2Cl_2: petroleum ether = 1:3) to afford the corresponding compound.

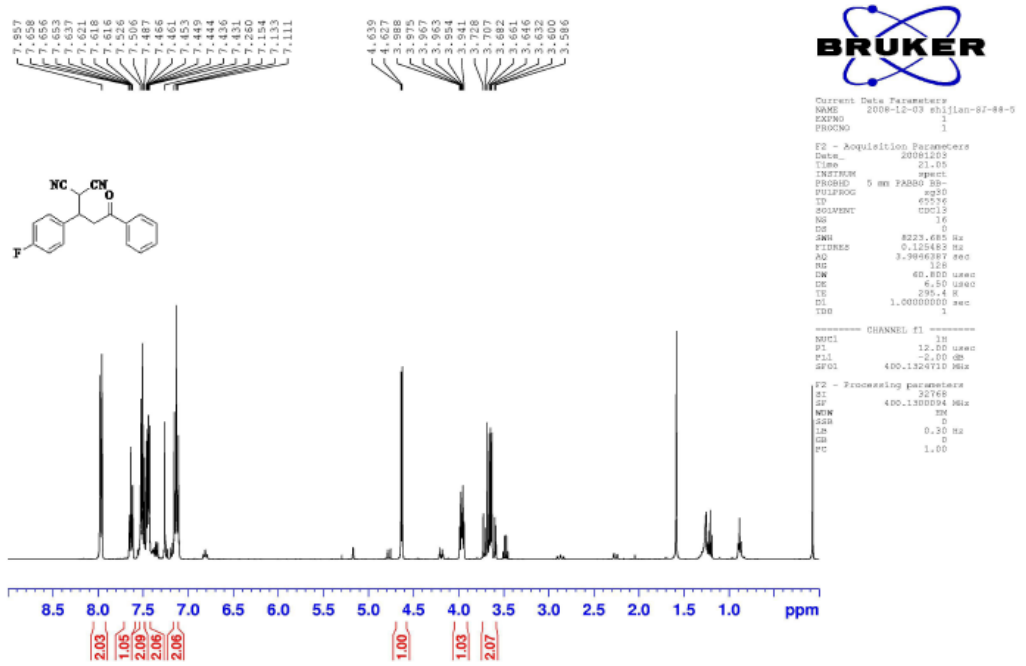
white solid; 22% yield; HPLC DAICEL CHIRALCEL IA, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 11.504 min (major) and 10.961 min (minor); IR 3226.99 (C=C), 2215.84 (-CN), 1730.19 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.62 (dd, 1H, J_1= 10.0 Hz, J_2= 25.2 Hz, Ar-CH), 3.946 (dd, 2H, J_1= 10.4 Hz, J_2= 24.8 Hz, C=C-CH_2), 7.32-7.87 (m, 10H, Ar-H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 42.3, 44.7, 48.0, 115.2, 128.0, 128.7, 128.8, 129.0, 129.3, 133.6, 135.8, 136.1, 194.9 ppm; HRMS (ESI-TOF) calcd for $C_{18}H_{13}N_2O$ ([M-H^+]) = 274.0873, Found 274.0865.





(H) Copies of NMR spectra for all compounds

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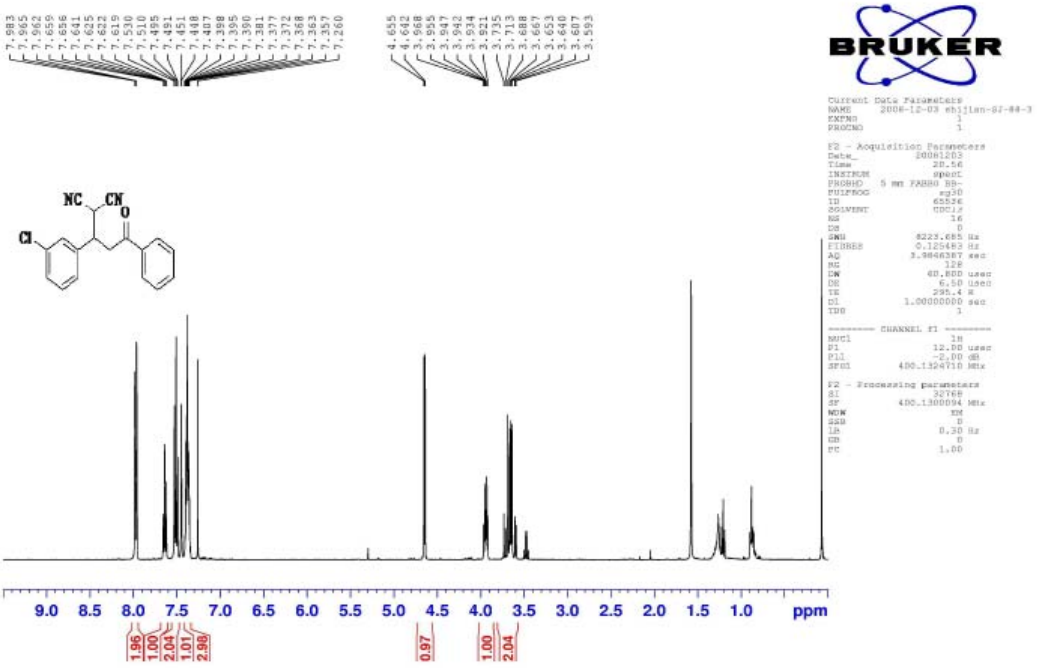
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TE 295.4 K
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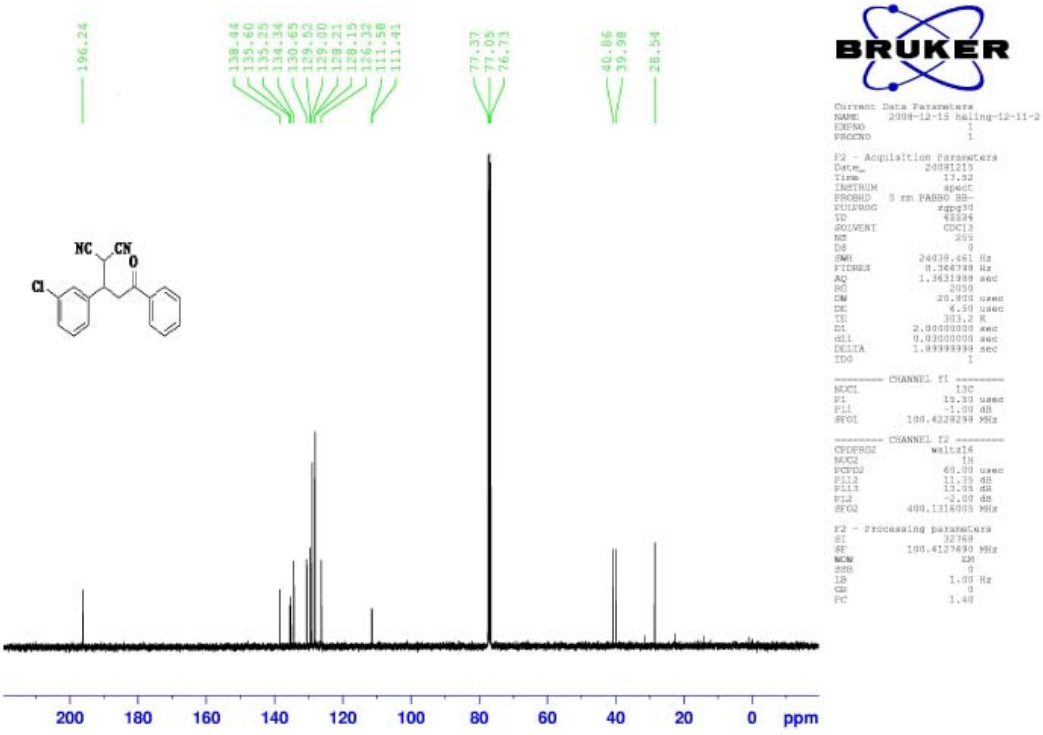
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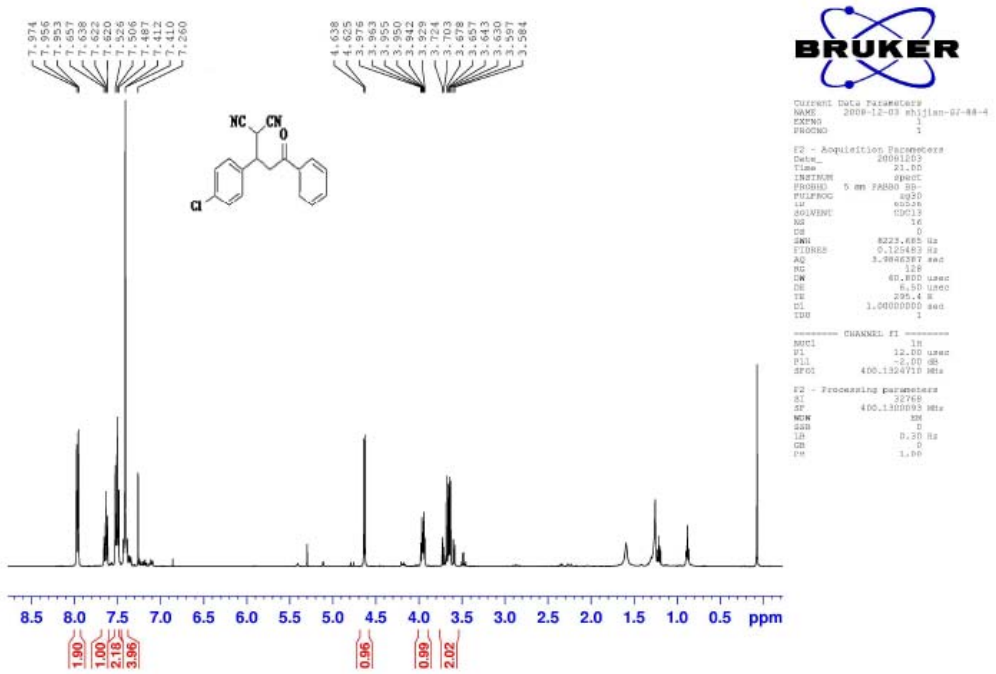
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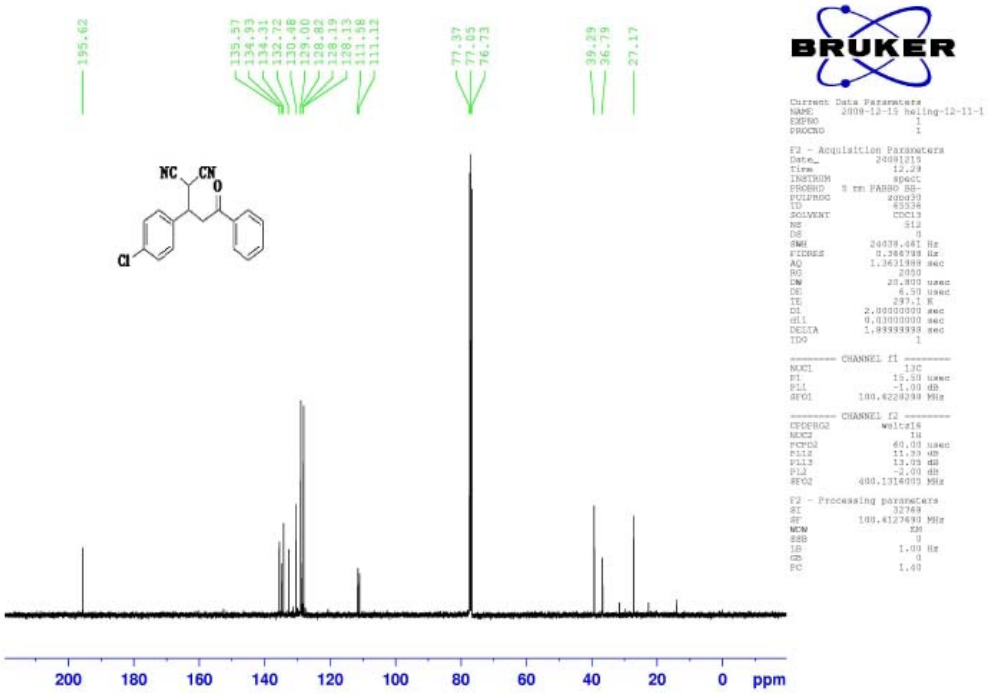
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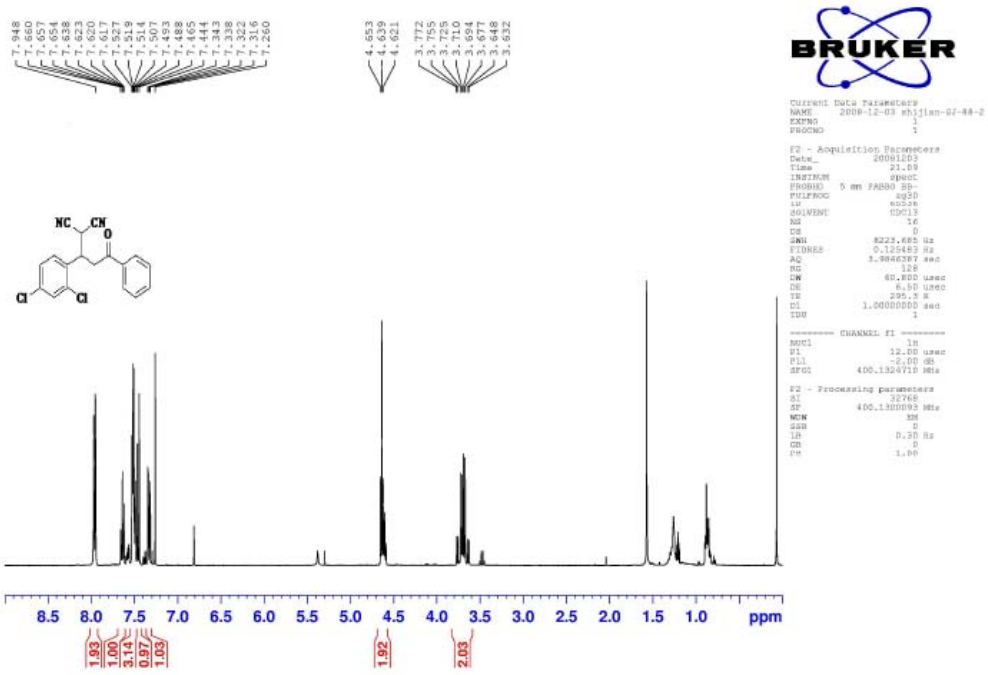
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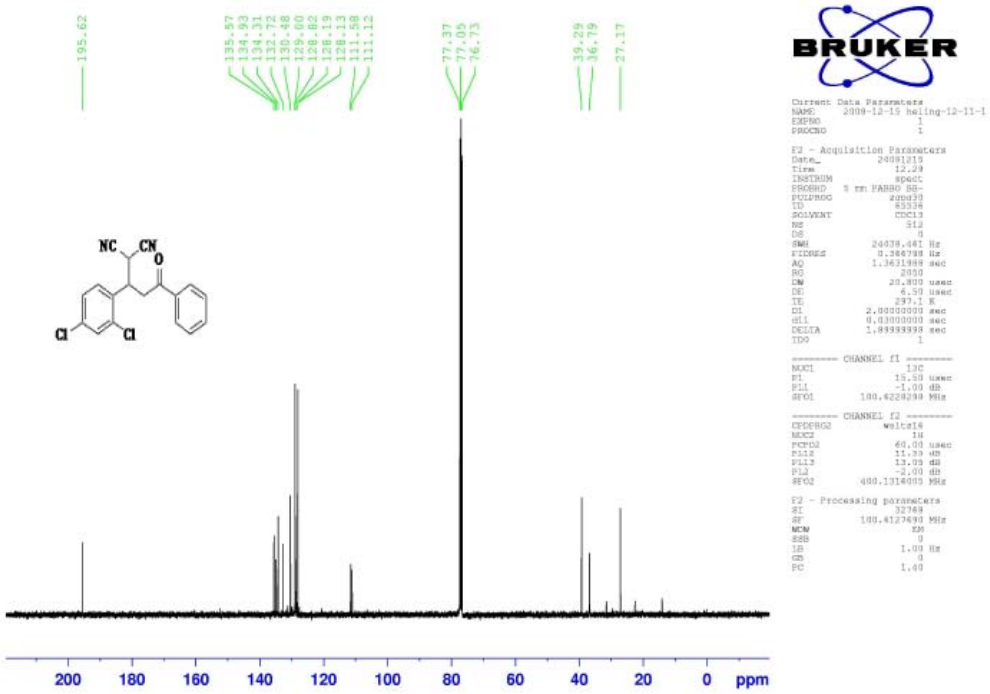
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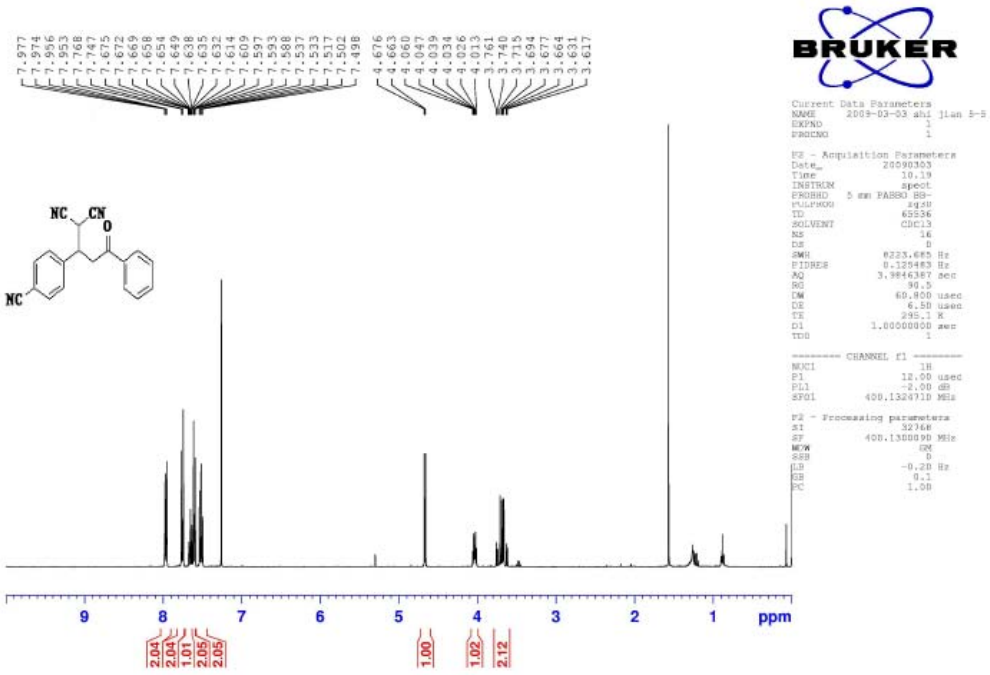
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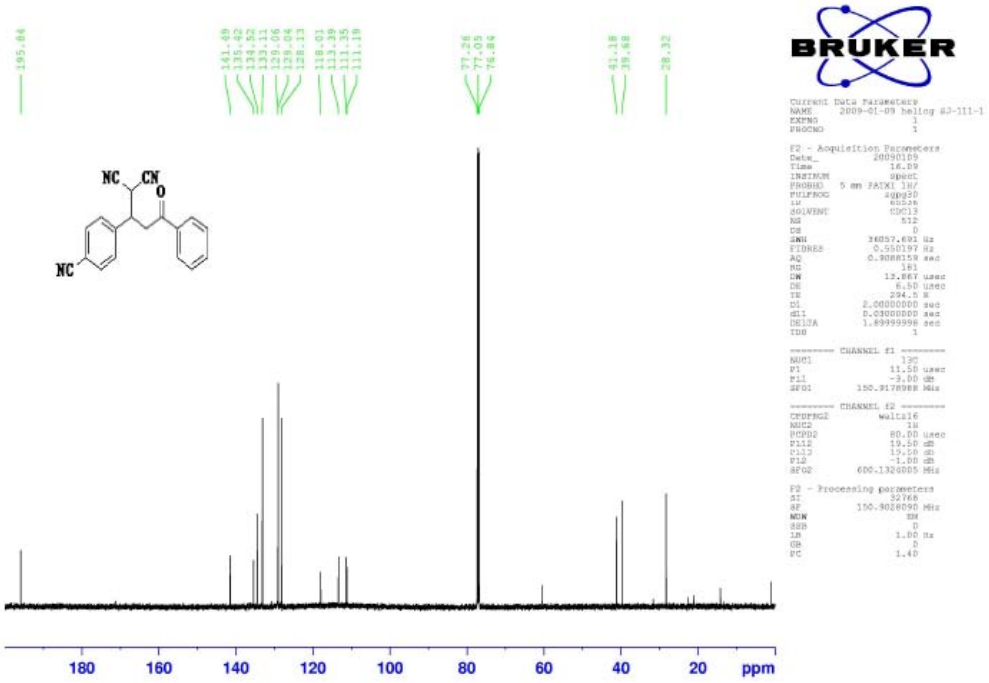
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BRUKER
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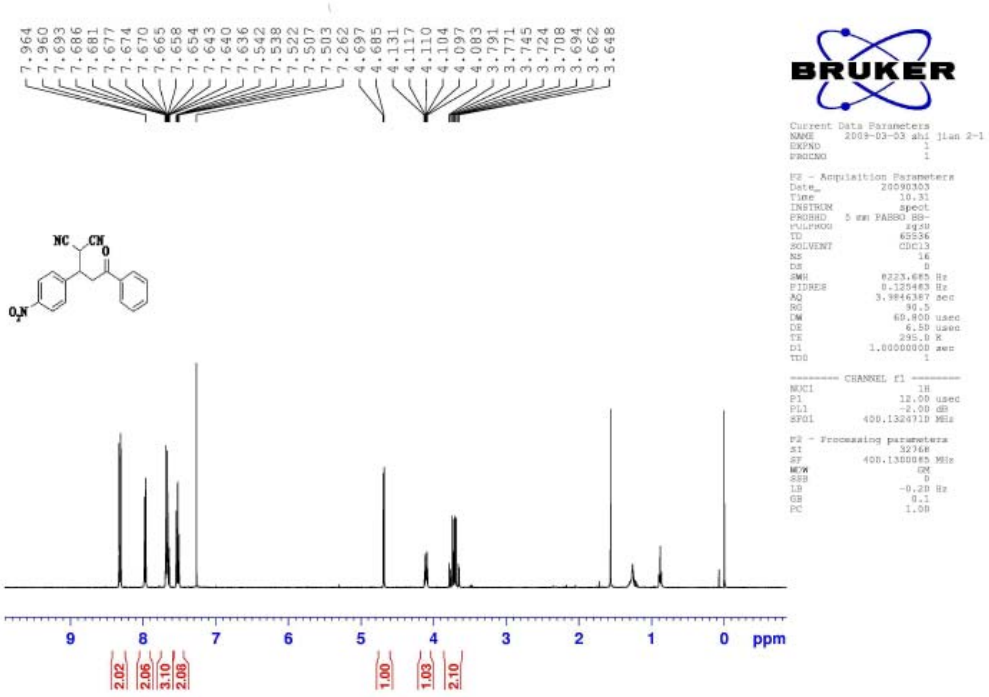
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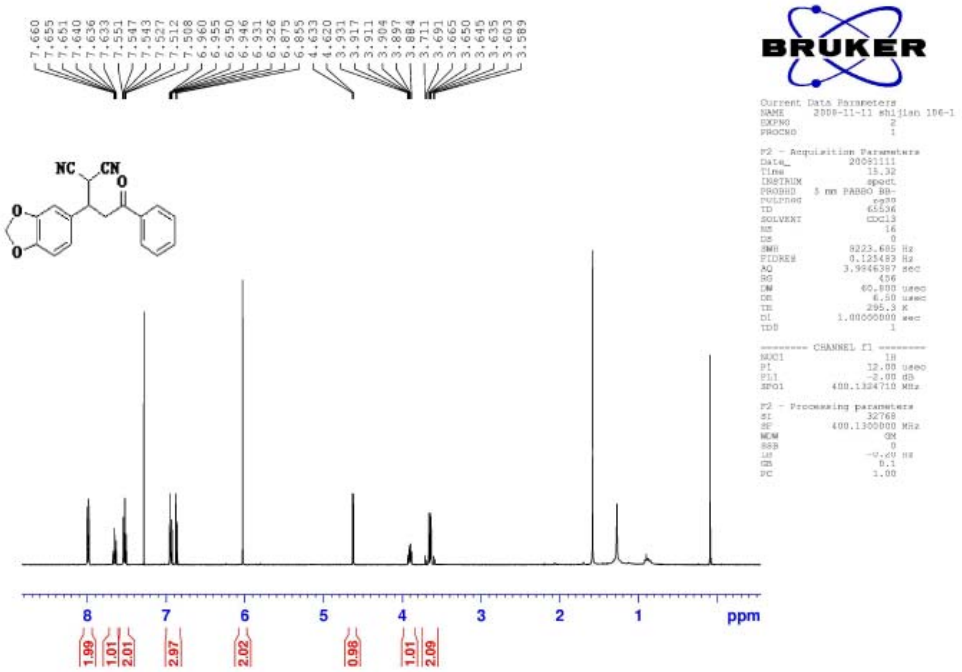
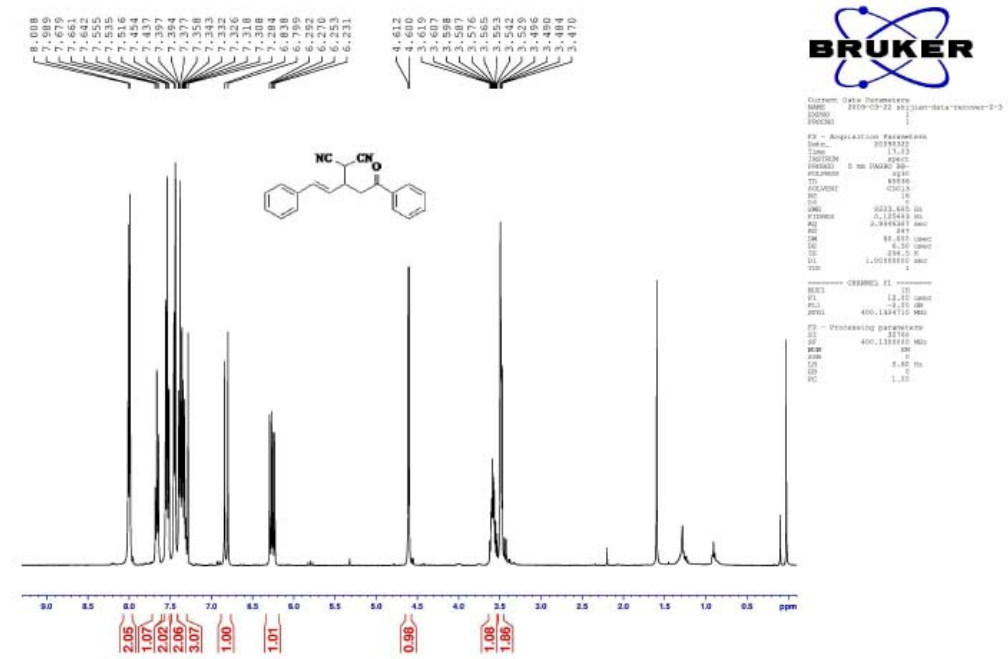
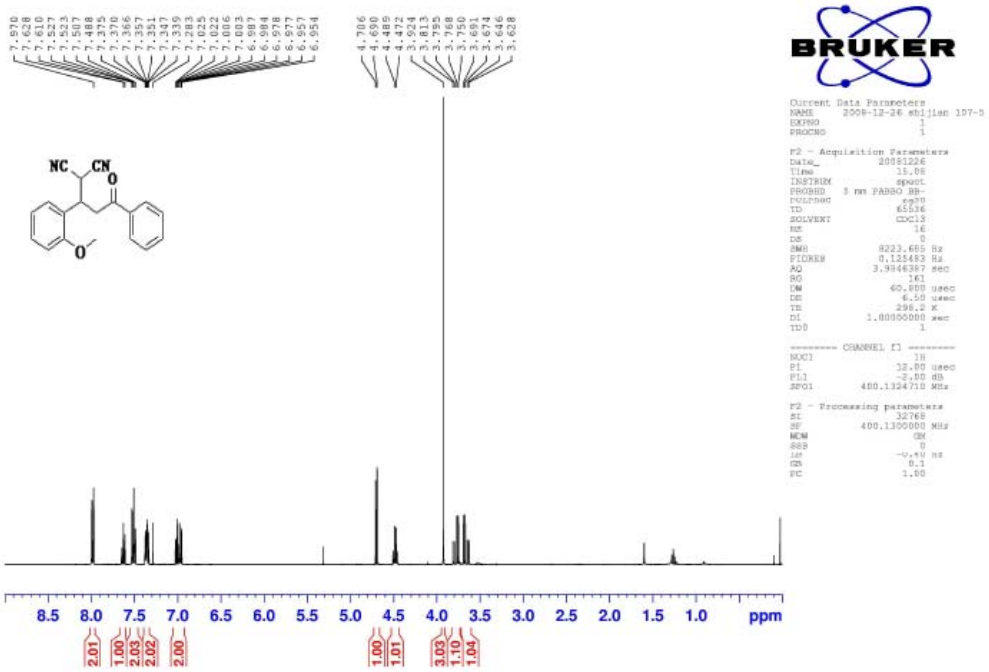
7.538
7.522
7.507
7.503
7.262
4.697
4.685
4.131
4.117
4.110
4.104
4.097
4.083
3.791
3.771
3.745
3.724
3.708
3.694
3.662
3.648
NC
CN
O
O2N
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.06
3.10
2.08
1.00
1.03
2.10

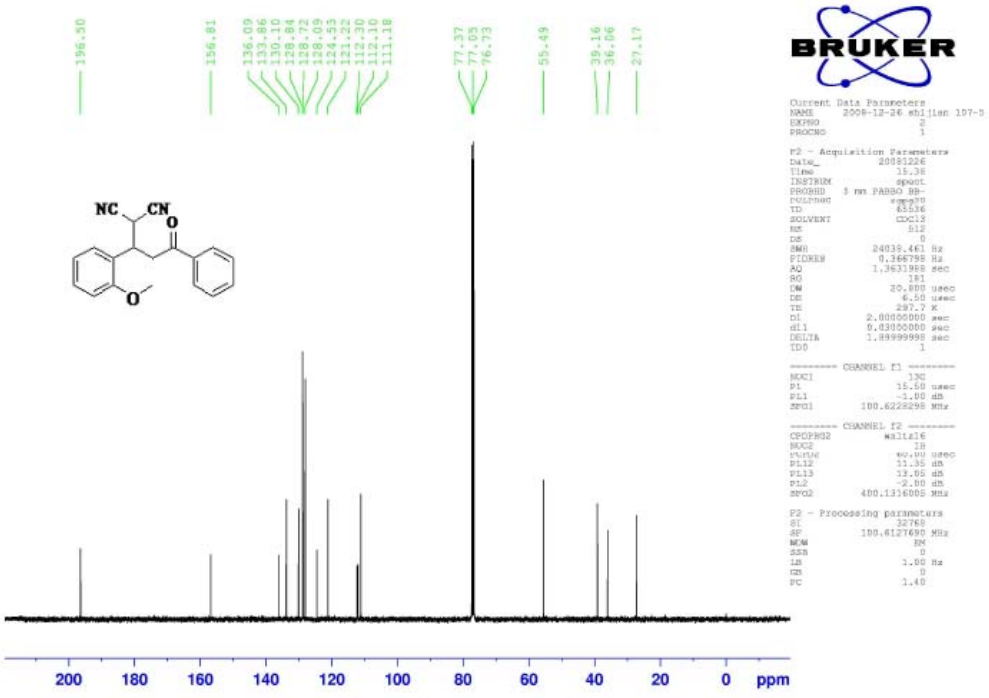
BRUKER
Current Data Parameters
NAME 2009-03-03 ahi jian 2-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090303
Time 10.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846367 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300085 MHz
WDW EM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00


7.660
7.655
7.651
7.640
7.636
7.633
7.551
7.547
7.543
7.527
7.512
7.508
6.960
6.955
6.950
6.946
6.931
6.926
6.875
6.855
4.633
4.620
3.931
3.917
3.911
3.904
3.897
3.884
3.711
3.691
3.665
3.650
3.645
3.635
3.603
3.589
NC
CN
O
O
O
BRUKER
Current Data Parameters
NAME 2008-11-11 shijian 106-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081111
Time 15.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 456
DW 60.800 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB -0.60 Hz
GB 0.1
PC 1.00
8
7
6
5
4
3
2
1
ppm
1.99
1.01
2.01
2.97
2.02
0.96
1.01
2.09

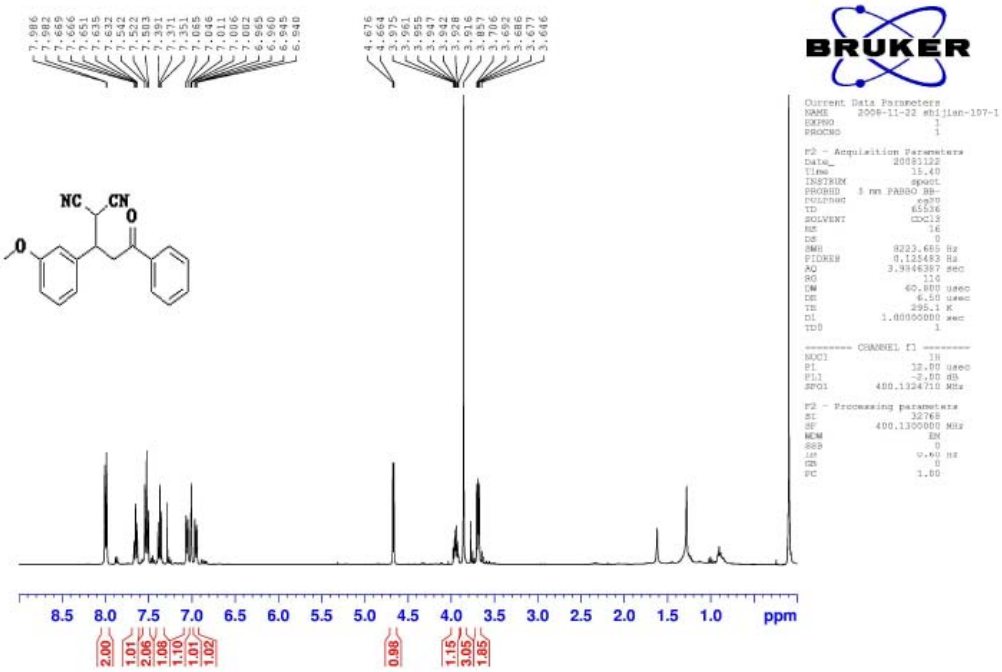

8.008
7.989
7.679
7.661
7.642
7.555
7.535
7.516
7.454
7.437
7.397
7.394
7.377
7.358
7.343
7.332
7.326
7.318
7.284
6.838
6.799
6.292
6.270
6.253
6.231
4.612
4.600
3.619
3.607
3.598
3.587
3.576
3.565
3.553
3.542
3.529
3.496
3.490
3.484
3.470
NC
CN
O
BRUKER
Current Data Parameters
NAME 2009-03-22 shijian-data-recover-2-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090322
Time 17.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
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
2.05
1.07
2.02
2.06
3.07
1.00
1.01
0.98
1.08
1.86

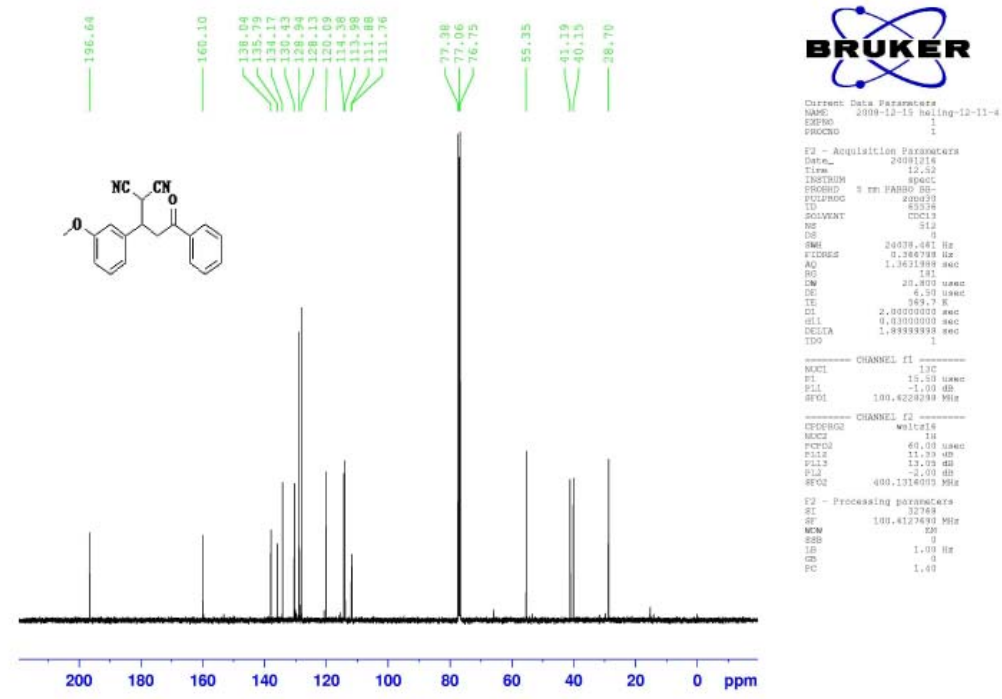

7.970
7.628
7.610
7.527
7.523
7.507
7.488
7.375
7.370
7.366
7.357
7.351
7.347
7.339
7.283
7.025
7.022
7.006
7.003
6.997
6.984
6.978
6.977
6.957
6.954
4.706
4.690
4.489
4.472
3.924
3.813
3.795
3.768
3.760
3.691
3.674
3.646
3.628
NC
CN
O
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.01
1.00
2.03
2.02
2.00
1.00
1.01
3.03
1.10
1.04

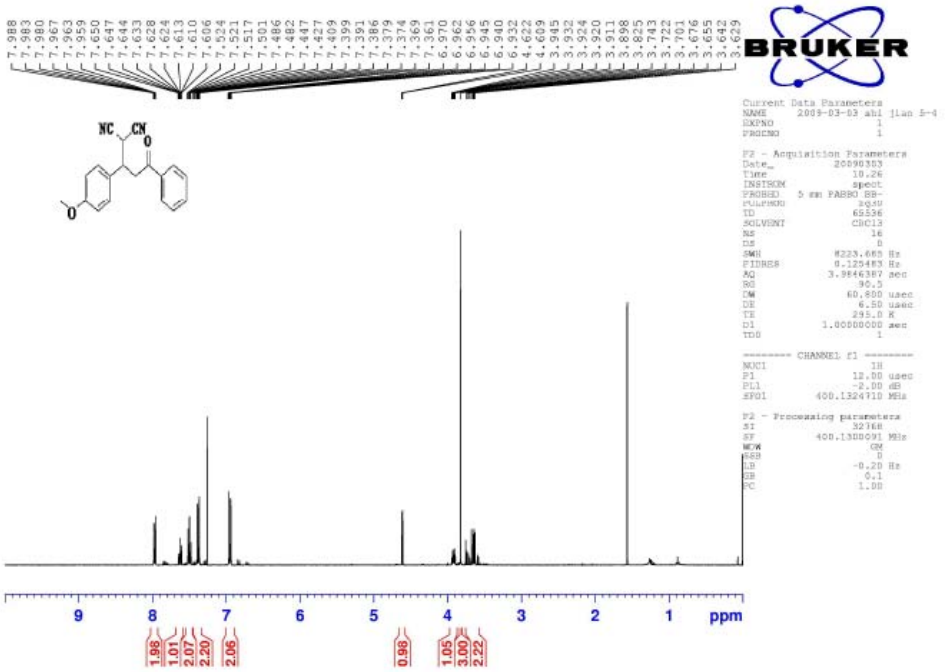
BRUKER
Current Data Parameters
NAME 2008-12-26 shijian 107-5
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081226
Time 15.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.00

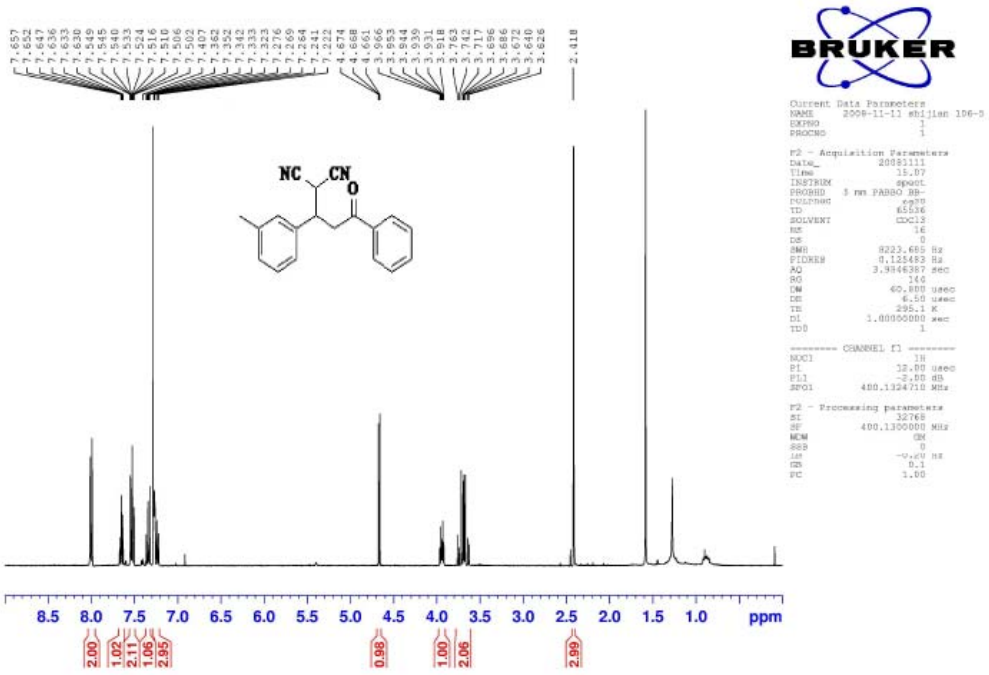

196.50
156.81
136.09
133.86
130.10
128.84
128.72
128.09
124.53
121.22
112.30
112.10
111.18
77.37
77.05
76.73
55.49
39.16
36.06
27.17
NC
CN
O
O
200 180 160 140 120 100 80 60 40 20 0 ppm

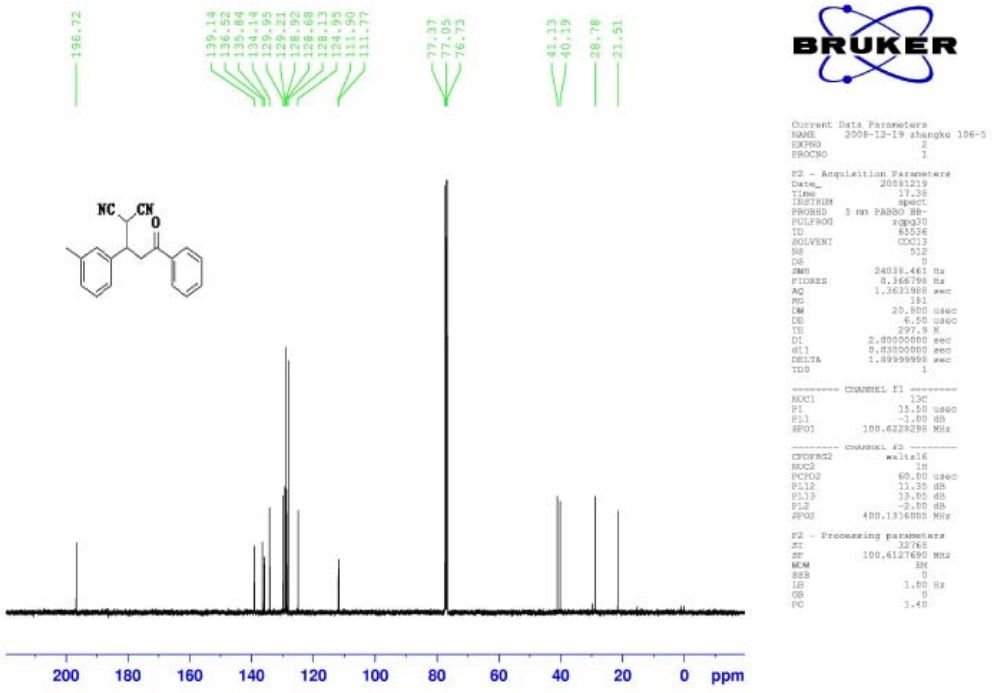
BRUKER
Current Data Parameters
NAME 2008-12-26 shijian 107-5
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081226
Time 15.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
d11 0.03050000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

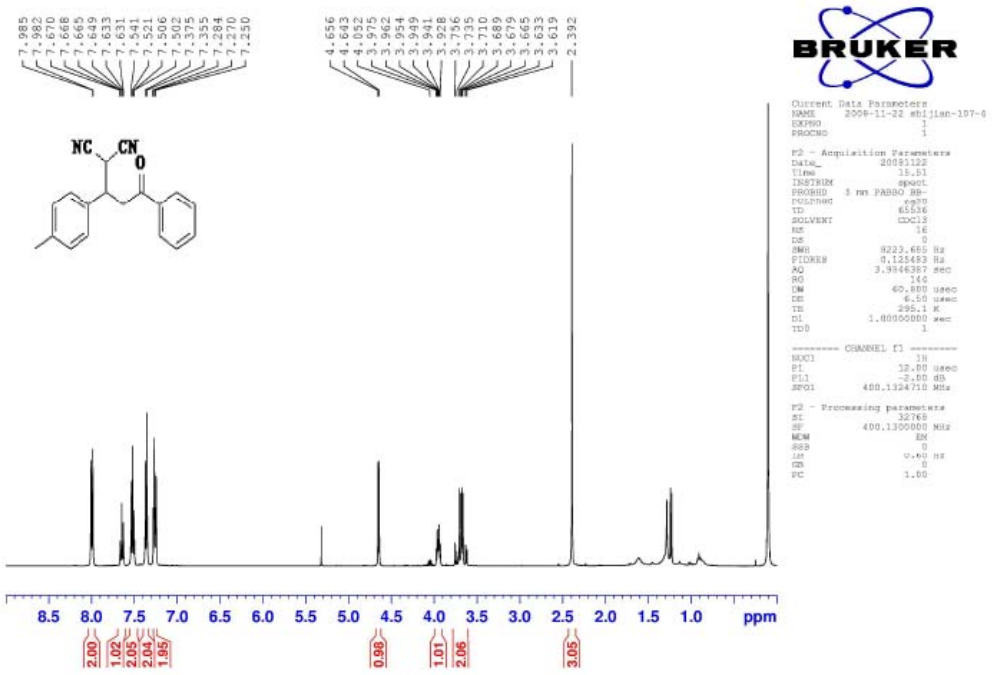

7.986
7.982
7.669
7.666
7.651
7.635
7.632
7.542
7.523
7.503
7.391
7.371
7.351
7.085
7.046
7.011
7.006
7.002
6.965
6.960
6.945
6.940
4.676
4.664
3.975
3.961
3.955
3.947
3.942
3.928
3.916
3.857
3.706
3.692
3.686
3.677
3.646
NC
CN
O
O
BRUKER
Current Data Parameters
NAME 2008-11-22 ebijlan-107-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081122
Time 15.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
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 ppm
2.00 1.01 2.06 1.08 1.10 1.01 1.02 0.98 1.15 3.05 1.85

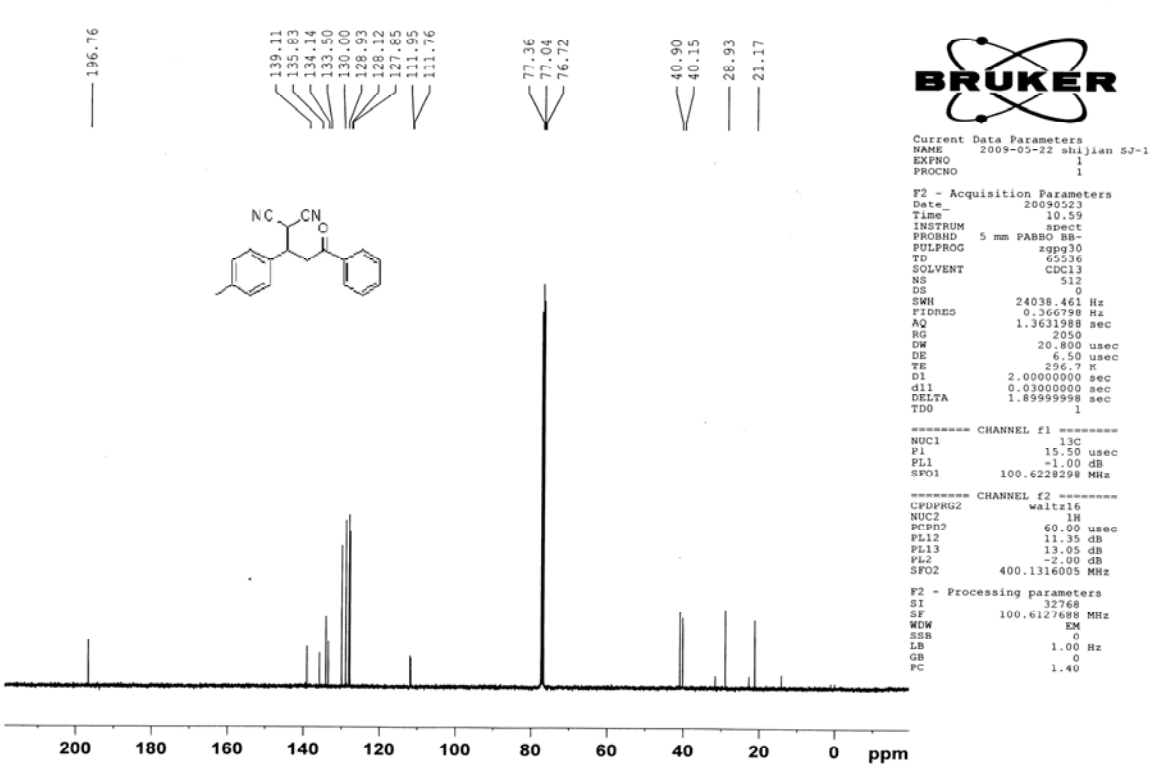

196.64
160.10
138.04
135.79
134.17
130.43
128.94
128.13
120.09
114.38
113.98
111.88
111.76
77.38
77.06
76.75
56.35
41.19
40.13
28.70
NC
CN
O
O
BRUKER
Current Data Parameters
NAME 2008-12-15 heling-12-11-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081216
Time 12.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631989 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 369.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.39 dB
PL13 13.03 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm


BRUKER
NC
CN
O
O
9
8
7
6
5
4
3
2
1
ppm
1.98
1.01
2.07
2.20
2.06
0.98
1.05
3.00
2.22


BRUKER
NC
CN
O
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
ppm
2.00
1.02
2.11
1.06
2.95
0.98
1.00
2.06
2.89


196.72
139.14
136.52
135.84
134.14
129.95
129.21
128.92
128.68
128.13
124.95
111.90
111.77
77.37
77.05
76.73
41.13
40.19
28.78
21.51
NC
CN
O
200
180
160
140
120
100
80
60
40
20
0
ppm


7.985
7.982
7.670
7.668
7.665
7.649
7.633
7.631
7.541
7.521
7.506
7.502
7.375
7.355
7.284
7.270
7.250
4.656
4.643
4.052
4.035
3.962
3.954
3.949
3.941
3.828
3.756
3.735
3.710
3.689
3.679
3.665
3.653
3.619
2.392
NC
CN
O
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
ppm
2.00
1.02
2.05
2.04
1.95
0.96
1.01
2.06
3.05



BRUKER

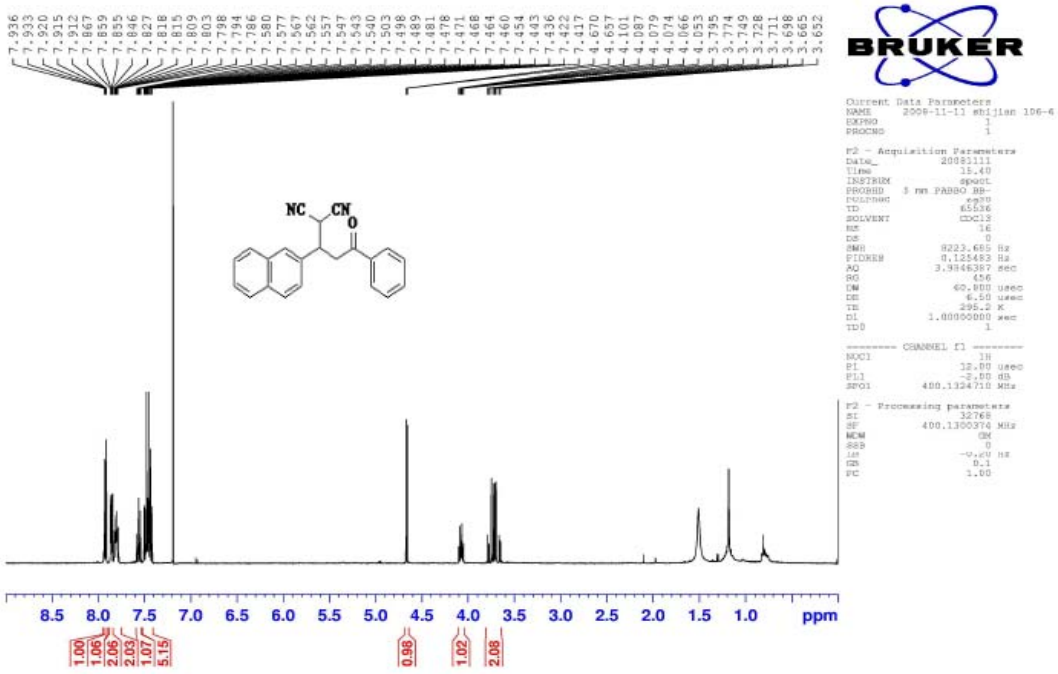
Current Data Parameters
NAME 2009-05-22 shijian SJ-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090523
Time 10.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127688 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



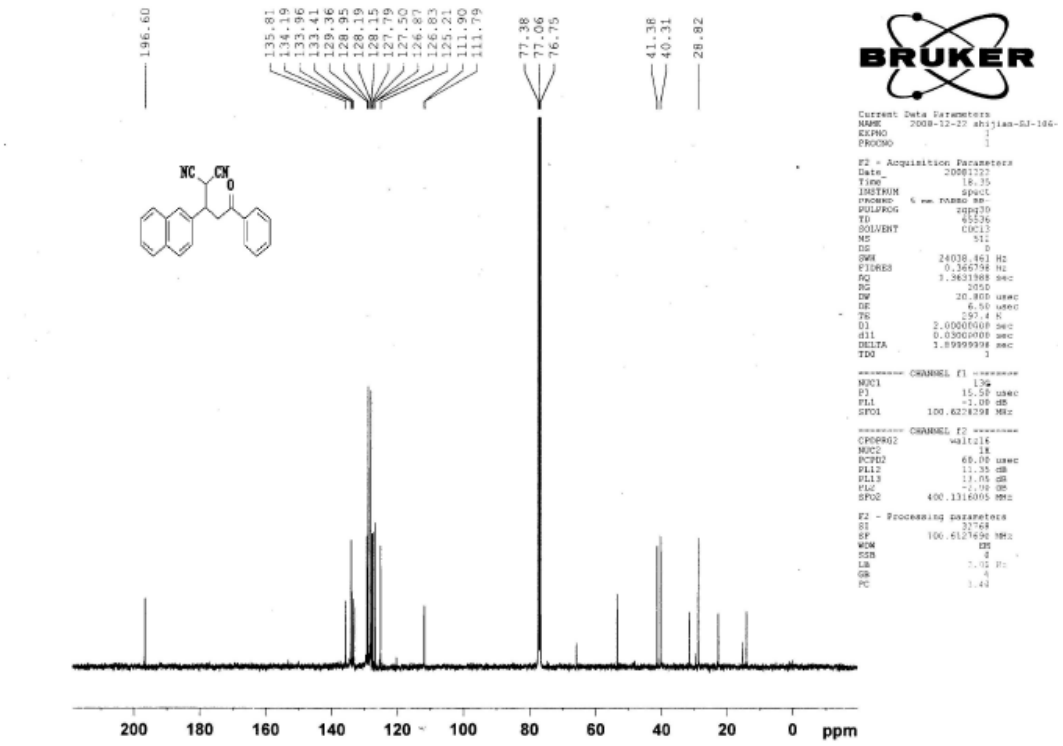
BRUKER

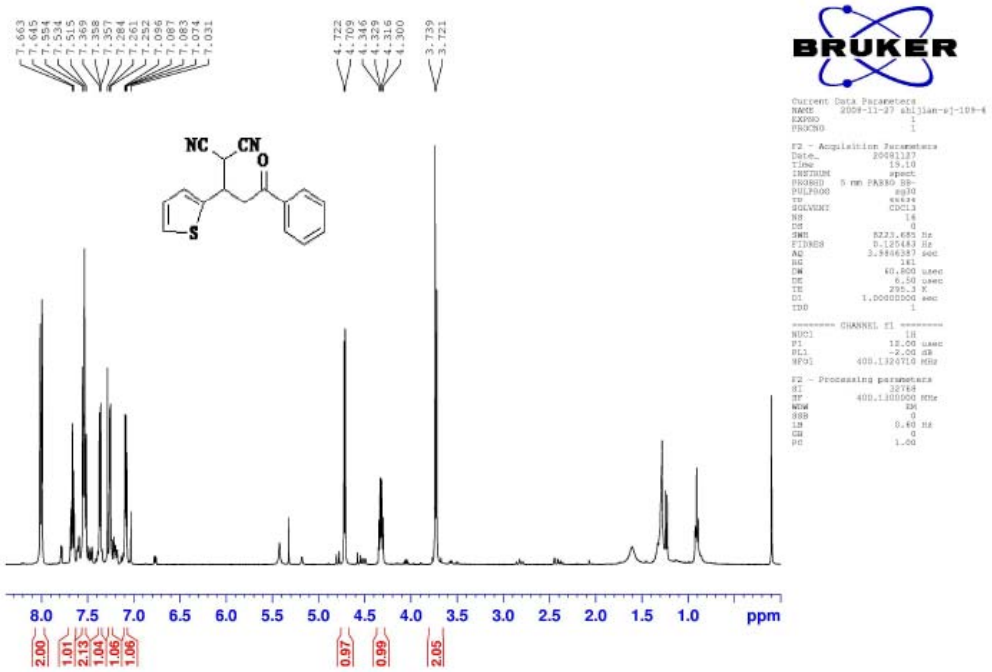
Current Data Parameters
NAME 2009-11-11 shijian 106-6
EXPNO 1
PROCNO 1

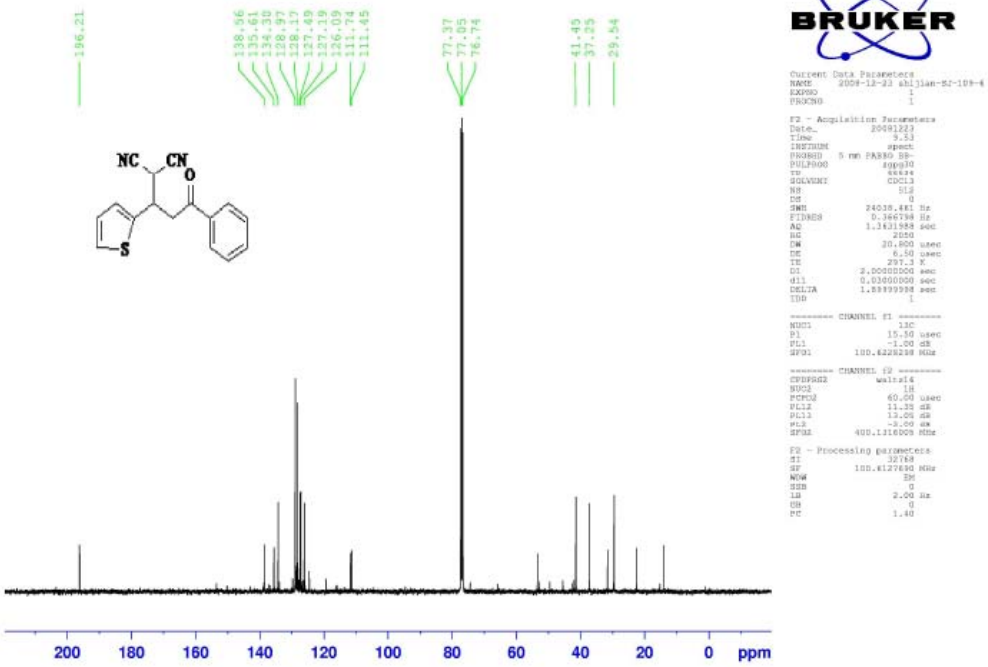
F2 - Acquisition Parameters
Date_ 20091111
Time 15.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 456
DW 60.800 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

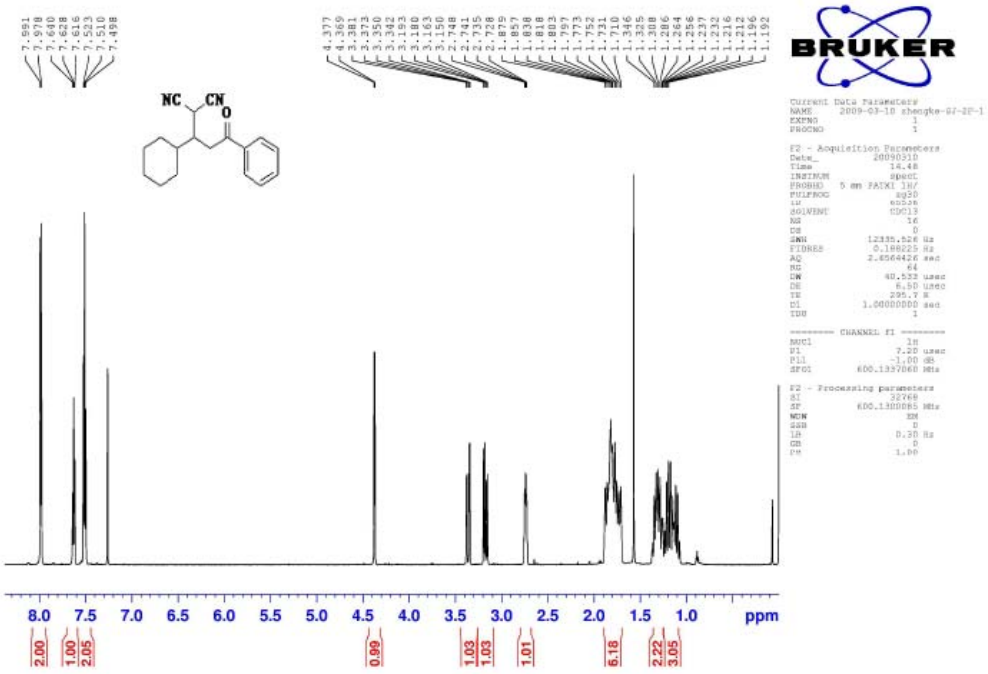
F2 - Processing parameters
SI 32768
SF 400.1300074 MHz
WDW EM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00


196.60
135.81
134.19
133.96
133.41
129.36
128.95
128.19
128.15
127.79
127.50
126.87
126.83
125.21
111.90
111.79
77.38
77.06
76.75
41.38
40.31
28.82
NC
CN
O
200
180
160
140
120
100
80
60
40
20
0
ppm

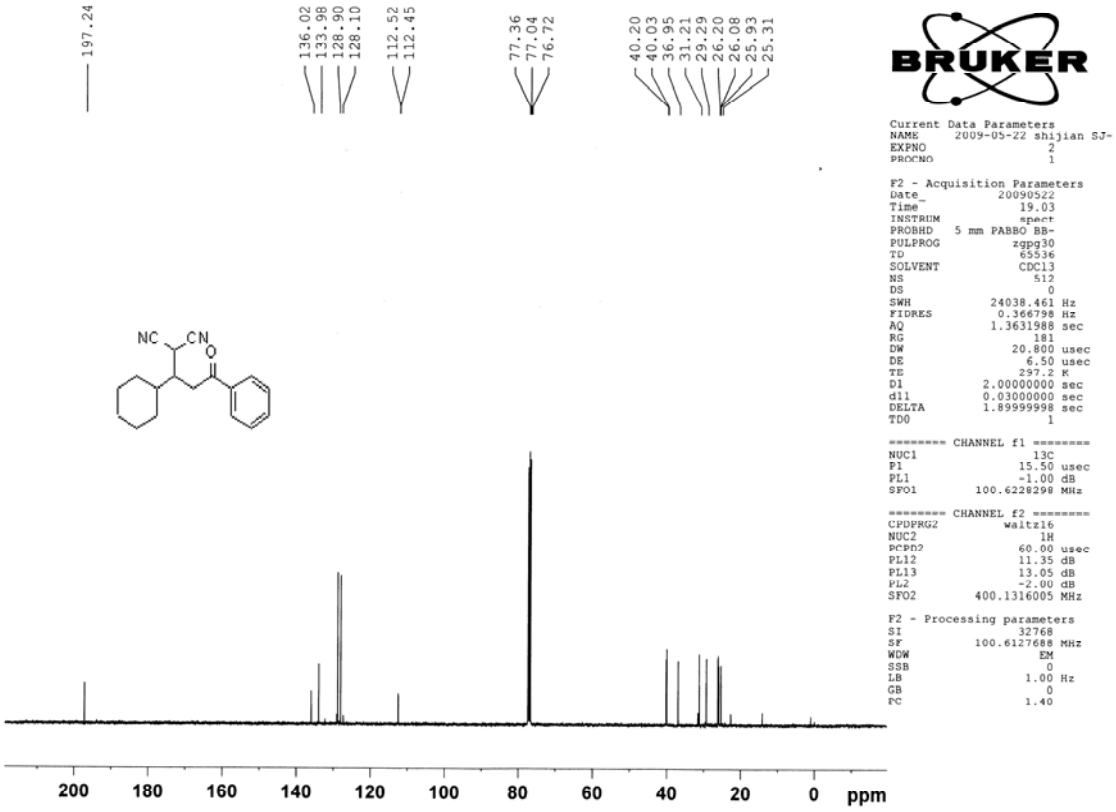

7.663
7.645
7.554
7.534
7.515
7.369
7.358
7.357
7.284
7.261
7.252
7.096
7.087
7.083
7.074
7.031
4.722
4.709
4.346
4.329
4.316
4.300
3.739
3.721
NC
CN
O
S
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.00
1.01
2.13
1.04
1.06
1.06
0.97
0.99
2.05

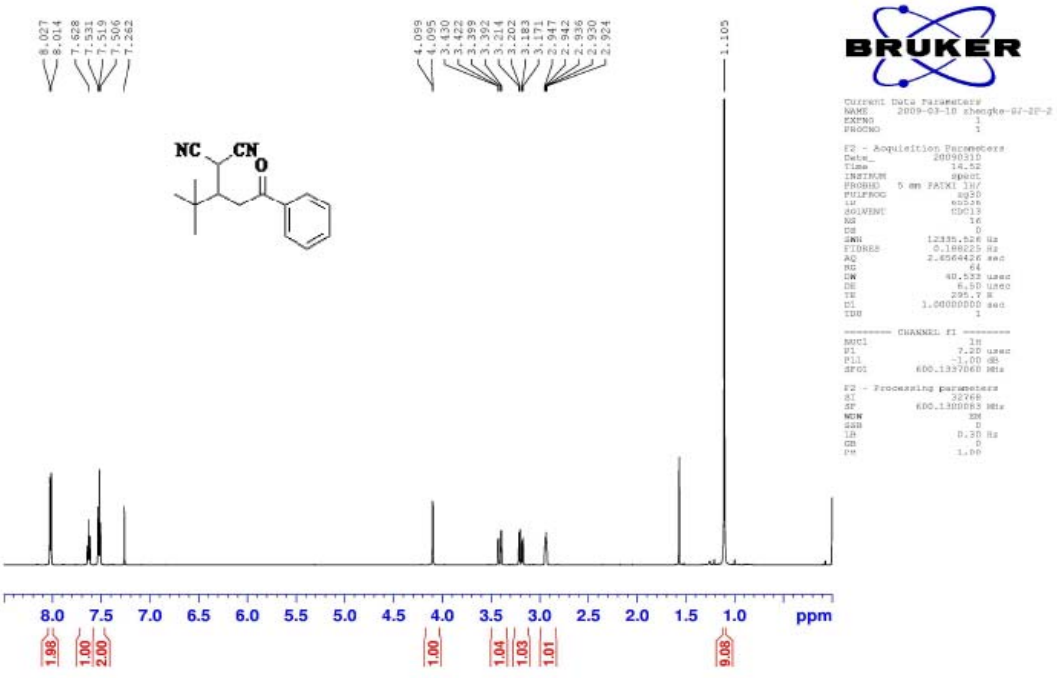

196.21
138.56
135.61
134.30
128.97
128.77
127.49
127.19
126.09
111.74
111.45
77.37
77.05
76.74
41.45
37.25
29.54
NC
CN
O
S
200
180
160
140
120
100
80
60
40
20
0
ppm

BRUKER


7.691
7.678
7.640
7.628
7.616
7.523
7.510
7.498
4.377
4.369
3.381
3.373
3.350
3.342
3.193
3.180
3.163
3.150
2.748
2.741
2.735
2.728
2.079
1.857
1.838
1.818
1.803
1.797
1.773
1.752
1.731
1.710
1.346
1.325
1.308
1.286
1.264
1.256
1.237
1.232
1.216
1.212
1.196
1.192
NC
CN
O
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
ppm
2.00
1.00
2.05
0.99
1.03
1.03
1.01
6.18
2.22
3.05

BRUKER


197.24
136.02
133.98
128.90
128.10
112.52
112.45
77.36
77.04
76.72
40.20
40.03
36.95
31.21
29.29
26.20
26.08
25.93
25.31
NC
CN
O
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME 2009-05-22 shijian SJ-
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090522
Time 19.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127688 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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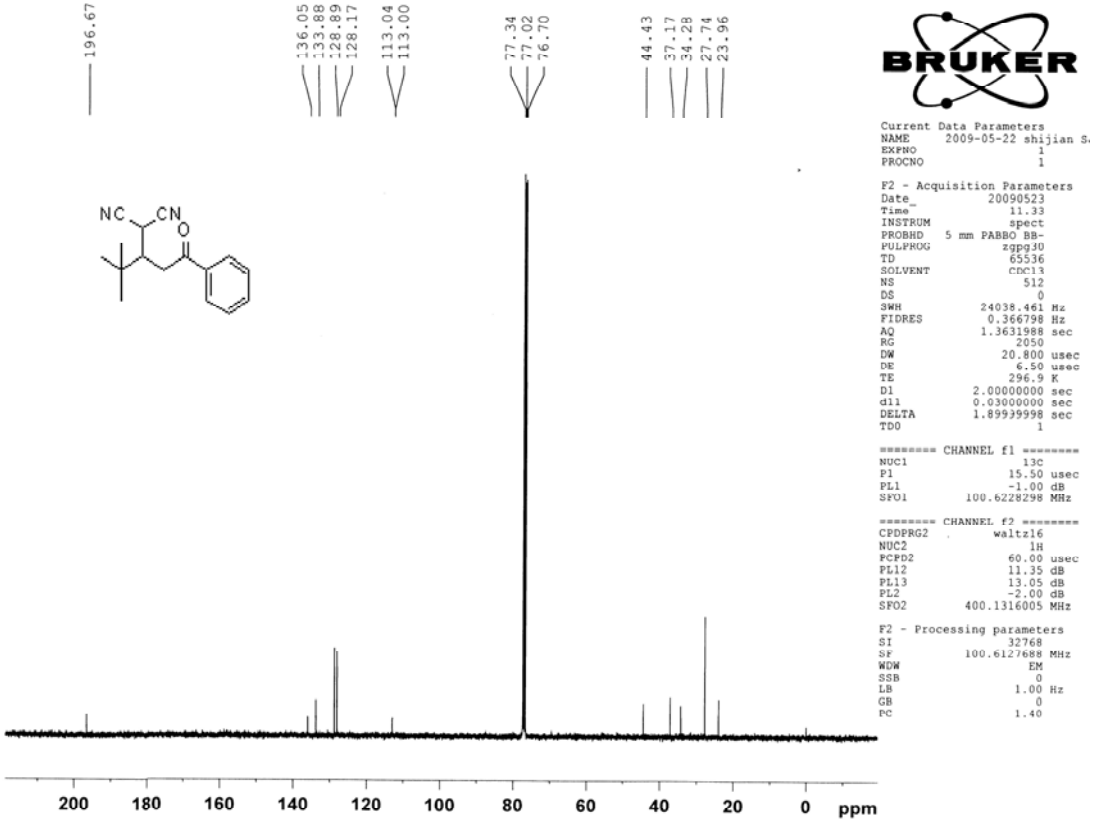
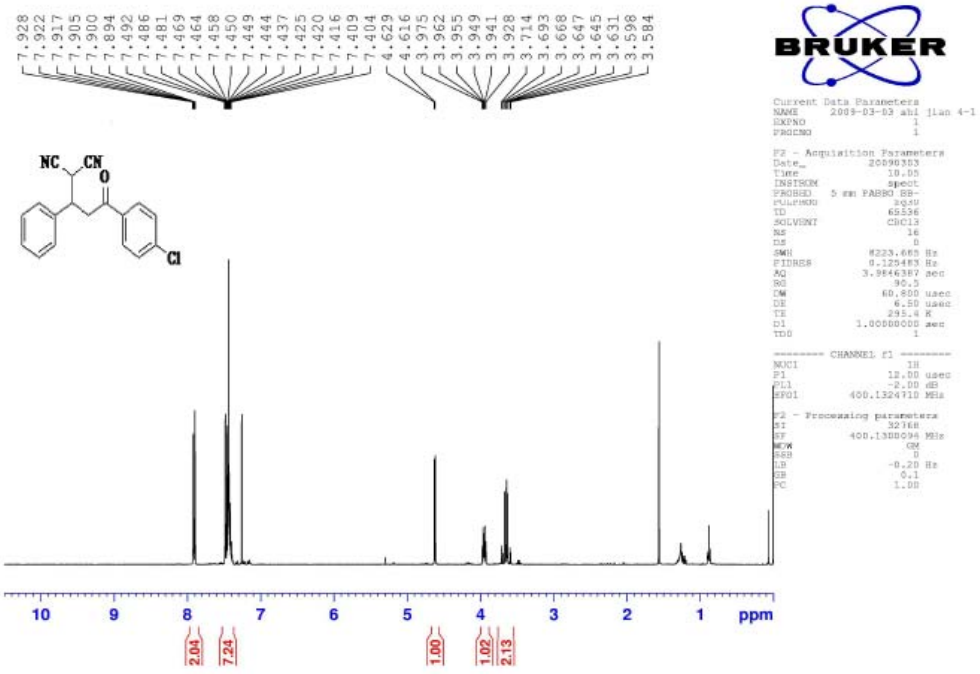
8.014
7.628
7.531
7.519
7.506
7.262
4.099
4.095
3.430
3.422
3.399
3.392
3.214
3.202
3.183
3.171
2.947
2.942
2.936
2.930
2.924
1.105
NC
CN
O
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.98
1.00
2.00
1.00
1.04
1.03
1.01
9.08
BRUKER
Current Data Parameters
NAME 2009-03-18 zhengke-07-2F-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090318
Time 14.52
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 64
DW 40.533 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.20 usec
PL1 -1.00 dB
SFO1 600.1397060 MHz
F2 - Processing parameters
SI 32768
SF 600.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



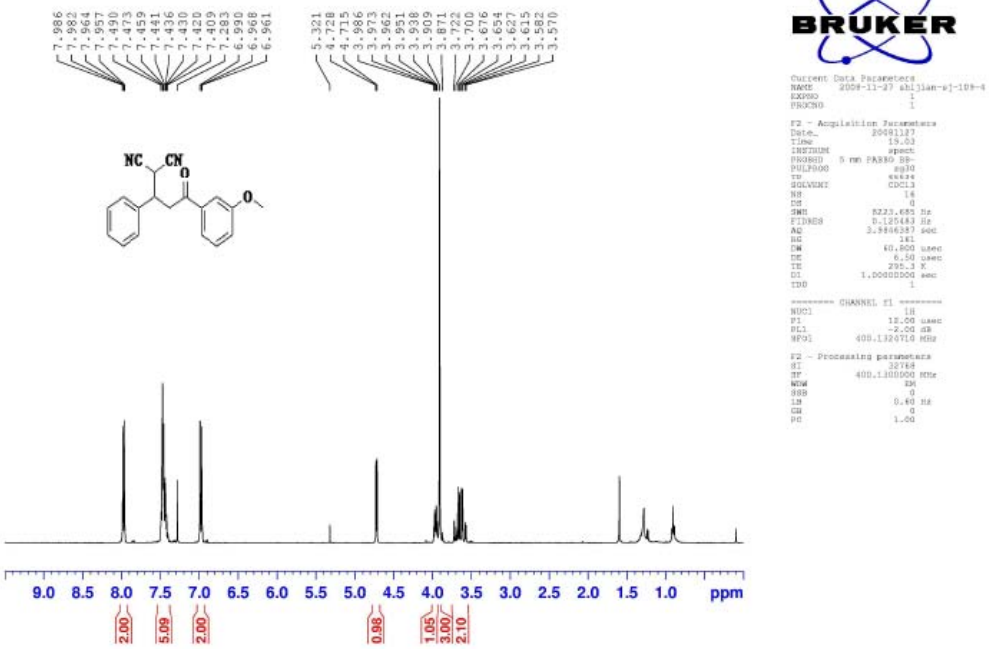
BRUKER

```
Current Data Parameters
NAME      2009-05-22 shijian S.
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20090523
Time                 11.33
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       0
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                    2050
DW                 20.800 usec
DE                  6.50 usec
TE                 296.9 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   15.50 usec
PL1                  -1.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2                60.00 usec
PL12                 11.35 dB
PL13                 13.05 dB
PL2                  -2.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF            100.6127688 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

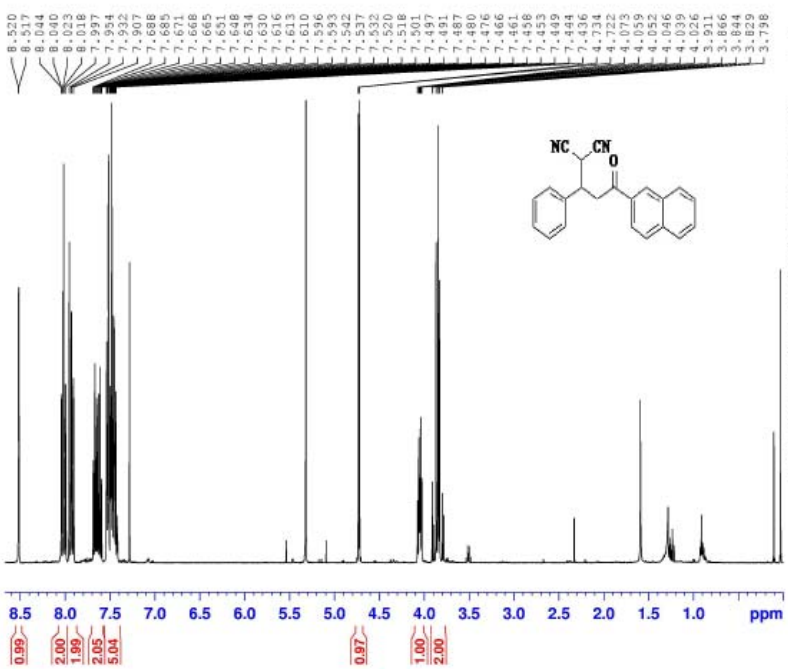


BRUKER

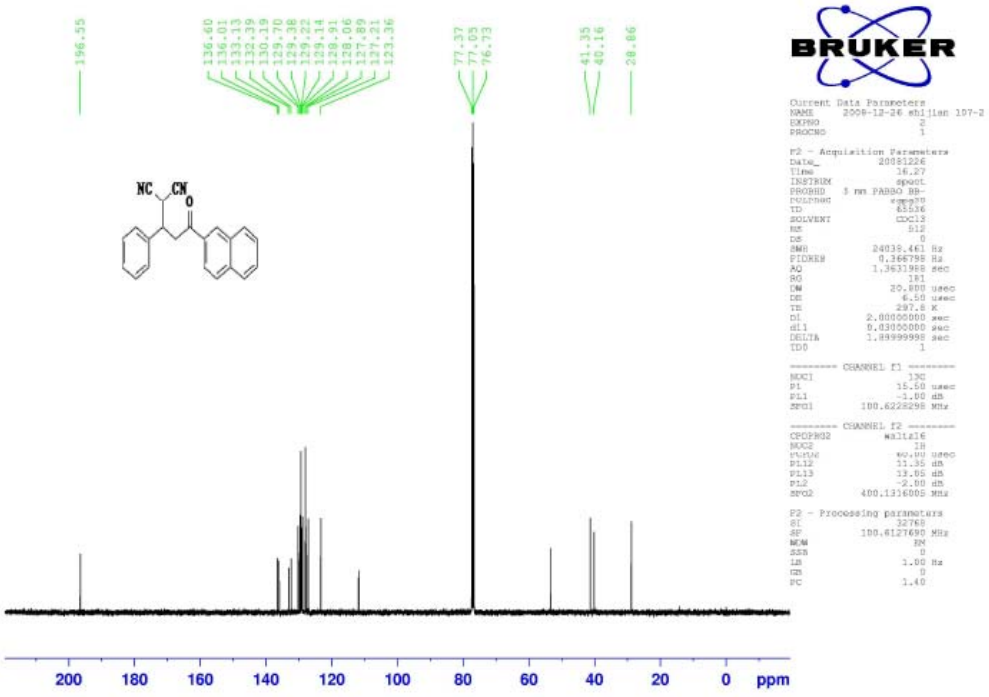
```
Current Data Parameters
NAME      2009-03-03 abl jian 4-1
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20090303
Time                 10.05
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       0
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    90.5
DW                 60.800 usec
DE                  6.50 usec
TE                 295.4 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                   12.00 usec
PL1                  -2.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                    32768
SF            400.1300094 MHz
WDW                      EM
SSB                       0
LB                    -0.20 Hz
GB                        0.1
PC                     1.00
```


7.986
7.982
7.964
7.957
7.490
7.473
7.459
7.441
7.436
7.430
7.420
7.409
7.283
6.990
6.968
6.961
5.321
4.728
4.715
3.986
3.973
3.962
3.951
3.938
3.909
3.871
3.722
3.700
3.676
3.654
3.627
3.615
3.582
3.570
NC
CN
O
O
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 ppm
2.00
5.09
2.00
0.98
1.05
3.00
2.10

BRUKER
Current Data Parameters
NAME 2009-11-27 shilan-wj-109-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091127
Time 19.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.40 Hz
GB 0
PC 1.00


8.520
8.517
8.044
8.040
8.023
8.018
7.997
7.954
7.932
7.907
7.688
7.685
7.671
7.668
7.665
7.651
7.648
7.634
7.630
7.616
7.613
7.610
7.596
7.593
7.542
7.537
7.532
7.520
7.518
7.501
7.497
7.491
7.487
7.480
7.476
7.466
7.461
7.458
7.453
7.449
7.444
7.436
4.734
4.722
4.073
4.059
4.052
4.046
4.039
4.026
3.911
3.866
3.844
3.829
3.798
NC
CN
O
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 ppm
0.99
2.00
1.99
2.05
5.04
0.97
1.00
2.00


BRUKER
Current Data Parameters
NAME 2009-12-26 shilan 197-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091226
Time 15.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00



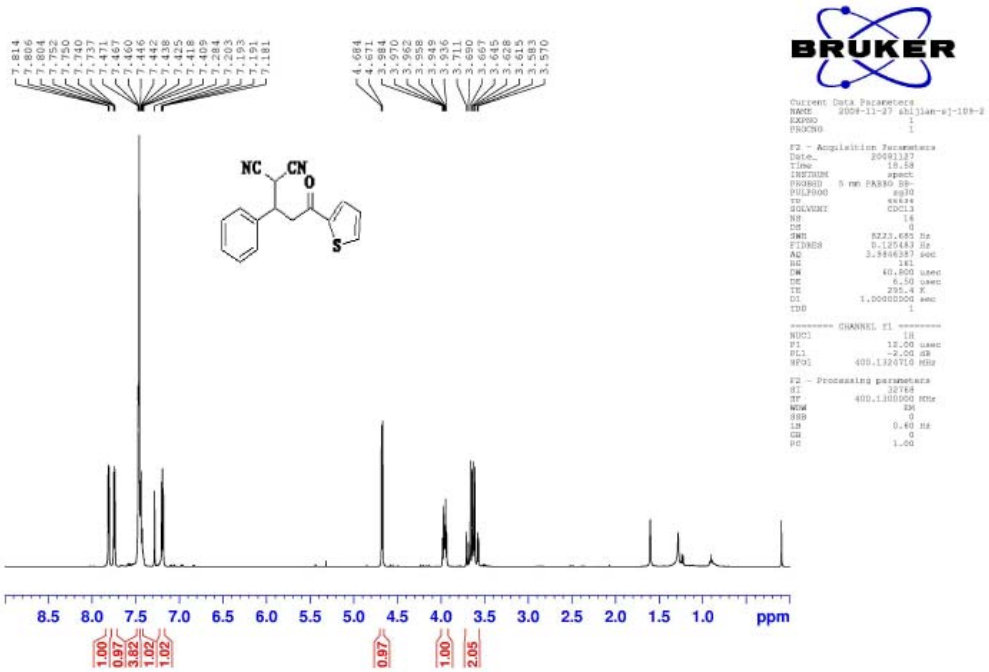
Current Data Parameters
NAME 2008-12-26 shijian 107-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081226
Time 16.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

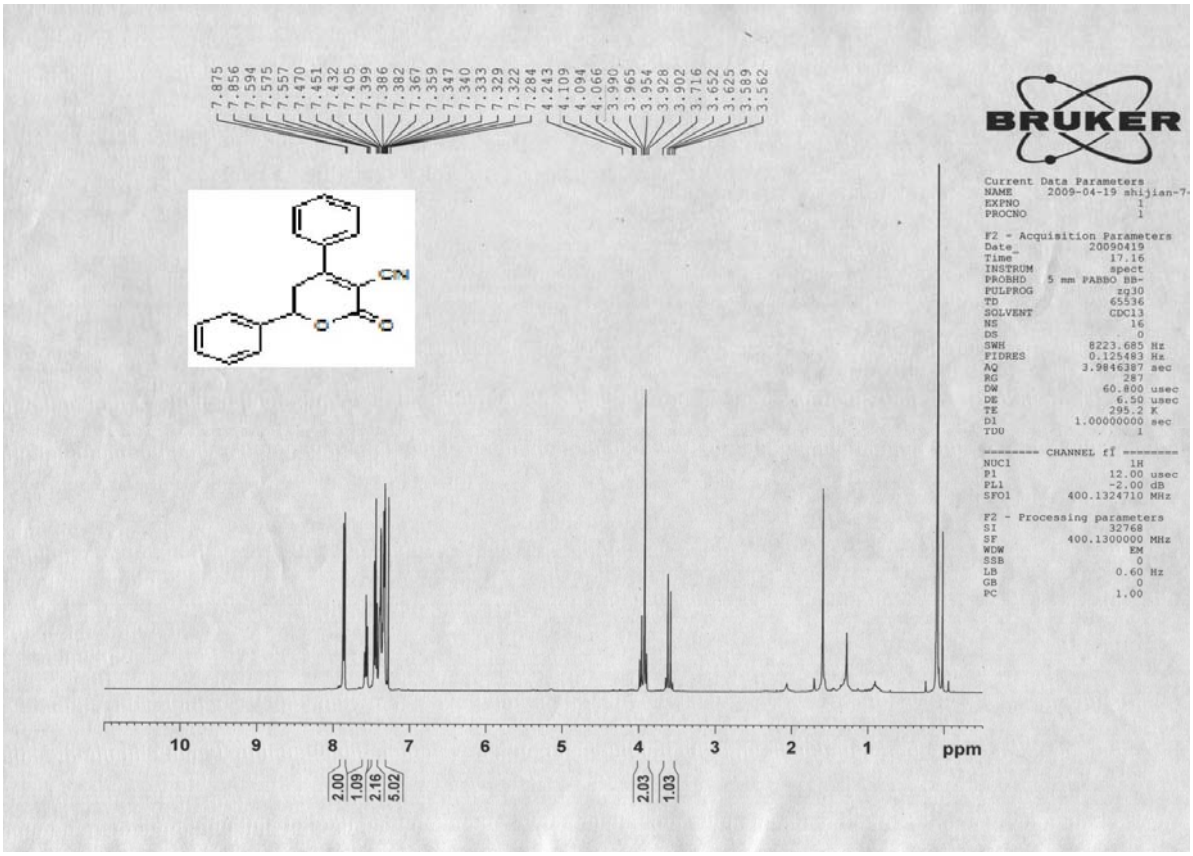


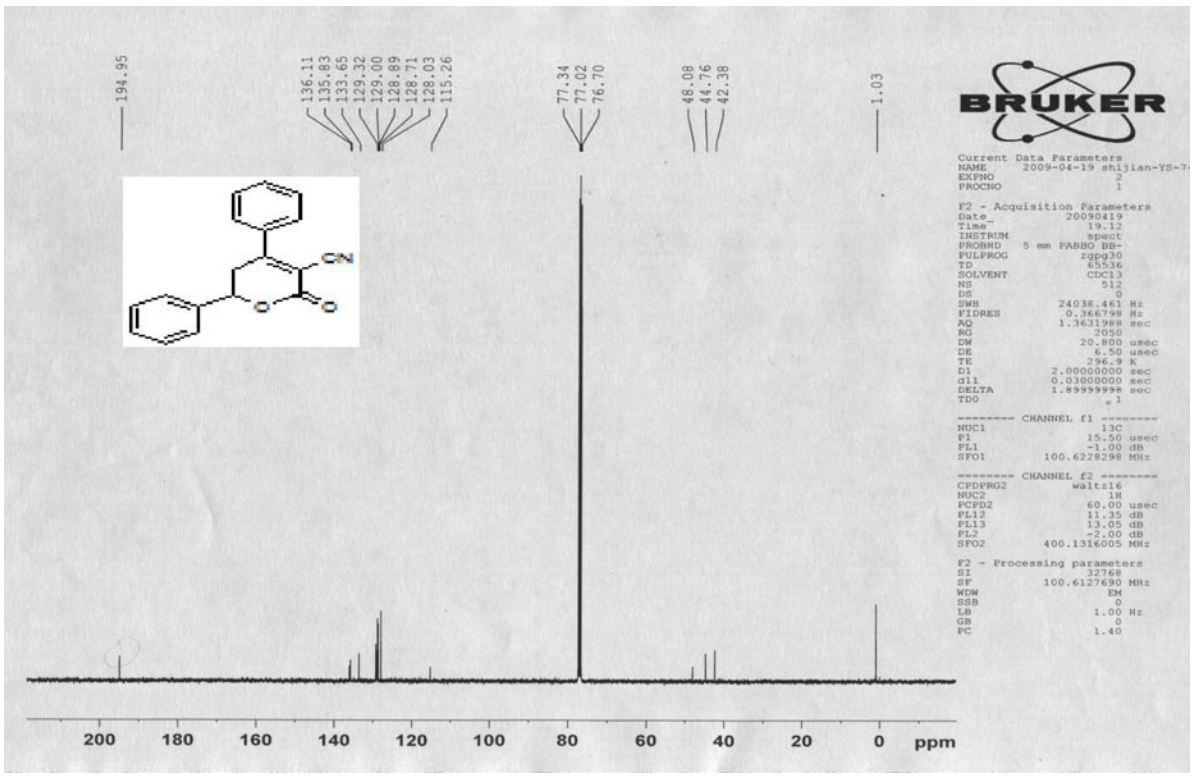
Current Data Parameters
NAME 2009-11-27 shijian-sj-109-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091127
Time 18.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1320710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00


7.875
7.856
7.594
7.575
7.557
7.470
7.451
7.432
7.405
7.399
7.386
7.382
7.367
7.359
7.347
7.340
7.333
7.329
7.322
7.284
4.243
4.109
4.094
4.066
3.990
3.965
3.954
3.928
3.902
3.716
3.652
3.625
3.589
3.562
CN
O
O
BRUKER
Current Data Parameters
NAME 2009-04-19 shijian-7-
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090419
Time_ 17.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.00
1.09
2.16
5.02
2.03
1.03


194.95
136.11
135.83
133.65
129.32
129.00
128.89
128.71
128.03
115.26
77.34
77.02
76.70
48.08
44.76
42.38
1.03
CN
O
O
BRUKER
Current Data Parameters
NAME 2009-04-19 shijian-YS-7-
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090419
Time_ 19.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

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2 (a) E. Solcaniova and S. Toma, *Org. Mag. Res*., 1980, **14**, 138; (b) K. Hino, Y. Nagai, H. Uno, Y. Masuda, M. Oka and T. Karasawa, *J. Med. Chem*., 1988, **31**, 107; (c) H. Xian, X. Linghong and I. N. Hong, *J. Org. Chem*., 1988, **53**, 4862; (d) J. Huang and Y.-Z. Huang, *J. Organomet. Chem*., 1991, **414**, 49; (e) D. S. Noyce and M. J. Jorgenson, *J. Am. Chem. Soc*., 1962, **84**, 4312; (f) B. C. Ranu and R. *J.* Jana, *Org. Chem.***,** 2005, **70**, 8621; (g) S. N. Lopez, M. V. Castelli, S. A. Zacchino, J. N. Dominguez, G. Lobo, J. Charris-Charris, J. C. G. Cortes, J. C. Ribas, C. Devia, A. M. Rodriguez and R. D. Enriz, *Bioorg. Med. Chem*., 2001, **9**, 1999; (h) P. Peach, D. J. Cross, J. A. Kenny, I. Mann, I. Houson, L. Campbell, T. Walsgroveb and M. Wills, *Tetrahedron*, 2006, **62**, 1864; (i) J. M. Concello, P. L. Bernad, M. Huerta, S. Garcia-Granda and M. R. Diaz, *Chem. Eur. J*., 2003, **9**, 5343; (j) D. Huang, J.-X. Wang, Y. Hu, Y. Zhang and J. Tang, *Synth. Commun*., 2002, **32**, 971; (k) J. Wu, D. Zhang and S. Wei, *Synth. Commun*., 2005, **35**, 1213.

3 a) M. Quinteiro, C. Seoane and J. L. Soto, *J. Heterocyclic. Chem*., 1978, **15**, 57; b) D. V. Tyndall, T. A. Nakib and M. J. Meegan, *Tetrahedron Lett*., 1988, **29**, 2703.