

Synthesis of 3-Indole Derivatives by Copper Sulfonato Salen-Catalyzed Three-Component Reactions in Water

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Supplementary Information

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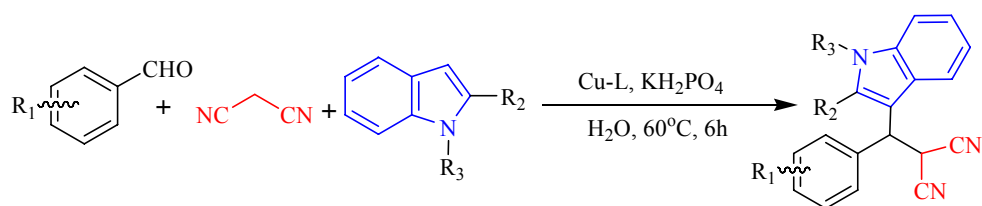
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1. General information: All reagents were purchased from commercial suppliers and used without further purification. Column chromatography was performed on silica gel (200–300 mesh), eluting with ethyl acetate and petroleum ether. Thin layer chromatography was carried out using Merck silica gel GF254 plates. ^1H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra were recorded at 100 MHz (Bruker DPX) with CDCl_3 or $\text{DMSO}-d_6$ as solvent. Chemical shifts are reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard. Coupling constants (J) are reported in Hz and refer to apparent peak multiplications.

2. General procedure for the synthesis of Ligands L^1 - L^8 ^{8|1,2|}

Ligands were prepared as previously described:^{1,2} disodium salicylaldehyde 5-sulfonates (20 mmol) was heated to reflux temperature and then diamine (10 mmol) was drop-wise added in methanol (25 mL) and stirred at reflux temperature for 2-3 h. The reaction mixture was then cooled to 0°C. A precipitate was formed. After filtration and washed by cold methanol, L^1 - L^8 were obtained in yields ranging from 78% to 92%.

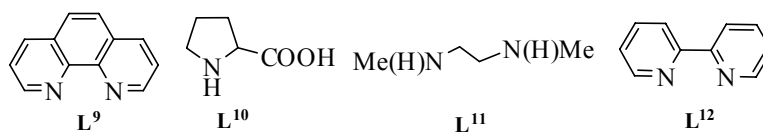
3. General procedure for the catalyzed three-component reaction between indole, aldehyde, and malononitrile in water



In a typical procedure, $\text{Cu}(\text{OAc})_2$ (0.19 mg, 0.001 mmol) and ligand 0.001 mmol was added 3.0 mL water and the mixture was stirred at room temperature for 2 hrs. Then, 13.6 mg KH_2PO_4 (0.1 mmol), aldehyde (0.1 mmol), the malononitrile (0.11 mmol), and indole (0.11 mmol) was added sequentially to form clear yellow solution. The resulting mixture was stirred at 60°C for 6hrs and then cooled to room temperature and extracted with ethyl acetate. The organic layer was dried over Na_2SO_4 and the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography (petroleum ether/AcOEt: 8:1 to 4:1) to give the desired product.

Besides the ligands L^1 - L^8 , the following ligands L^9 - L^{12} were also tried in this work, and the related results were listed below in Table 1.

Table 1. Screening of other ligands for the catalytic three-component reaction

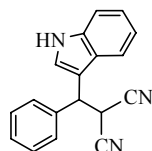


Entry	Metal(mol%)	L (mol%)	Base/acid	Yield(%) ^b
1	Cu(OAc) ₂ (5)	L ⁹ (5)	KH ₂ PO ₄	79
2	Cu(OAc) ₂ (5)	L ¹⁰ (5)	KH ₂ PO ₄	63
3	Cu(OAc) ₂ (5)	L ¹¹ (5)	KH ₂ PO ₄	48
4	Cu(OAc) ₂ (5)	L ¹² (5)	KH ₂ PO ₄	72

^a Unless otherwise specified, all reactions were carried out using metal salt and ligand 5 mol%, 1.0 equiv of acid/base, benzaldehyde 0.1 mmol, propanedinitrile 0.11 mmol, indole 0.11 mmol in water (3 mL) at 60 °C for 6 hours. ^b Isolated yield.

4. Experimental procedures and characterization data

2-((1H-indol-3-yl)(phenyl)methyl)malononitrile



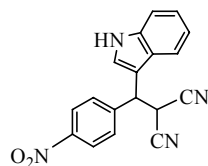
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a yellow solid in 96% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.23(s, 1H), 7.57(d, *J* = 2.4 Hz, 1H), 7.52(d, *J* = 8.0 Hz, 2H), 7.48(d, *J* = 8.0 Hz, 1H), 7.40-7.36(m, 3H), 7.31-7.27(m, 1H), 7.10(t, *J* = 8.0 Hz, 1H), 6.96(t, *J* = 7.6 Hz, 1H), 5.86(d, *J* = 9.2 Hz, 1H), 5.22(d, *J* = 9.6 Hz, 1H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 139.2, 136.0, 128.6, 128.0, 125.9, 122.7, 121.6, 118.9, 118.5, 114.1, 113.8, 112.3, 111.7, 42.4, 28.6 ppm;

MS (EI, *m/z*): 271 [M⁺]

2-((1H-indol-3-yl)(4-nitrophenyl)methyl)malononitrile



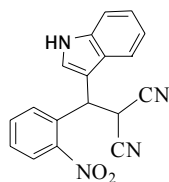
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a yellow solid in 97% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.34(bs, 1H), 8.27(d, *J* = 8.8 Hz, 2H), 7.82(d, *J* = 8.8 Hz, 2H), 7.64(d, *J* = 7.6 Hz, 1H), 7.50(d, *J* = 8.0 Hz, 1H), 7.42(d, *J* = 8.0 Hz, 1H), 7.12(t, *J* = 7.2 Hz, 1H), 6.99(t, *J* = 7.6 Hz, 1H), 6.01(d, *J* = 9.2 Hz, 1H), 5.52(d, *J* = 9.6 Hz, 1H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 147.5, 147.1, 136.6, 129.9, 126.2, 124.4, 123.6, 122.4, 119.6, 118.8, 114.0, 112.3, 111.7, 42.3, 28.6 ppm.

MS (EI, m/z): 316 [M⁺]

2-((1H-indol-3-yl)(2-nitrophenyl)methyl)malononitrile



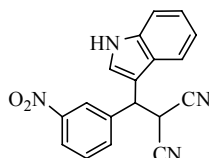
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a yellow solid in 93% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.35(s, 1H), 8.01(d, *J* = 8.0 Hz, 1H), 7.84-7.77(m, 2H), 7.61(t, *J* = 7.6 Hz, 1H), 7.54(s, 1H), 7.40(t, *J* = 7.6 Hz, 2H), 7.12(t, *J* = 7.6 Hz, 1H), 6.98(t, *J* = 7.6 Hz, 1H), 5.97(d, *J* = 9.2 Hz, 1H), 5.76(d, *J* = 8.8 Hz, 1H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 149.7, 136.5, 134.1, 132.9, 130.1, 130.0, 126.1, 125.5, 124.6, 122.4, 119.8, 118.7, 114.0, 113.9, 112.3, 111.0, 38.0, 28.9 ppm.

MS (EI, m/z): 316 [M⁺]

2-((1H-indol-3-yl)(3-nitrophenyl)methyl)malononitrile



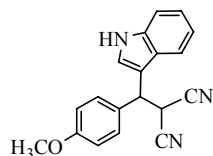
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a yellow solid in 94% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.34(s, 1H), 8.45(s, 1H), 8.18(d, *J* = 8.0 Hz, 1H), 8.02(d, *J* = 7.2 Hz, 2H), 7.73-7.67(m, 2H), 7.52(d, *J* = 8.0 Hz, 1H), 7.41(d, *J* = 8.0 Hz, 1H), 7.12(t, *J* = 7.6 Hz, 1H), 6.99(t, *J* = 7.6 Hz, 1H), 6.03(d, *J* = 9.2 Hz, 1H), 5.55(d, *J* = 9.6 Hz, 1H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 148.4, 141.9, 136.6, 135.3, 130.8, 126.2, 123.6, 123.4, 123.3, 122.4, 119.6, 118.8, 114.2, 114.0, 112.3, 111.8, 42.1, 28.8 ppm.

MS (EI, m/z): 316 [M⁺]

2-((1H-indol-3-yl)(4-methoxyphenyl)methyl)malononitrile



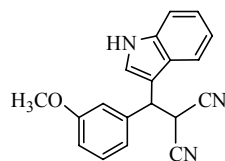
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a pale-yellow solid in 68% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.20(s, 1H), 7.54(d, *J* = 2.4 Hz, 1H), 7.44-7.38(m, 4H), 7.09(t, *J* = 8.0 Hz, 1H), 6.97-6.91(m, 3H), 5.77(d, *J* = 9.2 Hz, 1H), 5.14(d, *J* = 9.2 Hz, 1H), 3.73(s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 159.2, 136.6, 131.6, 129.7, 126.4, 123.0, 122.1, 119.3, 119.1, 114.6, 114.4, 113.1, 112.1, 55.5, 42.3, 29.4 ppm.

MS (EI, m/z): 301 [M⁺]

2-((1H-indol-3-yl)(3-methoxyphenyl)methyl)malononitrile



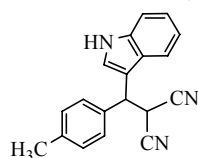
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a pale-yellow solid in 76% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.23(s, 1H), 7.58(s, 1H), 7.54(d, *J* = 8.0 Hz, 1H), 7.41(d, *J* = 8.4 Hz, 1H), 7.30(t, *J* = 7.6 Hz, 1H), 7.12(d, *J* = 6.8 Hz, 2H), 7.10(s, 1H), 6.99(t, *J* = 7.2 Hz, 1H), 6.87(d, *J* = 8.0 Hz, 1H), 5.88(d, *J* = 9.6 Hz, 1H), 5.36(d, *J* = 9.2 Hz, 1H), 3.74(s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 159.8, 141.3, 136.5, 130.2, 126.4, 123.2, 122.2, 120.6, 119.4, 119.1, 114.7, 114.6, 114.3, 113.1, 112.8, 112.2, 55.5, 42.9, 29.0 ppm.

MS (EI, *m/z*): 301 [M⁺]

2-((1H-indol-3-yl)(*p*-tolyl)methyl)malononitrile



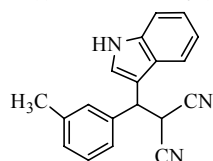
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a pale-yellow solid in 73% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.21(s, 1H), 7.55(d, *J* = 2.4 Hz, 1H), 7.46(d, *J* = 8.0 Hz, 1H), 7.39(d, *J* = 7.6 Hz, 3H), 7.17(d, *J* = 8.0 Hz, 2H), 7.10(t, *J* = 7.6 Hz, 1H), 6.95(t, *J* = 7.6 Hz, 1H), 5.81(d, *J* = 9.2 Hz, 1H), 5.16(d, *J* = 9.2 Hz, 1H), 2.27(s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 137.5, 136.7, 136.6, 129.6, 128.4, 126.4, 123.1, 122.1, 119.3, 119.1, 114.6, 114.4, 113.0, 112.1, 42.6, 29.2, 21.1 ppm.

MS (EI, *m/z*): 285 [M⁺]

2-((1H-indol-3-yl)(*m*-tolyl)methyl)malononitrile



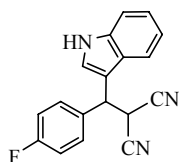
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a pale-yellow solid in 82% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.21(s, 1H), 7.55(d, *J* = 1.6 Hz, 1H), 7.50(d, *J* = 8.0 Hz, 1H), 7.39(d, *J* = 8.4 Hz, 1H), 7.33(d, *J* = 6.8 Hz, 2H), 7.25(t, *J* = 7.6 Hz, 1H), 7.09(t, *J* = 7.2 Hz, 2H), 6.96(t, *J* = 7.6 Hz, 1H), 5.83(d, *J* = 9.6 Hz, 1H), 5.16(d, *J* = 9.2 Hz, 1H), 2.28(s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 139.2, 137.7, 136.0, 128.5, 128.4, 125.9, 125.0, 122.7, 121.6, 118.9, 118.6, 114.1, 113.9, 112.4, 111.6, 42.4, 28.6, 21.1 ppm.

MS (EI, *m/z*): 285 [M⁺]

2-((4-fluorophenyl)(1H-indol-3-yl)methyl)malononitrile



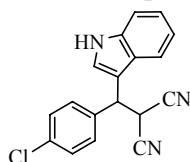
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a pale-yellow solid in 87% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.27(s, 1H), 7.5973-7.56(m, 3H), 7.47(d, *J* = 8.0 Hz, 1H), 7.41(t, *J* = 8.4 Hz, 1H), 7.23(t, *J* = 8.8 Hz, 2H), 7.11(t, *J* = 7.6 Hz, 1H), 6.98(t, *J* = 7.6 Hz, 1H), 5.86(d, *J* = 9.2 Hz, 1H), 5.29(d, *J* = 9.2 Hz, 1H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 163.3, 160.9, 136.6, 135.9, 135.9, 130.7, 130.6, 126.3, 123.2, 122.2, 119.5, 119.0, 116.1, 115.8, 114.5, 114.2, 112.7, 112.2, 42.1, 29.2 ppm.

MS (EI, *m/z*): 289 [M⁺]

2-((4-chlorophenyl)(1H-indol-3-yl)methyl)malononitrile



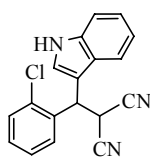
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a white solid in 93% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.26 (s, 1H), 7.57(d, *J* = 2.4 Hz, 1H), 7.54(d, *J* = 8.0 Hz, 2H), 7.45(d, *J* = 8.4 Hz, 3H), 7.39(d, *J* = 8.4 Hz, 1H), 7.10(t, *J* = 7.6 Hz, 1H), 6.97(t, *J* = 7.6 Hz, 1H), 5.86(d, *J* = 9.2 Hz, 1H), 5.284(d, *J* = 9.2 Hz, 1H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 138.7, 136.6, 133.0, 130.4, 129.1, 126.3, 123.3, 122.2, 119.5, 119.0, 114.4, 114.2, 112.4, 112.2, 42.1, 29.0 ppm.

MS (EI, *m/z*): 305 [M⁺]

2-((2-chlorophenyl)(1H-indol-3-yl)methyl)malononitrile



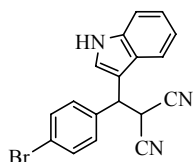
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a white solid in 91% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.31 (bs, 1H), 7.65-7.63(m, 1H), 7.55(t, 2H), 7.48(d, *J* = 8.0 Hz, 1H), 7.40(t, 2H), 7.36-7.32(m, 1H), 7.12(t, *J* = 7.2 Hz, 1H), 7.00(t, *J* = 7.6 Hz, 1H), 5.96(d, *J* = 10.0 Hz, 1H), 5.58(d, *J* = 9.6 Hz, 1H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 136.7, 136.5, 133.5, 130.4, 130.1, 129.5, 128.3, 126.2, 124.1, 122.3, 119.7, 118.6, 114.1, 113.9, 112.3, 111.6, 28.4 ppm.

MS (EI, *m/z*): 305 [M⁺]

2-((4-bromophenyl)(1H-indol-3-yl)methyl)malononitrile



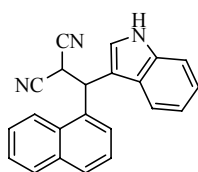
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a yellow solid in 89% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 7.98 (s, 1H), 7.41 (d, *J* = 8.3 Hz, 2H), 7.23-7.20 (m, 4H), 7.10 (t, *J* = 7.2 Hz, 1H), 6.97 (t, *J* = 7.6 Hz, 1H), 4.81 (d, *J* = 9.4 Hz, 1H), 4.54 (d, *J* = 9.4 Hz, 1H), 2.34 (s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 139.1, 136.6, 132.1, 130.8, 126.3, 123.3, 122.2, 121.6, 119.5, 119.0, 114.4, 114.2, 112.3, 112.2, 42.2, 28.9 ppm.

MS (EI, *m/z*): 349 [M⁺]

2-((1H-indol-3-yl)(naphthalen-1-yl)methyl)malononitrile



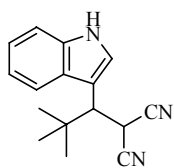
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a white solid in 84% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.19 (s, 1H), 8.49 (d, *J* = 8.0 Hz, 1H), 7.96 (t, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.77 (d, *J* = 7.2 Hz, 1H), 7.59-7.51 (m, 4H), 7.47 (d, *J* = 2.4 Hz, 1H), 7.35 (d, *J* = 8.4 Hz, 1H), 7.06 (t, *J* = 7.6 Hz, 1H), 6.93 (t, *J* = 8.0 Hz, 1H), 6.15 (d, *J* = 9.6 Hz, 1H), 5.99 (d, *J* = 9.6 Hz, 1H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 136.6, 135.5, 134.1, 131.4, 129.3, 128.7, 127.1, 126.4, 126.2, 125.8, 125.1, 124.6, 124.0, 122.0, 119.4, 119.1, 114.6, 113.0, 112.2, 38.1, 29.0 ppm.

MS (EI, *m/z*): 308 [M⁺]

2-(1-(1H-indol-3-yl)-2,2-dimethylpropyl)malononitrile



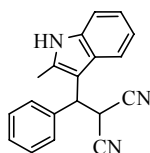
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a yellow solid in 88% yield.

¹H NMR (400 MHz, CDCl₃) δ = 8.41 (s, 1H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.57 (d, *J* = 1.6 Hz, 1H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.29-7.25 (m, 1H), 7.21 (t, *J* = 7.6 Hz, 1H), 4.30 (d, *J* = 8.4 Hz, 1H), 3.57 (d, *J* = 4.0 Hz, 1H), 1.58 (s, 9H) ppm

¹³C NMR (100 MHz, CDCl₃) δ = 135.1, 128.5, 122.7, 120.2, 118.2, 114.0, 113.5, 111.4, 111.1, 47.8, 35.7, 28.3, 26.0 ppm.

MS (EI, *m/z*): 251 [M⁺]

2-((2-methyl-1H-indol-3-yl)(phenyl)methyl)malononitrile



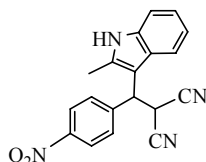
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a yellow solid in 92% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.31(s, 1H), 7.57(d, *J* = 8.0 Hz, 1H), 7.52(d, *J* = 7.6 Hz, 2H), 7.38(t, *J* = 7.6 Hz, 2H), 7.30-7.25(m, 2H), 7.02(t, *J* = 7.2 Hz, 1H), 6.92(t, *J* = 7.6 Hz, 1H), 6.03(d, *J* = 10.8 Hz, 1H), 5.23(d, *J* = 10.8 Hz, 1H), 2.51(s, 3H) ppm

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 139.4, 135.9, 134.8, 129.0, 128.0, 127.7, 126.3, 120.9, 119.2, 118.9, 114.8, 114.5, 111.3, 108.0, 43.0, 27.6, 12.2 ppm.

MS (EI, *m/z*): 285 [M⁺]

2-((2-methyl-1H-indol-3-yl)(4-nitrophenyl)methyl)malononitrile



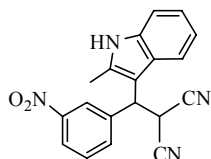
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a yellow solid in 95% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.24(s, 1H), 8.27(d, *J* = 8.4 Hz, 2H), 7.79(d, *J* = 8.4 Hz, 2H), 7.53(d, *J* = 8.0 Hz, 1H), 7.30(d, *J* = 8.0 Hz, 1H), 7.03(t, *J* = 7.2 Hz, 1H), 6.93(t, *J* = 7.6 Hz, 1H), 6.13(d, *J* = 10.4 Hz, 1H), 5.51(d, *J* = 10.4 Hz, 1H), 2.54(s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 147.1, 146.8, 135.9, 135.5, 129.3, 126.0, 124.2, 121.1, 119.5, 118.6, 114.4, 114.1, 111.5, 106.8, 42.6, 27.3, 12.2 ppm.

MS (EI, *m/z*): 330 [M⁺]

2-((2-methyl-1H-indol-3-yl)(3-nitrophenyl)methyl)malononitrile



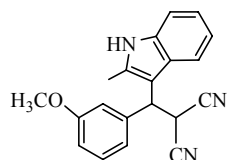
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a yellow solid in 94% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.24(s, 1H), 8.41(s, 1H), 8.17-8.15(m, 1H), 8.00(d, *J* = 8.0 Hz, 1H), 7.70(t, *J* = 8.0 Hz, 1H), 7.58(d, *J* = 8.0 Hz, 1H), 7.31(d, *J* = 8.0 Hz, 1H), 7.04(t, *J* = 7.6 Hz, 1H), 6.95(t, *J* = 7.6 Hz, 1H), 6.18(d, *J* = 10.8 Hz, 1H), 5.52(d, *J* = 10.8 Hz, 1H), 2.54(s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 148.3, 141.6, 135.9, 135.5, 134.8, 130.7, 126.1, 122.9, 122.7, 121.2, 119.5, 118.6, 114.4, 114.1, 111.6, 106.8, 42.4, 27.6, 12.2 ppm.

MS (EI, *m/z*): 330 [M⁺]

2-((3-methoxyphenyl)(2-methyl-1H-indol-3-yl)methyl)malononitrile



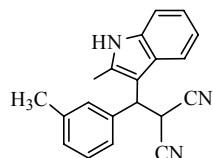
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a buff solid in 82% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.13(s, 1H), 7.62(d, *J* = 8.0 Hz, 1H), 7.31-7.28(m, 2H), 7.09(d, *J* = 8.0 Hz, 2H), 7.03(t, *J* = 7.2 Hz, 1H), 6.93(t, *J* = 7.6 Hz, 1H), 6.84(t, *J* = 7.6 Hz, 1H), 6.03(d, *J* = 10.8 Hz, 1H), 5.19(d, *J* = 10.8 Hz, 1H), 3.73(s, 3H), 2.51(s, 3H) ppm

¹³C NMR (100 MHz, CDCl₃) δ = 159.7, 140.9, 135.9, 134.8, 130.1, 126.3, 120.9, 120.3, 119.2, 118.9, 114.8, 114.3, 112.5, 111.4, 107.9, 55.5, 43.0, 27.6, 12.2 ppm

MS (EI, m/z): 315 [M⁺]

2-((2-methyl-1H-indol-3-yl)(m-tolyl)methyl)malononitrile



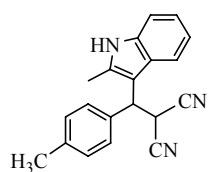
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/4) as a white solid in 85% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.10(s, 1H), 7.56(d, *J* = 8.0 Hz, 1H), 7.34(d, *J* = 8.0 Hz, 1H), 7.27(d, *J* = 6.8 Hz, 3H), 7.08(d, *J* = 7.6 Hz, 1H), 7.01(t, *J* = 7.2 Hz, 1H), 6.91(t, *J* = 7.6 Hz, 1H), 5.99(d, *J* = 10.8 Hz, 1H), 5.16(d, *J* = 10.8 Hz, 1H), 2.50(s, 3H), 2.28(s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 139.3, 138.1, 135.8, 134.7, 128.8, 128.4, 126.3, 124.7, 120.9, 119.2, 118.9, 114.8, 114.5, 111.3, 108.1, 43.0, 27.6, 21.6, 21.5, 12.2 ppm.

MS (EI, m/z): 299 [M⁺]

2-((2-methyl-1H-indol-3-yl)(p-tolyl)methyl)malononitrile



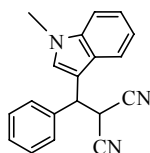
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/8) as a yellow solid in 83% yield.

¹H NMR (400 MHz, CDCl₃) δ = 8.05(s, 1H), 7.38(s, 1H), 7.36(d, *J* = 4.0 Hz, 1H), 7.31(t, *J* = 9.2 Hz, 3H), 7.21(s, 1H), 7.18(d, *J* = 3.6 Hz, 1H), 7.15(d, *J* = 8.0 Hz, 1H), 7.07(t, *J* = 7.6 Hz, 1H), 4.95(d, *J* = 9.6 Hz, 1H), 4.69(d, *J* = 9.6 Hz, 1H), 2.48(s, 3H), 2.36(s, 3H) ppm.

¹³C NMR (100 MHz, CDCl₃) δ = 137.8, 135.4, 134.1, 133.8, 129.7, 127.3, 126.2, 121.8, 120.1, 118.3, 112.8, 110.9, 108.1, 44.0, 27.8, 21.0, 12.4 ppm.

MS (EI, m/z): 299 [M⁺]

2-((1-methyl-1H-indol-3-yl)(phenyl)methyl)malononitrile



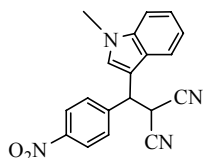
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/8) as a dark yellow solid in 77% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 7.52(t, 4H), 7.43(d, *J* = 8.0 Hz, 1H), 7.38(t, *J* = 7.6 Hz, 2H), 7.30(t, *J* = 7.2 Hz, 1H), 7.17(t, *J* = 7.2 Hz, 1H), 7.01(t, *J* = 7.6 Hz, 1H), 5.82(d, *J* = 9.2 Hz, 1H), 5.23(d, *J* = 9.2 Hz, 1H), 3.82(s, 3H) ppm

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 139.6, 137.0, 129.1, 128.5, 127.5, 126.7, 122.3, 119.5, 119.3, 114.5, 114.3, 112.1, 110.4, 42.7, 33.1, 29.2 ppm.

MS (EI, *m/z*): 285 [M⁺]

2-((1-methyl-1H-indol-3-yl)(4-nitrophenyl)methyl)malononitrile



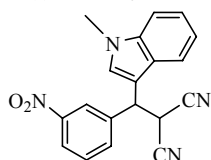
Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/8) as a yellow solid in 83% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.26(d, *J* = 8.4 Hz, 2H), 7.81(d, *J* = 8.4 Hz, 2H), 7.58(s, 1H), 7.54(d, *J* = 8.0 Hz, 1H), 7.45(d, *J* = 8.4 Hz, 1H), 7.19(t, *J* = 7.2 Hz, 1H), 7.03(t, *J* = 7.6 Hz, 1H), 5.97(d, *J* = 9.2 Hz, 1H), 5.53(d, *J* = 9.2 Hz, 1H), 2.51(s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 147.5, 147.0, 137.0, 129.9, 127.8, 126.6, 124.4, 122.5, 119.8, 119.1, 114.0, 110.9, 110.6, 42.1, 33.2, 28.6 ppm.

MS (EI, *m/z*): 330 [M⁺]

2-((1-methyl-1H-indol-3-yl)(3-nitrophenyl)methyl)malononitrile



Following the general procedure the title compound was isolated by flash chromatography (eluent: ethyl acetate/petrol ether =1/8) as a yellow solid in 80% yield.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.45(s, 1H), 8.22(t, *J* = 8.4 Hz, 1H), 8.01(d, *J* = 8.0 Hz, 1H), 7.73-7.69(m, 1H), 7.62(s, 1H), 7.56(d, *J* = 8.0 Hz, 1H), 7.45(d, *J* = 8.4 Hz, 1H), 7.19(t, *J* = 7.2 Hz, 1H), 7.04(t, *J* = 7.6 Hz, 1H), 5.99(d, *J* = 9.2 Hz, 1H), 5.56(d, *J* = 9.2 Hz, 1H), 3.84(s, 3H) ppm.

¹³C NMR (100 MHz, DMSO-*d*₆) δ = 147.9, 141.3, 134.8, 130.4, 130.2, 127.3, 126.1, 124.2, 122.9, 122.8, 122.0, 119.3, 118.6, 110.5, 110.1, 41.4, 32.8, 28.3 ppm.

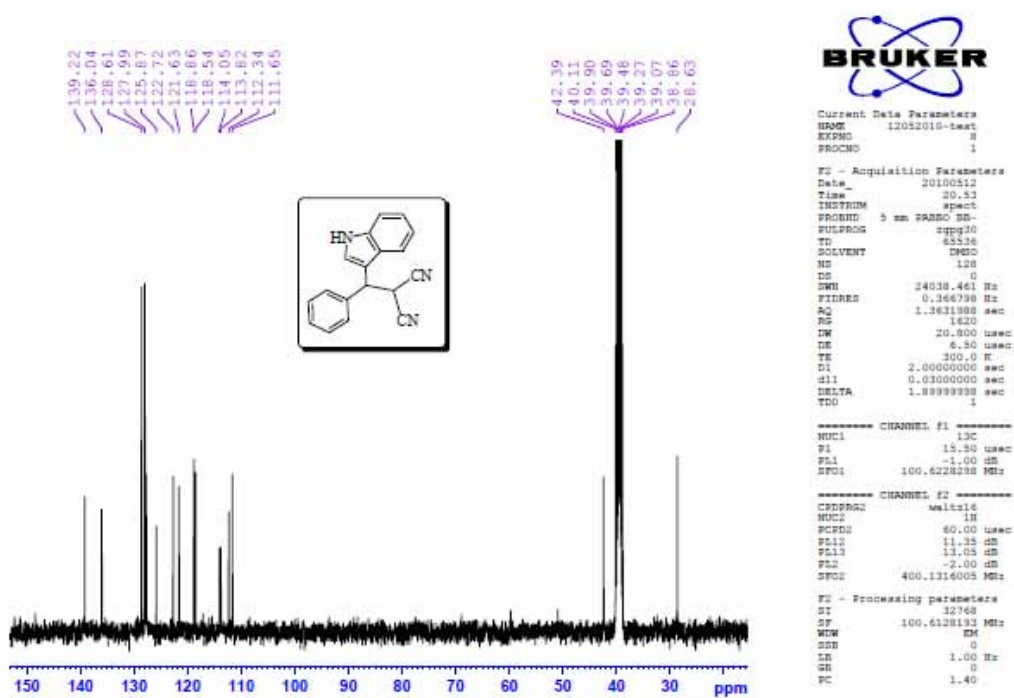
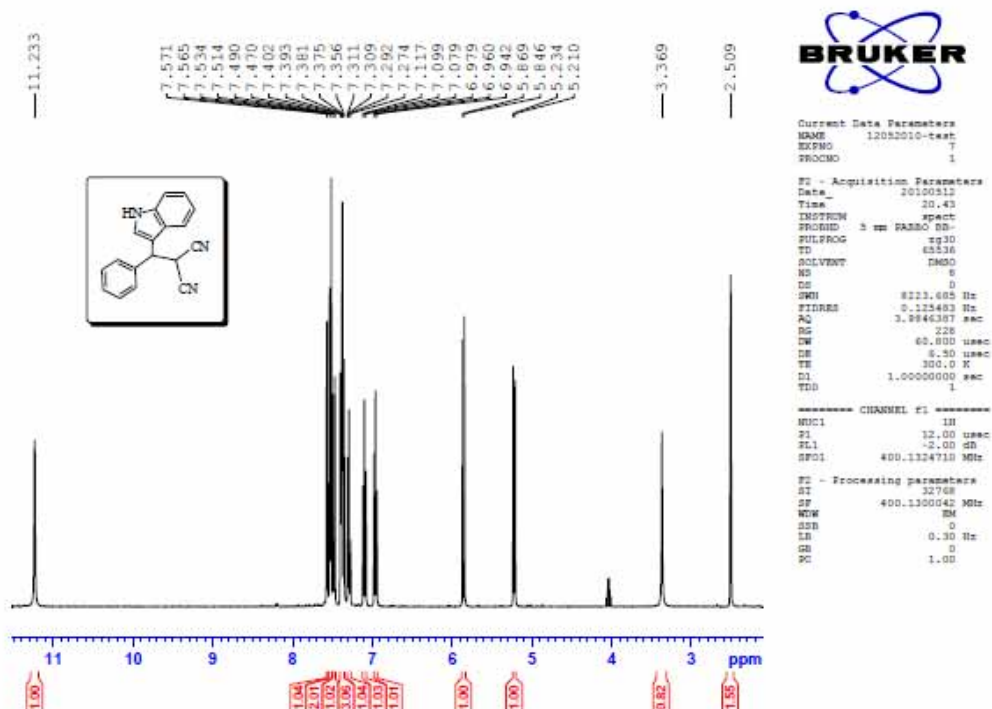
MS (EI, *m/z*): 330 [M⁺]

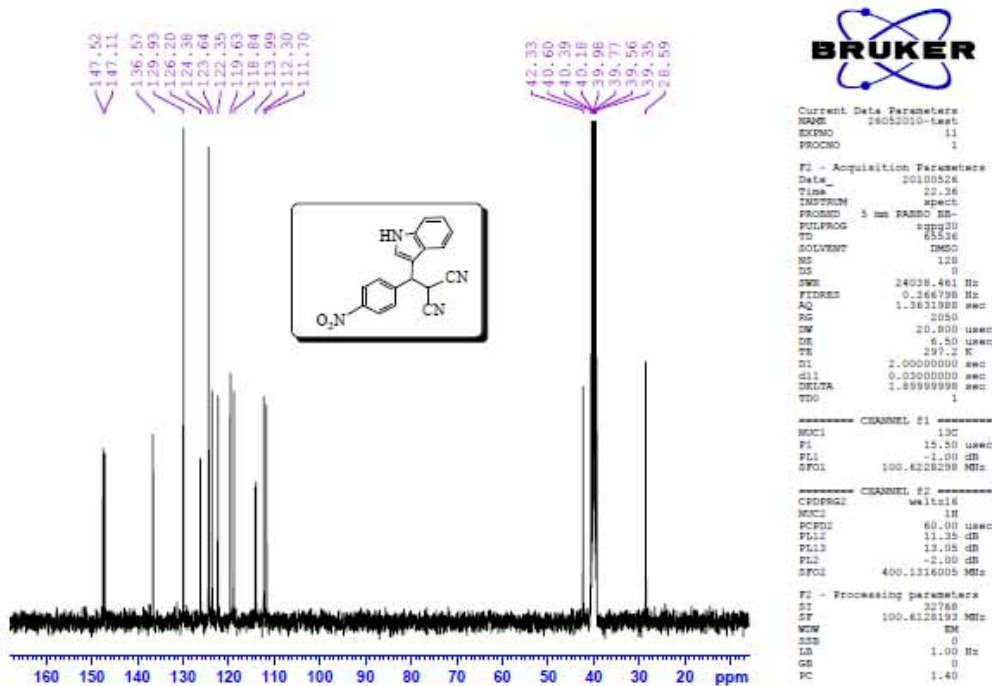
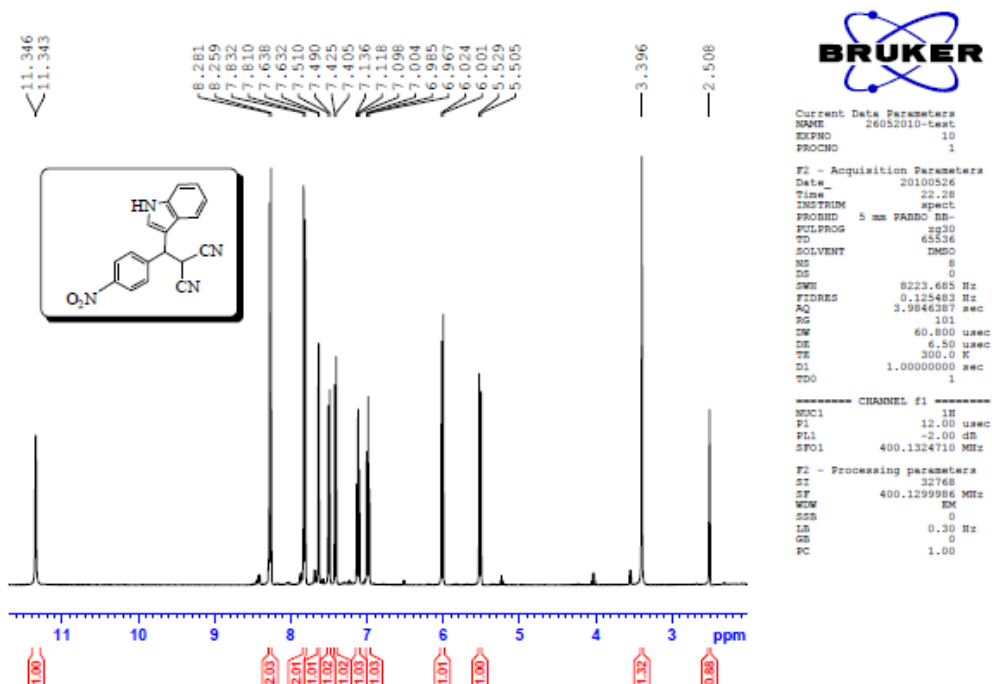
5. References

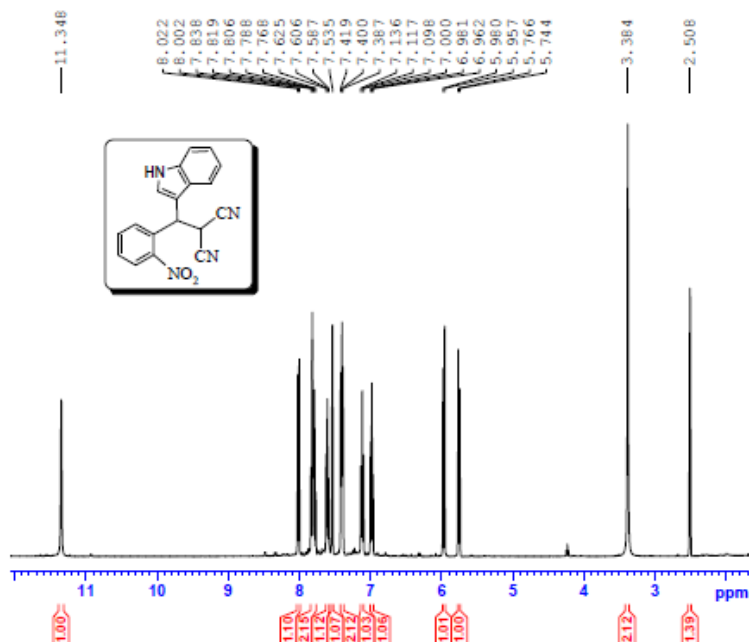
1. (a) Bhattacharjee, S.; Dines, T. J.; Anderson, J. A. J. *Catal.* 2004, 225, 398–407; (b) Choudary, B. M.; Ramani, T.;

- Maheswaran, H.; Pra-shant, L.; Ranganath, K. V. S.; Vijaykumar, K. *Adv. Synth. Catal.* 2006, 348, 493–498;
(c) Tan, R.; Yin, D. H.; Yu, N. Y.; Tao, L.; Fu, Z. H.; Yin, D. L. *J. Mol. Catal. A: Chem.* 2006, 259, 125–132.
2. (a) Larrow, J. F.; Jacobsen, E. N. *J. Org. Chem.* 1994, 59, 1939–1942; (b) Darensbourg, D. J.; Mackiewicz, R. M.; Rodgers, J. L.; Fang, C. C.; Billodeaux, D. R.; Reibenspies, J. H. *Inorg. Chem.* 2004, 43, 6024–6034.

6. ^1H NMR and ^{13}C NMR spectral for the products





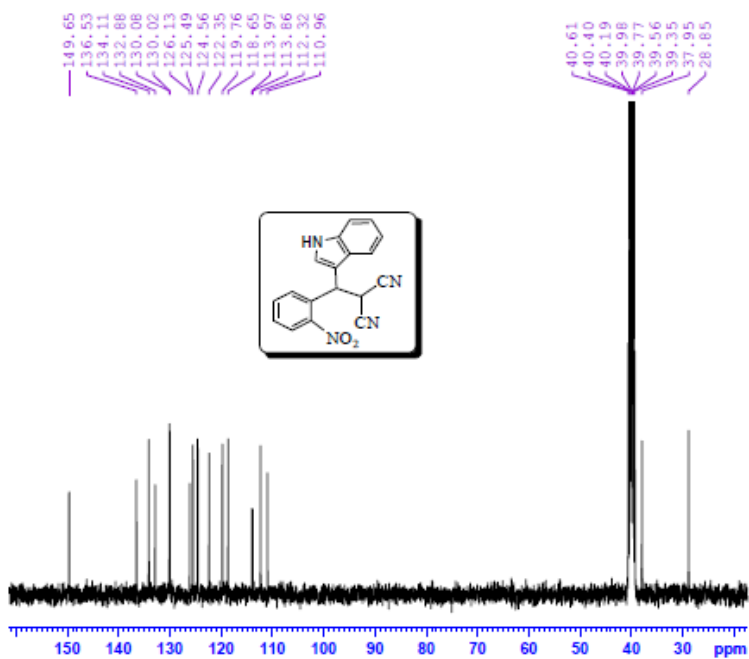


Current Data Parameters
NAME 19052010-test
EXPRNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100519
Time 21.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMF0
NS 16
DS 0
SWH 8223.485 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TDO 1

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

F2 - Processing parameters
SI 32768
SF 400.1299853 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



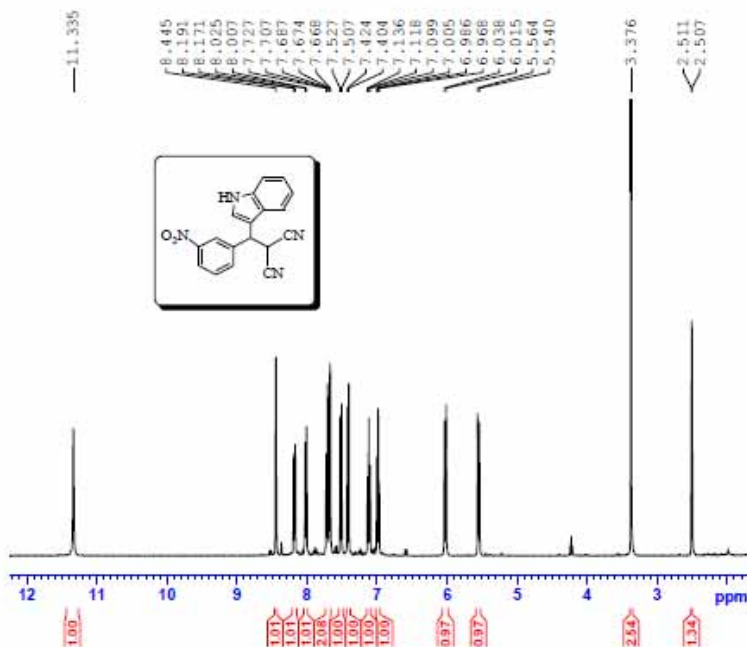
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EXPRNO 4
PROCNO 1

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PULPROG zgpg30
TD 65536
SOLVENT DMF0
NS 128
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 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 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
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SF 100.6128193 MHz
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SSB 0
LB 1.00 Hz
GB 0
PC 1.40

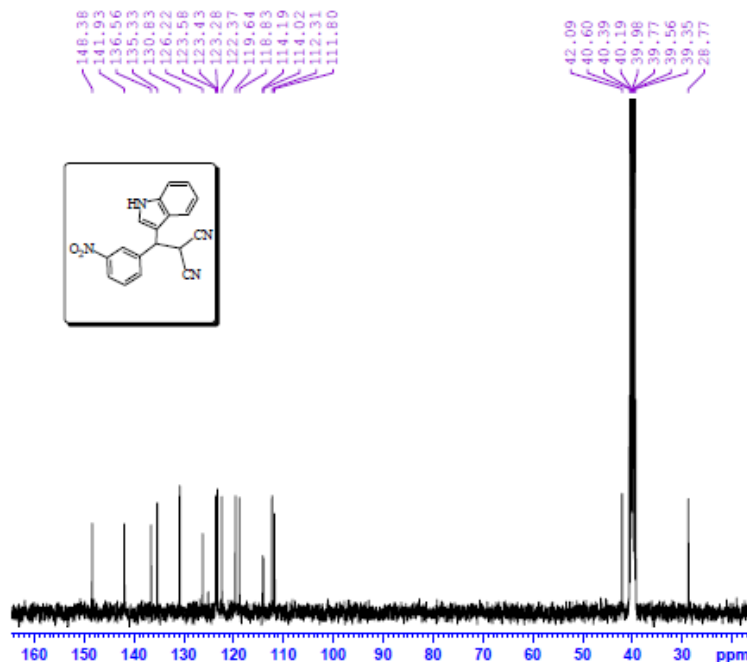


Current Data Parameters
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 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
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 Time 22.12
 INSTRUM spect
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 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 0
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846287 sec
 RG 120
 DW 60.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

----- CHANNEL f1 -----
 NUC1 1H
 P1 12.00 usec
 PL1 -2.00 dB
 SFO1 400.1124710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1238932 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



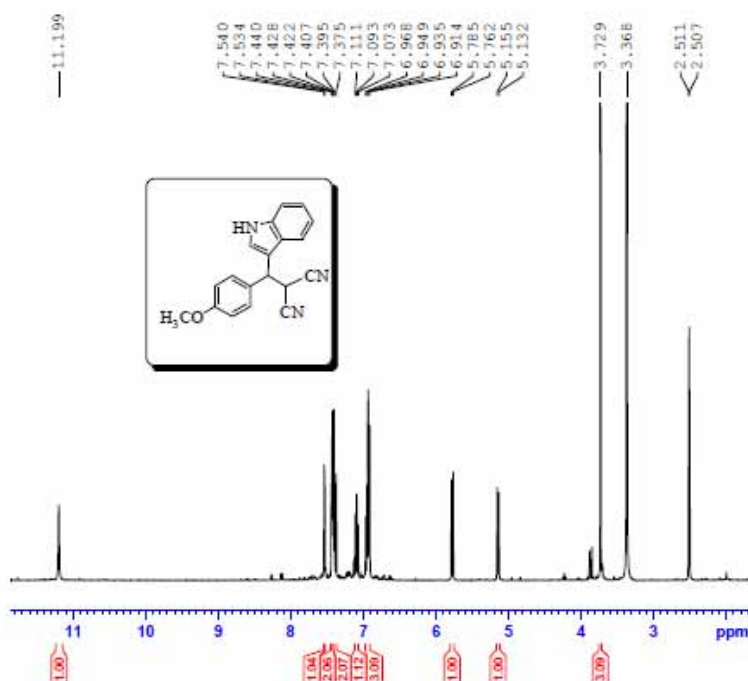
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 EXPNO 8
 PROCNO 1

F2 - Acquisition Parameters
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 Time 22.20
 INSTRUM spect
 PROBRD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 128
 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 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 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 100.6128193 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

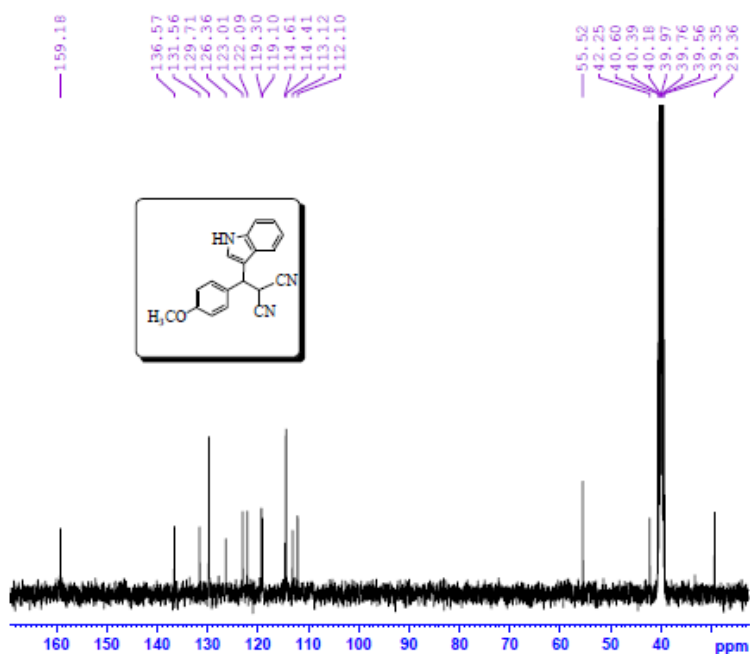


Current Data Parameters
 NAME 31052010-test
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100531
 Time_ 21.42
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 0
 SFO1 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9844387 sec
 RG 226
 DW 60.800 usec
 DE 6.50 usec
 TE 300.0 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 400.1299817 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



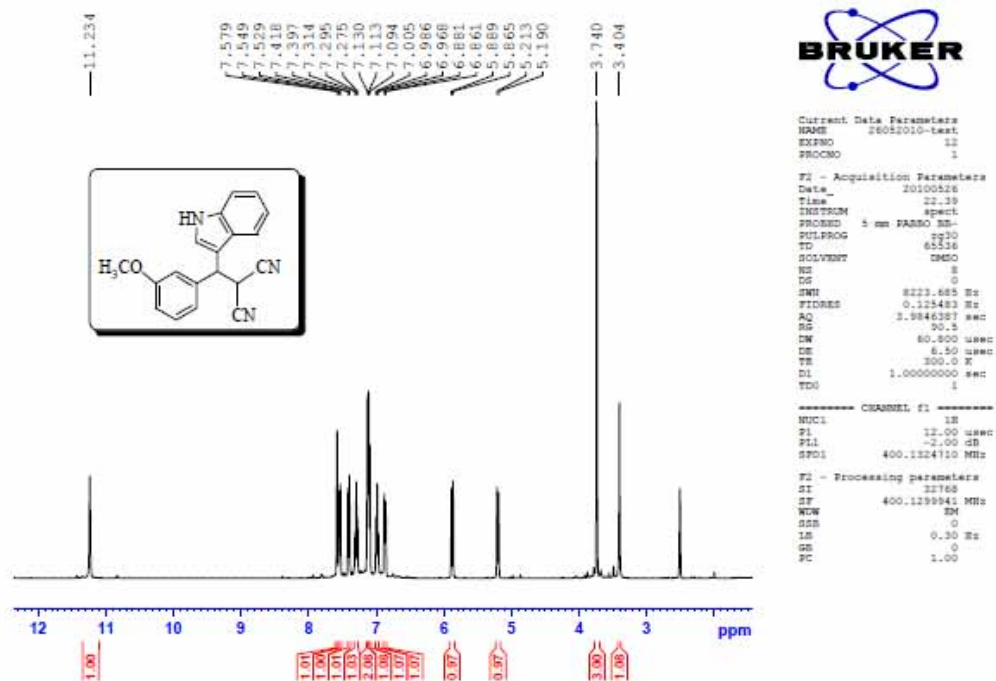
Current Data Parameters
 NAME 31052010-test
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
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 Time_ 21.51
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 128
 DS 0
 SFO1 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03800000 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
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 SF 100.6128193 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

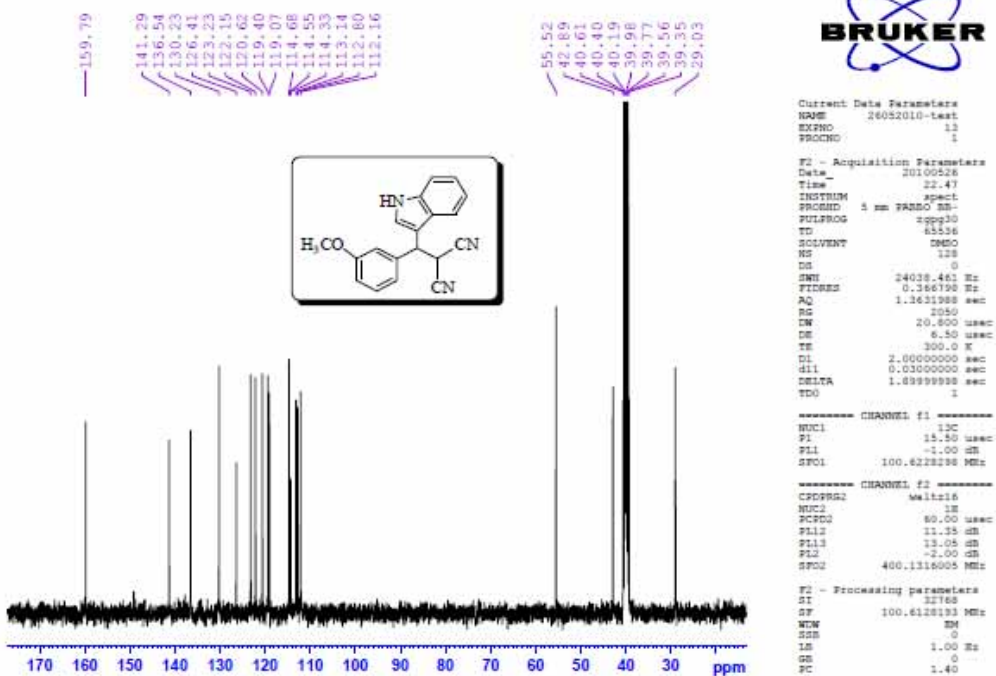


Current Data Parameters
NAME 26032010-Test
EXPNO 12
PROCNO 1

F1 - Acquisition Parameters
Date_ 20100526
Time 22.39
INSTRUM spect
PROBHD 5 mm PARO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 823.885 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 50.5
RW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.124110 MHz

F2 - Processing parameters
SI 32768
SF 400.1299841 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



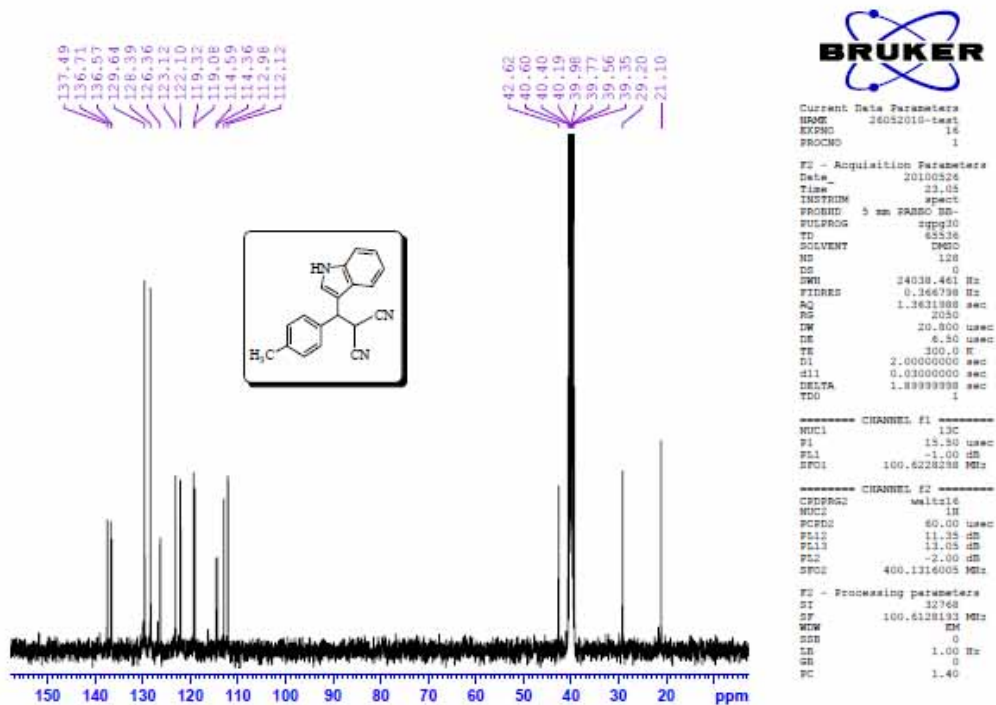
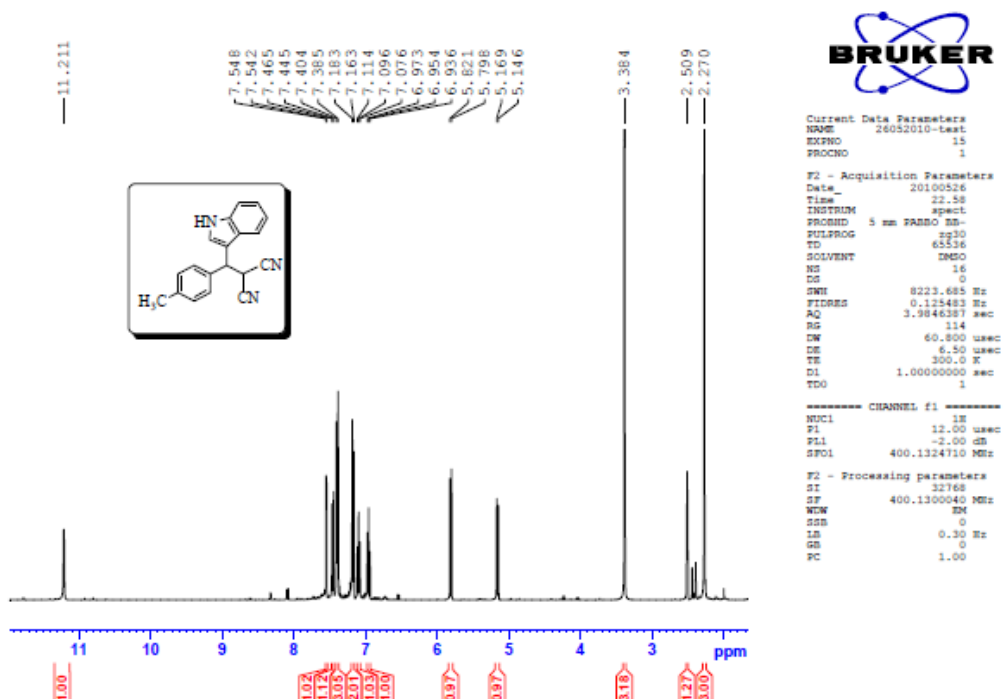
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EXPNO 13
PROCNO 1

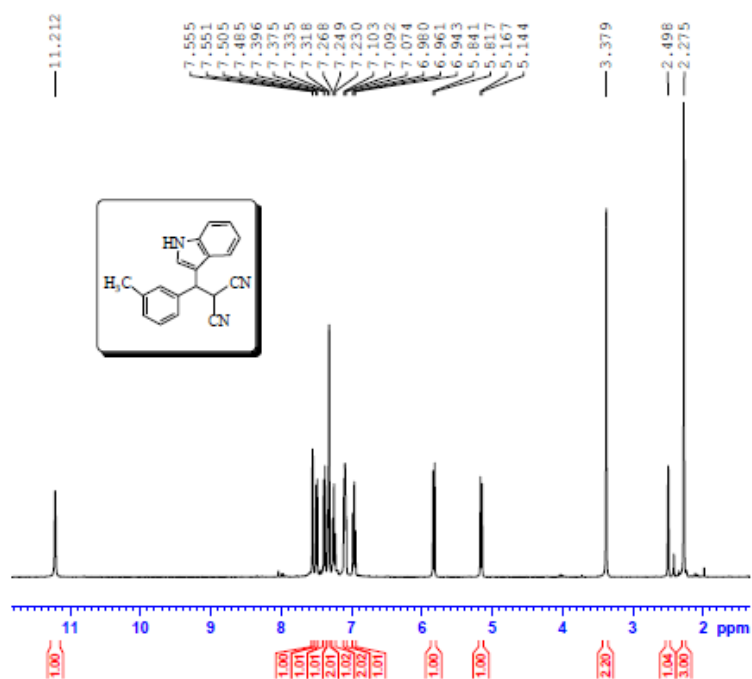
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Time 22.47
INSTRUM spect
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PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 128
DS 0
SWH 24038.461 Hz
FIDRES 0.366790 Hz
AQ 1.1631988 sec
RG 2050
RW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999999 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228238 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
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WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



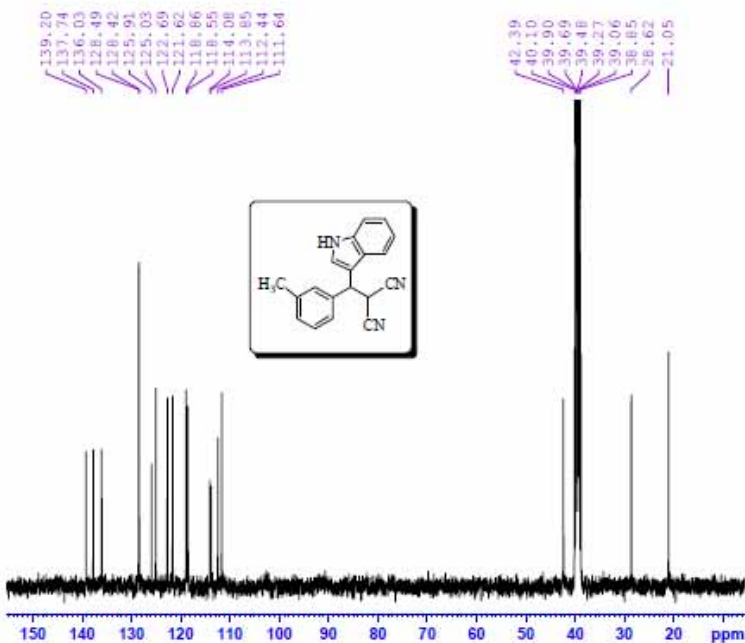


Current Data Parameters
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 PROCNO 1

F2 - Acquisition Parameters
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 Time 21.29
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 TD 65536
 SOLVENT dsgd
 NS 0
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.129483 Hz
 AQ 3.9846387 sec
 RG 101
 DW 60.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

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

F2 - Processing parameters
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 GB 0
 PC 1.00



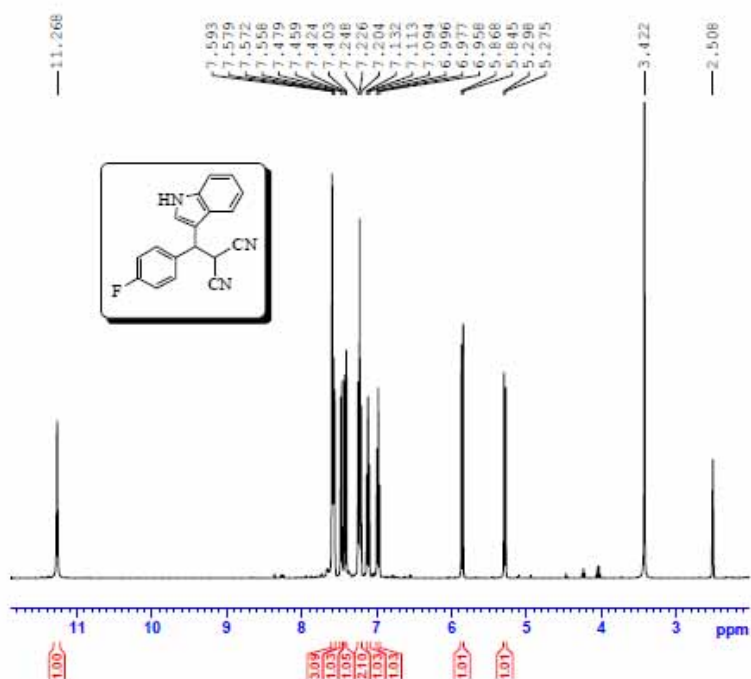
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 EXPNO 1
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 SOLVENT dsgd
 NS 128
 DS 0
 SWH 24030.461 Hz
 FIDRES 0.356798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.13000000 sec
 DELTA 1.89999998 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 15.50 usec
 PL1 -1.00 dB
 SFO1 100.6286298 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD12 60.00 usec
 PL12 11.35 dB
 PL13 13.05 dB
 PL2 -2.00 dB
 SFO2 400.1316003 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6128192 MHz
 MW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

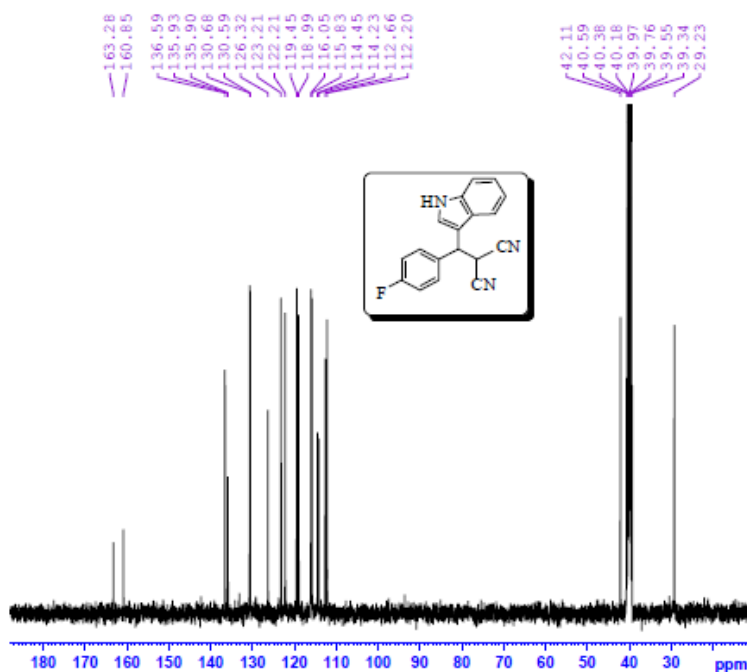


Current Data Parameters
 NAME 28052010-teat
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100528
 Time 22.11
 INSTRUM spect
 PROBRD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846287 sec
 RG 98.5
 DW 60.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

----- CHANNEL f1 -----
 NUCL1 1H
 P1 12.00 usec
 PL1 -2.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300024 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



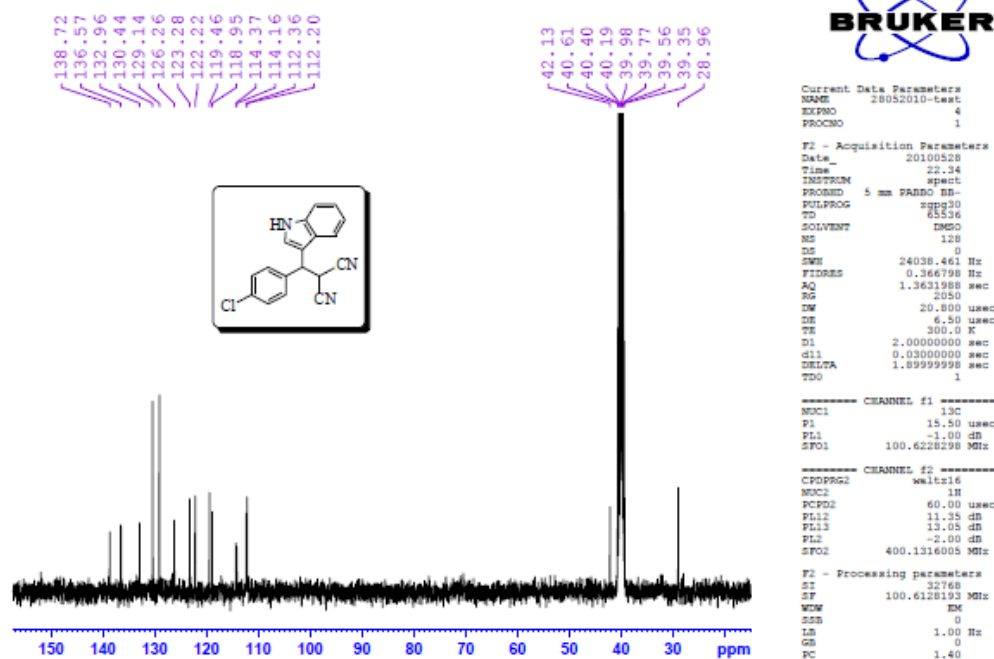
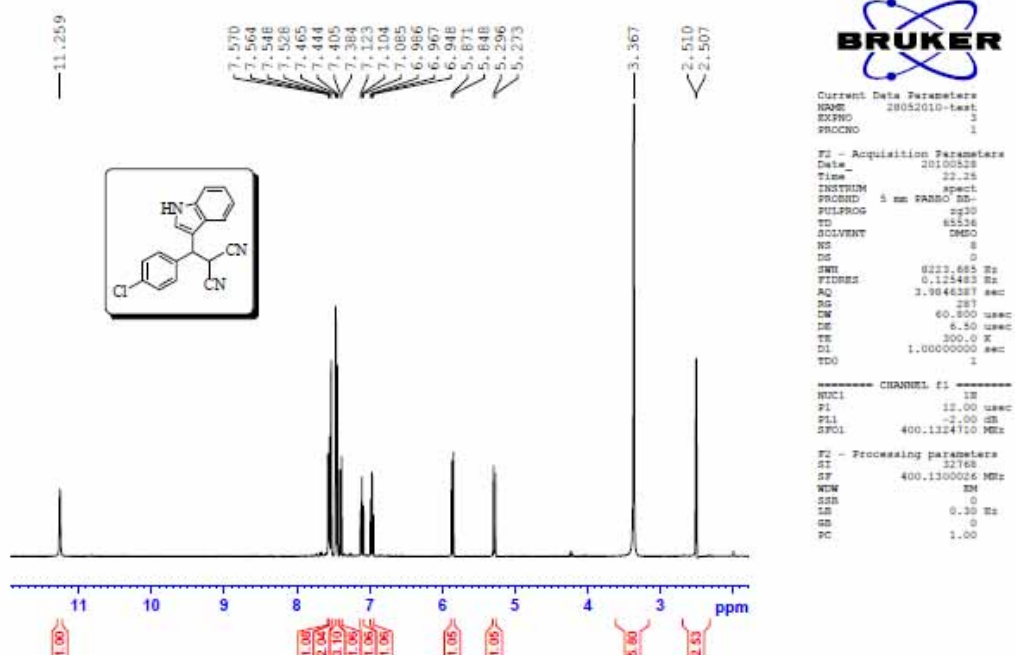
Current Data Parameters
 NAME 28052010-teat
 EXPNO 2
 PROCNO 1

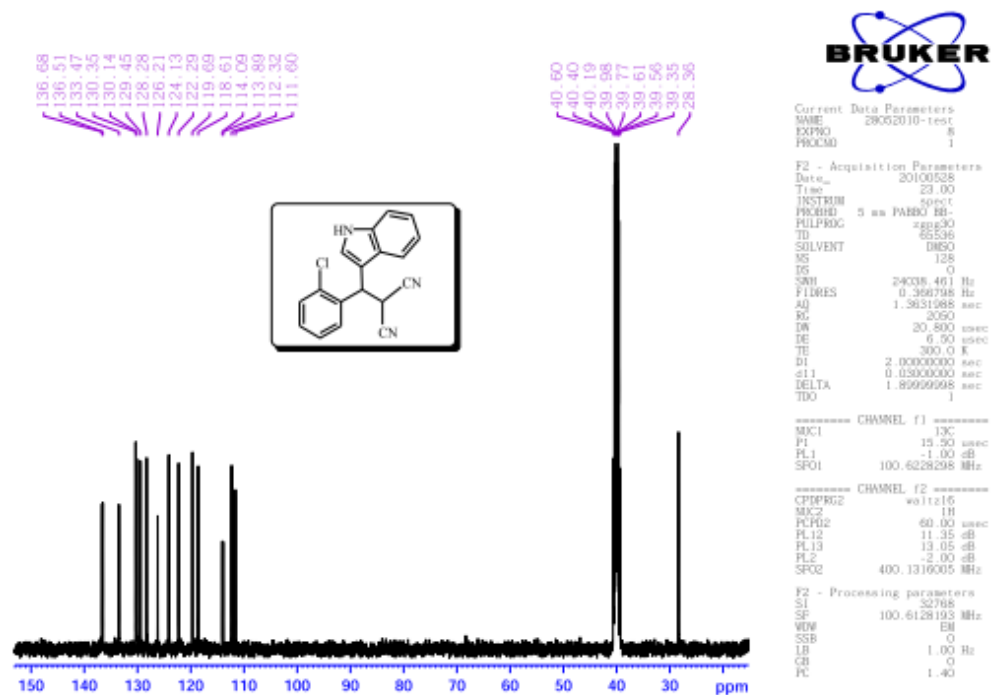
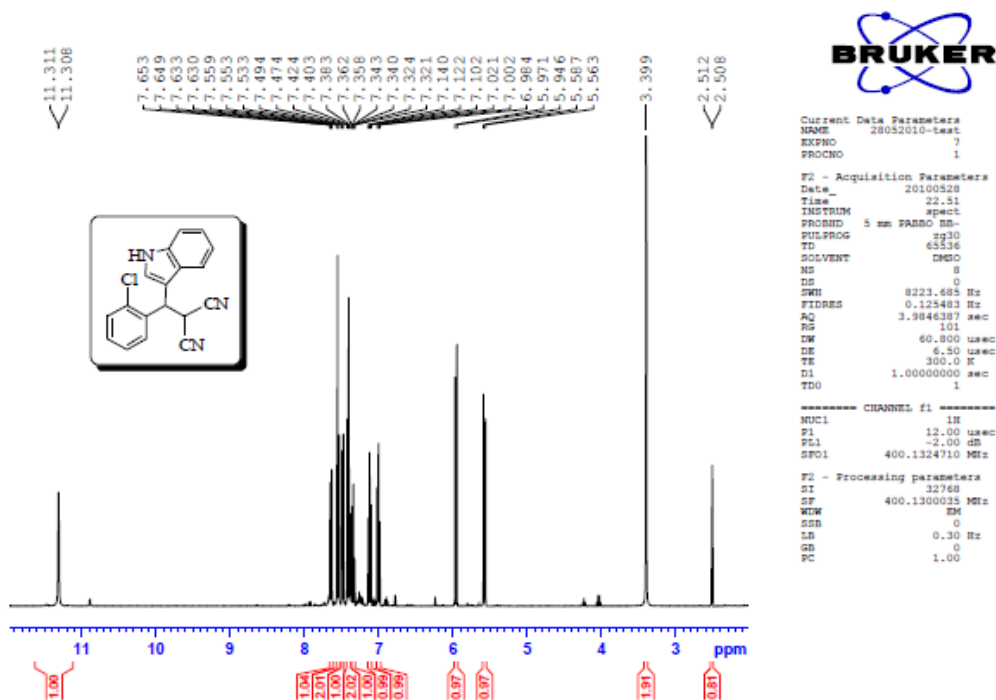
F2 - Acquisition Parameters
 Date_ 20100528
 Time 22.20
 INSTRUM spect
 PROBRD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 128
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2080
 DW 20.800 usec
 DE 6.50 usec
 TE 297.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

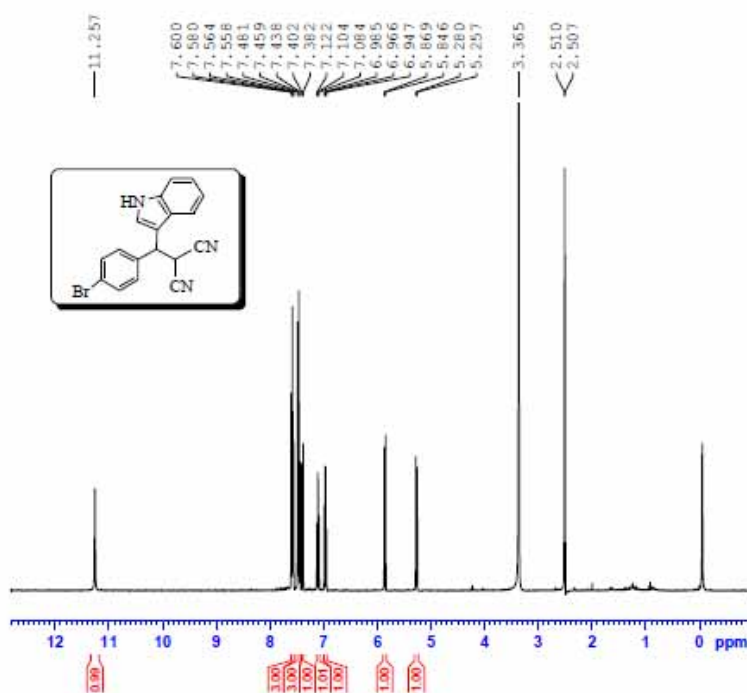
----- CHANNEL f1 -----
 NUCL1 13C
 P1 15.20 usec
 PL1 -1.00 dB
 SFO1 100.6228298 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUCL2 1H
 P2PRG 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 100.6128193 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40







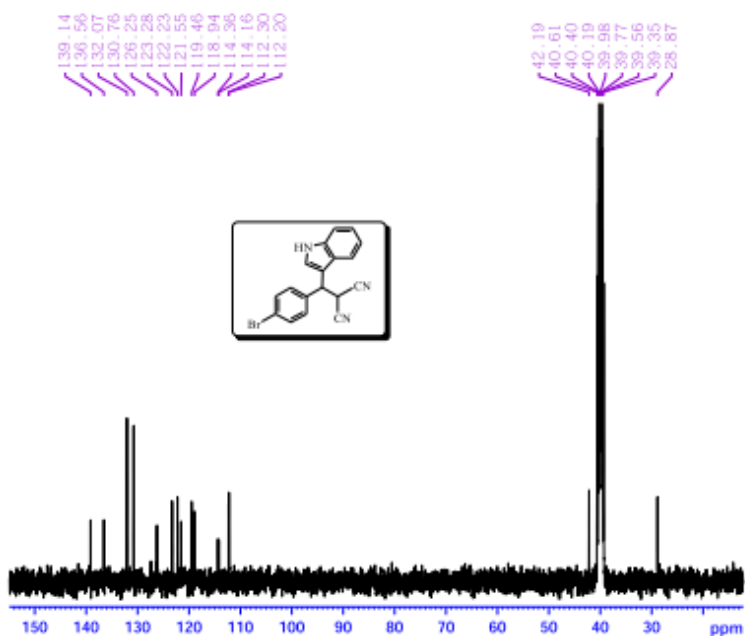
```

Current Data Parameters
NAME      28052010-test
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20100528
Time     22.38
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  DMSO
NS        8
DS        0
SWH       8222.865 Hz
FIDRES    0.125483 Hz
AQ         3.9046387 sec
RG         281
DM         60.800 usec
DE         6.50 usec
TE         300.2 K
D1         1.00000000 sec
TDO       1

===== CHANNEL f1 =====
NUC1      1H
P1        12.00 usec
PL1       -2.00 dB
SFO1     400.1214710 MHz

F2 - Processing parameters
SI        32768
SF        400.1299167 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



```

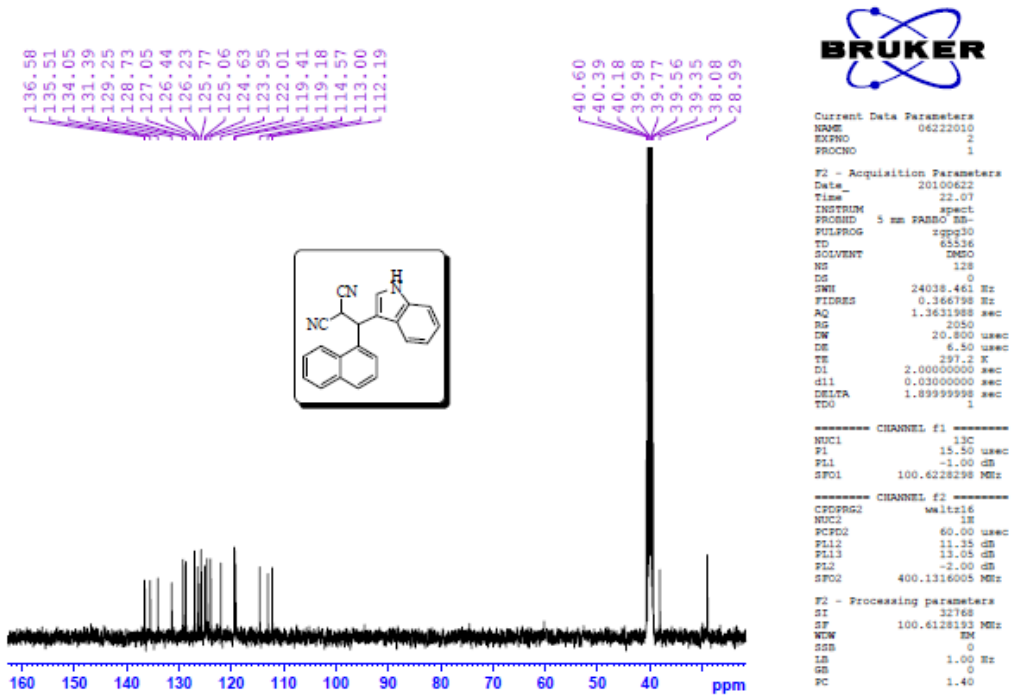
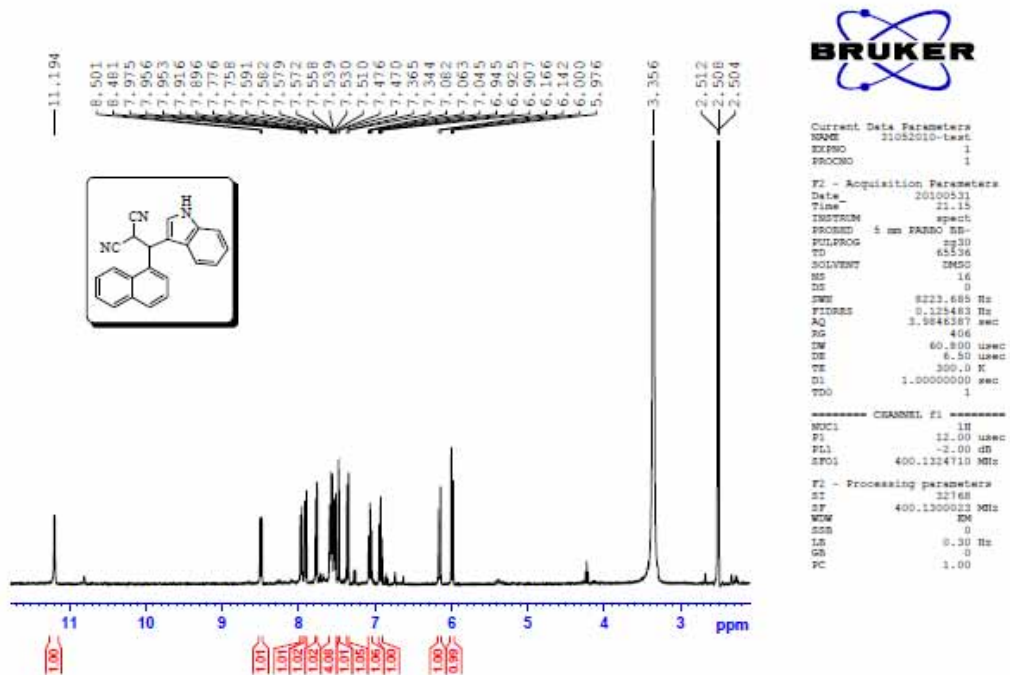
Current Data Parameters
NAME      28052010-test
EXPNO    6
PROCNO   1

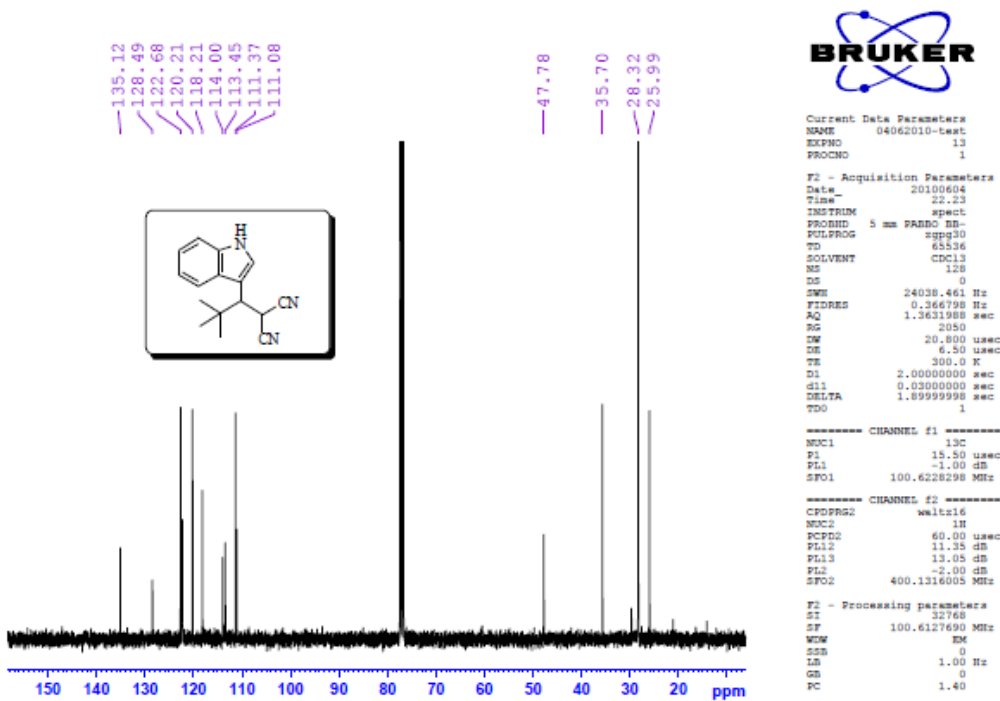
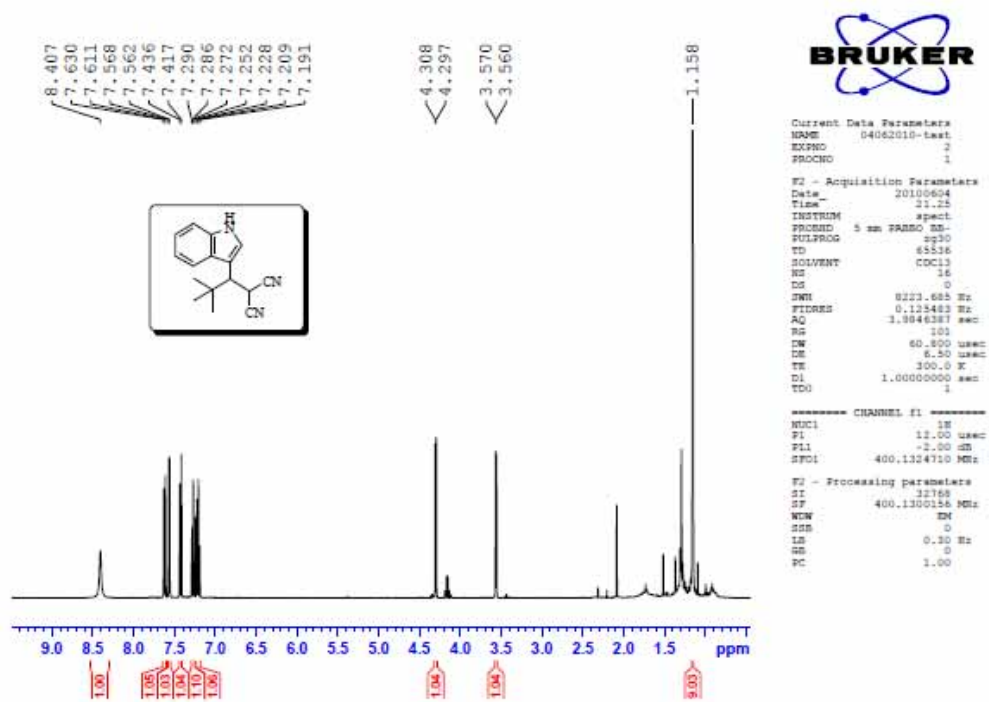
F2 - Acquisition Parameters
Date_    20100528
Time     22.47
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  DMSO
NS        128
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3831988 sec
RG         2000
DM         20.800 usec
DE         6.50 usec
TE         297.1 K
D1         2.00000000 sec
d11       0.03000000 sec
DELTA     1.89999998 sec
TDO       1

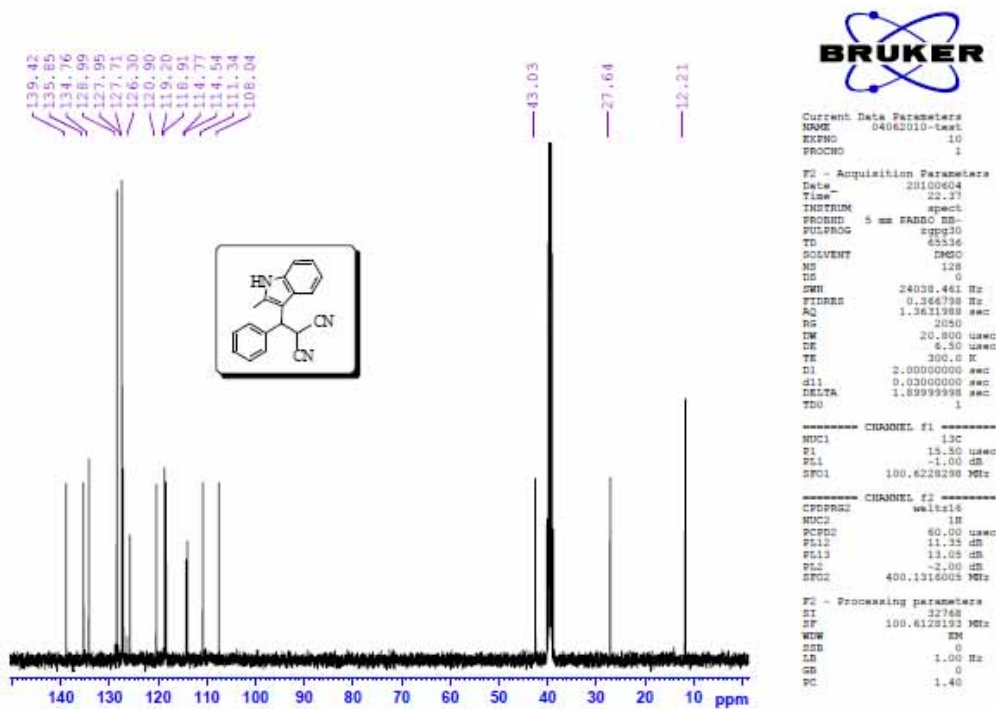
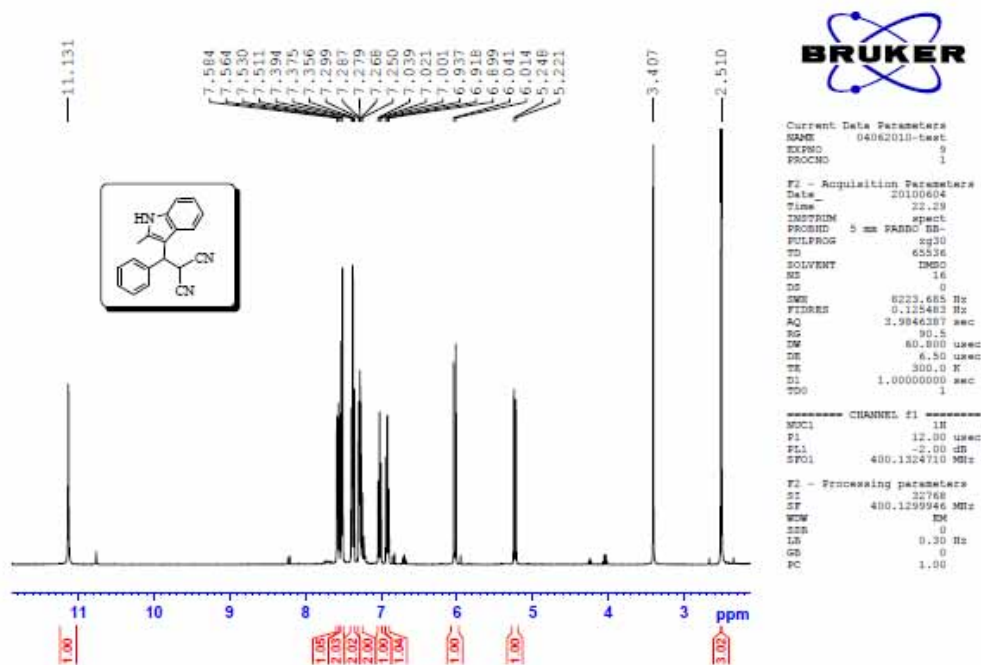
===== CHANNEL f1 =====
NUC1      13C
P1        15.50 usec
PL1       -1.00 dB
SFO1     100.6282698 MHz

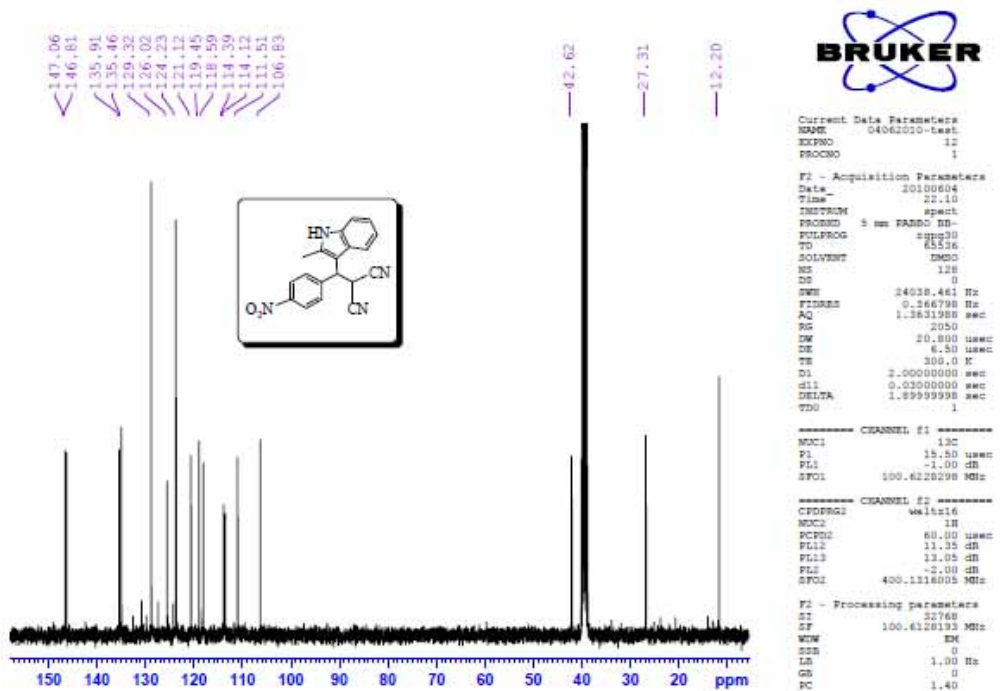
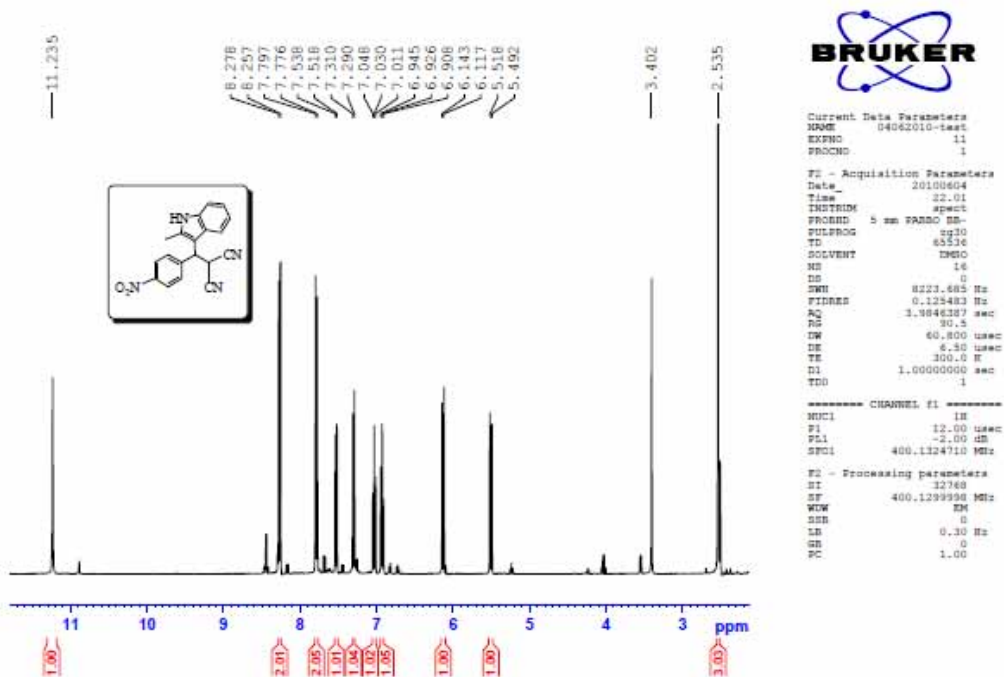
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    60.00 usec
PL12     11.35 dB
PL13     12.05 dB
PL2      -2.00 dB
SFO2     400.1316005 MHz

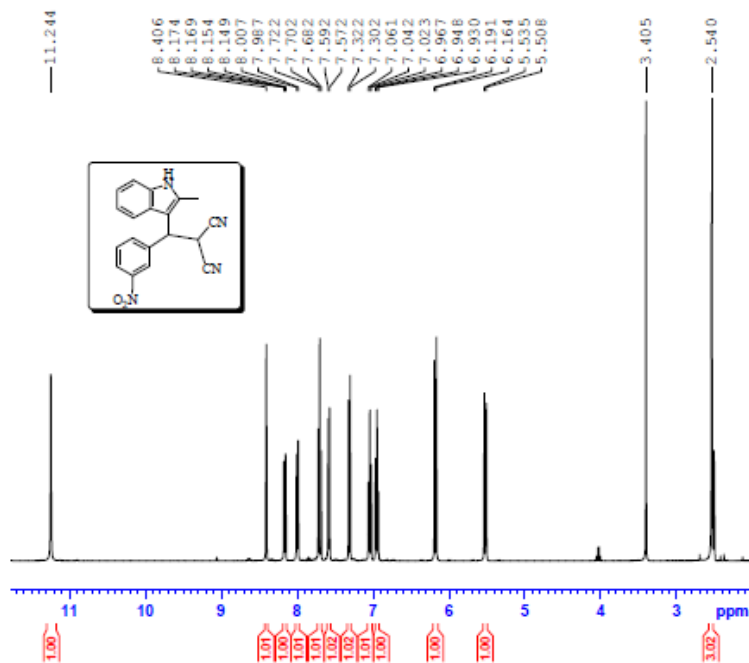
F2 - Processing parameters
SI        32768
SF        100.6128193 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```











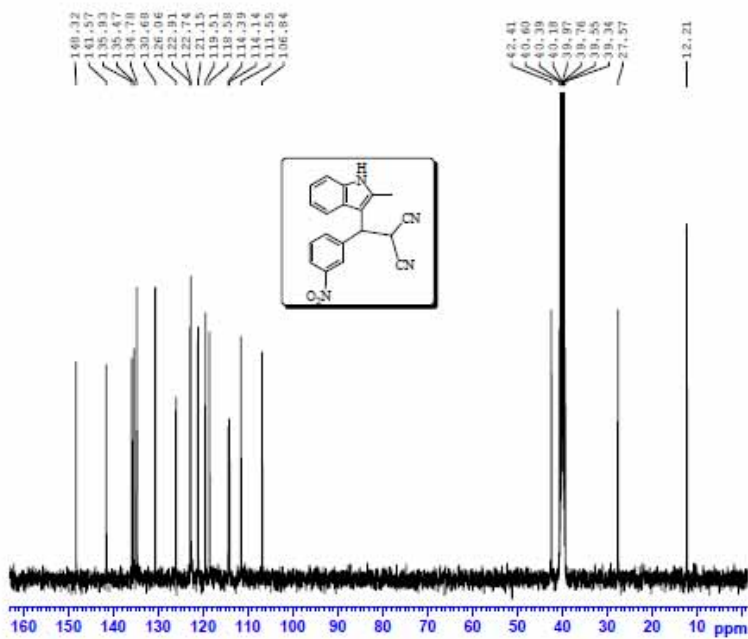
```

Current Data Parameters
NAME      06222010
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20100622
Time      22.11
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         8
DS         0
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         80.5
IN        60.800 usec
DE         6.50 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.00 usec
PL1        -2.00 dB
SFO1       400.1324110 MHz

F2 - Processing parameters
SI         32768
SF         400.1399993 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



```

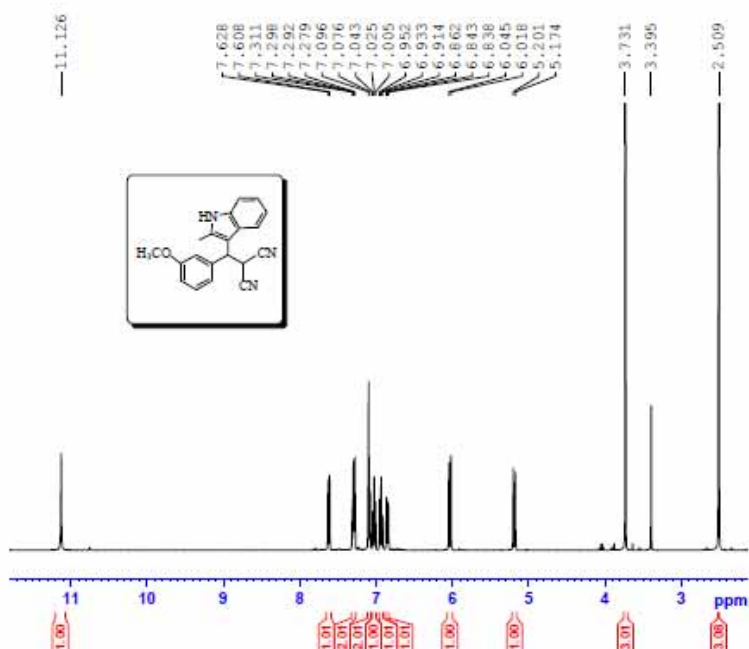
Current Data Parameters
NAME      06222010
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20100622
Time      22.20
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         128
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631888 sec
RG         2030
IN        20.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999998 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         15.20 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       400.1316003 MHz

F2 - Processing parameters
SI         32768
SF         100.6128193 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

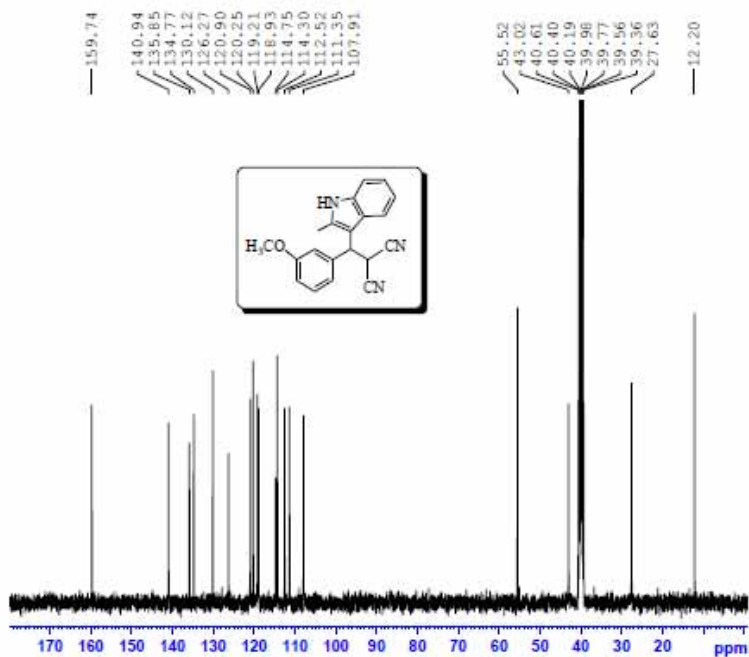


Current Data Parameters
NAME 21062010-Test
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100621
Time 21.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 0
DS 0
SWH 8223.463 Hz
FIDRES 0.123463 Hz
AQ 3.9846387 sec
RG 90.5
DM 60.800 usec
DE 6.40 usec
TE 300.0 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 400.1300071 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



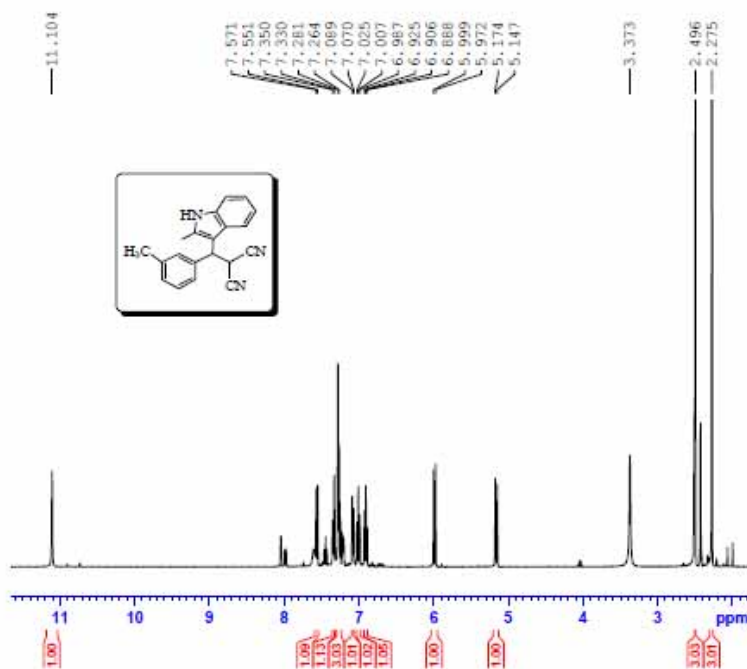
Current Data Parameters
NAME 21062010-Test
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100621
Time 21.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 128
DS 0
SWH 24028.441 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DM 20.800 usec
DE 6.50 usec
TE 300.0 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 100.6226298 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 100.6126193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

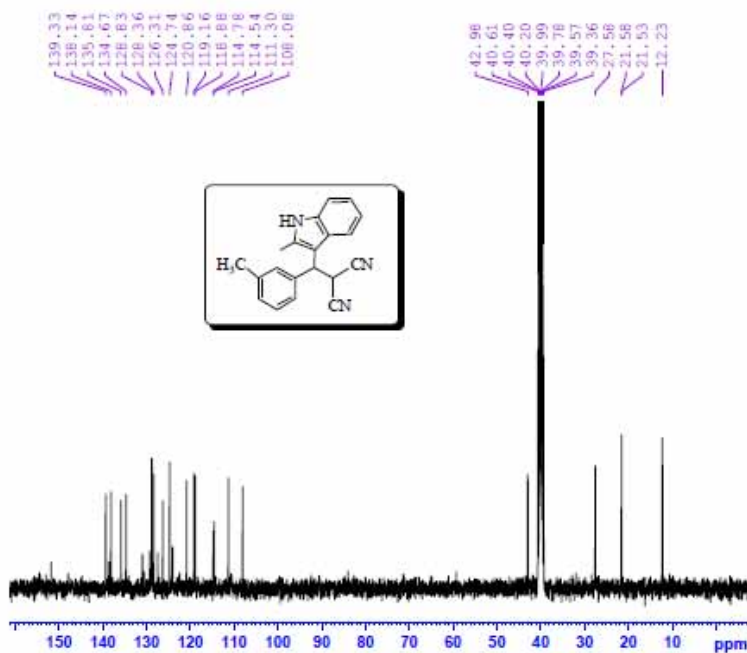


Current Data Parameters
NAME 21062010-test
EXPRO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100621
Time 22.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DM 60.000 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324110 MHz

F2 - Processing parameters
SI 32768
SF 400.1300042 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



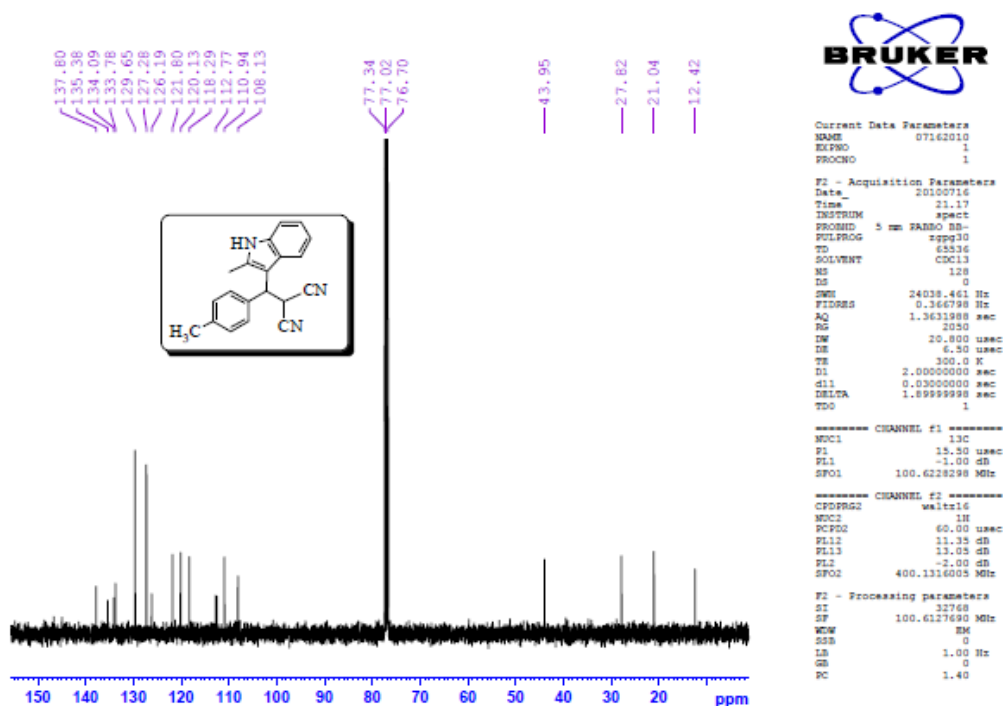
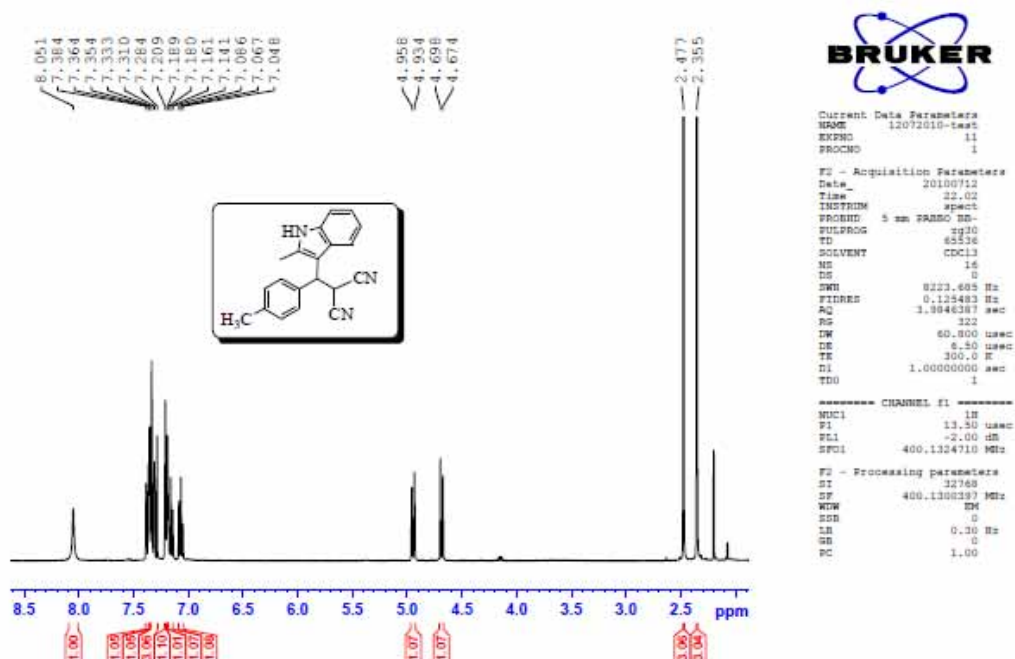
Current Data Parameters
NAME 21062010-test
EXPRO 4
PROCNO 1

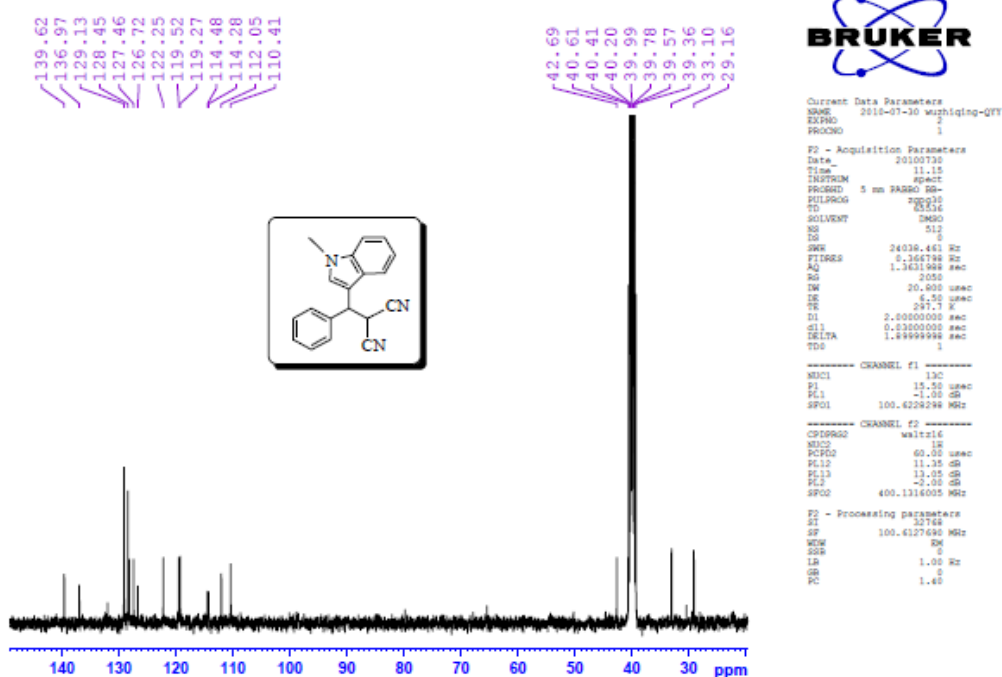
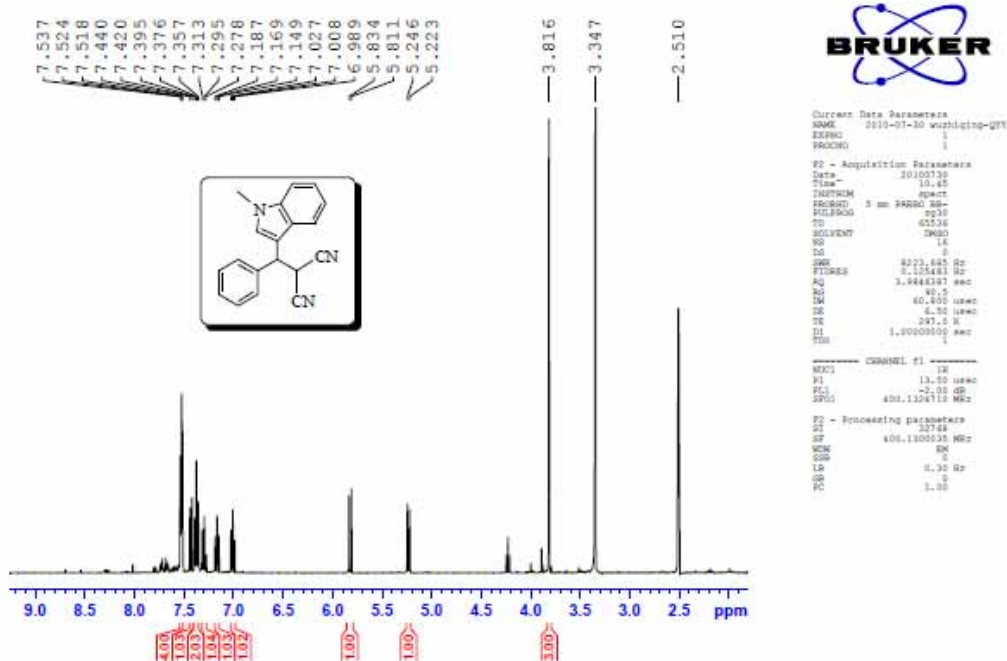
F1 - Acquisition Parameters
Date_ 20100621
Time 22.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 128
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3611988 sec
RG 2050
DM 20.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

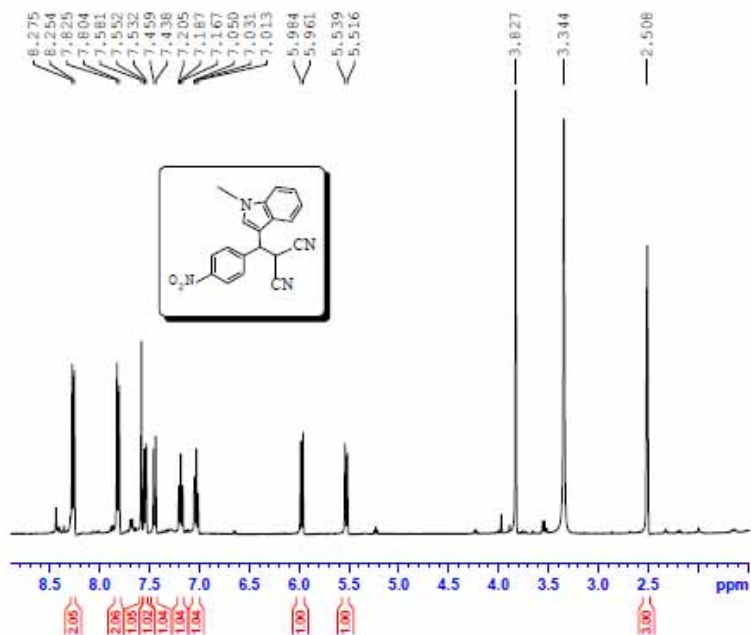
----- CHANNEL f1 -----
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

----- 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 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





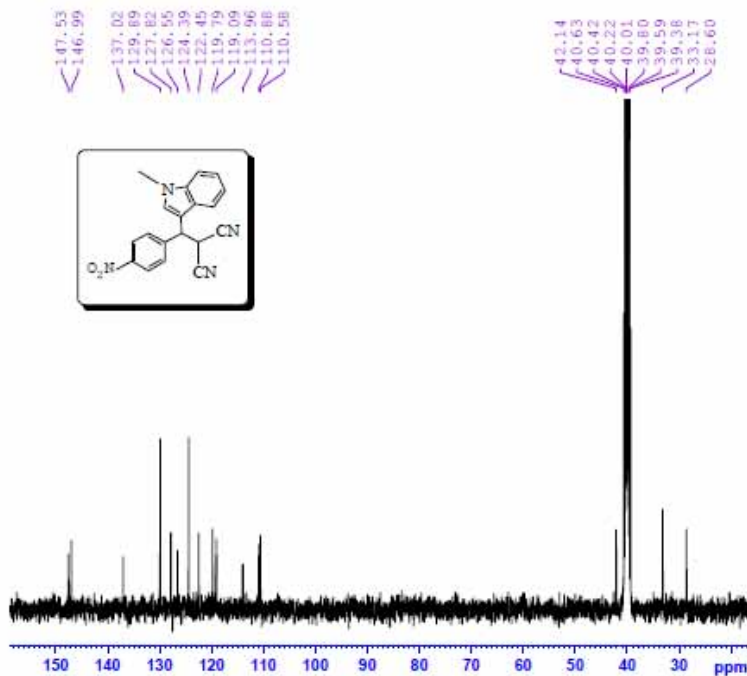


Current Data Parameters
 NAME 07082010
 EXPNO 10
 PROCNO 1

F1 - Acquisition Parameters
 Date_ 20100708
 Time 20.31
 INSTRUM spect
 PROBRD 5 mm F400 BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9246387 sec
 RG 256
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.50 usec
 PL1 -2.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300017 MHz
 WDW EM
 SS 0
 LB 0.30 Hz
 GB 0
 PC 1.00



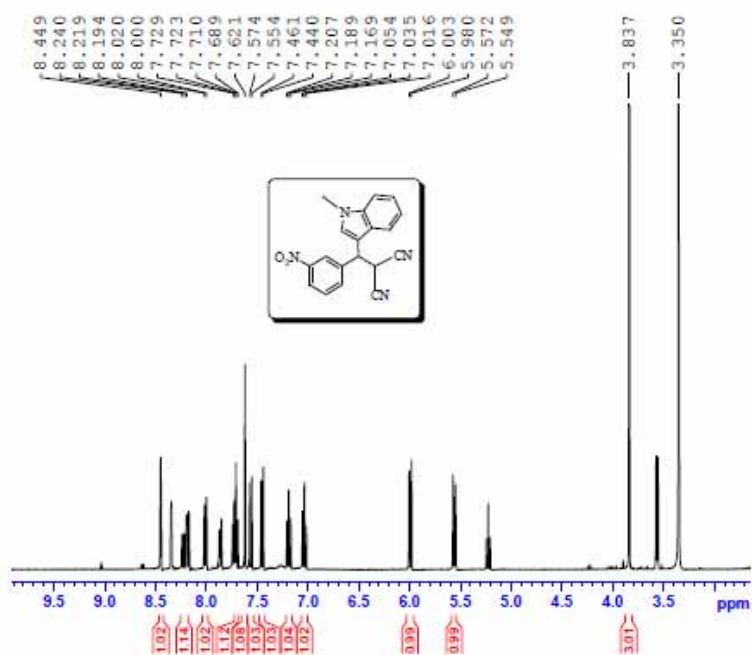
Current Data Parameters
 NAME 07082010
 EXPNO 11
 PROCNO 1

F1 - Acquisition Parameters
 Date_ 20100708
 Time 20.40
 INSTRUM spect
 PROBRD 5 mm F400 BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 128
 DS 0
 SWH 24028.481 Hz
 FIDRES 0.268798 Hz
 AQ 1.2631888 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 DELTA 1.88999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 15.50 usec
 PL1 -1.00 dB
 SFO1 100.6228238 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 60.00 usec
 PL12 11.25 dB
 PL13 13.05 dB
 PL2 -2.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6128193 MHz
 WDW EM
 SS 0
 LB 1.00 Hz
 GB 0
 PC 1.40

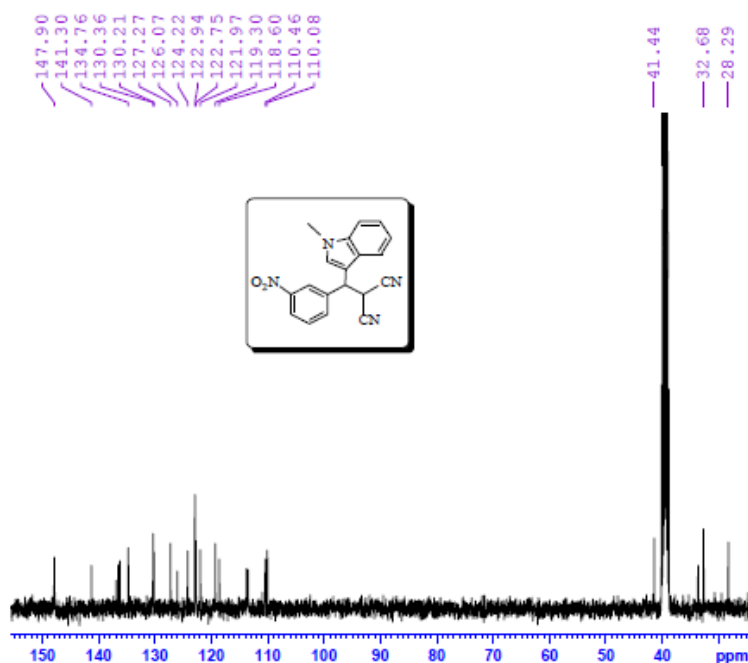


```
Current Data Parameters
NAME      07082010
EXPNO     8
PROCNO    1

F2 - Acquisition Parameters
Date_     20100708
Time      20.18
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         8
DS         0
SWH        8223.683 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         144
TW         60.808 usec
DE         8.50 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1

----- CHANNEL f1 -----
NUC1       1H
P1         13.50 usec
PL1        -2.00 dB
SFO1       400.1324710 MHz

F2 - Processing parameters
SI         32768
SF         400.1289824 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```



```
Current Data Parameters
NAME      07082010
EXPNO     9
PROCNO    1

F2 - Acquisition Parameters
Date_     20100708
Time      20.26
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         128
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3651988 sec
RG         2050
TW         20.800 usec
DE         6.50 usec
TE         300.0 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         100.6128193 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
```