

Electronic Supplementary Information

Design, synthesis and antiproliferative activity of indole analogues of indanocine

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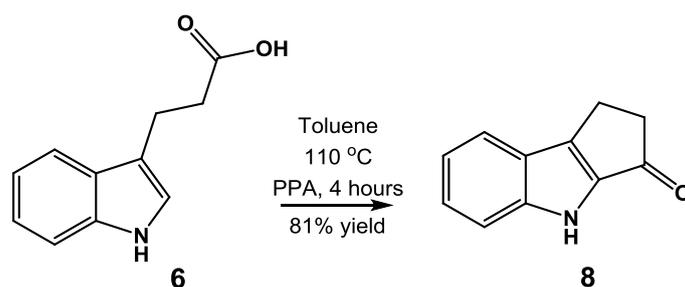
General Experimental

Chemicals, solvents and reagents used are commercially available and were used without further purification.

TLCs were carried out on Merck Aluminium backed TLC plates Silica Gel 60 F254 and viewed using UV light of wavelength 254 nm and then stained with potassium permanganate or 2,4-dinitrophenylhydrazine (DNP). Merck Silica Gel (0.040-0.063 mm) was used for column chromatography. Compounds were loaded as an oil, CHCl₃ solution or dry loaded by adsorption onto silica. Melting points were obtained using a Reichert-Jung heated-stage microscope. Infrared spectra were recorded on a Perkin-Elmer Spectrum RXI FT-IR system and all values are recorded in cm⁻¹.

NMR spectra were obtained on a Bruker Avance III (400 MHz) spectrometer. The chemical shifts are recorded in parts per million (ppm) with reference to tetramethylsilane. The coupling constants *J* are quoted to the nearest 0.5 Hz and are not corrected. The multiplicities are assigned as a singlet (s), doublet (d), triplet (t), doublet of doublets (dd), quartet (q) and multiplet (m). The symbols + and – after the carbon NMR chemical shifts indicate odd (CH and CH₃) and even (C and CH₂) numbers of attached protons respectively. Mass spectra and high resolution mass spectra were obtained on a micrOTOFTM from Bruker Daltonics (Bremen, Germany) coupled with an electrospray source (ESI-TOF) using an autosampler in an Agilent 1100 LC system. Data was processed using external calibration with the Bruker Daltonics software, DataAnalysisTM as part of the overall hardware control software, Compass 1.1TM.

1,4-Dihydrocyclopenta[*b*]indol-3(2*H*)-one **8**

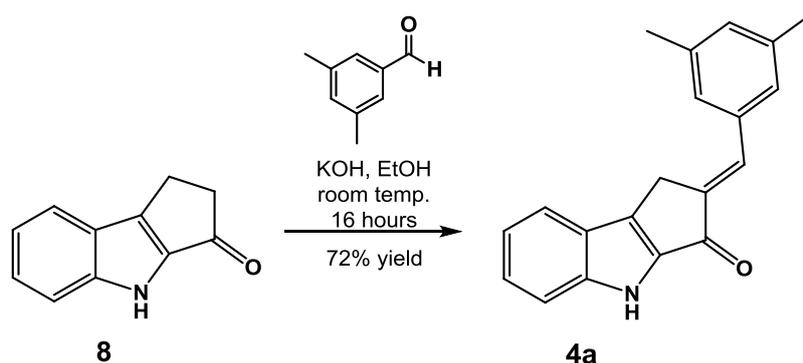


Following a previously reported procedure,¹ 3-(indole-3-yl)propanoic acid **6** (378 mg, 2 mmol, 1 eq.) was added to PPA (3.5 g, 35.7 mmol, 17.9 eq.) in toluene (20 mL) and the mixture was stirred at 110 °C for 4 hours. On cooling, ice water (80 mL) was added and the purple aqueous layer was extracted using CH₂Cl₂ (3 x 50 mL). The combined organic layers were washed with brine (30 mL), dried on Na₂SO₄, filtered and concentrated under reduced pressure to afford, without further purification, the desired compound **8** (278 mg, 81 % yield) as a beige solid.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.6. **IR** ν_{max} (liquid film): 3466 (NH), 3031 (CH) and 1682 (C=O). **m.p.** 255-258 °C [lit.² 250-252 °C]. **¹H NMR** (400 MHz; CDCl₃): δ_{H} 8.98 (1H, br. s. NH), 7.75 (1H, dd, $J = 8.0, 1.0$ Hz, ArCH), 7.52 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.44 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.23 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 3.17-3.14 (2H, m, COCH₂CH₂) and 3.08-3.05 (2H, m, COCH₂CH₂). **¹³C NMR** (100 MHz; CDCl₃): δ_{C} 194.5- (C=O), 147.2- (C), 143.8- (C), 137.4- (C), 127.4+ (ArCH), 123.6- (C), 121.6+ (ArCH), 120.8+ (ArCH), 113.5+ (ArCH), 41.0- (COCH₂CH₂) and 20.1- (COCH₂CH₂). **MS** m/z (+ESI) 172 (82 %, MH⁺) and 194 (100 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 172.0755, C₁₁H₁₀NO requires MH 172.0762 and found MNa⁺ 194.0572, C₁₁H₉NNaO requires MNa 194.0582.

Consistent with the spectroscopic data previously reported for this compound.³

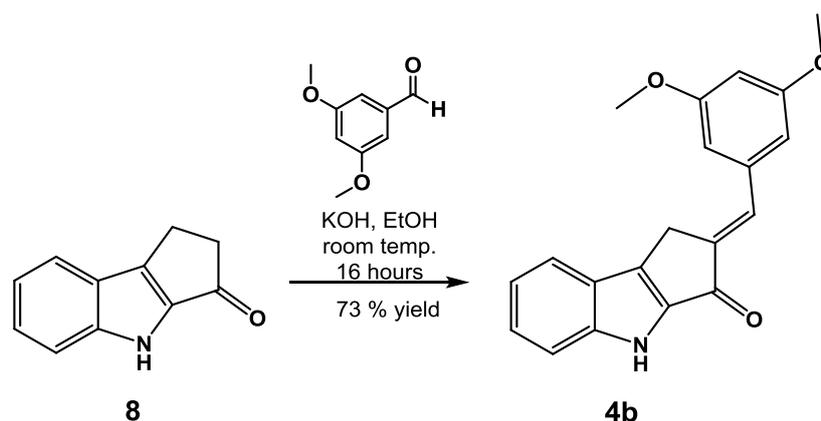
(E)-2-(3,5-Dimethylbenzylidene)-1,4-dihydrocyclopenta[*b*]indol-3(2*H*)-one 4a



A mixture of ketone **8** (171 mg, 1 mmol, 1 eq.) and 3,5-dimethylbenzaldehyde (0.15 mL, 1.1 mmol, 1.1 eq.) were treated with 4 % (w/v) ethanolic KOH (10 mL) and stirred at room temperature for 16 hours.⁴ The mixture was cooled in an ice-bath and the solid was filtered and washed with EtOH: H₂O (10 mL: 10 mL). H₂O (20 mL) was added to the filtrate which was then neutralised using AcOH, followed by a second filtration. The solid was again washed with EtOH: H₂O (10 mL: 10 mL) to afford compound **4a** (206 mg, 72 % yield) as a yellow solid.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.5. **IR** ν_{\max} (liquid film): 3464 (NH), 3028 (CH), 1678 (C=O) and 1627 (C=C). **m.p.** 288-292 °C. **¹H NMR** (400 MHz; (CD₃)₂SO): δ_{H} 11.90 (1H, br.s, NH), 7.80 (1H, d, $J = 8.0$ Hz, ArCH), 7.45 (1H, d, $J = 8.5$ Hz, ArCH), 7.37-7.33 (3H, m, ArCH and Ar'CH), 7.29 (1H, br.s, alkene CH), 7.14 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.05 (1H, s, Ar'CH), 4.04 (2H, s, CH₂) and 2.33 (6H, s, Ar'CH₃). **¹³C NMR** (100 MHz; (CD₃)₂SO): δ_{C} 181.8- (CO), 143.5- (C), 140.5- (C), 139.9- (C), 139.6- (C), 138.0- (C), 134.9- (C), 130.8+ (alkene CH), 130.8+ (Ar'CH), 128.1+ (ArCH or Ar'CH), 126.8+ (Ar'CH or ArCH), 122.7- (C), 121.7+ (ArCH), 120.3+ (ArCH), 113.6+ (ArCH), 26.3- (CH₂) and 20.9+ (Ar'CH₃). **MS** m/z (+ESI) 288 (100 %, MH⁺) and 310 (14 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 288.1369, C₂₀H₁₈NO requires MH 288.1388 and found MNa⁺ 310.1190, C₂₀H₁₇NNaO requires MNa 310.1208. **Analysis** (Found: H, 5.92; N, 4.86. C₂₀H₁₇NO requires H, 5.96; N, 4.87 %).

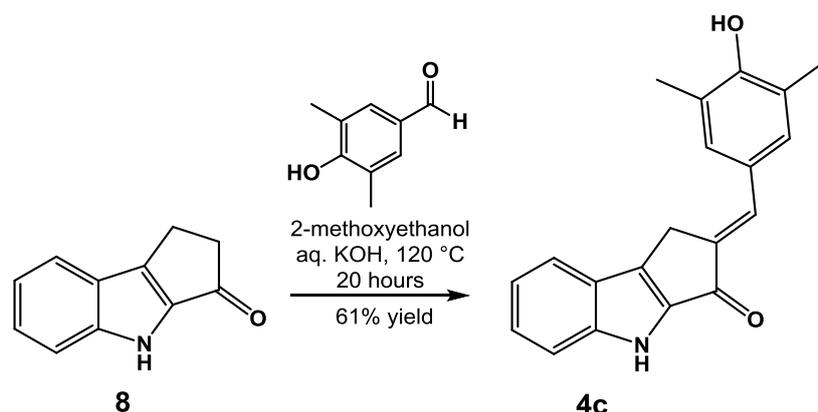
(E)-2-(3,5-Dimethoxybenzylidene)-1,4-dihydrocyclopenta[*b*]indol-3(2*H*)-one 4b



A mixture of ketone **8** (171 mg, 1 mmol, 1eq.) and 3,5-dimethoxybenzaldehyde (183 mg, 1.1 mmol, 1.1 eq.) were treated with 4 % (w/v) ethanolic KOH (10 mL) and stirred at room temperature for 16 hours.⁴ The mixture was cooled in an ice-bath and the solid was filtered and washed with EtOH: H₂O (10 mL: 10 mL). H₂O (15 mL) was added to the filtrate which was then neutralised using AcOH, followed by a second filtration. The solid was again washed with EtOH: H₂O (10 mL: 10 mL) to afford compound **4b** (232 mg, 73 % yield) as a yellow solid.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.3. **IR** ν_{\max} (liquid film): 3464 (NH), 3026 (CH), 1674 (C=O) and 1624 (C=C). **m.p.** >220 °C. **¹H NMR** (400 MHz; (CD₃)₂SO): δ_{H} 11.86 (1H, br.s, NH), 7.80 (1H, d, $J = 8.0$ Hz, ArCH), 7.45 (1H, d, $J = 8.5$ Hz, ArCH), 7.36 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.30 (1H, m, alkene CH), 7.14 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 6.90 (2H, d, $J = 2.0$ Hz, Ar'CH), 6.57 (1H, t, $J = 2.0$ Hz, Ar'CH), 4.03 (2H, d, $J = 1.5$ Hz, CH₂) and 3.81 (6H, s, Ar'OCH₃). **¹³C NMR** (100 MHz; (CD₃)₂SO): δ_{C} 181.7- (CO), 160.7- (C), 143.5- (C), 140.7- (C), 140.4- (C), 139.8- (C), 136.8- (C), 130.6+ (alkene CH), 126.9+ (ArCH), 122.6- (C), 121.8+ (ArCH), 120.3+ (ArCH), 113.6+ (ArCH), 108.3+ (Ar'CH), 101.3+ (Ar'CH), 55.4+ (Ar'OCH₃) and 26.2- (CH₂). **MS** m/z (+ESI) 320 (100 %, MH⁺) and 342 (9 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 320.1281, C₂₀H₁₈NO₃ requires *MH* 320.1287.

(E)-2-(4-Hydroxy-3,5-dimethylbenzylidene)-1,4-dihydrocyclopenta[*b*]indol-3(2*H*)-one 4c

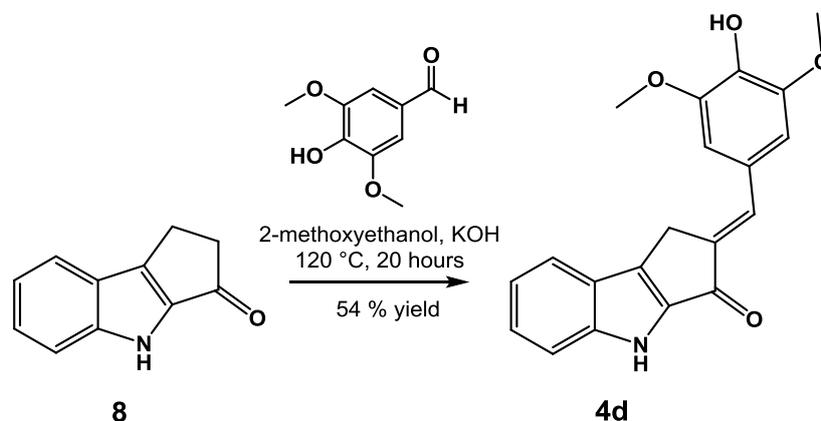


Ketone **8** (128 mg, 0.75 mmol, 1 eq.) and 3,5-dimethyl-4-hydroxybenzaldehyde (123 mg, 0.82 mmol, 1.1 eq.) were dissolved in 2-methoxyethanol (1 mL), followed by the addition of 1 % aqueous KOH solution (0.5 mL). The reaction mixture was heated at 120 °C for 20 hours. The mixture was cooled in an ice bath and the solid was filtered and washed with EtOH: H₂O (10 mL: 10 mL). H₂O (15 mL) was added to the filtrate which was then neutralised using AcOH, followed by a second filtration. The solid was again washed with EtOH: H₂O (10mL: 10mL) to afford compound **4c** (139 mg, 61 % yield) as a yellow solid.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.2. **IR** ν_{\max} (liquid film): 3605 (OH), 3463 (NH), 3036 (CH), 1677 (C=O) and 1601 (C=C). **m.p.** > 330 °C. **¹H NMR** (400 MHz; (CD₃)₂SO): δ_{H} 11.80 (1H, br.s, NH), 8.78 (1H, br.s, OH), 7.79 (1H, d, *J* = 8.0 Hz, ArCH), 7.44 (1H, d, *J* = 8.5 Hz, ArCH), 7.35-7.32 (3H, m, Ar'CH and ArCH), 7.22 (1H, br.s, alkene CH), 7.13 (1H, ddd, *J* = 8.0, 7.0, 1.0 Hz, ArCH), 3.97 (2H, s, CH₂) and 2.22 (6H, s, Ar'CH₃). **¹³C NMR** (100 MHz; (CD₃)₂SO): δ_{C} 182.1- (CO), 155.0- (C), 143.3- (C), 140.8- (C), 138.8- (C), 136.7- (C), 131.2+ (alkene CH), 131.0+ (Ar'CH), 126.5+ (ArCH), 126.1- (C), 124.7- (C), 121.5+ (ArCH), 120.2+ (ArCH), 118.0- (C), 113.6+ (ArCH), 26.3- (CH₂) and 16.6+ (Ar'CH₃). **MS** *m/z* (+ESI) 304 (100 %, MH⁺) and 326 (11 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 304.1334, C₂₀H₁₈NO₂ requires *MH* 304.1338 and found MNa⁺ 326.1170, C₂₀H₁₇NNaO₂ requires *MNa* 326.1157.

(E)-2-(4-Hydroxy-3,5-dimethoxybenzylidene)-1,4-dihydrocyclopenta[*b*]indol-3(2*H*)-one

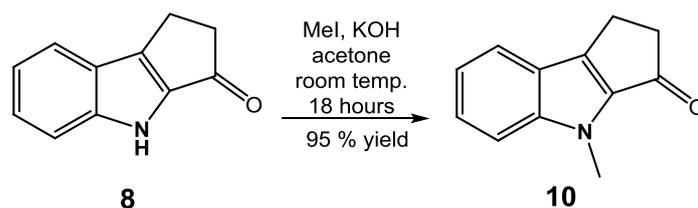
4d



Ketone **8** (171 mg, 1 mmol, 1 eq.) and 3,5-dimethoxy-4-hydroxybenzaldehyde (200 mg, 1.1 mmol, 1.1 eq.) were dissolved in 2-methoxyethanol (1 mL), followed by the addition of 1 % aqueous KOH solution (0.5 mL). The reaction mixture was heated at 120 °C for 20 hours. The mixture was cooled in an ice-bath and the solid was filtered and washed with EtOH: H₂O (10 mL: 10 mL). H₂O (15 mL) was added to the filtrate which was then neutralised using AcOH, followed by a second filtration. The solid was again washed with EtOH: H₂O (10 mL: 10 mL) to afford compound **4d** (182 mg, 54 % yield) as a yellow solid.

R_f (70 % EtOAc in light petroleum (b.p. 40-60 °C) 0.6. **IR** ν_{\max} (liquid film): 3529 (OH), 3011 (CH), 1673 (C=O) and 1624 (C=C). **m.p.** 237-239 °C. **¹H NMR** (400 MHz; (CD₃)₂SO): δ_{H} 11.86 (1H, br.s. NH), 8.94 (1H, s, OH), 7.80 (1H, d, *J* = 8.0 Hz, ArCH), 7.55 (1H, d, *J* = 8.5 Hz, ArCH), 7.40 (1H, ddd, *J* = 8.0, 7.0, 1.0 Hz, ArCH), 7.29 (1H, br.s, alkene CH), 7.17 (1H, ddd, *J* = 8.0, 7.0, 1.0 Hz, ArCH), 7.02 (2H, s, Ar'CH), 4.03 (2H, s, CH₂) and 3.85 (6H, s, Ar'OCH₃). **¹³C NMR** (100 MHz; (CD₃)₂SO): δ_{C} 182.0- (C=O), 148.1- (C), 143.3- (C), 140.7- (C), 139.0- (C), 137.7- (C), 137.3- (C), 131.6+ (alkene CH), 126.6+ (ArCH), 125.4- (C), 122.7- (C), 121.6+ (ArCH), 120.2+ (ArCH), 113.6+ (ArCH), 108.5+ (Ar'CH), 56.2+ (Ar'OCH₃) and 26.1- (CH₂). **MS** *m/z* (+ESI) 336 (100 %, MH⁺) and 358 (12 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 336.1228, C₂₀H₁₈NO₄ requires *MH* 336.1236 and found MNa⁺ 358.1050, C₂₀H₁₇NNaO₄ requires *MNa* 358.1055.

4-Methyl-1,4-dihydrocyclopenta[*b*]indol-3(2*H*)-one **10**

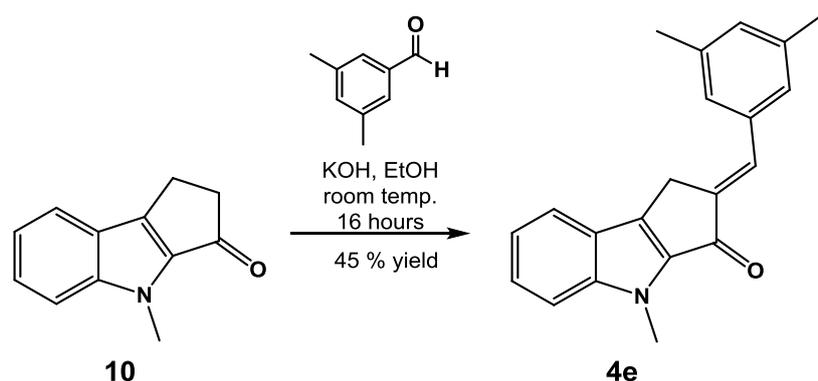


Following the procedure previously reported,⁵ MeI (2.4 eq.) was added to a rapidly stirred solution of the indole **8** (6.37 mmol) and KOH (2.9 eq.) in acetone (10 mL/ 1 mmol). After 18 hours at room temperature, the solvent was removed under reduced pressure and H₂O added and acidified to pH 1 with 6M HCl_(aq.). The aqueous layer was extracted three times with CH₂Cl₂ and the combined organic layers washed with brine, dried with Na₂SO₄, filtered and concentrated under reduced pressure to afford, without the need for further purification, the methylated product **10** (1.12 g, 95 % yield) as a beige solid.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.4. **m.p.** 138-142 °C [lit.⁶ 135.1-136.1 °C]. **IR** ν_{\max} (liquid film): 3033 (CH), 1681 (C=O). **¹H NMR** (400 MHz; CDCl₃): δ_{H} 7.69 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.41 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.36 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.17 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 3.91 (3H, s, NCH₃), 3.07-3.05 (2H, m, COCH₂CH₂) and 2.99-2.97 (2H, m, COCH₂CH₂). **¹³C NMR** (100 MHz; CDCl₃): δ_{C} 194.8- (C=O), 145.0- (C), 144.8- (C), 138.9- (C), 126.8+ (ArCH), 123.1- (C), 121.7+ (ArCH), 120.2+ (ArCH), 110.9+ (ArCH), 41.5- (COCH₂CH₂), 30.0+ (NCH₃) and 19.6- (COCH₂CH₂). **MS** m/z (+ESI) 186 (100 %, MH⁺). **HRMS** (+ESI) Found MH⁺ 186.0914, C₁₂H₁₂NO requires MH 186.0919 and found MNa⁺ 208.0736, C₁₂H₁₁NNaO requires MNa 208.0738.

Consistent with the spectroscopic data previously reported for this compound.^{1,6}

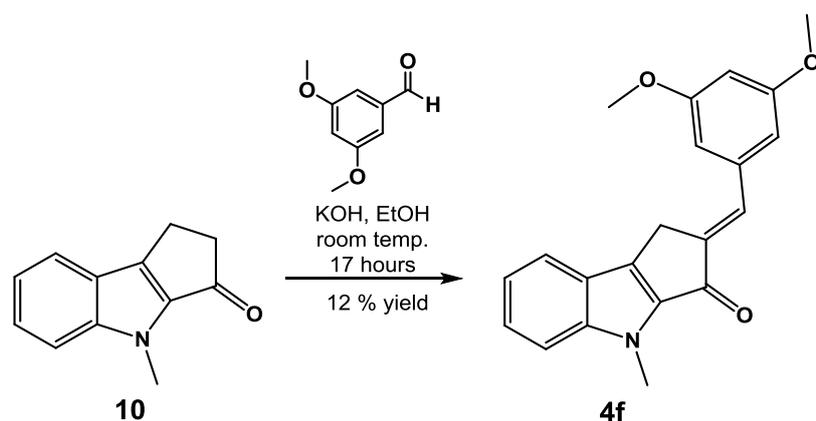
(E)-2-(3,5-Dimethylbenzylidene)-4-methyl-1,4-dihydrocyclopenta[*b*]indol-3(2*H*)-one 4e



A mixture of ketone **10** (185 mg, 1 mmol, 1 eq.) and 3,5-dimethylbenzaldehyde (0.15 mL, 1.1 mmol, 1.1 eq.) were treated with 4 % (w/v) ethanolic KOH (10 mL) and stirred at room temperature for 16 hours.⁴ The mixture was cooled in an ice-bath and the solid was filtered and washed with EtOH: H₂O (10 mL: 10 mL). H₂O (20 mL) was added to the filtrate which was then neutralised using AcOH, followed by a second filtration. The solid was again washed with EtOH: H₂O (10 mL: 10 mL) to afford compound **4e** (135 mg, 45 % yield) as a yellow solid.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.8. **IR** ν_{\max} (liquid film): 3029 (CH), 1678 (C=O) and 1628 (C=C). **m.p.** 174-177 °C. **¹H NMR** (400 MHz; CDCl₃): δ_{H} 7.76 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.47 (1H, t, $J = 1.5$ Hz, alkene CH), 7.42 (1H, ddd, $J = 8.5, 6.5, 1.0$ Hz, ArCH), 7.38 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.28 (2H, s, Ar'CH), 7.20 (1H, ddd, $J = 8.0, 6.5, 1.0$ Hz, ArCH), 7.02 (1H, s, Ar'CH), 4.00 (3H, s, NCH₃), 3.95 (2H, d, $J = 1.5$ Hz, CH₂) and 2.39 (6H, s, Ar'CH₃). **¹³C NMR** (100 MHz; CDCl₃): δ_{C} 183.4- (C=O), 144.6- (C), 140.9- (C), 139.8- (C), 138.6- (C), 138.3- (C), 135.5- (C), 132.0+ (alkene CH), 131.0+ (Ar'CH), 128.3+ (Ar'CH), 126.8+ (ArCH), 123.0- (C), 121.9+ (ArCH), 120.5+ (ArCH), 111.0+ (ArCH), 30.3+ (NCH₃), 26.5- (CH₂) and 21.4+ (Ar'CH₃). **MS** m/z (+ESI) 302 (100 %, MH⁺). **HRMS** (+ESI) Found MH⁺ 302.1535, C₂₁H₂₀NO requires MH 302.1545 and found MNa⁺ 324.1353, C₂₁H₁₉NNaO requires MNa 324.1364. **Analysis** (Found: H, 6.47; N, 4.29. C₂₁H₁₉NO requires H, 6.35; N, 4.65 %).

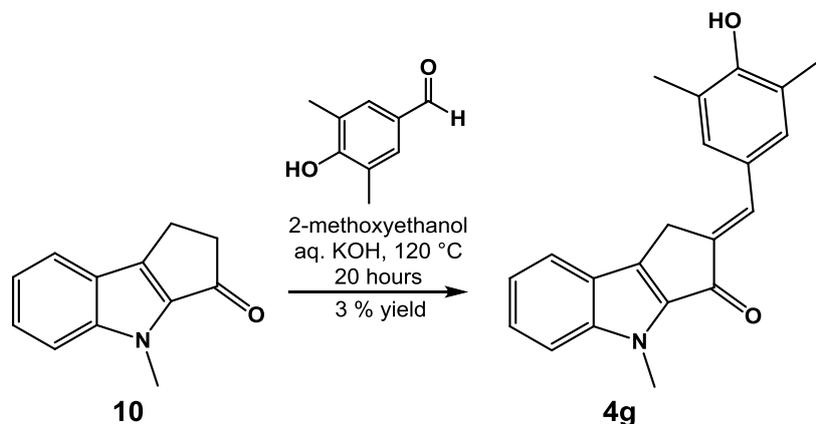
(E)-2-(3,5-Dimethoxybenzylidene)-4-methyl-1,4-dihydrocyclopenta[*b*]indol-3(2*H*)-one 4f



A mixture of ketone **10** (185 mg, 1 mmol, 1 eq.) and 3,5-dimethoxybenzaldehyde (183 mg, 1.1 mmol, 1.1 eq.) were treated with 4 % (w/v) ethanolic KOH (10 mL) and stirred at room temperature for 17 hours.⁴ The mixture was cooled in an ice-bath and the solid was filtered and washed with EtOH: H₂O (10 mL: 10 mL). H₂O (15 mL) was added to the filtrate which was then neutralised using AcOH, followed by a second filtration. The solid was again washed with EtOH: H₂O (10 mL: 10 mL) to collect 301 mg of crude product. Column chromatography [silica, light petroleum (b.p. 40-60 °C) - EtOAc gradient column], gave the product **4f** (40 mg, 12 % yield) as a yellow solid.

R_f (40 % EtOAc in light petroleum (b.p. 40-60 °C) 0.4. **IR** ν_{\max} (liquid film): 3014 (CH), 1677 (C=O) and 1592 (C=C). **m.p.** 178-180 °C. **¹H NMR** (400 MHz; CDCl₃): δ_{H} 7.73 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.44-7.37 (3H, m, ArCH and alkene CH), 7.20 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 6.81 (2H, d, $J = 2.0$ Hz, Ar'CH), 6.51 (1H, t, $J = 2.0$ Hz, Ar'CH), 4.01 (3H, s, NCH₃), 3.97 (2H, d, $J = 1.5$ Hz, CH₂) and 3.86 (6H, s, Ar'OCH₃). **¹³C NMR** (100 MHz; CDCl₃): δ_{C} 183.1- (C=O), 161.0- (C), 144.7- (C), 140.8- (C), 140.7- (C), 138.7- (C), 137.3- (C), 131.6+ (alkene CH), 127.0+ (ArCH), 122.9- (C), 121.9+ (ArCH), 120.6+ (ArCH), 111.0+ (ArCH), 108.5+ (Ar'CH), 101.2+ (Ar'CH), 55.5+ (Ar'OCH₃), 30.3+ (NCH₃) and 26.4- (CH₂). **MS** m/z (+ESI) 334 (100 %, MH⁺) and 356 (8 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 334.1445, C₂₁H₂₀NO₃ requires MH 334.1443 and found MNa⁺ 356.1270, C₂₁H₁₉NNaO₃ requires MNa 356.1263.

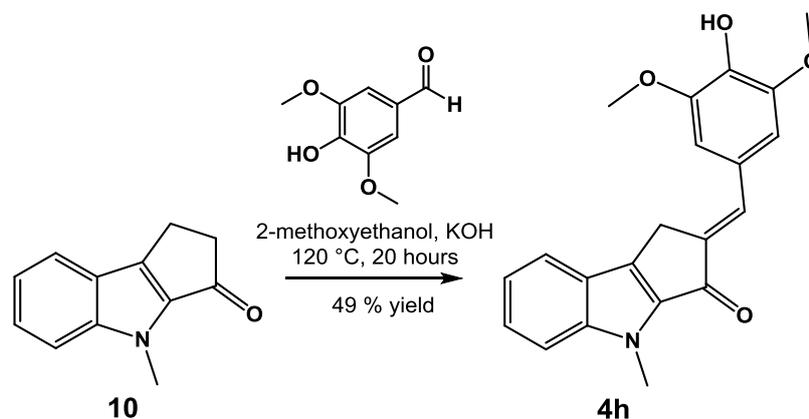
(E)-2-(4-Hydroxy-3,5-dimethylbenzylidene)-4-methyl-1,4-dihydrocyclopenta[*b*]indol-3(2*H*)-one **4g**



Ketone **10** (226 mg, 1.22 mmol, 1 eq.) and 3,5-dimethyl-4-hydroxybenzaldehyde (201 mg, 1.34 mmol, 1.1 eq.) were dissolved in 2-methoxyethanol (1.2 mL), followed by the addition of 1 % aqueous KOH solution (0.6 mL). The reaction mixture was heated at 120 °C for 20 hours. The mixture was cooled, H₂O (10 mL) was added and the mixture was neutralised using AcOH. The product was extracted using CHCl₃ (3 x 20 mL), the combined organic layers were washed with brine (20 mL), dried on Na₂SO₄, filtered and concentrated under reduced pressure. Column chromatography [silica, light petroleum (b.p. 40-60 °C) - EtOAc gradient column], gave the product **4g** (13 mg, 3 % yield) as yellow crystals.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C)) 0.5. **IR** ν_{\max} (liquid film): 3387 (OH), 3022 (CH), 1671 (C=O) and 1600 (C=C). **m.p.** 221-224 °C. **¹H NMR** (400 MHz; CDCl₃): δ_{H} 7.77 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.42-7.40 (3H, m, ArCH and alkene CH), 7.34 (2H, s, Ar'CH), 7.20 (1H, ddd, $J = 8.0, 6.5, 1.0$ Hz, ArCH), 4.86 (1H, s, OH), 4.02 (3H, s, NCH₃), 3.95 (2H, d, $J = 1.5$ Hz, CH₂) and 2.32 (6H, s, Ar'CH₃). **MS** m/z (+ESI) 318 (100 %, MH⁺) and 340 (34 %, MNa⁺). **HRMS** Found MH⁺ 318.1499, C₂₁H₂₀NO₂ requires MH 318.1494.

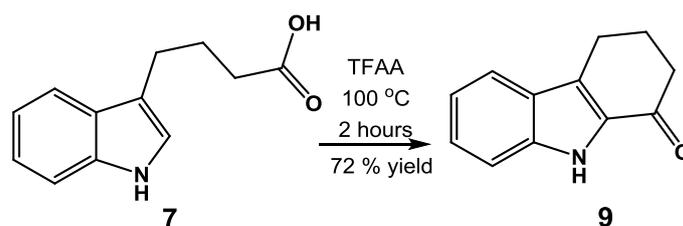
(E)-2-(4-Hydroxy-3,5-dimethoxybenzylidene)-4-methyl-1,4-dihydrocyclopenta[b]indol-3(2H)-one 4h



Ketone **10** (185 mg, 1 mmol, 1 eq.) and 3,5-dimethoxy-4-hydroxybenzaldehyde (200 mg, 1.1 mmol, 1.1 eq.) were dissolved in 2-methoxyethanol (1 mL), followed by the addition of 1 % aqueous KOH solution (0.5 mL). The reaction mixture was heated at 120 °C for 20 hours. The mixture was cooled in an ice-bath and the solid filtered and washed with EtOH: H₂O (10 mL: 10 mL). H₂O (15 mL) was added to the filtrate which was then neutralised using AcOH, followed by a second filtration. The solid was again washed with EtOH: H₂O (10 mL: 10 mL) to afford compound **4h** (171 mg, 49 % yield) as a yellow solid.

IR ν_{\max} (liquid film): 3690 (OH), 3464 (NH), 1677 (C=O) and 1627 (C=C). **m.p.** 191-194 °C. **¹H NMR** (400 MHz; (CD₃)₂SO): δ_{H} 8.97 (1H, br.s, OH), 7.80 (1H, d, $J = 8.0$ Hz, ArCH), 7.55 (1H, d, $J = 8.0$ Hz, ArCH), 7.40 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.29 (1H, br.s, alkene CH), 7.17 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.02 (2H, s, Ar'CH), 4.00 (2H, s, CH₂), 3.91 (3H, s, NCH₃) and 3.85 (6H, s, Ar'OCH₃). **¹³C NMR** (100 MHz; (CD₃)₂SO): δ_{C} 182.3- (C=O), 148.1- (C), 144.1- (C), 140.2- (C), 137.8- (C), 137.8- (C), 137.3- (C), 131.7+ (alkene CH), 126.6+ (ArCH), 125.2- (C), 122.3- (C), 121.8+ (ArCH), 120.3+ (ArCH), 111.5+ (ArCH), 108.5+ (Ar'CH), 56.2+ (Ar'OCH₃), 30.0+ (NCH₃) and 25.8- (CH₂). **MS** m/z (+ESI) 350 (100 %, MH⁺) and 372 (12 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 350.1376, C₂₁H₂₀NO₄ requires MH 350.1392 and found MNa⁺ 372.1189, C₂₁H₁₉NNaO₄ requires MNa 372.1212.

2,3,4,9-Tetrahydro-1*H*-carbazol-1-one **9**



Method A

4-(Indol-3-yl)butanoic acid **7** (1.22 g, 6 mmol, 1 eq.) in TFAA (0.92 mL, 6.6 mmol, 1.1 eq.) was heated at 100 °C in a sealed pressure tube for 4 hours. On cooling, CHCl₃ was added and the solvent was removed under reduced pressure. H₂O (20 mL) and EtOAc (20 mL) were added, the reaction mixture was adjusted to pH 7 using 2M NaOH_(aq.) and stirred at room temperature for 16 hours. The layers were separated and the aqueous layer extracted with EtOAc (2 x 20 mL). The combined organic layers were washed with brine (20 mL), dried on Na₂SO₄ and filtered and the solvent was removed under reduced pressure. Column chromatography [silica, light petroleum (b.p. 40-60 °C) - EtOAc gradient column] followed by recrystallisation (EtOAc), gave compound **9** (592 mg, 53 % yield) as a yellow solid.

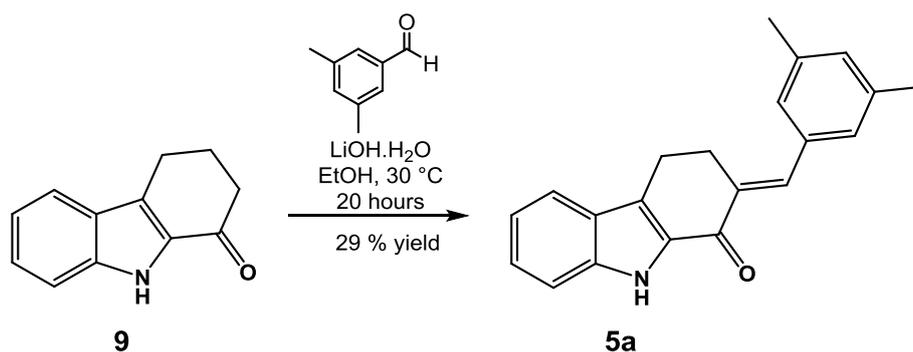
Method B

As above, except on a 2 mmol scale, heated for 2 hours and purification of the crude reaction mixture by column chromatography [silica, CH₂Cl₂ – MeOH gradient column] without aqueous work up, gave compound **9** (268 mg, **72 % yield**) as a pale yellow solid.

R_f (50 % EtOAc in light petroleum (b.p. 40-60 °C)) 0.5. **IR** ν_{\max} (liquid film): 3450 (NH), 3054 (CH) and 1666 (C=O). **m.p.** 170-172 °C [lit.⁷ 168-170 °C]. **¹H NMR** (400 MHz; CDCl₃): δ_{H} 9.31 (1H, br. s, NH), 7.66 (1H, d, $J = 8.0$ Hz, ArCH), 7.45 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.37 (1H, t, $J = 8.0$ Hz, ArCH), 7.15 (1H, t, $J = 7.5$ Hz, ArCH), 3.01 (2H, t, $J = 6.0$ Hz, COCH₂CH₂CH₂), 2.68 (2H, t, $J = 6.0$ Hz, COCH₂CH₂CH₂) and 2.27 (2H, quint, $J = 6.0$ Hz, COCH₂CH₂CH₂). **¹³C NMR** (100 MHz; CDCl₃): δ_{C} 191.5– (C=O), 137.9– (C), 131.2– (C), 129.6– (C), 127.0+ (ArCH), 125.8– (C), 121.3+ (ArCH), 120.3+ (ArCH), 112.6+ (ArCH), 38.2– (COCH₂CH₂CH₂), 25.0– (COCH₂CH₂CH₂) and 21.4– (COCH₂CH₂CH₂). **MS** m/z (+ESI) 186 (43 %, MH⁺) and 208 (100 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 186.0909, C₁₂H₁₂NO requires MH 186.0919 and found MNa⁺ 208.0726, C₁₂H₁₁NNaO requires MNa 208.0738.

Consistent with the spectroscopic data previously reported for this compound.^{7,8}

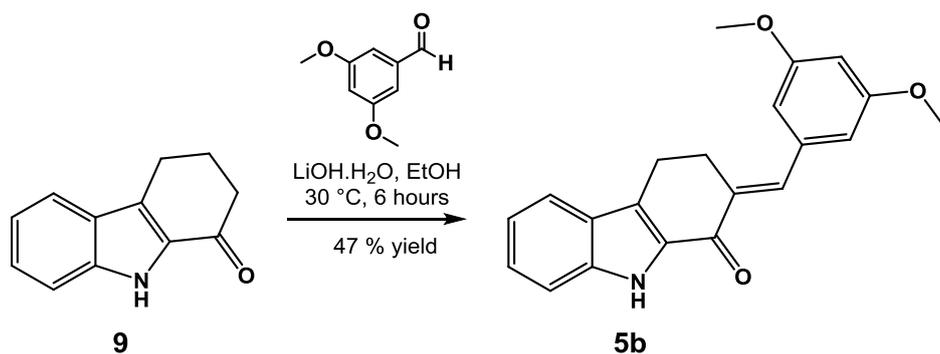
(E)-2-(3,5-Dimethylbenzylidene)-2,3,4,9-tetrahydro-1H-carbazol-1-one 5a



Following a previously reported procedure,⁹ ketone **9** (203 mg, 1.1 mmol, 1 eq.) and LiOH.H₂O (51 mg, 1.21 mmol, 1.1 eq.) in EtOH (3 mL) were stirred for 10 minutes, followed by the addition of 3,5-dimethylbenzaldehyde (0.15 mL, 1.1 mmol, 1.1 eq.). The reaction mixture was stirred at 30 °C for 20 hours. The solvent was removed under reduced pressure and the yellow residue was diluted with H₂O (10 mL) and EtOAc (10 mL). The mixture was neutralised with 6M HCl_(aq) followed by extraction with EtOAc (2 x 10 mL). The combined organic layers were washed with brine (10 mL), dried on Na₂SO₄, filtered and concentrated under reduced pressure. Column chromatography [silica, light petroleum (b.p. 40-60 °C) - EtOAc gradient column], gave the product **5a** (95 mg, 29 % yield) as a pale yellow solid.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.8. **IR** ν_{\max} (liquid film): 3460 (NH), 3046 (CH), 1651 (C=O) and 1601 (C=C). **m.p.** 209-211 °C (recrystallised from EtOAc). **¹H NMR** (400 MHz; CDCl₃): δ_{H} 9.21 (1H, br.s, NH), 7.80 (1H, br.s, alkene CH), 7.70 (1H, dd, $J = 8.0, 1.0$ Hz, ArCH), 7.48 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.41 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.20 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.11 (2H, s, Ar'CH), 7.04 (1H, s, Ar'CH), 3.30 (2H, td, $J = 6.5, 1.5$ Hz, CH₂CH₂C=CH), 3.11 (2H, t, $J = 6.5$ Hz, CH₂CH₂C=CH), 2.41 (6H, s, Ar'CH₃). **¹³C NMR** (100 MHz; CDCl₃): δ_{C} 181.1- (C=O), 138.6- (C), 138.0- (C), 136.1- (C), 136.0- (C), 135.7+ (alkene CH), 132.4- (C), 130.1+ (Ar'CH), 128.3- (C), 127.6+ (Ar'CH), 127.2+ (ArCH), 126.0- (C), 121.4+ (ArCH), 120.5+ (ArCH), 112.5+ (ArCH), 27.7- (CH₂CH₂C=CH), 21.4+ (Ar'CH₃) 20.9- (CH₂CH₂C=CH). **MS** m/z (+ESI) 302 (100 %, MH⁺) and 324 (32 %, MNa⁺). **HRMS** (+ESI) Found MH⁺, 302.1546, C₂₁H₂₀NO requires MH 302.1545 and found MNa⁺ 324.1363, C₂₁H₁₉NNaO requires MNa 324.1364.

(E)-2-(3,5-Dimethoxybenzylidene)-2,3,4,9-tetrahydro-1H-carbazol-1-one 5b



Method A

A mixture of ketone **9** (260 mg, 1.40 mmol, 1 eq.) and 3,5-dimethoxybenzaldehyde (256 mg, 1.54 mmol, 1.1 eq.) were treated with 4 % (w/v) ethanolic KOH (10 mL) and stirred at room temperature for 16 hours.⁴ The mixture was cooled in an ice-bath and the solid was filtered and washed with EtOH: H₂O (10 mL: 10 mL). H₂O (20 mL) was added to the filtrate which was then neutralised using AcOH, followed by a second filtration. The solid was again washed with EtOH: H₂O (10 mL: 10 mL) to afford compound **5b** (158 mg, 34 % yield) as a yellow solid.

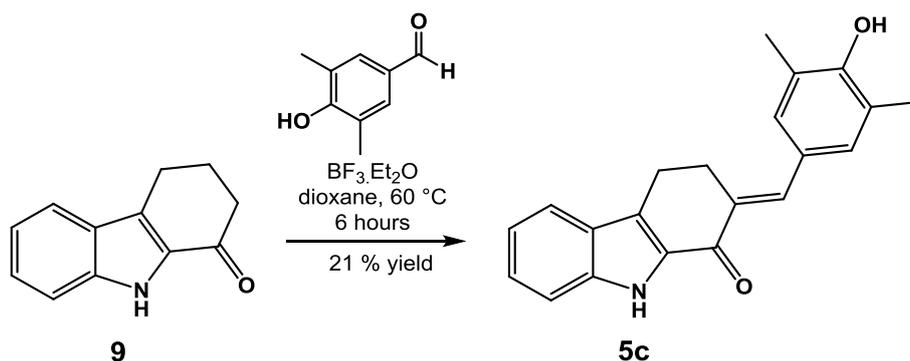
Method B

Following a previously reported procedure,⁹ ketone **9** (235 mg, 1.27 mmol, 1 eq.) and LiOH.H₂O (133 mg, 3.18 mmol, 2.5 eq.) in EtOH (2 mL) were stirred for 10 minutes, followed by the addition of 3,5-dimethoxybenzaldehyde (318 mg, 1.91 mmol, 1.5 eq.). The reaction mixture was stirred at 30 °C for 6 hours. The solvent was removed under reduced pressure and the yellow residue was diluted with water (20 mL) and EtOAc (20 mL). The mixture was neutralised with 6M HCl_(aq) followed by extraction with EtOAc (2 x 10 mL). The combined organic layers were washed with brine (20 mL), dried on Na₂SO₄, filtered and concentrated under reduced pressure to collect 456 mg of crude product. Column chromatography [silica, light petroleum (b.p. 40-60 °C) - EtOAc gradient column], gave the product **5b** (197 mg, **47 % yield**) as a yellow solid.

R_f (70 % EtOAc in light petroleum (b.p. 40-60 °C) 0.9. **IR** ν_{\max} (liquid film): 3460 (NH), 3026 (CH), 1652 (C=O) and 1597 (C=C). **m.p.** 168-170 °C. **¹H NMR** (400 MHz; CDCl₃): δ_{H} 8.96 (1H, br.s, NH), 7.76 (1H, br.s, alkene CH), 7.70 (1H, dd, $J = 8.0, 1.0$ Hz, ArCH), 7.48 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.42 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.20 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 6.63 (2H, dd, $J = 2.5, 0.5$ Hz, Ar'CH), 6.51 (1H, t, $J = 2.5$ Hz,

Ar'CH), 3.87 (6H, s, Ar'OCH₃), 3.30 (2H, td, *J* = 6.5, 2.0 Hz, CH₂CH₂C=CH) and 3.11 (2H, t, *J* = 6.5 Hz, CH₂CH₂C=CH). ¹³C NMR (100 MHz; CDCl₃): δ_C 181.7– (C=O), 160.8– (C), 140.0– (C), 138.1– (C), 138.0– (C), 135.4– (C), 135.3+ (alkene CH), 132.3– (C), 127.3+ (ArCH), 128.0– (C), 127.3+ (ArCH), 121.5+ (ArCH), 120.6+ (ArCH), 112.5+ (ArCH), 107.8+ (Ar'CH), 100.4+ (Ar'CH), 55.4+ (OCH₃), 27.7– (CH₂CH₂C=CH) and 20.9– (CH₂CH₂C=CH). **MS** *m/z* (+ESI) 334 (100 %, MH⁺) and 356 (9 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 334.1433, C₂₁H₂₀NO₃ requires *MH* 334.1443 and found MNa⁺ 356.1249, C₂₁H₁₉NNaO₃ requires *MNa* 356.1263. **Analysis** (Found: H, 5.74; N, 4.20. C₂₁H₁₉NO₃ requires H, 5.74; N, 4.20 %).

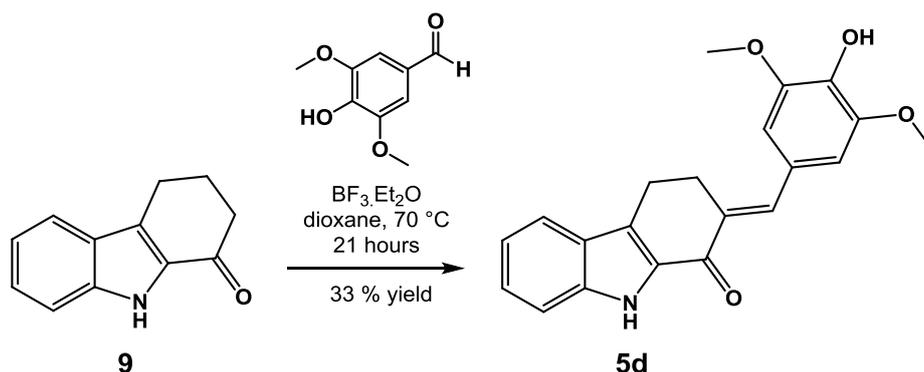
(E)-2-(4-Hydroxy-3,5-dimethylbenzylidene)-2,3,4,9-tetrahydro-1H-carbazol-1-one 5c



All glassware was dried under N_2 using a heatgun. Ketone **9** (192 mg, 1.04 mmol, 1 eq.) and 3,5-dimethyl-4-hydroxybenzaldehyde (156 mg, 1.04 mmol, 1 eq.) were added to the flask followed by anhydrous dioxane (2 mL). $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (0.4 mL, 3.12 mmol, 3 eq.) was added slowly and the reaction mixture was heated at 60°C for 6 hours.¹⁰ On cooling, H_2O (60 mL), 2M $\text{NaOH}_{(\text{aq})}$ (20 mL) and EtOAc (60 mL) were added to the reaction mixture. The layers were separated and the aqueous layer was extracted with EtOAc (2 x 60 mL). The combined organic layers were washed with brine (60 mL), dried on Na_2SO_4 , filtered and concentrated under reduced pressure to collect 343 mg of crude product. Column chromatography [silica, light petroleum (b.p. $40\text{--}60^\circ\text{C}$) - EtOAc gradient column], gave the product **5c** (70 mg, 21 % yield) as a yellow solid.

R_f (30 % EtOAc in light petroleum (b.p. $40\text{--}60^\circ\text{C}$)) 0.5. **IR** ν_{max} (liquid film): 3605 (OH), 3460 (NH), 3042 (CH), 1648 (C=O) and 1599 (C=C). **m.p.** $186\text{--}188^\circ\text{C}$. **¹H NMR** (400 MHz; CDCl_3): δ_{H} 8.80 (1H, br.s, NH), 7.73 (1H, br.s, alkene CH), 7.70 (1H, dd, $J = 8.0, 1.0$ Hz, ArCH), 7.46 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.41 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.20 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.17 (2H, s, Ar'CH), 4.84 (1H, br.s, OH), 3.32 (2H, td, $J = 6.5, 1.5$ Hz, $\text{CH}_2\text{CH}_2\text{C}=\text{CH}$), 3.11 (2H, t, $J = 6.5$ Hz, $\text{CH}_2\text{CH}_2\text{C}=\text{CH}$) and 2.35 (6H, s, Ar'CH₃). **¹³C NMR** (100 MHz; CDCl_3): δ_{C} 181.1– (C=O), 152.8– (C), 138.4– (C), 135.8+ (alkene CH), 134.1– (C), 132.5– (C), 130.7+ (Ar'CH), 128.2– (C), 127.9– (C), 127.0+ (ArCH), 126.0– (C), 123.2– (C), 121.3+ (ArCH), 120.4+ (ArCH), 112.5+ (ArCH), 27.7– ($\text{CH}_2\text{CH}_2\text{C}=\text{CH}$), 20.8– ($\text{CH}_2\text{CH}_2\text{C}=\text{CH}$) and 16.0+ (Ar'CH₃). **MS** m/z (+ESI) 318 (100 %, MH^+) and 340 (19 %, MNa^+). **HRMS** (+ESI) Found MH^+ 318.1486, $\text{C}_{21}\text{H}_{20}\text{NO}_2$ requires MH 318.1494 and found MNa^+ 340.1302, $\text{C}_{21}\text{H}_{19}\text{NNaO}_2$ requires MNa , 340.1313. **NOESY NMR** Interaction observed between 7.17 (2H, s, Ar'CH) and 3.32 (2H, td, $J = 6.5, 1.5$ Hz, $\text{CH}_2\text{CH}_2\text{C}=\text{CH}$) to confirm *E* configuration.

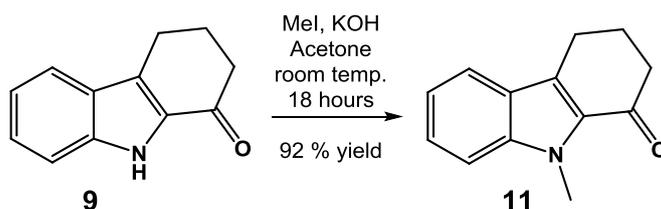
(E)-2-(4-Hydroxy-3,5-dimethoxybenzylidene)-2,3,4,9-tetrahydro-1H-carbazol-1-one 5d



All glassware was dried under N_2 using a heatgun. Ketone **9** (309 mg, 1.67 mmol, 2.5 eq.) and syringaldehyde (122 mg, 0.67 mmol, 1 eq.) were added to the flask followed by anhydrous dioxane (4 mL). $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (0.25 mL, 2.01 mmol, 3 eq.) was added slowly and the reaction mixture was heated at 70°C for 21 hours.¹⁰ On cooling, H_2O (30 mL), 2M $\text{NaOH}_{(\text{aq})}$ (10 mL) and EtOAc (30 mL) were added to the reaction mixture. The layers were separated and the aqueous layer was extracted with EtOAc (2 x 30 mL). The combined organic layers were washed with brine (30 mL), dried on Na_2SO_4 , filtered and concentrated under reduced pressure to collect 465 mg of crude product. Column chromatography [silica, light petroleum (b.p. $40\text{--}60^\circ\text{C}$) EtOAc gradient column], gave the product **5d** (78 mg, 33 % yield) as a yellow solid.

R_f (70 % EtOAc in light petroleum (b.p. $40\text{--}60^\circ\text{C}$)) 0.7. **IR** ν_{max} (liquid film): 3534 (OH), 3460 (NH), 3033 (CH), 1649 (C=O) and 1612 (C=C). **m.p.** $155\text{--}157^\circ\text{C}$. **¹H NMR** (400 MHz; CDCl_3): δ_{H} 9.09 (1H, br.s, NH), 7.78 (1H, br.s, alkene CH), 7.70 (1H, dd, $J = 8.0, 1.0$ Hz, ArCH), 7.47 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.42 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.20 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 6.76 (2H, s, Ar'CH), 5.72 (1H, s, OH), 3.97 (6H, s, Ar'OCH₃), 3.35 (2H, td, $J = 6.5, 1.5$ Hz, CH₂CH₂C=CH) and 3.13 (2H, t, $J = 6.5$ Hz, CH₂CH₂C=CH). **¹³C NMR** (100 MHz; CDCl_3): δ_{C} 180.7– (CO), 147.0– (C), 138.5– (C), 135.9+ (alkene CH), 135.6– (C), 134.7– (C), 132.4– (C), 128.0– (C), 127.4– (C), 127.2+ (ArCH), 126.0– (C), 121.4+ (ArCH), 120.5+ (ArCH), 112.5+ (ArCH), 107.2+ (Ar'CH), 56.5+ (Ar'OCH₃), 27.7– (CH₂CH₂C=CH) and 20.8– (CH₂CH₂C=CH). **MS** m/z (+ESI) 350 (100 %, MH^+). **HRMS** (+ESI) Found MH^+ 350.1394, $\text{C}_{21}\text{H}_{20}\text{NO}_4$ requires MH , 350.1392.

9-Methyl-2,3,4,9-tetrahydro-1*H*-carbazol-1-one **11**

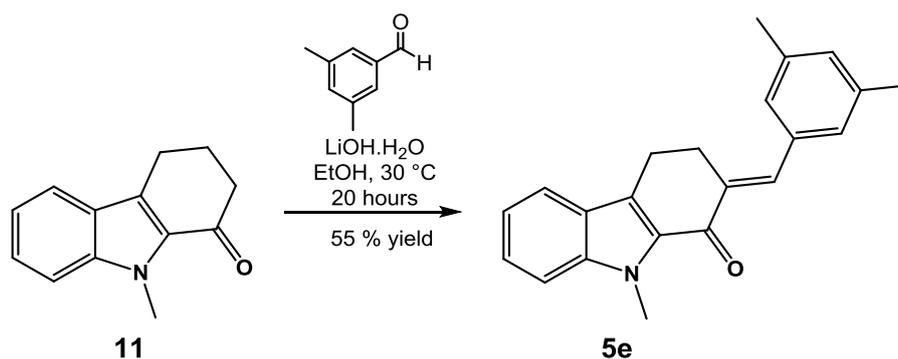


Following the procedure previously reported,⁵ MeI (2.4 eq.) was added to a rapidly stirred solution of the indole **9** (1.95 mmol) and KOH (2.9 eq.) in acetone (10 mL/ 1 mmol). After 18 hours at room temperature, the solvent was removed under reduced pressure and H₂O added and acidified to pH 1 with 6M HCl_(aq.). The aqueous layer was extracted three times with CH₂Cl₂ and the combined organic layers washed with brine, dried with Na₂SO₄, filtered and concentrated under reduced pressure to afford, without the need for further purification, the methylated product **11** (358 mg, 92 % yield) as a beige solid.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.8. **IR** ν_{\max} (liquid film): 3031 (CH) and 1662 (C=O). **m.p.** 96-99 °C [lit.¹¹ 100-101 °C]. **¹H NMR** (400 MHz; CDCl₃): δ_{H} 7.69 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.44 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 7.38 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.18 (1H, ddd, $J = 8.0, 7.0, 1.0$ Hz, ArCH), 4.11 (3H, s, NCH₃), 3.06 (2H, t, $J = 6.0$ Hz, COCH₂CH₂CH₂), 2.69 (2H, t, $J = 6.0$ Hz, COCH₂CH₂CH₂) and 2.26 (2H, quint, $J = 6.0$ Hz, COCH₂CH₂CH₂). **¹³C NMR** (100 MHz; CDCl₃): δ_{C} 192.3- (C=O), 139.7- (C), 130.5- (C), 129.2- (C), 126.7+ (ArCH), 124.8- (C), 121.3+ (ArCH), 120.0+ (ArCH), 110.3+ (ArCH), 40.0- (COCH₂CH₂CH₂), 31.5+ (NCH₃), 24.8- (COCH₂CH₂CH₂) and 21.9- (COCH₂CH₂CH₂). **MS** m/z (+ESI) 200 (100 %, MH⁺) and 222 (29 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 200.1068, C₁₃H₁₄NO requires MH 200.1075 and found MNa⁺ 222.0886, C₁₃H₁₃NNaO requires MNa 222.0895.

Consistent with the spectroscopic data previously reported for this compound.^{1,11}

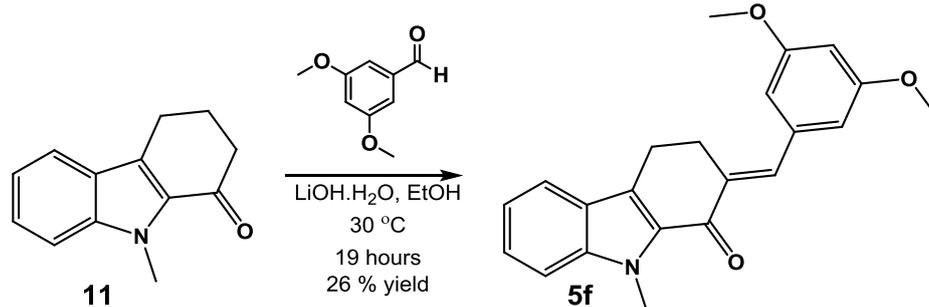
(E)-2-(3,5-Dimethylbenzylidene)-9-methyl-2,3,4,9-tetrahydro-1H-carbazol-1-one 5e



Following a previously reported procedure,⁹ ketone **11** (172 mg, 0.86 mmol, 2 eq.) and LiOH.H₂O (54 mg, 1.29 mmol, 3 eq.) in EtOH (2 mL) were stirred for 10 minutes, followed by the addition of 3,5-dimethylbenzaldehyde (58 mg, 0.43 mmol, 1 eq.). The reaction mixture was stirred at 30 °C for 20 hours and the solvent was removed under reduced pressure. Column chromatography [silica, light petroleum (b.p. 40-60 °C) - EtOAc gradient column], gave the product **5e** (75 mg, 55 % yield) as a yellow solid.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.9. **IR** ν_{\max} (liquid film): 3027 (CH), 1651 (C=O) and 1613 (C=C). **m.p.** 96-98 °C. **¹H NMR** (400 MHz; CDCl₃): δ_{H} 7.77 (1H, br.s, alkene CH), 7.69 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.45 (1H, td, $J = 8.5, 1.0$ Hz, ArCH), 7.41 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.19 (1H, ddd, $J = 8.0, 6.5, 1.0$ Hz, ArCH), 7.09 (2H, s, Ar'CH), 7.03 (1H, s, Ar'CH), 4.20 (3H, s, NCH₃), 3.24 (2H, td, $J = 6.0, 1.0$ Hz, CH₂CH₂C=C), 3.09 (2H, t, $J = 6.0$ Hz, CH₂CH₂C=C) and 2.40 (6H, s, Ar'CH₃). **¹³C NMR** (CDCl₃, 100 MHz): δ_{C} 181.9- (C=O), 140.4- (C), 137.9- (C), 137.2- (C), 136.3- (C), 135.4+ (alkene CH), 131.6- (C), 129.9+ (Ar'CH), 128.3- (C), 127.5+ (Ar'CH), 126.8+ (ArCH), 124.7- (C), 121.3+ (ArCH), 120.1+ (ArCH), 110.3+ (ArCH), 31.7+ (NCH₃), 27.7- (CH₂CH₂C=C), 21.4+ (Ar'CH₃) and 21.1- (CH₂CH₂C=C). **MS** m/z (+ESI) 316 (100 %, MH⁺) and 338 (15 %, MNa⁺). **HRMS** (+ESI) Found MH⁺ 316.1701, C₂₂H₂₂NO requires MH 316.1701 and found MNa⁺ 338.1517, C₂₂H₂₁NNaO requires MNa 338.1521.

(E)-2-(3,5-Dimethoxybenzylidene)-9-methyl-2,3,4,9-tetrahydro-1H-carbazol-1-one 5f



Method A

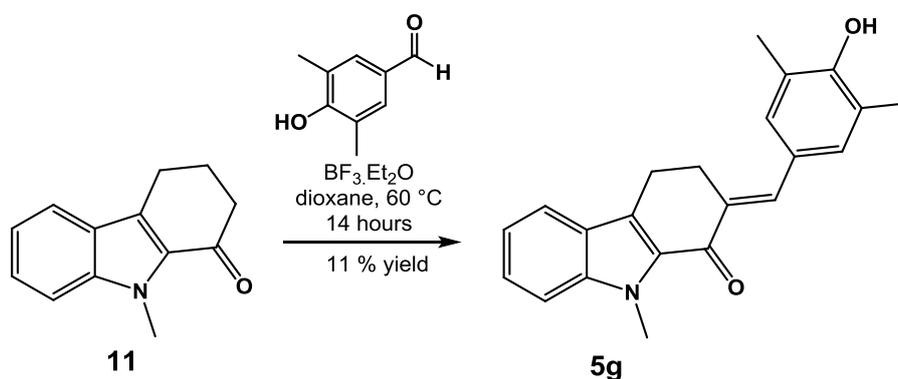
A mixture of ketone **11** (229 mg, 1.15 mmol, 1 eq.) and 3,5-dimethoxybenzaldehyde (211 mg, 1.27 mmol, 1.1 eq.) were treated with 4 % (w/v) ethanolic KOH (11.5 mL) and stirred at room temperature for 16 hours.⁴ The mixture was cooled in an ice-bath and the solid was filtered and washed with EtOH: H₂O (10 mL: 10 mL). H₂O (20 mL) was added to the filtrate which was then neutralised using AcOH, followed by a second filtration. The solid was again washed with EtOH: H₂O (10 mL: 10 mL) to afford **5f** (97 mg, 24 % yield) as a yellow solid.

Method B

Following a previously reported procedure,⁹ ketone **11** (139 mg, 0.70 mmol, 2 eq.) and LiOH.H₂O (44 mg, 1.05 mmol, 3 eq.) in EtOH (2 mL) were stirred for 10 minutes, followed by the addition of 3,5-dimethoxybenzaldehyde (58 mg, 0.35 mmol, 1 eq.). The reaction mixture was stirred at 30 °C for 19 hours and the solvent was removed under reduced pressure. Column chromatography [silica, light petroleum (b.p. 40-60 °C) - EtOAc gradient column], gave the product **5f** (31 mg, **26 % yield**) as yellow crystals.

R_f (30 % EtOAc in light petroleum (b.p. 40-60 °C) 0.8. **IR** ν_{\max} (liquid film): 3026 (CH), 1652 (C=O) and 1594 (C=C). **m.p.** 134-135 °C. **¹H NMR** (400 MHz; CDCl₃): δ_{H} 7.74 (1H, br.s, alkene CH), 7.69 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.46 (1H, ddd, $J = 8.5, 6.5, 1.0$ Hz, ArCH), 7.41 (1H, dt, $J = 8.5, 1.0$ Hz, ArCH), 7.19 (1H, ddd, $J = 8.0, 6.5, 1.0$ Hz, ArCH), 6.61 (2H, dd, $J = 2.5, 0.5$ Hz, Ar'CH), 6.50 (1H, t, $J = 2.5$ Hz, Ar'CH), 4.19 (3H, s, NCH₃), 3.86 (6H, s, Ar'OCH₃), 3.24 (2H, td, $J = 6.5, 1.0$ Hz, CH₂CH₂C=CH) and 3.09 (2H, t, $J = 6.5$ Hz, CH₂CH₂C=CH). **¹³C NMR** (100 MHz; CDCl₃): δ_{C} 181.7- (C=O), 160.7- (C), 140.5- (C), 138.3- (C), 138.0- (C), 134.9+ (alkene CH), 131.5- (C), 128.6- (C), 126.9+ (ArCH), 124.6- (C), 121.4+ (ArCH), 120.2+ (ArCH), 110.4+ (ArCH), 107.7+ (Ar'CH), 100.2+ (Ar'CH), 55.4+ (Ar'OCH₃), 31.7+ (NCH₃), 27.7- (CH₂CH₂C=CH) and 21.1- (CH₂CH₂C=CH). **MS** m/z (+ESI) 348 (100 %, MH⁺) and 370 (18 %, MNa⁺) **HRMS** (+ESI) Found MH⁺ 348.1606, C₂₂H₂₂NO₃ requires MH 348.1600 and found MNa⁺ 370.1423, C₂₂H₂₁NNaO₃ requires MNa 370.1419. **Analysis** (Found: H, 6.06; N, 3.93. C₂₂H₂₁NO₃ requires H, 6.09; N, 4.03 %).

(E)-2-(4-Hydroxy-3,5-dimethylbenzylidene)-9-methyl-2,3,4,9-tetrahydro-1H-carbazol-1-one 5g



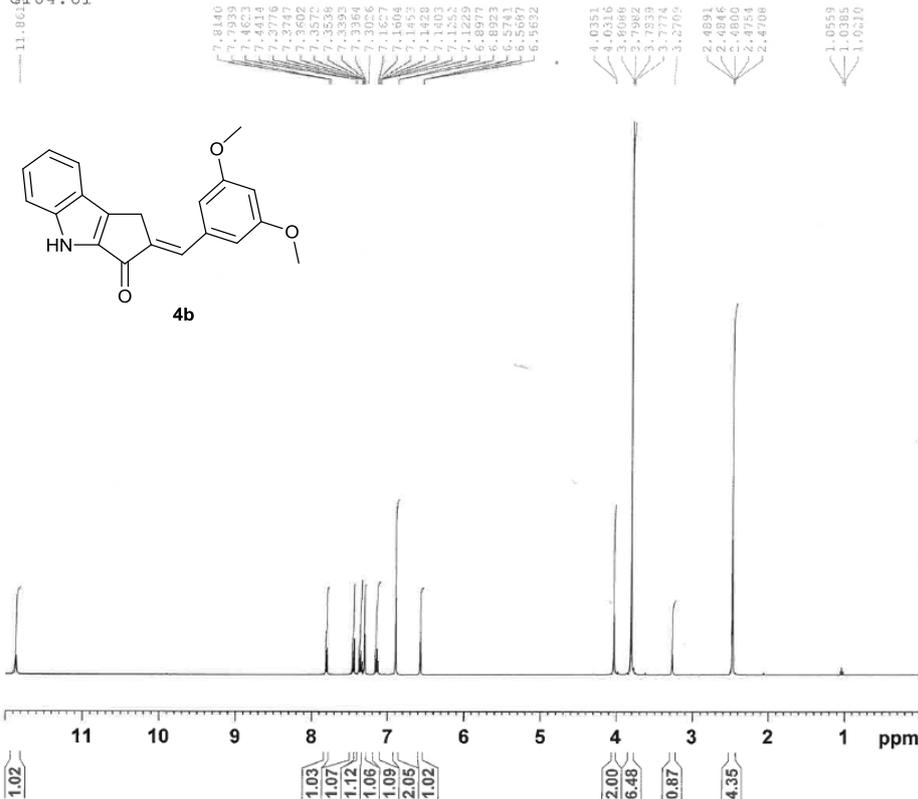
All glassware was dried under N_2 using a heatgun. Ketone **11** (106 mg, 0.53 mmol, 2 eq.) and 3,5-dimethyl-4-hydroxybenzaldehyde (41 mg, 0.27 mmol, 1 eq.) were added to the flask followed by anhydrous dioxane (2 mL). $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (0.1 mL, 0.8 mmol, 2.3 eq.) was added slowly and the reaction mixture was heated at 60°C for 14 hours.¹⁰ On cooling, H_2O (40 mL), 2M $\text{NaOH}_{(\text{aq})}$ (20 mL) and EtOAc (40 mL) were added to the reaction mixture. The layers were separated and the aqueous layer was extracted with EtOAc (2 x 40 mL). The combined organic layers were washed with brine (40 mL), dried on Na_2SO_4 , filtered and concentrated under reduced pressure to collect 139 mg of crude product. Column chromatography [silica, light petroleum (b.p. $40\text{--}60^\circ\text{C}$) - EtOAc gradient column], gave the product **5g** (10 mg, 11 % yield) as a yellow solid.

R_f (30 % EtOAc in light petroleum (b.p. $40\text{--}60^\circ\text{C}$)) 0.8. **IR** ν_{max} (liquid film): 3608 (OH), 3038 (CH), 1648 (C=O) and 1601 (C=C). **m.p.** $203\text{--}205^\circ\text{C}$. **¹H NMR** (400 MHz; CDCl_3): δ_{H} 7.72 (1H, br.s, alkene CH), 7.69 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.47-7.43 (1H, m, ArCH), 7.41 (1H, dt, $J = 8.0, 1.0$ Hz, ArCH), 7.19 (1H, ddd, $J = 8.0, 6.5, 1.0$ Hz, ArCH), 7.15 (2H, s, Ar'CH), 4.81 (1H, br.s, OH), 4.19 (3H, s, NCH_3), 3.26 (2H, td, $J = 6.5, 1.0$ Hz, $\text{CH}_2\text{CH}_2\text{C}=\text{CH}$), 3.09 (2H, t, $J = 6.5$ Hz, $\text{CH}_2\text{CH}_2\text{C}=\text{CH}$) and 2.33 (6H, s, Ar'CH₃). **¹³C NMR** (100 MHz; CDCl_3): δ_{C} 181.9- (CO), 152.6- (C), 140.4- (C), 135.5- (C), 135.4+ (alkene CH), 131.6- (C), 130.5+ (Ar'CH), 128.5- (C), 127.9- (C), 126.7+ (ArCH), 124.7- (C), 123.0- (C), 121.3+ (ArCH), 120.1+ (ArCH), 110.3+ (ArCH), 31.6+ (NCH_3), 27.6- ($\text{CH}_2\text{CH}_2\text{C}=\text{CH}$), 21.0- ($\text{CH}_2\text{CH}_2\text{C}=\text{CH}$) and 15.9+ (Ar'CH₃). **MS** m/z (+ESI) 332 (100 %, MH^+) and 354 (28 %, MNa^+). **HRMS** (+ESI) Found MH^+ 332.1634, $\text{C}_{22}\text{H}_{22}\text{NO}_2$ requires MH 332.1651 and found MNa^+ 354.1453, $\text{C}_{22}\text{H}_{21}\text{NNaO}_2$ requires MNa 354.1470. **NOESY NMR** Interaction observed between 7.15 (2H, s, Ar'CH) and 3.26 (2H, td, $J = 6.5, 1.0$ Hz, $\text{CH}_2\text{CH}_2\text{C}=\text{CH}$) to confirm *E* configuration.

References

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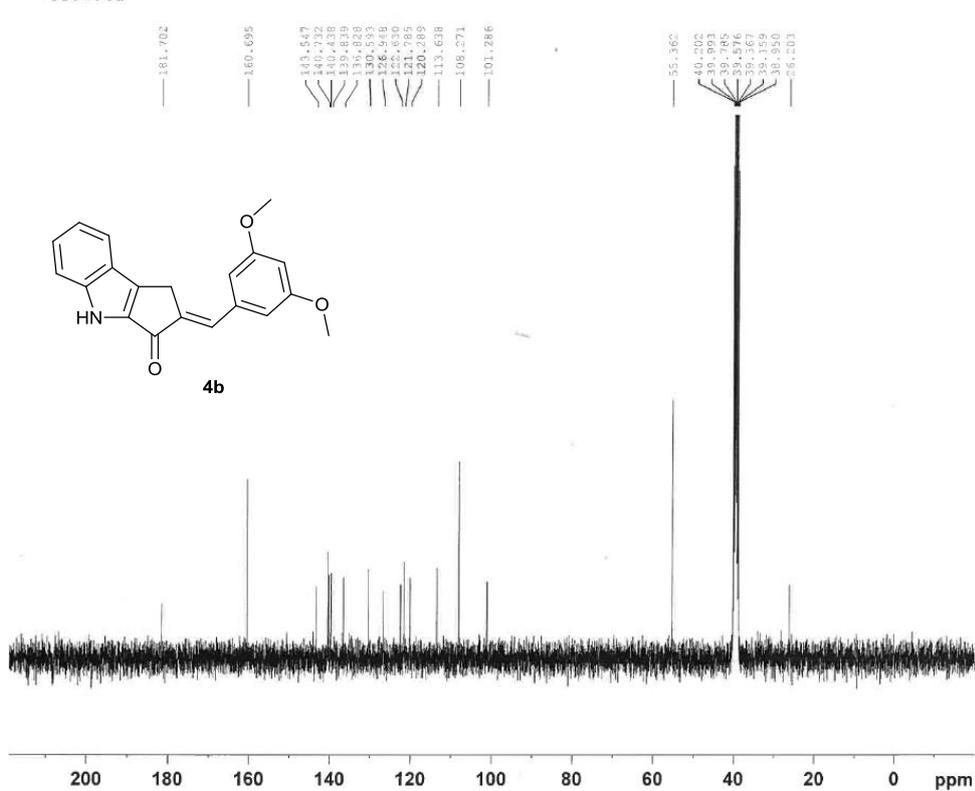
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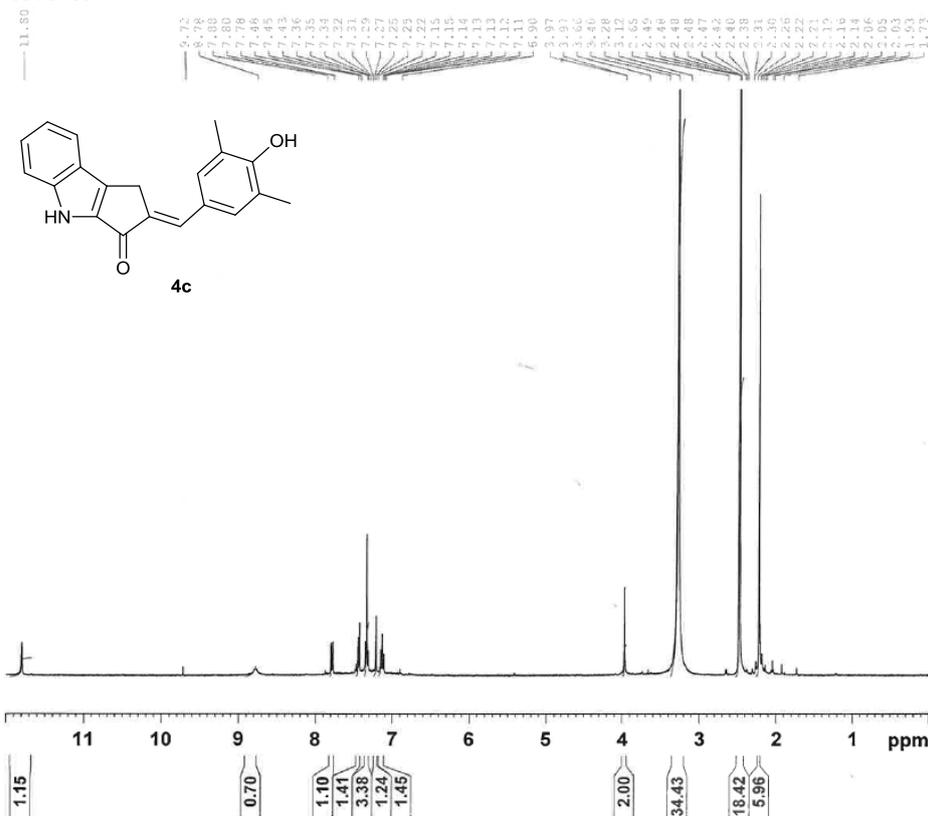


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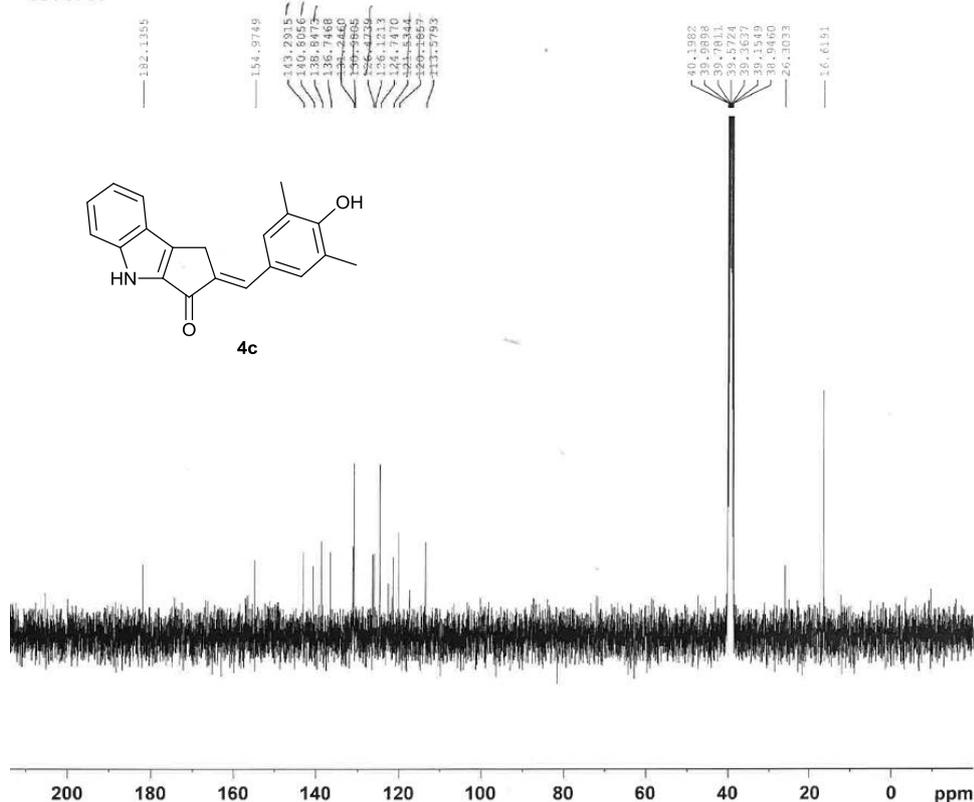
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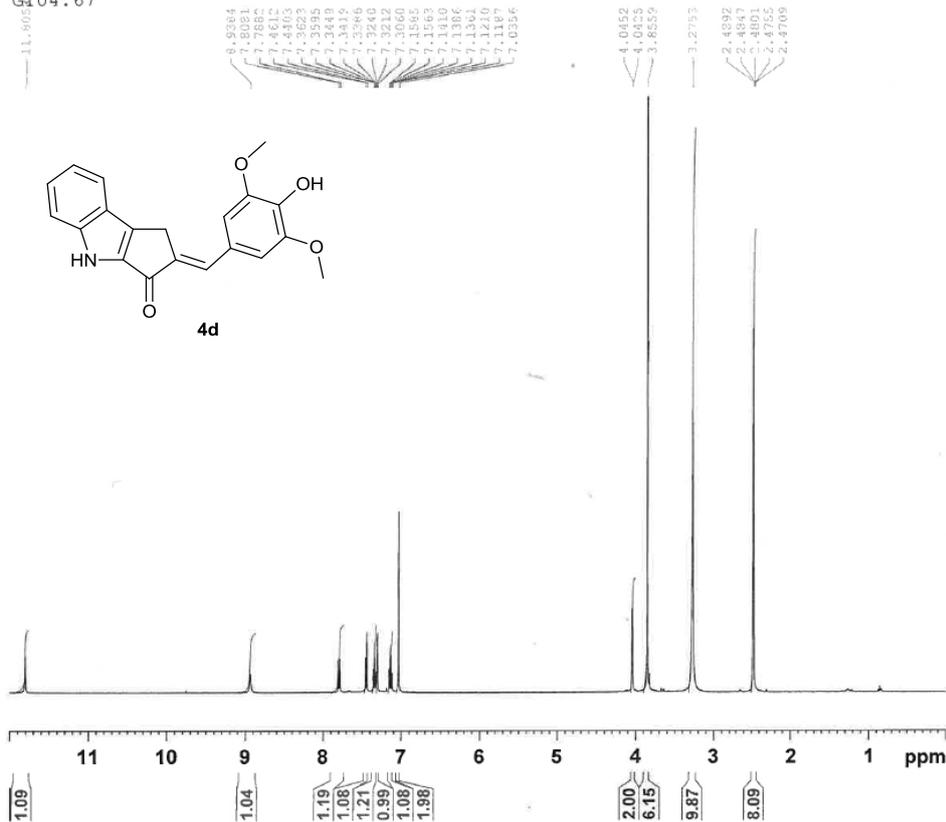
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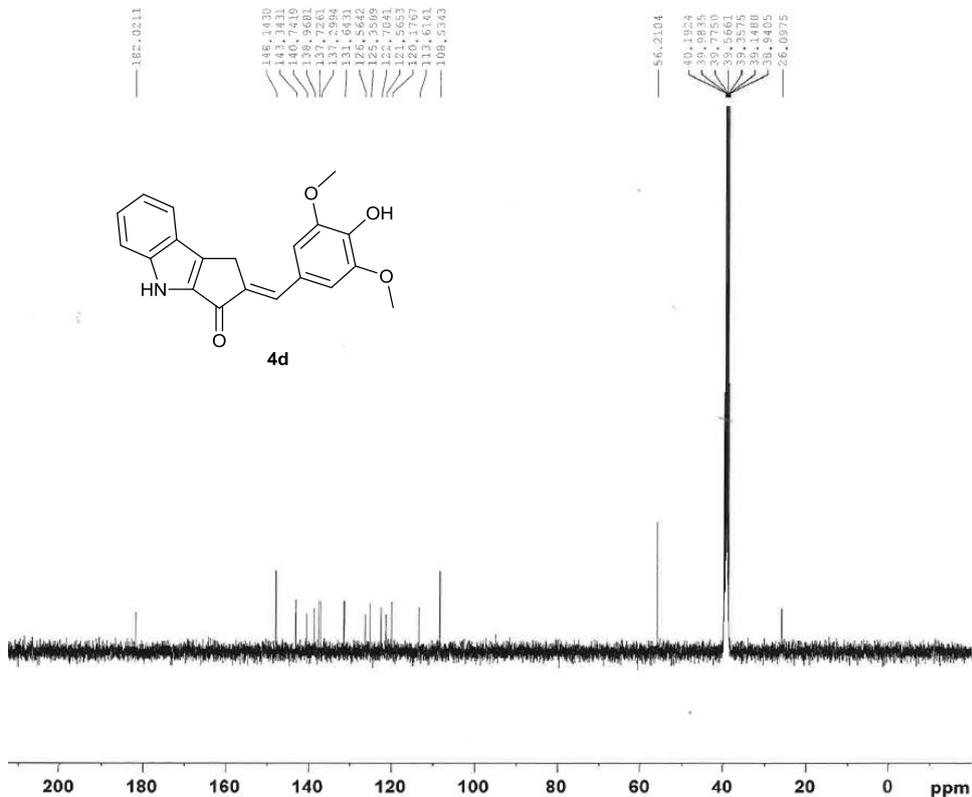
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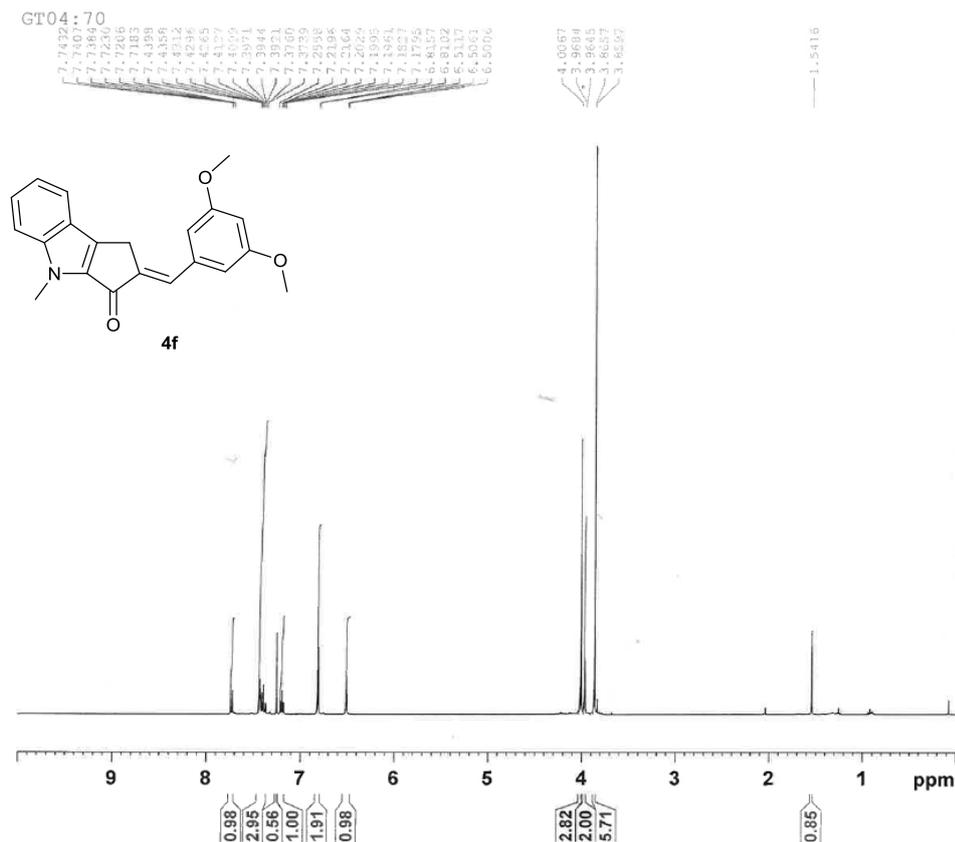


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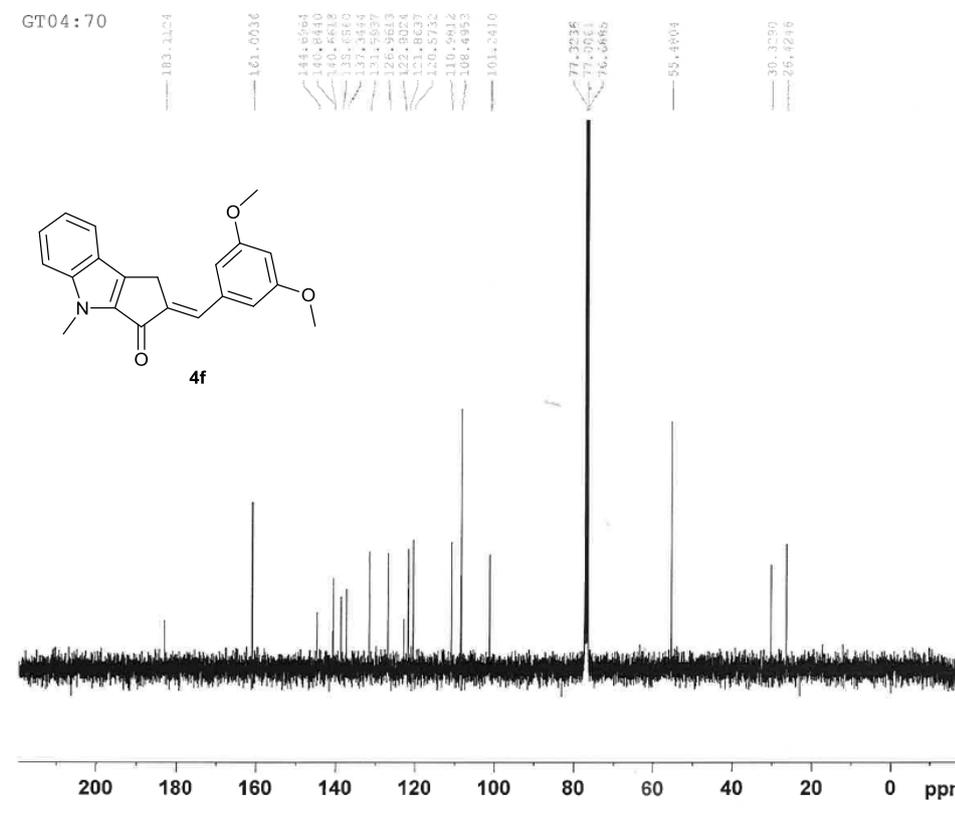
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NAME Jul106-2010-GT3369
 EXPNO 10
 PROCNO 1
 Date_ 20100707
 Time_ 0.46
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 237
 DW 60.800 usec
 DE 17.24 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

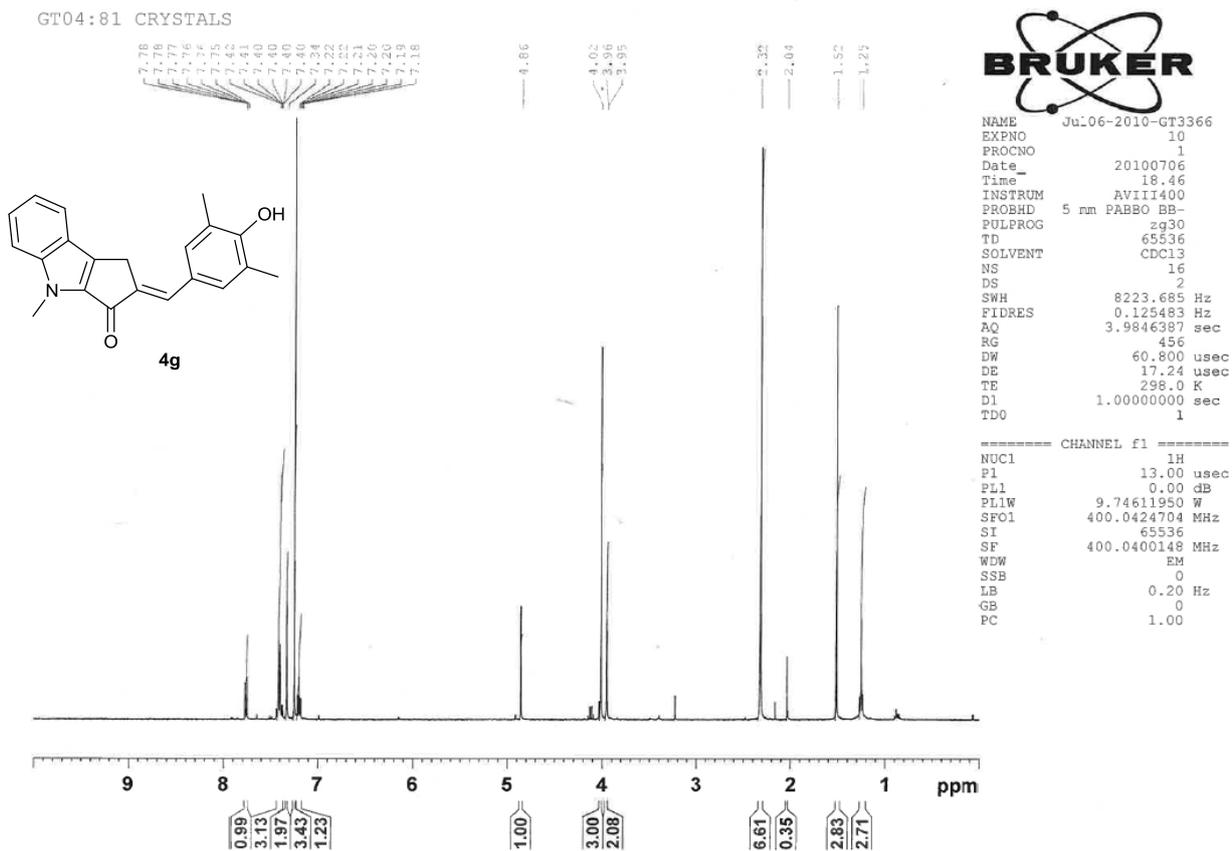
===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 0.00 dB
 PL1W 9.74611950 W
 SFO1 400.0424704 MHz
 SI 65536
 SF 400.0400148 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00



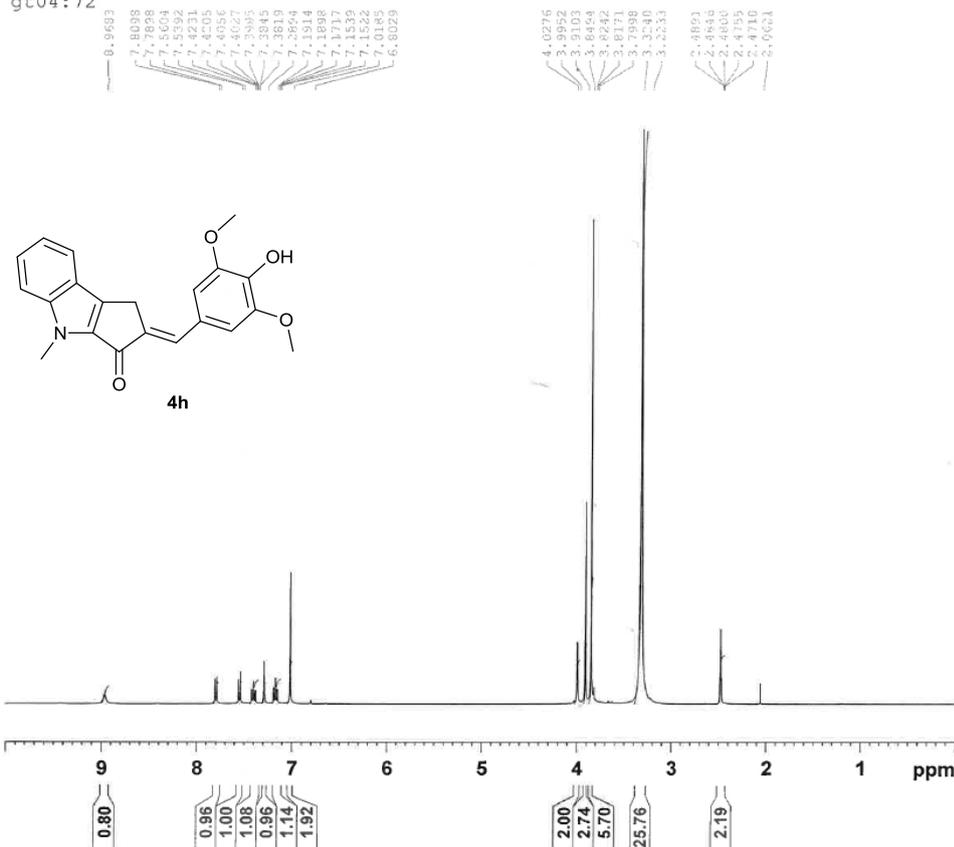
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 PROCNO 1
 Date_ 20100707
 Time_ 1.17
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.75 usec
 PL1 -2.00 dB
 PL1W 58.91986084 W
 SFO1 100.6001970 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.78 dB
 PL13 19.00 dB
 PL2W 9.74611950 W
 PL12W 0.25753233 W
 PL13W 0.12269637 W
 SFO2 400.0416002 MHz
 SI 65536
 SF 100.5901380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



gt04:72



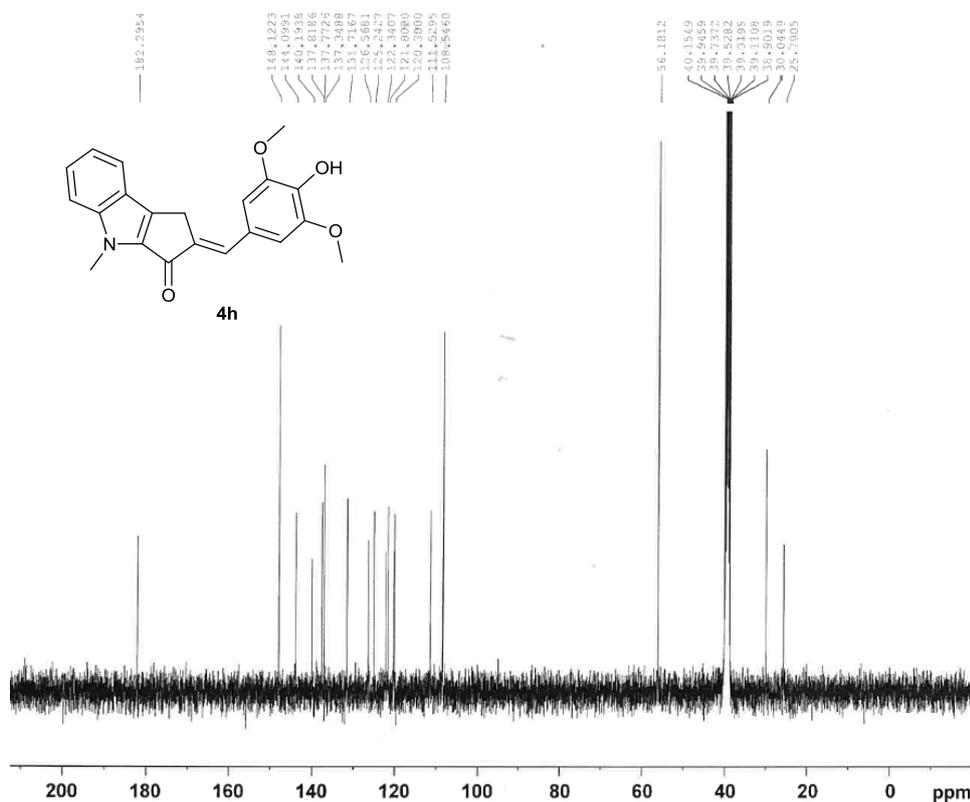
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NAME Jun10-2010-gt2760
EXPNO 10
PROCNO 1
Date_ 20100610
Time 16.55
INSTRUM AVIII400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 17.24 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
    
```

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===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 0.00 dB
PL1W 9.74611950 W
SF01 400.0424704 MHz
SI 65536
SF 400.0400148 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
    
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gt04:72



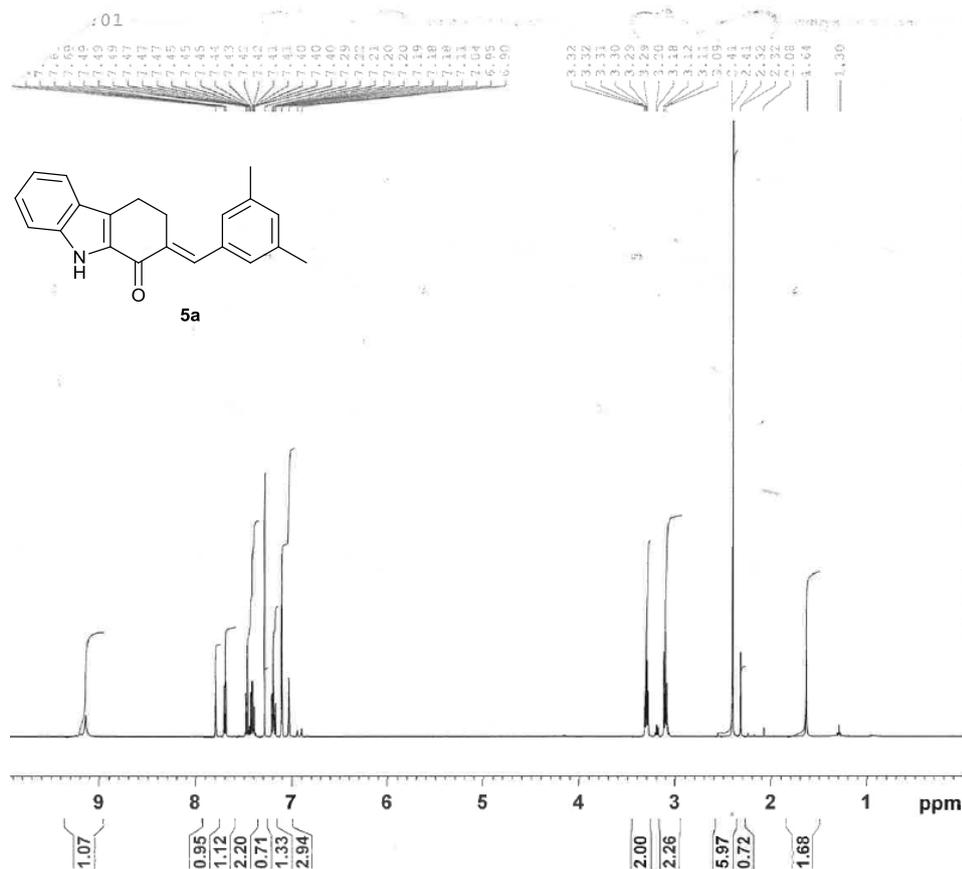
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NAME Jun10-2010-gt2760
EXPNO 11
PROCNO 1
Date_ 20100611
Time 4.29
INSTRUM AVIII400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631968 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
    
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===== CHANNEL f1 =====
NUC1 13C
P1 8.75 usec
PL1 -2.00 dB
PL1W 58.9196084 W
SF01 100.6001970 MHz

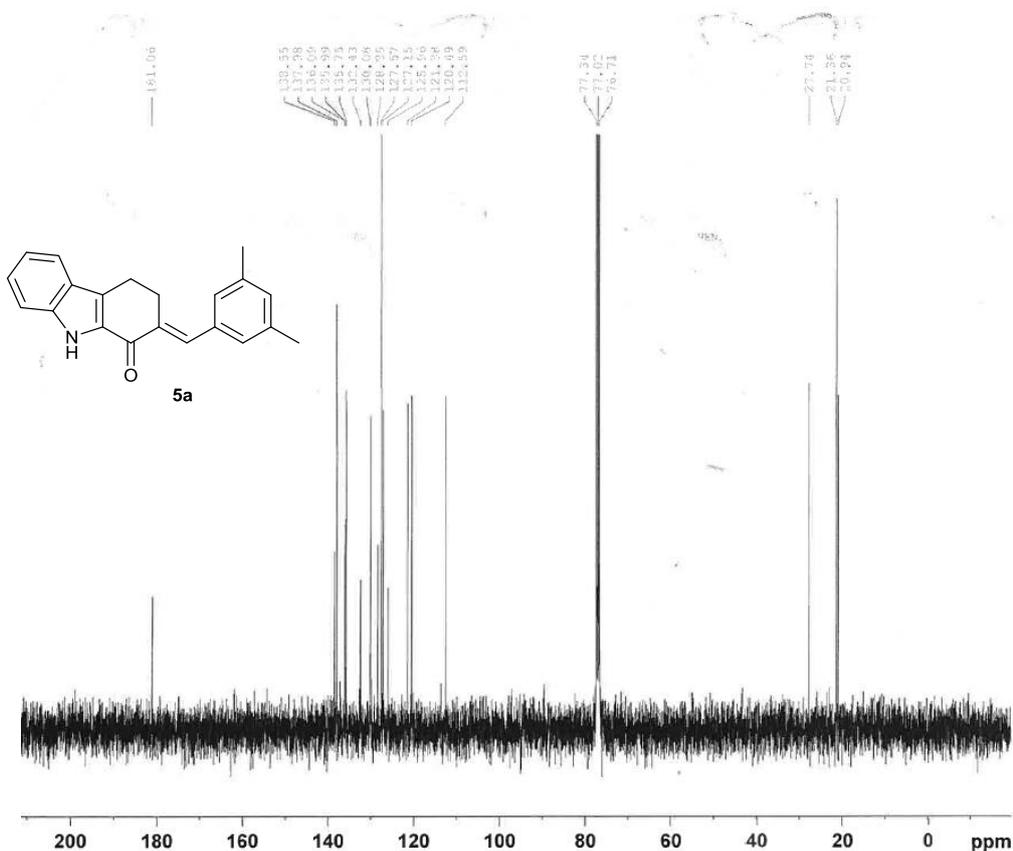
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.76 dB
PL13 14.00 dB
PL2W 9.74611950 W
PL12W 0.25753233 W
PL13W 0.12249697 W
SFO2 400.0416002 MHz
SI 65536
SF 100.5901883 MHz
WDW EM
SSB 0
LB 1.0 Hz
GB 0
PC 1.40
    
```



BRUKER

NAME Mar30-2010-GT0355
 EXPNO 11
 PROCNO 1
 Date_ 20100330
 Time_ 16.42
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 322
 DW 60.800 usec
 DE 17.24 usec
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

----- CHANNEL f1 -----
 NUC1 1H
 P1 13.00 usec
 PL1 0.00 dB
 PL1W 9.74611950 W
 SFO1 400.0424704 MHz
 SI 65536
 SF 400.0400000 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00

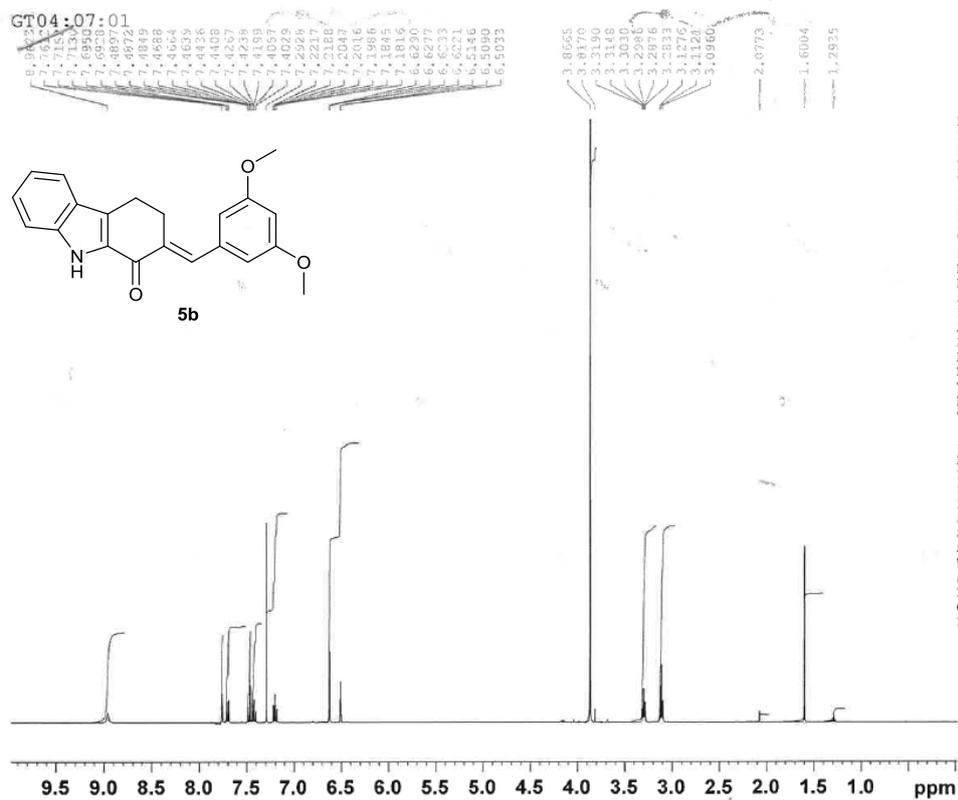


BRUKER

NAME Mar26-2010-GI0299
 EXPNO 10
 PROCNO 1
 Date_ 20100329
 Time_ 11.17
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

----- CHANNEL f1 -----
 NUC1 13C
 P1 8.75 usec
 PL1 -2.00 dB
 PL1W 58.91986084 W
 SFO1 100.6001970 MHz

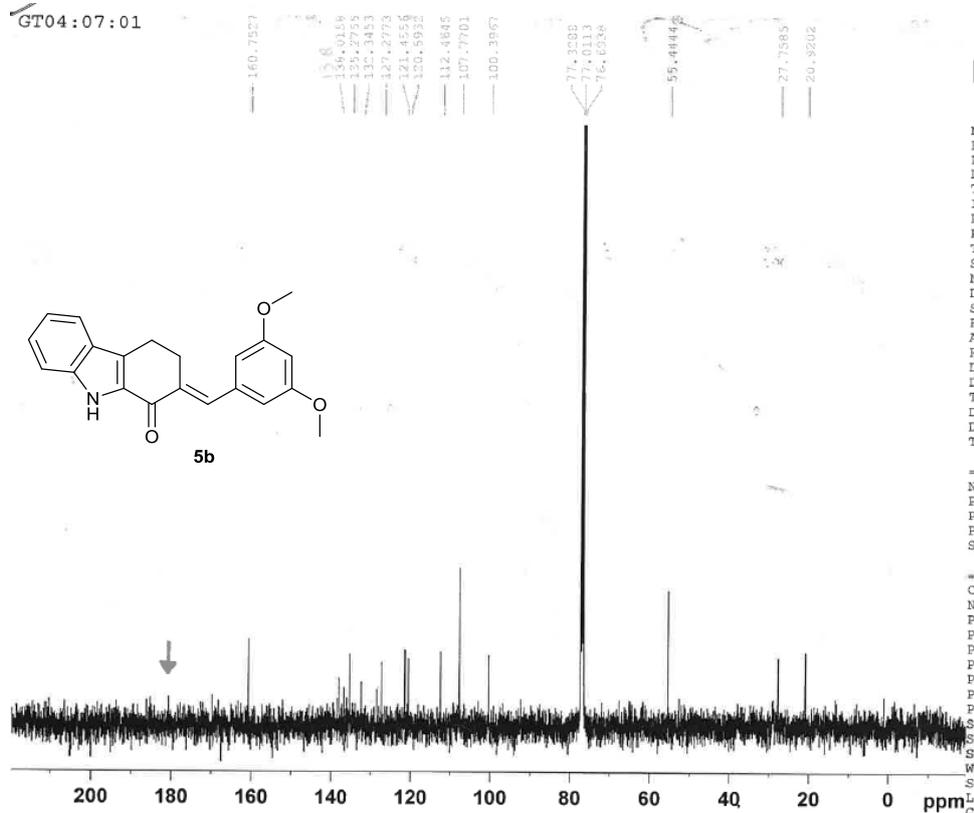
----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.78 dB
 PL13 19.00 dB
 PL2W 9.74611950 W
 PL12W 0.25753233 W
 PL13W 0.1226637 W
 CPO2 400.0416002 MHz
 SI 65536
 SF 100.5901380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



BRUKER

NAME Mar30-2010-GT0356
 EXPNO 10
 PROCNO 1
 Date_ 20100330
 Time 16.48
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 406
 DW 60.800 usec
 DE 17.24 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 0.00 dB
 PL1W 9.74611950 W
 SFO1 400.0424704 MHz
 SI 65536
 SF 400.0400000 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00



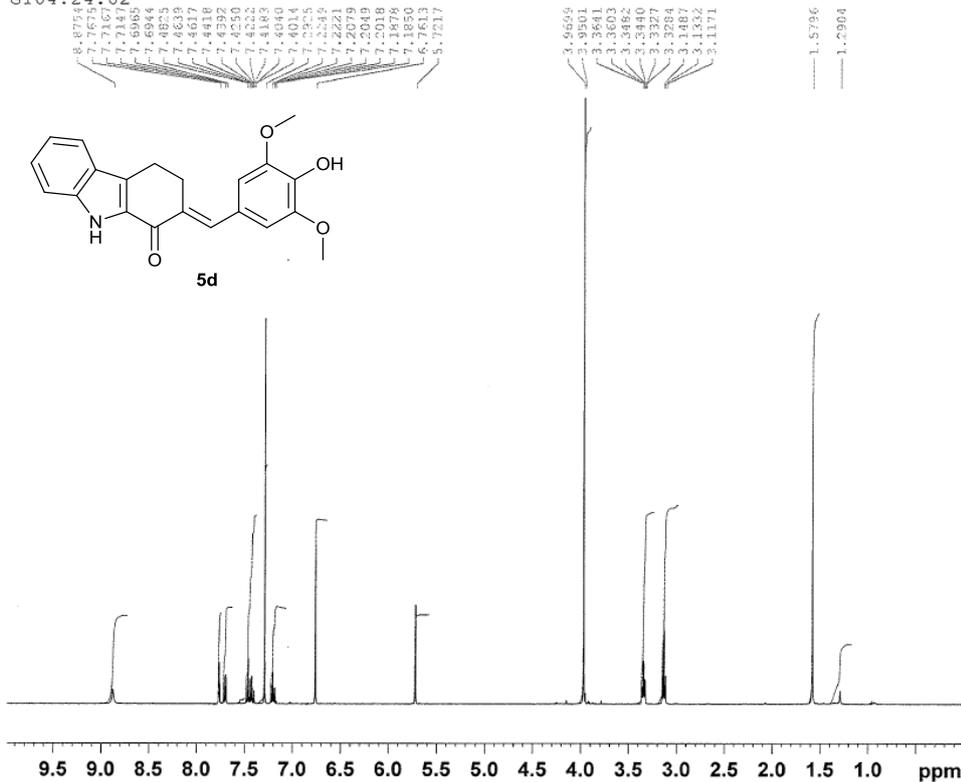
BRUKER

NAME Mar30-2010-GT0356
 EXPNO 11
 PROCNO 1
 Date_ 20100330
 Time 17.27
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.75 usec
 PL1 -2.00 dB
 PL1W 58.91986084 W
 SFO1 100.6001970 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.78 dB
 PL13 19.00 dB
 PL2W 9.74611950 W
 PL12W 0.25753233 W
 PL13W 0.12269637 W
 SFO2 400.0416002 MHz
 SI 65536
 SF 100.5901380 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

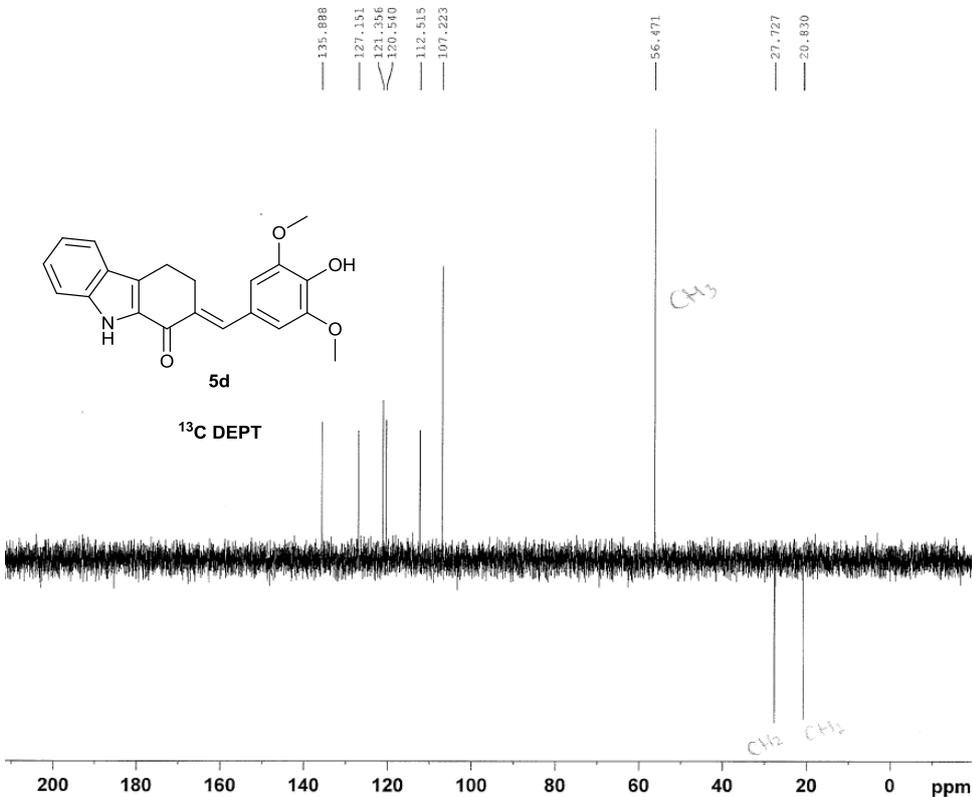
GT04:24:02



NAME May02-2010-GT1016
 EXPNO 10
 PROCNO 1
 Date_ 20100502
 Time 21:36
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQC 3.9846387 sec
 RG 456
 DW 60.800 usec
 DE 17.24 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 0.00 dB
 PL1W 9.74611950 W
 SFO1 400.0424704 MHz
 SI 65536
 SF 400.0400000 MHz
 EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00

GT04:24:02

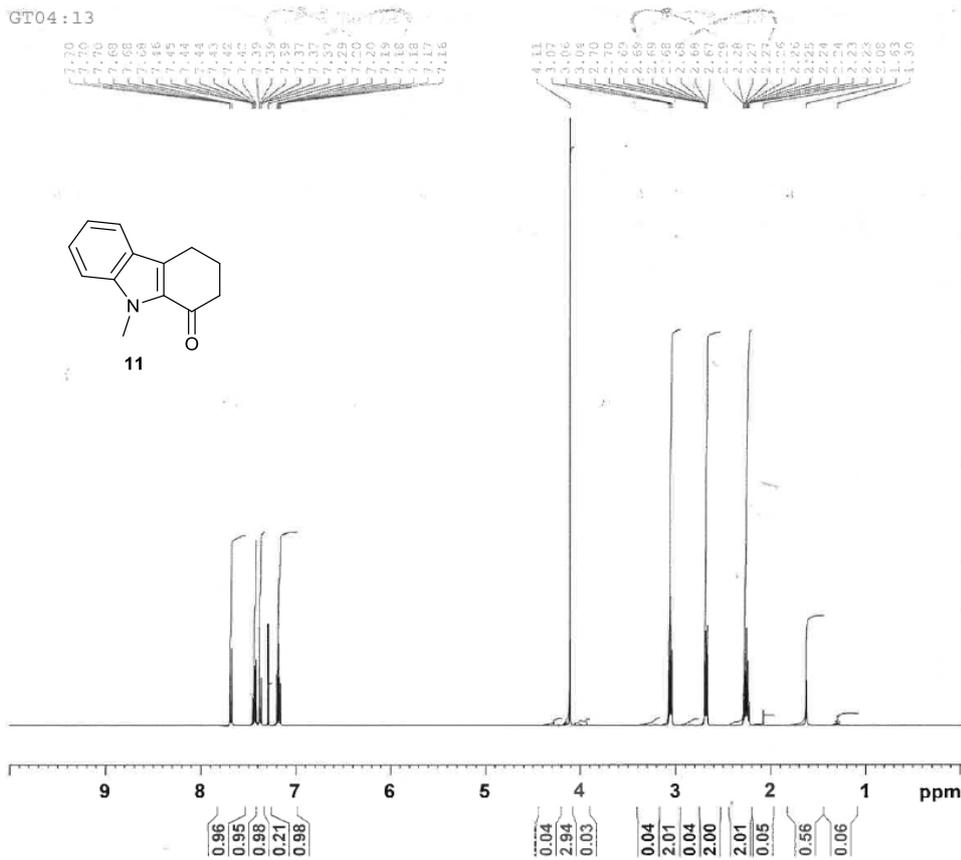
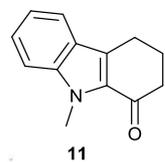


NAME Jul21-2010-GT4021
 EXPNO 12
 PROCNO 1
 Date_ 20100721
 Time 19:52
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG deptsp135
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQC 1.3631988 sec
 RG 2950
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 CNST2 145.0000000
 D1 2.00000000 sec
 D2 0.00344828 sec
 D12 0.00002000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.75 usec
 PL1 2000.00 usec
 PL0 120.00 dB
 PL1 2.00 dB
 PL1W 0.00000000 W
 PL1W 58.91986084 W
 SFO1 100.6001970 MHz
 SF2 132.00 MHz
 SFO2 400.0416002 MHz
 SI 65536
 SPNAME2 Crp60comp.4
 SFOAL2 0.500
 SFOFFS2 0.00 Hz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P3 13.00 usec
 P4 26.00 usec
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.78 dB
 PL2W 9.74611950 W
 PL12W 0.25753233 W
 SFO2 400.0416002 MHz
 SI 65536
 SF 100.5901380 MHz
 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

GT04:13

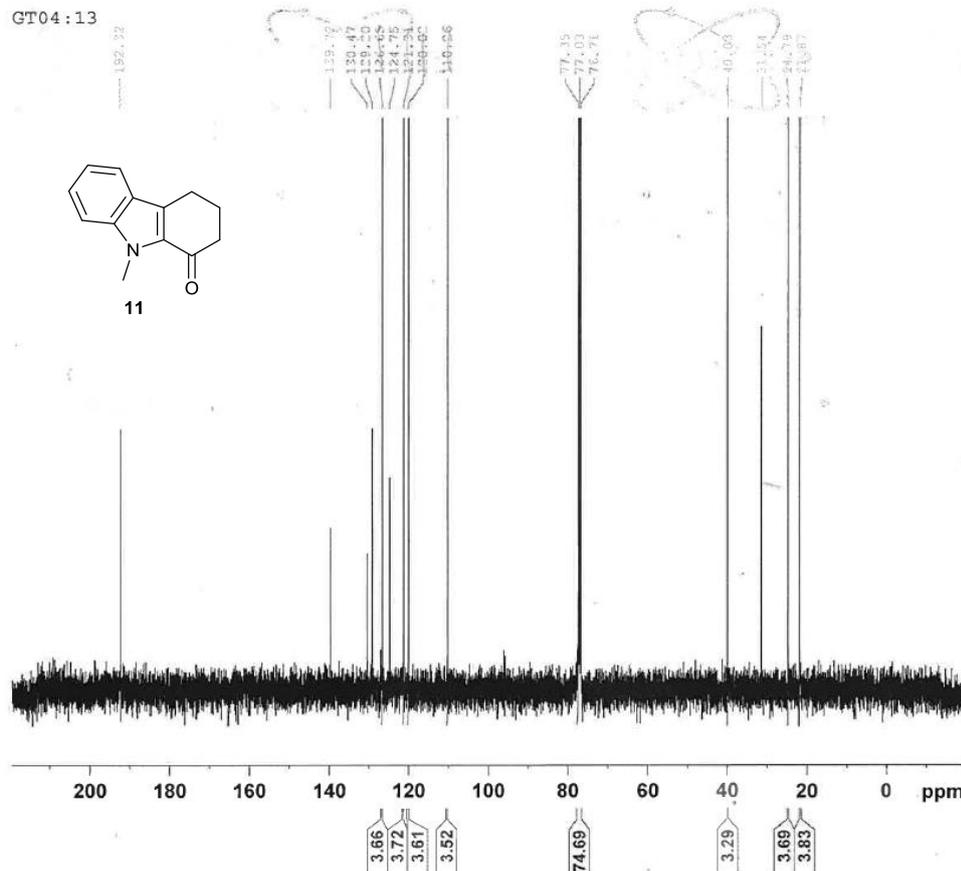
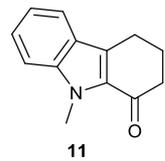


BRUKER

NAME Mar26-2010-GT0234
 EXPNO 21
 PROCNO 1
 Date_ 20100326
 Time_ 12.47
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 203
 DW 60.800 usec
 DE 17.24 usec
 TE 298.0 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 0.00 dB
 PL1W 9.74611950 W
 SFO1 400.0424704 MHz
 SI 65536
 SF 400.0400000 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00

GT04:13

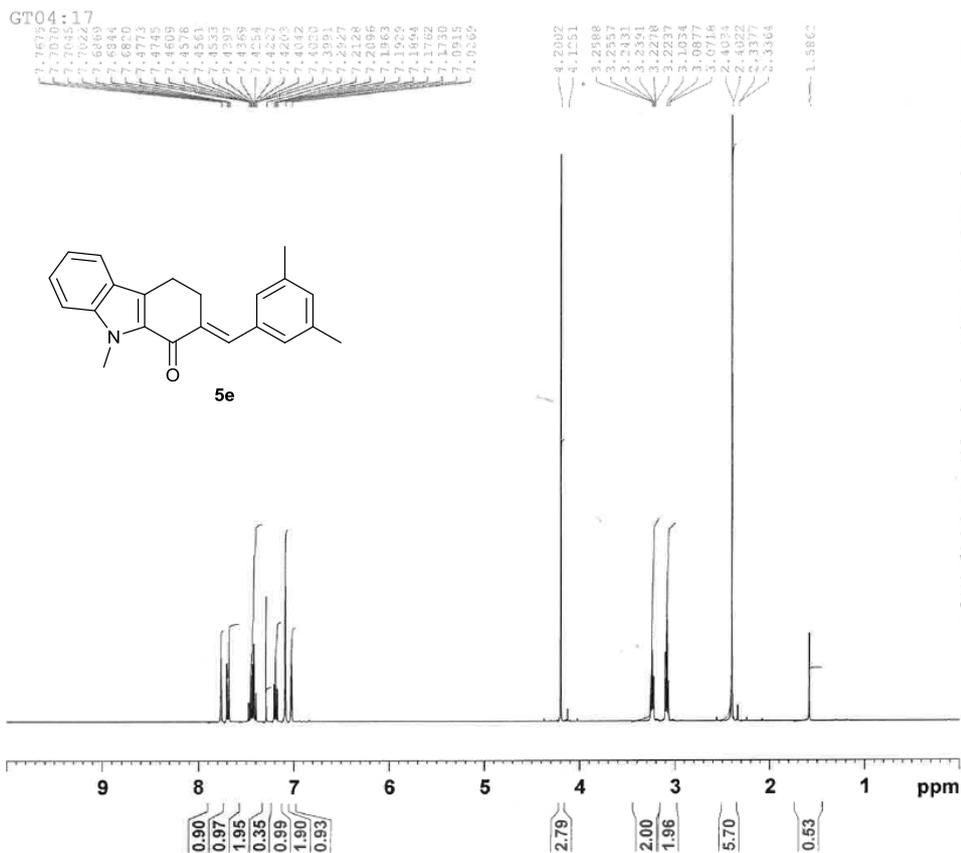


BRUKER

NAME Mar26-2010-GT0234
 EXPNO 20
 PROCNO 1
 Date_ 20100326
 Time_ 11.34
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

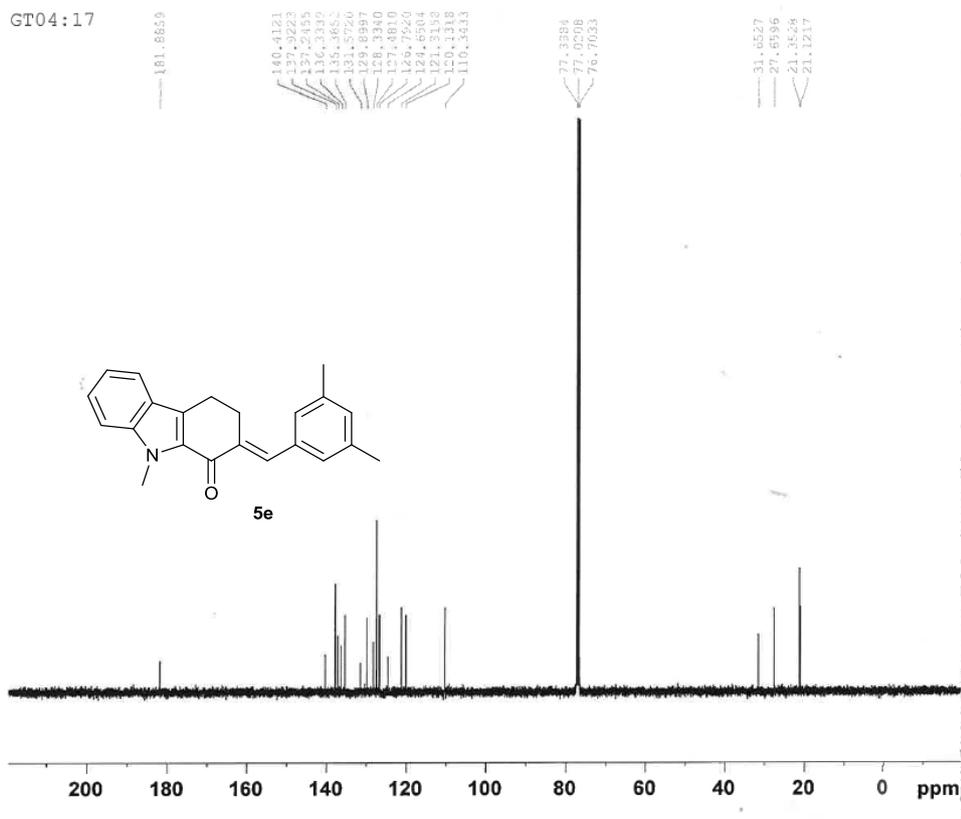
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.75 usec
 PL1 -2.00 dB
 PL1W 58.91986084 W
 SFO1 100.6001970 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.78 dB
 PL13 19.00 dB
 PL2W 9.74611950 W
 PL12W 0.25753233 W
 PL13W 0.12269637 W
 SFO2 400.0416002 MHz
 SI 65536
 SF 100.5901380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



NAME May07-2010-GT1133
 EXPNO 10
 PROCNO 1
 Date 20100507
 Time 15.22
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125493 Hz
 AQ 3.9846387 sec
 RG 228
 DW 60.800 usec
 DE 17.24 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

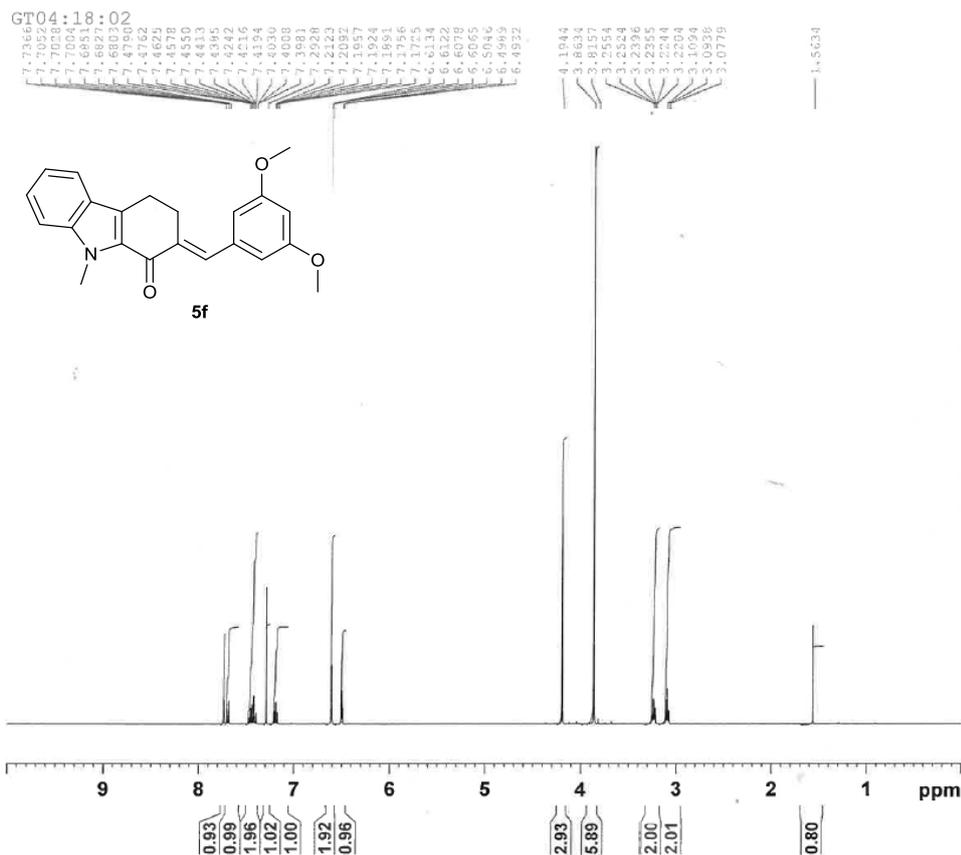
===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 0.00 dB
 PL1W 9.74611950 W
 SFO1 400.0424704 MHz
 SI 65536
 SF 400.0400000 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00



NAME May07-2010-GT1133
 EXPNO 11
 PROCNO 1
 Date 20100507
 Time 22.18
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

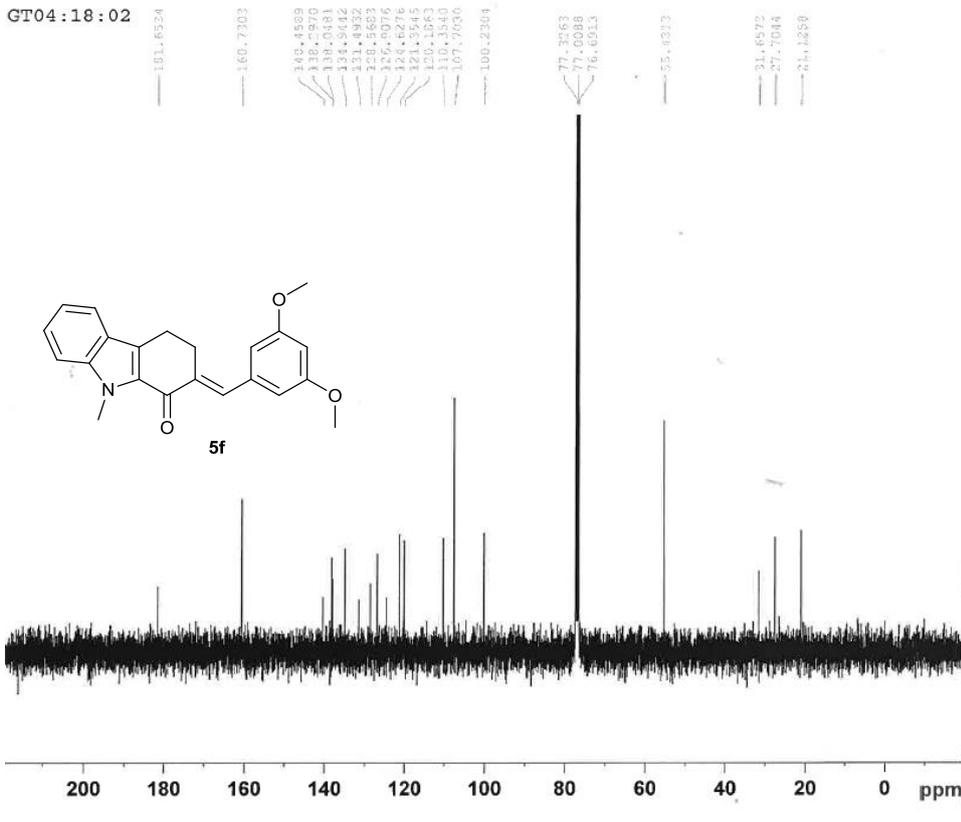
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.75 usec
 PL1 -2.00 dB
 PL1W 58.91986084 W
 SFO1 100.6001970 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.78 dB
 PL13 19.00 dB
 PL2W 9.74611950 W
 PL12W 0.25753233 W
 PL13W 0.12269637 W
 SFO2 400.0416002 MHz
 SI 65536
 SF 100.5901380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



NAME Apr01-2010-GT0430
 EXPNO 10
 PROCNO 1
 Date_ 20100401
 Time_ 11.52
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 362
 DW 60.800 usec
 DE 17.24 usec
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 0.00 dB
 PL1W 9.74611950 W
 SFO1 400.0424704 MHz
 SI 65536
 SF 400.0400000 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00



NAME Apr01-2010-GT0430
 EXPNO 11
 PROCNO 1
 Date_ 20100401
 Time_ 12.37
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.75 usec
 PL1 -2.00 dB
 PL1W 58.91986084 W
 SFO1 100.6001970 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.78 dB
 PL13 19.00 dB
 PL2W 9.74611950 W
 PL12W 0.25753233 W
 PL13W 0.12269637 W
 SFO2 400.0416002 MHz
 SI 65536
 SF 100.5901380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

COMPARE analyses

Results of the Standard COMPARE analysis of GI₅₀ values of compound **4c** (NSC 756592) with Synthetic Compounds.

Rank	Correlation	namecode	Target Vector ident For Display	Target Vector descript	Common Cell Lines
1	1	--DISCREE	NSC:S756592 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
2	0.842	--DISCREE	NSC:S756590 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	compound 5g	58
3	0.795	PUBLIC	NSC:S106969 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	CENTAUREIDIN	52
4	0.769	PUBLIC	NSC:S675003 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		54
5	0.725	PUBLIC	NSC:S750873 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		55
6	0.724	PUBLIC	NSC:S748541 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
7	0.723	PUBLIC	NSC:S748533 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
8	0.722	PUBLIC	NSC:S609397 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	B817373K328	56
9	0.715	--DISCREE	NSC:S756586 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
10	0.713	PUBLIC	NSC:S648581 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		57
11	0.7	PUBLIC	NSC:S119754 Endpt:GI50 Expld:AVGDATA hiConc:-4.9	COPTISINE CHLORIDE	57
12	0.7	PUBLIC	NSC:S686560 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
13	0.698	PUBLIC	NSC:S656160 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		53
14	0.697	PUBLIC	NSC:S659853 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	PANZEM NCD	58
15	0.695	PUBLIC	NSC:S638389 Endpt:GI50 Expld:AVGDATA hiConc:2.2		48
16	0.694	PUBLIC	NSC:S695588 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	AMINO MMI-S02 CI	55
17	0.683	PUBLIC	NSC:S680185 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		53
18	0.679	PUBLIC	NSC:S653008 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		41
19	0.679	PUBLIC	NSC:S673787 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		52
20	0.671	PUBLIC	NSC:S720716 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		57
21	0.671	PUBLIC	NSC:S645645 Endpt:GI50 Expld:AVGDATA hiConc:-6.0	ANTINEOPLASTIC-6456	55
22	0.671	PUBLIC	NSC:S712304 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		51
23	0.669	PUBLIC	NSC:S751957 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
24	0.659	PUBLIC	NSC:S643813 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
25	0.657	PUBLIC	NSC:S681683 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
26	0.646	PUBLIC	NSC:S698666 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	Indanocine 1	54
27	0.646	PUBLIC	NSC:S642198 Endpt:GI50 Expld:AVGDATA hiConc:-7.0	ANTINEOPLASTIC-6421	57
28	0.645	PUBLIC	NSC:S645646 Endpt:GI50 Expld:AVGDATA hiConc:-6.0	ANTINEOPLASTIC-6456	57
29	0.643	PUBLIC	NSC:S113764 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	U 32027	58
30	0.642	PUBLIC	NSC:S671178 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		45
31	0.64	PUBLIC	NSC:S656157 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		54
32	0.639	PUBLIC	NSC:S642321 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		44
33	0.634	PUBLIC	NSC:S667048 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-ETHYL ESTRADIOL	53
34	0.632	PUBLIC	NSC:S689466 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	DESMETHOXYCENTAUF	56
35	0.631	PUBLIC	NSC:S657275 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
36	0.631	PUBLIC	NSC:S671618 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-iodoestradiol	57
37	0.629	PUBLIC	NSC:S642321 Endpt:GI50 Expld:AVGDATA hiConc:-6.0		43
38	0.628	PUBLIC	NSC:S684423 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		57
39	0.626	PUBLIC	NSC:S649977 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		46
40	0.626	PUBLIC	NSC:S174589 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
41	0.625	PUBLIC	NSC:S643812 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	ANTINEOPLASTIC-6438	58
42	0.624	PUBLIC	NSC:S667047 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-PROPENYL ESTRADIC	57
43	0.623	PUBLIC	NSC:S123399 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
44	0.621	PUBLIC	NSC:S682429 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-(1-PROPYNYL)-3,17.B	57
45	0.62	PUBLIC	NSC:S702400 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		57
46	0.619	PUBLIC	NSC:S652809 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		54
47	0.616	PUBLIC	NSC:S281300 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		49
48	0.615	PUBLIC	NSC:S720872 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		54
49	0.61	PUBLIC	NSC:S671041 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-N-PROPOXYESTRADI	58
50	0.605	PUBLIC	NSC:S136513 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	DIBENZOYLFURAN DER	58

Results of the Standard COMPARE analysis of GI₅₀ values of compound **5c** (NSC 756586) with Synthetic Compounds.

Rank	Correlation	namecode	Target Vector id	For Display	Target Vector descriptor For	Common Cell Lines
1	1	--DISCREET	NSC:S756586	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
2	0.846	--DISCREET	NSC:S756590	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	compound 5g	60
3	0.788	PUBLIC	NSC:S240579	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
4	0.781	PUBLIC	NSC:S748533	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
5	0.744	PUBLIC	NSC:S642321	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		45
6	0.724	PUBLIC	NSC:S686560	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
7	0.722	PUBLIC	NSC:S638389	Endpt:GI50 Expld:AVGDATA hiConc:2.2		49
8	0.721	PUBLIC	NSC:S667049	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-ETHENYL ESTRADIOL	53
9	0.715	--DISCREET	NSC:S756592	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
10	0.705	PUBLIC	NSC:S643813	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
11	0.7	PUBLIC	NSC:S659853	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	PANZEM NCD	60
12	0.697	PUBLIC	NSC:S709581	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		51
13	0.695	PUBLIC	NSC:S703321	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
14	0.685	PUBLIC	NSC:S671043	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
15	0.683	PUBLIC	NSC:S681683	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
16	0.674	PUBLIC	NSC:S709566	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		52
17	0.674	PUBLIC	NSC:S675003	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
18	0.674	PUBLIC	NSC:S645620	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		46
19	0.672	PUBLIC	NSC:S123399	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
20	0.67	PUBLIC	NSC:S671041	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-N-PROPOXYESTRADIOL	60
21	0.669	PUBLIC	NSC:S297360	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		50
22	0.662	PUBLIC	NSC:S710266	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
23	0.662	PUBLIC	NSC:S642198	Endpt:GI50 Expld:AVGDATA hiConc:-7.0	ANTINEOPLASTIC-642198	59
24	0.66	PUBLIC	NSC:S642321	Endpt:GI50 Expld:AVGDATA hiConc:-6.0		44
25	0.659	PUBLIC	NSC:S648581	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
26	0.653	PUBLIC	NSC:S643812	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	ANTINEOPLASTIC-643812	60
27	0.652	PUBLIC	NSC:S647246	Endpt:GI50 Expld:AVGDATA hiConc:-6.0	ANTINEOPLASTIC-647246	56
28	0.652	PUBLIC	NSC:S625857	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		48
29	0.651	PUBLIC	NSC:S652890	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
30	0.649	PUBLIC	NSC:S645646	Endpt:GI50 Expld:AVGDATA hiConc:-6.0	ANTINEOPLASTIC-645646	58
31	0.646	PUBLIC	NSC:S641526	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	4'-HYDROXY-3',8-DIMETHOXY	44
32	0.645	PUBLIC	NSC:S682429	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-(1-PROPYNYL)-3,17.BETA.-	59
33	0.637	PUBLIC	NSC:S628949	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		45
34	0.635	PUBLIC	NSC:S683125	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
35	0.635	PUBLIC	NSC:S131734	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
36	0.634	PUBLIC	NSC:S673787	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		54
37	0.634	PUBLIC	NSC:S625863	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		46
38	0.634	PUBLIC	NSC:S709567	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		53
39	0.633	PUBLIC	NSC:S674256	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
40	0.631	PUBLIC	NSC:S402590	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
41	0.63	PUBLIC	NSC:S717356	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
42	0.63	PUBLIC	NSC:S709568	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		53
43	0.63	PUBLIC	NSC:S748541	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
44	0.625	PUBLIC	NSC:S667048	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-ETHYL ESTRADIOL	54
45	0.625	PUBLIC	NSC:S716893	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
46	0.624	PUBLIC	NSC:S667047	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-PROPENYL ESTRADIOL	59
47	0.622	PUBLIC	NSC:S179485	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	BREVICID	59
48	0.622	PUBLIC	NSC:S671042	Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-I-PROPOXYESTRADIOL	60
49	0.621	PUBLIC	NSC:S119754	Endpt:GI50 Expld:AVGDATA hiConc:-4.9	COPTISINE CHLORIDE	59
50	0.62	PUBLIC	NSC:S653564	Endpt:GI50 Expld:AVGDATA hiConc:-4.0		41

Results of the Standard COMPARE analysis of GI₅₀ values of compound **5g** (NSC 756590) with Synthetic Compounds.

Rank	Correlation	namecode	Target Vector ident For Display	Target Vector descriptor For Display	Common Cell Lines
1	1	--DISCREE	NSC:S756590 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
2	0.846	--DISCREE	NSC:S756586 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	compound 5c	60
3	0.842	--DISCREE	NSC:S756592 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	compound 4c	58
4	0.813	PUBLIC	NSC:S748533 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
5	0.785	PUBLIC	NSC:S675003 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
6	0.728	PUBLIC	NSC:S609397 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	B817373K328	58
7	0.724	PUBLIC	NSC:S751957 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
8	0.724	PUBLIC	NSC:S682429 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-(1-PROPYNYL)-3,17.BETA.-ESTRADIOL	59
9	0.718	PUBLIC	NSC:S748541 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
10	0.717	PUBLIC	NSC:S119754 Endpt:GI50 Expld:AVGDATA hiConc:-4.9	COPTISINE CHLORIDE	59
11	0.709	PUBLIC	NSC:S680185 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		55
12	0.698	PUBLIC	NSC:S671167 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
13	0.698	PUBLIC	NSC:S686560 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
14	0.697	PUBLIC	NSC:S674263 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
15	0.691	PUBLIC	NSC:S720872 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
16	0.691	PUBLIC	NSC:S695588 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	AMINO MMI-SO2 Cl	57
17	0.69	PUBLIC	NSC:S679431 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-ETHOXY-6-KETO-ESTRADIOL	59
18	0.69	PUBLIC	NSC:S681684 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
19	0.684	PUBLIC	NSC:S671169 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
20	0.683	PUBLIC	NSC:S106969 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	CENTAUREIDIN	54
21	0.68	PUBLIC	NSC:S710266 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
22	0.679	PUBLIC	NSC:S667048 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-ETHYL ESTRADIOL	54
23	0.678	PUBLIC	NSC:S683125 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		56
24	0.675	PUBLIC	NSC:S659853 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	PANZEM NCD	60
25	0.671	PUBLIC	NSC:S645645 Endpt:GI50 Expld:AVGDATA hiConc:-6.0	ANTINEOPLASTIC-645645	57
26	0.67	PUBLIC	NSC:S667049 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-ETHENYL ESTRADIOL	53
27	0.664	PUBLIC	NSC:S671041 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-N-PROPOXYESTRADIOL	60
28	0.663	PUBLIC	NSC:S673787 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		54
29	0.662	PUBLIC	NSC:S669229 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-TRANS-N-BUTEN-1-YL-ESTRADIOL	57
30	0.661	PUBLIC	NSC:S638389 Endpt:GI50 Expld:AVGDATA hiConc:2.2		49
31	0.66	PUBLIC	NSC:S653008 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		42
32	0.659	PUBLIC	NSC:S678473 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
33	0.659	PUBLIC	NSC:S674256 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
34	0.659	PUBLIC	NSC:S671618 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	2-IODOESTRADIOL	59
35	0.659	PUBLIC	NSC:S684423 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
36	0.658	PUBLIC	NSC:S671165 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
37	0.658	PUBLIC	NSC:S720716 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
38	0.656	PUBLIC	NSC:S665694 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
39	0.656	PUBLIC	NSC:S689466 Endpt:GI50 Expld:AVGDATA hiConc:-4.0	DESMETHOXYCENTAUREIDIN	58
40	0.655	PUBLIC	NSC:S240579 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
41	0.654	PUBLIC	NSC:S681683 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
42	0.654	PUBLIC	NSC:S51361 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
43	0.653	PUBLIC	NSC:S642198 Endpt:GI50 Expld:AVGDATA hiConc:-7.0	ANTINEOPLASTIC-642198	59
44	0.651	PUBLIC	NSC:S701101 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		58
45	0.651	PUBLIC	NSC:S123399 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
46	0.65	PUBLIC	NSC:S682597 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
47	0.65	PUBLIC	NSC:S648581 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
48	0.649	PUBLIC	NSC:S652893 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		59
49	0.648	PUBLIC	NSC:S746498 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		60
50	0.645	PUBLIC	NSC:S117028 Endpt:GI50 Expld:AVGDATA hiConc:-4.0		57