

Supporting Information for

A Proposal for the Mechanism-of-Action of Diazoparaquinone Natural Products.

Ken S. Feldman* and Kyle J. Eastman

*Department of Chemistry, The Pennsylvania State University, University Park, Pennsylvania 16802
USA*

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Moisture and oxygen sensitive reactions were carried out in flame-dried glassware under an argon or nitrogen atmosphere. Benzene was distilled from sodium benzophenone ketyl under an argon atmosphere, or passed through an activated alumina column immediately before use. Toluene was distilled from sodium 9-fluorenone ketyl under argon atmosphere, or passed through an activated alumina column immediately before use. Chlorobenzene, benzonitrile, anisole, m-xylene, 1,3-dimethoxybenzene, and 1,3-cyanobenzene were purged with nitrogen for 30 min prior to use (where applicable) and otherwise used as purchased. AIBN was recrystallized from ethyl alcohol. Bu_3SnH was distilled neat and stored under nitrogen. Purification of products via flash chromatography was performed with 32-63 μm silica gel or Analtech Uniplate 1000 micron preparative thin layer chromatography plates, with the solvent systems indicated. CH_2Cl_2 , EtOAc and acetone used in flash chromatography were of HPLC grade or passed through an activated alumina column prior to use. Melting points are uncorrected. Low- and high-resolution mass spectra were obtained according to the specified technique and were performed at The Huck Institute of the Life Sciences – Proteomics and Mass Spectrometry Core Facility at The Pennsylvania State University, University Park, PA. Copies of ^1H and ^{13}C NMR spectra are supplied as criteria of purity.

General Procedure 1. Phenylation of Prekinamycin and Derivatives. AIBN (1.1 equiv) in benzene (0.06 M) was added via syringe pump addition over a period of 1 h to a stirring solution of diazoquinones **10a**, **10b** or **10c** (1 equiv) and Bu_3SnH (1.1 equiv) in benzene (0.06 M) at 80 °C. When the addition was complete, the reaction solution was allowed to cool to room temperature. After reaching room temperature the reaction mixture was concentrated in vacuo. The resulting residue was purified by flash chromatography on silica gel using the specified eluent.

General Procedure 2. Aromatic Solvent Competition Experiments. AIBN (1.1 equiv) in an equimolar solution of benzene and the appropriate aromatic solvent (0.06 M) was added via syringe pump addition over a period of 1 h to a stirring solution of diazoquinone **10c** (1 equiv) and Bu_3SnH (1.1 equiv) also in an equimolar solution of benzene and the appropriate aromatic solvent (0.06 M) at 80 °C. When the addition was

complete, the reaction solution was allowed to cool to room temperature. After reaching room temperature the reaction mixture was diluted with CH₂Cl₂ and poured onto a silica gel column and purified eluting with CH₂Cl₂ with an increasing percentage of EtOAc from 0 % to 10 %. Purification furnished the clean benzene trapped product and in most cases a mixture of *o,m,p* or the 2,4,5 isomers of the given substituted aromatic trapped product. Relative rates were quantified by product mass comparison of the substituted aromatic adducts vs. the benzene addition product. Isolation of analytical samples of pure *o*, *m* or *p* (or the 2, 4 or 5 isomers) from chromatography permitted ¹H NMR identification. In cases where isomer separation was not achieved, the ratios were determined by inspection of ¹H NMR spectra of the mixtures.

General Procedure 3. Aromatic Solvent Competition Experiments. AIBN (1.1 equiv) in an equimolar solution of benzene and the appropriate aromatic solvent (0.06 M) was added via syringe pump addition over a period of 1 h to a stirring solution of diazoquinone **10c** (1 equiv) and Bu₃SnH (1.1 equiv) also in an equimolar solution of benzene and the appropriate solvent (0.06 M) at 80 °C. When the addition was complete, the reaction solution was allowed to cool to room temperature. After reaching room temperature the reaction mixture was diluted with CH₂Cl₂ and poured onto a silica gel column. Purification eluting with CH₂Cl₂ to remove tin residues, followed by an increase in polarity to 10% acetone in CH₂Cl₂ afforded a mixture of the benzene and substituted aromatic trapped products. Relative rates were quantified by ¹H NMR integration of the substituted aromatic adducts vs. the benzene addition product. Ratio determination was made through similar comparison of the pure *o,m,p* (or the 2,4,5) isomer(s) ¹H NMR spectra to that of the mixture.

General Procedure 4. Aromatic Solvent Competition Experiments with Varying Equivalents of Tin. General Procedures 2 and 3 were both used varying only in the equivalents of Bu₃SnH as indicated in Figure 1.

4,5,9-Trihydroxy-2-Methyl-11-Phenyl-Benzo[b]fluoren-10-one (10a). Following General Procedure 1, prekinamycin **1a** (18 mg, 0.057 mmol) was converted

into benzo[b]fluorenone **10a** (12.3 mg, 59%). mp 260 °C (dec); IR (neat): 3409, 1585 cm^{-1} ; Presumably, due to the partial free radical nature of **10a** analogous to kinobscurinone,¹ **10a** appears to be “NMR silent” exhibiting neither a ^1H - nor a ^{13}C NMR signature; ESI m/z relative intensity 391(MNa^+ 100); TOFHRMS (+ESI) Calcd for $\text{C}_{24}\text{H}_{16}\text{O}_4\text{Na}$: 391.0946, Found 391.0947.

Acetic Acid 9-Acetoxy-5-Hydroxy-2-Methyl-10-oxo-11-Phenyl-10H-Benzo[b]fluoren-4-yl Ester (10b). Following General Procedure 1, diazoquinone **1b** (17.5 mg, 0.044 mmol) was converted into benzo[b]fluorenone **10b** (9.8 mg, 50%). mp 200 °C (dec); IR (neat): 3330, 1789, 1766 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 9.39 (s, 1H), 7.88 (d, $J = 7.9$ Hz, 1H), 7.56 (t, $J = 8.0$ Hz, 2H), 7.55 (d, $J = 7.7$ Hz, 1H), 7.41-7.50 (m, 3H), 7.06 (d, $J = 8.0$ Hz, 1H), 7.06 (s, 1H), 7.05 (s, 1H), 2.51 (s, 3H), 2.36 (s, 3H), 2.32 (s, 3H), ^{13}C NMR (100 MHz, CDCl_3) δ 180.2, 170.2, 166.9, 151.3, 149.9, 147.9, 144.4, 143.3, 138.5, 135.7, 133.9, 133.8, 129.7, 128.8, 128.7, 128.2, 126.5, 125.4, 123.3, 123.2, 123.0, 122.6, 114.6, 21.7, 21.6, 21.3; ESI m/z relative intensity 475 (MNa^+ 100); TOFHRMS (+ESI) Calcd for $\text{C}_{28}\text{H}_{20}\text{O}_6\text{Na}$: 475.1158, Found 475.1142.

5-Hydroxy-4,9-dimethoxy-2-methyl-11-phenyl-benzo[b]fluoren-10-one (10c). Following General Procedure 1, diazoquinone **1c** (10.5 mg, 0.032 mmol) was converted into benzo[b]fluorenone **10c** (10.0 mg, 79%). mp 220 °C (dec); IR (neat): 3181, 1633 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 10.76 (s, 1H), 7.58 (d, $J = 7.6$ Hz, 1H), 7.56 (d, $J = 6.8$ Hz, 2H), 7.48 (t, $J = 8.2$ Hz, 1H), 7.45 (t, $J = 7.1$ Hz, 2H), 7.39 (d, $J = 7.2$ Hz, 1H), 7.00 (d, $J = 8.4$ Hz, 1H), 6.81 (s, 1H), 6.67 (s, 1H), 4.09 (s, 3H), 3.90 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 182.0, 161.8, 151.9, 151.1, 146.6, 144.3, 138.7, 136.9, 135.0, 134.6, 130.0, 129.4, 128.4, 128.2, 128.1, 120.0, 119.2, 117.8, 115.0, 114.9, 115.2, 56.9, 56.6, 22.2; ESI m/z relative intensity 497 (MH^+ 50); TOFHRMS (+ESI) Calcd for $\text{C}_{26}\text{H}_{21}\text{O}_4$: 397.1440, Found 397.1443.

Acetic Acid 4,5-Diacetoxy-2-Methyl-10-oxo-11-Phenyl-10H-benzo[b]fluoren-9-yl Ester (10d). From **10a**: Pyridine (0.26 mL, 3.1 mmol) and Ac_2O (0.31 mL, 3.1 mmol) were sequentially added to a mixture of **10a** (11.0 mg, 0.031 mmol) and DMAP

(1.0 mg, 0.008 mmol) in CH₂Cl₂ (1.0 mL) at room temperature. The dark purple mixture was stirred at room temperature for 30 min, turning to a dark red/orange color. At this time, the reaction solution was diluted with saturated aqueous NaHCO₃ (10 mL) and CH₂Cl₂ (10 mL). The layers were separated and the aqueous layer was extracted with CH₂Cl₂ (3x 10 mL). The combined organic layers were washed with water and brine, dried over Na₂SO₄, filtered and concentrated. The resulting dark red solid was purified by flash chromatography on silica gel (CH₂Cl₂) to yield the tri-acetate as a bright orange solid (8.1 mg, 52%).

From **10b**: Pyridine (0.070 mL, 0.90 mmol) and Ac₂O (0.85 mL, 0.90 mmol) were sequentially added to a mixture of **10b** (4.0 mg, 0.90 mmol) and DMAP (1.0 mg, 0.008 mmol) in CH₂Cl₂ (1.0 mL) at room temperature. The dark red mixture was stirred at room temperature for 30 min, changing to a dark red/orange color. At this time, the reaction solution was diluted with saturated aqueous NaHCO₃ (10 mL) and CH₂Cl₂ (10 mL). The layers were separated and the aqueous layer was extracted with CH₂Cl₂ (3X 10 mL). The combined organic layers were washed with water and brine, dried over Na₂SO₄, filtered and concentrated. The resulting dark red solid was purified by flash chromatography on silica gel (CH₂Cl₂). The tri-acetate was obtained as a orange solid (4.4 mg, 99%). mp 195 °C (dec); IR (neat): 1769, 1635 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.59 (d, *J* = 7.8 Hz, 1H), 7.58 (d, *J* = 7.3 Hz, 1H), 7.47-7.52 (m, 4H), 7.39 (d, *J* = 8.3 Hz, 1H), 7.01 (d, *J* = 7.7 Hz, 1H), 6.99 (s, 1H), 6.80 (s, 1H), 2.53 (s, 3H), 2.43 (s, 3H), 2.31 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 179.4, 169.6, 168.7, 167.6, 154.6, 151.4, 145.9, 145.2, 142.0, 141.1, 136.5, 133.8, 132.8, 129.4, 129.3, 129.3, 128.9, 128.0, 125.9, 125.3, 124.8, 124.2, 123.7, 121.8, 21.3, 21.2, 21.1, 20.9; ESI *m/z* relative intensity 517 (MNa⁺ 100); TOFHRMS (+ESI) Calcd for C₃₀H₂₂O₇Na: 517.1263, Found 517.1245.

4,5,9-Trimethoxy-2-Methyl-11-Phenyl-Benzo[b]fluoren-10-one (10e). From **10a**: Methyl iodide (0.042 mL, 0.68 mmol) was added to a mixture of **10a** (25.0 mg, 0.068 mmol) and K₂CO₃ (93.0 mg, 0.068 mmol) in DMF (2.0 mL) at room temperature. The dark purple mixture was stirred at room temperature for 12 h, turning to a dark red color. The reaction solution was then diluted with saturated aqueous NH₄Cl (10 mL) and Et₂O (10 mL). The layers were separated and the aqueous layer was extracted with Et₂O

(3X 10 mL). The combined organic layers were washed with water and brine, dried over Na₂SO₄, filtered and concentrated. The resulting light red solid was purified by flash chromatography on silica gel (CH₂Cl₂). The tri-methylether was obtained as a bright red solid (11.5 mg, 41%).

From **10b**: Methyl iodide (0.031 mL, 0.50 mmol) was added to a mixture of **10b** (20.0 mg, 0.050 mmol) and K₂CO₃ (69.0 mg, 0.050 mmol) in DMF (1.0 mL) at room temperature. The dark red mixture was stirred at room temperature for 12 h, turning to a light red color. The reaction was then diluted with saturated aqueous NH₄Cl (10 mL) and Et₂O (10 mL). The layers were separated and the aqueous layer was extracted with Et₂O (3X 10 mL). The combined organic layers were washed with water and brine, dried over Na₂SO₄, filtered and concentrated. The resulting light red solid was purified by flash chromatography on silica gel (CH₂Cl₂). The tri-methylether was obtained as a bright red solid (12.0 mg, 59%). mp 210 °C (dec); IR (neat): 1643 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.50-7.60 (m, 2H), 7.40-7.50 (m, 5H), 6.96 (dd, *J* = 6.7, 2.7 Hz, 1H), 6.76 (s, 1H), 6.59 (s, 1H), 4.02 (s, 3H), 3.96 (s, 3H), 3.89 (s, 3H), 2.32 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 181.5, 161.6, 155.2, 153.0, 151.7, 145.3, 140.3, 138.7, 134.3, 134.2, 130.1, 129.2, 128.3, 127.8, 126.8, 121.4, 118.3, 118.2, 117.4, 113.9, 113.6, 63.9, 56.1, 55.9, 21.7; ESI *m/z* relative intensity 411 (MH⁺ 100); TOFHRMS (+ESI) Calcd for C₂₇H₂₂O₄: 411.1596, Found 411.1596.

5-Hydroxy-4,9-Dimethoxy-2-Methyl-11-p-Tolyl-Benzo[b]fluoren-10-one

(11a). Following General Procedure 2, diazoquinone **1c** (20.0 mg, 0.060 mmol) was converted into a 2.2:1 mixture of benzo[b]fluorenone **11a** (*o,m,p* = 62:23:15) (7.4 mg, 30%) and benzo[b]fluorenone **10c** (3.6 mg, 15%). (*o,m,p* mixture) IR (neat): 3190, 1633 cm⁻¹; (*o*-isomer) ¹H NMR (500 MHz, CDCl₃) δ 10.64 (s, 1H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.50 (t, *J* = 8.1 Hz, 1H), 7.22-7.36 (m, 3H), 7.19 (d, *J* = 7.3 Hz, 1H), 7.01 (d, *J* = 8.3 Hz, 1H), 6.69 (s, 1H), 6.54 (s, 1H), 4.11 (s, 3H), 3.89 (s, 3H), 2.31 (s, 3H), 2.16 (s, 3H); (*o,m,p* mixture) ¹H NMR (500 MHz, CDCl₃) δ 10.76 (s, 0.4H, *m,p*), 10.68 (s, 0.6H, *o*) 7.60 (d, *J* = 7.7 Hz, 0.5H), 7.59 (d, *J* = 7.4 Hz, 0.5H), 7.47-7.51 (m, 1H), 7.33-7.36 (m, 1H), 7.22-7.30 (m, 2H), 7.19 (d, *J* = 6.8 Hz, 1H), 7.01 (d, *J* = 8.3 Hz, 1H), 6.85 (s, 0.25H), 6.79 (s, 0.25H), 6.69 (s, 1H), 6.54 (s, .5H), 4.11 (s, 1.8H, *o*), 4.10 (s, 1.2H, *m,p*),

3.90 (s, 1.2H, *m,p*) 3.89 (s, 1.8H, *o*), 2.35 (s, 1.8H, *m,p*), 2.31 (s, 1.8H, *o*), 2.16 (s, 3H); (*o,m,p* mixture) ^{13}C NMR (125 MHz, CDCl_3) δ 181.5, 161.3, 151.5, 150.6, 145.9, 144.2, 138.5, 136.7, 136.5, 135.1, 134.0, 129.9, 129.5, 128.5, 127.6, 126.6, 125.4, 121.3, 119.4, 118.9, 118.6, 117.4, 114.6, 114.4, 111.1, 56.53, 56.5, 56.5, 21.8, 21.7, 19.9; ESI m/z relative intensity 411 (MH^+ 30); TOFHRMS (+ESI) Calcd for $\text{C}_{27}\text{H}_{22}\text{O}_4$: 411.1596, Found 411.1611.

11-(4-Chloro-Phenyl)-5-Hydroxy-4,9-Dimethoxy-2-Methyl-Benzo[b]fluoren-10-one (11b). Following General Procedure 2, diazoquinone **1c** (20.0 mg, 0.060 mmol) was converted into a 1.5:1 mixture of benzo[b]fluorenone **11b** (*o,m,p* = 48:32:20) (9.5 mg, 37%) and benzo[b]fluorenone **10c** (5.0 mg, 21%). **11b** isomers were separated via preparative TLC (20% EtOAc in benzene). (*o*-isomer) mp 280 °C (dec); (*o*-isomer) IR (neat): 3178, 1630 cm^{-1} ; (*o*-isomer) ^1H NMR (500 MHz, CDCl_3) δ 10.76 (s, 1H), 7.60 (d, $J = 7.8$ Hz, 1H), 7.50 (m, 2H), 7.33 (m, 3H), 7.02 (d, $J = 8.2$ Hz, 1H), 6.69 (s, 1H), 6.58 (s, 1H), 4.11 (s, 3H), 3.90 (s, 3H), 2.33 (s, 3H); (*m*-isomer) mp 260 °C (dec); (*m*-isomer) IR (neat): 3378, 1630 cm^{-1} ; (*m*-isomer) ^1H NMR (500 MHz, CDCl_3) δ 10.82 (s, 1H), 7.60 (d, $J = 8.2$ Hz, 1H), 7.51 (m, 2H), 7.44 (d, $J = 6.8$ Hz, 1H), 7.38 (t, $J = 7.8$ Hz, 1H), 7.37 (s, 1H), 7.30 (d, $J = 8.3$ Hz, 1H), 6.75 (s, 1H), 6.71 (s, 1H), 4.12 (s, 3H), 3.91 (s, 3H), 2.36 (s, 3H); (*p*-isomer) mp 240 °C (dec); (*p*-isomer) IR (neat): 3166, 1631 cm^{-1} ; (*p*-isomer) ^1H NMR (500 MHz, CDCl_3) δ 10.81 (s, 1H), 7.60 (d, $J = 8.4$ Hz, 1H), 7.52 (d, $J = 8.6$ Hz, 2H), 7.50 (t, $J = 8.2$ Hz, 1H), 7.42 (d, 8.6 Hz, 2H), 7.30 (d, $J = 8.4$ Hz, 1H), 6.79 (s, 1H), 6.71 (s, 1H), 4.12 (s, 3H), 3.92 (s, 3H), 2.35 (s, 3H); (*o,m,p*-mixture) ^{13}C NMR (125 MHz, CDCl_3) δ 181.6, 181.5, 181.3, 161.3, 151.5, 151.4, 151.3, 151.1, 144.5, 143.5, 143.3, 142.3, 138.5, 136.6, 136.5, 136.4, 134.1, 133.8, 133.7, 133.2, 131.1, 130.7, 130.3, 129.5, 129.2, 129.1, 128.9, 128.3, 128.1, 127.9, 126.5, 121.1, 119.6, 119.5, 119.3, 118.5, 118.4, 118.3, 117.5, 114.7, 114.4, 114.2, 111.2, 111.1, 56.6, 56.5, 56.2, 56.1, 21.8, 21.7; ESI m/z relative intensity 431 (MH^+ 100); TOFHRMS (+ESI) Calcd for $\text{C}_{26}\text{H}_{20}\text{O}_4\text{Cl}$: 431.1050, Found 431.1060.

4-(5-Hydroxy-4,9-Dimethoxy-2-Methyl-10-oxo-10H-Benzo[b]fluoren-11-yl)-Benzonitrile (11c). Following General Procedure 2, diazoquinone **1c** (20.0 mg, 0.060

mmol) was converted into a 2.2:1 mixture of benzo[b]fluorenone **11c** (*o,m,p* = 43:25:32) (13.0 mg, 51%) and benzo[b]fluorenone **10c** (5.6 mg, 23%). **11c** isomers were separated via preparative TLC (20% EtOAc in benzene). (*o*-isomer) mp 265 °C (dec); (*o*-isomer) IR (neat): 3394, 2232, 1631 cm⁻¹; (*o*-isomer) ¹H NMR (500 MHz, CDCl₃) δ 10.87 (s, 1H), 7.77 (d, *J* = 7.6 Hz, 1H), 7.66 (t, *J* = 7.7 Hz, 1H), 7.61 (d, *J* = 7.9 Hz, 1H), 7.52 (t, *J* = 8.2 Hz, 1H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 1H), 7.03 (d, *J* = 8.2 Hz, 1H), 6.71 (s, 1H), 6.58 (s, 1H), 4.13 (s, 3H), 3.92 (s, 3H), 2.34 (s, 3H); (*m*-isomer) mp 265 °C (dec); (*m*-isomer) IR (neat): 3412, 2216, 1633 cm⁻¹; (*m*-isomer) ¹H NMR (500 MHz, CDCl₃) δ 10.88 (s, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.81 (s, 1H), 7.67 (d, *J* = 7.7 Hz, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.54 (t, *J* = 8.0 Hz, 1H), 7.47 (t, *J* = 8.1 Hz, 1H), 7.04 (d, *J* = 7.7 Hz, 1H), 6.72 (s, 1H), 6.70 (s, 1H), 4.13 (s, 3H), 3.93 (s, 3H), 2.37 (s, 3H); (*p*-isomer) mp 265 °C (dec); (*p*-isomer) IR (neat): 3412, 2223, 1631 cm⁻¹; (*p*-isomer) ¹H NMR (500 MHz, CDCl₃) δ 10.92 (s, 1H), 7.73 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.5 Hz, 2H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.52 (t, *J* = 8.2 Hz, 1H), 7.04 (d, *J* = 8.3 Hz, 1H), 6.72 (s, 1H), 6.71 (s, 1H), 4.13 (s, 3H), 3.92 (s, 3H), 2.36 (s, 3H); (*o,m,p*-mixture) ¹³C NMR (125 MHz, CDCl₃) δ 181.7, 181.6, 181.4, 161.5, 161.4, 152.4, 152.1, 151.6, 151.5, 143.1, 143.0, 142.9, 139.9, 139.6, 138.7, 138.6, 138.5, 136.4, 136.3, 136.2, 134.4, 133.0, 132.9, 132.3, 131.7, 131.5, 130.8, 130.4, 130.0, 129.7, 128.8, 127.9, 121.0, 119.5, 118.3, 118.1, 118.0, 117.9, 117.8, 115.2, 114.4, 114.3, 114.2, 113.1, 112.1, 111.4, 111.3, 111.3, 56.6, 56.5, 56.3, 56.3, 56.2, 21.8; ESI *m/z* relative intensity 444 (MNa⁺ 100); TOFHRMS (+ESI) Calcd for C₂₇H₁₉NO₄Na: 444.1212, Found 444.1215.

5-Hydroxy-4,9-Dimethoxy-11-(4-Methoxy-Phenyl)-2-Methyl-Benzo[b]fluoren-10-one (11d). Following General Procedure 2, diazoquinone **1c** (20.0 mg, 0.060 mmol) was converted into a 3.2:1 mixture of benzo[b]fluorenone **11d** (*o,m,p* = 76:16:12) (14.5 mg, 57%) and benzo[b]fluorenone **10c** (4.3 mg, 18%). (*o*-isomer) mp 210 °C (dec); (*o*-isomer) IR (neat): 3182, 1632 cm⁻¹; (*o*-isomer) ¹H NMR (500 MHz, CDCl₃) δ 10.70 (s, 1H), 7.58 (d, *J* = 7.9 Hz, 1H), 7.48 (t, *J* = 8.1 Hz, 1H), 7.36 (t, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 7.7 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 7.01 (d, *J* = 8.2 Hz, 1H), 7.0 (d, *J* = 8.3 Hz, 1H), 6.66 (s, 1H), 6.65 (s, 1H), 4.10 (s, 3H), 3.89 (s, 3H), 3.73 (s, 3H), 2.32 (s, 3H); (*o*-isomer) ¹³C NMR (125 MHz, CDCl₃) δ 181.3, 161.2, 157.4, 151.4, 150.4,

144.0, 142.8, 138.1, 136.5, 133.8, 130.7, 130.0, 129.2, 124.3, 121.6, 120.4, 119.4, 118.7, 117.3, 114.7, 114.6, 111.3, 110.9, 56.5, 56.1, 55.7, 21.8; (*m,p*-isomer) IR (neat): 3178, 2216, 1622 cm^{-1} ; (*m,p*-isomer) ^1H NMR (500 MHz, CDCl_3) δ 10.78 (s, 0.43H, *p*), 10.76 (s, 0.57H, *m*), 7.59 (d, $J = 7.3$ Hz, 1H), 7.58 (d, $J = 8.4$ Hz, 1H), 7.49 (t, $J = 8.0$ Hz, 1H), 7.37 (t, $J = 8.0$ Hz, 1H), 7.14 (d, $J = 7.6$ Hz, 0.5H), 6.98-7.06 (m, 2H), 6.93 (d, $J = 8.6$ Hz, 0.5H), 6.88 (s, 0.5H), 6.82 (s, 0.5H), 6.69 (s, 1H), 4.10 (s, 3H), 3.91 (s, 3H), 3.88 (s, 1.5H), 3.88 (s, 1.5H), 2.35 (s, 1.5H), 2.34 (s, 1.5H); (*p*-isomer) IR (neat): 3412, 2223, 1631 cm^{-1} ; (*m,p*-isomer) ^{13}C NMR (125 MHz, CDCl_3) δ 181.6, 181.3, 161.4, 161.2, 159.6, 159.2, 157.4, 151.4, 150.8, 150.4, 150.2, 149.4, 146.2, 145.8, 144.0, 142.8, 138.6, 138.2, 136.5, 136.4, 136.2, 136.1, 134.0, 133.8, 133.7, 131.2, 130.7, 130.0, 129.2, 128.9, 128.7, 128.3, 126.6, 124.3, 122.1, 122.0, 121.7, 121.6, 120.4, 119.8, 119.4, 118.9, 118.8, 118.7, 117.4, 117.3, 114.8, 114.7, 114.6, 114.4, 113.7, 113.6, 113.2, 111.3, 111.1, 110.9, 110.5, 56.5, 56.4, 56.1, 56.0, 55.7, 55.3, 55.2, 21.8, 21.7; ESI m/z relative intensity 449 ($\text{MNa}^+ 85$); TOFHRMS (+ESI) Calcd for $\text{C}_{27}\text{H}_{22}\text{O}_5\text{Na}$: 449.1365, Found 449.1346.

11-(3,5-Dimethyl-phenyl)-5-Hydroxy-4,9-Dimethoxy-2-Methyl-

Benzo[b]fluoren-10-one (11e). Following General Procedure 2, diazoquinone **1c** (20.0 mg, 0.060 mmol) was converted into a 3.14:1 mixture of benzo[b]fluorenone **11e** (2:4:5 = 50:50:0) (11.5 mg, 44%) and benzo[b]fluorenone **10c** (3.4 mg, 14%). **11e** isomers were separated via preparative TLC (1% EtOAc in CH_2Cl_2). (4-isomer) mp 220 $^\circ\text{C}$ (dec); (4-isomer) IR (neat): 3195, 1633 cm^{-1} ; (4-isomer) ^1H NMR (500 MHz, CDCl_3) δ 10.67 (s, 1H), 7.59 (d, $J = 7.3$ Hz, 1H), 7.49 (t, $J = 8.0$ Hz, 1H), 7.12 (s, 1H), 7.09 (d, $J = 7.7$ Hz, 1H), 7.05 (d, $J = 7.7$ Hz, 1H), 7.00 (d, $J = 8.2$ Hz, 1H), 6.68 (s, 1H), 6.58 (s, 1H), 4.11 (s, 3H), 3.89 (s, 3H), 2.37 (s, 3H), 2.30 (s, 3H), 2.13 (s, 3H); (4-isomer) ^{13}C NMR (125 MHz, CDCl_3) δ 181.5, 161.3, 151.4, 150.4, 146.1, 144.3, 138.4, 137.1, 136.5, 136.5, 133.9, 131.9, 130.8, 129.9, 128.4, 126.1, 121.4, 119.4, 118.6, 117.3, 114.5, 114.4, 111.1, 56.5, 56.1, 21.7, 21.3, 19.8; (2-isomer) mp 250 $^\circ\text{C}$ (dec); (2-isomer) IR (neat): 3194, 1633 cm^{-1} ; (2-isomer) ^1H NMR (500 MHz, CDCl_3) δ 10.64 (s, 1H), 7.60 (d, $J = 7.5$ Hz, 1H), 7.49 (t, $J = 7.8$ Hz, 1H), 7.18 (t, $J = 8.1$ Hz, 1H), 7.10 (d, $J = 7.6$ Hz, 2H), 7.01 (d, $J = 8.1$ Hz, 1H), 6.69 (s, 1H), 6.45 (s, 1H), 4.12 (s, 3H), 3.89 (s, 3H), 2.30 (s, 3H), 2.04 (s, 6H); (2-isomer) ^{13}C NMR (125 MHz, CDCl_3) δ 181.5, 161.3, 151.5, 150.3, 145.9, 143.3,

138.6, 136.6, 135.9, 134.8, 134.0, 130.8, 130.0, 127.1, 127.0, 121.1, 119.5, 118.0, 117.3, 114.5, 111.2, 56.4, 56.1, 20.7 20.1; ESI m/z relative intensity 425 (MH^+ 50); TOFHRMS (+ESI) Calcd for $C_{28}H_{25}O_4$: 425.1753, Found 425.1740.

11-(3,5-Dimethoxy-Phenyl)-5-Hydroxy-4,9-Dimethoxy-2-Methyl-

Benzo[b]fluoren-10-one (11f). Following General Procedure 2, diazoquinone **1c** (20.0 mg, 0.060 mmol) was converted into a 4.2:1 mixture of benzo[b]fluorenone **11f** (2:4:5 = 31:69:0) (16.2 mg, 59%) and benzo[b]fluorenone **10c** (3.4 mg, 14%). (4-isomer) mp 190 °C (dec); (4-isomer) IR (neat): 3195, 1633 cm^{-1} ; (4-isomer) 1H NMR (500 MHz, $CDCl_3$) δ 10.68 (s, 1H), 7.57 (d, $J = 7.6$ Hz, 1H), 7.46 (t, $J = 8.2$ Hz, 1H), 7.31 (d, $J = 8.9$ Hz, 1H), 6.99 (d, $J = 8.3$ Hz, 1H), 6.70 (s, 1H), 6.65 (s, 1H), 6.59 (m, 2H), 4.08 (s, 3H), 3.89 (s, 3H), 3.87 (s, 3H), 3.72 (s, 3H), 2.32 (s, 3H); (4-isomer) ^{13}C NMR (125 MHz, $CDCl_3$) δ 181.3, 161.2, 160.9, 158.9, 151.4, 150.0, 144.1, 142.8, 138.0, 136.5, 133.6, 131.5, 128.3, 121.8, 119.5, 118.8, 117.2, 116.8, 114.7, 114.5, 110.9, 104.5, 99.0, 56.5, 56.1, 55.6, 55.4, 21.8; (2-isomer) mp 180 °C (dec); (2-isomer) IR (neat): 3190, 1633 cm^{-1} ; (2-isomer) 1H NMR (500 MHz, $CDCl_3$) δ 10.80 (s, 1H), 7.58 (d, $J = 8.1$ Hz, 1H), 7.47 (t, $J = 8.1$ Hz, 1H), 7.30 (t, $J = 8.3$ Hz, 1H), 6.99 (d, $J = 8.2$ Hz, 1H), 6.65 (d, $J = 8.5$ Hz, 2H), 6.64 (s, 1H), 6.57 (s, 1H), 4.08 (s, 3H), 3.89 (s, 3H), 3.68 (s, 6H), 2.30 (s, 3H); (2-isomer) ^{13}C NMR (125 MHz, $CDCl_3$) δ 181.1, 161.1, 158.5, 151.5, 150.0, 144.0, 130.4, 138.1, 136.7, 136.3, 133.6, 129.1, 121.6, 119.5, 118.5, 117.2, 114.9, 114.5, 113.7, 113.3, 110.9, 110.6, 104.3, 56.2, 56.1, 56.0, 21.8; ESI m/z relative intensity 457 (MH^+ 30); TOFHRMS (+ESI) Calcd for $C_{28}H_{25}O_6$: 457.1651, Found 457.1653.

5-(5-Hydroxy-4,9-Dimethoxy-2-Methyl-10-oxo-10H-Benzo[b]fluoren-11-yl)-

Isophthalonitrile (11g). Due to the insolubility of 1,3-dicyanobenzene in benzene at 80 °C, the following experiment was run at a 13:1 ratio of benzene : 1,3-dicyanobenzene. Solid AIBN (11.0 mg, 0.066 mmol) was added portionwise over a period of 1 h to a stirring solution of diazoquinone **10c** (20.0 mg, 0.060 mmol), 1,3-dicyanobenzene (0.11g, .86 mmol) and Bu_3SnH (0.018 mL, 0.066 mmol) in benzene (1.0 mL, 11 mmol) at 80 °C. When the addition was complete, the reaction solution was allowed to cool to room temperature. After reaching room temperature the reaction mixture was diluted with

CH₂Cl₂ and purified with flash column chromatography, eluting with an increasing percentage of EtOAc from 0 % to 10 % in CH₂Cl₂. Purification furnished a 1:2.2 mixture of benzo[b]fluorenone **11g** (2:4:5 = 24:76:0) (6.5 mg, 24%) and benzo[b]fluorenone **10c** (12.4 mg, 52%). The ratio of **11g** : **10c**, ratioed up to equimolar amounts of benzene and 1,3-dicyanobenzene, is calculated to be 6.0:1. **11g** isomers were separated via preparative TLC (20% EtOAc in benzene). (4-isomer) mp 270 °C (dec); (4-isomer) IR (neat): 3213, 2526, 1633 cm⁻¹; (4-isomer) ¹H NMR (500 MHz, CDCl₃) δ 10.97 (s, 1H), 8.04 (s, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.63 (d, *J* = 8.1 Hz, 1H), 7.61 (d, *J* = 7.3 Hz, 1H), 7.53 (t, *J* = 8.1 Hz, 1H), 7.06 (d, *J* = 8.1 Hz, 1H), 6.72 (s, 1H), 6.54 (s, 1H), 4.13 (s, 3H), 3.93 (s, 3H), 2.35 (s, 3H); (4-isomer) ¹³C NMR (125 MHz, CDCl₃) δ 181.4, 161.5, 153.7, 151.7, 144.6, 141.8, 138.9, 137.4, 136.2, 136.2, 135.3, 134.8, 131.4, 131.3, 120.58, 119.6, 118.1, 117.2, 117.1, 116.2, 115.6, 115.0, 114.0, 112.3, 111.5, 56.6, 56.3, 21.8; (2-isomer) mp 290 °C (dec); (2-isomer) IR (neat): 3213, 2238, 1631 cm⁻¹; (2-isomer) ¹H NMR (500 MHz, CDCl₃) δ 10.99 (s, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.60 (t, *J* = 7.9 Hz, 1H), 7.53 (d, *J* = 8.2 Hz, 1H), 7.06 (d, *J* = 8.4 Hz, 2H) 6.72 (s, 1H), 6.48 (s, 1H), 4.13 (s, 3H), 3.93 (s, 3H), 2.34 (s, 3H); (2-isomer) ¹³C NMR (125 MHz, CDCl₃) δ 161.5, 153.9, 151.7, 144.4, 141.5, 138.8, 136.5, 136.3, 135.0, 134.8, 132.2, 131.9, 128.4, 120.5, 119.6, 118.2, 117.1, 116.4, 115.5, 115.0, 114.1, 111.6, 56.6, 56.4, 21.9; ESI m/z relative intensity 469 (MNa⁺ 100); TOFHRMS (+ESI) Calcd for C₂₈H₁₈N₂O₄Na: 469.1164, Found 469.1156.

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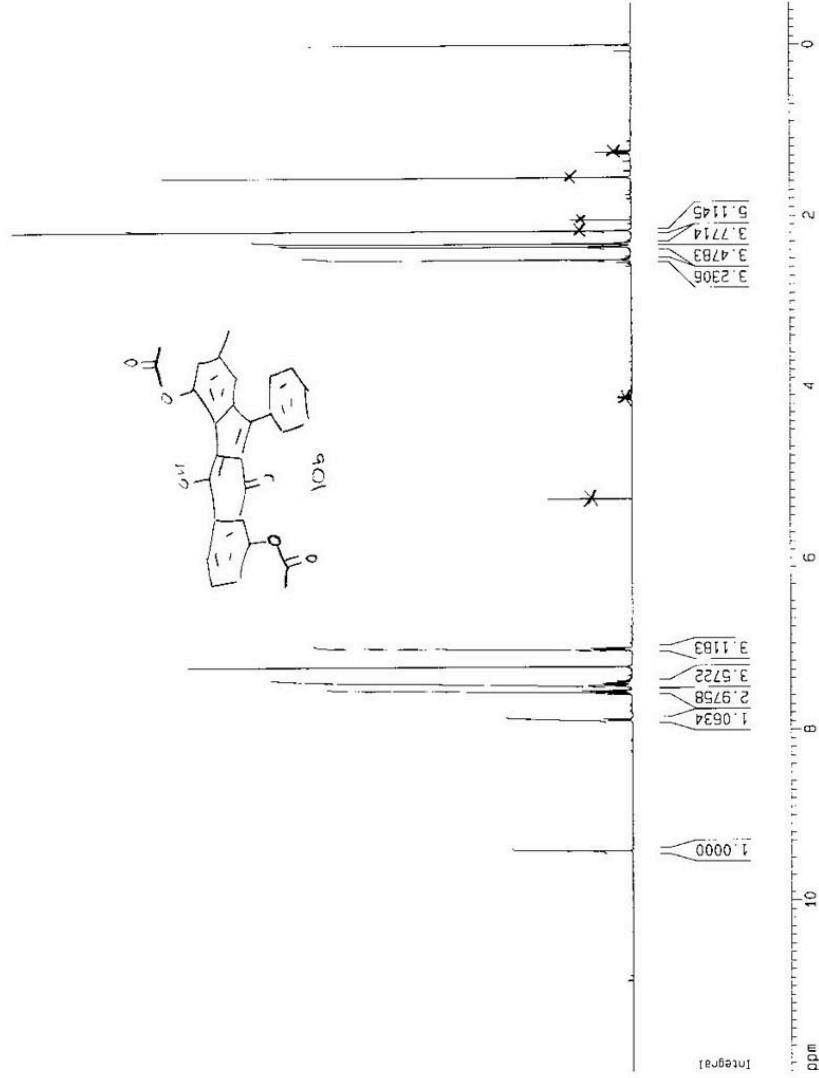
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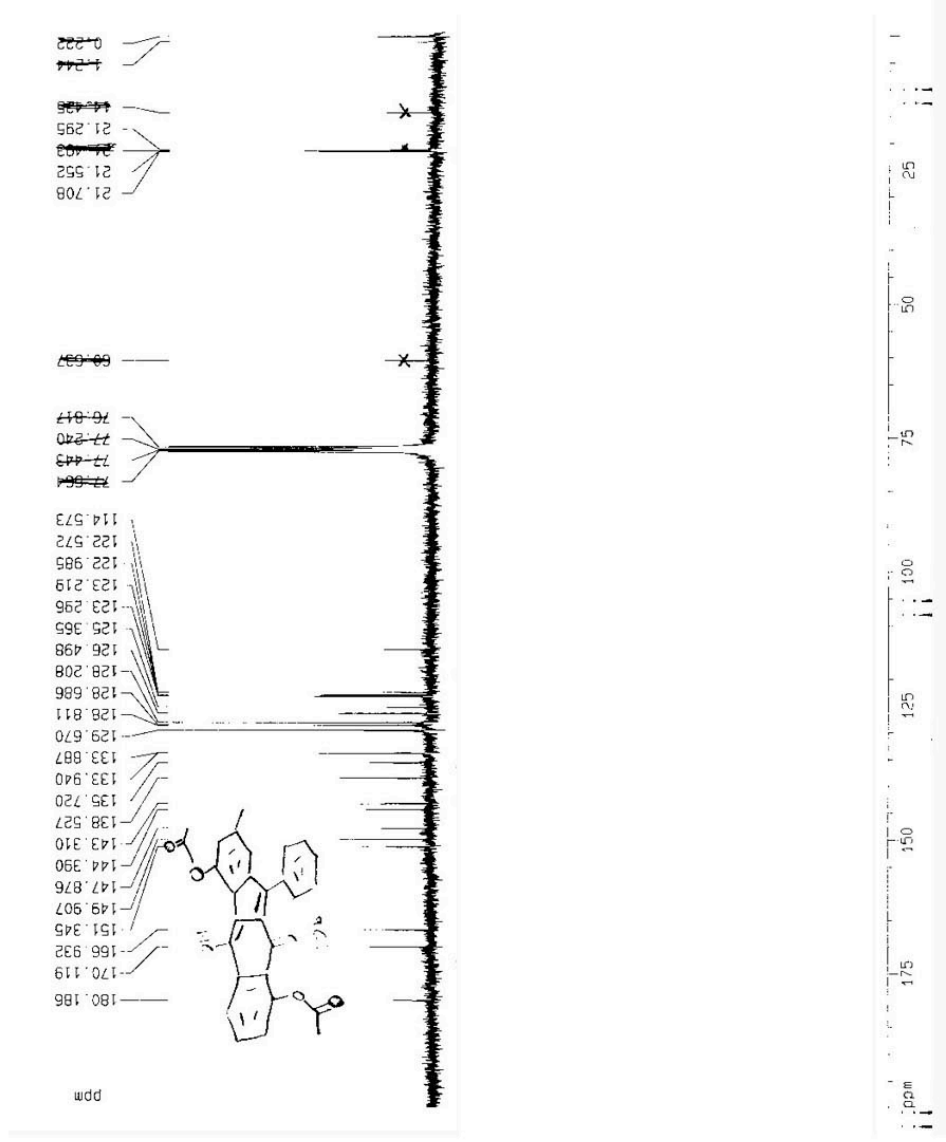
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1D NMR plot parameters

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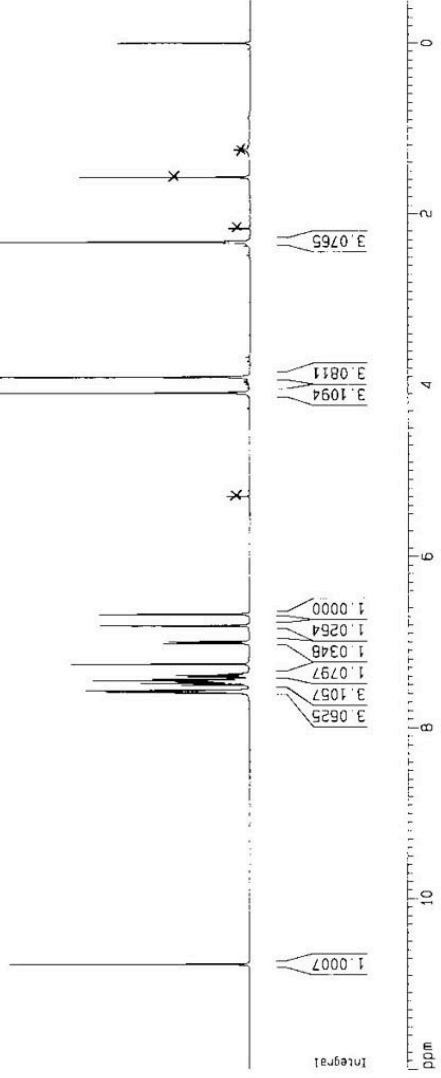
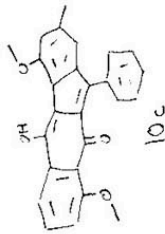
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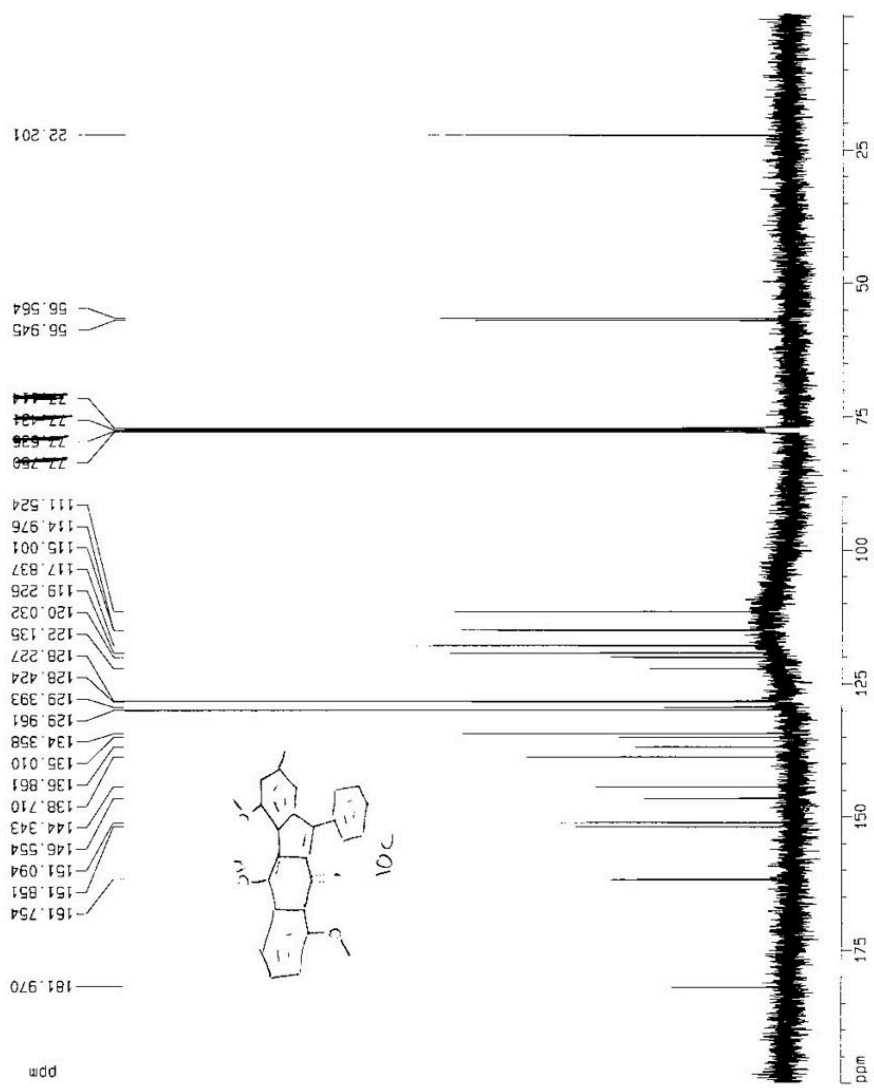
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1D NMR plot parameters
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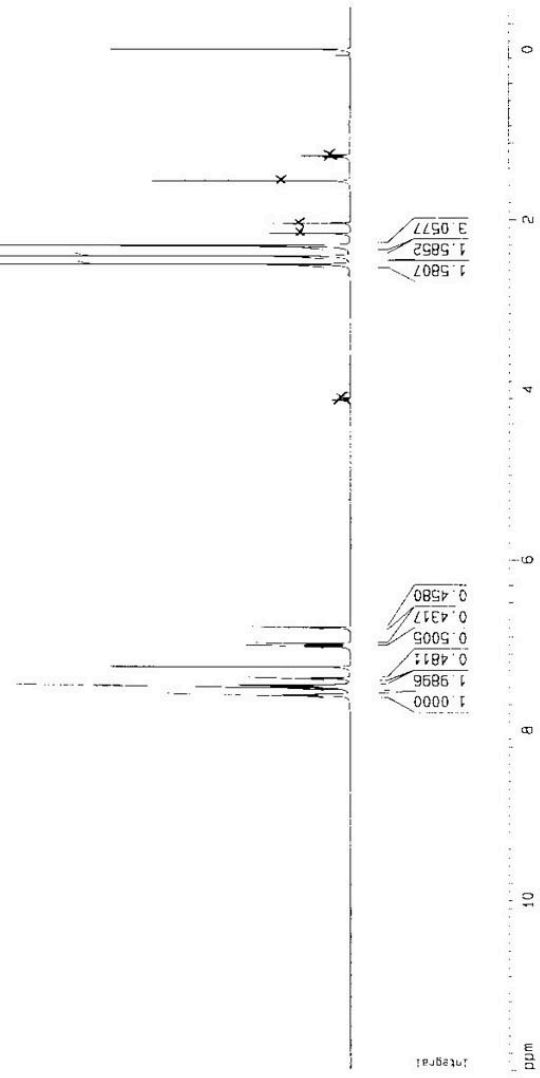
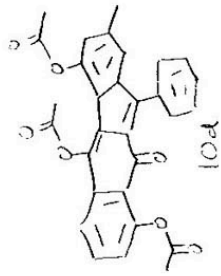


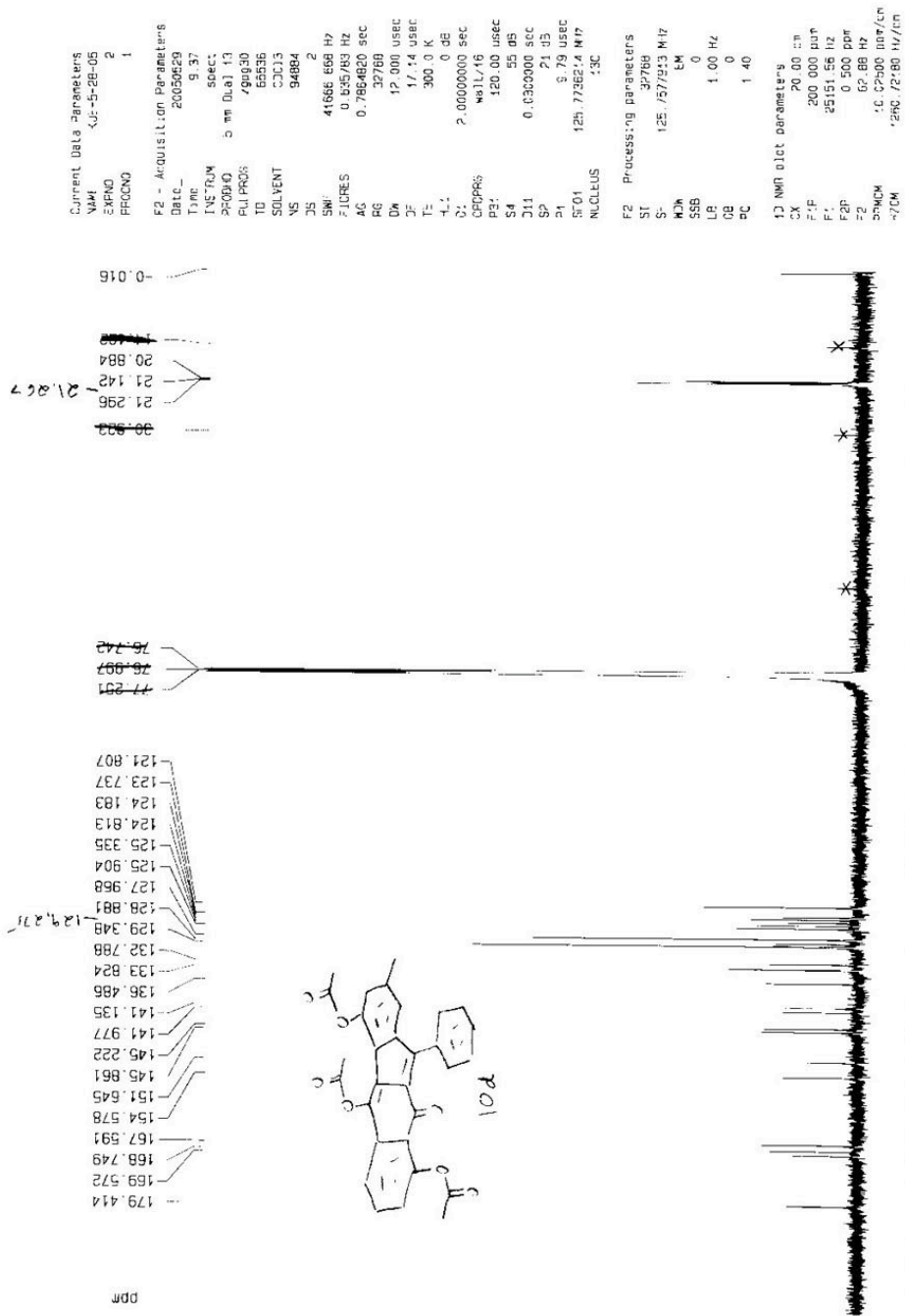
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13 NMR plot parameters
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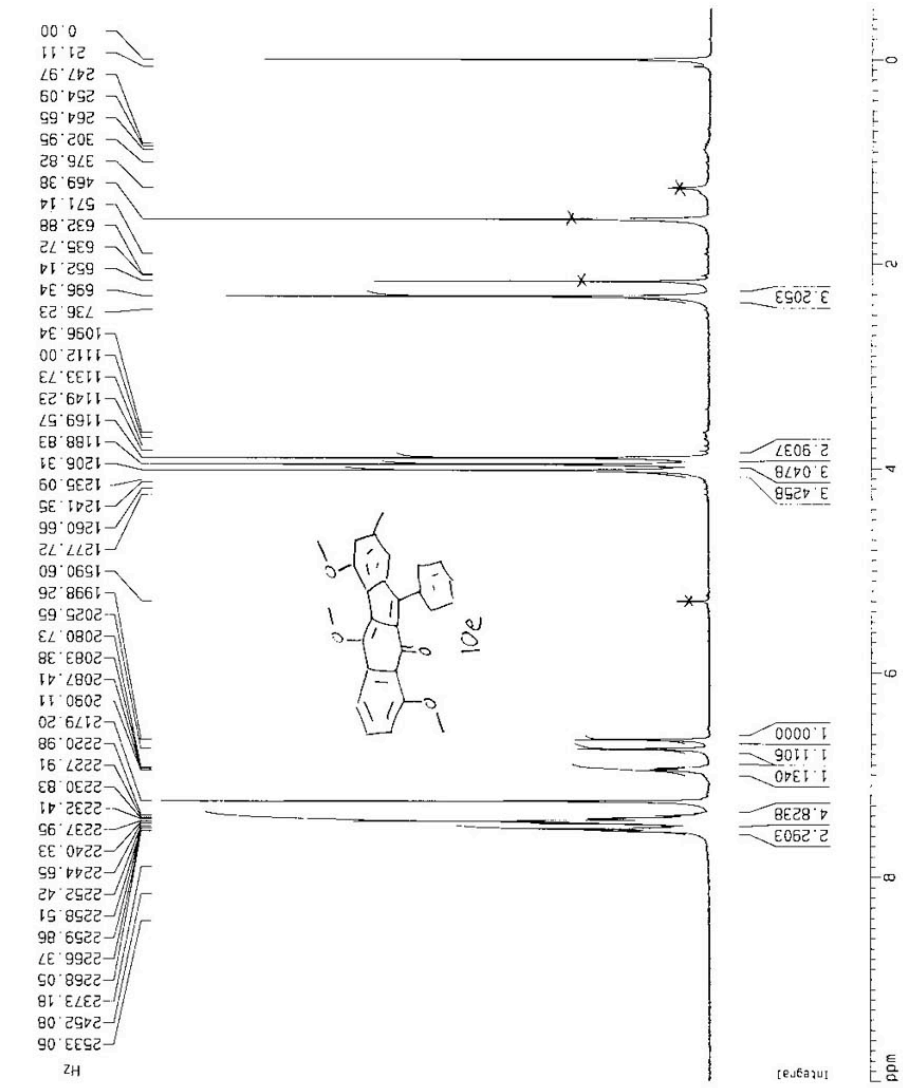
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1D NMR plot parameters
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Current Data Parameters
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SOLVENT  DMS-D6
NS       11454
DS       4
SWH      18832.364 Hz
FIDRES   0.267360 Hz
AQ       1.740336 sec
RG       6135
DM       26.856 usec
DE       6.00 usec
TE       300.0 K
D1       2.0000000 sec
d11      0.0300000 sec
d12      0.00002000 sec

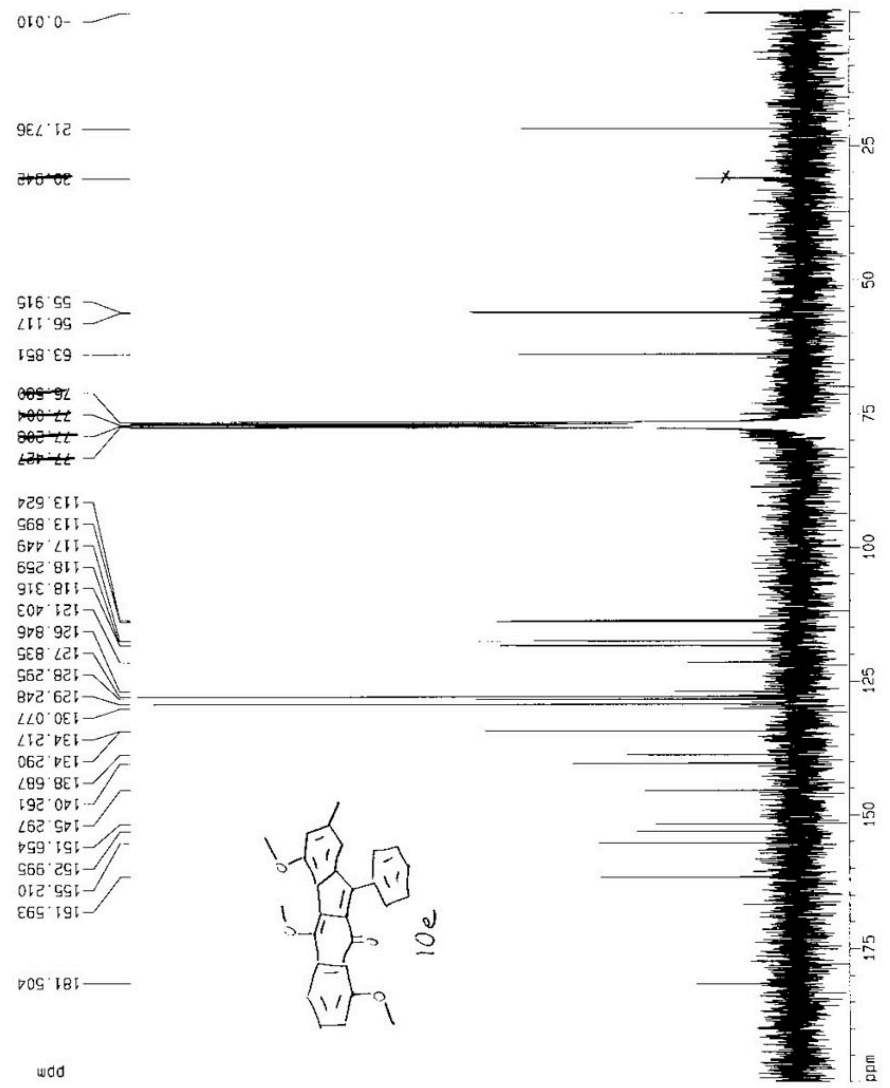
***** CHANNEL f1 *****
NUC1     13C
P1       11.80 usec
PL1      0.00 dB
SF01     75.4760200 MHz

***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2     1H
PCPD2    140.00 usec
PL2      0.00 dB
PL12     17.50 dB
PL13     17.50 dB
SF02     300.1312005 MHz

F2 - Processing parameters
SI       32768
SF       75.4677503 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40

1D NMR plot parameters
CX       20.00 cm
F1P      200.000 ppm
F2P      15093.50 Hz
F3P      -37.70 ppm
PRGCM    10.02500 Hz/cm
HZCM     755.56421 Hz/cm

```

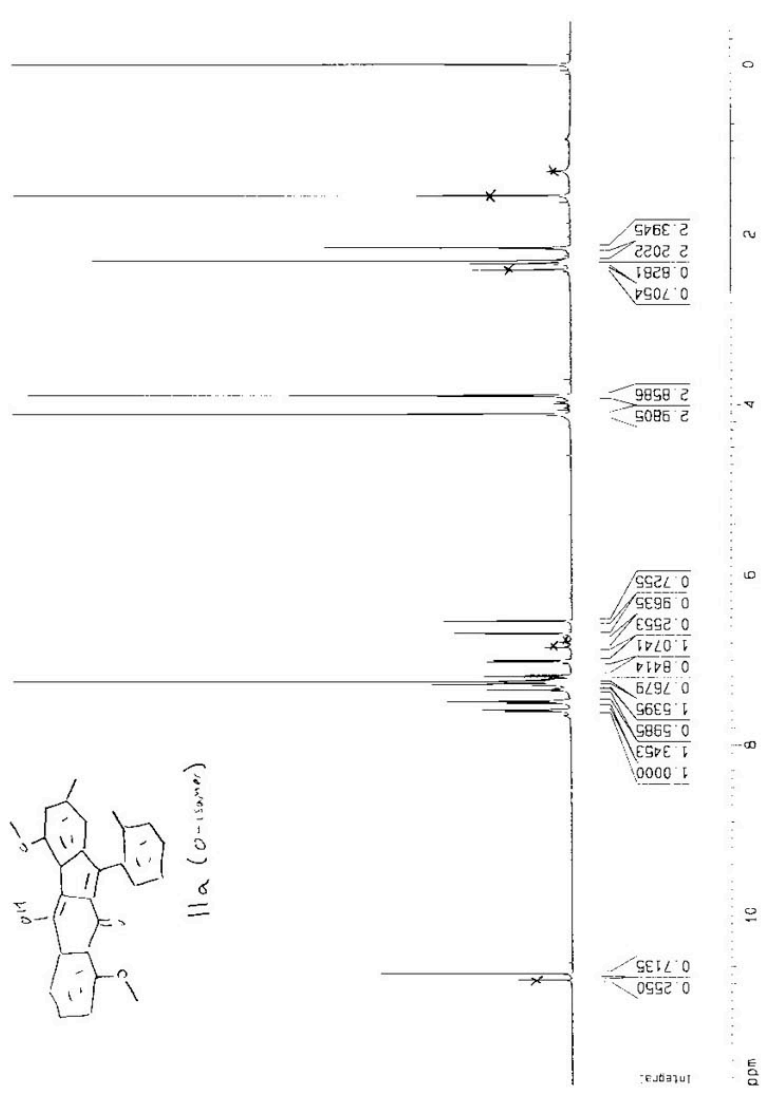
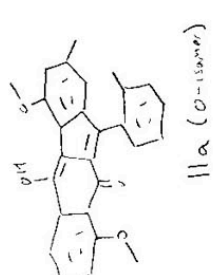


Current Data Parameters
 NAME: 4J-5-19-05
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20050519
 Time: 9:46
 INSTRUM: spect
 PROBHD: 5 mm Dui 13
 PULPROG: zgpg30
 TC: 32/BB
 SOLVENT: CDCl3
 NS: 256
 DS: 2
 SWH: 10416.667 Hz
 FIDRES: 0.317861 Hz
 AQ: 5.5728740 sec
 RG: 4096
 DA: 48.000 usec
 DE: 66.67 usec
 TE: 300.0 K
 L1: 0.05
 D1: 1.05000000 sec
 D11: 11.75 usec
 SFO1: 500.1330885 MHz
 NUC1EUS: 1H

F2 Processing parameters
 SI: 15384
 SF: 500.1330140 MHz
 WDM: nu
 SSB: 0
 GB: 0
 PC: 1.00

1D NMR plot parameters
 CX: 20.00 cr
 F1F: 12.000 ppr
 F1: 6001.56 Hz
 F2F: -0.500 ppr
 F2: -250.07 Hz
 PPMCM: 0.62400 ppr/cm
 HZCM: 312.5624 Hz/cm

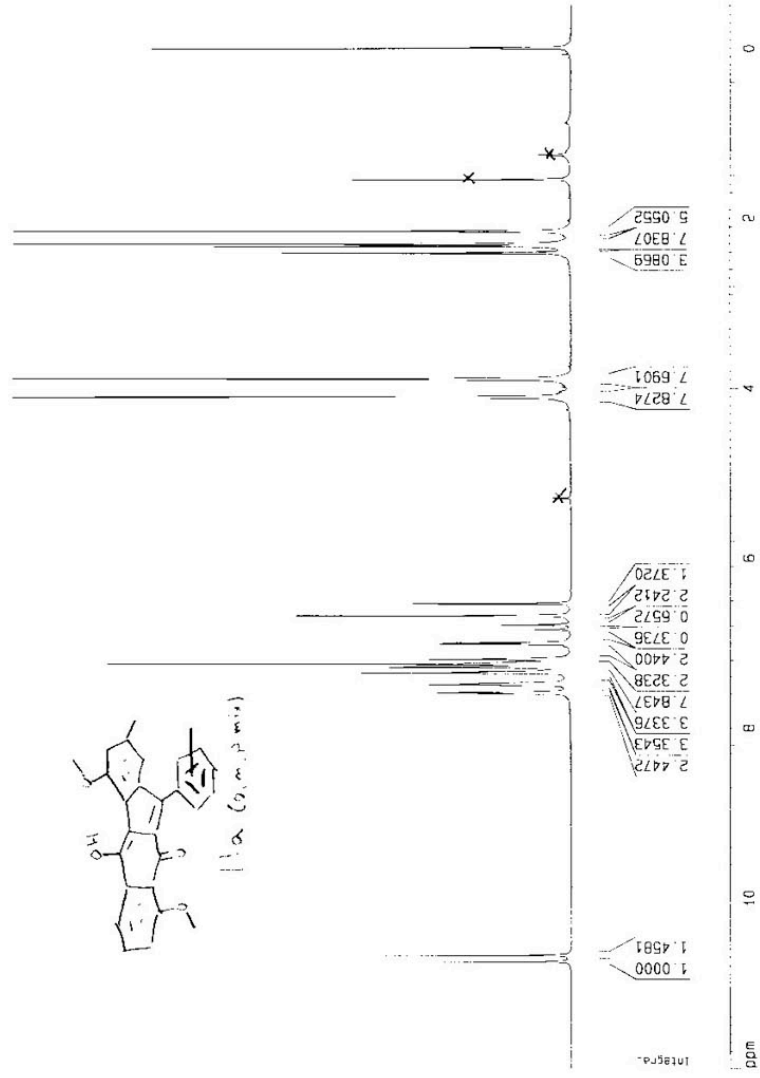


Current Data Parameters
 NAME: KJ-5-19-05
 EXPNO: 3
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20050519
 Time: 13:46
 INSTRUM: spect
 PROBHD: 5 mm Dual 13
 PULPROG: zg30
 TO: 32768
 SOLVENT: CDCl3
 NS: 256
 DS: 2
 SWH: 10416.667 MHz
 FIDRES: 0.317851 Hz
 AQ: 1.5728740 sec
 RG: 5048
 DA: 48.000 USEC
 DE: 88.27 USEC
 TE: 300.0 K
 DELT: 0.35
 D1: 1.0000000 sec
 S1: 11.75 USEC
 SFO1: 500.1330985 MHz
 NUCLEUS: 1H

F2 - Processing parameters
 SI: 16384
 SF: 500.1330154 MHz
 HSI: 1
 EQ: 0
 SSF: 0
 EB: 0
 GC: 0
 PC: 1.00

F2 NMR plot parameters
 CX: 20.00 cr
 CP: 17.000 ppm
 F1: 6001.56 Hz
 F2: -0.520 ppm
 FZ: 250.07 Hz
 GAMMA: 0.62600 ppm/cm
 GZCM: 313.081351 Hz/cm



```

Current Data Parameters
NAME      KJE-9-05
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20050610
Time     8:22
INSTRUM spect
PROBHD   5 mm Multino
PULPROG zgpg30
D        B5356
SOLVENT  DMS-D6
NS       10000
DS       4
SFO1     100.625 MHz
SFO2     0.267960 MHz
AQ       1.7400308 sec
RG       65304
DM       26.550 usec
DE       5.00 usec
TE       300.0 K
D1       2.00000000 sec
d11      0.03000000 sec
d12      0.00020000 sec

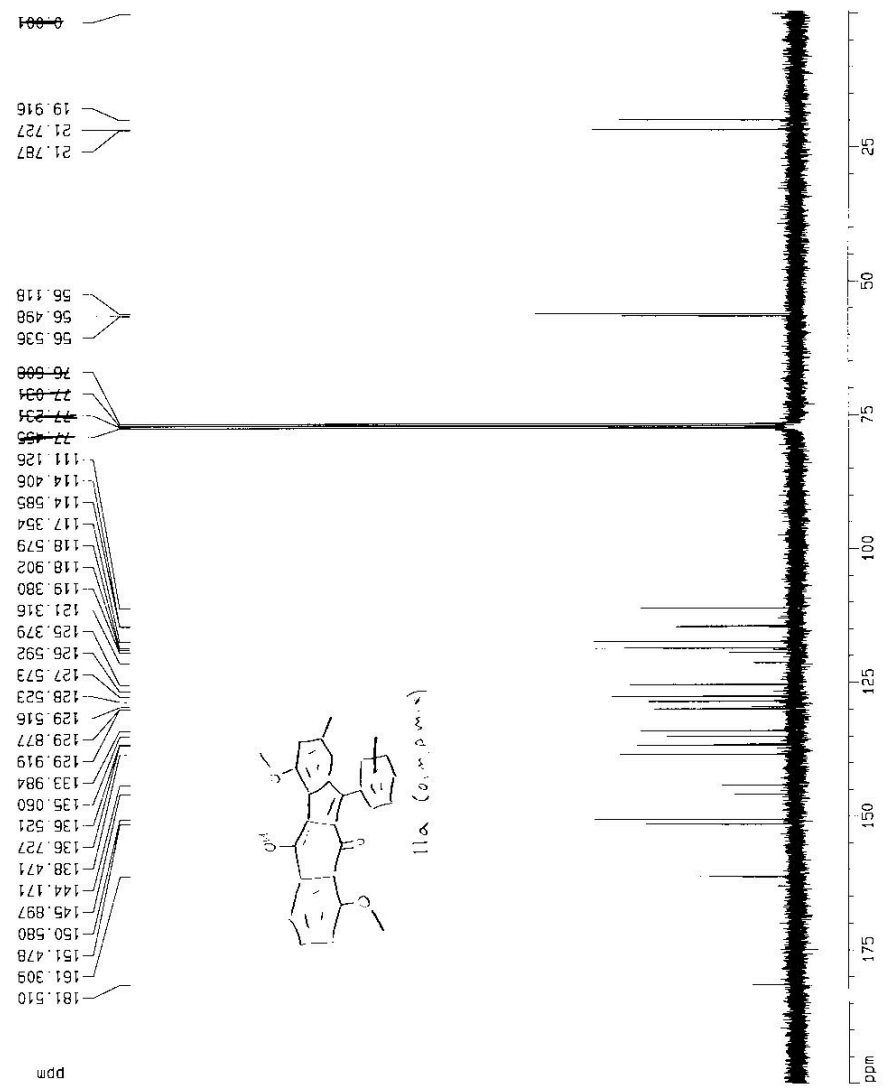
===== CHANNEL f1 =====
NUC1     13C
P1       11.80 usec
PL1      0.00 dB
SFO1     75.4760200 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
P2       110.00 usec
PL2      0.00 dB
PL3      17.50 dB
PL13     17.50 dB
SF02     300.1312000 MHz

F2 - Processing parameters
SI       32768
SF       75.4677481 MHz
RG       0
WDW      0
SSB      0
LB       0.00 Hz
GB       0
PC       1.40

ID NMR pict parameters
CX       20.00 cm
CT       200.00000000
F1       100.625 MHz
F2       300.13120000
PPMCM   10.00500000/cm
HZCM    756.56421 MHz/cm

```

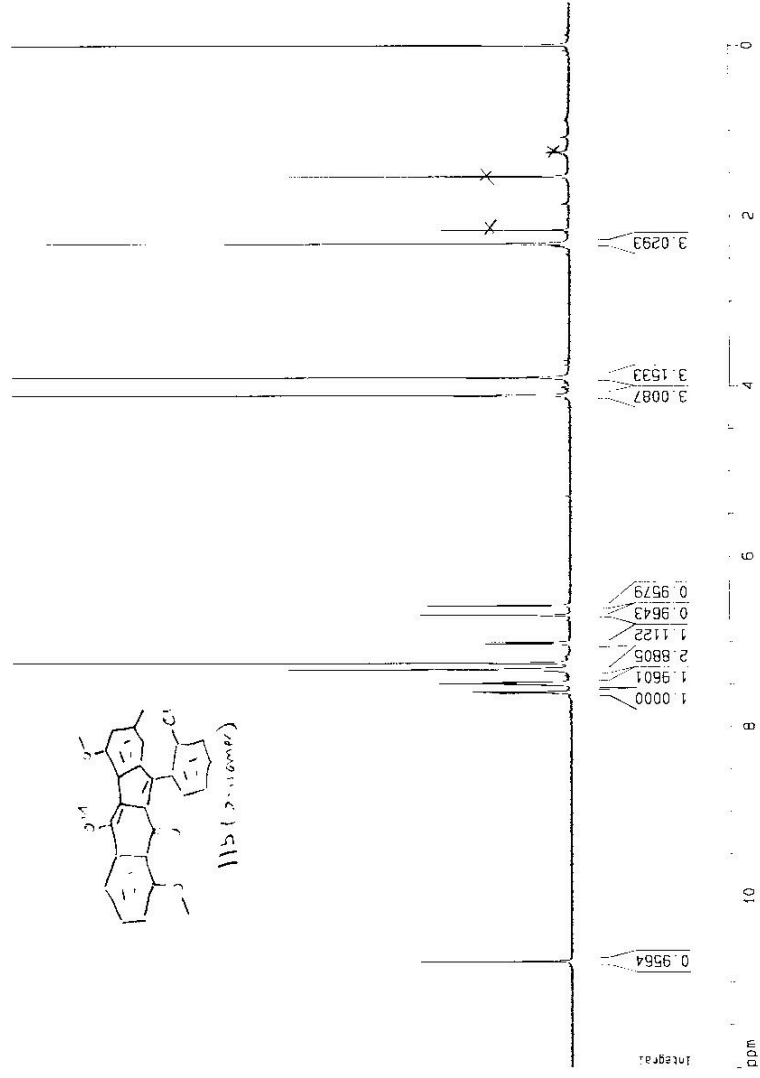


Current Data Parameters
 NAME KJE-5-1E-05
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050516
 Time 17:33
 INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG zgpg30
 TC 32756
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 10416.657 Hz
 FIDRES 0.317891 Hz
 AQ 1.5723740 sec
 RG 4095
 CW 48.000 usec
 DL 68.67 usec
 TE 300.0 K
 HL 0 dB
 O1 1.00000000 sec
 F1 11.75 usec
 SF01 500.133085 MHz
 NUCLEUS 1H

F2 Processing parameters
 SI 16284
 S 500.130139 MHz
 WDM nu
 SSB 0
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cr
 F/F 17.000 ppm
 F1 6001.55 Hz
 F2 -250.07 Hz
 FWHM 0.87500 ppm/cm
 FZCM 312.58124 Hz/cm

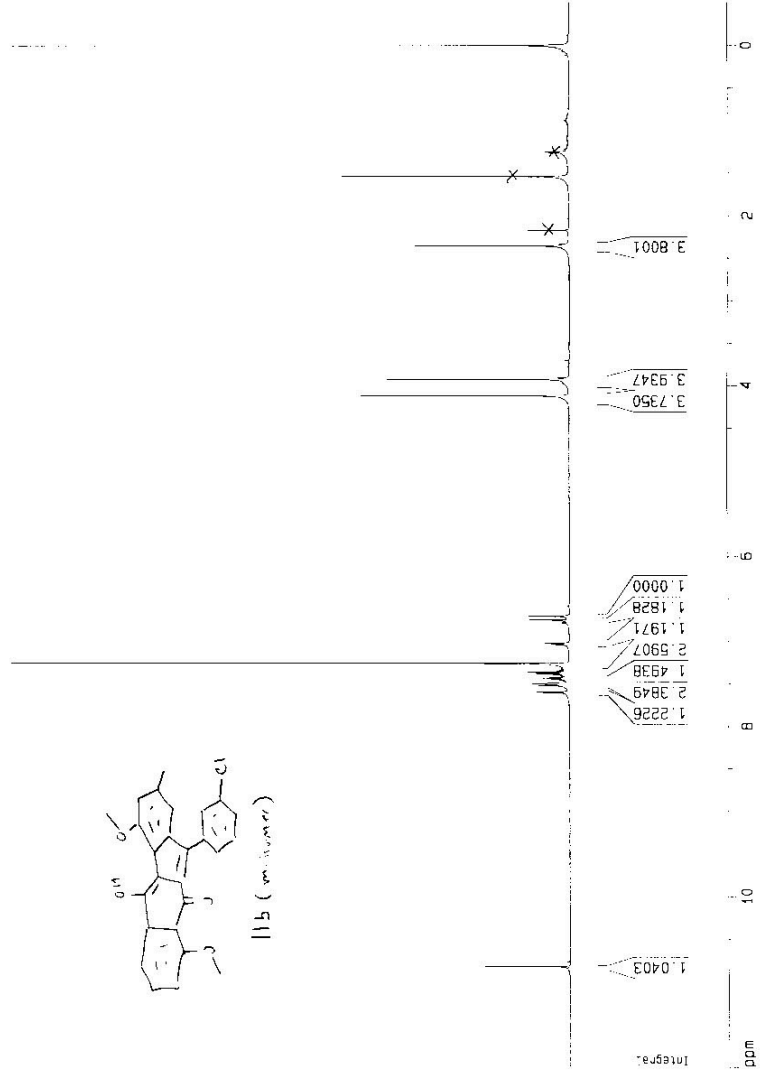
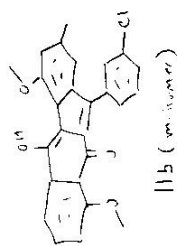


Current Data Parameters
 NAME 4J-5-16-05
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080516
 Time 10:01
 INSTRUM spect
 PROBHD 5 mm Dual 1:3
 PULPROG zgpg30
 TD 7930
 T2 32768
 SOLVENT CCL13
 NS 512
 DS 2
 SWH 10416.657 Hz
 FIDRES 0.314894 Hz
 AQ 1.5729740 sec
 RG 4096
 CW 48.000 usec
 DE 58.67 usec
 TE 300.0 K
 L1 0 dB
 D1 1.00000000 sec
 P1 11.75 usec
 SFO1 500 133085 MHz
 NUC1 13
 NUC2 1H

F2 Processing parameters
 SI 16384
 SF 500 1300133 MHz
 NU 1
 SSF 0
 LR 0.00 Hz
 GB 0
 FC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1 17.000 ppm
 F2 6001.56 Hz
 F3 -0.500 ppm
 F4 250.07 Hz
 SFO1 6.65900 ppm/Lin
 FIDM 312.38124 Hz/Lin

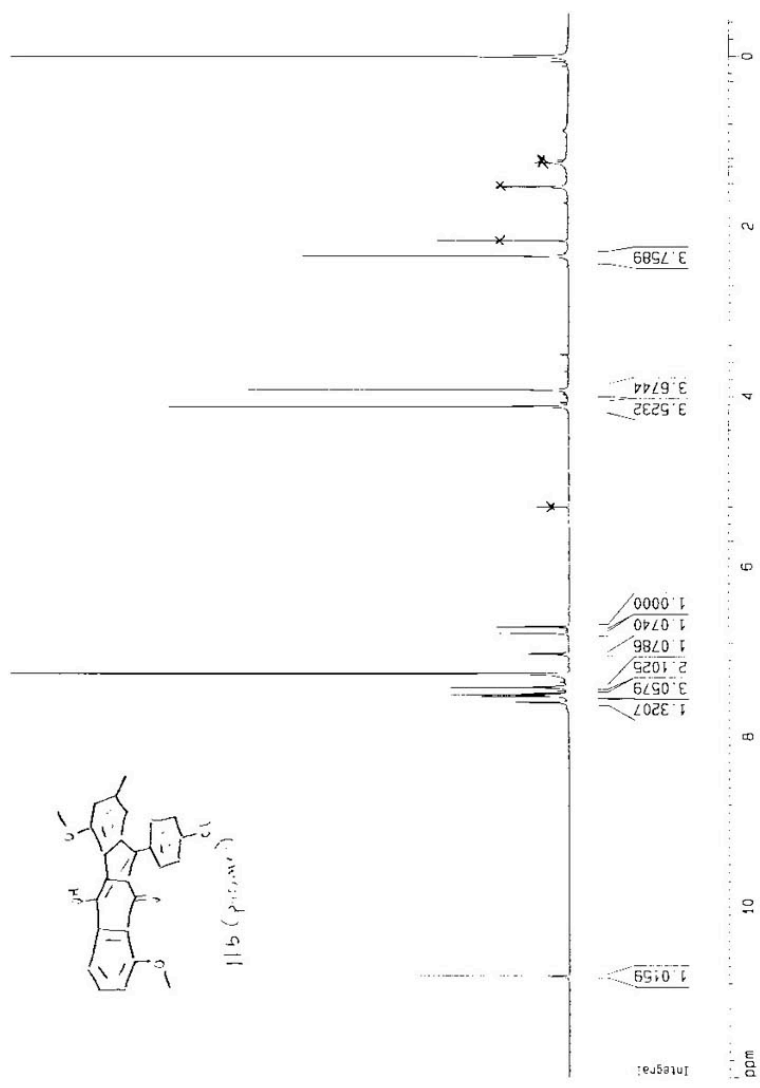


Current Data Parameters
 NAME 4E-5-13-05
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050513
 Time 17:16
 INSTRUM spect
 PROBHD 5 mm Dui: 11
 PULPROG zgpg30
 TC 32768
 SOLVENT CDCl3
 NS 512
 DS 2
 SWH 10416.657 Hz
 FIDRES 0.317851 Hz
 AQ 1.5729740 sec
 RG 4096
 CA 48.000 usec
 CB 68.67 usec
 TE 300.0 K
 LC 0.35
 PC 1.00000000 sec
 SI 11.75 usec
 SFO 500.1330885 MHz
 NUC1EL13 13C

F2 Processing parameters
 SI 15264
 SF 500.1330885 MHz
 WDM 1.00
 SSB 0
 GB 0
 PC 1.00

1D NMR list parameters
 CX 20.00 cm
 F1 19.000 ppm
 F2 -0.500 ppm
 F3 250.07 Hz
 DDM 0.62500 ppm/cm
 F4 312.58124 Hz/cm

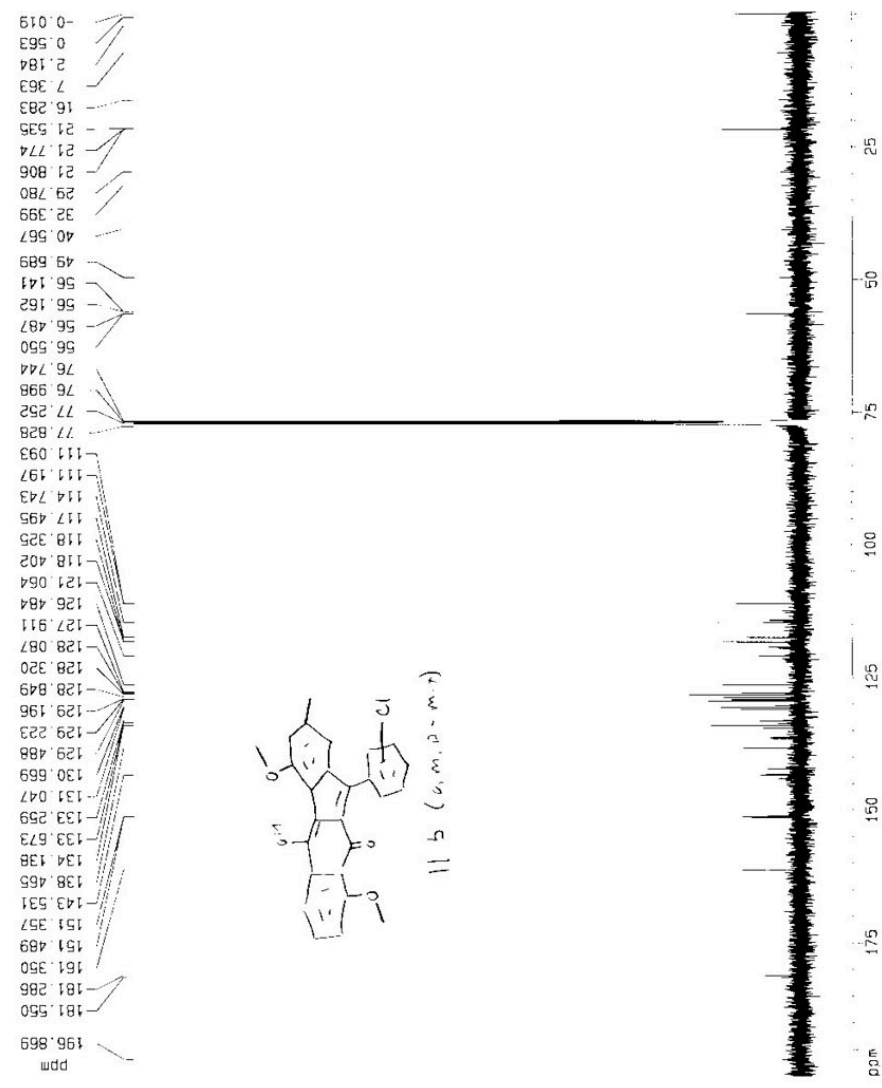


Current Data Parameters
 NAME KJF-5-17-05
 EXPNO 8
 PROCNO 1

F2 - Acquisition Parameters
 Ch.c. 20090518
 Time 15:16
 INSTRUM spect
 PROBHD 5 mm Dca1 13
 P1 PRG2 /gpg50
 T1 SFS36
 T2 SFS36
 SOLVENT CDCl3
 NS 2:71
 DS ?
 SM? 4:665,668 Hz
 FIDRES 0.633783 Hz
 AQ 0.7954820 sec
 RG 32768
 DR 12.000 usec
 DE 17.14 usec
 TE 303.2 K
 L1 0 dB
 L2
 L3
 J1 2.0000000 sec
 J2
 J3
 CDPORG wa.1716
 P31 120.00 usec
 S4 55 dB
 J11 0.0300000 sec
 SP 21 dB
 S1 9.79 usec
 S101 125.75792614 MHz
 NUCLEUS 13C

F2 Processing Parameters
 SI 125.757926 MHz
 SF 27269
 ADK EN
 SSB 0
 RB 1.00 Hz
 GB 0
 PC 0.54

1D NMR plot parameters
 CX 20.00 cr
 F1F 200.000 pprf
 S1 25151.5617
 F2P C 500.00F
 F2 62.88 Hz
 GAMMA 13C
 FWHM 1256.12180117/cm

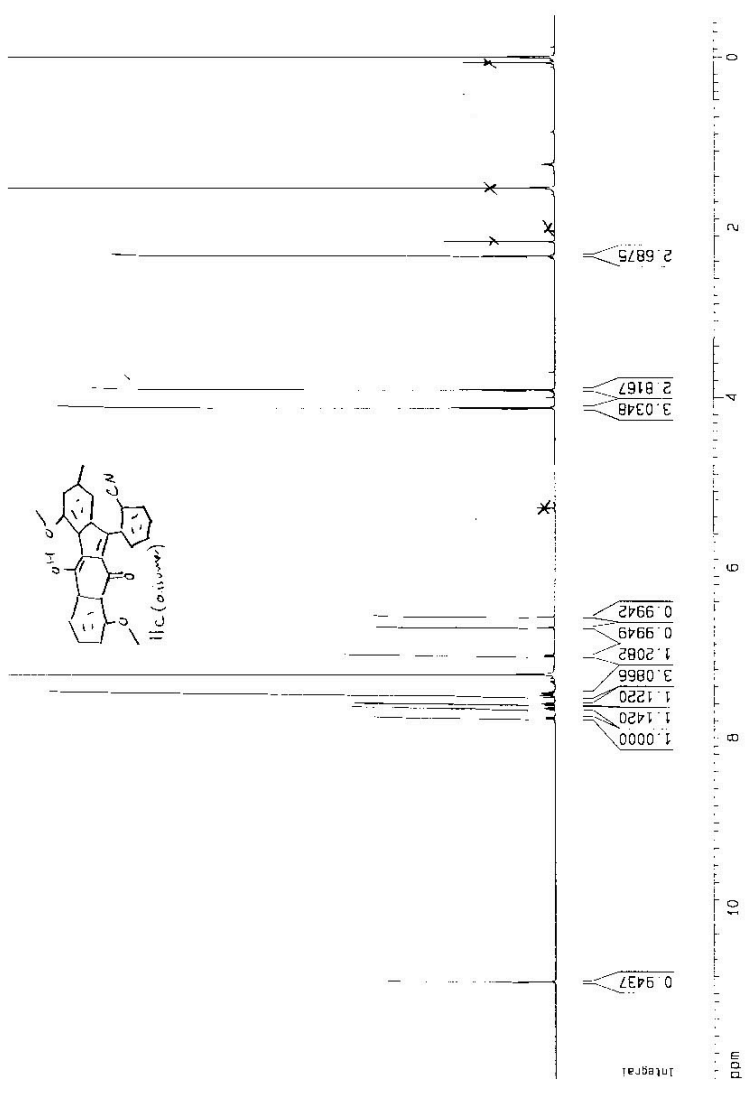


Current Data Parameters
 NAME KJF-5-05
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050509
 Time 10:01
 INSTRUM spect
 PROBHD 5 mm QNP 13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 6578.947 Hz
 FIDRES 0.200774 Hz
 AQ 2.4904180 sec
 RG 6192
 DA 76.000 usec
 DF 50.00 usec
 TE 300.0 K
 HL 0 dB
 DI 1.0000000 sec
 P1 11.75 usec
 SFO1 500.1327644 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 500.1300135 MHz
 RG 0
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cr
 F1 12.000 ppm
 F2 6001.55 Hz
 F3 -0.500 ppm
 F4 250.07 Hz
 PPMCM 0.62500 ppm/cm
 HZCM 312.58124 Hz/cm

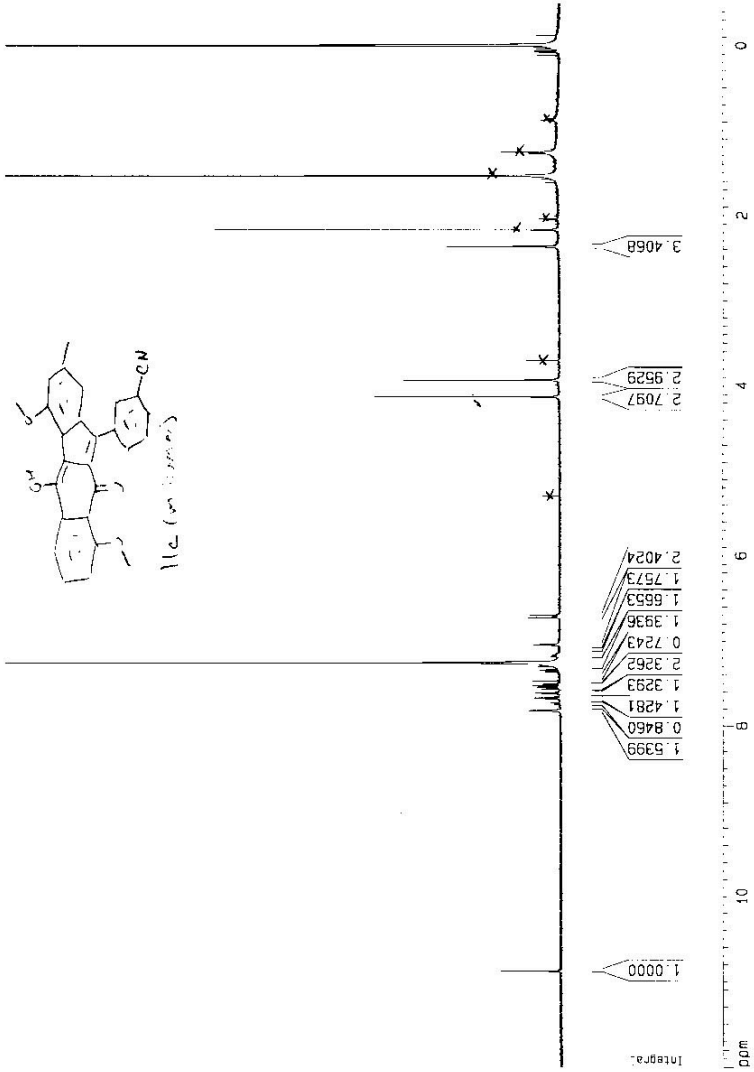


Current Data Parameters
 NAME KJL-5-7-05
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050507
 Time 13:11
 INSTRUM spect
 PROBD 5 mm Dui1 :3
 PULPROG zg30
 TO 32766
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 6578.947 Hz
 FIDRES 0.200774 Hz
 AQ 2.4904180 sec
 RG 61.92
 DW 76.000 usec
 DE 95.000 usec
 TE 300.0 K
 HL 0 dB
 D1 1.00000000 sec
 P1 11.75 usec
 SFO1 500.1327643 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 500.1300135 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1 12.000 ppr
 F1 6001.55 Hz
 F2 -0.500 ppr
 F2 -250.07 Hz
 PPMCM 0.62500 ppr/cm
 HZCM 312.58124 Hz/cm

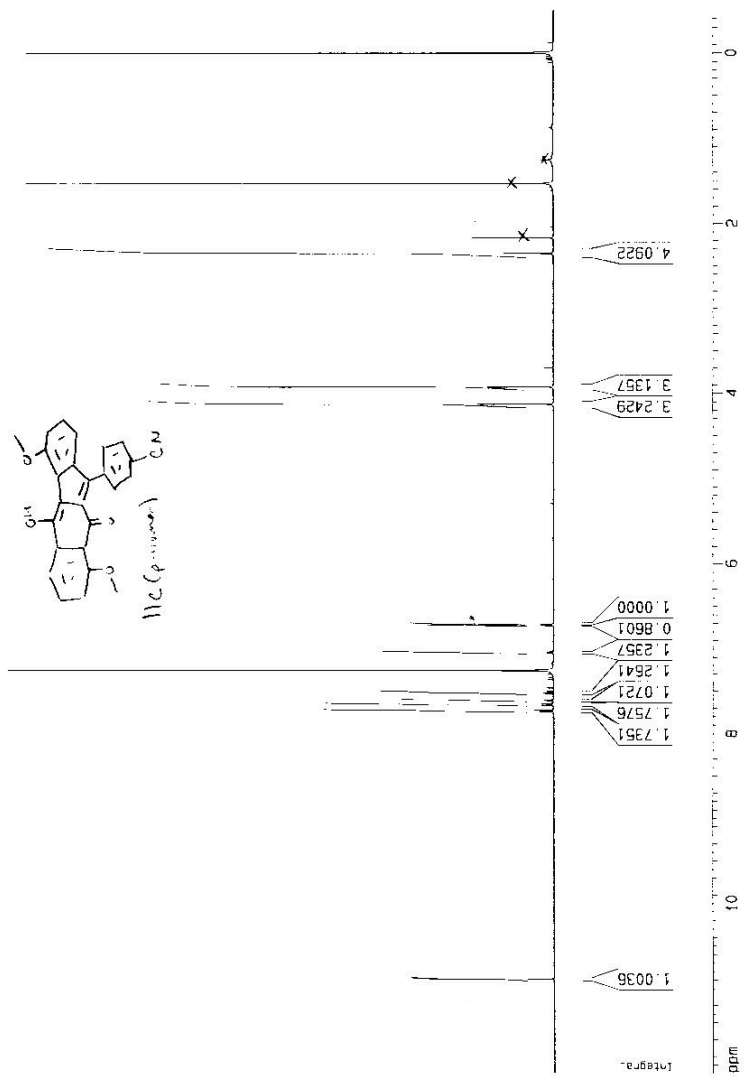


Current Data Parameters
 NAME KJE662005
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050505
 Time 13:28
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 512
 DS 2
 SMH 6493.506 Hz
 FIDRES 0.158166 Hz
 AQ 2.5231860 sec
 RG 8192
 DM 77.000 usec
 DE 96.25 usec
 TE 300.2 K
 L1 0 dB
 J1 1.00000000 sec
 P1 11.75 usec
 SFO1 300.1327665 MHz
 NUC1US 1H

F2 Processing parameters
 SI 16384
 SF 300.1300135 MHz
 WDW rh
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F4P 12.000 ppb
 F1 6001.95 Hz
 F2P -0.500 ppb
 F2 -250.07 Hz
 PPMCM 0.62500 ppb/cm
 HZCM 312.56124 Hz/cm

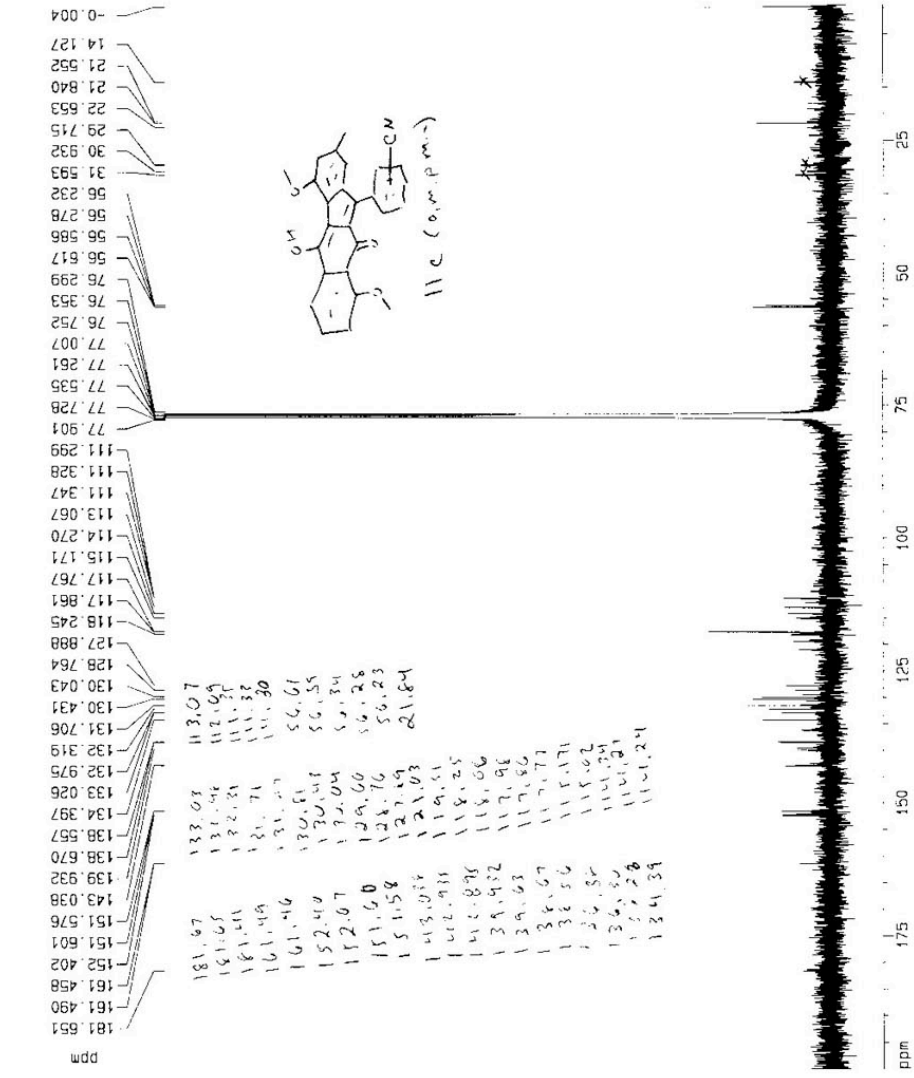


Current Data Parameters
 NAME: 4JL-5-9-05
 EXPNO: 4
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20050509
 Time: 15:35
 INSTRUM: spect
 PROBD: 5 mm Dual 1H
 PULPROG: zgpg30
 TO: 65536
 SOLVENT: CDCl3
 NS: 51200
 DS: 2
 SWH: 41666.668 Hz
 FIDRES: 0.635763 Hz
 AQ: 0.7864820 sec
 RG: 32768
 DW: 12.000 usec
 DE: 17.14 usec
 TE: 300.0 K
 HL: 0.98
 D2: 2.00000000 sec
 WALTZ16
 PCPDPRG: waltz16
 P3: 120.00 usec
 S4: 55 dB
 D7: 0.0300000 sec
 SP: 21 dB
 P1: 9.79 usec
 SF01: 125.7736214 MHz
 NUCLEUS: 13C

F2 Processing parameters
 SI: 32768
 SF: 125.757501 MHz
 AOK: E1
 SSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 0.30

1D NMR plot parameters
 CX: 20.00 cm
 F1P: 200.000 ppm
 F1: 25151.59 Hz
 F2P: -0.500 ppm
 F2: 62.66 Hz
 GAMMA: 1.002500 ppm/cm
 XCDM: 4260.72560 Hz/cm

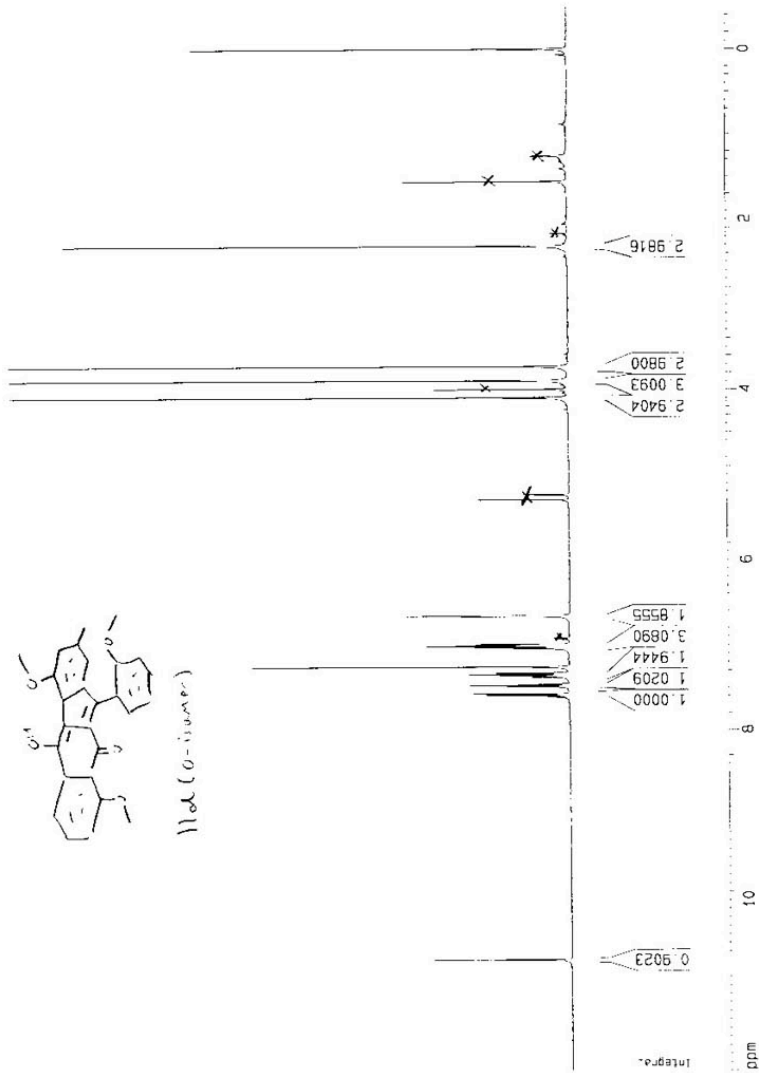


Current Data Parameters
 NAME: 6J-5-17-05
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20050517
 Time: 9:21
 INSTRUM: spect
 PROBHD: 5 mm Dbb-13
 PULPROG: zgpg30
 T2: 32.768
 SOLVENT: DMS-D6
 NS: 128
 DS: 2
 SWH: 10416.667 Hz
 FIDRES: 0.31785 Hz
 AQ: 1.5729140 sec
 RG: 2048
 DW: 48.000 usec
 DE: 68.0 usec
 TE: 300.0 K
 HL: 0 dB
 DI: 1.00000000 sec
 P1: 11.75 usec
 SFO1: 500.1330885 MHz
 NUC1EUS: 1H

F2 Processing parameters
 SI: 16384
 SF: 500.130044 MHz
 WCW: no
 SSB: 0
 GB: 0
 PC: 1.00

1D NMR plot parameters
 CX: 20.00 cm
 C1P: 12.000 ppm
 F1: 600.156 Hz
 F2P: -0.500 ppm
 F2: 250.07 Hz
 SFOCM: 0.67800 ppm/cm
 HZCM: 112.98124 Hz/cm

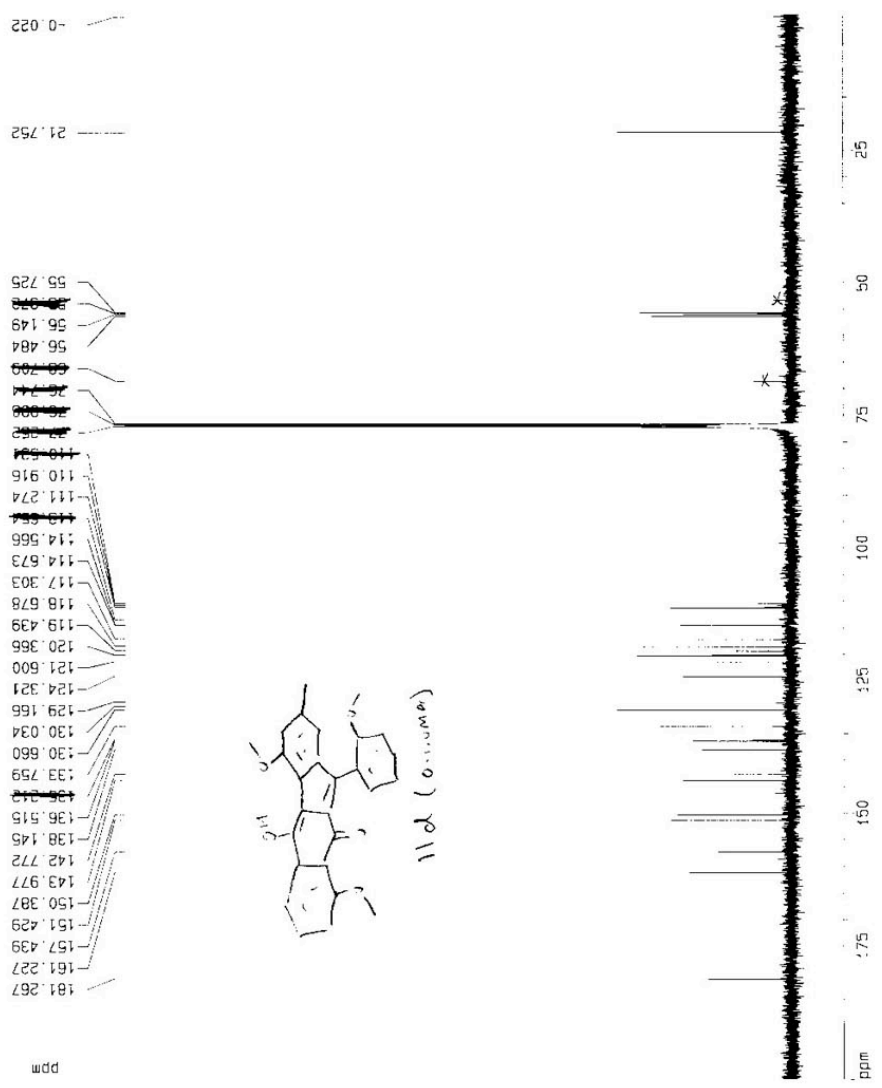


Current Data Parameters
 NAME: 4U-5 17-05
 EXPNO: 2
 PROCNO: 1

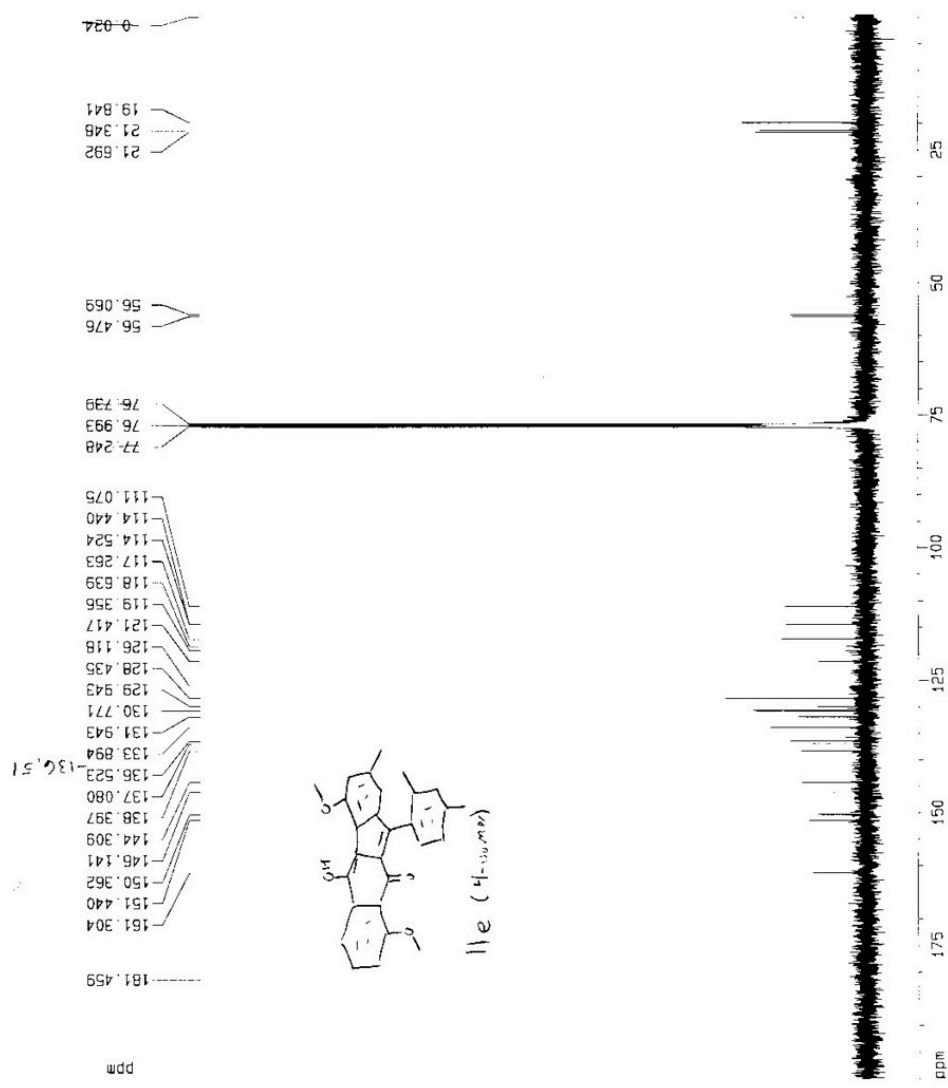
F2 - Acquistion Parameters
 Date_ : 20090517
 Time : 11.05
 INSTRUM : spect
 PROCNO : 13
 F1 FREQ : 799930
 TC : 65536
 SOLVENT : CDCl3
 NS : 6045
 DS : 2
 SWH : 41686.688 Hz
 FIDRES : 0.835462 Hz
 AQ : 0.7864820 sec
 SFO : 32768
 DQ : 12.000 USEC
 ZF : 17.74 USEC
 TE : 300.0 K
 L1 : 0 dB
 C2 : 2.00000000 sec
 C3 : 1.776
 P1 : 120.00 USEC
 S4 : 55 dB
 D11 : 0.0300000 sec
 S5 : 21 dB
 P4 : 5.79 USEC
 SFO1 : 125.738274 MHz
 NUCLEUS : 13C

F2 Processing Parameters
 SI : 32768
 SF : 125.7573826 MHz
 RM : EM
 SSB : 0
 GC : 1.00 Hz
 BA : 0
 PC : 1.40

13C NMR pint parameters
 CX : 20.00 cm
 F1P : 240.000 ppm
 F1 : 25151.56 Hz
 F2P : 0.500 ppm
 F2 : -52.88 Hz
 GAMMA : 0.02900 ppm/cm
 ZCEN : 2560.72180 Hz/cp



Current Data Parameters
 NAME KUL-5-11-05
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20050511
 Time 11.02
 INSTRUM spect
 PROBHD 5 mm Dual 13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 4228
 DS 2
 SWH 41666.668 Hz
 FJDRS 0.635783 Hz
 AQ 0.7864820 sec
 RG 32768
 DW 12.000 usec
 DE 17.14 usec
 TE 300.0 K
 HL 0 dB
 D1 2.00000000 sec
 waltz16
 CPDPRG 120.00 usec
 S4 55 dB
 D2 0.0300000 sec
 S2 21 dB
 P1 9.79 usec
 SF01 125.7736214 MHz
 NUCLEUS 13C
 F2 - Processing parameters
 SI 32768
 SF 125.7577926 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40
 ID NMR o1ct parameters
 CX 20.00 cm
 F1 200.000 ppm
 F2 25151.56 Hz
 F2P -0.500 ppm
 F2 -62.88 Hz
 F2MCM 10.02500 ppm/cm
 F2CM -280.72160 Hz/cm

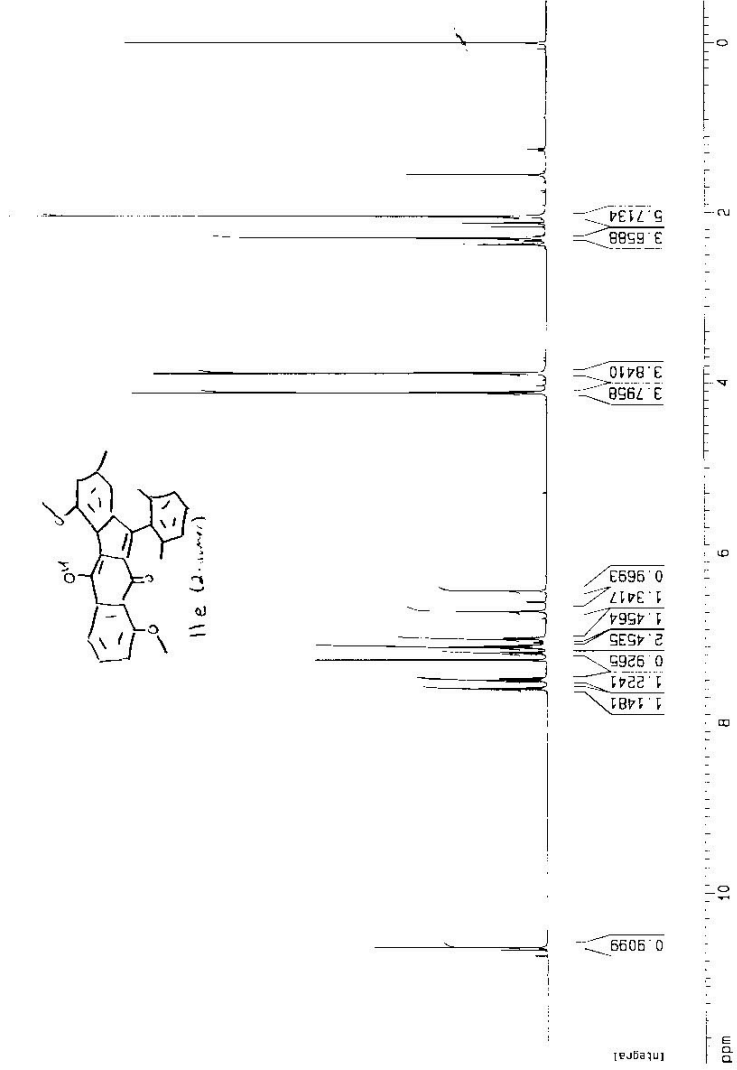
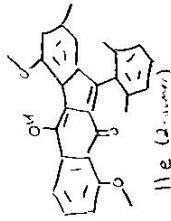


Current Data Parameters
 NAME ALI-5-11-05
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050511
 Time 15:35
 INSTRUM spect
 PROBRD 5 mm DLG1.13
 PULPROG zg30
 TD 32768
 SOLVENT DMS-D6
 NS 256
 DS 2
 SMH 10416.667 Hz
 FIDRES 0.317891 Hz
 AQ 1.5729540 sec
 RG 2048
 DM 48.000 usec
 DE 68.5/ usec
 TE 300.2 K
 NL 0 dB
 SI 1.00000000 sec
 P1 11.75 usec
 ST01 500.1330065 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 500.1300145 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1F 12.000 ppm
 F1 6001.56 Hz
 F2F -0.500 ppm
 F2 250.07 Hz
 PPM0M 0.82500 ppm/cm
 HZ0M 312.88124 Hz/cm



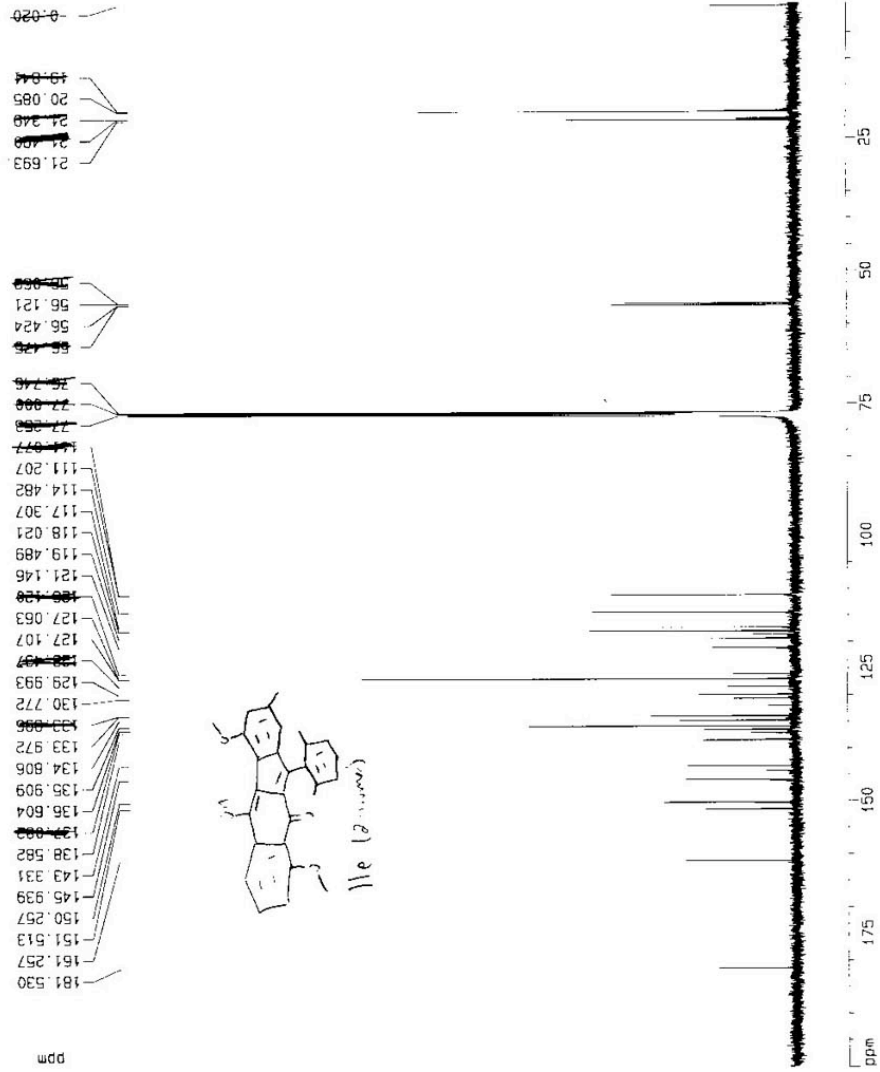
Current Data Parameters
 NAME KJL-5-11-05
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050511
 Time 16:37
 INSTRUM spect
 P-DBHD 5 mm QNP-1H
 P1 PRG zgpg30
 T1 85596
 SOLVENT CDCl3
 NS 23159
 DS 2
 SWH 41866.668 Hz
 FIDRES 0.635783 Hz
 AQ 0.7864620 sec
 RG 32769
 DM 12.000 usec
 DE 17.14 usec
 TE 300.0 K
 L1 0 dB
 J1 2.0000000 sec
 CDPRG waltz16

P31 120.00 usec
 S4 55 dB
 D1 0.0300000 sec
 S2 21 dB
 P 5.79 usec
 SF01 125.7736214 MHz
 NUCLEUS 13C

F2 Processing parameters
 SI 32768
 SF 125.757926 MHz
 KW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 25151.561 Hz
 F2P 0.500 ppm
 F2 62.88 Hz
 DNMCH 10.02500 ppm/cm
 1/DM 1260.72180 Hz/cm

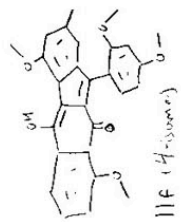
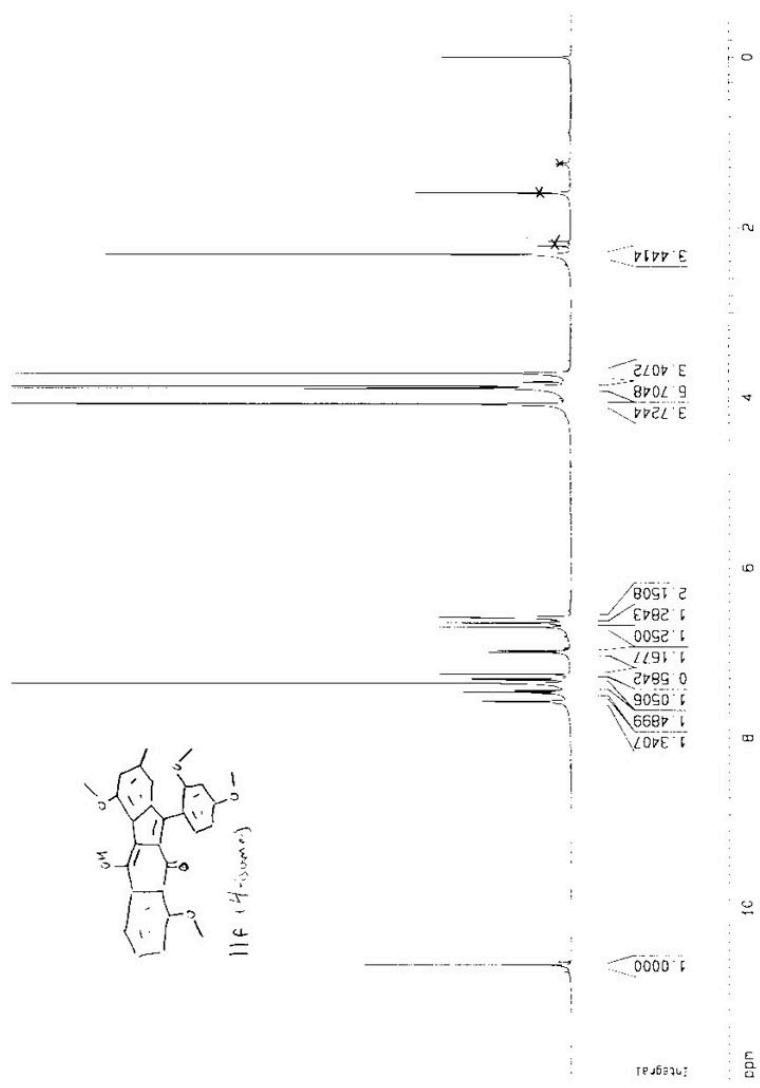


Current Data Parameters
 NAME KJF-5-26-05
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050526
 Time 12:21
 INSTRUM spect
 PROBHD 5 mm Dual 13
 PULPROG zgpg30
 T2 32766
 SOLVENT CDCl3
 NS 512
 DS 2
 SWH 10415.647 Hz
 FIDHLS 0.317801 Hz
 AQ 1.5729140 sec
 RG 1024
 DM 48 000 usec
 DE 58.57 usec
 TE 300.0 K
 RL1 0 dB
 D1 1.0000000 sec
 P1 11.75 usec
 SFO1 500.1330885 MHz
 NUCLEUS 1H

F2 Processing parameters
 SI 16384
 SF 500.1300164 MHz
 MDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR list parameters
 CX 20.00 cm
 F1F 12.000 ppm
 F1 6001.56 Hz
 F2F 0.500 ppm
 F2 250.07 Hz
 SFO1 0.62500 ppm/cm
 F2CM 312.58124 Hz/cm

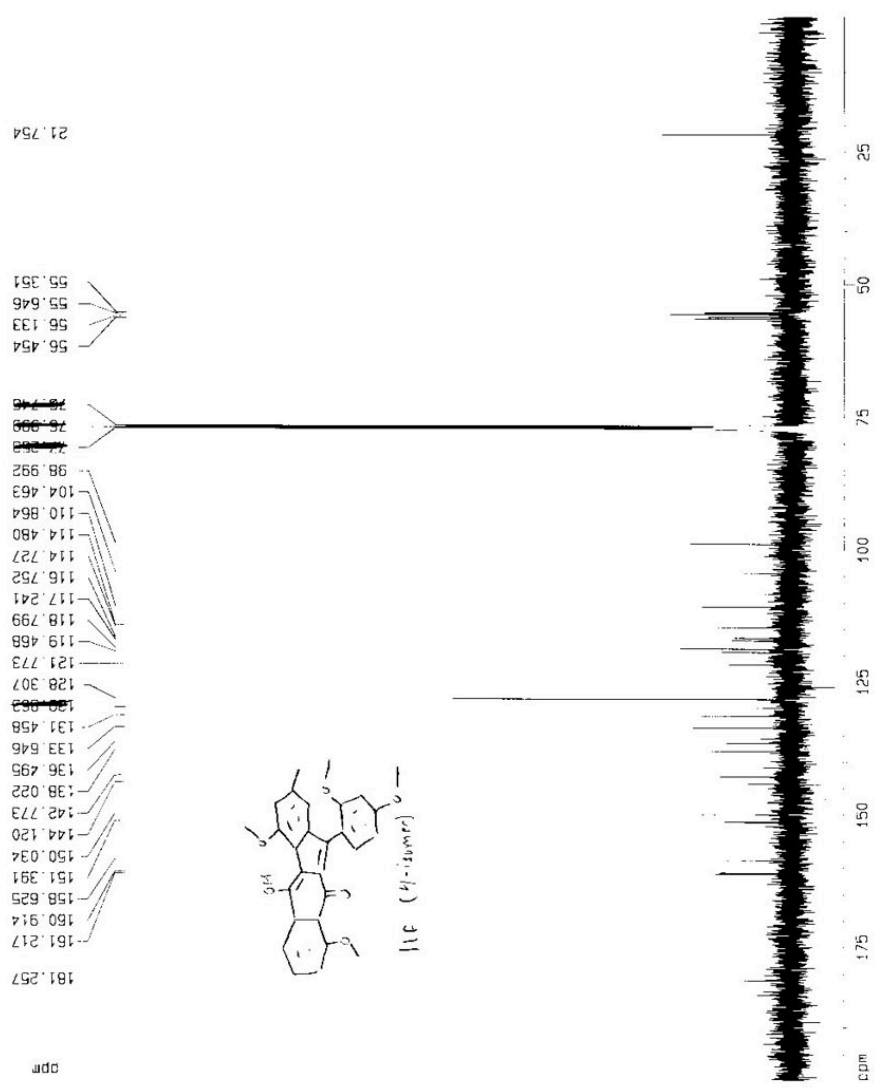


Current Data Parameters
 NAME: 5-26-05
 EXPNO: 2
 PROCNO: 1

F2 - Acquis. Parameters
 Date_: 20050527
 Time: 0:14
 INSTRUM: spect
 PROBHD: 5 mm QNP 1H/13
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 31458
 DS: 2
 SWH: 41566.668 Hz
 FIDRES: 0.635783 Hz
 AQ: 0.7864820 sec
 RG: 32768
 DW: 12.000 usec
 DE: 17.14 usec
 TE: 300.0 K
 L1: 0.05
 C1: 2.0000000 sec
 C2: 1.1716
 C3: 120.00 usec
 SFO1: 0.0300000 sec
 SFO2: 21.03
 SFO3: 9.79 usec
 SFO4: 128.735214 MHz
 NUC1: 13C

F2 Processing Parameters
 SI: 32768
 SF: 125.757539 MHz
 KCM: EM
 SSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 0.50

ID NMR plot parameters
 CX: 20.00 cm
 FWH: 200.000 bar
 F1: 25151.56 Hz
 F2: 0.500 bar
 F3: 62.88 Hz
 PPMCK: 10.00000 ppm/CM
 Y1CM: 1250 /2152 Hz/cm

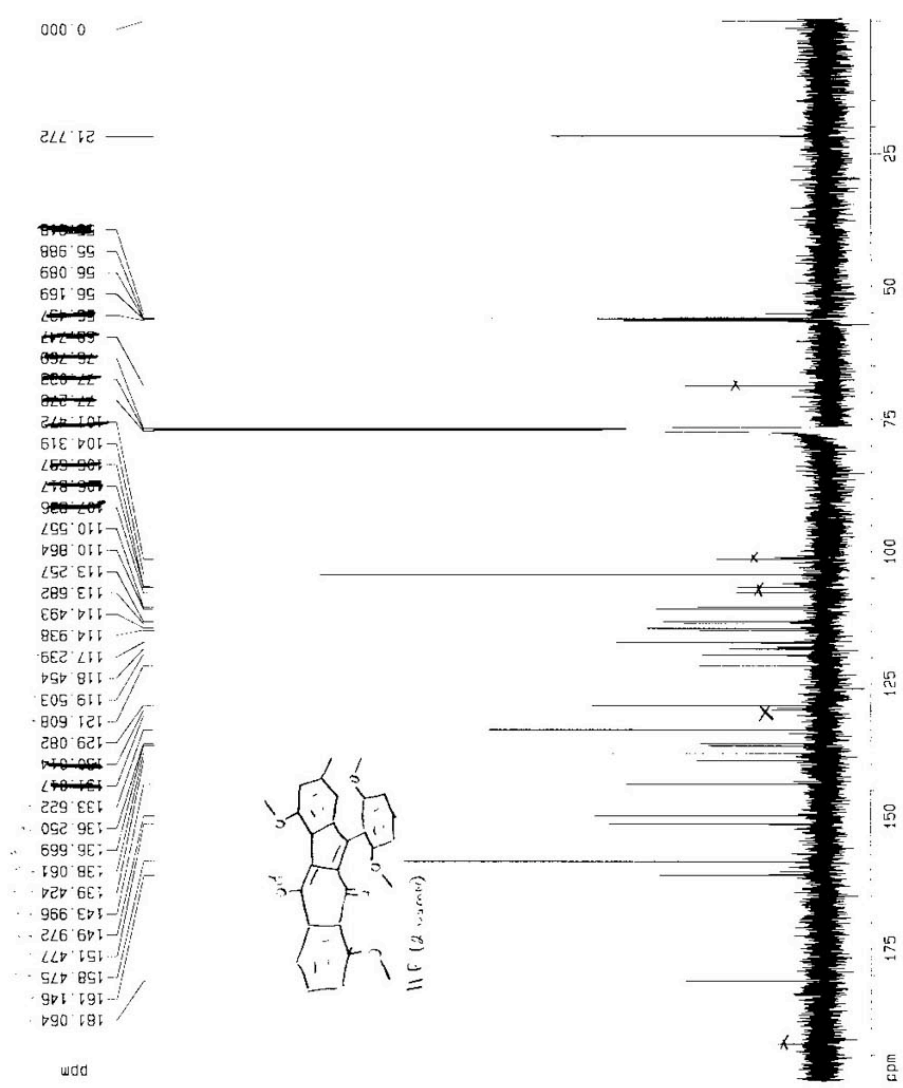


Current Data Parameters
 NAME 4J-B-10-05
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080610
 Time 0.25
 INSTRUM spect
 PULPROG 5 mm Dual 13
 TD 65536
 SOLVENT C3CL3
 NS 53056
 DS 2
 SWH 41665.658 Hz
 FIDRES 0.635763 Hz
 AQ 0.7964820 sec
 RG 32768
 CM 12.000 usec
 SFO 171.4 usec
 TE 300.0 K
 FL 0.00
 D1 2.0000000 sec
 CDPORG null716
 SFO 120.00 usec
 SA 55.00
 D11 0.0300000 sec
 SP 21.00
 P1 9.79 usec
 SF01 125.736214 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 32768
 SF 125.7571676 MHz
 XQM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1F 260.000 ppm
 F1 25151.56 Hz
 F2F -0.500 ppm
 F2 52.88 Hz
 PPKCM 10.07500 ppm/cm
 HZ/KM 260.72160 Hz/cm

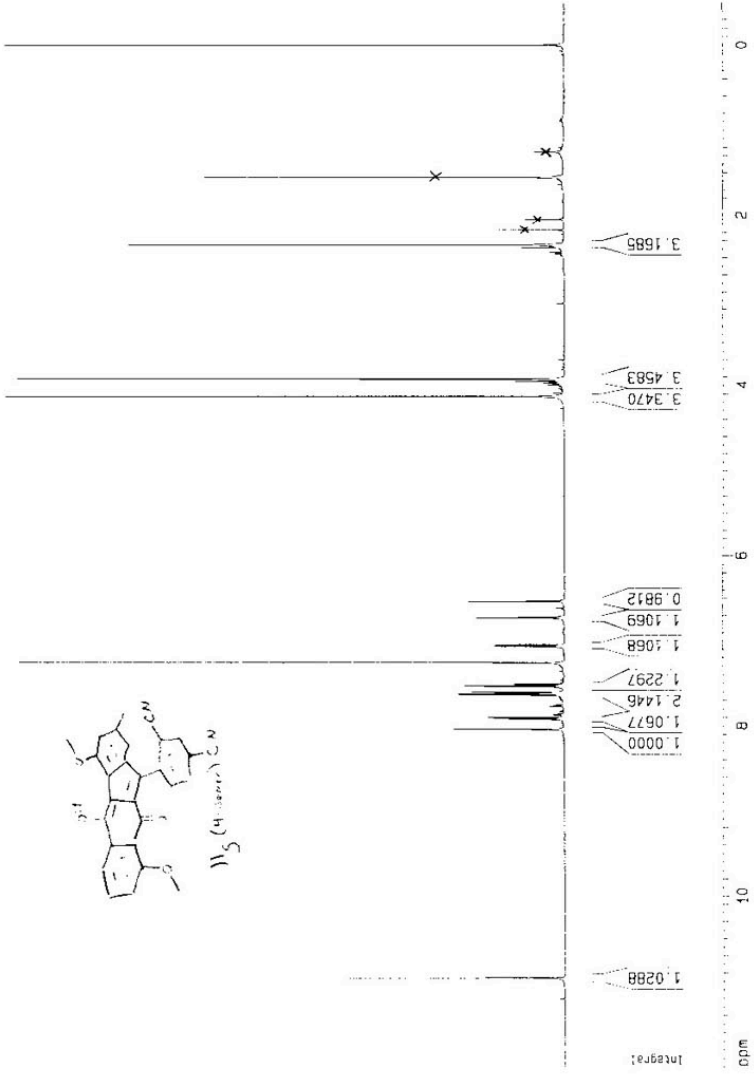


Current Data Parameters
 NAME Kuf-5-12-05
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050512
 Time 10:04
 INSTRUM spect
 PROBR0 31
 PULPROG zgpg30
 TD 32768
 SOLVENT c3cl3
 NS 256
 DS 2
 SWH 104416.667 Hz
 FIDRES 0.31781 Hz
 AQ 1.5729140 sec
 RG 4096
 DM 48.000 usec
 DQ 68.67 usec
 TE 300.0 K
 HL 0 dB
 GC 1.0000000 sec
 FI 11.75 usec
 SFO1 500.1330885 MHz
 NUC1E15 13C

F2 Processing Parameters
 SI 16384
 SF 500.1300132 MHz
 WCW 0
 SSB 0
 GB -3
 PC 1.00

1D NMR list parameters
 CX 20.00 cm
 F1 12.000 ppm
 F1 6001.56 Hz
 F2 -0.500 ppm
 F2 250.07 Hz
 PPMCM 0.62500 ppm/cm
 HZ/CM 312.58162 Hz/cm

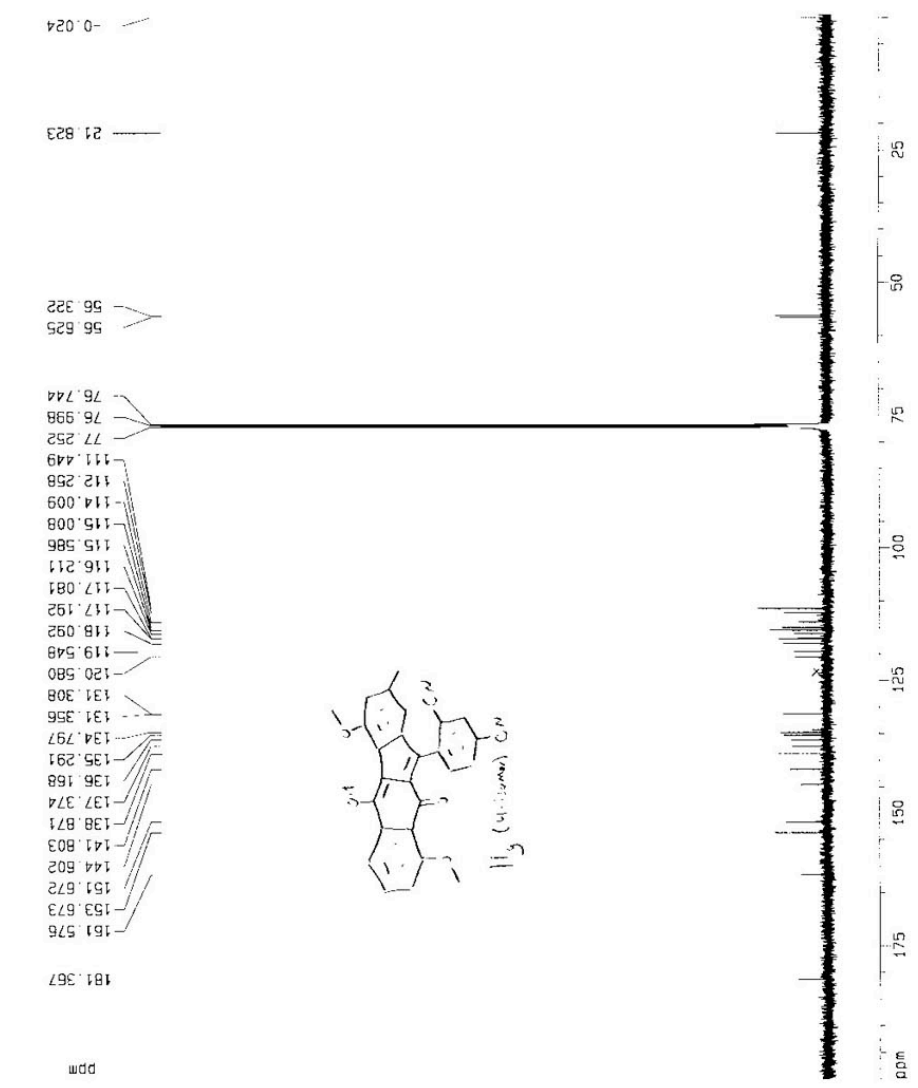


Current Data Parameters
 NAME KJE-5-12-05
 EXNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050512
 Time 11.46
 INSTRUM spect
 PULPROG zgpg30
 5 mm QNPc 13
 T2 490950
 T2 65536
 SOLVENT DMS
 NS 2370
 DS 2
 SWH 41666.668 Hz
 FIDRES 0.635763 Hz
 AQ 0.7664820 sec
 RG 32768
 DM 12.000 usec
 DT 17.74 usec
 TE 300.0 K
 L-1 0.03
 P1 7.0000000 sec
 P2 120.00 usec
 P3 120.00 usec
 P4 55.03
 D1 0.0300000 sec
 D2 21.03
 P5 9.79 usec
 SFO1 125.735214 MHz
 NUCLEUS 13C

F2 Processing parameters
 SI 32768
 SF 125.735214 MHz
 ACQ EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1 200.000 ppm
 F2 25151.56 Hz
 F2P 0.500 ppm
 F2 -62.68 Hz
 PPMCM 10.02500 ppm/cm
 HZCM 1260.72180 Hz/cm



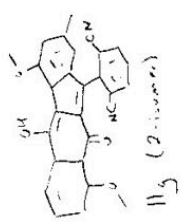
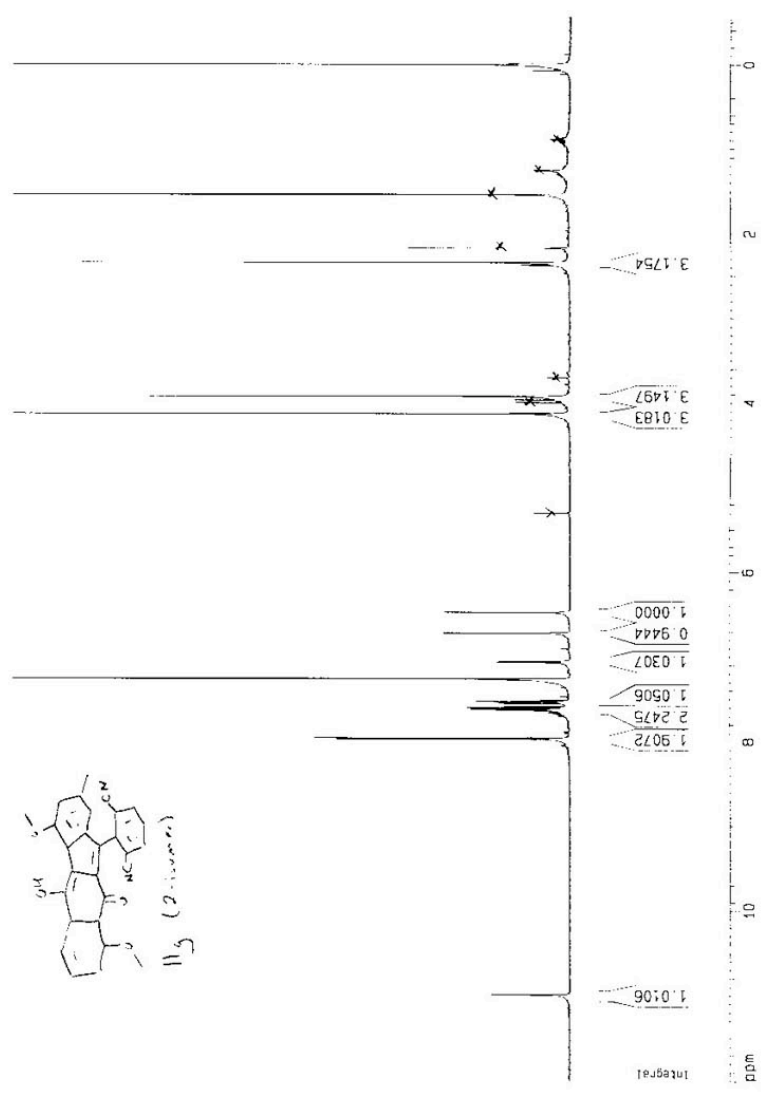
Current Data Parameters
 NAME: SLE-5-12-05
 EXPNO: 6
 PROCNO: 1

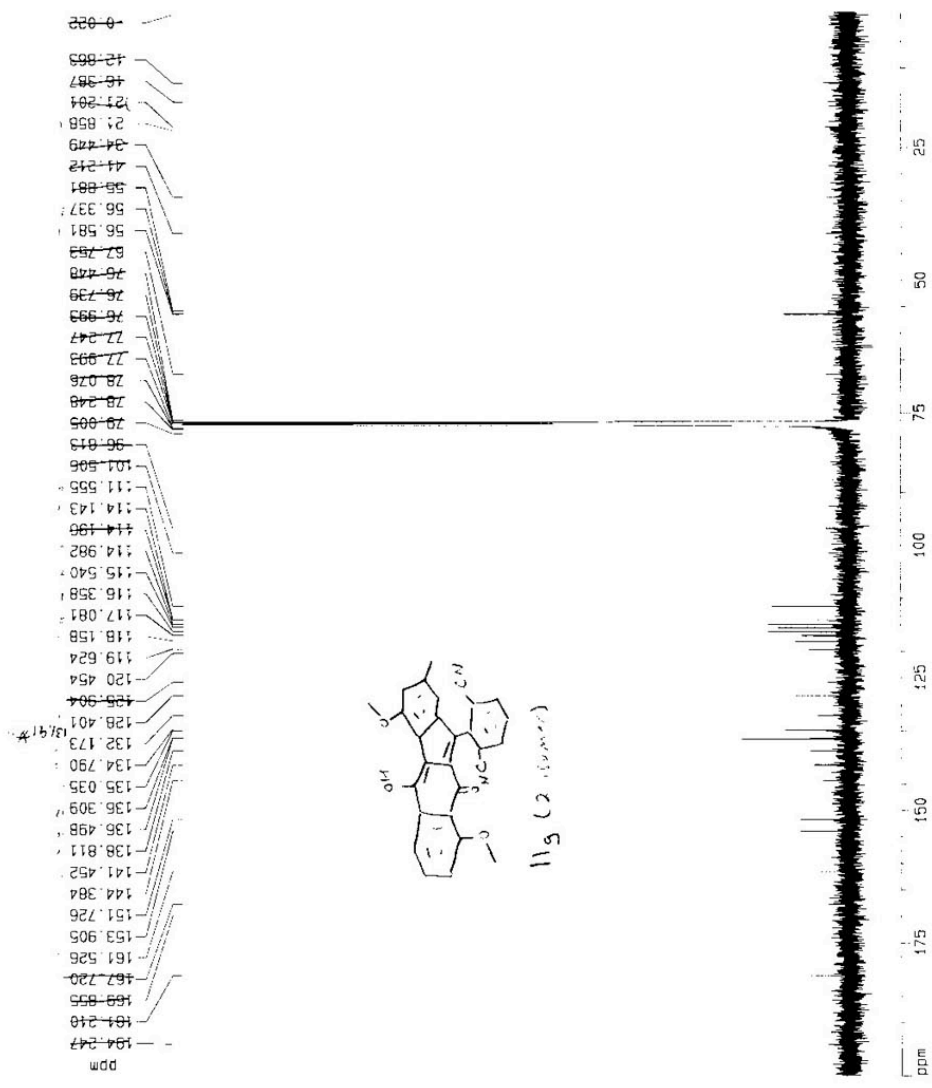
F2 - Acquisition Parameters

Date_: 20050512
 Time: 17.13
 INSTRUM: spect
 PROBHD: 5 mm QNP 1H
 PULPROG: zgpg30
 TD: 32768
 SOLVENT: CDCl3
 NS: 256
 DS: 2
 SWH: 10415.867 Hz
 FIDRES: 0.317851 Hz
 AQ: 1.572640 sec
 RG: 4096
 DA: 48.000 usec
 DE: 66.57 usec
 TE: 300.0 K
 HL: 0.05
 D1: 1.00000000 sec
 F2: 11.75 usec
 SFO1: 500.1320865 MHz
 NUC1: 1H

F2 Processing parameters
 SI: 16384
 SF: 500.1300134 MHz
 MZM: 0
 SSB: 0
 B: 0.00 Hz
 BB: 0
 PC: 1.00

1D NMR plot parameters
 CX: 20.00 cm
 F1P: 17.000 ppm
 F1: 6621.55 Hz
 F2P: -0.550 ppm
 F2: -290.07 Hz
 C13CM: 0.82800 ppm/cm
 M1CM: 314.06167 Hz/cm





Current Data Parameters
 NAME KJF-5-12-05
 EXPNO 8
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20050513
 Time 9:49
 INSTRUM spect
 PROBHD 5 mm QNP 13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8982
 DS 2
 SWH 41888.888 Hz
 FIDRES 0.635783 Hz
 AQ 0.7864820 sec
 RG 32768
 DM 12.000 usec
 DE 17.74 usec
 TE 300.0 K
 LC1 0.08
 J1 2.00000000 sec
 CPOPRG waltz16
 P31 120.00 usec
 S4 55 dB
 D11 0.0300000 sec
 S2 21 dB
 P1 9.79 usec
 S-01 125.736214 MHz
 NUCLEUS 13C
 F2 - Processing Parameters
 SI 32768
 SF 125.757920 MHz
 KM EM
 SS 0
 B 1.00 Hz
 GB 0
 PC 0.50
 LD NMR plot parameters
 CX 20.00 cr
 F1 200.000 phr
 F2 25151.55 Hz
 S2 0.500 ppm
 S3 -62.88 Hz
 GAMMA 10.02500 ppm/cm
 LCEN 1250.72160 Hz/cm

References

- (1) Gould, S. J.; Melville, C. R. *Tetrahedron Lett.* **1997**, 38, 1473-1476