

Two Palladium-Catalyzed Domino Reactions from One Set of Substrates/ Reagents: Efficient Synthesis of Substituted Indenes and *cis*-Stilbenoid Hydrocarbons from the Same Internal Alkynes and Hindered Grignard Reagents

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Supporting Information

General: NMR spectra were recorded on Varian 200 MHz or 600 MHz spectrometers. Chemical shifts were reported in ppm down field from internal tetramethylsilane. All yields reported refer to isolated yields (average of two runs) unless otherwise indicated, and the product purity was estimated to be greater than 95% as determined by ¹H NMR. Melting points were measured on a Fisher-Johns Melting Point Apparatus and uncorrected. Elemental analyses were performed by Atlantic Microlab, Inc. THF was distilled from sodium/benzophenone ketyl. 2,6-Dimethylphenylmagnesium bromide, 2-mesitylmagnesium bromide, 1-phenyl-1-propyne, 3-hexyne, 1,2-dibromomethane, anhydrous iron(III) chloride were purchased from Aldrich and used directly. Pd(OAc)₂ was a gift from Frontier Scientific, Inc. PPh₃ was purchased from Acros Organics and used directly. Anhydrous copper(II) chloride, Anhydrous copper(II) sulfate, silver carbonate, and Iron(III) chloride were purchased from Stem Chemical Inc. and were used as received. Other chemical reagents were purchased from Alfa Aesar and used without further purification. Bromopentamethylbenzene, pentamethylphenylmagnesium bromide were prepared according to reported methods.^{1,2} 2-Bromo-1-ethyl-3-methylbenzene, 2-bromo-1,3-diethylbenzene and 2-bromo-1-isopropyl-3-methylbenzene were prepared according to literature procedures.³ 1,2-Bis(4-methoxyphenyl)acetylene and 4-(1-Hexyn-1-yl)methylbenzene was prepared according to reported method.⁴

General Procedures for Pd(OAc)₂-Promoted Domino Reaction of Diphenylacetylene with Mesitylmagnesium Bromide:

- A. In glove box with nitrogen atmosphere, to a mixture of diphenylacetylene (44.5 mg, 0.25 mmol) and 0.5 ml THF (in a schlenk flask) was added palladium acetate (56 mg, 0.25 mmol). After stirred for 5-10 minutes, Grignard reagent (0.65 mL, 1M in THF, 0.65 mmol) was added. The mixture was allowed to stir at room temperature or 60°C (oil bath) or refluxing for 20 hours. After quenched with water, the reaction mixture was extracted with ethyl acetate (15 mL x 3). The organic layer was washed with brine and the solvent was evaporated under vacuum. The reaction mixtures were analyzed by ¹H NMR, from which the reaction conversion and the ratios of cyclization product : cross-coupling product : self-coupling product were be obtained.
- B. In glove box with nitrogen atmosphere, to a mixture of diphenylacetylene (44.5 mg, 0.25 mmol) and 0.5 mL THF (in a schlenk flask) was added palladium acetate (56 mg, 0.25

mmol) and PPh₃ (2 equiv., 131 mg, 0.5 mmol, or 4 equiv., 262 mg, 1.0 mmol). After stirred for 5-10 minutes, Grignard reagent (1.0 mL, 1M in THF, 1.0 mmol) was added. The mixture was allowed to stir under room temperature or 60°C (oil bath) or refluxing for 20 hours. After quenched with water, the reaction mixture was extracted with ethyl acetate (15 mL x 3). The organic layer was washed with brine and the solvent was evaporated under vacuum. The reaction mixtures were analyzed by ¹H NMR, from which the reaction conversion and the ratios of cyclization product: cross-coupling product : self-coupling product were be obtained.

General Procedure of the Oxidant Screening for Pd(OAc)₂-Catalyzed Domino Reaction of Diphenylacetylene with Mesitylmagnesium Bromide:

In glove box with nitrogen atmosphere, to a mixture of diphenylacetylene (89 mg, 0.5 mmol), oxidant (0.5 mmol) and 0.5 mL THF (in a Schlenk flask) was added palladium acetate (3.4 mg, 0.015 mmol). After stirred for 5-10 minutes, Grignard reagent (1.25 mL, 1M in THF, 1.25 mmol) was added. The mixture was allowed to stir under 60°C (oil bath) for 20 hours. After quenched with water, the reaction mixture was extracted with ethyl acetate (15 mL x 3). The organic layer was washed with brine and the solvent was evaporated under vacuum. The crude reaction mixtures were analyzed by ¹H NMR. Flash chromatography on silica gel (hexane: ethyl acetate = 100: 0 to 90: 10) gave the cyclization products.

General Procedure for Pd(OAc)₂-Catalyzed Annulative Domino Reaction of Internal Alkynes with Hindered Grignard Reagents:

In glove box with nitrogen atmosphere, to a mixture of alkyne (0.5 mmol), 1,2-dibromoethane (0.75 mmol, 65 µl) and 0.5 mL THF (in a schlenk flask) was added palladium acetate (3.4 mg, 0.015 mmol). After stirred for 5-10 minutes, Grignard reagent (1.25 mL, 1M in THF, 1.25 mmol) was added. The mixture was allowed to stir under 60°C (oil bath) for 20 hours. After quenched with water, the reaction mixture was extracted with ethyl acetate (15 mL x 3). The organic layer was washed with brine and the solvent was evaporated under vacuum. Flash chromatography on silica gel (hexane: ethyl acetate = 100: 0 to 90: 10) gave the cyclization products.

4,6-Dimethyl-2,3-diphenyl-1H-indene (1): white solid. m. p.: 86-87°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.403~7.345 (m, 3H), 7.313 (d, J= 7.2 Hz, 2H), 7.209 (s, 1H), 7.178 (d, J= 8.4 Hz, 2H), 7.139 (t, J= 7.2 Hz, 2H), 7.097 (t, J=7.2 Hz, 1H), 6.819 (s, 1H), 3.861 (s, 2H), 2.368 (s, 3H), 1.808 (s, 3H). ¹³C NMR (CDCl₃, 150 MHz): δ 143.118, 141.612, 141.367, 140.054, 139.278, 136.729, 134.756, 131.576, 130.386, 129.620, 128.560, 127.998, 127.851, 127.159, 126.457, 122.188, 40.595, 21.168, 19.834. Anal. calcd. for C₂₃H₂₀: C, 93.20%; H, 6.80%. Found: C, 93.00%; H, 6.69%.

4-Methyl-2,3-diphenyl-1H-indene (2): light yellow solid. M. p.: 118-119°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.412~ 7.367 (m, 4H), 7.325 (d, J= 7.8 Hz, 2H), 7.194 (d, J= 7.8 Hz, 2H), 7.160 (d, J= 7.8 Hz, 2H), 7.127 (t, J= 7.8 Hz, 2H), 6.992 (d, J= 7.8 Hz, 1H), 3.905 (s, 2H), 1.848 (s, 3H). ¹³C NMR (CDCl₃, 150 MHz): δ 144.101, 142.721, 141.478, 141.152, 139.137, 136.584, 131.940, 129.669, 129.553, 128.563, 128.022, 127.969, 127.225, 126.639, 124.933, 121.334, 40.844, 20.012. Anal. calcd. for C₂₂H₁₈: C, 93.57%; H, 6.43%. Found: C, 93.35%; H, 6.47%.

4,5,6,7-Tetramethyl-2,3-diphenyl-1H-indene (3): off-white solid. M. p.: 139-141°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.383 (t, J= 7.2 Hz, 2H), 7.345 (t, J= 7.2 Hz, 1H), 7.292 (d, J=7.2 Hz, 1H), 7.160 (d, J= 7.8 Hz, 2H), 7.134 (t, J= 7.8 Hz, 2H), 7.090 (t, J=7.8 Hz, 1H), 3.795 (s, 2H), 2.377 (s, 3H), 2.299 (s, 3H), 2.194 (s, 3H), 1.795 (s, 3H). ¹³C NMR (CDCl₃, 150 MHz): δ 142.156, 141.415, 140.474, 139.912, 139.147, 137.132, 134.573, 132.119, 129.676, 128.636, 128.205, 128.026, 127.906, 127.011, 126.319, 40.573, 16.368, 16.227, 16.119, 16.048. Anal. calcd. for C₂₅H₂₄: C, 92.54%; H, 7.46%. Found: C, 92.33%; H, 7.45%.

6-Methoxy-4-methyl-2,3-diphenyl-1H-indene (4): white solid. M. p.: 108-109°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.387 (t, J= 7.8 Hz, 2H), 7.361 (t, J= 7.2 Hz, 1H), 7.317 (d, J= 7.8 Hz, 2H), 7.167~ 7.121 (m, 4H), 7.087 (t, J= 7.2 Hz, 1H), 6.980 (d, J= 1.8 Hz, 1H), 6.560 (d, J= 1.8 Hz, 1H), 3.867 (s, 2H), 3.836 (s, 3H), 1.813 (s, 3H). ¹³C NMR (CDCl₃, 150 MHz): δ 157.782, 144.656, 141.132, 139.239, 138.843, 137.561, 136.743, 132.815, 129.592, 128.585, 127.995, 127.686, 127.177, 126.275, 115.188, 107.412, 55.463, 40.813, 20.069. Anal. calcd. for C₂₃H₂₀O: C, 88.43%; H, 6.45%. Found: C, 88.34%; H, 6.34%.

2,3-Bis(4-methoxyphenyl)-4,6-dimethyl-1H-indene (5): yellow solid. M. p.: 116-118°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.211 (d, J= 9.0 Hz, 2H), 7.179 (s, 1H), 7.139 (d, J= 9.0 Hz, 2H), 6.948 (d, J= 8.4 Hz, 2H), 6.802 (s, 1H), 6.703 (d, J= 8.4 Hz, 2H), 3.868 (s, 3H), 3.807 (s, 2H), 3.745 (s, 3H), 2.359 (s, 3H), 1.831 (s, 3H). ¹³C NMR (CDCl₃, 150 MHz): δ 158.670, 158.066, 142.778, 142.009, 139.741, 139.422, 134.233, 131.572, 131.182, 130.691, 130.301, 129.497, 128.890, 122.076, 114.012, 113.419, 55.115, 55.068, 40.419, 21.115, 19.875. Anal. calcd. for C₂₅H₂₄O₂: C, 84.24%; H, 6.79%. Found: C, 83.89%; H, 6.81%.

2,3-bis(4-methoxyphenyl)-4-methyl-1H-indene (6): white solid. M. p.: 131-132°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.354 (d, J= 7.8 Hz, 1H), 7.221 (d, J= 9.0 Hz, 2H), 7.152 (d, J= 8.4 Hz, 2H), 7.089 (t, J= 7.8 Hz, 1H), 6.971 (d, J= 7.2 Hz, 1H), 6.952 (d, J= 9.0 Hz, 2H), 6.710 (d, J= 8.4 Hz, 2H), 3.871 (s, 3H), 3.845 (s, 2H), 3.749 (s, 3H), 1.868 (s, 3H). ¹³C NMR (CDCl₃, 150 MHz): δ 158.742, 158.226, 144.545, 142.407, 140.859, 139.577, 131.580, 131.457, 130.766, 129.499, 129.362, 129.049, 124.506, 121.210, 114.041, 113.466, 55.162, 55.113, 40.667, 20.056. Anal. calcd. for C₂₄H₂₂O₂: C, 84.18%; H, 6.48%. Found: C, 83.84%; H, 6.38%.

2,3-Diethyl-4,6-dimethyl-1H-indene (7): colorless liquid. ¹H NMR (CDCl₃, 200 MHz): δ 7.056 (s, 1H), 6.825 (s, 1H), 3.217 (s, 2H), 2.640 (q, J= 7.6 Hz, 2H), 2.537 (s, 3H), 2.438 (q, J= 7.6 Hz, 2H), 2.320 (s, 3H), 1.130 (t, J= 7.6 Hz, 3H), 1.127 (t, J= 7.6 Hz, 3H). ¹³C NMR (CDCl₃, 150 MHz): δ 143.869, 143.233, 141.288, 139.182, 132.951, 129.939, 129.173, 122.138, 39.376, 21.261, 20.970, 19.889, 19.590, 15.290, 14.644.

2,3-Diethyl-4-methyl-1H-indene (8): colorless liquid. ¹H NMR (CDCl₃, 200 MHz): δ 7.230 (t, J= 3.6 Hz, 1H), 7.004 (d, J= 3.6 Hz, 1H), 7.699 (d, J=3.6 Hz, 1H), 3.256 (s, 2H), 2.667 (q, J= 7.2 Hz, 2H), 2.582 (s, 3H), 2.462 (q, J=7.2 Hz, 2H), 1.144 (t, J= 7.2 Hz, 6H). ¹³C NMR (CDCl₃, 150 MHz): δ 144.397, 143.993, 143.470, 139.447, 129.575, 129.175, 123.400, 121.185, 39.575, 21.298, 19.901, 19.750, 15.320, 14.625.

3-Butyl-4,6-dimethyl-2-*p*-tolyl-1H-indene/2-butyl-4,6-dimethyl-3-*p*-tolyl-1H-indene (9): ¹H NMR showed a 91: 9 ratio. Analytic sample of 3-butyl-4,6-dimethyl-2-*p*-tolyl-1H-indene was obtained by recrystallization of the mixture of 3-butyl-4,6-dimethyl-2-*p*-tolyl-1H-indene and 2-butyl-4,6-dimethyl-3-*p*-tolyl-1H-indene in hexanes. white solid. M. p.: 81-82°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.276 (d, J= 7.8 Hz, 2H), 7.204 (d, J= 7.8 Hz, 2H), 7.127 (s, 1H), 6.890 (s, 1H), 3.600 (s, 2H), 2.733 (t, J= 7.8 Hz, 2H), 2.583 (s, 3H), 2.382 (s, 3H), 2.352 (s, 3H), 1.604

(m, 2H), 1.375 (m, 2H), 0.894 (t, J = 7.2 Hz, 3H). ^{13}C NMR (CDCl_3 , 150 MHz): δ 144.160, 141.320, 140.432, 140.397, 136.163, 135.401, 133.986, 130.434, 130.300, 128.973, 128.267, 122.222, 41.700, 33.074, 27.482, 22.795, 21.166, 21.051, 19.720, 13.889. The structure of 3-butyl-4,6-dimethyl-2-*p*-tolyl-1H-indene was established by NOE effect: NOE effect observed when irradiated at the peak at δ 3.600 ppm: 7.276 (d, 0.60%), 7.127 (s, 0.65%). Anal. calcd. for $\text{C}_{22}\text{H}_{26}$: C, 90.98%; H, 9.02%. Found: C, 90.74%; H, 9.05%.

3,4,6-Trimethyl-2-phenyl-1H-indene (10): ^1H NMR showed a 92: 8 ratio. Analytic sample of 3,4,6-trimethyl-2-phenyl-1H-indene was obtained by recrystallization of the mixture of 3,4,6-trimethyl-2-phenyl-1H-indene and 2,4,6-trimethyl-3-phenyl-1H-indene in hexanes. White solid. m.p.: 108-110°C. ^1H NMR (CDCl_3 , 600 MHz) δ 7.414 ~ 7.389 (m, 4H), 7.271 (t, J = 6.6 Hz, 1H), 7.130 (s, 1H), 6.881 (s, 1H), 3.633 (s, 3H), 2.618 (s, 3H), 2.419 (s, 3H), 2.357 (s, 3H). ^{13}C NMR (CDCl_3 , 150 MHz) δ 143.6, 142.0, 140.2, 138.0, 136.0, 134.3, 130.9, 130.2, 128.6, 128.2, 126.4, 122.2, 41.2, 21.1, 20.3, 15.6. The structure of 3,4,6-trimethyl-2-phenyl-1H-indene was established by NOE effect: NOE effect observed when irradiated at the peak at δ 3.633 ppm: 7.400~7.390 (m, 0.9%), 7.130 (s, 0.65%).

3,4-Dimethyl-2-phenyl-1H-indene (11): ^1H NMR showed a 90: 10 ratio. Analytic sample of 3,4-dimethyl-2-phenyl-1H-indene was obtained by recrystallization of the mixture of 3,4-dimethyl-2-phenyl-1H-indene and 2,4-dimethyl-3-phenyl-1H-indene in hexanes. White solid. m.p.: 75-77°C. ^1H NMR (CDCl_3 , 600 MHz) δ 7.426 ~ 7.390 (m, 4H), 7.311 (d, J = 7.2 Hz, 1H), 7.282 (d, J = 7.2 Hz, 1H), 7.093 (t, J = 7.2 Hz, 1H), 7.058 (d, J = 7.2 Hz, 1H), 3.674 (s, 2H), 2.665 (s, 3H), 2.440 (s, 3H). ^{13}C NMR (CDCl_3 , 150 MHz) δ 144.6, 143.3, 141.3, 137.9, 136.2, 131.3, 129.4, 128.6, 128.3, 126.6, 124.6, 121.4, 41.4, 20.4, 15.6. The structure of 3,4-dimethyl-2-phenyl-1H-indene was established by NOE effect: NOE effect observed when irradiated at the peak at δ 3.674 ppm: 7.421~7.410 (m, 0.8%), 7.311 (d, 0.6%).

3-Ethyl-4-methyl-2-phenyl-1H-indene (12): ^1H NMR showed a 89: 11 ratio. Analytic sample of 3-ethyl-4-methyl-2-phenyl-1H-indene was obtained by recrystallization of the mixture of 3-ethyl-4-methyl-2-phenyl-1H-indene and 2-ethyl-4-methyl-3-phenyl-1H-indene in hexanes. White solid. m. p.: 46-47°C. ^1H NMR (CDCl_3 , 600 MHz) δ 7.405 ~ 7.397 (m, 4H), 7.316 (d, J = 6.6 Hz, 1H), 7.297 (m, 1H), 7.103 (t, J = 7.2 Hz, 1H), 7.074 (d, J = 7.2 Hz, 1H), 3.659 (s, 2H), 2.789 (q, J = 7.2 Hz, 2H), 2.654 (s, 3H), 1.269 (t, J = 7.2 Hz, 3H). ^{13}C NMR (CDCl_3 , 150 MHz) δ 143.8, 143.6, 142.4, 141.2, 138.1, 130.8, 129.6, 128.4, 128.3, 126.7, 124.5, 121.4, 41.9, 20.6, 19.8, 15.6.

3,4-Diethyl-2-phenyl-1H-indene (13): ^1H NMR showed a 85: 15 ratio. Analytic sample of 3,4-diethyl-2-phenyl-1H-indene was obtained by recrystallization of the mixture of 3,4-diethyl-2-phenyl-1H-indene and 2,4-diethyl-3-phenyl-1H-indene in hexanes. White solid. m. p.: 65-66°C. ^1H NMR (CDCl_3 , 600 MHz) δ 7.425 ~ 7.389 (m, 4H), 7.323 (d, J = 7.8 Hz, 1H), 7.307 ~ 7.292 (m, 1H), 7.161 (t, J = 7.8 Hz, 1H), 7.144 (d, J = 7.2 Hz, 1H), 3.661 (s, 2H), 2.977 (q, J = 7.8 Hz, 2H), 2.768 (q, J = 7.2 Hz, 2H), 1.300 (t, J = 7.2 Hz, 3H), 1.261 (t, J = 7.8 Hz, 3H). ^{13}C NMR (CDCl_3 , 150 MHz) δ 144.0, 142.4, 142.1, 141.6, 138.2, 137.7, 128.4, 128.3, 127.8, 126.7, 124.7, 121.3, 42.0, 25.5, 20.6, 17.1, 15.6. The structure of 3,4-diethyl-2-phenyl-1H-indene was established by NOE effect: NOE effect observed when irradiated at the peak at δ 3.661 ppm: 7.412~7.400 (m, 0.6%), 7.323 (d, 0.3%).

4-Ethyl-2,3-diphenyl-1H-indene (14): Off-white solid. m. p.: 80-81°C. ¹H NMR (CDCl₃, 600 MHz) δ 7.403 ~ 7.365 (m, 4H), 7.344 (d, J = 7.8 Hz, 1H), 7.341 (d, J = 7.8 Hz, 1H), 7.181 (t, J = 7.2 Hz, 2H), 7.156 ~ 7.140 (m, 3H), 7.130 ~ 7.108 (m, 1H), 7.069 (d, J = 7.8 Hz, 1H), 3.902 (s, 2H), 2.216 (q, J = 7.2 Hz, 2H), 0.872 (t, J = 7.2 Hz, 3H). ¹³C NMR (CDCl₃, 150 MHz) δ 143.4, 143.0, 141.5, 141.3, 139.2, 138.6, 136.6, 129.5, 128.5, 128.1, 128.0, 127.9, 127.3, 126.6, 125.1, 121.2, 41.0, 25.0, 16.3.

4-Isopropyl-2,3-diphenyl-1H-indene (15): White solid. m. p.: 79-80°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.400 ~ 7.358 (m, 4H), 7.337 ~ 7.323 (m, 2H), 7.238 ~ 7.221 (m, 2H), 7.144 ~ 7.137 (m, 4H), 7.120 ~ 7.097 (m, 1H), 3.895 (s, 2H), 2.739 (m, J = 6.6 Hz, 1H), 0.961 (d, J = 6.6 Hz, 6H). ¹³C NMR (CDCl₃, 150 MHz): δ 143.7, 142.9, 142.8, 141.6, 141.3, 139.5, 136.7, 129.3, 128.6, 128.1, 127.9, 127.3, 126.6, 125.3, 124.2, 121.1, 41.0, 26.8, 24.1.

General Procedure for Pd(OAc)₂-Catalyzed Domino Carbopalladation-Cross-Coupling Reactions of Internal Alkynes with Hindered Grignard Reagents:

In glove box with nitrogen atmosphere, to a mixture of alkyne (0.5 mmol), 1,2-dibromoethane (1.5 mmol, 130 μl and 0.5 mL THF (in a schlenk flask) was added palladium acetate (3.4 mg, 0.015 mmol) and PPh₃ (26.2 mg, 0.1 mmol). After stirred for 5-10 minutes, Grignard reagent (2.0 mL, 1M in THF, 2.0 mmol) was added. The mixture was allowed to stir under refluxing for 20 hours. After quenched with water, the reaction mixture was extracted with ethyl acetate (15 mL x 3). The organic layer was washed with brine and the solvent was evaporated under vacuum. Flash chromatography on silica gel (hexane: ethyl acetate = 100: 0 to 90: 10) gave the cross-coupling products.

1,2-Bis(2,4,6-trimethylphenyl)stilbene (16): white solid. M. p.: 177-178.5°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.047~ 7.025 (m, 6H), 6.928~ 6.911 (m, 4H), 6.641 (s, 4H), 2.167 (s, 6H), 2.052 (s, 12H). ¹³C NMR (CDCl₃, 150 MHz): δ 142.812, 140.323, 138.228, 136.729, 135.893, 131.150, 128.595, 127.152, 125.828, 21.722, 20.890. Anal. calcd. for C₃₂H₃₂: C, 92.26%; H, 7.74%. Found: C, 92.22%; H, 7.78%.

1,2-Bis(pentamethylphenyl)stilbene (17):⁵ white solid. m.p.: 252-254°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.057~ 7.027 (m, 6H), 7.000~ 6.987 (m, 4H), 2.110 (s, 12H), 2.105 (s, 6H), 2.009 (s, 12H). ¹³C NMR (CDCl₃, 150 MHz): δ 143.304, 141.545, 138.971, 132.803, 131.806, 131.761, 130.929, 127.176, 125.656, 20.026, 16.631, 16.413.

1,2-Bis(2,6-dimethylphenyl)-1,2-bis(4-methoxyphenyl)ethylene (18): white solid. M. p.: 204-206°C. ¹H NMR (CDCl₃, 600 MHz): δ 6.918 (t, J= 7.2 Hz, 2H), 6.873 (d, J= 9.0 Hz, 4H), 6.800 (d, J= 7.2 Hz, 4H), 6.621 (d, J= 9.0 Hz, 4H), 3.748 (s, 6H), 2.080 (s, 12H). ¹³C NMR (CDCl₃, 150 MHz): δ 157.589, 141.201, 138.863, 136.904, 135.054, 132.133, 127.587, 126.457, 112.713, 55.076, 21.782. Anal. calcd. for C₃₂H₃₂O₂: C, 85.68%; H, 7.19%. Found: C, 85.38%; H, 7.16%.

1,2-Bis(2,4,6-trimethylphenyl)-1,2-bis(4-methoxyphenyl)ethylene (19): white solid. M. p.: 195.5-196.5°C. ¹H NMR (CDCl₃, 600 MHz): δ 6.838 (d, J= 9.0 Hz, 4H), 6.627 (s, 4H), 6.598 (d, J= 9.0 Hz, 4H), 3.737 (s, 6H), 2.161 (s, 6H), 2.029 (s, 12H). ¹³C NMR (CDCl₃, 50 MHz): δ 157.434, 138.892, 138.573, 136.715, 135.646, 135.585, 132.165, 128.540, 112.645, 55.079, 21.711, 20.900. Anal. calcd. for C₃₄H₃₆O₂: C, 85.67%; H, 7.61%. Found: C, 85.79%; H, 7.64%.

3,4-Bis(2,4,6-trimethylphenyl)hex-3-ene (20):⁵ white solid. M. p.: 165-166°C. ¹H NMR (CDCl₃, 600 MHz): δ 6.648 (s, 4H), 2.472 (q, J= 7.2 Hz, 4H), 2.154 (s, 6H), 2.064 (s, 12H), 1.016 (t, J= 7.2 Hz, 6H). ¹³C NMR (CDCl₃, 150 MHz): δ 138.615, 138.471, 135.697, 134.880, 128.083, 28.228, 20.926, 20.803, 13.382.

3,4-Bis(pentamethylphenyl)hex-3-ene (21):⁵ white solid. M. p.: 175-177°C. ¹H NMR (CDCl₃, 600 MHz): δ 2.498 (q, J= 7.2 Hz, 4H), 2.112 (s, 6H), 2.025 (s, 12H), 2.004 (s, 12H), 1.059 (t, J= 7.2 Hz, 6H). ¹³C NMR (CDCl₃, 150 MHz): δ 139.449, 139.330, 131.965, 131.600, 131.112, 29.343, 19.647, 16.544, 16.442, 13.272.

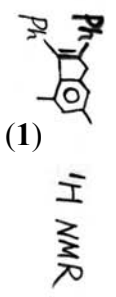
1,2-Bis(2,6-dimethylphenyl)prop-1-ene (22): white solid. m.p.: 120-121°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.295 (d, J= 4.2 Hz, 4H), 7.192 (q, J= 4.2 Hz, 1H), 6.929 (t, J= 7.8 Hz, 1H), 6.890 (t, J= 7.8 Hz, 1H), 6.852 (d, J= 7.8 Hz, 2H), 6.774 (d, J= 7.8 Hz, 2H), 2.216 (s, 3H), 2.202 (s, 6H), 2.084 (s, 6H). ¹³C NMR (CDCl₃, 150 MHz): δ 141.991, 141.966, 140.920, 137.862, 136.641, 135.777, 135.257, 130.090, 127.404, 127.383, 127.355, 126.316, 126.102, 126.032, 22.750, 21.778, 21.068. The stereochemistry of 1,2-bis(dimethylphenyl)prop-1-ene was established by NOE effect: NOE effect observed when irradiated at the peak at δ 2.084 ppm: 7.295 (d, 0.33%), 6.774 (d, 0.32%), 2.202 (s, 0.75%). Anal. calcd. for C₂₅H₂₆: C, 91.97%; H, 8.03%. Found: C, 91.41%; H, 7.94%.

1,2-Bis(2,4,6-trimethylphenyl)prop-1-ene (23): white solid. M. p.: 133-135°C. ¹H NMR (CDCl₃, 600 MHz): δ 7.275~7.259 (m, 4H), 7.181~7.153 (m, 1H), 6.682 (s, 2H), 6.598 (s, 2H), 2.173 (s, 3H), 2.165 (s, 3H), 2.162 (s, 6H), 2.132 (s, 3H), 2.041 (s, 6H). ¹³C NMR (CDCl₃, 150 MHz): δ 142.475, 139.287, 138.273, 137.883, 136.416, 135.686, 135.454, 135.296, 135.106, 130.086, 128.320, 128.306, 127.292, 125.842, 23.020, 21.697, 20.988, 20.837, 20.819. Anal. calcd. for C₂₇H₃₀: C, 91.47%; H, 8.53%. Found: C, 91.58%; H, 8.52%.

1,2-Bis(pentamethylphenyl)prop-1-ene (24): white solid. M. p.: 177-179 °C. ¹H NMR (CDCl₃, 600 MHz): δ 7.329 (d, J= 7.2 Hz, 2H), 7.275 (t, J= 7.2 Hz, 2H), 7.156 (t, J= 7.2 Hz, 1H), 2.219 (s, 3H), 2.123(s, 6H), 2.118 (s, 3H), 2.085 (s, 3H), 2.073 (s, 6H), 2.053 (s, 6H), 1.996 (s, 6H). ¹³C NMR (CDCl₃, 150 MHz): δ 143.063, 140.114, 138.966, 138.927, 137.112, 132.447, 132.362, 131.962, 131.716, 131.615, 130.375, 129.933, 127.307, 125.598, 24.145, 20.062, 19.641, 16.611, 16.513, 16.453, 16.380. The stereochemistry of 1,2-bis(pentamethylphenyl)prop-1-ene was established by NOE effect: NOE effect observed when irradiated at the peak at δ 2.219 ppm: 7.329 (d, 0.7%), 2.123 (s, 1.0%).

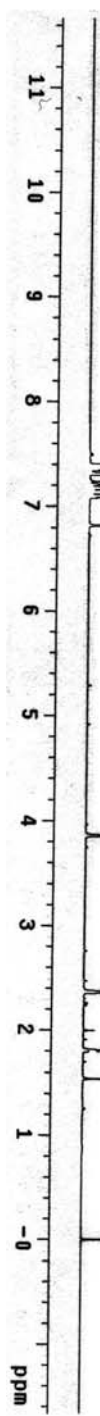
Reference:

1. Miller, A.R.; Curtin D.Y. *J. Am. Chem. Soc.* **1976**, 98, 1860-1865.
2. Hawkins, R. T.; Lennarz, W. J.; Snyder, H. R. *J. Am. Chem. Soc.* **1960**, 82, 3053-3059.
3. Furniss, B.S.; Hannaford, A. J.; Smith, P.W.G.; Tatchell, A.R. *Vogel's Textbook of Practical Organic Chemistry* (5th ed.), **1989**, P933-935.
4. Sonogashira, K.; Tohda, Y.; Hagihara, N. *Tetrahedron Lett.* **1975**, 4467.
5. Rathore, R.; Deselnicu, M. I.; Burns, C. L. *J. Am. Chem. Soc.* **2002**, 124, 14832-14833.



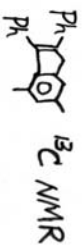
Dong_PIV_102_1H
 exp1 s2pu1

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file	ACQUISITION	exp	dpwr	nm	0
sfreq	599.942	dm	dot	nm	0
tn	H1	dmm		C	200
at	1.892	dseq		1.0	n
nd	3.027	dres			
sw	8000	homo			
fb	4000				
bs	32				
tpwr	56	dfreq2	DEC2	0	
pw	5.5	dn2			
d1	0	dpwr2		1	
tof	0	dot2		n	
nt	16	dmm2		C	
ct	0	dmf2		200	
atlock	n	dseq2		1.0	
gain	not used	dres2		n	
FLAGS		homo2	DEC3	0	
fl	n	dfreq3			
in	Y	dn3			
dp	Y	dpwr3		1	
hs	nm	dot3		n	
DISPLAY	-999.7	dmm3		C	
SP	7999.5	dmf3		200	
WP	102	dseq3		1.0	
VS	35	dres3		n	
SC	215	homo3	PROCESSING		
WC	1.20	wtfile			
h2mm	102.67	proc	not used		
IS	1000.1	fn			
rfl	0	math			
th	6	werr			
ins	6.000	wexp			
nm	cdc	wbs			
ph		wnt			
		wft			



Dong_PTV_102_13C

exp2 szpu1



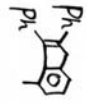
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 date Jun 14 2006 dfrq 599.942
 solvent CDC13 dn H1
 file ACQUISITION exp dpvr 39
 sfrq 150.871 dm 0
 tn 1.305 dmf mny
 nt 90272 dseq 16667
 SW 34707.2 dres 1.0
 fb 13000 homo n
 bs 8 DECC2 0
 tpr 54 dfrq2 0
 pw 6.0 dn2 1
 dl 3.000 dpvr2 0
 tof 3017.4 dot2 n
 nt 10000 dm2 n
 ct 0 dmm2 C
 alock n dmr2 16667
 gain not used dseq2 1.0
 flags n dres2
 n homo2 DECC3 0
 n n dfrq3
 n y dn3
 n y dpvr3 1
 n dn3 0
 DISPLAY -875.1 dm3 n
 SP 34706.6 dms3 n
 WP 145 dmm3 C
 VS 0 dmfr3 16667
 SC 0 dseq3
 WC 250 dres3 1.0
 hzmm 138.83 homo3
 IS 500.00 PROCESSING n
 rf1 12491.4 lb Wtfile 0.50
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 th 6
 rms 100.000
 nm cdc ph
 match
 werr
 wexp
 wbs
 wnt



Dong_PIV_108_1H

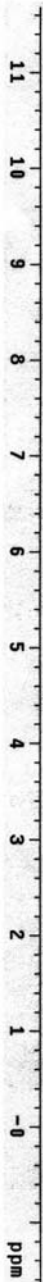
exp1 szpu1

SAMPLE	date	May 13 2006	dfreq	DEC. & VI	599.942
solvent	May 13 2006	dfreq <td>599.942</td> <td>HI</td> <td>0</td>	599.942	HI	0
FT1	CDCl3	dm	599.942	HI	0
ACQUISITION	exp	dpwr	599.942	HI	0
dfreq	599.942	dm	599.942	HI	0
tn	HI	dmm	599.942	HI	0
at	1.892	dntf	599.942	HI	0
np	30272	dseq	599.942	HI	0
sw	8000.0	dres	599.942	HI	0
td	4000	homo	599.942	HI	0
tpwr	55	dfreq2	599.942	HI	0
pw	6.0	dn2	599.942	HI	0
d1	3.000	dpwr2	599.942	HI	0
tof	0	dof2	599.942	HI	0
nt	8	dn2	599.942	HI	0
ct	8	dmm2	599.942	HI	0
atlock	not used	n	599.942	HI	0
gain	not used	n	599.942	HI	0
l1	n	homo2	599.942	HI	0
in	n	dfreq3	599.942	HI	0
dp	y	dn3	599.942	HI	0
hs	n	dpwr3	599.942	HI	0
DISPLAY	7981.7	dof3	599.942	HI	0
sp	7981.7	dm3	599.942	HI	0
vp	7981.7	dmm3	599.942	HI	0
vc	0	dof3	599.942	HI	0
sc	0	dmm3	599.942	HI	0
wc	220	dseq3	599.942	HI	0
hzm	0.35	dres3	599.942	HI	0
ls	343.34	homo3	599.942	HI	0
rfl	992.2	WfFile	599.942	HI	0
ft	30	Proc	599.942	HI	0
in	30	ft	599.942	HI	0
ms	2.000	math	599.942	HI	0
mm	2.000	math	599.942	HI	0
WERT					
WEXP					
WDS					
WNT					
WFL					



(2)

¹H NMR

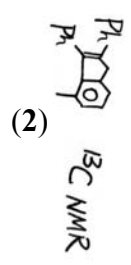


Dong_PIV_105_13C

```

exp2 s2pu1
SAMPLE
date May 13 2006 dfrq DEC. & VT 599.942
solvent 13CDCl3 dn HI
file ACQUISITION exp dpwr 39
sfrq 150.871 dm dof 0
in C13 dnm nny
at 1.309 dnf W
np 30272 dsdq 16667
rh 3418006 Res 1.0
bs 18000 HOMO n
lpwr 61 dfrq2 DEC2 0
d1 3.000 dpwr2 1
lof 3017.4 dof2 0
nt 5000 dm2 C
clock 684 dnm2 n
alock not used dmf2 16667
gath FLAGS n dmf2
11 n homoz 1.0
in n DEC3 0
dp y dfrq3
hs nm dn3
DISPLAY -876.8 dpwr3 1
sp 34706.8 dms 0
wp 4 dmf3 n
se 0 dsdq 16667
vc 250 dres3 C
hzzm 1.28 dres3 1.0
is 500.00 homos
rf1 12493.1 wflie
rfp 11615.8 pproc
th 7 n
ins cdc ph 100.000 math not used f

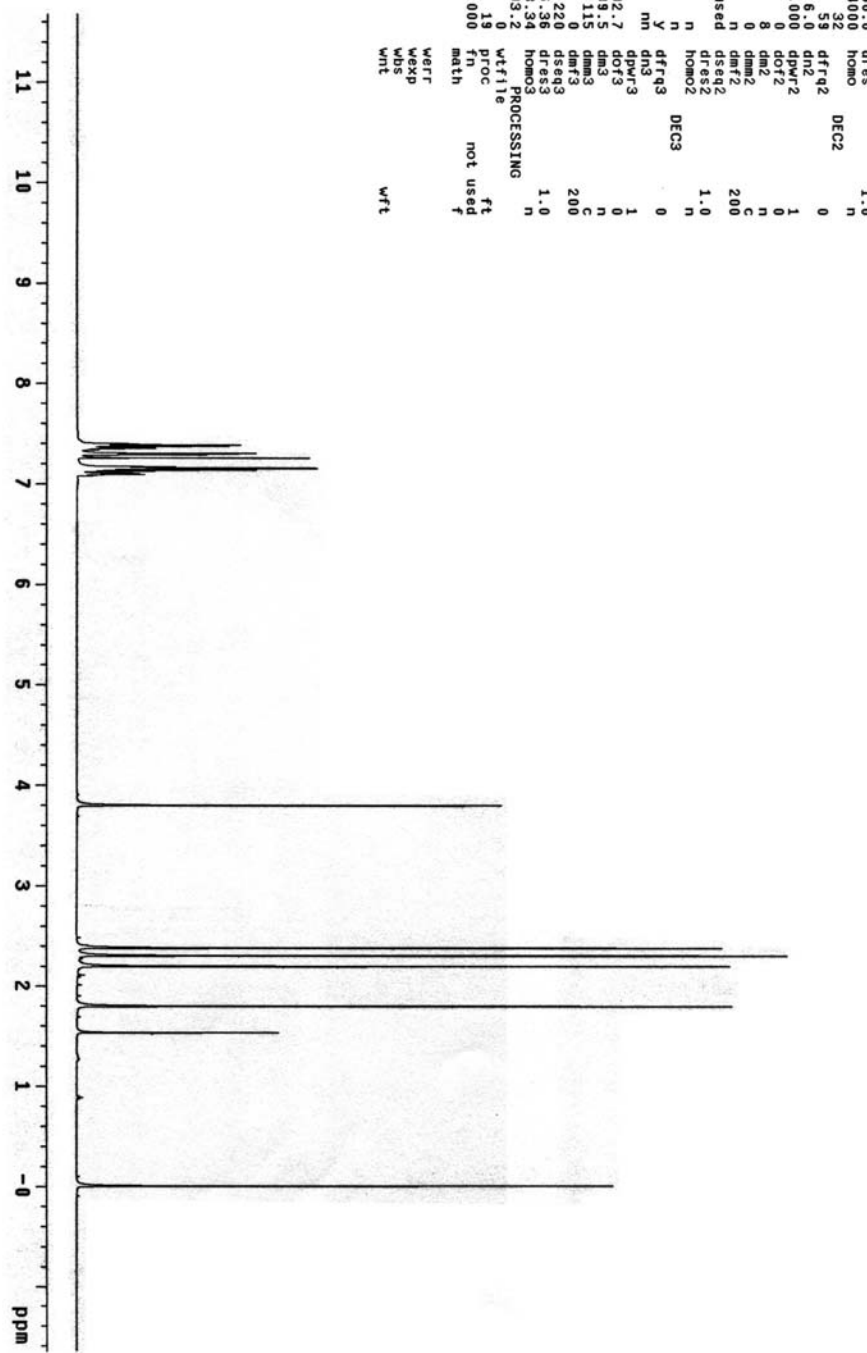
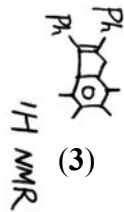
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Dong_PIV_104_1H

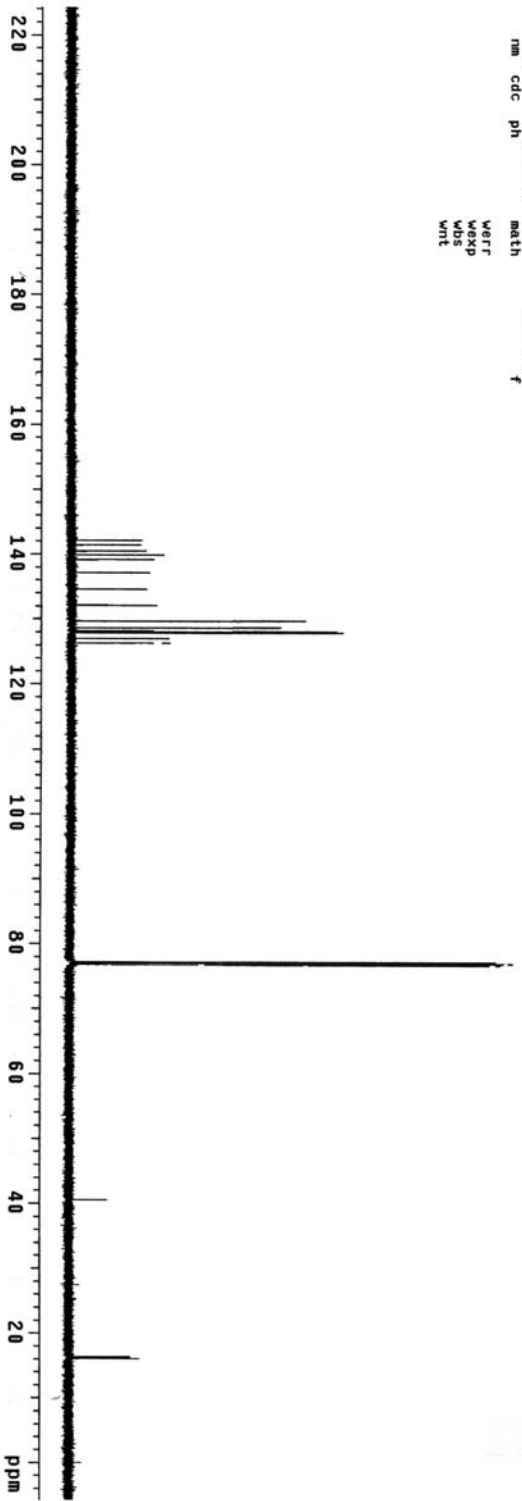
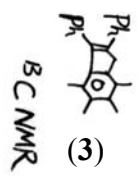
expl szpu1

date	SAMPLE	DEC. & VT
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solvent	CDC13	H1
f1	exp	30
ft1	exp	30
sfreq	599.942	mm
tn	H1	mm
at	1.892	dfes
np	30272	dfes
sw	8000.0	dfes
fb	4000	homo
bs	32	DEC2
tpwr	39	dfes2
pw	6.0	dm2
hi	3.000	dm2
tof	8	dm2
nt	8	dm2
ct	0	dm2
atlock	n	dm2
gain	not used	dm2
flags	not used	dm2
l1	n	homo2
l2	n	homo2
l3	n	homo2
l4	n	homo2
l5	n	homo2
l6	n	homo2
l7	n	homo2
l8	n	homo2
l9	n	homo2
l10	n	homo2
l11	n	homo2
l12	n	homo2
l13	n	homo2
l14	n	homo2
l15	n	homo2
l16	n	homo2
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l94	n	homo2
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l100	n	homo2



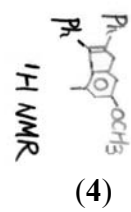
Dong_PIV_104_13C
exp2 szpu1

date	SAMPLE	5 2006	dfrc	DEC. & VT	599.942
solvent	CDCl3	exp	dn	HI	1
f1	ACQUISITION	exp	ddp	30	0
sfrcq	150.871	dm	dm	my	0
tn	C13	dmm	dm	w	1
at	1.300	dmt	dres	16667	1.0
np	90272	dseg	dres	1.0	0
sv	34707.2	dres	homo	DEC2	0
fb	19000	homo	homo	DEC3	0
vs	6	dfrc2	dfrc2	1	0
pw	3.000	dpwr2	dpwr2	1	0
dl	3017.4	dof2	dof2	n	0
nt	10000	dm2	dm2	n	0
ct	2632	dmm2	dmm2	c	16667
alock	not used	dmm2	dres2	1.0	0
gain	not used	dres2	dres2	1.0	0
ll	n	homo2	homo2	DEC3	0
ln	n	dfrc3	dfrc3	1	0
dn	y	dm3	dm3	n	0
hs	n	dfrc3	dfrc3	n	0
sp	DISPLAY	nm	nm	1	0
wp	-875.7	dof3	dof3	n	0
vs	34706.6	dm3	dm3	n	0
vc	73	dmm3	dmm3	c	16667
sc	0	dfr3	dfr3	1.0	0
wc	250	dseg3	dseg3	1.0	0
h2mm	16.65	dres3	dres3	1.0	0
h1	510.00	homo	homo	PROCESSING	n
rfl	12482.0	wfille	wfille	4	0
th	11615.6	proc	proc	not used	f
ins	100.000	fn	fn	math	0
nm	cdc	ph	ph	math	0
		weir	weir	0	0
		wexp	wexp	0	0
		wos	wos	0	0
		wnt	wnt	0	0

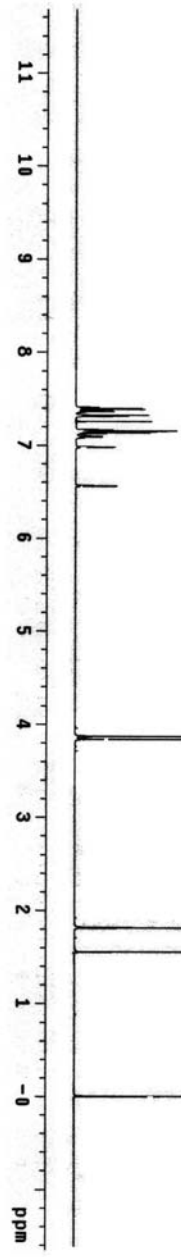


00mg_PIV_112_1M

expl s2pu1



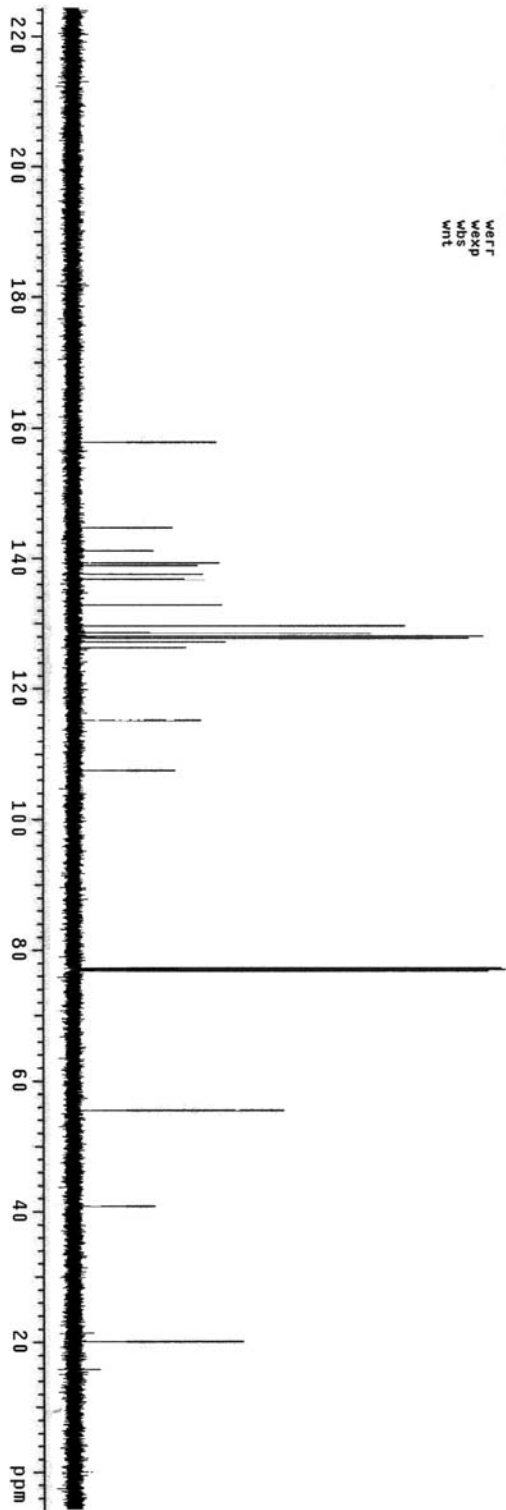
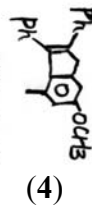
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 date Jun 7 2006 dfrq 599.942
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 file ACQUISITION exp dpwr H1
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 in H1 dnm mnm
 at 1.892 dmf 200
 np 30272 dseq 1.0
 sw 8000.0 dres homo
 fb 4000
 ds 32
 dpwr 59 dfrq2 DEC2 0
 di 57 dfrq2
 dt 0 dof2 1
 tof 0 dof2 0
 nt 8 dnm2 n
 ct 8 dnm2 n
 alock not used dnm2 200
 gain not used dseq2 1.0
 l1 n homo2 DEC3 0
 in n
 dn Y dfrq3
 dp n dn3 dpwr3 1
 hs nm
 DISPLAY
 sp -991.7 dof3 0
 wp 7999.5 dnm3 n
 wv 135 dnm3 n
 sc 215 dfrq3 200
 wc 0.23 dres3 1.0
 hzmm 39.57 homo3
 ffr1 992.2
 ffr2 0
 ffr3 22
 pproc not used
 fts math
 nm cdc ph 3.000
 werr
 wexp
 wds
 wnt wft



Donq_PIV_112_13C

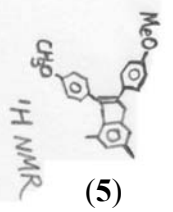
exp2 s2pul

date	SAMPLE	7 2006	dfreq	DEC. & VT	599.942
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solvent	CDC13		dm2	DEC2	0
file	ACQUISITION	exp	dm3	DEC3	0
sfreq	150.871	dm	dmf		
tn	C13	dm	dmf		
at	1.300	dmf	dseq		
np	90272	dseq	dres		
sw	34707.2	dres	homo		
fb	19000	homo			
bs	8				
tpwr	61	dfreq2			
pw	3.000	dm2			
tof	3017.4	dm2			
nt	30000	dm2			
ct	464	dm2			
atlock	n	dmf2			
gain	not used	dseq2			
11	FLAGS	dres2			
in	n	homo2			
dp	y	dfreq3			
hs	DISP	dm3			
SP	DISPLAY	dm3			
WD	-875.6	dm3			
VS	34706.6	dm3			
SC	71	dmf3			
WC	250	dseq3			
h2mm	27.48	dres3			
15	500.00	homo3			
rfl	1291.9	math			
trf	1015.8	math			
ins	100.000	math			
nm	cdc	math			
ph	100.000	math			
	werr				
	wexp				
	wds				
	wnt				



Dong_P1V_101_1H
 exp1 s2pu1

SAMPLE DEC. & VT
 date May 21 2006 dfrq 399.942
 solvent CHCl3 exp 30
 file C0013 dpr 30
 ACQUISITION dof 0
 sfrq 599.942 dm
 tn H1 dm
 at 1.892 dnt 200
 np 3.0272 dseq 200
 sp 80000 dres 1.0
 fb 40000 homo
 bs 4032
 tpwr 59 dfrq2 DEC2
 pw 6.0 dh2 0
 di 3.000 dpwr2 1
 tof 0 dof2 0
 nt 8 dm2 n
 st 0 dm3 n
 alock 0 dres2 200
 gain not used dres3 1.0
 flags
 i1 n homo2 n
 i2 n dfrq3 DEC3
 i3 y dfrq3 0
 dp n dh3 1
 ds dpwr3 0
 sp 2.000 dnt 0
 wp 7999.5 dnt3 n
 vs 87 dnt3 n
 sc 0 dnt3 200
 wc 220 dseq3 n
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 is 40.59 homo3
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 rfp 3
 th fn not used ft
 ins cdc ph 6.000 math
 warr
 wexp
 wds
 wnt wft



Dong_PIV_101_13C

exp2 s2pu1

SAMPLE date May 21 2006
solvent CDCl3
file ACQUISITION exp

sfreq 150.821
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 61
pw 5.0
d1 0.00
tof 3017.4
nt 30000
ct 992
alock n
gain not used

DEC 599.942
dfrq
dn
dpcr
dof
dmm
dmf
dseq
dres
homo

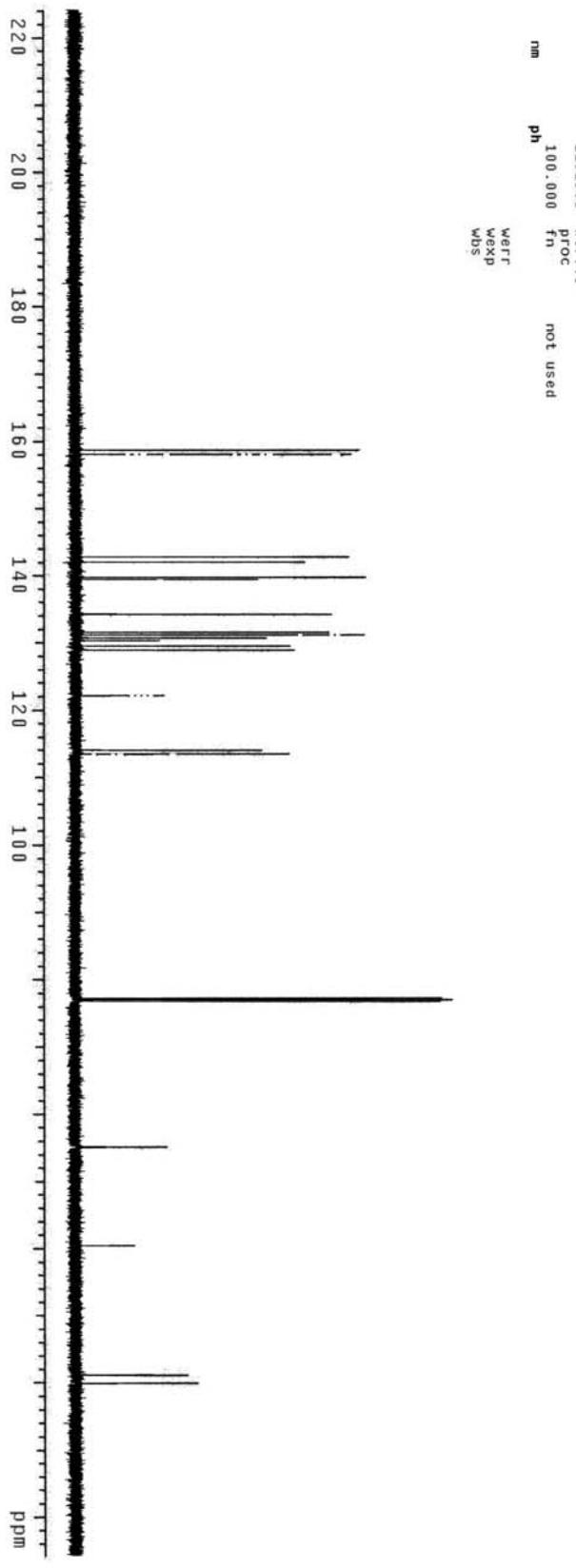
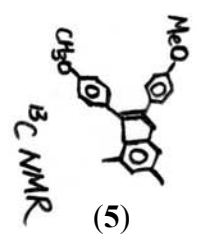
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dn3
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dmm3
dmf3
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590.00
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PROCESsing
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proc fn
nm ph
100.000
not used

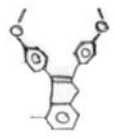
WEFT
wexp
wds



Dong_VIV_P4_01_1H
expt1 s2pu1

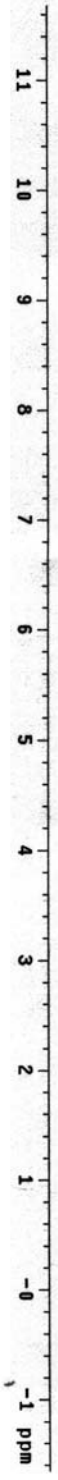
SAMPLE Jun 21 2006 dfrq DEC. & VT 589.942
solvent Jun 21 2006 dn H1
file CDC13 exp dpr 30
ACQUISITION exp dpr 30
sfrq 589.942 dn mm
tn H1 dmm C
at 1.892 dmf 200
np 30272 dseq 1.0
sw 8000.0 dres
fb 4000 homo
bs 58 dfrq2 DEC2
dpr 58
d1 0 dn2
d2 0 dn2
nt 16 dm2
ct 16 dmm2
atlock n dm2
gain not used dseq2 1.0
11 n dn2
in Y dfrq3 DEC3
dp n dn3
hs DISPLAY nm dpr3 1
sp -982.3 dof3 0
wp 7989.5 dms 0
vs 99 dms3 200
vc 250 dseq3
hzmm 0.52 dres3
15 18.72 homos 1.0
ffl 982.8 wfile PROCESSING
th 0 proc
ins 17 fn not used
nm 6.000 math

WERR
WEXP
WBS
WNT
WFT



(9)

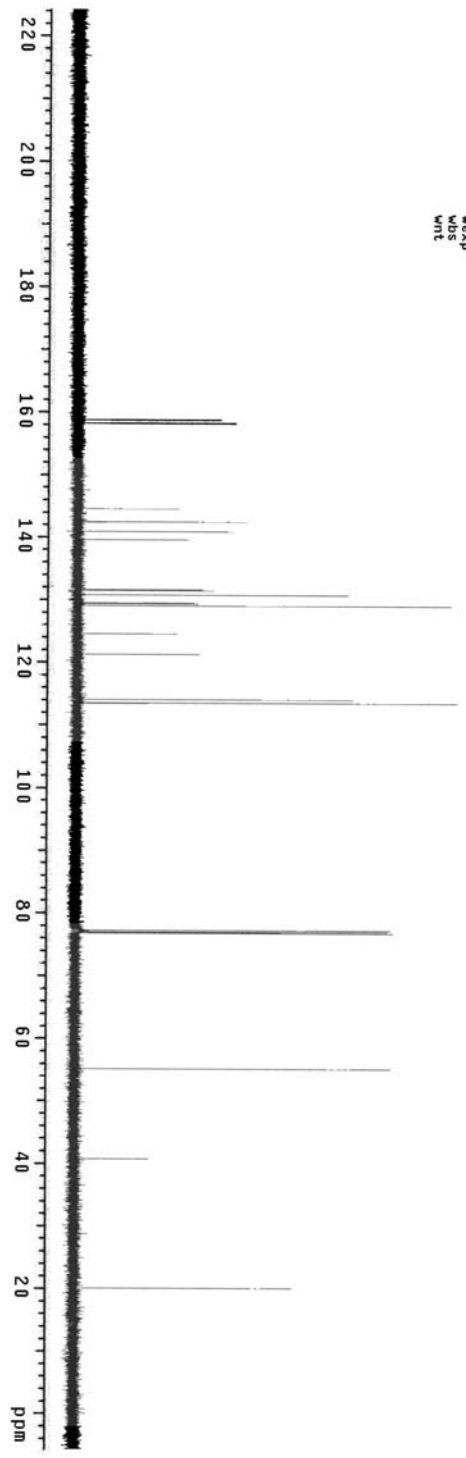
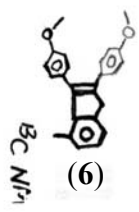
¹H NMR

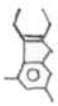


Dong_VIV_PA_G1_13C

exp2 s2pu1

date	SAMPLE	DEC. & VT	dfreq	599.942	H1
Jun 21 2006	CDC13	dm	30	0	
solvent	exp	dm	any	w	
11 ACQUISITION	150.871	dm	16667	1.0	
sfreq	C13	dmm			
tn	1.300	dmt			
at	50272	dseq			
np	3470.2	dres			
pw	19008	homo			
bs	61	dfreq2			
tpwr	5.0	dn2			
pl	3.000	dpwr2			
d1	3017.4	dot2			
nt	5000	dms2			
rt	752	dms2			
clock	not used	dres2			
gain	not used	dres2			
11	n	homo2			
in	n	DECS			
hs	y	dfreq3			
dp	mn	dms3			
sp	DISPLAY	dpwr3			
wp	-878.6	dm3			
vc	34708.6	dms3			
vs	65	dms3			
sc	0	dms3			
wc	250	dseq3			
h2mm	1.26	dres3			
rs	5000.00	dres3			
rfl	1290.0	homo3			
th	11615.8	homo3			
ins	8	proc			
nm	100.000	fn			
cdc	ph	math			
wt		not used			
Werr		ft			
Wexp		f			
Wts					
Wnt					



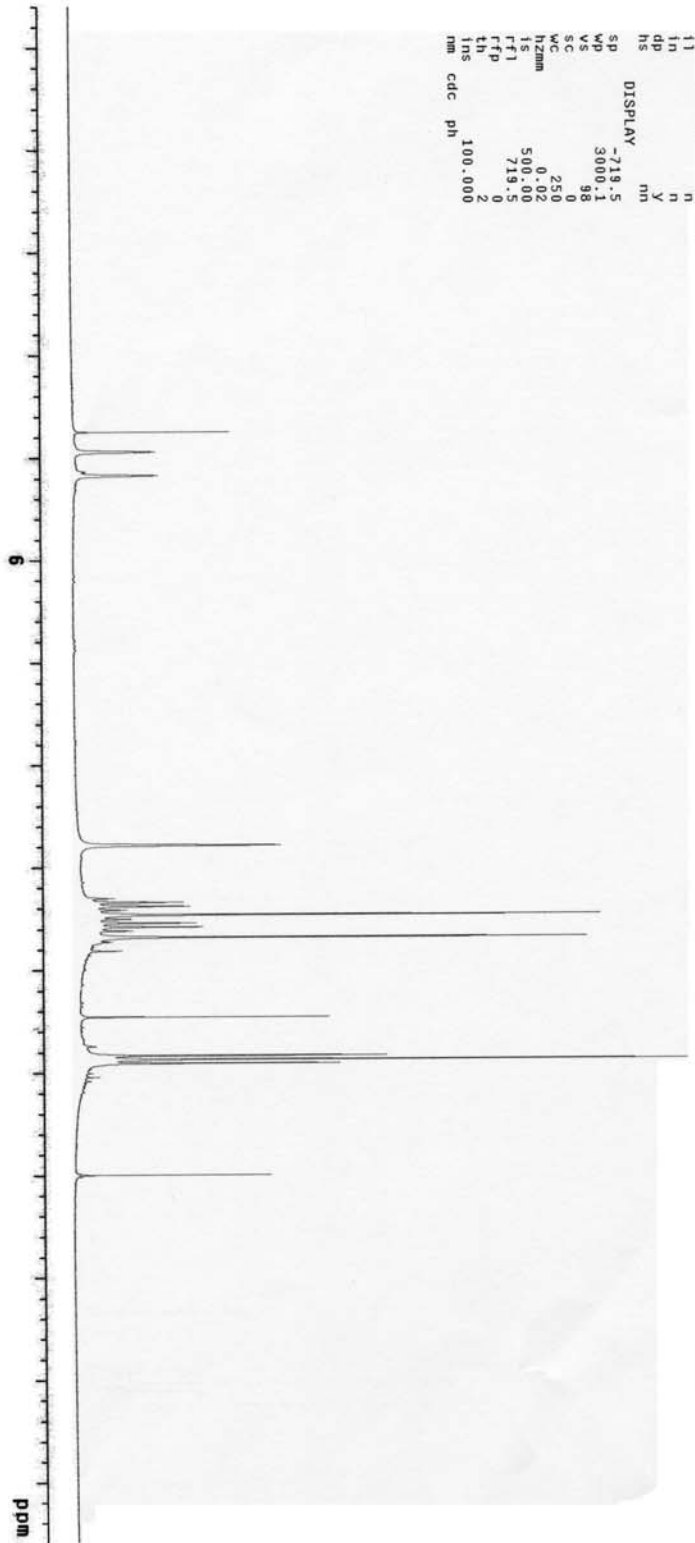


 ⑦

 1H NMR

```

Dong_1
expl std:
SAMPLE 2 2006
date Jun 12 2006
solvent CDCl3
FT ACQUISITION exp
sfrq 200.057
in H1
at 3.744 dm
np 22464 dseq
sw 3000.1 dres
fb 1600 homo
bs 4
tpwr 55
pw 7.0
d1 3.000
cp 64
ct 64
alock n
gain not used
flags not used
nt wnt
11 n
in n
dp y
hs nm
DISPLAY -719.5
sp
wp 3000.1
vs 98
sc 250
vc 0.02
h2mm 500.00
f1 719.5
rfp 0
th 2
nm cdc ph 100.000
  
```



D00ng_PIV_120_4H



¹H NMR

(8)

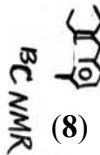
```

expt  szpul          SAMPLE          DEC. & VT          595.942
date   Jun 19 2006   CDC13          dn          H1
solvent                               exp          H1
file ACQUISITION 595.942   dm          30
sfreq 595.942      H1          mm
tn      1.892      H1          mm
at      30272     dsef          C
sw      8000.0    dres          200
fb      4000      homo          1.0
us      56        dfrq2     DEC2
dpwr    5.5      dn2
di      0         dpwr2     1
tof     0         dof2     1
nt      8         dm2      C
ct      8         dmm2     200
alock   n         dmf2     1.0
gain    not used dres2
flags   n         dn2      0
l1      n         dn3      0
in      y         dfrq3     DEC3
dp      nm          dn3
hs      DISPLAY   nm          dn3
sp      7981.2     dpwr3     1
wp      7981.0     dof3     1
vc      104       dmm3     C
sc      0         dmf3     200
wzmm    0.24      dres3     1.0
ls      102.67   homo3
rf1     595.942  wffile   PROCESSING
rfp     2         proc
tsh     2         fn
ins     1.000    math     not used
ms      cdc      ph

```



Dong_PIV_120_13C



```

exp2 s2pu1
SAMPLE
date Jun 21 2006 dfrq DEC. 2 VT 599.942
solvent CDC13 dn H1
file ACQUISITION exp dpwr 39
sfrq 150.871 dm nny 0
tn C13 dnm W
at 1.390 dmf 16667
np 1.222 dseq 1.0
sp 3470.72 dseq 1.0
fb 19000 homo
bs 8
tpwr 61 dfrq2 DEC2 0
pw 6.0 dn2
d1 3.000 dpwr2 1
tof 3017.4 dor2 0
nt 20000 dm2 n
dnt 9492 dm2 n
atlock gain not used 16667
gain FLAGS not used
11 n homo2 1.0
in n homo3
dp y dfrq3 DEC3 0
hs DISPLAY nm dn3
sp -872.3 dpwr3 1
wp 3470.6 dnt 1
vs 105 dms n
sc 0 dnm3 C
wc 250 dmf3 16667
h2am 31.89 dseq3
ts 500.00 dres3 1.0
fft 12488.6 homo3
tpp 11813.8 WtF18 PROCESSING
fns 8 Proc
ins 100.000 math not used
nm cdc ph
wert wexp
wbs
wnt

```

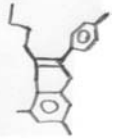


Domg_P1V_114_1H

expt s2p01

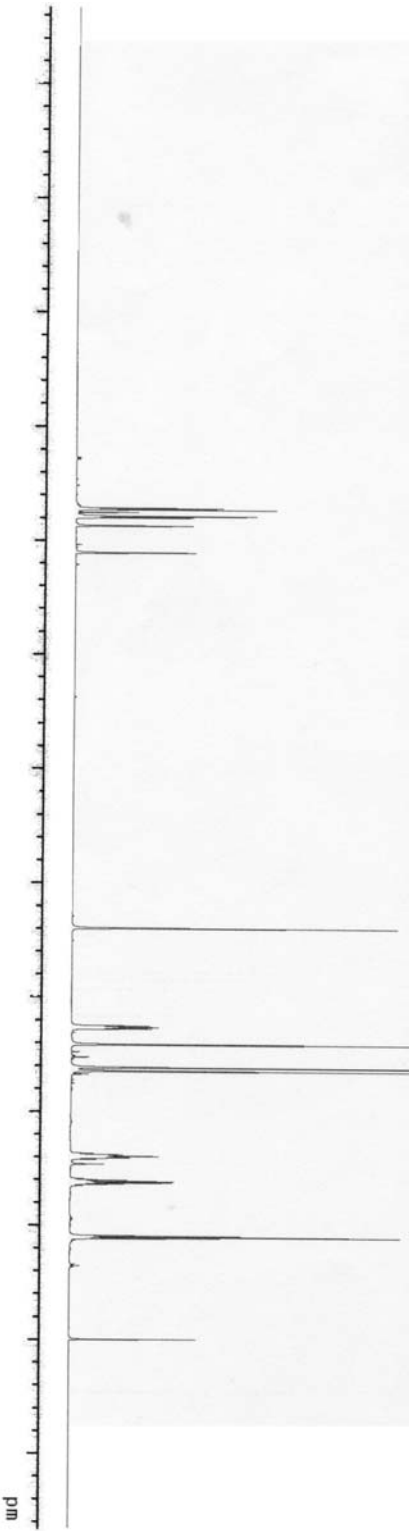
SAMPLE	date	Jun 8 2006	dfrc	DEC. & VT	599.942
solvent	CDCl3		dmf		91
file	ACQUISITION	exp	dpr		30
		dfrc	dm		0
frq	599.942		dm		mm
in	H1		dm		C
at	1.892		dse		200
np	30272		dres		1.0
sw	8000.0		homo		n
hb	4000		homo		n
ts	32		dfrc2	DEC2	0
tdpr	53		dm2		1
pw	3.000		dpr2		0
dl	0		dfc2		n
tof	8		dm2		C
nt	8		dm2		200
ct	n		dmf2		1.0
alock	not used		dres2		n
gain	not used		homo2	DEC3	0
ll	n		dfrc3		1
in	n		dmf3		n
ip	y		dmf3		n
hp	nm		dmf3		C
ns	DISPLAY	-998.4	dm3		200
sp	WP	7999.5	dm3		1.0
vs	SC	102	dmf3		n
wc	h2mm	32.250	dres3		n
h2mm	ts	223.51	homo3	PROCESSING	1.0
ts	ffl	998.9	homo3		n
ffl	proc	0	wfille		n
ftp	ft	0	proc		n
ins	ft	0	math		n
nm	cdc	2.000	math		n

WFT
WEXP
WBS
WNT



¹H NMR

(9)





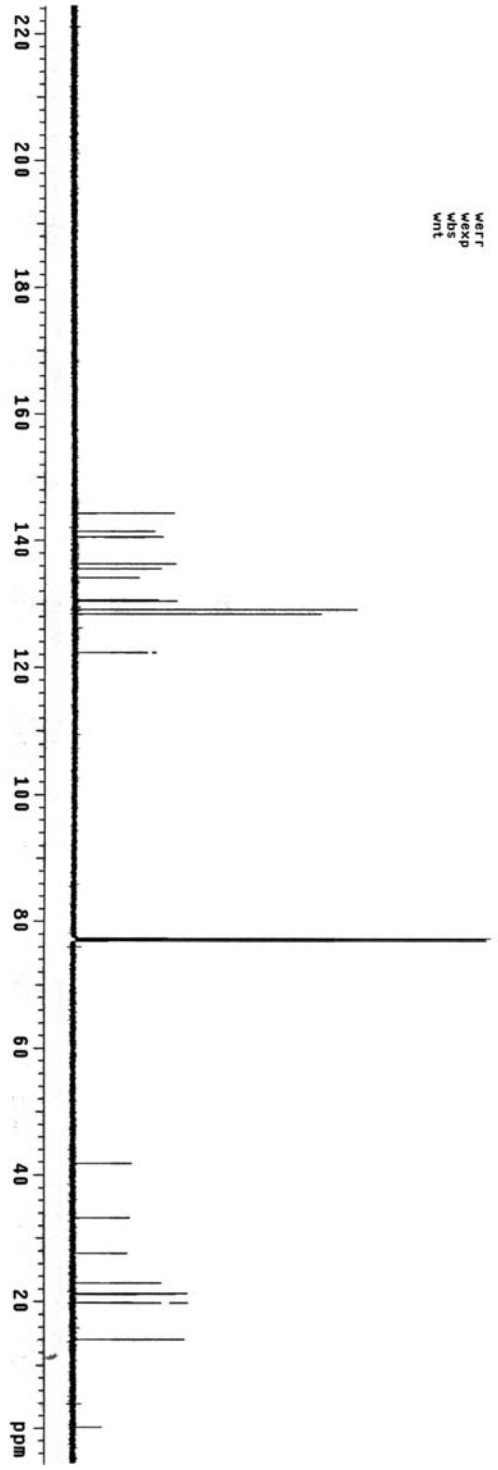
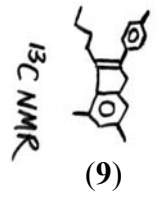
Dong_PIV_114_13C

exp2 s2pu1

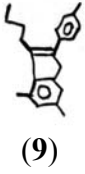
```

SAMPLE      Jun 8 2006      dfrq      DEC. & VT      599.942
solvent     Jun 8 2006      CDC13    dn          H1
f1file      ACQUISITION exp      dpuv       39
f2file      150.871      dot        14
tn          C13         dma        mvv        V
at          1.300      dmf        V
np          90272      dseq       16667
sw          34707.2   dres       1.0
fb          19000     homo
ds          81      dfrq2      DEC2       0
dpuv       6.0      dn2        0
d1          3.006     dpuv2      1
tof         3017.4    dot2       0
nt          30000     dma2       n
ct          8920     dma2       n
atlock     not used  dma2       C
gain       not used  dmf2       16667
          not used  dseq2      1.0
l1          n          dres2      n
in          n          homo2      n
dp          y          dfrq3      DEC3       0
hs          nm         dn3
          nm         dpuv3      1
          nm         dot3       0
          nm         dma3       n
          nm         dma3       C
          nm         dseq3      16667
          nm         dres3      1.0
          nm         homo3
          nm         proc
          nm         wfile
          nm         match
          nm         not used
          nm         ft
          nm         cdc
          nm         ph
          nm         weff
          nm         wexp
          nm         wbs
          nm         wnt

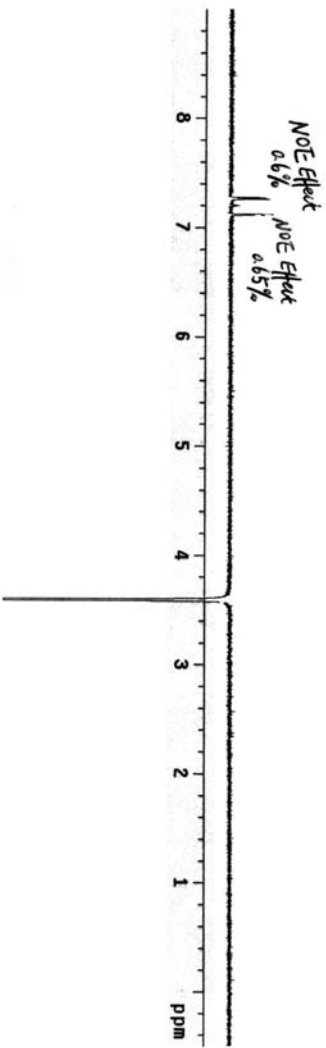
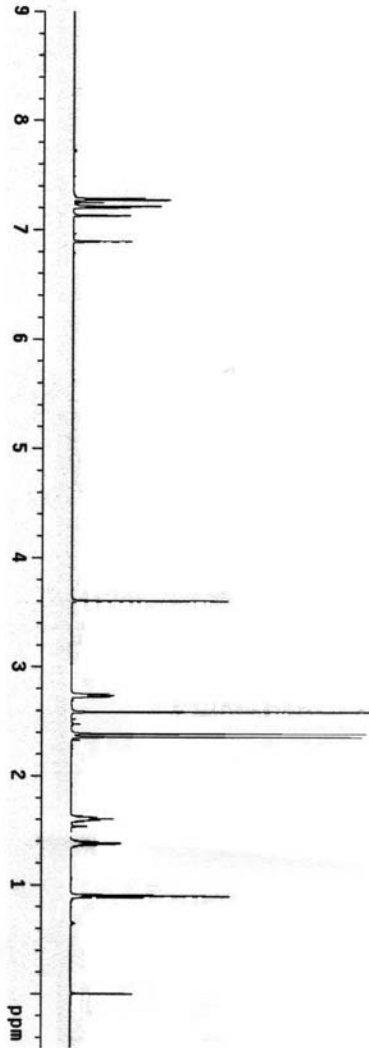
```



Dong_PIV_114_noesy1D
 Pulse Sequence: noesy1D
 Solvent: CDCl3
 Modulated Temperature
 INOVA-500 40000
 Relax delay 2.000 sec
 Pulse program
 Mixing 0.400 sec
 Acq. time 1.892 sec
 Width 8000.0 Hz
 180 repetitions
 OBSERVE N1 599.9393058 MHz
 F1 A1 500.1362928 MHz
 Total time 29 min, 25 sec



NOE Experiment

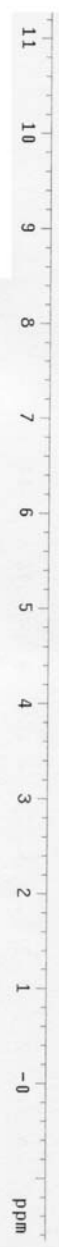


Ph C1=CC=C(C=C1)C2=CC=CC=C2
 (10)

¹H NMR

```

Donq_VIV_P109_1H
expt1 szpu1
SAMPLE
date Oct 3 2006 DEC. & VT
solvent CDC13 d1 d1 599.942
file exp dpwr 30
ACQUISITION 599.942 dm 0
sfrq 1.843 dm 200
tn 30272 dseg 1.0
mp 8000.0 dres 1.0
sw 4000 homo
bs 4 dfrq2 DEC2 0
tpwr 59 dfrq2
pw 6.0 d12 1
tof 3.000 dpwr2 1
nt 8 d12 1
ct 8 ddm2 n
atlock n dmf2 C
gain not used dseg2 200
flags n dres2 1.0
11 n homo2 DEC3 0
12 n dfrq3
13 n dn3
14 n dn3
15 n dn3
DISPLAY
sp -998.1 dof3 1
wp 7999.5 dm3 0
vc 119 ddm3 n
sc 220 dmf3 C
bzmm 30.00 dres3 200
ls 181.91 homo3 1.0
PROCESSING
ffl 0 wffile
fth 3 fproc
ins cdc ph 3.000 math not used
wfft wfft
wexp wexp
wbs wbs
wnt wnt
  
```





(10)

¹³C NMR

```

Dong_VIV_P109_13C
exp2 s2pu1
SAMPLE      date Oct 3 2008      dfrc DEC. & VT 599.942
solvent     Oct 3 2008      dn   H1
file        CDC13      exp   dpwr 39
ACQUISITION 150.871      dm   dof 0
sfrc        150.871      dm   dof 0
tp          1.300      dm   dof 0
nd          90272      dseg dres 16667
sw          34707.2      dres dres 1.0
fb          19000      homo 1.0
bs          8          dfrc2 DEC2 0
tpwr        61         dfrc2 dn2 0
pw          3.000      dpwr2 dn2 1
df          3017.4      dot2 dn2 0
ct          2978      dms2 dn2 0
ct          2978      dms2 dn2 0
atlock      not used   dseg2 dn2 16667
gain        not used   dres2 dn2 1.0
l1          n          homo2 dn2 0
ln          n          dfrc3 DEC3 0
dp          y          dfrc3 dn3 0
hs          n          dn3 0
DISPLAY     nm          dn3 0
sp          -872.5      dpc3 dn3 1
vs          34706.5      dpc3 dn3 0
vc          120         dms3 dn3 0
sc          0          dms3 dn3 0
wzmm        250         dseg3 dn3 16667
hzm         0.79        dres3 dn3 1.0
ls          500.00      homo3 dn3 1.0
rf1         12488.9      homo3 dn3 1.0
rf2         11615.9      1b   PROCESSING 0.50
rf3         100.000     wfile
nm          100.000     wfile
cdc         math       not used
ph          math       not used
werr       werr
wexp       wexp
wbs        wbs
wnt        wnt
  
```

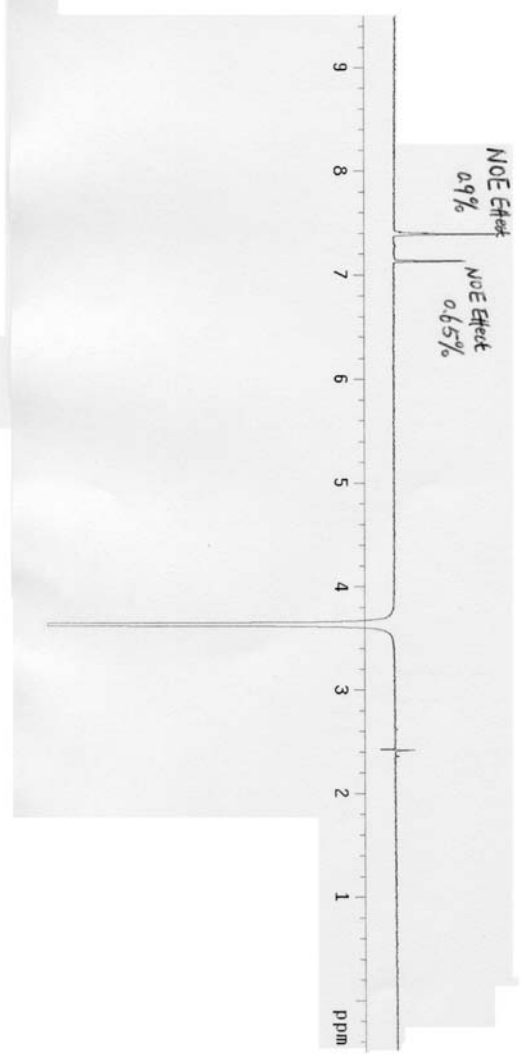
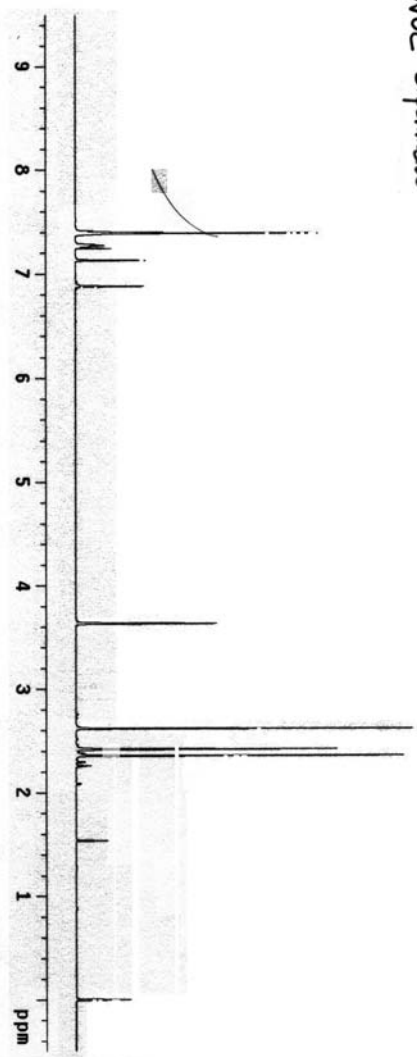


Dong_VIV_P109_noesy1D
 Pulse Sequence: noesy1D_da
 Solvent: CDCl3
 Ambient temperature
 INOVA-600 "nu600"
 Relax. delay 2.000 sec
 Pulse 90.0 degrees
 Mixing 0.400 sec
 Width 1.332 sec
 Width 8000.0 Hz
 248 Repetitions
 OBSERVE HI, 599.8393022 MHz
 DATA PROCESSING
 Line broadening 0.7 Hz
 FT size 32768
 Total time 1 hr, 19 min, 0 sec



(10)

NOE Experiment



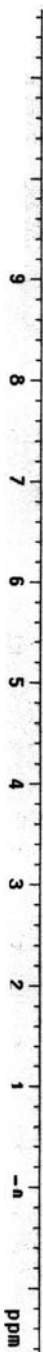
Dong_VIV_P105.

exp1 s2pu1



¹H NMR

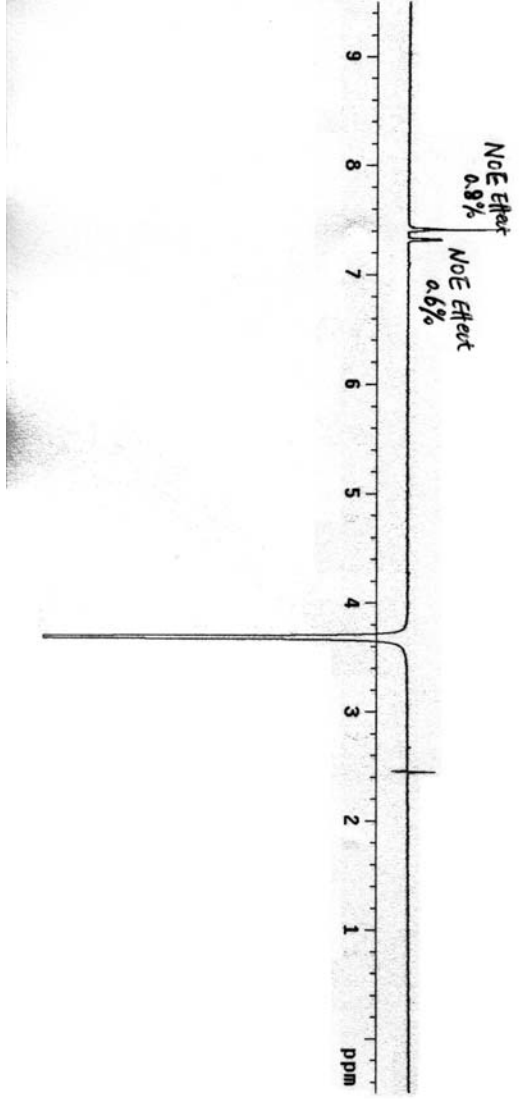
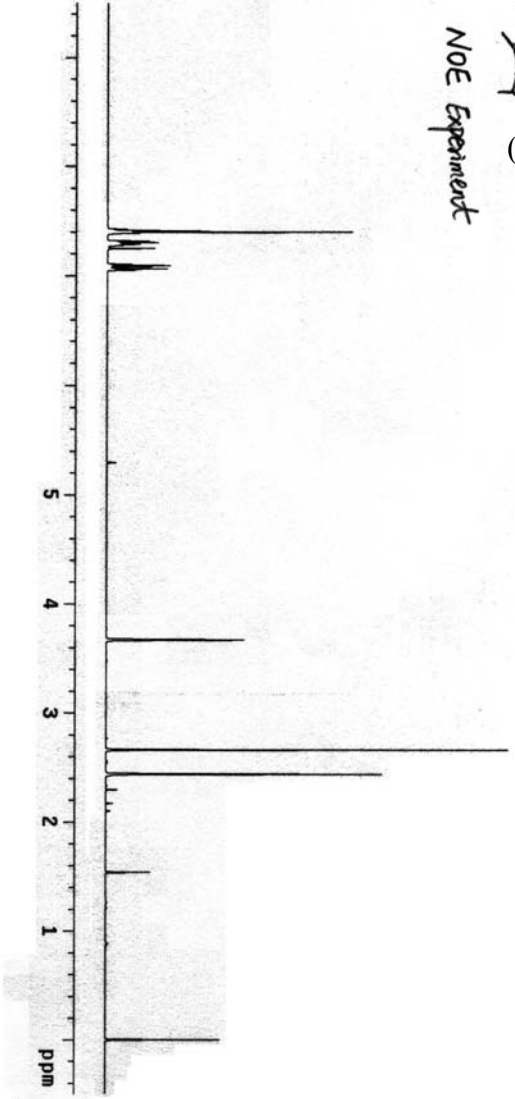
date	SAMPLE	DEC. & VT
Oct 3 2006	3.2006	599.942
solvent	CDC13	H1
file	exp	30
sfreq	599.942	mm
at	1.892	mm
np	30272	dseq
sw	8000.0	dres
fb	4000	homo
bs	4	DEC2
tpwr	59	dfrq2
pw	6.0	dn2
d1	3.000	dpwr2
tof	0	dof2
nt	8	dm2
st	8	dmf2
atlock	not used	dseq2
gain	not used	dres2
fl	n	homo2
in	y	dfrq3
dp	n	dn3
hs	n	dpwr3
sp	-994.3	dof3
wp	799.5	dm3
vs	95	dmf3
sc	220	dfrq3
hsc	0.49	dres3
hscnm	181.91	homo3
rfi	994.8	PROCESsing
th	0	vtfile
ins	5	proc
nm	3.000	fn
		math
		not used
		f
		ft
		werr
		wexp
		wds
		wnt
		wft



Dong_VIV_P105_noesy1D
 Pulse Sequence: noesy1D_1
 Solvent: CDCl3
 Ambient Temperature
 INOVA-600 400000
 Relax. delay 2.000 sec
 Pulse 90.0 degrees
 Mixing time 1.000 sec
 Acq time 1.038 sec
 Width 8000.0 Hz
 440 repetitions
 OBSERVE H1, 599.3392935
 DATA PROCESSING
 Line broadening 0.7 Hz
 FT size 32768
 Total time 1 hr. 20



NOE Experiment



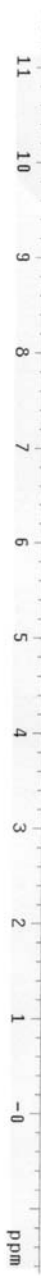
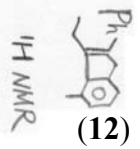
Doong_VIV_p112_1H

expt s2pu1

SAMPLE	date	DEC. & VI
6.2006	dfreq	599.942
CDC13	dn	H1
exp	dpwr	3.0
df	dozf	0
599.942	dm	mm
H1	damm	C
1.832	dmt	200
30272	dsesq	1.0
80000	drss	n
4000	homo	n
59	dfreq2	0
6.0	dn2	1
3.000	dpwr2	0
0	dozf2	n
8	dms2	n
8	dms2	200
0	dms2	C
0	dsesq2	1.0
not used	drss2	n
homo2	homo2	1.0
DEC3	DEC3	0

DISPLAY

SP	WP	VS	SC	WC	hazm	fz1	fz2	fn	proc	math	WARR	WOSP	Wnt	Wft
1004.2	799.5	119	30	220	181.31	1004.0	0	fn	not used	f				
nm	cdc	ph												




exp2 s2pu1

```

SAMPLE      DEC. & VI
date Oct 6 2006  dfrq 599.942
solvent Oct CDC13  dn   H1
file ACQUISITION exp dpwr 39
          exp dof   0
sfreq 150.871  dm   mny
          130.871  dm   mny
          111.871  dm   mny
at 1.300  dmf 16667
np 90272  dseq
sw 34707.2  dres 1.0
fb 19000  homo
bs 8  DECD
cpwr 8  dfrq2  DECD
          61  dfrq2  0
          3.000  dpwr2  1
          3017.4  dof2  0
nt 10000  dma2  n
          2752  dmm2  c
atlock not used  dmf2 16667
gain not used  dseq2 1.0
          n  dres2
          n  homo2
          n  DECD
          n  dn3
          Y  dfrq3  0
          n  dn3
          n  dpwr3  1
          n  dof3  0
          n  dma3  n
          n  dmm3  c
          n  dres3  16667
          76  dm3
          250  dseq3
          2.28  dres3  1.0
          500.00  homo3
          12498.7 1b  PROCESSING 0.50
          11615.8 1b  wffile
          10  wffile
          100.000  f1  not used
          mm  cdc  ph  math
          werr
          wexp
          wbs
          wnt

```

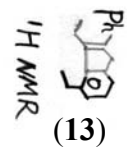


(12)

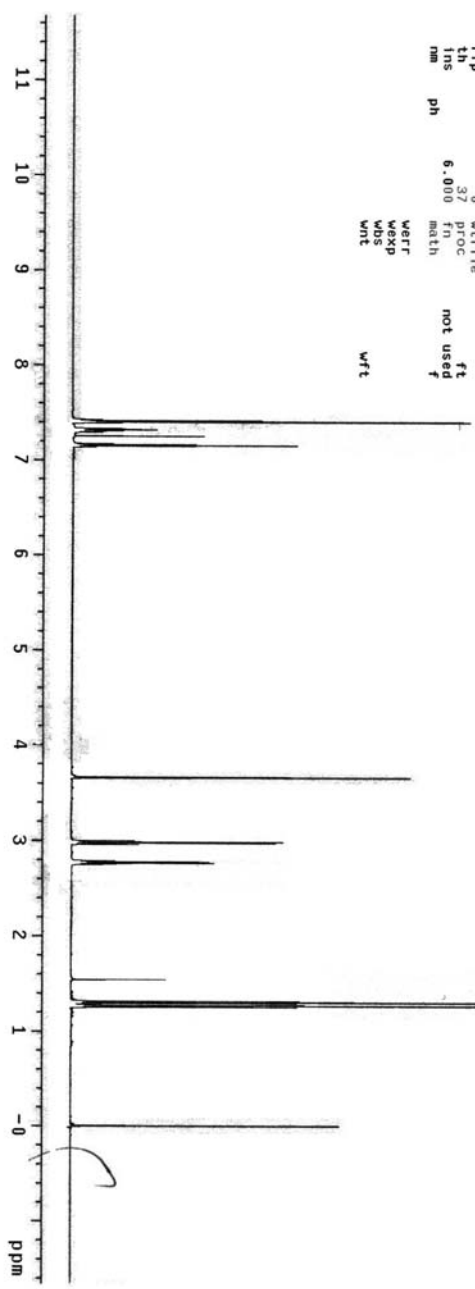
 13C NMR



exp1 s2pul



exp1 s2pul
 SAMPLE
 date Oct 20 2006
 solvent CDCl3
 F1 ACQUISITION exp
 sfrq 599.942
 ln H1
 at 1.892
 np 30272
 sw 8000.0
 ds 4000
 tpwr 59
 pw 6.0
 dl 3.000
 lof 0
 nt 8
 ct 8
 clock n
 gain not used
 flags n
 in n
 dp Y
 hs n
 DISPLAY
 sp -989.7
 wp 7999.5
 vs 94
 sc 30
 wc 220
 hzmm 0.07
 ls 227.39
 rfd 394.1
 th 37
 ins 6.000
 nm math
 ph not used
 WFT
 WEXP
 WMT
 WFT



Dong_VIV_P121_02_13C

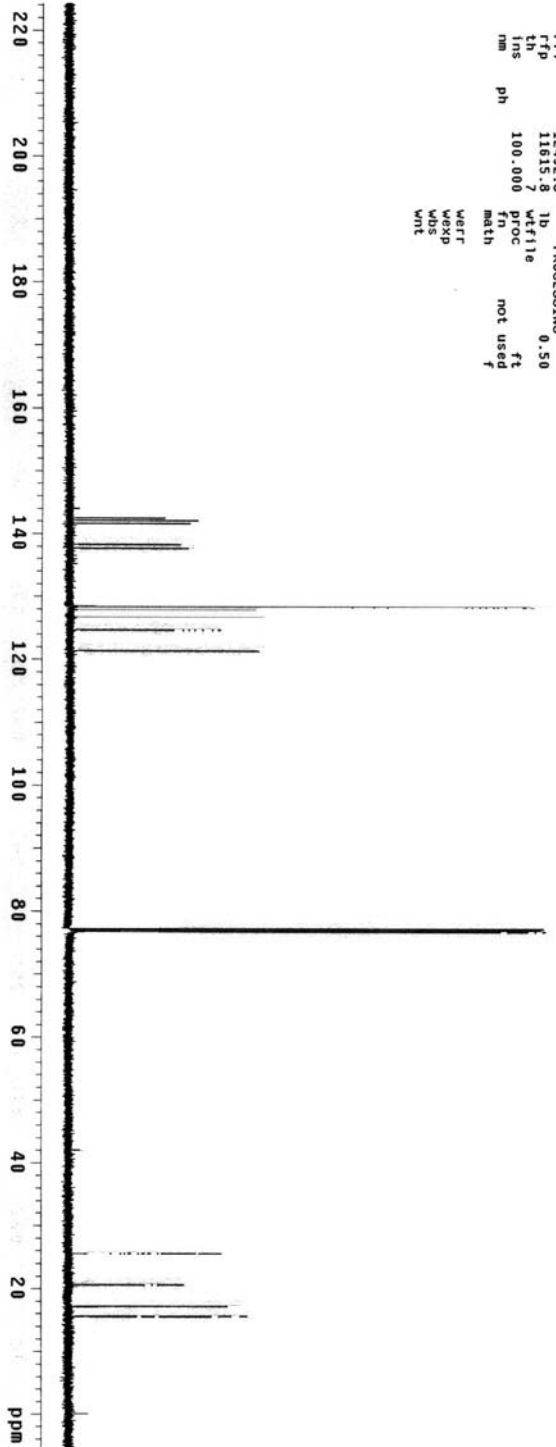
exp2 s2pu1

date	Oct 20 2006	dfreq	DEC. & VI	599.942	H1
solvent	CDC13	dn			39
file	ACQUISITION	exp	dpwr		16667
sfreq	150.871	dd	nmv		1.0
tn	1.300	dmm			
at	90272	dseq			
sw	34707.2	dres			
fb	19000	homo			
dsprf	6	dfreq2	DEC2	0	
dpwr	6	dn2			
dl	3.000	dpwr2		1	
tof	3017.4	dotf2		0	
nt	5000	dmm2		n	
ct	912	dmm2		C	
alock	not used	dseq2		16667	
gain	not used	dres2			
l1	Y	homo2	DEC3	1.0	
in	n	dfreq3		0	
dp	Y	dn3			
hs	n	dpwr3		1	
sp	DISP	dotf3		0	
wp	-876.5	dmm3		n	
v1	34706.5	dseq3		16667	
sc	0	dres3			
wc	250	homo3		1.0	
hzmm	34.35	PROCES			
rs	500.00	math			
rft1	12492.8	math			
rft2	11615.9	math			
rft3	100.000	math			
ins		math			
nm		math			

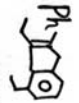


¹³C NMR

(13)

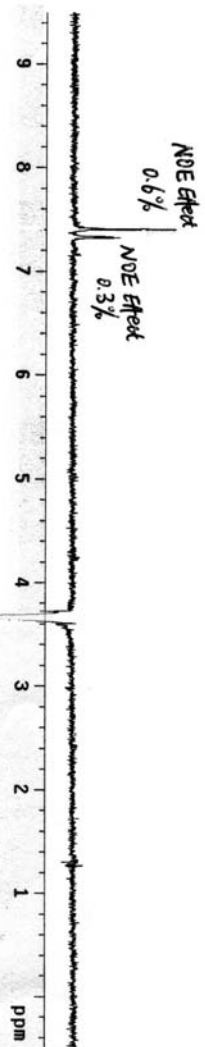
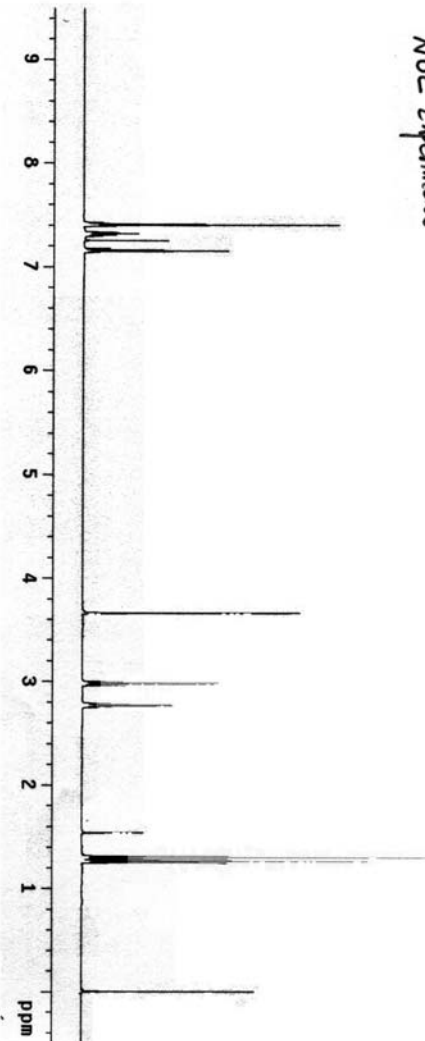


Donq_VIV_P121_G2_noesy10
 Pulse Sequence: noesy10_da
 Solvent: CDCl3
 Ambient temperature
 INOVA-600 "u600"
 Relax--delay 2.000 sec
 Pulse 90.0 degrees
 Mixing 0.400 sec
 Acq. time 1.892 sec
 F1den 6000.0 MHz
 F2den 6000.0 MHz
 OBSERVE H1 599.9392965 MHz
 DATA PROCESSING
 Line broadening 0.7 Hz
 FT size 32768
 Total time 1 hr, 28 min, 11 sec

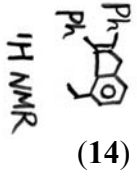


(13)

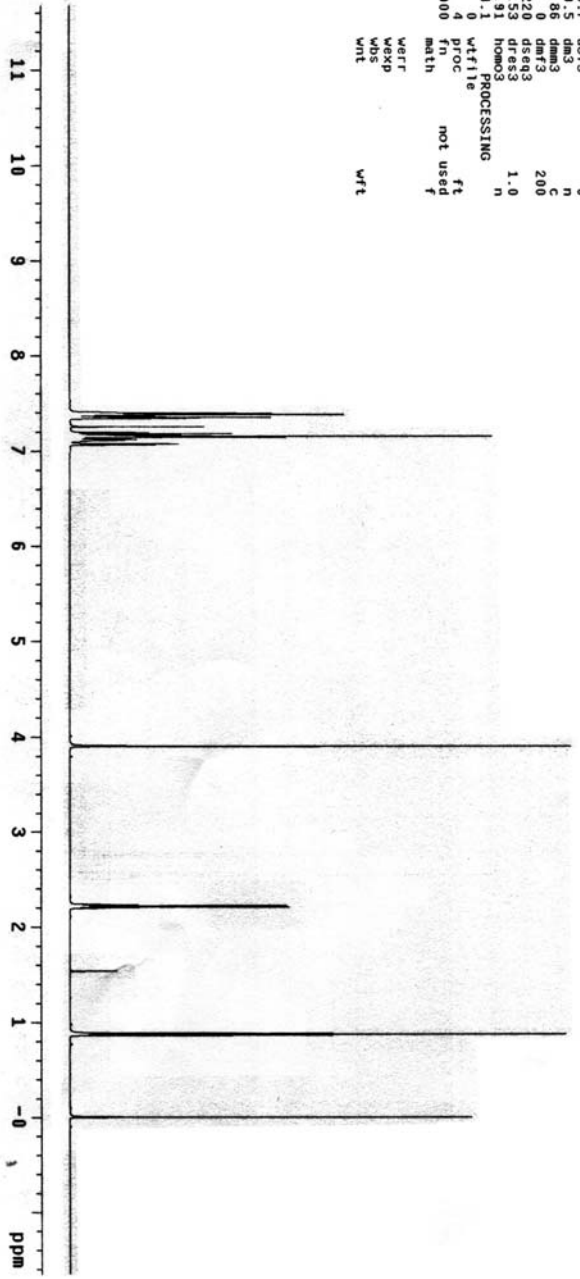
NOE Experiment



Dong_VIV_P117_G2_1H



```
expl s2pau1
SAMPLE DEC. & VI
date Oct13 2006 dfrq 599.942
solvent CDC13 dn H1
file ACQUISITION exp dpwr 30
sfreq 599.942 dof 30
in H1 dnm mnc
at 1.892 dmf 200
np 30272 dseq
sw 8000.0 dres 1.0
fb 4000 homo
dspr 5 4 dfrq2 DEC2 0
nu 6 0 dn2
di 3.000 dpwr2 1
tof 0 dof2 0
nt 8 dnm2 n
ct 8 dnm2 n
alock not used n dmf2 200
gain FLAGS n dseq2 1.0
l1 n homo2 DEC3 0
in Y dfrq3
dp nm dn3
hs DISPLAY
sp -983.7 dpwr3 1
wp 7993.3 dof3 0
vc 0 dnm3 n
sc 0 dmf3 200
wcc 220 dseq3
h2mm 181.91 homo3 PROCESSING 1.0
f1 394.1
f1p 0 wfile
f1m 0 pfile
f1s 3.000 math not used f
rm cdc ph
```



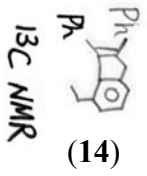
Dong_VIV_p117_02_130

exp2 s2pu1

```

SAMPLE DEC. & VT
date Oct 6 2006 dfreq 599.942
solvent CDC13 dn H1
file exp dpwr H1
ACQUISITION dof 39
sfrq 150.871 dm nmy
fn C13 dnm nmy
nc 1400 dnm 16667
sw 50272 dres 1.0
sb 34707.2 dres 1.0
fb 19000 homo n
bs 8
tpwr 61 dfreq2 DEC2 0
pw 6.0 dn2
d1 3.000 dpwr2 1
lof 3017.4 dof2 1
nt 10000 dm2 n
t1 019 dmf2 c
clock not used dres2 16667
gain not used dres2 1.0
ll n homo2 n
in y dfreq3 DEC3 0
dp n dn3
hs DISPLAY nm dn3 1
SP 7888.5 dpwr3 1
VP 31706.8 dof3 0
WC 70 dm3 n
SC 0 dmf3 c
WC 250 dres3 16667
hzm 138.83 dres3 1.0
IS 500.00 homo3
rf1 12504.9 lb PROCESSING 0.50
rfp 11615.8 wfl1e
th 10 p1oc not used f
ins 10 m1n
nm cdc ph math

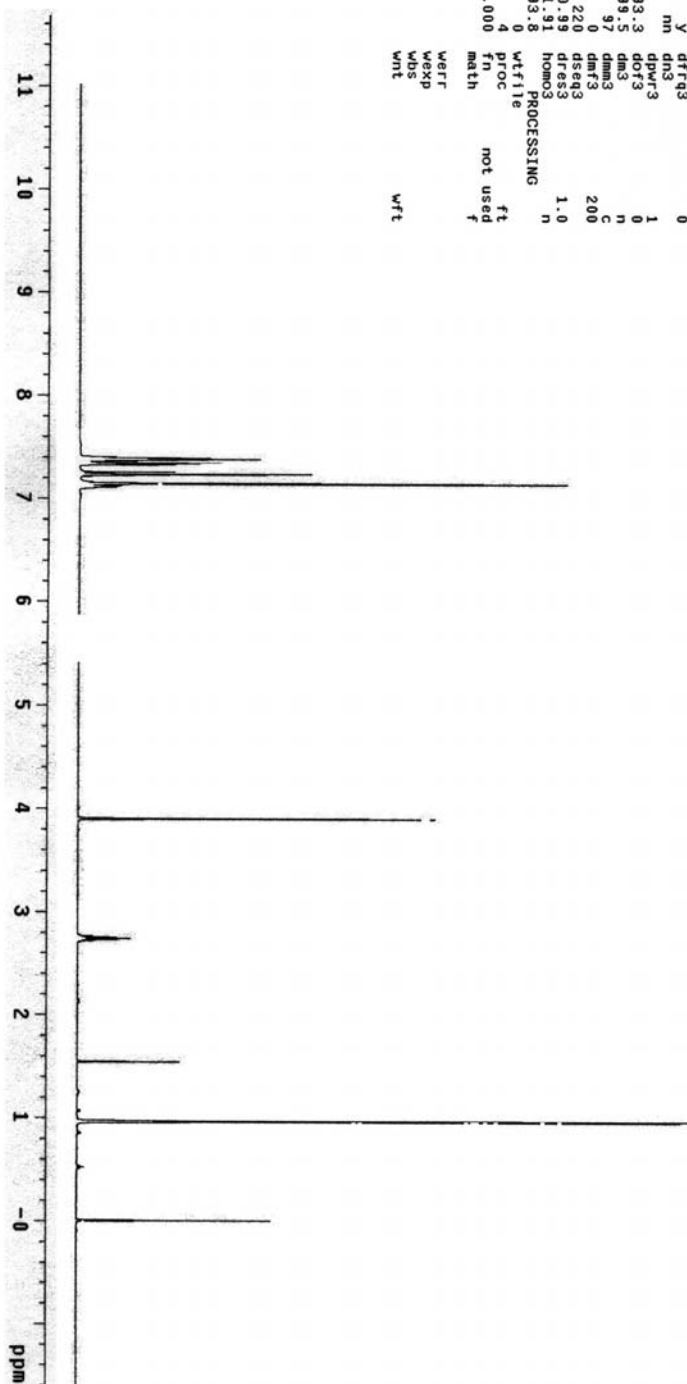
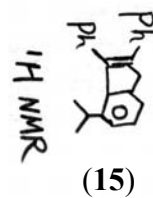
```



Dong_VIV_p116_g2_1H

expl s2pnt1

SAMPLE	DEC. & VT
date Oct 6 2006	dfrq 599.942
solvent CDCl3	dn HI
file ACQUISITION	exp dprvr 30
sfreq 599.942	dot 0
tn HI	dmm mnm
at 1.892	dntf 200
np 30272	dresq 200
sw 8000.0	dres 1.0
fb 4000	homo n
bs 4	DEC2 0
tpvr 59	dfrq2 0
pw 6.0	dn2 0
d1 3.000	dpvr2 1
tof 0	dot2 0
nt 8	dm2 n
ct 8	dmm2 C
atlock n	dmt2 200
gain not used	dresq2 1.0
flags n	dres2 n
l1 n	homo2 n
in n	dfrq3 DEC3 0
dp y	dn3 n
hs nm	dprvr3 1
sp DISPLAY -933.3	dot3 0
wp 7999.5	dms3 n
vs 97	dmm3 C
sc 0	dmt3 200
wc 220	dresq3 1.0
h2mm 0.99	dres3 n
rs 181.91	homo3 n
rfl 993.8	wtfile PROCESSING
fn 0	proc not used
tn 4	math n
ins cdc ph 2.000	verf wft
	weyp
	wbs
	wnt

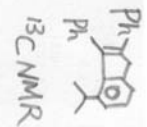


exp2 s2pu1

```

SAMPLE      DEC. & VT
date Oct 6 2006  dfrq 599.942
solvent CDCl3   dn   H1
file ACQUISITION exp  dpr 39
           150.871  dm  31
           C13     dm  16687
           at 1.300  dmf  W
           sw 90272  dseq 16687
           fb 34707.2 dres 1.0
           ds 19000  homo 1.0
           dpwr 8    DEQ2
           dp 61    dfrq2 0
           dt 3.00  dm2r2 1
           tof 3017.4 dof2 0
           nt 20000  dm2  n
           ct 568    dma2  C
           atlock n  dmf2  16687
           gain not used dseq2
           l1 n     dres2  1.0
           ln n     homo2  n
           ns Y     dfrq3  0
           dp n     dm3r3  1
           DISPLAY -882.9 dof3 0
           wp 34706.6 dm3  n
           vs 76    dma3  C
           sc 0     dmf3  16687
           wc 250   dseq3  1.0
           hzmm 138.83 dres3  n
           rs 500.00 homo3  1.0
           rft 12499.3 lb  PROCESSING
           rfp 1819.8  lf  0.50
           th 100.000  wf file
           rms cdc ph  math  not used
           werr
           wexp
           wds
           wnt

```



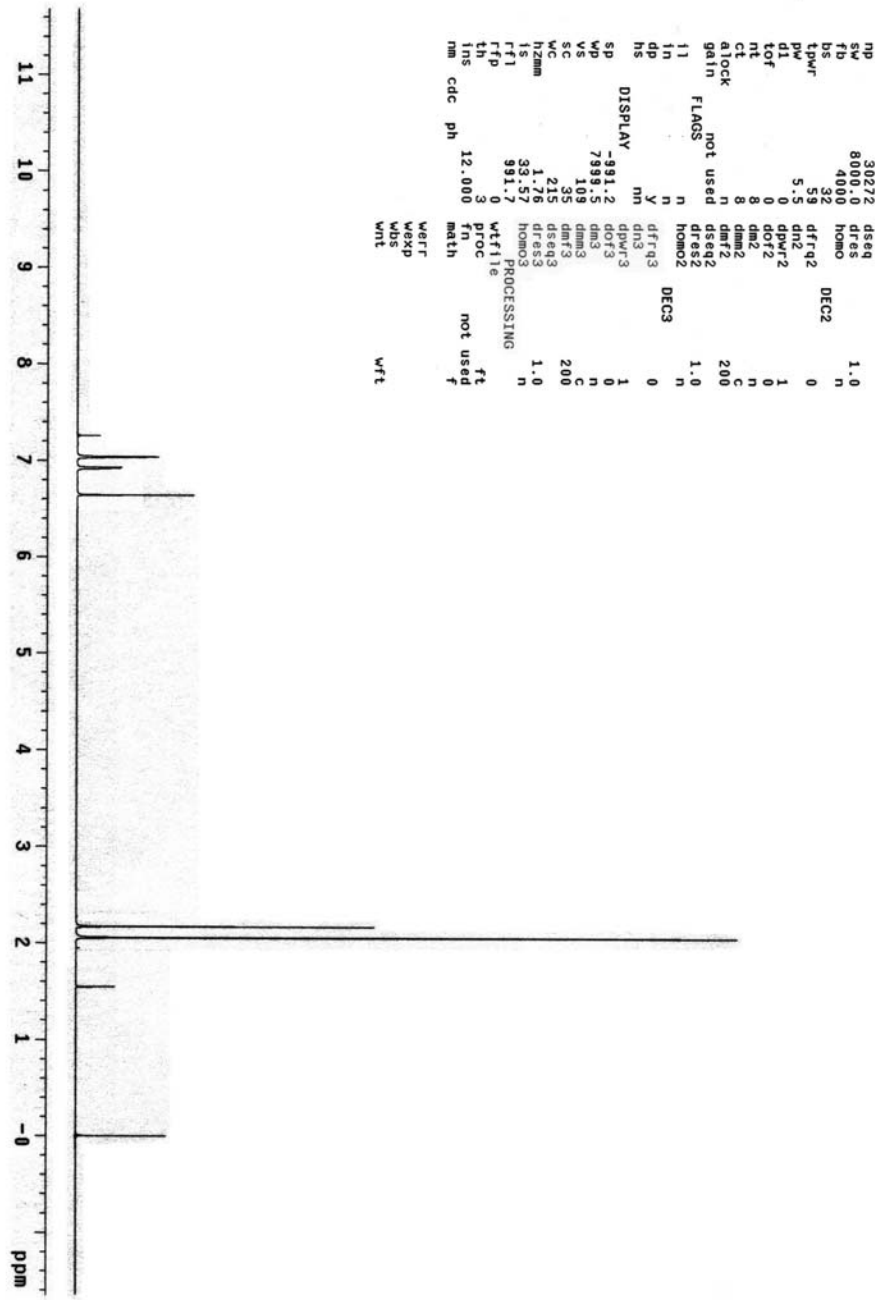
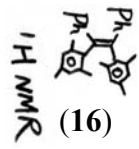
(15)



D0ng_PIV_121_1H

exp1 s2pul1

date	Jun 30 2006	DEC. & VT	599.942
solvent	CDCl3	dn	M1
file	exp	dpwr	30
str	599.942	dm	0
acq	1.891	dmr	200
np	30272	dres	1.0
sw	8000.0	homo	n
fb	4000	homo	n
bs	32	DEC2	0
tpwr	59	dftrq2	0
pw	5.5	dn2	1
dl	0	qpwr2	0
tof	0	doz2	n
td	8	dmz2	n
cl	8	dmz2	n
atlock	n	dmsq2	200
gain	not used	dres2	1.0
11	n	homo2	n
in	n	homo2	n
dp	y	dftrq3	DEC3
hs	nn	dn3	0
sp	DISPLAY-981.2	qpwr3	1
vd	7993.2	doz3	0
ve	109	dmz3	0
sc	35	dmr3	200
wc	215	dres3	1.0
hzmm	1.76	dres3	1.0
rs	33.57	homo3	n
rftl	991.7	homo3	n
lfs	0	wtfile	PROCESSING
lfs	0	proc	not used
lfs	3	match	f
nm	12.000	match	f
nm	cdc	ph	12.000
nm	cdc	ph	12.000
werr		werr	
wexp		wexp	
wbs		wbs	
wnt		wnt	
wft		wft	



Dong_PIV_121_13C

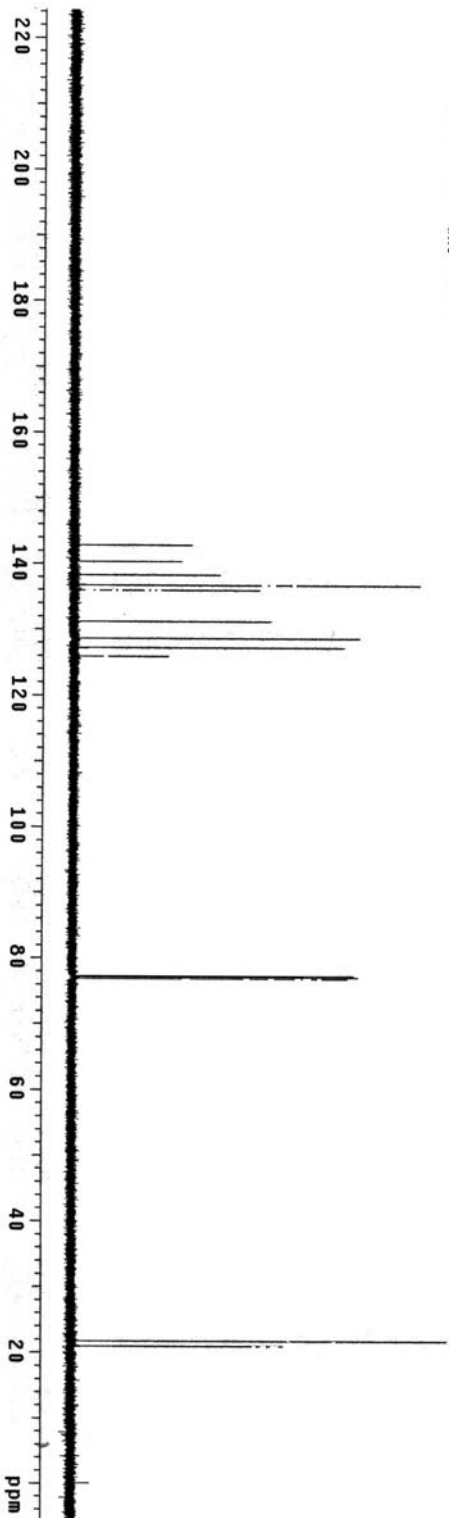
exp2 szpul1

date	Jun 30 2006	dfrq	DEC. & VT	539.942
solvent	CDCl3	dn	H1	39
file	exp	dpwr	0	nny
strq	150.871	dof	0	w
ACQUISITION		dm		
tn	1.523	dmm		16667
te	8.029	dms		1.0
nu	34707.2	dfe4		1.0
sw	19000	dfe8		n
fb		homo		n
bs	8	DEC2		0
tpwr	61	dfrq2		0
pl	6.6	dn2		1
d1	3.000	dpwr2		0
tof	3817.4	dof2		0
nt	10000	dm2		n
cl	328	dmm2		c
clock	not used	dms2		16667
gain	n	dfe2		1.0
11	n	dfe42		1.0
in	n	dfe82		n
dp	y	homo2	DEC3	0
hs	nn	dn3		1
DISPLAY	-874.6	dpwr3		0
sp	34706.6	dof3		n
wp	67	dms3		c
vs	0	dmm3		16667
sc	251	dfe3		1.0
hc	138.83	dfe43		1.0
h2mm	500.00	dfe83		n
15	12491.0	homo3	PROCESSING	n
rf1	11615.8	wtfile		ft
th	9	proc	not used	f
1ns	100.000	fn		
nm	cdc	math		
ph		weff		
		wekp		
		nds		
		mts		



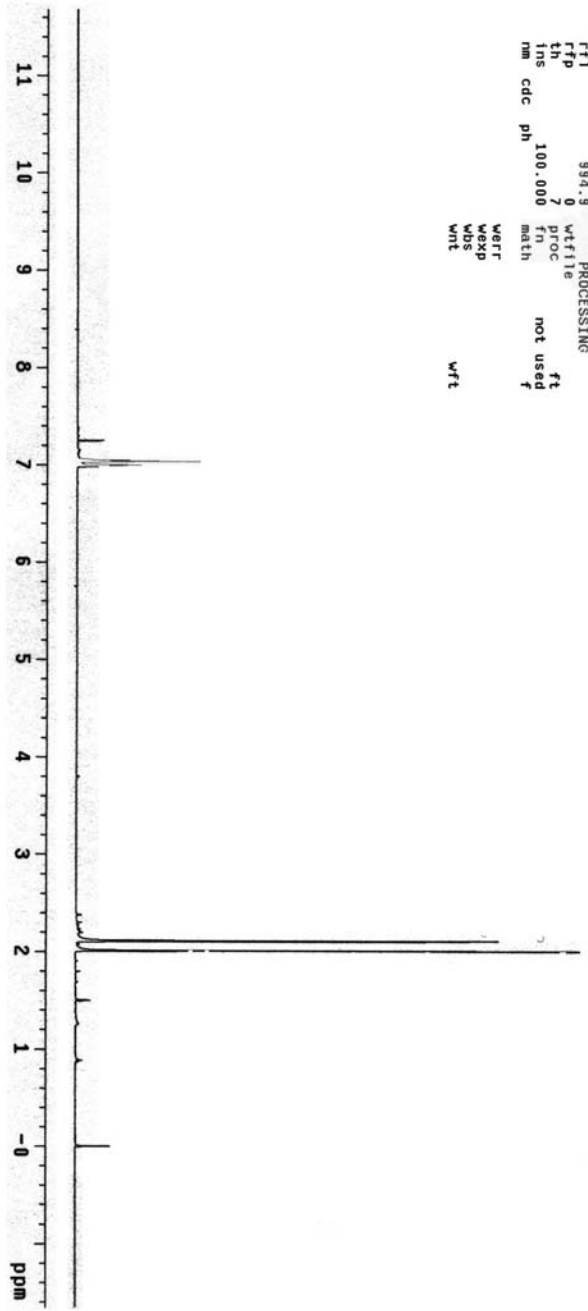
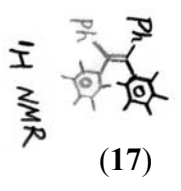
(16)

¹³C NMR



Dong_VIII_P157_G1_1H
 expt szput

SAMPLE DEC. & VT
 date Apr 19 2008 dfrq 599.942
 solvent CDC13 dn H1
 file ACQUISITION exp dpar 30
 sfrq 599.942 dm mnc
 tn HI dmm 200
 at 1.892 dmf
 np 30272 dseq
 sw 8000.0 dres
 fb 4000 homo 1.0
 bs 32
 tpar 59 dfrq2 DEC2
 dy 5.9 dm2 0
 dv 0 ddf2 1
 tof 0 dm2 1
 nt 8 dmm2 n
 ct 8 dmm2 n
 alock n dmf2 200
 gain not used dseq2
 flags n dres2 1.0
 11 n homo2 n
 in n dfrq3 DEC3
 dp y dm3 0
 ds DISPLAY nm dms 1
 sp -994.4 ddf3 1
 wp 7999.5 dms 1
 vs 83 dmm3 n
 sc 35 dmf3 200
 wc 215 dseq3
 hzmm 37.16 dres3 1.0
 ts 33.57 homo3
 ffl 994.9 wffile PROCESSING
 tpr 9 pproc
 tns cdc ph 100.000 math not used
 ft
 warr
 wexp
 wbs
 wnt wft



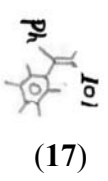
Dong_VIII_P157_G1_13C

exp2 s2pu1

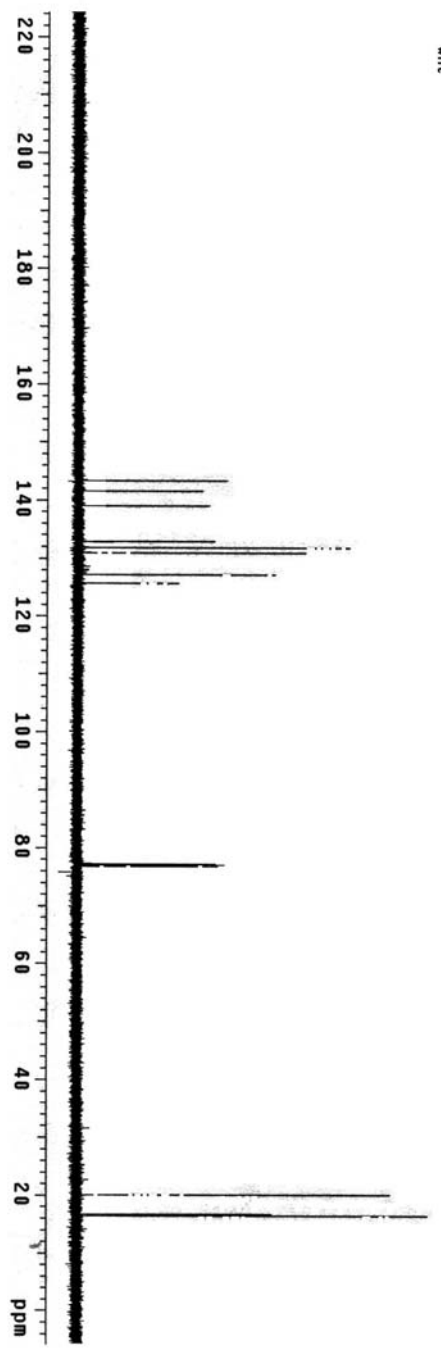
```

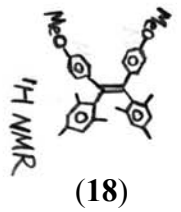
SAMPLE DEC. & VT
date Apr 18 2006 dfrq 539.942
solvent CDCl3 dn H1
file ACQUISITION exp dpr 39
sfreq 150.871 dm dot 0
in C13 dnm mny W
at 1.300 dmf 15877
np 30272 dseq 1.0
fh 34707.2 dres 1.0
bs 1300.8 homo 25.0
tpwr 61 temp
pw 6.0 dfrq2 DEC2 0
d1 3.000 dn2
lof 3017.4 dpwr2 1
nt 1000 dot2 0
ct 256 dm2 n
atlock n dm2 n
gain not used 1667 C
11 n dseq2 1.0
1n n dres2
1n n homo2 DEC3 0
hs DISPLAY mn dfrq3 0
sp -875.7 dpwr3 1
wp 34706.6 dot3 0
vs 57 dm3 n
sc 0 dnm3 C
wc 220 dm3 1667
hznm 157.76 dseq3
f1 570.09 dres3 1.0
f1 1248.4 homo3
th 11615.7 wfile PROCESSING n
ins cdc ph 100.000 proc not used f
nm math fn

```



¹³C NMR

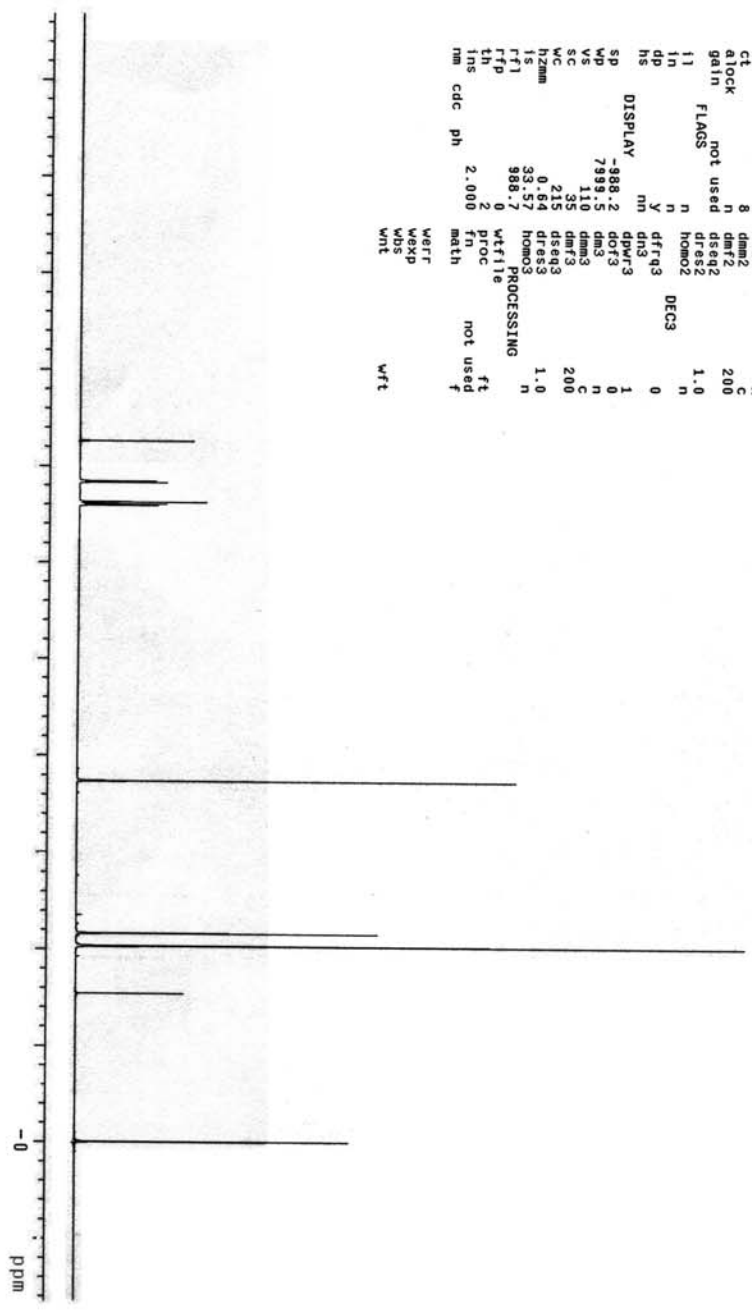




```

exp1 s2pu1
SAMPLE      date Jun 8 2006  dfrq DEC. & VT 598.942
solvent     Jun 8 2006  CDC13  dn          HI
F1 ACQUISITION  exp  dpr          30
F2 ACQUISITION  exp  dpr          30
sfrq       598.942  dn          0
tn         HI      dm          mnr
at         1.892   dseq       200
np         30272   dres       200
sw         8000.0  homo       1.0
fb         4000    homo
us         32      dfrq2     DEC2  0
dprf       5.3    dnr2      1
dn         0      ddf2      1
tof        8      dm2       n
nt         8      dm2       n
ct         8      dm2       n
alock      not used  dseq2     200
gain       not used  dres2     200
flags      not used  dres2     1.0
ll         n      homo2     n
ln         n      homo2     n
dp         y      dfrq3     DEC3  0
hs         y      dprf3     1
DISPLAY    -888.2   dprf3     1
wp         7999.5  dm3       n
vs         110    dm3       n
sc         35     dm3       n
wc         215    dseq3     200
hzmm       0.84   dres3     1.0
f1         398.7  homo3     n
f2         398.7  homo3     n
f3         398.7  homo3     n
proc       wftfile
nm cdc ph 2.000  math      not used
warrt
wexp
wds
wnt
wft

```

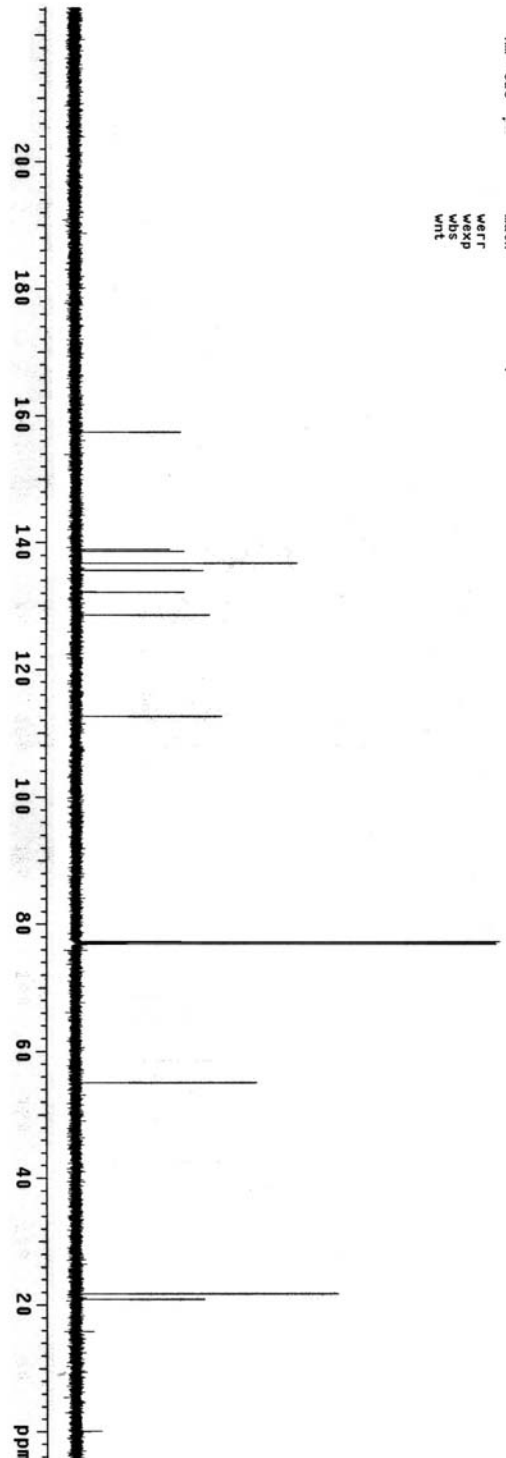
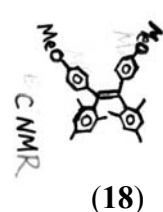


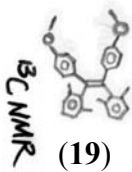
Doneg_PIV

```

SAMPLE 7 2006 dfr VT 599 42
date Jun CDC13 dfr H1
solvent CDC13 dfr H1
F1 ACQUISITION exp dfr 30
sfreq 150.871 dm dfr 0
tn C13 dmm W 7
at 1.300 dmf
np 90272 dseq
sw 34707.2 dres
td 13008 homo
tpwr 61 dfrq2
pw 6.0 dn2
d1 3.000 dpwr2
tof 3017.4 dof2
nt 30000 dm2
ct 1328 dmm2
atlock n dm2
gain not used dseq2
11 n dres2
in n homo2
dp n
hs Y dfrq3
DISPLAY nm dn3
sp -972.8 dpwr3 1
wp 34706.2 dms3 0
vc 0 dmf3 16667
sc 250 dseq3
wzmm 138.83 dres3 1.0
f1 500.00 homo3 PROCESSING
rf1 12489.2 wif1e
tn 11615.8 proc
ms 000 math not used
nm cdc

```





Dong_PIV_113_13C
exp2 s2pu1

date	SAMPLE	8 2006	dfreq	DEC. & VT	599.942
solvent	Jun	CDCl3	dn		38
ft		CDCl3	dpwr		38
ACQUISITION	exp		dof		0
sfreq	150.871	dm		nyy	W
tn	C13	dmm			
at	1.300	dmf			16667
np	90272	dseq			
sw	34707.2	dres			1.0
fd	19000	homo			n
bs	5	dfreq2	DEC2		0
dpwr	6.0	dp2			
dl	3.000	dpwr2			1
tof	9817.4	dof2			0
nt	30000	dm2			n
ct	1256	dmm2			C
atlock	n	dmf2			16667
gain	not used	dseq2			
FLAGS	n	dres2			1.0
11	n	homo2	DEC3		0
1n	y	dfreq3			
dp		dp3			
hs		dpwr3			1
DISPLAY	nh	dof3			0
sp	-871.4	dm3			n
wp	34706.6	dmm3			C
vs	100	dmf3			16667
sc	0	dseq3			
wc	250	dres3			1.0
hzmm	198.83	dres3			n
12	12987.0	homo	PROCESSING		
13	11615.8	wtfile			
ffp	11	proc			
th	11	fn			not used
lms	100.000	math			f
nm	cdc	ph			
		werr			
		wexp			
		wds			
		wnt			



Dong_P111_103_1H

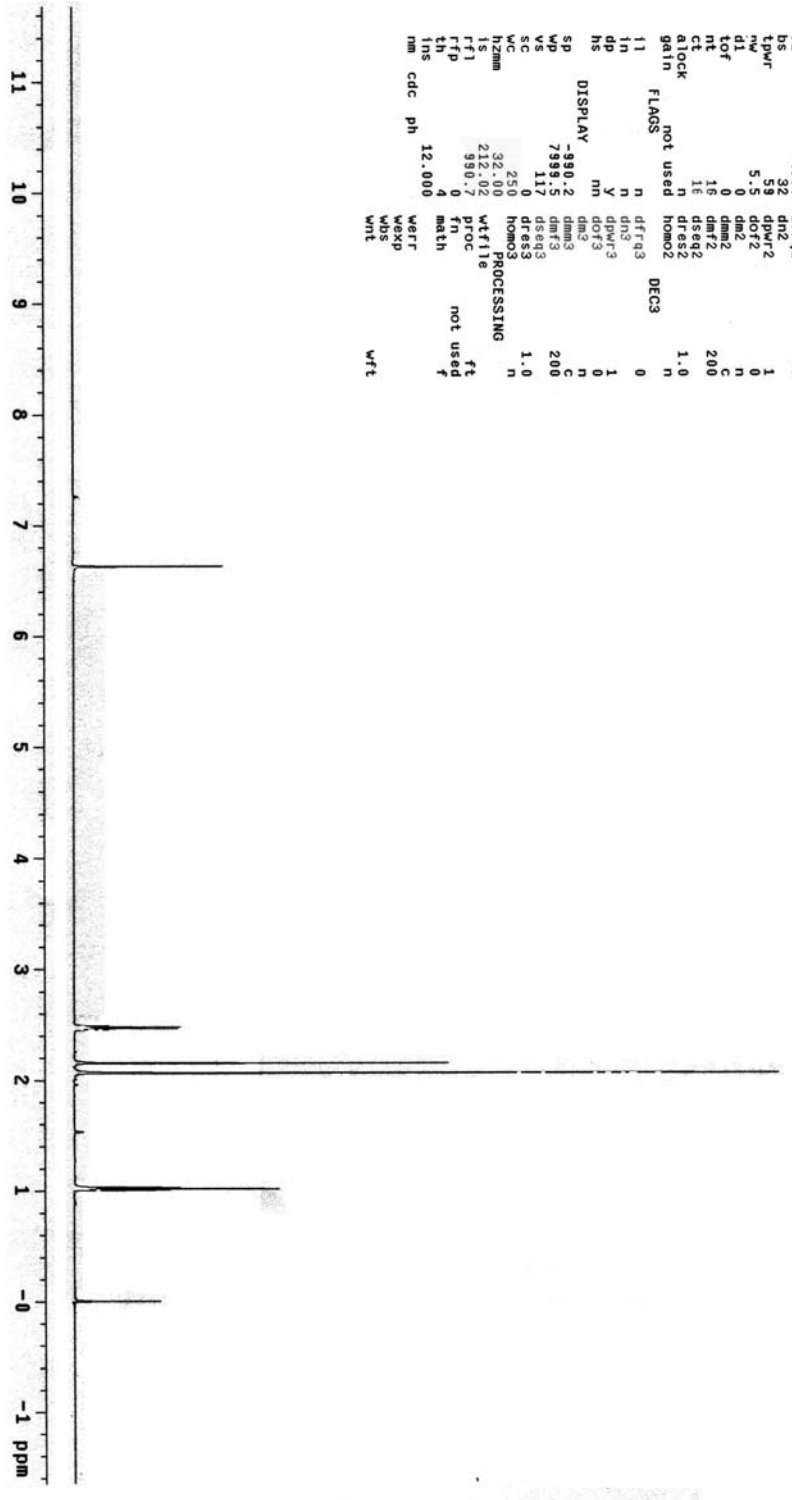


1H NMR (20)

```

exp4 s2pui
SAMPLE Nov 22 2005 DEC. & VI 599.942
date Nov 22 2005 dfrq dn H1
solvent CDC13 dn H1
file /data/hu/data~ dpwr 30
/dong/Nov2205_Dong~ dof 0
_P111_103_1H_11d dm nmh
_P111_103_1H_11d dm nmh
ACQUISIT 599.942 dmf 200
sfrq HI dseg
tn HI dseg
at 1.892 dres
nd 30272 homo
sw 8000.0 DECE2
fb 4000 dfrq2
ds 32 dnt2
lpwr 32 dpwr2
wv 5.3 dnt2
d1 0 dnt2
tof 0 dnt2
nt 16 dnt2
ct 16 dnt2
atlock n dres2
gain not used homo2
FLAGS not used DECE3
11 n dfrq3
11 n dnt3
11 y dnt3
hs nm dnt3
DISPLAY -990.2 dnt3
sp 7999.5 dnt3
vs 117 dnt3
sc 0 dnt3
WC 250 HOMOPROCESSING
Hzmm 32.250
f1 212.02 wfile
rfp 990.7 proc
th 0 fn
ins cdc ph 12.000 math
nm cdc ph Weir
Wexp
WDS
WNT wft

```






Dong_P111_103_13C

expd s2pu1

```

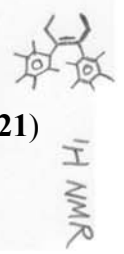
SAMPLE          DEC. & VT
date Nov 22 2005 dfrq 599.942
solvent CCl3      dn      H1
file /data/hu/data~ dpwr 39
/hong/NOV2005/Dong~ dof 0
_P111_103_13C.fid dm      nny
_ACQUISITION 158.871 dnm  W
sfreq 158.871      dm      15877
at C13             dres  1.0
np 1.300           dres  1.0
sw 90272          homo  25.0
fb 34707.2       temp  25.0
bs 19000         dfrq2  0
tpwr 61          dn2  dpr2  1
pw 3.0           dn2  dpr2  1
td 3017.4        dn2  dpr2  n
nt 30000         dmf2  16667
ct 568          dmf2  16667
atlock n         dseq2  1.0
gain not used   dres2  1.0
flags          homo2  n
11 n            dfrq3  0
in n            dn3  dpr3  1
dp y           dn3  dpr3  1
ns Y           dof3  0
DISPLAY -871.3  dms  0
sp 34706.6      dms3  n
vs 76          dmf3  16667
sc 0           dmf3  16667
wc 225         dseq3  1.0
hzmm 154.25    dres3  n
rf1 12987.6    homo2PROCESSING
rfi 11815.8    wfile
th 11          fn      not used
ns 100.000    math  f
nm           werr
           wexp
           wds
           wnt

```



 BC NMR (20)





```

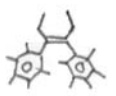
Donng_PIV_108_1H
exp1 szpui
SAMPLE DEC. & VT
date May 13 2006 dfrq 599.942
solvent CDCl3 dn H1
file exp dpwr 30
ACQUISITION dof 0
sfrq 599.942 dm mm
nu 1000000 dmf 200
at 1.892 dres 200
np 30272 dres 1.0
sw 8000.0 homo n
fb 4000 DECC2
bs 32 dfrq2 0
tpwr 59 dn2 1
pw 6.0 dn2 1
dl 3.000 dpwr2 1
lor 8 do2 0
hl 8 do2 0
ct 8 dms2 C
atlock n dsq2 200
gain not used dsq2 1.0
11 n homo2 n
in n dfrq3 DECC3 0
dp y dfrq3 0
hs DISPLAY nn dn3 1
sp -88.6 dpwr3 1
ve 789.5 dm3 n
sc 90 dms3 C
wc 220 dsq3 200
h2mm 36.36 dres3 1.0
is 343.34 homo3
rf1 889.1 WFFILE PROCESSING
rfp 0 WFFILE
ln 3 Proc
ns 3 Proc not used
nm cdc ph 12.000 math
werr werr
wexp wexp
wbs wbs
wnt wnt WFF
  
```



Dong_PIV

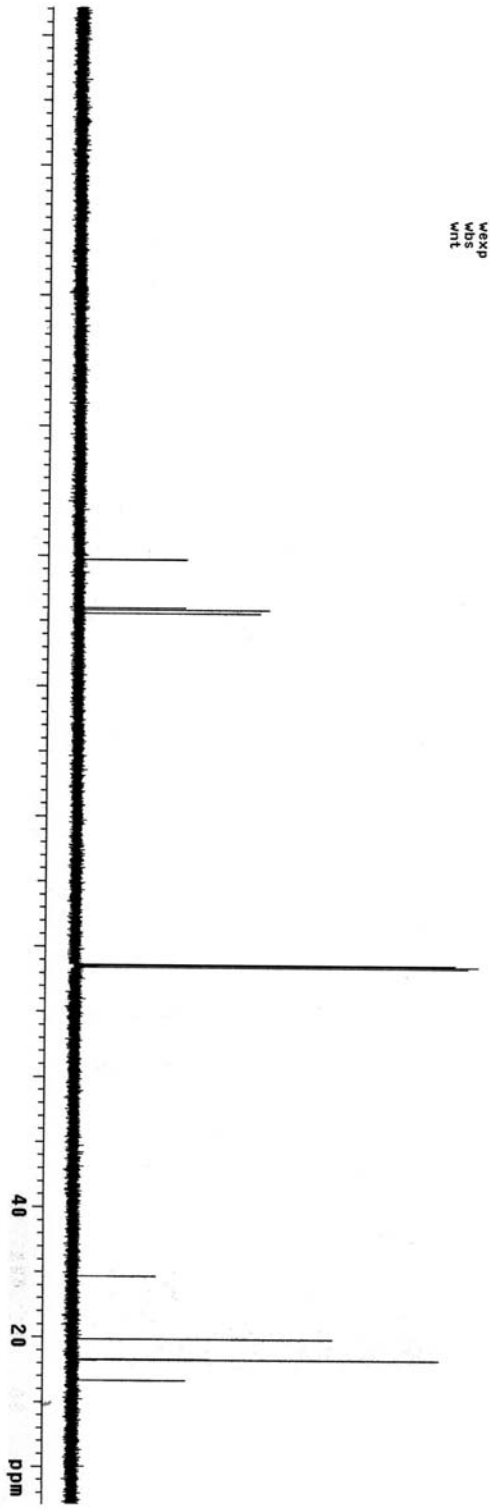
exp2

date	SAMPLE	May 13 2006	dfreq	142
solvent	CDC13	exp	dn	H1
file	ACQUISITION	exp	dpwr	39
sfreq	150.871	dm	dcf	0
tn	1.300	dmm	nmv	17
at	90272	dsef	dseq	0
sw	34707.2	dres	homo	n
fb	19000	homo	homo	n
bs	8	dfri	dfri	n
tpwr	60	dm2	dm2	1
dw	3.150	dpf2	dpf2	0
tof	3017.4	dm2	dm2	n
nt	5000	dmm2	dmm2	n
ct	984	dsef2	dsef2	7
atlock	not used	dres2	dres2	0
gain	-LAGS	homo2	homo2	n
l1	n	dfq3	dfq3	DEC3
l2	n	dfq3	dfq3	DEC3
l3	n	dfq3	dfq3	DEC3
l4	n	dfq3	dfq3	DEC3
l5	n	dfq3	dfq3	DEC3
l6	n	dfq3	dfq3	DEC3
l7	n	dfq3	dfq3	DEC3
l8	n	dfq3	dfq3	DEC3
l9	n	dfq3	dfq3	DEC3
l10	n	dfq3	dfq3	DEC3
l11	n	dfq3	dfq3	DEC3
l12	n	dfq3	dfq3	DEC3
l13	n	dfq3	dfq3	DEC3
l14	n	dfq3	dfq3	DEC3
l15	n	dfq3	dfq3	DEC3
l16	n	dfq3	dfq3	DEC3
l17	n	dfq3	dfq3	DEC3
l18	n	dfq3	dfq3	DEC3
l19	n	dfq3	dfq3	DEC3
l20	n	dfq3	dfq3	DEC3
l21	n	dfq3	dfq3	DEC3
l22	n	dfq3	dfq3	DEC3
l23	n	dfq3	dfq3	DEC3
l24	n	dfq3	dfq3	DEC3
l25	n	dfq3	dfq3	DEC3
l26	n	dfq3	dfq3	DEC3
l27	n	dfq3	dfq3	DEC3
l28	n	dfq3	dfq3	DEC3
l29	n	dfq3	dfq3	DEC3
l30	n	dfq3	dfq3	DEC3
l31	n	dfq3	dfq3	DEC3
l32	n	dfq3	dfq3	DEC3
l33	n	dfq3	dfq3	DEC3
l34	n	dfq3	dfq3	DEC3
l35	n	dfq3	dfq3	DEC3
l36	n	dfq3	dfq3	DEC3
l37	n	dfq3	dfq3	DEC3
l38	n	dfq3	dfq3	DEC3
l39	n	dfq3	dfq3	DEC3
l40	n	dfq3	dfq3	DEC3
l41	n	dfq3	dfq3	DEC3
l42	n	dfq3	dfq3	DEC3
l43	n	dfq3	dfq3	DEC3
l44	n	dfq3	dfq3	DEC3
l45	n	dfq3	dfq3	DEC3
l46	n	dfq3	dfq3	DEC3
l47	n	dfq3	dfq3	DEC3
l48	n	dfq3	dfq3	DEC3
l49	n	dfq3	dfq3	DEC3
l50	n	dfq3	dfq3	DEC3
l51	n	dfq3	dfq3	DEC3
l52	n	dfq3	dfq3	DEC3
l53	n	dfq3	dfq3	DEC3
l54	n	dfq3	dfq3	DEC3
l55	n	dfq3	dfq3	DEC3
l56	n	dfq3	dfq3	DEC3
l57	n	dfq3	dfq3	DEC3
l58	n	dfq3	dfq3	DEC3
l59	n	dfq3	dfq3	DEC3
l60	n	dfq3	dfq3	DEC3
l61	n	dfq3	dfq3	DEC3
l62	n	dfq3	dfq3	DEC3
l63	n	dfq3	dfq3	DEC3
l64	n	dfq3	dfq3	DEC3
l65	n	dfq3	dfq3	DEC3
l66	n	dfq3	dfq3	DEC3
l67	n	dfq3	dfq3	DEC3
l68	n	dfq3	dfq3	DEC3
l69	n	dfq3	dfq3	DEC3
l70	n	dfq3	dfq3	DEC3
l71	n	dfq3	dfq3	DEC3
l72	n	dfq3	dfq3	DEC3
l73	n	dfq3	dfq3	DEC3
l74	n	dfq3	dfq3	DEC3
l75	n	dfq3	dfq3	DEC3
l76	n	dfq3	dfq3	DEC3
l77	n	dfq3	dfq3	DEC3
l78	n	dfq3	dfq3	DEC3
l79	n	dfq3	dfq3	DEC3
l80	n	dfq3	dfq3	DEC3
l81	n	dfq3	dfq3	DEC3
l82	n	dfq3	dfq3	DEC3
l83	n	dfq3	dfq3	DEC3
l84	n	dfq3	dfq3	DEC3
l85	n	dfq3	dfq3	DEC3
l86	n	dfq3	dfq3	DEC3
l87	n	dfq3	dfq3	DEC3
l88	n	dfq3	dfq3	DEC3
l89	n	dfq3	dfq3	DEC3
l90	n	dfq3	dfq3	DEC3
l91	n	dfq3	dfq3	DEC3
l92	n	dfq3	dfq3	DEC3
l93	n	dfq3	dfq3	DEC3
l94	n	dfq3	dfq3	DEC3
l95	n	dfq3	dfq3	DEC3
l96	n	dfq3	dfq3	DEC3
l97	n	dfq3	dfq3	DEC3
l98	n	dfq3	dfq3	DEC3
l99	n	dfq3	dfq3	DEC3
l100	n	dfq3	dfq3	DEC3



(21)

¹³C NMR



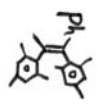
Dong_VIV_P34_1H

exp1 s2pul1

```

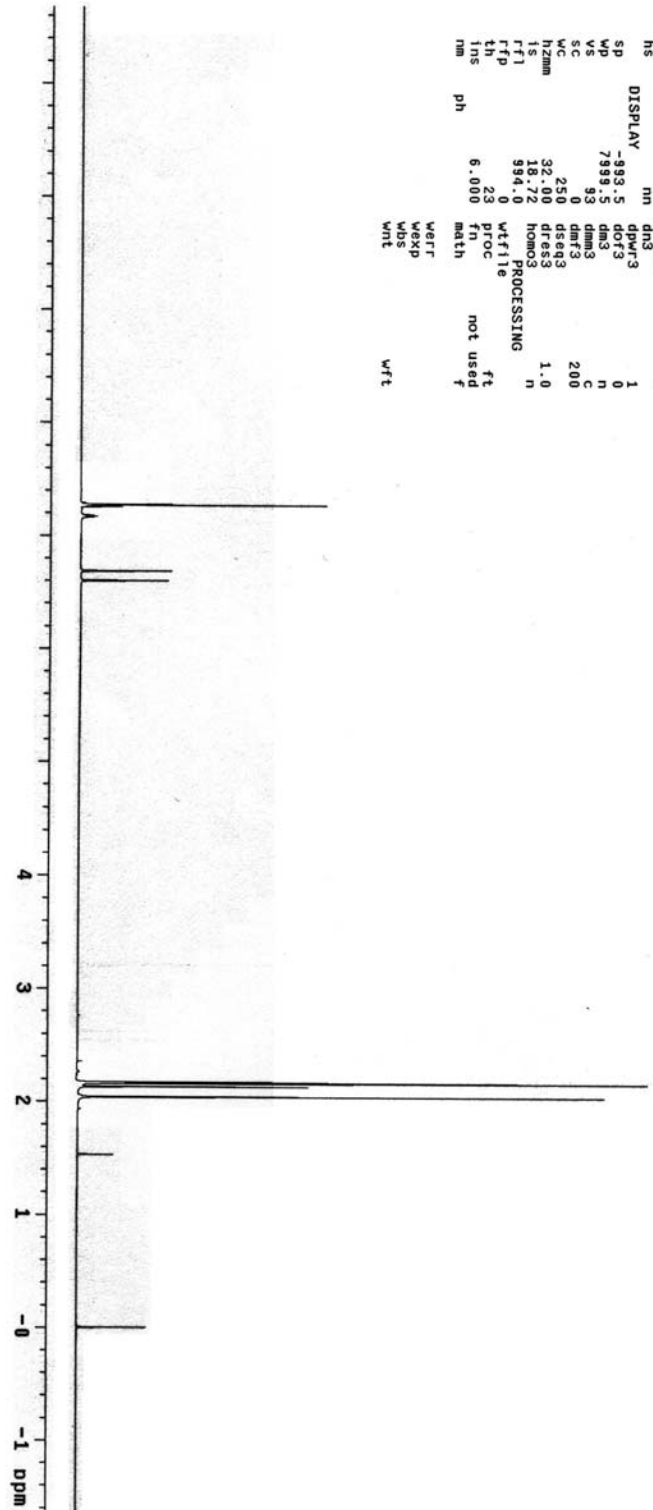
SAMPLE      date Jun 21 2006  DEC. & VT  539.942
solvent     Jun 21 2006  CDC13  dn
file        exp dof      dpwr      30
ACQUISITION 599.942  dm          0
           tn          H1 dnm      nmn
           dt          1.692  dmf      C
           sp          3272  dseq     200
           fb          8000  hres     1.0
           bs          4000  homo     n
           tpwr        58    dfrq2     DEC2
           dl          0     dn2      0
           tof         0     dpwr2    1
           nt          16    dof2     0
           ct          16    dm2      n
           atlock     not used  dmf2     C
           gain       not used  dseq2    200
           fl         not used  dres2    1.0
           in         n        hres2     n
           in         n        homo2     n
           dp         n        y dfrq3   DEC3
           hs         n        dn3      0
DISPLAY     -993.5  dpwr3    1
           wp         799.5  dof3     0
           vs         93    dm3      n
           sc         0     dnm3    C
           hc         250   dmf3     200
           hzmm       3280  dseq3    1.0
           is         18.72  hres3    n
           rfp        994.6  homo     n
           th         0     wtfile   n
           ins        23    proc     n
           nm         6.000  fn       not used
           ph         0     math     f
           werr       WEXP
           wds        WNT
           wnt        WFT

```



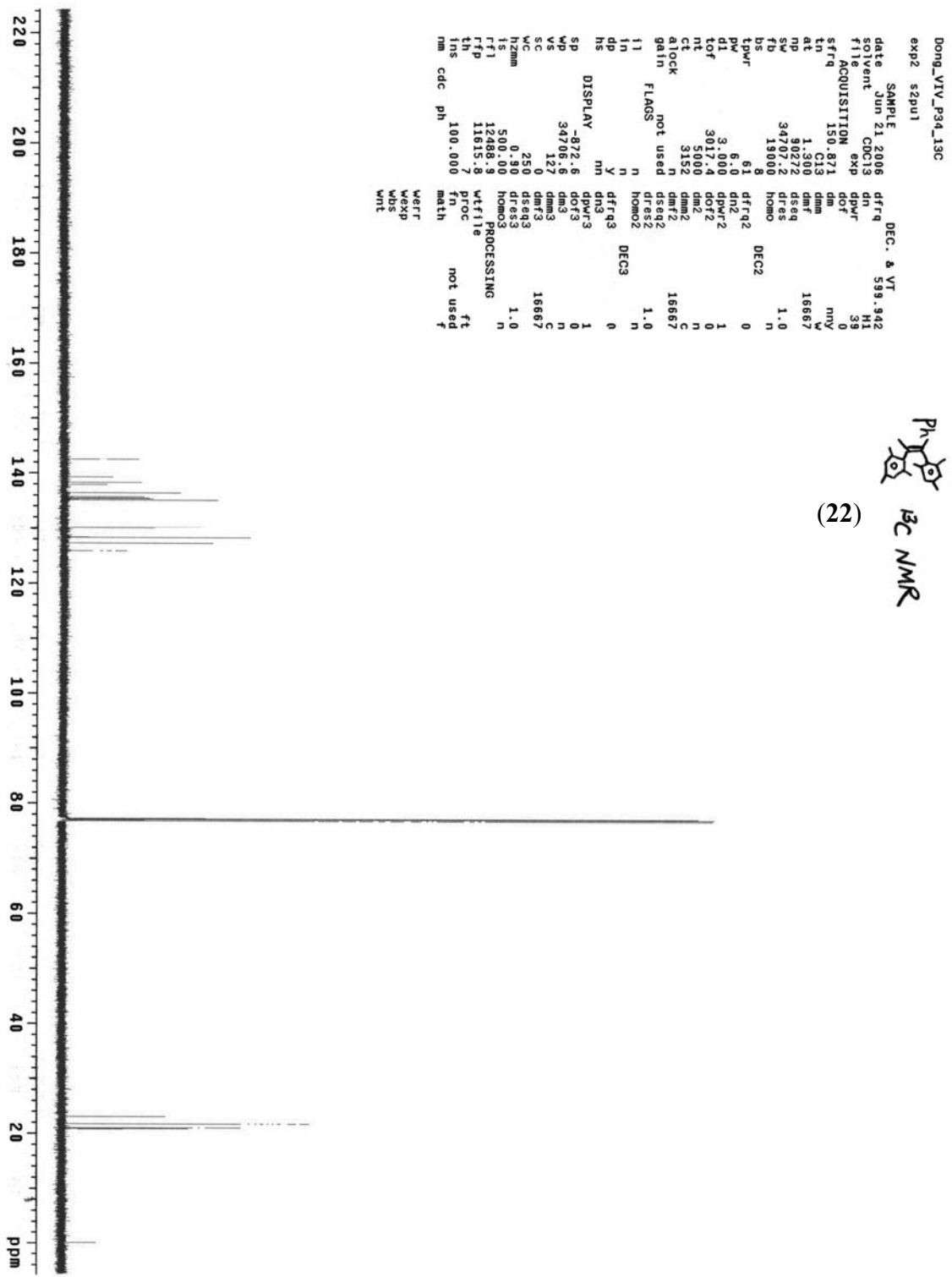
¹H NMR

(22)





(22)



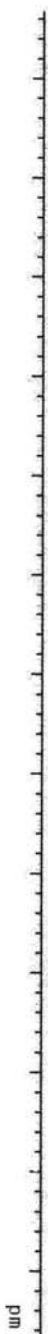
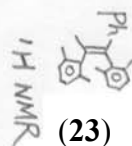
```

Donng_VIV_P34_13C
exp2 s2pu1
SAMPLE DEC. 8 VT
date Jun 21 2006 dfrc 539.942
solvent Jun 21 CDC13 dn H1
file ACQUISITION exp dpwr 39
sfrc 150.871 dm nny 0
fn 133 dnm w 16667
ns 50272 dfr 1.0
sw 34707.2 dres 1.0
fb 18000 homo
bs 8
tpwr 61 dfrc2 DEC2 0
pw 6.0 dn2
d1 3.000 dpwr2 1
lof 3017.4 dof2 0
nt 5000 dms2 n
rl 5152 dnm2 c
atlock not used dres2 16667
gain FLAGS n homo2 1.0
i1 n dfrc3 DEC3 0
in y dfrc3
dp nm dn3
hs DISPLAY -872.6 dpwr3 1
sp -872.6 dof3 0
wp 34706.9 dms3 n
vz 122 dmr3 c
sc 250 dmr3 16667
wc 250 dres3
hzmm 0.90 homo3 PROCESSING 1.0
ts 500.00 vffile
rf1 12488.9 vproc
th 11615.8 tr not used f
ins 100.000 math
nm cdc ph
  
```

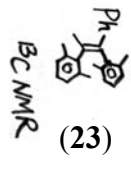
00mg_PIV_116_1H
expi szpul

SAMPLE Jun 30 2006 DEC. & VT
solvent CDC13 exp dn HI
file ACQUISITION exp dpwr 30
sfrq 599.942 dm 0
tn 1.81 dmm nmn
dd 30272 deag c
sw 8000.0 dres 200
fb 4000 homo 1.0
bs 32 DECD
tpwr 59 dfrq2 0
pv 5.5 dn2
dl 0 dpwr2 1
tof 0 dof2 0
nt 8 dm2 n
ct 8 dmm2 c
atlock n dmfr2 200
gain not used dres2 1.0
l1 n dres3
in n homo2 DECD
dn y dfrq3 0
hs DISPLAY nm dn3
sp -997.1 dpwr3 1
wp 7999.5 dof3 0
vs 90 dmm3 n
sc 35 dmf3 c
wc 215 dseq3 200
hzmin 0.62 dres3
ls 33.57 homo3 PROCESSING 1.0
rfp 997.6 wffile
th 3 proc
ins 9 fn
nm cdc ph 6.000 math not used f

werr
wexp
wbs
wnt
wfl



Dong_PTV_116_13C
exp2 szput1

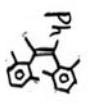


SAMPLE DEC. & VT
date Jun 14 2006 dfrq 599.942
file C038 dfrq 31
ACQUISITION exp dfrq 31
sfrq 150.871 dm 0
tn C15 dnm my
at 1.390 dmf 16667
sp 1.272 dseq 1.0
fb 3470.0 dms 1.0
bs 19000 homo
tpwr 54 dfrq2 DEC2 0
pw 6.0 dn2
dl 3.000 dpwr2 1
tof 3017.4 dot 0
ct 20000 dms 1
atlock n dmf2
gain not used dseq2 16667
FLAGS not used dres2 1.0
11 n dfrq3 DEC3 0
1n y dfrq3
dp nm dms
hs DISPLAY -873.1 dot3 1
sp 34706.6 dms 0
wp 162 dnm3 n
vs 0 dmf3 C
sc 250 dseq3 16667
wc 1.22 dfrq3 1.0
12mm 500.000 homo
rf1 12489.4 lb PROCESSING
th 11615.8 8 wtf file 0.50
fns 100.000 8 proc
nm cdc ph fn math not used
f

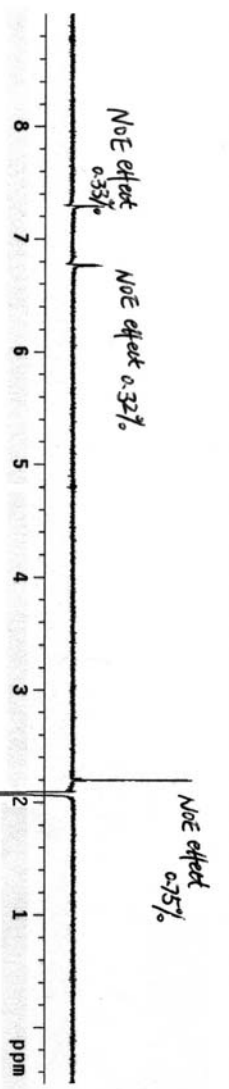
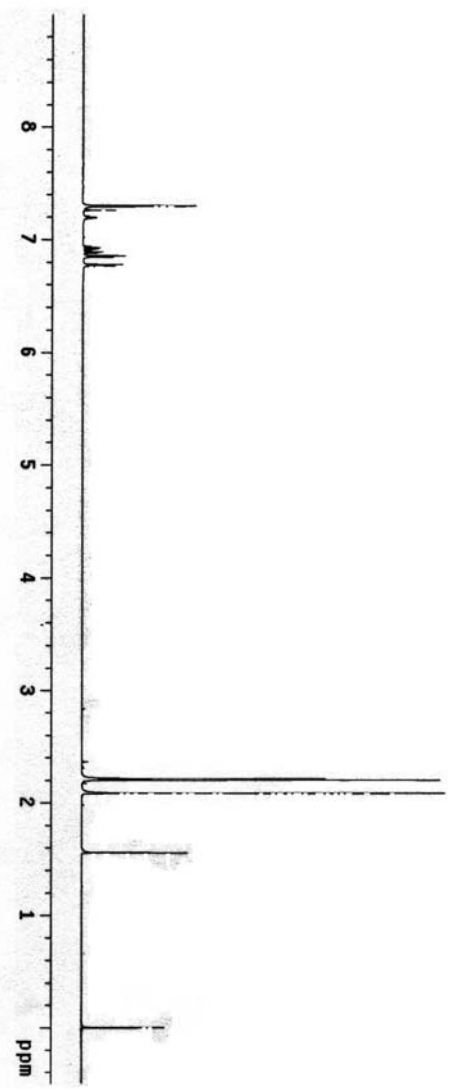
Wtfr
Wexp
Wds
Wnt



Donng_PTV_116_noesy1D
 Pulse Sequence: noesy1D_da
 Solvent: CDCl3
 Ambient temperature
 INOVA-600 "nb000"
 Relax. delay 2.000 sec
 Pulse 90.0 degrees
 Mixing 0.400 sec
 Acq time 1.892 sec
 Width 8000.0 Hz
 344 repetitions
 OBSERVE H1, 599.9393058 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 hr, 29 min, 28 sec



(23) NOE Experiment





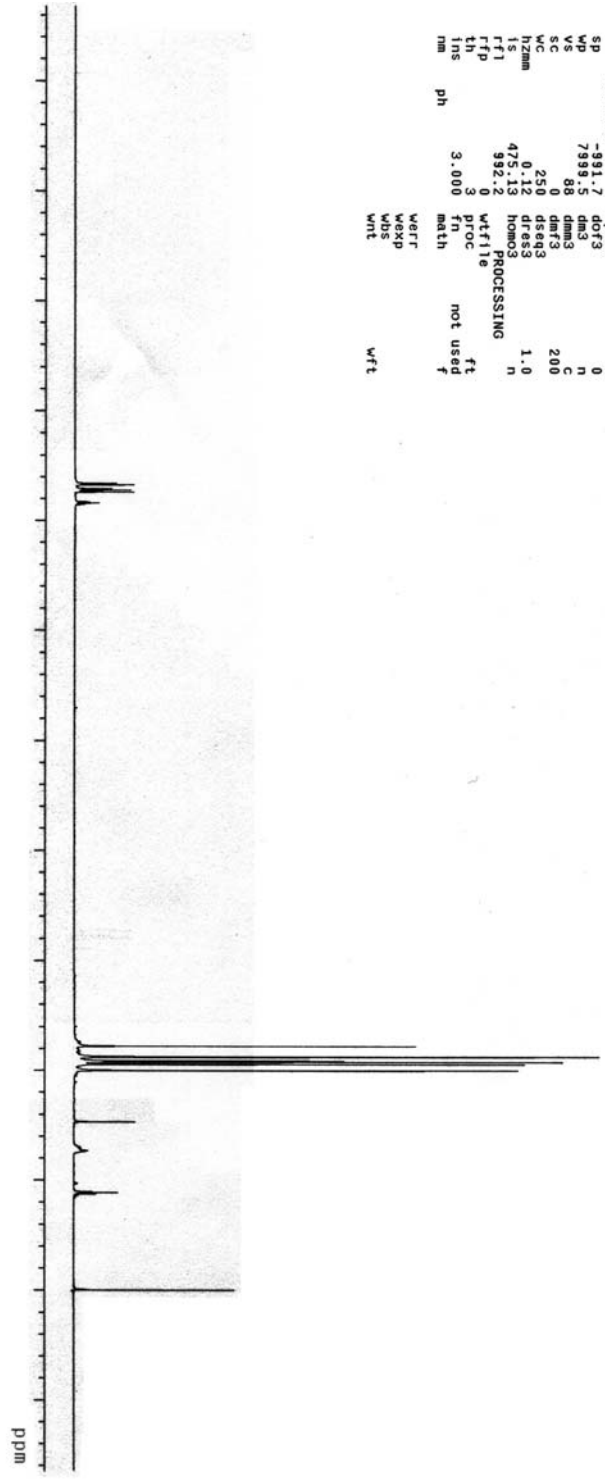
(24)

1H NMR

```

Dong_PIV_123_1H
exp1 s2pu1
SAMPLE
date Jun 23 2006 DEC. & VT 599.942
solvent CDC13 dn dpwr 30
file ACQUISITION exp dof 0
sfrq 599.942 dm mnm
tn 1.842 dmf1 n
nd 30272 dseq 200
sw 8000.0 dres 1.0
fb 4000 homo n
bs 4 dfrq2 DEC2 0
tpwr 58 dfrq3 0
pw 5.5 dn2 1
di 0 dpwr2 1
cof 0 ddf2 n
ct 18 ddm2 n
atlock n dmf2 200
gain not used dseq2 1.0
11 n homo2 n
1n n dfrq3 DEC3 0
dp Y dn3 1
ns DISPLAY nm dn3 0
SP -991.7 dof3 0
VP 7999.5 dm3 n
VE 88 dmf3 c
VC 0 dmf3 200
WC 250 dseq3 1.0
Hzmm 0.12 dres n
f1 473.13 HOMOPROCESSING
f2 392.6 wfile
f3 3.000 math
th 3 fn not used
ins 3 ft
nm ph 3.000 math wft
WEIR
WEXP
WOS
WNT

```



Dong_PIV_123_moesy1D
Pulse Sequence: moesy1D_da
Solvent: CDCl3
Ambient temperature
INOVA-600 "u600"
Relax. delay 2.000 sec
Pulse 90.0 degrees
Mixing 0.400 sec
Acq. time 1.892 sec
Width 8000.0 Hz
Spectral width
Observed H1 599.9392939 MHz
DATA PROCESSING
FT size 32758
Total time 1 hr, 24 min

(24) NOE

