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

Cheng-Guo Dong, Pik Yeung and Qiao-Sheng Hu*

Department of Chemistry, College of Staten Island and the Graduate Center of the City University of New York, Staten Island, New York 10314

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 ^{1}H 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-dibromethane, anhydrous iron(III) chloride were purchased from Aldrich and used directly. $Pd(OAc)_2$ was a gift from Frontier Scientific, Inc. PPh_3 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^{o}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 ^{1}H 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 PPh_3 (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 ^{1}H 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 ^{1}H 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{23}H_{20}$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{22}H_{18}$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{25}H_{24}$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{23}H_{20}O$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{25}H_{24}O_2$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{24}H_{22}O_2$: C, 84.18%; H, 6.48%. Found: C, 83.84%; H, 6.38%.

2,3-Diethyl-4,6-dimethyl-1H-indene (**7**): colorless liquid. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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**): ^{1}H NMR showed a 91: 9 ratio. Analytic sample of 3-butyl-4,6-dimethyl-2-*p*-tolyl-1H-indene was obtained by recrystalization 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. ^{1}H NMR ($CDCl_3$, 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 $C_{22}H_{26}$: 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 recrystalization 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. ^{1}H NMR ($CDCl_3$, 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).). ^{13}C NMR ($CDCl_3$, 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 PPh_3 (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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{32}H_{32}$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{32}H_{32}O_2$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{34}H_{36}O_2$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{25}H_{26}$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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 $C_{27}H_{30}$: 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. ^{1}H NMR ($CDCl_3$, 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). ^{13}C NMR ($CDCl_3$, 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%).

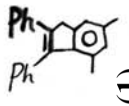
Reference:

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.

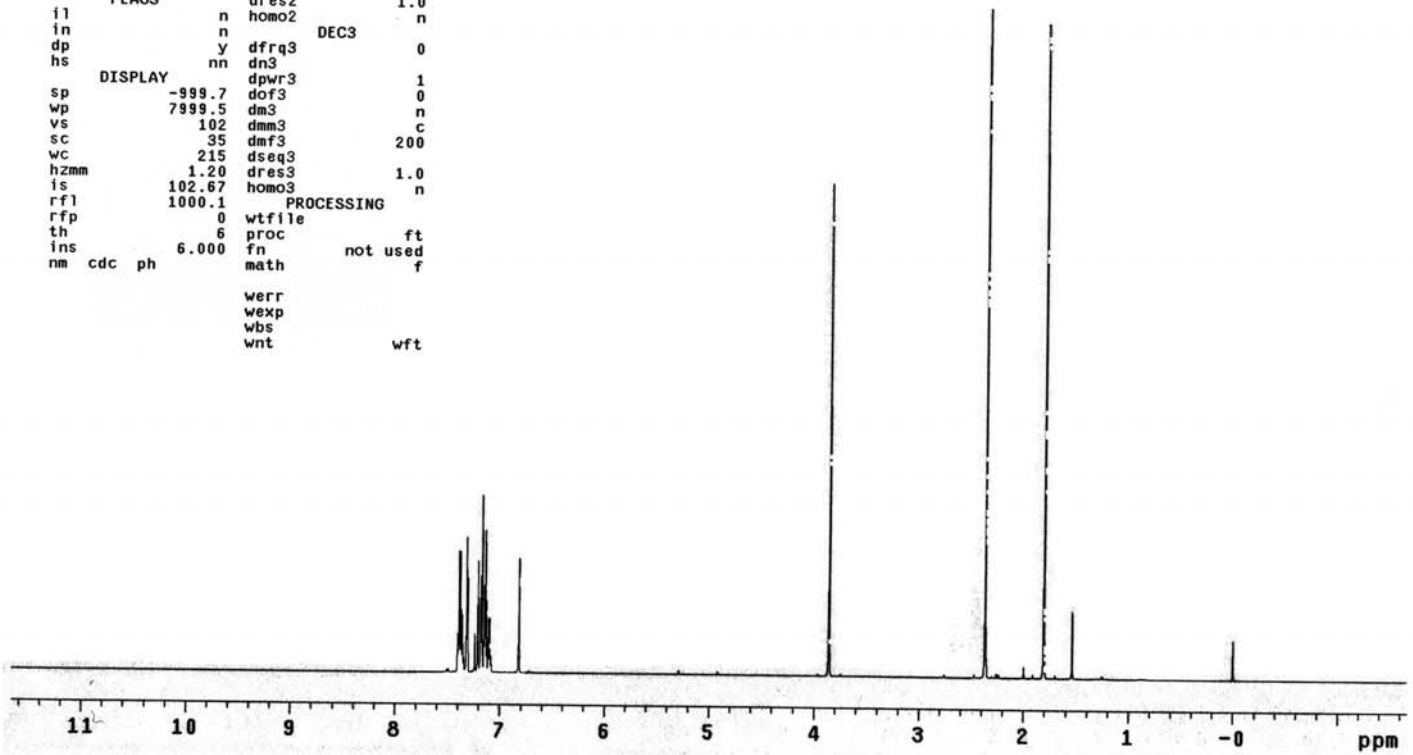
Dong_PIV_102_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jun 14 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.942	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	32	DEC2	
tpwr	58	dfrq2	0
pw	5.5	dn2	
d1	0	dpwr2	1
tof	0	dof2	0
nt	16	dm2	n
ct	0	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-999.7	dof3	0
wp	7999.5	dm3	n
vs	102	dmm3	c
sc	35	dmf3	200
wc	215	dseq3	
hzmm	1.20	dres3	1.0
is	102.67	homo3	n
rfl	1000.1	PROCESSING	
rfp	0	wtfile	
th	6	proc	ft
ins	6.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



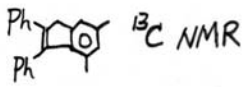
(1) ^{1}H NMR



Dong_PIV_102_13C

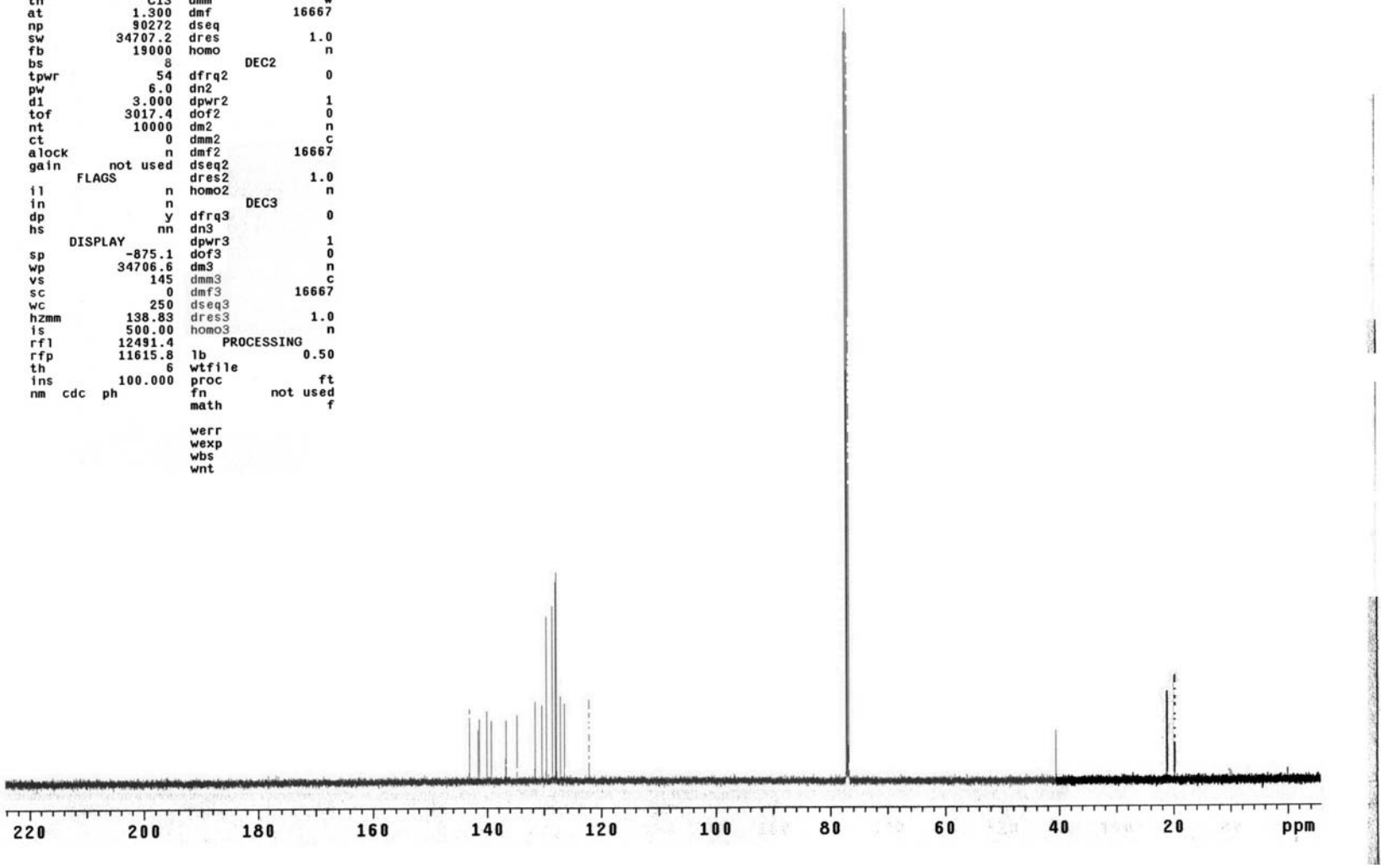
exp2 s2pul

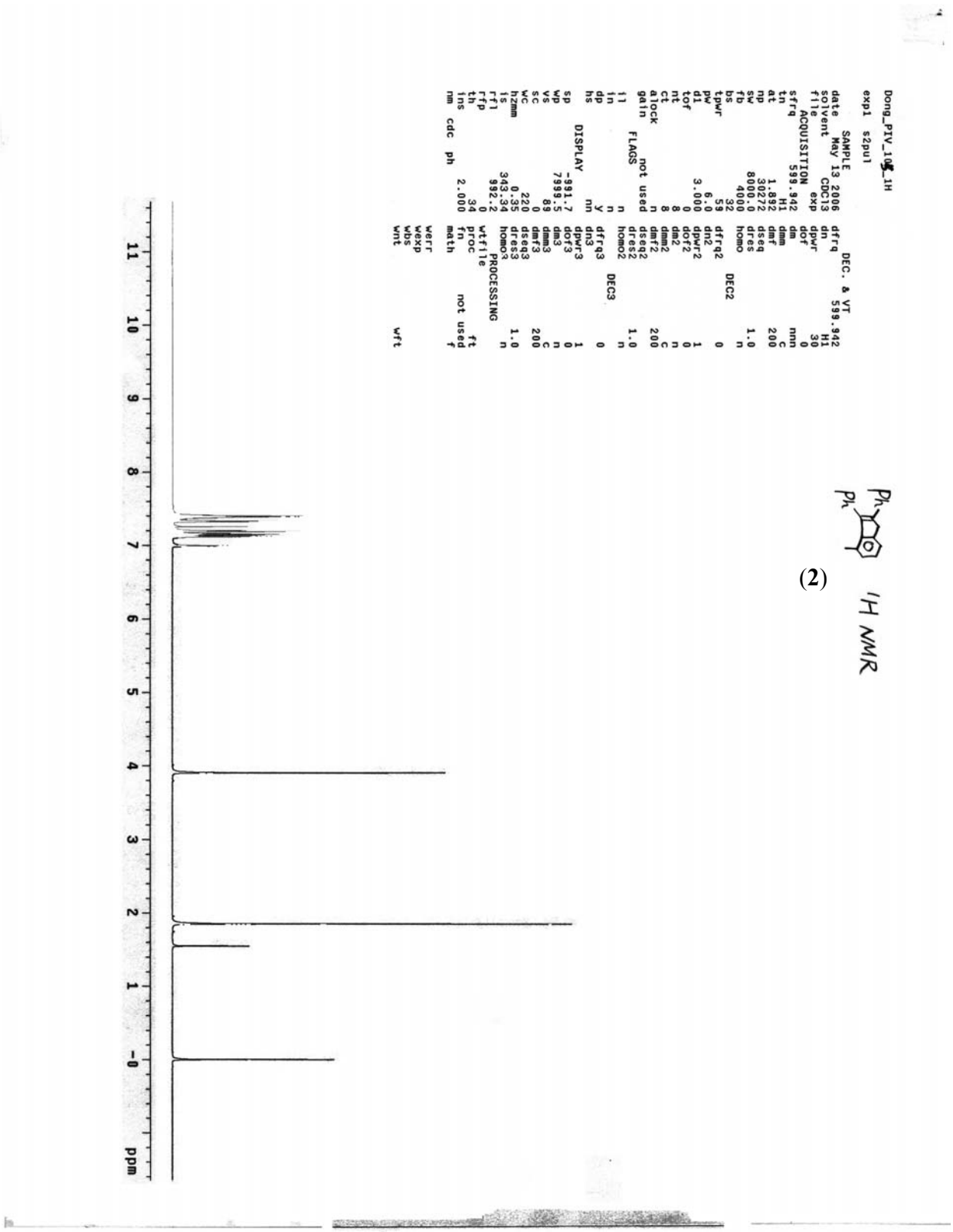
SAMPLE		DEC. & VT	
date	Jun 14 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	16667
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	DEC2	
tpwr	54	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	3017.4	dof2	0
nt	10000	dm2	n
ct	0	dmm2	c
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-875.1	dof3	0
wp	34706.6	dm3	n
vs	145	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	138.83	dres3	1.0
is	500.00	homo3	n
rfl	12491.4	PROCESSING	
rfp	11615.8	lb	0.50
th	6	wtfile	
ins	100.000	proc	ft
nm cdc ph		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



^{13}C NMR

(1)

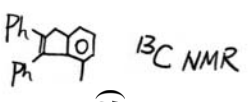



Dong_PIV_1H
exp1 s2pul
SAMPLE
date May 13 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 32
tpwr 59
pw 6.0
d1 3.000
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -991.7
wp 7999.5
vs 89
sc 0
wc 220
hzmm 0.35
is 343.34
rfl 992.2
rfp 0
th 34
ins 2.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 30
dof 0
dm nnn
dmf c
200
dseq
dres
homo 1.0
DEC2 n
dfrq2 0
dn2
dpwr2
dof2
dm2
dmm2 c
dmf2 200
dseq2
dres2
homo2 1.0
DEC3 n
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
Ph
Ph
1H NMR
(2)
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

Dong_PIV_105_13C

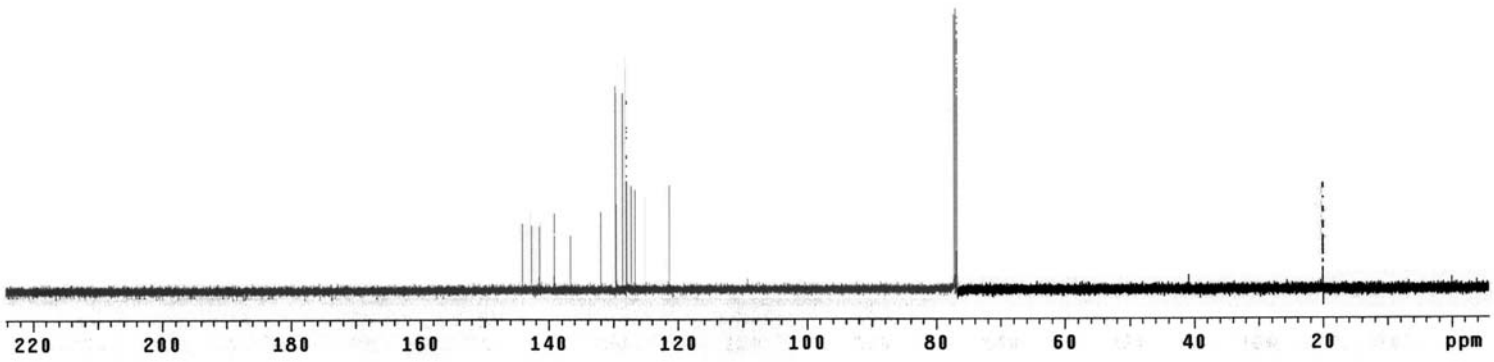
exp2 s2pul

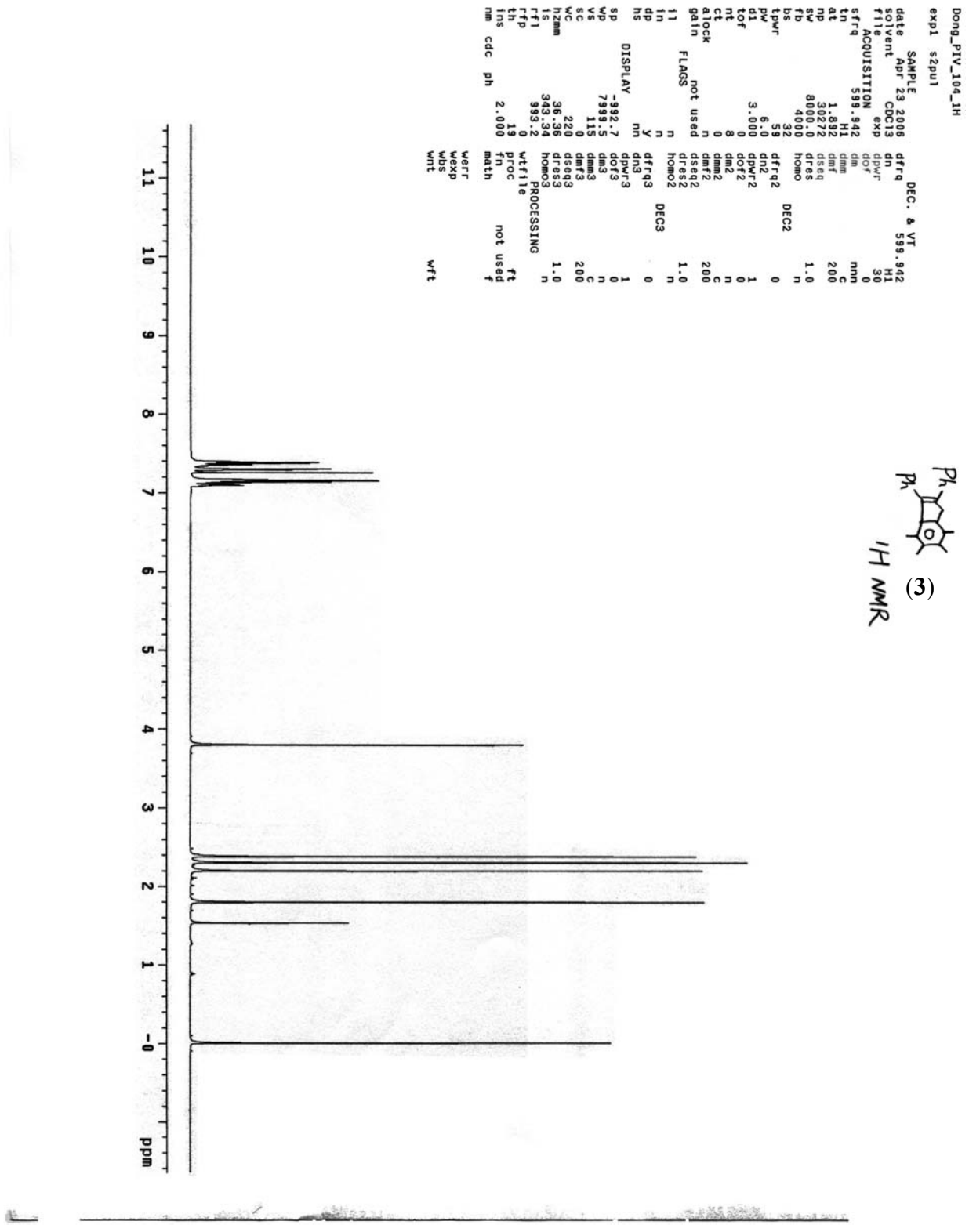
SAMPLE		DEC. & VT	
date	May 13 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	16667
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	DEC2	
tpwr	61	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	3017.4	dof2	0
nt	5000	dm2	n
ct	664	dmm2	c
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-876.8	dof3	0
wp	34706.6	dm3	n
vs	47	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	1.28	dres3	1.0
is	500.00	homo3	n
rfl	12493.1	PROCESSING	
rfp	11615.8	wtfile	
th	7	proc	ft
ins	100.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	



^{13}C NMR

(2)




Dong_PIV_104_1H
exp1 s2pul
SAMPLE
date Apr 23 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 32
tpwr 59
pw 6.0
d1 3.000
tof 0
nt 8
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -992.7
wp 7999.5
vs 115
sc 0
wc 220
hzmm 36.36
is 343.34
rfl 993.2
rfp 0
th 19
ins 2.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
11 10 9 8 7 6 5 4 3 2 1 -0 ppm

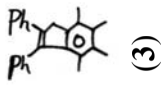
(**3**)

1H NMR

Dong_PIV_104_13C

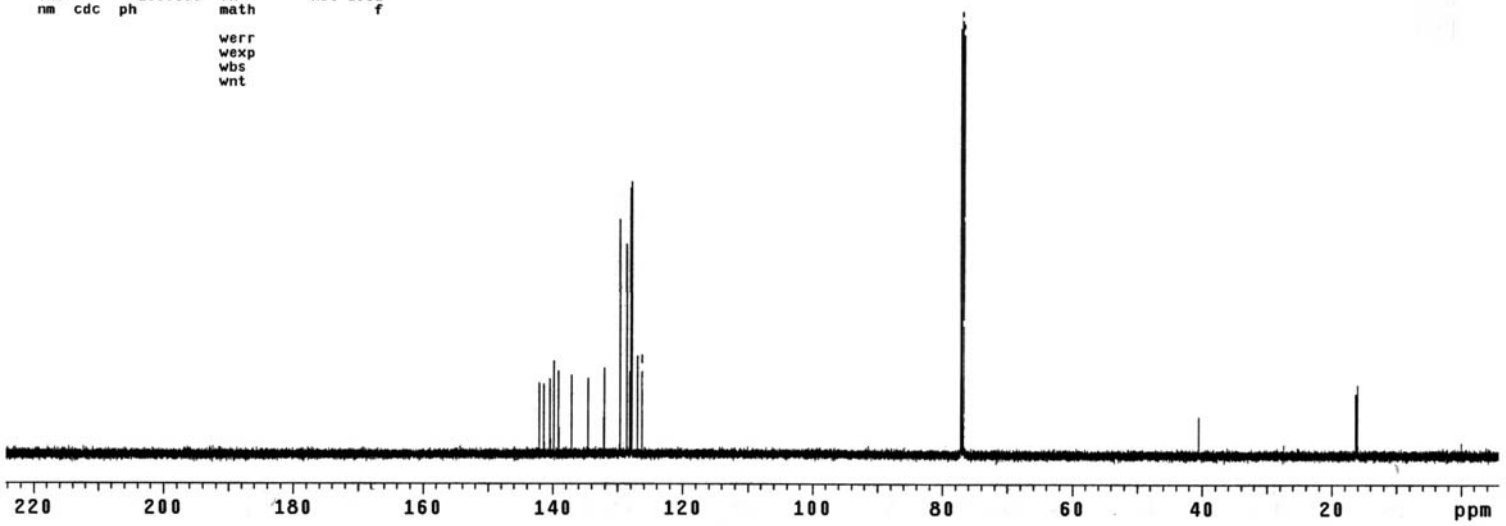
exp2 s2pul

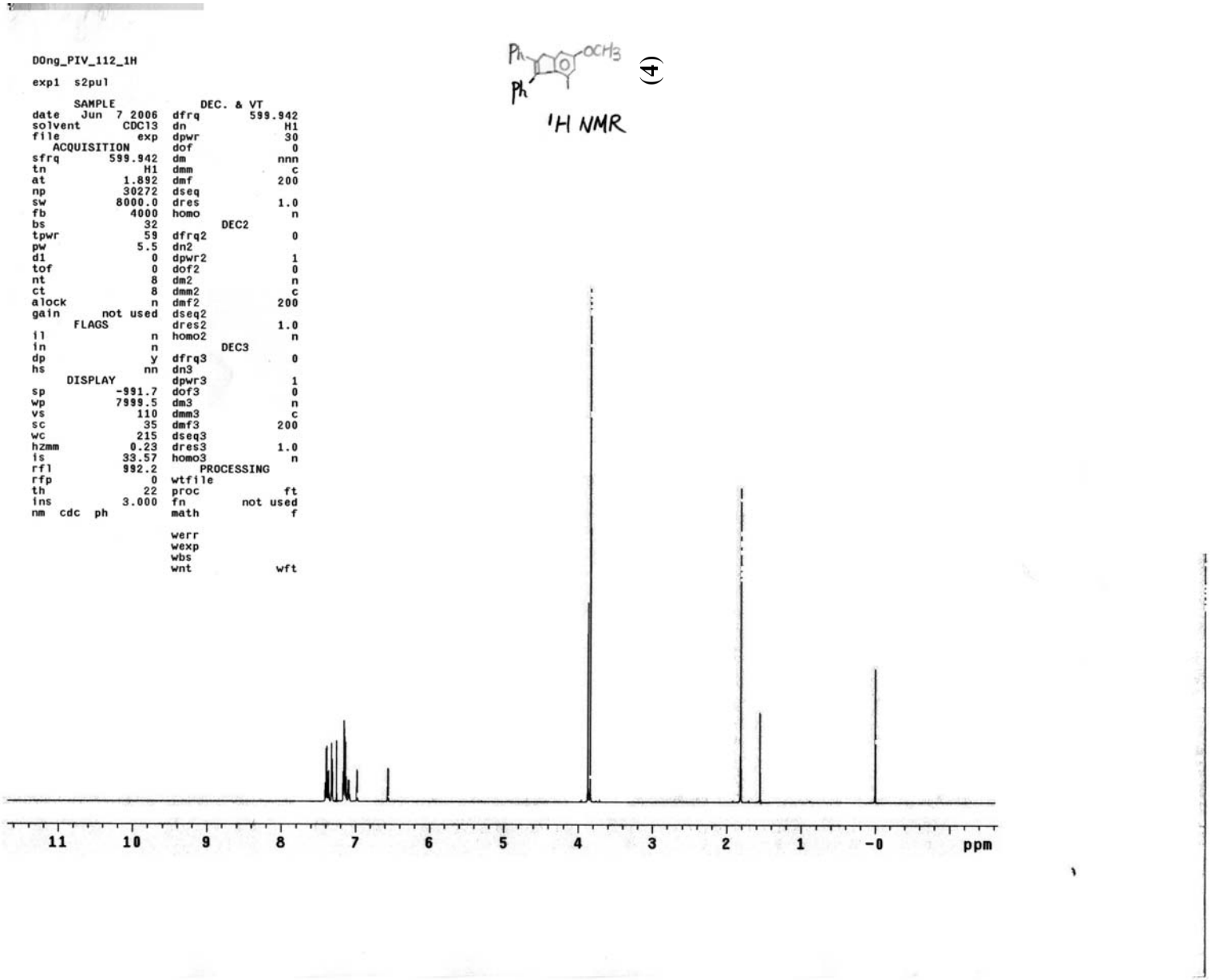
SAMPLE		DEC. & VT	
date	May 5 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	16667
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	DEC2	
tpwr	61	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	3017.4	dof2	0
nt	10000	dm2	n
ct	2632	dmm2	c
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-875.7	dof3	0
wp	34706.6	dm3	n
vs	73	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	16.65	dres3	1.0
is	500.00	homo3	n
rfl	12492.1	PROCESSING	
rfp	11615.8	wtfile	
th	4	proc	ft
ins	100.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	

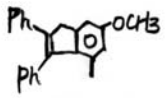


(3)

^{13}C NMR




DOng_PIV_112_1H
exp1 s2pul
SAMPLE
date Jun 7 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 32
tpwr 59
pw 5.5
d1 0
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -991.7
wp 7999.5
vs 110
sc 35
wc 215
hzmm 0.23
is 33.57
rfl 992.2
rfp 0
th 22
ins 3.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
Ph
OCH3
Ph
(4)
1H NMR
11
10
9
8
7
6
5
4
3
2
1
-0
ppm



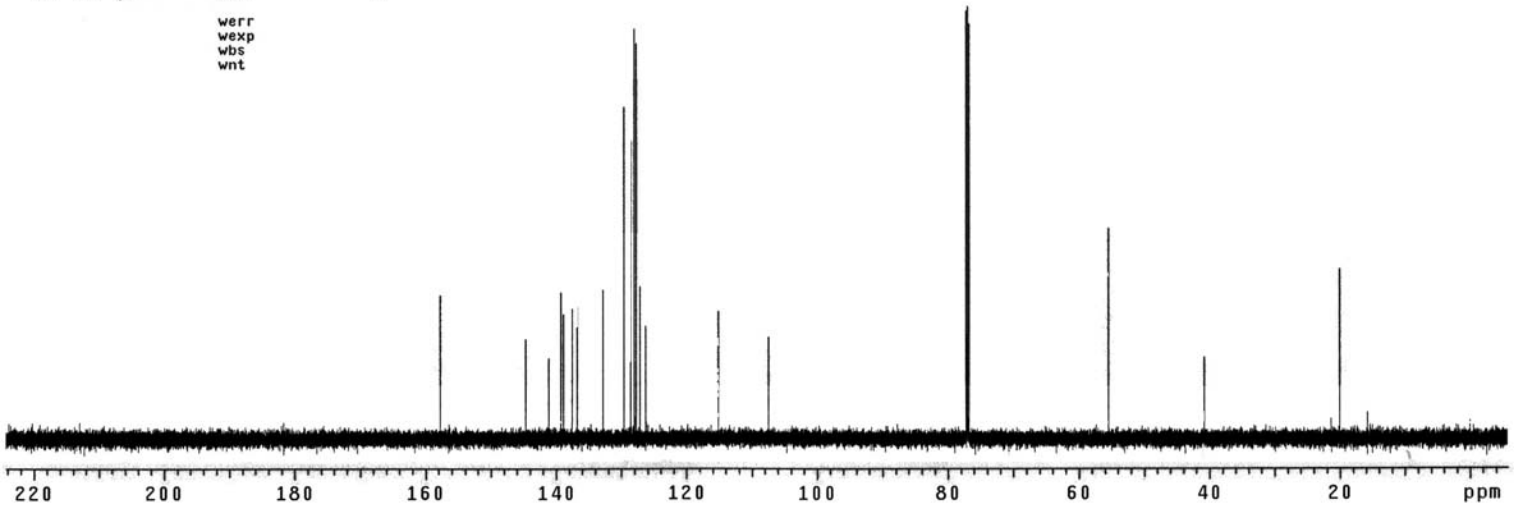
(4)

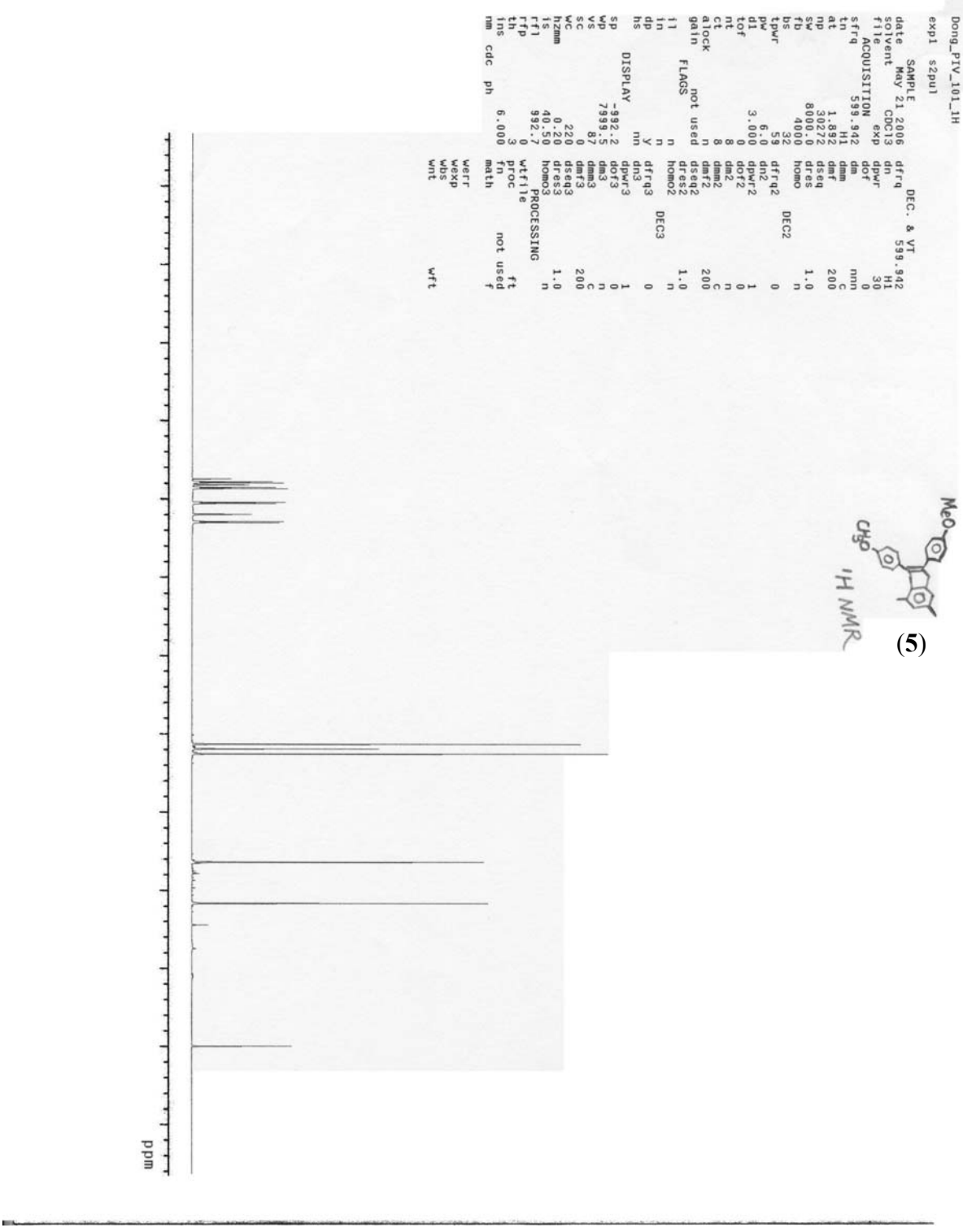
^{13}C NMR

Dong_PIV_112_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 7 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	16667
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	DEC2	
tpwr	61	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	3017.4	dof2	0
nt	30000	dm2	n
ct	464	dmm2	c
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-875.6	dof3	0
wp	34706.6	dm3	n
vs	71	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	27.48	dres3	1.0
is	500.00	homo3	n
rfl	12491.9	PROCESSING	
rfp	11615.8	wtfile	
th	8	proc	ft
ins	100.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	



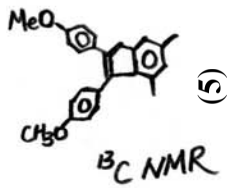

Dong_PIV_101_1H
exp1 s2pul
SAMPLE
date May 21 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 599.942 dfrq DEC. & VT
tn H1 dn 599.942
at 1.892 dmf H1
np 30272 dseq 30
sw 8000.0 dres 1.0 ni
fb 4000 homo
bs 32 DEC2
tpwr 59 dfrq2 0
pw 6.0 dn2
d1 3.000 dpwr2 1
tof 0 dof2 0
nt 8 dm2 n
ct 8 dmf2 c
alock n dmf2 200
gain FLAGS not used dseq2
dres2 1.0
ll n homo2 n
in n DEC3
dp y dfrq3 0
hs nn dn3
DISPLAY dpwr3 1
sp -992.2 dof3 0
wp 7999.5 dm3 n
vs 87 dmm3 c
sc 0 dmf3 200
wc 220 dseq3
hzmm 0.20 dres3 1.0
is 40.50 homo3 n
rfl 992.7 PROCESSING
rfp 0 wtfile
th 3 proc ft
ins 6.000 fn not used
nm cdc ph math f
werr
wexp
wbs
wnt wft
MeO
CHO
1H NMR
ppm

(5)

Dong_PIV_101_13C

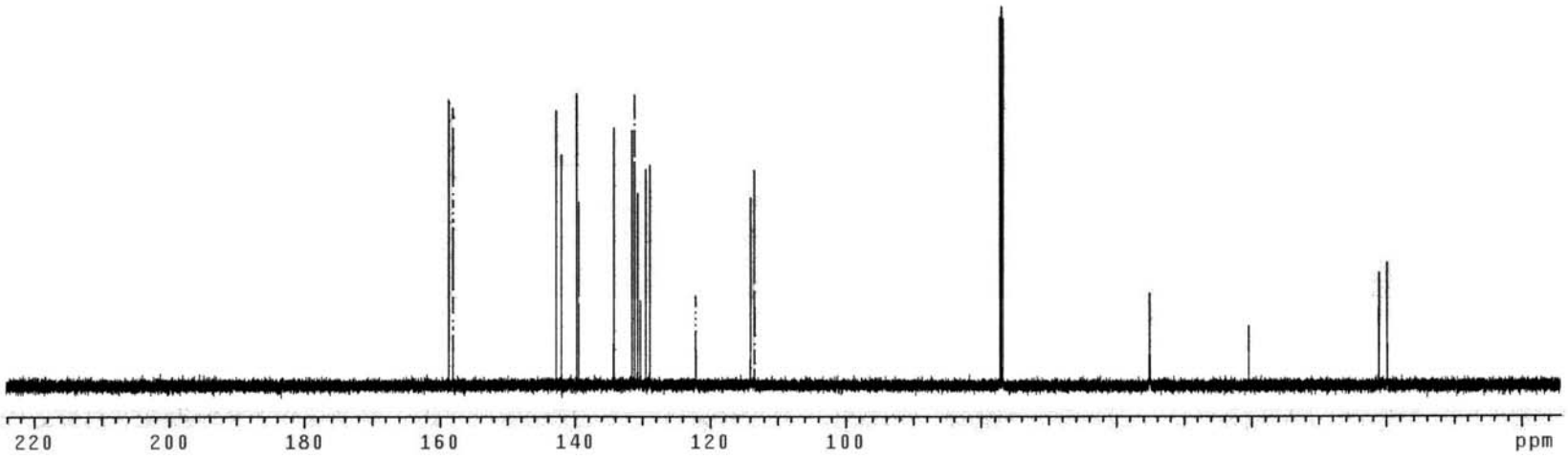
exp2 s2pul

SAMPLE		DEC	
date	May 21 2006	dfrq	599.942
solvent	CDCl3	dn	
file	exp	dpwr	
ACQUISITION		dof	
sfrq	150.871	dm	
tn	C13	dmm	
at	1.300	dmf	
np	90272	dseq	
sw	34707.2	dres	
fb	19000	homo	
bs	8	DEC2	
tpwr	61	dfrq2	
pw	6.0	dn2	
d1	000	dpwr2	
tof	3017.4	dof2	
nt	30000	dm2	
ct	992	dmm2	
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	
		homo2	
in		DEC3	
dp		dfrq3	
		dn3	
DISPLAY		dpwr3	
	-886.2	dof3	
wp	34706.6	dm3	
	60	dmm3	
	0	dmf3	16667
	250	dseq3	
	138.83	dres3	1.0
	500.00	homo3	
	12502.6	PROCESSING	
	11615.8	wtfile	
		proc	
	100.000	fn	not used
nm	ph		
		werr	
		wexp	
		wbs	



(5)

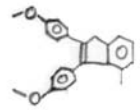
^{13}C NMR



Dong_VIV_P4_G1_1H

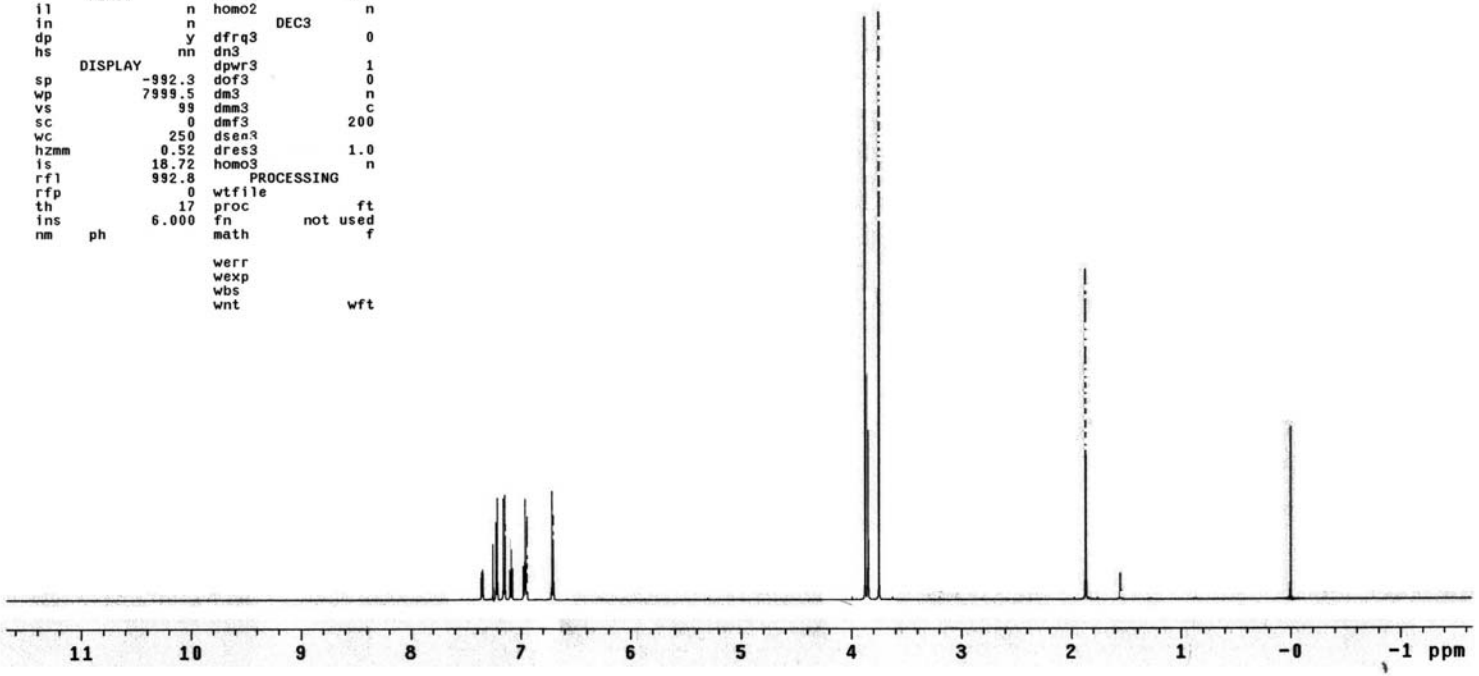
exp1 s2pul

SAMPLE		DEC. & VT	
date	Jun 21 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.942	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	58	dfrq2	0
pw	5.5	dn2	
d1	0	dpwr2	1
tof	0	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-992.3	dof3	0
wp	7999.5	dm3	n
vs	99	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	0.52	dres3	1.0
is	18.72	homo3	n
rfl	992.8	PROCESSING	
rfp	0	wtfile	
th	17	proc	ft
ins	6.000	fn	not used
nm ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft

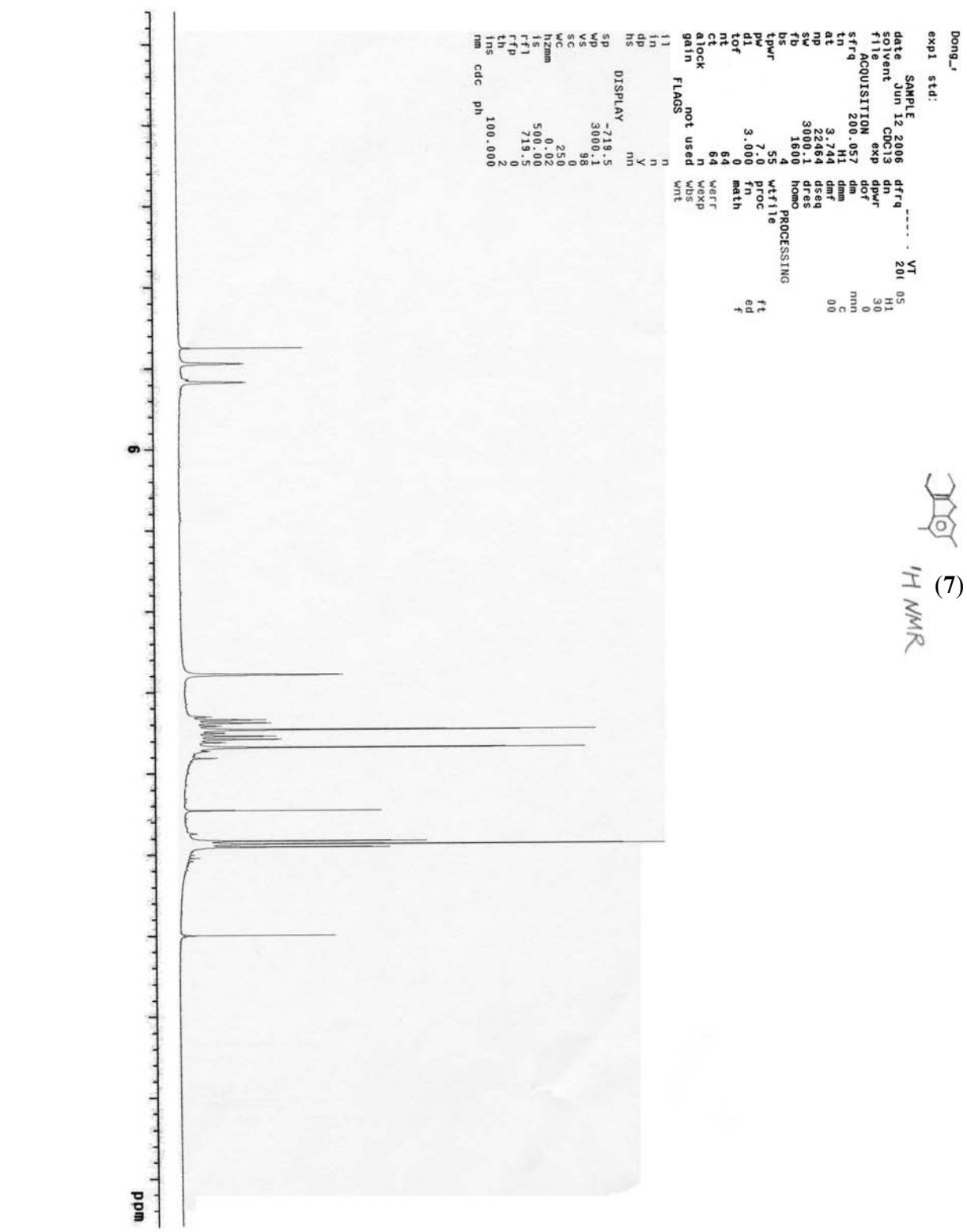


(9)

^1H NMR



Dong_VIV_P4_G1_13C
exp2 s2pul
SAMPLE
date Jun 21 2006
solvent CDCl3
file
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 61
pw 6.0
d1 3.000
tof 3017.4
nt 5000
ct 752
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -878.6
wp 34706.6
vs 65
sc 0
wc 250
hzmm 1.26
is 500.00
rfl 12494.9
rfp 11615.8
th 8
ins 100.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 39
dof 0
dm nny
dmm w
dmf 16667
dseq
dres
homo
DEC2
dfrq2
dn2
dpwr2
dof2
dm2
dmm2
dmf2
dseq2
dres2
homo2
DEC3
dfrq3
dn3
dpwr3
dof3
dm3
dmm3
dmf3
dseq3
dres3
homo3
PROCESSING
wtfile
proc
fn not used
math
werr
wexp
wbs
wnt
ft
f
(6)
13C NMR
220
200
180
160
140
120
100
80
60
40
20
ppm


Dong_1
expl std1
SAMPLE
date Jun 12 2006
solvent CDCl3
file
ACQUISITION
sfrq 200.057 H1
tn H1
at 3.744
np 22464
sw 3000.1
fb 1600
bs 4
tpwr 55
pw 7.0
d1 3.000
tof 0
nt 64
ct 64
alock n
gain n
FLAGS not used
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
PROCESSING
wtfile
proc
fn
math
werr
wexp
wbs
wnt
VT
201
ft
ed
f
DISPLAY
sp -719.5
wp 3000.1
vs 98
sc 0
wc 250
hzmm 0.02
is 500.00
rfl 719.5
rfp 0
th 2
ins
nm cdc ph 100.000
6
ppm

(7)

^1H NMR

Dong_PIV_110_13C
exp2 s2pul
SAMPLE
date Jun 14 2006
solvent CDCl3
file
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 54
pw 6.0
d1 3.000
tof 3017.4
nt 10000
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -873.6
wp 34706.6
vs 153
sc 0
wc 250
hzmm 138.83
is 500.00
rfl 12490.0
rfp 11615.8
th 6
ins 100.000
nm cdc ph
werr
wexp
wbs
wnt
dfrq DEC. & VT 599.942
dn H1
dof 39
dm 0
dmm nny
dmf w
dseq 16667
dres 1.0
homo n
dfrq2
dn2
dpwr2
dof2 0
dm2 n
dmm2 c
dmf2 16667
dseq2
dres2 1.0
homo2 n
DEC2
dfrq3
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 16667
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
DEC3
0
200
180
160
140
120
100
20
ppm

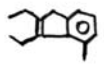
^{13}C NMR

(7)

DOng_PIV_120_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jun 19 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.942	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	32	DEC2	
tpwr	58	dfrq2	0
pw	5.5	dn2	
d1	0	dpwr2	1
tof	0	dof2	0
nt	8	dm2	n
ct	8	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-991.2	dof3	0
wp	7999.5	dm3	n
vs	104	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	0.24	dres3	1.0
is	102.67	homo3	n
rfl	991.7	PROCESSING	
rfp	0	wtfile	
th	2	proc	ft
ins	1.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



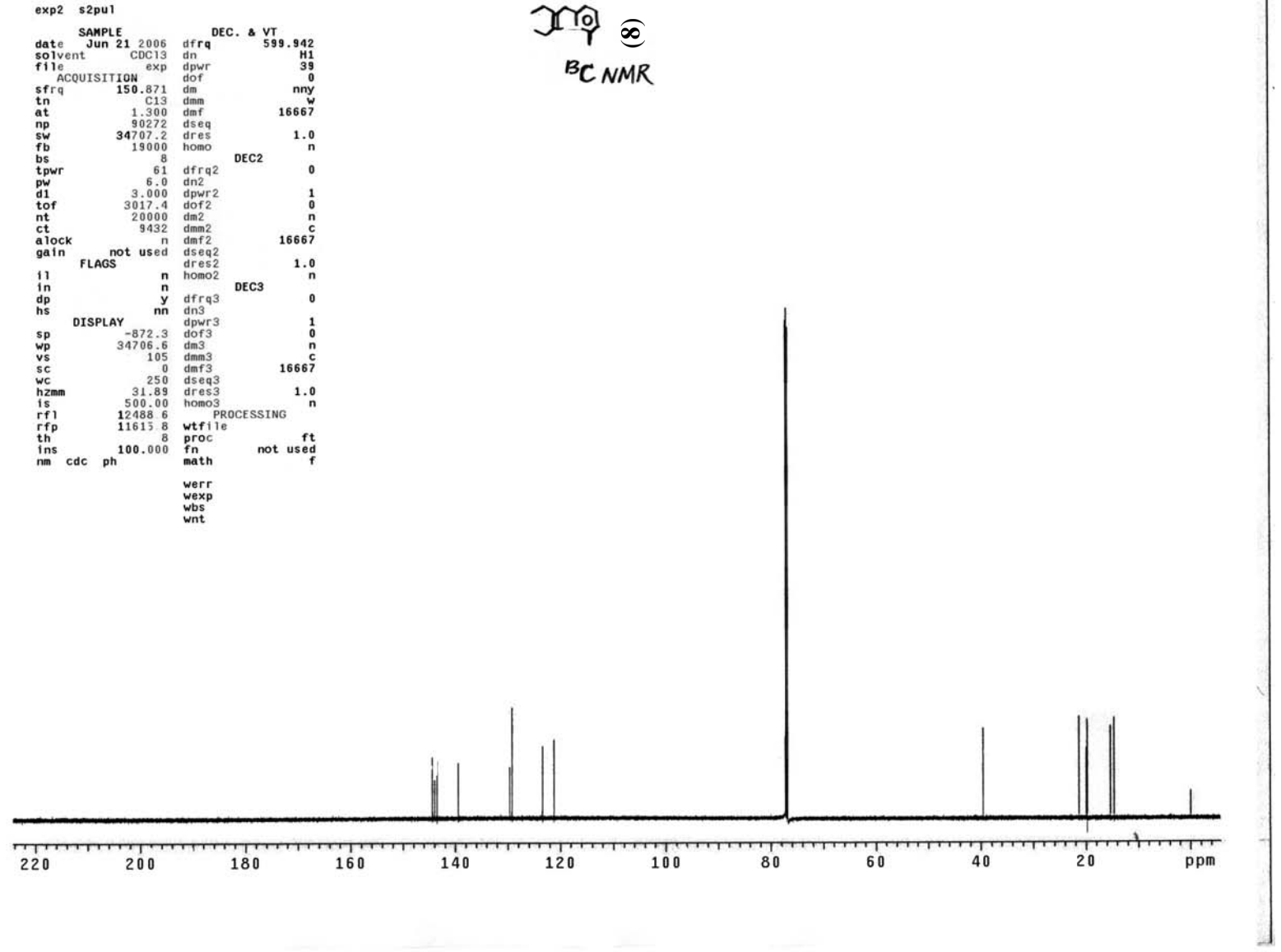
1H NMR

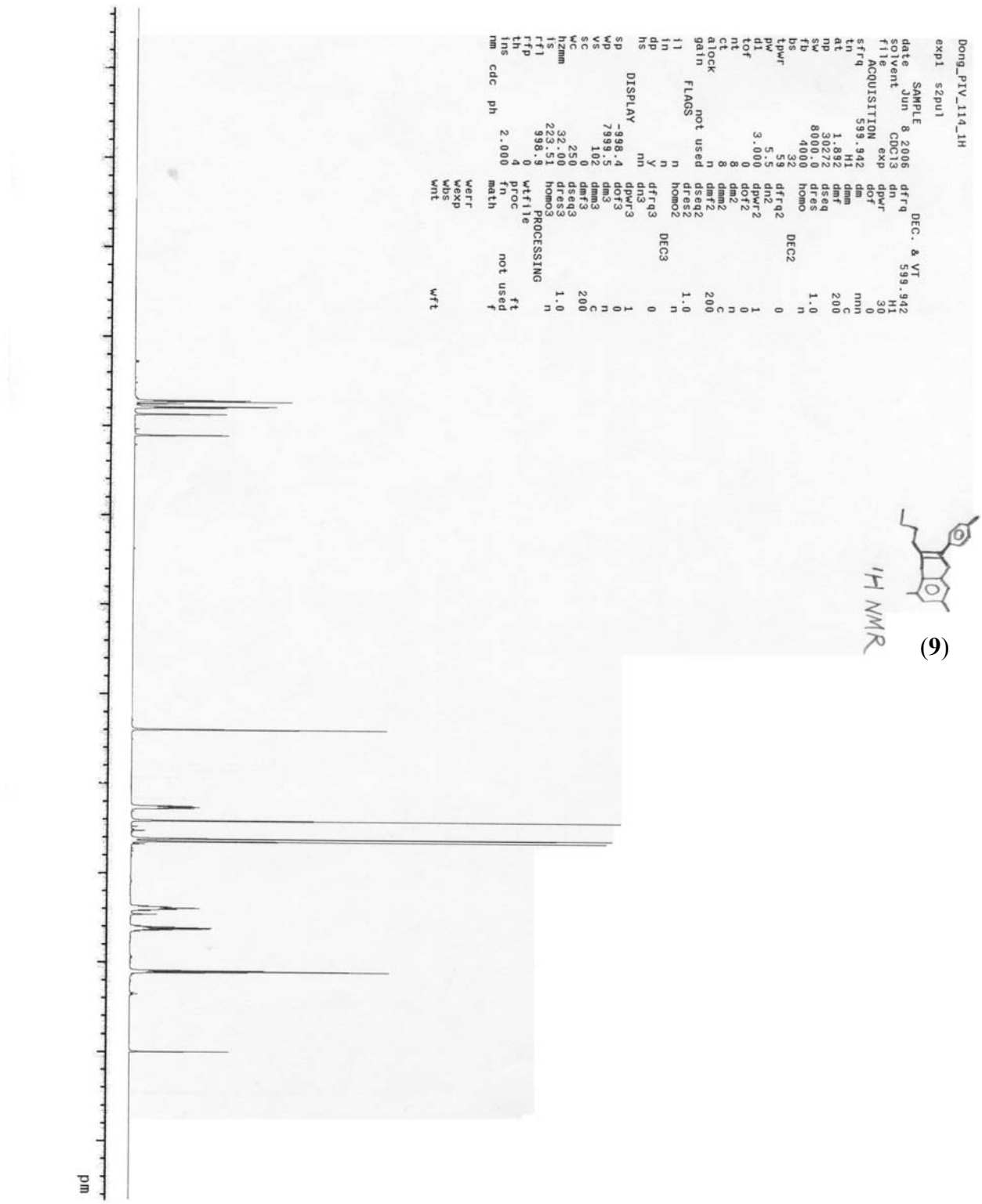
(8)

Dong_PIV_120_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 21 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	16667
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	DEC2	
tpwr	61	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	3017.4	dof2	0
nt	20000	dm2	n
ct	9432	dmm2	c
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-872.3	dof3	0
wp	34706.6	dm3	n
vs	105	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	31.89	dres3	1.0
is	500.00	homo3	n
rfl	12488.6	PROCESSING	
rfp	11615.8	wtfile	
th	8	proc	ft
ins	100.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	



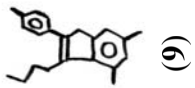

Dong_PIV_114_1H
expl s2pul
SAMPLE
date Jun 8 2006
solvent CDCl3
file
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 32
tpwr 59
pw 5.5
d1 3.000
tof 0
nt 8
ct 8
alock n
gain n
FLAGS
not used
n
il n
in n
dp y
hs nn
DISPLAY
sp -998.4
wp 7999.5
vs 102
sc 0
wc 250
hzmm 32.00
is 223.51
rfl 998.9
rfp 0
th 4
ins 2.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres
homo 1.0
n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2
homo2 1.0
n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
pm
1H NMR

(**9**)

Dong_PIV_114_13C

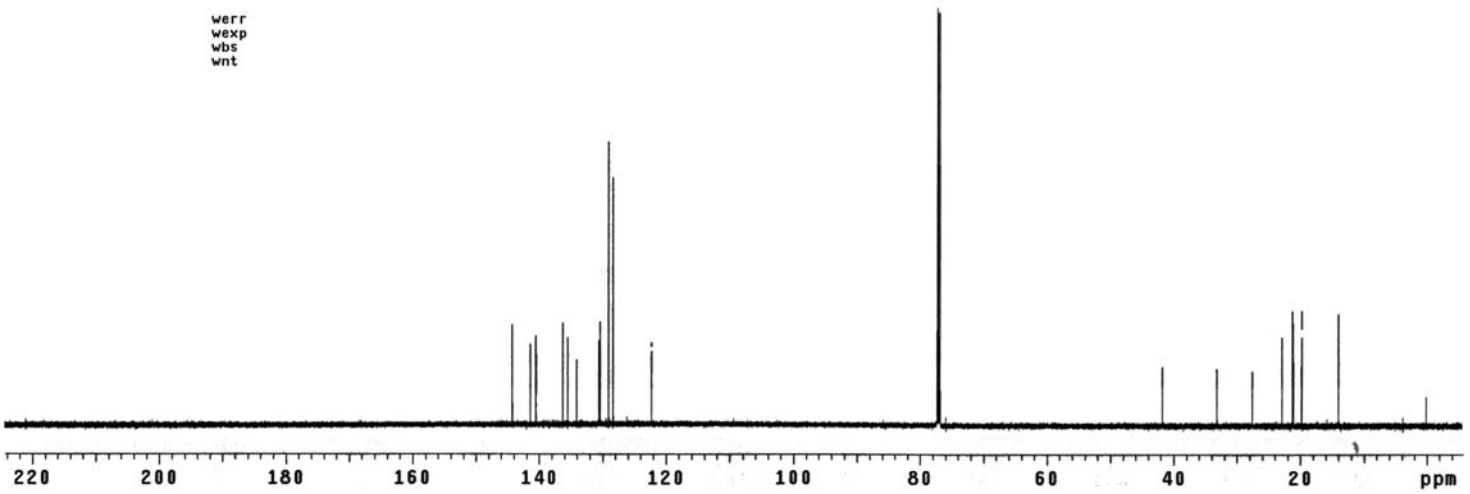
exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 8 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	16667
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	DEC2	
tpwr	61	dfrq2	0
pw	6.0	dn2	
d1	3.006	dpwr2	1
tof	3017.4	dof2	0
nt	30000	dm2	n
ct	8920	dmm2	c
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-872.6	dof3	0
wp	34706.6	dm3	n
vs	71	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	138.83	dres3	1.0
is	500.00	homo3	n
rfl	12488.9	PROCESSING	
rfp	11615.8	wtfile	
th	7	proc	ft
ins	100.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	



(9)

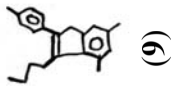
^{13}C NMR



Dong_PIV_114_noesy1D

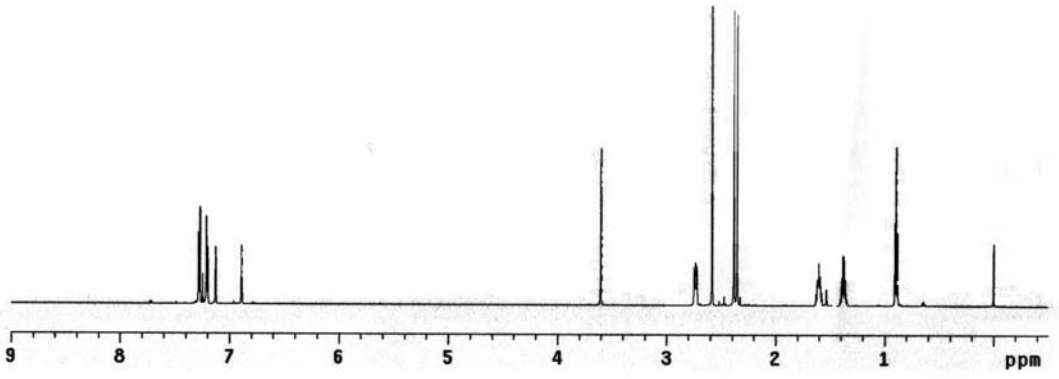
Pulse Sequence: noesy1D_da
Solvent: CDCl3
Ambient temperature
INOVA-600 "u600"

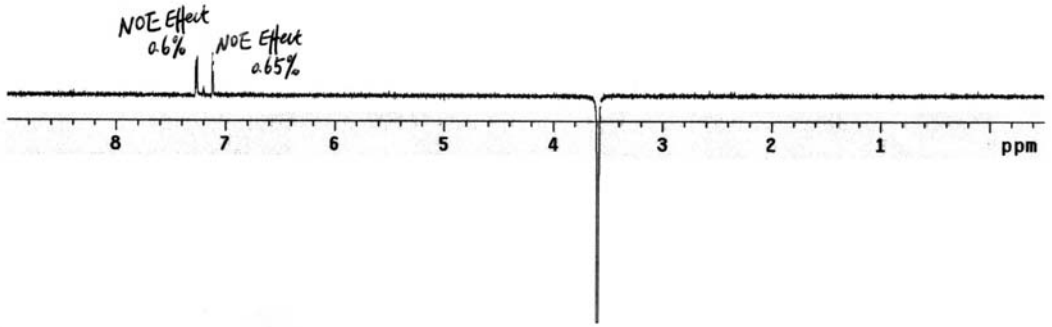
Relax delay 2.000 sec
Pulse 90.0 degrees
Mixing 0.400 sec
Acq. time 1.892 sec
Width 8000.0 Hz
160 repetitions
OBSERVE H1, 599.9393058 MHz
DATA PROCESSING
FT size 32768
Total time 29 min, 25 sec

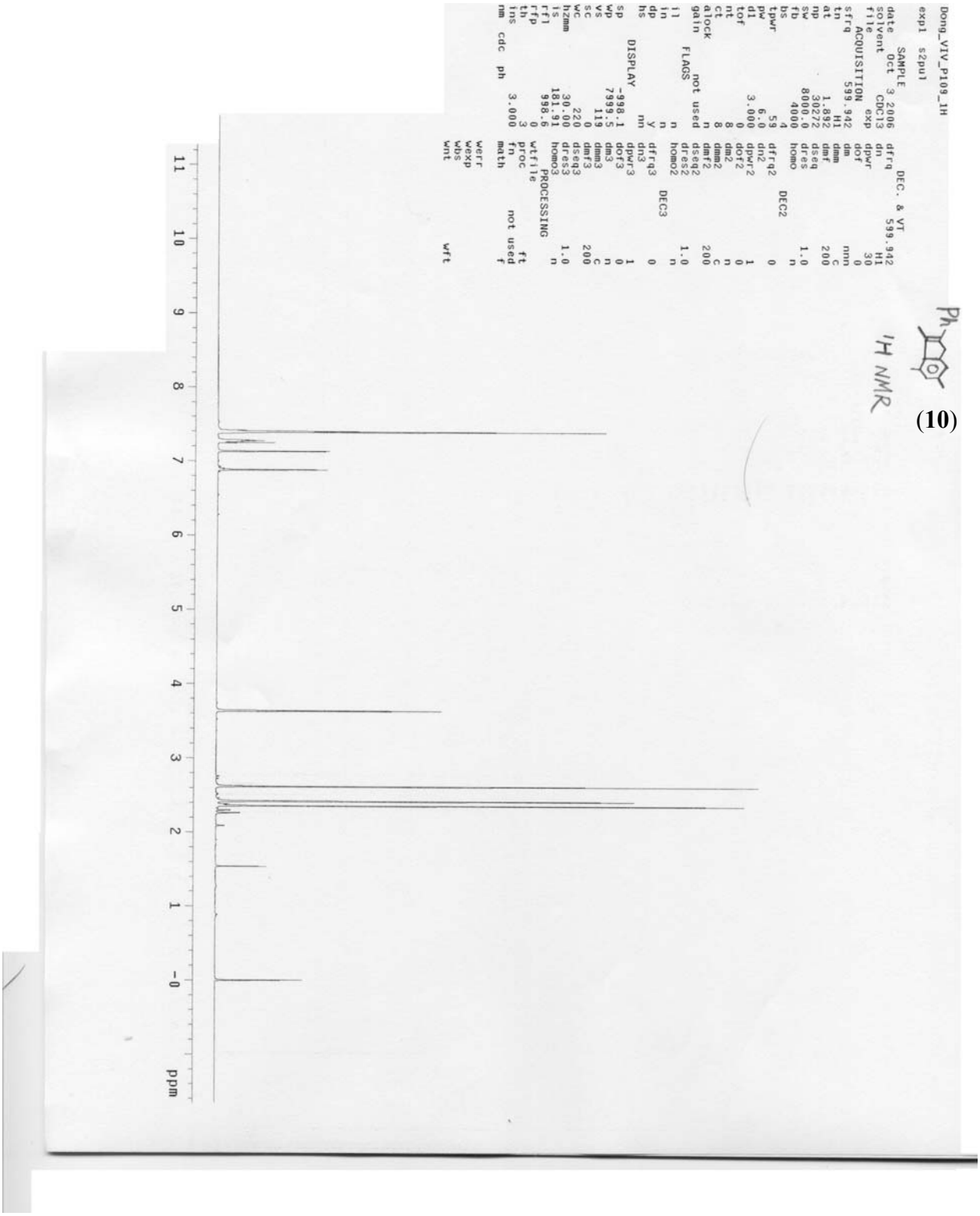


(9)

NOE Experiment



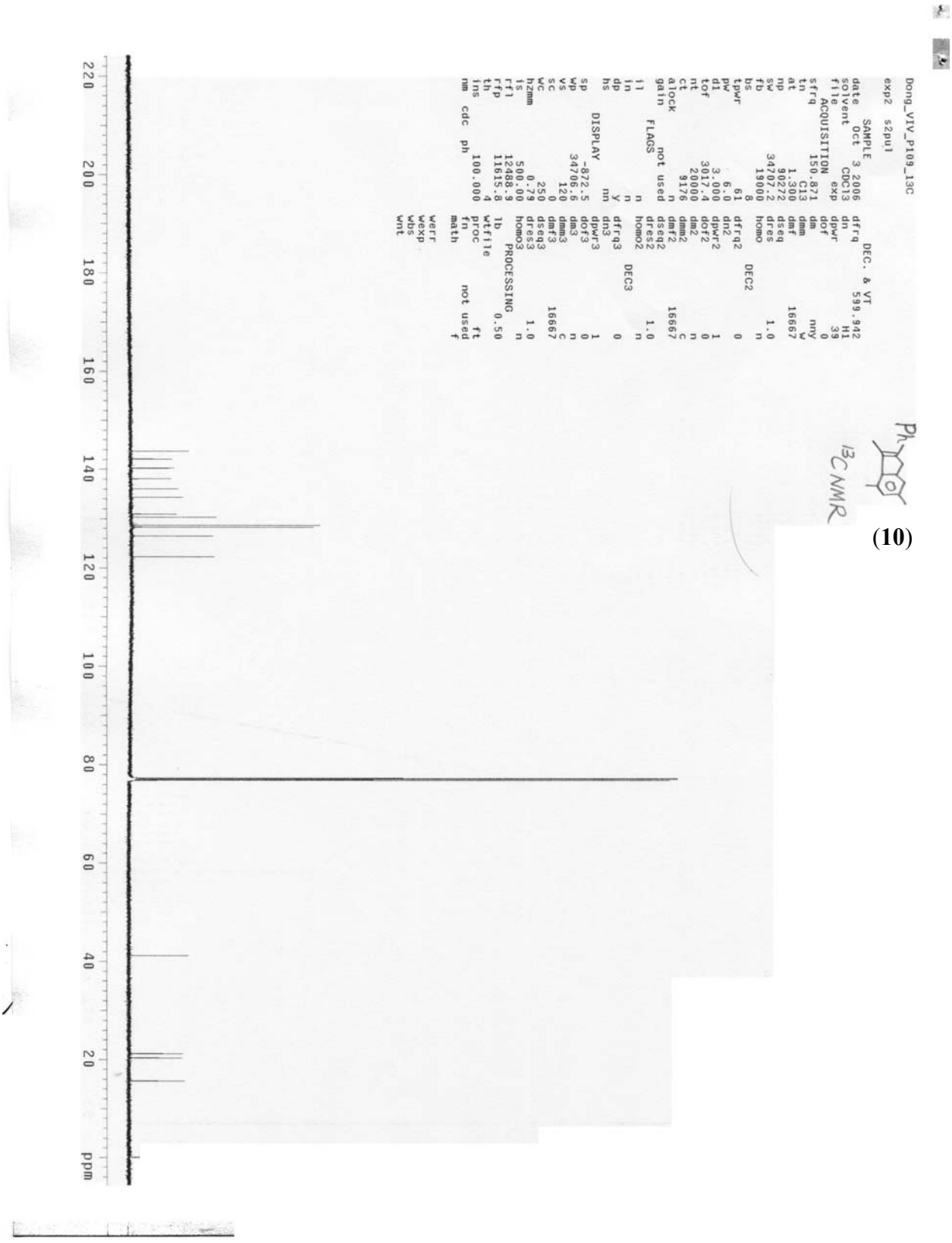



Dong_VIV_P109_1H
exp1 s2pul
SAMPLE
date Oct 3 2006
solvent CDCl3
file
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 4
tpwr 59
pw 6.0
d1 3.000
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
i1 n
in n
dp y
hs nn
DISPLAY
sp -998.1
wp 7999.5
vs 119
sc 0
wc 220
hzmm 30.00
is 181.91
rfl 998.6
rfp 0
th 3
ins 3.000
nm cdc ph
dfrq DEC. & VT 599.942
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
11 10 9 8 7 6 5 4 3 2 1 -0 ppm

Ph

1H NMR

(10)

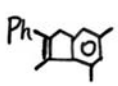

Dong_VIV_P109_13C
exp2 s2pul
SAMPLE
date Oct 3 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 61
pw 6.0
pl1 3.00
tof 3017.4
nt 20000
ct 9176
alock n
gain n
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -872.5
wp 34706.6
vs 120
sc 0
wc 250
hzmm 0.79
is 500.00
rfl 12488.9
rfp 11615.8
th 4
ins 100.000
nm cdc ph
dfrq DEC. & VT
dn H1
dpwr 39
dof 0
dm nny
dmm y
dmf 16667
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 16667
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 16667
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Ph
O
13C NMR
220
200
180
160
140
120
100
80
60
40
20
ppm

(10)

Dong_VIV_P109_noesy1D

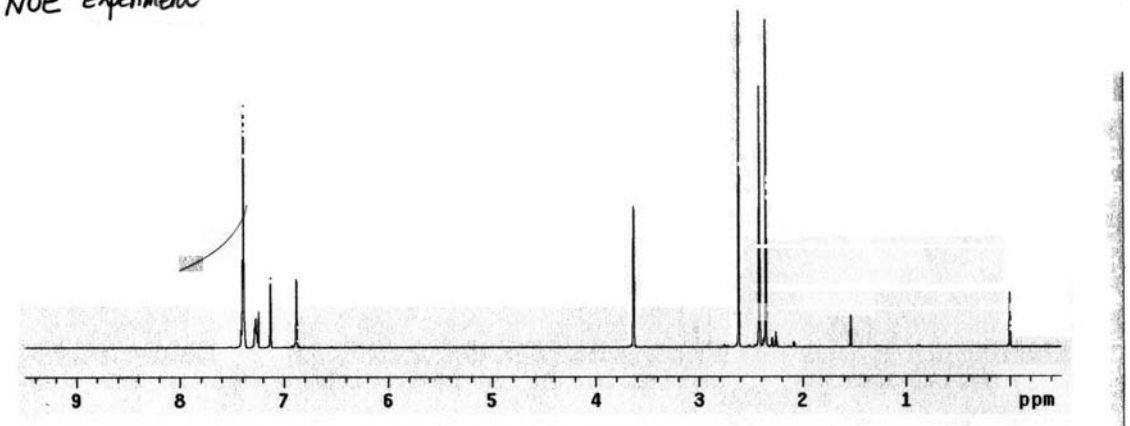
Pulse Sequence: noesy1D_da
Solvent: CDCl3
Ambient temperature
INOVA-600 "u600"

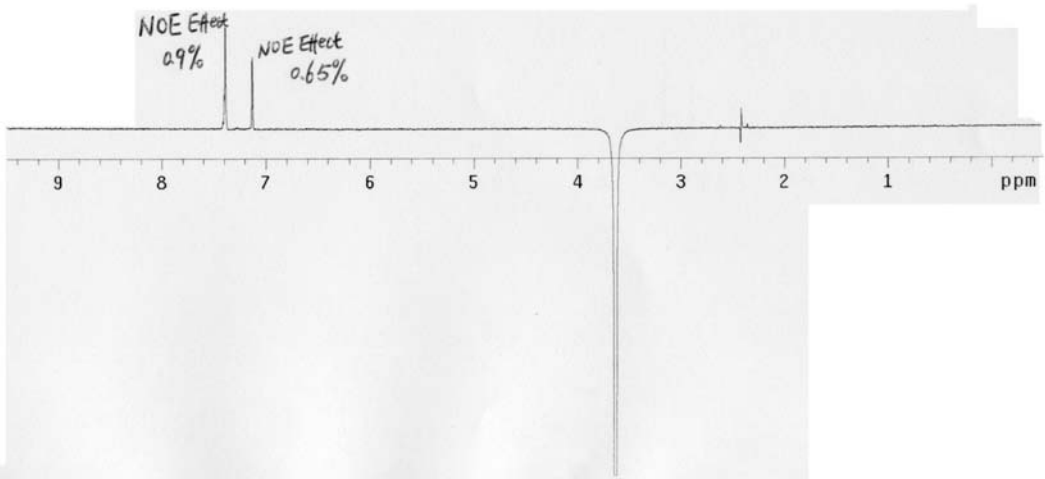
Relax. delay 2.000 sec
Pulse 90.0 degrees
Mixing 0.400 sec
Acq. time 1.892 sec
Width 8000.0 Hz
248 repetitions
OBSERVE H1, 599.9393022 MHz
DATA PROCESSING
Line broadening 0.7 Hz
FT size 32768
Total time 1 hr, 19 min, 0 sec



(10)

NOE Experiment





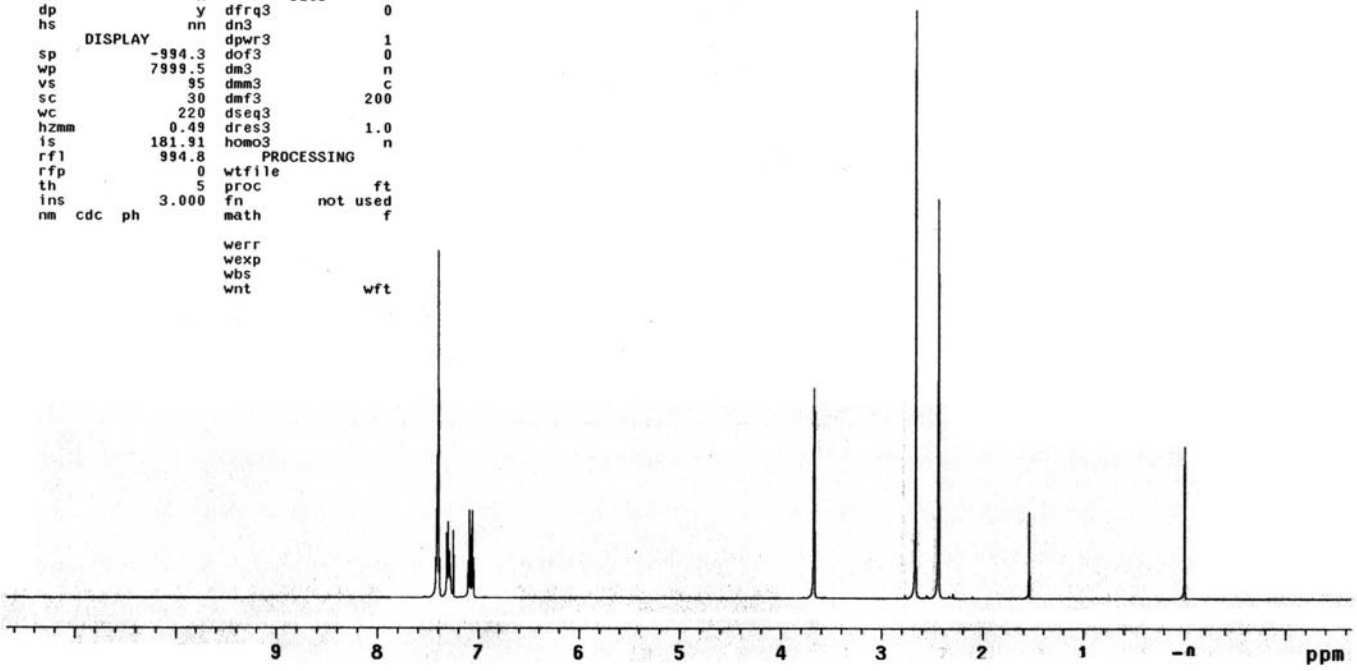
Dong_VIV_P105_

exp1 s2pul

SAMPLE		DEC. & VT	
date	Oct 3 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.942	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	59	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	0	dof2	0
nt	8	dm2	n
ct	8	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-994.3	dof3	0
wp	7999.5	dm3	n
vs	95	dmm3	c
sc	30	dmf3	200
wc	220	dseq3	
hzmm	0.49	dres3	1.0
is	181.91	homo3	n
rfl	994.8	PROCESSING	
rfp	0	wtfile	
th	5	proc	ft
ins	3.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft

(11)

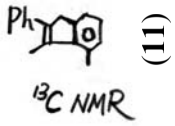
^{1}H NMR



Dong_VIV_P105_13C

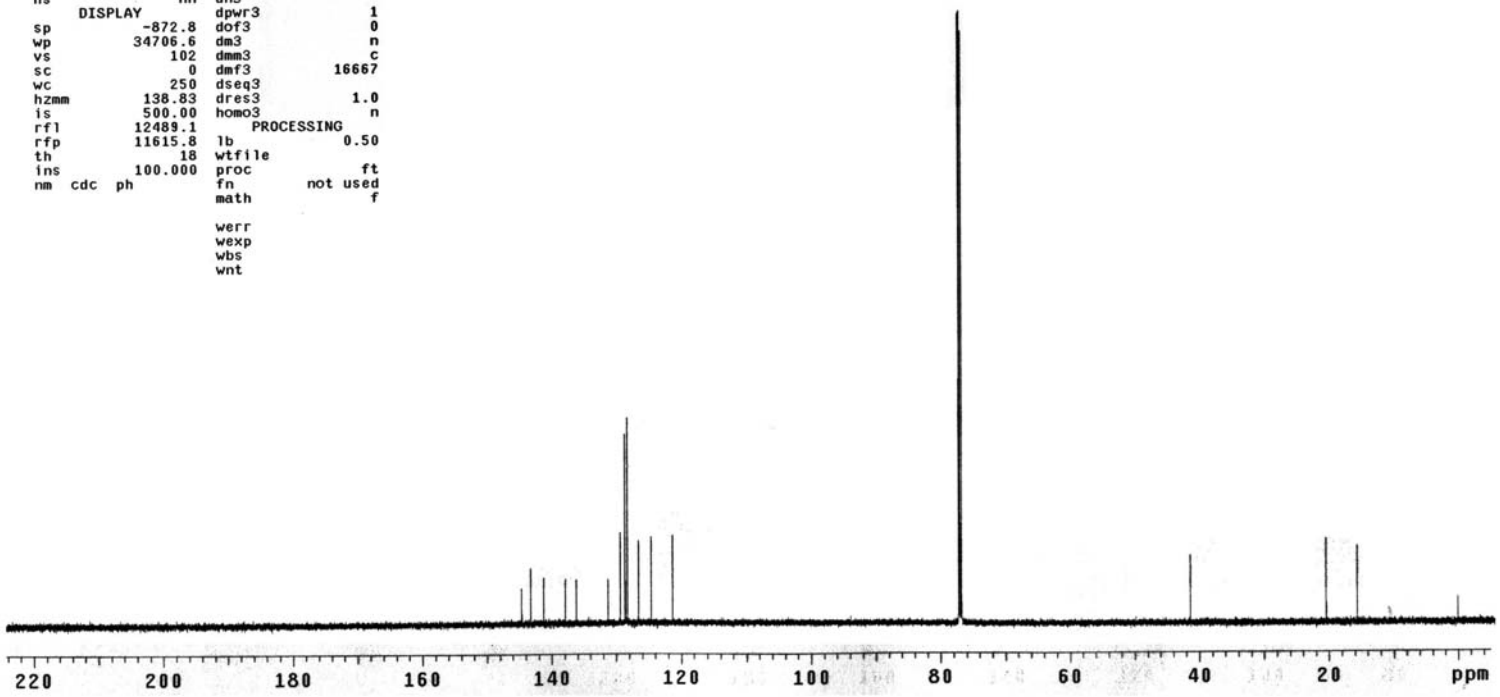
exp2 s2pul

SAMPLE		DEC. & VT	
date	Oct 3 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	16667
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	DEC2	
tpwr	61	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	3017.4	dof2	0
nt	10000	dm2	n
ct	2608	dmm2	c
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-872.8	dof3	0
wp	34706.6	dm3	n
vs	102	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	138.83	dres3	1.0
is	500.00	homo3	n
rfl	12489.1	PROCESSING	
rfp	11615.8	lb	0.50
th	18	wtfile	
ins	100.000	proc	ft
nm cdc ph		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



(11)

^{13}C NMR

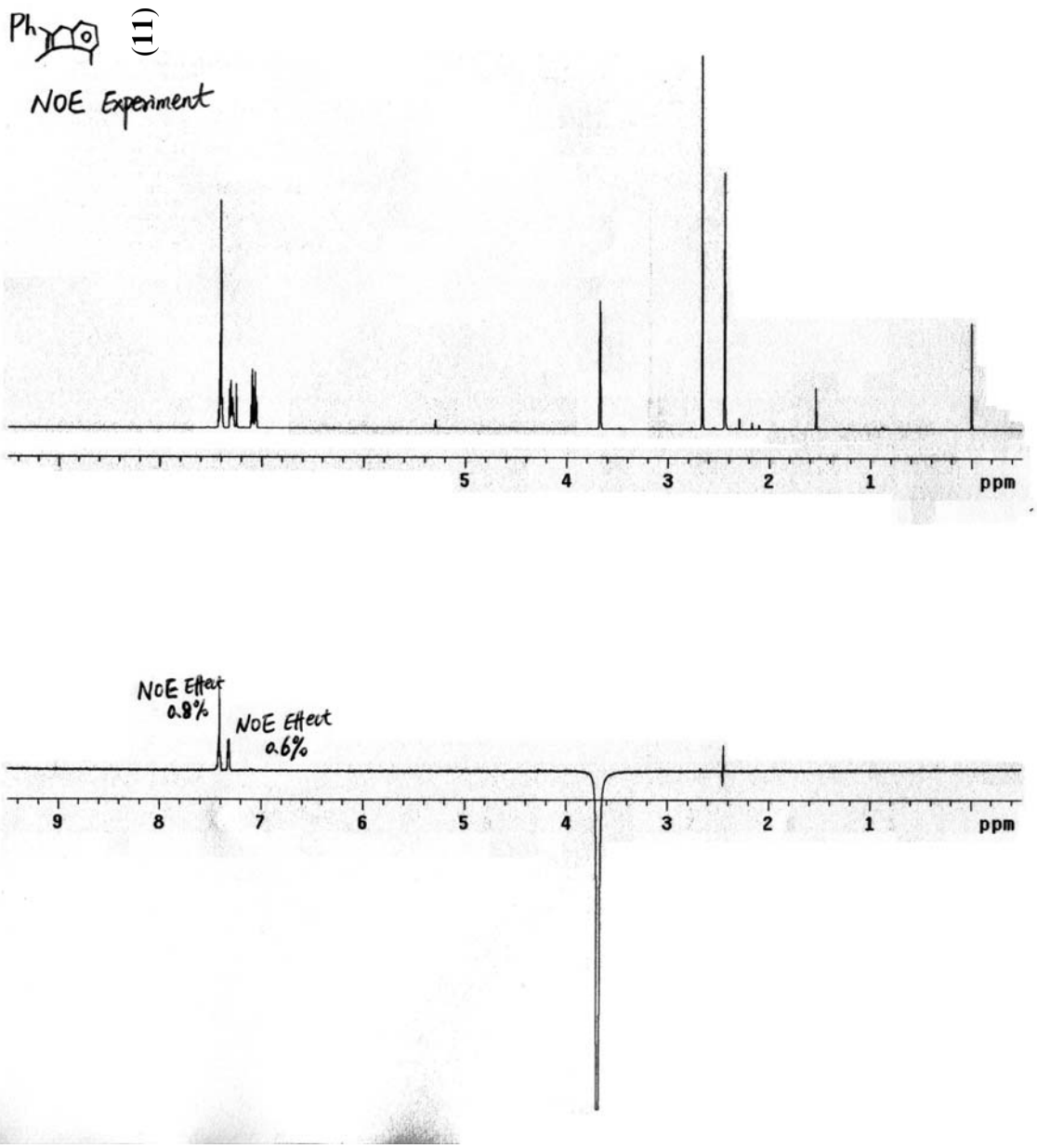


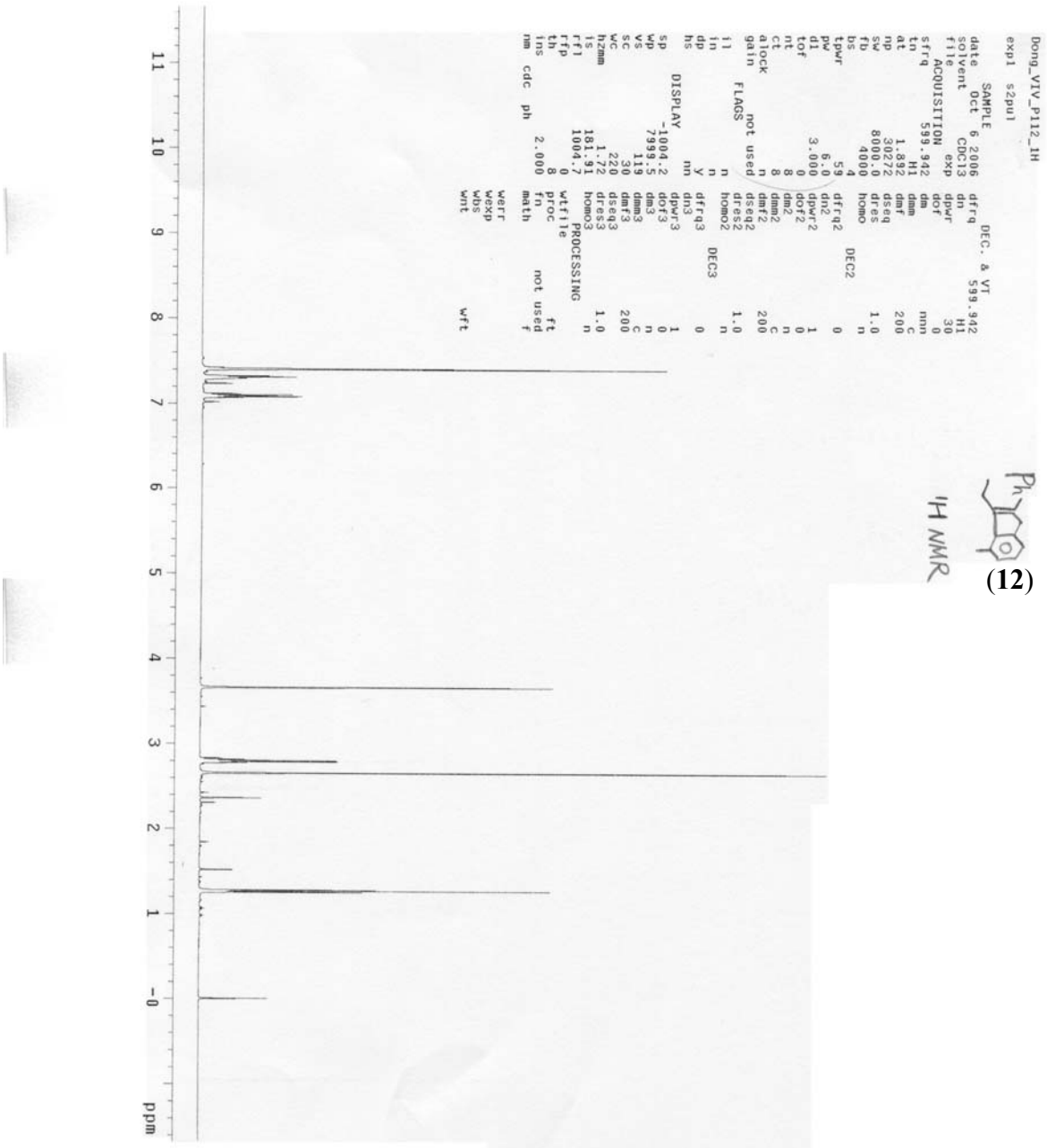
Dong_VIV_P105_noesy1D

Pulse Sequence: noesy1D_

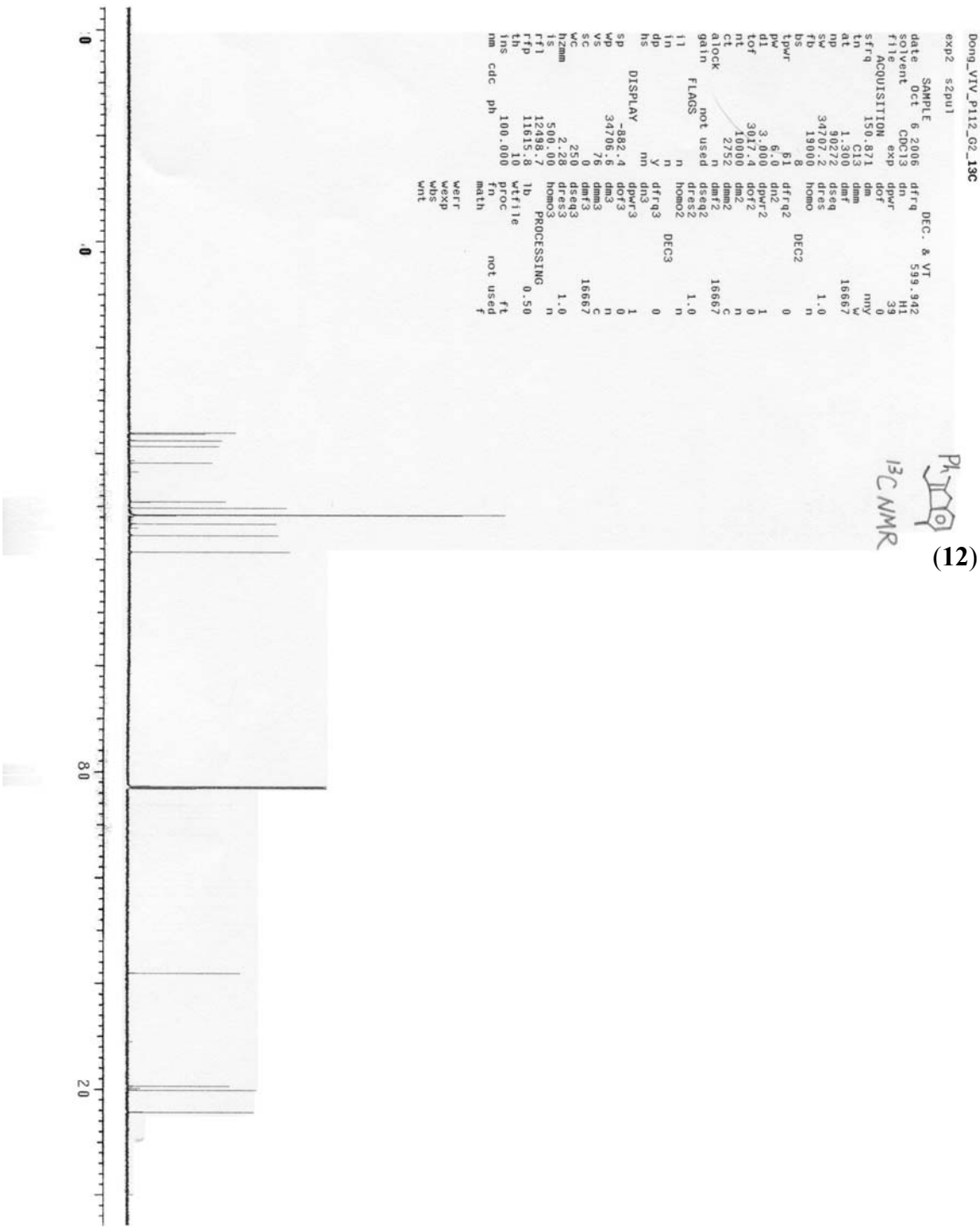
Solvent: CDCl3
Ambient temperature
INOVA-600 "u600"

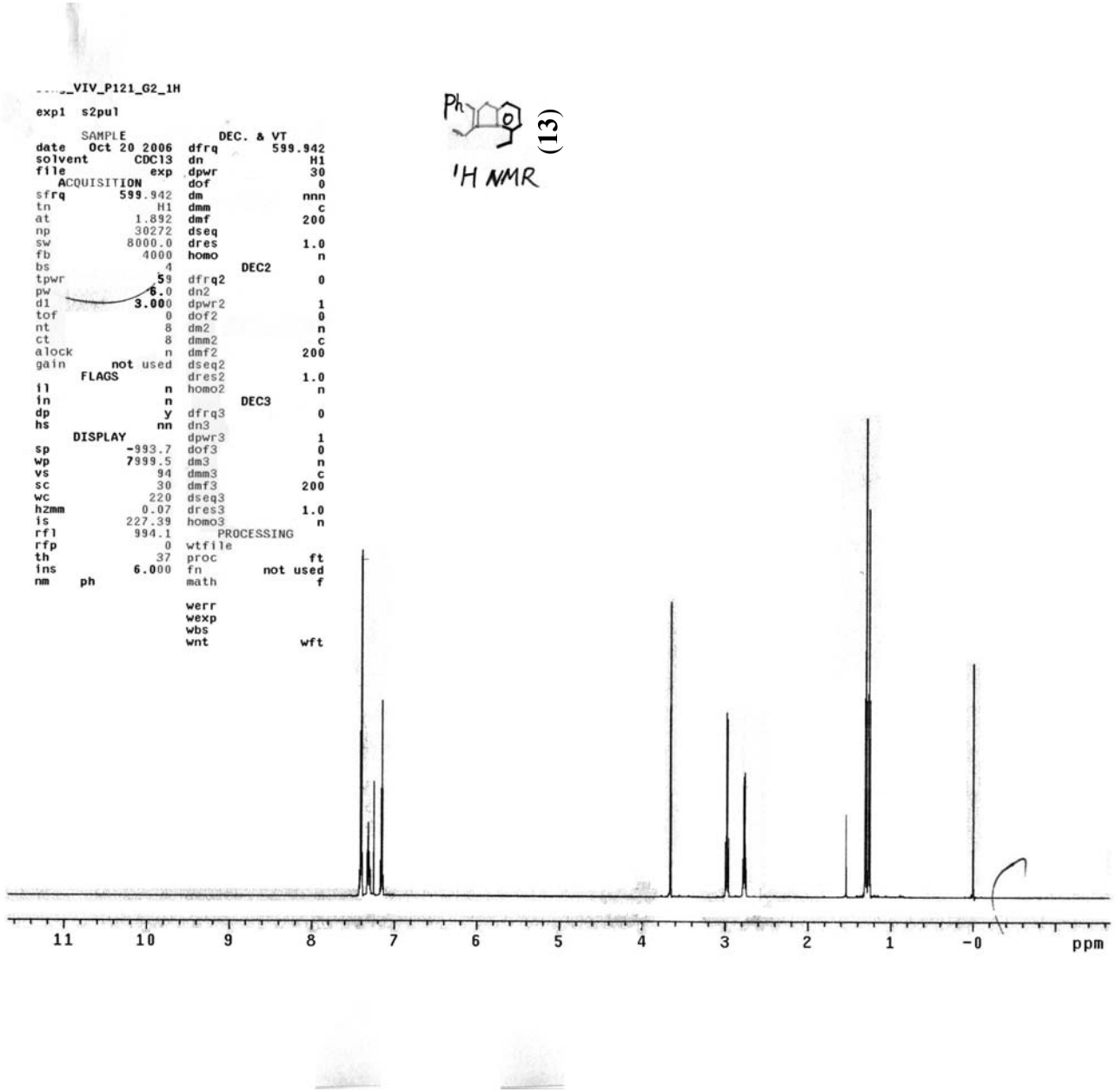
Relax. delay 2.000 sec
Pulse 90.0 degrees
Mixing 0.400 sec
Acq. time 1.892 sec
Width 8000.0 Hz
440 repetitions
OBSERVE H1, 599.9392938
DATA PROCESSING
Line broadening 0.7 Hz
FT size 32768
Total time 1 hr, 20

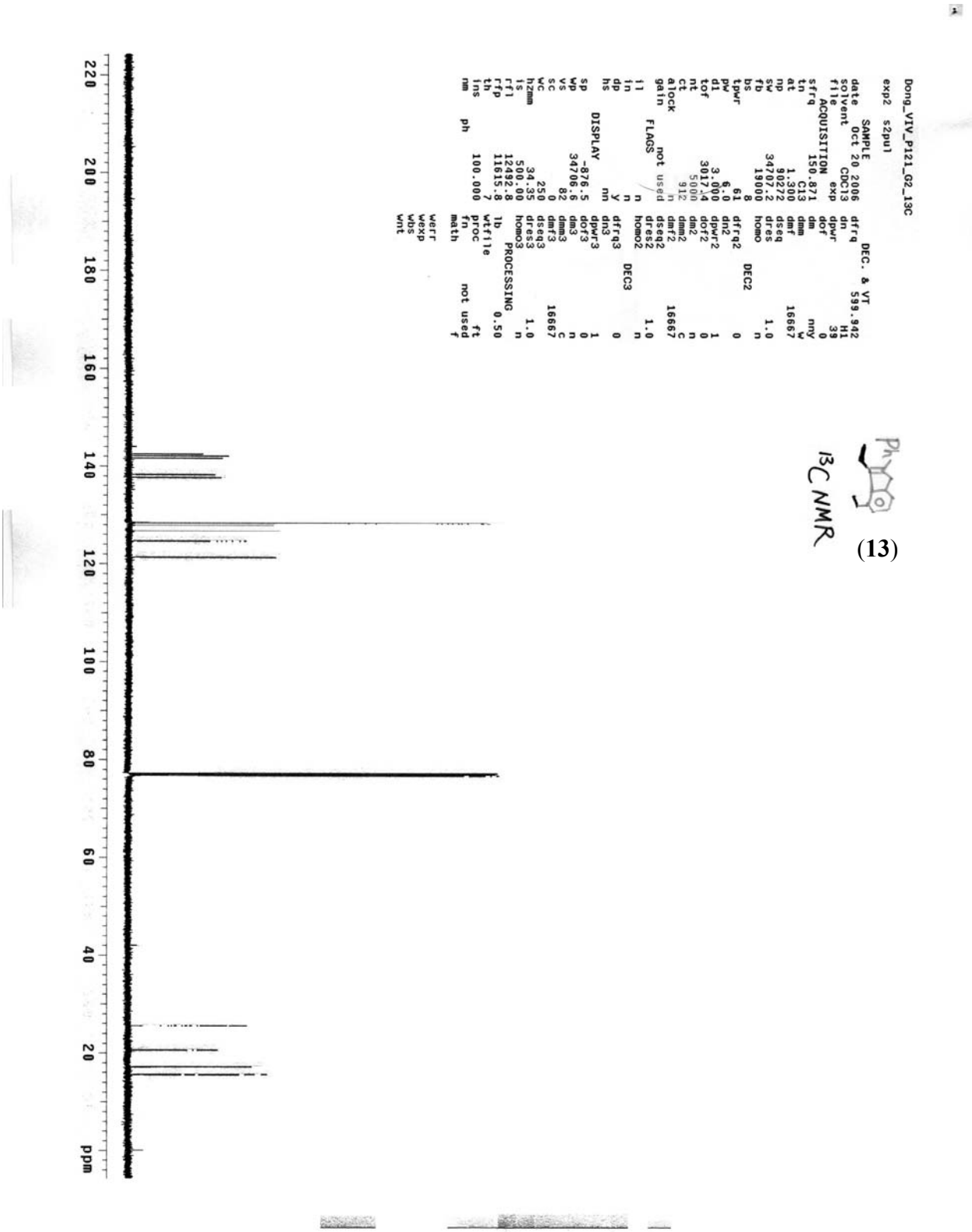



Dong_VIV_P112_1H
expl s2pul
SAMPLE
date Oct 6 2006
solvent CDCl3
file
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 4
tpwr 59
pw 6.0
d1 3.000
tof 0
nt 8
ct 8
alock n
gain n
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1004.2
wp 7999.5
vs 119
sc 30
wc 220
hzmm 1.72
is 181.91
rfl 1004.7
rfp 0
th 8
ins 2.000
nm cdc ph
dfrq DEC. & VT
dn 599.942
dpwr H1
dof 30
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
11 10 9 8 7 6 5 4 3 2 1 -0
ppm

(12)


Dong_VIV_P112_G2_13C
exp2 s2pul
SAMPLE
date Oct 6 2006
solvent CDCl3
file
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 61
pw 6.0
d1 3.000
tof 3017.4
nt 1000
ct 2752
alock n
gain 16667
FLAGS
not used
i1 n
in n
dp y
hs nn
DISPLAY
sp -882.4
wp 34706.6
vs 76
sc 250
wc 2.28
hzmm 500.00
is 12498.7
rfl 11615.8
rfp 16
th 100.000
ins
cdc ph
DEC. & VT
dfrq DEC
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo DEC2
dfrq2
dn2
dpwr2
dof2
dmm2
dmf2
dseq2
dres2
homo2 DEC3
dfrq3
dn3
dpwr3
dof3
dmm3
dmf3
dseq3
dres3
homo3
PROCESSING
lb
wtfile
proc
fn
math
599.942
H1
39
0
nny
w
16667
1.0
n
0
1
0
n
n
c
16667
1.0
n
0
1
0
n
n
c
16667
1.0
n
ft
not used
f
werr
wexp
wbs
wnt
13C NMR
(12)
0
80
20


_VIV_P121_G2_1H
exp1 s2pul
SAMPLE
date Oct 20 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 4
tpwr 59
pw 6.0
d1 3.000
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -993.7
wp 7999.5
vs 94
sc 30
wc 220
hzmm 0.07
is 227.39
rfl 994.1
rfp 0
th 37
ins 6.000
nm ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
Ph
(13)
1H NMR
11
10
9
8
7
6
5
4
3
2
1
-0
ppm


Dong_VIV_P121_02_13C
exp2 s2pul
SAMPLE
date Oct 20 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 61
pw 6.0
d1 3.000
tof 3017.4
nt 5000
ct 912
alock n
gain 39
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -876.5
wp 34706.6
vs 82
sc 0
wc 250
hzmm 34.35
is 500.00
rfl 12492.8
rfp 11615.8
th 100.000
ins 1
nm
ph
werr
wexp
wbs
wnt
DEC. & VT
dfrq 599.942
dn H1
dpwr 39
dof 0
dm nny
dmm w
dmf 16667
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2
dof2 1
dm2 0
dmm2 n
dmf2 c
dseq2 16667
dres2 1.0
homo2 n
DEC3
0
dfrq3
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 n
dmf3 c
dseq3 16667
dres3 1.0
homo3 n
PROCESSING
lb
wtfile
proc
fn
math
not used ft
not used f
220
200
180
160
140
120
100
80
60
40
20
ppm

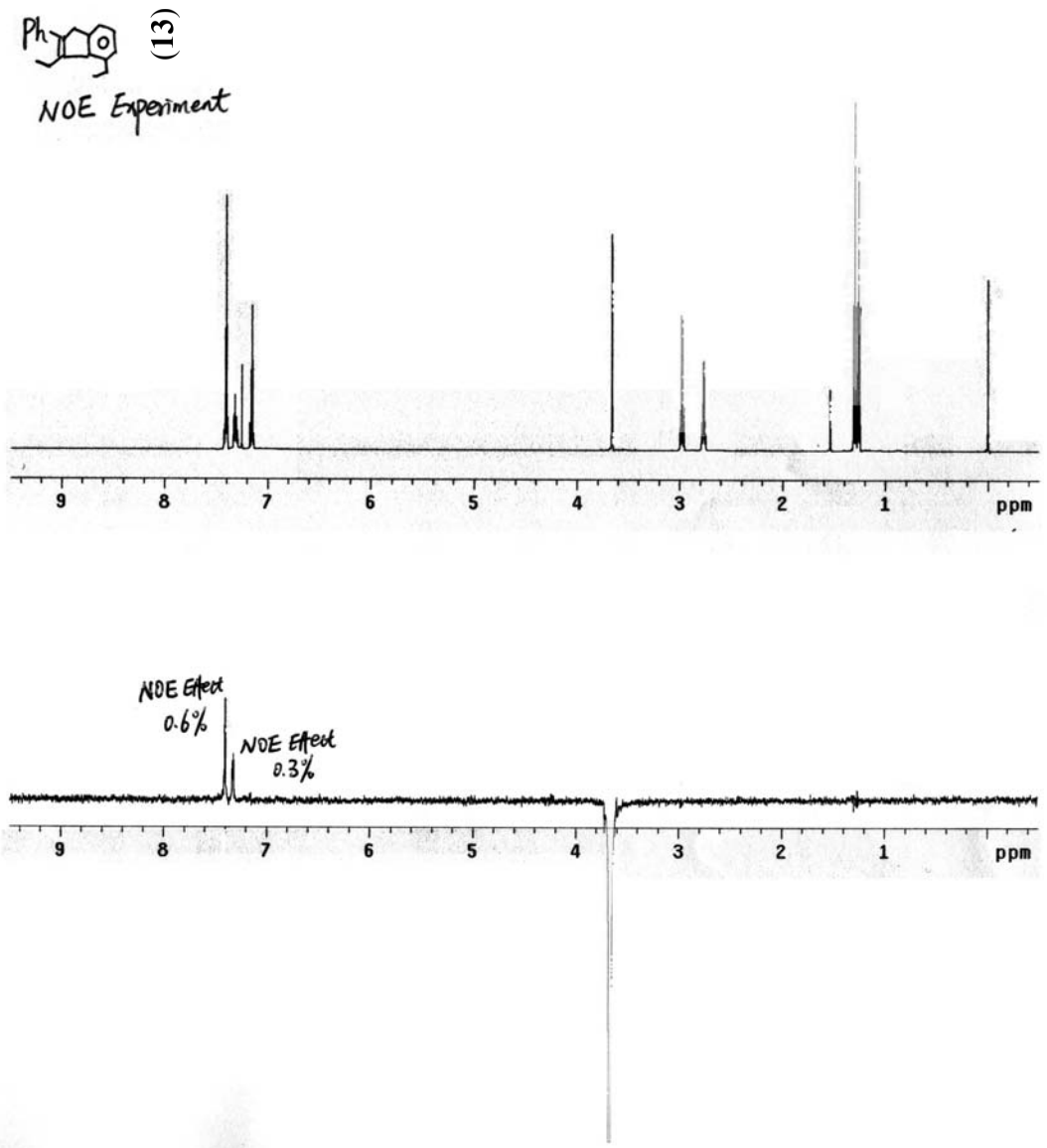
(13)

^{13}C NMR

Dong_VIV_P121_G2_noesy1D

Pulse Sequence: noesy1D_da
Solvent: CDCl3
Ambient temperature
INOVA-600 "u600"

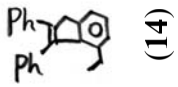
Relax. delay 2.000 sec
Pulse 90.0 degrees
Mixing 0.400 sec
Acq. time 1.892 sec
Width 8000.0 Hz
600 repetitions
OBSERVE H1, 599.9392965 MHz
DATA PROCESSING
Line broadening 0.7 Hz
FT size 32768
Total time 1 hr, 28 min, 11 sec



Dong_VIV_P117_G2_1H

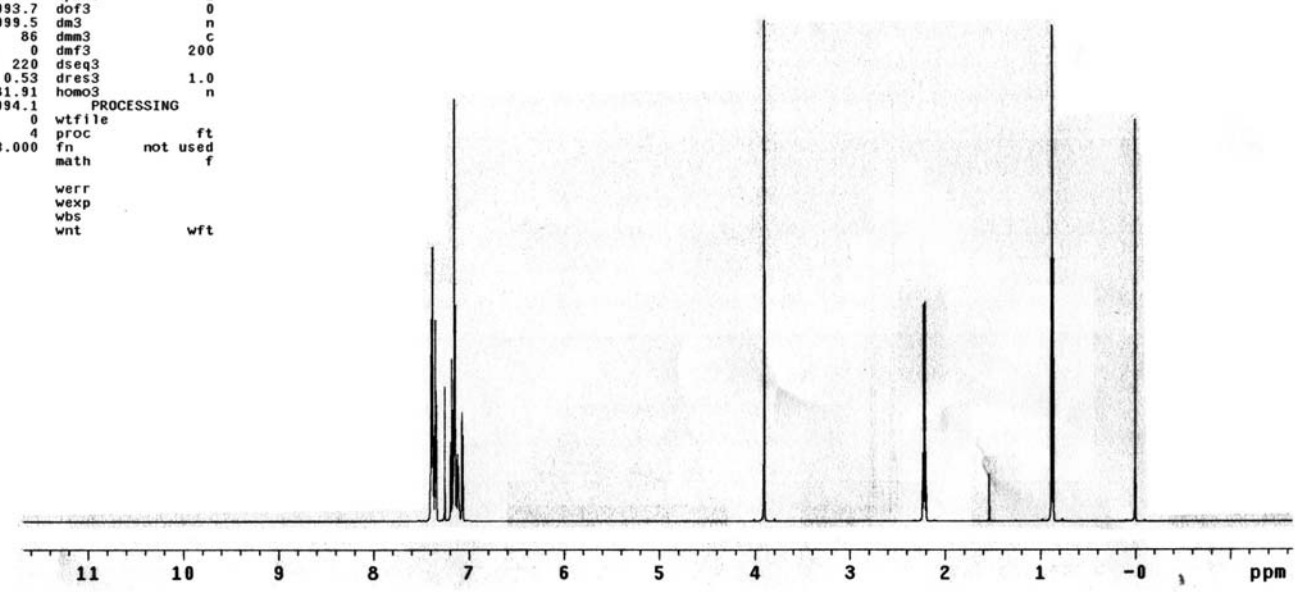
exp1 s2pul

SAMPLE		DEC. & VT	
date	Oct 13 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.942	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	59	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	0	dof2	0
nt	8	dm2	n
ct	8	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-993.7	dof3	0
wp	7999.5	dm3	n
vs	86	dmm3	c
sc	0	dmf3	200
wc	220	dseq3	
hzmm	0.53	dres3	1.0
is	181.91	homo3	n
rfl	994.1	PROCESSING	
rfp	0	wtfile	
th	4	proc	ft
ins	3.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



(14)

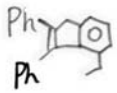
^{1}H NMR



Dong_VIV_P117_G2_13C

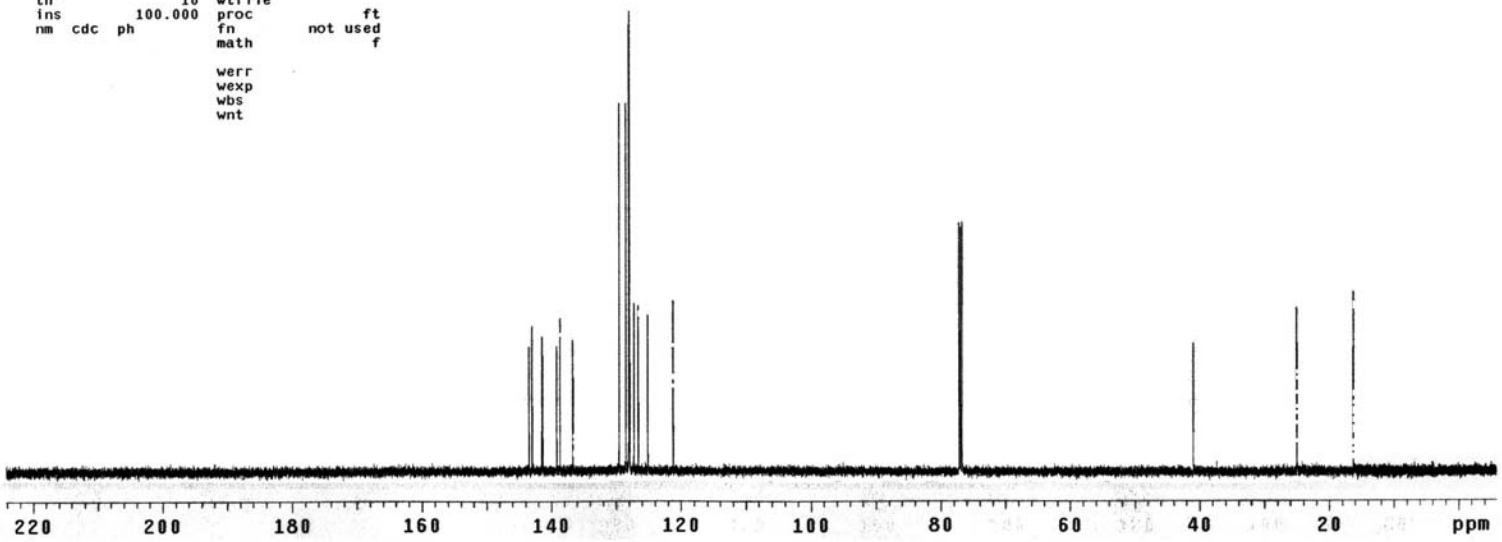
exp2 s2pul

SAMPLE		DEC. & VT	
date	Oct 6 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	16667
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	DEC2	
tpwr	61	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	3017.4	dof2	0
nt	10000	dm2	n
ct	616	dmm2	c
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-888.5	dof3	0
wp	34706.6	dm3	n
vs	76	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	138.83	dres3	1.0
is	500.00	homo3	n
rfl	12504.9	PROCESSING	
rfp	11615.8	lb	0.50
th	10	wtfile	
ins	100.000	proc	ft
nm cdc ph		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



(14)

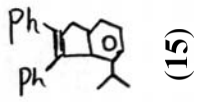
^{13}C NMR



Dong_VIV_P116_G2_1H

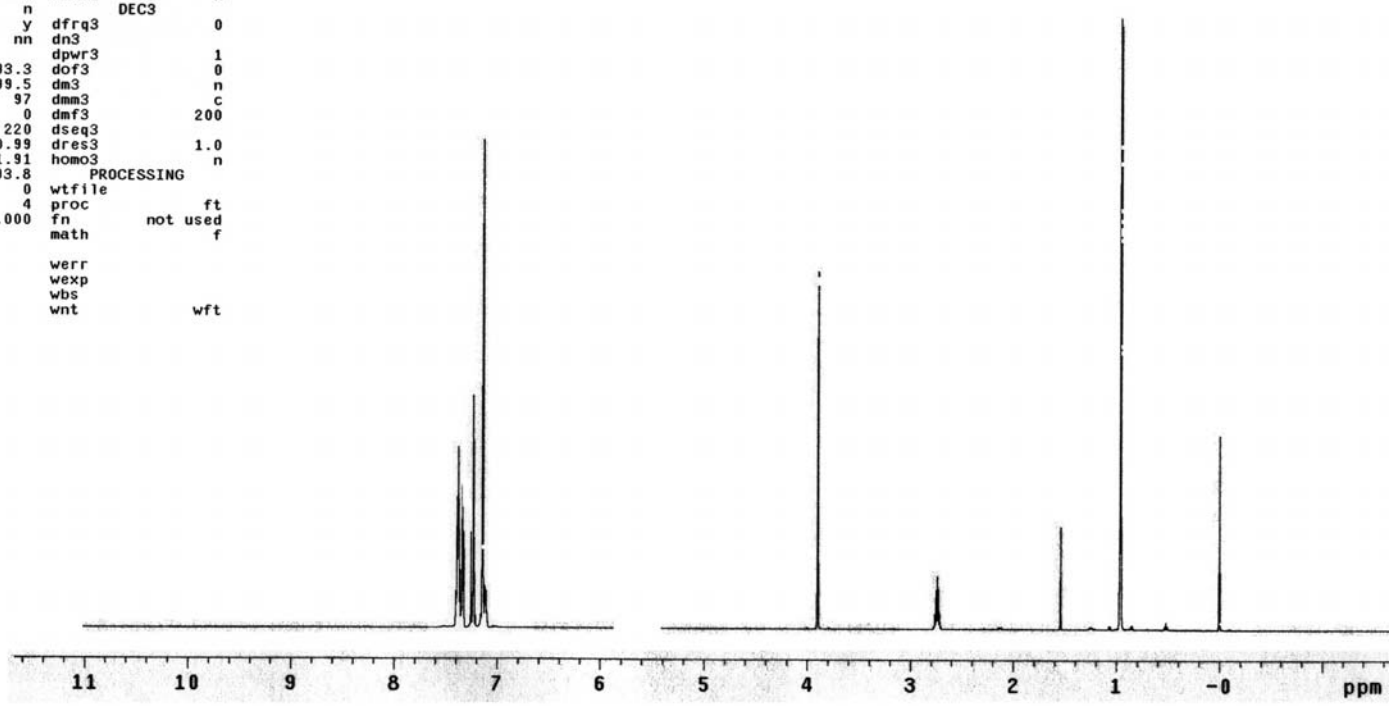
exp1 s2pul

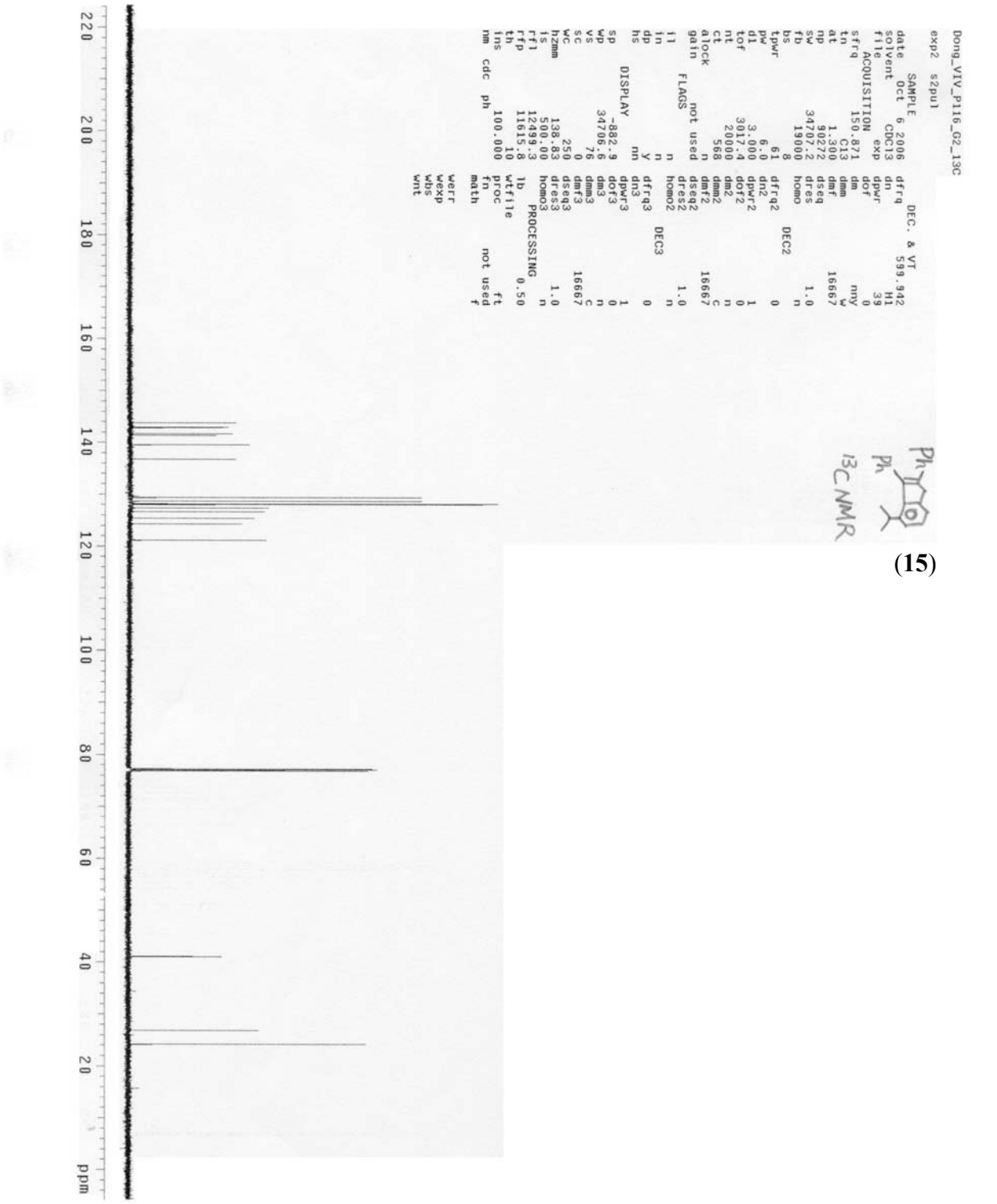
SAMPLE		DEC. & VT	
date	Oct 6 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.942	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	59	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	0	dof2	0
nt	8	dm2	n
ct	8	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-993.3	dof3	0
wp	7999.5	dm3	n
vs	97	dmm3	c
sc	0	dmf3	200
wc	220	dseq3	
hzmm	0.99	dres3	1.0
is	181.91	homo3	n
rfl	993.8	PROCESSING	
rfp	0	wtfile	
th	4	proc	ft
ins	2.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



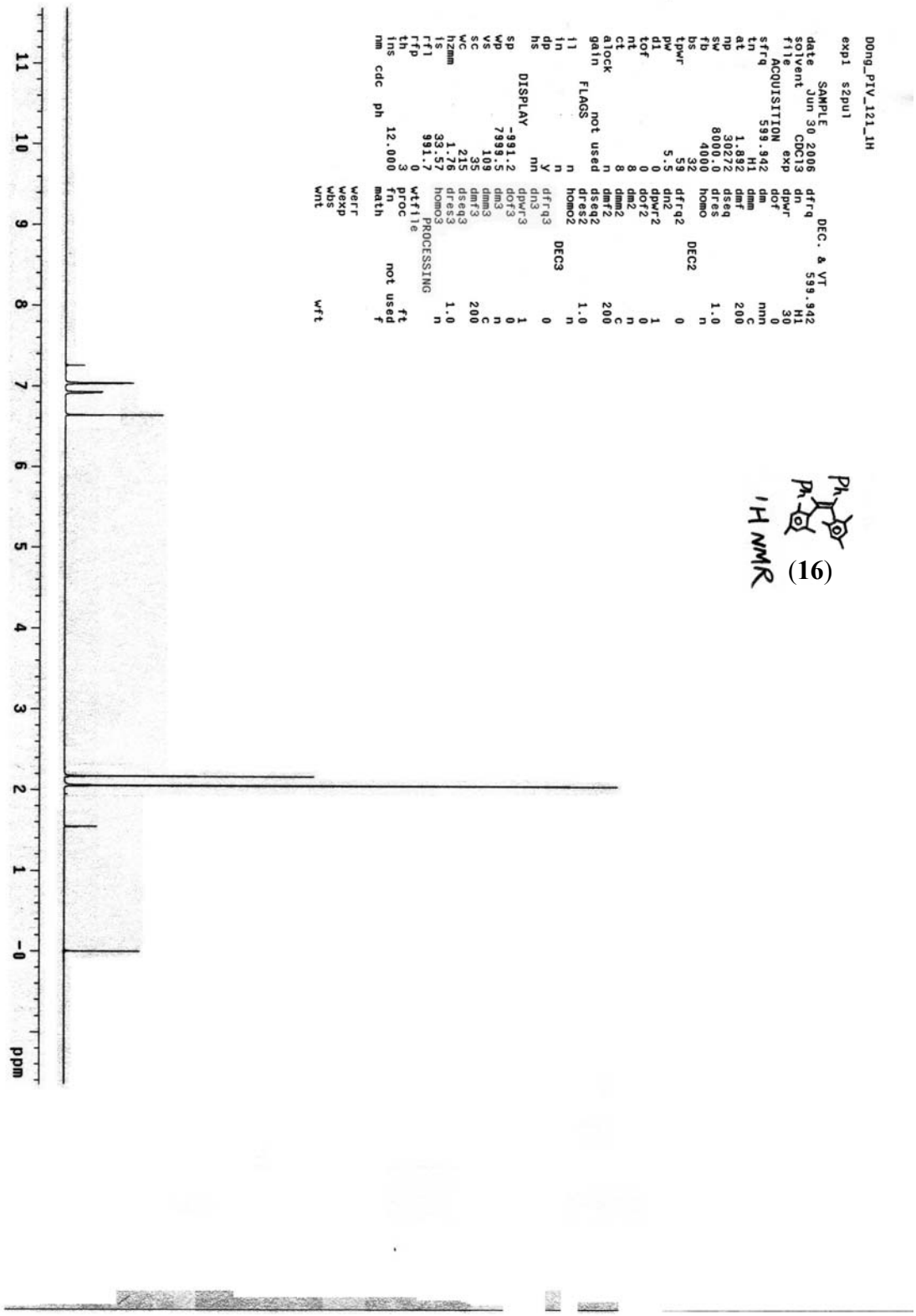
(15)

^1H NMR




Dong_VIV_P116_G2_13C
exp2 s2pul
SAMPLE
date Oct 6 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 61
pw 6.0
d1 3.000
tof 3017.4
nt 2000
ct 588
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -882.9
wp 34706.6
vs 76
sc 0
wc 250
hzmm 138.83
is 500.00
rfl 12499.3
rfp 11615.8
th 10
ins
nm cdc ph 100.000
werr
wexp
wbs
wnt
DEC. & VT
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
DEC2
dfrq2
dn2
dpwr2
dof2
dm2
dmm2
dmf2
dseq2
dres2
homo2
DEC3
dfrq3
dn3
dpwr3
dof3
dm3
dmm3
dmf3
dseq3
dres3
homo3
PROCESSING
lb
wtfile
proc
fn
math
599.942
H1
39
0
nny
w
16667
1.0
n
n
0
1
0
n
c
16667
c
16667
1.0
n
n
0
1
0
n
c
16667
1.0
n
0.50
ft
not used
f
13C NMR
220
200
180
160
140
120
100
80
60
40
20
ppm

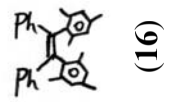
(15)


DOng_PIV_121_1H
exp1 s2pul
SAMPLE
date Jun 30 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 32
tpwr 59
pw 5.5
pl 0
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in y
dp nn
hs
DISPLAY
sp -991.2
wp 7999.5
vs 109
sc 35
wc 215
hzmm 1.76
is 33.57
rfl 991.7
rfp 0
th 3
ins 12.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
Ph
Ph
1H NMR
(16)
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

Dong_PIV_121_13C

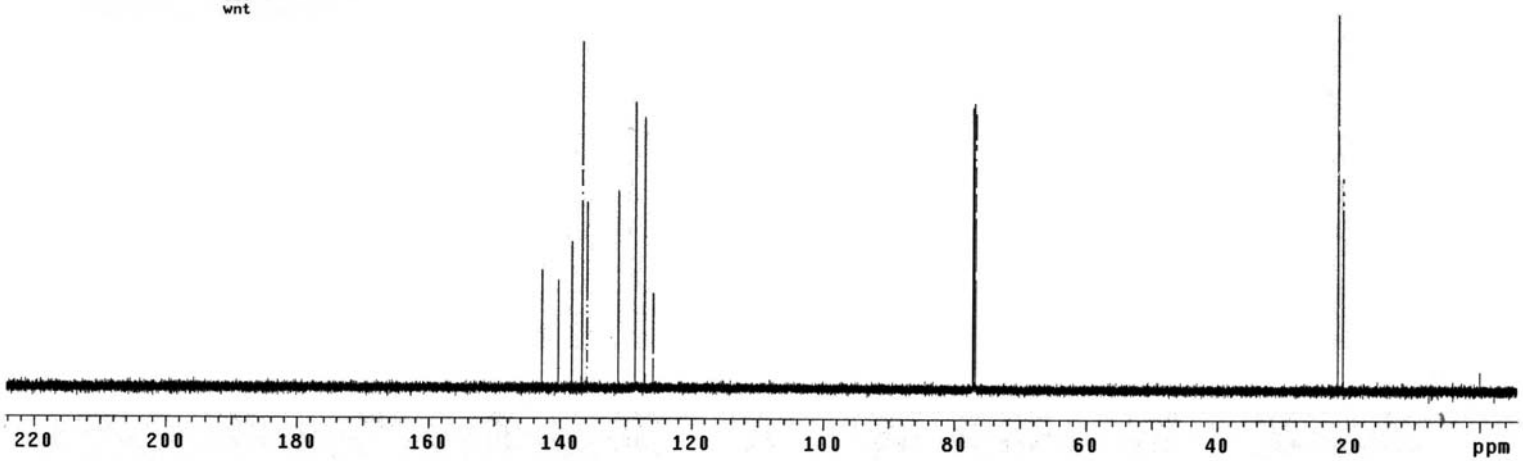
exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 30 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	16667
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	DEC2	
tpwr	61	dfrq2	0
pw	6.0	dn2	
a1	3.000	dpwr2	1
tof	3017.4	dof2	0
nt	10000	dm2	n
ct	528	dmm2	c
alock	n	dmf2	16667
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-874.6	dof3	0
wp	34706.6	dm3	n
vs	62	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	138.83	dres3	1.0
is	500.00	homo3	n
rfl	12491.0	PROCESSING	
rfp	11615.8	wtfile	
th	9	proc	ft
ins	100.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	



(16)

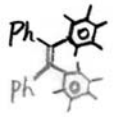
^{13}C NMR



Dong_VIII_P157_G1_1H

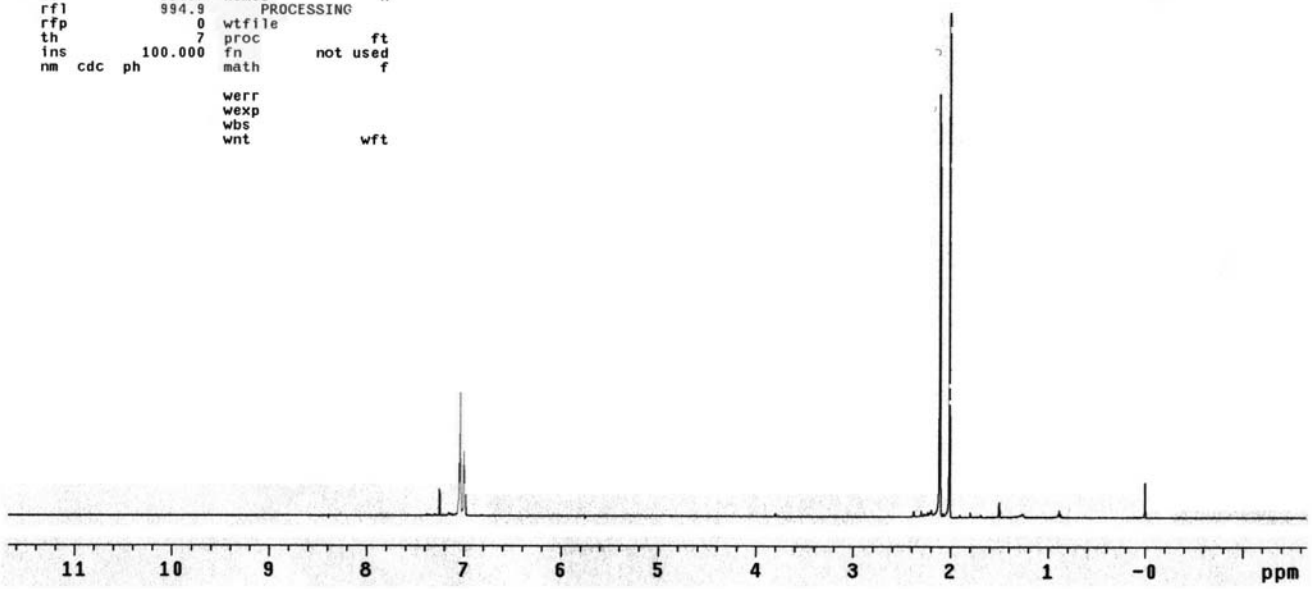
exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 19 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.942	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	32	DEC2	
tpwr	59	dfrq2	0
pw	5.5	dn2	
d1	0	dpwr2	1
tof	0	dof2	0
nt	8	dm2	n
ct	8	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-994.4	dof3	0
wp	7999.5	dm3	n
vs	83	dmm3	c
sc	35	dmf3	200
wc	215	dseq3	
hzmm	37.16	dres3	1.0
is	33.57	homo3	n
rfl	994.9	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	100.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



(17)

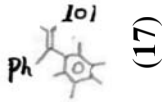
^1H NMR



Dong_VIII_P157_G1_13C

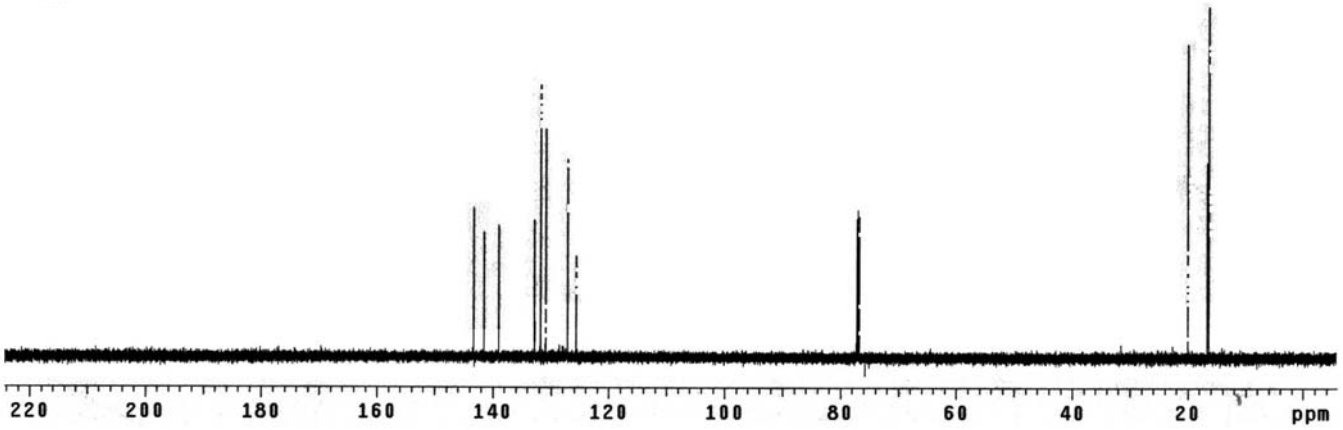
exp2 s2pul

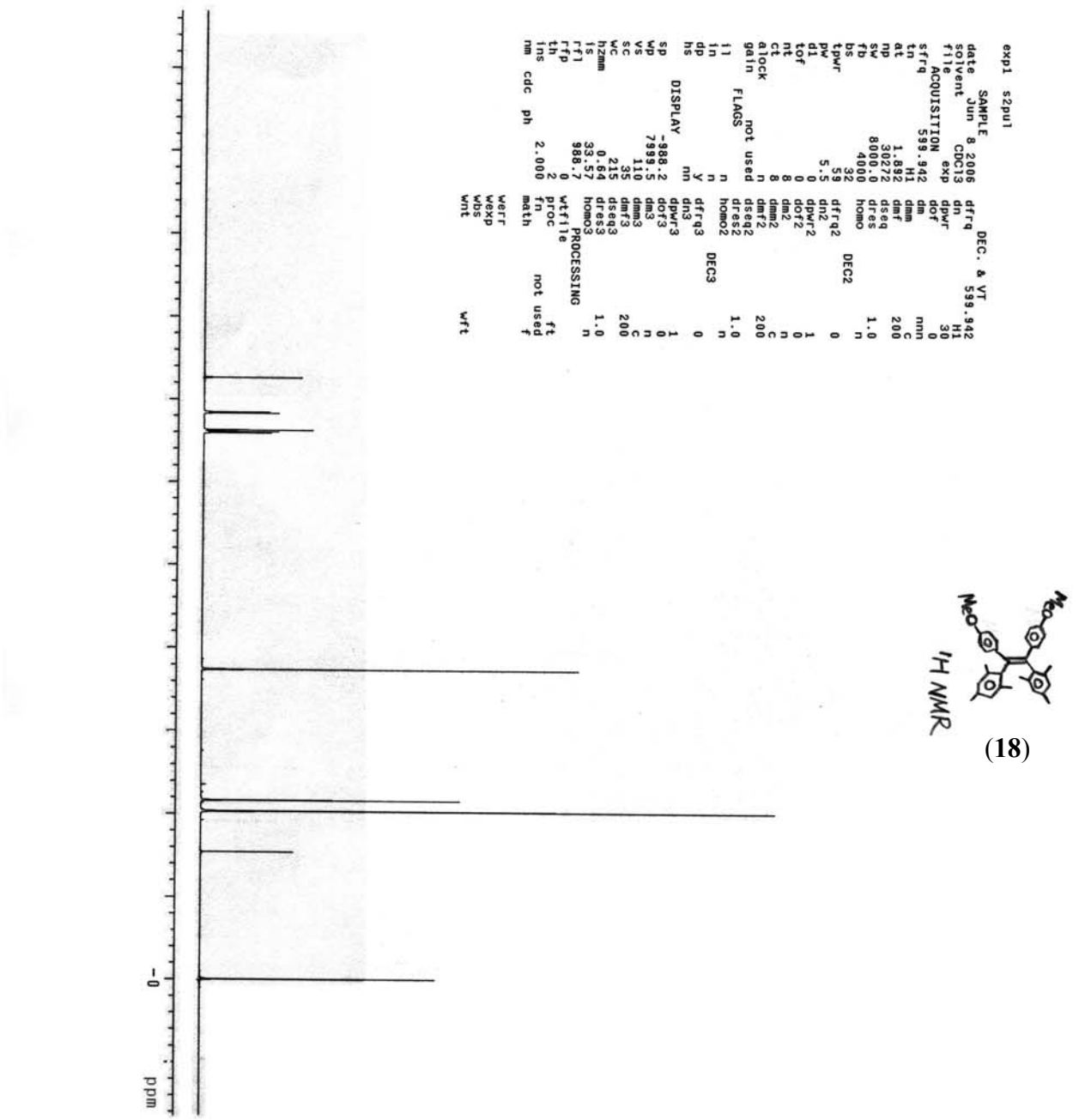
SAMPLE		DEC. & VT	
date	Apr 18 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	15877
np	90272	dseq	
sw	34707.2	dres	1.0
fb	19000	homo	n
bs	8	temp	25.0
tpwr	61	DEC2	
pw	6.0	dfrq2	0
d1	3.000	dn2	
tof	3017.4	dpwr2	1
nt	1000	dof2	0
ct	256	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	16667
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-875.7	dpwr3	1
wp	34706.6	dof3	0
vs	57	dm3	n
sc	0	dmm3	c
wc	220	dmf3	16667
hzmm	157.76	dseq3	
is	500.00	dres3	1.0
rfl	12492.1	homo3	n
rfp	11615.8	PROCESSING	
th	7	wtfile	
ins	100.000	proc	ft
nm cdc ph		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



(17)

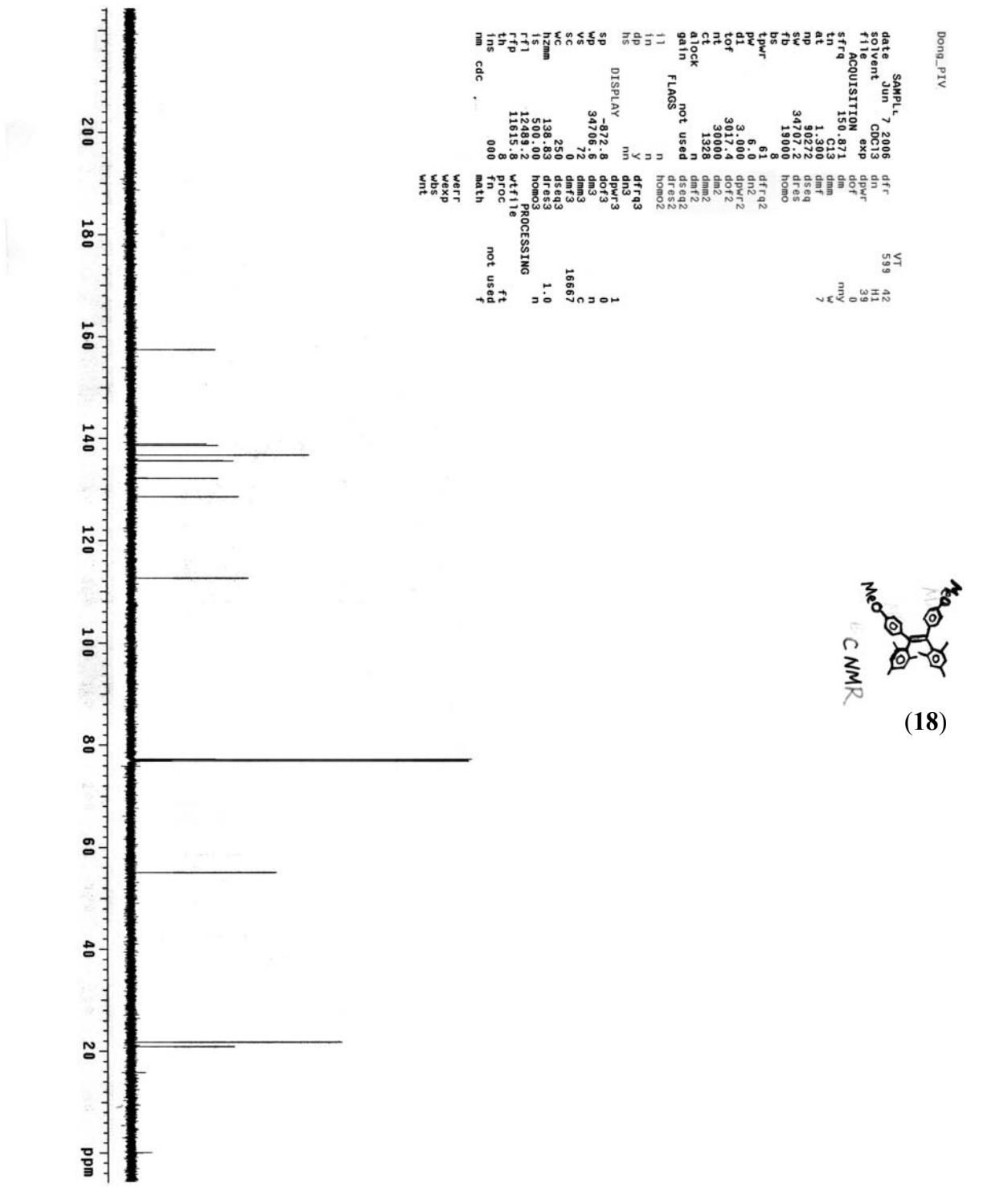
^{13}C NMR

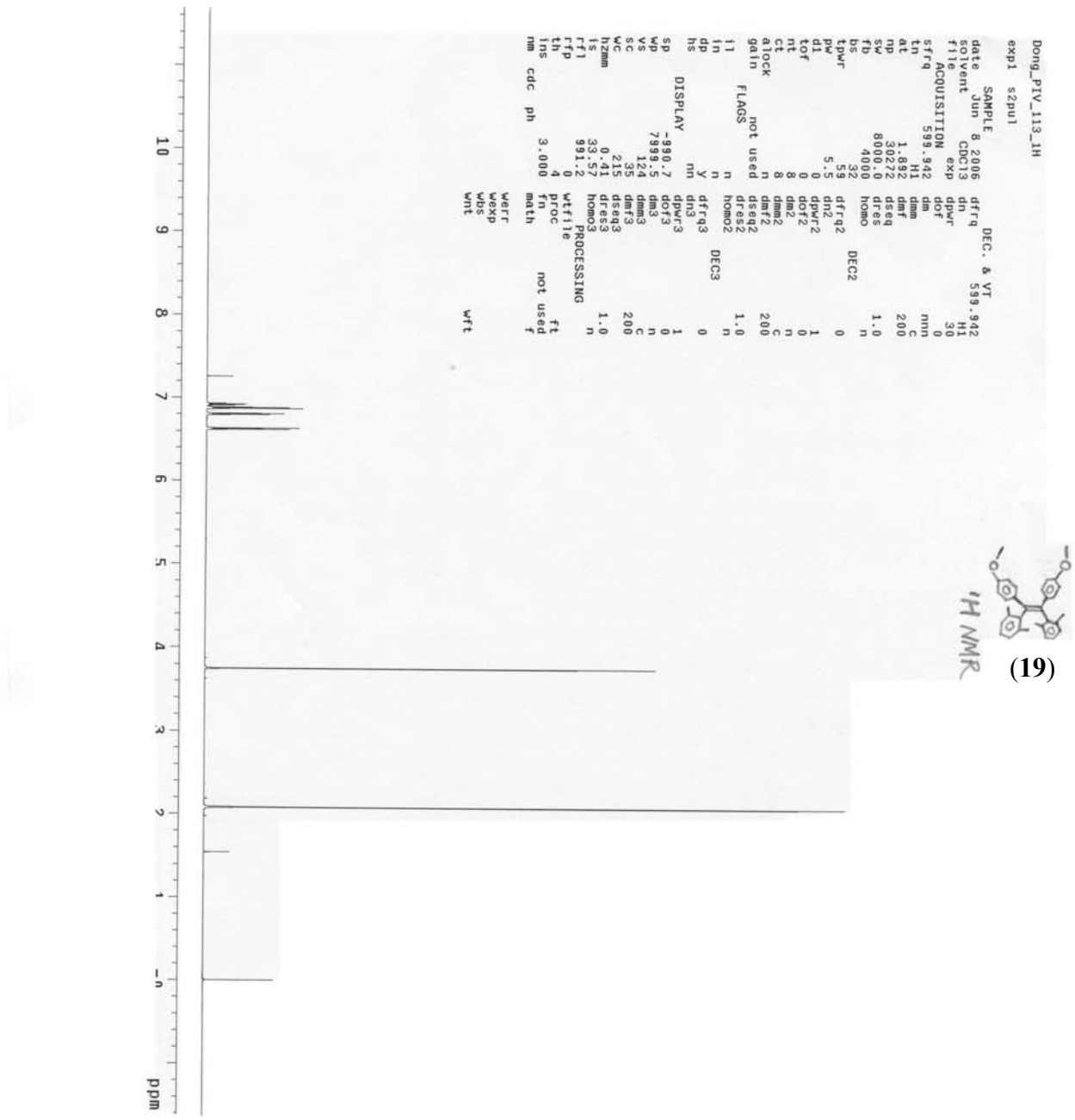



exp1 s2pul
SAMPLE
date Jun 8 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 32
tpwr 59
pw 5.5
d1 0
tof 0
nt 8
ct 8
alock n
gain n
FLAGS not used
il n
in n
dp y
hs nn
DISPLAY
sp -988.2
wp 7999.5
vs 110
sc 35
wc 215
hzmm 0.64
is 33.57
rfl 988.7
rfp 0
th 2
ins
nm cdc ph 2.000
dfrq DEC. & VT 599.942
dn H1
dof 30
dm nnn
dmm c
dmf 200
dseq
dres 1.0 n
homo
DEC2
dfrq2
dn2 0
dpwr2
dof2 1
dm2 0
dmm2 n
dmf2 c
dseq2 200
dres2
homo2 1.0
DEC3
dfrq3
dn3 0
dpwr3
dof3 1
dm3 0
dmm3 n
dmf3 c
dseq3 200
dres3
homo3 1.0
PROCESSING n
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
wft
0
ppm

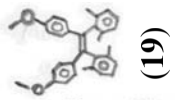
MeO
1H NMR

(18)


Dong_PIV
SAMPLE
date Jun 7 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 61
pw 6.0
d1 3.000
tof 3017.4
nt 30000
ct 1328
alock n
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -872.8
wp 3476.6
vs 72
sc 0
wc 250
hzmm 138.83
is 500.00
rfl 12489.2
rfp 11615.8
th 000
ins
nm cdc
werr
wexp
wbs
wnt
dfr
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
dfrq2
dn2
dpwr2
dof2
dm2
dmm2
dmf2
dseq2
dres2
homo2
dfrq3
dn3
dpwr3
dof3
dm3
dmm3
dmf3
dseq3
dres3
homo3
PROCESSING
wtfile
proc
fn
math
VT
599
42
H1
39
0
nny
w
7
1
0
n
c
16667
1.0
n
ft
not used
f
MeO
C NMR
(18)
200
180
160
140
120
100
80
60
40
20
ppm


Dong_PIV_113_1H
expl s2pul
SAMPLE
date Jun 8 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 599.942 H1
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 32
tpwr 59
pw 5.5
d1 0
tof 0
nt 8
ct 8
alock n
gain n
FLAGS not used
il n
in n
dp y
hs nn
DISPLAY
sp -990.7
wp 7999.5
vs 124
sc 35
wc 215
hzmm 0.41
is 33.57
rfl 991.2
rfp 0
th 4
ins 3.000
nm cdc ph
dfrq DEC. & VT
dn 599.942
dpwr H1
dof 30
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
10
9
8
7
6
5
4
3
2
1
0
ppm
'H NMR

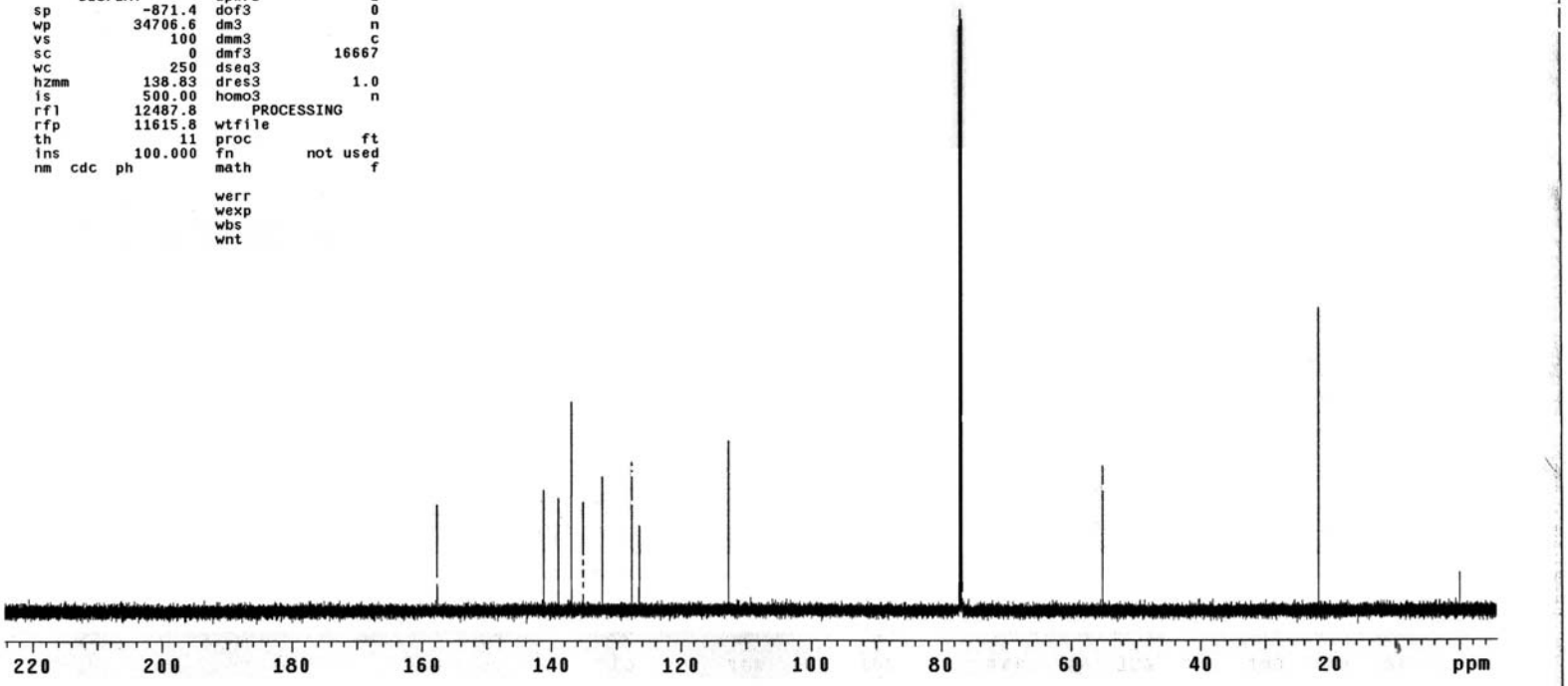
(19)

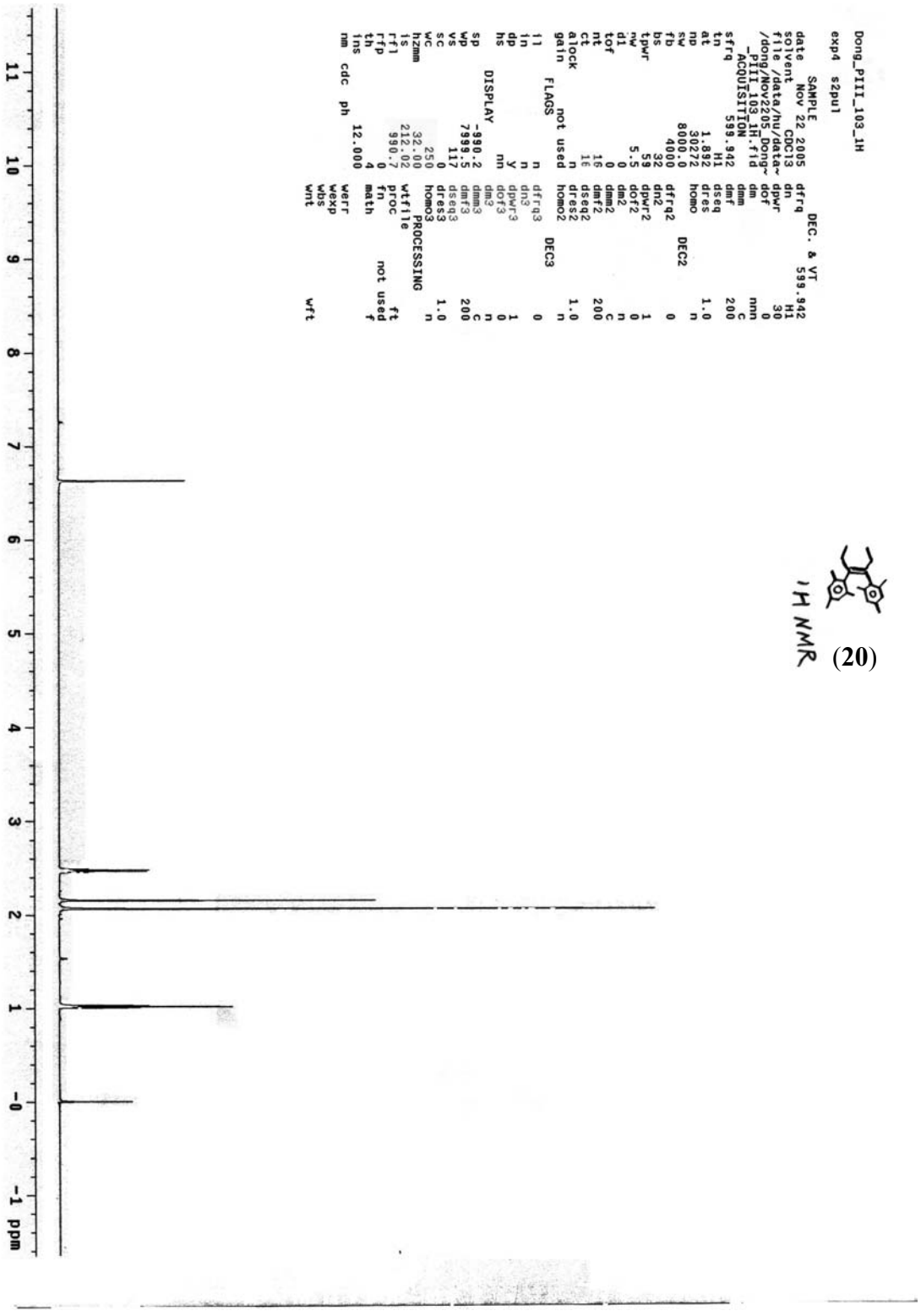


(19)

^{13}C NMR

```
Dong_PIV_113_13C

exp2  s2pul

      SAMPLE
date   Jun  8 2006
solvent      CDCl3
file           exp
   ACQUISITION
sfrq       150.871
tn             C13
at             1.300
np             90272
sw          34707.2
fb             19000
bs                 8
tpwr              61
pw               6.0
d1             3.000
tof           3017.4
nt             30000
ct              1256
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp            -871.4
wp           34706.6
vs               100
sc                 0
wc               250
hzmm          138.83
is            500.00
rfl          12487.8
rfp          11615.8
th                11
ins          100.000
nm  cdc  ph

      DEC. & VT
dfrq         599.942
dn                H1
dpwr              39
dof                0
dm               nny
dmm                w
dmf            16667
dseq
dres             1.0
homo               n
      DEC2
dfrq2              0
dn2
dpwr2              1
dof2               0
dm2                n
dmm2               c
dmf2           16667
dseq2
dres2            1.0
homo2              n
      DEC3
dfrq3              0
dn3
dpwr3              1
dof3               0
dm3                n
dmm3               c
dmf3           16667
dseq3
dres3            1.0
homo3              n
     PROCESSING
wtfile
proc              ft
fn         not used
math               f

werr
wexp
wbs
wnt
```




Dong_PIII_103_1H
exp4 s2pul
SAMPLE
date Nov 22 2005
solvent CDCl3
file /data/hu/data~
/dong_pIII_103_1H.fid
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
fb 8000.0
bs 32
tpwr 59
pw 5.5
d1 0
tof 16
nt 16
ct 1
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -990.2
wp 7999.5
vs 117
sc 0
wc 250
hzmm 32.00
is 212.02
rfl 990.7
rfp 0
th 4
ins 12.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dfrq2
dn2
dpwr2
dof2
dm2
dmm2
dmf2
dseq2
dres2
homo2
DEC3
dfrq3
dn3
dpwr3
dof3
dm3
dmm3
dmf3
dseq3
dres3
homo3
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm

1H NMR (**20**)

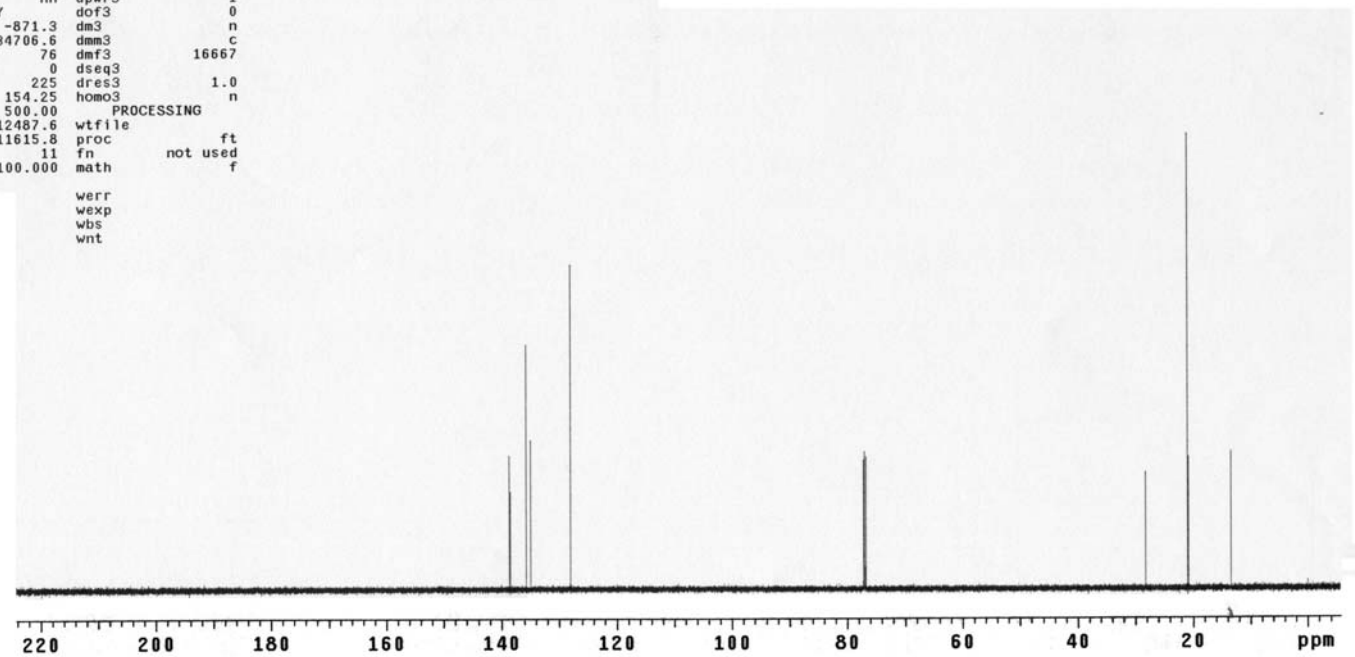
Dong_PIII_103_13C

exp4 s2pul

SAMPLE		DEC. & VT	
date	Nov 22 2005	dfrq	599.942
solvent	CDCl3	dn	H1
file	/data/hu/data~	dpwr	39
	/dong/Nov2205_Dong~	dof	0
	_PIII_103_13C.fid	dm	nny
ACQUISITION		dmm	w
sfrq	150.871	dmf	15877
tn	C13	dseq	
at	1.300	dres	1.0
np	90272	homo	n
sw	34707.2	temp	25.0
fb	19000	DEC2	
bs	8	dfrq2	0
tpwr	61	dn2	
pw	6.0	dpwr2	1
d1	3.000	dof2	0
tof	3017.4	dm2	n
nt	30000	dmm2	c
ct	568	dmf2	16667
alock	n	dseq2	
gain	not used	dres2	1.0
FLAGS		homo2	n
il	n	DEC3	
in	n	dfrq3	0
dp	y	dn3	
hs	nn	dpwr3	1
DISPLAY		dof3	0
sp	-871.3	dm3	n
wp	34706.6	dmm3	c
vs	76	dmf3	16667
sc	0	dseq3	
wc	225	dres3	1.0
hzmm	154.25	homo3	n
is	500.00	PROCESSING	
rfl	12487.6	wtfile	
rfp	11615.8	proc	ft
th	11	fn	not used
ins	100.000	math	f
nm ph			
		werr	
		wexp	
		wbs	
		wnt	

(20)

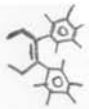
BC NMR



Dong_PIV_108_1H

exp1 s2pul

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	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	32	DEC2	
tpwr	59	dfrq2	0
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	0	dof2	0
nt	8	dm2	n
ct	8	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-988.6	dof3	0
wp	7999.5	dm3	n
vs	90	dmm3	c
sc	0	dmf3	200
wc	220	dseq3	
hzmm	36.36	dres3	1.0
is	343.34	homo3	n
rfl	989.1	PROCESSING	
rfp	0	wtfile	
th	3	proc	ft
ins	12.000	fn	not used
nm cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



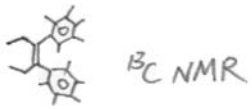
^1H NMR

(21)

Dong_PIV.

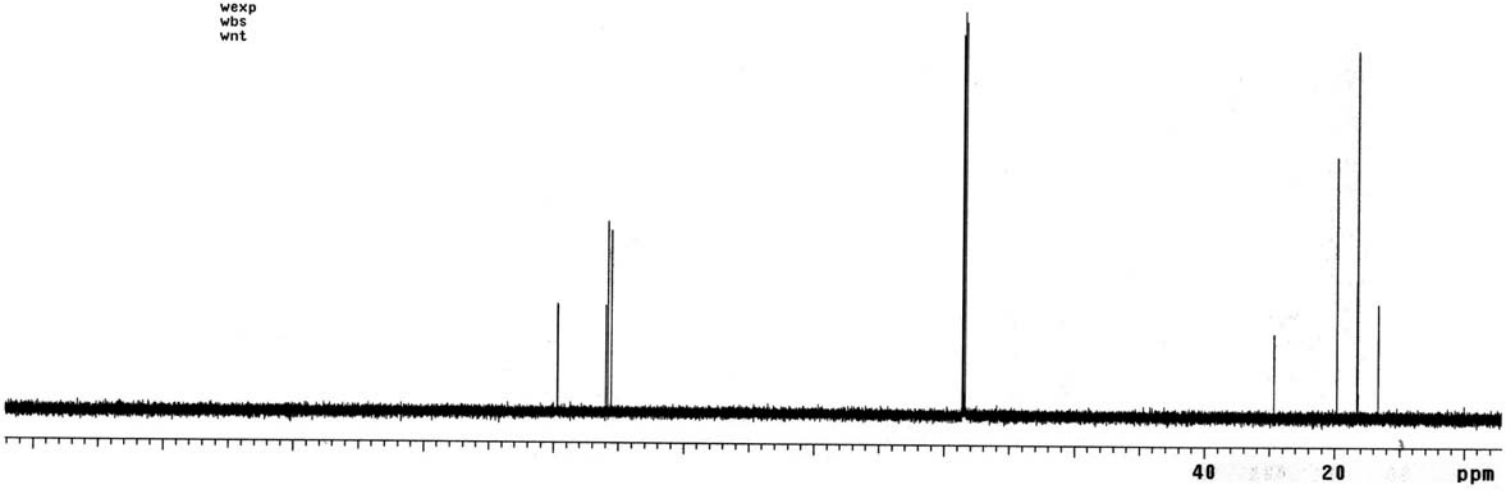
exp2

SAMPLE			
date	May 13 2006	dfrq	42
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	150.871	dm	nny
tn	C13	dmm	w
at	1.300	dmf	7
np	90272	dseq	
sw	34707.2	dres	0
fb	19000	homo	n
bs	8		
tpwr	61	dfr	
pw	6.0	dn2	
d1	3.000	dpwr2	1
tof	3017.4	dof2	0
nt	5000	dm2	n
ct	984	dmm2	c
alock	n	dmf2	7
gain	not used	dseq2	
LAGS		dres2	0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-874.1	dof3	0
wp	34706.6	dm3	n
vs	67	dmm3	c
sc	0	dmf3	16667
wc	250	dseq3	
hzmm	138.83	dres3	1.0
is	500.00	homo3	n
rfl	12490.4	PROCESSING	
rfp	11615.8	wtfile	
th	7	proc	ft
ins	000	fn	not used
nm		math	f
		werr	
		wexp	
		wbs	
		wnt	



^{13}C NMR

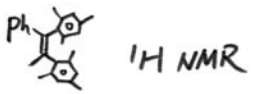
(21)



Dong_VIV_P34_1H

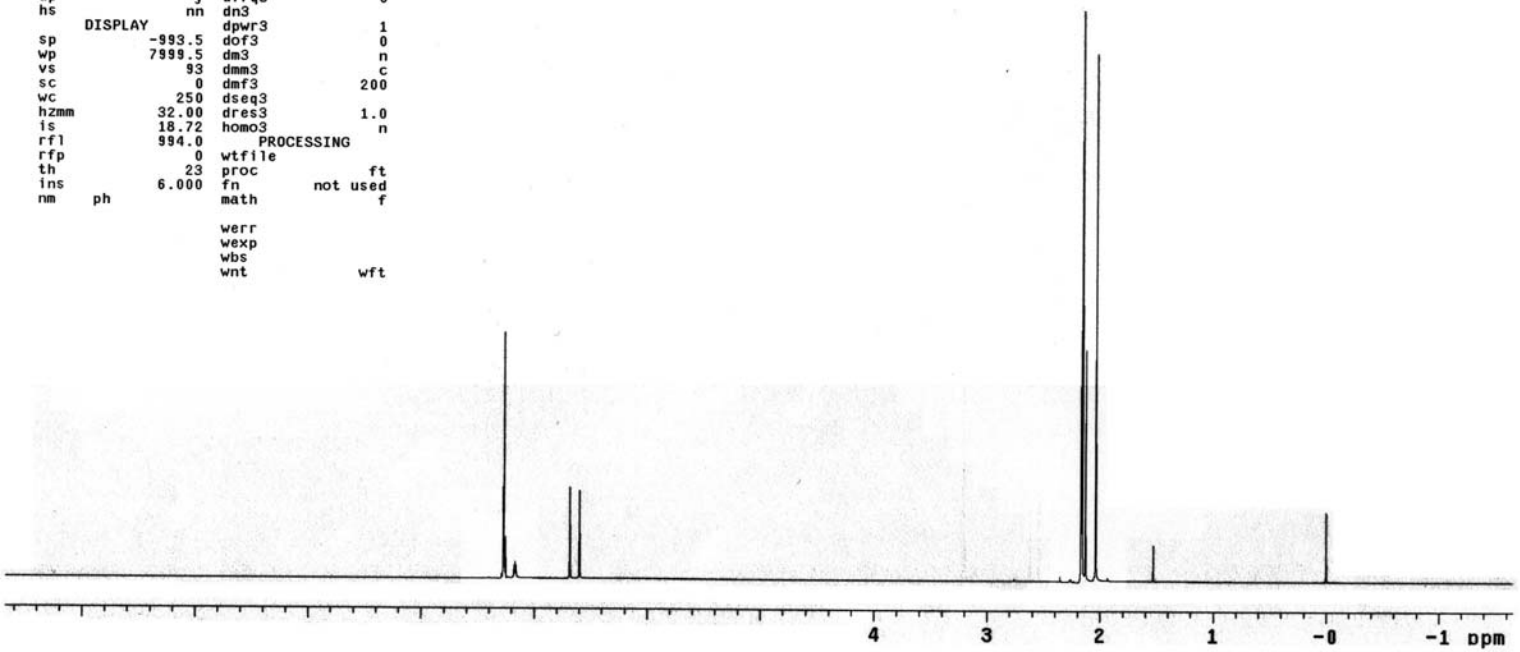
exp1 s2pul

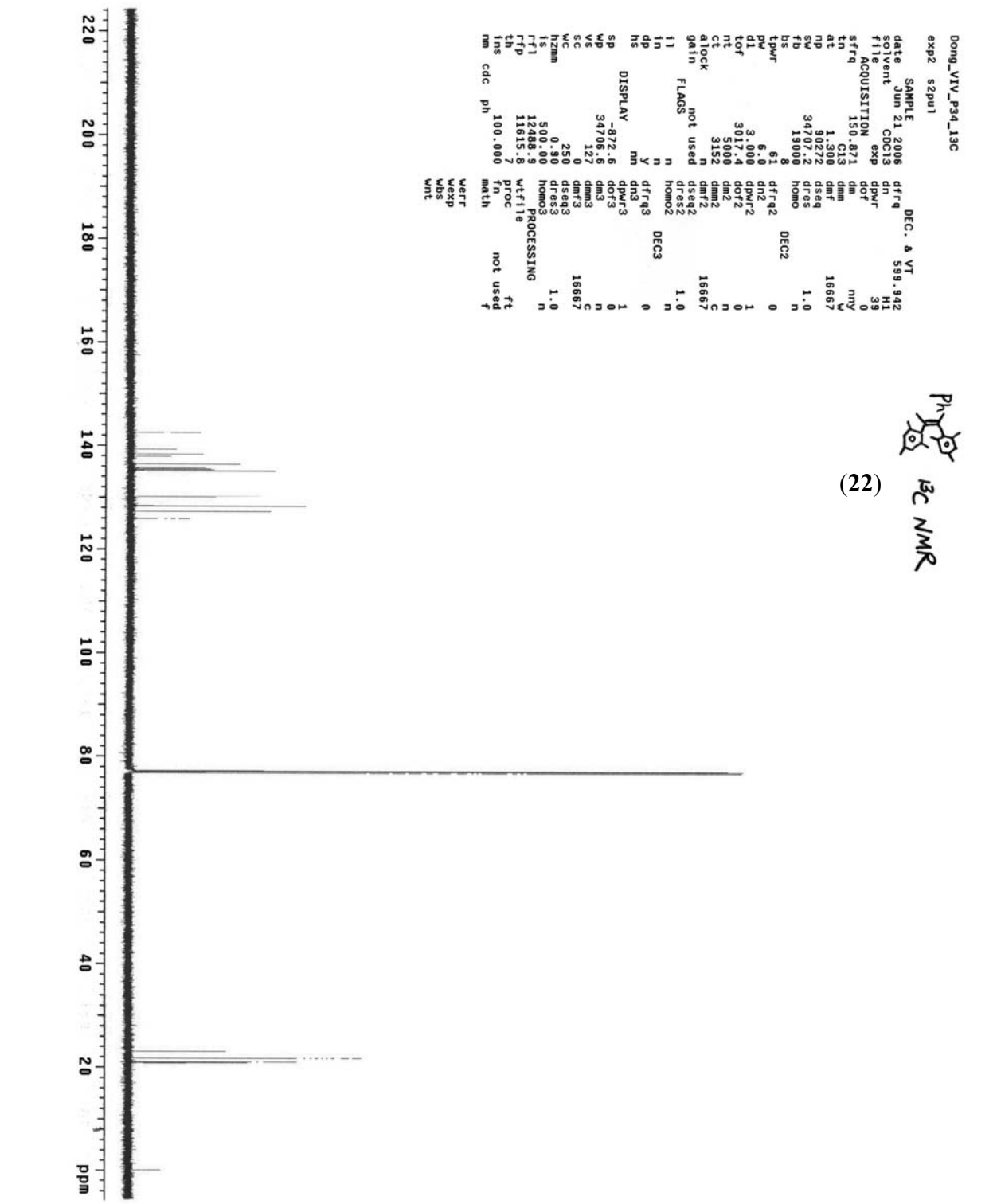
SAMPLE		DEC. & VT	
date	Jun 21 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.942	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	58	dfrq2	0
pw	5.5	dn2	
d1	0	dpwr2	1
tof	0	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-993.5	dof3	0
wp	7999.5	dm3	n
vs	93	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	32.00	dres3	1.0
is	18.72	homo3	n
rfl	994.0	PROCESSING	
rfp	0	wtfile	
th	23	proc	ft
ins	6.000	fn	not used
nm ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft

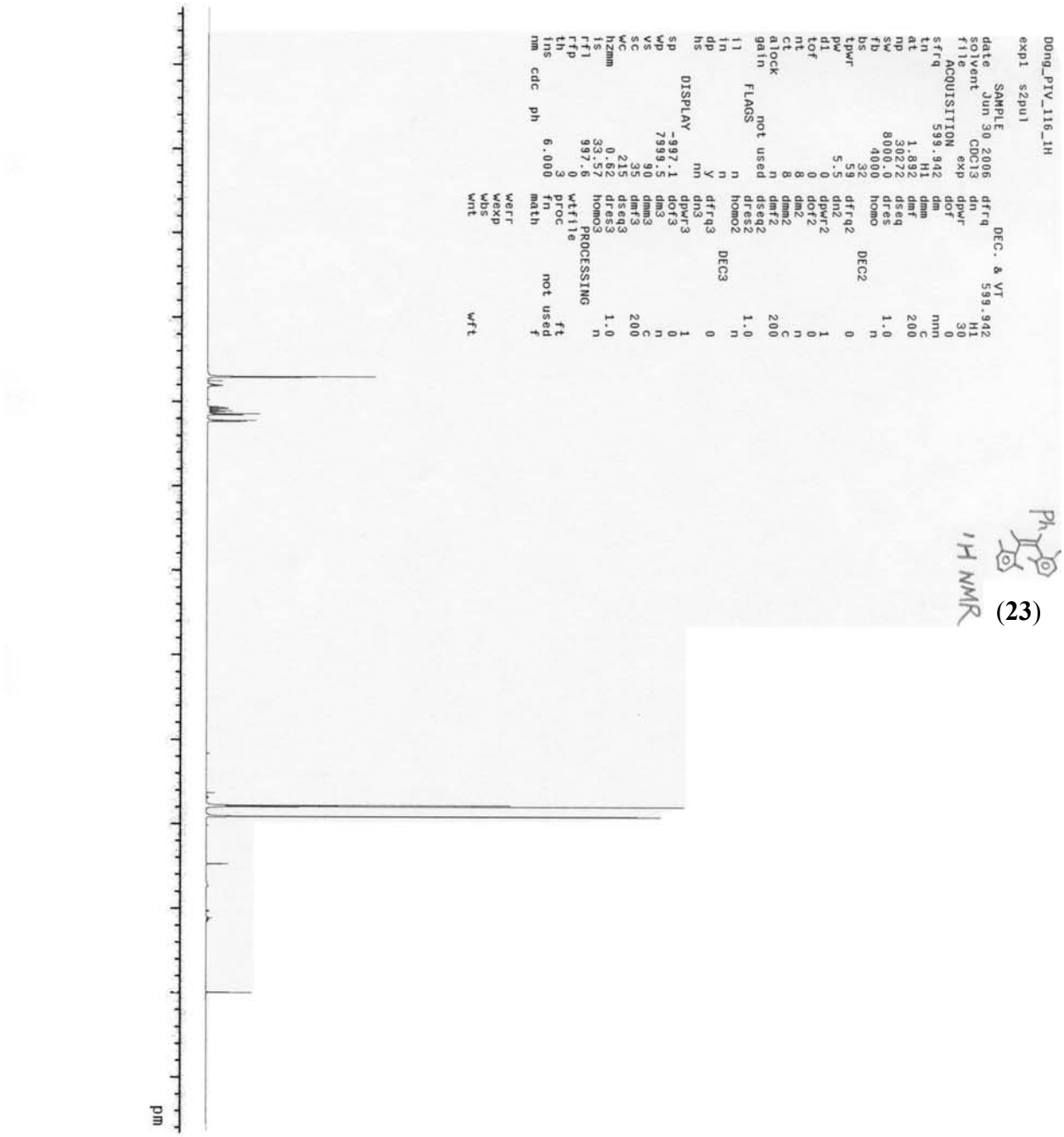


^{1}H NMR

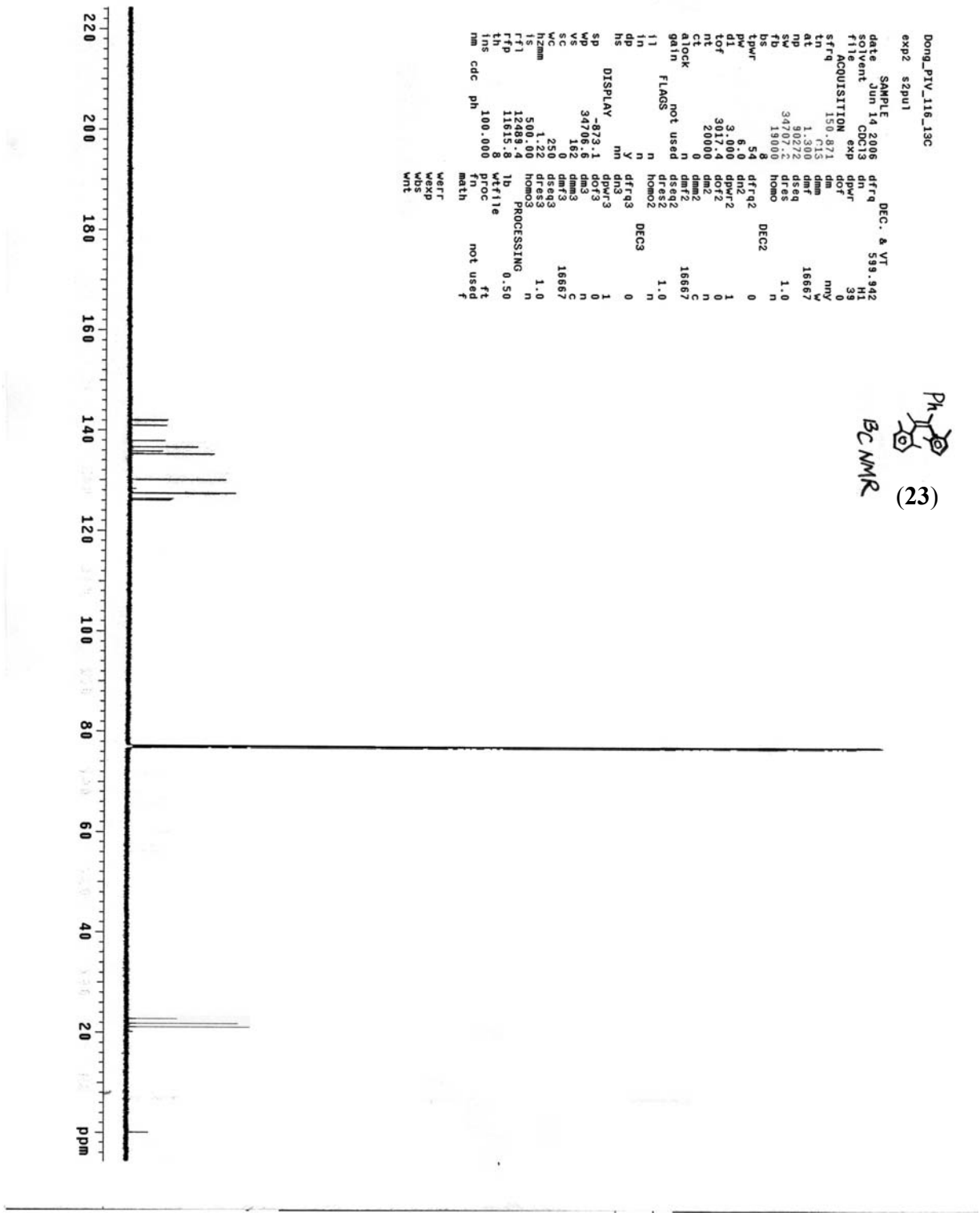
(22)




Dong_VIV_P34_13C
exp2 s2pul
SAMPLE
date Jun 21 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 98272
sw 34707.2
fb 19000
bs 8
tpwr 61
pw 6.0
d1 3.000
tof 3017.4
nt 5000
ct 3152
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -872.6
wp 34706.6
vs 127
sc 0
wc 250
hzmm 0.90
is 500.00
rfl 12488.9
rfp 11615.8
th 7
ins 100.000
nm cdc ph
DEC. & VT
dfrq 599.942
dn H1
dpwr 39
dof 0
dm nny
dmf 16667
dseq
dres 1.0
homo n
DEC2
dfrq2
dn2
dpwr2
dof2
dm2
dmm2
dmf2 16667
dseq2 c
dres2 1.0
homo2 n
DEC3
dfrq3
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 16667
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
(22)
13C NMR
220
200
180
160
140
120
100
80
60
40
20
ppm


D0ng_PIV_116_1H
expl s2pul
SAMPLE
date Jun 30 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 599.942
tn H1
at 1.892
np 30272
sw 8000.0
fb 4000
bs 32
tpwr 59
pw 5.5
pl 0
tof 8
nt 8
ct n
alock not used
gain
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -997.1
wp 7999.5
vs 90
sc 35
wc 215
hzmm 0.82
is 33.57
rfl 997.6
rfp 0
th 3
ins 6.000
nm cdc ph
werr
wexp
wbs
wnt
DEC. & VT
dfrq 599.942
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dres
homo 1.0
DEC2 n
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmf2 c
dseq2 200
dres2
homo2 1.0
DEC3 n
dfrq3
dn3 0
dpwr3
dof3 1
dm3 0
dmm3 n
dmf3 c
dseq3 200
dres3
homo3 1.0
PROCESSING n
wtfile
proc ft
fn not used
math f
wft
ppm

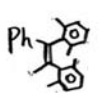
(**23**)


Dong_PIV_116_13C
exp2 s2pul
SAMPLE
date Jun 14 2006
solvent CDCl3
file
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 54
pw 6.00
dl 3.000
tof 3017.4
nt 20000
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -873.1
wp 34706.8
vs 162
sc 250
wc 1.22
hzmm 500.00
rfl 1249.4
rfp 11615.8
th 100.000
ins
cdc ph
dfrq DEC. & VT
dn 599.942
dpwr H1
dof 39
dm 0
nny y
dmm w
dmf 16667
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 16667
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 16667
dseq3
dres3 1.0
homo3 n
PROCESSING
lb
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Ph
BC NMR
(23)
220
200
180
160
140
120
100
80
60
40
20
ppm

Dong_PIV_116_noesy1D

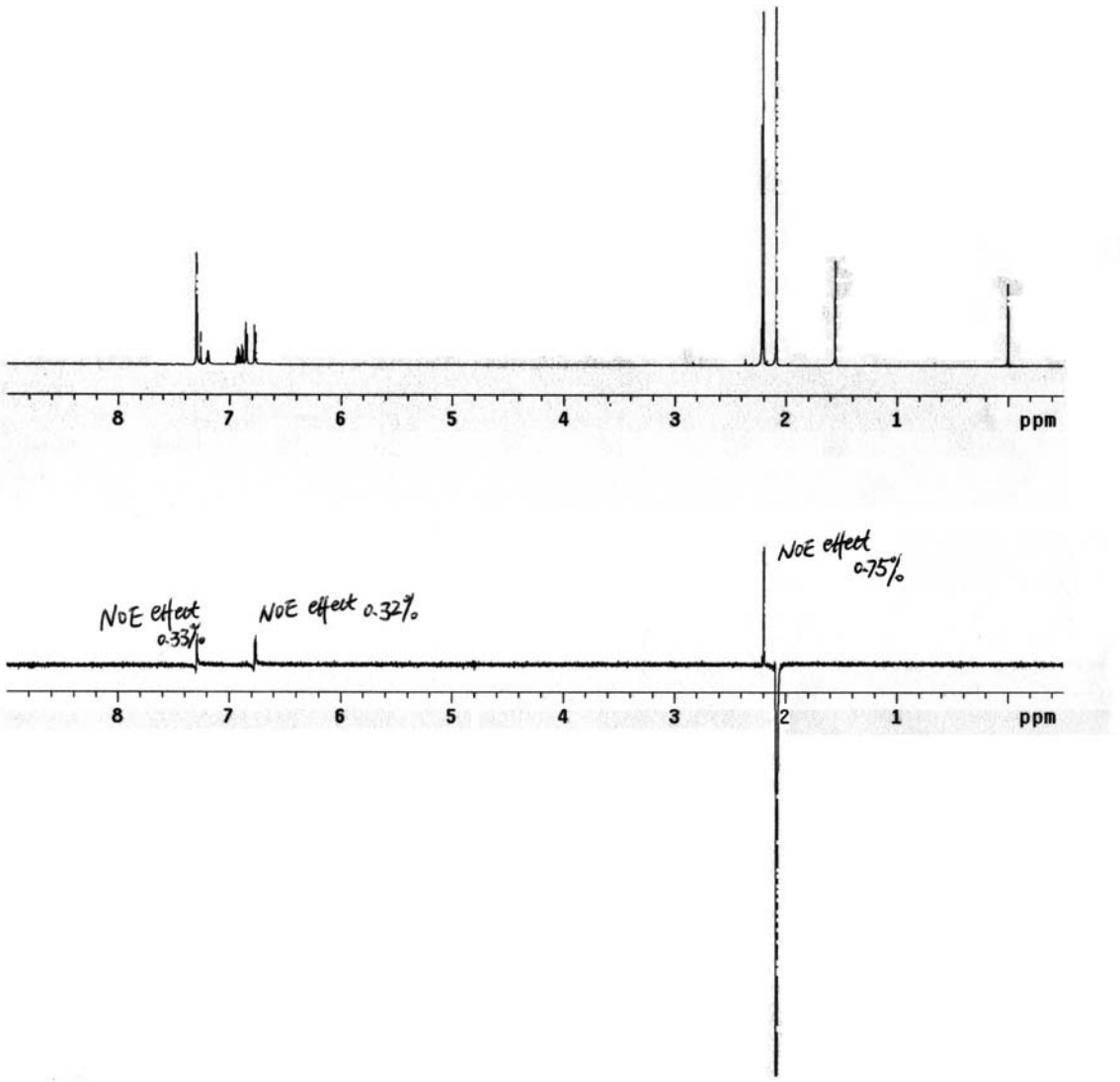
Pulse Sequence: noesy1D_da
Solvent: CDCl3
Ambient temperature
INOVA-600 "u600"

Relax. delay 2.000 sec
Pulse 90.0 degrees
Mixing 0.400 sec
Acq. time 1.892 sec
Width 8000.0 Hz
344 repetitions
OBSERVE H1, 599.9393058 MHz
DATA PROCESSING
FT size 32768
Total time 1 hr, 23 min, 28 sec



(23)

NOE Experiment



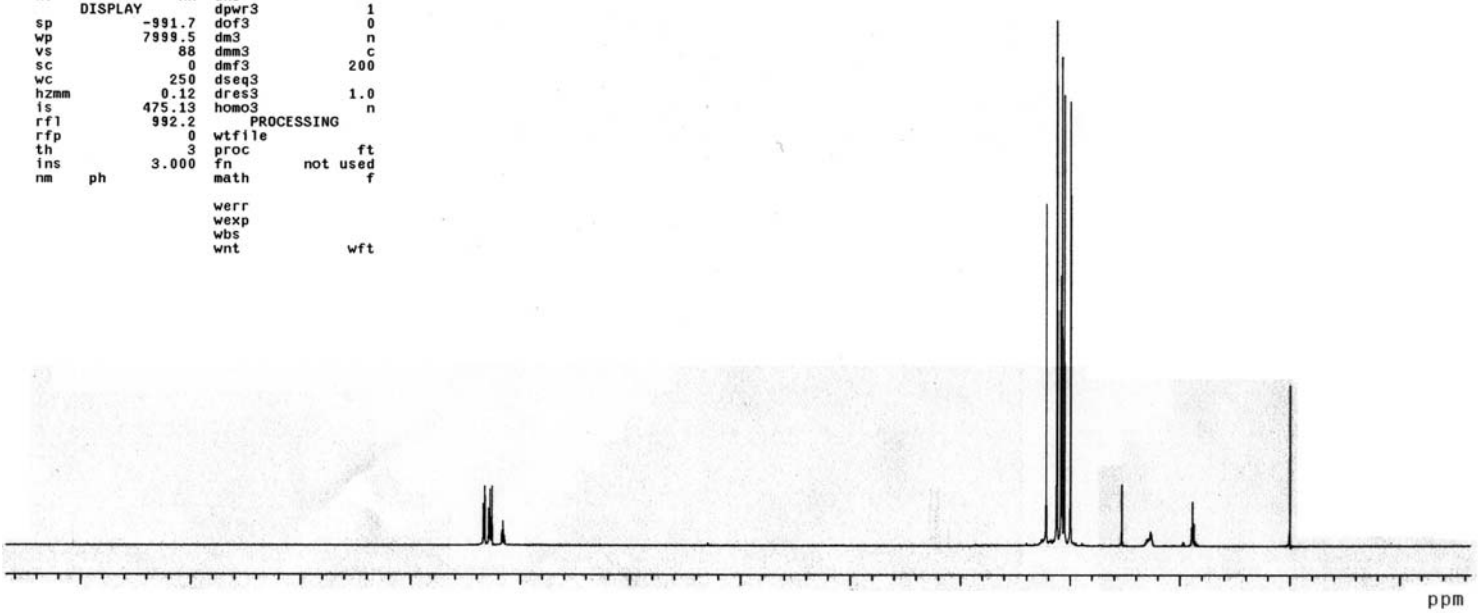
Dong_PIV_123_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jun 23 2006	dfrq	599.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.942	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	58	dfrq2	0
pw	5.5	dn2	
d1	0	dpwr2	1
tof	0	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-991.7	dof3	0
wp	7999.5	dm3	n
vs	88	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	0.12	dres3	1.0
is	475.13	homo3	n
rfl	992.2	PROCESSING	
rfp	0	wtfile	
th	3	proc	ft
ins	3.000	fn	not used
nm	ph	math	f
		werr	
		wexp	
		wbs	
		wnt	wft

(24)

1H NMR



Dong_PIV_123_13C
exp2 s2pul
SAMPLE
date Jul 19 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 150.871
tn C13
at 1.300
np 90272
sw 34707.2
fb 19000
bs 8
tpwr 54
pw 6.0
d1 3.000
tof 3017.4
nt 20000
ct 12896
alock n
gain n
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -873.3
wp 34706.6
vs 168
sc 0
wc 250
hzmm 1.73
is 500.00
rfl 12489.6
rfp 11615.8
th 22
ins 100.000
nm cdc ph
dfrq DEC. & VT
dn 599.942
dpwr H1
dof 39
dm 0
dmm nny
dmf w
dseq 16667
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2
dof2
dm2
dmm2 c
dmf2 16667
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 16667
dseq3
dres3 1.0
homo3 n
PROCESSING
lb
wtfile 0.50
proc
fn ft
math not used
werr f
wexp
wbs
wnt
220
200
180
160
140
120
100
80
60
40
20
ppm

(24)

^{13}C NMR

Dong_PIV_123_noesy1D

Pulse Sequence: noesy1D_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
440 repetitions
OBSERVE H1, 599.9392939 MHz
DATA PROCESSING
FT size 32768
Total time 1 hr, 24 min

(24) NOE