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Supporting Information

$Ce(OTf)_3$/PyBox Catalyzed Enantioselective Hosomi-Sakurai Reactions of Aldehydes with Allyltrimethylsilane

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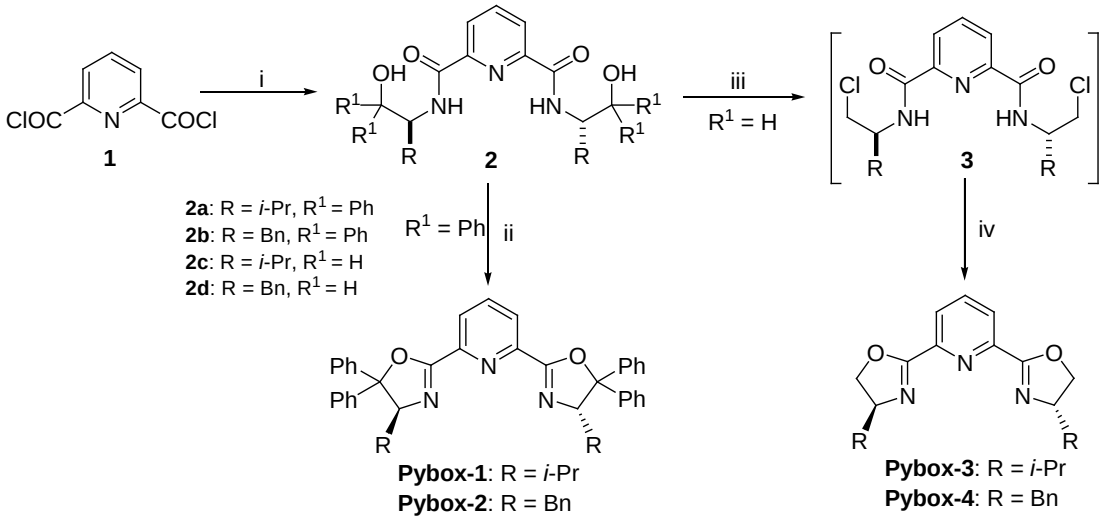
General Information

Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. All solvents used in the reactions were distilled from appropriate drying agents prior to use. Dichloromethane and trichloromethane were distilled from phosphorus pentoxide immediately prior to use. THF and toluene were distilled from sodium benzophenone ketone. Thionyl chloride and liquid aldehydes were purchased commercially and distilled before use. The 4 Å molecular sieves were purchased in a powder form, crushed, sieved at 100 mesh screen and then activated at 200^{o}C for 4 h. Reactions were monitored by thin layer chromatography (TLC) was performed on silica gel GF 254 (Qingdao, China). The TLC plates were visualized by exposure to ultraviolet light and/or immersion in a staining solution (phospho-molybdic acid) followed by heating on a hot plate. Flash column chromatography was undertaken on silica gel (200-300 mesh; Qingdao, China) using a proper eluent. Melting points were determined on a Yanaco MP-S3 micro melting point apparatus and are uncorrected. ^{1}H NMR and ^{13}C NMR was recorded on a Varian Inova-400 in deuterated solvent. ^{1}H chemical shifts are reported in ppm (δ) with the TMS or solvent resonance employed as the internal standard (TMS, δ 0.00 ppm; $CDCl_3$, δ 7.24 ppm). Data are reported as follows: chemical shift, number of equivalent nuclei (by integration), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, brs = broad singlet), coupling constant (Hz) and assignment. ^{13}C chemical shifts are reported in ppm (δ) with the TMS or solvent resonance employed as the internal standard (TMS, δ 0.00 ppm; $CDCl_3$, δ 77.0 ppm). Optical rotations were measured on a Autopol VI automatic polarimeter (Rudolph Research Analytical) and are reported as follows: concentration (*c* = g/dL), and solvent. The enantiomeric ratios were determined by high performance liquid chromatography (HPLC, Shimadzu LC-20AB) analysis employing a chiral stationary phase column specified in the individual experiment, by comparing the samples with the appropriate racemic mixtures.

The following abbreviations are used throughout: ethyl acetate (EtOAc), dichloromethane (CH_2Cl_2), chloroform ($CHCl_3$), tetrahydrofuran (THF), isopropanol (IPA), enantiomeric excess (*ee*), trimethylsilylchloride (TMSCl), melting point (m.p.), *tetra*-n-butylammonium fluoride (TBAF), 2,6-bis(oxazolinyl)pyridine (Pybox).

Catalyst Synthesis

Representative procedure for the synthesis of ligand PyBox-1~Pybox-4 (**Scheme 1**)



i) amino alcohol, CH_2Cl_2, Et_3N; ii) MsOH, CH_2Cl_2, reflux; iii) $SOCl_2$, $CHCl_3$, reflux; iv) NaOH, MeOH, rt

Scheme 1 synthesis of Pybox 1~Pybox 4

Pyridine-2,6-Dicarbonyl Dichloride (1) [1]. Pyridine-2, 6-dicarboxylic acid (0.418 g, 2.5 mmol) was dissolved in thionyl chloride (5 mL), the mixture was refluxed with stirring for 24 h under a nitrogen atmosphere until a homogenous brown solution was obtained.. The excess thionyl chloride was removed by distillation. The residue was placed under high vacuum for several hours to afford pyridine-2,6-dicarbonyl dichloride (0.501 g, 98.2%) as pale cream crystals (m.p. 55-58 ^oC). The product was moisture sensitive and used directly in the next procedure.

Amido Alcohols (2) [2, 3]. To a stirred solution of (*S*)-amino alcohol [4, 5] (2.0 mmol) and Et_3N (5.0 mmol, 0.7 mL) in CH_2Cl_2 (10 mL) was added dropwise a solution of pyridine-2,6-dicarbonyl dichloride (1.0 mmol, 0.204 g) in 10 mL of CH_2Cl_2 at 0 ^oC, and the mixture was stirred for 12 h (0 ^oC to rt). The reaction mixture was diluted with CH_2Cl_2 (10 mL) and then washed sequentially with aqueous $NaHCO_3$ (15 mL), water (30 mL) and brine (15 mL). The organic layer was dried with anhydrous Na_2SO_4. The filtrate was concentrated in vacuo, the residue was purified by column chromatography over silica gel to give corresponding pure amido alcohols **2**.

2,6-bis((*S*)-4,5-dihydro-4-isopropyl-5,5-diphenyloxazol-2-yl)pyridine (Pybox-1) [3b]. Compound **2a** (0.11 mmol, 70.6 mg) and methansesulfonic acid (0.66 mmol, 63.4 mg) were dissolved in CH_2Cl_2 (3 mL), the solution was refluxed for 6 h while keeping CaH_2 in an addition funnel for removing the water generated during the reaction. The reaction mixture was diluted with CH_2Cl_2

(3 mL), washed sequentially with aqueous $NaHCO_3$, water, and brine. The organic phase was dried with anhydrous Na_2SO_4. The filtrate was concentrated in vacuo, the residue was purified by column chromatography over silica gel to get the pure cyclized product **Pybox-1**(57.3 mg, yield 86%). White solid, m.p. 66-67 ^oC; $[\alpha]^{25}_D$ -252° (*c* 0.100, $CHCl_3$); R_f = 0.48 (4:1 hexane:EtOAc); ^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 8.22 (2H, dd, *J* = 8.0 Hz, *J* = 3.6 Hz, H-3), 7.92 (1H, t, *J* = 8.0 Hz, H-4), 7.63 (4H, d, *J* = 7.6 Hz, *J* = 7.2 Hz, Ph), 7.42-7.31 (8H, m, Ph), 7.30-7.22 (8H, m, Ph), 4.89 (2H, d, *J* = 4.8 Hz, H-4'), 1.95-1.90 (2H, m, C*H*CH_3), 1.06 (6H, d, *J* = 6.8 Hz, CH_3), 0.67 (6H, d, *J* = 6.4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 160.3 (C, C-2'), 146.8 (C, C-2), 144.9 (C, Ph), 140.3(C, Ph), 137.7 (CH, C-4), 128.3(CH, Ph), 127.9 (CH, Ph), 127.7 (CH, Ph), 127.3 (CH, Ph), 126.9 (CH, Ph), 126.3 (CH, Ph), 125.4 (CH, C-3), 93.7 (CH_2, C-5'), 80.3 (CH, C-4'), 30.3 (CH, C*H*CH_3), 21.9 (CH_3), 17.3 (CH_3).

2,6-bis((*S*)-4-benzyl-4,5-dihydro-5,5-diphenyloxazol-2-yl)pyridine (Pybox-2) [3b]. Using a procedure similar to that described above for the preparation of Pybox-1 from bisamide **2b**, bisamide **2b** (0.1 mmol, 73.8 mg) was treated with methansesulfonic acid (0.6 mmol, 57.7 mg) to afford **Pybox-2** (56.9 mg, 81%). White solid, m.p. 93-95 ^oC; $[\alpha]^{25}_D$ -361° (*c* 0.100, $CHCl_3$); R_f = 0.4 (4:1 hexane:EtOAc); ^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 8.21 (2H, d, *J* = 7.6 Hz, H-3), 7.89 (1H, t, *J* = 8.0 Hz, H-4), 7.54 (4 H, dd, *J* = 8.4 Hz, *J* = 3.2 Hz, Ph), 7.37-7.09 (26H, m, Ph), 5.24 (2H, t, *J* = 7.2 Hz, *J* = 6.0 Hz, H-4'), 2.67 (4H, d, *J* = 6.8 Hz, CH_2Ph). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 161.0 (C, C-2'), 147.3 (C, C-2), 143.8 (C, Ph), 140.3 (C, Ph), 138.8 (C, Ph), 137.3 (CH, C-4), 129.3 (CH, Ph), 128.4 (CH, Ph), 128.1 (CH, Ph), 127.9 (CH, Ph), 127.8 (CH, Ph), 127.6 (CH, Ph), 127.1 (CH, Ph), 126.5 (CH, Ph), 126.1 (CH, Ph), 125.9 (CH, C-3), 93.7 (CH_2, C-5'), 67.0 (CH, C-4'), 40.0 (CH_2, CH_2Ph).

2,6-bis((*S*)-4,5-dihydro-4-isopropyloxazol-2-yl)pyridine (Pybox-3) [6]. Thionyl chloride (25.0 mmol, 1.9 mL) was added with stirring to a solution of compound **2c** (2.0 mmol, 0.675 g) in chloroform (15 mL). The mixture was heated under reflux for 3 h, then cooled in ice bath. Water was added through the top of the condenser to quench the excess thionyl chloride and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic phases were washed with saturated aqueous $NaHCO_3$ (15 mL), water (30 mL) and brine (15 mL). The organic layer was dried with anhydrous $NaSO_4$. The filtrate was removed at reduced pressure to give compound **3** as a cream solid. The solid was dissolved in MeOH (15 mL) and treated immediately with

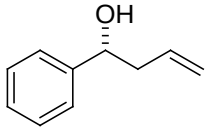
aqueous NaOH (5.3 mL, 3 mol·L^{-1}). The mixture was stirred at room temperature under a nitrogen atmosphere for 3 days, and then extracted with dichloromethane (2×30 mL). The extract was washed with water, brine and then dried with $NaSO_4$. The filtrate was concentrated in vacuo, the residue was purified by column chromatography over silica gel to get the pure cyclized product **Pybox-3** (0.374 g, yield 62%). White solid, m.p. 150-153 ^oC; $[\alpha]^{25}_D$ +124° (*c* 0.100, CH_2Cl_2); R_f = 0.31 (2:1, CH_2Cl_2:EtOAc); ^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 8.22 (2H, d, *J* = 8.0 Hz, H-3), 7.87 (1H, dd, *J* = 8.0 Hz, *J* = 7.6 Hz, H-4), 4.54 (2H, dd, *J* = 9.6 Hz, *J* = 8.4 Hz, H-5'A), 4.25-4.22 (2H, m, H-5'B), 4.18-4.12 (2H, m, H-4'), 1.87 (2H, m, C*H*CH_3), 1.05 (6H, d, *J* = 6.8 Hz, CH_3), 0.94 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 162.2 (C, C-2'), 146.9 (C, C-2), 137.2 (CH, C-4), 125.7 (CH, C-3), 72.9 (CH_2, C-5'), 71.0 (CH, C-4'), 32.8 (CH, C*H*CH_3), 19.1 (CH_3), 18.3 (CH_3).

2,6-bis((*S*)-4-benzyl-4, 5-dihydrooxazol-2-yl)pyridine (Pybox-4) [6]. Using a procedure similar to that described above for the preparation of Pybox-3 from bisamide **2d,** bisamide **2d** (0.5 mmol, 0.396 g) was treated with thionyl chloride to afford **Pybox-4** (0.134 g, 67%).. White solid, m.p. 155-156 ^oC; $[\alpha]^{25}_D$ -58° (*c* 0.100, CH_2Cl_2); R_f = 0.25 (2:1 CH_2Cl_2:EtOAc); ^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 8.22 (2H, d, *J* = 7.6 Hz, H-3), 7.89 (1H, t, *J* = 8.0 Hz, *J* = 7.6 Hz, H-4), 7.33-7.22 (10H, m, Ph), 4.67-4.61 (2H, m, H-4'), 4.46 (2H, t, *J* = 8.8 Hz, H-5'A), 4.26 (2H, dd, *J* = 8.8 Hz, *J* = 7.6 Hz, H-5'B), 3.27 (2H, dd, *J* = 13.6 Hz, *J* = 5.2 Hz, CH_AH_BPh), 2.74 (2H, dd, *J* = 13.6 Hz, *J* = 8.8 Hz, CH_AH_BPh); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 162.6 (C, C-2'), 146.7 (C, C-2), 137.6 (C, Ph), 137.3 (CH, C-4), 129.1 (CH, Ph), 128.5 (CH, Ph), 126.5 (CH, Ph), 125.7 (CH, C-3), 72.5 (CH_2, C-5'), 68.0 (CH, C-4'), 41.6 (CH_2, CH_2Ph).

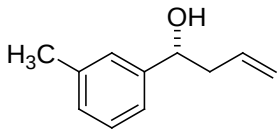
Representative procedure for asymmetric allylation of aldehydes

To an oven dried 10 mL round-bottom flask equipped with a magnetic stirring bar was added $Ce(OTf)_3$ (23.5 mg, 0.04 mmol), 4Å molecular sieve (80 mg) and anhydrous CH_2Cl_2 (1 mL). Pybox-1 (26.7 mg, 0.044 mmol) in CH_2Cl_2 (0.1 mL) was added and the mixture was stirred under nitrogen atmosphere at room temperature for 2 hours to afford a light white suspension. A mixture of benzaldehyde (21.2 mg, 0.2 mmol) and TMSCl (30.4 μL, 0.24 mmol) in CH_2Cl_2 (0.2 mL) was added to the resulting suspension. The mixture was then cooled to 0 ^oC followed by the addition of allyltrimethylsilane (63.5 μL, 0.4 mmol). The reaction mixture was stirred at room temperature for 30 h, then was quenched with saturated sodium bicarbonate solution (2 mL). The mixture was

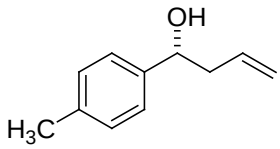
extracted with CH_2Cl_2 (3×10 mL), washed with brine, dried over Na_2SO_4, filtered and concentrated in vacuo. The residual crude product was purified via silica gel (20:1 hexane:EA) chromatography to afford the homoallylic alcohol as colorless oil.



Compound **2a**, (*R*)-1-phenylbut-3-en-1-ol, $C_{10}H_{12}O$, M=148.2, colorless viscous oil, $[\alpha]^{25}_D$ +50° (*c* 0.100, $CHCl_3$) [7]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.40-7.35 (3H, m, Ph), 7.32-7.28 (2H, m, Ph), 5.87-5.79 (1H, m, H-3), 5.22-5.15 (2H, m, H-4), 4.76 (1H, dd, *J* = 7.6 Hz, *J* = 7.2 Hz, H-1), 2.59-2.48 (2H, m, H-2), 2.08 (1H, brs, OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 143.8 (C, Ph), 134.4 (CH, C-3), 128.4 (CH, Ph), 127.5 (CH, Ph), 125.8 (CH, Ph), 118.4(CH_2, C-4), 73.3 (CH, C-1), 43.8 (CH_2, C-2). Enantiomeric excess was determined by HPLC (Chiracel OD-H, 99:1 hexanes: isopropanol, 1.0 mL/min, 230 nm): t_r (major) = 18.9 min, t_r (minor) = 21.4 min.

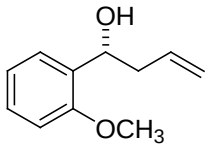


Compound **2b**, (*R*)-1-*m*-tolylbut-3-en-1-ol, $C_{11}H_{14}O$, M=162.2, colorless viscous oil, $[\alpha]^{25}_D$ +40° (*c* 0.100, CH_2Cl_2) [8]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.24 (1H, dd, *J* = 7.6 Hz, *J* = 7.2 Hz, Ar), 7.19-7.14 (2H, m, Ar), 7.09 (1H, d, *J* = 7.6 Hz, Ar), 5.85-5.77 (1H, m, H-3), 5.30-5.13 (2H, m, H-4), 4.71 (1H, dd, *J* = 8.0 Hz, *J* = 5.6 Hz, H-1), 2.54-2.48 (2H, m, H-2), 2.37 (3H, s, CH_3), 2.04 (1H, s, OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 143.9 (C, Ar), 138.1 (C, Ar), 134.6 (CH, C-3), 128.4 (CH, Ar), 128.3 (CH, Ar), 126.5 (CH, Ar), 122.9 (CH, Ar), 118.3 (CH_2, C-4), 73.4 (CH, C-1), 43.8 (CH_2, C-2), 21.5 (CH_3). Enantiomeric excess was determined by HPLC (Chiracel OD-H, 98:2 hexanes:isopropanol, 0.6 mL/min, 254 nm): t_r (major) = 16.5 min, t_r (minor) = 22.1 min.

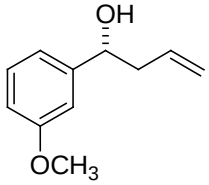


Compound **2c**, (*R*)-1-*p*-tolylbut-3-en-1-ol, $C_{11}H_{14}O$, M=162.2, colorless viscous oil, $[\alpha]^{25}_D$ +60° (*c* 0.100, CH_2Cl_2) [8]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.25 (2H, dd, *J* = 6.0 Hz, *J* = 2.0 Hz, Ar), 7.16 (2H, dd, *J* = 6.0 Hz, *J* = 2.0 Hz, Ar), 5.86-5.76 (1H, m, H-3), 5.19-5.11 (2H, m, H-4), 4.69 (1H, t, *J* = 6.4 Hz, H-1), 2.52-2.48 (2H, m, H-2), 2.35 (3H, s, CH_3), 2.04 (1H, brs, OH). ^{13}C

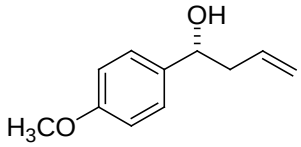
NMR (100 MHz, $CDCl_3$): δ (ppm) 140.9 (C, Ar), 137.1 (C, Ar), 134.6 (CH, C-3), 129.0 (CH, Ar), 125.7 (CH, Ar), 118.1 (CH_2, C-4), 73.2 (CH, C-1), 43.7 (CH_2, C-2), 21.1 (CH_3). Enantiomeric excess was determined by HPLC (Chiracel AS-H, 98:2 hexanes:isopropanol, 0.7 mL/min, 254 nm): t_r (major) = 14.1 min, t_r (minor) = 15.8 min.



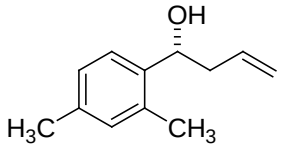
Compound **2d**, (*R*)-1-(2-methoxyphenyl)but-3-en-1-ol, $C_{11}H_{14}O_2$, M=178.2, colorless viscous oil, $[\alpha]^{25}_D$ +42° (*c* 0.100, CH_2Cl_2) [8]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.33 (1H, dd, *J* = 7.6 Hz, *J* = 1.6 Hz, Ar), 7.26-7.22 (1H, m, Ar), 6.96 (1H, td, *J* = 7.6 Hz, *J* = 1.2 Hz, Ar), 6.87 (1H, dd, *J* = 8.0 Hz, *J* = 1.2 Hz, Ar), 5.90-5.79 (1H, m, H-3), 5.16-5.08 (2H, m, H-4), 4.98-4.94 (1H, m, H-1), 3.85 (3H, s, OCH_3), 2.62-2.46 (3H, m, H-2 and OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 156.3 (C, Ar), 135.2 (CH, C-3), 131.7 (C, Ar), 128.2 (CH, Ar), 126.8 (CH, Ar), 120.6 (CH, Ar), 117.5 (CH_2, C-4), 110.4 (CH, Ar), 69.6 (CH, C-1), 55.2 (OCH_3), 41.8 (CH_2, C-2). Enantiomeric excess was determined by HPLC (Chiracel OD-H, 98:2 hexanes:isopropanol, 0.8 mL/min, 254 nm): t_r (minor) = 16.8 min, t_r (major) = 18.5 min.



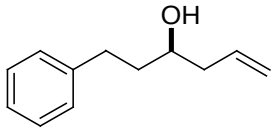
Compound **2e**, (*R*)-1-(3-methoxyphenyl)but-3-en-1-ol, $C_{11}H_{14}O_2$, M=178.2, colorless viscous oil, $[\alpha]^{25}_D$ +48° (*c* 0.100, CH_2Cl_2) [8]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.25 (1H, t, *J* = 8.0 Hz, Ar), 6.94-6.91 (2H, m, Ar), 6.81 (1H, qd, *J* = 8.0 Hz, *J* = 2.8 Hz, *J* = 1.2 Hz, Ar), 5.85-5.75 (1H, m, H-3), 5.19-5.12 (2H, m, H-4), 4.70 (1H, dd, *J* = 8.0 Hz, *J* = 1.6 Hz, H-1), 3.81 (3H, s, OCH_3), 2.54-2.46 (2H, m, H-2), 2.09 (1H, brs, OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 159.7 (C, Ar),145.6 (C, Ar), 134.4(CH, C-3), 129.4 (CH, Ar), 118.3 (CH, Ar), 118.0 (CH_2, C-4), 112.9 (CH, Ar), 111.3 (CH, Ar), 73.2 (CH, C-1), 55.2 (OCH_3), 43.7 (CH_2, C-2). Enantiomeric excess was determined by HPLC (Chiracel OD-H, 98:2 hexanes:isopropanol, 1.0 mL/min, 254 nm): t_r (major) = 26.7 min, t_r (minor) = 34.2 min.



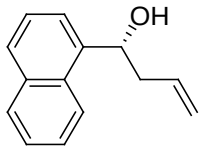
Compound **2f**, (*R*)-1-(4-methoxyphenyl)but-3-en-1-ol, $C_{11}H_{14}O_2$, M=178.2, colorless viscous oil, $[\alpha]^{25}_D$ +56° (*c* 0.100, $CHCl_3$) [7]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.28 (1H, dd, *J* = 6.8 Hz, *J* = 2.0 Hz, Ar), 6.88 (1H, dd, *J* = 6.8 Hz, *J* = 2.0 Hz, Ar), 5.84-5.74 (1H, m, H-3), 5.18-5.11 (2H, m, H-4), 4.68 (1H, t, *J* = 6.4 Hz, H-1), 3.80 (3H, s, OCH_3), 2.52-2.48 (2H, m, H-2), 2.02 (1H, brs, OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 159.0 (C, Ar), 136.0 (C, Ar), 134.6 (CH, C-3), 127.0 (CH, Ar), 118.2 (CH_2, C-4), 113.8 (CH, Ar), 72.9 (CH, C-1), 55.2 (OCH_3), 43.7 (CH_2, C-2). Enantiomeric excess was determined by HPLC (Chiracel OD-H, 95:5 hexanes:isopropanol, 0.8 mL/min, 230 nm): t_r (minor) = 13.1 min, t_r (major) = 13.9 min.



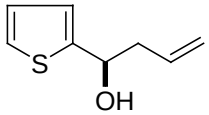
Compound **2g**, (*R*)-1-(2,4-dimethylphenyl)but-3-en-1-ol, $C_{12}H_{16}O$, M=176.3, colorless viscous oil, $[\alpha]^{25}_D$ +63° (*c* 0.100, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.36 (1H, d, *J* = 8.0 Hz, Ar), 7.04 (1H, d, *J* = 8.0 Hz, Ar), 6.96 (1H, s, Ar), 5.91-5.80 (1H, m, H-3), 5.20-5.13 (2H, m, H-4), 5.94 (1H, m, H-1), 2.52-2.41 (2H, m, H-2), 2.31 (3H, s, CH_3), 2.30 (3H, s, CH_3), 1.89 (1H, brs, OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 138.9 (C, Ar), 136.8 (C, Ar), 134.9 (CH, C-3), 134.3 (C, Ar), 131.1 (CH, Ar), 126.9 (CH, Ar), 125.2 (CH, Ar), 118.1 (CH_2, C-4), 69.6 (CH, C-1), 42.6 (CH_2, C-2), 20.9 (CH_3), 18.9 (CH_3). Enantiomeric excess was determined by HPLC (Chiracel OD-H, 95:5 hexanes:isopropanol, 0.8 mL/min, 220 nm): t_r (major) = 8.0 min, t_r (minor) = 8.8 min.



Compound **2h**, (*R*)-1-phenylhex-5-en-3-ol, $C_{12}H_{16}O$, M=176.3, colorless viscous oil, $[\alpha]^{25}_D$ +4° (*c* 0.100, $CHCl_3$) [9]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.31-7.25 (2H, m, Ph), 7.22-7.17 (3H, m, Ph), 5.87-5.77 (1H, m, H-5), 5.17-5.13 (2H, m, H-6), 3.68 (1H, m, H-3), 2.85-2.78 (1H, m, H-1A), 2.73-2.65 (1H, m, H-1B), 2.36-2.29 (1H, m, H-4A), 2.22-2.14 (1H, m, H-4B), 1.82-1.76 (2H, m, H-2), 1.64 (1H, s, OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 142.0 (C, Ph), 134.6 (CH, C-5), 128.4 (CH, Ph), 128.3 (CH, Ph), 125.8 (CH, Ph), 118.3 (CH_2, C-6), 69.9 (CH, C-3), 42.0 (CH_2, C-4), 38.4 (CH_2, C-2), 32.0 (CH_2, C-1). Enantiomeric excess was determined by HPLC (Chiracel OD-H, 95:5 hexanes:isopropanol, 1.0 mL/min, 254 nm): t_r (major) = 5.5 min, t_r (minor) = 10.5 min.



Compound **2i**, (*R*)-1-(naphthalen-5-yl)but-3-en-1-ol, $C_{14}H_{14}O$, M=198.3, colorless viscous oil, $[\alpha]^{25}_D$ +176° (c 0.100, $CHCl_3$) [7]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.08 (1H, d, *J* = 8.0 Hz, Ar), 7.88 (1H, dd, *J* = 7.6 Hz, *J* = 1.6 Hz, Ar), 7.79 (1H, d, *J* = 8.4 Hz, Ar), 7.67 (1H, d, *J* = 7.2 Hz, Ar), 7.56-7.47 (3H, m, Ar), 5.99-5.89 (1H, m, H-3), 5.53 (1H, m, H-1), 5.25-5.18 (2H, m, H-4), 2.80-2.74 (1H, m, H-2A), 2.65-2.58 (1H, m, H-2B), 2.28 (1H, d, *J* = 2.8 Hz, OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 139.4 (C, Ar), 134.7 (CH, C-3), 133.7 (C, Ar), 130.2 (C, Ar), 128.9 (CH, Ar), 127.9 (CH, Ar), 125.9 (CH, Ar), 125.4 (CH, Ar), 125.3 (CH, Ar), 122.9 (CH, Ar), 122.8 (CH, Ar), 118.2 (CH_2, C-4), 69.9 (CH, C-1), 42.8 (CH_2, C-2). Enantiomeric excess was determined by HPLC (Chiracel OD-H, 90:10 hexanes:isopropanol, 1.0 mL/min, 254 nm): t_r (major) = 8.1 min, t_r (minor) = 13.9 min.



Compound **2j**, (*R*)-1-(thiophen-2-yl)but-3-en-1-ol, $C_8H_{10}OS$, M=154.2, colorless viscous oil, $[\alpha]^{25}_D$ +38° (c 0.100, CH_2Cl_2) [8]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.25 (1H, dd, *J* = 4.8 Hz, *J* = 1.6 Hz, Ar), 6.99-6.96 (2H, m, Ar), 5.88-5.78 (1H, m, H-3), 5.22-5.14 (2H, m, H-4), 5.05-4.96 (1H, m, H-1), 2.64-2.60 (2H, m, H-2), 2.34 (1H, d, *J* = 4.0 Hz, OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 147.8 (C, Ar), 133.8 (CH, C-3), 126.6 (CH, Ar), 124.5 (CH, Ar), 123.7 (CH, Ar), 118.8 (CH_2, C-4), 69.3 (CH, C-1), 43.7 (CH_2, C-2). Enantiomeric excess was determined by HPLC (Chiracel OD-H, 97:3 hexanes:isopropanol, 0.6 mL/min, 220 nm): t_r (major) = 19.8 min, t_r (minor) = 21.5 min.

References:

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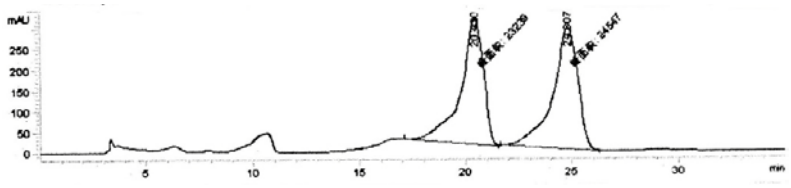
Copys of HPLC analysis of homoallylic alcohols

Compound **2a**: (*R*)-1-phenylbut-3-en-1-ol



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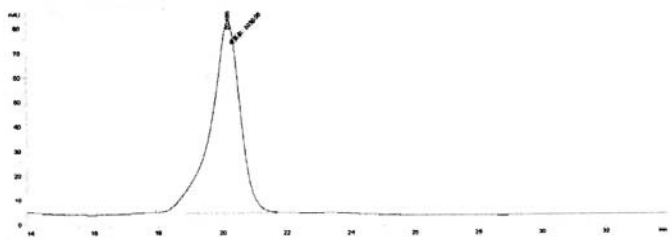
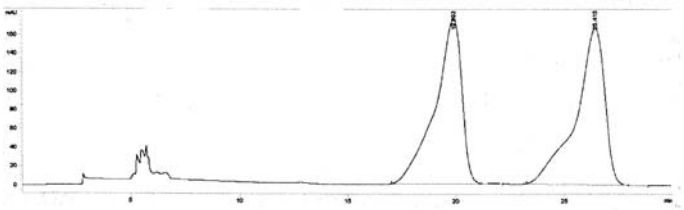
2	21.353	24547	295.4	73.5311



Peak	Ret. Time	Area	Height	Area %
1	20.186	2944.3	78.3	100.000

Compound **2b**: (*R*)-1-*m*-tolylbut-3-en-1-ol



Peak	Ret. Time	Area	Height	Area %
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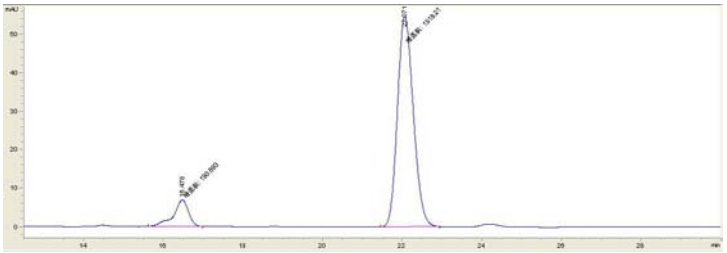
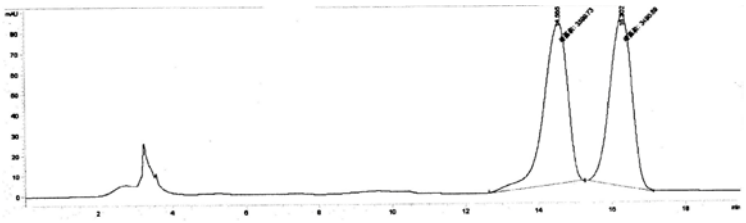


Peak	Ret. Time	Area	Height	Area %
1	16.478	190.7	7	11.159
2	22.071	1518.2	54.7	88.841

Compound **2c**: (*R*)-1-*p*-tolylbut-3-en-1-ol



Peak	Ret. Time	Area	Height	Area %
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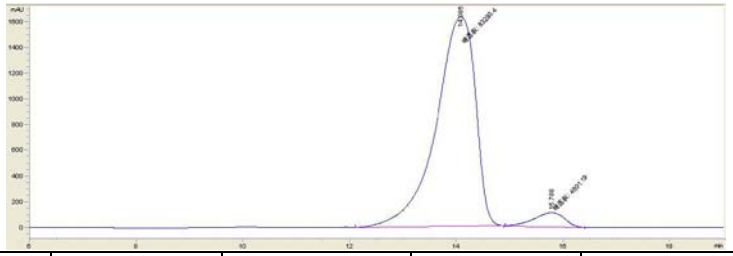
2	16.302	3490.9	84.3	49.232



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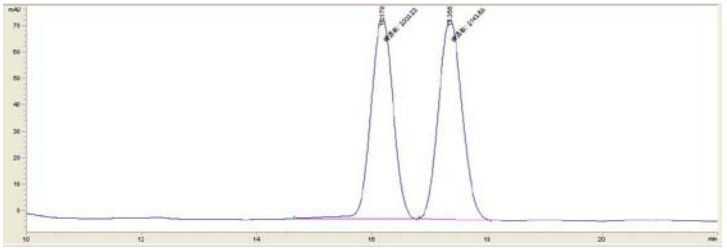
1	14.085	83293.4	1627.8	94.765
2	15.788	4601.2	113	5.235

Compound **2d**: (*R*)-1-(2-methoxyphenyl)but-3-en-1-ol



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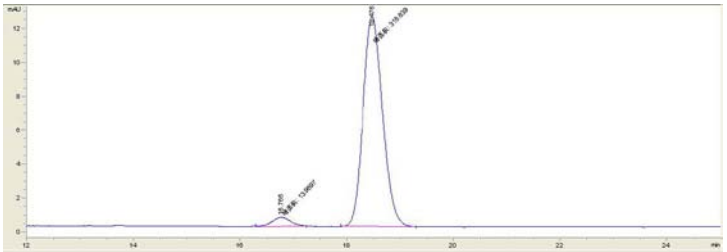

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1	16.765	14	5.5E-1	4.225
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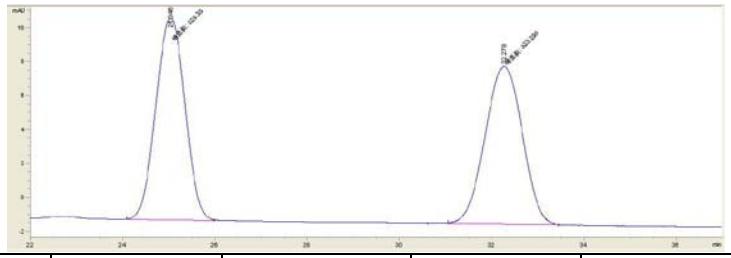
Compound **2e**: (*R*)-1-(3-methoxyphenyl)but-3-en-1-ol



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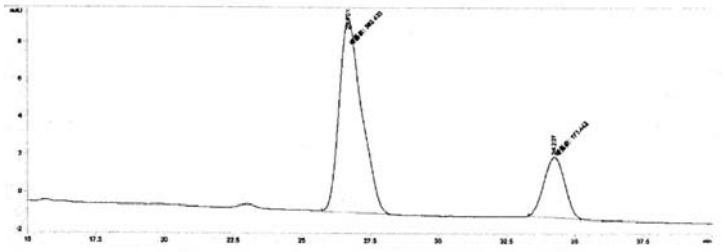
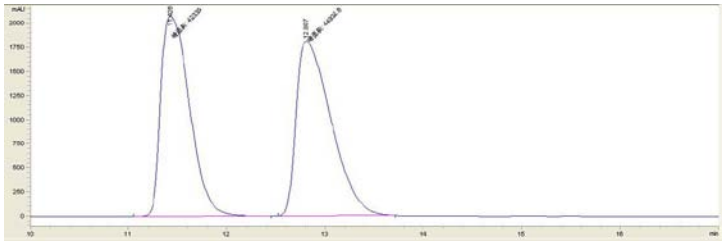


Peak	Ret. Time	Area	Height	Area %
1	26.701	592.4	10.3	77.556
2	34.237	171.4	3.2	22.444

Compound **2f**: (*R*)-1-(4-methoxyphenyl)but-3-en-1-ol



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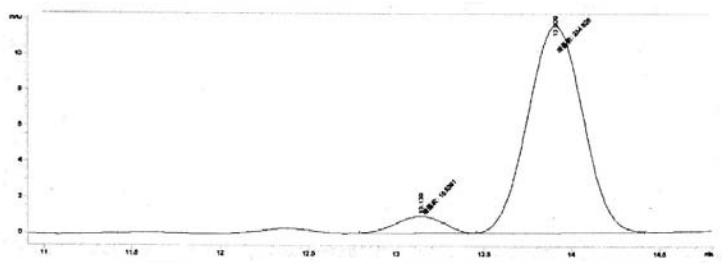
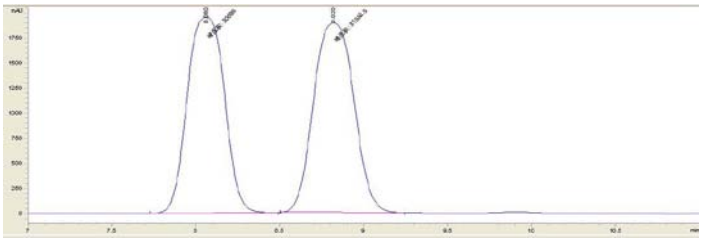


Peak	Ret. Time	Area	Height	Area %
1	13.139	18.5	9.7E-1	6.787
2	13.909	254.6	11.6	93.213

Compound **2g**: (*R*)-1-(2,4-dimethylphenyl)but-3-en-1-ol



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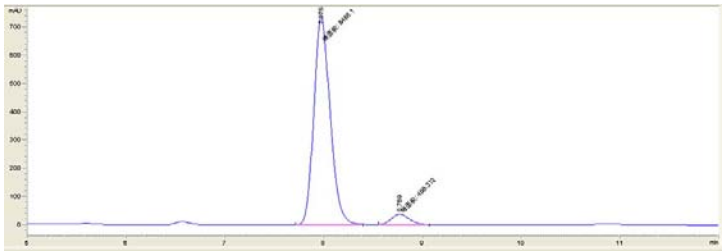
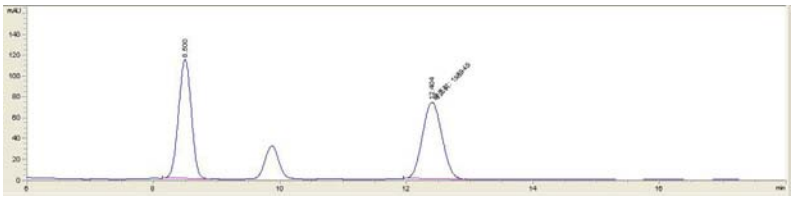


Peak	Ret. Time	Area	Height	Area %
1	7.975	8486.1	737	94.897
2	8.769	456.3	37	5.103

Compound **2h**: (*R*)-1-phenylhex-5-en-3-ol



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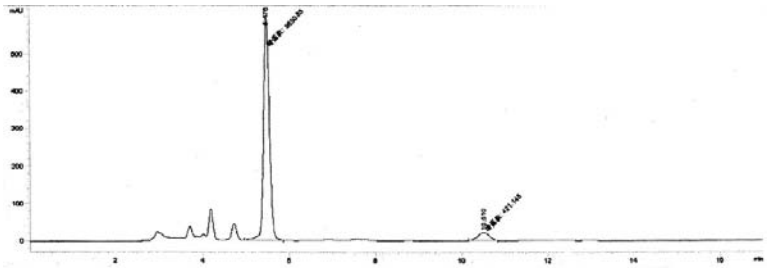

Peak	Ret. Time	Area	Height	Area %
1	5.476	5650.8	594.1	93.064
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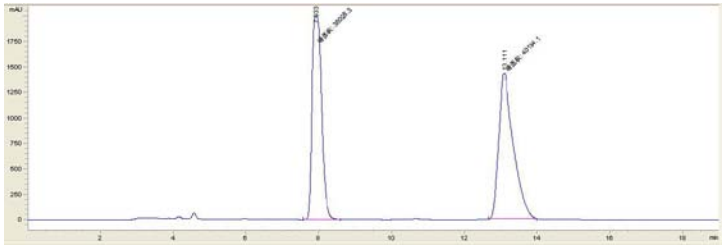
Compound **2i**: (*R*)-1-(naphthalen-5-yl)but-3-en-1-ol



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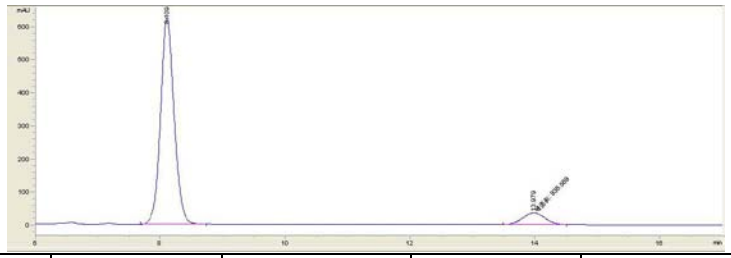

Peak	Ret. Time	Area	Height	Area %
1	8.109	9374.3	626.4	90.917
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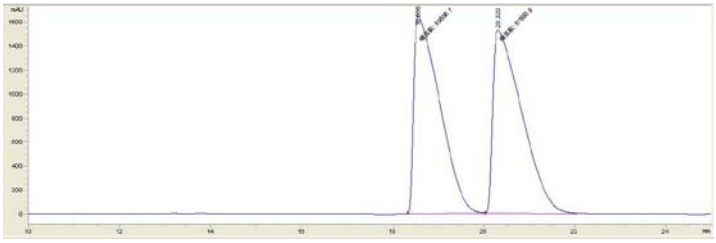
Compound **2j**: (*R*)-1-(thiophen-2-yl)but-3-en-1-ol



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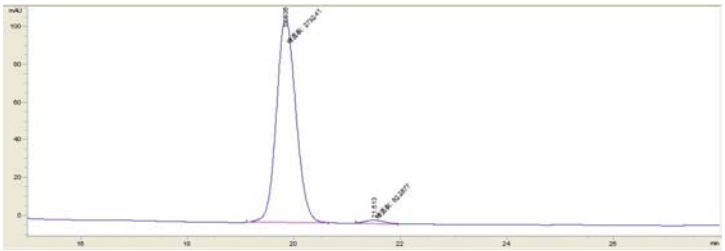

Peak	Ret. Time	Area	Height	Area %
1	19.836	2752.4	108.5	97.787
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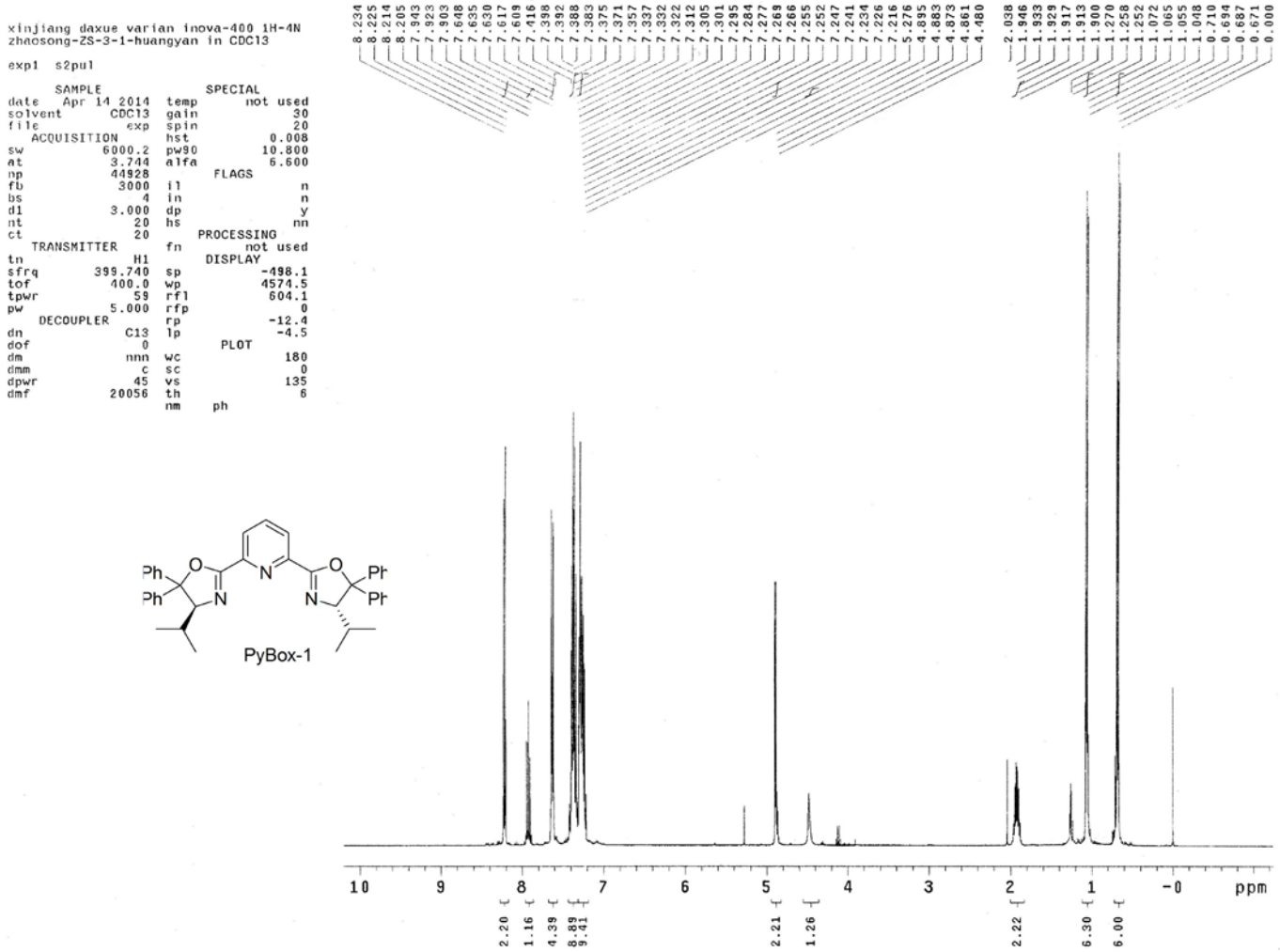
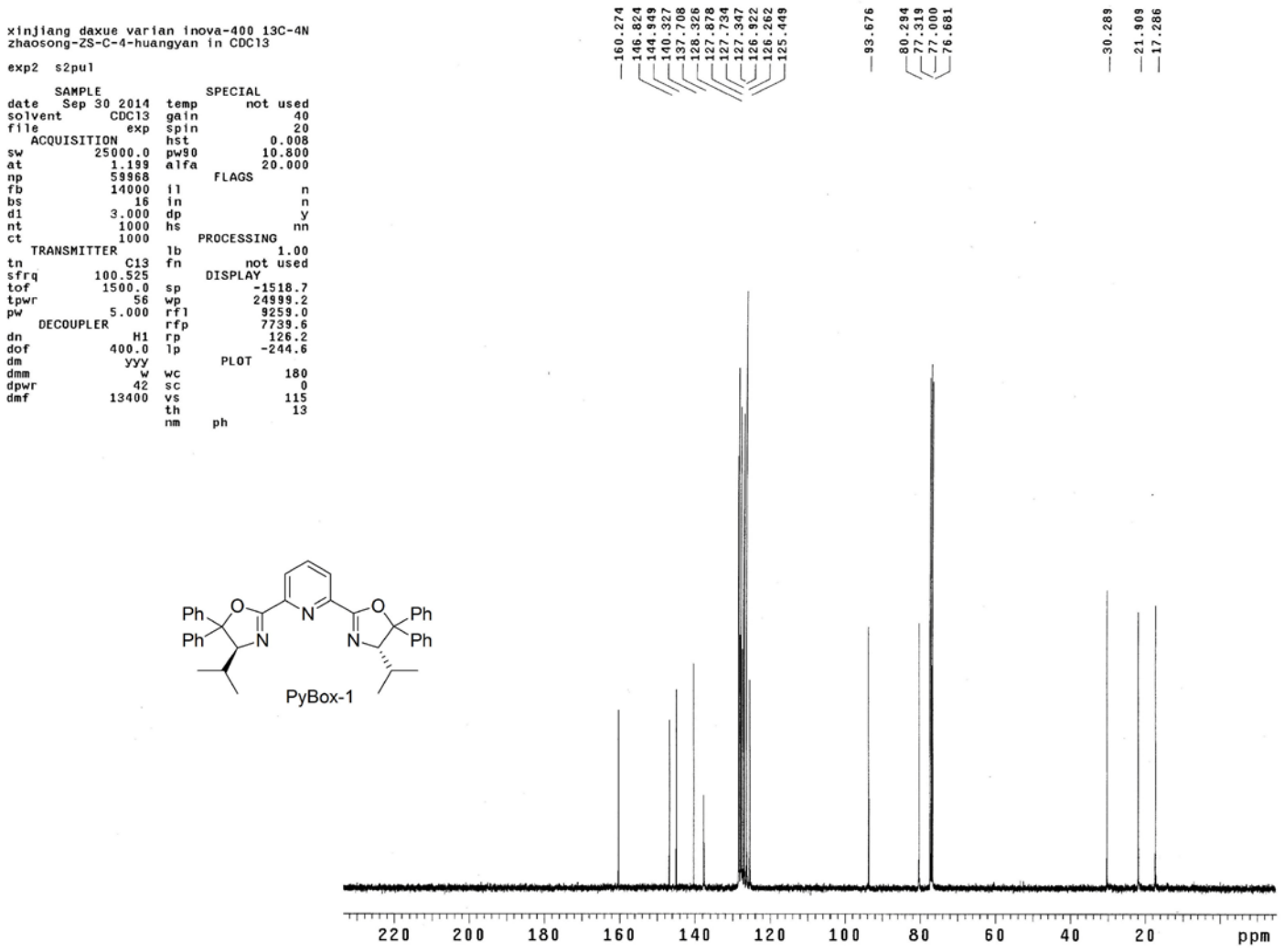
Copys of ^{1}H NMR and ^{13}C NMR spectra

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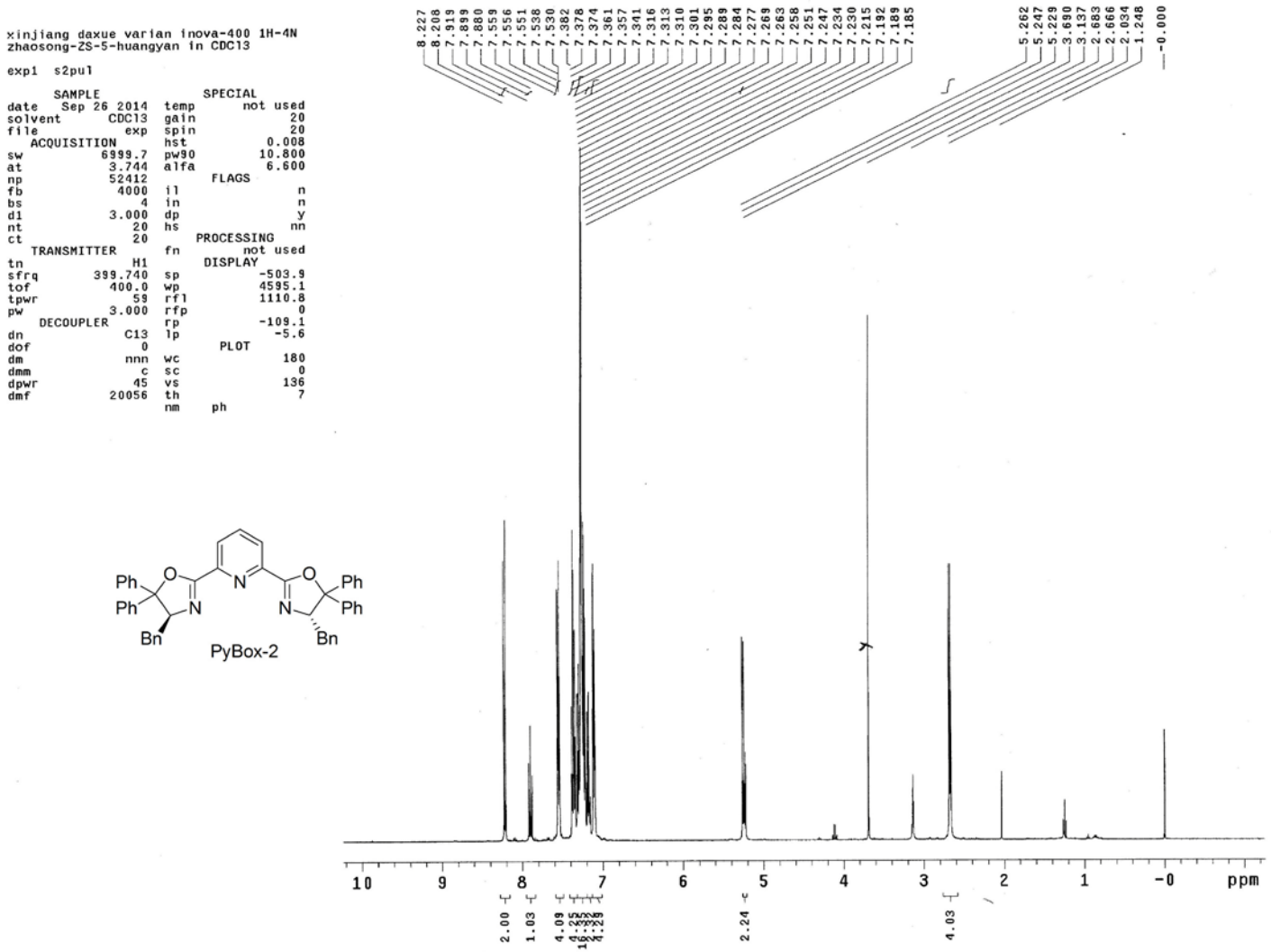
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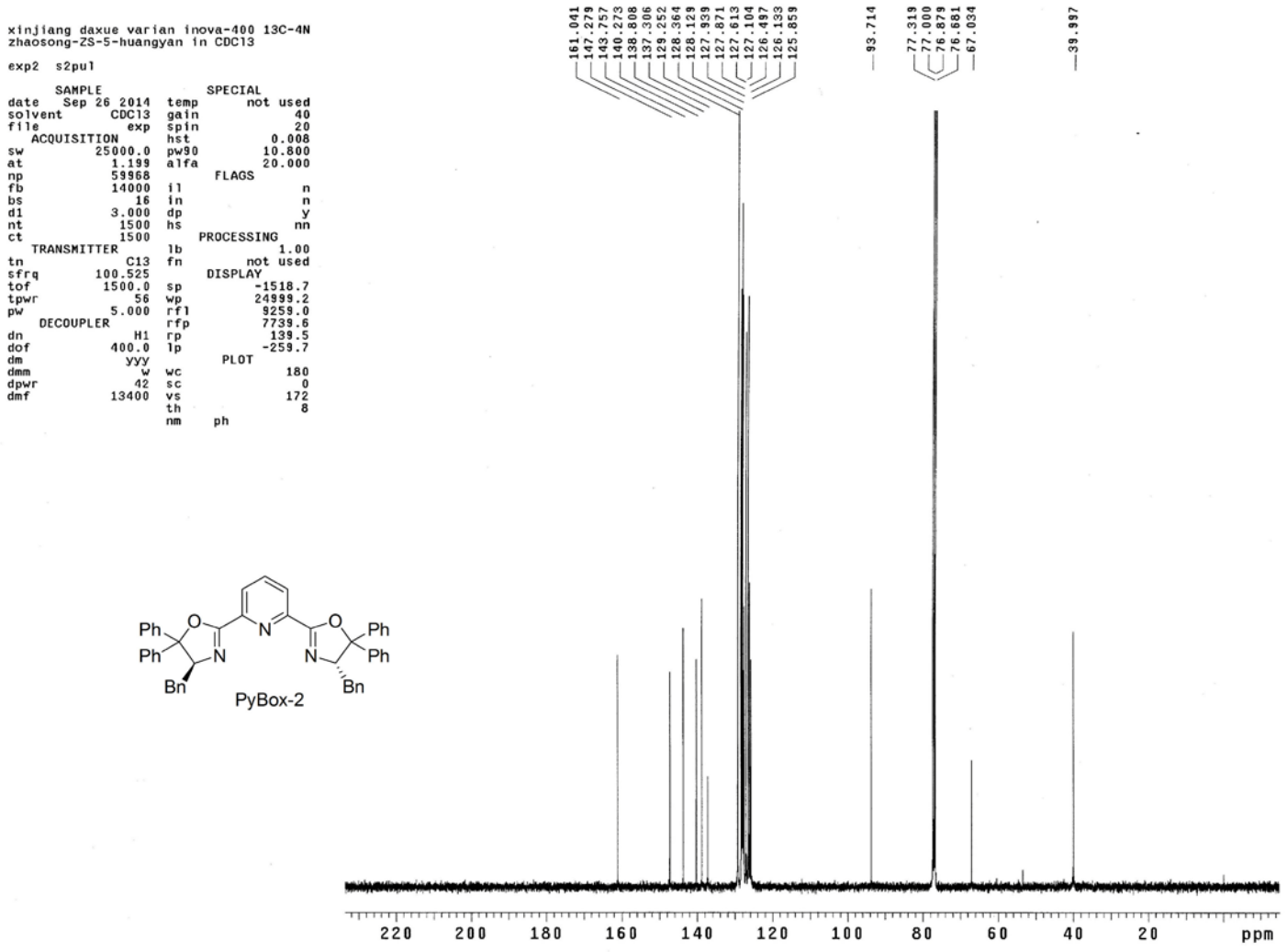
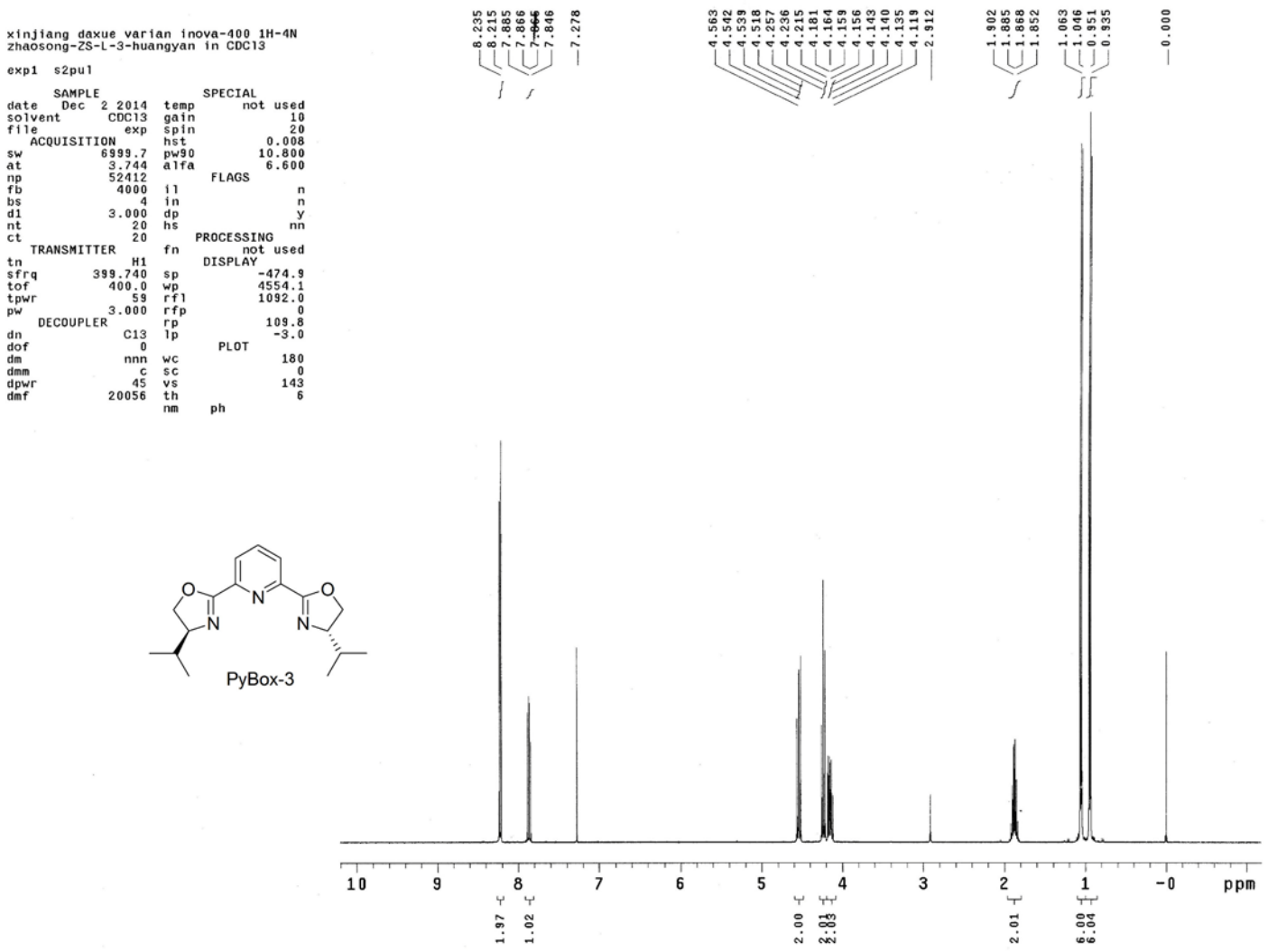
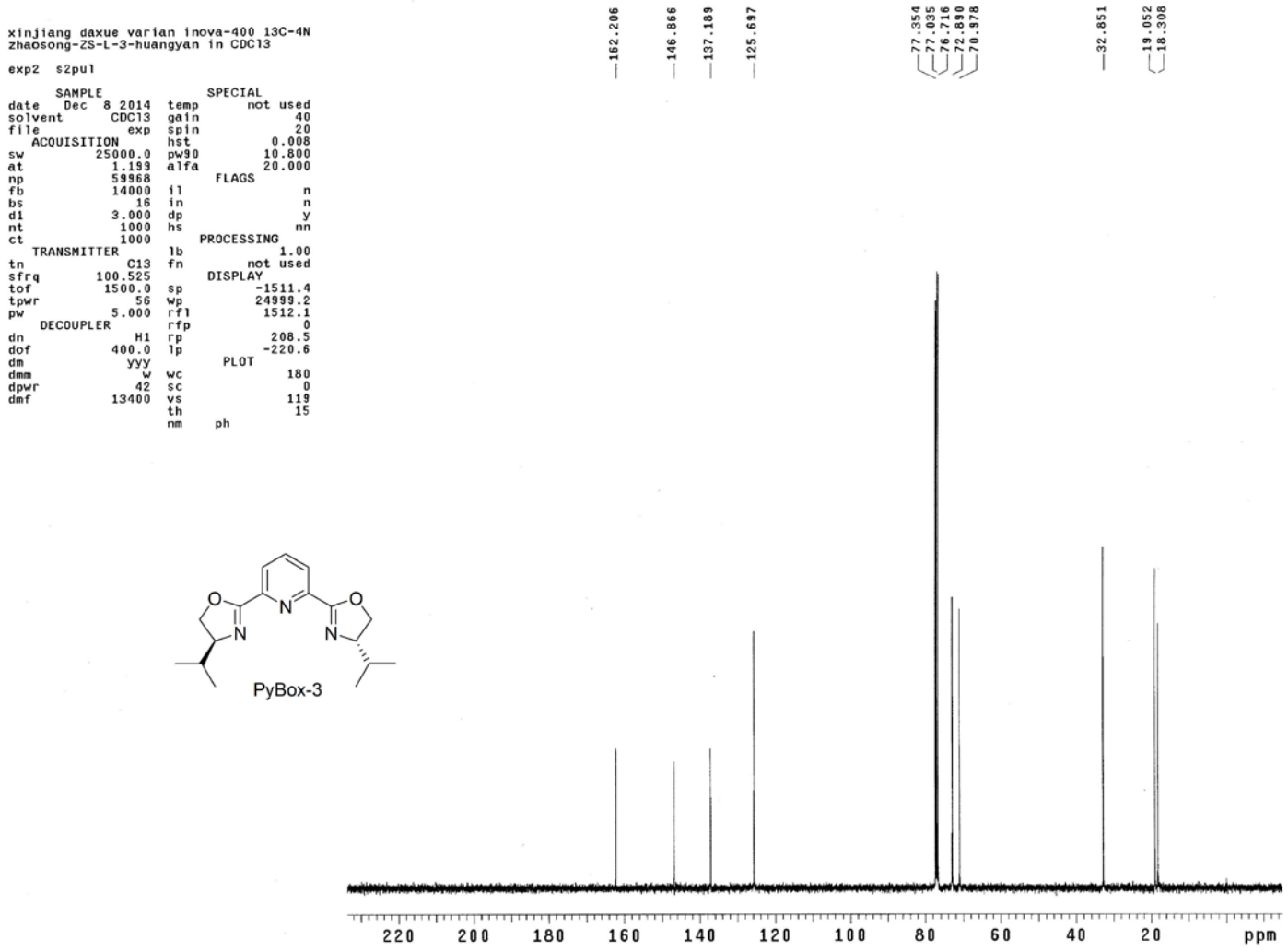
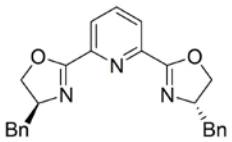
7.357
7.341
7.316
7.313
7.310
7.301
7.295
7.289
7.284
7.277
7.269
7.263
7.258
7.251
7.247
7.234
7.230
7.215
7.192
7.189
7.185
5.262
5.247
5.229
3.690
3.137
2.683
2.666
2.034
1.248
-0.000
Ph
Ph
O
N
N
O
Ph
Ph
Bn
Bn
PyBox-2
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.00
1.03
4.09
2.24
4.03


xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-5-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Sep 26 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 1500
ct 1500
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 40
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1518.7
wp 24999.2
rfl 9259.0
rfp 7739.6
rp 139.5
lp -259.7
PLOT
wc 180
sc 0
vs 172
th 8
nm ph
161.041
147.279
143.757
140.273
138.808
137.306
129.252
128.364
128.129
127.939
127.871
127.613
127.104
126.497
126.133
125.859
93.714
77.319
77.000
76.879
76.681
67.034
39.997
Ph
Ph
O
N
Bn
N
O
Ph
Ph
N
Bn
PyBox-2
220
200
180
160
140
120
100
80
60
40
20
ppm

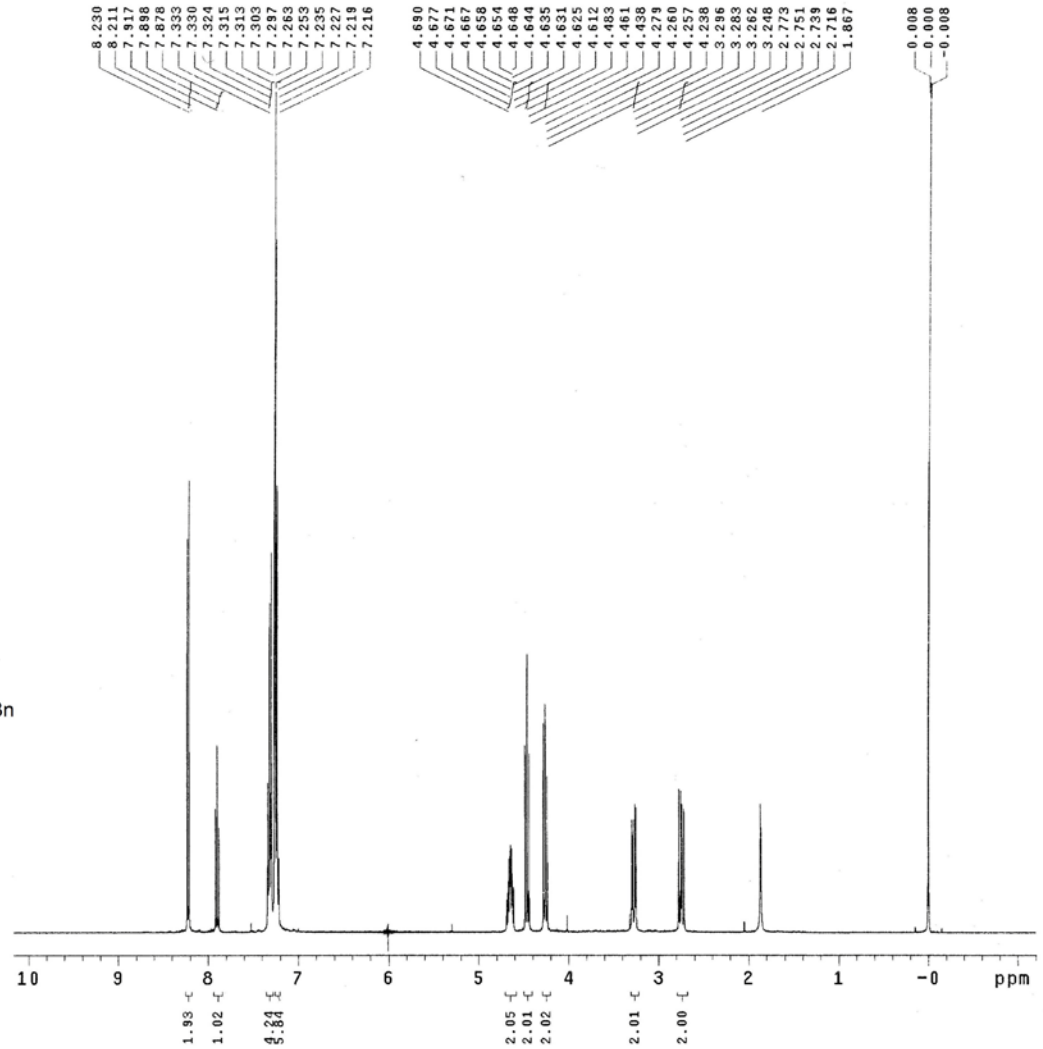

xinjiang daxue varian inova-400 1H-4N
zhaosong-ZS-L-3-huangyan in CDCl3
exp1 s2pul
SAMPLE
date Dec 2 2014
solvent CDCl3
file exp
ACQUISITION
sw 6999.7
at 3.744
np 52412
fb 4000
bs 4
d1 3.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 3.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 10
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -474.9
wp 4554.1
rfl 1092.0
rfp 0
rp 109.8
lp -3.0
PLOT
wc 180
sc 0
vs 143
th 6
nm ph
8.235
8.215
7.885
7.866
7.846
7.278
4.563
4.542
4.539
4.518
4.257
4.236
4.215
4.181
4.164
4.159
4.156
4.143
4.140
4.135
4.119
2.912
1.902
1.885
1.868
1.852
1.063
1.046
0.951
0.935
0.000
PyBox-3
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.97
1.02
2.00
2.01
2.03
2.01
6.00
6.04

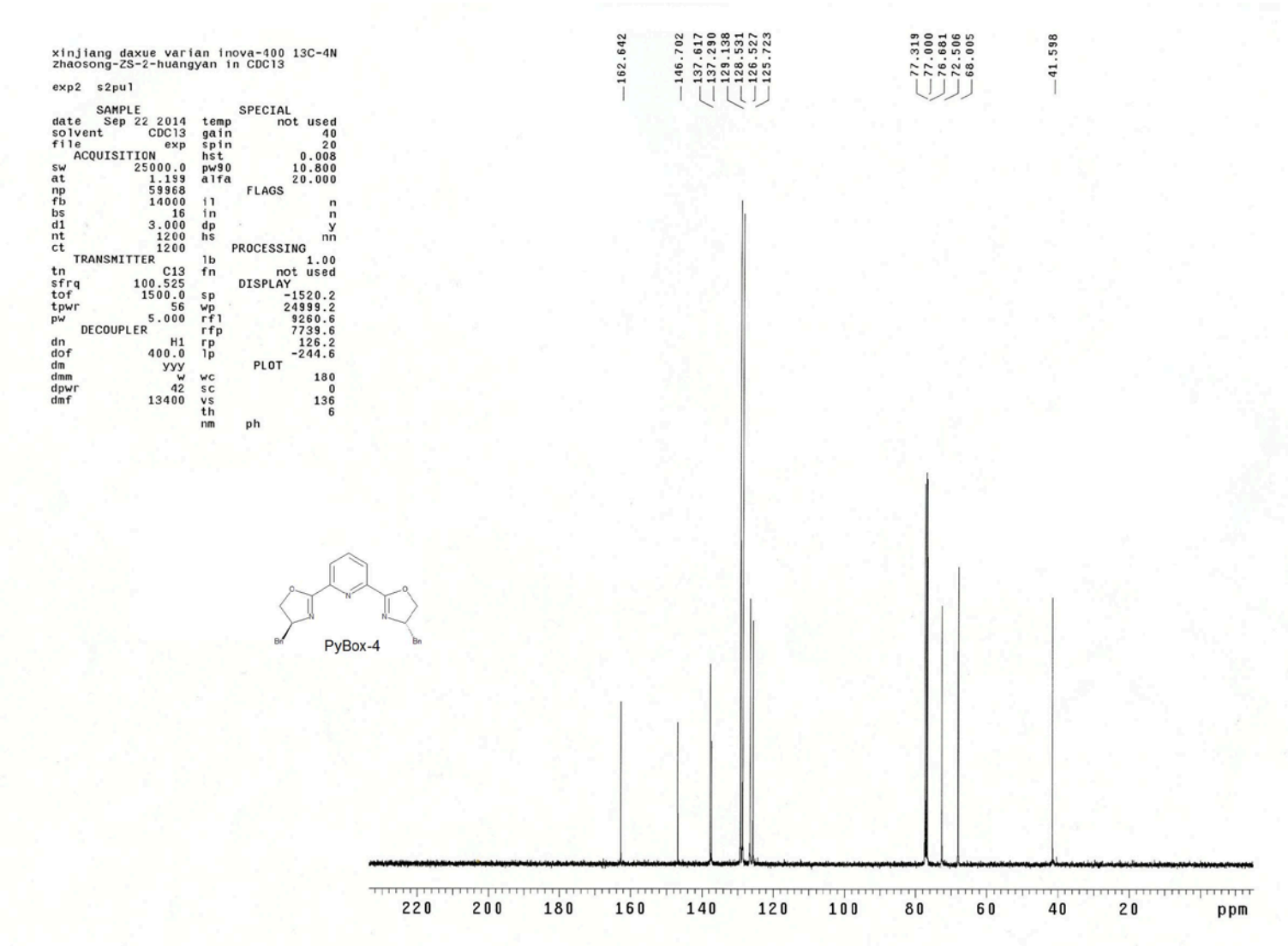

xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-L-3-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Dec 8 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 1000
ct 1000
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 40
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1511.4
wp 24998.2
rfl 1512.1
rfp 0
rp 208.5
lp -220.6
PLOT
wc 180
sc 0
vs 119
th 15
nm ph
162.206
146.866
137.189
125.697
77.354
77.035
76.716
72.830
70.978
32.851
19.052
18.308
O
N
N
O
PyBox-3
220
200
180
160
140
120
100
80
60
40
20
ppm

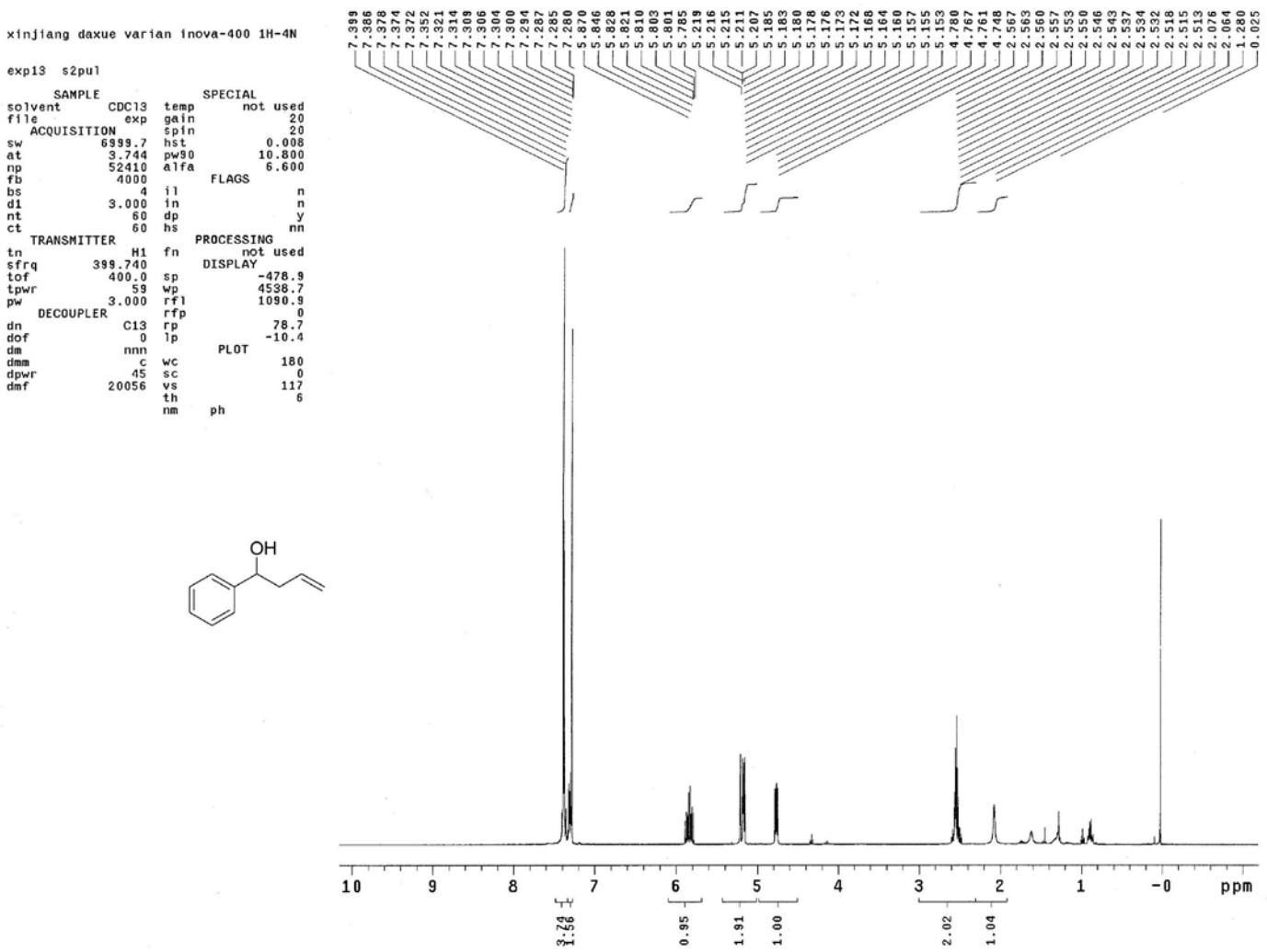
```
xinjiang daxue varian inova-400 1H-4N
zhaosong-ZS-1-huangyan in CDCl3

exp1  s2pul

      SAMPLE                SPECIAL
date    Nov 13 2013   temp        not used
solvent       CDCl3   gain              30
file            exp   spin              20
   ACQUISITION        hst            0.008
sw             6000.2  pw90           10.800
at              3.744  alfa            6.600
np             44928          FLAGS
fb              3000   il                n
bs                 4   in                n
d1             3.000   dp                y
nt               100   hs               nn
ct               100        PROCESSING
   TRANSMITTER        fn        not used
tn                H1         DISPLAY
sfrq         399.740   sp           -482.9
tof            400.0   wp           4545.7
tpwr              59   rf1           598.6
pw             3.000   rfp               0
    DECOUPLER         rp            -10.4
dn                C13   lp             -7.4
dof                 0          PLOT
dm                nnn   wc              180
dmm                 c   sc                0
dpwr               45   vs              309
dmf             20056   th                5
                       nm    ph
```

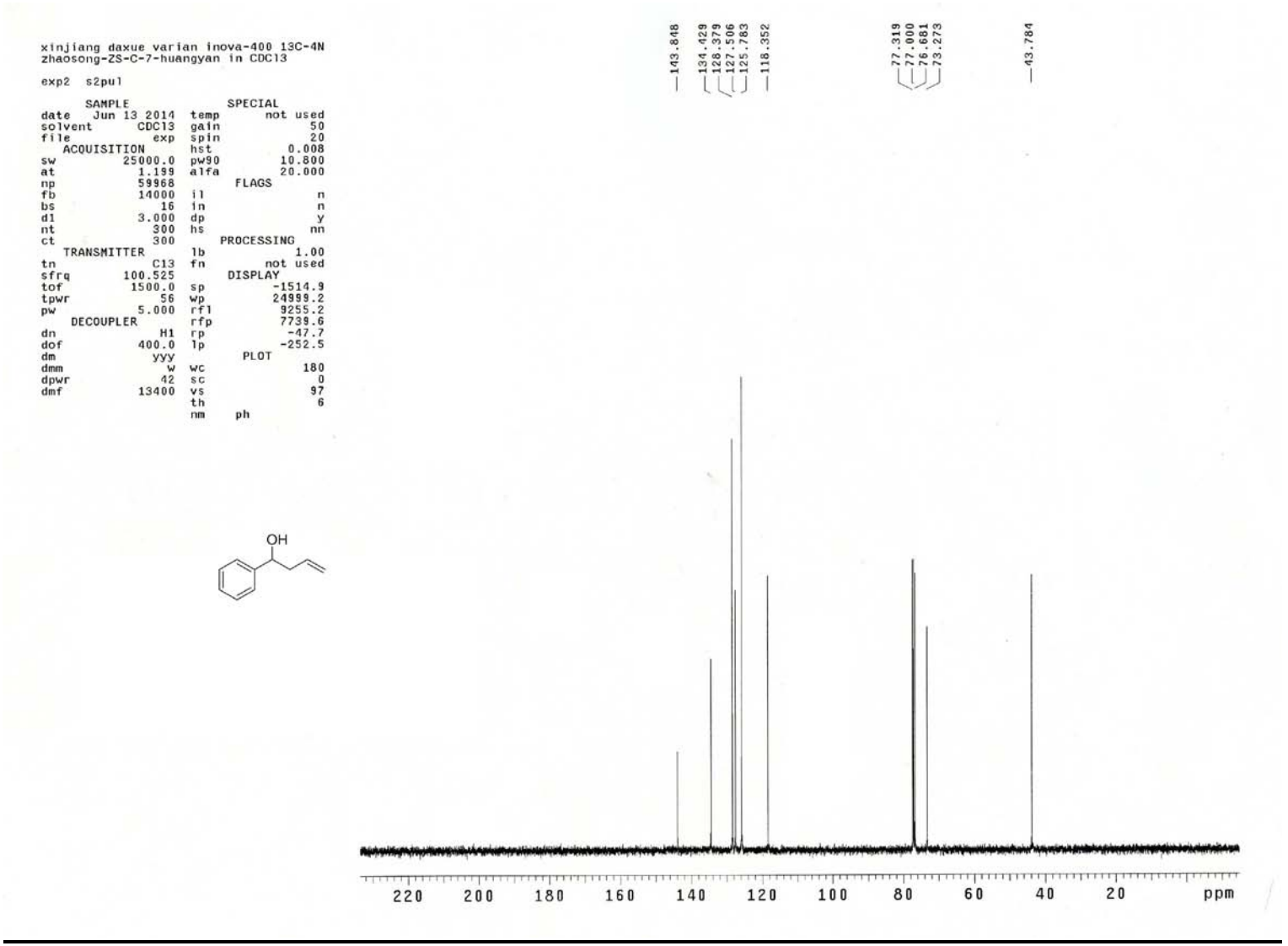


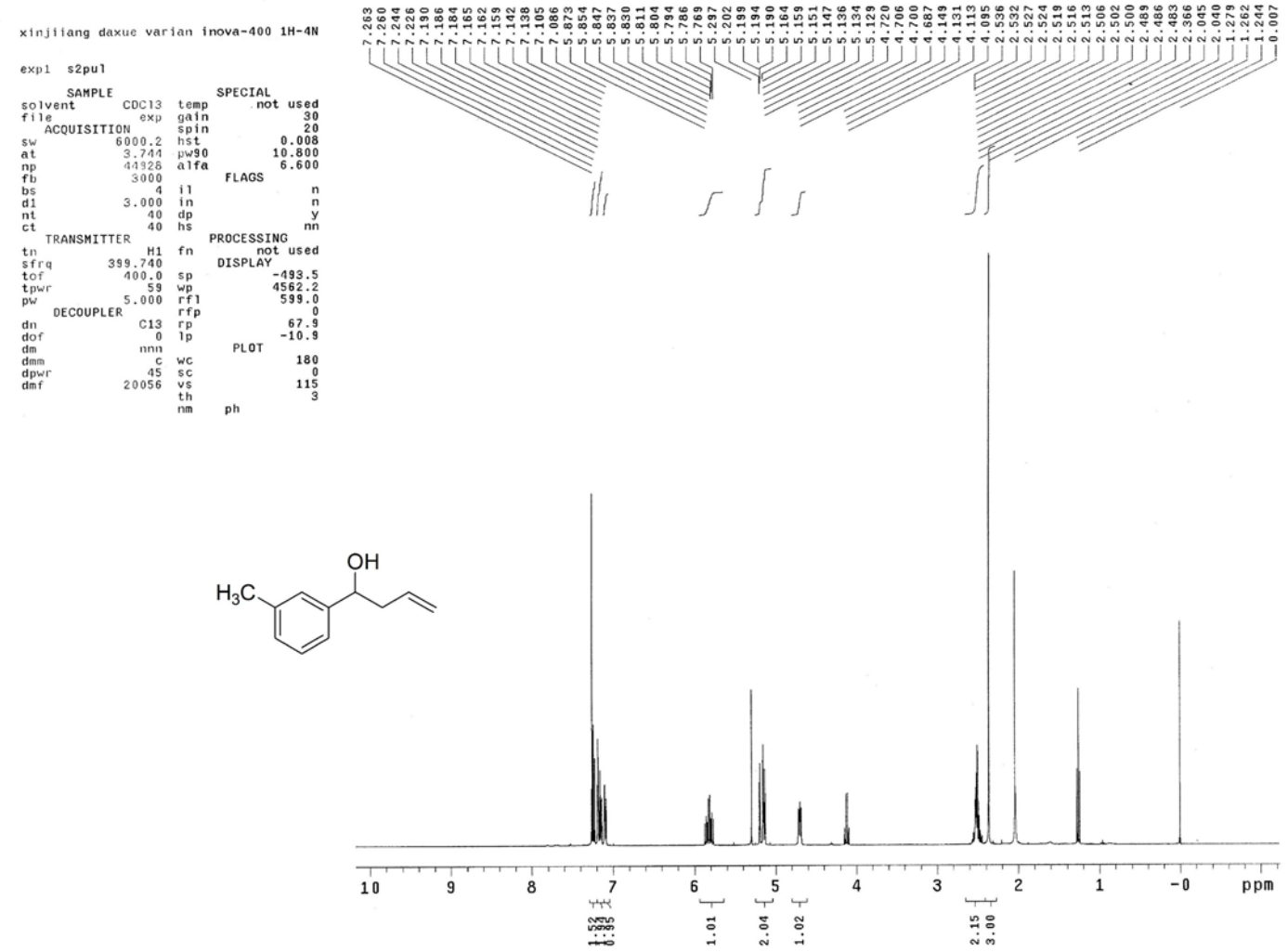
PyBox-4

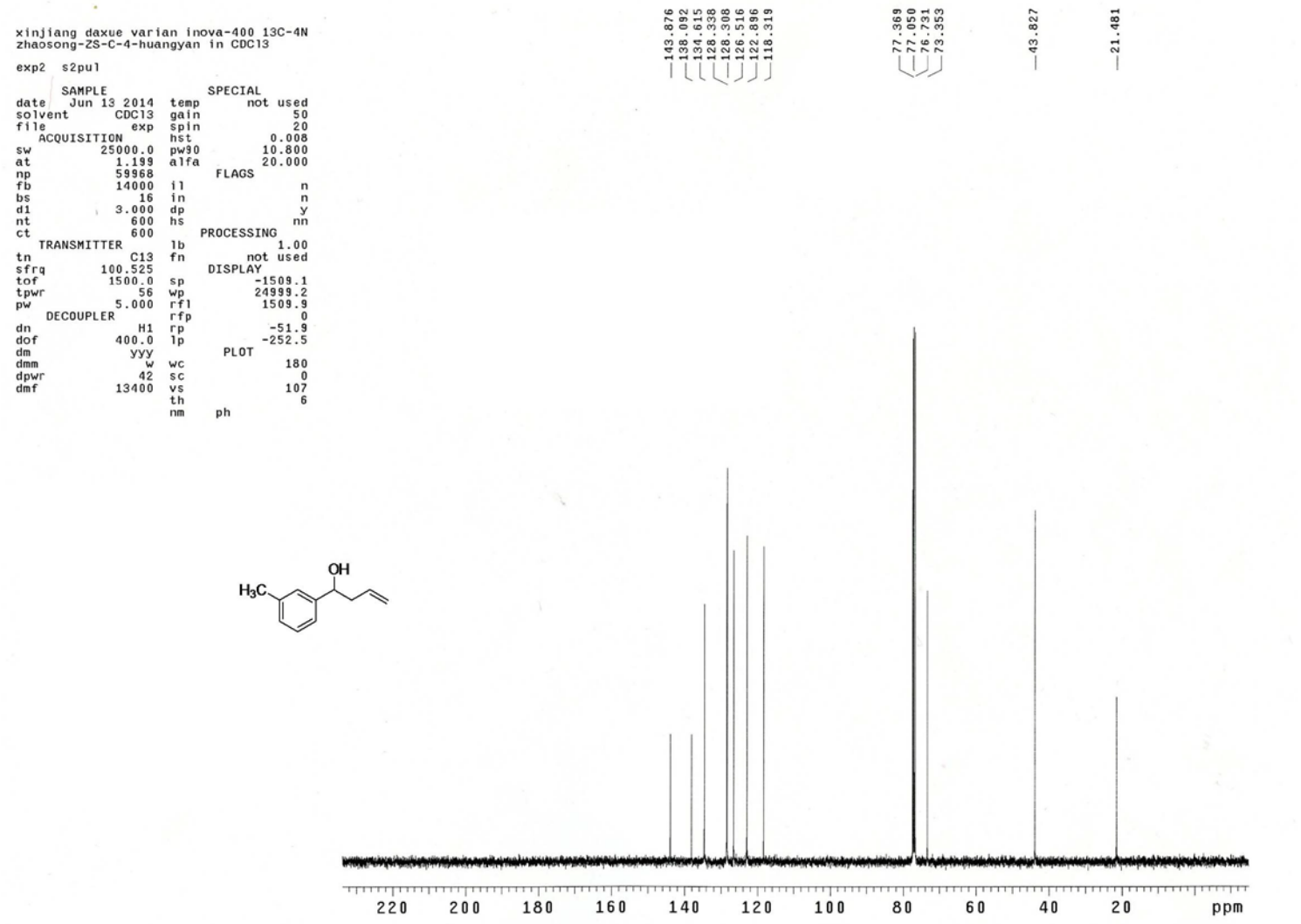


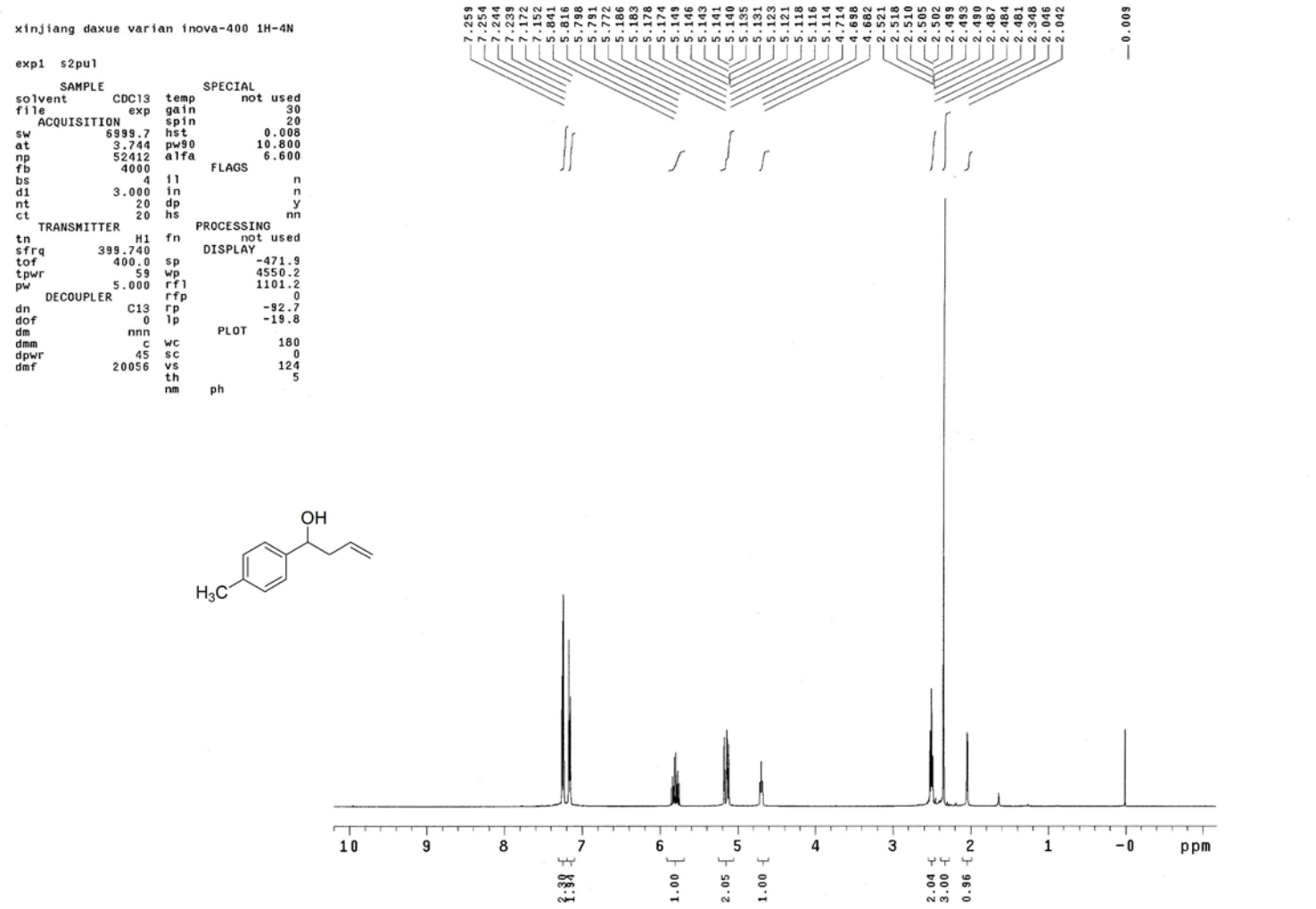

xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-2-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Sep 22 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 1200
ct 1200
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 40
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1520.2
wp 24999.2
rfl 9260.6
rfp 7739.6
rp 126.2
lp -244.6
PLOT
wc 180
sc 0
vs 136
th 6
nm ph
162.642
146.702
137.617
137.290
129.138
128.531
126.527
125.723
77.319
77.000
76.681
72.506
68.005
41.598
PyBox-4
220
200
180
160
140
120
100
80
60
40
20
ppm

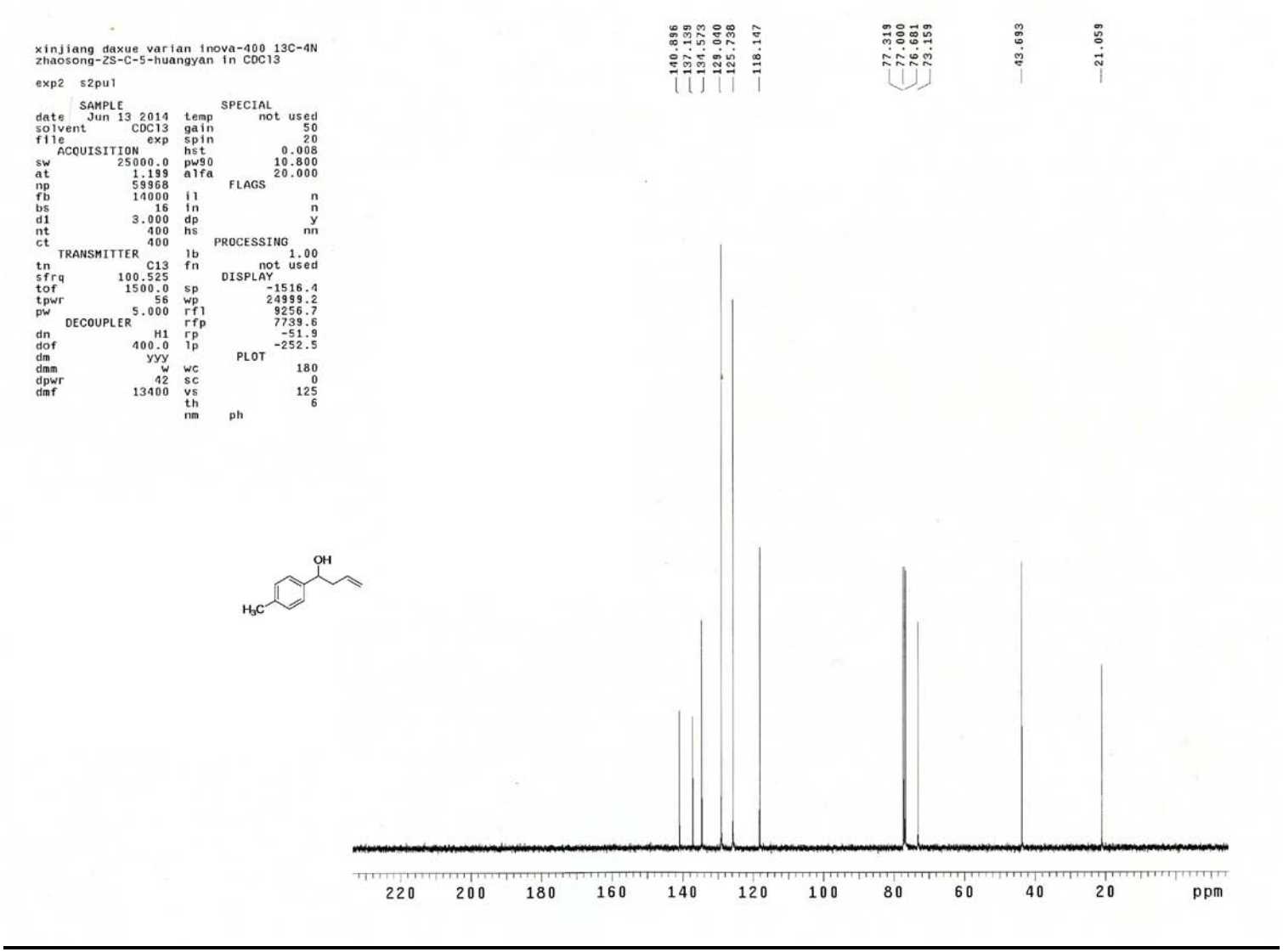

xinjiang daxue varian inova-400 1H-4N
exp13 s2pul
SAMPLE
solvent CDCl3
file exp
ACQUISITION
sw 6999.7
at 3.744
np 52410
fb 4000
bs 4
d1 3.000
nt 60
ct 60
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 3.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 20
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -478.9
wp 4538.7
rfl 1090.9
rfp 0
rp 78.7
lp -10.4
PLOT
wc 180
sc 0
vs 117
th 6
nm ph
OH
7.399 7.386 7.378 7.374 7.372 7.352 7.321 7.314 7.309 7.306 7.304 7.300 7.294 7.287 7.285 7.280 5.870 5.846 5.828 5.821 5.810 5.803 5.801 5.785 5.219 5.216 5.215 5.211 5.207 5.185 5.183 5.180 5.178 5.176 5.173 5.172 5.168 5.164 5.160 5.157 5.155 5.153 4.780 4.767 4.761 4.748 2.567 2.563 2.560 2.557 2.553 2.550 2.546 2.543 2.537 2.534 2.532 2.518 2.515 2.513 2.076 2.064 1.280 0.025
10 9 8 7 6 5 4 3 2 1 -0 ppm
3.74
1.56
0.95
1.91
1.00
2.02
1.04

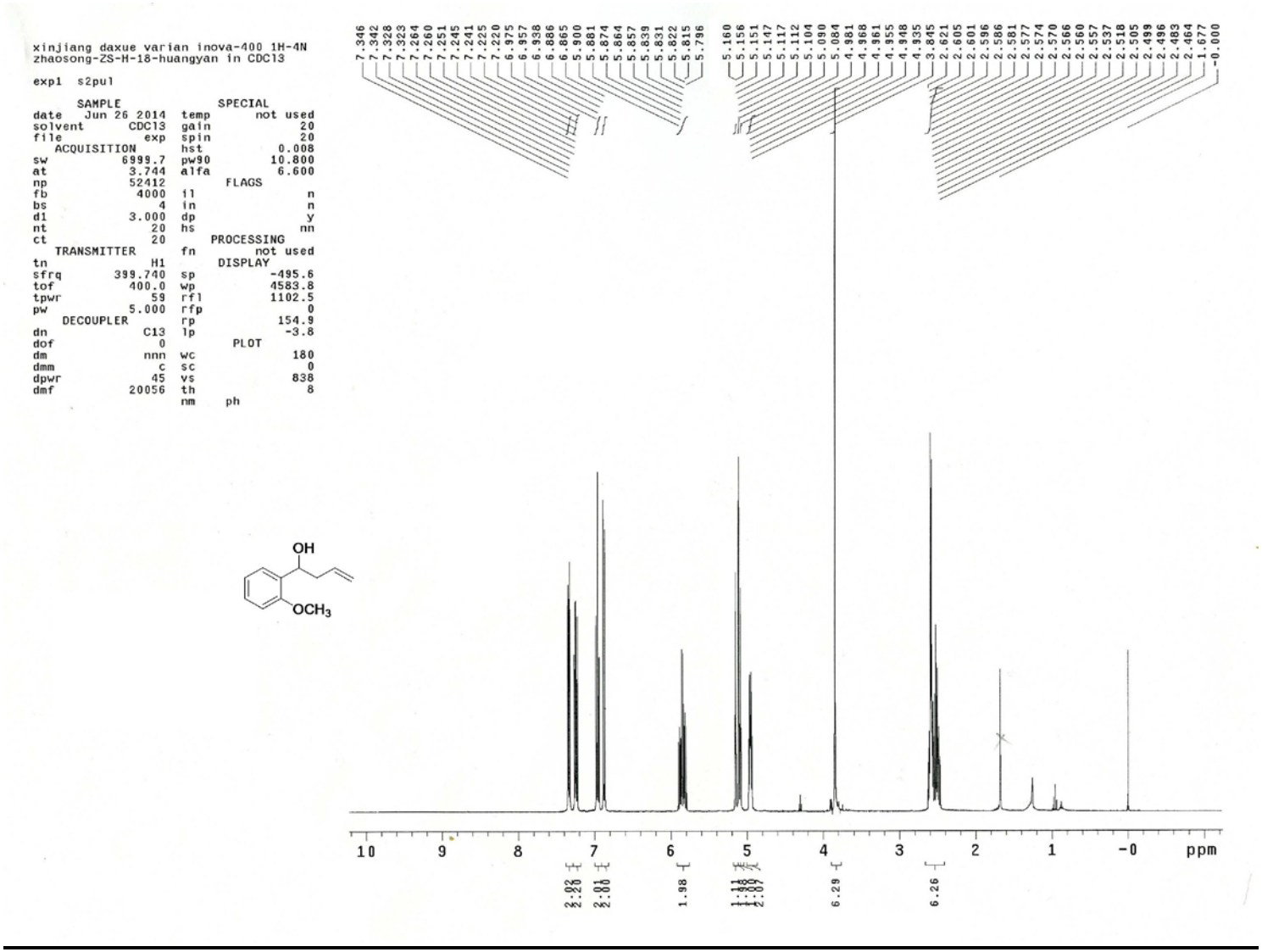

xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-C-7-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Jun 13 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 300
ct 300
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1514.9
wp 24999.2
rf1 9255.2
rfp 7739.6
rp -47.7
lp -252.5
PLOT
wc 180
sc 0
vs 97
th 6
nm ph
OH
143.848
134.429
128.379
127.506
125.783
118.352
77.319
77.000
76.681
73.273
43.784
220
200
180
160
140
120
100
80
60
40
20
ppm

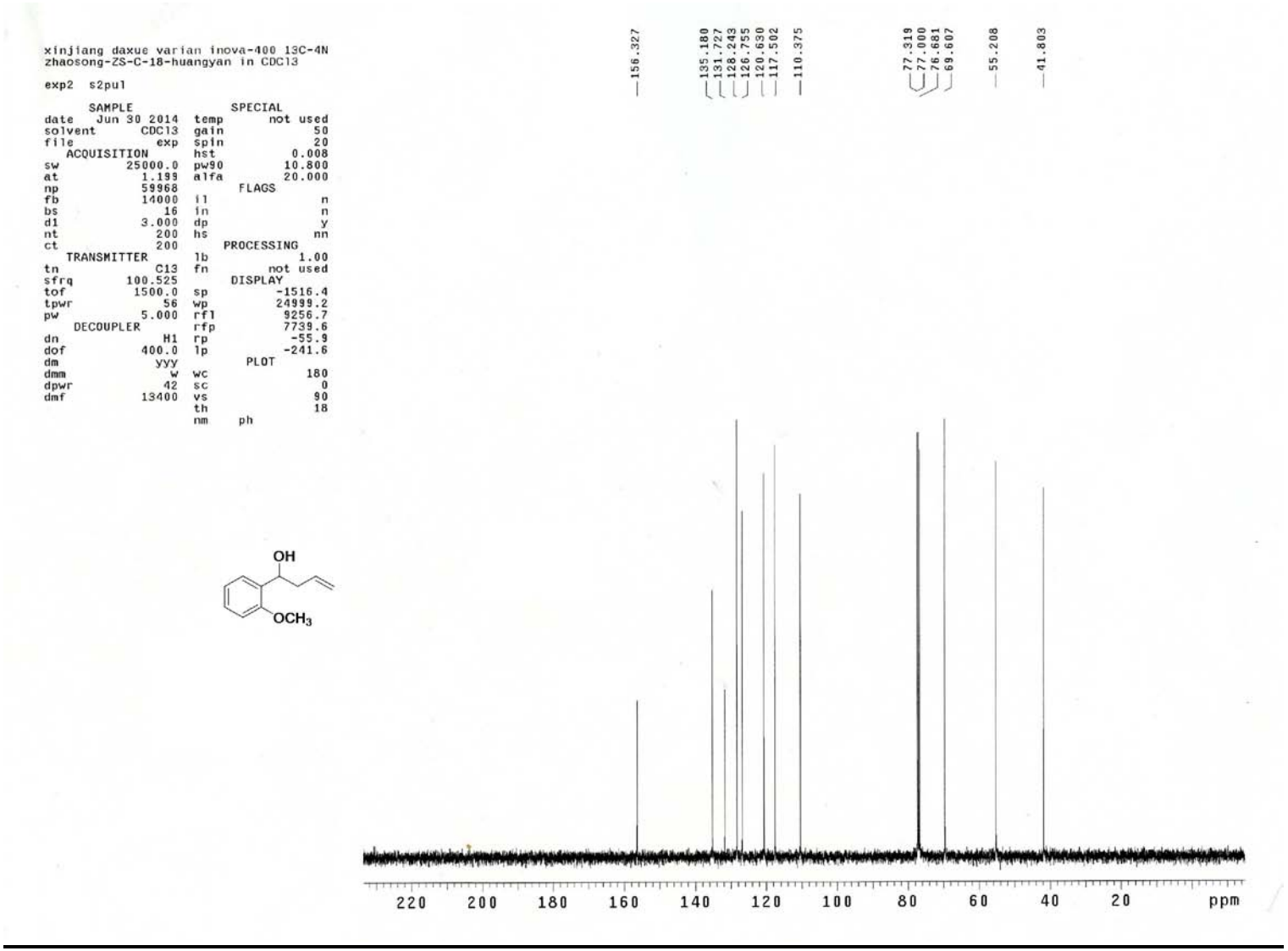

xinjiang daxue varian inova-400 1H-4N
expl s2pul
SAMPLE
solvent CDCl3
file exp
ACQUISITION
sw 6000.2
at 3.744
np 44928
fb 3000
bs 4
d1 3.000
nt 40
ct 40
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 30
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -493.5
wp 4562.2
rfl 599.0
rfp 0
rp 67.9
lp -10.9
PLOT
wc 180
sc 0
vs 115
th 3
nm ph
OH
H3C
ppm
1.52
0.95
1.01
2.04
1.02
2.15
3.00

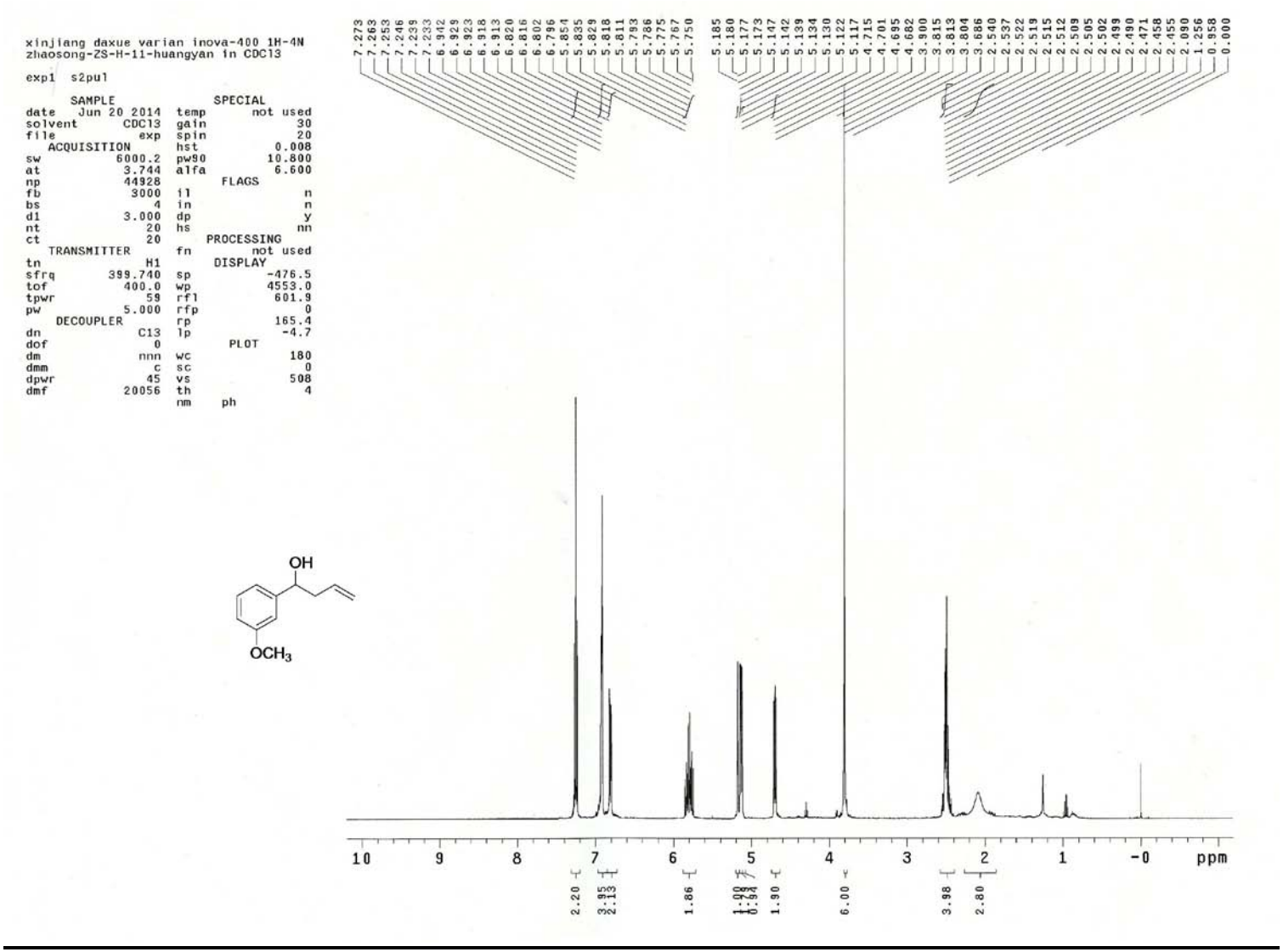

xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-C-4-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Jun 13 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 600
ct 600
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1509.1
wp 24999.2
rfl 1509.9
rfp 0
rp -51.9
lp -252.5
PLOT
wc 180
sc 0
vs 107
th 6
nm ph
143.876
138.092
134.615
128.338
128.308
126.516
122.896
118.319
77.369
77.050
76.731
73.353
43.827
21.481
OH
H3C
220
200
180
160
140
120
100
80
60
40
20
ppm

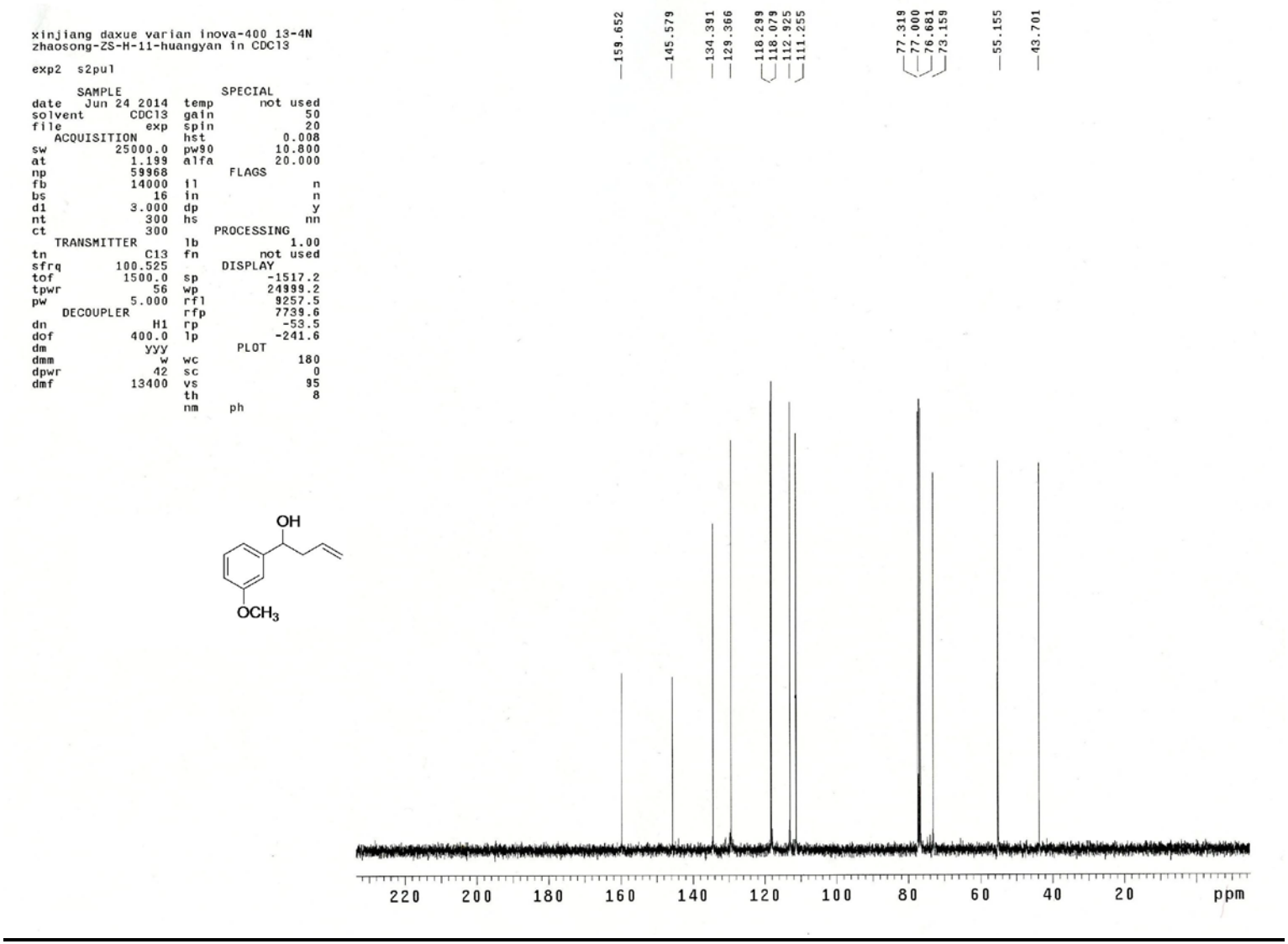

xinjiang daxue varian inova-400 1H-4N
exp1 s2pul
SAMPLE
solvent CDCl3
file exp
ACQUISITION
sw 6999.7
at 3.744
np 52412
fb 4000
bs 4
d1 3.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 30
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -471.9
wp 4550.2
rfl 1101.2
rfp 0
rp -92.7
lp -19.8
PLOT
wc 180
sc 0
vs 124
th 5
nm ph
7.259
7.254
7.244
7.239
7.172
7.152
5.841
5.816
5.798
5.791
5.772
5.186
5.183
5.178
5.174
5.149
5.146
5.143
5.141
5.140
5.135
5.131
5.123
5.121
5.118
5.116
5.114
4.714
4.698
4.682
2.521
2.518
2.510
2.505
2.502
2.499
2.493
2.490
2.487
2.484
2.481
2.348
2.046
2.042
0.009
OH
H3C
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.30
1.94
1.00
2.05
1.00
2.04
3.00
0.96

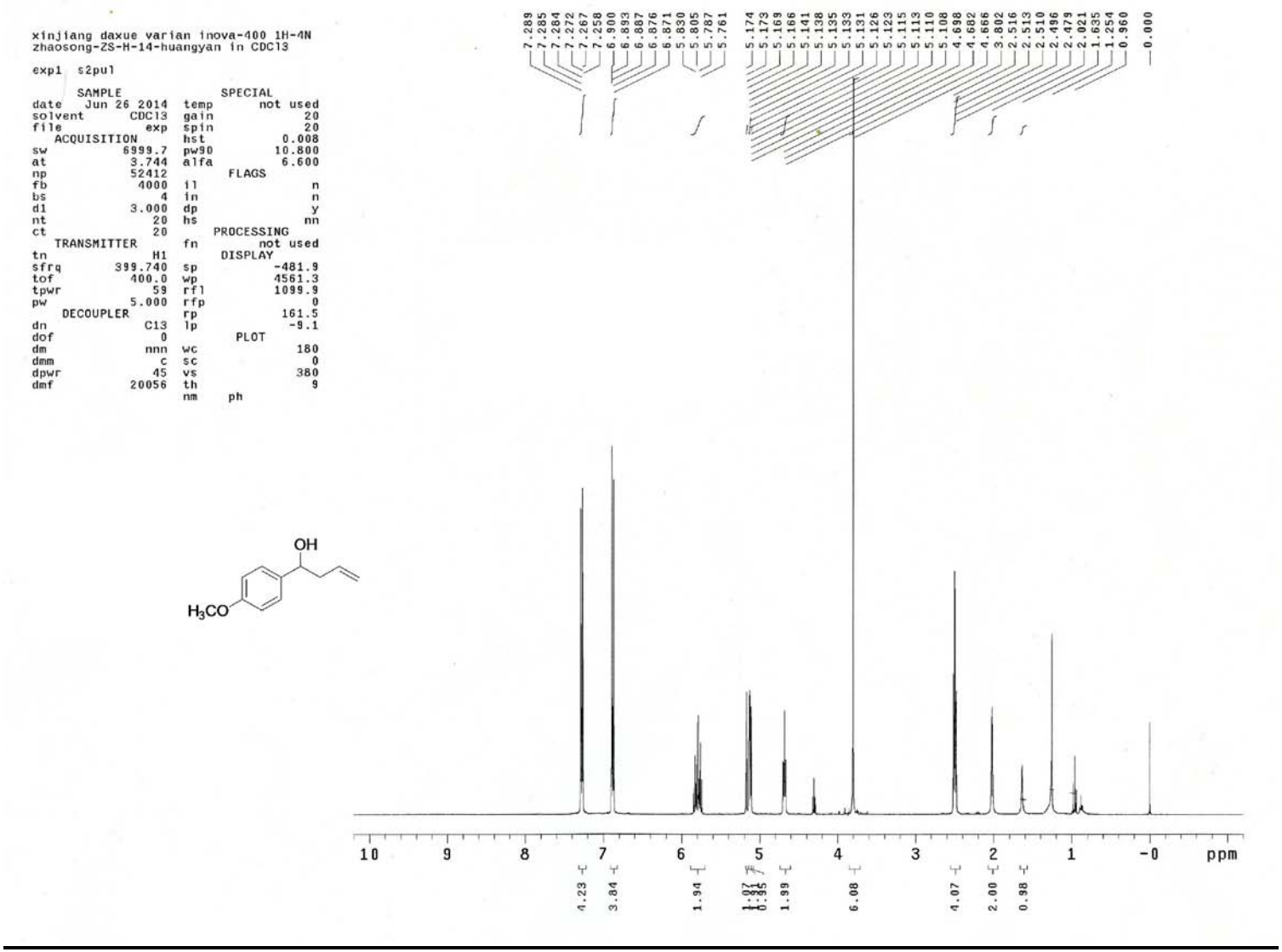

xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-C-5-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Jun 13 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 400
ct 400
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1516.4
wp 24999.2
rfl 9256.7
rfp 7739.6
rp -51.9
lp -252.5
PLOT
wc 180
sc 0
vs 125
th 6
nm ph
140.886
137.139
134.573
129.040
125.738
118.147
77.319
77.000
76.681
73.159
43.693
21.059
OH
H3C
220
200
180
160
140
120
100
80
60
40
20
ppm

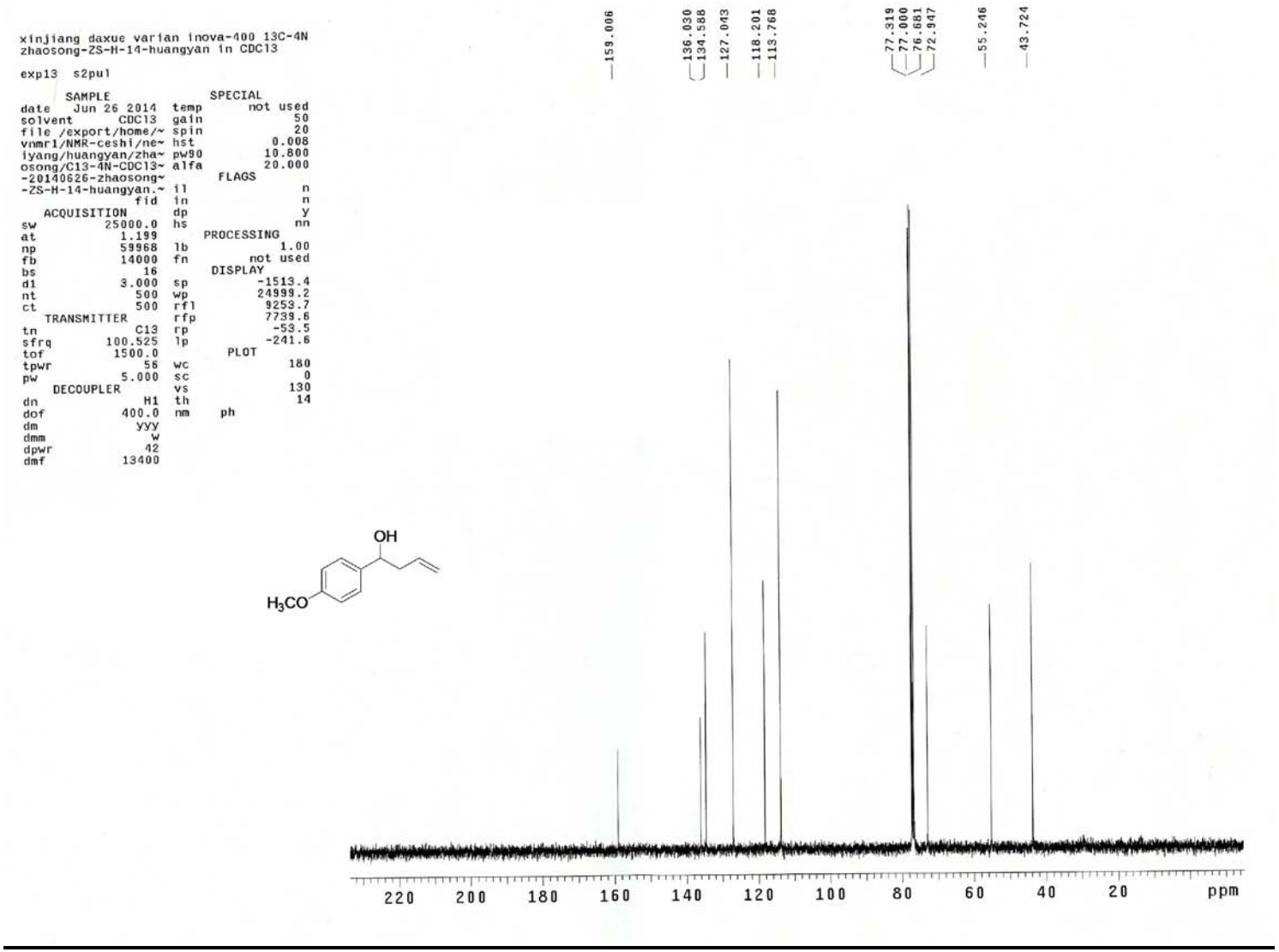

xinjiang daxue varian inova-400 1H-4N
zhaosong-ZS-H-18-huangyan in CDCl3
exp1 s2pul
SAMPLE
date Jun 26 2014
solvent CDCl3
file exp
ACQUISITION
sw 6999.7
at 3.744
np 52412
fb 4000
bs 4
d1 3.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 20
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -495.6
wp 4583.8
rfl 1102.5
rfp 0
rp 154.9
lp -3.8
PLOT
wc 180
sc 0
vs 838
th 8
nm ph
OH
OCH3
7.346
7.342
7.328
7.323
7.264
7.260
7.251
7.245
7.241
7.225
7.220
6.975
6.957
6.938
6.886
6.865
5.900
5.881
5.874
5.864
5.857
5.839
5.831
5.822
5.815
5.796
5.160
5.156
5.151
5.147
5.117
5.112
5.104
5.090
5.084
4.981
4.968
4.961
4.955
4.948
4.935
3.845
2.621
2.605
2.601
2.596
2.586
2.581
2.577
2.574
2.570
2.566
2.560
2.557
2.537
2.518
2.505
2.499
2.496
2.483
2.464
1.677
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.02
2.01
1.98
1.11
1.00
2.07
6.29
6.26

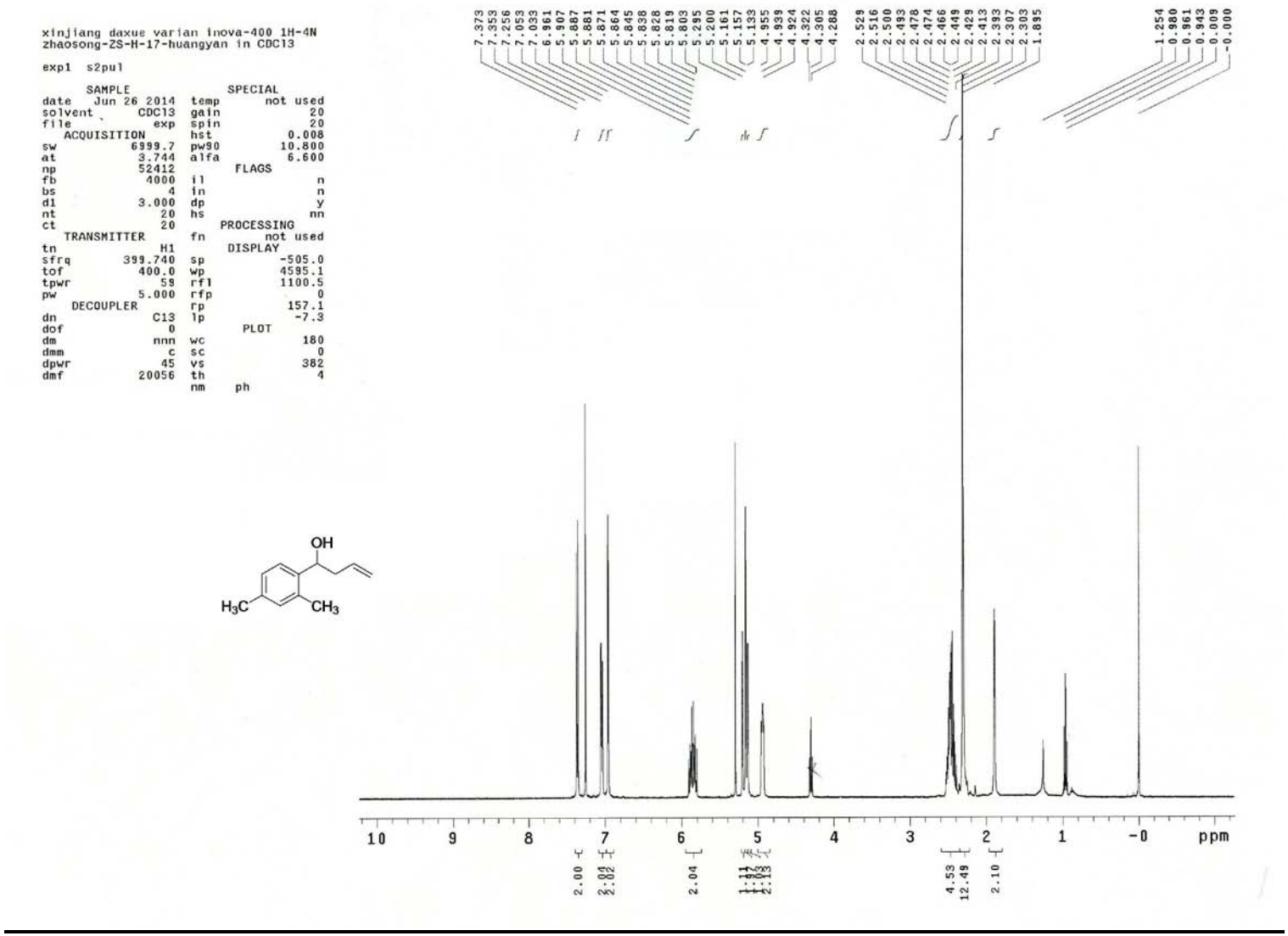

xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-C-18-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Jun 30 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 200
ct 200
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1516.4
wp 24999.2
rfl 9256.7
rfp 7739.6
rp -55.9
lp -241.6
PLOT
wc 180
sc 0
vs 90
th 18
nm ph
OH
OCH3
156.327
135.180
131.727
128.243
126.755
120.630
117.502
110.375
77.319
77.000
76.681
69.607
55.208
41.803
220
200
180
160
140
120
100
80
60
40
20
ppm

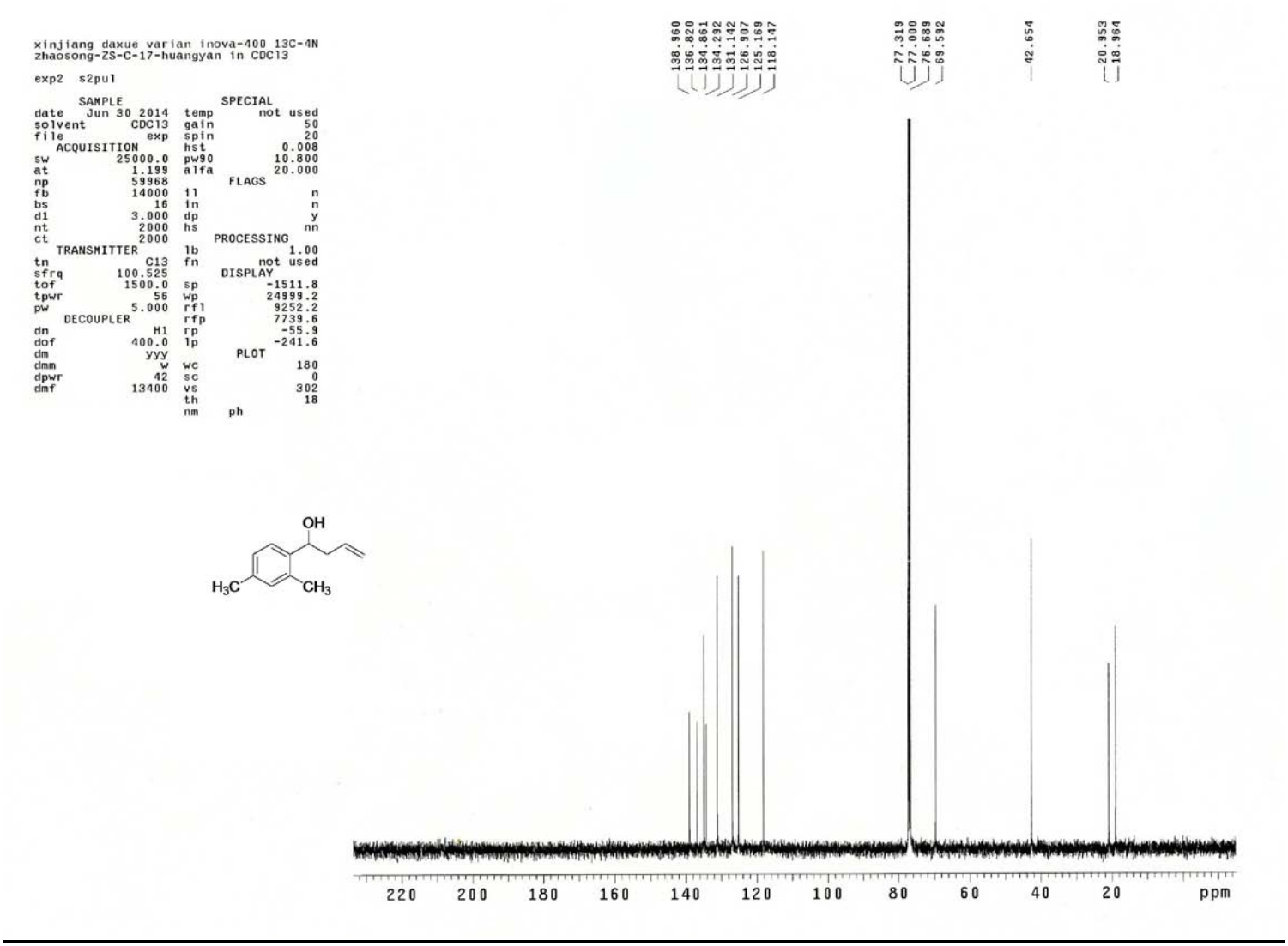

xinjiang daxue varian inova-400 1H-4N
zhaosong-ZS-H-11-huangyan in CDCl3
exp1 s2pul
SAMPLE
date Jun 20 2014
solvent CDCl3
file exp
ACQUISITION
sw 6000.2
at 3.744
np 44928
fb 3000
bs 4
d1 3.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 30
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -476.5
wp 4553.0
rfl 601.9
rfp 0
rp 165.4
lp -4.7
PLOT
wc 180
sc 0
vs 508
th 4
nm ph
OH
OCH3
ppm

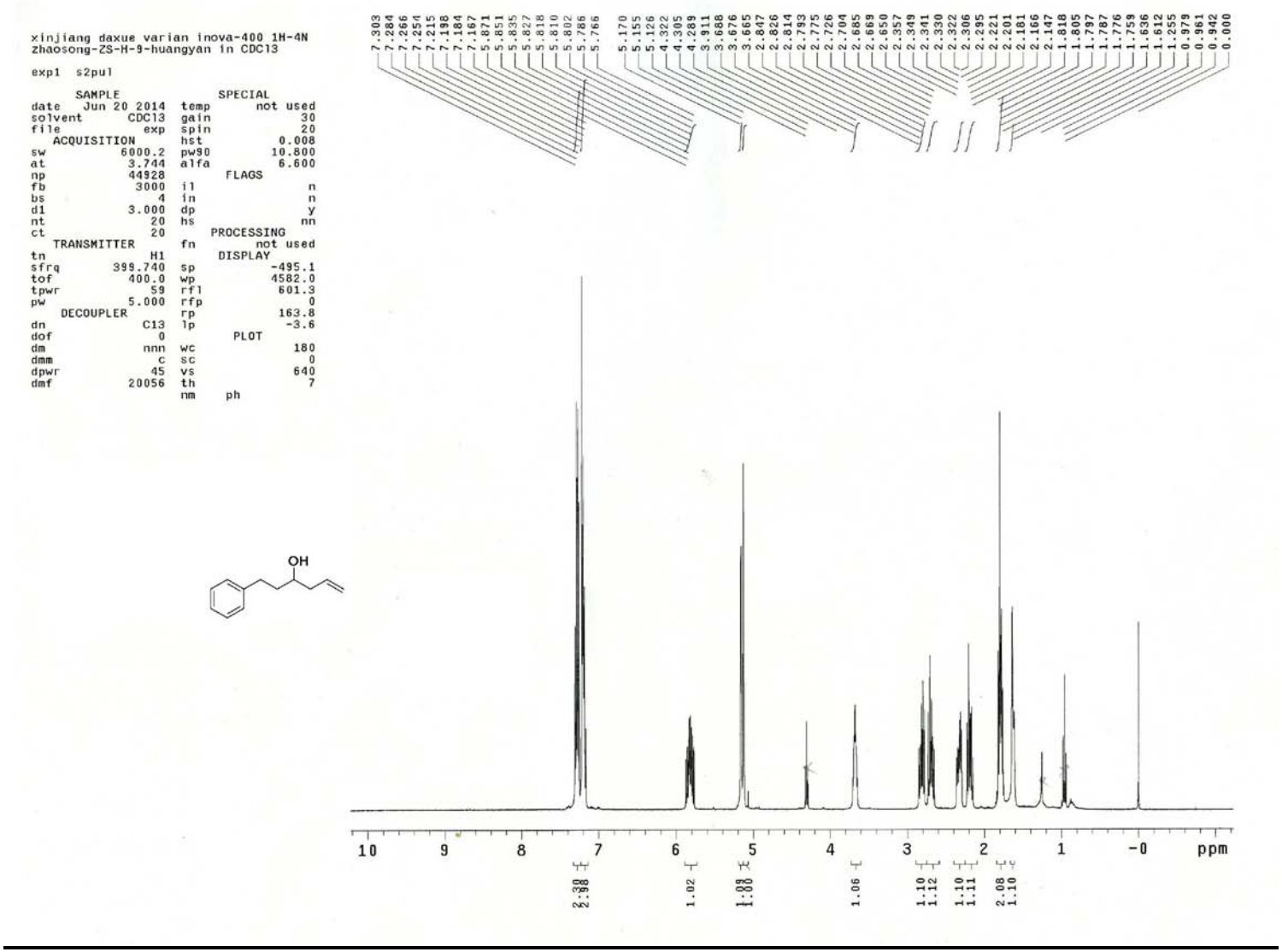

xinjiang daxue varian inova-400 13-4N
zhaosong-ZS-H-11-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Jun 24 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 300
ct 300
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1517.2
wp 24999.2
rf1 9257.5
rfp 7738.6
rp -53.5
lp -241.6
PLOT
wc 180
sc 0
vs 95
th 8
nm ph
159.652
145.579
134.391
129.366
118.299
118.079
112.925
111.255
77.319
77.000
76.681
73.159
55.155
43.701
OH
OCH3
220
200
180
160
140
120
100
80
60
40
20
ppm

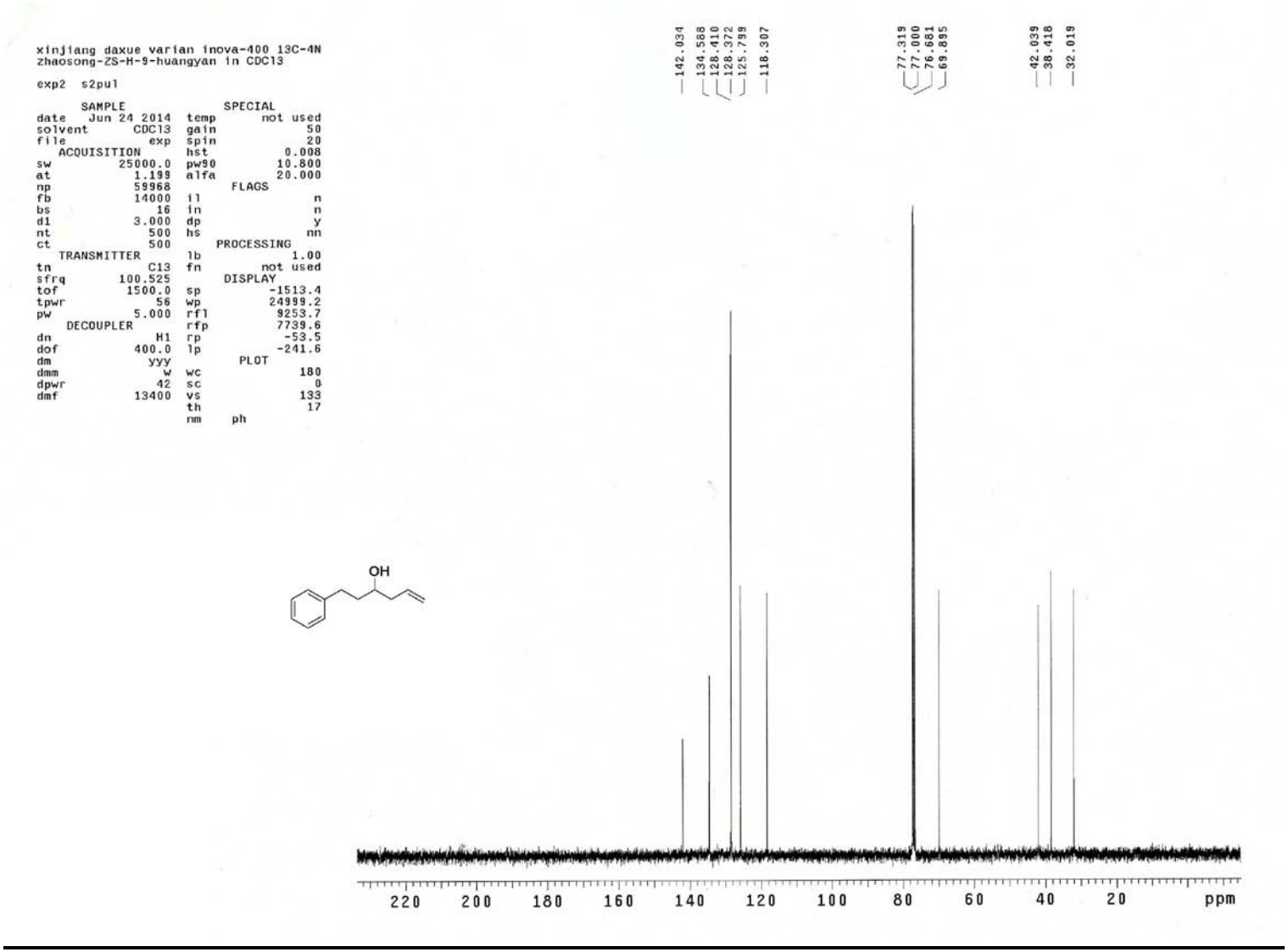

xinjiang daxue varian inova-400 1H-4N
zhaosong-ZS-H-14-huangyan in CDCl3
exp1 s2pul
SAMPLE
date Jun 26 2014
solvent CDCl3
file exp
ACQUISITION
sw 6999.7
at 3.744
np 52412
fb 4000
bs 4
d1 3.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 20
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -481.9
wp 4561.3
rfl 1099.9
rfp 0
rp 161.5
lp -9.1
PLOT
wc 180
sc 0
vs 380
th 9
nm ph
OH
H3CO
7.289 7.285 7.284 7.272 7.267 7.258 6.900 6.893 6.887 6.876 6.871 5.830 5.805 5.787 5.761
5.174 5.173 5.169 5.166 5.141 5.138 5.135 5.133 5.131 5.126 5.123 5.115 5.113 5.110 5.108 4.698 4.682 4.666 3.802 2.516 2.513 2.510 2.486 2.479 2.021 1.635 1.254 0.960 0.000
10 9 8 7 6 5 4 3 2 1 -0 ppm
4.23 3.84 1.94 1.07 1.00 0.95 1.99 6.08 4.07 2.00 0.98

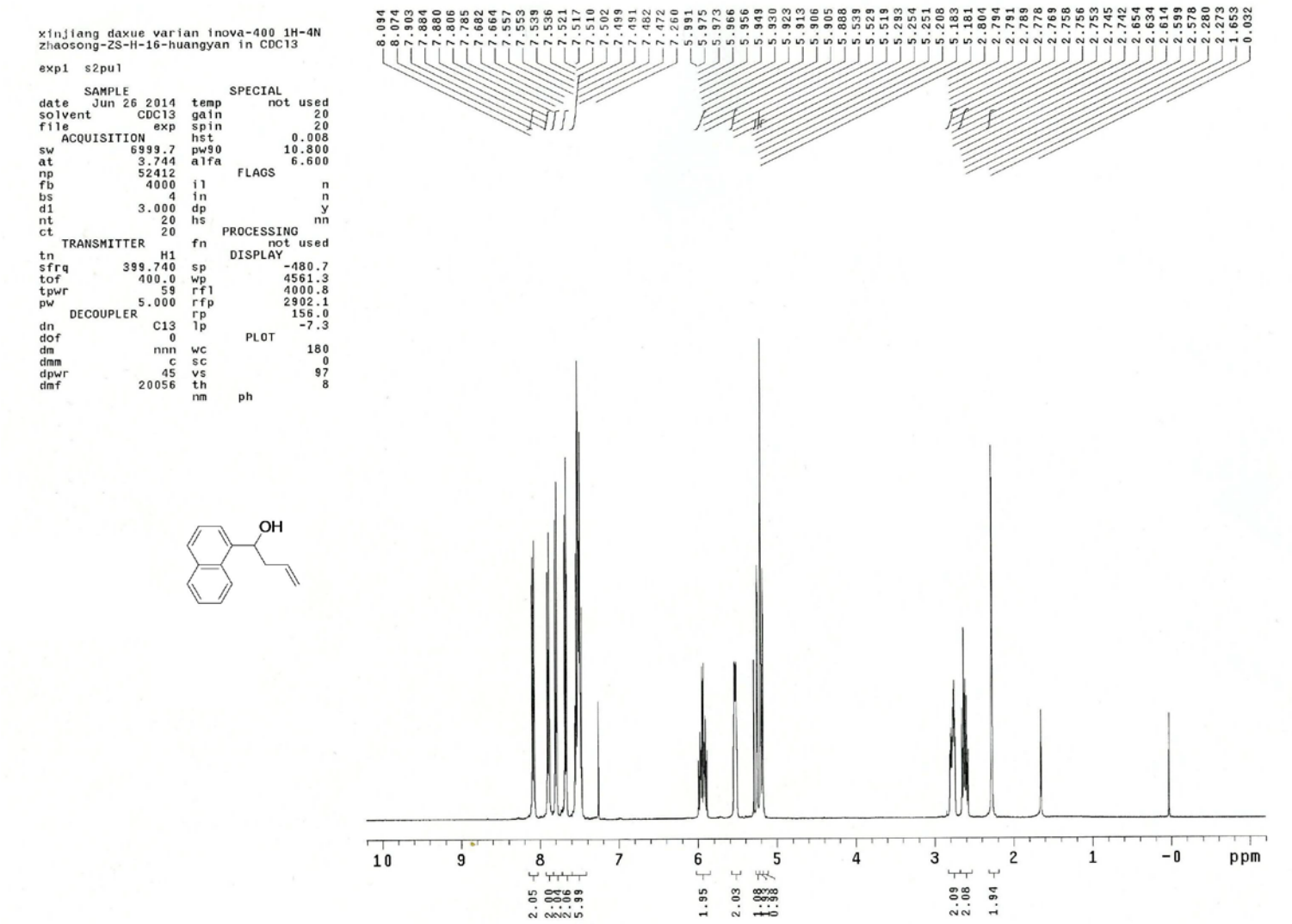

xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-H-14-huangyan in CDCl3
exp13 s2pul
SAMPLE
date Jun 26 2014
solvent CDCl3
file /export/home/~
vnmr1/NMR-ceshi/ne~
iyang/huangyan/zha~
osong/C13-4N-CDCl3~
-20140626-zhaosong~
-ZS-H-14-huangyan.~
fid
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 500
ct 500
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1513.4
wp 24999.2
rfl 9253.7
rfp 7739.6
rp -53.5
lp -241.6
PLOT
wc 180
sc 0
vs 130
th 14
nm ph
159.006
136.030
134.588
127.043
118.201
113.768
77.319
77.000
76.681
72.947
55.246
43.724
OH
H3CO
220
200
180
160
140
120
100
80
60
40
20
ppm

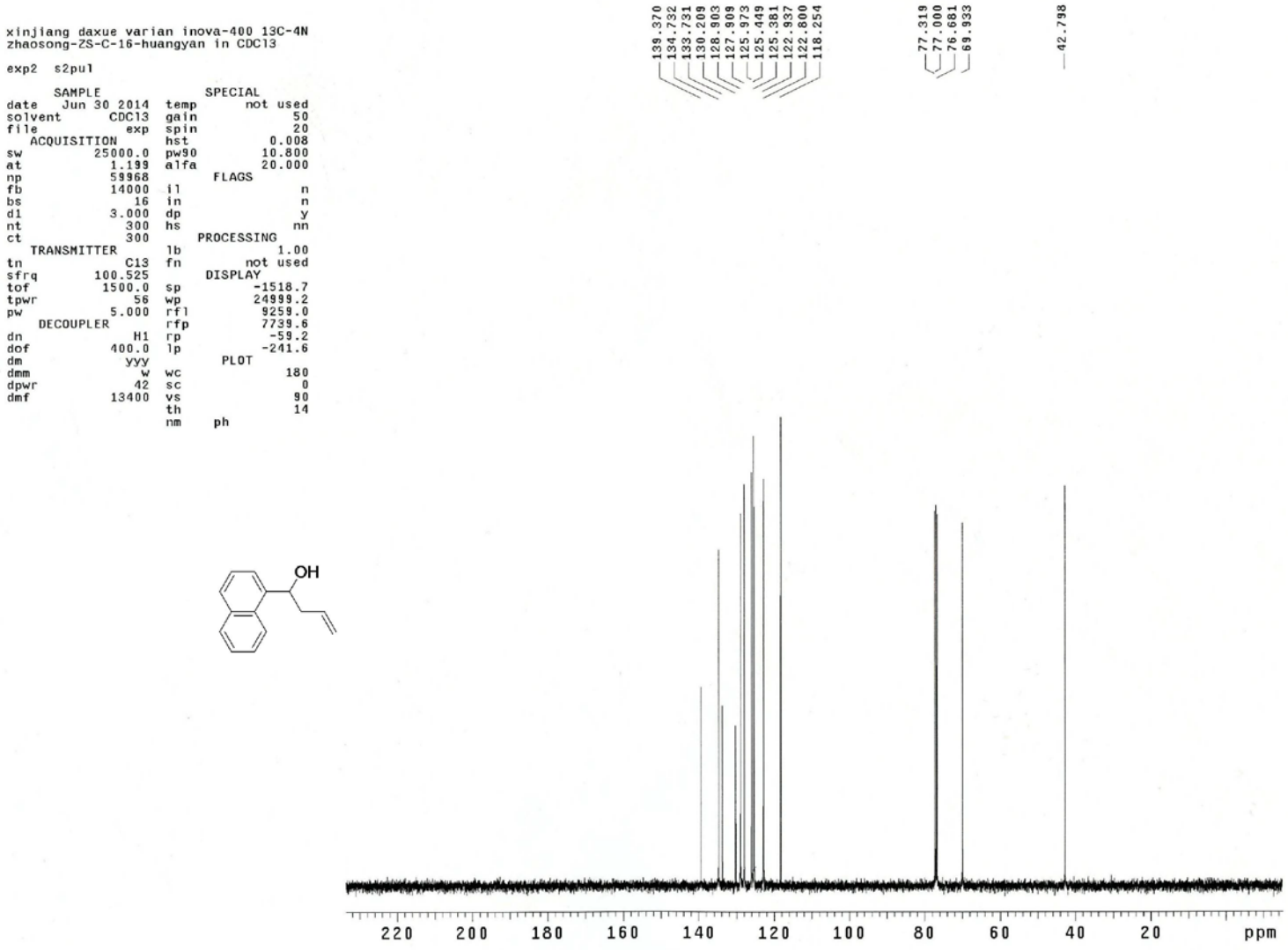

xinjiang daxue varian inova-400 1H-4N
zhaosong-ZS-H-17-huangyan in CDCl3
exp1 s2pul
SAMPLE
date Jun 26 2014
solvent CDCl3
file exp
ACQUISITION
sw 6999.7
at 3.744
np 52412
fb 4000
bs 4
d1 3.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 20
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -505.0
wp 4595.1
rfl 1100.5
rfp 0
rp 157.1
lp -7.3
PLOT
wc 180
sc 0
vs 382
th 4
nm ph
OH
H3C
CH3
7.373
7.353
7.256
7.053
7.033
6.961
5.907
5.887
5.881
5.871
5.864
5.845
5.838
5.828
5.819
5.803
5.295
5.200
5.161
5.157
5.133
4.955
4.939
4.924
4.322
4.305
4.288
2.529
2.516
2.500
2.493
2.478
2.474
2.466
2.449
2.429
2.413
2.393
2.307
2.303
1.895
1.254
0.980
0.961
0.943
0.009
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.00
2.02
2.04
4.53
12.49
2.10

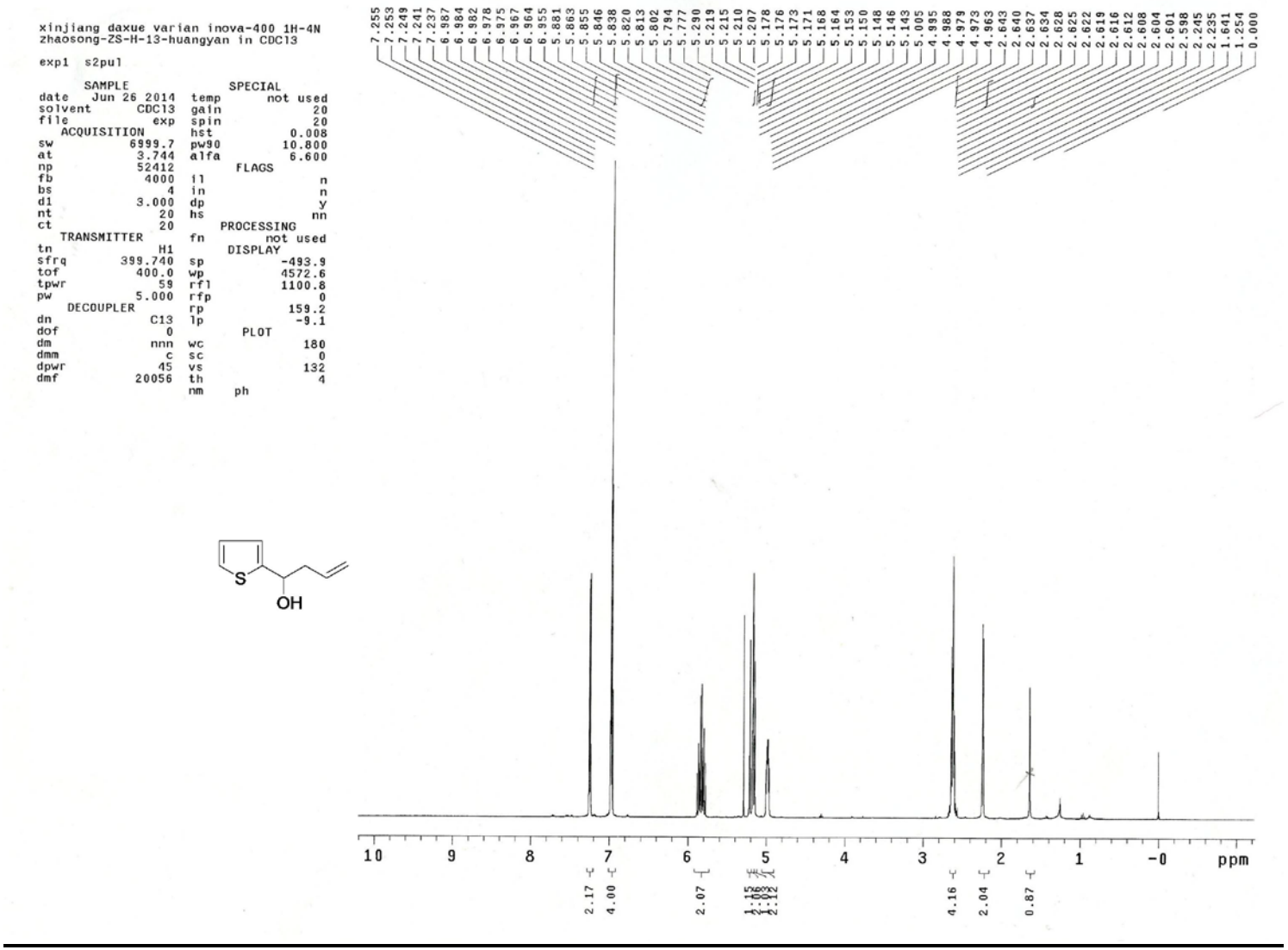

xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-C-17-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Jun 30 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 2000
ct 2000
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1511.8
wp 24999.2
rfl 9252.2
rfp 7739.6
rp -55.9
lp -241.6
PLOT
wc 180
sc 0
vs 302
th 18
nm ph
138.960
136.820
134.861
134.292
131.142
126.907
125.169
118.147
77.319
77.000
76.689
69.592
42.654
20.953
18.964
OH
H3C
CH3
220
200
180
160
140
120
100
80
60
40
20
ppm

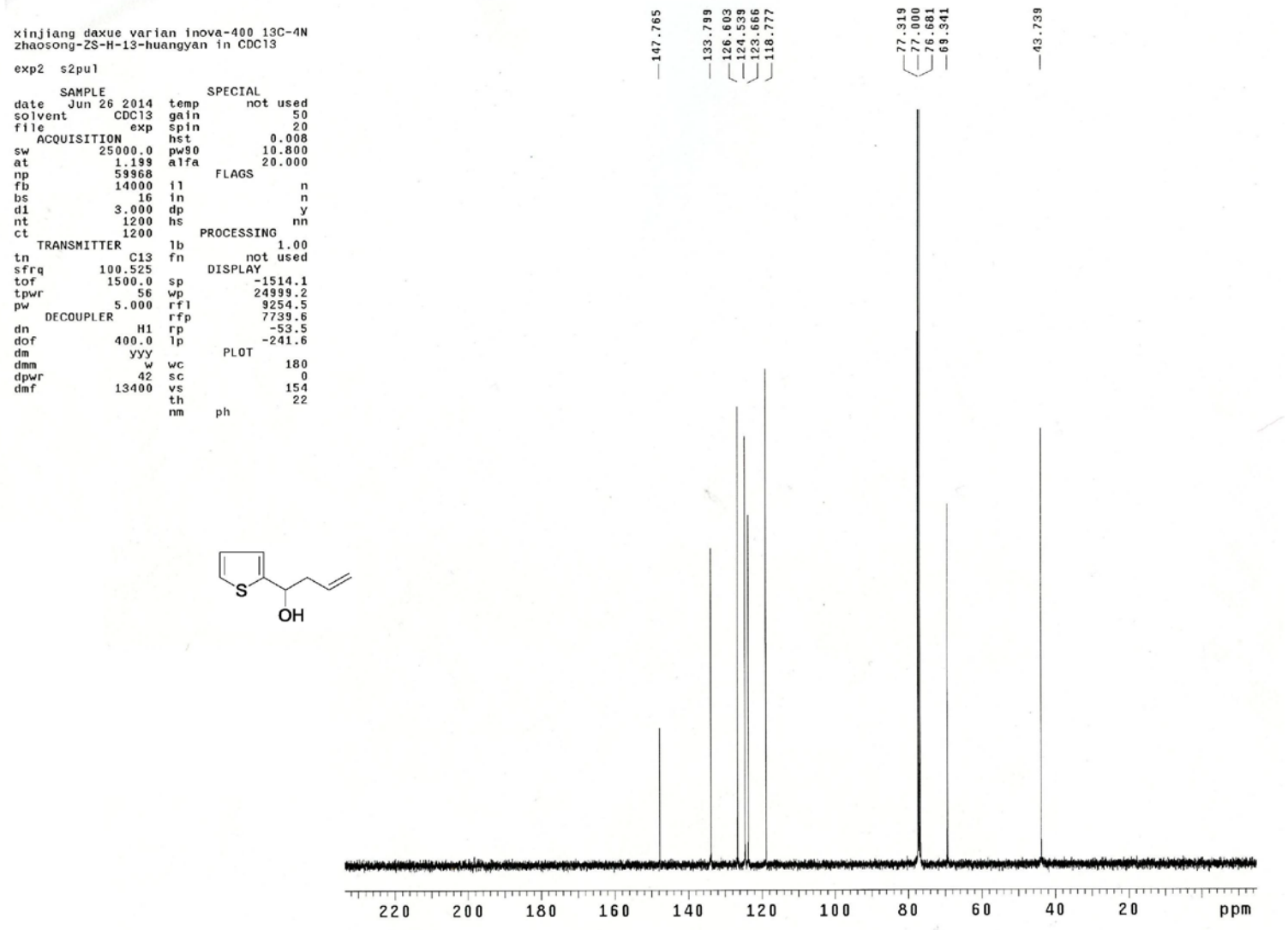

xinjiang daxue varian inova-400 1H-4N
zhaosong-ZS-H-9-huangyan in CDCl3
exp1 s2pul
SAMPLE
date Jun 20 2014
solvent CDCl3
file exp
ACQUISITION
sw 6000.2
at 3.744
np 44928
fb 3000
bs 4
d1 3.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 30
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -495.1
wp 4582.0
rfl 601.3
rfp 0
rp 163.8
lp -3.6
PLOT
wc 180
sc 0
vs 640
th 7
nm ph
OH
7.303
7.284
7.266
7.254
7.215
7.198
7.184
7.167
5.871
5.851
5.835
5.827
5.818
5.810
5.802
5.786
5.766
5.170
5.155
5.126
4.322
4.305
4.289
3.911
3.688
3.676
3.665
2.847
2.826
2.814
2.793
2.775
2.726
2.704
2.685
2.669
2.650
2.357
2.349
2.341
2.330
2.322
2.306
2.295
2.221
2.201
2.181
2.166
2.147
1.818
1.805
1.797
1.787
1.776
1.759
1.636
1.612
1.255
0.979
0.961
0.942
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.30
2.00
1.02
1.00
1.06
1.10
1.12
1.10
1.11
2.08
1.10


xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-H-9-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Jun 24 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 500
ct 500
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1513.4
wp 24999.2
rfl 9253.7
rfp 7739.6
rp -53.5
lp -241.6
PLOT
wc 180
sc 0
vs 133
th 17
nm ph
142.034
134.588
128.410
128.372
125.799
118.307
77.319
77.000
76.681
69.895
42.039
38.418
32.019
OH
220
200
180
160
140
120
100
80
60
40
20
ppm


xinjiang daxue varian inova-400 1H-4N
zhaosong-ZS-H-16-huangyan in CDCl3
exp1 s2pul
SAMPLE
date Jun 26 2014
solvent CDCl3
file exp
ACQUISITION
sw 6999.7
at 3.744
np 52412
fb 4000
bs 4
d1 3.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 20
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -480.7
wp 4561.3
rfl 4000.8
rfp 2902.1
rp 156.0
lp -7.3
PLOT
wc 180
sc 0
vs 97
th 8
nm ph
OH
8.094
8.074
7.903
7.884
7.880
7.806
7.785
7.682
7.664
7.557
7.553
7.539
7.536
7.521
7.517
7.510
7.502
7.499
7.491
7.482
7.472
7.260
5.991
5.975
5.973
5.966
5.956
5.949
5.930
5.923
5.913
5.906
5.905
5.888
5.539
5.529
5.519
5.283
5.254
5.251
5.208
5.183
5.181
2.804
2.794
2.791
2.789
2.778
2.769
2.758
2.756
2.753
2.745
2.742
2.654
2.634
2.614
2.599
2.578
2.280
2.273
1.653
0.032
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.05
2.00
2.04
2.06
5.99
1.95
2.03
1.08
0.98
2.09
2.08
1.94


xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-C-16-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Jun 30 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 300
ct 300
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1518.7
wp 24999.2
rfl 9259.0
rfp 7739.6
rp -59.2
lp -241.6
PLOT
wc 180
sc 0
vs 90
th 14
nm ph
OH
139.370
134.732
133.731
130.209
128.903
127.909
125.973
125.449
125.381
122.937
122.800
118.254
77.319
77.000
76.681
69.933
42.798
220
200
180
160
140
120
100
80
60
40
20
ppm


xinjiang daxue varian inova-400 1H-4N
zhaosong-ZS-H-13-huangyan in CDCl3
exp1 s2pul
SAMPLE
date Jun 26 2014
solvent CDCl3
file exp
ACQUISITION
sw 6999.7
at 3.744
np 52412
fb 4000
bs 4
d1 3.000
nt 20
ct 20
TRANSMITTER
tn H1
sfrq 399.740
tof 400.0
tpwr 59
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 20056
SPECIAL
temp not used
gain 20
spin 20
hst 0.008
pw90 10.800
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -493.9
wp 4572.6
rfl 1100.8
rfp 0
rp 159.2
lp -9.1
PLOT
wc 180
sc 0
vs 132
th 4
nm ph
S
OH
7.255
7.253
7.249
7.241
7.237
6.987
6.984
6.982
6.978
6.975
6.967
6.964
6.955
5.881
5.863
5.855
5.846
5.838
5.820
5.813
5.802
5.794
5.777
5.290
5.219
5.215
5.210
5.207
5.178
5.176
5.173
5.171
5.168
5.164
5.153
5.150
5.148
5.146
5.143
5.005
4.995
4.988
4.979
4.973
4.963
2.643
2.640
2.637
2.634
2.628
2.625
2.622
2.619
2.616
2.612
2.608
2.604
2.601
2.598
2.245
2.235
1.641
1.254
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.17
4.00
2.07
1.15
1.08
1.03
2.12
4.16
2.04
0.87


xinjiang daxue varian inova-400 13C-4N
zhaosong-ZS-H-13-huangyan in CDCl3
exp2 s2pul
SAMPLE
date Jun 26 2014
solvent CDCl3
file exp
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 14000
bs 16
d1 3.000
nt 1200
ct 1200
TRANSMITTER
tn C13
sfrq 100.525
tof 1500.0
tpwr 56
pw 5.000
DECOUPLER
dn H1
dof 400.0
dm yyy
dmm w
dpwr 42
dmf 13400
SPECIAL
temp not used
gain 50
spin 20
hst 0.008
pw90 10.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1514.1
wp 24999.2
rfl 9254.5
rfp 7739.6
rp -53.5
lp -241.6
PLOT
wc 180
sc 0
vs 154
th 22
nm ph
147.765
133.799
126.603
124.539
123.666
118.777
77.319
77.000
76.681
69.341
43.739
S
OH
220
200
180
160
140
120
100
80
60
40
20
ppm