Electronic Supporting Information

γ-Lactams and furan bispyrrolidines *via* iodine mediated cyclisation of homoallylamines

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General Experimental

Reagents and solvents used as commercially obtained from Sigma-Aldrich or Fisher. ^{1}HNMR spectroscopy was performed at 300 MHz on a Bruker AVIII300 NMR spectrometer or at 400 MHz on an AV400 NMR spectrometer. Proton decoupled ^{13}C NMR spectra were recorded at 100 MHz on a Bruker AVIII400 NMR spectrometer at room temperature. Chemical shifts (δ) are recorded in ppm relative to TMS (δ 0.00) for ^{1}HNMR or residual solvent and to chloroform (δ 77.0) for the ^{13}C NMR measurements, coupling constant *J* are expressed in Hertz, the PENDANT technique was used to aid ^{13}C NMR assignment in some cases, representative spectra are provided. Designation *cis* and *trans* refers to the relative orientation of carbon substituents on lactam rings synthesised herein. Mass spectrometry was performed using an Electrospray MS Waters LCT Time of flight Mass Spectrometer and with EI (GC/MS) Waters GCT Premier Time of Flight Mass Spectrometer. IR spectroscopy was recorded using a PerkinElmer 100FT-IR Spectrometer at room temperature. Melting points were measured using Stuart™ digital melting point apparatus (SMP10).

General Procedures

General procedure for the preparation of methyl-substituted homoallylamines (a)

Under nitrogen, activated zinc powder (2 equiv.) was mixed with3-bromo-2-methylprop-1-ene (1.5 equiv.) in dry THF (10 mL) and stirred for 30 minutes. The appropriate imine (1 equiv.) in dry THF (2 mL) was added to the reaction vessel. After the reaction was complete, as judged by TLC ethyl acetate/hexane 10%, it was

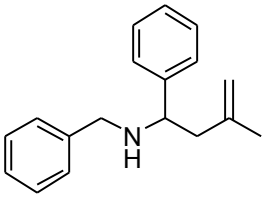
quenched with saturated sodium bicarbonate solution (5 mL) and extracted with ethyl acetate (3x 20 mL), dried over $MgSO_4$, concentrated *in vacuo* and purified by column chromatography.

General procedure for synthesis of γ-lactams (b)

To one equivalent of homoallylamine derivatives (**5a-q**) in ethyl acetate (25 mL) five equivalents of sodium bicarbonate and three equivalents of iodine were added, and the mixture stirred at room temperature for 24 hours. After the reaction was complete, as judged by TLC ethyl acetate/hexane, it was quenched with sodium thiosulfate (5 mL) and extracted with ethyl acetate. The organic layers were combined, concentrated *in vacuo* and purified by column chromatography. Where achieved, separation of diastereomers was performed by further column chromatography, with the *trans* isomer being typically more challenging to isolate.

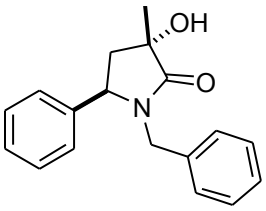
Synthesis

N-Benzyl-3-methyl-1-phenylbut-3-en-1-amine (5a)



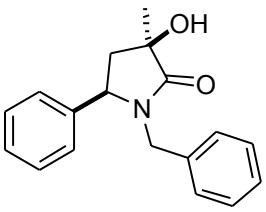
General procedure (**a)** was used. *N*-Benzylidene-1-phenylmethanamine (1.00 g, 5.0 mmol), 3-bromo-2-methylprop-2-ene (1.00 g, 10.0 mmol) and zinc powder (838 mg, 10.0 mmol) in dry THF (10 mL), pale yellow oil, 1.3 g, 99% yield. IR 3450, 3026, 2933, 1492. ^{1}H NMR (δ; 300 MHz, $CDCl_3$);1.72 (3H, s, CH_3), 1.85 (1H, s, NH), 2.37 (1H, dd, J_{ab} 14.0, J_{ac} 4.7, CHC*H*H), 2.45 (1H, dd, J_{ab} 14.0, J_{bc} 9.5, CHCH*H*), 3.56 (ABq, 1H, J_{AB} 13.5, ArC*H*H), 3.78 (ABq, 1H, J_{AB} 13.5, ArCH*H*), 3.84 (1H, dd, J_{ac} 4.7, J_{bc} 9.5, C*H*CHH), 4.84 (1H, app s, Olefin C*H*H), 4.88 (1H, app s, OlefinCH*H*),7.25-7.50 (10H, m, ArCH).^{13}C NMR (δ; 100MHz , $CDCl_3$); 22.1 (CH_3), 47.6 (CH), 51.5 (CH_2), 59.3 (CH), 113.4 (CH_2), 126.8 (ArCH), 127.0 (ArCH), 127.3 (ArCH),128.1 (ArCH), 128.3 (ArCH), 128.4 (ArCH), 140.6 (ArC), 142.7 (ArC), 144.3 (C). HRMS $[M+H]^+$calculated for the formula $C_{18}H_{22}N^+$ 252.1762; found 252.1752.[1]

cis-*N*-Benzyl-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*cis*-6a)



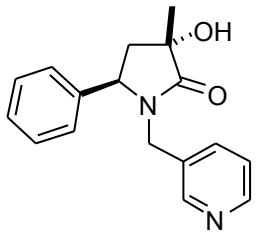
General procedure (**b**) was used. *N*-Benzyl-3-methyl-1-phenylbut-3-en-1-amine **5a** (156 mg, 0.60 mmol), iodine (460 mg, 1.80 mmol), sodium bicarbonate (254 mg, 3.03 mmol), ethyl acetate/petroleum ether 40%, Rf = 0.23, white crystal (mpt 155-156 °C), 63 mg, 37% yield. IR 3286, 3030, 2933, 1671 (s), 1576. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.55 (3H, s, CH_3), 1.92 (1H, dd, J_{ab} 13.0, J_{ac} 8.5, CHC*H*H), 2.59 (1H, dd, J_{ab} 13.0, J_{bc} 6.8, CHCH*H*), 3.57 (ABq, 1H, J_{AB} 14.6, ArCH*H*), 4.53 (1H, dd, J_{ac} 8.5, J_{bc} 6.8, C*H*CHH), 5.09 (ABq, 1H, J_{AB} 14.6, ArC*H*H), 7.04-7.43 (10H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.5 (CH_3), 44.0 (CH_2), 44.5 (CH_2), 57.8 (CH), 74.0 (COH), 126.9 (ArCH), 127.6 (ArCH), 127.9 (ArCH), 128.4 (ArCH), 128.6 (ArCH), 129.0 (ArCH), 135.8 (ArC), 139.0 (ArC), 177.1 (CO). HRMS $[M+H]^+$ calculated for the formula $C_{18}H_{19}NO_2^+$ 282.3519; found 282.3514.

trans-*N*-Benzyl-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*trans*-6a)



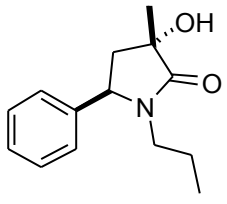
General procedure (**b**) was used. *N*-Benzyl-3-methyl-1-phenylbut-3-en-1-amine **5a** (156 mg, 0.60 mmol), iodine (460 mg, 1.80 mmol), sodium bicarbonate (254 mg, 3.03 mmol), ethyl acetate/ petroleum ether 40%, Rf = 0.25, white crystal (mpt 161-162 °C), 78 mg, 46% yield. IR 3286, 3030, 2933, 1671, 1576.^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.44 (3H, s, CH_3), 2.14 (1H, dd, J_{ab} 13.1, J_{ac} 8.4, CHC*H*H), 2.46 (1H, dd, J_{ab} 13.1, J_{bc} 6.0, CHCH*H*), 3.47 (ABq, 1H, J_{AB} 14.6, ArC*H*H), 4.16 (1H, dd, J_{ac} 8.4, J_{bc} 6.0, C*H*CHH), 5.06 (ABq, 1H, J_{AB} 14.6, ArCH*H*), 7.01-7.42 (10H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 25.2 (CH_3), 43.9 (CH_2), 44.5 (CH_2), 58.1 (CH), 74.3 (COH), 126.9 (ArCH), 127.6 (ArCH), 127.9 (ArCH), 128.2 (ArCH), 128.3 (ArCH), 128.6 (ArCH),129.0 (ArCH), 135.5 (ArC), 139.7 (ArC), 176.9 (CO). HRMS $[M+H]^+$ calculated for the formula $C_{18}H_{19}NO_2^+$ 282.3519; found 282.3514.

cis/trans-N-(3-Pyridyl)-3-hydroxy-3-methyl-5-phenyl-pyrrolidin-2-one (*cis/trans*-6b)



(Mixture of diastereoisomers maj/min) General procedure (**b**) was used. 3-Methyl-1-phenyl-*N*-(pyridin-3-ylmethyl)butan-1-amine **5b** (255 mg,1.01 mmol), iodine (769 mg, 3.03 mmol), sodium bicarbonate (424 mg, 5.05 mmol), ethyl acetate 100%. Rf = 0.2, yellow oil (gum), 0.25 g, 91% yield. IR 3323, 2927, 1668(s).^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.43 (3H, s, C$H_{3\ min}$), 1.56 (3H, s, C$H_{3\ maj}$), 1.94 (1H, dd, J_{ab} 13.8, J_{ac} 7.1, CHC*H*H$_{maj}$), 2.19 (1H, dd, J_{ab} 13.1, J_{ac} 7.7, CHCH*H*$_{min}$), 2.49 (1H, dd, J_{ab} 13.1, J_{bc} 6.8, CHC*H*H$_{min}$), 2.59 (1H, dd, J_{ab} 13.8, J_{bc} 7.4, CHCH*H*$_{maj}$), 3.64 (ABq, 1H, J_{AB} 17.1, PyrCH*H*$_{maj}$), 4.18 (1H, dd, J_{ac} 7.7, J_{bc} 6.8, C*H*CHH$_{min}$), 4.50 (1H, dd, J_{ac} 7.1, J_{bc} 7.4, C*H*CHH$_{maj}$), 4.97 (ABq, 1H, J_{AB} 15.4, PyrC*H*H$_{min}$) 7.06-7.46 (12H, m, ArC*H*), 7.49 (2H, dt, *J* 1.9, 7.9, PyrC*H*), 8.17 (1H, d, *J* 2.0, PyrC*H*), 8.24 (1H, d, *J* 2.0, PyrC*H*), 8.52 (2H, m, overlapping, PyrC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.5 (CH_3), 25.0 (CH_3), 42.1 (CH_2), 42.1 (CH_2), 43.9 (CH_2), 44.0 (CH_2), 58.2 (CH), 58.5 (CH), 73.9 (COH), 74.2 (COH), 123.6 (ArCH), 127.0 (ArCH), 127.7 (ArCH), 128.5 (ArCH), 128.8 (ArCH), 129.2 (ArCH), 131.5 (PyrC), 135.4 (ArCH), 136.1(ArCH), 136.2 (ArC), 138.5 (ArC), 139.2 (ArCH), 139.2 (ArCH), 149.1 (PyrCH), 149.1 (PyrCH), 149.2 (PyrCH), 149.2 (PyrCH), 149.7 (PyrCH), 149.7 (PyrCH), 149.9 (PyrCH), 149.9 (PyrCH), 150.7 (CO), 152.9 (CO). HRMS $[M+H]^+$ calculated for the formula $C_{17}H_{19}N_2O_2^+$ 283.1450; found 283.1447.

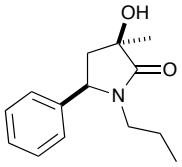
cis-N-Propyl-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*cis*-6c)



General procedure (**b**) was used. *N*-Propyl-3-methyl-1-phenylbut-3-en-1-amine **5c** (800 mg, 3.9 mmol), iodine (300 mg, 11.8 mmol), sodium bicarbonate (1650 mg, 19.6mmol), ethyl acetate/ hexane 50%, Rf = 0.25, yellow solid (mpt 95-96 °C),200 mg,22% yield. IR 3348, 2965, 1673 (s), 1457, 1366. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 0.84 (3H, t, *J* 7.2, CH_2CH_3), 1.39-1.48 (2H, m, CH_2CH_3), 1.49 (3H, s, CH_3), 1.88 (1H, dd, J_{ab}13.7, J_{ac}6.6, CHC*H*H), 2.55-2.70 (2H, m, NCH_2), 3.63 (1H, dt, J_{ab}13.7, J_{bc}6.6, CHCH*H*), 4.76 (1H, dd, J_{ac}6.6, J_{ac}6.6, C*H*CHH), 7.14-7.43 (5H, m, ArC*H*).^{13}C NMR (δ; 100 MHz, $CDCl_3$); 11.1 (CH_3), 19.9 (CH_2), 25.1 (CH_3), 42.4 (CH_2), 44.2

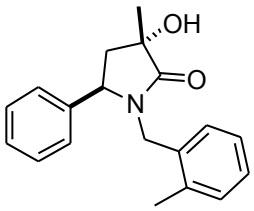
(CH_2), 58.8 (CH), 74.2 (C-OH), 126.7 (ArCH), 128.1 (ArCH), 129.0 (ArCH), 140.2 (ArC), 176.8 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{14}H_{19}NO_2Na^+$ 256.1318; found 256.1313.

trans-N-Propyl-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*trans*-6c)



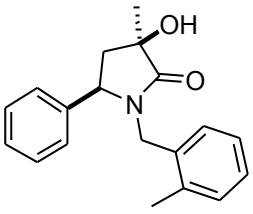
General procedure (**b**) was used. *N*-Propyl-3-methyl-1-phenylbut-3-en-1-amine **5c** (800 mg, 3.9 mmol), iodine (300 mg, 11.8 mmol), sodium bicarbonate (1.65 g, 19.6 mmol), ethyl acetate/hexane 50%. Rf = 0.27, yellow solid (mpt 84-85 °C), 120 mg, 13% yield. IR 3348, 2965, 1673 (s), 1457, 1366. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 0.84 (3H, t, *J* 7.4, CH_2CH_3), 1.38-1.48 (2H, m, CH_2CH_3), 1.49 (3H, s, CH_3), 1.88 (1H, dd, J_{ab} 13.0, J_{ac} 7.0, CHC*H*H), 2.55-2.70 (2H, m, NCH_2), 3.55-3.65 (1H, m, CHCH*H*), 4.46 (1H, t, *J* 7.0, C*H*CHH), 7.24-7.45 (5H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 11.2 (CH_3), 20.0 (CH_2), 24.9 (CH_3), 42.2 (CH_2), 44.5 (CH_2), 58.7 (CH), 74.0 (COH), 127.4 (ArCH), 128.5 (ArCH), 129.0 (ArCH), 139.4 (ArC), 176.8 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{14}H_{19}NO_2Na^+$ 256.1318; found 256.1313.

cis-N-(2-Methylbenzyl)-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*cis*-6d)



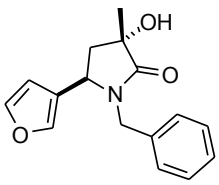
General procedure (**b**) was used. *N*-(2-Methylbenzyl)-3-methyl-1-phenylbut-3-en-1-amine **5d** (245 mg, 0.92 mmol), iodine (703mg, 2.77 mmol), sodium bicarbonate (388 mg, 4.60 mmol), ethyl acetate/petroleum ether 30%, Rf = 0.27, white solid (mpt 161-162 °C), 68mg, 25% yield. IR 3370, 2928, 1675(s), 1456. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.55 (3H, s, CH_3), 1.91 (1H, dd, J_{ab} 13.8, J_{ac} 6.0, CHC*H*H), 2.09 (3H, s, ArCH_3), 2.60 (1H, dd, J_{ab} 13.8, J_{bc} 8.1, CHCH*H*), 3.69 (ABq, 1H, J_{AB} 14.8, ArC*H*H), 4.44 (1H, dd, J_{ac} 6.0, J_{bc} 8.1, C*H*CHH), 5.10 (ABq, 1H, J_{AB} 14.8, ArCH*H*), 6.80 (1H, d, *J* 7.4, ArC*H*), 6.94-7.41 (8H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 19.0 (CH_3), 25.4 (CH_3), 42.5 (CH_2), 43.8 (CH_2), 58.0 (CH), 74.4 (COH), 125.8 (ArCH), 126.7 (ArCH), 127.7 (ArCH), 128.0 (ArCH), 129.0 (ArCH), 129.1(ArCH), 130.4 (ArCH),133.0 (ArC),136.9 (ArC), 140.0 (ArC), 176.2 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{19}H_{21}NO_2Na^+$ 318.1469; found 318.1470.

trans-N-(2-Methylbenzyl)-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*trans*-6d)



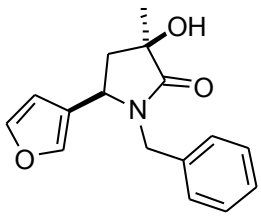
General procedure (**b**) was used. *N*-(2-Methylbenzyl)-3-methyl-1-phenylbut-3-en-1-amine **5d** (245 mg, 0.92 mmol), iodine (703 mg, 2.77 mmol), sodium bicarbonate (388 mg, 4.60 mmol), ethyl acetate/petroleum ether 30%, Rf = 0.27, colourless crystals (mpt 132-133 °C), 50 mg, 18% yield. IR 3370, 2926, 1675(s), 1456.^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.49 (3H, s, CH_3), 1.96 (1H, dd, J_{ab} 13.8, J_{ac} 6.1, CHC*H*H), 2.14 (3H, s, ArCH_3), 2.48 (1H, dd, J_{ab} 13.8, J_{bc} 8.0, CHCH*H*), 3.69 (ABq, 1H, J_{AB} 14.8, ArC*H*H), 4.13 (1H, dd, J_{ac} 6.1, J_{bc} 8.0, C*H*CHH), 4.44 (ABq, 1H, J_{AB} 14.8, ArCH*H*), 6.79 (1H, d, *J* 7.4, ArC*H*), 6.94-7.41 (8H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 18.9 (CH_3), 25.0 (CH_3), 42.1 (CH_2), 44.2 (CH_2), 58.0 (CH), 74.2 (COH), 125.8 (ArCH), 127.6 (ArCH), 127.8 (ArCH), 128.4 (ArCH), 128.9 (ArCH), 129.5 (ArCH), 130.5 (ArCH), 133.2 (ArC), 136.8 (ArC), 139.4 (ArC), 176.9 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{19}H_{21}NO_2Na^+$ 318.1469; found 318.1470.

cis-N-Benzyl-5-(furan-3-yl)-3-hydroxy-3-methylpyrrolidin-2-one (*cis*-6e)



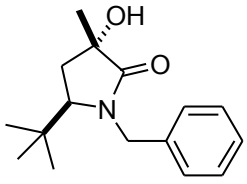
General procedure (**b**) was used. *N*-Benzyl-3-methyl-1-(furan-3-yl) but-3-en-1-amine **5e** (300 mg, 1.24 mmol), iodine (950 mg, 3.70 mmol), sodium bicarbonate (520 mg, 6.20mmol), ethyl acetate/petroleum ether 30%, Rf = 0.27, light yellow crystals (mpt 131-132 °C), 60 mg, 40% yield. IR 3356, 2932, 1663(s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.55 (3H, s, CH_3), 1.95 (1H, dd, J_{ab} 13.2, J_{ac} 8.1, CHC*H*H), 2.50 (1H, dd, J_{ab} 13.2, J_{bc} 6.9, CHCH*H*), 3.68 (ABq, 1H, J_{AB} 14.8, ArC*H*H), 4.55 (1H, J_{ac} 8.1, J_{bc} 6.9, C*H*CHH), 5.03 (ABq, 1H, J_{AB} 14.8, ArCH*H*), 6.23 (1H, dd, *J* 0.6, 1.5, FurC*H*), 7.08-7.48 (5H, overlapping m, ArC*H* & FurC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 25.1 (CH_3), 42.3 (CH_2), 44.4 (CH_2), 49.6 (CH), 74.2 (COH), 108.3 (FurCH), 124.3 (FurC), 127.6 (ArCH), 128.2 (ArCH), 128.7 (ArCH), 135.8(ArC), 140.8 (FurCH), 144.3(FurCH), 176.3 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{16}H_{17}NO_3Na^+$ 294.1105; found 294.1106.

trans-N-Benzyl-5-(furan-3-yl)-3-hydroxy-3-methylpyrrolidin-2-one (*trans*-6e)



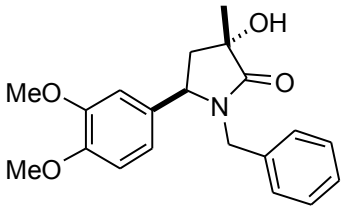
General procedure (**b**) was used. *N*-Benzyl-3-methyl-1-(furan-3-yl) but-3-en-1-amine **5e** (300 mg, 1.24 mmol), iodine (950 mg, 3.70 mmol), sodium bicarbonate (520 mg, 6.20mmol), ethyl acetate/petroleum ether 30%, Rf = 0.25, white crystals (mpt 120-121 °C), 40 mg, 35% yield. IR 3356, 2932, 1663(s).^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.44 (3H, s, CH_3), 2.17 (1H, dd, J_{ab}13.2, J_{ac}8.1, CHC*H*H), 2.38 (1H, dd, J_{ab}12.9, J_{bc}6.9, CHCH*H*), 3.61 (ABq, 1H, J_{AB} 14.6, ArC*H*H), 4.25 (1H, dd, J_{ac}8.1, J_{bc}6.9, C*H*CHH), 5.02 (ABq, 1H, J_{AB}14.6, ArCH*H*), 6.39 (1H, dd, *J* 0.6, 1.5, FurC*H*), 7.02-7.35 (5H, m, ArC*H*), 7.44 (2H, t, *J* 1.5, FurC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.4 (CH_3), 42.4 (CH_2), 44.3 (CH_2), 48.9 (CH), 73.9 (COH), 108.8 (FurCH), 123.7 (FurC), 127.7 (ArCH), 128.3 (ArCH), 128.7 (ArCH), 136.1 (ArC), 141.3 (FurCH), 144.3 (FurCH), 176.8 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{16}H_{17}NO_3Na^+$ 294.1105; found 294.1106.

cis/trans-N-Benzyl-5-(tert-butyl)-3-hydroxy-3-methylpyrrolidin-2-one (*cis/trans*-6f)



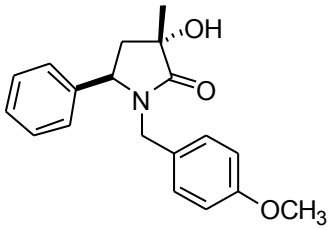
(Mixture of diastereoisomers maj/min) General procedure (**b**) was used. N-Benzyl-2,2,5-trimethylhex-5-en-3-amine **5f** (100 mg, 0.68 mmol), iodine (520 mg, 2.06 mmol), sodium bicarbonate (288 mg, 3.43 mmol), ethyl acetate/petroleum ether 80%, Rf = 0.65, pale yellow crystal, 120 mg, 67% yield. IR 3352, 2902, 1662(s). ^{1}HNMR (δ; 300 MHz, $CDCl_3$); 0.89 (9H, s, $CH_{3\ min}$), 0.93 (9H, s, $CH_{3\ maj}$), 1.45 (3H, s, $CH_{3\ min}$), 1.56 (3H, s, $CH_{3\ maj}$), 1.77 (1H, dd, J_{ab} 14.1, J_{ac} 7.3, CHC*H*H$_{min}$), 2.03 (1H, dd, J_{ab}4.8, J_{bc}8.2, CHCH*H*$_{min}$), 2.45 (1H, dd, J_{ab} 13.1, J_{ac} 6.9, CHC*H*H$_{maj}$), 2.59 (1H, dd, J_{ab} 13.1, J_{bc}7.8, CHCH*H*$_{maj}$), 3.21 (1H, dd, J_{ac} 7.3, J_{bc} 8.2, C*H*CHH $_{min}$), 4.17 (1H, t, *J* 7.3, C*H*CHH$_{maj}$), 5.06 (ABq, 2H, J_{AB} 14.4, ArC*HH*$_{maj}$), 5.30 (ABq, 2H, J_{AB} 15.7, ArC*HH* $_{min}$), 6.87-7.44 (10H, m, ArCH). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 22.1 (CH_3), 22.6 (CH_3), 26.9 (CH_3), 27.1 (CH_3), 36.6 (CH), 37.1 (CH), 44.3 (CH_2), 44.4 (CH_2), 62.3 (CH), 63.0 (CH), 74.1 (COH), 74.3 (COH), 127.0 (ArCH), 127.4 (ArCH), 127.7 (ArCH), 128.3 (ArCH), 128.5 (ArCH), 128.7 (ArCH), 129.0 (2ArCH), 139.1 (ArC), 139.8 (ArC), 177.0 (CO), 177.5 (CO). MS(ES+) $[M+Na]^+$ formula $C_{16}H_{23}NO_2Na^+$ found 284.15.

cis/trans-N-Benzyl-5-(3,4-dimethoxyphenyl)-3-hydroxy-3-methylpyrrolidin-2-one (*cis/trans*-6g)



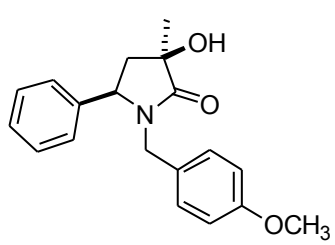
(Mixture of diastereoisomers maj/min) General procedure (**b**) was used.*N*-benzyl-1-(3,4-dimethoxyphenyl)-3-methylbut-3-en-1-amine **5g** (155 mg, 0.49 mmol), iodine (379 mg, 1.49 mmol), sodium bicarbonate (209 mg, 2.48 mmol), methanol/dichloromethane 5%, pale yellow precipitate, 110 mg, 65% yield. IR 3380, 1680(s), 1682(s).^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.45 (3H, s, C$H_{3\ min}$), 1.58 (3H, s, C$H_{3\ maj.}$), 1.92 (1H, dd, J_{ab}13.8, J_{ac}6.8, CHCHH$_{maj.}$), 2.18 (1H, dd, J_{ab}13.0, J_{bc} 8.2, CHCH$H_{maj.}$), 2.45 (1H, dd, J_{ab} 13.8, J_{ac} 6.8, CHCHH$_{min}$), 2.58 (1H, dd, J_{ab} 13.8, J_{bc} 7.5, CHCHH_{min}), 3.53 (ABq, 1H, J_{AB}14.8, PhCHH $_{min}$), 3.64 (ABq, 1H, J_{AB}14.8, PhCHH $_{maj}$), 3.83 (6H, s, OC$H_{3\ min}$), 3.92 (6H, s, OCH_3 $_{maj}$), 4.14 (1H, dd, J_{ac}6.8, J_{bc}7.5, CHCHH $_{min}$), 4.49 (1H, t, J 6.8, CHCHH $_{maj}$), 5.02 (1H, t, J 13.8, ArCHH $_{maj+min}$), 6.54 (2H, d, J 1.6, ArCH), 6.65-6.76 (2H, m, ArCH), 6.86 (2H, dd, J_{ab}3.0, J_{ac}8.2, ArCH), 6.98-7.03 (2H, m, ArCH), 7.05-7.11 (2H, m, ArCH), 7.28 (6H, d, J 3.7, ArCH).^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.4 (CH_3), 25.2 (CH_3), 29.7 (CH_2), 43.8 (CH_2), 43.9 (CH_2), 44.6 (CH), 44.7 (CH), 55.9 (2OCH_3), 57.9 (CH), 58.2 (CH), 74.1 (COH), 74.4 (COH), 109.6 (ArCH), 110.0 (ArCH), 110.8 (ArCH), 111.0 (ArCH), 111.3 (ArCH), 119.5 (ArCH), 120.5 (ArCH), 127.6 (ArCH), 128.0 (2ArCH), 128.4 (ArCH), 128.5 (ArC), 128.6 (ArCH), 128.9 (ArCH), 129.0 (ArC), 148.9 (ArCOCH_3), 149.1(ArC), 149.5 (ArCOCH_3), 176.6 (CO), 177.1 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{20}H_{24}NO_4Na^+$ 364.1519; found 364.1519.

cis-N-(4-Methoxybenzyl)-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*cis*-6h)



General procedure (**b**) was used. *N*-(4-Methoxylbenzyl)-3-methyl-1-phenylbut-3-en-1-amine **5h** (280 mg, 0.99 mmol), iodine (758 mg, 2.98 mmol), sodium bicarbonate (417 mg, 4.97 mmol), ethyl acetate/petroleum ether 40%, Rf = 0.23, colorless crystal (mpt 141-142 °C), 70 mg, 35% yield.. IR 3377, 2929, 1680(s).^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.55 (3H, s, CH_3), 1.90 (1H, dd, J_{ab} 13.7, J_{ac} 6.6, CHCHH), 2.57 (1H, dd, J_{ab} 13.8, J_{bc} 7.8, CHCHH), 3.51 (ABq, 1H, J_{AB} 14.5, ArCHH), 3.77 (3H, s, OCH_3), 4.51 (1H, t, J 7.2, CHCHH), 5.04 (ABq, 1H, J_{AB} 14.5, ArCHH), 6.78 (2H, d, J 8.6, ArCH), 6.98 (2H, d, J 8.6, ArCH), 7.06-7.17 (2H, m, ArCH), 7.29-7.43 (3H, m, ArCH). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 25.2(CH_3), 44.0 (CH_2), 55.2 (CH), 58.1 (CH), 74.3 (COH), 114.0 (ArCH), 127.0 (ArCH), 127.7 (ArC), 128.2 (ArCH), 129.0 (ArCH), 129.8 (ArCH), 140.0 (ArC), 159.0 (ArC), 176.8 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{19}H_{21}NO_3Na^+$ 334.1421; found 334.1419.

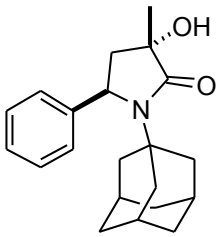
trans-N-(4-Methoxybenzyl)-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*trans*-6h)



General procedure (**b**) was used. *N*-(4-Methoxylbenzyl)-3-methyl-1-phenylbut-3-en-1-amine **5h** (280 mg, 0.99 mmol), iodine (758 mg, 2.98 mmol), sodium bicarbonate (417 mg, 4.97 mmol), ethyl acetate/ petroleum ether 40%, Rf = 0.25, colorless crystal (mpt 159-160 °C), 51 mg, 45% yield. IR 3377, 2929, 1680(s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.43 (3H, s, CH_3), 2.13 (1H, dd, J_{ab} 13.0, J_{ac} 6.6,

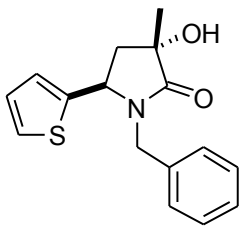
CHC*H*H), 2.45 (1H, dd, J_{ab} 13.0, J_{bc} 6.8, CHCH*H*), 3.41 (ABq, 1H, J_{AB} 14.4, ArC*H*H), 3.78 (3H, s, OCH_3), 4.14 (1H, dd, J_{ac} 6.6, J_{bc} 6.8, C*H*CHH), 5.01 (ABq, 1H, J_{AB} 14.4, ArCH*H*), 6.79 (2H, d, *J* 8.7, ArC*H*), 6.90 (2H, d, *J* 8.6, ArC*H*), 7.17-7.24 (2H, m, ArC*H*), 7.29-7.37 (3H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.4 (CH_3), 43.9 (CH_2), 44.2 (CH_2), 55.2(CH_3), 74.1 (COH), 114.0 (ArCH), 127.7 (ArCH), 128.0 (ArC), 128.4 (ArCH), 129.0 (ArCH), 129.8 (ArCH), 139.2 (ArC), 159.1 (ArC), 177.30 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{19}H_{21}NO_3Na^+$ 334.1421; found 334.1419.

cis/trans-N-(Adamantan-1-yl)-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*cis/trans*-6i)



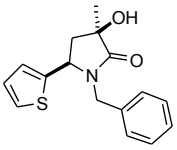
(Mixture of diastereoisomers maj/min) General procedure (**b**) was used. *N*-(Admantan-1-yl) phenylbut-3-ene **5i** (237 mg, 0.80 mmol), iodine (610 mg, 2.40 mmol), sodium bicarbonate (337 mg, 4.00 mmol), ethyl acetate (50 mL), ethylacetate/hexane 50%, white precipitate, 150 mg, 57% yield. IR 2904, 2849, 1657(s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.20-2.30 (24H, overlapping m, AdmantCH_2), 2.44 (1H, dd, J_{ab} 13.4, J_{ac} 8.3, CHC*H*H_{maj}), 2.63 (1H, dd, J_{ab} 12.9, J_{ac} 10.0, CHC*H*H_{min}), 2.73 (1H, dd, J_{ab} 13.4, J_{bc} 8.3, CHCH*H*$_{maj}$), 4.19 (1H, dd, J_{ab} 12.9, J_{bc} 9.8, CHCH*H*$_{min}$), 4.47-4.55 (1H, m, C*H*), 4.63-4.71 (1H, m, C*H*), 4.74 (1H, dd, J_{ac} 8.3, J_{bc}8.3, C*H*CHH_{maj}), 4.77 (1H, d, J_{ac} 10.0, J_{bc} 9.8, C*H*CHH $_{min}$), 5.04 (3H, d, *J* 9.6, C*H*), 7.08-7.53 (10H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 19.9 (CH_3), 26.5 (CH_3), 29.5 (CH), 29.7 (CH), 29.9 (CH), 36.2 (CH), 36.7 (CH), 41.8 ($2CH_2$), 42.2 ($2CH_2$), 49.5 (CH_2), 49.6 (CH_2), 54.6 (CH_2), 57.3 (CH), 59.3(CH), 77.8 (CH_2), 87.0 (CH), 88.7 (CH), 125.4 (ArCH), 126.1 (ArCH), 126.2 (ArCH), 126.6 (ArCH), 127.1 (ArCH), 127.2 (ArCH), 127.4 (ArCH), 127.8 (ArCH), 127.9 (ArCH), 128.0 (ArCH), 128.2 (ArCH), 128.7 (ArCH), 144.9 (ArC), 149.0 (ArC), 177.3 (CO), 177.8 (CO). HRMS $[M+H]^+$ calculated for the formula $C_{21}H_{28}NO_2^+$ 326.2122; found 326.2120.

cis-N-Benzyl-3-hydroxy-3-methyl-5-(thiophen-2-yl) pyrrolidin-2-one (*cis*-6j)



General procedure (**b**) was use. *N*-Benzyl-3-methyl-1-(thiophen-2-yl) but-3-en-1-amine **5j** (156 mg, 0.6mmol), iodine (460 mg, 1.80 mmol), sodium bicarbonate (254 mg, 3.03 mmol), ethyl acetate/ petroleum ether 40%, Rf = 0.35, white crystal (mpt 142-143 °C), 27 mg, 36% yield. IR 3292, 2940, 1671 (s).^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.45 (3H, s, CH_3), 2.31 (1H, dd, J_{ab} 13.0, J_{ac} 8.6, CHC*H*H), 2.53 (1H, dd, J_{ab} 13.0, J_{bc} 6.7, CHCH*H*), 3.59 (ABq, 1H, J_{AB} 14.7, ArC*H*H), 4.52 (1H, dd, J_{ac} 8.6, J_{bc} 6.7, C*H*CHH), 5.05 (ABq, 1H, J_{AB} 14.7, ArC*H*H), 6.88-7.12 (3H, m, ThioC*H*), 7.22-7.39 (5H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.2 (CH_3), 44.4 (CH_2), 44.9 (CH_2), 53.1 (CH), 73.8 (COH), 126.4 (ArCH), 126.8 (ArCH), 127.5 (ArCH), 127.7 (ArCH), 128.4 (ThioCH), 128.7 (ThioCH), 129.0 (ThioCH), 136.0 (ThioC), 142.7 (ArC), 176.9 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{16}H_{17}NO_2NaS^+$ 310.0876; found 310.0878.

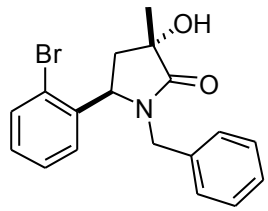
trans-N-Benzyl-3-hydroxy-3-methyl-5-(thiophen-2-yl) pyrrolidin-2-one (*trans*-6j)



General procedure (**b**) was used. *N*-Benzyl-3-methyl-1-(thiophen-2-yl) but-3-en-1-amine **5j** (156 mg, 0.60 mmol), iodine (460 mg, 1.80mmol), sodium bicarbonate (254 mg, 3.03 mmol),

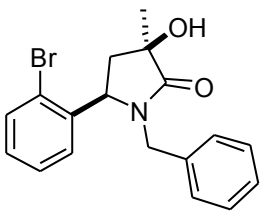
ethyl acetate/ petroleum ether 40%, Rf = 0.25, white crystal (mpt 134-135 °C), 23 mg 30% yield. IR 3292, 2940, 1671 (s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.62 (3H, s, CH_3), 2.09 (1H, dd, J_{ab}13.1, J_{ac}6.3, CHC*H*H), 2.65 (1H, dd, J_{ab} 13.1, J_{bc}7.7, CHCH*H*), 3.63 (ABq, 1H, J_{AB} 14.7, ArC*H*H), 4.86 (1H, t, *J* 7.1, C*H*CHH), 5.20 (ABq, 1H, J_{AB} 14.7, ArCH*H*), 6.80-7.36 (3H, m, ThioC*H*), 7.36-7.38 (5H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.0 (CH_3), 44.2 (CH_2), 44.7 (CH_2), 53.0 (CH), 73.9 (COH), 126.9 (ArCH), 127.0 (ArCH), 127.3 (ArCH), 127.9 (2ArCH), 128.5 (ThioCH), 128.6 (ThioCH), 129.0(ThioCH), 136.4 (ThioC), 142.9 (ArC), 177.1 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{16}H_{17}NO_2NaS^+$ 310.0876; found 310.0878.

cis-*N*-Benzyl-5-(2-bromophenyl)-3-hydroxy-3-methylpyrrolidin-2-one (*cis*-6k)



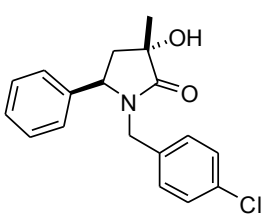
General procedure (**b**) was used. *N*-Benzyl-3-methyl-1-(2-bromophenyl)but-3-en-1-amine **5k** (200 mg, 0.60 mmol), iodine (460 mg, 1.8 mmol), sodium bicarbonate (250 mg, 3.0 mmol), ethyl acetate/petroleum ether 40%, Rf = 0.18,white crystal (mpt 125-126 °C), 74 mg, 34% yield. IR 3327, 2926, 1679(s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.45 (3H, s, CH_3), 1.89 (1H, dd, J_{ab}13.5, J_{ac}3.7, CHC*H*H), 2.66 (1H, dd, J_{ab}13.5, J_{bc} 8.9, CHCH*H*), 3.67 (ABq, 1H, J_{AB} 14.5, ArC*H*H), 4.91 (1H, dd, J_{ac} 3.7, J_{bc} 8.9, C*H*CHH), 5.16 (ABq, 1H, J_{AB} 14.5, ArC*H*H), 7.01-7.42 (8H, m, ArC*H*), 7.59 (1H, dd, *J* 8.0, 1.0, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.8 (CH_3), 42.6 (CH_2), 45.0 (CH_2), 56.3 (COH), 123.4 (ArC), 127.8 (ArCH), 128.0 (ArCH), 128.3 (ArCH), 128.6 (ArCH), 128.7 (ArCH), 129.4 (ArCH), 133.1 (ArCH), 135.3 (ArC), 147.0 (ArC), 176.5 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{18}H_{18}NO_2BrNa^+$ 282.0410; found 282.0419.

trans-*N*-Benzyl-5-(2-bromophenyl)-3-hydroxy-3-methylpyrrolidin-2-one (*trans*-6k)



General procedure (**b**) was used. *N*-Benzyl-3-methyl-1-(2-bromophenyl)but-3-en-1-amine **5k** (200 mg, 0.60 mmol), iodine (460 mg, 1.8 mmol), sodium bicarbonate (250 mg, 3.0 mmol), ethyl acetate/petroleum ether 40%, Rf = 0.25, white crystal (mpt 112-113 °C), 48 mg, 21% yield. IR 3327, 2926, 1679(s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.43 (3H, s, CH_3), 1.94 (1H, dd, J_{ab} 13.2, J_{ac} 7.3, CHC*H*H), 2.52 (1H, dd, J_{ab} 13.2, J_{bc} 7.3, CHCH*H*), 3.58 (ABq, 1H, J_{AB} 14.4, ArC*H*H), 4.73 (1H, t, *J* 7.2, C*H*CHH), 5.09 (ABq, 1H, J_{AB} 14.4, ArCH*H*), 6.99 (2H, dd, *J* 6.5, 2.8, ArC*H*), 7.15-7.45 (5H, m, ArC*H*), 7.55 (1H, d, *J* 7.3, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.8 (CH_3), 42.6 (CH_2), 45.0 (CH_2), 56.3 (COH), 123.4 (ArC), 127.8 (ArCH), 128.0 (ArCH), 128.3 (ArCH), 128.6 (ArCH), 128.7 (ArCH), 129.4 (ArCH), 133.1 (ArCH), 135.3 (ArC), 147.0 (ArC), 176.5 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{18}H_{18}NO_2BrNa^+$ 282.0410; found 282.0419.

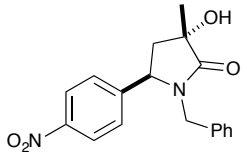
cis-*N*-(4-Chlorobenzyl)-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (*cis*-6l)



(Mixture of diastereoisomers maj/min) General procedure (**b**) was used. *N*-(4-Chlorobenzyl)-3-methyl-1-phenylbut-3-en-1-amine **5l** (160 mg, 0.56 mmol), iodine (426 mg, 1.68 mmol), sodium bicarbonate (235 mg, 2.79 mmol), ethyl acetate/hexane 50%, white precipitate,113 mg, 64% yield. IR 3286, 1681(s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$);

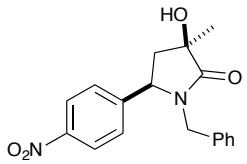
1.43 (3H, s, C$H_{3\ min}$), 1.58 (3H, s, C$H_{3\ maj}$), 1.94 (1H, dd, J_{ab} 13.8, J_{ac} 7.0, CHCHH$_{maj}$), 2.16 (1H, dd, J_{ab} 13.1, J_{ac} 8.5, CHCHH_{min}), 2.48 (1H, dd, J_{ab}13.1, J_{bc} 8.3, CHCHH$_{min}$), 2.61 (1H, dd, J_{ab} 13.8, J_{bc} 7.5, CHCHH_{maj}), 3.47(ABq, 1H, J_{AB} 14.5, ArCHH$_{min}$), 3.57 (ABq, 1H, J_{AB}14.7, ArCHH$_{maj}$), 4.14 (1H, dd, J_{ac}8.5, J_{bc}8.3, CHCHH$_{min}$), 4.54 (1H, dd, J_{ac}7.0, J_{bc}7.5, CHCHH$_{maj}$), 4.97 (ABq, 1H, J_{AB} 9.6, ArCHH_{min}), 5.02 (ABq, 1H, J_{AB} 9.8, ArCHH_{maj}), 6.91 (2H, d, J 8.5, ArCH), 7.00 (2H, d, J 8.5, ArCH), 7.10 (2H, d, J 8.1, ArCH), 7.12-7.38 (12H, m, ArCH). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.5 (CH_3), 25.1 (CH_3), 43.9 (CH_2), 44.1 (CH_2), 58.0 (CH), 58.3 (CH), 74.0 (COH), 74.3 (COH), 127.0 (ArCH), 127.7 (ArCH), 128.4 (ArCH), 128.6 (ArCH), 128.8 (ArCH), 129.1 (ArCH), 129.7 (ArCH), 129.9 (ArCH), 133.5 (ArC), 133.6 (ArC), 134.2 (ArC), 134.4 (ArC), 138.7 (ArC), 139.4 (ArC), 176.7 (CO), 176.8 (CO).HRMS $[M+H]^+$ calculated for the formula $C_{18}H_{19}ClNO_2^+$ 316.1110; found 316.1104.

cis-N-Benzyl-3-hydroxy-3-methyl-5-(4-nitrophenyl) pyrrolidin-2-one (*cis*-6m)



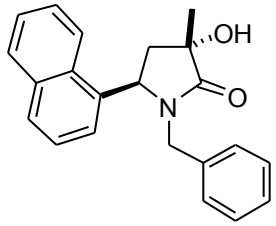
General procedure (**b**) was used. *N*-Benzyl-3-methyl-1-(4-nitrophenyl)but-3-en-1-amine **5m** (300 mg, 1.0 mmol), iodine (770 mg, 3.0 mmol), sodium bicarbonate (425 mg, 5.0 mmol), ethyl acetate/petroleum ether 60%, Rf = 0.25, light brown precipitate (mpt 238-239 ˚C), 88 mg, 25% yield. IR 3386, 2928, 1681(s), 1527, 1349, 698. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.57 (3H, s, CH_3), 1.88 (1H, dd, J_{ab} 13.8, J_{ac} 6.7, CHCHH), 2.65 (1H, dd, J_{ab} 13.8, J_{bc} 7.8, CHCHH), 3.60 (ABq, 1H, J_{AB} 14.7, ArCHH), 4.63 (1H, t, J 7.2, CHCHH), 5.16 (ABq, 1H, J_{AB} 14.7, ArCHH), 6.99-7.09 (2H, m, ArCH), 7.24- 7.37 (5H, m, ArCH), 8.26 (2H, d, J 8.7, ArCH). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 25.2 (CH_3), 43.7 (CH_2), 45.0 (CH_2), 57.6 (CH), 74.1 (COH), 124.4 (2ArCH), 127.7 (2ArCH), 127.9 (2ArCH), 128.2 (2ArCH), 128.8 (ArCH), 134.9 (ArC), 147.4 (ArC), 147.8 ($ArCNO_2$), 176.7 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{18}H_{18}N_2O_4Na^+$ 349.1166; found 349.1164.

trans-N-Benzyl-3-hydroxy-3-methyl-5-(4-nitrophenyl) pyrrolidin-2-one (*trans*-6m)



General procedure (**b**) was used. *N*-benzyl-3-methyl-1-(4-nitrophenyl)but-3-en-1-amine **5m** (300 mg, 1.0 mmol), iodine (770 mg, 3.0 mmol), sodium bicarbonate (425 mg, 5.0 mmol), ethyl acetate/ petroleum ether 60%, yellow oil, 100 mg, 30% yield. IR 3386, 2928, 1681(s), 1527, 1349, 698. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.47 (3H, s, CH_3), 2.10 (1H, dd, J_{ab} 13.2, J_{ac} 7.6, CHCHH), 2.50 (1H, dd, J_{ab} 13.3, J_{bc} 7.3, CHCHH), 3.54 (ABq, 1H, J_{AB} 14.6, ArCHH), 4.28 (1H, t, J 7.4, CHCHH), 5.16 (ABq, 1H, J_{AB} 14.6, ArCHH), 7.17- 7.50 (2H, m, ArCH), 7.35-7.15 (5H, m, ArCH), 8.24 (2H, d, J 6.9, ArCH). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 25.2 (CH_3), 43.6 (CH_2), 44.9 (CH_2), 57.6 (CH), 74.0 (COH), 124.3 (2ArCH), 127.7 (2ArCH), 127.9 (2ArCH), 128.2 (2ArCH), 128.8 (ArCH), 134.9 (ArC), 147.4 (ArC), 147.8 (ArC), 176.7 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{18}H_{18}N_2O_4Na^+$ 349.1166; found 349.1164.

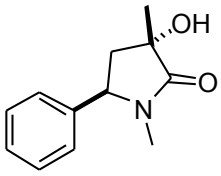
cis/trans-N-Benzyl-3-hydroxy-3-methyl-5-(naphthalen-1-yl)pyrrolidin-2-one (*cis/trans*-6n)



(Mixture of diastereoisomers maj/min) General procedure (**b**) was used. *N*-benzyl-3-methyl-1-(naphthalen-1-yl)but-3-en-1-amine **5n** (126 mg, 0.418 mmol), iodine (318

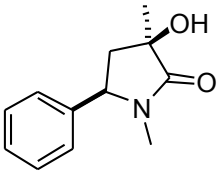
mg, 1.2 mmol), sodium bicarbonate (175 mg, 2.09 mmol), ethyl acetate/hexane 50%, white precipitate,100 mg, 72% yield. IR 3355, 2985, 1676(s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.53 (3H, s, C$H_{3\ maj.}$), 1.95 (3H, s, C$H_{3\ min}$), 2.17 (1H, dd, J_{ab}13.4, J_{ac}7.2, CHCHH $_{maj}$), 2.50 (1H, dd, J_{ab}13.1, J_{ac}7.1, CHCHH$_{min}$), 2.64 (1H, dd, J_{ab} 13.4, J_{bc} 7.4, CHCHH_{maj}), 2.82 (1H, dd, J_{ab}13.1, J_{bc} 9.9, CHCHH_{min}), 3.35 (1H, dd, J 14.4, ArCHH$_{min}$), 3.70 (2H, d, J 14.4, ArCHH$_{maj}$), 4.61 (1H, dd, J_{ac}7.1, J_{bc}9.9, CHCHH$_{min}$), 5.41 (1H, dd, J_{ac}7.2, J_{bc}7.4, CHCHH$_{maj}$), 5.23 (2H, d, J 14.5, ArCHH_{maj}), 5.34 (2H, d, J 14.5, ArCHH_{min}), 6.93 (4H, t, J 7.5, NaphCH), 7.06- 8.24 (20H, m, ArCH). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.9 (CH_3), 25.0 (CH_3), 41.4 (CH_2), 44.0 (CH_2), 44.9 (CH_2), 45.0 (CH_2), 45.3 (CH_2), 52.3 (CH), 60.5 (CH), 74.3 (COH), 121.7 (ArCH), 123.1 (ArCH), 123.8 (ArCH), 124.9 (ArCH), 125.8 (ArCH), 126.4 (ArCH), 126.6 (ArCH), 127.0 (ArCH), 127.7 (ArCH), 128.4 (ArCH), 128.5 (ArCH), 128.6 (ArCH), 128.7 (ArCH), 129.0 (ArCH), 129.5 (ArCH), 130.1 (ArC), 131.0 (ArC), 133.9 (ArC), 135.6 (ArC), 135.9 (ArC), 177.4 (2CO). HRMS $[M+H]^+$ calculated for the formula $C_{22}H_{22}NO_2^+$ 332.4150; found 332.41314.

***cis*-3-Hydroxy-1,3-dimethyl-5-phenylpyrrolidin-2-one (*cis*-6o)**



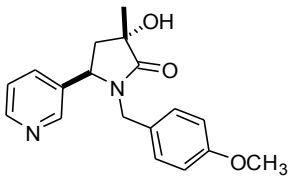
General procedure (**b**) was used. *N*, 3-dimethyl-1-phenylbut-3-en-1-amine **5o** (500 mg, 2.80 mmol), iodine (2.15 mg, 8.5 mmol), sodium bicarbonate (1.18 g, 14.1 mmol), ethyl acetate/petroleum ether 70%, Rf = 2.28, white crystal (mpt 159-160 ˚C). 135 mg, 24% yield. IR 3306, 2928, 1668, 1454, 1256. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.48 (3H, s, CH_3), 2.13 (1H, dd, J_{ab} 13.2, J_{ac} 8.1, CHCHH), 2.54 (1H, dd, J_{ab} 13.2, J_{bc} 6.9, CHCHH), 2.65 (3H, s, NCH_3), 4.34 (1H, dd, J_{ac} 8.1, J_{bc} 6.9, CHCHH), 7.22-7.43 (5H, m, ArCH). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 25.3 (CH_3), 28.7 (CH_3), 44.3 (CH_2), 61.4 (CH), 74.1 (COH), 126.5 (ArCH), 128.1 (ArCH), 129.1 (ArCH), 140.3 (ArC), 177.0 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{12}H_{15}NO_2Na^+$ 228.0996; found 228.1000.

***trans*-3-Hydroxy-1,3-dimethyl-5-phenylpyrrolidin-2-one (*trans*-6o)**



General procedure (**b**) was used. *N*, 3-dimethyl-1-phenylbut-3-en-1-amine **5o** (500 mg, 2.80 mmol), iodine (2150 mg, 8.47 mmol), sodium bicarbonate (1180 mg, 14.10 mmol), ethyl acetate/petroleum ether 70%, Rf = 0.29, white crystal (mpt 168-169 ˚C), 118 mg, 19% yield. IR 3306, 2928, 1668, 1454, 1256. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.51 (3H, s, CH_3), 1.90 (1H, dd, J_{ab} 13.2, J_{ac} 7.0, CHCHH), 2.66 (1H, dd, J_{ab} 13.8, J_{bc} 7.7, CHCHH), 2.72 (3H, s, NCH_3), 4.65 (1H, dd, J_{ac}7.0, J_{bc}7.7, CHCHH), 7.12-7.44 (5H, m, ArCH). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.8 (CH_3), 28.4 (CH_3), 44.4 (CH_2), 61.0 (CH), 74.1 (COH), 127.3 (ArCH), 128.5 (ArCH), 129.0 (2ArCH), 139.6 (ArC), 177.3 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{12}H_{15}NO_2Na^+$ 228.0996; found 228.1000.

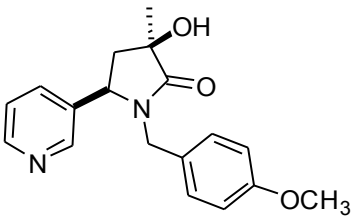
***cis-N*-(4-Methoxybenzyl)-3-hydroxy-3-methyl-5-(pyridin-3-yl)pyrrolidin-2-one (*cis*-6p)**



General procedure (**b**) was used. *N*-benzyl-3-methyl-1-phenylbut-3-en-1-amine **5p** (350 mg, 1.24 mmol), iodine (940 mg, 3.70 mmol), sodium bicarbonate (520 mg, 6.19 mmol), the product was separated with crystalisation from dichloromethane/petroleum ether 50%, white crystal (mpt 172-173 ˚C), 150 mg, 51% yield. IR 2934, 1689 (s), 1611, 1512, 1431, 1245. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.39 (3H, s, CH_3), 2.08 (1H, dd, J_{ab} 13.8, J_{ac} 6.9,

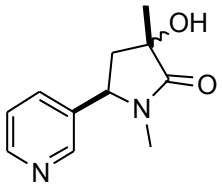
CHC*H*H), 2.41 (1H, dd, J_{ab} 13.8, J_{bc} 7.6, CHCH*H*), 3.35 (ABq, 1H, J_{AB} 14.5, ArC*H*H), 3.71 (3H, s, OCH_3), 4.13 (1H, t, *J* 7.5, C*H*CHH), 4.96 (ABq, 1H, J_{AB} 14.5, ArCH*H*), 6.77 (4H, dt, *J* 1.9, 1.9, ArC*H*), 7.38 (2H, dt, *J* 1.9 ,1.9, PyrC*H*), 8.38(1H, d, *J* 1.9, PyrC*H*), 8.55 (1H, dd, *J* 4.7, 1.6, PyrC*H*). ^{13}C NMR (δ; 100 MHz, CDCl3); 24.9 (CH_3), 43.9 (CH_2), 44.1 (CH_2), 55.2 (CH_3), 55.9 (OCH_3), 74.0 (COH), 114.1 (ArCH), 123.9 (ArCH), 127.1 (ArC), 129.6 (ArCH), 134.6 (ArCH), 135.4 (ArC), 149.0 (ArCH), 149.7 (ArCH), 159.1 (ArC), 176.8 (CO). HRMS $[M+H]^+$ calculated for the formula $C_{18}H_{21}N_2O_3^+$ 313.1541; found 313.1552.

trans-N-(4-Methoxybenzyl)-3-hydroxy-3-methyl-5-(pyridin-3-yl)pyrrolidin-2-one (*trans*-6p)



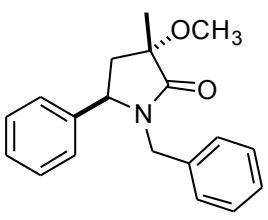
General procedure (**b**) was used. *N*-(4-methoxybenzyl)-3-methyl-1-3-pyridylbut-3-en-1-amine **5p** (350 mg, 1.24 mmol), iodine (940 mg, 3.70 mmol), sodium bicarbonate (520 mg, 6.19 mmol), dichloromethane/petroleum ether 50%, yellow oil, 140 mg, 48% yield. IR 2934, 1689 (s), 1611, 1512, 1431, 1245. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.55 (3H, s, CH_3), 1.88 (1H, dd, J_{ab} 13.7, J_{ac} 6.9, CHC*H*H), 2.58 (1H, dd, J_{ab} 13.7, J_{bc} 7.0, CHCH*H*), 3.51 (ABq, 1H, J_{AB} 14.5, ArC*H*H), 3.78 (1H, s, OCH_3), 4.53 (1H, t, *J* 7.3, C*H*CHH), 5.04 (ABq, 1H, J_{AB} 14.5, ArCH*H*), 6.83 (4H, dt, *J* 1.9, 1.9, ArC*H*), 7.39 (1H, dt, *J* 1.9, 1.9, PyrC*H*), 8.38 (1H, d, *J* 1.9, PyrC*H*), 8.61 (1H, dd, *J* 1.6, 4.7, PyrC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.9 (CH_3), 43.9 (CH_2), 44.1 (CH_2), 55.2 (CH), 55.9 (CH), 74.0 (COH), 114.1 (ArCH), 123.9 (ArCH), 127.1 (ArC), 129.6 (ArCH), 134.4 (ArCH), 135.4 (ArC), 149.0 (ArCH), 149.7 (ArCH), 159.1 (ArC), 176.8 (CO). HRMS $[M+H]^+$ calculated for the formula $C_{18}H_{21}N_2O_3^+$ 313.1541; found 313.1552.

3-Hydroxy-1,3-dimethyl-5-(pyridin-3-yl)pyrrolidin-2-one (6q)



General procedure (**b**) was used. *N*,3-dimethyl-1-(pyridin-3-yl)but-3-en-1-amine **5q** (400 mg, 2.27 mmol), iodine (1.73 g, 1.80 mmol), sodium bicarbonate (950 mg, 11.30 mmol), methanol/ dichloromethane 5%, Rf = 0.24, pale yellow crystal (mpt 168-169 ˚C), 150 mg, 36% yield. IR 3356, 2927, 1679(s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.51 (3H, s, CH_3), 1.89 (1H, dd, J_{ab} 13.8, J_{ac} 6.7, CHC*H*H), 2.62-2.77 (4H, overlapping m, CHCH*H* & NCH_3), 4.69 (1H, app t, *J* 7.2, C*H*CHH), 7.36 (1H, dd, *J* 4.8, 7.9, PyrC*H*), 7.51 (1H, dt, *J* 7.9, 1.9, PyrC*H*), 8.52 (1H, s, PyrC*H*), 8.62 (1H, d, *J* 3.8, PyrC*H*).^{13}C NMR (δ; 100 MHz, $CDCl_3$); 25.2 (CH_3), 28.7 (CH_3), 43.9 (CH_2), 59.1 (CH), 73.9 (COH), 124.0 (ArCH), 133.8 (ArCH), 135.7 (ArC), 148.6 (ArCH), 149.8 (ArCH), 176.7 (CO). HRMS $[M+H]^+$ calculated for the formula $C_{11}H_{15}N_2O_2^+$ 207.1134 found 207.1136.

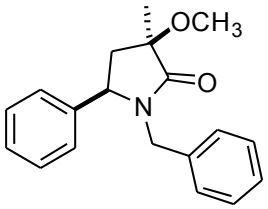
cis-N-Benzyl-3-methoxy-3-methyl-5-phenylpyrrolidin-2-one (*cis*-7a)



N-Benzyl-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one **6a** (0.07 g, 0.25 mmol) in dry DMF (10 mL), was added sodium hydride (0.01 g, 0.5 mmol), the reaction mixture was stirred for 30 minutes, then iodomethane (0.7 mL, 0.5 mmol) was added and stirred for another 18 hours at room temperature (TLC was monitored), quenched by addition of (10 mL) water and extracted with ethyl acetate, the combined organic layer was washed with brine and dried with magnesium sulfate to give the alkylated product in quantitative yield, the single

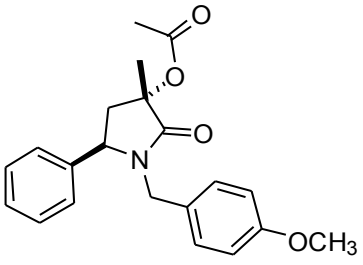
diastereoisomer was separated by column chromatography using silica gel and ethyl acetate/petroleum ether 30%, Rf = 0.21, colorless solid (mpt 82-83 ˚C), 34 mg, 50% yield. IR 3360, 2928, 1683 (s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.51 (3H, s, CH_3), 1.85 (1H, dd, J_{ab} 13.9, J_{ac} 7.6, CHC*H*H), 2.55 (1H, dd, J_{ab} 13.9, J_{bc} 7.1, CHCH*H*), 3.36 (3H, s, CH_3), 3.50 (ABq, 1H, J_{AB} 14.6, ArC*H*H), 4.46 (1H, t, *J* 7.3, C*H*CHH), 5.13 (ABq, 1H, J_{AB} 14.6, ArCH*H*), 6.94- 7.47 (10H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 21.5 (CH_3), 40.2 (CH_2), 44.5 (CH_2), 51.7 (CH_3), 57.2 (CH), 79.3 (*C*OCH_3), , 127.3 (ArCH), 127.5 (ArCH), 128.1 (ArCH), 128.3 (ArCH), 128.6 (ArCH), 129.0 (ArCH), 136.0 (ArC), 139.7 (ArC), 174.7 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{19}H_{21}NO_2Na^+$ 318.1470; found 318.1478.

trans-*N*-Benzyl-3-methoxy-3-methyl-5-phenylpyrrolidin-2-one (*trans*-7b)



N-Benzyl-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one **6a** (0.07 g, 0.25 mmol) in dry DMF (10 mL), was added sodium hydride (0.01 g, 0.5 mmol), the reaction mixture was stirred for 30 minutes, then iodomethane (0.7 mL, 0.5 mmol) was added and stirred for another 18 hours at room temperature (TLC was monitored), quenched by addition of (10 mL) water and extracted with ethyl acetate, the combined organic layer was washed with brine and dried with magnesium sulfate to give the alkylated product, the single diastereoisomer was separated by column chromatography using silica gel and ethyl acetate/petroleum ether 30%, Rf = 0.25, colorless solid (mpt 75-76 ˚C), 33 mg, 49% yield. IR 3360, 2928, 1683. ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.42 (3H, s, CH_3), 2.17 (1H, dd, J_{ab} 13.3, J_{ac} 7.9, CHC*H*H), 2.26 (1H, dd, J_{ab} 13.3, J_{bc} 7.3, CHCH*H*), 3.43 (3H, s, OCH_3), 3.47 (ABq, 1H, J_{AB} 14.5, ArC*H*H), 4.12 (1H, app.t, *J* 7.6, C*H*CHH), 5.12 (ABq, 1H, J_{AB} 14.5, ArCH*H*), 6.92-7.46 (10H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 20.0 (CH_3), 40.2 (CH_2), 44.5 (CH_2), 51.7 (CH_3), 58.3 (CH), 78.5 (*C*OCH_3), 126.8 (ArCH), 127.1 (ArCH), 127.7 (ArCH), 128.4 (ArCH), 128.4 (ArCH), 128.6 (ArCH), 136.0 (ArC), 139.8 (ArC), 173.7 (CO). HRMS $[M+Na]^+$ calculated for the formula $C_{19}H_{21}NO_2Na^+$ 318.1470; found 318.1478.

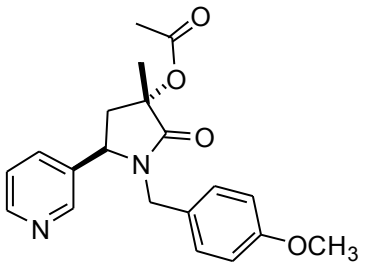
cis-*N*-(4-Methoxybenzyl)-3-methyl-2-oxo-5-phenylpyrrolidin-3-yl acetate (*cis*-8a)



N-(4-Methoxybenzyl)-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (35 mg, 0.11 mmol), triethylamine (22 mg, 0.22 mmol), DMAP (5.80 mg, 0.011 mmol) and acetic anhydride (22 mg, 0.22 mmol) were added in (10 mL) dry dichloromethane under nitrogen and stirred at room temperature for 24 hours with TLC monitoring. The reaction was quenched with (30 mL) brine extracted with ethyl acetate (3x 25 mL) and the combined organic layer was washed with water and dried with $MgSO_4$ and the product was obtained as colourless crystals after recrystalised from dichloromethane/petroleum ether 10%, colourless crystals (mpt 121-122 ˚C), 36 mg, 92% yield. IR 3286, 3030, 2933, 1671(s), 1669(s). ^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.56 (3H, s, CH_3), 1.89 (1H, dd, J_{ab} 14.4, J_{ac} 4.8, CHC*H*H), 2.03 (3H, s, COCH_3), 2.73 (1H, dd, J_{ab} 14.4, J_{bc} 9.6, CHCH*H*), 3.44 (ABq, 1H, J_{AB} 14.5, ArC*H*H), 3.71 (3H, s, OCH_3), 4.44 (1H, dd, J_{ac} 4.8, J_{bc} 9.6, C*H*CHH), 5.02 (ABq, 1H, J_{AB} 14.5, ArCH*H*), 6.85 (4H, dt, *J*

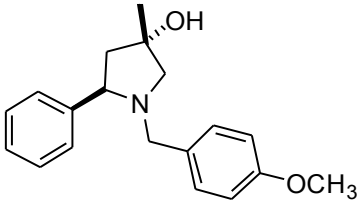
2.9, 2.6, ArC*H*), 7.00-7.33 (5H, m, ArC*H*). ^{13}C NMR (δ; 100 MHz, $CDCl_3$); 20.2 (CH_3), 24.7 (CH_3), 40.0 (CH_2), 43.4 (CH_2), 54.1 (CH), 56.7 (*C*OCH_3), 79.0 ($COCOCH_3$), 112.6 (ArCH), 125.7 (ArCH), 126.0 ($ArCOCH_3$), 127.0 (ArCH), 128.0 (ArCH), 129.2 (ArCH), 139.5 (ArC), 158.0 (ArC), 169.0 (CO), 171.9 (CO). HRMS $[M+H]^+$ calculated for the formula $C_{21}H_{23}NO_4^+$ 353.4116; found 354.4115.

cis-N-(4-Methoxybenzyl)-3-methyl-2-oxo-5-(pyridin-3-yl)pyrrolidin-3-ylacetate (*cis*-8b)



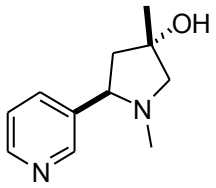
N-(4-Methoxybenzyl)-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (135 mg, 0.43 mmol), triethylamine (80 mg, 0.86 mmol), DMAP (23 mg, 0.043 mmol) and acetic anhydride (88 mg, 0.86 mmol) were added in (10 mL) dry dichloromethane under nitrogen and stirred at room temperature for 24h with TLC monitoring. The reaction was quenched with (5 mL) brine extracted with ethyl acetate and the combined organic layer was washed with water and *in vacuo* evaporated solvent gave yellow solid (mpt 193-194 ˚C), 123 mg, 56% yield. IR 3457, 2931, 1736 (s), 1701 (s).^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.56 (3H, s, CH_3), 1.89 (1H, dd, J_{ab} 14.4, J_{ac} 4.8, CHC*H*H), 2.03 (3H, s, COCH_3), 2.73 (1H, dd, J_{ab} 14.4, J_{bc} 4.8, CHCH*H*), 3.44 (ABq, 1H, J_{AB} 14.5, ArC*H*H), 3.71 (3H, s, COCH_3), 4.44 (1H, dd, J_{ac} 4.8, J_{bc} 4.8, C*H*CHH), 5.02 (ABq, 1H, J_{AB} 14.5, ArCH*H*), 6.85 (4H, dd, J_{ab} 8.6, J_{ac} 69.4, ArC*H*), 7.00-7.33 (6H, m, ArC*H*).^{13}C NMR (δ; 100 MHz, $CDCl_3$); 20.2 (CH_3), 24.7 (CH_3), 40.0 (CH_2), 43.4 (CH_2), 54.1 (CH), 56.7 (*C*OCH_3), 79.0 (*C*$OCOCH_3$), 112.6 (2ArCH), 125.7 (ArCH), 126.0 ($ArCOCH_3$), 127.0 (ArCH), 128.0 (ArCH), 129.2 (ArCH), 139.5 (ArC), 158.0 (ArC), 169.0 (CO), 171.9 (CO). HRMS $[M+H]^+$ calculated for the formula $C_{20}H_{23}N_2O_4^+$ 355.1661; found 355.1658.

cis-N-(4-Methoxybenzyl)-3-methyl-5-phenylpyrrolidin-3-ol (*cis*-9a)



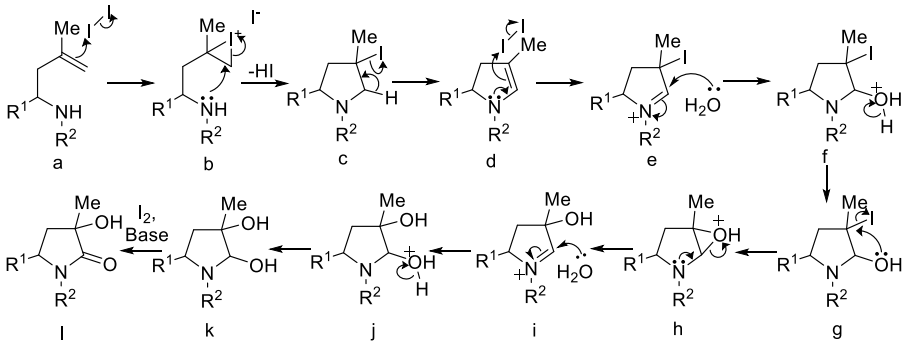
cis-N-(4-Methoxybenzyl)-3-hydroxy-3-methyl-5-phenylpyrrolidin-2-one (30 mg, 0.096 mmol) in dry THF (5 mL) was added drop wise to the refluxed solution of $LiAlH_4$ (18 mg, 0.48 mmol) in dry THF (5 mL) for three hours, the reaction was quenched with (20 mL) water and extracted with dichloromethane (3 x 20 mL) the combined organic layer was washed with brine and dried with magnesium sulfate and *in vacuo* evaporated solvent and column chromatography ethyl acetate/hexane 30% gave colorless oil 16 mg, 56% yield. IR 3393, 2925, 1510, 1245.^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.44 (3H, s, CH_3), 1.82 (1H, dd, J_{ab} 12.0, J_{ac} 9.0, CHC*H*H), 2.20 (1H, dd, J_{ab}12.0, J_{bc}6.0, CHCH*H*), 2.43 (1H, d, *J* 10.0, C*H*H), 3.11 (1H, d, *J* 10.8 CH*H*), 3.72-3.84 (6H, m, OCH_3, ArC*H*$'$*H*$''$ & C*H*CHH), 6.83 (2H, d, *J* 8.6, ArC*H*), 7.18 (2H, d, *J* 8.5, ArC*H*), 7.23-7.53 (5H, m, ArC*H*).^{13}C NMR (δ; 100 MHz, $CDCl_3$); 29.3 (CH_3), 51.8 (CH_2), 55.2 (CH), 56.9 ($2CH_2$), 67.4 (CH_2), 113.5 ($ArOCH_3$), 127.2 (ArCH), 127.4 (ArCH), 128.4 (ArCH), 129.6 (ArCH), 131.4 (ArC), 158.4 (ArC). HRMS $[M+H]^+$ calculated for the formula $C_{19}H_{24}NO_2^+$ 298.1802; found 298.1807.

cis-N-Methyl-3-methyl-5-(pyridin-3-yl)pyrrolidin-3-ol (*cis*-9b)



cis-3-Hydroxy-1,3-dimethyl-5-phenylpyrrolidin-2-one **6q** (70 mg, 0.34 mmol) in dry THF (5 mL) was added to refluxed solution of $LiAlH_4$ (128 mg, 3.4 mmol) in dry THF (5 mL) for three hours with TLC monitoring (methanol/DCM 10%), the reaction was quenched with (20 mL) water and extracted with dichloromethane (3 x 20 mL) the combined organic layer was washed with brine and dried with magnesium sulfate and *in vacuo* evaporated solvent and column chromatography (methanol/ dichloromethane/ petroleum ether 1:7:2) gave yellow oil 58 mg, 89% yield. IR 3361, 2966, 2777.^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.39 (3H, s, CH_3), 1.90 (1H, dd, J_{ab} 7.7, J_{ac} 1.6, CHC*H*H), 1.92 (1H, dd, J_{ab} 7.7, J_{bc} 1.6, CHCH*H*), 2.19 (3H, s, NCH_3), 2.32-2.43 (2H, m, NC*H*H), 3.07 (1H, dd, *J* 1.5, 9.7, NCH*H*), 3.22 (1H, dd, J_{ac} 1.6, J_{bc} 1.6, C*H*CHH), 7.29 (2H, dd, *J* 6.7, 3.8, PyrC*H*), 7.74 (1H, dt, *J* 7.8, 1.8, PyrC*H*), 8.53 (1H, s, PyrC*H*).^{13}C NMR (δ; 100 MHz, $CDCl_3$); 24.9 (CH_3), 39.9 (CH_3), 51.2 (CH_2), 53.8 (COH), 68.2 (CH), 70.7 (CH_2), 123.7 (PyrCH), 134.9 (PyrCH), 138.0 (PyrC), 148.8 (PyrCH), 149.43 (PyrCH). HRMS $[M+H]^+$ calculated for the formula $C_{11}H_{17}N_2O^+$ 193.1344; found 193.1341.

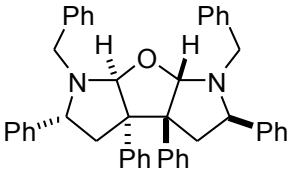
Suggested Mechanism for lactam formation



Cesium carbonate mediated aerobic oxidation may play a role

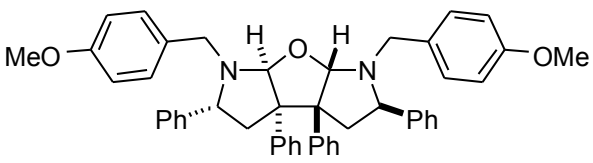
Fused tricyclic compounds:

N,N'-Dibenzyl-2,3a,3b,5-tetraphenyldecahydrofuro[2,3-b:5,4-b']dipyrrole (11a)



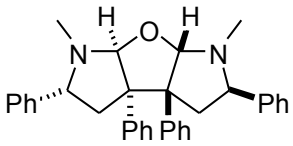
N-Benzyl-1,3-diphenylbut-3-en-1-amine **10a** (500 mg, 1.5mmol), iodine (1. 21 g, 4.78 mmol), sodium bicarbonate (4.67 g, 7.97 mmol), column chromatography ethyl acetate/ hexane 10%, Rf = 0.7, crystalisation with ethyl acetate/ petroleum ether 10%, colorless crystal (mpt 190-192 ˚C), 266 mg, 48% yield. IR 3060, 2850, 1601, 1493, 1454, 1365, 1142.^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.85 (2H, dd, J_{ab} 13.8, J_{ac} 7.6, CHC*H*H), 2.67 (2H, dd, J_{ab} 13.8, J_{bc} 8.4, CHCH*H*), 3.60 (ABq, 2H, J_{AB} 12.9, ArC*H*H), 3.87 (ABq, 2H, J_{AB} 12.9, ArC*HH*), 3.99 (2H, t, *J* 8.0, C*H*CHH), 5.61 (2H, s, OC*H*), 7.06-7.48 (30H, overlapping m, ArC*H*).^{13}C NMR (δ; 100 MHz, $CDCl_3$); 47.4 (CH_2), 50.8 (CH_2), 61.8 (C), 65.9 (CH), 98.0 (CH), 126.3 (ArCH), 126.9 (ArCH), 127.0 (ArCH), 127.5 (ArCH), 128.2 (ArCH), 128.3 (ArCH), 128.9 (ArCH), 129.1 (ArCH), 139.4 (ArC), 143.3 (ArC), 144.2 (ArC). HRMS $[M+H]^+$ calculated for formula $C_{46}H_{43}N_2O^+$ 639.3360; found 639.3375.

N,N'-Bis(4-methoxybenzyl)-2,3a,3b,5-tetraphenyldecahydrofuro[2,3-b:5,4-b']dipyrrole (11b)



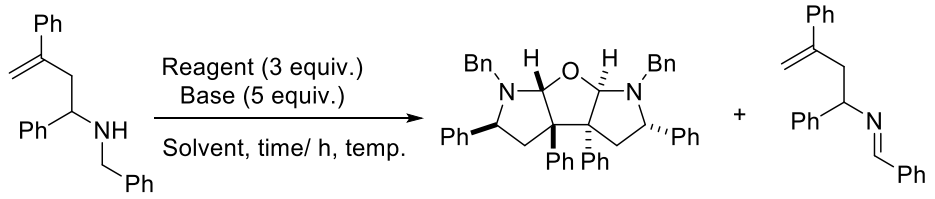
N-(4-Methoxybenzyl)-1,3-diphenylbut-3-en-1-amine **10b** (310 mg, 0.90 mmol), iodine (687 mg, 2.70 mmol), sodium bicarbonate (379 mg, 4.50 mmol), column chromatography ethyl acetate/ hexane 10%, Rf = 0.7, crystalisation with ethyl acetate/ petroleum ether 10%, colorless crystal (mpt 215-216 ˚C), 120 mg, 20% yield. IR 2858, 1614, 1512.^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.84 (2H, dd, J_{ab} 13.8, J_{ac} 7.6, CHC*H*H), 2.67 (2H, dd, J_{ab} 13.8, J_{bc} 8.4, CHCH*H*), 3.53 (ABq, 2H, J_{AB} 12.6, ArC*H*H), 3.66 (ABq, 2H, J_{AB} 12.6, ArCH*H*), 3.87 (6H, s, OCH_3), 3.97 (2H, t, *J* 8.0, C*H*CHH), 5.58 (2H, s, OC*H*), 6.93 (4H, d, *J* 8.6, ArC*H*), 7.06-7.28 (20H, overlapping m, ArC*H*), 7.37 (4H, d, *J* 8.5, ArC*H*).^{13}C NMR (δ; 100 MHz, $CDCl_3$); 47.5 (CH_2), 50.2 (CH_2), 55.3 (CH_3), 61.7 (C), 65.8 (CH), 98.0 (CH), 113.6 (ArCH), 126.2 (ArCH), 126.9 (ArCH), 127.5 (ArCH), 128.3 (ArCH), 128.9 (ArCH), 130.2 (ArCH), 131.4 (ArC), 143.4 (ArC), 144.2 (ArC), 158.6 (ArC). HRMS $[M+H]^+$ calculated for the formula $C_{48}H_{47}N_2O_3^+$ 699.3587; found 699.3591.

1,6-Dimethyl-2,3a,3b,5-tetraphenyldecahydrofuro[2,3-b:5,4-b']dipyrrole (11c)



N-Methyl-1,3-diphenylbut-3-en-1-amine **10c** (265 mg, 1.12 mmol), iodine (850 mg, 3.34 mmol), sodium bicarbonate (468 mg, 5.58 mmol), ethyl acetate/ hexane 10%, Rf = 0.7, crystalisation with ethyl acetate/ petroleum ether 10%. Colorless crystal (mpt 195-196 ˚C), 120 mg, 22% yield. IR 3050, 2800, 1602, 1491.^{1}H NMR (δ; 300 MHz, $CDCl_3$); 1.93 (2H, dd, J_{ab} 13.8, J_{ac} 7.6, CHC*H*H), 2.44 (6H, s, NCH_3), 2.82 (2H, dd, J_{ab} 13.8, J_{bc} 8.5, CHCH*H*), 3.97 (2H, t, *J* 8.0, C*H*CHH), 5.82 (2H, s, OC*H*), 7.04-7.36 (20H, m, ArC*H*).^{13}C NMR (δ; 100 MHz, $CDCl_3$); 34.6 (CH_3), 47.7 (CH_2), 62.0 (PhC), 66.8 (CH), 103.1 (CH), 126.3 (ArCH), 127.0 (ArCH), 127.5 (ArCH), 127.6 (ArCH), 128.2 (ArCH), 129.0 (ArCH), 143.0 (ArC), 144.0 (ArC). HRMS $[M+H]^+$ calculated for the formula $C_{34}H_{35}N_2O^+$ 487.2728; found 487.2749.

Table 1: Condition screening for synthesis of **11a**



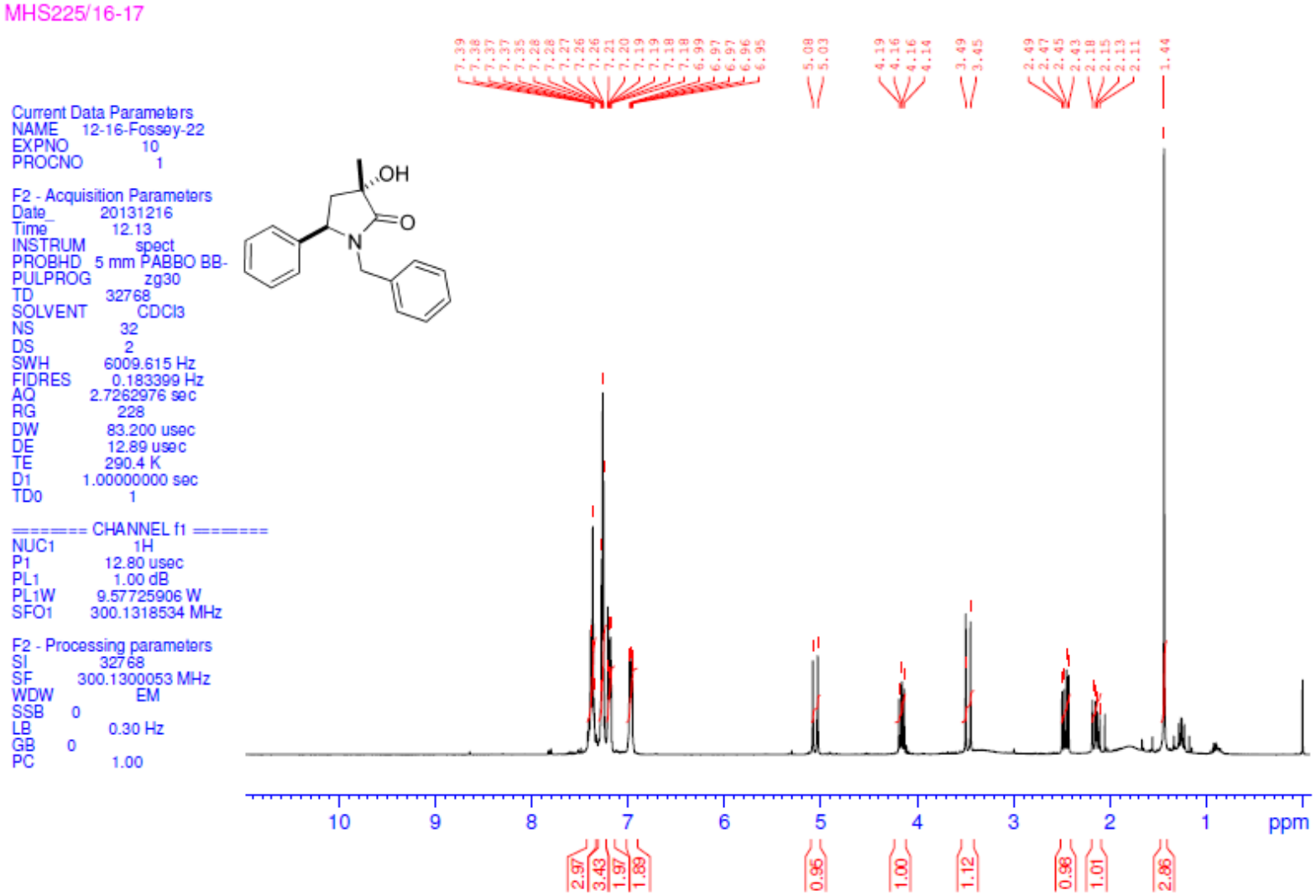
10a **11a** ***imine* I**

Entry	Solvent	Base	T/ °C	Time /h	Reagent	10a %	11a %	I %
1	**EtOAc**	**$NaHCO_3$**	**r.t.**	**24**	**I_2**	**52**	**48**	**-**
2	EtOAC	$NaHCO_3$	Reflux	24	I_2	81	19	-
3	EtOAc	$NaHCO_3$	r.t	48	I_2	83	17	-
4	EtOAc	$NaHCO_3$	r.t	72	I_2	89	11	-
5	EtOAc	LiOH	r.t	24	I_2	80	20	-
6	EtOH	$NaHCO_3$	r.t	24	I_2	>50	(*)	-
7	MeCN	$NaHCO_3$	r.t	48	I_2	69	31	-
8	EtOAc	Cs_2CO_3	r.t	24	I_2	-	-	>99
9	EtOAc	$NaHCO_3$	r.t	24	NIS	73	27	-
10	EtOAc	$NaHCO_3$	r.t	24	Br_2	82	18	-
11	MeCN	LiOH	r.t	24	I_2	67	33	-
12	$CHCl_3$	$NaHCO_3$	r.t	24	I_2	>99	-	-
13	THF	$NaHCO_3$	r.t	24	I_2	81	19	-

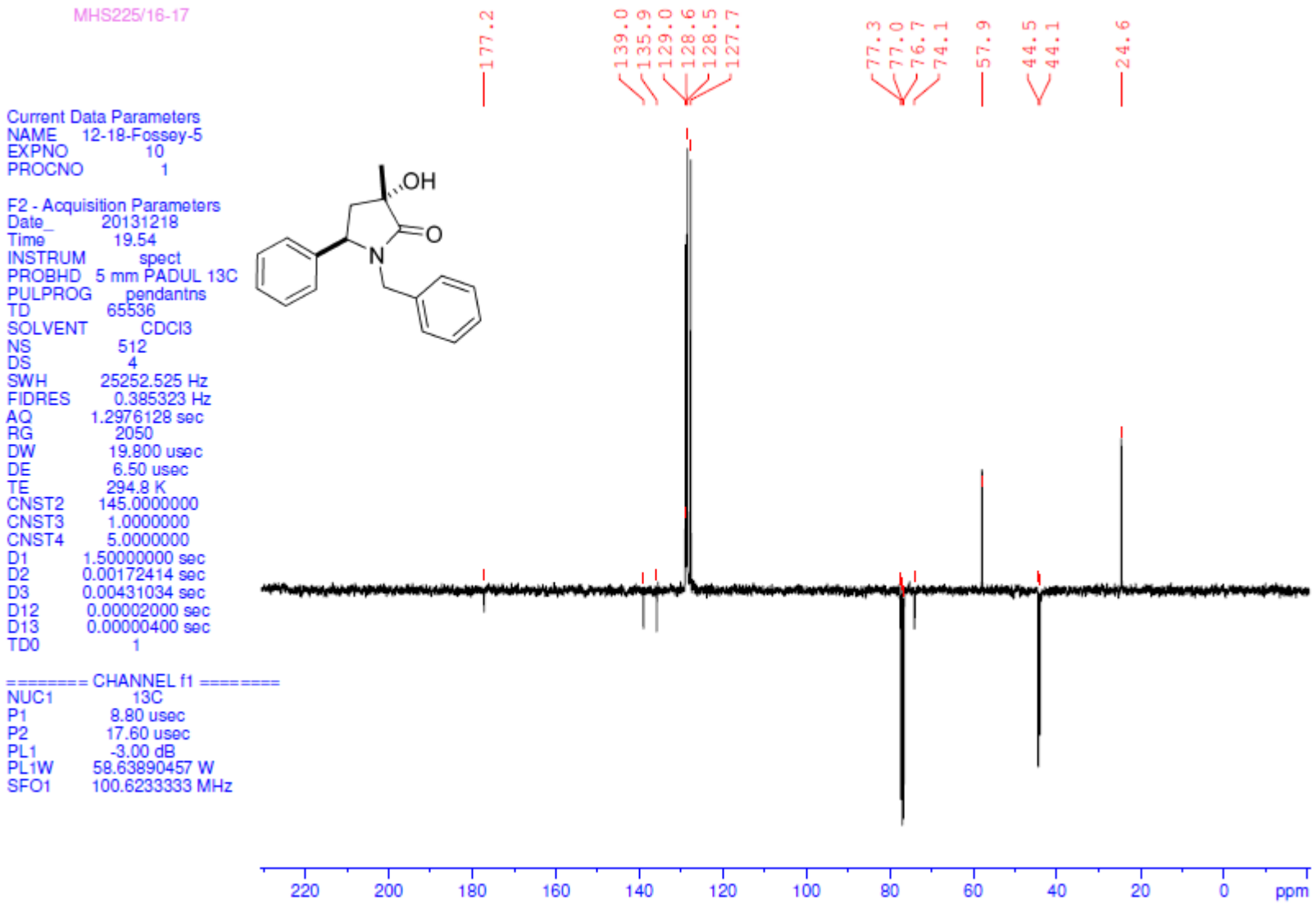
* unknown product

NMR Spectra

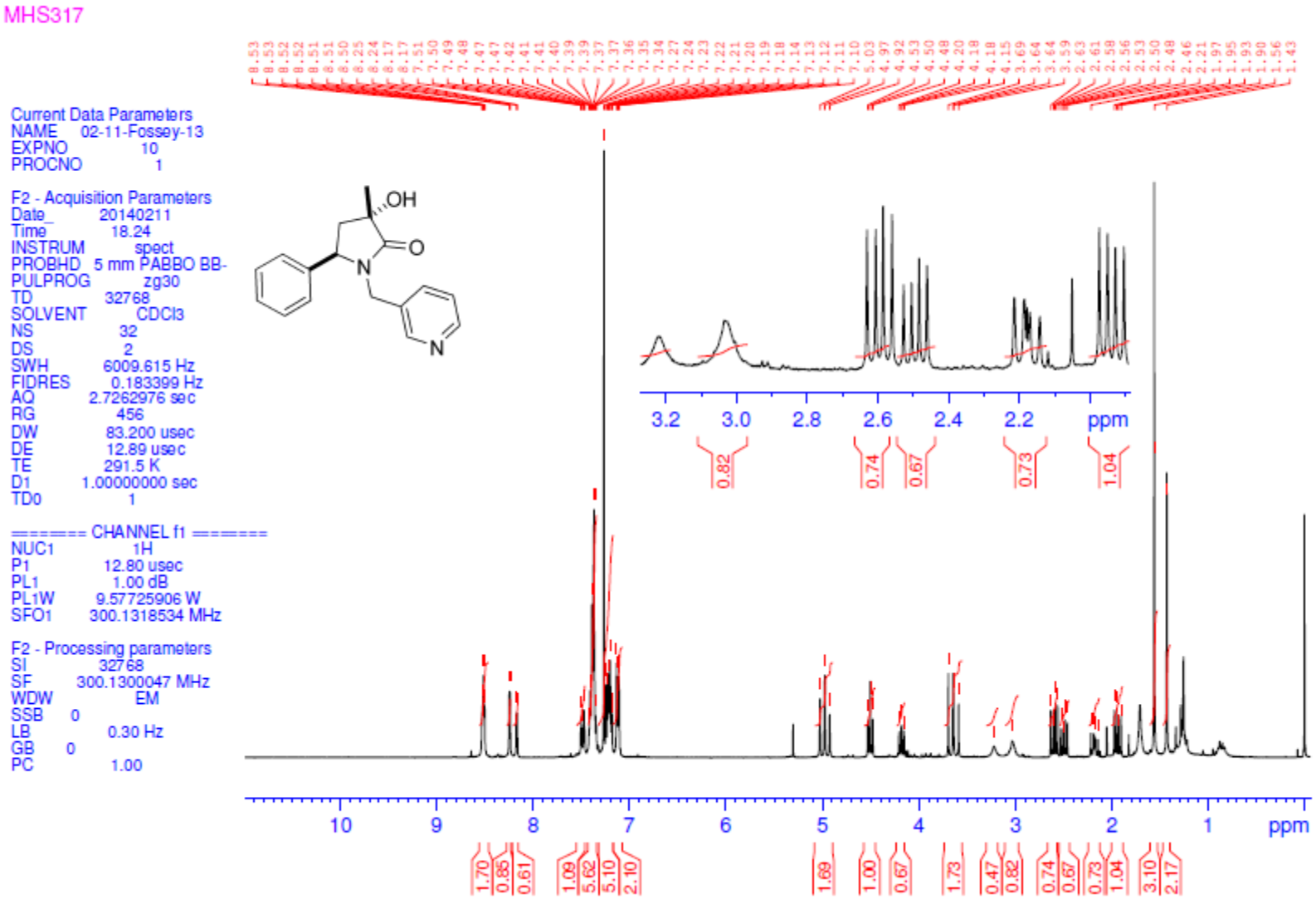
^{1}H NMR Spectra of compound *cis*-6a



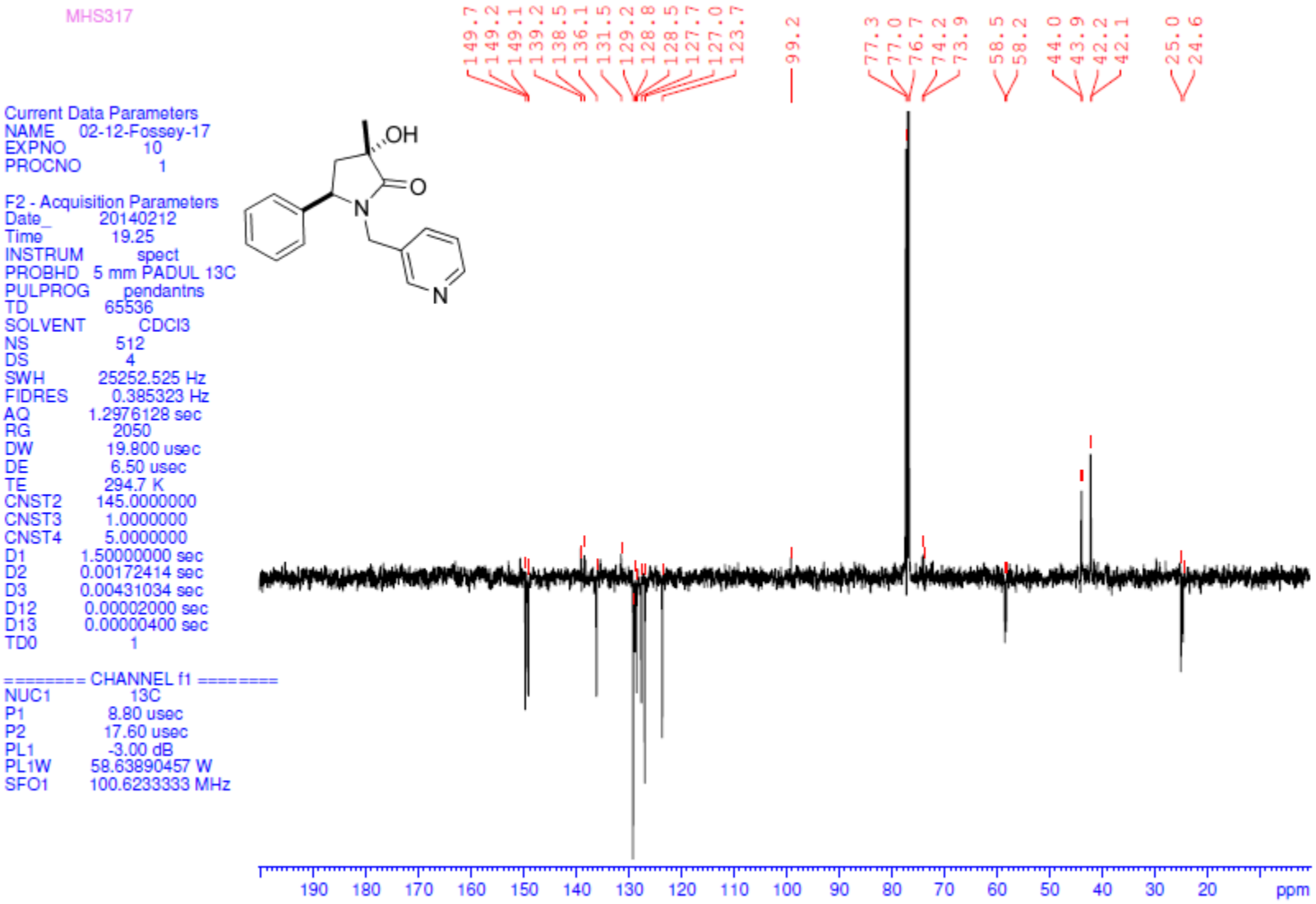
ESI M. A **^{13}C NMR Spectra of compound *cis*-6a**



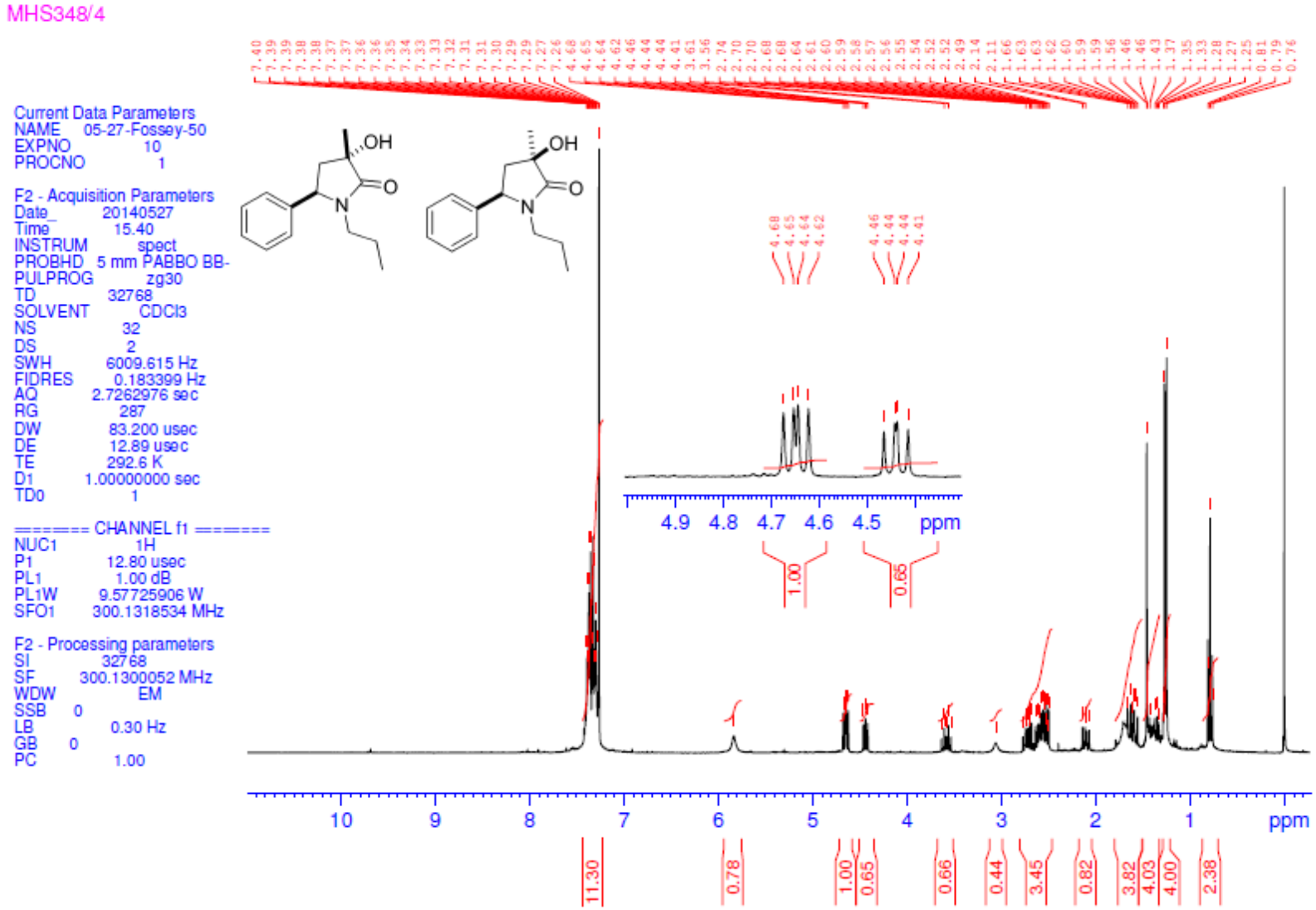
^{1}H NMR Spectra of compounds *cis*-6b and *trans*-6b (mixture of diastereoisomers)



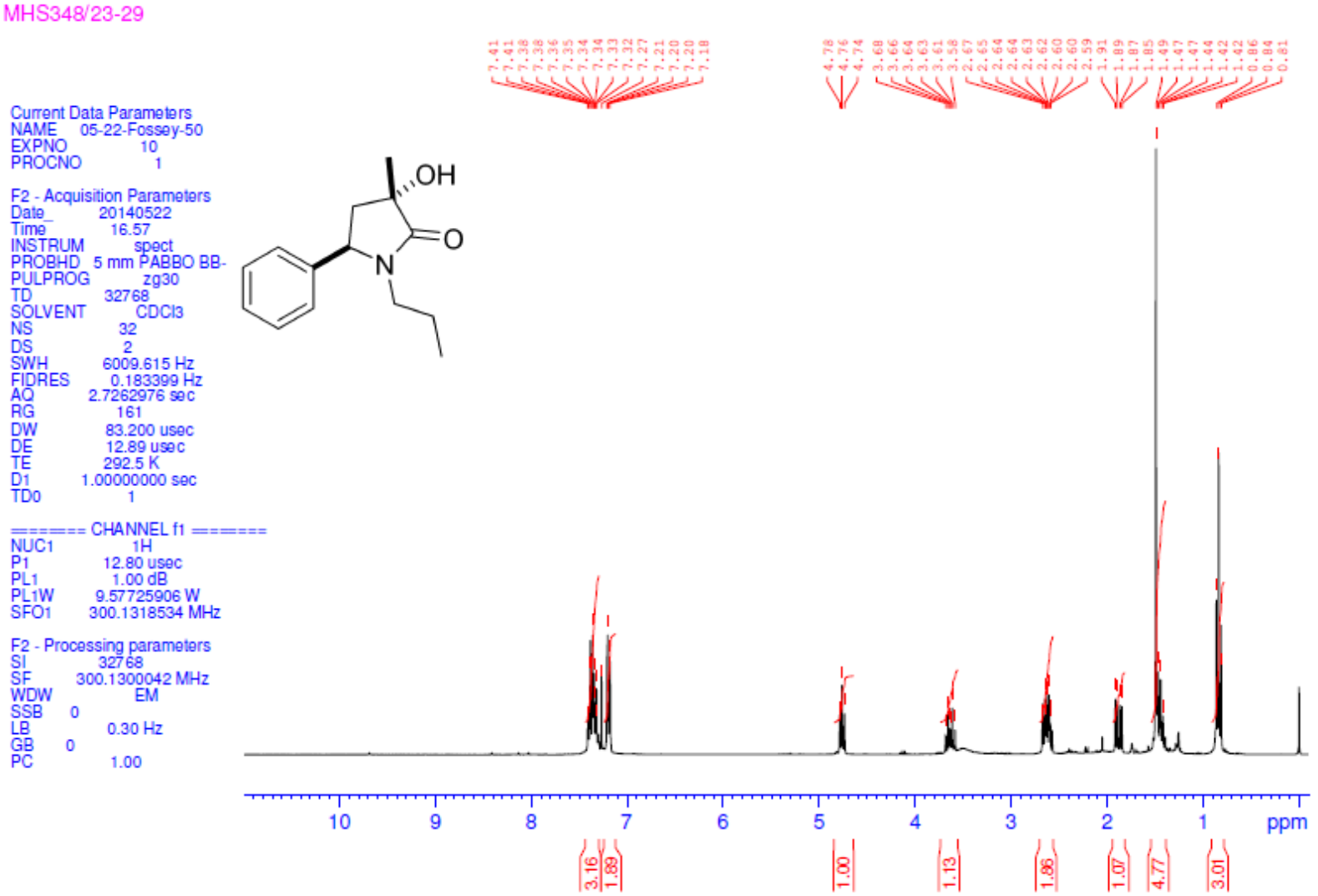
^{13}C NMR Spectra of compounds *cis*-6b and *trans*-6b (mixture of diastereoisomers)



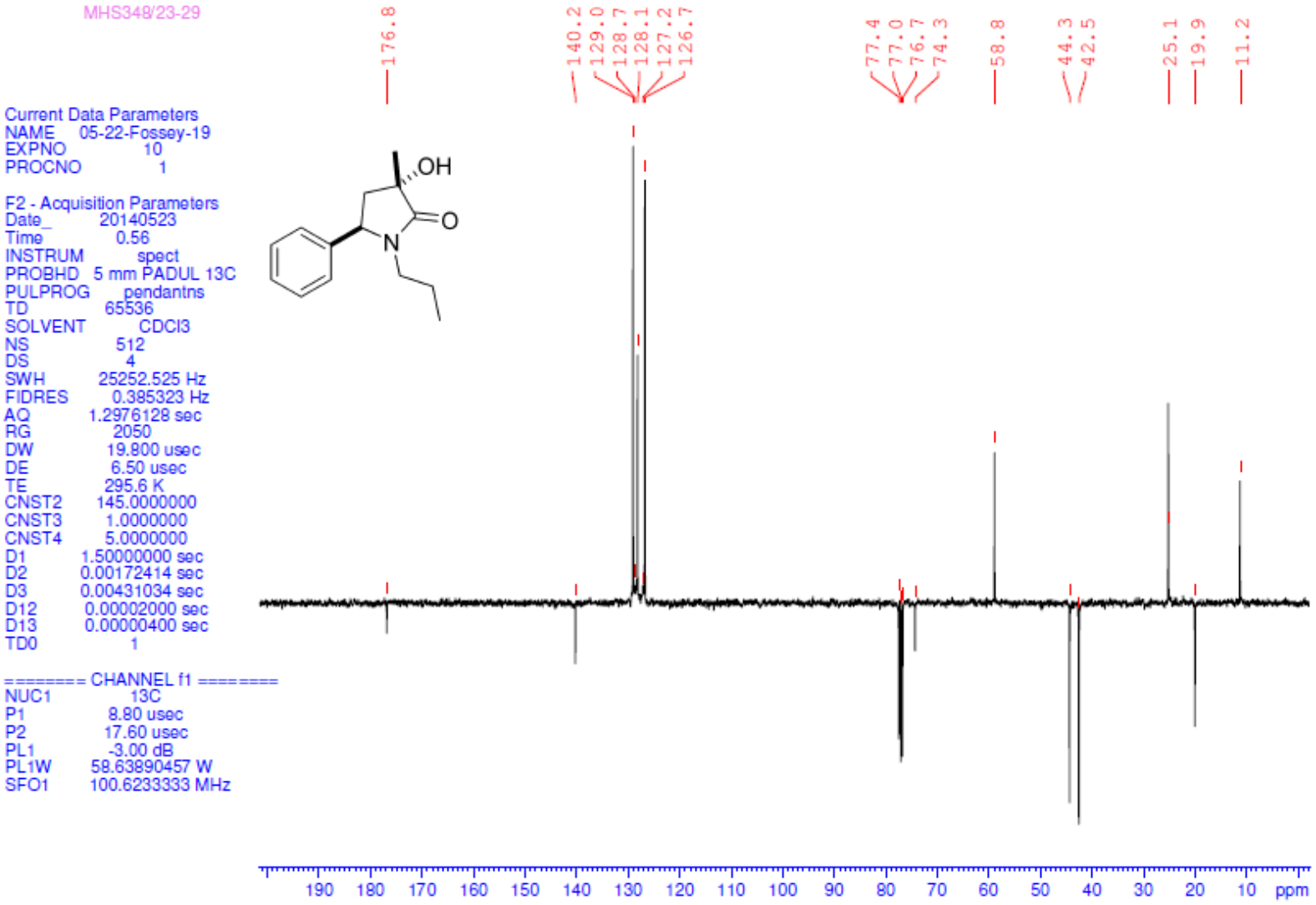
 ^{1}H NMR Spectra of compounds *cis*-6c and *trans*-6c (mixture of diastereoisomers)



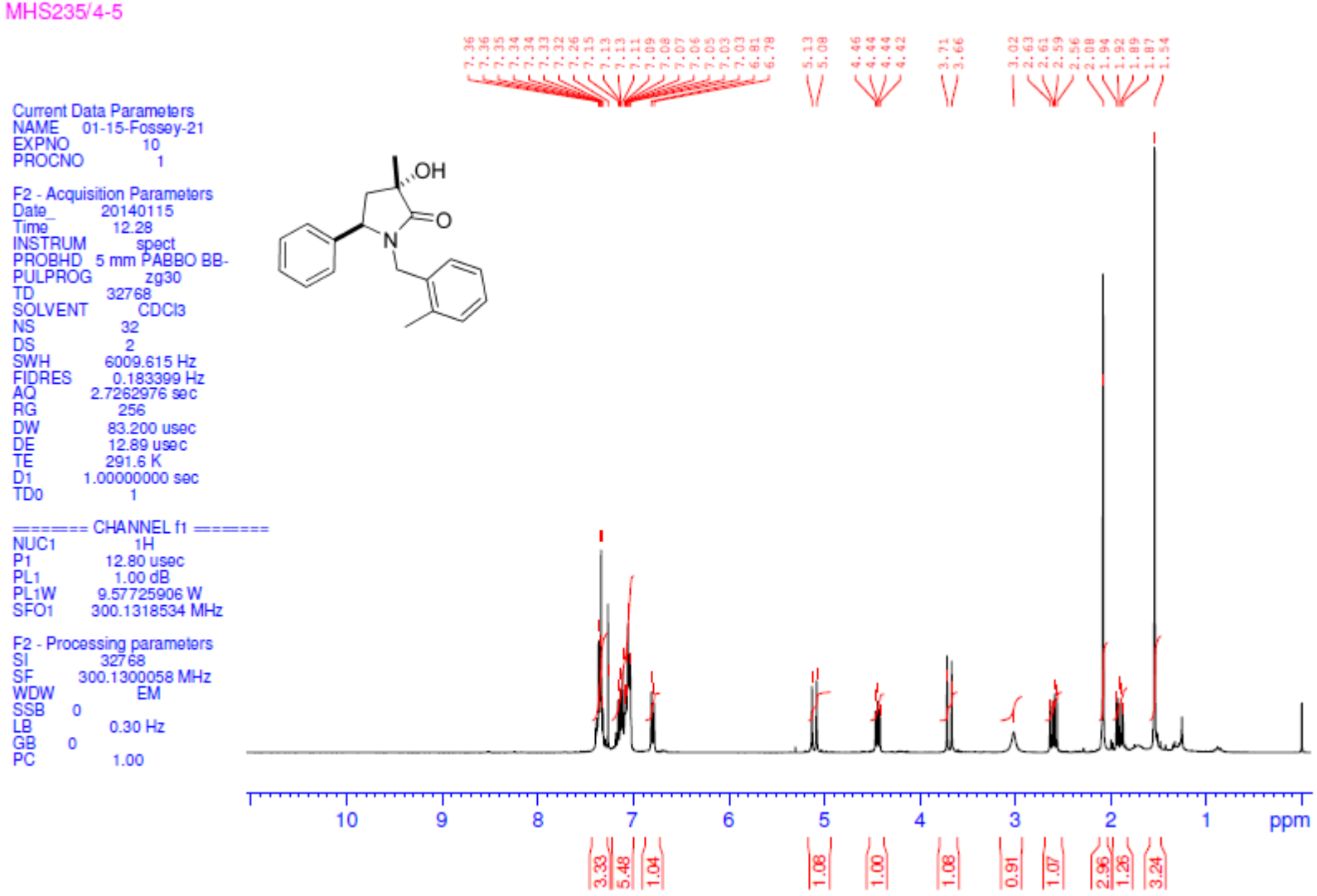
^{1}H NMR Spectra of compound *cis*-6c



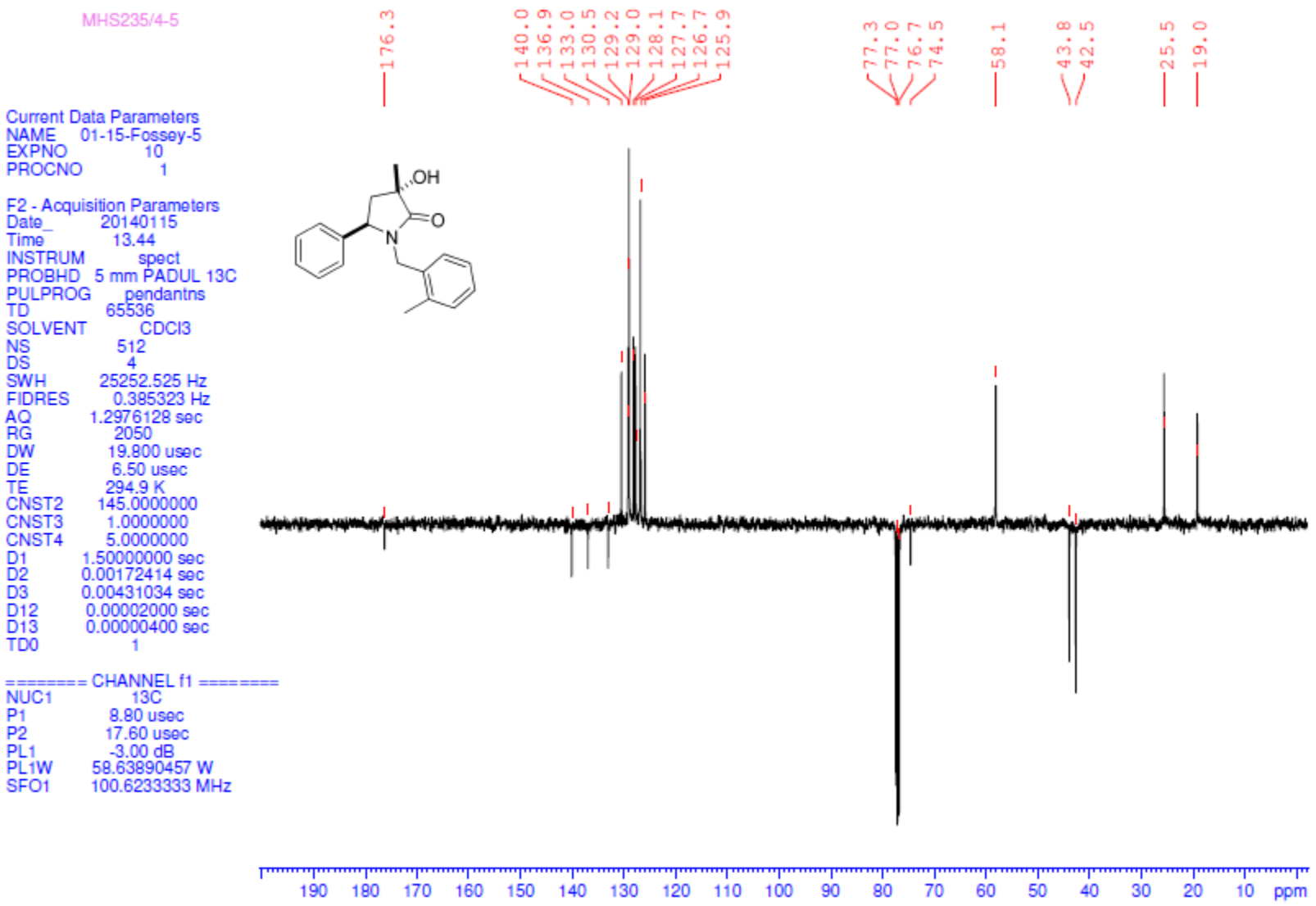
^{13}C NMR Spectra of compound *cis*-6c



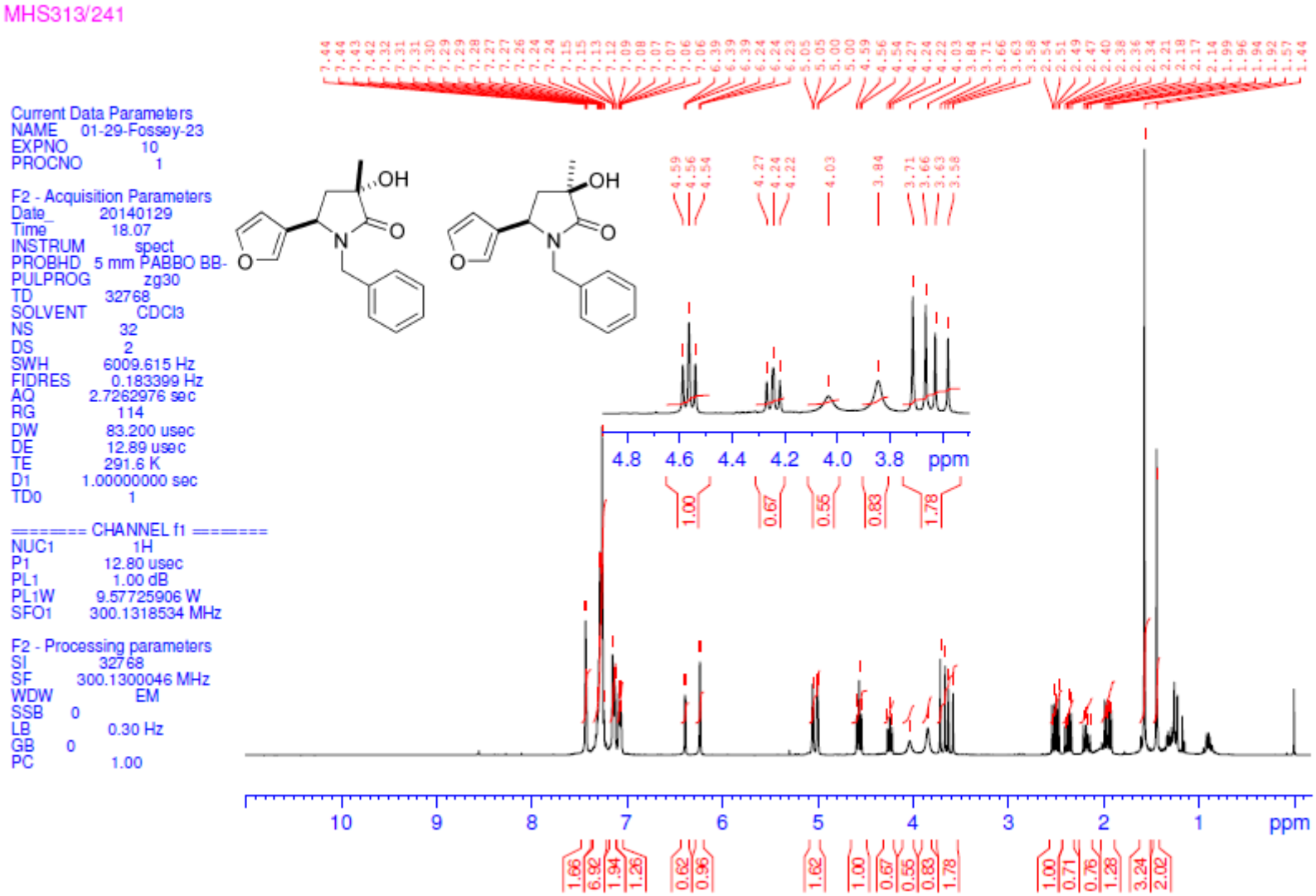
^{1}H NMR Spectra of compound *cis*-6d



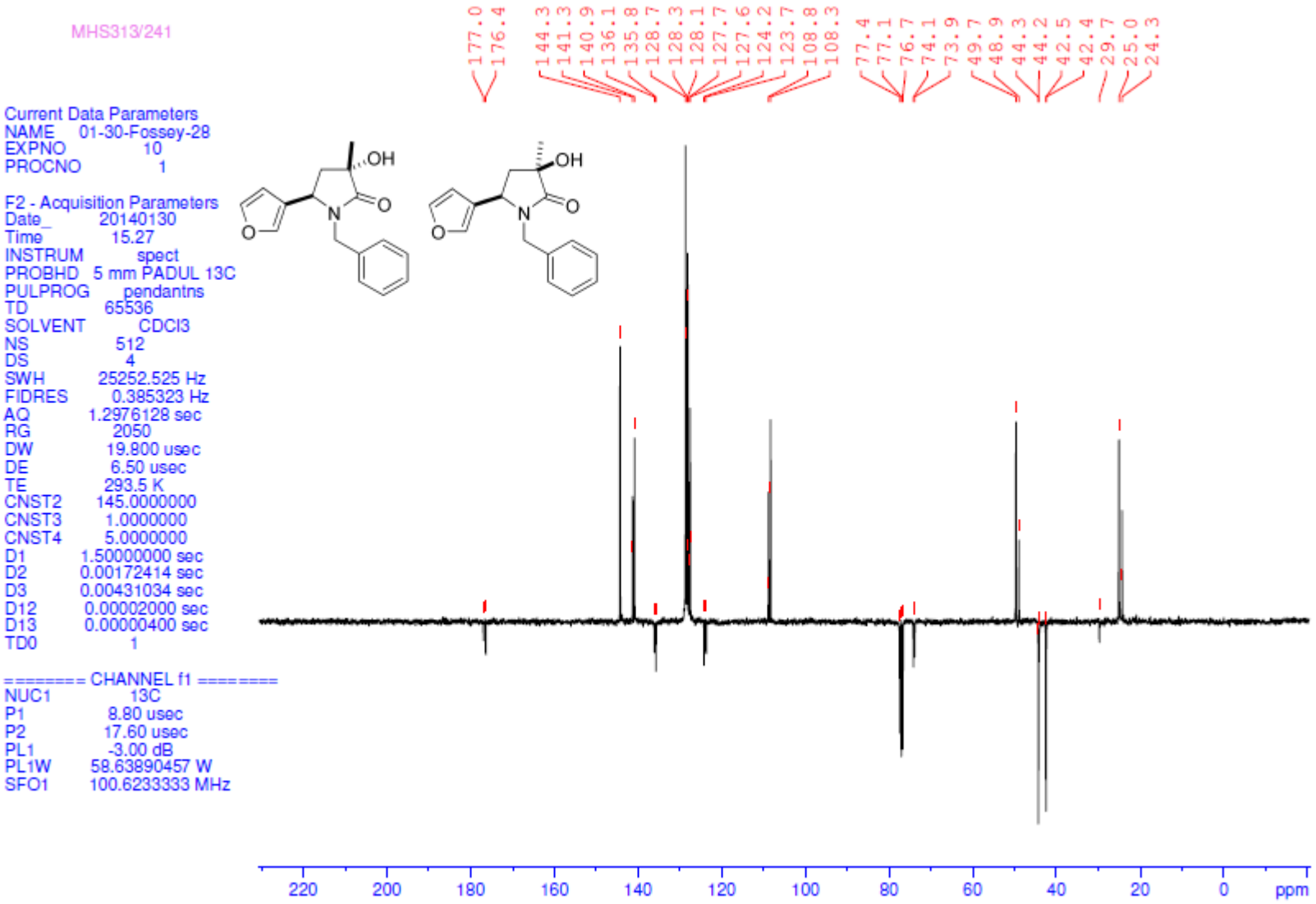
^{13}C NMR Spectra of compound *cis*-6d



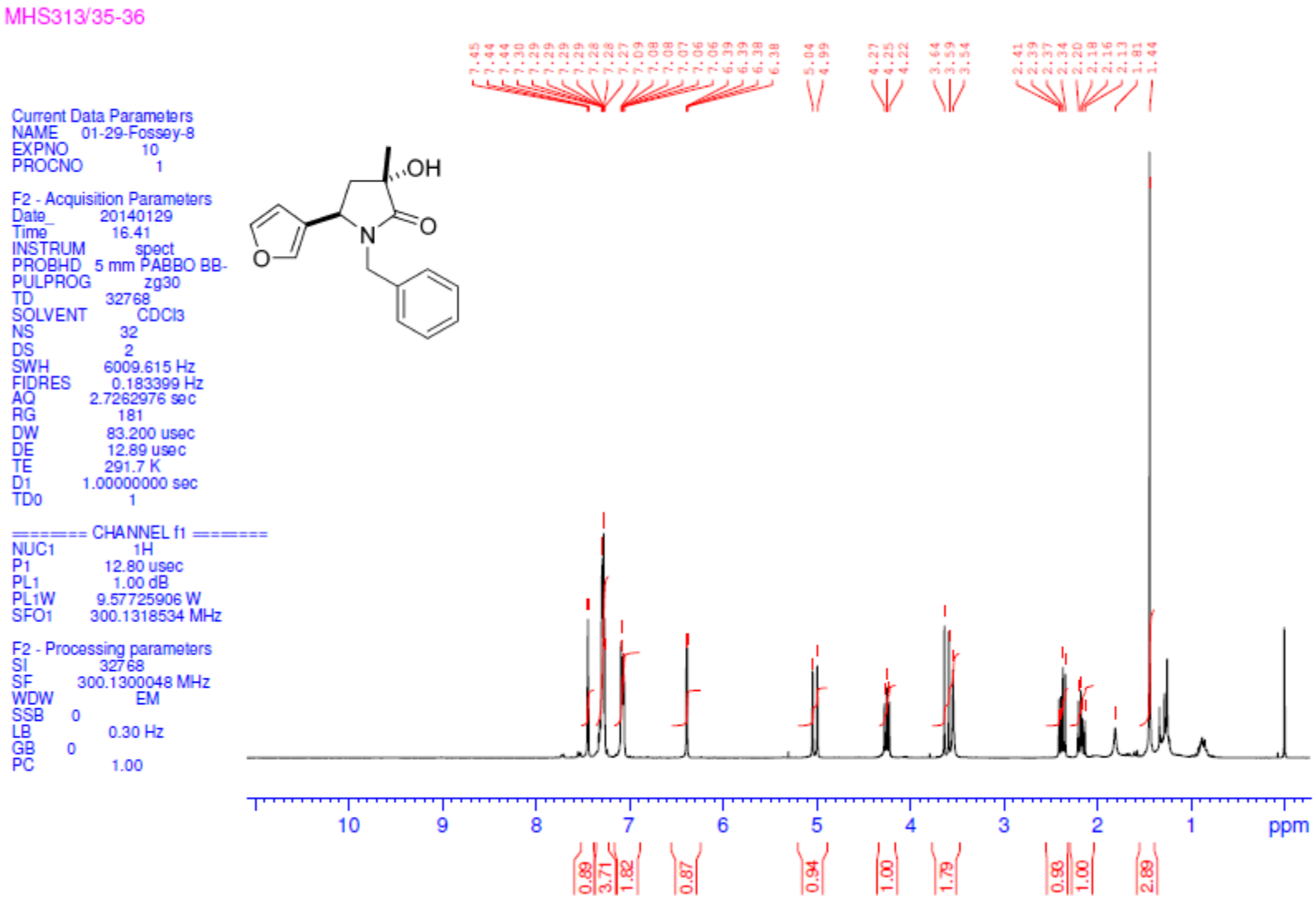
^{1}H NMR Spectra of compounds *cis*-6e and *trans*-6e (mixture of diastereoisomers)



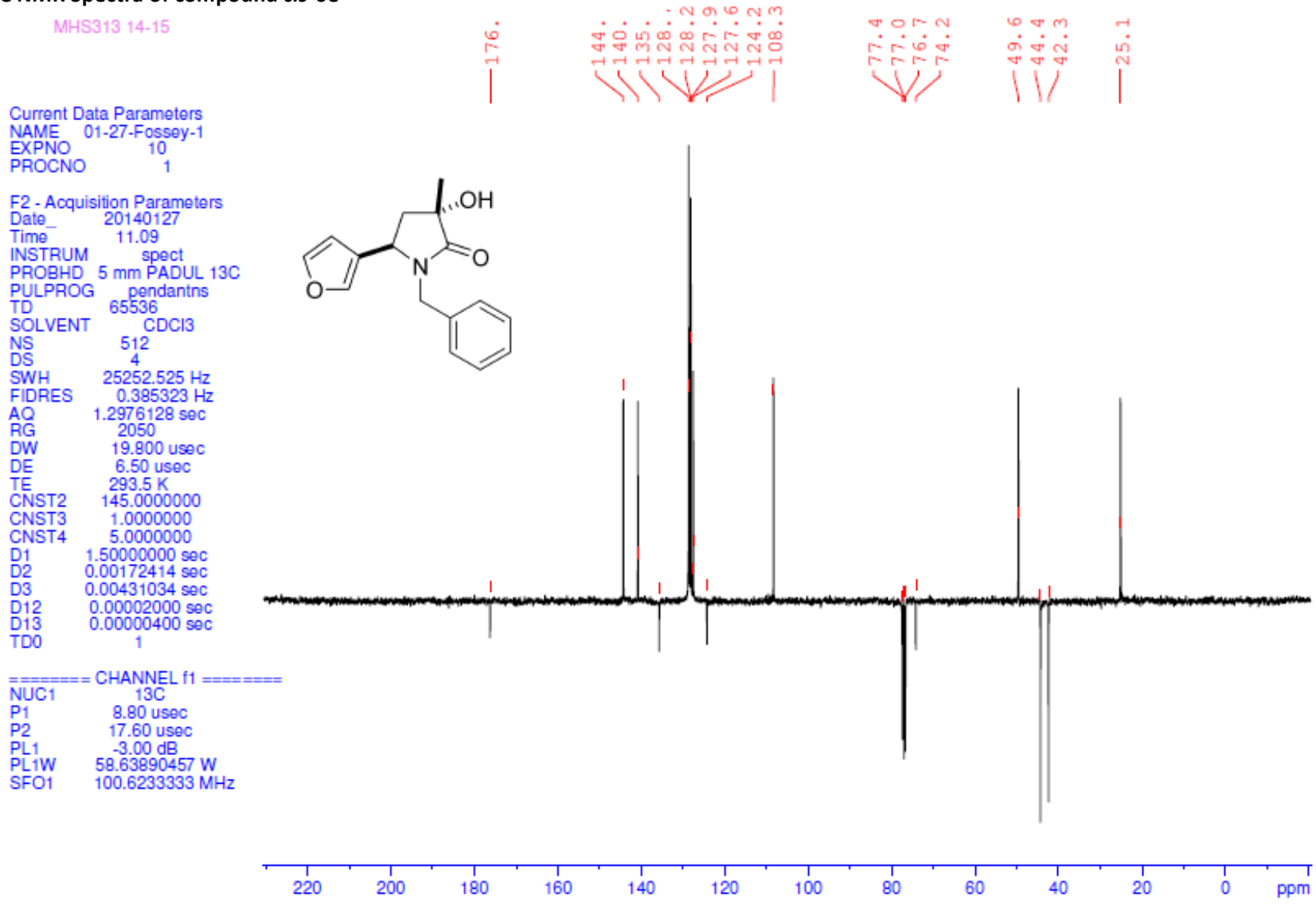
^{13}C NMR Spectra of compounds *cis*-6e and *trans*-6e (mixture of diastereoisomers)



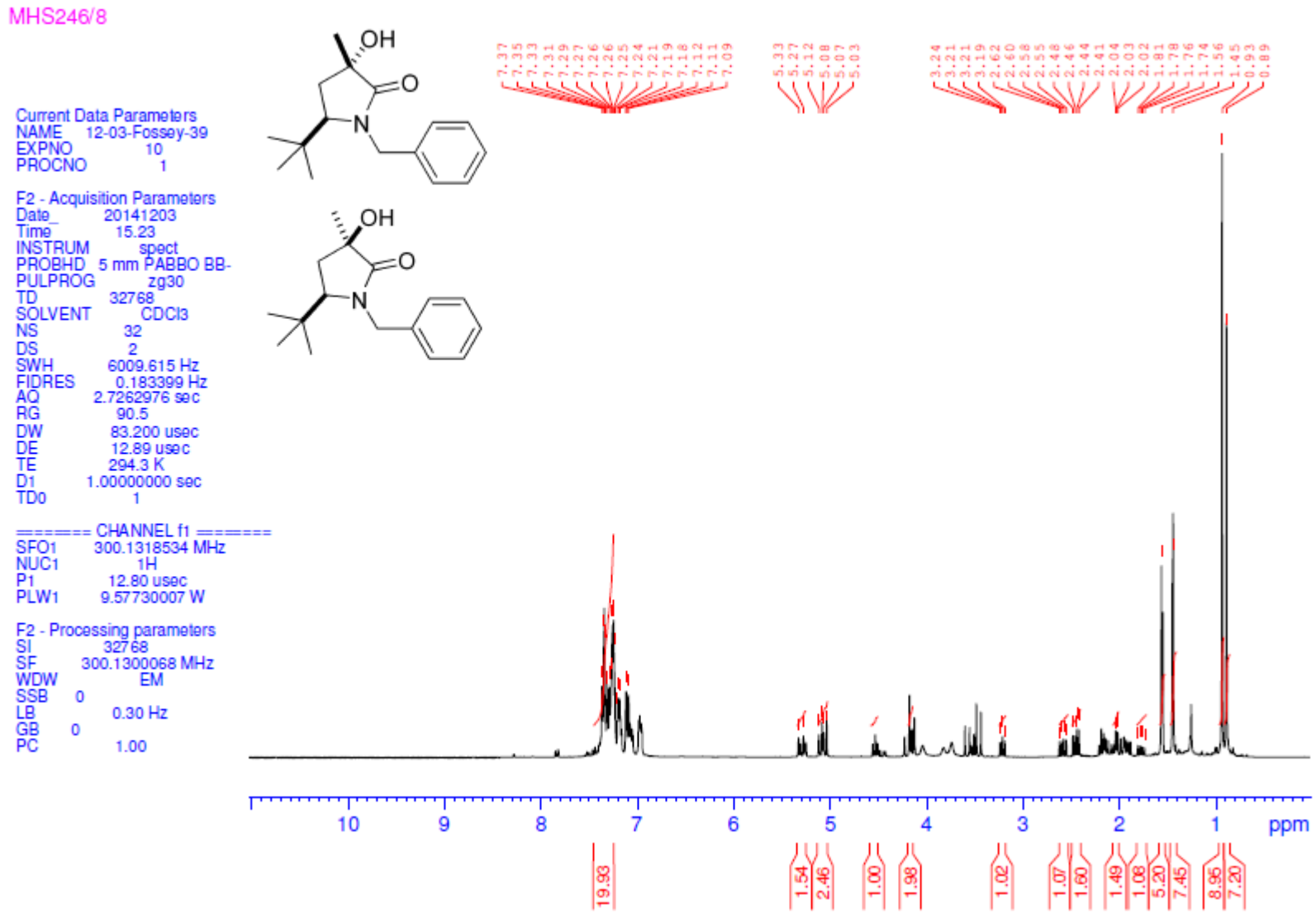
^{1}H NMR Spectra of compound *cis*-6e



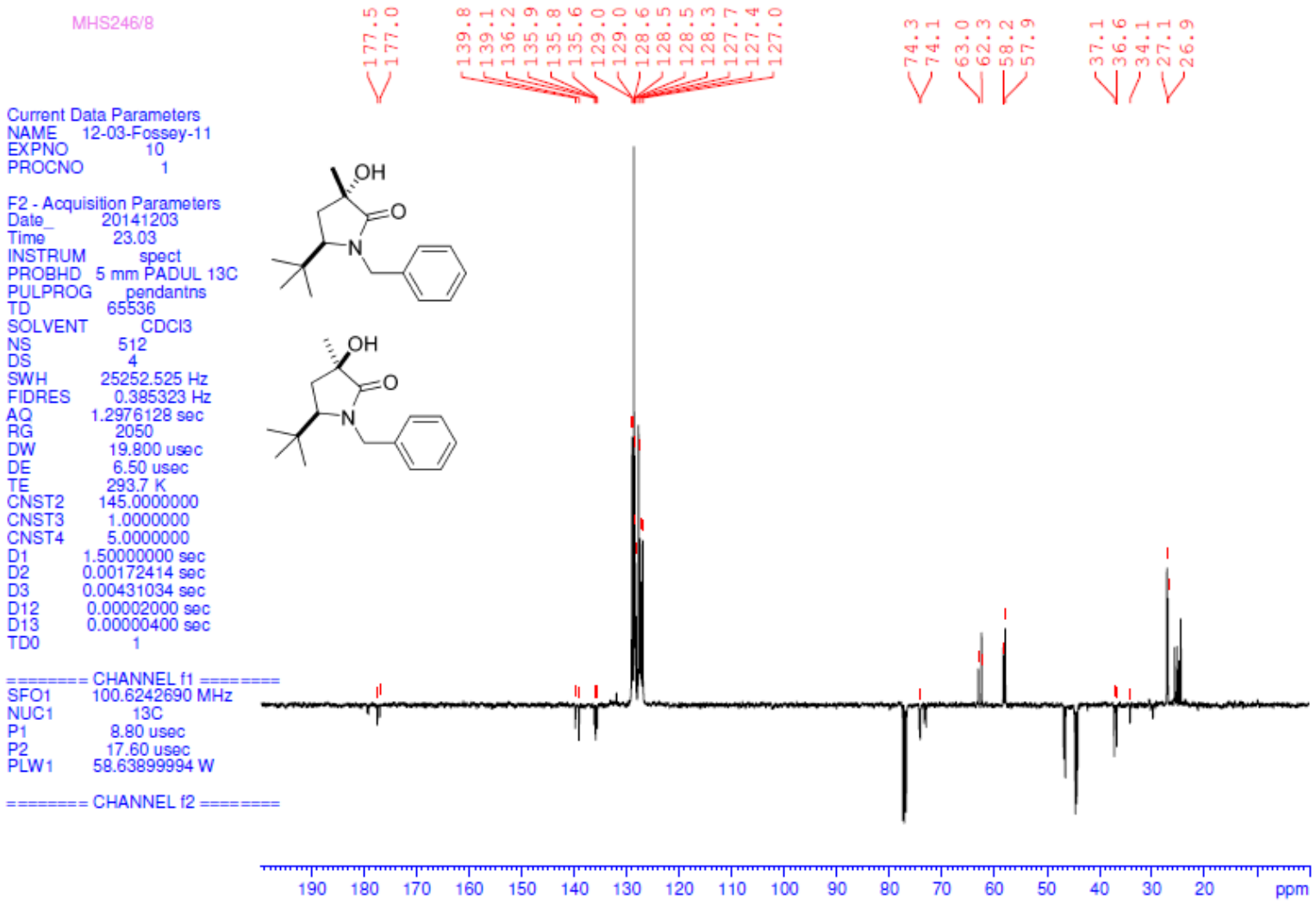
^{13}C NMR Spectra of compound *cis*-6e



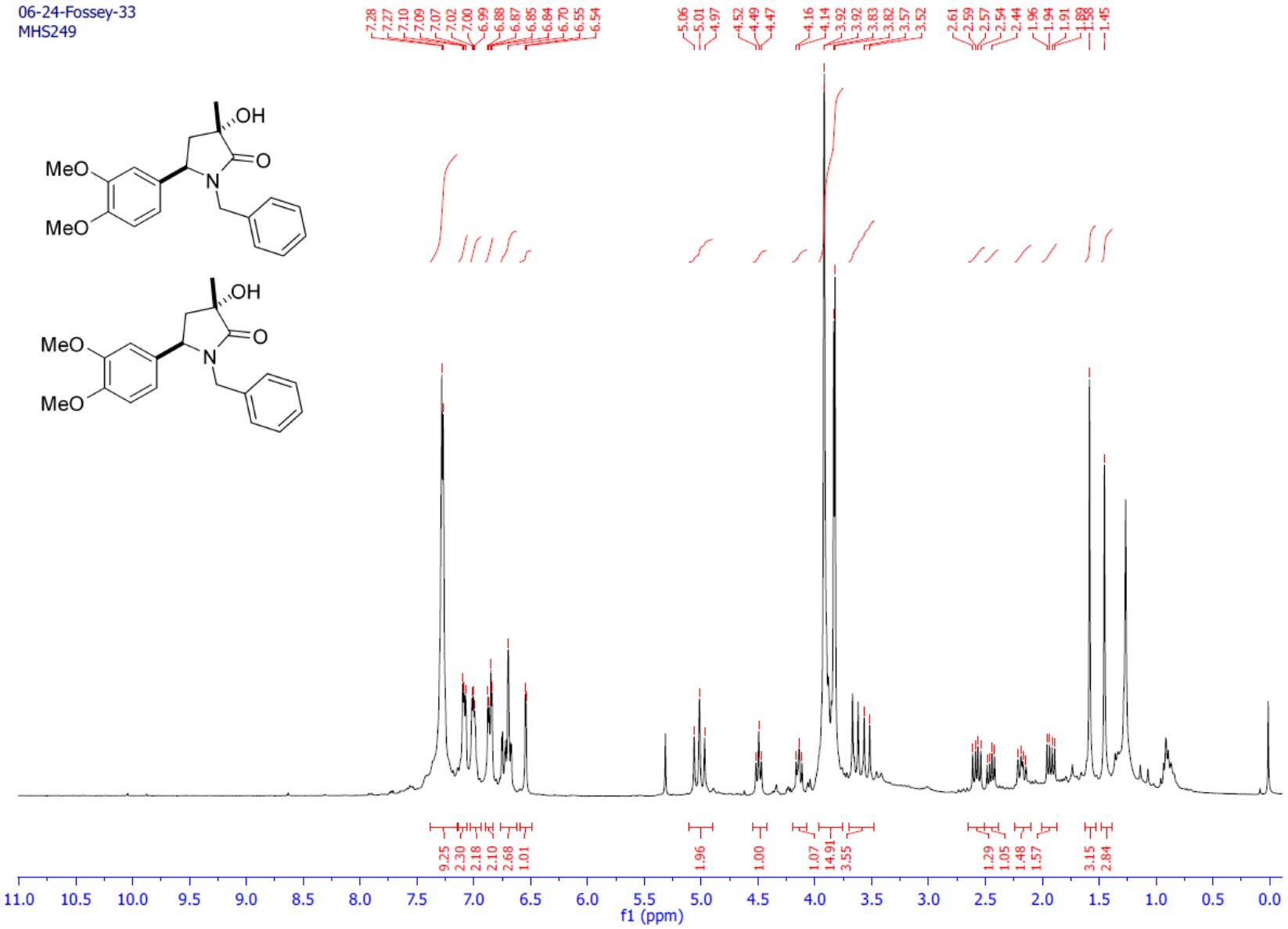
^{1}H NMR Spectra of compounds *cis*-6f and *trans*-6f (Mixture of diastereoisomers)



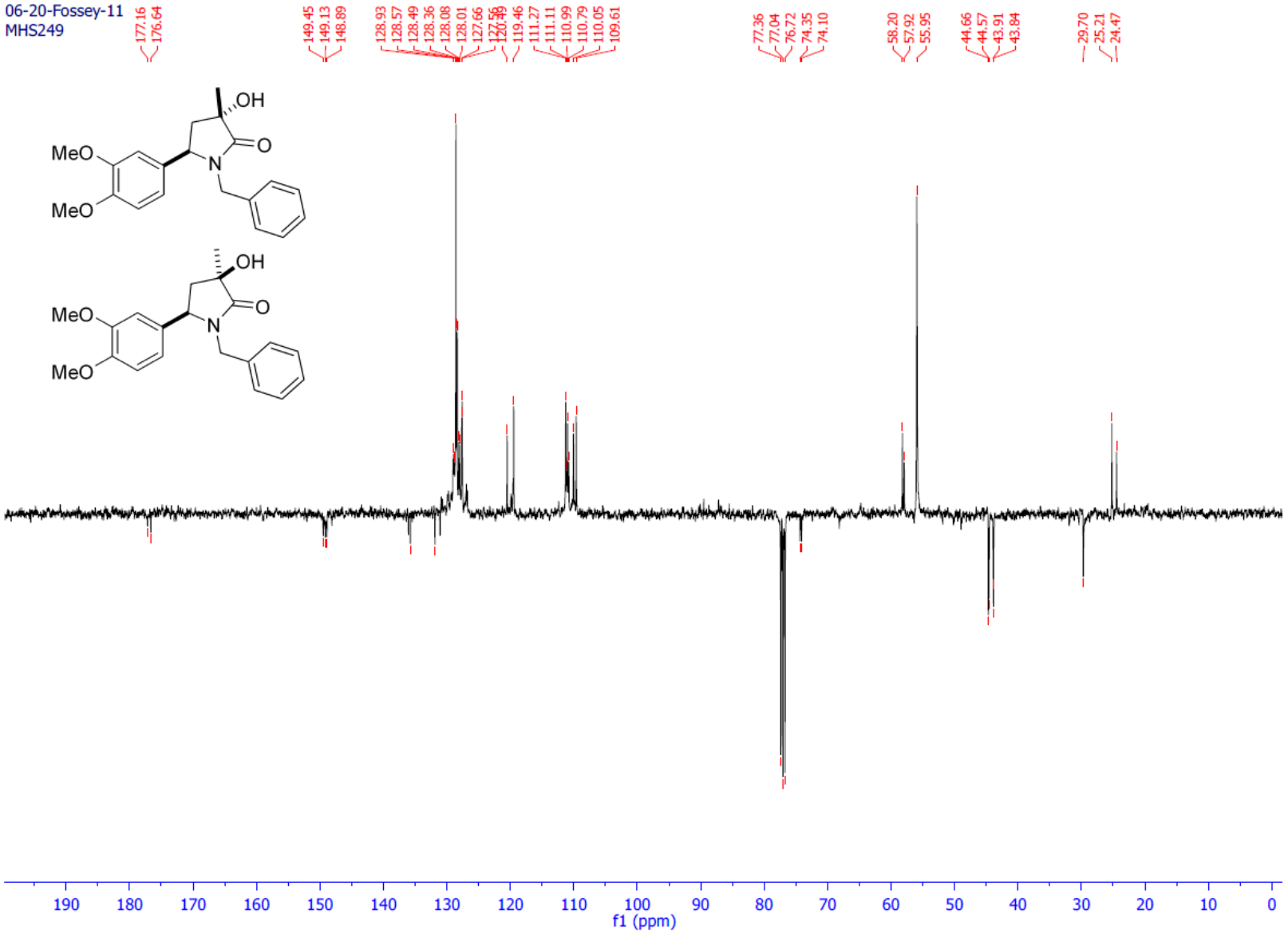
^{13}C NMR Spectra of compounds *cis*-6f and *trans*-6f (mixture of diastereoisomers)



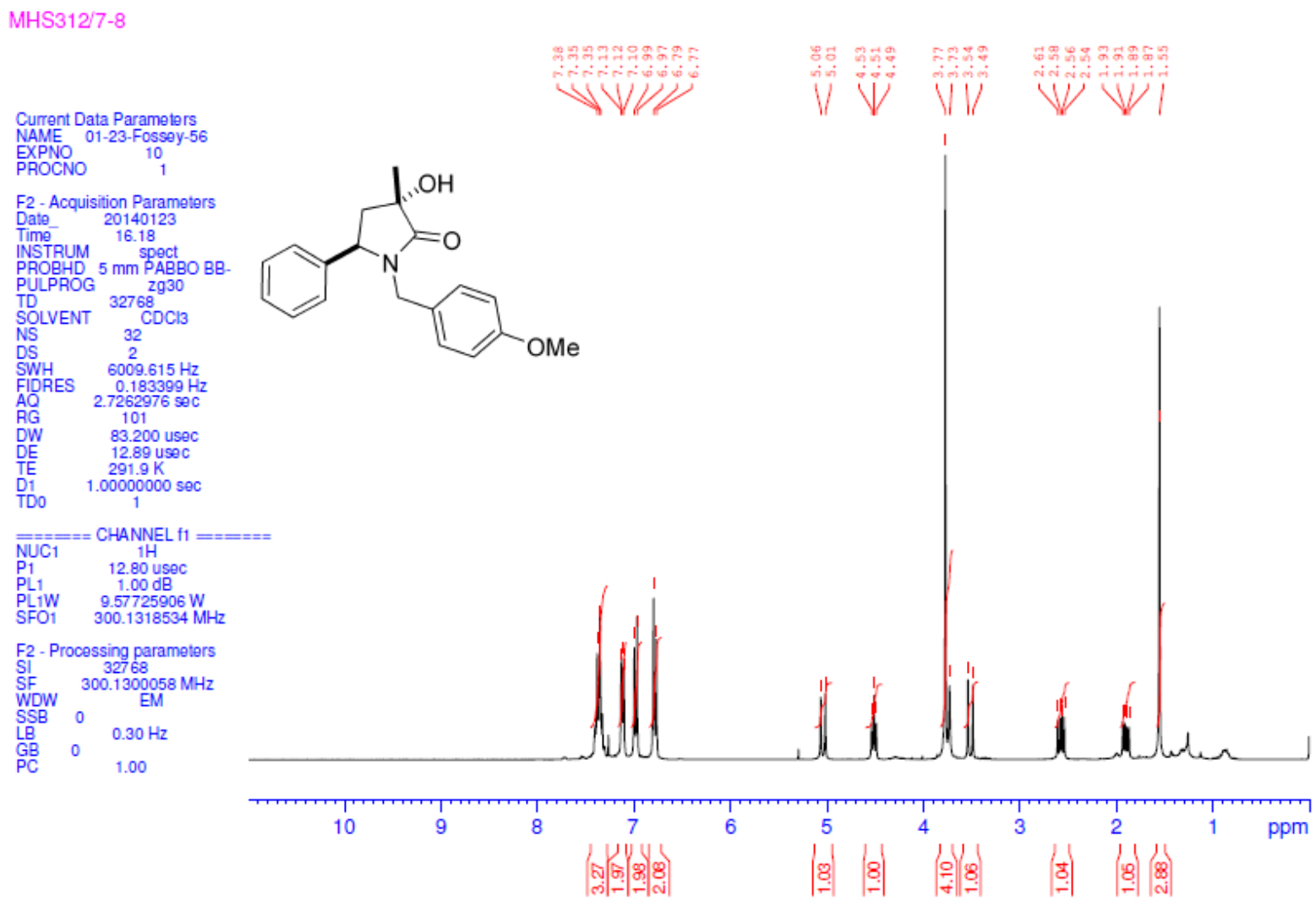
^{1}H NMR Spectra of compounds *cis*-6g and *trans*-6g (mixture of diastereoisomers)



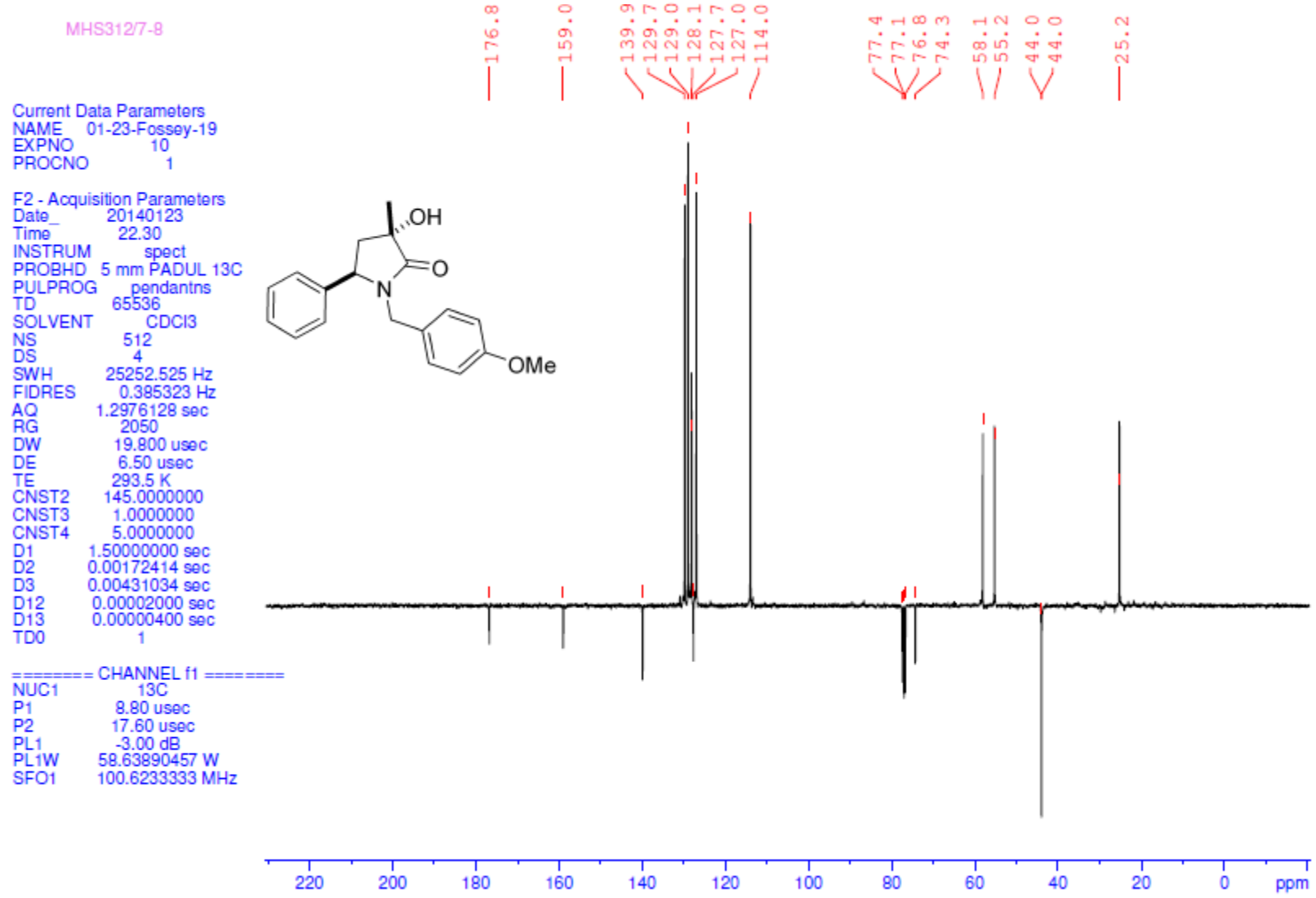
^{13}C NMR Spectra of compounds *cis*-6g and *trans*-6g (Mixture of diastereoisomers)



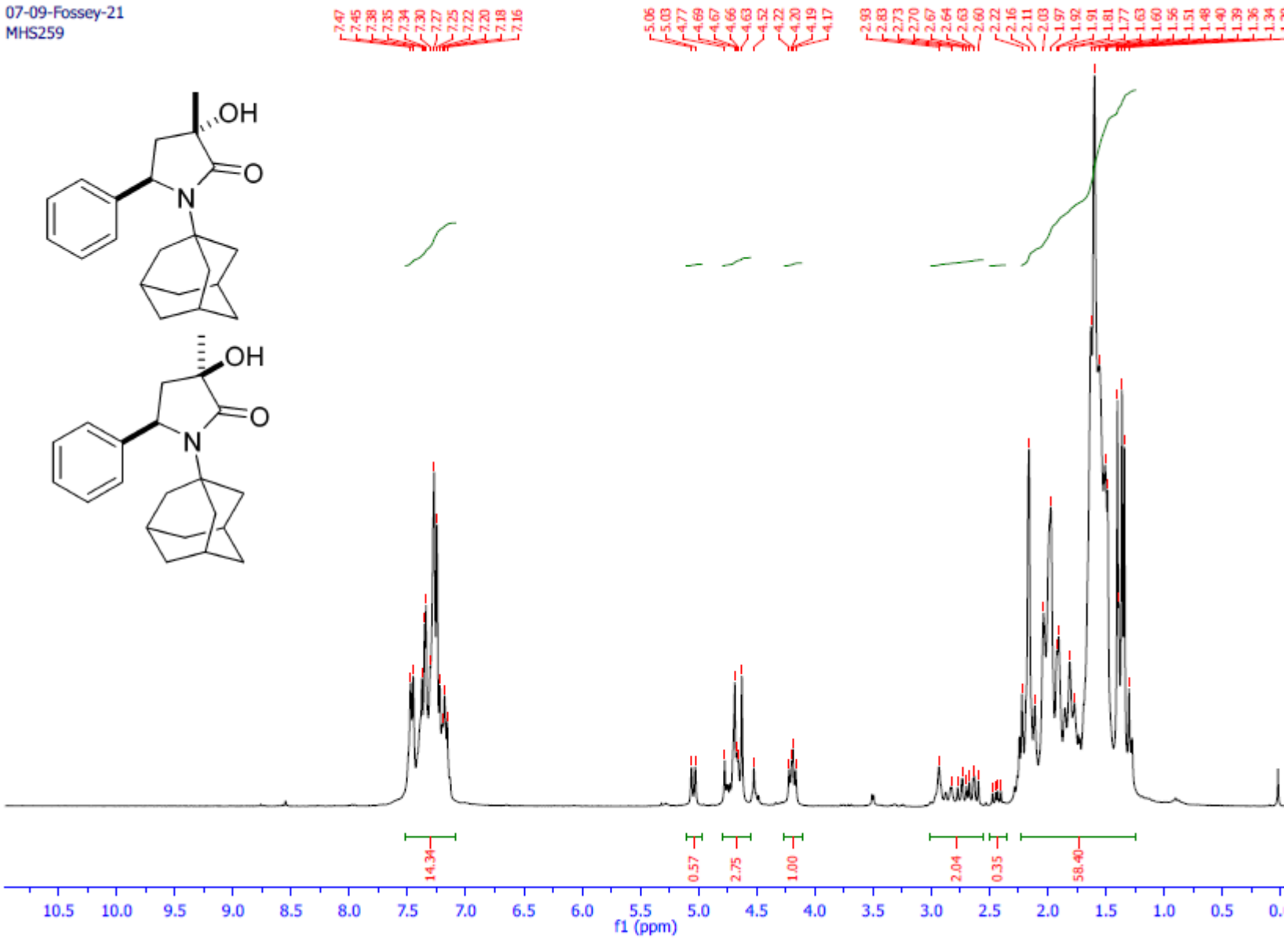
^{1}H NMR Spectra of compound *cis*-6h



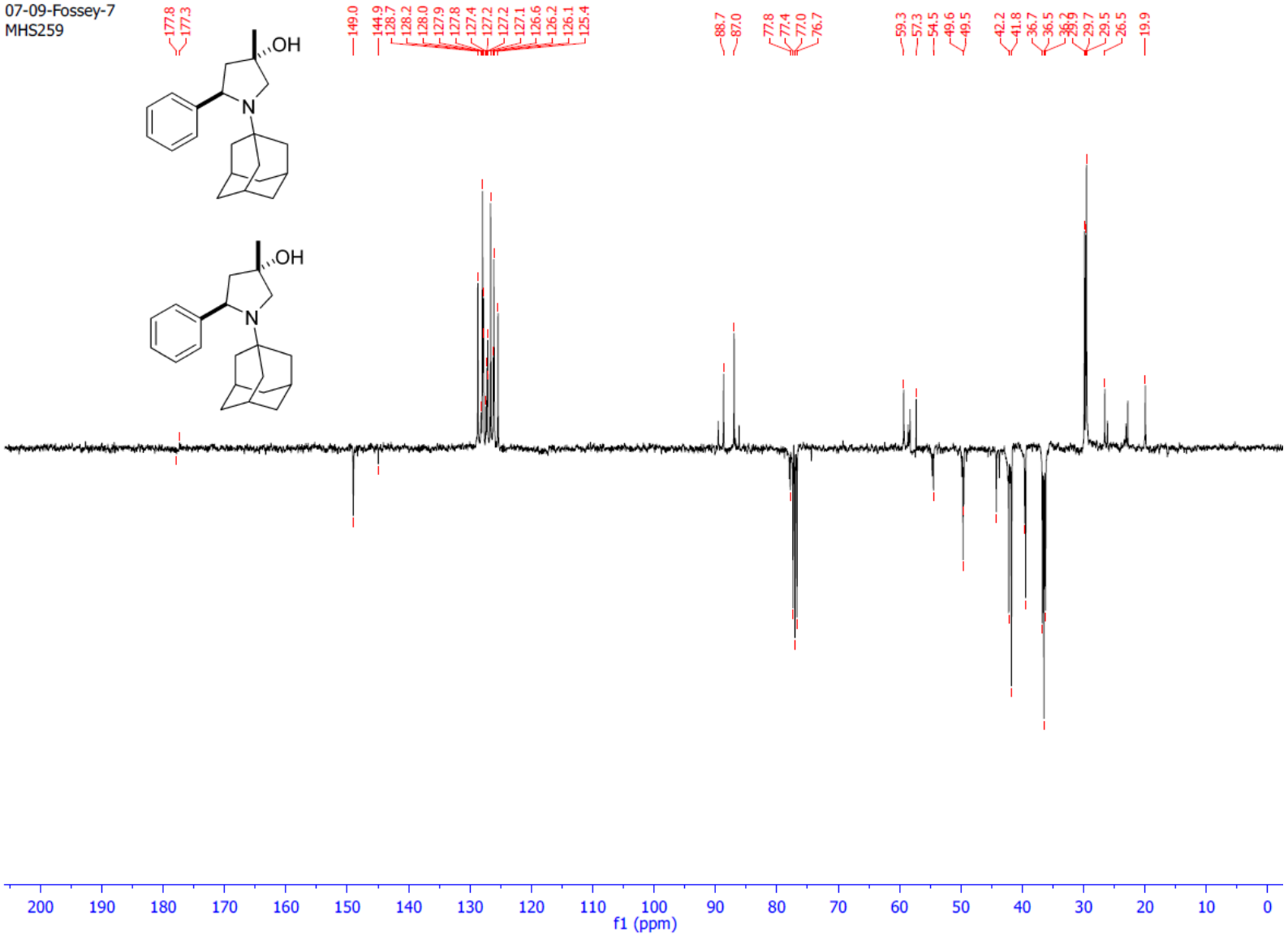
^{13}C NMR Spectra of compound *cis*-6h



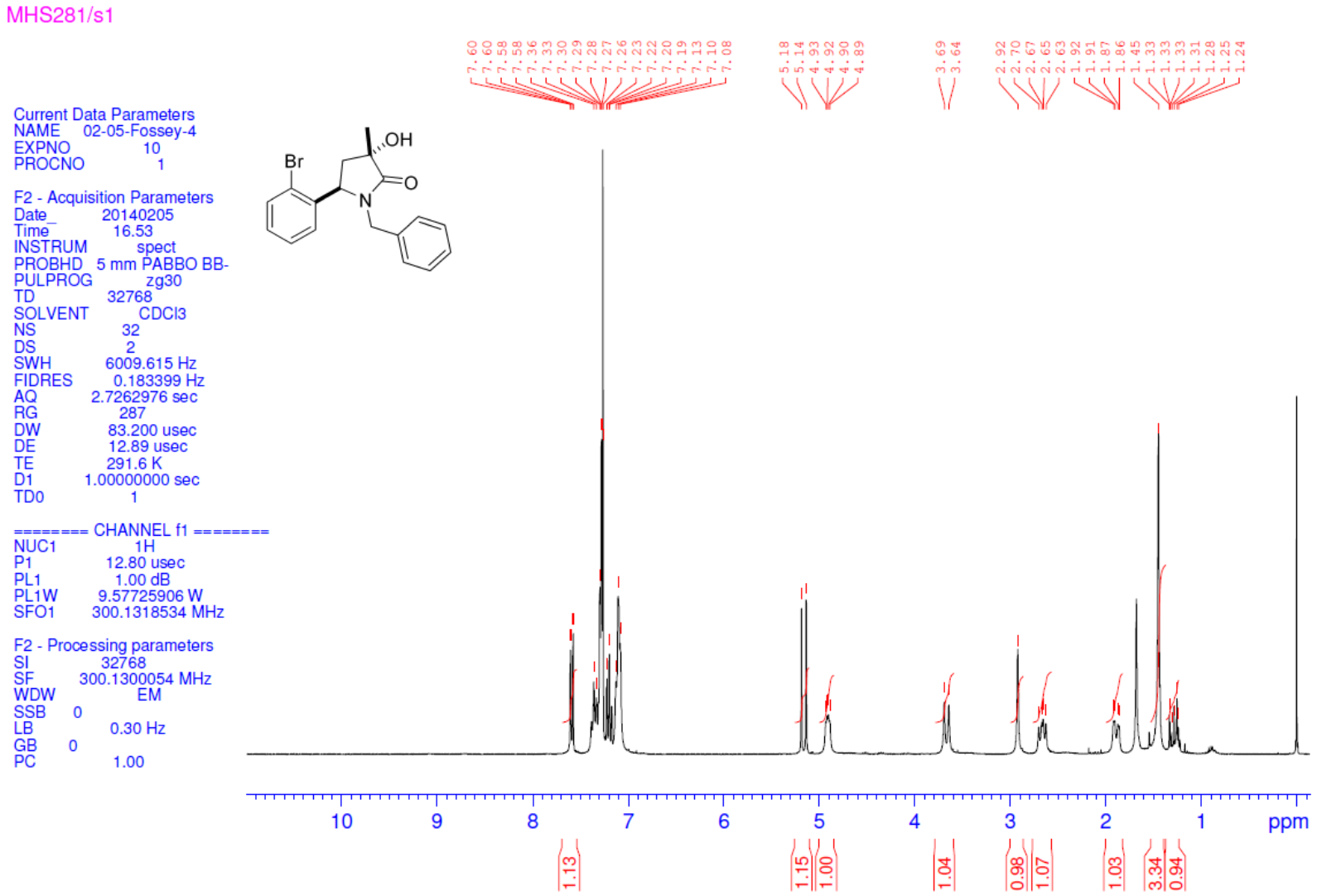
^{1}H NMR Spectra of compounds *cis*-6i and *trans*-6i (mixture of diastereoisomers)



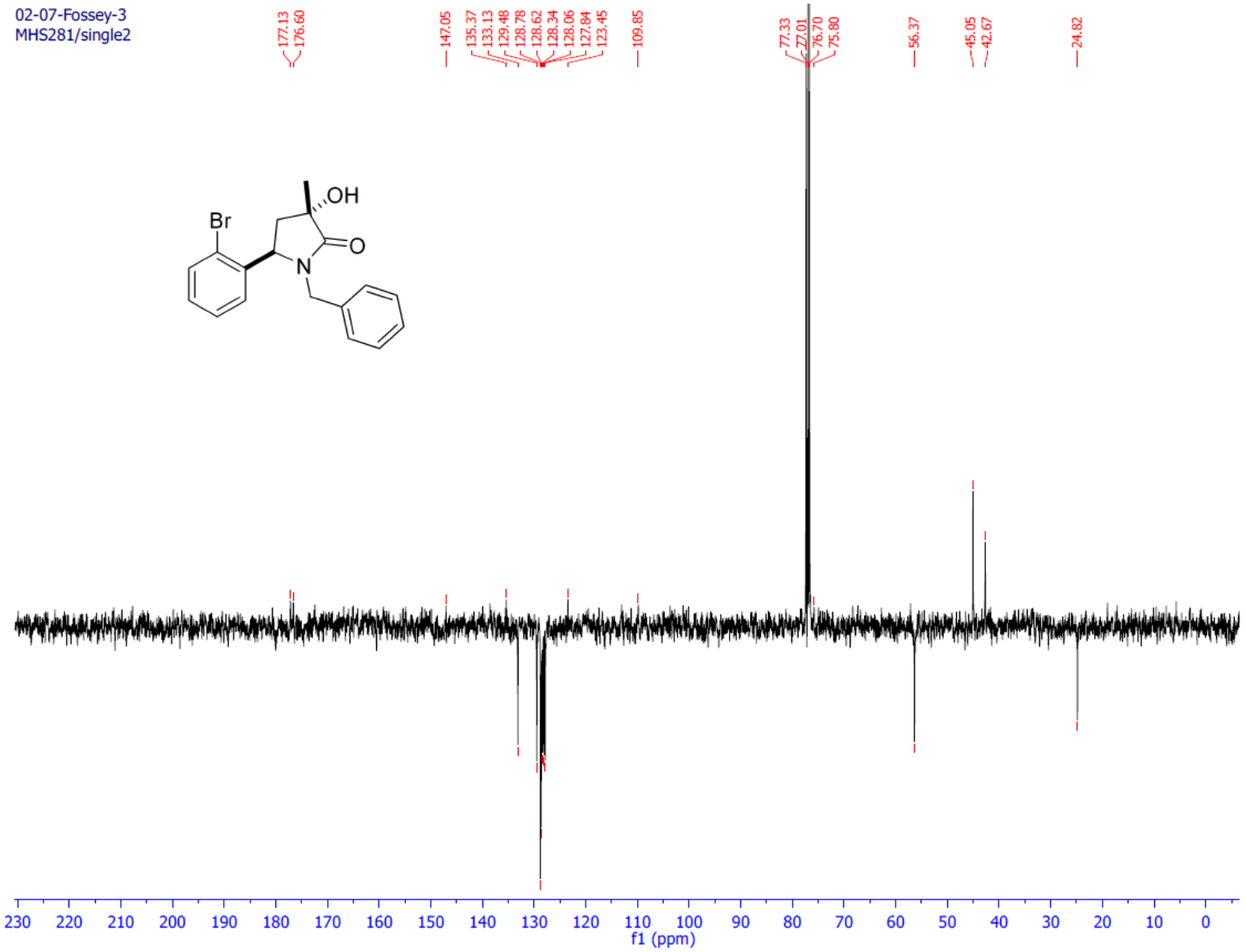
^{13}C NMR Spectra of compounds *cis*-6i and *trans*-6i (mixture of diastereoisomers)



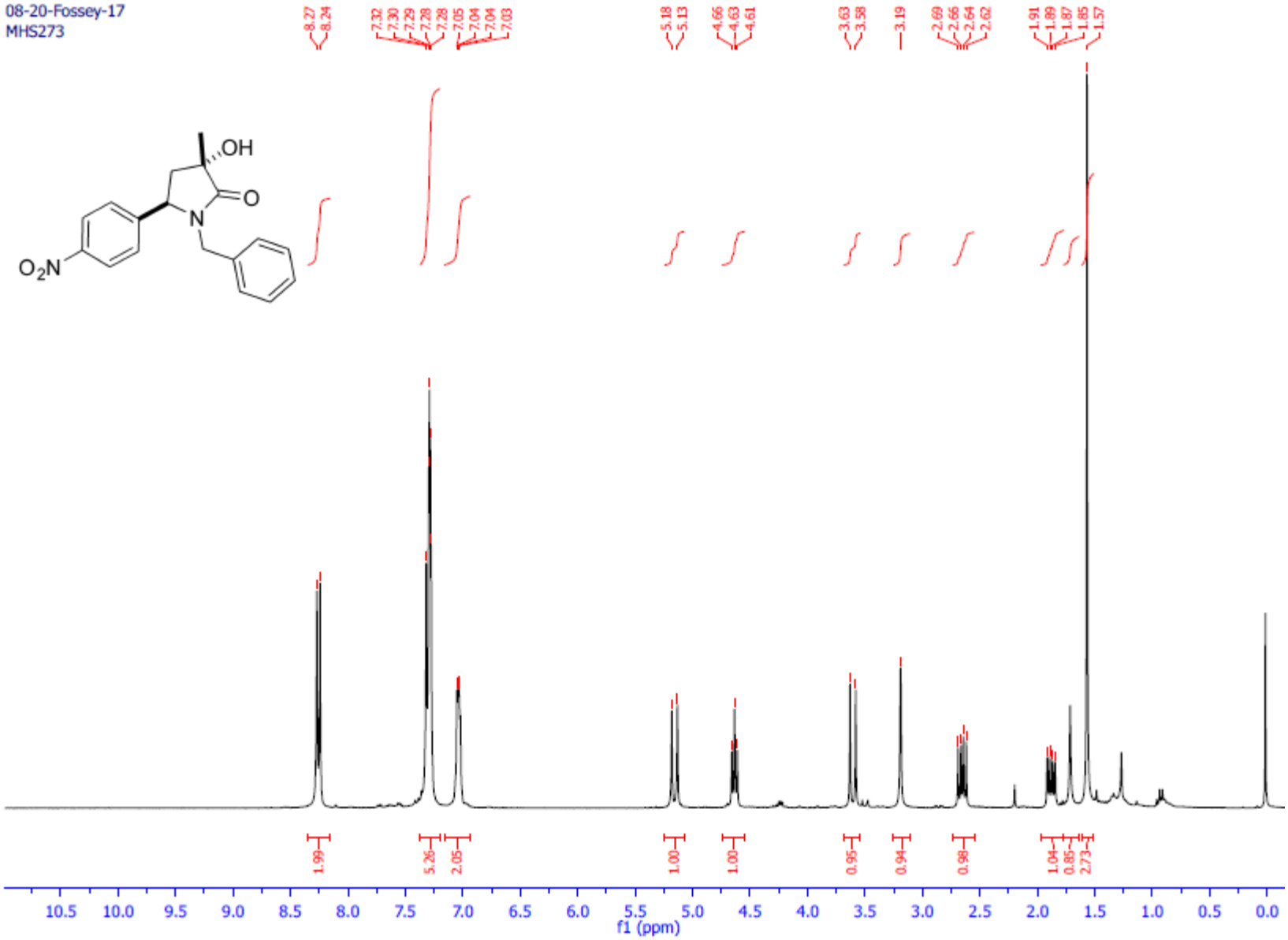
^{1}H NMR Spectra of compound *cis*-6k



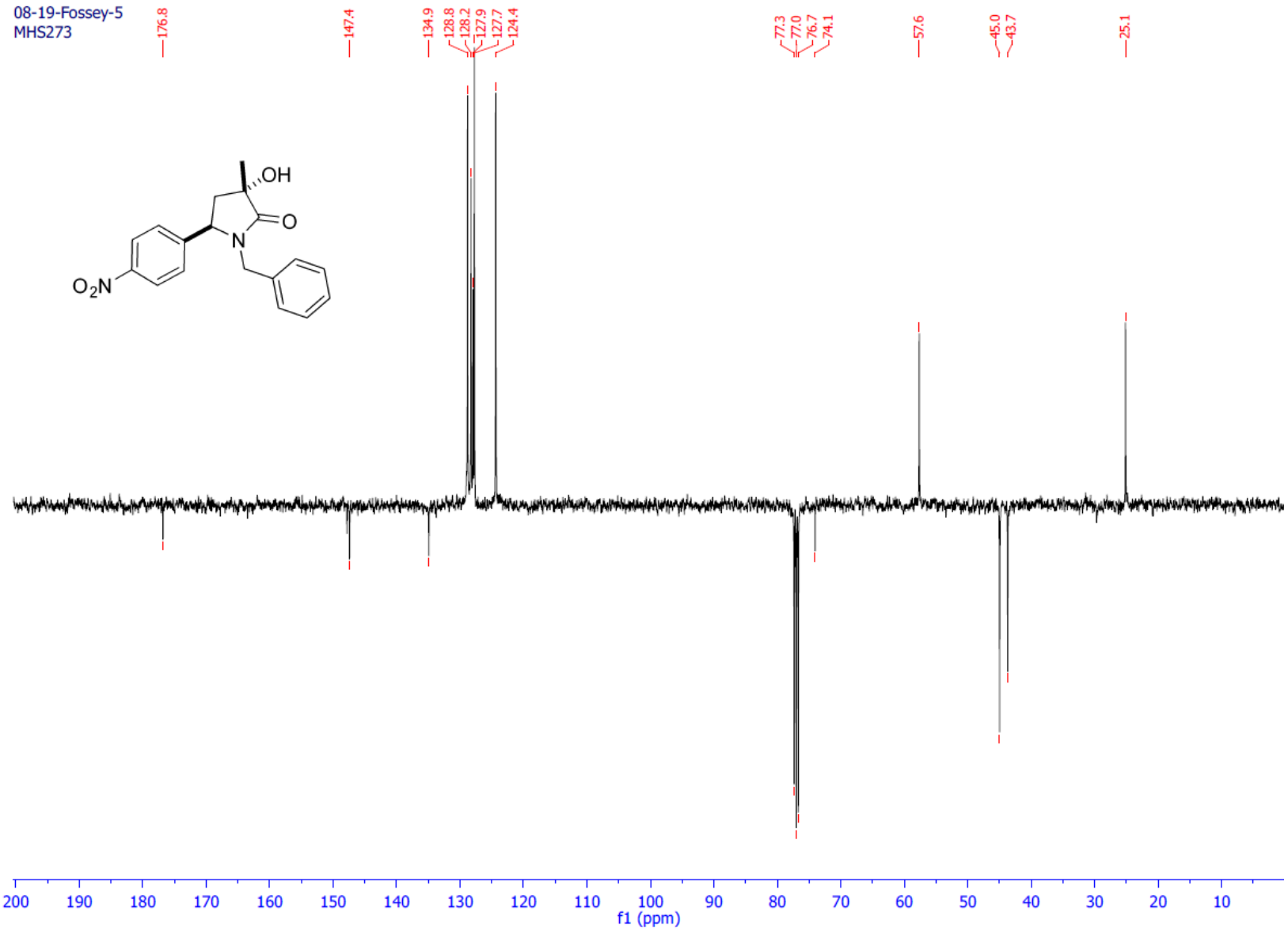
^{13}C NMR Spectra of compound *cis*-6k



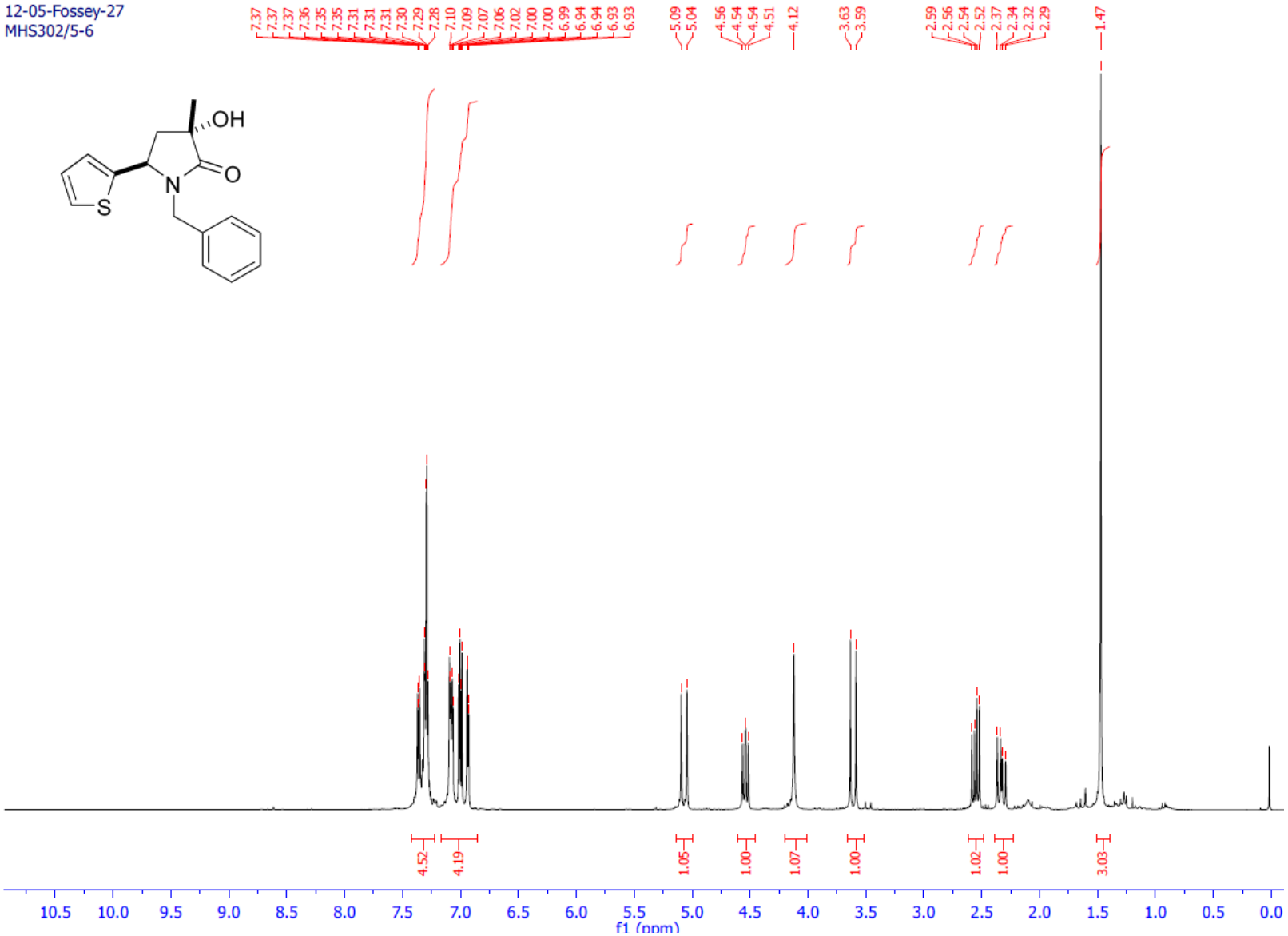
^{1}H NMR Spectra of compound *cis*-6m



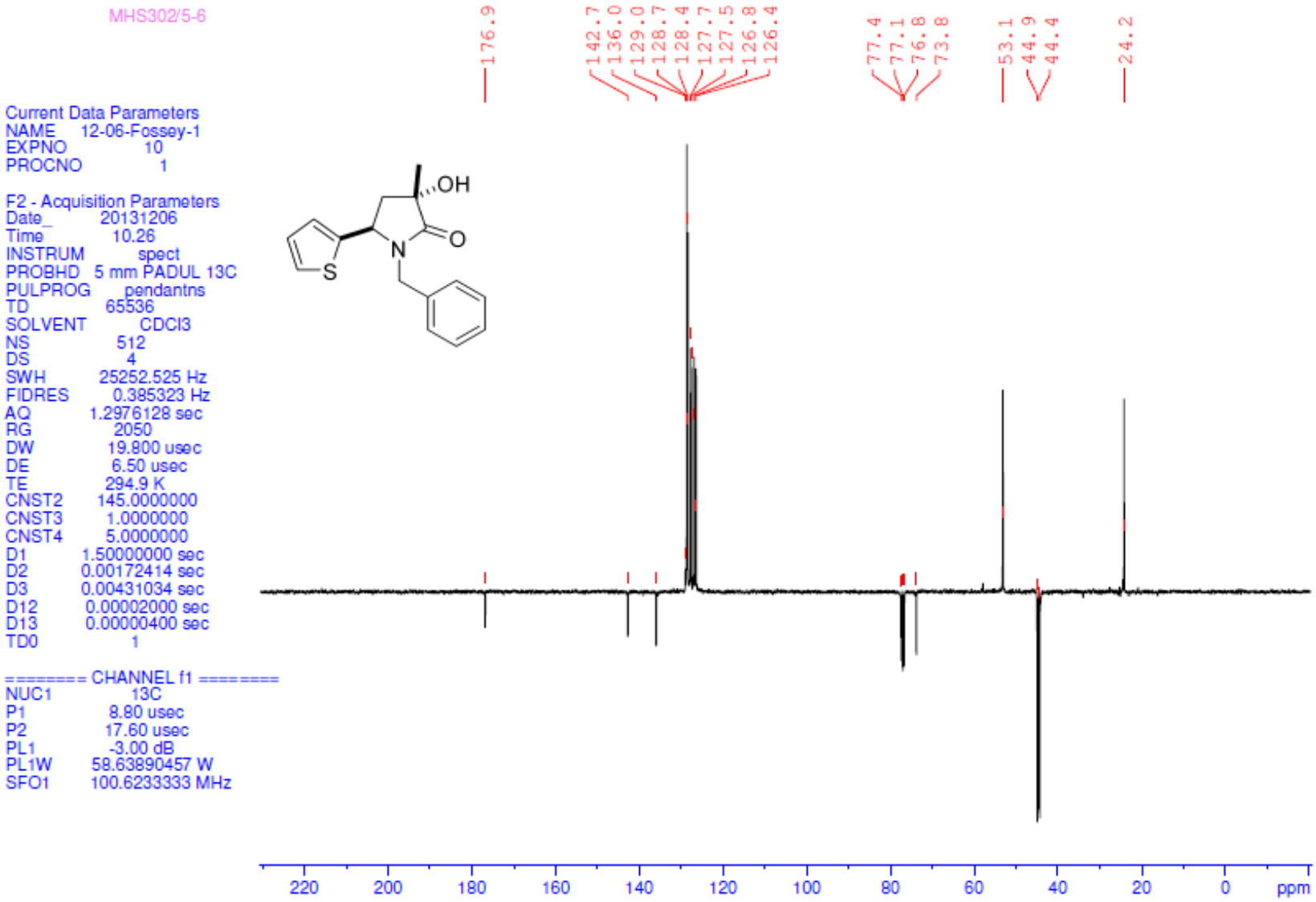
^{13}C NMR Spectra of compound *cis*-6m



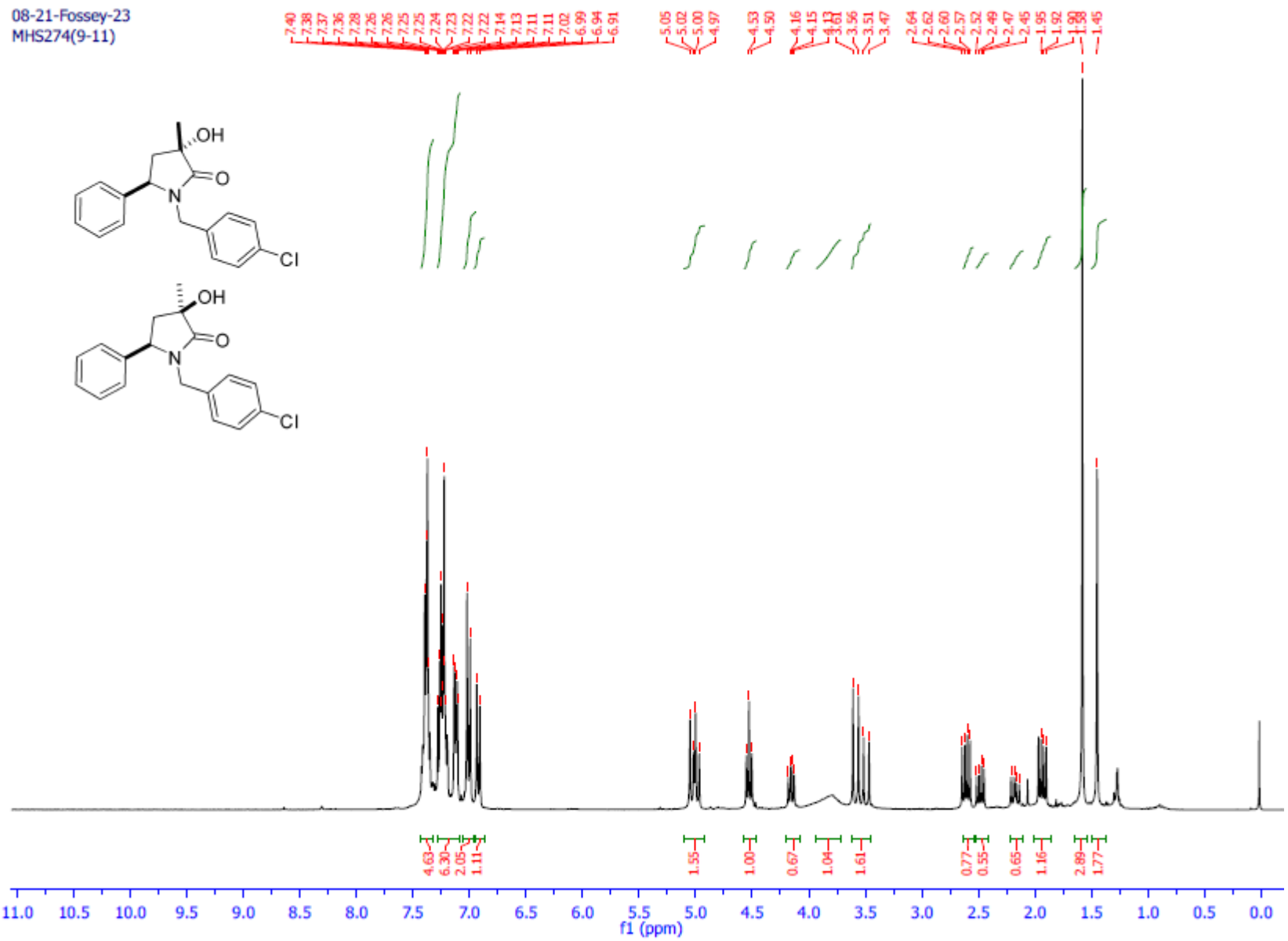
^{1}H NMR Spectra of compound *cis*-6j



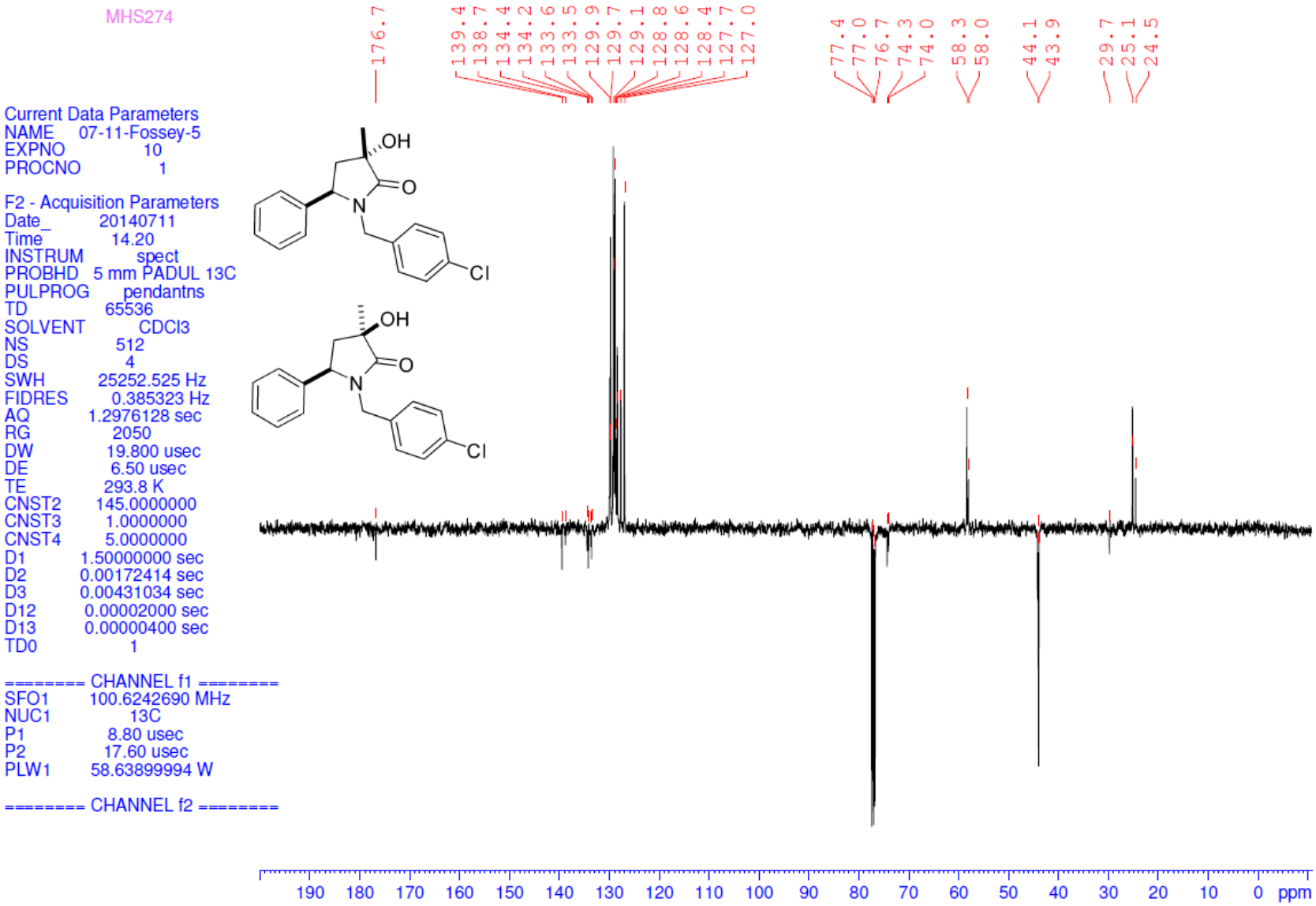
^{13}C NMR Spectra of compound *cis*-6j



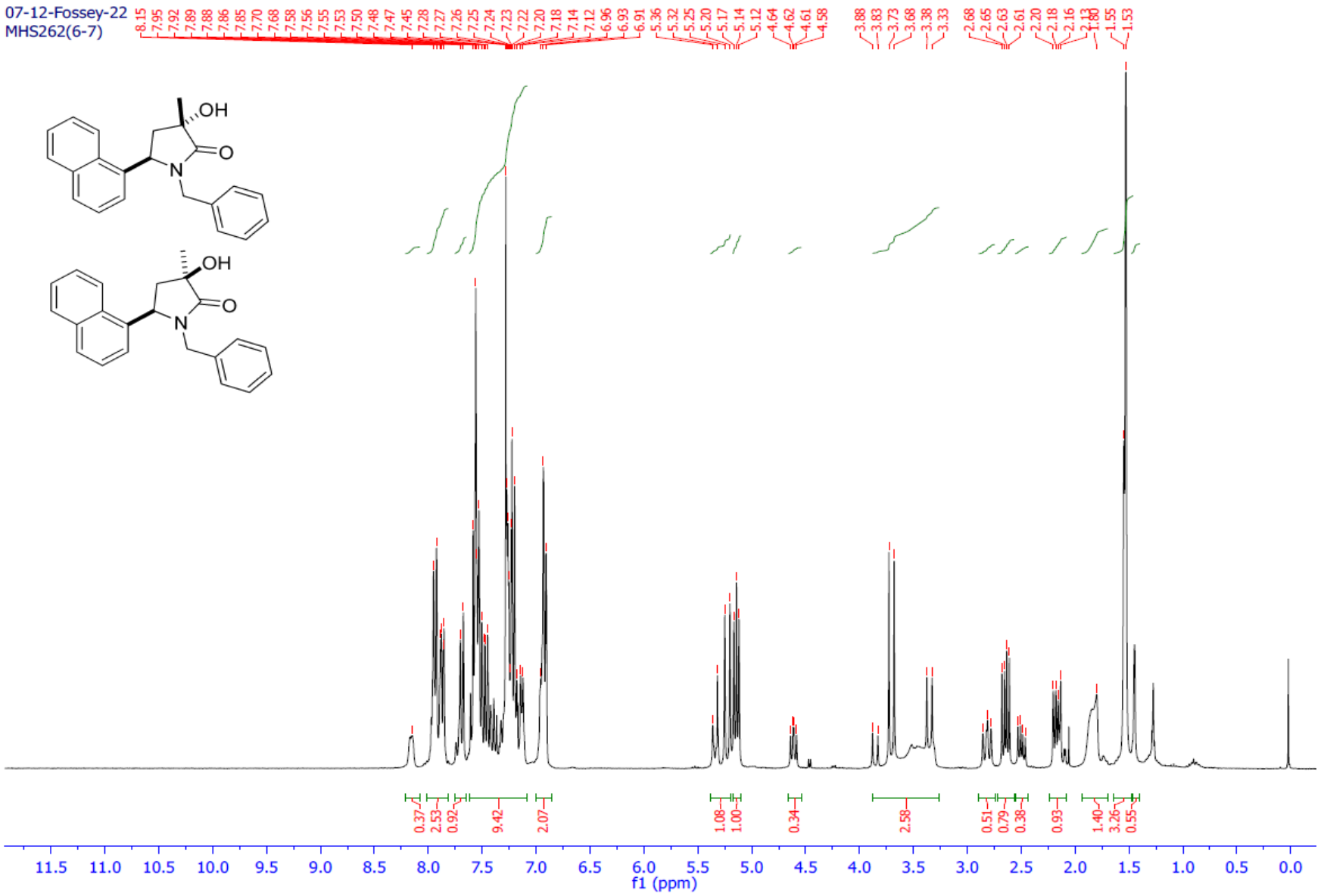
^{1}H NMR Spectra of compounds *cis*-6l and *trans*-6l (mixture of diastereoisomers)



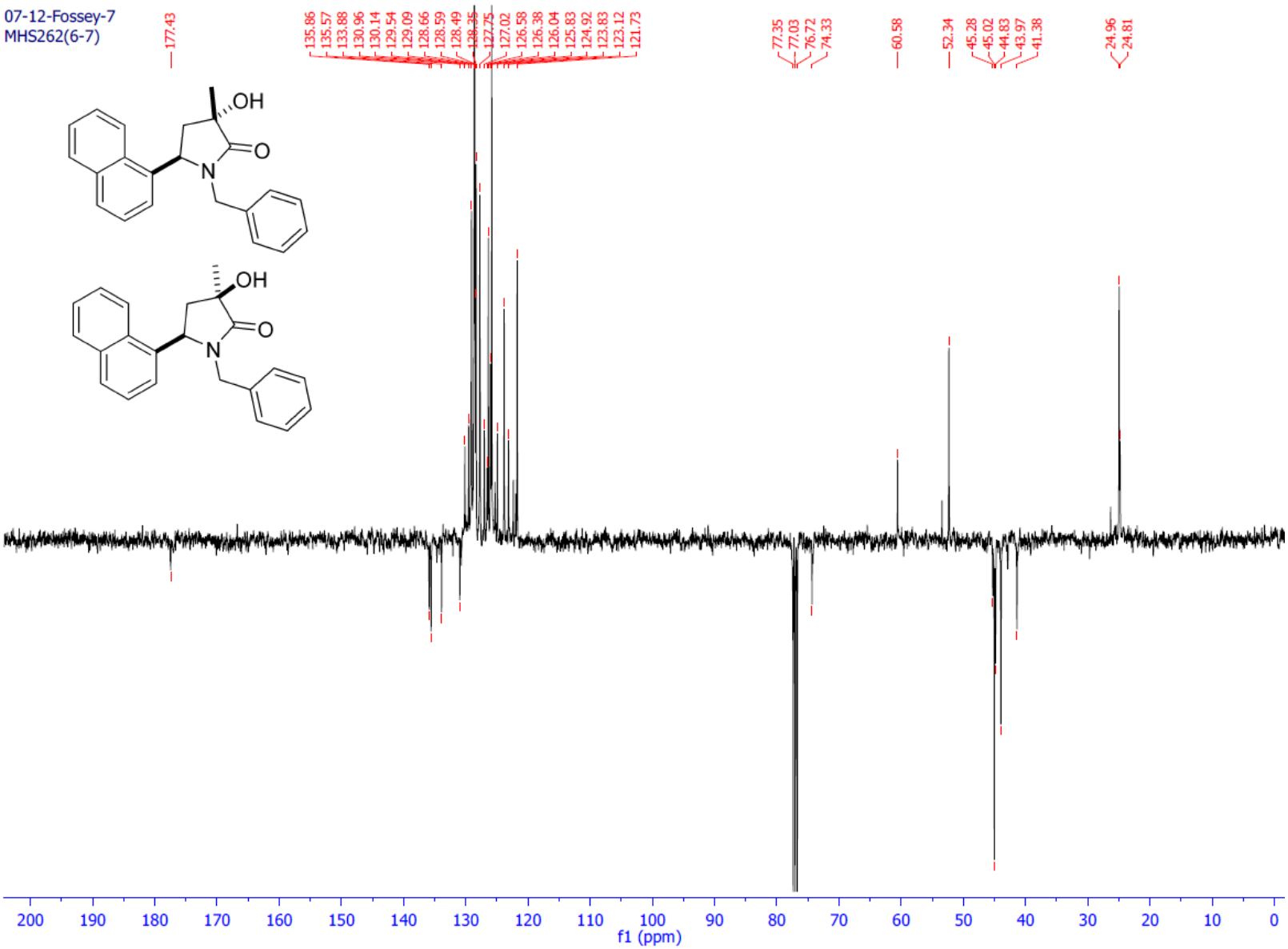
^{13}C NMR Spectra of compounds *cis*-6l and *trans*-6l (mixture of diastereoisomers)



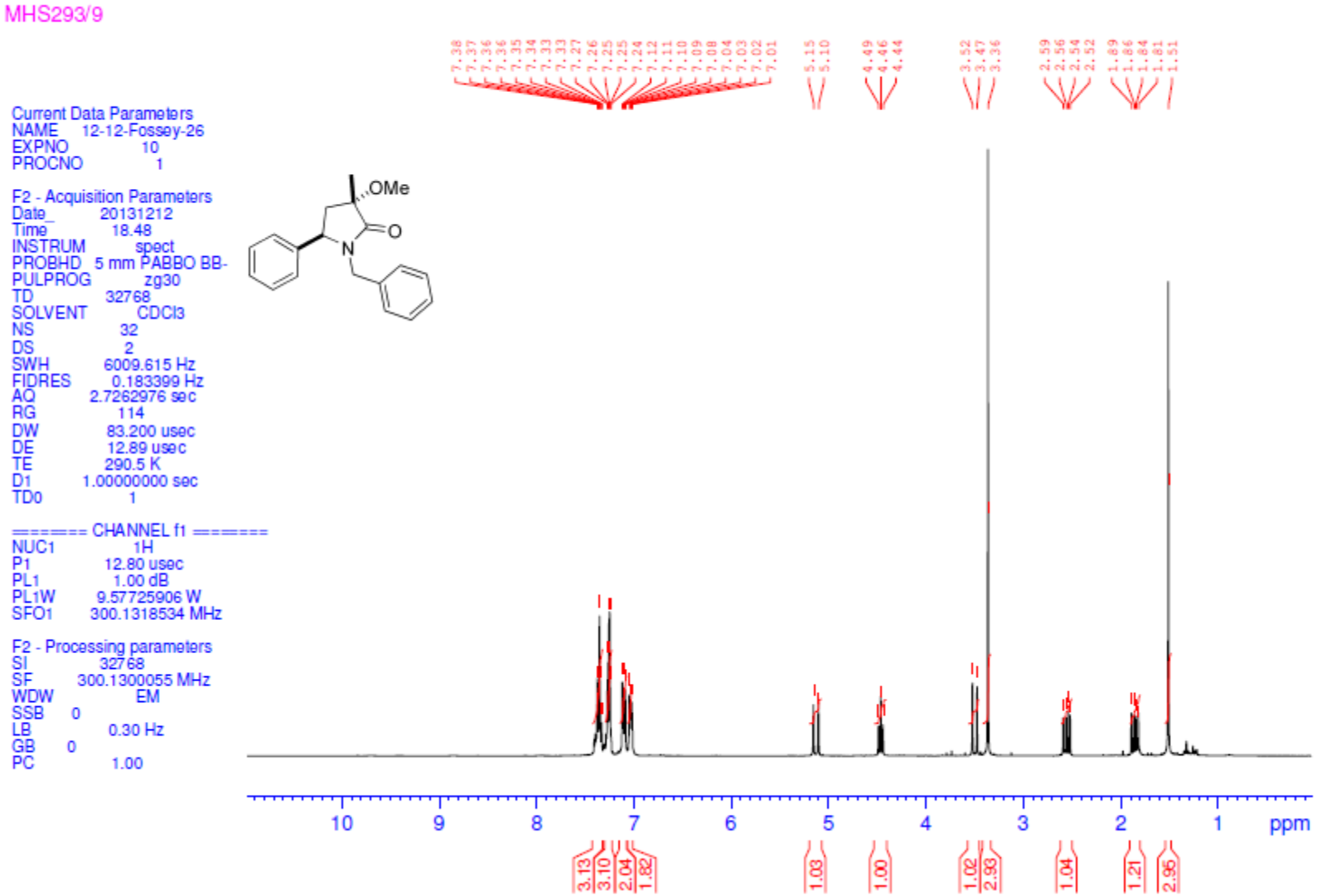
^{1}H NMR Spectra of compounds *cis*-6n and *trans*-6n (mixture of diastereoisomers)



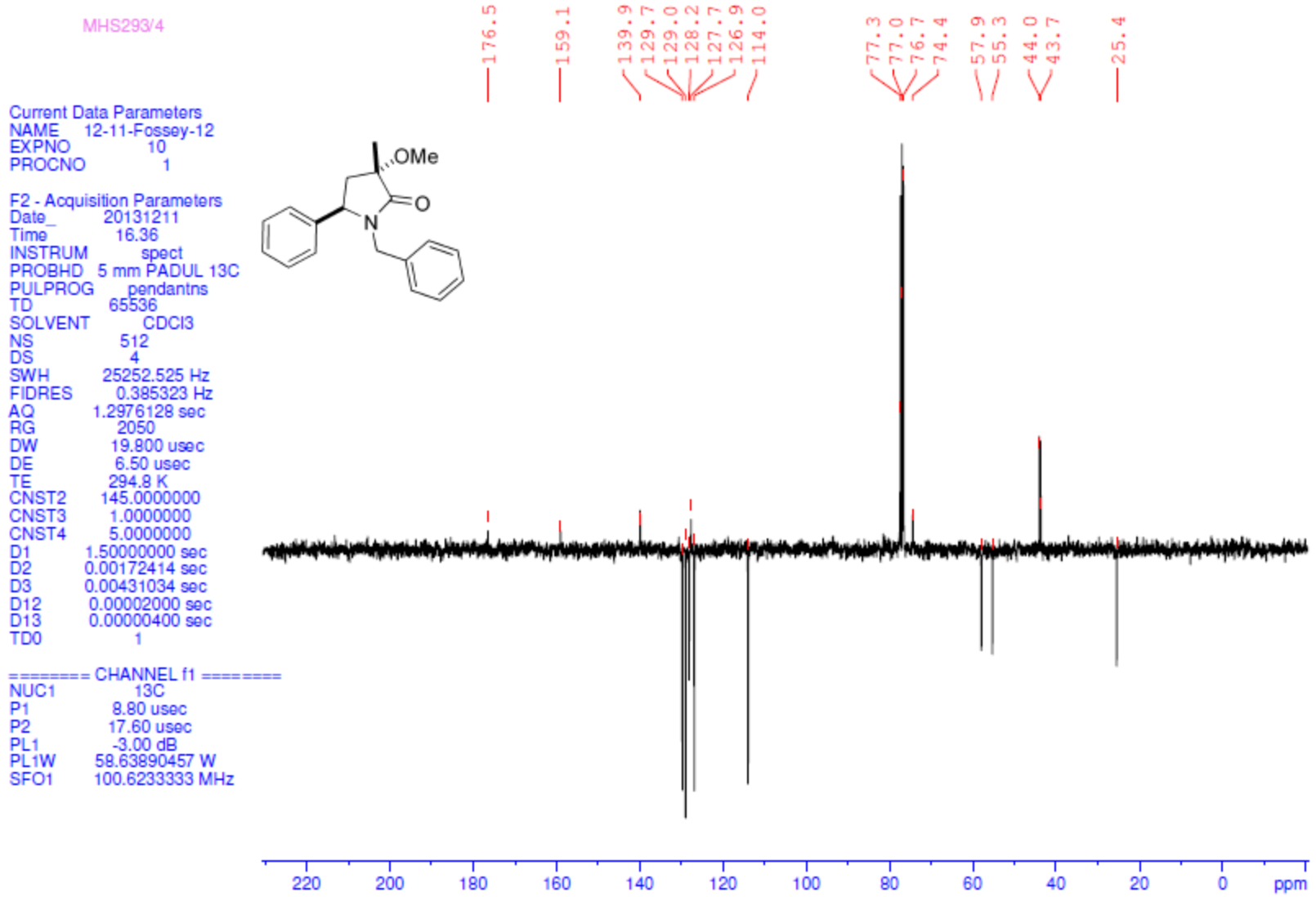
^{13}C NMR Spectra of compounds *cis*-6n and *trans*-6n (mixture of diastereoisomers)



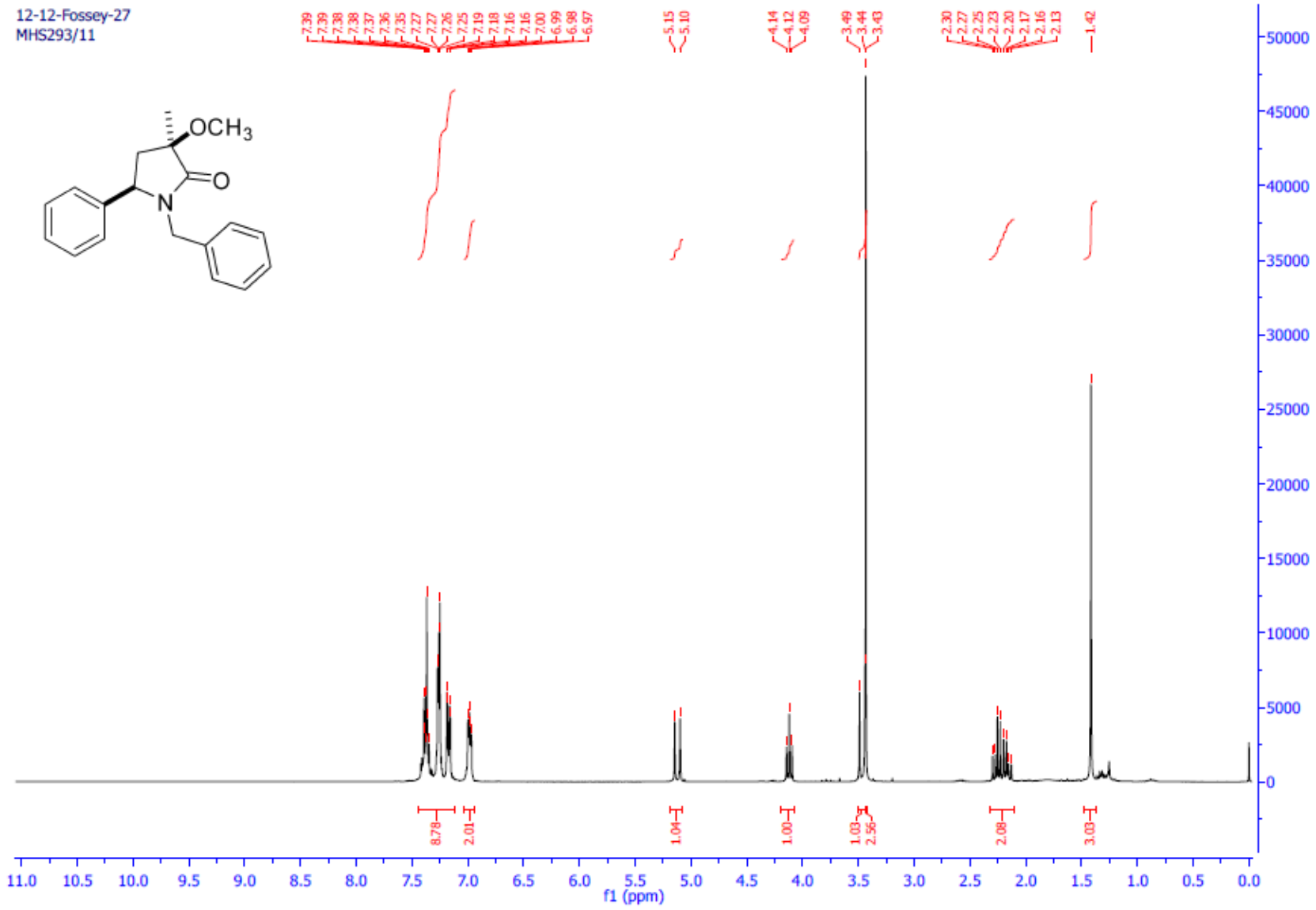
^{1}H NMR Spectra of compound 7a



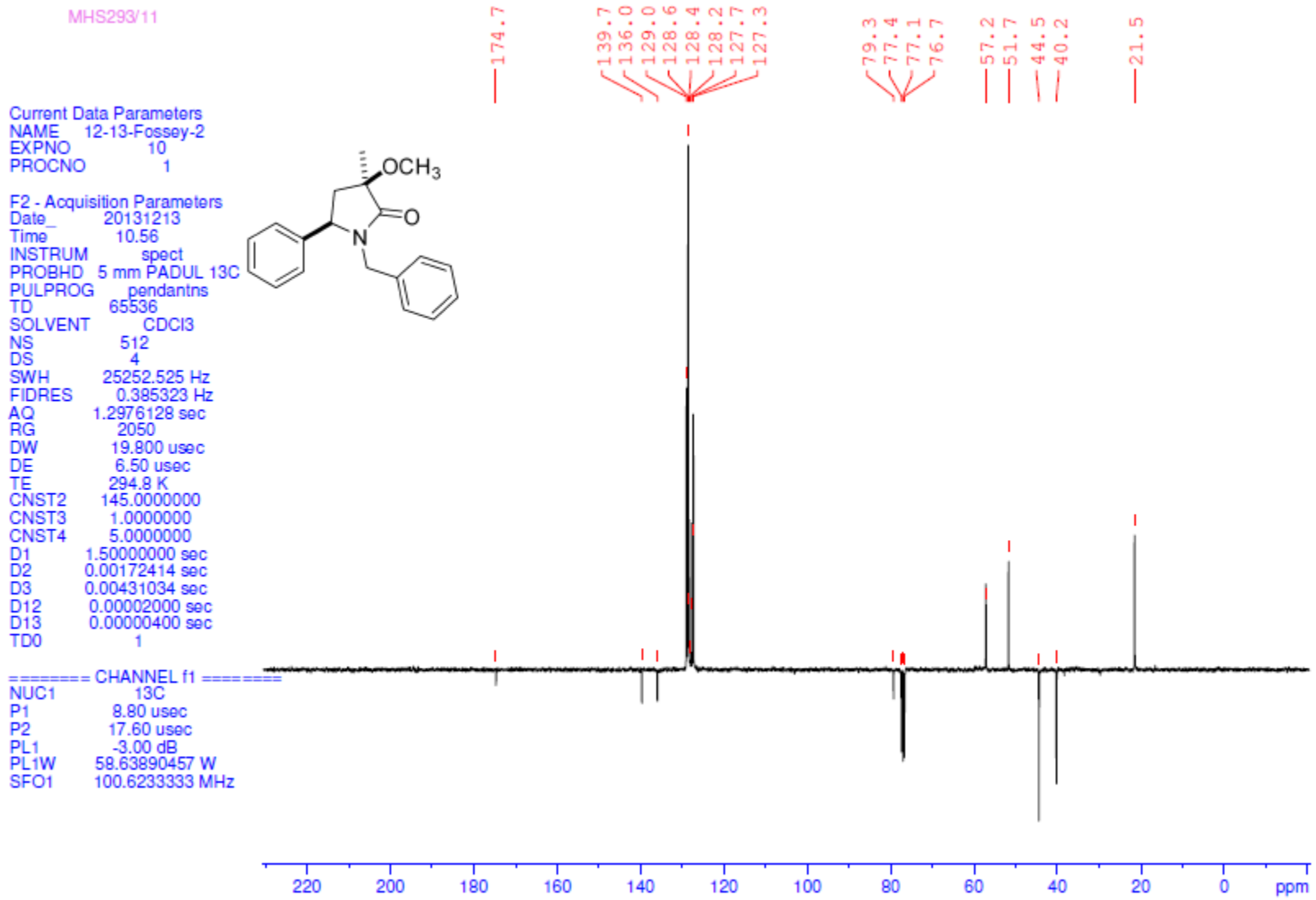
^{13}C NMR Spectra of compound 7a



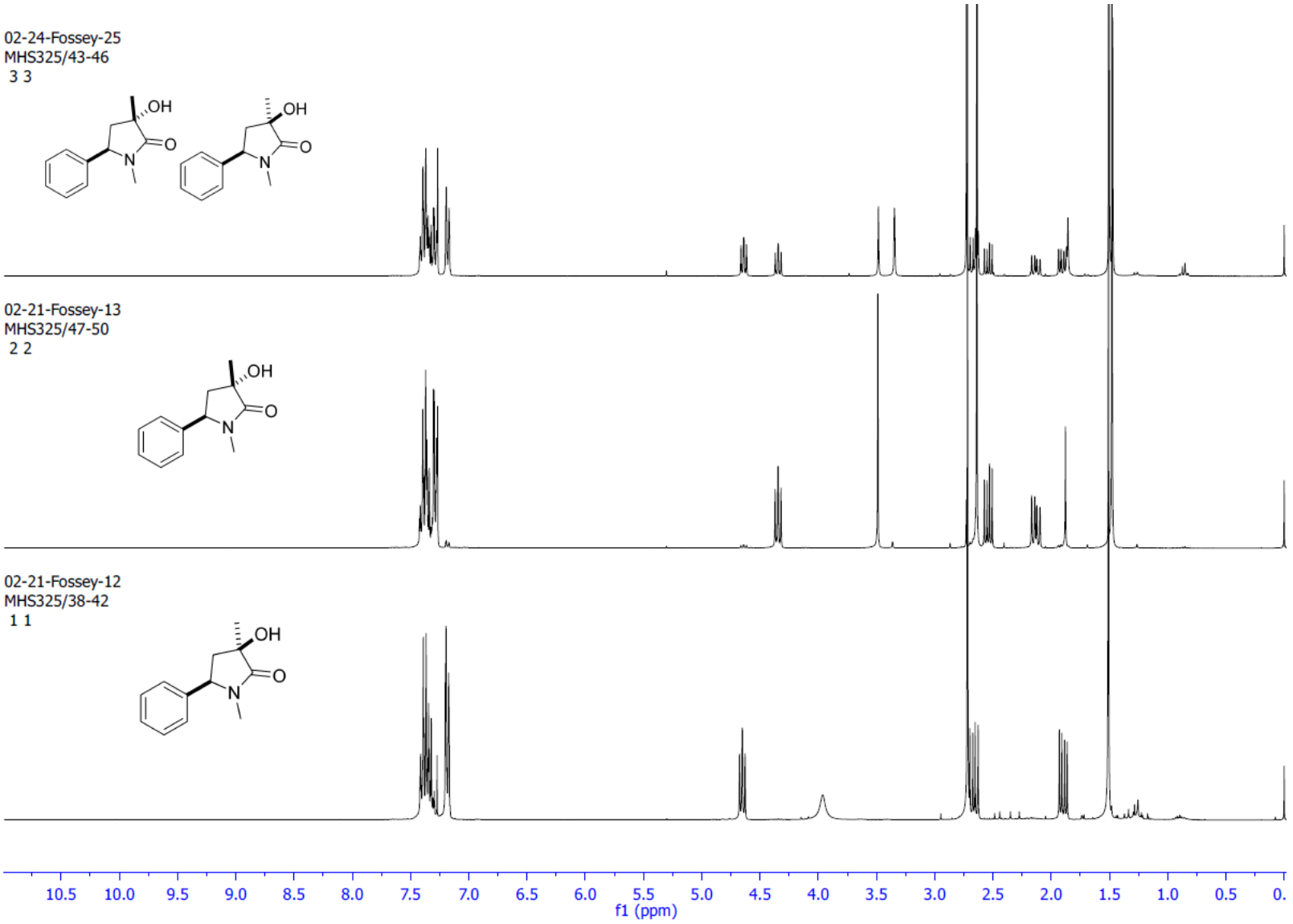
^{1}H NMR Spectra of compound *trans*-7b



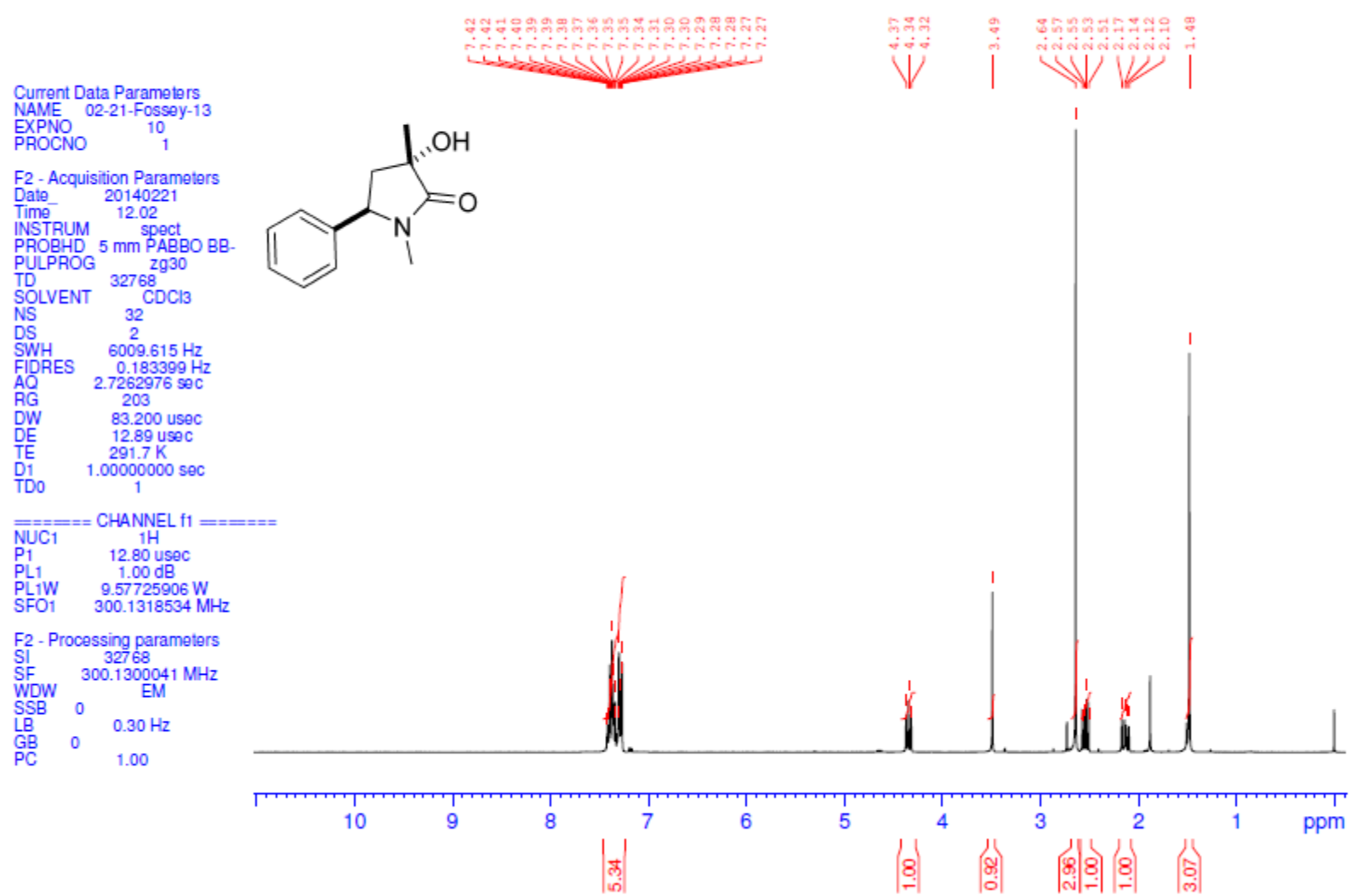
^{13}C NMR Spectra of compound *trans*-7b



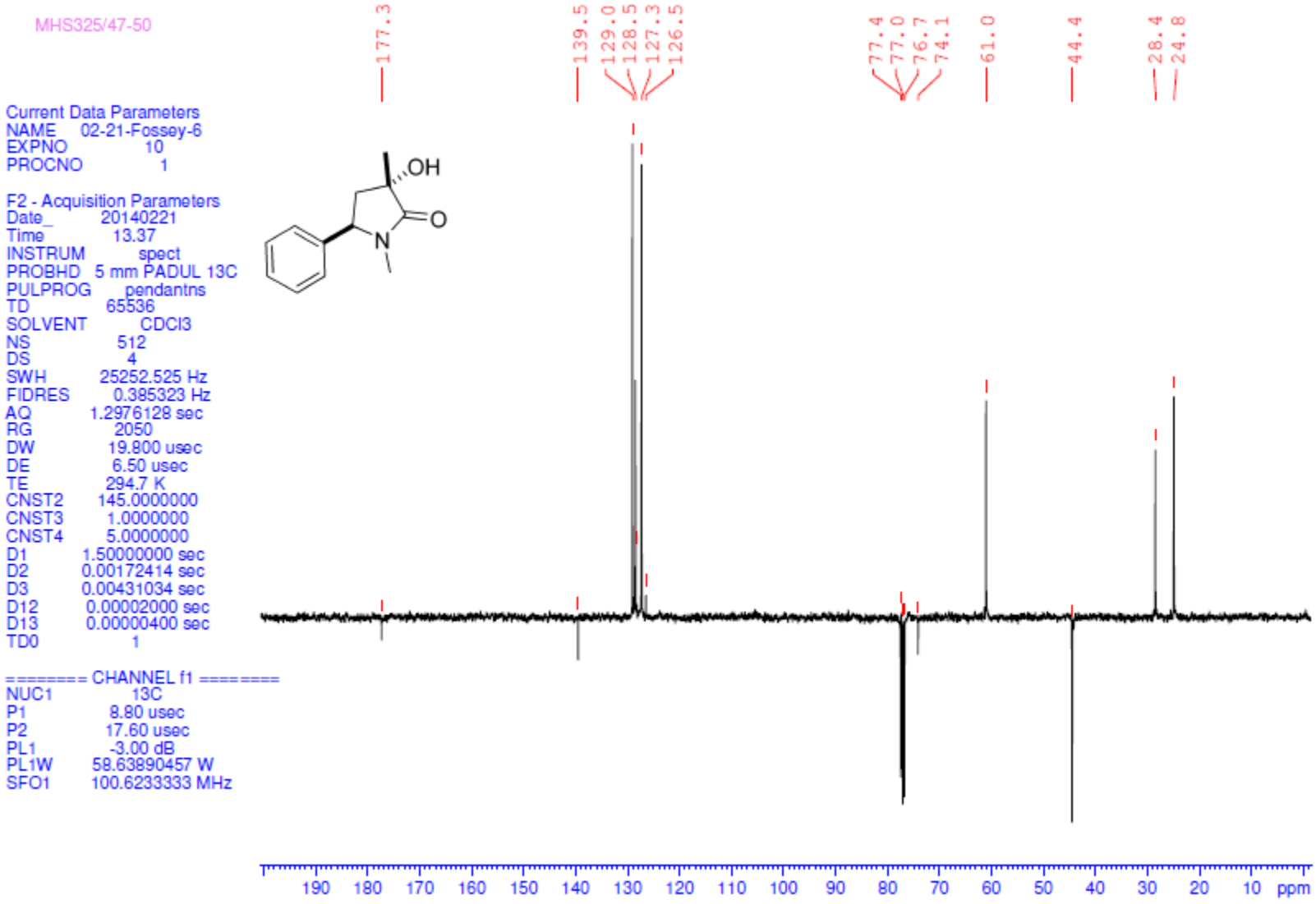
^{1}H NMR Spectrum of compounds *cis*-6o and *trans*-6o mixture of diastereoisomers and the separated diastereoisomers



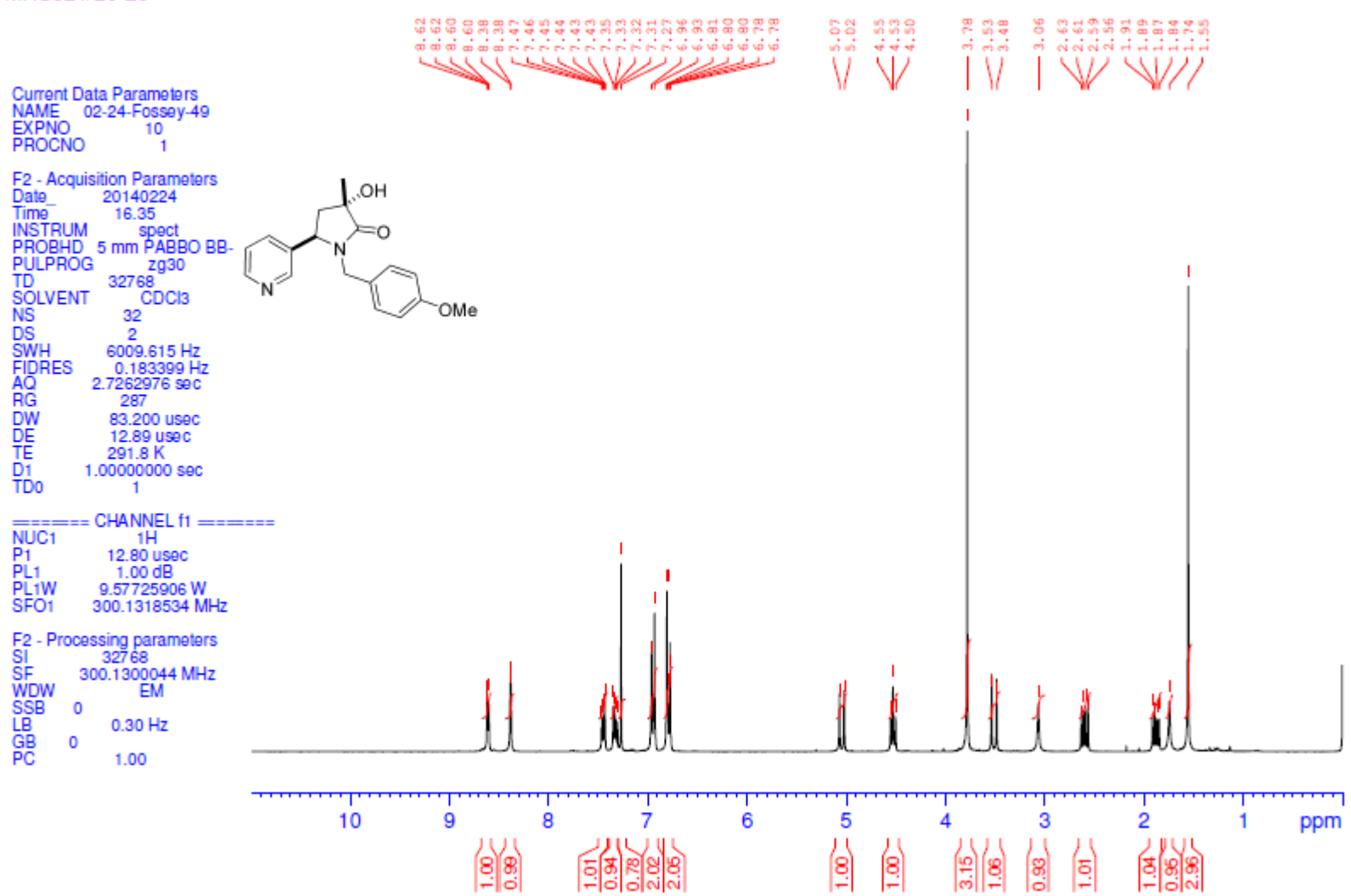
^{1}H NMR Spectra of compound *cis*-6o



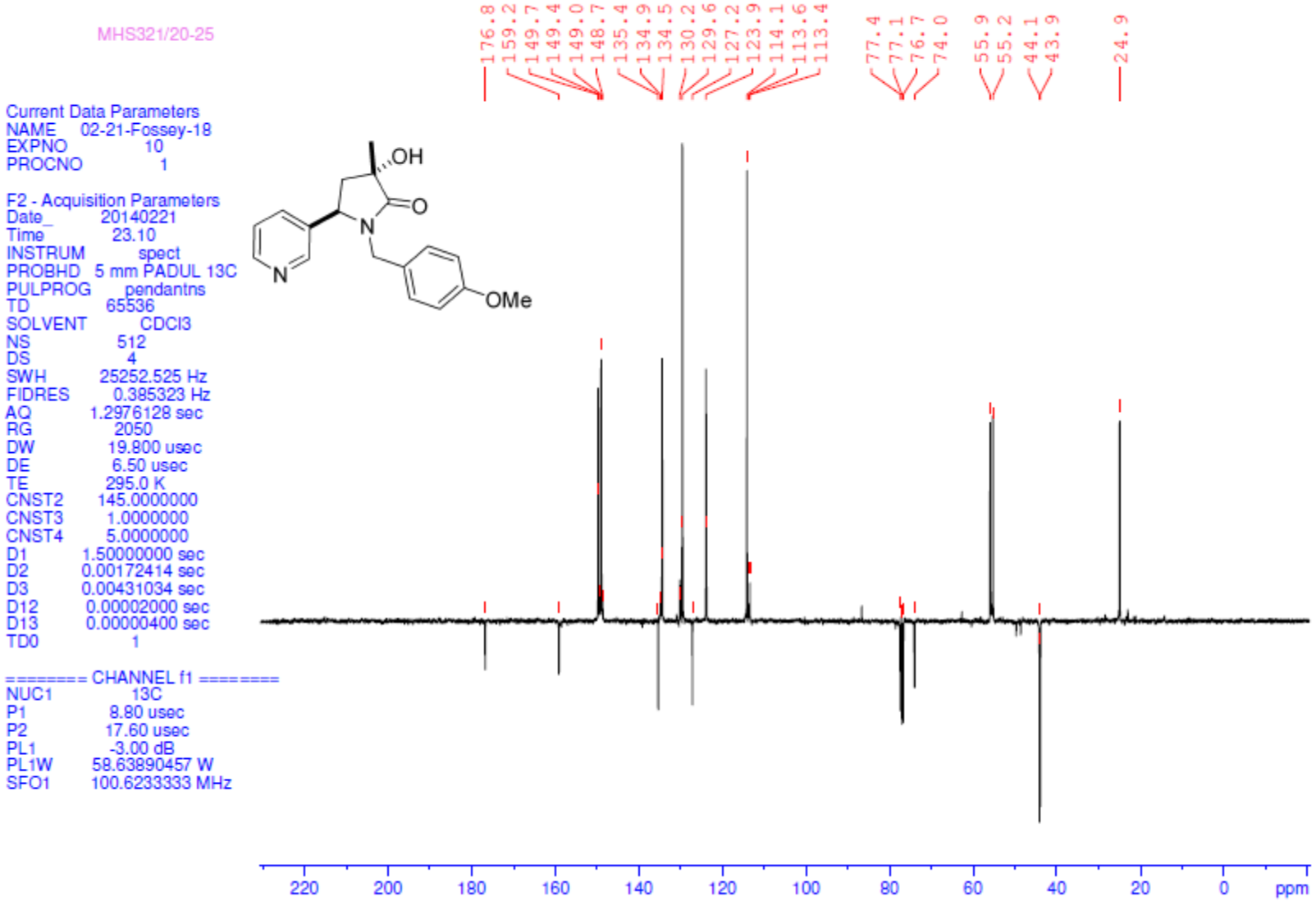
^{13}C NMR Spectra of compound *cis*-6o



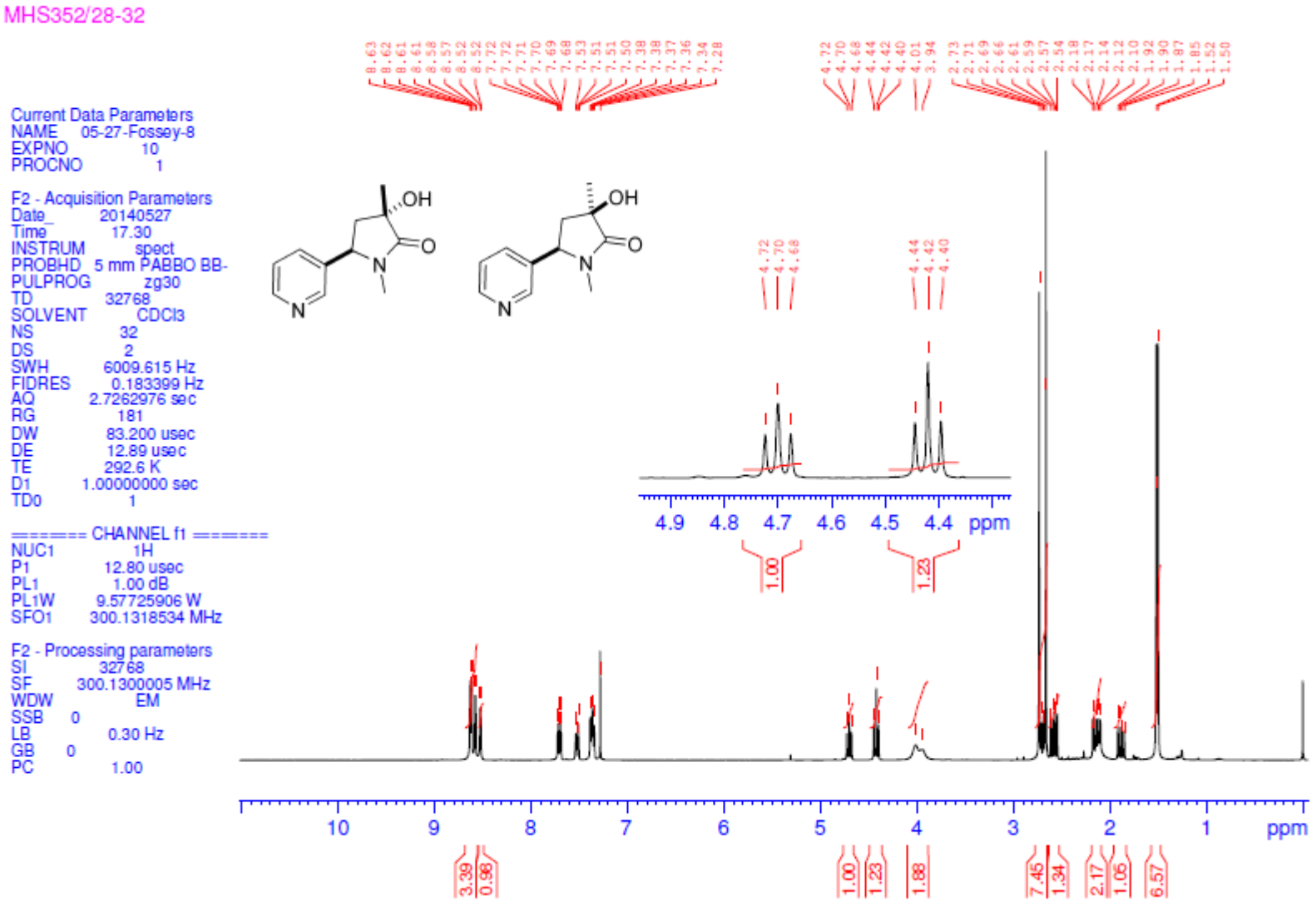
^{1}H NMR Spectra of compound *cis*-6p



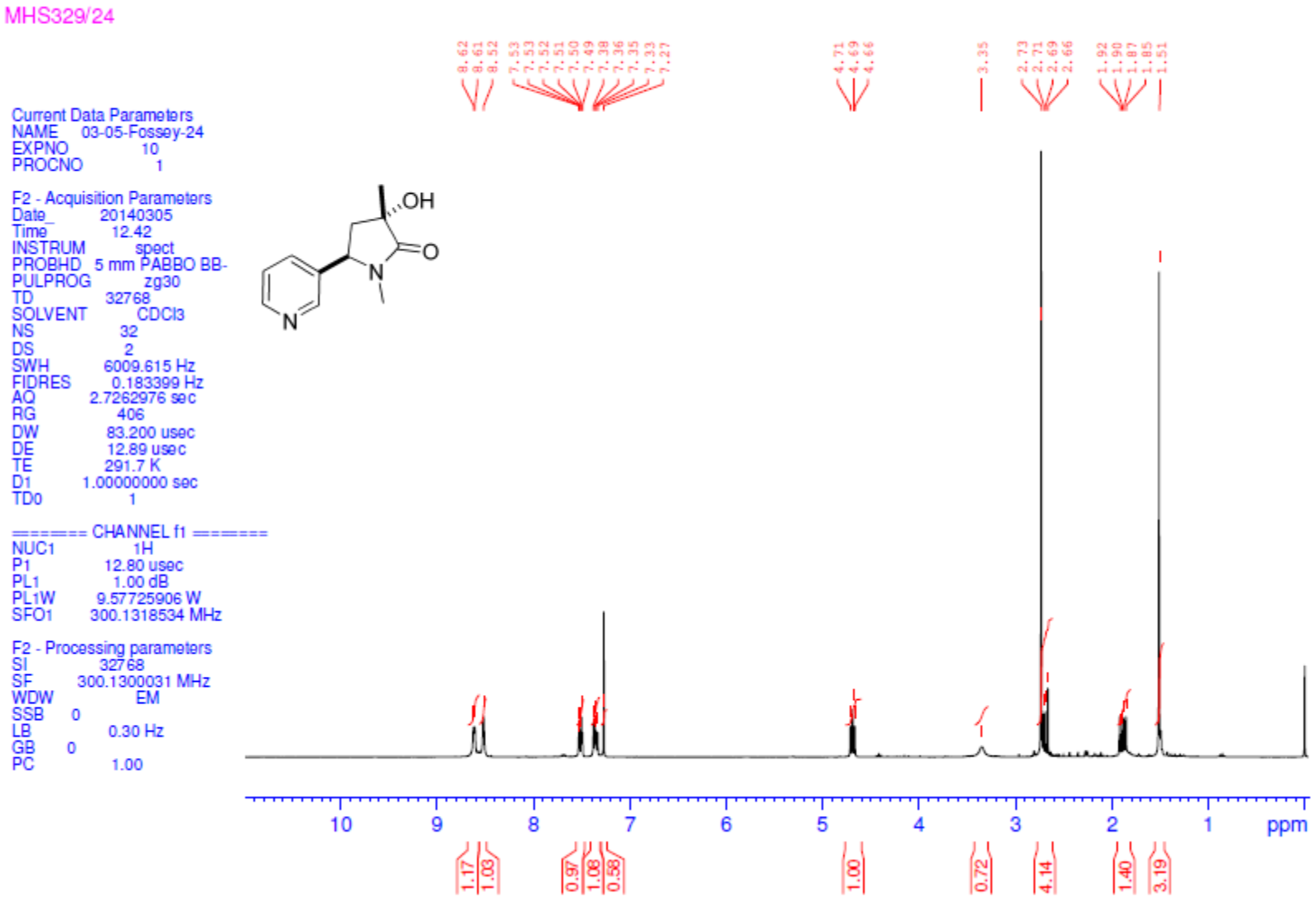
^{13}C NMR Spectra of compound *cis*-6p



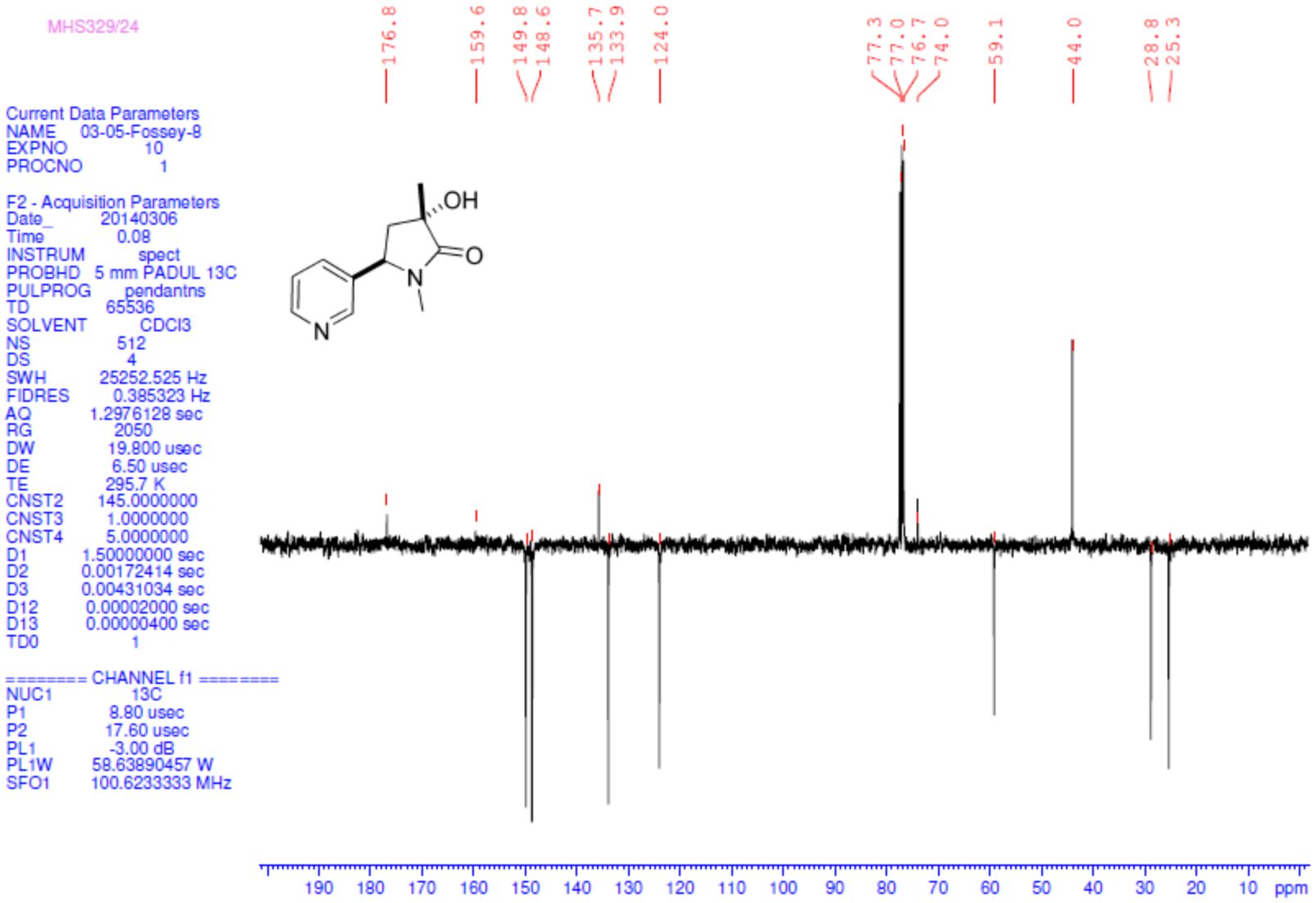
^{1}H NMR Spectra of compounds *cis*-6q and *trans*-6q (mixture of diastereoisomers)



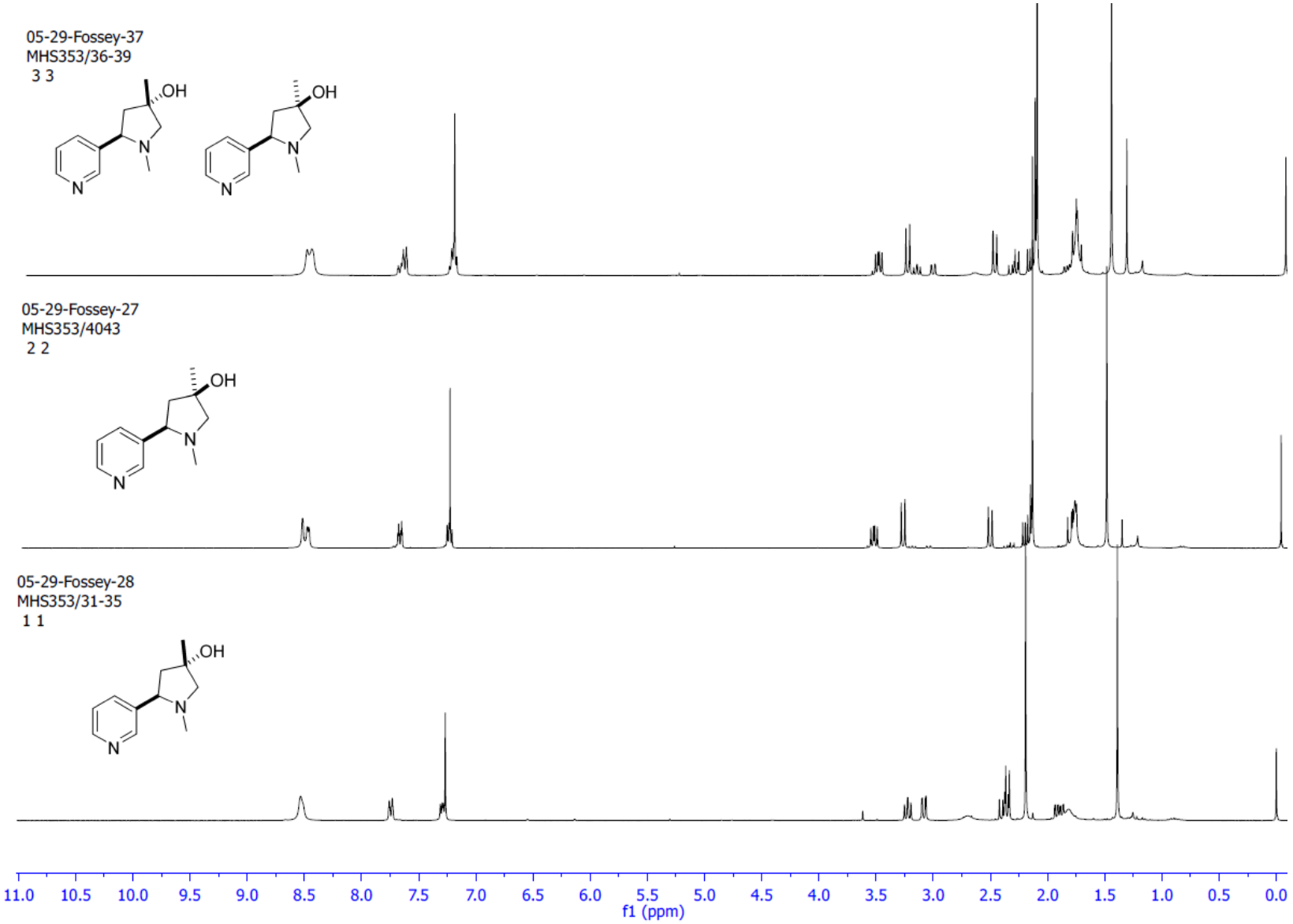
^{1}H NMR Spectra of compound *cis*-6q



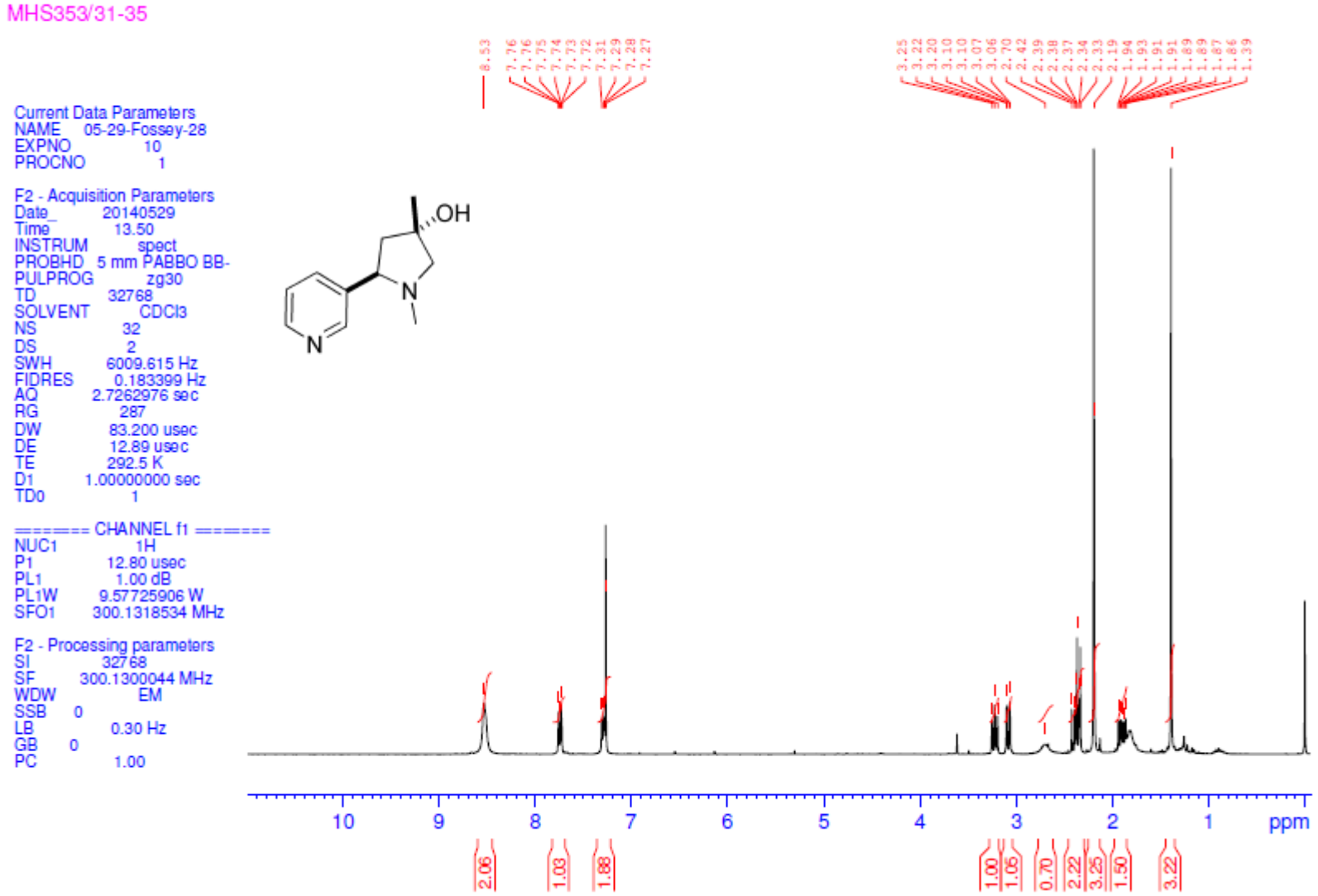
^{13}C NMR Spectra of compound *cis*-6q



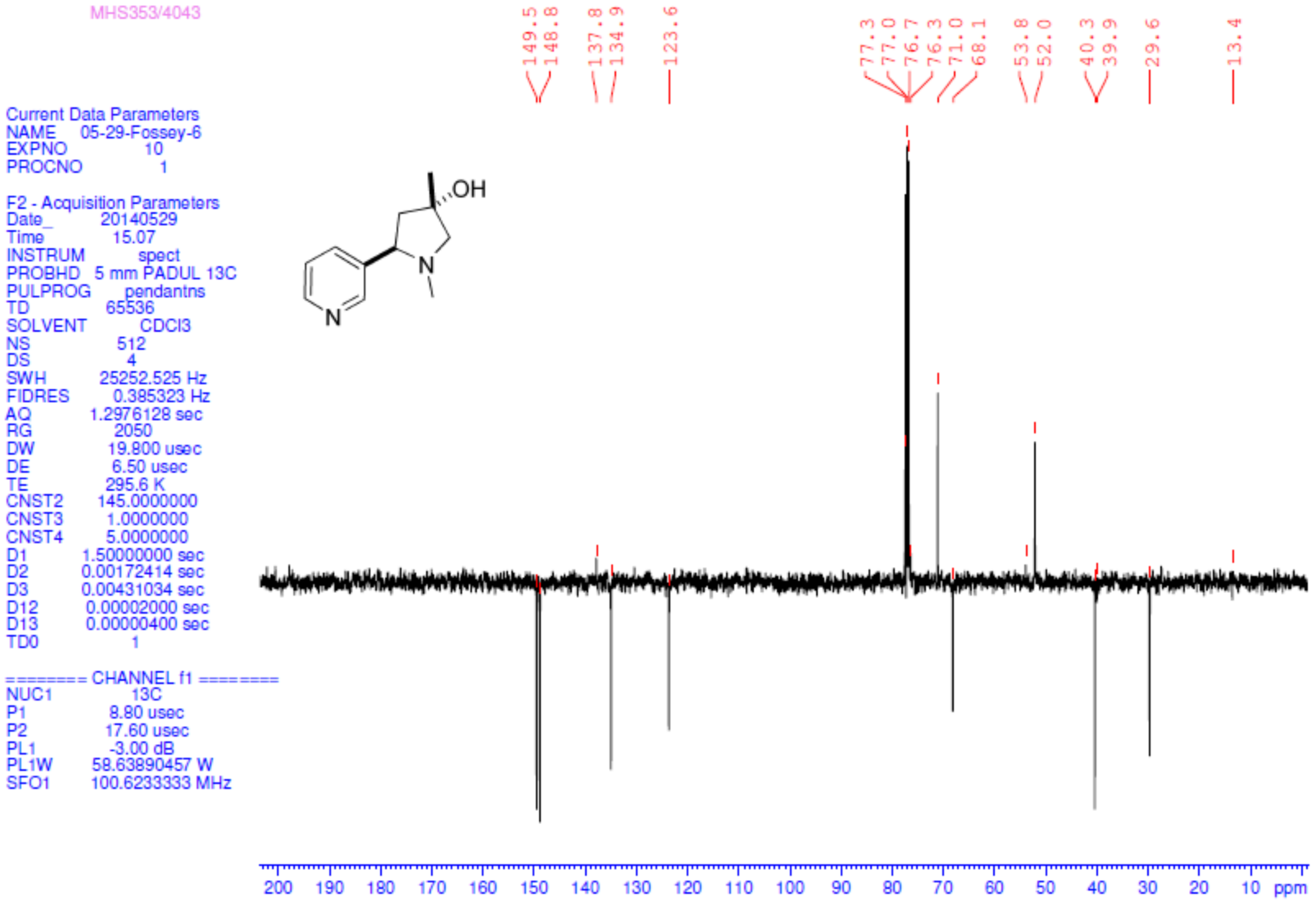
^{1}H NMR Spectrum of compounds *cis*-9b and *trans*-9b mixture of diastereoisomers and the separated diastereoisomers



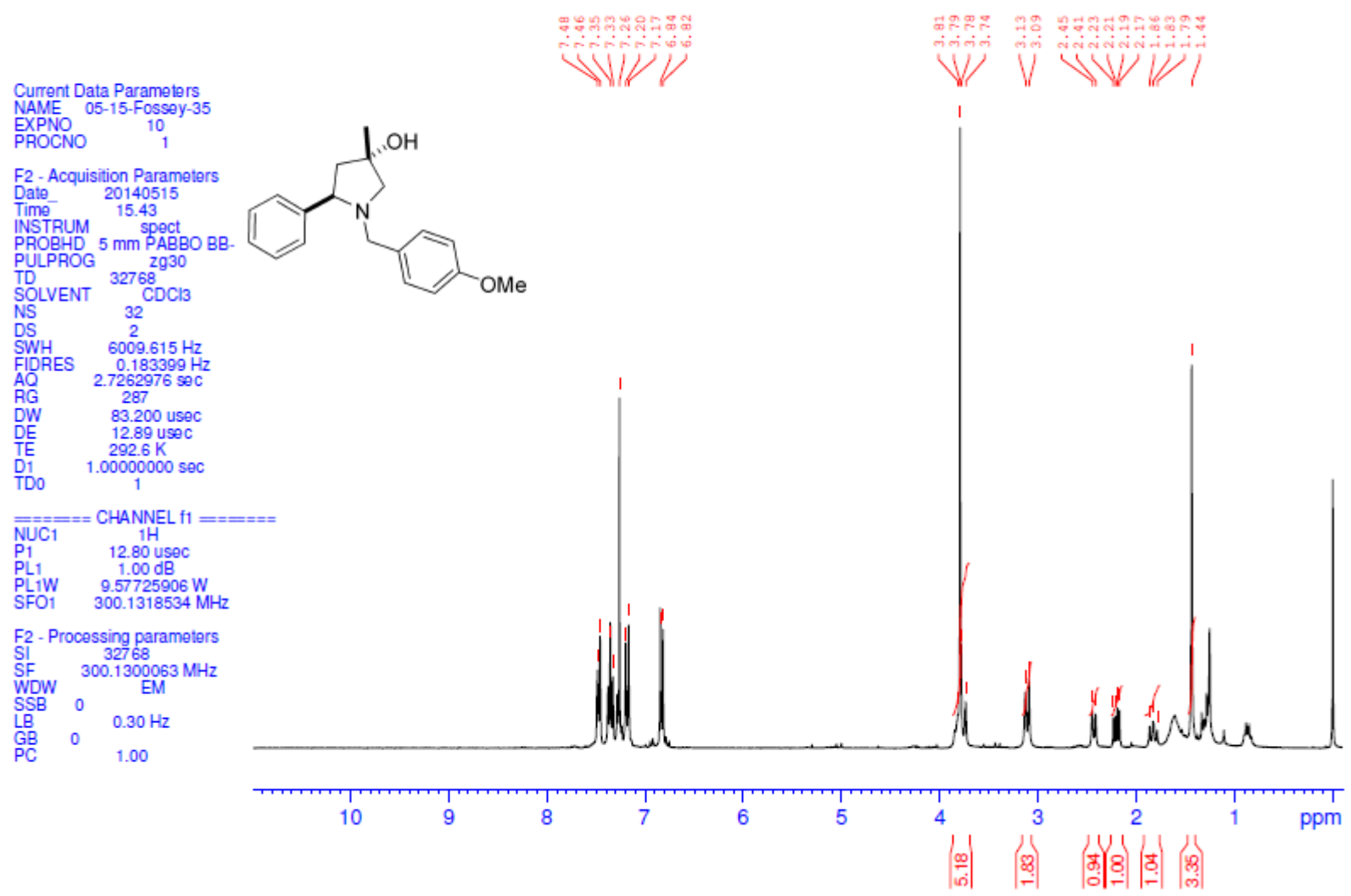
^{1}H NMR Spectra of compound *cis*-9b



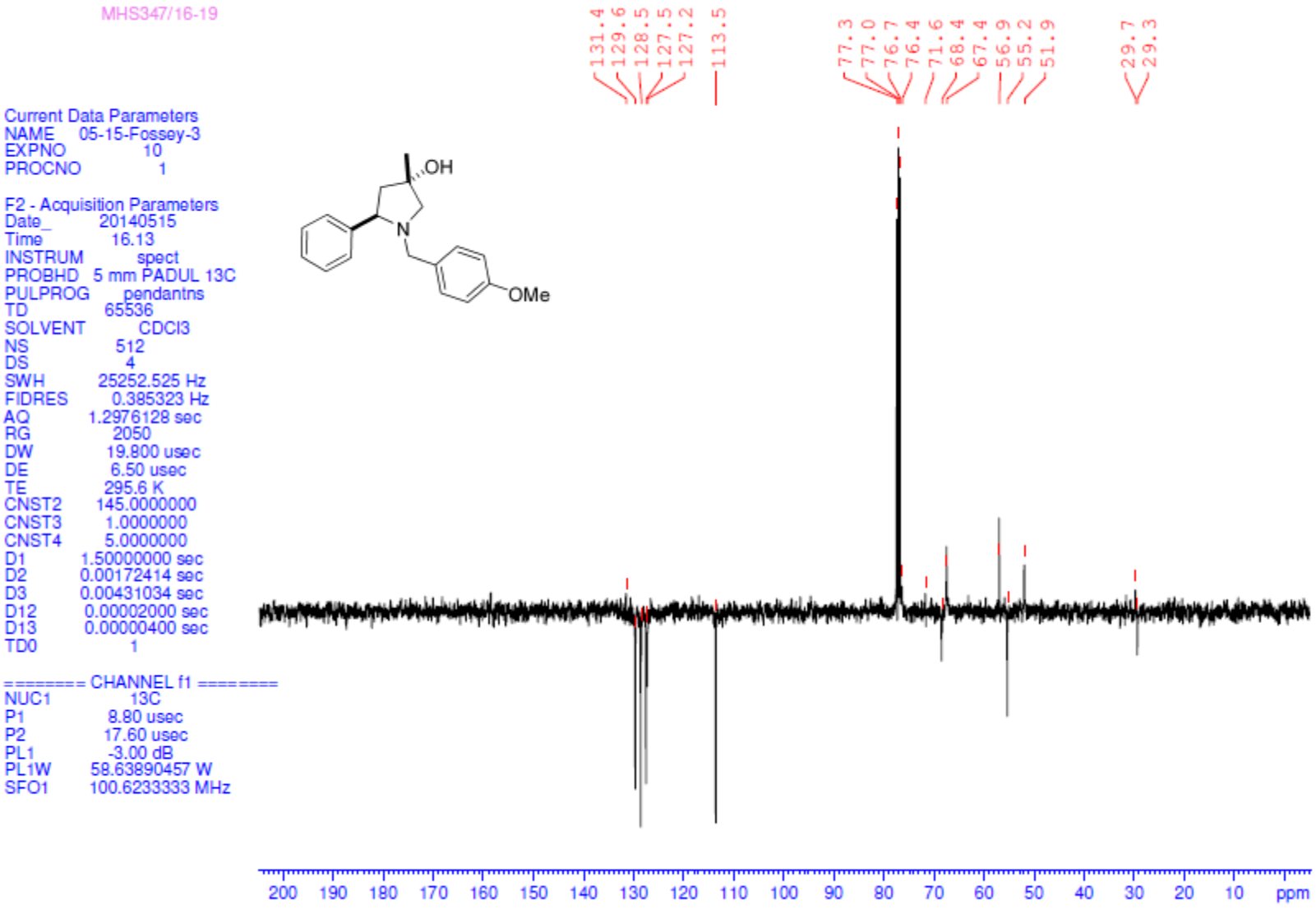
^{13}C NMR Spectra of compound *cis*-9b



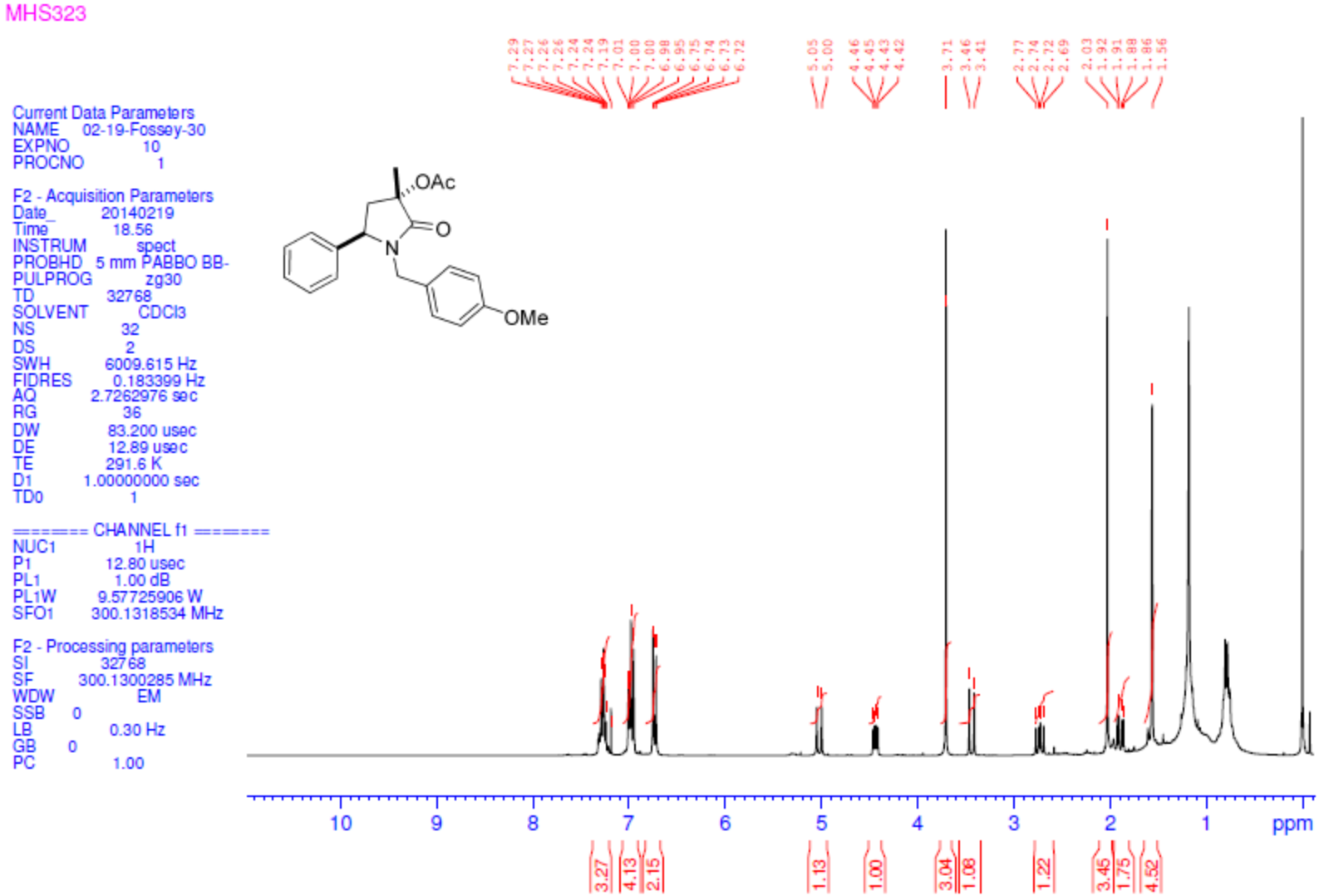
^{1}H NMR Spectra of compound *cis*-9a



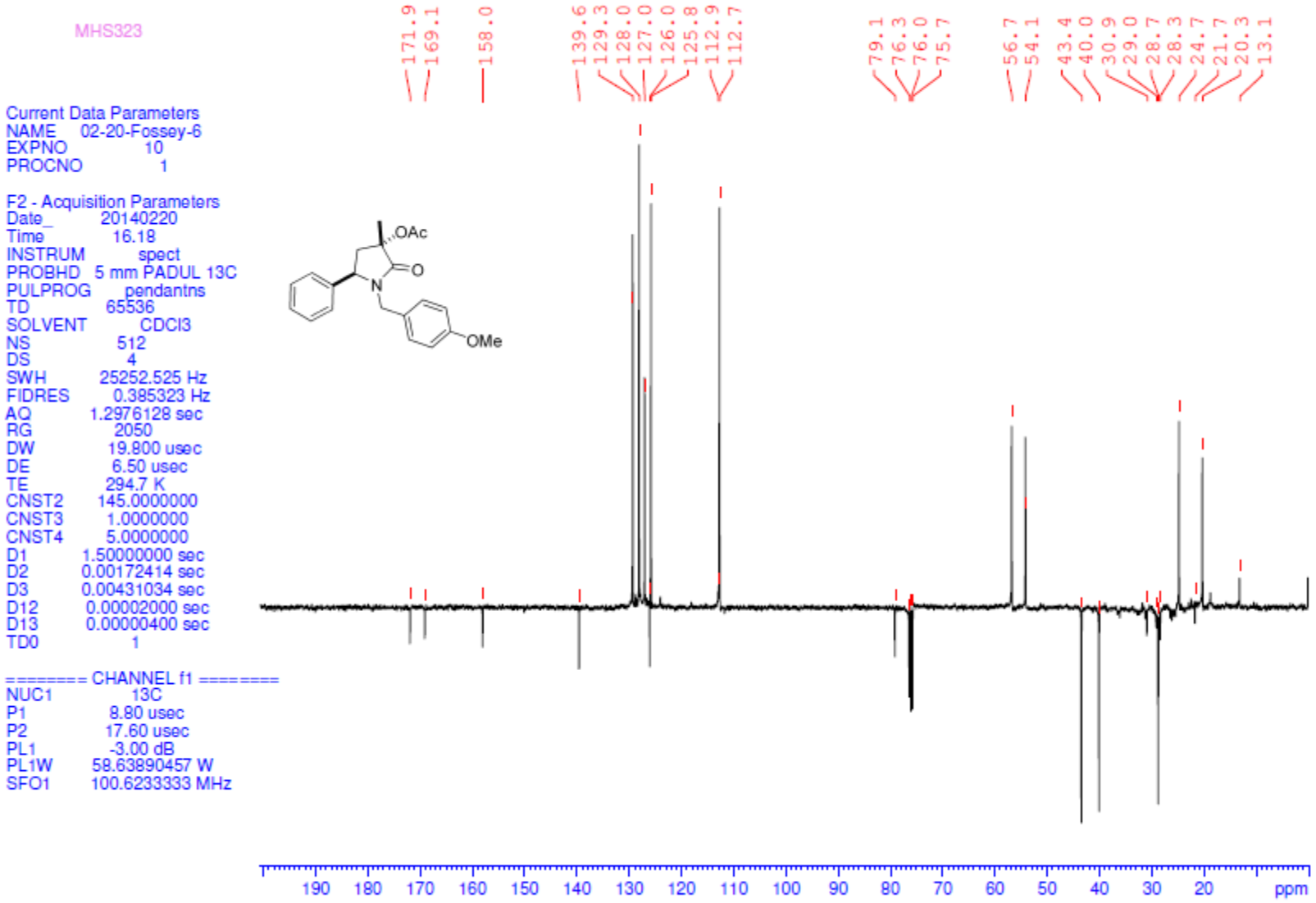
^{13}C NMR Spectra of compound *cis*-9a



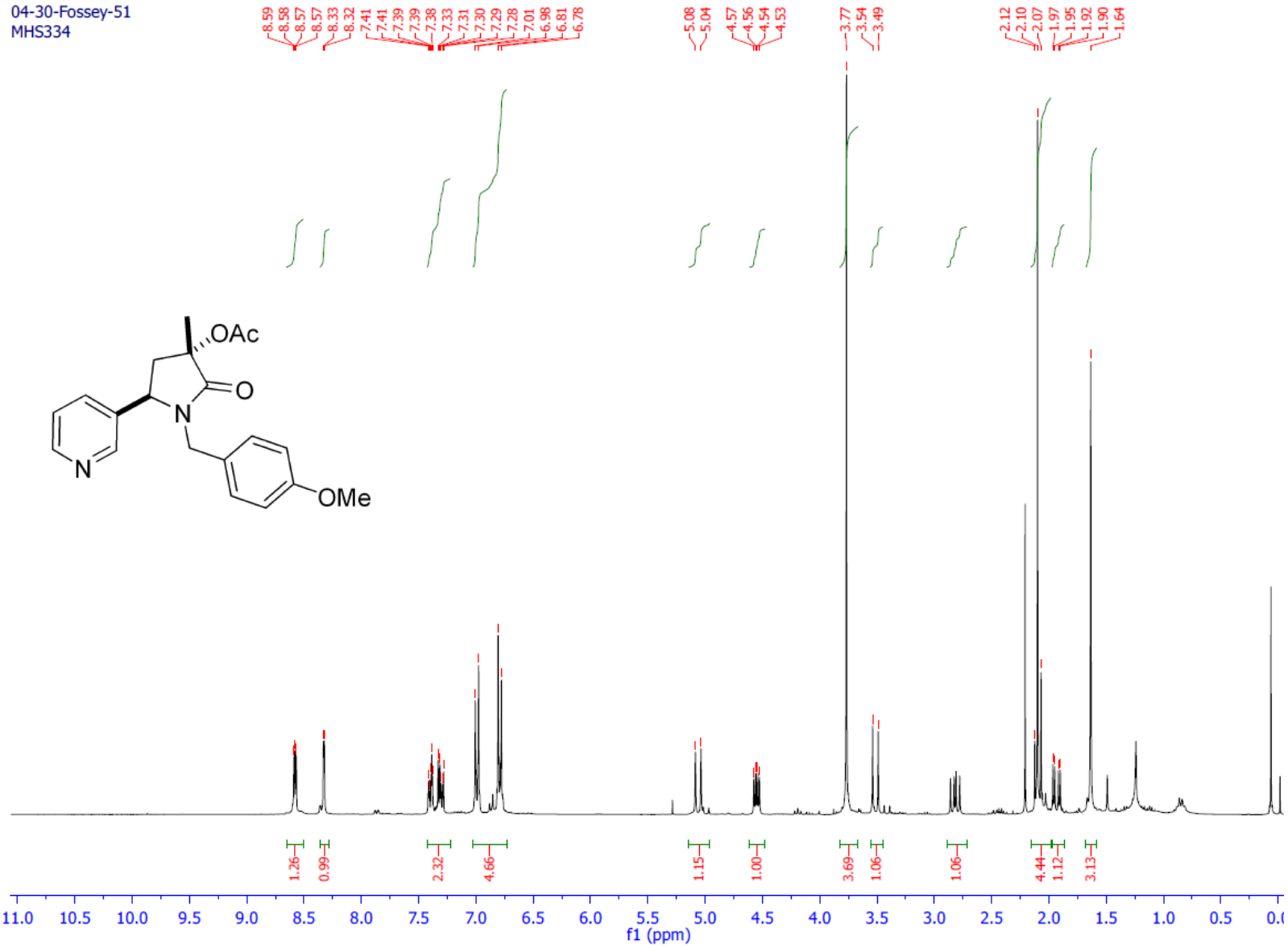
^{1}H NMR Spectra of compound *cis*-8a



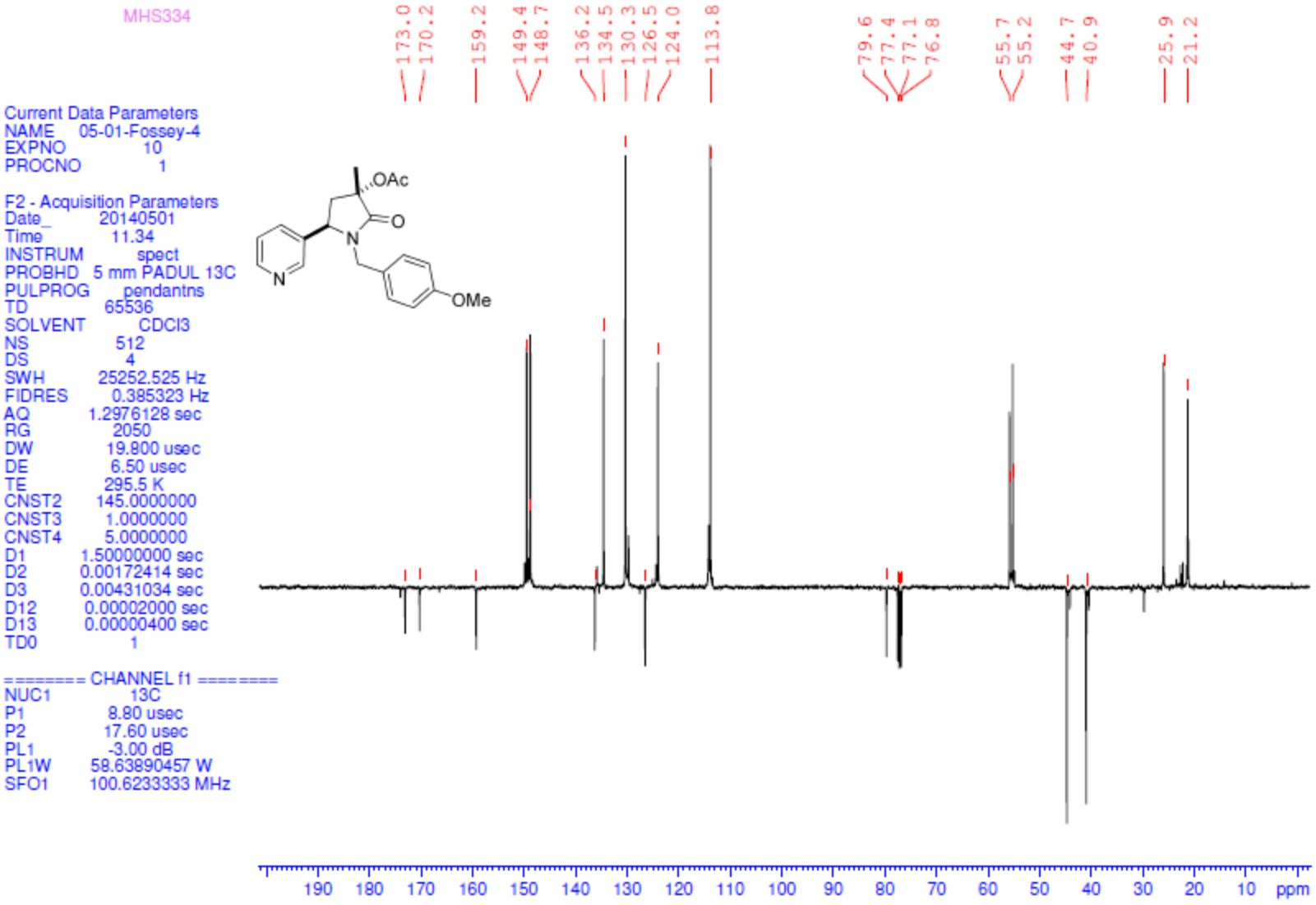
^{13}C NMR Spectra of compound *cis*-8a



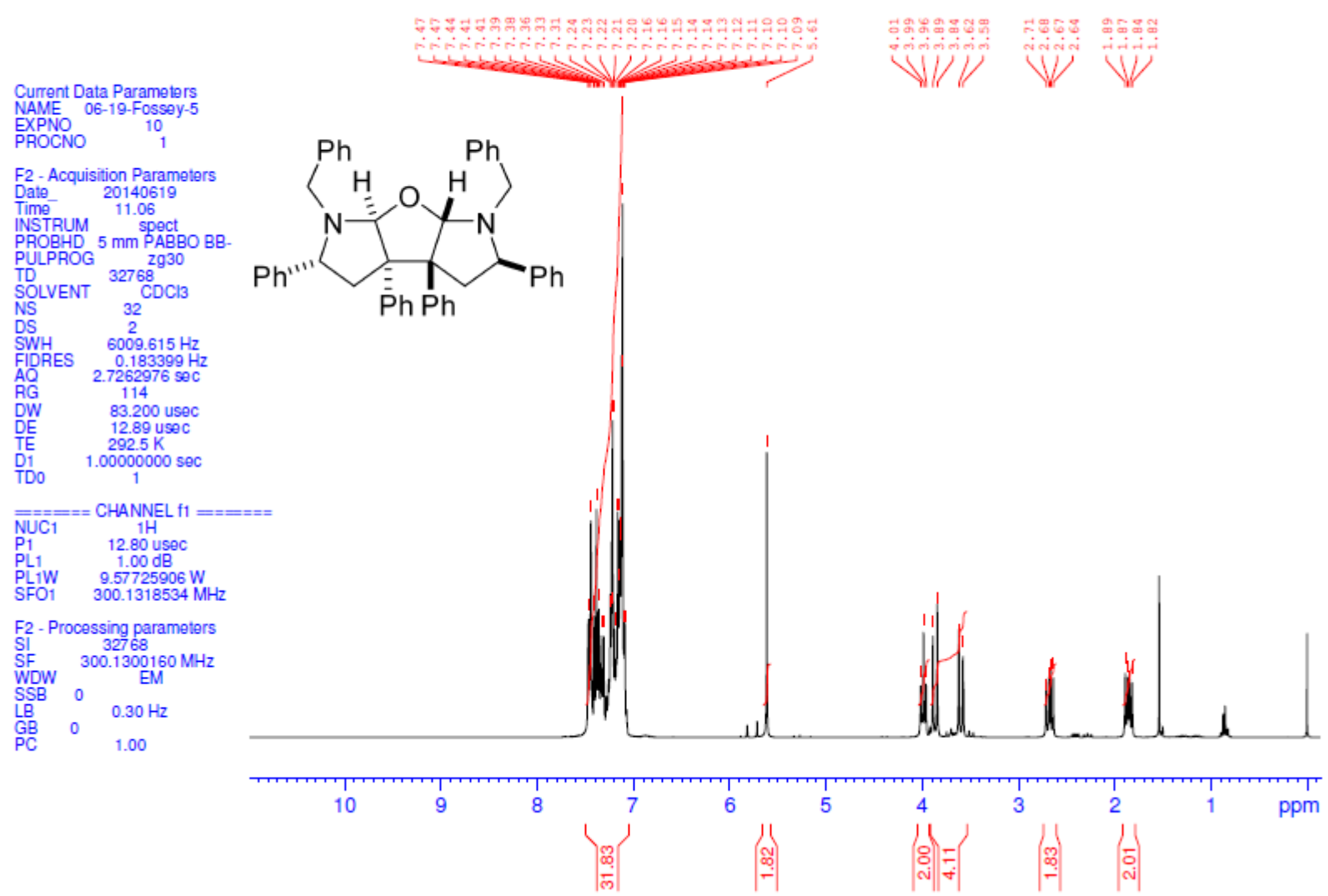
^{1}H NMR Spectra of compound *cis*-8b



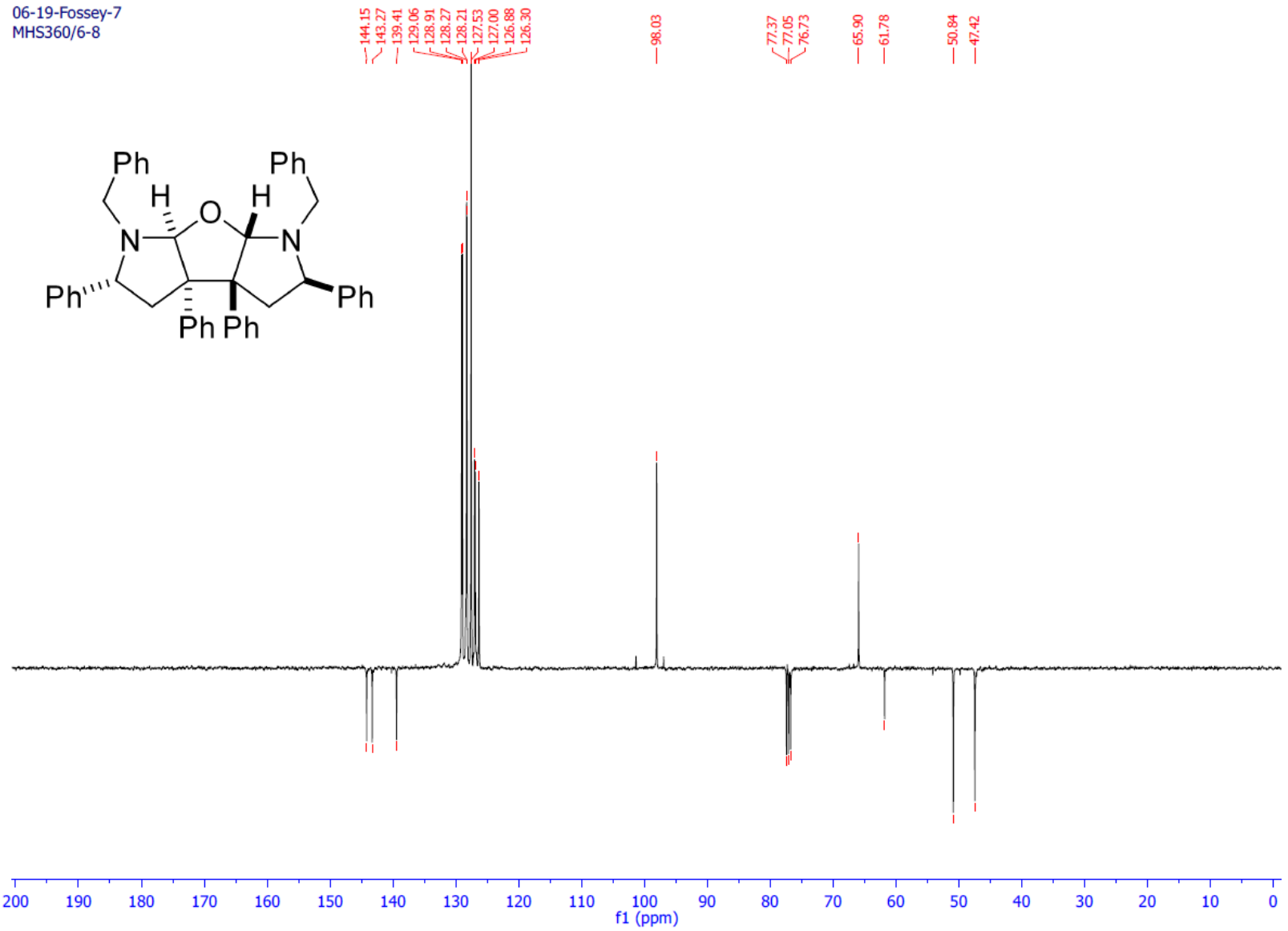
^{13}C NMR Spectra of compound *cis*-8b



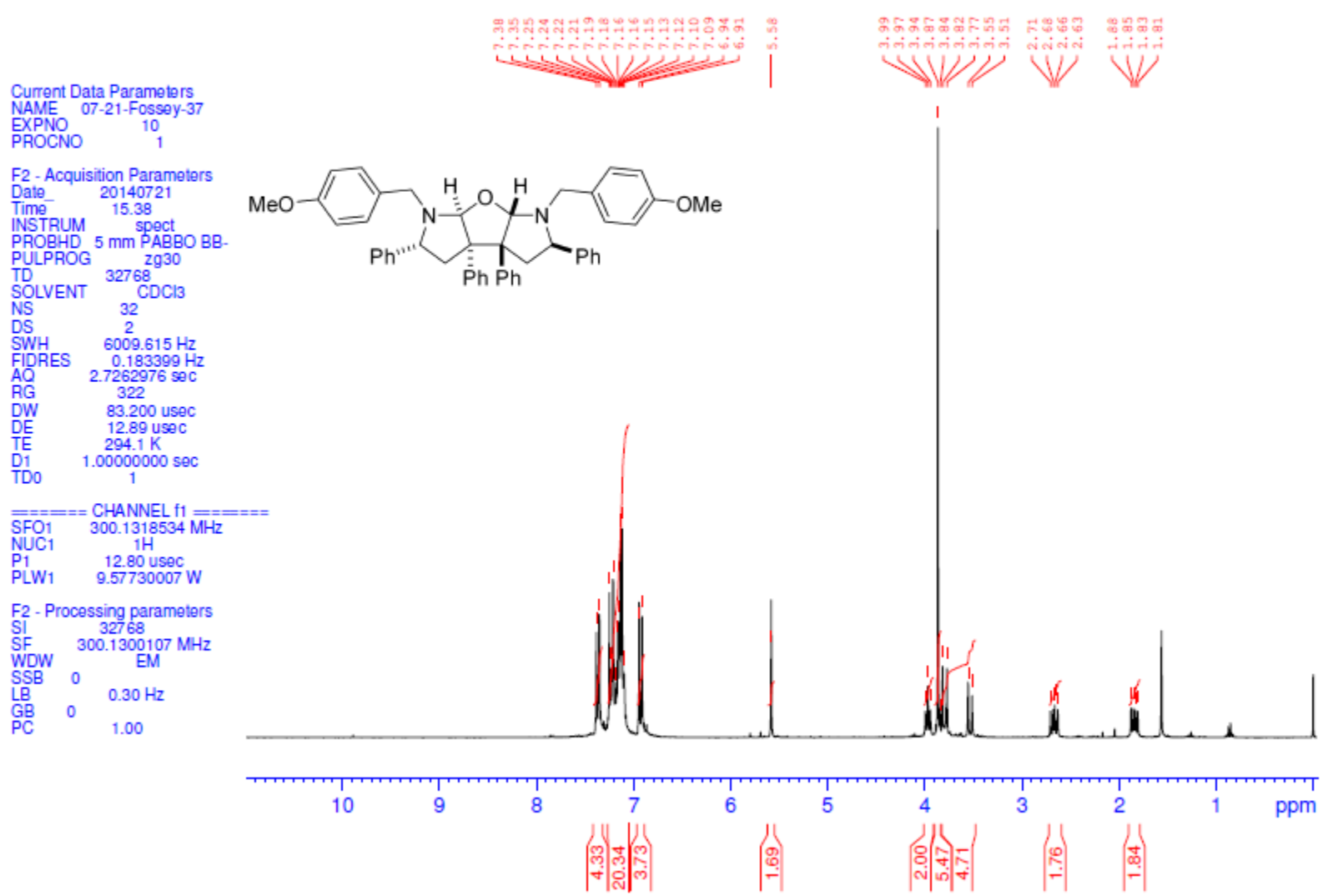
^{1}H NMR Spectra of compound 11a



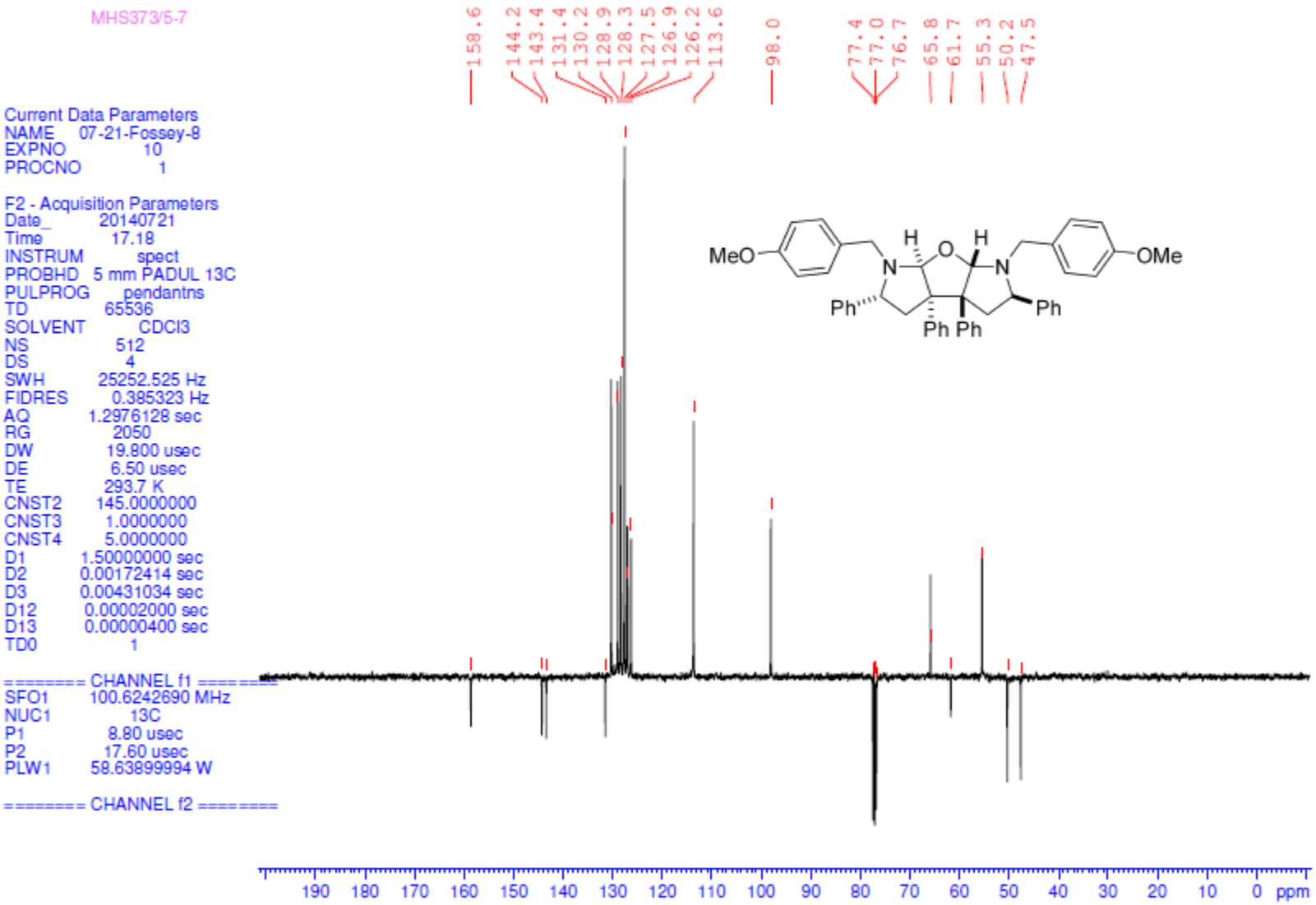
^{13}C NMR Spectra of compound 11a



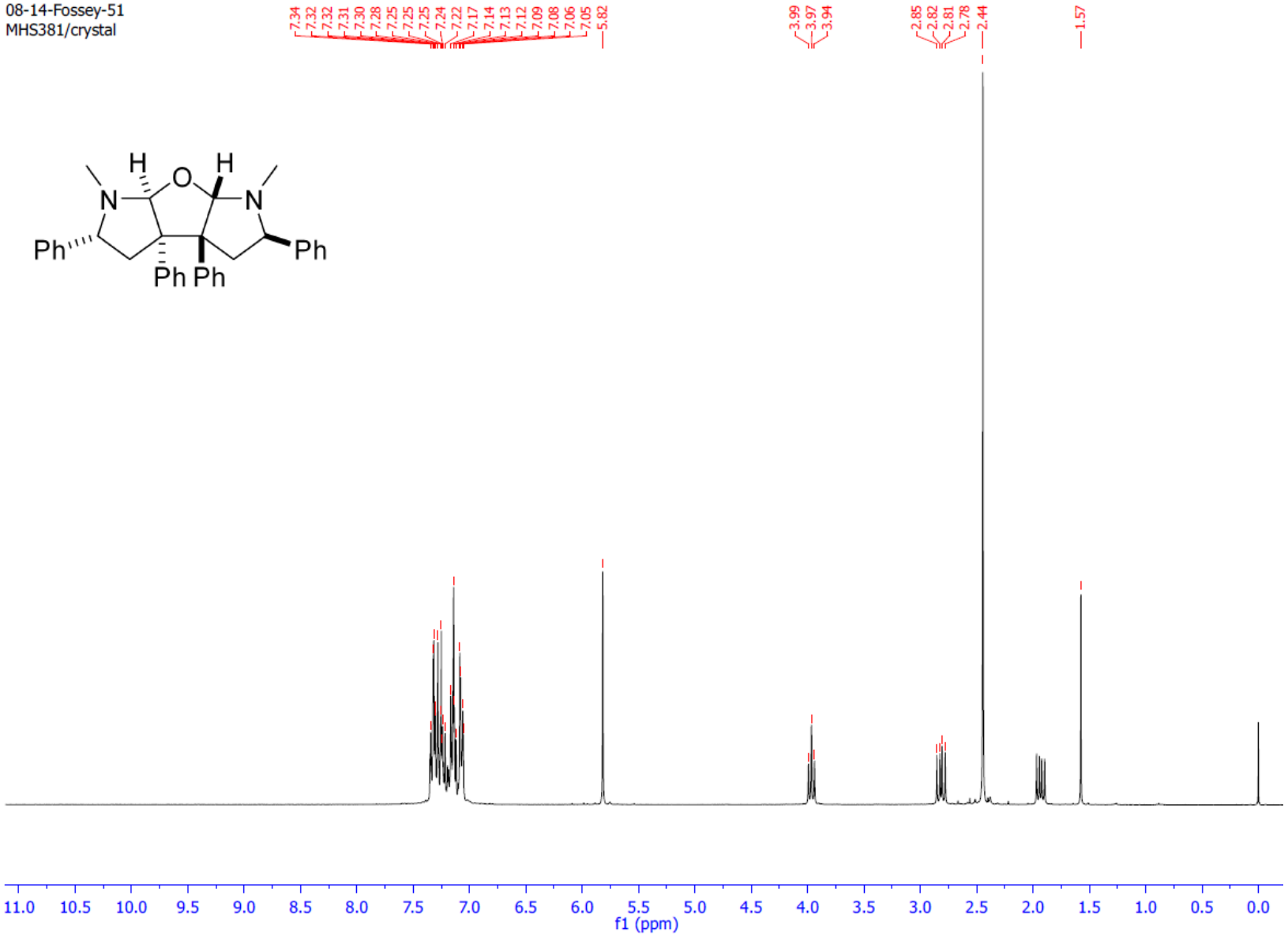
^{1}H NMR Spectra of compound 11b



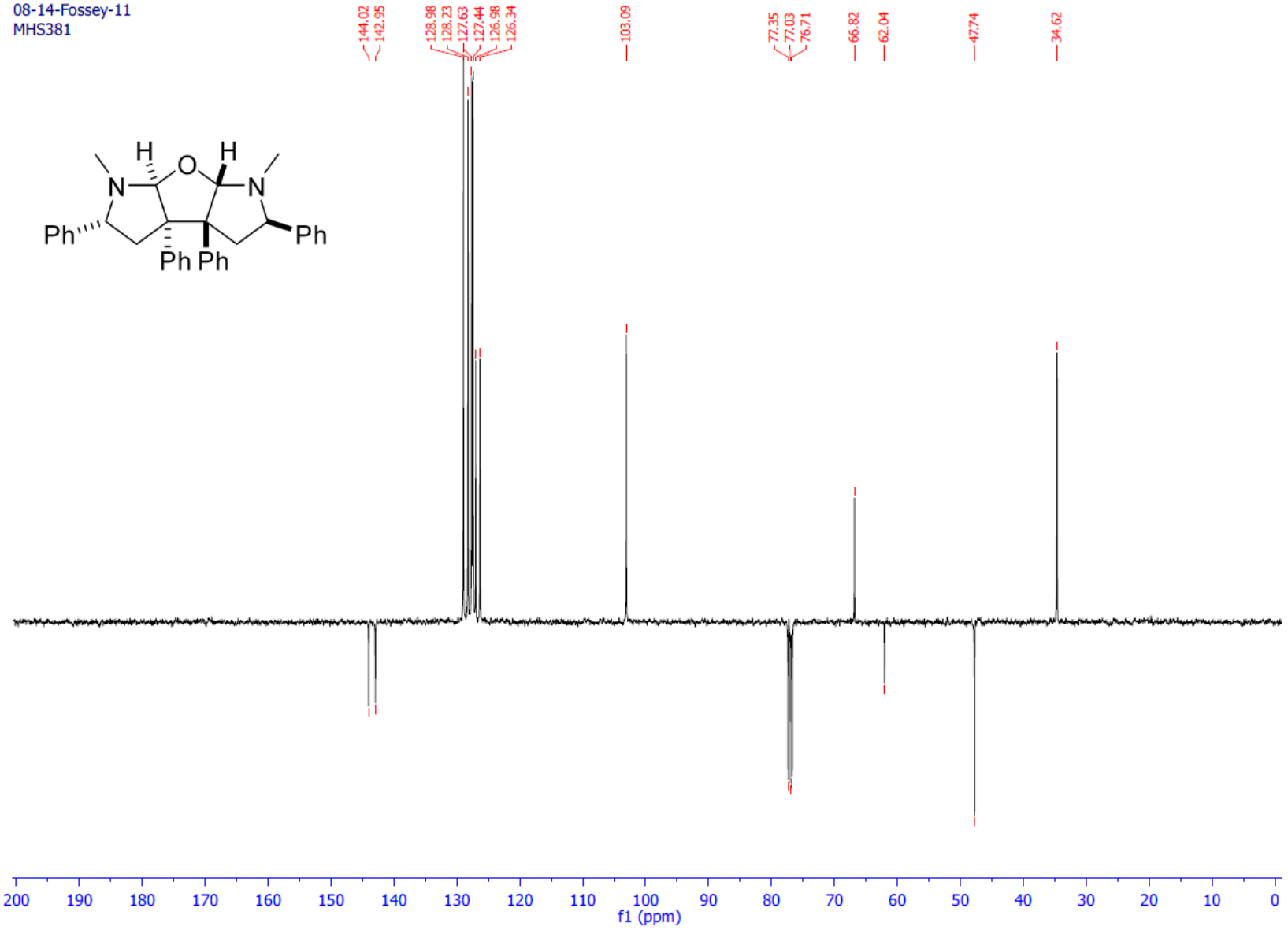
^{13}C NMR Spectra of compound 11b



^{1}H NMR Spectra of compound 11c



^{13}C NMR Spectra of compound 11c



X-Ray Crystallography Data

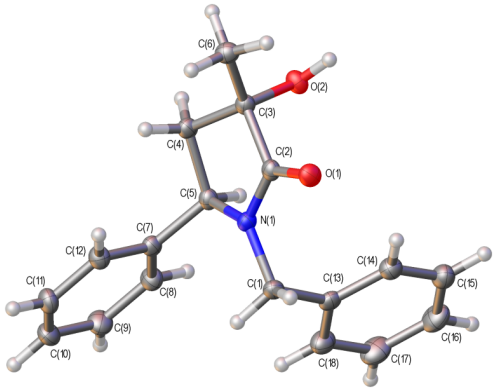


Figure S1: X-ray crystal structure of **6a-*cis*** with ellipsoids drawn at the 50 % probability level.

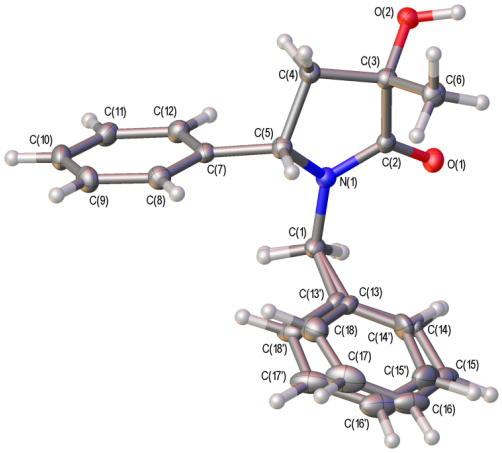


Figure S2: X-ray crystal structure of **6a-*trans*** with ellipsoids drawn at the 50 % probability level. The phenyl group C(13)-C(18)/C(13')-C(18') is disordered over two positions with a refined occupancy ratio of 54(1):46(1).

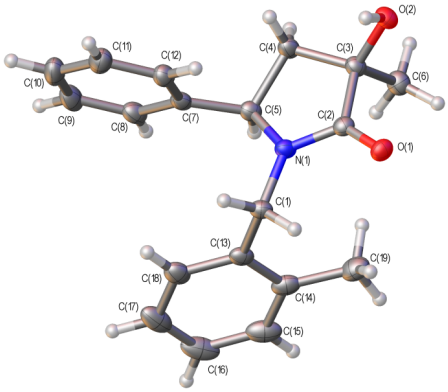


Figure S3: X-ray crystal structure of **6d-*trans*** with ellipsoids drawn at the 50 % probability level.

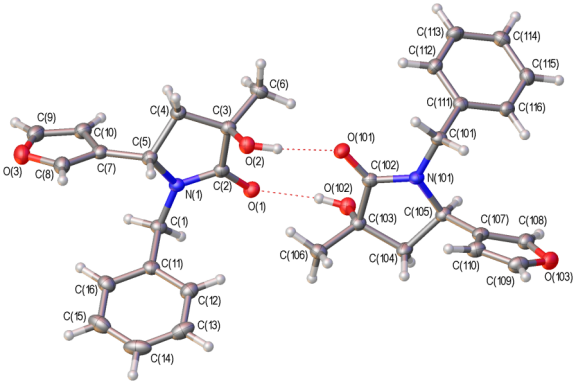


Figure S4: X-ray crystal structure of **6e-*cis*** with ellipsoids drawn at the 50 % probability level. The structure contains two crystallographically-independent molecules in the asymmetric unit.

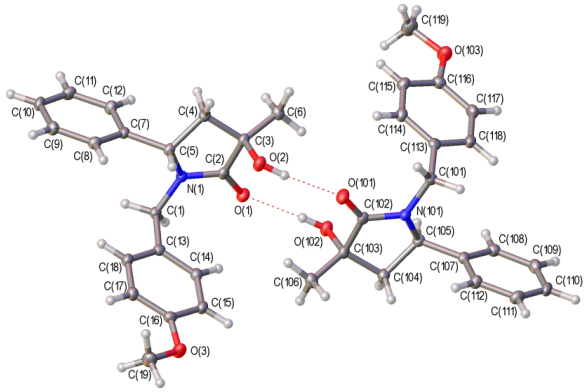


Figure S5: X-ray crystal structure of **6h-*cis*** with ellipsoids drawn at the 50 % probability level. The structure contains two crystallographically-independent molecules in the asymmetric unit.

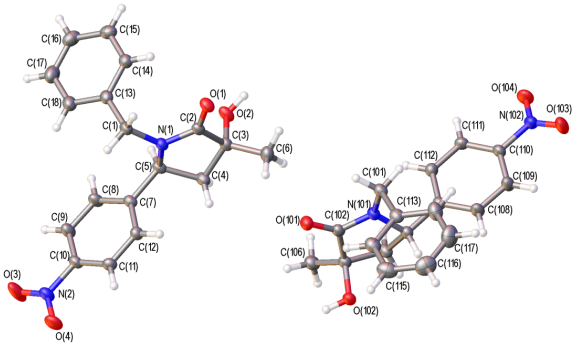


Figure S6: X-ray crystal structure of **6m-*cis*** with ellipsoids drawn at the 50 % probability level. The structure contains two crystallographically-independent molecules in the asymmetric unit.

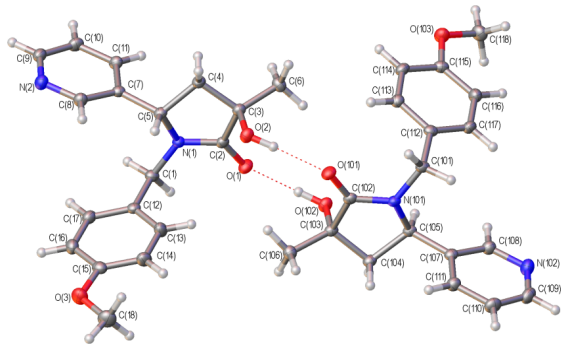


Figure S7: X-ray crystal structure of **6p-*cis***with ellipsoids drawn at the 50 % probability level. The structure contains two crystallographically-independent molecules in the asymmetric unit.

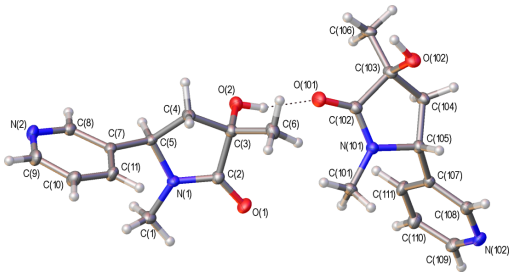


Figure S8: X-ray crystal structure of **6q-*cis*** with ellipsoids drawn at the 50 % probability level. The structure contains two crystallographically-independent molecules in the asymmetric unit.

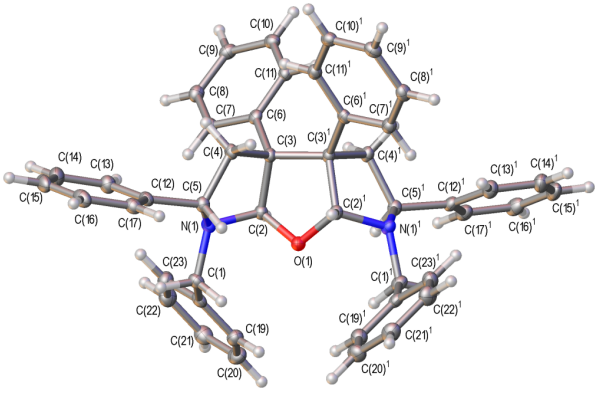


Figure S9: X-ray crystal structure of **11a** with ellipsoids drawn at the 50 % probability level

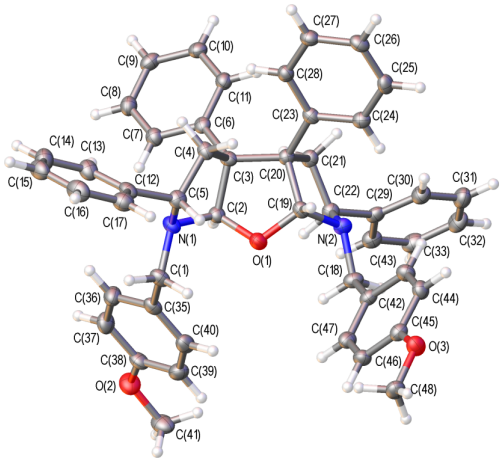


Figure S10: X-ray crystal structure of **11b** with ellipsoids drawn at the 50 % probability level.

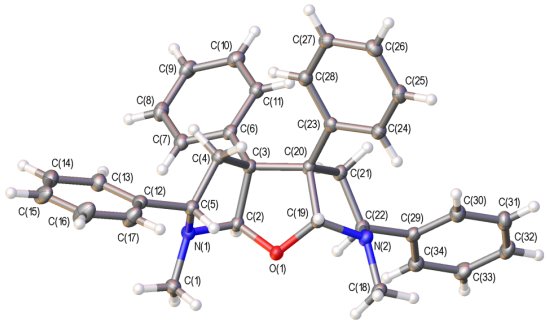


Figure S11: X-ray crystal structure of **11c** with ellipsoids drawn at the 50 % probability level.

Crystal structure determination of 6a-*cis*: $C_{18}H_{19}NO_2$ (*M* =281.34): monoclinic, space group $P2_1/c$ (no. 14), *a* = 7.63367(18) Å, *b* = 29.2305(5) Å, *c* = 6.84862(15) Å, *β* = 102.295(2)°, *V* = 1493.12(6) Å^3, *Z* = 4, *T* = 99.94(19) K, *μ*(CuKα) = 0.646 mm^{-1}, *Dcalc* = 1.252 g/mm^3, 10140 reflections measured (11.866 ≤ 2θ ≤ 148.962), 2899 unique (R_{int} = 0.0248, R_{sigma} = 0.0231) which were used in all calculations. The final R_1 was 0.0367 ($I>2\sigma(I)$) and $wR_2(F_2)$ was 0.0934 (all data).

Crystal structure determination of 6a-*trans*: $C_{18}H_{19}NO_2$ (*M* =281.34): monoclinic, space group *C*2/c (no. 15), *a* = 20.2930(2) Å, *b* = 6.84225(8) Å, *c* = 22.5979(2) Å, *β* = 90.8593(9)°, *V* = 3137.36(5) Å^3, *Z* = 8, *T* = 99.94(17) K, *μ*(CuKα) = 0.615 mm^{-1}, *Dcalc* = 1.191 g/mm^3, 28439 reflections measured (7.826 ≤ 2θ ≤ 149.016), 3181 unique (R_{int} = 0.0244, R_{sigma} = 0.0109) which were used in all calculations. The final R_1 was 0.0343 ($I>2\sigma(I)$) and $wR_2(F_2)$ was 0.1396 (all data).

Crystal structure determination of 6d-*trans*: $C_{19}H_{21}NO_2$ (M =295.37): orthorhombic, space group Pbca (no. 61), a = 12.4972(2) Å, b = 14.1613(2) Å, c = 17.5538(3) Å, V = 3106.62(9) Å^3, Z = 8, T = 100.01(10) K, μ(CuKα) = 0.645 mm^{-1}, Dcalc = 1.263 g/cm^3, 15485 reflections measured (10.078° ≤ 2θ ≤ 148.89°), 3132 unique (R_{int} = 0.0249, R_{sigma} = 0.0184) which were used in all calculations. The final R_1 was 0.0379 (I>2σ(I)) and $wR_2(F_2)$ was 0.0966 (all data).

Crystal structure determination of 6e-*cis*: $C_{16}H_{17}NO_3$ (*M* =271.30): orthorhombic, space group $P2_12_12_1$ (no. 19), *a* = 6.86998(8) Å, *b* = 15.21957(20) Å, *c* = 27.0563(4) Å, *V* = 2828.95(6) Å^3, *Z* = 8, *T* = 100.00(10) K, *μ*(CuKα) = 0.717 mm^{-1}, *Dcalc* = 1.274 g/mm^3, 15654 reflections measured (6.534 ≤ 2θ ≤ 148.936), 5473 unique (R_{int} = 0.0239, R_{sigma} = 0.0248) which were used in all calculations. The final R_1 was 0.0289 ($I>2\sigma(I)$) and $wR_2(F_2)$ was 0.0703 (all data).

Crystal structure determination of 6h-*cis*: $C_{19}H_{21}NO_3$ (*M* =311.37): triclinic, space group *P*-1 (no. 2), *a* = 6.98428(14) Å, *b* = 15.3845(4) Å, *c* = 16.6168(4) Å, *α* = 110.497(2)°, *β* = 97.0023(18)°, *γ* = 102.2736(19)°, *V* = 1596.16(7) Å^3, *Z* = 4, *T* = 100.00(10) K, *μ*(CuKα) = 0.703 mm^{-1}, *Dcalc* = 1.296 g/mm^3, 30499 reflections measured (5.814 ≤ 2θ ≤ 149.132), 6446 unique (R_{int} = 0.0314, R_{sigma} = 0.0265) which were used in all calculations. The final R_1 was 0.0420 ($I>2\sigma(I)$)and$wR_2(F_2)$ was 0.1611 (all data).

Crystal structure determination of 6m-*cis*: $C_{18}H_{18}N_2O_4$ (M = 326.34): triclinic, space group P-1 (no. 2), a = 7.87442(17) Å, b = 13.1736(2) Å, c = 15.8461(3) Å, α = 88.7943(13)°, β = 76.7697(16)°, γ = 88.4490(15)°, V = 1599.38(5) Å^3, Z = 4, T = 100.01(10) K, μ(CuKα) = 0.798 mm^{-1}, *Dcalc* = 1.355 g/cm^3, 55778 reflections measured (5.73° ≤ 2θ ≤ 148.794°), 6470 unique (R_{int} = 0.0294, R_{sigma} = 0.0141) which were used in all calculations. The final R_1 was 0.0380 (I>2σ(I)) and wR_2 was 0.1058 (all data).

Crystal structure determination of 6p-*cis*: $C_{18}H_{20}N_2O_3$ (M =312.36): triclinic, space group P-1 (no. 2), a = 6.8071(3) Å, b = 15.0202(7) Å, c = 15.8559(8) Å, α = 90.798(4)°, β = 101.156(4)°, γ = 100.035(4)°, V = 1564.30(13) Å^3, Z = 4, T = 99.98(10) K, μ(CuKα) = 0.739 mm^{-1}, *Dcalc* = 1.326 g/cm^3, 11128 reflections measured (5.688° ≤ 2θ ≤ 149.102°), 6195 unique (R_{int} = 0.0285, R_{sigma} = 0.0384) which were used in all calculations. The final R_1 was 0.0391 (I>2σ(I)) and wR_2(F_2) was 0.1067 (all data).

Crystal structure determination of 6q-*cis*: $C_{11}H_{14}N_2O_2$ (M =206.24): triclinic, space group P-1 (no. 2), a = 7.5414(3) Å, b = 11.8446(5) Å, c = 12.2635(5) Å, α = 80.154(4)°, β = 88.093(4)°, γ = 88.519(3)°, V = 1078.48(8) Å^3, Z = 4, T = 100.00(10) K, μ(CuKα) = 0.724 mm^{-1}, *Dcalc* = 1.270 g/mm^3, 7212 reflections measured (7.32 ≤ 2θ ≤ 148.982), 4221 unique (R_{int} = 0.0222, R_{sigma} = 0.0333) which were used in all calculations. The final R_1 was 0.0399 (I>2σ(I)) and wR_2(F_2) was 0.1031 (all data).

Crystal structure determination of 11a: $C_{46}H_{42}N_2O$ (M =638.81): monoclinic, space group C2/c (no. 15), a = 12.55604(14) Å, b = 15.09459(13) Å, c = 18.96430(17) Å, β = 105.8657(10)°, V = 3457.35(6) Å^3, Z = 4, T = 99.9(2) K, μ(CuKα) = 0.557 mm^{-1}, *Dcalc* = 1.227 g/cm^3, 32364 reflections measured (9.378° ≤ 2θ ≤ 148.662°), 3515 unique (R_{int} = 0.0309, R_{sigma} = 0.0135) which were used in all calculations. The final R_1 was 0.0364 (I>2σ(I)) and wR_2(F_2) was 0.0919 (all data).

Crystal structure determination of 11b: $C_{48}H_{46}N_2O_3$ (M =698.87): triclinic, space group P-1 (no. 2), a = 10.1705(3) Å, b = 10.2903(3) Å, c = 19.4528(5) Å, α = 99.778(2)°, β = 99.679(2)°, γ = 106.888(2)°, V = 1868.03(9) Å^3, Z = 2, T = 99.99(11) K, μ(CuKα) = 0.600 mm^{-1}, *Dcalc* = 1.242 g/cm^3, 31249 reflections measured (14.244° ≤ 2θ ≤ 140.124°), 7054 unique (R_{int} = 0.0352, R_{sigma} = 0.0246) which were used in all calculations. The final R_1 was 0.0863 (I>2σ(I)) and wR_2(F_2) was 0.2339 (all data).

Crystal structure determination of 11c: $C_{34}H_{34}N_2O$ (M =486.63): monoclinic, space group $P2_1/c$ (no. 14), a = 11.9986(2) Å, b = 10.60180(16) Å, c = 20.9045(4) Å, β = 104.8514(19)°, V = 2570.37(8) Å^3, Z = 4, T = 100.00(10) K, μ(CuKα) = 0.580 mm^{-1}, *Dcalc* = 1.258 g/cm^3, 17451 reflections measured (7.622° ≤ 2θ ≤ 148.838°), 5186 unique (R_{int} = 0.0340, R_{sigma} = 0.0268) which were used in all calculations. The final R_1 was 0.0404 (I>2σ(I)) and wR_2(F_2) was 0.1100 (all data).

The datasets were measured on an Agilent SuperNova diffractometer using an Atlas detector. The data collections were driven and processed and numerical absorption corrections based on gaussian integration over multifaceted crystal models were applied using CrysAlisPro.[2] The structures were solved using ShelXS[3] and refined by a full-matrix least-squares procedure on F^2

in ShelXL.[3] All non-hydrogen atoms were refined with anisotropic displacement parameters. For **6a-*cis***, **6a-*trans***, **6d-*trans***, **6e-*cis***, **6h-*cis***, **6m-*cis***,**6p-*cis*** and**6q-*cis*** the hydrogen atoms belonging to the hydroxyl group(s) (O(2) and O(102)) were located in the electron density and freely refined. All remaining hydrogen atoms for all ten structures were added at calculated positions and refined by use of a riding model with isotropic displacement parameters based on the equivalent isotropic displacement parameter (U_{eq}) of the parent atom. In **6e-*cis***, **6h-*cis***, **6m-*cis***, **6p-*cis*** and **6q-*cis*** there are two crystallographically-independent molecules in the asymmetric unit. In **6a-*trans*** the phenyl group C(13)-C(18)/C(13')-C(18') is disordered over two positions with a refined occupancy ratio of 54(1):46(1). Figures were produced using OLEX2.[4] The CIFs for **6a-*trans***, **6a-*cis***, **6d-*trans***, **6e-*cis***, **6h-*cis***, **6p-*cis***, **6q-*cis***, **11a**, **11b**, **11c** and **6m-*cis*** have been deposited with the CCDC and have been given the deposition numbers 1028121-1028130 and 1038737 respectively.

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