SUPPORTING INFORMATION

Methoxy-Directed Aryl-to-Aryl 1,3-Rhodium Migration

Jing Zhang,[†] Jun-Feng Liu,[‡] Angel Ugrinov,[†] Anthony F. X. Pillai,[†] Zhong-Ming Sun,*[,‡] and Pinjing Zhao*[,†]

[†]Department of Chemistry and Biochemistry, North Dakota State University, Fargo, ND 58102, United States
[‡]State Key Laboratory of Rare Earth Resource Utilization, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences (CAS), Changchun, Jilin 130022, China

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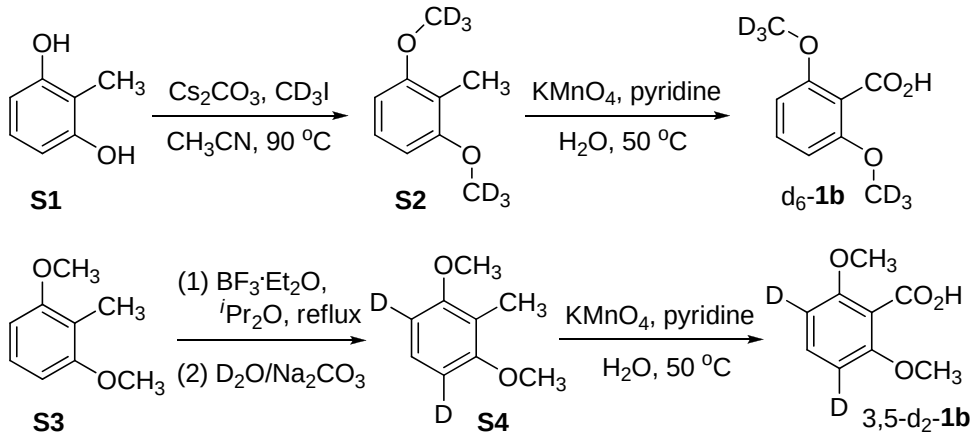
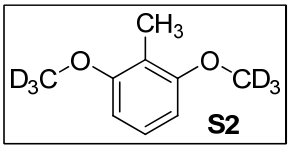
General Experimental Procedure and Reagent Availability

Unless otherwise noted, all manipulations were carried out under a nitrogen atmosphere using standard Schlenk-line or glovebox techniques. All glassware was oven-dried for at least 1 h prior to use. THF, diethyl ether, toluene, benzene, hexane and pentane were degassed by purging with nitrogen for 45 min and dried with a solvent purification system (MBraun MB-SPS). Pyridine, C_6D_6, C_7D_8, D_2O, and THF-d_8 were degassed by purging with nitrogen and dried over activated 3 Å molecular sieves. Other reagents and substrates were purchased from VWR, Strem, Aldrich or Alfa-Aesar and were used as received. TLC plates were visualized by exposure to ultraviolet light or by exposure to I_2 sealed in a bottle at room temperature. Organic solutions were concentrated by rotary evaporation at ~10 torr. Flash column chromatography was performed with 32–63 microns silica gel.

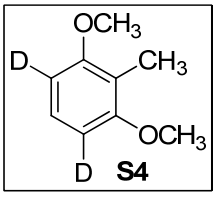
GC analyses were carried on Shimadzu GC-2010 with *n*-dodecane as internal standard material. ^{1}H NMR spectra were obtained on a 400 or 500 MHz spectrometer, and chemical shifts were recorded relative to residual protiated solvent. ^{2}H NMR spectra were obtained on a 76.7 MHz spectrometer. ^{13}C NMR spectra were obtained at 100 MHz, and chemical shifts were recorded relative to the solvent resonance. Both ^{1}H and ^{13}C NMR chemical shifts were reported in parts per million downfield from tetramethylsilane ($\delta = 0$). ^{31}P NMR spectra were obtained at 121.5 MHz, and chemical shifts were reported in parts per million downfield of 85% H_3PO_4 ($\delta = 0$). ^{19}F NMR spectra were obtained at 282.4 MHz, and all chemical shifts were reported in parts per million upfield of CF_3COOH ($\delta = -78.5$ ppm).

Synthesis of Deuterium-Labeled 2,6-Dimethoxybenzoic Acid Derivatives: General Scheme





Synthesis of Deuterium-Labeled (2 x CD_3) 2,6-Dimethoxy Toluene Derivative S2. Compound **S2** was synthesized according to a modified literature procedure.[1] Into a 250 mL round-bottom flask was placed a mixture of 2-methylresorcinol (**S1**, 993 mg, 8.0 mmol, 1.0 equiv.) and Cs_2CO_3 (10.43g, 32.0 mmol, 4.0 equiv.) in 100 mL of acetonitrile. The mixture was refluxed at 90 °C for 1 hour and then CD_3I (3.5 g, 24 mmol, 99.9% D) was injected under nitrogen. The whole mixture was continuously refluxed for 12 hours. After the reaction was cooled, all volatile materials were removed under reduced pressure. The residue was extracted into Et_2O (2 × 100 mL), washed with brine (3 x 20 mL), and dried over anhydrous $MgSO_4$. The solution was then filtered and concentrated under reduced pressure to remove all volatiles to afford the crude product. Further purification was achieved by flash-column chromatography (EtOAc: Hexane, 1:5) and gave 760 mg of product (yield 77%, D% > 99.5%) as a white solid. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.12 (t, 1H, *J* = 8.4 Hz), 6.54 (d, 2H, *J* = 8.4 Hz), 2.13 (s, 3H), ^{2}H-NMR (76.7 MHz, CH_2Cl_2): δ 3.85 (s). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 158.9, 126.5, 115.1, 104.1, 55.0 (very weak, m), 8.4.

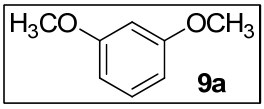


S4

Synthesis of Deuterium-Labeled (4,6-d_2) 2,6-Dimethoxy Toluene Derivative S4. Compound **S4** was synthesized according to a modified literature procedure.[2] To a solution of 1.0 g of 2,6-dimethoxytoluene (**S3**, 6.57 mmol, 1.0 equiv) in 40 mL of $^{i}Pr_2O$, 10 mL of $BF_3{\cdot}Et_2O$ (81.0 mmol, 12.3 equiv) was added under nitrogen. The mixture was refluxed at 85 °C for 12 hours and then quenched using 15 mL of a 10% Na_2CO_3/D_2O solution and continuously stirred for 4 hours. The organic layer was separated and washed with H_2O (2 × 5 mL), dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure to afford the crude product. Further purification was achieved by flash-column chromatography (EtOAc: Hexane, 1:5) and gave 880 mg of product **S4** as a white solid (yield 88%, D% = 70% based on ^{1}HNMR). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.15 (s, broad, 1H), 6.57 (dd, 0.6 H, J_1 = 8.4 Hz, J_2 = 1.6 Hz), 3.85 (s, 6H), 2.16 (s, 3H), ^{2}H-NMR (76.7 MHz, CH_2Cl_2): δ 6.57 (s). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 158.6 (d, J = 4.1 Hz), 126.4 (t, J = 10.2 Hz), 114.7, 103.7, 55.9, 8.4.

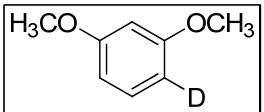
General Procedure for the Oxidation of Deuterium-Labeled 2,6-Dimethoxy Toluene Derivatives (S2, S4) to Form Deuterium-Labeled 2,6-Dimethoxy Benzoic Acid Derivatives (d_6-1b, 3,5-d_2-1b). The oxidation was carried out according to a modified literature procedure.[3] Into a 100 mL round-bottom flask was placed into 500 mg of **S2** or **S4** (3.67 mmol, 1.0 equiv), 5.0 mL of pyridine, and 20 mL of H_2O under stirring. The mixture was heated at 50 °C for 12 hours during which time 1.8 g of $KMnO_4$ (11.3 mmol, 3.0 equiv.) was added in small portions over a period of 1 hour. After cooling down to room temperature, the mixture was filtered through Celite and washed twice with 20 mL of H_2O. The combined filtrate was concentrated to ~5 mL under reduced pressure and then treated by 2 mL of 40% H_2SO_4. The resulting white deposit was collected, washed with 30 mL of cold water (~4 ºC), and dried at 60 °C to give the benzoic acid product as a white solid: 328 mg for d_6-**1b** (49% yield) and 302 mg for 3,5-d_2-**1b** (46%) respectively, without loss of deuterium content (D%) from reactants **S2** and **S4**.

General Procedure for Rh(I)-Catalyzed Protodecarboxylation of 2,6-Dialkoxy Substituted Benoic Acids. Into a 4 mL scintillation vial equipped with a magnetic stir bar was placed hydroxy(1,5-cyclooctadiene)rhodium(I) dimer ([(cod)Rh(OH)]$_2$, 1.5 mg, 0.0034 mmol, 0.015 equiv), DPPP ligand (3.0 mg, 0.007 mmol, 0.03 equiv), and 1.5 mL of toluene. The mixture was stirred at room temperature for 5 minutes until the materials were completely dissolved. Next, the mixture was charged with benzoic acid substrate (0.225 mmol, 1.0 equiv), Na_2CO_3 (24.0 mg, 1.0 equiv), and H_2O or D_2O (250 μL, degassed). The vial was then sealed with a silicone-lined screw-cap, transferred out of the glovebox, and stirred at 120 °C for 30 hours. After the reaction mixture was cooled to room temperature, all volatile materials were removed under reduced pressure. The residue was extracted into ethyl acetate (30 mL), washed with brine (3 x 20 mL), and dried over anhydrous $MgSO_4$. The solution was filtered and the filtrate was concentrated under reduced pressure to give the crude product. Further purification was achieved by flash-column chromatography (EtOAc: Hexane, 1:10). Isolated yields are based on the average of two runs under identical conditions.



9a

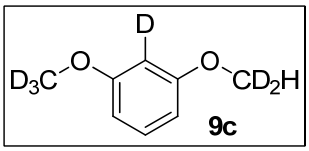
1,3-Dimethoxybenzene (**9a**) was acquired by Rh(I)-catatlyzed protodecarboxylation of 2,6-dimethoxybenzoic acid (**1b**) in H_2O/toluene mixed solvent following the general procedure as a colorless oil in 64% yield. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.18 (t, 1H, J= 8.4 Hz), 6.51 (dd, 2H, J_1 = 8.4 Hz, J_2 = 2.4 Hz), 6.47 (t, 1H, J = 2.0 Hz), 3.78 (s, 6H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 161.1, 130.1, 106.4, 100.7, 55.4.



9b

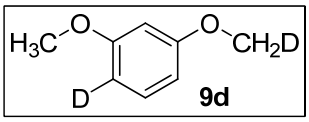
4-*d*-1,3-Dimethoxybenzene (**9b**) was acquired by Rh(I)-catatlyzed protodecarboxylation of 2,6-dimethoxybenzoic acid (**1b**) in D_2O/toluene mixed solvent following the general procedure as a colorless oil in 61% yield. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.18 (t, 1H, J= 8.4 Hz), 6.50 (dt, 1.3 H, J_1 = 7.2 Hz, J_2 = 1.2 Hz), 6.46 (d, 1H, J = 2.4 Hz), 3.78 (s, 6H); ^{2}H-NMR (76.7 MHz,

CH_2Cl_2): δ 6.50 (s, 1.0 D), 3.77 (s, trace). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 161.1, 130.0 (d, J = 8.1 Hz), 106.4, 100.7, 55.4. D% >90% based on ^{1}H-NMR.



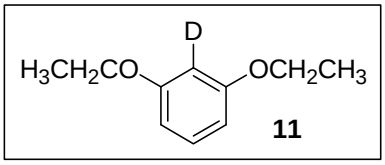
9c

Compound **9c** was acquired by Rh(I)-catatlyzed protodecarboxylation of methoxy-deuterated 2,6-dimethoxybenzoic acid (d_6-**1b**) in H_2O/toluene following the general procedure as a colorless oil in 67% yield. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.16 (m, 1H), 6.42-6.52 (m, 2H), 3.73 (s, 1H); ^{2}H-NMR (76.7 MHz, CH_2Cl_2): δ 6.51 (s, 1D), 3.76 (s, 5D). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 161.1, 130.0 (m), 106.4, 100.7, 54.9 (t, J = 21.6 Hz). D% >90% based on ^{1}H-NMR.



9d

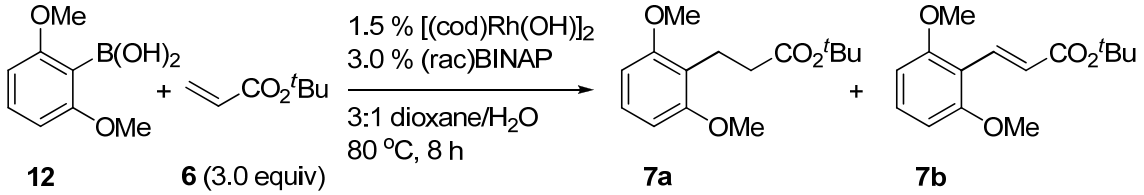
Compound **9d** was acquired by Rh(I)-catatlyzed protodecarboxylation of 3,5-deuterated 2,6-dimethoxybenzoic acid (3,5-d_2-**1b**) in H_2O/toluene mixed solvent following the general procedure as a colorless oil in 59% yield. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.22 (m, 1H), 6.54 (dd, 1.4 H, J_1 = 6.8 Hz, J_2 = 2.4 Hz), 6.50 (t, 1H, J = 2.0 Hz), 3.83 (s, 3H), 3.81 (t, J = 2.4 Hz); ^{2}H-NMR (76.7 MHz, CH_2Cl_2): δ 6.55 (s, 1D), 3.81 (s, 1D). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 161.1, 130.0 (d, J = 10.8 Hz), 106.4, 100.7, 55.5, 55.2 (t, J = 18.5 Hz). D% ~60% based on ^{1}H-NMR (Note: 70% deuterium content for the reactant 3,5-d_2-**1b**).



11

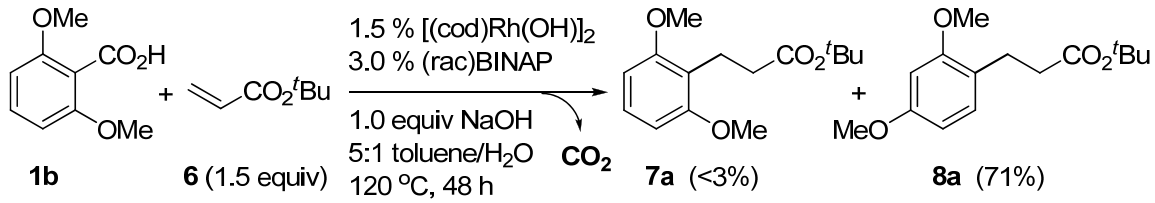
Compound **11** was acquired by Rh(I)-catatlyzed protodecarboxylation of 2,6-diethoxybenzoic acid (**10**) in D_2O/toluene mixed solvent following the general procedure as a colorless oil in 85% yield. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.14 (t, 1H, J = 8.0 Hz), 6.47 (d, 2H, J = 8.4 Hz), 4.00 (q, 4H, J= 7.2 Hz), 1.39 (t, 6H, J = 6.8 Hz). ^{2}H-NMR (76.7 MHz, CH_2Cl_2): 6.41 (s). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 160.4, 130.0, 106.9, 101.6, 63.6, 15.0. D% >90% based on ^{1}H-NMR.

Synthesis of 3-(2,6-Dimethoxyphenyl)-Propionic Acid *tert*-Butyl Ester (7a)



Compound **7a** was independently synthesized for comparison using a modified literature procedure.[4] Into a 4 mL scintillation vial equipped with a magnetic stir bar was placed 2,6-dimethoxyphenylboronic acid (**12**, 182 mg, 1.0 mmol), *tert*-butyl acrylate (**6**, 302 mg, 3.0 mmol), $[(cod)Rh(OH)]_2$ (6.8 mg, 0.015 mmol, 0.015 equiv), BINAP ligand (18.7 mg, 0.030 mmol, 0.030 equiv) and mixed dioxane/H_2O (1.5 mL : 0.5 mL). The vial was then sealed with a silicone-lined screw-cap, transferred out of the glovebox, and stirred at 80 °C for 8 hours. After the reaction, the mixture was quenched by H_2O (50 mL) and extracted by EtOAc (25 mL × 2). The combined organic phase was washed by saturated $NaHCO_3$ (30 mL × 2), brine (30 mL × 2) and concentrated under reduced pressure to give the crude product as a 2:1 mixture of **7a** and olefination product **7b** in 65% overall yield by GC analysis. Further purification by flash-column chromatography (10% ethyl acetate in hexane) gave **7a** as a colorless oil (107 mg, 43%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.11 (t, 1H, *J* = 8.0 Hz), 6.50 (d, 2H, *J* = 8.4 Hz), 3.78 (s, 6H), 2.91 (t, 2H, *J* = 8.0 Hz), 2.39 (t, 2H, *J* = 8.0 Hz), 1.42 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 173.4, 158.5, 127.3, 117.4, 103.8, 79.9, 55.8, 34.9, 28.3, 18.9. HRMS: calcd for $C_{15}H_{22}O_4Na^+$ 289.1410, found 289.1398; $C_{11}H_{14}O_4Na^+$ 233.0784, found 233.0792 (*t*-Bu group loss).

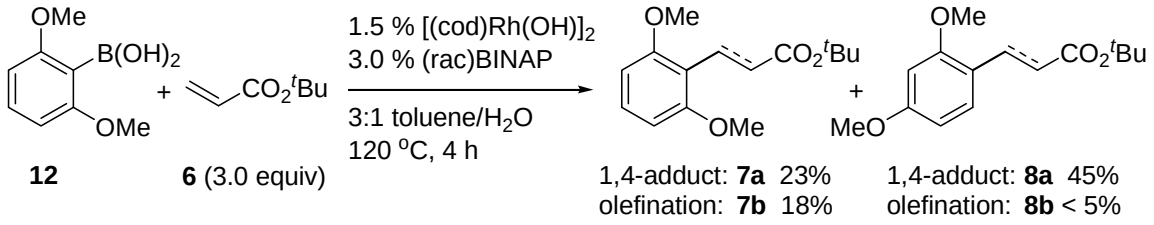
Rh(I)-Catalyzed Decarboxylative Conjugate Addition to Form 1,3-Migration Product 8a.



Into a 20 mL scintillation vial equipped with a magnetic stir bar was placed $[(cod)Rh(OH)]_2$ (15 mg, 0.034 mmol), BINAP (42 mg, 0.068 mmol), and 10.0 mL of toluene. The mixture was stirred at 60 °C for 1 hour until the materials were completely dissolved to form a stock solution.

Next, into a 4 mL screw-cap vial equipped with a magnetic stir bar was placed 2,6-dimethoxybenzoic acid **1b** (41.0 mg, 0.225 mmol, 1.0 equiv), *t*-butyl acrylate **6** (43.3 mg, 0.338 mmol, 1.5 equiv equiv), NaOH (9.0 mg, 1.0 equiv), H_2O (200 μL, degassed), and 1.0 mL of the Rh/phosphine stock solution in toluene (containing 0.015 equiv of $[(cod)Rh(OH)]_2$ and 0.030 equiv of BINAP). The vial was sealed with a silicone-lined screw-cap, transferred out of the glovebox, and stirred at 120 °C for 48 hours. After the reaction mixture was cooled, all volatile materials were removed under reduced pressure. The residue was extracted into ethyl acetate (30 mL), washed with brine (3 × 20 mL), dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to give the crude product. Further purification was achieved by flash-column chromatography (EtOAc/Hexane) to give **8a** as colorless oil (42.5 mg, 71% yield). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.04 (d, 1H, J = 8.0 Hz), 6.43 (d, 1H, J = 2.4 Hz), 6.39 (dd, 1H, J_1 = 8.0 Hz, J_2 = 2.4 Hz), 3.80 (s, 3H), 3.79 (s, 3H), 2.82 (t, 2H, J = 8.0 Hz), 2.47 (t, 2H, J = 8.0 Hz), 1.42 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 173.1, 159.6, 158.6, 130.3, 121.8, 103.9, 80.2, 55.4, 35.9, 28.3, 25.7. HRMS: calcd for $C_{15}H_{22}O_4Na^+$ 289.1410, found 289.1408. $C_{11}H_{14}O_4Na^+$ 233.0784, found 233.0787 (*t*-Bu group loss).

Rh(I)-Catalyzed Areneboronic Acid Conjugate Addition to Form 1,3-Migration Product 8a.



Into a 20 mL scintillation vial equipped with a magnetic stir bar was placed $[(cod)Rh(OH)]_2$ (15 mg, 0.034 mmol), BINAP (42 mg, 0.068 mmol), and 10.0 mL of toluene. The mixture was stirred at 60 °C for 1 hour until the materials were completely dissolved to form a stock solution. Next, into a 4 mL scintillation vial equipped with a magnetic stir bar was placed 2,6-dimethoxy-benzeneboronic acid (**12**, 41 mg, 0.225 mmol, 1.0 equiv), *tert*-butyl acrylate (**6**, 68 mg, 0.675 mmol, 3.0 equiv), 1.0 mL of the Rh/BINAP stock solution (0.015 equiv $[(cod)Rh(OH)]_2$ and 0.030 equiv BINAP ligand) and 330 μL of H_2O to make a 3:1 toluene/H_2O solution. The vial was then sealed with a silicone-lined screw-cap, transferred out of the glovebox, and stirred at 120 °C for 4 hours. The yields of **7a**, **7b**, **8a** and **8b** were determined by GC analysis.

Table S1. Summary of Cell Parameters, Data Collection and Structural Refinements.

Empirical formula	$C_{45}H_{37}O_4P_2Rh$ **2b**	$C_{50}H_{42}NO_4P_2Rh$ **3b**	$C_{100}H_{84}N_2O_8P_4Rh_2$ **4b**
Formula weight	806.60	885.70	1771.39
Temperature, K		293(2)	
Wavelength, λ(Å)		Mo K_α , 0.71073	
space group	*C*2/c	*P*-1	$P2_1/n$
a/Å	13.498(5)	13.0849(9)	21.6627(17)
b/Å	26.851(10)	13.2803(9)	17.5320(14)
c/Å	14.881(5)	14.9486(11)	25.769(2)
α, deg	90	78.0370(10)	90
β, deg	101.799(7)	70.5080(10)	98.035(2)
γ, deg	90	87.3840(10)	90
V, $Å^3$	5279(3)	2394.8(3)	9690.8(13)
Z	4	2	4
D_{calcd}, $g.cm^{-3}$	1.015	1.228	1.214
μ, mm^{-1}	0.415	0.464	0.459
F(000)	1656	912	3648
Theta range, deg	1.52 to 24.96	1.48 to 27.00	1.15 to 23.28
hkl ranges	[-15, 11], [-30, 31], ±17	[-15, 16], ±16, ±19	[-24, 23], [-18, 19], ±28
Refltns collected/unique	13886/4605 (R_{int} = 0.0544)	21525/10303(R_{int} = 0.0304)	62598/13914(R_{int} = 0.0528)
GOOF on F^2	1.001	0.998	1.077
R_1, wR_2 ($I>2\sigma(I)$)[a]	0.0528/ 0.1269	0.0475/0.1387	0.0399/0.1223
R_1, wR_2 (all data)	0.0889/0.1355	0.0660/0.1490	0.0519/0.1283
Largest diff. peak and hole (e. $Å^{-3}$)	0.402 / -0.678	0.472 / -0.596	0.511 /-0.577

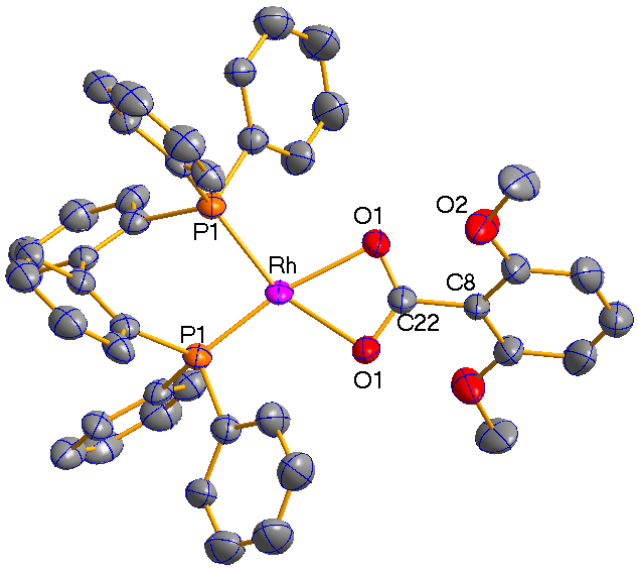
[a] $R_1 = \Sigma||Fo| - |Fc||/\Sigma|Fo|$, $wR_2 = \{\Sigma w[(Fo)^2 - (Fc)^2]^2/\Sigma w[(Fo)^2]^2\}^{1/2}$

Table S2. Selected Bond Lengths [Å] and Bond Angles [degree].

		$C_{45}H_{39}O_4P_2Rh$ **2b**	
Rh(1)-O(1)	2.161(3)	Rh(1)-P(1)	2.1763(12)
Rh(1)-O(1)#1	2.161(3)	Rh(1)-P(1)#1	2.1763(12)
Rh(1)- C(22)	2.505(6)	P(1)-C(13)	1.821(5)
P(1)-C(7)	1.823(4)	C(21)-O(2)	1.328(6)
C(22)-O(1)	1.284(4)	C(24)-O(2)	1.443(5)
O(1)-Rh(1)-O(1) #1	61.66(16)	O(1) #1-Rh(1)-P(1)	103.68(9)
O(1)-Rh(1)-P(1)	165.23(8)	P(1)-Rh(1)-P(1) #1	91.02(6)
		$C_{50}H_{44}NO_4P_2Rh$ **3b**	
Rh(1)-O(1)	2.110(2)	Rh(1)-N(1)	2.136(3)
Rh(1)-P(2)	2.2298(9)	Rh(1)-P(1)	2.1987(8)
P(1)-C(13)	1.836(3)	P(1)-C(7)	1.840(3)
P(1)-C(1)	1.841(4)	P(2)-C(25)	1.836(3)
P(2)-C(31)	1.840(3)	P(2)-C(19)	1.857(3)
O(1)-Rh(1)-N(1)	81.48(11)	O(1)-Rh(1)-P(2)	94.22(7)
N(1)-Rh(1)-P(2)	173.77(10)	O(1)-Rh(1)-P(1)	172.87(7)
N(1)-Rh(1)-P(1)	92.32(9)	P(2)-Rh(1)-P(1)	92.23(3)
		$C_{113}H_{112}N_2O_8P_4Rh_2$ **4b**	
Rh(1)-O(5)	2.120(2)	Rh(1)-N(1)	2.145(3)
Rh(1)-P(2)	2.1892(9)	Rh(1)-P(1)	2.2099(9)
Rh(2)-O(1)	2.094(3)	Rh(2)-N(2)	2.141(3)
Rh(2)-P(3)	2.2029(10)	Rh(2)-P(4)	2.1841(9)
P(1)-C(7)	1.828(4)	P(1)-C(1)	1.855(4)
P(1)-C(13)	1.840(3)	P(2)-C(25)	1.835(4)
P(2)-C(19)	1.844(3)	P(2)-C(31)	1.855(4)
P(3)-C(57)	1.833(4)	P(3)-C(51)	1.838(4)
P(3)-C(63)	1.837(4)	P(4)-C(69)	1.836(3)
P(4)-C(75)	1.838(4)	P(4)-C(81)	1.843(4)

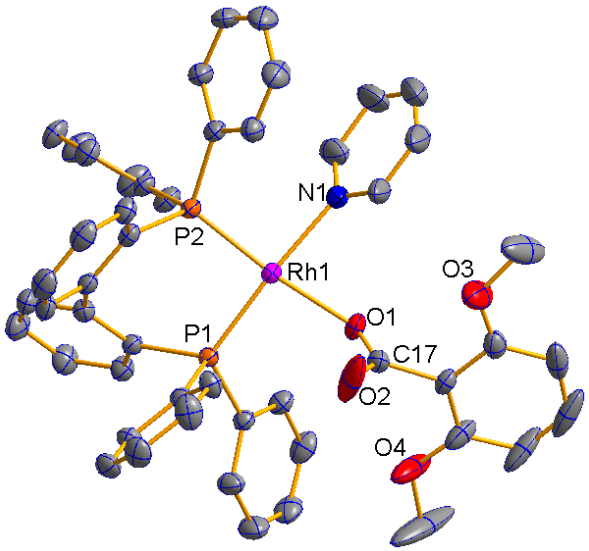
O(5)-Rh(1)-N(1)	85.35(10)	O(5)-Rh(1)-P(2)	176.26(7)
N(1)-Rh(1)-P(2)	94.52(8)	O(5)-Rh(1)-P(1)	89.39(7)
N(1)-Rh(1)-P(1)	170.71(8)	P(2)-Rh(1)-P(1)	91.22(3)
O(1)-Rh(2)-N(2)	80.43(12)	O(1)-Rh(2)-P(4)	171.01(10)
N(2)-Rh(2)-P(4)	92.80(9)	O(1)-Rh(2)-P(3)	95.32(9)
N(2)-Rh(2)-P(3)	168.48(9)	P(4)-Rh(2)-P(3)	92.42(4)

Symmetry transformations used to generate equivalent atoms: #1: -x+1, y, -z+1/2



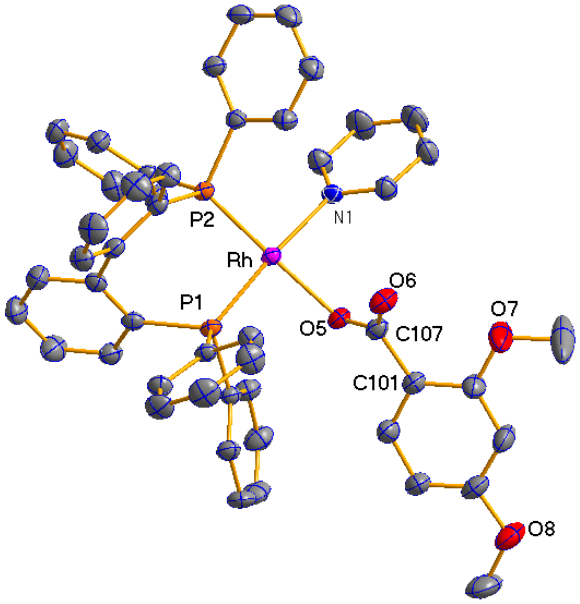
Preparation of {(BIPHEP)RhOCO[η^2-OCO(2,6-OMe-C_6H_3)]}·hexane (2b). Into a 20 mL scintillation vial equipped with a magnetic stir bar was placed $[(COD)Rh(\mu\text{-}OH)]_2$ (15.0 mg, 0.034 mmol, 1.0 equiv), BIPHEP (40.0 mg, 0.076 mmol, 2.2 equiv), and 4.0 mL of THF. The mixture was stirred at 80 °C for 1 hour. After cooling down to room temperature, all volatile materials were removed under reduced pressure, and the residue was re-dissolved using THF (4.0 mL). 12.4 mg of 2,6-dimethoxybenzoic acid (0.068 mmol, 2.0 equiv) was added as a powder and the mixture was stirred at 60 °C for 2 hours to reach full conversion, as determined by ^{31}P

NMR spectroscopy. The solvent THF was removed under reduced pressure, and the residue was extracted into 3.0 mL of toluene. After gentle heating at 60 °C (~ 30 min), the solution was filtered through a pad of tightly packed glass wool. The filtrate was transferred into a 7 mL vial, carefully layered with hexane (3 mL), and left undisturbed. Maroon-colored block crystals of **2b** were obtained after three days (38.0 mg, 61% yield based on Rh). This complex is air-sensitive and satisfactory element analysis could not be obtained. ^{31}P-NMR spectra are provided to demonstrate purity. ^{31}P-NMR (121.5 MHz, toluene): δ 53.86 (d, J_{Rh-P} = 196.8 Hz).



Preparation of {(BIPHEP)RhOCO[(2,6-(OMe)$_2$-C$_6$H$_3$)(pyridine)]}·(toluene) (3b). Into a 20 mL scintillation vial equipped with a magnetic stir bar was placed [(COD)Rh(μ-OH)]$_2$ (15.0 mg, 0.034 mmol, 1.0 equiv), BIPHEP (40.0 mg, 0.076 mmol, 2.2 equiv), and 4.0 mL of THF. The mixture was stirred at 80 °C for 1 hour. After cooling down to room temperature, all volatile materials were removed under reduced pressure, and the residue was re-dissolved using THF (4.0 mL). 2,6- Dimethoxybenzoic acid (12.4 mg, 0.068 mmol, 2.0 equiv) was added as a powder and the mixture was stirred at 60 °C for 2 hours to reach full conversion, as determined by ^{31}P NMR spectroscopy. The solvent THF was removed under reduced pressure, and the residue was added 8.0 mg of pyridine (0.10 mmol, 1.5 equiv) and then extracted into 3.0 mL of toluene to form a homogeneous solution. The solution was filtered through a pad of tightly packed glass wool. The filtrate was transferred into a 7 mL vial, carefully layered with hexane (3.0 mL), and

left undisturbed. Red needle-like crystals of **3b** were obtained after three days (34.0 mg, 52% yield based on Rh). This complex is air-sensitive and satisfactory element analysis could not be obtained. ^{31}P-NMR spectra are provided to demonstrate purity. ^{31}P-NMR (202.3 MHz, toluene, 263.0 K): δ 54.1 (broad dublet, $J_{Rh\text{-}P}$ = 166.1 Hz), 48.6 (d, $J_{Rh\text{-}P}$ = 141.2 Hz).



Preparation of {(BIPHEP)Rh[(2,4-OMe-C_6H_3)(pyridine)]}·(toluene)(hexane) (4b). Into a 4 mL scintillation vial equipped with a magnetic stir bar was placed **{(BIPHEP)RhOCO[(2,6-OMe-C_6H_3)(pyridine)]}·(toluene)** (100 mg, 0.10 mmol, 1.0 equiv), pyridine (12 mg, 0.15 mmol, 1.5 equiv), and 3.0 mL of toluene. The mixture was stirred at 120 °C for 3 hours to reach full conversion. After cooling down to room temperature, the solution was filtered through a pad of tightly packed glass wool. The filtrate was transferred into a 7 mL vial, carefully layered with hexane (3.0 mL), and left undisturbed. Red needle-like crystals of **4b** were obtained after three days (18 mg, 18% yield based on Rh). The yield could be increased to 71% (^{1}HNMR yield using dodecahydrotriphenylene as internal standard) under 1 atm of CO_2 after heating at 120°C for 2 hours with >95% conversion. This complex is air-sensitive and satisfactory element analysis could not be obtained. ^{31}P-NMR spectra are provided to demonstrate purity. ^{31}P-NMR (202.3 MHz, toluene, 263.0 K): δ 55.7 (broad dublet, $J_{Rh\text{-}P}$ = 169.7 Hz), 50.4 (d, $J_{Rh\text{-}P}$ = 145.0 Hz).

References

1. J. C. Lee, J. Y. Yuk, S.H. Cho, *Synth. Commun.* **1995**, *25*, 1367-1370.

2. A. Kreuchunas, *J. Org. Chem.* **1956**, *21*, 368-369.

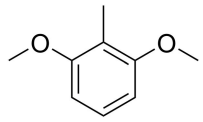
3. A. R. Makriyannis, A. Charalambous, C. J. van der Schyf, A. Makriyannis, *J. Labelled. Comp. Radiopharm.* **1987**, *24*, 1479-1482.

4. T. Hayashi, M. Takahashi, Y. Takaya, M. Ogasawara, *J. Am. Chem. Soc.* **2002**, *124*, 5052-5058.

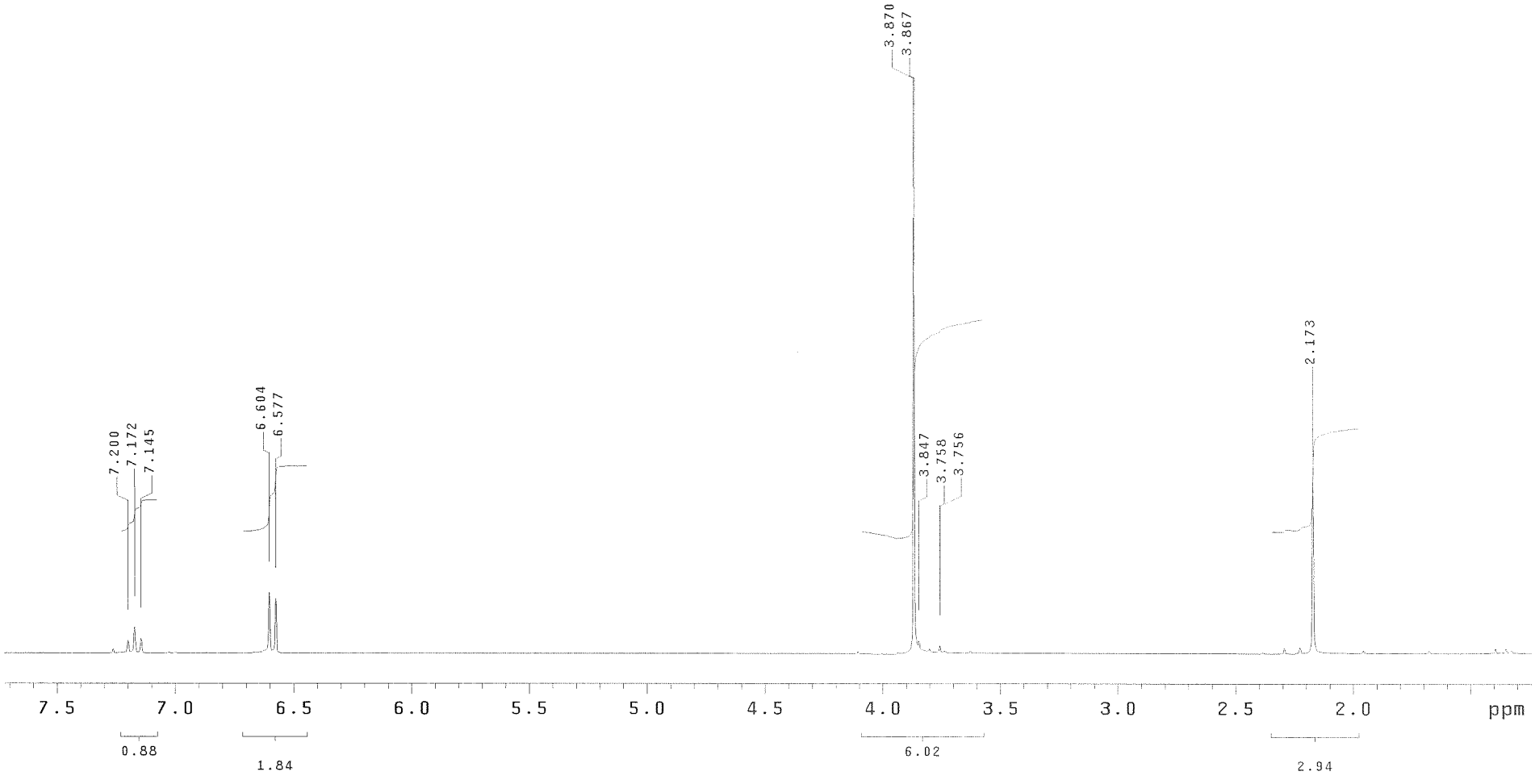
zsun-III-142-H1

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
Operator: zsun
File: zsun-III-142-H1
Mercury-300BB "vnmr300"

 Relax. delay 1.000 sec
 Pulse 66.3 degrees
 Acq. time 1.997 sec
 Width 3301.4 Hz
 40 repetitions
OBSERVE   H1, 300.1377222 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 16384
Total time 0 min, 01sec



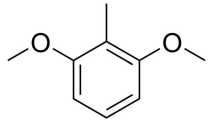
S3



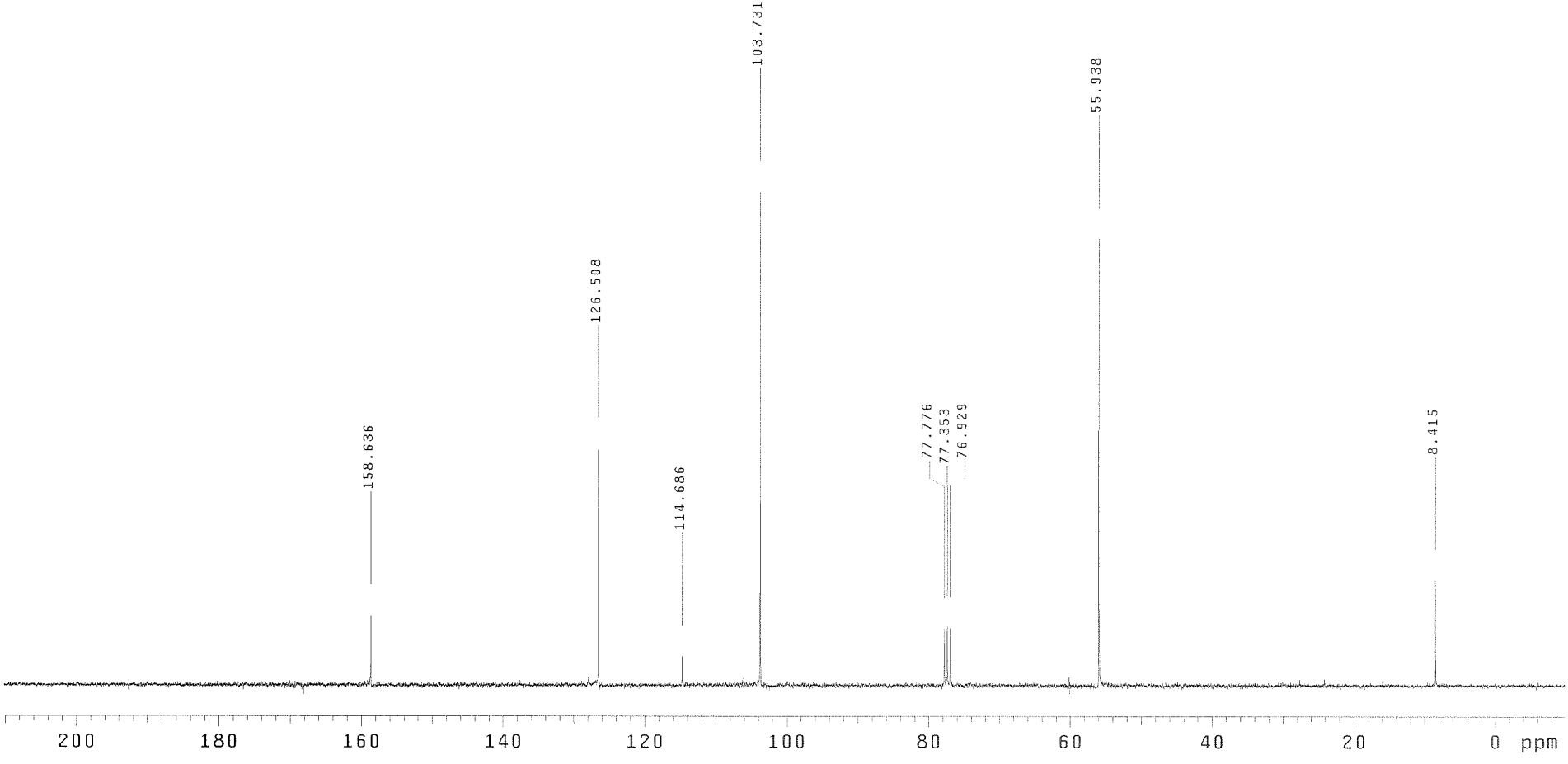
zsun-III-142-C13

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
Operator: zsun
File: zsun-III-142-C13
Mercury-300BB "vnmr300"

 Relax. delay 0.200 sec
 Pulse 44.6 degrees
 Acq. time 1.927 sec
 Width 16611.3 Hz
 204 repetitions
OBSERVE C13, 75.4696723 MHz
DECOUPLE H1, 300.1391844 MHz
 Power 43 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 30 min, 52 sec



S3

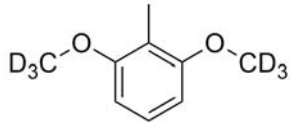


zsun-III-143-H1

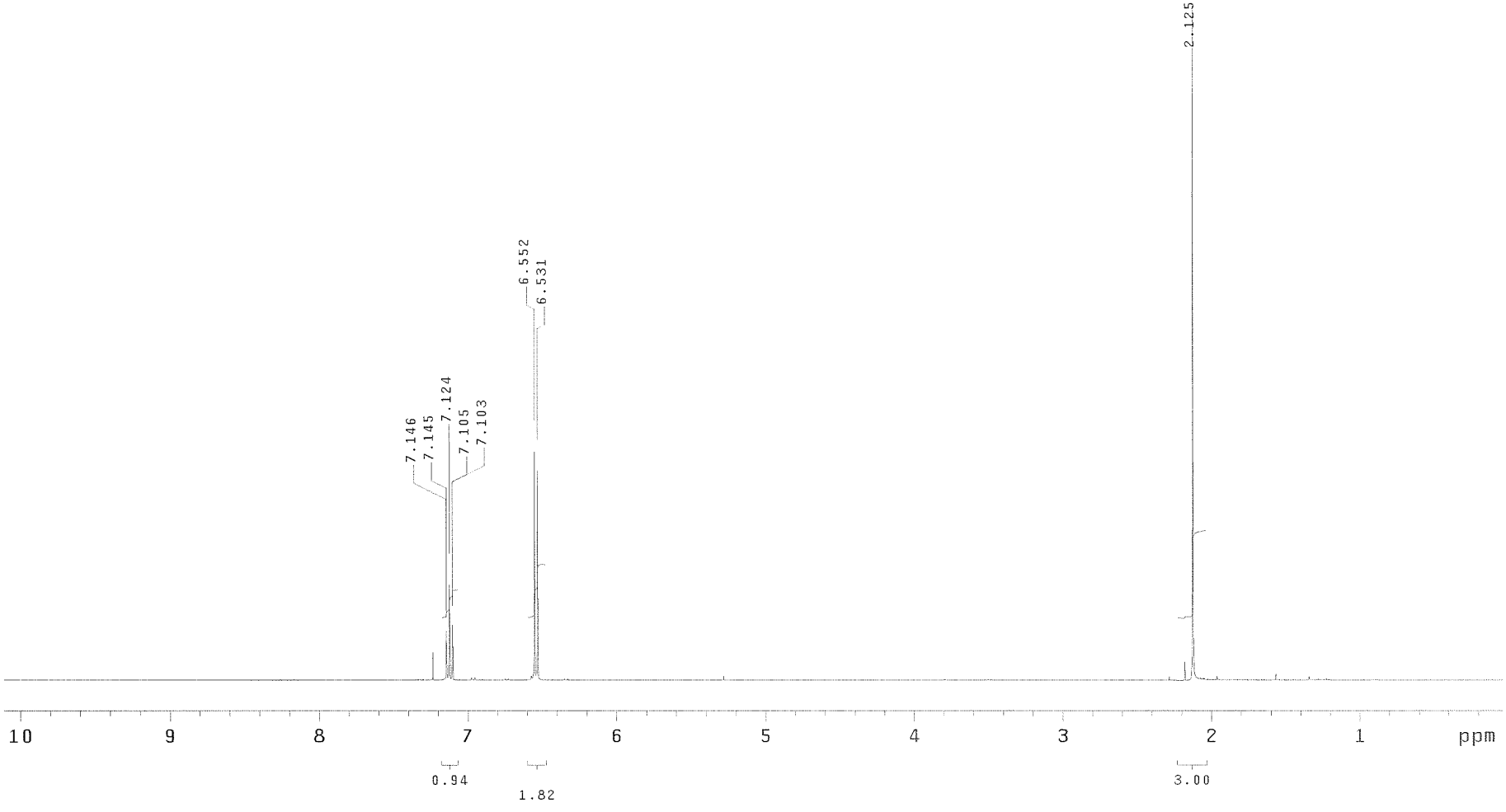
Pulse Sequence: s2pul

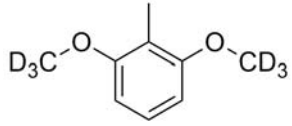
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: zsun-III-143-H1
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



S2



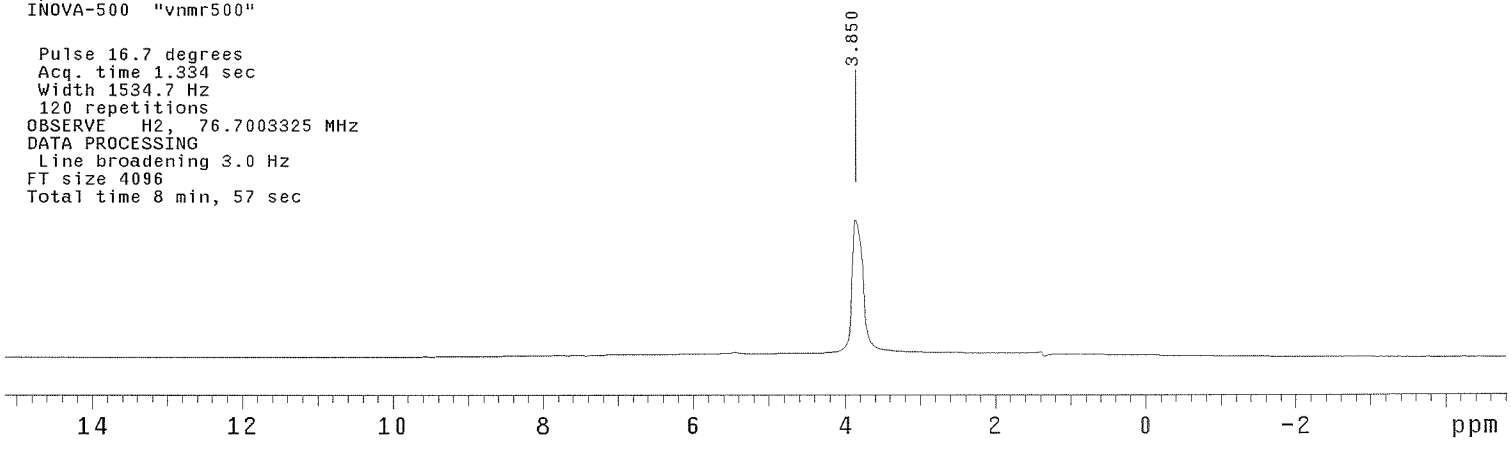


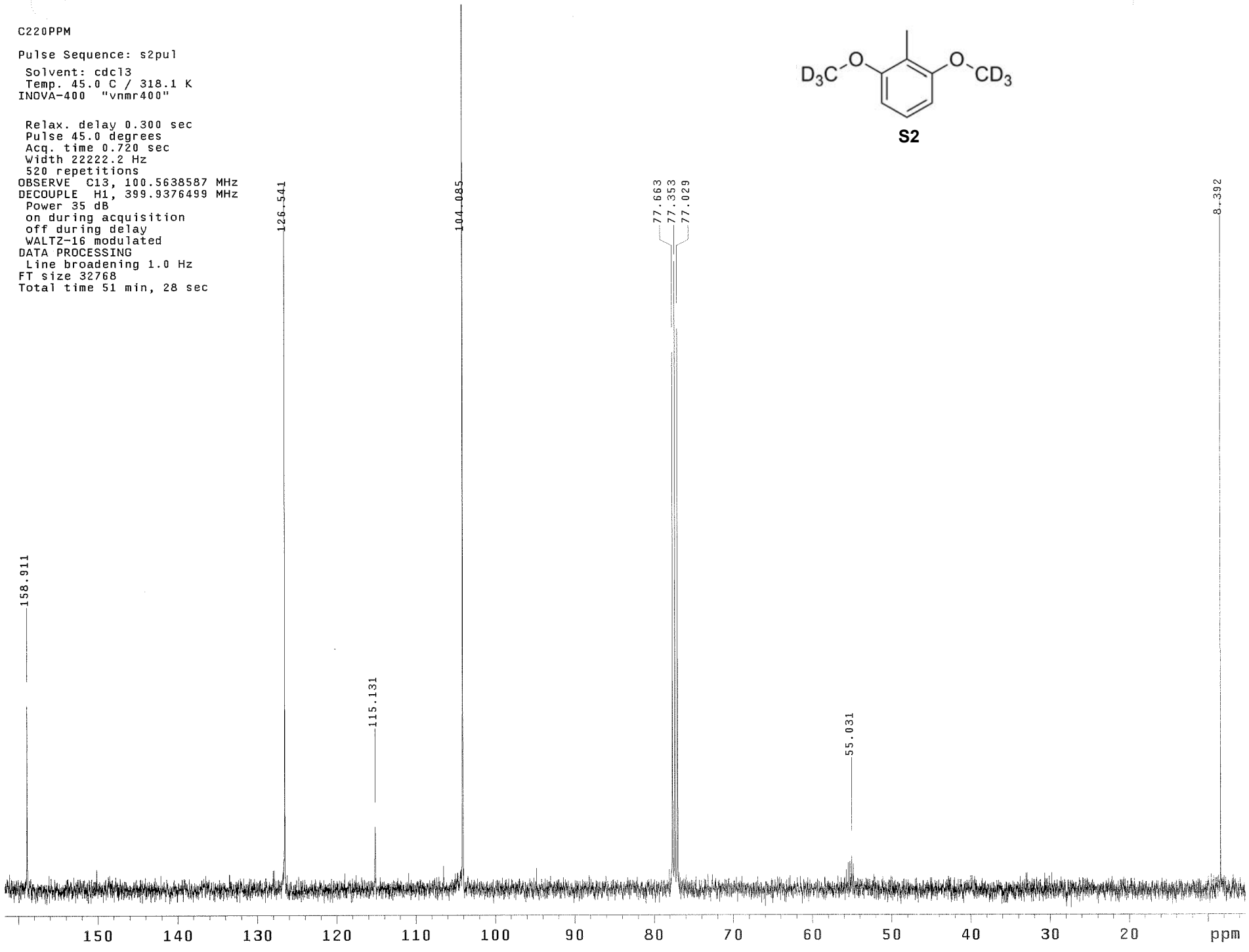
S2

zsun-III-143-H2

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: zsun
INOVA-500 "vnmr500"

Pulse 16.7 degrees
Acq. time 1.334 sec
Width 1534.7 Hz
120 repetitions
OBSERVE H2, 76.7003325 MHz
DATA PROCESSING
Line broadening 3.0 Hz
FT size 4096
Total time 8 min, 57 sec




C220PPM
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 45.0 C / 318.1 K
INOVA-400 "vnmr400"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.720 sec
Width 22222.2 Hz
520 repetitions
OBSERVE C13, 100.5638587 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 51 min, 28 sec
158.911
126.541
115.131
104.085
77.663
77.353
77.029
55.031
8.392
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

D3C
O
O
CD3

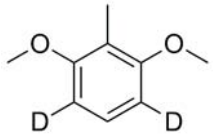
S2

zsun-IV-58-H1

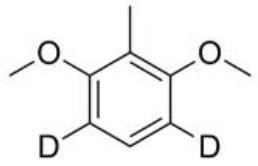
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



S4

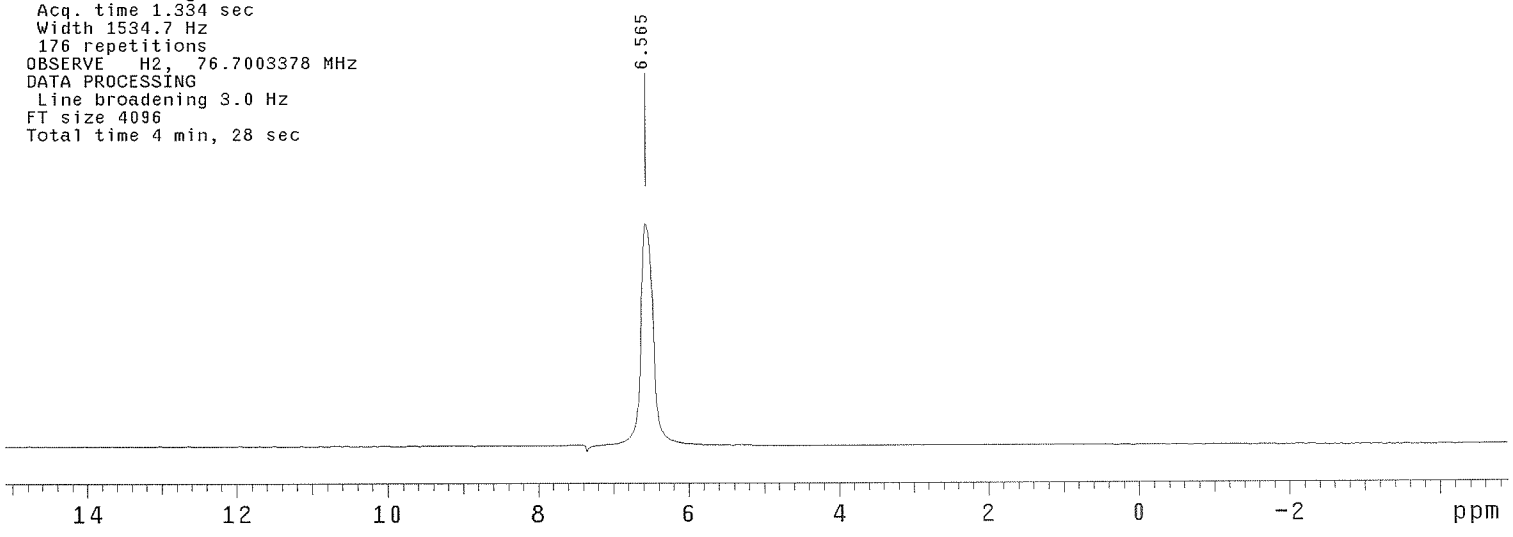


S4

zsun-IV-58-H2

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: zsun
INOVA-500 "vnmr500"

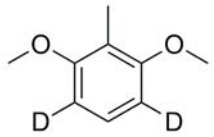
Pulse 16.7 degrees
Acq. time 1.334 sec
Width 1534.7 Hz
176 repetitions
OBSERVE H2, 76.7003378 MHz
DATA PROCESSING
Line broadening 3.0 Hz
FT size 4096
Total time 4 min, 28 sec



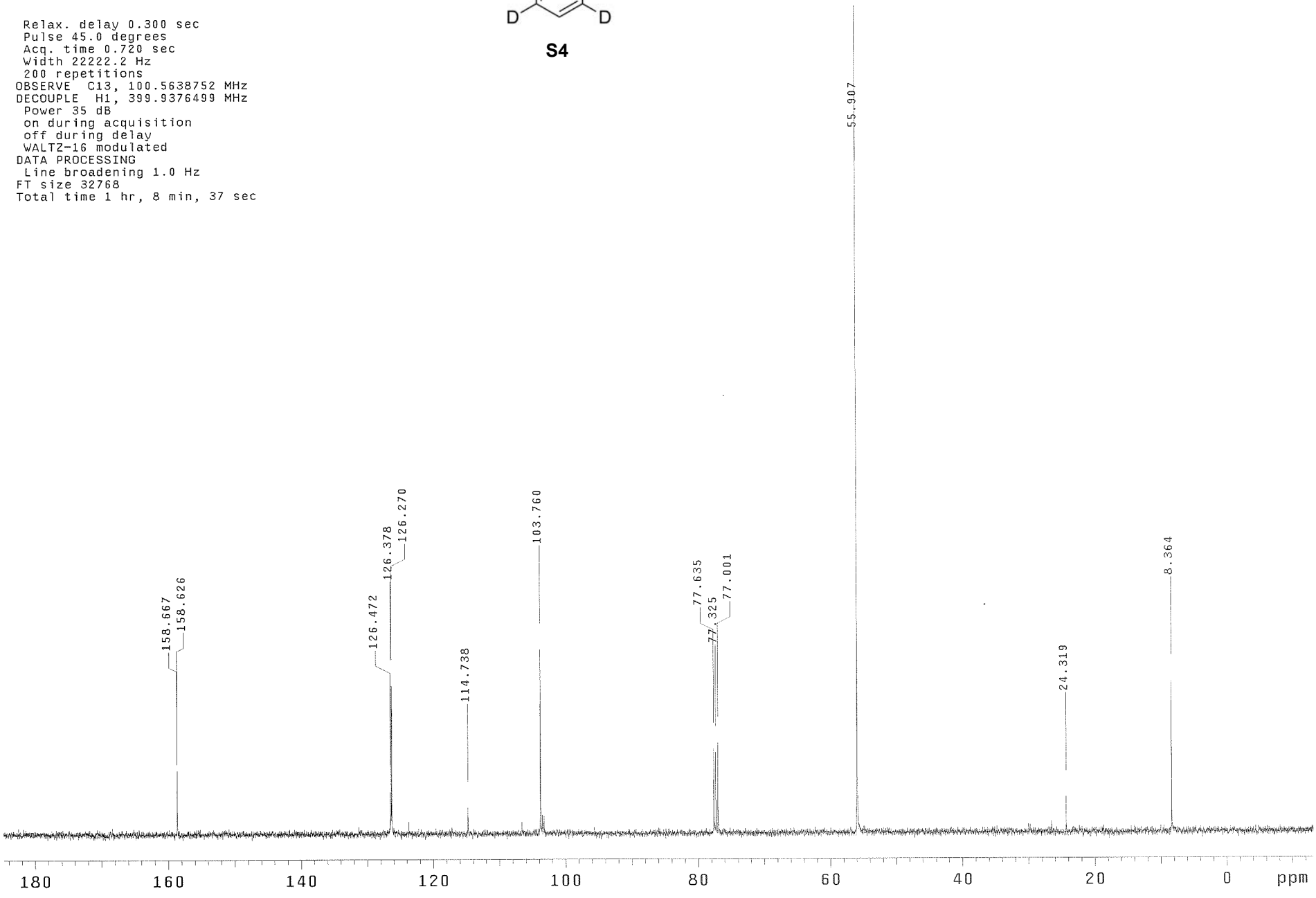
zsun-IV-58-C13

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.720 sec
Width 22222.2 Hz
200 repetitions
OBSERVE C13, 100.5638752 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 1 hr, 8 min, 37 sec



S4

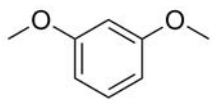


zsun-IV-14-Standard-H1

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
32 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



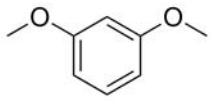
9a

zsun-IV-14-Standard-H1

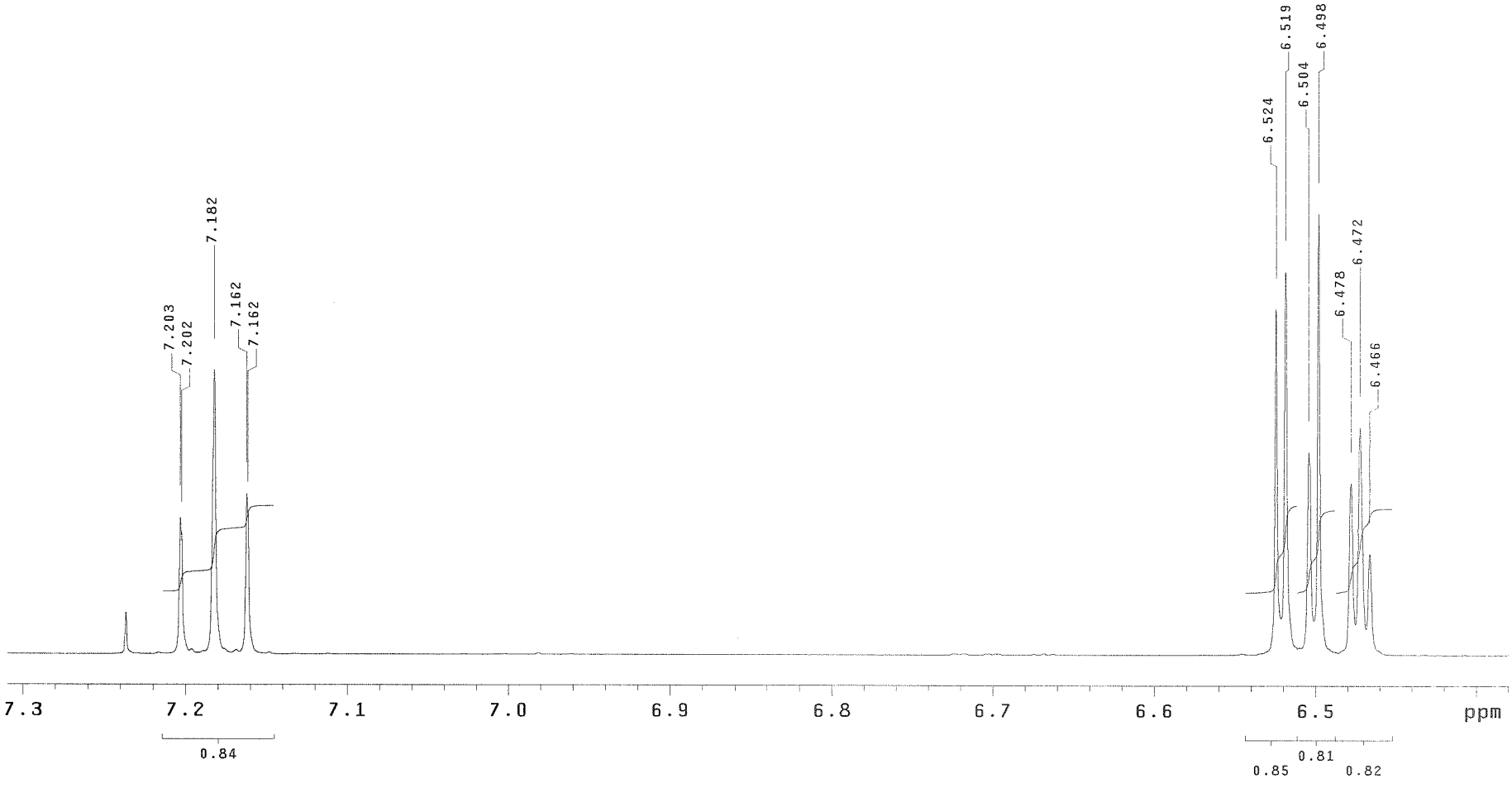
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
40 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



9a

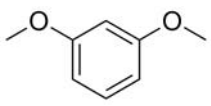


zsun-IV-14-Standard-C13

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.720 sec
Width 22222.2 Hz
200 repetitions
OBSERVE C13, 100.5638752 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 min, 53 sec



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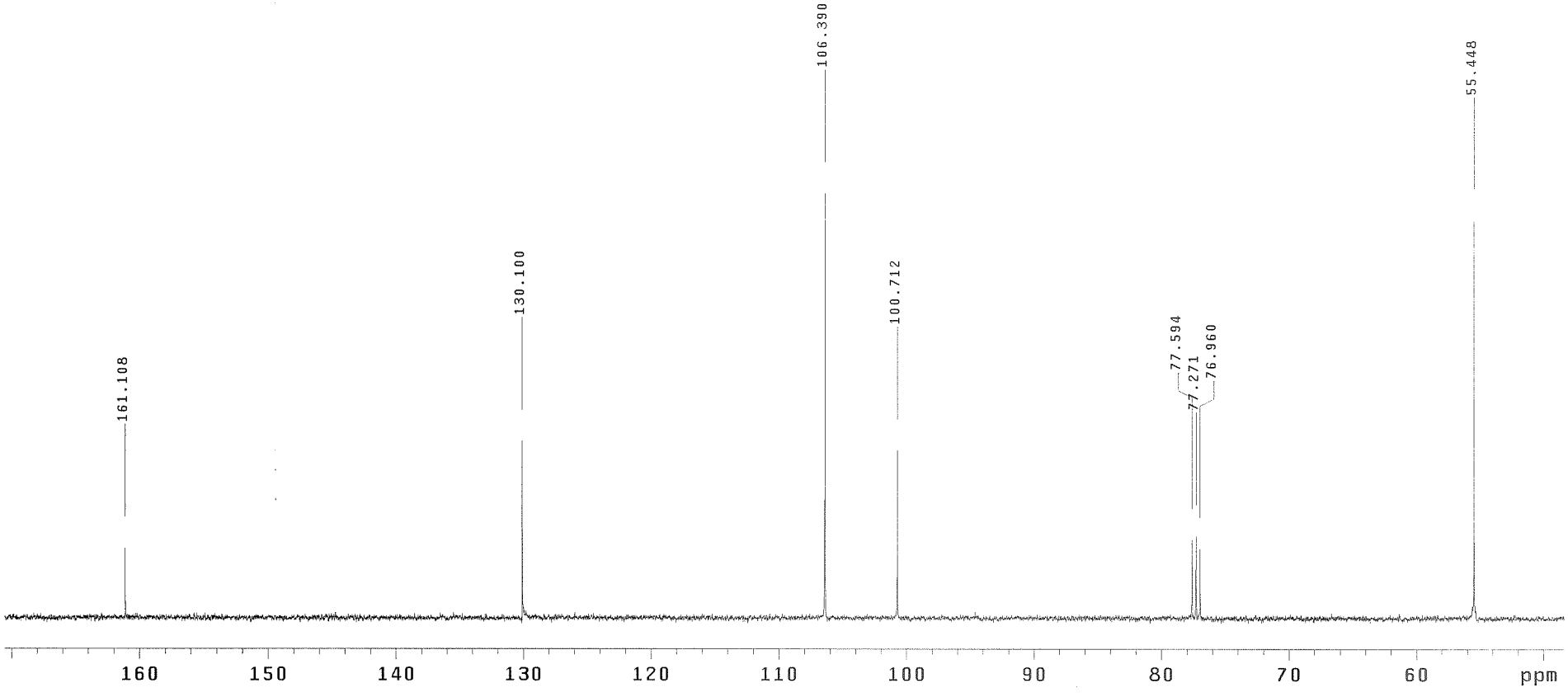
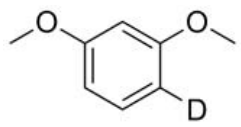


zsun-IV-68-H1

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



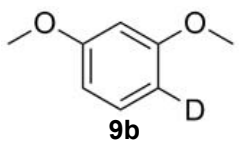
9b

zsun-IV-68-H1

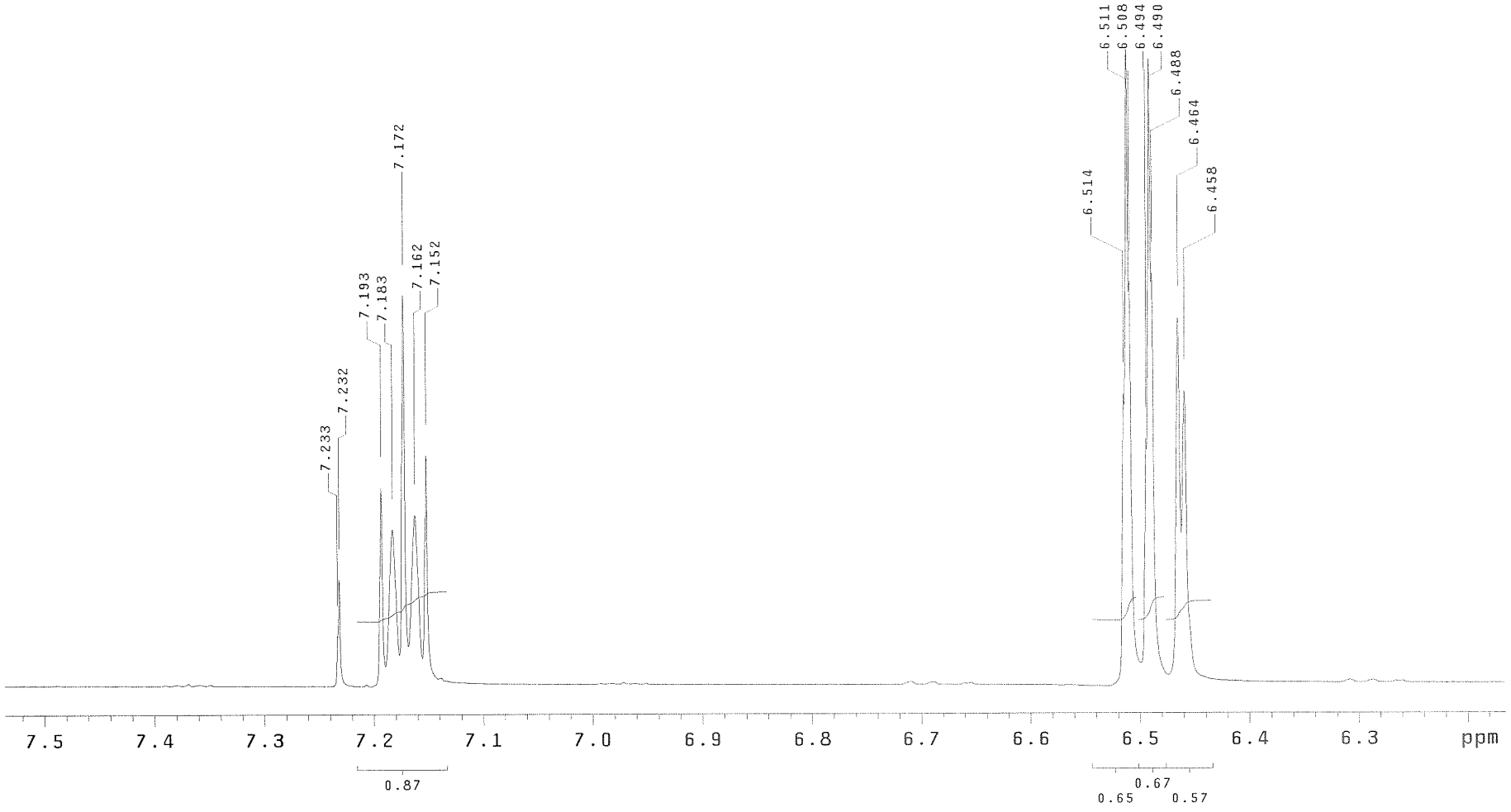
Pulse Sequence: s2pul

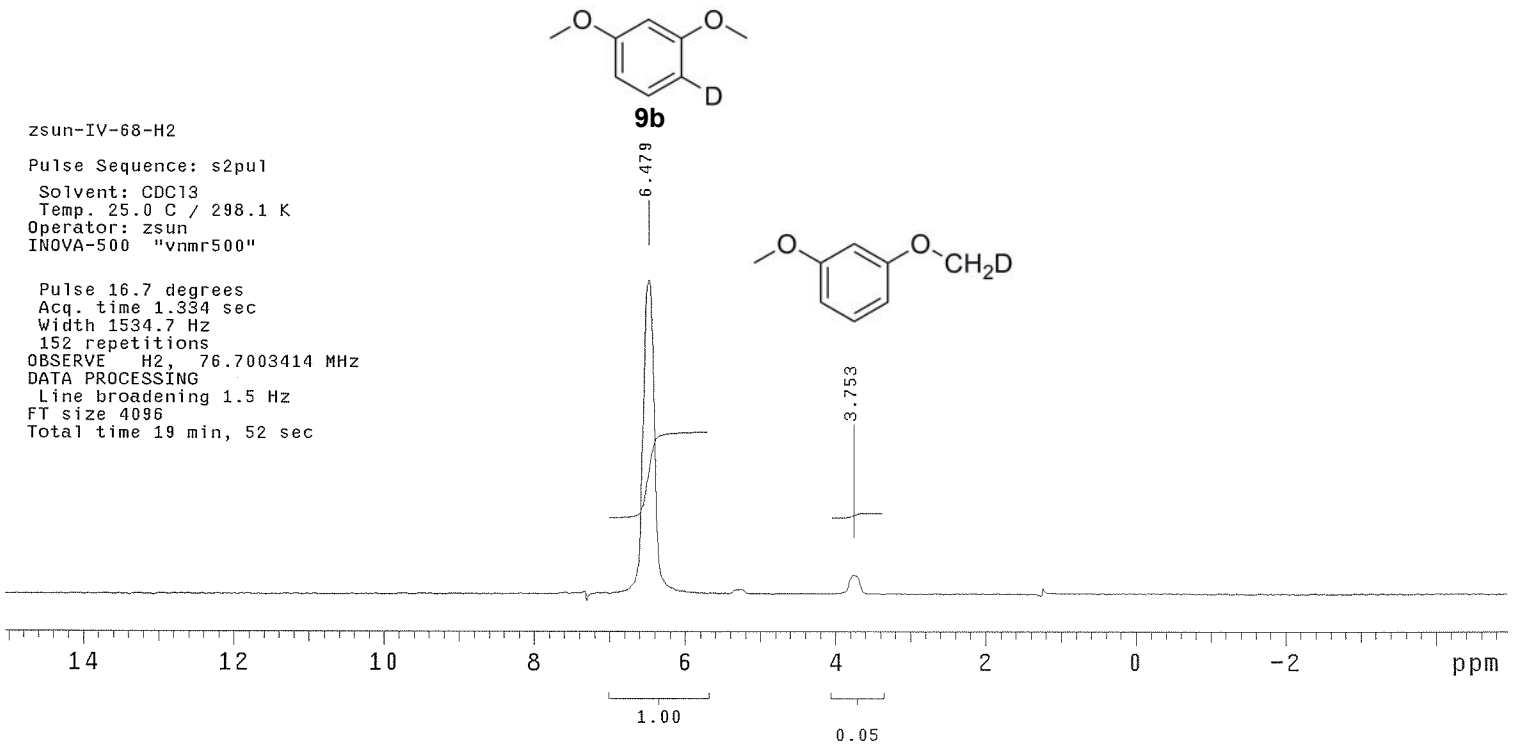
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



9b



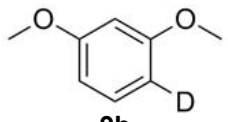

9b
zsun-IV-68-H2
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: zsun
INOVA-500 "vnmr500"
Pulse 16.7 degrees
Acq. time 1.334 sec
Width 1534.7 Hz
152 repetitions
OBSERVE H2, 76.7003414 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 4096
Total time 19 min, 52 sec
6.479
3.753
1.00
0.05
14
12
10
8
6
4
2
0
-2
ppm

zsun-IV-68-C13

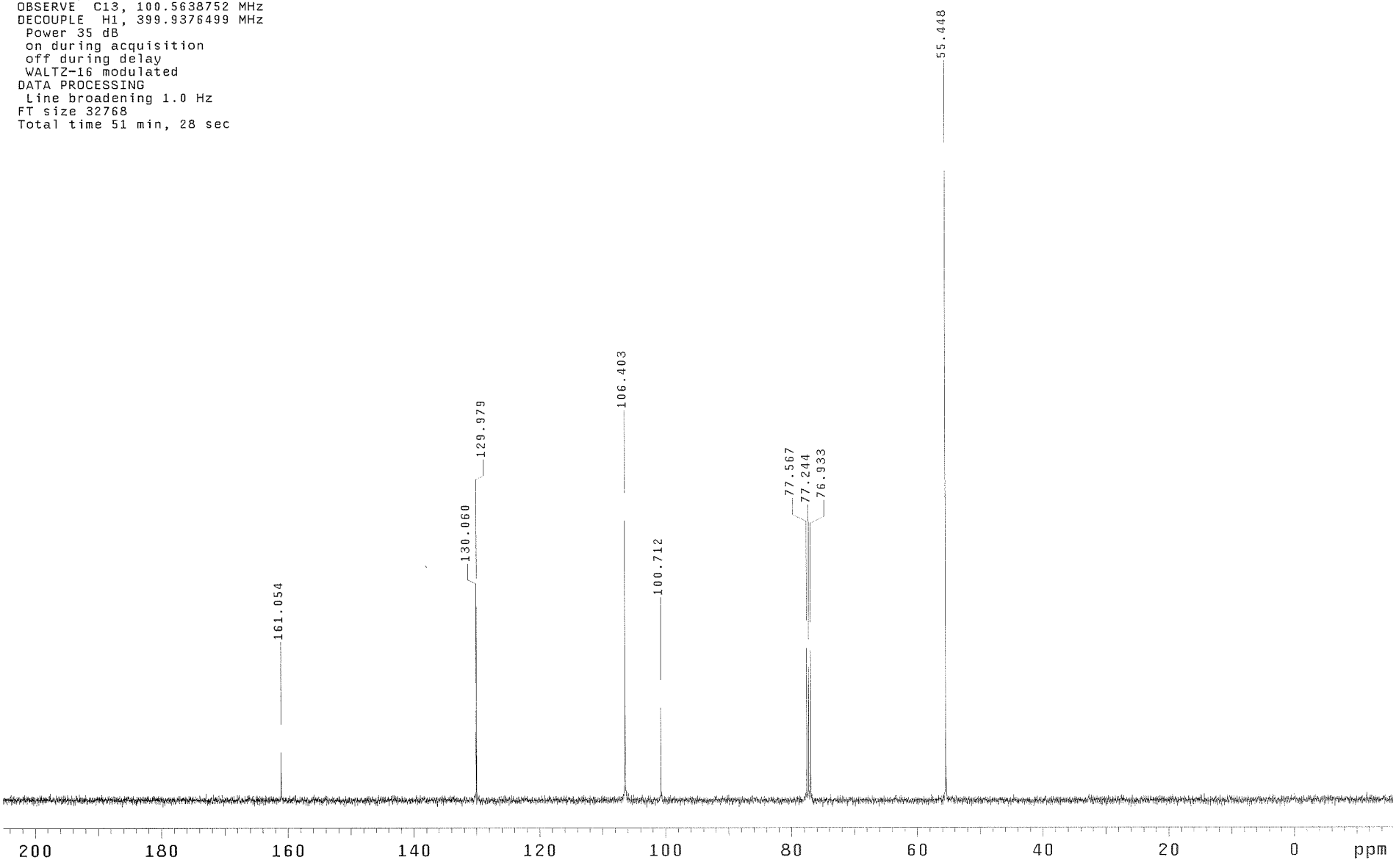
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.720 sec
Width 22222.2 Hz
268 repetitions
OBSERVE C13, 100.5638752 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 51 min, 28 sec



9b

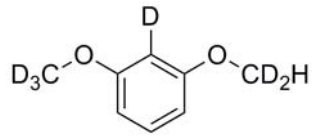


zsun-iv-79-H1

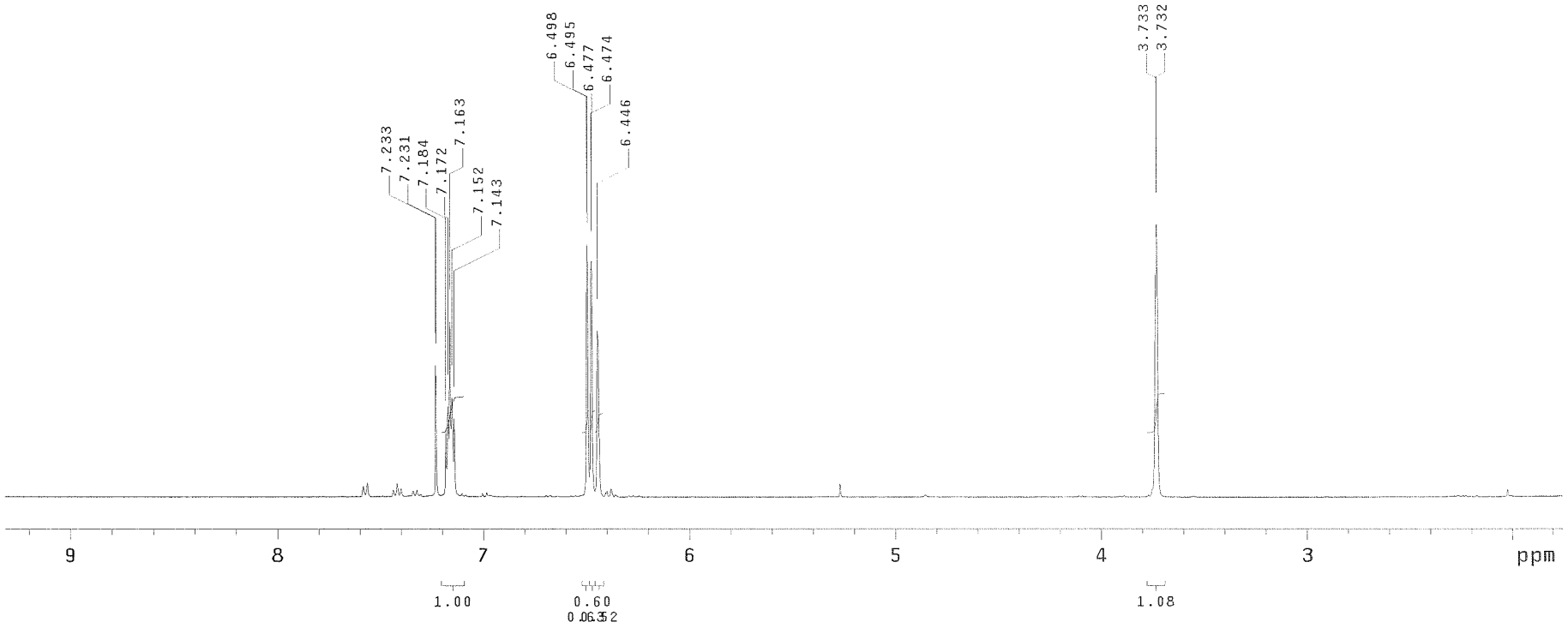
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



9c

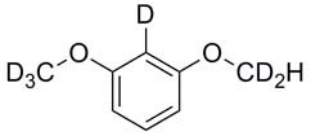


zsun-iv-79-H1

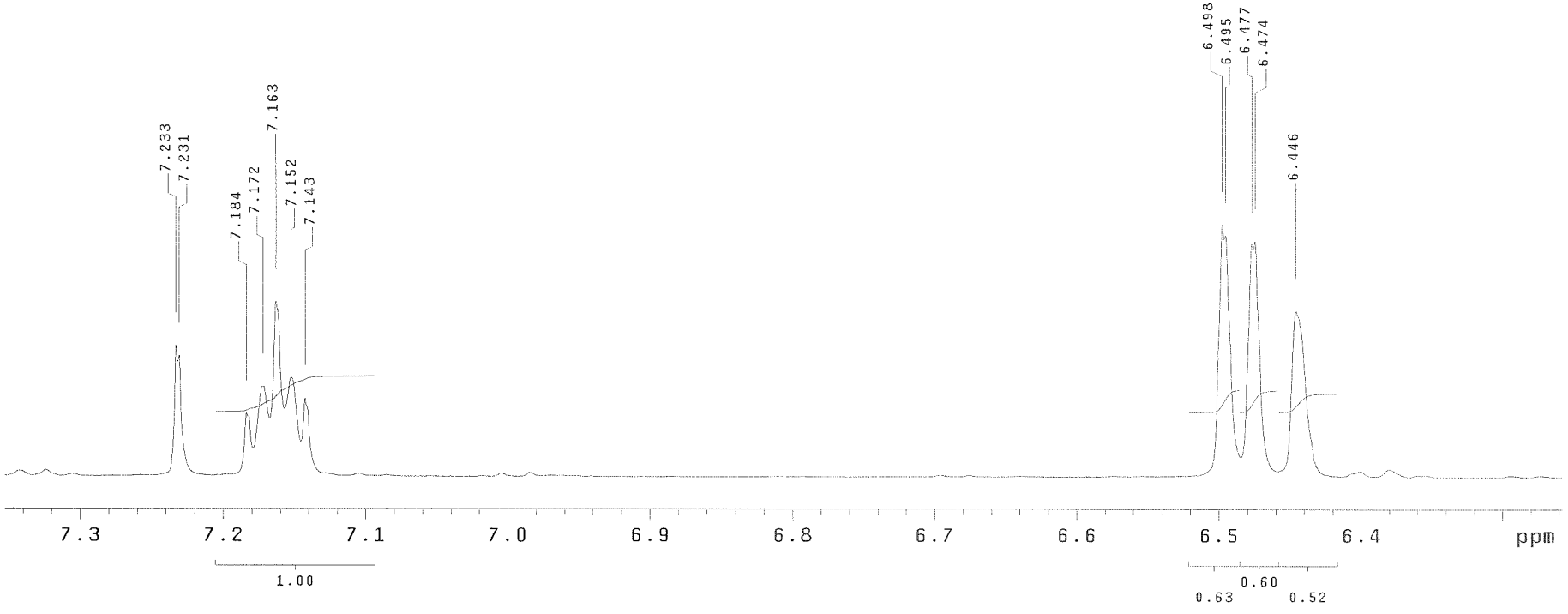
Pulse Sequence: s2pul

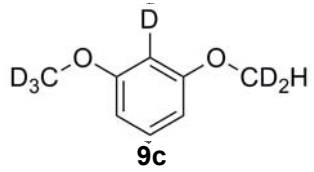
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec

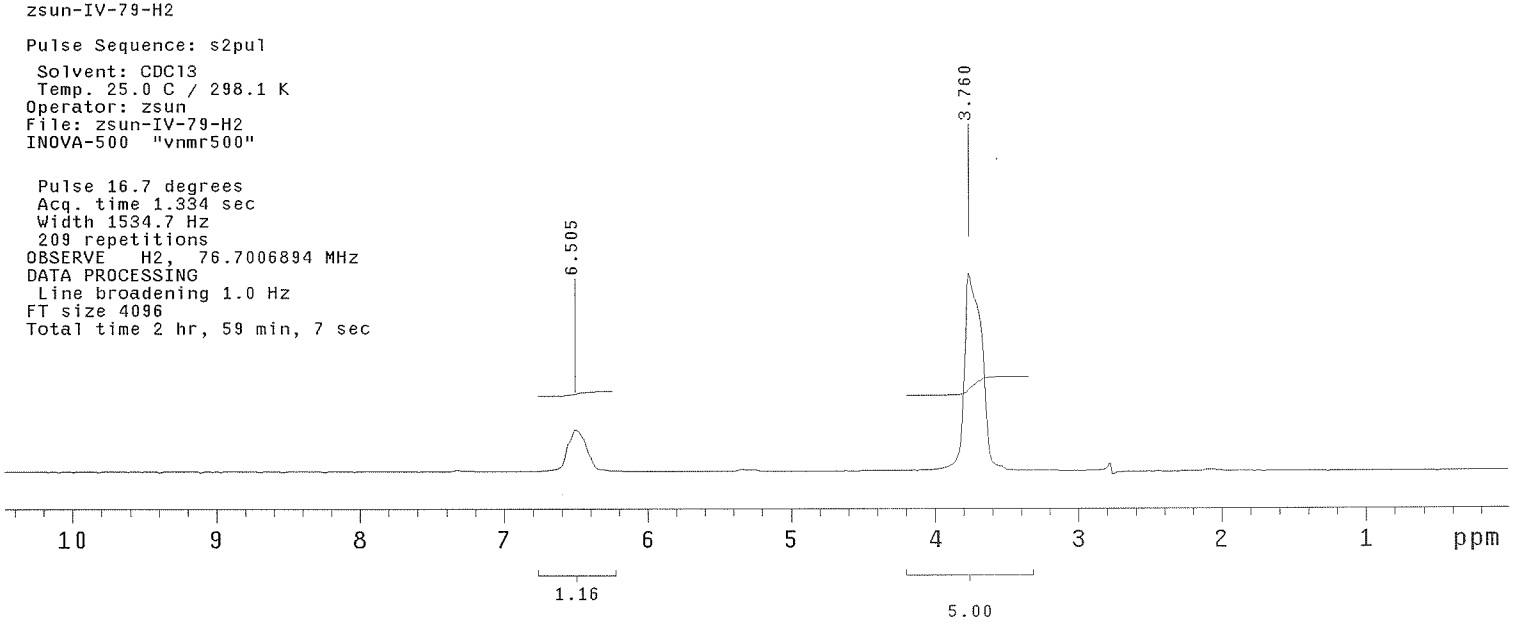


9c




D
D_3C
O
O
CD_2H

9c

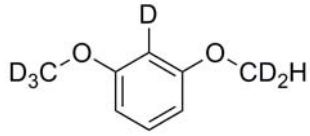

zsun-IV-79-H2
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: zsun
File: zsun-IV-79-H2
INOVA-500 "vnmr500"
Pulse 16.7 degrees
Acq. time 1.334 sec
Width 1534.7 Hz
209 repetitions
OBSERVE H2, 76.7006894 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 4096
Total time 2 hr, 59 min, 7 sec
6.505
3.760
10
9
8
7
6
5
4
3
2
1
ppm
1.16
5.00

zsun-IV-79-C13

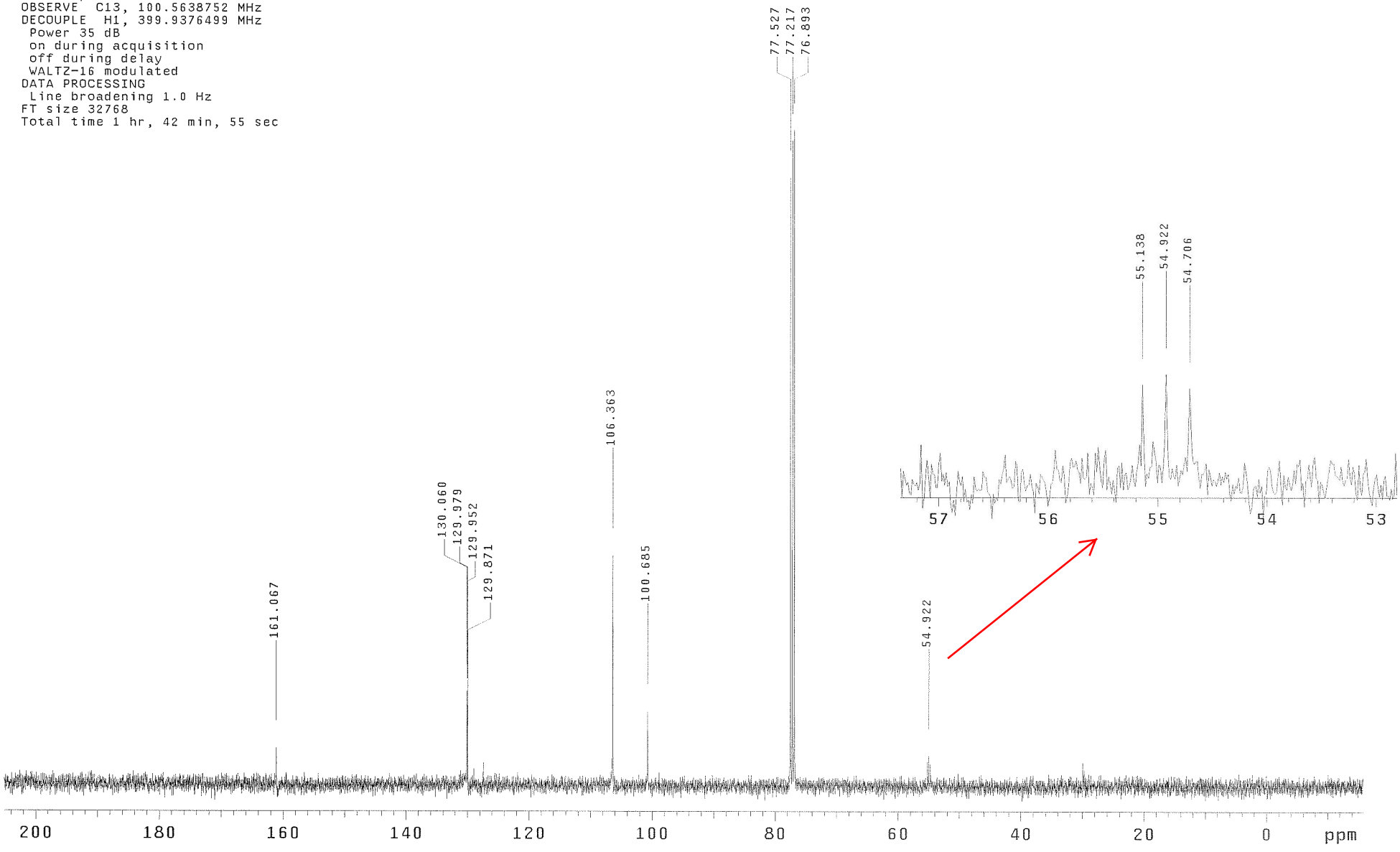
Pulse Sequence: s2pul

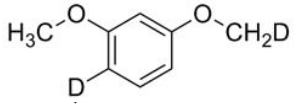
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.720 sec
Width 22222.2 Hz
728 repetitions
OBSERVE C13, 100.5638752 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 1 hr, 42 min, 55 sec

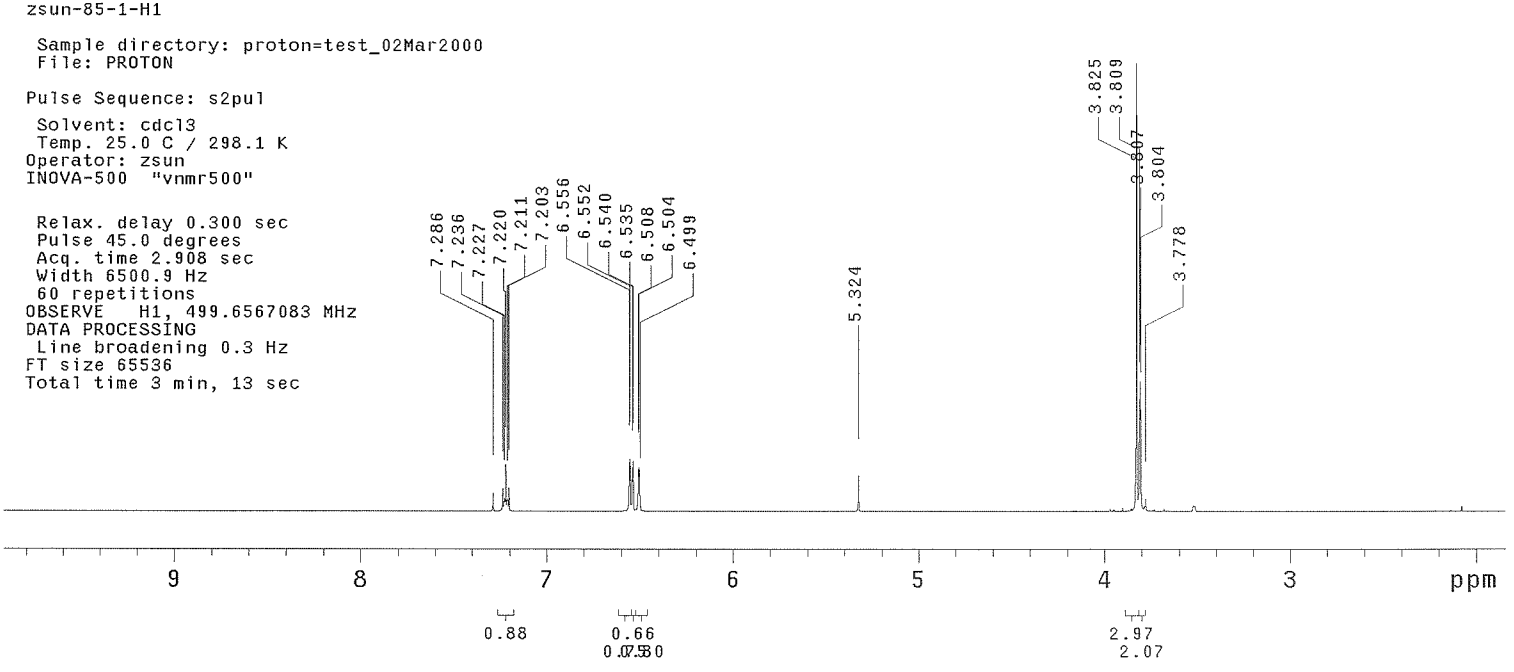


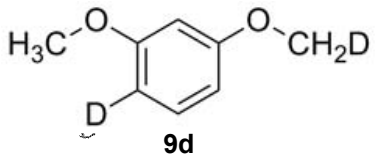
9c




H3C
O
O
CH2D
D

9d


zsun-85-1-H1
Sample directory: proton=test_02Mar2000
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: zsun
INOVA-500 "vnmr500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 2.908 sec
Width 6500.9 Hz
60 repetitions
OBSERVE H1, 499.6567083 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 3 min, 13 sec
7.286
7.236
7.227
7.220
7.211
7.203
6.556
6.552
6.540
6.535
6.508
6.504
6.499
5.324
3.825
3.809
3.807
3.804
3.778
9
8
7
6
5
4
3
ppm
0.88
0.66
2.97
2.07



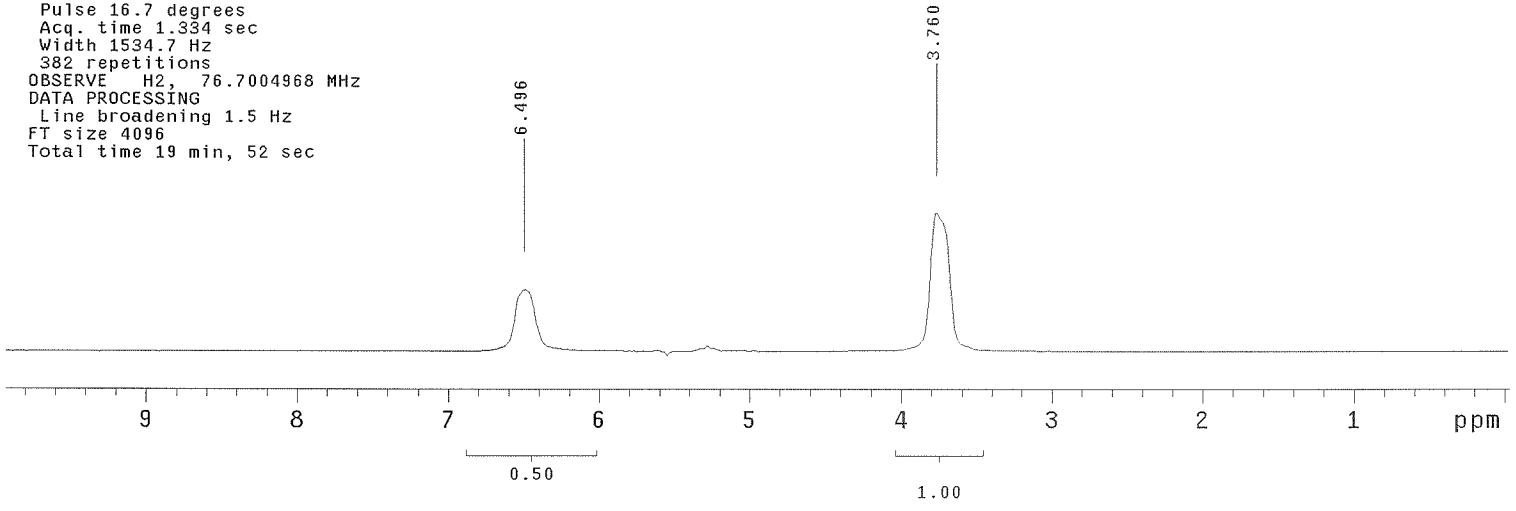
9d

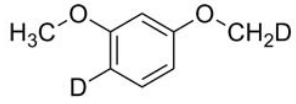
zsun-IV-85-1-H2

Pulse Sequence: s2pul

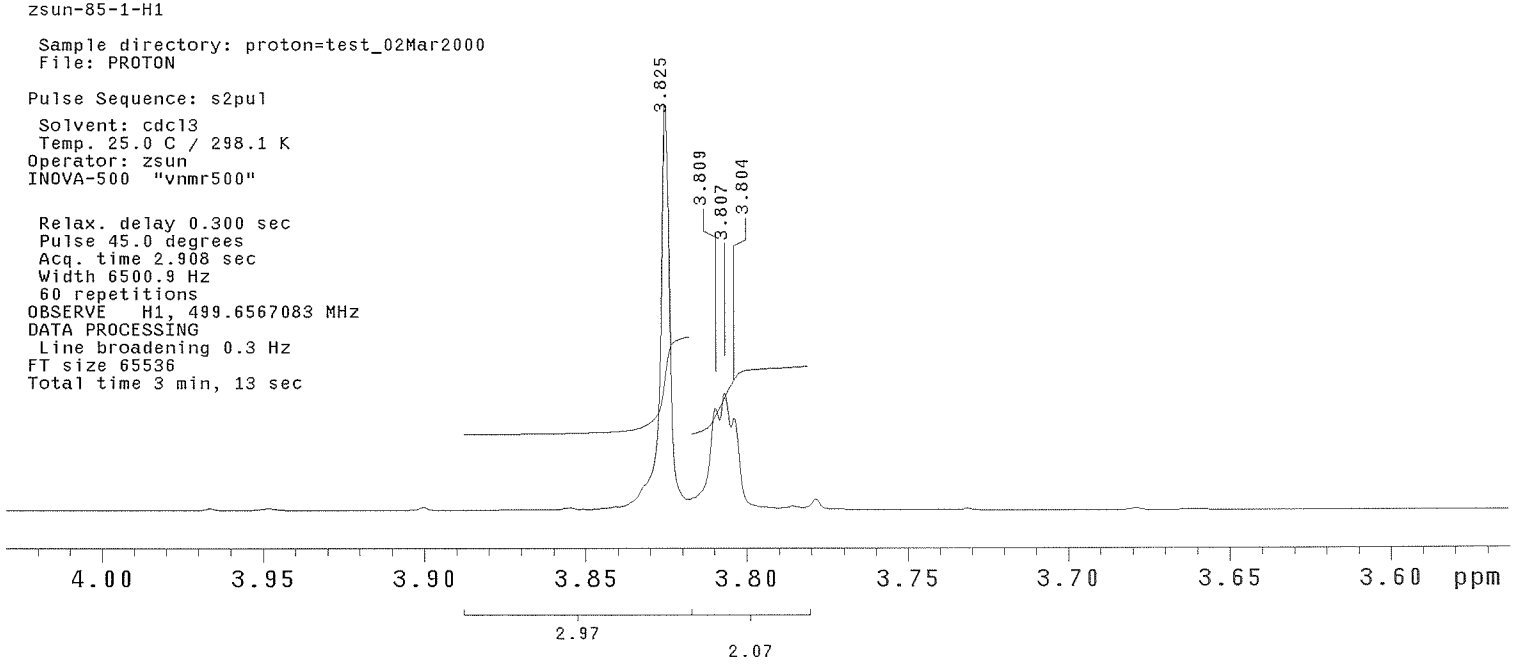
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: zsun
INOVA-500 "vnmr500"

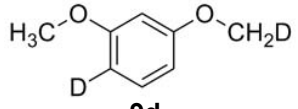
Pulse 16.7 degrees
Acq. time 1.334 sec
Width 1534.7 Hz
382 repetitions
OBSERVE H2, 76.7004968 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 4096
Total time 19 min, 52 sec




H3C
O
O
CH2D
D

9d


zsun-85-1-H1
Sample directory: proton=test_02Mar2000
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: zsun
INOVA-500 "vnmr500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 2.908 sec
Width 6500.9 Hz
60 repetitions
OBSERVE H1, 499.6567083 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 3 min, 13 sec
3.825
3.809
3.807
3.804
4.00
3.95
3.90
3.85
3.80
3.75
3.70
3.65
3.60
ppm
2.97
2.07



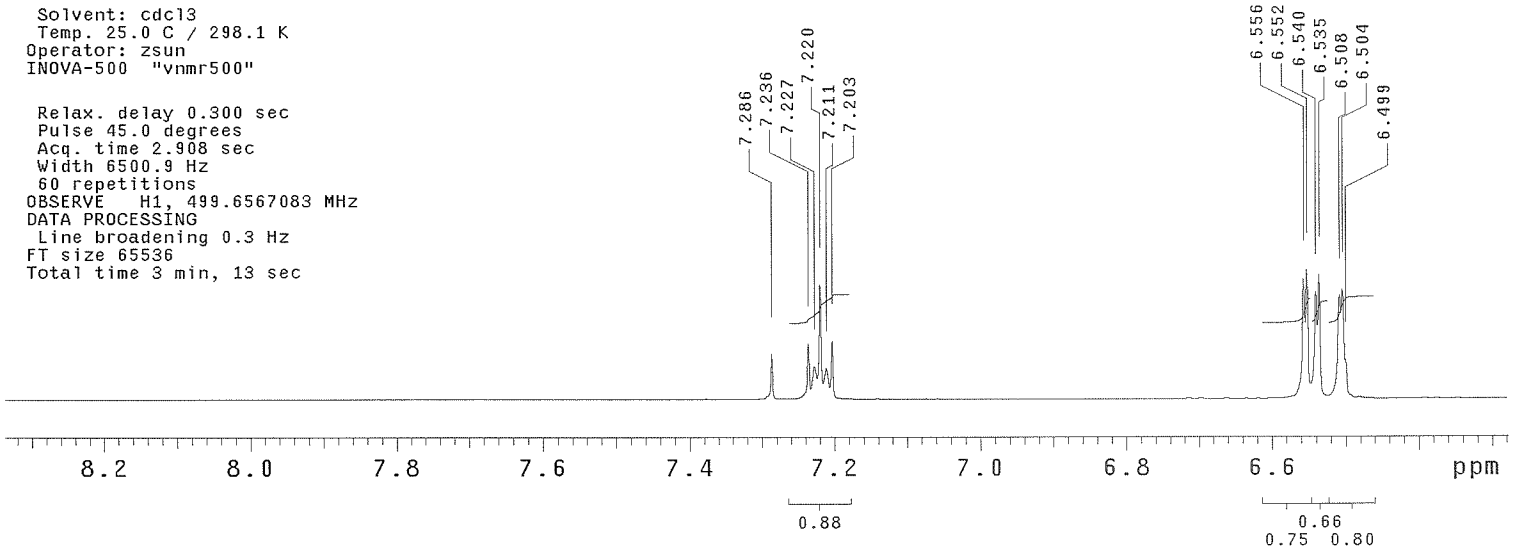
9d

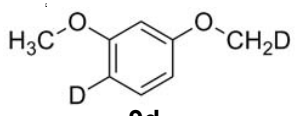
```
zsun-85-1-H1

 Sample directory: proton=test_02Mar2000
 File: PROTON

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: zsun
INOVA-500  "vnmr500"

 Relax. delay 0.300 sec
 Pulse 45.0 degrees
 Acq. time 2.908 sec
 Width 6500.9 Hz
 60 repetitions
OBSERVE   H1, 499.6567083 MHz
DATA PROCESSING
 Line broadening 0.3 Hz
FT size 65536
Total time 3 min, 13 sec
```





9d

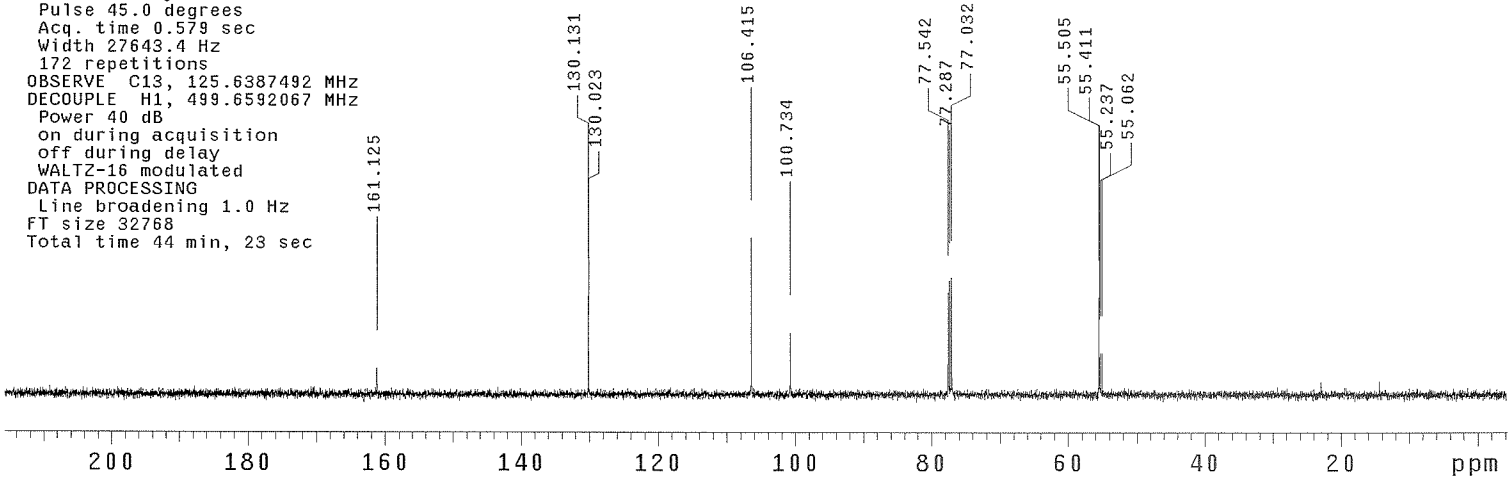
zsun-IV-85-1-C13

Sample directory: eb-chloroform_01Mar2000
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: zsun
INOVA-500 "vnmr500"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.579 sec
Width 27643.4 Hz
172 repetitions
OBSERVE C13, 125.6387492 MHz
DECOUPLE H1, 499.6592067 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 44 min, 23 sec

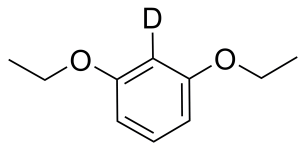


zsun-IV-60-2-H1

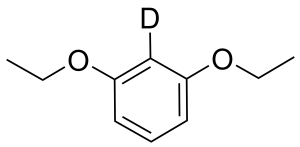
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

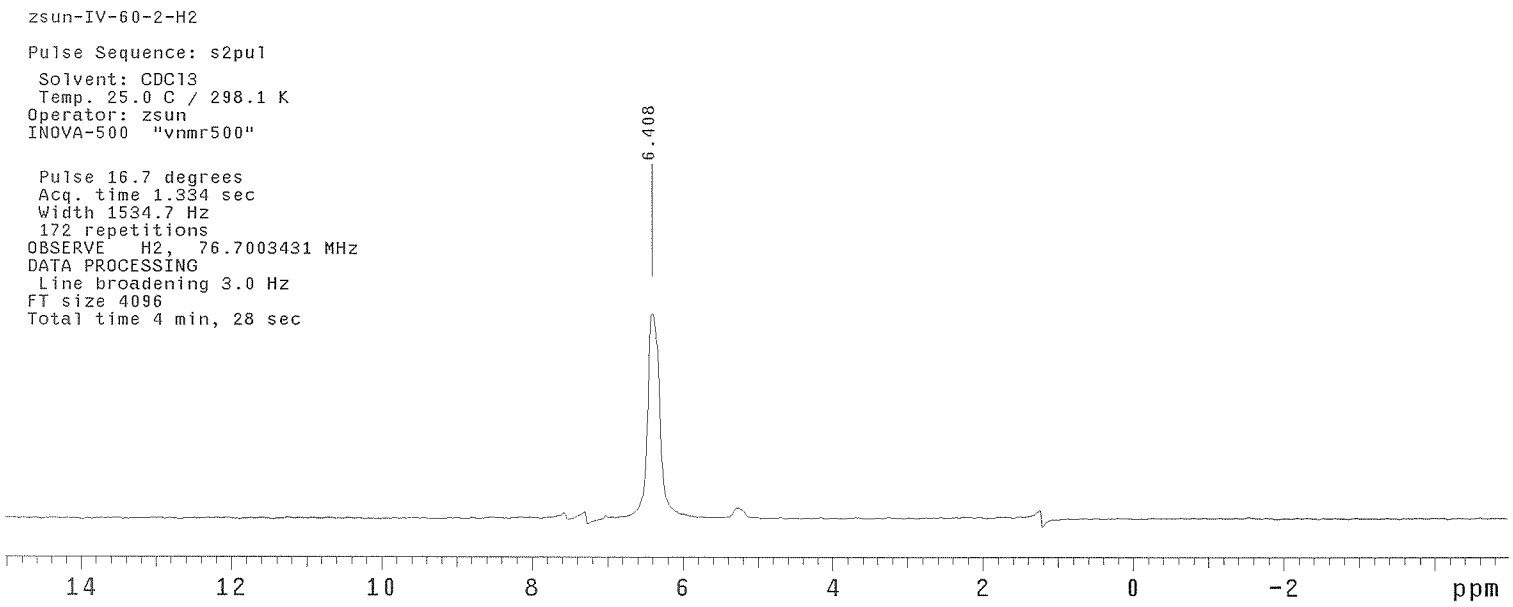
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



11


D
O
O

11

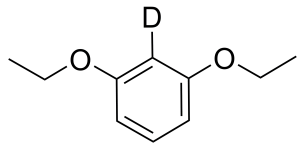

zsun-IV-60-2-H2
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: zsun
INOVA-500 "vnmr500"
Pulse 16.7 degrees
Acq. time 1.334 sec
Width 1534.7 Hz
172 repetitions
OBSERVE H2, 76.7003431 MHz
DATA PROCESSING
Line broadening 3.0 Hz
FT size 4096
Total time 4 min, 28 sec
6.408
14
12
10
8
6
4
2
0
-2
ppm

zsun-IV-60-2-C13

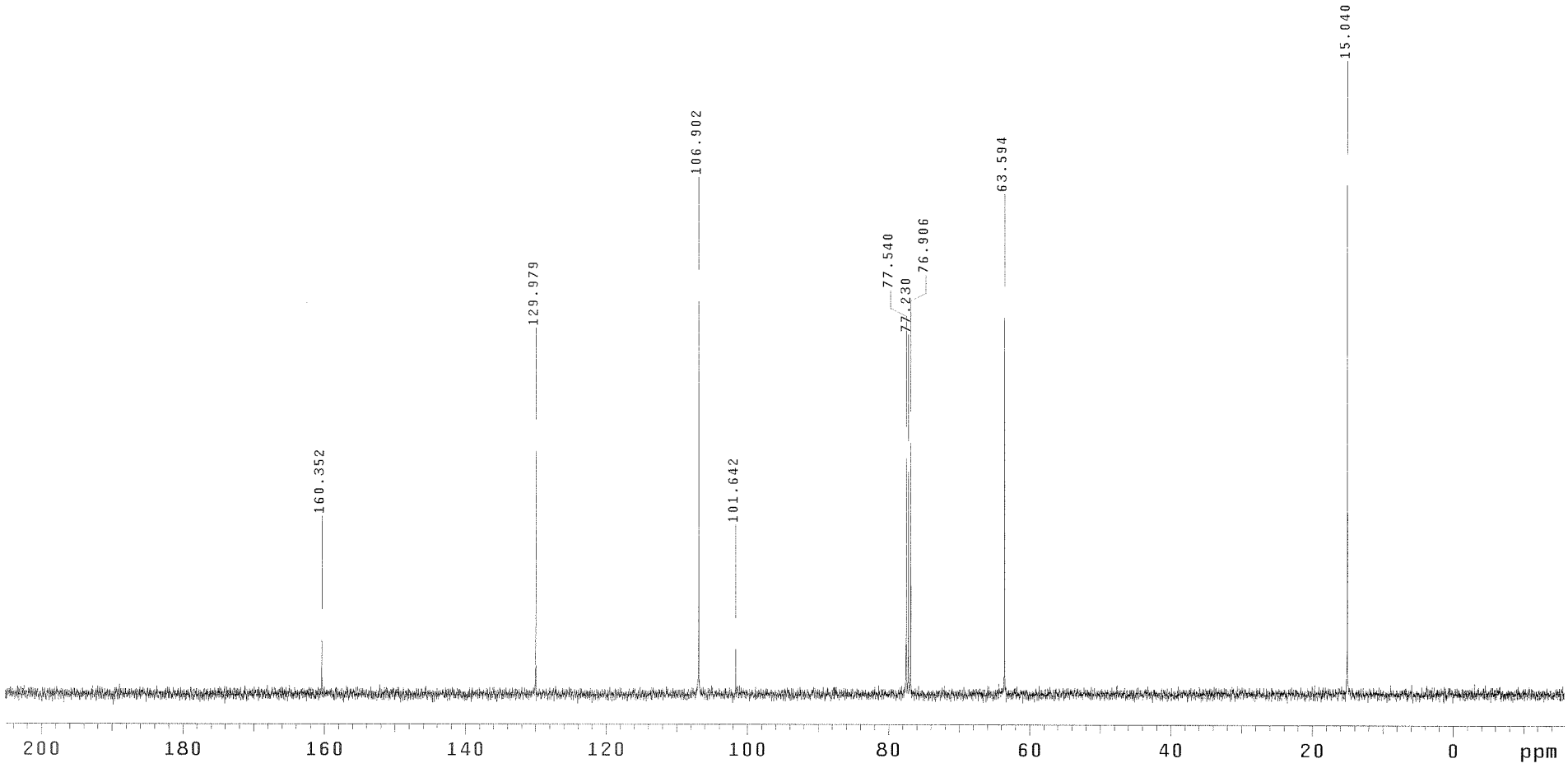
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.720 sec
Width 22222.2 Hz
384 repetitions
OBSERVE C13, 100.5638752 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 1 hr, 8 min, 37 sec



11

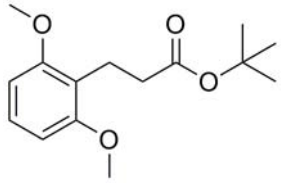


zsun-III-163-1-side-product

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



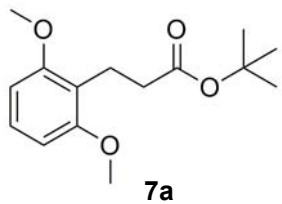
7a

zsun-III-163-1-side-product-C13

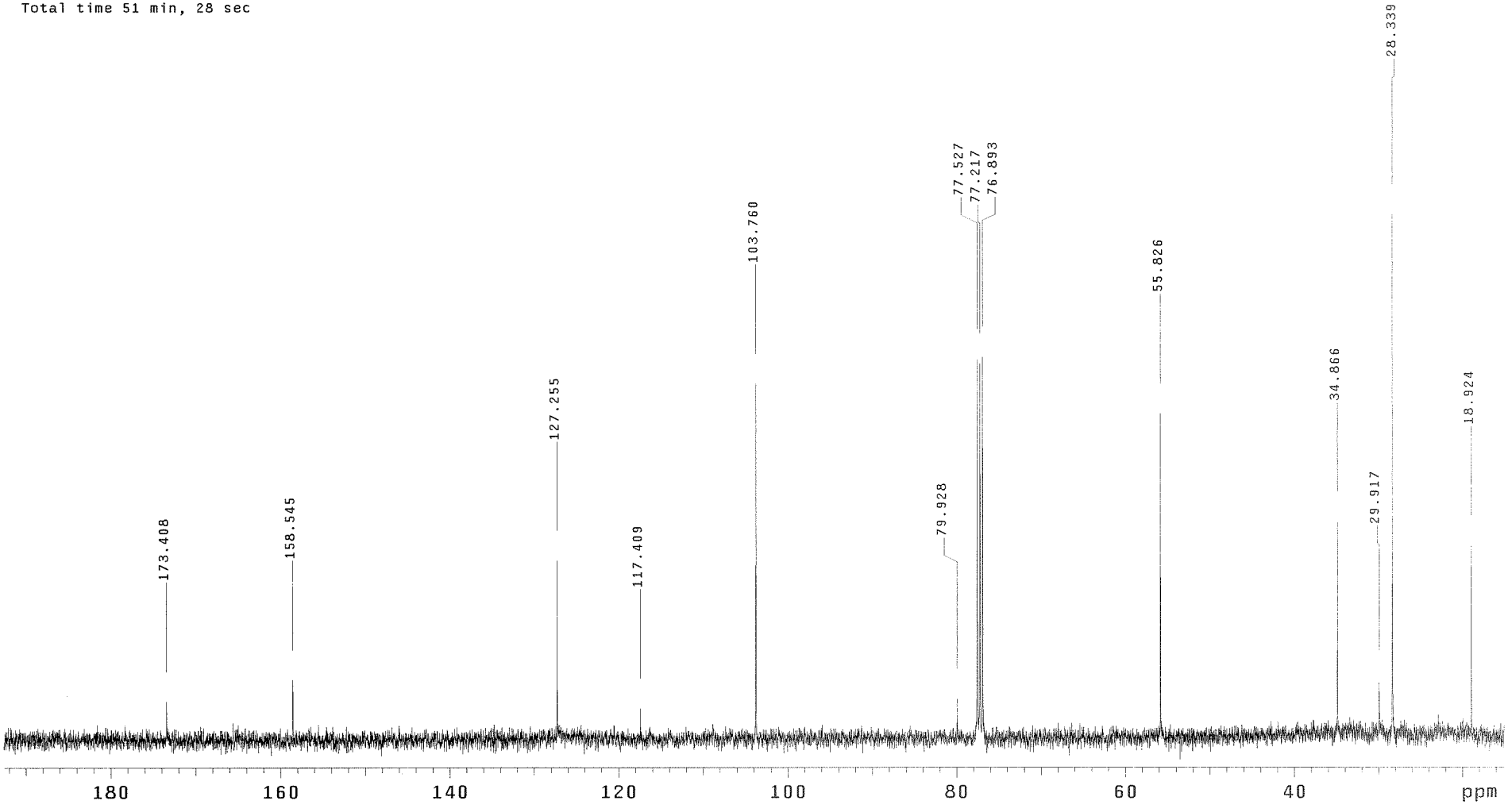
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.720 sec
Width 22222.2 Hz
476 repetitions
OBSERVE C13, 100.5638752 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 51 min, 28 sec

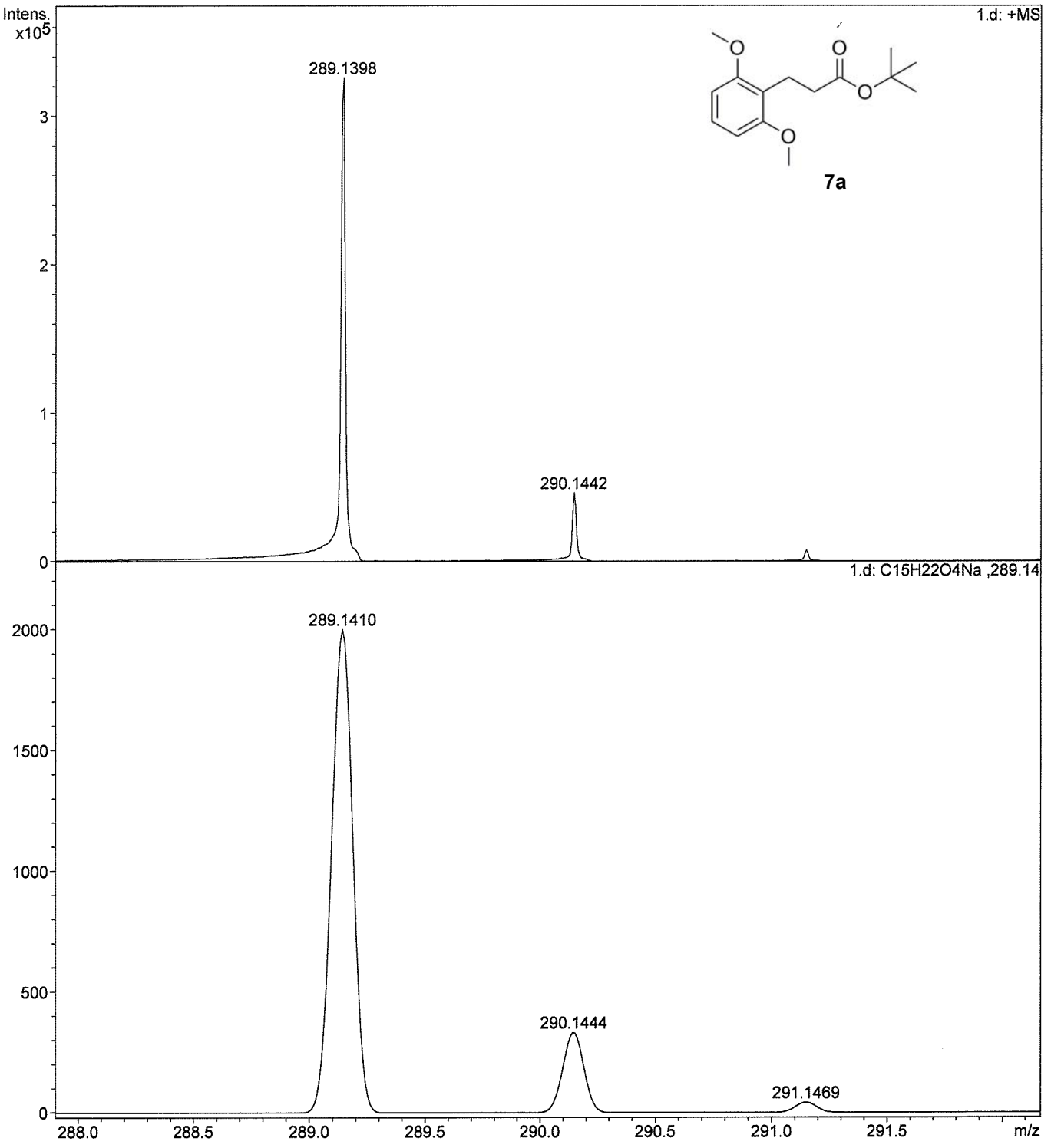


7a



Generic Display Report

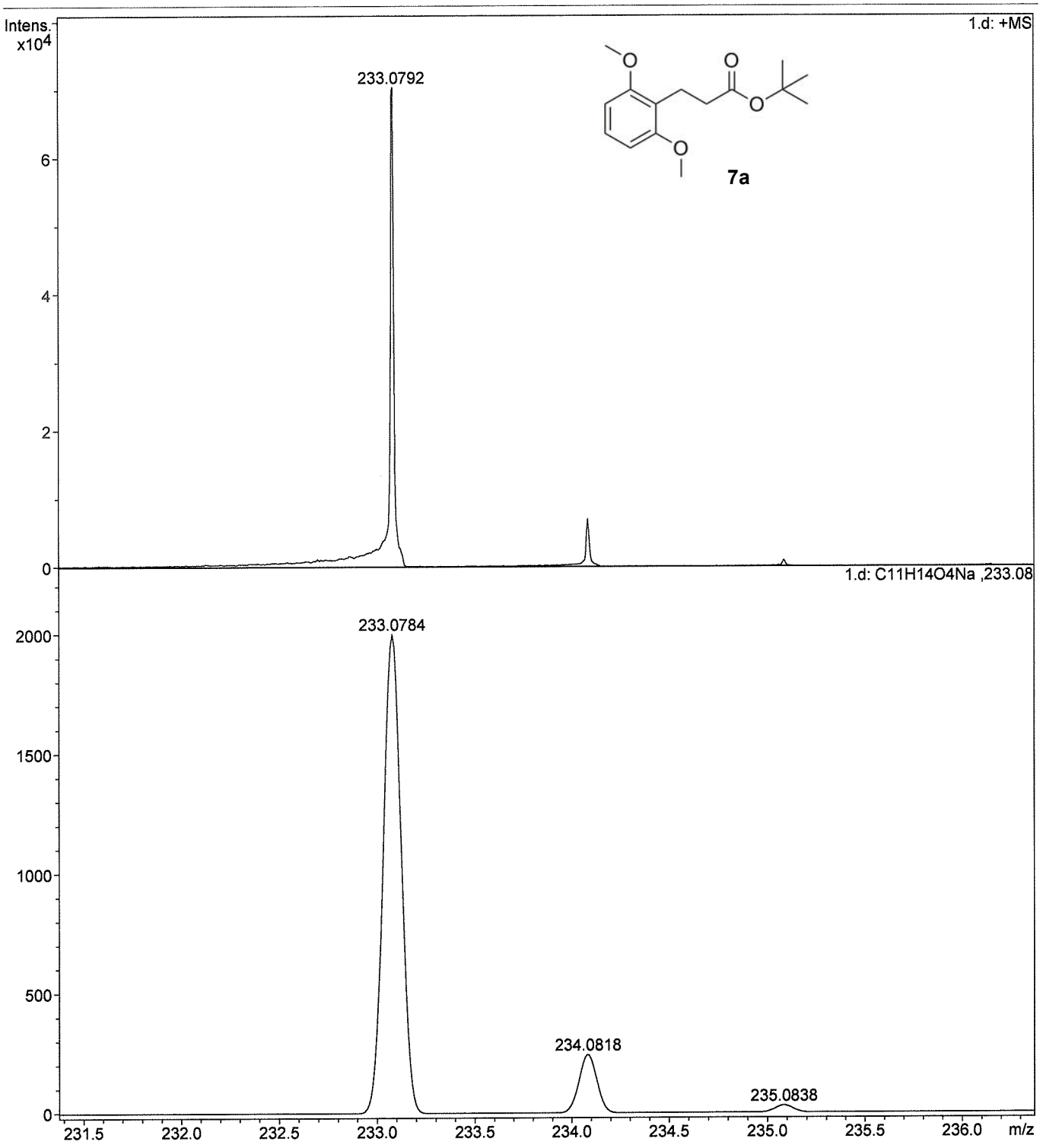
Analysis Info		Acquisition Date	12/9/2009 2:00:50 PM
Analysis Name	D:\Bruker\data\zhosun\I-201b\1.d		
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	I-201b	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		



Generic Display Report

Analysis Info

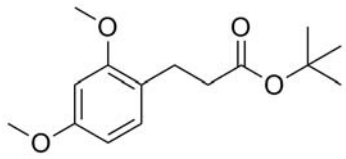
Analysis Name	D:\Bruker\data\zhosun\I-201b\1.d	Acquisition Date	12/9/2009 2:00:50 PM
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	I-201b	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		



zsun-II-56-H1

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: zsun-II-56-H1
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356399 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



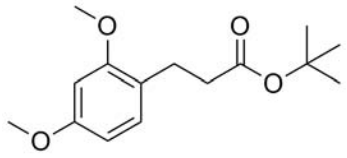
8a

zsun-II-56-H1

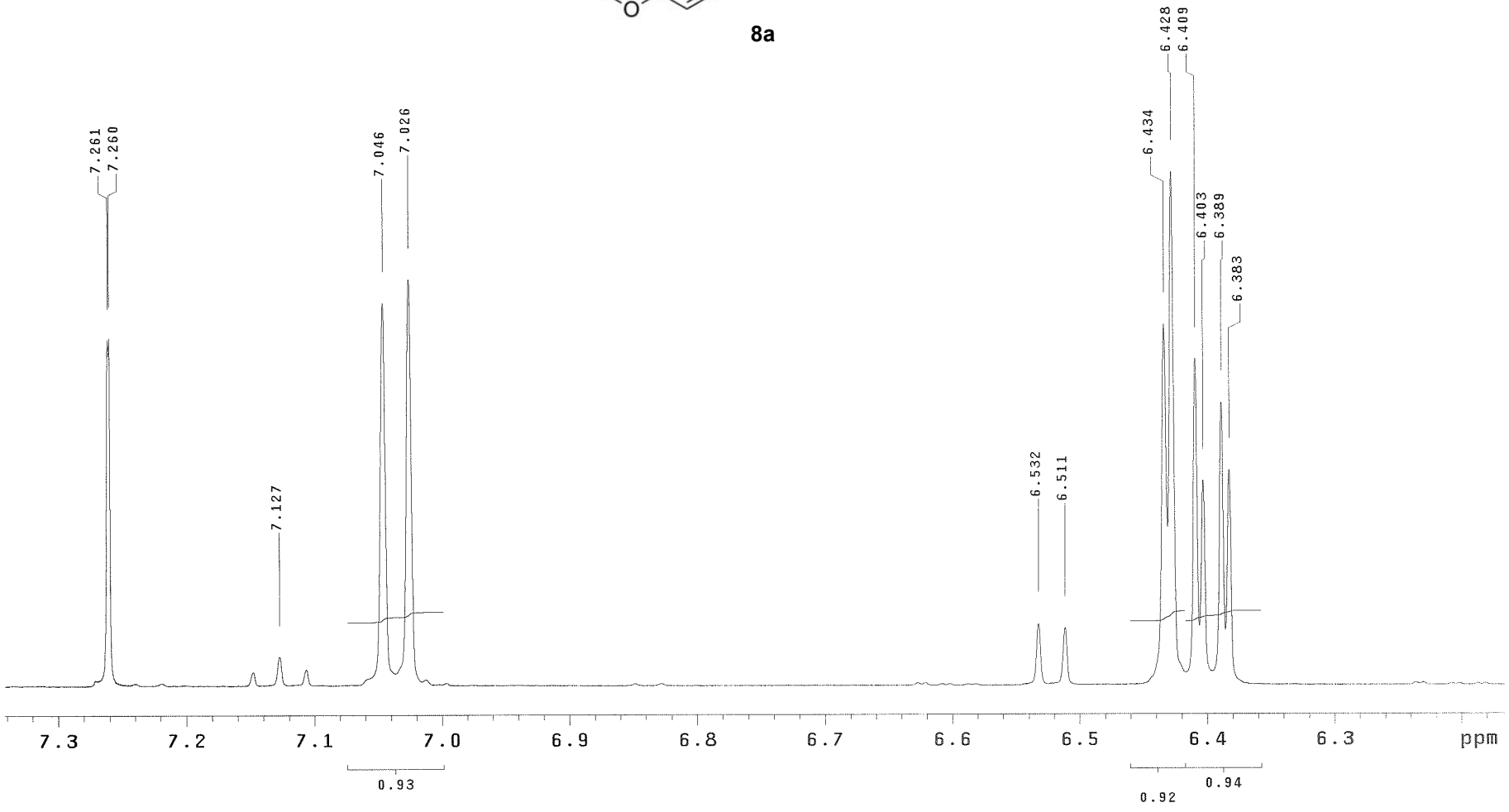
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: zsun-II-56-H1
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356399 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



8a

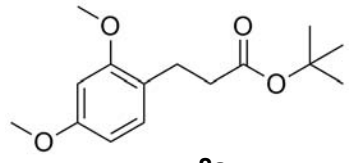


zsun-II-56-H1

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: zsun-II-56-H1
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 3.334 sec
Width 4662.0 Hz
60 repetitions
OBSERVE H1, 399.9356399 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec

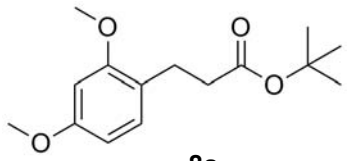


8a

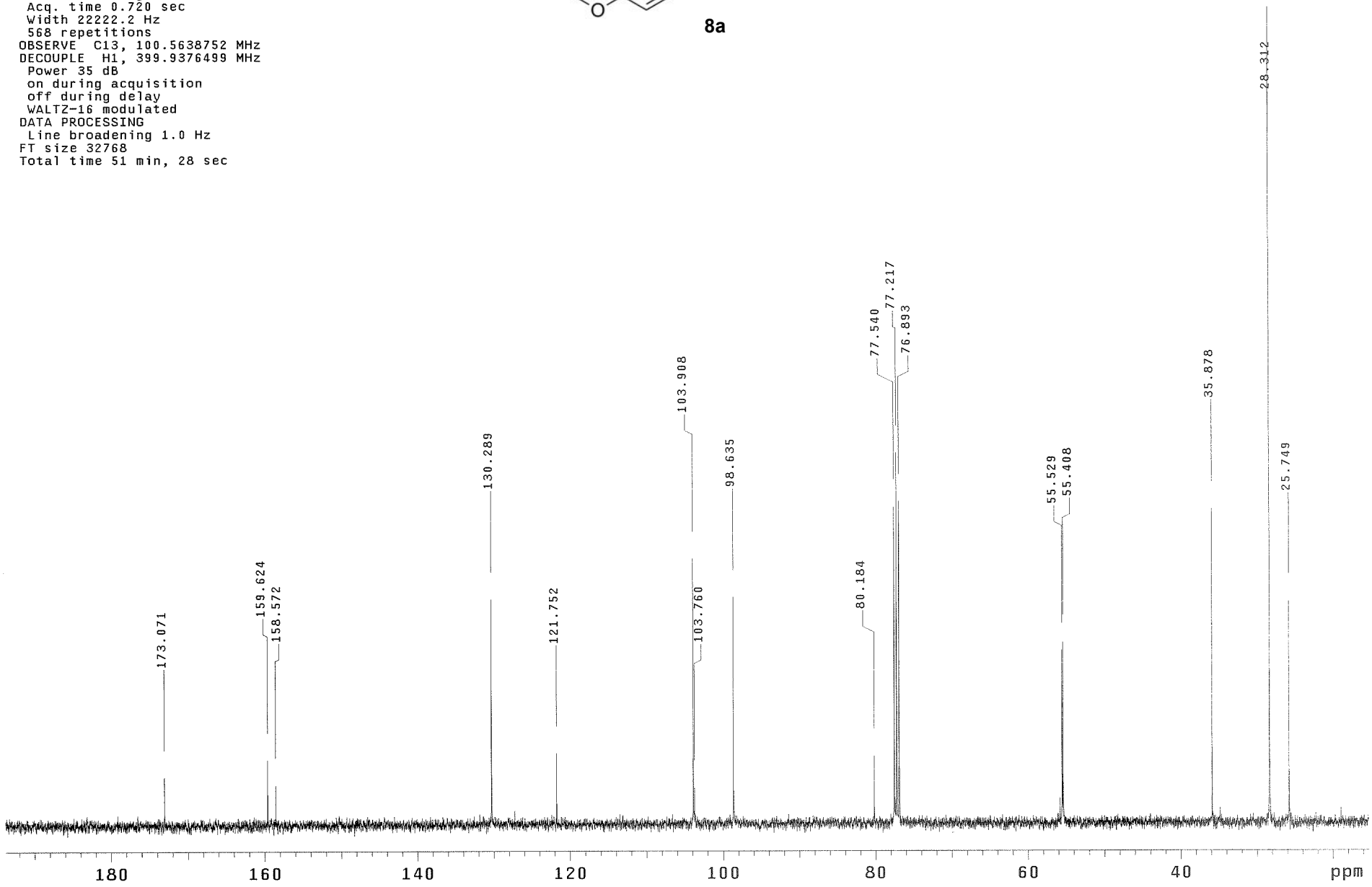
zsun-II-56-C13

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-400 "vnmr400"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.720 sec
Width 22222.2 Hz
568 repetitions
OBSERVE C13, 100.5638752 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 51 min, 28 sec

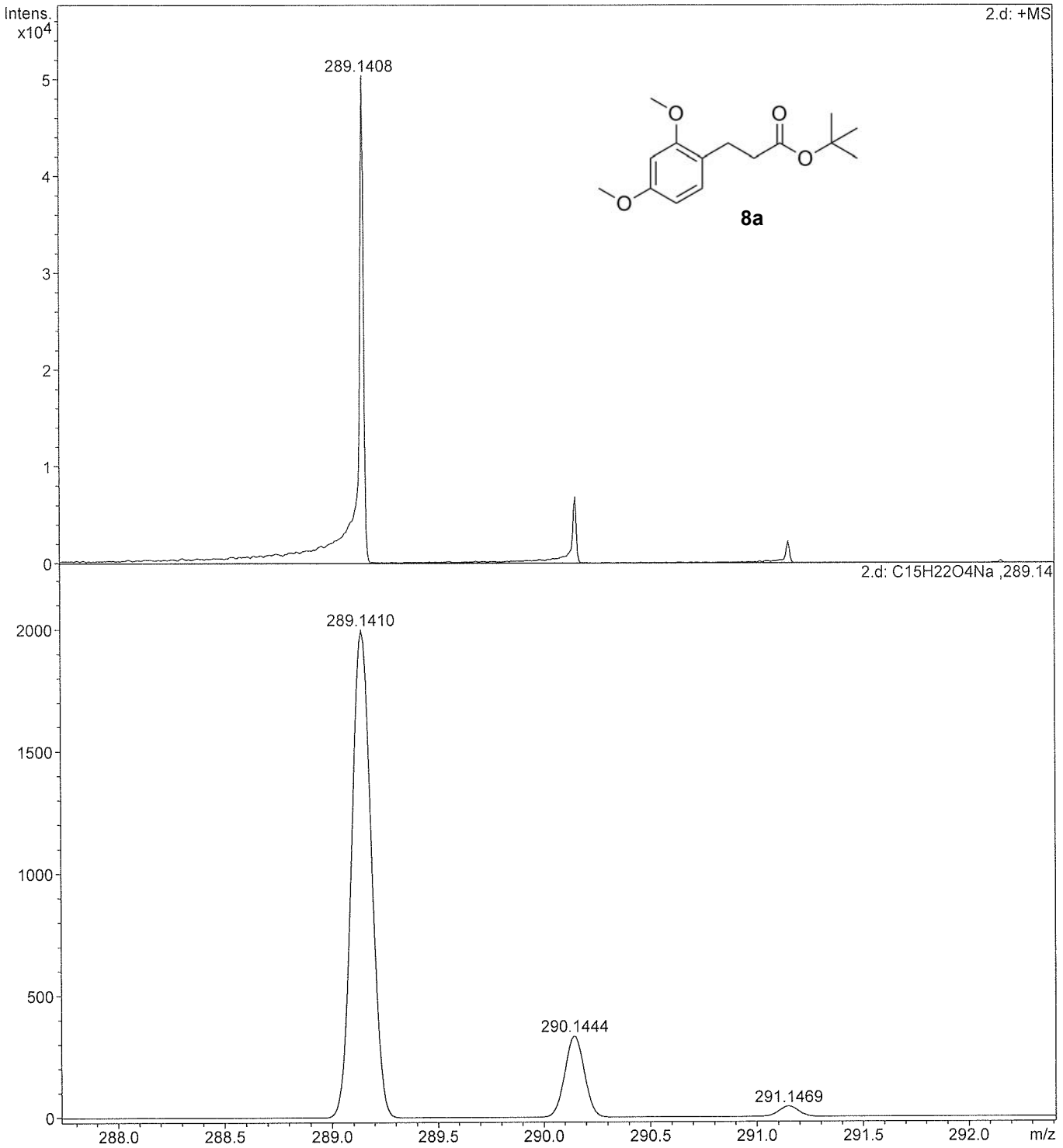


8a



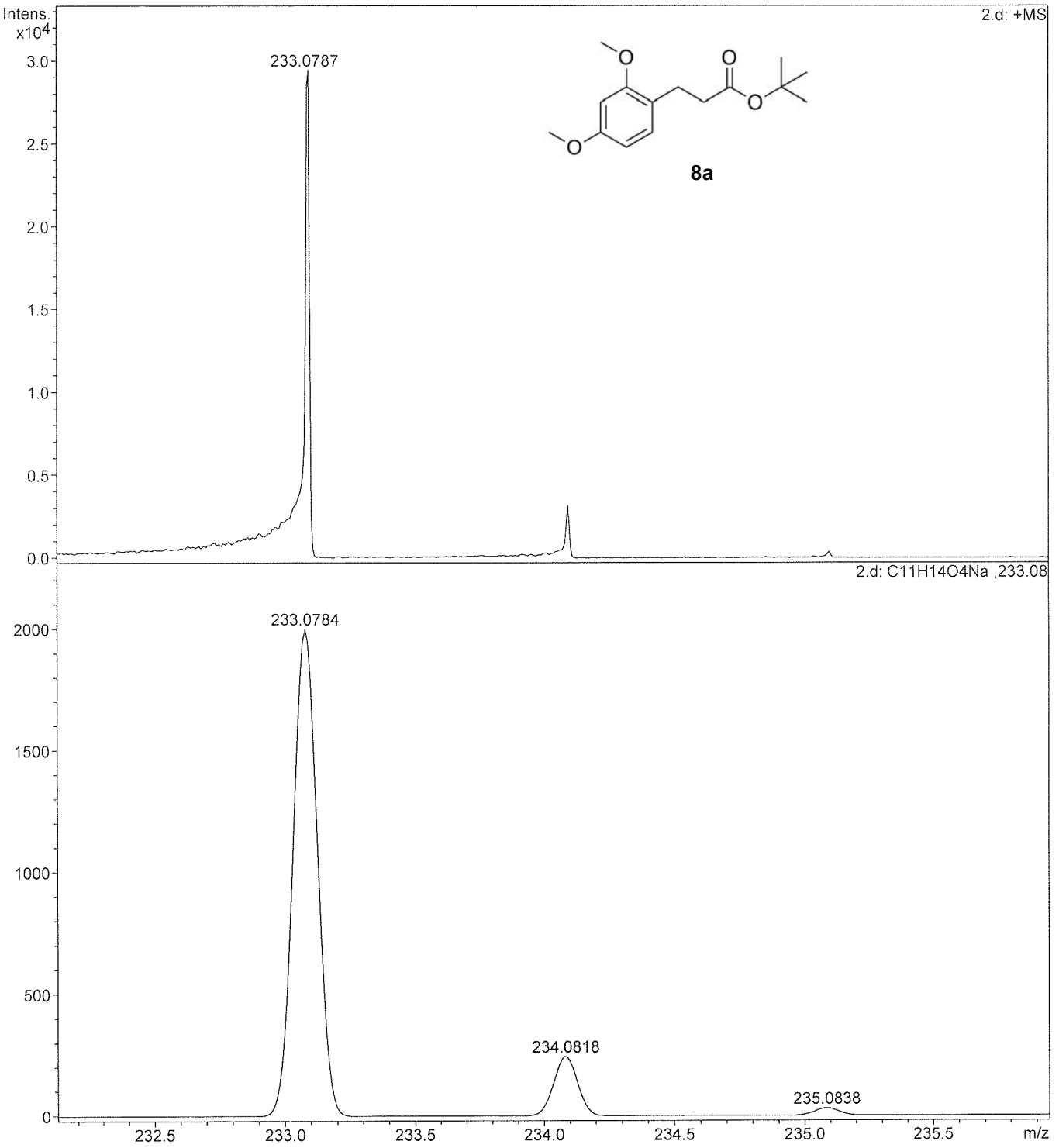
Generic Display Report

Analysis Info		Acquisition Date	8/27/2009 5:02:58 PM
Analysis Name	D:\Bruker\data\zhosun\II-56-standard\2.d		
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	II-56-standard	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		



Generic Display Report

Analysis Info			
Analysis Name	D:\Bruker\data\zhosun\II-56-standard\2.d	Acquisition Date	8/27/2009 5:02:58 PM
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	II-56-standard	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		



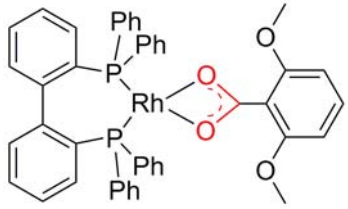
zsun-II-155a-31P

Sample directory: P-31-1903_13Mar2003

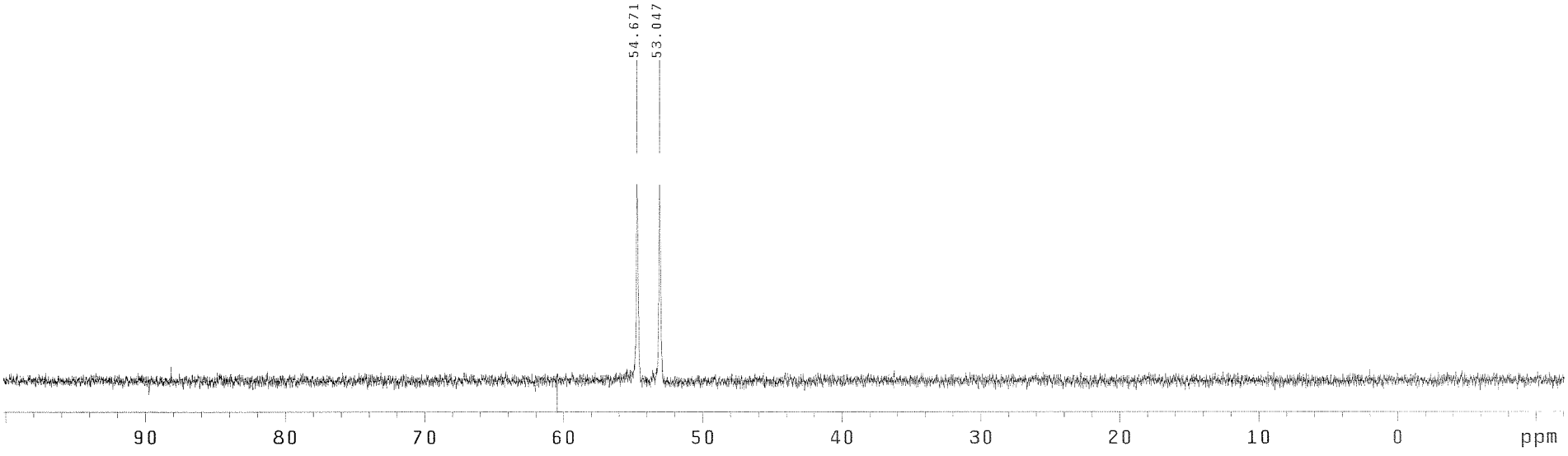
Pulse Sequence: s2pul

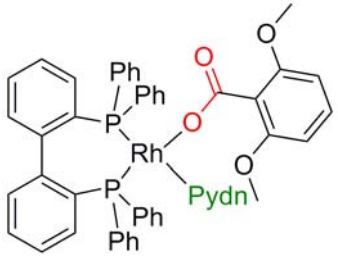
Solvent: CDCl3
Ambient temperature
Operator: zsun
File: zsun-II-155a-31P
Mercury-300BB "vnmr300"

Relax. delay 1.000 sec
Pulse 35.0 degrees
Acq. time 1.600 sec
Width 18214.9 Hz
120 repetitions
OBSERVE P31, 121.4927885 MHz
DECOUPLE H1, 300.1391844 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 23 min, 38 sec



2b

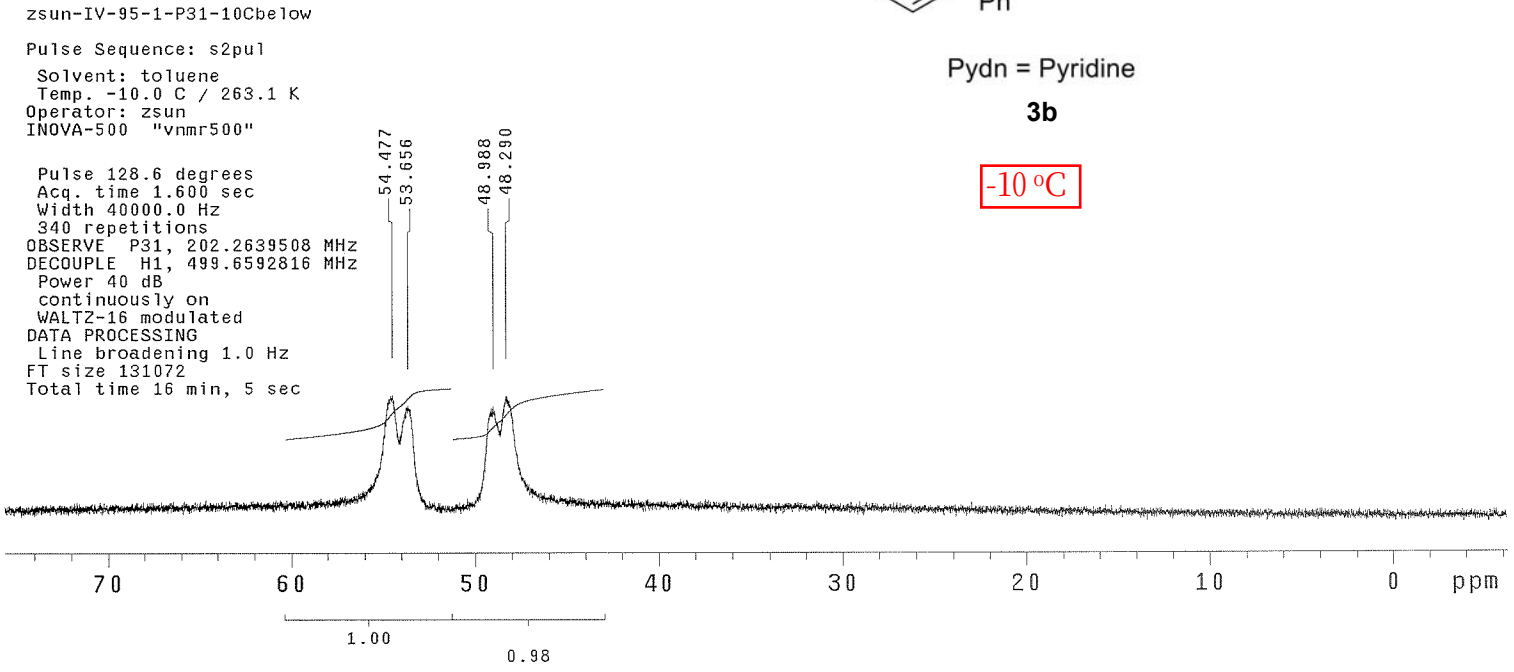


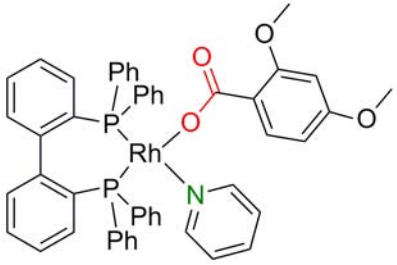


Pydn = Pyridine

3b

-10 °C





4b

-10 °C

zsun-IV-95-2-P31-below10C

Pulse Sequence: s2pul

Solvent: toluene
Temp. -10.0 C / 263.1 K
Operator: zsun
INOVA-500 "vnmr500"

Pulse 128.6 degrees
Acq. time 1.600 sec
Width 40000.0 Hz
520 repetitions
OBSERVE P31, 202.2637320 MHz
DECOUPLE H1, 499.6592816 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 40 min, 53 sec