Supporting Information

Rh(I)-Catalyzed Decarboxylative Transformations of Arene-Carboxylic Acids: Ligand- and Reagent-Controlled Selectivity towards Hydrodecarboxylation or Heck-Mizoroki Products

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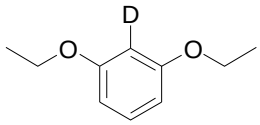
General. Unless otherwise noted, all manipulations were carried out under a nitrogen atmosphere using standard Schlenk-line or glovebox techniques. All glassware was oven-dried for at least 1 h prior to use. THF, diethyl ether, toluene, benzene, hexane and pentane were degassed by purging with nitrogen for 45 min and dried with a solvent purification system (MBraun MB-SPS). Pyridine, C_6D_6, C_7D_8 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 a Shimadzu GC-2010 with *n*-dodecane as the internal standard. ^{1}H NMR spectra were obtained on a 400 MHz spectrometer, and chemical shifts were recorded relative to residual protiated solvent. ^{13}C NMR spectra were obtained at 100 MHz, and chemical shifts were recorded to the solvent resonance. Both ^{1}H and ^{13}C NMR chemical shifts were reported in parts per million downfield from tetramethylsilane ($\delta = 0$). ^{19}F NMR spectra were obtained at 282.4 MHz, and all chemical shifts were reported in parts per million upfield of CF_3COOH (δ = -78.5 ppm). High-resolution mass spectra were obtained at a Bruker Daltronics BioTOF HRMS spectrometer.

General Procedure for Rh(I)-Catalyzed Hydrodecarboxylation. Method A (for the synthesis of **2a-i):** Into a 20 mL scintillation vial equipped with a magnetic stir bar was placed hydroxy(1,5-cyclooctadiene)rhodium(I) dimer (5 mg, 0.011 mmol), DPPP (11 mg, 0.082 mmol), and 8.0 mL of toluene. The mixture was stirred at room temperature for 5 minutes until the materials were completely dissolved to form a homogeneous stock solution. Next, into a 4 mL screw-cap vial equipped with a magnetic stir bar was placed the benzoic acid substrate (0.225 mmol), NaOH (9.0 mg, 1.0 equiv), H_2O (150 µL, degassed), and 0.8 mL of the Rh/phosphine stock solution (containing 0.005 equiv of hydroxy(1,5-cyclooctadiene)rhodium(I) dimer and 0.010 equiv of DPPP). The vial was sealed with a silicone-lined screw-cap, transferred out of the glovebox, and stirred at 90 or 110 °C for 5~12 hours. After the reaction mixture was cooled, 500 mg of Na_2SO_4 was added into the mixture to remove H_2O. Characterization of the products was carried out by ^{19}F NMR analysis, with reaction yields determined by ^{19}F NMR integration using

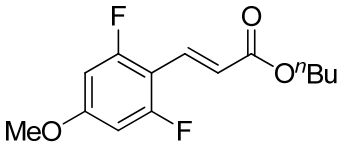
C_6F_6 as the internal standard (sealed in a capillary). The spectroscopic data (NMR, GC) matched those reported in the literatures or those acquired with commercially available authentic samples. **Method B (**for the synthesis of **2j-l, 2o**): Similar to **Method A**, but with a Rh/DPPP loading of 1-3%, 1.5 mL of toluene, 1.0 equiv NaOH or Na_2CO_3 as added base, and heating at 100 or 120 °C for 18-30 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 x 20 mL), dried over anhydrous $MgSO_4$, filtered and concentrated. Further purification was achieved by flash-column chromatography. Isolated yields are based on the average of two runs under identical conditions. **Method C (**for the synthesis of **2m, 2n):** Similar to **Method A**, with 1% Rh/DPPP, THF/H_2O (2.0 mL/350 μL), heating at 90 or 100 °C for 16~24 hours. After the reaction mixture was cooled, the mixture was quenched with H_2O (40 mL) and extracted by Et_2O (40 mL). The organic layer was collected and washed by saturated aqueous $NaHCO_3$ (2 × 20 mL) and brine (2 × 20 mL), dried over anhydrous $MgSO_4$, filtered and concentrated. Further purification was achieved by flash-column chromatography. All decarboxylative products are known compounds and previously reported. Their analytical data are identical with those reported in the literature. ^{1}H, ^{2}H and ^{13}C NMR data for deuterium-labeled **2l-*d*$_1$** is provided below as an example to support reported purity of the products.



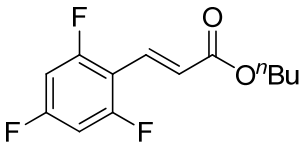
2l-*d*$_1$: Prepared from 2,6-diethoxybenzoic acid (**1l**) according to the general procedure (**Method B**) using D_2O in place of H_2O. Chromatography (5% ethyl acetate in hexane) gave **2l-*d*$_1$** as a colorless oil (32.0 mg, 85%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7. 14 (t, 1H, *J* = 8.0 Hz), 6.47 (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): δ 6.41 (s). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 160.4, 130.0, 106.9, 101.6, 63.6, 15.0.

General Procedure for Rh(I)-Catalyzed Decarboxylative Heck-Mizoroki Reaction. Into a 20 mL scintillation vial equipped with a magnetic stir bar was placed hydroxy(1,5-cyclooctadiene)rhodium(I) dimer (10 mg, 0.022 mmol), (R,R)-DIOP (28 mg, 0.054 mmol), and 15.0 mL of toluene. The mixture was stirred at room temperature for 10 minutes until the

materials were completely dissolved to form a homogeneous stock solution. Next, into a 4 mL screw-cap vial equipped with a magnetic stir bar was placed the benzoic acid substrate (0.225 mmol), olefin substrate (0.68 mmol, 3.0 equiv), NaOH (9.0 mg, 1.0 equiv), H_2O (150 µL, degassed), and 1.5 mL of the Rh/phosphine stock solution (containing 0.015 equiv of hydroxy(1,5-cyclooctadiene)rhodium(I) dimer and 0.030 equiv of (R,R)-DIOP). The vial was 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, all volatile materials were removed under reduced pressure. The residue was extracted into ethyl acetate (30 mL), washed with brine (3 x 20 mL), dried over anhydrous $MgSO_4$, filtered and concentrated. Further purification was achieved by flash-column chromatography. Isolated yields are based on the average of two runs under identical conditions.

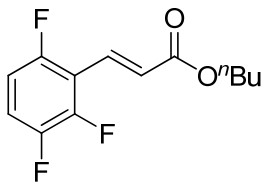


4b: Prepared from 2,6-difluoro-4-methoxybenzoic acid (**1a**) and *n*-butyl acrylate (**3a**) according to the general procedure. Chromatography (10% ethyl acetate in hexane) gave **4b** as a light-yellow oil (47.8 mg, 79%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.68 (d, 1H, *J* = 16.4 Hz), 6.58 (d, 1H, *J* = 16.4 Hz), 6.46 (d, 2H, *J* = 10.8 Hz), 4.18 (t, 2H, *J* = 6.4 Hz), 3.79 (s, 3H), 1.65 (quintet, 2H, *J* = 7.2 Hz), 1.42 (sextet, 2H, *J* = 7.2 Hz), 0.94 (t, 3H, *J* = 7.6 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 167.6, 162.8 (dd, J_1 = 253.6 Hz, J_2 = 10.4 Hz), 162.1, 131.0, 121.6 (t, *J* = 8.1 Hz), 105.6, 98.6 (d, *J* = 26.9 Hz), 64.6, 56.1, 31.0, 19.4, 13.9. ^{19}F-NMR (282.4 MHz, $CDCl_3$): δ -109.3 (d, *J* = 9.0 Hz). HRMS: calcd for $C_{14}H_{168}O_3F_2Na^+$ 293.0960, found 293.0958.

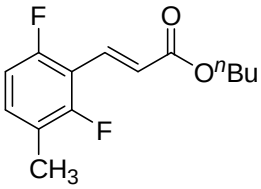


4c: Prepared from 2,4,6-trifluorobenzoic acid (**1d**) and *n*-butyl acrylate (**3a**) according to the general procedure. Chromatography (5% ethyl acetate in hexane) gave **4c** as a light-yellow oil (40.6 mg, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.66 (d, 1H, *J* = 16.8 Hz), 6.70 (t, 2H, *J* = 8.4

Hz), 6.65 (d, 1H, *J* = 16.4 Hz), 4.20 (t, 2H, *J* = 6.8 Hz), 1.67 (quintet, 2H, *J* = 6.8 Hz), 1.42 (sextet, 2H, *J* = 7.6 Hz), 0.94 (t, 3H, *J* = 7.6 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 167.1, 163.7 (d, *J* = 252.3 Hz), 162.2 (d, *J* = 269.7 Hz), 129.9, 129.1, 128.2, 101.1 (t, *J* = 24.2 Hz), 64.9, 30.9, 19.4, 13.9. ^{19}F-NMR (282.4 MHz, $CDCl_3$): δ -104.8 (s, 1F), -107.3 (s, 2F). HRMS: calcd for $C_{13}H_{13}O_2F_3Na^+$ 281.0760, found 281.0758.

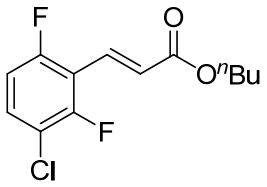


4d: Prepared from 2,3,6-trifluorobenzoic acid (**1c**) and *n*-butyl acrylate (**3a**) according to the general procedure. Chromatography (5% ethyl acetate in hexane) gave **4d** as a light-yellow oil (42.2 mg, 81%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.70 (d, 1H, *J* = 16.4 Hz), 7.12 (m, 1H), 6.87 (triplet of quartet, 1H, J_1 = 9.6 Hz, J_2 = 2.4 Hz), 6.74 (d, 1H, *J* = 16.4 Hz), 4.21 (t, 2H, *J* = 6.8 Hz), 1.70 (quintet, 2H, *J* = 6.8 Hz), 1.40 (sextet, 2H, *J* = 7.6 Hz), 0.95 (t, 3H, *J* = 7.2 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 166.8, 157.0 (d, *J* = 250.9 Hz), 149.4 (d, *J* = 263.0 Hz), 147.5 (dd, J_1 = 244.1 Hz, J_2 = 9.4 Hz), 130.1, 125.7 (t, *J* = 8.1 Hz), 117.9 (q, *J* = 10.8 Hz), 114.3 (t, *J* = 12.2 Hz), 111.3 (dt, J_1 = 24.3 Hz, J_2 = 4.1 Hz), 65.0, 30.9, 19.4, 13.9. ^{19}F-NMR (282.4 MHz, $CDCl_3$): δ -116.0 (s, 1F), -113.7 (dd, 1F, J_1 = 21.5 Hz, J_2 = 9.0 Hz), -142.3 (s, 1F). HRMS: calcd for $C_{13}H_{13}O_2F_3Na^+$ 281.0760, found 281.0763.

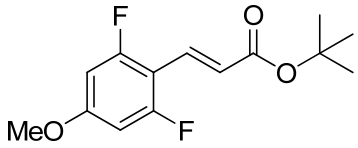


4e: Prepared from 2,6-difluoro-3-methylbenzoic acid and *n*-butyl acrylate according to the general procedure. Chromatography (5% ethyl acetate in hexane) gave **4e** as a light-yellow oil (39.1 mg, 68%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7. 74 (d, 1H, *J* = 16.4 Hz), 7.10 (quartet, 1H, *J* = 6.4 Hz), 6.78 (dt, 1H, J_1 = 6.0 Hz, J_2 = 0.8 Hz), 6.70 (d, 1H, *J* = 16.8 Hz), 4.19 (t, 1H, *J* = 6.8 Hz, 2H), 2.21 (t, 3H, *J* = 1.2 Hz), 1.65 (quintet, 2H, *J* = 6.8 Hz), 1.40 (sextet, 2H, *J* = 6.4 Hz), 0.95 (t, 3H, *J* = 7.6 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 167.3, 161.4 (dd, J_1 = 27.0 Hz, J_2 =

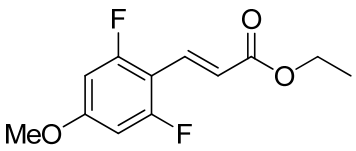
6.8 Hz), 158.6 (dd, J_1 = 28.3 Hz, J_2 = 6.8 Hz), 132.5 (dd, J_1 = 9.4 Hz, J_2 = 6.7 Hz), 131.3, 124.2 (t, J = 8.8 Hz), 121.1 (dd, J_1 = 17.6 Hz, J_2 = 4.0 Hz), 112.1 (t, J = 16.2 Hz), 111.2 (dd, J_1 = 23.0 Hz, J_2 = 4.1 Hz), 64.8, 30.9, 19.4, 14.3, 13.9. ^{19}F-NMR (282.4 MHz, $CDCl_3$): δ -113.85 (s, 1F), -114.81 (s, 1F). HRMS: calcd for $C_{14}H_{16}O_2F_2Na^+$ 277.1011, found 277.1024.



4f: Prepared from 2,6-difluoro-3-chlorobenzoic acid (**1b**) and *n*-butyl acryalte (**3a**) according to the general procedure. Chromatography (5% ethyl acetate in hexane) gave **4f** as a light-yellow oil (49.4 mg, 73%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.70 (d, 1H, J = 16.4 Hz), 7.35 (m, 1H), 6.90 (dt, 1H, J_1 = 9.6 Hz, J_2 = 2.0 Hz), 6.73 (d, 1H, J = 16.4 Hz), 4.21 (t, 2H, J = 6.8 Hz), 1.69 (quintet, 2H, J = 6.8 Hz), 1.40 (sextet, 2H, J = 7.6 Hz), 0.95 (t, 3H, J = 7.6 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 166.8, 159.83 (dd, J_1 = 310.2 Hz, J_2 = 6.0 Hz), 155.3 (d, J = 310.2 Hz), 131.2 (d, J = 9.5 Hz), 130.1, 125.8 (t, J = 8.1 Hz), 117.6 (d, J = 18.8 Hz), 114.1 (t, J = 14.9 Hz), 112.5 (dd, J_1 = 24.3 Hz, J_2 = 4.1 Hz), 65.0, 30.9, 19.4, 13.9. ^{19}F-NMR (282.4 MHz, $CDCl_3$): δ -110.9 (s, 1F), -111.7 (s, 1F). HRMS: calcd for $C_{13}H_{13}O_2F_2ClNa^+$ 297.0464, found 297.0478.



4h: Prepared from 2,6-difluoro-4-methoxybenzoic acid (**1a**) and *tert*-butyl acrylate according to the general procedure. Chromatography (10% ethyl acetate in hexane) gave **4h** as a light-yellow oil (43.5 mg, 72%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.58 (d, 1H, J = 16.4 Hz), 6.50 (d, 1H, J = 16.4 Hz), 6.44 (d, 2H, J = 10.4 Hz), 3.78 (s, 3H), 1.50 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 166.8, 162.8 (dd, J_1 = 250.9 Hz, J_2 = 9.5 Hz), 161.9 (t, J = 14.8 Hz), 130.0, 121.5 (t, J = 8.1 Hz), 105.7 (t, J = 14.8 Hz), 98.5 (dd, J_1 = 27.0 Hz, J_2 = 6.8 Hz), 80.7, 56.1, 28.4. ^{19}F-NMR (282.4 MHz, $CDCl_3$): δ -109.5 (d, J = 12.1 Hz). HRMS: calcd for $C_{14}H_{16}O_3F_2Na^+$ 293.0960, found 293.0958.



4i: Prepared from 2,6-difluoro-4-methoxybenzoic acid (**1a**) and ethyl acrylate according to the general procedure. Chromatography (10% ethyl acetate in hexane) gave **4i** as a light-yellow oil (40.7 mg, 75%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.67 (d, 1H, J = 16.4 Hz), 6.57 (d, 1H, J = 16.4 Hz), 6.45 (d, 2H, J = 10.4 Hz), 4.23 (quartet, 2H, J = 7.2 Hz), 3.78 (s, 3H), 1.30 (t, 3H, J = 6.8 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 167.5, 162.9 (dd, J_1 = 252.2 Hz, J_2 = 10.8 Hz), 162.2 (t, J = 13.5 Hz), 131.0, 121.6 (t, J = 9.4 Hz), 105.6 (t, J = 14.8 Hz), 98.6 (dd, J_1 = 22.9 Hz, J_2 = 8.1 Hz), 60.7, 56.1, 14.5. ^{19}F-NMR (282.4 MHz, $CDCl_3$): δ -109.3 (d, J = 12.1 Hz). HRMS: calcd for $C_{12}H_{12}O_3F_2Na^+$ 265.0647, found 265.0649.

8

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
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60 repetitions
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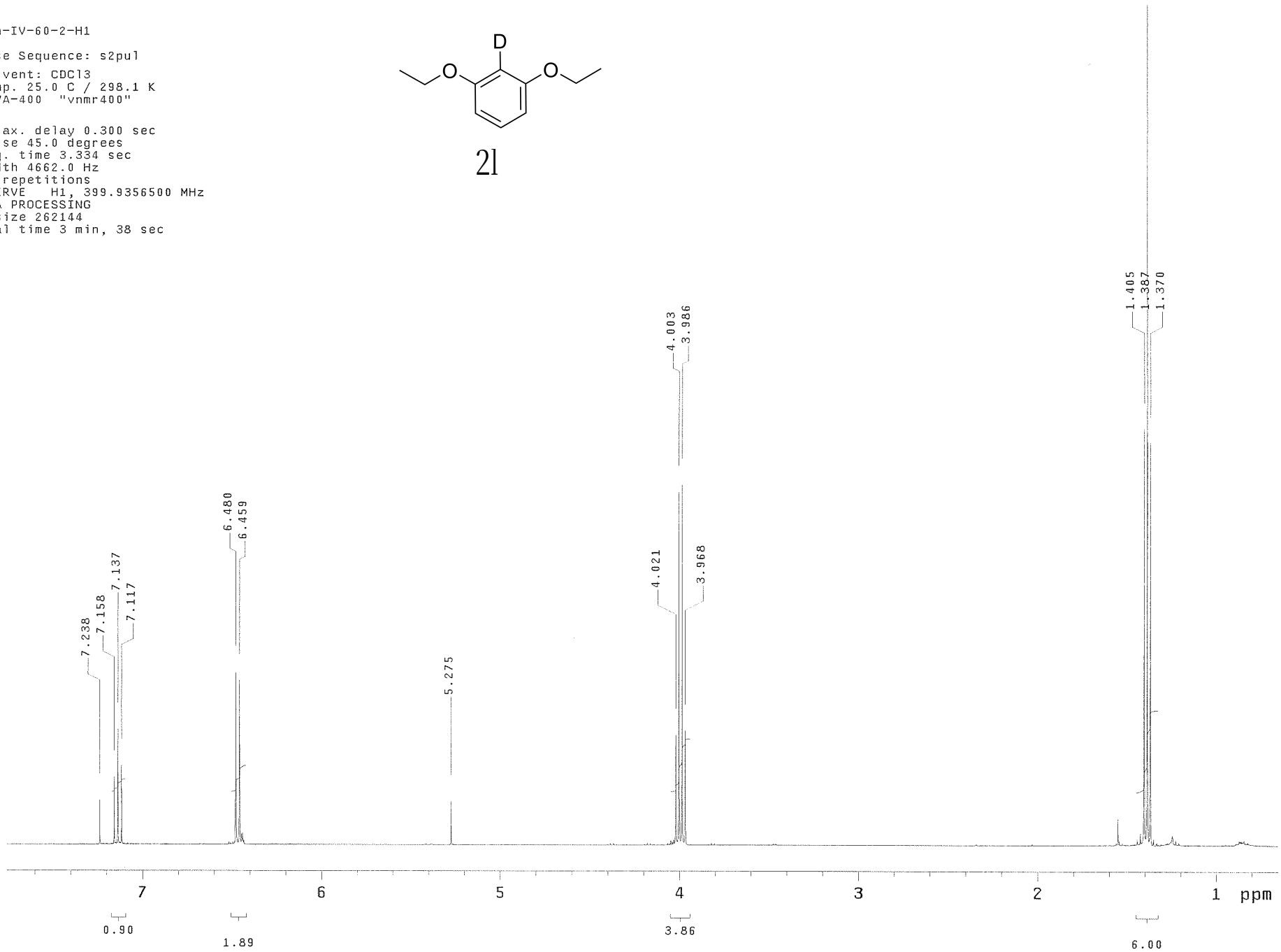
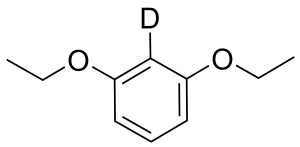
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec

21





2l

zsun-IV-60-2-H2

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

Pulse 16.7 degrees
Acq. time 1.334 sec
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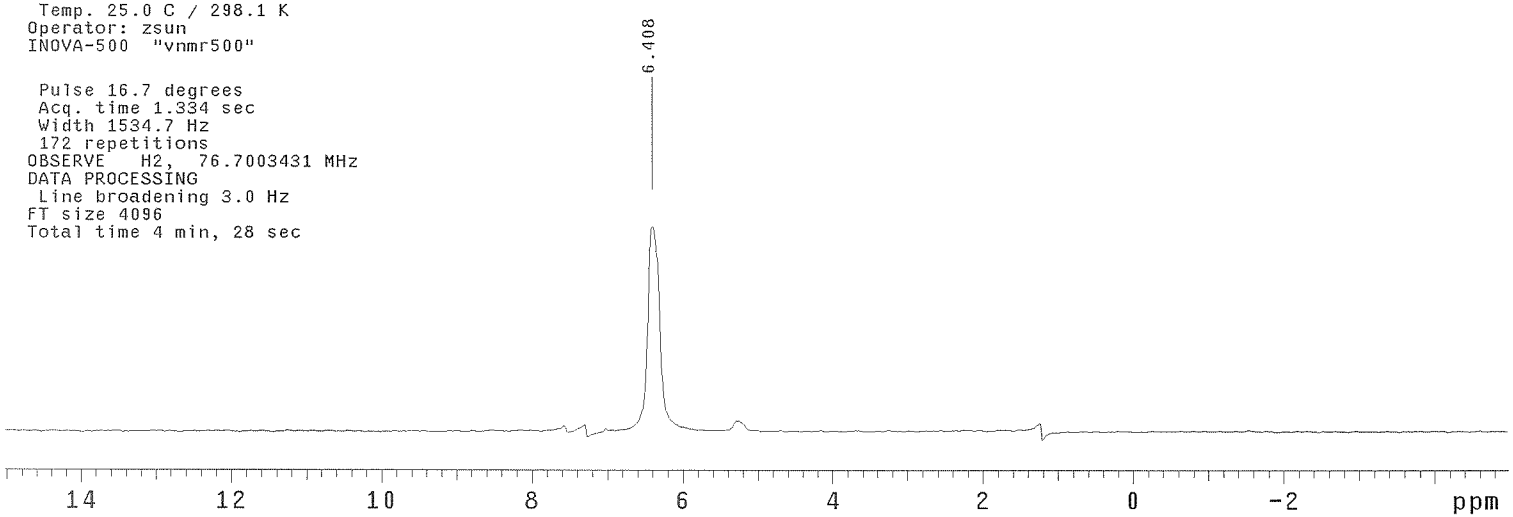
DATA PROCESSING
Line broadening 3.0 Hz
FT size 4096
Total time 4 min, 28 sec



10

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
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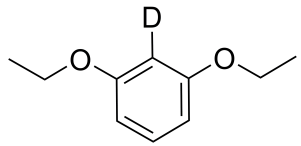
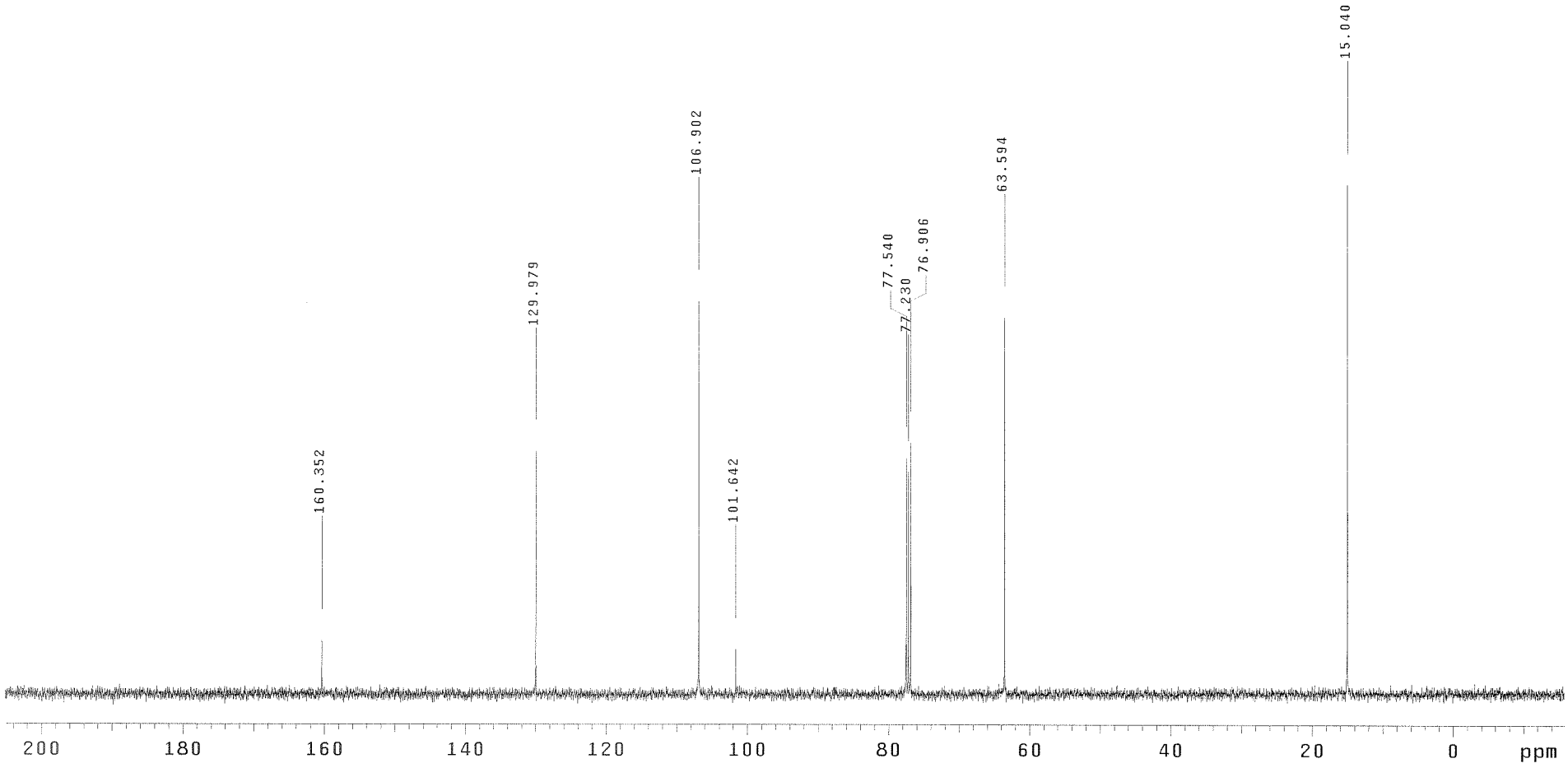
FT size 32768
Total time 1 hr, 8 min, 37 sec



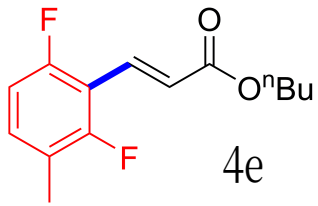
21



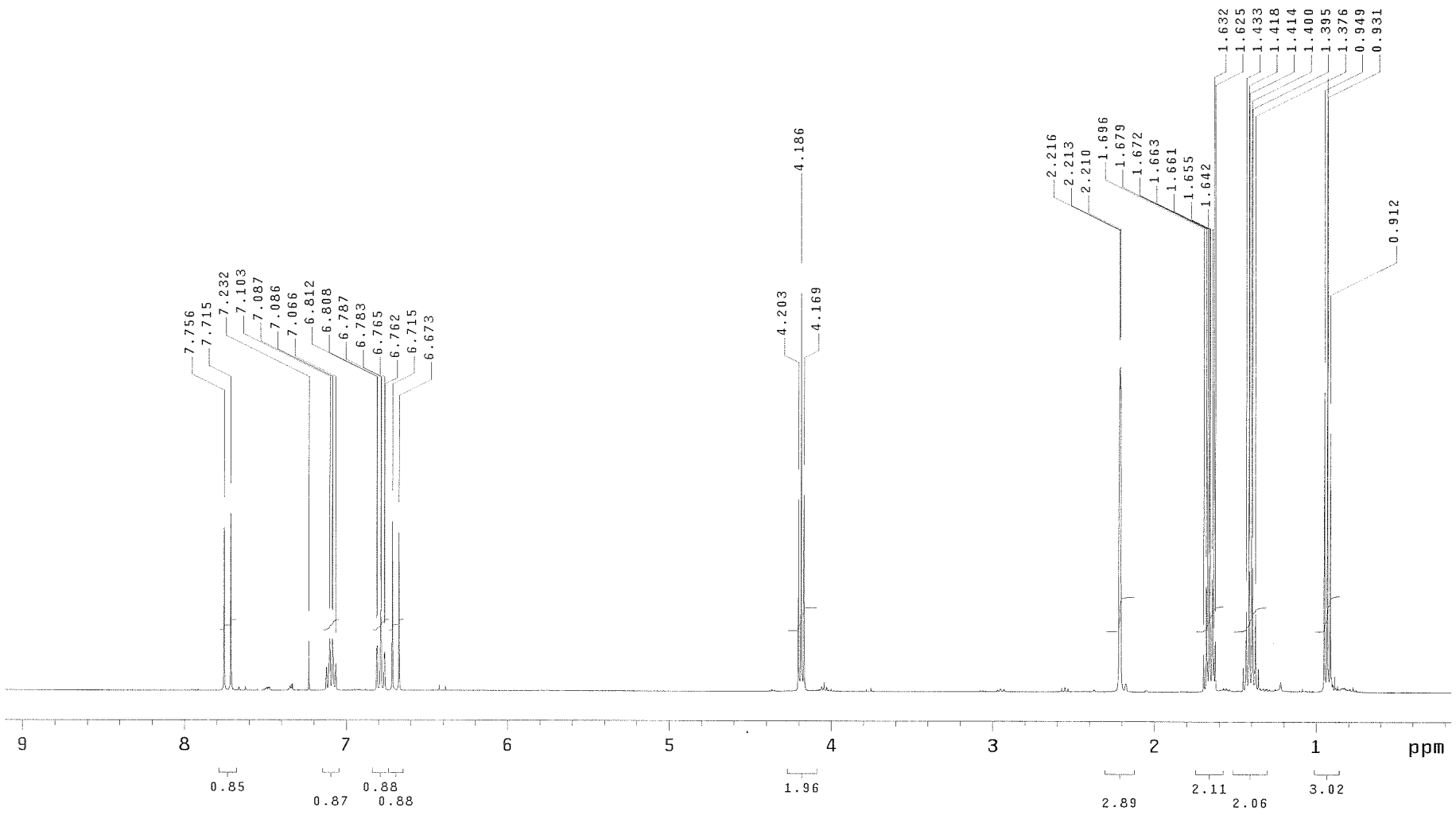
zsun-IV-74-2-H1

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



4e



zsun-IV-74-2-C13

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Solvent: CDCl3
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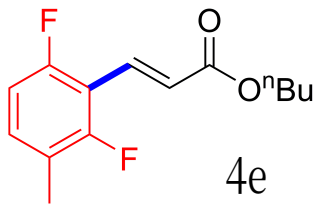
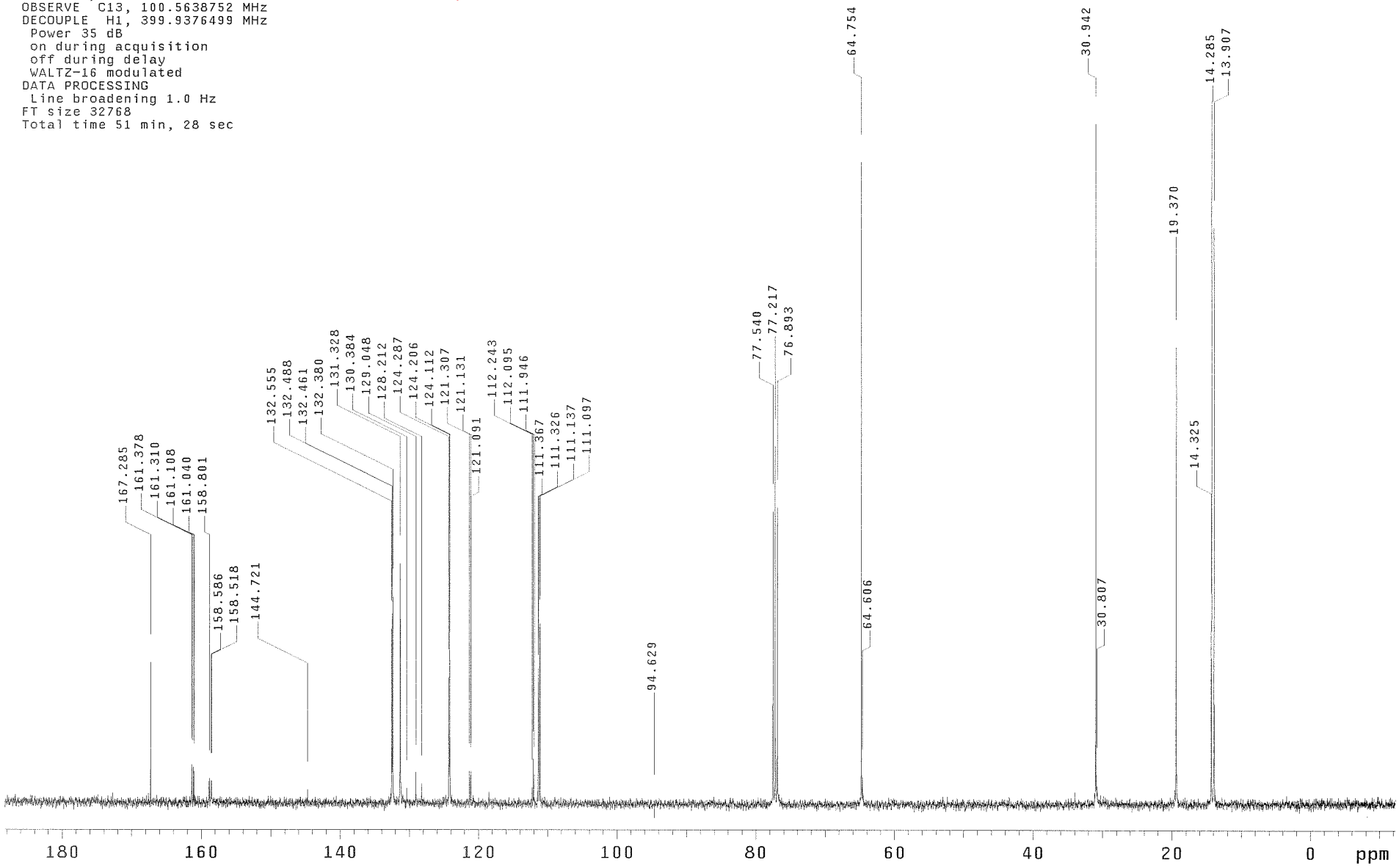
Width 22222.2 Hz
1044 repetitions
OBSERVE C13, 100.5638752 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 51 min, 28 sec



4e



13

zsun-IV-74-2-C13

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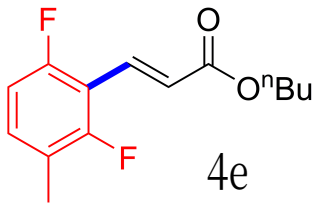
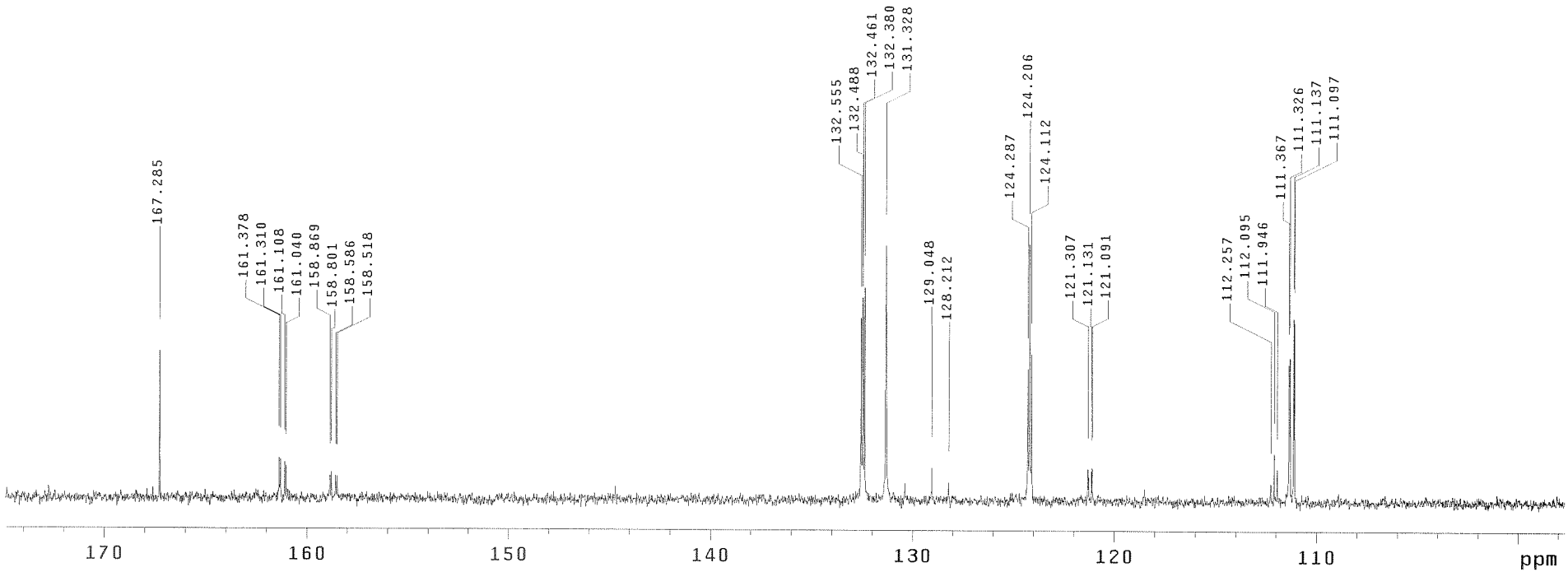
Pulse 45.0 degrees
Acq. time 0.720 sec
Width 22222.2 Hz
952 repetitions
OBSERVE C13, 100.5638752 MHz
DECOUPLE H1, 399.9376499 MHz
Power 35 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 51 min, 28 sec



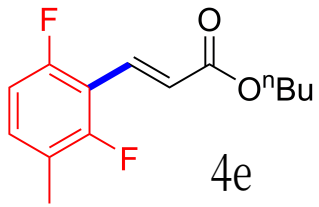
4e



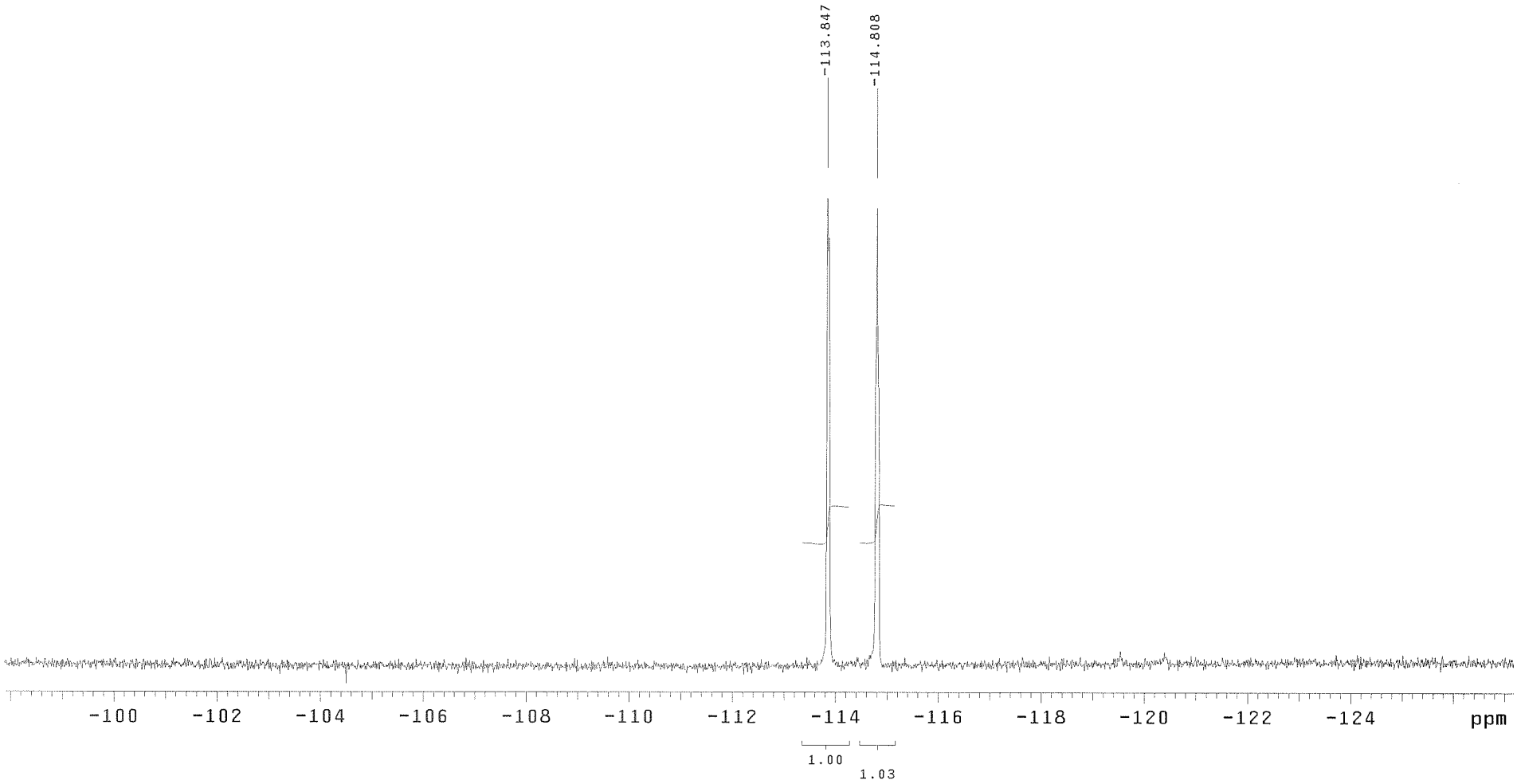
zsun-IV-74-2-F19

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Operator: zsun
Mercury-300BB "vnmr300"

Relax. delay 4.000 sec
Pulse 30.6 degrees
Acq. time 0.300 sec
Width 50000.0 Hz
40 repetitions
OBSERVE F19, 282.4118209 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 1 hr, 4 min, 26 sec



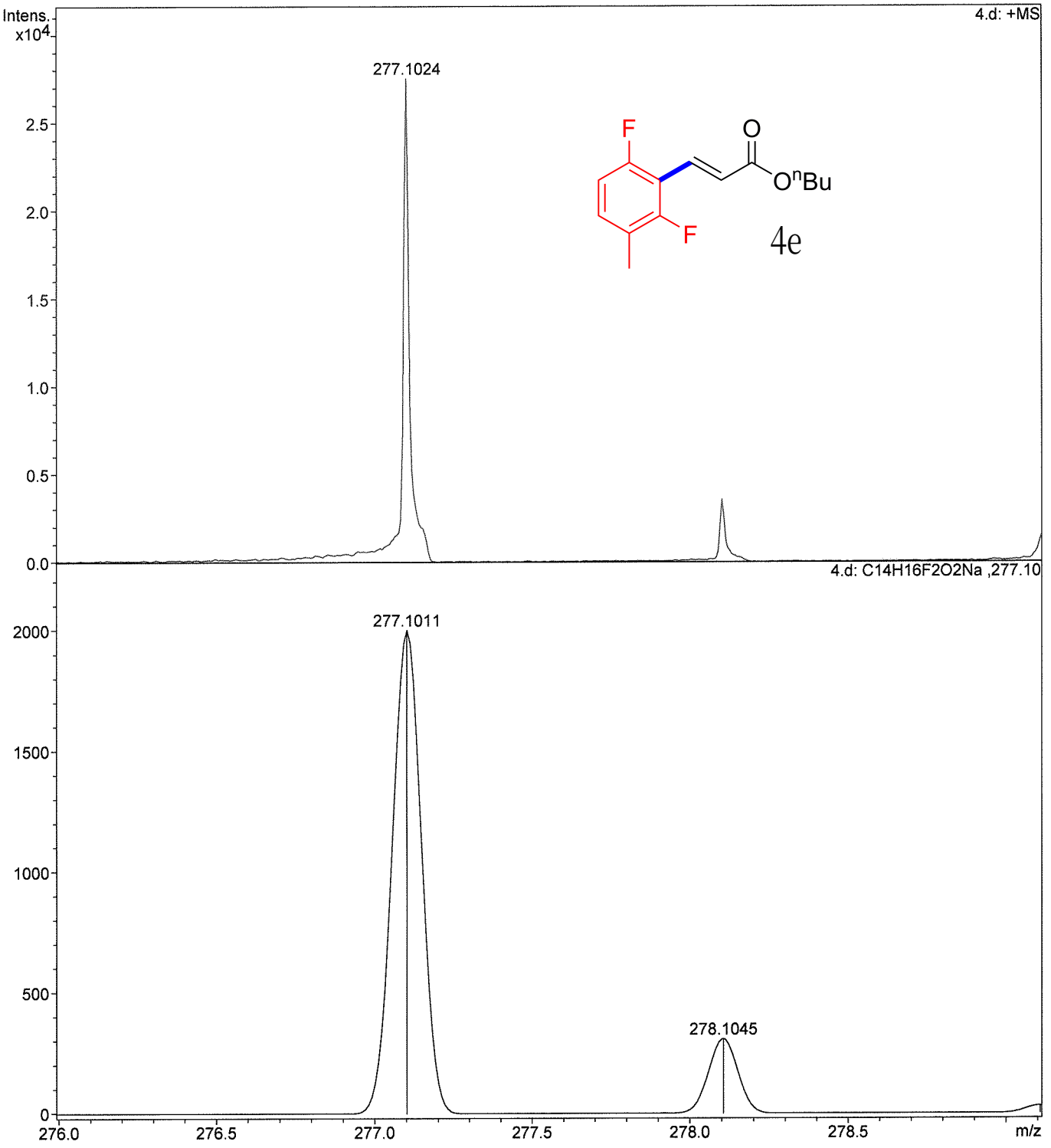
4e



Generic Display Report

Analysis Info

Acquisition Date	11/20/2009 4:05:42 PM		
Analysis Name	D:\Bruker\data\zhosun\IV-85-1\4.d		
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	IV-85-1	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		

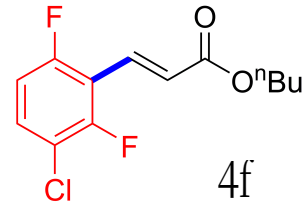


zsun-IV-43-1-H1

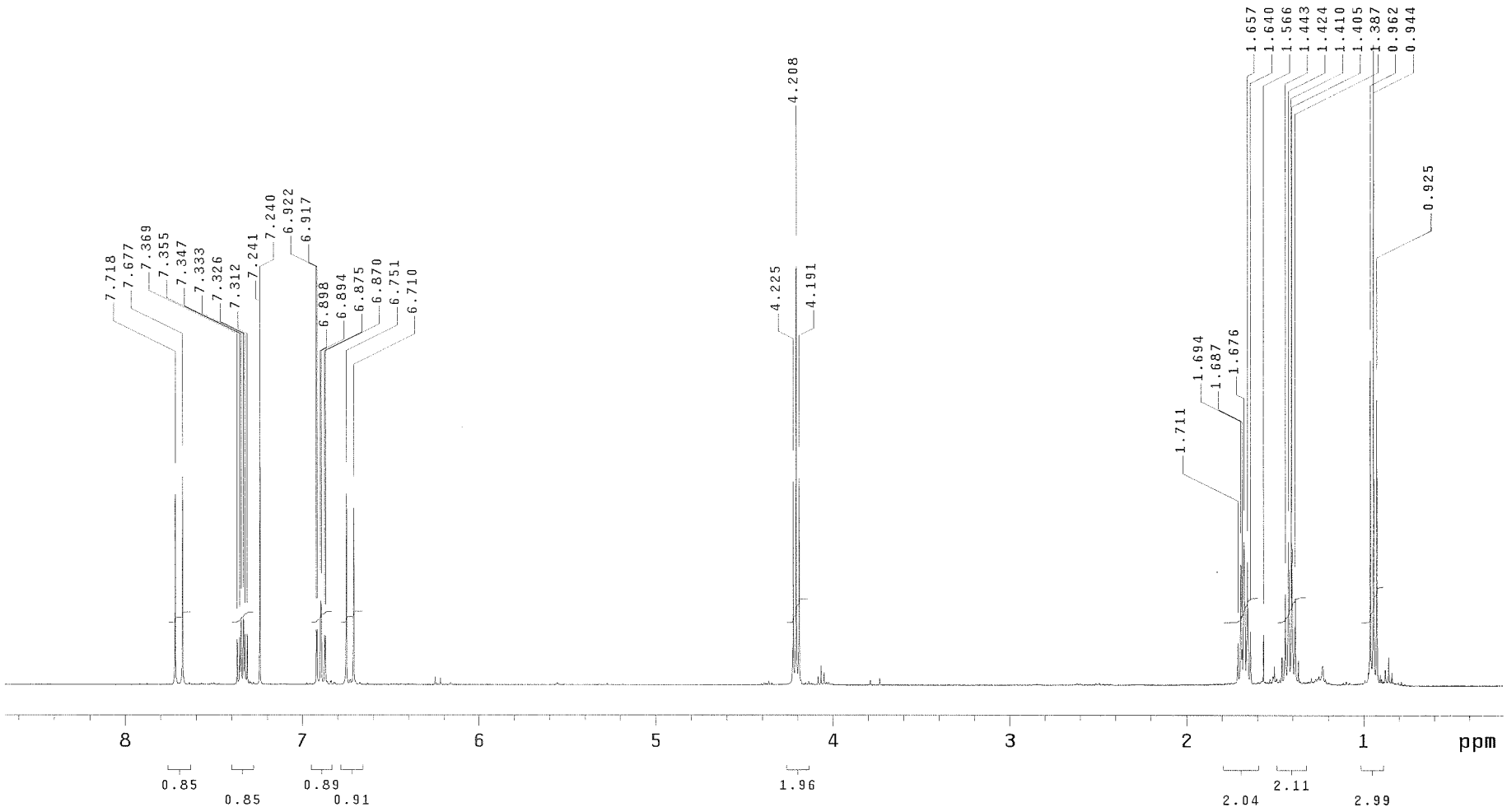
Pulse Sequence: s2pul

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
36 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



4f



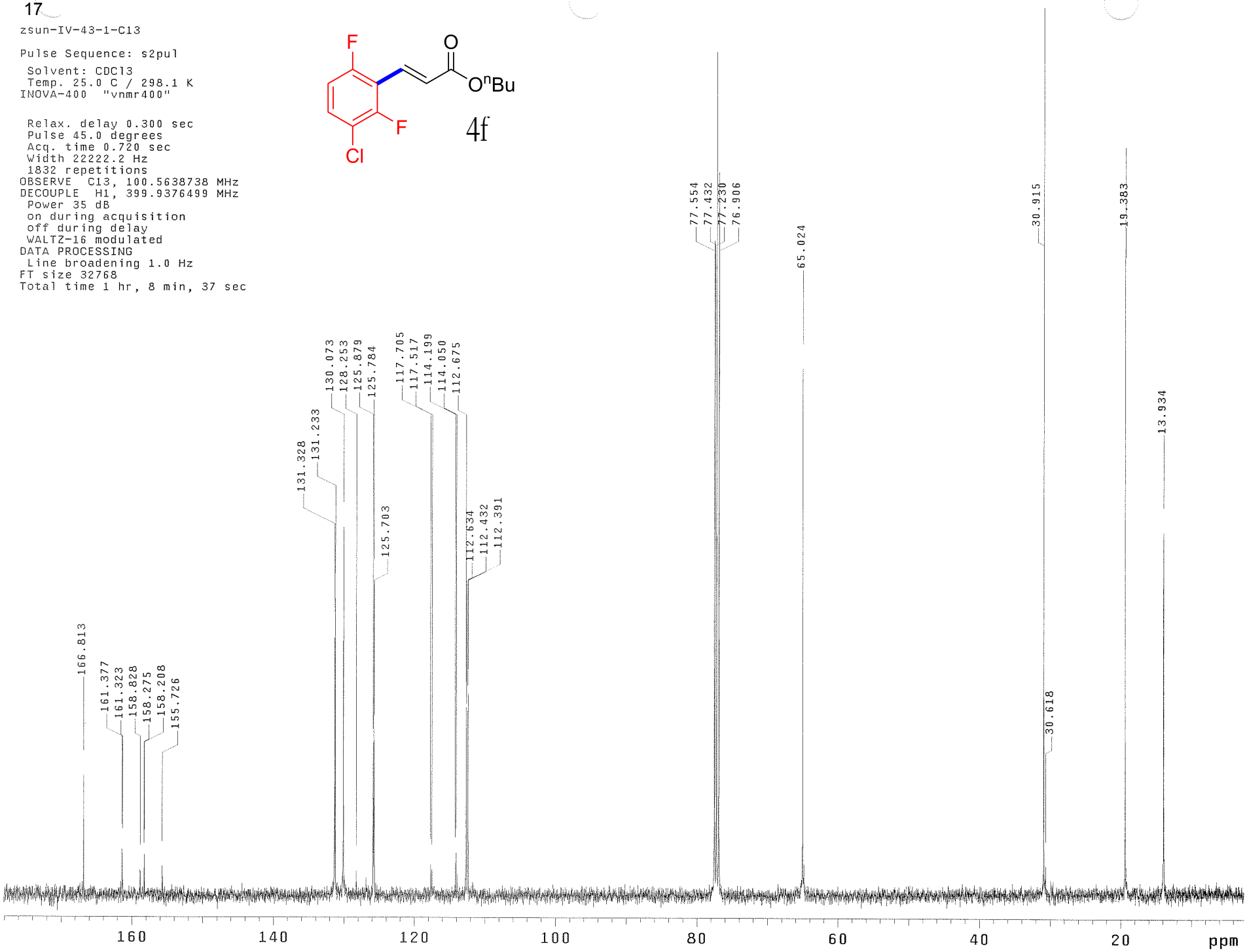
zsun-IV-43-1-C13

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
1832 repetitions
OBSERVE C13, 100.5638738 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

4f

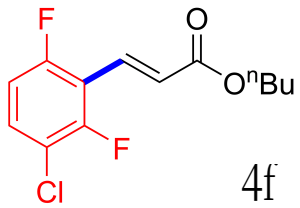


zsun-IV-43-1-C13

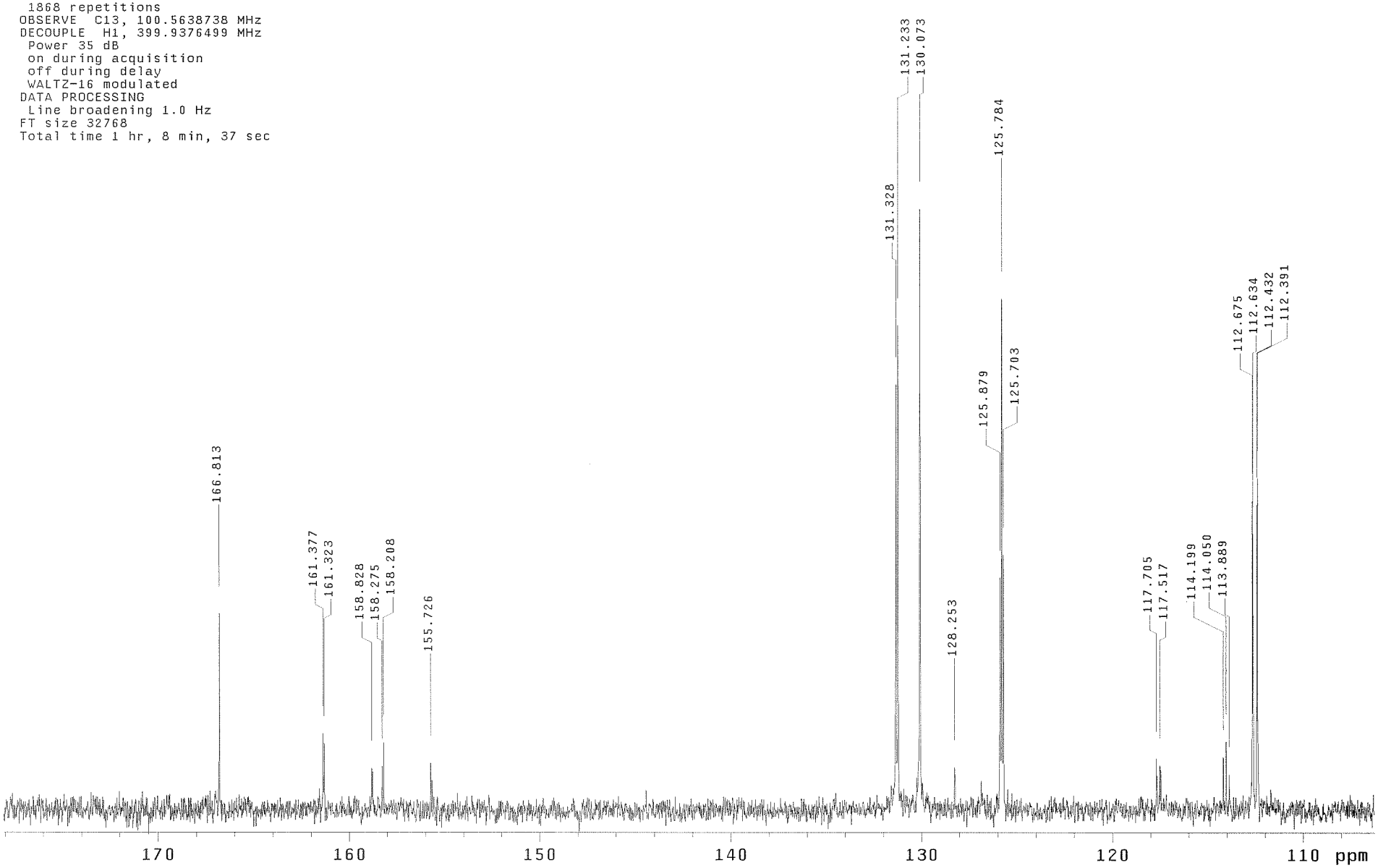
Pulse Sequence: s2pul

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
1868 repetitions
OBSERVE C13, 100.5638738 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



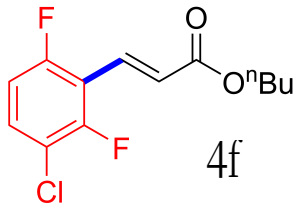
4f



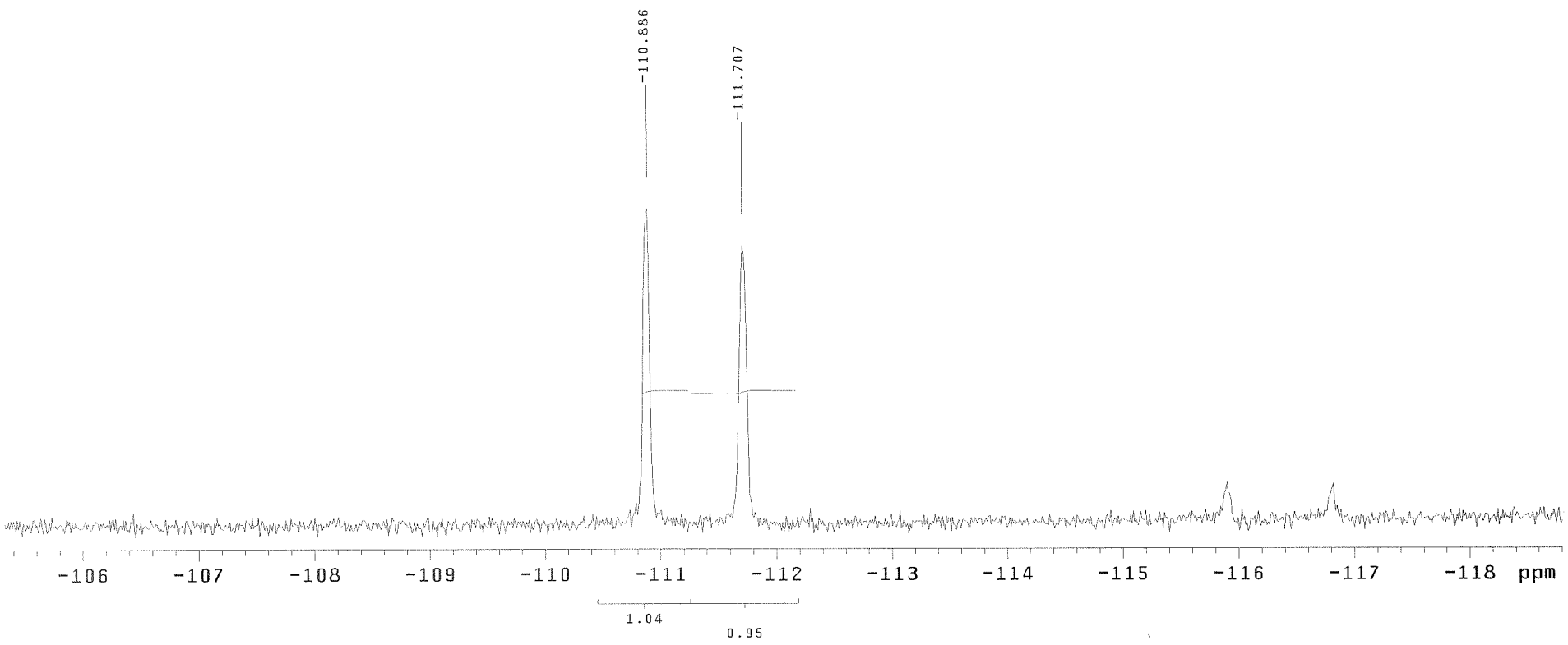
zsun-IV-22-1-F19

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Operator: zsun
Mercury-300BB "vnmr300"

 Relax. delay 4.000 sec
 Pulse 30.6 degrees
 Acq. time 0.300 sec
 Width 50000.0 Hz
 152 repetitions
OBSERVE F19, 282.4095227 MHz
DATA PROCESSING
 Line broadening 0.3 Hz
FT size 32768
Total time 32 min, 13 sec



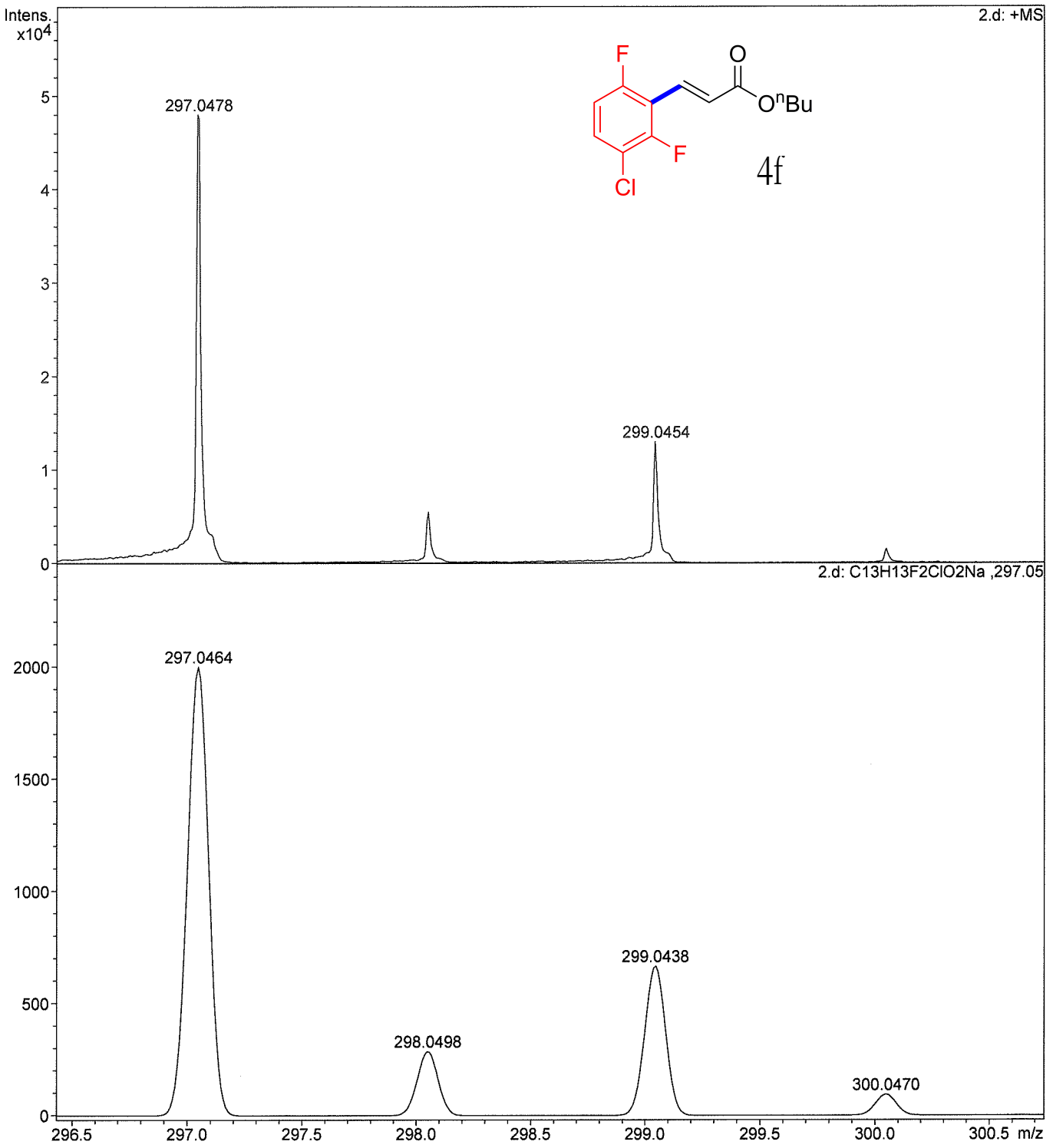
4f



Generic Display Report

Analysis Info

Acquisition Date	10/23/2009 3:48:04 PM		
Analysis Name	D:\Bruker\data\zhosun\IV_43-1\2.d		
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	IV_43-1	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		



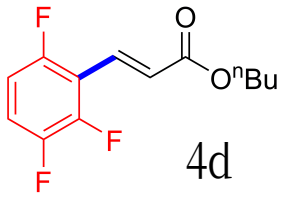
21

zsun-IV-43-2-H1

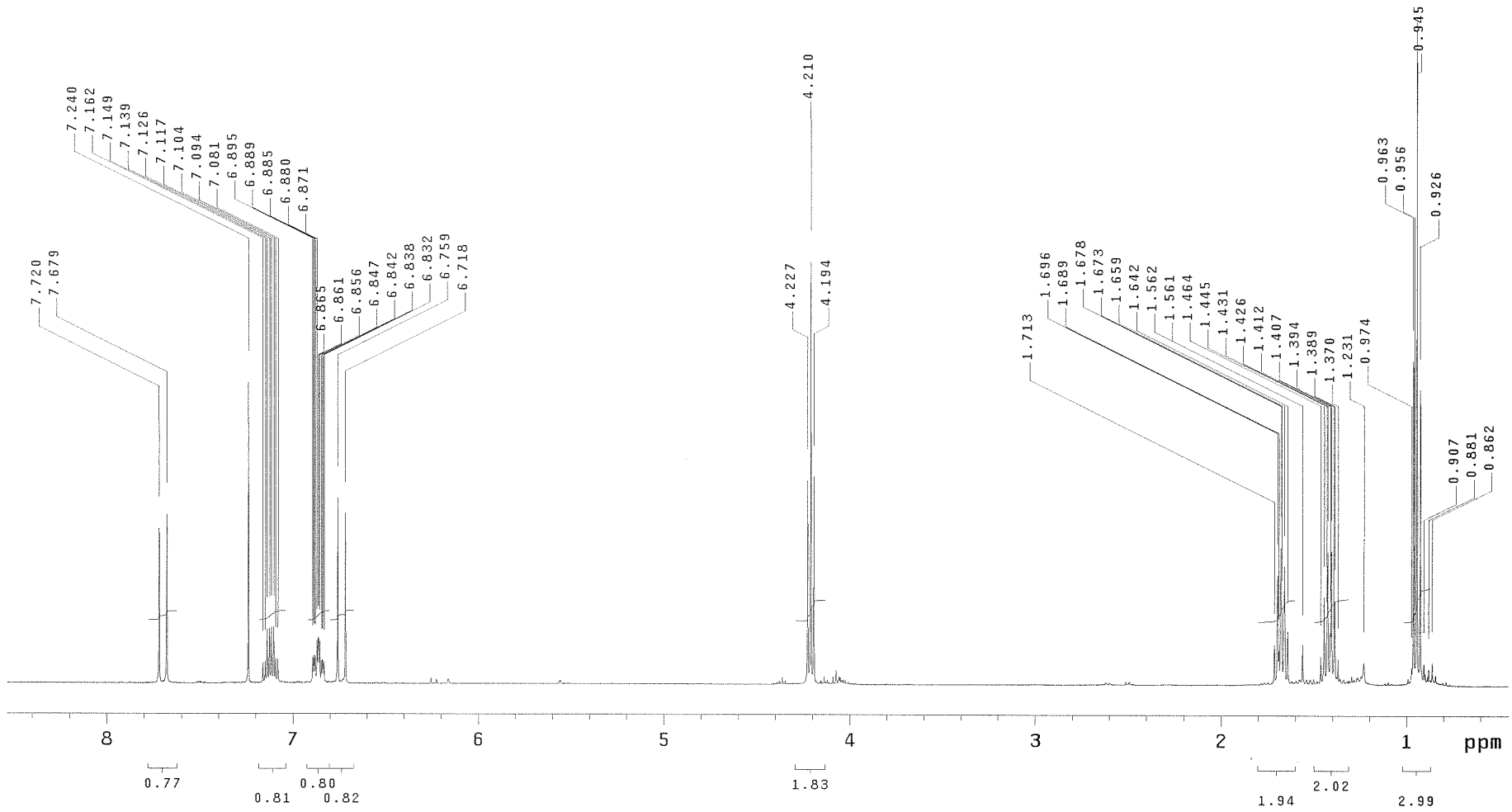
Pulse Sequence: s2pul

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



4d

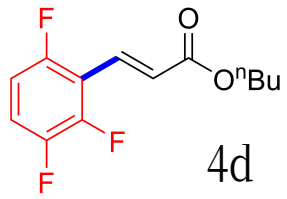


zsun-IV-43-2-H1

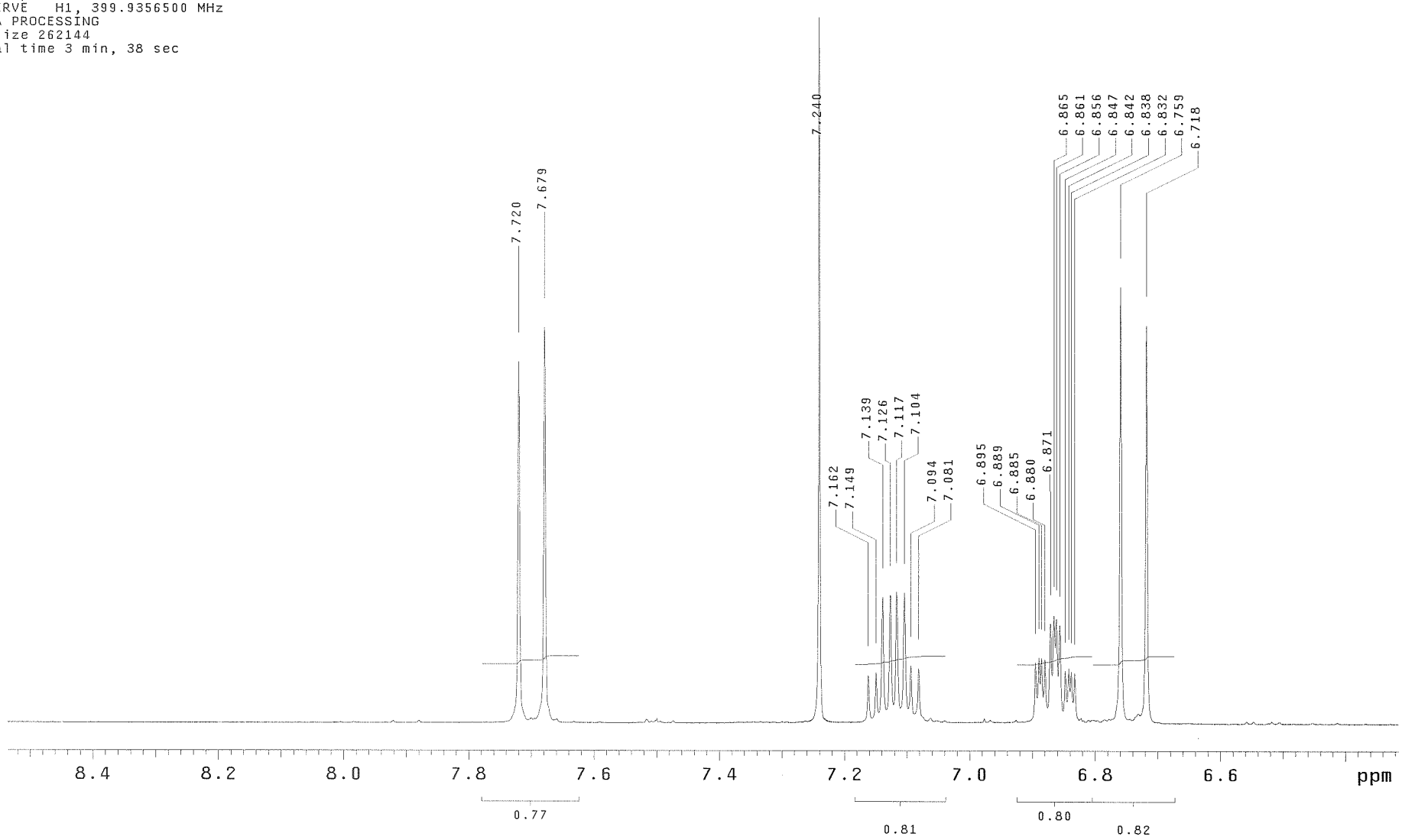
Pulse Sequence: s2pul

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



4d



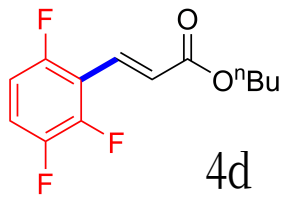
23

zsun-IV-43-2-C13

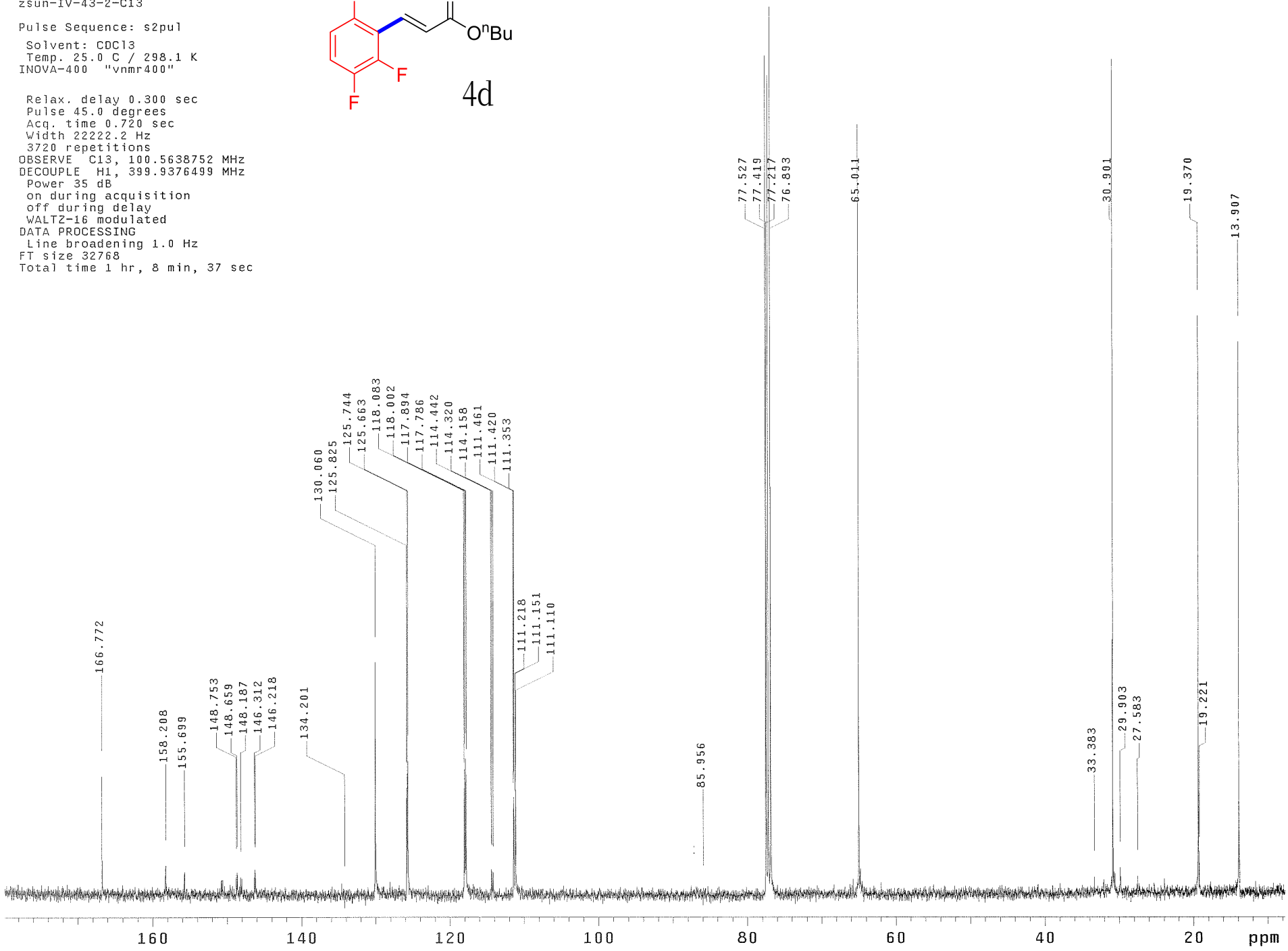
Pulse Sequence: s2pul

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
3720 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



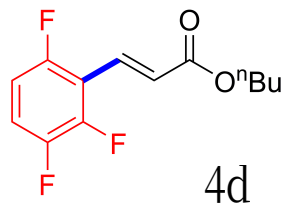
4d



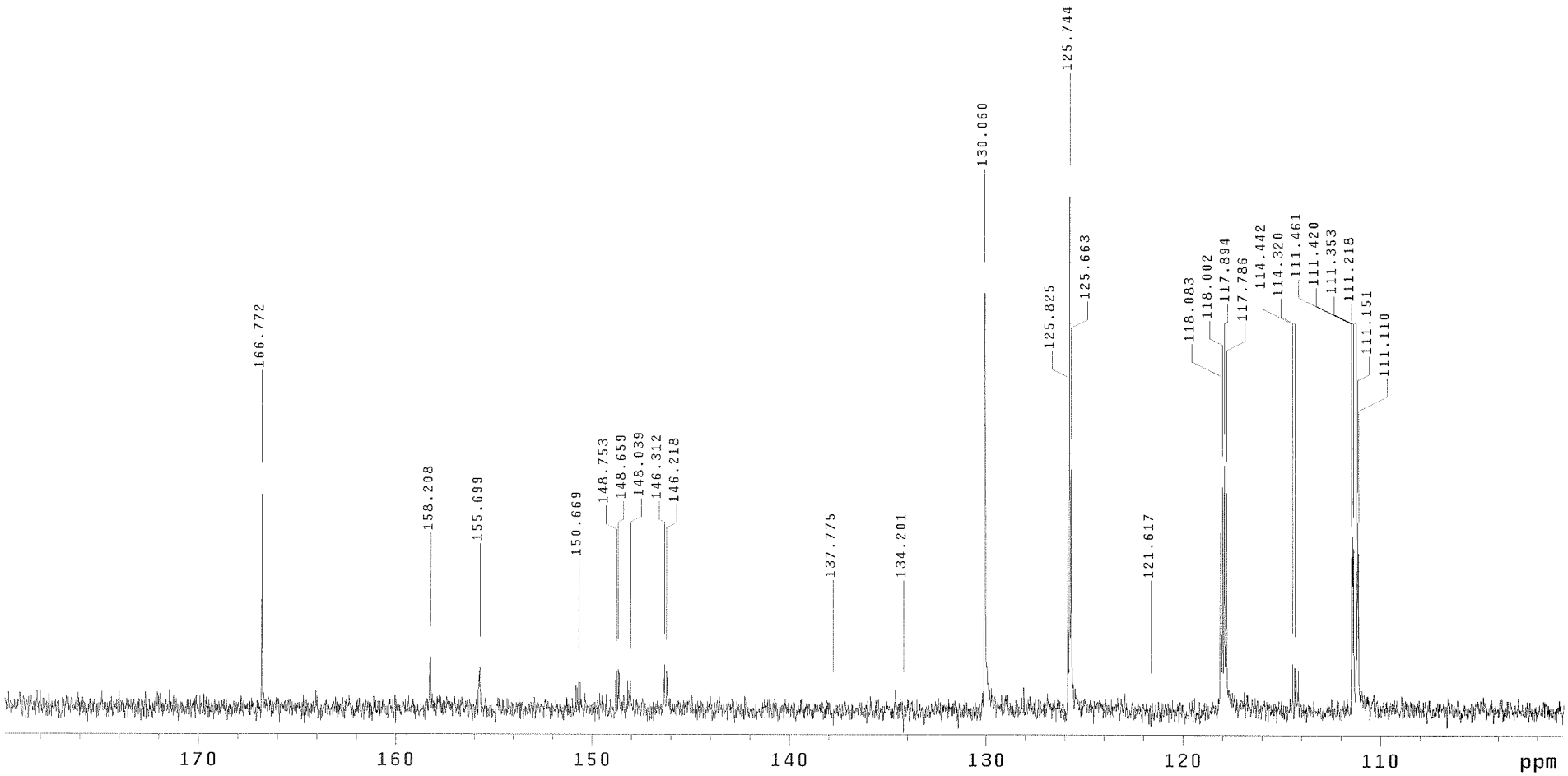
zsun-IV-43-2-C13

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
3780 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



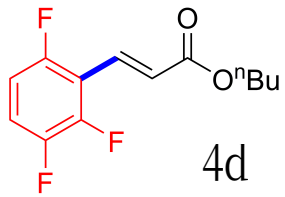
4d



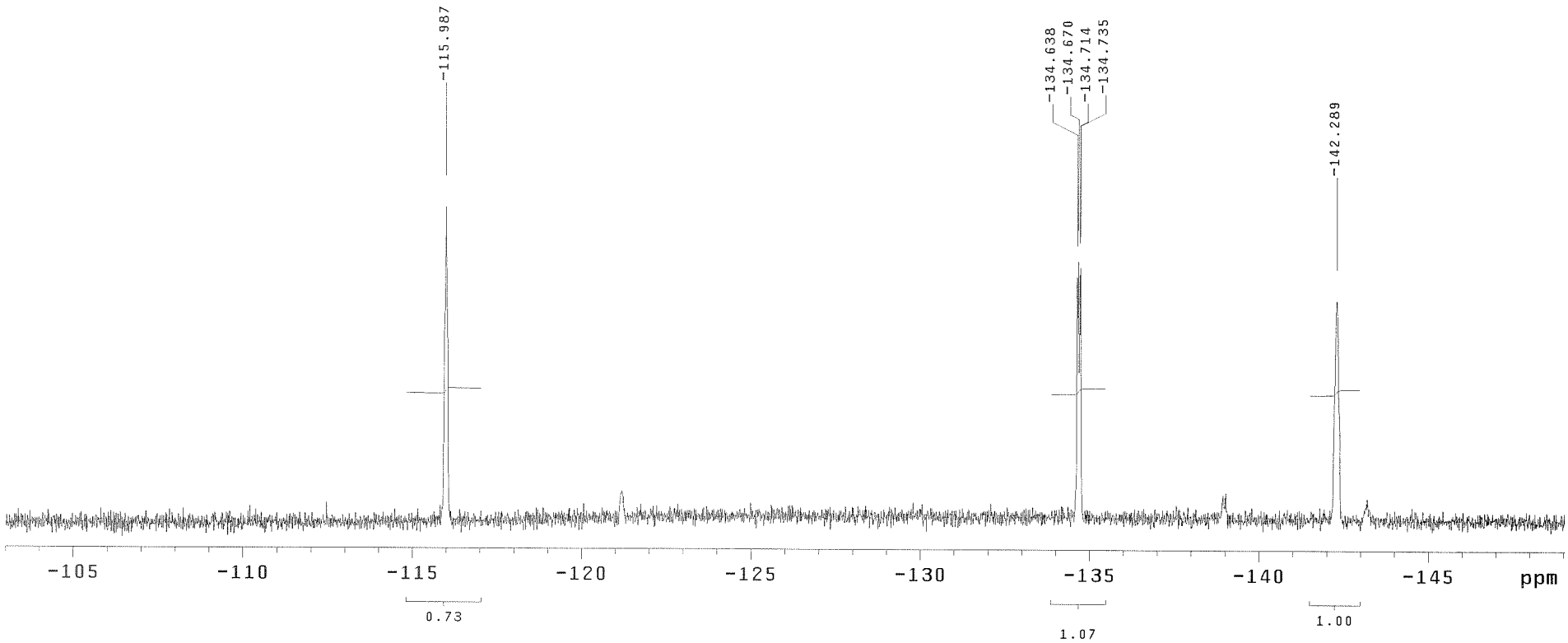
zsun-IV-22-2-F19

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Operator: zsun
Mercury-300BB "vnmr300"

 Relax. delay 4.000 sec
 Pulse 30.6 degrees
 Acq. time 0.300 sec
 Width 50000.0 Hz
 96 repetitions
OBSERVE F19, 282.4095227 MHz
DATA PROCESSING
 Line broadening 0.3 Hz
FT size 32768
Total time 32 min, 13 sec



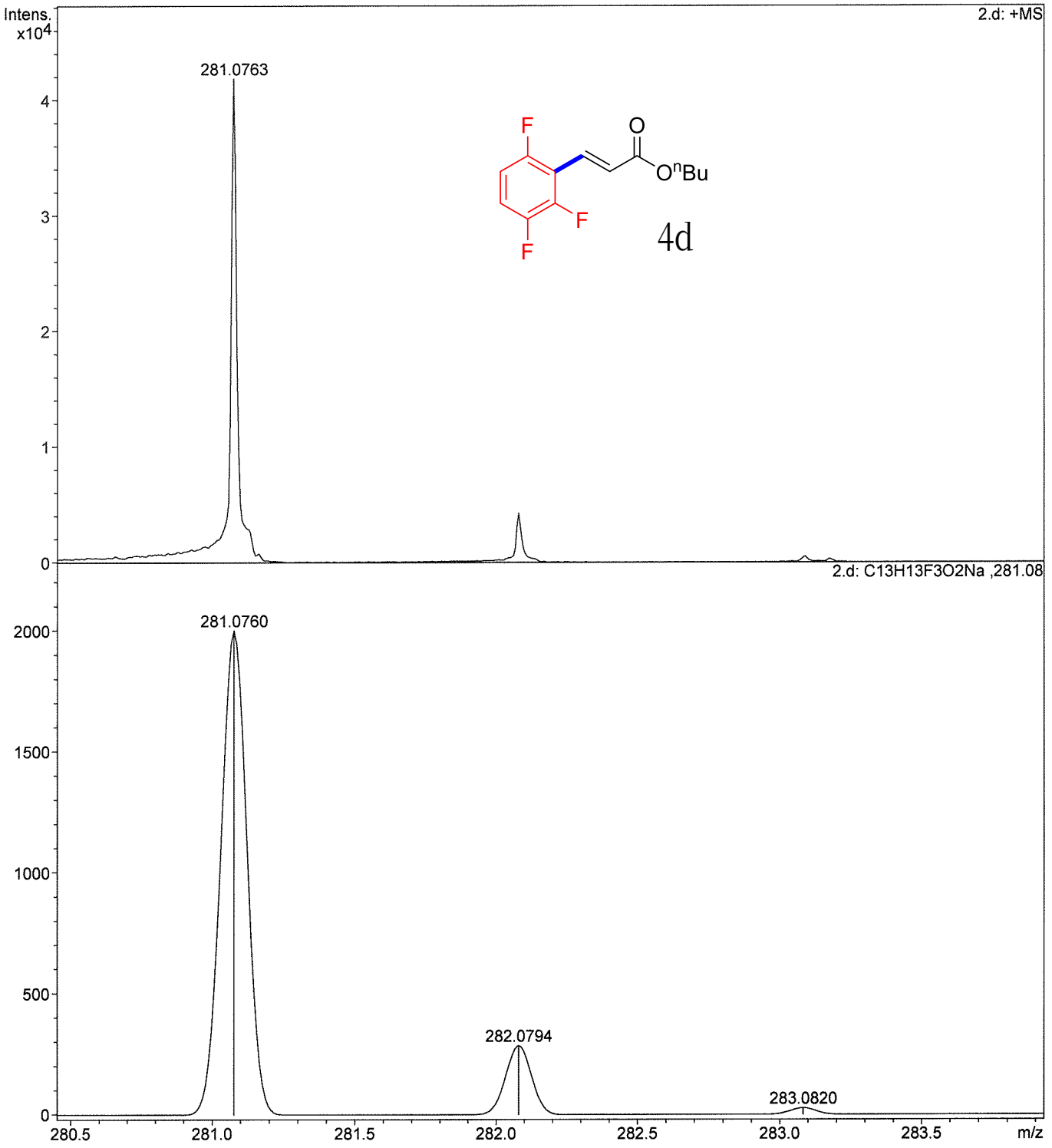
4d



Generic Display Report

Analysis Info

Acquisition Date	10/23/2009 3:55:13 PM		
Analysis Name	D:\Bruker\data\zhosun\IV_43-2\2.d		
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	IV_43-2	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		

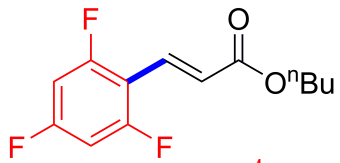


zsun-IV-22-3-H1

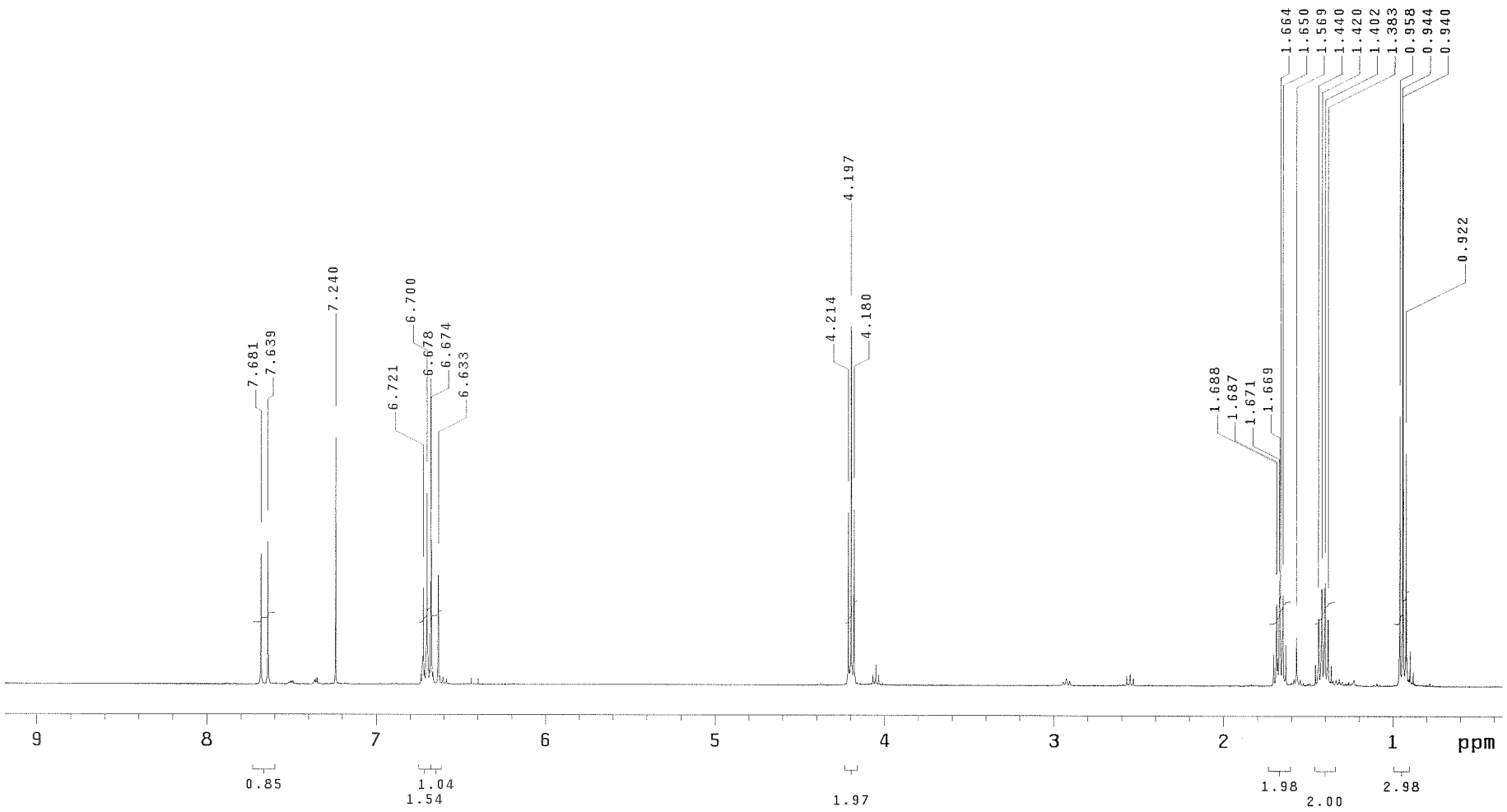
Pulse Sequence: s2pul

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
44 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



4c

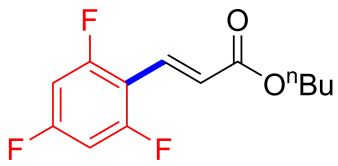


zsun-IV-22-3-C13

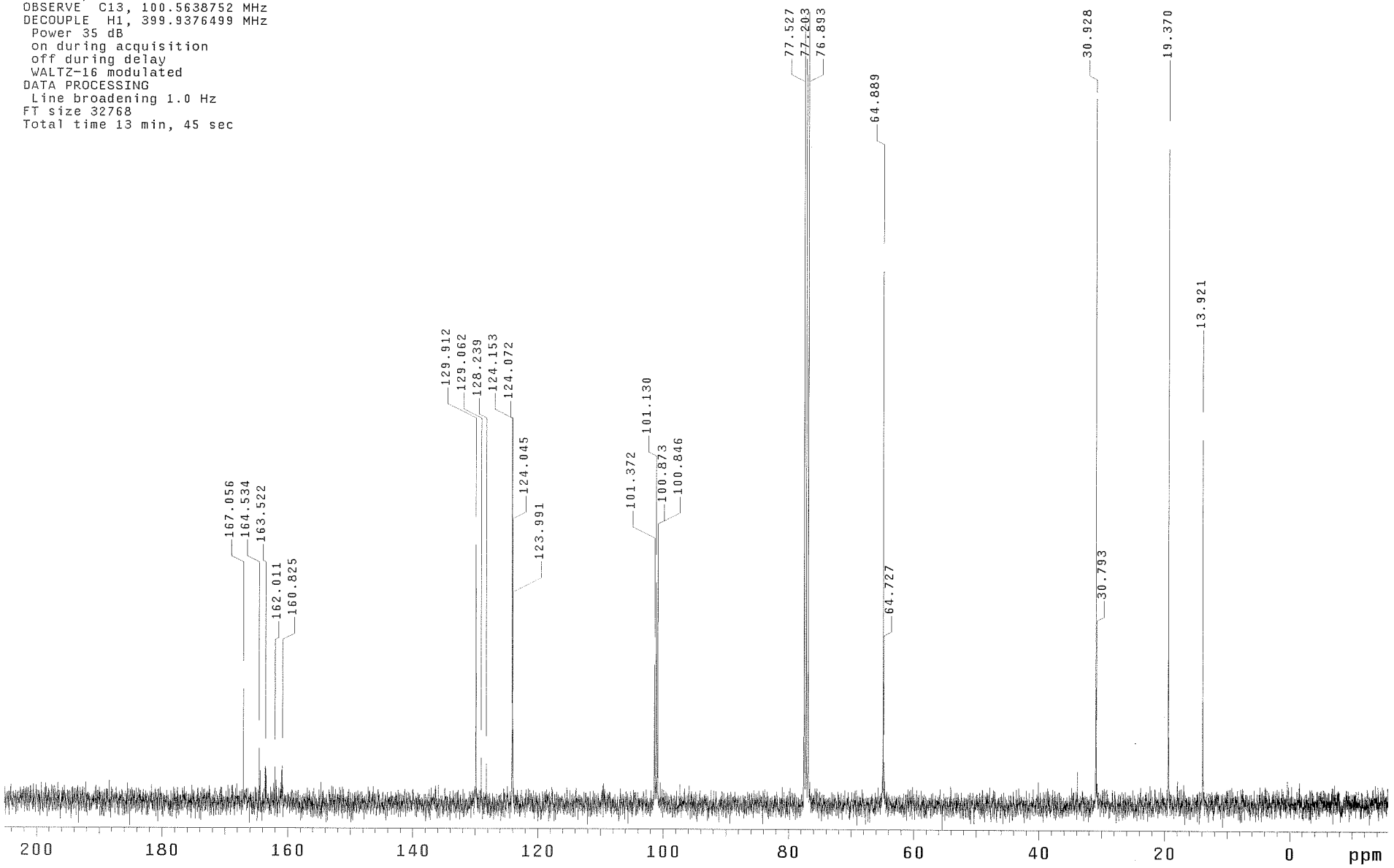
Pulse Sequence: s2pul

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
800 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 13 min, 45 sec



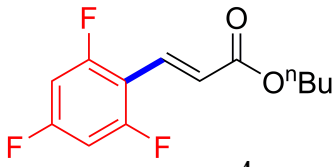
4c



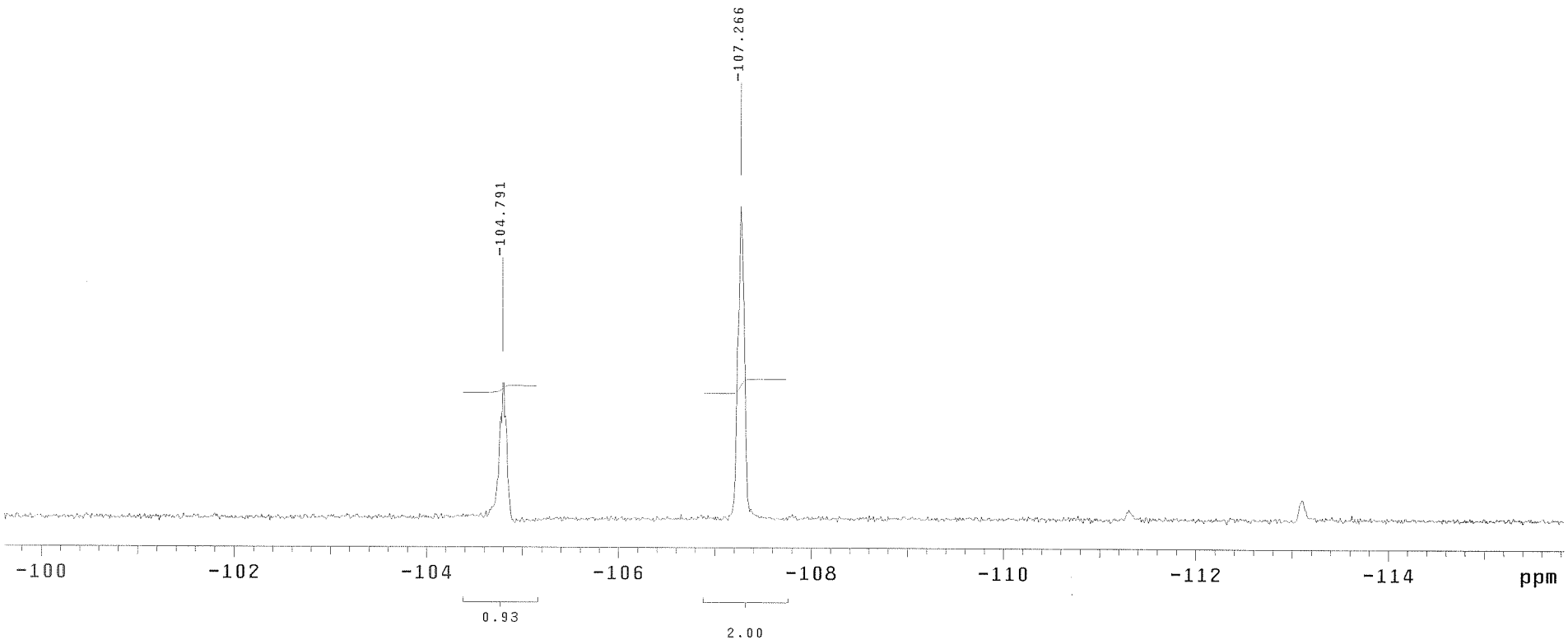
zsun-IV-22-3-F19

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Operator: zsun
File: zsun-IV-22-3-F19
Mercury-300BB "vnmr300"

 Relax. delay 4.000 sec
 Pulse 30.6 degrees
 Acq. time 0.300 sec
 Width 50000.0 Hz
 104 repetitions
OBSERVE F19, 282.4095227 MHz
DATA PROCESSING
 Line broadening 0.3 Hz
FT size 32768
Total time 32 min, 13 sec



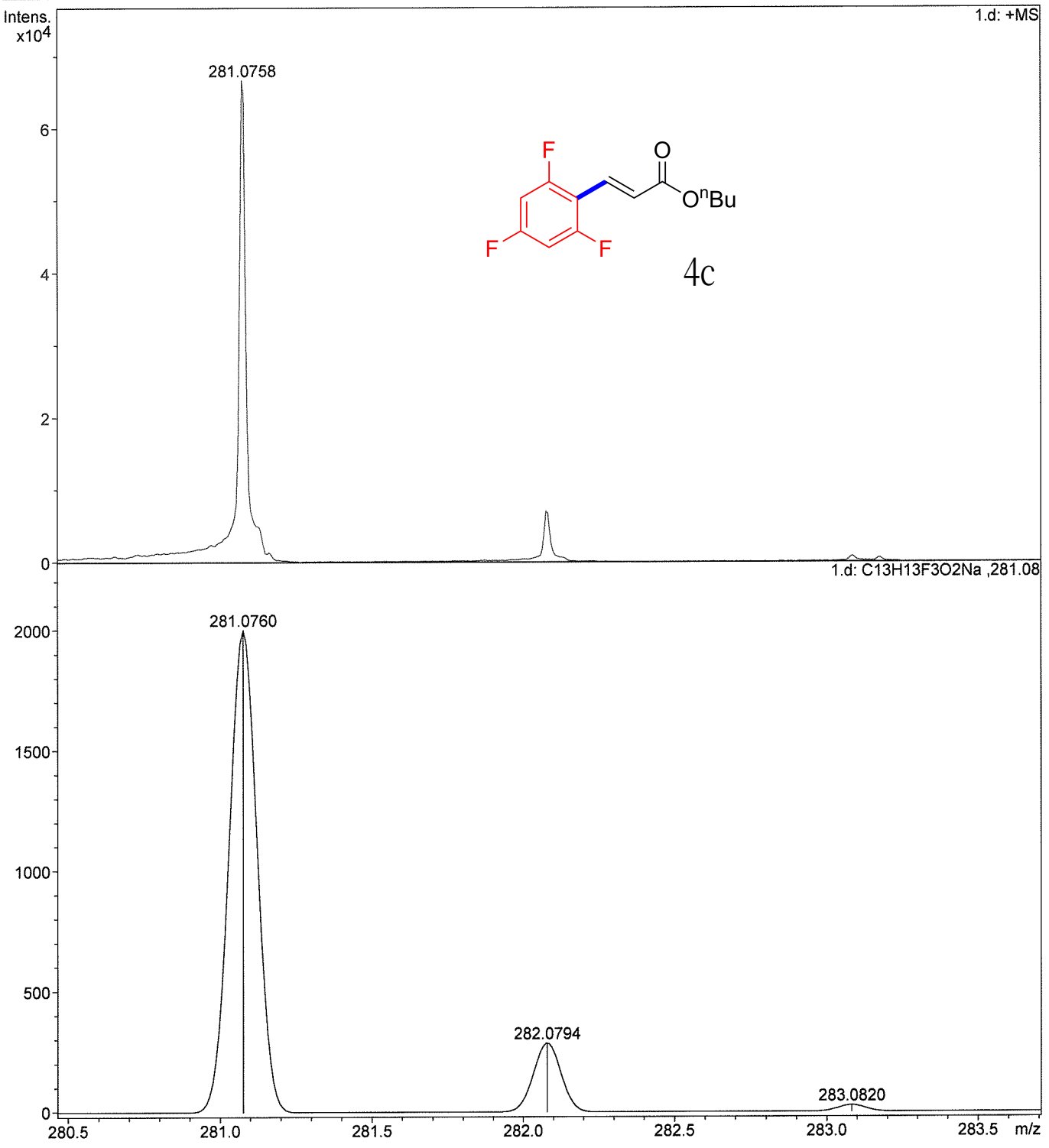
4c



Generic Display Report

Analysis Info

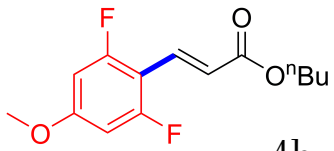
Acquisition Date	10/23/2009 3:57:29 PM		
Analysis Name	D:\Bruker\data\zhosun\IV_22-3\1.d		
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	IV_22-3	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		



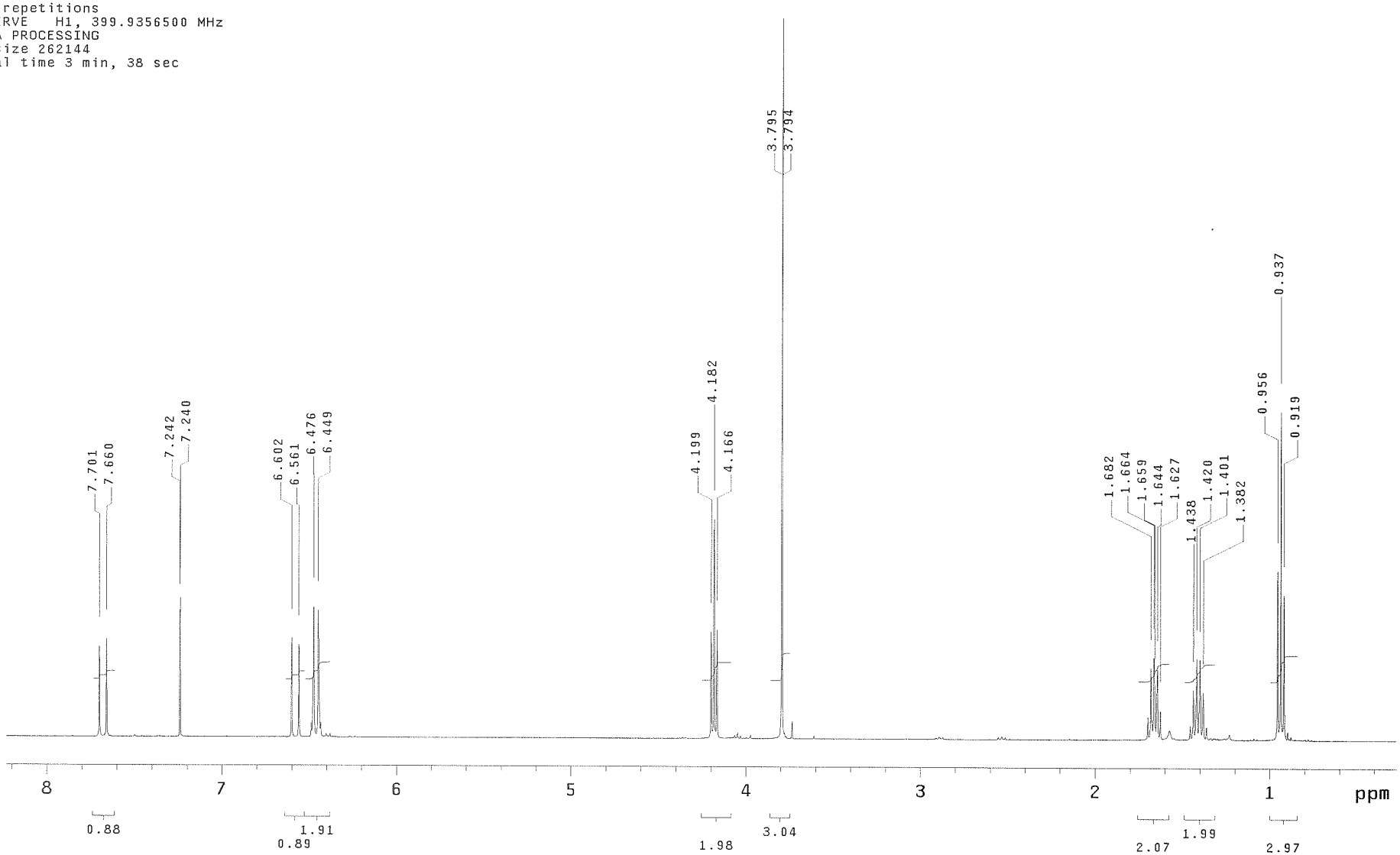
zsun-IV-22-8-H1

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
44 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



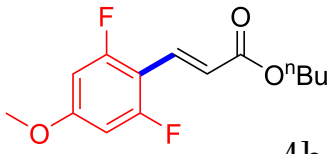
4b



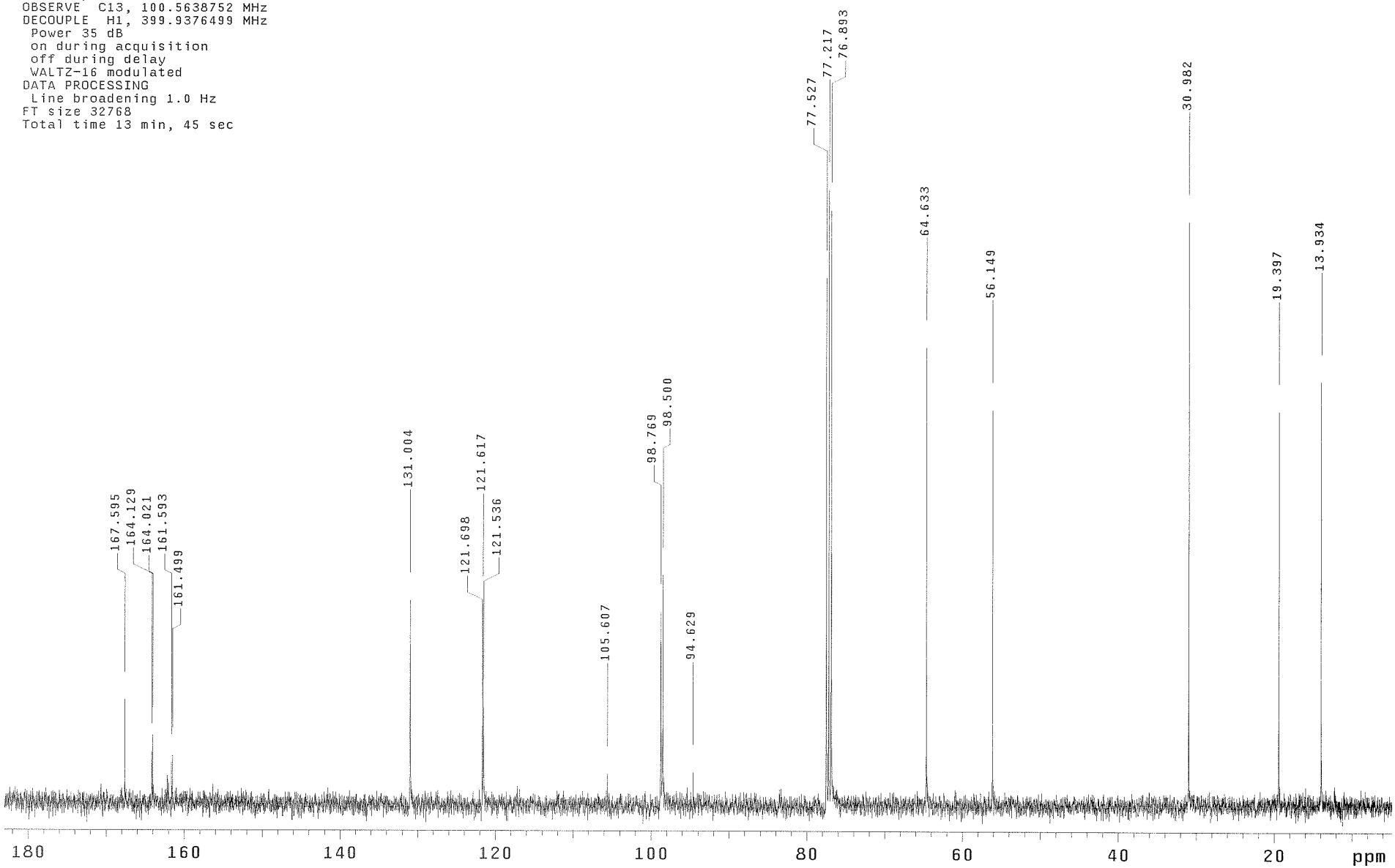
zsun-IV-22-8-C13

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
584 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 13 min, 45 sec



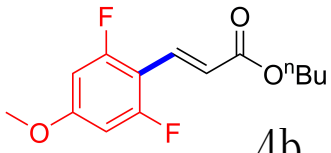
4b



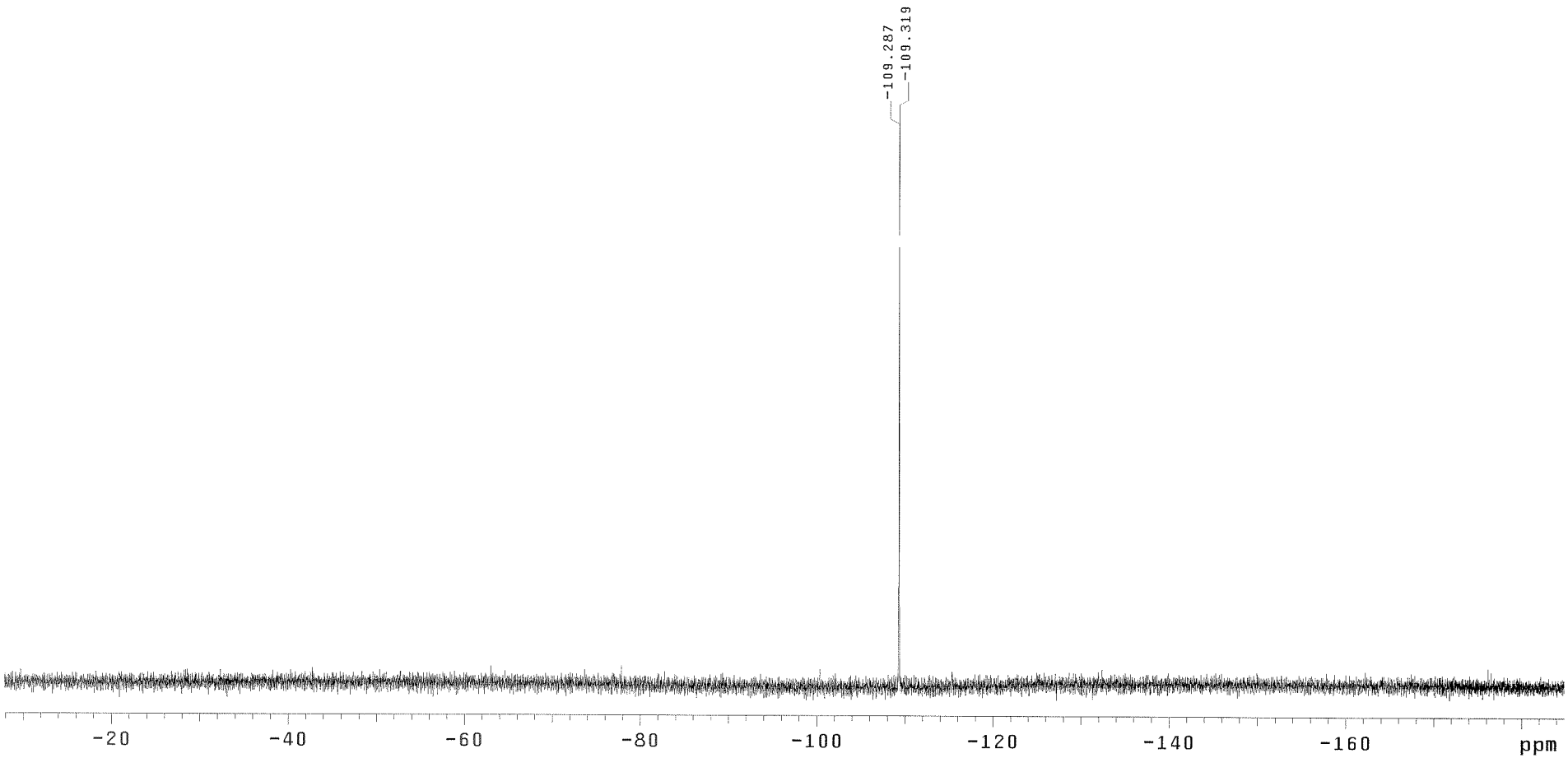
zsun-IV-22-8-F19

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Operator: zsun
Mercury-300BB "vnmr300"

 Relax. delay 4.000 sec
 Pulse 30.6 degrees
 Acq. time 0.300 sec
 Width 50000.0 Hz
 24 repetitions
OBSERVE F19, 282.4095258 MHz
DATA PROCESSING
 Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 0 sec



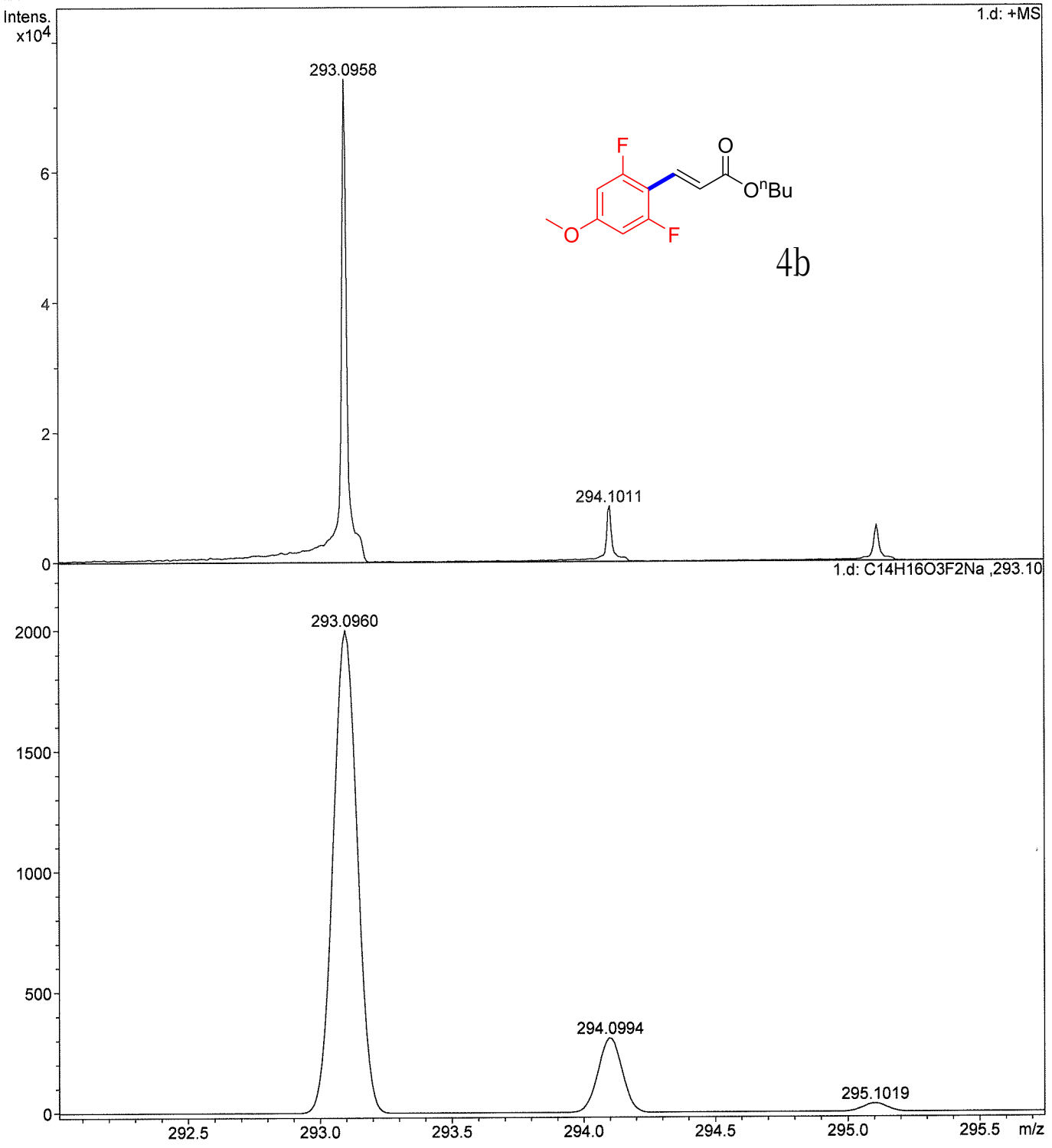
4b



Generic Display Report

Analysis Info

Acquisition Date	10/23/2009 4:15:14 PM		
Analysis Name	D:\Bruker\data\zhosun\IV_22-8\1.d		
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	IV_22-8	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		

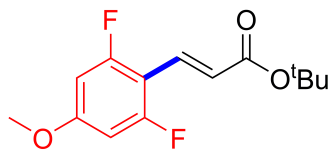


zsun-IV-35-1-H1

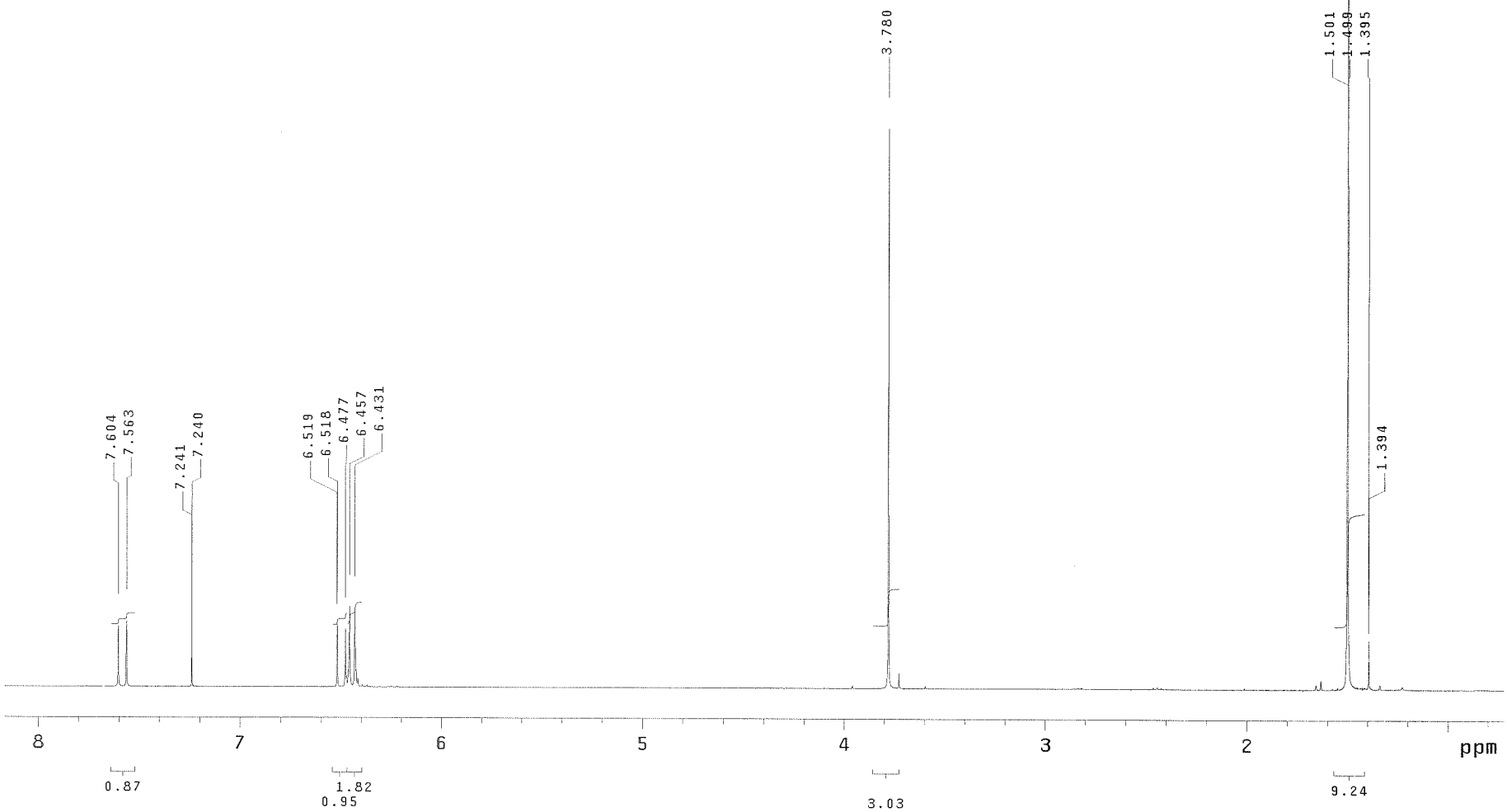
Pulse Sequence: s2pul

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
48 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



4h

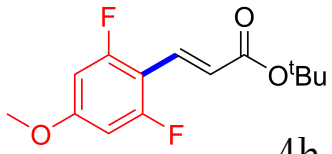


zsun-IV-35-1-H1

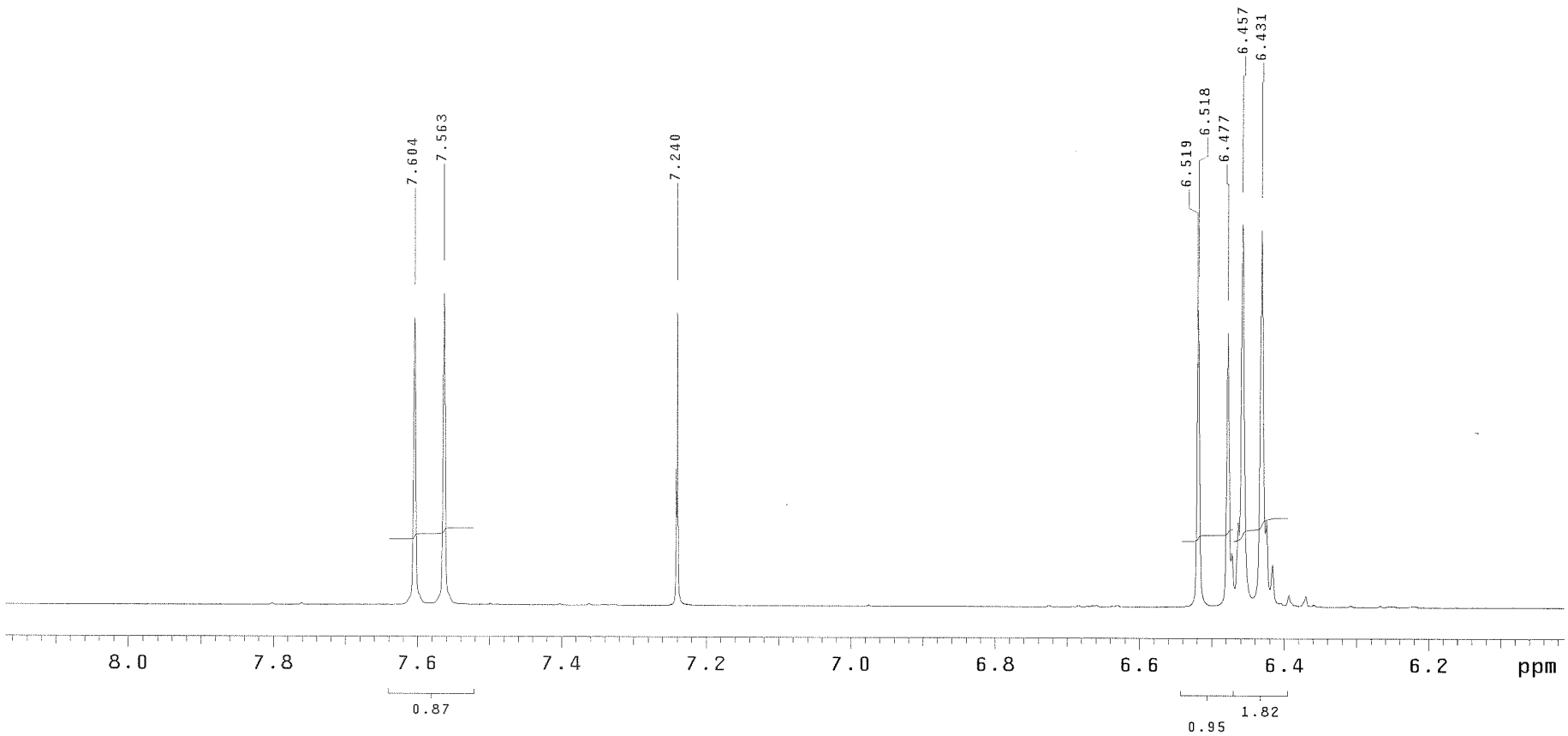
Pulse Sequence: s2pul

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



4h

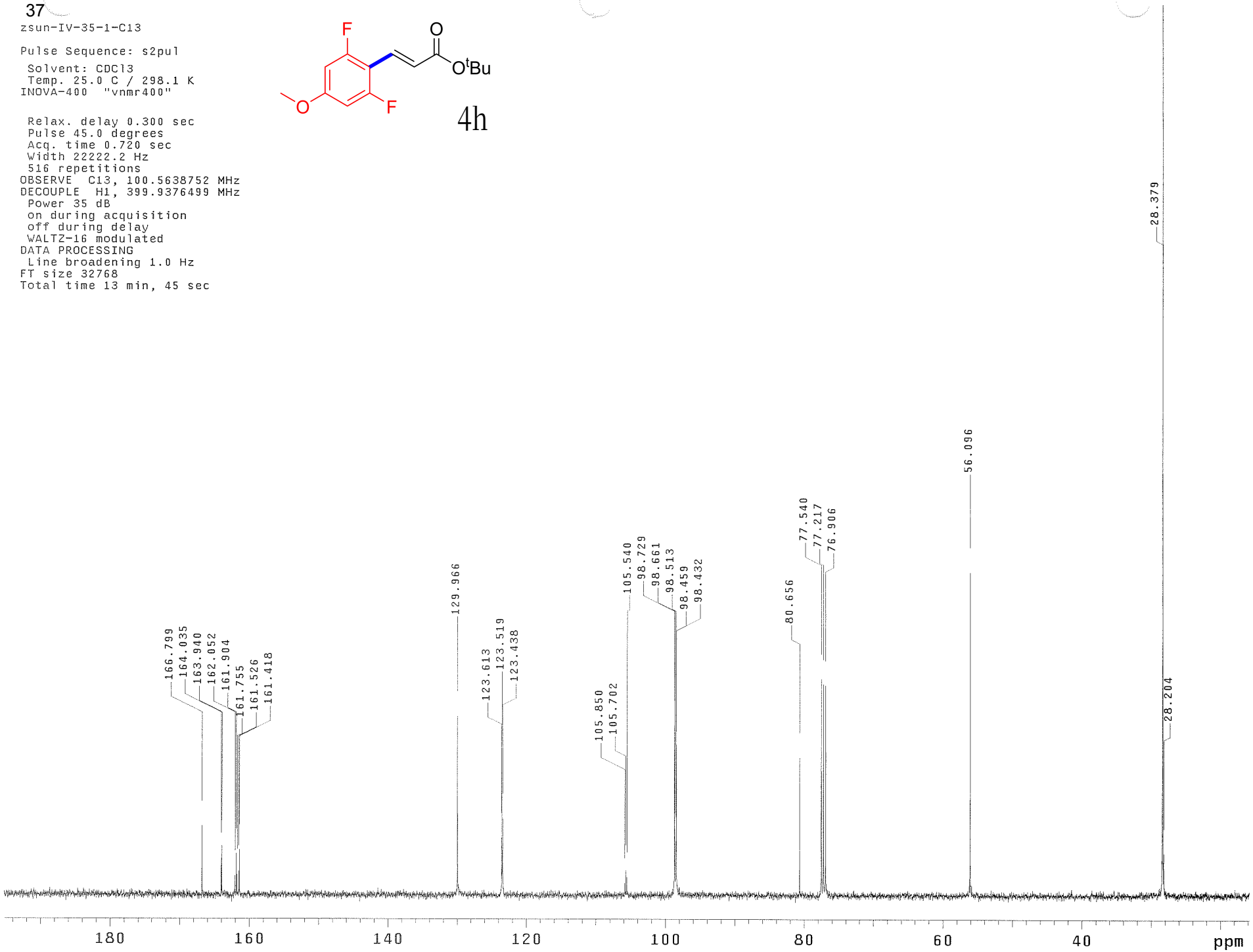


zsun-IV-35-1-C13

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
516 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 13 min, 45 sec

4h

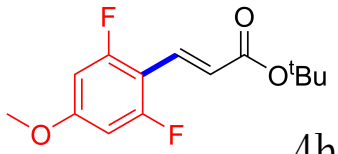


zsun-IV-35-1-C13

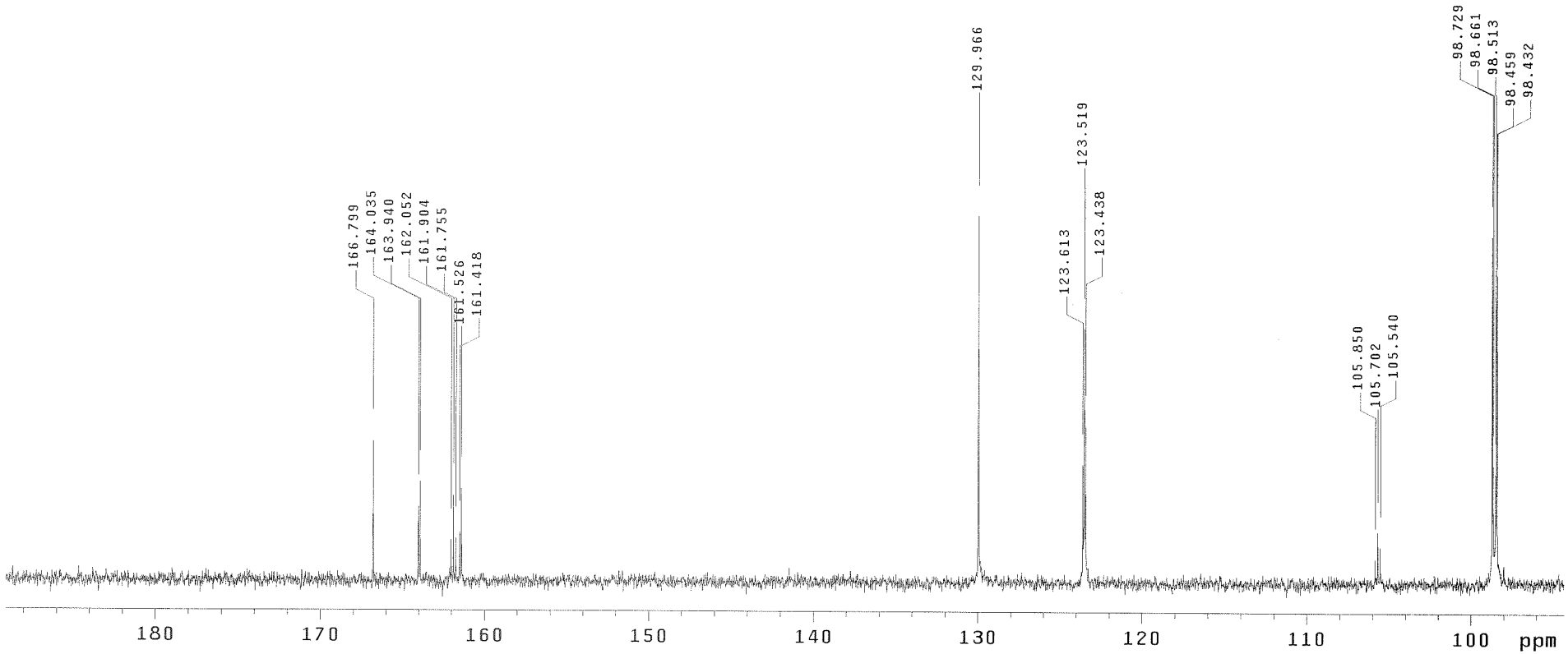
Pulse Sequence: s2pul

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
540 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 13 min, 45 sec



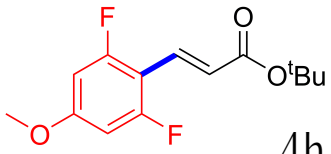
4h



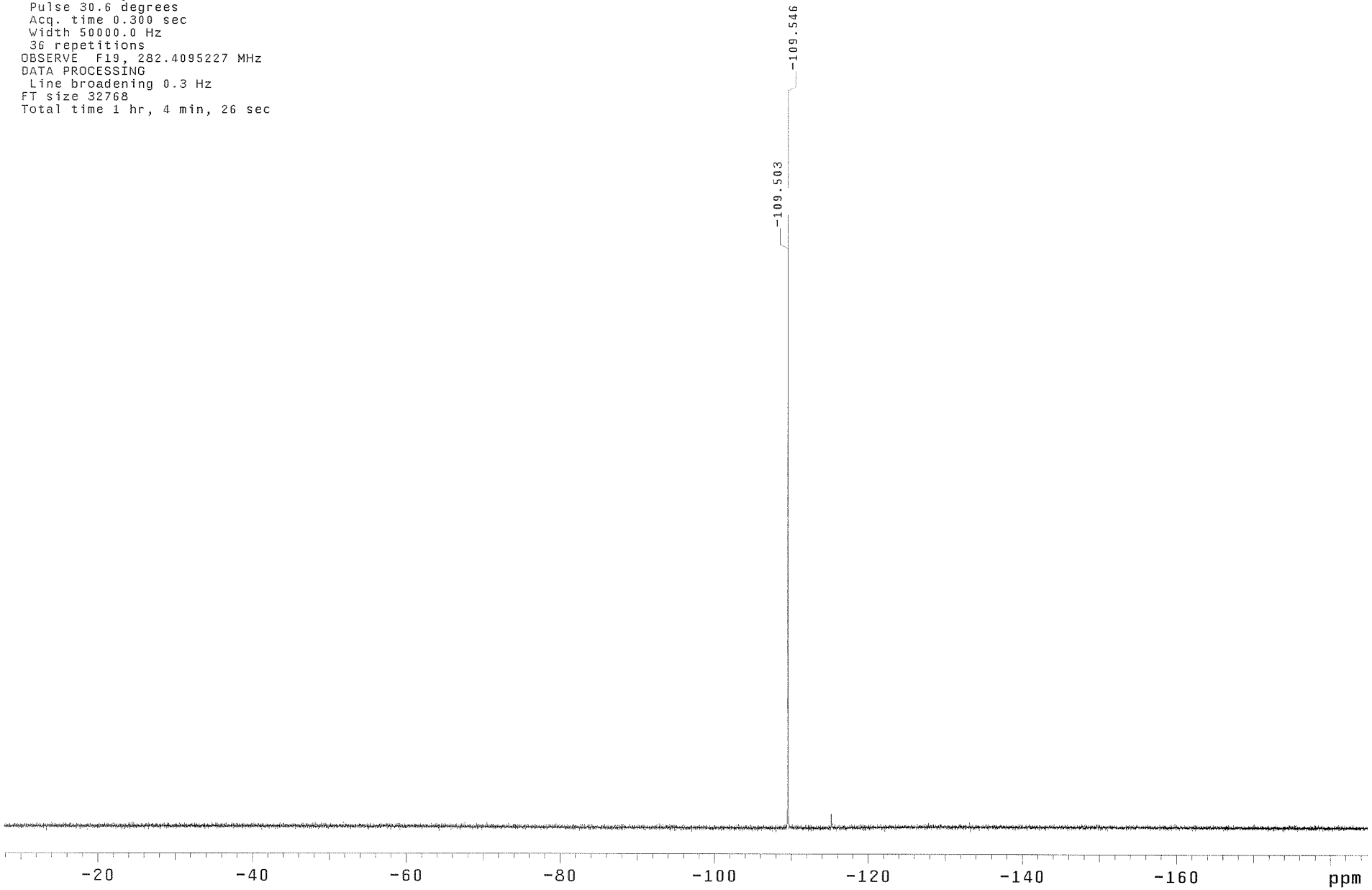
zsun-IV-35-1-F19

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Operator: zsun
Mercury-300BB "vnmr300"

 Relax. delay 4.000 sec
 Pulse 30.6 degrees
 Acq. time 0.300 sec
 Width 50000.0 Hz
 36 repetitions
OBSERVE F19, 282.4095227 MHz
DATA PROCESSING
 Line broadening 0.3 Hz
FT size 32768
Total time 1 hr, 4 min, 26 sec



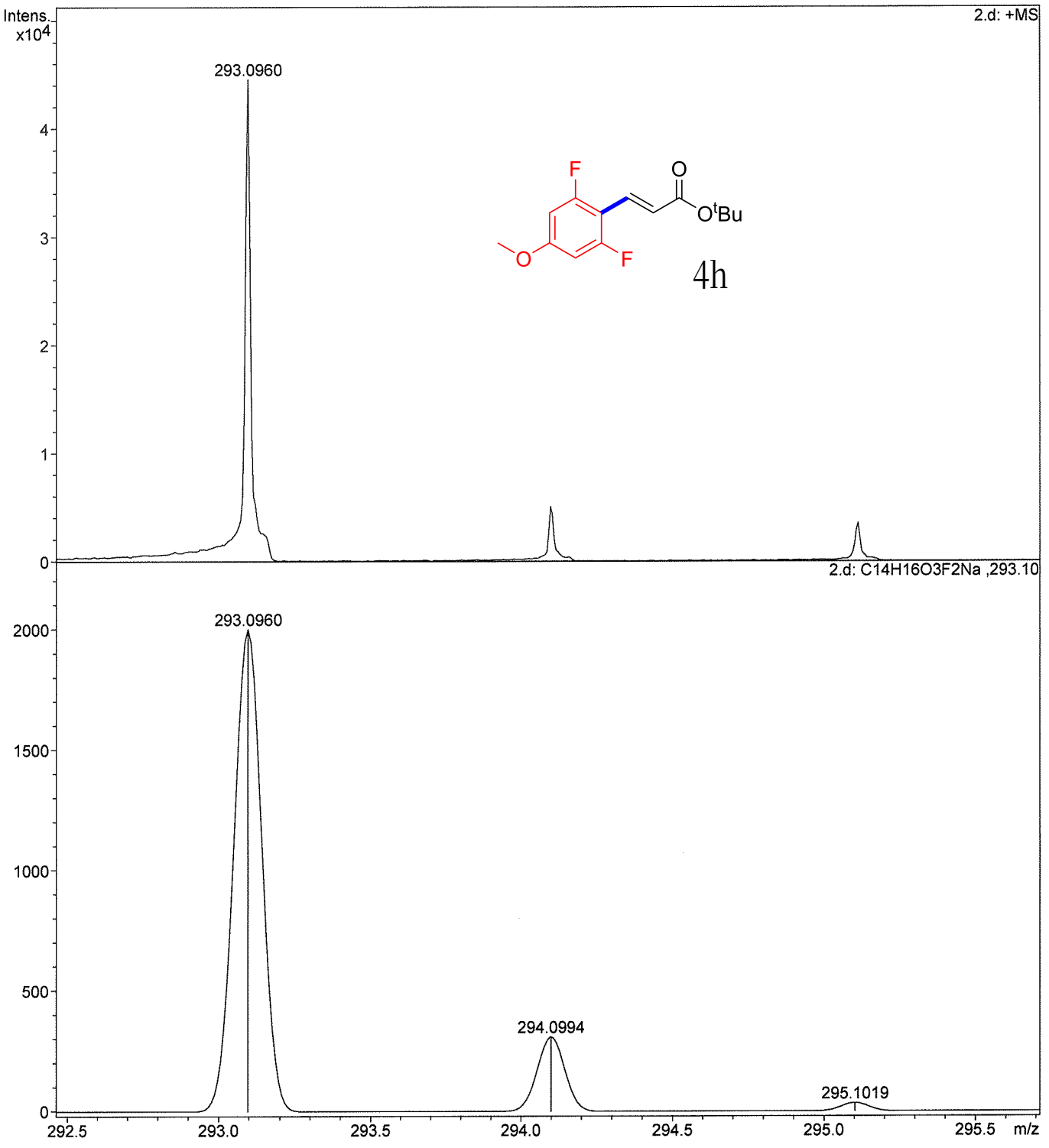
4h



Generic Display Report

Analysis Info

Acquisition Date	10/23/2009 4:13:09 PM		
Analysis Name	D:\Bruker\data\zhosun\IV_35-1\2.d		
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	IV_35-1	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments		



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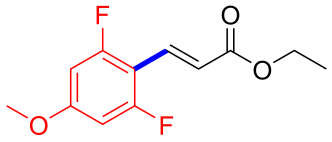
zsun-IV-35-2-H1

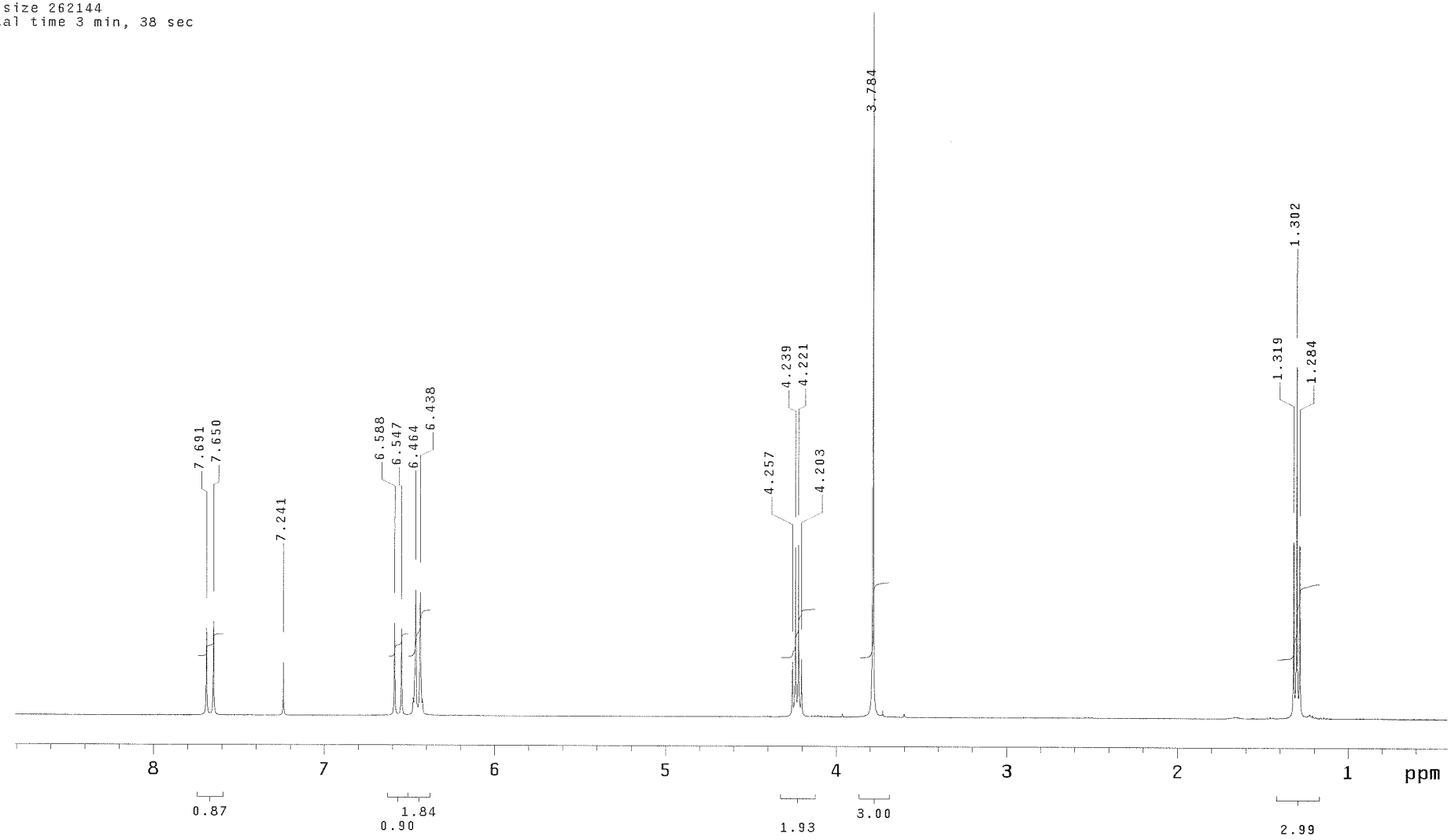
Pulse Sequence: s2pul

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
32 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
FT size 262144
Total time 3 min, 38 sec



4i

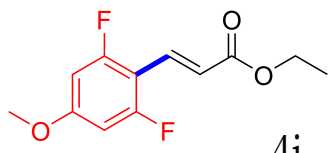


zsun-IV-35-2-C13

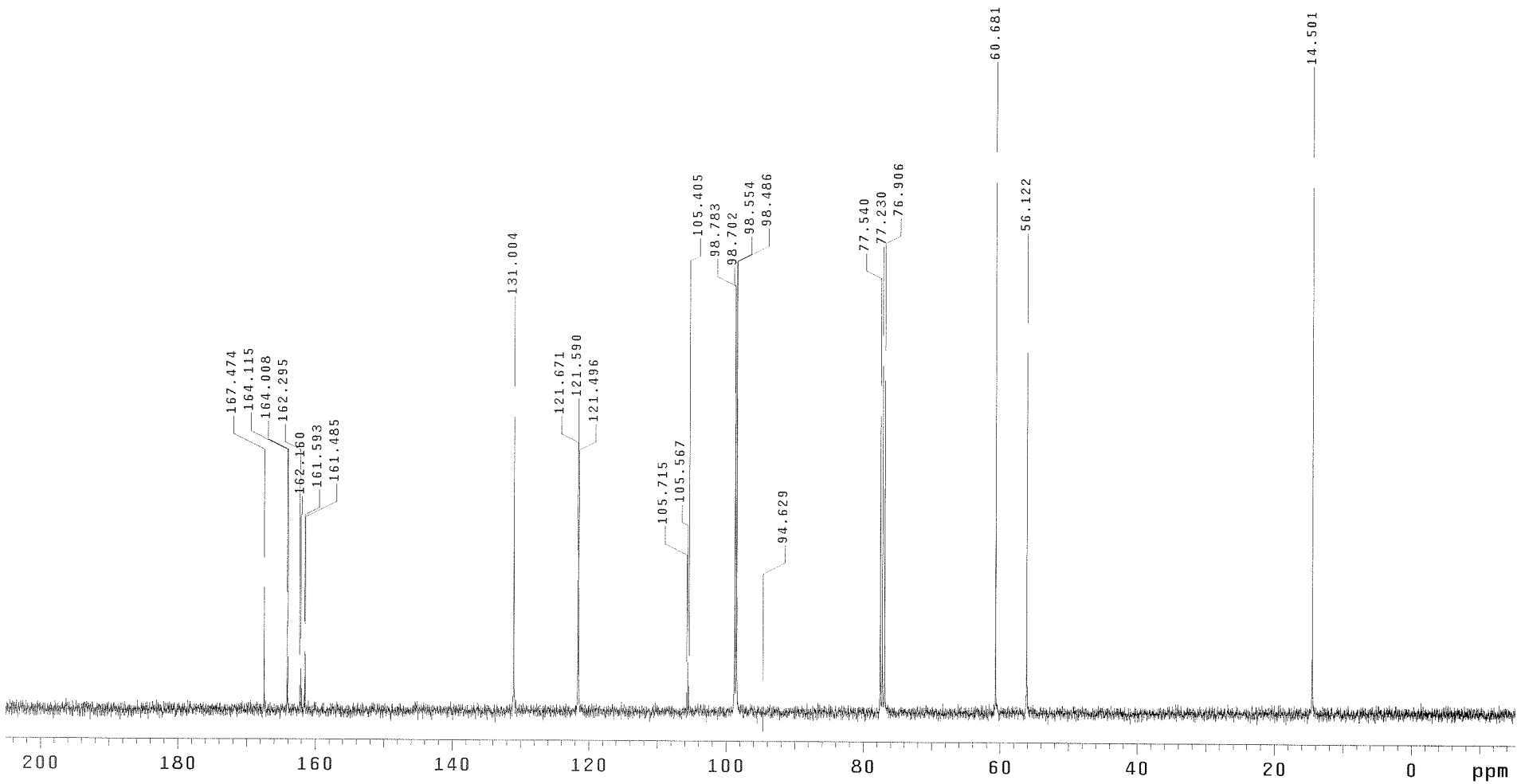
Pulse Sequence: s2pul

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
504 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 13 min, 45 sec



4i

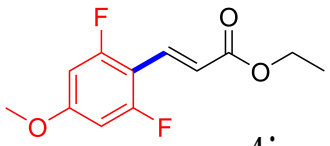


zsun-IV-35-2-C13

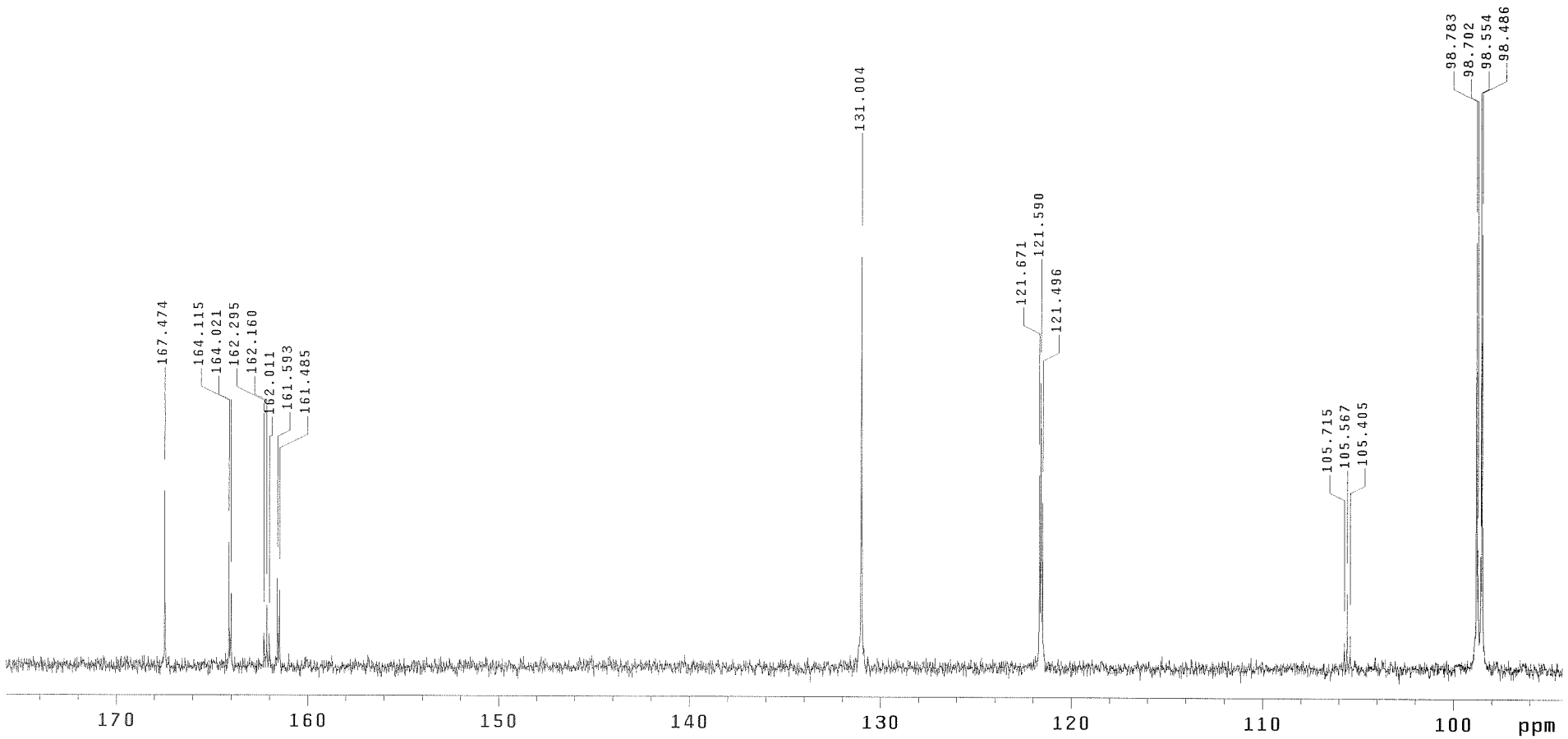
Pulse Sequence: s2pul

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
612 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 13 min, 45 sec



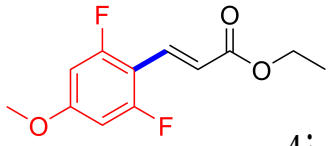
4i



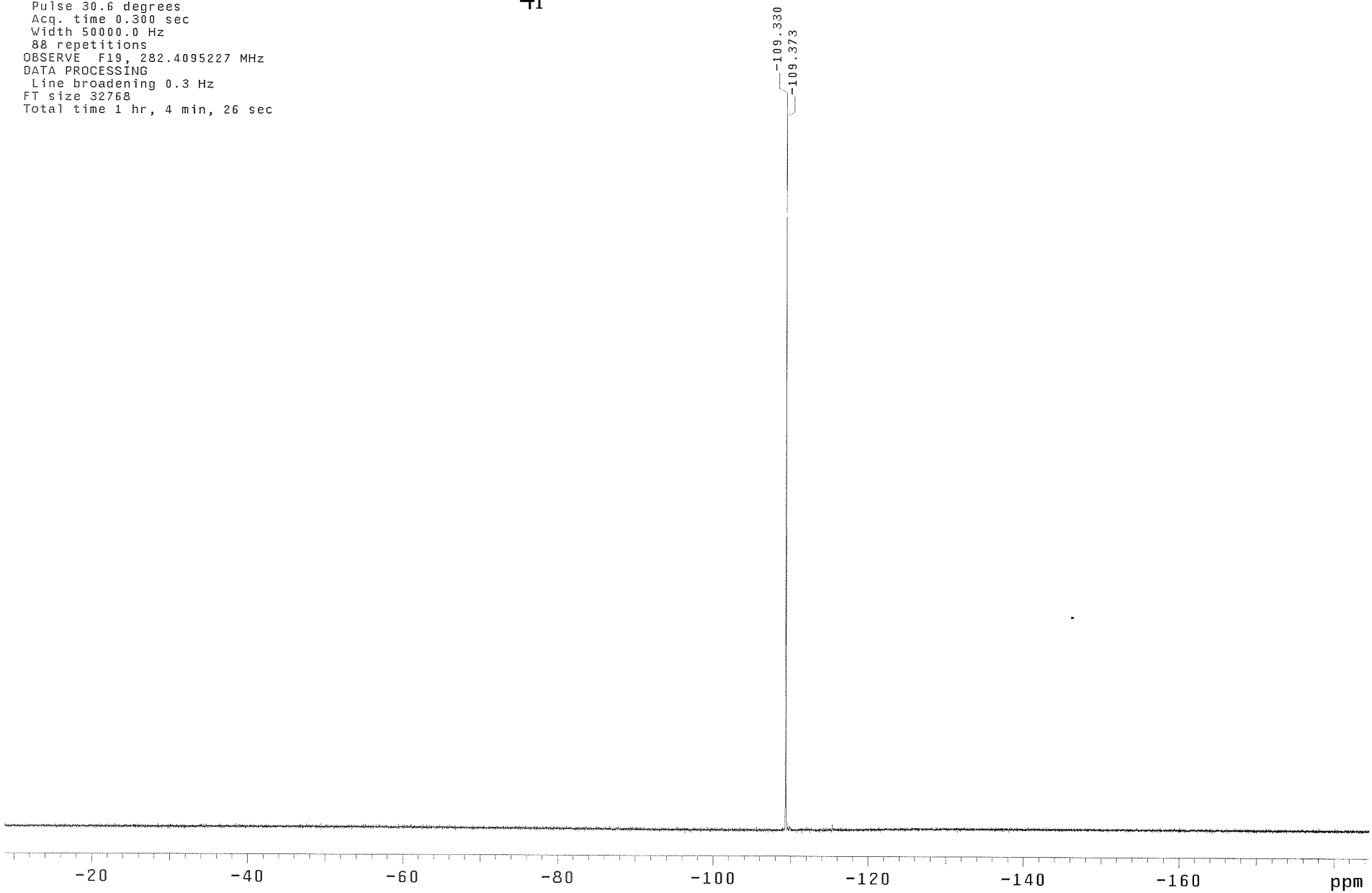
zsun-IV-35-2-F19

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Operator: zsun
Mercury-300BB "vnmr300"

 Relax. delay 4.000 sec
 Pulse 30.6 degrees
 Acq. time 0.300 sec
 Width 50000.0 Hz
 88 repetitions
OBSERVE F19, 282.4095227 MHz
DATA PROCESSING
 Line broadening 0.3 Hz
FT size 32768
Total time 1 hr, 4 min, 26 sec



4i



Generic Display Report

Analysis Info

Acquisition Date	10/23/2009 4:21:29 PM		
Analysis Name	D:\Bruker\data\zhosun\IV_35-2\1.d		
Method	user-1pass_pos_mid.tofpar	Operator	operator name
Sample Name	IV_35-2	Instrument	BioTOF II
Comment	Free format commentsFree format commentsFree format comments