

Supporting Information

Palladium-Catalyzed Oxidative Carbocyclization/Arylation of Enallenes

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Contents

General information	S2
Preparation of starting materials	S2
Selected optimization results	S3
General procedures and spectral data	S4
Copies of spectra	S10

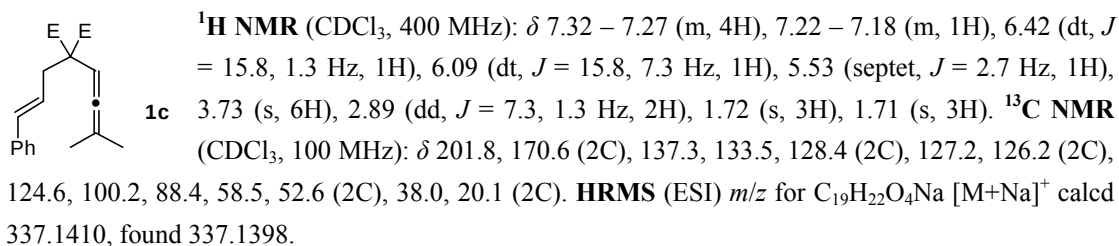
Experimental section

General information

Unless otherwise noted, all reagents were used as received from commercial suppliers. Pd(OAc)₂ was obtained from Pressure Chemicals and used without further purification. All the arylboronic acids were purchased from Sigma-Aldrich Co. LLC.. Palladium-catalyzed oxidative carbocyclization/arylation of enallenes was performed without any efforts to exclude moisture, i.e. reagent grade THF can be used without any drying/purification. The H₂O content of this THF was typically 50-150 ppm, measured using a Metrohm 831F (KF-titrator). Dry solvents for other necessary reactions (THF) were obtained from a VAC Solvent Purifier. Reactions were monitored using thin-layer chromatography (SiO₂). TLC plates were visualized with UV light (254 nm), iodine treatment or using Hanessians stain. Flash chromatography was carried out with 60Å (particle size 35-70 µm) normal flash silica gel. NMR spectra were recorded at 400 MHz (H) and at 100 MHz (C), respectively. Chemical shifts (δ) are reported in ppm, using the residual solvent peak in CDCl₃ (H: δ = 7.26 and C: δ = 77.0 ppm) as internal standard, and coupling constants (*J*) are given in Hz. HRMS were recorded using ESI-TOF techniques. The stereochemistry of **2d** and **2e**, respectively, was determined by comparing coupling constants with analogous compounds from our previous studies.¹

Preparation of starting materials

Enallene substrates **1a** – **1f** were prepared as reported in the literature.² Characterization of enallene **1c** is given below:



(1) Persson, A. K. Å.; Jiang, T.; Johnson, M.; Bäckvall, J.-E. *Angew. Chem. Int. Ed.* **2011**, 50, 6155.

(2) Franzén, J.; Bäckvall, J.-E. *J. Am. Chem. Soc.* **2003**, 125, 6056.

Optimization of solvent in carbocyclization/arylation of enallene 1a to 2aa

To a solution enallene **1a** (23.8 mg, 0.1 mmol) in the indicated solvent (1 mL) of was added Pd(OAc)₂ (1.1 mg, 0.005 mmol, 5 mol%), BQ (13.0 mg, 0.12 mmol) and PhB(OH)₂ (12.2 mg, 0.1 mmol). The mixture was heated at 40 °C for 16 hours. The solvent was evaporated followed by the addition of anisole (11.0 μL, 0.1 mmol) as the internal standard. The crude mixture was analyzed by ¹H NMR spectroscopy and the results are summarized in Table 1.

Table 1. Solvent effect

Entry	Solvent	Yield of 2a (%) ^a	Yield of 4a (%) ^a
1	acetone	90	7
2 ^b	acetonitrile	0	0
3	DCM	67	4
4	DCE	64	5
5	dioxane	86	2
6	ethyl acetate	63	3
7	THF	92	4
8	toluene	53	1

^a crude ¹H NMR yield. ^b No reaction in acetonitrile.

Optimization of temperature in carbocyclization/arylation of enallene 1a to 2aa

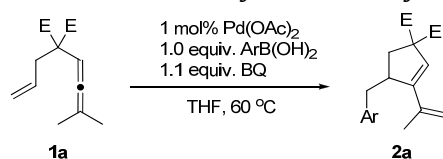
To a solution of enallene **1a** (47.6 mg, 0.2 mmol) in THF (2 mL) was added Pd(OAc)₂ (0.5 mg, 0.002 mmol, 1 mol%), BQ (23.8 mg, 0.22 mmol) and PhB(OH)₂ (24.4 mg, 0.2 mmol). The mixture was heated at the indicated temperature until completion. The solvent was evaporated followed by the addition of anisole (22.0 μL, 0.2 mmol) as the internal standard. The crude mixture was analyzed by ¹H NMR spectroscopy and the results are summarized in Table 2.

Table 2. Temperature influence

Entry	Temp (°C)	Time (h)	Yield of 2a (%) ^{a,b}
1	40	16	77
2	50	3.5	83
3	60	2	86 (83)
4	70	1	75

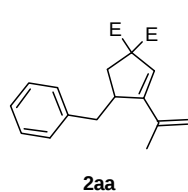
^a crude ¹H NMR yield. ^b isolated yield in parenthesis.

General procedure for the oxidative carbocyclization/arylation of enallene 1a

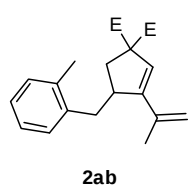


To a solution of enallene **1a** (47.6 mg, 0.2 mmol) in THF (2 mL) was added Pd(OAc)₂ (0.5 mg, 0.002 mmol, 1 mol%), BQ (23.8 mg, 0.22 mmol) and PhB(OH)₂ (24.4 mg, 0.2 mmol). The mixture was heated at 60 °C for 2 hours. The solvent was evaporated and the residue was purified by flash column chromatography (pentane/ethyl acetate v/v 40:1) giving the arylated product **2a** as a colorless oil (53 mg, 83 %).

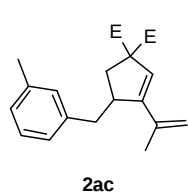
Spectral data of compounds 2aa – 2ar (NMR and HRMS)



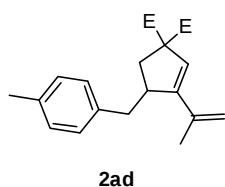
¹H NMR (CDCl₃, 400 MHz): δ 7.33 – 7.21 (m, 5H), 5.82 (br. s, 1H), 5.24 (br. s, 1H), 5.18 (br. s, 1H), 3.77 (s, 3H), 3.71 (s, 3H), 3.28 (m, 1H), 3.13 (dd, J = 13.9, 3.0 Hz, 1H), 2.54 (dd, J = 13.9, 2.2 Hz, 1H), 2.37 (dd, J = 14.0, 9.0 Hz, 1H), 2.35 (dd, J = 13.7, 11.5 Hz, 1H), 2.00 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.9, 171.7, 151.4, 140.8, 137.9, 129.0 (2C), 128.3 (2C), 126.0, 124.2, 115.4, 65.3, 52.8, 52.7, 46.2, 40.0, 36.0, 21.2. **HRMS** (ESI) m/z for C₁₉H₂₂O₄Na [M+Na]⁺ calcd 337.1410, found 337.1403.



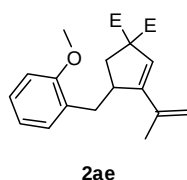
¹H NMR (CDCl₃, 400 MHz): δ 7.29 (m, 1H), 7.19 – 7.10 (m, 3H), 5.84 (br. s, 1H), 5.21 (br. s, 1H), 5.15 (br. s, 1H), 3.78 (s, 3H), 3.72 (s, 3H), 3.37 (m, 1H), 3.14 (dd, J = 14.3, 3.3 Hz, 1H), 2.51 (dd, J = 14.3, 2.6 Hz, 1H), 2.46 – 2.38 (m, 2H), 2.35 (s, 3H), 2.01 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.83 (2C), 152.0, 138.8, 138.0, 136.2, 130.2, 129.7, 126.1, 125.8, 124.2, 115.3, 65.4, 52.8, 52.7, 43.9, 37.4, 36.1, 21.3, 19.8. **HRMS** (ESI) m/z for C₂₀H₂₄O₄Na [M+Na]⁺ calcd 351.1567, found 351.1573.



¹H NMR (CDCl₃, 400 MHz): δ 7.20 (m, 1H), 7.06 – 7.02 (m, 3H), 5.82 (br. s, 1H), 5.24 (br. s, 1H), 5.18 (br. s, 1H), 3.77 (s, 3H), 3.71 (s, 3H), 3.27 (m, 1H), 3.10 (dd, J = 13.9, 2.9 Hz, 1H), 2.55 (dd, J = 13.9, 2.2 Hz, 1H), 2.41 – 2.31 (m, 2H), 2.36 (s, 3H), 2.00 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.9, 171.7, 151.5, 140.8, 137.9, 137.8, 129.7, 128.2, 126.7, 126.0, 124.2, 115.4, 65.3, 52.7, 52.7, 46.2, 39.9, 36.1, 21.4, 21.1. **HRMS** (ESI) m/z for C₂₀H₂₄O₄Na [M+Na]⁺ calcd 351.1567, found 351.1569.

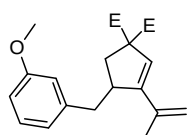


¹H NMR (CDCl₃, 400 MHz): δ 7.13 (m, 4H), 5.81 (br. s, 1H), 5.24 (br. s, 1H), 5.18 (br. s, 1H), 3.77 (s, 3H), 3.71 (s, 3H), 3.25 (m, 1H), 3.09 (dd, J = 13.9, 2.9 Hz, 1H), 2.54 (dd, J = 13.9, 2.2 Hz, 1H), 2.39 – 2.31 (m, 2H), 2.34 (s, 3H), 2.00 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.9, 171.7, 151.4, 137.8, 137.7, 135.4, 129.0 (2C), 128.9 (2C), 124.1, 115.4, 65.3, 52.7, 52.7, 46.2, 39.5, 36.0, 21.1, 21.0. **HRMS** (ESI) m/z for C₂₀H₂₄O₄Na [M+Na]⁺ calcd 351.1567, found 351.1562.



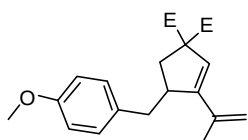
2ae

¹H NMR (CDCl₃, 400 MHz): δ 7.27 (dd, J = 7.4, 1.7 Hz, 1H), 7.20 (ddd, J = 7.9, 7.9, 1.7 Hz, 1H), 6.91 (ddd, J = 7.4, 7.4, 1.0 Hz, 1H), 6.85 (m, 1H), 5.78 (br. s, 1H), 5.49 (br. s, 1H), 5.18 (br. s, 1H), 3.84 (s, 3H), 3.79 (s, 3H), 3.70 (s, 3H), 3.34 (m, 1H), 3.28 (dd, J = 13.2, 2.6 Hz, 1H), 2.55 (dd, J = 13.9, 1.9 Hz, 1H), 2.28 (dd, J = 13.9, 8.6 Hz, 1H), 2.17 (dd, J = 13.2, 11.6 Hz, 1H), 1.99 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 172.1, 171.9, 157.6, 152.3, 137.8, 131.3, 129.4, 127.3, 123.6, 120.3, 115.9, 110.0, 65.4, 55.0, 52.8, 52.7, 44.0, 36.2, 35.6, 21.1. **HRMS** (ESI) m/z for C₂₀H₂₄O₅Na [M+Na]⁺ calcd 367.1516, found 367.1534.



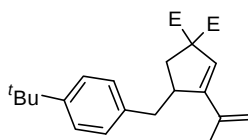
2af

¹H NMR (CDCl₃, 400 MHz): δ 7.21 (t, J = 7.8 Hz, 1H), 6.84 (d, J = 7.6 Hz, 1H), 6.80 (m, 1H), 6.75 (m, 1H), 5.81 (br. s, 1H), 5.22 (br. s, 1H), 5.17 (br. s, 1H), 3.81 (s, 3H), 3.76 (s, 3H), 3.70 (s, 3H), 3.28 (m, 1H), 3.09 (dd, J = 13.9, 3.0 Hz, 1H), 2.53 (dd, J = 13.9, 2.2 Hz, 1H), 2.41 – 2.32 (m, 2H), 1.99 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.8, 171.6, 159.6, 151.3, 142.4, 137.8, 129.2, 124.2, 121.4, 115.4, 114.9, 111.1, 65.3, 55.0, 52.7, 52.7, 46.0, 40.0, 36.1, 21.1. **HRMS** (ESI) m/z for C₂₀H₂₄O₅Na [M+Na]⁺ calcd 367.1516, found 367.1515.



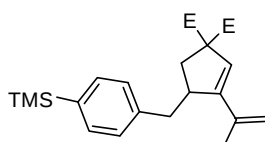
2ag

¹H NMR (CDCl₃, 400 MHz): δ 7.16 (m, 2H), 6.84 (m, 2H), 5.80 (br. s, 1H), 5.21 (br. s, 1H), 5.16 (br. s, 1H), 3.79 (s, 3H), 3.75 (s, 3H), 3.70 (s, 3H), 3.22 (m, 1H), 3.05 (dd, J = 13.9, 3.1 Hz, 1H), 2.52 (dd, J = 13.9, 2.2 Hz, 1H), 2.35 (dd, J = 14.0, 8.7 Hz, 1H), 2.31 (dd, J = 13.9, 11.2 Hz, 1H), 1.98 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.9, 171.7, 157.9, 151.4, 137.9, 132.9 (2C), 129.9, 124.1, 115.4, 113.7 (2C), 65.3, 55.2, 52.8, 52.7, 46.4, 39.0, 36.0, 21.1. **HRMS** (ESI) m/z for C₂₀H₂₄O₅Na [M+Na]⁺ calcd 367.1516, found 367.1538.



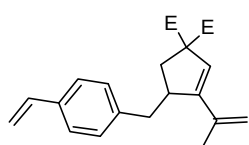
2ah

¹H NMR (CDCl₃, 400 MHz): δ 7.33 (m, 2H), 7.18 (m, 2H), 5.82 (br. s, 1H), 5.24 (br. s, 1H), 5.18 (br. s, 1H), 3.77 (s, 3H), 3.71 (s, 3H), 3.28 (m, 1H), 3.11 (dd, J = 14.0, 3.0 Hz, 1H), 2.54 (dd, J = 14.0, 2.3 Hz, 1H), 2.39 (dd, J = 14.0, 8.6 Hz, 1H), 2.35 (dd, J = 14.0, 11.9 Hz, 1H), 2.00 (br. s, 3H), 1.33 (s, 9H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.8, 171.7, 151.5, 148.7, 137.8, 137.7, 128.6 (2C), 125.2 (2C), 124.1, 115.4, 65.3, 52.7, 52.6, 46.1, 39.4, 36.2, 34.3, 31.3 (3C), 21.1. **HRMS** (ESI) m/z for C₂₃H₃₀O₄Na [M+Na]⁺ calcd 393.2036, found 393.2050.



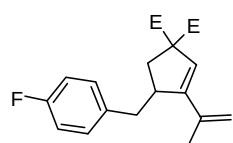
2ai

¹H NMR (CDCl₃, 400 MHz): δ 7.48 (m, 2H), 7.26 (m, 2H), 5.84 (br. s, 1H), 5.25 (br. s, 1H), 5.15 (br. s, 1H), 3.77 (s, 3H), 3.71 (s, 3H), 3.31 (m, 1H), 3.14 (dd, J = 14.0, 2.9 Hz, 1H), 2.55 (dd, J = 14.0, 2.2 Hz, 1H), 2.42 – 2.36 (m, 2H), 2.01 (br. s, 3H), 0.28 (s, 9H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.8, 171.6, 151.4, 141.4, 137.8, 137.5, 133.4 (2C), 128.4 (2C), 124.2, 115.4, 65.3, 52.7, 52.6, 46.0, 39.9, 36.1, 21.1, – 1.1 (3C). **HRMS** (ESI) m/z for C₂₂H₃₀O₄SiNa [M+Na]⁺ calcd 409.1806, found 409.1803.



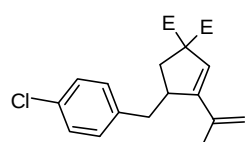
2aj

¹H NMR (CDCl₃, 400 MHz): δ 7.35 (m, 2H), 7.21 (m, 2H), 6.71 (dd, J = 17.6, 10.9 Hz, 1H), 5.82 (br. s, 1H), 5.72 (dd, J = 17.6, 0.8 Hz, 1H), 5.23 (br. s, 1H), 5.21 (dd, J = 10.9, 0.8 Hz, 1H), 5.18 (br. s, 1H), 3.75 (s, 3H), 3.70 (s, 3H), 3.27 (m, 1H), 3.10 (dd, J = 14.0, 3.1 Hz, 1H), 2.53 (dd, J = 14.0, 2.3 Hz, 1H), 2.39 (dd, J = 13.9, 11.2 Hz, 1H), 2.37 (dd, J = 13.9, 8.6 Hz, 1H), 2.00 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.8, 171.6, 151.3, 140.5, 137.8, 136.6, 135.4, 129.2 (2C), 126.2 (2C), 124.3, 115.4, 113.0, 65.3, 52.8, 52.7, 46.1, 40.0, 36.0, 21.1. **HRMS** (ESI) m/z for C₂₁H₂₄O₄Na [M+Na]⁺ calcd 363.1567, found 363.1572.



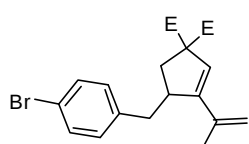
2ak

¹H NMR (CDCl₃, 400 MHz): δ 7.19 (m, 2H), 6.97 (m, 3H), 5.80 (br. s, 1H), 5.19 (br. s, 1H), 5.16 (br. s, 1H), 3.74 (s, 3H), 3.70 (s, 3H), 3.23 (m, 1H), 3.05 (dd, J = 14.0, 3.3 Hz, 1H), 2.49 (dd, J = 14.0, 2.2 Hz, 1H), 2.37 (dd, J = 13.9, 11.3 Hz, 1H), 2.34 (dd, J = 13.9, 8.6 Hz, 1H), 1.98 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.8, 171.6, 164.4 (d, J = 243.9 Hz, 1C), 151.1, 137.9, 136.4 (d, J = 3.4 Hz, 1C), 130.4 (d, J = 7.6 Hz, 2C), 124.4, 115.4, 115.0 (d, J = 21.0 Hz, 2C), 65.3, 52.8, 52.7, 46.2, 39.0, 35.9, 21.1. **HRMS** (ESI) m/z for C₁₉H₂₁O₄FNa [M+Na]⁺ calcd 355.1316, found 355.1315.



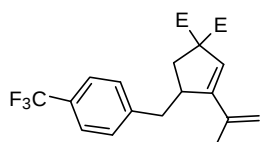
2al

¹H NMR (CDCl₃, 400 MHz): δ 7.26 (m, 2H), 7.17 (m, 2H), 5.80 (br. s, 1H), 5.18 (br. s, 1H), 5.16 (br. s, 1H), 3.74 (s, 3H), 3.70 (s, 3H), 3.23 (m, 1H), 3.05 (dd, J = 13.9, 3.3 Hz, 1H), 2.48 (dd, J = 13.9, 2.2 Hz, 1H), 2.38 (dd, J = 13.9, 11.2 Hz, 1H), 2.34 (dd, J = 13.9, 8.6 Hz, 1H), 1.98 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.8, 171.6, 151.0, 139.2, 137.8, 131.8, 130.4 (2C), 128.4 (2C), 124.5, 115.4, 65.3, 52.8, 52.8, 46.0, 39.2, 35.9, 21.1. **HRMS** (ESI) m/z for C₁₉H₂₁O₄ClNa [M+Na]⁺ calcd 371.1021, found 371.1029.



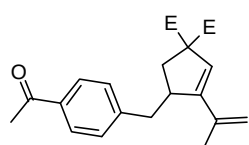
2am

¹H NMR (CDCl₃, 400 MHz): δ 7.41 (m, 2H), 7.11 (m, 2H), 5.80 (br. s, 1H), 5.18 (br. s, 1H), 5.16 (br. s, 1H), 3.74 (s, 3H), 3.70 (s, 3H), 3.23 (m, 1H), 3.03 (dd, J = 13.9, 3.2 Hz, 1H), 2.47 (dd, J = 13.9, 2.2 Hz, 1H), 2.37 (dd, J = 13.7, 11.2 Hz, 1H), 2.34 (dd, J = 13.7, 8.6 Hz, 1H), 1.98 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.8, 171.6, 151.0, 139.7, 137.8, 131.4 (2C), 130.8 (2C), 124.5, 119.8, 115.4, 65.3, 52.8, 52.8, 46.0, 39.3, 35.9, 21.1. **HRMS** (ESI) m/z for C₁₉H₂₁O₄BrNa [M+Na]⁺ calcd 415.0515, found 415.0511.



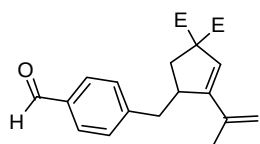
2an

¹H NMR (CDCl₃, 400 MHz): δ 7.55 (m, 2H), 7.35 (m, 2H), 5.83 (br. s, 1H), 5.20 (br. s, 1H), 5.18 (br. s, 1H), 3.74 (s, 3H), 3.70 (s, 3H), 3.28 (m, 1H), 3.14 (dd, J = 13.9, 3.0 Hz, 1H), 2.52 – 2.46 (m, 2H), 2.34 (dd, J = 13.9, 8.7 Hz, 1H), 1.99 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.7, 171.5, 150.9, 144.9, 137.8, 129.4, 128.4 (q, J = 32.2 Hz, 1C), 125.2 (q, J = 3.7 Hz, 1C), 124.3 (q, J = 271.8 Hz, 1C), 124.6, 115.5, 65.4, 52.8, 52.8, 45.8, 39.7, 35.8, 21.1. **HRMS** (ESI) m/z for C₂₀H₂₁O₄F₃Na [M+Na]⁺ calcd 405.1284, found 405.1301.



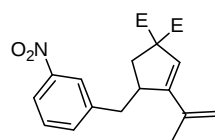
2ao

¹H NMR (CDCl₃, 400 MHz): δ 7.88 (m, 2H), 7.32 (m, 2H), 5.81 (br. s, 1H), 5.19 (br. s, 1H), 5.16 (br. s, 1H), 3.74 (s, 3H), 3.68 (s, 3H), 3.28 (m, 1H), 3.13 (dd, J = 13.9, 3.1 Hz, 1H), 2.57 (s, 3H), 2.48 – 2.42 (m, 2H), 2.32 (dd, J = 13.9, 8.6 Hz, 1H), 1.97 (s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 197.7, 171.6, 171.4, 151.0, 146.6, 137.7, 135.2, 129.2 (2C), 128.4 (2C), 124.5, 115.4, 65.3, 52.8, 52.7, 45.8, 39.9, 35.9, 26.4, 21.1. **HRMS** (ESI) m/z for C₂₁H₂₄O₅Na [M+Na]⁺ calcd 379.1516, found 379.1517.



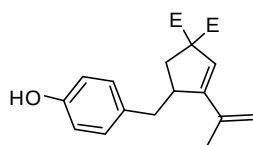
2ap

¹H NMR (CDCl₃, 400 MHz): δ 9.96 (s, 1H), 7.80 (m, 2H), 7.40 (m, 2H), 5.81 (br. s, 1H), 5.19 (br. s, 1H), 5.16 (br. s, 1H), 3.74 (s, 3H), 3.68 (s, 3H), 3.28 (m, 1H), 3.15 (dd, J = 13.9, 3.2 Hz, 1H), 2.52 – 2.45 (m, 2H), 2.31 (dd, J = 13.9, 8.6 Hz, 1H), 1.97 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 191.9, 171.6, 171.5, 150.9, 148.2, 137.7, 134.6, 129.8 (2C), 129.7 (2C), 124.6, 115.5, 65.3, 52.8, 52.7, 45.8, 40.1, 35.8, 21.1. **HRMS** (ESI) m/z for C₂₀H₂₂O₅Na [M+Na]⁺ calcd 365.1359, found 365.1356.



2aq

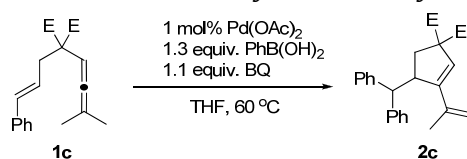
¹H NMR (CDCl₃, 400 MHz): δ 8.04 (m, 2H), 7.58 (d, J = 7.5 Hz, 1H), 7.45 (m, 1H), 5.82 (br. s, 1H), 5.18 (br. s, 1H), 5.17 (br. s, 1H), 3.72 (s, 3H), 3.68 (s, 3H), 3.30 (m, 1H), 3.15 (dd, J = 13.9, 3.3 Hz, 1H), 2.56 (dd, J = 13.9, 10.9 Hz, 1H), 2.43 (dd, J = 14.0, 2.3 Hz, 1H), 2.34 (dd, J = 14.0, 8.4 Hz, 1H), 1.97 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.5, 171.3, 150.5, 148.2, 142.6, 137.7, 135.5, 129.1, 124.8, 123.7, 121.2, 115.5, 65.2, 52.8, 52.7, 45.6, 39.3, 35.6, 21.0. **HRMS** (ESI) m/z for C₁₉H₂₁O₆NNa [M+Na]⁺ calcd 382.1261, found 382.1250.



2ar

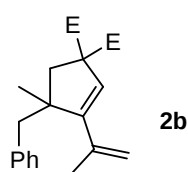
¹H NMR (CDCl₃, 400 MHz): δ 7.08 (m, 2H), 6.77 (m, 2H), 5.79 (br. s, 1H), 5.53 (br. s, 1H), 5.20 (br. s, 1H), 5.16 (br. s, 1H), 3.76 (s, 3H), 3.71 (s, 3H), 3.23 (m, 1H), 3.03 (dd, J = 14.0, 3.1 Hz, 1H), 2.51 (dd, J = 14.0, 2.3 Hz, 1H), 2.37 (dd, J = 14.0, 8.6 Hz, 1H), 2.30 (dd, J = 14.0, 11.1 Hz, 1H), 1.98 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 172.1, 172.0, 154.1, 151.6, 137.9, 132.6, 130.1 (2C), 124.0, 115.5, 115.2 (2C), 65.4, 52.9, 52.8, 46.3, 39.0, 36.1, 21.1. **HRMS** (ESI) m/z for C₁₉H₂₂O₅Na [M+Na]⁺ calcd 363.1567, found 363.1558.

General procedure for the oxidative carbocyclization/arylation of other enallenes

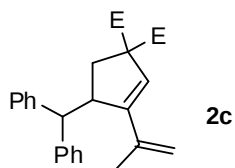


To a solution of enallene **1c** (50.5 mg, 0.2 mmol) in THF (2 mL) was added Pd(OAc)₂ (0.5 mg, 0.002 mmol, 1 mol%), BQ (23.8 mg, 0.22 mmol) and PhB(OH)₂ (33.4 mg, 0.26 mmol). The mixture was heated at 60 °C for 7 hours. The solvent was evaporated and the residue was purified by flash column chromatography (pentane/ethyl acetate v/v 40:1) giving the arylated product **2c** as a colorless oil (39 mg, 59 %).

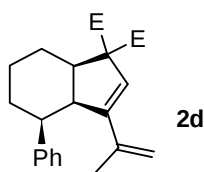
Spectral data of compounds 2b – 2e and 6c (NMR and HRMS)



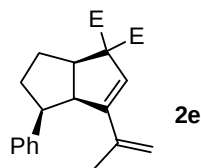
¹H NMR (CDCl₃, 400 MHz): δ 7.27 – 7.24 (m, 2H), 7.21 – 7.18 (m, 3H), 5.74 (s, 1H), 5.33 (br. s, 1H), 5.17 (m, 1H), 3.70 (s, 3H), 3.63 (s, 3H), 2.91 (d, J = 13.5 Hz, 1H), 2.87 (d, J = 13.5 Hz, 1H), 2.78 (d, J = 14.1 Hz, 1H), 2.11 (d, J = 14.1 Hz, 1H), 1.99 (m, 3H), 1.31 (s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.8, 171.5, 153.8, 138.6, 138.2, 130.6 (2C), 127.8 (2C), 126.1, 125.7, 115.5, 63.2, 52.8, 52.7, 51.0, 45.0, 44.1, 27.5, 23.7. **HRMS** (ESI) m/z for C₂₀H₂₄O₄Na [M+Na]⁺ calcd 351.1567, found 351.1568.



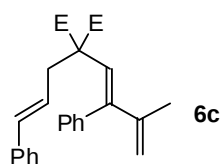
¹H NMR (CDCl₃, 400 MHz): δ 7.30 – 7.26 (m, 2H), 7.26 – 7.15 (m, 6H), 6.96 – 6.94 (m, 2H), 5.75 (d, J = 1.1 Hz, 1H), 5.02 (m, 1H), 4.98 (br. s, 1H), 4.58 (d, J = 4.4 Hz, 1H), 4.00 – 3.95 (m, 1H), 3.69 (s, 3H), 3.46 (s, 3H), 3.03 (dd, J = 14.5, 9.4 Hz, 1H), 2.56 (dd, J = 14.5, 3.4 Hz, 1H), 1.94 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.6, 171.0, 150.4, 143.8, 141.6, 138.2, 129.8 (2C), 128.8 (2C), 128.2 (2C), 127.7 (2C), 126.2, 126.1, 125.7, 115.4, 65.8, 52.9, 52.6, 52.3, 47.9, 34.2, 21.5. **HRMS** (ESI) m/z for C₂₅H₂₆O₄Na [M+Na]⁺ calcd 413.1723, found 413.1722.



¹H NMR (CDCl₃, 400 MHz): δ 7.22 – 7.21 (m, 4H), 7.16 – 7.13 (m, 1H), 5.51 (m, 1H), 4.69 (m, 1H), 4.53 (m, 1H), 3.73 (s, 3H), 3.67 (s, 3H), 3.20 (m, 1H), 2.98 (ddd, J = 12.4, 4.4, 3.4 Hz, 1H), 1.87 – 1.80 (m, 2H), 1.73 – 1.69 (m, 1H), 1.48 – 1.38 (m, 4H), 0.77 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 170.4, 169.6, 153.5, 144.3, 143.4, 128.8 (2C), 127.6 (2C), 125.8, 125.6, 113.4, 67.3, 52.5, 52.1, 50.3, 46.3, 44.2, 25.9, 25.1, 23.8, 20.8. **HRMS** (ESI) m/z for C₂₂H₂₆O₄Na [M+Na]⁺ calcd 377.1723, found 377.1720.



¹H NMR (CDCl₃, 400 MHz): δ 7.16 – 7.07 (m, 5H), 5.68 (d, J = 1.0 Hz, 1H), 4.49 (br. s, 1H), 4.46 (m, 1H), 3.95 – 3.90 (m, 1H), 3.87 – 3.83 (m, 1H), 3.80 (s, 3H), 3.73 (s, 3H), 3.32 (m, 1H), 1.95 – 1.67 (m, 4H), 1.53 (br. s, 3H). **¹³C NMR** (CDCl₃, 100 MHz): δ 171.2, 171.2, 149.7, 143.9, 138.1, 128.7 (2C), 127.5 (2C), 125.6, 124.9, 115.6, 69.8, 54.5, 52.9, 52.2, 49.2, 47.6, 34.7, 29.0, 20.4. **HRMS** (ESI) m/z for C₂₁H₂₄O₄Na [M+Na]⁺ calcd 363.1567, found 363.1563.

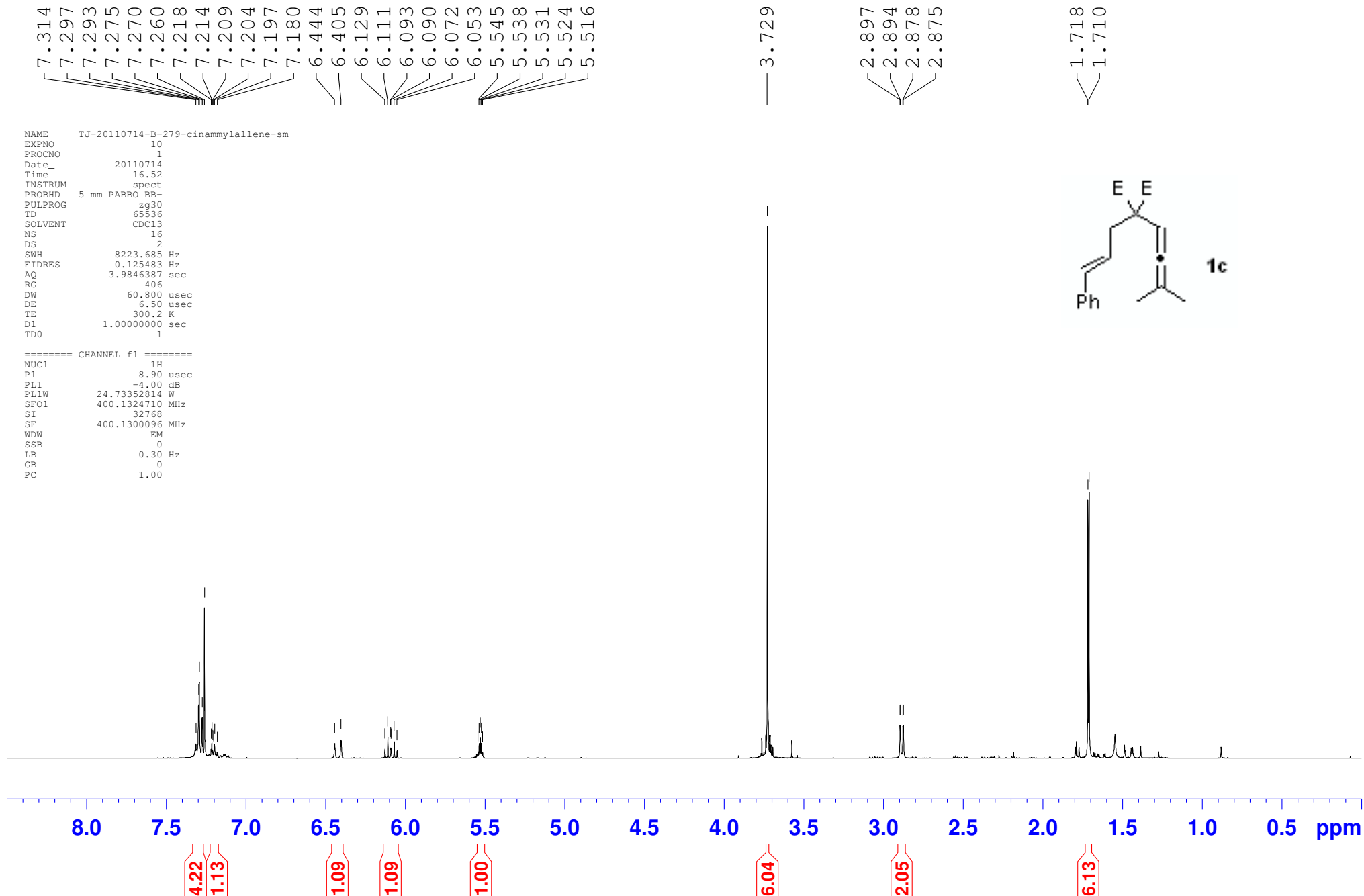


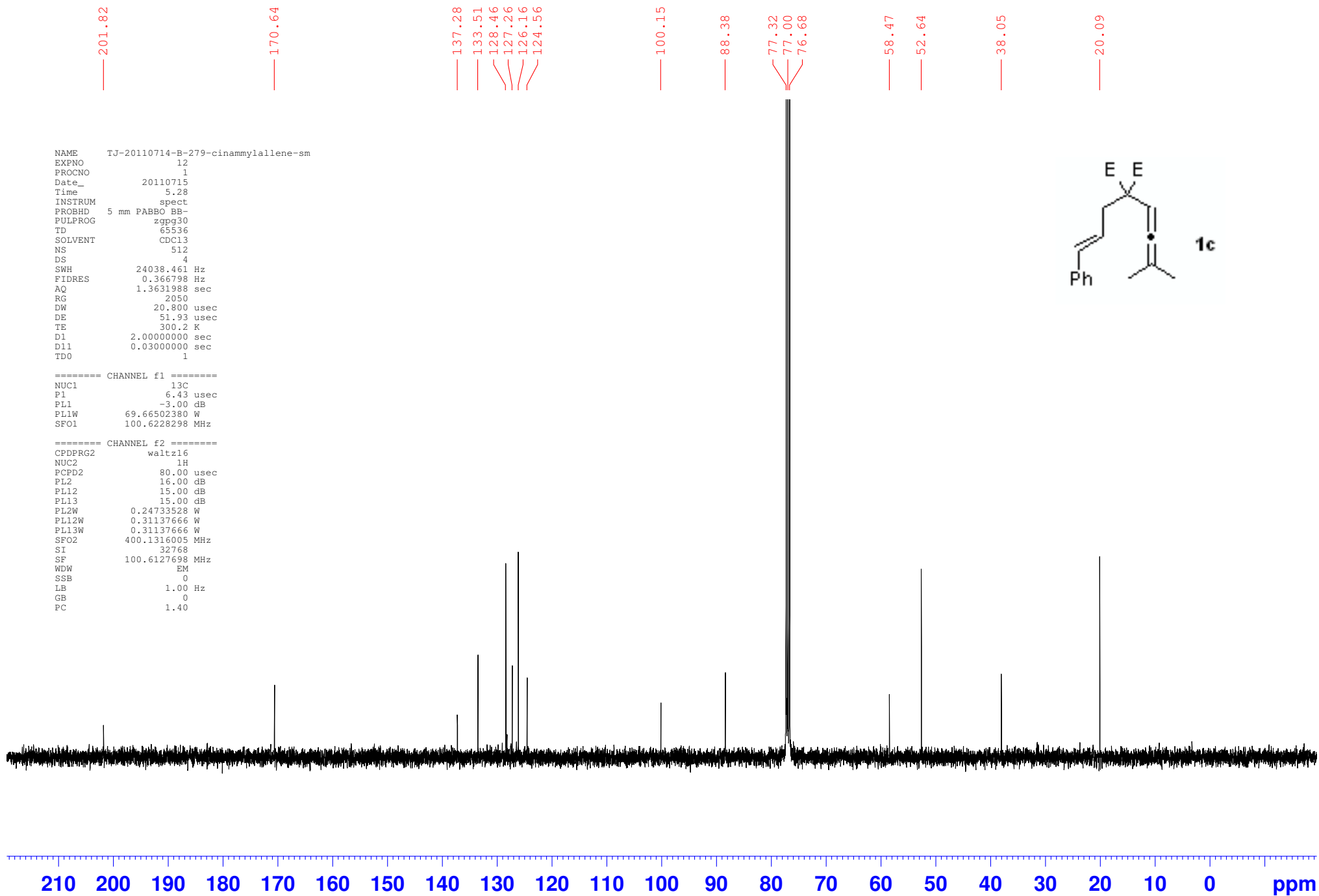
6c

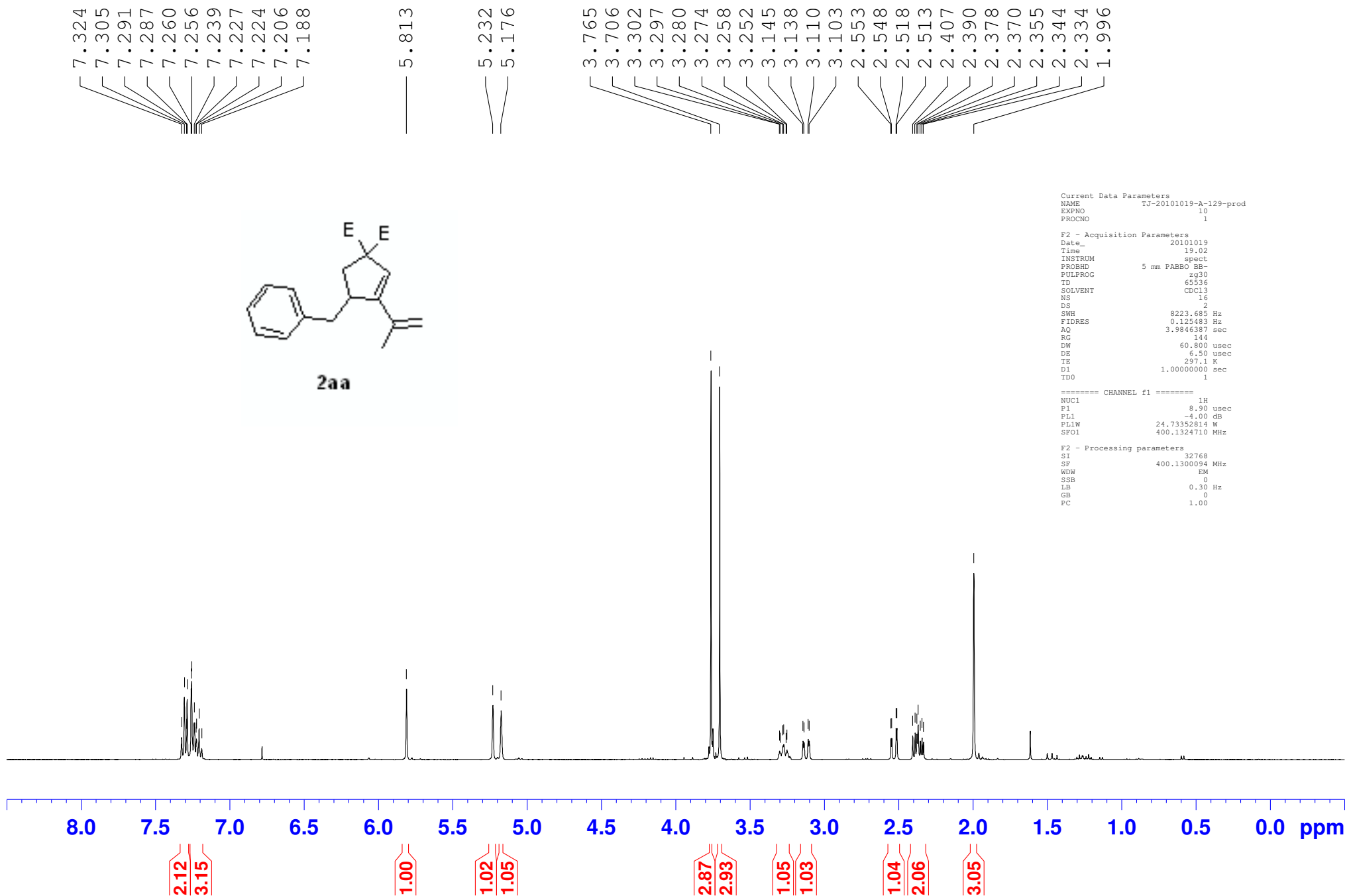
^1H NMR (CDCl_3 , 400 MHz): δ 7.33 – 7.29 (m, 7H), 7.22 (m, 1H), 7.04 (m, 2H), 6.48 (s, 1H), 6.39 (d, J = 15.7 Hz, 1H), 6.09 (dt, J = 15.7, 7.5 Hz, 1H), 5.02 (br. s, 1H), 4.41 (m, 1H), 3.54 (s, 6H), 2.80 (dd, J = 7.5, 1.2 Hz, 2H), 2.06 (m, 3H).

^{13}C NMR (CDCl_3 , 100 MHz): δ 170.4 (2C), 145.4, 144.5, 137.4, 137.2, 133.6, 129.9 (2C), 128.5, (2C), 127.6 (2C), 127.4, 127.4, 126.2 (2C), 125.2, 124.4,

117.7, 59.3, 52.5 (2C), 40.8, 20.9. **HRMS** (ESI) m/z for $\text{C}_{25}\text{H}_{26}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$ calcd 413.1723, found 413.1721.







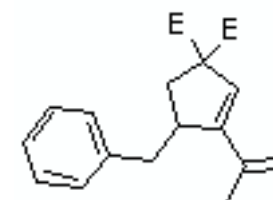
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EXPNO 11
PROCNO 1

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FIDRES 0.366798 Hz
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D11 0.03000000 sec
TD0 1

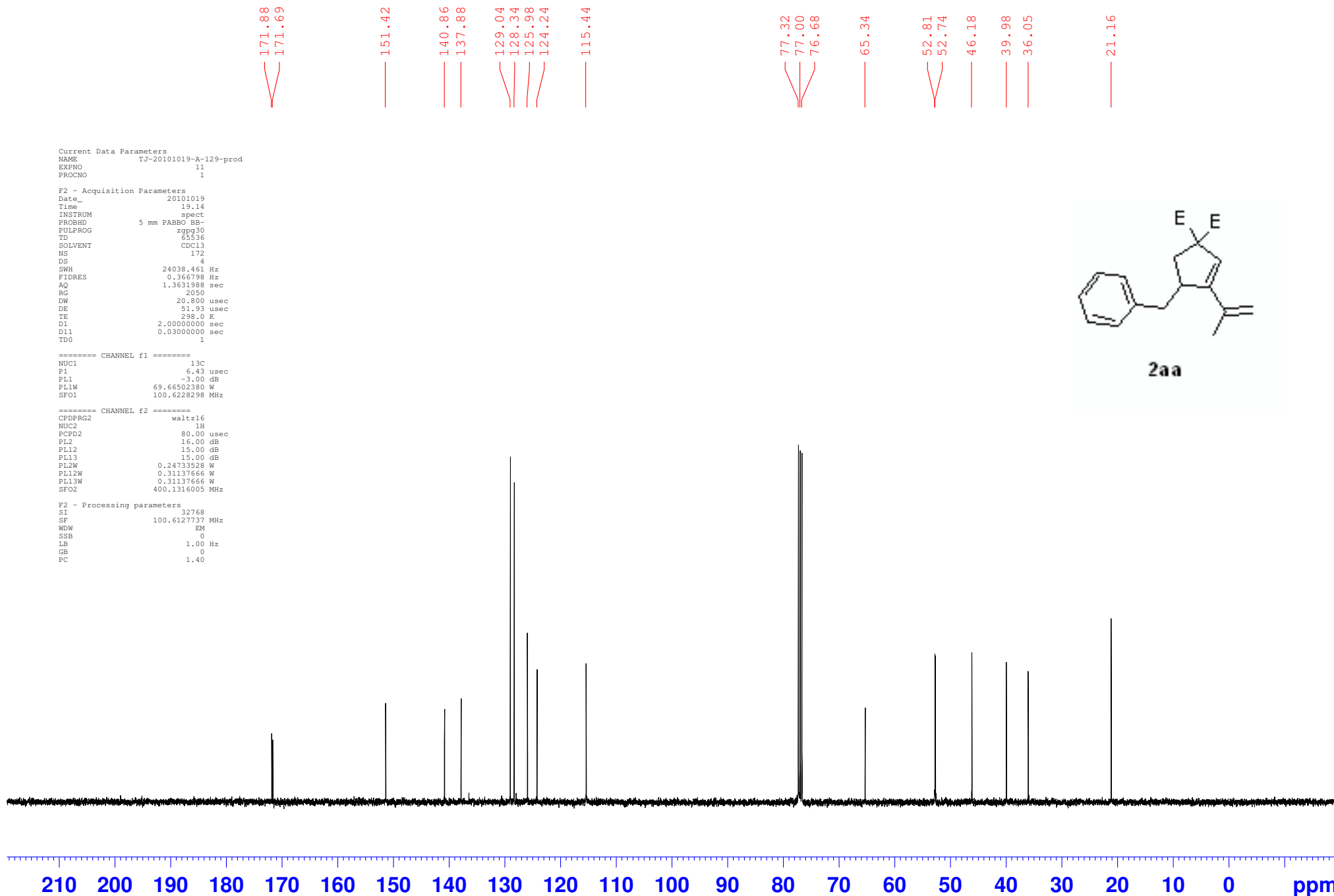
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SFO1 100.6228298 MHz

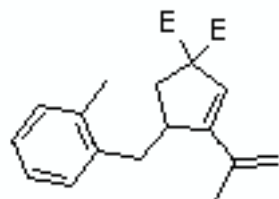
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PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz

F2 - Processing parameters
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SF 100.6127737 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

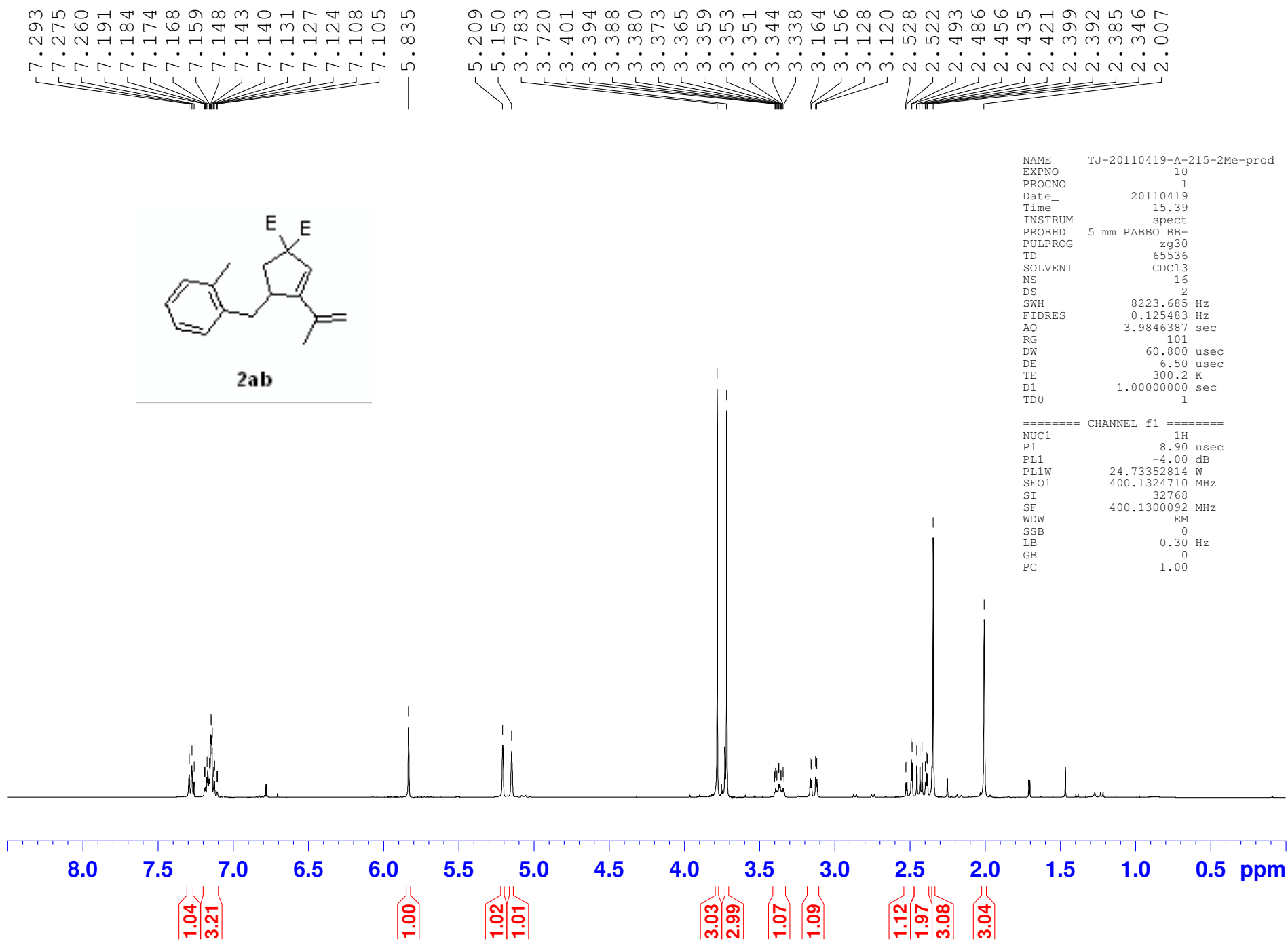


2aa





2ab



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PULPROG   zgpg30
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DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG          2050
DW          20.800 usec
DE          51.93 usec
TE          300.2 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1

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===== CHANNEL f1 =====
NUC1       13C
P1          6.43 usec
PL1        -3.00 dB
PL1W       69.66502380 W
SFO1       100.6228298 MHz

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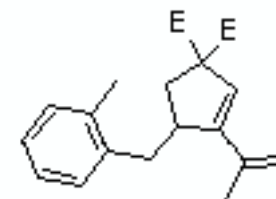
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PCPD2       80.00 usec
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PL12        15.00 dB
PL13        15.00 dB
PL2W        0.24733528 W
PL12W       0.31137666 W
PL13W       0.31137666 W
SFO2       400.1316005 MHz
SI          32768
SF         100.6127758 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

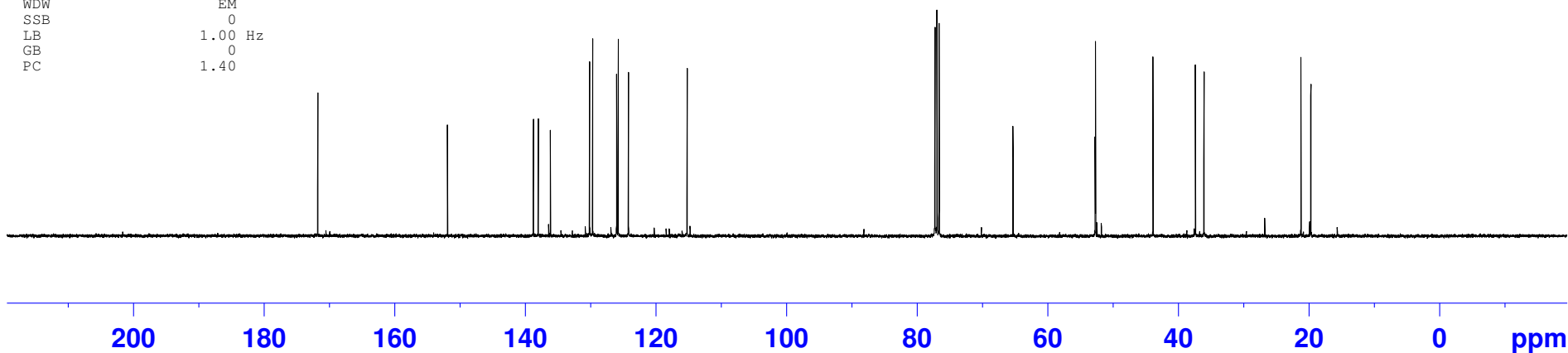
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2ab



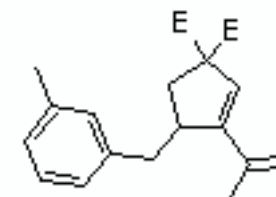
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$$\begin{array}{r} 5.243 \\ 5.182 \end{array}$$

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6	3.271	3.254	3.249
7	3.254	3.249	3.243
8	3.249	3.243	3.117
9	3.243	3.117	3.110
10	3.117	3.110	3.083
11	3.110	3.083	3.076
12	3.083	3.076	2.568
13	3.076	2.568	2.563
14	2.568	2.563	2.533
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16	2.533	2.528	2.409
17	2.528	2.409	2.387
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19	2.387	2.373	2.357
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21	2.357	2.345	2.339
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24	2.311	2.002	
25	2.002		

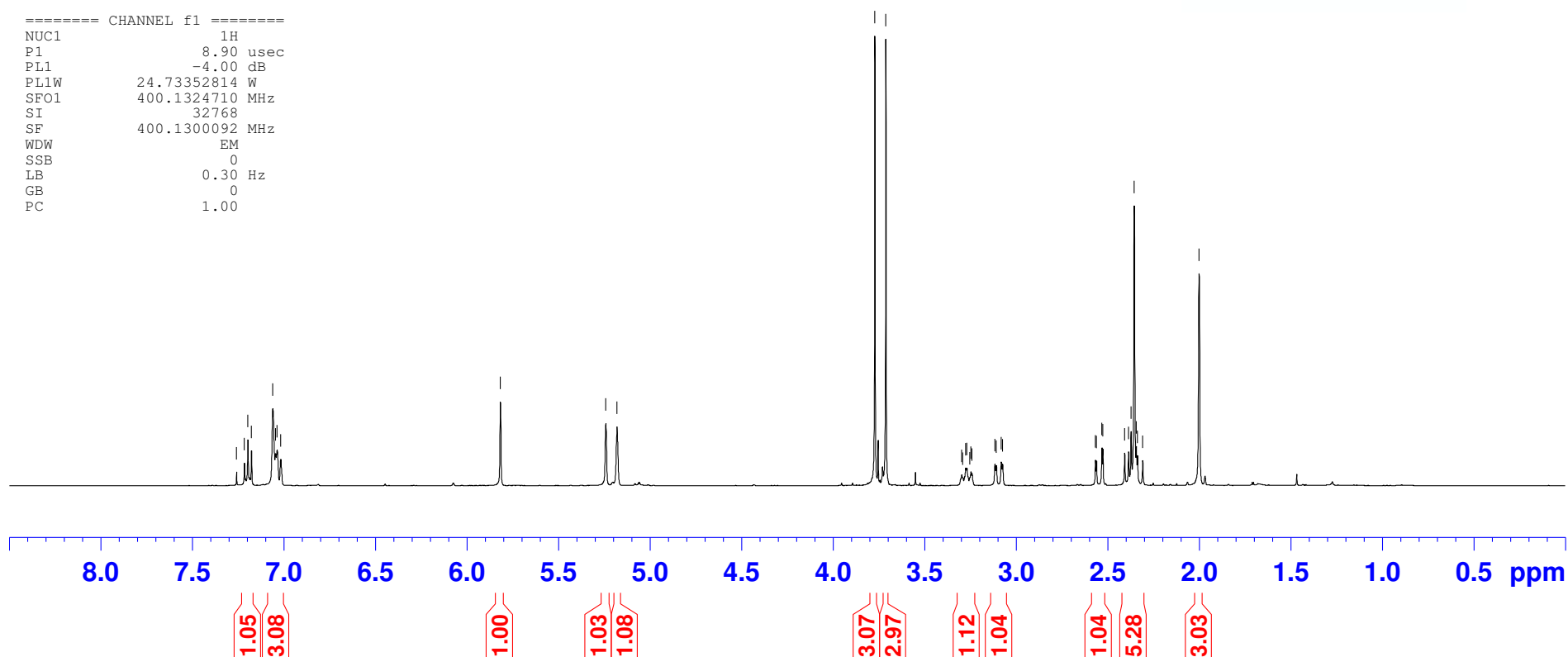
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SI                  32768
SF                  400.1300092 MHz
WDW                  EM
SSB                  0
LB                  0.30 Hz
GB                  0
PC                  1.00

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2ac



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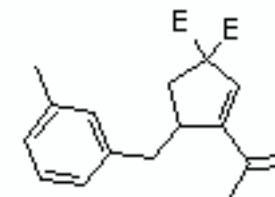
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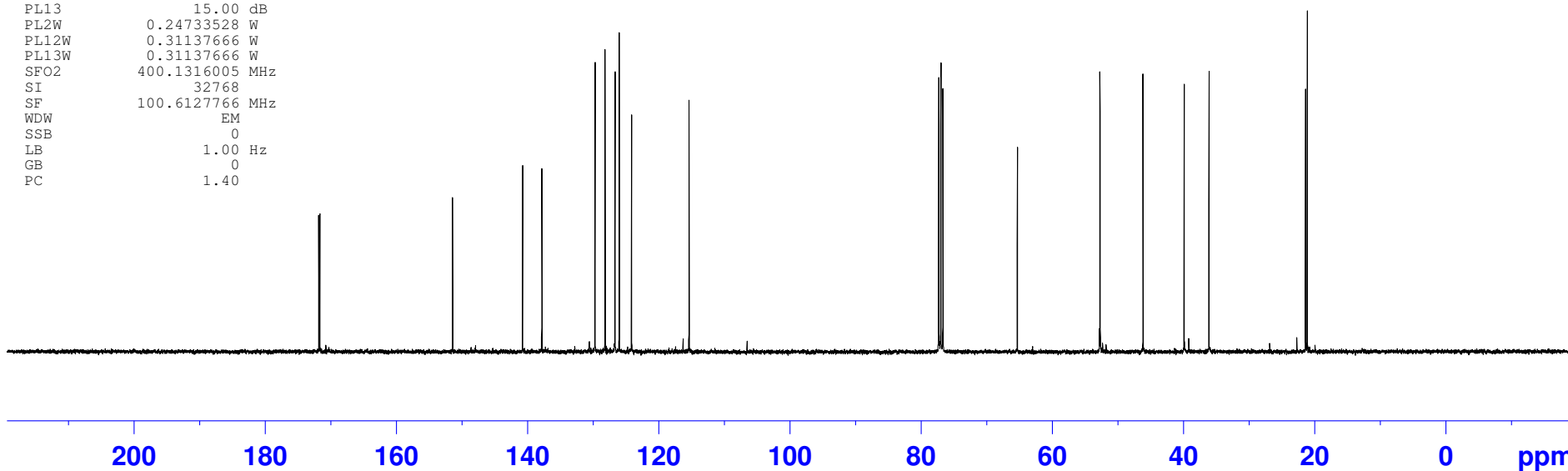
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 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
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 RG 2050
 DW 20.800 usec
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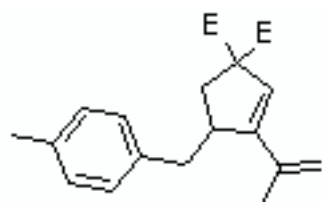
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 SFO1 100.6228298 MHz

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 NUC2 1H
 PCPD2 80.00 usec
 PL2 16.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 0.24733528 W
 PL12W 0.31137666 W
 PL13W 0.31137666 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127766 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



2ac





2ad

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7.107

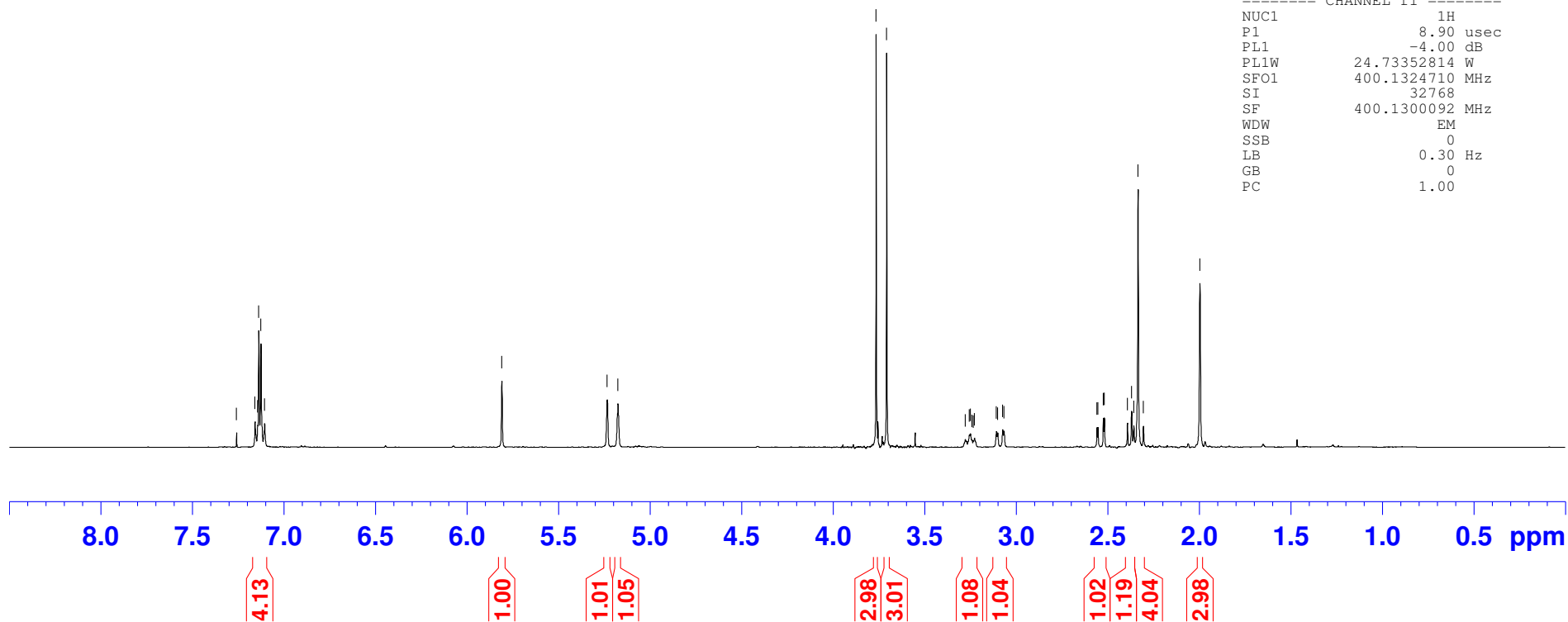
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2.307
1.998

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PROCNO 1
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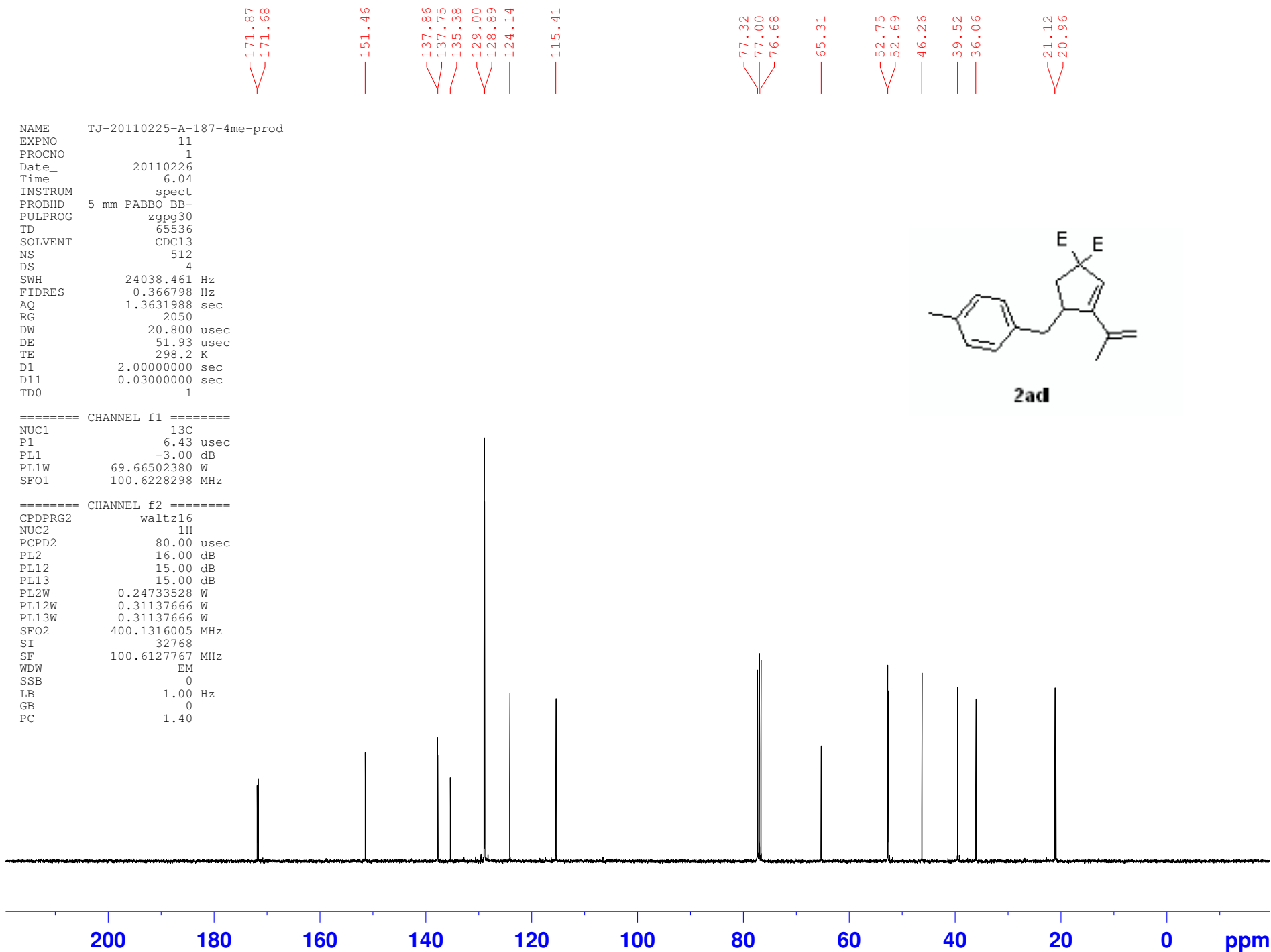
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NAME TJ-20110225-A-187-4me-prod
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 PROCNO 1
 Date_ 20110226
 Time 6.04
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 512
 DS 4
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 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 51.93 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.43 usec
 PL1 -3.00 dB
 PL1W 69.66502380 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
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 NUC2 1H
 PCPD2 80.00 usec
 PL2 16.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 0.24733528 W
 PL12W 0.31137666 W
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 SI 32768
 SF 100.6127767 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

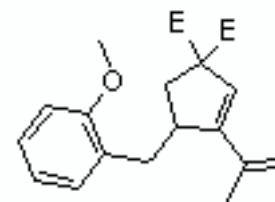


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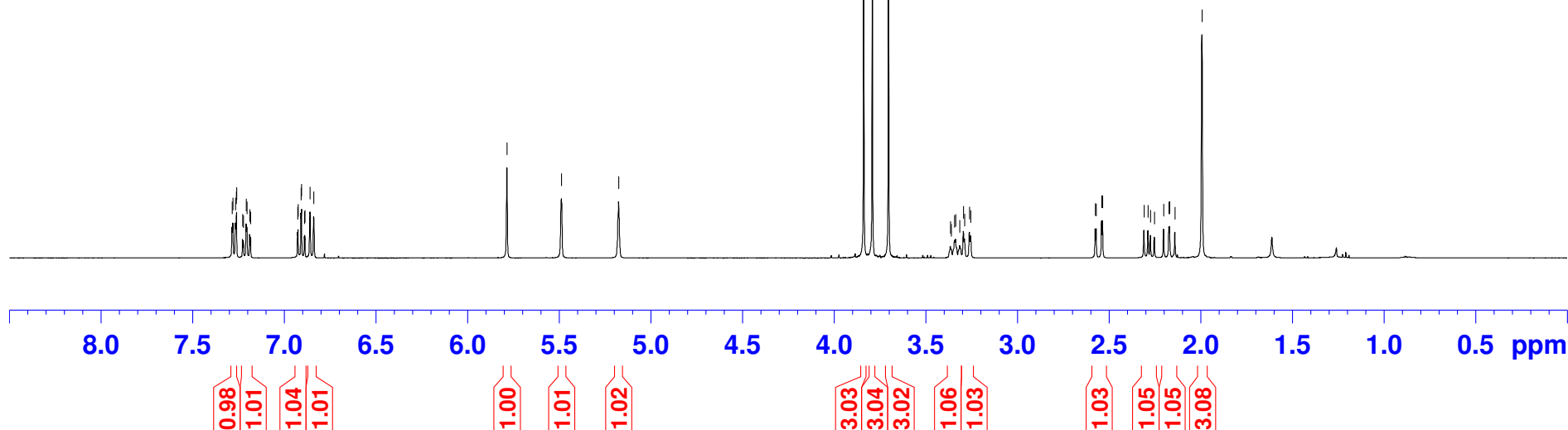
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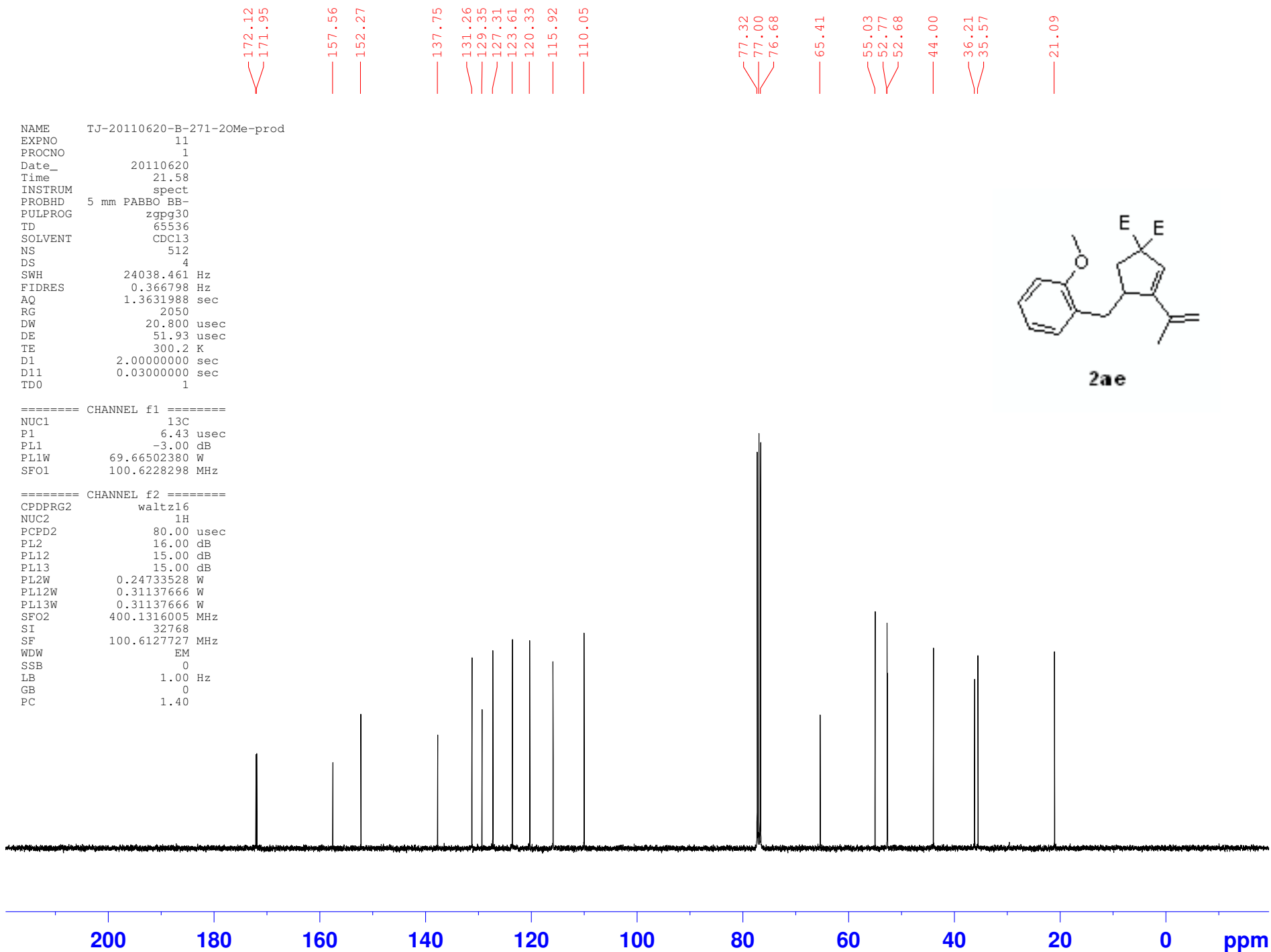
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AQ 3.9846387 sec
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DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

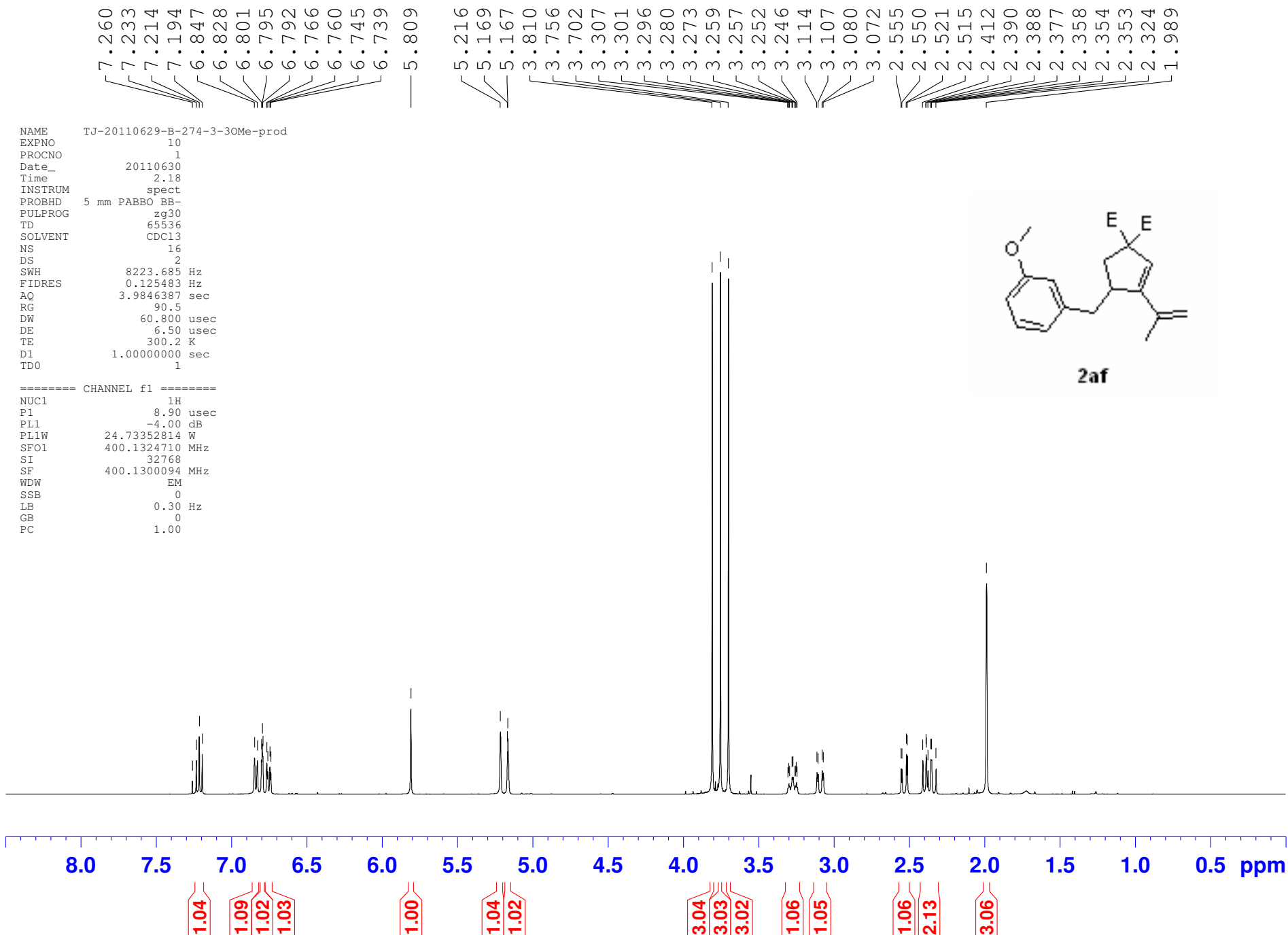
===== CHANNEL f1 =====
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



2ae







NAME TJ-20110629-B-274-3-3OMe-prod
 EXPNO 11
 PROCNO 1
 Date_ 20110630
 Time 2.50
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 51.93 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.43 usec
 PL1 -3.00 dB
 PL1W 69.66502380 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 16.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 0.24733528 W
 PL12W 0.31137666 W
 PL13W 0.31137666 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127774 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

171.81
171.61

159.56

151.33

142.40

137.83

129.24

124.25

121.42

115.38

114.88

111.12

77.32
77.00
76.68

65.32

55.05

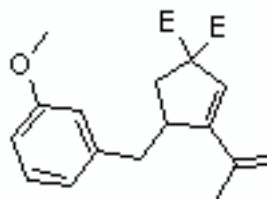
52.73

52.68

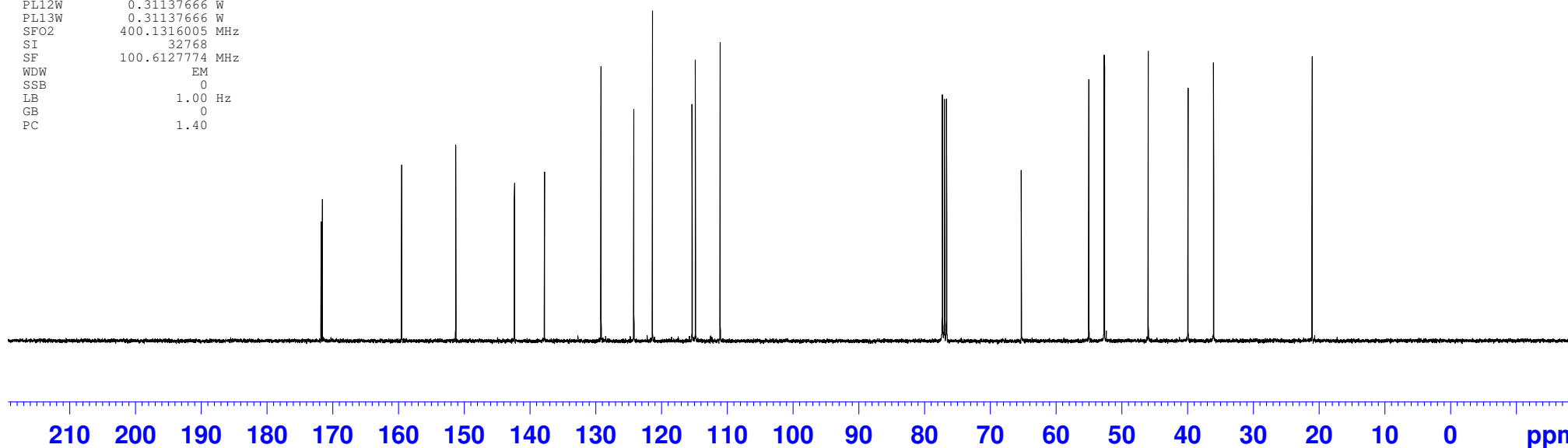
46.02

39.97

36.07



2af

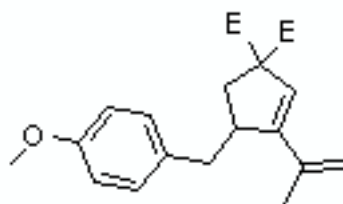


7.260
7.168
7.164
7.147
6.854
6.849
6.838
6.833

5.796

5.213
5.162

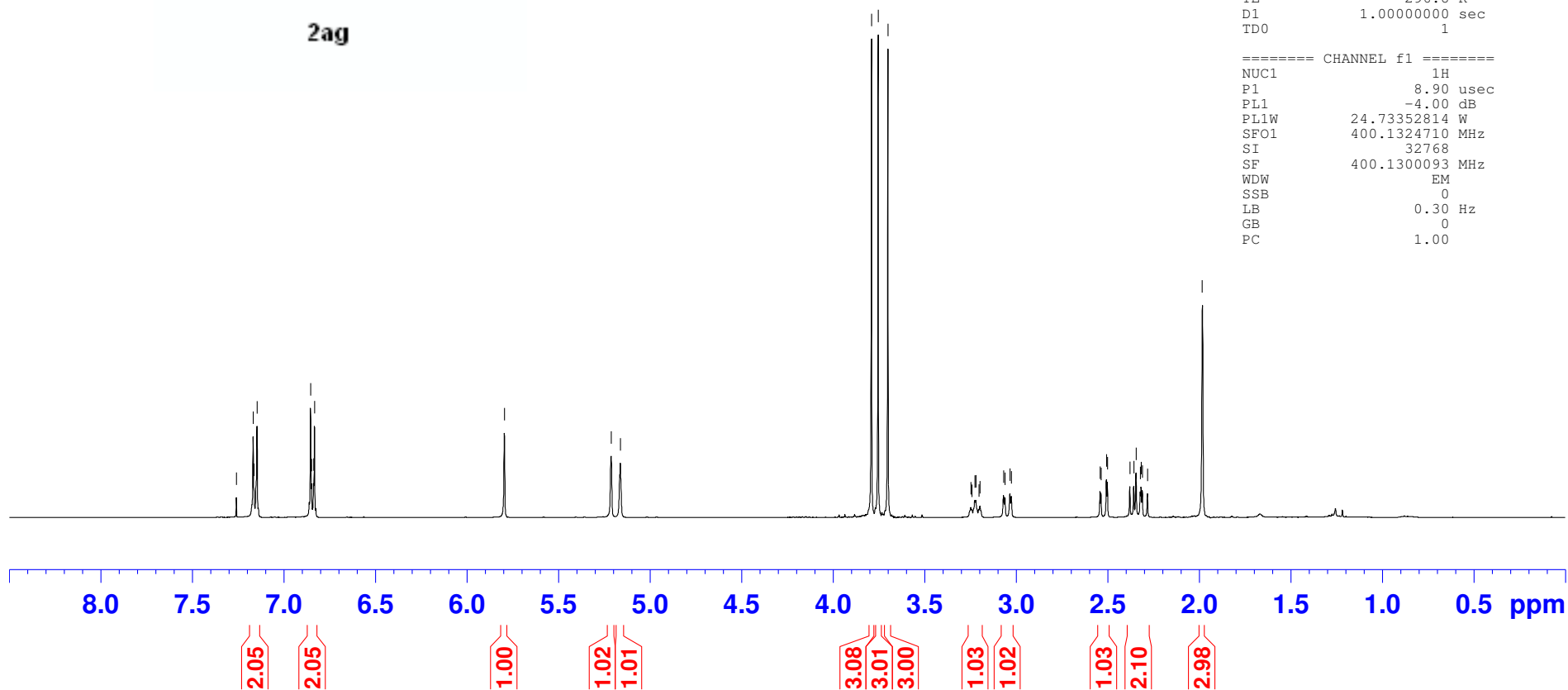
3.791
3.754
3.701
3.248
3.242
3.226
3.220
3.204
3.198
3.069
3.062
3.035
3.027
2.542
2.537
2.507
2.502
2.379
2.358
2.345
2.322
2.318
2.311
2.283
1.985

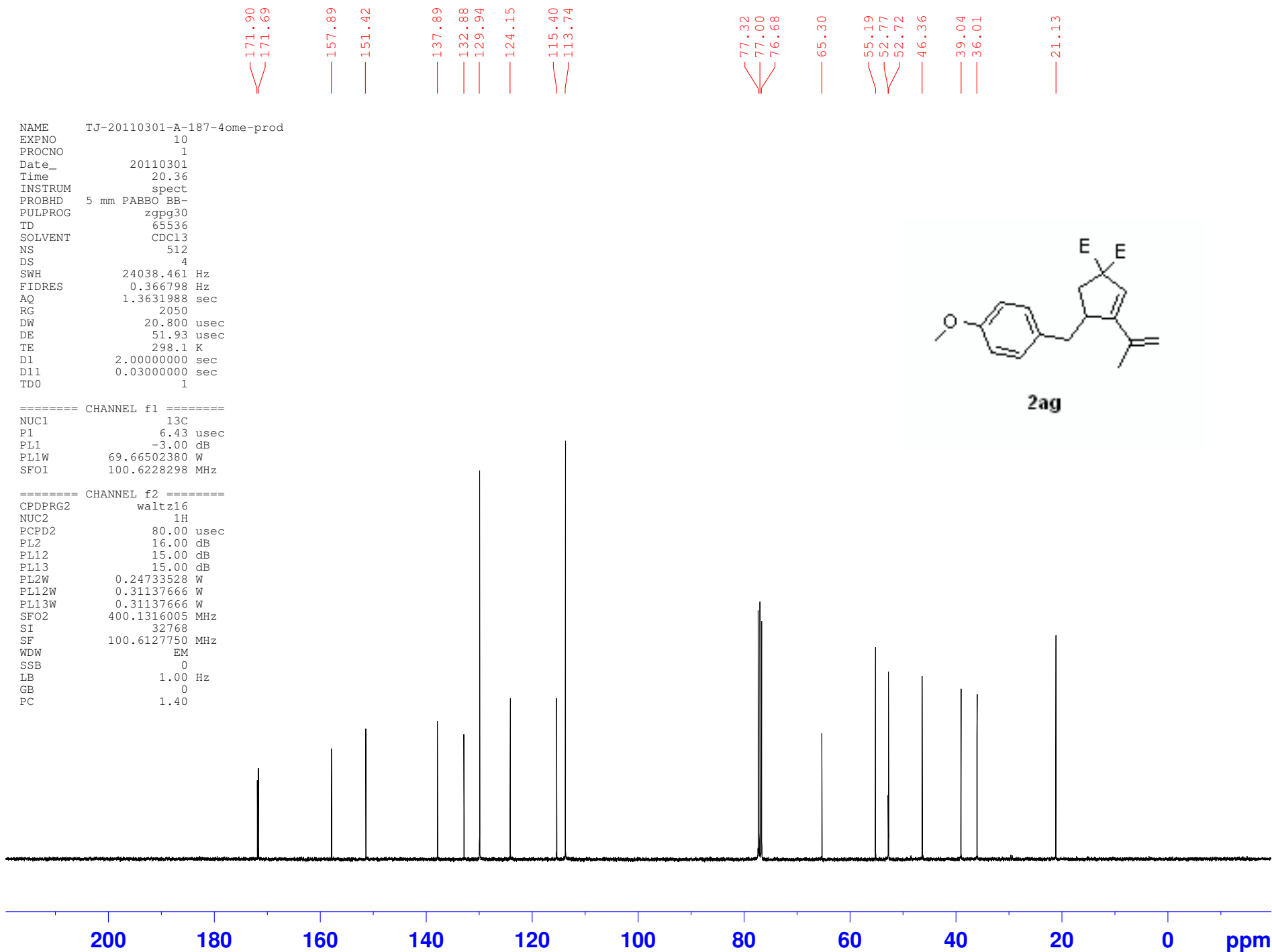


2ag

NAME TJ-20110228-A-187-4ome-prod
EXPNO 10
PROCNO 1
Date_ 20110228
Time 16.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1

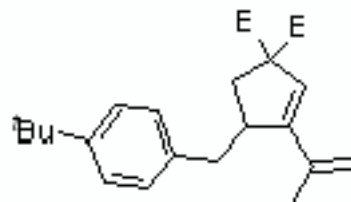
===== CHANNEL f1 =====
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



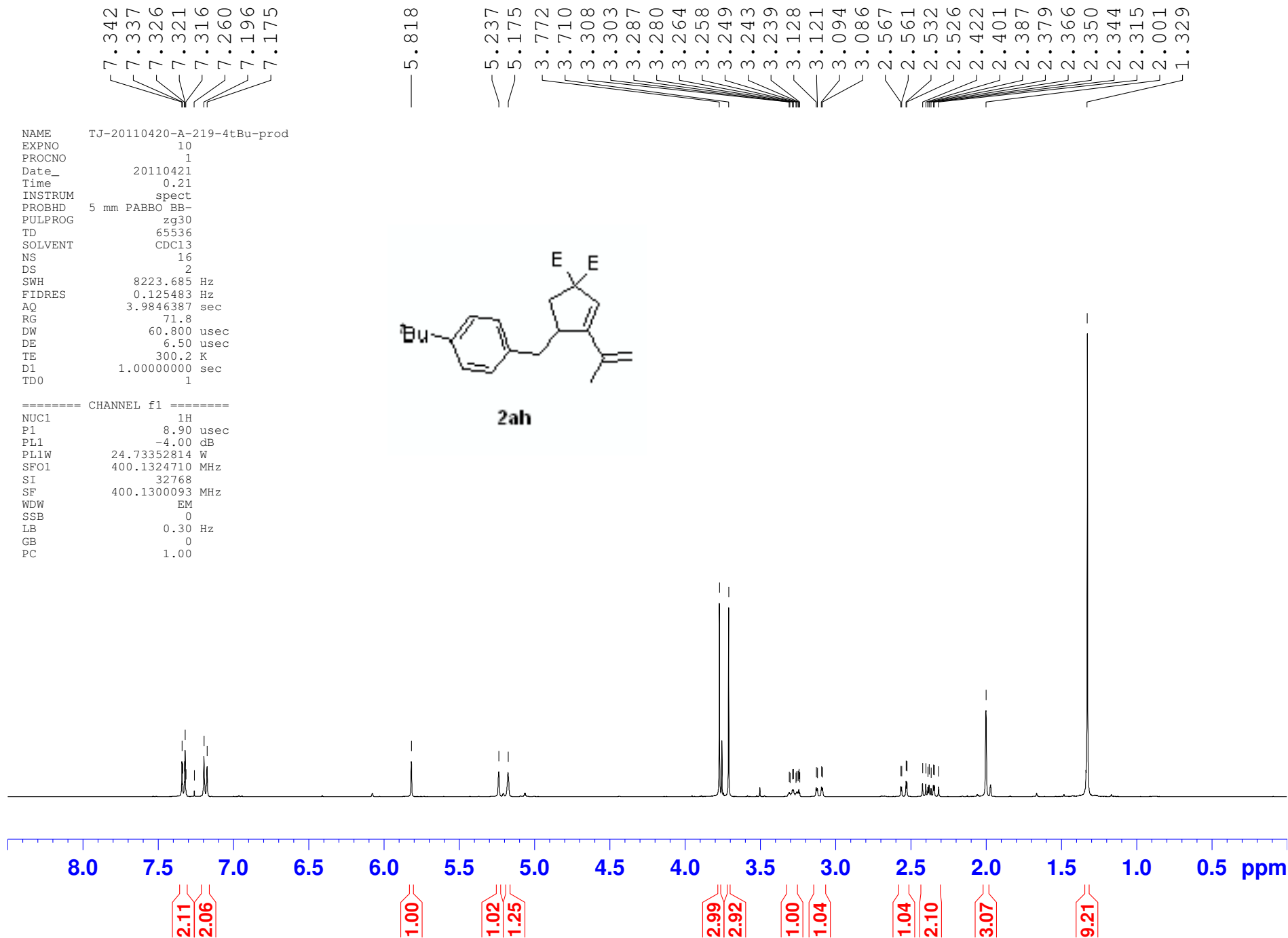


NAME TJ-20110420-A-219-4tBu-prod
 EXPNO 10
 PROCNO 1
 Date_ 20110421
 Time 0.21
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 71.8
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.90 usec
 PL1 -4.00 dB
 PL1W 24.73352814 W
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300093 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



2ah



171.85
171.66

151.51
148.68

137.85
137.72

128.60
125.17
124.10

115.38

77.30
76.99
76.67

65.31

52.72
52.66

46.07

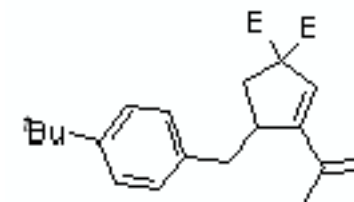
39.45
36.20
34.28
31.34

21.11

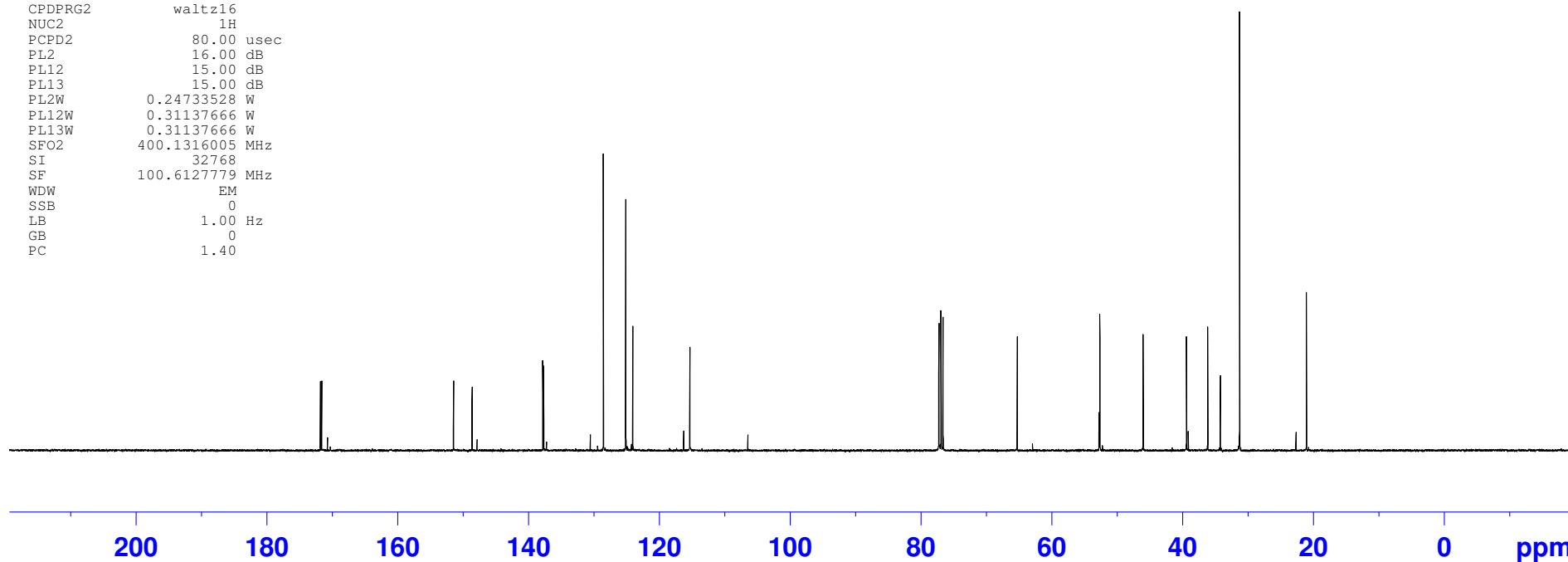
NAME TJ-20110420-A-219-4tBu-prod
EXPNO 11
PROCNO 1
Date_ 20110421
Time_ 0.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

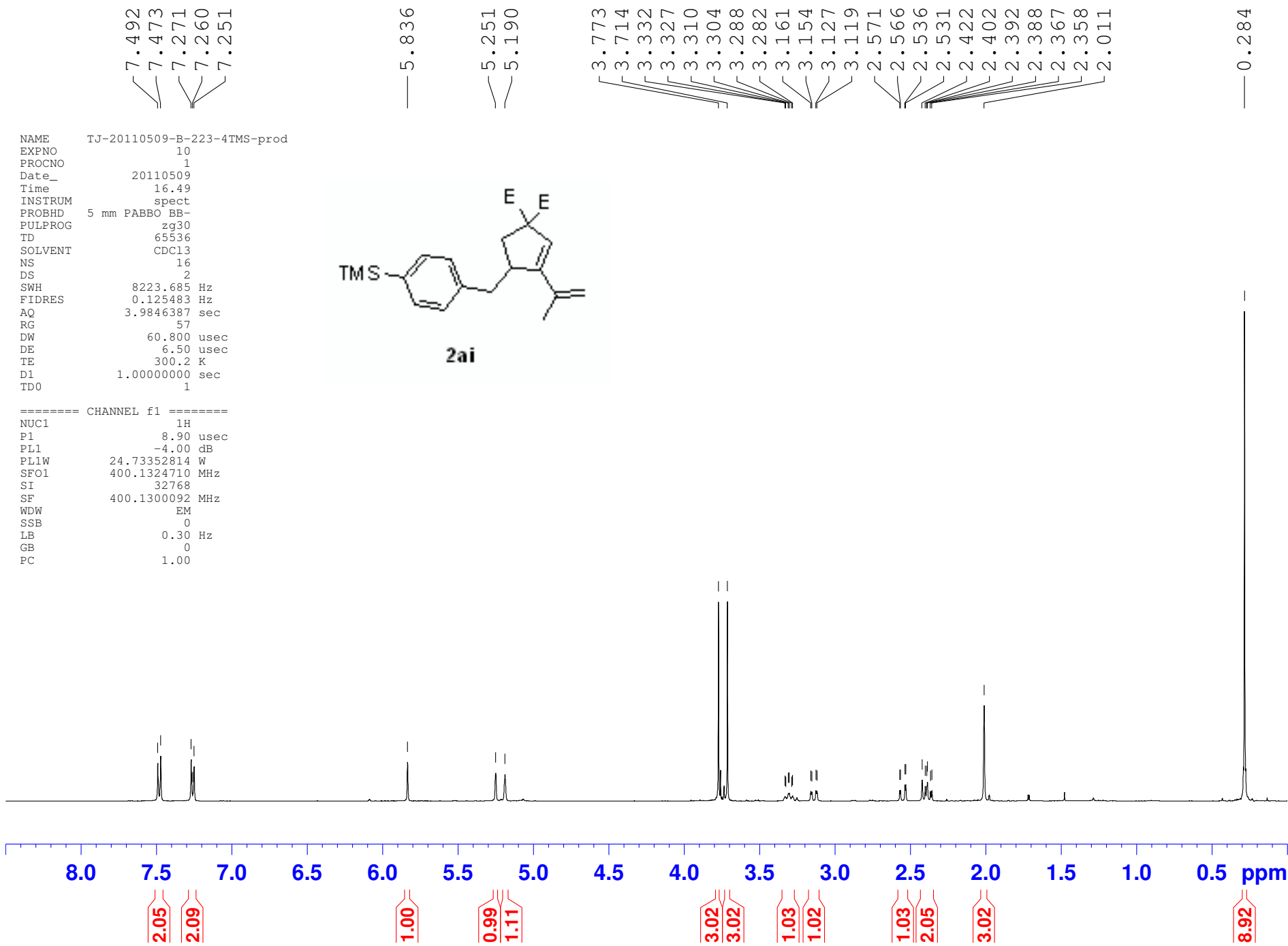
===== CHANNEL f1 =====
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz

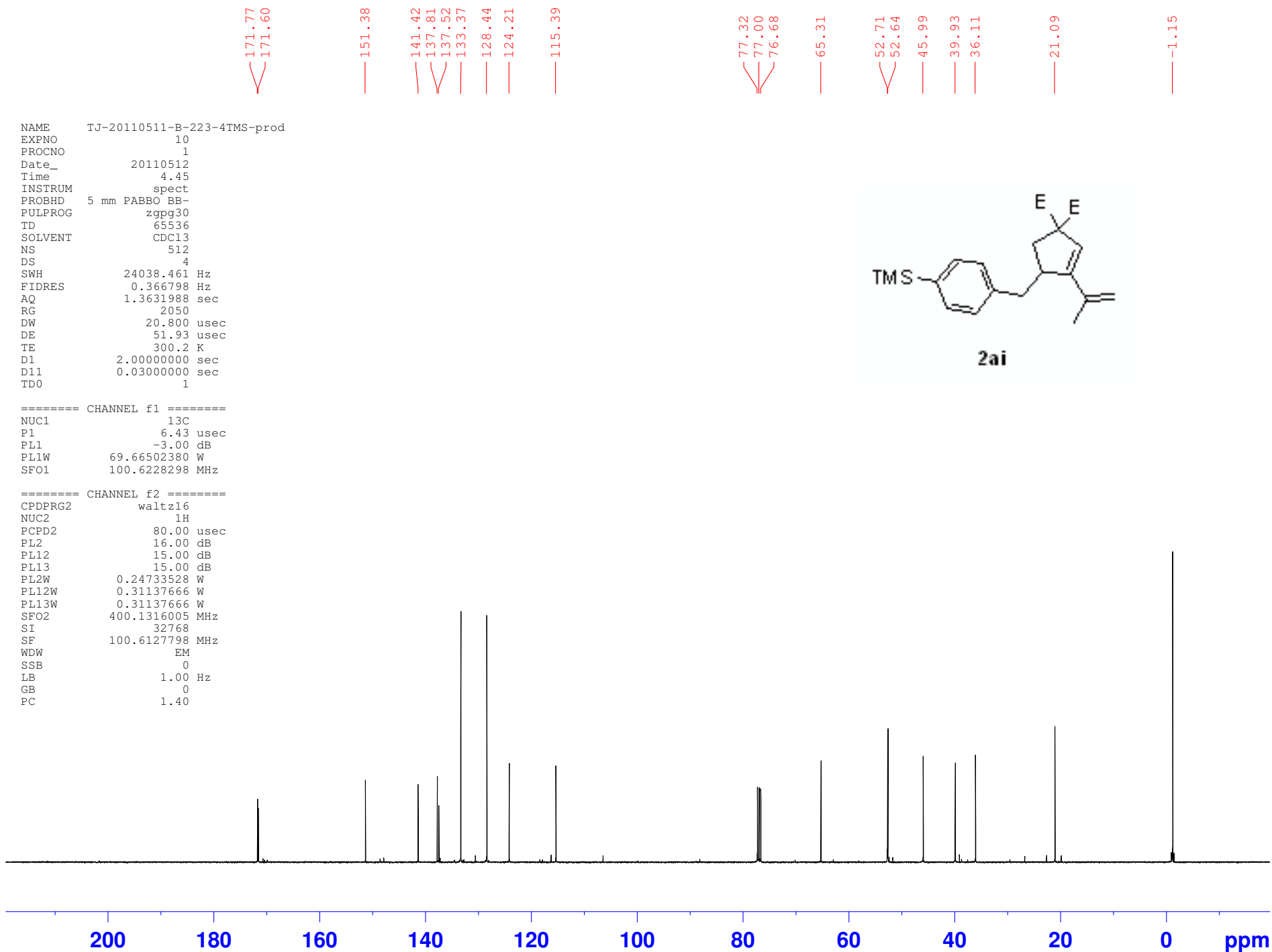
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127779 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

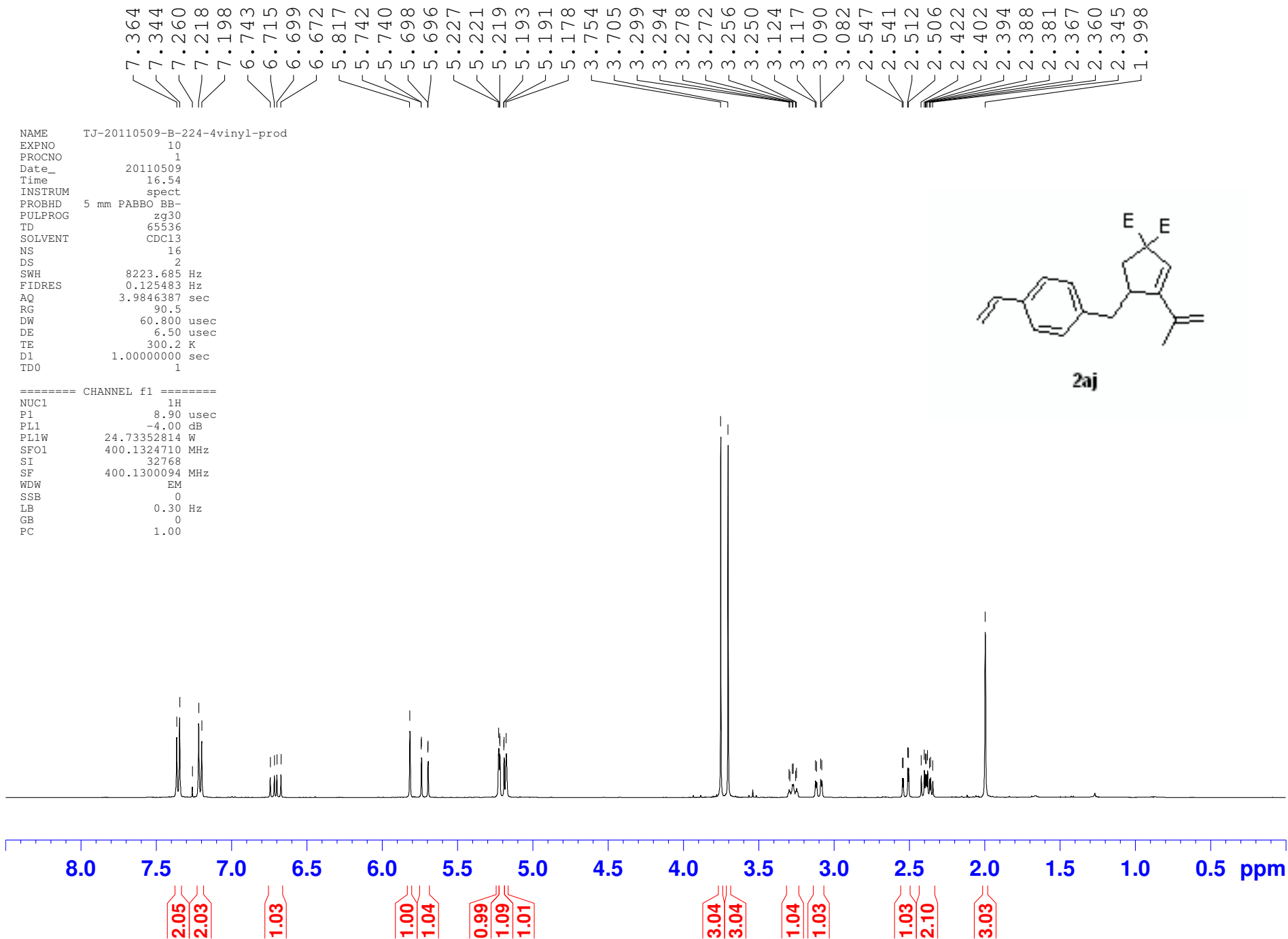


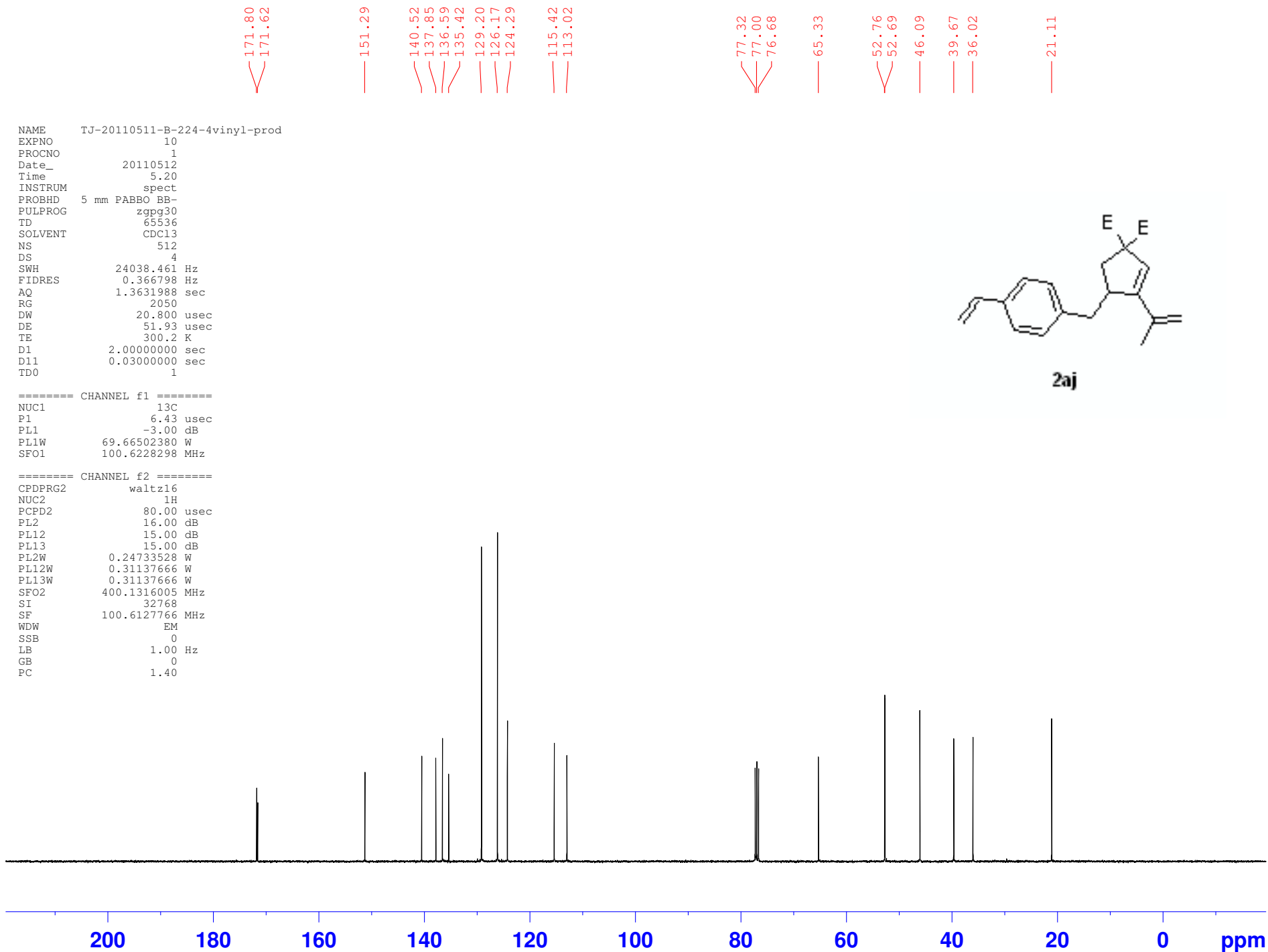
2ah





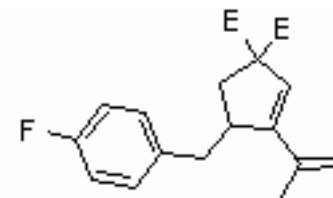




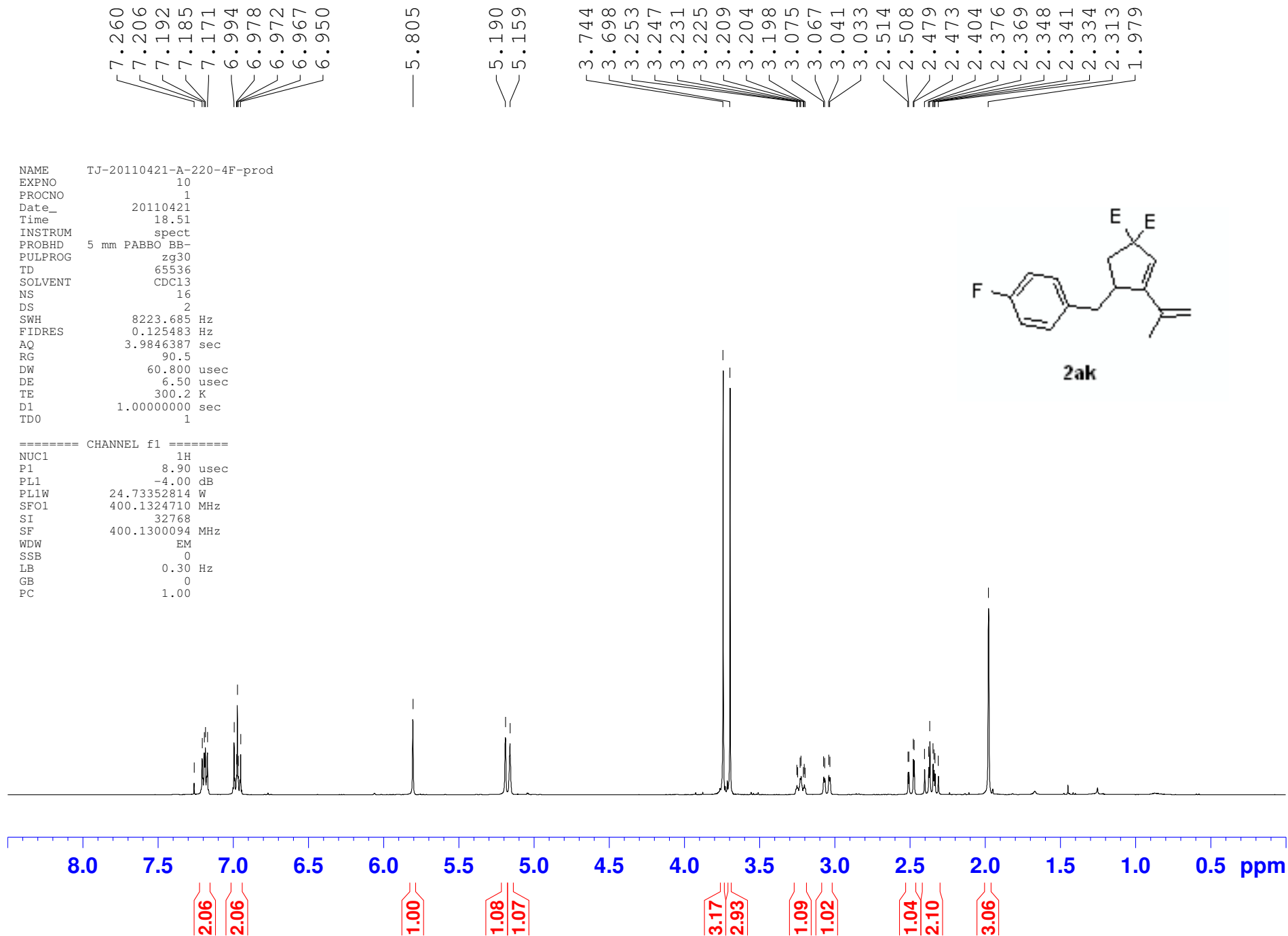


NAME TJ-20110421-A-220-4F-prod
 EXPNO 10
 PROCNO 1
 Date_ 20110421
 Time 18.51
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 90.5
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.90 usec
 PL1 -4.00 dB
 PL1W 24.73352814 W
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



2ak

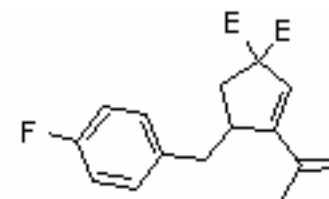




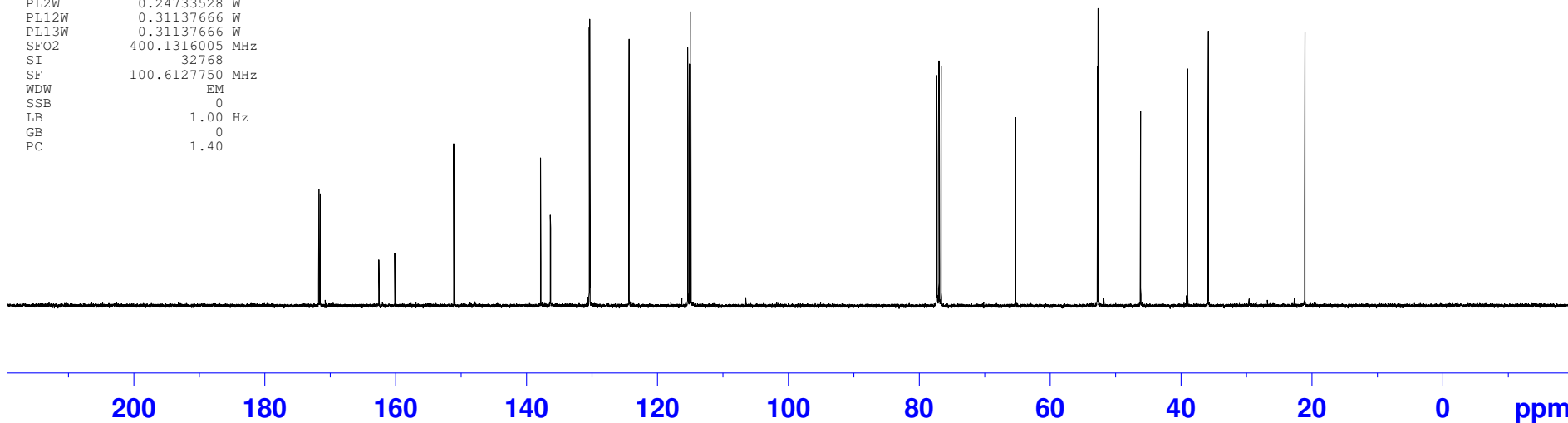
NAME TJ-20110421-A-220-4F-prod
EXPNO 11
PROCNO 1
Date_ 20110422
Time 7.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

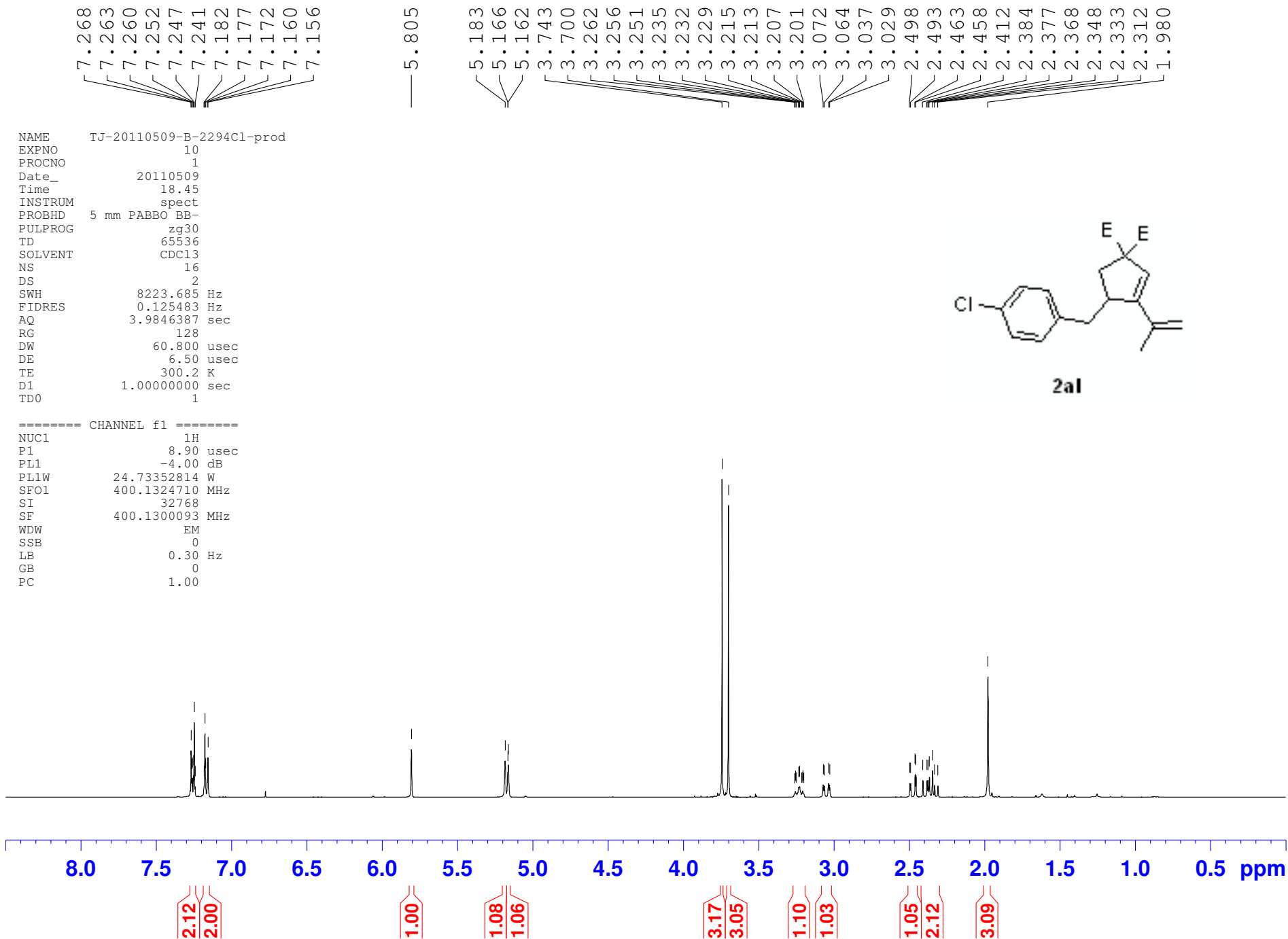
===== CHANNEL f1 =====
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz

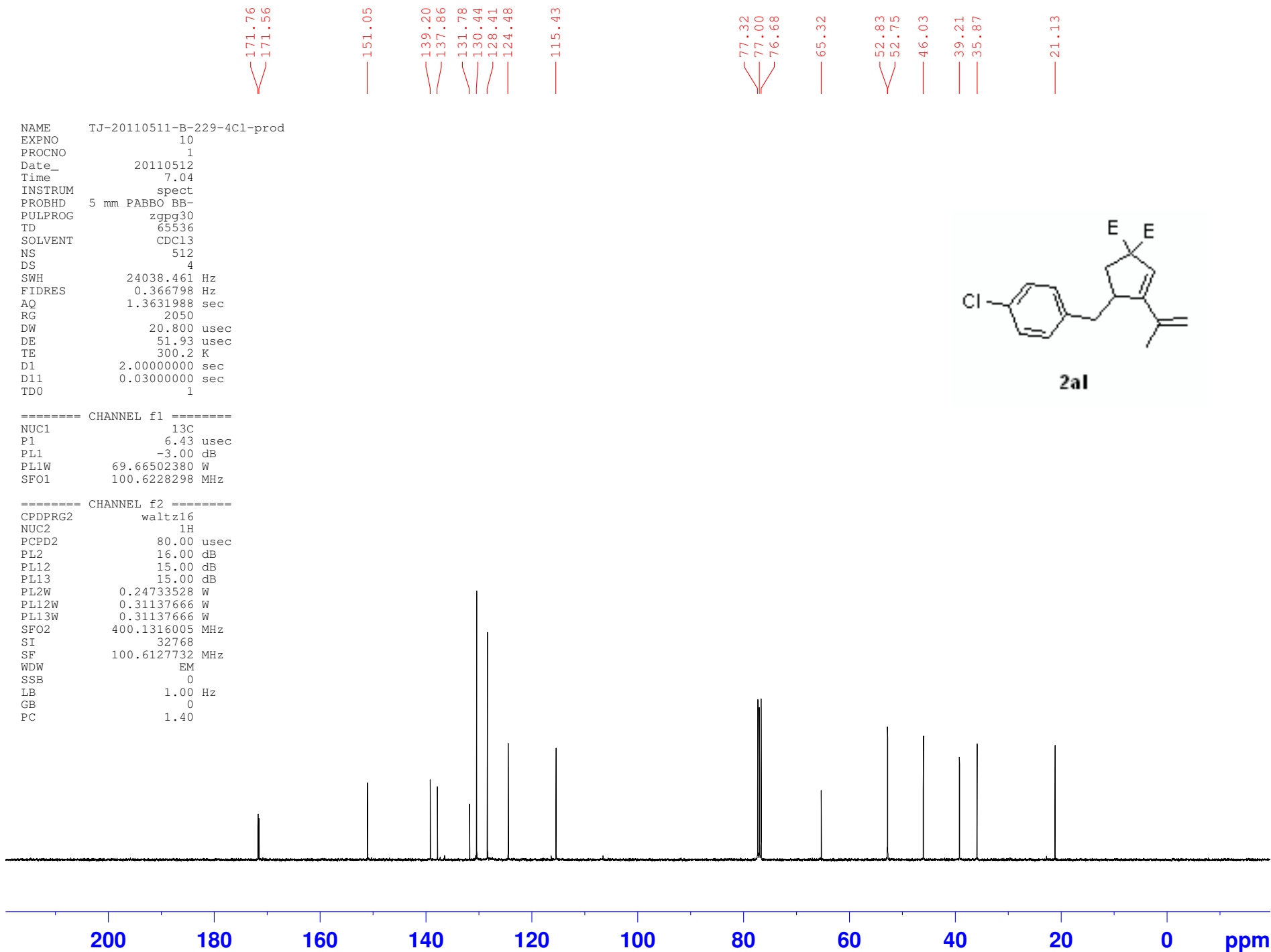
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127750 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

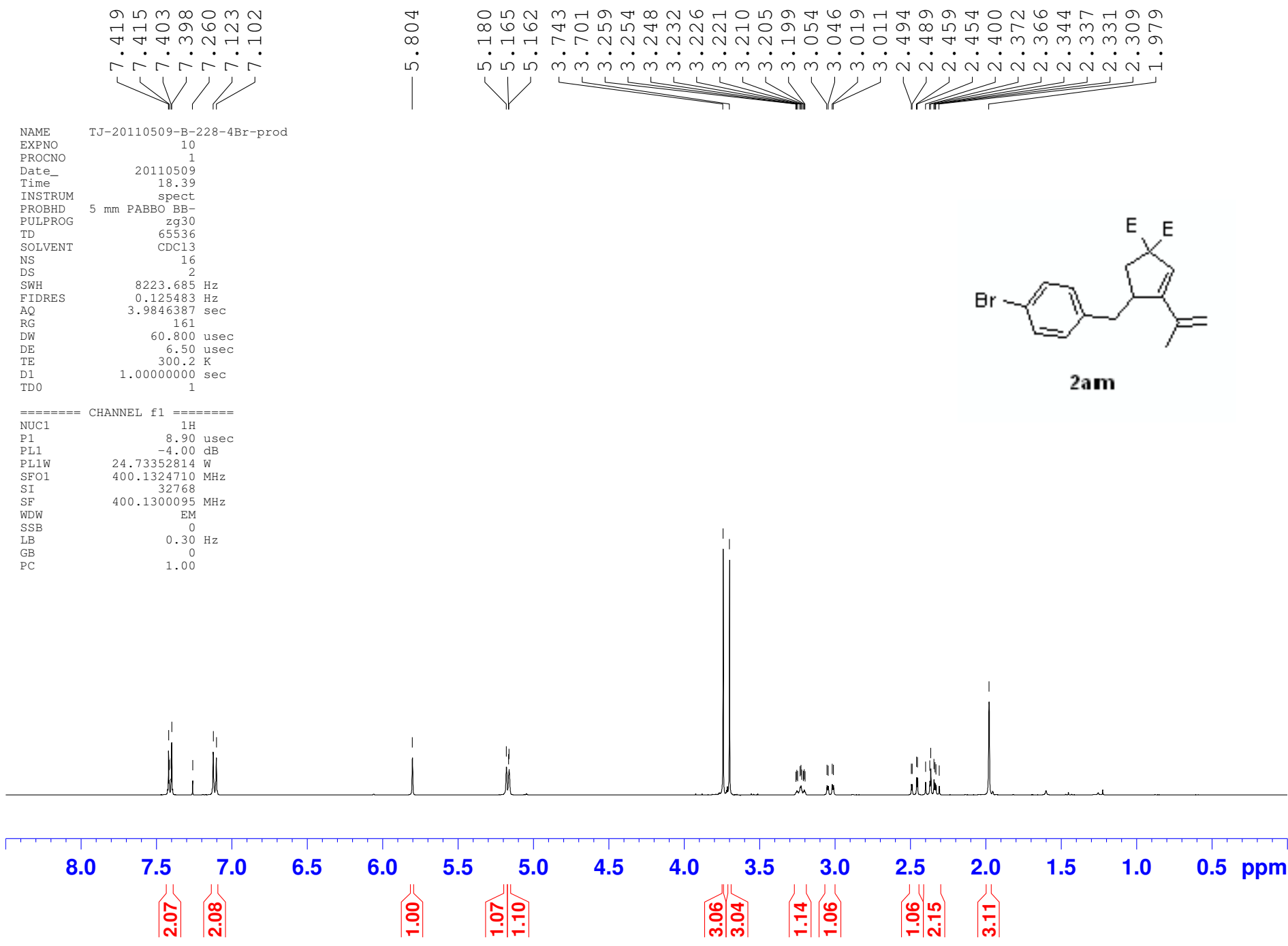


2ak





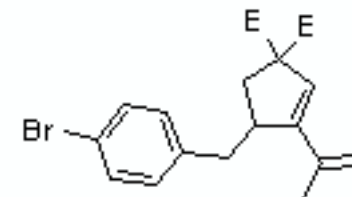




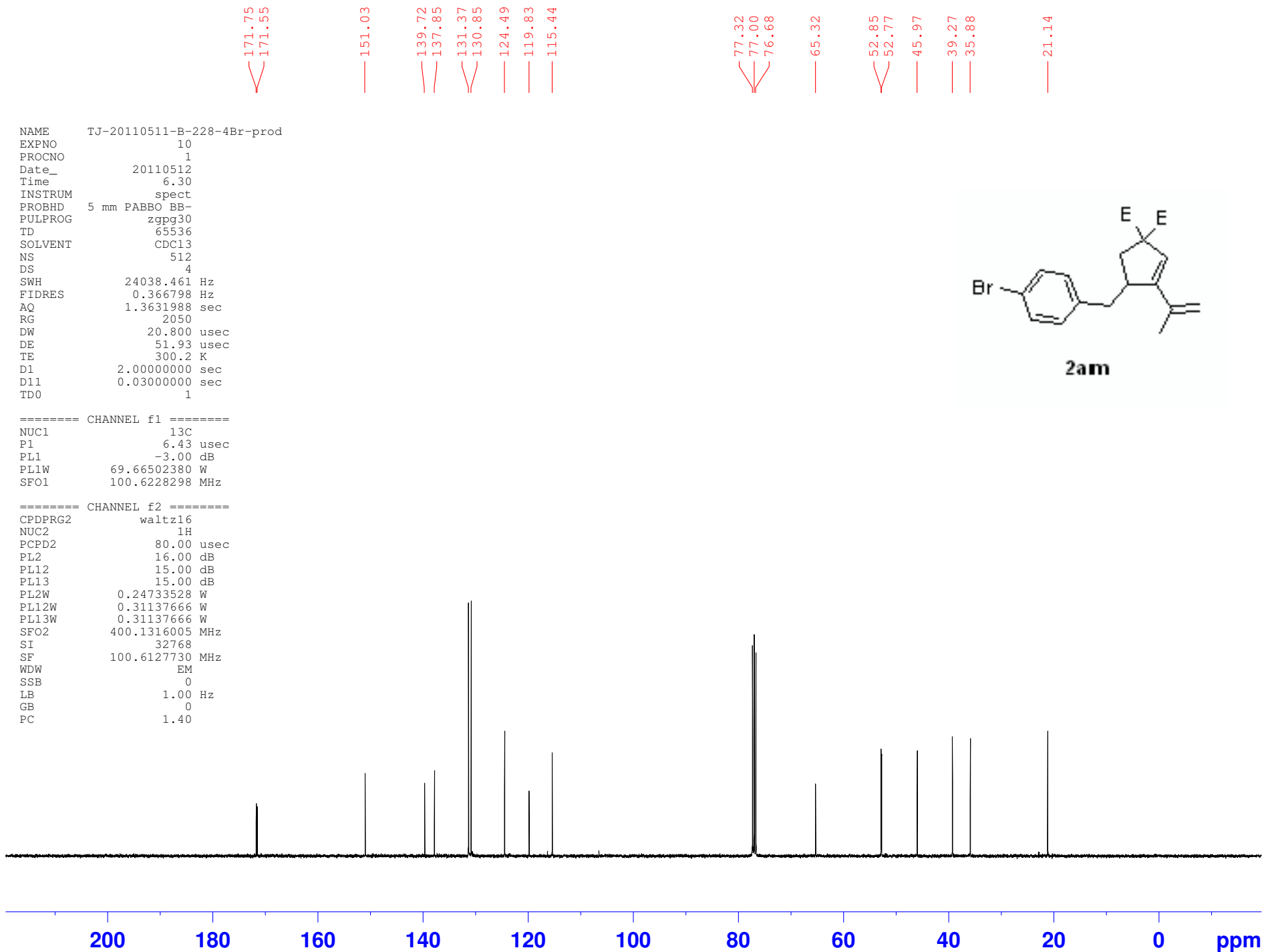
NAME TJ-20110511-B-228-4Br-prod
 EXPNO 10
 PROCNO 1
 Date_ 20110512
 Time 6.30
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 51.93 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

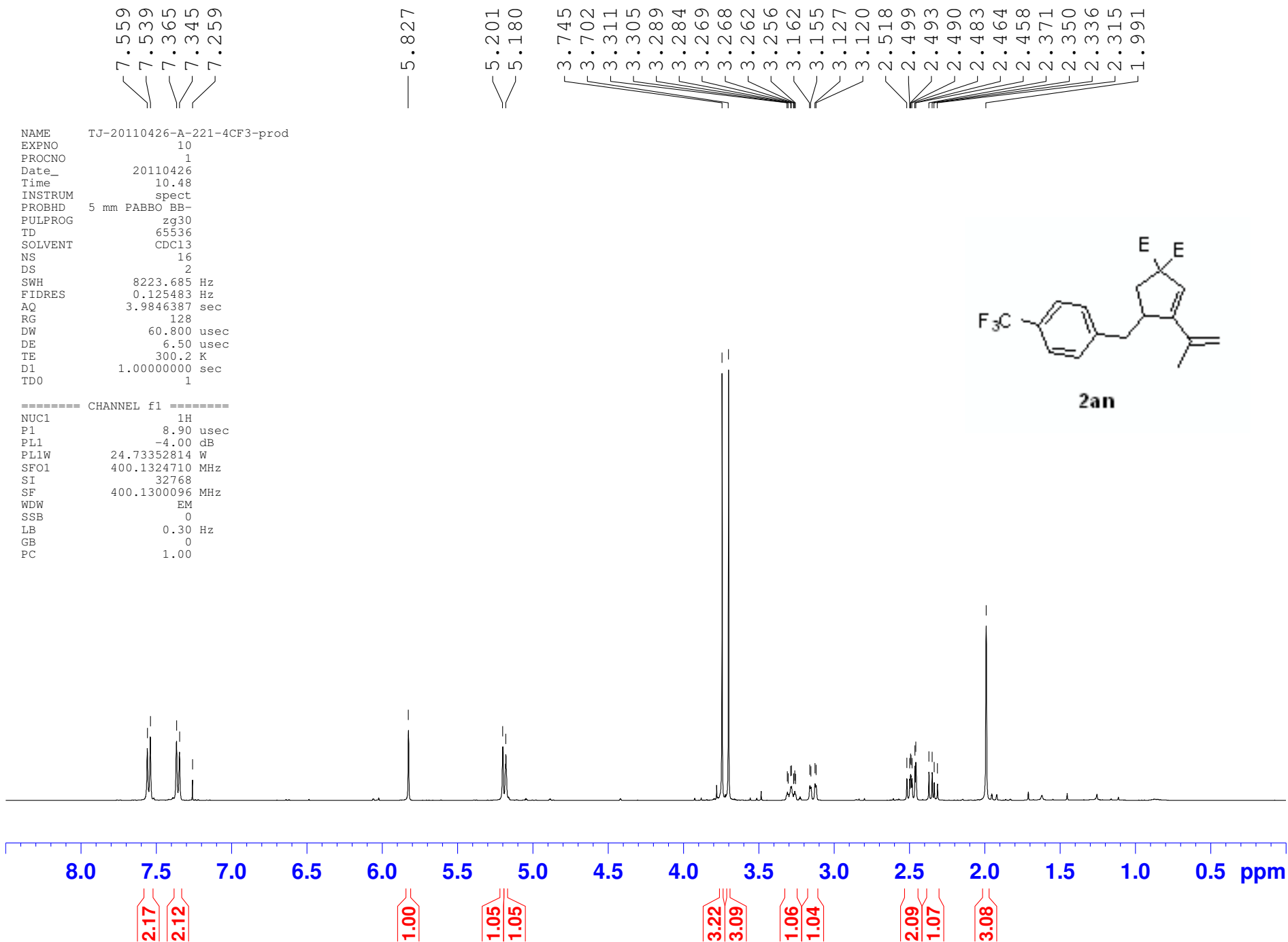
===== CHANNEL f1 =====
 NUC1 13C
 P1 6.43 usec
 PL1 -3.00 dB
 PL1W 69.66502380 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 16.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 0.24733528 W
 PL12W 0.31137666 W
 PL13W 0.31137666 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127730 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



2am

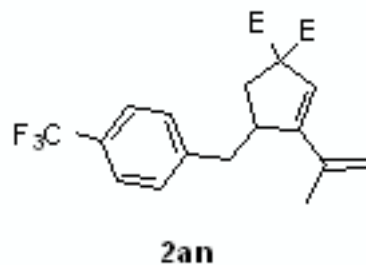




NAME TJ-20110426-A-221-4CF3-prod
 EXPNO 20
 PROCNO 1
 Date_ 20110426
 Time 23.05
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 51.93 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.43 usec
 PL1 -3.00 dB
 PL1W 69.66502380 W
 SFO1 100.6228298 MHz

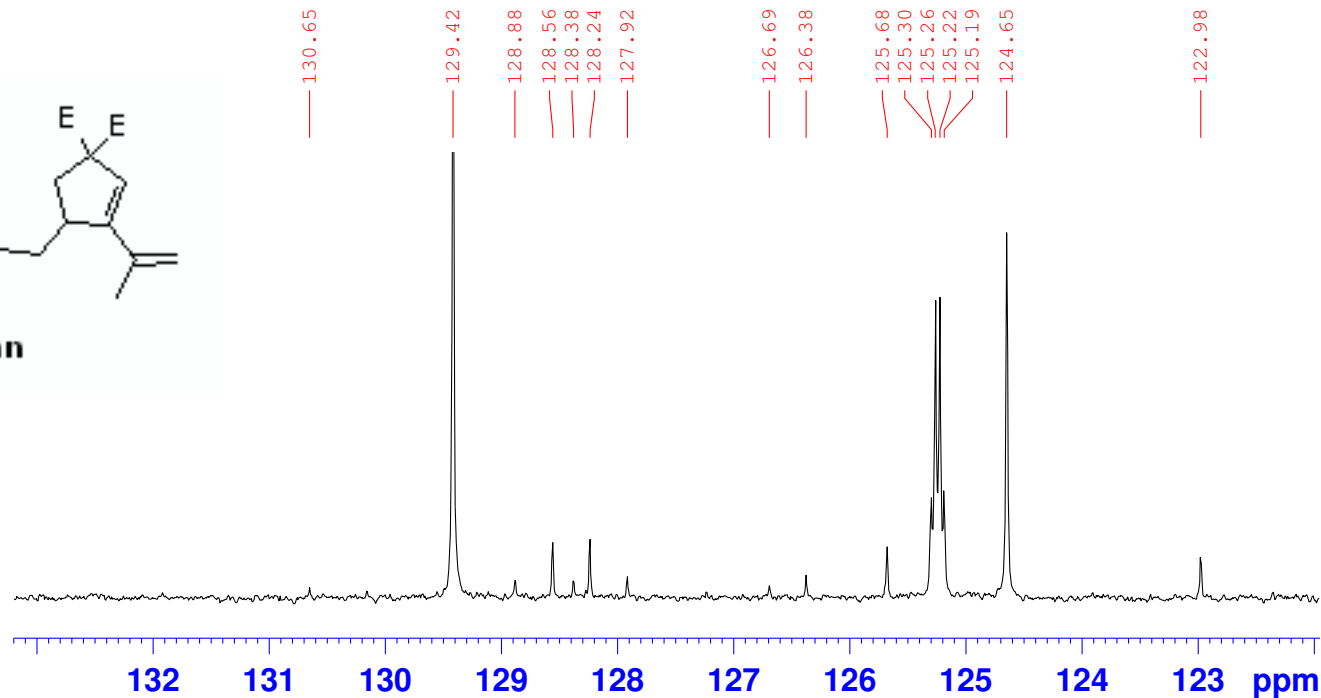
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 16.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 0.24733528 W
 PL12W 0.31137666 W
 PL13W 0.31137666 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127715 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



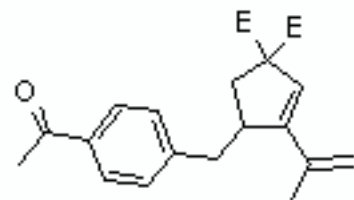
171.71
 171.52
 150.94
 144.92
 137.85
 130.65
 129.42
 128.88
 128.56
 128.38
 128.24
 127.92
 126.69
 126.38
 125.68
 125.30
 125.26
 125.22
 125.19
 124.65
 122.98
 115.47

77.32
 77.00
 76.68
 65.36
 52.85
 52.77
 45.86
 39.70
 35.86
 21.13

130.65
 129.42
 128.88
 128.56
 128.38
 128.24
 127.92
 126.69
 126.38
 125.68
 125.30
 125.26
 125.22
 125.19
 124.65
 122.98



200 180 160 140 120 100 80 60 40 20 0 ppm



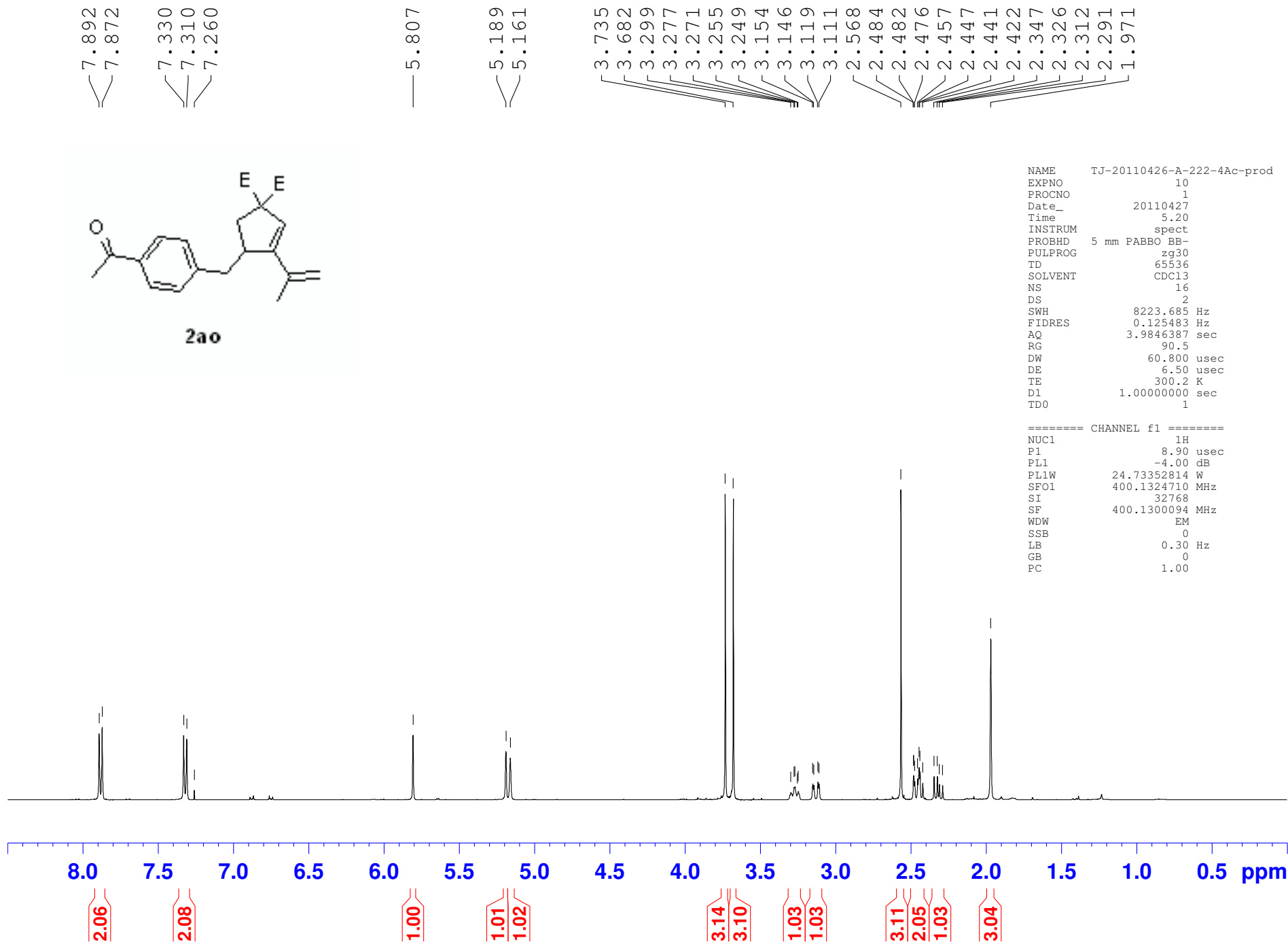
2ao

```

NAME      TJ-20110426-A-222-4Ac-prod
EXPNO     10
PROCNO    1
Date_     20110427
Time      5.20
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        90.5
DW        60.800 usec
DE        6.50 usec
TE        300.2 K
D1        1.00000000 sec
TD0       1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        8.90 usec
PL1       -4.00 dB
PL1W      24.73352814 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300094 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



— 197.74

— 171.63
— 171.47

— 150.96

— 146.59

— 137.75

— 135.17

— 129.22

— 128.45

— 124.49

— 115.44

77.31
76.99
76.67

— 65.27

52.79
52.70

— 45.77

— 39.88

— 35.87

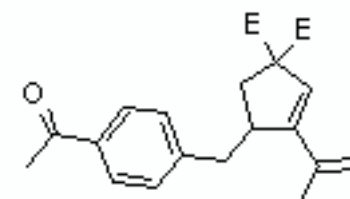
— 26.45

— 21.07

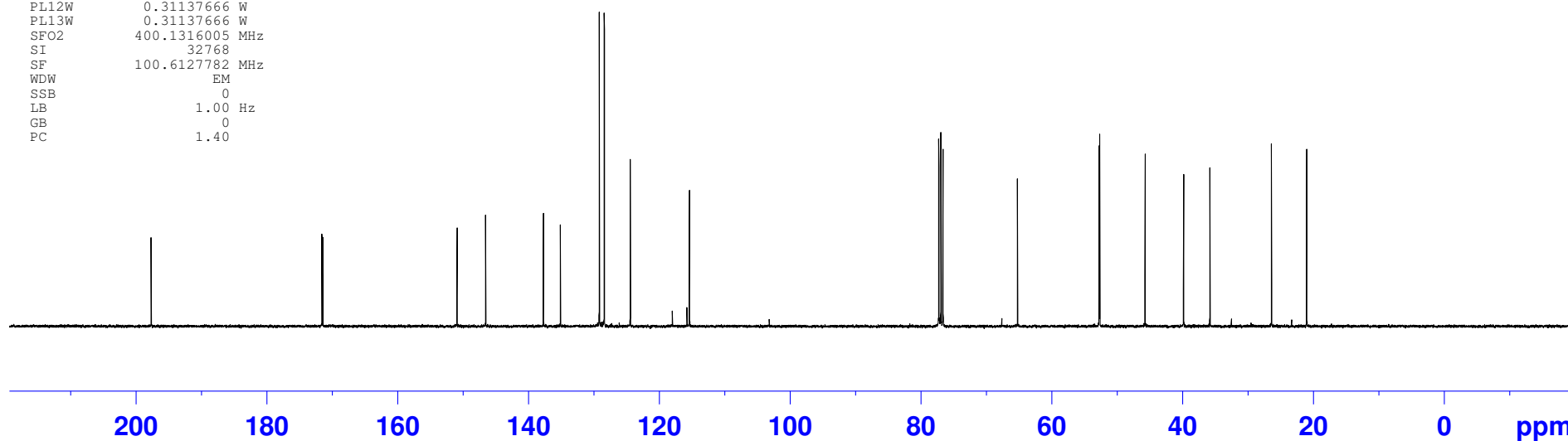
NAME TJ-20110426-A-222-4Ac-prod
EXPNO 11
PROCNO 1
Date_ 20110427
Time 5.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127782 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

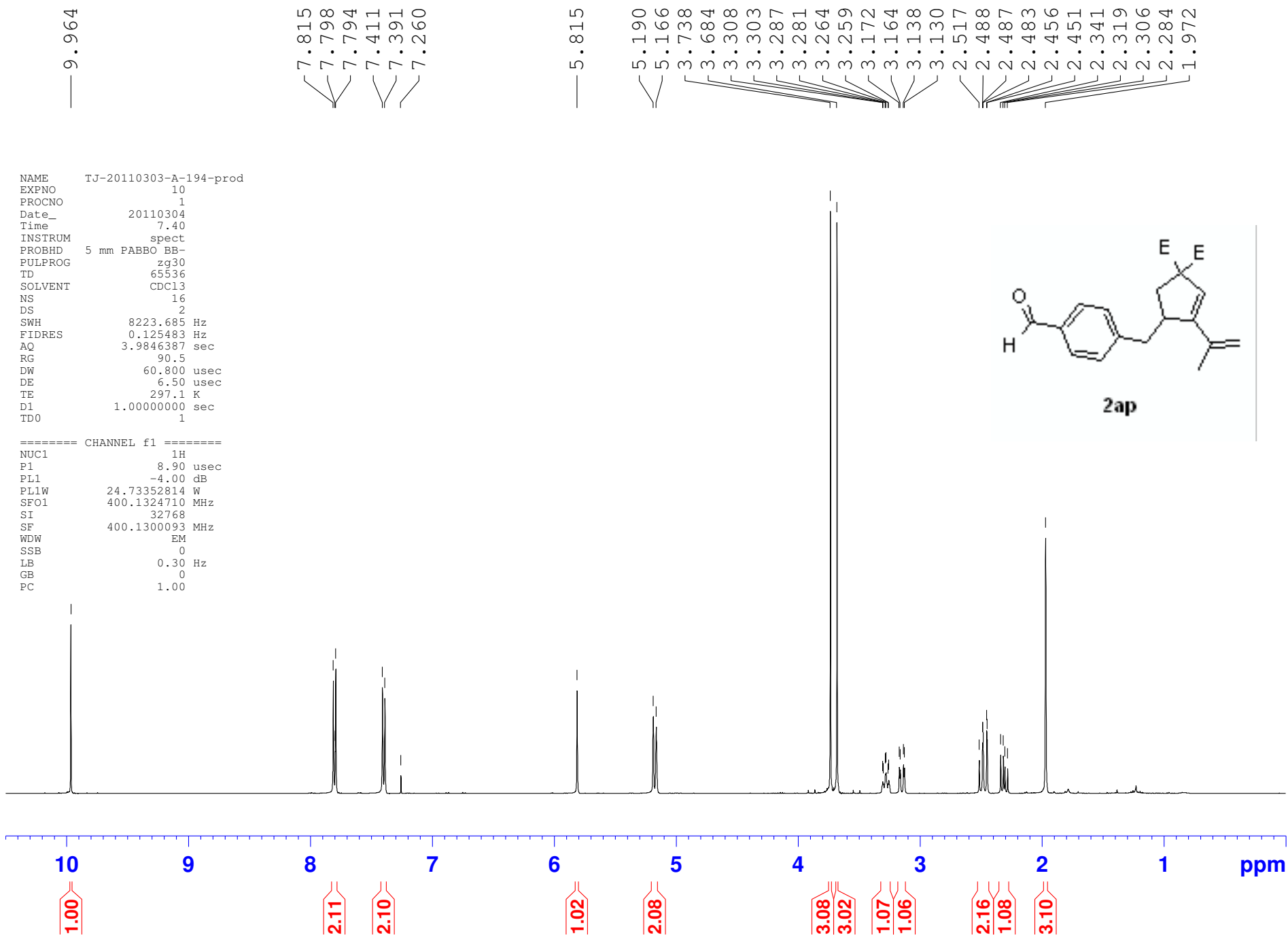


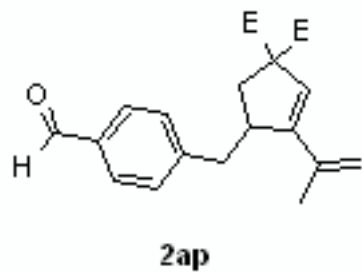
2ao



NAME TJ-20110303-A-194-prod
 EXPNO 10
 PROCNO 1
 Date_ 20110304
 Time 7.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 90.5
 DW 60.800 usec
 DE 6.50 usec
 TE 297.1 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.90 usec
 PL1 -4.00 dB
 PL1W 24.73352814 W
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300093 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





```

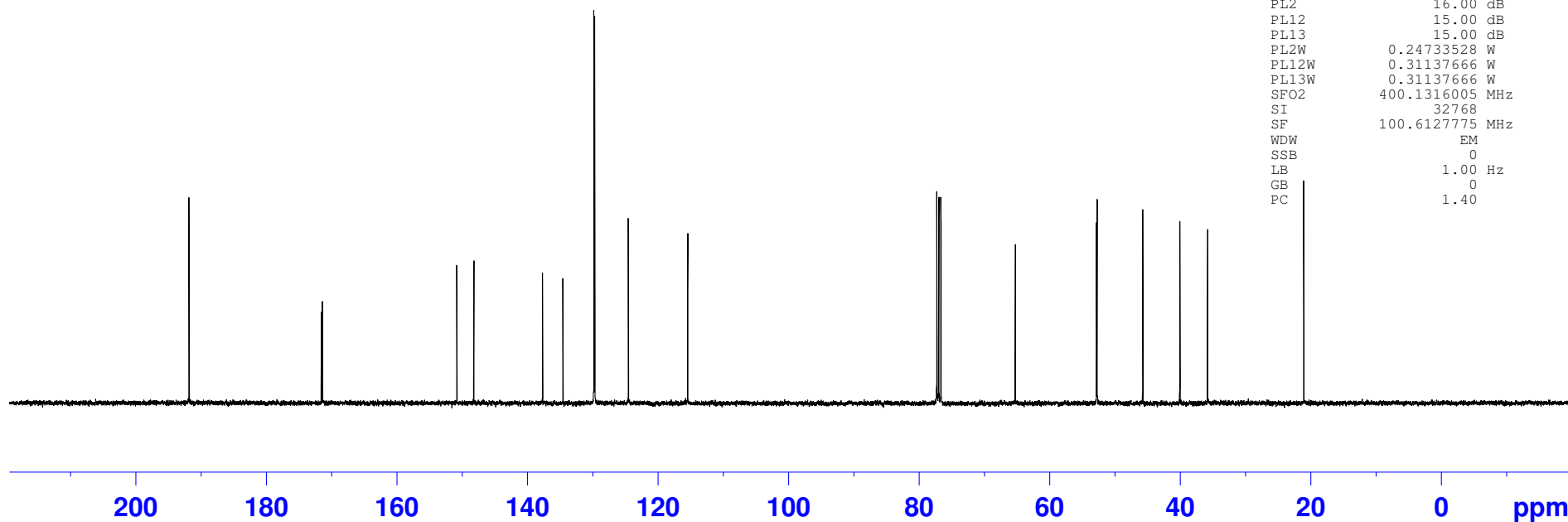
NAME      TJ-20110303-A-194-prod
EXPNO     11
PROCNO    1
Date_     20110304
Time      7.52
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         172
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.800 usec
DE         51.93 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

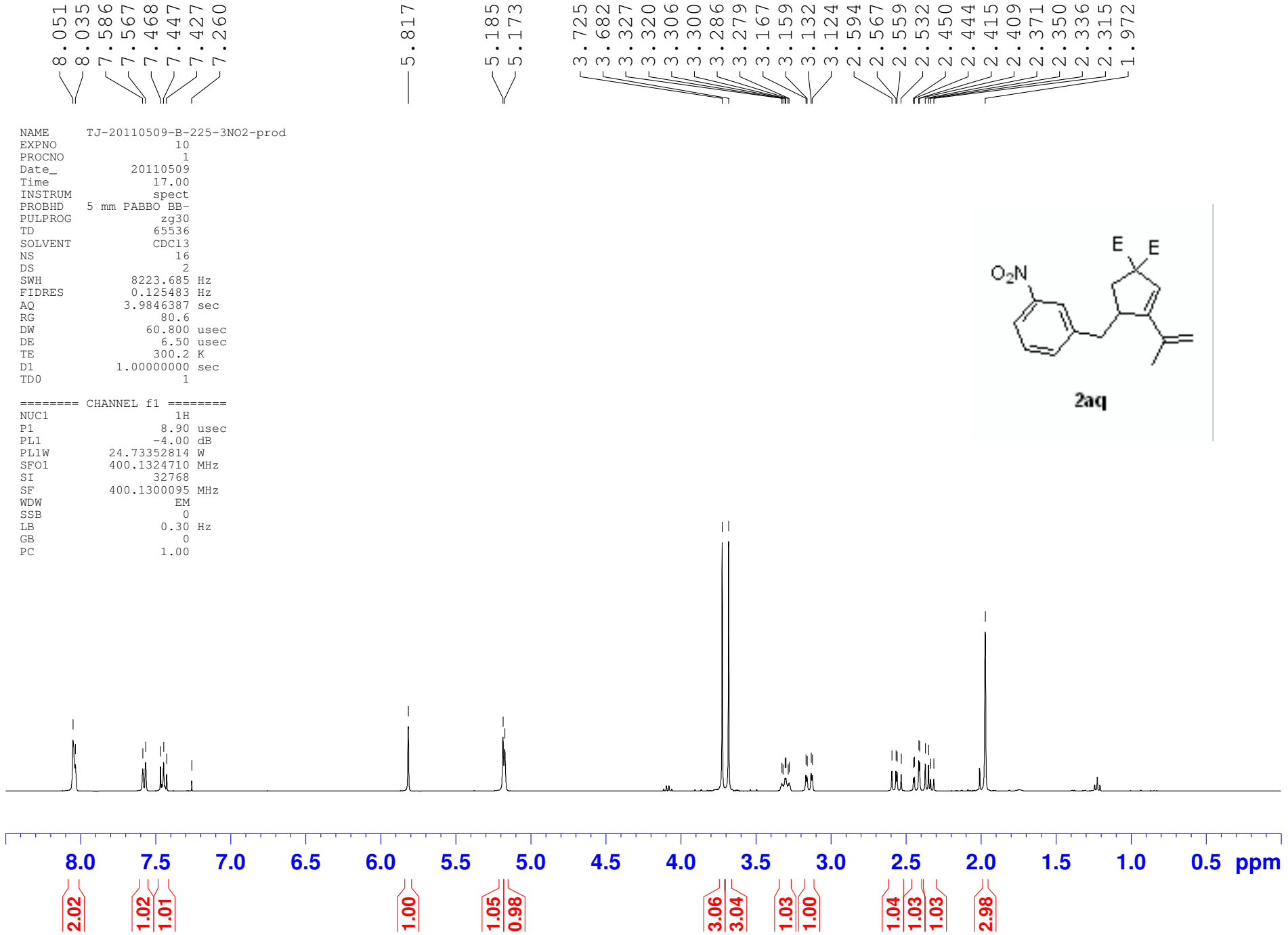
```

===== CHANNEL f1 =====
NUC1       13C
P1         6.43 usec
PL1        -3.00 dB
PL1W       69.66502380 W
SFO1       100.6228298 MHz
  
```

```

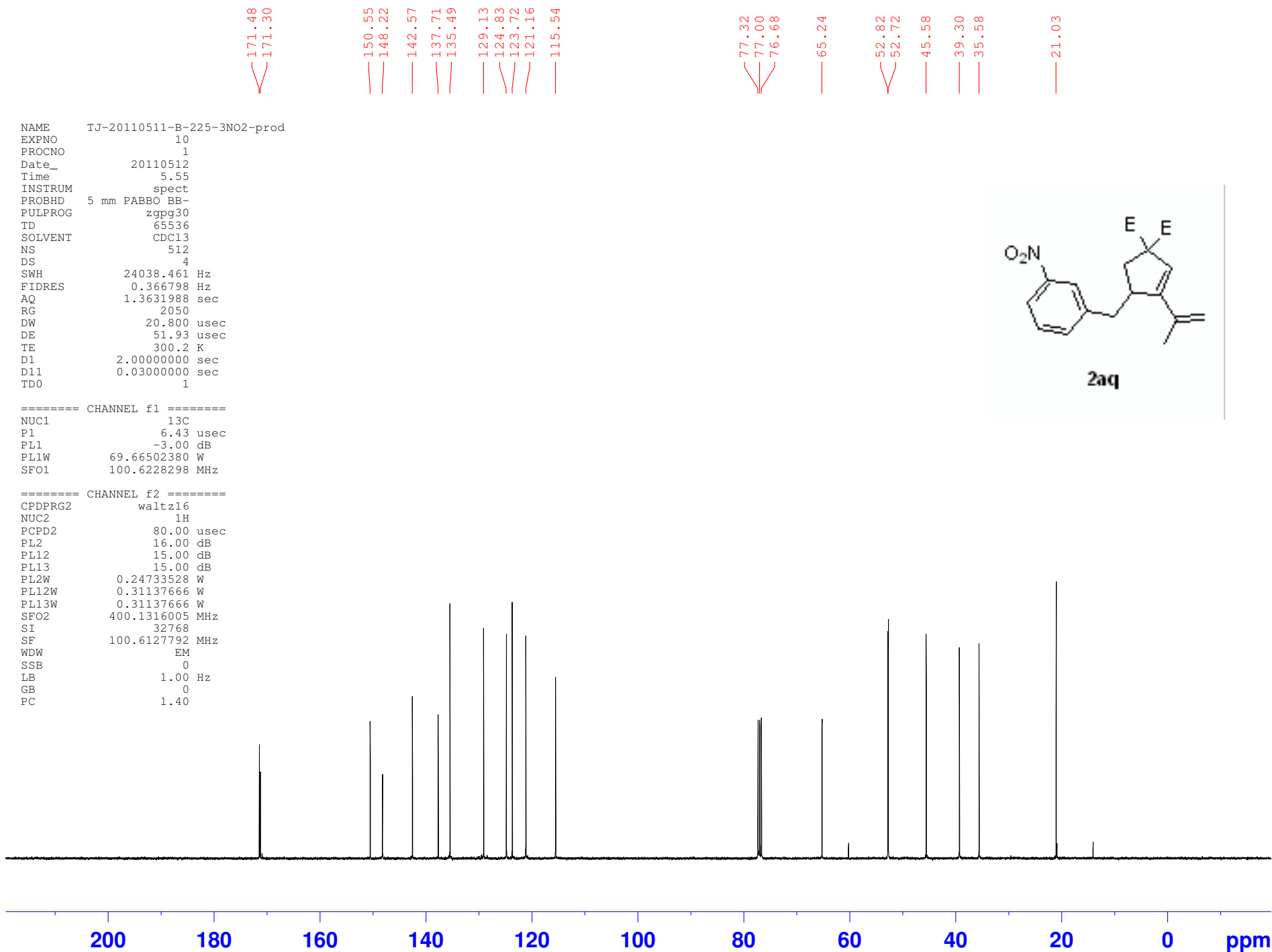
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2      80.00 usec
PL2        16.00 dB
PL12       15.00 dB
PL13       15.00 dB
PL2W       0.24733528 W
PL12W      0.31137666 W
PL13W      0.31137666 W
SFO2       400.1316005 MHz
SI         32768
SF         100.6127775 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

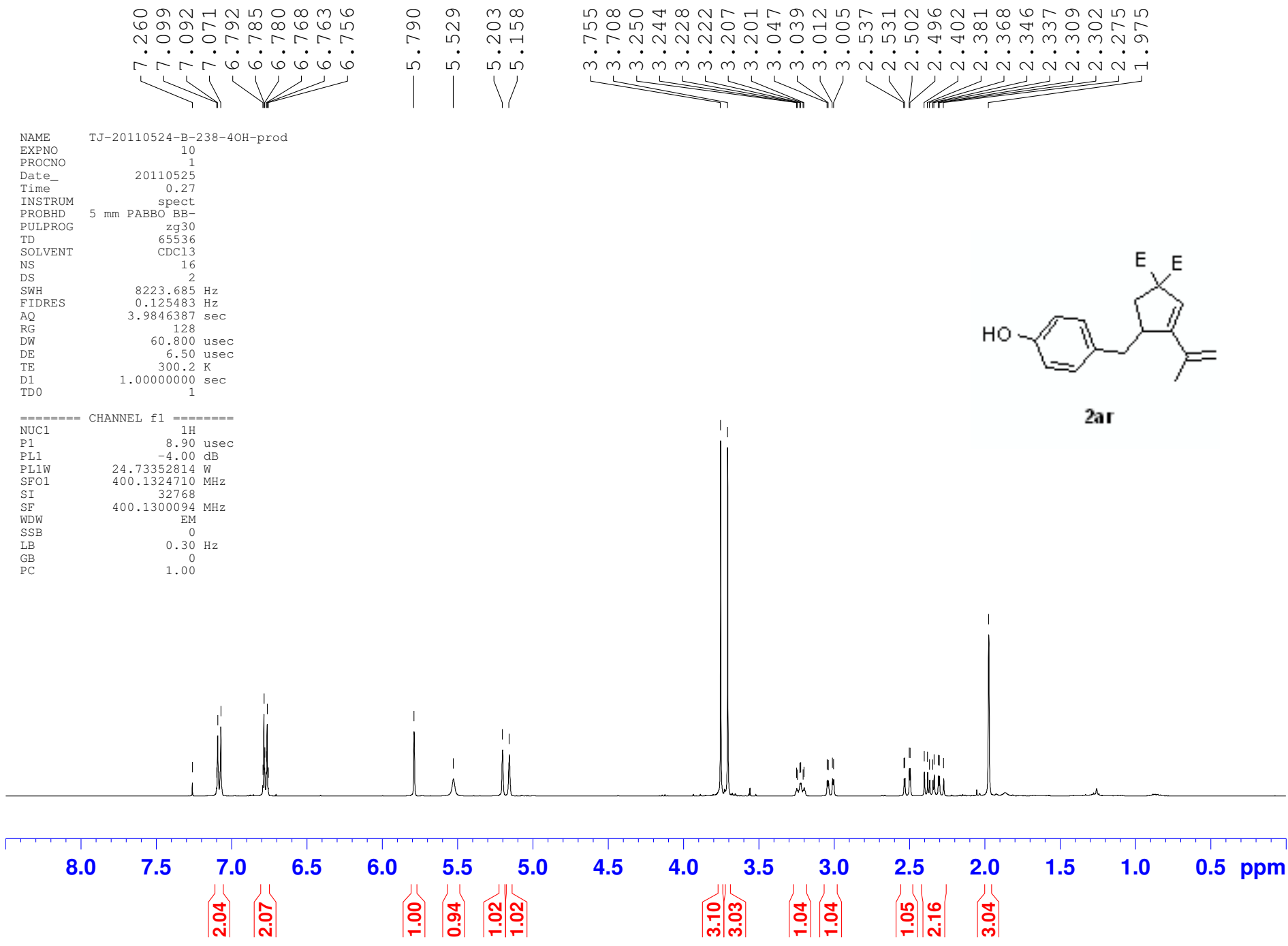




NAME TJ-20110509-B-225-3NO2-prod
EXPNO 10
PROCNO 1
Date_ 20110509
Time 17.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





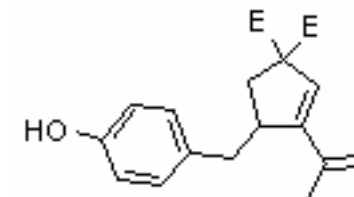
¹³C NMR chemical shifts (ppm):
 172.12, 171.96, 154.07, 151.55, 137.89, 132.63, 130.10, 124.02, 115.47, 115.20

¹³C NMR chemical shifts (ppm):
 77.32, 77.00, 76.68, 65.36, 52.90, 52.85, 46.32, 39.05, 36.07, 21.12

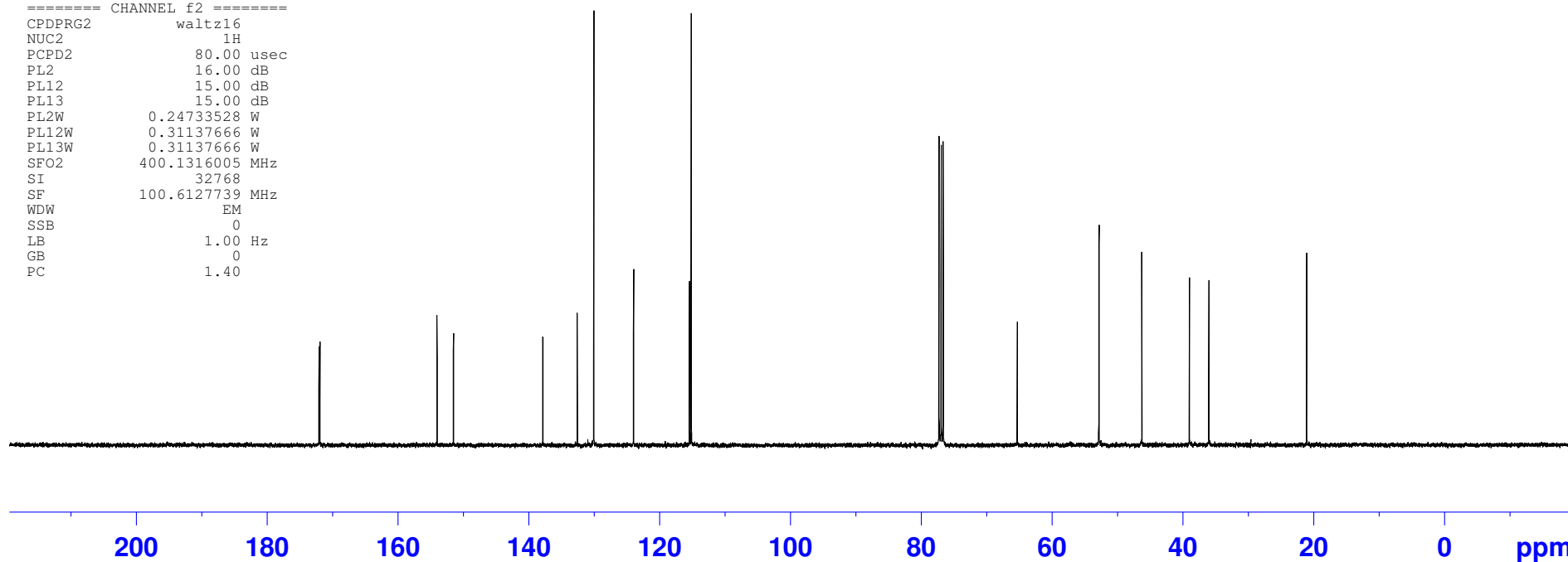
NAME TJ-20110524-B-238-4OH-prod
 EXPNO 11
 PROCNO 1
 Date_ 20110525
 Time 0.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 51.93 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.43 usec
 PL1 -3.00 dB
 PL1W 69.66502380 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 16.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 0.24733528 W
 PL12W 0.31137666 W
 PL13W 0.31137666 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127739 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



2ar



7.274
7.271
7.267
7.260
7.256
7.253
7.250
7.236
7.212
7.207
7.202
7.190
7.183
7.173

— 5.735
5.335
5.173
5.169
5.166

3.698
3.630

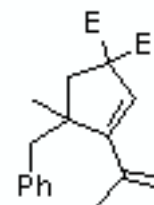
2.933
2.900
2.887
2.853
2.800
2.764

2.132
2.097
1.989
1.987

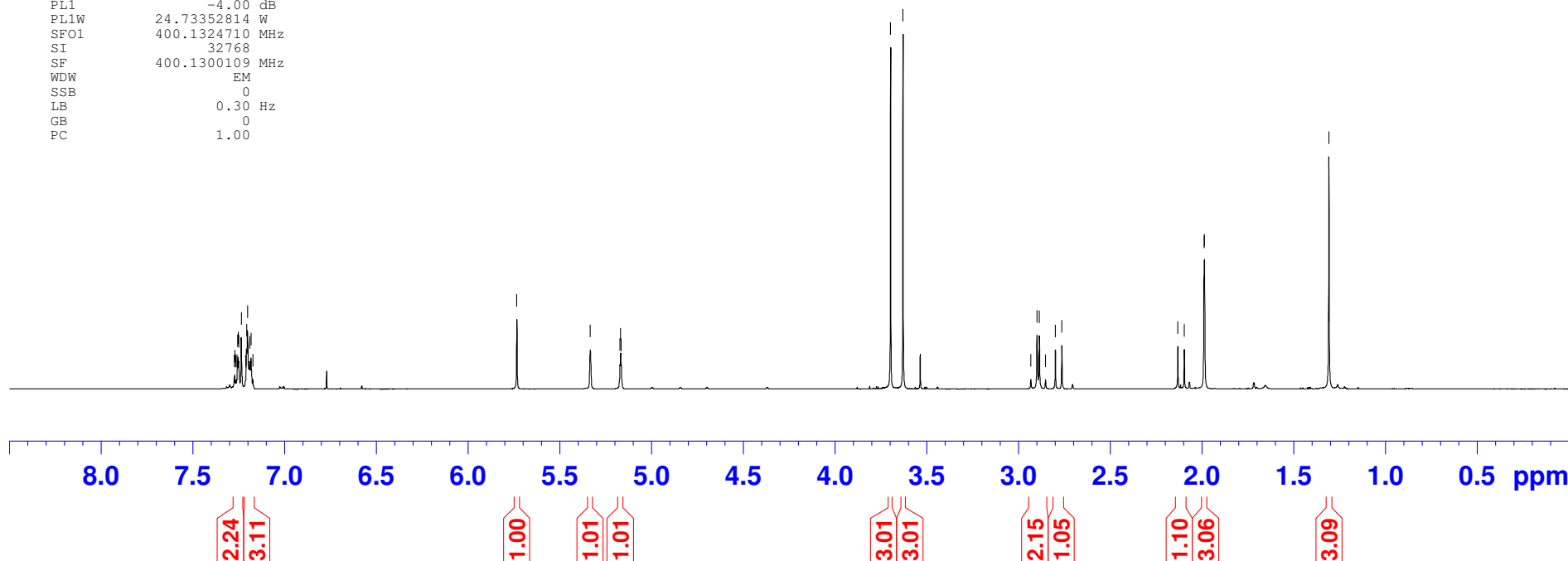
— 1.309

NAME TJ-20110512-B-242-internalMe-prod
EXPNO 10
PROCNO 1
Date_ 20110513
Time 5.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300109 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



2b



NAME TJ-20110512-B-242-internalMe-prod
 EXPNO 11
 PROCNO 1
 Date_ 20110513
 Time 5.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 51.93 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.43 usec
 PL1 -3.00 dB
 PL1W 69.66502380 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 16.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 0.24733528 W
 PL12W 0.31137666 W
 PL13W 0.31137666 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127751 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

171.80
171.48

153.84

138.55
138.18

130.56
127.80

126.06
125.67

115.45

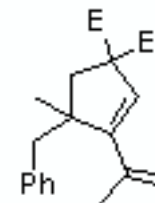
77.32
77.00
76.68

63.22

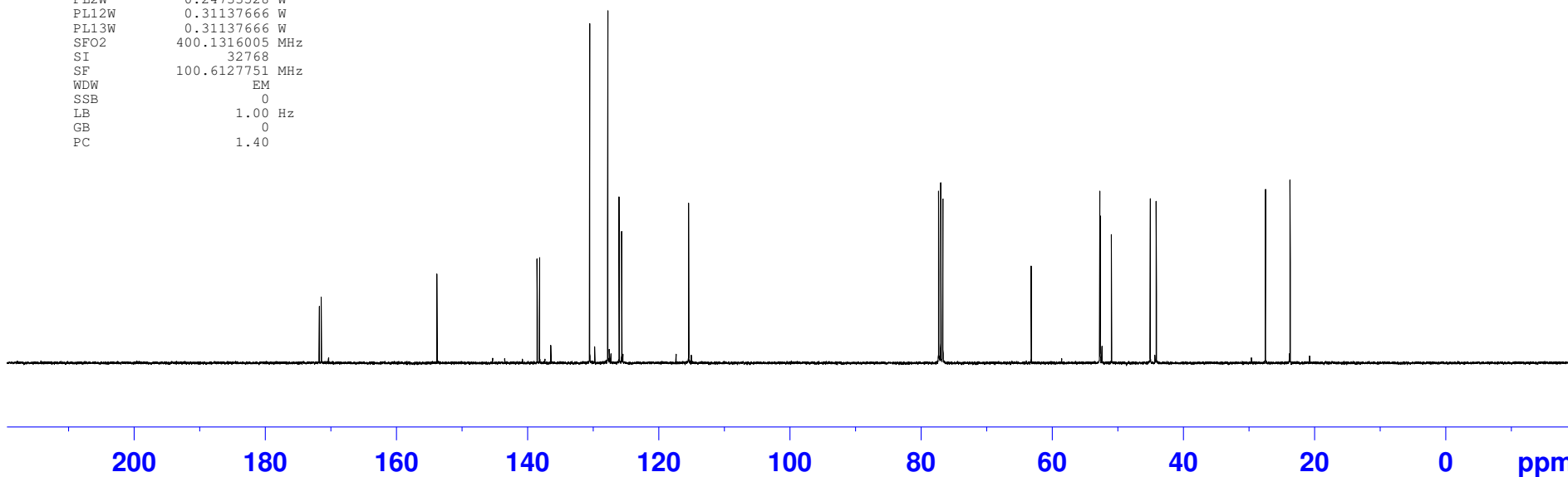
52.76
52.68

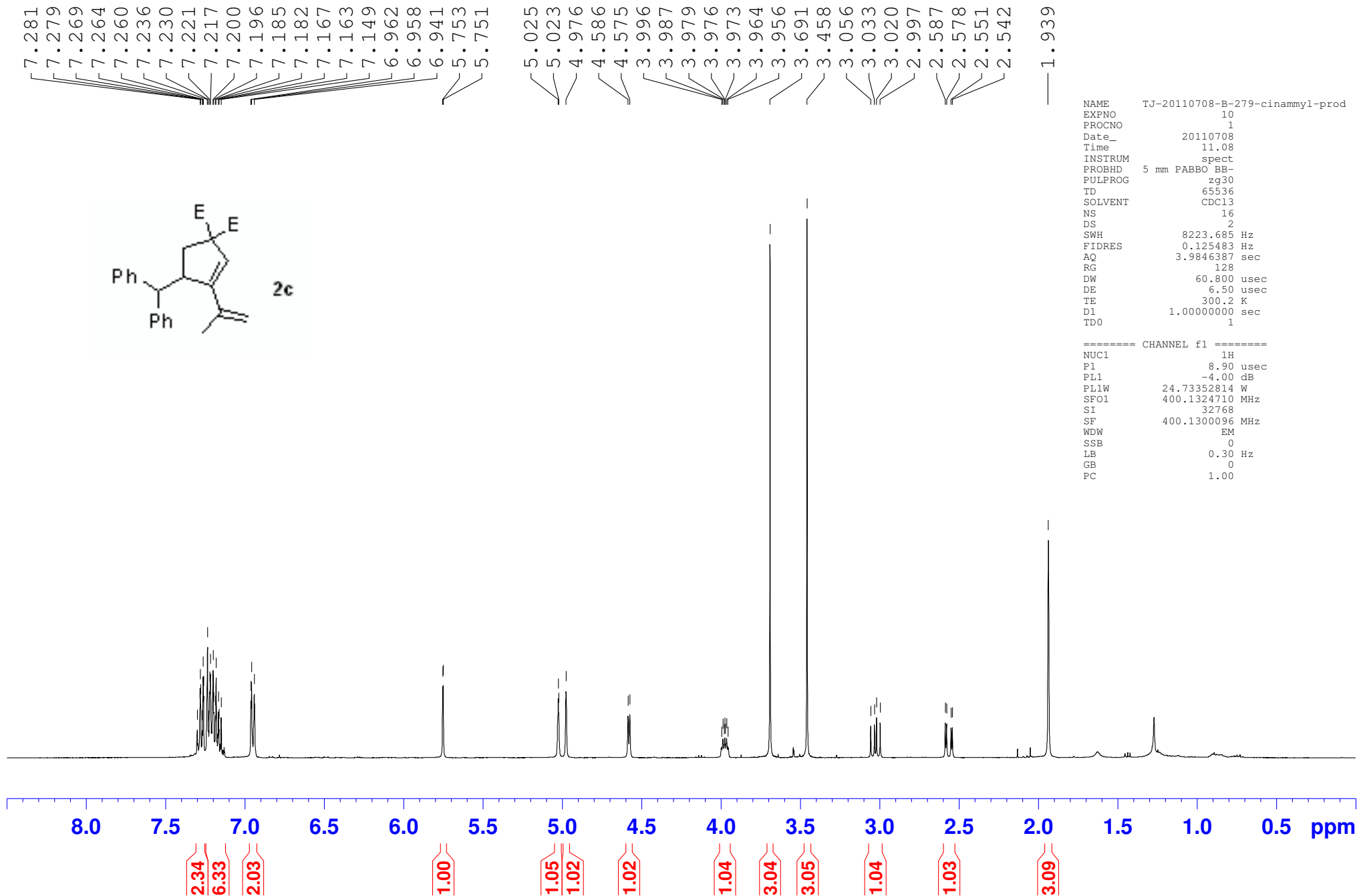
50.96
45.05
44.13

27.48
23.72



2b

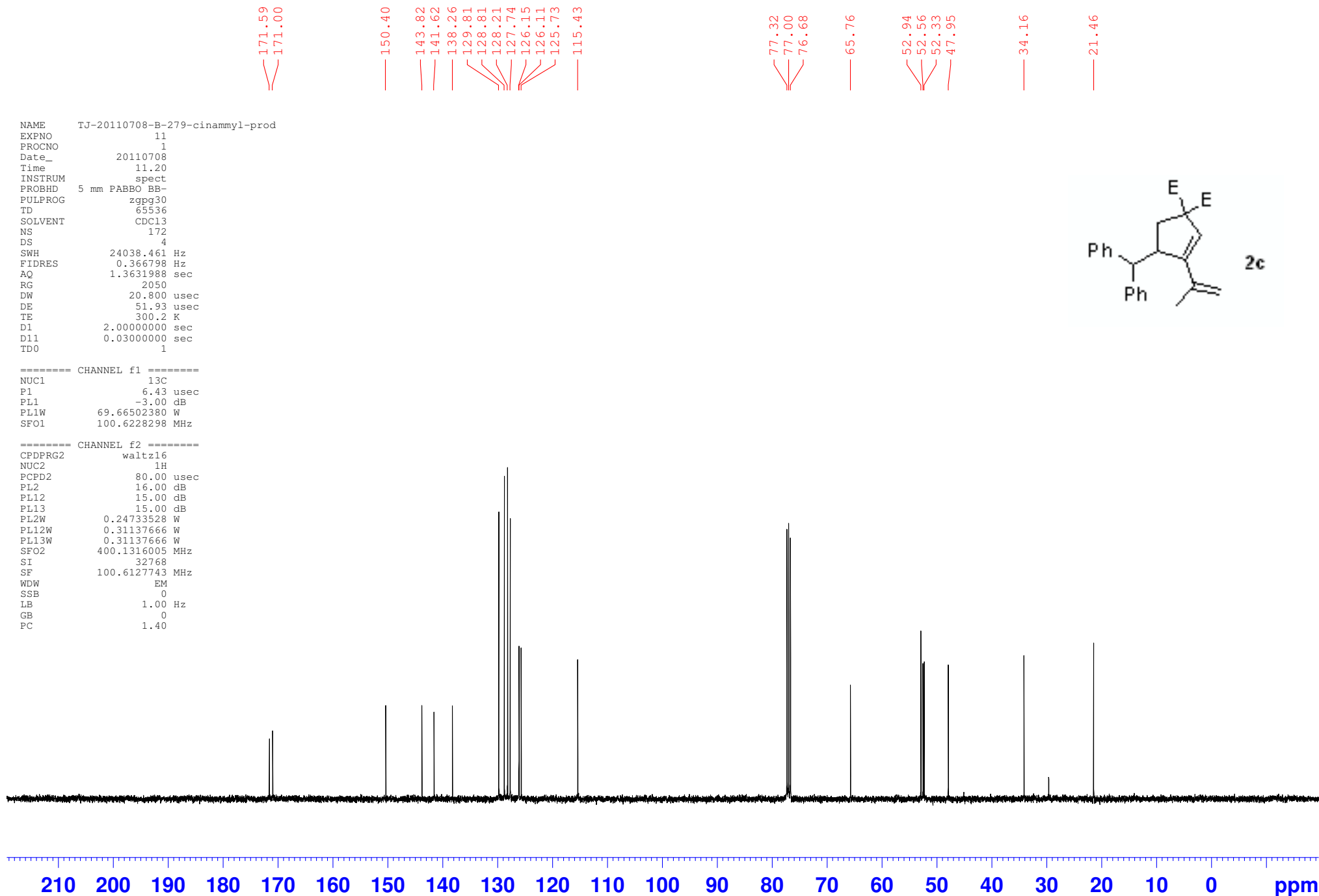
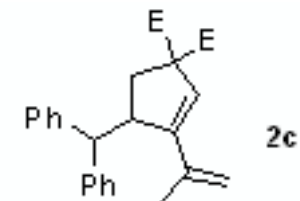




NAME TJ-20110708-B-279-cinammyl-prod
 EXPNO 11
 PROCNO 1
 Date_ 20110708
 Time 11.20
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 172
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 51.93 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

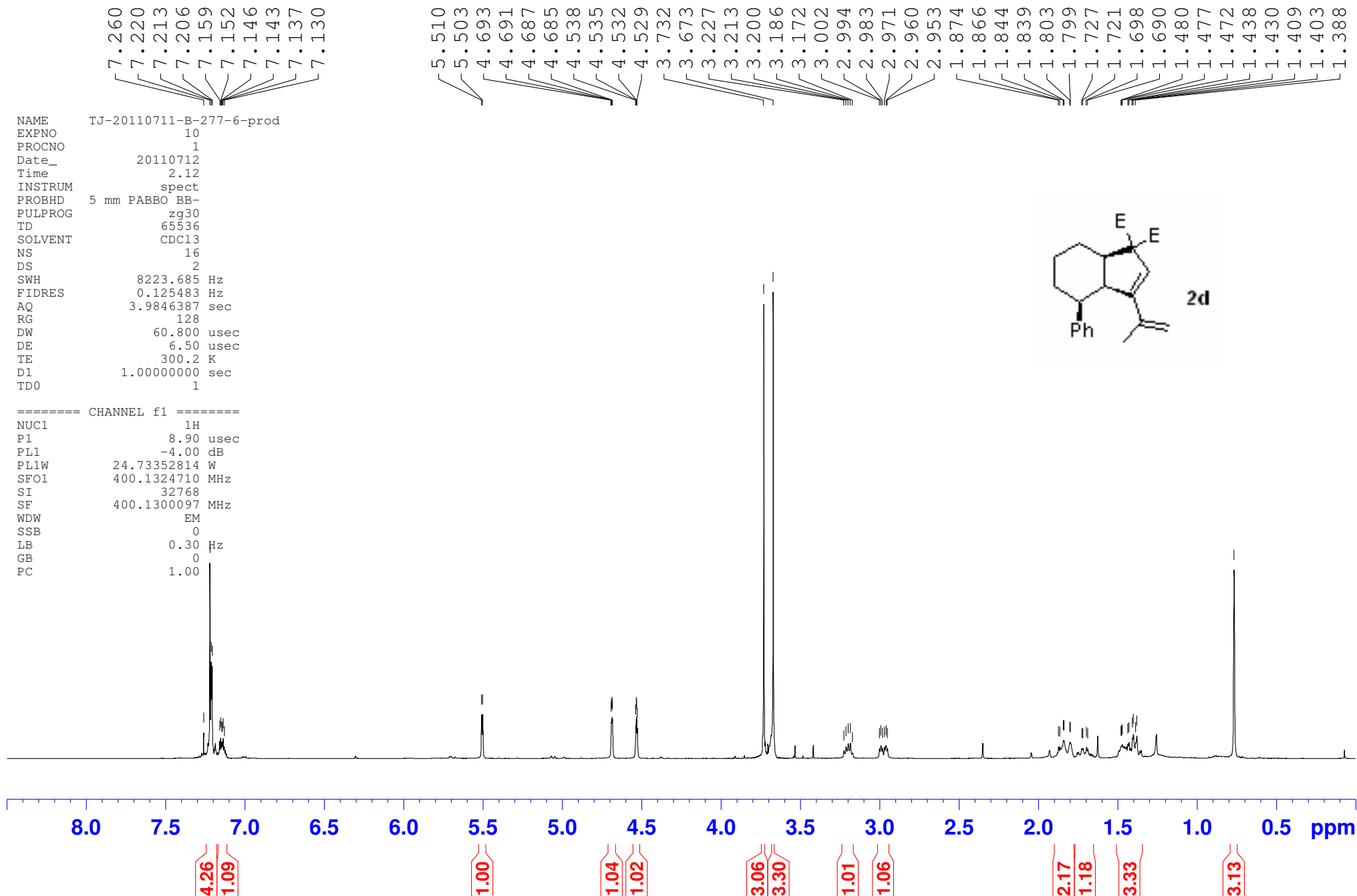
===== CHANNEL f1 =====
 NUC1 13C
 P1 6.43 usec
 PL1 -3.00 dB
 PL1W 69.66502380 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 16.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 0.24733528 W
 PL12W 0.31137666 W
 PL13W 0.31137666 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127743 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



NAME TJ-20110711-B-277-6-prod
 EXPNO 10
 PROCNO 1
 Date_ 20110712
 Time 2.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 128
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.90 usec
 PL1 -4.00 dB
 PL1W 24.73352814 W
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300097 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



NAME TJ-20110711-B-277-6-prod
 EXPNO 11
 PROCNO 1
 Date_ 20110712
 Time 2.44
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 51.93 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.43 usec
 PL1 -3.00 dB
 PL1W 69.66502380 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 16.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 PL2W 0.24733528 W
 PL12W 0.31137666 W
 PL13W 0.31137666 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127728 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

170.44
169.64

153.47

144.30
143.37

128.76
127.58
125.79
125.63

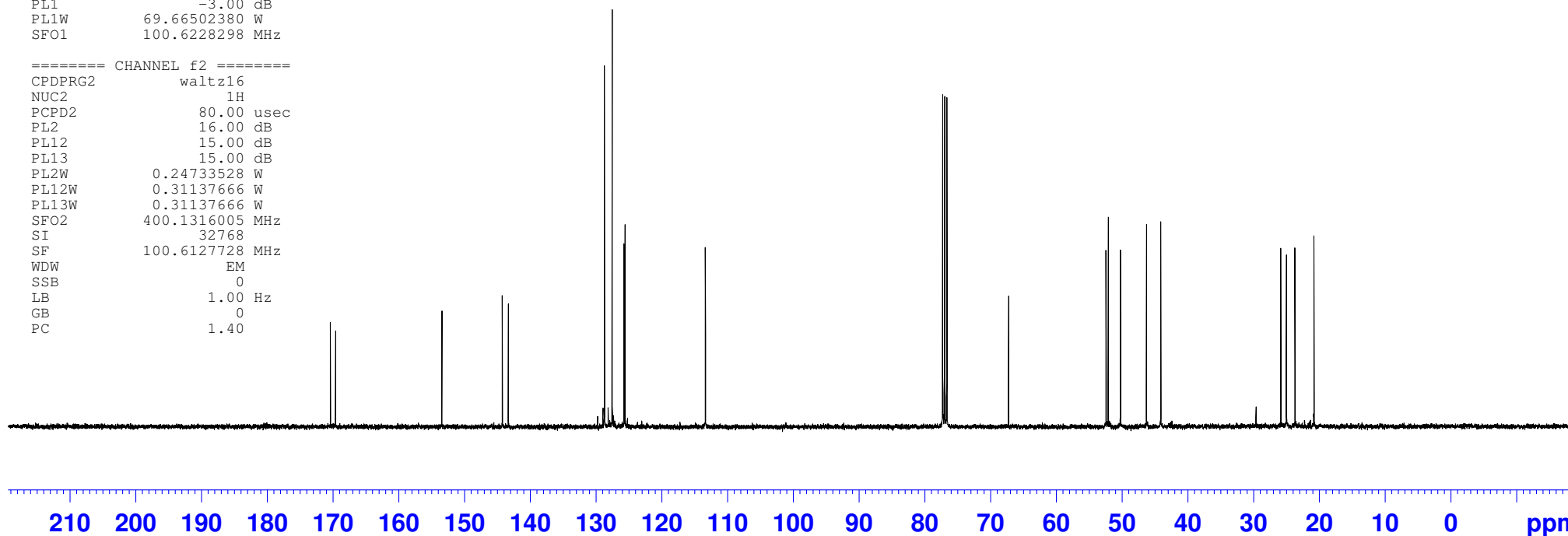
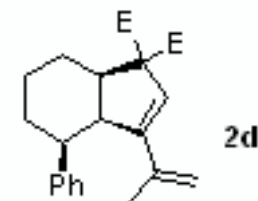
113.41

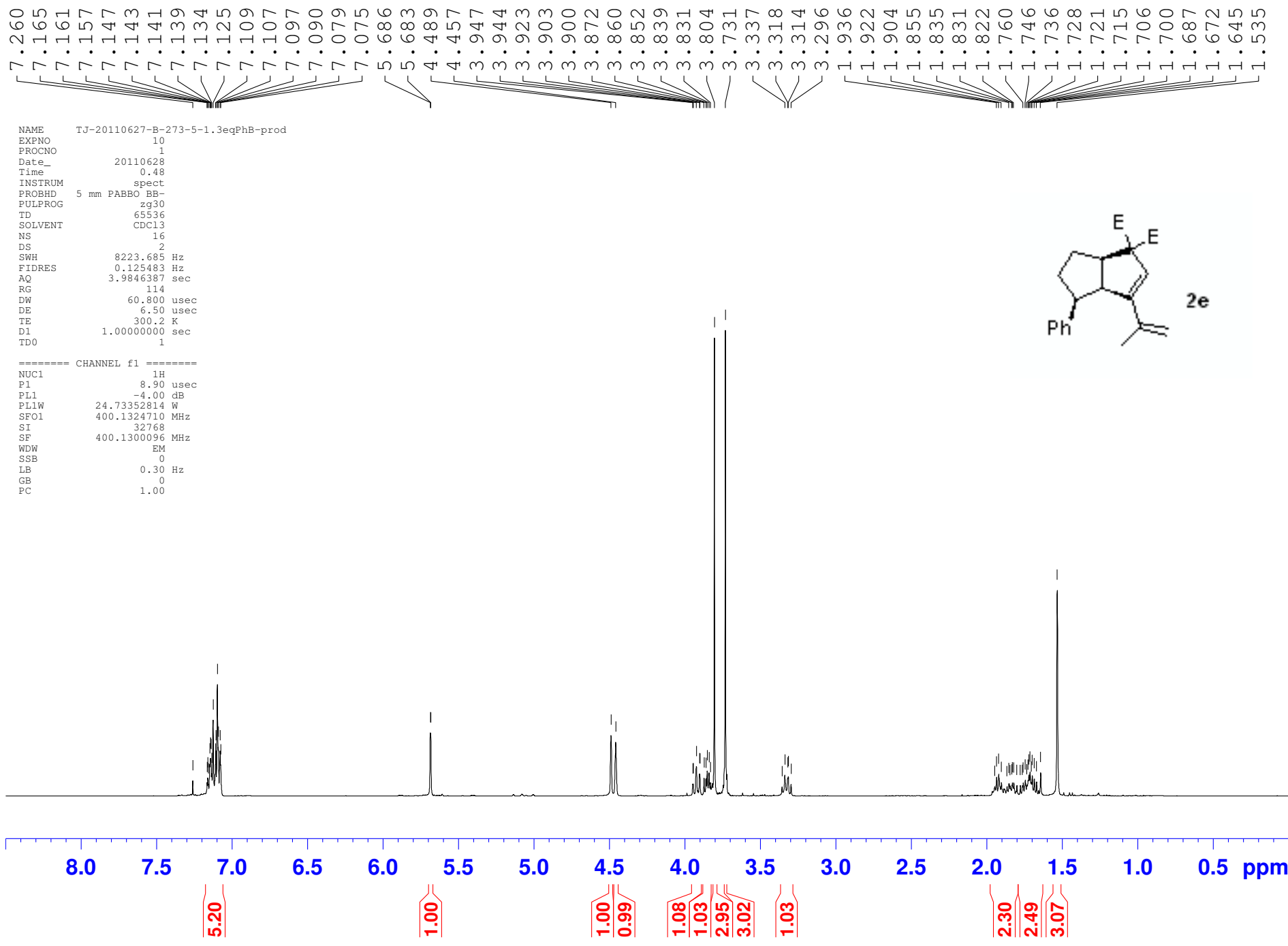
77.32
77.00
76.69

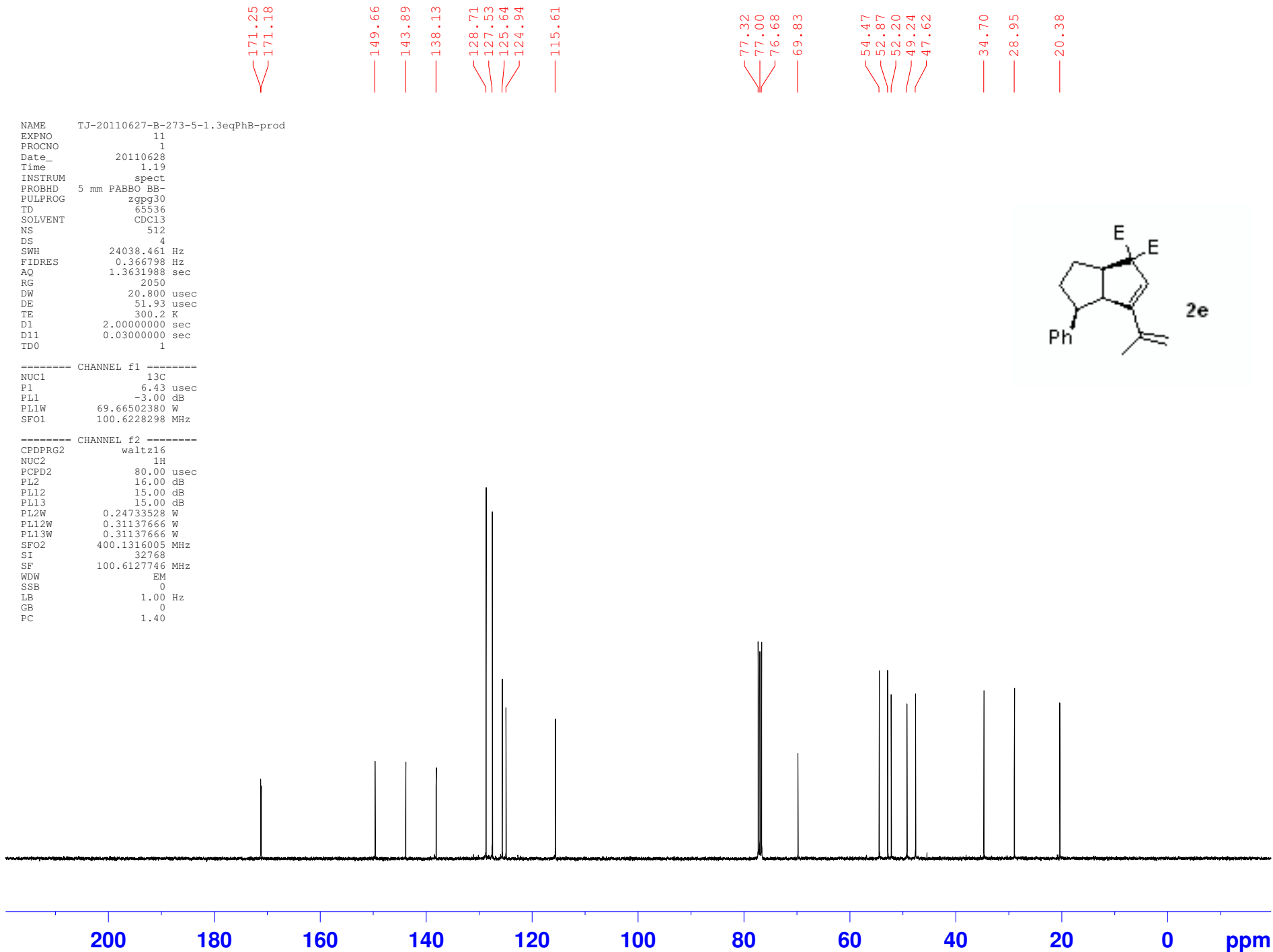
67.30

52.48
52.15
50.29
46.33
44.16

25.90
25.06
23.75
20.85







4mg

170.41

145.39

144.47

137.43

137.19

133.55

129.87

128.48

127.62

127.42

127.35

126.22

125.19

124.43

117.71

77.32

77.00

76.68

59.33

52.50

40.82

20.88

```

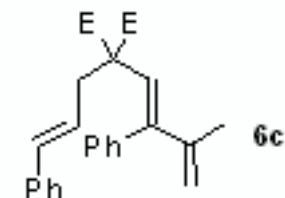
NAME      TJ-20110719-B-279-cinammyl-frac65-75
EXPNO     10
PROCNO    1
Date_     20110719
Time      23.25
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        1024
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        2050
DW        20.800 usec
DE        51.93 usec
TE        300.2 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        6.43 usec
PL1       -3.00 dB
PL1W      69.66502380 W
SFO1      100.6228298 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       16.00 dB
PL12      15.00 dB
PL13      15.00 dB
PL2W      0.24733528 W
PL12W     0.31137666 W
PL13W     0.31137666 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6127701 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

4mg

7.330
7.325
7.316
7.311
7.308
7.306
7.289
7.260
7.228
7.212
7.054
7.035
6.483
6.409
6.370
6.128
6.109
6.090
6.069
6.051

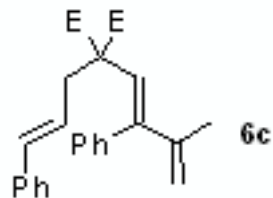
5.016

4.410
4.406

3.537

2.811
2.808
2.793
2.790

2.060
2.059



```

NAME      TJ-20110719-B-279-cinammyl-frac65-
EXPNO     11
PROCNO    1
Date_     20110719
Time      23.27
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        406
DW        60.800 usec
DE        6.50 usec
TE        300.2 K
D1        1.00000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      1H
P1        8.90 usec
PL1       -4.00 dB
PL1W      24.73352814 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300097 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

