SUPPORTING INFORMATION FOR:

Single and Double Suzuki–Miyaura Couplings with Symmetric Dihalobenzenes

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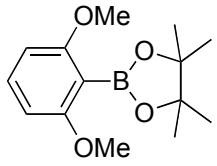
^{1}H and ^{13}C NMR spectra of compounds **4**, **5**-*meta*, **5**-*para*, **S1**, **S5**, **S13**, and **14**–**18**. S14–S35

Characterization Data

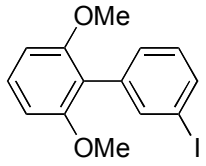
Pinacolyl 2,6-dimethoxyphenylboronate (**4**)



4

Mp 82–84 °C (hexanes); IR (film) 2977, 2936, 2836, 1599, 1584, 1466, 1430, 1378, 1344, 1307, 1244, 1146, 1107, 1058, 859, 777, 725, 678 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 (t, *J* = 8.3 Hz, 1 H), 6.46 (d, *J* = 8.4 Hz, 2 H), 3.76 (s, 6 H), 1.38 (s, 12 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4, 131.4, 103.3, 83.7, 55.7, 24.7; MS (EI) *m/z* 264 (M^+); HRMS (EI) *m/z* calcd for $C_{14}H_{21}BO_4$ (M^+) 264.1533, found 264.1532.

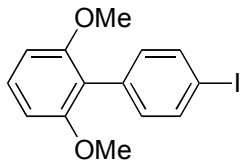
2,6-Dimethoxy-3'-iodobiphenyl (**5**-*meta*)



5-*meta*

Mp 85–86 °C (DCM/ethanol); IR (film) 2935, 2835, 1589, 1471, 1432, 1244, 1110, 788, 729, 695 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (t, *J* = 1.7 Hz, 1 H), 7.65 (ddd, *J* = 7.8, 1.7, 1.1 Hz, 1 H), 7.35–7.27 (m, 2 H), 7.15 (t, *J* = 7.8 Hz, 1 H), 6.65 (d, *J* = 8.1 Hz, 2 H), 3.75 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.5, 139.7, 136.4, 135.6, 130.3, 129.4, 129.2, 117.9, 104.1, 93.7, 55.9; MS (EI) *m/z* 340 (M^+); HRMS (EI) *m/z* calcd for $C_{14}H_{13}IO_2$ (M^+) 339.9960, found 339.9967.

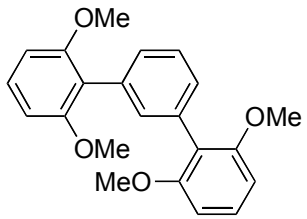
2,6-Dimethoxy-4'-iodobiphenyl (**5**-*para*)



5-*para*

Mp 129–130 °C (DCM/ethanol); IR (film) 2988, 2937, 2833, 1588, 1469, 1431, 1249, 1110, 999, 820, 779, 729, 701 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2 H), 7.29 (t, *J* = 8.3 Hz, 1 H), 7.11 (d, *J* = 8.4 Hz, 2 H), 6.65 (d, *J* = 8.1 Hz, 2 H), 3.74 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.5, 136.8, 133.7, 133.0, 129.0, 118.3, 104.2, 92.6, 55.9; MS (EI) *m/z* 340 (M^+); Anal. Calcd for $C_{14}H_{13}IO_2$: C 49.4%, H 3.9. Found: C 49.7, H 4.0.

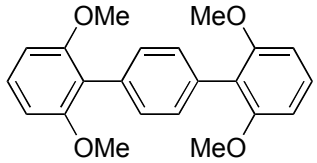
2,2",6,6"-Tetramethoxy-1,1':3',1"-terphenyl (**6**-*meta*)



6-*meta*

Grewal, R. S.; Hart, H.; Vinod, T. K. *J. Org. Chem.* **1992**, *57*, 2721–2726.

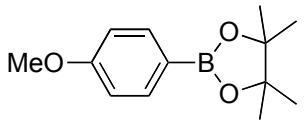
2,2",6,6"-Tetramethoxy-1,1':4',1"-terphenyl (**6**-*para*)



6-*para*

Kern, W.; Ebersbach, H. W.; Ziegler, I. *Makromolekulare Chemie* **1959**, *31*, 154–180.

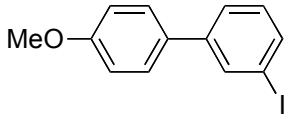
Pinacolyl 4-methoxyphenylboronate (**7**)



7

Ishiyama, T.; Murata, M.; Miyaura, N. *J. Org. Chem.* **1995**, *60*, 7508–7510.

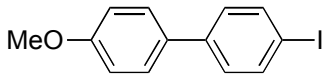
3-Iodo-4'-methoxybiphenyl (**S1**; Table 2, entry 3, monocoupled product)



S1

Mp 111–112 °C (DCM/ethanol); IR (film) 2959, 2931, 2836, 1607, 1554, 1518, 1251, 1037, 1018, 838, 781, 684 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 (t, *J* = 1.7 Hz, 1 H), 7.63 (ddd, *J* = 8.0, 1.7, 1.2 Hz, 1 H), 7.52–7.46 (m, 3 H), 7.14 (t, *J* = 7.8 Hz, 1 H), 6.98 (d, *J* = 9.0 Hz, 2 H), 3.85 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.6, 143.1, 135.7, 135.5, 132.1, 130.4, 128.2, 126.0, 114.3, 94.8, 55.4; MS (EI) *m/z* 310 (M^+); Anal. Calcd for $C_{13}H_{11}IO$: C 50.4%, H 3.6. Found: C 50.2, H 3.8.

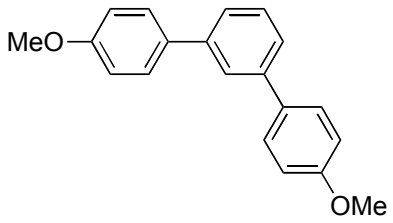
4-Iodo-4'-methoxybiphenyl (**S2**; Table 2, entry 4, monocoupled product)



S2

Spivey, A. C.; Diaper, C. M.; Adams, H.; Rudge, A. J. *J. Org. Chem.* **2000**, *65*, 5253–5263.

4,4"-Dimethoxy-1,1':3',1"-terphenyl (**S3**; Table 2, entry 3, biscoupled product)

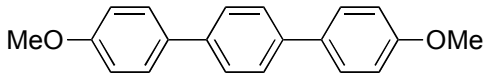


S3

* Commercially available

Mueller, G. P.; Honaker, C. B. *J. Am. Chem. Soc.* **1951**, *73*, 2377–2379.

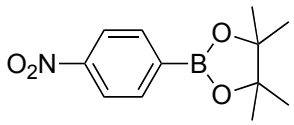
4,4"-Dimethoxy-1,1':4',1"-terphenyl (**S4**; Table 2, entry 4, biscoupled product)



S4

Tao, X.; Zhao, Y.; Shen, D. *Synlett* **2002**, 757–760.

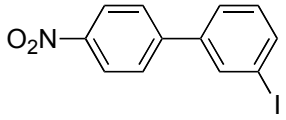
Pinacolyl 4-nitrophenylboronate (**8**)



8

* Commercially available

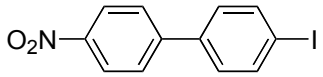
3-Iodo-4'-nitrobiphenyl (**S5**; Table 2, entry 5, monocoupled product)



S5

Mp 125–127 °C (DCM/ethanol); IR (film) 2917, 2849, 1597, 1557, 1513, 1468, 1344, 1108, 854, 782, 768, 748, 687 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.30 (d, *J* = 9.0 Hz, 2 H), 7.96 (t, *J* = 1.8 Hz, 1 H), 7.78 (dt, *J* = 8.1, 1.4 Hz, 1 H), 7.70 (d, *J* = 9.0 Hz, 2 H), 7.58 (ddd, *J* = 7.8, 1.8, 0.9 Hz, 1 H), 7.23 (t, *J* = 8.0 Hz, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.4, 145.9, 140.9, 137.8, 136.3, 130.8, 127.9, 126.6, 124.2, 95.0; MS (EI) *m*/*z* 325 (M^+); HRMS (EI) *m*/*z* calcd for $C_{12}H_8INO_2$ (M^+) 324.9600, found 324.9608.

4-Iodo-4'-nitrobiphenyl (**S6**; Table 2, entry 6, monocoupled product)

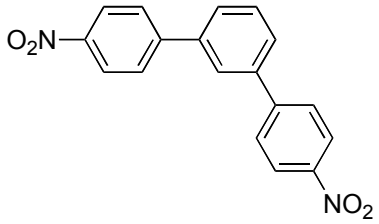


S6

* Commercially available

1) Sarma, J. A. R. P.; Allen, F. H.; Hoy, V. J.; Howard, J. A. K.; Thaimattam, R.; Kumar, B.; Desiraju, G. R. *Chem. Commun.* **1997**, 101–102. 2) Harley-Mason, J.; Mann, F. G. *J. Chem. Soc.* **1940**, 1374–1385.

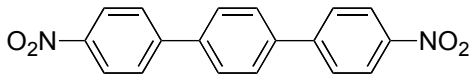
4,4"-Dinitro-1,1':3',1"-terphenyl (**S7**; Table 2, entry 5, biscoupled product)



S7

Descotes, G.; Praly, J. P. *Bull. Soc. Chim. Fr.* **1976**, 901–910.

4,4"-Dinitro-1,1':4',1"-terphenyl (**S8**; Table 2, entry 6, biscoupled product)

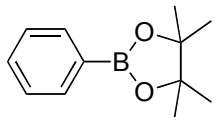


S8

* Commercially available

Chaumeil, H.; Le Drian, C.; Defoin, A. *Synthesis* **2002**, 757–760.

Pinacolyl phenylboronate (**9**)

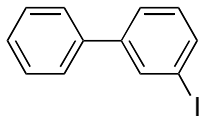


9

* Commercially available

Ishiyama, T.; Murata, M.; Miyaura, N. *J. Org. Chem.* **1995**, *60*, 7508–7510.

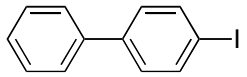
3-Iodobiphenyl (**S9**; Table 2, entry 7, monocoupled product)



S9

Dektar, J. H.; Hacker, N. P. *J. Org. Chem.* **1990**, *55*, 639–647.

4-Iodobiphenyl (**S10**; Table 2, entry 8, monocoupled product)

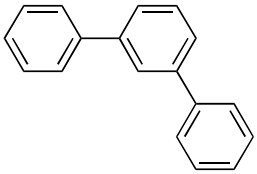


S10

* Commercially available

Dektar, J. H.; Hacker, N. P. *J. Org. Chem.* **1990**, *55*, 639–647.

1,1':3',1"-Terphenyl (**S11**; Table 2, entry 7, biscoupled product)

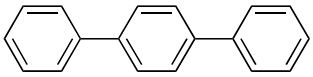


S11

* Commercially available

Asao, N.; Nogami, T.; Lee, S.; Yamamoto, Y. *J. Am. Chem. Soc.* **2003**, *125*, 10921–10925.

1,1':4',1"-Terphenyl (**S12**; Table 2, entry 8, biscoupled product)

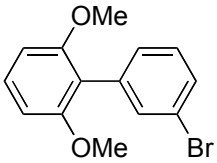


S12

* Commercially available

Voronkov, M. G.; Deryagina, E. N.; Sukhomazova, E. N.; Klochkova, L. G. *Zhurnal Organicheskoi Khimii* **1975**, *11*, 1131.

3-Bromo-2',6'-dimethoxybiphenyl (**S13**; Table 3, entry 4, monocoupled product)

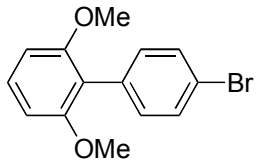


S13

Mp 79–80 °C (DCM/ethanol); IR (film) 2937, 2835, 1589, 1472, 1242, 1111, 790, 729, 694, 658 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 (dd, J = 2.1, 1.2 Hz, 1 H), 7.45 (ddd, J = 6.0, 3.0, 2.0 Hz, 1 H), 7.33–7.28 (m, 3 H), 6.66 (d, J = 8.4 Hz, 2 H), 3.75 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.5,

136.3, 133.9, 129.7, 129.7, 129.2, 129.1, 121.7, 118.0, 104.1, 55.9; MS (EI) *m/z* 292/294 (M^+); Anal. Calcd for $C_{14}H_{13}BrO_2$: C 57.4%, H 4.5. Found: C 57.3, H 4.4.

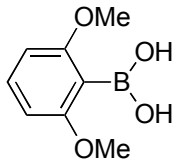
4-Bromo-2',6'-dimethoxybiphenyl (**S14**; Table 3, entry 5, monocoupled product)



S14

von dem Bussche-Huennefeld, C.; Helgeson, R. C.; Buerhing, D.; Knobler, C. B.; Cram, D. J. *Croat. Chim. Acta* **1996**, *69*, 447–458.

2,6-Dimethoxyphenylboronic acid (**10**)

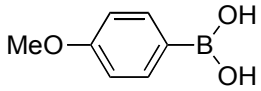


10

* Commercially available

von dem Bussche-Huennefeld, C.; Helgeson, R. C.; Buerhing, D.; Knobler, C. B.; Cram, D. J. *Croat. Chim. Acta* **1996**, *69*, 447–458.

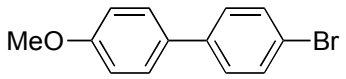
4-Methoxyphenylboronic acid (**11**)



11

* Commercially available

4-Bromo-4'-methoxybiphenyl (**S15**; Table 3, entry 10, monocoupled product)

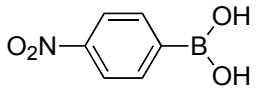


S15

* Commercially available

Spivey, A. C.; Diaper, C. M.; Adams, H.; Rudge, A. J. *J. Org. Chem.* **2000**, *65*, 5253–5263.

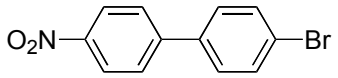
4-Nitrophenylboronic acid (**12**)



12

* Commercially available

4-Bromo-4'-nitrobiphenyl (**S16**; Table 3, entry 12, monocoupled product)

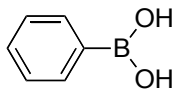


S16

* Commercially available

Kim, Y.; Han, K.; Ha, C.-S. *Macromolecules* **2002**, *35*, 8759–8767.

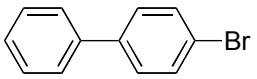
Phenylboronic acid (**13**)



13

* Commercially available

4-Bromobiphenyl (**S17**; Table 3, entry 13, monocoupled product)

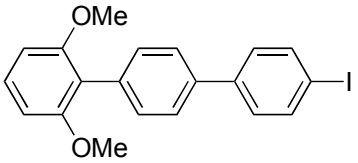


S17

* Commercially available

Berthiol, F.; Kondolff, I.; Doucet, H.; Santelli, M. *J. Organomet. Chem.* **2004**, *689*, 2786–2798.

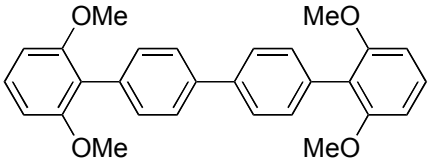
2,6-Dimethoxy-4"-iodo-1,1':4',1"-terphenyl (**14**)



14

Mp 157–158 °C (DCM/ethanol); IR (film) 2932, 2835, 1588, 1470, 1245, 1105, 1001, 814, 735 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.4 Hz, 2 H), 7.60 (d, *J* = 8.4 Hz, 2 H), 7.45 (d, *J* = 8.4 Hz, 2 H), 7.40 (d, *J* = 8.4 Hz, 2 H), 7.31 (t, *J* = 8.6 Hz, 1 H), 6.69 (d, *J* = 8.4 Hz, 2 H), 3.77 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.7, 140.8, 138.2, 137.8, 133.7, 131.5, 129.0, 128.8, 126.2, 118.8, 104.2, 92.77, 56.0; MS (EI) *m/z* 416 (M^+); HRMS (EI) *m/z* calcd for $C_{20}H_{17}IO_2$ (M^+) 416.0273, found 416.0274.

2,2'",6,6'"-Tetramethoxy-1,1':4',1":4",1'"-quaterphenyl (**15**)

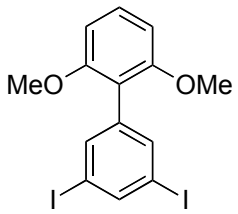


15

Mp 285 °C (DCM/ethanol); IR (film) 2989, 2936, 2835, 1586, 1468, 1431, 1247, 1110, 821, 782, 732 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.1 Hz, 4 H), 7.45 (d, *J* = 8.4 Hz, 4 H), 7.30 (t, *J* = 8.4

Hz, 2 H), 6.69 (d, J = 8.4 Hz, 4 H), 3.78 (s, 12 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.8, 139.6, 132.8, 131.2, 128.7, 126.5, 119.2, 104.3, 56.0; MS (EI) m/z 426 (M^+); Anal. Calcd for $C_{28}H_{26}O_4$: C 78.9%, H 6.1. Found: C 78.7, H 6.4.

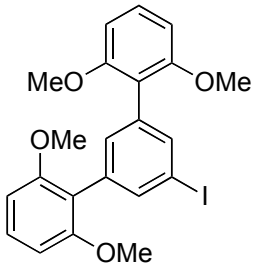
3,5-Diiodo-2',6'-dimethoxybiphenyl (**16**)



16

Mp 170–172 °C (DCM/ethanol); IR (film) 2917, 2835, 1590, 1532, 1472, 1428, 1396, 1249, 1236, 1108, 846, 778, 700 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (t, J = 1.7 Hz, 1 H), 7.64 (d, J = 1.5 Hz, 2 H), 7.29 (t, J = 8.6 Hz, 1 H), 6.62 (d, J = 8.7 Hz, 2 H), 3.75 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.4, 143.1, 139.2, 138.0, 129.7, 116.4, 104.0, 93.9, 55.9; MS (EI) m/z 466 (M^+); HRMS (EI) m/z calcd for $C_{14}H_{12}I_2O_2$ (M^+) 465.8927, found 465.8936.

5'-Iodo-2,2",6,6"-tetramethoxy-1,1':3',1"-terphenyl (**17**)

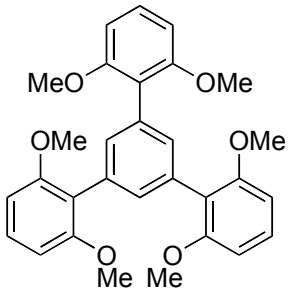


17

Mp 220–222 °C (DCM/ethanol); IR (film) 2936, 2837, 1589, 1472, 1433, 1416, 1249, 1112, 784, 731, 702 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (d, J = 1.2 Hz, 2 H), 7.35 (t, J = 1.2 Hz, 1 H), 7.27 (t, J = 8.6 Hz, 2 H), 6.64 (d, J = 8.4 Hz, 4 H), 3.75 (s, 12 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.7, 138.0,

134.9, 133.4, 128.9, 118.3, 104.2, 92.7, 55.9; MS (EI) *m*/*z* 476 (M^+); HRMS (EI) *m*/*z* calcd for $C_{22}H_{21}IO_4$ (M^+) 476.0485, found 476.0485.

5'-(2,6-dimethoxyphenyl)-2,2",6,6"-tetramethoxy-1,1':3',1"-terphenyl (**18**)



18

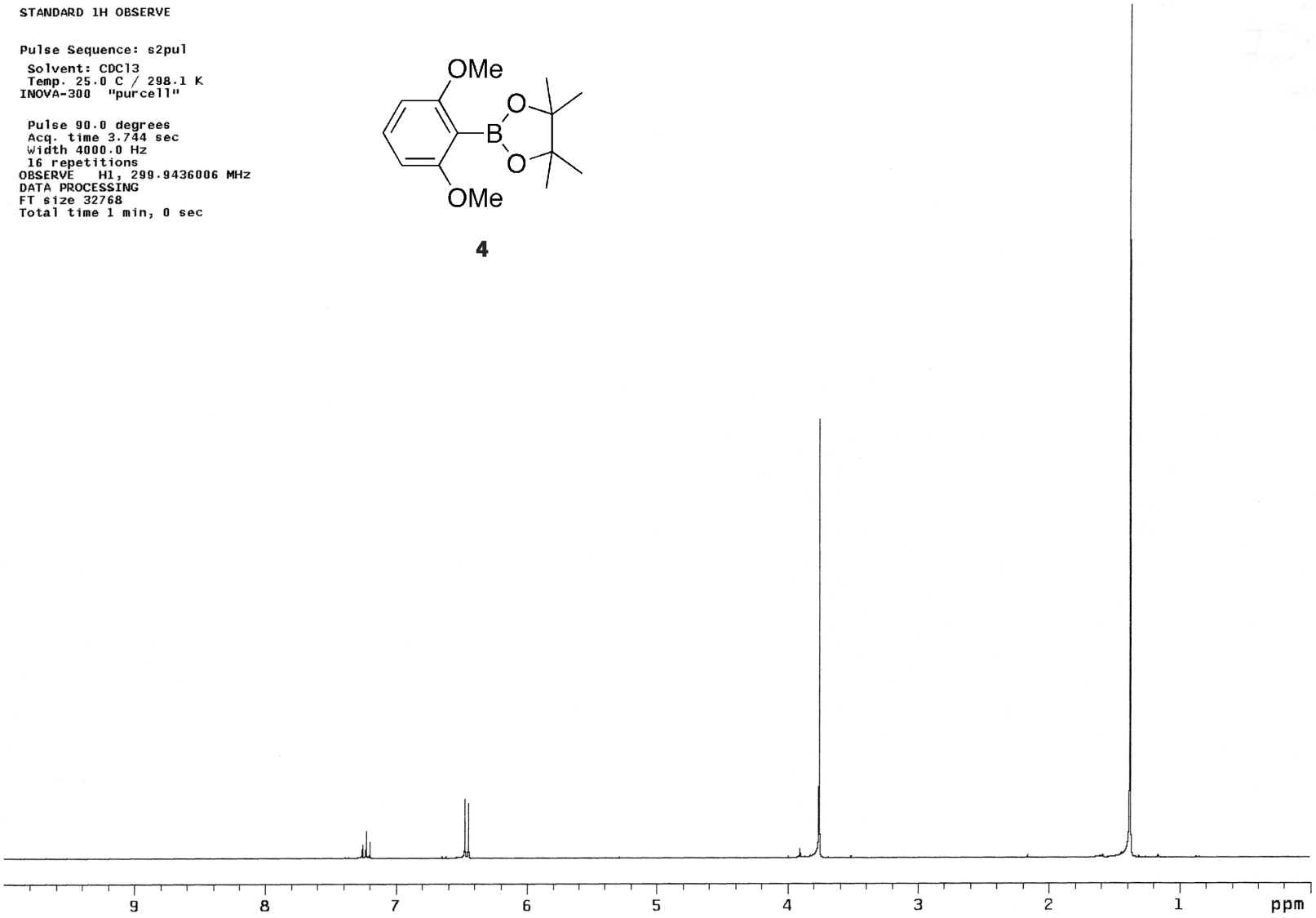
Mp 253–254 °C (DCM/ethanol); IR (film) 2936, 2836, 1599, 1586, 1471, 1431, 1414, 1248, 1236, 1111, 779, 729, 707 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.41 (s, 3 H), 7.24 (t, *J* = 8.3 Hz, 3 H), 6.66 (d, *J* = 8.4 Hz, 6 H), 3.73 (s, 18 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.0, 132.5, 131.4, 128.0, 120.4, 104.6, 56.0; MS (EI) *m*/*z* 486 (M^+); HRMS (EI) *m*/*z* calcd for $C_{30}H_{30}O_6$ (M^+) 486.2042, found 486.2047.

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436006 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

4

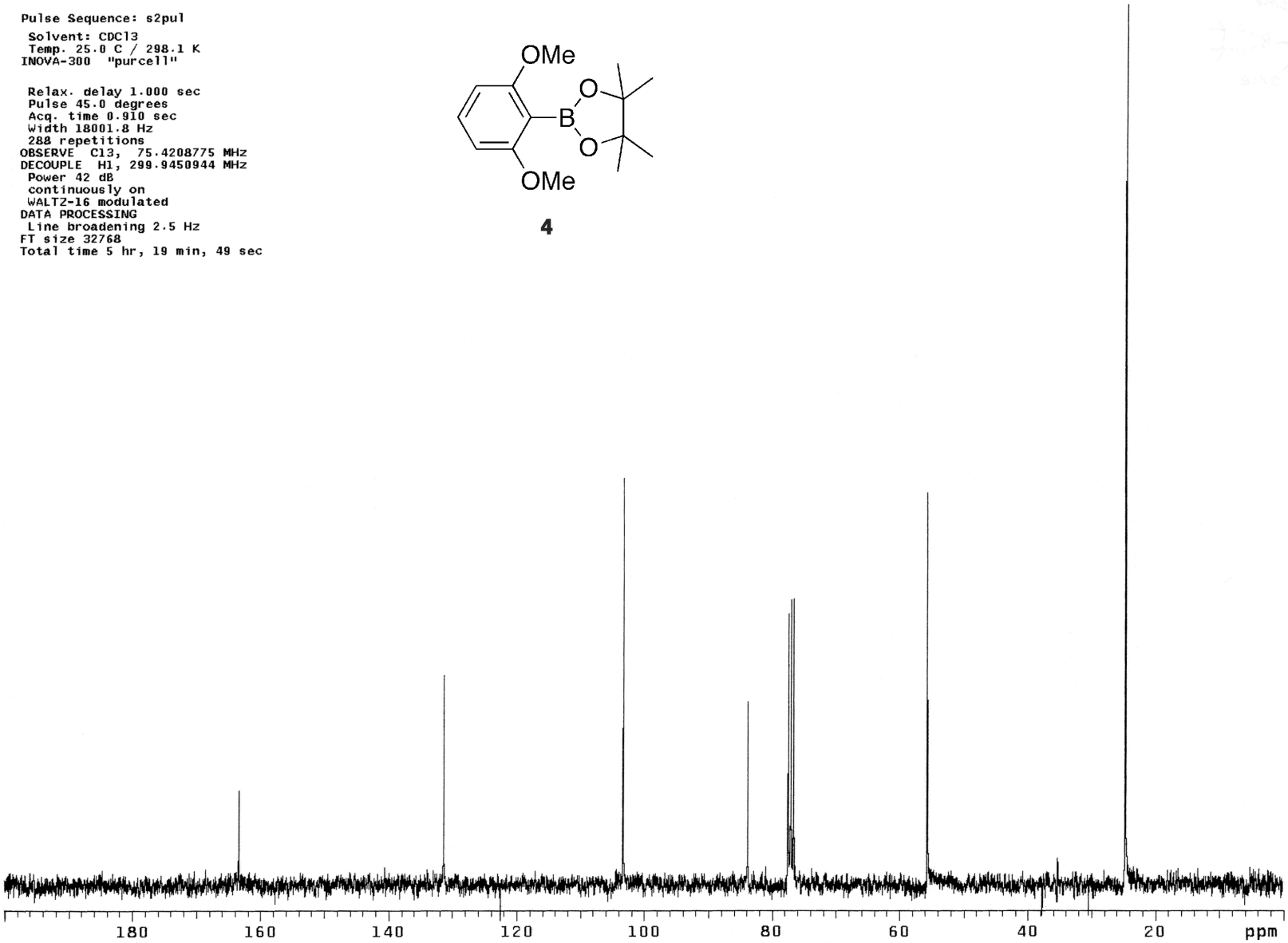


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
288 repetitions
OBSERVE C13, 75.4208775 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

4

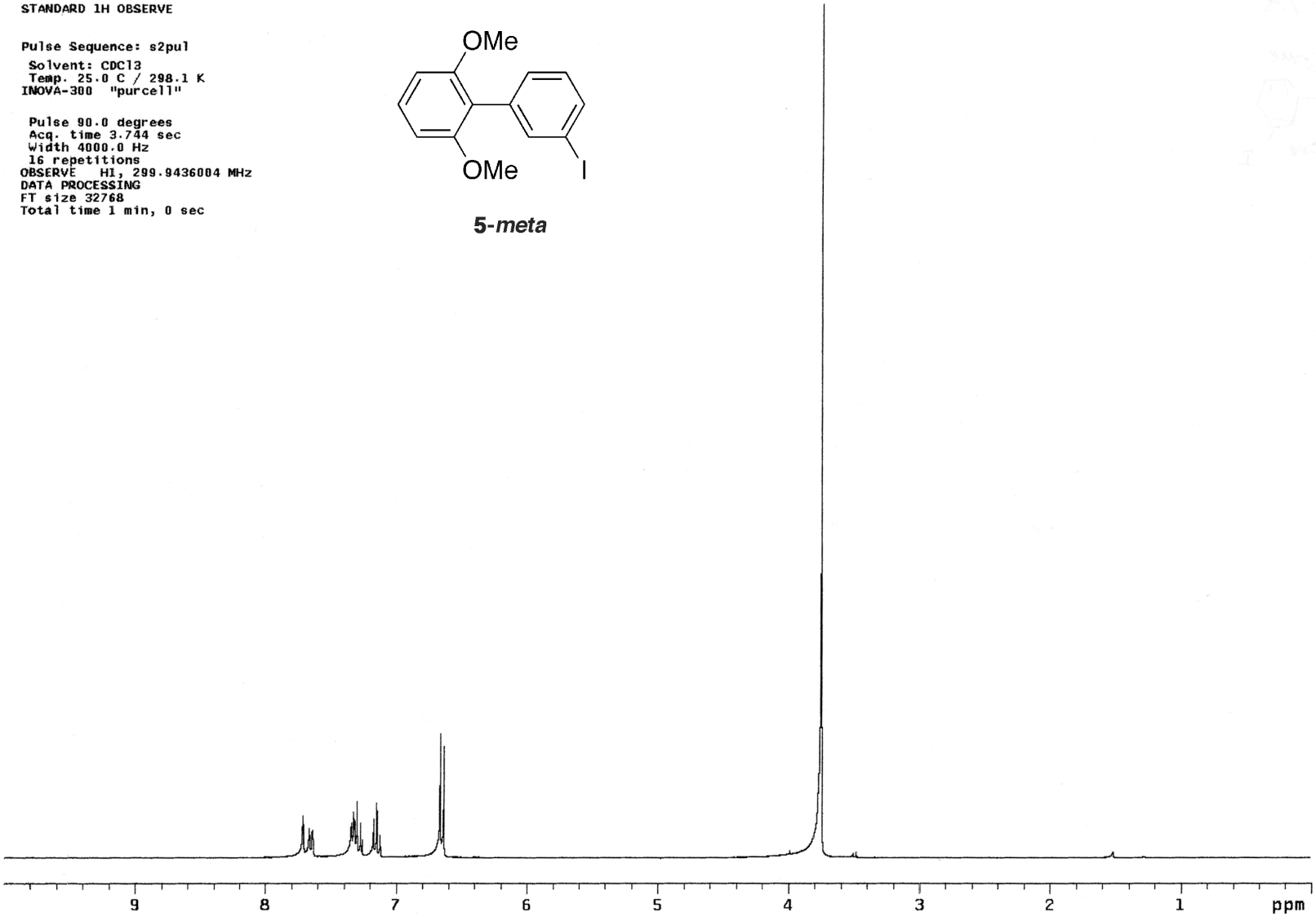


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436004 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

5-*meta*

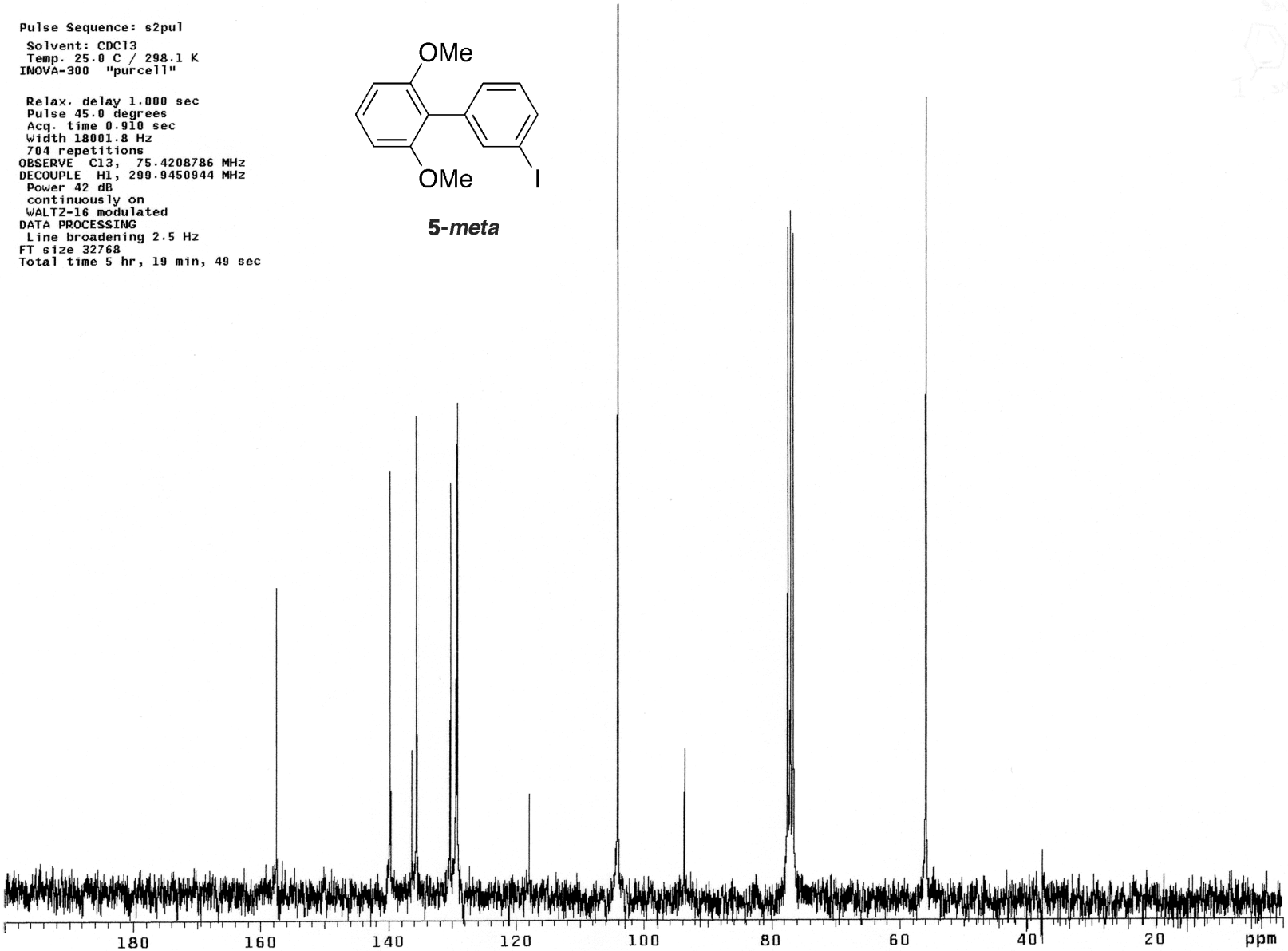


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
704 repetitions
OBSERVE C13, 75.4208786 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

5-*meta*

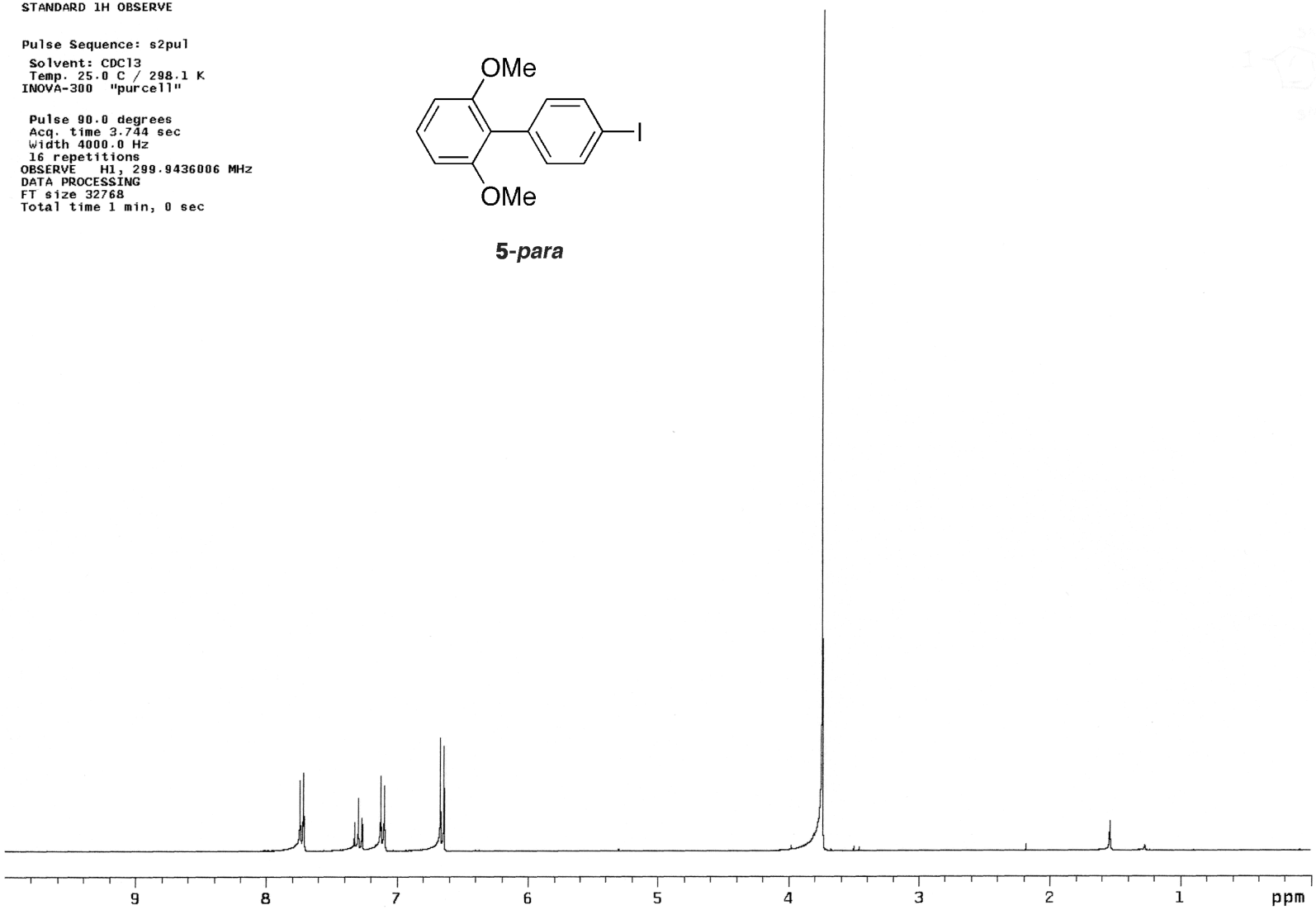


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436006 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

5-*para*

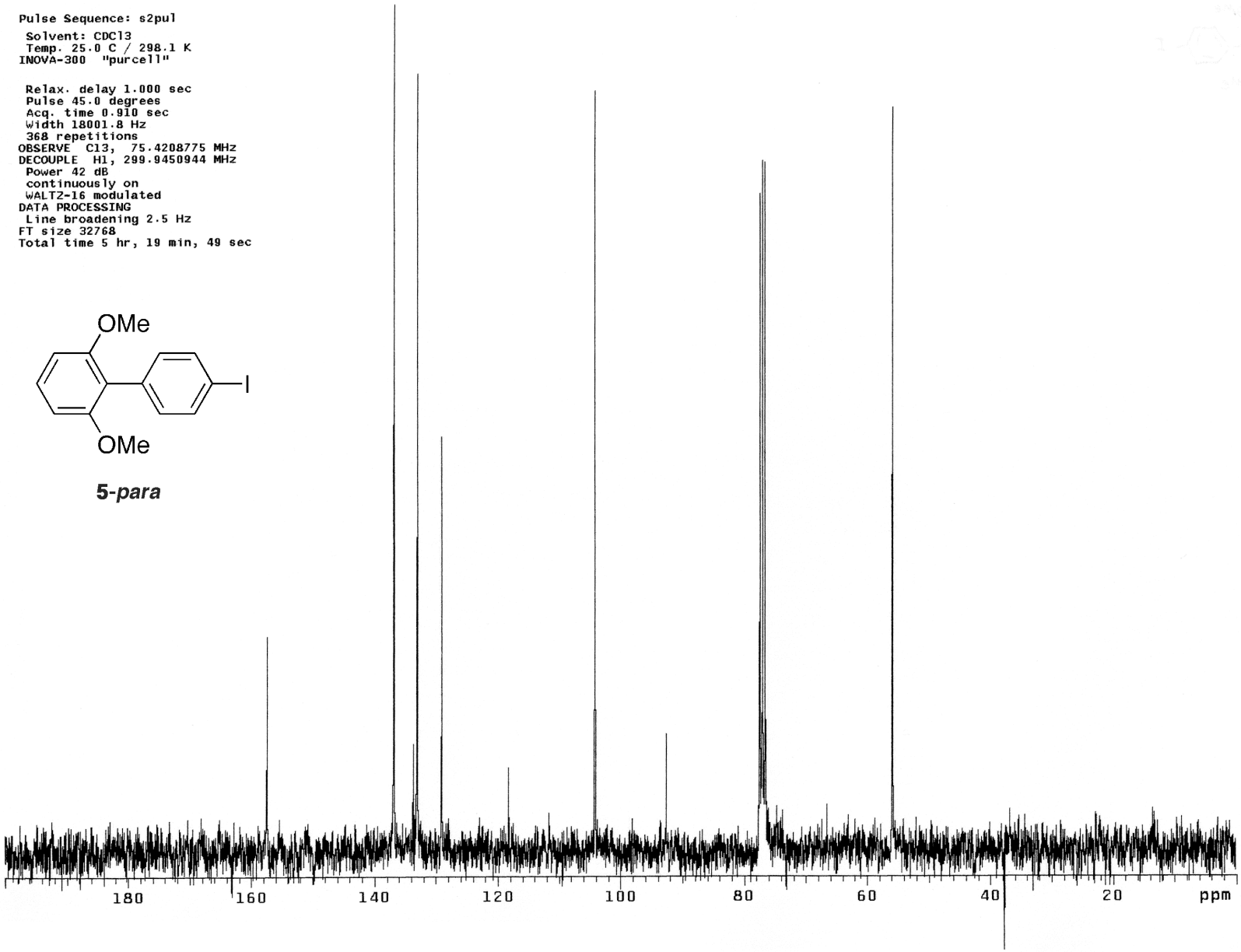


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
368 repetitions
OBSERVE C13, 75.4208775 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

5-*para*

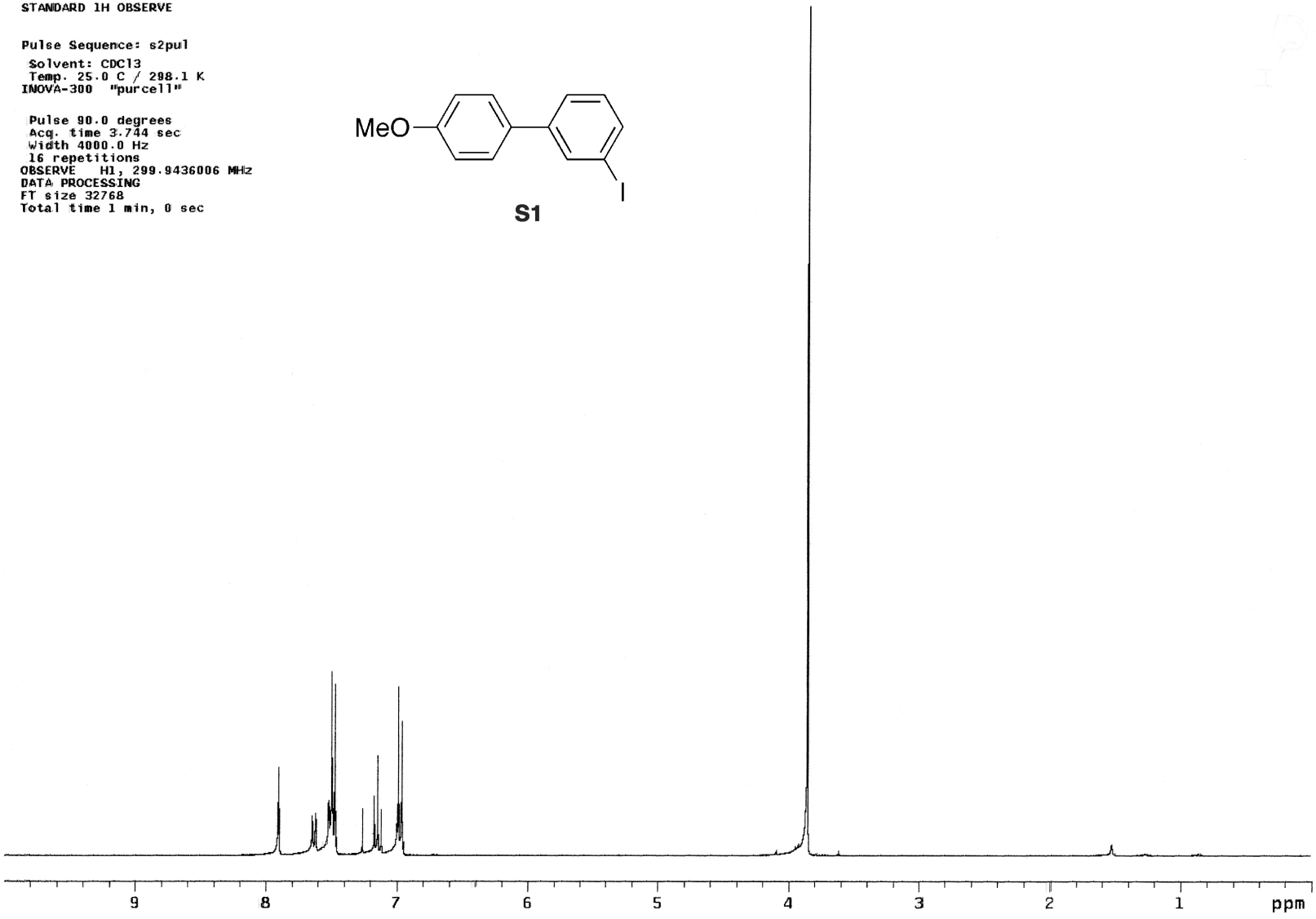


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436006 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

S1

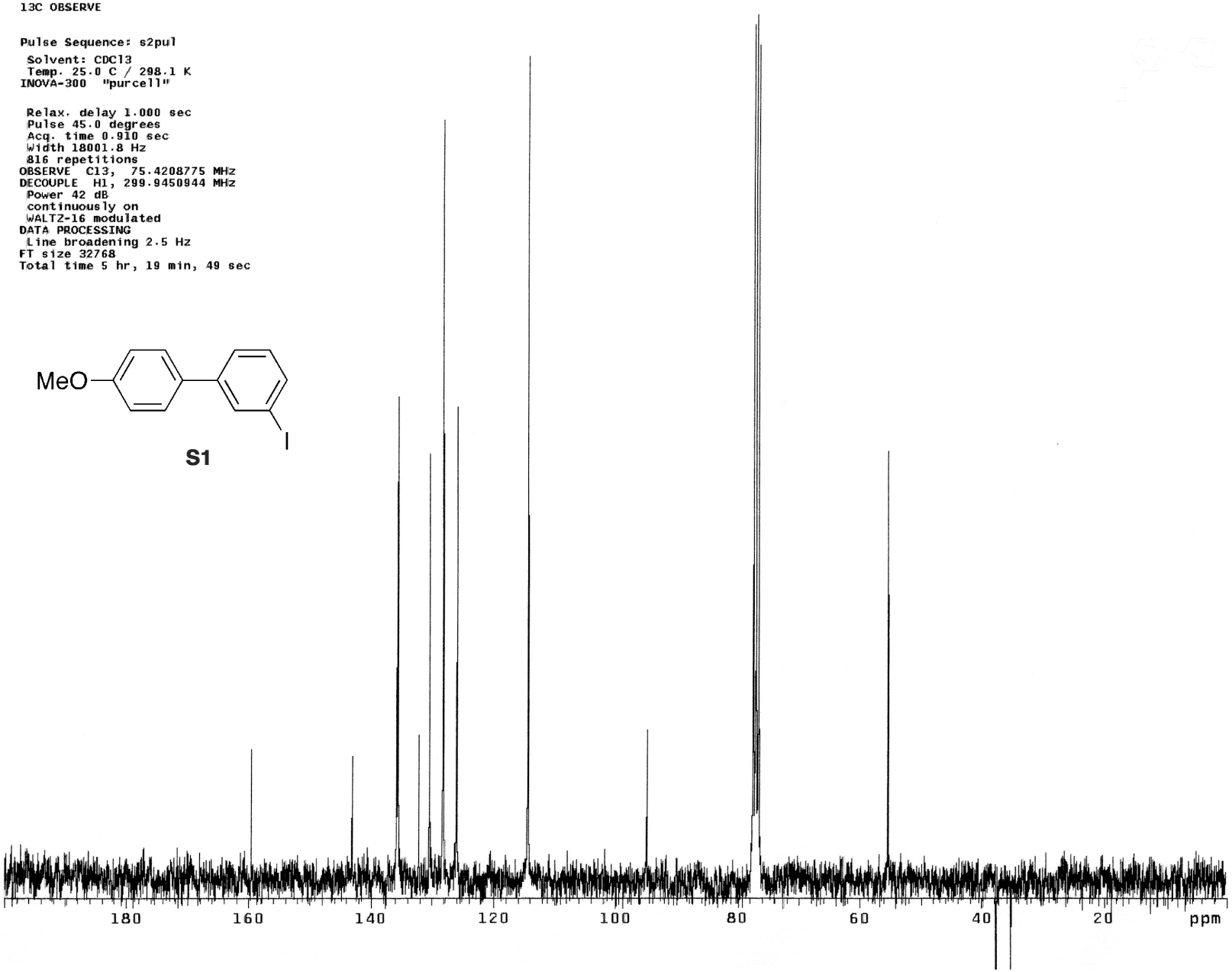


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
816 repetitions
OBSERVE C13, 75.4208775 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

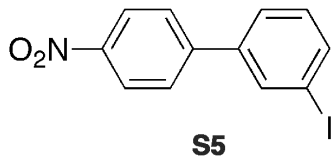
S1



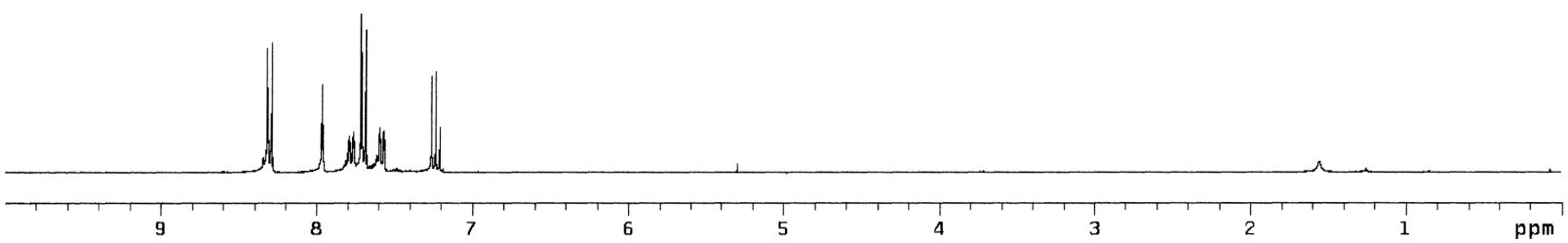
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436006 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec



S5

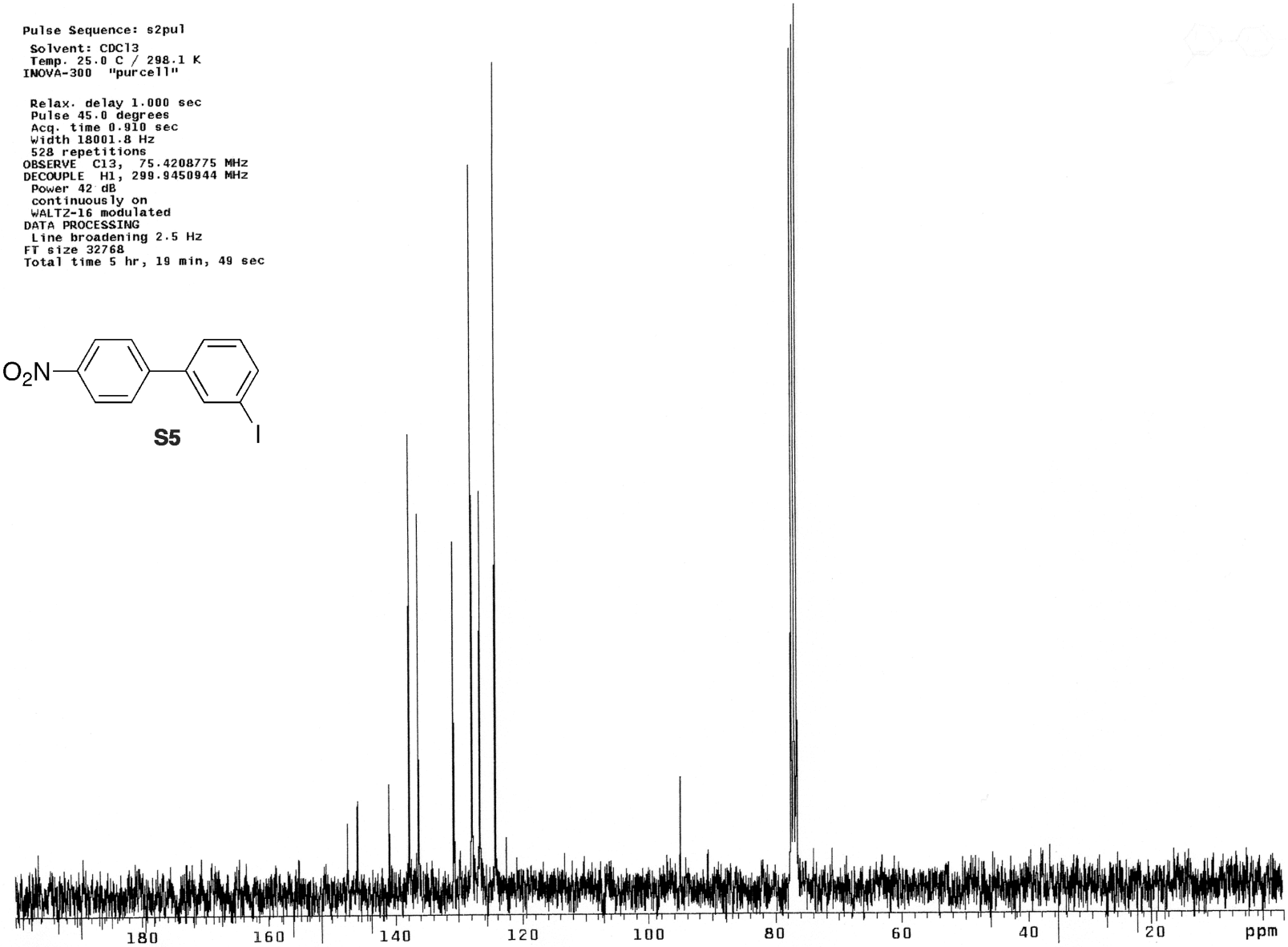


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
528 repetitions
OBSERVE C13, 75.4208775 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

S5

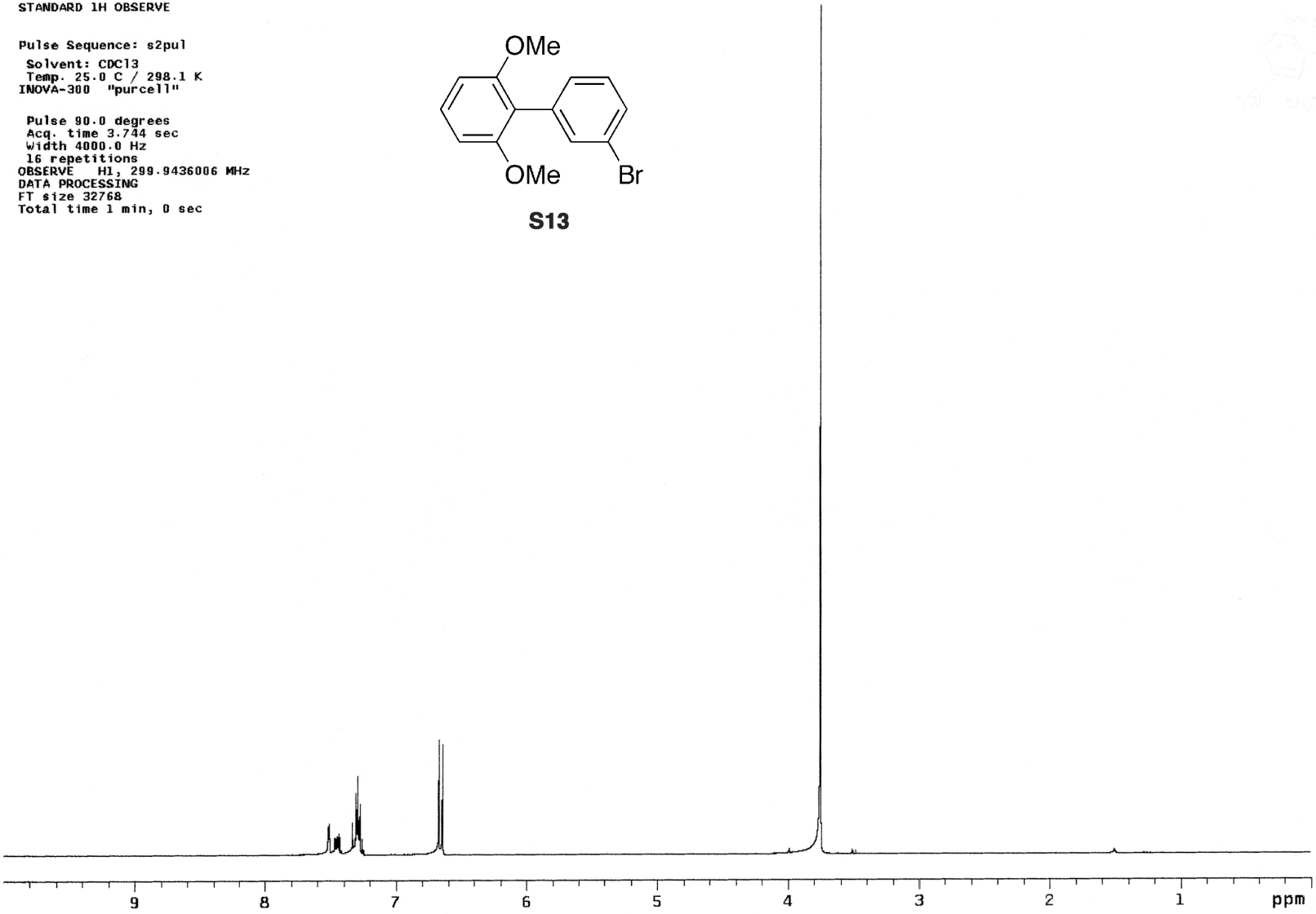


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436006 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

S13

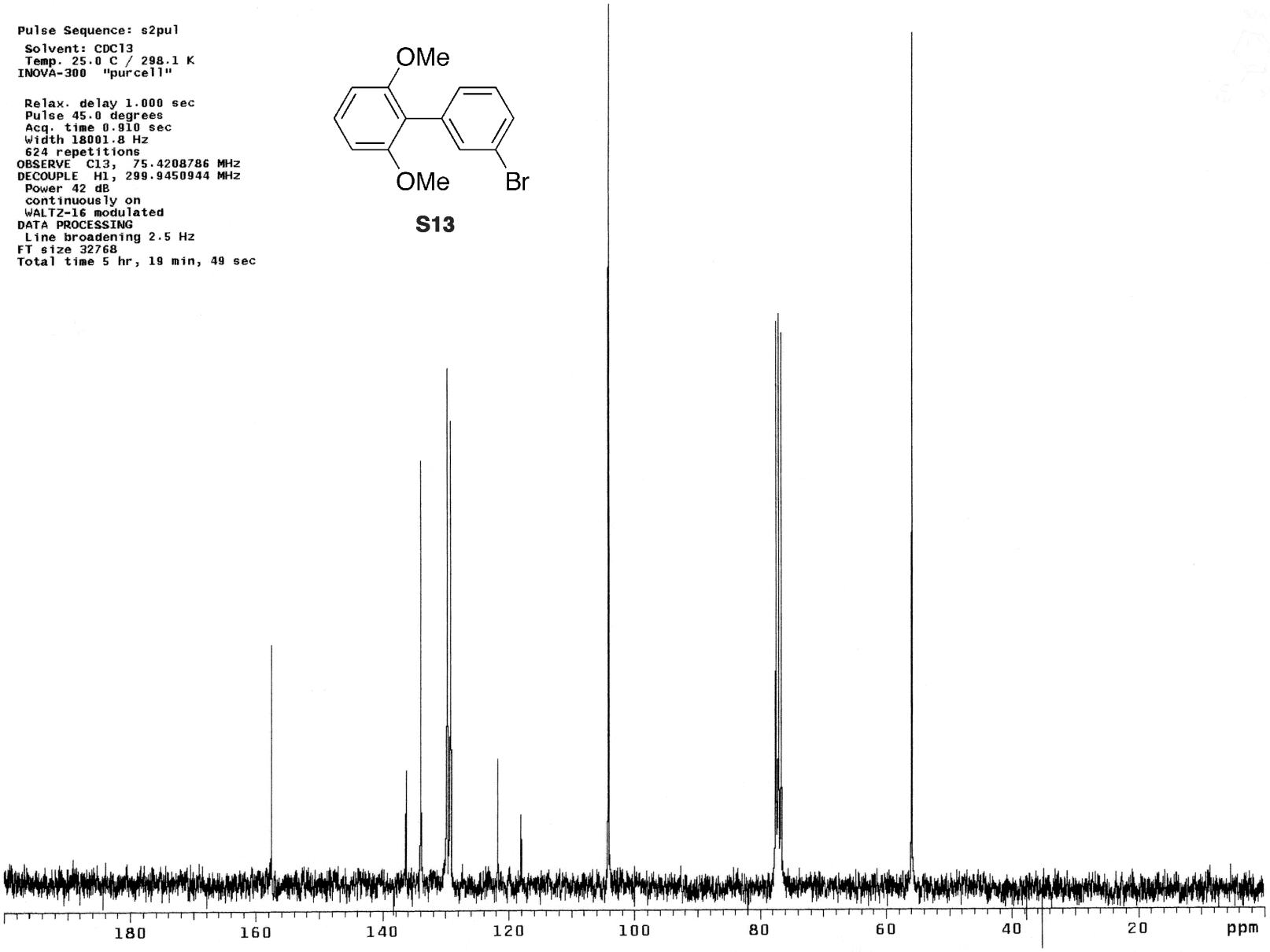


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
624 repetitions
OBSERVE C13, 75.4208786 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

S13

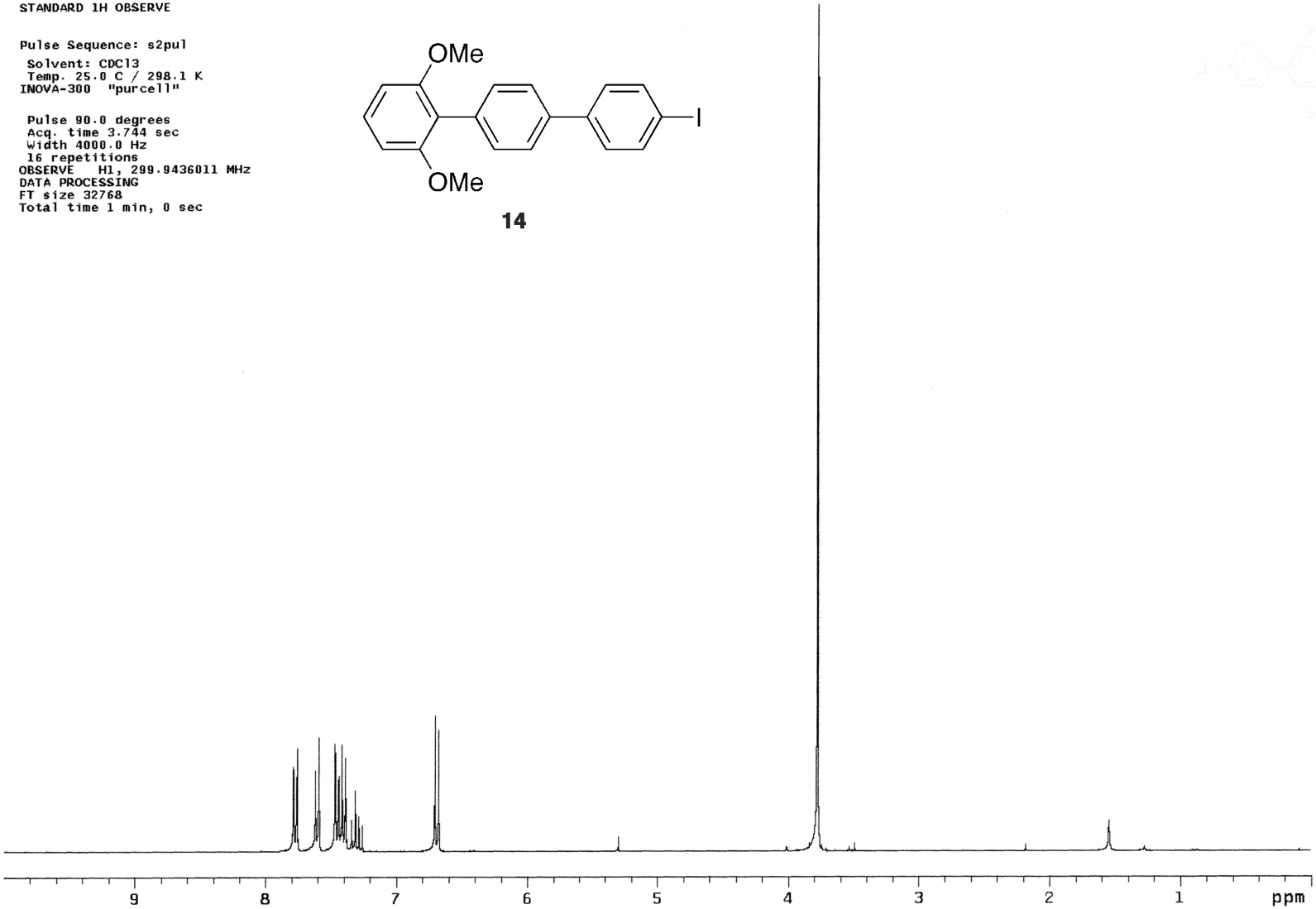


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436011 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

14

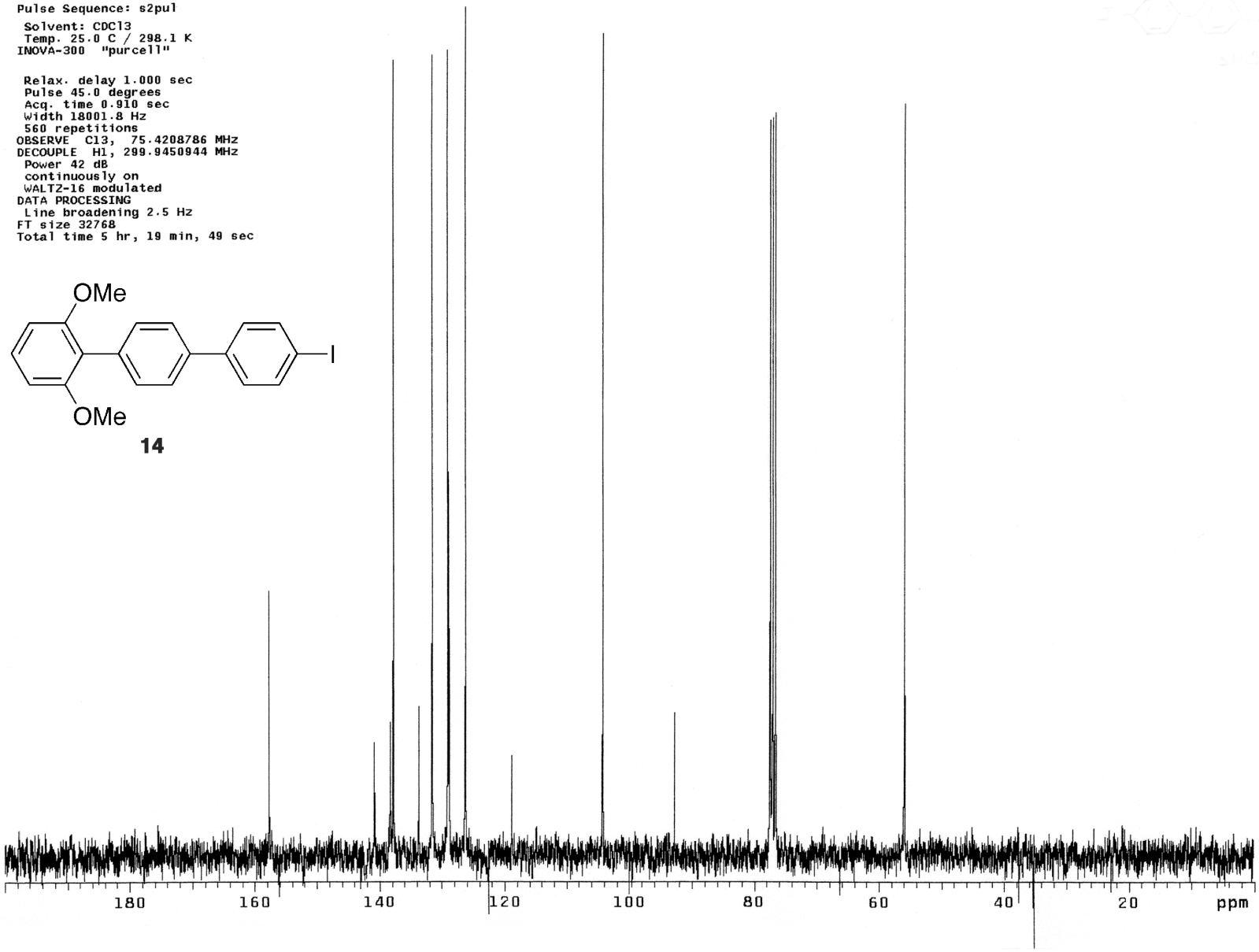


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
560 repetitions
OBSERVE C13, 75.4208786 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

14

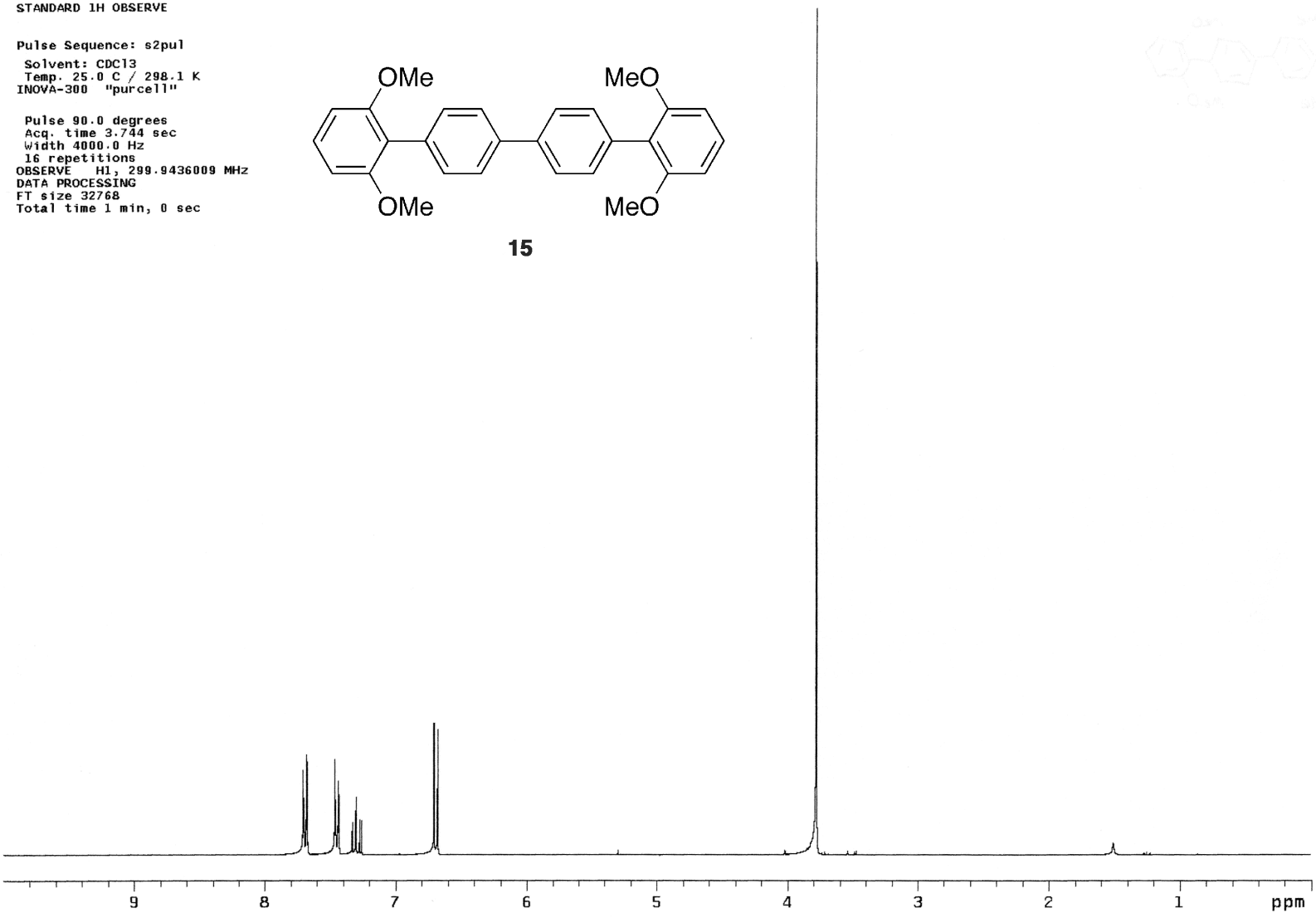


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436009 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

15

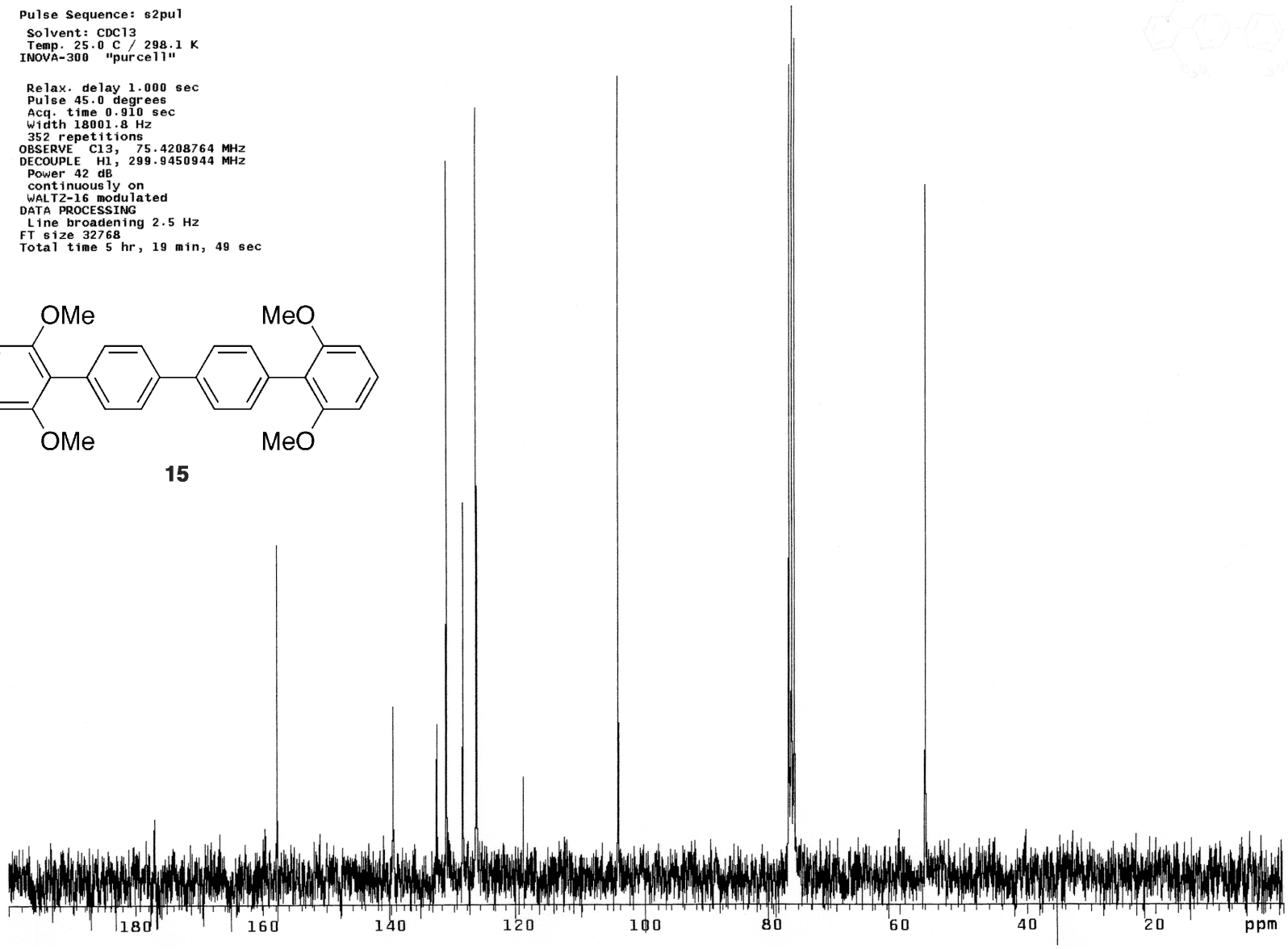


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
352 repetitions
OBSERVE C13, 75.4208764 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

15

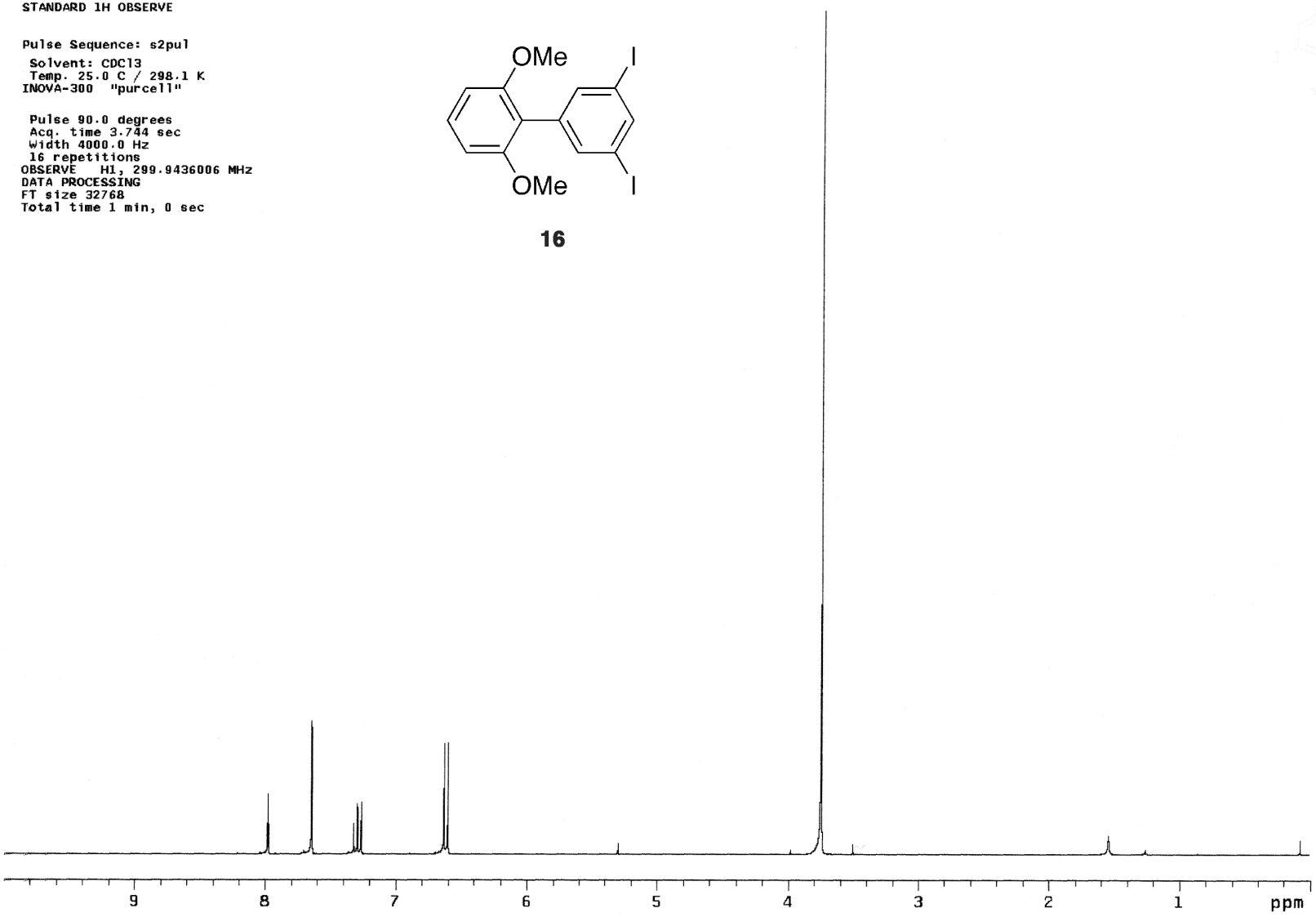


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436006 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

16

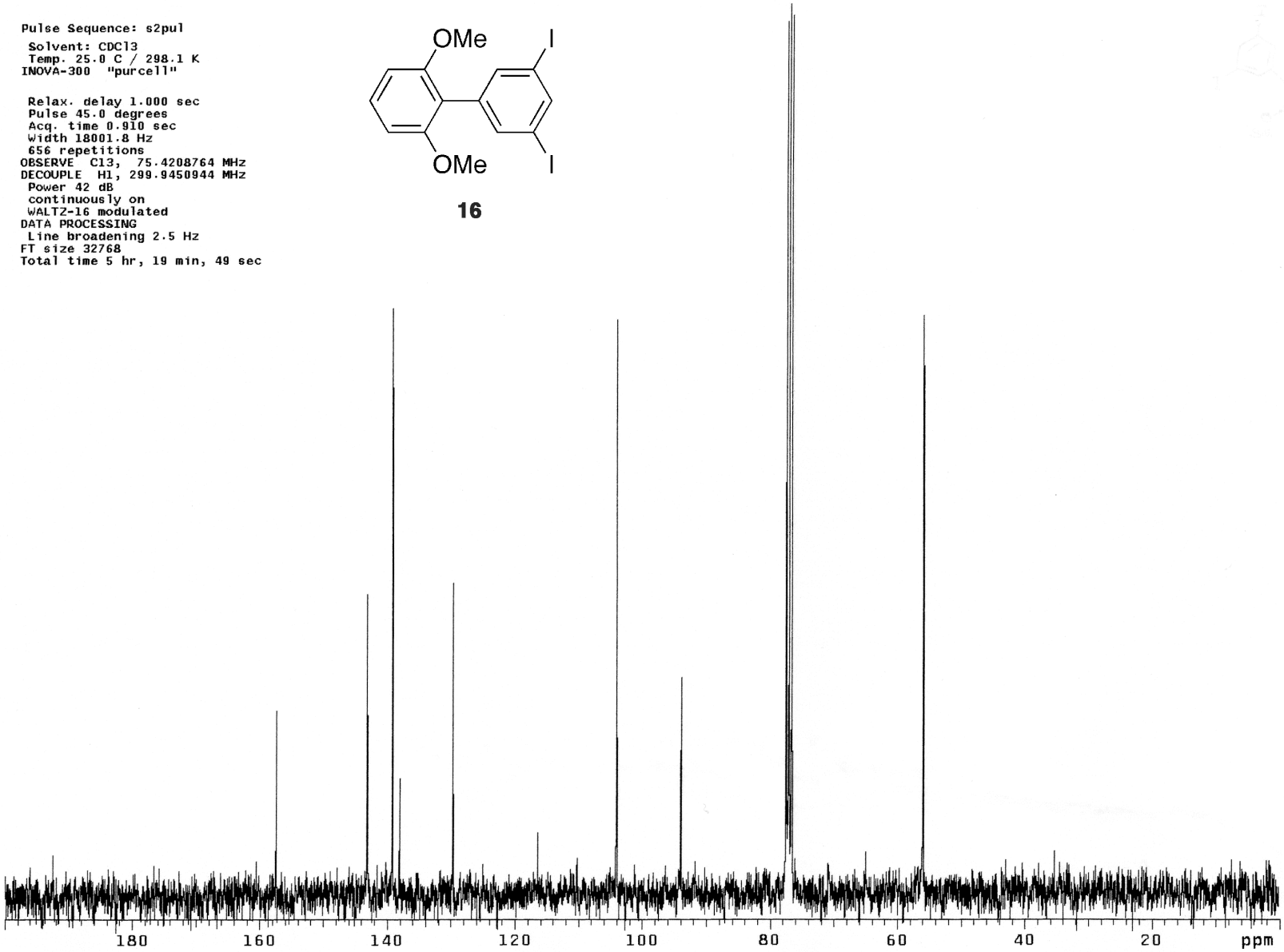


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
656 repetitions
OBSERVE C13, 75.4208764 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

16

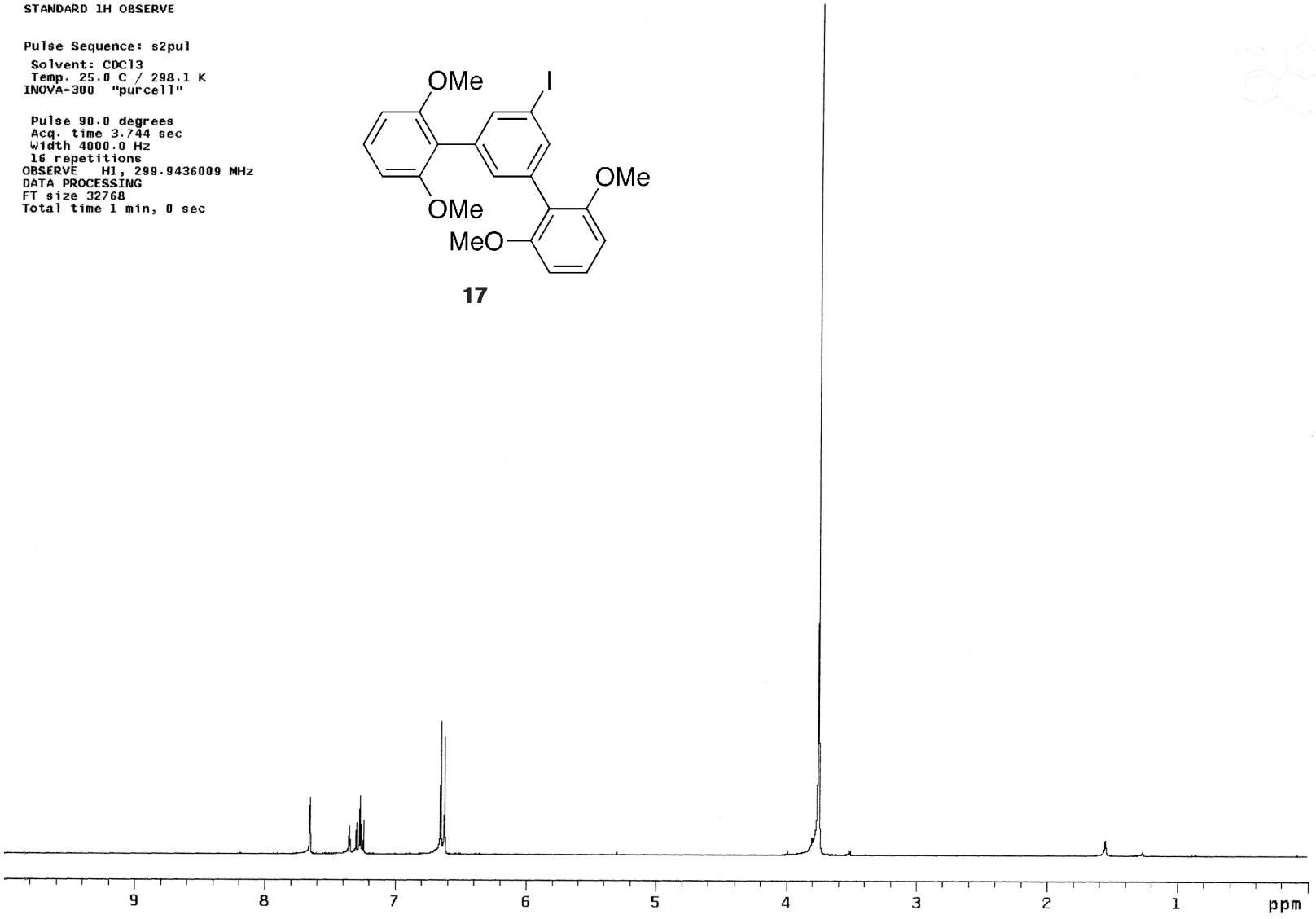


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436009 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

17

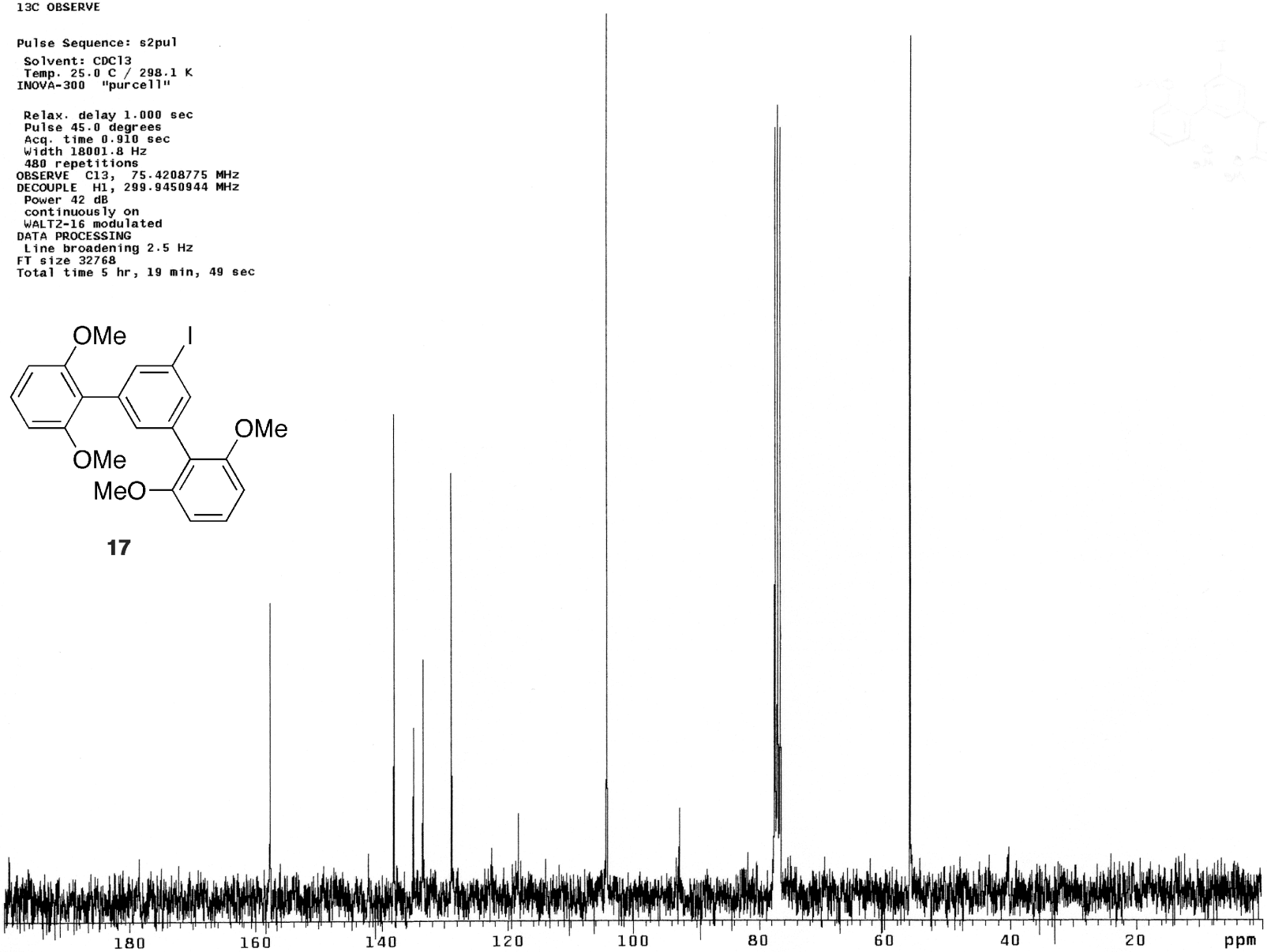


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
480 repetitions
OBSERVE C13, 75.4208775 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

17



STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Pulse 90.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 299.9436009 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 0 sec

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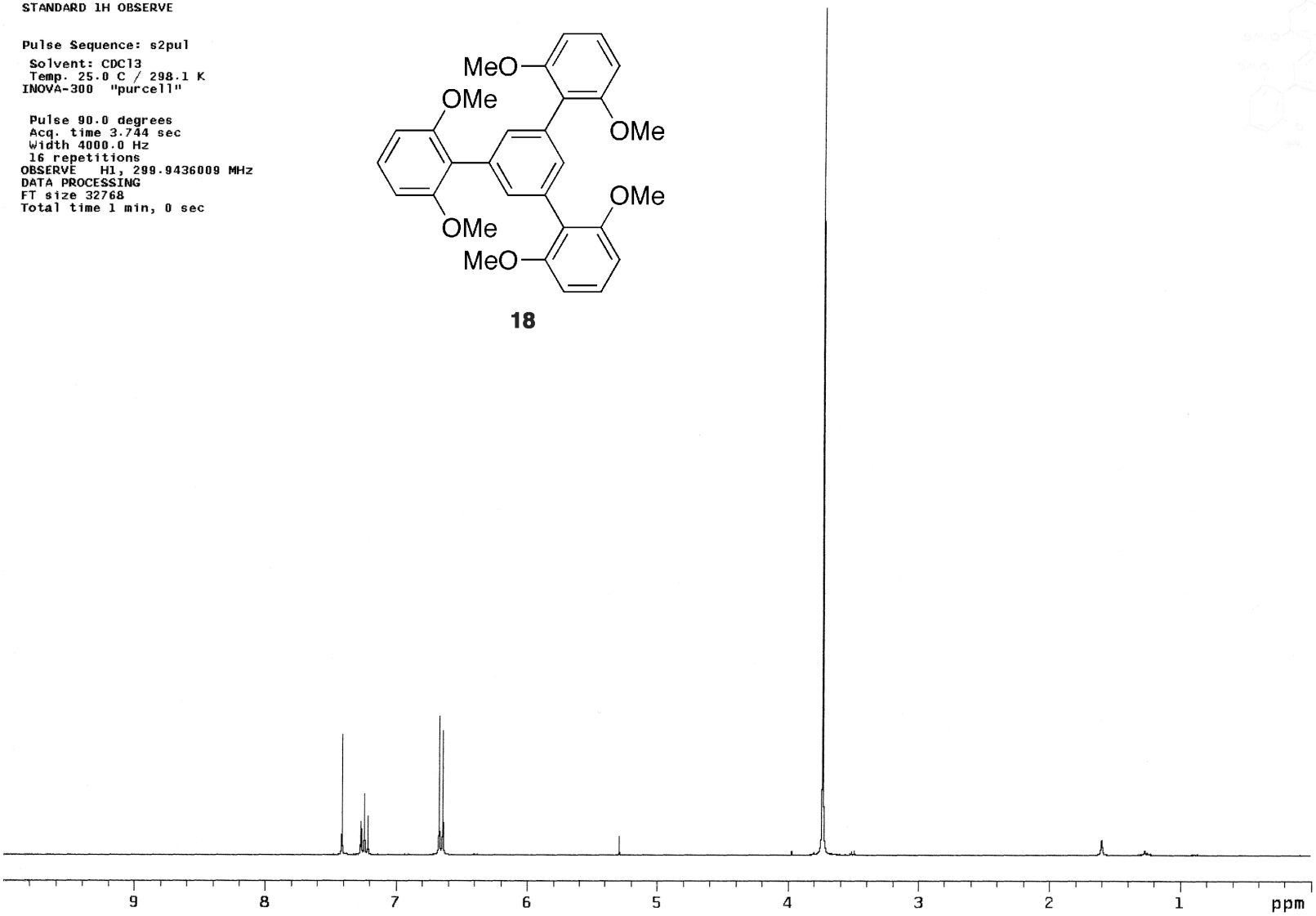
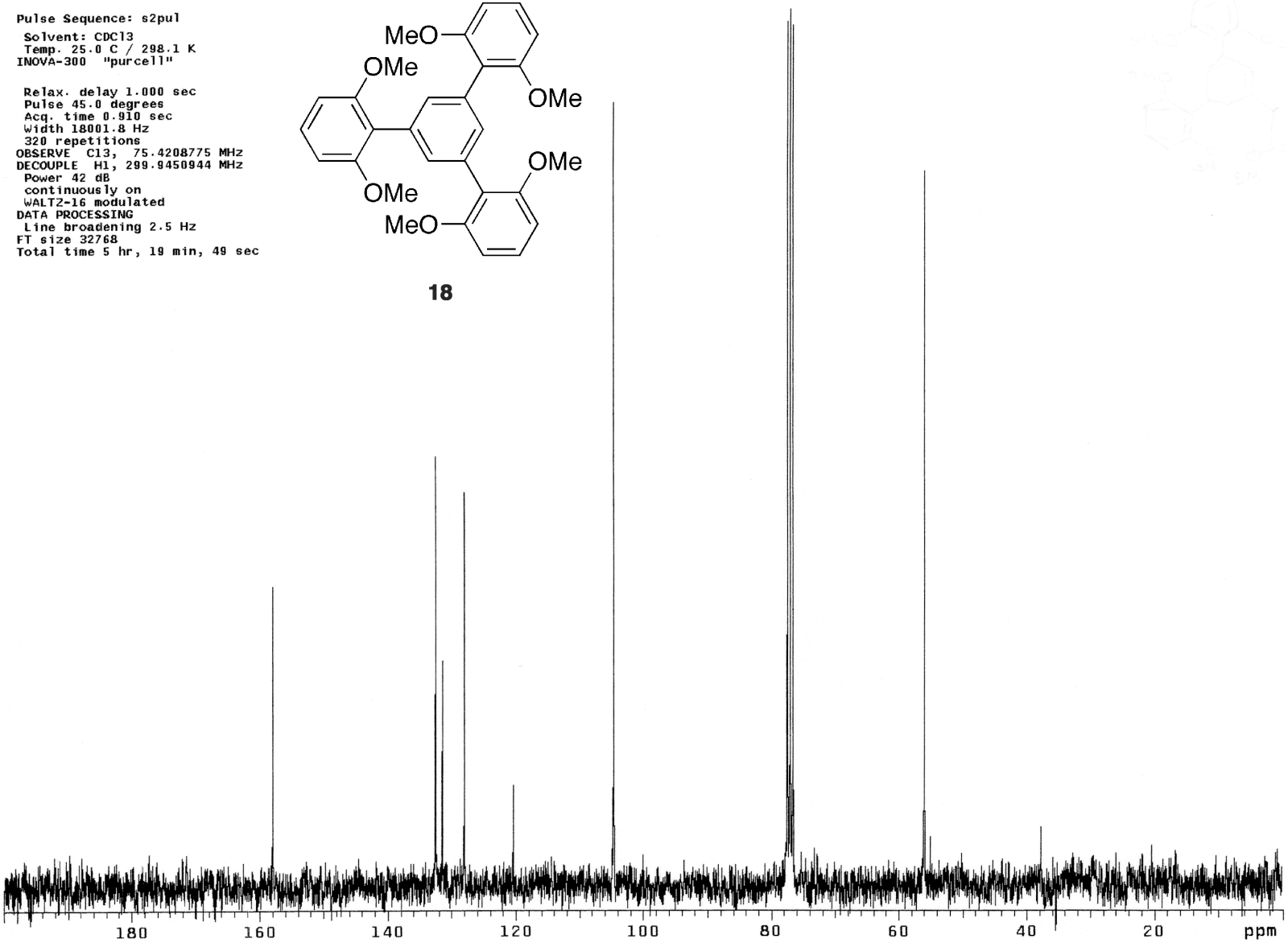


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-300 "purcell"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.910 sec
Width 18001.8 Hz
320 repetitions
OBSERVE C13, 75.4208775 MHz
DECOUPLE H1, 299.9450944 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.5 Hz
FT size 32768
Total time 5 hr, 19 min, 49 sec

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