Electronic Supplementary Material (ESI) for Green Chemistry

Supporting Information

A practicable environmentally benign one-pot synthesis of 2-arylbenzofurans at room temperature

Lian-Yan Liao, Gang Shen, Xue Zhang and Xin-Fang Duan*
College of Chemistry, Beijing Normal University, Beijing, 100875, China.
Email: xinfangduan@vip.163.com

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General procedure for debenzylation by hydrogenation (Scheme 2)

Benzyloxy-substituted benzofuran (**4hn, 4jo** or **4pn,** 0.5 mmol) was dissolved in a mixture of THF (20 mL) and CH_3OH (10 mL). To the solution, palladium on carbon (10%, 0.08 g) was added. The mixture was hydrogenated under 1 atm of H_2 at ambient temperature. After completion of debenzylation (monitored by tlc), the reaction mixture was filtered and evaporated to provide the crude product. Column chromatography on silica gel with petroleum ether-ethyl acetate as an eluent gave the desired compound.

Characterization data for known benzofurans

2-(4-Methoxyphenyl)benzofuran (4ae)[1]

White solid; m.p. = 147.5-148.4 °C (lit. 148-149 °C); IR(KBr) 1610, 1505, 1454, 1249, 1023, 835, 799, 751; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.48 (d, *J* = 7.1 Hz, 2H), 7.46 (d, *J* = 9.8 Hz, 1H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.11-7.20 (m, 2H), 6.91 (d, *J* = 8.9 Hz, 2H), 6.82 (s, 1H), 3.79 (s, 3H).

2-(4-Methylphenyl)benzofuran (4af)[2]

White solid; mp = 127-128.5 °C (lit. 128-129 °C); IR(KBr) 1505, 1451, 1257, 1169, 1033, 919, 825, 801; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.69 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.12-7.21 (m, 4H), 6.89 (s, 1H), 2.32 (s, 3H).

2-(3,4-Dimethoxyphenyl)benzofuran (4ag)[3]

White solid; m.p. = 120.3-121.5 °C (lit. 118.9 °C); ν_{max} (KBr)/cm^{-1} 1606, 1573, 1508, 1453, 1442, 1251, 1227, 1160, 1138, 1020, 940, 859, 799, 754; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.57 (d, *J* = 7.3 Hz, 1H),

7.52 (d, J = 7.8 Hz, 1H), 7.45 (dd, J_1 = 8.4 Hz, J_2 = 1.2 Hz, 1H), 7.39 (s, 1H), 6.96-7.29 (m, 2H), 6.96 (d, J = 8.4 Hz, 1H), 6.92 (s, 1H), 4.0 (s, 3H), 3.94 (s, 3H).

5-(Benzofuran-2-yl)benzo[d][1,3]dioxole(4ah)[3]

White solid; m.p. = 102.5-104.1 °C (lit. 101.3-101.8 °C); IR (KBr) 1502, 1488, 1452, 1254, 1234, 1042, 934, 799, 749, 737. ^{1}H NMR ($CDCl_3$, 400MHz) δ 7.48 (d, J = 7.2 Hz, 1H), 7.42 (d, J = 8.0 Hz, 1H), 7.33 (dd, J_1 = 8.1 Hz, J_2 = 1. 6 Hz, 1H), 7.25 (d, J = 1.5 Hz, 1H), 7.12-7.21 (m, 2H), 6.80-6.83 (m, 2H), 5.95 (s, 2H).

2-(Thiophen-2-yl)benzofuran (4ai)[4]

Pale yellow solid; m.p. = 94.1-95.1 °C (lit. 95-96.5 °C); IR (KBr) 1586, 1450, 1257, 997, 849, 802, 750; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.48 (d, J = 7.3 Hz, 1H), 7.42 (d, J_1 = 4.6 Hz, J_2 = 3.0 Hz, 2H), 7.28 (d, J = 5.0 Hz, 1H), 7.13-7.22 (m, 2H), 7.04 (dd, J_1 = 4.9 Hz, J_2 = 3.7 Hz, 1H), 6.80 (s, 1H).

7-Methoxy-2-phenylbenzofuran (4ga)[4]

White solid; m.p. = 79.5-80.8 °C (lit. 79-80 °C); IR (KBr) 1622, 1591, 1498, 1483, 1269, 1119, 1100, 795, 770, 727, 684; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.82 (d, J = 7.6 Hz, 2H), 7.29-7.39 (m, 2H), 7.27 (d, J = 7.3 Hz, 1H), 7.05-7.12 (m, 2H), 6.94 (s, 1H), 6.73 (d, J = 7.4 Hz, 1H), 3.98 (s, 3H).

7-Methoxy-2-(4-methoxyphenyl)benzofuran (4ge)[5]

White solid; m.p. = 78-79 °C (lit. 80-81 °C); IR (KBr) 1615, 1588, 1506, 1488, 1434, 1308, 1251, 1215, 1176, 1097, 834, 804, 729; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.85 (d, J = 8.8 Hz, 2H), 6.87- 7.17 (m, 5H), 6.86 (d, J = 1.5 Hz, 1H), 4.01 (s, 3H), 3.85 (s, 3H).

Corsifuran C (4ie)[6]

White solid; m.p. = 169.5-171.1 °C (lit. 163 °C); IR (KBr) 1610, 1505, 1472, 1437, 1250, 1206, 1176, 1143, 1020, 916, 833, 791; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.70 (d, J = 8.8 Hz, 2H), 7.31 (d, J = 8.8 Hz, 1H), 6.95 (d, J = 2.4 Hz, 1H), 6.90 (d, J = 8.8 Hz, 2H), 6.79 (d, J = 2.5 Hz, 1H), 6.78 (dd, J_1 = 8.9 Hz, J_2 = 4.7 Hz, 1H), 3.79 (s, 3H), 3.78 (s, 3H).

2-(4-Methylphenyl)-5-methoxybenzofuran (4if)[7]

White solid; m.p. = 125.6-127.2 °C; IR (KBr) 1609, 1597, 1472, 1445, 1430, 1209, 1180, 1145, 1029, 916, 841, 825, 799; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.75 (d, *J* = 8.2 Hz, 2H), 7.41 (d, *J* = 8.9 Hz, 1H), 7.25-7.27 (m, 2H), 7.04 (d, *J* = 2.6 Hz, 1H), 6.87-6.92 (m, 2H), 3.87 (s, 3H), 2.41 (s, 3H).

6-Benzyloxy-2-phenylbenzofuran (4ja)[8]

White solid; m.p. 116.5-117.8 °C. IR (KBr)[1] 1622, 1491, 1450, 1301, 1264, 1156, 1019, 820, 761, 731, 691. ^{1}H NMR ($CDCl_3$, 400MHz) δ 7.82 (d, *J* = 8.5 Hz, 2H), 7.39-7.50 (m, 7H), 7.30-7.37 (m, 2H), 7.15 (d, *J* = 1.9 Hz, 1H), 6.95-6.98 (m, 2H), 5.14 (s, 2H).

2-(4-Chlorophenyl)-5-methylbenzofuran (4kk)[9]

White solid; m.p. = 190.8-191.9 °C (lit. 178-180 °C). IR (KBr) 1612, 1579, 1462, 1403, 1260, 1093, 1034, 1010, 828, 803, 505; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.70 (d, *J* = 8.6 Hz, 2H) 7.33 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 6.2 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 1H), 6.87 (s, 1H), 2.37 (s, 3H).

5-Chloro-2-(4-methoxyphenyl)benzofuran (4le)[10]

White solid; m.p. = 162.8-164.7 °C (lit. 164-165 °C); IR (KBr) 1609, 1505, 1445, 1255, 1177, 1040, 833, 809, 796; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.71 (d, *J* = 8.8 Hz, 2H), 7.44 (d, *J* = 1.8Hz, 1H), 7.33 (d, *J* = 8.6 Hz, 1H), 7.12 (dd, J_1 = 8.6 Hz, J_2 = 2.0 Hz, 1H), 6.91(d, *J* = 8.8 Hz, 2H), 6.76 (s, 1H), 3.80 (s, 3H).

5-Iodo-2-(4-methoxyphenyl)benzofuran (4ne)[11]

white solid. mp 207.0-208.8 °C. IR (KBr) 1610, 1504, 1441, 1252, 1177, 1038, 835, 804, 791. ^{1}H NMR ($CDCl_3$, 400MHz) δ 7.88 (d, *J* = 1.9 Hz, 1H), 7.78 (d, *J* = 8.9 Hz, 2H), 7.78 (dd, *J* = 8.5 Hz, *J* = 1.8 Hz, 1H), 7.28 (s, 1H), 6.98 (d, *J* = 8.9 Hz, 2H), 6.81 (d, *J* = 0.7 Hz, 1H), 3.87 (s, 3H).

5-Bromo-2-(3,4-dimethoxyphenyl)-7-methoxybenzofuran (4og)[3]

White solid; mp 204.5-205.8°C; IR (KBr) 1597, 1513, 1483, 1464,

1427, 1279, 1264, 1230, 1141, 1070, 1022, 960, 851, 800; ^{1}H NMR ($CDCl_3$, 400MHz) δ 7.45 (dd, J_1 = 8.4 Hz, J_2 = 2.0 Hz, 1H), 7.35(d, J = 1.9 Hz, 1H), 7.30(d, J = 1.6 Hz, 1H), 6.94 (d, J = 8.4Hz, 1H), 6.90 (d, J = 1.6 Hz, 1H), 6.82 (s, 1H), 4.03 (s, 3H), 3.09 (s, 3H), 3.94(s, 3H).

5-(6-(benzyloxy)benzofuran-2-yl)-6-methoxy-benzo[d][1,3] dioxole (Scheme2; 4jo)[4]

According to the general procedure for benzofurans, the compound **4jo** was prepared from **1j** (0.46 g, 2.0 mmol) and **2o** (1.0 g, 2.2 mmol) in 77% yield (0.58 g).

White solid; m.p. 145.5-146.3 °C (lit. 146.9-148.0 °C); IR (KBr) 1622, 1506, 1488, 1267, 1194, 1155, 1124, 1014 , 818, 741. ^{1}H NMR ($CDCl_3$, 400MHz) δ 7.40 (d, J = 6.9 Hz, 3H), 7.31-7.36 (m, 3H), 7.28 (d, J = 7.2 Hz, 1H), 7.07 (s, 1H), 7.03 (d, J = 1.3 Hz, 1H), 6.85 (dd, J_1 = 8.5 Hz , J_2 = 2.2 Hz, 1H), 6.55 (s, 1H), 5.91 (s, 2H), 5.05 (s, 2H), 3.86 (s, 3H).

Cicerfuran (Scheme 2)[4]

According to reported procedure for debenzylation by hydrogenation, the title compound was obtained from **4jo** (0.19 g, 0.5 mmol) in 93 % yield (0.14 g, overall yield from **1j**: 72%).

White solid; m.p.= 153.4-154.9 °C (lit. 153.4-154.7 °C); IR (KBr) 3481, 1624, 1507, 1488, 1466, 1452, 1370, 1262, 1197, 1176, 1147, 1041, 1023, 934, 820; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.41 (s, 1H), 7.31 (d, J = 8.3 Hz, 1H), 7.06 (s, 1H), 6.90 (d, J = 1.9 Hz, 1H), 6.67 (dd, J_1 = 8.4 Hz, J_2 = 2.2 Hz, 1H), 6.56 (s, 1H), 5.91 (s, 2H), 3.86 (s, 3H).

Erythbidin E (Scheme 2)[12]

According to the general procedure for benzofurans, **4hn** was prepared from **1h** (0.30 g, 2.0 mmol) and **2n** (1.2 g, 2.2 mmol) in 79% yield (0.69 g).

The title compound was prepared following the general procedure for debenzylation by hydrogenation from **4hn** (0.22 g, 0.5 mmol) in 94% yiled (0.12 g, overall yield from **1h**: 74%)

White solid; m.p.= 146.8-148.5 °C; IR (KBr) 3422, 1624, 1491, 1271, 1149, 1109, 1017, 976, 823. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.3 (d, J =

9.1 Hz, 1H), 7.36 (d, J = 8.5 Hz, 1H), 6.99 (d, J = 2.0 Hz, 1H), 6.83 (dd, J_1 = 8.5 Hz, J_2 = 2.3 Hz, 1H), 6.77 (s, 1H), 6.41-6.44 (m, 2H), 3.80 (s, 3H).

Ebenfuran I (Scheme 2)[13]

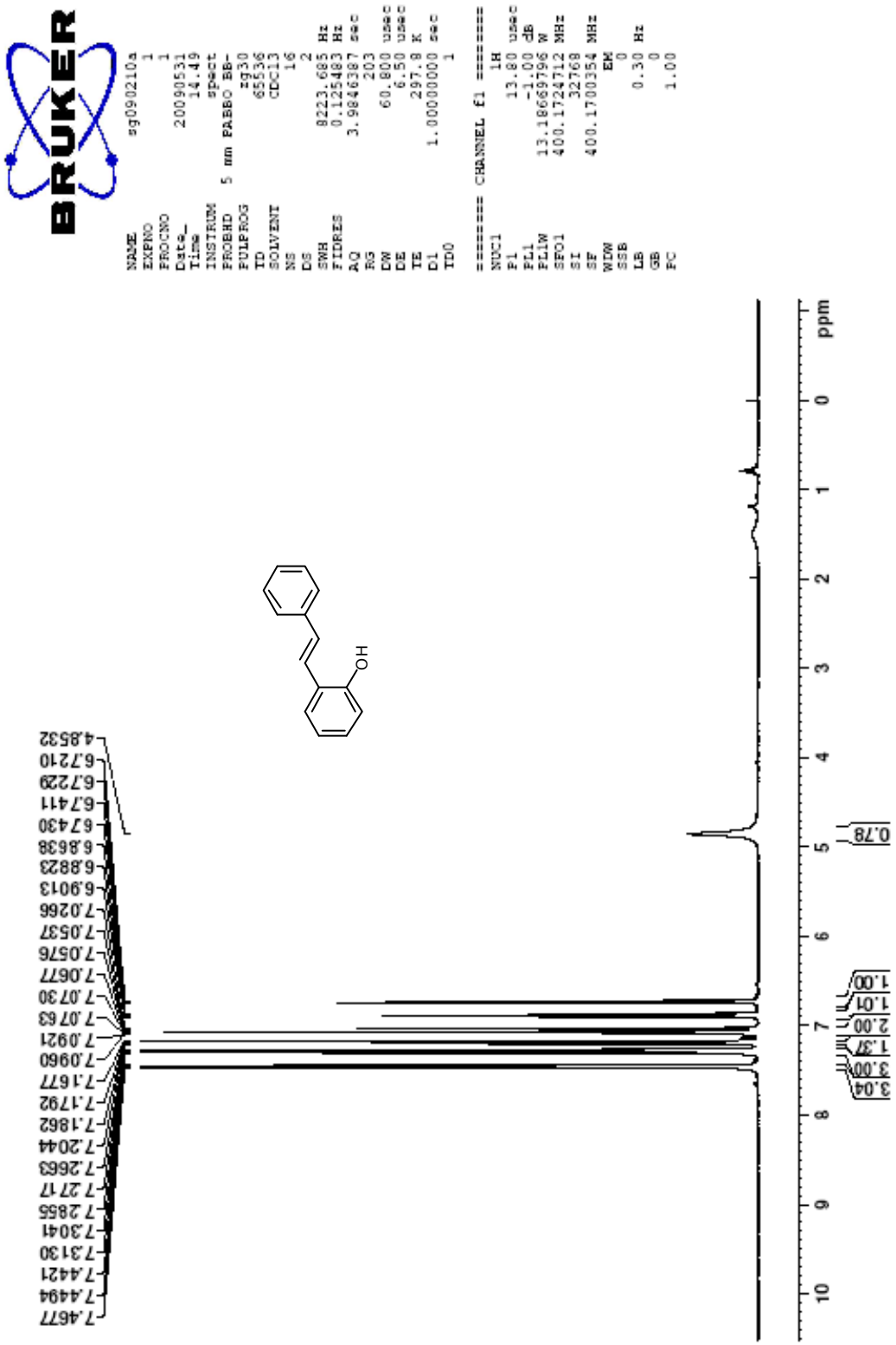
According to the general procedure for benzofurans, **4pn** was prepared from **1p** (0.39 g, 1.5 mmol) and **2n** (0.9 g, 1.7 mmol) in 70% yield (0.57 g).

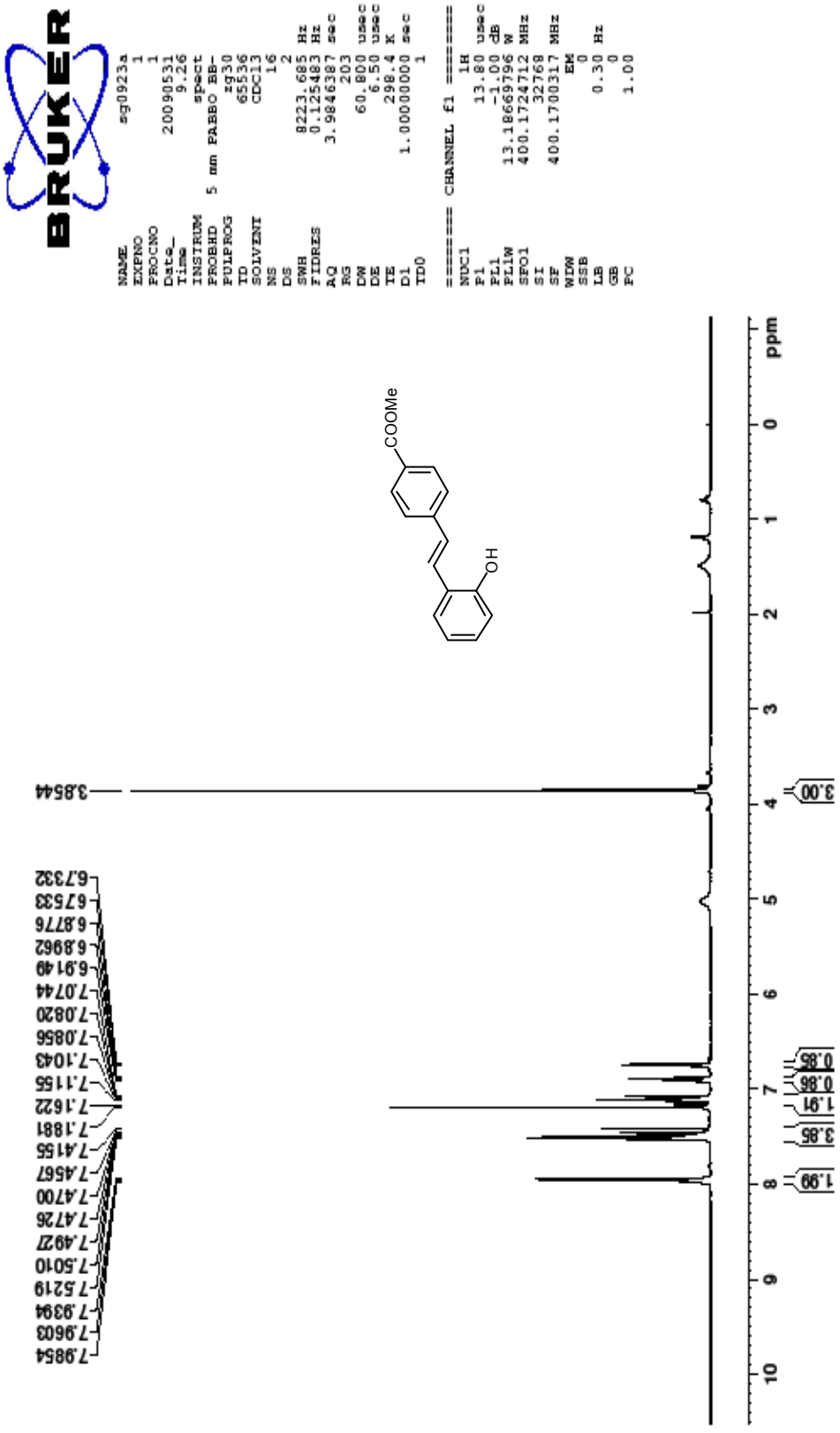
The title compound was prepared following the general procedure for debenzylation by hydrogenation from **4pn** (0.27 g, 0.5 mmol) in 97% yiled (0.13 g, overall yield from **1p**: 68%).

Pale yellow solid; mp 183.4-184.7 °C; IR (KBr) 3503, 1625, 1602, 1486, 1453, 1377, 1325, 1280, 1255, 1149, 1117, 1007, 974, 864, 503; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.40 (d, J = 9.2 Hz, 1H), 6.99 (d, J = 2.2 Hz, 2H), 6.71 (s, 1H), 6.40-6.43 (m, 2H), 3.89 (s, 3H).

References

1. N. Takeda, O. Miyata, and T. Naito, *Eur. J. Org. Chem.,* 2007, 1491.
2. C. Y. Chen, and P. G. Dormer, *J. Org. Chem.,* 2005, **70**, 6964.
3. X. F. Duan, J. X. Feng, and Z. B. Zhang, *Synthesis,* 2010, 515.
4. X. F. Duan, J. Zeng, Z. B. Zhang, G. F. Zi, *J. Org. Chem.,* 2007, **72**, 10283.
5. M. Watanabe, M. Date, K. Kawanishi, T. Hori, and S. Furukawa, *Chem. Pharm. Bull.,* 1991, **39**, 41.
6. A. Guy, J. P. Guette, and G. Lang, *Synthesis,* 1980, 222.
7. J. Bonnamour, M. Piedrafita, and C. Bolm, *Adv. Synth. Catal.,* 2010, **352**, 1577.
8. R. G. Cooke, R. M. McQuilkin, *Austr. J. Chem.,* 1969, **22**, 2395.
9. A. Arcadi, S. Cacchi, M. D. Rosario, G. Fabrizi, and F. Marinelli, *J. Org. Chem.,* 1996, **61**, 9280.
10. E. A. Jaseer, D. J. C. Prasad, and G. Sekar, *Tetrahedron,* 2010, **66**, 2077.
11. M. Ono, M. P. Kung, C. Hou, H. F. Kung, *Nucl. Med. Bio*., 2002, **29**, 633.
12. (a) H. Tanaka, H. Hattori, M. Sato, R. Yamaguchi, T. Fukai, T. Tanaka, E. Sakai, *Heterocycles,* 2007, **71**, 1779; (b) J. L. Ingham, P. M. Dewick, *Phytochemistry*, 1978, **17**, 535.
13. (a) M. Halabalaki, X. Alexi, N. Aligiannis, M. N. Alexis, A. L. Skaltsounis, *J. Nat. Prod.,* 2008, **71**, 1934; (b) M. Halabalaki, N. Aligiannis, Z. Papoutsi, S. Mitakou, P. Moutsatsou, C. Sekeris, A. L. Skaltsounis, *J. Nat. Prod.,* 2000, **63**, 1672.

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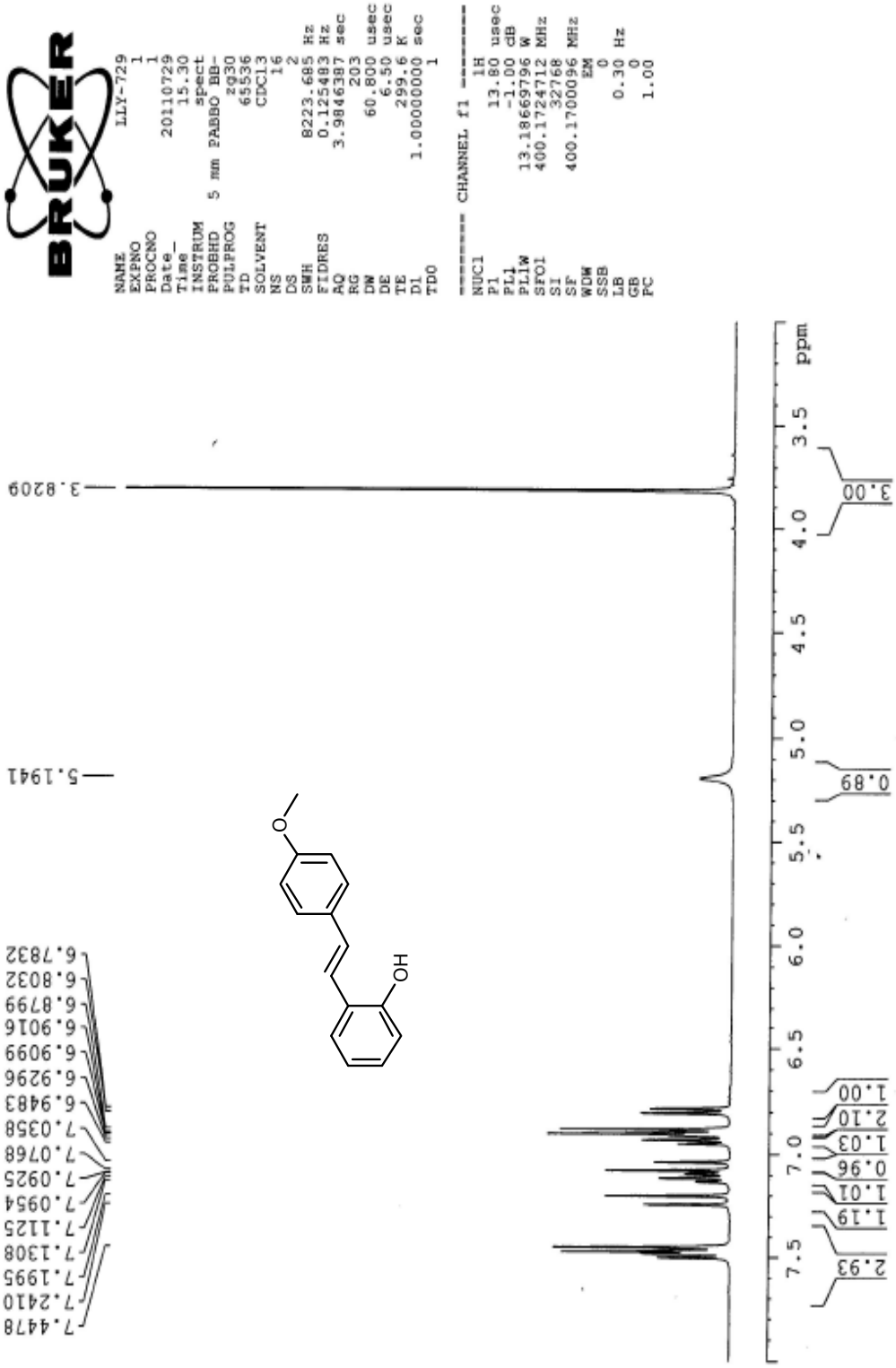

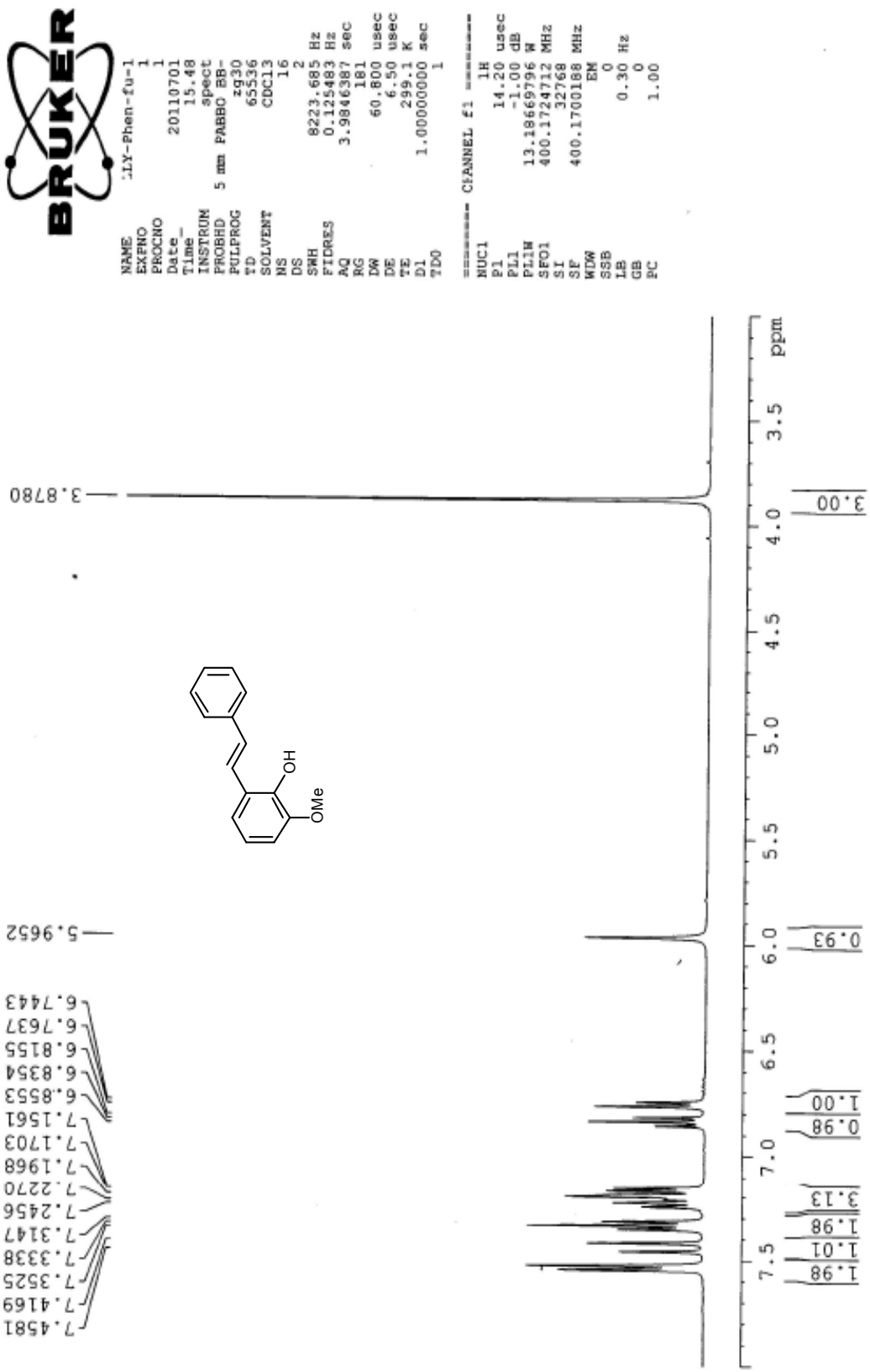
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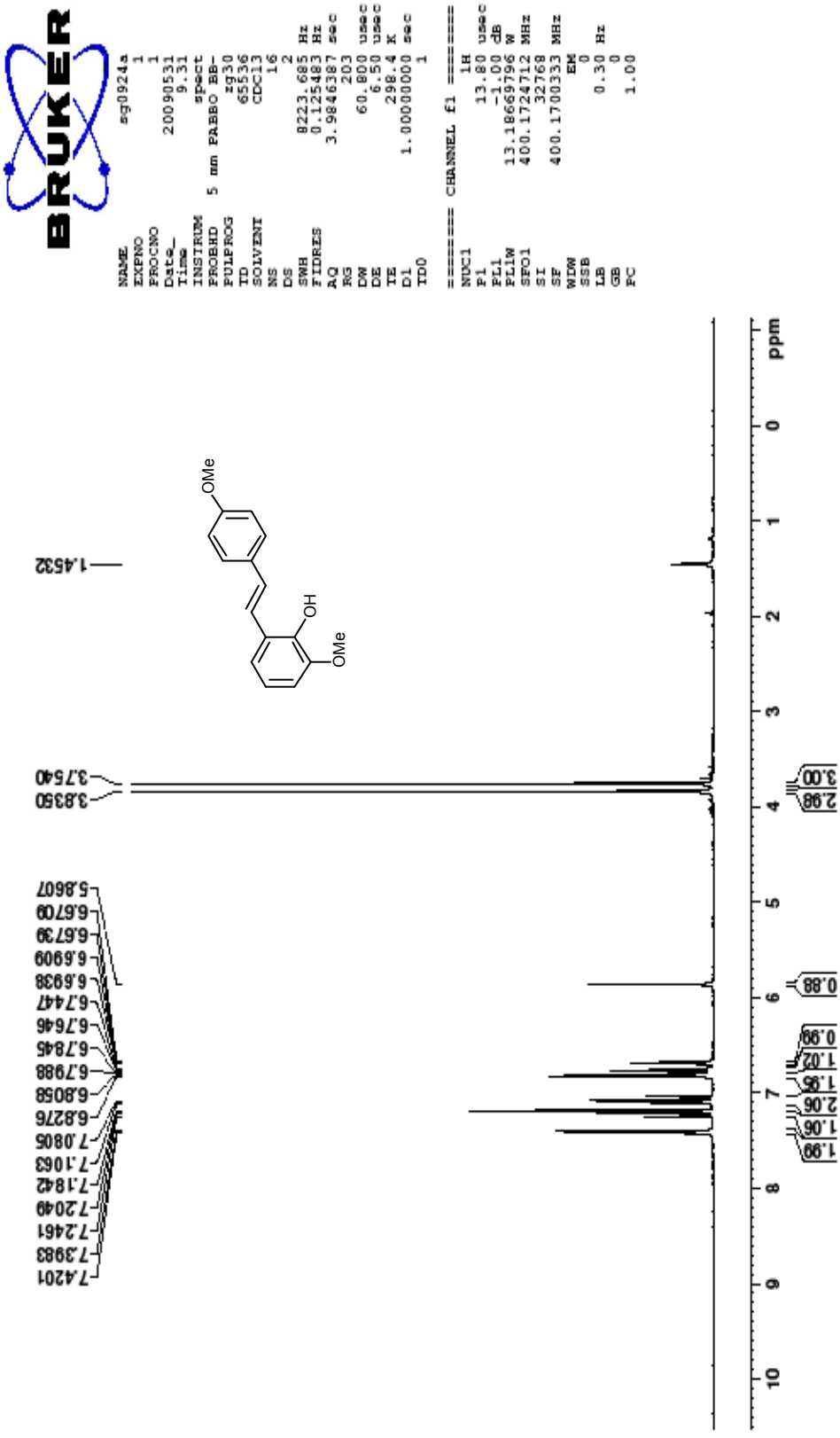

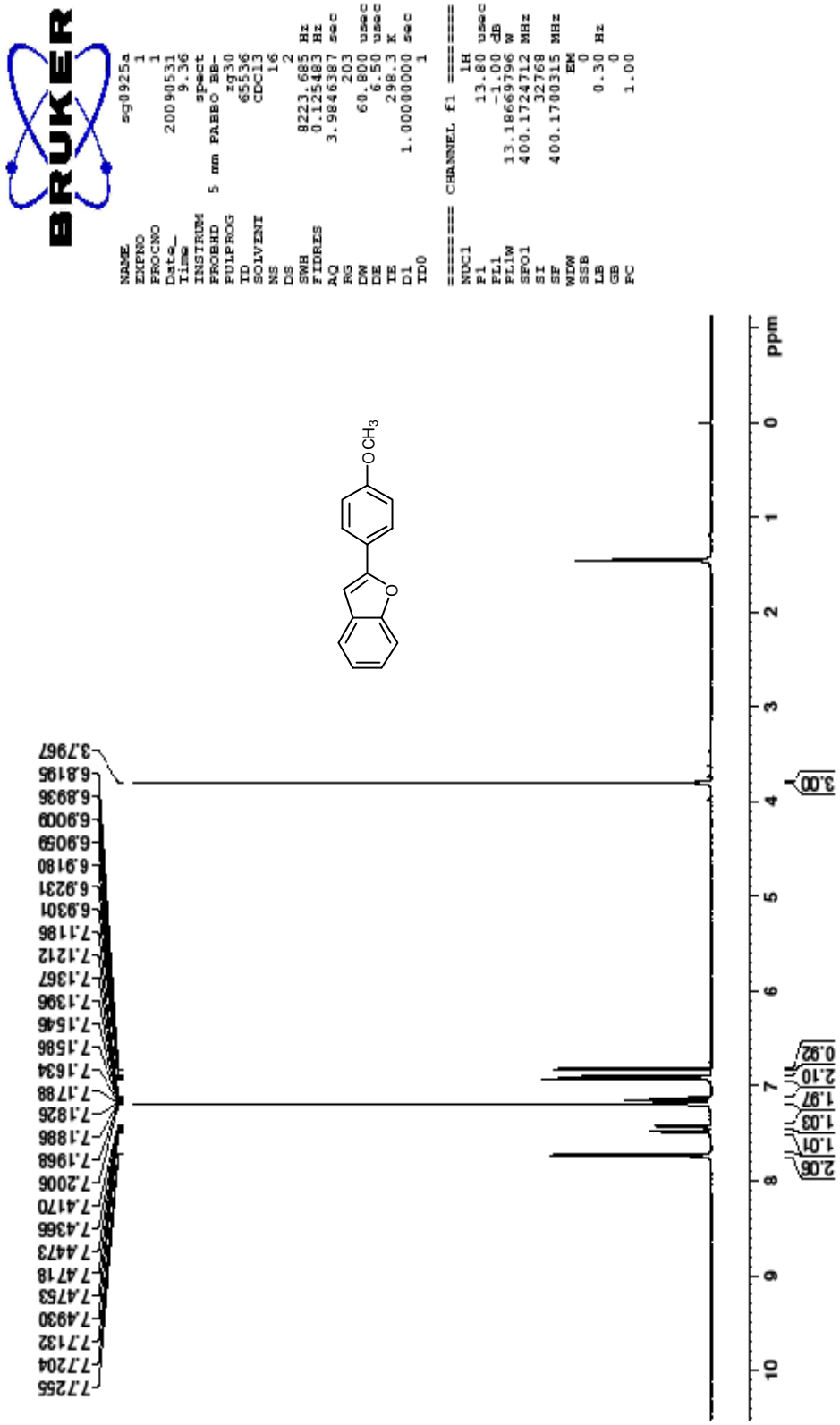
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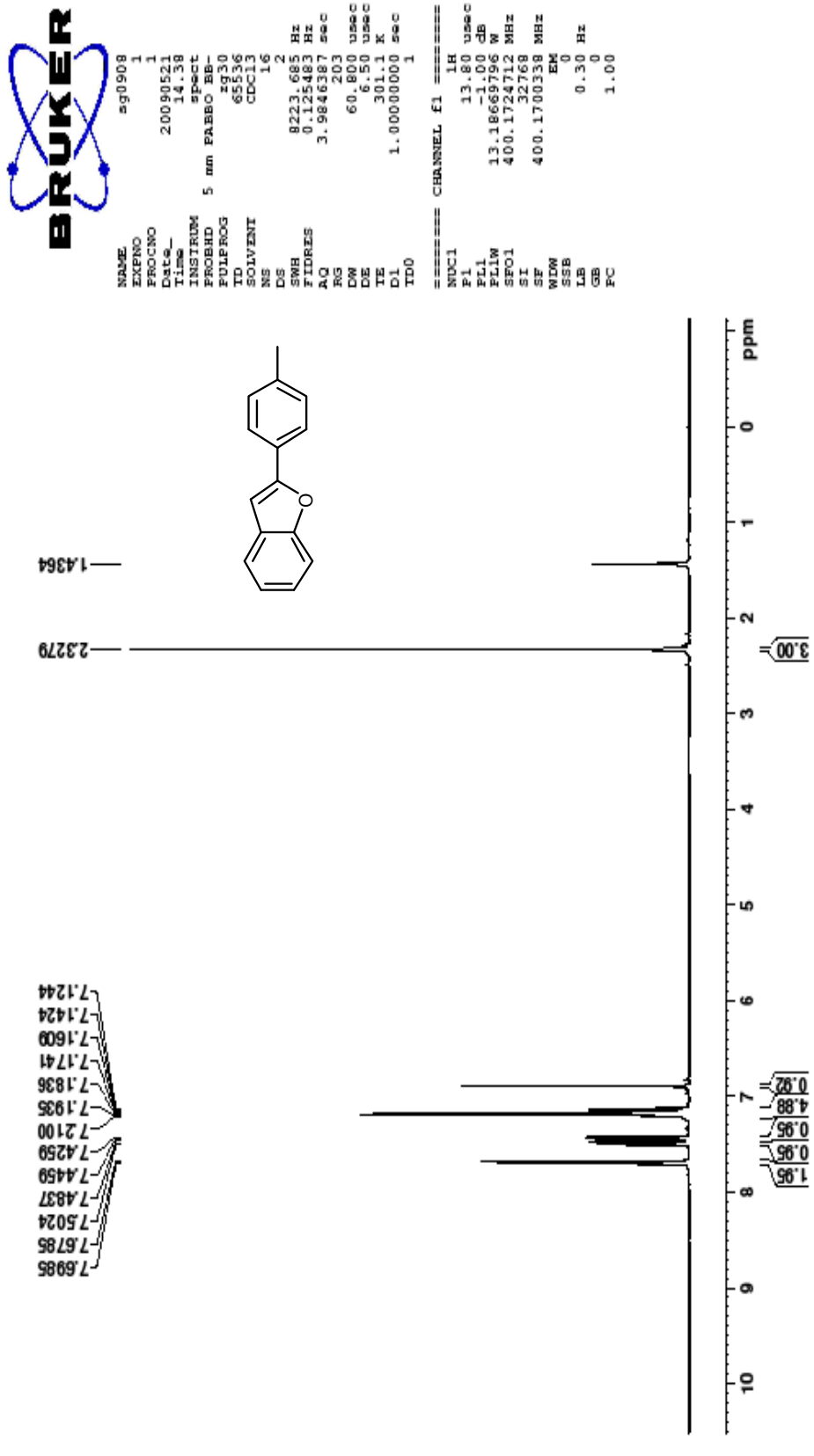

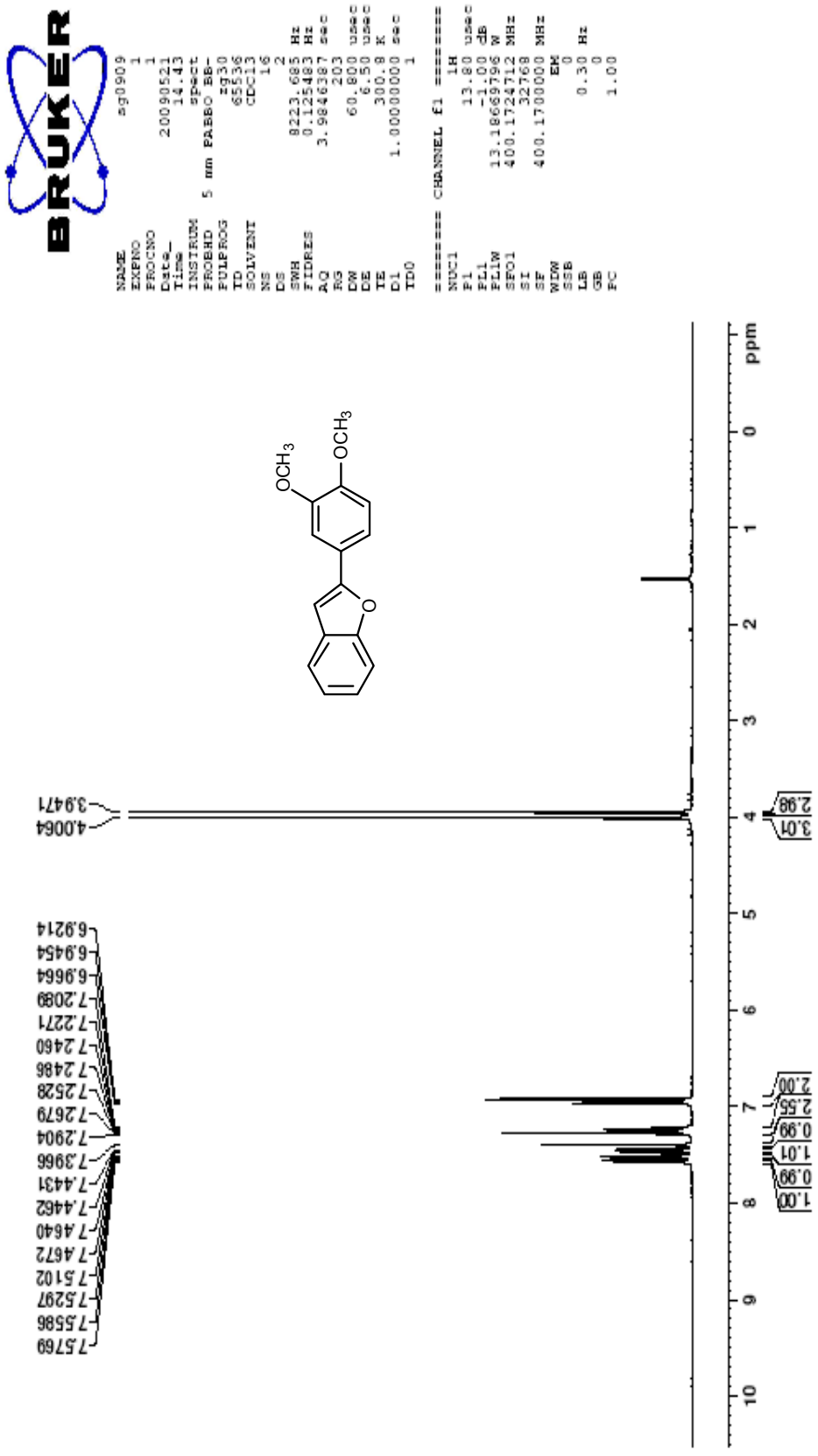
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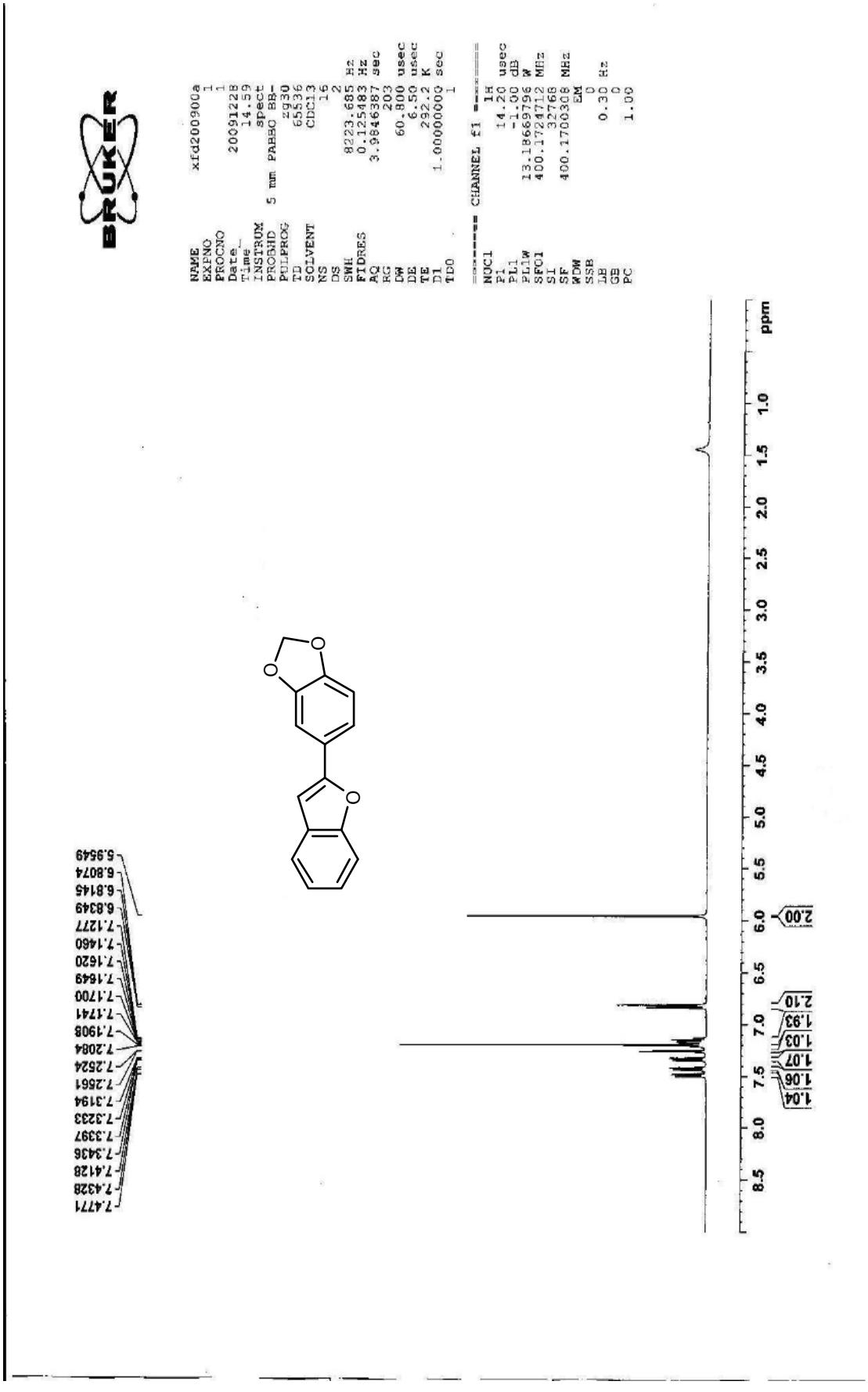
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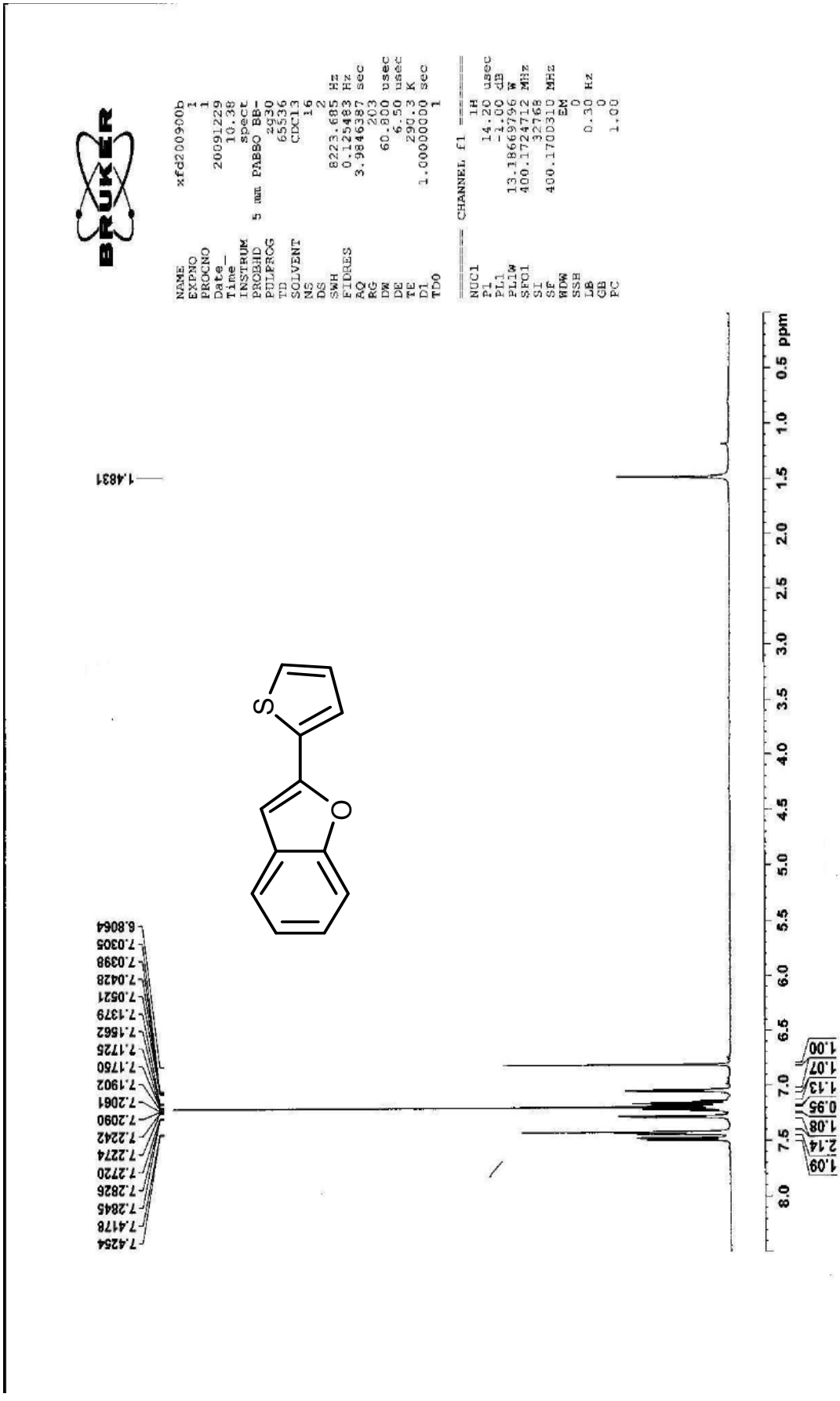
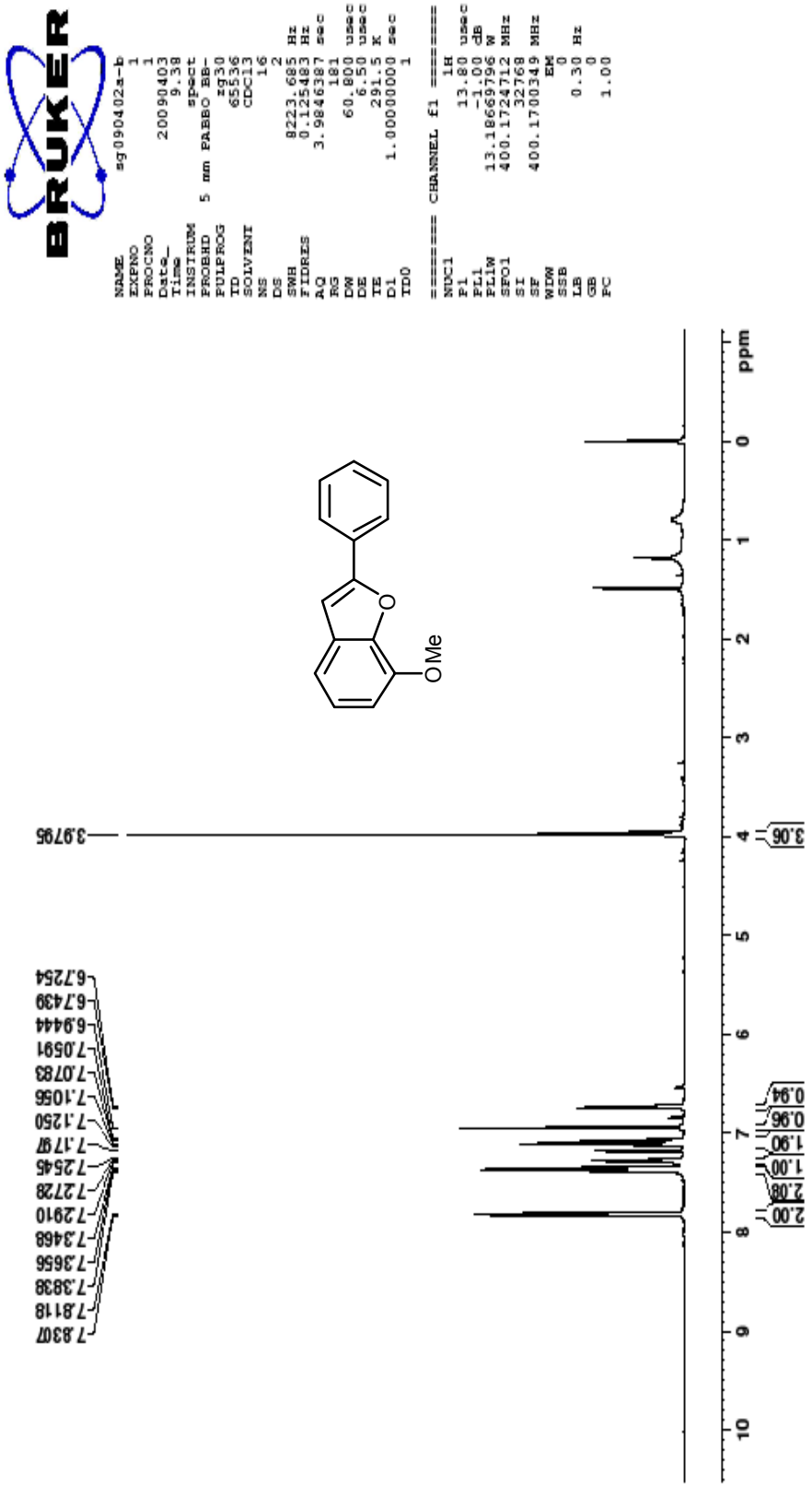
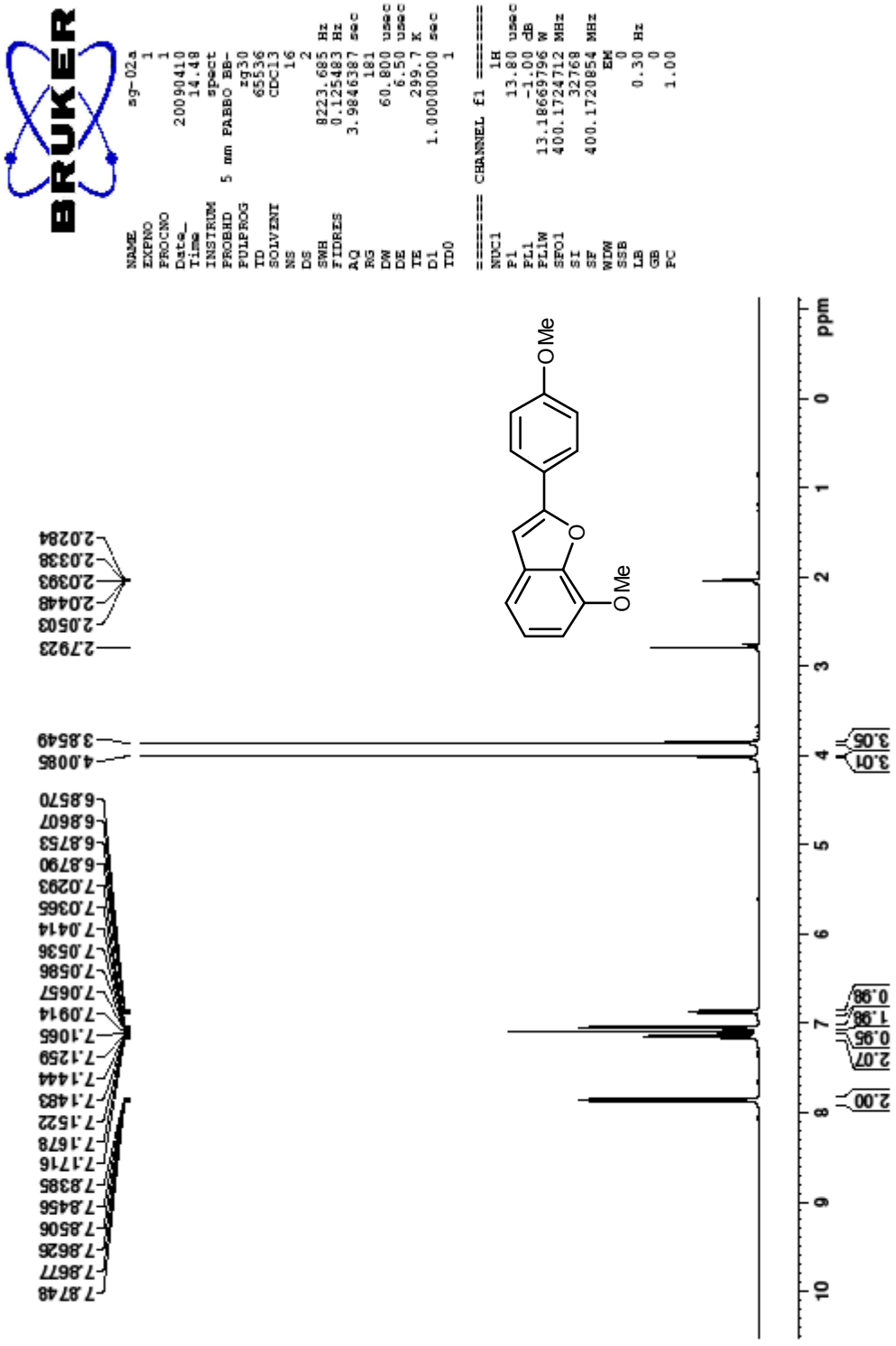
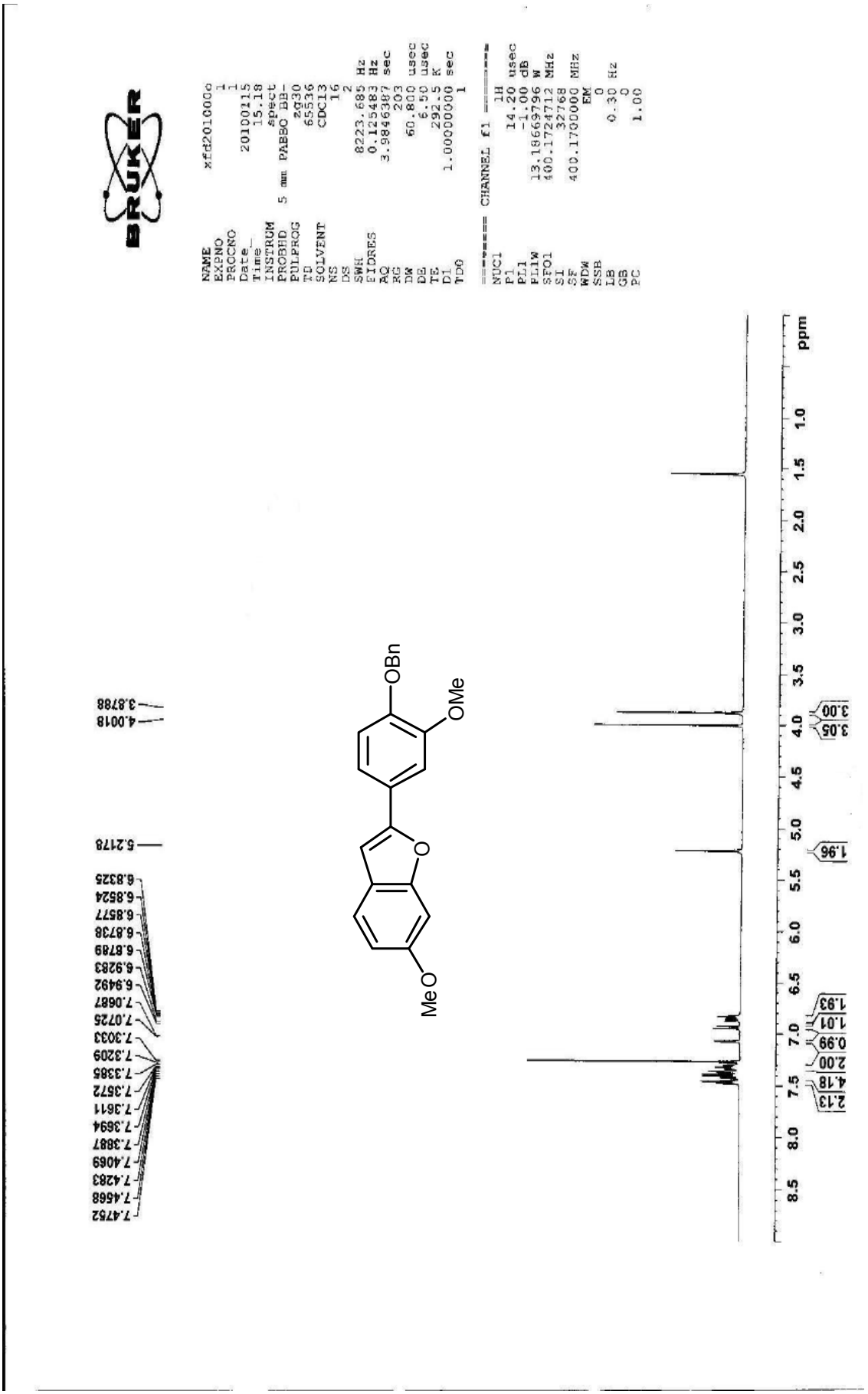
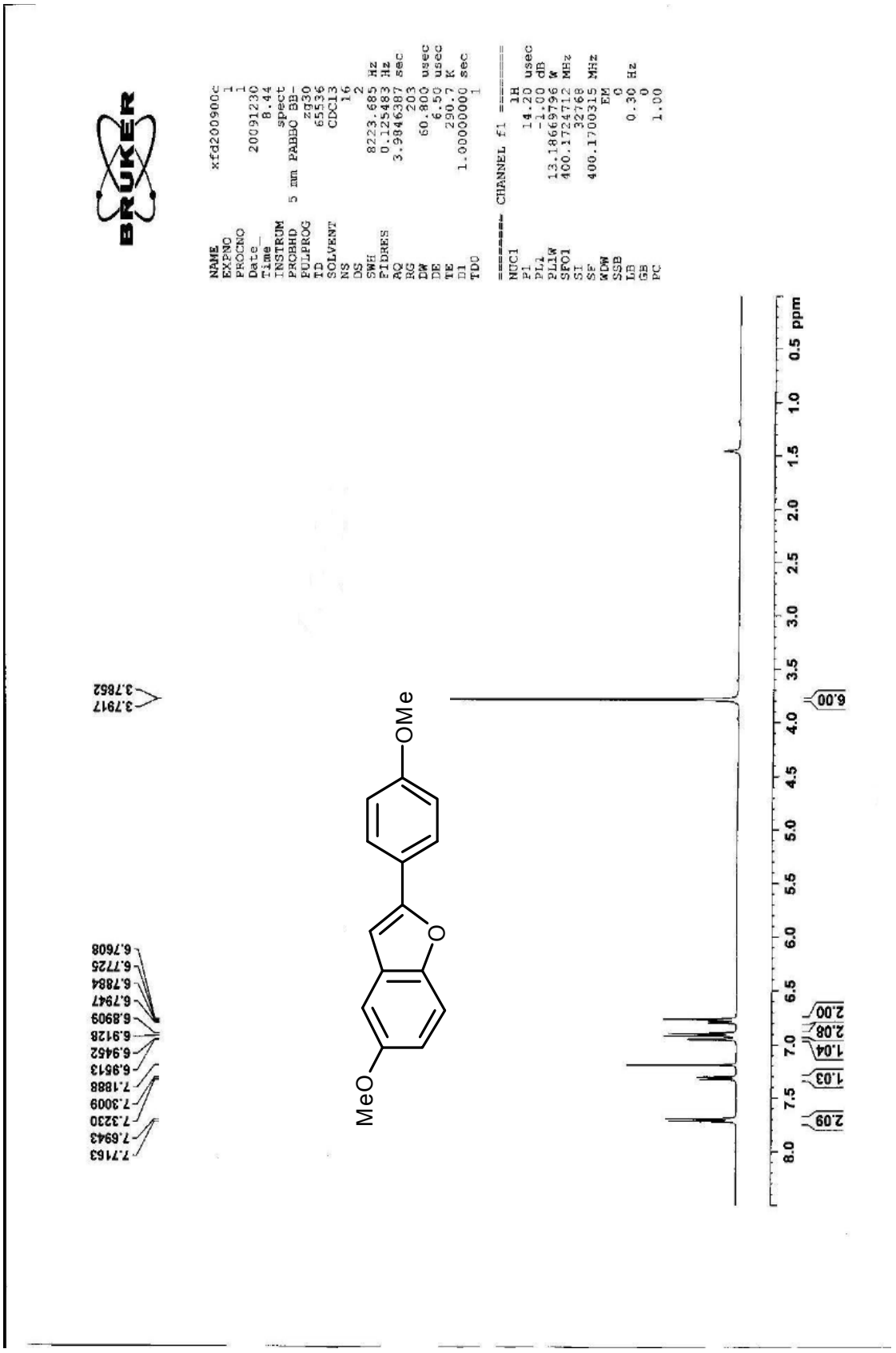
7.3397
7.3233
7.3194
7.2561
7.2524
7.2084
7.1908
7.1741
7.1700
7.1649
7.1620
7.1460
7.1277
6.8349
6.8145
6.8074
5.9549

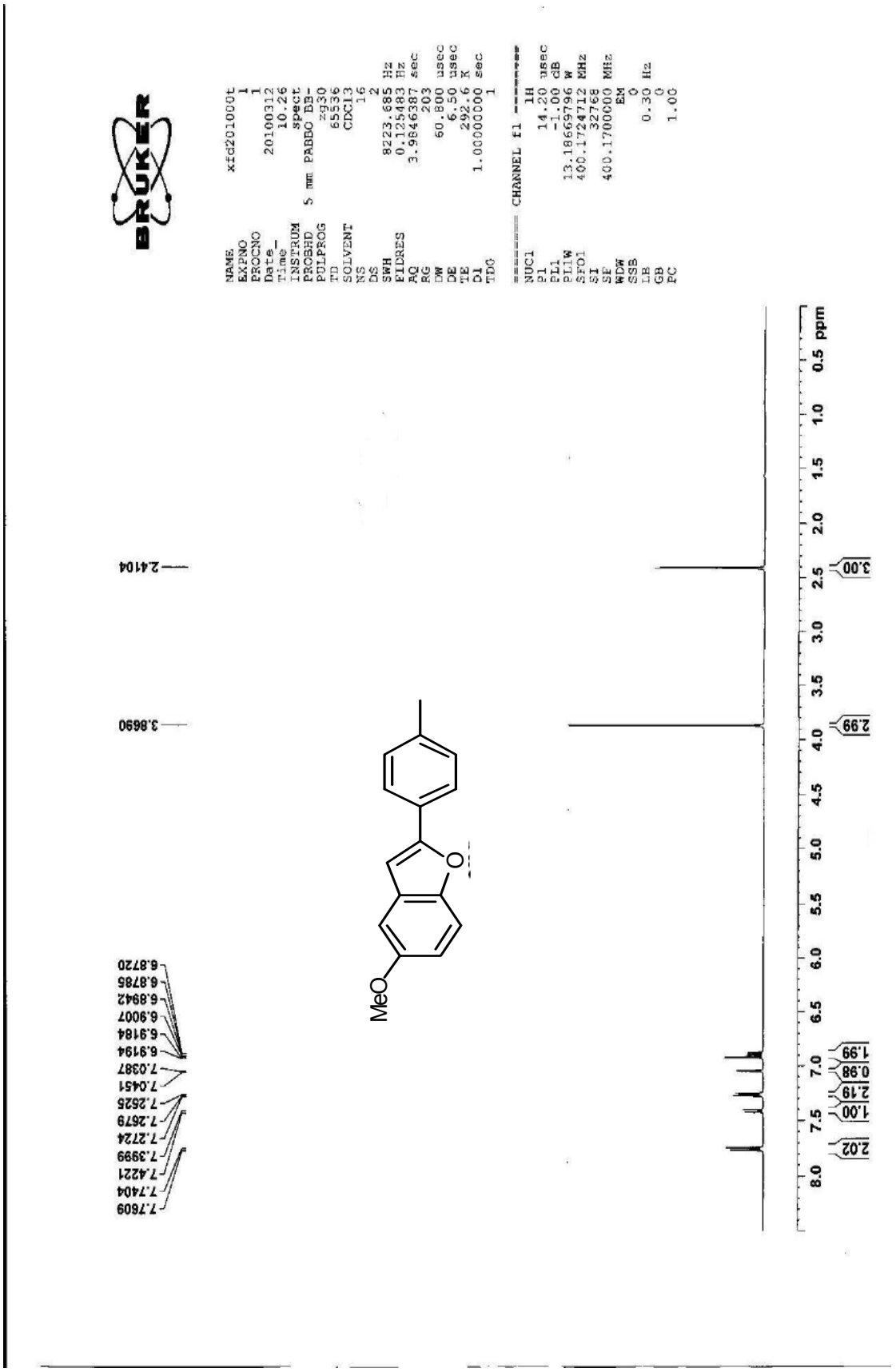

BRUKER
NAME xfd200900b
EXPNO 1
PROCNO 1
Date_ 20091229
Time 10.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 290.3 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700310 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.4831
7.4254
7.4178
7.2845
7.2826
7.2720
7.2274
7.2242
7.2090
7.2061
7.1902
7.1760
7.1725
7.1662
7.1379
7.0521
7.0428
7.0398
7.0305
6.8064
S
O
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5 ppm
1.09
2.14
1.08
0.95
1.13
1.07
1.00

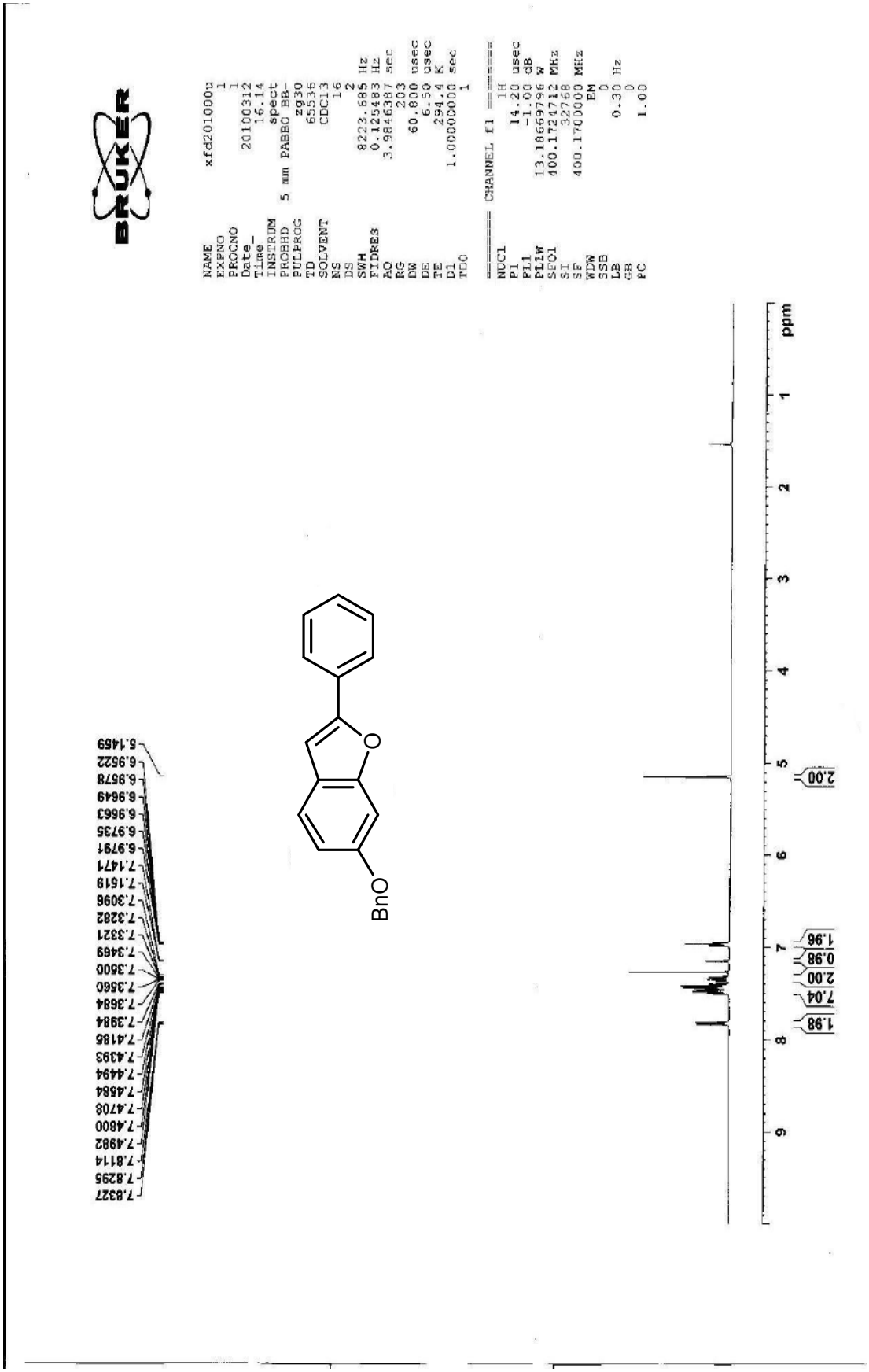

BRUKER
NAME sg090402s-b
EXPNO 1
PROCNO 1
Date_ 20090403
Time 9.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 291.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700349 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
3.9795
7.8307
7.8118
7.3838
7.3656
7.3468
7.2910
7.2728
7.2545
7.1767
7.1250
7.1056
7.0783
7.0591
6.9444
6.7439
6.7254
3.06
2.00
2.08
1.00
1.90
0.96
0.94
ppm

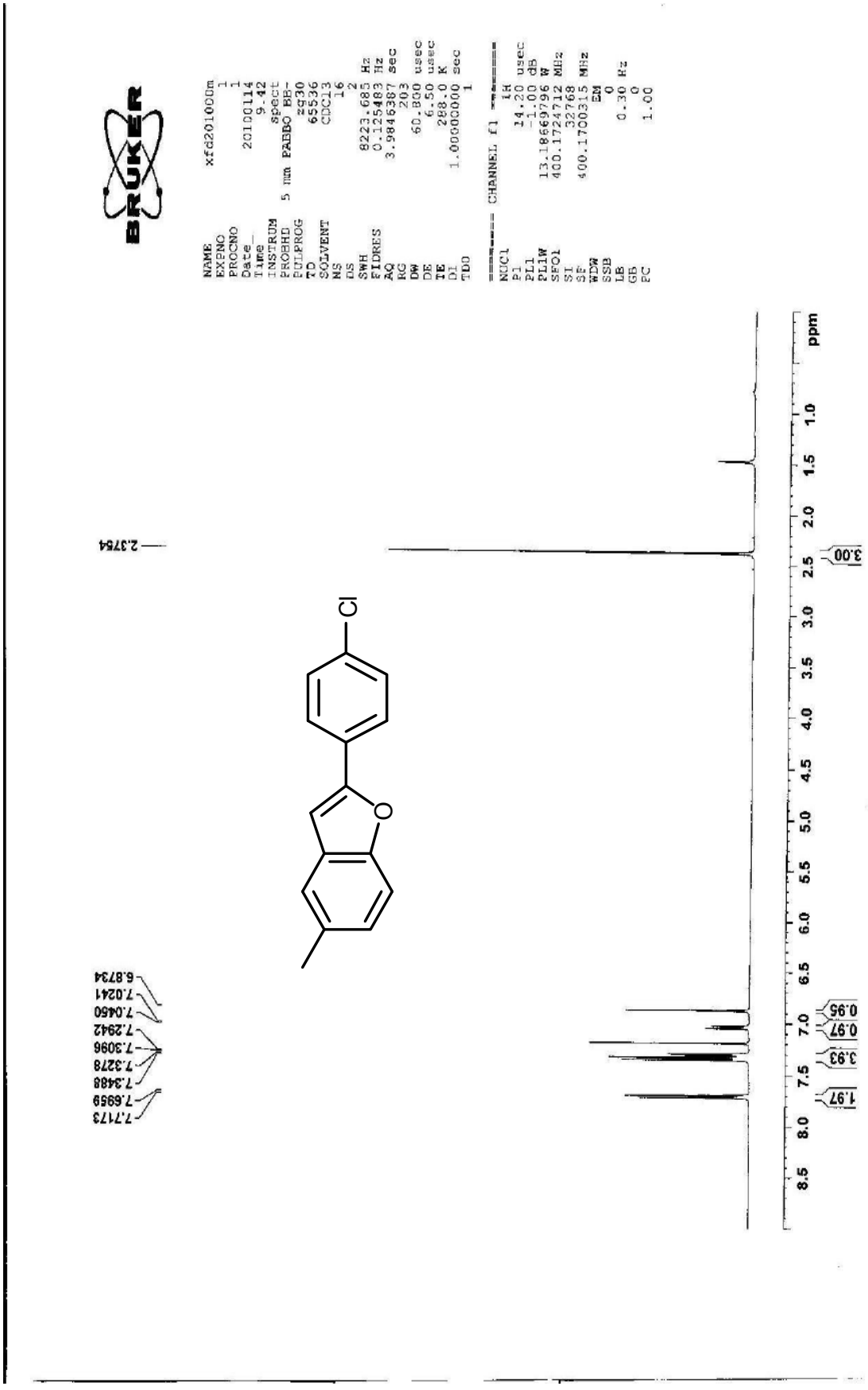

BRUKER
NAME sg-02a
EXPNO 1
PROCNO 1
Date_ 20090410
Time 14.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 299.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1720854 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
OMe
ppm
2.0284
2.0338
2.0393
2.0448
2.0503
2.7923
3.8549
4.0085
6.8570
6.8607
6.8753
6.8790
7.0293
7.0365
7.0414
7.0536
7.0586
7.0657
7.0914
7.1065
7.1259
7.1444
7.1483
7.1522
7.1678
7.1716
7.8385
7.8456
7.8506
7.8626
7.8677
7.8748
3.06
3.01
0.98
1.98
0.95
2.07
2.00

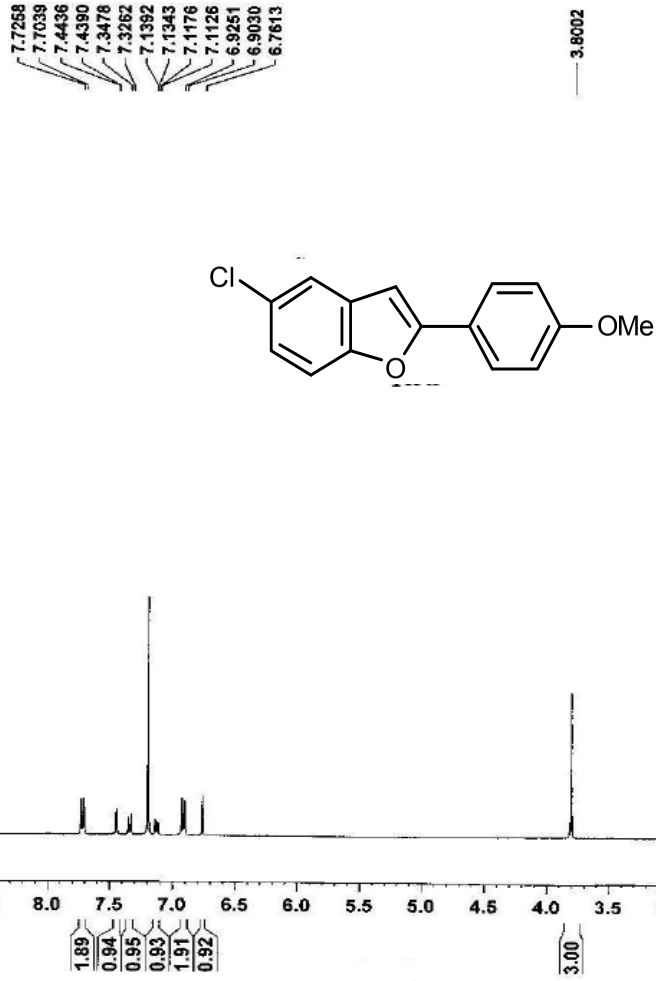

BRUKER
NAME xfd201000o
EXPNO 1
PROCNO 1
Date_ 20100115
Time 15.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 2930
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OBn
OMe
MeO
3.8788
4.0018
5.2178
6.8325
6.8524
6.8577
6.8738
6.8789
6.9283
6.9492
7.0687
7.0725
7.3033
7.3209
7.3385
7.3572
7.3611
7.3694
7.3887
7.4069
7.4283
7.4568
7.4752
3.00
3.05
1.96
1.93
1.01
0.99
2.00
4.18
2.13
ppm


BRUKER
NAME xfd200900c
EXPNO 1
PROCNO 1
Date_ 20091230
Time 8.44
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 203
DW 60.800 usec
DE 6.50 usec
TE 290.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700315 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.7917
3.7852
6.7608
6.7725
6.7884
6.7947
6.8909
6.9128
6.9462
6.9613
7.1888
7.3009
7.3230
7.6943
7.7163
OMe
MeO
O
6.00
2.09
1.03
1.04
2.08
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5 ppm


BRUKER
NAME xfd20100t
EXPNO 1
PROCNO 1
Date_ 20100312
Time 10.26
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 203
DW 60.800 usec
DE 6.50 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.4104
3.8690
6.8720
6.8785
6.8942
6.9007
6.9184
6.9194
7.0387
7.0451
7.2625
7.2679
7.2724
7.3999
7.4221
7.7404
7.7609
MeO
O
3.00
2.99
2.02
1.00
2.19
0.98
1.99
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5 ppm




BRUKER
NAME xfg20100Dm
EXPNO 1
PROCNO 1
Date_ 20100114
Time_ 9.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 2g30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700315 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cl
O
2.3754
6.8734
7.0241
7.0460
7.2942
7.3096
7.3278
7.3488
7.6959
7.7173
3.00
0.95
0.97
3.93
1.97
ppm

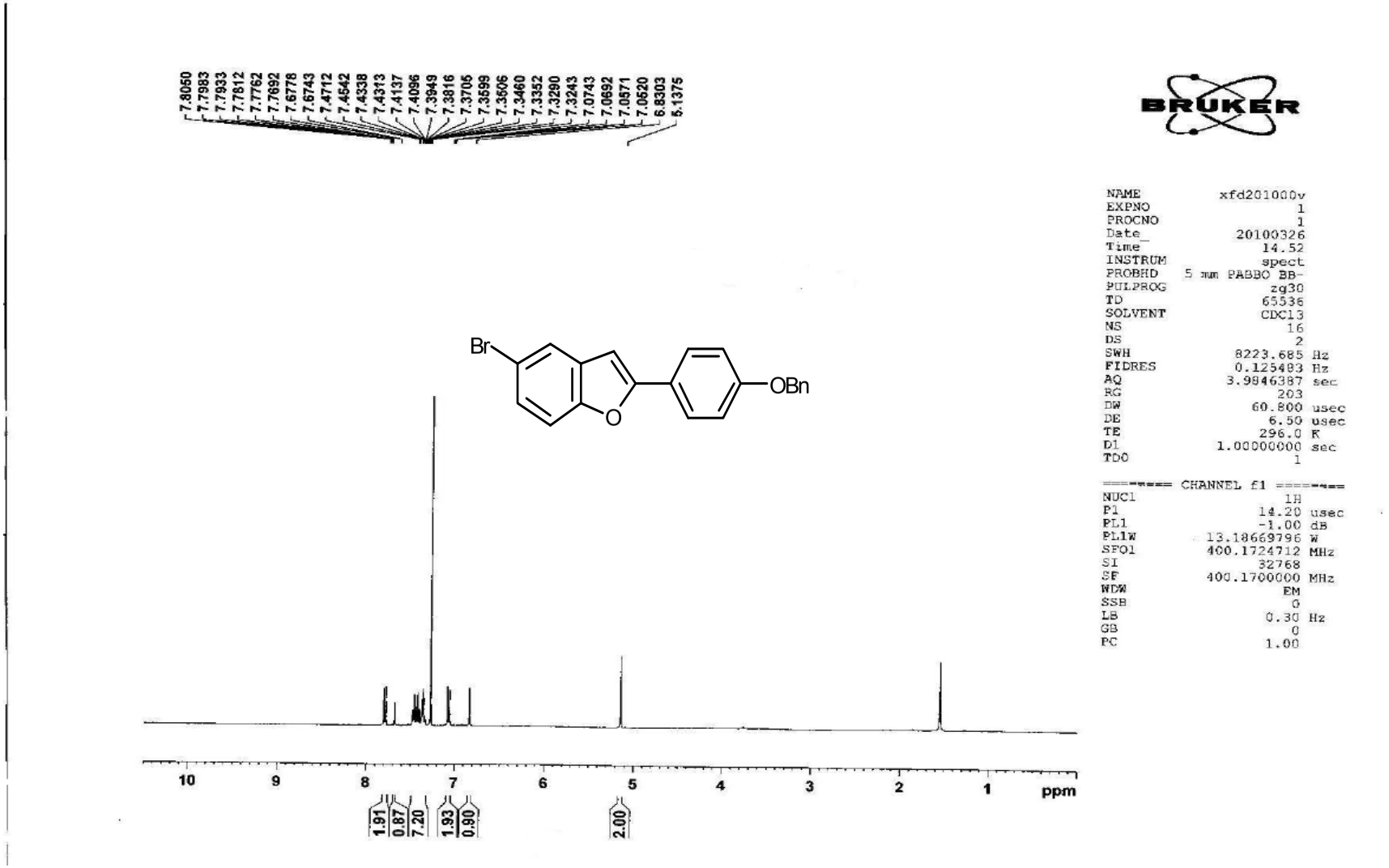


BRUKER

NAME	xfd2010001
EXPNO	1
PROCNO	1
Date_	20100113
Time	10.36
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	203
DW	60.800 usec
DE	6.50 usec
TE	287.2 K
D1	1.00000000 sec
TD0	1

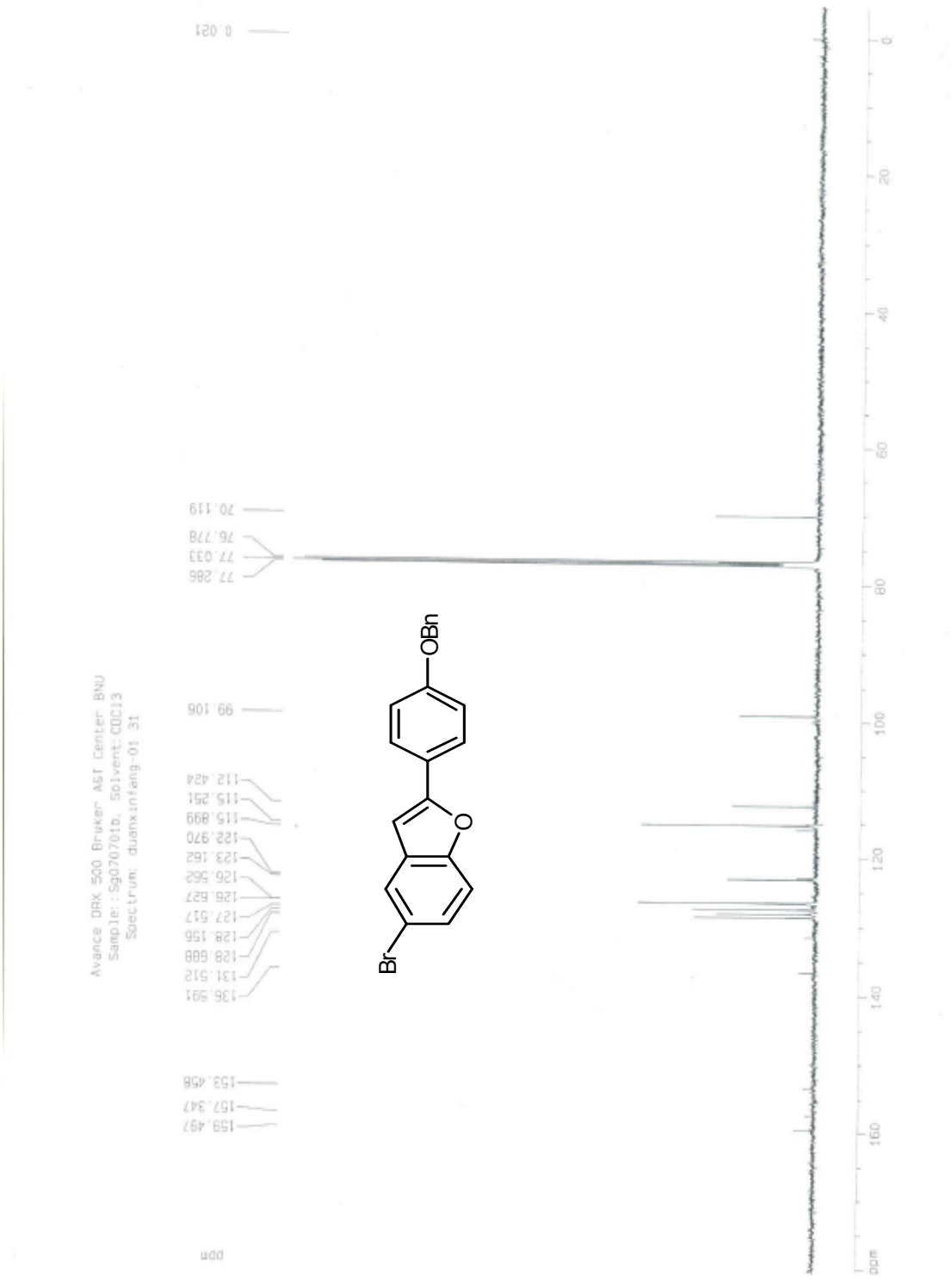
======== CHANNEL f1 ========

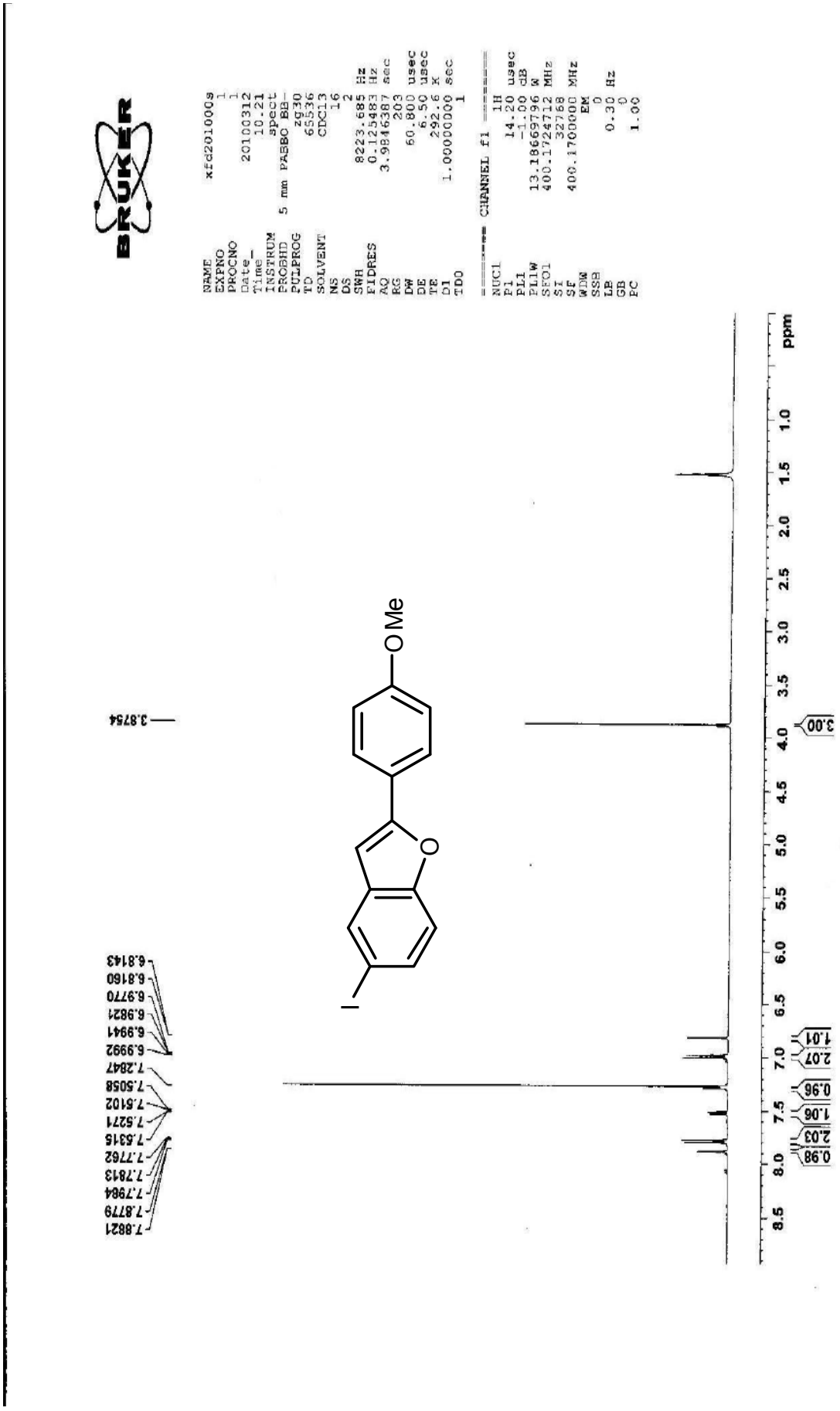
NUC1	1H
P1	14.20 usec
PL1	-1.00 dB
PL1W	13.18669796 W
SFO1	400.1724712 MHz
SI	32768
SF	400.1700306 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

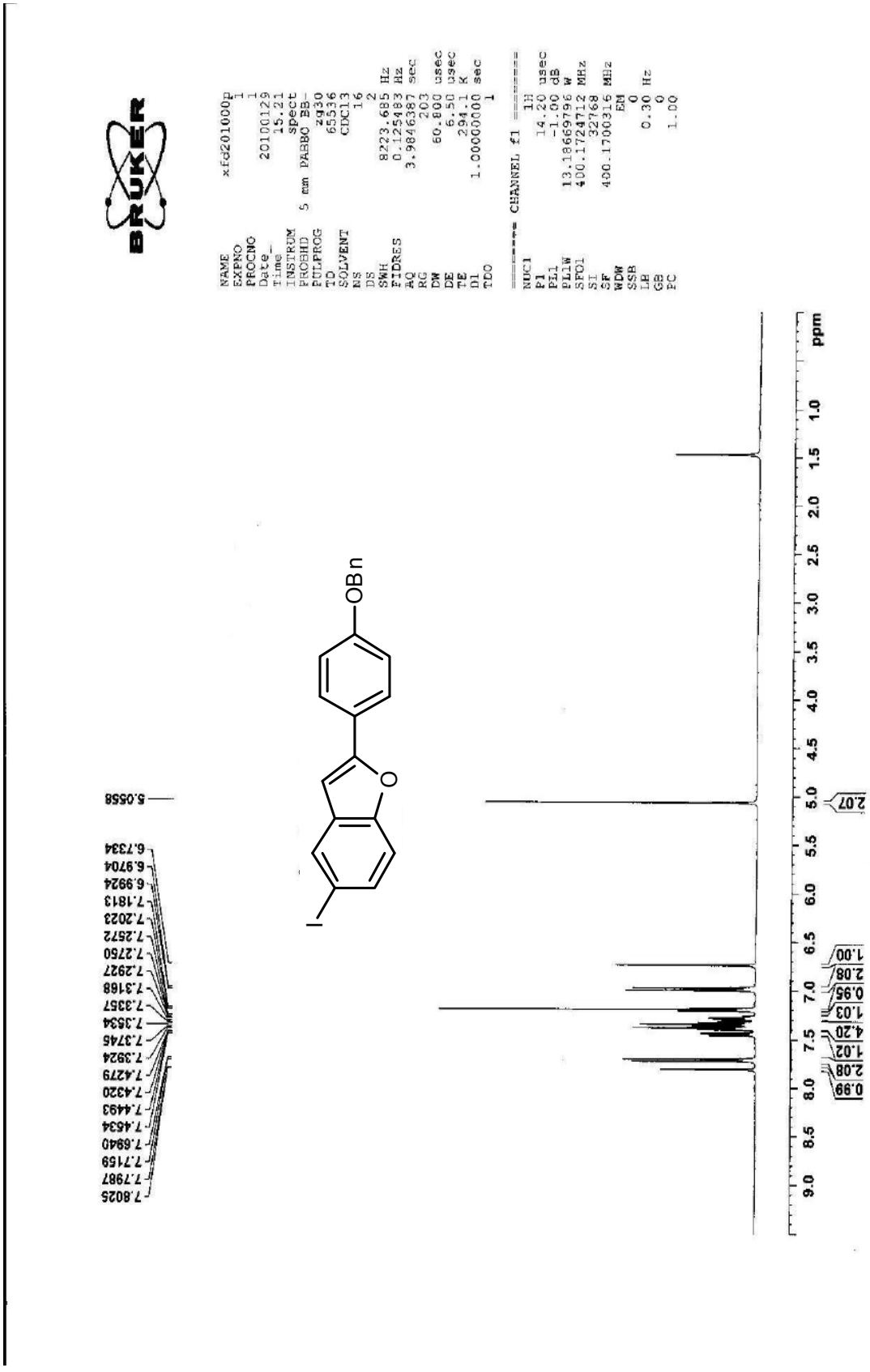


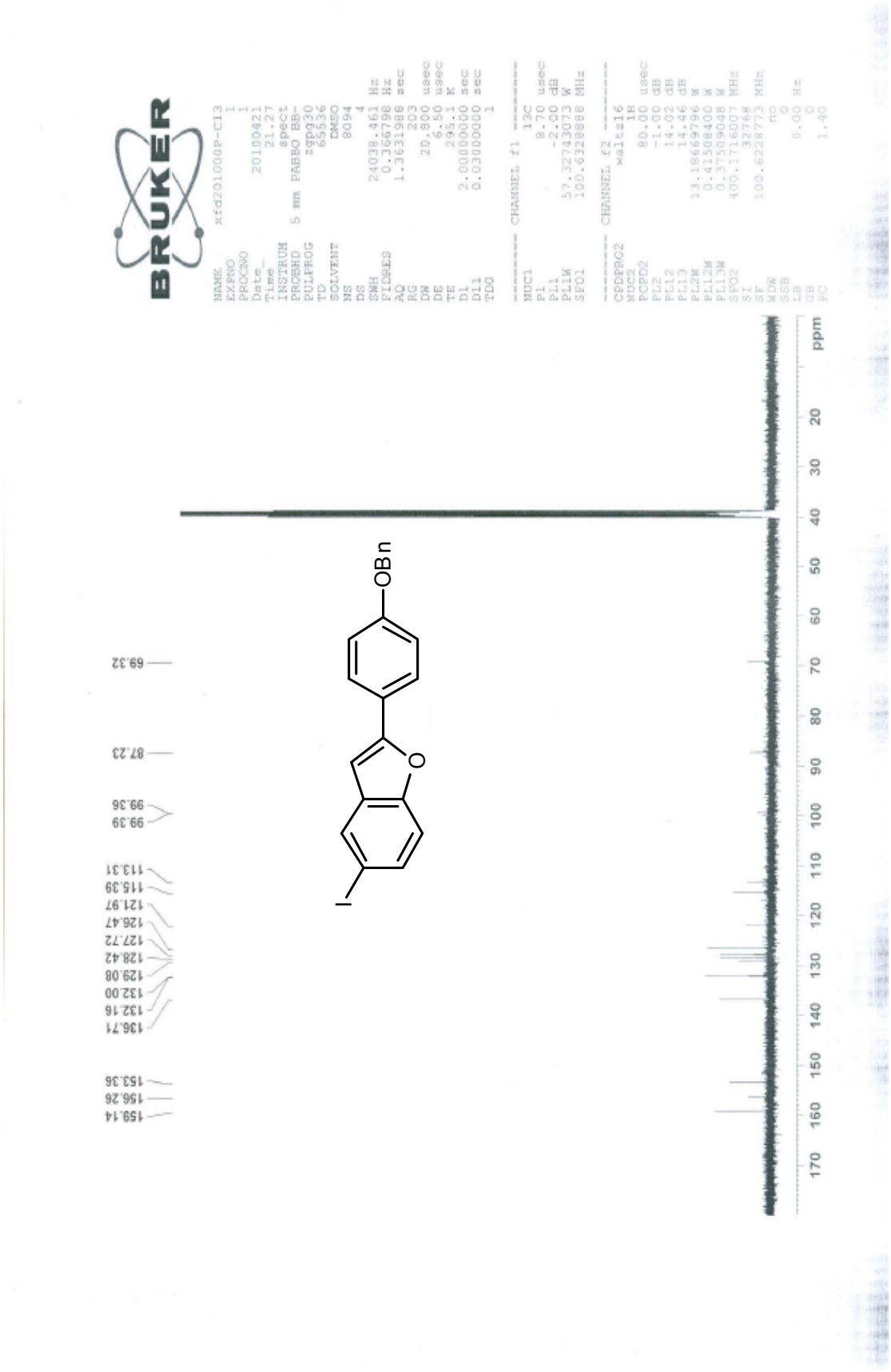
BRUKER

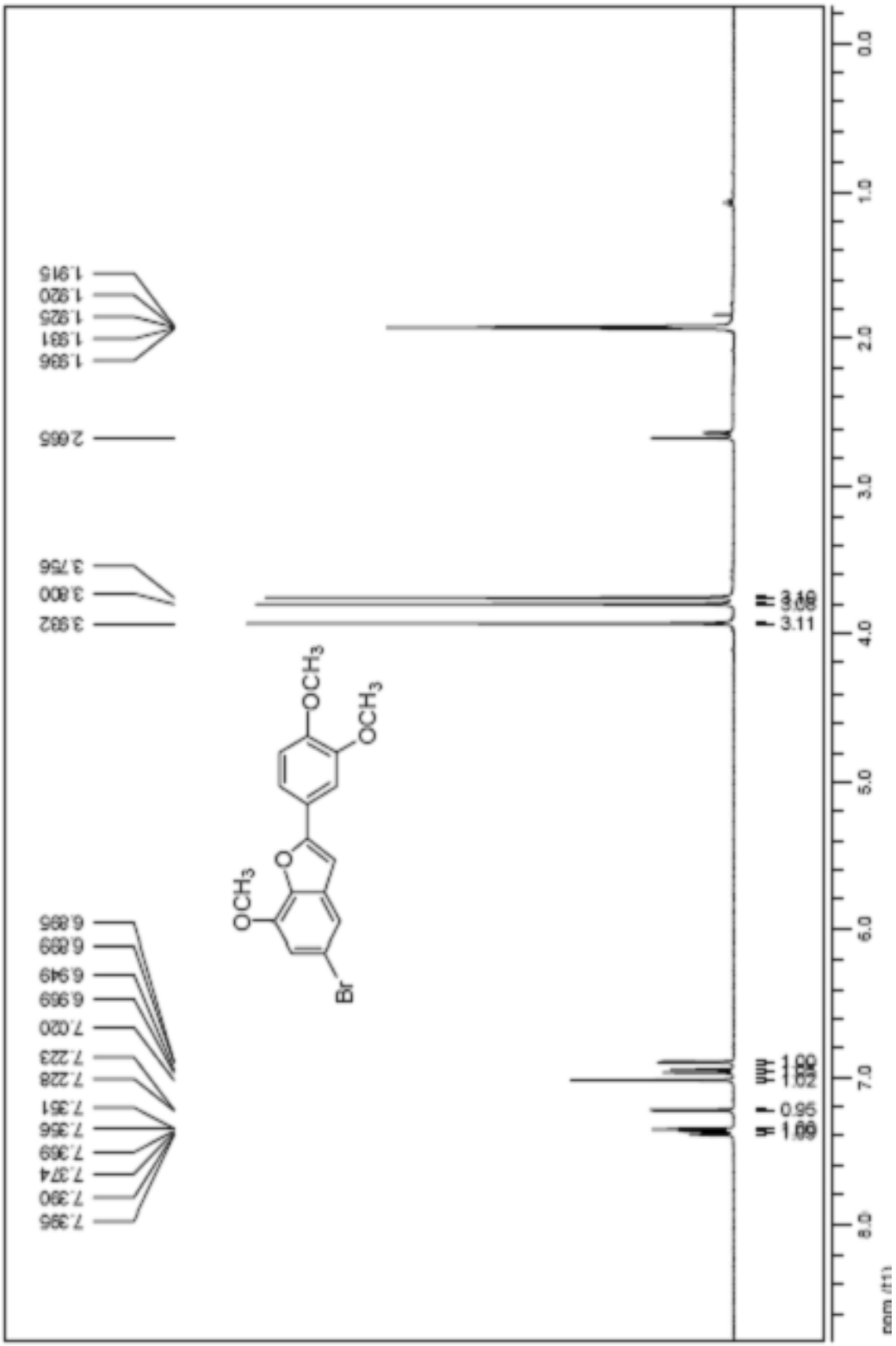
NAME	xfd201000v
EXPNO	1
PROCNO	1
Date_	20100326
Time	14.52
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	296.0 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	14.20 usec
PL1	-1.00 dB
PL1W	13.18669796 W
SFO1	400.1724712 MHz
SI	32768
SF	400.1700000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

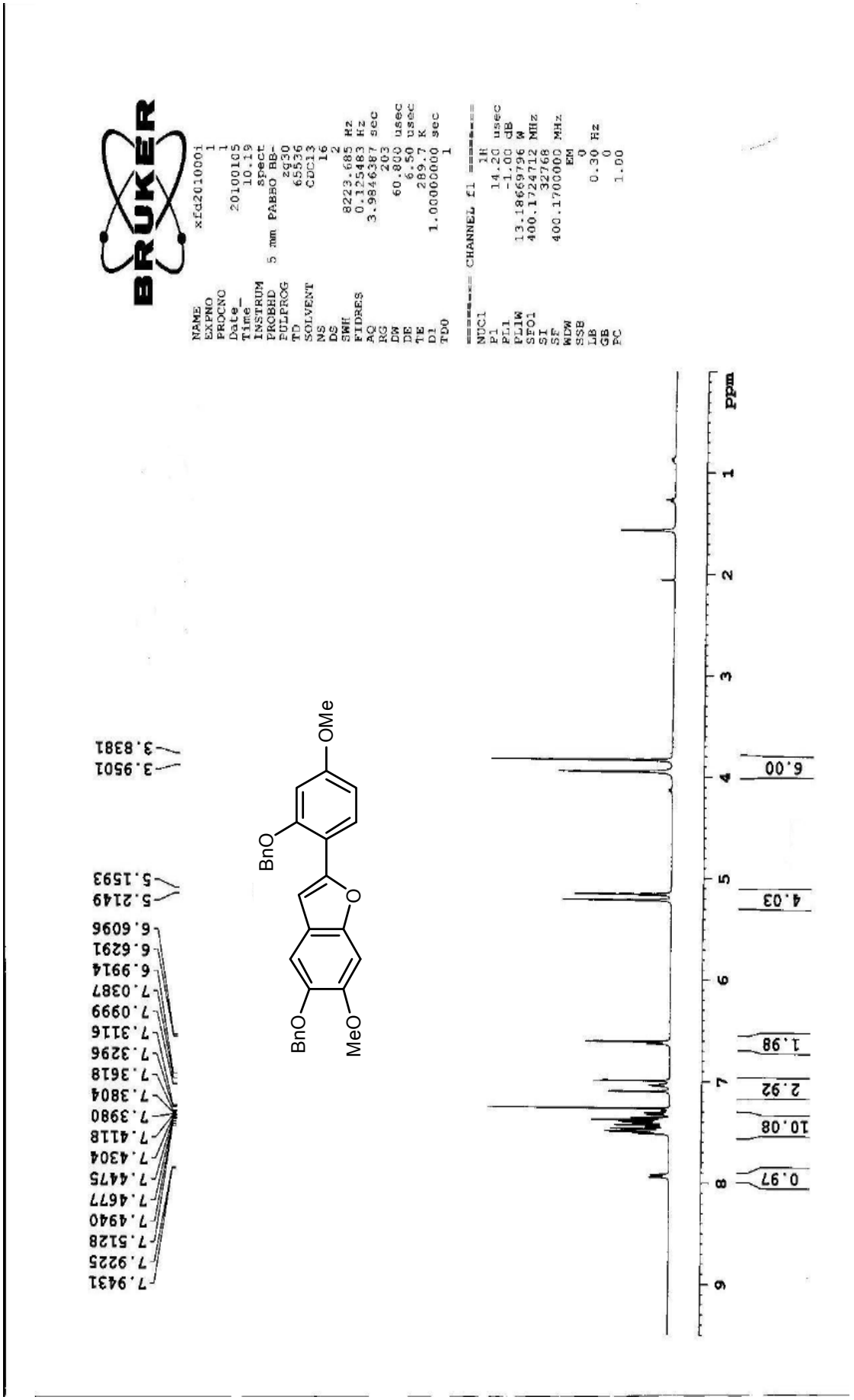

OBn
Br
O
Avance DRX 500 Bruker AST Center BNU
Sample: : Sg0707018, Solvent: CDCl3
Spectrum: duanxinfang-01 31
ppm

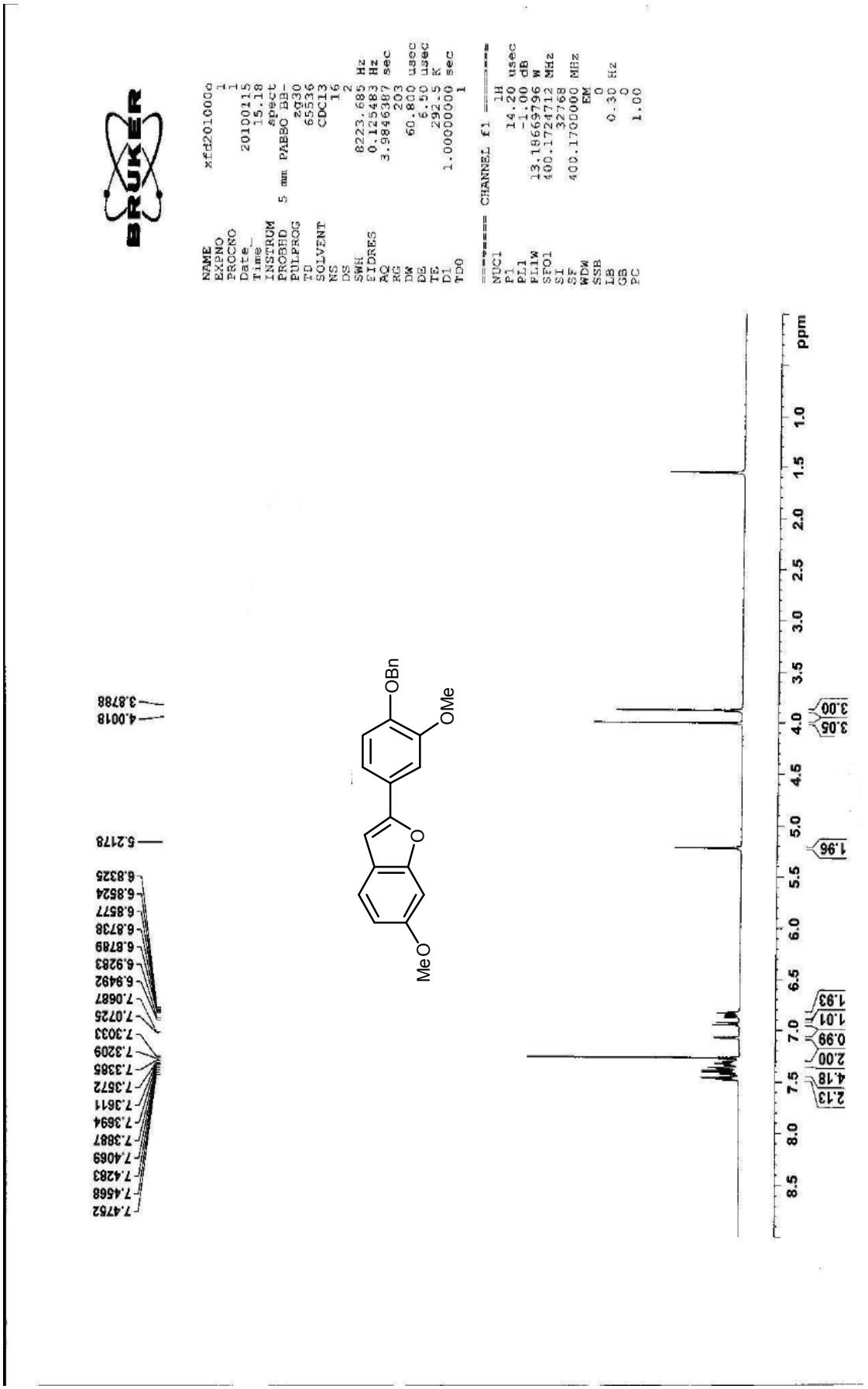

BRUKER
NAME xfg201000s
EXPNO 1
PROCNO 1
Date_ 20100312
Time 10.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.8754
7.8821
7.8779
7.7984
7.7813
7.7762
7.5315
7.5271
7.5102
7.5058
7.2847
6.9992
6.9941
6.9821
6.9770
6.8160
6.8143
O
Me
I
3.00
1.01
2.07
0.96
1.06
2.03
0.98
ppm

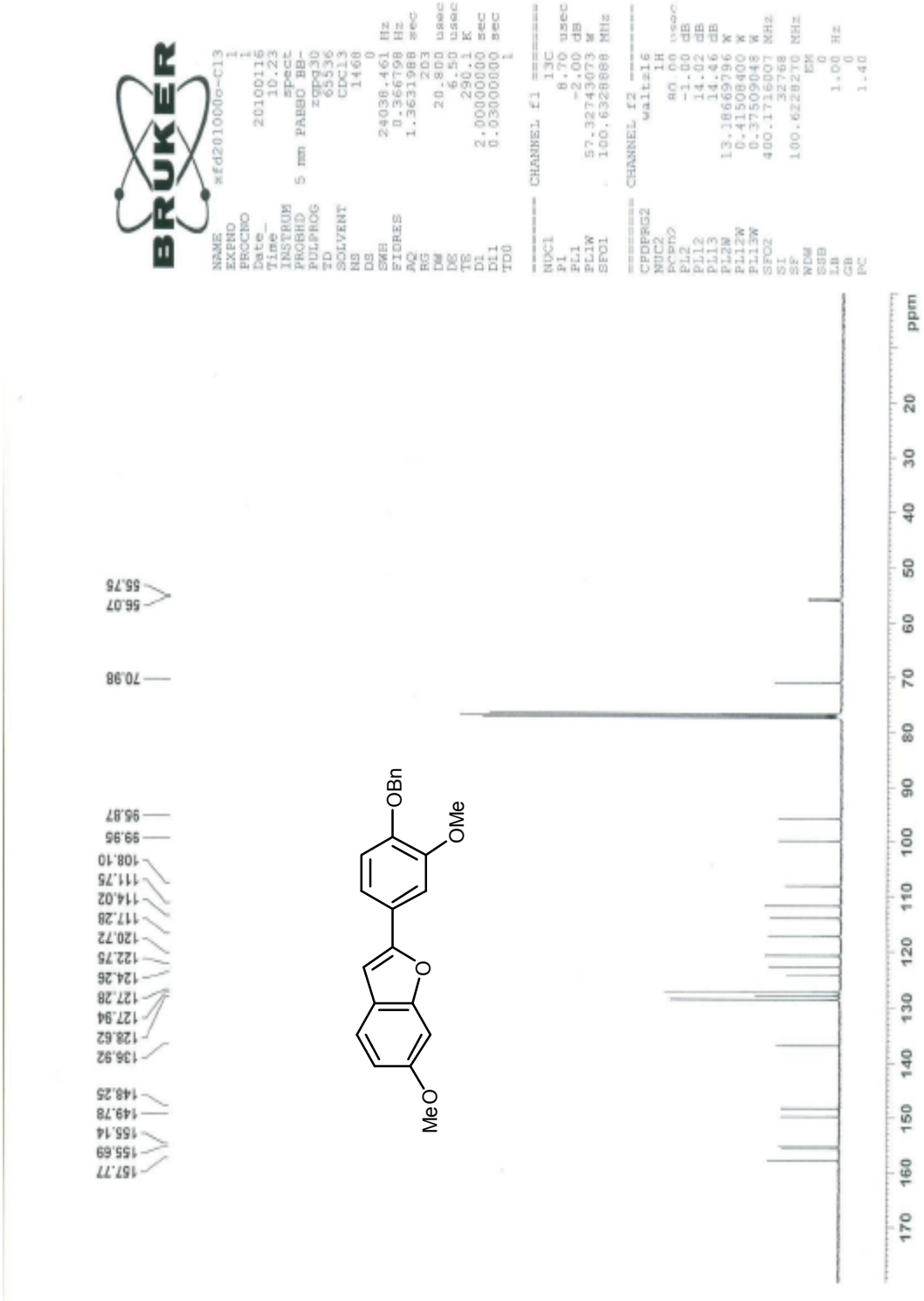

BRUKER
NAME xfg201000p
EXPNO 1
PROCNO 1
Date_ 20100129
Time_ 15.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700316 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OBn
O
I
5.0558
6.7334
6.9704
6.9924
7.1813
7.2023
7.2572
7.2750
7.2927
7.3168
7.3357
7.3534
7.3745
7.3924
7.4279
7.4320
7.4493
7.4534
7.6940
7.7159
7.7987
7.8025
2.07
1.00
2.08
0.95
1.03
4.20
1.02
2.08
0.99
ppm

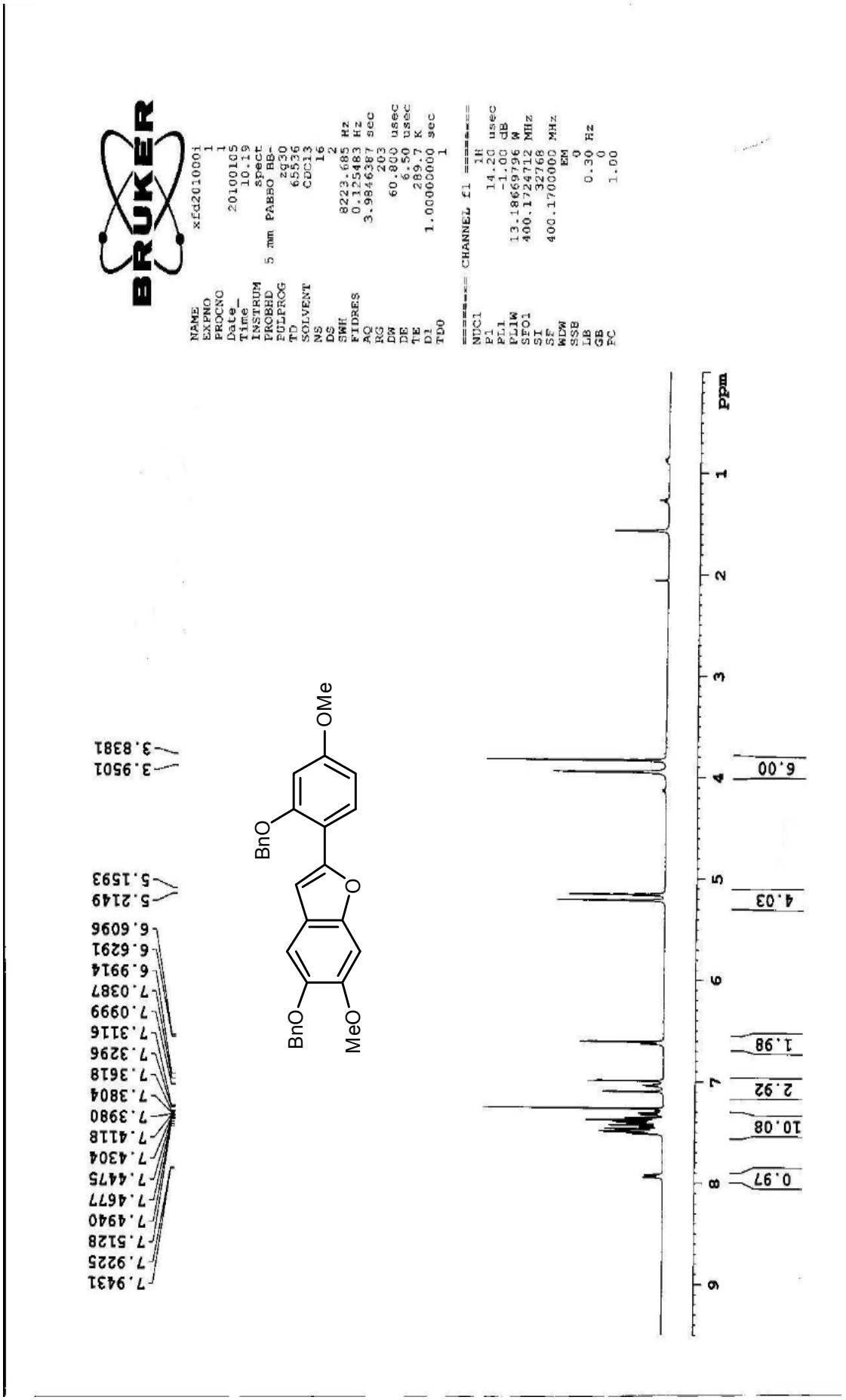

BRUKER
OBn
O
I
69.32
87.23
99.36
99.39
113.31
115.39
121.97
126.47
127.72
128.42
129.08
132.00
132.16
136.71
153.36
156.26
159.14
ppm

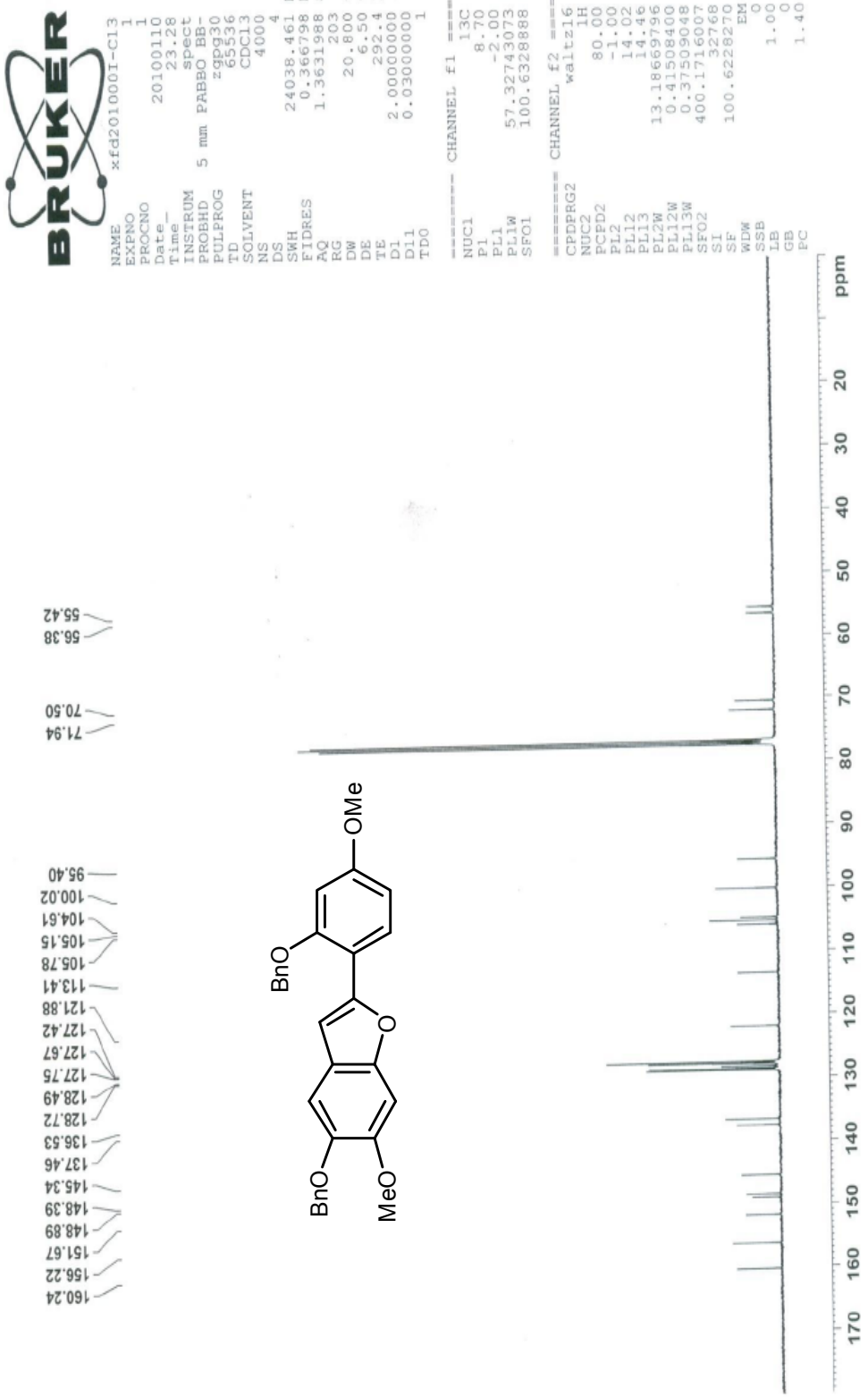

1.915
1.920
1.925
1.931
1.936
2.665
3.756
3.800
3.882
3.11
0.95
6.885
6.889
6.949
6.969
7.020
7.223
7.228
7.351
7.356
7.369
7.374
7.390
7.395
OCH3
OCH3
OCH3
O
Br
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
ppm (t1)

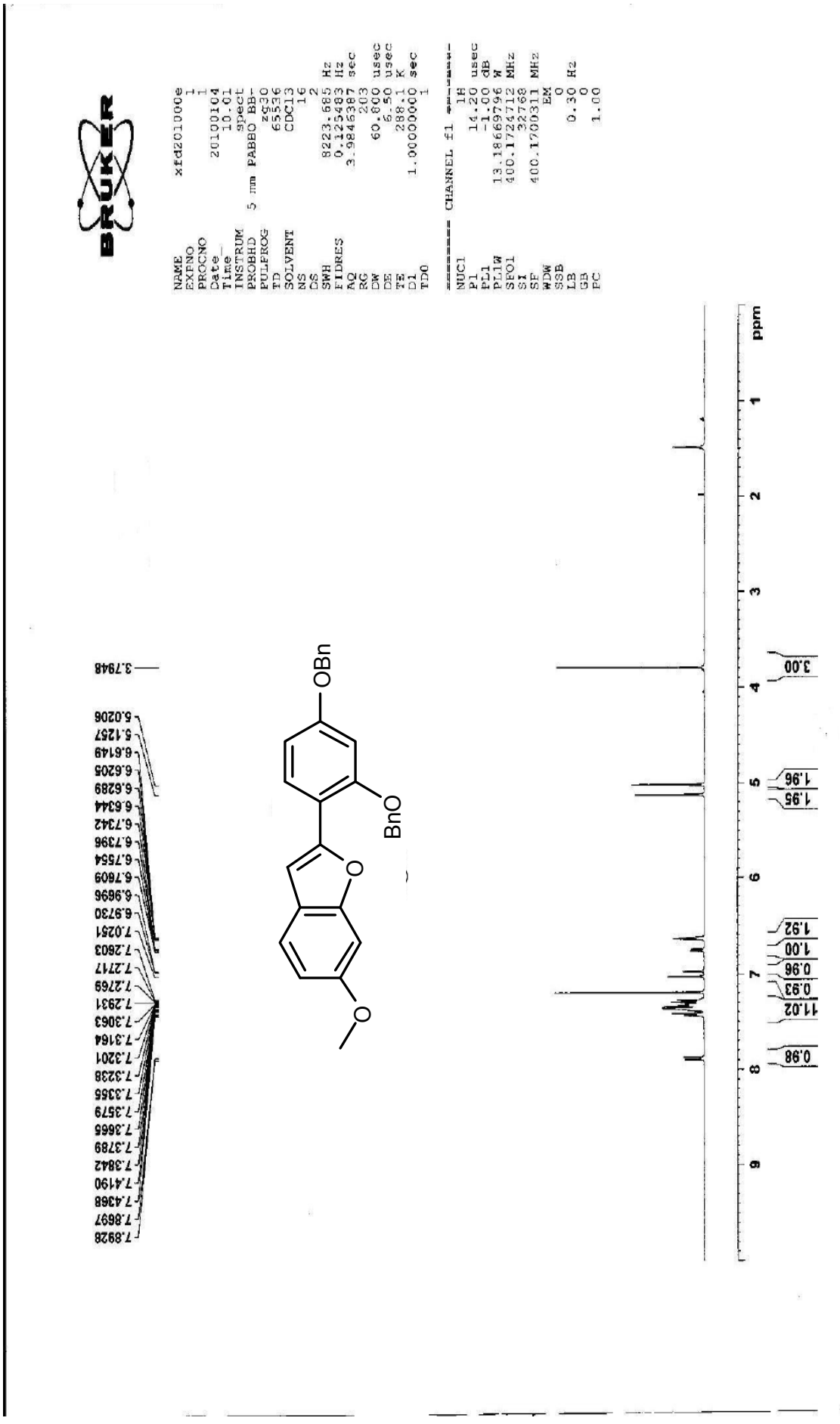

BRUKER
NAME xfd201000i
EXPNO 1
PROCNO 1
Date_ 20100105
Time 10.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 289.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.8381
3.9501
5.1593
5.2149
6.6096
6.6291
6.9914
7.0387
7.0999
7.3116
7.3296
7.3618
7.3804
7.3980
7.4118
7.4304
7.4475
7.4677
7.4940
7.5128
7.9225
7.9431
BnO
OMe
BnO
MeO
O
6.00
4.03
1.98
2.92
10.08
0.97
ppm

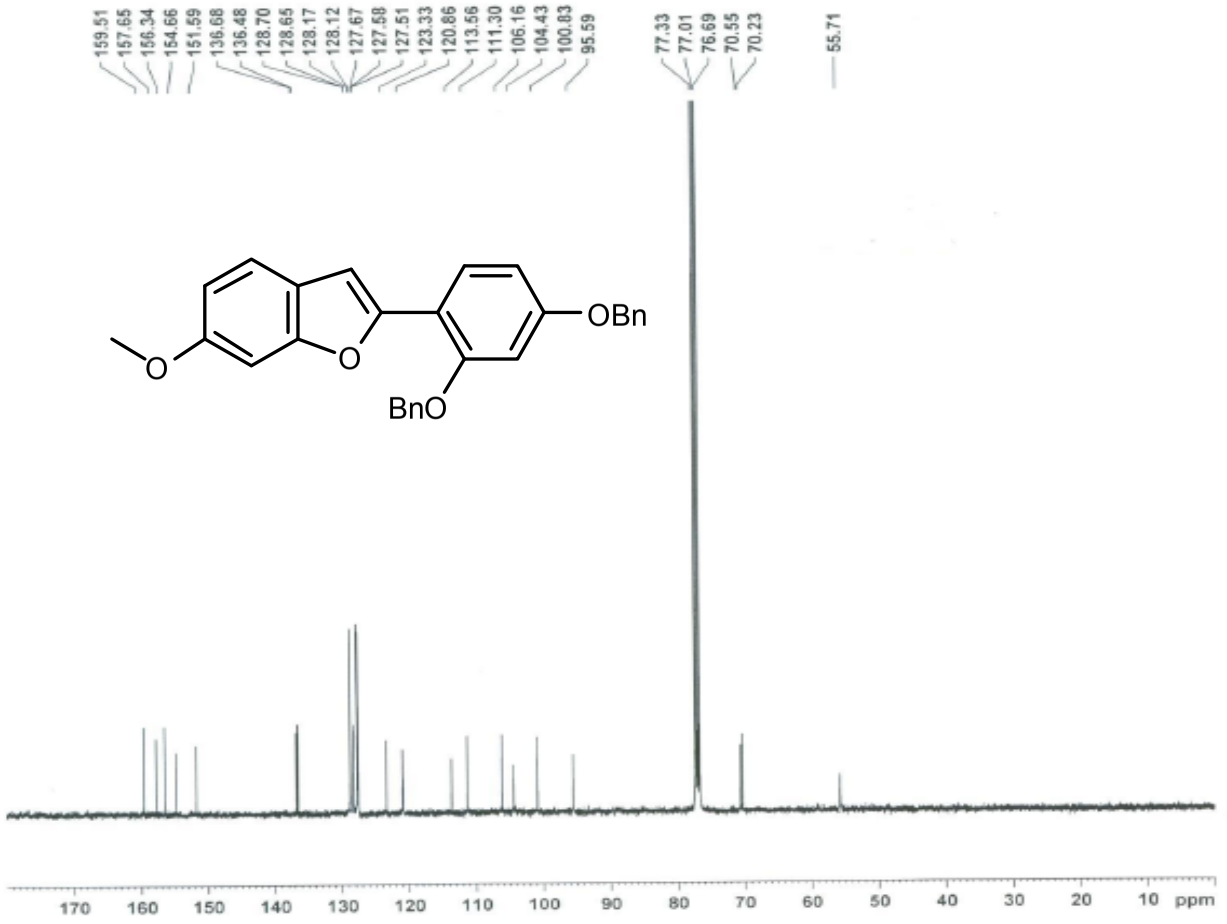



BRUKER
NAME xfd2010000o-c13
EXPNO 1
PROCNO 1
Date_ 20100116
Time 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1460
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 290.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.02 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.41508400 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OBn
OMe
MeO
55.76
56.07
70.98
95.87
99.96
108.10
111.75
114.02
117.28
120.72
122.75
124.26
127.28
127.94
128.62
136.92
148.25
149.78
155.14
155.69
157.77
ppm



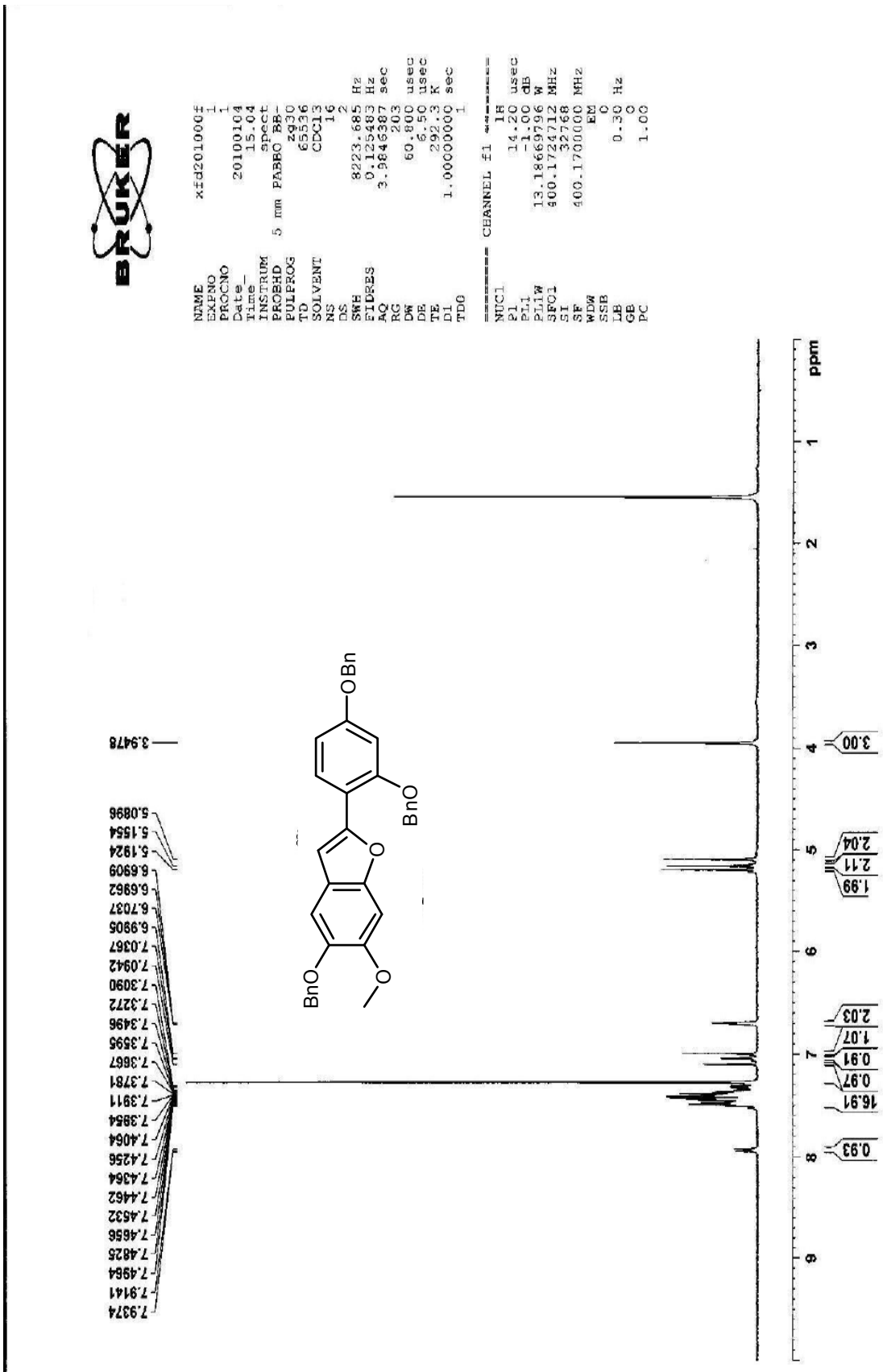

BRUKER
NAME xfd20100I-C13
EXPNO 1
PROCNO 1
Date_ 20100110
Time_ 23.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 292.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.02 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.41508400 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm
55.42
56.38
70.50
71.94
95.40
100.02
104.61
105.15
105.78
113.41
121.88
127.42
127.67
127.75
128.49
128.72
136.53
137.46
145.34
148.39
148.89
151.67
156.22
160.24
BnO
OMe
O
BnO
MeO

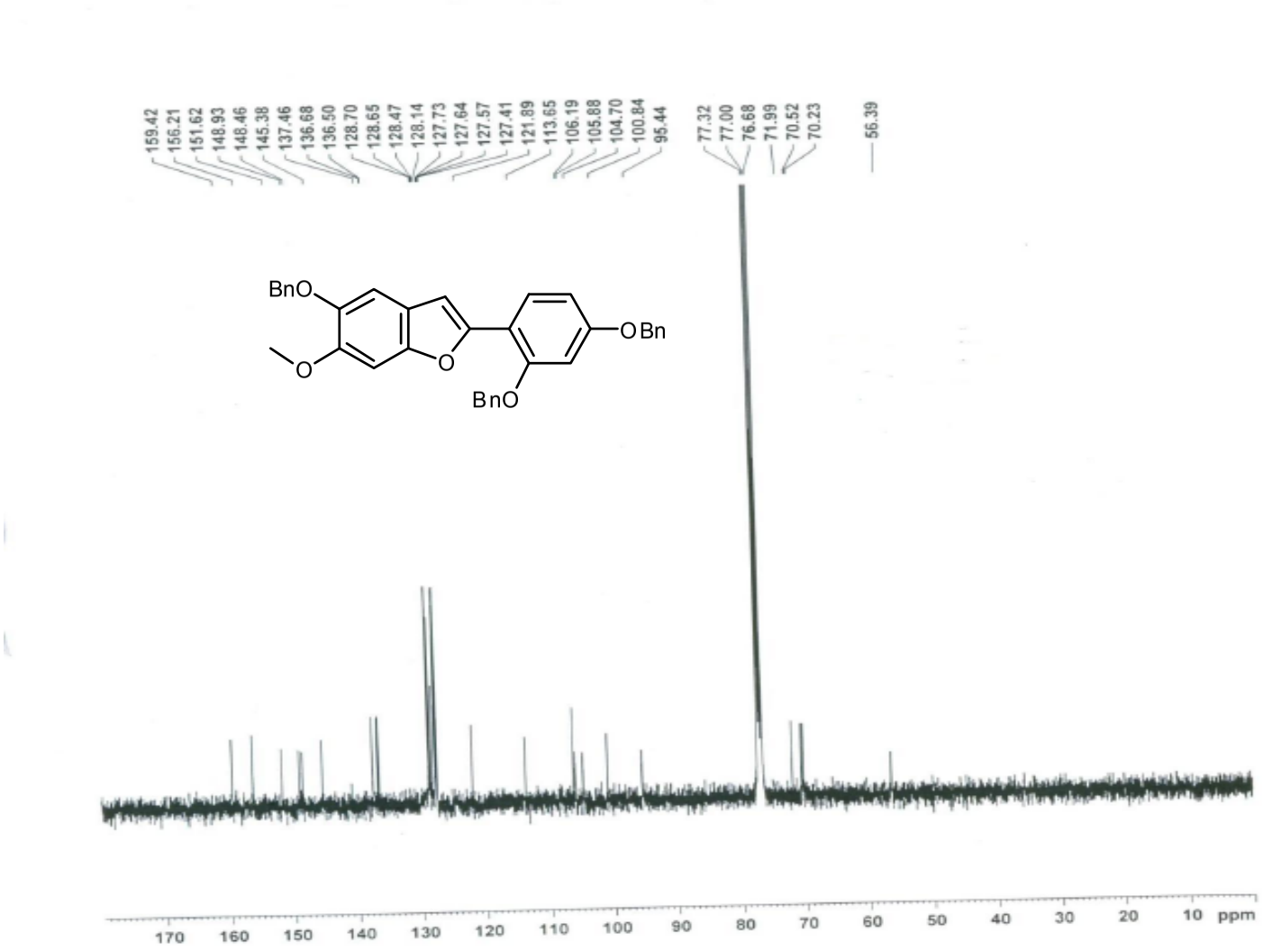




BRUKER

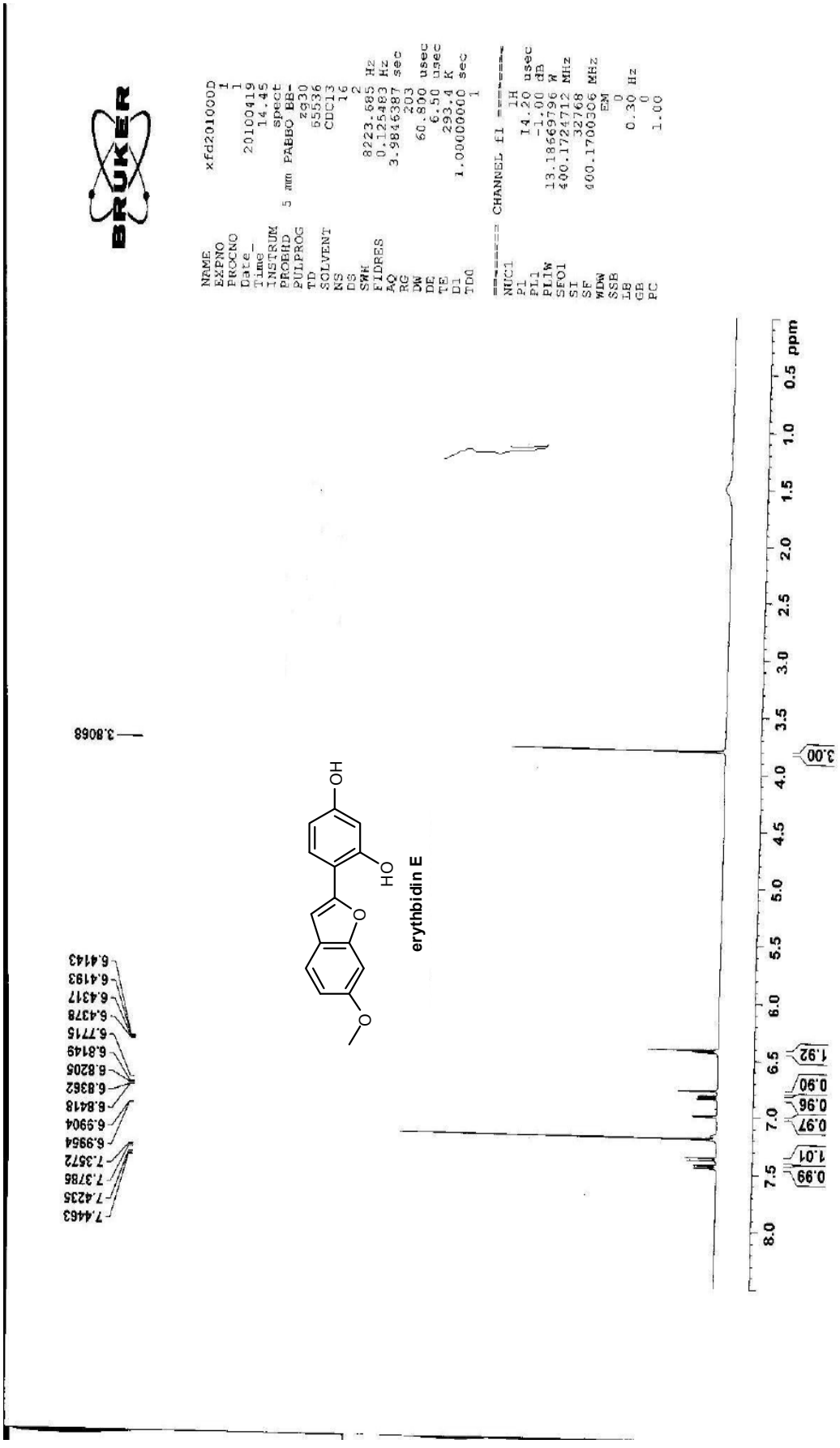
NAME	xfd201000e-C13	
EXPNO	1	
PROCNO	1	
Date_	20100106	
Time	2.27	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	3000	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	20.800	usec
DE	6.50	usec
TE	295.6	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	8.70	usec
PL1	-2.00	dB
PL1W	57.32743073	W
SFO1	100.6328888	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	-1.00	dB
PL12	14.02	dB
PL13	14.46	dB
PL2W	13.18669796	W
PL12W	0.41508400	W
PL13W	0.37509048	W
SFO2	400.1716007	MHz
SI	32768	
SF	100.6228270	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

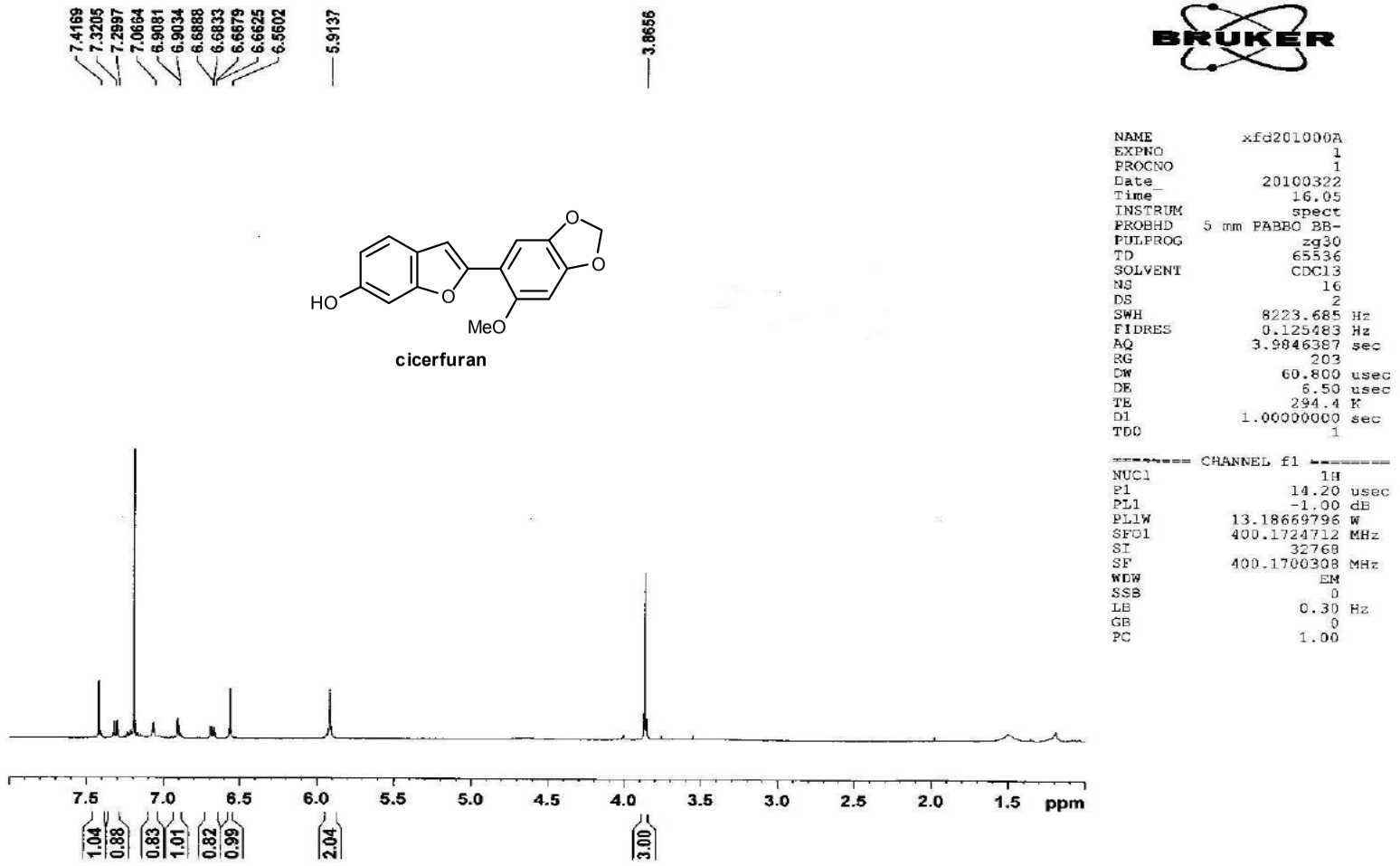

BRUKER
NAME xfd201000f
EXPNO 1
PROCNO 1
Date_ 20100104
Time_ 15.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OBn
BnO
BnO
O
O
3.9478
5.0896
5.1554
5.1924
6.6909
6.6962
6.7037
6.9905
7.0367
7.0942
7.3090
7.3272
7.3496
7.3595
7.3667
7.3781
7.3911
7.3954
7.4064
7.4256
7.4364
7.4462
7.4532
7.4656
7.4825
7.4964
7.9141
7.9374
3.00
1.99
2.11
2.04
2.03
1.07
0.91
0.97
16.91
0.93
ppm



BRUKER

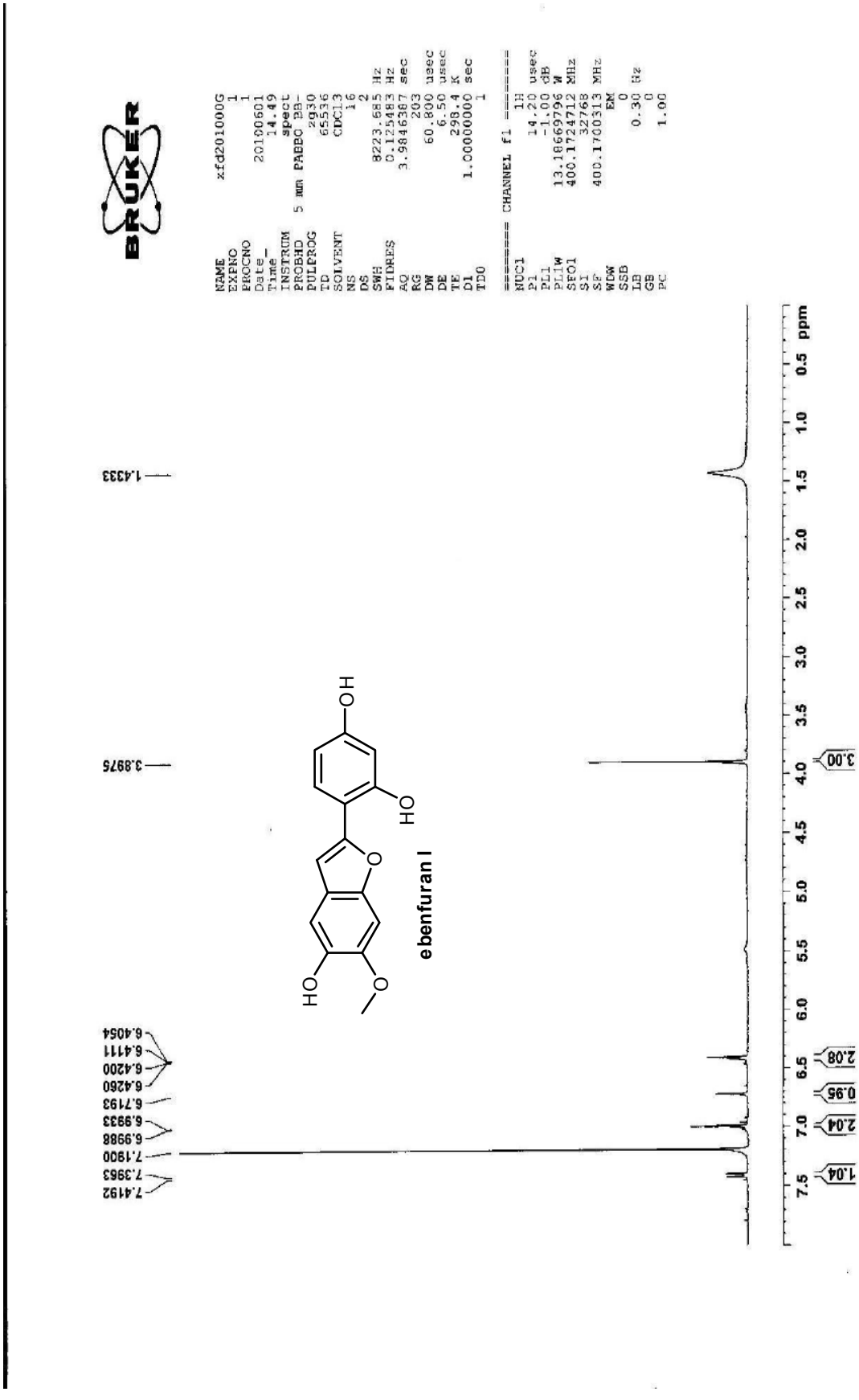
NAME	xfd201000f-C13
EXPNO	1
PROCNO	1
Date_	20100106
Time	5.23
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3000
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.70 usec
PL1	-2.00 dB
PL1W	57.32743073 W
SFO1	100.6328888 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.02 dB
PL13	14.46 dB
PL2W	13.18669796 W
PL12W	0.41508400 W
PL13W	0.37509048 W
SFO2	400.1716007 MHz
SI	32768
SF	100.6228270 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


BRUKER
NAME xfd20100DD
EXPNO 1
PROCNO 1
Date_ 20100419
Time 14.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 2930
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700306 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.8068
7.4463
7.4235
7.3786
7.3572
6.9954
6.9904
6.8418
6.8362
6.8205
6.8149
6.7715
6.4378
6.4317
6.4193
6.4143
OH
HO
O
O
erythbidin E
3.00
1.92
0.90
0.96
0.97
1.01
0.99
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5 ppm



BRUKER

NAME	xfd201000A	
EXPNO	1	
PROCNO	1	
Date_	20100322	
Time	16.05	
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	203	
DW	60.800	usec
DE	6.50	usec
TE	294.4	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	14.20	usec
PL1	-1.00	dB
PL1W	13.18669796	W
SFO1	400.1724712	MHz
SI	32768	
SF	400.1700308	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	


BRUKER
NAME xfd201000G
EXPNO 1
PROCNO 1
Date_ 20100601
Time_ 14.49
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 203
DW 60.800 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700313 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.4333
3.8975
6.4054
6.4111
6.4200
6.4260
6.7193
6.9933
6.9988
7.1800
7.3963
7.4192
OH
HO
O
HO
O
ebenfuran I
3.00
2.08
0.95
2.04
1.04
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm