A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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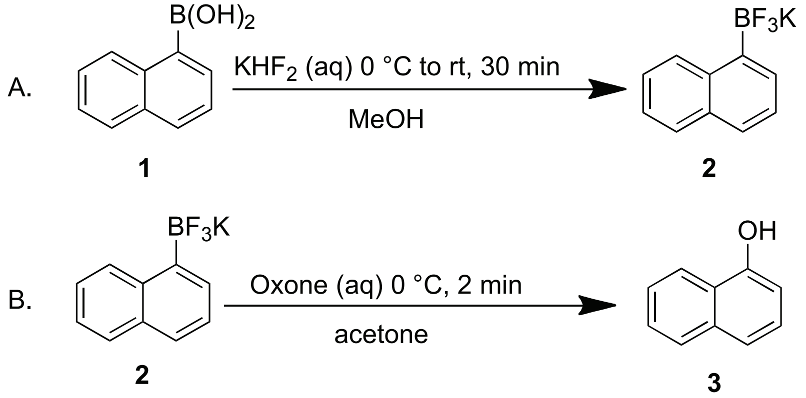
Synthesis of 1-Naphthol via Oxidation of Potassium 1-Naphthyltrifluoroborate



Submitted by Gary A. Molander, Sabahat Zahra Siddiqui, and Nicolas Fleury-Brégeot.[1]
Checked by David Hughes.

1. Procedure

*A. Potassium 1-Naphthyltrifluoroborate (**2**).* A 1-L round-bottomed flask equipped with a 4-cm oval PTFE-coated magnetic stir bar and a thermocouple thermometer (Note 1) is charged with 1-naphthaleneboronic acid (22.0 g, 128 mmol, 1.0 equiv) and methanol (80 mL) (Notes 2 and 3). The solution is cooled to 5 ºC using an ice-bath. A solution of KHF_2 (30.3 g dissolved in 100 mL water, 388 mmol, 3.0 equiv) is added in 5 portions over 10 min, resulting in a thick white slurry (Note 4). The ice bath is removed and the mixture is stirred for 20 min (Note 5). The stir bar is removed and the mixture is concentrated by rotary evaporation (45 ºC bath temp, 100 to 20 mmHg) (Note 6). Acetonitrile (2 x 200 mL) flushes are used to azeotropically remove the remaining water, with the mixture concentrated by rotary evaporation after each flush (45 ºC bath temp, 100 to 20 mmHg) (Notes 7 and 8). The flask is equipped with a 3-cm oval PTFE-coated magnetic stir bar and acetonitrile (300 mL) is added. The flask is placed in a heating mantle and the stirred mixture is heated at reflux for 90 min (Notes 9 and 10). After cooling to ambient temperature over 90 min, the resulting solids are removed by filtration through a 150-mL medium porosity sintered glass funnel and rinsed with acetonitrile (2 x 50 mL)

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(Note 11). The filtrate is concentrated by rotary evaporation (55 ºC bath temp, 70 mmHg) to afford potassium 1-naphthyltrifluoroborate (**2**) as a white solid (29.9 g) as a hemi-acetonitrile solvate (Note 12). The solid is heated under vacuum (70 mmHg) for 18–24 h at 90 ºC to provide the de-solvated product (26.5 g, 89% yield) (Notes 13 and 14).

B. 1-Naphthol (3). A 1-L 3-necked round-bottom flask is equipped with a 3-cm oval PTFE-coated magnetic stir bar and a 500-mL addition funnel. The two side necks are sealed with septa, one of which is pierced with a thermocouple thermometer (Note 1). The flask is charged with potassium 1-naphthyltrifluoroborate (26.0 g, 111 mmol, 1.0 equiv) and acetone (300 mL) (Note 15). The mixture is stirred to dissolve all solids, then cooled to 3 ºC using an ice bath. The addition funnel is charged with a solution of Oxone® (72 g dissolved in 300 mL water, 117 mmol, 1.05 equiv), which is added to the flask over 10 min (Note 16). The reaction mixture is stirred for an additional 5 min, then quenched with aq HCl (0.1 M, 360 mL, 0.3 equiv), added in one portion. The contents are transferred to a 2-L separatory funnel and extracted with dichloromethane (3 x 250 mL). The combined organic layers are washed with half-saturated aq. NaCl (200 mL), then dried by filtering through a bed of Na_2SO_4 (150 g) in a 350-mL sintered glass funnel. The filtrate is concentrated in portions in a 1-L flask by rotary evaporation (40 ºC, 200 to 20 mmHg) to an orange oil, then flushed with toluene (75 mL) and concentrated (50 ºC, 20 mmHg) to provide a brown wet solid (40 g). The product is recrystallized in the same flask by adding 15 mL of toluene and warming in a 60 ºC water bath to dissolve all solids. A 3-cm oval PTFE-coated magnetic stir bar is added and the flask is equipped with a 150-mL addition funnel. The stirred mixture is allowed to cool to room temperature, upon which crystallization occurs. *n*-Heptane (140 mL) is added via the addition funnel over 1 h. After stirring for 4 h, the suspension is filtered through a 150-mL medium porosity sintered glass funnel, washed with 4:1 *n*-heptane:toluene (2 x 15 mL), and air-dried to afford 1-naphthol (12.0 g) as an off-white to slightly pink solid. A second crop of product is obtained as follows. The filtrate and washes from the initial crystallization are concentrated by rotary evaporation (50 ºC, 20 mmHg) in a l-L flask to afford 4 g of solids. Toluene (15 mL) is added along with a 3-cm oval PTFE-coated magnetic stir bar. The mixture is warmed in a 60 ºC water bath to dissolve all solids, then allowed to cool to room temperature, upon which crystallization occurs. *n*-Heptane (60 mL) is added via a 150-mL addition funnel over 1 h, then the mixture is stirred for 14 h at room temperature. The suspension is filtered through a 150-mL medium porosity sintered glass funnel, washed with 4:1 *n*-heptane:toluene (2 x 10 mL), and air-dried to afford 1-naphthol as a pink solid (2.32 g). The two crops are combined (14.3 g, 89 % yield) (Notes 17 and 18).

2. Notes

1. The internal temperature was monitored using a J-Kem Gemini digital thermometer with a Teflon-coated T-Type thermocouple probe (12-inch length, 1/8 inch outer diameter, temperature range –200 to +250 °C).

2. The following reagents and solvents were used as received for Step A: 1-naphthaleneboronic acid (Oakwood), methanol (Fisher Optima), acetonitrile (Fisher Optima, water content 80 ppm based on Karl Fischer titration) and KHF_2 (Acros 99+%). Potassium hydrogen fluoride must be handled carefully as it is corrosive. In the presence of water, it slowly releases HF. As a consequence, when using normal glassware, etching of the flask may occur with time. The solution of KHF_2 was prepared fresh just prior to use. The submitters report the synthesis of organotrifluoroborates has also been carried out in Nalgene® bottles with a screw-cap lid fitted with a mechanical stirrer.

3. The submitters prepared 1-naphthaleneboronic acid as follows. An oven-dried 500-mL, 3-necked, round-bottomed flask, equipped with a stirring bar and a reflux condenser, is charged with oven-dried magnesium turnings (11.5 g, 459 mmol, 2.8 equiv, activated in an oven at 160 ºC for 48 h) and flushed with N_2. The magnesium turnings are covered with dry tetrahydrofuran (50 mL) and stirred at 470 rpm. The reaction is initiated by the addition of iodine crystals (60 mg, 0.14 mol %) and a small portion (approximately 0.5 mL) of neat 1-bromonaphthalene. If necessary, the reaction flask can be further warmed using an oil bath. Once the reaction is started (as evident by the disappearance of the yellowish color of iodine) the flask is heated in a 75 ºC oil bath, and 1-bromonaphthalene (34.0 g, 164 mmol, 1 equiv) in dry THF (155 mL) is added dropwise over 25 min. The dark green solution is refluxed for 17 h. The reaction is allowed to cool over 40 min, then the solution of 1-naphthalenemagnesium bromide is transferred by cannula in a dropwise fashion over 1 h to an N_2-flushed 1-L round-bottom flask containing a stirred solution of triethyl borate (42 mL, 246 mmol, 1.5 equiv) in dry THF (100 mL) placed in a dry-ice/acetone bath.

After the addition is complete, the cold bath is removed and the resulting brownish green solution is stirred for 5 h at room temperature. The color of the solution changes from dark brown to white and a precipitate is formed. The reaction flask is then placed in an ice bath and aqueous HCl (100 mL, 2 M, 1.2 equiv) is added dropwise over 10 min. The white precipitate dissolves to form a light yellow, clear solution. After 1 h stirring at room temperature, the reaction mixture is extracted with diethyl ether (2 x 150 mL) and the combined organic layers are washed with distilled water (3 x 300 mL). The organic layer is dried over anhydrous sodium sulfate (50 g) and the solvents are removed on a rotary evaporator at 35 ºC at 75 mmHg. After drying under vacuum overnight, 1-naphthaleneboronic acid is obtained as a white solid (22.0–22.9 g, 78–81% yield). If necessary, the product may be recrystallized using 200 mL of distilled water and 20 mL of THF, sonicated for 5–7 min, refrigerated overnight, filtered, and dried under vacuum. Analytical data: mp: 194–196 °C. IR (cm^{-1}): 3261, 1575, 1508, 1348, 1318, 807, 778; ^{1}H NMR (500 MHz, DMSO-d^6) δ: 7.62–7.49 (m, 3 H), 7.78–7.76 (m, 1 H), 8.02–7.91 (m, 2 H), 8.35 (m, 3 H); ^{13}C NMR (125 MHz, acetone-d^6) δ: 125.6, 125.9, 126.1, 128.7, 129.3, 129.7, 132.6, 133.4, 136.1; ^{11}B NMR (128 MHz, acetone-d^6) δ: 30.6.

4. The reaction is run open to air. The temperature rose to 15 ºC at the end of the addition. If the slurry becomes too thick to stir, it should be broken up with a spatula.

5. The submitters monitored completion of the reaction by ^{11}B-NMR. The disappearance of the boronic acid peak at 30.7 ppm indicated the completion of the reaction.

6. The pressure is reduced gradually to minimize bumping. About 120 mL of solvent was removed, leaving a residue of 97 g of wet solids in the flask.

7. The pressure is reduced gradually to minimize bumping. After each acetonitrile flush, the mixture was concentrated to 50–52 g of solids.

8. The submitters removed water by lyophilization for 24 h.

9. The mixture remains heterogeneous as the product dissolves, but the residual KHF_2 remains undissolved.

10. The submitters subjected the crude material to Soxhlet extraction using 700 mL of acetonitrile in a 1-L round-bottomed flask placed in an oil bath (102 ºC and 280 rpm) for 6 h. The filtrate was concentrated on a rotary evaporator under reduced pressure at (40 ºC, 150 mmHg) until dry material was obtained.

11. The water content of the acetonitrile solution was determined to be 570 ppm based on Karl Fischer titration. Higher levels of water can result in some dissolution of KHF_2.

12. The hemi-solvate was determined based on ^{1}H NMR analysis in acetone-d^6. The solvate remained intact upon vacuum drying 2 days at 40 ºC.

13. The submitters report the crude product can be purified by dissolving the material in acetonitrile (50 mL) and precipitating with diethyl ether (300 mL).

14. Potassium 1-naphthyltrifluoroborate has the following physical and spectroscopic properties: mp: > 300 ºC. IR (cm^{-1}): 2980, 2884, 2360, 1382, 940, 668, 651; ^{1}H NMR (400 MHz, acetone-d^6) δ: 7.30–7.36 (m, 3 H), 7.64 (d, J = 8.2 Hz, 1 H), 7.74–7.77 (m, 2 H), 8.57–8.59 (m, 1 H); ^{13}C NMR (100 MHz, acetone-d^6) δ: 124.5, 124.8, 125.9, 126.7, 128.4, 130.0 (q, $J_{C\text{-}F}$ = 3.2 Hz), 131.4, 134.5, 138.1, 147 (very broad); ^{11}B NMR (128 MHz, acetone-d^6) δ: 4.06; ^{19}F NMR (377 MHz, acetone-d^6) δ: –138.6; HRMS (ESI) *m/z* calcd. for $C_{10}H_7BF_3$ (M-K) 195.0593, found 195.0593. Anal calcd for $C_{10}H_7BF_3K$: C, 51.31; H, 3.01; Found: C, 51.50; H, 2.81.

15. The following reagents and solvents were used as received for Step B: acetone (Fisher certified ACS), Oxone® (Sigma-Aldrich), toluene (Fisher Optima), *n*-heptane (99%, ReagentPlus, Sigma-Aldrich), dichloromethane (Fisher, certified ACS reagent, stabilized) and 6 N HCl (Fisher). The solution of Oxone was prepared fresh just prior to use.

16. The reaction was carried out open to air. The temperature rose to 32 ºC during the addition. The reaction mixture becomes milky white during the addition.

17. 1-Naphthol has the following physical and spectroscopic properties: mp: 95–96 ºC (lit. mp 95 ºC^{2a}, 94 ºC^{2b}); IR (cm^{-1}): 3300, 1597, 1358, 1269, 1083, 789, 765; R_f: 0.4 (hexanes/EtOAc 4:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ: 5.17 (s, 1 H), 6.83 (dd, J = 7.4, 0.7 Hz, 1 H), 7.30–7.34 (m, 1 H), 7.43 (d, J = 8.3 Hz, 1 H), 7.48–7.53 (m, 2 H), 7.81–7.85 (m, 1 H), 8.17–8.20 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ: 108.8, 121.0, 121.7, 124.6, 125.5, 126.0, 126.7, 127.9, 135.0, 151.5; MS (EI, 70eV) 144 (M+, 100), 116 (62), 115 (81), 89 (22), 63 (18); Purity by GC: 99% (t_R = 11.2 min, conditions: Agilent DB35MS column; 30 m x 0.25 mm; initial temp 60 °C, ramp at 20 °C/min to 280 °C, hold 15 min)

18. A yield of 87% was obtained at half scale.

Safety and Waste Disposal Information

3. Discussion

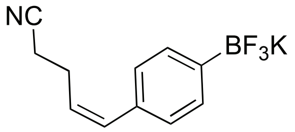
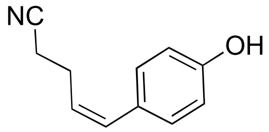
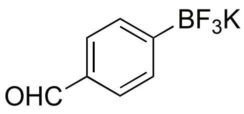
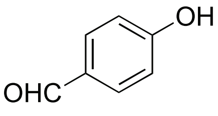
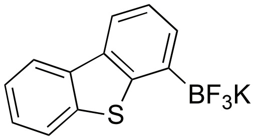
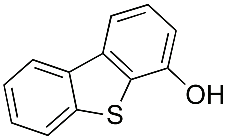
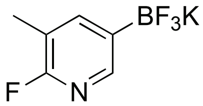
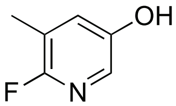
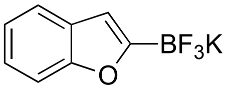
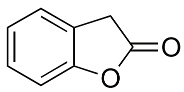
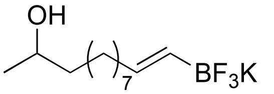
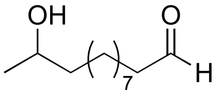
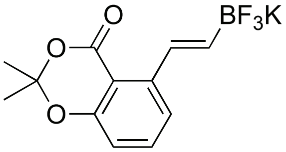
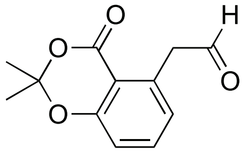
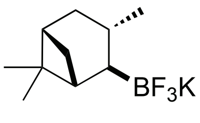
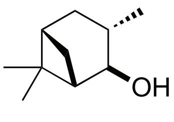
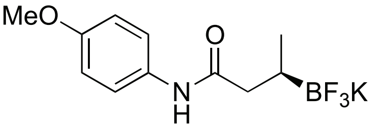
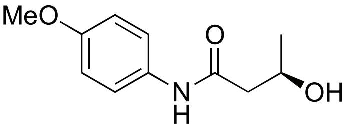
Boron containing reagents are among the most versatile building blocks used in chemistry. Historically, boranes and boronic acids are the most encountered, but over the past decades, modified boron compounds, which overcome some of the limitations associated with the earlier boron reagents, have emerged.[3]

Potassium organotrifluoroborates, quaternary boron salts, represent a relatively new class of reagents that are air and moisture stable and are efficient partners in boron-based chemical transformations.[4] They are easily prepared by addition of KHF_2 to various organoboron reagents,[5] and once synthesized they can be stored indefinitely without particular precaution. The organotrifluoroborate group acts as a boronic acid surrogate that can be carried through several chemical transformations[6] before being reacted or deprotected to yield the boronic acid.[7] These features make them very attractive reagents, and they are the subject of many studies. They are mainly used as coupling partners in the Suzuki-Miyaura reaction[8] but are also very useful reagents in other transformations.[9]

Treatment of organotrifluoroborate salts with oxone[10] represents a very efficient and practical method to oxidize these boron reagents. Oxone is an inexpensive reagent that readily oxidizes a large range of organotrifluoroborates in high yields within minutes. The rapidity, ease of execution, and non-toxicity of the oxidizing agent, associated with the advantages inherent to the potassium organotrifluoroborates, make the procedure described above a very convenient, environmentally sound, and attractive transformation. The process is exceedingly general. Aromatic trifluoroborates are transformed to phenols, alkyltrifluoroborates are converted to the corresponding alcohols in a stereospecific manner (complete retention of configuration), and alkenyltrifluoroborates can be used to access aldehydes (Table 1). These transformations occur with great chemoselectivity, as other oxidizable groups such as alkenes, aldehydes, as well as sulfides and amines remain untouched under the mild reaction conditions.

Table 1. Oxidation of Various Organotrifluoroborates with Oxone

$$\text{R-BF}_3\text{K} \xrightarrow[\text{acetone : H}_2\text{O} \\ \text{rt, 2 min}]{\text{Oxone}} \text{R-OH}$$

entry	starting material	product	isolated yield (%)
1			98
2			99
3			95
4			94
5			97
6			99
7			99
8			98
9			97

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Appendix

Chemical Abstracts Nomenclature; (Registry Number)

1-Bromonaphthalene; (90-11-9)
Triethyl borate; (150-46-9)
Magnesium; (7439-95-4)
Iodine; (7553-56-2)
1-Naphthaleneboronic acid; (13922-41-3)
Potassium hydrogen fluoride; (7789-29-9)
Potassium 1-Naphthyltrifluoroborate: Borate(1-), trifluoro-1-naphthalenyl-, potassium (1:1), (*T*-4)-; (166328-07-0)
Oxone; (70693-62-8)
1-Naphthol; (90-15-3)

Professor Gary Molander received his B.S. degree at Iowa State University in 1975, working with Professor Richard C. Larock. He obtained his Ph.D. degree in 1979 under the direction of Professor Herbert C. Brown. He joined Professor Barry Trost's group at the University of Wisconsin, Madison as a National Institutes of Health postdoctoral fellow in 1980, and in 1981 he accepted an appointment at the University of Colorado, Boulder. In 1999 he joined the faculty at the University of Pennsylvania. His research interests focus on the development of new synthetic methods for organic synthesis and natural product synthesis.

Sabahat Zahra Siddiqui was born in Lahore, Pakistan. She received her B.Sc degree from Lahore College for Women University (Roll of Honor), M.Sc degree in Organic Chemistry from Government College University, Lahore (Certificate of Merit) and M.Phil in Organic Chemistry from Punjab University, Lahore (Certificate of Merit). Currently, she is a Lecturer and a PhD scholar at Government College University, Lahore. She won an IRSIP scholarship 2011-2012 from the Higher Education Commission of Pakistan to visit the University of Pennsylvania, USA as a visiting research scholar where her research focused on synthetic methods using organotrifluoroborates under the guidance of Prof. Gary Molander.

Dr. Nicolas Fleury-Brégeot studied chemistry in France at the ESCOM (Ecole Supérieure de Chimie Organique et Minérale), where he received a Master of Science degree in 2005. He then joined the ICSN (Institut de Chimie des Substances Naturelles) in Gif-sur-Yvette where he obtained his Ph.D. in 2008 under the supervision of Dr. Angela Marinetti. After working for one year as a postdoctoral research associate in Tarragona (Spain) with Prof. Carmen Claver on asymmetric hydrogenation, he moved to Philadelphia. He is currently pursuing research in the group of Prof. Gary Molander as a postdoctoral research associate at the University of Pennsylvania. His research focuses on the development of new synthetic methods involving organotrifluoroborates.

```
NAME           32077-209
EXPNO                  4
PROCNO                 1
Date_           20121025
Time               16.42
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT          Acetone
NS                    32
DS                     2
SWH             6578.947 Hz
FIDRES          0.200774 Hz
AQ             2.4904180 sec
RG                  90.5
DW                76.000 usec
DE                  6.50 usec
TE                 300.0 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.75 usec
PL1                -2.00 dB
PL1W         13.05791473 W
SFO1         400.3324722 MHz
SI                 16384
SF           400.3300046 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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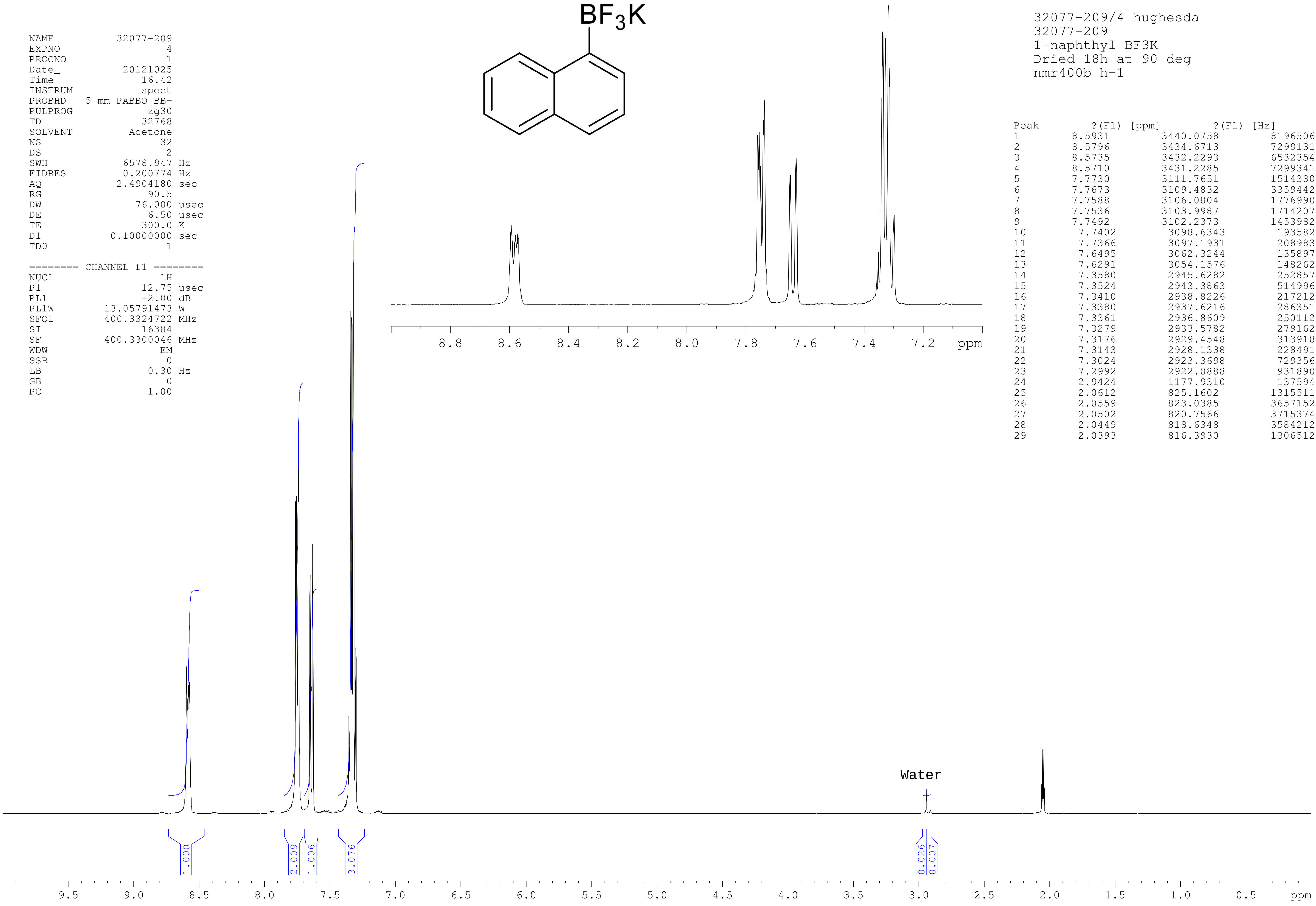


32077-209/4 hughesda
32077-209
1-naphthyl BF3K
Dried 18h at 90 deg
nmr400b h-1

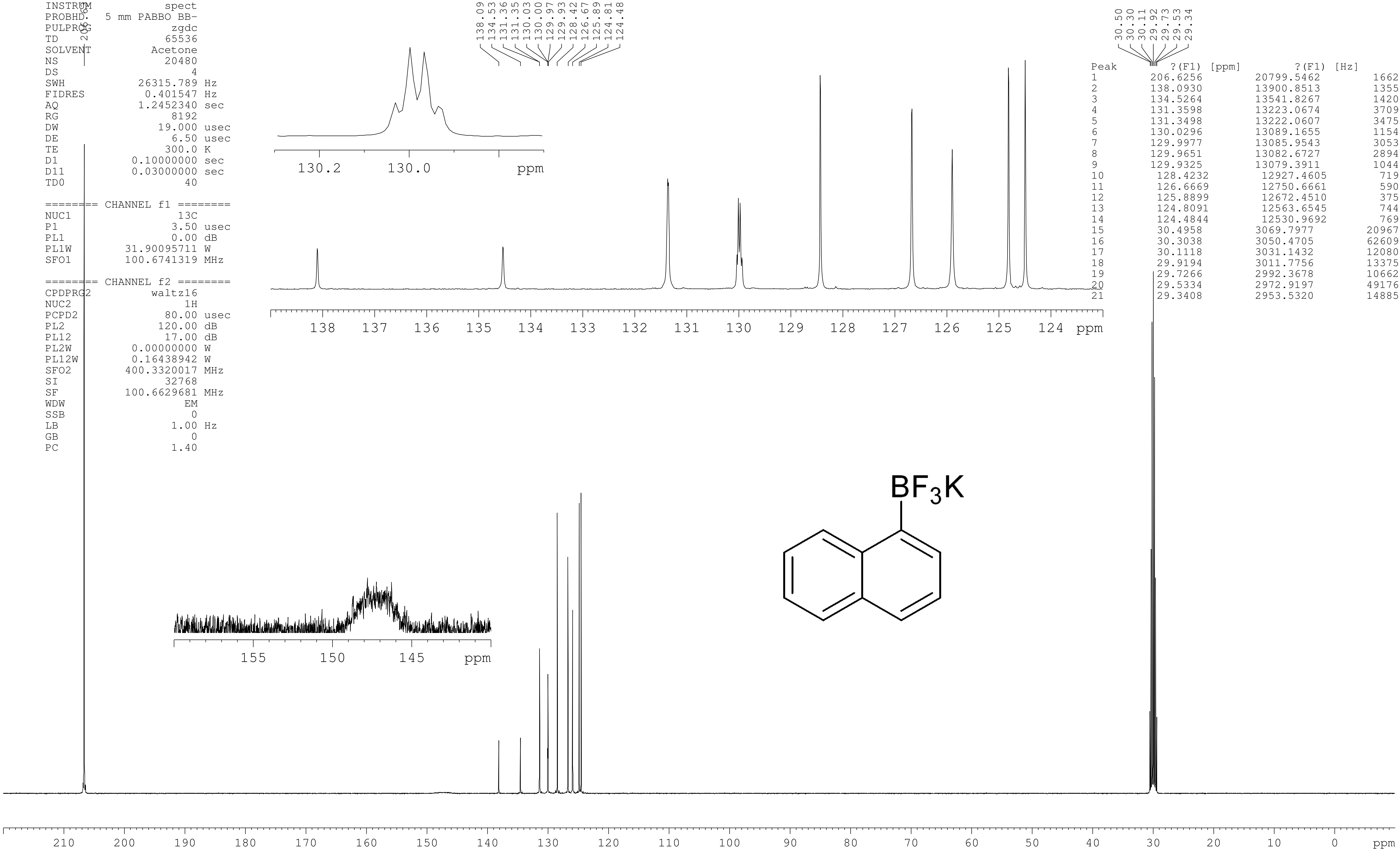
```
Peak        ?(F1) [ppm]        ?(F1) [Hz]
1        8.5931         3440.0758         8196506.1
2        8.5796         3434.6713         7299131.3
3        8.5735         3432.2293         6532354.3
4        8.5710         3431.2285         7299341.4
5        7.7730         3111.7651         1514380.0
6        7.7673         3109.4832         3359442.8
7        7.7588         3106.0804         17769909.
8        7.7536         3103.9987         17142073.
9        7.7492         3102.2373         14539829.
10        7.7402         3098.6343         19358287
11        7.7366         3097.1931         20898309
12        7.6495         3062.3244         13589763
13        7.6291         3054.1576         14826203
14        7.3580         2945.6282         2528576.
15        7.3524         2943.3863         5149962.
16        7.3410         2938.8226         21721231
17        7.3380         2937.6216         28635181
18        7.3361         2936.8609         25011249
19        7.3279         2933.5782         27916236
20        7.3176         2929.4548         31391880
21        7.3143         2928.1338         22849145
22        7.3024         2923.3698         7293569.
23        7.2992         2922.0888         9318907.
24        2.9424         1177.9310         1375948.
25        2.0612        825.1602         1315511.3
26        2.0559        823.0385         3657152.5
27        2.0502        820.7566         3715374.5
28        2.0449        818.6348         3584212.8
29        2.0393        816.3930         1306512.1
```

32077-209/5 hughesda
32077-209
1-naphthalene BF3K
Dried 18 h at 90 deg
nmr400b c-13

```
NAME            32077-209
EXPNO                   5
PROCNO                  1
Date_            20121025
Time                16.48
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zgdc
TD                  65536
SOLVENT           Acetone
NS                  20480
DS                      4
SWH             26315.789 Hz
FIDRES           0.401547 Hz
AQ              1.2452340 sec
RG                   8192
DW                 19.000 usec
DE                   6.50 usec
TE                  300.0 K
D1             0.10000000 sec
D11            0.03000000 sec
TD0                    40

======== CHANNEL f1 ========
NUC1                  13C
P1                   3.50 usec
PL1                  0.00 dB
PL1W          31.90095711 W
SFO1          100.6741319 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                120.00 dB
PL12                17.00 dB
PL2W           0.00000000 W
PL12W          0.16438942 W
SFO2          400.3320017 MHz
SI                  32768
SF            100.6629681 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



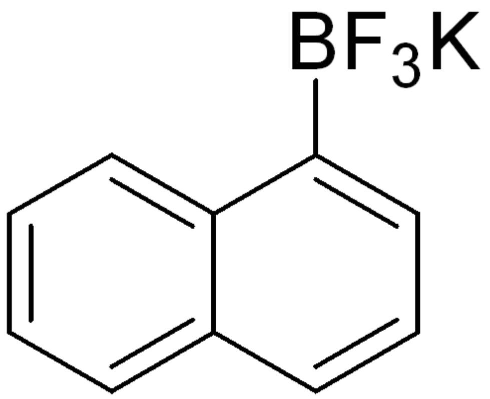
```
Peak       ?(F1) [ppm]        ?(F1) [Hz]          In
1          206.6256           20799.5462          16629453
2          138.0930           13900.8513          13550672
3          134.5264           13541.8267          14204242
4          131.3598           13223.0674          37097515
5          131.3498           13222.0607          34752835
6          130.0296           13089.1655          11547391
7          129.9977           13085.9543          30534750
8          129.9651           13082.6727          28946513
9          129.9325           13079.3911          10442327
10          128.4232           12927.4605           7192276
11          126.6669           12750.6661           5902417
12          125.8899           12672.4510           3756084
13          124.8091           12563.6545           7440300
14          124.4844           12530.9692           7694079
15          30.4958           3069.7977          209674755
16          30.3038           3050.4705          626098780
17          30.1118           3031.1432          120802708
18          29.9194           3011.7756          133759982
19          29.7266           2992.3678          106624927
20          29.5334           2972.9197          491769136
21          29.3408           2953.5320          148853001
```

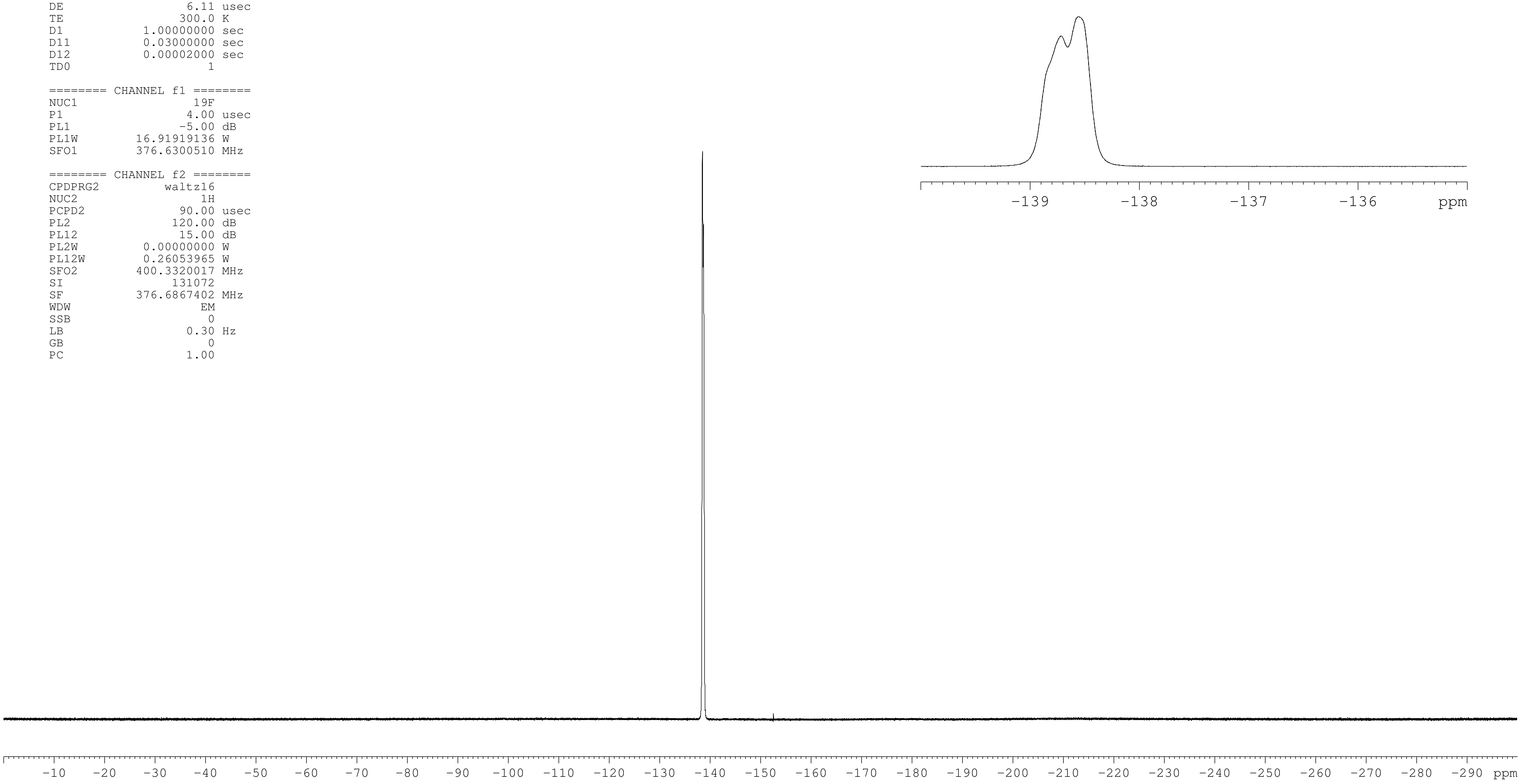
32077-209/2 hughesda
32077-209
1-Naphthalene BF3K
nmr400b f-19

```
NAME             32077-209
EXPNO                    2
PROCNO                   1
Date_             20121024
Time                  7.29
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgfhigqn
TD                  262144
SOLVENT            Acetone
NS                     192
DS                       4
SWH          116959.063 Hz
FIDRES         0.446163 Hz
AQ            1.1207156 sec
RG                    4096
DW                4.275 usec
DE                 6.11 usec
TE                300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   19F
P1                 4.00 usec
PL1               -5.00 dB
PL1W        16.91919136 W
SFO1        376.6300510 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2             90.00 usec
PL2              120.00 dB
PL12              15.00 dB
PL2W         0.00000000 W
PL12W        0.26053965 W
SFO2        400.3320017 MHz
SI                  131072
SF          376.6867402 MHz
WDW                     EM
SSB                      0
LB                 0.30 Hz
GB                       0
PC                    1.00
```



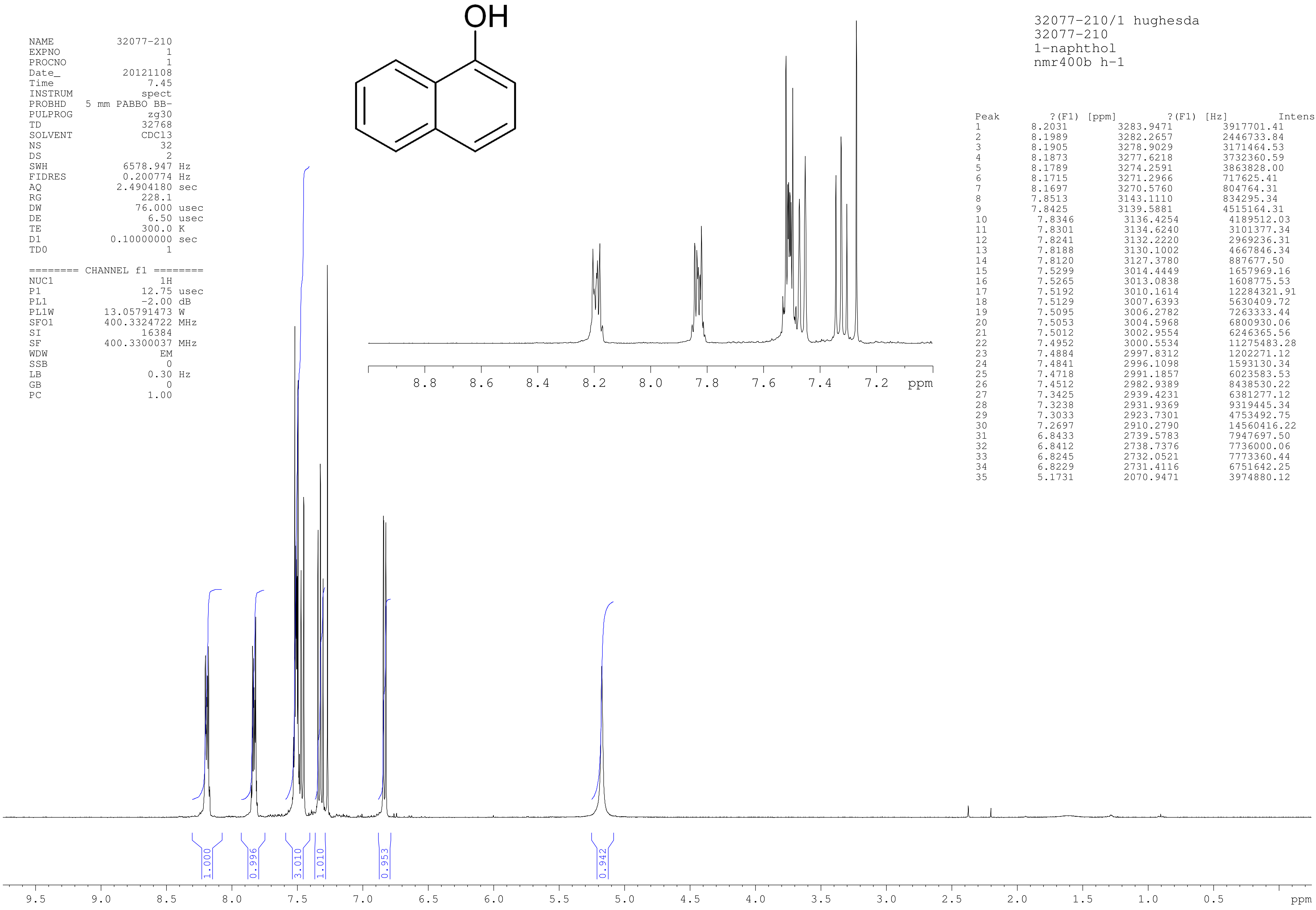


32077-210/1 hughesda
32077-210
1-naphthol
nmr400b h-1

NAME	32077-210
EXPNO	1
PROCNO	1
Date_	20121108
Time	7.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDC13
NS	32
DS	2
SWH	6578.947 Hz
FIDRES	0.200774 Hz
AQ	2.4904180 sec
RG	228.1
DW	76.000 usec
DE	6.50 usec
TE	300.0 K
D1	0.10000000 sec
TD0	1

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

NUC1	1H
P1	12.75 usec
PL1	-2.00 dB
PL1W	13.05791473 W
SFO1	400.3324722 MHz
SI	16384
SF	400.3300037 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



Peak	?(F1) [ppm]	?(F1) [Hz]	Intensit
1	8.2031	3283.9471	3917701.41
2	8.1989	3282.2657	2446733.84
3	8.1905	3278.9029	3171464.53
4	8.1873	3277.6218	3732360.59
5	8.1789	3274.2591	3863828.00
6	8.1715	3271.2966	717625.41
7	8.1697	3270.5760	804764.31
8	7.8513	3143.1110	834295.34
9	7.8425	3139.5881	4515164.31
10	7.8346	3136.4254	4189512.03
11	7.8301	3134.6240	3101377.34
12	7.8241	3132.2220	2969236.31
13	7.8188	3130.1002	4667846.34
14	7.8120	3127.3780	887677.50
15	7.5299	3014.4449	1657969.16
16	7.5265	3013.0838	1608775.53
17	7.5192	3010.1614	12284321.91
18	7.5129	3007.6393	5630409.72
19	7.5095	3006.2782	7263333.44
20	7.5053	3004.5968	6800930.06
21	7.5012	3002.9554	6246365.56
22	7.4952	3000.5534	11275483.28
23	7.4884	2997.8312	1202271.12
24	7.4841	2996.1098	1593130.34
25	7.4718	2991.1857	6023583.53
26	7.4512	2982.9389	8438530.22
27	7.3425	2939.4231	6381277.12
28	7.3238	2931.9369	9319445.34
29	7.3033	2923.7301	4753492.75
30	7.2697	2910.2790	14560416.22
31	6.8433	2739.5783	7947697.50
32	6.8412	2738.7376	7736000.06
33	6.8245	2732.0521	7773360.44
34	6.8229	2731.4116	6751642.25
35	5.1731	2070.9471	3974880.12

32077-210/2 hughesda
32077-210
1-naphthol
nmr400b c-13

```
NAME           32077-210
EXPNO                  2
PROCNO                 1
Date_           20121108
Time                7.50
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  1823
DS                     4
SWH            26315.789 Hz
FIDRES          0.401547 Hz
AQ             1.2452340 sec
RG                  8192
DW                19.000 usec
DE                  6.50 usec
TE                 300.0 K
D1            0.10000000 sec
D11           0.03000000 sec
TD0                   40

======== CHANNEL f1 ========
NUC1                 13C
P1                  3.50 usec
PL1                 0.00 dB
PL1W         31.90095711 W
SFO1         100.6741319 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2               120.00 dB
PL12               17.00 dB
PL2W          0.00000000 W
PL12W         0.16438942 W
SFO2         400.3320017 MHz
SI                 32768
SF           100.6630367 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```