Electronic Supplementary Material (ESI) for Green Chemistry

A Highly Active Magnetically Recoverable Nano Ferrite-Glutathione-Copper (Nano-FGTCu) Catalyst for Huisgen 1, 3-Dipolar Cycloadditions

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Supporting Information

Table of Contents

Experimental section

Synthesis of magnetic nano-ferrites

$FeSO_4 \cdot 7H_2O$ (13.9 g) and $Fe_2(SO_4)_3$ (20 g) were dissolved in 500 mL water in a 1000 mL beaker. Ammonium hydroxide (25%) was added slowly to adjust the pH of the solution to 10. The reaction mixture was then continually stirred for 1 h at 60 ◦C. The precipitated nanoparticles were separated magnetically, washed with water until the pH reached 7, and then dried under vacuum at 60 ◦C for 2 h. Ferrite was characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM) . This magnetic nano-ferrite (Fe_3O_4) was then used for further chemical modification.

Surface modification of nano-ferrites

Nano-Fe_3O_4 (0.5 g) was dispersed in water (15 mL) and methanol(5 mL) and sonicated for 15 min. Glutathione (reduced form) (0.2 g) dissolved in water (5 mL) was added to this solution and again sonicated for 2 h. The glutathione-functionalized nanomaterial (nano-organocatalyst) was then isolated by centrifugation, washed with water and methanol, and dried under vacuum at 50-60 °C.

Synthesis of nano-FGT-Cu, catalyst

Glutathione-functionalized nano-Fe_3O_4 (1 gm) was dispersed in water-methanol mixture (1:1) and $CuCl_2.2H_2O$ (100 mg) solution in water was added to the reaction mixture. Hydrazine monohydrate solution in water was added drop wise to bring the pH of this mixture to 9, followed by addition of 0.1 gm of $NaBH_4$. The reaction mixture was then stirred for 24 h at room temperature. The product was allowed to settle, washed several times with water and acetone, and dried under vacuum at 60 ◦C for 2 h. Catalyst characterization by X-ray diffraction (XRD) (Fig. 1d) and transmission electron microscopy (TEM) (Fig. 1c) confirm the anchoring of Cu nanoparticles on ferrite surfaces. The weight percentage of Cu in the catalyst was found to be 1.57% by ICP-AES analysis.

Synthesis of triazole from benzyl azide an alkyne

Benzyl azide (1.2 mmol), alkyne (1.0 mmol) and nano-FGT-Cu catalyst (100 mg) were placed in a crimp-sealed thick-walled glass tube equipped with a pressure sensor and a magnetic stirrer and water (4 mL) was added to the reaction mixture. The reaction tube was placed inside the cavity of a CEM Discover focused microwave synthesis system, operated at 120 ^{o}C (temperature

monitored by a built-in infrared sensor), power 100 Watt and pressure 10–60 psi for 8-12 min (Table 2). After completion of the reaction, the catalyst was easily removed from reaction mixture using an external magnet. After separation of catalyst, solid product was filtered off or extracted with ethyl acetate and recrystallized.

Synthesis of triazole via in situ generation of alkyl azide

Alkyl halides (1.2 mmol) , NaN_3(1.5 mmol), and alkyne (1.0 mmol)and nano-FGT-Cu catalyst(100 mg) were placed in a crimp-sealed thick-walled glass tube equipped with a pressure sensor and a magnetic stirrer and 4 mL of water was added to the reaction mixture. The reaction tube was placed inside the cavity of a CEM Discover focused microwave synthesis system, operated at 120 ^{o}C (temperature monitored by a built-in infrared sensor), power 100 Watt and pressure 10–60 psi for 10-20 min (Table 3 and 4). After completion of the reaction, the catalyst was easily removed from reaction mixture using an external magnet. After separation of catalyst solid product was filtered off or extracted with ethyl acetate and recrystallized or purified by column chromatography.

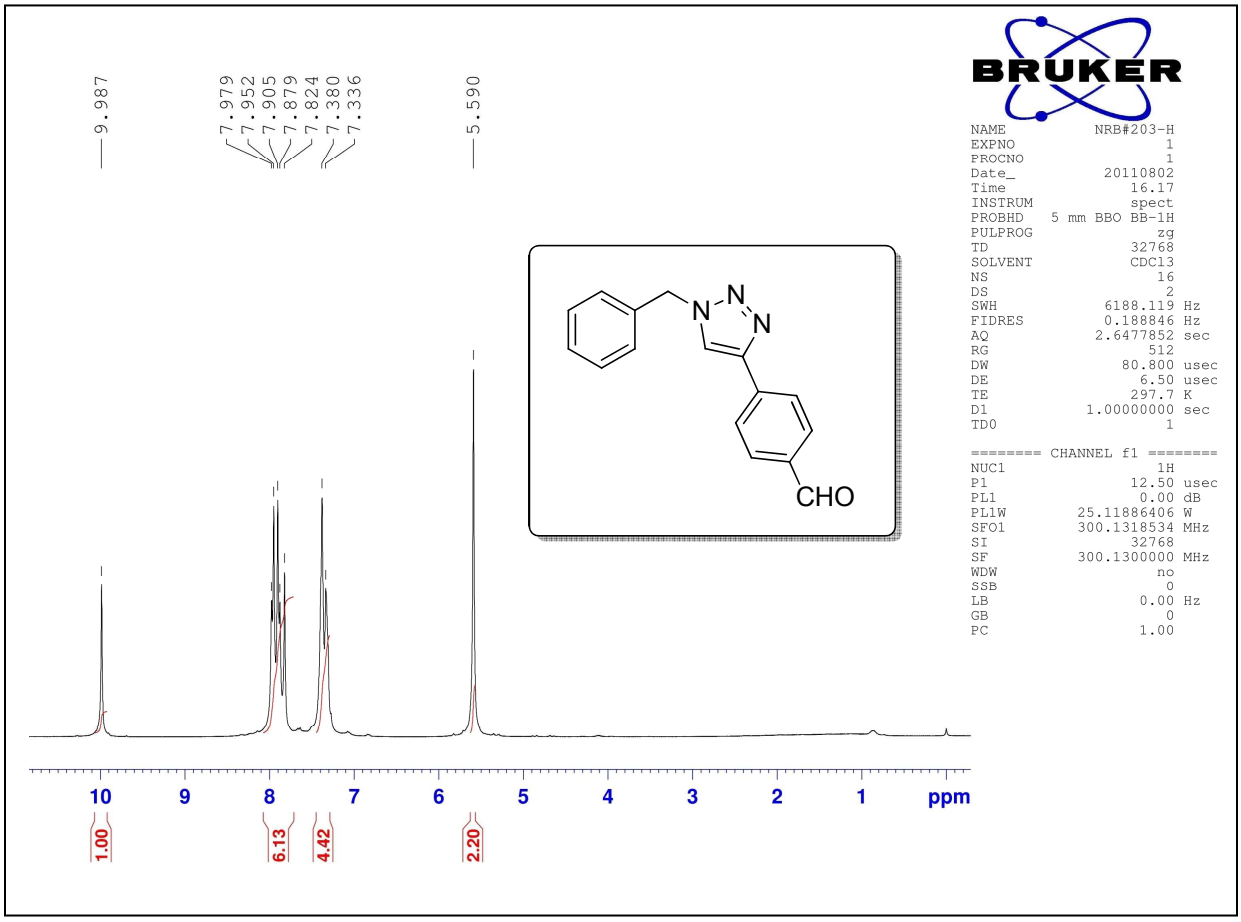
All products listed in Table 2-4 are known in the literature (except Table 3 entry 5) and were identified by comparison of their FT-IR, ^{1}H, and ^{13}C NMR with literature data.[1] The characterization data for a representative compound, Table 3, entry 5 is given below.

4-(1-benzyl-1H-1, 2, 3-triazol-4-yl)benzaldehyde (Table 3 entry 5). White solid, Yield 86%, FT-IR (cm^{-1}): 3130(w), 2839(m), 1690(s), 1608(s), 1572(m), 1305(m), 1213(s), 1171(s), 834(s), 810(s), 720(s), 701(s), 680(s); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H : 9.98(1H, s), 7.97-7.82(6H, m), 7.38-7.33(4H, m), 5.59 (2H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ_C : 191.68, 146.86, 136.33, 135.76, 134.36, 130.34, 129.24, 128.95, 128.14, 126.01, 120.81, 54.37, Analysis calculated for $C_{16}H_{13}N_3O$: C 72.99, H 4.98, N 15.96; Found: C 72.97, H 4.96, N 15.98.

Reference

1. (a) M. Liu and O. Reiser, *Org. Lett.* 2011, **13**, 1102; (b) B. Saha, S. Sharma, D. Sawant and B. Kundu, *Synlett*, 2007, 1591; (c) F. Alonso, Y. Moglie, G. Radivoy and M. Yus, *Org. Biomol. Chem.*, 2011, **9**, 6385; (d) S. Ozcubukcu, E. Ozkal, C. Jimeno, and M. A. Pericas, *Org. Lett.*, 2009, **11**, 4680; (e) J. R. Cabrero-Antonino, T. Garcia, P. Rubio-

Marques, J. A. Vidal-Moya, A. Leyva-Perez, S. S. Al-Deyab, S. I. Al-Resayes, U. Diaz, and A. Corma, *ACS Catal.*, 2011, **1**, 147; (f) C. Shao, X. Wang, Q. Zhang, S. Luo, J. Zhao, and Y. Hu, *J. Org. Chem*., 2011, **76**, 6832; (g) X. Meng, X. Xu, T. Gao, B. Chen, *Eur. J. Org. Chem.*, 2010, **28**, 5409; (h) S. Chassaing, A. S. S. Sido, A. Alix, M. Kumarraja, P. Pale, J. Sommer, *Chem. Europ. J.*, **2009**, **15**, 1582.



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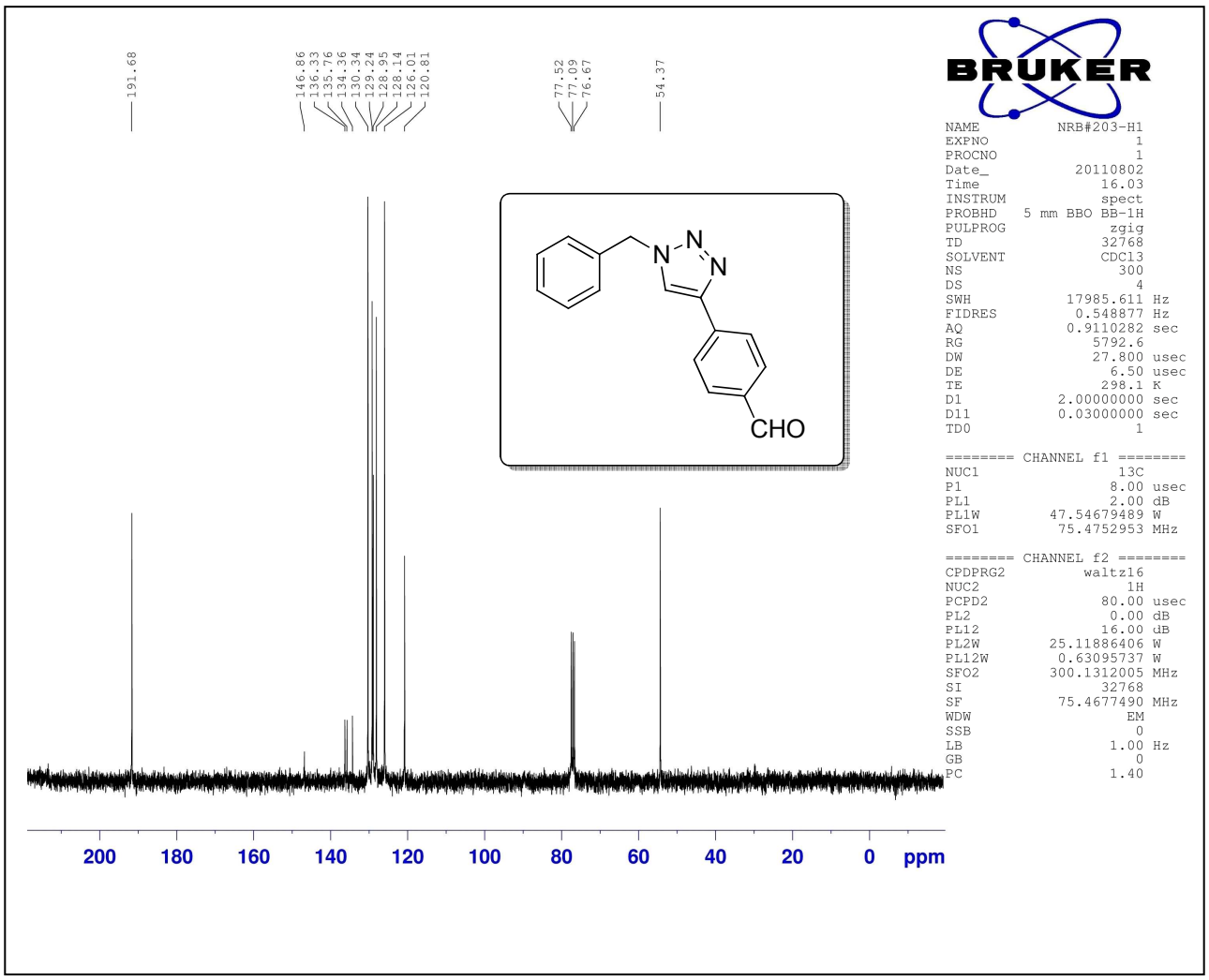

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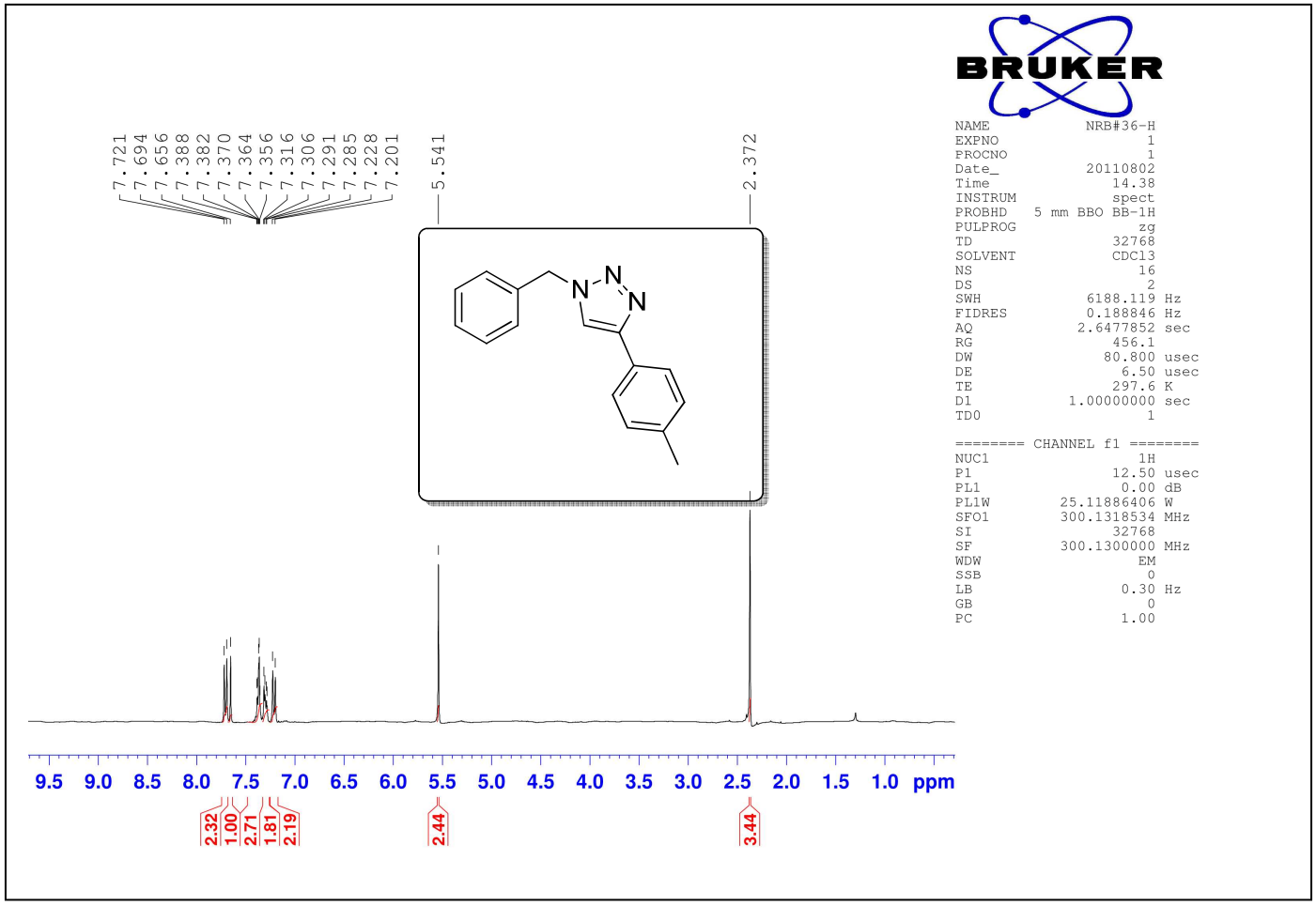
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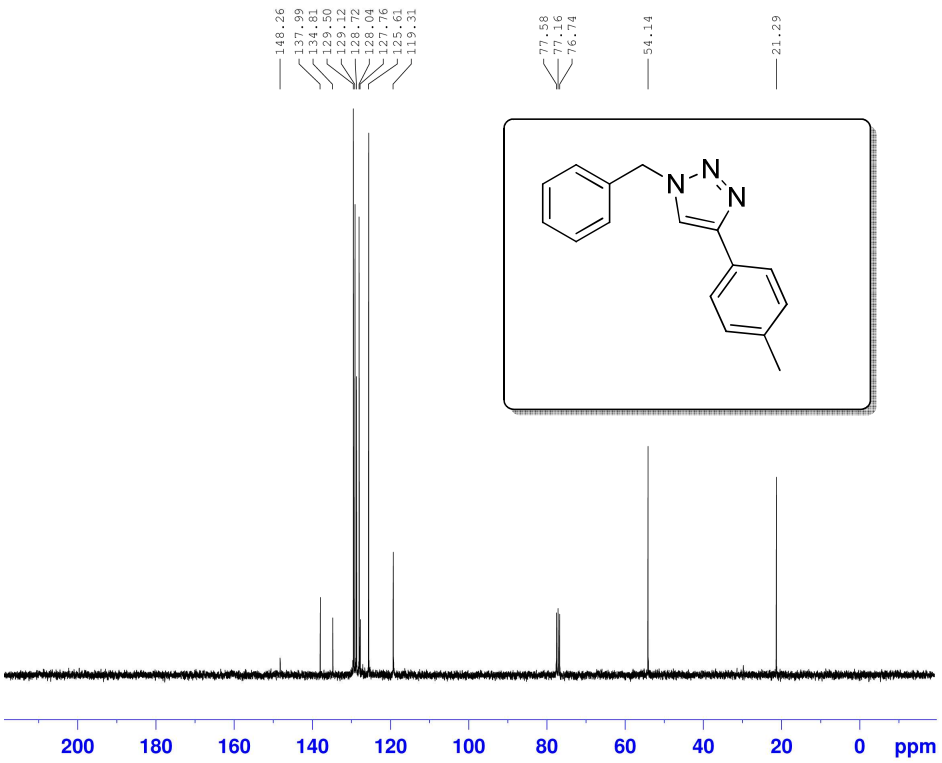

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PL12W	0.63095737 W
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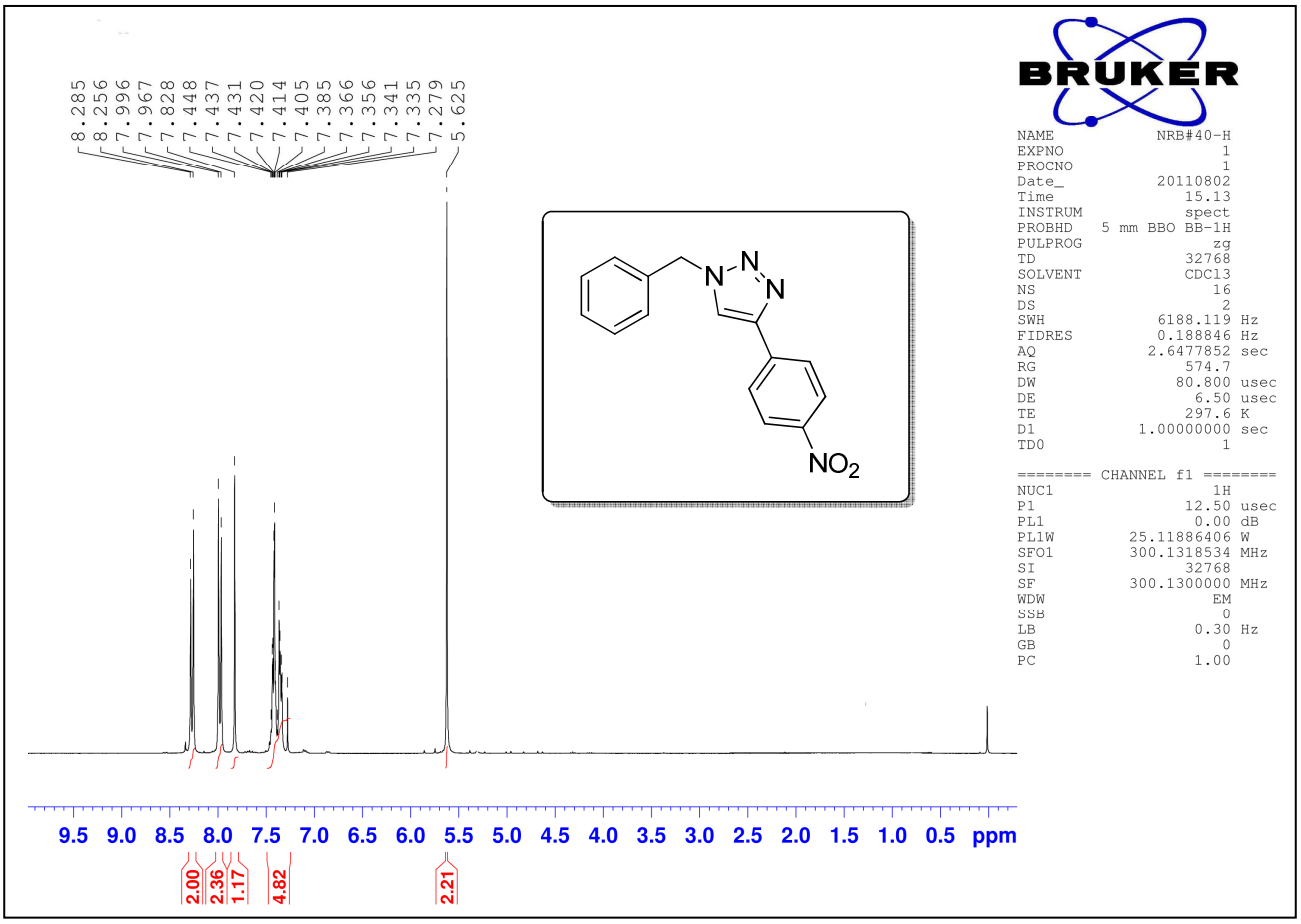

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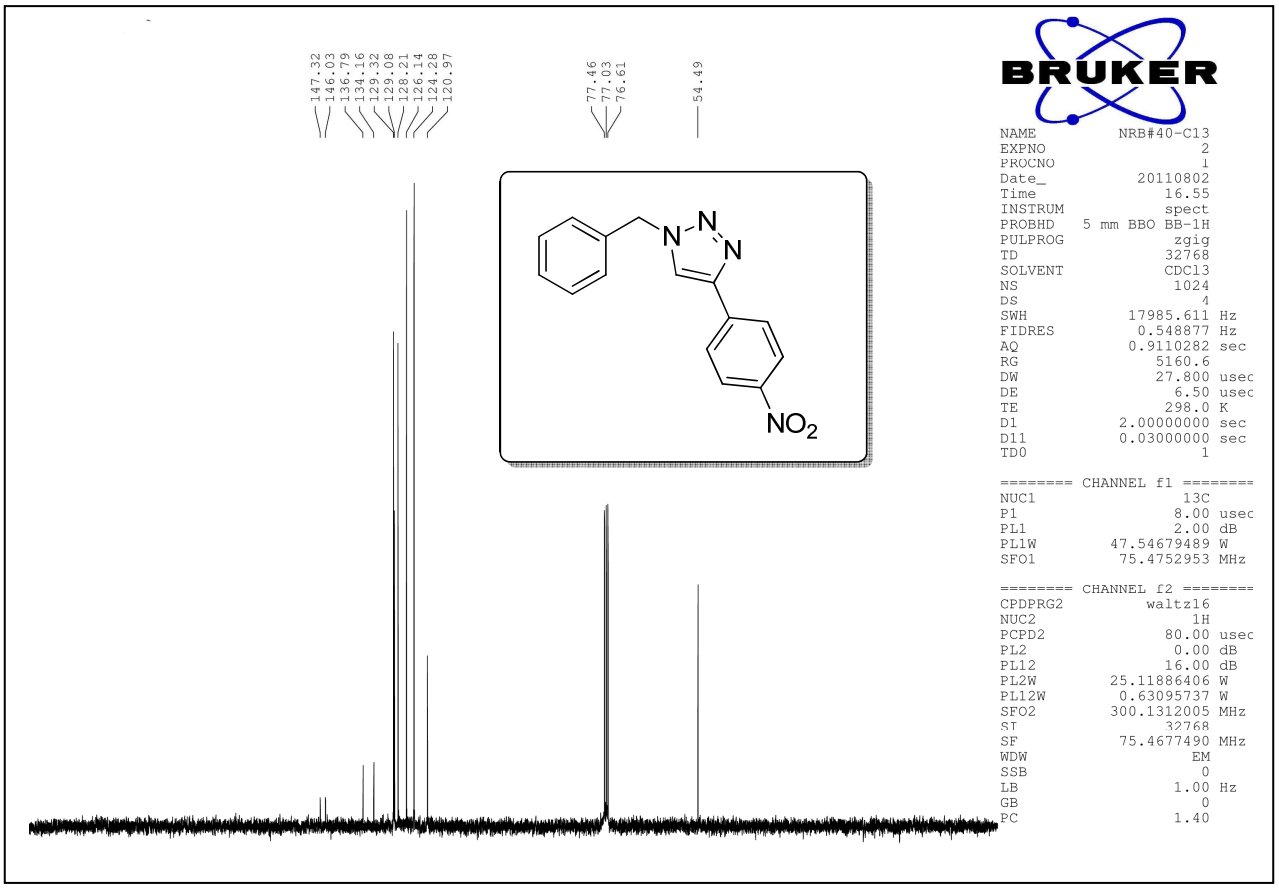

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PL12	16.00 dB
PL2W	25.11886406 W
PL12W	0.63095737 W
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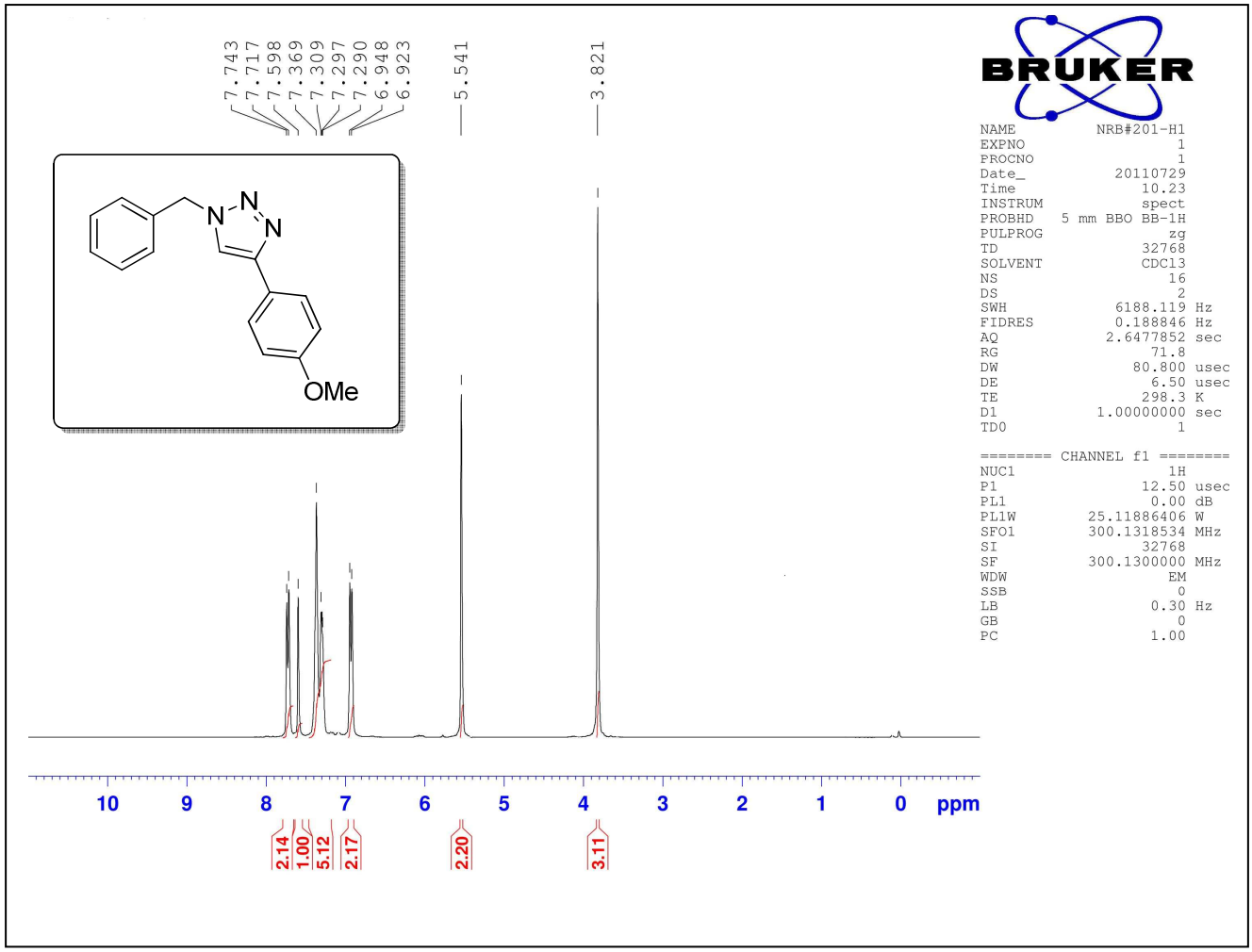

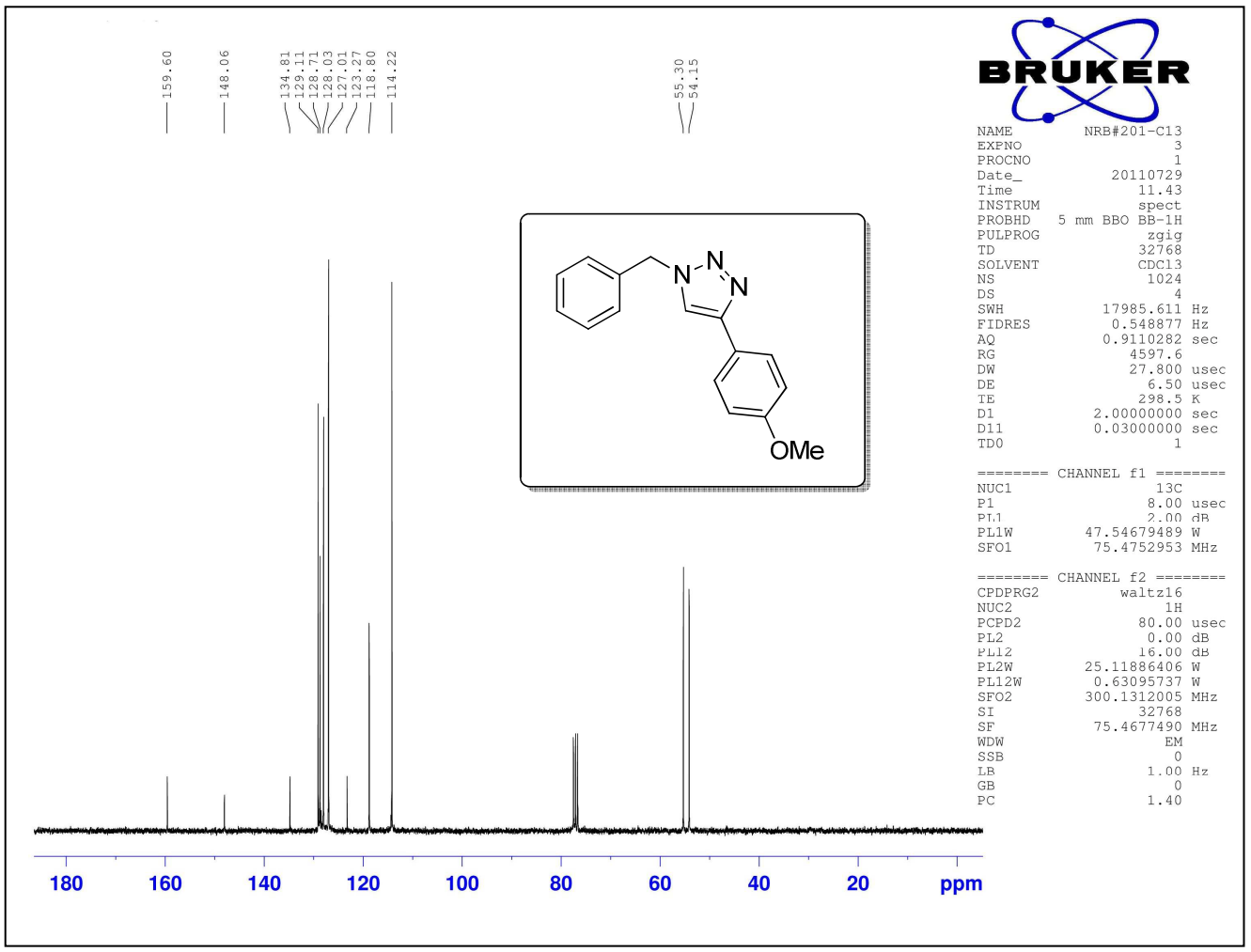

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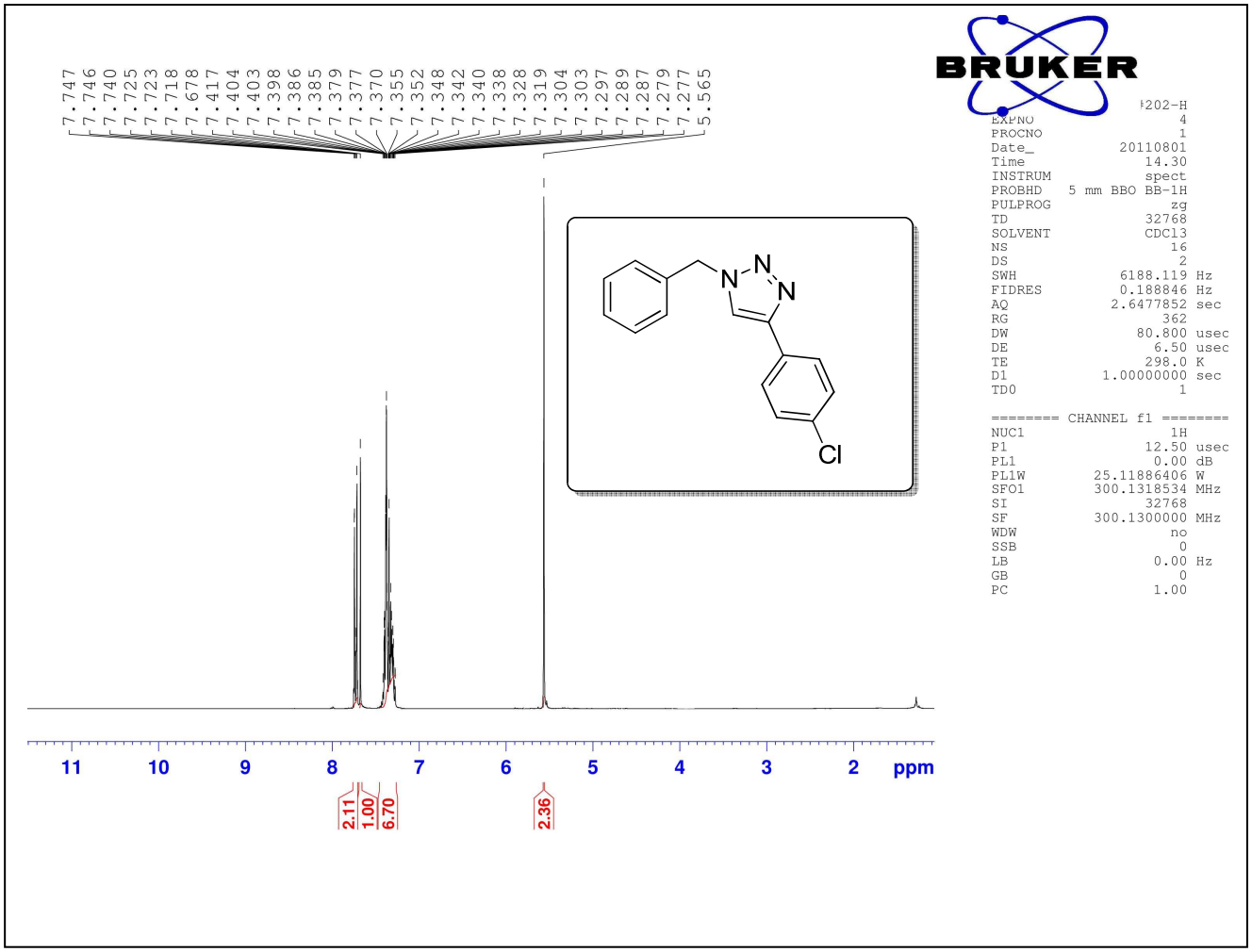
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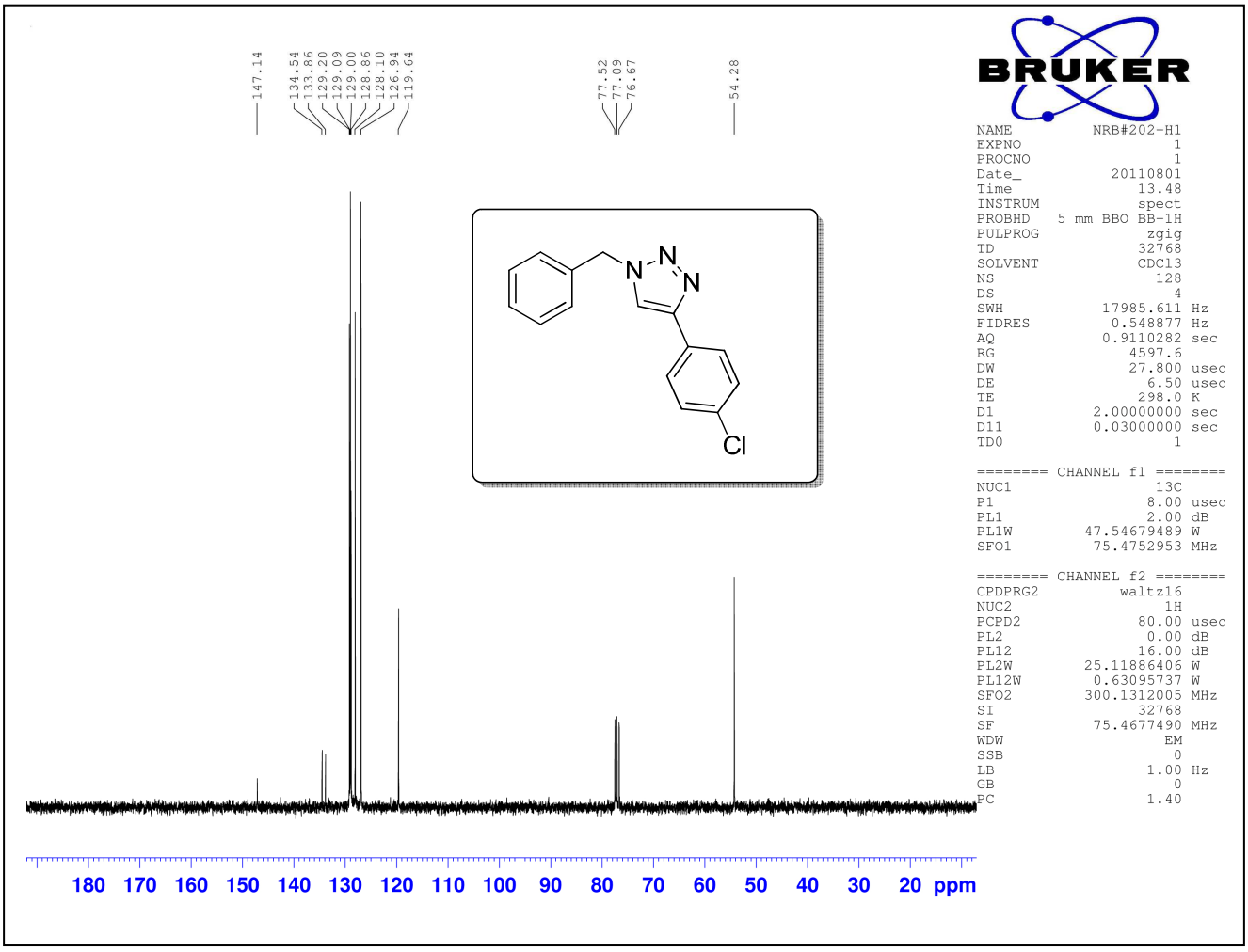

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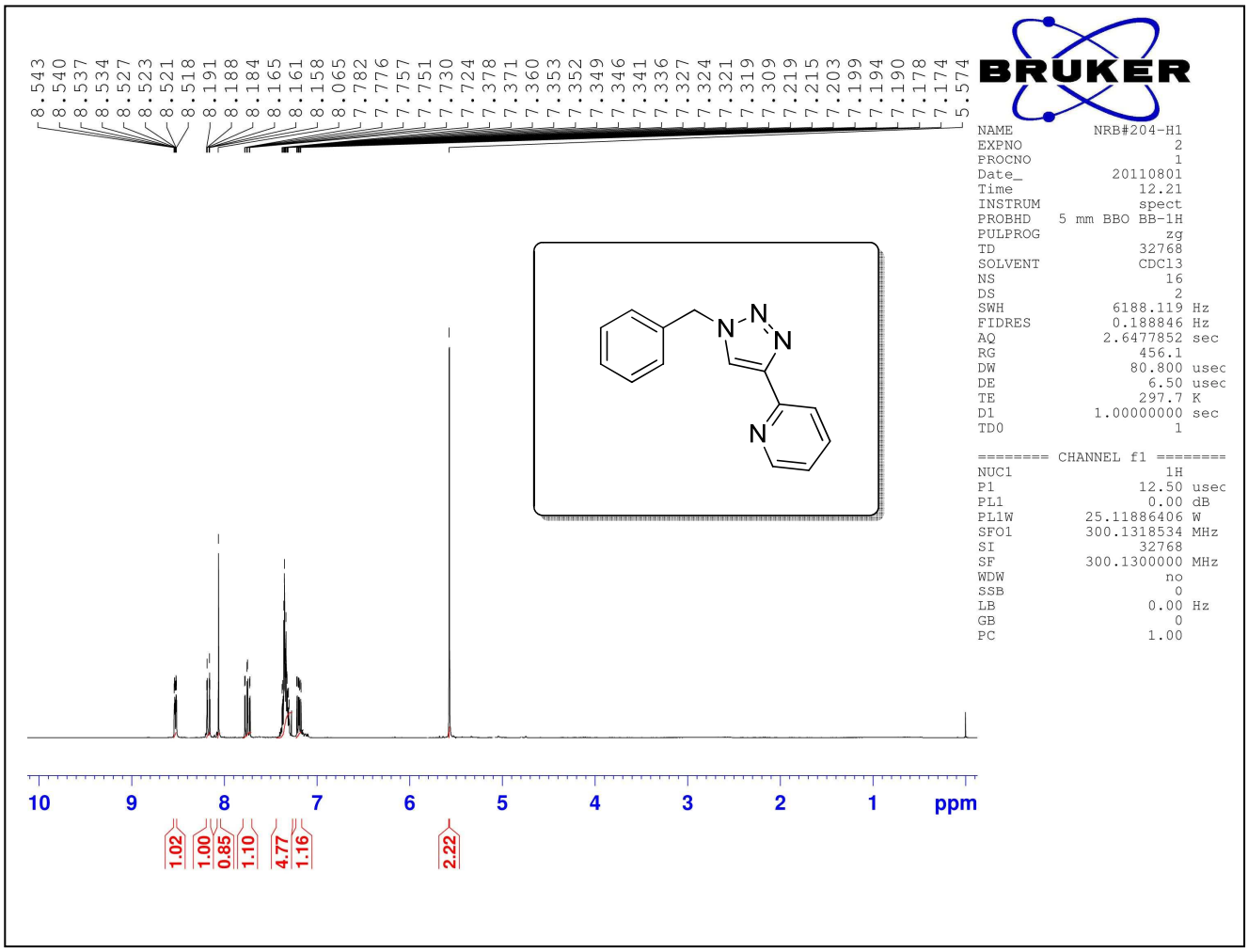
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RG 456.1
DW 80.800 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
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SI 32768
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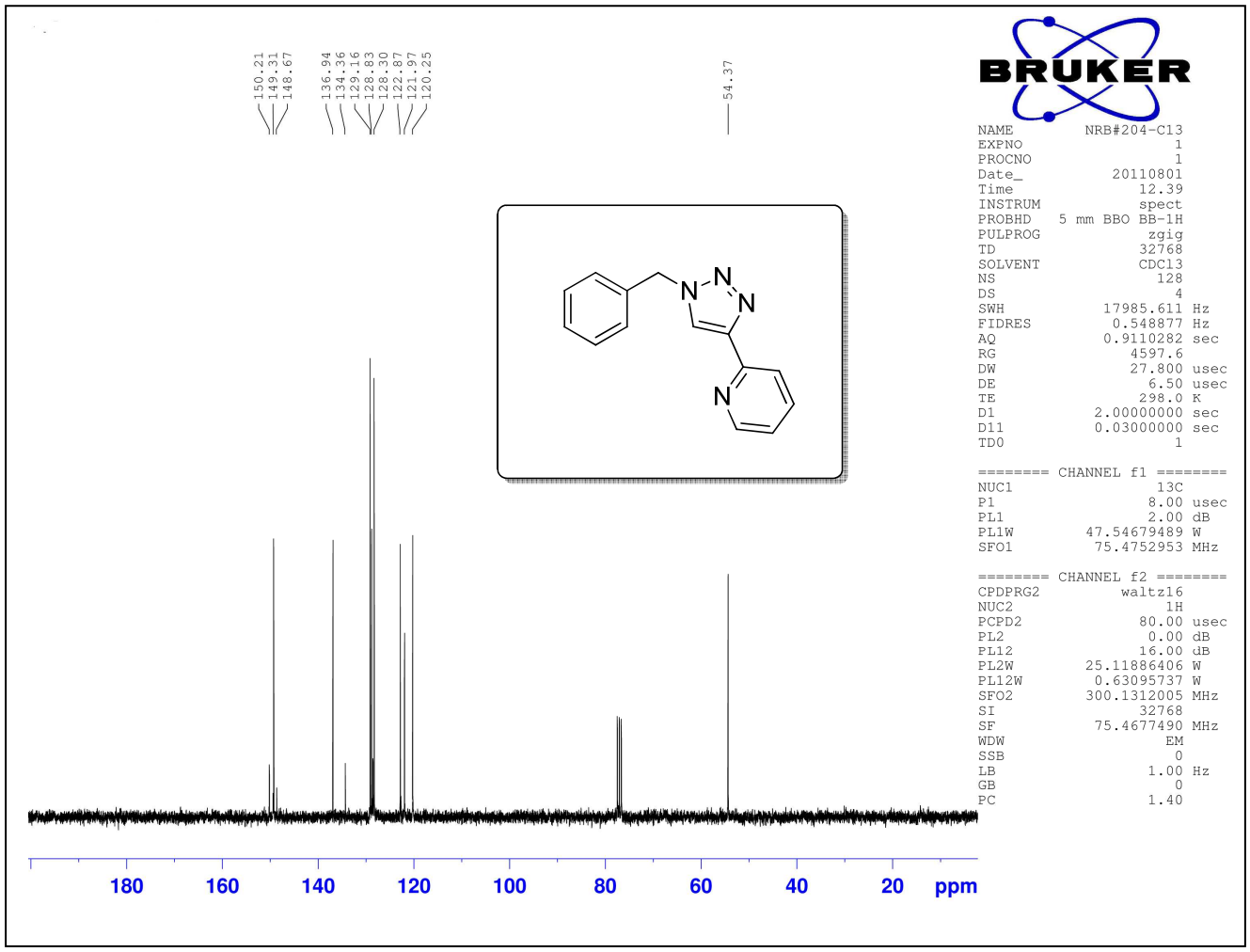

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PL12W	0.63095737 W
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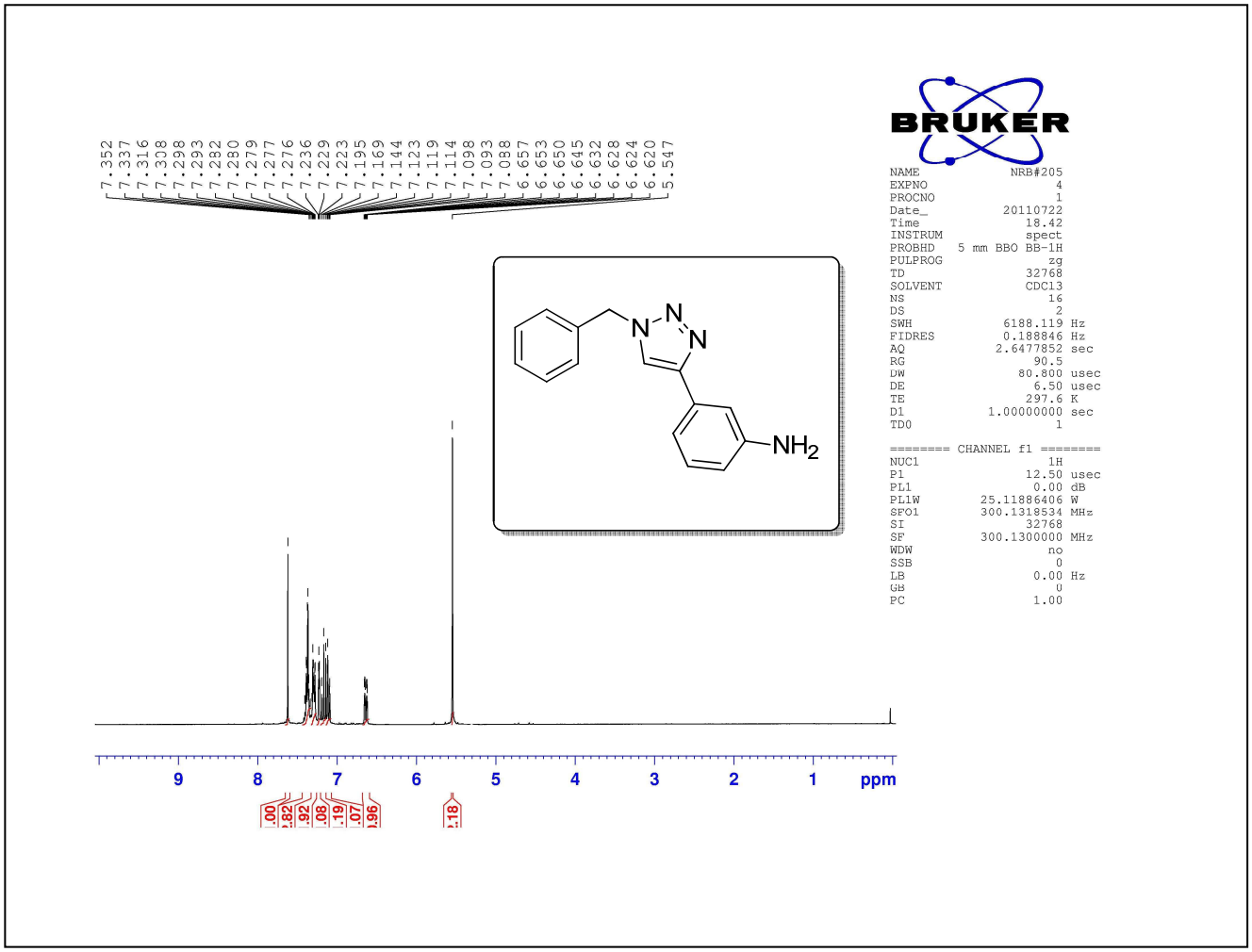

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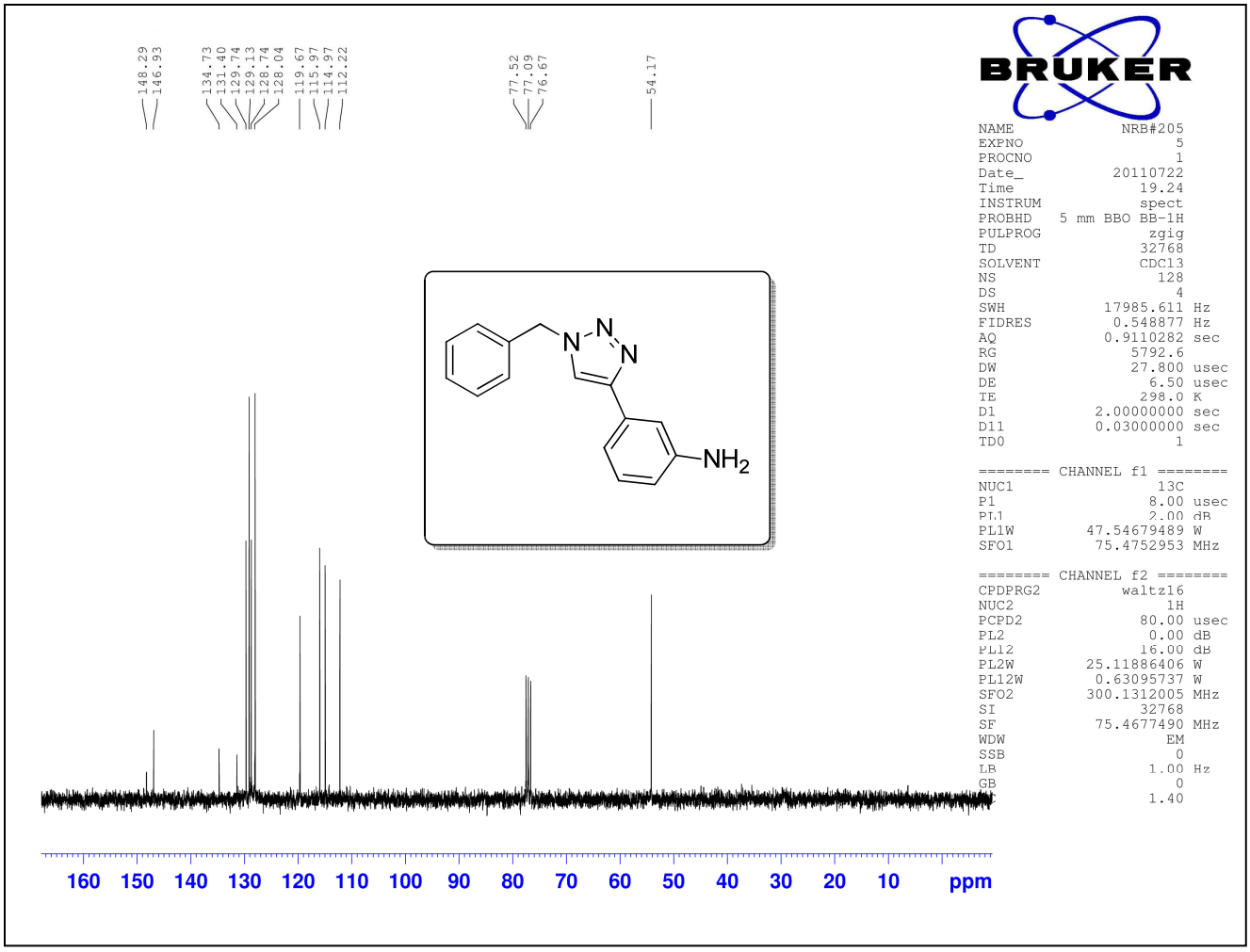
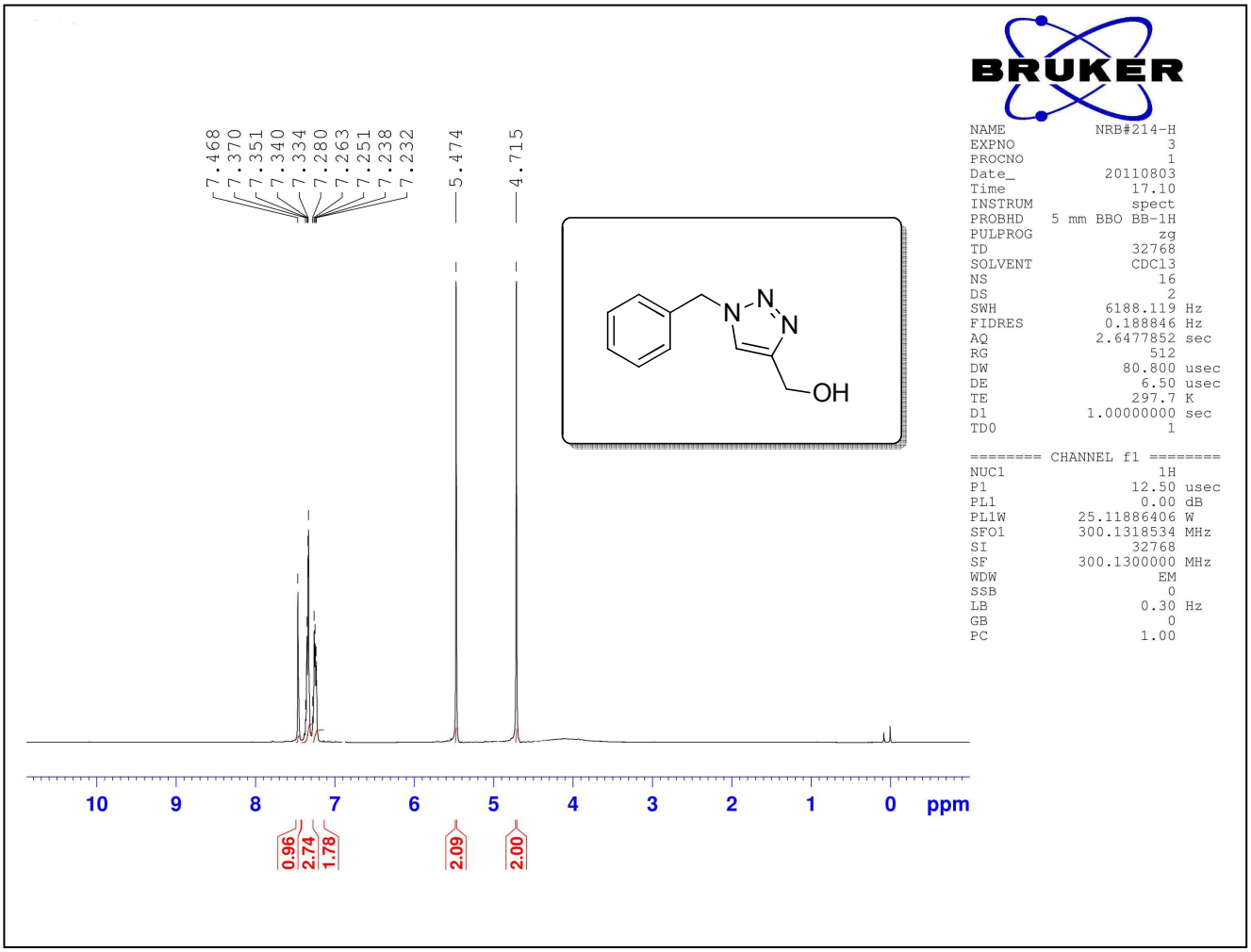
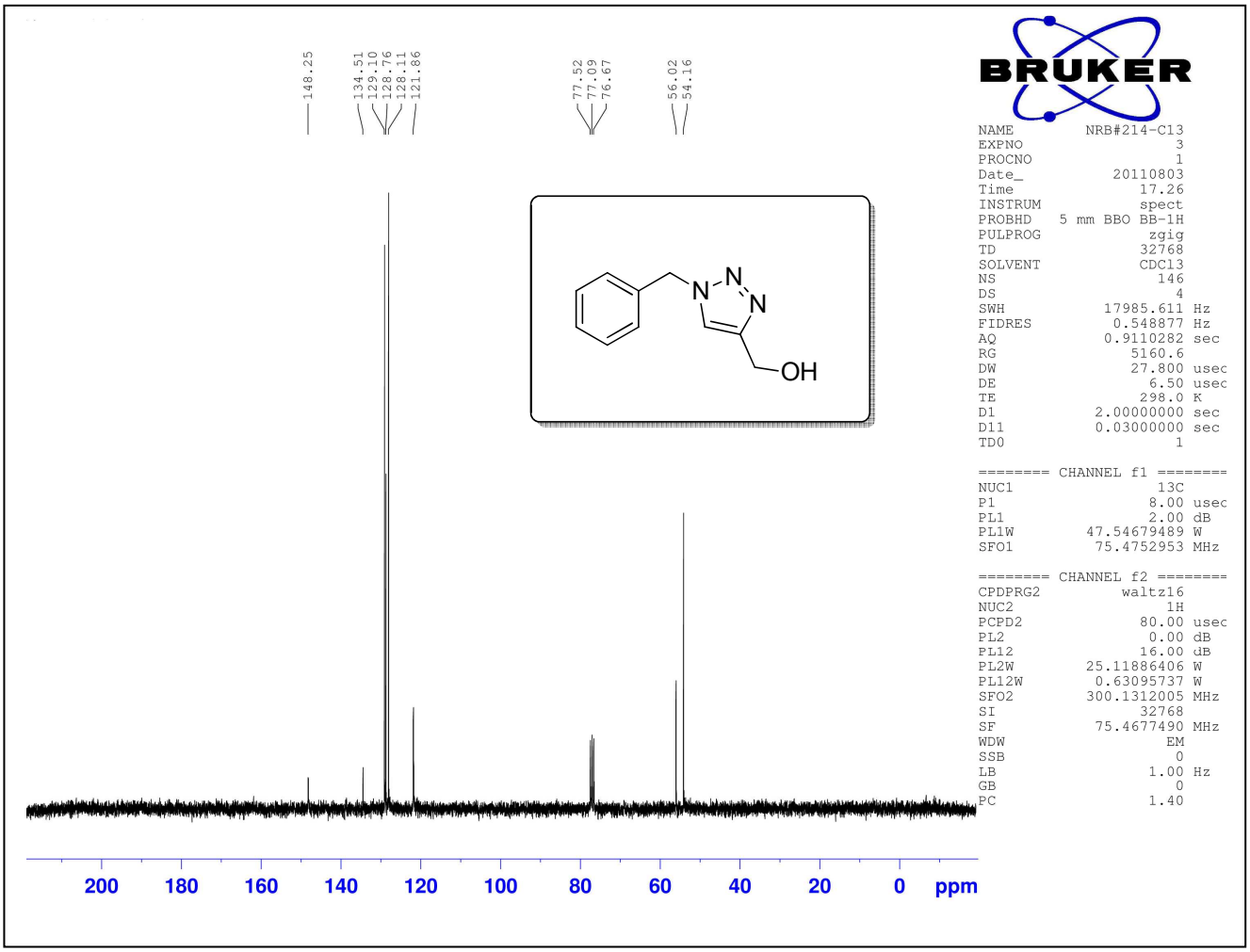
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



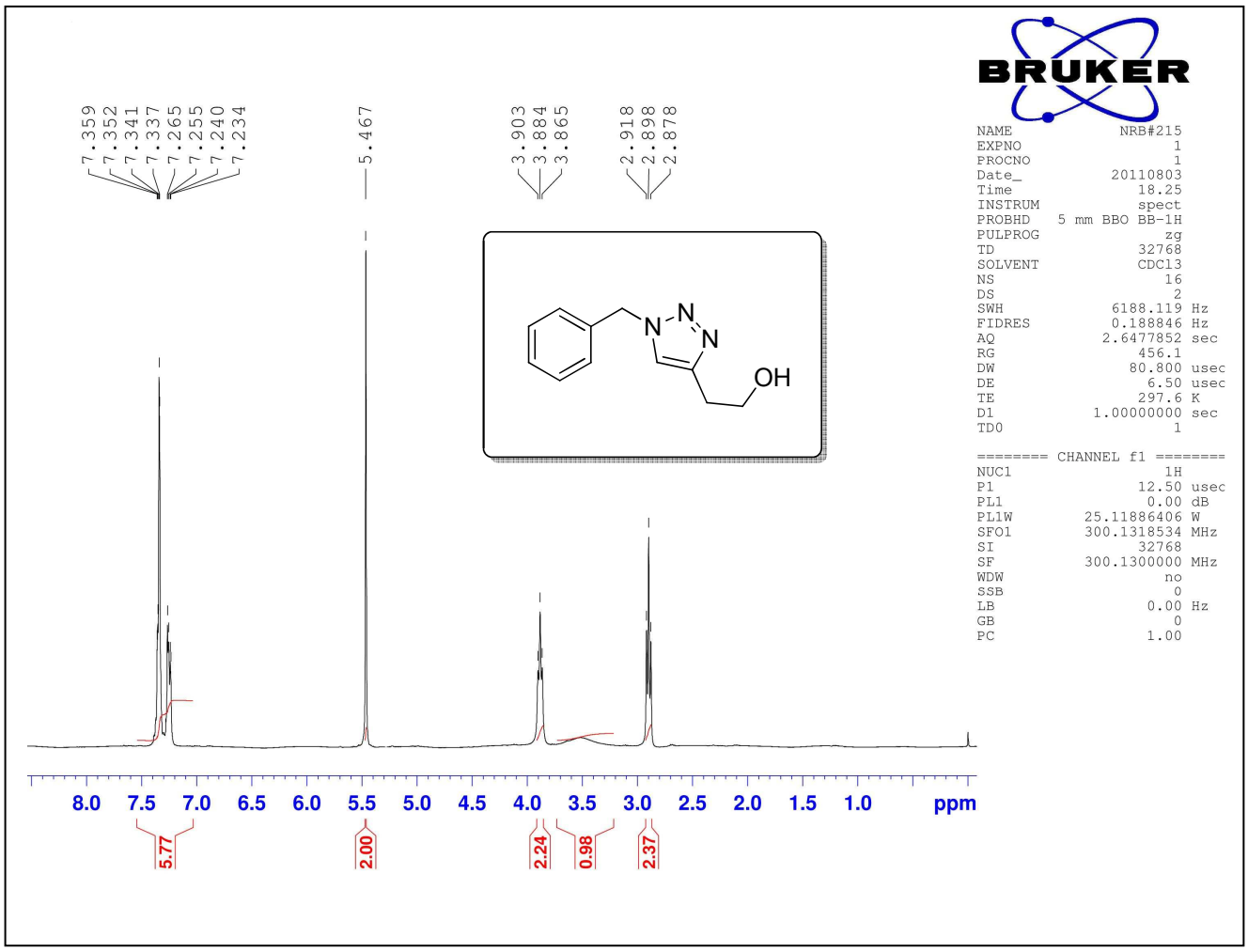

```
NAME                 NRB#205
EXPNO                      5
PROCNO                     1
Date_               20110722
Time                   19.24
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zgig
TD                    32768
SOLVENT               CDCl3
NS                      128
DS                        4
SWH              17985.611 Hz
FIDRES            0.548877 Hz
AQ                0.9110282 sec
RG                   5792.6
DW                  27.800 usec
DE                   6.50 usec
TE                  298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   8.00 usec
PL1                  2.00 dB
PL1W          47.54679489 W
SFO1           75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  0.00 dB
PL12                16.00 dB
PL2W          25.11886406 W
PL12W          0.63095737 W
SFO2          300.1312005 MHz
SI                    32768
SF             75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
                      1.40
```

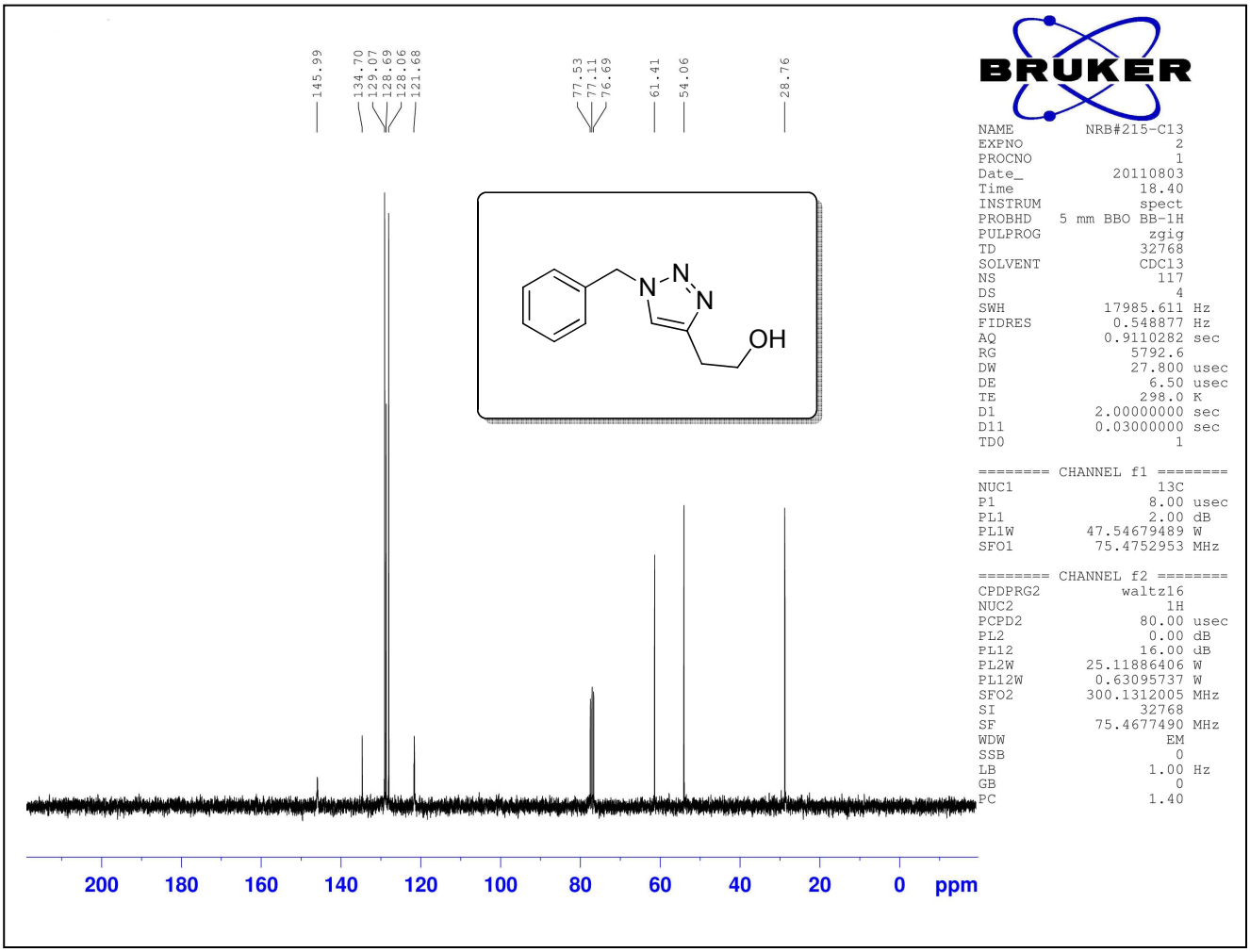

7.468
7.370
7.351
7.340
7.334
7.280
7.263
7.251
7.238
7.232
5.474
4.715
N
N
N
OH
10
9
8
7
6
5
4
3
2
1
0
ppm
0.96
2.74
1.78
2.09
2.00
BRUKER
NAME NRB#214-H
EXPNO 3
PROCNO 1
Date_ 20110803
Time 17.10
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 512
DW 80.800 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 0.00 dB
PL1W 25.11886406 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

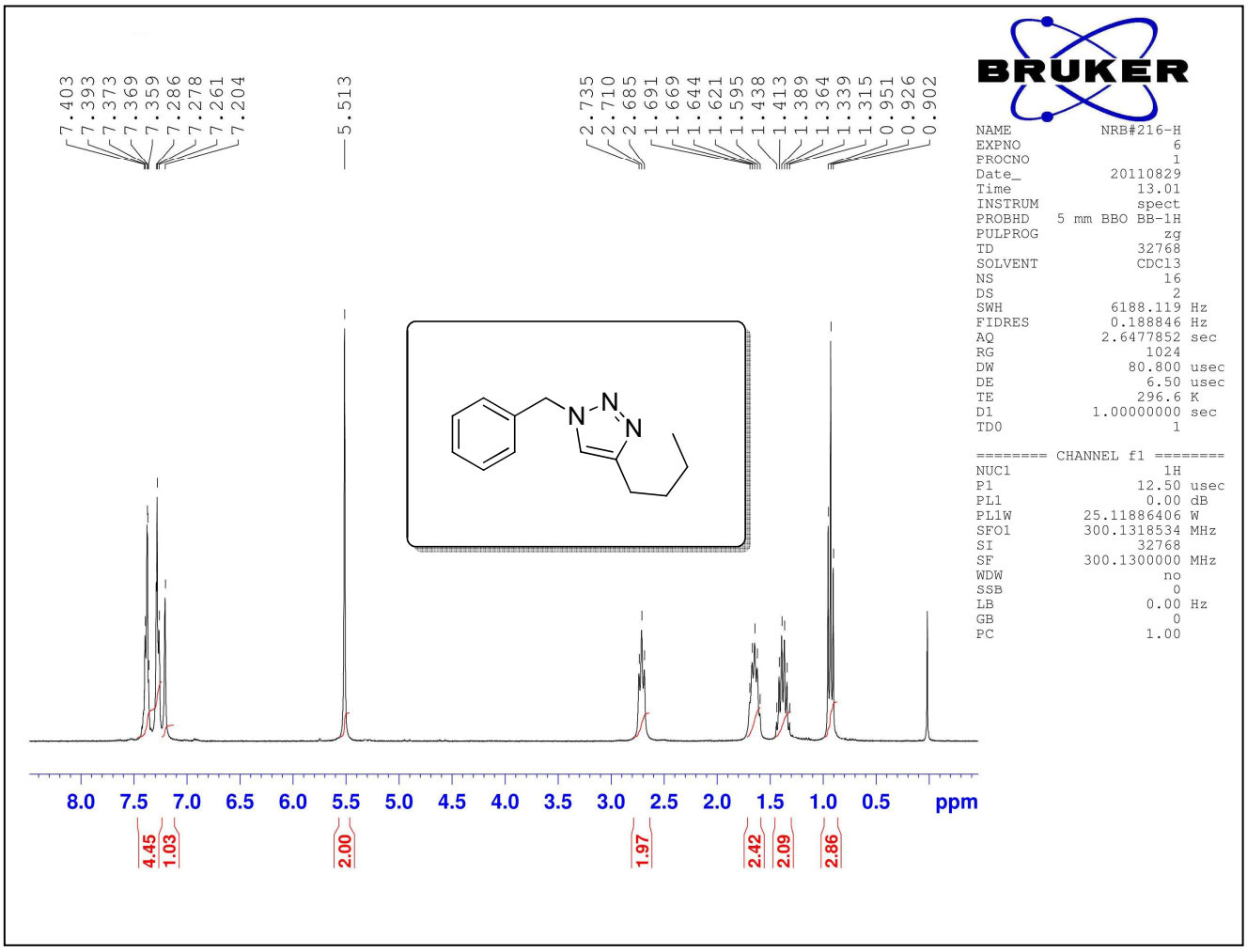
NAME	NRB#214-C13
EXPNO	3
PROCNO	1
Date_	20110803
Time	17.26
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	146
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	5160.6
DW	27.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	2.00 dB
PL1W	47.54679489 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	16.00 dB
PL2W	25.11886406 W
PL12W	0.63095737 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

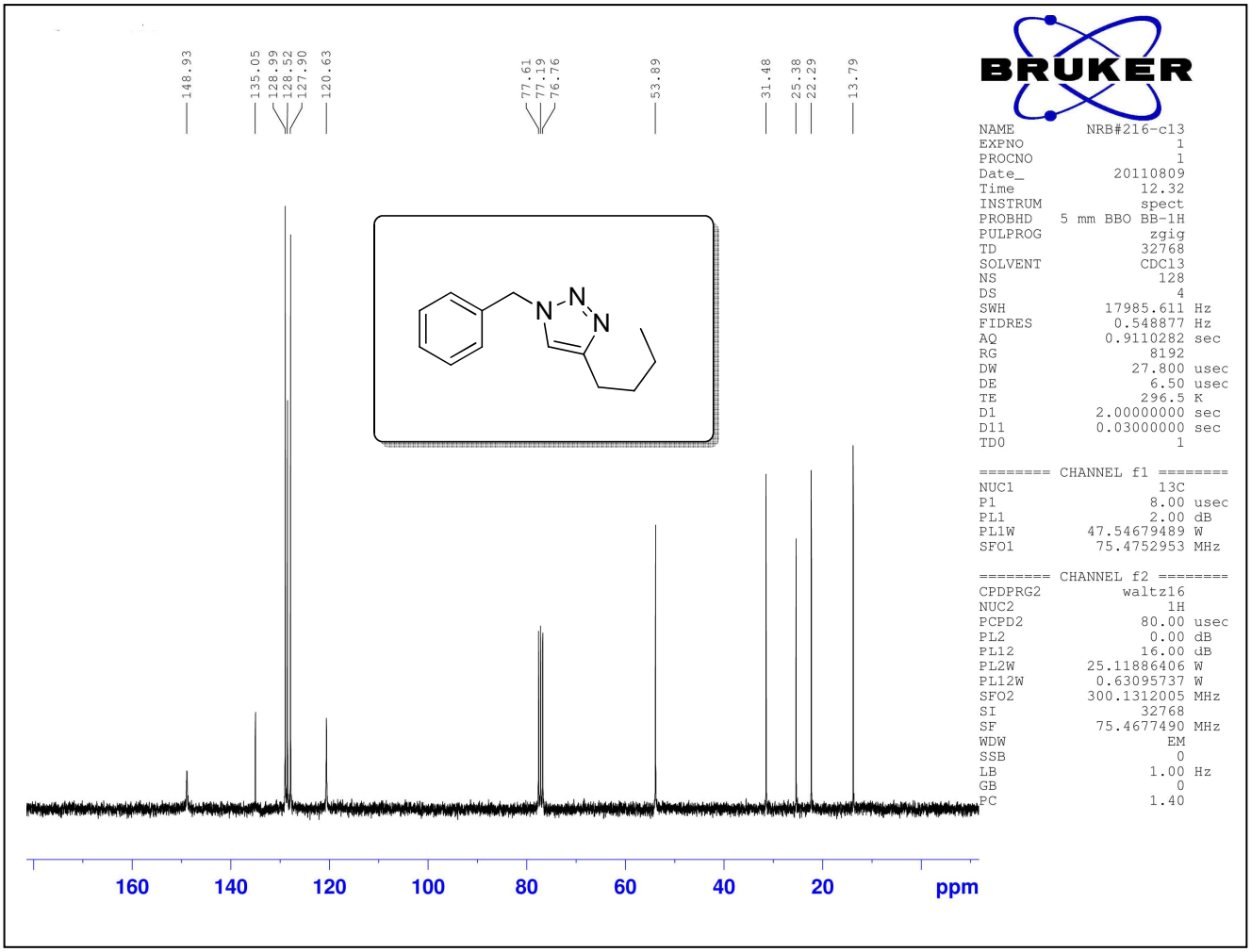

7.359
7.352
7.341
7.337
7.265
7.255
7.240
7.234
5.467
3.903
3.884
3.865
2.918
2.898
2.878
N
N
N
OH
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
5.77
2.00
2.24
0.98
2.37
BRUKER
NAME NRB#215
EXPNO 1
PROCNO 1
Date_ 20110803
Time 18.25
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 456.1
DW 80.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 0.00 dB
PL1W 25.11886406 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

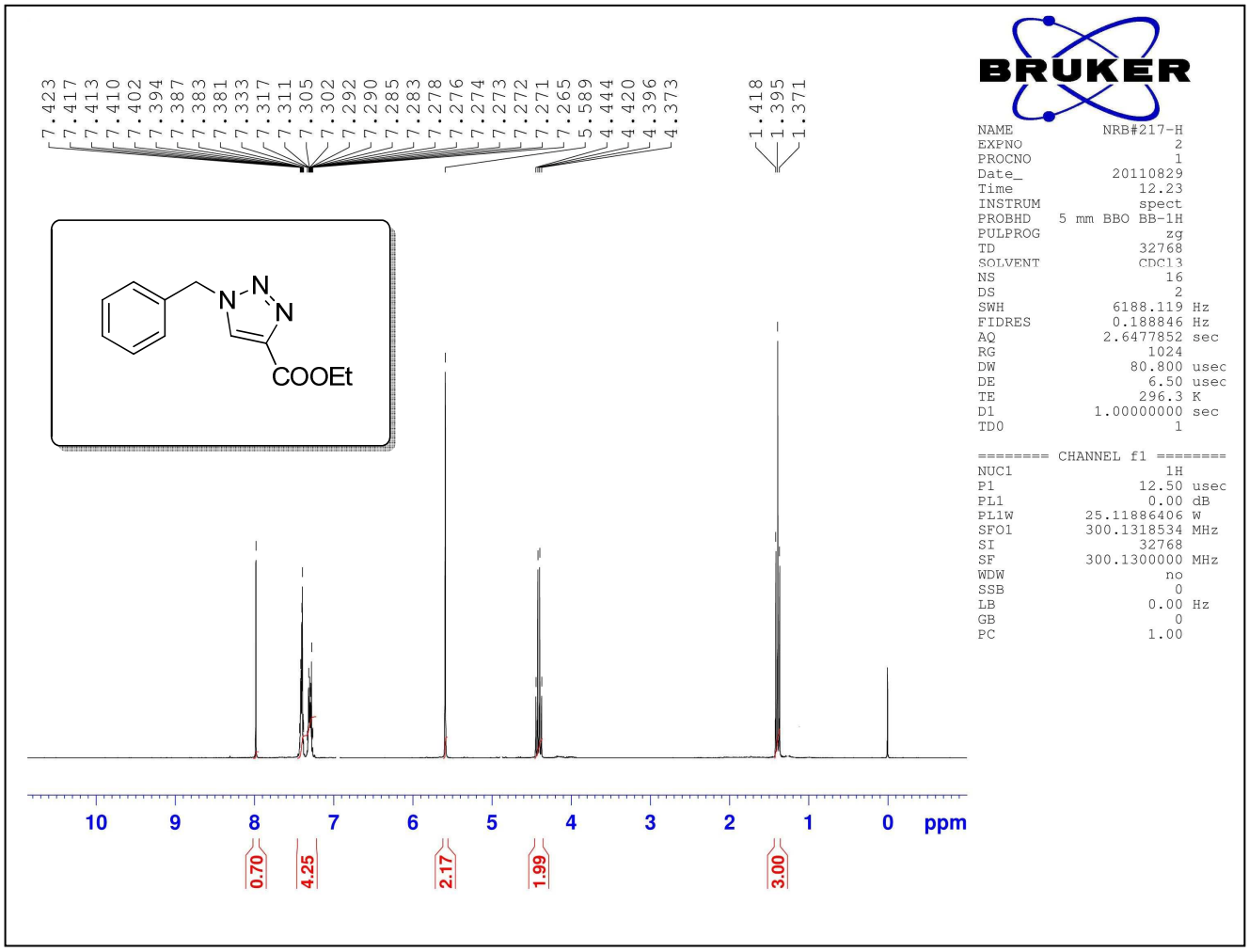


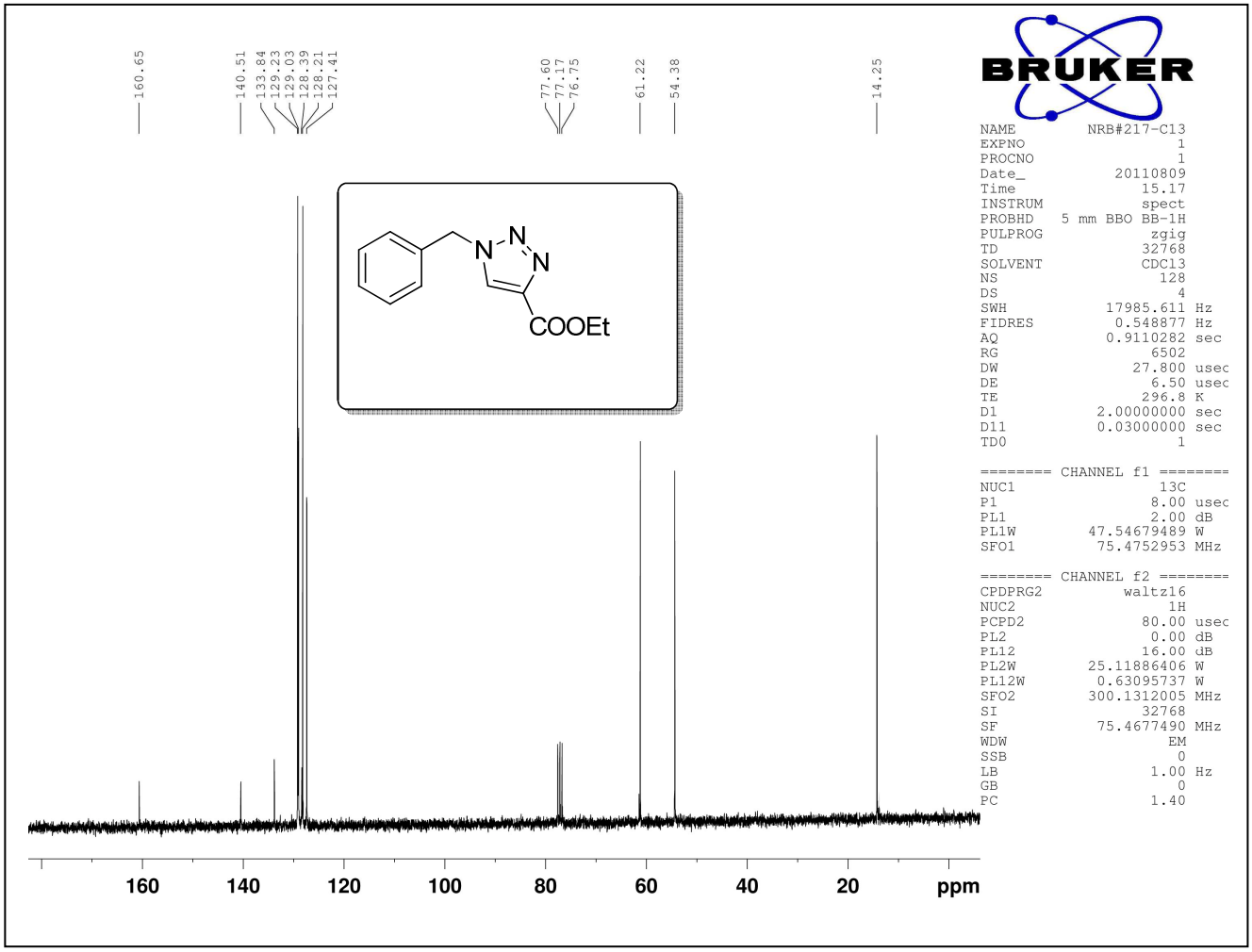
BRUKER

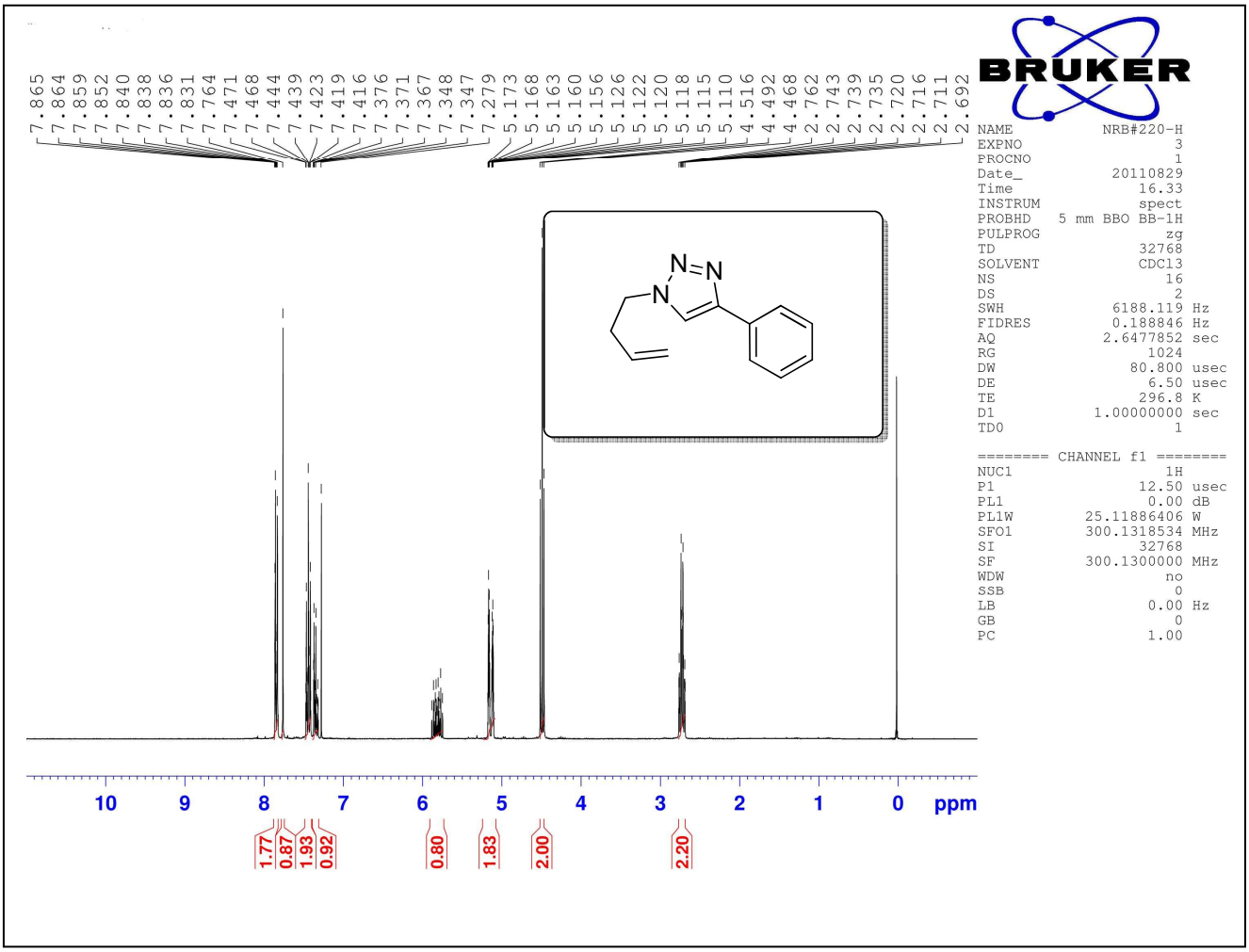
NAME	NRB#215-C13
EXPNO	2
PROCNO	1
Date_	20110803
Time	18.40
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	117
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	5792.6
DW	27.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	2.00 dB
PL1W	47.54679489 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	16.00 dB
PL2W	25.11886406 W
PL12W	0.63095737 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

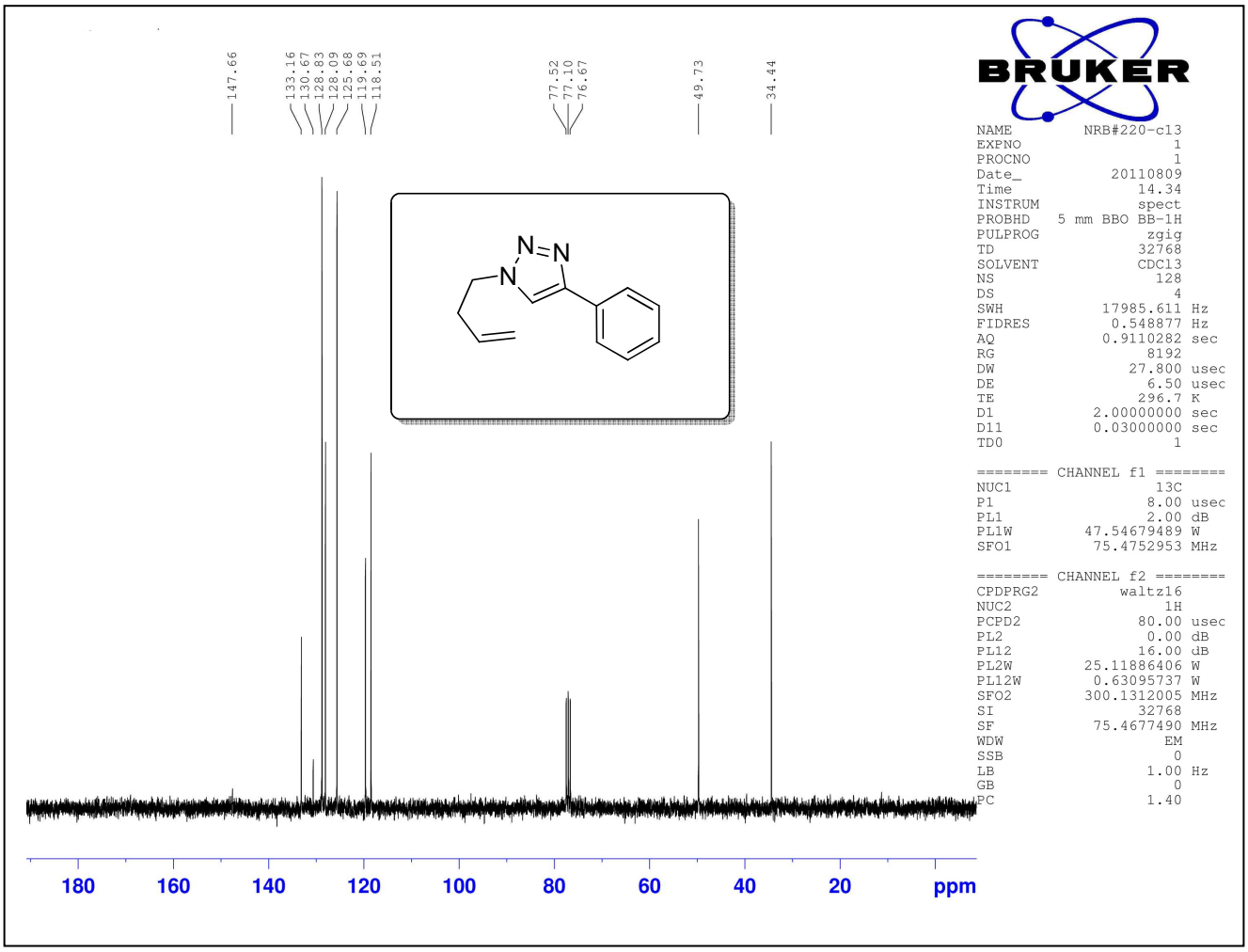

7.403
7.393
7.373
7.369
7.359
7.286
7.278
7.261
7.204
5.513
2.735
2.710
2.685
1.691
1.669
1.644
1.621
1.595
1.438
1.413
1.389
1.364
1.339
1.315
0.951
0.926
0.902
BRUKER
NAME NRB#216-H
EXPNO 6
PROCNO 1
Date_ 20110829
Time 13.01
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 1024
DW 80.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 0.00 dB
PL1W 25.11886406 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
N
N
N
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
4.45
1.03
2.00
1.97
2.42
2.09
2.86


148.93
135.05
128.99
128.52
127.90
120.63
77.61
77.19
76.76
53.89
31.48
25.38
22.29
13.79
BRUKER
NAME NRB#216-c13
EXPNO 1
PROCNO 1
Date_ 20110809
Time 12.32
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 128
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 sec
RG 8192
DW 27.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 2.00 dB
PL1W 47.54679489 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL2W 25.11886406 W
PL12W 0.63095737 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
N
N
160
140
120
100
80
60
40
20
ppm


7.423
7.417
7.413
7.410
7.402
7.394
7.387
7.383
7.381
7.333
7.317
7.311
7.305
7.302
7.292
7.290
7.285
7.283
7.278
7.276
7.274
7.273
7.272
7.271
7.265
5.589
4.444
4.420
4.396
4.373
1.418
1.395
1.371
N
N
N
COOEt
10
9
8
7
6
5
4
3
2
1
0
ppm
0.70
4.25
2.17
1.99
3.00
BRUKER
NAME NRB#217-H
EXPNO 2
PROCNO 1
Date_ 20110829
Time 12.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 1024
DW 80.800 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 0.00 dB
PL1W 25.11886406 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

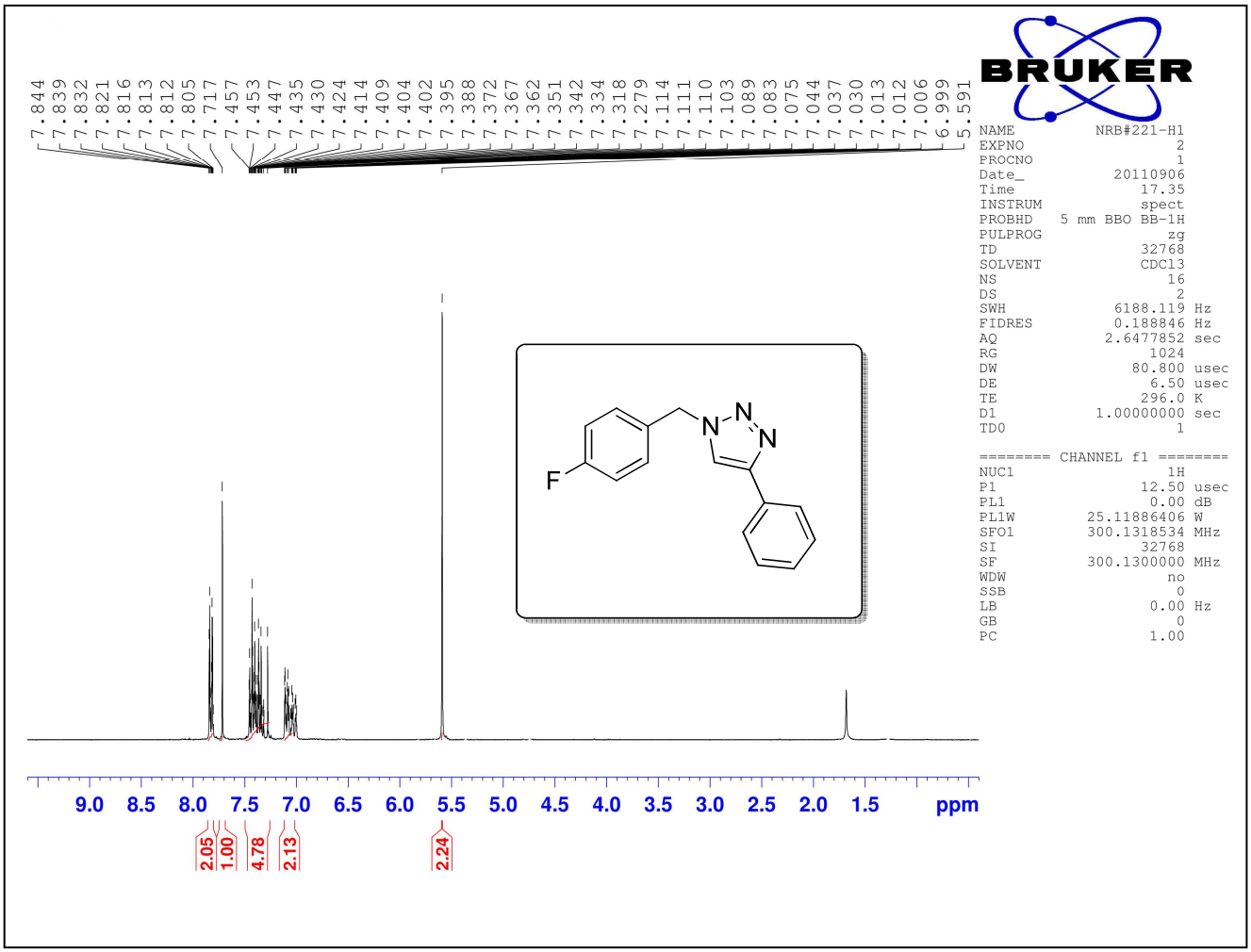


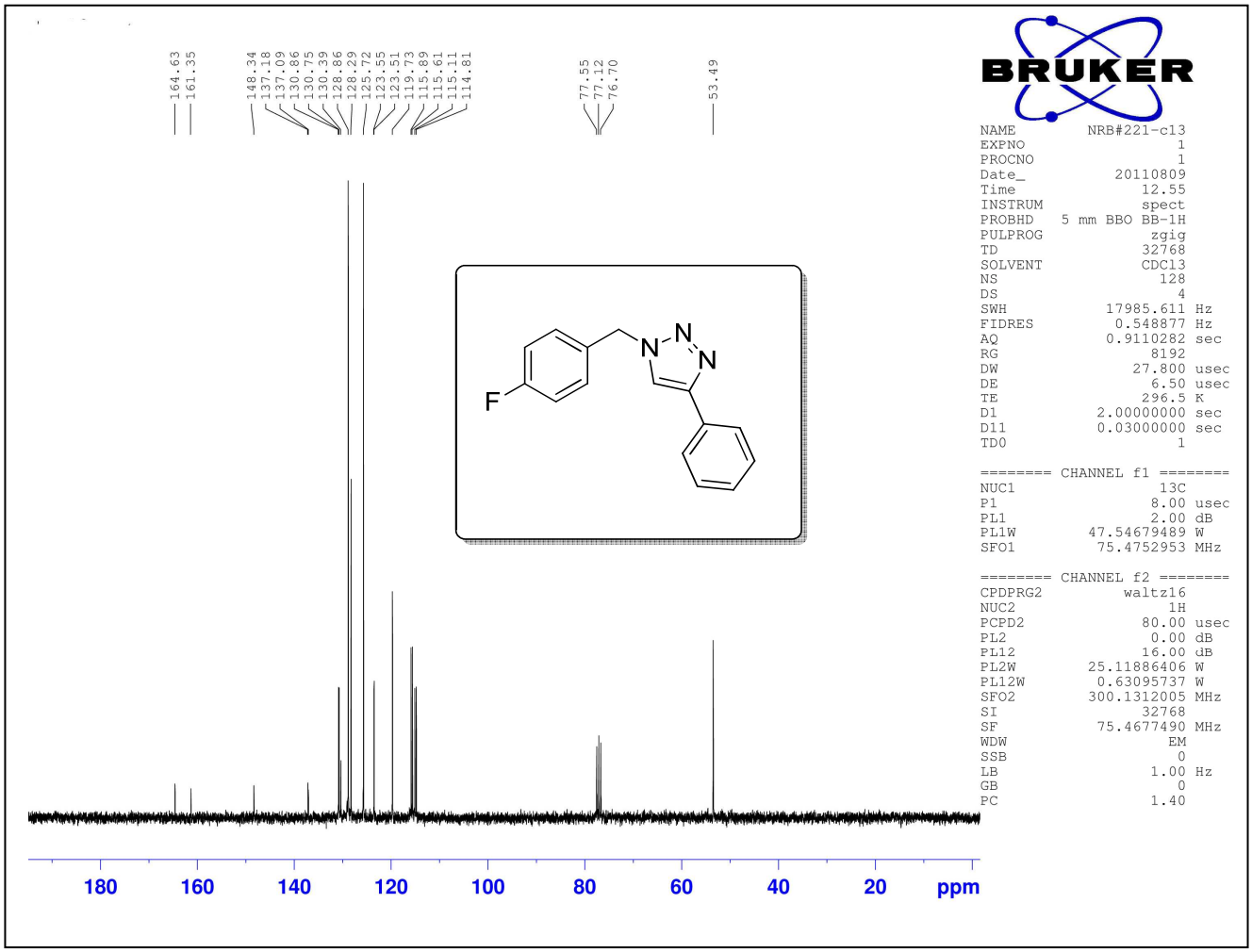




BRUKER

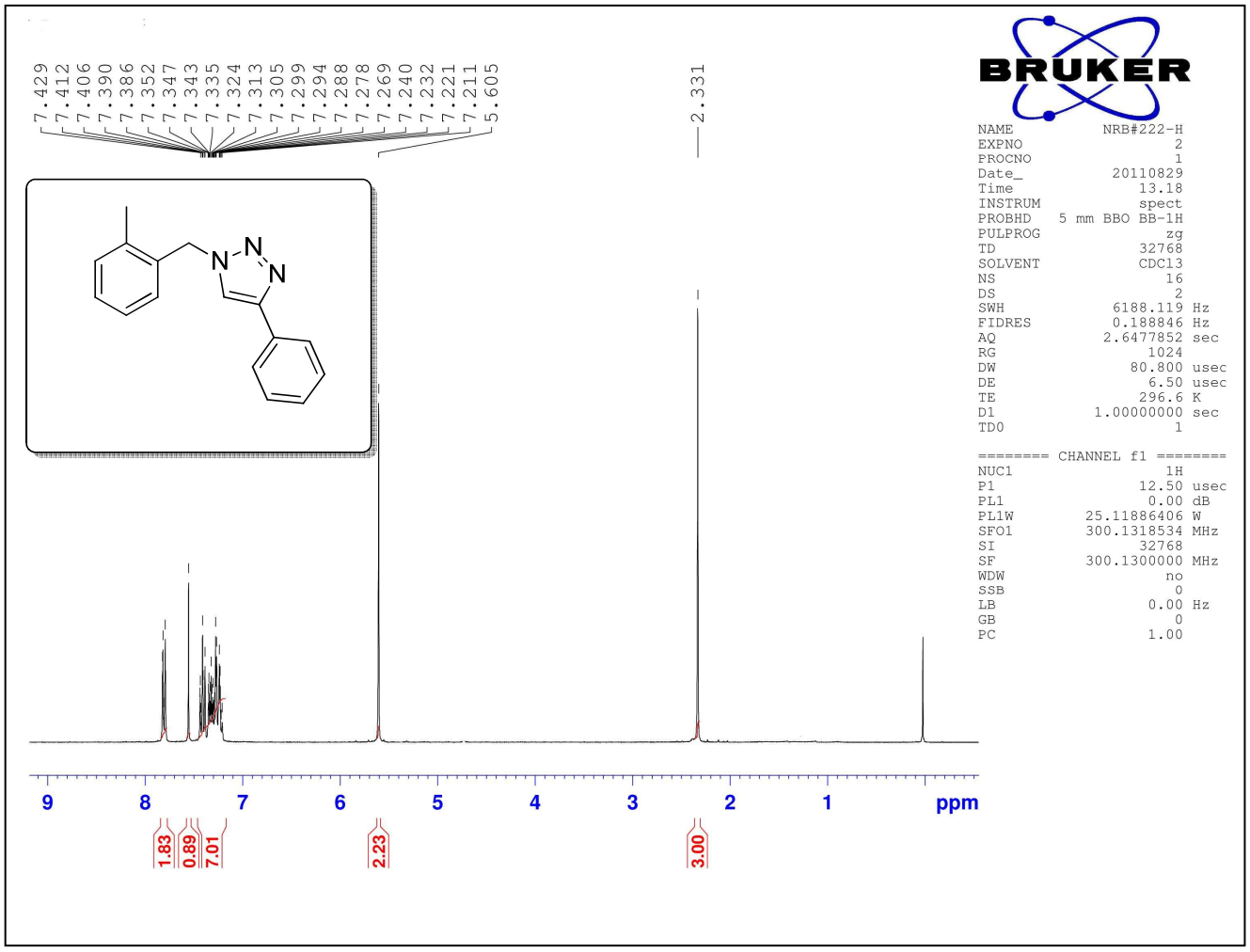
NAME	NRB#220-c13
EXPNO	1
PROCNO	1
Date_	20110809
Time	14.34
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	128
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	296.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	2.00 dB
PL1W	47.54679489 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	16.00 dB
PL2W	25.11886406 W
PL12W	0.63095737 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


7.844 7.839 7.832 7.821 7.816 7.813 7.812 7.805 7.717 7.457 7.453 7.447 7.435 7.430 7.424 7.414 7.409 7.404 7.402 7.395 7.388 7.372 7.367 7.362 7.351 7.342 7.334 7.318 7.279 7.114 7.111 7.110 7.103 7.089 7.083 7.075 7.044 7.037 7.030 7.013 7.012 7.006 6.999 5.591
BRUKER
NAME NRB#221-H1
EXPNO 2
PROCNO 1
Date_ 20110906
Time 17.35
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 1024
DW 80.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 0.00 dB
PL1W 25.11886406 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
F
N
N
N
9.0 8.5 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 ppm
2.05
1.00
4.78
2.13
2.24



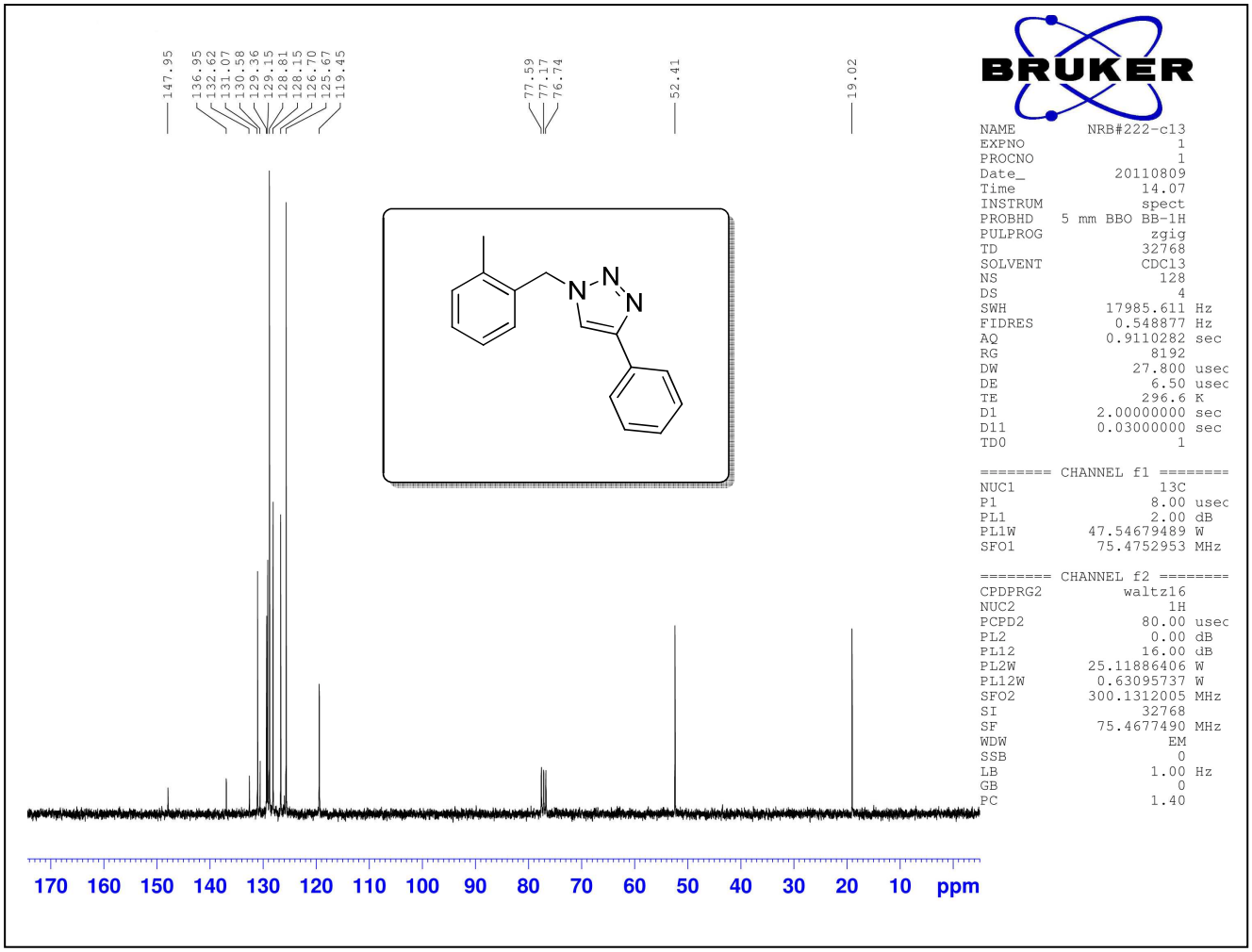
BRUKER

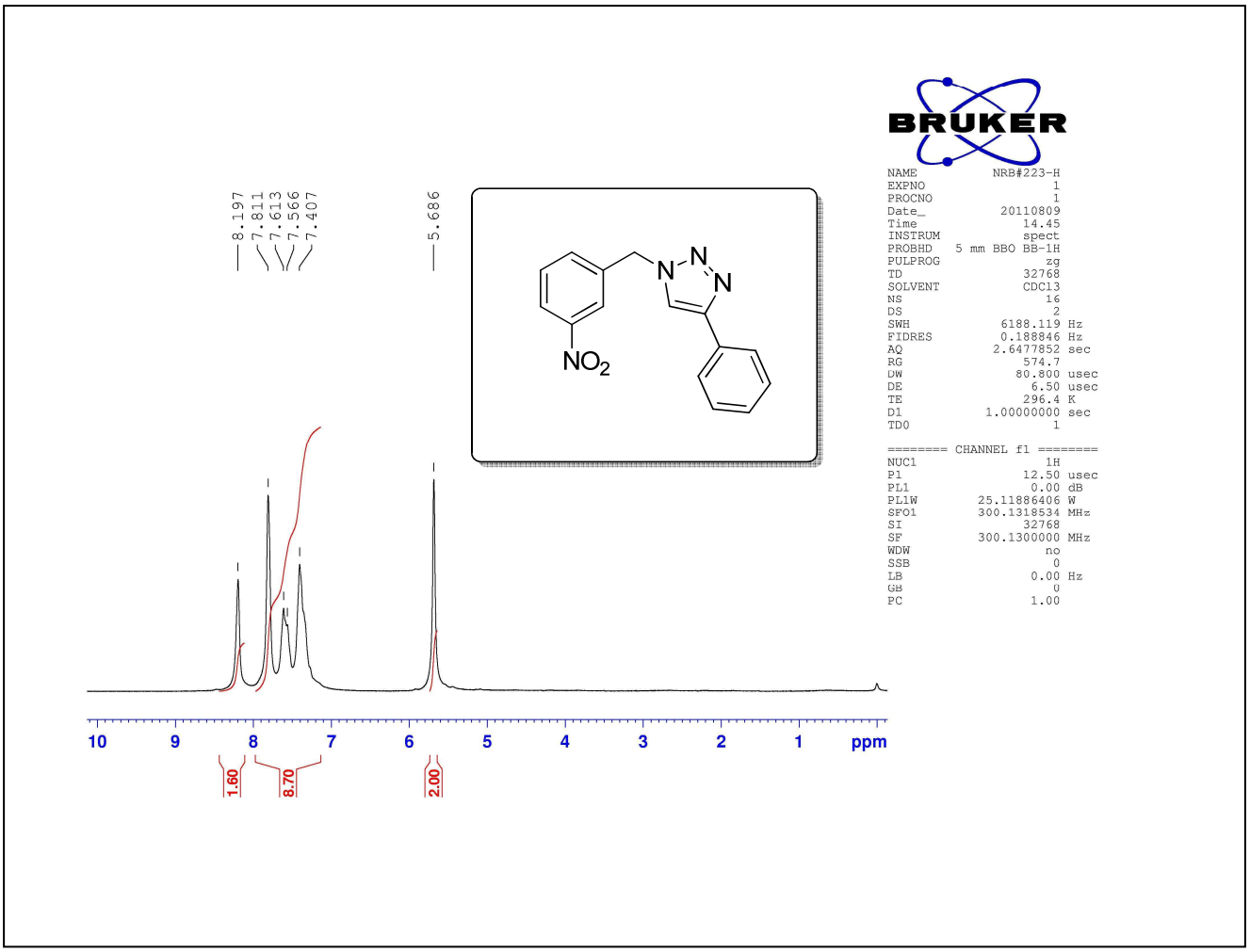
NAME	NRB#221-c13
EXPNO	1
PROCNO	1
Date_	20110809
Time	12.55
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	128
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	296.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	2.00 dB
PL1W	47.54679489 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	16.00 dB
PL2W	25.11886406 W
PL12W	0.63095737 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

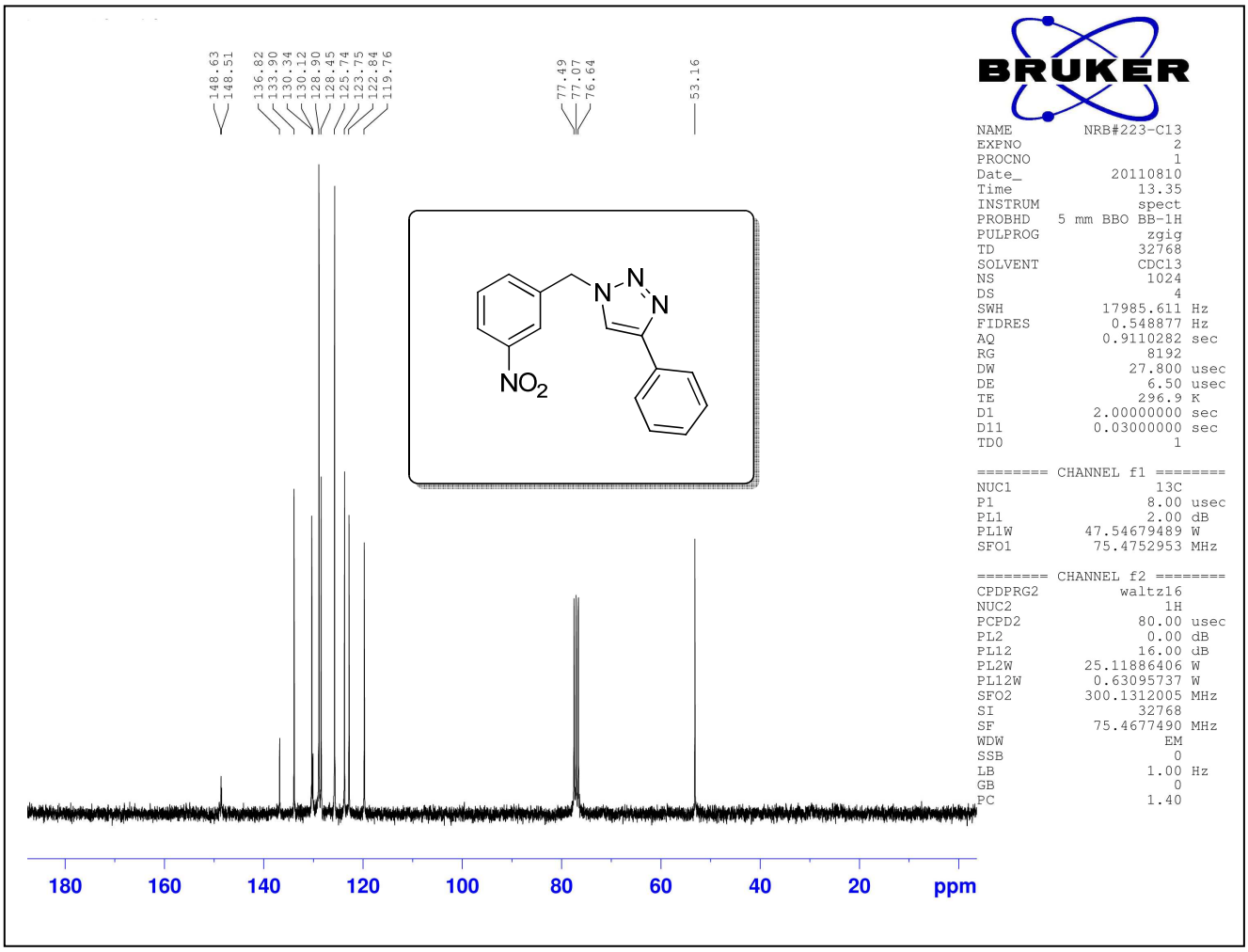
NAME	NRB#222-H
EXPNO	2
PROCNO	1
Date_	20110829
Time	13.18
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6188.119 Hz
FIDRES	0.188846 Hz
AQ	2.6477852 sec
RG	1024
DW	80.800 usec
DE	6.50 usec
TE	296.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.50 usec
PL1	0.00 dB
PL1W	25.11886406 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

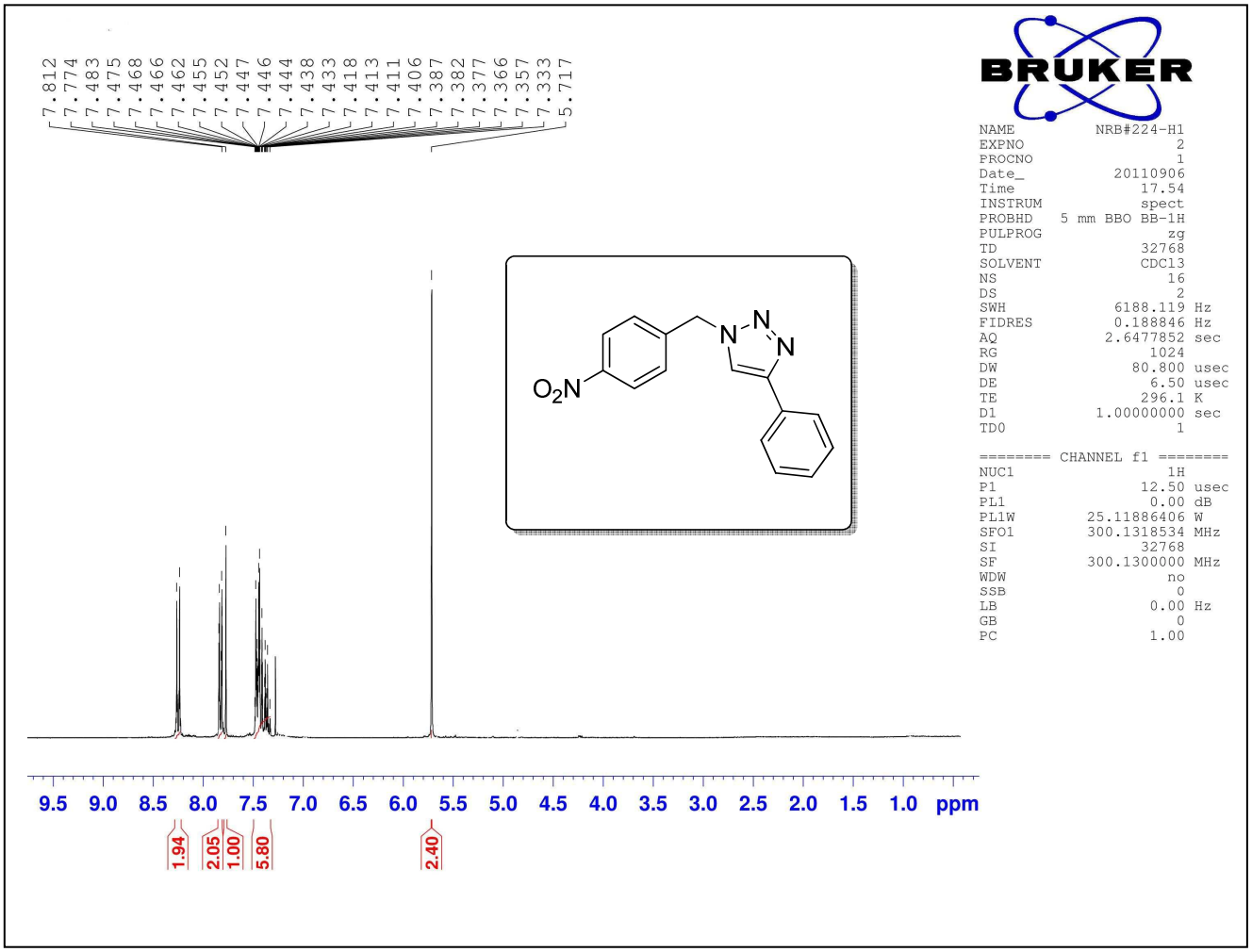


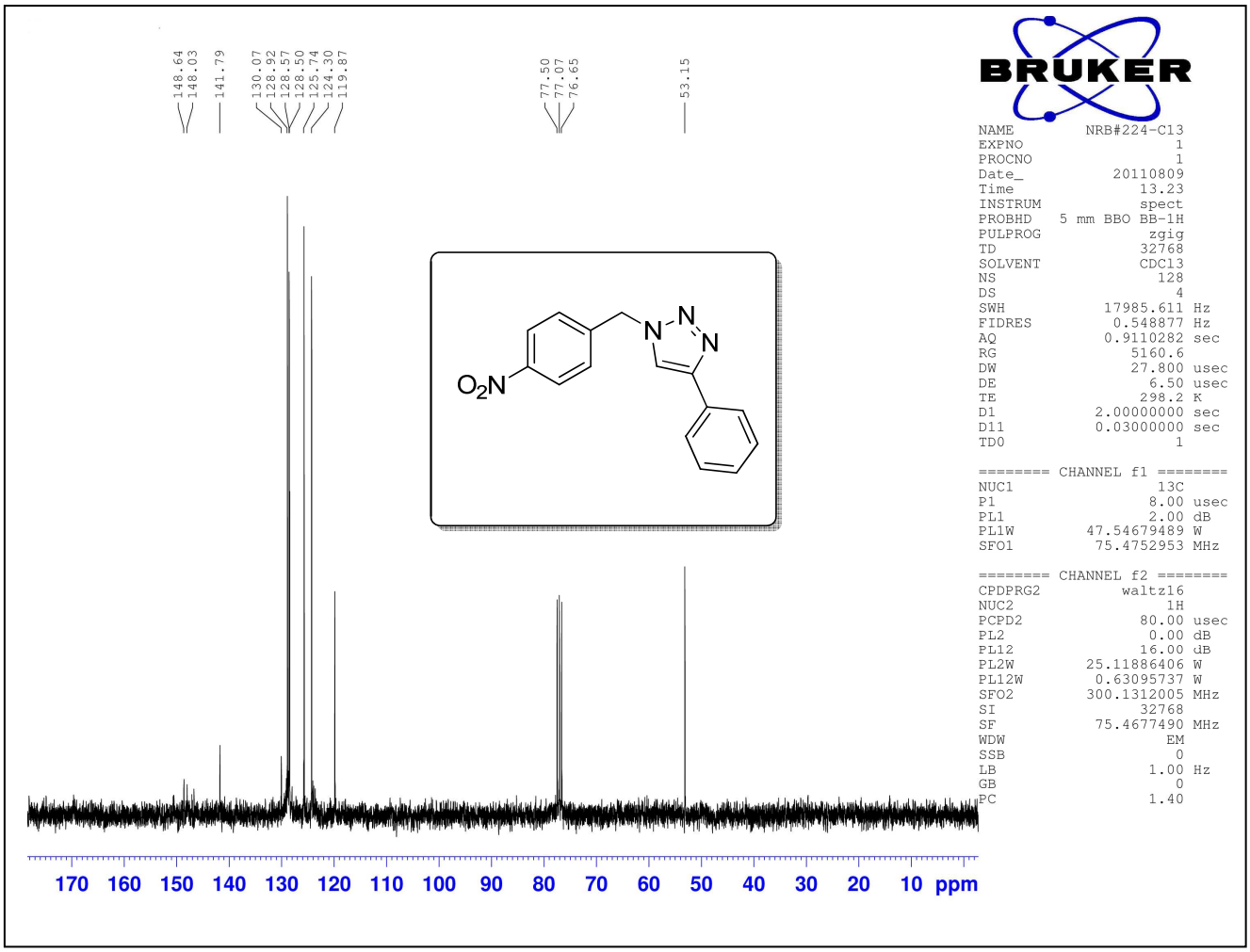


BRUKER

NAME	NRB#223-H
EXPNO	1
PROCNO	1
Date_	20110809
Time	14.45
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6188.119 Hz
FIDRES	0.188846 Hz
AQ	2.6477852 sec
RG	574.7
DW	80.800 usec
DE	6.50 usec
TE	296.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.50 usec
PL1	0.00 dB
PL1W	25.11886406 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00






148.64
148.03
141.79
130.07
128.92
128.57
128.50
125.74
124.30
119.87
77.50
77.07
76.65
53.15
BRUKER
NAME NRB#224-C13
EXPNO 1
PROCNO 1
Date_ 20110809
Time 13.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 128
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 sec
RG 5160.6
DW 27.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 2.00 dB
PL1W 47.54679489 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL2W 25.11886406 W
PL12W 0.63095737 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O2N
N
N
N
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm