B DENT GLOBAL
CUSTOM SYNTHESIS SPECIALISTS

Certificate of Analysis

B Dent Global Limited certifies that this reference material meets or exceeds the specifications stated in this data sheet.

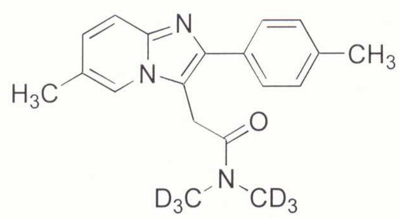
Barry R. Dent, Director.
10 September 2001

Name: Zolpidem-(N,N-*dimethyl-d*$_6$) (*N,N*,6-trimethyl-2-(4-methylphenyl)imidazo[1,2-a]pyridine-3-acetamide-(N,N-*dimethyl-d*$_6$) [as an incompletely characterised hydrobromide salt]

CAS Number: none

Lot Number: BDG 2410

Structure:



(free base)

Appearance: white to off-white crystalline solid

Chemical Purity: 98 + %

Isotopic Purity: Under 0.5% d_0

Molecular Weight: $C_{19}H_{15}D_6N_3O$ = 313.43 (free base)

Storage and Handling: Protect from light. Susceptible to static electricity.

Melting Point: The bulk of the sample melted at 280-288°C with decomposition occurring above 268°C.

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Identity and Purity:

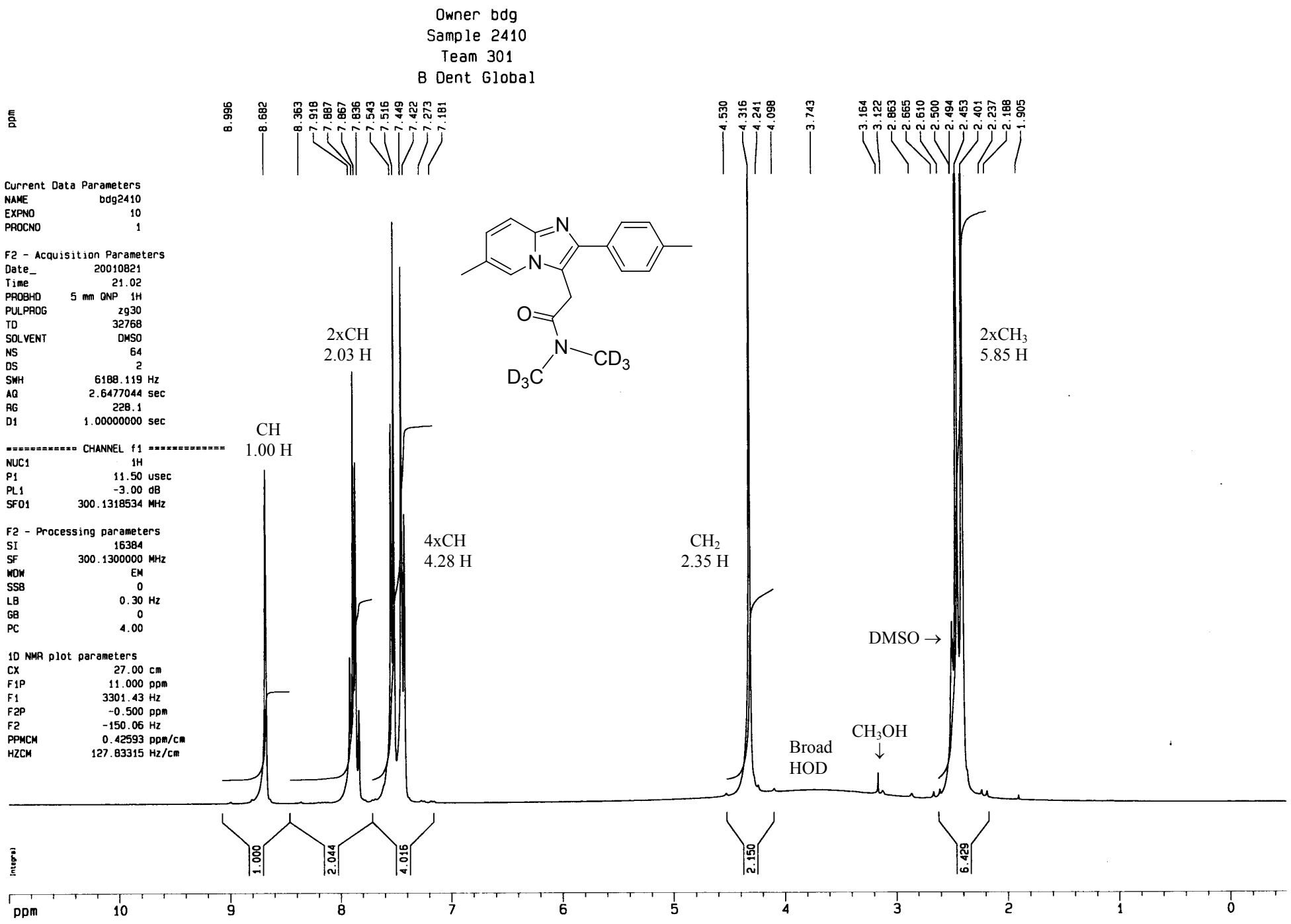
Neither the **carbon-13 and proton NMR** spectra (attached) disclose the presence of any impurities save for a small amount of methanol (0.1% w/w, a solvent used during crystallisation) and all signals exhibit the coupling patterns and parity expected for the specified compound. In the proton NMR, two very small signals are seen for the *N*-methyl protons, arising from protium impurities in the deuterated reagent used in the synthesis. In the carbon-13 NMR, the corresponding signals have collapsed to two low-intensity multiplets, indicating clean deuteration.

High-resolution mass spectrum (EI^+) Found: **m/z 313.2062**; predicted mass for $C_{19}H_{15}D_6N_3O$ ($M^{+\cdot}$) correct within -0.2 ppm. A peak at m/z 307 (d_0) is at the background level only.

HPLC: The attached chromatogram shows a relatively symmetrical peak at 7.02 minutes (**99.9 area %**). Two other peaks (2.23 and 2.59 minutes) were detected, but these were also present in the solvent blank.

Microanalysis: Found: C 55.11, H/D 7.20 (corrected for D), Br 18.78, N 10.00 %. Cl, not detected. $C_{19}H_{15}D_6N_3O.HBr.H_2O$ requires C 55.34, H 4.40, D 2.93, Br 19.38, N 10.19 %.

Note: The free base of the product was exposed to HCl and HBr during synthesis. When it was purified it appears to have chromatographed on normal phase silica as a salt. The elemental analyses most closely match those expected for a monohydrobromide monohydrate, and we believe it will be reasonable to use this composition when any correction for salts is applied during work with the compound.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Sample 2410
Team 301
B Dent Global
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8.363
7.918
7.887
7.867
7.836
7.543
7.516
7.449
7.422
7.273
7.181
4.530
4.316
4.241
4.098
3.743
3.164
3.122
2.863
2.665
2.610
2.500
2.494
2.453
2.401
2.237
2.188
1.905
Current Data Parameters
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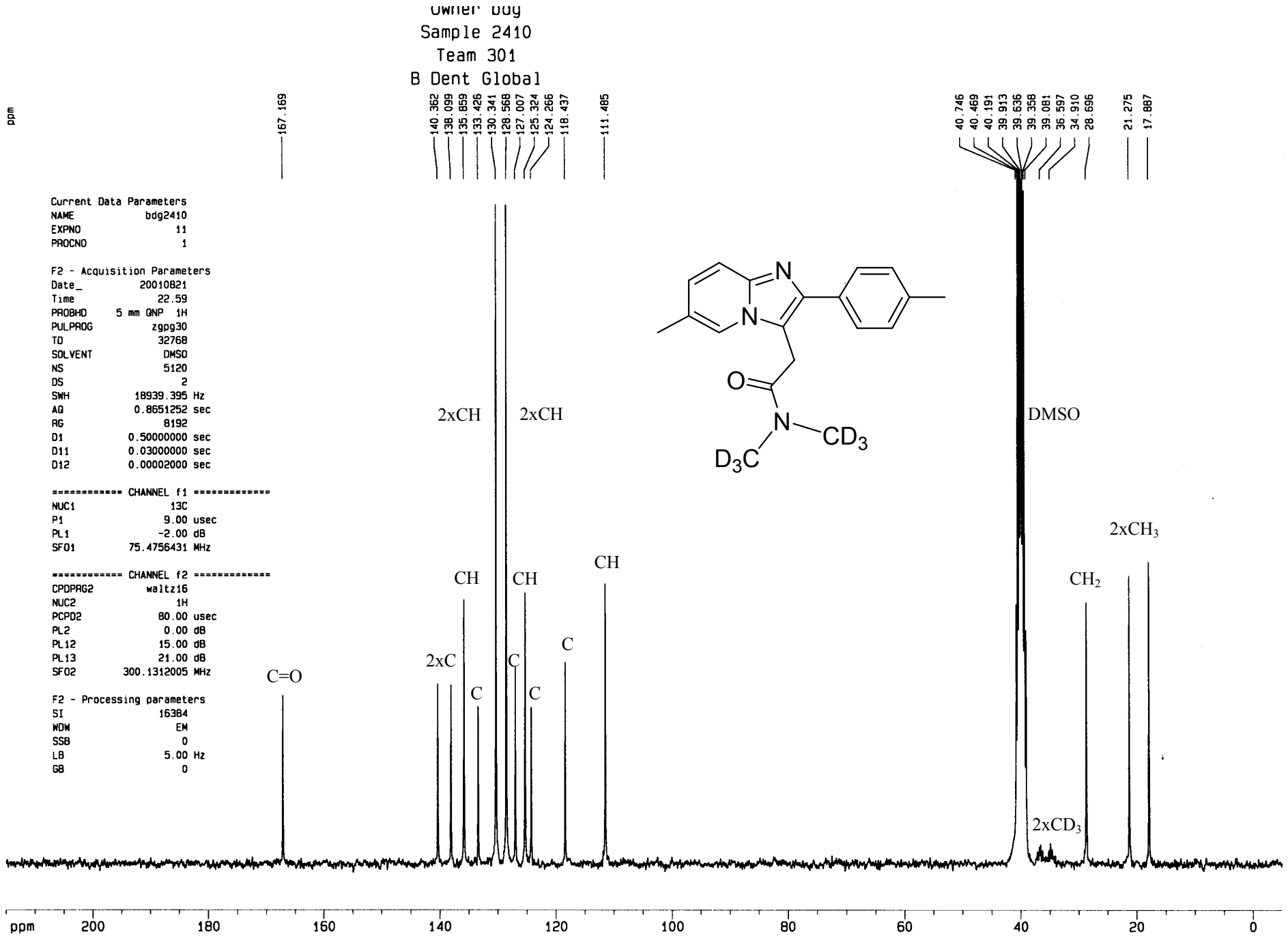
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010821
Time 21.02
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 64
DS 2
SWH 6188.119 Hz
AQ 2.6477044 sec
RG 228.1
D1 1.00000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 27.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.42593 ppm/cm
HZCM 127.83315 Hz/cm
N
N
O
N
CD3
D3C
CH
1.00 H
2xCH
2.03 H
4xCH
4.28 H
CH2
2.35 H
Broad
HOD
CH3OH
DMSO →
2xCH3
5.85 H
Integral
1.000
2.044
4.016
2.150
6.429
ppm
10
9
8
7
6
5
4
3
2
1
0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Team 301
B Dent Global
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127.007
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124.266
118.437
111.485
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40.469
40.191
39.913
39.636
39.358
39.081
36.597
34.910
28.696
21.275
17.887
Current Data Parameters
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PROCNO 1
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PULPROG zgpg30
TD 32768
SOLVENT DMSO
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DS 2
SWH 18939.395 Hz
AQ 0.8651252 sec
RG 8192
D1 0.50000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
========== CHANNEL f1 ==========
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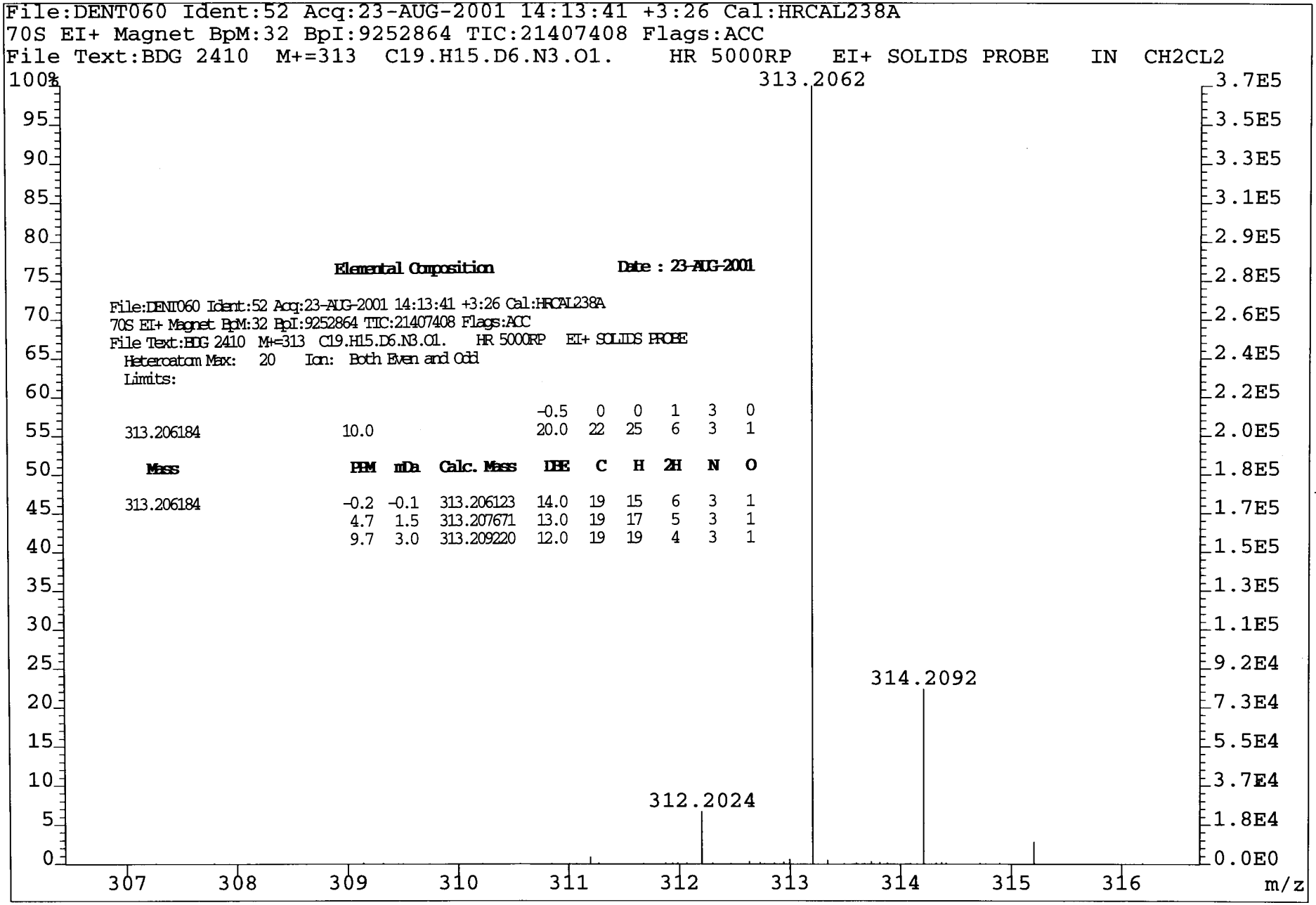
P1 9.00 usec
PL1 -2.00 dB
SF01 75.4756431 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 21.00 dB
SF02 300.1312005 MHz
F2 - Processing parameters
SI 16384
WDW EM
SSB 0
LB 5.00 Hz
GB 0
2xCH
2xCH
DMSO
CH
CH
CH
2xC
C
C
C
C=O
CH2
2xCH3
2xCD3
N
N
O
N
CD3
D3C
ppm
200
180
160
140
120
100
80
60
40
20
0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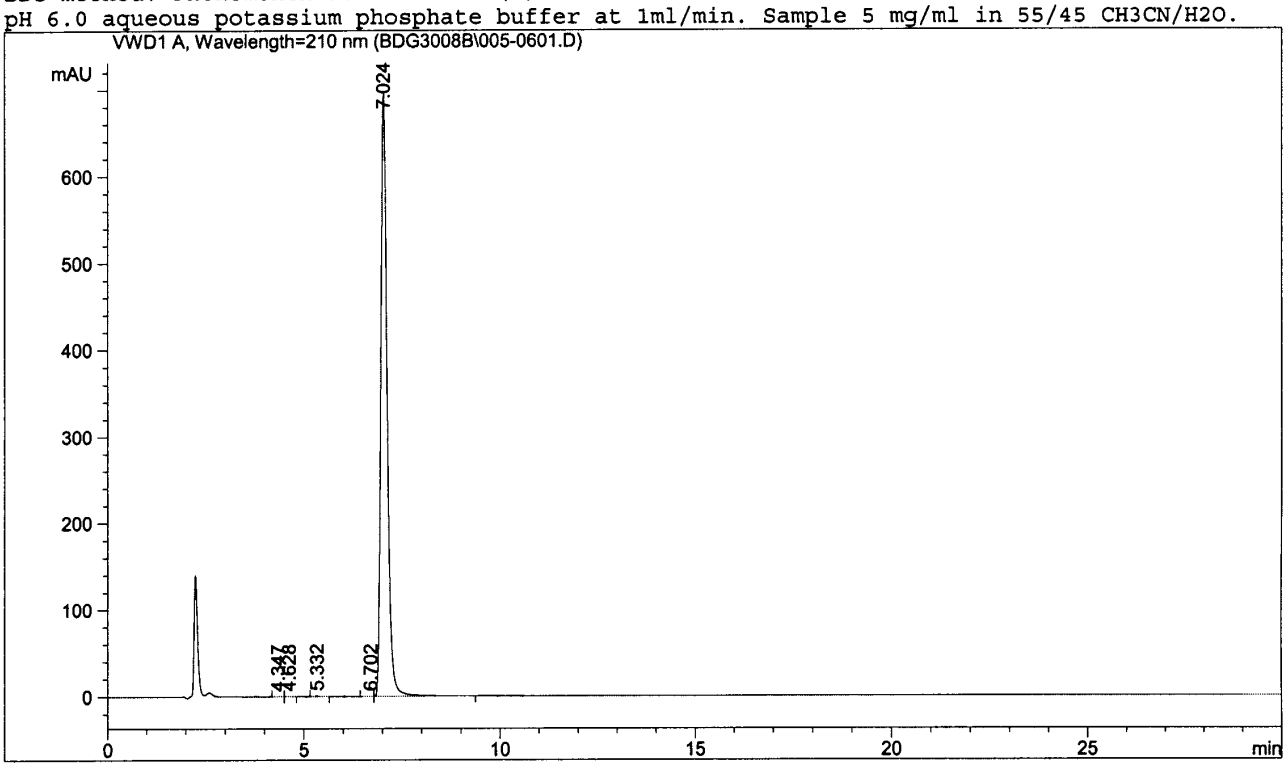
70S EI+ Magnet BpM:32 BpI:9252864 TIC:21407408 Flags:ACC
File Text:BDG 2410 M+=313 C19.H15.D6.N3.O1. HR 5000RP EI+ SOLIDS PROBE IN CH2CL2
313.2062
314.2092
312.2024
Elemental Composition
Date : 23-AUG-2001
File:DENT060 Ident:52 Acq:23-AUG-2001 14:13:41 +3:26 Cal:HRCAL238A
70S EI+ Magnet BpM:32 BpI:9252864 TIC:21407408 Flags:ACC
File Text:BDG 2410 M+=313 C19.H15.D6.N3.O1. HR 5000RP EI+ SOLIDS PROBE
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:
-0.5 0 0 1 3 0
313.206184 10.0 20.0 22 25 6 3 1
Mass PPM mDa Calc. Mass DBE C H 2H N O
313.206184 -0.2 -0.1 313.206123 14.0 19 15 6 3 1
4.7 1.5 313.207671 13.0 19 17 5 3 1
9.7 3.0 313.209220 12.0 19 19 4 3 1
100% 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
3.7E5 3.5E5 3.3E5 3.1E5 2.9E5 2.8E5 2.6E5 2.4E5 2.2E5 2.0E5 1.8E5 1.7E5 1.5E5 1.3E5 1.1E5 9.2E4 7.3E4 5.5E4 3.7E4 1.8E4 0.0E0
307 308 309 310 311 312 313 314 315 316 m/z

```
==========================================================================
Injection Date  : 8/30/2001 10:13:39 AM          Seq. Line :   6
Sample Name     : BDG 2410 10x dil                Location : Vial 5
Acq. Operator   : LKBadmin                            Inj :   1
                                                Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\2\METHODS\BDG002.M
Last changed    : 8/30/2001 10:13:02 AM by LKBadmin
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\BDG002.M
Last changed    : 8/30/2001 10:53:47 AM by LKBadmin
BDG method. Phenomenex Luna 5um C18(2) 250 x 4.6mm. 40:60 CH3CN
pH 6.0 aqueous potassium phosphate buffer at 1ml/min. Sample 5 mg/ml in 55/45 CH3CN/H2O.
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: VWD1 A, Wavelength=210 nm
```

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.347	MF	0.1277	1.08155	1.41182e-1	0.0139
2	4.628	FM	0.1562	2.14483	2.28849e-1	0.0277
3	5.332	MM	0.1506	5.04827	5.58525e-1	0.0651
4	6.702	MF	0.1591	1.33321	1.39626e-1	0.0172
5	7.024	FM	0.1852	7746.14648	697.22906	99.8761
Totals :				7755.75434	698.29725	

```
 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```