BDG SYNTHESIS

Certificate of Analysis

This material is a research-grade material prepared by custom synthesis. The quantity available is limited, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of research-grade materials. Research materials often contain higher levels of residual solvents and/or water, and we urge you to use the corrected purity where needed rather than the raw HPLC purity.

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

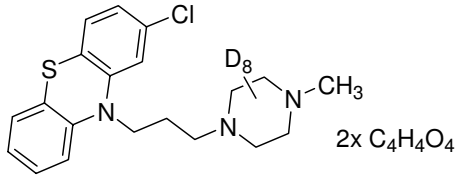
Barry Dent

Barry R. Dent, PhD, Director
27 July 2004

Name: Prochlorperazine-d_8 dimaleate

CAS Number: none (84-02-6 unlabelled)

Structure:



Molecular Weight: $C_{20}H_{16}D_8ClN_3S \bullet 2C_4H_4O_4$ = 614.14

Lot Number: BDG 4591

Appearance: Pale yellow, crystalline solid

Corrected Purity: 99.4 % (HPLC) – 0.2 % (methanol) = 99.2 %

Isotopic Purity: Under 0.5 % d_0

Expiry Date: 27 July 2009
Because of the small amount of material available it is not possible to perform formal storage stability studies. This expiry date is assigned from experience gained with the material in the laboratory and/or on storage.

Storage and Handling:
Temperature: ambient laboratory temperature; may be refrigerated.
Humidity: not believed to be hygroscopic; may be handled in normal laboratory atmosphere.
Light: store in an amber vial and protect from bright light.
Caution: Only experienced laboratory personnel should handle the material.

• Custom synthesis of analytical reference standards, metabolites, stable isotope labelled compounds • Contract research • BDG Synthesis is a division of B Dent Global Limited

Mailing:
BDG Synthesis
PO Box 38 627, Wellington Mail Centre,
Wellington, New Zealand.

Shipping:
BDG Synthesis
Gracefield Research Centre, Building F,
Gracefield Road, Lower Hutt, New Zealand.

Phone: + 64 4 569 0520
Fax: + 64 4 569 0521

info@bdg.co.nz
www.bdg.co.nz

Identity and Purity:

Source of Material
The material was made by an unambiguous synthetic route, using literature procedures where possible; starting materials were purchased from reputable sources and all intermediates were checked for identity by NMR.

Proton NMR Spectrum
Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Isotopic labelling: signals at the site / sites of deuteration are absent, compared with what would be expected for unlabelled material, indicating clean deuteration.
Residual solvents: a small amount of methanol (0.2 % *w/w*) is observed.
Impurities: no significant impurities are evident in the spectrum.

Carbon-13 NMR Spectrum
Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Isotopic labelling: signals at the sites of deuteration have collapsed to small multiplets compared with what would be expected for unlabelled material, indicating clean deuteration.

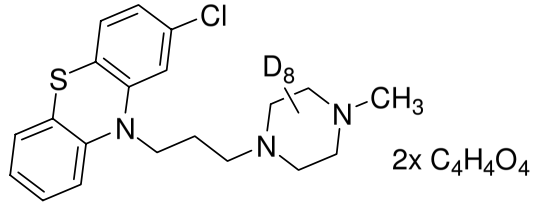
High-resolution mass spectrum (ESI+): found *m/z* 382.1932. $C_{20}H_{17}D_8ClN_3S$ $[M+H]^+$ requires *m/z* 382.1952. The deviation of 5.0 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for d_0 material was seen (detection limit about 0.5 %).

HPLC: A sharp, symmetrical peak is observed (99.4 area %). The non-integrated peak on the solvent front is maleic acid. Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis:	Found:	C 54.54, H 3.68, D 2.94, N 6.88 %
$C_{20}H_{16}D_8ClN_3S\bullet 2C_4H_4O_4$	requires:	C 54.76, H 3.94, D 2.62, N 6.84 %

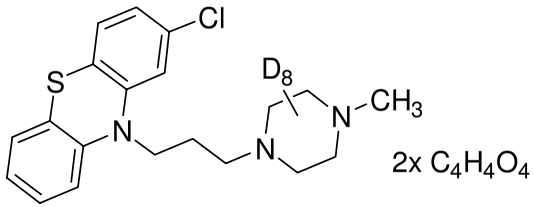
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

BDG SYNTHESIS



Proton NMR spectrum of Prochlorperazine-d_8 dimaleate (BDG4591) in DMSO-d_6.

BDG SYNTHESIS



Carbon-13 NMR spectrum of Prochloperazine-d_8 dimaleate (BDG 4591) in DMSO-d_6.

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==========================================================================
Injection Date  : 7/16/04 11:18:17 AM          Seq. Line :   2
Sample Name     : BDG4591                       Location : Vial 52
Acq. Operator   : YRLman                            Inj :   1
                                               Inj Volume : 10 µl
Acq. Method     : N:\LC1100_2\1\METHODS\LC40176A.M
Last changed    : 7/1/04 5:09:17 PM by YRLman
Analysis Method : N:\LC1100_2\1\METHODS\LC40176A.M
Last changed    : 7/16/04 1:13:49 PM by YRLman
                  (modified after loading)

BDG - isocratic analysis of prochlorperazine on Luna C18, 5um, 250 x 4.6mm ID.  # LC40176
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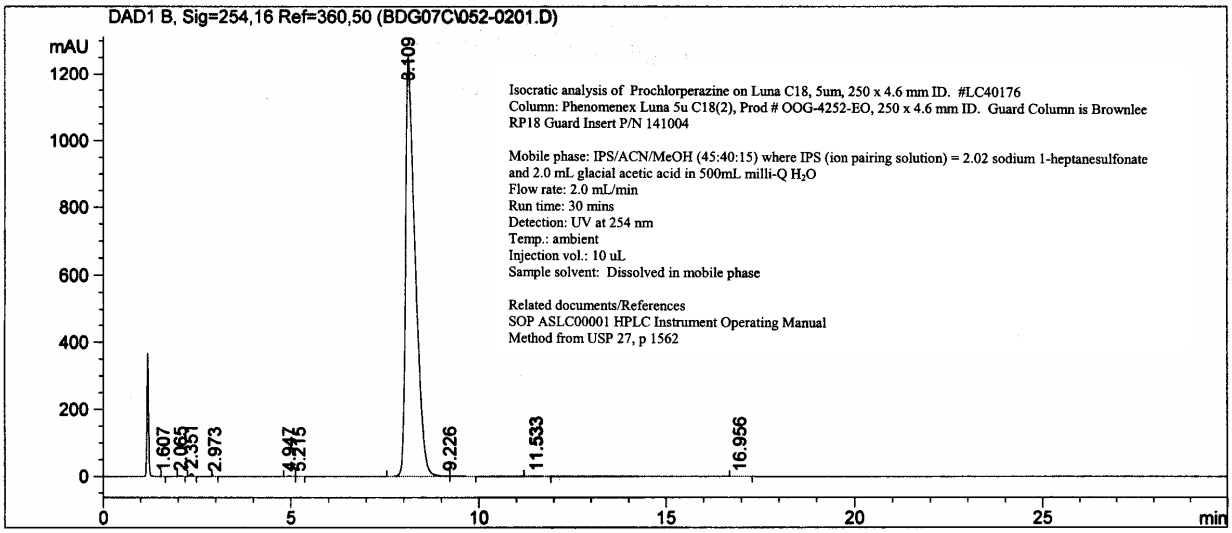


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==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,50
```

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.607	MM	0.0630	2.18071	5.76665e-1	9.782e-3
2	2.065	BB	0.0714	6.26325	1.36960	0.0281
3	2.351	BB	0.0741	39.87692	8.29801	0.1789
4	2.973	MM	0.0943	1.92773	3.40631e-1	8.647e-3
5	4.947	MF	0.1455	9.11727	1.04425	0.0409
6	5.215	FM	0.1425	2.06292	2.41213e-1	9.254e-3
7	8.109	MF	0.2953	2.21708e4	1251.14014	99.4520
8	9.226	MF	0.2881	33.21417	1.36065	0.1490
9	11.533	MM	0.3205	23.28146	1.21086	0.1044
10	16.956	MM	0.3919	4.23131	1.79930e-1	0.0190
Totals :				2.22930e4	1265.76194	

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