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.

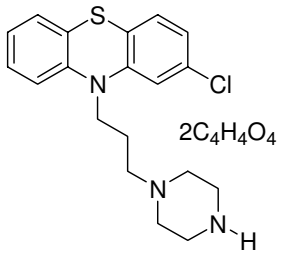
Barry Dent

Barry R. Dent, PhD, Director
11 August 2004

Name: *N*-Desmethylprochlorperazine dimaleate

CAS Number: 49780-18-9

Structure:



Molecular Weight: $C_{19}H_{22}ClN_3S \bullet 2C_4H_4O_4$ = 592.06

Lot Number: BDG 4609

Appearance: Pale yellow, crystalline solid

Purity by HPLC: 99.6 %

Expiry Date: 11 August 2005
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.
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

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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.
Residual solvents: a trace (under 0.1 %) of methanol is observed.
Impurities: no significant impurities are evident in the spectrum.

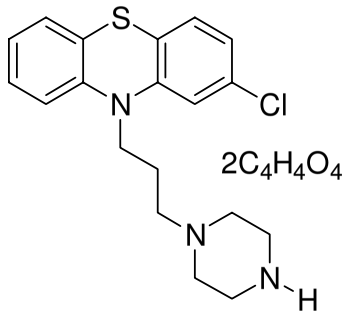
Carbon-13 NMR Spectrum
Identity: the signals are consistent with the proposed structure and in accord with literature where available.

High-resolution mass spectrum (ESI+): found *m/z* 360.1285. $C_{19}H_{23}ClN_3S$ $[M+H]^+$ requires *m/z* 360.1296. The deviation of 2.9 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC: A broad, symmetrical peak is observed (99.6 area %). The 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.

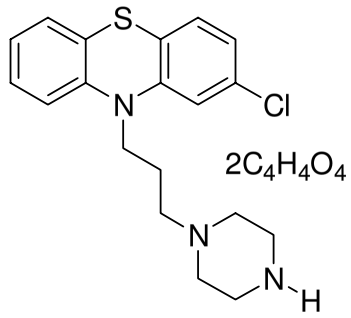
Elemental Analysis: Found: C 54.54, H 5.20, N 7.00 %
$C_{19}H_{22}ClN_3S \bullet 2C_4H_4O_4$ requires: C 54.77, H 5.11, N 7.10 %
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

BDG SYNTHESIS



Proton NMR spectrum of *N*-Desmethylprochlorperazine dimaleate (BDG 4609) in DMSO-d_6.

BDG SYNTHESIS



Carbon-13 NMR spectrum of
N-Desmethylprochlorperazine dimaleate
(BDG 4609) in DMSO-d_6.

Data File D:\HPCHEM\1\DATA\2004\AUG04\BDG08B\052-0301.D Sample Name: BDG4609

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==========================================================================
Injection Date  : 8/9/04 11:00:23 AM          Seq. Line :   3
Sample Name     : BDG4609                     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    : 8/9/04 1:18:12 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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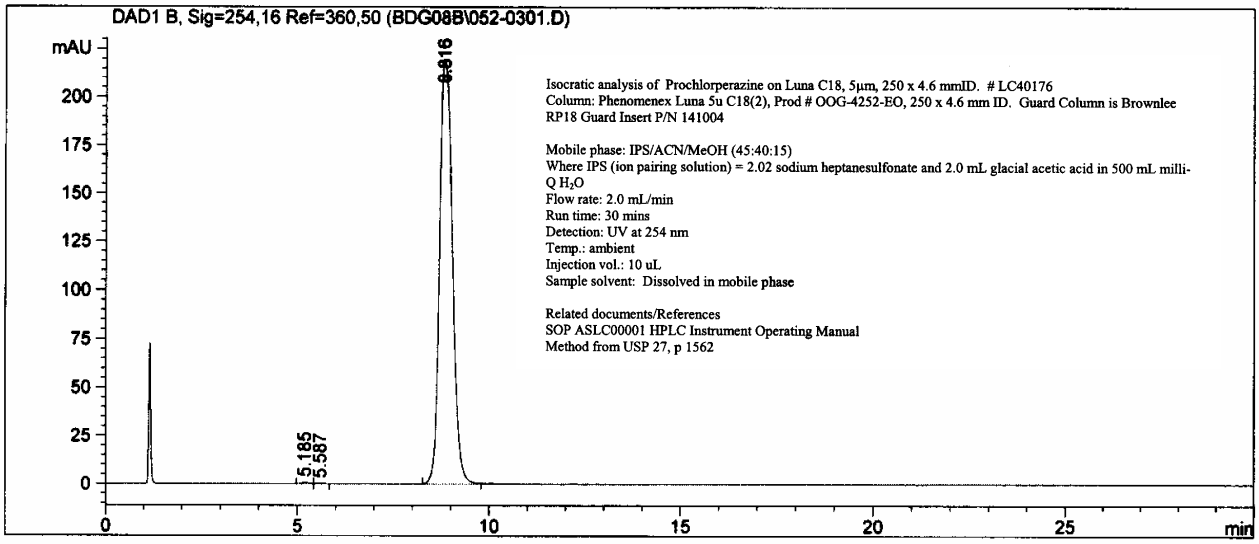


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==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.185 MF    0.2160   11.92665 9.20341e-1   0.2320
   2   5.587 FM    0.2745    4.26976 2.59206e-1   0.0830
   3   8.816 MM    0.3887 5125.30273  219.78038  99.6850

Totals :                   5141.49915  220.95993

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