



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.

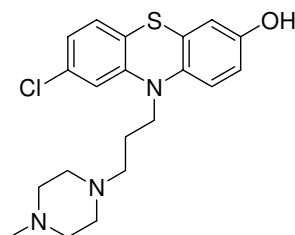
Barry Dent

Barry R. Dent, PhD, Director
15 December 2005

Name: 7-Hydroxyprochlorperazine

CAS Number: 52172-19-7

Structure:



Molecular Weight: $C_{20}H_{24}ClN_3OS = 389.95$

Lot Number: BDG 3912.6

Appearance: Tan, crystalline solid

Corrected Purity: 97.7 % (HPLC) – 7.6 % (acetone) = 90.1 %

Expiry Date: 15 December 2006

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.

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 considerable amount of acetone (4.9 % w/w) is observed.
Impurities: no significant impurities are evident in the spectrum.

Carbon-13 NMR Spectrum

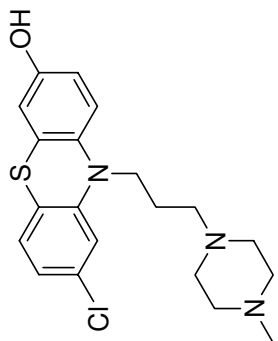
Identity: the signals are consistent with the proposed structure and in accord with literature where available.

High-resolution mass spectrum (EI+): found m/z 389.1329. $C_{20}H_{24}^{35}ClN_3OS [M]^+$ requires m/z 389.1329. The deviation of 0.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC: A sharp, symmetrical peak is observed (97.7 area %). 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.



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Proton NMR spectrum of 7-Hydroxyprochlorperazine
(BDG 3912.6) in CD₃OD

Acetone
4.9 % w/w

NCH₃
3.23 H

Methanol

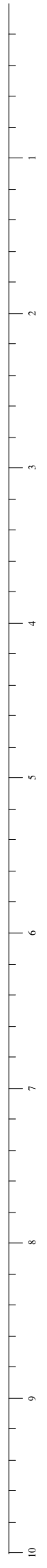
5xNCH₂
9.32 H

CH₂
2.10 H

HOD

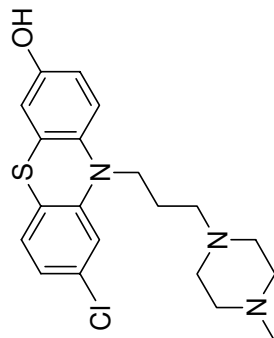
NCH₂
1.96 H

6xCH
6.02 H

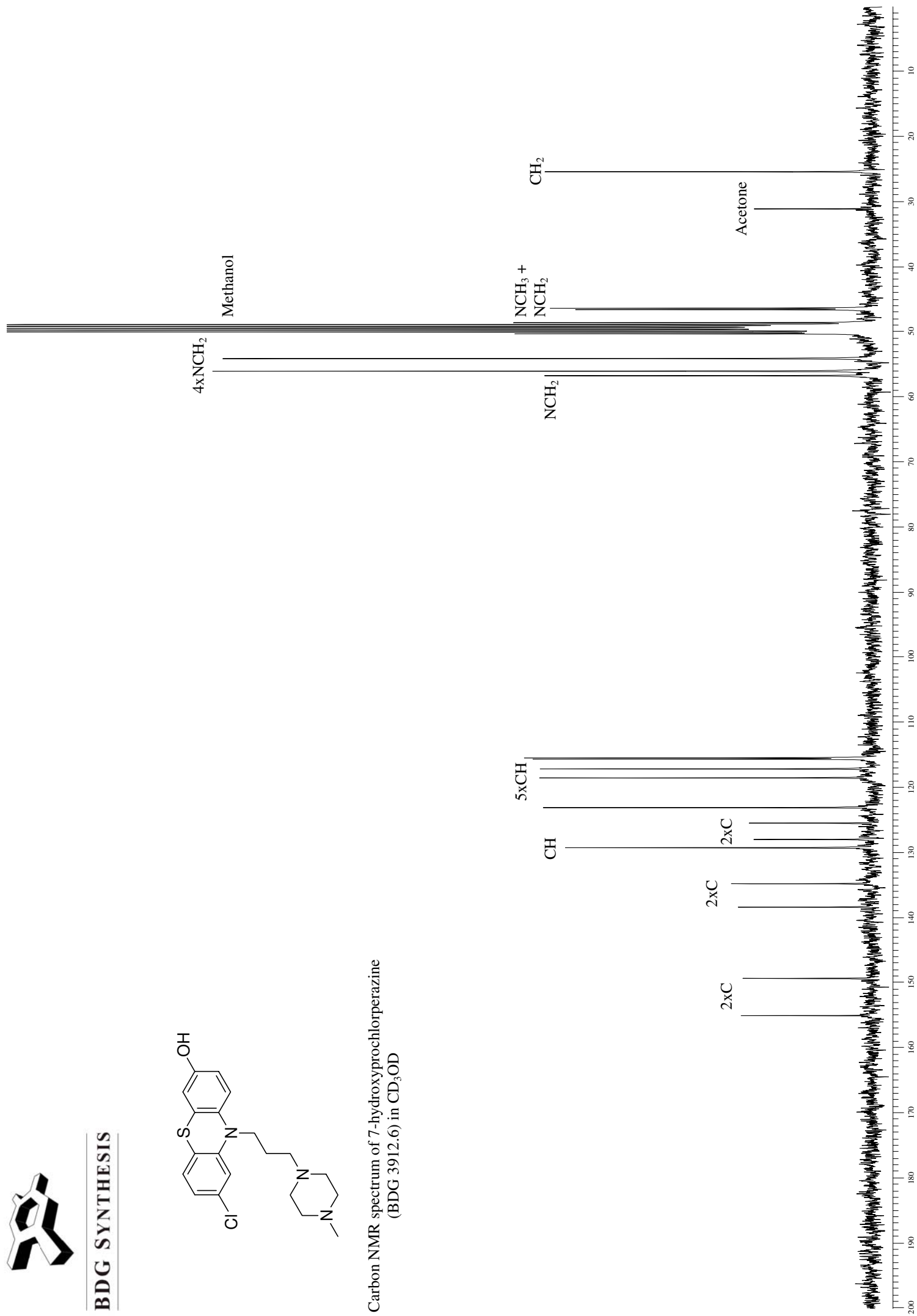




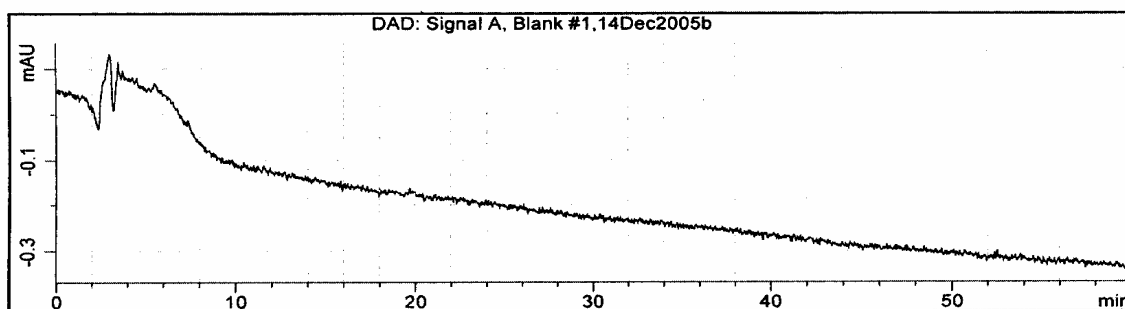
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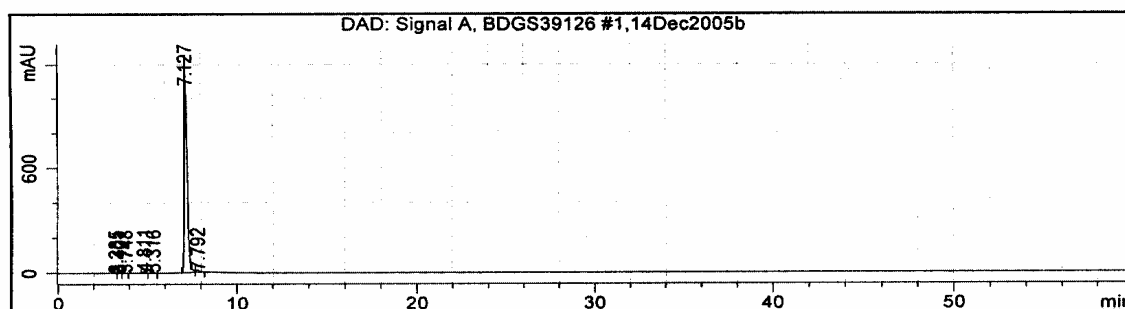
Carbon NMR spectrum of 7-hydroxyprochlorperazine
(BDG 3912.6) in CD₃OD



Signals of Blank #114Dec2005b



Signals of BDGS39126 #114Dec2005b



Results Table :

RT	Peak Type and Separation Code	Peak Area	Peak Height	Peak Width	Time Start	Time End	Symmetry
3.24	BV	6.4815	0.8156	0.1087	3.04	3.31	1.800
3.40	VB	6.6401	0.6846	0.1332	3.31	3.58	0.593
3.75	BB	6.9389	0.9227	0.1097	3.62	3.94	0.810
4.81	BB	78.3663	9.6799	0.1205	4.66	5.04	0.719
5.32	BB	36.3940	4.0571	0.1347	5.17	5.56	0.690
7.13	BV	13691.5202	1256.1834	0.1652	6.92	7.67	0.497
7.79	VB	181.1240	12.0616	0.2172	7.67	8.19	0.672

Initial Events :

Events Selected : Sequence Global DAD Events

Initial Event Name	Initial Event Value
Area Reject	0.0000
Slope Sensitivity	5.0000
Peak Width	0.0500
Shoulder Detection Mode	Disabled
Height Reject	0.0000
For All Signals	
Tail Peak Skim Height Ratio	0.00
Front Peak Skim Height Ratio	0.00
Skim Valley Ratio	20.00
Baseline Correction	Classical
Tangent Skim Mode	Standard

Summary

Peak Retention	Area % 254nm
3.24	0.05
3.40	0.05
3.75	0.05
4.81	0.56
5.32	0.26
7.13	97.74
7.79	1.29