

BDG SYNTHESIS

Certificate of Analysis

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

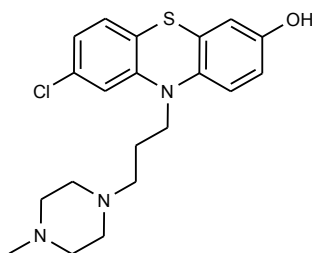
Barry Dent

Barry R. Dent, PhD, Director
11 February 2010

Name: 7-Hydroxyprochlorperazine

CAS Number: 52172-19-7

Structure:



Molecular Weight: $C_{20}H_{24}ClN_3OS = 389.94$

Lot Number: BDG 3912.6

Appearance: Tan, crystalline solid

Corrected Purity: 95.9 % (HPLC) - 6.5 % (acetone) = 89.4 %

Re-test Date: 11 February 2011

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

Proton NMR Spectrum

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

Residual Solvents: a small amount of acetone (6.5 % 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. $C_{20}H_{24}ClN_3OS$ $[M]^+$ 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 (95.9 %). 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 61.26, H 6.28, N 9.85 %
$C_{20}H_{24}ClN_3OS$	Requires:	C 61.60, H 6.20, N 10.78 %

The elemental analyses fall within generally accepted limits (+/- 0.4 %) for establishing the molecular formula given, except the result for nitrogen. 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).

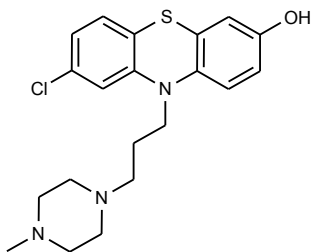
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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. This compound is intended for use as an analytical reference material and it is not for human administration. Structures are shown with relative stereochemistry unless otherwise specified.

The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

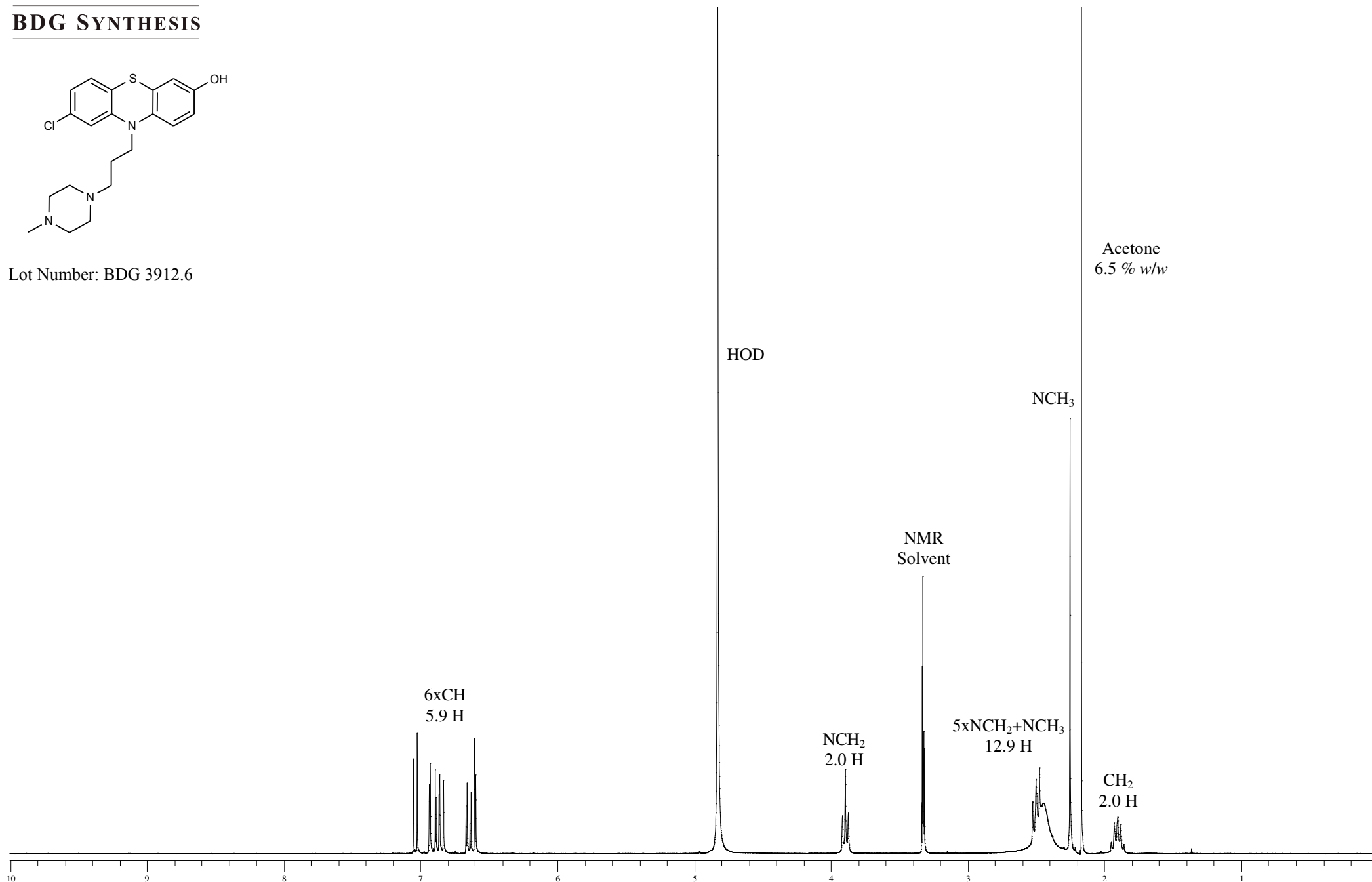


Proton NMR Spectrum of 7-Hydroxyprochlorperazine in Methanol-d₄

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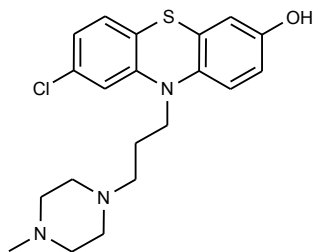
Lot Number: BDG 3912.6



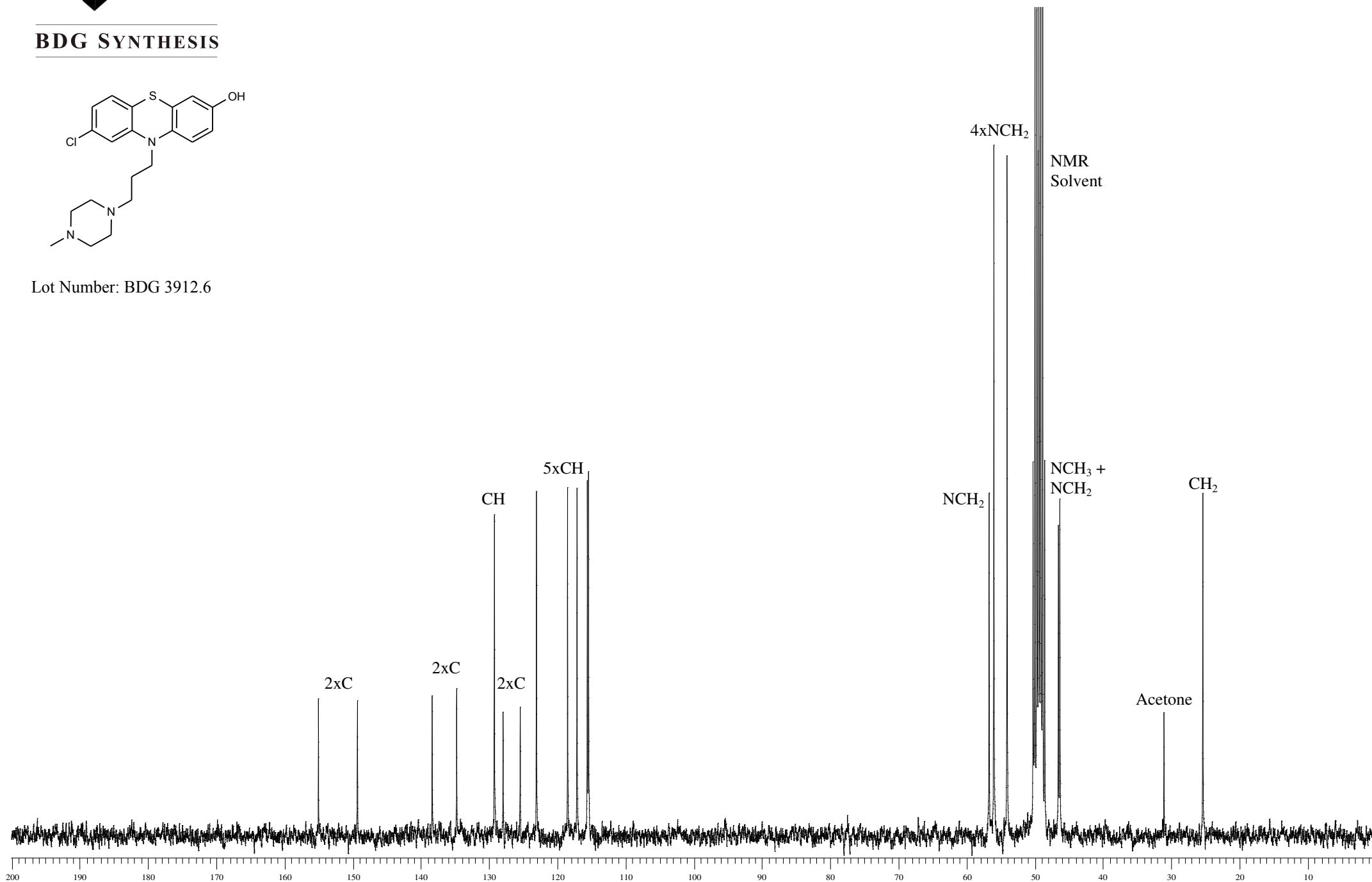


Carbon-13 NMR Spectrum of 7-Hydroxyprochlorperazine in Methanol-d₄

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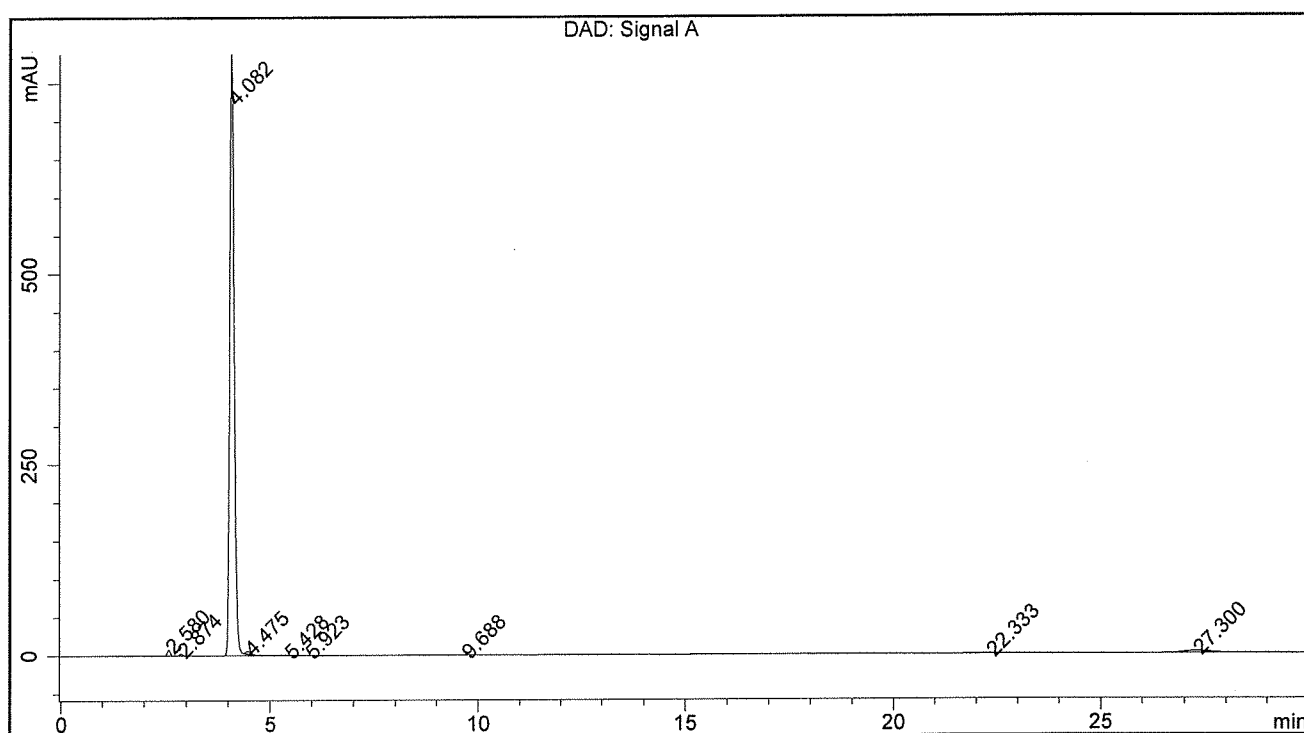
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BDG - Analysis of 7-Hydroxyprochlorperazine

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 45:40:15 IPS : Acetonitrile : Methanol
 IPS = 20 mM Heptanesulfonic Acid sodium salt + 70 mM Acetic Acid
 Flow Rate : 2.0 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 254 nm

Sample Name	BDG 3912.6	Instrument	AnalyticalLC01
Acquisition	11/02/2010, 09:03:29	Method (rev.)	LC10000a (4)
Sequence	BDG_11Feb2010a	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	2.58 min	7.8675	41.8858	0.0801 min	0.668 %
2	2.87 min	2.4696	14.1450	0.0888 min	0.226 %
3	4.08 min	795.4103	6010.5800	0.1162 min	95.925 %
4	4.47 min	3.4760	26.0074	0.1173 min	0.415 %
5	5.43 min	0.3801	4.4390	0.1642 min	0.071 %
6	5.92 min	0.4491	4.7209	0.1626 min	0.075 %
7	9.69 min	0.2857	5.3341	0.2478 min	0.085 %
8	22.33 min	0.4617	20.6456	0.5357 min	0.329 %
9	27.30 min	2.8305	138.1699	0.6675 min	2.205 %