



BDG SYNTHESIS

Certificate of Analysis

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

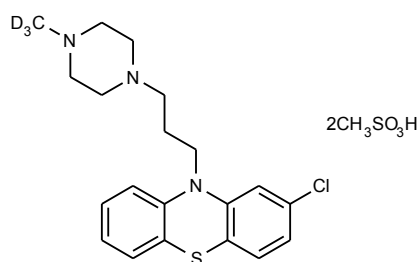
Neil Beare

Neil Beare, PhD, Director
15 March 2016

Name: Prochlorperazine-d₃ Dimesylate

CAS Number: 51888-09-6 (unlabelled)

Structure:



Molecular Weight: C₂₀H₂₁D₃ClN₃S · 2CH₄O₃S = 569.17

Lot Number: BDG 1464

Appearance: Off-white, crystalline solid

Corrected Purity: 98.9 % (HPLC) - 0.2 % (chloroform) - 2.2 % (water) = 96.5 %

Isotopic Purity: Under 0.5 % d₀

Re-test Date: 15 March 2021

Storage and Handling:

Temperature:	refrigerate for prolonged storage; may be handled and shipped at ambient temperature.
Humidity:	not believed to be hygroscopic; may be handled in normal laboratory atmosphere.
Light:	protect from strong sunlight.
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.

Isotopic Labelling: signals at the site of deuteration are absent, compared with what would be expected for unlabelled material, indicating clean deuteration.

Residual Solvents: a small amount of chloroform (0.2 % w/w) and a trace (under 0.1 % w/w) of ethanol are 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 site of deuteration have collapsed to small multiplets compared with what would be expected for unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (TOF MS ES+)

Found m/z 377.1644. $C_{20}H_{22}D_3ClN_3S$ $[M+H]^+$ requires m/z 377.1646. The deviation of 0.5 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 (98.9 %). 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 45.42, H 5.22, D 1.03, N 7.18 %
$C_{20}H_{21}D_3ClN_3S \cdot 2CH_4O_3S \cdot 0.7H_2O$	Requires:	C 45.42, H 5.27, D 1.04, N 7.22 %, H_2O 2.17 %
$C_{20}H_{21}D_3ClN_3S \cdot 2CH_4O_3S$	Requires:	C 46.42, H 5.14, D 1.06, N 7.38 %

The elemental analyses fall slightly outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the "best-fit" hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we recommend that the "best-fit" water content be used when determining corrected purity.

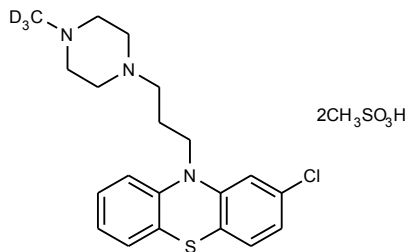
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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.

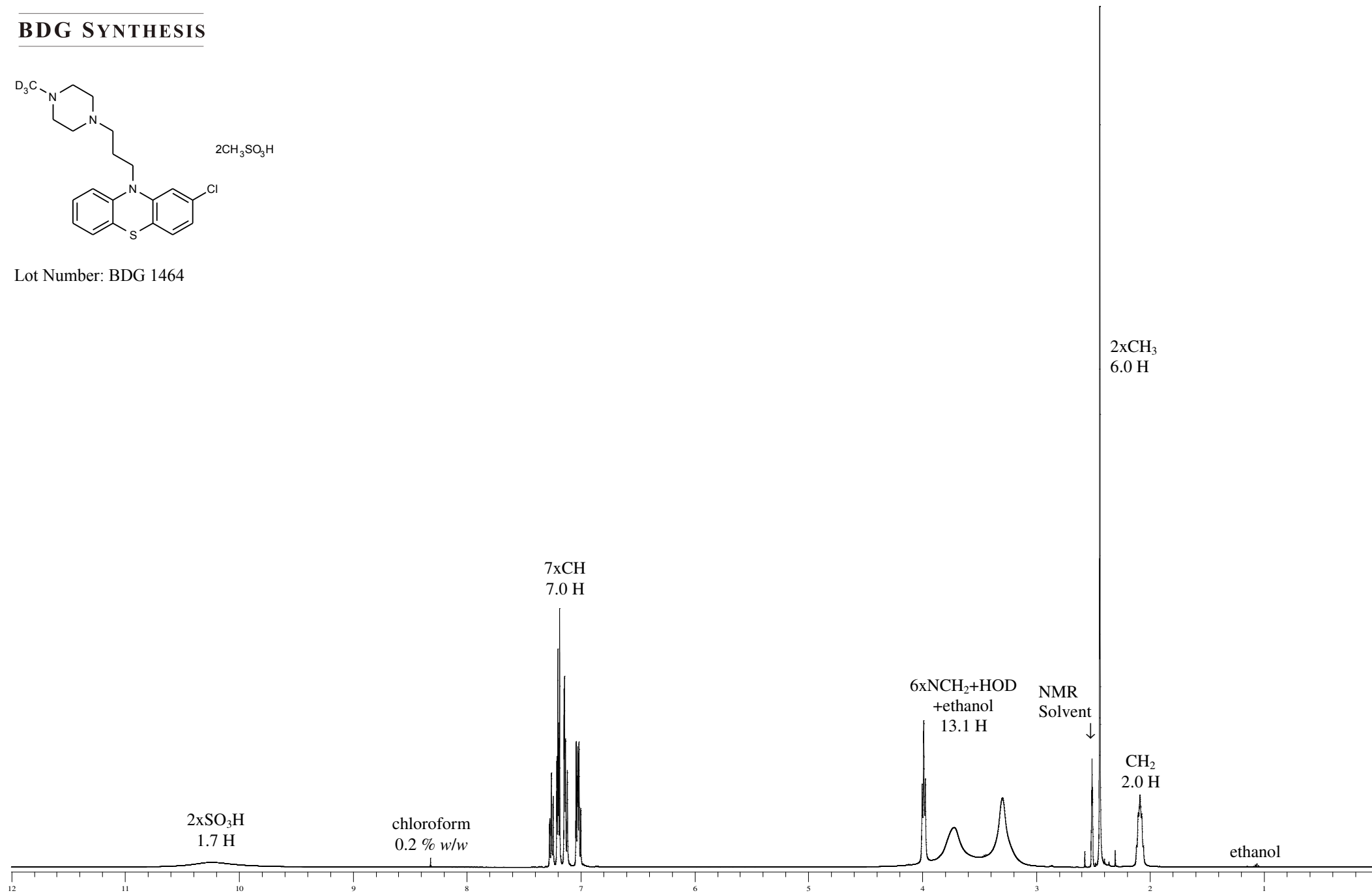


Proton NMR Spectrum of Prochlorperazine-d₃ Dimesylate in DMSO-d₆

BDG SYNTHESIS



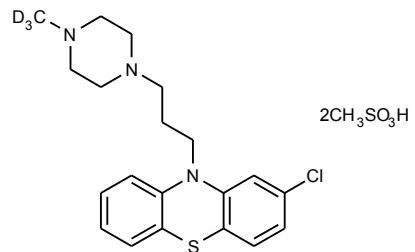
Lot Number: BDG 1464



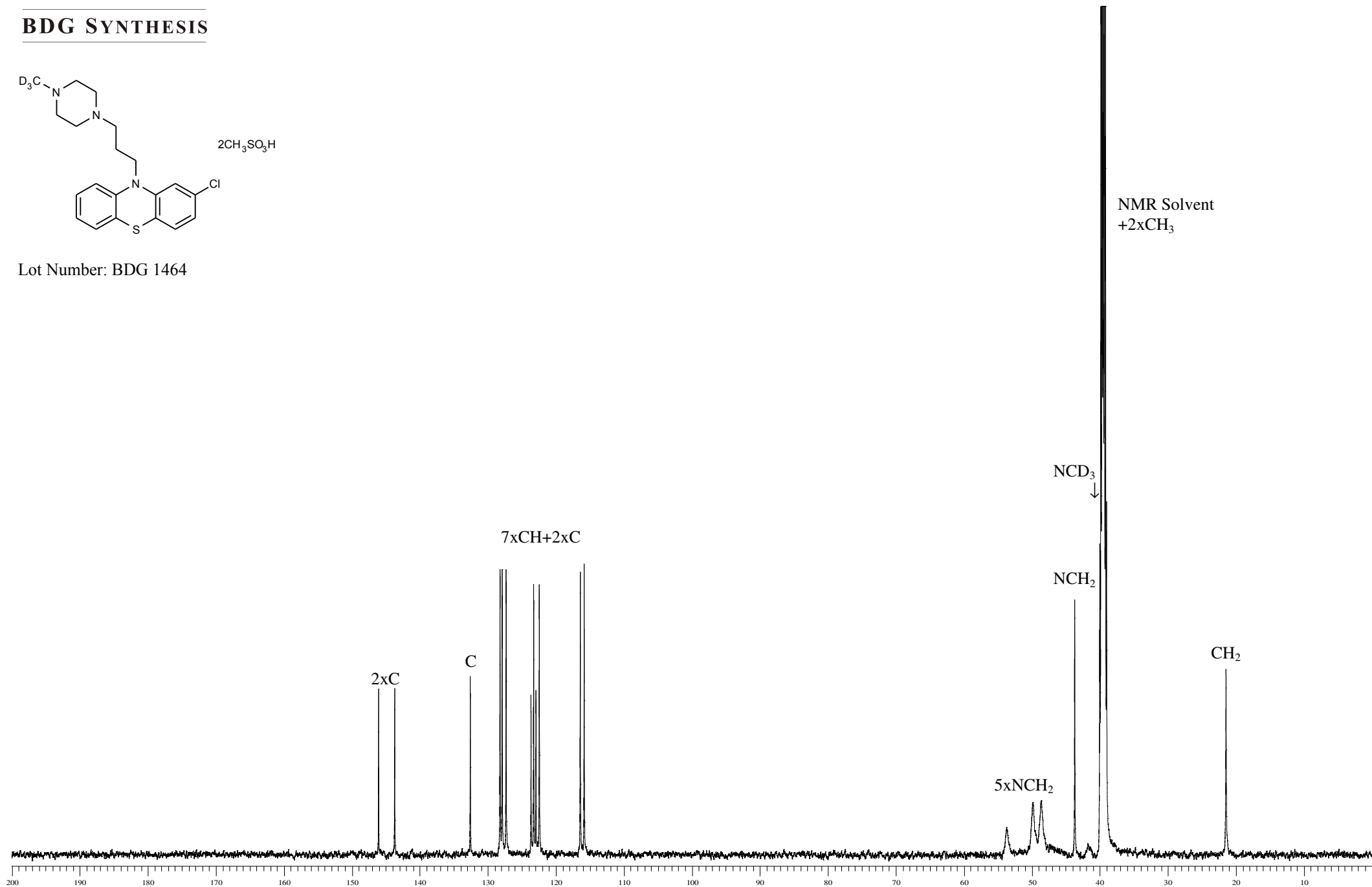


Carbon-13 NMR Spectrum of Prochlorperazine-d₃ Dimesylate in DMSO-d₆

BDG SYNTHESIS



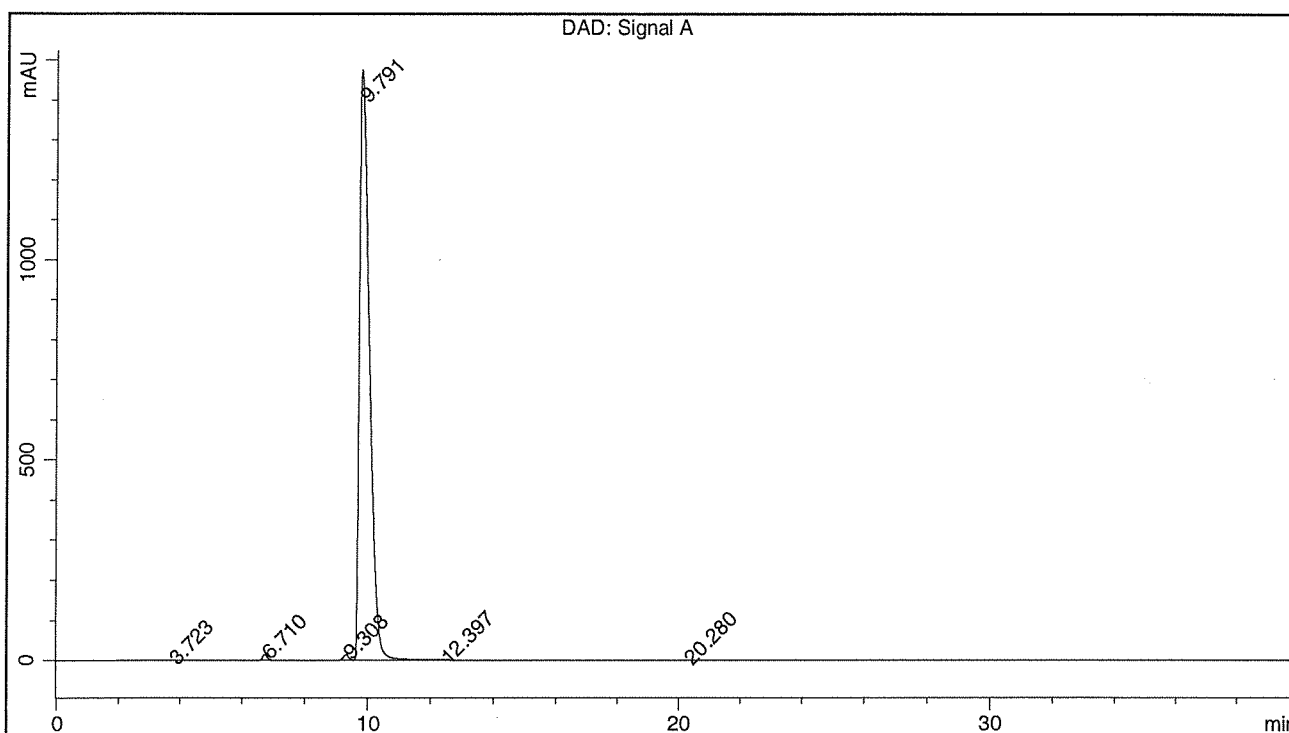
Lot Number: BDG 1464



BDG - Analysis of Prochlorperazine-d3 Dimesylate

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 39 :48 :13 IPS : Acetonitrile : Methanol
 IPS = 20 mM Heptanesulfonic Acid Sodium salt + 70 mM Acetic Acid
 Flow Rate : 1.0 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 30 C
 Injection Volume : 10 uL
 Detection : UV at 257 nm

Sample Name	BDG 1464	Instrument	AnalyticalLC01
Acquisition	15/03/2016, 13:57:05	Method (rev.)	LC10001b (6)
Sequence	BDG_15Mar2016c - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.72 min	0.5294	3.8952	0.1098 min	0.012 %
2	6.71 min	14.7232	150.7214	0.1554 min	0.459 %
3	9.31 min	14.4879	170.9473	0.1797 min	0.521 %
4	9.79 min	1475.7819	32490.7248	0.3349 min	98.945 %
5	12.40 min	0.5164	8.8395	0.2337 min	0.027 %
6	20.28 min	0.5000	11.9804	0.2935 min	0.036 %