



BDG SYNTHESIS

Certificate of Analysis

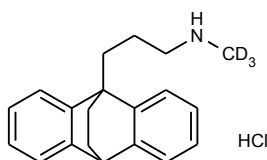
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Barry Dent

Barry R. Dent, PhD, Director
4 May 2013

Name: Maprotiline-d₃ HCl
CAS Number: 10347-81-6 (unlabelled)

Structure:



Molecular Weight: C₂₀H₂₀D₃N·HCl = 316.88

Lot Number: BDG 1495

Appearance: White, crystalline solid

Purity By HPLC: 99.7 %

Isotopic Purity: Under 0.5 % d₀

Re-test Date: 4 May 2018

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. A small singlet of an unknown impurity is observed at 1.2 ppm.

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

Residual Solvents: no residual solvents 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 (ESI+)

Found m/z 281.2095. $C_{20}H_{21}D_3N$ $[M+H]^+$ requires m/z 281.2097. The deviation of 0.7 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 (99.7 %). 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 75.61, H 6.40, D 1.83, N 4.49 %
$C_{20}H_{20}D_3N \cdot HCl$	Requires:	C 75.81, H 6.68, D 1.91, N 4.42 %

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).

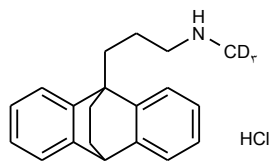
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

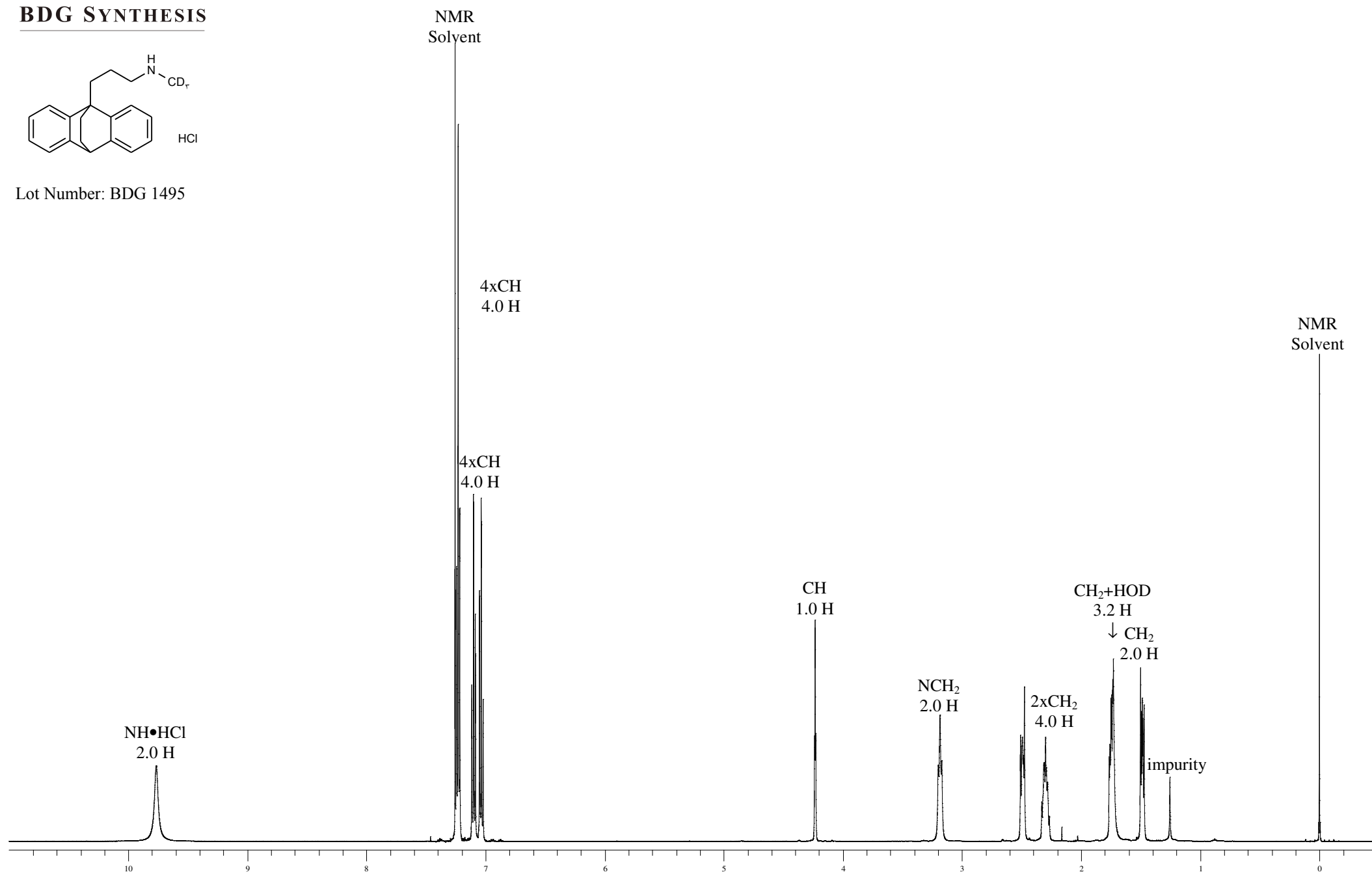


Proton NMR Spectrum of Maprotiline-d₃ HCl in CDCl₃

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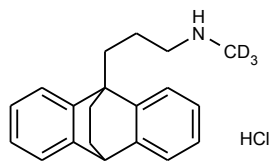
Lot Number: BDG 1495



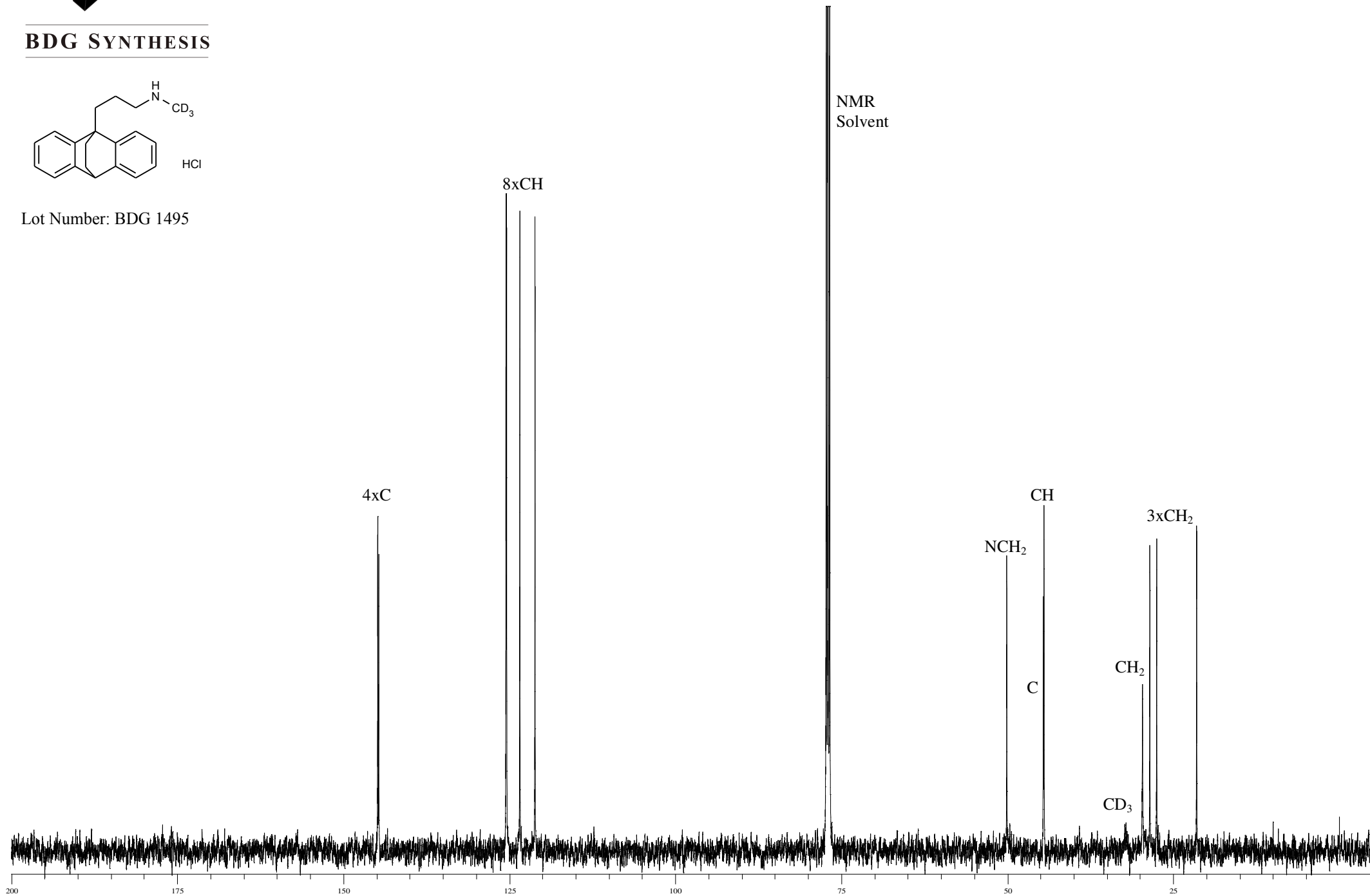


Carbon-13 NMR Spectrum of Maprotiline-d₃ HCl in CDCl₃

BDG SYNTHESIS



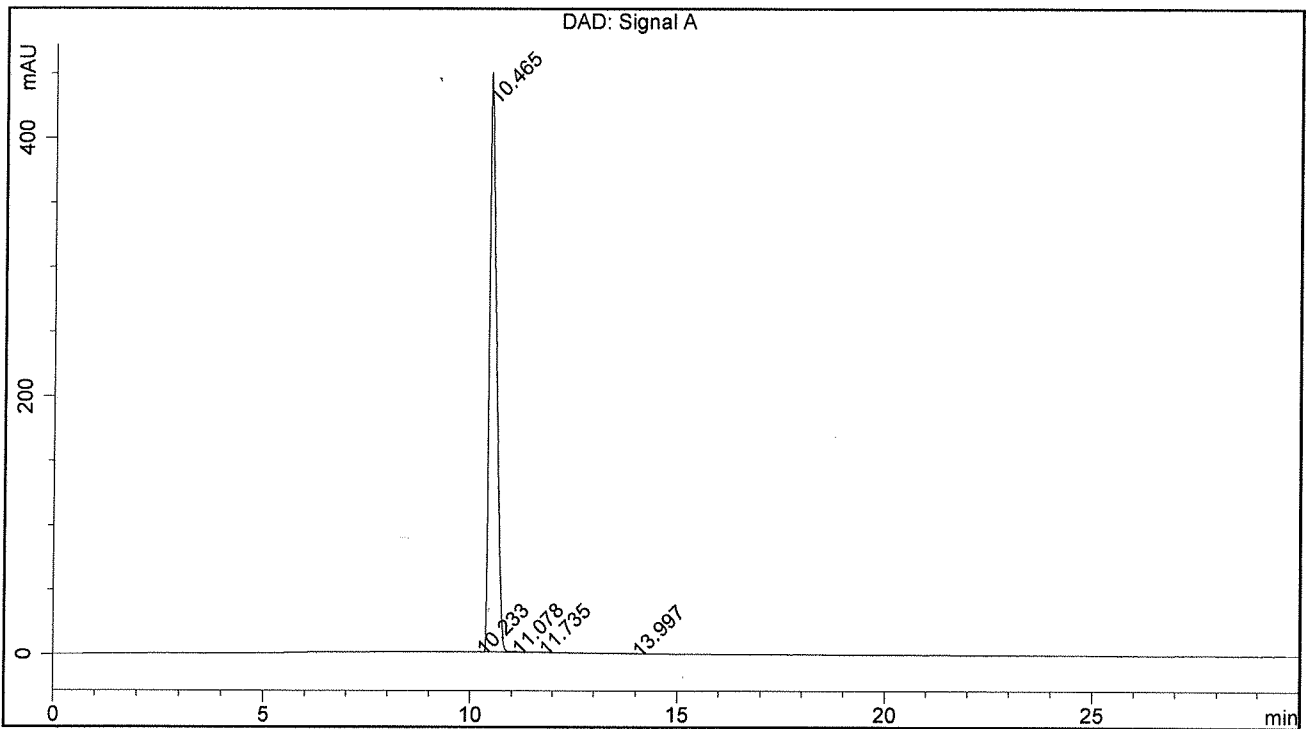
Lot Number: BDG 1495



BDG - Analysis of Maprotiline-d3 HCl

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase A : Water + 0.05% Trifluoroacetic acid
 Mobile Phase B : Acetonitrile + 0.05% Trifluoroacetic Acid
 Gradient (A:B) : T0=70:30, T30=10:90, T35=70:30, T40=70:30
 Flow Rate : 1.0 mL/min
 Sample Solvent : 70:30 Water : Acetonitrile
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 272 nm

Sample Name	BDG 1495	Instrument	AnalyticalLC01
Acquisition	04/05/2013, 14:32:26	Method (rev.)	LC10205a (9)
Sequence	BDG_04May2013a - Reprocessed	Vial Position	43
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	10.23 min	0.3906	2.1712	0.0868 min	0.041 %
2	10.46 min	449.0783	5271.9318	0.1670 min	99.723 %
3	11.08 min	0.6700	8.0894	0.1669 min	0.153 %
4	11.74 min	0.2735	2.0521	0.1115 min	0.039 %
5	14.00 min	0.3069	2.3492	0.1153 min	0.044 %