



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.

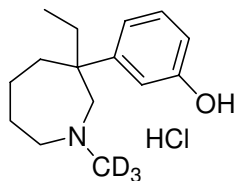
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

Barry Dent

Barry R. Dent, PhD, Director.
13 September 2006

Name: Meptazinol-d₃ HCl
CAS Number: 59263-76-2 (unlabelled)

Structure:



Molecular Weight: C₁₅H₂₀D₃NO•HCl = 272.84

Lot Number: BDG 6562.2

Appearance: White, crystalline powder

Purity by HPLC: 99.9 %

Isotopic Purity: Under 0.5 % d₀

Expiry Date: 13 September 2011
This expiry date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage stability studies because of the small amount of material available.

Storage and Handling:

Temperature: ambient laboratory temperature; may be refrigerated.

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:

Source of Material

The material was made by an unambiguous synthetic route, using literature procedures where possible; the starting material was client-supplied (Meptazinol HCl, Batch No. X-001) 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. The complexity of the spectrum indicates two rotamers of the product are present in solution.

Isotopic labelling: signals at the site of deuteration are greatly diminished, compared with the spectrum of 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.

All signals are duplicated indicating that two rotamers of the product are present in solution

Isotopic labelling: signals at the site of deuteration have collapsed to small multiplets compared with the spectrum of unlabelled material, indicating clean deuteration.

High-resolution mass spectrum (ESI+): found m/z 237.2035. $C_{15}H_{21}D_3NO$ $[M+H]^+$ requires m/z 237.2041. The deviation of 2.5 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 somewhat broadened, slightly tailing peak is observed (99.9 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.

Elemental Analysis: Found: C 66.19, H 7.61, D 2.18, N 5.23 %

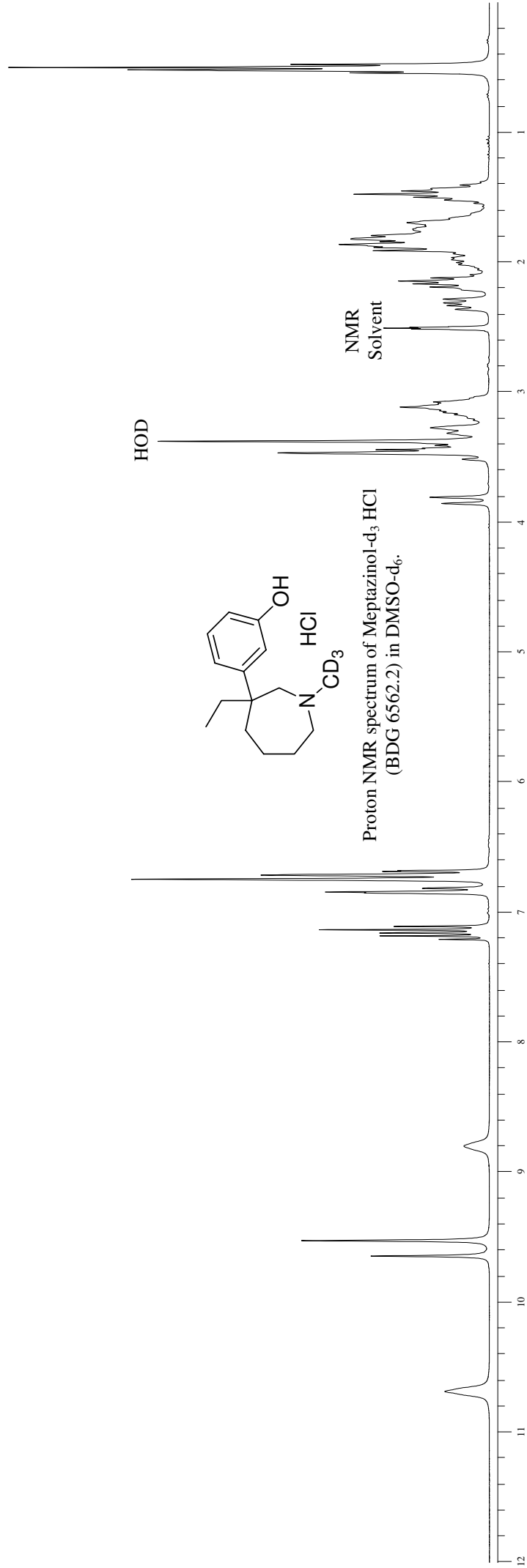
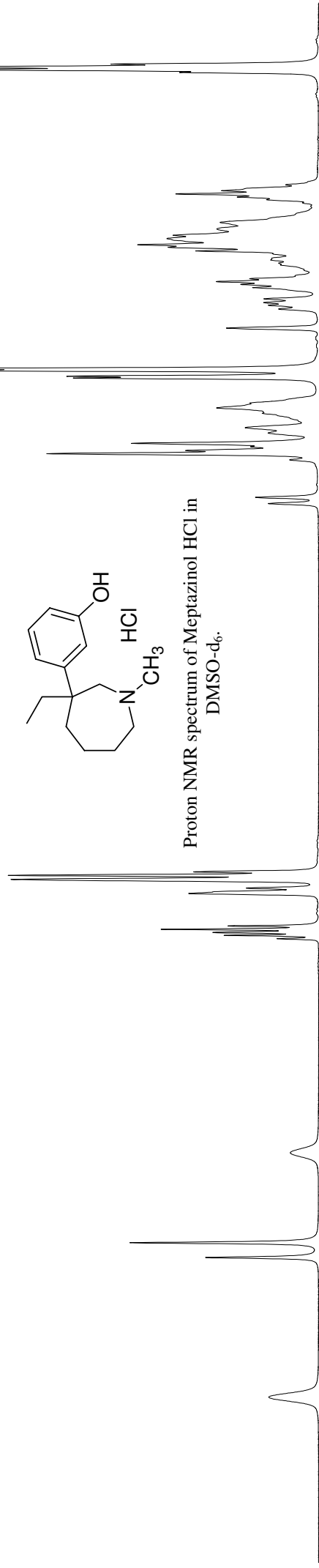
$C_{15}H_{20}D_3NO \cdot HCl$ requires: C 66.03, H 7.76, D 2.22, N 5.13 %

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).

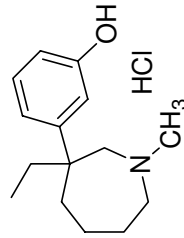


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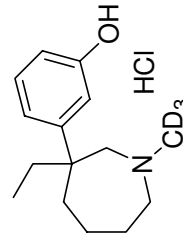
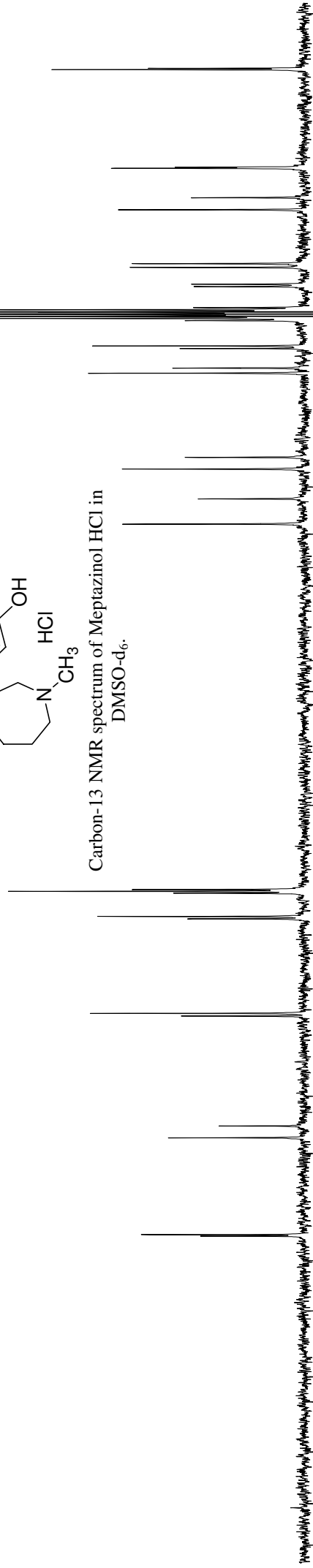




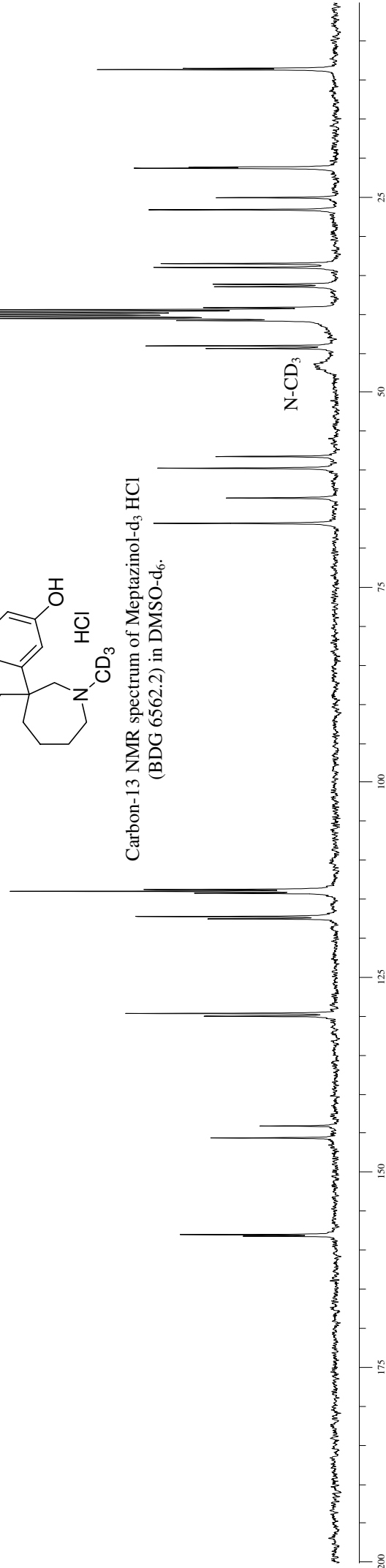
NMR
Solvent



Carbon-13 NMR spectrum of Meptazino-1-ol HCl in DMSO-d₆.



Carbon-13 NMR spectrum of Meptazino-1-ol HCl (BDG 6562.2) in DMSO-d₆.

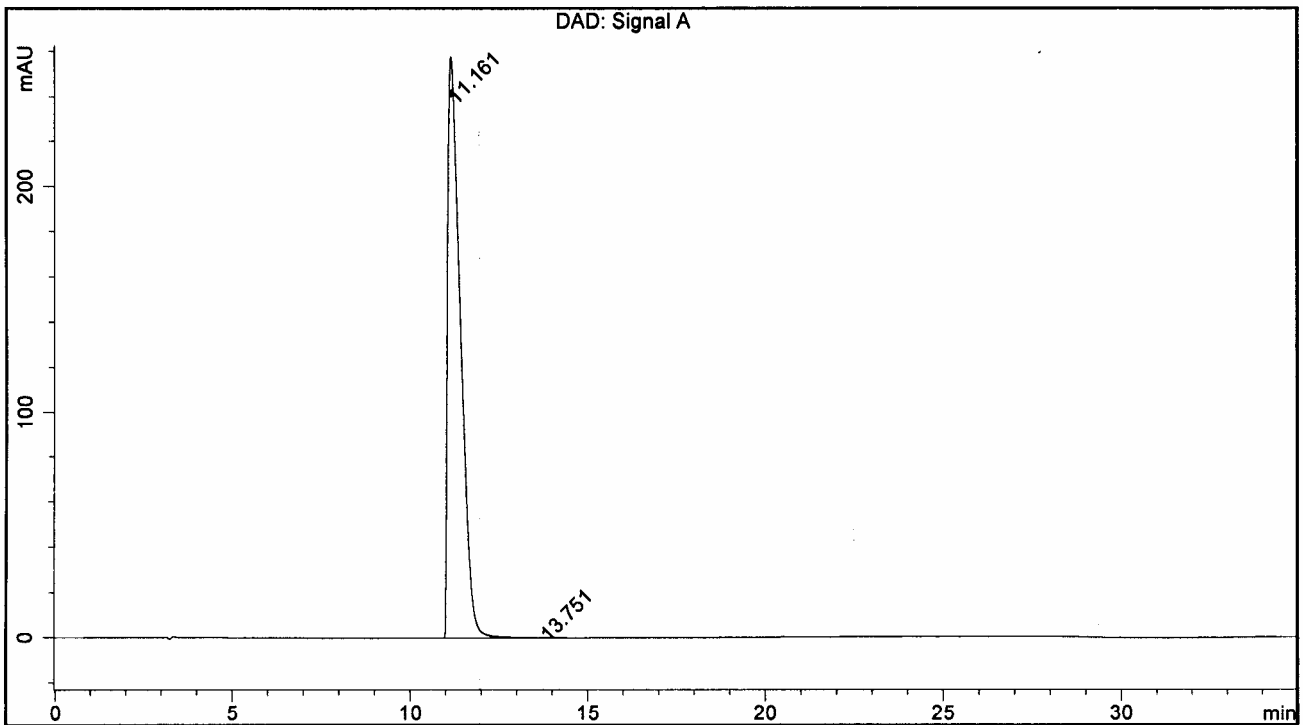


Solvation Analytical Report

BDG - Analysis of Meptazinol-d3 hydrochloride

Column : Phenomenex Luna C18 (2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase A : 10 mM Ammonium Acetate
 Mobile Phase B : Acetonitrile
 Gradient : T0=80:20, T20=20:80, T25=20:80, T30=80:20, T35=80:20
 Flow Rate : 1 mL/min
 Sample Solvent : 1:1 Acetonitrile:Water
 Column Temperature : 35C
 Injection Volume : 10 uL
 Detection : UV at 275 nm

Sample Name	BDG 6562.2	Instrument	AnalyticalLC01
Acquisition	11/09/2006, 16:48:52	Method (rev.)	LC10087a
Sequence	BDG_11Sep2006b - Reprocessed	Vial Position	1
Operator	LC10087a	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	11.16 min	257.5276	6494.6978	0.3862 min	99.955 %
2	13.75 min	0.1554	2.9003	0.2457 min	0.045 %