



BDG SYNTHESIS

Certificate of Analysis

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

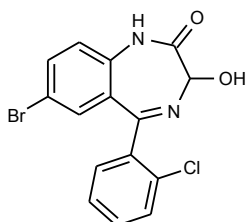
Barry Dent

Barry R. Dent, PhD, Director
9 July 2011

Name: 3-Hydroxyphenazepam

CAS Number: 70030-11-4

Structure:



Molecular Weight: $C_{15}H_{10}BrClN_2O_2 = 365.61$

Lot Number: BDG 11283.2

Appearance: White powder

Corrected Purity: 97.2 % (HPLC) - 0.3 % (ethanol) - 0.8 % (ethyl acetate) = 96.1 %

Re-test Date: 9 July 2012

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.
Residual Solvents: small amounts of ethanol (0.25 % w/w) and ethyl acetate (0.84 % w/w) 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.

High-resolution Mass Spectrum (TOF MS ES+)

Found m/z 386.9514. $C_{15}H_{10}^{79}Br^{35}ClN_2NaO_2 [M+Na]^+$ requires m/z 386.9512. The deviation of 0.5 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (97.2 %). 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

$C_{15}H_{10}BrClN_2O_2$	Found:	C 49.67, H 2.61, N 7.66 %
	Requires:	C 49.28, H 2.76, N 7.66 %

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).

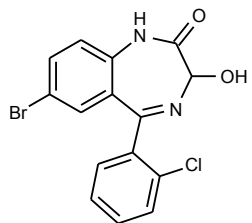
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

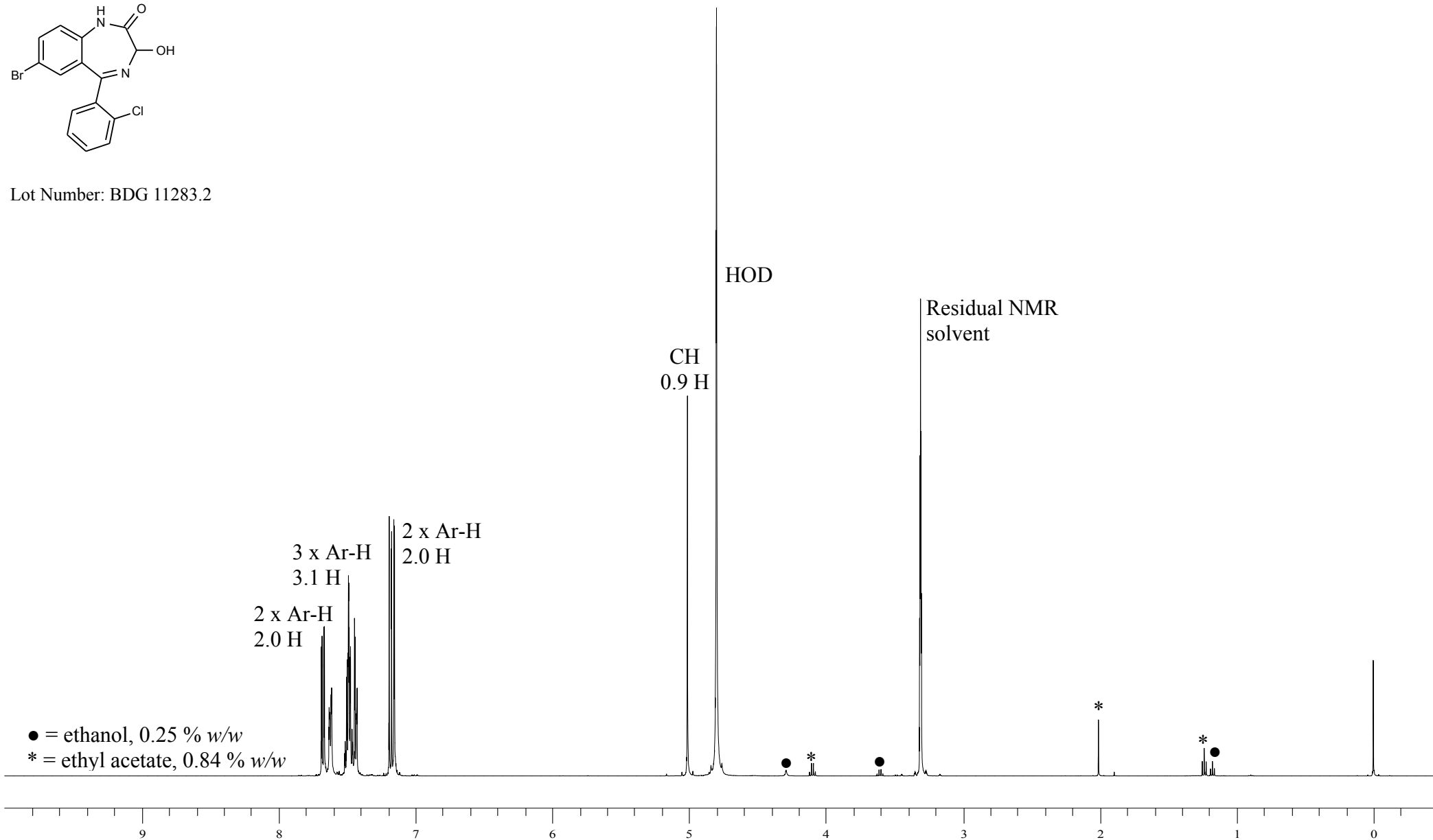


Proton NMR Spectrum of 3-Hydroxyphenazepam in Methanol-d₄

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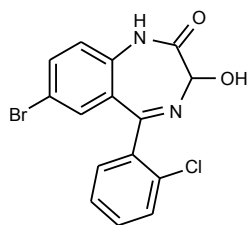
Lot Number: BDG 11283.2



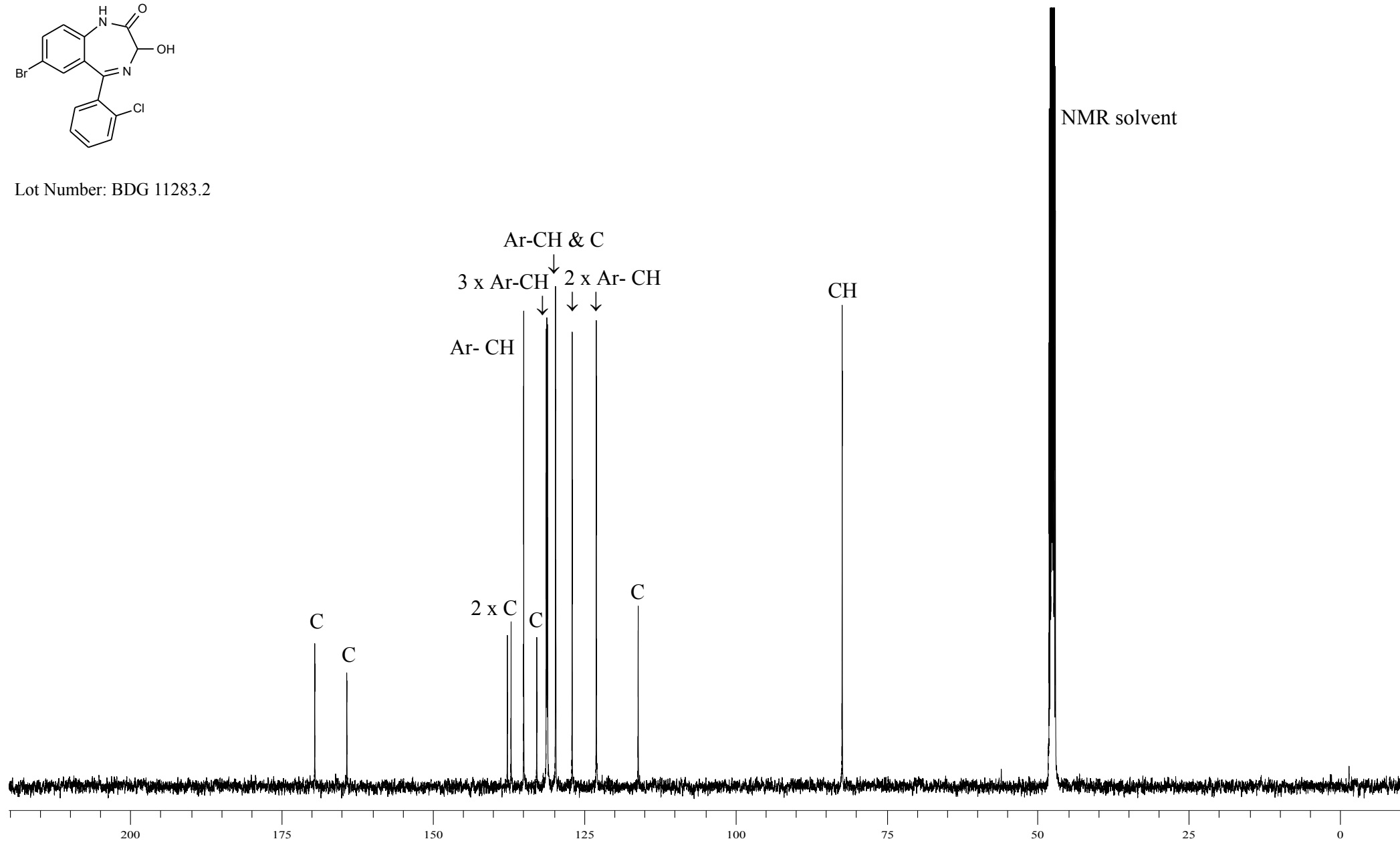


Carbon-13 NMR Spectrum of 3-Hydroxyphenazepam in Methanol-d₄

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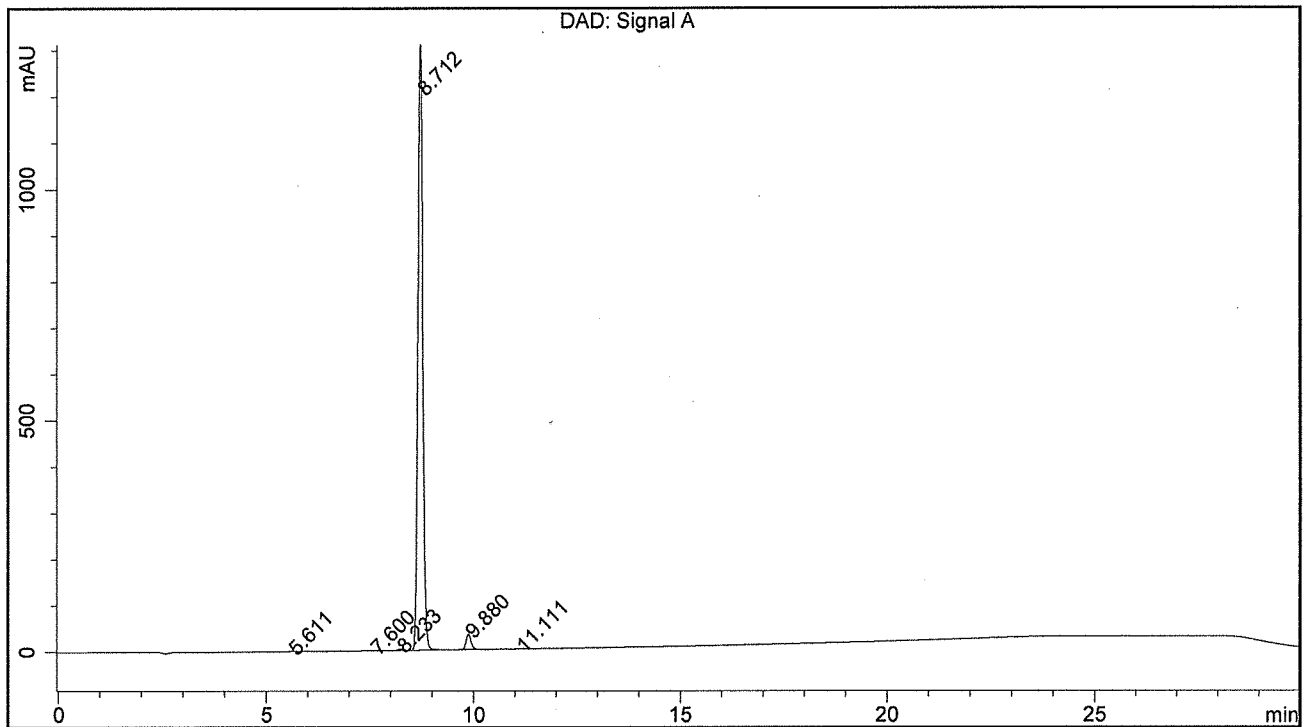
Lot Number: BDG 11283.2



BDG - Analysis of 3-Hydroxyphenazepam

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase A: 60:40:0.1 Water : Acetonitrile : Trifluoroacetic Acid
 Mobile Phase B: 30:70:0.1 Water : Acetonitrile : Trifluoroacetic Acid
 Gradient (A:B) : T0=100:0, T20=0:100, T25=0:100, T28=100:0
 Flow Rate : 1.0 mL/min Sample Solvent : 1:1 Water : Acetonitrile
 Column Temperature : 20C Injection Volume : 10 uL Detection : UV at 235 nm

Sample Name	BDG 11283.2	Instrument	AnalyticalLC01
Acquisition	09/07/2011, 13:44:02	Method (rev.)	LC10448a (7)
Sequence	BDG_09Jul2011d - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	5.61 min	0.2374	1.5494	0.1001 min	0.014 %
2	7.60 min	1.0833	7.9448	0.1116 min	0.073 %
3	8.23 min	2.9623	22.4337	0.1164 min	0.206 %
4	8.71 min	1337.1160	10604.7453	0.1225 min	97.227 %
5	9.88 min	32.3398	268.4621	0.1288 min	2.461 %
6	11.11 min	0.2178	2.0864	0.1417 min	0.019 %