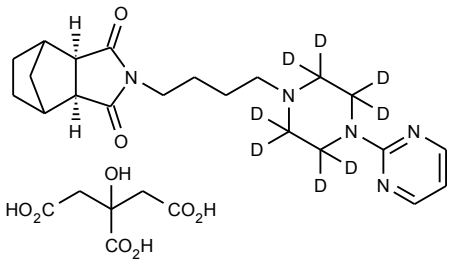
BDG SYNTHESIS

Certificate of Analysis

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

Barry Dent

Barry R. Dent, PhD, Director
21 June 2007

Name:	**Tandospirone-d_8 Citrate**
CAS Number:	112457-95-1 (unlabelled)
Structure:	
Molecular Weight:	$C_{21}H_{21}D_8N_5O_2.C_6H_8O_7$ = 583.66
Lot Number:	BDG 6867.3-02
Appearance:	White, crystalline solid
Corrected Purity:	99.5 % (HPLC) - 0.2 % (diethyl ether) - 0.3 % (2-propanol) = 99.0 %
Isotopic Purity:	Under 0.5 % d_0
Re-test Date:	21 June 2012
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.

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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 sites of deuteration are absent, compared with what would be expected for unlabelled material, indicating clean deuteration.
Residual Solvents: small amounts of diethyl ether (0.2 % w/w) and 2-propanol (0.3 % 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.
Isotopic Labelling: signals at the sites 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* 392.2908. $C_{21}H_{22}D_8N_5O_2$ $[M+H]^+$ requires *m/z* 392.2896. The deviation of 3.0 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 (99.5 %). 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 55.76, H 5.04, D 2.78, N 12.09 %
$C_{21}H_{21}D_8N_5O_2.C_6H_8O_7$	Requires:	C 55.56, H 5.01, D 2.76, N 12.00 %

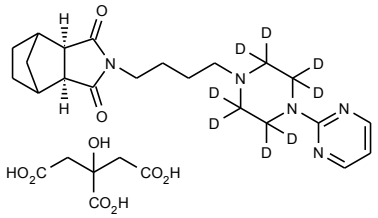
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

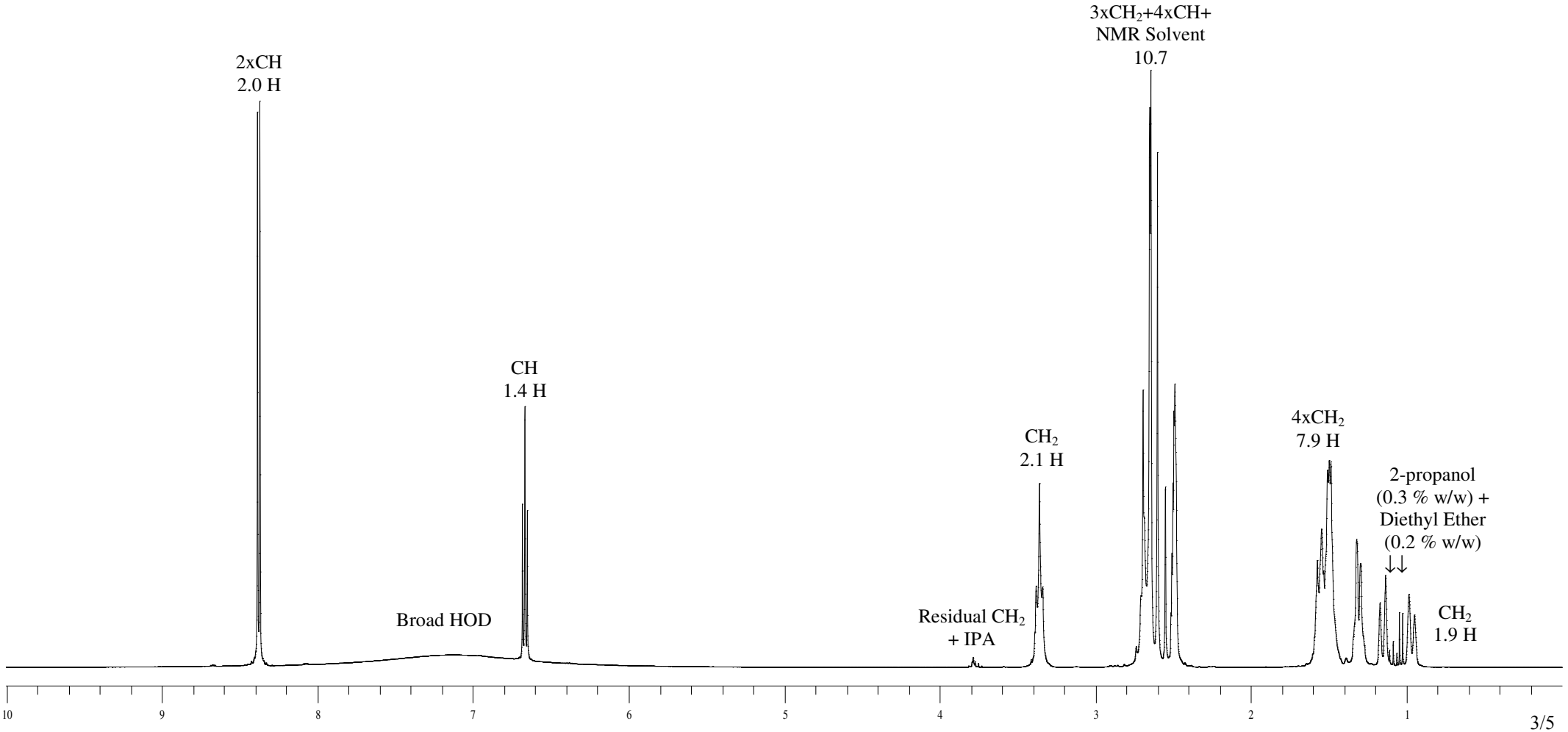
The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

BDG SYNTHESIS

Proton NMR Spectrum of Tandospirone-d_8 Citrate in DMSO-d_6

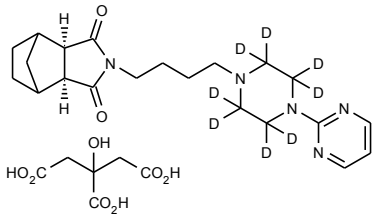


Lot Number: BDG 6867.3



BDG SYNTHESIS

Carbon-13 NMR Spectrum of Tandospirone-d_8 Citrate in DMSO-d_6

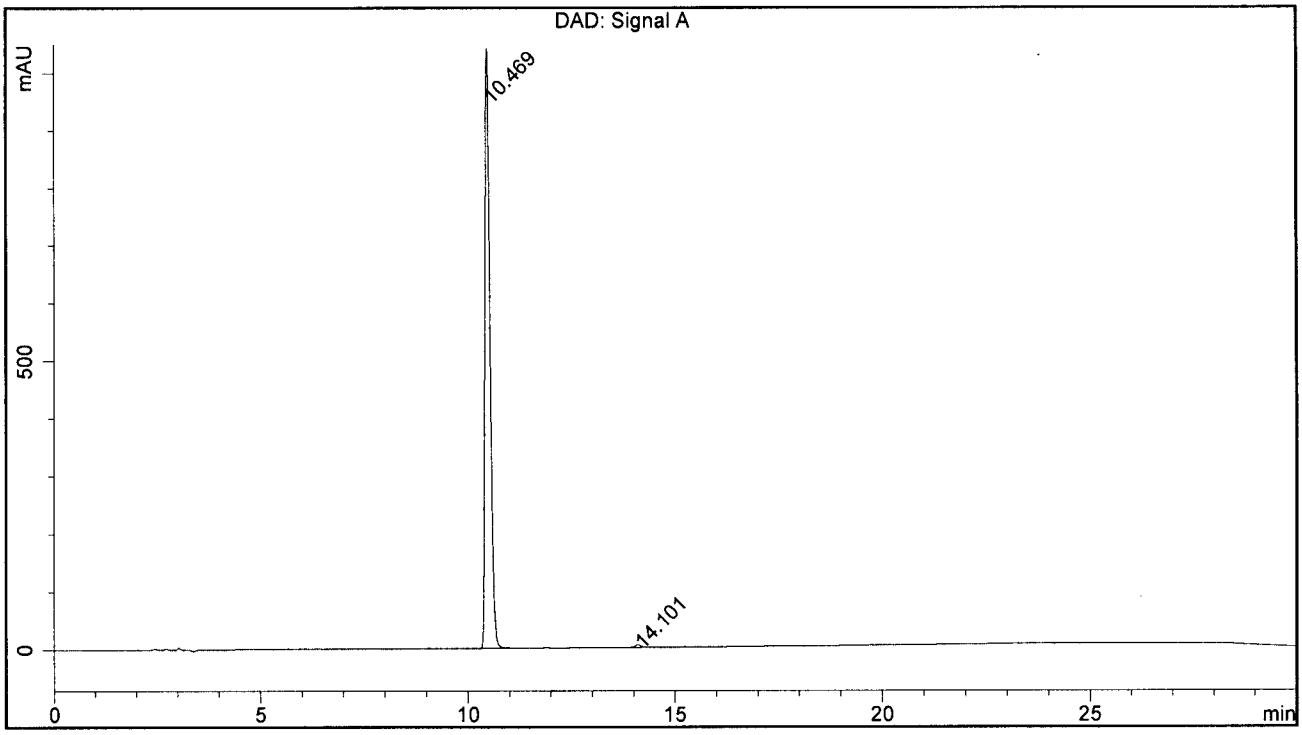


Lot Number: BDG 6867.3

BDG - Analysis of Tandospirone-d8 Citrate

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 + 0.1% Trifluoroacetic Acid
Mobile Phase B: Acetonitrile + 0.1% Trifluoroacetic Acid
Gradient (A:B) : T0=80:20, T20=50:50, T25=50:50, T30=80:20, T35=80:20
Flow Rate : 1.0 mL/min
Sample Solvent : 1:1 A:B
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 238 nm

Sample Name	BDG 6867.3	**Instrument**	AnalyticalLC01
Acquisition	19/06/2007, 19:29:55	**Method (rev.)**	LC10171a (7)
Sequence	BDG_19Jun2007d	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	10.47 min	1040.5188	8903.0941	0.1318 min	99.520 %
2	14.10 min	4.8678	42.9428	0.1329 min	0.480 %