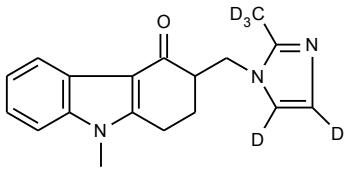
BDG SYNTHESIS

Certificate of Analysis

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

Barry Dent

Barry R. Dent, PhD, Director
5 November 2012

Name:	**Ondansetron-d_5**
CAS Number:	99614-02-5 (unlabelled)
Structure:	
Molecular Weight:	$C_{18}H_{14}D_5N_3O$ = 298.39
Lot Number:	BDG 12620
Appearance:	White, crystalline solid
Corrected Purity:	99.8 % (HPLC) - 2.4 % (water) = 97.4 %
Isotopic Purity:	Under 0.5 % d_0
Re-test Date:	5 November 2017

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.

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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 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.
Isotopic Labelling: signals at the sites 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* 299.1918. $C_{18}H_{15}D_5N_3O$ $[M+H]^+$ requires *m/z* 299.1920. 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 somewhat broadened, slightly tailing peak is observed (99.8 %). 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 70.70, H 4.85, D 3.28, N 13.86 %
$C_{18}H_{14}D_5N_3O{\cdot}0.4H_2O$	Requires:	C 70.74, H 4.88, D 3.30, N 13.75 %, H_2O 2.36 %
$C_{18}H_{14}D_5N_3O$	Requires:	C 72.45, H 4.73, D 3.37, N 14.08 %

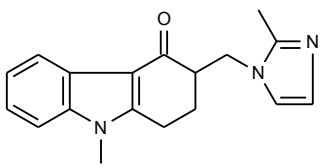
The elemental analyses fall somewhat outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the “best-fit” hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we recommend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

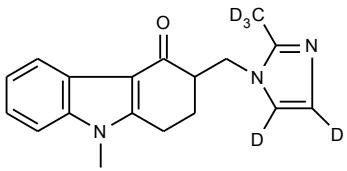
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

BDG SYNTHESIS

Proton NMR Spectrum of Ondansetron (top) and Ondansetron-d_5 (bottom) in $CDCl_3$

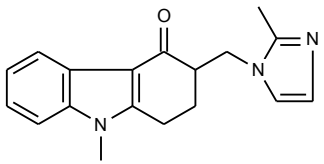


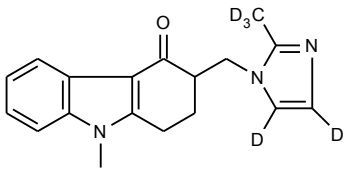


Lot Number: BDG 12620

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Ondansetron (top) and Ondansetron-d_5 (bottom) in $CDCl_3$





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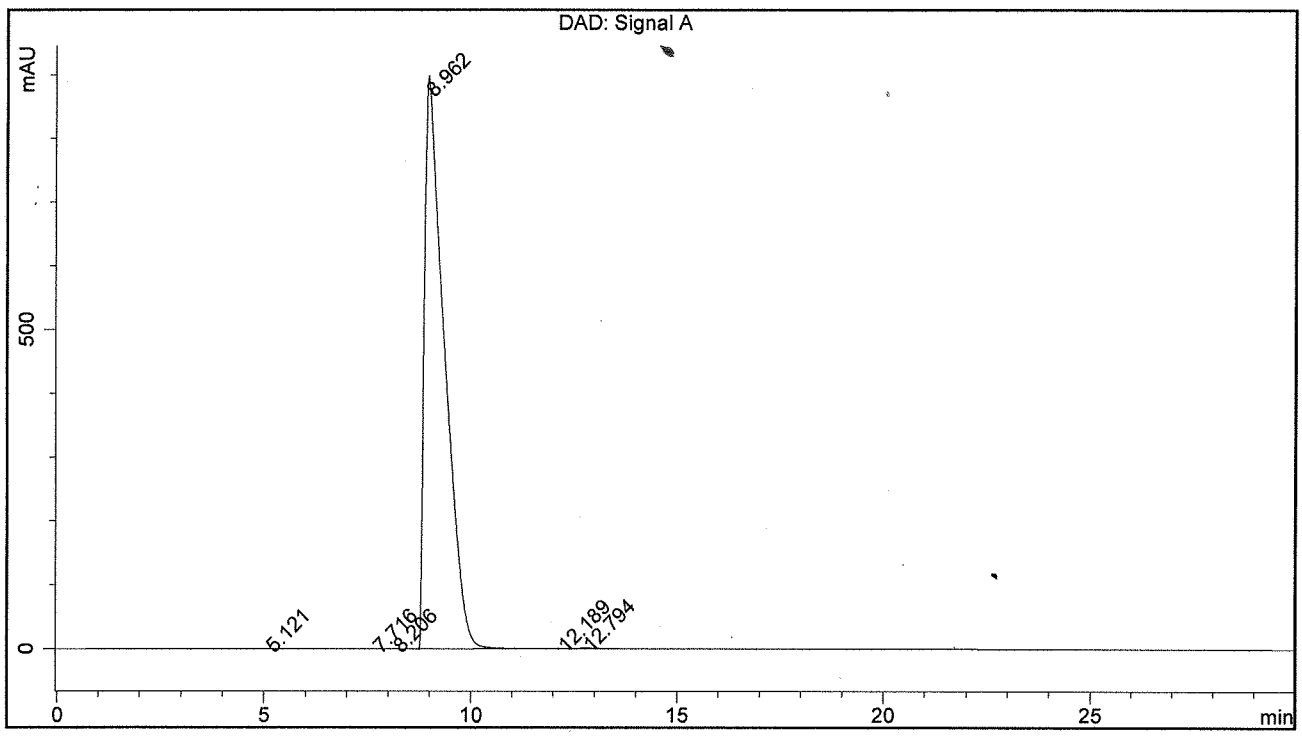
BDG - Analysis of Ondansetron-d5

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 75:25 10 mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile
Flow Rate : 1.0 mL/min
Sample Solvent : Initial Mobile Phase
Injection Volume : 10 uL
Column Temperature : 20C
Detection : UV at 248 nm

Sample Name	BDG 12620	Instrument	AnalyticalLC01
Acquisition	05/11/2012, 19:51:55	Method (rev.)	LC10441a (5)
Sequence	BDG_05Nov2012b - Reprocessed	Vial Position	2
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	5.12 min	0.2171	1.9224	0.1173 min	0.006 %
2	7.72 min	0.1503	1.5294	0.1307 min	0.005 %
3	8.21 min	0.2802	3.0392	0.1405 min	0.010 %
4	8.96 min	898.1839	30083.7786	0.4608 min	99.820 %
5	12.19 min	0.3685	5.7028	0.2005 min	0.019 %
6	12.79 min	1.5437	42.1627	0.3766 min	0.140 %