BDG SYNTHESIS

Certificate of Analysis

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

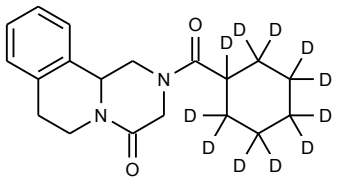
Barry Dent

Barry R. Dent, PhD, Director
22 October 2010

Name: **Praziquantel-d_{11}**

CAS Number: 55268-74-1 (unlabelled)

Structure:



Molecular Weight: $C_{19}H_{13}D_{11}N_2O_2$ = 323.47

Lot Number: BDG 11102.3

Appearance: Off-white, crystalline solid

Corrected Purity: 98.5 % (HPLC) - 0.4 % (hexanes) = 98.1 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 22 October 2015

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. Some signals are split due to the presence of conformers.
Isotopic Labelling: small signals at about 8 and 5 % of the intensities expected for unlabelled material, are observed for two of the sites of deuteration indicating that a small amount H/D exchange has occurred.
Residual Solvents: a small amount of hexanes (0.4 % *w/w*) and a trace (under 0.1 % *w/w*) of ethyl acetate are observed.
Impurities: traces of unidentified impurities are seen in the baseline.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available. Some signals are split due to the presence of conformers.
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 (TOF MS ES+)

Found *m/z* 346.2437. $C_{19}H_{13}D_{11}N_2NaO_2$ $[M+Na]^+$ requires *m/z* 346.2426. The deviation of 3.2 ppm is within normally accepted limits for the establishment of identity by HRMS. Signals for M-1, M-2 and M-3 species are also observed, but any M-11 peak is at or below the background level of 0.5 % (detection limit). We conclude that the material comprises substantially d_{11} material with lesser amounts of d_{10}, d_9 and d_8 species present because of a small amount of H/D exchange on the cyclohexyl group.

HPLC

A sharp, symmetrical peak is observed (98.5 %). 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 70.59, H 4.14, D 7.01, N 8.50 %
$C_{19}H_{13}D_{11}N_2O_2$	Requires:	C 70.55, H 4.05, D 6.85, N 8.66 %

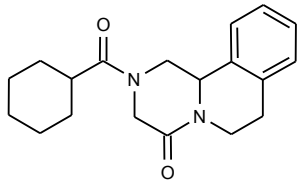
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

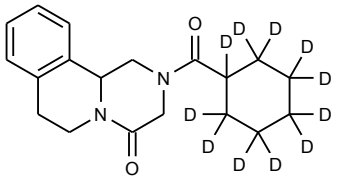
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

BDG SYNTHESIS

Proton NMR Spectrum of Praziquantel (top) and Praziquantel-d_{11} (bottom) in Methanol-d_4

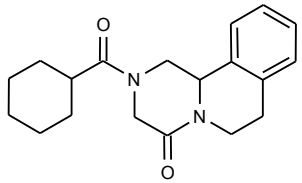


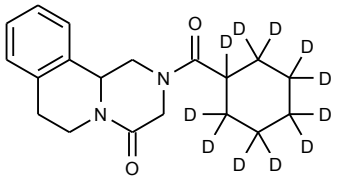


Lot Number: BDG 11102.3

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Praziquantel (top) and Praziquantel-d_{11} (bottom) in Methanol-d_4



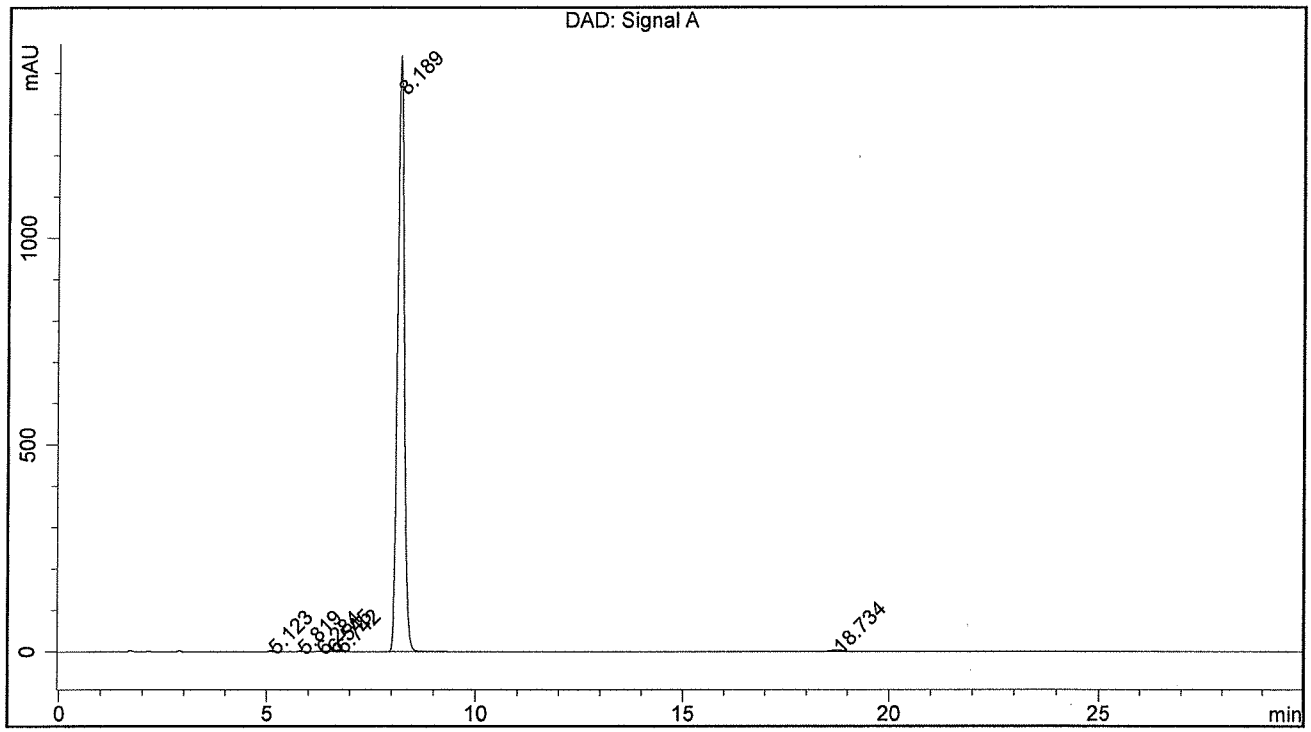


Lot Number: BDG 11102.3

BDG - Analysis of Praziquantel-d11

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 50:50 10 mM diPotassium Hydrogen Phosphate pH=7.0 : Acetonitrile
Flow Rate : 1.0 mL/min
Sample Solvent : Mobile phase
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 210nm

Sample Name	BDG 11102.3	Instrument	AnalyticalLC01
Acquisition	22/10/2010, 11:03:05	Method (rev.)	LC10404a (3)
Sequence	BDG_22Oct2010b	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	5.12 min	3.2508	32.8103	0.1618 min	0.203 %
2	5.82 min	2.6567	20.6768	0.1188 min	0.128 %
3	6.28 min	1.1633	9.9025	0.1453 min	0.061 %
4	6.55 min	4.0159	30.6883	0.1192 min	0.190 %
5	6.74 min	4.5188	39.9099	0.1330 min	0.247 %
6	8.19 min	1441.9156	15942.8343	0.1730 min	98.527 %
7	18.73 min	4.3222	104.3348	0.3656 min	0.645 %