BDG SYNTHESIS

Certificate of Analysis

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

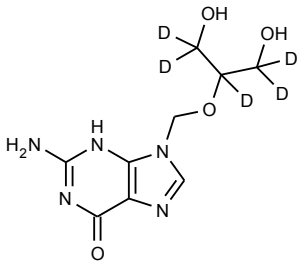
Barry Dent

Barry R. Dent, PhD, Director
7 March 2012

Name: **Ganciclovir-d_5**

CAS Number: 82410-32-0 (unlabelled)

Structure:



Molecular Weight: $C_9H_8D_5N_5O_4$ = 260.26

Lot Number: BDG 12459.5

Appearance: White, crystalline solid

Corrected Purity: 95.2 % (HPLC) - 6.5 % (water) = 88.7 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 7 March 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 absent, compared with the spectrum of unlabelled material, indicating clean deuteration.
Residual Solvents: no residual solvents 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.
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* 261.1363. $C_9H_9D_5N_5O_4$ $[M+H]^+$ requires *m/z* 261.1360. The deviation of 1.1 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 (95.2 %). 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

	Found:	C 38.56, H 3.49, D 3.49, N 24.04 %
$C_9H_8D_5N_5O_4 \cdot 1.0H_2O$	Requires:	C 38.84, H 3.62, D 3.62, N 25.17 %, H_2O 6.47 %
$C_9H_8D_5N_5O_4$	Requires:	C 41.53, H 3.10, D 3.87, N 26.91 %

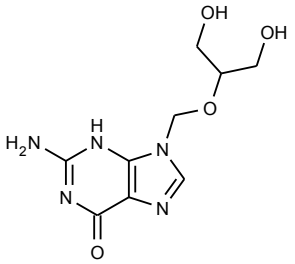
The elemental analyses fall somewhat outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the "best-fit" hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we recommend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

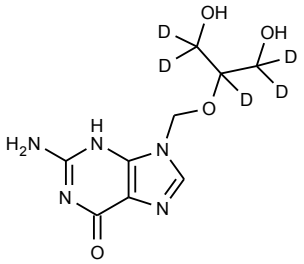
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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 Ganciclovir (top) and Ganciclovir-d_5 (bottom) in DMSO-d_6

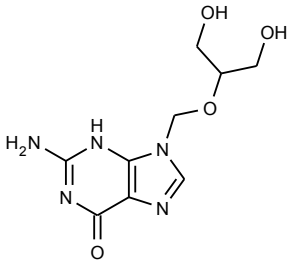


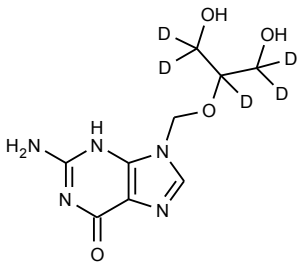


Lot Number: BDG 12459.5

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Ganciclovir (top) and Ganciclovir-d_5 (bottom) in DMSO-d_6



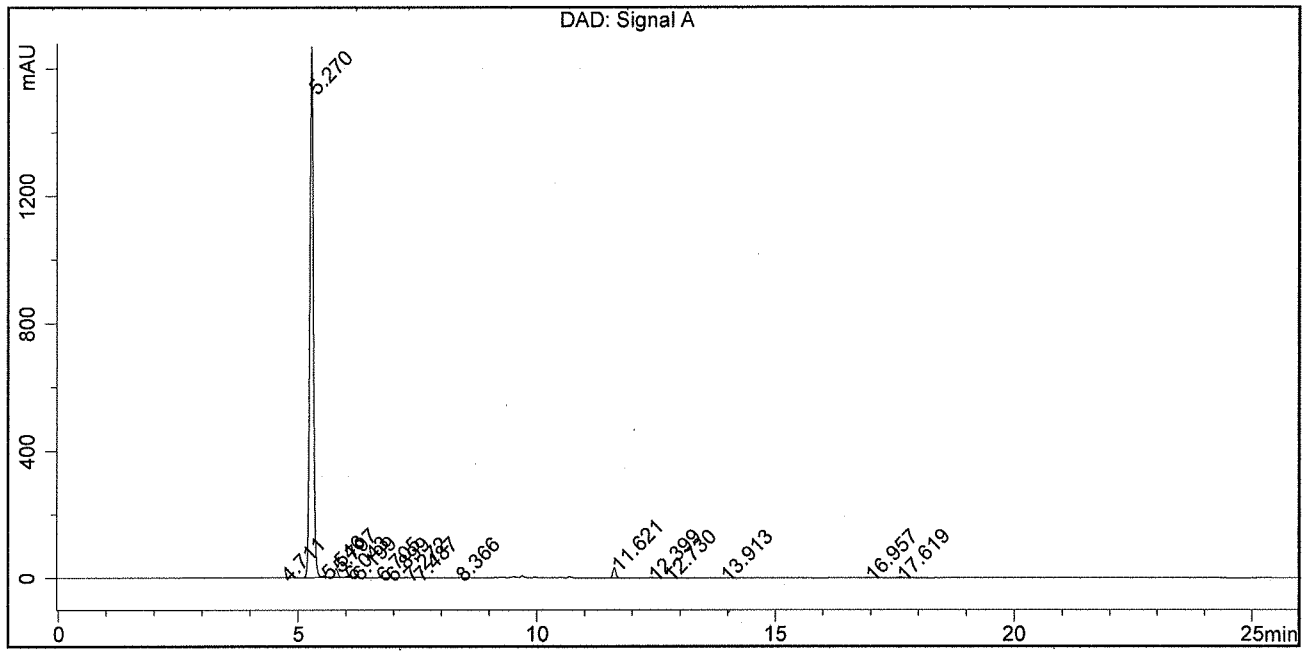


Lot Number: BDG 12459.5

BDG- Analysis of Ganciclovir-d5

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm . . . . . Guard : Security Guard C18 RP 4 x 3 mm
Mobile Phase A : 20 mM Potassium diHydrogen Phosphate + 5 mM Sodium Heptanesulphonate to pH=2.2.
Mobile Phase B : Acetonitrile . . . . . Gradient (A:B) : T0=95:5, T20=30:70, T23=95:5, T26=95:5
Flow Rate : 1.0 mL/min . . . . . Sample Solvent : Mobile Phase . . . . . Injection Volume : 10 uL
Column Temperature : 20C . . . . . Detection : UV at 254 nm

Sample Name	BDG 12459.5	Instrument	AnalyticalLC01
Acquisition	07/03/2012, 10:28:48	Method (rev.)	LC10495a (7)
Sequence	BDG_07Mar2012a - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.71 min	0.8429	6.6682	0.1203 min	0.075 %
2	5.27 min	1669.4793	8497.5044	0.0814 min	95.227 %
3	5.55 min	4.1027	17.1104	0.0667 min	0.192 %
4	5.80 min	28.2032	138.0519	0.0771 min	1.547 %
5	6.04 min	4.6255	23.4629	0.0812 min	0.263 %
6	6.20 min	3.1425	14.5588	0.0741 min	0.163 %
7	6.70 min	0.9135	4.1119	0.0705 min	0.046 %
8	6.90 min	0.8522	6.8708	0.1301 min	0.077 %
9	7.27 min	0.6913	5.6739	0.1158 min	0.064 %
10	7.49 min	1.6691	13.7698	0.1303 min	0.154 %
11	8.37 min	0.9285	18.8685	0.2891 min	0.211 %
12	11.62 min	32.3183	140.0371	0.0686 min	1.569 %
13	12.40 min	1.2262	6.2268	0.0792 min	0.070 %
14	12.73 min	0.6381	3.0255	0.0753 min	0.034 %
15	13.91 min	0.4980	2.5950	0.0808 min	0.029 %
16	16.96 min	1.1414	7.9642	0.1074 min	0.089 %
17	17.62 min	2.6965	16.9191	0.0952 min	0.190 %