



BDG SYNTHESIS

Certificate of Analysis

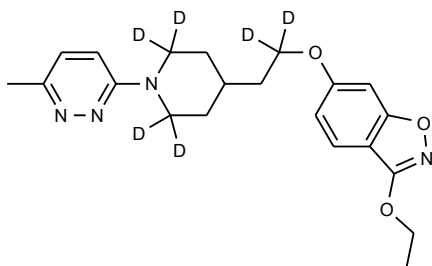
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Barry Dent

Barry R. Dent, PhD, Director
30 July 2014

Name: Vapendavir-d₆
CAS Number: 439085-51-5 (unlabelled)

Structure:



Molecular Weight: C₂₁H₂₀D₆N₄O₃ = 388.49

Lot Number: BDG 16140.3

Appearance: White, crystalline solid

Purity By HPLC: 98.5 %

Isotopic Purity: Under 0.5 % d₀

Re-test Date: 30 July 2019

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. Prone to static.

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: a trace (under 0.1 % w/w) of ethyl acetate is observed.

Impurities: a trace of an unidentified impurity is 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 what would be expected for unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found m/z 389.2458. $C_{21}H_{21}D_6N_4O_3$ $[M+H]^+$ requires m/z 389.2460. The deviation of 0.5 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, 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 65.00, H 5.18, D 3.11, N 14.50 %
$C_{21}H_{20}D_6N_4O_3$	Requires:	C 64.92, H 5.19, D 3.11, N 14.42 %

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).

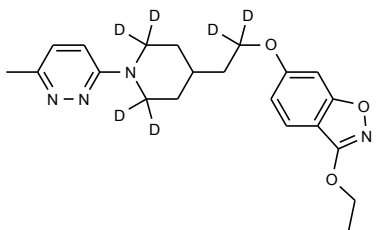
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

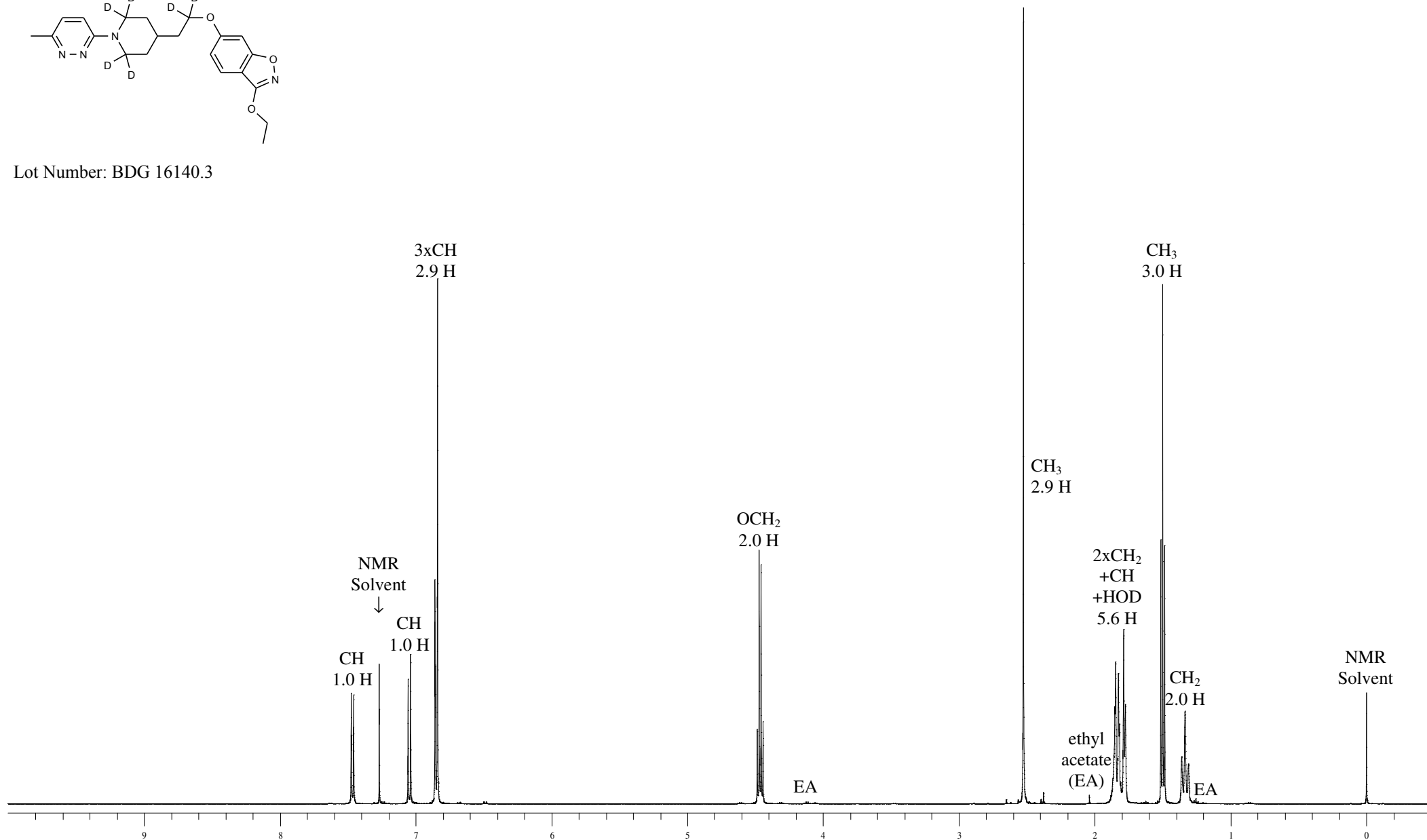


Proton NMR Spectrum of Vapendavir-d₆ in CDCl₃

BDG SYNTHESIS



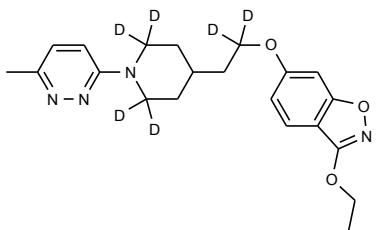
Lot Number: BDG 16140.3



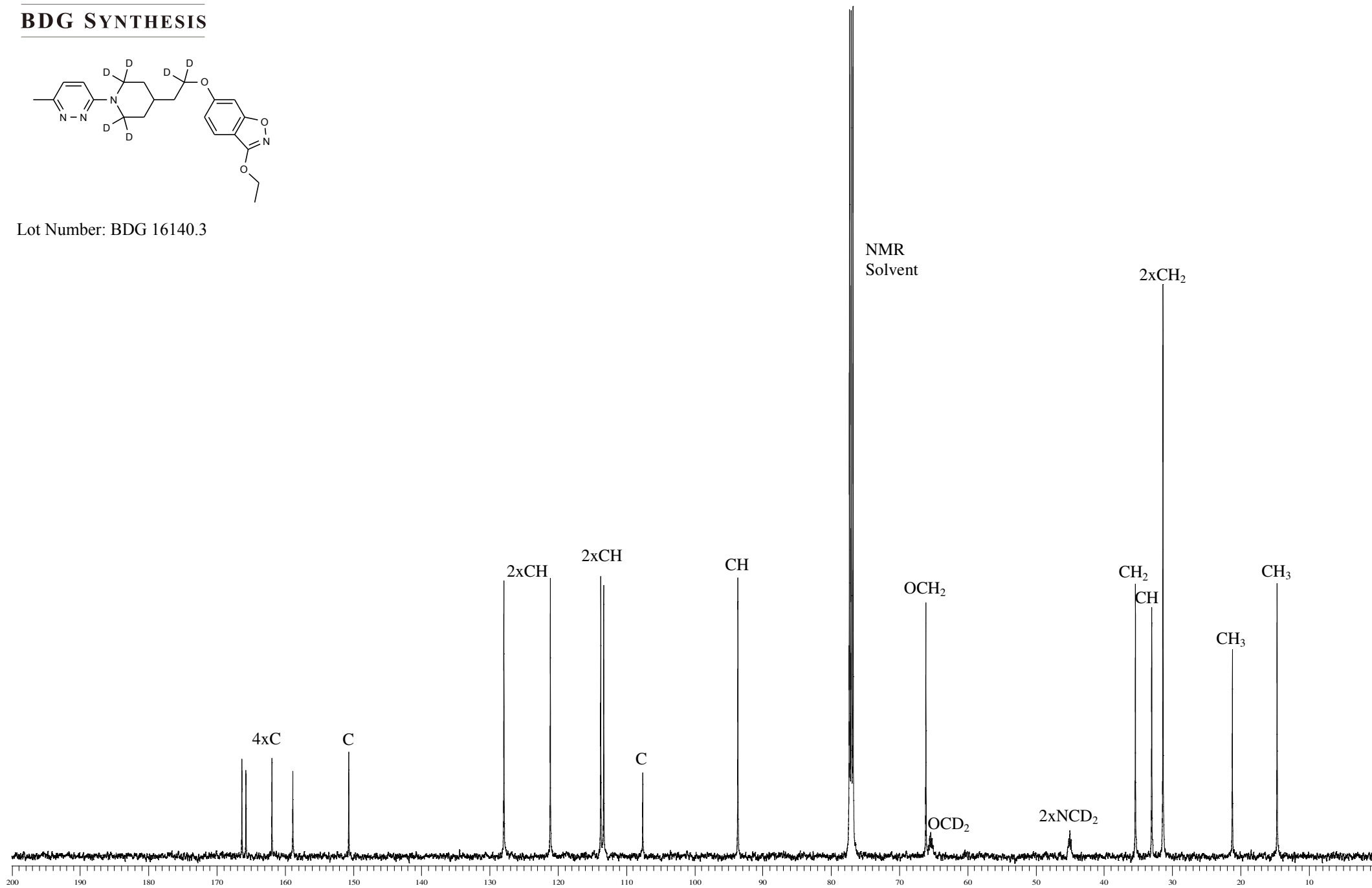


Carbon-13 NMR Spectrum of Vapendavir-d₆ in CDCl₃

BDG SYNTHESIS



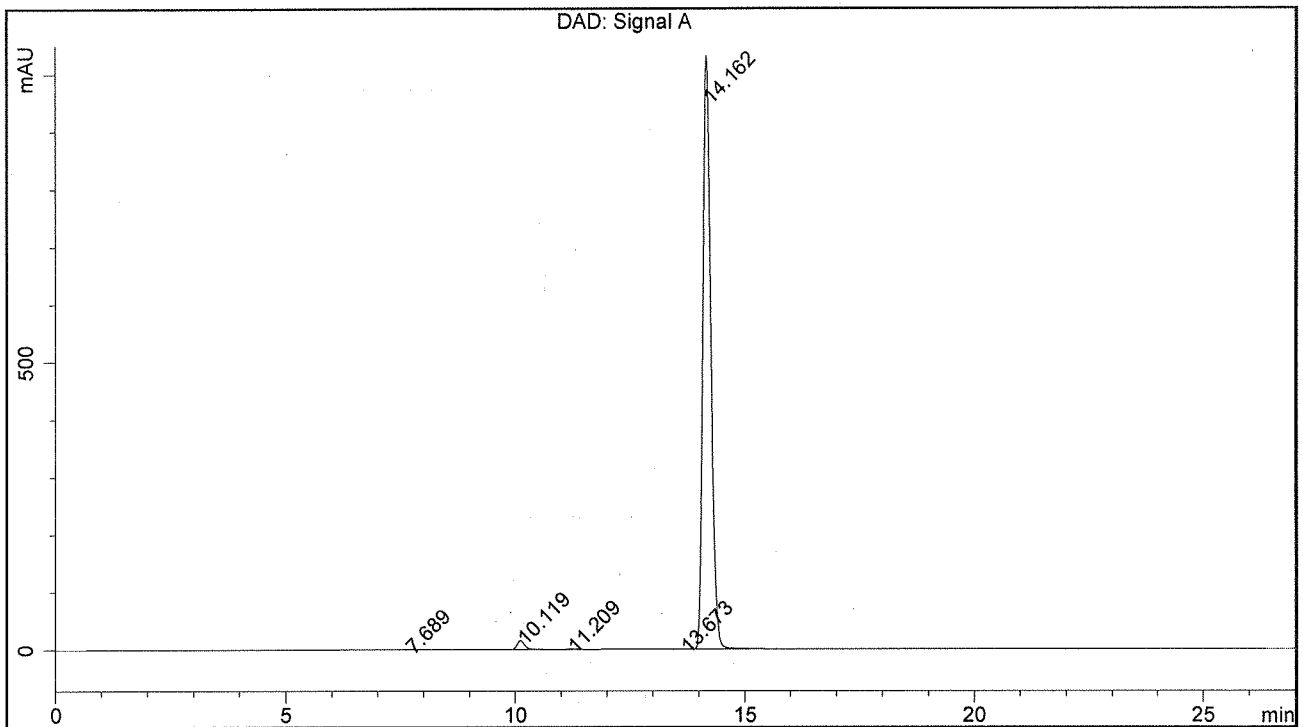
Lot Number: BDG 16140.3



BDG - Analysis of Vapendavir-d6

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase A : 50:50 10 mM diPotassium Hydrogen Phosphate pH=7.0 : Acetonitrile
 Mobile Phase B : 25:75 10 mM diPotassium Hydrogen Phosphate pH=7.0 : Acetonitrile
 Gradient (A:B) : T0=100:0, T15=0:100, T25=0:100, T26=100:0, T30=100:0
 Flow Rate : 1.0 mL/min
 Sample Solvent : 30:70 Water : Acetonitrile
 Injection Volume : 10 uL
 Detection : UV 250 nm

Sample Name	BDG 16140.3	Instrument	AnalyticalLC01
Acquisition	30/07/2014, 13:25:19	Method (rev.)	LC10624a (8)
Sequence	BDG_30Jul2014c - Reprocessed	Vial Position	2
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	7.69 min	0.7625	9.1154	0.1775 min	0.072 %
2	10.12 min	15.3670	173.9300	0.1740 min	1.364 %
3	11.21 min	0.7052	7.9676	0.1618 min	0.062 %
4	13.67 min	0.2571	2.5923	0.1256 min	0.020 %
5	14.16 min	1030.0952	12554.5232	0.1882 min	98.481 %