



BDG SYNTHESIS

Certificate of Analysis

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

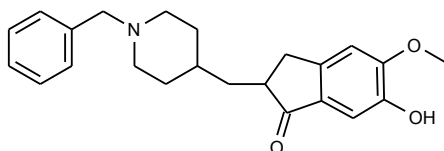
Barry Dent

Barry R. Dent, PhD, Director
28 November 2010

Name: 6''-O-Desmethyldonepezil

CAS Number: 147451-81-8

Structure:



Molecular Weight: $C_{23}H_{27}NO_3 = 365.47$

Lot Number: BDG 4825.2

Appearance: Cream, crystalline solid

Corrected Purity: 98.2 % (HPLC) - 2.0 % (ethyl acetate) - 1.3 % (water) = 94.9 %

Re-test Date: 28 November 2011

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.

Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

Residual Solvents: a small amount of ethyl acetate (2.0 % w/w) is 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.

High-resolution Mass Spectrum (ESI+)

Found m/z 366.2063. $C_{23}H_{28}NO_3$ $[M+H]^+$ requires m/z 366.2064. The deviation of 0.1 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A somewhat broadened, symmetrical peak is observed (98.2 %). 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 74.03, H 7.60, N 3.76 %
$C_{23}H_{27}NO_3 \cdot 0.4H_2O$	Requires:	C 74.13, H 7.52, N 3.76 %
$C_{23}H_{27}NO_3$	Requires:	C 75.59, H 7.45, N 3.83 %

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).

Karl-Fischer Analysis

	Found:	H ₂ O 1.3 %
$C_{23}H_{27}NO_3 \cdot 0.4H_2O$	Requires:	H ₂ O 1.9 %

Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

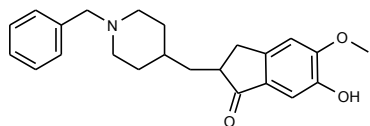
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

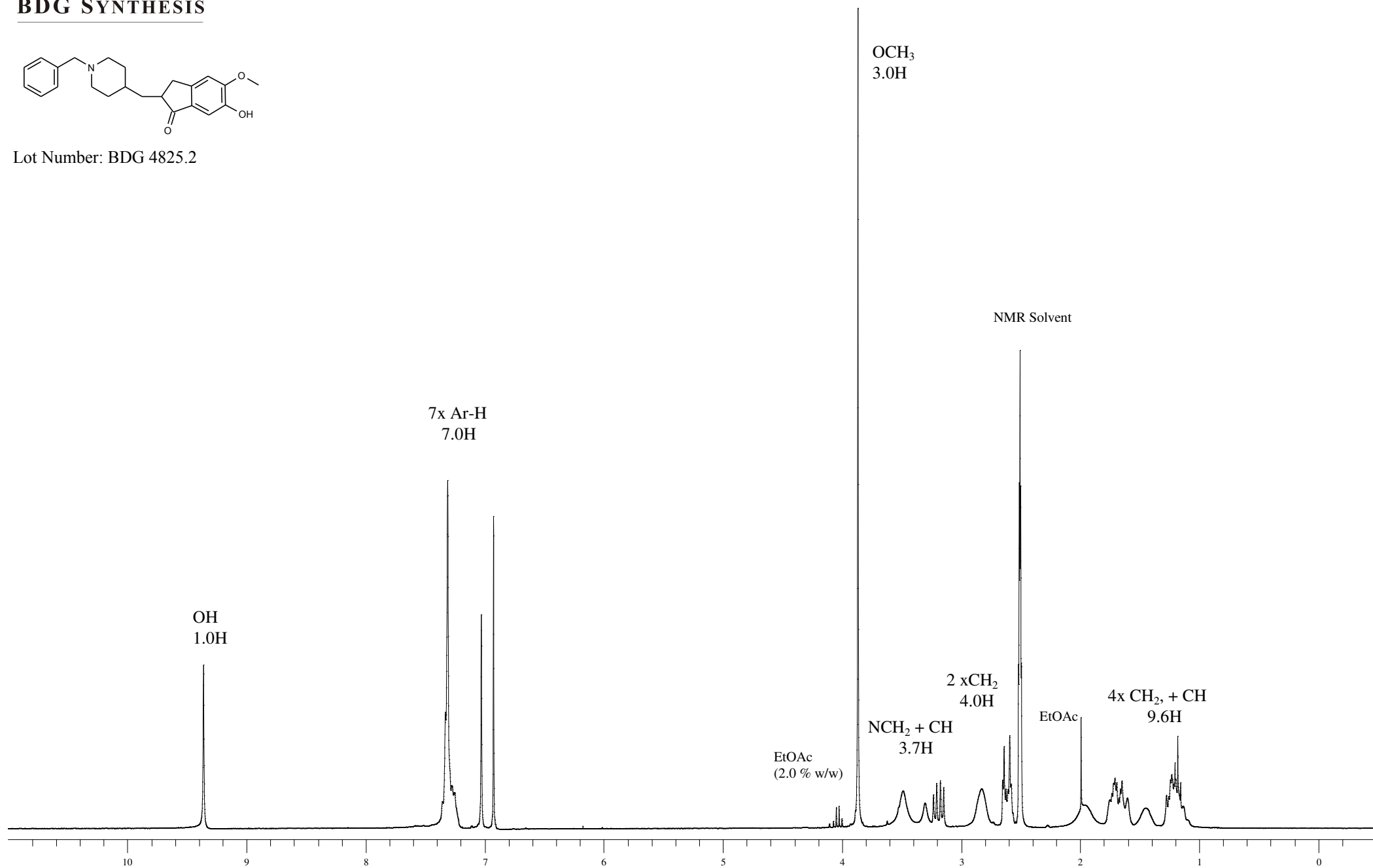


Proton NMR Spectrum of 6''-O-Desmethyldonepezil in DMSO-d₆

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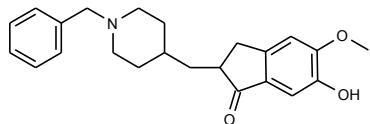
Lot Number: BDG 4825.2



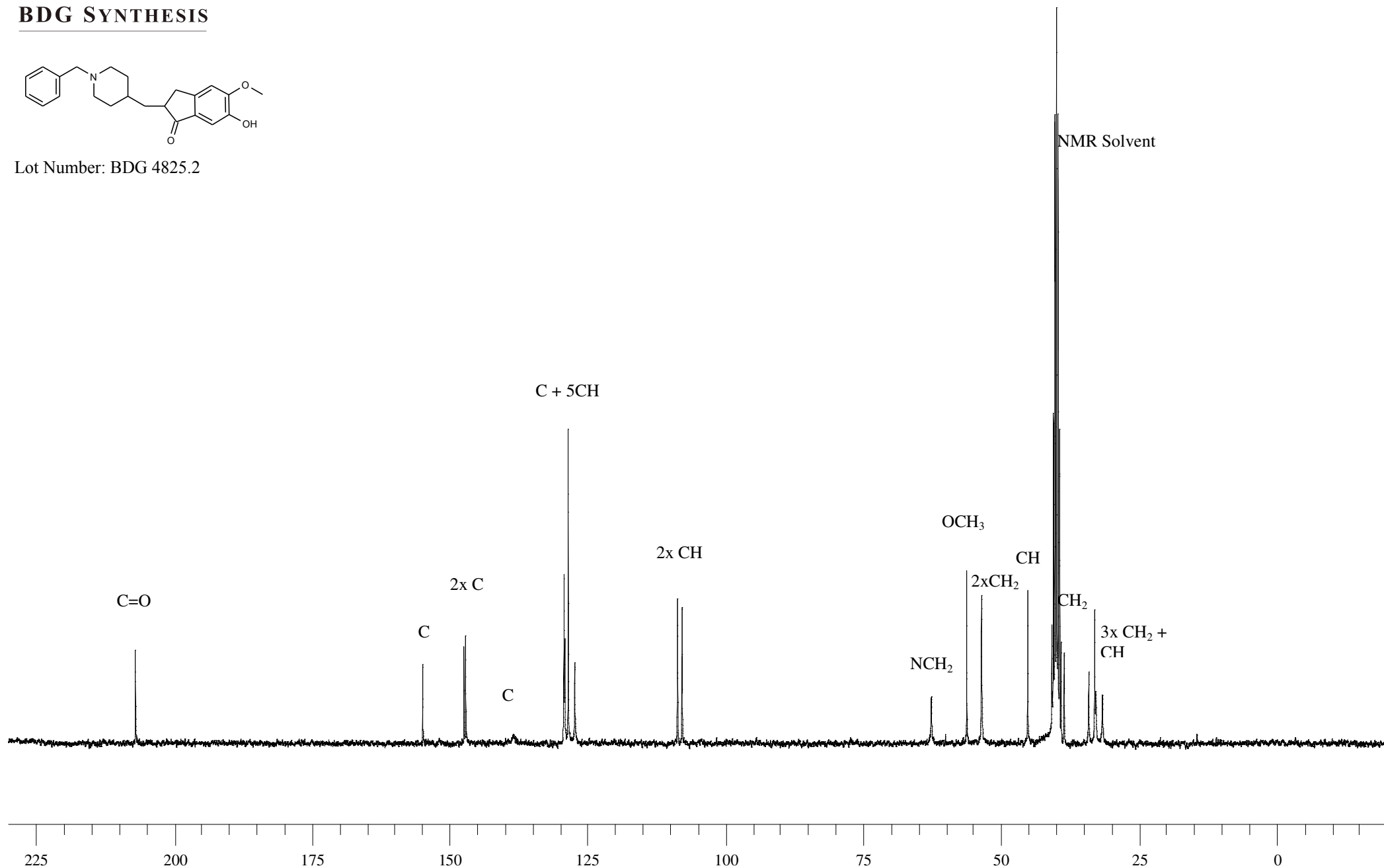


Carbon-13 NMR Spectrum of 6''-*O*-Desmethyldonepezil in DMSO-d₆

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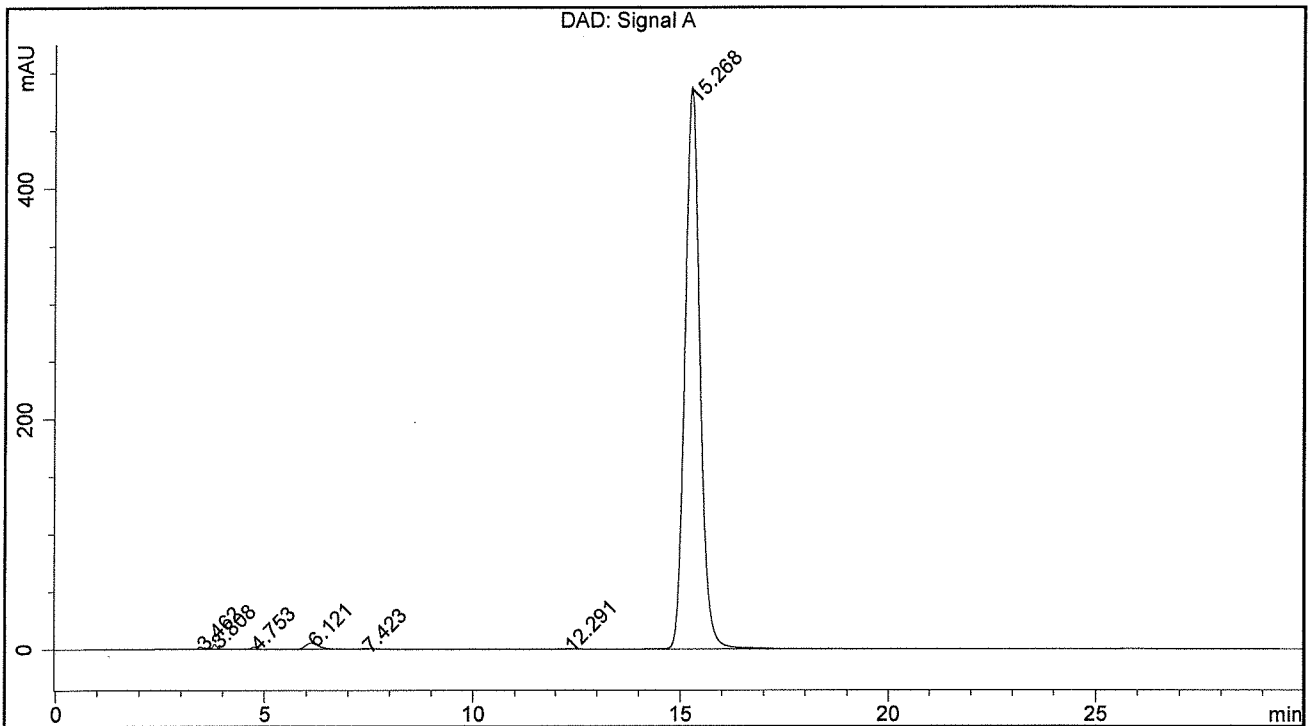
Lot Number: BDG 4825.2



BDG - Analysis of 6"-O-Desmethyldonepezil

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 4 x 3 mm
 Mobile Phase : 30:70 10 mM diPotassium Hydrogen Phosphate pH=7.0 : Methanol
 Flow Rate : 1.0 mL/min
 Sample Solvent : 1:2 Water : Methanol
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 268 nm

Sample Name	BDG 4825.2	Instrument	AnalyticalLC01
Acquisition	28/11/2010, 12:35:03	Method (rev.)	LC10416a (8)
Sequence	BDG_28Nov2010f - Reprocessed	Vial Position	23
Operator	solvation010\cerityadmin	Injection	1 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.46 min	2.0064	16.5234	0.1221 min	0.132 %
2	3.81 min	4.0875	32.6047	0.1191 min	0.261 %
3	4.75 min	1.9632	17.6625	0.1390 min	0.141 %
4	6.12 min	5.2865	131.1113	0.3753 min	1.049 %
5	7.42 min	0.6483	16.3184	0.3496 min	0.131 %
6	12.29 min	0.4186	11.6002	0.3513 min	0.093 %
7	15.27 min	487.6835	12269.4156	0.3855 min	98.193 %