



BDG SYNTHESIS

Certificate of Analysis

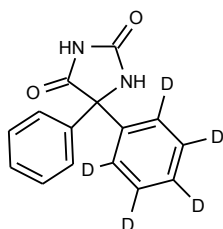
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Barry Dent

Barry R. Dent, PhD, Director
21 August 2007

Name: Phenytoin-d₅
CAS Number: 57-41-0 (unlabelled)

Structure:



Molecular Weight: C₁₅H₇D₅N₂O₂ = 257.30
Lot Number: BDG 7430.1
Appearance: White powder
Corrected Purity: 100.0 % (HPLC) - 0.4 % (diethyl ether) = 99.6 %
Isotopic Purity: Under 0.5 % d₀
Re-test Date: 21 August 2012
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. The material is susceptible 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 on the aromatic rings are absent, as shown by NMR integrals, when compared with the spectrum of unlabelled material, indicating clean deuteration.

Residual Solvents: a small amount of diethyl ether (0.4 % 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.

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 280.1098. $C_{15}H_7D_5N_2NaO_2$ $[M+Na]^+$ requires m/z 280.1105. The deviation of 2.5 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 sharp, symmetrical peak is observed (100.0 %). 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 70.12, H 2.86, D 4.08, N 10.98 %
$C_{15}H_7D_5N_2O_2$	Requires:	C 70.02, H 2.74, D 3.91, N 10.89 %

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).

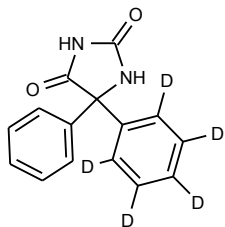
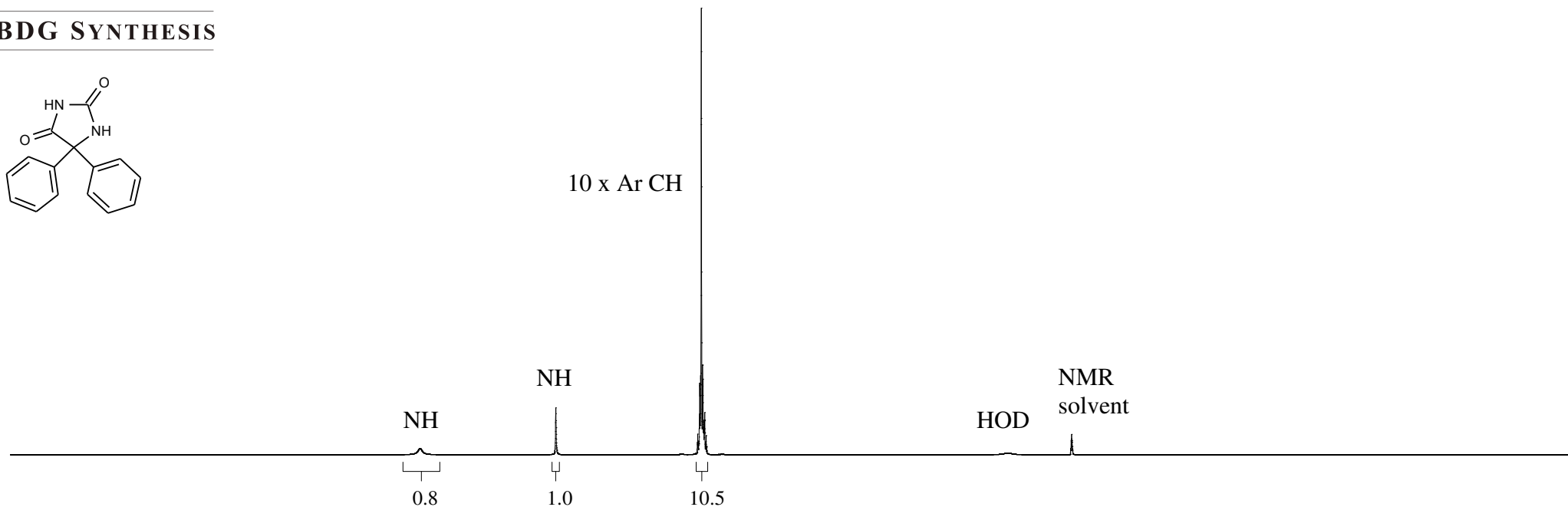
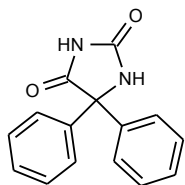
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

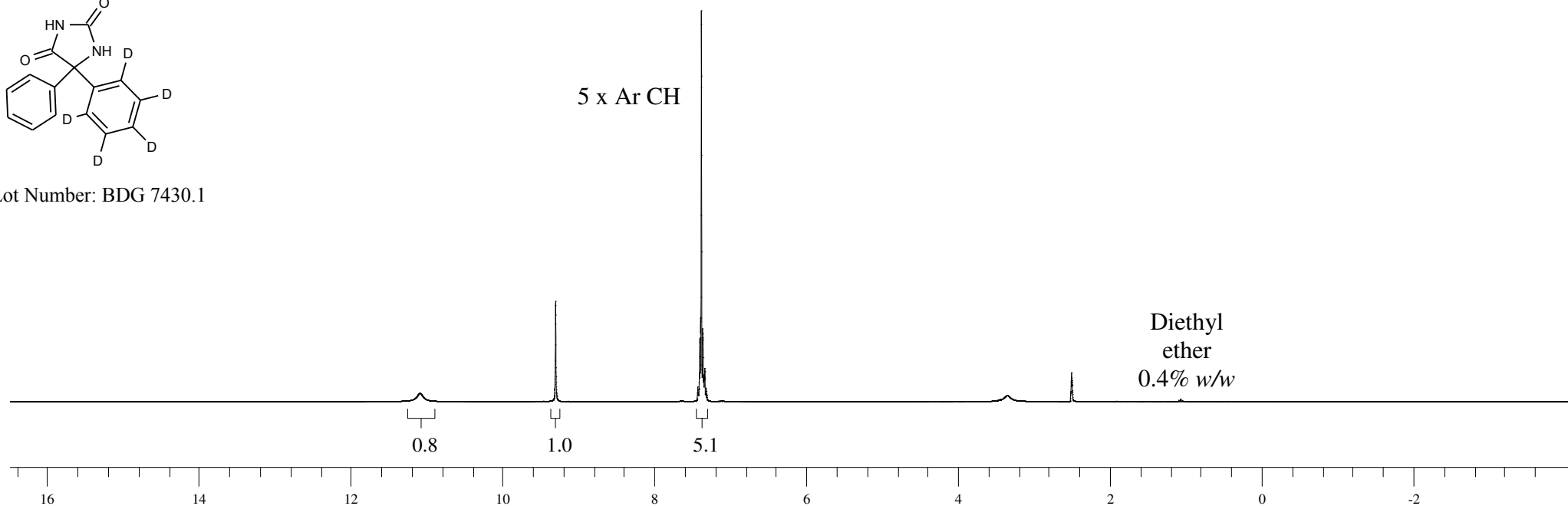


Proton NMR Spectrum of Phenytoin (top) and Phenytoin-d₅ (bottom) in DMSO-d₆

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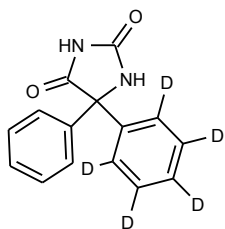
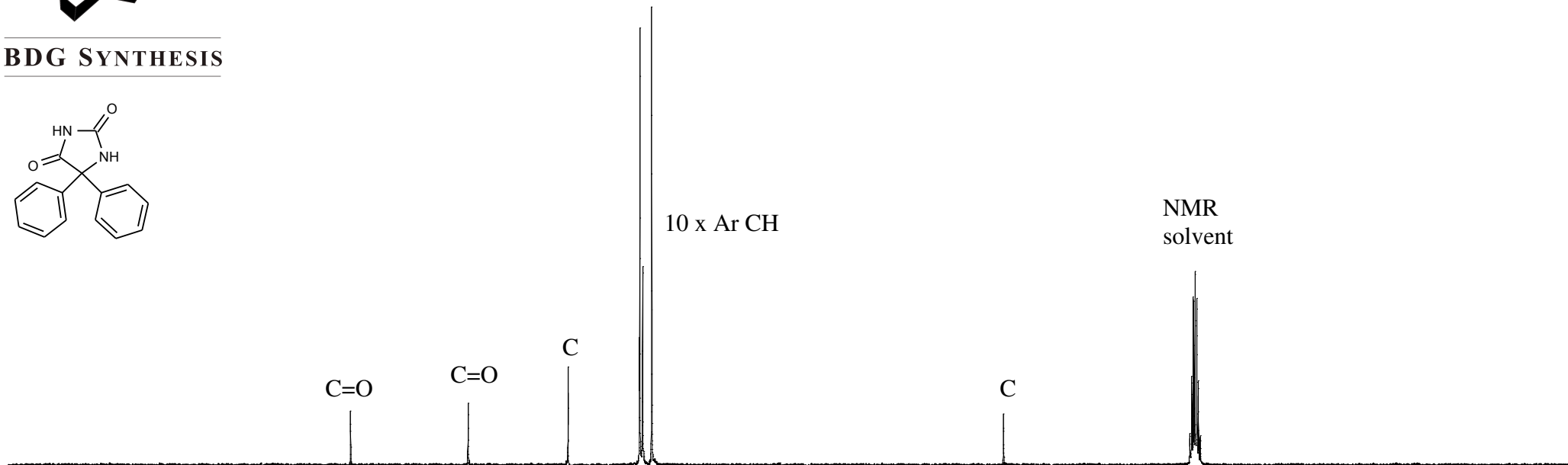
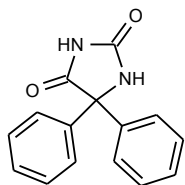
Lot Number: BDG 7430.1



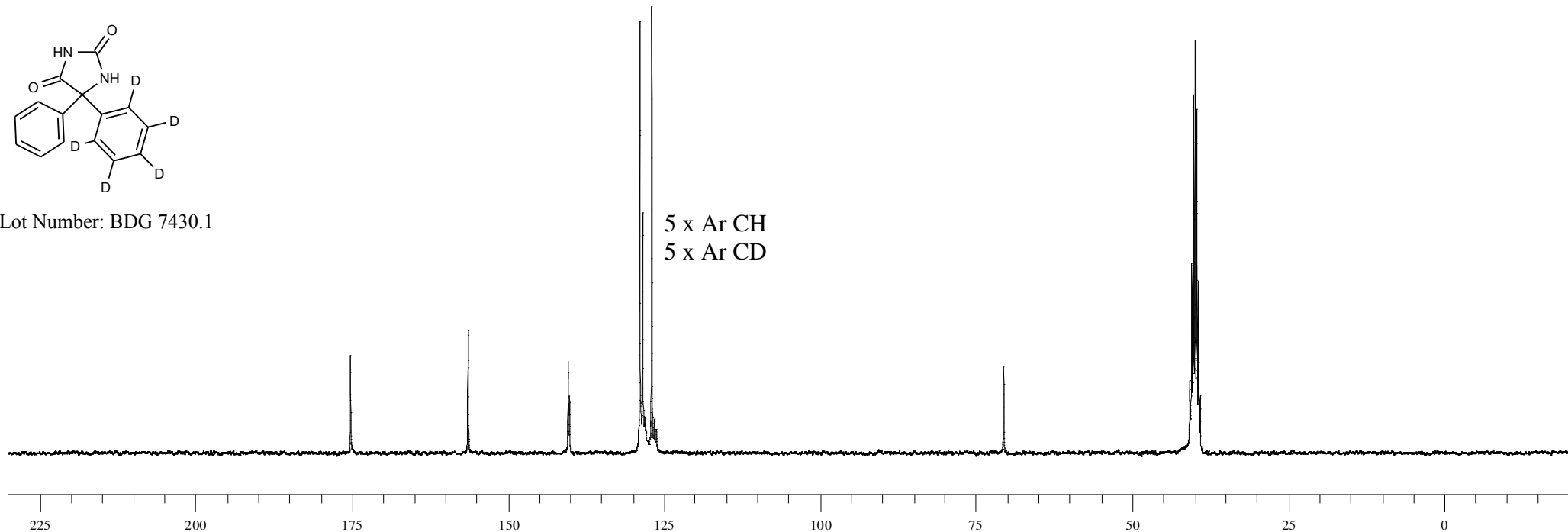


Carbon-13 NMR Spectrum of Phenytoin (top) and Phenytoin-d₅ (bottom) in DMSO-d₆

BDG SYNTHESIS



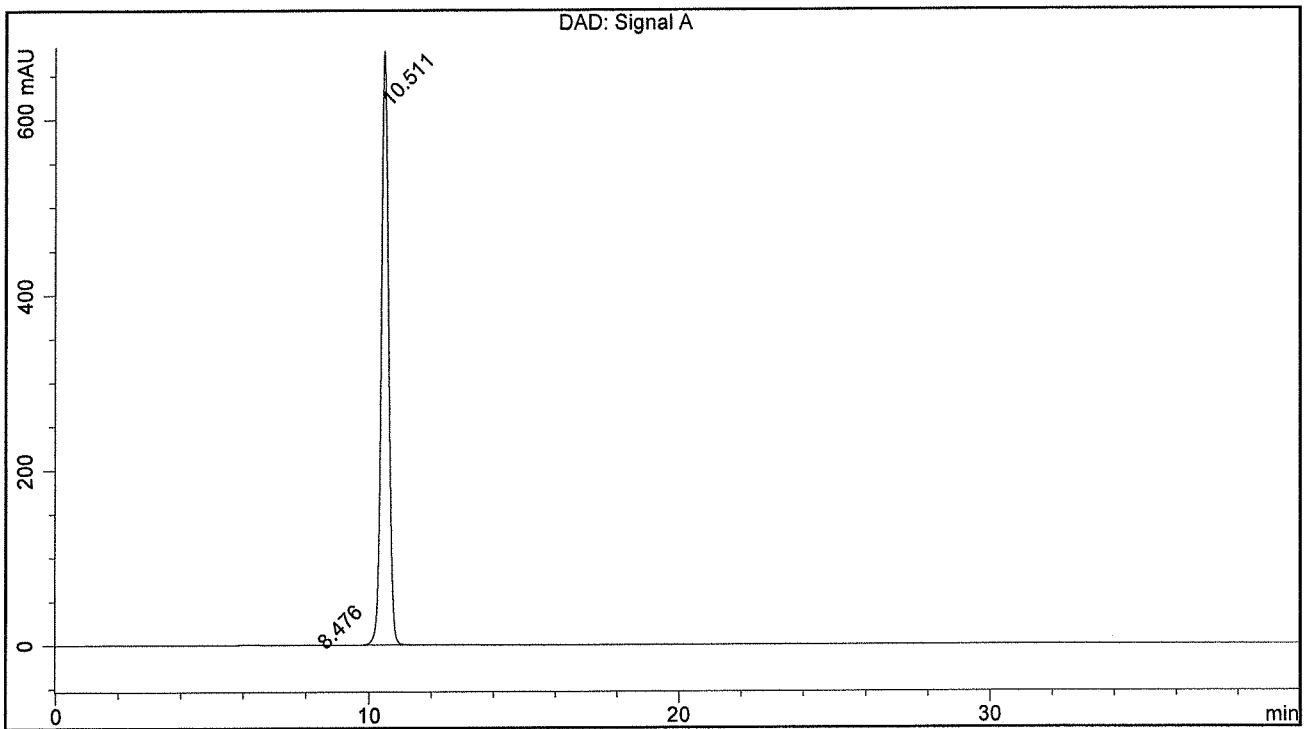
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BDG - Analysis of Phenytoin-d5

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 45:55 Water : Methanol
 Flow Rate : 1.0 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 220 nm

Sample Name	BDG 7430.1	Instrument	AnalyticalLC01
Acquisition	15/08/2007, 18:52:32	Method (rev.)	LC10183a
Sequence	BDG_15Aug2007b - Reprocessed	Vial Position	1
Operator	LC10183a	Injection	1 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	8.48 min	0.0639	1.0436	0.2025 min	0.009 %
2	10.51 min	677.8951	11195.9989	0.2547 min	99.991 %