



## BDG SYNTHESIS

### Certificate of Analysis

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

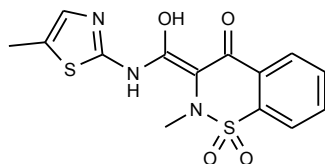
*Barry Dent*

Barry R. Dent, PhD, Director  
26 February 2013

**Name:** Meloxicam

**CAS Number:** 71125-38-7

**Structure:**



**Molecular Weight:**  $C_{14}H_{13}N_3O_4S_2 = 351.40$

**Lot Number:** BDG 3669

**Appearance:** Pale, yellow, crystalline solid

**Corrected Purity:** 99.1 % (HPLC) - 0.2 % (diethyl ether) = 98.9 %

**Re-test Date:** 26 February 2018

**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 diethyl ether (0.2 % w/w) is 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 or are manifest as collapsed multiplets, which is sometimes observed for nitrogen-containing compounds that can adopt several conformations in solution.

### High-resolution Mass Spectrum (TOF MS ES+)

Found  $m/z$  374.0237.  $C_{14}H_{13}N_3NaO_4S_2$   $[M+Na]^+$  requires  $m/z$  374.0245. The deviation of 2.1 ppm is within normally accepted limits for the establishment of identity by HRMS.

### HPLC

A sharp, slightly tailing peak is observed (99.1 %). 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

$C_{14}H_{13}N_3O_4S_2$	Found:	C 48.10, H 3.71, N 11.89, S 18.15 %
	Requires:	C 47.85, H 3.73, N 11.96, S 18.25 %

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).

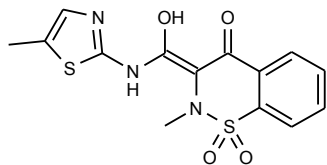
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

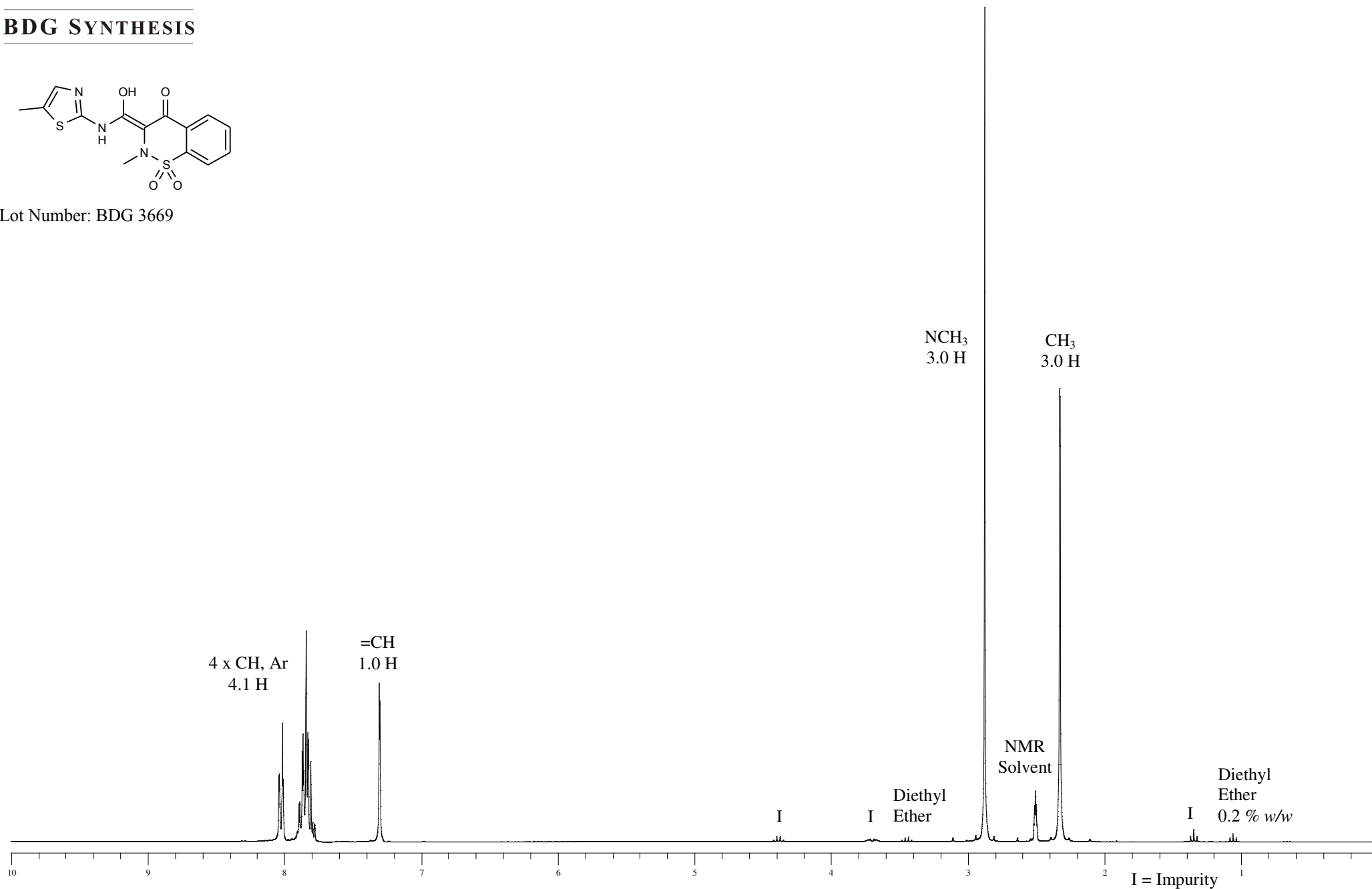


Proton NMR Spectrum of Meloxicam in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



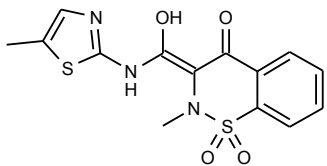
Lot Number: BDG 3669



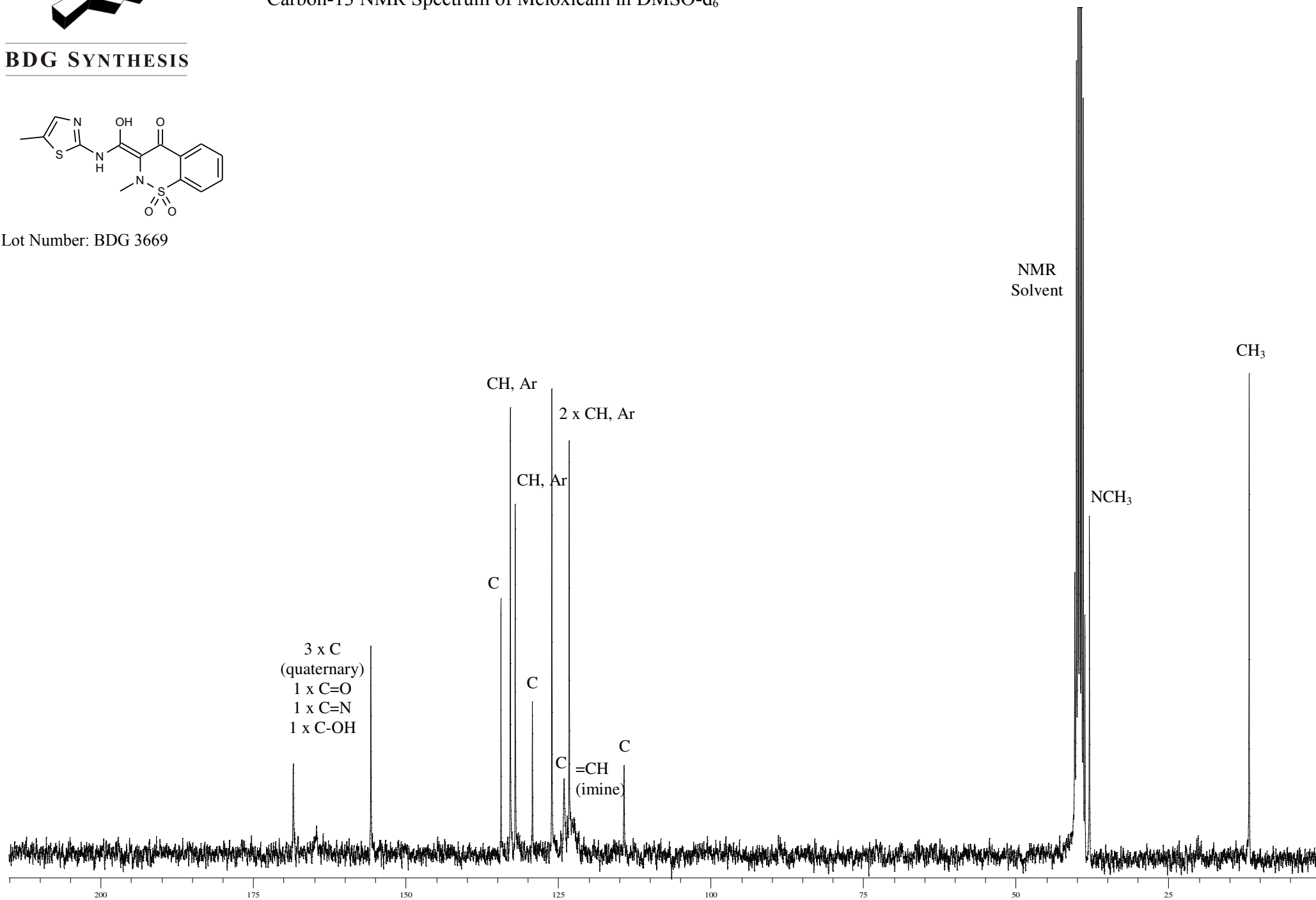


# Carbon-13 NMR Spectrum of Meloxicam in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



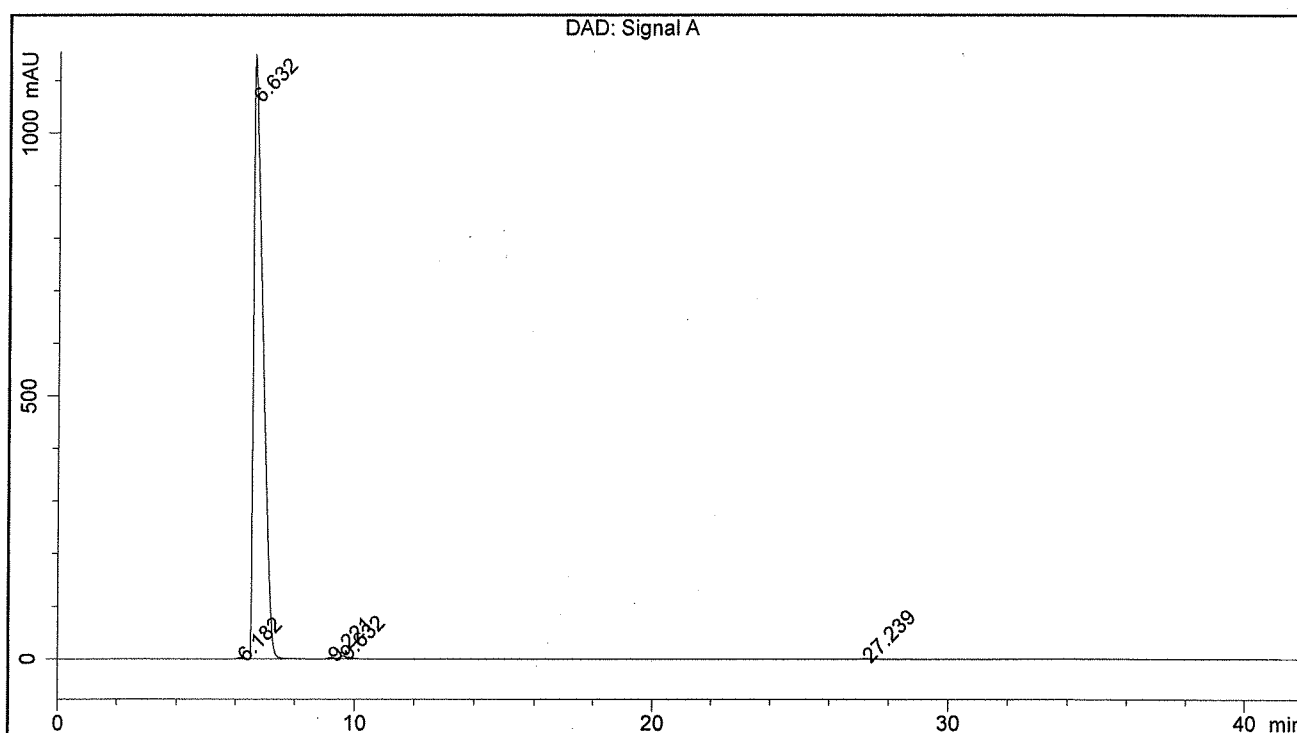
Lot Number: BDG 3669



BDG - Analysis of Meloxicam

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 0.1% Potassium diHydrogen Phosphate pH=6.0 : Methanol  
 Mobile Phase B : 30:70 0.1% Potassium diHydrogen Phosphate pH=6.0 : Methanol  
 Gradient ( A:B ) : T0=100:0, T25=100:0, T30=0:100, T40=0:100, T41=100:0, T45=100:0  
 Flow Rate : 1.0 mL/min  
 Sample Solvent : Mobile Phase  
 Column Temperature : 45C  
 Injection Volume : 20 uL  
 Detection : UV at 365 nm

<b>Sample Name</b>	BDG 3669	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	26/02/2013, 12:38:20	<b>Method (rev.)</b>	LC10564a ( 8)
<b>Sequence</b>	BDG_26Feb2013b - Reprocessed	<b>Vial Position</b>	1
<b>Operator</b>	solvation010\cerityadmin	<b>Injection</b>	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	6.18 min	2.8277	40.9005	0.2168 min	0.160 %
2	6.63 min	1144.3614	25403.0288	0.3190 min	99.145 %
3	9.22 min	2.4223	36.0828	0.2258 min	0.141 %
4	9.63 min	5.7166	108.1063	0.2766 min	0.422 %
5	27.24 min	0.9655	33.9596	0.4903 min	0.133 %