

BDG SYNTHESIS

Certificate of Analysis

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

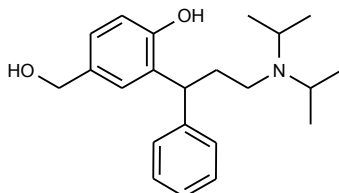
Barry Dent

Barry R. Dent, PhD, Director
2 June 2011

Name: 5-Hydroxymethyltolterodine

CAS Number: 200801-70-3

Structure:



Molecular Weight: $C_{22}H_{31}NO_2 = 341.49$

Lot Number: BDG 5187.8

Appearance: White, crystalline solid

Corrected Purity: 99.1 % (HPLC) - 0.3 % (diethyl ether) = 98.8 %

Re-test Date: 2 June 2016

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 (0.3 % 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 342.2450. $C_{22}H_{32}NO_2$ $[M+H]^+$ requires m/z 342.2435. The deviation of 5.2 ppm is somewhat outside normally accepted limits for the establishment of identity by HRMS, and the mass spectral data should be considered in conjunction with other identity criteria.

HPLC

A sharp, symmetrical 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

	Found:	C 77.54, H 9.35, N 4.12 %
$C_{22}H_{31}NO_2$	Requires:	C 77.38, H 9.15, N 4.10 %

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).

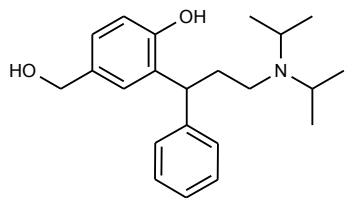
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

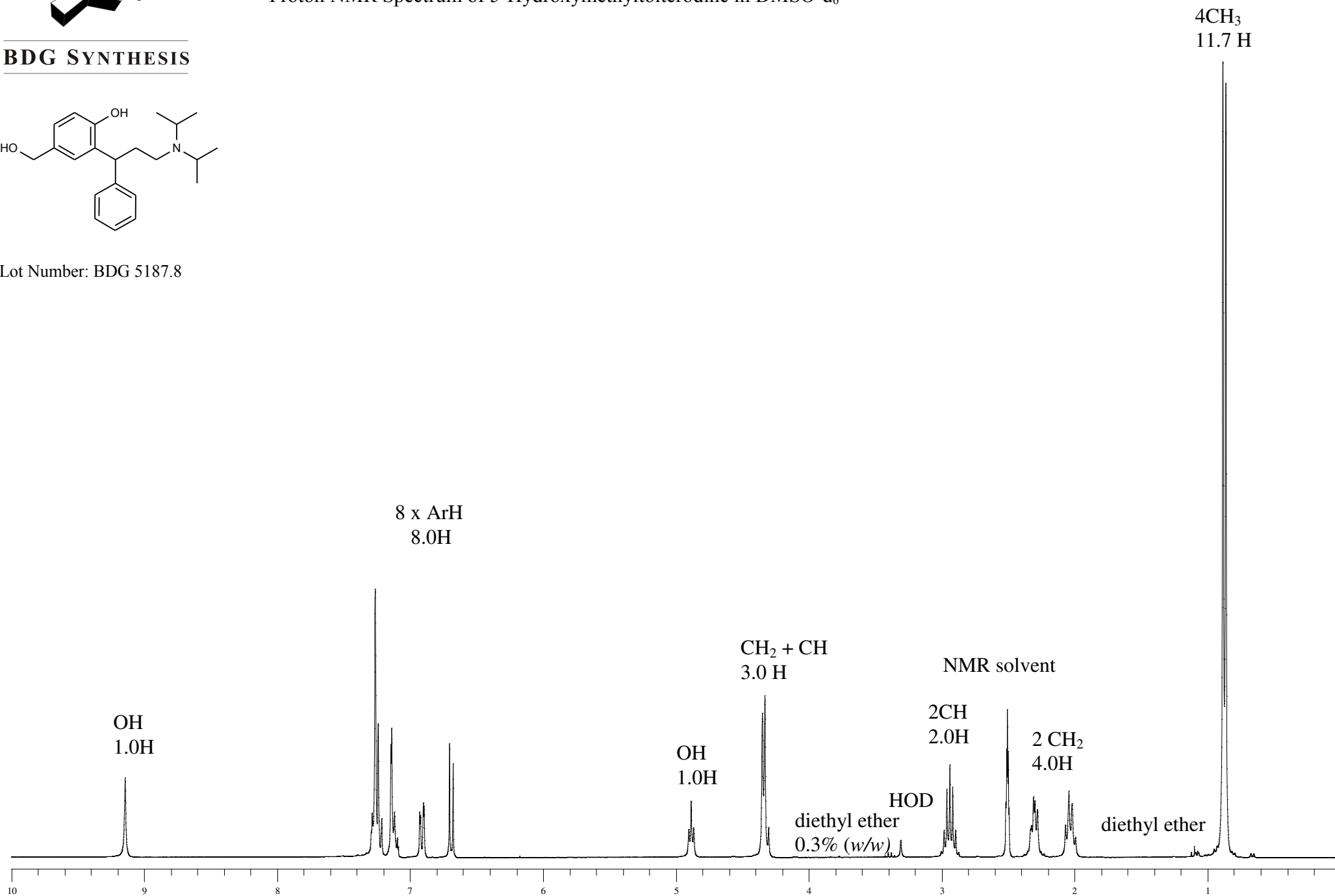


BDG SYNTHESIS

Proton NMR Spectrum of 5-Hydroxymethyltolterodine in DMSO-d₆



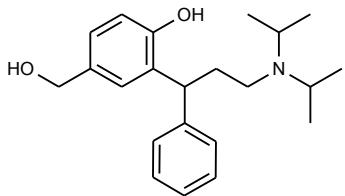
Lot Number: BDG 5187.8



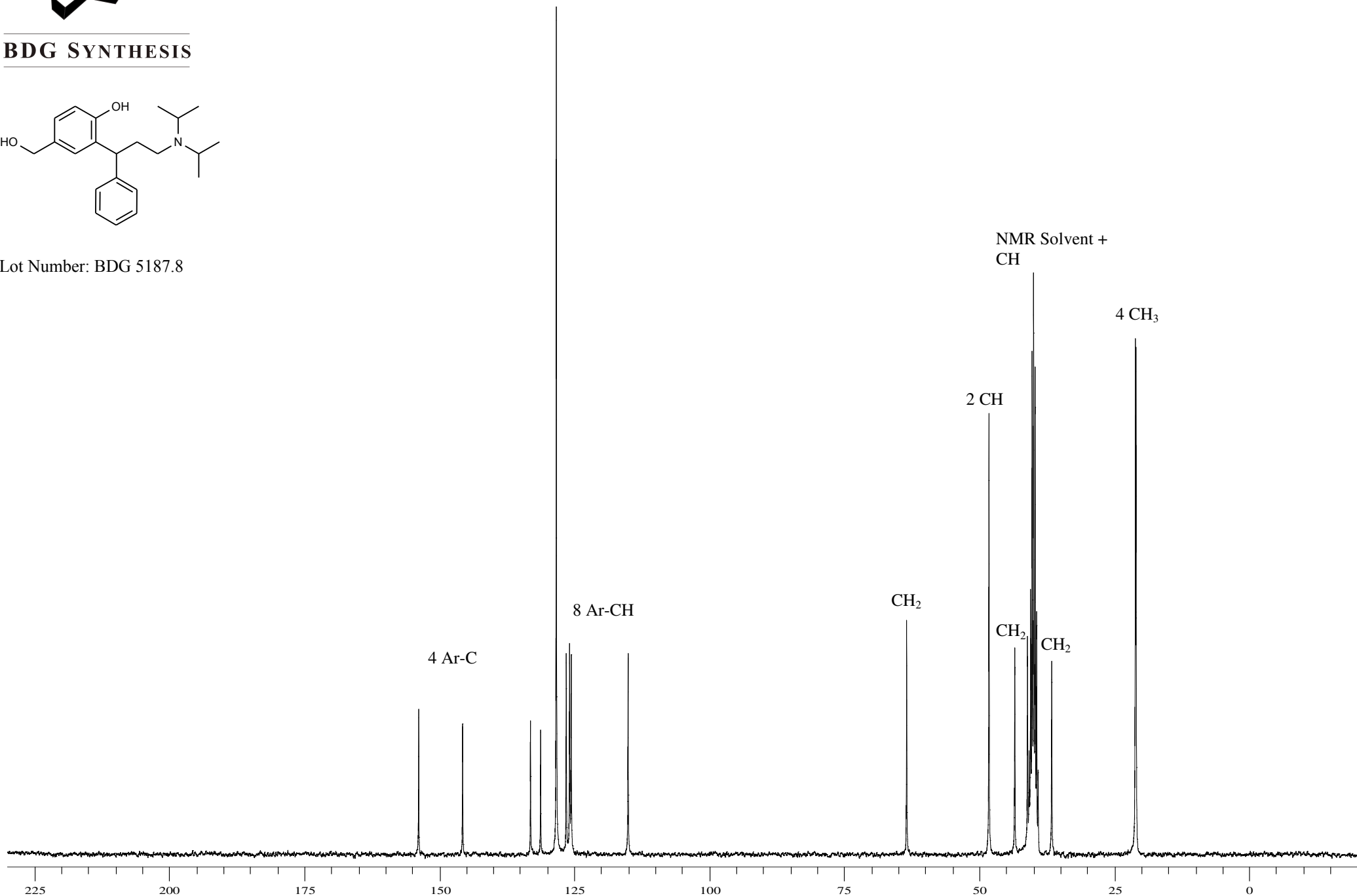


Carbon-13 NMR Spectrum of 5-Hydroxymethyltolterodine in DMSO-d₆

BDG SYNTHESIS



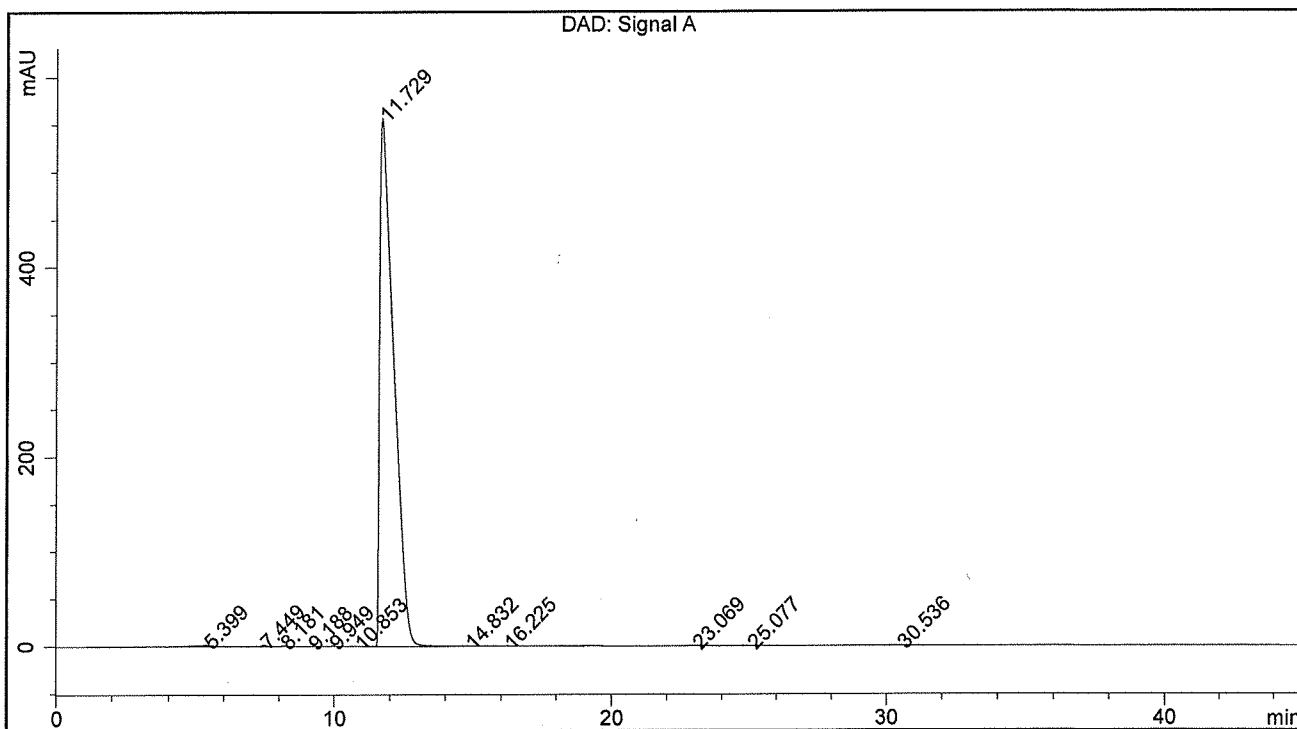
Lot Number: BDG 5187.8



BDG - Analysis of 5-Hydroxymethyltolterodine

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 78:22 10 mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile
 Flow Rate : 1.0 mL/min
 Column Temperature : 20C
 Sample Solvent : Mobile Phase
 Injection Volume : 10 uL
 Detection : UV at 225 nm

Sample Name	BDG 5187.8	Instrument	AnalyticalLC01
Acquisition	01/06/2011, 19:38:47	Method (rev.)	LC10445a (11)
Sequence	BDG_01Jun2011c - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	5.40 min	1.5923	82.5315	0.6527 min	0.418 %
2	7.45 min	1.8365	22.3794	0.1822 min	0.113 %
3	8.18 min	0.7099	10.2744	0.2049 min	0.052 %
4	9.19 min	0.1575	2.3780	0.2001 min	0.012 %
5	9.95 min	0.3313	7.5519	0.3220 min	0.038 %
6	10.85 min	0.1672	3.6308	0.2813 min	0.018 %
7	11.73 min	557.0120	19542.5871	0.4973 min	99.054 %
8	14.83 min	0.3658	11.3241	0.3852 min	0.057 %
9	16.22 min	0.2421	5.6839	0.3139 min	0.029 %
10	23.07 min	0.4626	17.2143	0.4502 min	0.087 %
11	25.08 min	0.2101	12.7723	0.7173 min	0.065 %
12	30.54 min	0.1784	10.8242	0.7238 min	0.055 %