

## BDG SYNTHESIS

### Certificate of Analysis

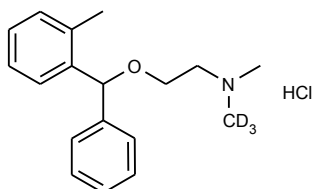
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

*Neil Beare*

Neil Beare, PhD, Director  
23 September 2014

**Name:** Orphenadrine-d<sub>3</sub> HCl  
**CAS Number:** 83-98-7 (unlabelled free base)

**Structure:**



**Molecular Weight:** C<sub>18</sub>H<sub>20</sub>D<sub>3</sub>NO·HCl = 308.86  
**Lot Number:** BDG 1468  
**Appearance:** Off-white, crystalline solid  
**Corrected Purity:** 85.4 % (HPLC) - 0.8 % (ethanol) = 84.6 %  
**Isotopic Purity:** Under 0.5 % d<sub>0</sub>  
**Re-test Date:** 23 September 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.

## 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 site of deuteration are greatly diminished, compared with what would be expected for unlabelled material, indicating clean deuteration.

Residual Solvents: a small amount of ethanol (0.8 % w/w) is observed.

Impurities: 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 site of deuteration have collapsed to small multiplets compared with what would be expected for unlabelled material, indicating clean deuteration.

Impurities: significant impurities are evident in the spectrum.

### High-resolution Mass Spectrum (ESI+)

Found  $m/z$  273.2040.  $C_{18}H_{21}D_3NO$   $[M+H]^+$  requires  $m/z$  273.2046. The deviation of 2.2 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 (85.4 %). 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 66.86, H 6.92, D 1.98, N 4.38 %
$C_{18}H_{20}D_3NO \cdot HCl$	Requires:	C 70.00, H 6.85, D 1.96, N 4.53 %

The elemental analyses fall substantially outside those expected.

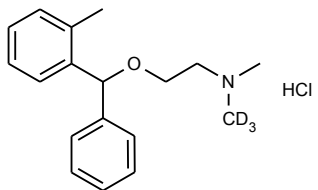
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

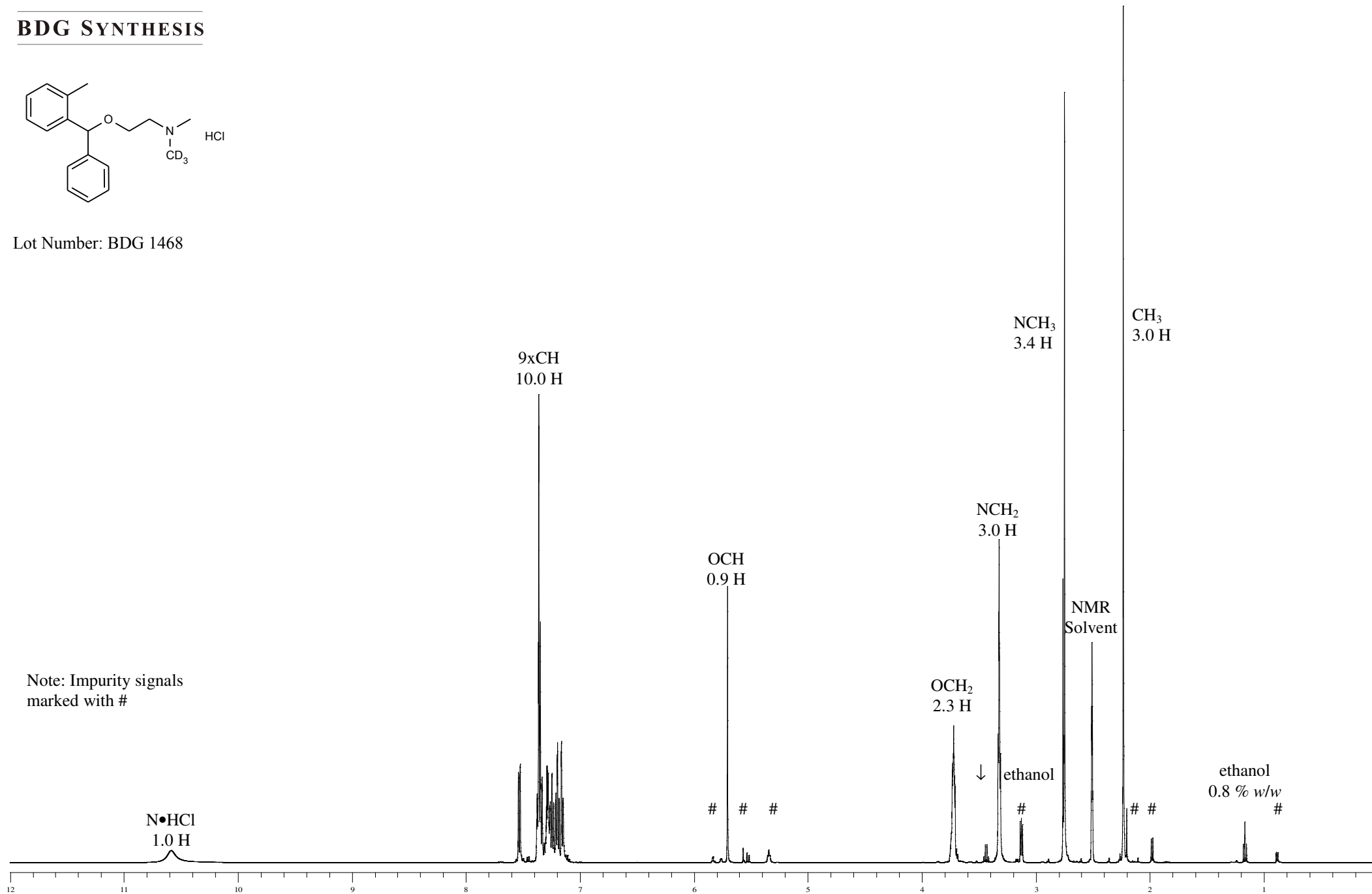


Proton NMR Spectrum of Orphenadrine-d<sub>3</sub> HCl in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



Lot Number: BDG 1468

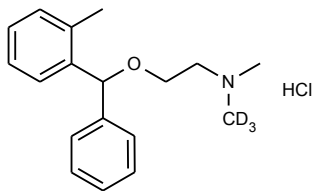


Note: Impurity signals marked with #

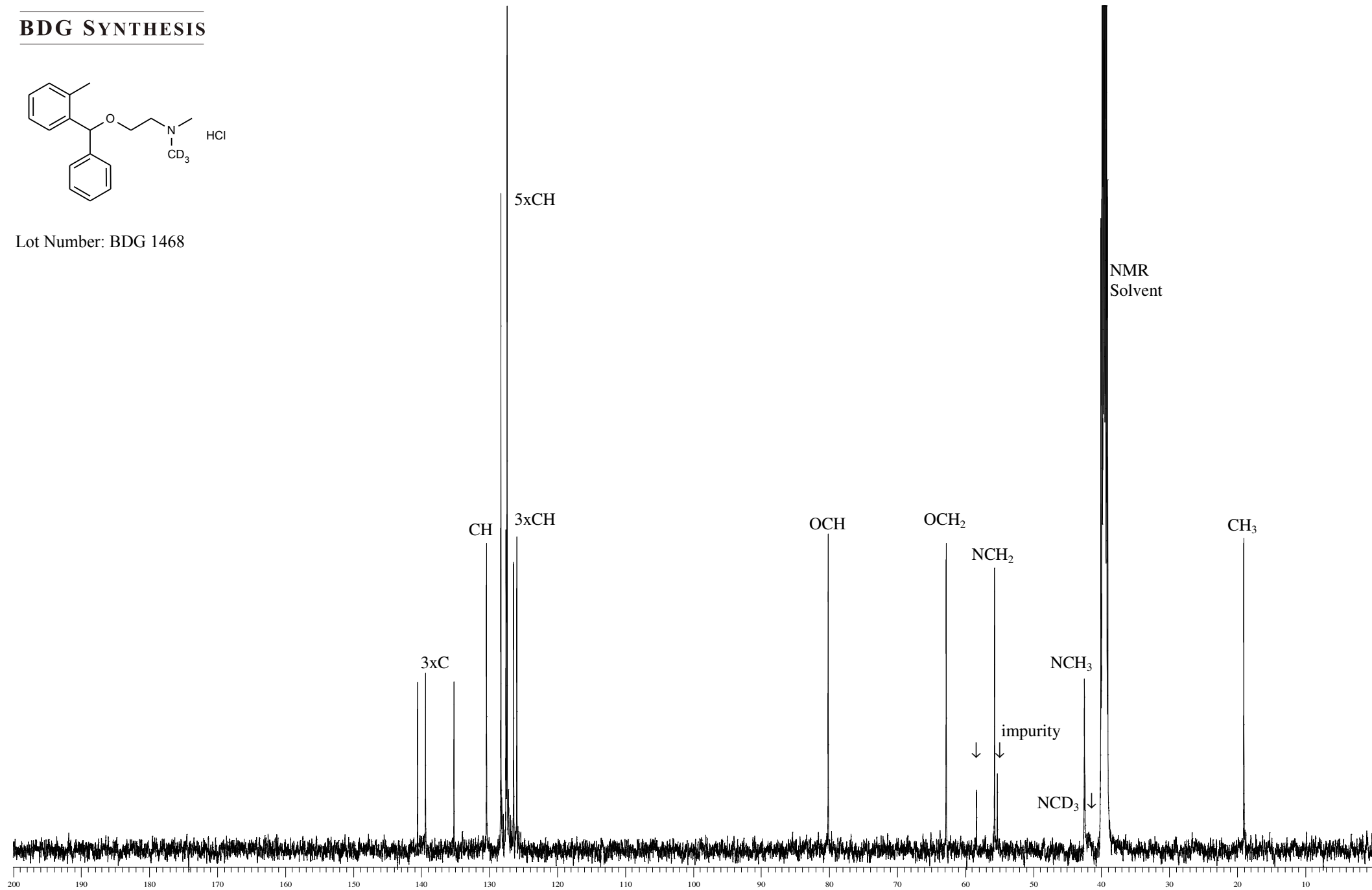


# Carbon-13 NMR Spectrum of Orphenadrine-d<sub>3</sub> HCl in DMSO-d<sub>6</sub>

## BDG SYNTHESIS



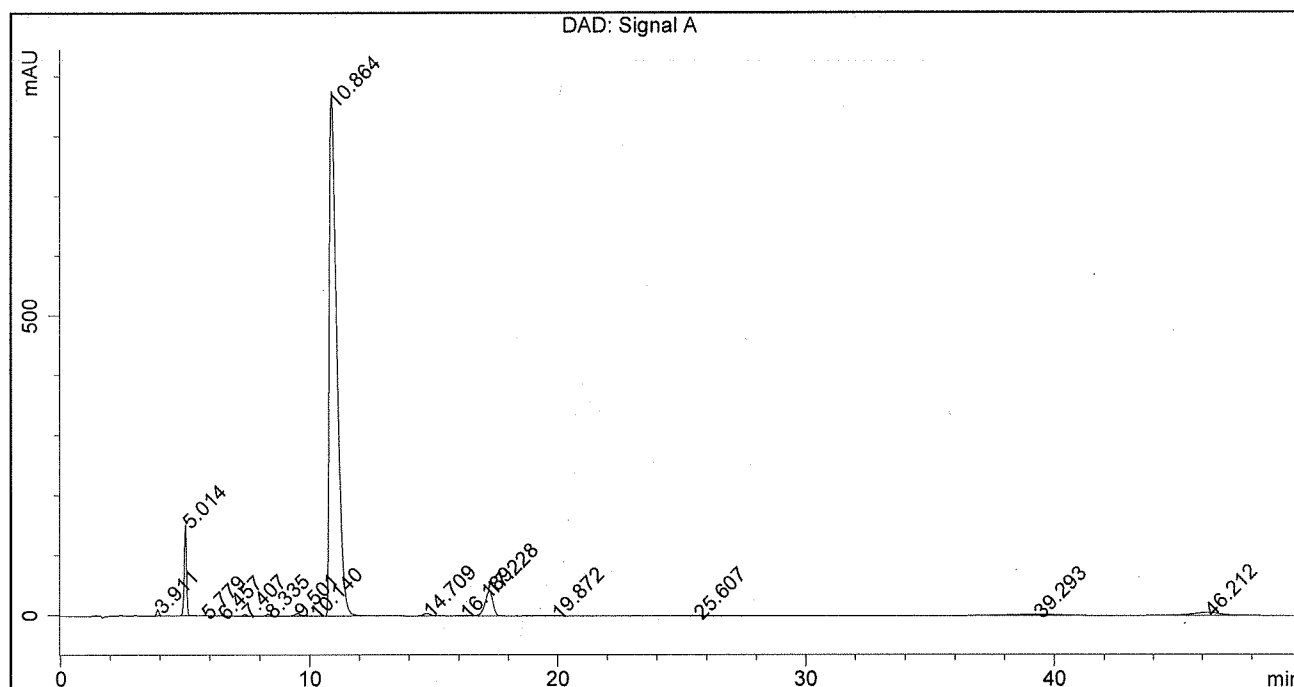
Lot Number: BDG 1468



BDG - Analysis of Orphenadrine-d3 HCl

Column : Phenomenex Luna C18(2) 5 um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase : 30:45:25 25 mM Ammonium Phosphate pH=7.9 : Methanol : Acetonitrile  
 Flow Rate : 1.5 mL/min . . . . . Column Temperature : 40 C . . . . . Detection: UV 220 nm  
 Sample Solvent : 50:50 Water : Acetonitrile . . . . . Injection Volume : 10 uL

<b>Sample Name</b>	BDG 1468	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	23/09/2014, 14:20:25	<b>Method (rev.)</b>	LC10630a ( 11)
<b>Sequence</b>	BDG_23Sep2014c - Reprocessed	<b>Vial Position</b>	33
<b>Operator</b>	solvation010\cerityadmin	<b>Injection</b>	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.91 min	10.4241	68.0572	0.1022 min	0.318 %
2	5.01 min	151.9680	995.1695	0.1024 min	4.647 %
3	5.78 min	0.4935	7.2584	0.1856 min	0.034 %
4	6.46 min	0.6171	5.3815	0.1277 min	0.025 %
5	7.41 min	2.8752	25.3849	0.1350 min	0.119 %
6	8.34 min	3.0987	40.1846	0.1893 min	0.188 %
7	9.50 min	4.1757	80.5389	0.2690 min	0.376 %
8	10.14 min	0.5906	12.6689	0.2643 min	0.059 %
9	10.86 min	875.0965	18288.5034	0.3198 min	85.393 %
10	14.71 min	4.0968	89.5770	0.3271 min	0.418 %
11	16.19 min	1.1396	27.8911	0.3095 min	0.130 %
12	17.23 min	41.0016	910.3274	0.3410 min	4.251 %
13	19.87 min	0.4063	10.1912	0.3066 min	0.048 %
14	25.61 min	0.5361	19.3376	0.4328 min	0.090 %
15	39.29 min	2.2098	362.6076	1.9284 min	1.693 %
16	46.21 min	5.3281	473.6702	1.0492 min	2.212 %