



## BDG SYNTHESIS

### Certificate of Analysis

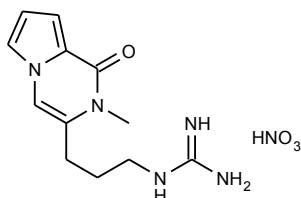
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

*Barry Dent*

Barry R. Dent, PhD, Director  
13 August 2011

**Name:** Peramine Nitrate  
**CAS Number:** 102482-94-0 (free base)

**Structure:**



**Molecular Weight:**  $C_{12}H_{17}N_5O \cdot HNO_3 = 310.31$   
**Lot Number:** BDG 5128.7  
**Appearance:** Off-white, crystalline solid  
**Corrected Purity:** 99.1 % (HPLC) - 0.9 % (methanol) - 0.9 % (water) = 97.3 %  
**Re-test Date:** 13 August 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 methanol (0.9 % 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  $m/z$  248.1509.  $C_{12}H_{18}N_5O$   $[M+H]^+$  requires  $m/z$  248.1506. The deviation of 1.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

### 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 44.91, H 5.65, N 26.97 %
$C_{12}H_{17}N_5O \cdot HNO_3 \cdot 0.4H_2O$	Requires:	C 45.39, H 5.97, N 26.47 %
$C_{12}H_{17}N_5O \cdot HNO_3$	Requires:	C 46.45, H 5.85, N 27.08 %

The elemental analyses fall somewhat outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the "best-fit" hydrated molecular formula is given.

### Karl-Fischer Analysis

	Found:	H <sub>2</sub> O 0.9 %
$C_{12}H_{17}N_5O \cdot HNO_3 \cdot 0.4H_2O$	Requires:	H <sub>2</sub> O 2.3 %

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.

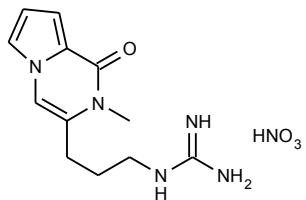
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

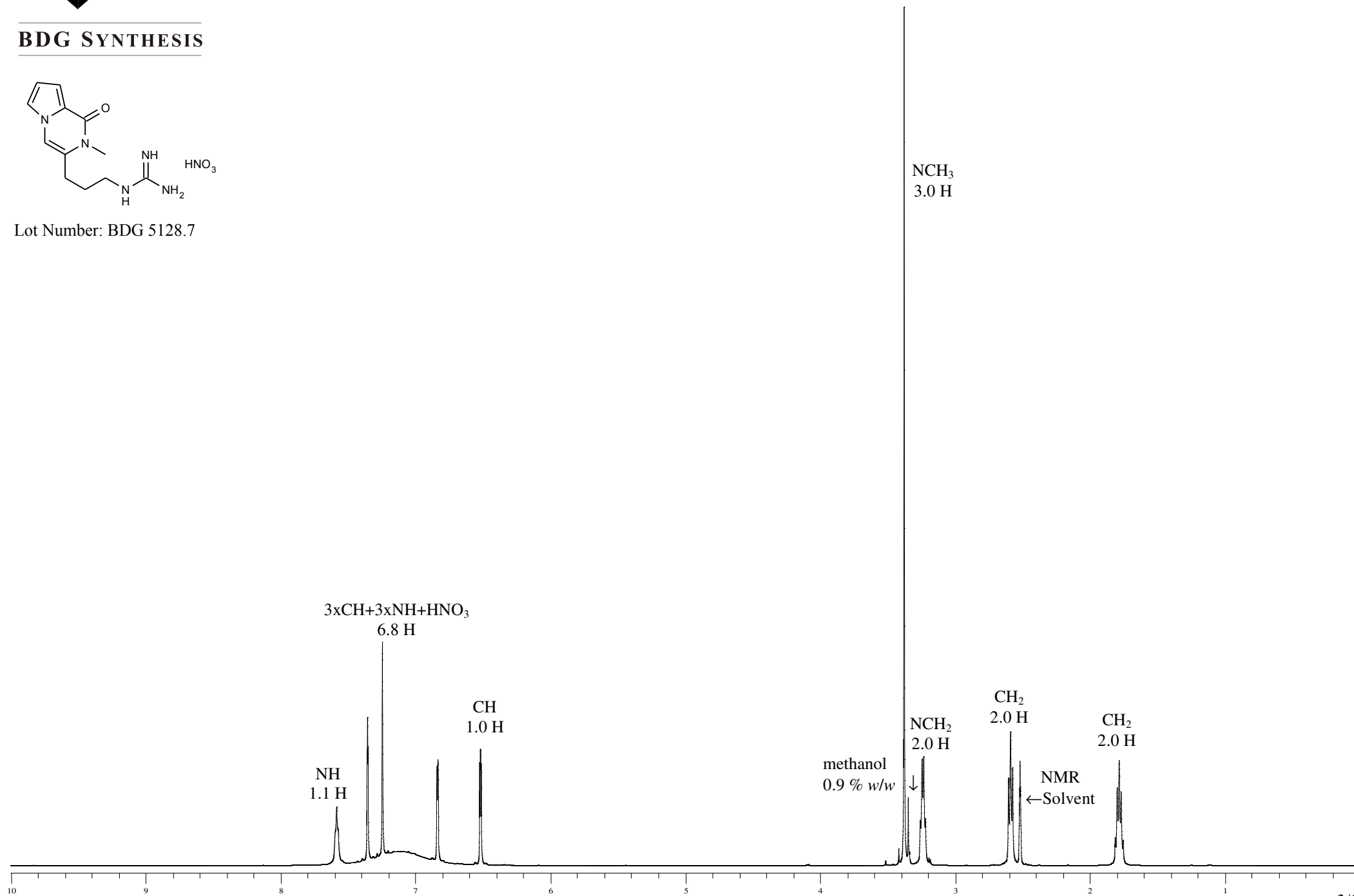


Proton NMR Spectrum of Peramine Nitrate in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



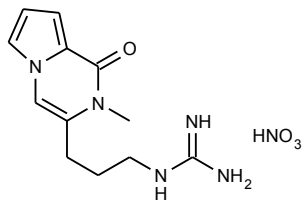
Lot Number: BDG 5128.7



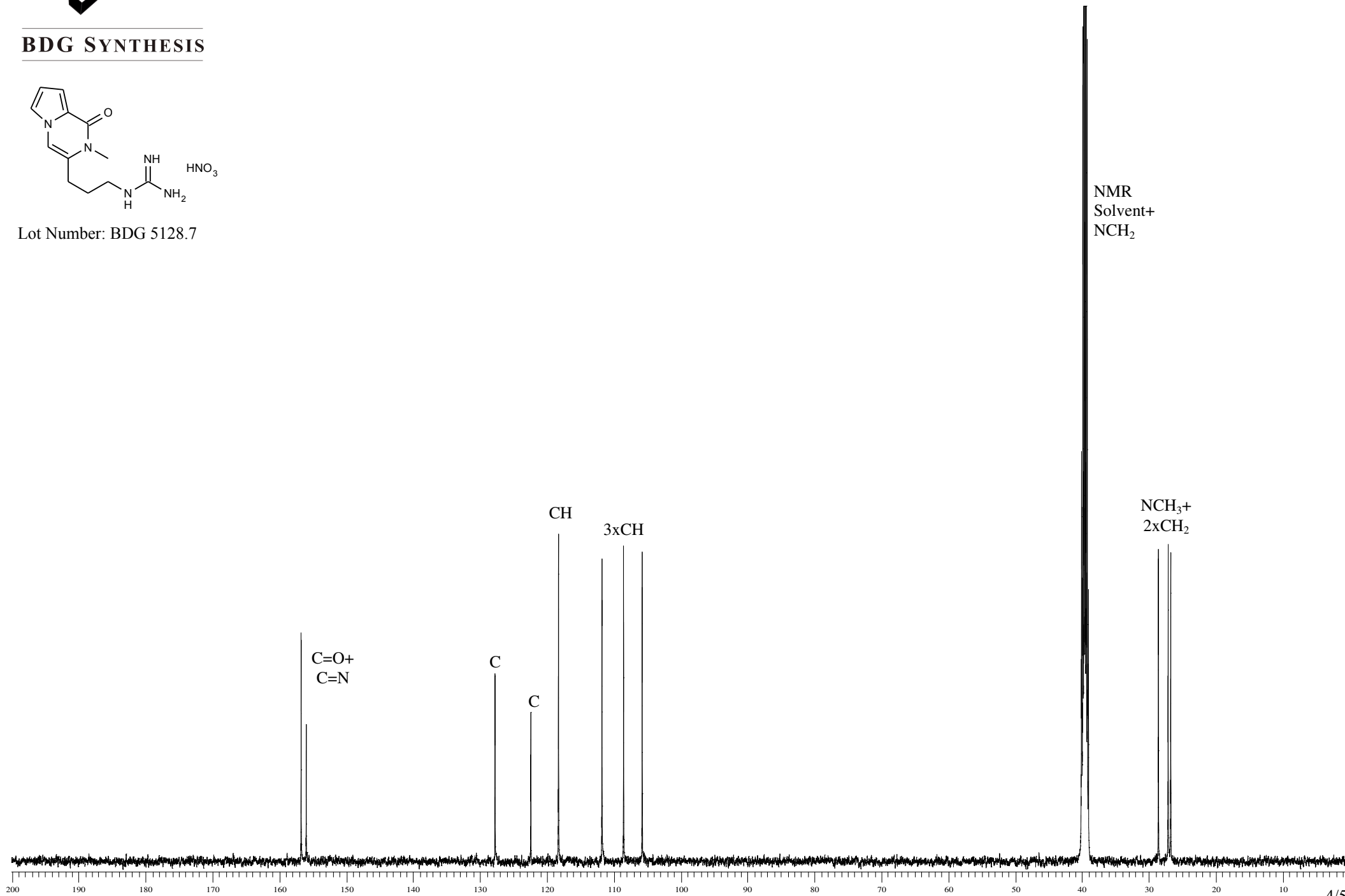


Carbon-13 NMR Spectrum of Peramine Nitrate in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



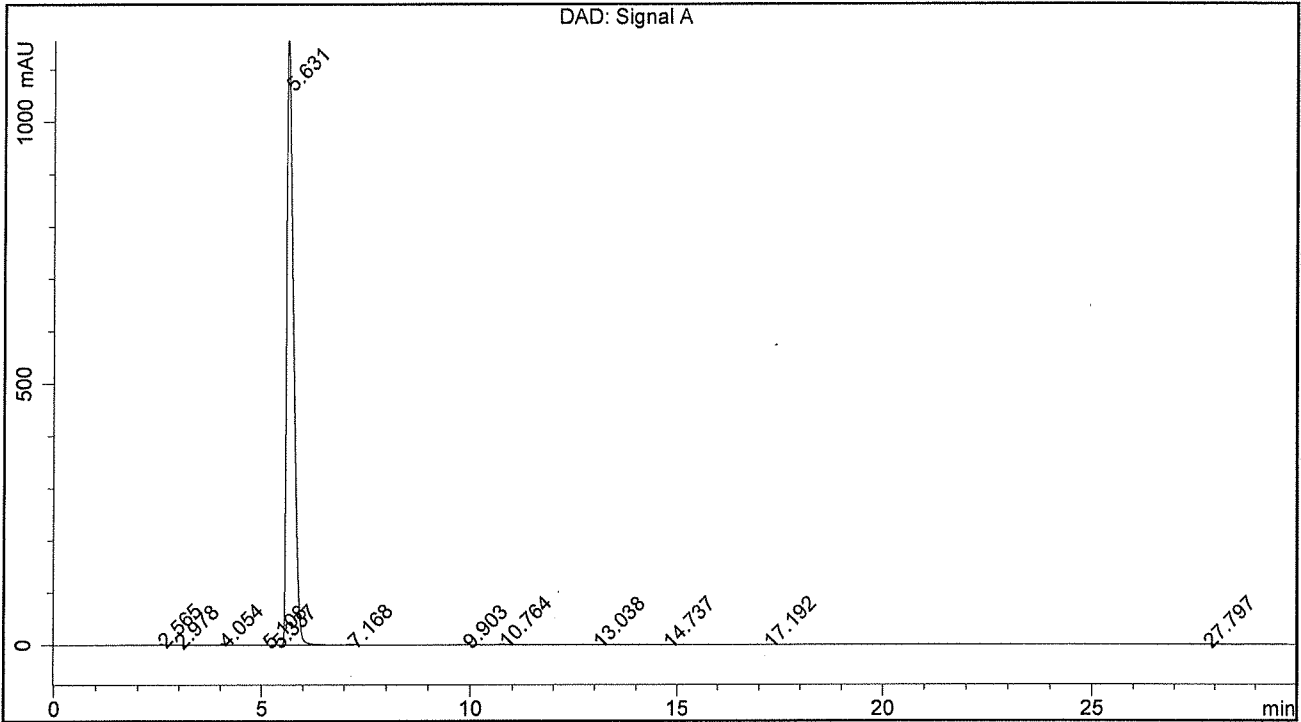
Lot Number: BDG 5128.7



BDG - Analysis of Peramine Nitrate

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase : 85:15 25 mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile  
 Injection Volume : 10 uL . . . . . Flow Rate : 1.0 mL/min . . . . . Column Temperature : 20C  
 Sample Solvent : Mobile Phase . . . . . Detection : UV at 285 nm

<b>Sample Name</b>	BDG 5128.7	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	12/08/2011, 11:06:04	<b>Method (rev.)</b>	LC10454a ( 9)
<b>Sequence</b>	BDG_12Aug2011e - 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	2.56 min	2.7896	13.8930	0.0741 min	0.103 %
2	2.98 min	0.2895	2.3933	0.1165 min	0.018 %
3	4.05 min	2.7783	15.3824	0.0846 min	0.114 %
4	5.11 min	0.4436	4.5373	0.1473 min	0.033 %
5	5.34 min	0.6852	5.6812	0.1247 min	0.042 %
6	5.63 min	1169.2290	13430.5483	0.1700 min	99.117 %
7	7.17 min	1.9047	22.5687	0.1782 min	0.167 %
8	9.90 min	0.1635	2.8081	0.2164 min	0.021 %
9	10.76 min	1.4400	20.0814	0.2107 min	0.148 %
10	13.04 min	0.1157	2.2071	0.2425 min	0.016 %
11	14.74 min	0.4799	11.6014	0.2900 min	0.086 %
12	17.19 min	0.2284	5.1342	0.2742 min	0.038 %
13	27.80 min	0.3967	13.3499	0.4046 min	0.099 %