BDG SYNTHESIS

# Certificate of Analysis

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

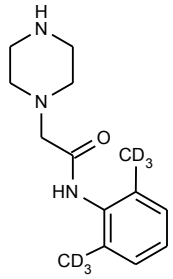
Barry Dent

Barry R. Dent, PhD, Director
5 December 2009

**Name:** **RS-94287-$d_6$**

**CAS Number:** 5294-61-1 (unlabelled)

**Structure:**




**Molecular Weight:** $C_{14}H_{15}D_6N_3O$ = 253.37

**Lot Number:** BDG 8951

**Appearance:** Off-white, crystalline solid

**Purity By HPLC:** 99.7 %

**Isotopic Purity:** Under 0.5 % $d_0$

**Re-test Date:** 5 December 2014

**Storage and Handling:**

| | |
|---|---|
| Temperature: | ambient laboratory temperature; may be refrigerated. |
| 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. |





Mailing:
BDG Synthesis
PO Box 38 627, Wellington Mail Centre,
Wellington, New Zealand.

Shipping:
BDG Synthesis
Gracefield Research Centre, Building F,
Gracefield Road, Lower Hutt, New Zealand.

Phone: + 64 4 569 0520
Fax: + 64 4 569 0521

info@bdg.co.nz
www.bdg.co.nz

## 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 absent, compared with the spectrum of unlabelled material, indicating clean deuteration.
Residual Solvents: no residual solvents are 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 site 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* 254.2138. $C_{14}H_{16}D_6N_3O$ $[M+H]^+$ requires *m/z* 254.2134. The deviation of 1.5 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 (99.7 %). 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 66.39, H 6.11, D 4.89, N 16.70 % |
| $C_{14}H_{15}D_6N_3O$ | Requires: | C 66.36, H 5.97, D 4.77, N 16.58 % |

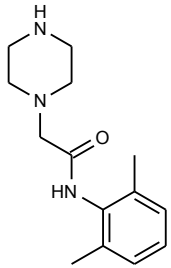
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

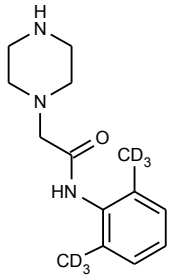
The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

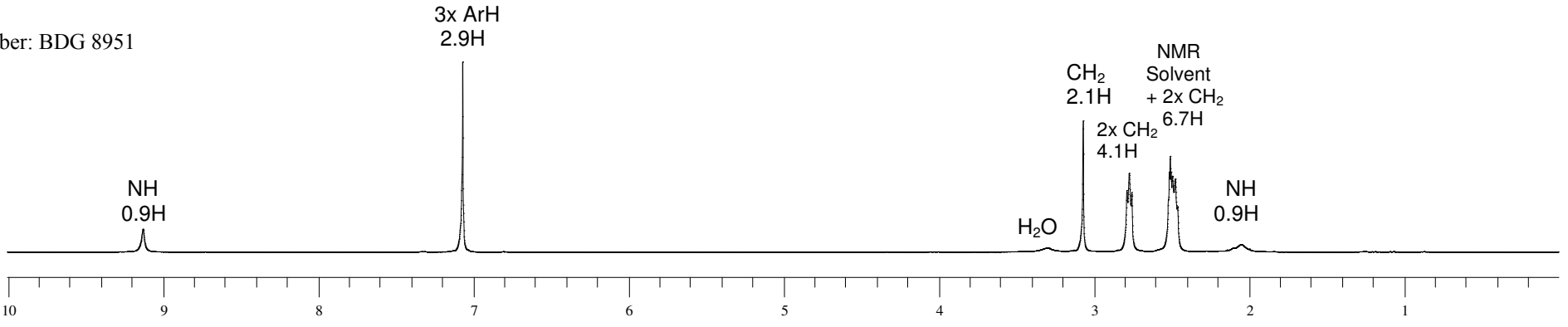
BDG SYNTHESIS

Proton NMR Spectrum of RS-94287 (top) and RS-94287-$d_6$ (bottom) in DMSO-$d_6$








Lot Number: BDG 8951

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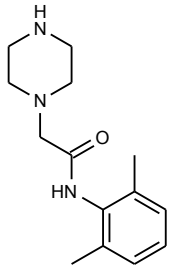
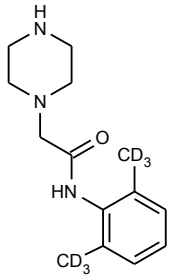
Carbon-13 NMR Spectrum of RS-94287 (top) and RS-94287-$d_6$ (bottom) in DMSO-$d_6$





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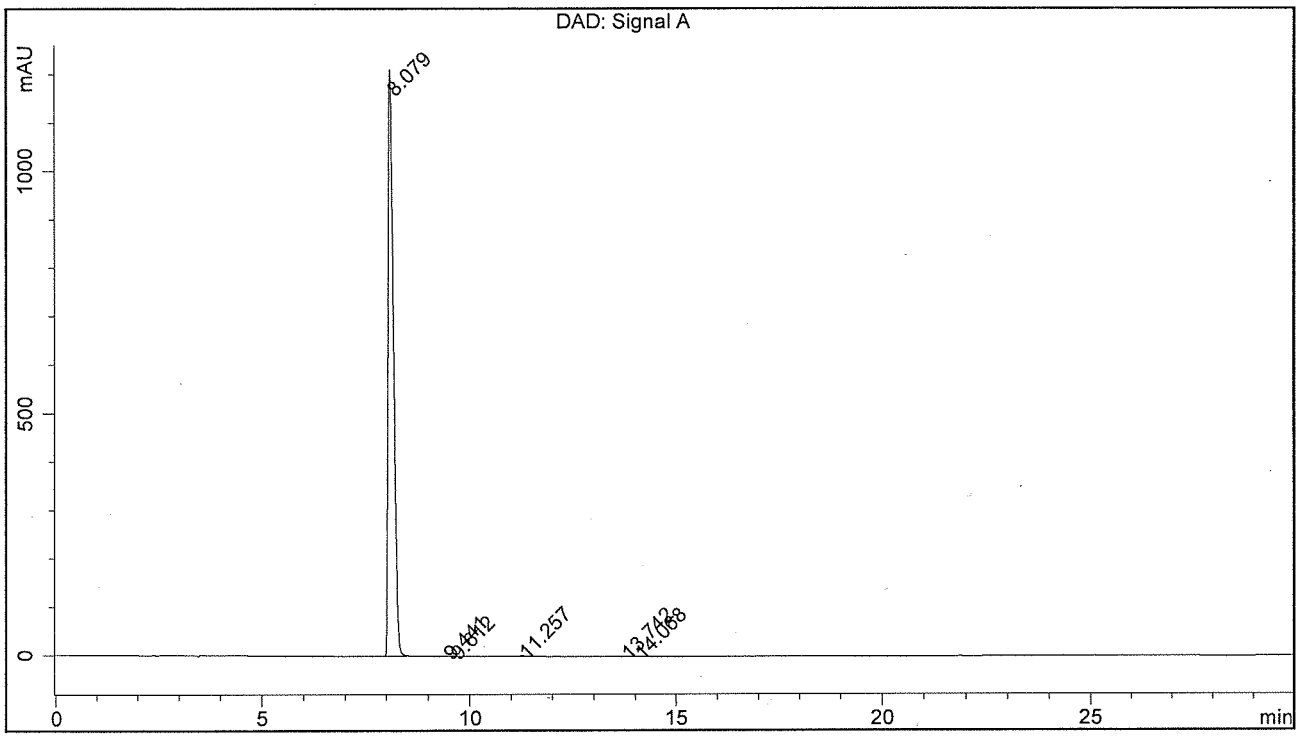
BDG - Analysis of RS-94287-d6

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase A : 25 mM Potassium diHydrogen Phosphate pH=3.0
Mobile Phase B : Acetonitrile
Gradient ( A:B ) : T0=90:10, T20=40:60, T24=40:60, T27=90:10, T30=90:10
Flow Rate : 1.0 mL/min
Sample Solvent : 4:1 Water : Acetonitrile
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV 225 nm

| Sample Name | BDG 8951 | Instrument | AnalyticalLC01 |
|---|---|---|---|
| Acquisition | 05/12/2009, 09:09:05 | Method (rev.) | LC10354c ( 2) |
| Sequence | BDG_05Dec2009a | Vial Position | 1 |
| Operator | solvation010\cerityadmin | Injection | 1 of 1 |




**Area Percent Report**

| Peak# | RT | Peak Height | Peak Area | Width | Area % |
|---|---|---|---|---|---|
| 1 | 8.08 min | 1206.8434 | 10901.1207 | 0.1414 min | 99.752 % |
| 2 | 9.44 min | 0.5161 | 4.0097 | 0.1146 min | 0.037 % |
| 3 | 9.61 min | 0.4201 | 3.1639 | 0.1119 min | 0.029 % |
| 4 | 11.26 min | 2.0953 | 11.1568 | 0.0821 min | 0.102 % |
| 5 | 13.74 min | 0.4535 | 4.5593 | 0.1393 min | 0.042 % |
| 6 | 14.07 min | 0.6752 | 4.2079 | 0.0947 min | 0.039 % |