

# **Certificate of Analysis**

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

Barry Dent

Barry R. Dent, PhD, Director 16 October 2011

Name: Hexadiline HCl

CAS Number: 3626-67-3 (free base)

**Structure:** 

N HCI

**Molecular Weight:**  $C_{19}H_{33}N\cdot HCl = 311.93$ 

**Lot Number:** BDG 14046.3

**Appearance:** White, crystalline solid

Purity By HPLC: 99.8 %

**Re-test Date:** 16 October 2012

**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: store in an amber vial and protect from bright light.

Caution: only experienced laboratory personnel should handle the material.

Avoid strongly acidic or basic conditions that may cause

isomerisation of the double bond.

Version 1 (dd399) 1/5

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### **Identity and Purity**

#### **Proton NMR Spectrum**

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

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.

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

Found m/z 276.2693.  $C_{19}H_{34}N$  [M+H]<sup>+</sup> (free base) requires m/z 276.2691. The deviation of 0.7 ppm is within normally accepted limits for the establishment of identity by HRMS.

#### **HPLC**

A somewhat broadened, slightly tailing peak is observed (99.8 %). 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 73.20, H 10.95, Cl 11.39, N 4.48 %

C<sub>19</sub>H<sub>33</sub>N·HCl Requires: C 73.16, H 10.99, Cl 11.37, N 4.49 %

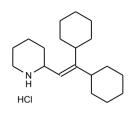
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

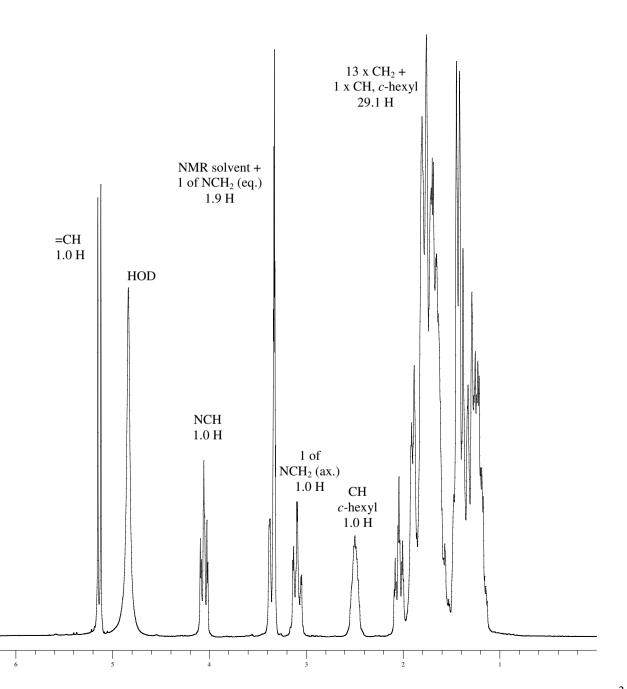
The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.



## **BDG SYNTHESIS**



Lot Number: BDG 14046.3

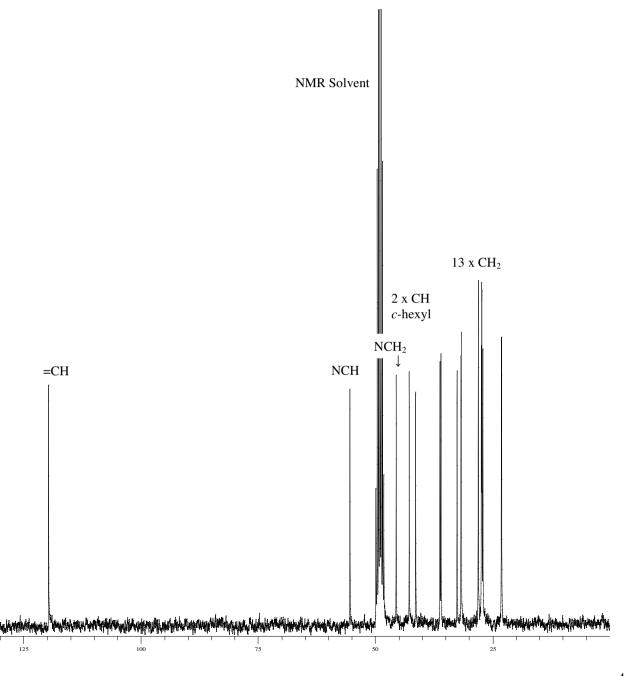


 $=CR_2$ 



# N H

Lot Number: BDG 14046.3



#### BDG - Analysis of Hexadiline HCI

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm Guard : Phenomenex Security Guard 18 RP 4 x 3 mm

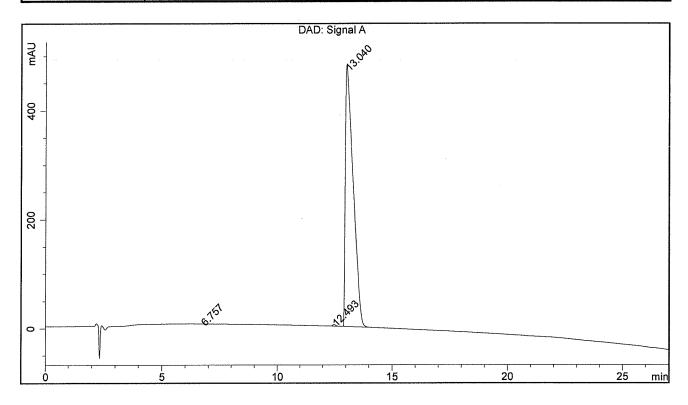
Mobile Phase A: 60:40:0.05 Water: Acetonitrile: Trifluoroacetic Acid Mobile Phase B: 20:80:0.05 Water: Acetonitrile: Trifluoroacetic Acid Gradient (A:B): T0=100:0, T25=0:100, T27=100:0, T30=100:0

Flow Rate: 1.0 mL/min

Sample Solvent : 1:1 Water : Acetonitrile

Column Temperature : 20C Injection Volume : 10 uL Detection : UV at 210 nm

Sample Name	BDG 14046.3	Instrument	AnalyticalLC01 LC10463a(5)
Acquisition	16/10/2011, 13:54:07	Method (rev.)	
Sequence	BDG_16Oct2011b - Reprocessed	Vial Position	2
Operator	solvation010\cerityadmin	Injection	2 of 2



#### **Area Percent Report**

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	6.76 min	0.2076	2.6552	0.1632 min	0.023 %
2	12.49 min	2.9258	24.7536	0.1327 min	0.218 %
3	13.04 min	482.2937	11327.7558	0.3260 min	99.759 %