BDG SYNTHESIS

Certificate of Analysis

This material is a research-grade material prepared by custom synthesis. The quantity available is limited, 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 research-grade materials. Research 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.

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

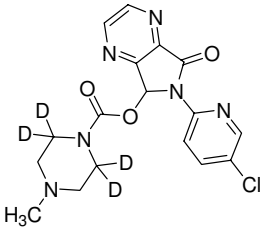
Barry Dent

Barry R. Dent, PhD, Director
7 November 2006

Name: Zopiclone-d_4

CAS Number: none (43200-80-2 unlabelled)

Structure:



Molecular Weight: $C_{17}H_{13}D_4ClN_6O_3$ = 392.84

Lot Number: BDG 6607.1

Appearance: Cream, crystalline solid

Corrected Purity: 99.6 % (HPLC) – 0.3 % (ethanol) = 99.3 %

Isotopic Purity: Under 0.5 % d_0

Expiry Date: 7 November 2011
This expiry date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage stability studies because of the small amount of material available.

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.

• Custom synthesis of analytical reference standards, metabolites, stable isotope labelled compounds • Contract research • BDG Synthesis is a division of B Dent Global Limited

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Identity and Purity:

Source of Material
The material was made by an unambiguous synthetic route, using literature procedures where possible; starting materials were purchased from reputable sources and all intermediates were checked for identity by NMR.

Proton NMR Spectrum
Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Isotopic labelling: signals at the sites of deuteration are absent, compared with what would be expected for unlabelled material, indicating clean deuteration.
Residual solvents: a small amount of ethanol (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.
Isotopic labelling: signals at the sites of deuteration have collapsed to small multiplets compared with what would be expected for unlabelled material, indicating clean deuteration.

High-resolution mass spectrum (ESI+): found *m/z* 415.1190. $C_{17}H_{13}D_4ClN_6NaO_3$ $[M+Na]^+$ requires *m/z* 415.1194. The deviation of 0.9 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 broad, slightly tailing peak is observed (99.6 area %). 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 51.67, H 3.56, D 2.14, N 21.09 %
$C_{17}H_{13}D_4ClN_6O_3$ requires: C 51.98, H 3.34, D 2.05, N 21.39 %
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).

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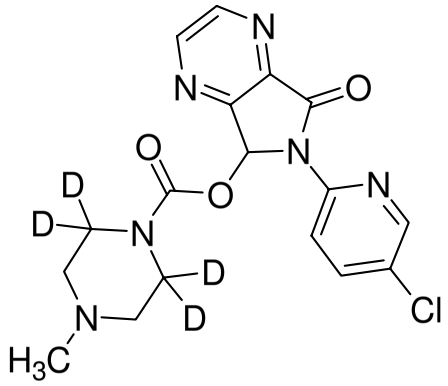
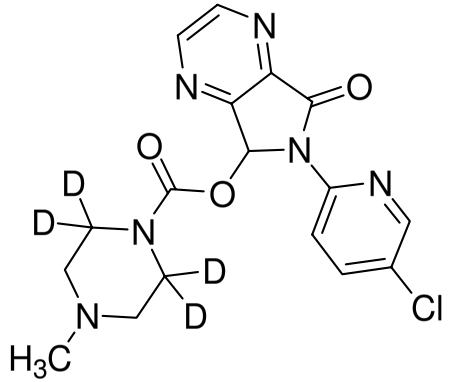


Proton NMR spectrum of Zopiclone-d_4 (BDG 6607.1)
in DMSO-d_6.

BDG SYNTHESIS

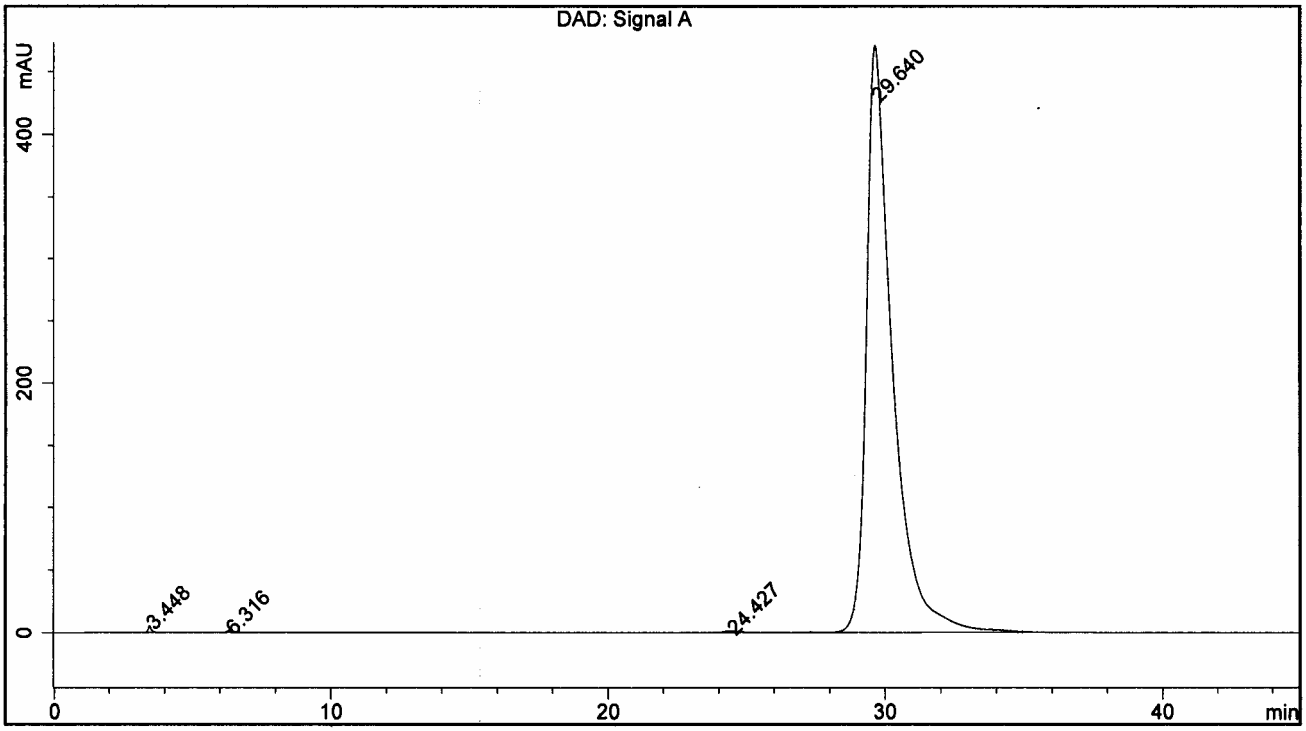


Carbon-13 NMR spectrum of Zopiclone-d_4
(BDG 6607.1) in DMSO-d_6.

BDG - Analysis of Zopiclone-d4

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 38:62 Acetonitrile : 28 mM SDS in 10 mM Sodium Dihydrogen Phosphate pH 3.5
Flow Rate : 1.5 mL/min
Sample Solvent : Mobile Phase
Column Temperature : 30C
Injection Volume : 10 uL
Detection : UV at 303 nm

Sample Name	BDG 6607.1	Instrument	AnalyticalLC01
Acquisition	06/11/2006, 19:58:33	Method (rev.)	LC10101a
Sequence	BDG_06Nov2006a - Reprocessed	Vial Position	2
Operator	LC10101a	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.45 min	5.3285	43.4715	0.1172 min	0.141 %
2	6.32 min	1.4891	26.1172	0.2566 min	0.085 %
3	24.43 min	1.2141	51.0380	0.5064 min	0.166 %
4	29.64 min	470.4080	30679.7197	0.9209 min	99.608 %