BDG SYNTHESIS

Certificate of Analysis

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

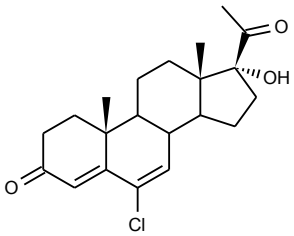
Barry Dent

Barry R. Dent, PhD, Director
5 May 2012

Name: **Chlormadinone**

CAS Number: 1961-77-9

Structure:



Molecular Weight: $C_{21}H_{27}ClO_3$ = 362.89

Lot Number: BDG 9175

Appearance: Yellow, crystalline solid

Purity By HPLC: 98.2 %

Re-test Date: 5 May 2013

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.

Mailing:
BDG Synthesis Limited
PO Box 38627, Wellington Mail Centre,
Wellington 5045, New Zealand.

Shipping:
BDG Synthesis Limited
Gracefield Research Centre, Building F,
69 Gracefield Rd, Lower Hutt 5010, 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.
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 (ESI+)

Found *m/z* 385.1549. $C_{21}H_{27}ClNaO_3$ $[M+Na]^+$ requires *m/z* 385.1546. The deviation of 0.8 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (98.2 %). 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 69.63, H 7.62 %
$C_{21}H_{27}ClO_3$	Requires:	C 69.50, H 7.50 %

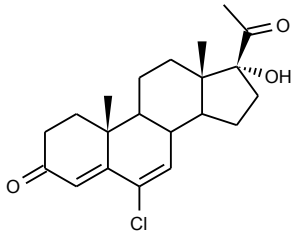
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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

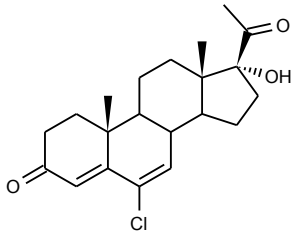
Proton NMR Spectrum of Chlormadinone in $CDCl_3$



Lot Number: BDG 9175

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Chlormadinone in $CDCl_3$



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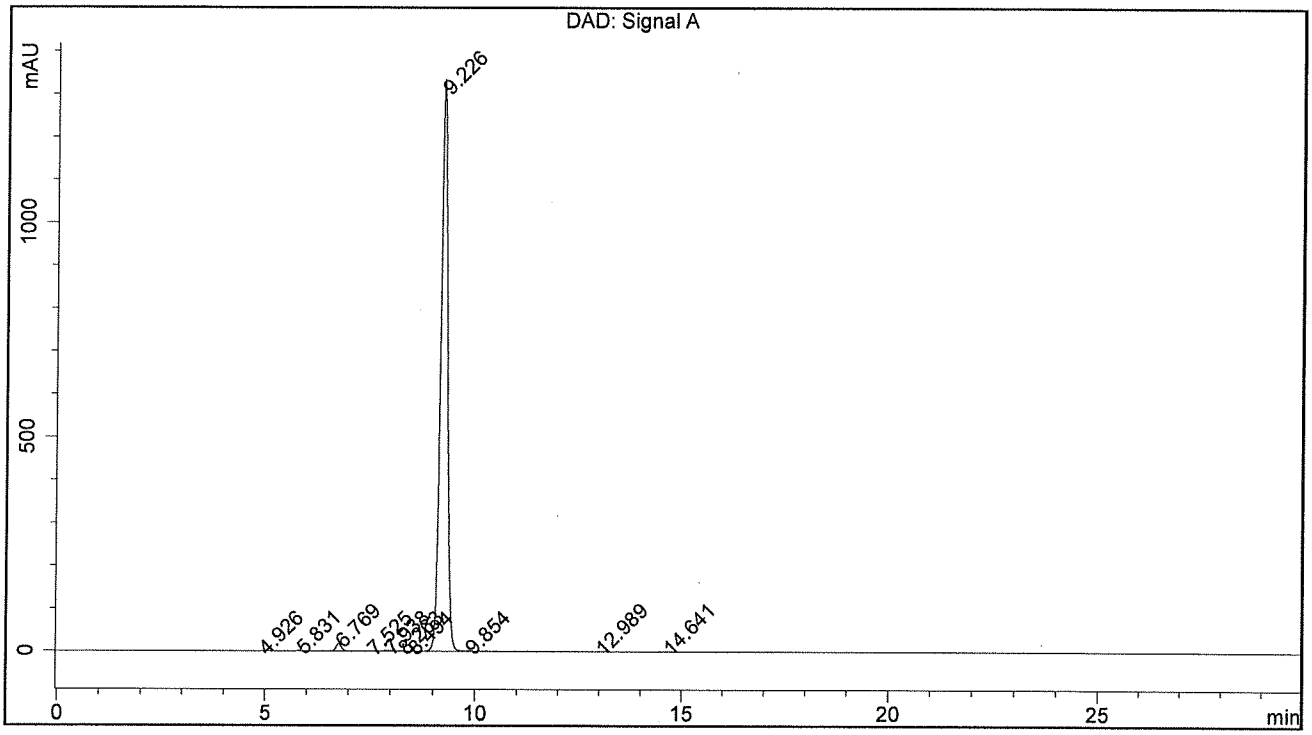
BDG - Analysis of Chlormadinone

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex SecurityGuard C18 4 x 3mm
Mobile Phase : 40:60 20 mM diPotassium Hydrogen Phosphate pH=6.0 : Acetonitrile
Flow Rate : 1.0 mL/min
Sample Solvent : Mobile Phase
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 286 nm

Sample Name	BDG 9175	Instrument	AnalyticalLC01
Acquisition	05/05/2012, 18:06:05	Method (rev.)	LC10507a (5)
Sequence	BDG_05May2012e - Reprocessed	Vial Position	54
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.93 min	0.4020	3.0860	0.1176 min	0.020 %
2	5.83 min	1.6530	15.2051	0.1393 min	0.097 %
3	6.77 min	17.6803	153.1120	0.1370 min	0.978 %
4	7.53 min	0.3318	3.5156	0.1596 min	0.022 %
5	7.94 min	2.0053	22.4502	0.1746 min	0.143 %
6	8.26 min	0.5264	5.4914	0.1617 min	0.035 %
7	8.49 min	0.3416	3.4224	0.1529 min	0.022 %
8	9.23 min	1332.9474	15372.8596	0.1826 min	98.172 %
9	9.85 min	0.6918	53.3201	0.9352 min	0.341 %
10	12.99 min	0.1046	2.0546	0.2811 min	0.013 %
11	14.64 min	0.9890	24.6460	0.3467 min	0.157 %