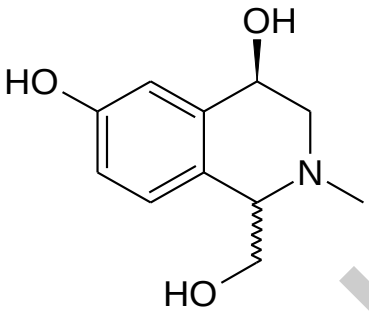
Our Formula. Your Success.

Reference Material Product Information Sheet

Epichem's Quality System conforms to ISO9001:2015 as certified by ECAAS Pty Ltd - Certification number 616061.



Name	(4*R*)-4,6-dihydroxy-1-(hydroxymethyl)-*N*-methyl-1,2,3,4-tetrahydroisoquinoline
Synonym(s)	1-(hydroxymethyl)-*N*-methyl-1,2,3,4-tetrahydroisoquinoline-4,6-diol
Epichem Item #	EPL-AA109 Batch 10
CAS #	Not available
Molecular Formula	$C_{11}H_{15}NO_3$
Molecular Weight	209.25 g/mol
Appearance	Pale amber powder
Melting Point	47.8-67.4°C
Combustion Analysis	Required (%): C:63.1; H:7.2; N:6.7. Found (%): C:60.9; H:7.6; N:6.4
Purity*	91.4%
Date of Manufacture	22 January 2020
Storage Requirements	Hygroscopic. Protect from heat, light and moisture.
Special Precautions	**This compound is for laboratory use only. Its toxicological properties may not have been fully established. It should be handled only by suitably qualified personnel.**
Intended Use	This compound is suitable for the identification of impurities and degradants in pharmaceutical materials. The purity assay is considered as relative contribution.
Date of Shipment	TBA
	This certificate is valid for one year from the date of shipment provided the substance is unopened and stored under the recommended conditions.
Retest Date	TBA (Proper Storage and Handling Required)

** NATA accreditation does not cover the performance of this service*

Epichem Pty Ltd, Suite 5, 3 Brodie-Hall Drive, Bentley WA 6102, Australia
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I. Identity

The identity of this product was established using the following analyses:

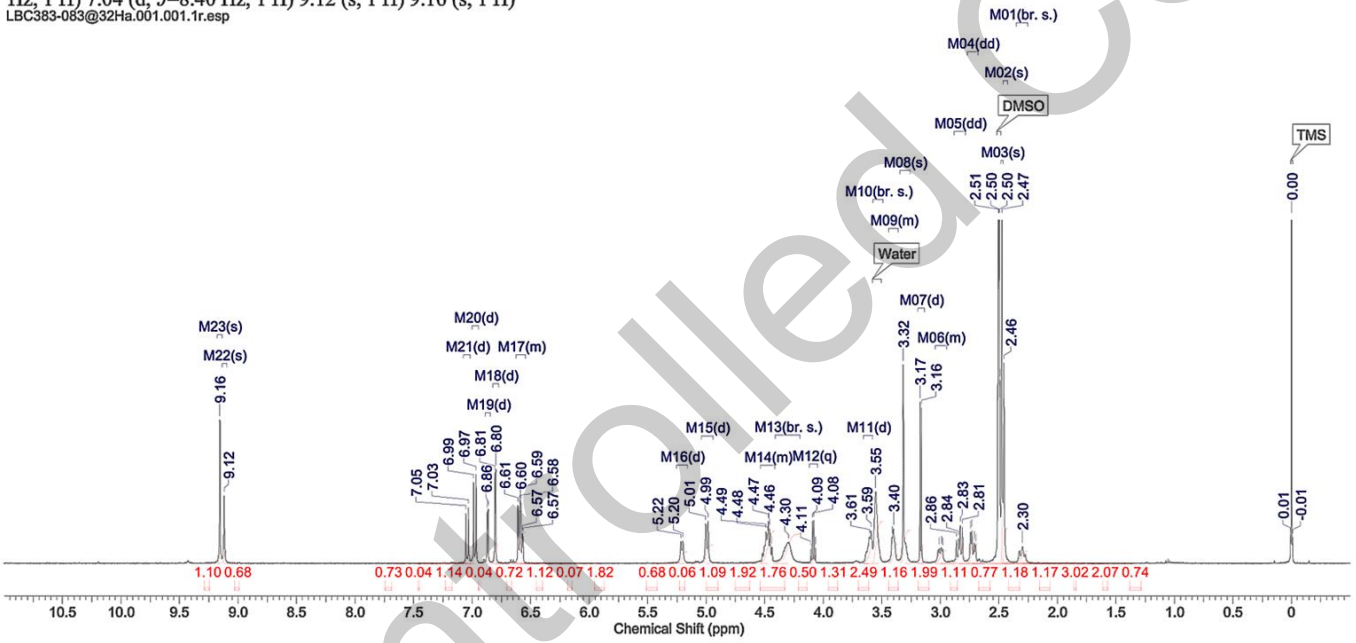
Ia. [1]HNMR Spectrum

Conditions: 400MHz, DMSO-d_6

[1]HNMR spectrum consistent with chemical structure.

Acquisition Time (sec)	3.7547	*Comment*	LBC383-083@32Ha 1H DMSO {E:\dataexternal\epichem} cygoh 18				
Date	09 Dec 2019 17:18:56			*Date Stamp*	09 Dec 2019 17:18:56		
File Name	\\naphthalene\company\NMR files\LBC383-083@32Ha\1\pdata\1\1r					*Frequency (MHz)*	400.13
Nucleus	1H	*Number of Transients*	8	*Origin*	spect	*Original Points Count*	24038
Owner	nmr	*Points Count*	32768	*Pulse Sequence*	zg	*Receiver Gain*	181.00
SW(cyclical) (Hz)	6402.05	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2797.3445	*Spectrum Type*	STANDARD
Sweep Width (Hz)	6401.85	*Temperature (degree C)*	25.429				

^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 2.30 (br. s., 1 H) 2.46 (s, 2 H) 2.47 (s, 3 H) 2.72 (dd, *J*=12.41, 4.79 Hz, 1 H) 2.84 (dd, *J*=12.21, 7.52 Hz, 1 H) 2.95 - 3.04 (m, 1 H) 3.17 (d, *J*=5.28 Hz, 1 H) 3.32 (s, 2 H) 3.36 - 3.44 (m, 1 H) 3.55 (br. s., 3 H) 3.60 (d, *J*=5.47 Hz, 1 H) 4.09 (q, *J*=5.28 Hz, 1 H) 4.30 (br. s., 2 H) 4.41 - 4.54 (m, 2 H) 5.00 (d, *J*=7.82 Hz, 1 H) 5.21 (d, *J*=6.25 Hz, 1 H) 6.55 - 6.63 (m, 2 H) 6.80 (d, *J*=2.54 Hz, 1 H) 6.87 (d, *J*=2.15 Hz, 1 H) 6.98 (d, *J*=8.40 Hz, 1 H) 7.04 (d, *J*=8.40 Hz, 1 H) 9.12 (s, 1 H) 9.16 (s, 1 H)



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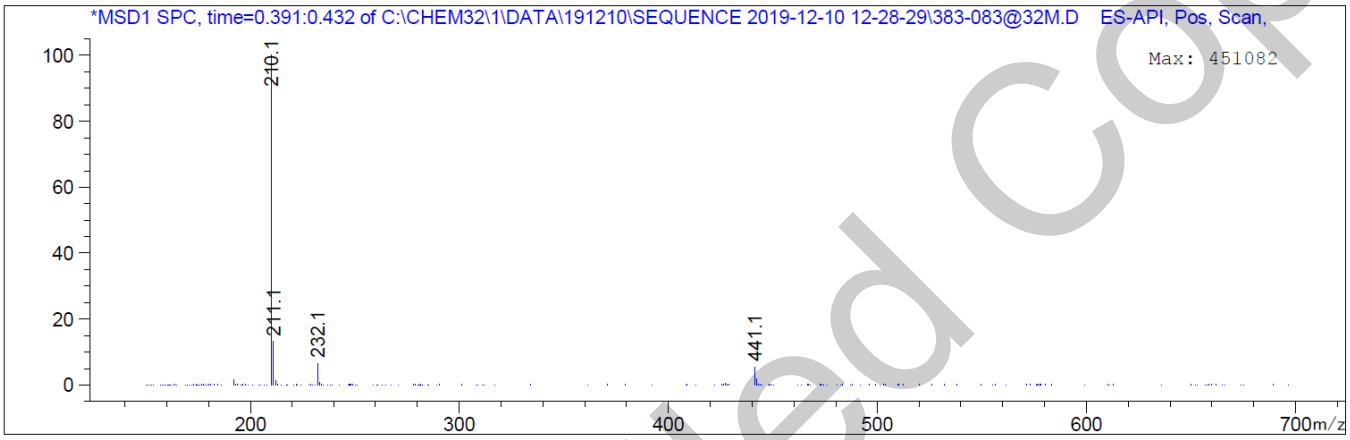
Ib. Mass Spectrum

The mass spectrum of this material was analysed by Liquid Chromatography Mass Spectroscopy (LCMS) using in-house EM005.WI08.

Method: ACN/water gradient (+ 0.1% formic acid).

ZORBAX SB-C8, 4.6 x 30 mm, 3.5 micron.

Retention Time (MS)	MS Area	Mol. Weight or Ion
0.409	3056344	211.05 I
		210.10 I



Theoretical value: 210.1 $[M+H]^+$.

The signal of the Mass Spectrum is consistent with the theoretical value and its interpretation is consistent with the structural formula.

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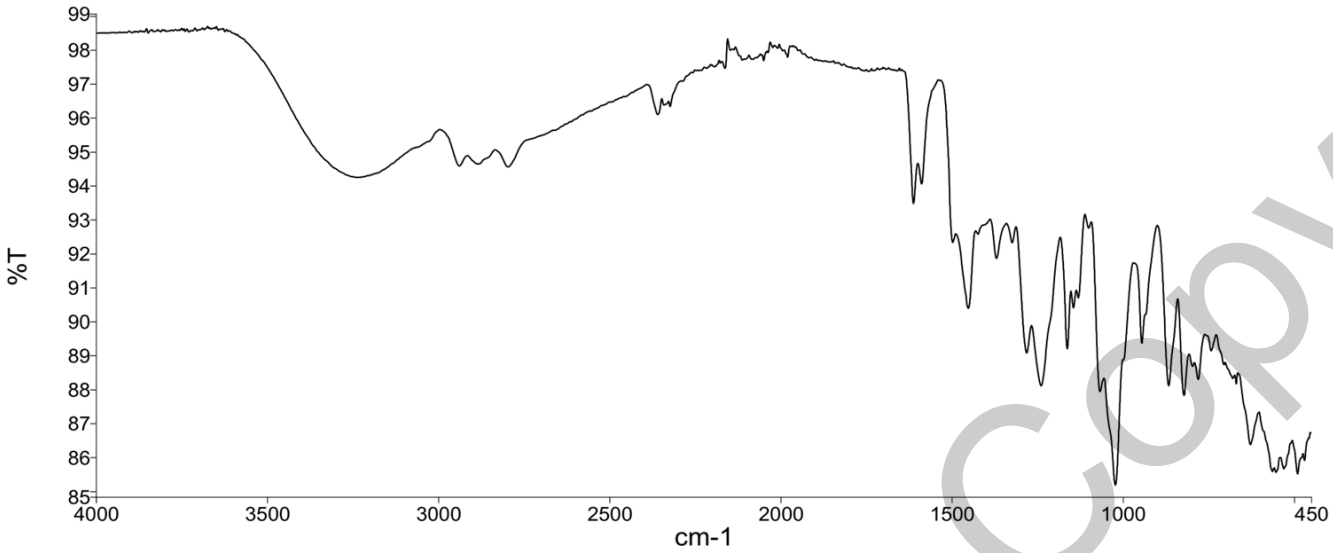
Ic. IR Spectrum

The infra-red spectrum of this material was analysed by Fourier-Transform Infra-red Spectroscopy (FTIR) using in-house EM005.WI09.



The interpretation of the signals of the Fourier Transform Infra-red Spectrum is consistent with the structural formula.

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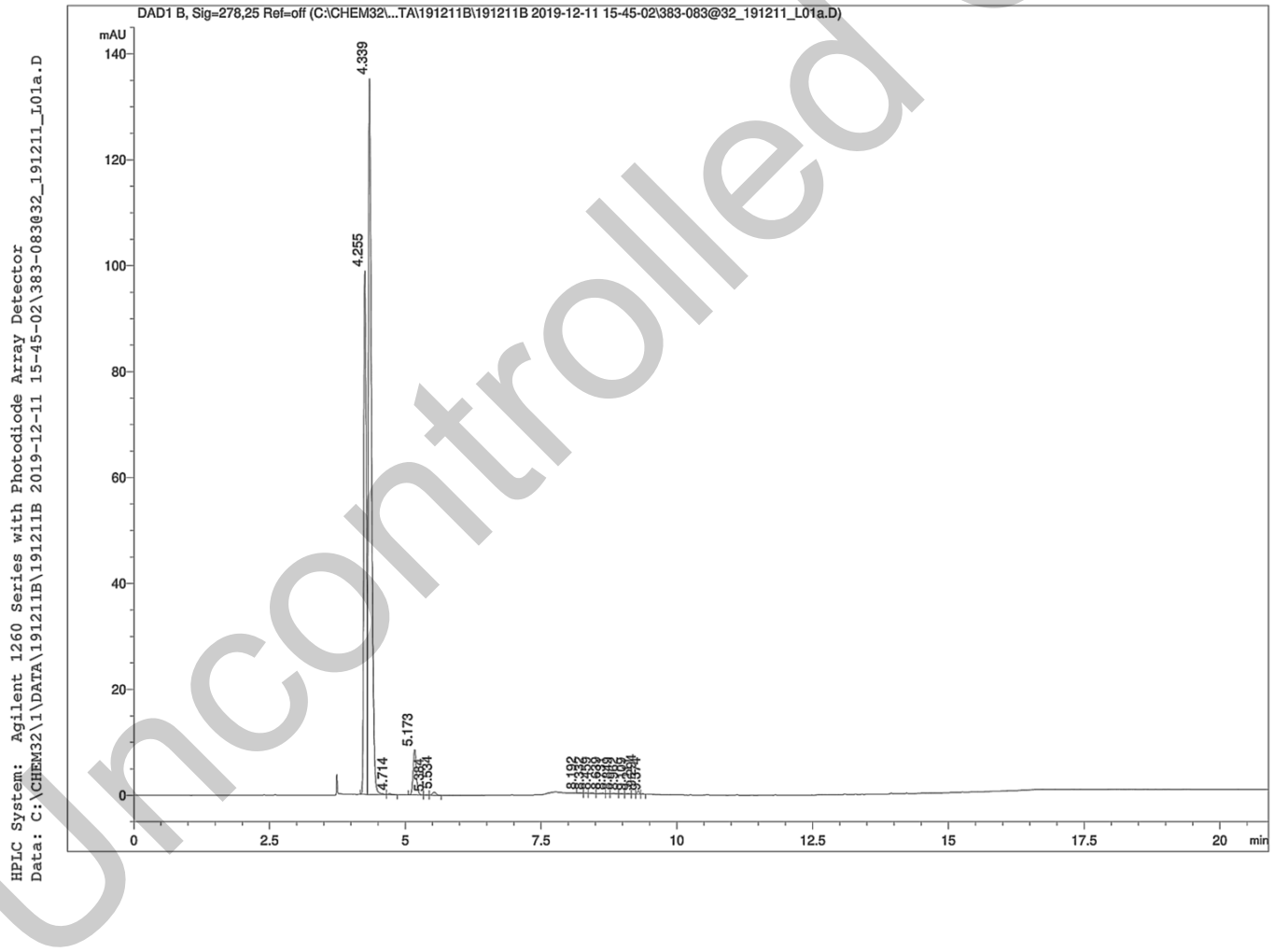
II. Purity

The purity of this material was analysed by high performance liquid chromatography (HPLC) using in-house EM005.WI07.

HPLC Conditions:

Column	Conditions				Detector	Injector
Fortis H2o 4.6 x 100mm 3 micron	25ºC				DAD 278nm	Auto 2.0 µL 1.4 mg/mL in water + 0.1% (v/v) TFA
	Time (min)	% Line A (Water + 0.1% (v/v) TFA)	% Line B (Acetonitrile + 0.1% (v/v) TFA)	Flow rate (mL/min)		
	0.00	99.5	0.5	1.0		
	5.00	97	3	1.0		
	14.20	5	95	1.0		
	19.20	5	95	1.0		
	20.20	100	0.5	1.0		
	26.20	100	0.5	1.0		



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Area Percent Report – Sorted by Signal

Peak Number	Retention Time (rounded)	Area	Area % (rounded)
1	4.25	318.19	34.32
2	4.34	565.35	60.98
3	4.71	0.87	0.09
4	5.17	35.99	3.88
5	5.38	0.26	0.03
6	5.53	2.70	0.29
7	8.19	0.19	0.02
8	8.33	0.10	0.01
9	8.46	0.27	0.03
10	8.64	0.34	0.04
11	8.73	0.16	0.02
12	8.85	0.51	0.05
13	8.96	0.20	0.02
14	9.11	0.14	0.01
15	9.21	0.06	0.01
16	9.29	1.61	0.17
17	9.37	0.19	0.02
Totals			100 (rounded)

For the calculation the system peaks were ignored. The content of the analyte was determined as a ratio of the peak area of the analyte and the cumulative areas of the purities, added up to 100%.

Results:

Average 95.3% (average of 10 duplicate analyses)

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III. Water Content

Method: Karl-Fischer titration using in-house EM005.WI04.

Results:

Average 0.5%

IV. Ash Content

Method: Combustion adjuvant added

Result:

Contains 1.0% ash.

V. Residual Solvents

Method: ^{1}HNMR

Result:

2.6% Methanol by ^{1}H NMR analysis.

VI. Final Result

Chromatographic purity (HPLC)	95.3%
Water content	0.5%
Ash content	1.0%
Residual solvents	2.6%
Purity*	91.4%

This purity is assessed to be 91.4%.

Product Reviewed By:

John Moursounidis, PhD
Head Reference Standards

Product Released By:

Boon Tan
Quality Manager

Release Date: 22 January 2020

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The calculation of the purity follows the formula:

$$\text{Purity}(\%) = \frac{((\text{Chromatographicpurity[HPLC]})x(100 - (\text{watercontent} + \text{ashcontent} + \text{volatilecontents})))}{100}$$

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