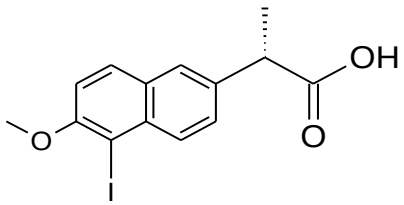
Our Formula. Your Success.

Reference Material Product Information Sheet

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



Name	(2*S*)-2-(5-iodo-6-methoxynaphthalen-2-yl)propanoic acid
BP Name	Naproxen Impurity D
Synonym(s)	5-Iodonaproxen
Epichem Item #	EPL-AA185 Batch 1
CAS #	116883-62-6
Molecular Formula	$C_{14}H_{13}IO_3$
Molecular Weight	356.16 g/mol
Appearance	Yellow crystalline powder
Melting Point	186.0-192.6°C
Combustion Analysis	Required (%): C:47.2; H:3.7; N:0.0. Found (%): C:47.3; H:3.5; N:0.0.
Purity*	99.2%
Optical Purity*	99.7% by HPLC; $[\alpha]_D^{25.9}$ +41 (*c*1.00, $CHCl_3$)
Date of Manufacture	23 October 2015
Storage Requirements	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 stored under the recommended conditions.
Retest Date	TBA (Proper Storage and Handling Required)

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

EPL-AA185 Batch 1

I. Identity

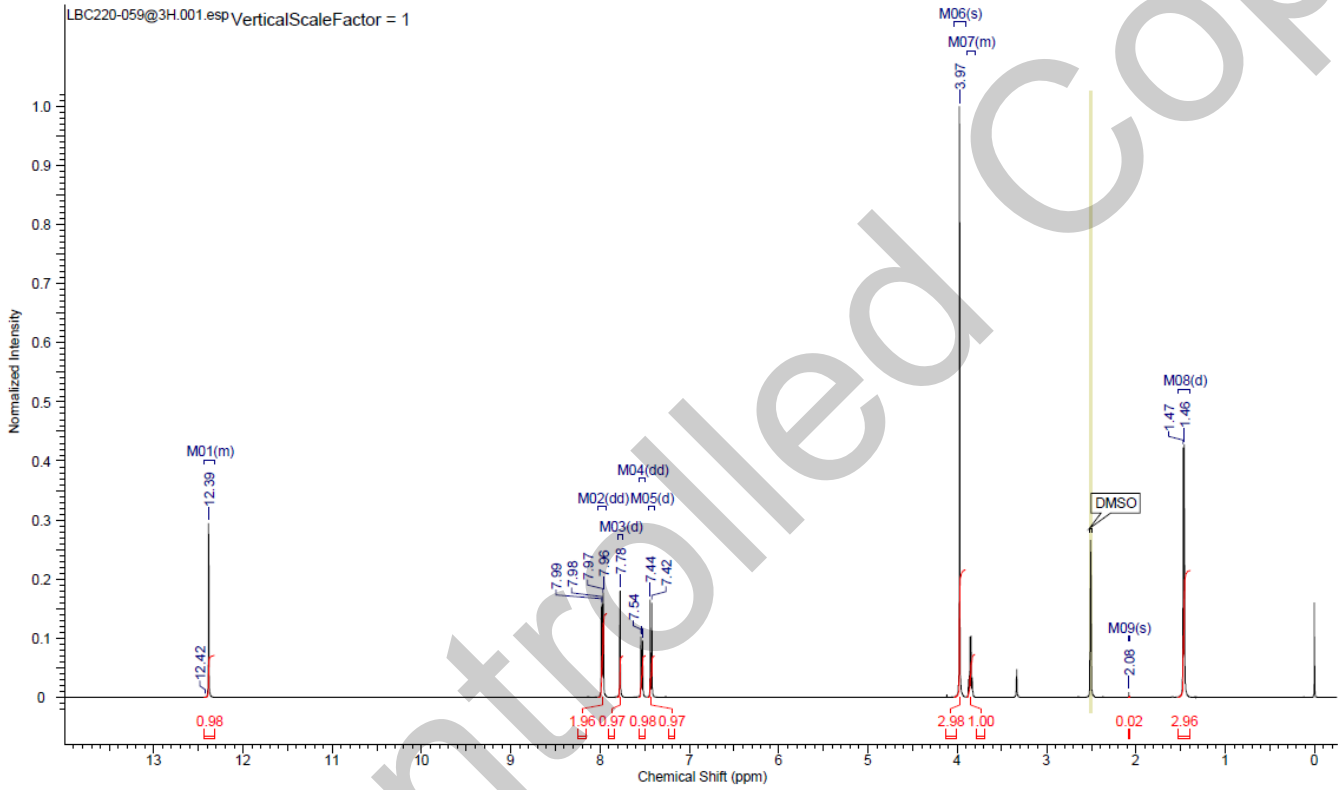
The identity of this product was established using the following analyses:

Ia. ^{1}HNMR Spectrum

Conditions: 400 MHz, DMSO-d_6

^{1}HNMR spectrum consistent with chemical structure.

Acquisition Time (sec)	3.2768	*Date*	14 Oct 2015 14:04:48		*Date Stamp*	14 Oct 2015 14:04:48	
File Name	\\NAPHTHALENE\Company\UWA NMR\LBC220-059@3H\1\fid				*Frequency (MHz)*	500.10	*Nucleus* 1H
Number of Transients	16	*Origin*	spect	*Original Points Count* 32768	*Owner*	Epichem	*Points Count* 32768
Pulse Sequence	zg30	*Receiver Gain*	121.41	*SW(cyclical) (Hz)* 10000.00	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)* 3086.1375
Spectrum Type	STANDARD	*Sweep Width (Hz)*	9999.70	*Temperature (degree C)* 24.911			



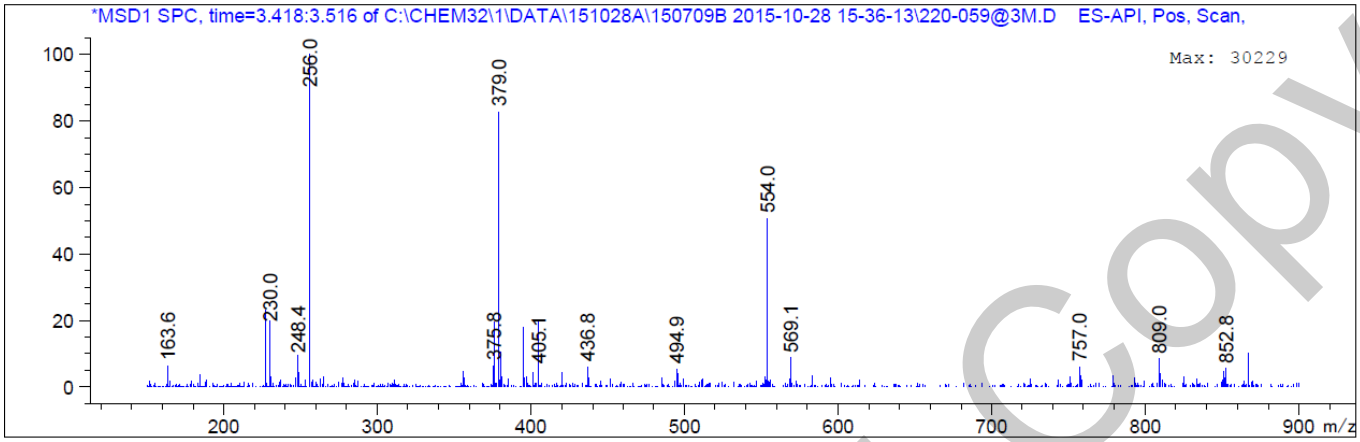
EPL-AA185 Batch 1

Ib. Mass Spectrum

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

Method: 5% to 100% ACN in water gradient (+0.1% formic acid)

Poroshell 120 EC-C18, 4.6 x 50mm, 2.7 micron



Theoretical value: 379.0 $[M+Na]^+$.

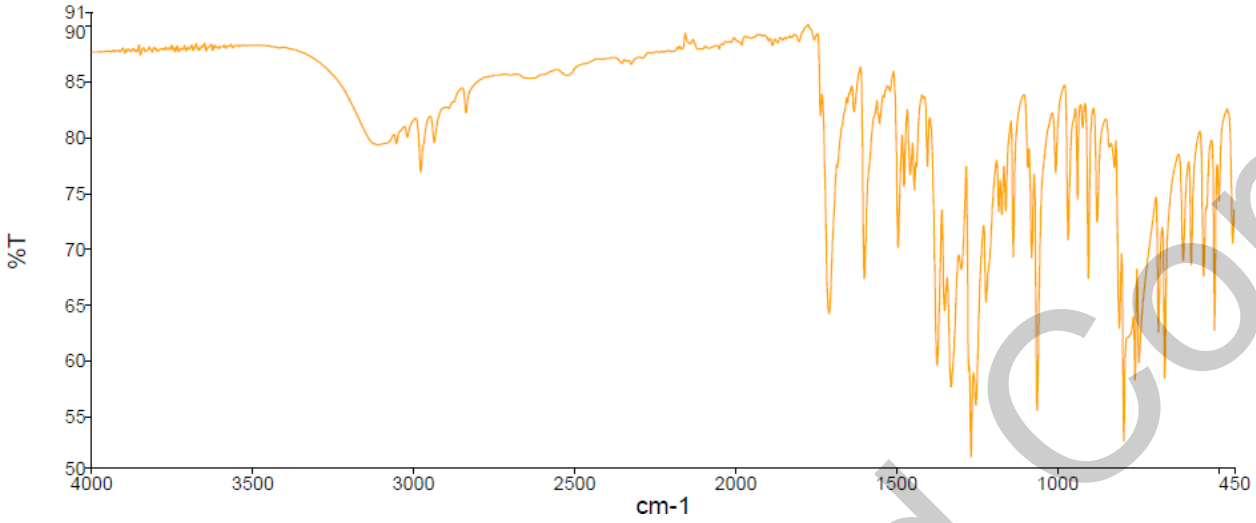
The signal of the Mass Spectrum is consistent with the theoretical value and its interpretation is consistent with the structural formula.

Epichem Pty Ltd, Suite 5, 3 Brodie-Hall Drive, Bentley WA 6102, Australia
Tel + 61 (0)8 6167 5200 Fax + 61 (0)8 6167 5201 www.epichem.com.au ABN 80 106 769 902

Ic. IR Spectrum

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

Method: Fourier Transform Infrared (FTIR) Spectroscopy



The signals of the IR spectrum and their interpretation are consistent with the structural formula.

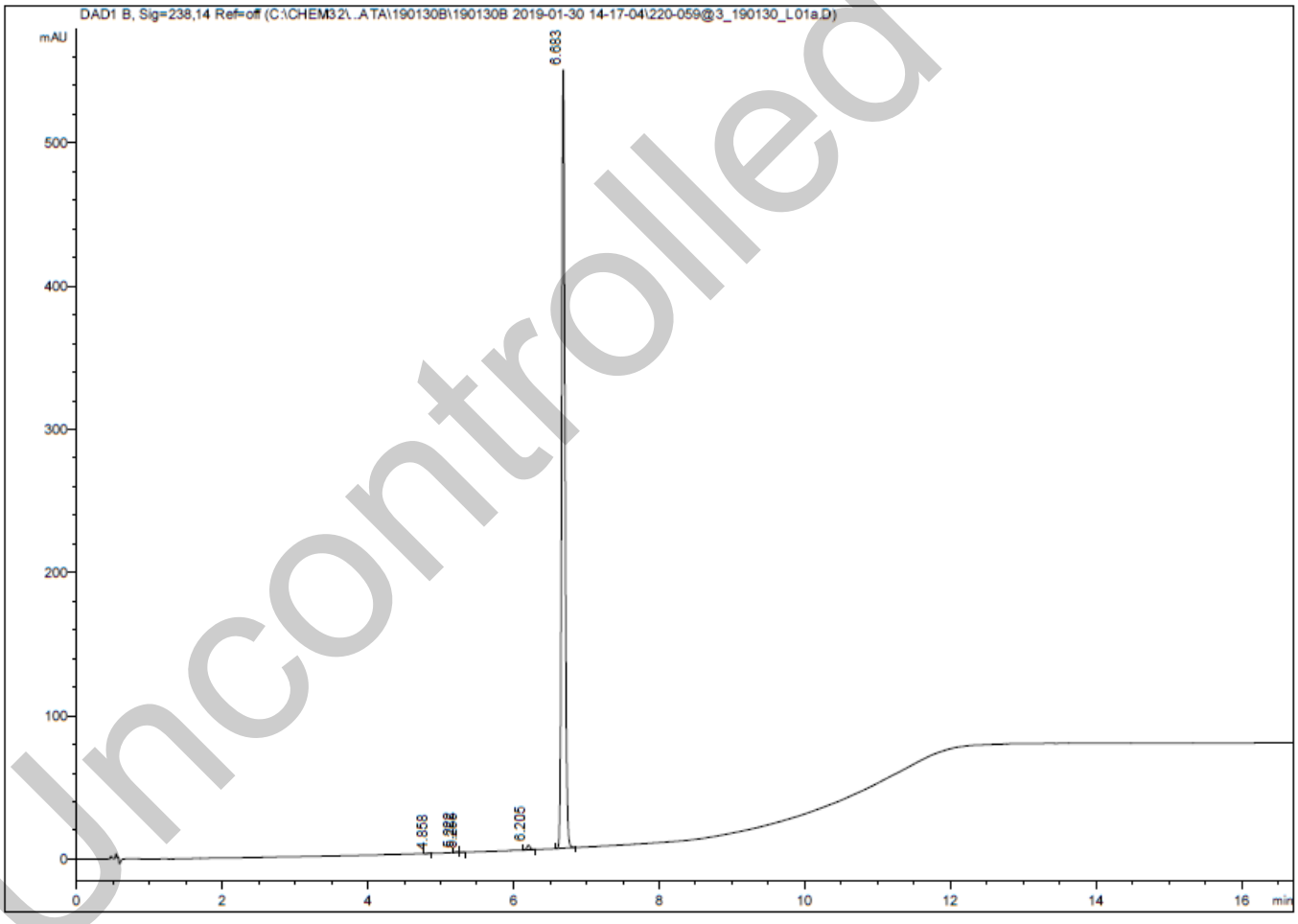
Epichem Pty Ltd, Suite 5, 3 Brodie-Hall Drive, Bentley WA 6102, Australia
Tel + 61 (0)8 6167 5200 Fax + 61 (0)8 6167 5201 www.epichem.com.au ABN 80 106 769 902

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
Agilent Poroshell 120 EC-C18 4.6 x 50mm 2.7 micron	25ºC				DAD 238nm	Auto 1.0 µL 0.20 mg/mL in 100% acetonitrile
	Time (min)	% Line A (Water + 0.1% (v/v) TFA)	% Line B (Acetonitrile + 0.1% (v/v) TFA)	Flow rate (mL/min)		
	0.00	75	25	1.0		
	7.00	40	60	1.0		
	10.50	5	95	1.0		
	15.50	5	95	1.0		
	16.50	75	25	1.0		
	19.50	75	25	1.0		



EPL-AA185 Batch 1

Area Percent Report – Sorted by Signal

Peak Number	Retention Time (rounded)	Area	Area % (rounded)
1	4.86	0.21	0.01
2	5.22	2.10	0.12
3	5.25	0.70	0.04
4	6.21	10.18	0.58
5	6.68	1731.82	99.24
Totals			100.0

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 99.3% (average of 10 duplicate runs)

Epichem Pty Ltd, Suite 5, 3 Brodie-Hall Drive, Bentley WA 6102, Australia
Tel + 61 (0)8 6167 5200 Fax + 61 (0)8 6167 5201 www.epichem.com.au ABN 80 106 769 902

III. Water Content

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

Results:

Average 0.2%

IV. Ash Content

Method: BP2015 Ash (Appendix XI-J) WS001/26829

Result:

Contains <0.1% ash.

V. Residual Solvents

Method: ^{1}HNMR

Result:

Contains: acetonitrile <0/1%

VI. Final Result

Chromatographic purity (HPLC)	99.3%
Water content	0.1%
Ash content	<0.1%
Residual solvents	<0.1%
Purity*	99.2%

This purity is assessed to be 99.2%.

Product Reviewed By:

John Moursounidis, PhD
Head Reference Standards

Product Released By:

Boon Tan
Quality Manager

Release Date: 8 February 2019

**NATA accreditation does not cover the performance of this service.*
The calculation of the purity follows the formula:

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

EPL-AA185 Batch 1