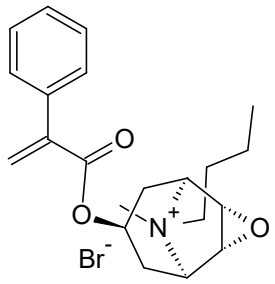
Our Formula. Your Success.

Reference Material Product Information Sheet

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



Name	(1α,2β,4β,5α,7β)-9-butyl-9-methyl-7-((1-oxo-2-phenyl-2-propenyl)oxy)-3-oxa-9-azoniatricyclo[3.3.1.$0^{2,4}$]nonane bromide
BP/EP Name	Hyoscine Butylbromide Impurity G
USP Name	Not listed.
Synonym(s)	Apohyoscine butylbromide; Aposcopolamine butylbromide; Apobuscopan
Epichem Item #	EPL-AA238 Batch 5
CAS #	92714-23-3
Molecular Formula	$C_{21}H_{28}NO_3$.Br
Molecular Weight	422.37 g/mol
Appearance	Off-white powder
Melting Point	63.7-89.0°C
Combustion Analysis	Required (%):C:59.7, H:6.7, N:3.3. Found (%):C:52.4, H:6.4, N:3.0.
Purity	97.2%
Date of Manufacture	7 February 2022
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 stored under the recommended conditions.
Retest Date	TBA (Proper Storage and Handling Required)

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:

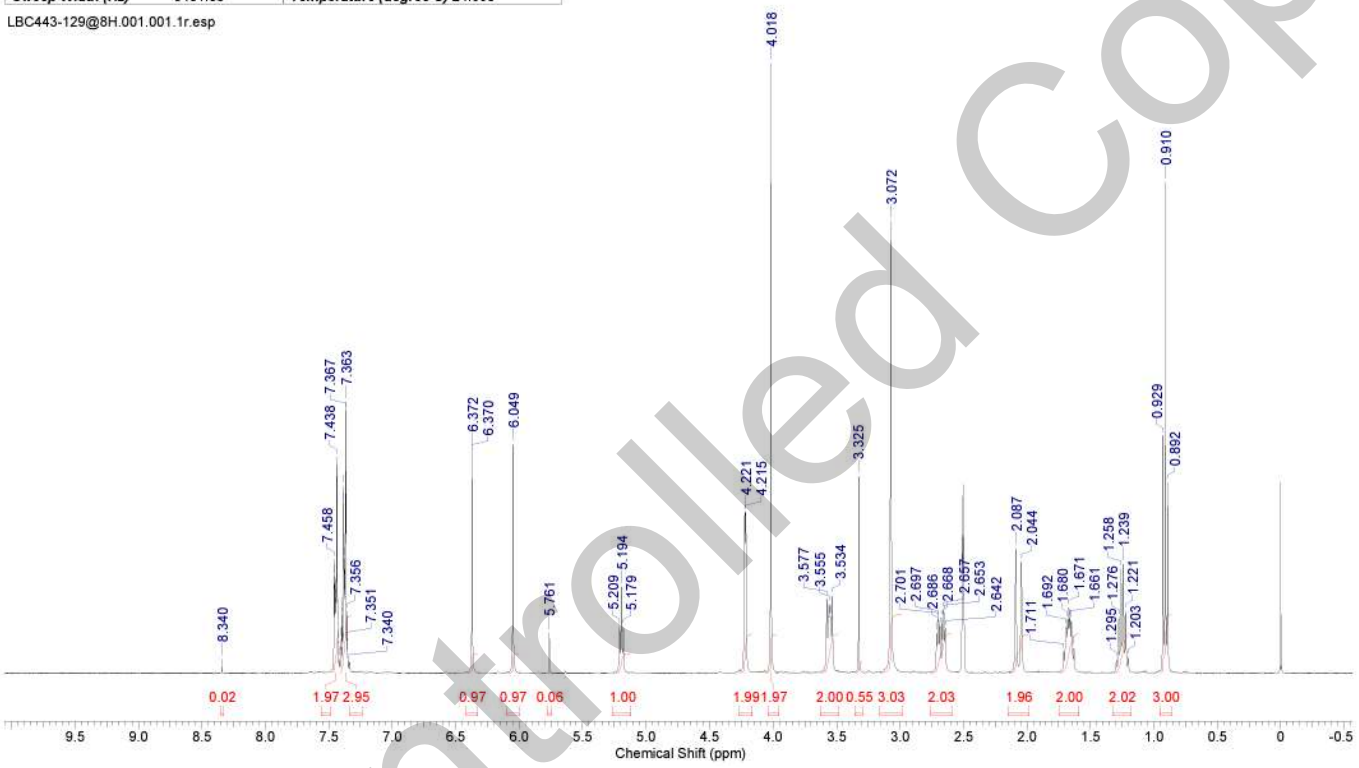
Ia. [1]HNMR Spectrum

Conditions: 400 MHz, DMSO-d_6

[1]HNMR spectrum consistent with chemical structure.

Acquisition Time (sec)	3.7547	***Comment***	LBC443-129@8H 1H DMSO {E:\dataexternal\epichem} cygoh 2				
Date	22 Mar 2023 08:29:52			***Date Stamp***	22 Mar 2023 08:29:52		
File Name	\\naphthalene\company\015 NMR files\LBC443-129@8H\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***	114.00
SW(cyclical) (Hz)	6402.05	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	2799.3291	***Spectrum Type***	STANDARD
Sweep Width (Hz)	6401.85	***Temperature (degree C)***	24.996				

LBC443-129@8H.001.001.1r.esp



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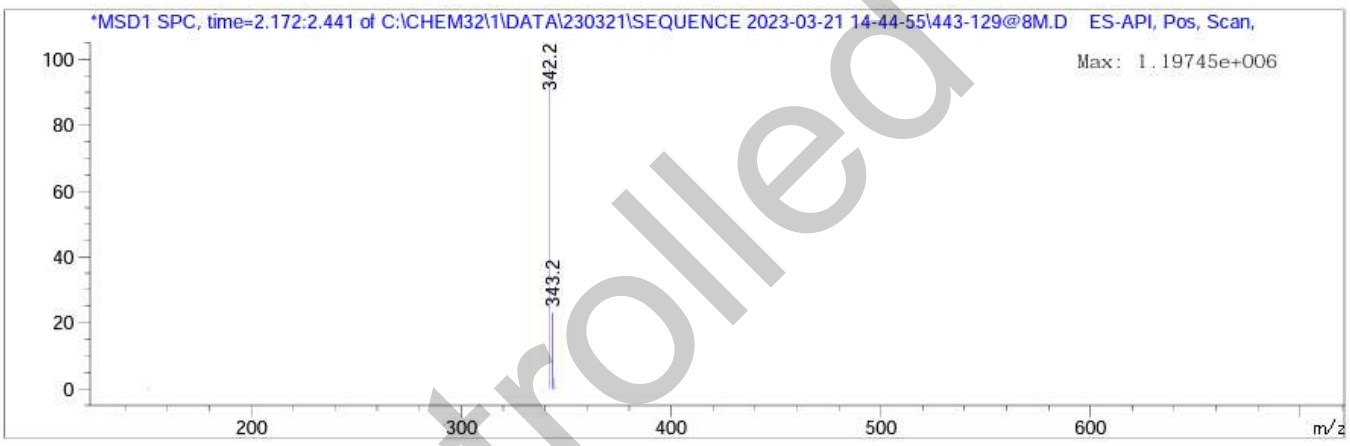
Ib. Mass Spectrum

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
2.203	28649452	343.20 I
		342.20 I
2.751	740777	460.05 I
		459.15 I
		458.00 I
		456.10 I
		356.20 I
		343.20 I
		342.20 I



Theoretical values: 342.2 $[M]^+$.

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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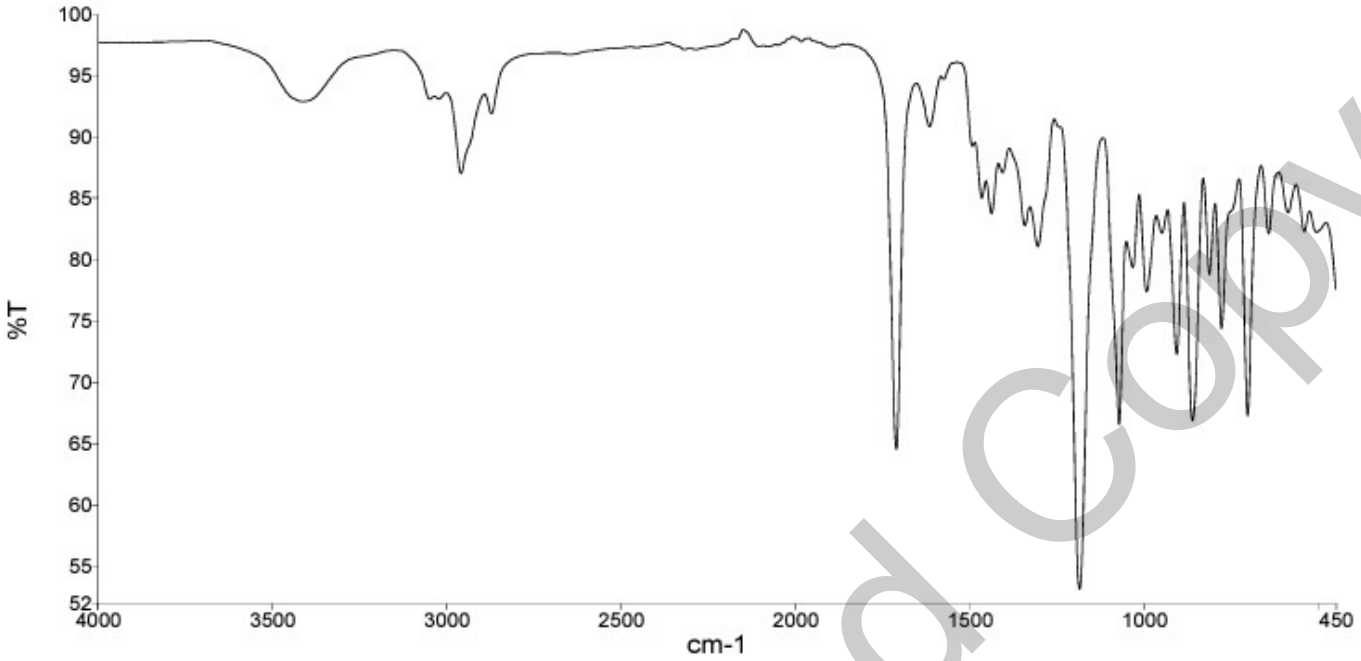
Ic. IR Spectrum

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



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

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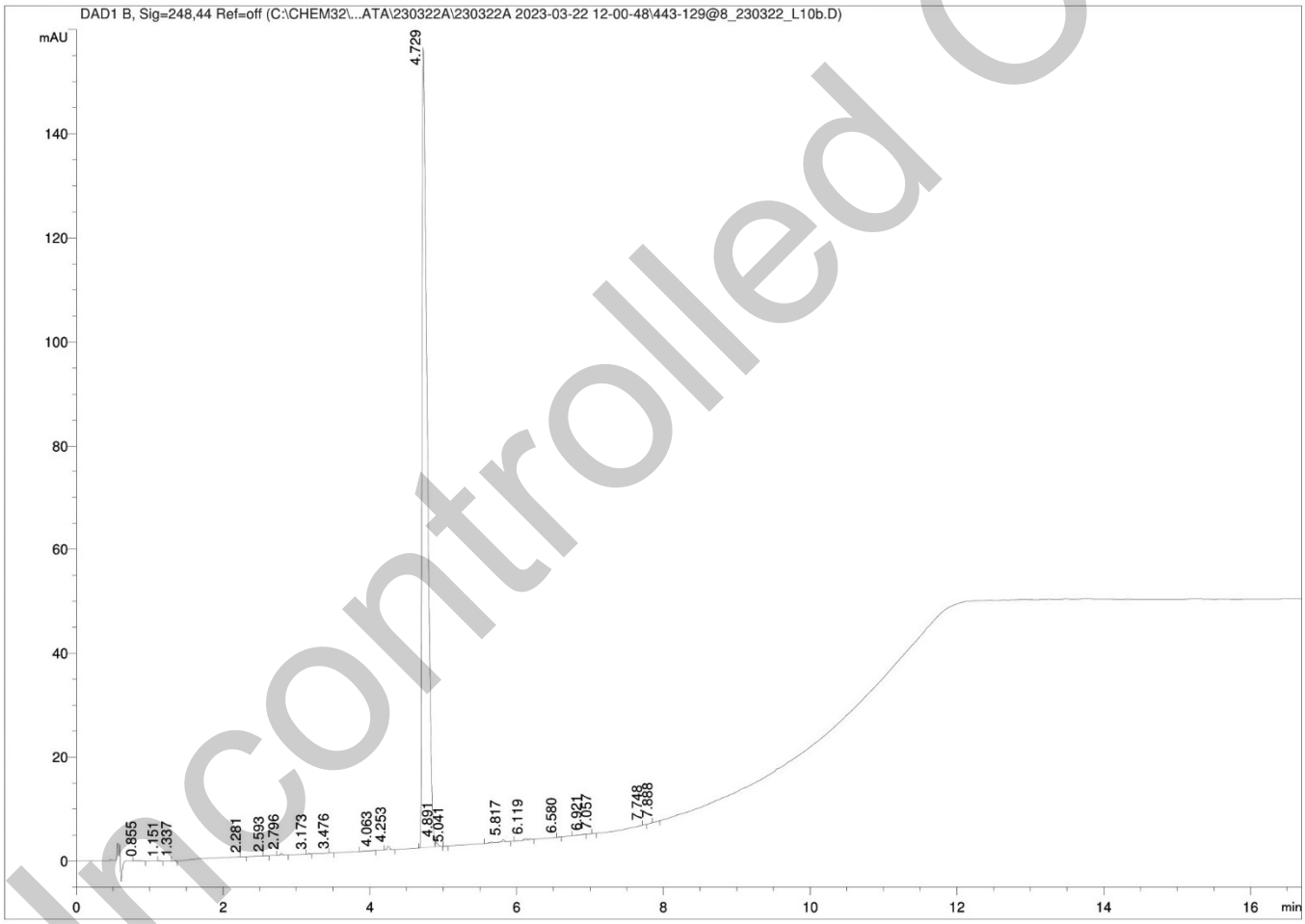
II. Purity

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

HPLC Conditions:

Column	Conditions				Detector	Injector
Agilent Poroshell 120 EC-C18 4.6 x 50mm 2.7 micron	25ºC				DAD 248nm	Auto 1.0 µL 1.8 mg/mL in 100% acetonitrile (NO MODIFIERS)
	Time (min)	% Line A (Water + 0.1% (v/v) TFA)	% Line B (Acetonitrile + 0.1% (v/v) TFA)	Flow rate (mL/min)		
	0.00	80	20	1.0		
	6.00	50	50	1.0		
	10.50	5	95	1.0		
	15.50	5	95	1.0		
	16.50	80	20	1.0		
	19.50	80	20	1.0		



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Area Percent Report – Sorted by Signal

Peak Number	Retention Time (rounded)	Area	Area % (rounded)
1	0.85	0.46	0.06
2	1.15	0.12	0.02
3	1.34	0.24	0.03
4	2.28	0.08	0.01
5	2.59	0.09	0.01
6	2.80	0.84	0.11
7	3.17	0.21	0.03
8	3.48	0.10	0.01
9	4.06	0.34	0.04
10	4.25	1.86	0.24
11	4.73	755.25	98.54
12	4.89	2.09	0.27
13	5.04	0.09	0.01
14	5.82	2.26	0.30
15	6.12	1.38	0.18
16	6.58	0.08	0.01
17	6.92	0.60	0.08
18	7.06	0.11	0.01
19	7.75	0.08	0.01
20	7.89	0.12	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 98.5% (average of 10 duplicate runs)

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III. Water Content

Method: Karl-Fischer titration using in-house EM005.WI04.
Results:
Average 0.4%

IV. Ash Content

Method: Gravimetric determination of total ash by combustion in accordance with Ph. Eur. 2.4.16
Result:
Contains 0.3% ash.

V. Residual Solvents

Method: ^{1}HNMR
Result:
0.6% Dichloromethane by ^{1}H NMR analysis.

VI. Final Result

Chromatographic purity (HPLC)	98.5%
Water content	0.4%
Ash content	0.3%
Residual solvents	0.6%
Purity	97.2%

This purity is assessed to be 97.2%

Product Reviewed By:

Jacob Heppell, PhD
Chemist

Product Released By:

Carol Worth, PhD
Quality Manager

Release Date: 31 March 2023

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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