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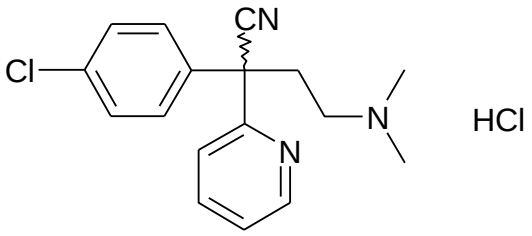
# Reference Material Product Information Sheet

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




| | |
|---|---|
| **Name** | (2*RS*)-2-(4-chlorophenyl)-4-(dimethylamino)-2-(pyridin-2-yl)butanenitrile hydrochloride |
| **USP Name** | Chlorpheniramine Nitrile |
| **BP Name** | Chlorphenamine Impurity D hydrochloride |
| **Synonym(s)** | Chlorpheniramine Impurity D hydrochloride; (2*RS*)-2-(4-chlorophenyl)-4-(dimethylamino)-2-(pyridin-2-yl)butyronitrile hydrochloride; Chlorpheniramine Nitrile hydrochloride |
| **Epichem Item #** | EPL-AA17 Batch 7 |
| **CAS #** | 65676-21-3 (free base) |
| **Molecular Formula** | $C_{17}H_{19}Cl_2N_3$ |
| **Molecular Weight** | 336.27 g/mol |
| **Appearance** | Off-white powder |
| **Melting Point** | 194.0-197.7°C |
| **Combustion Analysis** | Required (%): C:60.7; H:5.7; N:12.5. Found (%): C:60.5; H:5.6; N:12.4. |
| **Purity*** | 99.5% |
| **Date of Manufacture** | 29 April 2019 |
| **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 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*





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## I. Identity

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

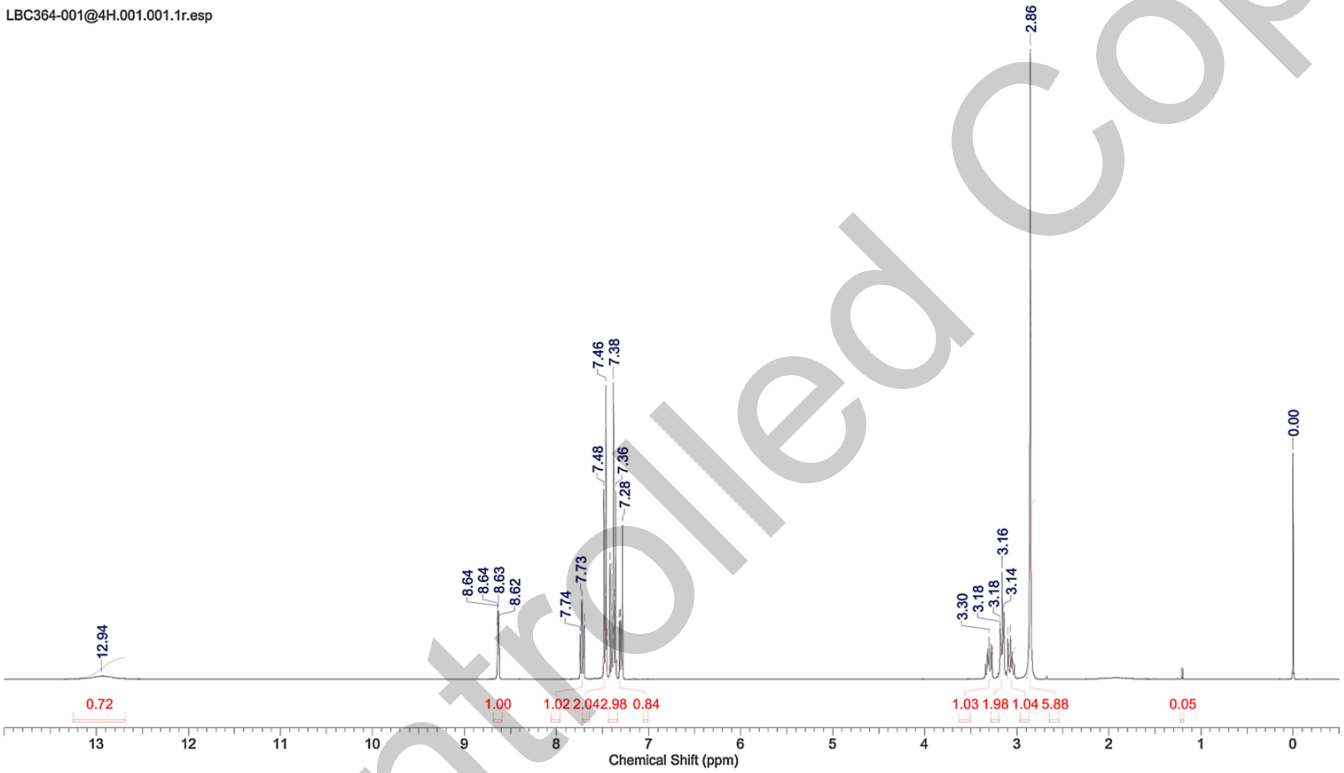
### Ia. $^{1}$HNMR Spectrum

Conditions: 400 MHz, DMSO-$d_6$

$^{1}$HNMR spectrum consistent with chemical structure.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| *Acquisition Time (sec)* | 3.7547 | *Comment* | LBC364-001@4H 1H CDCl3 {E:\dataexternal\epichem} cygoh 5 | | | *Date* | 15 Apr 2019 17:38:08 |
| *Date Stamp* | 15 Apr 2019 17:38:08 | | | *File Name* | \\naphthalene\company\NMR files\LBC364\LBC364-001@4H\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* | 90.50 | *SW(cyclical) (Hz)* | 6402.05 | *Solvent* | CHLOROFORM-d | | |
| *Spectrum Offset (Hz)* | 2800.0854 | *Spectrum Type* | STANDARD | *Sweep Width (Hz)* | 6401.85 | *Temperature (degree C)* | 26.945 |

LBC364-001@4H.001.001.1r.esp





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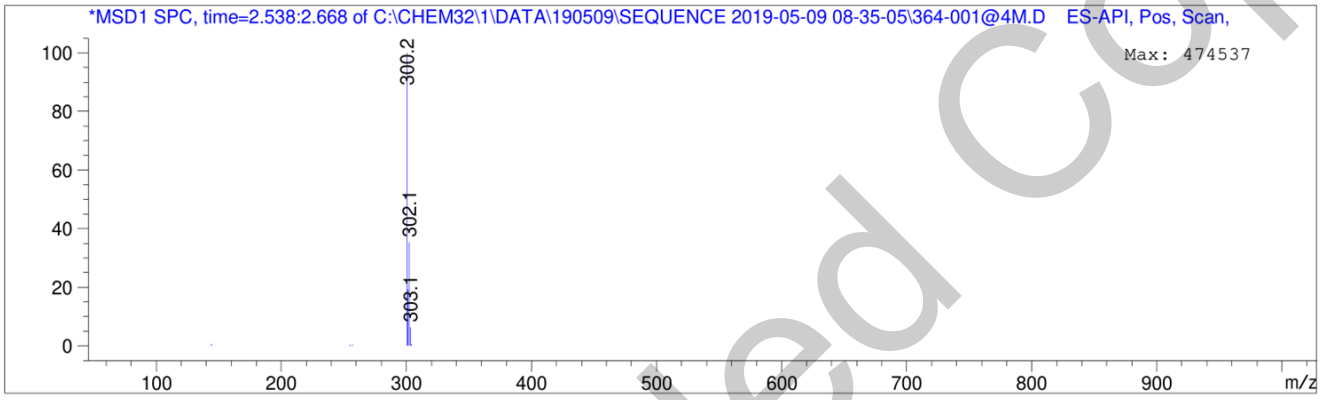
## Ib. Mass Spectrum

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 |
|---|---|---|
| 2.583 | 8480721 | 302.15 I |
| | | 301.20 I |
| | | 300.20 I |




Theoretical value: 300.2 $[M\text{-}HCl+H]^+$.

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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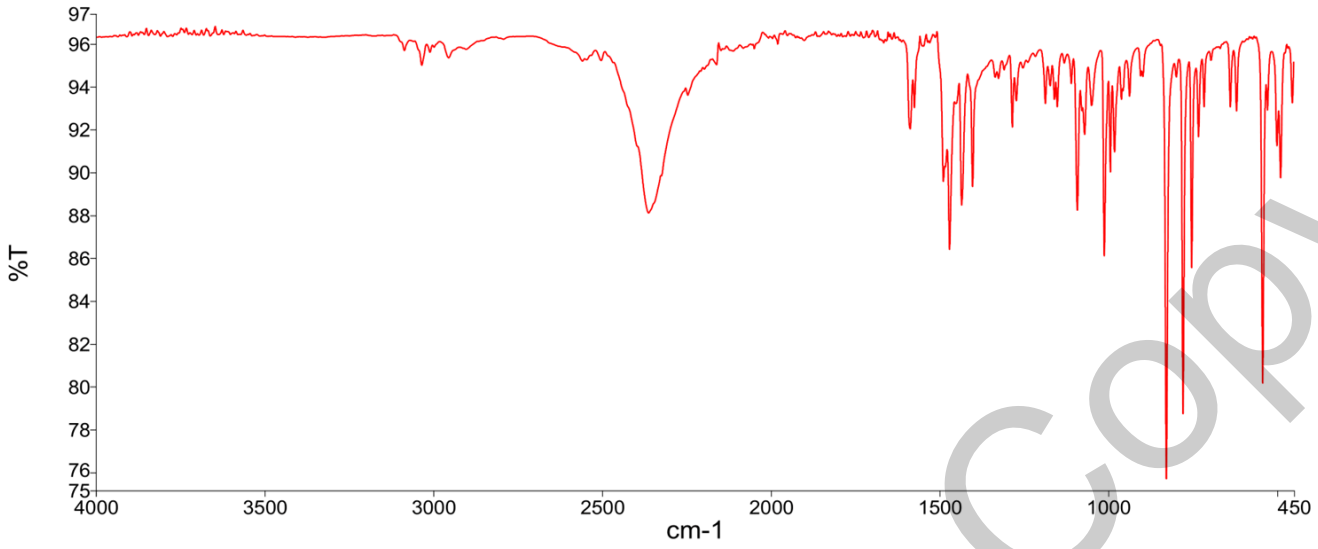
## Ic. IR Spectrum

The infra-red spectrum of this material was analysed by Fourier-Transform 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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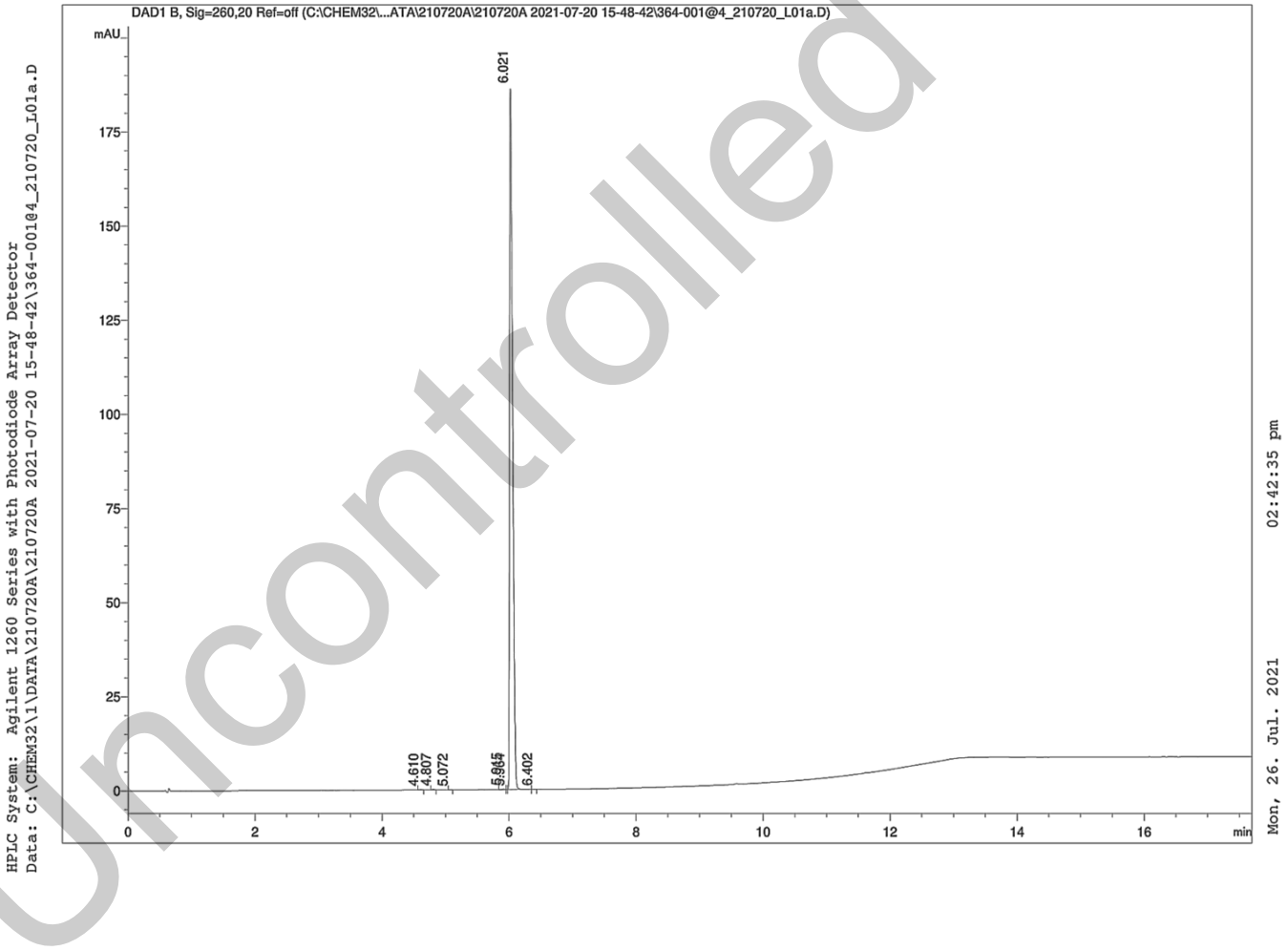
## II. Purity

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<br><br>4.6 x 50mm<br><br>2.7 micron | 25ºC | | | | DAD 260nm | Auto<br>1.0 µL<br>1.0 mg/mL in 100% water (+0.1% 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 | 90 | 10 | 1.0 | | |
| | 6.00 | 60 | 40 | 1.0 | | |
| | 11.50 | 5 | 95 | 1.0 | | |
| | 16.50 | 5 | 95 | 1.0 | | |
| | 17.50 | 90 | 10 | 1.0 | | |
| | 20.50 | 90 | 10 | 1.0 | | |





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**Area Percent Report – Sorted by Signal**

| Peak Number | Retention Time (rounded) | Area | Area % (rounded) |
|---|---|---|---|
| 1 | 4.61 | 0.25 | 0.04 |
| 2 | 4.81 | 0.31 | 0.05 |
| 3 | 5.07 | 0.05 | 0.01 |
| 4 | 5.92 | 0.41 | 0.07 |
| 5 | 5.96 | 0.05 | 0.01 |
| 6 | 6.02 | 615.21 | 99.81 |
| 7 | 6.40 | 0.08 | 0.01 |
| 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 99.8% (average of 10 duplicate analyses)





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## III. Water Content

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

**Results:**

Average 0.2%

## IV. Ash Content

Method: BP 2019 Appendix XIJ Method II

**Result:**

Contains <0.1% ash.

## V. Residual Solvents

Method: $^{1}$HNMR

**Result:**

0.1% isopropanol detected by $^{1}$H NMR analysis.

## VI. Final Result

| | |
|---|---|
| Chromatographic purity (HPLC) | 99.8% |
| Water content | 0.2% |
| Ash content | <0.1% |
| Residual solvents | 0.1% |
| Purity* | 99.5% |

This purity is assessed to be 99.5%.

Product Reviewed By:

James Rixson, PhD
Head of Production

Product Released By:

Jason Chaplin, PhD
Principal Chemist

Release Date: 30 July 2021

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

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





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