



CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D824b.2018.01

Compound Name: ( $\pm$ )-4-Hydroxyamphetamine hydrochloride

Description: Off white powder

Collection number: D824b

Chemical Formula:  $C_9H_{13}NO \cdot HCl$

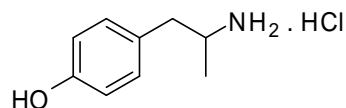
CAS number: 876-26-6 (HCl salt), 103-86-6 (base)

Structure:

Batch number: 09-D-16

Molecular Weight: 187.7 (HCl salt), 151.2 (base)

Release date: 14<sup>th</sup> September 2009



Synonyms: ( $\pm$ ) 4-(2-Aminopropyl)phenol hydrochloride  
( $\pm$ ) *p*-(2-Aminopropyl)phenol hydrochloride  
( $\pm$ )-Hydroxyamphetamine hydrochloride  
4-Hydroxyamphetamine hydrochloride  
*p*-Hydroxyamphetamine hydrochloride  
Paredrine hydrochloride  
 $\alpha$ -Methyl-*p*-tyramine hydrochloride  
 $\alpha$ -Methyltyramine hydrochloride

Purity (mass fraction):  $97.7 \pm 1.0\%$  (95% coverage interval)

Purity estimate obtained from a combination of traditional analytical techniques. The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and  $^1H$  NMR. Supporting evidence is provided by quantitative  $^1H$  NMR using a certified potassium hydrogen maleate internal standard and elemental microanalysis.

GC-FID: Instrument: Varian CP-3800  
Column: VF-1MS, 30 m  $\times$  0.32 mm I.D.  $\times$  0.25  $\mu$ m  
HP-5, 30 m  $\times$  0.32 mm I.D.  $\times$  0.25  $\mu$ m (2012)  
Program: 120  $^{\circ}C$  (10 min), 25  $^{\circ}C/min$  to 300  $^{\circ}C$  (3 min)  
Injector: 250  $^{\circ}C$  Detector Temp: 320  $^{\circ}C$   
Carrier: Helium Split ratio: 20/1  
Relative peak area response of main component:  
Initial analysis: Mean = 99.1%,  $s = 0.03\%$  (10 sub samples in duplicate, July 2009)  
Re-analysis: Mean = 98.7%,  $s = 0.18\%$  (5 sub samples in duplicate, June 2010)  
Re-analysis: Mean = 98.8%,  $s = 0.13\%$  (5 sub samples in duplicate, June 2011)  
Re-analysis: Mean = 98.7%,  $s = 0.02\%$  (5 sub samples in duplicate, May 2012)  
Re-analysis: Mean = 98.9%,  $s = 0.02\%$  (5 sub samples in duplicate, April 2015)  
Re-analysis: Mean = 99.1%,  $s = 0.03\%$  (5 sub samples in duplicate, March 2018)

QNMR: Instrument: Bruker Avance III-400  
Field strength: 400 MHz Solvent:  $D_2O$   
Internal standard: Potassium hydrogen maleate (98.6% mass fraction)  
Purity estimate: Mean = 98.1%,  $s = 0.25\%$  (5 sub samples, August 2009)

Thermogravimetric analysis: Initial non volatile residue < 0.1 % mass fraction (August 2009)  
Volatile content not determined due to volatility of the material

Karl Fischer analysis: Moisture content 0.5% mass fraction (July 2009)  
Moisture content 0.4% mass fraction (June 2010)  
Moisture content 0.5% mass fraction (June 2011)  
Moisture content 0.5% mass fraction (May 2012)  
Moisture content 0.8% mass fraction (April 2015 and March 2018)

Accredited for compliance with ISO Guide 34.

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### Expiration of certification

The property values are valid till 28<sup>th</sup> March 2021, i.e. three years from the date of re-certification provided the **unopened** material is handled and stored in accordance with the recommendations below. The material as issued in the unopened container and stored as recommended below should be suitable for use beyond this date, subject to confirmation of batch stability from the issuing body.

The expiry date/shelf life does not apply to sample bottles that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

The long-term stability of the compound in solution has not been examined.

This material has demonstrated stability over a minimum period of three years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

### Homogeneity assessment

The homogeneity of the material was assessed using purity assay by GC-FID on ten randomly selected 1-2 mg samples of the material. The material was judged to be homogeneous at this level of sampling as the variation in analysis results between samples was not significantly different at a 95% confidence level from that observed on repeat analysis of the same sample.

### Metrological traceability

The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. The purity was derived by subtraction of the mass of impurities from the mass of the reference material. Organic purity is traceable to the SI-derived coherent unit one through chromatographic separation and response factor determination of individual components. Volatile and non-volatile residue content is directly traceable to mass through use of Karl Fischer and thermogravimetric analysis. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

### Recommended storage

When not in use this material should be stored at or below 25 °C in a closed container in a dry, dark area.

### Intended use

For *in vitro* laboratory analysis only.

### Caution

Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

### Legal notice

Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,  
Team Leader,  
Chemical Reference Materials, NMI.  
Dated: 6 April, 2018.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 6<sup>th</sup> April 2018.