

Australian Government



# **National Measurement Institute**

## **REFERENCE MATERIAL ANALYSIS REPORT**

#### Report ID: D1027.2015.01

Compound Name: ( $\pm$ )-*N*-Methylbutylone hydrochloride Collection Number: D1027 Chemical Formula: C<sub>13</sub>H<sub>17</sub>NO<sub>3</sub>.HCl CAS Number: 17763-12-1 Structure:

Description: White solid Batch Number: 14-D-27 Molecular Weight: 271.7 (HCl), 235.3 (base) Release date: 10<sup>th</sup> October 2014

.HCI

Synonyms:  $(\pm)$ -1-(1,3-Benzodioxol-5-yl)-2-(dimethylamino)-1-butanone hydrochloride  $(\pm)$ -2-(Dimethylamino)-3',4'(methylenedioxy)-butyrophenone hydrochloride

Purity (mass fraction):  $99.9 \pm 1.4\%$  (95% coverage interval)

The purity value was 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, Karl Fischer analysis and <sup>1</sup>H NMR. Supporting evidence is provided by quantitative nuclear magnetic resonance (QNMR) and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800		
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm		
Program: 160 °C (15		160 °C (15 min), 30 °C/mi	in to 300 °C (5 min)	
	Injector:	250 °C	Detector Temp: 320 °C	
	Carrier:	Helium	Split ratio: 20/1	
	Relative peak area response of main component as the free base:			
	Initial analysis:	Mean = 99.9%, $s = 0.01\%$ (10 sub samples in duplicate, August 2014)		
	Re-analysis:	Mean = 99.7%, $s = 0.02\%$ (5 sub samples in duplicate, August 2015)		
Karl Fischer analysis:		Moisture content $< 0.2\%$ mass fraction (September 2014)		
Karl Fischer	analysis:	Moisture content $< 0.2\%$ n	nass fraction (September 2014)	
Karl Fischer	analysis:		nass fraction (September 2014) nass fraction (August 2015)	
Karl Fischer QNMR:	analysis: Instrument:			
	·	Moisture content $< 0.2\%$ n		
	Instrument:	Moisture content < 0.2% n Bruker Avance-III-500	nass fraction (August 2015) Solvent: D <sub>2</sub> O (4.79 ppm)	
	Instrument: Field strength: Internal standard: Initial analysis:	Moisture content < 0.2% m Bruker Avance-III-500 500 MHz Maleic acid (98.7% mass f Mean (0.77 ppm) = 100.19	nass fraction (August 2015) Solvent: $D_2O$ (4.79 ppm) fraction) %, s = 0.5% (5 sub samples, August 2014)	
	Instrument: Field strength: Internal standard:	Moisture content < 0.2% m Bruker Avance-III-500 500 MHz Maleic acid (98.7% mass f Mean (0.77 ppm) = 100.19 Mean (6.09 ppm) = 100.29	nass fraction (August 2015) Solvent: $D_2O$ (4.79 ppm) fraction)	

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Spectroscopic and other characterisation data				
GC-MS:	Instrument: Column: Program: Injector: Carrier:	Agilent 6890/5973 TG-1MS, 30 m x 0.25 mm 160 °C (15 min), 30 °C/min 180 °C Helium, 1.0 mL/min	•	
		of the free base is reported along with the major peaks in the mass spectrum. ted as mass/charge ratios and (in brackets) as a percentage relative to the base		
	Free base (12.0 min	se (12.0 min): 149 (7), 121 (3), 86 (100), 71 (8), 65 (4) m/z		
ESI-MS:	Instrument: Operation: Ionisation: EM voltage: Cone voltage: Peak:	Micromass Quatro LC Mic Positive ion mode, direct in ESI spray voltage at 3.5 kV 650 V 20 V 236.4 (M+H <sup>+</sup> ) m/z	ifusion at 10 μL/min	
<sup>1</sup> H NMR:	Instrument: Field strength: Spectral data:	(3H, s), 5.11 (1H, dd, J = 3	Solvent: D <sub>2</sub> O (4.79 ppm) 2.08 (1H, m), 2.16 (1H, m), 2.86 (3H, s), 2.98 .9, 6.3 Hz), 6.09 (2H, m), 6.98 (1H, d, <i>J</i> = 8.3 z), 7.67 (1H, dd, <i>J</i> = 1.9, 8.3 Hz) ppm	
<sup>13</sup> C NMR:	Instrument: Field strength: Spectral data:	Bruker Avance III-500 126 MHz δ 7.0, 22.1, 40.1, 43.6, 70.0 194.6 ppm	Solvent: D <sub>2</sub> O 0, 102.7, 107.7, 108.5, 126.7, 128.0, 148.4, 153.8,	
Melting point:		220-222 °C		
Microanalysis:		Found: C = 57.6%; H = 6.8%; N = 5.2%, Cl = 13.0 (September, 2014) Calc: C = 57.5%; H = 6.7%; N = 5.2%, Cl = 13.1 (Calculated for $C_{13}H_{17}NO_3.HCl$ )		

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# **Expiration of certification**

The property values are valid till 10<sup>th</sup> August 2018, 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 been given a shelf life of three years from the date of re-certification. The material will be retested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

In the absence of stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last 10 years.

#### Homogeneity assessment

The homogeneity of the material was assessed using purity assay by GC-FID on ten randomly selected 1-2 mg sub 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.

#### **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: 12 August, 2015.

Characterisation data and property values specified in this report supersede those in all reports issued prior to  $12^{\text{th}}$  August 2015.



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