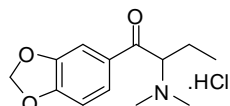




REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D1027.2015.01

Compound Name: (±)- <i>N</i> -Methylbutylone hydrochloride	Description: White solid
Collection Number: D1027	Batch Number: 14-D-27
Chemical Formula: C ₁₃ H ₁₇ NO ₃ .HCl	Molecular Weight: 271.7 (HCl), 235.3 (base)
CAS Number: 17763-12-1	Release date: 10 th October 2014
Structure:	



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

Purity (mass fraction): 99.9 ± 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 ¹H 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 min), 30 °C/min 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)
Moisture content < 0.2% mass fraction (August 2015)

QNMR: Instrument: Bruker Avance-III-500
Field strength: 500 MHz Solvent: D₂O (4.79 ppm)
Internal standard: Maleic acid (98.7% mass fraction)
Initial analysis: Mean (0.77 ppm) = 100.1%, s = 0.5% (5 sub samples, August 2014)
Initial analysis: Mean (6.09 ppm) = 100.2%, s = 0.5% (5 sub samples, August 2014)
Initial analysis: Mean (7.66 ppm) = 100.0%, s = 0.5% (5 sub samples, August 2014)

Expiration of certification

The property values are valid till 10th 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 re-tested 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 12th August 2015.