



REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D1018.2017.01

Compound Name: 2-(3,4-Dimethyl-2,5-dimethoxyphenyl)-N-[(2-methoxybenzyl)]ethylamine hydrochloride

Description: Off white solid

Collection Number: D1018

Chemical Formula: C₂₀H₂₇NO₃.HCl

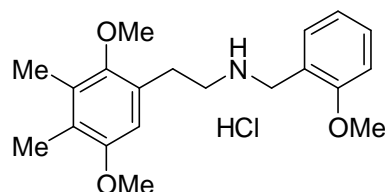
CAS Number: 1354632-65-7 (base)

Structure:

Batch Number: 14-D-17

Molecular Weight: 365.9 (HCl), 329.4 (base)

Release date: 17th October 2014



Synonyms: 25G-NBOMe
2-(3,4-Dimethyl-2,5-dimethoxyphenyl)-N-(2-methoxybenzyl)ethanamine hydrochloride
2,5-Dimethoxy-N-[(2-methoxyphenyl)methyl]-3,4-dimethylbenzeneethanamine hydrochloride

Purity (mass fraction): 97.8 ± 1.2% (95% coverage interval)

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 headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID: Instrument: Varian CP-3800
Column: HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
Program: 120 °C (1 min), 20 °C/min to 250 °C (5 min), 30 °C/min to 300 °C (3 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 = 98.3%, s = 0.03% (10 sub samples in duplicate, July 2014)

GC-FID: Instrument: Varian CP-3800
Column: HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
Program: 120 °C (1 min), 20 °C/min to 250 °C (5 min), 30 °C/min to 300 °C (3 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 = 98.0%, s = 0.02% (7 sub samples in duplicate, July 2014)
Re-analysis: Mean = 98.8%, s = 0.11% (5 sub samples in duplicate, July 2015)
Re-analysis: Mean = 98.3%, s = 0.03% (5 sub samples in duplicate, May 2016)
Re-analysis: Mean = 98.0%, s = 0.08% (5 sub samples in duplicate, April 2017)

Karl Fischer analysis: Moisture content 0.1% mass fraction (July 2014, July 2015 and May 2016)
Moisture content 0.2% mass fraction (April 2017)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m × 0.25 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 10 °C/min to 300 °C (3 min)
	Injector:	250 °C Transfer line temp: 280 °C
	Carrier:	Helium, 1.0 mL/min Split ratio: 20/1
	The retention times of the free base and <i>N</i> -acetyl derivative are reported along with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Free base (22.3 min): 298 (M^+ -OMe, 13), 180 (7), 150 (42), 121 (100), 91 (26) m/z	
	<i>N</i> -acetyl (24.5 min): 371 (M^+ , 11), 192 (93), 177 (13), 150 (8), 121 (100), 91 (30) m/z	
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 µm
	Program:	50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)
	Injector:	150 °C Transfer line temp: 280 °C
	Carrier:	Helium, 1.2 mL/min Split ratio: 50/1
	Solvents detected:	Diethyl ether, isopropanol
TLC:	Kieselgel 60F ₂₅₄	Dichloromethane/Methanol/conc NH ₃ (11:1:drop) Single spot observed, R _f = 0.53. Visualisation with UV light (254 nm)
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm ⁻¹
	Peaks:	2939, 2837, 2743, 2667, 2622, 2477, 1600, 1585, 1494, 1482, 1466, 1438, 1407, 1253, 1231, 1118, 1090, 1032, 1015, 767, 477 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance -500
	Field strength:	500 MHz Solvent: d ₆ -DMSO (2.50 ppm)
	Spectral data:	δ 2.04 (3H, s), 2.12 (3H, s), 2.98 (2H, m), 3.08 (2H, m), 3.58 (3H, s), 3.73 (3H, s), 3.84 (3H, s), 4.14 (2H, s), 6.66 (1H, s), 7.00 (1H, dt, <i>J</i> = 1.0, 7.5 Hz), 7.10 (1H, dd, <i>J</i> = 0.6, 7.7 Hz), 7.42 (1H, ddd, <i>J</i> = 1.7, 7.6, 8.3 Hz), 7.51 (1H, dd, <i>J</i> = 1.7, 7.5 Hz), 9.22 (2H, br s) ppm Isopropanol (0.1%) and diethyl ether (0.2%) estimated mass fraction was observed in the ¹ H NMR
¹³ C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	125 MHz Solvent: d ₆ -DMSO (39.52 ppm)
	Spectral data:	δ 11.9, 12.6, 26.3, 44.9, 47.0, 55.6, 60.8, 109.5, 111.1, 119.8, 120.4, 124.2, 127.0, 130.4, 130.8, 131.5, 150.1, 153.2, 157.5 ppm
Melting point:		170 °C
Microanalysis:		Found: C = 65.5 %; H = 7.9 %; N = 3.8 %; Cl = 9.4% (August 2014) Calc: C = 65.7 %; H = 7.7 %; N = 3.8 %; Cl = 9.7 % (for C ₂₀ H ₂₈ ClNO ₃)

Expiration of certification

The property values are valid till 6th April 2020, 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.

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.

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 sub samples of the material. The material was judged to be sufficiently 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, protected from ambient moisture and light.

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: 27 April, 2017.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 27th April, 2017