



REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D1001.2016.01

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

Description: Off white solid

Collection Number: D1001

Chemical Formula: C₁₈H₂₂INO₃.HCl

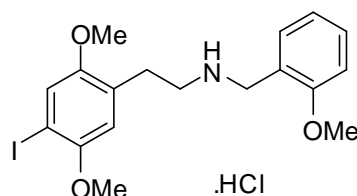
CAS Number: 1043868-97-8

Structure:

Batch Number: 13-D-26

Molecular Weight: 463.7 (HCl), 427.3 (base)

Release date: August 2013



Synonyms: 4-Iodo-2,5-dimethoxy-*N*-[(2-methoxyphenyl)methyl]benzeneethanamine hydrochloride
2-(4-Iodo-2,5-dimethoxyphenyl)-*N*-[(2-methoxyphenyl)methyl]ethanamine hydrochloride
2-(4-Iodo-2,5-dimethoxyphenyl)-*N*-(2-methoxybenzyl)ethanamine hydrochloride
25I-NBOMe

Purity (mass fraction): 92.1 ± 2.2% (95% coverage interval)

The purity value was obtained from a combination of traditional analytical. The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by HPLC with UV detection, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR. Supporting evidence is provided by headspace GC-MS analysis of occluded solvent and elemental microanalysis.

Note: 2-(2,5-Dimethoxyphenyl)-*N*-[(2-methoxybenzyl)]-ethylanamine hydrochloride estimated at 5.4% is present in this material.

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
Column: X-bridge C-18, 5 μm (4.6 mm x 150 mm)
Column oven: 40 °C
Mobile Phase: Acetonitrile/MilliQ water (50:50)
The aqueous phase was buffered at pH 10.8 using 20mM NH₄OAc
Flow rate: 1 mL/min
Detector: SPD-M20A PDA 2998 operating at 210 nm
Relative peak area response of main component:
Initial analysis: Mean = 94.3%, s = 0.06% (10 sub samples in duplicate, July 2013)
Re-analysis: Mean = 94.8%, s = 0.06% (5 sub samples in duplicate, May 2014)
Re-analysis: Mean = 95.3%, s = 0.4% (5 sub samples in duplicate, June 2015)
Re-analysis: Mean = 94.6%, s = 0.03% (5 sub samples in duplicate, August 2016)

Thermogravimetric analysis: Volatile content 1.4% and non volatile residue < 0.2% mass fraction (July 2013)

Karl Fischer analysis: Moisture content 2.3% mass fraction (August 2013)
Moisture content 3.0% mass fraction (June 2014)
Moisture content 2.5% mass fraction (May 2015)
Moisture content 2.8% mass fraction (June 2016)

Spectroscopic and other characterisation data

LC-MS:	Instrument:	Waters 2695 (HPLC)/Micromass Quatro	
	Column:	X-Bridge C-18, 150 mm × 4.6 mm I.D. × 5 µm	
	Column temp:	40 °C	
	Solvent system:	2% formic acid [10% v/v], acetonitrile [50% v/v], MilliQ water [40% v/v]	
	Flow rate:	0.2 mL/min	
	Sample prep:	1000 µg/g in 2% formic acid [10% v/v], acetonitrile [50% v/v], MilliQ water [40% v/v]	
	Injection volume:	10 µL	
	Ionisation mode:	Electrospray positive ion	
	Capillary voltage:	3.5 kV	Cone voltage: 5 V
	Source temp:	130 °C	Desolvation gas temperature: 350 °C
	Cone gas flow rate:	28 L/hr	Desolvation gas flow rate: 770 L/hr
	The retention time of 2-(4-iodo-2,5-dimethoxyphenyl)- <i>N</i> -[(2-methoxybenzyl)]ethylamine hydrochloride is reported along with the major peak in the mass spectrum. The latter is reported as a mass/charge ratio.		
	4.8 min:	428.0 (M+H ⁺) 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:	Isopropanol, diethyl ether	
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Hexane/ethyl acetate/diethyl amine (15/10/0.2) Single spot observed, R _f = 0.34. Visualisation with UV at 254 nm	
IR:	Instrument:	Biorad FTS3000MX FT-IR	
	Range:	4000-400 cm ⁻¹ , KBr powder	
	Peaks:	3597, 3440, 2934, 2831, 2764, 2462, 2521, 2488, 2416, 2347, 1589, 1492, 1454, 1253, 1216, 1034, 865, 767 cm ⁻¹	
¹ H NMR:	Instrument:	Bruker Avance-400	
	Field strength:	400 MHz	Solvent: d ₆ -DMSO (2.50 ppm)
	Spectral data:	δ 2.97 (2H, m), 3.06 (2H, m), 3.73 (3H, s), 3.77 (3H, s), 3.83 (3H, s), 4.11 (2H, s), 6.91 (1H, s), 7.00 (1H, dt, <i>J</i> = 0.8, 7.5 Hz), 7.08 (1H, d, <i>J</i> = 7.8 Hz), 7.32 (1H, s), 7.41 (1H, dt, <i>J</i> = 1.7, 7.9 Hz), 7.49 (1H, dd, <i>J</i> = 1.9, 7.5 Hz), 9.23 (2H, s) ppm	
	Isopropanol estimated at 0.03% mass fraction was observed in the ¹ H NMR		
	Diethyl ether estimated at 0.03% mass fraction was observed in the ¹ H NMR		
¹³ C NMR:	Instrument:	Bruker Avance-400	
	Field strength:	101 MHz	Solvent: d ₆ -DMSO (39.52 ppm)
	Spectral data:	δ 26.5, 44.8, 45.7, 55.6, 56.2, 56.8, 84.0, 111.1, 113.8, 119.7, 120.4, 121.4, 126.3, 130.8, 131.5, 151.8, 152.0, 157.5 ppm	
Melting point:	156-159 °C (dec.)		
Microanalysis:	Found: C = 46.7%; H = 5.1%; N = 3.1%; Cl = 7.2%; I = 26.3% (June, 2013) Calculated: C = 46.6%; H = 5.0%; N = 3.0%; Cl = 7.7%; I = 27.4% (Calculated for C ₁₈ H ₂₂ INO ₃ ·HCl)		

Expiration of certification

The property values are valid till 9th August 2019, 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 HPLC with UV detection 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 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: 24 August, 2016.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 24th August 2016.