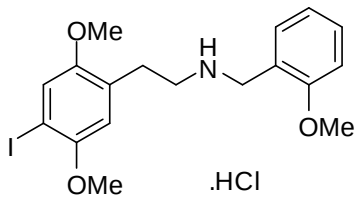
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D1001: 2-(4-Iodo-2,5-dimethoxyphenyl)-N-[(2-methoxybenzyl)] ethylamine hydrochloride

Report ID: D1001.2022.02

Chemical Formula: $C_{18}H_{22}INO_3$.HCl

Molecular Weight: 463.7 g/mol (HCl), 427.3 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
13-D-26	1043868-97-8 (HCl)	91.4 ± 1.9%

IUPAC name: 2-(4-Iodo-2,5-dimethoxyphenyl)-N-(2-methoxybenzyl)ethanamine hydrochloride (1:1).

Expiration of certification: The property values are valid till 3 May 2027, i.e. five 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.

Description: Off-white solid prepared by synthesis, and certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium cap or screw top cap.

Intended use: This reference material is recommended for qualitative analysis only and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

Instructions for use: Equilibrate the bottled material to room temperature before opening.

Recommended storage: When not in use this material should be stored at or below 25 ºC in a closed container in a dry, dark area.

Stability: This material has demonstrated stability over a minimum period of three years. The long-term stability of the compound in solution has not been examined.

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.

Safety: Treat as a hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust. Refer to the provided safety data sheet.

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
21 September 2022

This report supersedes any issued prior to 21 September 2022.

NATA Accreditation No. 198 / Corporate Site No. 14214.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. Impurities of related structure were assessed by HPLC with UV detection. The purity value was obtained by mass balance from a combination of traditional analytical techniques, including HPLC with UV detection. The purity value is calculated as per Equation 1.

Supporting evidence is provided by thermogravimetric analysis, Karl Fischer analysis, ^{1}H NMR spectroscopy, qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

Note: 2-(2,5-Dimethoxyphenyl)-*N*-[(2-methoxybenzyl)]-ethylamine 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 NH_4OAc
	Flow rate:	1 mL/min
	Detector:	Shimadzu SPD-M20A PDA operating at 210 nm
	Relative peak area of the 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)
	Re-analysis:	Mean = 93.9%, s = 0.03% (5 sub samples in duplicate, August 2019)
	Re-analysis:	Mean = 93.5%, s = 0.07% (5 sub samples in duplicate, May 2022)
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) Moisture content 2.7% mass fraction (July 2019) Moisture content 2.7% mass fraction (May 2022)

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 ºC
	Desolvation gas temp:	350 ºC
	Cone gas flow rate:	28 L/hr
	Desolvation gas flow:	770 L/hr
	The retention time of 2-(4-iodo-2,5-dimethoxyphenyl)-N-[(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 60F254. 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, J = 0.8, 7.5 Hz), 7.08 (1H, d, J = 7.8 Hz), 7.32 (1H, s), 7.41 (1H, dt, J = 1.7, 7.9 Hz), 7.49 (1H, dd, J = 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
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)