



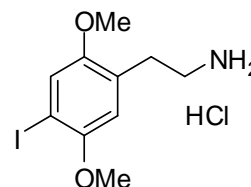
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D922: 2,5-Dimethoxy-4-iodophenylethylamine hydrochloride

Report ID: D922.2023.01

Chemical Formula: C₁₀H₁₄INO₂.HCl

Molecular Weight: 343.6 g/mol (HCl), 307.1 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
07-D-16	64584-32-3 (HCl) 69587-11-7 (base)	99.2 ± 0.6%

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

IUPAC name: 2-(4-Iodo-2,5-dimethoxyphenyl)ethanamine hydrochloride

Expiration of certification: The property values are valid till 16 May 2033, ten 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 powder prepared by synthesis, 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 certified reference material is suitable for use as a primary calibrator.

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 °C in a closed container in a dry, dark area.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance all impurities are quantified as a mass fraction and subtracted from 100%.

Stability: This material has demonstrated stability over a minimum period of ten years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials. 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 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.

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.
18 May 2023

This report supersedes any issued prior to 18 May 2023.

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

Legal notice: Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100 \% - I_{\text{ORG}}) \times (100 \% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by elemental microanalysis.

GC-FID: Instrument: Agilent 6890
 Column: HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 100 °C (1 min), 10 °C/min to 200 °C (4 min), 30 °C/min to 300 °C (3 min)
 Injector: 250 °C
 Detector Temp: 320 °C
 Carrier: Helium
 Split ratio: 20/1

Relative mass fraction of the main component:

Initial analysis: Mean = 99.6%, s = 0.05% (10 sub samples in duplicate, October 2007)
 Re-analysis: Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, October 2008)
 Re-analysis: Mean = 99.6%, s = 0.04% (5 sub samples in duplicate, October 2009)
 Re-analysis: Mean = 99.5%, s = 0.04% (5 sub samples in duplicate, September 2013)
 Re-analysis: Mean = 99.5%, s = 0.06% (4 sub samples in duplicate, July 2018)
 Re-analysis: Mean = 99.6%, s = 0.05% (5 sub samples in duplicate, May 2023)

GC-FID: Instrument: Varian CP-3800
 Column: HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 120 °C (1 min), 8 °C/min to 180 °C (6 min), 30 °C/min to 300 °C (3 min)
 Injector: 200 °C
 Detector Temp: 320 °C
 Carrier: Helium
 Split ratio: 20/1

Relative mass fraction of the main component:

Initial analysis: Mean = 99.5%, s = 0.03% (5 sub samples in duplicate, November 2010)

Thermogravimetric analysis: Volatile content not determined and non volatile residue 0.52% (October 2007)
 Volatile content 0.22% mass fraction (October 2008)
 Volatile content not determined and non volatile residue 0.24% (November 2010)

Karl Fischer analysis: Moisture content ≤ 0.2% mass fraction (November 2007 – May 2023)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.20 μm
	Program:	100 °C (1 min), 15 °C/min to 250 °C, (2 min), 40 °C/min to 300 °C (1 min)
	Injector:	250 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	20/1
	The retention time of the free base is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Free base (8.8 min):	307 (M ⁺ , 15), 278 (100), 263 (18), 247 (6), 105 (3), 91 (4), 77 (4) <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Methanol/Conc. NH ₃ (200/3) Single spot observed, R _f = 0.3-0.4. Visualisation with UV at 254 nm.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm ⁻¹ , KBr powder
	Peaks:	2953, 2904, 2753, 2660, 2557, 2458, 2046, 1601, 1489, 1434, 1381, 1306, 1215, 1047, 1025, 853, 791, 729, 706 cm ⁻¹ .
¹ H NMR:	Instrument:	Bruker DMX500
	Field strength:	500 MHz
	Solvent:	D ₂ O (4.80 ppm)
	Spectral data:	δ 2.94 (2H, t, <i>J</i> = 7.0 Hz), 3.22 (2H, t, <i>J</i> = 7.0 Hz), 3.80 (3H, s), 3.82 (3H, s), 6.90 (1H, s), 7.42 (1H, s) ppm
¹³ C NMR:	Instrument:	Bruker Gyro-300
	Field strength:	75 MHz
	Solvent:	D ₂ O
	Spectral data:	δ 28.0, 39.3, 56.1, 57.1, 83.4, 114.4, 122.2, 126.3, 152.0, 152.3 ppm
Melting point:	251-253 °C	
Microanalysis:	Found:	C = 35.1 %, H = 4.6 %; N = 4.1% (October 2007)
	Calculated:	C = 35.0 %, H = 4.4 %; N = 4.1% (Calc for C ₁₀ H ₁₄ INO ₂ .HCl)