Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

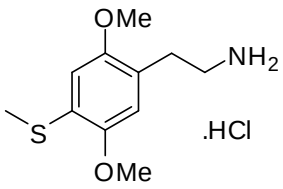
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1019: 2,5-Dimethoxy-4-methylthiophenethylamine hydrochloride

Report ID: D1019.2024.01

Chemical Formula: $C_{11}H_{17}NO_2S.HCl$

Molecular Weight: 263.8 g/mol (HCl) 227.3 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
14-D-18	61638-10-6 (HCl) 61638-09-3 (base)	99.4 ± 0.6%

IUPAC name: 2-[2,5-Dimethoxy-4-(methylsulfanyl)phenyl]ethanamine hydrochloride (1:1).

Expiration of certification: The property values are valid till 19 February 2029, 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 powder 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 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 ºC 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: In the absence of long term 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 ten 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 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.
22 February 2024

This report supersedes any issued prior to 22 February 2024.

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 indicative purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis, and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100 % - I_{ORG}) x (100 % - I_{VOL} – I_{NVR}) Equation 1

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

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

GC-FID:	Instrument:	Varian CP-3800 or Agilent 8890
	Column:	VF-1ms or HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	120 °C (1 min), 12 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component as the free base:	
	Initial analysis:	Mean = 99.99%, s = 0.001% (10 sub samples in duplicate, June 2014)
	Re-analysis:	Mean = 99.97%, s = 0.014% (7 sub samples in duplicate, May 2015)
	Re-analysis:	Mean = 99.8%, s = 0.04% (5 sub samples in duplicate, April 2016)
	Re-analysis:	Mean = 99.97%, s = 0.003% (5 sub samples in duplicate, February 2024)
Karl Fischer analysis:		Moisture content 0.2% mass fraction (June 2014, May 2015 and April 2016, February 2024)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (May 2015). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material and/or degradation at elevated temperatures.

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 250 °C (5 min), 30 °C/min to 300 °C (2 min)
	Injector:	250 °C,
	Split ratio:	20/1
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the free base compound is reported 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 (13.7 min):	227 (M^+, 32), 198 (100), 183 (51), 167 (31), 152 (19), 137 (8), 91 (7) *m/z*
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	6 V
	Peak:	228.2 ($M+H^+$) *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
	Split ratio:	50/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Solvents detected:	Ethanol, acetone
TLC:	Conditions:	Kieselgel 60F_{254}. Ammonia/methanol (1.5/100) Single spot observed, R_f = 0.3
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3213, 2953, 2905, 2656, 2558, 2454, 2041, 1606, 1502, 1491, 1393, 1209, 1040, 843, 788, 725, 624, 438 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 2.41 (3H, s), 2.91 (2H, t, *J* = 6.9 Hz), 3.18 (2H, t, *J* = 6.9 Hz), 3.80 (3H, s), 3.81 (3H, s), 6.83 (1H, s), 6.86 (1H, s) ppm
		Ethanol estimated at 0.3% mass fraction was observed in the ^{1}H NMR.
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	D_2O
	Spectral data:	δ 13.9, 27.6, 39.6, 56.2, 56.4, 110.6, 114.0, 122.7, 125.5, 149.8, 152.1 ppm
Melting point:		242-243 °C
Microanalysis:	Found:	C = 50.2%; H = 7.1%; N = 5.3%, Cl = 13.3%; S = 12.1% (June 2014)
	Calculated:	C = 50.1%; H = 6.9%; N = 5.3%, Cl = 13.4%; S = 12.2% (Calculated for $C_{11}H_{17}NO_2S.HCl$)