



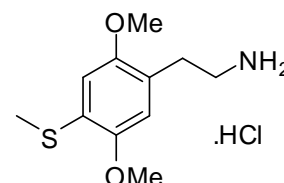
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

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

Report ID: D1019.2016.04

Chemical Formula: C₁₁H₁₇NO₂S.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)	99.3 ± 1.8%

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

Expiration of certification: The property values are valid till 8 April 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.

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 reference material is recommended for qualitative analysis only.

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.

Stability: In the absence of long term stability data the stability of this material has been judged 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

Report ID: D1019.2016.04
Product release date: 2 July 2014

measurement.gov.au

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.

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. Impurities of related structure were assessed by GC-FID. The purity estimate was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID. 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 thermogravimetric analysis, Karl Fischer analysis and ^1H NMR spectroscopy qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID: Instrument: Varian CP-3800
Column: VF-1ms, 30 m \times 0.32 mm I.D. \times 0.25 μm
Program: 120 $^\circ\text{C}$ (1 min), 12 $^\circ\text{C}/\text{min}$ to 300 $^\circ\text{C}$ (3 min)
Injector: 250 $^\circ\text{C}$
Detector Temp: 320 $^\circ\text{C}$
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)

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

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:	Free base compound:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μ m
	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 <i>m/z</i>
	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) <i>m/z</i>
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	6 V
	Peak:	228.2 ($M+H^+$) <i>m/z</i>
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μ m
	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 ₂ O (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 0.5% mass fraction was observed in the ¹ H NMR
¹³ C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	D ₂ O
	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 ₂ S.HCl)