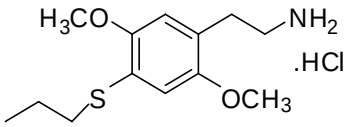
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D919: 2,5-Dimethoxy-4-(propylthio)phenethylamine hydrochloride



Report ID: D919.2022.01

Chemical Formula: $C_{13}H_{21}NO_2S.HCl$

Molecular Weight: 291.8 g/mol (HCl), 255.4 g/mol (free base)

Certified value

Batch No.	CAS No.	Purity (mass fraction)
07-D-11	850140-15-7 (HCl) 207740-26-9 (base)	99.1 ± 0.3%

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

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

Expiration of certification: The property values are valid till 6 December 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 powder prepared by synthesis or sourced from an external supplier, 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 approach all impurities are quantified as a mass fraction and subtracted from 100%.

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

Caution: 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.
19 December 2022

This report supersedes any issued prior to 19 December 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. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, 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 elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890N or Agilent 7890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 180 °C (8 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium, 2.0 mL/min
	Split ratio:	20/1
	Relative mass fraction of the main component	
	Initial analysis:	Mean = 98.9%, s= 0.05% (5 sub samples in duplicate, June 2018)
	Re-analysis:	Mean = 99.2%, s= 0.04% (5 sub samples in duplicate, December 2022)
GC-FID:	Instrument:	Varian CP3800 or Agilent 6890N
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 200 °C (3 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C or 200°C
	Detector Temp:	320 °C
	Carrier:	Helium, 2.0 mL/min
	Split ratio:	20/1
	Relative mass fraction of the main component	
	Initial analysis:	Mean = 98.8%, s = 0.06% (10 sub samples in duplicate, September 2007)
	Re-analysis:	Mean = 99.1%, s = 0.02% (5 sub samples in duplicate, October 2008)
	Re-analysis:	Mean = 99.1%, s= 0.03% (5 sub samples in duplicate, October 2009)
	Re-analysis:	Mean = 99.0%, s = 0.02% (5 sub samples in duplicate, October 2010)
	Re-analysis:	Mean = 99.0%, s = 0.04% (5 sub samples in duplicate, August 2013)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (November 2007 to October 2010) Moisture content < 0.2% mass fraction (August 2013, June 2018 and December 2022)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	ZB-5, 28 m × 0.25 mm I.D. × 0.25 μm
	Program:	60 °C (6 min), 10 °C/min to 100 °C, 15 °C/min to 250 °C, (2 min), 50 °C/min to 300 °C (6 min)
	Injector:	250 °C
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	20/1
	Scan range:	50-550 *m/z*
	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.	
	Parent (18.5 min):	255 (M^+, 29), 226 (100), 183 (48), 169 (21), 153 (21) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. MeOH:NH_3 (100:1.5) Single spot observed, R_f = 0.54. Visualisation with iodine
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	2955, 2906, 2654, 2462, 2041, 1601, 1499, 1389, 1208, 1039, 811, 737 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX500
	Field strength:	500 MHz
	Solvent:	d_4-MeOH (3.30 ppm)
	Spectral data:	δ 1.02 (3H, t, *J* = 7.3 Hz), 1.62 (2H, m), 2.86 (2H, t, *J* = 7.2 Hz), 2.93 (2H, t, *J* = 7.3 Hz), 3.12 (2H, t, *J* = 7.2 Hz), 3.81 (3H, s), 3.82 (3H, s), 6.83 (1H, s), 6.91 (1H, s) ppm
^{13}C NMR:	Instrument:	Bruker Gyro-300
	Field strength:	75 MHz
	Solvent:	d_4-MeOH (49.0 ppm)
	Spectral data:	δ 13.7, 23.5, 29.7, 35.2, 40.9, 56.5, 57.1, 114.1, 115.3, 124.3, 126.1, 153.2, 153.4 ppm
Melting point:		195-197 °C
Microanalysis:	Found:	C = 53.4%; H = 7.6%; N = 4.8%; S = 10.8% (October 2007)
	Calculated:	C = 53.5%; H = 7.6%; N = 4.8%; S = 11.0% (Calculated for $C_{13}H_{21}NO_2S.HCl$)