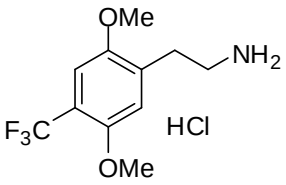
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D1039: 4-(Trifluoromethyl)-2,5-dimethoxyphenethylamine hydrochloride

Report ID: D1039.2016.04

Chemical Formula: $C_{11}H_{14}F_3NO_2.HCl$

Molecular Weight: 285.7 g/mol (HCl), 249.2 (base)



Property value

Batch No.	CAS No.	Purity estimate
14-D-39	159277-13-1 (HCl) 159277-08-4 (base)	97.9 ± 3.2%

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

Expiration of certification: The property values are valid till 13 May 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 ºC 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 nine 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 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.

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 and qualitative elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800/Agilent 6890
	Column:	VF-1/HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	120 °C (1 min), 5 °C/min to 180 °C, 20 °C/min to 300 °C (5 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 = 98.6%, s = 0.08% (9 sub samples in duplicate, June 2015)
	Re-analysis:	Mean = 98.4%, s = 0.03% (5 sub samples in duplicate, May 2016)
Karl Fischer analysis:		Moisture content 0.2% mass fraction (June 2015) Moisture content 0.5% mass fraction (May 2016)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (June 2015). The volatile content (e.g. organic solvents and/or water) could not be determined using thermogravimetric analysis.

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	120 °C (1 min), 5 °C/min to 180 °C, 20 °C/min to 300 °C (3 min)
	Injector:	250 °C,
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the free base 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 (8.8 min):	249 (M^+, 25), 220 (100), 205 (20), 201 (21), 200 (11), 189 (12), 127 (16) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Diethyl ether/TBME/diethyl amine (45/45/10) Single spot observed, R_f = 0.3
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2951, 2904, 1511, 1468, 1410, 1386, 1325, 1268, 1220, 1189, 1129, 1053, 1029, 871 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 2.95 (2H, m), 3.00 (2H, m), 3.81 (3H, s), 3.84 (3H, s) 7.12 (1H, s), 7.19 (1H, s), 8.21 (3H, br s) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 28.0, 38.1, 56.2, 56.6, 109.6 (q, J_{CF} = 5 Hz), 115.6 (q, J_{CF} = 30.3 Hz), 115.8, 123.7 (q, J_{CF} = 272.9 Hz), 131.4, 150.6, 150.8 (q, J_{CF} = 1.7 Hz) ppm
^{19}F NMR:	Instrument:	Bruker Avance III-500
	Field strength:	470 MHz
	Solvent:	DMSO-d_6
	Spectral data:	δ -60.3 ppm
Melting point:		258-259 °C
Microanalysis:	Found:	C = 45.9%; H = 5.3%; N = 4.9%; Cl = 12.4%; F = 19.9% (June, 2015)
	Calculated:	C = 46.3%; H = 5.3%; N = 4.9%; Cl = 12.4%; F = 20.0% (Calculated for $C_{11}H_{14}F_3NO_2$.HCl)