



REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D1007.2016.01

Compound Name: **4-Chloro-2,5-dimethoxyphenethylamine hydrochloride**

Description: Off white solid

Collection Number: D1007

Chemical Formula: C₁₀H₁₄ClNO₂.HCl

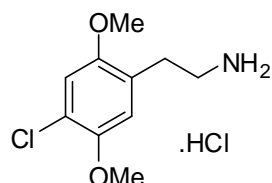
CAS Number: 88441-15-0

Structure:

Batch Number: 13-D-27

Molecular Weight: 252.1 (HCl), 215.7 (base)

Release date: 1st October 2013



Synonyms: 2C-C
4-Chloro-2,5-dimethoxy-benzenethaamine hydrochloride

Purity (mass fraction): 98.9 ± 1.5% (95% coverage interval)

The purity value was obtained from a combination of traditional analytical techniques by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR spectroscopy. Supporting evidence is provided by headspace GC-MS analysis of occluded solvent and elemental microanalysis.

GC-FID: Instrument: Agilent 6890
Column: HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
Program: 150 °C (16 min), 20 °C/min to 300 °C (3 min)
Injector: 250 °C Detector Temp: 320 °C
Carrier: Helium Split ratio: 20/1

Relative peak area response of main component as the free base:

Initial analysis: Mean = 98.8%, s = 0.10% (10 sub samples in duplicate, September 2013)
Re-analysis: Mean = 98.9%, s = 0.09% (5 sub samples in duplicate, August 2014)
Re-analysis: Mean = 98.8%, s = 0.21% (5 sub samples in duplicate, July 2015)
Re-analysis: Mean = 99.2%, s = 0.06% (4 sub samples in duplicate, June 2016)

Thermogravimetric analysis: Non-volatile residue < 0.2% mass fraction (September 2013). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.

Karl Fischer analysis: Moisture content 0.2% mass fraction (September 2013)
Moisture content < 0.1% mass fraction (August 2014 & July 2015)
Moisture content 0.15% mass fraction (June 2016)

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 μ m Program: 150 $^{\circ}$ C (16 min), 20 $^{\circ}$ C/min to 300 $^{\circ}$ C (5 min) Injector: 250 $^{\circ}$ C Transfer line temp: 280 $^{\circ}$ C Carrier: Helium, 1.0 mL/min Split ratio: 20/1
	The retention time of the free base derivative 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 (12.4 min): 217 (M^+ , 2), 215 (M^+ , 13), 188 (33), 186 (100), 173 (11), 171 (30), 155 (11), 99 (7), 91 (6), 77 (13) m/z
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 $^{\circ}$ C (5 min), 7 $^{\circ}$ C/min to 120 $^{\circ}$ C, 15 $^{\circ}$ C/min to 220 $^{\circ}$ C (8.3 min) Injector: 150 $^{\circ}$ C Transfer line temp: 280 $^{\circ}$ C Carrier: Helium, 1.2 mL/min Split ratio: 50/1 Solvents detected: Diethyl ether
TLC:	Conditions: Kieselgel 60F ₂₅₄ . Diethyl ether/TBME/diethyl amine (45:45:10) Single spot observed, R_f = 0.3. Visualisation with UV at 254 nm
IR:	Instrument: Biorad FTS3000MX FT-IR Range: 4000-400 cm^{-1} , KBr powder Peaks: 3216, 3005, 2938, 2900, 2841, 2737, 2698, 2605, 2449, 2037, 1502, 1492, 1464, 1394, 1308, 1210, 1033, 843, 786 cm^{-1}
^1H NMR:	Instrument: Bruker Avance-400 Field strength: 400 MHz Solvent: CD_3OD (3.31 ppm) Spectral data: δ 2.96 (2H, t, J = 7.4 Hz), 3.14 (2H, t, J = 7.4 Hz), 3.81 (3H, s), 3.84 (3H, s), 6.99 (1H, s), 7.03 (1H, s) ppm Diethyl ether estimated at 0.04% mass fraction was observed in the ^1H NMR
^{13}C NMR:	Instrument: Bruker Avance-400 Field strength: 101 MHz Solvent: CD_3OD (49.0 ppm) Spectral data: δ 29.6, 40.6, 56.6, 57.3, 114.2, 116.6, 122.6, 125.5, 150.6, 153.1 ppm
Melting point:	230-231 $^{\circ}$ C
Microanalysis:	Found: C = 47.8%; H = 5.9%; N = 5.7% Cl = 27.8% (September, 2013) Calc: C = 47.6%; H = 6.0%; N = 5.6% Cl = 28.1% (Calculated for $\text{C}_{10}\text{H}_{14}\text{ClNO}_2\text{HCl}$)

Expiration of certification

The property values are valid till 8th June 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.

The long-term stability of the compound in solution has not been examined.

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

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.

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.

Intended Use

For *in vitro* laboratory analysis only.

Caution

Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice

Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 17 June, 2016.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 17th June 2016.