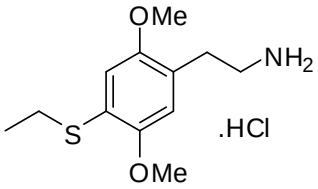
# REFERENCE MATERIAL PRODUCT INFORMATION SHEET

## NMIA D1040: 4-(Ethylthio)-2,5-dimethoxyphenethylamine hydrochloride

Report ID: D1040.2019.03

Chemical Formula: $C_{12}H_{19}NO_2S.HCl$

Molecular Weight: 277.8 g/mol (HCl), 241.3 g/mol (base)




## Property value

| Batch No. | CAS No. | Purity estimate |
|---|---|---|
| **14-D-40** | **681160-71-4** | **94.5 ± 2.4%** |

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

**Synonyms:** 4-(Ethylthio)-2,5-dimethoxy-benzeneethanamine, hydrochloride
2,5-Dimethoxy-4-(ethylthio)phenethylamine hydrochloride
2C-T-2 hydrochloride

**Expiration of certification:** The property values are valid till 4 January 2022, 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 solid 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 should be used 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:** 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.
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. The 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 elemental microanalysis.

| | | |
|---|---|---|
| GC-FID: | Instrument: | Varian CP-3800 |
| | Column: | HP-5, 30 m × 0.32 mm I.D. × 0.25 µm |
| | Program: | 160 °C (1 min), 5 °C/min to 220 °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 = 97.3%, s = 0.11% (10 sub samples in duplicate, May 2015) |
| | Re-analysis: | Mean = 97.6%, s = 0.03% (5 sub samples in duplicate, November 2017) |
| | Re-analysis: | Mean = 97.5%, s = 0.06% (5 sub samples in duplicate, January 2019) |
| Thermogravimetric analysis: | | Volatile content < 0.1 % and non-volatile residue < 0.2 % mass fraction (July 2018) |
| Karl Fischer analysis: | | Moisture content 1.4% mass fraction (May 2015)<br>Moisture content 2.4% mass fraction (October 2017)<br>Moisture content 2.3% mass fraction (January 2019) |

## Spectroscopic and other characterisation data

| | | |
|---|---|---|
| GC-MS: | Parent compound: | |
| | Instrument: | Agilent 6890/5973 |
| | Column: | HP-1MS, 30 m x 0.25 mm I.D. x 0.25 µm |
| | Program: | 160 °C (1 min), 5 °C/min to 220 °C (2 min), 5 °C/min to 300 °C (5 min) |
| | Injector: | 250 °C |
| | Transfer line temp: | 280 °C |
| | Carrier: | Helium, 1.0 mL/min |
| | Split ratio: | 20/1 |
| | The retention times of the free base derivative are 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 (9.3 min): | 241 ($M^+$, 31), 212 (100), 211 (53), 197 (17), 183 (40), 153 (16) *m/z* |
| TLC: | Conditions: | Kieselgel 60$F_{254}$. Diethyl ether/TBME/ diethyl amine (45/45/10)<br>Single spot observed, $R_f$ = 0.38. Visualisation with UV at 254 nm |
| IR: | Instrument: | Bruker Alpha FT-IR |
| | Range: | 4000-400$cm^{-1}$, neat |
| | Peaks: | 2953, 2897, 1490, 1392, 1206, 1037, 849, 810 $cm^{-1}$ |
| $^1$H NMR: | Instrument: | Bruker Avance III-500 |
| | Field strength: | 500 MHz |
| | Solvent: | $d_4$-methanol (3.31 ppm) |
| | Spectral data: | δ 1.26 (3H, t, *J* = 7.5 Hz), 2.91 (2H, q, *J* = 7.5 Hz), 2.94 (2H, t, *J* = 7.5 Hz), 3.13 (2H, t, *J* = 7.5 Hz), 3.82 (3H, s), 3.83 (3H, s), 6.85 (1H, s), 6.91 (1H, s) ppm<br>Isopropanol estimated at 0.2% mass fraction was observed in the $^1$H NMR |
| $^{13}$C NMR: | Instrument: | Bruker Avance III-500 |
| | Field strength: | 126 MHz |
| | Solvent: | $d_4$-methanol (49.0 ppm) |
| | Spectral data: | δ 14.6, 27.0, 29.7, 40.8, 56.5, 57.0, 113.9, 115.2, 124.3, 125.9, 153.1, 153.2 ppm |
| Melting point: | | 176-181 °C |
| Microanalysis: | Found: | C = 50.6%; H = 7.2%; N = 5.0%; Cl = 13.1%; S = 11.2% (June, 2015) |
| | Calculated: | C = 51.9%; H = 7.3%; N = 5.0%; Cl = 12.8%; S = 11.5% (Calculated for $C_{12}H_{19}NO_2S.HCl$) |