



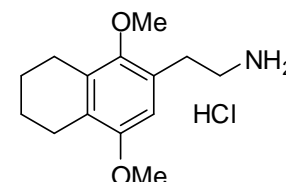
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1052: 2-(1,4-Dimethoxy-5,6,7,8-tetrahydronaphthalen-2-yl)ethylamine hydrochloride

Report ID: D1052.2016.04

Chemical Formula: C₁₄H₂₁NO₂.HCl

Molecular Weight: 271.8 g/mol (HCl), 235.2 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
15-D-06	952006-59-6 (base)	99.2 ± 0.7%

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

IUPAC name: 2-(1,4-Dimethoxy-5,6,7,8-tetrahydro-2-naphthalenyl)ethanamine

Expiration of certification: The property values are valid till 11 March 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 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 °C 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%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

Stability: In the absence of long term stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated 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

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
28 September 2022

This report supersedes any issued prior to 28 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. The certified purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include GC-FID, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR spectroscopy. 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

The certified purity value by qNMR was obtained using the one-proton singlet at 6.62 ppm measured against a certified internal standard of potassium hydrogen maleate.

Supporting evidence is provided by elemental microanalysis.

GC-FID: Instrument: Agilent 6890
Column: HP-1, 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 mass fraction of the main component:
Initial analysis: Mean = 99.8%, s = 0.02% (10 sub samples in duplicate, April 2015)
Re-analysis: Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, March 2016)

Karl Fischer analysis: Moisture content < 0.2% mass fraction (April 2015)
Moisture content 0.2% mass fraction (March 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 degradation at elevated temperatures.

QNMR: Instrument: Bruker Avance-III-500
Field strength: 500 MHz
Solvent: DMSO-*d*₆ (2.50 ppm)
Internal standard: Potassium hydrogen maleate (100.0% mass fraction)
Initial analysis: Mean (6.62 ppm) = 99.0%, s = 0.2% (5 sub samples, April 2015)

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 μ m
	Program:	160°C (1 min), 5 °C/min to 220 °C, 20 °C/min to 300 °C (5 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium
	Scan range:	50-550 <i>m/z</i>
	The retention time of the parent 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 (10.7 min):	235 (M^+ , 10), 206 (100), 204 (49), 191 (94), 175 (18), 115 (12), 91 (13) <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Diethyl ether/TBME/diethyl amine (45/45/10) Single spot observed, $R_f = 0.2$
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1} , neat
	Peaks:	3026, 2935, 1588, 1517, 1480, 1461, 1428, 1229, 1108, 946 cm^{-1}
¹ H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	MeOH- <i>d</i> ₄ (3.31 ppm)
	Spectral data:	δ 1.71-1.76 (4H, m), 2.58 (2H, t, <i>J</i> = 5.6 Hz), 2.73 (2H, t, <i>J</i> = 5.6 Hz), 2.95 (2H, t, <i>J</i> = 7.5 Hz), 3.15 (2H, t, <i>J</i> = 7.5 Hz), 3.70 (3H, s), 3.78 (3H, s), 6.61 (1H, s) ppm Methanol estimated at 0.01% mass fraction was observed in the ¹ H NMR
¹³ C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	MeOH- <i>d</i> ₄ (49 ppm)
	Spectral data:	δ 23.49, 23.54, 24.3, 25.0, 29.7, 41.6, 55.9, 61.0, 109.6, 127.1, 127.6, 133.1, 151.3, 155.3 ppm
Melting point:		247-251 °C
Microanalysis:	Found:	C = 62.0%; H = 8.3%; N = 5.2%; Cl = 12.9% (May, 2015)
	Calculated:	C = 61.9%; H = 8.2%; N = 5.2%; Cl = 13.0% (Calculated for C ₁₄ H ₂₁ NO ₂ .HCl)