



REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D1053.2015.02

Compound Name: 2-(1, 4-Dimethoxynaphthalene)ethanamine hydrochloride

Description: Off white solid

Collection Number: D1053

Chemical Formula: C₁₄H₁₇NO₂.HCl

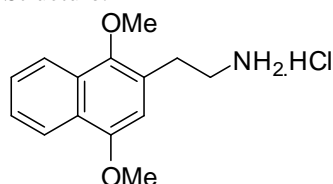
CAS Number: 207740-21-4 (free base)

Batch Number: 15-D-07

Molecular Weight: 267.8 (HCl), 231.1 (base)

Release date: 14th May 2015

Structure:



Synonyms: 2C-G-N hydrochloride

Purity (mass fraction): 98.4 ± 1.9% (95% coverage interval)

The purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (QNMR). The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR. The purity estimate by QNMR was obtained using the one proton singlet at 6.86 ppm, 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 peak area response of main component as the free base:
Initial analysis: Mean = 99.7%, s = 0.06% (10 sub samples in duplicate, April 2015)

Thermogravimetric analysis: Non volatile residue 0.3 % mass fraction (May 2015).

Karl Fischer analysis: Moisture content 0.2% mass fraction (April 2015)

QNMR: Instrument: Bruker Avance-III-500
Field strength: 500 MHz Solvent: d₆-DMSO (2.50 ppm)
Internal standard: Potassium hydrogen maleate (100% mass fraction)
Initial analysis: Mean (6.86 ppm) = 98.2%, s = 0.4% (6 sub samples, May 2015)

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 µm
	Program:	160 °C (1 min), 5 °C/min to 220 °C, 20 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Carrier:	Helium, 1.0 mL/min
		Transfer line temp: 280 °C
		Split ratio: 20/1
	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.	
	Free base (10.8 min): 231 (M^+ , 23), 202 (65), 187 (100), 128 (23), 115 (21) m/z	
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Diethyl ether/TBME/diethyl amine (45/45/10) Single spot observed, $R_f = 0.19$. Visualisation with UV at 254 nm
IR:	Instrument:	Bruker Alpha FT-IR
	Range:	4000-400 cm^{-1} , neat
	Peaks:	2899, 1591, 1522, 1458, 1445, 1372, 1265, 1103, 1084, 1000, 831, 765 cm^{-1}
^1H NMR:	Instrument:	Bruker Avance III 500
	Field strength:	500 MHz
	Spectral data:	Solvent: CD_3OD (3.31 ppm) δ 3.15 (2H, t, $J = 7.5$ Hz), 3.26 (2H, t, $J = 7.5$ Hz), 3.91 (3H, s), 4.01 (3H, s), 6.77 (1H, s), 7.47 (1H, dd, $J = 7.5, 8.4$ Hz), 7.56 (1H, dd, $J = 7.5, 8.4$ Hz), 8.03 (1H, d, $J = 8.4$ Hz), 8.19 (1H, d, $J = 8.4$ Hz) ppm
^{13}C NMR:	Instrument:	Bruker Avance III 500
	Field strength:	126 MHz
	Spectral data:	Solvent: CD_3OD (49.0 ppm) δ 29.6, 41.4, 56.3, 62.6, 106.2, 122.8, 123.4, 125.7, 126.4, 127.5, 127.9, 129.9, 148.9, 153.7 ppm
Melting point:	259-263°C	
Microanalysis:	Found: C = 62.8%; H = 6.8%; N = 5.3%; Cl = 13.0% (May, 2015) Calc: C = 62.8%; H = 6.8%; N = 5.2%; Cl = 13.2% (Calculated for $\text{C}_{14}\text{H}_{17}\text{NO}_2 \cdot \text{HCl}$)	

Expiration of certification

The property values are valid till 17th April 2018, i.e. three years from the date of 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 been given a shelf life of three years from the date of certification. The material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

In the absence of 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 10 years.

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 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:

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Dated: 7 March, 2016.