



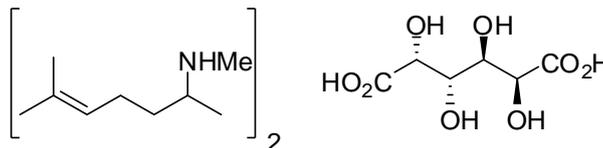
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D940: Isometheptene mucate

Report ID: D940.2019.03

Chemical Formula: C₂₄H₄₈N₂O₈

Molecular Weight: 492.7 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
09-D-07	7492-31-1	99.5 ± 0.5%

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

IUPAC name: N,6-Dimethyl-5-hepten-2-ammonium - D-galactarate

Expiration of certification: The property values are valid till 23 July 2029, i.e. ten 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 sourced from an external supplier, 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: 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: This material has demonstrated stability over a minimum period of 5 years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. 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 ¹H NMR on five randomly selected 20 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.
20 September 2022

This report supersedes any issued prior to 20 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 by quantitative nuclear magnetic resonance (qNMR). A combination of the three-proton doublet at 1.31 ppm, and the three-proton singlet at 2.68 ppm were measured against a certified internal standard of potassium hydrogen maleate or glycine.

Supporting evidence is provided by GC-FID, Karl-Fischer analysis, thermogravimetric analysis and elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	70 °C (15 min), 50 °C/min to 300 °C (5 min)
	Injector:	200 °C
	Carrier:	Helium
	Detector Temp:	320 °C
	Split ratio:	20/1
	Relative mass fraction of the main component as the free base:	
	Initial analysis:	Mean = 99.9%, s = 0.1% (10 sub samples in duplicate, April 2009)
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (February 2009 and May 2019)
Thermogravimetric analysis:		Volatiles content not determined due to the nature of the material. Non-volatile residue < 0.2% mass fraction (April 2009)
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	D ₂ O
	Internal standard:	Potassium hydrogen maleate
	Initial analysis:	Mean = 99.4%, s = 1.1% (3 sub samples, March 2009)
	Re-analysis:	Mean = 99.6%, s = 0.9% (5 sub samples, June 2010)
	Re-analysis:	Mean = 99.9%, s = 0.4% (5 sub samples, July 2013)
	Re-analysis:	Mean = 99.4%, s = 0.6% (5 sub samples, June 2016)
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	D ₂ O
	Internal standard:	Glycine (100.0% mass fraction)
	Initial analysis:	Mean (2.68 ppm) = 100.2%, s = 0.1% (4 sub samples, July 2019)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 5 μ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	Isometheptene 142 (M+H ⁺) <i>m/z</i>
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Negative ion mode, direct infusion at 5 μ L/min
	Ionisation:	ESI spray voltage at 3.2 kV negative ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	Mucate 209 (M-H ⁺) <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F254. diethyl ether/tert-butyl methyl ether/diethylamine (45/45/10) Single spot observed, R _f = 0.5. Visualisation with vanillin
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm ⁻¹ , neat
	Peaks:	3287, 3163, 2960, 2740, 2441, 1590, 1424, 1367, 1107, 1050, 971, 767 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D ₂ O (4.79 ppm)
	Spectral data:	δ 1.31 (6H, d, <i>J</i> = 6.6 Hz), 1.54-1.65 (2H, m), 1.64 (6H, s), 1.71 (6H, d, <i>J</i> = 0.7 Hz), 1.73-1.82 (2H, m), 2.11 (4H, m), 2.68 (6H, s), 3.22 (2H, m), 3.95 (2H, s), 4.25 (2H, s), 5.19 (2H, m) ppm
¹³ C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	D ₂ O
	Spectral data:	δ 14.9, 16.8, 23.1, 24.7, 29.6, 32.2, 55.0, 71.4, 71.7, 122.3, 134.7, 179.4 ppm
Melting point:		146-148 °C
Microanalysis:	Found:	C = 58.7%; H = 10.0%; N = 5.7% (February, 2009)
	Calculated:	C = 58.5%; H = 9.8%; N = 5.7% (Calculated for C ₂₄ H ₄₈ N ₂ O ₈)