



CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D940.2016.02

Compound Name: Isometheptene mucate

Collection Number: D940

Chemical Formula: $C_{24}H_{48}N_2O_8$

CAS Number: 7492-31-1

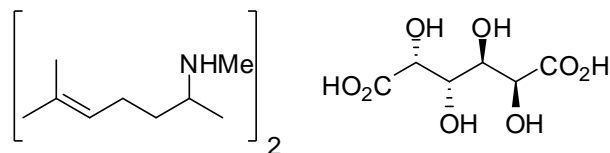
Structure:

Description: Off white solid

Batch Number: 09-D-07

Molecular Weight: 492.7

Release Date: 6th June 2009



Synonyms: Isometheptene galactarate
Octinum

Purity (mass fraction): 99.4 ± 2.3 % (95 % coverage interval)

The purity value was obtained by quantitative nuclear magnetic resonance (qNMR) analysis against an internal standard of potassium hydrogen maleate. Supporting evidence is provided by GC-FID, Karl Fischer analysis, thermogravimetric analysis and elemental microanalysis.

QNMR: Instrument: Bruker Avance-III-500
Field strength: 500 MHz Solvent: D₂O
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 = 99.7%, s = 0.9% (5 sub samples, June 2016)

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: 250 °C Detector Temp: 320 °C
Carrier: Helium Split ratio: 20/1
Relative peak area response of main component:
Initial analysis: Mean = 99.9%, s = 0.1% (10 sub samples in duplicate, April 2009)

Thermogravimetric analysis: Volatile contents not determined due to the nature of the material
Non volatile residue < 0.2 % mass fraction (April 2009)

Karl Fischer analysis: Moisture content 0.21% mass fraction (February 2009)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro Micro
	Operation:	Positive ion mode, direct infusion at 5 $\mu\text{L}/\text{min}$
	Ionisation:	ESI spray voltage at 3.2 kV negative ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	Isometheptene 142 ($\text{M} + \text{H}^+$) m/z
	Instrument:	Micromass Quatro Micro
	Operation:	Negative ion mode, direct infusion at 5 $\mu\text{L}/\text{min}$
	Ionisation:	ESI spray voltage at 3.2 kV negative ion
	EM voltage:	500 V
	Cone voltage:	20 V
	Peak:	Mucate 209 ($\text{M} - \text{H}^+$) m/z
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . diethyl ether/tert-butyl methyl ether/diethylamine (45/45/10) Single spot observed, $R_f = 0.52$. Visualisation with vanillin
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1} , KBr powder
	Peaks:	3287, 3163, 2960, 2740, 2441, 1590, 1424, 1367, 1107, 1050, 971, 767 cm^{-1}
¹ H NMR:	Instrument:	Avance-400
	Field strength:	400 MHz Solvent: D ₂ O (4.79 ppm)
	Spectral data:	δ 1.31 (6H, d, $J = 6.6$ Hz), 1.54-1.65 (2H, m), 1.64 (6H, s), 1.71 (6H, d, $J = 0.7$ 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:	Avance-400
	Field strength:	100 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 $^{\circ}\text{C}$
Microanalysis:		Found: C = 58.7 %; H = 10.0 %; N = 5.7% (February 2009)
		Calc: C = 58.5 %; H = 9.8 %; N = 5.7 % (Calculated for C ₂₄ H ₄₈ N ₂ O ₈)

Expiration of certification

The property values are valid till 23rd 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.

Metrological Traceability

The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. The purity was derived by subtraction of the mass of impurities from the mass of the reference material. Organic purity is traceable to the SI-derived coherent unit one through chromatographic separation and response factor determination of individual components. Volatile and non-volatile residue content is directly traceable to mass through use of Karl Fischer and thermogravimetric analysis. 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).

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: 14 July, 2016.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 14th July 2016.