



REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D903.2011.03

Compound Name: (2S*, 3R*)-2-Methyl-3-[3,4-(methylenedioxy)phenyl]-glycidic acid methyl ester

Description: White solid

Collection No: D903

Chemical Formula: C₁₂H₁₂O₅

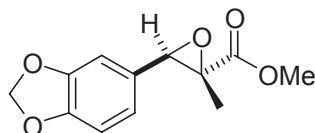
CAS No: N/A

Structure:

Batch No: 05-D-16

Molecular Weight: 236.2

Batch production completed: October 2005



Synonyms: *trans*-Methyl 2-methyl-3-[3,4-(methylenedioxy)phenyl]-glycidate

Purity (mass fraction): 99.7 ± 1.8% (95% coverage interval)

Purity estimate obtained by subtraction from 100% of total impurities by HPLC-PDA and Karl Fischer. Supporting evidence is provided by headspace GC-MS analysis of occluded solvents and elemental microanalysis.

HPLC Instrument: Waters Model 1525 Binary pump, 717 plus autosampler
Column: Alltech Alltima C18, 5µm (4.6 mm x 150 mm)
Mobile Phase: Acetonitrile/Water (1:1)
Flow rate: 1 mL/min
Detector: Waters PDA 996 at Max Plot
Relative peak area response of main component:
Initial analysis: Mean = 99.97%, s = 0.01 (7 samples in duplicate, October 2005)
Re-analysis: Mean = 99.91%, s = 0.02 (5 sub samples in duplicate, April 2008)
Current analysis: Mean = 99.92%, s = 0.01 (4 sub samples in duplicate, April 2011)

GC-MS: Instrument: HP 5890/5971A
Column: BPX-5, 30 m x 0.25 mm I.D. x 0.30 µm
Program: 100 °C (1 min), 10 °C/min to 250 °C, hold (5min)
Injector: 220°C Transfer line temp: 280 °C
Carrier: Helium, 1.0 mL/min Split ratio: 20/1

The retention time of the parent compound 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.

12.1min: 236 (M⁺, 8), 177 (14), 165 (100), 134 (39), 104 (24), 76 (46), 65 (14) m/z

TLC: Conditions: Kieselgel 60F₂₅₄. Hexane/ethyl acetate/methanol (70:29:1)
Single spot observed, R_f = 0.42. Visualisation with UV at 254 nm

IR: Instrument: Biorad FTS300MX FT-IR.
Range: 4000-400cm⁻¹, KBr pellet.
Peaks: 3072, 3000, 2955, 2923, 2845, 1742, 1509, 1496, 1449, 1406, 1307, 1293, 1252, 1197, 1166, 1094, 1033, 922, 814, 739, 591, 560 cm⁻¹

¹H NMR: Instrument: Bruker DMX-500
Field strength: 500 MHz Solvent: CDCl₃
Spectral data: δ 1.32 (3H, s), 3.80 (3H, s), 4.23 (1H, s), 5.96 (2H, s), 6.78 (3H, m) ppm

¹³C NMR: Instrument: Bruker DMX-500
Field strength: 125 MHz Solvent: CDCl₃
Spectral data: δ 12.5, 52.7, 59.9, 62.3, 101.2, 107.0, 108.2, 120.3, 127.5, 147.6, 147.7, 171.2 ppm

Melting point: 59-61 °C

Microanalysis: Found: C = 60.8%; H = 4.9% (September 2005)
Found: C = 61.2%; H = 5.2% (June 2007)
Calc: C = 61.0%; H = 5.1% (Calculated for C₁₂H₁₂O₅)

Thermogravimetric analysis: Volatile content < 0.1% mass fraction at 50 °C and non volatile residue < 0.2% mass fraction (December 2005 & March 2007).

Karl Fischer analysis: Moisture content 0.7% mass fraction (May 2007)
Moisture content 0.5% mass fraction (April 2008)
Moisture content < 0.1% mass fraction (April 2011)

Expiration of certification

The property values are valid till 12th April 2016, i.e. five 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.

In the absence of stability data the measurement uncertainty at the 95% confidence 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 HPLC-UV on 4 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 20 °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: 23 July, 2012.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 23rd July 2012.



This document is issued in accordance with NATA's accreditation requirements.

Accredited for compliance with ISO Guide 34.

This document shall not be reproduced except in full.

Accreditation Number : 198

Corporate Site Number : 14214

