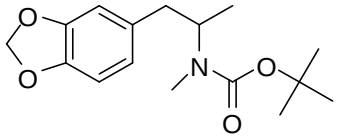
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1069: (±)-N-tert-Butoxycarbonyl-3,4-methylenedioxy-methamphetamine



Report ID: D1069.2023.01

Chemical Formula: $C_{16}H_{23}NO_4$

Molecular Weight: 293.4 g/mol

Certified value

Batch No.	CAS No.	Purity (mass fraction)
17-D-03	**1228259-70-8**	**99.6 ± 0.4%**

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

IUPAC name: 2-Methyl-2-propanyl [1-(1,3-benzodioxol-5-yl)-2-propanyl]methylcarbamate.

Expiration of certification: The property values are valid till 25 August 2028, 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.

Description: Clear oil 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 ºC 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%.

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 10 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 seven 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.
29 August 2023

This report supersedes any issued prior to 29 August 2023.

NATA Accreditation No. 198 / Corporate Site No. 14214.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100 % - I_{ORG}) x (100 % - I_{VOL} – I_{NVR}) Equation 1

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

Warning: **This material is sensitive to the quality of the silanised glass liner when injected at elevated temperature (~ 250 ºC) into a GC instrument.**

GC-FID:	Instrument:	Varian CP-3800/Agilent 8890
	Column:	VF-1MS/HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 20 °C/min to 180 °C (6 min), 20 °C/min to 300 °C (3 min)
	Injector:	180 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.8%, s = 0.01% (7 sub samples in duplicate, March 2017)
	Re-analysis:	Mean = 99.9%, s = 0.02% (5 sub samples in duplicate, April 2018)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, April 2019)
	Re-analysis:	Mean = 99.8%, s = 0.05% (5 sub samples in duplicate, July 2021)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, August 2023)
Karl Fischer analysis:		Moisture content 0.5% mass fraction (March 2017 and April 2018) Moisture content 0.3% mass fraction (April 2019, August 2021) Moisture content 0.3% mass fraction (April 2019, August 2023)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (March 2017). The volatile content (e.g. organic solvents and/or water) could not be determined by thermogravimetric analysis.

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	HP-1MS, 30 m × 0.25 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 300 °C (3 min)
	Injector:	180 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	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.	
	Parent (14.1 min):	293 (M^+, 1), 220 (5), 163 (6), 158 (13), 135 (27), 102 (33), 58 (100), 41 (49) *m/z*
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μL/min
	Ionisation:	ESI spray voltage at 3.0 kV positive ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	316.1 ($M+Na^+$) *m/z*
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m × 0.25 mm I.D. × 1.4 μm
	Program:	50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)
	Injector:	150 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	None
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate (1:9) Single spot observed, R_f = 0.4. Visualisation with UV at 254 nm and/or vanillin stain.
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2974, 1684, 1489, 1441, 1345, 1334, 1244, 1141, 1038, 929, 802, 770 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm) at 340K
	Spectral data:	δ 1.08 (3H, d, *J* = 6.9 Hz), 1.30 (9H, s), 2.62 (3H, s), 2.59 (1H, dd, *J* = 6.3, 13.9 Hz), 2.64 (1H, dd, *J* = 8.9, 13.9 Hz), 4.24 (1H, m), 5.92 (2H, s), 6.61 (1H, dd, *J* = 1.3, 7.8 Hz), 6.71 (1H, d, *J* = 1.3 Hz), 6.77 (1H, d, *J* = 7.9 Hz) ppm Ethyl acetate estimated at 0.03% and dichloromethane at 0.12% mass fraction was observed in the ^{1}H NMR
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	DMSO-d_6 (39.5 ppm) at 340K
	Spectral data:	δ 17.5, 27.6, 27.8, 39.0*, 51.6, 77.9, 100.3, 107.6, 108.9, 121.5, 132.8, 145.2, 146.8, 154.4 ppm *Observed *via* HSQC correlation
Melting point:		N/A
Microanalysis:	Found:	C = 65.4%; H = 7.7%; N = 4.6% (March 2017)
	Calculated:	C = 65.5%; H = 7.9%; N = 4.8% (Calculated for $C_{16}H_{23}NO_4$)