Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

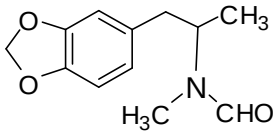
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D538: N-Formyl-3,4-methylenedioxymethamphetamine

Report ID: D538.2021.02

Chemical Formula: $C_{12}H_{15}NO_3$

Molecular Weight: 221.3 g/mol



Property value

Batch No.	CAS No.	Purity by GC-FID
97-000923	**154148-22-8**	**98%**

IUPAC name: *N*-[1-(1,3-Benzodioxol-5-yl)-2-propanyl]-N-methylformamide

Expiration of certification: The property values are valid till 17 August 2026, 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.

Description: Light amber waxy solid/oil prepared by synthesis, 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 material should be considered for qualitative or semi quantitative work only.

Recommended storage: When not in use, this material should be stored at or below 4 ºC in a closed container in a dry, dark area.

Stability: This material has demonstrated stability over a minimum period of five 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 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.

Safety: Treat as 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.
September 14, 2022

This report supersedes any issued prior to 14 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 purity value was obtained by GC-FID.

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

GC-FID:	Instrument:	Agilent 6890N or 8890
	Column:	HP-1 Capillary, 30 m x 0.32 mm I.D. x 0.25 µm
	Program:	80 °C (2 min), 15 °C/min to 300 °C (3 min)
	Injector Temp:	150 °C
	Detector Temp:	320 °m
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.9 %, s = 0.10% (7 sub samples in duplicate, June 1998)
	Re-analysis:	Mean = 98.6 %, s = 0.13% (5 sub samples in duplicate, November 2009)
	Re-analysis:	Mean = 98.9 %, s = 0.08% (7 sub samples in duplicate, September 2014)
	Re-analysis:	Mean = 98.8 %, s = 0.126% (5 sub samples in duplicate, August 2021)
Karl Fischer analysis:		Moisture content 0.3% mass fraction (November 2009) Moisture content 0.9% mass fraction (September 2014) Moisture content 0.8% mass fraction (August 2021)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5970B
	Ionisation:	EI, 70 eV
	Scan Range:	40-450 *m/z*
	Column:	HP Ultra-2, 12 m x 0.22 mm I.D. x 0.1 μm film thickness
	Temp Program:	70 °C to 300 °C at 10 °C/min
	Injector Temp:	230 °C
	Transfer line temp:	280 °C
	Carrier gas:	Helium, 1 mL/min
	Split ratio:	10/1
	Peaks:	162 (100), 86 (98), 58 (98), 135 (30), 51 (25), 221 (10) *m/z*
HPLC:	Method:	Peak area percentage of total, triplicate mean > 99.8%, s < 0.2%
	Column:	Alltima C-18, 5 µm x 4.6 mm x 150 mm
	Mobile Phase:	Methanol/water (80/20)
	Flow Rate:	0.8 mL/min
	Wavelength:	290 nm
TLC:	Conditions:	Kieselgel 60F_{254}. Diisopropylether/diethylether/diethylamine (45/45/10). Single spot observed, R_f = 0.68
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-500 cm^{-1}, liquid film on KCl
	Peaks:	1665, 1490, 1441, 1405, 1324, 1247, 1190, 1036, 920, 810 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Key spectral data:	δ 1.2 (3H, d, *J* =7.2 Hz), 2.6 (2H, m), 2.8 (3H, s), 3.7 (1H, m), 5.9 (2H, s), 6.6 (3H, m), 7.8 (1H, s) ppm Ethyl acetate was observed at 0.2% mass fraction in the ^{1}H NMR (in chloroform-d_1, December 2006) and 0.51% mass fraction of chloroform in the ^{1}H NMR (in DMSO-d_6, January 2010)