Australian Government
Department of Industry, Science and Resources

National Measurement Institute

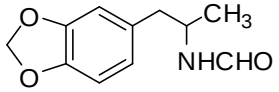
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D498: (±)-N-Formyl-3,4-methylenedioxyamphetamine

Report ID: D498.2021.02

Chemical Formula: $C_{11}H_{13}NO_3$

Molecular Weight: 207.2 g/mol



Property value

Batch No.	CAS No.	Purity estimate
97-000052	**67669-00-5**	**99.9 ± 0.3%**

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

Expiration of certification: The property values are valid till 11 November 2031, 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: White crystals prepared by synthesis or sourced from an external supplier, 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 reference material should be used for qualitative analysis only.

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 4 ºC in a closed container in a dry, dark area.

Stability: This material has demonstrated stability over a minimum period of ten 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 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.
14 September 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 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 elemental microanalysis.

GC-FID:	Column:	HP-1 Capillary, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	120 °C (1 min), 10 °C/min to 180 °C (5 min), 30 °C/min to 300 °C (3 min)
	Injector Temp:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.1% (7 sub samples in duplicate, August 1997)
	Re-analysis:	Mean = 99.8%, s = 0.1% (3 sub samples in duplicate, September 2004)
	Re-analysis	Mean = 99.9%, s = 0.003% (5 sub samples in duplicate, September 2007)
	Re-analysis	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, August 2012)
	Re-analysis	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, May 2017)
	Re-analysis	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, November 2021)
Karl Fischer analysis:		Moisture content ≤ 0.1% mass fraction (September 2007and August 2012) Moisture content < 0.1% mass fraction (May 2017 and November 2021)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5970B
	Ionisation:	EI/70 eV
	Scan Range:	400 – 450 *m/z*
	Column:	HP Ultra-2, 12 m x 0.22 mm I.D. x 0.1 μm
	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
	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.	
	Parent (10.5 min):	207(M^+, 20) 178 (10), 162(100), 147 (20), 135(73), 121 (20), 105 (10), 77 (32), 72 (50), 51 (23), 44 (56) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/acetone (75/25) Single spot observed, R_f = 0.8
IR:	Instrument:	FT-IR, BioRad WIN FTS40
	Range:	4500 - 500 cm^{-1}, KBr pellet
	Peaks:	3305, 1655, 1539, 1501, 1484, 1440, 1394, 1241, 1040, 937 cm^{-1}
^{1}H NMR:	Field Strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Peaks	δ 1.1 (3H, m), 2.7 (2H, m), 3.7 (1H, m) 4.3 (1H, m), 5.9 (2H, s), 6.6 (3H, m), 8.1 (1H, s) ppm
Melting Point:		91 - 93 °C