Australian Government
Department of Industry, Science and Resources

National Measurement Institute

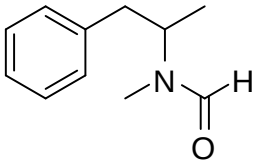
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D502b: N-Formylmethamphetamine

Report ID: D502b.2022.02

Chemical Formula: $C_{11}H_{15}NO$

Molecular Weight: 177.2 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
15-D-05	**42932-20-7**	**96.2 ± 1.0%**

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

IUPAC: *N*-Methyl-*N*-(1-phenyl-2-propanyl)formamide

Expiration of certification: The property values are valid till 14 February 2027, 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: Pale yellow 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 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: 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. 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 ten 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 qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800 or Agilent 6890N	
	Column:	VF-1MS or HP-1, 30 m × 0.32 mm I.D. × 0.25 μm	
	Program:	120 °C (1 min), 10 °C/min to 150 °C (3 min), 30 °C/min to 300 °C (3 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative mass fraction of the main component:		
	Initial analysis:	Mean = 99.6%, s = 0.01% (10 sub samples in duplicate, March 2015)	
	Re-analysis:	Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, February 2016)	
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, March 2017)	
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, February 2018)	
	Re-analysis:	Mean = 99.6%, s = 0.03% (5 sub samples in duplicate, May 2019)	
	Re-analysis:	Mean = 99.5%, s = 0.01% (5 sub samples in duplicate, February 2022)	
Thermogravimetric analysis:		Non-volatile residue < 0.2% mass fraction (March 2015). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material and/or degradation at elevated temperatures.	
Karl Fischer analysis:		Moisture content 2.9% mass fraction (March 2015) Moisture content 2.1% mass fraction (February 2016) Moisture content 2.2% mass fraction (February 2017) Moisture content 2.0% mass fraction (March 2018) Moisture content 2.1% mass fraction (March 2019) Moisture content 2.6% mass fraction (February 2022)	

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	HP-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Transfer line temp:	300 °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/spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (11.0 min):	177 (M^+, 5), 118 (48), 91 (58), 86 (100), 58 (97) *m/z*
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 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:	Dichloromethane
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/acetone (1/1) Single spot observed, R_f = 0.7. Visualisation with UV at 254 nm
IR:	Instrument:	Bruker Alpha FT-IR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	3481, 3027, 2972, 2933, 2859, 1657, 1494, 1453, 1429, 1326, 1245, 1111, 1075, 1050, 746, 700, 629, 525 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III 500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (Spectrum referenced to residual DCM at 5.30 ppm)
	Spectral data:	This compound appears as two rotomers in a ratio of 2.7:1
	Minor rotomer	δ 1.17 (3H, d, *J* = 6.8 Hz), 2.73-2.88 (2H, m), 2.79 (3H, s), 3.80 (1H, m), 7.10-7.26 (3H, m), 7.26-7.33 (2H, m), 7.96 (1H, s) ppm
	Major rotomer	δ 1.31 (3H, d, *J* = 6.8 Hz), 2.73-2.88 (2H, m), 2.81 (3H, s), 4.79 (1H, m), 7.10-7.26 (3H, m), 7.26-7.33 (2H, m), 7.79 (1H, s) ppm
		Dichloromethane and ethanol estimated at 0.6% and 0.2% mass fraction respectively were observed in the ^{1}H NMR.
^{13}C NMR:	Instrument:	Bruker Avance III 500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 16.9, 19.1, 25.0, 29.9, 39.7, 40.8, 48.1, 56.1, 126.5, 126.9, 128.5, 128.8, 128.9, 129.0, 137.9, 138.1, 162.6, 162.8 ppm
Microanalysis:	Found:	C = 73.2%; H = 8.6%; N = 7.9% (March, 2015)
	Calculated:	C = 74.5%; H = 8.5%; N = 7.9% (Calculated for $C_{11}H_{15}NO$)