Australian Government
Department of Industry, Science and Resources
National Measurement Institute
NATA WORLD RECOGNISED ACCREDITATION

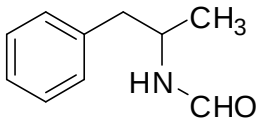
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D447c: (±)-N-Formylamphetamine

Report ID: D447c.2023.01

Chemical Formula: $C_{10}H_{13}NO$

Molecular Weight: 163.2 g/mol



Property value

Batch No.	CAS No.	Purity estimate
11-D-20	**22148-75-0**	**96.1 ± 2.6%**

IUPAC name: N-(1-Phenyl-2-propanyl)formamide

Expiration of certification: The property values are valid till 22 November 2033, 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: Clear liquid prepared by synthesis, and certified for identity and purity by NMIA. Packaged in sealed amber glass ampoules.

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 ten randomly selected 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.

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.
7 December 2023

This report supersedes any issued prior to 7 December 2023.

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:

This reference material has NOT been extensively quantified by the Chemical Reference Materials team, NMI. This material should be considered for use in qualitative analysis only.

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The 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:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 20 °C/min to 120 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 97.0%, s = 0.02% (10 samples in duplicate, February 2012)
	Re-analysis:	Mean = 97.0%, s = 0.02% (7 sub samples in duplicate, March 2013)
	Re-analysis;	Mean = 97.2%, s = 0.01% (5 sub samples in duplicate, February 2014)
	Re-analysis;	Mean = 97.2%, s = 0.03% (6 sub samples in duplicate, January 2017)
	Re-analysis;	Mean = 97.4%, s = 0.04% (5 sub samples in duplicate, November 2019)
	Re-analysis;	Mean = 97.1%, s = 0.01% (5 sub samples in duplicate, November 2023)
Thermogravimetric analysis:		The volatile content could not be determined because of the inherent volatility of the material and non-volatile residue < 0.2% mass fraction (February 2012)
Karl Fischer analysis:		Moisture content 1.1% mass fraction (February 2012) Moisture content 2.5% mass fraction (February 2013) Moisture content 0.9% mass fraction (November 2023)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1ms, 30 m × 0.25 mm I.D. × 0.25 μm
	Program:	60 °C (1min), 10 °C/min to 300 °C (3 min)
	Injector:	250 °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.	
	11.8 min:	161 (M^+- 2), 118 (100), 117 (22), 91 (49), 72 (94), 65 (16), 44 (62) *m/z*
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-700 cm^{-1}, neat oil
	Peaks:	1659, 1532, 1496, 1450, 1380, 1243, 769, 733 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 1.11 (2.3H, d, *J* = 6.6 Hz), 1.23 (0.7H, d, *J* = 6.6 Hz), 2.63-2.83 (2H, m), 3.69 (0.2H, sep), 4.31 (0.7H, sep), 5.56 (0.8H, bs), 5.76 (0.2H, bs), 7.10-7.28 (5H, m), 7.76 (0.2H, d, *J* = 12 Hz), 8.03 (0.7H, s) ppm
		Doubling of the signals is due to restricted rotation about the amide bond.
^{13}C NMR:	Instrument:	Bruker Avance III -400
	Field strength:	100 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data:	δ 20.1, 22.0, 42.4, 44.6, 45.1, 50.0, 126.7, 127.0, 128.5, 128.8, 129.5, 137.3, 137.7, 160.6, 163.8 ppm
		Doubling of the signals is due to restricted rotation about the amide bond.
Microanalysis:	Found:	C = 72.8%; H = 8.3%; N = 8.5% (February 2012)
	Calculated:	C = 72.8%; H = 8.1%; N = 8.5% (Calc. for $C_{10}H_{13}NO$. $0.1H_2O$)
	Calculated:	C = 73.6%; H = 8.0%; N = 8.6% (Calc. for $C_{10}H_{13}NO$)