Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

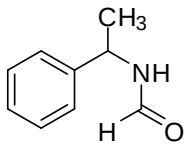
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D526: N-Formyl-1-phenylethylamine

Report ID: D526.2020.03

Chemical Formula: $C_9H_{11}NO$

Molecular Weight: 149.2 g/mol



Property value

Batch No.	CAS No.	Purity estimate
98-000301	**6948-01-2**	**99.6 ± 0.3%**

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

IUPAC name: *N*-(1-Phenylethyl)formamide

Expiration of certification: The property values are valid till 30 June 2030, 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: Pale yellow solid 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 may be used for instrument calibration.

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.

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 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 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.
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 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:	Instrument:	Agilent 6890N, 7890 or Varian CP3800
	Column:	HP-1 or VF-1MS, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 250 °C, 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 > 99.9%, s = 0.1% (7 sub samples in duplicate, March 1998)
	Re-analysis:	Mean = 99.4%, s = 0.13% (5 sub samples in duplicate, June 2007)
	Re-analysis:	Mean = 99.3%, s = 0.14% (5 sub samples in duplicate, June 2010)
	Re-analysis:	Mean = 99.7%, s = 0.07% (5 sub samples in duplicate, June 2015)
HPLC:	Column:	Econosphere C-18, 5 μm (4.6 mm x 250 mm)
	Mobile Phase:	Methanol/MilliQ water (60:40) The aqueous phase was buffered at pH 8.5 using 0.5% diethylamine
	Flow rate:	1.5 mL/min
	Detector:	UV operating at 260 nm
	Relative peak area response of main component:	
	Initial analysis:	Mean = 98.9%, s = 0.1% (3 sub samples in duplicate, May 1998)
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (June 2007, 2010, 2015 and July 2020)
Thermogravimetric analysis:		Non-volatile residue < 0.2% mass fraction (June 2007) Volatile content not determined due to volatility of the material

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP6890/5973
	Ionisation:	EI/70ev
	Column:	DB-1MS, 25 m x 0.2 mm I.D. x 0.33 μm
	Program:	100 °C (1 min), 10 °C/min to 250 °C, 25 °C to 300 °C (1 min)
	Injector:	250 °C
	Transfer line:	310 °C
	Carrier:	Helium, 1.8 mL/min
	Split:	20/1
	Scan Range:	35-450 *m/z*
	No published reference spectrum for *N*-formyl-1-phenylethylamine is available. The retention time of the material is reported along with the major peaks in the mass spectrum. The latter are reported in mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	6.9 min:	149 (M^+, 100), 148 (82), 134 (68), 105 (27), 104 (46), 91 (9), 79 (60), 77 (54), 51 (20), 42 (16) *m/z*, consistent with expected fragmentation pattern.
TLC:	Conditions:	Kieselgel 60F254 plate, solvent system containing ethyl acetate/acetone/ammonia (60/35/2.5); Single spot observed under UV,Rf = 0.50.
IR:	Instrument:	FT-IR, BIORAD WIN FTS40
	Range:	4000 - 400 cm^{-1}, KBr pellet
	Peaks:	1660, 1533, 1452, 1381, 1238, 762, 699 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ Major rotamer: 1.50 (3H, d, *J* = 7.0 Hz), 4.64-4.70 (1H, m,), 6.14 (1H, s), 7.26-8.13 (5H, m,), 8.11-8.13 (1H, m) ppm δ Minor rotamer: 1.54 (3H, d, *J* = 7.0 Hz), 5.16-5.22 (1H, m,), 6.27 (1H, s) 7.26-8.13 (5H, m,), 8.11-8.13 (1H, m) ppm Product was observed as two rotamers in a ratio of ~4:1
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77.0 ppm)
	Spectral data:	δ Major rotamer: 21.7, 47.5, 126.1, 127.5, 128.7, 142.5, 160.3 ppm δ Minor rotamer: 23.5, 51.6, 125.7, 127.7, 128.9 142.8, 164.1 ppm
Microanalysis:	Found:	C = 72.8%; H = 7.4% (June 2007)
	Calculated:	C = 72.5%; H = 7.4% (Calculated for $C_9H_{11}NO$)