Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

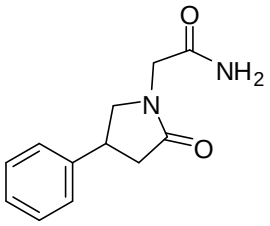
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D891b: Carphedon

Report ID: D891b.2020.01 (Bottled 150909)

Chemical Formula: $C_{12}H_{14}N_2O_2$

Molecular Weight: 218.3 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
13-D-32	**77472-70-9**	**98.9 ± 0.5%**

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

IUPAC name: 2-(2-Oxo-4-phenyl-1-pyrrolidyl) acetamide

Expiration of certification: The property values are valid till 15 July 2025, 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: Off-white powder 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 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% confidence 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 a 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.
21 July 2020

This report supersedes any issued prior to 21 July 2020

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 20844. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

Legal notice: Neither NMIA as a representative of the Commonwealth of Australia, nor any person acting on NMIA's behalf, assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this document.

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:	Agilent 7890A
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	180 °C (1 min), 5 °C/min to 240 °C, 20 °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 = 99.5%, s = 0.10% (10 sub samples in duplicate, December 2013)
GC-FID:	Instrument:	Agilent 7890A
	Column:	HP-1MS, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	180 °C (1 min), 5 °C/min to 240 °C, 20 °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 = 99.5%, s = 0.12% (10 sub samples in duplicate, December 2013)
	Re-analysis:	Mean = 99.4%, s = 0.05% (5 sub samples in duplicate, November 2014)
	Re-analysis:	Mean = 99.5%, s = 0.01% (5 sub samples in duplicate, August 2017)
	Re-analysis:	Mean = 99.4%, s = 0.05% (5 sub samples in duplicate, July 2020)
Thermogravimetric analysis:		Volatile content 0.3% and non volatile residue < 0.2% mass fraction (November 2013)
Karl Fischer analysis:		Moisture content 0.2% mass fraction (December 2013) Moisture content 0.2% mass fraction (March 2014) Moisture content 0.3% mass fraction (October 2014) Moisture content 0.4% mass fraction (August 2017) Moisture content 0.5% mass fraction (June 2020)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	180 °C (1 min), 5 °C/min to 240 °C, 20 °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. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (8.71 min):	218 (M^+, 15), 200 (13), 175 (13), 174 (65), 160 (57), 145 (34), 129 (20), 117 (35), 115 (14), 105 (18), 104 (100), 103 (24), 91 (20), 78 (16), 77 (19) *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:	Hexane
TLC:	Conditions:	Kieselgel 60F254. Chloroform/methanol (9/1) Single spot observed, Rf = 0.39. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3334, 3177, 1699, 1686, 1668, 1447, 1384, 1320, 1297, 1260, 1203, 1161, 1082, 1051, 1035, 947, 931, 911, 753, 721, 699, 644 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Solvent:	CD_3OD (3.31 ppm)
	Spectral data:	δ 2.59 (1H, dd, *J* = 8.7, 16.9 Hz), 2.84 (1H, dd, *J* = 9.1, 16.8 Hz), 3.55 (1H, dd, *J* = 7.5, 9.4 Hz), 3.70 (1H, quintet, *J* = 8.7 Hz), 3.84 (1H, dd, *J* = 8.6, 9.2 Hz), 4.02 (1H, d, *J* = 16.7 Hz), 4.06 (1H, d, *J* = 16.7 Hz), 7.24 (1H, m), 7.31-7.33 (4H, m) ppm Hexane estimated at 0.1% mass fraction was observed in the ^{1}H NMR
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	125 MHz
	Solvent:	$CDCl_3$
	Spectral data:	δ 37.3, 38.3, 46.3, 55.4, 126.7, 127.2, 128.9, 141.7, 170.4, 174.9 ppm.
Melting point:		125-129 °C
Microanalysis:	Found:	C = 66.1%; H = 6.5%; N = 12.9% (December 2013)
	Calculated:	C = 66.0%; H = 6.5%; N = 12.8% (Calculated for $C_{12}H_{14}N_2O_2$)