Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

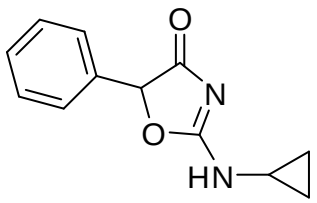
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D914: Cyclazodone

Report ID: D914.2024.01 (Bottled 191016)

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

Molecular Weight: 216.2 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
06-D-15	**14461-91-7**	**99.6 ± 0.4%**

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

IUPAC name: 2-(Cyclopropylamino)-5-phenyl-1,3-oxazol-4(5H)-one.

Expiration of certification: The property values are valid till 14 February 2034, 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 powder prepared by synthesis and 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.
16 February 2024

This report supersedes any issued prior to 16 February 2024.

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 elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890N
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 15 °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 = 0.01% (7 sub samples in duplicate, January 2007)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, January 2010)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, November 2014)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, October 2019)
	Re-analysis:	Mean = 99.7%, s = 0.03% (5 sub samples in duplicate, February 2024)
Thermogravimetric analysis:		Volatile content < 0.1% and non volatile residue < 0.2 % mass fraction
Karl Fischer analysis:		Moisture content < 0.3% mass fraction (January 2008, 2009 & 2010, October 2014 September 2019, and February 2024)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 6890/5973
	Column:	ZB-5, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	100 °C (1 min), 15 °C/min to 300 °C, (2 min)
	Injector:	200°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 to charge ratios and (in brackets) as a percentage relative to the base peak.	
	10.9 min:	216 (M+, 6), 189 (6), 132 (17), 118 (100), 90 (26), 77 (25), 68 (24), 55 (25) *m/z*
TLC:	Conditions:	Kieselgel 60F254. Dichloromethane / methanol (90/10) Single spot observed, Rf = 0.6. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	3251, 3036, 2945, 1745, 1651, 1485, 1387, 1342, 1256, 1199, 1013, 816, 715 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz
	Solvent:	DMSO-d_6 (2.5 ppm)
	Spectral data:	δ 0.64 (2H, m), 0.75 (2H, m), 2.82 (1H, m), 5.73 (0.58H, s), 5.79 (0.42H, s), 7.28 – 7.43 (5H, m), 9.13 (0.59H, bs), 9.44 (0.41H, bs) ppm. (2 rotational isomers observed)
^{13}C NMR:	Instrument:	Bruker DMX600
	Field strength:	125 MHz
	Solvent:	DMSO-d_6 (39.5 ppm)
	Spectral data:	δ 5.88, 5.91, 6.0, 23.0, 24.9, 82.1, 82.8, 126.2, 126.3, 128.7, 134.4, 134.5, 176.4, 185.4, 185.9 ppm. Two rotational isomers observed) in the NMR spectrum.
Melting point:		138-143°C
Microanalysis:	Found:	C = 67.0 %; H = 5.7 %; N = 13.0 % (December 2006)
	Calculated:	C = 66.7 %; H = 5.6 %; N = 13.0 % (Calculated for $C_{12}H_{12}N_2O_2$)