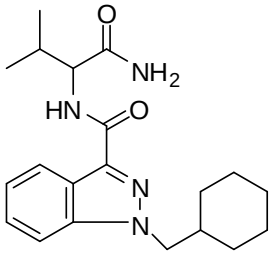
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D1049: AB-CHMINACA



Report ID: D1049.2022.01

Chemical Formula: $C_{20}H_{28}N_4O_2$

Molecular Weight: 356.5 g/mol

Property value

Batch No.	CAS No.	Purity estimate
15-D-01	**1185887-21-1**	**95.6 ± 0.5%**

IUPAC name: *N*-[(2S)-1-Amino-3-methyl-1-oxo-2-butanyl]-1-(cyclohexylmethyl)-1H-indazole-3-carboxamide.

Expiration of certification: The property values are valid till 28 September 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: White powder sourced from an external supplier, 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 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 25 ºC in a closed container in a dry, dark area.

Stability: In the absence of long term stability data the stability of this material has been judged from stability trials conducted on similar materials by NMI Australia over the last ten years.This material has demonstrated stability over a minimum period of three 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 HPLC with UV detection 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.
13 October 2022

This report supersedes any issued prior to 13 October 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 purity value was obtained by mass balance from a combination of traditional analytical techniques, including HPLC with UV detection, 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.

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT auto sampler
	Column:	Grace Alltima C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	35 °C
	Mobile Phase:	Acetonitrile/MilliQ water (60:40 v/v) A = MilliQ water; B = Acetonitrile
	Flow rate:	0.8 mL/min
	Detector:	Shimadzu SPD-M20A PDA operating at 300 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 98.6%, s = 0.01% (10 sub samples in duplicate, March 2015)
	Re-analysis:	Mean = 98.6%, s = 0.01% (5 sub samples in duplicate, February 2016)
	Re-analysis:	Mean = 98.6%, s = 0.02% (5 sub samples in duplicate, February 2017)
	Re-analysis:	Mean = 98.5%, s = 0.01% (5 sub samples in duplicate, January 2018)
	Re-analysis:	Mean = 98.4%, s = 0.01% (5 sub samples in duplicate, September 2022)
Karl Fischer analysis:		Moisture content 1.2-1.4% mass fraction (March 2015, February 2016, March 2017, January 2018 and September 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.

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:	100 °C (1 min), 15 °C/min to 280 °C (10 min), 30 °C/min to 300 °C (10 min)
	Injector:	250 °C,
	Split ratio:	30/1
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the parent compound is reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (17.8 min):	356 (M^+, 2), 312 (100), 241 (100), 145 (78), 131 (19), 103 (15), 55 (23) *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:	Chloroform
TLC:	Conditions:	Kieselgel 60F_{254}. Dichloromethane/methanol (20:1) Single spot observed, R_f = 0.4
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2924, 2851, 1649, 1525, 1490, 1175, 778, 748 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 0.89 (3H, d, *J* = 6.8 Hz), 0.94 (3H, d, *J* = 6.8 Hz), 0.98-1.24 (5H, m), 1.47 (2H, bdd, *J* = 14.4, 21.1 Hz), 1.53-1.73 (3H, m), 1.92 (1H, m), 2.09 (1H, octet, *J* = 6.7 Hz), 4.34 (2H, d, *J* = 7.1 Hz), 4.42 (1H, dd, *J* = 6.3, 9.0 Hz), 7.25 (1H, s), 7.26 (1H, t, *J* = 7.7 Hz), 7.44 (1H, m), 7.68 (1H, s), 7.69 (1H, d, *J* = 8.7 Hz), 7.77 (1H, d, *J* = 8.7 Hz), 8.15 (1H, d, *J* = 8.2 Hz) ppm
		Chloroform estimated at 1.6% mass fraction was observed in the ^{1}H NMR
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 18.0, 19.4, 25.1, 25.15, 25.8, 30.0, 30.1, 31.4, 38.5, 54.6, 56.8, 110.7, 121.6, 121.8, 122.5, 126.7, 136.4, 141.2, 161.4, 172.7 ppm
Melting point:		73-76 °C
Microanalysis:	Found:	C = 65.4%; H = 7.8%; N = 15.1% (March, 2015)
	Calculated:	C = 67.4%; H = 7.9%; N = 15.7% (Calculated for $C_{20}H_{28}N_4O_2$)