Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

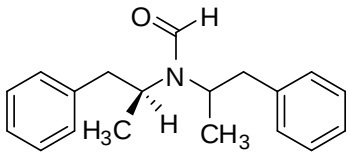
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D465: Di-(β-phenylisopropyl)formamide

Report ID: D465.2021.02

Chemical Formula: $C_{19}H_{23}NO$

Molecular Weight: 281.4 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
96-49036	**71685-26-2**	**97.6 ± 0.8%**

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

Synonyms: *N*-Formyl-DPIA;
Di-(1-phenylisopropyl) formamide
N,N-bis(β-phenylisopropyl) formamide

Expiration of certification: The property values are valid till 12 August 2031, 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: Waxy pale green 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.

Note: This material was prepared via reductive amination of benzyl methyl ketone with dexamphetamine sulfate (S configuration). This material is one of two possible diastereoisomers (SS or SR). The exact stereochemistry has not been determined.

Intended use: This certified reference material may be used for instrument calibration.

Instructions for use: Equilibrate the bottled material to room temperature before opening.

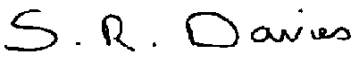
Recommended storage: When not in use, this material should be stored at or below 4 ºC in a closed container in a dry, dark area.

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 measurement uncertainty at the 95% coverage 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 five 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 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.



Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
14 September 2022

This report supersedes any issued prior to 14 September 2022.

NATA Accreditation No. 198 / Corporate Site No. 14214.

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:	HP5890
	Column:	Zebron ZB-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 250 °C (4 min)
	Injector:	250°C
	Detector Temp:	315 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 97.8 %, s = 0.2% (5 sub samples in duplicate, July 2006)
GC-FID:	Instrument:	Varian CP3800 and Agilent HP 8890
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm or HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	200 °C (1 min), 2 °C/min to 220 °C, 10 °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 = 98.2 %, s = 0.3% (5 sub samples in duplicate, July 2009)
	Re-analysis:	Mean = 98.4 %, s = 0.2% (5 sub samples in duplicate, February 2015)
	Re-analysis:	Mean = 98.4 %, s = 0.09% (5 sub samples in duplicate, August 2021)
Thermogravimetric analysis:		Volatile content not determined due to volatility of the material Initial non-volatile residue < 0.2 % mass fraction
Karl Fischer analysis:		Moisture content 0.4% mass fraction (July 2009) Moisture content < 0.1% mass fraction (February 2015 and August 2021)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5971A
	Column:	ZB-5
	Program:	150 °C (1 min) 10 °C/min to 300 °C (1 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.	
	11.0 min:	281 (M^+, <1), 190 (60), 162 (4), 119 (25), 91 (100), 72 (10) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform / Ethyl acetate (4/1) Single spot observed, R_f = 0.5. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS40 FT-IR
	Range:	4000-400cm^{-1}, KBr pellet
	Peaks:	1669, 1496, 1454, 1434, 1374, 1315, 1272, 747, 701 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 0.99 (3H, d, *J* = 6.8 Hz), 1.21 (3H, d, *J* = 6.7 Hz), 2.72 (1H, dd, *J* = 13.5, 8.5 Hz), 2.84 (1H, dd, *J* = 13.4, 6.3 Hz), 2.92 (1H, dd, *J* = 13.4, 8.2 Hz), 3.10 (1H, dd, *J* = 13.3, 7.1 Hz), 3.56-3.62 (1H, m), 4.02 (1H, bs), 7.11 (2H, d, *J* = 7.1 Hz), 7.20-7.24 (4H, m), 7.26-7.31 (4H, m), 8.19 (1H, s) ppm
^{13}C NMR:	Instrument:	Bruker DMX600
	Field strength:	150 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 17.6, 20.2, 40.2, 43.3, 51.3, 54.4, 126.3, 126.7, 128.3, 128.6, 129.0, 129.2, 137.9, 139.0, 162.4 ppm
Microanalysis:	Found:	C = 81.2%, H = 8.3%; N = 5.0% (August 2006)
	Calculated:	C = 81.1%, H = 8.2%; N = 5.0% (Calculated for $C_{19}H_{23}NO$)