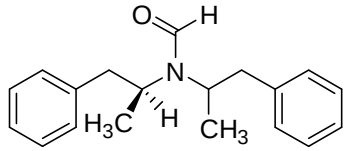
REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D465.2015.01

Compound Name: **Di-(β-phenylisopropyl)formamide**
Description: Waxy pale green solid
Collection Number: D465 Batch Number: 96/49036
Chemical Formula: $C_{19}H_{23}NO$ Molecular Weight: 281.4
CAS Number: 71685-26-2 Release Date: 28th August 2006
Structure:



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

Purity (mass fraction): 97.6 ± 0.8% (95 % coverage interval)

The purity value was obtained from a combination of traditional analytical techniques by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR. Supporting evidence is provided by elemental microanalysis.

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.

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 peak area response of main component:		
	Initial analysis:	Mean = 97.8 %, s = 0.2% (5 sub samples in duplicate, July 2006)	
GC-FID:	Instrument:	Varian CP3800	
	Column:	VF-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 peak area response of 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)	
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)	

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5971A	
	Column:	ZB-5	
	Program:	150 ^{o}C (1 min) 10 °C/min to 300^{o}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 = 0.50. 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)	
		Calc: C = 81.1%, H = 8.2%; N = 5.0% (Calculated for $C_{19}H_{23}NO$)	

Expiration of certification
The property values are valid till 3rd February 2020, 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.

The long-term stability of the compound in solution has not been examined.

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.

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.

Intended Use
For ***in vitro*** laboratory analysis only.

Caution
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice
Neither NMI nor any person acting on NMI'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 certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 4th March 2015.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 4th March 2015.