Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

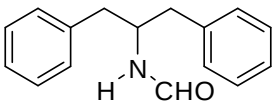
# REFERENCE MATERIAL PRODUCT INFORMATION SHEET

## NMIA D970: N-(α-Benzylphenethyl)formamide

Report ID: D970.2021.02

Chemical Formula: $C_{16}H_{17}NO$

Molecular Weight: 239.3 g/mol




## Propertyl value

| Batch No. | CAS No. | Purity estimate |
|---|---|---|
| **11-D-11** | **788-09-0** | **99.8 ± 1.0%** |

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

**IUPAC name:** *N*-(1,3-Diphenyl-2-propanyl)formamide.

**Expiration of certification:** The property values are valid till 10 November 2026, 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 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.

**Intended use:** This reference material should be used for qualitative analysis only and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

**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:** 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 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.

**Caution:** 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.
20 September 2022

This report supersedes any issued prior to 20 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. Impurities of related structure were assessed by GC-FID. The purity estimate was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID. 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 Karl Fischer analysis, $^{1}$H NMR spectroscopy and qualitative elemental microanalysis.

| | | |
|---|---|---|
| GC-FID: | Instrument: | Varian CP-3800 or Agilent 6890N/8890 |
| | Column: | HP-5 or HP-1, 30.0 m × 0.32 mm I.D. × 0.25 μm |
| | Program: | 150 °C (1 min), 10 °C/min to 240 °C, 30 °C/min to 300 °C (5 min) |
| | Injector: | 200 °C |
| | Detector Temp: | 320 °C |
| | Carrier: | Helium |
| | Split ratio: | 20/1 |
| | Relative peak area of the main component: | |
| | Initial analysis: | Mean = 99.8%, s = 0.01% (10 sub samples in duplicate, July 2011) |
| | Re-analysis: | Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, May 2012) |
| | Re-analysis: | Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, April 2013) |
| | Re-analysis: | Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, March 2014) |
| | Re-analysis: | Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, March 2017) |
| | Re-analysis: | Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, November 2021) |
| Karl Fischer analysis: | | Moisture content < 0.1% mass fraction (April 2013, February 2017 & November 2021) |

## Spectroscopic and other characterisation data

| | | |
|---|---|---|
| GC-MS: | Instrument: | Agilent 6890/5973 |
| | Column: | TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm |
| | Program: | 60 °C (1 min), 10 °C/min to 300 °C (3 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 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 (18.8 min): | 237 (3), 194 (71), 148 (45), 120 (100), 103 (23), 91 (48), 77 (15), 65 (13) *m/z* |
| ESI-MS: | Instrument: | Waters Acquity UPLC |
| | Operation: | Positive ion mode, direct infusion at 10 µL/min |
| | Ionisation: | ESI spray voltage at 3.0 kV positive ion |
| | EM voltage: | 650 V |
| | Cone voltage: | 20 V |
| | Peak: | 240 ($M+H^+$) *m/z* |
| IR: | Instrument: | Biorad FTS3000MX FT-IR |
| | Range: | 4000-400 $cm^{-1}$, KBr powder |
| | Peaks: | 3281, 3085, 3063, 3029, 2953, 2917, 2859, 2765, 1661, 1539, 1522, 1496, 1454, 1388, 1255, 1086, 1032, 711, 754, 733, 701, 498 $cm^{-1}$ |
| $^1$H NMR: | Instrument: | Bruker Avance III |
| | Field strength: | 400 MHz |
| | Solvent: | $CDCl_3$ (7.26 ppm) |
| | Spectral data: | δ 2.67-2.83 (2H, m), 2.87-2.97 (2H, m), 3.78 (0.25H, m), 4.56 (0.75H, m), 5.34 (0.70H, d, *J* = 7.5 Hz), 5.59 (0.3H, t, *J* = 10.8 Hz), 7.14-7.37 (10H, m), 7.46 (0.26H, d, *J* = 11.8 Hz), 8.03 (0.74H, d, *J* = 1.2 Hz) ppm |
| | n-Hexane (0.03%) mass fraction estimated by $^1$H NMR spectrum. | |
| $^{13}$C NMR: | Instrument: | Bruker Avance III |
| | Field strength: | 100 MHz |
| | Solvent: | $CDCl_3$ (77.16 ppm) |
| | Spectral data: | δ 39.9, 42.4, 50.4, 55.9, 126.8, 127.1, 128.5, 128.7, 128.9, 129.47, 129.54, 137.2, 137.7, 160.8, 163.9 ppm |
| Melting point: | | 84-86 °C |
| Microanalysis: | Found: | C = 80.3%; H = 7.3%; N = 5.8% (August, 2011) |
| | Calculated: | C = 80.3%; H = 7.2%; N = 5.9% (Calculated for $C_{16}H_{17}NO$) |