Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

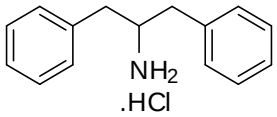
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D971: α-Benzylphenethylamine hydrochloride

Report ID: D971.2021.02

Chemical Formula: $C_{15}H_{17}N.HCl$

Molecular Weight: 247.8 g/mol (HCl), 211.3 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
11-D-12	**7763-96-4 (HCl)** **4275-43-8 (base)**	**99.8 ± 0.3%**

IUPAC name: 1,3-Diphenyl-2-propanammonium chloride

Expiration of certification: The property values are valid till 17 June 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: 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 reference material is recommended 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: This material has demonstrated stability over a minimum period of ten 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.

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 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, 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:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program	120 °C (1 min), 20 °C/min to 250 °C (2 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of main component as the free base:	
	Initial analysis:	Mean = 99.97%, s = 0.03% (10 sub samples in duplicate, July 2011)
	Re-analysis:	Mean = 99.96%, s = 0.02% (5 sub samples in duplicate, July 2012)
GC-FID:	Instrument:	Varian CP-3800 or Agilent 7890
	Column:	HP-5, 30.0 m × 0.32 mm I.D. × 0.25 μm
	Program	120 °C (1 min), 20 °C/min to 250 °C (2 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of main component as the free base:	
	Initial analysis:	Mean = 99.98%, s = 0.02% (10 sub samples in duplicate, July 2011)
	Re-analysis:	Mean = 99.97%, s = 0.007% (5 sub samples in duplicate, May 2016)
	Re-analysis:	Mean = 99.98%, s = 0.003% (5 sub samples in duplicate, June 2021)
Karl Fischer analysis:		Moisture content ≤ 0.1% mass fraction (August 2011, July 2012, May 2016 and May 2021)
Thermogravimetric analysis:		The volatile content could not be determined using thermogravimetric analysis. Non-volatile residue < 0.1% mass fraction (August 2011)

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
	Split ratio:	30/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention times of the free base and *N*-acetyl derivative are 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.	
	Free base (15.7 min):	120 (M-$C_6H_5CH_2$, 100), 103 (9), 91 (15), 77 (5), 65 (4) *m/z*
	N-Acetyl (18.6 min):	194 (M-NHAc, 13), 162 (39), 120 (100), 91 (21), 77 (4), 65 (5) *m/z*
ESI-MS:	Instrument:	Micromass Quatro Micro
	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:	212 ($M+H^+$) *m/z*
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-500 cm^{-1}, KBr powder
	Peaks:	3026, 2896, 2866, 2757, 2596, 2565, 2458, 1604, 1568, 1504, 1454, 1378, 1268, 1114, 987, 748, 699, 502, 421 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	600 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 2.94 (4H, d, *J* = 7 Hz), 3.75 (1H, quintet, *J* = 7.2, 7.0 Hz), 7.26-7.31 (6H, m), 7.36-7.38 (4H, m) ppm
		Isopropanol (0.07%) and diethyl ether (0.03%) mass fraction estimated by ^{1}H NMR spectrum.
^{13}C NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	151 MHz
	Solvent:	MeOH-d_4 (49.0 ppm)
	Spectral data:	δ 39.7, 55.6, 128.5, 130.1, 130.4, 137.0 ppm
Melting point:		204-206 °C
Microanalysis:	Found:	C = 71.2%; H = 7.4%; N = 5.5%; Cl = 15.6% (July, 2011)
	Found:	C = 71.1%; H = 7.1%; N = 5.6%; Cl = 15.5% (August, 2011)
	Calculated:	C = 72.7%; H = 7.3%; N = 5.7%; Cl = 14.3% (Calculated for $C_{15}H_{17}N.HCl$)