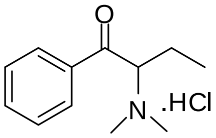
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1011: 2-(Dimethylamino)-1-phenyl-1-butanone hydrochloride



Report ID: D1011.2016.04

Chemical Formula: $C_{12}H_{27}NO.HCl$

Molecular Weight: 227.7 g/mol (HCl), 191.3 g/mol (base)

Certified value

Batch No.	CAS No.	Purity (mass fraction)
14-D-03	**2576-13-8 (HCl)** **2576-12-7 (base)**	**99.8 ± 1.7%**

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

IUPAC name: 2-(Dimethylamino)-1-phenyl-1-butanone hydrochloride (1:1).

Expiration of certification: The property values are valid till 26 February 2019, i.e. three 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 certified reference material is suitable for use as a primary calibrator.

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.

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: In the absence of long term stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last 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.
21 September 2022

This report supersedes any issued prior to 21 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.

$$\text{Purity} = (100\,\% - I_{ORG}) \times (100\,\% - I_{VOL} - I_{NVR}) \qquad \text{Equation 1}$$

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by HPLC with UV detection, qualitative headspace GC-MS analysis of occluded solvents andelemental microanalysis.

Warning: This material is sensitive to the quality of the silanised glass liner when injected at elevated temperature (~ 250 ºC) into a GC instrument.

GC-FID:	Instrument:	Agilent 6890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	100 °C (1 min), 5 °C/min to 120 °C (2 min), 20 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the free base:	
	Initial analysis:	Mean = 99.8%, s = 0.01% (10 sub samples in duplicate, January 2014)
	Re-analysis:	Mean = 99.6%, s = 0.1% (5 sub samples in duplicate, March 2015)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, February 2016)
HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	X-Bridge C-18, 5 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	Acetonitrile/MilliQ water (40:60 v/v), the aqueous phase was buffered at pH 10.8 using 20mM NH_4OAc and NH_4OH
	Flow rate:	1.5 mL/min
	Detector:	Shimadzu SPD-M20A PDA operating at 225 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.004% (7 sub samples in duplicate, March 2014)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (February 2014, March 2015 and February 2016)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (March 2014). 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:	Free base compound:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	100 °C (1 min), 5 °C/min to 120 °C (2 min), 20 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Split ratio:	30/1
	Transfer line temp:	300°C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	The retention time of the free base 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.	
	Free base (9.3 min):	105 (10), 86 (100), 77 (22), 71 (28), 51 (9), 42 (14) *m/z*
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 µL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	192.2 (M+H$^+$) *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:	Diethyl ether
TLC:	Conditions:	Kieselgel 60F$_{254}$. NH$_3$/methanol (3:200) Single spot observed, R$_f$ = 0.7. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3342, 2943, 2733, 2658, 2504, 1687, 1596, 1451, 1346, 1238, 1168, 1046, 1005, 942, 838, 744, 703, 505 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-600
	Field strength:	600 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 0.76 (3H, t, *J* = 7.5 Hz), 1.97 (1H, m), 2.03 (1H, m), 2.82 (3H, bs), 2.87 (3H, bs), 5.56 (1H, bt, *J* = 5.2 Hz), 7.61 (2H, t, *J* = 7.6 Hz), 7.76 (1H, t, *J* = 7.4 Hz), 8.10 (2H, d, *J* = 7.3 Hz), 10.76 (1H, s) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-600
	Field strength:	151 MHz
	Solvent:	DMSO-d_6 (39.5 ppm)
	Spectral data:	δ 8.60, 21.4, 40.7, 42.2, 67.8, 128.8, 129.2, 134.8, 134.9, 196.3 ppm
Melting point:		189-190 °C
Microanalysis:	Found:	C = 63.6%; H = 8.1%; N = 6.2%; Cl% = 15.6% (February 2014)
	Calculated:	C = 63.3%; H = 8.0%; N = 6.2%; Cl% = 15.6% (Calculated for C$_{12}$H$_{17}$NO.HCl)