Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

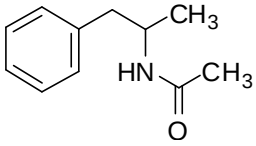
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D445b: *N*-Acetylamphetamine

Report ID: D445b.2022.02

Chemical Formula: $C_{11}H_{15}NO$

Molecular Weight: 177.2 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
11-D-21	**14383-60-9**	**99.4 ± 0.9%**

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

Synonyms: Acetylamphetamine
N-Acetyl phenylisopropylamine
N-(α-Methylphenethyl) acetamide
N-(1-Methyl-2-phenylethyl) acetamide

Expiration of certification: The property values are valid till 24 February 2027, 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: 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%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

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 detection 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 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.
14 September 2022

This report supersedes any issued prior to 14 September 2022.

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

Legal notice: Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

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:	Varian CP-3800 or Agilent 8890	
	Column:	VF-1MS or HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm	
	Program:	120 °C (12 min), 30 °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 = 99.7%, s = 0.03% (10 samples in duplicate, May 2012)	
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, July 2017)	
	Re-analysis:	Mean = 99.7%, s = 0.03% (5 sub samples in duplicate, February 2022)	
GC-FID:	Instrument:	Varian CP-3800	
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 μm	
	Program:	120 °C (12 min), 30 °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 = 99.7%, s = 0.01% (10 samples in duplicate, May 2012)	
Thermogravimetric analysis:		The volatile content could not be determined because of the inherent volatility of the material. Non-volatile residue < 0.2% mass fraction (July 2012)	
Karl Fischer analysis:		Moisture content ≤ 0.1% mass fraction (July 2012, July 2017 and January 2022)	

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1ms, 30 m × 0.25 mm I.D. × 0.25 μm
	Program:	60 °C (1min), 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 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.	
	12.1 min:	177 (M^+, 1), 118 (44), 91 (35), 86 (58), 65 (11), 44 (100) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Diisopropylether/diethyl ether/diethylamine (45/45/10). Single spot observed, R_f = 0.3.
IR:	Instrument:	Biorad FTS 3000 Excalibur FT-IR
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks	3248, 3082, 2969, 2856, 1637, 1447, 703 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 1.11 (3H, d, *J* = 6.7 Hz), 1.92 (3H, s), 2.71 (1H, dd, *J* = 7.2, 13.6 Hz), 2.83 (1H, dd, *J* = 5.6, 13.4 Hz), 4.26 (1H, sep, *J* = 6.8 Hz), 5.3 (1H, bs), 7.17 (2H, d, *J* = 7.2 Hz), 7.22 (1H, t, *J* = 7.3 Hz), 7.29 (1H, t, *J* = 7.5 Hz) ppm
		Hexane estimated at 0.14% mass fraction was observed in the ^{1}H NMR ($CDCl_3$). Chloroform estimated at 0.09% mass fraction was observed in the ^{1}H NMR (d_6-DMSO).
^{13}C NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$
	Spectral data:	δ 19.8, 23.4, 42.3, 46.0, 126.3, 128.3, 129.3, 137.8, 169.2 ppm
Melting point:		93-95 °C
Microanalysis:	Found:	C = 74.7%; H = 8.6%; N = 7.9% (May 2012)
	Calculated:	C = 74.5%; H = 8.5%; N = 7.9% (Calc. for $C_{11}H_{15}NO$)