



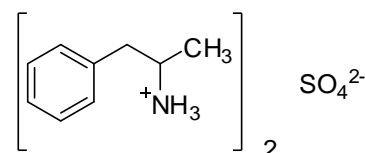
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D420d: (±)-Amphetamine sulfate

Report ID: D420d.2019.04 (Bottled 150423)

Chemical Formula: $(C_9H_{13}N)_2 \cdot H_2SO_4$

Molecular Weight: 368.5 g/mol (H_2SO_4)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
15-D-04	60-13-9	99.2 ± 0.5%

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

IUPAC name: 1-Phenyl-2-propanamine sulfate (2:1)

Expiration of certification: The property values are valid till 17 December 2024, 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.

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 °C 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 three years. The measurement uncertainty at the 95% confidence 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 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 14 September 2022.

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

CIPM MRA notice: This certificate is consistent with the capabilities that are included in Appendix C of the CIPM MRA drawn up by the CIPM. Under the CIPM MRA, all participating institutes recognize the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C. The "CIPM MRA Logo" and this statement attest only to the measurement(s) applied for determining the certified values on the certificate (for details see <http://www.bipm.org>).

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 from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include GC-FID, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

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

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

The purity estimate by QNMR was obtained using a combination of the two proton multiplet at 3.59 ppm, the four proton doublet at 2.90 ppm and the six proton doublet at 1.26 ppm against a certified internal standard of maleic acid.

Supporting evidence is provided by elemental microanalysis.

GC-FID: Instrument: Varian CP-3800
 Column: HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 60 °C (3 min), 10 °C/min to 150 °C (6 min), 30 °C/min to 300 °C (3 min)
 Injector: 180 °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 = 99.6%, s = 0.06% (10 sub samples in duplicate, March 2015)
 Re-analysis: Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, February 2016)
 Re-analysis: Mean = 99.6%, s = 0.05% (5 sub samples in duplicate, January 2019)
 Re-analysis: Mean = 99.7%, s = 0.01% (6 sub samples in duplicate, December 2019)

Thermogravimetric analysis: Volatile content < 0.1% and non-volatile residue < 0.2% mass fraction (March 2016)

Karl Fischer analysis: Moisture content ca 0.2% mass fraction (March 2015, January 2019, November 2019)
 Moisture content ca 0.1% mass fraction (February 2016)

QNMR: Instrument: Bruker Avance-III-500
 Field strength: 500 MHz
 Solvent: D₂O (4.79 ppm)
 Internal standard: Potassium hydrogen maleate (98.7% mass fraction)
 Initial analysis: Mean (1.26 ppm) = 98.9%, s = 0.4% (5 sub samples, March 2015)
 Initial analysis: Mean (2.90 ppm) = 99.0%, s = 0.4% (5 sub samples, March 2015)
 Initial analysis: Mean (3.59 ppm) = 99.2%, s = 0.4% (5 sub samples, March 2015)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	HP-1, 30 m x 0.25 mm I.D. x 0.25 μ m
	Program:	60 °C (3 min), 10 °C/min to 150 °C, 30 °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 free base is reported along 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 (8.8 min):	120 (4), 91 (16), 65 (8), 44 (100) <i>m/z</i>
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1} , KBr pellet
	Peaks:	3414, 3028, 2981, 2939, 2587, 2496, 1976, 1599, 1497, 1456, 1391, 1123, 743, 701, 618 cm^{-1}
¹ H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D ₂ O (4.79 ppm)
	Spectral data:	δ 1.28 (3H, d, <i>J</i> = 6.6 Hz), 2.92 (2H, d, <i>J</i> = 7.2 Hz), 3.60 (1H, sextet, <i>J</i> = 6.7 Hz), 7.29 (2H, d, <i>J</i> = 7.0 Hz), 7.33 (1H, t, <i>J</i> = 7.3 Hz), 7.40 (2H, t, <i>J</i> = 7.3 Hz) ppm
	Ethanol estimated at < 0.1% mass fraction was observed in the ¹ H NMR	
¹³ C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	D ₂ O
	Spectral data:	δ 17.4, 40.0, 49.0, 127.3, 128.9, 129.3, 136.1 ppm
Melting point:	>300 °C	
Microanalysis:	Found:	C = 58.8%; H = 7.8%; N = 7.6% (March, 2015)
	Calculated:	C = 58.7%; H = 7.7%; N = 7.6% (Calculated for (C ₉ H ₁₃ N) ₂ .H ₂ SO ₄)