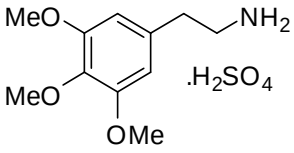
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D768b: Mescaline hydrogen sulfate



Report ID: D768b.2020.03

Chemical Formula: $C_{11}H_{17}NO_3.H_2SO_4$

Molecular Weight: 309.3 g/mol (sulfate), 211.3 g/mol (base)

Property value

Batch No.	CAS No.	Purity estimate
13-D-18	**5967-42-0**	**96.9 ± 1.5 %**

IUPAC name: 2-(3,4,5-Trimethoxyphenyl)ethanammonium sulfate.

Expiration of certification: The property values are valid till 15 October 2025, 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 solid 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 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.

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.
15 September 2022

This report supersedes any issued prior to 15 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. 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.

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

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

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian 3800
	Column:	VF-1ms, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 250 °C, 30 °C/min to 300 °C (3 min)
	Injector:	200°C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component as the free base:	
	Initial analysis:	Mean = 99.5%, s = 0.04% (10 sub samples in duplicate, November 2013)
	Re – analysis:	Mean = 99.8%, s= 0.01% (5 sub samples in duplicate, October 2020)
Karl Fischer analysis:		Moisture content ~2.0% mass fraction (November 2013, March 2014 and October 2020)
Thermogravimetric analysis:		Volatile content 1.8% and non-volatile residue < 0.2% mass fraction (November 2013)

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:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the free base compound are 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.	
	Free base (14.4 min): 211 (M^+, 22), 182 (100), 167 (51), 151 (14), 148 (11), 136 (7), 107 (5) *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
	Split ratio:	50/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Solvents detected:	Ethanol
TLC:	Conditions:	Kieselgel 60F_{254} Methanol/$NH_{3(aq)}$ (100:1.5) Single spot observed, R_f = 0.20 Visualization with UV light (254 nm)
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3148, 2949, 1590, 1510, 1424, 1225, 1129, 848 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 2.95 (2H, t, *J* = 7.3 Hz), 3.27 (2H, t, *J* = 7.3 Hz), 3.75 (3H, s), 3.86 (6H, s), 6.68 (2H, s) ppm
	Ethanol estimated at 0.05% mass fraction was observed in the ^{1}H NMR	
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	151 MHz
	Solvent:	MeOH-d_4 (49.0 ppm)
	Spectral data:	δ 34.8, 42.0, 56.6, 61.0, 107.1, 134.1, 138.1, 154.8 ppm
Melting point:		142-145 °C
Microanalysis:	Found:	C = 44.2%; H = 6.5%; N = 4.6% (December 2013)
	Calculated:	C = 42.7%; H = 6.2%; N = 4.5% (Calculated for $C_{11}H_{17}NO_3.H_2SO_4$)