Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

NATA
ACCREDITED FOR TECHNICAL COMPETENCE

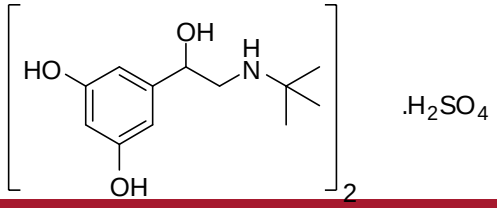
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA M647b: Terbutaline sulfate

Report ID: M647b.2016.02

Chemical Formula: $(C_{12}H_{19}NO_3)_2.H_2SO_4$

Molecular Weight: 548.6 g/mol



Property value

Batch No.	CAS No.	Purity estimate
11-D-29	**23031-32-5**	**99.0%**

IUPAC name: 5-{1-Hydroxy-2-[(2-methyl-2-propanyl)amino]ethyl}-1,3-benzenediol sulfate**.**

Expiration of certification: The property values are valid till 7 November 2021, 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: Off-white powder sourced from an external supplier, 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 should be used 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: In the absence of long term stability data the stability of this material has been judged from stability trials conducted on similar materials by NMI Australia over the last ten years.This material has demonstrated stability over a minimum period of five 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 ^{1}H qNMR on five 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.
4 May 2020

This report supersedes any issued prior to 4 May 2020

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS.

Supporting evidence is provided by thermogravimetric analysis, Karl Fischer analysis, headspace GC-MS analysis of occluded solvents and elemental microanalysis.

QNMR:	Instrument:	Bruker Avance-DMX-600
	Field strength:	600 MHz
	Solvent:	D_2O (4.8 ppm)
	Internal standard:	Sodium acetate (60.1% mass fraction)
	Initial analysis:	Mean (1.38 ppm) = 99.9%, s = 0.3% (5 sub samples, November 2011)
	Initial analysis:	Mean (6.40 ppm) = 100.0%, s = 0.4% (5 sub samples, November 2011)
	Re-analysis:	Mean (1.34 ppm) = 99.0%, s = 0.38% (5 sub samples, November 2011)
	Re-analysis:	Mean (1.35 ppm) = 99.7%, s = 0.25% (5 sub samples, October 2016)
Karl Fischer analysis:		Moisture content ≤ 0.1% mass fraction (November 2011, November 2013 and November 2016)
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction (November 2011)

Spectroscopic and other characterisation data

GC-MS:	*Tris*-TMS derivative:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	100 °C (1 min), 15 °C/min to 220 °C, 10 °C/min to 250 °C, 30°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 tris-TMS derivative 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.	
	Tris-TMS (10.3 min):	426 (3), 356 (91), 336 (4), 280 (6), 265 (4), 147 (4), 86 (100), 75 (24), 73 (53), 57 (13) *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:	15 V
	Peak:	226 $(M+H^+)$ *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
	Split ratio:	50/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Solvents detected:	Methyl acetate, isopropanol
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/concentrated ammonia (200/3) Single spot observed, R_f = 0.50. Visualisation with UV at 254 nm.
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3332, 3055, 2973, 2814, 2668, 2505, 1610, 1488, 1343, 1313, 1205, 1158, 1130, 1110, 1054, 975, 857 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.80 ppm)
	Spectral data:	δ 1.38 (18H, s), 3.15 (2H, dd, *J* = 9.7, 12.8 Hz), 3.25 (2H, dd, *J* = 3.2, 12.8 Hz), 4.87 (2H, dd, *J* = 3.2, 9.7 Hz), 6.38 (2H, t, *J* = 2.2 Hz), 6.50 (4H, d, *J* = 2.2 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-400
	Field strength:	101 MHz
	Solvent:	D_2O
	Spectral data:	δ 24.7, 47.4, 57.4, 69.1, 102.5, 105.0, 143.1, 157.1 ppm
Microanalysis:	Found:	C = 52.8%; H = 7.5%; N = 5.2% (January 2012)
	Calculated:	C = 52.5%; H = 7.4%; N = 5.1% (Calculated for $(C_{12}H_{29}NO_3.HCl)_2.H_2SO_4$)