Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

NATA ACCREDITED FOR TECHNICAL COMPETENCE

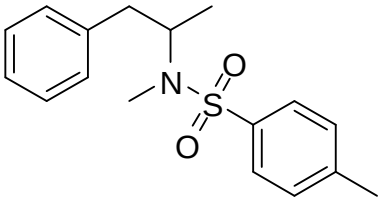
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D1071: p-Tosyl methamphetamine

Report ID: D1071.2021.01

Chemical Formula: $C_{17}H_{21}NO_2S$

Molecular Weight: 303.4 g/mol



Property value

Batch No.	CAS No.	Purity by GC-FID
17-D-11	**34542-10-4**	**99.5% ± 0.03%**

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

IUPAC name: *N*-Tosyl-*N*-methyl-1-phenyl-2-propanamine

Expiration of certification: The property values are valid till 30 August 2026, 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 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 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.

Metrological traceability: Metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established. This is not a certified reference material.

Stability: At the recommended storage conditions this material has demonstrated stability for a 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.
14 September 2021

This report supersedes any issued prior to 14 September 2021.

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 value was obtained by GC-FID only.

Supporting evidence is provided by ^{1}H NMR spectroscopy and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800/Agilent 8890
	Column:	HP-5/HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	120 °C (1 min), 15 °C/min to 220 °C (8 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.5%, s =0.03% (10 sub samples in duplicate, November 2017)
	Re-analysis:	Mean = 99.5%, s = 0.03% (5 sub samples in duplicate, October 2018)
	Re-analysis:	Mean = 99.5%, s = 0.01% (5 sub samples in duplicate, August 2021)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	HP1-MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	120 °C (1 min), 15 °C/min to 220 °C (8 min), 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 parent compound 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.	
	Parent (15.1 min):	212 (M^+-C_7H_7, 93), 155 (42), 91 (100), 65 (19) *m/z*
ESI-MS:	Instrument:	Waters Acquity TQ API mass sprectrometer
	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:	40 V
	Peak:	326.2 ($M+Na^+$, 100), 342.2 ($M+K^+$, < 10) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate (9:1) Single spot observed, R_f = 0.3
IR:	Instrument:	FT-IR, Biorad WIN FTS3000MX
	Range:	4000-400 cm^{-1}, KBr
	Peaks:	1595, 1492, 1452, 1311, 1288, 1149, 1084, 999, 942, 884, 819, 752, 704, 691, 647, 550 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 0.82 (3H, d, *J* = 6.7 Hz), 2.36 (3H, s), 2.55 (1H, dd, *J* = 7.3, 13.4 Hz), 2.63 (1H, dd, *J* = 7.7, 13.4 Hz), 2.66 (3H, s), 4.15 (1H, m), 7.14 (2H, d, *J* = 7.6 Hz), 7.20 (1H, t, *J* = 7.3 Hz), 7.27 (2H, d, *J* = 7.3, 7.6 Hz), 7.34 (2H, d, *J* = 8.1 Hz), 7.53 (2H, d, *J* = 8.1 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	DMSO-d_6 (39.50 ppm)
	Spectral data:	δ 16.3, 21.0, 27.6, 39.6, 53.9, 126.2, 126.8, 128.3, 129.0, 129.8, 136.5, 138.5, 142.9 ppm
Melting point:		65-69 °C [63-64 °C reported for *N*-Tosyldeoxyephedrine EH Gold and E Babad, J. Org. Chem., **1972**, 37(13), 2208-2210]
Microanalysis:	Found:	C = 67.1%; H = 7.2%; N = 4.3%; S% = 10.3% (December 2017)
	Calculated:	C = 67.3%; H = 7.0%; N = 4.6%; S% = 10.6% (Calculated for $C_{17}H_{21}NO_2S$)