Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

NATA
ACCREDITED FOR TECHNICAL COMPETENCE

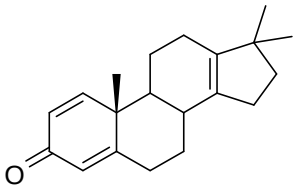
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D576: 17,17-Dimethyl-18-norandrosta-1,4,13(14)-trien-3-one

Report ID: D576.2019.02

Chemical Formula: $C_{20}H_{26}O$

Molecular Weight: 282.4 g/mol



Property value

Batch No.	CAS No.	Purity estimate
99-S-05	77702-25-1	96.4%

IUPAC name: 10,17,17-Trimethylgona-1,4,13-trien-3-one.

Expiration of certification: The property values are valid till 21 June 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: Pale yellow solid 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 -20 ºC in a closed container in a dry, dark area.

Stability: This material has limited stability in solution. A solution in methanol stored at 4 ˚C decomposed to a purity of 85% (GC-FID relative peak area) within six months. If working solutions of high purity are required we recommend the user make a stock solution of the material, sub-divide into separate vials, and remove the solvent under nitrogen. Store the separate dried aliquots at -20 ˚C and make up a working solution periodically from one of these aliquots. Replace with fresh material when the purity of the analyte in the solution drops below an acceptable threshold value.

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.
17 February 2020

This report supersedes any issued prior to 17 February 2020

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 20844. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

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 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 qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

Note: The organic component of this material has shown signs of steady decomposition since 1999. This material has not been fully certified by the Chemical Reference Materials team at NMI and should be considered for use in qualitative analysis only.

GC-FID:	Instrument:	HP 5890
	Column:	ZB-1, 29.5 m x 0.32 mm I.D. x 0.25 µm
	Program:	180 °C (1 min), 10 °C/min to 220 °C, 20 °C/min to 300 °C (6 min)
	Injector:	250 °C
	Detector Temp:	320° C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 98.3%, s = 0.04% (10 sub samples, February 1999)
	Re-analysis:	Mean = 97.6%, s = 0.15% (5 sub samples in duplicate, July 2006)
GC-FID:	Instrument:	Agilent 6890 or 7890
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 µm
	Program:	160 °C (1 min), 10 °C/min to 190 °C (22 min), 30 °C/min to 300 °C (5 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 97.0%, s = 0.03% (5 sub samples in duplicate, September 2009)
	Re-analysis:	Mean = 96.0%, s = 0.11% (5 sub samples in duplicate, July 2014)
	Re-analysis:	Mean = 96.6%, s = 0.09% (5 sub samples in duplicate, June 2019)
Thermogravimetric analysis:		Volatile content < 0.1% and non-volatile residue < 0.2% mass fraction
Karl Fischer analysis:		Moisture content 0.1% mass fraction (July 2014) Moisture content < 0.1% mass fraction (May 2019)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP5890/5971
	Column:	BPX5, 30m x 0.25mm I.D. x 0.25 µm
	Program:	180 °C (1 min), 15 °C/min to 300 °C (3 min)
	Injector:	260 °C
	Transfer line temp:	280 °C
	Carrier:	Helium
	Split ratio: 30/1	
	Mono-TMS derivative:	
	Instrument:	HP6890/5973
	Column:	HP Ultra 1, 17 m x 0.25 mm ID x 0.22 µm
	Program:	170 °C, 3 °C/min to 234 °C, 10 °C/min to 265 °C (3 min)
	Injector:	280 °C
	Transfer line temp:	300 °C
	Carrier:	Helium
	Split ratio:	15/1
	The retention times of the parent compound and *mono*-TMS derivative are reported along with the major peaks in the mass spectrum/spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent:	(9.7 min): 282 (M^+, 57), 267 (46), 161 (86), 147 (40), 122 (100), 105 (84) *m/z*
	Mono-TMS:	(5.1 min): 354 (M^+, 37), 339 (53), 324 (5), 206 (27), 148 (42), 133 (100) *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
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	A mixture of light petroleum solvents (pentanes, hexanes and heptanes)
TLC:	Conditions:	Kieselgel 60F_{254} Ethyl acetate/hexane (1:3) Single spot observed, R_f = 0.48 (3 sub samples)
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm-1, KBr pellet
	Peaks:	1660, 1624, 1601 cm^{-1}
^{1}H NMR:	Instrument:	Bruker ARX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 0.92 (3H, s), 0.96 (3H, s), 1.20 (3H, s), 6.07 (1H, m), 6.24 (1H, dd), 7.14 (1H, dd) ppm
	Light petroleum solvents estimated at 0.1% mass fraction were observed in the ^{1}H NMR	
^{13}C NMR:	Instrument:	Bruker ARX-500
	Field strength:	125 MHz
	Solvent:	$CDCl_3$ (77 ppm)
	Spectral data:	δ 18.5, 22.0, 24.0, 26.4, 26.6, 29.9, 32.5, 33.3, 36.5, 39.4, 43.4, 45.4, 49.4, 124.2, 127.5, 134.2, 141.9, 155.7, 169.0, 186.4 ppm
Microanalysis:	Found:	C = 85.0%, H = 9.4% (April 1999)
	Calculated:	C = 85.1%, H = 9.3% (Calculated for $C_{20}H_{26}O$)