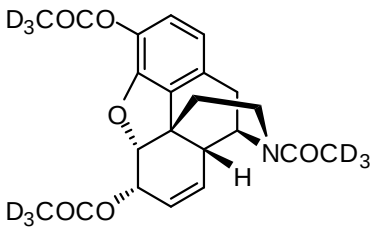
Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

NATA ACCREDITED FOR TECHNICAL COMPETENCE

DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

NMIA D894: d9- Triacetylnormorphine



Report ID: D894.2011.04

Chemical Formula: $C_{22}H_{14}D_9NO_6$

Molecular Weight: 406.5 g/mol

Property value

Batch No.	CAS No.	Purity estimate
03-D-15	Not available	80% minimum

Expiration of certification: The property values are valid till 23 August 2014, i.e. three 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 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: The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard 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 4 ºC in a closed container in a dry, dark area.

Stability: This material is very sensitive to hydrolysis and should be used accordingly. To minimise degradation this material should be handled in non-protic solvent or those without acidic or basic by-products. Recommended solvents are acetonitrile, acetone, THF, ethyl acetate, di-isopropyl ether and dichloromethane.

Homogeneity assessment: The homogeneity of the material was assessed using purity assay by GC-FID 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.
5 August 2021

This report supersedes any issued prior to 5 August 2021.

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 elemental microanalysis.

Isotopic Purity: 99 atom % D, as determined by Aldrich Chemical Company for the parent acetic anhydride-d_6 (Cat # 17,564-1) which was used in the synthesis of D894.

Isotopic Purity: $d_9 \approx (0.99)^9 \sim 91\%$

$d_0 \approx (0.01)^9 \sim 0\%$

The main component of this material is d_9- triacetylnormorphine. d_8-, d_7-, d_6-, d_5-, d_4-, d_3-, d_2-, d_1- and d_0-Triacetylnormorphine are also present. The stated chemical purity of the analyte represents the combined mass fractions of deuterated d_9-, d_8-, d_7-, d_6-, d_5-, d_4-, d_3-, d_2-, d_1- and d_0- triacetylnormorphine in the material.

The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard only.

GC-FID:	Instrument:	Agilent 6890N
	Column:	HP-1, 29.5 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (2 min), 20 °C/min to 250 °C (8 min), 40 °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 = 88.2%, s = 0.11% (7 sub samples in duplicate, July 2005)
	Re-analysis:	Mean = 87.8%, s = 0.05% (5 sub samples in duplicate, August 2007)
	Re-analysis:	Mean = 87.1%, s = 0.1% (5 sub samples in duplicate, September 2008)
	Re-analysis:	Mean = 87.2%, s = 0.4% (5 sub samples in duplicate, August 2011)
Karl Fischer analysis:		Moisture content of 1.5% mass fraction (April 2007) Moisture content of 1.4% mass fraction (August 2008) Moisture content of 1.8% mass fraction (August 2011)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5471A
	Column:	BPX-5, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	160 °C, 20 °C/min to 300 °C (5 min)
	Injector:	250 °C
	Transfer line temp:	320 °C
	Carrier:	Helium, 1.0 mL/min.
	Split ratio:	20/1
	The retention time of the parent compound is 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.	
	Parent (10.33 min):	406 (M+, 19), 405 (8), 404 (5), 362 (8), 361 (8), 281 (9), 255 (8), 254 (13), 253 (10), 237 (12), 236 (10), 211 (44), 210 (96), 209 (39), 208 (10), 207 (26), 182 (12), 91 (14), 90 (100), 89 (42), 74 (15), 73 (16), 46 (51), 45 (26), 44 (11) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/Acetone (3:2) Single spot observed, R_f = 0.4 (2 replicates)
IR:	Instrument:	Biorad FTS 3000 MXFT-IR
	Range:	4000-400 cm^{-1}, KBr
	Peaks:	3250, 2972, 2260, 1762, 1736, 1630, 1493, 1449, 1034, 818 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	DMSO-*d*$_6$
	Duplicate resonances appear due to restricted rotation of the amide bond. Rotamers A and B exist in a ratio of ca. 3:2 respectively. Ethyl acetate 0.4% mass/mass was observed in the ^{1}H NMR.	
	Key spectral data:	**A** δ 3.03 (1H, ddd, *J* = 3.4, 13.5, 13.5 Hz), 3.72 (1H, dd, *J* = 4.6, 14.0 Hz), 5.23 (1H, m), 5.58 (2H, m), 6.61 (1H, d, *J* = 8.2 Hz), 6.79 (1H, d, *J* = 8.2 Hz) ppm **B** δ 4.35 (1H, dd, *J* = 5.0, 13.9 Hz), 4.62 (1H, m), 5.23 (1H, m), 6.64 (1H, d, *J* = 8.2 Hz), 6.79 (1H, d, *J* = 8.2 Hz) ppm
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	125 MHz
	Solvent:	DMSO-*d*$_6$ (39.5 ppm)
	Spectral data:	δ 15.0, 21.7, 24.8, 29.7, 30.4, 30.8, 34.6, 35.2, 35.4, 43.4, 43.4, 47.5, 53.0, 60.7, 68.2, 68.3, 89.0, 89.0, 120.4, 120.5, 123.1, 123.1, 129.7, 129.7, 129.8, 131.6, 132.1, 132.3, 132.3, 150.0, 169.1, 169.2, 170.7 ppm
Microanalysis:	Found:	C = 63.6%; H = 8.4%; N = 3.9% (May, 2005)
	Calculated:	C = 63.5%; H = 8.0%; N = 3.4% (based on $C_{22}H_{14}D_9NO_6$ + ½ H_2O)