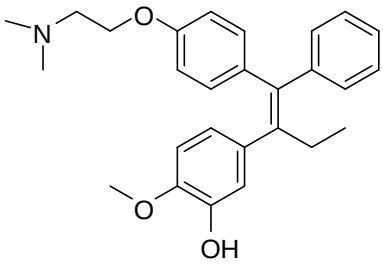
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D921: 3-Hydroxy-4-methoxy-tamoxifen



Report ID: D921.2019.03

Chemical Formula: $C_{27}H_{31}NO_3$

Molecular Weight: 417.5 g/mol

Property value

Batch No.	CAS No.	Purity by ^{1}H NMR
07-S-03	Not available	85%

Synonyms: Tamoxifen metabolite
Z-2-[4-(1-Phenyl-2-(3-hydroxy-4-methoxyphenyl)-1-butenyl)phenoxy]-N,N-dimethylethanamine.

Expiration of certification: The property values are valid till 23 August 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: 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 4 ºC in a closed container in a dry, dark area.

Stability: This material is unstable in solution when exposed to light. 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.

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 November 2022

This report supersedes any issued prior to 15 November 2022.

NATA Accreditation No. 198 / Corporate Site No. 14214.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The purity value was obtained by quantitative nuclear magnetic resonance (qNMR). The two-proton triplet at 2.75 ppm was measured against a certified internal standard of dimethyl sulfone.

Supporting evidence is provided by thermogravimetric analysis, Karl Fischer analysis and elemental microanalysis.

QNMR:	Instrument:	Bruker DMX-600 or Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Internal standard:	Dimethyl sulfone (100.0% mass fraction)
	Initial analysis:	Mean (2.75 ppm) = 85.7%, s = 0.5% (3 sub samples, August 2007)
	Re-analysis:	Mean (2.75 ppm) = 86.0%, s = 0.9% (3 sub samples, November 2008)
	Re-analysis:	Mean (2.75 ppm) = 86.5%, s = 0.3% (3 sub samples, November 2016)
	Re-analysis:	No significant change in the ^{1}H NMR spectrum (August 2019)
Thermogravimetric analysis:		Initial volatile content 1.0 % and non volatile residue < 0.2 % mass fraction
Karl Fischer analysis:		Moisture content 1.8% mass fraction (November 2013) Moisture content 1.9% mass fraction (November 2016) Moisture content 1.7% mass fraction (August 2019)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	HP-2, 17 m × 0.20 mm I.D × 0.25 μm
	Program:	80 °C (0.5 min), 50 °C/min to 200 °C (0.5 min), 15 °C/min to 310 °C (5 min)
	Injector:	290 °C
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio: 10/1	
	TMS derivative:	
	Instrument:	Agilent 6890/5973
	Column:	Ultra 1, 17 m x 0.2 mm I.D x 0.11 μm
	Program:	178 °C (0.2 min), 3 °C/min to 229 °C, 10 °C/min to 265 °C, 30 °C/min to 310°C (2 min)
	Injector:	250 °C
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	14/1
		The retention times of the parent compound and TMS derivative are 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 (9.5 min):	417 (M^+, 20), 239 (3), 165 (3), 72 (35), 58 (100) *m/z*
	TMS (18.8 min):	489 (M^+, 21), 417 (2), 72 (42), 58 (100) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/methanol (1/1) Single spot observed, R_f = 0.53. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	2954, 2868, 2836, 2787, 1608, 1574, 1508, 1468, 1282, 1243, 1173, 1028, 834, 799, 701 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX 500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$
	Spectral data:	δ 0.91 (3H, t, *J* = 7.3 Hz), 2.32 (6H, s), 2.39 (2H, q, *J* = 7.4 Hz), 2.69 (2H, t, *J* = 5.7 Hz), 3.82 (3H, s), 3.96 (2H, t, *J* = 5.7 Hz), 6.55-6.63 (4H, m), 6.75-6.79 (3H, m), 7.20-7.24 (3H, m), 7.31-7.32 (2H, m) ppm
^{13}C NMR:	Instrument:	Bruker DMX 500
	Field strength:	125 MHz
	Solvent:	$CDCl_3$
	Spectral data:	δ 13.6, 28.9, 45.7, 55.7, 58.1, 65.4, 110.1, 113.4, 115.7, 121.8, 126.4, 128.0, 129.4, 131.7, 135.6, 135.9, 137.7, 140.8, 144.0, 144.9, 145.0, 156.5 ppm
Melting point:		127-130 °C
Microanalysis:	Found:	C = 76.3 %; H = 7.6 %; N = 3.3% (August 2007)
	Calculated:	C = 77.7 %; H = 7.5 %; N = 3.4% (Calculated for $C_{27}H_{31}NO_3$)