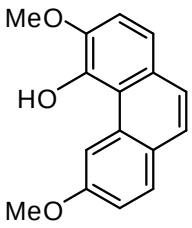
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D715: Thebaol



Report ID: D715.2022.02

Chemical Formula: $C_{16}H_{14}O_3$

Molecular Weight: 254.3 g/mol

Property value

Batch No.	CAS No.	Purity estimate
01-D-016	481-81-2	99.2 ± 1.6%

IUPAC name: 3,6-Dimethoxy-4-phenanthrenol.

Expiration of certification: The property values are valid till 18 January 2032, i.e. ten 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: Yellowish crystalline solid 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: This reference material is recommended for qualitative analysis only and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

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 has demonstrated stability over a minimum period of five years. This material is susceptible to oxidation in solution.

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 2022

This report supersedes any issued prior to 14 September 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. Impurities of related structure were assessed by GC-FID. The purity estimate was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID and thermogravimetric analysis. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100\ \% - I_{ORG}) \times (100\ \% - I_{VOL} - I_{NVR}) \qquad \text{Equation 1}$$

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

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

Warning: **This material is sensitive to oxidation in solution.**

GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	200 °C (1 min), 15 °C/min to 300 °C (6 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, November 2011)
GC-FID:	Instrument:	Agilent 6890N
	Column:	HP-1 Capillary, 30 m × 0.32 mm I.D. × 0.5 μm
	Program:	200 °C (1min), 15 °C/min to 300 °C (6 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.5%, s = 0.04 (10 sub samples in duplicate, March 2002)
	Re-analysis:	Mean = 99.5%, s = 0.02 (5 sub samples in duplicate, December 2008)
	Re-analysis:	Mean = 99.5%, s = 0.06 (5 sub samples in duplicate, November 2016)
	Re-analysis:	Mean = 99.6%, s = 0.02 (5 sub samples in duplicate, January 2022)
Thermogravimetric analysis:		Volatile content 0.5% and non-volatile content < 0.2% mass fraction (February 2002)
		Volatile content 0.2% and non-volatile content < 0.2% mass fraction (December 2005)
		Volatile content 0.6% and non-volatile content < 0.2% mass fraction (December 2008)
		Volatile content 0.8% and non-volatile content < 0.2% mass fraction (November 2011)
		Volatile content 0.8% and non-volatile content < 0.2% mass fraction (November 2016)
		Volatile content 0.4% and non-volatile content < 0.2% mass fraction (January 2022)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 5890/5471A
	Column:	BPX-5, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	200 °C (1 min), 10 °C /min to 300 °C (5 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 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 (8.1 min):	254 (M^+, 94), 239 (100), 211 (29), 168 (30), 139 (49) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform + 1% methanol Single spot observed, R_f = 0.6 (3 replicates)
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr
	Peaks:	3448, 3160, 2993, 2827, 1827, 1602, 1436, 1242, 837, 579 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 9.25 (1H, d, *J* = 2.6 Hz), 7.76 (1H, d, *J* = 8.6 Hz), 7.53 (1H, d, *J* = 8.8 Hz), 7.49 (1H, d, *J* = 8.8 Hz), 7.41 (1H, d, *J* = 8.6 Hz), 7.27 (1H, d, *J* = 8.5 Hz), 7.24 (1H, dd, *J* = 2.6, 8.7 Hz), 4.04 (3H, s), 4.01 (3H, s) ppm
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	125 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 157.9, 143.7, 143.6, 131.3, 129.0, 128.9, 127.1, 125.4, 124.6, 119.5, 118.5, 115.9, 111.1, 110.3, 57.0, 55.4 ppm.
Melting point:		91–92.5 °C
Microanalysis:	Found:	C = 75.6%; H = 5.6% (February, 2002)
	Calculated:	C = 75.6%; H = 5.6% (Calculated for $C_{16}H_{14}O_3$)