Australian Government
Department of Industry, Science and Resources

National Measurement Institute

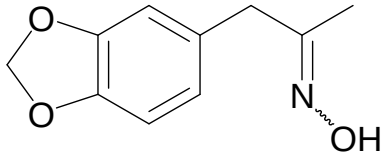
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D890: 3,4-Methylenedioxybenzyl methyl ketoxime

Report ID: D890.2021.03

Chemical Formula: $C_{10}H_{11}NO_3$

Molecular Weight: 193.2 g/mol



Property value

Batch No.	CAS No.	Purity estimate
05-D-01	52271-42-8	99.5 ± 0.3 %

Expiration of certification: The property values are valid till 2 February 2031, 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: 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: 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 25 ºC in a closed container in a dry, dark area.

Stability: This material has demonstrated stability over a minimum period of ten 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 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.

Caution: 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.
13 October 2022

This report supersedes any issued prior to 19 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. The certified 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.

Note: This material exists as a 20:1 mix of isomers, by ^{1}H NMR. The purity statement relates to the major isomer.

GC-FID:	Instrument:	Agilent 6890N or 7890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °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 = 98.9%, s = 0.03% (7 sub samples in duplicate, March 2005)
	Re-analysis:	Mean = 99.1%, s = 0.11% (5 sub samples in duplicate, June 2008)
	Re-analysis:	Mean = 99.5%, s = 0.009% (5 sub samples in duplicate, June 2011)
	Re-analysis:	Mean = 99.5%, s = 0.03% (5 sub samples in duplicate, April 2016)
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, February 2021)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (March 2005). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.
Karl-Fischer analysis:		Moisture content ≤ 0.2% mass fraction (June 2008, June 2011, April 2016 and January 2021)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 5890/5971A
	Column:	BPX-5, 30 m x 0.25 mm I.D. x 0.30 μm
	Program:	100 °C (1 min), 10 °C/min to 250 °C
	Injector:	220°C
	Transfer line temp:	280 °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 (10.9 min):	193 (M^+, 54), 160 (18), 146 (36), 135 (100), 118 (15), 105 (16), 77 (41), 51 (29) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/ethyl acetate (9:1) Single spot observed, R_f = 0.3. Visualisation with UV at 254 nm.
IR:	Instrument:	Biorad FTS300MX FT-IR.
	Range:	4000-400 cm^{-1}, KBr pellet.
	Peaks:	3236, 2897, 1488, 1446, 1246, 1040, 946, 922, 811, 773, 670 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$
	Spectral Data:	δ 1.81 (3H, s), 3.40 (2H, s), 5.93 (2H, s), 6.68 (1H, d, *J* = 7.9 Hz), 6.71 (1H, s), 6.75 (1H, d, *J* = 7.8 Hz), 8.92 (1H, bs) ppm
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	150 MHz
	Solvent:	$CDCl_3$
	Spectral data:	δ 13.1, 19.6, 34.4, 41.7, 100.9, 108.3, 109.3, 122.0, 130.3, 146.4, 147.8, 157.7 ppm
Melting point:		87-89 °C
Microanalysis:	Found:	C = 62.2%; H = 5.9%
	Calculated:	C = 62.2%; H = 5.7% (Calculated for $C_{10}H_{11}NO_3$)