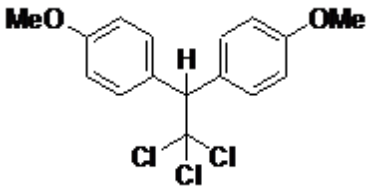
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1305: Methoxychlor



Report ID: P1305.2017.03

Chemical Formula: $C_{16}H_{15}Cl_3O_2$

Molecular Weight: 345.6 g/mol

Certified value

Batch No.	CAS No.	Purity (mass fraction)
96/024237	72-43-5	99.7 ± 0.5%

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

IUPAC name: 1,1'-(2,2,2-Trichloro-1,1-ethanediyl)bis(4-methoxybenzene)

Expiration of certification: The property values are valid till 29 November 2027, 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: White crystal 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 certified reference material is suitable for use as a primary calibrator.

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.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

Stability: This material has demonstrated stability over a minimum period of ten years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. 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.

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.
10 August 2022

This report supersedes any issued prior to 10 August 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 from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include 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

The certified purity value by qNMR was obtained using a combination of the six-proton singlet at 3.8 ppm, the one-proton singlet at 5.0 ppm and the two-proton doublet at 6.9 ppm against a certified internal standard of dimethyl sulfone.

Supporting evidence is provided by qualitative elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	180 °C (1 min), 12 °C/min to 300 °C (1 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.7%, s = 0.2% (4 sub samples in duplicate, May 1996)
	Re-analysis:	Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, February 2007)
	Re-analysis:	Mean = 99.8%, s = 0.003% (5 sub samples in duplicate, September 2012)
	Re-analysis:	Mean = 99.7%, s = 0.02% (8 sub samples in duplicate, November 2017)
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (February 2007, January 2012, October 2017)
QNMR:	Instrument:	Bruker Avance-III-400
	Field strength:	400 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Internal standard:	Dimethyl sulfone (100.0% mass fraction)
	Initial analysis:	Mean (3.8 ppm) = 99.8%, s = 0.3% (5 sub samples, January 2013)
	Initial analysis:	Mean (5.0 ppm) = 99.6%, s = 0.3% (5 sub samples, January 2013)
	Initial analysis:	Mean (6.9 ppm) = 99.9%, s = 0.3% (5 sub samples, January 2013)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 6890/5973
	Column:	ZB-5ms, 28 m x 0.25 mm I.D.x 0.25 µm
	Program:	180° C (1 min), 12° C/min to 300°C (1 min)
	Injector:	250° 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 mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	7.62 min:	346 (M+, 2), 308 (5), 274 (20), 228 (17), 227 (100), 152 (9) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Cyclohexane/di-isopropyl ether/diethylamine (52/40/8) Single spot observed, R_f = 0.38. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS40 FT-IR
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks:	1612, 1511, 1464, 1308, 1254, 1184, 1033, 853, 832, 795, 712, 575, 556 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 3.80 (6H, s), 5.00 (1H, s), 6.89 (4H, d, *J* = 8.9 Hz), 7.55 (4H, d, *J* = 8.8 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Gyro-300
	Field strength:	75 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data:	δ 55.2, 69.7, 102.5, 113.6, 130.6, 131.1, 159.1 ppm
Melting point:		88.5-89.0 °C
Microanalysis:	Found:	C = 55.8%; H = 4.5% (March, 2007)
	Calculated:	C = 55.6%; H = 4.4% (Calculated for $C_{16}H_{15}Cl_3O_2$)