Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

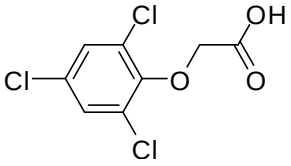
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1375: 2,4,6-Trichlorophenoxyacetic acid

Report ID: P1375.2024.01

Chemical Formula: $C_8H_5Cl_3O_3$

Molecular Weight: 255.49 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
96/104158	**575-89-3**	**99.4 ± 0.3%**

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

IUPAC name: 2,4,6-Trichlorophenoxyacetic acid.

Expiration of certification: The property values are valid till 11 April 2034, 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: Small, white, needle-like crystals 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 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 all impurities are quantified as a mass fraction and subtracted from 100%.

Stability: This material has demonstrated stability over a minimum period of 5 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.
12 April 2024

This report supersedes any issued prior to 12 April 2024.

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

Legal notice: Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

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.

GC-FID:	Instrument:	HP5890 or Varian CP-3800
	Column:	ZB-1, HP-1, or DB-17, Capillary, 30 m × 0.32 mm × 0.25 μm
	Program:	120 °C (2 min), 10 °C/min to 250 °C (1 min), 30 °C/min to 280 °C (3 min)
	Injector:	250 °C
	Detector Temp:	315 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main componentas TMS derivative:	
	Initial analysis:	Mean = 99.6%, s = 0.19% (6 sub samples in duplicate, September 1996)
	Re-analysis:	Mean = 99.4%, s = 0.05% (5 sub samples in duplicate, June 2008)
	Re-analysis:	Mean = 99.4%, s = 0.03% (5 sub samples in duplicate, May 2013)
	Re-analysis:	Mean = 99.4%, s = 0.03% (5 sub samples in duplicate, September 2019)
	Re-analysis:	Mean = 99.4%, s = 0.02% (5 sub samples in duplicate, April 2024)
HPLC:	Instrument:	LKB Bromma HPLC pump with a HP1047A refractive index detector
	Column:	Alltima C-18 5 mm (4.6 mm x 250 mm)
	Mobile Phase:	Acetonitrile/water/TFA (60/40/0.05)
	Relative mass fraction of the main component: Mean of 6 replicates > 99.9% (impurities < 0.1%)	
Karl Fischer analysis:		Moisture content ≤ 0.2% mass fraction (July 2008 and May 2013) Moisture content < 0.1% mass fraction (September 2019 and April 2024)
Thermogravimetric analysis:		Volatiles content and non-volatile residue < 0.3% mass fraction (October 2005)

Spectroscopic and other characterisation data

GC-MS:	Derivatised as the methyl ester.	
	Instrument:	Finnigan MAT GCQ
	Column:	BPX5 capillary, 30 m × 0.22 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 15 °C/min to 280 °C (3 min)
	Injector:	300 °C
	Split ratio:	20/1
	Transfer line temp:	275 °C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	The retention time of the methyl esteris 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.	
	Methyl ester:	274, 272, 270, 268 (<1, 7, 21, 20; M^+); 237 (11), 235 (61), 233 (100); 215 (1), 213 (8), 211 (35), 209 (39); 201 (2), 199 (20), 197 (53). 195 (58) *m/z*
ES-MS:	Instrument:	TSQ 700 Finnigan MAT triple quadrupole
	Operation:	Negative ion mode
	Ionisation:	ESI spray voltage at 4.5 kV positive ion
	Scan Range:	50-600 *m/z*
	Peaks (% rel. intensity):	259, 257, 255, 253 (3, 29, 100, 99; M^+-H); 201, 199, 197, 195 (3, 25, 73, 86) *m/z*
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks:	1733, 1710, 1460, 1426, 1257, 1232, 1042, 858, 769 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.28 ppm)
	Spectral data:	δ 4.68 (2H, s), 7.36 (2H, s) ppm
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	125 MHz
	Solvent:	$CDCl_3$ (77.01 ppm)
	Spectral data:	δ 129.0, 129.7, 130.9, 148.8, 170.4 ppm
Melting point:		184.5-186 °C
Microanalysis:	Found:	C = 37.9%; H = 2.0% (June 2008)
	Calculated:	C = 37.6%; H = 2.0% (Calculated for $C_8H_5Cl_3O_3$)