Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

NATA
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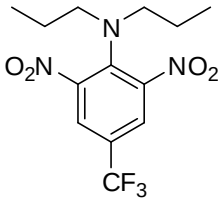
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1408b: Trifluralin



Report ID: P1408b.2022.01

Chemical Formula: $C_{13}H_{16}F_3N_3O_4$

Molecular Weight: 335.3 g/mol

Certified value

Batch No.	CAS No.	Purity (mass fraction)
21-AV-01	**1582-09-8**	**99.7 ± 0.4%**

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

IUPAC name: 2,6-Dinitro-N,N-dipropyl-4-(trifluoromethyl)aniline

Expiration of certification: The property values are valid till 14 September 2024, i.e. three years from the date of 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. The material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

Description: Orange needle 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.

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: At the recommended storage conditions this material has demonstrated stability for a period of at least ten years. The measurement uncertainty includes components for long term stability at the recommended storage conditions, and accelerated stability trials conducted at 40 ºC and 75% humidity for a 14 day period.

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 nine 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.
15 December 2022

This report supersedes any issued prior to 15 December 2022

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 quantitative NMR (qNMR), qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis. The purity value obtained by qNMR was determined using combination of the two-proton singlet at 8.50 ppm, the four-proton sextet at 1.49 ppm and the six-proton triplet at 0.8 ppm were measured against a certified internal standard of dimethyl fumarate.

GC-FID:	Instrument:	Agilent 8890
	Column:	HP-1MS, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	100 °C (0.5 min), 10 °C/min to 250 °C, 30 °C/min to 300 °C (3 min)
	Injector:	230 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.01% (9 sub samples in duplicate, September 2021)
	Re-analysis:	Mean = 99.8%, s = 0.01% (10 sub samples in duplicate, December 2022)
Karl Fischer analysis:		Moisture content ≤ 0.1% mass fraction (September 2021)
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction (September 2021)
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Internal standard:	Dimethyl fumarate (99.9% mass fraction)
	Initial analysis:	Mean (8.50 ppm) = 100.1%, s = 0.2% (5 sub samples, September 2021)
	Initial analysis:	Mean (1.49 ppm) = 99.7%, s = 0.04% (4 sub samples, September 2021)
	Initial analysis:	Mean (0.80 ppm) = 99.9%, s = 0.2% (5 sub samples, September 2021)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	DB-5MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	80 °C (1 min), 20 °C/min to 300 °C (3 min)
	Injector:	230 °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 trifluralin is 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 (8.2 min):	335 (M^+, 4), 316 (7), 306 (100), 290 (14), 264 (72), 248 (15), 206 (4) 145 (10), 43 (62) *m/z*
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μm
	Program:	50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)
	Injector:	150 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	Isopropanol
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform Single spot observed, R_f = 0.9. Visualisation with UV at 254 nm.
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2972, 2942, 2880, 1626, 1560, 1523, 1407, 1350, 1285, 1177, 1124, 904, 874, 709, 662 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 0.80 (6H, t, *J* = 7.3 Hz), 1.50 (4H, sextet, *J* = 7.3 Hz), 2.93 (4H, t, *J* = 7.3 Hz), 8.50 (2H, s) ppm
		Isopropanol was quantified at 0.1% mass fraction.
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 10.9, 20.5, 53.3, 120.6 (q, *J* = 35.4 Hz), 122.4 (q, *J* = 272 Hz), 126.9 (q, *J* = 3.6 Hz), 140.3, 145.5 ppm
Melting point:		48-50 °C
Microanalysis:	Found:	C = 46.6%; H = 4.7%; N = 12.6% (September, 2021)
	Calculated:	C = 46.6%; H = 4.8%; N = 12.5% (Calculated for $C_{13}H_{16}F_3N_3O_4$)