



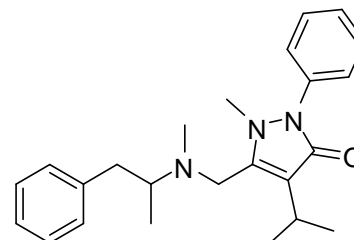
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D893: Famprofazone

Report ID: D893.2021.01 (Bottled 090407)

Chemical Formula: C₂₄H₃₁N₃O

Molecular Weight: 377.5 g/mol



Certified value

Batch No.

CAS No.

Purity (mass fraction)

05-D-10

22881-35-2

97.4 ± 0.4%

IUPAC name: 4-Isopropyl-1-methyl-5-[[methyl(1-phenyl-2-propanyl)amino]methyl]-2-phenyl-1,2-dihydro-3H-pyrazol-3-one

Expiration of certification: The property values are valid till 16 February 2026, i.e. five 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 sourced from an external supplier, 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 25 °C 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

Stability: This material has demonstrated stability over a minimum period of five 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 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.
15 March 2021

This report supersedes any issued prior to 15 March 2021

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 20844. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

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 ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

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

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: Varian-CP3800
 Column: VF-1MS, 30 m x 0.32 mm I.D. x 0.25 μm
 Program: 220 °C (1 min), 40 °C/min to 270 °C (10 min), 40 °C/min to 300 °C (1 min)
 Injector: 250 °C
 Detector Temp: 320 °C
 Carrier: Helium
 Split ratio: 20/1
 Relative mass fraction of main component:
 Initial analysis: Mean = 97.6%, s = 0.2% (7 sub samples in duplicate, August 2005)
 Re-analysis: Mean = 97.6%, s = 0.1% (5 sub samples in duplicate, July 2008)

GC-FID: Instrument: Agilent 7890
 Column: HP-1MS, 30m x 0.32 mm I.D. x 0.25 μm
 Program: 220 °C (1 min), 30 °C/min to 270 °C (10 min), 30 °C/min to 300 °C (3 min)
 Injector: 250 °C
 Detector Temp: 320 °C
 Carrier: Helium
 Split ratio: 20/1
 Relative mass fraction of main component:
 Initial analysis: Mean = 97.6%, s = 0.1% (5 sub samples in duplicate, July 2011)
 Re-analysis: Mean = 97.3%, s = 0.1% (5 sub samples in duplicate, April 2016)
 Re-analysis: Mean = 97.6%, s = 0.1% (5 sub samples in duplicate, February 2021)

Thermogravimetric analysis: Volatile content < 0.1% and non volatile residue < 0.2 % mass fraction (August 2005 and July 2006).

Karl Fischer analysis: Moisture content < 0.1% mass fraction (July 2008)
 Moisture content 0.3% mass fraction (July 2011)
 Moisture content 0.2% mass fraction (May 2016 and March 2021).

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5971A
	Column:	BPX-5, 30 m × 0.25 mm I.D. × 0.30 μm
	Program:	220 °C (2 min), 10 °C/min to 290 °C (5 min), 10 °C/min to 300 °C
	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 as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (12.3 min):	377 (M ⁺ , 3), 286 (100), 229 (31), 214 (8), 186 (4), 146 (5), 136 (17), 91 (48) <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Methanol/Conc NH ₃ (200/3) Single spot observed, R _f = 0.8. Visualisation with UV at 254 nm.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm ⁻¹ , KBr pellet
	Peaks:	3059, 2960, 1650, 1609, 1593, 1496, 1454, 1375, 1342, 1141, 1077, 1031, 765, 756, 735, 698 cm ⁻¹
¹ H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	CDCl ₃
	Spectral data:	δ 1.05 (3H, d, <i>J</i> = 6.6 Hz), 1.29 (6H, t, <i>J</i> = 6.7 Hz), 2.31 (3H, s), 2.56 (1H, dd, <i>J</i> = 7.0, 13.5 Hz), 2.75 (3H, s), 2.83 (1H, heptet, <i>J</i> = 7.0 Hz), 2.89 (1H, dd, <i>J</i> = 7.6, 13.5 Hz), 3.05 (1H, m), 3.51 (2H, s), 7.17-7.23 (4H, m), 7.26-7.31 (4H, m), 7.40 (2H, t, <i>J</i> = 7.9 Hz) ppm.
¹³ C NMR:	Instrument:	Bruker DMX-500
	Field strength:	125.5 MHz
	Solvent:	CDCl ₃
	Spectral data:	δ 13.0, 21.1, 21.2, 24.3, 35.0, 36.1, 39.8, 48.4, 59.4, 117.0, 123.4, 125.7, 125.9, 128.1, 128.8, 129.2, 135.2, 140.3, 151.7, 165.1 ppm
Melting point:	132-134 °C (Lit 132-133 °C)	
Microanalysis:	Found:	C = 76.3%, H = 8.3%, N = 11.2% (August 2005)
	Calculated:	C = 76.4%, H = 8.3%, N = 11.1% (Calculated for C ₂₄ H ₃₁ N ₃ O)