



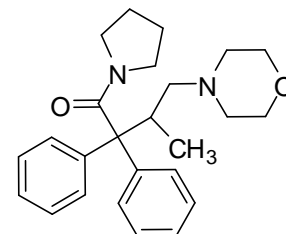
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D944: Racemoramide

Report ID: D944.2021.02

Chemical Formula: C₂₅H₃₂N₂O₂

Molecular Weight: 392.5 g/mol



Property value

Batch No.	CAS No.	Purity estimate
09-D-18	545-59-5	96.9 ± 1.9%

IUPAC name: 3-Methyl-4-(4-morpholinyl)-2,2-diphenyl-1-(1-pyrrolidinyl)-1-butanone.

Expiration of certification: The property values are valid till 18 June 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: 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 is recommended 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 °C in a closed container in a dry, dark area.

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

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.
13 October 2022

This report supersedes any issued prior to 20 September 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. Impurities of related structure were assessed by GC-FID. The purity estimate was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID. 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 thermogravimetric analysis, Karl Fischer analysis, ¹H NMR spectroscopy and elemental microanalysis.

GC-FID: Instrument: Varian CP-3800 or Agilent 7890
 Column: HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 200 °C (1 min), 2 °C/min to 230 °C (25 min), 30 °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 = 98.5%, s = 0.06% (5 sub samples in duplicate, July 2011)
 Re-analysis: Mean = 98.6%, s = 0.03% (5 sub samples in duplicate, May 2012)
 Re-analysis: Mean = 98.4%, s = 0.04% (6 sub samples in duplicate, April 2015)
 Re-analysis: Mean = 98.4%, s = 0.09% (5 sub samples in duplicate, March 2018)
 Re-analysis: Mean = 98.7%, s = 0.05% (3 sub samples in duplicate, June 2021)

Karl Fischer analysis: Moisture content < 0.2% mass fraction (June 2009, July 2010, June 2011 and May 2012)
 Moisture content 0.2% mass fraction (April 2015)
 Moisture content 0.6% mass fraction (March 2021)

Thermogravimetric analysis: Initial volatile content 0.4% and non volatile residue 0.8% mass fraction (June 2009)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	VF-1MS, 15 m × 0.25 mm I.D. × 0.25 μm
	Program:	180 °C (1 min), 10 °C/min to 300 °C (2 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 as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent compound (10.3 min):	306 (2), 265 (75), 236 (4), 194 (4), 178 (3), 165 (8), 128 (52), 100 (100), 70 (4), 56 (10), 42 (4) <i>m/z</i>
ESI-MS:	Instrument:	Micromass Quatro Micro
	Operation:	Positive ion mode, direct infusion at 5 μL/min
	Ionisation:	ESI spray voltage at 3.2 kV positive ion
	EM voltage:	500 V
	Cone voltage:	30 V
	Peak:	393 (M+H) ⁺ <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Chloroform/ethyl acetate (4/1) Single spot observed, R _f = 0.20. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm ⁻¹ , KBr powder
	Peaks:	2969, 2882, 2851, 2797, 1620, 1395, 1114, 1008, 861, 704 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance III 500
	Field strength:	500 MHz
	Solvent:	CDCl ₃ (7.26 ppm)
	Spectral data:	δ 0.76 (3H, d, <i>J</i> = 6.7 Hz), 1.19 (1H, bt, <i>J</i> = 10.5 Hz), 1.35-1.48 (2H, m) 1.50-1.69 (2H, m), 2.12-2.20 (2H, m), 2.28-2.40 (2H, m), 2.56-2.68 (3H, m), 3.24 (1H, m), 3.45-3.67 (6H, m), 7.22-7.38 (6H, m), 7.41-7.48 (4H, m) ppm. Hexane at 0.5% mass fraction was observed in the ¹ H NMR (June 2009 and July 2010)
¹³ C NMR:	Instrument:	Bruker Avance III 500
	Field strength:	126 MHz
	Solvent:	CDCl ₃ (77.00 ppm)
	Spectral data:	δ 15.6, 23.0, 26.1, 39.2, 47.5, 54.4, 63.2, 63.7, 67.3, 126.4, 126.7, 127.8, 127.9, 129.0, 129.2, 137.3, 138.9, 172.1 ppm
Melting point:		168-170 °C
Microanalysis:	Found:	C = 76.3%; H = 8.4%; N = 7.2% (June 2009)
	Calculated:	C = 76.5%; H = 8.2%; N = 7.1% (Calculated for C ₂₅ H ₃₂ N ₂ O ₂)