Australian Government
Department of Industry, Science and Resources

National Measurement Institute

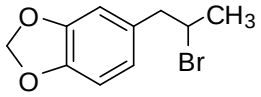
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D455: 2-Bromosafrole

Report ID: D455.2021.02

Chemical Formula: $C_{10}H_{11}BrO_2$

Molecular Weight: 243.1 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
96-006658	**5463-71-8**	**92.4 ± 0.8%**

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

Synonyms: 2-Bromosaffrole
1-(3,4-Methylenedioxyphenyl) -2-bromopropane
5-(2-Bromopropyl)-1,3-benzodioxole

Expiration of certification: The property values are valid till 16 August 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: Pale yellow oil prepared by synthesis, 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 may be used for instrument calibration.

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 five years. The measurement uncertainty at the 95% coverage 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 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 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.
14 September 2022

This report supersedes any issued prior to 14 September 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 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 and quantitative NMR with dimethyl terephthalate as the internal standard.

GC-FID:	Instrument:	Agilent 6890
	Column:	HP-1 capillary, 30 m x 0.32 mm I.D. x 0.25 μm film thickness
	Program:	80 °C (1 min), 40 °C /min to 130 °C (14 min), 30 °C /min to 300 °C (1 min)
	Injector:	150 °C
	Detector:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	98% (December 1995)
	Re-analysis:	Mean = 93.0%, s = 0.06% (5 sub samples in duplicate, August 2008)
	Re-analysis:	Mean = 92.7%, s = 0.05% (5 sub samples in duplicate, September 2013)
GC-FID:	Instrument:	Agilent 8890
	Column:	HP-1MS, 30 m x 0.32 mm x 0.25 μm
	Program:	80 °C (1 min), 20 °C /min to 140 °C (14 min), 20 °C /min to 300 °C (3 min)
	Injector:	150 °C
	Detector:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 92.9%, s = 0.05% (5 sub samples in duplicate, August 2021)
QNMR:	Instrument:	Bruker DMX-400
	Field strength:	400 MHz
	Solvent:	$CDCl_3$
	Internal standard:	Dimethyl terephthalate
	Purity estimate:	92.6 % (mass fraction, 5 sub samples, s = 0.52%, July 2008)
Karl Fischer Analysis:		Moisture content 0.14% mass fraction (July 2008) Moisture content < 0.1% mass fraction (September 2013 and August 2021)

Spectroscopic and other characterisation data

HPLC:	Method:	Peak area percentage of total = 96.6%, (average of replicates)
	Column:	Radial-pak cartridge Novapak C-18, 4 μm x 8 mm x 100 mm
	Mobile Phase:	Methanol/water/diethylamine (60/40/0.5) pH 8.5 buffered with orthophosphoric acid 50%
	Flow Rate:	1.2 mL/min
	Wavelength:	255 nm
GC-MS:	Instrument:	Agilent 6890/5973
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.25 μm
	Temp Program:	100 °C (2 min), 40 °C/min to 140 °C (12 min), 30 °C/min to 300 °C (2 min)
	Injector Temp:	150 °C
	Transfer line temp:	320 °C
	Carrier gas:	Helium, 1 mL/min
	Split ratio:	20/1
	Peaks:	244, 242, 163, 135, 105, 77, 51 *m/z* Conforms with the published spectrum for 2-bromosaffrole
TLC:	Conditions:	Kieselgel 60F_{254}. Diisopropylether/diethylether/diethylamine (45/45/10) Single spot observed, R_f = 0.6
IR:	Instrument:	FT-IR, BIORAD WIN FTS40
	Range:	4000-400 cm^{-1} (liquid film on KCl)
	Peaks:	1490, 1444, 1251, 1040, 930, 810 cm^{-1}
^{1}H NMR	Instrument:	Bruker DMX600
	Field Strength:	600 MHz
	Solvent:	$CDCl_3$
	Spectral Data	δ 1.69 (3H, d, *J* = 6.6 Hz), 2.98 (1H, dd, *J* = 7.2, 14.0 Hz), 3.14 (1H, dd, *J* = 7.0, 14.1 Hz), 4.21-4.27 (1H, m), 5.95 (2H, s), 6.66 (1H, dd, $J_{5,6}$ = 7.9, $J_{2,6}$ = 1.5 Hz), 6.70 (1H, d, $J_{2,6}$ = 1.5 Hz), 6.76 (1H, d, $J_{5,6}$ = 6.8 Hz) ppm
^{13}C NMR	Instrument	Bruker DMX600
	Field Strength	600 MHz
	Spectral Data	δ: 26.0, 47.6, 51.3, 101.4, 108.6, 109.9, 122.7, 132.7, 146.8, 148.0 ppm
Microanalysis:	Found:	C = 50.5%, H = 4.8 % (July 2008)
	Calculated:	C = 49.4%, H = 4.6% (Calculated for $C_{10}H_{11}BrO_2$)