



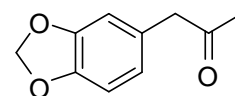
# REFERENCE MATERIAL PRODUCT INFORMATION SHEET

## NMIA D810b: 3,4-Methylenedioxyphenyl acetone

Report ID: D810b.2020.03

Chemical Formula: C<sub>10</sub>H<sub>10</sub>O<sub>3</sub>

Molecular Weight: 178.2 g/mol



### Property value

Batch No.	CAS No.	Purity estimate
08-D-08	4676-39-5	93.2 ± 1.7%

**IUPAC name:** 1-(1,3-Benzodioxol-5-yl)acetone

**Expiration of certification:** The property values are valid till 14 May 2023, i.e. three 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, and certified for identity and purity by NMIA. Packaged in amber glass ampoule.

**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 4 °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.  
15 September 2022

This report supersedes any issued prior to 15 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.

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### 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 value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID and Karl Fisher analysis. The purity value is calculated as per Equation 1.

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

$I_{\text{ORG}}$  = Organic impurities of related structure,  $I_{\text{VOL}}$  = volatile impurities,  $I_{\text{NVR}}$  = non-volatile residue.

Supporting evidence is provided by headspace GC-MS.

**Warning:** This material is sensitive to the quality of the silanised glass liner when injected at elevated temperature (~ 250 °C) into a GC instrument.

GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	80 °C (1 min), 15°C/min to 100 °C, 20 °C/min to 300 °C (5 min)
	Injector:	180 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 98.2%, s = 0.3% (10 injections, February 2008)
	Re-analysis:	Mean = 95.4%, s = 0.05% (5 sub samples in duplicate, May 2011)
	Re-analysis:	Mean = 94.5%, s = 0.1% (5 sub samples in duplicate, April 2014)
	Re-analysis:	Mean = 92.8%, s = 0.1% (5 sub samples in duplicate, March 2017)
	Re-analysis:	Mean = 93.4%, s = 0.03% (5 sub samples in duplicate, May 2020)
Karl Fischer analysis:	Moisture content 0.2% mass fraction (June 2011)	
	Moisture content 0.3% mass fraction (March 2014, 2017 and 2020)	

### Spectroscopic and other characterisation data

GC-MS: Instrument: Agilent 5890/5971  
Column: ZB-5, 30 m x 0.25 mm I.D. x 0.25  $\mu$ m  
Program: 60 °C (1 min), 10 °C/min to 250 °C (1 min)  
Injector: 250 °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 the parent compound 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 (10.6 min): 178 ( $M^+$ , 30), 136 (11), 135 (100), 105 (5), 77 (17), 51 (8), 43 (6) *m/z*

IR: Instrument: Biorad WIN FTS3000 MX FTIR  
Range: 4000-400  $\text{cm}^{-1}$ , NaCl plates  
Peaks: 2901, 1709, 1488, 1355, 1238, 1030, 924  $\text{cm}^{-1}$

$^1\text{H}$  NMR: Instrument: Bruker DMX-600  
Field strength: 600 MHz  
Solvent:  $\text{CDCl}_3$  (7.26 ppm)  
Spectral data:  $\delta$  2.14 (3H, s), 3.60 (2H, s), 5.94 (2H, s), 6.63 (1H, dd,  $J = 1.4, 7.9$  Hz), 6.67 (1H, d,  $J = 1.4$  Hz), 6.76 (1H, d,  $J = 7.9$  Hz) ppm

$^{13}\text{C}$  NMR: Instrument: Bruker DMX-600  
Field strength: 150 MHz  
Solvent:  $\text{CDCl}_3$  (77.2 ppm)  
Spectral data:  $\delta$  29.0, 50.4, 101.0, 108.4, 109.7, 122.4, 127.7, 146.6, 147.8, 206.5 ppm

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