



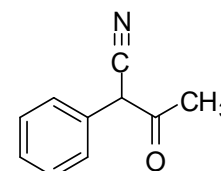
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D965: α -Acetylphenylacetone nitrile

Report ID: D965.2022.01

Chemical Formula: C₁₀H₉NO

Molecular Weight: 159.2 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
11-D-03	4468-48-8	99.4 ± 0.6%

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

IUPAC name: 3-Oxo-2-phenylbutanenitrile.

Expiration of certification: The property values are valid till 5 September 2027, 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 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 three years. The measurement uncertainty at the 95% confidence 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 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.
23 September 2022

This report supersedes any issued prior to 23 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. The certified purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include 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

The purity value by qNMR was obtained using the three-proton singlet at 2.25 ppm measured against a certified internal standard of dimethyl sulfone.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument: Varian CP-3800 Column: TG-17MS, 30 m × 0.32 mm I.D. × 0.25 μm Program: 70 °C (1 min), 20 °C/min to 200 °C, 30 °C/min to 280 °C (10 min) Injector: 250 °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% (10 sub samples in duplicate, March 2011) Re-analysis: Mean = 99.9%, s = 0.01% (4 sub samples in duplicate, October 2014) Re-analysis: Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, July 2017)
GC-FID:	Instrument: Varian CP-3800 Column: HP-5, 30 m × 0.32 mm I.D. × 0.25 μm Program: 80 °C (1 min), 15 °C/min to 200 °C, 20 °C/min to 300 °C (3min) Injector: 250 °C Detector Temp: 320 °C Carrier: Helium Split ratio: 20/1 Relative mass fraction of the main component: Initial analysis: Mean = 99.95%, s = 0.003% (10 sub samples in duplicate, March 2011)
HPLC:	Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT auto sampler Column: ACE Excel Amide C-18, 5 μm (4.6 mm x 250 mm) Column oven: 40 °C Mobile Phase: A = Milli-Q water; B = Acetonitrile both with 0.1% TFA 0-8min 40%B, 8-10min 40-85%B, 10-15min 85%B, 15-17min 85-40%B, 17-23min 40%B Flow rate: 1.0 mL/min Detector: Shimadzu SPD-M20A PDA operating at 263 nm Relative mass fraction of the main component: Initial analysis: Mean = 99.7%, s = 0.09% (5 sub samples in duplicate, September 2022)
Karl Fischer analysis:	Moisture content < 0.1% mass fraction (March 2011) Moisture content 0.2% mass fraction (October 2014) Moisture content 0.1% mass fraction (August 2017) Moisture content < 0.1% mass fraction (June 2022)
Thermogravimetric analysis:	The volatile content could not be determined due to the inherent volatility of this material. The non volatiles are below the detection limit (March 2011)
QNMR:	Instrument: Bruker Avance-400 Field strength: 400 MHz Solvent: CDCl ₃ (7.26 ppm) Internal standard: Dimethyl sulfone (100.0% mass fraction) Initial analysis: Mean (2.25 ppm) = 99.11%, s = 0.06% (5 sub samples, April 2011)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μ m
	Program:	60 °C (1 min), 10 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	30/1
	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 (9.6 min):	159 (M^+ , 5), 117 (100), 89 (20), 63 (10), 43 (70) m/z
ESI -MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	5 V
	Peak:	160.2 ($M+H^+$) m/z
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μ m
	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:	Chloroform, hexane and ethyl acetate
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1} , KBr powder
	Peaks:	3134, 2987, 2700, 2216, 1637, 1497, 1389, 1364, 1336, 1306, 1281, 1023, 918, 762, 690 cm^{-1}
1H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 2.26 (3H, s), 4.69 (1H, s), 7.32-7.48 (5H, m) ppm.
	Keto-enol tautomers were observed for α -acetylphenylacetonitrile. Ethyl acetate estimated at 0.08% and n-hexane estimated at 0.03% mass fractions were observed in the 1H NMR ($CDCl_3$). Chloroform estimated at 0.07% mass fraction was observed in the 1H NMR (CD_3OD)	
^{13}C NMR:	Instrument:	Bruker Avance III-400
	Field strength:	100 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data:	δ 27.0, 51.6, 116.3, 128.1, 129.5, 129.8, 129.8, 196.6 ppm
Melting point:	89-93 °C	
Microanalysis:	Found:	C = 75.6%; H = 5.7%; N = 8.8% (April 2011)
	Calculated:	C = 75.5%; H = 5.7%; N = 8.8% (Calculated for $C_{10}H_9NO$)