



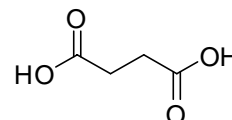
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA QNMR018: Succinic acid

Report ID: QNMR018.2023.01

Chemical Formula: C₄H₆O₄

Molecular Weight: 118.1 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
20-Q-03	110-15-6	99.3 ± 0.4%

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

IUPAC name: Succinic acid

Expiration of certification: The property values are valid till 14 June 2026, 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. The material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

Description: Off-white powder sourced from an external supplier, and certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium 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. 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: In the absence of long term stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last ten years. The measurement uncertainty at the 95% confidence interval also includes a stability component determined from accelerated stability trials conducted at 40 °C and 75% humidity for a 14 day period.

Homogeneity assessment: The homogeneity of the material was assessed using purity assay by qNMR on eight randomly selected 20 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.
8 August 2023

This report supersedes any issued prior to 8 August 2023

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 traditional mass balance combining ^1H NMR to determine the ratio of succinic acid and fumaric acid in the sample, thermogravimetric analysis and Karl Fischer analysis.

Supporting evidence is provided by quantitative nuclear magnetic resonance (qNMR), qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis. The purity value by qNMR was obtained using the four-proton singlet at 2.68 ppm measured against a certified internal standard of dimethyl sulfone.

QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	D ₂ O (4.79 ppm)
	Internal standard:	Dimethyl sulfone (100.0% mass fraction)
	Initial analysis:	Mean (2.68 ppm) = 99.5%, s = 0.2% (8 sub samples, January 2021)

Karl Fischer analysis:	Moisture content 0.6% mass fraction (January 2021)
	Moisture content 0.7% mass fraction (May 2022)
	Moisture content 0.6% mass fraction (January 2023)
	Moisture content 0.5% mass fraction (June 2023)
Thermogravimetric analysis:	Volatiles content 0.3% and non-volatile residue < 0.01% mass fraction (October 2020)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Negative ion mode, direct infusion at 20 $\mu\text{L}/\text{min}$
	Ionisation:	ESI spray voltage at 2.5 kV negative ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	116.8 $[\text{M-H}]^-$ m/z
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μm
	Program:	50 $^{\circ}\text{C}$ (5 min), 7 $^{\circ}\text{C}/\text{min}$ to 120 $^{\circ}\text{C}$, 15 $^{\circ}\text{C}/\text{min}$ to 220 $^{\circ}\text{C}$ (8.3 min)
	Injector:	150 $^{\circ}\text{C}$
	Transfer line temp:	280 $^{\circ}\text{C}$
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	None
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1} , neat
	Peaks:	2929, 2728, 2625, 2532, 1683, 1407, 1303, 1194, 1174, 891, 800, 580 543 cm^{-1}
^1H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 2.68 (4H, s) ppm Fumaric acid estimated at 0.1% mass fraction was observed in the ^1H NMR
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	D_2O
	Spectral data:	δ 28.7, 177.0 ppm
Melting point:		190-193 $^{\circ}\text{C}$
Microanalysis:	Found:	C = 40.7%; H = 5.3% (October, 2020)
	Calculated:	C = 40.7%; H = 5.1% (Calculated for $\text{C}_4\text{H}_6\text{O}_4$)