



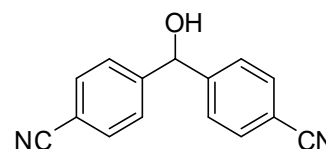
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D909c: Bis-(4-cyanophenyl) methanol

Report ID: D909c.2019.01 (Ampouled 170525)

Chemical Formula: C₁₅H₁₀N₂O

Molecular Weight: 234.2 g/mol



Certified value

Batch No.	CAS No.	Mass per ampoule
13-D-33	134521-16-7	993 ± 46 µg

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

IUPAC name: 4,4'-(Hydroxymethylene)dibenzonitrile.

Expiration of certification: The property values are valid till 18 July 2022, 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: The compound is supplied as a dried aliquot in a sealed ampoule and is intended for a single use to prepare a standard solution containing D909c. This material was prepared by synthesis, certified for identity and purity by NMIA.

Intended use: This certified reference material is suitable for use as a primary calibrator.

Instructions for use: Open the ampoule and carefully rinse the interior at least three times with a suitable organic solvent (e.g. methanol). This will transfer 993 ± 46 µg of anhydrous bis-(4-cyanophenyl) methanol.

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.

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%.

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 HPLC with UV detection on seven randomly selected ampoules 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 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.
12 August 2019

This report supersedes any issued prior to 12 August 2019

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 20844. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

Legal notice: Neither NMIA as a representative of the Commonwealth of Australia, nor any person acting on NMIA's behalf, assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this document.

Characterisation Report:

HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Alltima C-18, 5 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A = MilliQ water; B = Methanol 0-12 min 45% B; 12-17 min 45-80% B; 17-21 min 80% B; 21-22 min 80-45% B; 22-30 min 45% B
	Flow rate:	1 mL/min
	Detector:	Waters 2998 PDA operating at 238 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.7%, s = 0.04% (5 ampoules in duplicate, July 2019)
HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Alltima C-18, 5 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A = MilliQ water; B = Acetonitrile 0-12 min 45% B; 12-17 min 45-80% B; 17-25 min 80% B; 25-28 min 80-45% B; 28-45 min 45% B
	Flow rate:	1 mL/min
	Detector:	Shimadzu SPD-M20A PDA operating at 238 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.5%, s = 0.09% (7 ampoules in duplicate, June 2017)
	Re-analysis:	Mean = 99.6%, s = 0.02% (5 ampoules in duplicate, July 2018)

The following analytical data was obtained on the bulk material subsequently used in the preparation of the ampoules.

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 HPLC with UV detection, 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.

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

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Alltima C-18, 5 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A = MilliQ water; B = Acetonitrile 0-12 min 45% B; 12-17 min 45-80% B; 17-25 min 80% B; 25-28 min 80-45% B; 28-45 min 45% B
	Flow rate:	1mL/min
	Detector:	Shimadzu SPD-M20A PDA operating at 238 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.3%, s = 0.04% (10 sub samples in duplicate, December 2013)
	Re-analysis:	Mean = 99.5%, s = 0.07% (5 sub samples in duplicate, December 2014)
Thermogravimetric analysis:	Volatile content 0.4% and non volatile residue < 0.2% mass fraction (November 2013)	
Karl Fischer analysis:	Moisture content < 0.1% mass fraction (December 2013 and October 2014)	

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:	200 °C (1 min), 10 °C/min to 250 °C (8 min), 20 °C/min to 300 °C (3 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 (7.14 min):	234 (M^+ , 15), 233 (15), 218 (28), 190 (11), 130 (100), 104 (45), 102 (34), 77 (13) 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, acetone
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Chloroform/acetone (23/2) Single spot observed, R_f = 0.38. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1} , KBr powder
	Peaks:	3431, 3330, 3092, 3057, 2227, 1927, 1603, 1499, 1410, 1324, 1194, 1065, 874, 816, 766 cm^{-1}
¹ H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Solvent:	CD ₃ OD (3.31 ppm)
	Spectral data:	δ 5.90 (1H, s), 7.58 (4H, m), 7.69 (4H, m) ppm Chloroform and acetone estimated at 0.3% and 0.1% mass fraction respectively were observed in the ¹ H NMR
¹³ C NMR:	Instrument:	Bruker DMX-500
	Field strength:	126 MHz
	Solvent:	CDCl ₃ (77.0 ppm)
	Spectral data:	δ 102.0, 106.9, 108.3, 128.6, 131.9, 148.7, 153.1, 190.2 ppm
Microanalysis:	Found:	C = 76.6%; H = 4.4%; N = 11.9% (December, 2013)
	Calculated:	C = 76.9%; H = 4.3%; N = 12.0% (Calculated for C ₁₅ H ₁₀ N ₂ O)