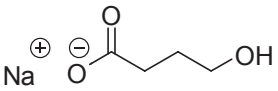
REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D812b.2015.01

Compound Name: **Sodium γ-hydroxybutyrate**
Collection Number: D812b
Chemical Formula: $C_4H_7O_3Na$
CAS Number: 502-85-2
Structure:



Description: White powder
Batch Number: 12-D-18
Molecular Weight: 126.1
Release date: 27th March 2013

Synonyms: Sodium oxybate
4-Hydroxybutyric acid sodium salt
GHB (sodium salt)

Purity (mass fraction): 99.3 ± 3.0% (95% coverage interval)

Sodium γ-hydroxybutyrate is extremely hygroscopic. In a closed container this material has been shown to absorb more than 10% moisture and in some cases the sample has become liquid. This material should be considered for use in qualitative analysis only.

The purity value was obtained from quantitative nuclear magnetic resonance (QNMR). The purity estimate by QNMR was obtained using a combination of the two-proton triplet at 3.57 ppm, the two-proton triplet at 2.21 ppm and the two-proton multiplet at 1.79 ppm against a certified internal standard of potassium hydrogen maleate. Supporting evidence is provided by headspace GC-MS analysis of occluded solvents and elemental microanalysis.

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Ascentis C-18, 2.7 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	Acetonitrile/MilliQ water (15:85) The aqueous phase was buffered at pH 3.3 using 20mM NH_4HCO_2 and HCO_2H
	Flow rate:	0.15 mL/min
	Detector:	Shimadzu LT-II ELSD
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.9%, s = 0.08% (10 sub samples in duplicate, March 2013)
Thermogravimetric analysis:		Volatile content < 0.1%. Non volatile residue not determined (March 2013)
Karl Fischer analysis:		Moisture content 1.2% mass fraction (April 2015)
QNMR:	Instrument:	Bruker Avance-III-400
	Field strength:	400 MHz Solvent: D_2O (4.79 ppm)
	Internal standard:	Potassium hydrogen maleate (99.4% mass fraction)
	Re-analysis:	Mean (3.57 ppm) = 99.3%, s = 0.3% (3 sub samples, March 2014)
	Re-analysis:	Mean (1.79 ppm) = 99.3%, s = 0.3% (3 sub samples, March 2014)
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz Solvent: D_2O (4.79 ppm)
	Internal standard:	Potassium hydrogen maleate (100% mass fraction)
	Re-analysis:	Mean (3.56 ppm) = 99.5%, s = 0.3% (6 sub samples, April 2015)
	Re-analysis:	Mean (2.21 ppm) = 99.5%, s = 0.3% (6 sub samples, April 2015)
	Re-analysis:	Mean (1.79 ppm) = 99.5%, s = 0.3% (6 sub samples, April 2015)

Spectroscopic and other characterisation data

LC-MS:	Instrument:	Waters 2695 (HPLC)/Micromass Quatro	
	Column:	Ascentis C-18, 150 mm × 4.6 mm I.D. × 2.7 μm	
	Column temp:	40 °C	
	Solvent system:	A 2% Formic acid in MilliQ water [10% v/v] B Acetonitrile [15% v/v] C MilliQ water [75% v/v]	
	Flow rate:	0.15 mL/min	
	Sample prep:	100 μg/g in MeOH/MilliQ water (25:75)	
	Injection volume:	30 μL	
	Ionisation mode:	Electrospray negative ion	
	Capillary voltage:	3.0 kV	Cone voltage: 25 V
	Source temp:	130 ºC	Desolvation gas temperature: 350 ºC
	Cone gas flow rate:	27 L/hr	Desolvation gas flow rate: 750 L/hr
	The retention time of sodium γ-hydroxybutyrate is reported along with the major peak in the mass spectrum. The latter is reported as a mass/charge ratio.		
	9.80min:	102.9 ($M\text{-}H^+$) 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 °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:	Acetone	
IR:	Instrument:	Biorad FTS3000MX FT-IR	
	Range:	4000-400 cm^{-1}, KBr powder	
	Peaks:	3341, 2941, 2875, 1554, 1420, 1069, 1017, 922, 869, 668 cm^{-1}	
^{1}H NMR:	Instrument:	Bruker Avance-400	
	Field strength:	400 MHz	Solvent: D_2O (4.79 ppm)
	Spectral data:	δ 1.76 (2H, quintet, J = 6.8 Hz), 2.20 (2H, d, J = 7.9 Hz), 3.57 (2H, d, J = 6.8 Hz) ppm	
		Butyrolactone and γ-hydroxybutyrate methyl ester were both estimated at 0.1% mass fraction each in the ^{1}H NMR.	
^{13}C NMR:	Instrument:	Bruker Avance-400	
	Field strength:	101 MHz	Solvent: D_2O
	Spectral data:	δ 28.4, 34.0, 61.5, 183.1 ppm	
Melting point:		146-149 °C	
Microanalysis:		Found: C = 37.9%; H = 5.5% (March, 2013) Calc: C = 38.1%; H = 5.6% (Calculated for $C_4H_7O_3Na$)	

Expiration of certification
The property values are valid till 29th April 2018, 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 long-term stability of the compound in solution has not been examined.

This material has been given a shelf life of three years from the date of re-certification.

Homogeneity assessment
The homogeneity of the material was assessed using purity assay by HPLC with ELS detection on ten 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.

Recommended storage
When not in use, this material should be stored at or below 25 ºC in a closed container, protected from ambient moisture and light.

Intended Use
For ***in vitro*** laboratory analysis only.

Caution
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice
Neither NMI nor any person acting on NMI'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 certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 30 April, 2015.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 30th April 2015.