

# Certificate of measurement



4005

## Soft drinking water - metals

Certified Reference Material LGC6027

### Certified values

Constituent <sup>1,2</sup>	Number of laboratories	Units	Certified value <sup>3</sup>	Uncertainty <sup>4</sup>	Coverage factor, $k$ <sup>4</sup>
Aluminium	18	µg/L	196.1	7.3	2.09
Antimony	16	µg/L	5.21	0.24	2.13
Arsenic	17	µg/L	10.00	0.35	2.09
Barium	18	µg/L	115.7	3.5	2.10
Beryllium	15	µg/L	5.09	0.22	2.10
Boron	17	µg/L	1006	49	2.11
Cadmium	17	µg/L	5.09	0.24	2.11
Chromium	18	µg/L	49.9	1.1	2.06
Cobalt	16	µg/L	4.87	0.17	2.12
Copper	18	µg/L	1995	66	2.03
Iron <sup>1</sup>	1	µg/L	200.0	2.6	2.18
Lead <sup>1</sup>	1	µg/L	10.15	0.20	2.00
Lithium	11	µg/L	10.41	0.64	2.20
Manganese	18	µg/L	49.9	1.1	2.09
Molybdenum	13	µg/L	4.62	0.40	2.18
Nickel	18	µg/L	20.01	0.50	2.07
Selenium	17	µg/L	10.21	0.39	2.11
Strontium	13	µg/L	496	24	2.14
Thallium	10	µg/L	4.88	0.32	2.26
Uranium	10	µg/L	4.95	0.26	2.26
Vanadium	14	µg/L	4.93	0.21	2.16
Zinc	18	µg/L	613	19	2.10

  

Calcium <sup>1</sup>	1	mg/L	8.53	0.16	2.00
Magnesium	17	mg/L	1.026	0.035	2.10
Potassium	18	mg/L	0.367	0.023	2.09
Sodium	16	mg/L	4.36	0.29	2.05

### Notes:

- 1) Calcium, iron and lead were determined at LGC using Isotope Dilution Inductively Coupled Plasma Mass Spectrometry (IDMS).
- 2) The remaining constituents were certified using the results of an inter-laboratory comparison using different methods (see Table 1). Robust estimates of dispersion and location were used as the values of the concentration determined by participant laboratories. Each data set was obtained in a different laboratory and/or using a different method of measurement.
- 3) The results are traceable to the SI through the physical and chemical standards used by LGC and the inter-laboratory study participant laboratories. The certified values are reported to the same number of decimal places as the uncertainties (reported to 2 significant figures).
- 4) The quoted uncertainty is the half-width of the expanded uncertainty interval calculated using a coverage factor ( $k$ ), which gives a level of confidence of approximately 95 %.

Date of Issue: November 2018

Signed: \_\_\_\_\_

Gill Holcombe (Mrs)

for the Government Chemist

Material number: LGC6027

Batch number: 001

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## Material Preparation

Soft drinking water was sourced from the Bury (Lancashire, UK) potable mains supply. The water was filtered through a set of 8 µm, 1.2 µm and 0.2 µm in-line filters and acidified by addition of nitric acid to give a final concentration of approximately 0.1 % nitric acid and pH < 2.0. High purity metal standards were used to spike the base material to reach the target levels, reflecting the regulatory limits in the European Drinking Water Directive <sup>[1]</sup> and the UK Water Supply (Water Quality) Regulations 2016 <sup>[2]</sup>.

The material was bottled in 250 mL portions in screw cap high density polyethylene bottles and stored at (5 ± 4) °C.

## Homogeneity Assessment

The homogeneity of the material was assessed for each of the elements of interest. Analysis was carried out at LGC by inductively coupled plasma optical emission spectrometry (ICP-OES) for boron, copper, magnesium, potassium, sodium, strontium and zinc, with all other elements analysed by inductively coupled plasma mass spectrometry (ICP-MS) apart from calcium, iron and lead where an assessment was made using IDMS.

The results showed the material was fit-for-purpose for a sample size of 1 mL which is the recommended minimum sample intake.

## Stability

The nature of the material is such that deterioration is not anticipated over its lifetime when stored under the recommended conditions. The material will be monitored at LGC and customers will be notified of any changes in the certified values.

## Certification

The material was certified using results from characterisation studies organised by LGC. This was a combination of IDMS measurements carried out at LGC (for calcium, iron and lead) and an inter-laboratory study. The decision to use IDMS for the characterisation of the material for calcium, iron and lead was based on, respectively, the method bias observed from the inter-laboratory results for calcium (ICP-MS vs ICP-OES), the large spread of the results observed for iron and the known difficulties of the measurement of lead.

### IDMS characterisation

Samples were analysed at LGC using a method accredited to ISO/IEC 17025. The samples were spiked with a solution containing enriched <sup>42</sup>Ca (ORNL, USA), <sup>57</sup>Fe (Isoflex, USA) and <sup>206</sup>Pb (NIST, USA). Ten units were analysed in duplicate over two batches using two independent sets of calibrants.

Measurements were performed using the Agilent 8800 ICP-QQQ-MS in H<sub>2</sub> mode for iron, and in standard mode for lead. For calcium, the Element2 (SN03936E) was used. The doubly charged Sr<sup>2+</sup> produces an interference at masses 42 and 44, which are the masses measured for the determination of calcium. Thus, a higher concentration of calcium would be determined in samples if this interference is not removed. The Agilent 8800 does not have the capability to eliminate the Sr<sup>2+</sup> contribution, hence the use of the Element2 in high resolution.

The samples were bracketed with the calibration standard blend. The primary standard was NIST SRM 3109a for calcium, NIST SRM 3126a for iron and NIST SRM 3128 for lead. Independent standards were prepared from secondary sources.

The certified value was assigned as the mean of the duplicate measurements.

### Interlaboratory study characterisation

Units of the candidate material were distributed to laboratories that had previously agreed to participate in the inter-laboratory study. Participant laboratories chose suitable methods with which they were familiar which included ICP-MS, ICP-OES and IC (ion chromatography). The number of laboratories that used a particular method is shown in Table 1.

The data from the inter-laboratory study were processed using a robust statistical approach, after screening laboratories based on their performance in analysing a separate QC sample. The certified value for each constituent was assigned as the robust estimate of location of the accepted laboratory data. Uncertainties were based on the robust estimate of dispersion (taking into account the number of laboratories and corrected for the efficiency of the estimator), and combined with the uncertainties related to homogeneity and stability.

**Table 1**

Constituent	ICP-MS	ICP-OES	IC	Total
Aluminium	12	6		18
Antimony	16			16
Arsenic	16	1		17
Barium	14	4		18
Beryllium	13	2		15
Boron	12	5		17
Cadmium	16	1		17
Chromium	14	4		18
Cobalt	14	2		16
Copper	14	4		18
Lithium	7	4		11
Magnesium	10	7		17
Manganese	13	5		18
Molybdenum	10	3		13
Nickel	16	2		18
Potassium	10	7	1	18
Selenium	17			17
Sodium	10	6		16
Strontium	9	4		13
Thallium	10			10
Uranium	10			10
Vanadium	13	1		14
Zinc	14	4		18

## Participants

The following laboratories participated in the inter-laboratory study for this material:

Affinity Water	UK
Anglian Water	UK
British Geological Survey	UK
Cavendish Nuclear	UK
Concept Life Sciences	UK
CSIR Stellenbosch Environmental Laboratory	South Africa
DCWW Welsh water	UK
Edinburgh Scientific Services	UK
Environmental Protection Agency - Ireland	Ireland
FIZ GmbH	Germany
i2 Analytical Ltd	UK
Northern Ireland Water	UK
Northumbrian Water Scientific Services	UK
Seibersdorf Labor GmbH	Austria
South East Water	UK
Severn Trent Water	UK
South East Water	UK
South West Water	UK
Tayside Scientific Services	UK
Thames Water	UK
The Institute of Naval Medicine	UK
Wessex Water	UK
Affinity Water	UK

## Accreditation

Property values on this certificate are within LGC's scope of accreditation to ISO 17034.

## Intended Use

This reference material is primarily intended for use in the development, validation or quality control of analytical methods for the determination of metals in soft drinking water. The material may also be applicable to other similar matrices where more clearly matched reference materials are not available.

## Instructions for Use

Before use, the material should be thoroughly mixed by inversion, and allowed to equilibrate to room temperature. The minimum amount of sample to be used is 1 mL.

During the analysis of calcium by quadrupole ICP-MS, a significant interference was observed at  $m/z$  44, with smaller effects found on  $m/z$  42 and 43. The interference was caused by the presence of strontium in the material forming a doubly charged ion (i.e.  $^{88}\text{Sr}^{2+}$  has a  $m/z$  of 44). As 42, 43 and 44 are the main calcium isotopes available for ICP-MS analysis, users should be aware that the presence of strontium in LGC6027 may cause a positive bias for the measurement of calcium by quadrupole ICP-MS, unless appropriate measures are taken to significantly minimise such interferences. Those include the use of sector field ICP-MS (at high resolution) or chemical separation prior to ICP-MS analysis.

After use, the bottle should be securely sealed and returned to storage at  $(5 \pm 4) ^\circ\text{C}$ .

## Storage

The material should be stored at  $(5 \pm 4) ^\circ\text{C}$  in its original packaging.

## Shelf Life

If stored under the recommended conditions the certified values will remain valid for 12 months from the date of shipment (see page 6 for shipment date).

## Metrological Traceability

For the analytes characterised through the interlaboratory study, the results are considered traceable to the SI through the physical and chemical standards used by the participant laboratories.

For calcium, iron and lead, the results are traceable to the SI through the primary calibration standards used for the calibration of the IDMS analysis. These were NIST SRM 3109a for calcium, NIST SRM 3126 for iron and NIST SRM 3128 for lead.

## References

[1] Drinking Water Directive. Council Directive 98/83/EC of 3 November 1998 on the quality of water intended for human consumption.

[2] Statutory Instrument 2016 No. 614, Water, England and Wales, The Water Supply (Water Quality) Regulations 2016.

Unit Number

Date of Shipment

### Legal Notice

The values quoted in this certificate are the best estimate of the true values within the stated uncertainties and based on the techniques described herein. No warranty or representation, express or implied, is made that the use of the product or any information, material, apparatus, method or process which is the subject of or referred to in this certificate does not infringe any third party rights. Further, save to the extent: (a) prohibited by law; or (b) caused by a party's negligence; no party shall be liable for the use made of the product, any information, material, apparatus, method or process which is the subject of or referred to in this certificate. In no event shall the liability of any party exceed whichever is the lower of: (i) the value of the product; or (ii) £500,000; and any liability for loss of profit, loss of business or revenue, loss of anticipated savings, depletion of goodwill, any third-party claims or any indirect or consequential loss or damage in connection herewith is expressly excluded.

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