# Certificate of measurement



4005

# Processed meat – Proximates, chloride, hydroxyproline & metals

Certified Reference Material LGC7155

# **Certified values**

Constituent <sup>1</sup>	Number of laboratories	Certified value <sup>2,3</sup> Uncertainty <sup>4</sup> (g/100 g) (g/100 g)		Coverage factor, k
Moisture	18	55.13	0.43	2.03
Nitrogen	18	2.202	0.046	2.02
Total fat	13	24.23	0.59 2	
Ash	17	17 3.229		2.08
Chloride	12	1.377	0.072 2	
Hydroxyproline	10	10 0.359		2.20
	Number of laboratories	Certified value <sup>2,3</sup> (mg/100 g)	Uncertainty <sup>4</sup> (mg/100 g)	Coverage factor, k
Magnesium	9	11.02	11.02 0.73	
Phosphorus	11	236 13		2.20
Potassium	9	187.4	7.4 8.3 2.23	
Sodium	12	1110	63	2.13

#### Notes:

- 1. The values were assigned using the results of an inter-laboratory study.
- 2. Unweighted robust estimate of location, (for magnesium and potassium, the unweighted mean value) of the concentration obtained by participant laboratories. Each data set was obtained in a different laboratory.
- 3. The results are traceable to the SI.
- 4. The uncertainty quoted is the half-width of the expanded uncertainty interval calculated using the coverage factor (k) shown in the table, which gives a level of confidence of approximately 95 %.

Date of Issue: August 2019

Signed: \_\_\_\_\_ Gill Holcombe (Mrs) for the Government Chemist



#### **Indicative Values**

Analyte	Unit	Value <sup>1</sup>		
Calcium	mg/100 g	8		
Iron	mg/100 g	0.6		
Notes:  1. Median of inter-laboratory results. For calcium, occasional high values were obtained in the homogeneity study.				
Analyte	Unit	Range <sup>2</sup>		
Nitrate (as NO <sub>3</sub> ) mg/kg 0.6 to 2		0.6 to 25.1		
Notes:  2. Range of inter-laboratory mean results reported				

#### Calculated value

Constituent	Unit	Calculated value <sup>1</sup>	Expanded uncertainty <sup>2</sup>
Salt (NaCl) 1	g/100 g	2.27	0.12
Notoo:			

#### Notes:

- 1. Calculated from the chloride content.
- 2. The quoted uncertainty is the half-width of the expanded uncertainty interval.

# **Material Preparation**

The material was prepared using a commercial pork-based processed meat. After thorough mincing and mixing, it was sealed in sachets in 50 g portions and irradiated to sterilise using a dose of 18 KGy.

# **Homogeneity Assessment**

The material was tested for between-sachet homogeneity by analysing randomly selected units from across the fill run. The homogeneity data for all analytes was examined and deemed fit-for-purpose, with the exception of calcium. The mass of the material taken for the homogeneity assessment of each analyte is shown in the following table and represents the minimum weight which should be taken for analysis.

Analyte	Moisture	Nitrogen	Total fat	Ash	Chloride	Hydroxyproline	Metals
Mass (g)	5	0.25	3	5	5	3	1

The uncertainty contribution from the homogeneity assessment was incorporated into the combined uncertainty figure.

# **Stability**

The nature of the material is such that deterioration is not anticipated over its lifetime when stored under the recommended conditions. The uncertainty associated with long-term instability was considered to be negligible compared with the uncertainty contributions from characterisation and possible inhomogeneity, and was therefore assigned a value of zero. The material will be monitored at LGC and customers will be notified of any changes in the certified values.

#### Certification

This material has been certified by LGC using the results of an inter-laboratory characterisation study.

Units of the candidate material were distributed to laboratories that had previously agreed to participate in the study. Participant laboratories were given a free choice of methods, but asked to use those with which they were familiar. For the total fat determination, laboratories were asked to use a method which included an acid hydrolysis step.

The data from the inter-laboratory study were processed, after screening laboratories based on their performance in analysing a separate QC sample. Where ten or more acceptable sets of results were received, 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. Where less than ten acceptable sets of results were received, the mean and standard deviation of results were used to calculate the equivalent parameters after review for statistical outliers.

The uncertainty associated with each certified value was calculated by combining the characterisation uncertainty from the inter-laboratory study with the uncertainty calculated from the homogeneity study. This combined uncertainty was then multiplied by a coverage factor (k), calculated separately for each analyte, according to the Welch-Satterthwaite equation, taking into account the effective degrees of freedom.

# Methods used by Participating Laboratories

Techniques used in the characterisation of this material are summarised below:

Constituent	Techniques used
Moisture	Measured as loss on drying using a variety of conditions.
Nitrogen	There were two approaches used by participants. Some used a Kjeldahl method and others used instrumentation based on the Dumas principle.
Total fat	Laboratories used acid hydrolysis followed by solvent extraction (variety of solvents)
Ash	Measured by gravimetry after heating to temperatures between 510 °C and 600 °C.
Chloride	Laboratories employed a variety of methods. Some used a method based on a coulometric titration; others used titration with a colorimetric end point. The preparation procedures included ashing.
Hydroxyproline	Labs generally used spectrophotometric methods following acid hydrolysis.
Metals	Laboratories used a variety of methods including ICP-OES, ICP-MS, AAS following sample preparation, generally by microwave digestion.

### Accreditation

Property values on this certificate are within LGC's scope of accreditation to ISO 17034 as a reference material producer.

#### Intended Use

This material is intended for use in the development, validation or quality control of analytical methods for the determination of major constituents and selected additional analytes in meat and meat products. The material may also be applicable to other matrices and procedures where suitable reference materials are not available.

#### Instructions for Use

Immediately prior to analysing the material, massage the sachet gently to mix the contents, paying particular attention to the corners, and then open carefully. Transfer the entire contents to a suitable mixer or blender and homogenise thoroughly. Store in an airtight jar.

#### **Storage**

The material should be stored at  $(5 \pm 4)$  °C in its original sealed sachet. Once prepared, the material should be stored in a closed container in a fridge at  $(5 \pm 4)$  °C and used within one week.

#### **Shelf Life**

If stored under the recommended conditions, the certified values will remain valid for 12 months from the date of shipment (see page 5 for shipment date).

# **Metrological Traceability**

The certified values are considered to be traceable to the SI through the physical and chemical standards used by the participants in the interlaboratory study.

# Participants in the Inter-laboratory Study

The following laboratories took part in the inter-laboratory study for this material.

Aqualab Ireland
Ashwood UK Ltd UK
Control Foods Poland

Czech Agricultur and Fodd Inspection Authority Czech Republic

Edinburgh Scientific Services UK
Foodtest Laboratories UK

Ing. Josef Nemec, Laborator Pisek Czech Republic

Lancashire County Scientific Services UK

Maxxam Analytics Canada

National Laboratory for Health, Environment and Food (Slovenia - Maribor)

Slovenia

National Laboratory for Health, Environment and Food (Slovenia – Novo Mesto)

Slovenia

Noon Products

UK
Salamon and Seaber Limited

UK
SGS – Ashby

UK

SGS Agri Food Lab – South Africa
State Veterinary and Food Institute Bratislava
VestfolLAB A/S

Worcestershire Scientifc Services

South Africa
Slovakia
Norway

Unit Number	Date of Shipment

# **Legal Notice**

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