Setting standards in analytical science

European Reference Materials

CERTIFICATE OF ANALYSIS

ERM®- BB501a

Processed meat – Proximates, chloride, nitrate and hydroxyproline			
Constituent	Number of laboratories	Certified value [2,3]	Uncertainty [4]
Moisture [1] (g/kg)	18	618	7
Nitrogen [1] (g/kg)	17	23.0	0.7
Total fat [1] (g/kg)	16	151	7
Ash [1] (g/kg)	18	33.2	0.9
Chloride (Cl) (g/kg)	17	14.5	0.5
Hydroxyproline (g/kg)	13	3.3	0.3
Nitrate (NO_3) (g/kg)	9	0.209	0.032

1) The measurand is defined by the methods used by participant laboratories (see page 3).

2) Unweighted median value of the mass fraction 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 the participant laboratories.

4) The expanded uncertainty quoted is the half-width of the 95 % confidence interval. A coverage factor k, (approximately equal to 2 for all analytes) was calculated separately for each analyte according to the Welch-Satterthwaite equation (see ISO Guide to the expression of uncertainty in measurement, 1995 (GUM)).

This certificate is valid for 12 months from the date of shipment provided the sample is stored under the recommended conditions.

The minimum amount of sample to be used for each analyte is given on page 2.

NOTE

European Reference Material ERM®-BB501a was produced and certified under the responsibility of LGC according to the principles laid down in the Technical Guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

Accepted as an ERM®, Teddington, September 2007.

Signed: _____

Dr John Marriott, UK Government Chemist
LGC Limited
Queens Road
Teddington
Middlesex
TW11 0LY, UK

UKAS REFERENCE MATERIALS
4005

Queens Road, Teddington, Middlesex TW11 0LY Tel: +44 (0)20 8943 7000 Fax: +44 (0)20 8943 2767 Web: www.lgc.co.uk

Calculated Values		
Constituent	Value g/kg	Uncertainty g/kg
Salt (NaCl) [1]	23.9	0.7
Sodium nitrate ($NaNO_3$) [2]	0.286	0.043
1) Calculated from the chloride content quoted on page 1. 2) Calculated from the nitrate content quoted on page 1.		

DESCRIPTION OF THE SAMPLE

The material is a pork-based processed meat containing dried pork protein and pea starch. After thorough mincing and mixing, it was sealed in retort pouches in 180 g portions and heat treated for 45 minutes at 122 °C. The material is stored at LGC at 20 °C ± 5 °C.

The material was tested for homogeneity by analysing randomly selected samples from across the fill run. The weight of 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	Nitrate
Weight (g)	5	1	2.5	5	5	5	10

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

INTENDED USE

This material is intended for use in the development, validation or quality control of analytical methods for the determination of the major constituents and selected additives in foods. The methods used to provide the data for assigning the values to the material are summarised later in this certificate. The material may also be applicable to other matrices and procedures where suitable reference materials are not available.

CERTIFICATION

This material has been certified by LGC by means of an inter-laboratory exercise. The certified values are based on the median of laboratory medians following the elimination of results from laboratories failing to meet acceptable QC performance.

The total uncertainty for each analyte was calculated by combining the characterisation uncertainty from the inter-laboratory exercise with the uncertainty calculated from the homogeneity study, and a contribution for possible long-term instability. This combined uncertainty was then multiplied by a coverage factor calculated separately for each analyte according to the Welch-Satterthwaite equation for effective degrees of freedom. This gives an expanded uncertainty with a 95 % confidence factor.

Queens Road, Teddington, Middlesex TW11 0LY Tel: +44 (0)20 8943 7000 Fax: +44 (0)20 8943 2767 Web: www.lgc.co.uk

ANALYTICAL METHODS USED FOR CERTIFICATION

Participating laboratories were free to choose any suitable method with which they were familiar, apart from the measurement of fat content, where an acid hydrolysis step was requested to ensure the measurement of bound as well as free fat. The methods used are summarised below.

Moisture
Measured as loss on drying using a variety of conditions. Procedures included microwave drying, vacuum oven drying and hot air oven drying.

Nitrogen
There were two approaches used by participants. Some used a Kjeldahl method (variety of catalysts) and others used instrumentation based on the Dumas principle.

Total Fat
One laboratory used an NMR procedure on a dried sample. The remaining laboratories used acid hydrolysis followed by solvent extraction (variety of solvents).

Ash
All laboratories used dry ashing in muffle furnace. The end temperature varied between 500 °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 and dissolution, cold or hot water extraction and acid extraction.

Hydroxyproline
Samples were prepared by acid digestion (sulfuric or hydrochloric) followed by determination of hydroxyproline by chloramine 'T' oxidation and colorimetry using 4-dimethylaminobenzaldehyde.

Nitrate
Samples were extracted using hot water or hot borax solution. Nitrate levels were measured using anion exchange HPLC with UV detection, or colorimetrically using sulfanilamide and naphthylethylenediamine hydrochloride, measured at a wavelength of 538 nm.

SAFETY INFORMATION

Refer to material safety data sheet.

INSTRUCTIONS FOR USE

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

STORAGE

The material should be stored at 20 °C ± 5 °C in the original sealed sachet. Once opened, the sample should be treated as fresh meat.

Queens Road, Teddington, Middlesex TW11 0LY Tel: +44 (0)20 8943 7000 Fax: +44 (0)20 8943 2767 Web: www.lgc.co.uk

PARTICIPANTS

The number of participants' results used in the calculation of the certified values is given in the table on page one. The following organisations took part in the interlaboratory exercise:

ALcontrol Laboratories	UK
Birmingham City Laboratories	UK
Central Laboratories	UK
CCFRA Technology Limited	UK
Food Safety & Nutrition Centre - National Institute of Health	Portugal
General Chemical State Laboratory	Greece
Grampian Country Pork Suffolk Ltd	UK
ILS Limited	UK
Laboratoire Intercommunal	Belgium
Lancashire County Council	UK
LGC Limited	UK
Princes Foods Ltd	UK
Public Analysts Laboratory, Dublin	Ireland
RHM Technology	UK
Salamon & Seaber Limited	UK
State Veterinary and Food Institute, Bratislava	Slovakia
Sun Valley Foods Ltd	UK
West Yorkshire Analytical Services	UK
Zavod Za Zdravstveno Varstvo Koper	Slovenia

Queens Road, Teddington, Middlesex TW11 0LY Tel: +44 (0)20 8943 7000 Fax: +44 (0)20 8943 2767 Web: www.lgc.co.uk

Unit Number:	Shipment Date:

LEGAL NOTICE

The certified values quoted in this certificate are LGC's best estimate of the true values within the stated uncertainties and based on the techniques described in this certificate. In support of LGC's aim of providing high quality materials, the reference material was produced taking into account appropriate international guidelines for the preparation and certification of reference materials. None of LGC (Holdings) Limited, its subsidiaries, its contractors nor any person acting on their behalf:

(a) make any warranty or representation, express or implied, that the use of any information, material, apparatus, method or process disclosed in this document does not infringe any privately owned rights; or

(b) assume any liability with respect to, or for damages resulting from, the use of any information, material, apparatus, method or process disclosed in this document save for loss or damage arising solely and directly from the negligence of LGC (Holdings) Limited or any of its subsidiaries. In such circumstances (i.e. our proved negligence) the total amount recoverable shall not exceed £1,000,000.

Production of this reference material and certificate was supported under contract with the Department for Innovation, Universities and Skills as part of the National Measurement System Chemical and Biological Metrology Knowledge Base Programme.

Queens Road, Teddington, Middlesex TW11 0LY Tel: +44 (0)20 8943 7000 Fax: +44 (0)20 8943 2767 Web: www.lgc.co.uk