

JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements

CERTIFICATE OF ANALYSIS

ERM[®] - BB130

PORK MUSCLE		
Measurand in the reconstituted material ¹⁾	Mass fraction	
	Certified value ³⁾ [µg/kg]	Uncertainty ⁴⁾ [µg/kg]
Chloramphenicol ²⁾	0.230	0.021
<p>1) Value is applicable to the material when reconstituted according to the specified procedure (page 3).</p> <p>2) The measurand is defined by using quantification with liquid chromatography-isotope dilution tandem mass spectrometry and gas chromatography-isotope dilution mass spectrometry. Different sample preparation procedures (extraction and clean-up) were applied, but none of those procedures included a β-glucuronidase digestion step.</p> <p>3) The certified value is the unweighted mean of 13 accepted set of results, each set being obtained in a different laboratory, using the calibration substance provided. The values are traceable to the International System of Units (SI).</p> <p>4) Expanded uncertainty with a coverage factor $k = 2$ corresponding to a level of confidence of about 95 % estimated in accordance with ISO Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM), ISO, 2008.</p>		

This certificate is valid for one year after purchase.

Sales date:

The minimum amount of sample to be used is 5 g reconstituted material (prepared using 1.25 g of powder).

Accepted as an ERM[®], Geel, June 2010
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INFORMATION ONLY
Signed:

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Additional Material Information

	Mass Fraction
	Interval [$\mu\text{g}/\text{kg}$] ³
Chloramphenicol ^{1,2)}	0.236 - 0.269

1) Value is applicable to the material when reconstituted according to the specified procedure (page 3).
2) The measurand is defined by using quantification with liquid chromatography-isotope dilution tandem mass spectrometry. Different sample preparation procedures (extraction and clean-up) were applied, but all procedures included a β -glucuronidase digestion step.
3) Mean values obtained in the two laboratories operating methods that included a β -glucuronidase digestion in the sample preparation part.

NOTE

European Reference Material ERM[®]-BB130 was produced and certified under the responsibility of the Institute for Reference Materials and Measurements of the European Commission's Joint Research Centre according to the principles laid down in the technical guidelines of the European Reference Materials[®] co-operation agreement between BAM-IRMM-LGC. Information on these guidelines is available on the internet (<http://www.erm-crm.org>).

DESCRIPTION OF THE SAMPLE

One unit contains 7.5 g of lyophilised pork muscle tissue filled under inert gas in a 100 mL amber glass bottle. The content corresponds to 30 g of fresh muscle tissue derived from animals to which chloramphenicol has been administered (incurred material). The water mass fraction of the lyophilised powder is 20.5 ± 0.7 g/kg.

ANALYTICAL METHOD USED FOR CERTIFICATION

All results were obtained employing liquid chromatography-isotope dilution tandem mass spectrometry and gas chromatography-isotope dilution mass spectrometry methods. Different sample preparation procedures (extraction and clean-up) were applied. None of the methods used for assigning the certified value included a β -glucuronidase digestion step during sample preparation.

PARTICIPANTS

Agence Française de Sécurité Sanitaire des Aliments, Laboratoire d'Etudes et de Recherches sur les Médicaments Vétérinaires et les Désinfectants, Fougères (FR)
(Measurements performed under ISO/IEC 17025 accreditation; COFRAC 1-0247)

Agri-Food and Biosciences Institute, Veterinary Sciences Division, Belfast (GB)
(Measurements performed under ISO/IEC 17025 accreditation; UKAS 2632)

Aveyron Labo, Rodez (FR)
(Measurements performed under ISO/IEC 17025 accreditation; COFRAC 1-1706)

Bundesamt für Verbraucherschutz und Lebensmittelsicherheit (BVL), Berlin (DE)
(Measurements performed under ISO/IEC 17025 accreditation; AKS-PL-12005)

Central Laboratory of Veterinary Control and Ecology (CLVCE), Sofia (BG)
(Measurements performed under ISO/IEC 17025 accreditation; BAS 51)

C.E.R. Groupe, Laboratoire d'Hormonologie, Marloie (BE)
(Measurements performed under ISO/IEC 17025 accreditation; Beltest 103-T)

Chemisches und Veterinäruntersuchungsamt Freiburg, Freiburg (DE)
(Measurements performed under ISO/IEC 17025 accreditation; SAL-BW-L14-03-03)

DTU - National Food Institute, Søborg (DK)
(Sample preparation part performed under ISO/IEC 17025 accreditation; DANAK 350)

- Elintarviketurvallisuusvirasto Livsmedelssäkerhetsverketto (EVIRA), Helsinki (FI)
(Measurements performed under ISO/IEC 17025 accreditation; FINAS T014)
- Eurofins Analytics, Wiertz-Eggert-Jörissen, Hamburg (DE)
(Measurements performed under ISO/IEC 17025 accreditation; DAP-PL-1453.80)
- Institut Scientifique de Santé Publique, Bruxelles (BE)
(Measurements performed under ISO/IEC 17025 accreditation; BELAC 081-TEST)
- LGC Limited, Teddington (GB)
(Measurements performed under ISO/IEC 17025 accreditation; UKAS 0003)
- Norges Veterinærhøgskole, Oslo (NO)
(Measurements performed under ISO/IEC 17025 accreditation; NORSK AKKREDITERING TEST137)
- Österreichische Agentur für Gesundheit und Ernährungssicherheit GmbH, Wien (AT)
(Measurements performed under ISO/IEC 17025 accreditation; Bundesministerium für Wirtschaft und Arbeit, Id 189)
- RIKILT – Institute of Food Safety, Wageningen (NL)
(Measurements performed under ISO/IEC 17025 accreditation; RvA L014)
- Ústav pro státní kontrolu veterinárních biopreparátů a léčiv (ÚSKVBL), Brno (CZ)
(Measurements performed under ISO/IEC 17025 accreditation; Czech Accreditation Institute, 621/2007)

SAFETY INFORMATION

The usual laboratory safety precautions apply.

INSTRUCTIONS FOR USE

This material is intended to be used for method performance control and validation purposes.

For assessing the method performance, the measured value of a CRM is compared with the certified value following a procedure described by Linsinger [Comparison of a measurement result with the certified value, ERM Application Note 1, July 2005, <http://www.erm-crm.org>]. The procedure is described here in brief:

- Calculate the absolute difference between mean measured value and the certified value (Δ_m).
- Combine measurement uncertainty (u_{meas}) with the uncertainty of the certified value (u_{CRM}):

$$u_{\Delta} = \sqrt{u_{meas}^2 + u_{CRM}^2}$$

- Calculate the expanded uncertainty (U_{Δ}) from the combined uncertainty (u_{Δ}) using a coverage factor of two ($k = 2$), corresponding to a confidence interval of approximately 95 %.
- If $\Delta_m \leq U_{\Delta}$ then there is no significant difference between the measurement result and the certified value, at a confidence level of about 95 %.

Reconstitution of the sample

- Allow the bottle to warm up to ambient temperature before opening. Shake bottle vigorously for 30 s to ensure re-homogenisation of the content.
- Weigh accurately an aliquot of 1.25 ± 0.01 g. The weighing should be performed immediately after opening of the vial to minimise water uptake by the lyophilised powder.
- Add an accurately weighed amount of 3.75 ± 0.01 g of distilled water to the powder.
- In case the working instruction of the laboratory's method foresees a higher sample intake than 5 g of reconstituted material, the 1:3 m/m ratio of powder to distilled water has to be maintained.
- Mix to a homogeneous sample, for instance by vortexing the mixture for at least 1 min at maximum speed. Proceed with the sample preparation as foreseen in the laboratory's working instruction without unnecessary delay.

Extraction step in sample preparation procedure

- Please note that after reconstitution, the typical duration of the extraction step (addition of extraction solution, agitation, centrifugation) before further sample manipulation (e.g. evaporation, clean-up) was between 10 and 60 min in the laboratories contributing to the certified value. Any unnecessary delay during extraction (e.g. leaving the sample in the extraction solution at room temperature for a non-controlled time span) shall be avoided.

Dispose in accordance with good laboratory practice.

STORAGE

The material should be stored at a temperature of -20 ± 2 °C.

However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

LEGAL NOTICE

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NOTE

A detailed technical report is available on www.irmm.jrc.be. A paper copy can be obtained from the Joint Research Centre, Institute for Reference Materials and Measurements on request.