JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements

CERTIFICATE OF ANALYSIS

ERM®- BB492

PARTIALLY SKIMMED MILK		
	Mass fraction	
	Certified value [2] [µg/kg]	Uncertainty [3] [µg/kg]
Oxytetracycline (sum of oxytetracycline and 4-epi-oxytetracycline) [1]	101	11

1) Oxytetracycline as measured by liquid chromatography - tandem mass spectrometry and liquid chromatography - diode array detection. Different sample preparation procedures (extraction and clean-up) were applied.

2) The value is applicable to the material when reconstituted according to the specified procedure (page 3). The certified value is the unweighted mean of 10 accepted set of results, each set being obtained in a different laboratory, using the calibration substance provided. The value is traceable to the International System of Units (SI).

3) Expanded uncertainty with a coverage factor k = 2 corresponding to a level of confidence of approximately 95 % estimated in accordance with ISO Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM), ISO, 2008.

This certificate is valid for one year after purchase.

Sales date:

The minimum amount of sample to be used is 1 g reconstituted material.

NOTE

European Reference Material ERM®-BB492 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).

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Signed:

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DESCRIPTION OF THE SAMPLE

One unit contains approximately 5.5 g of spray-dried partially skimmed milk filled under inert gas in a 30 mL amber glass vial. The water mass fraction of the spray-dried powder is 2.50 ± 0.08 g/100 g.

ANALYTICAL METHOD USED FOR CERTIFICATION

All results were obtained employing liquid chromatography - tandem mass spectrometry and liquid chromatography - diode array detection methods. Different sample preparation procedures (extraction and clean-up) were applied.

PARTICIPANTS

Agence Nationale de Sécurité Sanitaire Agence Nationale de Médicament Vétérinaire, Fougères, FR
(Measurements performed under ISO/IEC 17025 accreditation; COFRAC 1-0247)

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(Measurements performed under ISO/IEC 17025 accreditation; UKAS 1642)

Universiteit Gent, Vakgroup Farmacologie, Toxicologie en Biochemie, Gent, BE
(Measurements in conformity with GLP according to Directive 2004/9/EC)

SAFETY INFORMATION

The usual laboratory safety precautions apply.

INSTRUCTIONS FOR USE AND INTENDED USE

Reconstitution of the sample

- Allow the bottle to warm up to ambient temperature; shake vigorously for at least 30 s before opening.
- Weigh accurately an aliquot of 1.00 ± 0.01 g. The weighing should be performed immediately after opening of the vial to minimise water uptake by the powder.
- Add an accurately weighed amount of 8.15 ± 0.01 g of distilled water to the powder.
- The sample has to be homogenised by adding a magnetic stirring bar to the powder/water mixture, and stirring for 10 - 15 min at room temperature.
- In case the working instruction of the laboratory's method foresees a higher/lower sample intake than 9.15 g of reconstituted material, the 1:8.15 m/m ratio of powder to distilled water has to be maintained. It shall be noted that most methods have smaller sample intakes per analysis, and that the sample intake usually is an aliquot of the amount of reconstituted milk.

The certified value is not related to dry mass. Laboratories may wish to verify whether the dry mass content of the material is above 95 m/m % by applying procedure ISO 5537:2004/IDF 26:2004. In any case, water uptake shall be avoided while handing the material (see below).

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

For assessing the method performance, the measured value of a CRM is compared with the certified value following a procedure described in: 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 %.

Dispose in accordance with good laboratory practice.

STORAGE

The material should be stored at a temperature of - 20 ± 5 °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.

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