CERTIFICATE OF ANALYSIS

ERM®- BD213a

Yeast – Total chromium and Cr(III)		
Constituent	Certified value[1,2] (mg/kg)	Uncertainty[3] (mg/kg)
Total chromium	305.5	5.0

1. The certified value is the mass fraction of total chromium determined using double Isotope Dilution Inductively Coupled Plasma Mass Spectrometry (ID-ICP-MS). The value has not been corrected for moisture content.
2. The certified value is traceable to the SI through the use of NIST SRM 3112a.
3. The quoted uncertainty is the half-width of the expanded uncertainty interval calculated using a coverage factor (*k*) of 2, which gives a level of confidence of approximately 95 %.

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

The minimum amount of sample to be used for total chromium analysis is 0.2 g.

NOTE

European Reference Material ERM®-BD213a 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-JRC-LGC. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

Accepted as an ERM®, Teddington, June 2017.

Signed: _____

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Indicative Value[2]		
Constituent	Mass fraction[3] (mg/kg)	Uncertainty[4] (mg/kg)
Cr(III)[1]	302	47

1. Not within LGC's scope of accreditation to ISO Guide 34.
2. The indicative value is the mass fraction of Cr(III) determined using double spike species specific ID-ICP-MS. The value has not been corrected for moisture content. This value is classified as indicative because a full uncertainty budget was not developed for the characterisation method. An indicative value can be used to monitor method performance but not to check bias.
3. The indicative value is traceable to the SI through the use of NIST SRM 3112a and NIST SRM136f (see Traceability).
4. The quoted uncertainty is the half-width of the expanded uncertainty interval calculated using a coverage factor (*k*) of 4.3, which gives a level of confidence of approximately 95 %. The uncertainty value includes an allowance associated with the homogeneity of the material and a component derived from the data obtained during characterisation.

The minimum amount of sample to be used for Cr(III) analysis is 0.1 g.

DESCRIPTION OF THE MATERIAL

The powder form of a commercially-available food supplement of chromium-enriched yeast was donated by PharmaNord ApS (Vejle, Denmark) for the production of ERM®-BD213a. The yeast is in the form of a pale yellow fine powder. The bulk was dispensed as 7 g units in 15 mL amber glass bottles sealed by means of tamper-proof polycone-lined polyethylene screw caps. The material was stored at (-20 ± 10) °C after bottling.

The water content of the material was measured as loss on drying at (102 ± 1) ºC until constant mass was achieved and found to be approximately 5 % m/m (note that this value is provided for information only).

INTENDED USE

The primary intended use of this reference material is for the validation and performance monitoring of new and existing methods for the quantification of total chromium in yeast. The material can also be used for the performance monitoring of procedures for the quantification of chromium species in yeast. The material may also be applicable to other similar matrices where suitable reference materials are not available.

Information on how to compare an analytical result with the certified value can be found in ERM Application Note 1; www.erm-crm.org.

ACCREDITATION

Property values on this certificate are within LGC's scope of accreditation to ISO Guide 34 unless otherwise indicated in the tables of values.

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ANALYTICAL METHODS USED FOR CERTIFICATION

The mass fraction of total chromium was determined at LGC using double ID-ICP-MS following microwave digestion with HNO_3 and H_2O_2. The method is accredited to ISO/IEC 17025. The analyses were carried out using an Agilent 7700 ICP-MS (Agilent Technologies, Cheshire, UK) in helium cell gas mode. The calibration standard used was SRM 3112a from NIST (Gaithersburg, MD, USA). The spike solution contained enriched ^{53}Cr (Oak Ridge National Laboratory, USA). The certified value was confirmed with measurements carried out by the National Measurement Institute of China – NIM (Beijing, China) following a method also based on double isotope dilution calibration. The mass fraction of Cr(III) was determined at LGC using double spike species specific ID-ICP-MS with alkaline hydrolysis. The method is LGC's implementation of a published method (1) with calibration carried out using ^{53}Cr-enriched Cr(VI) and ^{50}Cr-enriched Cr(III). The calibration standards were produced from commercial sources and characterised in-house using SRM 136f and SRM 3112a, with SRM 979 (NIST, Gaithersburg, MD, USA) being used to correct for instrumental mass bias. The analyses were carried out using an Agilent 8800 HPLC-ICP-QQQ (Agilent Technologies, Cheshire, UK) operated in ammonia cell gas mode. The indicative value quoted was confirmed with measurements carried out by NIM using an independent implementation of the same measurement technique.

HOMOGENEITY

An experimental assessment of the homogeneity was carried out on twelve randomly selected units analysed in duplicate using the characterisation method described above. The level of homogeneity of the material in terms of its total chromium and Cr(III) mass fractions was considered satisfactory on sample intakes of 0.2 g and 0.1 g, respectively.

STABILITY

An experimental stability assessment was conducted over a period of 6 months observing the overall bioburden level (bacteria and fungi) of the material. No significant increase in bioburden was observed with time at either -20 °C or 4 °C.

Given its chemical stability, the mass fraction of total chromium is not expected to change with time. Equally, the mass fraction of Cr(III) is not expected to change in the type of reducing environment provided by the yeast. As such, no experimental stability assessment was carried out for either total chromium or Cr(III). The material will, however, be subject to periodical re-testing at LGC and, in the unlikely event of changes being observed, these will be communicated to users.

Additionally, the effect of one freeze/thaw cycle on the mass fraction of Cr(III) was found not to be significant.

MEASUREMENT UNCERTAINTY

The magnitude of each of the components that contribute to the combined uncertainty associated with the quoted values is given in the table below:

	Standard Uncertainty (mg/kg)	
	Total chromium	Cr (III)
Characterisation	1.542	10.39
Homogeneity	1.938	2.69

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TRACEABILITY

The certified value (total chromium) is traceable to the SI through the use of NIST SRM 3112a.

The indicative value Cr(III) is traceable to the SI through the use of NIST SRM 3112a and NIST SRM136f.

NIST SRM 3112a was used to confirm the total chromium content of the isotopically enriched $^{50}Cr(III)$ standard produced from ^{50}Cr enriched Cr_2O_3 (CK Gas). Chromium species distribution in this isotopically-enriched standard was checked using HPLC-ICP-MS (to verify that the total chromium corresponds with that of the peak of Cr(III)). NIST SRM 136f was used to determine the Cr(VI) content of the isotopically-enriched $^{53}Cr(VI)$ standard produced from ^{53}Cr enriched Cr_2O_3 (CK Gas). Note also that NIST SRM 979 was used to correct for instrumental mass bias.

COMMUTABILITY

A commutability study has not been carried out on ERM®-BD213a. Laboratories using this reference material to validate methods that do not rely on total digestion or hydrolysis of the test samples, or that are known to have a commutability issue, should check its commutability against the characterisation method.

SAFETY INFORMATION

Refer to material safety data sheet.

INSTRUCTIONS FOR USE

Prior to use, the unit should be allowed to equilibrate to ambient temperature (20 ± 5) °C. Attention is drawn to the fact that the values quoted are not corrected for moisture content.

STORAGE

Unopened units of the material should be stored at (-20 ± 10) °C.

Previous experience with this type of material at LGC suggests that storage of opened units at (5 ± 4) °C should not significantly affect the total chromium and Cr(III) content; however, a formal experimental study has not been conducted on opened units. Regarding changes in the overall bioburden level (bacteria and fungi) at this temperature, the study carried out over a period of 6 months (see Stability) was on unopened units and cannot be extrapolated to open ones. However, changes to the bioburden levels are not expected at this temperature unless water is added to the sample.

Note that the effect of one freeze/thaw cycle did not significantly change the mass fraction of Cr(III).

LGC cannot take responsibility for changes to the quoted values if a unit is opened and then stored for later use, or if a unit is frozen and thawed repeatedly.

REFERENCES

(1) Calculations of double spike isotope dilution results revisited. Juris Meija, Lu Yang, Joseph A. Caruso, Zoltan Mester: Journal of Analytical Atomic Spectrometry, 2006, Vol. 21, pp. 1294-1297.

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Unit Number:	Shipment Date:

LEGAL NOTICE

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