



CERTIFIED REFERENCE MATERIAL BCR[®] – 633

CERTIFICATE OF ANALYSIS

ANHYDROUS BUTTER FAT		
	Mass fraction	
	Certified value ¹⁾	Uncertainty ²⁾
<i>β</i> -Apo-8'-carotenic acid ethyl ester	26.5 mg/kg	1.4 mg/kg
<i>β</i> -Sitosterol	530 mg/kg	29 mg/kg
Stigmasterol	147 mg/kg	11 mg/kg
<i>n</i> -Heptanoic acid triglyceride	10.6 g/kg	0.4 g/kg

1) Unweighted mean value of the means of accepted sets of results, each set being obtained in a different laboratory and according to reference methodology (see Annex in the certification report). The certified values are traceable to the methodology applied.

2) Estimated expanded uncertainty with a coverage factor $k = 2$, corresponding to a level of confidence of about 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1993.

This certificate is valid for one year after purchase.

Sales date:

The minimum amount of sample to be used is defined by the reference method employed for each analyte.

NOTE

This material has been certified by BCR (Community Bureau of Reference, the former reference materials programme of the European Commission). The certificate has been revised under the responsibility of IRMM.

Brussels, October 2002

Latest revision: September 2013

Signed: _____

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Indicative Values	
	Mass fraction
	Indicative value ¹⁾ [mg/kg]
Vanillin	226
1) Unweighted mean value of the means of 7 accepted sets of results.	

DESCRIPTION OF THE SAMPLE

BCR-633 is supplied in units of about 5 g each in amber glass ampoules, which were filled under inert gas conditions (nitrogen).

ANALYTICAL METHOD USED FOR CERTIFICATION

The tracers foreseen in the regulations and incorporated in this reference material are compounds of different origin and exhibit different chemical structures and properties. Thus, several different methods have to be applied to determine their concentrations: the β -ester, which shows a strong absorbance at 449 nm, appears in an insoluble form and has an orange colour; therefore a photometric method is performed, using the organic solvent petrol ether to dissolve the standards and the butter oil sample.

For vanillin, a chromatographic method exists with an extraction procedure as the sample preparation step. Separation with subsequent detection is accomplished by reversed-phase HPLC and UV at 306 nm. For the sterols, a GC-FID (GC-MS) analysis is the method of choice, using a saponification of the non-sterol compounds with KOH in ethanolic solution, extraction of the unsaponifiables (sterols) with diethyl ether, and finally derivatisation (trimethylsilylation) of the analytes prior to GC separation. The detection is usually accomplished using a FID.

For 4 of the 5 tracers certified here, EU reference methods are existing, which define in detail the procedure to be followed. However, for C₇-TG, no such reference method exists yet. So far, both fatty acid methyl ester (FAME) analysis by GC as well as a direct determination of C₇-TG, using GC/FID (GC-MS) are most widely used.

PARTICIPANTS

- **Preparation of material**
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- **Organisation, statistical analysis and preparation of the report**
European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Geel (BE)

SAFETY INFORMATION

The usual laboratory safety precautions apply.

INSTRUCTIONS FOR USE

Before opening an ampoule and taking out a portion for analysis, it must be guaranteed that the content is a liquid and properly mixed to ensure homogeneity. The following procedure is recommended:

- 1) Immerse the ampoule in a water bath at a temperature not exceeding 50 °C, with occasional agitation until the fat is completely melted.
- 2) When a clear bright oil is obtained, mix the contents by repeated inversion for at least 30 s.
- 3) Before significant cooling can occur, score the neck of the ampoule at the thinnest part of its head and remove the head manually or by application of a respective device.
- 4) Immediately transfer the contents of the ampoule in a clean and dry vial, which can be tightly sealed.

The reference material should be used on the day of opening. The contents of opened ampoules should not be stored for future use. The reference methods define the amount of sample to be taken for the individual parameters.

Care has been taken to ensure that the certified value represents the "true" value. 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.

Dispose in accordance with good laboratory practice.

STORAGE

The samples must be stored unopened - 30 °C until use. The material is solid at ambient temperature.

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NOTE

A technical report on the production of BCR-633 is available on the internet (<http://www.irmm.jrc.be>). A paper copy can be obtained from IRMM on request.