



Czech Metrology Institute
Dept. Of Reference Materials Certification
Radiová 3
102 00 Prague 10

CERTIFICATE

No. 1014-CM-7007-09

CERTIFIED REFERENCE MATERIAL CZ 7007

PAHs and some pesticides in extra virgin olive oil

Use:

The material is intended for use in verification of analytical procedures and validation of analytical methods and for ensuring traceability. It is not intended for use as a calibrant.

Description of the material:

The sample consists of two 75ml amber glass bottles with a PTFE insert and screw cap packed into a small paper box. Each bottle contains 50 ± 0.5 ml of a virgin olive oil. The bottle designated OO-LL contains the original olive oil (blank), the bottle designated OO-HL contains spiked oil, which is the subject of certification. Both materials are stabilized with 0.05% BHT.

Leader Subcontractor: ANALYTIKA[®] spol. s r.o., Ke Klíčovu 2a/816, 190 00 Prague 9, Czech Republic

Project manager: Doc. RNDr. Václav Sychra, CSc.

Certifying body responsible person: Ing. Jan Beránek

Manufacture, testing and certification of this material were carried out in compliance with the Czech Metrology Institute Methodical procedure No. 017/MP/C001 and ISO/REMCO Guide 34.

Date of issue: 1.3.2009

Valid until: 1.3.2013

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Ing. Vladimír Peršl,
Director of CMI OI Prague

**Certified values and their uncertainties
and non-certified values of contents of analytes in CZ7007**

Analyte	Mass fraction		Number of accepted sets of data
	Mean of the lab. means [$\mu\text{g}/\text{kg}$] ¹	Uncertainty [$\mu\text{g}/\text{kg}$] ^{2,3}	
Anthracene	3.72	0.26	8
Benzo[a]anthracene	4.78	0.38	7
Benzo[b]fluoranthene	3.18	0.30	7
Benzo[k]fluoranthene	2.22	0.19	7
Benzo[ghi]perylene	2.19	0.18	8
Benzo[a]pyrene	2.47	0.18	9
Chrysene	6.21	0.43	8
Dibenzo[a,h]anthracene	0.50	0.06	8
Fluoranthene	15.7	1.2	8
Indeno[1,2,3-cd]pyrene	2.44	0.18	8
Phenanthrene	42.3	3.8	8
Pyrene	14.6	1.2	7
Chlorpyriphos–methyl	182	13	8
Endosulphan sulphate	186	13	7
Non-certified analytes			
Benzo[j]fluoranthene	1.57	(0.15)	4
Benzo[c]fluorene	1.68	(0.31)	5
Cyclopenta[cd]pyrene	3.16	(0.38)	5
Dibenzo[a,e]pyrene	2.31	(0.34)	5
Dibenzo[a,h]pyrene	2.83	(0.38)	6
Dibenzo[a,i]pyrene	2.67	(0.28)	6
Dibenzo[a,l]pyrene	2.80	(0.35)	6
5–methylchrysene	1.53	(0.15)	4

- 1) This value is the unweighed mean of the means of accepted sets of results
- 2) Expanded combined uncertainty (k=2)
- 3) The uncertainty of non-certified values are presented in parentheses

Instructions for use:

For analysis the sample should be taken as it is (after tempering to 20°C). The material in the bottle should be rehomogenized before use by manual or mechanical shaking for 1-2 minutes. The bottle should be closed again as soon as possible. Before closing, it is recommended to refill the bottle (the space over the oil) with an inert gas (preferably with argon). The recommended sample intake is 0.5-3g (depending on the analytical method used). The material has to be stored in dark at 5-8°C (in the fridge). At low temperature precipitation of a solid phase can occur. However, the precipitate dissolves completely when the sample is tempered to 20°C. This fact does not affect the quality of analytical data.

Characterization:

Characterization was performed in accordance with ISO REMCO Guide 35. Results based on homogeneity testing (within-bottle and between-bottle) and on isochronous stability tests (performed 6 and 18 months after the production of the material) have shown that the contribution of inhomogeneity as well as of non-stability to the total uncertainty of

the certified values is insignificant (for all analytes tested). The material is stable in the temperature range of (-20;+25)°C. The certified and non-certified values of analytes were derived from results of an interlaboratory comparison in which 11 selected laboratories took part. Analytical methods used covered an extraction (or dilution) with one or with a mixture of solvents and a clean-up in one or several steps. Separation and quantification was carried out by GC-MS or HPLC. (Refer to Certification Report for details).

Metrological traceability:

For calibration, the participating laboratories use two sets of standards: own calibrants and mixed calibration solutions prepared gravimetrically (from the best defined primary analyte compounds available at the market) and delivered by the producer.

For some analytes, the traceability was realized by using certified reference standards solution SRM 1647a (NIST, USA) and matrix CRM BCR 458 and 459.

Participating laboratories:

Material preparation: ANALYTIKA[®] spol. s r.o. Prague, Czech Republic

Homogeneity and stability study: Metrological and Testing Laboratory, Institute of Chemical Technology, Prague, Czech Republic

Certification analyses:

- Central Science Laboratory, York, United Kingdom
- Eurofins Analytik, WEJ Contaminants, Hamburg, Germany
- Eurofins Dr. Specht Laboratorien GmbH, Hamburg, Germany
- Münster Analytical Solutions (MAS) GmbH, Münster, Germany
- State Veterinary and Food Institute, Bratislava (Slovakia)
- Ekocentrum Ostrava, Ostrava, Czech Republic
- State Veterinary Institute, Department of Food Testing, Prague, Czech Republic
- Czech Agricultural and Food Inspection, Prague, Czech Republic
- Metrological and Testing Laboratory, Institute of Chemical Technology, Prague, Czech Republic
- Department of Chemistry and Analysis of Food, Institute of Chemical Technology, Prague, Czech Republic
- Institute of Health Ostrava, Department of laboratories of Occupational Hygiene, Frýdek-Místek, Czech Republic

Note: A detailed Certification Report on the sample preparation, the analysis procedures and the treatment of the analytical data is available on Internet (www.analytika.net). A paper copy can be obtained from the producer on explicit request.

CMI responsible person: Ing. Jan Beránek