



# Czech Metrology Institute

Certifying Body for Reference Materials

Radiová 3

102 00 Praha 10

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## CERTIFICATE

No. 0217-CM-7006-06

### **POLYCHLORODIBENZO-p-DIOXINS, POLYCHLORODIBENZO FURANS AND TOXIC POLYCHLORO BIPHENYLS IN SEWAGE SLUDGE OF MIXED ORIGIN**

**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  $60 \pm 0.5$  g of dried sewage sludge powder (a particle size of less than 100  $\mu\text{m}$ ) in amber glass bottles with a polypropylene insert and screw cap. The total I-TEQ value is approximately 150 ng/kg. The material is radiationally sterilised by  $^{60}\text{Co}$  at a dose of 25 kGy.

**Leader Subcontractor:** ANALYTIKA<sup>®</sup> spol. s r.o., Ke Klíčovu 2a/816, 190 00 Prague 9  
Czech Republic

**Project leader:** Dr. Václav Sychra, Ph. D.

**Certifying body responsible person:** Ing. Jan Tichý

Production, testing and certification of this material followed strictly ISO REMCO Guide 34.

Date of issue: 20.12.2006

Expiry date: 20.12.2011

Date of certification: 7.12.2006

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Dr. Jiří Tesař, PhD.

Deputy director of CMI

## Certified values and their uncertainties

Analyte	Mass fraction		Number of accepted sets of data
	Certified value (ng/kg) <sup>1)3)</sup>	Uncertainty (ng/kg) <sup>2)</sup>	
2,3,7,8 TeCDD (D48)	4.5	0.3	7
1,2,3,7,8 PeCDD (D54)	2.1	0.3	7
1,2,3,4,7,8 HxCDD (D66)	2.6	0.5	7
1,2,3,6,7,8 HxCDD (D67)	5.0	0.9	9
1,2,3,7,8,9 HxCDD (D70)	3.7	0.6	9
1,2,3,4,6,7,8 HpCDD (D73)	65	10	7
OCDD (D75)	519	74	7
2,3,7,8 TeCDF (F83)	110	17	8
1,2,3,7,8 PeCDF (F94)	157	21	8
2,3,4,7,8 PeCDF (F114)	87	11	6
1,2,3,4,7,8 HxCDF (F118)	376	63	9
1,2,3,6,7,8 HxCDF (F121)	102	13	8
1,2,3,7,8,9 HxCDF (F124)	11.0	2.2	7
2,3,4,6,7,8 HxCDF (F130)	19.8	2.8	6
1,2,3,4,6,7,8 HpCDF (F131)	256	41	9
1,2,3,4,7,8,9 HpCDF (F134)	110	17	8
OCDF (F135)	1590	290	9
PCB 77	2380	370	10
PCB 81	108	16	6
PCB 126	169	32	9
PCB 169	25	4	8
PCB 105	3430	495	10
PCB 114	169	36	6
PCB 118	15800	2300	10
PCB 123	121	30	6
PCB 156	9140	1300	8
PCB 157	802	130	10
PCB 167	4130	670	10
PCB 189	1860	260	8

- 1) This value is the unweighed mean of the means of accepted sets of results
- 2) Uncertainties are combined uncertainties multiplied by a coverage factor k=2
- 3) Corrected for dry mass at 105<sup>0</sup>C

## Non-certified values of contents of some other analytes present in CZ 7006\*

Metals		Polycyclic AromaticHydrocarbons (PAHs)	
Analyte	Mass fraction <sup>a)</sup> mg/kg <sup>c)</sup>	Analyte	Mass fraction <sup>a)</sup> mg/kg
Ag	6.75	acenaphtene	0.53
As	17.3	anthracene	0.20
Be	0.82	benzo(a)anthracene	0.61
Cd	1.47	benzo(b)fluoranthene	0.33
Co	16.8	benzo(k)fluoranthene	0.21
Cr	498	benzo(g,h,i)perylene	0.21
Cu	318	benzo(a)pyrene	0.23
Hg	1.48	chrysene	0.53
Mn	1410	dibenzo(a,h)anthracene	0.04
Mo	4.15	fluoranthene	3.84
Ni	41.2	fluorene	0.51
Pb	174	indeno(1,2,3-c,d)pyrene	0.23
Se	1.41	naphtalene	1.32
Sn	15.2	phenanthrene	1.92
Tl	0.17	pyrene	2.27
V	39.5	<b>Some other organic analytes</b>	
Zn	782	Analyte	Mass fraction <sup>b)</sup> , µg/kg
Al	(19800) <sup>d)</sup>	Σ HCH,α,β,γ,δ	(52) <sup>d)</sup>
Ca	(98200)	2,4' - DDD	(670)
Fe	(21500)	4,4' - DDD	(1650)
K	(5650)	2,4' - DDT	(190)
Mg	(4940)	4,4' - DDT	(850)
Na	(5470)	1,2,3 – trichlorobenzene	(280)
		pentachlorobenzene	(200)
		hexachlorobenzene	(6000)

Other PCBs ("markers")		Brominated Flame Retardants (BFRs)	
Analyte	Mass fraction <sup>a)</sup> , µg/kg	Analyte	Mass fraction <sup>a)</sup> , µg/kg
PCB - 28	26.3	PBDE - 28	0.77
PCB - 52	14.6	PBDE - 47	62.2
PCB - 101	49.3	PBDE - 49	2.23
PCB - 138	115	PBDE - 66	1.33
PCB - 153	153	PBDE - 85	3.50
PCB - 170	64.8	PBDE - 99	59.8
PCB - 180	123	PBDE - 100	14.0
		PBDE - 153	4.05
		PBDE - 154	4.60
		PBDE - 183	6.25
		PBDE - 209	55.6
		HBCD	79.2

\*Analyte contents were derived (unless otherwise stated) from a minimum of 3 data sets

a) Corrected for dry mass at 105<sup>o</sup>C

b) Corrected for dry mass at 40<sup>o</sup>C

c) Aqua regia extraction according to ISO-11885

d) Informative analyses only from 2 labs

**Instructions for use:**

For analysis the sample should be taken as it is. Its dry mass should be determined on non-analysed portions oven dried at 105<sup>0</sup>C till the constant mass. The water content is approximately 6% by mass. The recommended sample intake is 5g. The material in the bottle should be rehomogenized before use by manual or mechanical shaking for 1-2 minutes. The bottle should be open several minutes after shaking to avoid possible emission of fine particles of the sample from the bottle and their sedimentation in the surroundings. Storage of the material at room temperature (15-30°C) in the dark is recommended. Since the material is toxic, handling with it should be allowed only to staff trained for working with toxic materials.

**Characterization:**

Characterization was performed in accordance with ISO REMCO Guide 35. 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 with one or a mixture of solvents and a clean-up in one or several steps. Separation and quantification was carried out by GC-HRMS using two chromatographic phases of different polarity.

**Metrological traceability:**

BCR 614 (S<sub>0</sub>-S<sub>5</sub>), BCR 365 and ERM AC 820-822 were used for calibration by most of participating laboratories. Each laboratory analysed simultaneously with the samples also the matrix reference material BCR – 677.

**Participating laboratories****Material preparation:**

ANALYTIKA<sup>®</sup> spol. s r.o. Prague, Czech Republic

Sub-suppliers:

Ecolab s.r.o., Radlík, Czech Republic (sourcing, preliminary analyses, packing)

Institute of Nuclear Physics, Řež u Prahy (radiational sterilization)

Safina a.s. Vestec u Prahy, Czech Republic (homogenization)

ÚNS a.s. Kutná Hora, Czech Republic (milling, sieving)

**Homogeneity and stability study:**

Axys-Varilab s.r.o., Vrané nad Vltavou – Skochovice, Czech Republic

**Certification analyses:**

- ALS Czech Republic s.r.o. (former Ecochem a.s.), Prague, Czech Republic
- Axys Analytical Services Ltd., Sidney, Canada
- Axys-Varilab s.r.o., Vrané nad Vltavou – Skochovice, Czech Republic
- CARSO, Centre d'Analyse et de Recherche sur de Substances Organiques, Lyon, France
- Institute of Chemical Technology, Metrological Laboratory, Prague, Czech Republic
- Institute of Health, Ostrava, laboratory Frýdek-Místek, Czech Republic
- MAS, Münster Analytical Solutions GmbH, Münster, Germany
- Nablabs Laboratories, Espoo, Finland
- SAL, Scientific Analysis Laboratories Ltd., Manchester, United Kingdom
- VITO, Vlaamse Instelling voor Technologisch Onderzoek, Mol, Belgium
- Water Research Institute T. G. M., Prague, 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](http://www.analytika.net)). A paper copy can be obtained from the producer on explicit request.