

National Institute for Environmental Studies

Certificate of Analysis

NIES CRM No. 24 Fly Ash II

This environmental certified reference material (CRM) was prepared by the National Institute for Environmental Studies (NIES) with the cooperation of the Japan Environmental Sanitation Center (JESC) for use in calibrating and managing the precision of the analytical equipment used for the chemical analysis of polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and dioxin-like PCBs (DL-PCBs : IUPAC No. 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, 189) in incinerator ash. This material was used in the environmental analytical quality control program conducted by the Ministry of Environment of Japan in FY 2001.

Preparation of the CRM

Fly ash was collected from a general waste incineration plant with stoker-type incinerators in Japan. Fifteen kilograms of fly ash were sieved using a 100-mesh (150 μ m) sieve and then homogenized. Ten kg of the homogenized material were packed into 666 pre-washed amber glass bottles in December 2001, each bottle containing 15 g. The CRM production process was conducted in accordance with ISO Guide 34.

Homogeneity

Six bottles were randomly selected from the entire batch and analyzed for PCDDs, PCDFs and DL-PCBs. No significant variations in homogeneity between bottles and within bottles were observed. It was adjudged that the material was sufficiently homogenous for its intended purpose.

Certified and Reference Values

Of the measured values reported by 18 organizations, those with z-scores of 2 or more, obtained by use of robust statistics, were rejected. Certified values for PCDDs and PCDFs were decided in accordance with ISO Guide 35. The uncertainty attached to the certified values is the expanded uncertainty using a coverage factor $k = 2$, corresponding to the half-width of a confidence interval of approximately 95 % (Table 1). Reference values only are given for DL-PCBs because of the large analytical variation (Table 2). All certified and reference values are reported on a dry mass basis. Chemical analysis of this material was conducted according to Attached Table 1 of the Ministry of Health, Labour and Welfare of Japan Notification No. 192, and the Ministry of the Environment of Japan Notification No. 80.

Precautions for Storage and Handling for Analysis

1. This CRM should be stored in a sealed state in a cool dark place.
2. Prior to weighing aliquots for analysis, the bottle should be allowed to stand at room temperature, and the contents of the bottle should be shaken gently.

3. The certified and reference values for this material are based on dry mass. For correction of the measured values to dry mass, moisture content measurements should be made on a separate portion of the material taken from the same bottle. The material should be dried for 2 hours at 105 °C before weighing. The moisture content measured by NIES was approximately 5 %. This value, however, varies depending on the storage environment. The moisture content should be measured using the recommended method each time analysis is undertaken, and the analytical value should be corrected to dry mass.
4. It is recommended that a sample of approximately 1 g is used for analysis.
5. Do not use equipment made from organic materials such as plastics for weighing this CRM.
6. Precautions should be taken to avoid inhalation of the material.
7. Stability tests demonstrated that the analytical values for PCDDs and PCDFs in this CRM remained within the certified limits for a period of at least five years.

Expiry Date of Certification

The expiry date for the certified values of this CRM is March 2032, assuming that above mentioned storage conditions are adhered to. NIES will announce via its website if any changes in the contents are noticed within the term of validity.

Collaborating Laboratories in Analysis

The certified and reference values for this CRM were based on the analytical values from the following participating organizations:

National Institute for Environmental Studies; Center for Environmental Science in Saitama; Chiba Prefectural Environmental Research Center; Environmental Control Center Co., Ltd., Analysis Center; Environmental Pollution Control Center, Osaka Prefectural Government; Environmental Research Center Co., Ltd.; Gifu Prefectural Research Institute for Health and Environmental Sciences; Hokkaido Institute of Environmental Sciences; Japan Food Research Laboratories; Japan Quality Assurance Organization, Kansai Testing Center; METOCEAN Environment Inc., Institute of Environmental Ecology; Miyagi Prefectural Institute of Public Health and Environment; Nagano Research Institute for Health and Pollution; Nagasaki Prefectural Institute of Public Health and Environmental Sciences; Oita Prefectural Institute of Health and Environment; Shimadzu Techno-Research, Inc.; Sumika Chemical Analysis Service, Ltd., Ehime Branch; Tokyo Metropolitan Research Institute for Environmental Protection

Organization Cooperating in the Preparation of this CRM

Japan Environmental Sanitation Center

Technical Information

Technical information and the latest reports regarding this material can be obtained from the website.

<http://www.nies.go.jp/labo/crm-e/index.html>

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Table 1. Certified values for PCDDs and PCDFs

Component name		Mass fraction		Reference	
		Certified value ng/g-dry	Uncertainty ng/g-dry	TEF *	Toxicity equivalence ng-TEQ/g-dry **
PCDD isomer	2,3,7,8-TeCDD	4.2	0.6	1	4.2
	1,2,3,7,8-PeCDD	28	4	1	28
	1,2,3,4,7,8-HxCDD	39	7	0.1	3.9
	1,2,3,6,7,8-HxCDD	38	7	0.1	3.8
	1,2,3,7,8,9-HxCDD	49	8	0.1	4.9
	1,2,3,4,6,7,8-HpCDD	250	60	0.01	2.5
	OCDD	280	70	0.0003	0.084
PCDF isomer	2,3,7,8-TeCDF	7.1	1.7	0.1	0.71
	1,2,3,7,8-PeCDF	23	8	0.03	0.69
	2,3,4,7,8-PeCDF	23	5	0.3	6.9
	1,2,3,4,7,8-HxCDF	43	8	0.1	4.3
	1,2,3,6,7,8-HxCDF	45	8	0.1	4.5
	1,2,3,7,8,9-HxCDF	2.9	1.2	0.1	0.29
	2,3,4,6,7,8-HxCDF***	38	9	0.1	3.8
	1,2,3,4,6,7,8-HpCDF	160	50	0.01	1.6
	1,2,3,4,7,8,9-HpCDF	18	7	0.01	0.18
	OCDF	63	21	0.0003	0.019
PCDD congener	TeCDDs	55	8		4.2
	PeCDDs	170	20		28
	HxCDDs	390	60		12.6
	HpCDDs	420	80		2.5
	OCDD	280	70		0.084
	Total PCDDs	1300	200		47
PCDF congener	TeCDFs	220	30		0.71
	PeCDFs	330	50		7.59
	HxCDFs	400	50		12.89
	HpCDFs	260	80		1.78
	OCDF	63	21		0.019
	Total PCDFs	1300	200		23
Total (PCDDs + PCDFs)		2600	400		70

All certified values were determined based on dry mass.

The uncertainty attached to the certified values is the expanded uncertainty using a coverage factor $k = 2$, corresponding to the half-width of a confidence interval of approximately 95%.

* TEF, Toxic Equivalency Factor WHO-TEF(2005) TOXICOLOGICAL SCIENCES 93(2), 223-241 (2006)

** TEQ, Toxic Equivalent

- *** 1) The certified value of 2,3,4,6,7,8-HxCDF was calculated by subtracting the 1,2,3,6,8,9-HxCDF value quantified using column RH-12ms from the 2,3,4,6,7,8+1,2,3,6,8,9-HxCDF value quantified using column BPX-DXN. The uncertainty of this certified value was calculated based on the value of 2,3,4,6,7,8-HxCDF after subtraction.
- 2) The average value of 2,3,4,6,7,8+1,2,3,6,8,9-HxCDF was 45.1 ng/g-dry and the standard deviation of this value was 0.7 ng/g-dry, when we measured n=5 during the stability test in 2021.

Table 2. Reference values for DL-PCBs

Component name	IUPAC No.	Mass fraction	Reference		
		Reference value ng/g-dry	TEF *	Toxicity equivalence ng-TEQ/g-dry **	
Non-ortho-type	3,3',4,4'-TeCB	77	3.4	0.0001	0.00034
	3,4,4',5'-TeCB	81	0.45	0.0003	0.00014
	3,3',4,4',5'-PeCB	126	4.4	0.1	0.44
	3,3',4,4',5,5'-HxCB	169	2.2	0.03	0.066
	Total		10		0.51
Mono-ortho-type	2,3,3',4,4'-PeCB	105	2.2	0.00003	0.000066
	2,3,4,4',5'-PeCB	114	0.15	0.00003	0.0000045
	2,3',4,4',5'-PeCB	118	1.7	0.00003	0.000051
	2',3,4,4',5'-PeCB	123	0.45	0.00003	0.000014
	2,3,3',4,4',5'-HxCB	156	1.9	0.00003	0.000057
	2,3,3',4,4',5'-HxCB	157	1.4	0.00003	0.000042
	2,3',4,4',5,5'-HxCB	167	1.2	0.00003	0.000036
	2,3,3',4,4',5,5'-HpCB	189	2.0	0.00003	0.000060
	Total		11		0.00033
Total PCBs			21		0.51

All reference values were determined based on dry mass.

* TEF, Toxic Equivalency Factor

WHO-TEF(2005) TOXICOLOGICAL SCIENCES 93(2), 223-241 (2006)

** TEQ, Toxic Equivalent