

National Institute for Environmental Studies

Certificate of Analysis

Certified Reference Material 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 (IUPAC No. 81, 77, 126, 169, 123, 118, 105, 114, 167, 156, 157, 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 stocker-type incinerators in Japan. Fifteen kilograms of fly ash were sieved using a 100-mesh (150 μm) 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 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 a confidence interval of approximately 95% (Table 1). Reference values only are given for 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.

Expiration Date of Certification

The expiration date for the certified values of this CRM is March 2022, assuming that above mentioned storage conditions are adhered to. NIES will announce via its website (www.nies.go.jp) 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; and Tokyo Metropolitan Research Institute for Environmental Protection.

Organization Cooperating in the Preparation of this CRM

Japan Environmental Sanitation Center

Organization Responsible for Preparation, Certification, and Distribution

The Environmental Analytical Chemistry Research Laboratory within the Laboratory of Intellectual Fundamentals for Environmental Studies, NIES, takes entire responsibility for this CRM. Research reports and technical information regarding this material can be obtained from the following address.

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Table 1. NIES CRM No. 24 Fly Ash II Certified values for PCDDs and PCDFs

| Component name | | Certified value ng/g-dry | Reference | |
|-----------------------|---------------------|-----------------------------|-----------|---|
| | | | 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 | — | — |
| | PeCDDs | 170 ± 20 | — | — |
| | HxCDDs | 390 ± 60 | — | — |
| | HpCDDs | 420 ± 80 | — | — |
| | OCDD | 280 ± 70 | — | — |
| | Total PCDDs | 1300 ± 200 | — | 47 |
| PCDF congener | TeCDFs | 220 ± 30 | — | — |
| | PeCDFs | 330 ± 50 | — | — |
| | HxCDFs | 400 ± 50 | — | — |
| | HpCDFs | 260 ± 80 | — | — |
| | OCDF | 63 ± 21 | — | — |
| | Total PCDFs | 1300 ± 200 | — | 23 |
| Total (PCDDs + PCDFs) | | 2600 ± 400 | — | 70 |

*: WHO-TEF (2005)

Table 2. NIES CRM No. 24 Fly Ash II Reference values for PCBs

| Component name | | IUPAC No. | Reference value ng/g-dry | Reference | |
|-----------------------------|-----------------------|-----------|-----------------------------|-----------|--------------------------------------|
| | | | | TEF* | Toxicity equivalence ng-TEQ/g-dry |
| Non- <i>ortho</i> isomer | 3,4,4',5'-TeCB | 81 | 0.45 | 0.0003 | 0.00014 |
| | 3,3',4,4'-TeCB | 77 | 3.4 | 0.0001 | 0.00034 |
| | 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- <i>ortho</i> isomer | 2',3,4,4',5'-PeCB | 123 | 0.45 | 0.00003 | 0.000014 |
| | 2,3',4,4',5'-PeCB | 118 | 1.7 | 0.00003 | 0.000051 |
| | 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',5,5'-HxCB | 167 | 1.2 | 0.00003 | 0.000036 |
| | 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 |
| | 2,3,3',4,4',5,5'-HpCB | 189 | 2.0 | 0.00003 | 0.000060 |
| | Total | | 11 | — | 0.00033 |
| Total PCBs | | | 21 | — | 0.51 |

*: WHO-TEF (2005)