

National Institute for Minamata Disease, Ministry of the Environment, Japan

# Reference Material Certificate NIMD-01 Human Hair

This reference material (NIMD-01) is a homogenized human hair produced in accordance with ISO Guide 35 (JIS Q 0035). NIMD-01 can be used to validate analytical methods or equipment, or to operate quality management in quantitative analysis of trace elements in hair samples.

# 1. Certified Value and Indicative Value

Certified values of NIMD-01 shown below are traceable to the International System of Units (SI). Expanded uncertainties of certified values are obtained by multiplying standard uncertainties by the coverage factor k = 2.

| Class               | Elements              | Value<br>(mg/kg) | Expanded<br>Uncertainty<br>(mg/kg) | Analytical Method<br>(refer to<br>annotations below) |
|---------------------|-----------------------|------------------|------------------------------------|--|
| Certified<br>Value  | Methyl Mercury (MeHg) | 0.634            | 0.071                              | 1),2),3)   |
|                     | Total Mercury (THg)   | 0.794            | 0.050                              | 4),5),6)   |
|                     | Copper (Cu)           | 12.8             | 1.4                                | 7)   |
|                     | Zinc (Zn)             | 234              | 29                                 | 7)   |
|                     | Selenium (Se)         | 1.52             | 0.29                               | 7)   |
| Indicative<br>Value | Arsenic (As)          | 0.17             | 0.03                               | -  |

Analytical method

1) GC-electron capture detector

2) HPLC-chemiluminescence

3) Ethylation-GC-cold vapor atomic fluorescence

4) Thermal decomposition atomic absorption

5) Cold vapor atomic absorption

6) Cold vapor atomic fluorescence

7) Inductively coupled plasma mass spectrometry

#### 2. Minimum Sampling Volume

Sampling volume for an analysis should be 20 mg or more to ensure homogeneity.

## 3. Characterization and Value Assignment

The certified values were assigned on the basis of valid datasets after evaluation of validity and exclusion of statistical outliers of the original dataset reported from participants of an interlaboratory study. This process was based on instructions and reporting formats that meet the criteria of JIS Q 0035:2008. Uncertainties of the certified values were determined by combining uncertainties obtained from the inter-laboratory study, a homogeneity test, and a stability test.

## 4. Period of Validity

The period of validity for this reference material is one year from the date of shipment, provided that the conditions described in "7. Instruction on Storage" are met. Storage at -20 degrees Celsius is expected to enable longer-term preservation; in such cases, the corresponding expiration date will be posted on the producer's website (http://nimd.env.go.jp/english/).

#### 5. Product Form

NIMD-01 is provided in gray powder form and put in a glass bottle. Each bottle contains approximately 3 g of reference material.

#### 6. Homogeneity

After NIMD-01 was put into 1200 bottles, 12 of them were selected by stratified sampling and analyzed for methyl mercury, total mercury, copper, zinc, arsenic and selenium. The homogeneity of the sample was evaluated by analysis of variance. The value of uncertainty, which is derived from homogeneity, is included in the uncertainty of the certified value. Therefore, the homogeneity of NIMD-01 is ensured within the range of uncertainty of certified value.

## 7. Instruction on Storage

Store in a clean place and avoid light and high temperature (less than 35 degrees Celsius, frozen/refrigerated storage recommended).

## 8. Instruction for Handling and Use

- (1) Although NIMD-01 is sterilized with  $\gamma$ -ray irradiation, the potential risk as a source of diseases should be considered.
- (2) To homogenize the contents, shake the bottle well before use.
- (3) Leave the bottle at room temperature for more than 30 minutes before measuring sample mass for analytical use. Sampling volume for an analysis should be 20 mg or more to ensure homogeneity.

(4) Moisture rate of NIMD-01 is approximately 5%. All of the certified values were calculated with dry mass. Therefore, measure moisture content of samples to correct obtained values. To measure moisture content, follow the instructions. Measure out samples (>100 mg) in weighing bottles and leave them in an oven at 85 degrees Celsius for 4 hours. After heating, close the lid immediately and let them cool in a desiccator for 30 minutes. Measure their mass and compare them with those measured before heating. The weight difference can be calculated as a moisture content. To avoid evaporation and degradation of components, samples for metrological analysis and those for moisture content measurement should be prepared separately.

## 9. Production Method

To produce NIMD-01, hair materials were collected in Hanoi city by Hanoi School of Public Health. The original hair materials were washed, crushed by roll-press method and pin-mil method, homogenized by mixing and placed into glass bottles. All of the processing operations were conducted by IDEA Consulting, Inc.

#### 10. Reference Material Producer

The producer of NIMD-01 is the National Institute for Minamata Disease, Ministry of the Environment, Japan.

## 11. Participants Involved in Characterization of NIMD-01

Property values of NIMD-01 were characterized by an inter-laboratory study that was conducted by the institutions shown below.

| No.      | Institution  | Country   |
|----------|--|-----------|
| 1        | Balai Besar Laboratorium Kesehatan (BBLK)  | Indonesia |
| 2        | Dong-A University  | Korea     |
| 3        | IDEA Consultants, Inc.   | Japan     |
| 4        | Institut national de santé publique du Québec                                    | Canada    |
| <b>5</b> | International Mercury Lab.   | Japan     |
| 6        | Jožef Stefan Institute   | Slovenia  |
| 7        | Kagoshima University   | Japan     |
| 8        | Lumex Instruments  | Russia    |
| 9        | Milestone General.K.K  | Japan     |
| 10       | National Institute for Environmental Studies (NIES)                              | Japan     |
| 11       | National Institute for Minamata Disease (NIMD)                                   | Japan     |
| 12       | Nippon Instruments Corporation (NIC)   | Japan     |
| 13       | Pusat Penelitian Dan Pegembangan Kualitas Dan Laboratorium<br>Lingkungan (P3KLL) | Indonesia |

| No. | Institution  | Country      |
|-----|--|--------------|
| 14  | SHIMADZU Techno-Research, Inc.                           | Japan        |
| 15  | St. Petersburg University                                | Russia       |
| 16  | Tshwane University of Technology                         | South Africa |
| 17  | University of Ottawa                                     | Canada       |
| 18  | University of Rochester School of Medicine and Dentistry | US           |
| 19  | University of Southern Denmark                           | Denmark      |

Institutions are in alphabetical order

#### 12. Access to Information

Information on any important revision regarding NIMD-01 will be announced at the website of the producer (http://nimd.env.go.jp/english/). Technical information on NIMD-01 can be acquired from the contact address shown below.

## 13. Replicate of Certificate

Indicate as copy when replicating this certificate.

April 10, 2025 National Institute for Minamata Disease Ministry of the Environment, 4058-18 Hama, Minamata City, Kumamoto, 867-0008, Japan TEL +81-966-63-3111 • FAX +81-966-61-1145 http://nimd.env.go.jp/english/

Certification Revision History

April 10, 2025 (Updated expiration conditions: storage temperature and unopened status) October 1, 2023 (Change of expiration) November 1, 2021 (Change of expiration) October 1, 2020 (Change of expiration) August 1, 2020 (Change of expiration) August 1, 2019 (Original certificate date)