Guidance document on test methods to be used for the tier-2 threshold for tailings from mining other than primary mercury mining

Note by the secretariat

1. As is explained in paragraph 15 of document UNEP/MC/COP.5/9, the group of technical experts on mercury waste thresholds worked online to develop a guidance document on test methods to be used for the tier-2 threshold for tailings from mining other than primary mercury mining, as requested by the Conference of the Parties to the Minamata Convention on Mercury in decision MC-4/6.

2. The group of technical experts developed the guidance document through drafting by a small group of experts and agreement by email communication. The guidance document has been posted on the Convention website¹ and is set out in the annex to the present note, without formal editing.

¹ See https://mercuryconvention.org/en/meetings/cop5#sec1564.
Annex*

Guidance document on test methods to be used for the tier-2 threshold for tailings from mining other than primary mercury

I. Background: context from Minamata Convention Article 11

1. Paragraph 2 of Article 11 of the Minamata Convention provides that the definition of mercury waste excludes overburden, waste rock and tailings from mining, except from primary mercury mining, unless they contain mercury or mercury compounds above thresholds defined by the Conference of the Parties. The Conference of the Parties, in its decision MC-3/5, decided that, at the time, there was no need to develop thresholds for overburden and waste rock from mining other than primary mercury mining. The Conference of the Parties, in its decision MC-4/6, defined the following two-tier thresholds above which tailings from mining other than primary mercury mining are not excluded from the definition of mercury waste:

(a) Tier-1 threshold to be applied first: 25 mg/kg total mercury content;
(b) Tier-2 threshold to be applied to tailings above the tier-1 threshold: 0.15 mg/L in the leachate using an appropriate test method simulating the leaching of mercury at the site where the tailings are deposited.

2. The Tier 2 threshold of 0.15 mg Hg/L leachate has been derived from the drinking water concentration limit of WHO (0.006 mg Hg/L), applying the dilution/attenuation factor of the ground layer below the mine tailings at the point of control (200 m away downstream).¹

3. This document provides guidance on test methods to be used for the tier-2 threshold described above.

II. Definition and relevant characteristics of mine tailings

4. The intrinsic mineralogical characteristics of specific mine tailings will dictate the extent to which leaching of mercury is possible, and accordingly, how the mine tailings must be managed to prevent impacts. To understand mine tailings characteristics, leachate release and how to interpret the results of leach tests, it is useful to review what mine tailings are and how they arise.

5. Figure 1 summarises how mine tailings result from a sequence of steps that begins with discovery of an orebody and ends with subsequent concentration processes. Overburden, waste rock and tailings are defined in the report of the United States Environmental Protection Agency to the Congress² as follows:

(a) Overburden: Consolidated or unconsolidated material overlying the mined area
(b) Waste rock: Rock that must be broken and disposed of to gain access to and excavate the ore; valueless rock that must be removed or set aside before the milling process
(c) Tailings: A large-volume waste consisting of the materials remaining after the valuable constituents of the ore have been removed by physical or chemical beneficiation, including crushing, grinding, sorting, and concentration by a variety of methods

6. Many mining operations do not require a concentration step, so no tailings are generated at those sites. Concentration processes are more typically undertaken at hard rock metal mines.

7. Ore is naturally occurring material made up of minerals, which are inorganic compounds having an orderly internal structure and characteristic crystal form and physical properties. In most cases, the main difference in characteristics between the original deposit and the corresponding mine tailings is that the exposed surface area of tailings is much greater, which increases the possibility for reaction with the environment. This can be seen in the particle size distribution of the tailings (See 3a below).

8. In contrast to production of most non-ferrous metal concentrates, copper or gold can be chemically extracted from the bulk composition of crushed and milled ores, or in the case of gold, from tailings. These additional processing steps eliminate or dramatically reduce the mercury content of tailings compared to the native mercury content of the ores they originate from.

9. Otherwise, the orderly internal structure, crystal form and physical properties of the contained minerals are preserved through the process of mining, milling and concentration. In other words, except where leaching is a deliberate part of the production process, the natural minerals present in the mined rock remain the same in the tailings without having undergone any chemical modification. The physical properties of contained minerals are the key drivers of any release of constituents from mine tailings. In most cases, these minerals are quite insoluble in water (See 3a below).

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6 In specific circumstances, sulfide minerals can oxidise in contact with air to produce sulfuric acid, which, if not neutralised by carbonate minerals in the rock, can dissolve constituents. As guidance on specific situations, standard practices to actively manage tailings to prevent deleterious mine drainage are available in the GARD Guide developed by International Network for Acid Prevention (INAP), http://gardguide.com.
10. Mine tailings are expected to be placed at mine tailings management facilities, such as tailings dams. Seepage from mine tailings management facilities depends, amongst other things, on the hydraulic conductivity of the mine tailings. Once deposited on a storage facility, mine tailings become compacted which reduces hydraulic conductivity. The vertical hydraulic conductivity of deposited mine tailings is typically very low given its fine particle size distribution and placement in horizontal lenses (See 3b & 3c below).

11. Climatic conditions can also play a part. In dry climates, limited rainfall may reduce leaching and transport of any constituents that may be leached. In wetter climates, mine tailings may instead be maintained in saturated conditions to prevent mobilisation of elements through oxidation. Other conditions such as freezing/thawing and concentrated storms also affect leaching (See 3b, 3c & 3d below).

III. Factors that influence leach test results

A. Particle size distribution and mineral texture of the sample

12. Leaching describes the release of substances by dissolution from the surface of particles and/or into the pores of a solid matrix. If mercury-bearing minerals are fine grained and liberated during processing, the resultant large surface area will increase their availability for potential leaching. If mercury-bearing minerals remain encapsulated within other minerals, they will be less available for potential leaching (and in some cases may remain effectively immobilised). Whether leaching actually occurs depends on the intrinsic chemical solubility of the individual minerals under the conditions at sites where tailings are deposited. Then, the ability of a leachate to migrate through tailings also depends, amongst other things, on the particle size distribution.

13. Mineral texture defines the general character of a rock, determined by its component particles in terms of grain size and shape, degree of crystallinity and arrangement. The texture of tailings reflects the availability of minerals to potential leaching. Then, the mineral grain structure of the tailings will also affect the transport of any reaction products. Tight grains (displaying low porosity and low permeability) will not easily allow the rapid transport of constituents needed for a reaction to occur, or the flushing out of reaction products.

14. Available content, expressed in mg/kg dry weight, is the fraction of the total concentration of a constituent in the solid phase (mg/kg-dry) that potentially may leach over a reasonably near-term timeframe (e.g., 100 y). As explained above, these will be somewhat dependent on the particle size distribution of the sample, mineral texture of the sample, and the intrinsic chemical solubility of the exposed minerals.

B. Testing Liquid/Solid ratio

15. The Liquid/Solid ratio affects liquid-solid equilibrium over the residence time of eluant in a leach test. For example, in a low ratio system, a build-up of reaction products may result in precipitation of secondary minerals, which cement the tailings, and thereby increase their resistance to leaching (i.e., via encapsulation and/or reduced hydraulic conductivity), lowering the reaction rate or stopping it. In a high ratio system, a build-up of reaction products is less likely, as any precipitated secondary minerals are more readily flushed, which in turn allows for less constrained leaching.

16. This is reflected in studies that have shown that the amount of a given element leached from a given material during leach testing varies with the Liquid/Solid ratio used. Such results depend on a

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8 Hydraulic conductivity can be understood as a characteristic of a formation that relates to its capacity to transmit water through its internal, interconnected pathways (ISO 6107:2021).
complex relationship between severable variables including type of material, solubility limits, test duration, pH, etc.

C.  Test duration

17. Leachate concentration can be near-equilibrium based (if the L/S ratio is low), or kinetically controlled by weathering and diffusion. Most of the tests listed in Section 5 are best suited to measuring near-equilibrium based leachate concentrations. They are conservative in the sense that they do not capture the effect of mineralogical weathering or provide information on reaction rates or release rates of constituent elements more typical at sites where tailings are deposited. For this type of information, repeated testing would be needed on the same material exposed to weathering for different (i.e., longer) durations.

D. Test pH control

18. The pH of the eluant used in a leach test affects the solubility and mobility of the leached elements. For example, some elements such as lead and copper are more soluble in acidic solutions, while others such as arsenic and selenium are more soluble in alkaline solutions. In extreme cases, the pH of the eluant can also affect the stability of the tailings sample itself, which can negatively affect the accuracy and reliability of the leach test results.

Figure 2
General leaching behaviour of anions and cations at different pHs

IV. Key considerations in selecting or defining Leach test methods for the Tier-2 threshold

19. Most of the tests listed in Section 5 have been developed for the characterization of waste, but not for mine tailings specifically. Therefore, when selecting or defining leach test methods for the Tier-2 threshold, it is important to consider the unique properties and potential environmental impacts associated with mine tailings. The data from the leaching tests is one aspect to consider, and it should be adjusted at least for the L/S ratio and the grain size/surface area used in the modelling.

A. Liquid/Solid ratio

20. The choice of liquid to solid ratio in leaching tests is based on a balance between the need to simulate realistic conditions and the practical constraints of the test. A higher liquid to solid ratio may provide a more realistic simulation of landfill conditions, but it may also require longer test durations, which can make the test more difficult and expensive to perform.

21. Typically, an L/S ratio of 10:1 is used to represent a realistic worst-case scenario for landfill conditions. This means that if the waste material can pass the leaching test under these conditions, it is likely to be suitable for disposal in a landfill without posing a significant risk to the environment.

22. As the L/S ratio affects liquid-solid equilibrium over the residence time of eluent in a leach test, the L/S ratio of the chosen test should not differ ±20% from a ratio of 10:1.

B. Test pH

23. At sites where tailings are deposited, weathering processes lead to changes in pH and can ultimately control release. To understand these processes, repeated testing would be needed on the same material exposed to weathering for long periods. However, an initial near-equilibrium state may be observed at a neutral pH, which can be due to slow oxidation, dissolution and interaction of fresh minerals at circum neutral conditions. Most of the tests listed in Section 5 are best suited to measuring such near-equilibrium based leachate concentrations.

24. As no in-situ pH control exists at sites where tailings are deposited, to avoid introducing the undesirable effects described in 3c above, and to accurately capture the initial near-equilibrium state, it is recommended to use an eluant at neutral pH (such as deionized water) without pH control (buffering).

C. Test duration

25. As oxidation, dissolution and interaction of “fresh” minerals in tailings are known to be slow enough to approximate initial near-equilibrium conditions, and weathering processes control release from tailings over the longer term, the duration of the chosen test shall not exceed 24-hours.

V. Overview of Test conditions to be used by Parties

26. This section provides an overview of available test methods used by Parties, compiled by the group of technical experts on mercury waste thresholds under the Minamata Convention.

27. The test methods are intended to provide information about how the waste may be expected to behave in the natural or man-made conditions where waste could be stored or used. The applicability of the test results is therefore naturally different from one country to another, since the natural conditions (rain, evaporation, drainage, frost, etc.) are different from one country to another and also within large countries.

28. Leaching tests are, from a chemical point of view, partial extractions and the result depends on strict compliance with the test conditions (particle size, leaching, duration, temperature, agitation, solid/liquid separation - decantation, centrifugation, frontal filtration, tangential filtration, filter pore size, tolerance of a filter cake, volume of leaching to be filtered, etc.) to obtain good repeatability and reproducibility.

(a) Shake Test: European Standard (EN) 12457-1, EN 12457-2, EN 12457-4

(i) EN 12457-1 and -2: 175-g test portion from a > 2-kg sample of <4 mm in size fraction is mixed by rotation with deionized water at a liquid to solid ratio of 2:1 or 10:1 and shaken once for 24 hours (equivalent to EPA Method 1316).

(ii) EN 12457-4: 175-g test portion from a > 2-kg sample of <10-mm size fraction is mixed by rotation once for 24 hours using deionized water with a liquid to solid ratio of 10:1.

(iii) The suspension is allowed to settle for 15 minutes, and then filtered with a 0.45-µm membrane under vacuum or pressure. If a filter cake builds up (acting as an additional filter and resulting in increased filtration of colloids and microparticles, and lower concentration int the filtrate), a preliminary centrifugation is compulsory (see details in the standards).

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(b) **Japanese Leaching Test No. 13**

(i) This test was developed to achieve rapid short-term extraction of leachable constituents of mining materials.

(ii) 50-g test with 0.5-5 mm with 500 mL deionized water placed in a 1000 ml bottle. Liquid-solid ratios is 10. It is shaken horizontally for 6 hours. The shake speed is 200 rpm.

(c) **Leaching Environmental Assessment Framework (LEAF): EPA Method 1314 and 1316**

(i) A leaching evaluation framework for estimating constituent release from solid materials consisting of four separate methods (EPA 1313, 1314, 1315, 1316) under key environmental conditions and waste properties known to significantly affect constituent release.

(ii) For example, Method 1314 is an up-flow percolation column test designed to evaluate constituent releases from solid materials as a function of cumulative L/S and provides estimate of pore water concentrations and illustrates how leaching behaviour changes as the cumulative L/S ratio increases.

(iii) The Tier 2 threshold of 0.15 mg/L was derived from test data using Method 1314 using L/S ratio of 10:1 and deionized water. For applying this threshold, the same test condition is recommended.

(iv) Method 1316 is an equilibrium-based leaching test intended to provide eluate solutions over a range of L/S. Data provides mass release information as a function of L/S, similar to Method 1314 data, however, Method 1316 eluate concentrations are often higher than Method 1314, reflecting the nature of the batch test where constituents are not sequentially removed from the system at each L/S as with a flow-through percolating column.

(v) The batch method may be useful when characterizing materials with physical properties that make flow through tests impractical.

(d) **Ministry of Environmental Protection, China: HJ 557-2010 Solid waste – Extraction procedure for leaching toxicity – Horizontal vibration method.**

(i) 100-g dry basis test portion (or equivalent) of <3-mm size fraction with deionized water, with a liquid to solid ratio of 10:1, placed in a 2000 ml bottle. It is shaken horizontally for 8 hours at room temperature, then stilled for 16 hours. The shake speed is 110 rpm. The amplitude of vibration is 40 mm.

(ii) If sample contains liquid, extraction fluid should be filtered with a pressured 0.45-µm filter membrane prior to analysis. If percent solids of sample ≤ 9%, the filtrate is the leach solution, should be used for analysis directly. If percent solids of sample >9%, the filter residue (the test) should be tested under the above extraction procedure, the filtrate should be mixed with the leach solution of the filter residue for analysis.

(e) **Federal Ministry of Environment, Switzerland: Test methods in the domain of waste and contaminated sites: Solid test method No.22**

(i) The sampled material is tested if possible in the form in which it will be landfilled.

(ii) The size of the pieces is not reduced as much as possible. When a large quantity of samples has been taken, a representative portion of at least 100 g is withdrawn. If a pre-grinding is necessary for practical reasons (introduction into the test container), one will not go below a size of 0.5 cm.

(iii) For neutral leaching, distilled or demineralised water with a conductivity less than or equal to 10 μS cm−1 is used. At least 100 g of sample are introduced into the leaching vessel, where they are mixed with ten times the amount of water, with reference to the dry weight of the sample. The leaching vessel is

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15 Méthodes de mesure dans le domaine des déchets et des sites pollués.
hermetically sealed and placed for 24 hours in a rotary agitator set to one rotation per minute.

(iv) This test also provides for the use of CO2 saturation to maintain lower pH.

29. Test conditions for the leach test methods reviewed in this section is summarised below.

<table>
<thead>
<tr>
<th>Leach Test</th>
<th>L/S Ratio</th>
<th>Test Duration</th>
<th>Sample Size</th>
<th>pH Control</th>
<th>Test Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shake Test (EN 12457-1, -2, -4)</td>
<td>10:1</td>
<td>24 hours</td>
<td>175g</td>
<td>Neutral</td>
<td>&lt;4mm or &lt;10mm</td>
</tr>
<tr>
<td>Japanese Leaching Test No. 13</td>
<td>10:1</td>
<td>6 hours</td>
<td>50g</td>
<td>Neutral</td>
<td>0.5-5mm</td>
</tr>
<tr>
<td>LEAF (EPA 1314 &amp; 1316)</td>
<td>10:1b</td>
<td>24 hoursa</td>
<td>Varies</td>
<td>Neutralb</td>
<td>Varies</td>
</tr>
<tr>
<td>HJ 557-2010 Solid Waste</td>
<td>10:1</td>
<td>24 hours</td>
<td>100g</td>
<td>Neutralc</td>
<td>&lt;3mm</td>
</tr>
<tr>
<td>Swiss solid test method No.22</td>
<td>10:1</td>
<td>24 hours</td>
<td>100g</td>
<td>Neutrald</td>
<td>Not reduced as much as possible</td>
</tr>
</tbody>
</table>

a EN 12457-1 and -2 also provide for L/S ratio of 2:1 but 10:1 is recommended to test for the Tier-2 threshold.

b LEAF tests can collect data over a range of L/S ratios and pH ranges, but results corresponding to L/S ratio and neutral pH are recommended to be used for the Tier-2 threshold, for more information see https://www.epa.gov/hw-sw846/leaching-environmental-assessment-framework-leaf-methods-and-guidance.

c This test method also provides for CO2 saturated water but neutral pH is recommended to test for the Tier-2 threshold.

VI. Cost Factors in Testing

30. Testing costs can vary worldwide due to factors such as regional labour costs, market competition, regulatory requirements, and overhead expenses. However, the complexity of the methods and the equipment required can have a significant impact on the overall cost.

31. The test methods can be categorized into different levels:

   (a) **Low Complexity**: The Shake Test methods (EN 12457-1, EN 12457-2, EN 12457-4) are relatively straightforward and involve mixing the sample with deionized water and shaking it for a specified duration. The associated equipment requirements are generally simple and may include containers or bottles and a shaking apparatus. These tests are likely to have lower equipment costs.

   (b) **Moderate Complexity**: The Japanese Leaching Test No. 13 and the HJ 557-2010 method require specific shaking conditions and may involve additional equipment such as horizontal shakers or vibration apparatus. These methods are moderately complex and may have slightly higher equipment costs compared to the Shake Test methods.

   (c) **Higher Complexity**: The Leaching Environmental Assessment Framework (LEAF: EPA Methods 1314 and 1316) and the Solid test method No.22 methods can be more involved due to their multiple evaluation methods, flow-through percolation columns, CO2 injection systems, and specific setups. These methods are likely to have higher equipment costs due to their increased complexity and specialized requirements.

VII. How to collect a representative tailing sample

32. Waste from the extractive industry differs considerably from the waste types and sampling scenarios typically addressed in guidance documents. The approach to sampling described in this document is primarily focused on the requirements to undertake mineralogical and geochemical testing of mining waste.

33. **In situ** tailings are not typically sampled at a regular frequency due to accessibility constraints and health and safety risks. Instead, tailings are sampled at an accessible point prior to their deposition in a tailings storage facility, such as from a thickener underflow stream or a tailings pump box typically located at the processing facility, or less commonly from a spigot located at the tailings storage facility.
34. Tailings storage facilities should be included in surface and groundwater monitoring programs\textsuperscript{16}, such that the performance of tailings storage facilities, in terms of effects of any tailings seepage on water quality, can be evaluated (e.g., actual mercury concentrations in groundwater at relevant monitoring wells that can be compared to the WHO Drinking Water Standard of 0.006 mg/L).

35. CEN/TR 16365:2012 Characterization of waste - Sampling of waste from extractive industries - gives further specific information on sampling for testing of waste from the extractive industry to support the development of appropriate sampling plans.

\textsuperscript{16} global-industry-standard-on-tailings-management.pdf (globaltailingsreview.org).