Guidance on Analysis of Glass Matrices for Toxics in Packaging

Toxics in packaging laws prohibit the intentional use of any amount of four heavy metals in packaging – lead, cadmium, mercury, and hexavalent chromium. If the metals are incidentally present (defined as an unintended or undesired ingredient) in the packaging material, the laws restrict the sum of the total concentration of these four metals to less than 100 parts per million. Manufacturers and suppliers[1] of packaging must certify to these two requirements – no intentional use and the sum of total concentration of the four metals do not exceed 100 ppm – in order to sell or distribute packaging or for their customers to sell or distribute packaged products in states with the law.[2]

This document provides guidance for manufacturers and suppliers of glass packaging, retailers, and analytical testing laboratories for measuring heavy metals in packaging when seeking to demonstrate compliance with toxics in packaging requirements. This guidance is based on the results of a study to evaluate analytical methods for determining the total concentrations of lead and cadmium in glass samples. The study was conducted by the Toxics in Packaging Clearinghouse (TPCH) under contract to the California Department of Toxic Substances Control (DTSC). The complete report, *Glass Matrix Test Methods Evaluation for Toxics in Packaging*, is available on the TPCH website.

Background

State toxics in packaging laws restrict the total concentration of metals in packaging. As early as 2007, TPCH and its member states began to suspect that regulated entities and commercial analytical laboratories were experiencing difficulty in evaluating packaging samples for compliance with state toxics in packaging laws. It was suspected that insufficient dissolution of packaging samples was occurring in samples being tested, resulting in inaccurate results and underreporting of regulated metals in packaging samples. Measuring the total concentration of metals requires

[1] "Supplier" means: Any person, firm, association, partnership, or corporation who sells, offers for sale, or offers for promotional purposes packages or packaging components which shall be used by any other person, firm, association, partnership, or corporation to package (a) product(s).

[2] For more information on toxics in packaging requirements, visit the Toxics in Packaging Clearinghouse website at www.toxicsinpackaging.org.

that a sample be completely dissolved in solution for detection by conventional laboratory instrumentation (such as Inductively Coupled Plasma/ICP spectroscopy) that relies on the detection of metals in a solution. In other words, in order to effectively and accurately measure total metals in a sample, the sample matrix must be completely digested/dissolved to liberate the metals in order to be measured. In 2011, TPCH released a test method evaluation report and guidance for analytical testing of packaging for compliance with state toxics in packaging requirements, focusing on polyvinylchloride (PVC) packaging which is a difficult to dissolve material.

This guidance document continues the efforts of TPCH to provide guidance to industry on appropriate analytical test methods to determine total concentration of metals, focusing on glass matrices. Like PVC, glass matrices are also difficult to dissolve into solution and require careful attention in the selection of sample preparation methods to achieve the objectives of toxics in packaging requirements, which are based on total, and not just "leachable", or "recoverable" metal concentrations.

Guidance on test methods for the evaluation of glass matrices

Based on the findings of the glass matrix test method evaluation study, TPCH recommends the use of:

- **EPA SW-846 Method 3052 or comparable method using hydrofluoric acid (HF) for the analysis of glass matrices when traditional laboratory "wet" chemistry analysis is required.** The analysis method should include the application of an external heat source (e.g., microwave digestion) to assist in acid digestion.

  Method 3052 and comparable sample preparation methods using HF effectively liberate lead (Pb) and cadmium (Cd) from the glass matrix for the determination of total metals concentration. Sample preparation methods using HF without the application of an external heat source such as microwave, oven bomb or hot plate do not perform as well. EPA Method 3050B, which is designed to measure "total recoverable metals" is inadequate in the liberation of Pb and Cd from glass matrix samples, and in the TPCH study, consistently failed to accurately measure Pb or Cd in glass matrices.

  Method 3052 requires the use of HF for silica-based materials, such as glass. Laboratories capable of performing Method 3052 are commercially available in the U.S. and globally. In the TPCH study, commercial laboratories offering Method 3052 included those that perform consumer product testing, environmental analyses, and specialty testing targeting glass, inorganic substances, and metals.

- **Laboratories with a demonstrated capability to perform EPA SW-846 Method 3052 and complete sample dissolution.** Laboratories may state that they are capable of performing EPA SW-846 Method 3052 on glass samples; however, in its recent study TPCH found that an assertion by the laboratory in this regard was insufficient to confirm the laboratory's technical capability. It is important to select laboratories that employ effective QA/QC procedures, and to clearly communicate with the laboratory that the intended outcome is to measure the total concentration of the restricted metals in the sample, which can only be achieved with complete sample dissolution.

- **XRF analysis for compliance screening of glass samples for toxics in packaging laws.**[3] XRF spectroscopy offers an alternative to EPA Method 3052 and the use of HF for the non-destructive analysis of metals in glass matrices. In the TPCH glass matrices test method evaluation, XRF results were often, but not always, comparable to EPA Method 3052 in determining the concentration of Pb and Cd. In the study, XRF analysis detected the presence of Pb and Cd in all samples known to contain these substances and did not produce false positives when the margin of error was taken into account.

  When restricted metals are detected above 100 ppm using XRF analysis further action is warranted. For example:

  — If Pb concentration is greater than 100 pm, the sample should be considered non-compliant with most state toxics in packaging laws and action taken to prevent the glass packaging from entering the supply stream and/or removing non-compliant glass from the supply stream.[4] Analytical testing using EPA Method 3052 may be undertaken to confirm Pb concentrations and to verify the compliance status of the glass.

  — If Pb concentration is close to 100 ppm (e.g., within +/- 25 ppm), analytical testing using EPA Method 3052 should be considered to confirm Pb concentrations.

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[3] It is important to note one limitation of XRF analysis when used for compliance screening. XRF instruments only detect total chromium, not hexavalent chromium, which is another metal restricted by state toxics in packaging laws. This limitation does not preclude the use of XRF analysis as an alternative to laboratory "wet" chemistry. Instead, if chromium is detected using XRF, further analysis for chromium may be warranted to determine if the chromium is hexavalent chromium.

[4] The Model Toxics in Packaging Legislation currently provides an exemption for the use of recycled content (exemption 5c) with a threshold limit of 200 ppm. States are not bound by the Model but are strongly encouraged to adopt revised provisions to stay in sync. However, as of the date of this publication, only one TPCH member state (New Hampshire) has adopted these revisions. Other member states have allowed the exemption for recycled content to lapse or maintain an exemption for recycled content with a threshold limit of 100 ppm. State toxics in packaging laws prevail over the Model Legislation. Parties testing glass packaging should confirm the applicable threshold limit.

— If chromium is detected above 100 ppm, analytical testing may be needed to confirm the valence state of chromium, and specifically to determine if hexavalent chromium is present in the sample. Alternatively, knowledge of the raw material inputs into the process, and specifically the source and valence state of the chromium introduced into the manufacturing process, may substitute for further laboratory analysis.

General guidance for regulated entities
(such as packaging suppliers, manufacturers, distributors, purchasers, and retailers)

When requesting testing services from laboratories, it is important to communicate testing requirements and data quality objectives. Toxics in packaging laws require analysis of all packaging components for **total concentration** of the four restricted metals, which is possible only through **complete sample decomposition**. If complete sample decomposition is not achieved, this fact must be reported on the test report, as it strongly impacts the accuracy of the results. This is very important when working with laboratories that typically conduct analyses for "total recoverable" metals (i.e., leaching for hazardous waste or site characterization) as they might not be as familiar with requests for total concentration of metals in products, packaging, or otherwise unique matrices.

Regulated entities should be proactive:

- Communicate the data quality objective of "complete sample decomposition" to laboratories and request that laboratories include in their test reports information on the degree of sample decomposition. This information will provide regulated entities with some assurance that appropriate test methods were used by the laboratory for determining compliance with state toxic in packaging laws. Providing decomposition information in test reports will save all stakeholders (regulated entities, laboratories, state agencies) the time of having to ask for this information or sort through laboratory records for this information, if laboratory test reports are requested and scrutinized by state agencies.

- If test reports indicate the presence of the four metals restricted by state laws in the sample, it is prudent to follow up with laboratories to determine the degree to which the sample was decomposed, if this information is not available on the test report. If the sample was documented as not being completely decomposed, the analysis, including sample preparation, should be repeated. Re-analyzing samples is particularly important if any amount of the restricted metals is detected in the initial test, since further or complete decomposition of the sample matrix may result in detection of one or more of the restricted metals in excess of the regulatory limits.

- Conventional communication mechanisms with laboratories may not be sufficient to achieve the above-mentioned goals. For example, many laboratories utilize standardized

test request forms, where the customer checks a box for "toxics in packaging" testing that may not provide the details needed to the lab personnel performing the actual test procedure. Further, customer service representatives may not be familiar with the technical details for sample preparation methods specific to toxics in packaging or packaging materials. Address these concerns through detailed conversations with the laboratory, including assurances from the technical staff, before securing laboratory testing services.

General guidance for testing laboratories

Regulated entities rely on analytical testing laboratories to assess compliance with toxics in packaging regulations. The adoption of appropriate sample preparation methodologies and their execution to achieve complete decomposition of packaging matrices are critical to ensure confidence in testing results and assure compliance.

Laboratories that perform testing services for toxics in packaging should:

- Evaluate current sample preparation methods used in determining the restricted metals content for glass matrices to ensure that the methods used achieve complete decomposition of the sample. Complete sample decomposition should be the objective of selected methods such as EPA SW-846 Method 3052 or a comparable methodology.

- Add a comment field to test reports that documents whether the sample was completely decomposed (e.g., percent dissolution of the sample). TPCH has found that the data quality objective of "complete sample decomposition" is the critical factor in determining the accuracy of test results for toxic in packaging requirements. A simple statement of the test method used does not convey this information. Providing decomposition information in test reports allows all stakeholders (regulated entities, laboratories, state agencies) to save time by not having to return to the lab to retrieve this information if test reports are scrutinized by state agencies.

- Expect to re-analyze samples if complete sample decomposition is not achieved. Some matrices may require experimentation with sample preparation methods until complete sample decomposition can be achieved.

Please contact TPCH or visit the TPCH website for more information.

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