Guidance on Laboratory Analysis 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 component or material, the laws restrict the total concentration of these four metals combined to less than 100 parts per million. Suppliers[1] and manufacturers of packaging must certify to these two requirements – no intentional use and the sum 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 suppliers of packaging and packaging components, manufacturers and distributors of packaged products, 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 round-robin study to evaluate the performance of testing laboratories in determining compliance with toxics in packaging statutes. 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, *Laboratory Round Robin Test Project: Assessing Performance in Measuring Toxics in Packaging*, is available on the TPCH website.

Background

For the past five years, TPCH has screened packaging for compliance with state toxics in packaging laws using x-ray fluorescent (XRF) analysis. XRF analysis is a rapid and inexpensive screening tool for measuring the elemental composition of samples, including the four metals

[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.

restricted by state laws – cadmium, lead, mercury, and hexavalent chromium.[3] Companies were notified of the XRF screening results that showed heavy metals above the 100 ppm limit, and asked to demonstrate compliance with state laws or remove non-compliant packages from retail shelves. In response, companies often submitted laboratory test reports showing "passing" test results and claimed compliance with toxics in packaging requirements for many of the packages that failed the TPCH screening tests.

Although XRF is recognized as only a screening tool, TPCH has experienced good correlation between laboratory analysis and XRF, particularly when metals are present above the 100 ppm threshold. The underlying cause of discrepancies between XRF and laboratory analysis appeared to be in the selection of dissolution methods for preparing packaging samples for analysis. Simply put, if the sample is not completely digested, the restricted metals, if present, are not sufficiently liberated from the packaging material and cannot be accurately measured by the laboratory analytical equipment, since analytical instruments, such as ICP, measure the concentration of substances liberated into the solution. When complete dissolution of the matrix was achieved, the analytical results demonstrated a much better correlation with XRF screening results.

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 total sample decomposition is <u>not</u> 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 absolute total concentration of metals in products, packaging, or otherwise unique matrices.

Regulated entities should be proactive:

- Communicate the data quality objective of "total sample decomposition" to laboratories and request that laboratories include in their test reports information on sample decomposition. This information will provide regulated entities with some assurance

[3] XRF measures total chromium, not hexavalent chromium. If chromium is detected using XRF, laboratory analysis would be needed to determine if the chromium is hexavalent chromium.

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 scrutinized by state agencies. Additional information on sample preparation methods is available on the TPCH website, Frequently Asked Questions.

- If test reports indicate that any amount of the four metals restricted by state laws are present in the sample, it is prudent to follow up with laboratories to determine whether the sample was totally decomposed, if this information is not available on the test report. If the sample was not documented as being totally decomposed, the analysis, including sample preparation, should be repeated. Some matrices may require experimentation with sample preparation methods until complete sample matrix decomposition is achieved. 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 ideal for achieving 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.

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 total 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 different types of packaging materials, including difficult to digest matrices such as PVC, 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 an equivalent methodology. Additional information on sample preparation methods is available on the TPCH website, Frequently Asked Questions.

- Note that EPA SW-846 Method 3052 does NOT require the use of hydrofluoric acid for decomposition of organically-based matrices like PVC. Over the years, a number of laboratories or their customers have reported to TPCH that the laboratory would not use Method 3052 as it requires the use of hydrofluoric acid. A careful review of the sample preparation procedure for Method 3052 reveals that hydrofluoric acid is not required, but a combination of other acids (e.g., hydrochloric acid, nitric acid, hydrogen peroxide), combined with microwave digestion, may accomplish the goal of complete sample matrix decomposition.

- Add a comment field to test reports that documents whether the sample was totally decomposed (e.g., percent dissolution of the sample). TPCH has found that the data quality objective of "total 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 total sample decomposition is not achieved. Some matrices may require experimentation with sample preparation methods until total sample decomposition can be achieved.

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

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