TPCH Research Bulletin
December 2012

XRF Screening of Packaging Components: Inks & Colorants

Since 2006, the Toxics in Packaging Clearinghouse (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 restricted by state laws -- cadmium, lead, mercury, and hexavalent chromium.[1] This report summarizes the results of the fourth XRF screening project of the TPCH. This project screened inks and colorants in single-use shopping and mail order bags for the presence of lead and cadmium. Two previous projects by the TPCH identified inks and colorants as a potential source of lead and cadmium in packaging.

Background

The use of four metals -- lead, cadmium, mercury, and hexavalent chromium-- are restricted by state laws in nineteen U.S. states. These laws prohibit the intentional use of any amount of these four metals in any packaging or packaging component, such as inks and colorants. If the metals are incidentally present (defined as an unintended or undesired ingredient) in the packaging component or material, the laws restrict the sum total concentration of these four metals to less than 100 parts per million.

The TPCH supports and helps coordinate the implementation of states' toxics in packaging laws. Nine states -- California, Connecticut, Iowa, Minnesota, New Hampshire, New

[1] 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.

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Jersey, New York, Rhode Island, and Washington --are members of the TPCH. The TPCH, which is administered by the Northeast Recycling Council, Inc. (NERC), serves as a central location for processing information requests from external constituencies and promoting compliance with the laws. The TPCH also undertakes research projects such as this compliance screening project to assist member states in identifying potential sources of non-compliant packaging and conducting outreach to industry to bring about change.

Methodology

The TPCH and its member states collected retail packaging samples from November 2011 through February 2012. Members focused mostly on shopping and mailing bags, but also secured some food packaging with printing inks.

The packaging samples were screened using an Olympus Innov-X Systems DELTA handheld XRF instrument, following manufacturer recommended procedures for RoHS mode.[2] Each sample was analyzed for 60 seconds. A single measurement was taken of each sample unless the initial reading detected one of the target metals at a concentration of 100 ppm or greater. When any of the target metals was detected in the initial measurement a duplicate reading was taken.

Multiple packaging components of individual samples were isolated and screened, if applicable and feasible. For example, some packaging samples contained more than one color ink or some bags were a solid color with contrasting print. Isolation of individual packaging components was aided by the camera built into the DELTA instrument. The camera allowed the technician to locate and center the analyzer window over the packaging component of interest. Samples failing the XRF screening-- >100 ppm of one of the restricted metals -- were sent to the Washington State Department of Ecology, where they were screened again using XRF analyses, and then forwarded to the Washington State Manchester Environmental Laboratory for confirmatory testing. The Laboratory followed EPA SW Method 3052 for the preparation of

[2]Olympus Innov-X System User Manual, Delta Family: Handheld XRF Analyzers, June 2010; and Quick Start Guide, DELTA Family of Handheld XRF Analyzers, August 2010.

samples and EPA Method 200.8 for analysis. EPA Method 245.5 was used for sample preparation and analysis for mercury.

Packaging Samples

A total of 132 packaging samples were screened. Packaging was obtained from ten states with toxics in packaging laws, from east coast to west coast. Ninety-five percent of the packaging samples (125) were shopping or mailing bags. Seven samples (5%) were food packaging. Similarly, 95 percent of the samples were inks or colorants on plastic, and 5 percent were inks on paper-based packaging. The project screened a total of 181 unique inks and colorants applied to or used to print on base materials (plastic or paper). A broad range of colors and shades of colors were screened, including blue, red, green, yellow, black, gray, orange, and purple.

Results

Three packaging samples, less than 3 percent of the samples screened, failed the XRF screening for lead. The failing samples were all solid-colored plastic bags, two yellow and one red. The concentration of lead in all three packaging samples was approximately 10,000 ppm, or 1 percent by weight. Only one of the bags was marked with the country of origin, and in that case it was the U.S.

XRF screening was repeated by the Washington Department of Ecology prior to laboratory analyses at the Washington State Manchester Environmental Laboratory. Both the Washington State XRF screening and laboratory analysis confirmed that these packages were not in compliance with state laws. The table on the next page summarizes the XRF and laboratory results.

Sample #	Packaging Type	Description	TPCH XRF Olympus Innov-X DELTA Pb (ppm)	WA XRF Niton XL3t Pb (ppm)	WA Laboratory Analysis Pb (ppm)
1	Charitable donation bag	Yellow plastic bag	8,952	9,789	9,730
2	Retail shopping bag	Yellow plastic bag	9,831	8,380	10,600
3	Retail shopping bag	Red plastic bag	11,179	11,966	498

The XRF screening performed by the TPCH and the Washington Department of Ecology returned consistent results. Two of the three laboratory results were also consistent with the XRF analysis, while one was significantly lower. Regardless of the discrepancy, the lead concentration in the packaging sample was well over the 100 ppm threshold for determining compliance.[3]

While the reason for the discrepancy is unknown, there are several possible explanations, including: incomplete digestion of the sample prior to laboratory analysis; a lower metal concentration in the portion of the bag subjected to the laboratory analysis compared to the XRF sample; or inaccurate XRF readings resulting from spectral interference.

Discussion

Overall, the TPCH member states were pleased with the high level of compliance with state toxics in packaging laws. An early XRF screening project by the TPCH, released in 2007, showed almost 17 percent non-compliance for plastic shopping bags of a total of 60 samples screened. The TPCH also included some retail shopping bags that failed in the 2007 project in

[3] State toxics in packaging laws prohibit the intentional use of any amount of the four regulated metals. The incidental presence (defined as unintended ingredients) of the four metals combined cannot exceed 100 ppm.

the current screening, and the XRF results on these new bags indicated they were in compliance. The TPCH and member states are encouraged by these results, which may indicate manufacturers and distributors of plastic shopping and mailing bags are paying more attention to sourcing and testing for compliance with toxics in packaging laws.

The TPCH learned from previous screening projects that compliance could be problematic with yellow plastic shopping bags. The results of this project confirm that retailers should proceed with caution when specifying or purchasing yellow plastic shopping bags. It is likely that the bags tested for this project contained lead chromate pigment, since the XRF analyses of these bags also showed the presence of chromium. The TPCH will continue to monitor these types of packaging.

No inks used to print on bags --plastic or paper -- failed the screening tests. The TPCH expects that these results are accurate; however, the TPCH is cognizant that the results could possibly be compromised by the thickness of the ink on the substrate. The minimum recommended sample thickness for XRF analysis is 5 mm. The thin layer of printing ink on the surface of the substrate does not meet this requirement, although most samples had enough ink coverage to create a sample several layers thick. In contrast, the shopping bags that were infused with color could be easily folded to achieve the recommended sample thickness.

In conclusion, inks and colorants are ideally tested for compliance with toxics in packaging requirements before they are applied, blended, or otherwise used in packaging applications. Inks and colorants are considered packaging components under state toxics in packaging laws, and therefore, must individually meet the law's requirements. If testing of the finished product is the only option or is undertaken for quality assurance purposes, then care should be taken to isolate the inks to the extent possible and apply best practices for analysis. If any of the regulated metals are detected, further investigation should be undertaken to separate the individual packaging components and ensure that the inks or colorants do not violate state toxic in packaging requirements.