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An Assessment of Heavy Metals in Packaging: Screening Results Using a Portable X-Ray Fluorescence Analyzer

Final Report

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The Toxics in Packaging Clearinghouse

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I. Executive Summary

Nineteen U.S. states have toxics in packaging laws that prohibit the sale or distribution of packaging containing intentionally added cadmium, lead, mercury, and hexavalent chromium, and set limits on the incidental concentration of these materials in packaging. The purpose of these laws is to prevent the use of toxic heavy metals in packaging materials that enter landfills, waste incinerators, recycling streams, and ultimately, the environment.

With funding from the U.S. Environmental Protection Agency, the Toxics in Packaging Clearinghouse (TPCH) initiated the first comprehensive test program of packaging in the U.S. TPCH screened 355 packaging samples between October 2005 and February 2006 for the presence of the four restricted metals using a portable x-ray fluorescence (XRF) analyzer. The packaging samples were selected to represent different packaging materials (aluminum, glass, paper, plastic, and steel) and product types, mostly in the retail sector.

Of the packages tested, 16% exceeded the screening threshold of 100 parts per million (ppm) for the presence of one or more of the restricted heavy metals, and may be in violation of state toxics in packaging laws. Cadmium and lead were the most frequently detected of the four regulated metals. Historically, these metals were used in colorants and inks, and as stabilizers to retard the degradation of plastics exposed to heat and ultraviolet light. The average cadmium concentration detected in the samples that failed the screening test was 449 ppm while the average lead concentration was 1,740 ppm. Test results for one package, a plastic mailing bag, indicated that the package was almost 1% (10,000 ppm) lead by weight.

There were two types of packaging that dominated the samples failing the screening test:

1) **Flexible polyvinylchloride (PVC) packages**. This “heavy-duty” plastic material is frequently found in packaging of textiles, cosmetics, inexpensive toys, and pet supplies. Examples of the packages tested are the zippered bags used to package home furnishings, such as comforters, and the plastic pouches used to package pet toys. In the TPCH screening project, 61% of this packaging type was not in compliance with toxics in packaging laws, due to excessive levels of cadmium and/or lead. Almost all of the flexible PVC packaging samples tested were from products imported from Asia, according to the product label. Interestingly, all PVC “blister packs,” which are semi-rigid and in this study were mostly imported from Asia, passed the screening tests.

2) **Inks and colorants used on plastic shopping/mailing bags**. Lead was most often found in the shopping bags that failed the screening test, but the XRF instrument

also detected mercury and chromium in some samples. The elevated levels of the restricted metals again appear to be largely from packages of products imported from Asia, where solvent-based inks that contain these heavy metals are still used.

The TPCH test program was designed to screen packages for the presence of the four restricted metals. Based on the results of the test program, companies selling or distributing packages that failed the screening test received notification of the test results. TPCH requested that these companies certify compliance with state toxics in packaging laws and provide supporting analytic data, or notify TPCH of non-compliant packages and discontinue the sale and distribution of the package.

Working with companies to determine the compliance status of the packages was more challenging than anticipated. Companies verified the TPCH test results, acknowledging that their packages were not in compliance with state toxics in packaging requirements, for only 15% of packages (8 of 52 failure notifications). Companies claimed compliance and submitted supporting documentation for almost 70% of the packages that failed the TPCH screening tests. Companies made no claims for the remaining 15% of the failure notifications, most often citing that the product was discontinued and therefore the packaging was not available for testing. In addition, several companies simply did not respond to multiple TPCH notices and the file was turned over to state agencies for possible enforcement action.

There are several possible explanations for the discrepancy between the screening test results and company claims. First, suppliers or raw materials changed and the package tested was not manufactured with the same material as the TPCH test package. Second, in some cases, TPCH suspects that conventional laboratory preparation methods are insufficient to adequately digest the packaging sample and liberate the metals, resulting in the measurement of “recoverable” metals, not a true total concentration of the restricted metals. Finally, the XRF technology has its limitations as well. For example, XRF detects total chromium, not hexavalent chromium, which might have contributed to some false positives for hexavalent chromium.

As a result of the compliance-screening project, the Toxics in Packaging Clearinghouse began a new outreach and education campaign aimed at increasing awareness of state restrictions on heavy metals in packaging. The California Department of Toxic Substances Control (DTSC), a TPCH member, is also undertaking a project designed to compare different test methodologies for determining total concentration of the restricted metals in packaging samples. This project will provide guidance to the regulated community on acceptable test methods. TPCH, in conjunction with its member states, also plans to conduct additional compliance screening programs in the future to detect trends in compliance with state toxics in packaging requirements.

II. Introduction

Nineteen U.S. states have toxics in packaging laws that prohibit the sale or distribution of packaging containing intentionally added cadmium (Cd), lead (Pb), mercury (Hg), and hexavalent chromium (Cr^{+6}), and set limits on the incidental concentration of these materials in packaging. The purpose of these laws is to prevent the use of toxic heavy metals in packaging materials that enter landfills, waste incinerators, recycling streams, and ultimately, the environment.

Box 1

States with Toxics in Packaging Laws

* Indicates TPCH Member State

*** California**	*** New Hampshire**
*** Connecticut**	*** New Jersey**
Florida	*** New York**
Georgia	Pennsylvania
Illinois	*** Rhode Island**
*** Iowa**	Vermont
*** Maine**	Virginia
Maryland	Washington
*** Minnesota**	Wisconsin
Missouri	

With funding from the U.S. Environmental Protection Agency, the Toxics in Packaging Clearinghouse (TPCH) initiated the first comprehensive test program of packaging in the U.S. on behalf of its member states, all of which have toxics in packaging laws. Ten additional U.S. states have similar laws, based on the Model Toxics in Packaging Legislation. A list of states that adopted the Model Legislation is provided in Box 1.

Based on the results of the study, TPCH launched an outreach initiative to educate the packaging supply chain about toxics in packaging requirements, and to bring non-compliant packages into compliance, thereby reducing the presence of toxic heavy metals in packaging.

A. Background on Toxics in Packaging Legislation

Nineteen states have toxics in packaging laws based on the (formerly “CONEG”) Model Legislation. State toxics in packaging laws prohibit the intentional use of *any amount* of lead, cadmium, mercury, and hexavalent chromium in packaging or individual packaging components, such as inks, adhesives, or labels. If the regulated metals are unintentionally present, for example, as a contaminant in raw material feedstocks, then these state laws limit the total concentration of the sum of the metals to below 100 ppm in any package or individual packaging component. Limited exemptions are available for recycled-content, reusable containers, and packages regulated by other federal and state laws.

These requirements apply to all packaging and packaging components offered for sale or for promotional purposes by the manufacturer and distributor (including importers) in states with toxics in packaging legislation. The state laws further require self-certification by companies, and require companies to produce a Certificate of

Compliance upon request. Most TPCH member states have included in their laws the ability to levy substantial monetary penalties for non-compliance.

The Toxics in Packaging Clearinghouse coordinates implementation of the legislation on behalf of its member states, and serves as a single point of contact for companies seeking further information, clarification of specific details, or an exemption to toxics in packaging requirements. Manufacturers, distributors, and retailers must deal directly with states that have adopted toxics in packaging legislation but are not members of the TPCH. For more information on toxics in packaging legislation and the Clearinghouse, visit www.toxicsinpackaging.org.

III. Methodology

The TPCH compliance assessment project was designed to screen packaging for compliance with the Model Legislation and state laws based on the Model. Packages, mostly from the retail market, were screened for the presence of the four restricted heavy metals -- cadmium, lead, mercury, and hexavalent chromium[1] -- using a portable x-ray fluorescence (XRF) analyzer. The XRF testing device allowed TPCH to make a rapid determination whether a package was likely to pass or fail the toxics in packaging requirement for the total concentration of the four heavy metals.

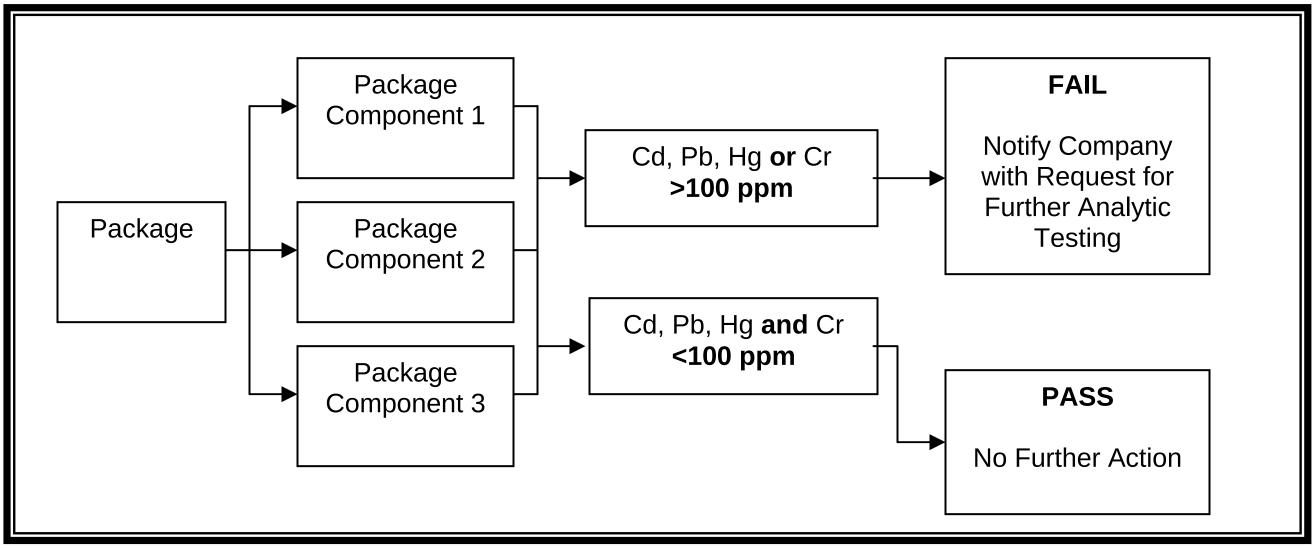
As depicted in Figure 1, if the concentration of one or more of the restricted metals exceeded 100 ppm in one or more packaging components, the package “failed” and the product manufacturer or distributor was notified of the test results. One hundred parts per million (100 ppm) was chosen as the trigger for notification letters in the compliance test program because this concentration level indicates potential non-compliance due to intentional introduction and/or incidental presence. The screening test protocol did not differentiate between intentional and incidental presence of heavy metals.

Manufacturers and distributors selling suspected non-compliant packaging were provided with information on the Model Toxics in Packaging Legislation and state laws based on the Model. TPCH requested that these companies certify compliance and provide supporting analytic data, or notify the TPCH that the package was not in compliance with state laws and discontinue the distribution and sale of the package in TPCH member states.

The screening results were also used to identify patterns of potential non-compliance within specific sectors of the marketplace. These market sectors are the subject of ongoing outreach efforts aimed at educating the supply chain about the legal requirements that prohibit the use of toxic heavy metals in packaging.

[1] The NITON XRF analyzer measures total chromium, not hexavalent chromium. If total chromium exceeded 100 ppm, TPCH requested a Certificate of Compliance from the company with laboratory test results documenting the level of hexavalent chromium in the packaging.

Figure 1: XRF Screening Test Protocol



The following sections describe the packaging selection and acquisition protocols, the test procedures, and the outreach strategy.

A. Sample Selection and Acquisition

TPCH screened 355 packages, representing different product sectors, packaging types (e.g., bags, boxes, and caps), and material types (aluminum, glass, paper plastics, and steel) as outlined in Box 2. Packaging was selected for testing in two ways: 1) randomly from among the variety of packaging materials and products in the marketplace; and 2) targeted based on previously reported or alleged violations or characteristics that may increase the likelihood of non-compliance (such as product type, material type, ink colors, manufacturing location).

Table 1 summarizes the types of packages tested by product sector. Packaging samples were collected in all nine TPCH member states, and generally were acquired by TPCH members and staff through routine business or personal purchases. TPCH members and staff purchased additional products, as needed, to obtain the desired distribution of package types. Collection forms recorded information on the sample, its purchase, and chain of custody. All samples were delivered to TPCH (either hand delivered or by U.S. mail) for testing, where the sample was assigned a sample number. Descriptive information on each sample was recorded directly into the XRF software prior to testing.

Each of the 355 packaging samples was separated into individual packaging components, resulting in a total of 570 packaging components screened over the course of this project. For example, a soda bottle was separated into three packaging components: the resin bottle, the cap, and the paper or plastic label. It was not always possible to separate all packaging components; ideally, colorants, inks, and adhesives

Box 2: Major Characteristics of Packaging Considered in Sample Selection

The following characteristics and descriptions are not meant to be a definitive list but rather a starting point. The lists below include retail and other classes of packaging. The test program selected mostly retail packaging.

Product category
Agricultural & garden; animal care; apparel; automotive; building/construction; cleaning & chemicals; distribution; electrical & electronic; entertainment; food & beverage; food service; furniture; hardware & machinery; jewelry; healthcare; household goods; novelty/promotional; paints, coatings & adhesives; personal care; retail trade (e.g., shopping bags); sporting goods; stationary/office supplies; textiles (other than apparel); toys & games.[1]

Package construction
Bag; barrel; blister pack & clamshell; bottle; box; can; crate; drum; envelope; jar; laminate (includes aseptic); pallet; pouch (bag or sack holding several items); strapping; tank; tube; wrap/film.[2]

Packaging components
Adhesives; closures; cushioning material; electronic components; fasteners; handles; inks, dyes, and pigments; labels (printed on package, separate label affixed to package); seals; tags.

Materials
Aluminum; ceramic; composites; glass; paper; paperboard; plastic (different resin types); steel; wood.

Origin
Domestic; import.

Color
Natural; red; yellow; orange; green; blue; white; gray; black; etc.

[1] *This list of product categories is drawn from the North American Industrial Classification System – NAICS – at www.census.gov/epcd/naics02/naicod02.htm. Some categories were combined or customized to enhance applicability to packaging.*

[2] *The package construction categories are based on ASTM Standard Terminology of Packaging and Distribution Environments (ASTM D996-95).*

should be tested individually. Since this project acquired finished packages (e.g., shopping bags with graphic designs) and not the raw materials, packaging components were isolated to the extent possible.

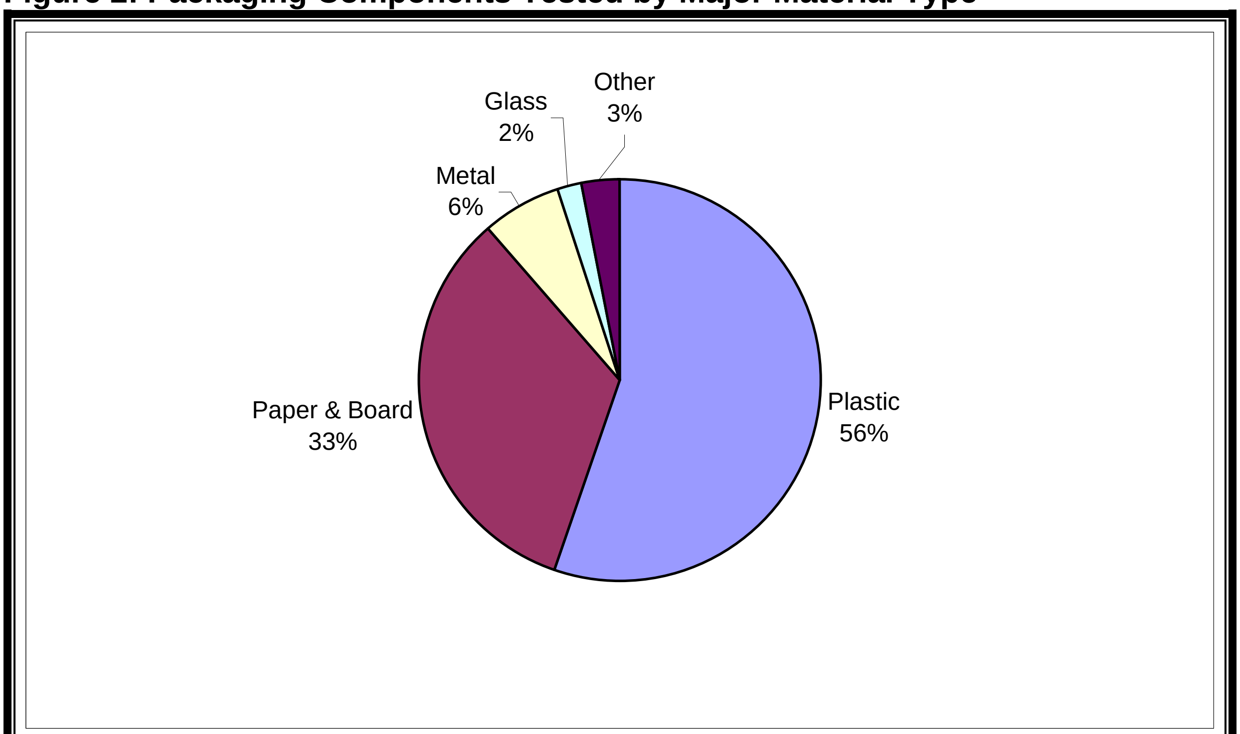
Figure 2 provides a breakdown of the major materials in the packaging components tested.

Table 1: Types of Packages Tested by Product Sector

Random Selection		Targeted Selection[1]	
Product Sector	**No. of packages**	**Product Sector or Package Type**	**No. of packages**
Grocery (Food)	30	Shopping Bags	68
Beverage	24	Toys & Games	19
Personal/Healthcare	16	Produce Bags	17
Electrical/Electronic	14	Textile & Home Furnishing Bags	16
Apparel/Accessories	11	Mailing/Shipping Bags	14
Cleaning Products	10	Grocery In-store/Self Service Bags	13
Entertainment	10	Inexpensive Novelty Items	12
Hardware & Automotive	8	Mexican Candy/Food Products	12
Household Goods	7	Steel Strapping	9
Fast Food	6	Steel Cans	5
Office Supplies	6	Eastern Herb/Food Products	5
Pet Supplies	6	Cosmetic/Toiletry Bags	4
Sporting Goods	6	Tools- Hand & Power	1
Cosmetics	5	Light Up Packages	1
Subtotal	159	**Subtotal**	196
Total Packaging Samples: 355			

[1] *Targeted based on prior knowledge or alleged non-compliance.*

Figure 2: Packaging Components Tested by Major Material Type



B. Sample Preparation and Test Procedures

The test program was designed to screen packages for the presence of the four restricted metals. Testing was performed using a NITON XLt 797, which uses nondestructive energy-dispersive x-ray fluorescence technology to determine the elemental composition of samples. Although portable, all testing was performed with the analyzer docked into a stationary test stand with x-ray shielding as shown below. The instrument was connected by serial cable to a laptop computer that allowed remote, hands-free equipment operation.

The NITON XLt quantitatively measured up to 25 elements simultaneously. The analyzer displayed results as the concentration (ppm) level of each element detected in the sample, or indicated that the element was below the limit of detection (LOD). The reading for each element was accompanied by a range of uncertainty (i.e., +/- error) for the sample, which was expressed in ppm. The instrument also provides the x-ray spectrum for each analyzed sample. These measurements, along with the user data inputs, were stored in the NITON Data Transfer (NDT©) software, which cannot be modified, thus ensuring the integrity of test results. The test results were exported into a spreadsheet format for subsequent analysis.

XRF instrument in test stand

For this project, the concentration of the four metals (cadmium, lead, mercury, and chromium) was evaluated as pass (< 100 ppm or below the limit of detection) or fail ($\geq$ 100 ppm), taking into consideration the reported range of uncertainty for each metal. The test results were compiled and analyzed for trends in compliance and non-compliance with state toxics in packaging laws, including:

- Percentage of samples that passed and failed screening test; and
- Characteristics of failed samples, including product sector, material types and which restricted metals were detected above the screening threshold.

To prepare packaging samples for testing, the products were removed from the packages and individual packaging components were isolated to the extent possible. The accuracy of XRF measurements is affected by measurement duration, sample thickness, and sample positioning. Samples were either directly measured (non-destructive) or mechanically prepared (e.g., cut in squares and layered; folded) to meet a minimum sample thickness of 5 mm; and positioned directly over the detection window. The measurement time for all samples was 120 seconds, and duplicate readings, at a minimum, were taken for each packaging component. The concentration of each metal reported in subsequent sections of this report is the average of the measurements taken on individual packaging components.

Table 2: Limits of Detection in mg/kg for a Measurement Time of 120 seconds

Element	Matrix			
	Polyvinylchloride (PVC) with 2% Antimony	Polyethylene (PE) with 2% Antimony	PVC with 2% Bromine	PE with 2% Bromine
Cd	18	16	13	30
Pb	16	10	40	30
Hg	20	10	30	15
Cr	20	10	20	10

Source: NITON white paper, "Screening Materials for RoHS Compliance with the NITON XLt Analyzer, June 2005.

As shown in Table 2, the accuracy of the NITON instrument is sufficient to meet the objective of this screening project, that is, to identify samples that exceed the 100 ppm screening threshold for one of the restricted heavy metals. Table 2 shows the limits of detection (LOD) of the XRF analyzer for the target heavy metals with a 120-second measurement time for each sample. The samples contained two common plastics additives (antimony and bromine) that may interfere with XRF elemental analysis. For all the target substances, the LOD is between 10 to 40 mg/kg (ppm).

Equipment calibration and testing followed the standard operating procedures developed by NITON for the specified model and the detection of the four restricted heavy metals.[2] The plastics analysis mode was used for all samples, except metals. The metal alloy mode was used for metal samples, along with an alloy certified reference material. A polyethylene (PE) matrix reference sample that contained the target metals was used to verify the precision of the analyzer and validate the plastics mode test results. The following procedures were followed during every test session:

- Using NITON Standard Operating Procedures (SOP) (11 August 2005), calibrate the detector and test the multi-element reference sample. Take three 120-second measurements and average the results. Verify that the results are within the range of acceptable values for each reference metal as outlined in the SOP.
- Repeat the calibration and reference sample tests at a minimum of every two hours.
- Repeat the calibration and reference sample tests at the completion of the test session.

The equipment operator was certified in the safety and operation of the NITON instrument.

C. Outreach to the Packaging Supply Chain

The TPCH test program was designed to screen packages. Based on the results of the test program, companies selling or distributing packages that failed the screening test received notification of the test results. TPCH requested that these companies

[2] NITON Standard Operating Procedure: Screening of RoHS/WEEE elements in Plastics using the NITON Lt 794 X-Ray Fluorescence Analyzer Version 4.2 and higher, dated 11 Aug 2005. Similar SOPs were used for metallic samples. Additional information on this instrument and XRF technology can be found at www.niton.com.

certify compliance to state toxic in packaging laws and provide supporting analytic data, or notify TPCH of non-compliant packages and discontinue the sale and distribution of the package. The Certificate of Compliance required a company official to certify to the two requirements of state toxic in packaging laws: 1) the restricted heavy metals were not intentionally added; and 2) if any metals were incidentally present, that the sum of the concentration levels did not exceed 100 ppm.

A Compliance Review Committee comprised of five TPCH member states and TPCH program staff reviewed all company submissions and requested additional information as needed. From the outset, the objective of the outreach was to educate companies and their supply chain about toxics in packaging requirements and to work with them to eliminate the use of heavy metals. Companies that did not respond to the requests were referred to member states for appropriate action, including enforcement.

IV. Results

A. XRF Screening Results

TPCH screened 355 packaging samples between October 2005 and February 2006 for the presence of the four restricted metals (lead, cadmium, mercury, and hexavalent chromium) using a portable x-ray fluorescence analyzer. The packages included 570 packaging components such as bottles, bags, boxes, wraps, labels, caps, inks, and tape and ties. The packaging samples were selected to represent different packaging materials (aluminum, composites, glass, paper, plastic, and steel), product types, and packaging components, mostly in the retail sector, as described above.

The XRF analyzer detected concentrations of one or more of the restricted heavy metals in excess of 100 ppm in 57 packaging samples (see Table 3) -- 16% of all packages tested.[3] Of the total packaging components tested (570), 60 components, or 11%, failed the screening test. Some of these packages may have failed the screening test due to recycled content, which is exempt from toxics in packaging requirements in most states, or due to limitations of the XRF technology. For example, the XRF detects total chromium, not hexavalent chromium. When the six packages are removed that are likely to have failed the screening test as a result of total chromium or recycled content, 14% of the packages tested were likely to be in violation of state toxics in packaging requirements.

Cadmium and lead were the most frequently detected of the four regulated metals (Table 4). Historically, these metals were used in colorants and inks, and as stabilizers in PVC to retard the degradation of plastics exposed to heat and ultraviolet light.[4] While industry experts say these manufacturing practices have been largely

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[3] These results include packages that failed the screening test due to total chromium. The NITON XRF analyzer measures total chromium, not hexavalent chromium. Laboratory testing is needed to determine if the chromium is hexavalent chromium.
[4] International Cadmium Association, available at http://www.cadmium.org/app_stab.html, accessed on October 30, 2005.

Table 3: Summary of All Failed Packaging Samples (i.e., >100 ppm of Restricted Metals)

PKG #	Material Type	Product Category	Origin	Cd	Pb	Hg	Cr
1	Plastic	Shopping Bag	No information			243	
2a	Synthetic Fabric	Sporting Goods	China				459
2b	Synthetic Fabric	Sporting Goods	China				1,611
3a	PVC	Textiles	Pakistan	752			
3b	PVC	Textiles	Pakistan	901			
4	Plastic	Shopping Bag	No information		624		176
5	PVC	Textiles	China	561			
6	Plastic	Electrical/Electronic	Japan			123	
7	Glass	Beverage	Italy		147		442
8	Plastic	Shopping Bag	Singapore		243		
9	Plastic	Apparel	No information		148		
10	Glass	Beverage	Mexico			473	2,320
11	Plastic	Shopping Bag	No information		1,296		494
12	Plastic	Mailing/shipping	No information		226		
13	PVC	Textiles	Pakistan	393			
14	Glass	Beverage	USA				371
15	PVC	Cosmetics	China	274			
16	Plastic	Apparel	China		128		
17	PVC	Cosmetics	China		257		
18	PVC	Textiles	China	408			
19	Plastic	Shopping Bag	No information		9,334		2,548
20	PVC	Toys & Games	China	235			
21	PVC	Textiles	No information	430	404		
22	Plastic	Household Goods	China		270		
23	PVC	Toys & Games	China	504	137		
24	Alloy	Steel Strapping	No information		200		
25	PVC	Toys & Games	China	764			
26	PVC	Textiles	USA	529			
27	PVC	Textiles	China	350			
28	PVC	Textiles	China	449			
29	Plastic	Mailing/shipping	No information		8,889		1,166
30	Solder	Novelty	China		13,628		
31	Plastic	Retail Deli Bag	No Information		309		126
32	Plastic	Shopping Bag	USA			154	
33	PVC	Textiles	China	764	103		
34	PVC	Textiles	China	253			
35	PVC	Pet Supplies	China	483			
36	PVC	Cosmetics	China	502	115		
37	PVC	Pet Supplies	China	525			
38a	PVC	Electrical/Electronic	China		1,461		
38b	Unknown	Electrical/Electronic	Mexico-China				336
39	Plastic	Shopping Bag	No information		3,809		
40	Alloy	Personal Care	No information				300
41	Glass	Beverage	No information				581
42	Glass	Beverage	USA				555

Table 3 (cont.) Summary of All Failed Packaging Samples (> 100 ppm of Restricted Metals)

PKG #	Material Type	Product Category	Origin	Cd	Pb	Hg	Cr
43	PVC	Textiles	USA-Asia	269	417		
44	Glass	Beverage	Germany		127		700
45	PVC	Electrical/Electronic	China	321			
46	PVC	Textiles	China	330			
47	Plastic	Shopping Bag	No information		216		107
48	PVC	Pet Supplies	China	282			
49	PVC	Pet Supplies	China	439			
50	Plastic	Shopping Bag	No information				181
51	Plastic	Personal Care	USA	273			
52	Plastic	Mailing/shipping	No information			132	
53	Alloy	Steel Strapping	No information		400		
54	Alloy	Steel Strapping	No information		600		
55	Plastic	Shopping Bag	No information			135	
56	PVC	Textiles	Pakistan	988			
57	Glass	Beverage	Australia				241

Legend:
- (yellow) Confirmed non-compliance with toxics in packaging requirements
- (green) Industry documentation demonstrated that chromium is not likely to be hexavalent; or the recycled content exemption applies.

phased out in the U.S., this study found that packages of imported products continue to utilize raw materials that contain heavy metals. Twenty-four (24) of the 25 samples with elevated cadmium levels were flexible PVC packages, and of these, 23 were imported products. The average cadmium concentration detected in the samples that failed the screening test was 449 ppm while the average lead concentration was 1,740 ppm. Test results for two packages, a plastic shopping bag and a plastic mailing bag, indicated a lead content of almost 1% of the total package weight (9,334 ppm and 8,889 ppm, respectively).

Table 4: Summary of Results >100 ppm by Restricted Heavy Metal

Restricted Metal	Samples with >100 ppm Detected	Mean (ppm)	Median (ppm)	Range (ppm)	Comments
Cadmium	25	479	436	235 - 988	24 of 25 samples were flexible PVC; at least 21 of the 25 were imported
Lead	25	1740	270	103 - 13,628	
Mercury	6	210	145	123 - 473	
Chromium[1]	18	706	451	107 – 2,548	

[1] *XRF measures total chromium, not hexavalent chromium (Cr+6), which is the regulated metal.*

There were two types of packaging that dominated the samples failing the screening test (that is, one or more of the restricted heavy metals detected at a concentration > 100 ppm):

1) **Flexible polyvinylchloride (PVC) packages**. This "heavy-duty" plastic material is frequently used to package textiles, cosmetics, inexpensive toys, and pet supplies. Examples of the packages tested include zipper bags used to package bedding and other home furnishings, such as comforters, and the plastic pouches used to package pet toys and chews. As shown in Table 5, 25 of the 41 flexible PVC packages tested (61%) exceeded the screening limit of 100 ppm, due to excessive levels of cadmium and/or lead. Almost all of the products in flexible PVC packages were imported from Asia (at least 35 of 41), according to the product label. Interestingly, all PVC "blister packs" and clamshells, which are semi-rigid and in this study mostly imported from Asia as well, passed the screening tests. Blister packs and clamshells were used to package a variety of products, from office supplies and novelty items to hardware and toys.

The screening results indicated a high prevalence (about 80%) of the restricted heavy metals in flexible PVC packaging from some product sectors. For example, 13 of the 16 flexible PVC bags/pouches for textile and home furnishings products (e.g., mattress pads, comforters, tablecloths, shower curtains) had elevated levels of cadmium and/or lead, as shown in Table 6. In the pet supply sector, four of the five flexible PVC pouches tested with the XRF contained cadmium greater than 100 ppm.

Table 5: Analysis of All Flexible PVC Samples

		PASS < 100 ppm for all metals		FAIL > 100 ppm for any one metal	
	Total Samples	**Samples**	**%**	**Samples**	**%**
Packages	41	16	39.1	25	60.9
Packaging Components[1]	45	19	42.2	26	57.8
Product Origin[2]	47 [3,4]				
Asia	39	15	38.5	24	61.5
U.S.	5	3	0.6	2	0.4
Mexico	1	1		0	
No information	2	1		1	

[1] *Four packaging samples had two flexible PVC packaging components.*
[2] *Package origin is assumed to be the same as product origin unless otherwise specified.*
[3] *Total number of samples is based on total number of packaging components since three packages had one component that passed and one that failed.*
[4] *The countries of origin add up to 47 (not 45) because one product was labeled "Made in USA-Asia" and the package had one component that passed and one component that failed. This package was counted towards both countries of origin under pass and fail.*

Table 6: Samples Failing for One or More Metals > 100 ppm by Product Category

Product Category	Total Samples	Samples > 100 ppm	% of Samples >100ppm	Comments on Samples > 100 ppm
Textiles	16	13	81.3	All samples >100 ppm were flexible PVC bags
Shopping Bags - All - Plastic - Paper	 68 60 8	 10 10 0	 14.7 16.7 0	Inks & colorants suspected; all samples >100ppm were plastic bags
Beverage	24	7	29.2	All samples >100 ppm were glass; six of seven samples are likely to be "false positives" due to total chromium reading, which is probably not hexavalent chromium[1]
Cosmetic/personal care	9	5	55.6	Three of five samples >100 ppm were flexible PVC bags
Pet Supplies - All - Flexible PVC	6 5	4 4	66.7 80.0	The four samples >100 ppm were flexible PVC
Toys/Games	19	3	15.8	All samples >100 ppm were inexpensive toys in flexible PVC packaging
Electrical/Electronic	14	3	21.4	Samples >100 ppm were various materials (solder, plastic tape, & flexible PVC bag)
Mailing/Shipping	14	3	21.4	Inks & colorants suspected; all samples >100 ppm were plastic bags
Steel Strapping	9	3	33.3	
Other[2]		5		

[1] Industry documentation demonstrated that chromium is not likely to be hexavalent.
[2] *Product categories included apparel, novelty products, household goods, and sporting goods.*

2) **Inks and colorants used on plastic shopping/mailing bags.** Lead was the most frequently detected restricted heavy metal in shopping bags that failed the screening tests, but mercury and chromium were also detected in some samples.[5] Similar to the flexible PVC samples, the elevated levels of restricted metals appear to be largely from imported products, where solvent-based inks that contain the heavy metals are still used.[6]

[5] The XRF measures total chromium and does not differentiate between types of chromium (e.g., trivalent or hexavalent.) Only hexavalent chromium is restricted by state toxics in packaging laws.
[6] Countries of origin statistics are not available for shopping and mailing bags since this information was not often printed on the package.

B. Company Responses

The TPCH test program was designed to screen packages for the presence of the four restricted metals. Based on the results of the test program, companies selling or distributing packages that failed the screening test received notification of the test results. Companies were required to certify compliance with state toxics in packaging laws and to provide supporting analytic data, or to notify TPCH of non-compliant packages and discontinue the sale and distribution of the package. A total of 44 companies were notified about 52 packages that failed the screening test. Several companies were notified about two or more packages. In five instances, the companies could not be located so no notifications were sent.

Working with companies to determine the compliance status of the packages was more challenging than anticipated. Table 7 summarizes companies' responses overall and by packaging type. Companies verified the TPCH test results and acknowledged that their packages were not in compliance with state toxics in packaging requirements for only 15% (8 of 52 failure notifications) of packages. Companies claimed compliance and submitted supporting documentation for almost 70% of the packages that failed the TPCH screening tests. Companies made no claims for the remaining 8 packages (15% of failure notifications), most often citing that the product was discontinued and therefore the packaging was not available for testing.

There are several possible explanations for the discrepancy between the screening test results and company claims. First, suppliers or raw materials changed and the package tested was not manufactured with the same material as the TPCH test package. Second, in some cases, TPCH suspects that conventional laboratory

Table 7: Company Compliance Claims for Failing XRF Results (> 100 ppm)

		Company Claim					
		Compliance		**Non-compliance**		**No Claim[1]**	
	Total Samples	**Samples**	**%**	**Samples**	**%**	**Samples**	**%**
All Packages	52	36	69.2	8	15.4	8	15.4
By Package Type							
Flexible PVC, including bag, pouches, twist ties	24	14	58.3	4	16.7	6	25.0
Inks/colorants on plastic bags, e.g., shopping/mailing bags	17	13	76.5	3	17.6	1	5.9
Glass bottles[2]	7	7	100.0	0	0.0	0	0.0
Other[3]	4	2		1		1	

[1]For six packages, the company claimed that the product was no longer available for testing. For the three remaining packages, the companies have not submitted documentation.
[2] Glass bottles without label except one sample where the vitrified label was tested.
[3] Other package types included rigid non-PVC plastic, printed wire board with lead solder, and plastic tape.

preparation methods are insufficient to adequately digest the packaging sample, resulting in the measurement of “recoverable” metals in the solution, not a true total concentration of the restricted metals in the original matrix. (See further discussion of test methods in Box 3 on page 20.) Third, the detection of heavy metals was attributable to recycled content, which is exempt from toxics in packaging requirements in some states. Finally, the XRF technology has its limitations as well. For example, XRF detects total chromium, not hexavalent chromium, which might have attributed to some false positives for heavy metal concentrations. However, if the packages that are likely to have failed the screening tests due to total chromium (not hexavalent) and the recycled content exemption are removed from the sample size, then the confirmation of non-compliance is still only 17% of the packages tested.

C. Additional Test Results

After receiving numerous analytic test results from companies that refuted TPCH XRF measurements indicating violations of state laws, TPCH sent several samples to the California Department of Toxic Substance Control’s (DTSC) analytical laboratory for further testing and validation of TPCH test results. California DTSC analyzed the samples using XRF technology, as well as Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES), a commonly used laboratory analytical technique for determining metals concentration in soil samples.[7] California DTSC then forwarded these same samples to its XRF vendor, Oxford Instruments, for further confirmation of XRF test measurements.

Table 8 summarizes the XRF test results obtained by TPCH, California DTSC, and Oxford Instruments using portable and bench-top energy-dispersive XRF technology, as well as the measurements obtained by California DTSC using ICP-AES. The XRF measurements obtained by the three organizations, using three different XRF models manufactured by two companies, indicate that the packages are in violation of state restrictions on heavy metals in packaging. The variation in the metal concentrations detected by the three organizations is likely due to such factors as sample thickness and the location where the measurement was taken on the sample,[8] rather than the accuracy of the XRF technology.

The ICP-AES test results obtained by California DTSC stand in stark contrast to the collective XRF results. The ICP-AES results are at least an order of magnitude less than the XRF results. The ICP-AES only detected metal concentrations over 100 ppm when the XRF results indicated concentrations greater than 1,000 ppm. Based on these ICP-AES results, two of the three samples would be in violation of state laws, while one

[7] Samples were digested with 1:1 HNO3 (and 30% H2O2, and 1:1 HCl, if applicable) over a hot plate. Digests were cooled, filtered and made to final volume with deionized H2O (EPA SW-846 3050B). Metal analysis of the digests was by ICP-AES (EPA SW-846 6010B).

[8] XRF analysis requires minimal sample preparation and is non-destructive. Measurements are therefore subject to the variation in metal concentration found in the manufactured package; for example, the ink to resin ratio on different areas of a shopping bag.

Table 8: Comparison of California XRF and ICP Results (ppm)

		XRF			ICP-AES
Sample Description	**Restricted Metal**	**TPCH NITON XLt 797**	**CA DTSC[1] Oxford X-Met 3000TX**	**Oxford Instruments XGT1000**	**CA DTSC**
Shopping Bag 1	**Pb**	1,296	718	1,163	138
	Cr	494	279	161	30.2
Shopping Bag 2	**Pb**	9,334	12,752	9,203	322
	Cr	2,548	2,188	1,617	71.6
Textile Bag – Flexible PVC	**Cd**	430	360	591	20.4
	Pb	404	432	565	19.2

1 *CA DTSC XRF testing was performed using Oxford Instrument, X-MET 3000TX; results are the average of two readings; shopping bag samples were 32 layers thick (2-3 mm); the textile bag was 8 layers thick (1mm).*

2 *Oxford Instruments tested the samples using a bench top energy-dispersive x-ray fluorescence spectrometer, Model XGT 1000WR-Type II.*

sample (the textile bag) would appear to be in compliance with the 100 ppm limit of state toxics in packaging laws. Indeed, using ICP-AES, most of the TPCH samples shown in Table 3 (page 13), would have "passed" rather than "failed" the compliance test, since the median concentration of the restricted metals in all the TPCH samples that failed the XRF screening test was below 500 ppm, as summarized previously in Table 4 (page 14).

The Connecticut Department of Environmental Protection obtained similar results when it submitted four different TPCH flexible PVC samples to an accredited contract laboratory for analysis. The instructions given to the laboratory were to analyze for "total metals concentration" in the samples. Table 9 summarizes the results of the laboratory tests compared to the TPCH XRF measurements.

Given the results obtained by both California and Connecticut using conventional laboratory test methods, it is not surprising that many of the companies that received

Table 9: Connecticut Laboratory Results Compared to TPCH XRF Analysis (ppm)

Description of Flexible PVC Sample	**Contract Laboratory Results / TPCH XRF Results**			
	Cadmium (ppm)	**Chromium total (ppm)**	**Mercury (ppm)**	**Lead (ppm)**
Toy Bag	21.2 / 500	<0.50 / ND	<0.10 / ND	11.8 / 137
Small Electrical Appliance Bag	17.3 / 320	<0.50 / ND	<0.10 / ND	<0.50 / ND
Textile Bag 1	31.8 / 990	<0.50 / ND	<0.10 / ND	<0.50 / ND
Textile Bag 2	31.2 / 528	<0.50 / ND	<0.10 / ND	<0.50 / ND

Box 3: Conventional Test Methods

"Conventional" laboratory techniques refer to the traditional methods that environmental laboratories have been using to test for "total" metals in soils, sediment, and other solid samples for waste characterization and site characterization purposes. Traditionally, this has been EPA SW-846 Method 3050B. The scope and application of Method 3050B indicates that it is not a total digestion technique for most metals. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available." It further states that if absolute total digestion is required, use Method 3052.

The scope and application of Method 3052 indicates that it is applicable to the microwave assisted acid digestion of organic matrices and other complex matrices and that the technique is not appropriate for regulatory applications that require the use of leachate preparations (such as Method 3050). It further states that Method 3052 is appropriate for those applications requiring a total decomposition in response to a regulation that requires total sample decomposition.

Preliminary analysis by California DTSC of a limited number of additional samples using more rigorous sample digestion (such as microwave techniques) seemed to liberate more of the metals of interest from hard-to-digest matrices (such as plastics), resulting in higher concentrations in the reported test results. California DTSC plans to pursue a study to determine the validity of this hypothesis, and evaluate appropriate sample preparation and analysis techniques to determine compliance with state toxics in packaging laws.

failure notifications from TPCH claimed compliance based on independent laboratory test results. TPCH and its member states, in consultation with experts and laboratory personnel, suspect that the destructive preparation methods used to prepare the samples for subsequent analysis contributed to the discrepancy, resulting in incomplete digestion of the sample, and therefore, incomplete recovery of the metals actually present in the sample. For a brief discussion of conventional laboratory test methods, see Box 3, above.

V. Discussion and Next Steps

A. Why Are Toxics Detected in Packaging Now?

The packaging industry is constantly changing as new technologies are introduced and as companies and manufacturing locations shift. There is evidence that suggests these changes may lead to greater levels of non-compliance. For example, electronic components that contain circuit boards are on the rise as part of packaging. The TPCH recently found two such packages with a circuit board containing lead solder that was used to power a blinking light to draw consumer attention. In another example, lead crystals are being used to decorate water bottles that are marketed as a "fashion accessory." In both cases, the packaging component is a crossover technology from another industry that may not be familiar with packaging regulations.

Increasingly, products sold in the U.S. are imported from other countries. As manufacturing has moved off shore, state toxics in packaging requirements may not be transferred, properly translated, or understood in other countries. According to shopping bag suppliers, for example, U.S. companies have moved away from solvent-based inks to water-based inks, in part due to the regulation of air emissions and in part due to state toxics in packaging requirements. The use of solvent-based inks, according to these same suppliers and supported by the findings of this study, is still commonplace in Asian countries.

TPCH outreach to the packaging supply chain also revealed complacency among manufacturers and distributors. Until recently, toxics in packaging laws were not aggressively enforced. Several companies selling shopping bags acknowledged to TPCH staff that toxics in packaging requirements had "fallen off the radar screen" due to a lack of state attention to and enforcement of the laws. With ever-increasing pressure to reduce costs to remain competitive and a supply of less expensive imports, there was little incentive to comply with toxics in packaging requirements.

B. Quality and Compliance Assurance

The TPCH screening project did encounter manufacturers who thought they were taking the necessary steps to ensure the quality of their supply, including compliance with state toxics in packaging laws. These companies included toxics in packaging requirements in their purchasing specifications and terms and conditions, as well as required certification of the supply. Upon inspection and testing, however, they were surprised to learn that a package or packaging component was not in compliance with state laws. Simply stating material or packaging specifications, taking a supplier's assurances, or requiring a Certificate of Compliance was not enough. TPCH learned this lesson first-hand as well, and as a result, recommends additional due diligence steps for manufacturers and distributors as discussed in the conclusions section below.

C. Test Methodologies

There were significant differences between the screening results obtained by the TPCH using the NITON XRF analyzer and the tests performed by laboratories that used conventional laboratory methods, such as SW-846 test methods. TPCH undertook preliminary comparison testing to learn why different tests were yielding different results. Testing using XRF technology at member state California DTSC and Oxford Instruments confirmed the levels of metals detected during the TPCH screening process.

Tests by California DTSC on the same samples but using conventional laboratory analysis techniques (ICP-AES) did not find similarly high levels. It may be that conventional laboratory methods insufficiently digest the sample, so that results for the regulated metals are significantly lower. The California DTSC is designing a follow-up study to further compare different sample preparation and test methods for determining total concentration of the restricted metals in packaging materials. This project will provide guidance to the regulated community on appropriate test methods.

D. Understanding U.S. Toxics in Packaging Requirements as Compared to the European Union Requirements

Finally, based on company queries and submissions to the TPCH, it seems that the regulated community may be unaware of and/or misunderstands the Model Legislation and U.S. state toxics in packaging requirements based on the Model. Legislation in the U.S. has two distinct requirements: 1) no intentional introduction of any amount of the four restricted metals; and 2) the sum of the incidental presence (that is, not intentional) of the four metals cannot exceed 100 ppm. These requirements are in contrast to the European Union Packaging Directive, which does not have the “no intentional introduction” requirement. Rather, the European Union only restricts the presence of the same four heavy metals to a 100 ppm maximum allowable concentration in any package or packaging component. From the numerous company submissions reviewed by TPCH member states over the course of this project, it became apparent that many companies thought that the Model Legislation and U.S. state laws allow the use of heavy metals as long as the concentration does not exceed 100 ppm.

If companies are using a 100 ppm threshold for compliance and if the test methods currently used to measure heavy metal concentration are not adequate for detecting these substances in some materials, then the amount of heavy metals entering the waste stream and subsequently the environment could be higher than expected. This possibility makes it even more important that TPCH and its member states conduct education and outreach to the packaging supply chain to emphasize the “no intentional use” requirements of state toxics in packaging laws.

VI. Conclusions

The results of this project clearly demonstrate that toxic heavy metals in packaging are still an issue almost 15 years after many states enacted laws prohibiting these substances in packaging. The greatest threat to the quality of packaging materials and compliance with state laws appears to be packages of imported products. Given the amount and short-lived nature of packaging, lead and cadmium, in particular, are being continuously fed into the solid waste and recycling streams via discarded packaging, and potentially released into the environment.

Companies selling or distributing packaging materials and packaged products need to work with and educate their suppliers to ensure compliance with state restrictions on the use of heavy metals in packaging. Packaging specifications, written compliance certification, or the “word” of suppliers is insufficient to document or ensure compliance, based on the experience of TPCH over the course of this project. At a minimum, the supply chain should require analytic test results from the supplier prior to purchasing the packaging material or packaged product. Additionally, a quality assurance program should include periodic “spot checks” to determine if heavy metals are present in the packaging in order to verify the validity of supplier claims.

TPCH and its member states support the use of XRF technology to screen packages and packaging materials for compliance with state requirements. Further work is needed to define appropriate analytical laboratory techniques suitable for determining heavy metal concentration in packaging materials.

TPCH and its member states are committed to increased outreach and education of the regulated community about toxics in packaging and state requirements. TPCH encourages additional states that have toxics in packaging laws to become members of the Clearinghouse in order to broaden the impact of outreach efforts, and to take advantage of the efficiencies embodied in sharing resources towards a common goal. States without legislation are also encouraged to adopt the Model Toxics in Packaging Legislation to increase the overall effectiveness of the legislation, and to avoid becoming a dumping ground for packaging that contains heavy metals and are illegal to sell in states with toxics in packaging laws.

Finally, TPCH, in conjunction with member states, plans to conduct compliance screening programs in the future to detect compliance trends with state toxics in packaging requirements, and assess the impact of its outreach efforts.

