

Enhancing Trilateral Understanding of Flame Retardants and Their Use in Manufactured Items

**Analysis of Select Flame Retardants Contained in
Office and Household Furniture**

Summary Report – Phase II

December 2015



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List of Abbreviations and Acronyms

Br	Bromine
CEC	Commission for Environmental Cooperation
Cl	Chlorine
DBDPE	Decabromobiphenyl ethane; or 1,1'-(1,2-Ethanediy)bis[2,3,4,5,6-pentabromobenzene]
EC	Environment Canada
g	gram(s)
GCMS	gas chromatography–mass spectrometry
HBCD	Hexabromocyclododecane
kg	kilogram(s) (1000 grams, or 1×10^3 grams)
ng	nanogram(s) (or 1×10^{-9} grams)
P	Phosphorus
PIP	Phenol, isopropylated, phosphate (3:1)
ppm	parts per million
QAPP	quality assurance project plan
Semarnat	<i>Secretaría de Medio Ambiente y Recursos Naturales</i> (Ministry of Environment and Natural Resources [Mexico])
TBB	2,3,4,5-Tetrabromobenzoic acid 2-ethylhexylester
TBE	1,1'-[1,2-Ethane-diylbis(oxy)]bis[2,4,6-tribromobenzene]; or 1,2-Bis(2,4,6-tribromophenoxy) ethane; or 1,1,2,2-Tetrabromoethane
TBEP	Ethanol, 2-butoxy-, phosphate (3:1); or Tri(2-butoxyethyl) phosphate
TBPH	1,2-Benzenedicarboxylic acid, 3,4,5,6-Tetrabromo-bis(2-ethylhexyl) ester; or Bis(2-ethylhexyl) tetrabromophthalate
TCEP	Tris(2-chloroethyl)phosphate
TCP	Phosphoric acid, tris(methyphenyl) ester
TCPP	2-Propanol, 1-chloro-phosphate, mixture of isomers (contains 6145-73-9)
TCPP isomer	1-Propanol, 2-chloro-, phosphate (isomer of TCPP)
TDCPP	2-Propanol, 1,3-dichloro-, phosphate (3:1)
TEP	Triethyl phosphate; or Phosphoric acid, triethyl ester
TIC	total ion chromatogram
TPP	triphenyl phosphate
XRF	X-ray fluorescence

Abstract

Canada, Mexico, and the United States, in their roles as Parties to the Commission for Environmental Cooperation (CEC), have taken a common interest in carrying out a case study to better understand the presence of 16 emerging flame retardants in consumer products. As a result, a project entitled Enhancing Trilateral Understanding of Flame Retardants and Their Use in Manufactured Items (CEC 2014) is being supported under the CEC's Operational Plan 2013–2014, through a project team consisting of representative members from the CEC Secretariat, Health Canada (HC), Environment Canada (EC), *Instituto Nacional de Ecología y Cambio Climático* (INECC) under the *Secretaría de Medio Ambiente y Recursos Naturales* (Semarnat), and the United States Environmental Protection Agency (EPA). The results of Phase 2 of this project are discussed in this report. These results cover the targeted product sampling and analysis of a select list of emerging flame retardants. Phase 2 was carried out in two parts: 1) a screening technique was used to determine the presence of bromine, chlorine, and phosphorus, elements which could indicate the presence of one of the 16 flame retardants of interest, and 2) a quantitative analysis of samples taken from the products was performed. The results of the XRF screening and GC/MS laboratory analyses are discussed in terms of general trends across product type, sample category, and country.

Introduction

In response to an increasing amount of scientific data, international communities are recognizing that certain chemicals contained within a variety of consumer products can lead to detrimental effects on the environment and human health. These chemicals are introduced into consumer products for a number of reasons, depending upon the product use. Flame retardants are of particular interest because several studies have identified adverse health effects for these substances. The fact that they have been found in a variety of locations, including the environment (Segev 2009), wildlife (Hale 2001), and humans (Hooper 2000), has only increased concerns.

Canada, Mexico, and the United States, as Parties to the Commission for Environmental Cooperation (CEC), have a common interest in better understanding the degree to which consumer products may be treated with flame retardants. Of particular interest are several “emerging” flame retardants that have been developed as drop-in substitutes for older or restricted flame retardants.

A project entitled *Enhancing Trilateral Understanding of Flame Retardants and Their Use in Manufactured Items* (CEC 2014) was being supported under the CEC’s Operational Plan 2013–2014. The Project Team consisted of representative members from the CEC Secretariat, Health Canada (HC), Environment Canada (EC), *Instituto Nacional de Ecología y Cambio Climático* (INECC) under the *Secretaría de Medio Ambiente y Recursos Naturales* (Semarnat), and the United States Environmental Protection Agency (EPA).

The project was constructed and executed in two phases. Phase 1 was contracted to Eastern Research Group, Inc. (ERG) (CEC 2015). The two primary goals of this phase were to 1) identify products containing at least one from an initial list of 46 flame retardants of common interest, and to 2) obtain information and understand perspectives on the future use of flame retardants in foam products. A third objective was to gather information on the levels at which flame retardants are claimed to be included in various products. A final goal was to develop a scoping document to recommend items to be purchased and evaluated for the presence of flame retardants, using a sampling plan with generally known analytical methodologies. For Phase 2, the CEC contracted Intertek to perform targeted product sampling and analysis for the identification and quantitation of emerging flame retardants from a select list based on the outcome of the work performed by ERG. The focused list of 16 flame retardants was recommended through collaboration between the CEC and its member representatives. The remainder of this summary discusses the outcomes of Phase 2 of *Enhancing Trilateral Understanding of Flame Retardants and Their Use in Manufactured Items*.

Study Objectives

The objectives of Phase 2 were to:

1. develop and implement a quality assurance project plan (QAPP);
2. purchase/acquire manufactured items commonly used by consumers;
3. prepare samples of these manufactured items for testing; and
4. determine the presence and concentration of selected flame retardants in specific manufactured items, through qualitative and quantitative analyses as required.

The flame retardant testing (Objective 4) took place in two parts. Initially, a screening technique—X-ray fluorescence (XRF)—was used to determine the presence of bromine (Br), chlorine (Cl), and phosphorus (P), which are elements that could indicate the presence of one of the 16 flame retardants of interest (see Table 1 below for the list of flame retardants). In the second part, a quantitative analysis was performed for samples exhibiting Br, Cl, or P concentrations above a threshold. The

quantitative analysis provided both identification and measurements of concentration of the flame retardants present in samples taken from the products.

Table 1. Flame Retardants Evaluated in Phase 2

CAS RN*	Acronym	Flame Retardant—Chemical Name
13674-84-5	TCPP	2-Propanol, 1-chloro-, phosphate; (TCPP); or 1-Propanol, 2-chloro-, phosphate; Mixture of isomers (contains 6145-73-9)
6145-73-9	TCPP ISOMER	1-Propanol, 2-chloro-phosphate (isomer of TCPP)
13674-87-8	TDCPP	2-Propanol, 1,3-dichlorophosphate
26040-51-7	TBPH	1,2-Benzenedicarboxylic acid, 3,4,5,6-tetrabromo-bis(2-ethylhexyl) ester; or Bis(2-ethylhexyl) tetrabromophthalate
84852-53-9	DBDPE (DBE-209)	1,1'-(1,2-Ethanediy)bis[2,3,4,5,6-pentabromobenzene]; or Decabromodiphenyl ethane
183658-27-7	TBB	2-Ethylhexylester 2,3,4,5-tetrabromobenzoate; or 2,3,4,5-Tetrabromobenzoic acid 2-ethylhexyl ester
78-40-0	TEP	Triethyl phosphate; or Phosphoric acid, triethyl ester
78-51-3	TBEP	Ethanol, 2-butoxy-, phosphate (3:1)
1330-78-5	TCP	Phosphoric acid, tris(methylphenyl) ester; or tricresyl phosphate
26446-73-1		Phosphoric acid, bis(methylphenyl) phenyl ester
68937-41-7	PIP	Phenol, isopropylated, phosphate (3:1)
77098-07-8		1,2-Benzenedicarboxylic acid, 3,4,5,6-tetrabromo-; 3,4,5,6-Tetrabromo-1,2-benzenedicarboxylic acid, mixed esters with diethylene glycol and propylene glycol; Tetrabromophthalate Diol (TBPA Diol, mixed esters)
20566-35-2		2-(2-Hydroxyethoxy)ethyl 2-hydroxypropyl 3,4,5,6-tetrabromobenzenedicarboxylate; 2-(2-Hydroxyethoxy)ethyl 2-hydroxypropyl ester; 2-(2-Hydroxyethoxy)ethyl 2-hydroxypropyl 3,4,5,6-tetrabromophthalate
115-96-8	TCEP	Tris(2-chloroethyl) phosphate
37853-59-1	TBE	1,1'-[1,2-Ethane-diylbis(oxy)]bis[2,4,6-tribromobenzene]; or 1,2-Bis(2,4,6-tribromophenoxy) ethane
25637-99-4	HBCD	Hexabromocyclododecane
115-86-6	TPP	Triphenyl phosphate

*Chemical Abstract Service Registry Number.

Methodologies

Intertek selected an average of 45 furniture products from each country (Canada, Mexico, and the US). The products were purchased between December 2014 and April 2015 from at least three major retailers in each country, and for each country approximately 10 office chairs and 35 upholstered products (i.e., sofas and/or chairs) were chosen. Products selected were in the low-to-moderate end of the price spectrum. The products purchased in this study were described as specifically as possible. This included images of the items' physical appearances, and describing uses, trade names, and retailer information. According to the available information on point of origin, there were 54 products from China, 4 from Asia, 6 from Canada, and 5 from the US. For another 63 products, the country of export or manufacture could not be determined. Due to this overall lack of sufficient information on country of origin, as well as the small sample size, no correlations were drawn between origin and quantitative results.

Each product (e.g., sofa, chair) was photographed in its purchased state, then taken apart into homogeneous constituent samples for screening (see Table 2 for definitions of sample categories), each of which was photographed.

Table 2. Sample Category Descriptions

Sample Category	Description
Fabric	Flexible material, typically woven, consisting of a network of natural or synthetic fibers
Foam	Substance that is formed by trapping pockets of gas in a solid
Other	Includes leather, imitation leather, plastics, straps, and piping
Padding	Fibrous material, typically formed in sheets from materials that include cotton, wool, and/or synthetic fibers
Stuffing	Loose, fibrous material used for cushioning

Screening Experiments Using X-ray Fluorescence (XRF)

An Olympus Delta-model portable XRF analyzer was used for screening all samples. If the results of the screening showed that the concentrations of Cl, P, or Br were above the screening limit of 300 parts per million (ppm), the samples were carried forward for more-specific identification and quantitation of the individual flame retardants. If the samples did not exhibit Cl, Br, and P concentrations above 300 ppm, then no further analyses were carried out for these samples.

Laboratory Analysis Using Gas Chromatography–Mass Spectrometry (GC/MS)

Samples that required further analysis were prepared using an organic solvent extraction, followed by flame retardant analysis using gas chromatography–mass spectrometry (GC/MS). Samples were cut into small pieces, placed in a vial with methylene chloride, and extracted by sonication for 1 hour at a temperature of 50–55 °C. After sonication, the samples were allowed to cool to room temperature, filtered to remove particulates, and analyzed using an Agilent 6890N GC System coupled to an Agilent 5975 Mass Selective Detector.

Summary of Results

XRF Screening

Of the total of 132 products purchased for testing, each one ended up being flagged in the screening process because the concentration of Cl, P, and/or Br was above the threshold limit of 300 ppm in at least one sample (fabric, foam, padding, stuffing, other) collected from it. A comparison of the data shows that the largest contributing factor identifying a product as possibly containing a flame retardant is the presence of chlorinated compounds. In fact, all 132 products contained at least one sample with Cl concentrations greater than 300 ppm (>300 ppm), while on average, about 40% of the products tested contained at least one sample with >300 ppm P, and 39% of the products tested contained at least one sample with >300 ppm Br. Table 3 gives a summary of the XRF screening results from all the products obtained from Canada, Mexico, and the US.

Table 3. Summary of Overall XRF Screening Results for Products across All Countries

Product	Number of Products	Number of Products with at Least One Sample* with Cl, P, and/or Br >300 ppm	Number of Products with Cl >300 ppm	Number of Products with P >300 ppm	Number of Products with Br >300 ppm
Chair	47	47 (100%)	47 (100%)	23 (49%)	16 (34%)
Office Chair	33	33 (100%)	33 (100%)	15 (45%)	12 (36%)
Ottoman	2	2 (100%)	2 (100%)	1 (50%)	0 (0%)
Sofa	50	50 (100%)	50 (100%)	16 (32%)	24 (48%)
Total	132	132 (100%)	132 (100%)	55 (42%)	52 (39%)

*Sample = fabric, foam, padding, stuffing, other.

A total of 717 samples were created from the 132 products purchased. Of these 717 samples, 559 (or 78%) exhibited concentrations of Cl, P, and/or Br above the XRF screening limit of 300 ppm, and were therefore brought forward for quantitative analysis. Table 4 gives the number and percentage of samples that showed concentrations of one of at least one of the substances above the 300 ppm limit, by country. The results indicate that the percentages of samples showing concentrations exceeding the 300 ppm screening limit were fairly equal among the products purchased in Canada, Mexico, and the US.

Table 4. Summary of XRF Screening Results for Each Country

Country	Total Number of Samples	Number of Samples with Cl, P or Br >300 ppm	% of Samples with Cl, P or Br > 300 ppm
Canada	245	193	79%
Mexico	177	138	78%
US	295	228	77%
Total	717	559	78%

An alternative examination of the XRF screening sample results is presented in Table 5, where the element-specific XRF results are shown for each sample type (e.g., fabric, foam), across all countries. The results show that fabrics and foams produced the greatest number of samples with concentrations of Cl, P, and/or Br above 300 ppm. The fabric and foam categories accounted for 73% of the results above 300 ppm, largely due to the presence of high concentrations of Cl. The number of samples with Cl above 300 ppm (552) was considerably higher than that with P (80) or Br (72) above 300 ppm. Interestingly, the number of samples with P above 300 ppm was highest for foams, while the number of samples with Br above 300 ppm was highest for fabrics.

Table 5. Detailed XRF Screening Results across All Countries

Sample Category	Number of Samples	Number of Samples with Cl, P or Br >300 ppm	Number of Samples with Cl >300 ppm	Number of Samples with P >300 ppm	Number of Samples with Br >300 ppm
Fabric	261	207	206	2	49
Foam	246	203	198	74	9
Other	65	54	53	2	10
Padding	67	44	44	2	1
Stuffing	78	51	51	0	3
Total	717	559	552	80	72

Flame Retardant Results

Product-related trends, based on country

Of the 16 original flame retardants targeted in this study, six (TCPP, TDCPP, TPP, TBEP, TBPH, and TBB) were detected in samples, based on GC/MS analysis following XRF screening. For North America as a whole, close to half (i.e., 61) of the 132 products tested contained at least one of the 16 flame retardants, as shown in Table 6. These flame retardants were found in 53% of chairs, 45% of office chairs, 100% of ottomans, and 38% of sofas. On a country-by-country basis, 22% (29) of the total products from Canada contained flame retardants, in comparison to 17% (22) from the US, and

8% (10) from Mexico. The presence of flame retardants according to product type (e.g., chairs versus sofas) also varied by country. Canada exhibited the highest number of sofas and office chairs with detected flame retardants, while Mexico exhibited the least. The number of chairs sourced from the US which contained flame retardants was nearly double that of either Mexico or Canada. The number of ottomans with detected flame retardants was equal in Mexico and the US.

Table 6. Percentage of Products Containing Flame Retardants, for North America as a Whole and for Canada, Mexico, and the US Individually

Product Type	Total Number of Products Tested	Number of Products Containing Flame Retardants			
		North America	Canada	Mexico	US
Chair	47	25 (53%)	7 (15%)	6 (13%)	12 (26%)
Office Chair	33	15 (45%)	10 (30%)	2 (6%)	3 (9%)
Ottoman	2	2 (100%)	0 (0%)	1 (50%)	1 (50%)
Sofa	50	19 (38%)	12 (24%)	1 (2%)	6 (12%)
Total	132	61 (46%)	29 (22%)	10 (8%)	22 (17%)

Sample type–related trends, based on country

Organophosphate flame retardants were the dominant flame retardants detected in the samples. TCPP was the most frequently detected flame retardant measured in samples collected from products across all countries. As shown in Table 7, TCPP was most frequently detected in samples from Canada, followed by the US, and then lastly Mexico. TDCPP was the second most common flame retardant, with samples from Mexico exhibiting higher detection frequencies than those from Canada or the US. TPP was the third most frequently detected flame retardant, with samples from Canada exhibiting the highest detection frequencies. TBPH, TBB, and TBEP each were found in samples from Canada but at relatively low frequencies.

Table 7. Number and Detection Frequency of Samples Containing TCPP, TDCPP, or TPP, across All Countries

Samples	Canada	Mexico	US
Containing > 300 ppm Cl, Br, and/or P (by XRF)	193	138	228
Containing TCPP	45 (23%)	19 (14%)	43 (19%)
Containing TDCPP	8 (4%)	11 (8%)	5 (2%)
Containing TPP	9 (5%)	1 (0.1%)	2 (1%)
Containing TBPH	1 (<0.5%)	0 (0%)	0 (0%)
Containing TBB	1 (<0.5%)	0 (0%)	0 (0%)
Containing TBEP	2 (1%)	0 (0%)	0 (0%)
Total	717		

Distribution of flame retardants in samples

TCPP was present in a total of 54 products (e.g., chairs, sofas) from across North America. Among samples collected from these products, TCPP was most frequently detected in the foam samples—91%—and was detected in 24% of the fabric samples, 11% of the padding samples, 4% of the stuffing samples, and 13% of the “other” sample category (Table 8). It is important to note that the number of samples collected per type (foam vs fabric) within a product were not necessarily equal. When detected in products, TDCPP and TPP were also most dominant in foam versus other sample types, while TBPH, TBB and TBEP, when detected, were only found in the foam.

Table 8. Distributions of Detected Flame Retardants, by Sample Type

Flame Retardant (FR)	Number of Products in Which FR Detected	% of Products with FR in Foam	% of Products with FR in Fabric	% of Products with FR in Padding	% of Products with FR in Stuffing	% of Products with FR in Other
TCPP	54	91%	24%	11%	4%	13%
TDCPP	15	73%	7%	7%	13%	13%
TPP	11	82%	9%	0%	0%	18%
TBPH	1	100%	0%	0%	0%	0%
TBB	1	100%	0%	0%	0%	0%
TBEP	2	100%	0%	0%	0%	0%

Presence of flame retardants in foam vs. upholstery

The presence of flame retardants detected in foams and upholstery in chairs, office chairs, ottomans, and sofas is presented in Table 9. Here the term “upholstery” is defined as the material (cloth, leather, vinyl, etc.) used to cover furniture, and was based on samples from the “fabric” sample category as well as a subset of the “other” sample type category. Results show that when flame retardants were present in a chair, they were mainly found in the foam, as opposed to in the upholstery. The same is true for ottomans and sofas. However, the results for office chairs do not necessarily exhibit this trend, and appear to be dependent on the type of flame retardant. For instance, when TCPP was detected in office chairs, it was more often found in foam samples, whereas when TDCPP and TPP were present, they were more often found in upholstery.

Table 9. Distributions of Detected Flame Retardants in Foam and Upholstery Samples

Product	Description	Number (and Percentage*) of Detections					
		TCPP	TDCPP	TPP	TBPH	TBB	TBEP
Chair	Foam	32 (76%)	9 (100%)	1 (100%)	0 (0%)	0 (0%)	0 (0%)
	Upholstery	10 (24%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)
Office Chair	Foam	14 (67%)	1 (33%)	1 (25%)	0 (0%)	0 (0%)	0 (0%)
	Upholstery	7 (33%)	2 (67%)	3 (75%)	0 (0%)	0 (0%)	0 (0%)
Ottoman	Foam	5 (83%)	4 (100%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)
	Upholstery	1 (17%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)
Sofa	Foam	25 (89%)	4 (80%)	7 (100%)	1 (100%)	1 (100%)	2 (100%)
	Upholstery	3 (11%)	1 (20%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)

* Calculated percentages are based on the number of detections of a specific flame retardant in foams and upholstery samples for a given product (chair, office chair, ottoman, sofa).

XRF and GC/MS Results Correlation

Although a direct correlation of the XRF and GC/MS results was not clearly identified in this study, XRF may still be used to provide guidance for determining the potential for the presence of a chlorinated, organophosphorus, or brominated flame retardant (Stapleton 2011; van Bergen and Stone 2014). A variety of causes may contribute to the absence of a strong correlation between XRF and GC/MS results, including the possible presence of additional compounds containing Cl, P, and/or Br. These compounds could be flame retardants that are not included in the scope of this work, or could be other compounds containing these elements. Further evaluation of XRF as a flame retardant screening tool is needed to clearly understand the advantages and limitations of this technique.

Conclusion

In this work, portable XRF screening and GC/MS laboratory analyses have been used to determine the presence of 16 specific flame retardants in products obtained in Canada, Mexico, and the United States. The XRF results reveal that 78% of the samples (559 of 717) taken from the products exhibited concentrations of Cl, P, and/or Br above the XRF screening limit of 300 ppm. The percentage of samples exceeding the 300 ppm screening limit was fairly even amongst Canada (79%), Mexico (78%), and the US (77%). The GC/MS results from these pre-screened samples indicated that the majority of the flame retardants were found in the foam (approximately 90% of the time). Fabrics exhibited the next highest occurrence of flame retardants (7–24%), followed by “other” (13–18%), and lastly by padding (7–11%) and stuffing (4–13%). The approximate ranges of concentrations found in the samples were as follows: TCPP, 100–89,000 ppm; TDCPP, 275–58,000 ppm; TPP, 200–11,500 ppm; TBEP, 750–1700 ppm; TBPH, 14,000 ppm (one result); and TBB, 39,000 ppm (one result). Overall, 46% of the products tested positive for one of the specific flame retardants targeted in this study.

Further evaluation of the flame retardant distribution in the products revealed that for chairs, ottomans, and sofas, flame retardants are mainly found in the foam, as opposed to in the upholstery. The results for office chairs are different in that the distribution of flame retardants in foams versus upholstery depends on which flame retardant is present. For office chairs, when TCPP is present it is more often found in foam samples, whereas when TDCPP and TPP are present, they are more often found in upholstery samples. These types of relationships can provide general guidance on which flame retardants are more likely to be found in different parts of a product.

XRF was a useful screening tool in identifying samples containing the presence of higher levels of elements of the target flame retardants. While correlations in concentrations of the results of the XRF screening and the GC/MS were not found, these results were also not unexpected as XRF measures contributions from all Cl, P, and Br compounds present in a material.

An additional limitation of both the XRF and GC/MS analyses is the absence of certified reference materials (CRMs) in matrices relevant to the samples analyzed in this work that contain the 16 flame retardants. Without CRMs it is difficult to determine absolute uncertainties for the XRF and GC/MS analyses. To further define the uncertainties in laboratory analyses for flame retardants, round-robin testing with a robust sampling and analysis methodology is suggested. This approach would establish a better understanding of the variability between cooperating partner laboratories during sample generation, the extraction processes, and the instrumental analyses.

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