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Identification of Phthalate and Alternative Plasticizers, Flame Retardants, and Unreacted Isocyanates in Infant Crib Mattress Covers and Foam

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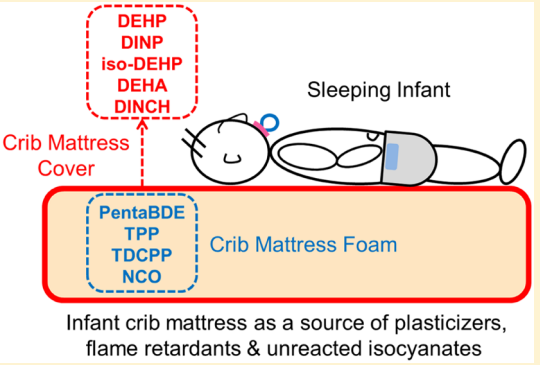
Supporting Information

ABSTRACT: Infants spend most of their time sleeping, where they are in intimate contact with their crib mattresses. In this study, we analyzed the cover and foam layers of 20 new and used crib mattresses for selected chemical additives. Seventeen of the 20 crib mattress covers contained at least one identifiable plasticizer, with concentrations ranging from 1 to >35% by weight. Sixty percent of the covers contained a total plasticizer content of >9% by weight. Nine of the 20 covers contained either bis(2-ethylhexyl) phthalate (DEHP) or diisononyl phthalate (DINP). In contrast, phthalate alternatives, including diisononyl 1,2-cyclohexanedicarboxylic acid (DINCH) and bis(2-ethylhexyl) isophthalate (iso-DEHP), were the most frequently identified plasticizers in crib mattresses manufactured after the U.S. Consumer Product Safety Improvement Act (CPSIA) went into effect. Flame retardants, including pentabromodiphenyl ether (pentaBDE) congeners and triphenyl phosphate (TPP), and unreacted isocyanates (NCO) were also identified in crib mattresses with polyurethane foam.



INTRODUCTION

Infants spend a considerable amount of their time sleeping, typically 12−13 h/day, making the sleep microenvironment particularly important in contributing to both their acute and chronic exposures to indoor air pollutants, particularly those originating in crib mattresses on which they sleep. The infant sleep microenvironment can be defined as the space encompassing a crib mattress, bedding, and crib and volume of air above these items that includes both an infant's breathing zone (BZ) and thermal plume. Crib mattresses are typically assembled in multiple layers, with a thick layer of polyurethane or polyester foam (inner springs are also used) encased within a thin, waterproof, and soft polyvinyl chloride (PVC) cover to protect the mattress foam and to provide an easy-to-clean surface. Different classes of chemical additives can be used in each material layer, which may migrate from the mattress to the air around a sleeping infant and accumulate in settled mattress dust and on bedding fibers.

The soft and flexible nature of crib mattress covers suggests the use of plasticizers;[1,2] however, the occurrence of such additives in this particular baby product, as well as their concentrations, has not been previously reported in the literature. To meet various flammability standards, brominated and organophosphate flame retardants are often added to the foam padding at percent levels, ranging from 0.1 to 10% by weight.[3] Crib mattresses have also been shown to be an important indoor source of volatile organic compounds (VOCs), with emission rates comparable to those of flooring materials, wallcoverings, and plastic toys.[4] In addition, studies have suggested that unreacted isocyanates may remain in polyurethane foam-based consumer products if excess levels of toluene diisocyanate (TDI) are used in the foam's production.[5,6] Health effects of the various chemical additives found in crib mattresses are discussed in the Supporting Information.

An infant's exposure via inhalation and dermal pathways to these compounds may be augmented by several factors. First, infants are in very close contact with their crib mattress and can receive elevated levels of exposure because of the source-proximity effect intrinsic to the sleep microenvironment. Recent laboratory studies have shown that BZ concentrations of gases (e.g., VOCs) and particles (e.g., resuspended mattress dust) released from mattresses are greater than those in the bulk bedroom air by factors typically in the range of 1.5−2.5.[4,7−9] Second, heat transfer from a sleeping infant (skin temperature of ∼36 °C)[10,11] to their crib mattress can increase the rates of emission of VOCs and semi-volatile organic compounds (SVOCs) by factors ranging from 2 to 10,

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Table 1. Description of Crib Mattress Samples Analyzed in This Study and a Summary of the Plasticizers Detected in These Samples at Concentrations of ≥1.0 mg/g in Covers or ≥0.1 mg/g in Foam

Crib Mattress Samples[1]			Plasticizers (mg/g)											
New Samples (n = 9)			DEHP		iso-DEHP		DINP		DINCH		DEHA		Σ Plasticizers[4]	
ID[2]	Year Manufactured	Retail Cost	Cover	Foam	Cover	Foam	Cover	Foam	Cover	Foam	Cover	Foam	Cover	Foam
1	2011	53USD							93.7				93.7	
2	2011	43USD							68.8	0.1	4.8		73.6	0.1
3	2011	39USD	83.1	0.8			28.5						111.6	0.8
4	2011	35USD		1.4					34.9				34.9	1.4
5	2011	89USD			352.5								352.5	
6	2011	70USD							15.4				15.4	
7	2011	120USD			276.5								276.5	
8	2011	148USD		0.5										0.5
20	2011	22USD	125.7	4.2									125.7	4.2
Detection Frequency			22.2%	44.4%	22.2%	n.d.	11.1%	n.d.	44.4%	11.1%	11.1%	n.d.	88.9 %	55.5 %
Mean Concentration (mg/g)			104.4	1.7	314.5		28.5		53.2	0.1	4.8		135.5	1.4
Used Samples (n = 11)														
ID	Year Manufactured	Usage												
9	2009	1 yr.							103.1	0.1		0.2	103.1	0.3
10	2005	2 yr.												
11	2008	2 yr.					149.8				44.1		193.9	
12	2005	2 yr.	43.2	0.9			40.9				53.9		138.0	0.9
13	2001	2 yr.	3.3				207.1	63.6			40.7		251.1	63.6
14	2003	3.5 yr.					306.2	0.6			14.4		320.6	0.6
15	1993	10 yr.		7.9			42.0	16.5					42.0	24.4
16	2007	4 yr.		8.0	60.0	14.4						0.1	60.0	22.5
17	2007	3 yr.	4.4	0.4			93.0						97.4	0.4
18	2003	< 1 yr.	32.9	5.3			118.5	8.5			2.5	0.4	153.9	14.2
19	2000	Unk.		10.6										10.6
Detection Frequency			36.4%	54.5%	9.1%	9.1%	63.6%	36.4%	9.1%	9.1%	45.5%	27.3%	81.8 %	81.8 %
Mean Concentration (mg/g)			21.0	5.5	60.0	14.4	136.8	22.3	103.1	0.1	31.1	0.2	151.1	15.3

[1]: All samples, excluding 19, manufactured in the U.S. Sample 19 was manufactured and used in Finland and had a fabric cover. Samples 2, 3, 6-8, 20, 10, 13-16, 18, and 19 were manufactured with polyurethane foam; samples 7 and 8 were manufactured with a fraction of soy-derived foam; and samples 1, 4, 5, 9, 11, 12, and 17 were manufactured with polyester foam (as listed by manufacturer).
[2]: Sample ID consistent with crib mattress samples analyzed in Boor et al. (4).
[3]: Blank cell: plasticizer not detected (n.d.) at concentrations ≥ 1.0 mg/g Cover or ≥ 0.1 mg/g Foam.
Nomenclature: DEHP: Bis(2-ethylhexyl) phthalate; iso-DEHP: Bis(2-ethylhexyl) isophthalate; DINP: Diisononyl phthalate; DINCH: Diisononyl 1,2-cyclohexanedicarboxylic acid; DEHA: Bis(2-ethylhexyl) adipate
[4]: Total plasticizer content in each crib mattress cover or foam layer
not detected (n.d.) ≥ 0.1 to 1 mg/g (for foam only) > 1 to 10 mg/g > 10 to 100 mg/g > 100 mg/g

depending on the particular compound.[4,12] Lastly, infants can receive much larger doses of a chemical additive released from their mattress compared to adults because they inhale approximately 10 times more air per body mass than adults and have skin surface area to body mass ratios 3 times greater than that of adults (Figure S1 of the Supporting Information). The latter is especially important when considering an infant's exposure to plasticizers in the crib mattress cover via contact transfer between the skin and the cover and air-to-skin uptake.[13,14]

In previous papers,[4,12] we studied the emission dynamics of VOCs from crib mattresses and examined the influence of temperature on emissions of phthalates from PVC products, including crib mattress covers. In this paper, we focus on levels and trends associated with the usage of various chemicals in crib mattresses over the past decade. The main objective was to investigate the occurrence of phthalate and alternative plasticizers in new and used crib mattresses and to determine their concentrations. Our secondary objective was to identify selected flame retardants, unreacted isocyanates (NCO), and several lower-volatility compounds in crib mattress foam. The new data gathered from this study will help to inform policy decisions in the United States, educate parents and the general public, and advance research on infant exposures to pollutants in sleep microenvironments, all of which are urgently needed.

MATERIALS AND METHODS

Sample Collection and Extraction. A collection of 20 new and used crib mattresses were analyzed in this study. Nine new crib mattresses, made by different manufacturers, were purchased from an online retail store in 2011. Eleven used crib mattresses, manufactured between 1993 and 2009, were obtained through donations in Austin, TX (10 mattresses), and Helsinki, Finland (one mattress). The samples are described in further detail by Boor et al.[4] For each crib

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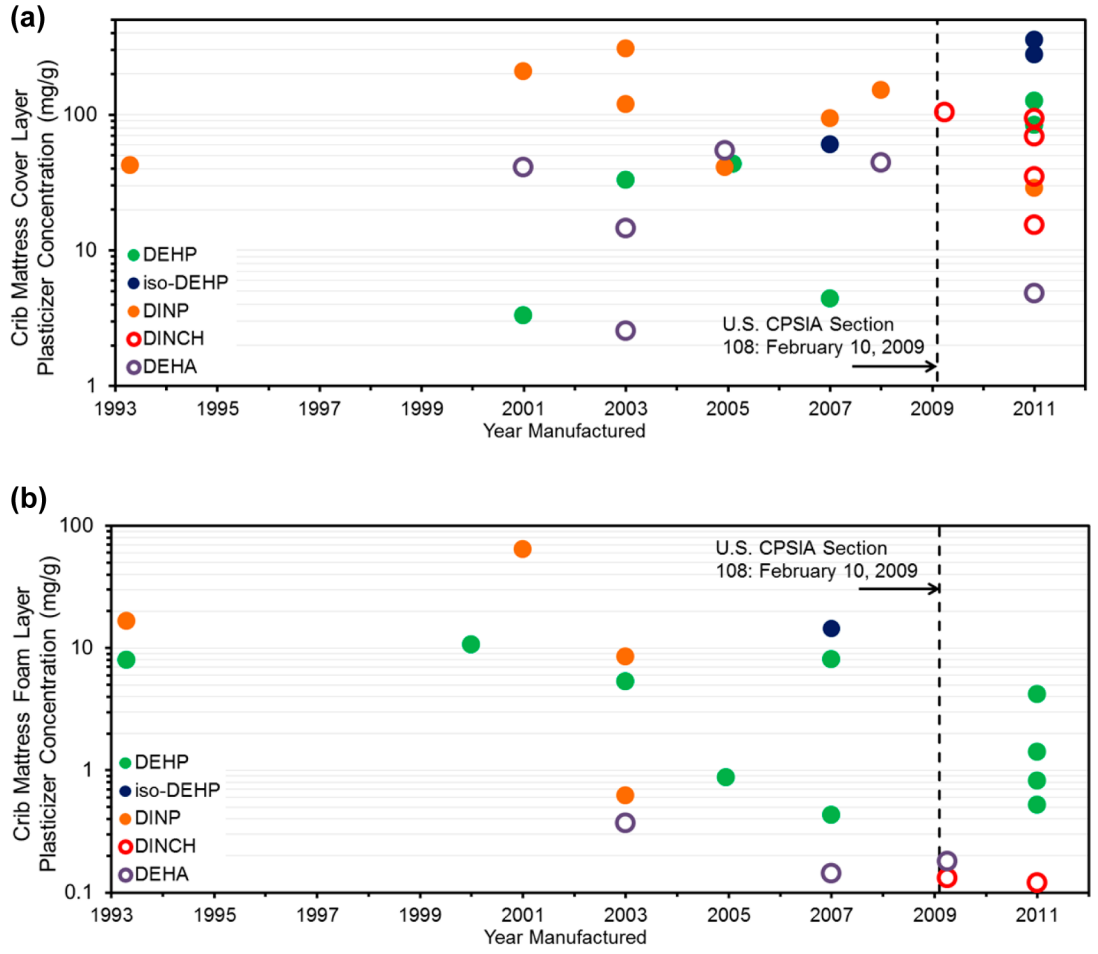

Figure 1. Plasticizer concentrations in cover (a) and foam (b) layers vs the year crib mattress samples were manufactured. Date on which the U.S. Consumer Product Safety Improvement Act (CPSIA) Section 108 was enacted in the United States (February 10, 2009) denoted by a dashed line.

mattress, a sample of both the cover layer and the underlying foam layer were examined.

Sample Analysis by PAS-FT-IR and Mass Spectrometry. Crib mattress cover and foam samples were first screened for possible phthalate content via nondestructive photoacoustic (PAS) Fourier transform infrared (FT-IR) spectroscopy, as described in the Supporting Information. This screening-level analysis suggested that phthalates, or compounds with similar chemical structure, were likely present in many of the mattress covers (Figure S3a of the Supporting Information). Foam samples were also screened for the presence of NCO (Figure S3b of the Supporting Information).

Following PAS-FT-IR screening analysis, all crib mattress cover and foam samples were extracted via ultrasonication in hexane and analyzed by gas chromatography and mass spectrometry (GC−MS) following our previously published methods for phthalate and alternative plasticizers with minor modifications.[12,15] All samples were analyzed for eight plasticizers: bis(2-ethylhexyl) phthalate (DEHP), bis(2-ethylhexyl) isophthalate (iso-DEHP), diisononyl phthalate (DINP), diisononyl 1,2-cyclohexanedicarboxylic acid (DINCH), bis(2-ethylhexyl) adipate (DEHA), dimethyl phthalate (DMP), di-*n*-butyl phthalate (DnBP), and butyl benzyl phthalate (BBP). However, the later three were not detected in any samples. Samples were also analyzed for selected flame retardant additives, including triphenyl phosphate (TPP), tris(1,3-dichloro-2-propyl)phosphate (TDCPP), and pentabromodiphenyl ether (pentaBDE) congeners, along with several additional compounds, such as phenol. Detailed information about the extraction and analytical methods can be found in the Supporting Information.

RESULTS AND DISCUSSION

Identification and Quantification of Plasticizers in Crib Mattress Covers and Foam. Table 1 provides an overview of phthalate and alternative plasticizers detected in the new and used crib mattress covers and foam. Seventeen of the 20 crib mattress covers contained at least one identifiable plasticizer at concentrations greater than 1 mg/g (0.1% by weight), and the most common plasticizer detected was DINP, found in 40% of all samples. The mean total plasticizer contents of new and used covers were 135.5 and 151.1 mg/g, respectively; 60% of the samples contained more than 90 mg/g of plasticizers, and 20% contained over 200 mg/g of plasticizers (samples 5, 7, 13, and 14). The plasticizer content is dependent on several factors, including the amount added during the initial manufacture, the homogeneity of its distribution throughout the product, the extraction and analytical methods employed in this study, and the volatilization of the plasticizer from the crib mattress over time, which, for SVOCs, is typically only a small fraction of the initially added mass.[15,16] The plasticizer concentrations in crib mattress covers are comparable to those reported for a vinyl pillow protector (140 mg of DEHP/g),[17] a nursing pillow cover (144

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mg of DINP/g),[18] and a children's sofa (210 mg of DEHP/g).[19] Plasticizers were not identified in three mattress covers: samples 8, 10, and 19. Sample 19 had a fabric cover (the only nonsoft PVC cover tested), and samples 8 and 10 may have contained a plasticizer not included among the eight target compounds.

DINCH, marketed under the name Hexamoll,[20] was the most frequently detected plasticizer in new covers (44.4% of samples), and DINP was the most frequently detected plasticizer in used covers (63.6% of samples). The two phthalate plasticizers, DEHP and DINP, were detected in nine of the 20 covers. Samples 3, 12, 13, 17, and 18 contained both phthalates, with DINP typically occurring at concentrations greater than those of DEHP. A similar observation of the co-occurrence of DEHP and DINP was also made in soft PVC flooring products.[12] The highest detected concentration of DEHP was found in the new crib mattress (sample 20, 125.7 mg/g) with the lowest retail cost (22USD). However, in general, no trends were observed between plasticizer concentrations and crib mattress quality. Two of the new crib mattresses (samples 7 and 8) were marketed as "eco or green mattresses" and manufactured with a fraction of soy-derived foam. Sample 8 did not contain target plasticizers beyond 1 mg/g, and sample 7 contained 276.5 mg of iso-DEHP/g. DEHA was primarily detected in crib mattresses made in 2008 and earlier, whereas two of the three samples containing iso-DEHP, marketed under the name "Flexol Plasticizer 380",[21,22] were made in 2011. DEHA was always detected in the presence of another plasticizer and may be used as part of a plasticizer mixture in covers.

Plasticizer concentrations in crib mattress polyurethane or polyester foam were typically in the range of 0.1−10 mg/g, although several foam samples contained higher concentrations, such as sample 13 (63.6 mg of DINP/g). These concentrations are comparable to levels of flame retardants in crib mattress foam.[3] Dibutyl phthalate (DBP) was also detected in two foam samples (Table S1 of the Supporting Information). The identified plasticizers may have originated during the manufacturing process, as they can be used as dispersion media for catalysts, dyes, fillers, and stabilizers in urethane foam feedstocks.[23] There were 13 occurrences of paired detection of a specific plasticizer in both the cover and foam layers, and higher plasticizer concentrations were associated with older mattresses (Figure 1). This suggests that, over time, the foam behaved as a sorptive reservoir for gas-phase plasticizers released from the overlying cover layer or from products found elsewhere in a residence, although greater concentrations of plasticizers may have been used during the initial manufacture of the foam in older mattresses.

Trends Associated with the 2008 U.S. CPSIA. Although concentrations of a specific plasticizer in crib mattress covers did not show strong associations with the age of the mattress, trends in the occurrence of plasticizers were observed in regard to the U.S. Consumer Product Safety Improvement Act of 2008 (CPSIA), as shown in Figure 1. The CPSIA limits the concentrations of DEHP, DBP, and BBP in children's toys or child-care articles to <0.1% by weight (1 mg/g).[24] Furthermore, a recent report by the Chronic Hazard Advisory Panel (CHAP) to the U.S. Consumer Product Safety Commission (CPSC) recommended that DINP be permanently included in this list.[25] However, the inclusion of a crib mattress in the definition of a child-care article is not clear.[26−28]

Since the CPSIA went into effect, there has been more frequent use of phthalate alternatives, including DINCH and iso-DEHP, which were identified in seven of the 10 cover samples manufactured from 2009 to 2011. The use of DINCH as a phthalate replacement in crib mattresses follows the trend observed in other soft PVC products,[29] and isophthalates (iso-DEHP) are being used in the same manner.[22] However, DEHP concentrations in two of the crib mattress covers manufactured after the CPSIA went into effect (sample 3, 83.1 mg/g; sample 20, 125.7 mg/g) exceeded the 1 mg/g limit of the CPSIA for child-care articles. Considering all 20 crib mattresses, nine would not have met the current CPSIA limits or CHAP recommendations as they contained either DEHP or DINP at concentrations greater than 1 mg/g.

Additional Compounds Detected in Crib Mattresses. Polybrominated diphenyl ether (PBDE) congeners associated with the pentaBDE mixture were identified in three crib mattress foam samples: sample 10 (2.2 mg/g), sample 15 (5.5 mg/g), and sample 18 (14.7 mg/g) (Table S1 of the Supporting Information). Samples 15 and 18 were manufactured before the 2004 pentaBDE phase-out,[30] whereas sample 10 was manufactured in 2005. TPP was identified in the three samples containing pentaBDE congeners, as well as several additional foam samples and two crib mattress covers, where it may have been used as a plasticizer.[31,32] The co-occurrence of the two flame retardants is likely the result of both being used in combination during the manufacture of the foam[3] or adsorption of gas-phase TPP or pentaBDEs in a residence over time. In total, eight of the 20 crib mattress foam layers contained pentaBDE congeners, TPP, or TDCPP (in sample 7 with partial soy-derived foam), all of which were manufactured with polyurethane foam. All crib mattresses with a label indicating that it met one of three flammability standards [Technical Bulletin (TB) 116, TB 117, or FF 4-72] contained either pentaBDEs, TPP, or both. NCO, as identified through PAS-FT-IR analysis, was detected in all 13 polyurethane foam samples (Table S1 of the Supporting Information), likely at greater levels in new mattresses than in used (Figure S4 of the Supporting Information). Further discussion of NCO can be found in the Supporting Information. Additionally, phenol, 1-decene, 1-nonanol, and palmitic acid were detected in crib mattresses, the latter two of which were likely adsorbed over time by the mattress, as discussed by Boor et al.[4]

Implications for SVOC Transport and Infant Exposure in the Sleep Microenvironment. The findings from this study, along with Stapleton et al.[3] and Boor et al.,[4] collectively demonstrate that infant crib mattresses can contain SVOCs, including plasticizers and flame retardants, VOCs, and unreacted isocyanates. Given the slow and nearly constant rate of emission of plasticizers from soft PVC materials,[16,33] it is likely that crib mattresses are a constant plasticizer source in the infant sleep microenvironment, which is important, considering the long lifetime of a crib mattress (~10 years), mattress reuse in families with multiple children, and purchasing of used mattresses through second-hand venues. This is in contrast to VOC emissions that tend to decay over time.

It is expected that the chemical additives identified in this study will volatilize from the crib mattress; however, experimental data on the emissions of SVOCs from mattresses, pillows, and bedding are limited.[34,35] Kemmlein et al.[36] reported an emission rate of 0.012 μg m^{-2} h^{-1} for tris(2-chloroisopropyl)phosphate (TCPP) from a mattress, and our parallel study evaluated the emissions of DINCH (sample 9)

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and DEHA (sample 11) from the cover layers.[12] Crib mattress covers act as a diffusive barrier to the transport of VOCs originating in the underlying foam[4] and may do the same for flame retardants as they migrate from the foam to ambient air. This may partially explain the paired detection of TPP in the cover and foam layers of sample 15.

The volatilized gas-phase plasticizers and flame retardants will tend to partition to and accumulate in surface dust particles (e.g., refs 37 and 38). As infants move and crawl in their cribs, they will likely resuspend settled mattress dust[7,8,39] and thus be exposed to particle-phase plasticizers and flame retardants. Oral exposure may also occur via hand-to-mouth transfer of mattress dust during sleep periods.[40] The gas-phase SVOCs may also adsorb to bed sheets, where they may subsequently desorb during sleep periods, as the temperature of the bedding fibers increases.[13] In addition, dermal uptake of plasticizers and flame retardants in infants may occur as they sleep via direct contact transfer between the skin and crib mattress cover or surface-sorbed SVOCs on bed sheets, and air-to-skin transport. The latter is linked to concentrations of gas-phase pollutants released from the mattress, which are much greater near the mattress surface than in the bulk bedroom air.[4,9]

■ ASSOCIATED CONTENT

Supporting Information

Additional information as denoted in the text. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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