Phthalates, Alkylphenols, Pesticides, Polybrominated Diphenyl Ethers, and Other Endocrine-Disrupting Compounds in Indoor Air and Dust

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Chemicals identified as endocrine-disrupting compounds (EDCs) have widespread consumer uses, yet little is known about indoor exposure. We sampled indoor air and dust in 120 homes, analyzing for 89 organic chemicals identified as EDCs. Fifty-two compounds were detected in air and 66 were detected in dust. These are the first reported measures in residential environments for over 30 of the compounds, including several detected at the highest concentrations. The number of compounds detected per home ranged from 13 to 28 in air and from 6 to 42 in dust. The most abundant compounds in air included phthalates (plasticizers, emulsifiers), *o*-phenylphenol (disinfectant), 4-nonylphenol (detergent metabolite), and 4-*tert*-butylphenol (adhesive) with typical concentrations in the range of 50−1500 ng/m^3. The penta- and tetrabrominated diphenyl ethers (flame retardants) were frequently detected in dust, and 2,3-dibromo-1-propanol, the carcinogenic intermediate of a flame retardant banned in 1977, was detected in air and dust. Twenty-three pesticides were detected in air and 27 were detected in dust, the most abundant being permethrins and the synergist piperonyl butoxide. The banned pesticides heptachlor, chlordane, methoxychlor, and DDT were also frequently detected, suggesting limited indoor degradation. Detected concentrations exceeded government health-based guidelines for 15 compounds, but no guidelines are available for 28 compounds, and existing guidelines do not consider endocrine effects. This study provides a basis for prioritizing toxicology and exposure research for individual EDCs and mixtures and provides new tools for exposure assessment in health studies.

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Introduction

Current widespread interest in a range of health effects potentially associated with endocrine-disrupting compounds (EDCs) has made exposure assessment for these compounds a priority. Studies of potential health effects associated with EDCs have been hampered by lack of information about the major sources of exposure to EDCs. Furthermore, because many EDCs act additively through a common mechanism of action or have antagonistic or other interactive effects by operating at different points in cell signaling systems, consideration of exposure to mixtures is critical in studies of health effects (*1*−*7*). These questions are particularly important in relation to indoor environments, which have been identified as an important source of chemical exposures (*8*−*11*). People spend a large fraction of their time indoors, and indoor sources of chemicals, coupled with limited ventilation and slow chemical degradation processes, cause increased pollutant concentrations indoors. In fact, indoor air specifically has been described as "one of the most serious environmental risks to human health" (*8*).

Many high production volume chemicals—including some already identified as EDCs—have consumer uses (e.g., in plastics, detergents, and other household and consumer products) that make them potentially important indoor contaminants. While a number of comprehensive exposure studies have been conducted or are underway to characterize residential exposures to selected contaminants, particularly volatile organic compounds, pesticides, and polyaromatic hydrocarbons (PAHs), these studies have been limited to a small number of compounds and have focused on characterizing exposure pathways and sources (*12*−*18*). We were unable to locate exposure data for many of our compounds of interest, including alkylphenols, parabens, polybrominated diphenyl ethers (PBDEs), and many of the estrogenic phenolic compounds such as bisphenol A. We located only one (unpublished) study of substantial size that has characterized phthalate concentrations in indoor air (*18*).

The primary objective of this study is to provide an assessment of household exposure to a broad suite of organic chemicals that have been identified as EDCs. Indoor air and dust were selected for analysis because many EDCs are used in consumer products and building materials (*6*, *19*), so these chemicals would be expected indoors. Indoor air has been identified as an important source of chemical exposure, while house dust has been demonstrated to be an important exposure pathway in young children (*20*). Dust also provides a record of chemicals that have been used in the home historically since degradation processes indoors are typically slow (*21*).

The chemicals targeted for analysis included phthalates, alkylphenols, pesticides, parabens, PBDEs, PAHs, polychlorinated biphenyls (PCBs), and other estrogenic phenols such as bisphenol A. These compounds were selected if there was evidence that they were EDCs, if they were reported to be present in commercial products or building materials, and/or if they were compatible with one of two analytical methods being used for these samples. We previously reported on the selection of target compounds and methods for measuring them in air and dust (*22*).

This paper describes the analytical results for indoor air and house dust samples from 120 homes on Cape Cod, MA. Air and dust samples were analyzed for 89 target chemicals, many identified as EDCs. The large number of homes provides insight into population distributions of exposure to

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TABLE 1. Number of Analytes and Related Data Collection by Chemical Group for Samples Taken in 120 Homes on Cape Cod, MA[a]

	no. of analytes			related data collection	
chemical group	dust	air	urine[b]	interview[b]	GIS-based[b]
pesticides	38	39	13	+	+
alkylphenols	7	7		~	
phthalates	10	9	8	~	
PCBs, PAHs, PBDEs	10	10		~	
parabens	3	3			
other estrogenic phenols and misc.	18	20			
estrogenic activity (E-SCREEN MCF-7 bioassay)[b]		+			

[a] +, data of this type were collected in this study. ~, limited questions related to sources of these compounds were included in the interview. [b] These data will be reported in subsequent papers.

target compounds, and the large number of analytes provides insight into typical mixtures of EDCs to which people are exposed. Table 1 provides an overview of the study design. In addition to the air and dust samples, we collected a urine sample from a resident of the home and a detailed questionnaire about product use and home construction. We also used a geographic information system (GIS) to estimate the relative exposure at each home from historical wide-area pesticide use (*23*). Finally, air samples were extracted, and total estrogenic activity was determined using an MCF-7 cell proliferation assay (E-SCREEN) (*24*). Relationships across these measures will be reported separately. This household exposure study was conducted as part of a case-control epidemiologic study of breast cancer on Cape Cod, MA (*25*).

Methods

Participant Selection. Eligible women were either breast cancer cases or age-matched controls, were currently alive and residing on Cape Cod, and had lived in their home at least 10 yr at the time of the sampling. To enhance variability across subjects and improve the precision of estimates of upper and lower percentiles of exposure distributions for pesticides, we oversampled individuals with higher and lower potential for pesticide exposure based on self-reported pesticide use and a GIS-derived measure of historical wide-area application of persistent pesticides. Sampling was conducted in two rounds of 60 homes per round, beginning in June 1999 and ending in September 2001. All sample collection and analyses were the same for both rounds, although minor changes were made to the target analyte list between rounds.

Sample Collection. *Air.* The 24-h indoor air samples of particulate $<5\,\mu$m and vapor phase materials were collected using a quiet indoor flow-controlled model SP-280 pump (Air Diagnostics and Engineering, Harrison, ME) modified to collect three parallel 160-mm URG personal pesticide sampling cartridges (University Research Glassware, Chapel Hill, NC). Each URG cartridge contained an impactor-equipped inlet ($10\,\mu$m at 4 L/min) followed by a glass cartridge that was fitted with a 25-mm quartz fiber filter followed by a 3.0-g bed of XAD-2 resin sandwiched between two $1^{13}/_{16}$ in. diameter polyurethane foam plugs. Preparation of the URG cartridges is described in our earlier paper (*22*). Pumps were operated at a constant flow rate of 20−24 L/min. Flow control valves were used to control flow rates for the three parallel URG cartridges so that two samples were collected at flow rates of 8−9 L/min, and a third was collected at 4 L/min. Actual flow rates were determined at the beginning and end of the 24-h sample collection period using a high-flow Gilian Gilibrator primary standard flow calibrator (Environmental Monitoring Supply). The two URGs collected at the higher flow rate were used for extraction and analysis by the two analytical methods, while the third URG was used to collect duplicate or other samples. The total volume of air sampled ranged from 10 to 14 m^3 for the primary samples and from 4 to 6 m^3 for the duplicate samples.

On day 1 of sample collection, the pump was placed in a frequently used room of the home, such as the living room or family room, and the URGs were suspended so that the intakes were directed downward 4 ft from the floor. The pump was then calibrated and turned on. On day 2, the URGs were disconnected, and the flow was checked. URGs were stored at −4 °C and then shipped on dry ice to Southwest Research Institute (SWRI) in San Antonio, TX, where they were extracted and analyzed.

Dust. Dust samples were collected using a Eureka Mighty-Mite vacuum cleaner, 9 amp, modified to collect dust into a 19 × 90 mm cellulose extraction thimble (Whatman Inc., Clifton, NJ). Because of the number of our target analytes associated with plastic materials, a custom crevice tool with a holder for the extraction thimble was constructed of PTFE Teflon so dust did not contact any plastic parts of the vacuum. Dust sample collection did not begin until the air sample collection was complete. Sample collection was accomplished by slowly and lightly drawing the crevice tool just above the surface of rugs, upholstery, wood floors, windowsills, ceiling fans, and furniture in each room. Sampling was conducted in the most frequently used rooms of the house, usually 4−5 rooms and including hallways. Unfinished/semifinished areas such as basements, attics, and garages were not sampled. Using this technique and collecting for 45−90 min, approximately 4 g of dust was collected per sample. Cellulose thimbles containing dust were removed and placed in precleaned, certified glass jars with Teflon-lined lids (Environmental Sampling Supply, Oakland, CA). Samples were stored at −4 °C until they were shipped overnight on dry ice to SWRI. Prior to extraction, dust was tapped out of the thimbles, weighed, and sieved to $<150\,\mu$m. These samples were split into aliquots for extraction and analysis by each of the two methods. Fourteen samples were split into a larger number of aliquots, with and without spiking with target compounds to determine recovery efficiency. Final sample masses of aliquots used for extraction and analysis ranged from 0.047 to 1.6 g per method (median 0.385 g).

Chemical Analysis. Chemical analysis of air and dust samples was conducted at SWRI. Two GC/MS analytical methods were used to analyze a total of 88 target compounds in air and 86 compounds in dust samples (total of 89 different compounds). One method targets neutrally extracted pesticides, phthalates, PAHs, PBDEs, and PCBs. The second method, which requires derivitazation of the extract prior to analysis, targets alkylphenols—specifically 4-nonylphenol, 4-octylphenol, and their mono- and diethoxylates as well as parabens and other phenols and biphenyls identified as EDCs. The chlorpyrifos metabolite and degradation product 3,5,6-trichloropyridinol and the methoxychlor metabolite/degradation product 2,2-bis(*p*-hydroxyphenyl)-1,1,1-trichloroethane (HPTE) and some chlorinated phenols were also included as target analytes of the phenols method. All target analytes are included in Supporting Information Tables S1 (air) and S2 (dust).

Neutrals/Phthalates Extraction and Analysis. Each sieved ($<150\,\mu$m) dust sample was spiked with the required amount of surrogate solution, 40 ng/mL *p*-terphenyl-d_{14}, and/or matrix spike solutions (in hexane) depending on the actual size of the dust sample. The spiked dust samples were equilibrated for 30 min at room temperature and then Soxhlet

extracted using 6% diethyl ether in hexane for 16 h. The extracts were concentrated to 10 mL, and a 1-mL aliquot was cleaned by running through a florisil column (elution with 20 mL 10% acetone in hexane). When less than 2 g of sieved dust was available, proportionately smaller amounts of surrogates were spiked, and extracts were concentrated to proportionately smaller volumes. The florisil eluent was concentrated to a final volume of 2 mL with 10% ether in hexane for analysis by GC/MS.

The contents of each URG (XAD-2/PUF/filter) were Soxhlet extracted for 16 h in 150 mL of 6% ether in hexane solution with 100 mL of surrogate solution of *p*-terphenyl-d_{14} at 2.0 ng/mL. After being cooled, if water was visibly present in any of the extracts, the extract was passed through a glass drying tube containing sodium sulfate. The extracts were concentrated to 2 mL and quantitatively transferred to a 3.7-mL vial, and the final volume was adjusted using 10% diethyl ether in hexane.

Analysis for the neutral target analytes was performed using an Agilent 6890/5973 (or a Thermoquest MD800) GC/MS in selected ion monitoring (SIM) mode. A 60 m × 0.25 mm i.d. DB-5MS column was used as the GC analytical column. The GC/MS instrument was scanned to monitor two or four selected ions per analyte. The base peak ion (or the second most intense peak if there was interference with the base peak) was used as the quantification ion for each compound (*22*). Quantification was performed using labeled PAHs as internal standards (naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene -d_{12}, perylene-d_{12}). The percent relative standard deviation (% RSD) of each analyte was maintained within 30% during the initial five-point standard calibration. A continuing calibration standard was processed at the beginning and end of each sequence of 15 samples. The percent difference of each analyte in the mid-level standard was generally maintained within 40% of the initial calibration value during continuing calibrations.

Phenols Extraction and Analysis. Dust samples were extracted by acidifying with 1 mL of 1:1 sulfuric acid/water (after adding 2,4,6-tribromophenol as the surrogate standard and matrix spike solutions as required), equilibrating spiked samples for 30 min at room temperature, and extracting with three portions of 18 mL of optima-grade dichloromethane (DCM) (sonicated 10 min per extraction). The three extracts were combined and evaporated under nitrogen at less than 45 °C.

The contents of each URG (quartz filter/PUF/XAD-2) were extracted 3 times with 50 mL of optima-grade DCM, 10 min shaking per extraction (after adding 2,4,6-tribromophenol as the surrogate standard and matrix spike solutions as required). After each extraction, the DCM was decanted through a glass drying tube (1.5 in. diameter, 5 in. length, HGF Scientific, Inc., Stafford, TX) containing a glass wool plug. After the last extraction, the PUF was added to the drying tube to remove any residual DCM. The extracts were concentrated to 1.0 mL under nitrogen using a N-EVAP analytical evaporator at 35−40 °C. All glassware was washed with acidified DCM (3 mL of HCl/600 mL of DCM) prior to use.

Dust and air extracts were derivatized with *N*,*O*-bis-(trimethylsilyl) trifluoroacetamide (BSTFA) at 60 °C for 60 min. Analysis was performed using an Agilent 6890/5973 GC/MS system in SIM mode. A 30 m × 0.25 mm i.d. DB-5.625 column was used as the GC analytical column. Quantification was performed using 3,4,5-trichlorophenol as the internal standard. A continuing calibration standard was processed at the beginning and end of each sequence of 15 samples. The percent difference of each analyte in the mid-level standard was maintained within 40% of the initial calibration value during continuing calibrations.

QA/QC. Extensive QA/QC measures were conducted to ensure accuracy and reliability of measurements. Of particular concern was the possibility of field and laboratory contamination with ubiquitous target compounds in plastics and other common products, so a high proportion of blank samples was included in this study.

Air. Potential sample contamination by target compounds was evaluated using both laboratory solvent and matrix (URG contents including quartz filter/PUF/XAD-2) blanks as well as field matrix blanks shipped to the laboratory with samples. Analysts were blinded to the identity of field blanks. A total of 36 neutrals and 35 phenols blank samples were analyzed along with the 120 field samples reported here. These included field blanks ($n = 7$), matrix blanks ($n = 23$), and solvent blanks (6 neutrals, 5 phenols). The nominal analyte reporting limit in this study was the analyte level in the lowest standard of the initial five-point calibration curve. When an interfering compound was present so that the presence of a target analyte at the detection limit was obscured, the reporting limit of the analyte was raised to the size of the false interfering peak. Method Reporting Limits (MRLs)are listed in Table 2 (detected analytes) and in Table S1 in Supporting Information (all analytes). Phthalates, alklyphenols, and bisphenol A were the only compounds detected in any blanks. Target analytes were reported as not detected in samples if they were present at less than the mean + 3 SD of the amount in blank samples.

Method performance was evaluated using matrix spike samples. Over the course of the sample collection, 16 phenols or 17 neutrals PUF/XAD-2 preparations were spiked with target compounds. Average recoveries ranged from 40% to 220%; data in tables and figures are qualified for any compounds with average recoveries less than 60% or greater than 150% or for compounds with highly variable recoveries (>50% of spikes outside the 60−150% recovery range).

Full-scan confirmational analyses were performed on two air sample extracts to verify large quantifications of *o*-phenyl phenol, propoxur, and phthalates. In addition, the two air samples with highest concentrations of 2,3-dibromo-1-propanol were confirmed by full scan.

Duplicate air samples (field duplicates; $n = 10$) were also analyzed by both neutrals and phenols methods to characterize reproducibility. Percent differences for field duplicate samples were typically between 15 and 25%. For a few compounds, average percent differences between field duplicates were higher than 30% (carbaryl, 33%; piperonyl butoxide, 39%; pentachlorophenol, 42%; 2,3-dibromo-1-propanol, 41%). The analyte *o*-phenyl phenol was included as a target analyte in both analytical methods for air and for dust samples as another check of the reliability of these methods. Percent differences between measurements by the two methods averaged 31%, and the two measures were well correlated (Pearson correlation coefficient, 0.87), although the phenols method tended to report slightly lower values than the neutrals method for this compound.

Breakthrough was not specifically evaluated, however "sandwich" combinations of XAD-2 between two layers of PUF have been shown to efficiently trap semivolatile organic chemicals with vapor pressures up to 10^{-3} kPa (*26*), so we expect these target compounds to be efficiently trapped with this preparation.

Dust. Potential sample contamination by target compounds was evaluated for dust samples by running 27 neutrals and 22 phenols solvent blanks. Matrix or field blanks are not readily available for house dust samples. Certain phthalates, nonyl- and octylphenol diethoxylate, and 2-*sec*-butylphenol were the only target compounds detected in solvent blanks. These target analytes were reported as not detected in samples if they were present at less than the mean + 3 SD of the blank samples.

TABLE 2. Summary Data for Detected Chemicals in Indoor Air (ng/m³)[a]

chemical	no. of homes sampled	MRL[b]	% >RL	min	median	max	chemical	no. of homes sampled	MRL[b]	% >RL	min	median	max
Alkylphenols and Alkylphenol Ethoxylates													
4-nonylphenol	120	3	100	21	110	420	nonylphenol ethoxycarboxylate	30	18	7	<RL	<RL	18
nonylphenol monoethoxylate	120	6	95	<RL	17	73	octylphenol monoethoxylate	120	10	93	<RL	8.6	50
nonylphenol diethoxylate	120	4	33	<RL	<RL	26	octylphenol diethoxylate	120	8	5	<RL	<RL	120
Phthalates													
diethyl phthalate[c]	120	75	100	130	590	4300	dicyclohexyl phthalate	102	2	21	<RL	<RL	280
di-*n*-butyl phthalate[d]	120	21	100	52	220	1100	bis(2-ethylhexyl) adipate	120	3	99	<RL	9.0	66
benzyl butyl phthalate	120	31	44	<RL	<RL	480	di-*n*-propyl phthalate	120	3	15	<RL	<RL	27
bis(2-ethylhexyl) phthalate	102	59	68	<RL	77	1000	diisobutyl phthalate	120	2	100	11	61	990
Parabens													
butyl paraben	120	4	8	<RL	<RL	3.2	methyl paraben	120	1	67	<RL	2.9	21
ethyl paraben	120	1	3	<RL	<RL	4.0							
Polycyclic Aromatic Hydrocarbons (PAHs)													
anthracene	90	1	1	<RL	<RL	3.7	pyrene	90	1	27	<RL	<RL	3.4
Polychlorinated Biphenyls (PCBs) and Polychlorinated Diphenyl Ethers (PBDEs)													
PCB 52	120	1	31	<RL	<RL	25	PCB 153	119	1	6	<RL	<RL	6.7
PCB 105	116	1	3	<RL	<RL	3.6							
Pesticides													
4,4′-DDD	90	1	3	<RL	<RL	3.5	lindane	90	2	1	<RL	<RL	110
4,4′-DDE	90	1	2	<RL	<RL	5.1	methyl parathion[d]	90	2	6	<RL	<RL	92
4,4′-DDT	90	1	10	<RL	<RL	30	pentachlorophenol[d]	120	1	58	<RL	1.6	34
bendiocarb	90	6	4	<RL	<RL	120	*cis*-permethrin	120	1	3	<RL	<RL	3.7
carbaryl	120	2	11	<RL	<RL	22	*trans*-permethrin	120	2	3	<RL	<RL	5.4
α-chlordane	120	1	51	<RL	0.10	61	*o*-phenylphenol (neutrals method)	120	1	100	12	71	970
γ-chlordane	120	1	53	<RL	0.22	83							
chlorothalonil	90	1	17	<RL	<RL	36	*o*-phenyl phenol (phenols method)	120	1	100	9.8	70	590
chlorpyrifos	120	1	38	<RL	<RL	92							
3,5,6-trichloro-2-pyridinol[d]	120	1	13	<RL	<RL	7.3	piperonyl butoxide	90	1	6	<RL	<RL	110
diazinon	120	1	40	<RL	<RL	550	prometon	90	2	1	<RL	<RL	4.3
dieldrin	90	2	4	<RL	<RL	3.0	propoxur[e]	120	4	47	<RL	<RL	110
heptachlor	120	1	44	<RL	<RL	71	trifluralin[d,f]	90	1	10	<RL	<RL	23
Phenols and Miscellaneous													
2,3-dibromo-1-propanol	85	1	9	<RL	<RL	200	*p*-phenylphenol	120	1	1	<RL	<RL	1.5
2,4-dihydroxybenzophenone[d]	85	1	1	<RL	<RL	1.2	2,4-dichlorophenol	120	1	28	<RL	<RL	6.0
4,4′-methylenediphenol[d]	120	1	3	<RL	<RL	4.9	4-nitrophenol	120	1	17	<RL	<RL	7.0
4-*tert*-butylphenol	120	1	100	3.4	16	290							

[a] Additional summary statistics in Table S1 in Supporting Information. [b] MRL is the typical method reporting limit (RL) reported as median reporting limit for nondetect samples. Some samples had higher or lower RLs due to smaller or larger sample sizes, respectively, or due to interferences. For chemicals with detects in all samples, MRL is derived from matrix blank samples and assumes typical sample size (11.6 m^3). For chemicals detected in blanks, MRL is the mean + 3 SD of the levels in matrix blanks and assumes typical sample size. [c] Average of matrix spike recoveries was high (150−220%). [d] Matrix spike recoveries were variable (>50% of spikes outside the range of 60−150%). [e] Interference from XAD-2 breakdown affects propoxur identification and quantification. [f] Average of matrix spike recoveries was low (40−60%).

TABLE 3. Summary Statistics for Household Dust Samples (μg/g)[a]

chemical	no. of homes sampled	MRL[b]	% >RL	min	median	max	chemical	no. of homes sampled	MRL[b]	% >RL	min	median	max
Alkylphenols and Alkylphenol Ethoxylates													
4-nonylphenol	118	1	80	<RL	2.58	8.68	4-octylphenol	118	0.2	2	<RL	<RL	0.090
nonylphenol monoethoxylate	118	2	86	<RL	3.36	15.6	octylphenol monoethoxylate	118	0.5	50	<RL	0.13	1.99
nonylphenol diethoxylate	118	2	86	<RL	5.33	49.3							
nonylphenol ethoxycarboxylate	30	3	93	<RL	2.12	9.45	octylphenol diethoxylate	118	0.2	69	<RL	0.306	2.12
Phthalates													
diethyl phthalate	119	4	89	<RL	4.98	111	dicyclohexyl phthalate	101	0.8	77	<RL	1.88	62.7
di-*n*-butyl phthalate	119	24	98	<RL	20.1	352	bis(2-ethylhexyl) adipate[c,d]	119	0.4	100	0.935	5.97	391
benzyl butyl phthalate[e]	119	3	100	3.87	45.4	1310	di-*n*-hexyl phthalate	119	0.1	76	<RL	1.1	30.6
bis(2-ethylhexyl) phthalate[e]	101	8	100	16.7	340	7700	diisobutyl phthalate	119	1	95	<RL	1.91	39.1
Parabens													
butyl paraben	118	0.2	22	<RL	<RL	3.92	methyl paraben	118	0.3	90	<RL	0.978	8.24
ethyl paraben	118	0.2	9	<RL	<RL	2.18							
Polycyclic Aromatic Hydrocarbons (PAHs)													
anthracene	89	0.2	13	<RL	<RL	3.05	benz[*a*]anthracene	119	0.3	76	<RL	0.499	10.0
pyrene	89	1.2	96	<RL	1.33	39.8	benzo[*a*]pyrene	119	0.4	85	<RL	0.712	18.1
Polychlorinated Biphenyls (PCBs) and Polybrominated Diphenyl Ethers (PBDEs)													
PCB 52	119	0.2	8	<RL	<RL	15.7	PBDE 47	89	0.4	45	<RL	<RL	9.86
PCB 105	119	0.2	9	<RL	<RL	16.5	PBDE 99	89	0.4	55	<RL	0.304	22.5
PCB 153	119	0.2	16	<RL	<RL	35.3	PBDE 100	89	0.3	20	<RL	<RL	3.40
Pesticides													
4,4′-DDD	119	0.2	9	<RL	<RL	0.718	heptachlor	119	0.2	3	<RL	<RL	0.549
4,4′-DDE	119	0.2	13	<RL	<RL	0.738	lindane	119	0.4	2	<RL	<RL	1.04
4,4′-DDT	119	0.3	65	<RL	0.279	9.61	malathion	119	0.2	3	<RL	<RL	1.48
alachlor	119	0.3	1	<RL	<RL	0.221	methoxychlor	119	0.5	54	<RL	0.240	12.9
bendiocarb[c,d]	114	0.2	12	<RL	<RL	40.7	methyl parathion	119	0.3	3	<RL	<RL	0.992
carbaryl[c,d]	119	0.4	43	<RL	<RL	34.4	pentachlorophenol	118	0.3	86	<RL	0.793	7.96
α-chlordane	119	0.3	39	<RL	<RL	9.97	*cis*-permethrin	119	0.3	45	<RL	<RL	61.9
γ-chlordane	119	0.3	41	<RL	<RL	10.6	*trans*-permethrin	119	0.4	53	<RL	0.387	98.0
chlorothalonil	119	0.2	19	<RL	<RL	3.20	*o*-phenylphenol (neutrals method)	119	0.4	67	<RL	0.283	1.67
chlorpyrifos	119	0.2	18	<RL	<RL	228							
3,5,6-trichloro-2-pyridinol	118	0.2	31	<RL	<RL	44.7	*o*-phenylphenol (phenols method)	118	0.3	73	<RL	0.303	2.40
cypermethrin[c]	119	1	5	<RL	<RL	172							
diazinon	119	0.2	14	<RL	<RL	51.0	piperonyl butoxide[d]	119	0.2	66	<RL	0.426	624
dicofol (ketone form)	119	0.4	6	<RL	<RL	3.54	prometon	119	0.3	1	<RL	<RL	0.095
dieldrin	119	0.4	12	<RL	<RL	4.89	propoxur[c]	119	0.2	42	<RL	<RL	12.6
Phenols and Miscellaneous													
2,3-dibromo-1-propanol	88	0.2	6	<RL	<RL	42.8	4-*tert*-butylphenol	118	0.2	5	<RL	<RL	1.12
2,4-dihydroxybenzophenone	88	0.7	63	<RL	0.515	9.36	bisphenol A[d]	118	0.2	86	<RL	0.821	17.6
3-biphenylol	118	0.2	2	<RL	<RL	0.170	*p*-phenylphenol	118	0.2	5	<RL	<RL	2.40
4,4′-biphenyldiol[d]	118	0.3	6	<RL	<RL	3.89	2,4-dichlorophenol	118	0.2	5	<RL	<RL	0.227
4,4′-methylenediphenol	118	0.2	7	<RL	<RL	0.934	4-nitrophenol[c]	118	0.4	42	<RL	<RL	4.25
4-cumylphenol	118	0.2	3	<RL	<RL	0.542							

[a] Additional summary statistics in Table S2 in Supporting Information. [b] MRL is the typical method reporting limit (RL) reported as median reporting limit for nondetect samples. Some samples had higher or lower RLs due to smaller or larger sample sizes, respectively, or due to interferences. For chemicals with detects in all samples, MRL is derived from solvent blank samples and assumes typical sample size (0.38 g). For chemicals with detects in solvent blanks, MRL is the mean + 3 SD of the levels in blanks and assumes typical sample size. [c] Average of matrix spike recoveries was high (150−220%). [d] Matrix spike recoveries were variable (>50% of spikes outside the range of 60−150%). [e] Spike recovery not determined.

Method performance (percent recoveries) was evaluated using matrix spiked ($n = 14$) samples. Average recoveries ranged from 40% to 220%.

Full-scan confirmational analyses were performed on nine dust sample extracts to verify large quantifications of bendiocarb, carbaryl, chlordane, chlorpyrifos, cypermethrin, DDT, methoxychlor, permethrin, piperonyl butoxide (PBO), propoxur, phthalates, PCB congeners, and PBDE 99. The 2,3-dibromo-1-propanol detects were also confirmed by full-scan GC/MS of three dust samples.

Duplicate dust samples (laboratory splits; $n = 4$) were also analyzed to characterize reproducibility. Average percent differences between duplicates were less than 20% with the exception of carbaryl (59%), bis(2-ethylhexyl) adipate (30%), benz[*a*]anthracene (39%), benz[*a*]pyrene (40%), and piperonyl butoxide (22%).

Data Analysis. The unadjusted descriptive statistics were calculated using the standard formulas for simple random samples. Data below the limit of detection were set equal to zero, which will cause the sample mean to be biased low.

Adjusted geometric mean concentrations and confidence intervals were calculated for target compounds after adjusting for stratified sampling. To achieve this, data and detection limits were log transformed. If there were no data below the limit of detection in a stratum, the usual within stratum arithmetic mean and standard deviation were calculated. When there were data below the limit of detection in a stratum, the normal distribution maximum likelihood estimates for the mean and standard deviation, assuming left censoring at the log detection limit were calculated. If there were no values above the detection limit within a stratum, the previous estimate does not exist.

After the within strata estimates were obtained, the adjusted means and their standard errors were calculated using the standard formulas for stratified samples (*27*). Since the data were sampled separately from cases and controls and participants in the first round were limited to women over 65 yr, the sample is more complex than a stratified random sample from the nine exposure cells. However, for the purposes of summarizing the data, they were assumed to have the simple stratified structure.

The 95% confidence intervals for the adjusted means were calculated using a *t*-distribution, with the Satterthwaite approximation to the degrees of freedom. These confidence intervals assume a normal distribution within the population. Since this assumption is probably not true for this population, the confidence intervals should be regarded as only approximate. The mean, standard error, and confidence intervals were exponentiated back to the original scale of the concentration data. It is important to realize that the estimate of the geometric mean in the original scale is consistent for the median of a log-normal distribution rather than the mean. The confidence interval in the original scale should be interpreted as a confidence interval for the median of the concentration values.

Results and Discussion

Summary Statistics. Summary data for all detected compounds are shown in Tables 2 (air) and 3 (dust), and Tables S1 and S2 in Supporting Information provide more detailed statistics and include target compounds that were not detected. Chemicals are divided into the following groups: (1) alkylphenols; (2) phthalates; (3) parabens; (4) PAHs, PCBs, and PBDEs; (5) pesticides; and (6) phenols and miscellaneous. The summary tables (Tables 2 and 3) show the number of samples tested for each detected analyte, the percent of samples with detectable levels, the method reporting limit, and the median and range for the raw data. Tables S1 and S2 in Supporting Information include additional descriptive statistics for the raw data (arithmetic mean, range of detects, and the median, 75th, and 90th percentile concentrations detected). In addition, Tables S1 and S2 (Supporting Information) present geometric means and confidence intervals for the data after (i) adjusting for stratification in the participant selection process based on self-reported and GIS-based opportunities for pesticide exposure and (ii) using maximum likelihood estimates with left censoring for non-detects. Comparison of the adjusted geometric means with the medians of the raw data show few differences, suggesting that the adjustments and parametric assumptions are in agreement with the raw results.

In all, 52 of 88 target compounds were detected in indoor air and 66 of 86 compounds were detected in house dust. The most frequently detected compounds were phthalates, which are ubiquitous in plastics, building materials, food packaging, and personal care products, and alkylphenols, which are impurities or degradation products of the alkylphenol polyethoxylates that are used in detergents and personal care products and as inert ingredients in pesticide formulations. Three phthalates were detected in air in 100% of homes, and three different phthalates were detected in dust in 100% of homes. Nonylphenol was also detected in air in 100% of homes. Other frequently detected chemicals in air and dust samples include methyl paraben, which is used in personal care products; PBDEs, which are flame retardants with properties similar to PCBs; and bisphenol A, which is a constituent of polycarbonate plastics. Pesticides detected in at least half the homes included DDT, methoxychlor, pentachlorophenol, permethrin, and the synergist piperonyl butoxide (PBO) (dust) and chlordane and pentachlorophenol (air). The disinfectant *o*-phenyl phenol was detected in air in 100% of homes and was detected in a majority of dust samples. The number of target chemicals detected per sample ranged from 13 to 28 for air samples (mean 19) and from 6 to 42 for dust samples (mean 26). Figures 1 and 2 show concentration distributions for the most commonly detected compounds in air and dust, grouped by chemical class; and chemicals and pesticides detected at highest concentrations are summarized in Table 4.

Most Abundant Compounds. *Phthalates.* Phthalates, many of which have been characterized as EDCs due to their ability to interfere with androgen action (*28*, *29*), were detected at the highest concentrations in both air and dust, although different phthalates dominated the two media. In indoor air, diethyl phthalate (DEP) and di-*n*-butyl phthalate (DBP) were present at the highest concentrations. The 90th percentile concentrations in indoor air were 1560 and 426 ng/m^3 for DEP and DBP, respectively. These are the same phthalates observed to be most abundant in human urine samples reported by the CDC for a cross-section of U.S. adults (*30*). In dust, diethyl hexyl phthalate (DEHP) and butyl benzyl phthalate (BBP) were the chemicals detected at the highest concentrations. The 90th percentile concentrations for these phthalates in dust were 854 and 277 μg/g dust, respectively. In addition, high concentrations of an unidentified phthalate with >7 carbon chain were detected (approximate concentration range 4−800 μg/g), and this compound interfered with detection of diisononyl phthalate.

In the absence of data, most estimates of exposure to phthalates have concluded that inhalation is not an important route of exposure (*29*). However, the high indoor air concentrations detected here and the correspondence between phthalates abundant in air and urine suggest that inhalation exposures may be important. While exposure estimates based on ambient air concentrations may appear to be an insignificant portion of total exposure, actual exposure by inhalation is likely to be higher than would be estimated on the basis of ambient indoor air concentrations because phthalate-containing product use may result in

TABLE 4. Most Abundant Chemicals

Ten Chemicals with Highest 90th Percentile Concentrations	
air (ng/m^3)[a]	dust (μg/g)[a]
diethyl phthalate (1,600) *100*	bis(2-ethylhexyl) phthalate (854) *100*
o-phenylphenol (440) *100*	benzyl butyl phthalate (277) *100*
di-*n*-butyl phthalate (430) *100*	di-*n*-butyl phthalate (43.9) *98*
4-nonylphenol (230) *100*	nonylphenol diethoxylate (18.9) *86*
bis(2-ethylhexyl) phthalate (210) *68*	bis(2-ethylhexyl) adipate (16.6) *100*
diisobutyl phthalate (150) *100*	*trans*-permethrin (16.5) *53*
benzyl butyl phthalate (68) *44*	piperonyl butoxide (15.1) *66*
4-*tert*-butylphenol (43) *100*	diethyl phthalate (10.8) *89*
nonylphenol monoethoxylate (41) *95*	nonylphenol monoethoxylate (8.55) *86*
bis(2-ethylhexyl) adipate (22) *99*	*cis*-permethrin (7.04) *45*

10 Pesticides with Highest 90th Percentile Concentrations	
air (ng/m^3)[a]	dust (μg/g)[a]
o-phenylphenol (440) *100*	*trans*-permethrin (16.5) *53*
heptachlor[b] (19) *44*	piperonyl butoxide (15.1) *66*
propoxur (16) *49*	*cis*-permethrin (7.04) *45*
γ-chlordane[b] (12) *53*	methoxychlor[b] (3.38) *54*
chlorpyrifos (12) *38*	4,4′-DDT[b] (3.19) *65*
pentachlorophenol[b] (10) *58*	pentachlorophenol[b] (2.42) *86*
diazinon (9.0) *40*	chlorpyrifos[b] (1.87) *18*
α-chlordane[b] (8.8) *51*	carbaryl (1.72) *43*
chlorothalonil (3.4) *17*	propoxur (1.70) *42*
3,5,6-trichloro-2-pyridinol (1.1) *13*	bendiocarb (1.11) *12*

[a] Percent detection in italics. [b] Indicates banned or restricted-use pesticide (at time of sample collection).

personal air concentrations that are much higher than ambient concentrations.

Alkylphenols. Alkylphenols, particularly 4-nonylphenol (4-NP) and its mono- and diethoxylates, were also among the most abundant compounds detected (4-NP 90th percentile in air, 230 ng/m^3; NP2EO in dust, 18.9 μg/g) (see Tables 2 and 3 and Tables S1 and S2 in Supporting Information). In addition to being present at high concentrations relative to other compounds detected, 4-NP was detected in 100% of indoor air samples. These data provide the first evidence that 4-NP is an important contaminant of indoor air, although lower concentrations have been reported in outdoor air (*31*). This result contrasts with conclusions by others that 4-NP is not volatile and would be unlikely to be a significant air contaminant (*32*, *33*). Nonylphenol, octylphenol, and their small ethoxylates have been identified as EDCs because of their ability to mimic estrogen action (*24*).

Parabens and Phenols. Several other estrogenic compounds, presumably originating from consumer products, were commonly detected in air. These include the disinfectant *o*-phenyl phenol (90th percentile, 440 ng/m^3), 4-*tert*-butyl phenol (90th percentile, 43 ng/m^3), and methyl paraben (90th percentile, 11 ng/m^3).

Pesticides. Pesticides detected at the highest concentrations include the currently used pesticide permethrin and the synergist piperonyl butoxide (PBO) in dust (Table 4). Other pesticides detected at relatively high concentrations include heptachlor, propoxur, chlordane, chlorpyrifos, and pentachlorophenol in air and methoxychlor, DDT, pentachlorophenol, chlorpyrifos, carbaryl, and propoxur in dust (Table 4, Figures 1 and 2). The 90th percentile concentrations for these pesticides ranged from 10 to 19 ng/m^3 in air and from 1.7 to 17 μg/g in dust. The prevalence indoors of pesticides that have been banned or restricted for many years, such as DDT, chlordane, heptachlor, methoxychlor, dieldrin, and pentachlorophenol, suggests that degradation indoors is negligible. This observation is further supported by the abundance of DDT in dust relative to its degradation product DDE (Figure 2).

Brominated Flame Retardants. PBDEs, which are flame retardants widely used in foams and other plastics, were detected in dust samples with a concentration distribution similar to the carcinogenic PAHs, benzo[*a*]pyrene, and benz-[*a*]anthracene (Figure 2), with 90th percentile concentrations ranging from 0.7 to 4.1 μg/g dust. We targeted tetra- and pentabrominated BDEs, which originate from polyurethane foams. PCBs, which have a similar mechanism of endocrine toxicity to PBDEs, were also detected in air and dust samples but at somewhat lower concentrations (Figure 2).

Another notable finding in this study was detects of the mutagen and carcinogen 2,3-dibromo-1-propanol (*34*) in both dust and air samples. This chemical is described as an intermediate in the production of the flame retardant TRIS (tris(2,3-dibromo-1-propyl)phosphate), which was banned in 1977, and also as a urinary metabolite of TRIS (*34*). We detected it in both indoor air (9% of 85 homes with detects and a wide range of concentrations with maximum of 200 ng/m^3) and house dust (6% of 88 homes with maximum of 42.8 μg/g dust).

Toxicity Data and Implications. For over 30 EDCs that we detected in indoor air and dust, including alkylphenols, PBDEs, 2,3-dibromo-1-propanol, parabens, and some phenols (e.g., bisphenol A, 4-*tert*-butyl phenol), our measurements are the first that we know of in these media. In some cases, these are the first we are aware of in any media. The exposure data reported here provide a basis for prioritizing EDCs for more comprehensive toxicity testing and for assessing potential risks once toxicity testing is complete. The compounds listed in Table 4, for example, provide a starting point for prioritization based on chemical concentrations, and consideration of preliminary toxicity data would suggest prioritization of additional compounds, such as the brominated flame retardants.

Comparison with Available Government Risk Evaluations. We sought to compare our detected concentrations with risk-based media concentrations that have been developed for air, and we compared our dust concentrations with residential soil risk-based concentrations, which are designed to protect a small child from toxicant exposure via soil ingestion.

Of the measurements that we were able to compare with EPA risk-based concentrations (*35*, *36*), measurements in our study exceeded risk-based concentrations in at least one home for DEHP, PCBs, DDT, chlordane, dieldrin, heptachlor, and lindane (dust and air) and for benzo[*a*]pyrene, benz-[*a*]anthracene, chlorpyrifos, dicofol, and pentachlorophenol (dust only). However, because these EPA guidelines do not consider endocrine effects, these comparisons are of limited usefulness. In addition, we were unable to locate any risk-based media concentrations for 28 of the chemicals that we detected in homes in this study, including alkylphenols, parabens, some phthalates and pesticides, and most of the phenolic compounds, so we cannot evaluate the potential health risks associated with the detected concentrations using these types of data. Given the evidence of exposure reported here for EDCs, it is important to note the limitations in available toxicity data so that further work in this area can be prioritized. Furthermore, given that we detected so many EDCs and others report that mixtures at sub-threshold concentrations act additively (*4*, *7*), our results provide additional evidence that consideration of mixtures is important in assessing EDC exposure.

Indoor Sources. For virtually all the target compounds where comparison data are available, levels detected in indoor air are higher than those reported by others for outdoor air (*9*, *12*, *14*, *22*, *37*, *38*), confirming that most of these chemicals

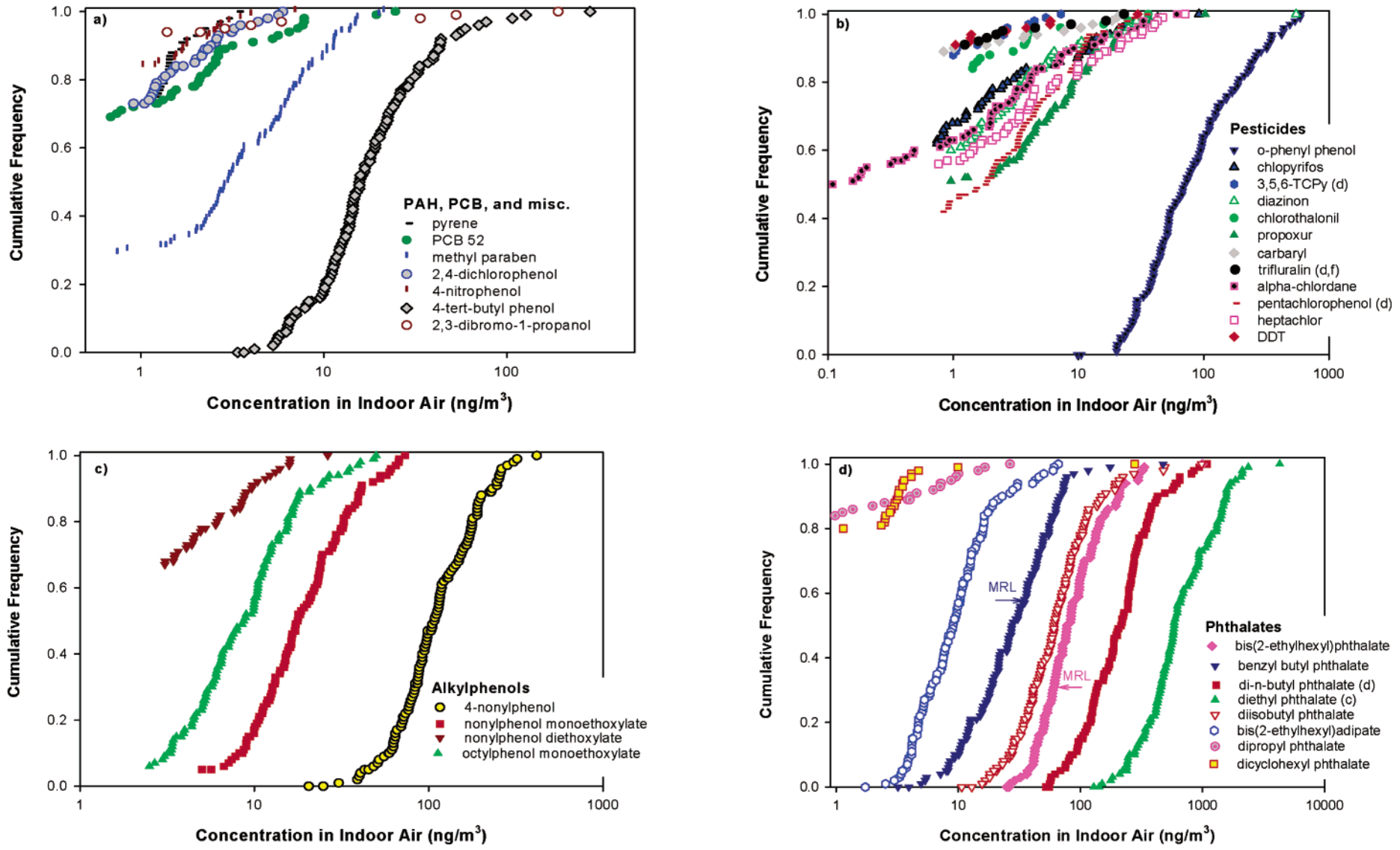

FIGURE 1. Cumulative frequency distributions of measured levels of the most frequently detected chemicals in indoor air samples from 120 homes. Distributions are truncated at the reporting level, and concentrations are shown on a log scale on the *x*-axis. Footnotes for specific chemicals refer to notes in Table 2. Chemicals are grouped into classes: (a) PAHs, PCBs, and misc.; (b) pesticides; (c) alkylphenols; and (d) phthalates.

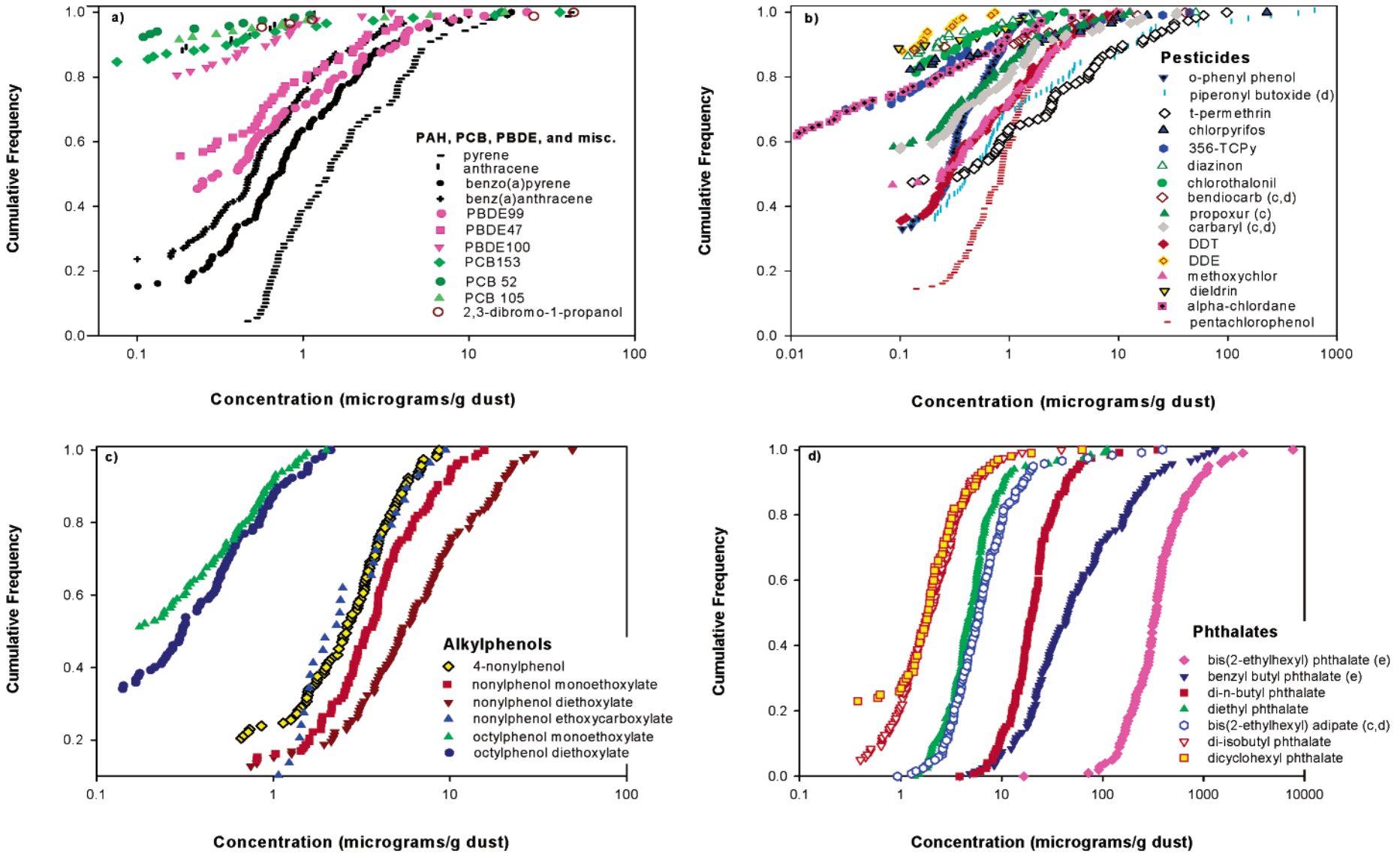

FIGURE 2. Cumulative frequency distributions of measured levels of frequently detected chemicals in indoor dust samples from 120 homes. Distributions are truncated at the reporting level, and concentrations are shown on a log scale on the *x*-axis. Footnotes for specific chemicals refer to notes in Table 3. Chemicals are grouped into classes: (a) PAHs, PCBs, PBDEs, and misc.; (b) pesticides; (c) alkylphenols; and (d) phthalates.

originate in household products and materials. For example, one study of outdoor air in urban New York/New Jersey reported that average levels of 11 nonylphenol isomers combined were in the range of 10 ng/m^3 (*31*), while in our study the average concentration of 4-nonylphenol was 130 ng/m^3. Median outdoor concentrations of DBP were reported to be 18 ng/m^3 in a suburban California location (*18*) as compared with a median indoors in our study of 210 ng/m^3. While environmental regulatory programs have traditionally focused on outdoor ambient air, surface water, drinking water, and hazardous industrial processes, little attention has been paid to the home environment.

Regional Variation. Comparison of these data with other studies can provide insights about regional, demographic, and temporal patterns in exposure to these compounds. Where comparison data were available (primarily for pesticides, PCBs, PAHs, and some phthalates), levels detected in our study (on Cape Cod, MA) are similar to levels reported elsewhere—especially for air concentrations (*9*, *12*−*14*, *18*, *39*−*42*). Some regional differences observed for dust levels were reported in ref *40*. Briefly, dust concentrations of PAHs on Cape Cod appear lower than on Long Island, NY, but higher than in many other regions of the United States (Iowa; Seattle, WA; Los Angeles, CA); levels of PCBs in dust appear higher on Cape Cod than in Iowa and Los Angeles, CA, but similar to or lower than Seattle, WA; Detroit, MI; and Long Island NY; levels of pesticides in Cape Cod house dust appear higher than other regions for DDT, carbaryl, chlordane, methoxychlor, pentachlorophenol, and propoxur; and levels appear lower than other regions for diazinon and permethrin. For chlorpyrifos and *o*-phenyl phenol in dust, Cape Cod levels are higher than some regions and lower than others (*40*). Compared with PBDE levels in indoor dust reported from Germany (*43*) and the United Kingdom (*44*), PBDE levels reported here were 5−10 times higher. These comparisons must be interpreted with caution considering differences between studies in methods of sample collection and demographics of study populations.

Individuals with Highest Measurements. As is typical for environmental measurement data, the exposure distributions for most analytes are highly skewed. Thus, the maximum concentration detected is often much higher than even the 90th or 95th percentiles. This finding suggests that (for each analyte) a small proportion of the population (e.g., 1%) receives substantially higher exposures than the majority. Since most health-based standards are derived to protect the 90th or 95th percentile-exposed individual in a population, these standards may not be adequately protective of the highest exposed 1% of the population who have exposures that are substantially higher, sometimes by orders of magnitude. For example, the maximum air concentrations for DDT and diazinon were 58 and 61 times higher than the 90th percentile concentrations, respectively. In dust samples, maximum concentrations for diazinon, chlorpyrifos, and PCB 153 were 228, 122, and 89 times higher than 90th percentile concentrations, respectively. The flame retardant 2,3-dibromo-1-propanol, while it was detected in fewer than 10% of the homes, was detected over a very large concentration range—the maximum detected concentrations in both air and dust were at least 200 times higher than the MRL.

Tools for Health Studies and Source Identification. There is great interest in conducting epidemiologic studies to evaluate effects of exposures to EDCs, but limitations in exposure assessment tools have impeded progress. Our study was designed in part to develop improved exposure tools for EDCs and to address some key data gaps—for example, these data provide a basis for prioritizing the development of exposure biomarkers. Data on key sources of these compounds and factors that affect exposure levels allow for further development of exposure assessment and source reduction tools and provide insight into exposure characterizations in health studies that have already been completed.

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Supporting Information Available

More detailed summary statistics for these data and a list of chemicals that were not detected in this study (Tables S1 and S2). This material is available free of charge via the Internet at http://pubs.acs.org.

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