Distribution Agreement

Phthalate exposures in California over 2007-2018: estimates from municipal wastewater discharge reports

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A thesis submitted to the Faculty of the
Rollins School of Public Health of Emory University
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Abstract

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Phthalates are a useful group of chemicals with over 470 million pounds produced or imported in the United States each year. Phthalates primary applications are as plasticizers (making plastics soft). Some phthalates are even used in personal care products like fragrances or cosmetics. However, some phthalates like di(2-ethylhexyl) phthalate were shown to produce adverse health effects in male infants. Due to the ubiquitous use and potential health effects, it is of public health importance to estimate population level exposures to phthalates. To efficiently estimate population wide exposures to phthalates, this paper uses wastewater discharge reports. Contaminants measured in wastewater is made publicly available by the United State Environmental Protection Agency. 6 phthalates were considered in this analysis – butyl benzyl, di-n-octly, di (2-ethylhexyl), dibutyl, diethyl, and dimethyl phthalate as these are listed on the United States Environmental Protection Agency's list of 126 priority pollutants. Restricted maximum likelihood mixed-effects linear regression models were utilized to explain temporal and spatial heterogeneity. Butyl benzyl phthalate per-capita geometric mean mass discharge appears to increase. Decreases in per-capita geometric mean masses were seen in di(2-ethylhexyl) phthalate and diethyl phthalate over the 11-year period.

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Introduction:

Phthalates are a broad class of chemicals that are used in a wide variety of products and produced in high volumes – over 470 million pounds in the United States alone **[1]**. This paper will focus on 6 phthalates that are listed on the United States Environmental Protection Agency's (EPA) priority substance list: butyl benzyl phthalate (BBP), di(2-ethylhexyl) phthalate (DEHP), di-n-octyl phthalate (DnOP), dibutyl phthalate (DBP), diethyl phthalate (DEP), and dimethyl phthalate (DMP) **[2]**. These 6 phthalates are used to produce a range of products like polyvinyl chloride products – vinyl flooring **[3]**, food conveyor belts **[3]**, hosing **[4]**, wallpaper **[5]**, medical devices like blood bags **[5]**, packing film **[6, 7]**, and personal care products like fragrances and cosmetics (**Table 1**)**[6, 8]**. Exposures to these phthalates occur through multiple routes with food being the primary source for BBP **[3]**, DEHP **[5]**, DBP **[7]**, and DEP **[6]**. Medical products and indoor air are important sources for DEHP **[5]** while cosmetics and personal care products are important sources of DBP and DEP **[6, 7]**. DnOP is lacking in exposure data but some studies have quantified DnOP in household dust **[4]**. DMP represents less than 1% of phthalate use in the United States, but is used as a solvent in a variety of products like paints, chemicals, fragrances and cosmetics **[8]**. Due to the frequent use of phthalates in everyday products, there is some concern for health effects **[1]**. The WHO has classified phthalates as endocrine disrupting chemicals **[9]**. The National Toxicology Program reported some to serious concern of adverse health effects for male infants exposed to DEHP **[5]**. The NTP also reported clear evidence of developmental toxicity in laboratory animals for BBP **[3]** while DBP could possibly cause developmental effects at high exposures **[7]**.

Estimating population-level exposures to these 6 phthalates has public health significance. Currently the Centers for Disease Control and Prevention includes these 6 phthalates in its national biomonitoring program **[10]**. The state of California also conducts biomonitoring of phthalates through the Biomonitoring California initiative **[11]**. This project consolidates biomonitoring results from studies conducted in California, which may not be population-representative.

Another way to estimate population-level exposures though is through wastewater analysis. Wastewater analysis offers an efficient way to estimate population wide exposures to many chemicals including phthalates **[12]**. Typically, wastewater analysis studies include systematic sampling of wastewater influent and effluent to track markers of exposure of public health or environmental importance **[13]**. For example, a large European study estimated illicit drug use across 21 countries using wastewater analysis **[14]**. Another study used wastewater analysis to estimate phthalate exposures in major Chinese cities as well **[12]**. This paper attempts a similar approach to wastewater analysis to explain temporal and spatial trends in phthalate exposures over an 11-year period in the state of California. Rather than direct sampling at wastewater facilities, wastewater data collected by the EPA under the Clean Water Act was used.

We will discuss how the EPA wastewater data is collected, the creation of service area population dataset, estimate average per capita exposure to 6 phthalates listed on EPA's priority substance list, and describe trends seen in this estimated average per capita exposure. Finally, we will attempt to compare results from this analysis to the biomonitoring reports from the state of California.

Materials and Methods:

Wastewater data

The Clean Water Act's National Pollution and Discharge Elimination System (NPDES) program requires all wastewater dischargers to hold a permit, monitor wastewater dischargers and report monitoring data through Discharge Monitoring Reports (DMR) **[15]**. Municipal wastewater facilities in California must sample and analyze wastewater for phthalates at least once per year or more as described in their NPDES permit **[16]**. Phthalates are analyzed using EPA method 606 **[17]**. Laboratories conducting the wastewater sample analysis are certified by the California Department of Public Health Environmental Laboratory Accreditation Program **[16]**. Once the analysis is complete, the municipal wastewater facility completes the DMR, reporting phthalates discharged as pounds per year and submits this form to the EPA **[16]**. The data from DMRs are made publicly available through the EPA's Enforcement and Compliance History Online (ECHO) portal **[18]**.

The DMR data is accessed through the Water Pollution Search database found in ECHO **[19]**. Discharges from municipal wastewater facilities (Standard Industrial Classification Code 4952 **[20]**) in the state of California were downloaded for each year (2007-2018) as Microsoft Excel™ files. These annual files then appended in Stata 15.1 IC **[21]**. The appended dataset included all pollutants reported, so Chemical Abstract Service (CAS) Number (**Table 1)** was used to identify the 6 phthalates and all other pollutants were dropped resulting in the final dataset. These 6 phthalates were considered because they are listed on the EPA's priority pollutant list **[2]**.

Population

The population size served by each municipal wastewater facility in each year was obtained from at least one of four distinct data sources: facility reports, direct correspondence with facility operators; the California Wastewater Survey; or, if a facility served only its city population, the California's Department of Finance's yearly census data. The supplemental file reports the source for each facility. Additional calculations were required to determine service population for a few municipal wastewater facilities. These calculations and descriptions can be found in the appendix.

The California Water Board conducts a yearly survey of wastewater facilities to assess sewer rates and connections **[22]**. Participation is voluntary with roughly 61% of facilities participating in 2016-2017 **[22]**. This survey includes service area populations from 2012 to 2017. The survey was not conducted in 2009-2010, 2010-2011 **[22]**. Previous surveys do not report service area populations in counts but rather as an index (1,000-9,999 = pop index 2) so pre-2009 surveys were not used in this analysis.

<u>Biomonitoring California</u>

Biomonitoring California is a state wide initiative to report environmental chemicals in Californians **[11]**. Because the project relies on studies conducted in the state of California, participants' samples come from the individual studies which may not be population-representative **[11]**. This initiative reported biomarkers of phthalates for adults in the Central Valley Region (2011-2013), firefighters in southern California (2010), and 3 distinct cohorts of pregnant women from northern California (2007), Salinas Valley (2005), and San Francisco (2010) **[23]**. Urine samples were taken from these participants and analyzed for phthalate metabolites by using solid phase extraction with high performance liquid chromatography tandem mass spectrometry (SPE-HPLC-

MS/MS) **[24]**. The limit of detection for phthalates was 0.190 nanogram per milliliter **[23]**. Geometric means were not calculated for metabolites detected in fewer than 65% of a study's participants. The metabolites and their parent phthalates are reported in **Table 4.**

<u>Data analysis and model fitting</u>

Phthalate discharges were reported in mass units per year (pounds/year). The reported mass discharge was divided by the facility population in the observed year and then natural log-transformed. We recoded years for analysis as 'years since 2007'.

Restricted maximum likelihood mixed-effects linear regression model of log-per capita municipal wastewater facility phthalate discharges regressed on year

We first estimated linear mixed effects models by restricted maximum likelihood with a random intercept for facility and fixed effect for year since 2007, with unstructured covariance matrix for the random effects. A random slope within facility by year was then added to allow for possible heterogeneity in temporal trends across facilities. Q-normal plots of the standardized residuals were generated to test fit of model. Results were back-transformed to original scale and plotted as geometric means with units of pounds per year per person. Data and statistical analysis were carried out in Stata 15.1 IC **[21]**.

Inverse variance-weighted fixed-effects meta-regression of Biomonitoring California reported geometric means for phthalates

All 5 reports from Biomonitoring California were downloaded as a single Microsoft Excel™ file. Chemicals with only 1 observation per year were dropped. Studies performed over multiple years were assigned year that the first sample was collected. The reported geometric means were natural log transformed then regressed on year using variance weighted least squares models. Results are reported as geometric mean ratios.

Results and Discussion

BBP

From 2007 to 2018, BBP was reported a total of 45 times with a minimum of 1 reported discharge in 2018 and maximum of 9 reported discharges in 2007 (**Table 1)**. 25 unique municipal wastewater facilities reported discharges of BBP over the 11-year period. On average, each facility reported 1.8 discharges of BBP with a minimum of 1 and maximum of 6 reported discharges by a single facility. The fixed effect on year was 0.022 (95% Confidence Interval (CI): -0.122, 0.166) (**Table 5, Figure 2**). The random intercept [0.207 (95% CI: 0.006, 7.67)] and random slope [0.054 (95% CI: 0.020, 0.150)] were smaller than the residual [0.708 (95% CI: 0.370, 1.36)]. The random intercept describes spatial patterns, that is the variance between facilities. The random slope describes temporal patterns or the variance at a specific time. The residual is unexplained variance within a facility. The fixed effect indicates a 2.2% (95% CI: -11.5, 18.0) increase in per-person geometric mean mass discharges of BBP per year. Most of the variance in this upward trend of BBP was not explained by variations between facilities (random intercept) or temporal variations (random slope). The large residual indicates that the trend for BBP discharges is mostly explained by the variance within facilities.

DEHP

DEHP was the most reported discharged phthalate in this analysis with 371 reported discharges from 2007 to 2018 (**Table 1**). The minimum number of reported discharges was 24 in 2018 while the maximum number of reported discharges was 52 in 2008. 112 unique municipal wastewater facilities reported discharges of DEHP with an average of 3.3 reported discharges per facility. The minimum reported discharges from a single facility was 1 and the maximum number of reported discharges from a single facility was 10. The fixed effect on year for DEHP was -0.069 (95% CI: -0.124, -0.015) (**Table 6, Figure 3)**. The random intercept [1.77 (95% CI: 1.14, 2.75)] and random slope [0.003 (95% CI: 0.0001, 0.119)] were smaller than the residual [2.06 (95% CI: 1.71, 2.49)]. The fixed effect indicates a 6.6% (95% CI: -11.6, -1.5) decrease in per-person geometric mean mass of DEHP discharged from municipal wastewater facilities. Temporal variations explain very little of the decreasing trend of per-person amount of DEHP being discharged. Between facility, spatial, variance does explain some of this trend in DEHP discharges, but the larger residual indicates that most of the trend is explained by within facility variations.

DEP

There were 50 total reported discharges of DEP, a minimum of 1 discharge in 2018 and a maximum of 11 discharges in 2009 (**Table 1)**. 35 unique municipal wastewater facilities reported discharging DEP. The average number of reported discharges per facility was 1.4 with a minimum of 1 reported discharge from a single facility and a maximum of 6 reported discharges from a single facility. The fixed effect on year for DEP was -0.413 (95% CI: -0.612, -0.213) (**Table 7, Figure 4**). The random intercept [1.02 (95% CI: 0.165, 6.29)] and random slope [0.070 (95% CI: 0.019, 0.260) were smaller than the residual [1.58 (95% CI: 0.807, 3.09)]. The fixed effect indicates a 33.8% (95% CI: -45.8, -19.2) decrease in per-person geometric mean mass of DEP discharge from facilities. The variance between facilities does explain some of the trend of DEP discharges, but within facility variance is still larger and explains more of the trend of DEP discharges.

Wastewater and Biomonitoring Trends

Per-person geometric mean masses DEHP (6.6%), and DEP (33.8%) are decreasing each year in California from 2007 to 2018. In contrast, per person geometric mean masses of BBP is increasing slightly by 2.2% each year. The variance within facilities explains most of the trends seen for BBP, DEHP, and DEP. For DEHP and DEP, spatial patterns – the random intercept or between facility variations – do explain some of the decreasing trend of per person DEHP and DEP discharges. The random intercept for BBP explains less than half of the total variance in the increasing trend. Temporal variations were very small for all 3 phthalates and did not explain the trends seen in phthalate discharges.

The wastewater study is consistent with the biomonitoring program except for BBP. Mono-ethyl phthalate, the metabolite of DEP, geometric mean ratio decreased by 0.94 per year. Mono-benzyl phthalate, metabolite of BBP, geometric mean ratio decreased by 0.91 per year and Mono-(2-ethyl-5-carboxypentyl) phthalate, metabolite of DEHP, geometric mean ratio by 0.89 per year. Mono-butyl phthalate, metabolite of DBP and BBP, geometric mean ratio decreased by 0.88 per year. The biomonitoring reports are limited because the volunteers were recruited due to their exposure (firefighters) or susceptibility (pregnant women) which do not necessarily reflect the general population of California.

This wastewater analysis does contain limitations. First, relying on the publicly available dataset made it difficult to quantify measurement error of the laboratories analyzing facilities' wastewater samples. Second, low observation numbers increase the uncertainty in our estimates. We were unable to predict estimates for DnOP, DBP or DMP because of low observations including some years with 0 reports of measured DnOP or DMP in wastewater. Third, the population data was collected from a variety of sources with imperfect estimates of service population. For example, facilities requiring census data do not account for citizens using septic tank systems and thus who do not contribute to the wastewater stream. Lastly, we were unable to discern if any of the phthalates came from non-human sources such as runoff or disposal of products into the sewage system. These additional sources could overestimate the average exposure. This analysis was useful in harnessing publicly available data to estimate population level exposures to 3 phthalates: BBP, DEHP, and DEP. We recommend further collaboration with wastewater facilities to improve estimates of populations served by municipal wastewater facilities. Additionally, further research should use direct sampling to provide finer time scale resolution and more accurate measure of phthalates.

Appendix

Tables

Table 1: 6 reported phthalates, corresponding Chemical Abstract Numbers (CAS) and primary uses of each phthalate. The uses are not a comprehensive list [5-10].

Phthalate	CAS Number	Uses
Butyl Benzyl Phthalate	85-68-7	Vinyl tile; plasticizer for PVC in food conveyor belts, carpet tile, artificial leather
Di-n-octyl Phthalate	117-84-0	Ingredient for phthalate mixture C6-10 phthalate; flooring, carpet tiles, tarps, garden hoses
Di(2-ethylhexyl) Phthalate	117-81-7	Flooring, wallpaper, auto upholstery, raincoats; medical devices such as blood bags and tubing
Dibutyl Phthalate	84-74-2	Latex adhesives, plasticizer for cellulose plastics, solvent for dyes
Diethyl Phthalate	84-66-2	Plasticizer for packaging film, solvent or vehicle for fragrances in hair sprays, nail polishes, and perfumes
Dimethyl Phthalate	131-11-3	Paints, rubber; solvent for cosmetics, creams, perfumes, shampoos

Table 2: Number of reported discharges of 6 phthalates from municipal wastewater facilities in California by year (2007 to 2018).

Year	**BBP**	**DEHP**	DNOP	DBP	**DEP**	DMP	All
2007	8	32	1	6	8	4	59
2008	5	43	2	9	4	1	64
2009	3	34	0	5	7	2	51
2010	3	36	0	5	3	0	47
2011	4	38	0	8	7	0	57
2012	3	33	4	6	7	0	53
2013	5	27	0	11	4	1	48
2014	4	29	3	8	2	3	49
2015	5	26	2	5	4	1	43
2016	2	31	2	3	1	2	41
2017	2	22	2	3	2	2	33
2018	1	20	0	5	1	0	27
Total	45	371	16	74	50	16	572

Table 3: Per person average of pounds of phthalates discharged from municipal wastewater facilities in California (2007-2018).

Phthalate	Geometric Mean Mass (95% CI) (pounds per year per person)
BBP	$1.72 x10^{-4}$ ($8.11 x10^{-5}$, $3.66 x10^{-4}$)
DEHP	$6.43 x10^{-4}$ ($4.29 x10^{-4}$, $9.58 x10^{-4}$)
DNOP	$1.19\ x10^{-5}$ ($1.2 x10^{-6}$, $1.2 x10^{-4}$)
DBP	$1.95 x10^{-4}$ ($7.9 x10^{-5}$, $4.8 x10^{-4}$)
DEP	$2.86 x10^{-4}$ ($1.10 x10^{-4}$, $7.42 x10^{-4}$)
DMP	$1.68 x10^{-4}$ ($2.1 x10^{-5}$, $1.3 x10^{-3}$)

Table 4: Average mass results of fixed-effects inverse-weighted meta-regressions per year of reported phthalate metabolites from California biomonitoring study.

Metabolite	Phthalate	Geometric Mean Ratio (95% CI)
Mono-benzyl phthalate (MBzP)	BBP	0.91 (0.84, 0.98)
Mono-(3-carboxypropyl) phthalate (MCPP)	DNOP	1.17 (1.09, 1.26)
Mono-(2-ethyl-5-carboxypentyl) phthalate (MECPP)	DEHP	0.89 (0.83, 0.95)
Mono-ethyl phthalate (MEP)	DEP	0.94 (0.90, 0.98)
Mono-n-butyl phthalate (MnBP)	DBP, BBP	0.88 (0.86, 0.91)

Table 5: Restricted maximum likelihood mixed-effects linear regression model of log-per capita municipal wastewater facility BBP discharges regressed on year

Fixed Effects	Coefficient (95% Confidence Interval)	Standard Error
Intercept	-8.96 (-9.47, -8.44)	-34.13
Year	0.022 (-0.122, 0.166)	0.073
Random Effect	Random effect variance estimate (95% Confidence Interval)	Standard Error
Random slope on year	0.054 (0.020, 0.150)	0.028
Random intercept	0.207 (0.006, 7.67)	0.382
Residual	0.708 (0.370, 1.35)	0.235

Table 6: Restricted maximum likelihood mixed-effects linear regression model of log-per capita municipal wastewater facility di(2-ethylhexyl) phthalate discharges regressed on year

Fixed Effects	Coefficient (95% Confidence Interval)	Standard Error
Intercept	-7.35 (-7.75, -6.95)	0.204
Year	-0.069 (-0.124, -0.015)	0.028
Random Effects	Random effect variance estimate (95% Confidence Interval)	Standard Error
Random slope on year	0.003 (0.0001, 0.119)	0.006
Random intercept	1.77 (1.14, 2.75)	0.399
Residual	2.06 (1.71, 2.49)	0.198

Table 7: Restricted maximum likelihood mixed-effects linear regression model of log-per capita municipal wastewater facility diethyl phthalate discharges regressed on year

Fixed Effects	Coefficient (95% Confidence Interval)	Standard Error
Intercept	-7.90 (-8.73, -7.07)	0.102
Year	-0.413 (-0.612, -0.213)	0.423
Random Effects	Random effect variance estimate (95% Confidence Interval)	Standard Error
Random slope on year	0.070 (0.019, 0.260)	0.047
Random Intercept	1.02 (0.165, 6.29)	0.946
Residual	1.58 (0.807, 3.09)	0.540

Figures

Figure 1: Unfitted spaghetti plot of observed phthalate discharges from municipal wastewater facilities.

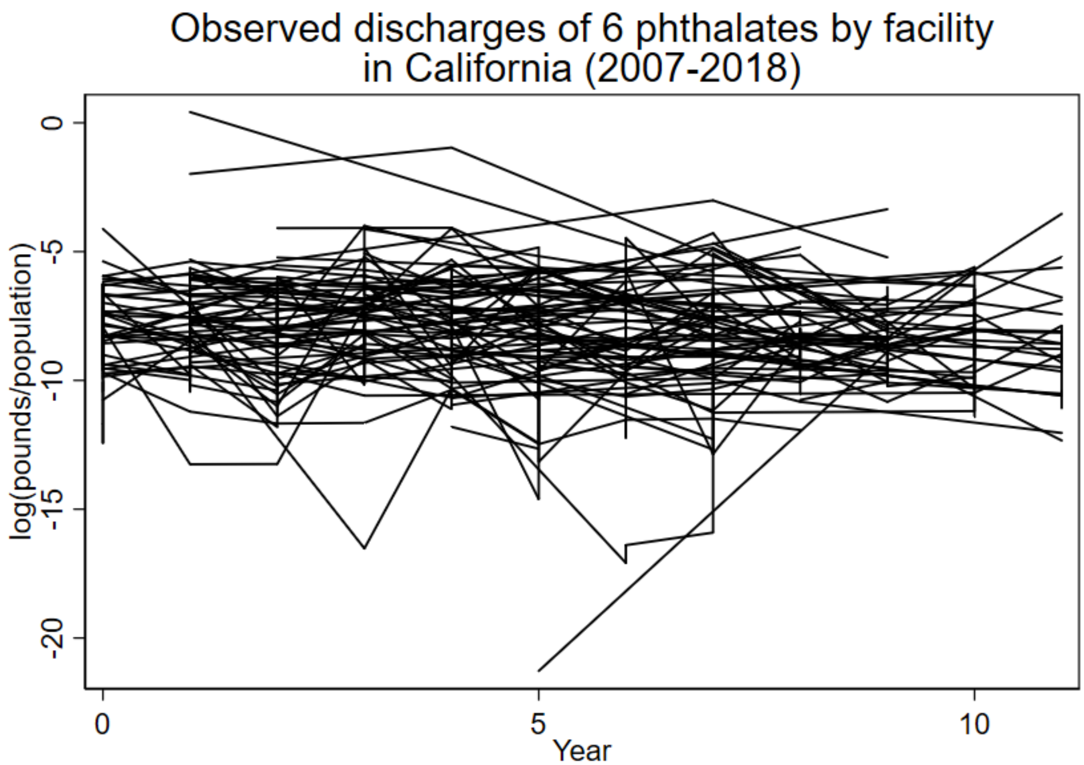


Figure 2: Trend in geometric mean mass of BBP discharged per person accounting for spatial and temporal random effects.

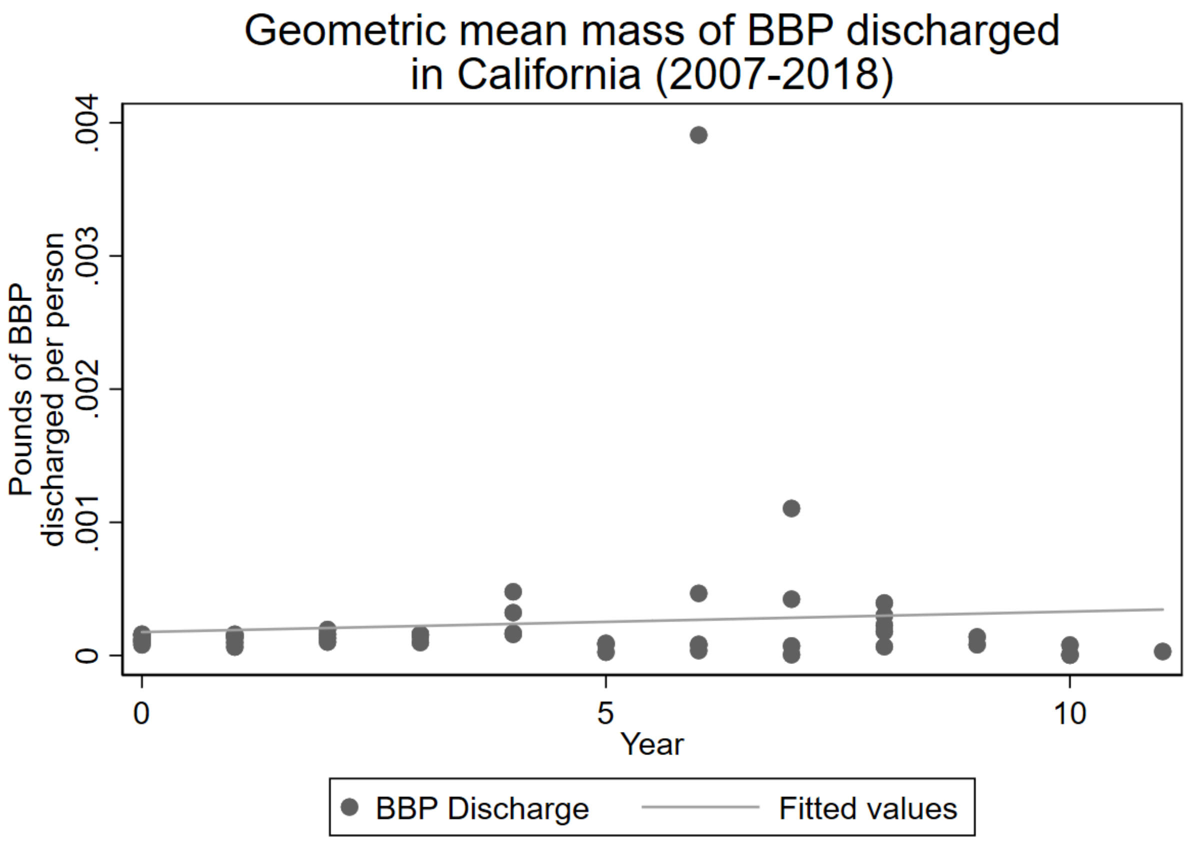


Figure 3: Trend in geometric mean mass of DEHP discharged per person accounting for spatial and temporal random effects.

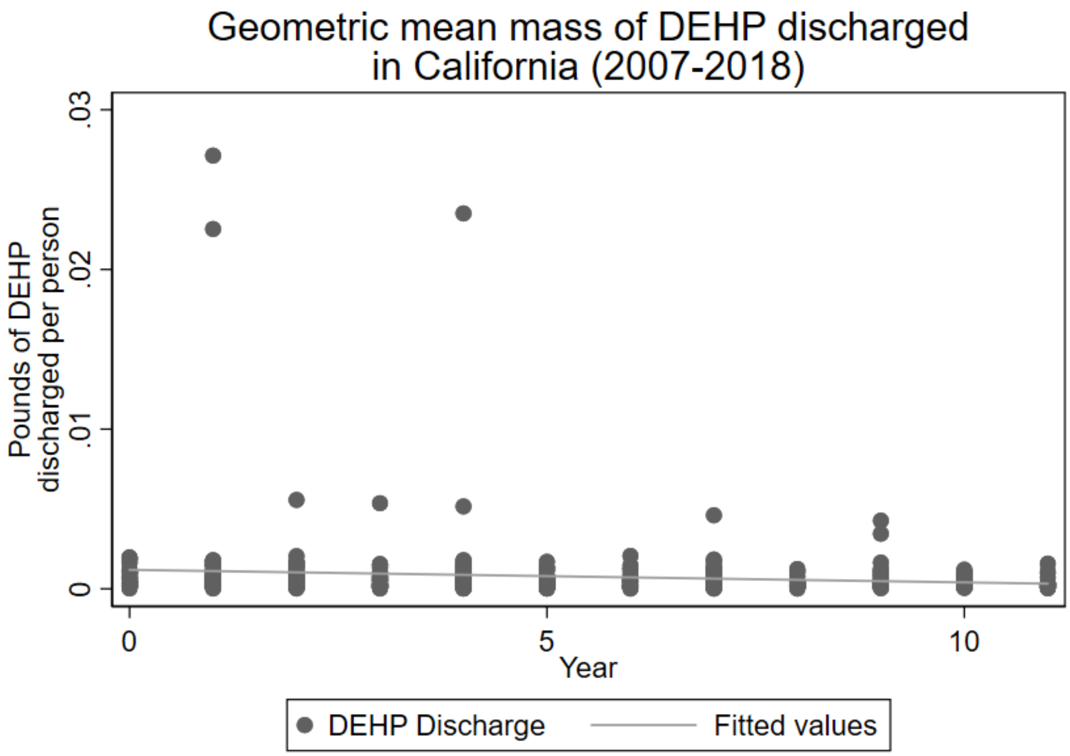
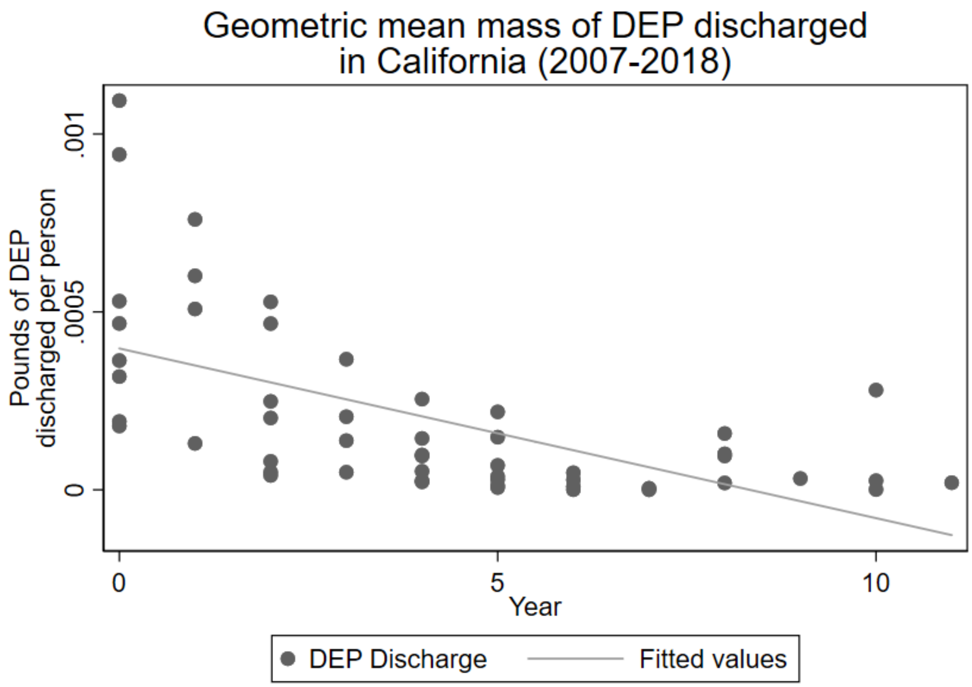


Figure 4: Trend in geometric mean mass of DEP discharged per person accounting for spatial and temporal random effects.



Population calculations

The City of Atwater Wastewater Treatment Plant includes the census population from the City on Winston because the city of Atwater treats wastewater from the city of Winton. The City of Tracy also treats wastewater from a neighbor city, Lathrop which was added to Tracy's census population. The Turlock facility includes the population of Keyes and Denair.

The Michelson Treatment Plant population treats 57% of the wastewater in its sanitary district. By multiplying the reported sanitary district population by 57% provides an estimate of the service population for Michelson Treatment Plant. The Orange County Treatment Plant 1 receives 60% of sanitary district's wastewater which serves 81% of the county. Census data provided the county population which was multiplied by the proportion living in the sanitary district (0.81) and then the flows directed to the treatment plant (0.60).

In Lake Elsinore population calculations required using the equivalent dwelling unit (EDU) which is a unit of wastewater equal to the amount generated by a single family **[25]**. So, 1 EDU is the amount of wastewater a single family would generate. To determine number of people per EDUs, the county population was divided by the number of residential EDUs in the county. Finally, this quotient was multiplied by the residential EDU in Lake Elsinore. North San Mateo Treatment Plant service population was derived from the product of the reported service connections and EDU over the time period.

The City and County of San Francisco Treatment Plant services 80% of the population of the sanitary district and this proportion was multiplied by census population. The Anderson Water Pollution Control Plant reported 93% of the county population serviced by the facility. Clear Creek and Redding facilities both serve a portion of the total population. Clear Creek reported serving 33% of Shasta county with Redding serving 50%.

City of Red Bluff only reported 2007 population but in 2018 showed past population growth of 0.4% which remained steady over the 11-year period. Lastly, the Linda County Wastewater Plant reported a 2005 population with a steady 0.0148% population growth through 2018. The population growth numbers were used to predict population for the years which had reported phthalate discharges.

residual within cluster variation dwarfs what is explained by baseline between places (spatial variation) and place specific time trends.

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