



ELSEVIER

Contents lists available at ScienceDirect

MethodsX

journal homepage: [www.elsevier.com/locate/mex](http://www.elsevier.com/locate/mex)

## Method Article

# A method for evaluating indoor phthalate exposure levels in collected dust



Yuxuan Zhao, Yuexia Sun\*, Qinghao Zhang, Qingnan Zhang, Jing Hou

Tianjin Key Laboratory of Indoor Air Environmental Quality Control, School of Environmental Science and Engineering, Tianjin University, China

## A B S T R A C T

This paper provides a method for evaluating indoor phthalate exposure levels in collected dust, including dust collection, chemical analysis and quality control. Indoor dusts were collected with a self-made equipment and pretreated by the Soxhlet extractor method. Six phthalates, i.e., diethyl phthalate (DEP), di-n-butyl phthalate (DnBP), di-isobutyl phthalate (DiBP), benzyl butyl phthalate (BBzP), di (2-ethylhexyl) phthalate (DEHP) and diisononyl phthalate (DiNP) in the dust were analyzed by gas chromatography mass spectrometry (GC-MS).

- Measures were taken to avoid background contamination of phthalates during sampling and chemical analysis to accurately evaluate the indoor phthalates exposure levels.
- Filter sock and self-made mouthpiece mounted on vacuum cleaner were made of nylon and aluminum respectively.
- Dust from surface of plastic products was avoided. The pretreatment of vessels and analysis of blank samples were conducted to reduce “noise” from background contamination.

© 2020 The Author(s). Published by Elsevier B.V.

This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>)

## A R T I C L E I N F O

**Method name:** A method for evaluating indoor phthalate exposure levels in collected dust

**Keywords:** Gas chromatography mass spectrometry (GC-MS), Indoor dust, Vacuum cleaner, Phthalate

**Article history:** Received 20 September 2020; Accepted 13 December 2020; Available online 17 December 2020

## Specifications Table

Subject Area	Environmental Science
More specific subject area	Indoor air quality
Method name	A method for evaluating indoor phthalate exposure levels in collected dust
Name and reference of original method	
Resource availability	

DOI of original article: [10.1016/j.scitotenv.2020.136965](https://doi.org/10.1016/j.scitotenv.2020.136965)

\* Corresponding author.

E-mail address: [yuexiasun@tju.edu.cn](mailto:yuexiasun@tju.edu.cn) (Y. Sun).

<https://doi.org/10.1016/j.mex.2020.101187>

2215-0161/© 2020 The Author(s). Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>)

## Method details

Phthalates are widely used in daily necessities, such as household furnishings, cosmetics, medical devices, children's toys, food packaging, cleaning materials and insecticides [2]. Phthalates are not chemically bonded to consumer products. They will with time escape by evaporating or by liquid extraction. The vapor pressure strongly depends on temperature. Evaporated during high temperature peaks, phthalates will likely condense at normal indoor temperatures and form small particles or adsorb to other particles. Therefore, phthalate concentration in indoor dust can provide a reliable indication of indoor contamination level [1]. Due to the wide use of plastic products and ubiquitous exposure to phthalates, how to avoid background contamination in the period of sampling and analyzing is a challenge. This paper aims to develop a method to estimate exposure of phthalate indoors through collected dusts.

### Dust collection indoors

Samples of settled dust were collected using filter socks made of nylon mounted on an aluminum mouthpiece connected to household vacuum cleaner. Dusts were from molding, window frames and/or shelves in children's bedroom [3]. Dusts from plastic surface were forbidden. Each sample contained at least 20–100 mg dust. After sampling, the filter sock was taken off and wrapped in aluminum foil. Samples should be stored at  $-20\text{ }^{\circ}\text{C}$  before analysis.

The filter sock is nylon with 1200 holes per square inch. The dimension of filter sock is shown in Fig. 1. A self-made aluminum mouthpiece was used to replace the original plastic mouthpiece, as shown in Fig. 2. One end of the mouthpiece was pressed into a flat mouth shape. The mouthpiece was 30mm in diameter and 190 mm in length. The gap in the flat part of the mouthpiece was 7 mm.

The detailed steps for dust collection were:

- Preparation: Wash hands. Prepare the vacuum cleaner, filter sock and aluminum mouthpiece.
- Attach the filter sock to the vacuum cleaner: Put the filter sock inside the vacuum cleaner pipe and fold out the edge of the filter sock. Attach the mouthpiece so that the filter sock sticks out a little beyond the mouthpiece, as shown in Fig. 3.
- Dust collection: Collect at least 20–100 mg dust from door/window/picture frames or shelves indoors. To get enough dust, dust can also be collected from skirting line, but not from the floor. Avoid collecting dust from plastic surface and plastic products.
- Sample preservation: Detach the mouthpiece and take off the filter sock. Fold the filter sock, roll it together and wrap it with aluminum foil. Label the sample and store it in the freezer at  $-20\text{ }^{\circ}\text{C}$  before analysis.
- Rinse the mouthpiece with clean water and dry it for the next vacuuming.

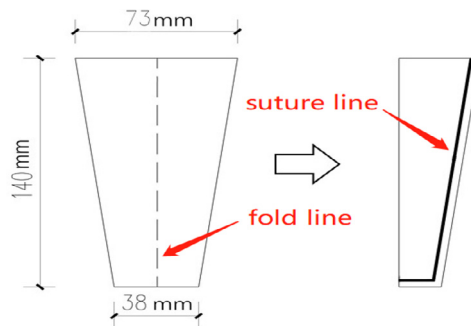


Fig. 1. Filter sock for dust collection.

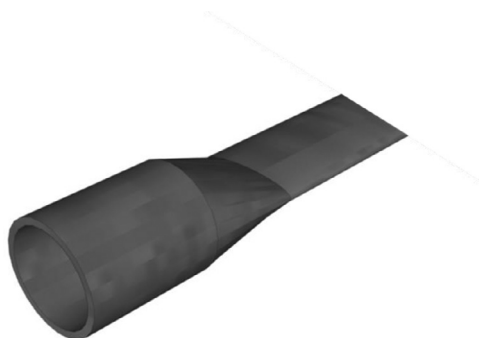


Fig. 2. Self-made mouthpiece.

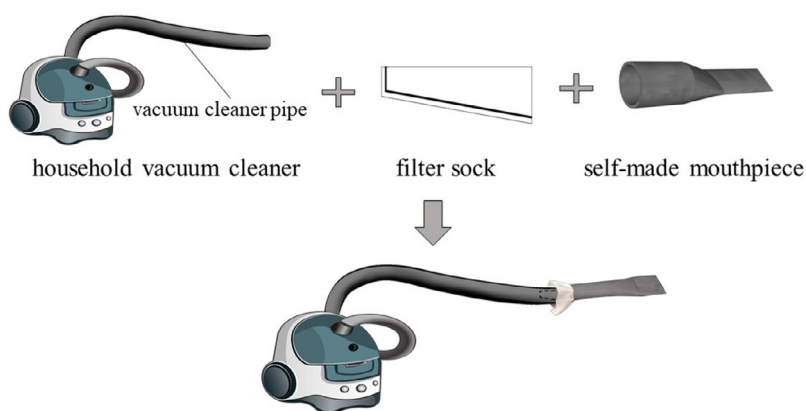


Fig. 3. Dust collection equipment.

### Chemical analysis and quality control

An internal standard method was used to obtain the phthalate concentration with gas chromatography mass spectrometry (GC-MS) analysis. Six phthalates in dust were analyzed: Diethyl phthalate (DEP), di-isobutyl phthalate (DiBP), di-n-butyl phthalate (DnBP), benzyl butyl phthalate (BBzP), di (2-ethylhexyl) phthalate (DEHP) and di-isobutyl phthalate (DiNP). Benzyl benzoate (BB), the internal standard, with a purity of  $\geq 99\%$  were purchased from AccuStandard Inc. (New Haven, CT).

#### *Pretreatment of vessels for chemical analysis*

Vessels made of glass were used in the chemical analysis. All the vessels were cleaned with chromic acid lotion to avoid environmental phthalate contamination. The glass vessels were soaked in chromic acid lotion for 2 h, then washed with distilled water followed by ultrapure water, and finally heated in a muffle furnace at 400 °C for 2 h. Note that plastics vessels were avoided during chemical analysis.

#### *Dust weighting and extraction*

Dust samples were put through sieves with an aperture of 0.25 mm to remove fabric and hair. A 100 mg dust sample was wrapped with filter paper. It was extracted in Soxhlet extractor for 6 h

**Table 1**

Retention time and quantitative ion for six phthalates and internal standard BB.

Phthalates	CAS registry number	Retention time (min)	Quantitative ion
DEP	84-66-2	9.7	149
DiBP	84-69-5	13.3	149
DnBP	84-74-2	14.5	149
BBzP	85-68-7	18.9	149
DEHP	117-81-7	20.9	149
DiNP	68,515-48-0	21.8	293
BB	120-51-4	6.9	105

**Table 2**

Regression equations for target phthalates.

Phthalate	Regression equations <sup>a</sup>	Correlation coefficients (R <sup>2</sup> )
DEP	$Y = 0.539X + 0.1365$	0.9962
DiBP	$Y = 0.286X + 0.1654$	0.9987
DnBP	$Y = 0.296X - 0.4023$	0.9992
BBzP	$Y = 0.702X + 0.2473$	0.9989
DEHP	$Y = 0.594X - 5.8037$	0.9974
DiNP	$Y = 1.165X + 0.2008$	0.9956

<sup>a</sup> X- ratio of peak area of phthalate to BB in the mixed standard solution; Y- ratio of the concentration of phthalate to BB in the mixed standard solution.

(5 cycles) at 70 °C using dichloromethane. Then the extract was concentrated to 1 mL by a rotary evaporator.

### GC-MS analysis

Dust samples were analyzed by an Agilent 6890 N gas chromatograph and a 5975C mass spectrometry detector. Gas chromatograph was performed using a fuse silica capillary column (HP-5, 30.0 m × 250 μm × 0.25 μm) and a splitless injector with a temperature of 250 °C. Nitrogen with purity ≥ 99.99% was used as carrier gas at a flow rate of 1 mL/min. The initial temperature was 80 °C, which was maintained for 2 min. Then the temperature rose to 220 °C at a rate of 10 °C /min and maintained for 1 min. Finally, it rose to 300 °C at a rate of 20 °C /min and maintained for 5 min. The process was 26 min.

The mass spectrometer used an electron impact ion source (EI) with a collision energy of 70 eV and selected ion monitoring mode (SIM). The temperature of interface and ion source were set at 280 °C and 250 °C respectively.

Retention time and quantitative ion for DEP, DiBP, DnBP, BBzP, DEHP, DiNP and BB are shown in Table 1.

Mixed standard stock solution of DEP, DiBP, DnBP, BBzP, DEHP and DiNP were prepared at a concentration of 100 mg/L. The mixed standard stock solution was diluted with dichloromethane to obtain a series of concentrations with 0.2 mg/L, 0.5 mg/L, 1 mg/L, 2 mg/L, 5 mg/L, 10 mg/L, 20 mg/L, 40 mg/L, 60 mg/L, 80 mg/L and 100 mg/L, 200 mg/L, 300 mg/L and 400 mg/L. The internal standard (BB) was added to a series of mixed standard solution, and the concentration of BB was kept at 1 mg/L.

A series of mixed standard solution were used to establish standard curves, with equations shown in Table 2. The correlation coefficients ranged from 0.9956 to 0.9992.

The ratio of peak area of phthalate to BB in the sample solution, x, was plugged into the regression equation to figure out the ratio of the concentration of phthalate to BB in the sample solution, i.e., y. The concentration of phthalate in the sample solution, c (mg/L), was calculated as the production of y and the concentration of BB in the sample solution (i.e., 1 mg/L). The concentration of phthalate in

**Table 3**  
Field blank values for six target phthalates.

Phthalate	Field blanks		
	Average of field blanks( $\mu\text{g}$ )	Standard deviations( $\mu\text{g}$ )	3 times of SD of field blanks( $\mu\text{g}$ ) <sup>a</sup>
DEP	0.138	0.001	0.004
DiBP	0.553	0.077	0.230
DnBP	0.431	0.061	0.180
BBzP	0.255	0.004	0.010
DEHP	1.298	0.194	0.580
DiNP	0.230	0.007	0.020

<sup>a</sup> Values in the table were rounded.

dust samples was calculated by Eq. (1).

$$w = 0.1 \times \frac{c \times V}{m} \quad (1)$$

Where  $w$  is the concentration of target phthalate in dust sample, with a unit of  $\mu\text{g/g}$ ;  $V$  is the volume of the extraction liquid, i.e., 1 mL;  $m$  is the mass of the extracted dust, i.e., 100 mg.

### Quality control

The above steps were used to analyze 6 blank filter socks without any dust. The average blank values (standard deviations) for six target phthalates are shown in Table 3. The limits of quantification (LOQ) were defined as 3 times of standard deviation of field blanks.

The exposure level of indoor phthalate was obtained through subtracting the concentration of target phthalate in dust sample (i.e.,  $w$  ( $\mu\text{g/g}$ )) by the related average blank value, and was compared with LOQ. If the difference was lower than LOQ, half value of LOQ was nominated as the exposure level and used for further statistical analysis.

### Summary

This paper provides a dust collect method and chemical analysis process for evaluating phthalate concentration indoors. Caution must be taken to avoid background phthalate contamination. Dust was collected using nylon filter socks. Dust was drawn into socks through an aluminum mouthpiece connected to a household vacuum cleaner. Glass vessels for chemical analysis were cleaned by chromic acid lotion and heated in a muffle furnace at 400 °C. Dust samples were extracted with dichloromethane in a Soxhlet extractor. A gas chromatography mass spectrometry (GC-MS) method was used to measure phthalate concentration in dust. The reliability and accuracy of the exposure level of phthalates indoors are guaranteed in our study.

### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### References

- [1] B. Gevao, A.N. Al-Ghadban, M. Bahloul, S. Uddin, J. Zafar, Phthalates in indoor dust in Kuwait: implications for non-dietary human exposure, *Indoor Air* 23 (2012) 126–133, doi:[10.1111/ina.12001](https://doi.org/10.1111/ina.12001).
- [2] U. Heudorf, V. Mersch-Sundermann, J. Angerer, Phthalates: toxicology and exposure, *Int. J. Hyg. Environ. Health*. 210 (2007) 623–634, doi:[10.1016/j.ijheh.2007.07.011](https://doi.org/10.1016/j.ijheh.2007.07.011).
- [3] Q.H. Zhang, Y.X. Sun, Q.N. Zhang, J. Hou, P. Wang, X.R. Kong, J. Sundell, Phthalate exposure in Chinese homes and its association with household consumer products, *Sci. Total Environ.* 719 (2020) 136965, doi:[10.1016/j.scitotenv.2020.136965](https://doi.org/10.1016/j.scitotenv.2020.136965).