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Simultaneous extraction of di(2-ethylhexyl) phthalate and nonionic surfactants from house dust Concentrations in floor dust from 15 Danish schools

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Abstract

Static extraction, supercritical fluid extraction (SFE), pressurized liquid extraction (PLE) and Soxhlet extraction were compared for simultaneous extraction of di(2-ethylhexyl) phthalate (DEHP) and nonionic surfactants from house dust. Homogenized office floor dust from a vacuum cleaner dust bag ("standard dust") was used for the evaluation. One portion of the extracts was used for analysis of nonionic surfactants with LC–MS and another portion was used for DEHP analysis with GC–MS. The extraction yield of DEHP was comparable for all the methods whereas SFE and PLE were the most efficient extraction techniques for the nonionic surfactants. The PLE extraction was found most suitable as a routine method for simultaneous extraction of both types of compounds and was used in a field study of floor dust from 15 Danish schools. The mean concentration of DEHP in the school dust samples was ~4 times higher than observed in other studies of dust from homes in different countries. The concentrations of nonionic surfactants were one order of magnitude lower than soap and linear alkylbenzene sulfonates measured in other studies of floor dust from offices and other public buildings. However, for the first time nonionic surfactants have been identified in house dust. © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

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The phthalate ester di(2-ethylhexyl) phthalate (DEHP) [1] and a limited number of surfactants [2] have been shown to possess adjuvant effects that increase the health damaging potential of common allergens. In addition, phthalate esters are suspected

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to have several other health effects and airborne surfactants may be airway (mucous membrane) irritants in the indoor environment [3]. An epidemiological study has shown that development of bronchial obstruction in children was associated with the presence of poly(vinyl chloride) (PVC) in homes [4]. Based on another study it was proposed that deposition of DEHP in the lungs increases the risk of inducing inflammation that is characteristic of asthma [5]. Phthalate esters are used as plasticizers in PVC and are slowly emitted as vapors. They are common pollutants in indoor air [6,7] and surface house dust [5,8,9]. The existence of phthalate esters in indoor air may be due to resuspension of sedimented dust [5] and/or emission from building products, furniture, etc. [10]. Much less is known about sources and amounts of surfactants in the indoor environment. Only two studies have emerged until now. The first study found up to 0.5% fatty acid salts (soaps) in floor dust from eight offices [11]. The second study found that linear alkylbenzene sulfonates (LASs) are also important components of house dust [12]. In order to estimate the exposure to phthalates esters and surfactants in the indoor environment there is a need for methods to measure these compounds in air and dust. The few studies of phthalate esters in house dust [5,8,9] have used static solvent extraction [5] and static solvent extraction combined with sonication [8,9] and have not paid much attention to the extraction process. In addition, the content of nonionic surfactants in house dust has not been studied before. The aim of the present study was to develop a method for the simultaneous extraction of phthalate esters and nonionic surfactants from floor dust by comparison of different extraction techniques. Homogenized office dust ("standard dust") from a vacuum cleaner dust bag was used for the evaluation. One portion of the extract was used for DEHP analysis with gas chromatography combined with mass spectrometry (GC-MS) and another portion was used for analysis of nonionic surfactants with liquid chromatography combined with mass spectrometry (LC-MS). The most optimal methods with regard to extraction efficiency and analysis time was validated and used in a field study for analysis of floor dust collected in 15 Danish schools.

2. Experimental

2.1. Chemicals and materials

GC-MS standards were DEHP (Pestanal grade, Riedel-de Haën), and hexachlorobenzene (HCB: internal standard, I.S.) (Pestanal grade, Riedel-de Haën). LC-MS standards were Nonidet 40 [commercial nonylphenolpolyethoxylate (NPEO₂)] and alcoholpolyethoxylates (AEO_x) with six ethoxylate groups and alkyl chain length of ten $(C_{10}EO_6)$, twelve $(C_{12}EO_6)$, fourteen $(C_{14}EO_6)$, sixteen $(C_{16}EO_6)$, and eighteen $(C_{18}EO_6)$ carbon atoms (Fluka). The I.S.s were the ethylphenol pentaethylene glycol (EtPEO₆) and hexylphenol pentaethylene glycol (HPEO₆) obtained by synthesis as described elsewhere [13]. Ethylacetate (for chromatography grade, Fluka), heptane ('purum' grade, Fluka or HPLC grade, Rathburn), methanol (analytical reagent grade, Merck), acetone (HPLC grade, Rathburn or analytical reagent grade, Merck), dichloromethane (HPLC grade, Rathburn), and cyclohexane (LiChrosolv, Merck) were used as solvents. LC-MS eluents were water (obtained from a Millipore purification system, Bedford, MA, USA) and methanol (Buffer A) both containing 5 mM ammonium acetate (Merck) and 0.5 mM trichloroacetic acid (Merck). Gasses were helium (He) (5.0, Hydrogas), carbon dioxide (CO₂) (ECD-Qualität 5.2, AGA), and nitrogen (N_2) (5.0, Hydrogas). For supercritical fluid extraction (SFE) and pressurized liquid extraction (PLE) Ottawa Sand (20-30 mesh, Fisher), Hydromatrix (Dionex), or anhydrous sodium sulfate (BDH) was used as fillers and glass fiber filters (GF/B, Whatman) was used in both ends of the extraction cells.

2.2. Extraction study

2.2.1. "Standard dust" preparation

"Standard dust" was produced as follows: house dust was collected in an office building with a standard industrial vacuum cleaner. Fibers were cut by a pair of scissors and the dust homogenized by sieving (500 μ m, 12 DIN). Large objects such as clips were sorted out. However, both a particle fraction and a fiber fraction were obtained. The ratio of the particle to fiber fraction was ca. 4:1 (w/w). The two "standard dust" fractions were stored separately in glass flasks in a refrigerator.

2.2.2. General procedure for all extractions

Prior to the extraction the particle and fiber fractions of the "standard dust" were weighed accurately and mixed in a ~4:1 ratio to give portions of ~0.5 g or ~1 g dust. All flasks etc. were rinsed with methanol prior to use. Then the extraction was performed as described below separately for each type of extraction. After the extraction the solvent was changed by gentle evaporation at 40 °C to almost dryness with charcoal filtered N2, addition of 100 µl ethylacetate, evaporation to almost dryness, and finally addition of 10 ml heptane. The extraction cycle was repeated up to three times to test for exhaustive extraction (except for Soxhlet extraction that has many cycles). For each experiment 4-5portions of "standard dust" and one blank was extracted. The extract was divided into two portions for analysis of DEHP and nonionic surfactants, respectively.

2.2.3. Static extraction in a flask

This extraction was similar to the method used by Øie et al. [5] for extraction of phthalates from house dust. The "standard dust" was weighed into a 50-ml glass stopped Erlenmeyer flask and 20 ml of methanol was added. The dust was mixed thoroughly with the methanol by shaking and left for extraction without shaking at ambient temperature for 48 h. It was then shaken again, transferred to a vial and centrifuged (1000 rpm for 5 min). The solvent of the clear supernatant was then changed as described above. The extract was finally treated as described in the general procedure.

2.2.4. Supercritical fluid extraction

Two different types of SFE equipment were used. The extraction parameters used were within the traditional ranges for SFE of polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) in environmental samples [14]. For collection of the analytes in a liquid an ISCO model 210D pump and an SFX-210 extractor was used. The "standard dust" was weighed, mixed with sodium

sulfate and placed in the 10-ml extraction cell. The flow-rate of the CO_2 was adjusted to 2 ml/min with an ISCO coaxially heated restrictor set to 80 °C. For different experiments the extractor temperature was set to 80 (SFE 1), 100, 120 and 150 °C, respectively. The pressure was 365 bar for all experiments. A static extraction of 5 or 10 min was performed prior to a 30- or 40-min dynamic extraction. Extracted analytes were collected by placing the outlet of the restrictor into a 25-ml vial containing 15 ml of acetone. For collection of the analytes on a solidphase trap a Hewlett-Packard HP 7680T supercritical fluid extraction unit was used (SFE 2). The "standard dust" was weighed, mixed with sodium sulfate and placed in the 7-ml extraction cell. The flow-rate of the CO₂ was set to 1 ml/min and a density of 0.80 g/ml (365 bar). The extraction temperature was set to 100 °C, and the temperatures of the nozzle and trap were 45 and 20 °C, respectively. A static extraction of 5 min was performed prior to a 40-min dynamic extraction. Extracted analytes were collected on a solid-phase trap containing Florisil (supplied by Hewlett-Packard). Elution of the trapped analytes was done with 1.4 ml dichloromethane at 1 ml/min, followed by 6 ml of acetone and another 6-ml portion of dichloromethane (for reconditioning of the trap). The extract was finally treated as described in the general procedure.

2.2.5. Pressurized liquid extraction

Extractions were performed using a Dionex ASE 200 system [15]. The "standard dust" was weighed into the cell and the dead volume was filled with (precleaned) Ottawa sand placed in the stainless steel extraction cell (11 ml). To prevent clogging of the metal frit, a filter paper (diameter 19.1 mm) supplied by Dionex was placed at the exit of the cell. The extraction was started by pumping the solvent into the cell. The cell was then preheated for 5 min to reach the set temperature (100 °C), followed by a static extraction of 5 min at constant temperature and pressure (140 bar). After the static extraction the pressure was released and the extract was collected in a 25-ml glass vial. The rinsing volume was 60% of the extraction cell volume as set by the software. Finally, pure N₂ was purged through the extraction cell for 1 min to assure that the solvent (and

analytes) was completely transferred to the collection vial. The extraction cycle was repeated once to evaluate the exhaustiveness of the extraction cycle. Two experiments were performed with different solvents, cyclohexane–acetone (1:1) (PLE 1) and methanol (PLE 2). The extract was finally treated as described in the general procedure.

2.2.6. Soxhlet extraction

The extraction was performed as standard Soxhlet extraction. The "standard dust" was weighed into the extraction thimble, ~ 25 ml of methanol were placed in a round-bottomed flask and the extraction was carried out at 60 °C for 12 h. The extract was finally treated as described in the general procedure.

2.3. Analyses

2.3.1. DEHP

Two different GC-MS systems were used for analysis of DEHP. In the extraction study split injection GC-MS was used for both identification and quantification. In the field study thermal desorption (TD) and GC-MS was used for identification and control for interference and TD-GC with flame ionization detection (FID) was used for quantification. Before the GC-MS analysis of the phthalate esters, ~ 1 ml of the extract was accurately weighed into a 1.5-ml vial and spiked with 100 µl of a solution containing ~500 ng/µl hexachlorobenzene in heptane as the I.S. A Hewlett-Packard model 5972 GC-MS with a constant He (carrier gas) pressure of 103 kPa (1 p.s.i.=6894.76 Pa) was equipped with $30 \text{ m} \times 0.25 \text{ mm}$ I.D. Chrompack CP Sil 8 CB Low Bleed/MS (0.5 µm film thickness) column. A 2-ml volume of the extract was injected in the split mode (split 1:7.5) at an injector temperature of 260 °C. Temperature programming was 180 °C, held 1 min, increased to 320 °C at 30 °C/min, and held for 5 min. The MS transfer line temperature was 275 °C. The MS was operated in the electron impact ionization mode (70 eV) using selected ion monitoring (SIM) and scan mode (m/z 30-400). For the quantification of DEHP m/z 149 was used. Standards for six-points calibration curves were run in each series of samples and each 10th sample was a control standard. Before the TD-GC-MS/FID analysis the solvent was changed to methanol as described in

Section 2.2.2. The solution was directly injected (5-30 µl) onto Tenax TA in stainless steel tubes and analyzed by TD-GC-MS for identification and by TD-GC-FID for quantification as described elsewhere [16]. Both systems were Perkin-Elmer GC Autosystem XL/TurboMass MS or FID, respectively. The systems were running with a constant He (carrier gas) pressure of ~20 p.s.i. resulting in a flow-rate of $\sim 1 \text{ ml/min}$ at 120 °C (calculated). They were equipped with 60 m×0.25 mm I.D. Chrompack CP Sil 8 CB low bleed/MS (0.25 µm film thickness) columns. The GC temperature programming was 120 °C, held 2 min, increased to 300 °C at 15 °C/ min, and held for 8 min. The MS parameters and the external calibration (no I.S. was used) were as for the split injection GC-MS method. The FID temperature was 275 °C. The limit of detection (LOD) was estimated as three times the standard deviation of DEHP in methanol (5 ng/ μ l) injected (5 μ l) onto Tenax TA tubes (n = 13).

2.3.2. Nonionic surfactants

Before the analysis the solvent was evaporated and the extract redissolved in same volume of Buffer A containing ~1 μ g/ml of the I.S.s (EtPEO₆ and HPEO₆). The chromatographic system consisted of a HP 1100 HPLC system (Hewlett-Packard, Palo Alto, CA, USA) with a C₁₈ analytical column. This was connected to an Esquire-LC (ion trap) mass spectrometer (Bruker Daltonics, Bremen, Germany) with an electrospray interface operated in the positive ionization mode. The compounds were detected as ammonium adduct ions. The details of the method are described elsewhere [13]. The nonylphenol and alcohol polyethoxylates with 6–15 ethoxlate groups were quantified. Results below the lowest calibration standards were not reported (~LOD).

2.4. Field study

2.4.1. Floor dust sampling

Floor dust sampling was done with a specially designed vacuum cleaner HVS3 (Cascade Stack Sampling Systems, OR, USA) [17]. HSV3 was modified to ensure a more constant suction pressure and volume as described previously [18]. Recently, the design and use of the HSV3 has been standardized [19]. In 15 Danish schools dust was col-

lected from 3 to 10 m² before the daily floor cleaning in each of 2–5 similar classrooms with identical floor covering. Shortly after collection the samples were divided as previously described [18] and stored in small glass vials at -18 °C. Approximately 10% of each sample was used for analysis of DEHP and nonionic surfactants. Before the extraction the dust samples were pooled to one sample for each school.

2.4.2. Extraction and analysis

The analytical method for DEHP was modified compared to the extraction study as described in Section 2.3.1. PLE 1 was used for the extractions of the school dust samples. The extracts were divided into two portions for analysis of phthalate esters and nonionic surfactants, respectively.

2.4.3. Validation of the field study methods

For estimation of the recovery five 1-g portions of "standard dust" were spiked with DEHP (4467 μ g/g dust), extracted with PLE 1 and analyzed with TD–GC–FID. Analysis of PLE 1 extracts of the "standard dust" was used to compare GC–MS and TD–GC–FID.

For estimation of the recovery three 1-g portions of "standard dust" were spiked with $C_{18}EO_6$ (~15 µg/g dust) extracted with PLE 1 and analyzed. Analysis of four PLE 1 extracts of "standard dust" was used for an independent validation by comparison of LC–MS and LC–MS–MS [20].

3. Results and discussion

3.1. Extraction study

The "standard dust" was produced in order to compare different extraction methods. It was produced from office floor dust that has a natural content of a wide spectrum of different compounds (see Figs. 1 and 2, and cf. [8,9,11,12,18,21–24]).

The preconcentration (evaporation) step of the solvent change removed a large fraction of the dibutyl phthalate (DBP) in the extracts whereas DEHP was quantitatively recovered [16]. Therefore only DEHP is reported despite the fact that the

"standard dust" also contained DBP and butyl benzyl phthalate.

Fig. 3 shows that the yield of DEHP was generally comparable for all the methods but with the SFE and PLE methods as the most efficient. The blank values were <1% of the DEHP content of the "standard dust". No significant difference in the yield of DEHP extracted with SFE at different temperatures was observed.

The nonionic surfactants were not measured in the PLE 2 and the Soxhlet extracts. In the SFE 2 extracts all measured concentrations were below the lowest calibration standards. This was probably due to the solid-phase trap that retained the nonionic surfactants but not the phthalate esters. In all chromatograms of blanks no peaks of AEO, or NPEO, were found. Fig. 3 shows that the yield of $C_{10}EO_r$ was different for the static, the SFE 1, and the PLE 1 extractions. The other homologues of the AEO_x and NPEO_y were extracted equally well with SFE 1 and PLE 1 but much less efficient with the static extraction. The higher yield of $C_{10}EO_x$ relative to the other homologues with the SFE 1 method might be due to a relatively higher polarity of $C_{10}EO_x$ combined with a good extraction efficiency for polar compounds with SFE (CO₂) at 80 °C and 365 bar (0.8 g/ml). This is supported by the tendency to a decreasing yield of $C_{10}EO_r$ with increasing SFE temperature (at constant pressure) while the other homologues and NPEO_x had a constant yield (see Fig. 4). This indicates that the extraction efficiency of nonionic surfactants depends more on the SFE solvent power and thereby on the fluid and less on the extraction temperature as for the extraction of fat and oil [14,25,26].

3.2. Method validation

The PLE extraction was found most suitable as a routine method for simultaneous extraction of both types of compounds because the time consumption was low, the PLE equipment was easy to use, and the yield was high and comparable to SFE. The PLE 1 extraction (cyclohexanol-acetone) including solvent change was chosen for the field study.

For DEHP two extraction cycles were sufficient to obtain exhaustive extraction with PLE 1 where the last cycle increased the yield with 3%. Recovery of DEHP spiked on "standard dust" was $111\pm15\%$







Fig. 2. (A) Extracted and stacked LC–MS chromatograms of a PLE 1 extract of a blank ($C_{10}EO_x$ trace) and of AEO_x in the "standard dust. (B) LC–MS spectrum of $C_{10}EO_x$ in the PLE 1 "standard dust" extract.

(95% confidence). The content of DEHP in the "standard dust" based on four PLE 1 extracts and the GC-MS method (extraction study) was $1250\pm120 \ \mu g/g$ dust (95% confidence). The content of DEHP in the "standard dust" based on the field study methods was $1190\pm100 \ \mu g/g$ dust (n=10).

Also for AEO_x two extraction cycles were used to obtain exhaustive extraction with PLE 1 where the last cycle increased the yield with a maximum of 3%. Recovery of $C_{18}EO_6$ spiked on the dust prior to the extraction was $127\pm27\%$ (95% confidence). The results for the nonionic surfactants in the "standard



Di(2-ethylhexyl)phthalate (DEHP)







Fig. 3. Absolute yield of DEHP and nonionic surfactants from extraction of "standard dust" with different methods. The error bars are the 95% confidence intervals for 4–5 extractions. See Experimental for details.

Non-ionic surfactants (AEO_{x-6-15} and NPEO_{x-6-15})



C10EOX C12EOX C14EOX C16EOX NPEOX

Fig. 4. Absolute yield of nonionic surfactants from SFE 1 extraction of "standard dust" with increasing temperature and constant pressure. The error bars are the 95% confidence intervals for five extractions.



Non-ionic surfactants (AEO_{x=6-15} and NPEO_{x=6-15})

Fig. 5. PLE 1 extracts of "standard dust" analyzed for nonionic surfactants with two different LC-MS methods. The error bars are the 95% confidence intervals of four extractions.

dust" were independently validated by use of LC– MS–MS [20] as shown in Fig. 5. The good agreement between the two different LC–MS methods (ion trap mass spectrometry and tandem mass spectrometry) is crucial for the validation of the analysis of the nonionic surfactants.

For quantitative analysis of DEHP, FID was preferred as the detection method because of its high stability and large linear dynamic range. However, the extracts had to be analyzed by TD–GC–MS to ensure absence of interfering compounds in the DEHP peak. The LOD for DEHP was estimated to 11 ng as the absolute amount on the Tenax TA tube. DEHP in the blanks were below this LOD. Taking into account the variable amounts of dust in the school samples this corresponds to a maximum LOD for DEHP of 87 μ g/g dust. This is below any of the measured values in the field study (see Fig. 6).

The lowest concentration LC–MS calibration standards were 0.05 ng/µl for NPEO₆ and 0.1 ng/µl AEO₆. Results below these values were not reported. All blanks were below these values. Taking into account the variable amounts of dust in the school samples this corresponds to a maximum report limit for NPEO_{x=6-15} of 2.3 µg/g dust NPEO_{x=6-15} of 4.6 µg/g dust.

We are aware of only three studies of phthalates in house dust. They all used GC–MS and direct injection of the extract. The extraction methods varied from static extraction with methanol [5] to ultrasonic extraction with acetone–cyclohexane [8] and toluene [9]. Only the last mentioned study had a limited description of quantitative validation of the method. They found recoveries of 80–115% of a series of compounds including DEHP. The LOD (not defined) of DEHP was stated to be ~1 μ g/g dust that is far below the maximum value found in this study. Their instrumental LOD is thus ~1/10th that of the TD–GC–FID method used in this study. This may be due to larger variation of TD and nonreproducible interference combined with the nonspecific FID.

Based on the good agreement between the different analytical methods and the recoveries we consider the field study methods as sufficiently precise and sensitive for determination of DEHP and nonionic surfactants in house floor dust.

3.3. Field study

The mass of the dust in the pooled samples used for the analysis of nonionic surfactants and DEHP were between 0.4 and 1.6 g. The floor dust concentrations in each classroom were between 0.16 and 3.5 g/m^2 (mean = 0.52 g/m²). This is approximately twice the values for samples collected with the same equipment in 12 offices (mean = 0.24 g/m², min = 0.04 g/m², max = 0.89 g/m²) [18].

Fig. 6 shows the concentrations and analytical variations of the studied compounds in floor dust from these particular sampling locations and particular sampling days (cross-sectional study). However, they may illustrate the concentration levels and variability representative for Danish schools. It is not



Di(2-ethylhexyl)phthalate (DEHP)

Non-ionic surfactants (AEO_{x=6-15} and NPEO_{x=6-15})



Fig. 6. DEHP and nonionic surfactants found in single samples of floor dust from 15 Danish schools (A–O). The error bars of DEHP are the 95% confidence intervals for 3–6 analyses. The relative error of the NPEO_x and AEO_x results are 16% and 6–19%, respectively. This is based on 95% confidence intervals of the analysis of four extracts of the "standard dust" (see text and Fig. 5).

the aim of this paper to evaluate the health impact of the measured concentrations.

DEHP concentrations were very high compared to other studies (see Table 1). The levels of DEHP found in the other studies (all from homes) appear to be comparable. Other differences are age of the sampled dust, sampling technique, treatment of the dust (dust fraction), extraction and analytical methods. For the first time nonionic surfactants have been measured in floor dust. Therefore the results cannot be compared to other studies. However, the observed concentrations of these surfactants in the floor dust were much lower than the soaps (total fatty acid salts) found in floor dust from eight offices (up to 5000 μ g/g dust) [11] and LASs (total linear alkylbenzene sulfonates) in floor dust from seven public buildings (up to 1500 μ g/g dust) [12].

Study	Sampling/ treatment	Mean	90% percentile	95% percentile	Building type	No. of samples
Denmark (this study)	HVS 3	3214	6404	7063	Schools	15
Germany 2001 [9]	Vacuum cleaner, sieving $< 63 \ \mu m$			2600	Homes	286
Denmark 2001 [27]	Filter using vacuum cleaner	858	1761	2595	Homes	23
Germany 1997 [8]	Vacuum cleaner, particle fraction		1600	2000	Homes	272
Norway 1997 [5]	Filter using vacuum cleaner	640			Homes	38

Table 1							
DEHP concentrations (µg/g dust)	measured in s	surface dust	from diff	ferent bui	ildings in	different	countries

4. Conclusions

DEHP is easily extracted from house dust using various techniques. Nonionic surfactants are most efficiently extracted from house dust with SFE and PLE. Taking time consumption and easiness into account PLE extraction was found the most suitable technique for simultaneous extraction of DEHP and nonionic surfactants from house dust.

The results of the field study of floor dust from schools showed that DEHP could be up to nearly 1% (w/w). This is four times higher than the mean concentrations found in other studies of house dust from homes. For the first time nonionic surfactants have been identified in house dust.

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