Identification and semi-quantification of known and novel contaminants in indoor dust by ion-mobility high-resolution mass spectrometry and estimation of risks for human exposure

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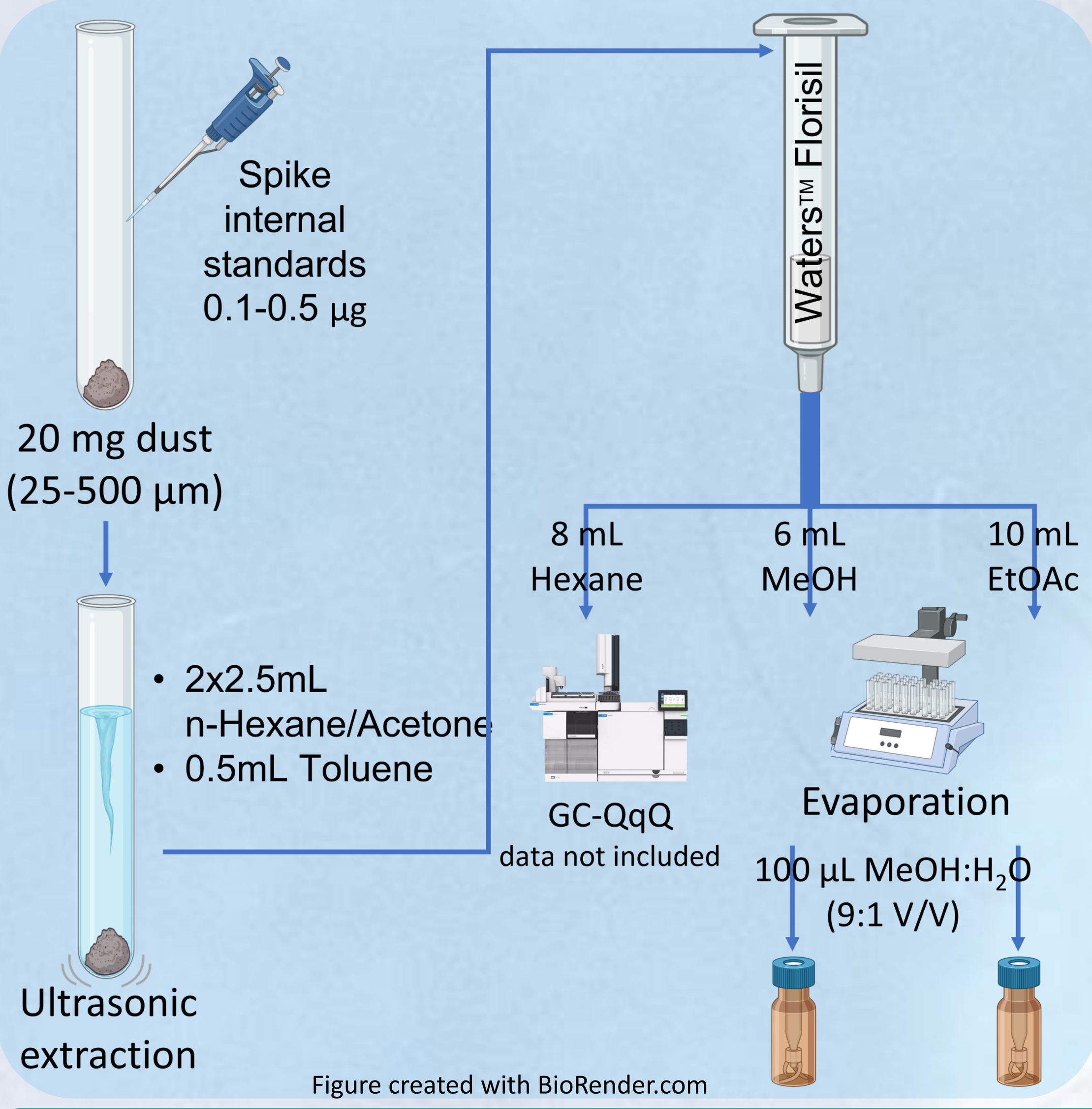
INTRODUCTION

- Dust is important for exposure since humans spend 80% of their time indoors
- Toddlers are a highly exposed risk group due to hand-to-mouth contact
- Dust functions as a sink of indoor contaminants which has been underlined by the results of quantification studies on several classes of contaminants
- Due to the ever-evolving chemical exposure there is a complementary need for the identification of **contaminants of emerging concern (CECs)**

- Screening of **46 Flemish dust samples** from houses, offices, and leisure venues
- **Drift tube ion-mobility (IM) mass spectrometry (MS)** was applied as an additional separation dimension to liquid chromatography (LC) and high-resolution mass spectrometry (HRMS)
- IM-MS allows for the calculation of **collision cross-section values (CCS)**, to further increase identification confidence.

MATERIALS AND METHODS

Sample Preparation[(1)]



Instrumentation

Agilent 6560 HPLC-IM-QTOF

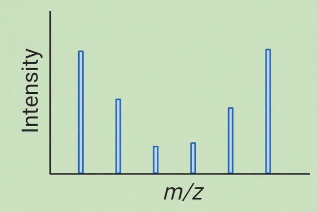
- **Column**: InfinityLab Poroshell 120 EC-C18
- **ESI+**: (A) H_2O + 0.1% FA
  (B) MeOH + 0.1% FA
- **Mass range:** m/z 100-1500
- **Acquisition:** QTOF only – data dependent acquisition and IM-MS 4bit multiplexing
- **Software:** Mass Profiler Professional, Agilent Masshunter, IM Browser

Data Processing

QC and IS RT stability – mass accuracy – stable signal intensities

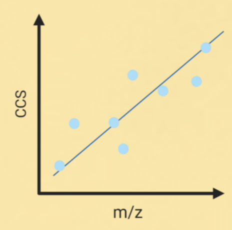
Target/ Suspect screening

- Target screening with in-house standards
- Suspect list with known and novel CECs (n > 4000)
- Match based on retention time (< 0.2 min.), m/z (< 7ppm), isotopic (score > 80) pattern and fragmentation pattern (manual matching)



IM-MS analysis

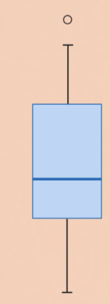
- CCS values for all compounds
- $[M+H]^+$/$[M+Na]^+$ adducts
- Match with established database (ΔCCS < 2%) and m/z[2]
- Comparison with m/z-CSS trendlines



Semi-Quantification

- Calibration curves of selection of ref. standards
- Structural similarity between calibrant & suspect[3]
- Consideration of one adduct

$$Conc._{Susp.} = \frac{Area_{Susp.}/Area_{IS}}{Response_{factor}}$$



RESULTS

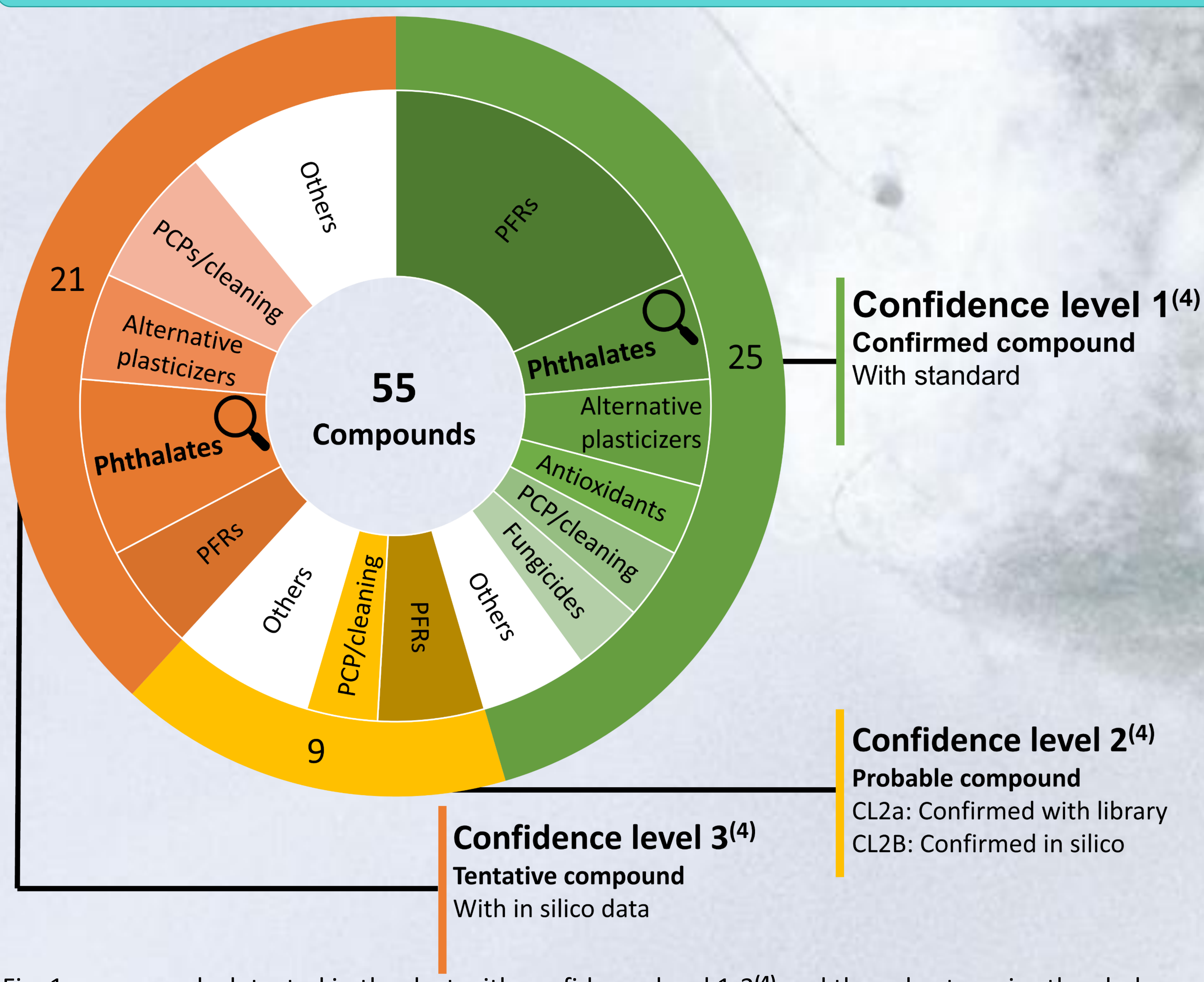


Fig. 1: compounds detected in the dust with confidence level 1-3[(4)] and the subcategories they belong to. With PFR: phosphate flame retardant; PCP: personal care products.

Phthalate identification

Tab. 1: Summary of detected phthalates. With DF; detection frequency, EDI; estimated daily intake of toddlers for the 95th percentile. HQ; hazard quotient based on U.S. EPA. (2017). Exposure Factors Handbook Chapter 5 (Update).

Compound	DF [%]	CL	Estimated Conc. [µg/g]	EDI [mg/kg (bw)/day]	HQ
Diisodecyl phthalate (DIDP)	97.8	CL 1	3.2-90	5.71E-05	-
Diisononyl phthalate (DINP)	100	CL 1	12.7-437	5.71E-05	2.49E-03
Diethylhexyl phthalate (DEHP)	91.3	CL 1	8.0-448	1.62E-04	8.10E-03
Decyl nonyl phthalate	93.5	CL 3	2.5-645	9.77E-05	4.89E-03
Decyl undecyl phthalate	82.6	CL 3	1.7-478	3.65E-05	1.82E-03
Undecyl dodecyl phthalate	4.3	CL 3	6.4-31	-	-
Diundecyl phthalate	45.7	CL 3	0.5-122	-	-
Diheptyl phthalate (DHP)	89.2	CL 3	0.7-50	1.34E-05	6.7E-04

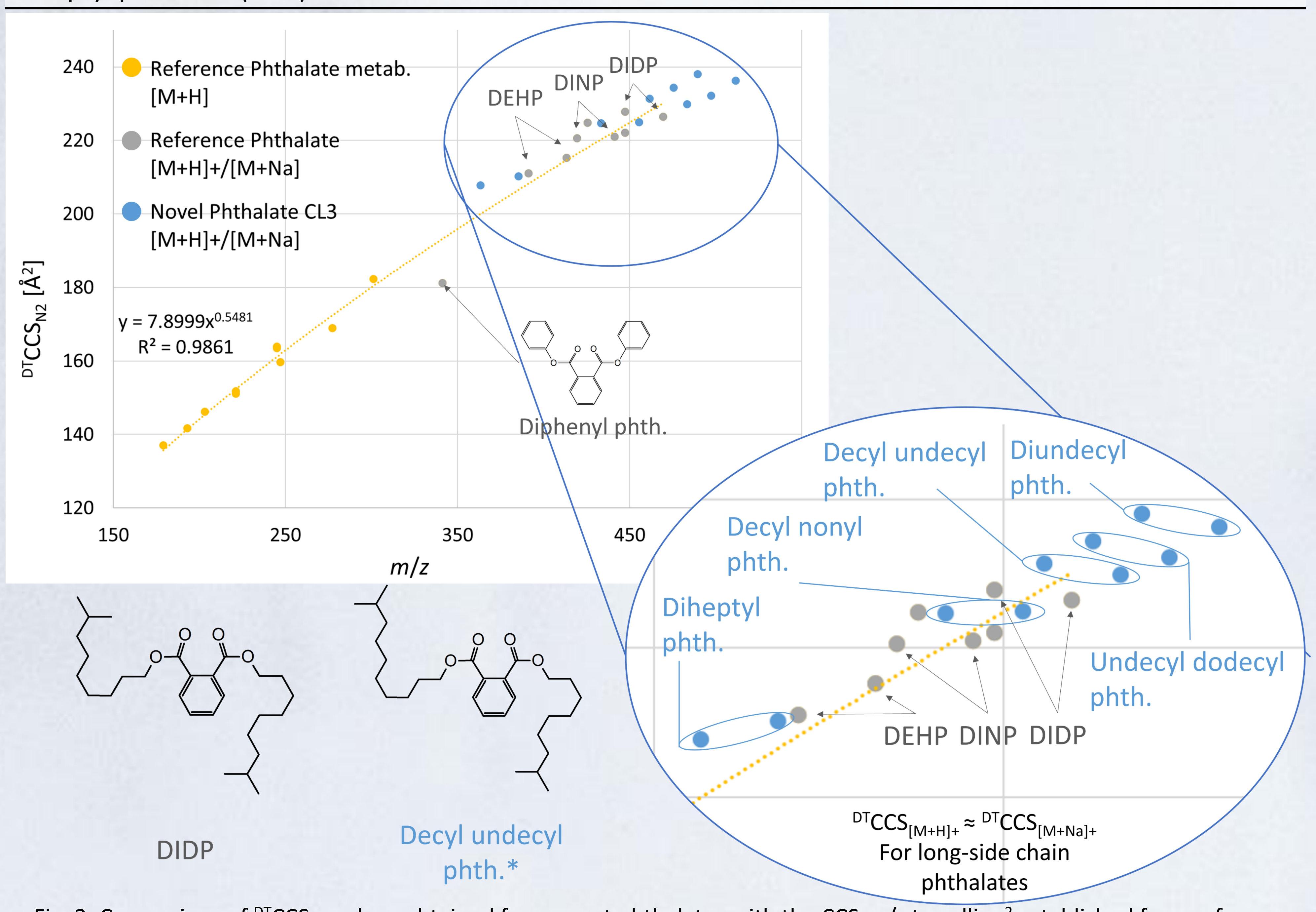


Fig. 2: Comparison of $^{DT}CCS_{N2}$ values obtained for suspect phthalates with the CCS-m/z trendline[2] established from ref. $^{DT}CCS_{N2}$ values of known phthalates and their metabolites (Metab.). *CL3 with uncertain alkyl chain branching

DISCUSSION AND CONCLUSIONS

- 55 compounds from various classes were detected in dust
- Detected **phthalates with uneven side chains** show unstudied variability for the class of phthalates
- Concentration of novel phthalates is **in the same order** as the concentration of well-known reference phthalates
- $^{DT}CSS_{N2}$ can function as an additional identification parameter by **matching with established CCS-m/z trendlines**
- Semi-quantification allows exposure assessment by giving an indication of concentration
- Exposure to phthalates through dust ingestion did result in **no potential risk** for toddlers, the most exposed group

[1]Christia et al. 2021; Chemosphere 263: 127817.
[2]Belova et al. 2021; *Anal. Chem.* 93(16): 6428-6436.
[3]Malm et al. 2021; *Molecules.* 26(12): 3524
[4]Schymanski et al. 2014; *Environ. Sci. Technol.* 48(4): 2097-2098.

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