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Title

Determination of chlordecone in indoor dust by GC/MS/MS and associated human exposure in the French West Indies

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Highlights

- First analysis of chlordecone (CLD) in indoor dust using a novel GC/MS/MS method
- Limit of detection of 8 ng/g and limit of quantification of 16 ng/g
- Quantified CLD concentrations in dust samples ranged from 51 to 247 ng/g
- Higher concentrations are found in rural areas than in urban areas
- Indoor dust ingestion appears to be a negligible pathway for CLD exposure (< 1,5%)

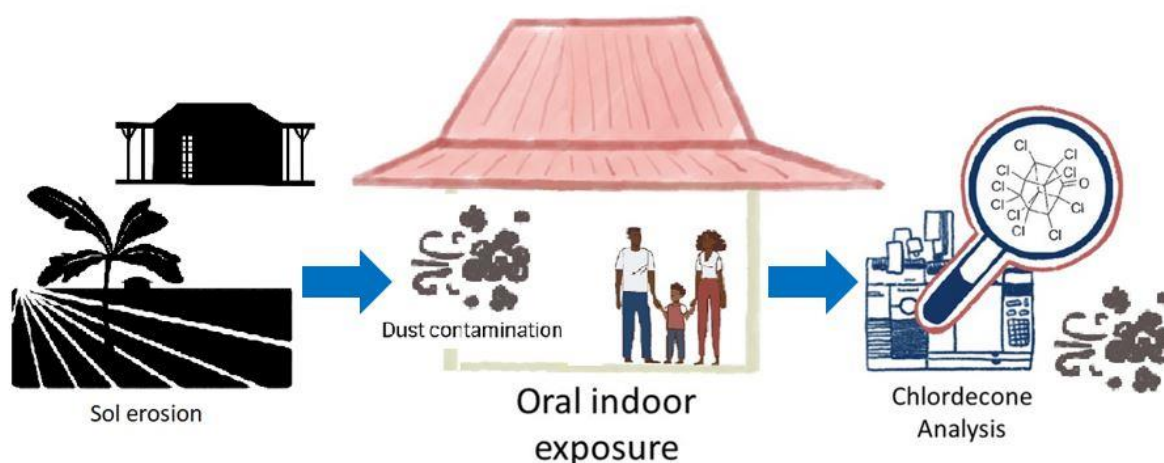
Abstract

Chlordecone (CLD) is an organochlorine pesticide with various toxicities, widely used from 1972 to 1993 in the French West Indies lands (FWI, Guadeloupe and Martinique). It can remain in environment leading to indoor contamination by pollutants coming from outside. Therefore, investigating indoor pollutants is necessary due to the potential indoor dust ingestion through hand-to-mouth contact, in particular for children. No chemical analytical methods measure CLD in indoor dust and we propose to investigate its presence in this specific compartment, in Martinique. CLD was analyzed from dust samples taken from homes and schools (n=47). We extracted CLD from dust with pressure liquid extraction (PLE) using a suitable mixture solvent of hexane and ethyl acetate (85:15). This was followed with gas chromatography coupled to tandem spectrometry (GC/MS/MS) analysis. A sampled mass of 25 mg optimal for analysis. Concentrations of CLD in indoor dust varied from < 16 to 247 ng/g. Exposure to CLD through indoor dust ingestion was also assessed and does not exceed 1.5% and 0.1%, of the acceptable daily intake, in childrens and adults, respectively. However, this exploratory study was conducted on a very limited number of dust samples limits the generalization to the whole French West Indies population.

Environmental implication

Chlordecone is a pesticide widely used on banana plants in the French West Indies. Although its use has been banned since 1993, the molecule is still present in soils and contaminates the trophic chains up to humans. Contamination levels in food, soil, and water, have already been described, but not indoor dust. As humans spend most of their time indoors, dust can be a vector that contributes to human exposure. This study, through the development of a novel analysis method, completes existing data by assessing chlordecone contamination of indoor dust.

Graphical Abstract



Keywords

- public health, indoor contamination, exposome, pesticide, chlordecone, household dust

Abbreviations

- ASE: accelerated solvent extraction
- CLD: chlordecone
- ISTD: internal standard
- LOD: limit of detection
- LOQ: limit of quantification
- TCNB: tetrachloronitrobenzene

- GC/MS/MS: gaz chromatography coupled with tandem mass spectrometer

CRedit authorship contribution statement

- Marie Dufresne: Data curation, Visualization, Writing - Original draft
- Gaëlle Raffy: Methodology, Investigation, Validation, Writing – Review & Editing
- Jean Guimberteau: Methodology, Ressources, Data Curation, Writing - Review & Editing
- Nathalie Bonvallet: Methodology, Writing - Review & Editing
- Aurélien Lecorgne: Formal Analysis, Data Curation, Writing - Review & Editing
- Emilie Surget: Methodology, Resources, Writing - Review & Editing
- Carole Boullanger: Methodology, Writing - Review & Editing, Supervision
- Luc Multigner: Methodology, Writing – Review & Editing
- Olivier Ramalho: Methodology, Writing- Review & Editing
- Philippe Glorennec: Methodology, Writing- Review & Editing
- Barbara Le Bot: Conceptualization, Funding acquisition, Project administration, Writing- Review & Editing; Validation.

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1 Introduction

Chlordecone (CLD) also known as Curlone® or Kepone®, is an organochlorine pesticide widely used to control banana weevils (*Cosmopolites sordidus*) from 1972 to 1993 in the French West Indies (FWI) [1]. CLD is among the persistent organic pollutants (POPs) listed in the Stockholm Convention [2].

Because of its chemical properties, i.e. low volatility (vapor pressure = $3.1 \cdot 10^{-5}$ Pa), apolar ($4.51 < \log Kow < 5.41$), and high affinity to organic matter ($Koc = 15850$), and its low biotic and abiotic degradation, CLD is still present in soils (the most contaminated exceed the concentration of 1mg/g [3]) where it was applied and contaminates local water resources, foodstuffs with highest concentrations at 320.71 $\mu\text{g}/\text{kg}$ fw observed in sweet potatoes. It is also found in human biomonitoring studies with a highest CLD serum concentration at 15.41 $\mu\text{g}/\text{L}$ [4], [5], [6], [7], [8]. Experimental studies *in vitro* and *in vivo* have shown that CLD is neurotoxic, reprotoxic, and carcinogenic [6], with well-recognized estrogenic properties [9]. In epidemiological studies, CLD exposure is associated with an increased risk of prostate cancer, preterm birth, and impaired child neurodevelopment [10], [11], [12], [13], [14].

Exposure to pesticides and organic pollutants in indoor environments is a growing concern. Residents are exposed to pollutants in indoor environments through various pathways, including inhalation, dermal contact or ingestion of contaminated environmental matrix (through air or sprayed products, deposited particles, contaminated food, etc). Recently, non-dietary ingestion of environmental chemicals from indoor dust has become an issue of concern and must be considered in health risk assessments [15], [16]. The composition, amount, and origin of indoor dust can vary significantly depending on specific inhabitant activities [17]. Indoor dust also comes from outdoor dust and external soil particles, and

outdoor pollutants can also reach housing [18], [19], [20]. Children are particularly vulnerable due to their proximity to the ground and exploratory behavior, leading them to ingest particles by hand-to-mouth contact [21]. Studies have suggested that dust particles with a diameter $< 100 \mu\text{m}$ are more representative of exposure through hand-to-mouth contact in children [22], [23], [24]. Indoor dust, both suspended in the air and deposited on surfaces, can be inhaled, swallowed, and ingested. For this study, only the oral route was investigated. CLD was not found in ambient air during the pesticide national exploratory French campaign, which included the FWI [25]. This may be explained by the low vapor pressure of CLD.

Currently, there is no chemical analytical methods for measuring CLD in settled indoor dust. Analytical method exploring CLD concentrations in soil matrices use pressurized liquid extraction with dichloromethane and acetone, or solid phase micro extraction and GC/MS analysis [26], [27].

Here, we aimed to (i) develop and validate a method for analyzing CLD in dust, (ii) measure the concentration of CLD in indoor settled dust, and (iii) provide a preliminary assessment of human exposure via indoor dust ingestion.

2 Materials and Methods

2.1 Solvents and Reagents

A powder of standard CLD (CAS n°143-50-0, $\text{C}_{10}\text{Cl}_{10}\text{O}$, 491 g/mol) (purity 99.7%) manufactured and distributed by Sigma-Aldrich (Saint Louis, USA) was used for preparing the calibration standards and sample spiking. A commercial solution of ^{13}C -CLD certified at 100 $\mu\text{g/mL}$ in toluene (99%) manufactured by Cambridge Isotope Laboratories (CIL) (Tewksbury, USA) and distributed by LGC Standards (Augsburg, Germany) was used as the internal standard. A powder of standard CLD (purity 87.87%) manufactured by Dr. Ehrenstorfer (Wesel, Nordrhein-Westfalen, Germany) and distributed by LGC Standards (Augsburg, Germany) was used as an external control to validate the calibration range. 2,3,4,5-tetranitrochlorobenzene (TCNB) manufactured and distributed by Dr. Ehrenstorfer (Wesel, Nordrhein-Westfalen, Germany) was used as an injection tracer to monitor the GC injection process.

Acetone, dichloromethane, and hexane solvents purchased by Carlo Erba (Val-de-Reuil, France) were all pestipur grade or equivalent. Ethyl acetate was purchased from Fischer Scientific (Illkirch, France). Celite® 545 (particle size 0.02-0.1 mm) was purchased from LGC Standards (Augsburg, Germany) and heated to 550°C for 2 h before each experiment.

2.2 Dust samples

Indoor dust samples from six dwellings and two schools in Martinique, one of the two main islands of the FWI in the Caribbean, were collected over one year from November 2022 to November 2023. Dust collection occurred monthly in both schools (n=24) and quarterly in all six dwellings, except one, where only three dust samples were collected (n=23). In schools, samples were only collected in classrooms. In dwellings, samples gather dust from all the inside rooms. Half of the sampling sites were located in an urban area (municipality of Lamentin) and the other half in a rural area next to agricultural plots (municipality of Macouba). Urban sites were selected according to a population density > 2000 inhabitants/km². Dust samples were collected using a vacuum cleaner in dedicated vacuum bag, according to a defined protocol (detailed in Supplementary Information). Then bags were stored in specific aluminized polyethylene bags for transport. As schools were closed during school and public holidays, the schedule had to be adjusted to collect samples only when the schools were open. When the schools were closed in August, only two residences were sampled, and school sampling was postponed until November to ensure temporal consistency of the data. This planning ensured the effective capture of monthly and seasonal variations in both studied areas. Collected samples were transferred in dry ice box under controlled temperature, and within 15 days to the Leres Laboratory in Rennes.

Composite dust (named Dust A, B, C), prepared from mixed indoor dust from home interiors, coming from mainland French dwellings collected since 2014 and available in the laboratory collection, were used for analytical development. This composite dust has varying organic matter content ranging from 20 to 60%. Settled dust from anonymous vacuum bag donations collected from various Martinican dwellings was also used for analytical development.

Before analysis, all dust samples were sieved through a stainless steel grid precleaned with dichloromethane using a vibratory sieve shaker (Retsch, Haan, Germany) to obtain particles < 100 μm . All samples were stored for analysis at -20°C in 20 mL amber glass vials with inox caps.

Standard reference material (SRM) 2585[®] (organic contaminants in house dust) prepared by the National Institute of Standards & Technology (NIST) was also used for analytical development [28]. This dust was collected from indoor environments (homes, hotels, motels, and cleaning services) from different states in the USA from 1993 to 1994. The dust was gamma-irradiated and then sieved to a size range of 90-100 μm before storage at room temperature ($18-25^{\circ}\text{C}$). However, there is no information or certified data concerning CLD quantities in the SRM 2585[®] samples.

2.3 Dust sample preparation

For each sample, 25 mg of dust was weighed on a calibrated microbalance (Sartorius, Göttingen, Germany) and then mixed with conditioned Celite[®] 545 to disperse the dust during extraction. The mix was tamped into 1 mL extraction cells and spiked with a volume of 4 μL of an internal standard solution (ISTD) of ^{13}C -CLD at 1 mg/L. Two successive pressure liquid extractions (PLE) were performed using a Dionex[™] ASE[™] 350 (Thermo Scientific) instrument in a solvent mixture (hexane and ethyl acetate 85:15). The heating program was set to 100°C with a pressure of 1600 PSI. The extracted samples were then concentrated to a drop using a nitrogen flow evaporator (Organomation, Berlin, Germany) and finally diluted in 1 ml hexane, spiked with TCNB, and transferred to glass amber vials. Extracts were vortexed prior to GC/MS/MS injection. After analysis, samples were stored at -20°C . All glassware and syringes were rinsed twice before use with the appropriate solvent (hexane, dichloromethane, or acetone) to avoid contamination. After use, the glassware was washed thoroughly with diluted nitric acid, rinsed and dried..

2.4 GC/MS/MS conditions

The vial contents were analyzed using a 7890B gas chromatograph coupled to a 7010B triple quadrupole mass spectrometer (Agilent Technologies, California, USA). A volume of 1 μL was injected in the splitless mode through an injector equipped with an insert containing glass wool. The syringe was rinsed

four times before and after each injection with hexane. An RTX-PCB (dimensions 15 m x 0.25 mm x 0.25 μm) apolar fused silica column (Restek, Lisses, France) was used for separation. The heating program started at 58°C, followed by an initial temperature ramp of 45°C/min until reaching 250°C and then a second temperature ramp of 30°C/min to reach a plateau of 300°C, which was maintained for 8 min. The vector gas (Helium) flow rate was set to 1.5 mL/min and the source temperature to 230°C.

Multiple-reaction-monitoring (MRM) transitions, including quantifier and qualifier transitions, were monitored for each compound based on their intensity and specificity. The most intense and specific MRM transitions were selected for monitoring (Supplementary Info). The quantifier transition (271.9 > 236.9) was used to determine the analyte response from the peak area after calibration. Qualifier transitions were used to ensure that the measured peak corresponded to the analyte, rather than the matrix or other types of interference. The identification of CLD was confirmed when both its retention time and the ratio between the quantifier and qualifier were the same in the dust extract and calibration standards. The ^{13}C -CLD ISTD was used to calculate the concentration of the native CLD.

Identification and quantification data were acquired and processed using MassHunter B.10 software (Agilent Technologies, California, USA) and exported to Microsoft Excel 2016 to express the results.

2.5 QA/QC validation

The following parameters were assessed as part of the evaluation of the method performance: calibration, extraction recovery, precision, limits of detection (LOD) and quantification (LOQ), blank management, and conservation of the dust sample. We based our analytical development and validation on the NF T90-210 and SWGTOX Practices standards [29], [30].

Standards, ranging from 0.2 to 4 ng/mL and diluted in hexane, were injected to establish a five-point calibration curve, based on the relative response factor between the response of CLD and the response of ^{13}C -CLD. A quadratic model was used to fit the curve. This curve was used for quantification if (i) the determination coefficient (r^2) of the curve was > 0.995, (ii) the drift from the theoretical values for the calibration points, extracted dust control, and CLD external control was < $\pm 25\%$, and (iii) the concentration found in the dust samples was within the working range, without exceeding 10% of the

most concentrated calibration point. Otherwise, diluted extracts were prepared. Positive values for CLD were confirmed by comparing the retention time and MRM transition ratios between the calibration samples and dust samples.

Extraction efficiency was assessed by monitoring the area of ^{13}C -CLD ISTD in the samples, in comparison to the mean area of ^{13}C -CLD ISTD in the calibration standards.

The precision of the method was assessed in the presence and in the absence of matrix : without matrix, by spiking celite at two levels at 16 ng/g and 80 ng/g ; with matrix by spiking one of the anonymous dust sample from a Martinican dwelling also at 16 ng/g and 80 ng/g. These tests were carried out under reproducible conditions.

The limit of quantification (LOQ) was defined as the lowest concentration of CLD for which the relative standard deviation (RSD) of five replicate analyses was $< 20\%$ and the signal/noise ratio > 10 in accordance to Gonzáles et al. [31]. The limit of detection (LOD) was defined as the lowest concentration of CLD for which the signal/noise ratio was > 3 .

Dust samples were stored in amber glass vials at -18°C upon reception in the laboratory. Conservation of the concentrations in the dust sample was monitored over 10 months.

2.6 Exposure assessment

Dust from hand-to-mouth contact or object-to-mouth contact can be ingested involuntarily by both adults and children. The daily intake of CLD through dust ingestion was calculated using the following formula (1):

$$DI_{CLD} = \frac{[CLD]_{dust} \times Q_{dust}}{BW} \quad (1)$$

where DI_{CLD} is the daily intake of ingested CLD in ng/kg of BW/day, $[CLD]_{dust}$, the CLD concentration (P50, min and max) in dust in ng/g measured in this study, Q_{dust} the daily mass of matrice ingested in mg/day, estimated for dust at maximum values of 60 mg/d for adults and 100 mg/d for children [32], and BW body weight in kg, considered to be 80 kg for adults < 16 year old and 10 kg for children below

2 year old [33]. The exposure was then expressed as a percentage of the acceptable daily intake (ADI=170 ng/kg BW/d) updated by the French agency for food, environmental and occupational health, the most recent and protective reference value for humans for CLD [34], [35].

2.7 Statistical analysis

Descriptive statistics concern $n = 47$ out of 49 collected samples, belonging to 6 dwellings and 2 schools. Two samples were not included in the calculations due to an insufficient quantity of dust collected. The data were statistically analyzed and plotted using GraphPad Prism v10 and Excel 2016 software. Nonparametric Kruskal Wallis followed with Dunn's multiple comparisons test were used to indicate significant differences between groups (type of buildings and geographical area) while a Shapiro-Wilk test indicated a non normal distribution for urban groups.

3 Results

3.1 Method validation

3.1.1 Selection of extraction solvent

Various organic solvents, used individually or mixed, were tested for the extraction and the results are presented in Table 1. Dichloromethane allowed quantitative extraction of the molecule, with a stable recovery rate of 104% between assays (CV = 7%), but a purification step was needed to increase the clarity of the extract (Supplementary Info). Although hexane alone showed nearly null recovery rates, using a mixture of 85% hexane and 15% of a polar solvent, such as acetone, ethyl acetate, or diethyl ether, increased the extraction rate to up to 89% for the hexane/ethyl acetate mixture, without the need of a purification step. The hexane with ethyl acetate (85:15) solvent mixture was therefore selected for CLD extraction from the dust samples. As shown in Table 1, the absolute recovery is greater than 100% for this solvent, most likely because of passivation in the presence of matrix compared to calibration standards. The CLD accuracy, assessed using ^{13}CLD for quantification, is correct (89%). The internal standard is therefore needed for an accurate quantification of CLD.

Table 1 - Comparison of solvents for the ASE extraction step

Solvent	Accuracy (native CLD) (%)	CV (%)	Recovery rate (¹³ C CLD) (%)	CV (%)
Hexane	0	0	2	44
Dichloromethane	104	7	64	8
Acetone	57	39	46	3
Dichloromethane/Acetone (50 :50)	53	49	4	37
Hexane/Acetone (85:15)	87	4	179	7
Hexane/Diethyl ether (85:15)	69	9	218	7
Hexane/Ethyl acetate (85:15)	89	10	152	12

Recovery rate assays were carried out on n=3 repetitions with Martinique dust samples

3.1.2 Optimisation of the test sample mass

Dust represents a complex matrix likely to interfere with the analysis. Reducing the sample mass from 50 to 25 mg had an impact on the variability of the results, improving reproducibility by a factor of 2 (Table 2). The smaller test sample mass of 25 mg generated less matrix interference and was therefore selected for the analytical protocol.

Table 2 – Dust sample weight determination (mg) for extraction

Sample intake	Recovery rate (native CLD) (%)	CV (%)	Recovery rate (¹³ C CLD) (%)	CV (%)
25 mg	99	12	116	17
50 mg	104	21	70	21

Extraction efficiency (%) and variability (%) for n=3 repetitions with Martinique dust samples

3.1.3 Extraction recovery

The recovery of ¹³C-CLD, measured in 46 dust samples was 115 ± 30 %. This variability is a reflection of the variability of dust matrix between the different samples.

3.1.4 Precision of the method

The precision of the method, assessed at two spiking levels, and with and without matrix is shown in Table 3. Both the accuracy and precision were satisfactory, with the accuracy ranging from 100 to 115% and the CV from 10 to 14%.

Table 3 – Method precision and accuracy for n replicates under reproducible conditions

	Target concentration (ng/g)	n (under reproducible conditions)	Measured concentration	Accuracy (%)	CV (%)
Without matrix	16	9	16.5 ± 1.8	103%	12 %
With matrix	16	8	18.5 ± 2.3	115%	14 %
Without matrix	80	10	79.7 ± 8.0	100%	10 %
With matrix	80	10	83.8 ± 7.7	105%	10 %

3.1.5 Limits of detection and quantification

Validation criteria for the LOQ were fulfilled at 0.4 µg/L in extracts and the LOD at 0.2 µg/L. In the matrix, the LOQ was equivalent to 16 ng/g and the LOD to 8 ng/g. A value of 4 ng/g (½ LOD) was assigned to samples with concentrations below the LOD for graphical representations and calculations.

3.1.6 Blank and interference management

Contamination was examined in the solvents and extracted samples. According to our validation criteria, signal responses in blanks had to be < 50% of the LOQ. All blanks showed CLD concentrations below the LOQ in Table 3, indicating no contamination throughout the preparation and injection process.

According to a study of Bichon et al. [36], certain compounds, such as nonachlor, can interfere with CLD detection. The close retention time with molecules of interest could lead to an overestimation of CLD concentrations. Our results for CLD interference showed good separation, with no possible confusion between compounds (Supplementary Info).

3.1.7 Conservation

Conservation tests over time are shown in Supplementary Info. After 10 months, the variation of the concentration did not exceed 30%. The storage of dust samples at -20°C in glass amber vials is therefore validated for at least 10 months.

3.2 CLD detection in dust samples

CLD concentrations were below the LOD (8 ng/g) for 40 % (n=19) of samples, between LOD and LOQ for 19 % (n=9) of samples, and 40 % (n=19) provided quantifiable concentrations (> 16 ng/g). The median concentration was estimated at 12 ng/g, between the LOD and LOQ. Otherwise, the quantified

CLD concentration ranged from a minimum of 51 ng/g to a maximum of 247 ng/g. During the analysis, we found no CLD in the SRM 2585® reference dust. Household dust samples were compared to school dust samples and those obtained in rural areas to urban areas (Fig. 1). We observed significant differences between the urban and rural areas for both types of buildings. Median concentration in rural household and school dust samples (110.1 ng/g and 80.8 ng/g respectively) are higher than urban household and school dust samples (median concentrations below LOD).

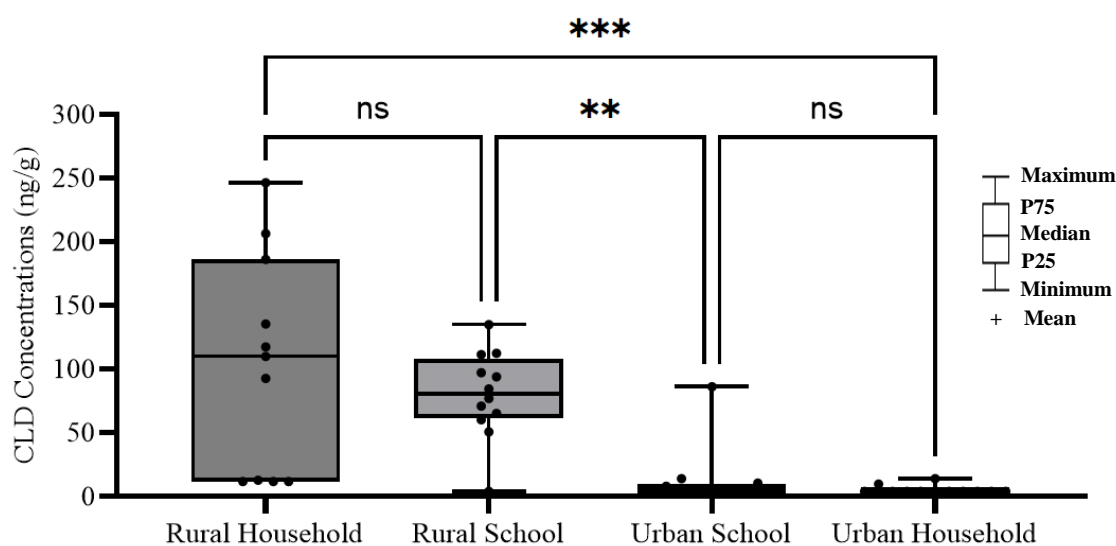
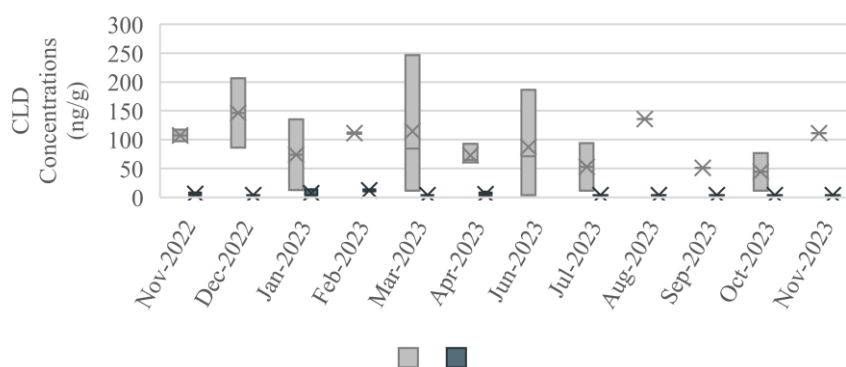


Fig. 1 - CLD concentrations in indoor dust (ng/g) according to building type and urban or rural CLD area. ns = non significant difference ($p_{\text{value}} > 0.999$). ** = significant difference ($p_{\text{value}} < 0.006$). *** = significant difference ($p_{\text{value}} < 0.0007$). Shapiro-Wilk test indicated a non normal distribution for urban groups ($p_{\text{value}} < 0.001$)

3.2.1 Temporal CLD concentrations in dust samples

CLD concentrations over time based on the one-year study are presented in Table 3 (details in Supplementary Information). CLD concentrations in rural areas did not appear to vary over time. In urban areas, the CLD concentrations remained low (< LOQ) throughout the year. Constant contamination in dust in rural areas observed. Highest median CLD concentrations in dust (> 100 ng/g) were observed in rural areas during the months of November and June, except in April.

(A)



(B)

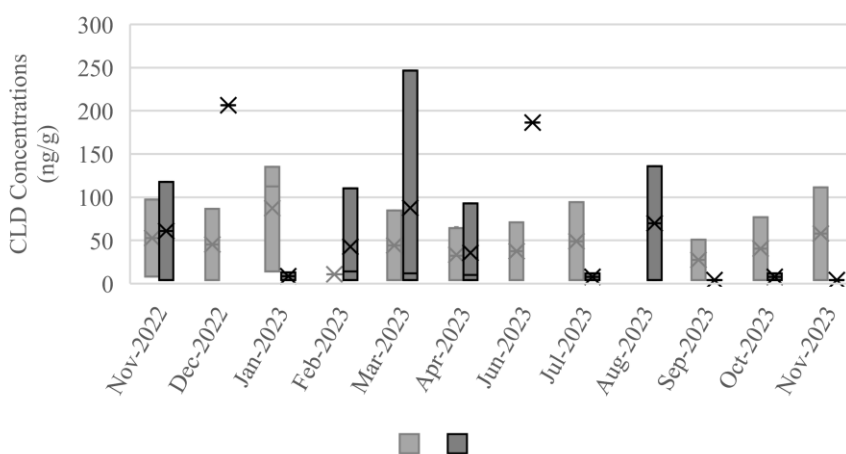


Fig. 2 – Evolution of CLD concentrations in Martinique dust samples in one year, in school (n=24) and household (n=23) (A) and rural area (n=24) and urban area (n=23) (B)

3.3 CLD exposure assessment via indoor dust

Daily exposure due to the intake of CLD via indoor dust in 6 dwellings and 2 schools in Martinique, and associated ADI percentages, are presented in Table 4. It varies from 0.006 to 0.185 ng/kg BW/d and 0.08 to 2.47 ng/kg BW/d for adults and children, respectively. LOD value of 8 ng/g was used for the calculation.

Table 4 - Daily intake of CLD through dust ingestion based on contamination data from 6 dwellings and 2 schools (associated ADI percentage, according to the value of Anses, 2021). DI was calculated with median concentrations obtained (P50), minimum concentrations obtained (MIN) and maximum concentrations obtained (MAX).

DI (ng/kg BW/day)	P50	MIN	MAX
Adults	0.009 (0.006%)	0.006 (0.004%)	0.185 (0.11%)
Children	0.12 (0.07%)	0.08 (0.05%)	2.47 (1.46%)

4 Discussion

4.1 Optimization of the analytical method

4.1.1 Dust sample mass

The mass of the dust, as well as that of the celite used, had an impact on CLD extraction. Dust is a complex matrix [17], [37], [38] and matrix effects can be observed : weak and strong interactions exerted by the matrix components could significantly retain CLD, making it difficult to extract from the dust [39]. Lower yields were thus observed for a higher sample mass (> 50 mg). Due to the reduction of the matrix effect, the LOD and LOQ were maintained, even when the sample mass was lowered. Reducing the mass of the dust sample for CLD analysis makes it possible to keep a larger amount of residual dust and conduct further exposure studies for other pollutants.

Furthermore, a mass of > 50 mg of dust requires the introduction of a purification step to eliminate interference, which has a significant impact on the cost of the analysis and the risk of clogging the GC/MS/MS instrument. To offset the matrix effect, celite is used to disperse the dust during the

extraction and fill the volume of the extraction cells. Although chemically inert, celite could have an antagonistic effect on this compound if present in too high a proportion. Without molecular attachment to other attractive compounds, unstabilized CLD would be extracted less well from the dust.

4.1.2 Organic matter content

During analytical development, composite dust samples with an organic content ranging from 20 to 60% were used. It is generally agreed that organic matter has an impact on organochlorine pesticide retention in soil and, by extension, in the dust matrix [40]. We could have expected lower recovery rates for CLD extractions due to higher absorption with a high organic matter content (approximately 60%). However, we did not observe significant differences in CLD recovery depending on the organic matter content of the three types of composite dust, showing that the extraction method is fit for purpose and efficient for a large range of dust types.

4.1.3 Organic solvent

Due to electronegative chlorine atoms, the CLD molecule has many dipoles, making it a polar compound. The caged-structured conformation of CLD makes it highly stable in the environment [41]. This physical-chemical aspect suggested extraction using apolar solvents. A polar solvent could be used to pull CLD out from the matrix, combined with an apolar solvent to stabilize its extraction. This association was considered due to the compound's affinity for polar solvents, even though it is typically poorly soluble in water [26]. A dichloromethane/acetone mix (50:50) could have been used. However, extraction with these versatile solvents requires an additional purification step before injection into GC/MS/MS. To facilitate routine operations and reduce handling time and the materials required, it is better to limit the number of preparation steps. Using only a small portion of polar solvent to extract the CLD from an apolar solvent limits the extraction of other potentially interfering substances.

4.1.4 Extraction condition

During assay development, we tested microwave extraction (data not shown), with satisfying recovery rates for CLD. However, the use of microwaves during extraction could have deleterious effects on the stability of CLD in the extracts. As reported in recent studies [42], [43], a decrease in the concentration

found in matrices was observed using microwaves, likely linked to CLD degradation. This extraction process may therefore not be a suitable method for this molecule and should be avoided.

The extraction with dichloromethane is not specific to CLD and other materials were also present in the extract with a purification step needed. The recovery of ^{13}C -CLD (64%) might have been lowered by this step. When using a hexane/ethyl acetate mixture, a high recovery rate was observed, which may be attributed to matrix effects that were not mitigated by any purification step. Nevertheless, the method demonstrated acceptable accuracy (89%). The 85/15 ratio was selected based on prior reports utilizing a hexane/diethyl ether system [44]. In our study, ethyl acetate proved to be a more effective solvent than diethyl ether for extracting CLD from the dust matrix (89% vs. 69%).

4.2 Transfer of CLD from outdoor to indoor environments

4.2.1 Soil contamination and transfer from outdoor to indoor environments

The dust samples were regularly collected from two places, located in the center (Lamentin) and North (Macouba) of the Island. These two areas do not represent the entire island of Martinique. Otherwise, the interactive map of CLD pollution in soil, published by the Geomatics Resource Center and Geographic Data Infrastructure (GéoMartinique - Pollution des sols par la chlordécone en Martinique), indicates the presence of areas at risk of contamination also in the eastern part of the island (historical presence of banana crops). According to Devault [45], CLD concentrations in 600 soil samples ranged from 0.01 to 17.4 $\mu\text{g/g}$ of soil, whereas the minimum and maximum quantified values of CLD in indoor settled dust in this study were respectively 51 and 247 ng/g . As such, CLD concentrations in soil remain higher than those found in indoor settled dust by at least two orders of magnitude. The contamination of CLD does not come from domestic sources, but practically all from external particles. These particles are transported by erosion, wind and human transfers, and are deposited in the indoor environment. In addition, the structure of buildings in the FWI had large, unglazed openings. This design allows outdoor contamination, such as soil particles and pollution, to enter more easily due to wind, ventilation, and human activity. During Lent, from February to April, strong winds contribute to the transfer of outdoor particles indoors, which settle after the windy period, concentrating pollutants such as CLD in dust. This

could explain the higher observed CLD concentrations in households in June. Also, the duration of the 1-year sampling period may be insufficient to accurately capture long-term trends in CLD deposition or to account for the impacts of extreme weather events, such as hurricanes. Contamination might differ between ground-level houses and upper-floor flats, not studied here, suggesting further research that includes building specifications. Finally, the composition of indoor dust may vary according to the activity of the inhabitants. Clothes and shoes worn outdoors for the entire day can pick up particles from the outside and then, once back home or indoors, contribute to contamination from outdoors [46]. Pets, whose fur retain particles of potentially contaminated soil, can bring pollution indoors [47]. Further studies on indoor dust pollution in different locations and in a larger number of homes and schools allow to complete this initial data to obtain a more representative overview of the situation.

4.3 Exposure assessment via indoor dust ingestion

This study provides a first glimpse of CLD contamination in indoor dust. According to obtained results, dust ingestion alone does not appear to pose a risk to human health, with only 0.11 to 1.46% of the ADI. Medians were used to calculate daily intake. The value of the median between the LOD and the LOQ has a measurement uncertainty due to an unquantified value. However, using this measure or LOQ, has no impact on the assessment of exposure, which remains very low (<1%) via the ingestion of contaminated dust. Most importantly, the estimations of human exposure provided here are also very low compared to dietary exposure data for the FWI population estimated in 2013. These ranged from 20 to 700 ng/kg BW/d depending on the population (adult/children) and the more or less conservative assumptions made during the calculations. Moreover, it will be relevant to compare these data with updated dietary exposure data knowing that it is assumed that dietary exposure of FWI population are decreasing taking into account the actions implemented by public health authorities, associations, and residents. However, the small number of collected samples and the restricted areas covered by this study does not make it possible to provide a confident representation of CLD dust contamination in indoor environments and exposure of FWI populations.

5 Conclusion

Many analytical methods are available to investigate CLD in environmental matrix, such as water, soil, biological fluids, and foodstuffs, but thus far not in indoor settled dust. The method developed provides satisfactory and reproducible results for CLD analysis in this complex matrix. The analytical performance of this method enables us to measure low CLD concentration levels (LOD = 8 ng/g). Accelerated extraction using dichloromethane is more suited for extracting CLD from indoor dust rather than micro-wave extraction. GC/MS/MS was used for its analysis.

This study measured CLD for the first time in the dust of 6 indoor dwellings and 2 schools in the French West Indies on a small number of samples (n=47). The median CLD concentration in indoor dust samples is estimated at 12 ng/g, between LOD and LOQ. Preliminary estimation of CLD exposure from indoor dust ingestion based on these 47 samples indicates that the contribution of CLD exposure does not exceed 0.1 and 1.5% of the ADI in adults and children, respectively, and are very low compared to dietary exposures estimated in 2017 for the FWI population.

These initial results suggest that exposure through ingestion of CLD contaminated dust contribute to a negligible part to ADI. However, this conclusion obtained on a very limited number of samples needs to be confirmed by a larger study.

Some recommendations can be made for the reduction of contamination indoor. The use of different pairs of shoes dedicated to indoor and outdoor use and systematically wiping feet before entering the house could help to reduce contamination of the indoor environment. In addition, frequent cleaning of the indoor environment and cleaning surfaces (furniture, floors, objects) helps to limit indoor dust pollution [48], [49], [50], [51]. Wet cleaning is also recommended to more efficiently capture settled dust, without the resuspension of particles into the air [52].

6 References

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7 Environmental implication

Chlordecone is a pesticide widely used on banana plants in the French West Indies. Although its use has been banned since 1993, the molecule is still present in soils and contaminates the trophic chains up to humans. Contamination levels in food, soil, and water, have already been described, but not indoor dust. As humans spend most of their time indoors, dust can be a vector that contributes to human exposure. This study, through the development of a novel analysis method, completes the data by assessing chlordecone contamination of indoor dust.

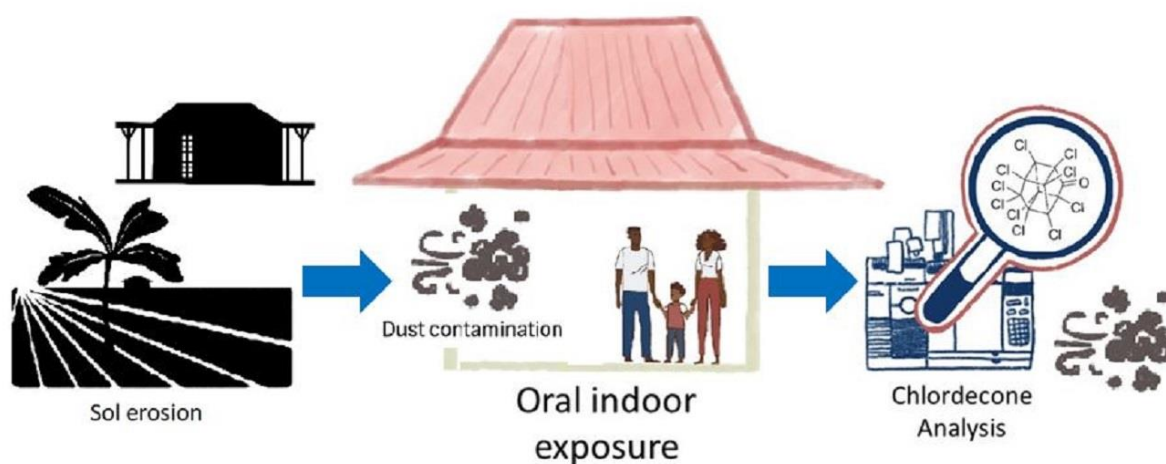
Declaration of interests

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.

☒ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Barbara Le Bot reports financial support was provided by French Biodiversity Office. Marie Dufresne reports financial support was provided by University of Rennes EHESP School of Public Health. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Graphical abstract



7.1 Highlights

- First analysis of chlordecone (CLD) in indoor dust using a novel GC/MS/MS method
- Limit of detection of 8 ng/g and limit of quantification of 16 ng/g
- Quantified CLD concentrations in dust samples ranged from 51 to 247 ng/g
- Higher concentrations are found in rural areas than in urban areas
- Indoor dust ingestion appears to be a negligible pathway for CLD exposure (< 1,5%)