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# Analysis of Semi-Volatile Organic Compounds (SVOCs) in Micro-Chamber Thermal Extracted Emissions from Household Dust

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## Abstract

Settled house dust acts as a critical sink and surrogate medium for indoor air quality assessment and exposure monitoring. Many hazardous chemical compounds, including biocides and semi-volatile organic compounds (SVOCs), exhibit prolonged stability when bound to dust matrices, often accumulating in significantly higher concentrations than in surrounding air. In this study, household dust samples collected from vacuum cleaner bags in Nigerian residences were sieved (<150 µm fractions) and subjected to an innovative thermal extraction technique. Aliquots were heated directly within a micro-chamber thermal extractor (µ-CTE) at temperatures ranging from 40 °C to 120 °C. The emitted chemical vapors were captured using sorbent tubes containing Tenax (Q-Tenax) and subsequently analyzed via thermal desorption-gas chromatography-mass spectrometry (TD/GC/MS). The systematic detection of key target analytes confirmed the viability of µ-CTE heating as a rapid, solventless approach to vaporize and quantify loose SVOC fractions. To validate this method, comparative evaluations were conducted against conventional solvent extraction-GC/MS method. While conventional solvent extraction provided total mass loadings via aggressive matrix dissolution, the µ-CTE method effectively characterized the readily bioaccessible, vaporizable fractions. This method demonstrates strong repeatability with relative standard deviations (RSD) generally between 9% and 27%, presenting a rapid, green analytical tool for indoor environmental health assessments.

## Introduction

Growing public health concerns regarding chronic chemical exposure within indoor exposure in residential environments have driven rigorous investigations into indoor contaminant loadings (Weber et al., 2011). Modern humans spend over 90% of their lifespan inside buildings, where they encounter complex mixtures of volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) off-gassing from architectural coatings, polyvinyl chloride (PVC) floorings, plasticizers, electronics, and consumer goods (Mohammed & Crump 2013). Due to their characteristically low vapor pressures, SVOCs slowly migrate from host polymer matrices into the surrounding indoor

air, partition onto airborne suspended particulate matter, and ultimately settle out into indoor dust repositories contact (Morawska, 2004; Morawska & Salthammer, 2006).

Settled household dust serves as an effective surrogate and archival medium for indoor pollutant monitoring. It is easily sampled via everyday vacuum bags or specialized collection devices, providing stable, integrated concentration metrics over time. For many persistent SVOC groups, including phthalate esters and flame retardants, concentration profiles in dust are substantially higher than those found in air. These contaminated dust deposits present a direct exposure hazard to human occupants, particularly infants and toddlers through intentional or involuntary ingestion via hand-to-mouth pathways, dermal contact, and the inhalation of re-suspended fractions. Consequently, modeling the phase partitioning, bioaccessibility, and dynamic source-sink interactions of dust-bound pollutants is essential for evaluating internal exposure doses and associated health risks, such as endocrine disruption, respiratory diseases, and neurodevelopmental impairments (Raffy, *et al*. 2018; Bi *et al*., 2018).

The conventional determination of SVOC profiles in settled dust relies on liquid solvent extraction (e.g. pressurized solvent extraction, Soxhlet extraction, or sonication) using toxic organic solvents like dichloromethane or hexane, followed by laborious filtration, concentration, and cleanup steps before gas chromatography-mass spectrometry (GC/MS) analysis. Although robust for quantifying absolute total mass loadings, solvent extraction destroys the structural matrix and fails to differentiate between tightly encapsulated polymers and the freely vaporizable, bioaccessible pollutant fractions that present immediate health risks. Furthermore, these workflows are time-consuming and generate hazardous chemical waste (Guan, *et al.*, 2024, Al-Alam, et al., 2024).

To overcome these challenges, this study evaluates the application of a Micro-Chamber Thermal Extractor (μ-CTE) as a direct, solventless sampling gateway coupled with Thermal Desorption-Gas Chromatography-Mass Spectrometry (TD/GC/MS). While the μ-CTE was originally engineered to characterize rapid material emission dynamics from planar building components, textiles, and coatings, this work explores its novel modification to screen and desorb organic contaminants directly from loose household dust matrices. By adjusting operational variables like mass loading, temperature, and sweeping carrier flows, this paper presents a rapid, reproducible, and green analytical methodology. The performance of this system is validated using residential samples from distinct geographical regions (the UK and Nigeria) alongside National Institute of Standards and Technology (NIST) Standard Reference Material (SRM 2585).

## Methodology

### Method Development and Calibration

Analytical standard stock solutions comprising eight target representative chemicals (benzene, hexanal, nonanal, limonene, diethyl phthalate [DEP], diisobutyl phthalate [DIBP], dibutyl phthalate [DBP], and diethylhexyl phthalate [DEHP]) were formulated by dissolving 0.4 g of each chemical into a 10 mL volumetric flask with high-purity methanol, achieving an initial target stock concentration. Serial dilutions were systematically performed to generate multi-point calibration standards down to an operating level of 40 mg $mL^{-1}$.

For instrumental calibration, 1 μL aliquots of each diluted standard solution were precisely spiked onto conditioned Q-Tenax (Tenax-TA) sorbent tubes, immediately followed by the application of 5 μL of n-toluene as an internal standard to normalize analytical variance. Spiked calibration tubes were subsequently processed via the TD/GC/MS assembly. Linear calibration curves were constructed by plotting the integrated detector peak area responses against known reference mass inputs via automated quantitation software.

## Dust Sample Processing and μ-CTE Extraction Setup

Household settled dust matrices were harvested from residential vacuum cleaner bags across selected homes. Twenty six (26) samples were collected within IDH location, and another 26 samples from FH homes within the same location. An SRM (standard reference dust was obtained from US and analysed alongside the vacuum cleaner bag dust samples. The crude dust was processed in a laboratory fume hood using a stainless-steel mesh sieve with a 150 μm aperture to isolate the fine particle fraction (<150 μm), which represents the dominant vehicle for human dust ingestion and dermal adherence. Personnel wore mandatory respiratory and eye protections during sieving to mitigate dust inhalation risks. The isolated fractions were preserved inside sealed, pre-cleaned glass jars under dark storage conditions.

Direct thermal extractions were conducted using a commercial Micro-Chamber Thermal Extractor (μ-CTE; Markes International Ltd., Llantrisant, UK). The instrument consists of six isolated cylindrical micro-chamber cells, each measuring 28 mm in depth by 45 mm in diameter.

The operational extraction parameters were adapted from material emission protocols outlined by Brown and Crump (2008), with modifications for loose powder matrices. Approximately 5 g of sieved dust was accurately weighed on a calibrated five-figure analytical balance (Sartorius®) into individual heated stainless-steel chamber pots. To stabilize the powder within the swift gas stream, each pot was lined at the base with a glass micro-fibre filter paper (GF/A) previously pre-conditioned in an oven at 200°C for 1 hour. A matching sheet of conditioned GF/A filter paper was placed over the dust sample bed. This dual-filter encapsulation prevented dust particles from becoming entrained in the air stream and contaminating the sampling lines or the sorbent bed.

Duplicate pots were processed for each discrete dust sample alongside a baseline filter-only control pot to monitor system backgrounds. The μ-CTE block temperature was adjusted to its operational peak of 120 °C, and a high-purity, conditioned zero-air carrier flow was driven through all chambers at a constant rate of 70 mL $min^{-1}$. Volatilized organic emissions swept out of the cell were trapped directly onto attached Q-Tenax sorbent collection tubes. The initial screening runtime was set to 2 hours, using sequential tube replacements every 40 minutes to track desorption rates over time.

## Method Optimization and TD/GC/MS Conditions

Initial screening utilizing the 5 g sample mass resulted in severe analytical detector saturation due to the abundance of target phthalates and aldehydes outgassing from the dust. To achieve accurate quantification within the linear dynamic range of the instrument, three modifications were introduced:

- The sample mass loading was scaled down from 5 g to 1 g.
- The total thermal extraction window was reduced from three consecutive 40-minute stages to a single 40-minute sampling sequence, minimizing tube consumption and preventing analyte breakthrough.
- The TD/GC/MS split injection ratio was increased to 10:1.

Desorbed tubes were evaluated on an automated thermal desorption system (TD-100, Markes International Ltd.) coupled to a Gas Chromatograph-Mass Spectrometer (GC/MS). Sorbent tubes underwent primary thermal desorption at 320 °C for 20 minutes under high-purity helium carrier gas, re-focusing volatile analytes onto a secondary Peltier-cooled cold trap maintained at -10 °C. Once primary desorption concluded, the cold trap was rapidly heated to 320 °C to release the analyte band onto the capillary gas chromatography column for separation and mass spectral detection.

## Data and Statistical Analysis

Replicate analytical datasets were compiled, and descriptive statistical profiles (means, standard deviations (SD), and relative standard deviations (RSD %) were calculated using Microsoft Excel and automated chromatographic quantitation platforms.

## Results and Discussion

### Screening Characterization and Quantification Boundaries

Initial un-split μ-CTE extractions provided an overview of the volatile and semi-volatile chemicals present within household dust matrices indicating the presence of a wide range of VOCs and SVOCs released from the heated dust. A representative Total Ion Chromatogram (TIC) acquired during the first extraction stage is shown in Figure 1.

Some target compounds were selected for detailed quantitative analysis which included benzene, hexanal, nonanal, limonene, DEP, DIBP, DBP, and DEHP. These selections were based on their documented toxicity profiles, structural representation across various chemical classes, and regular occurrence in indoor environmental health literature.

The quantitative operational boundaries of the calibrated TD/GC/MS configuration were derived from initial multi-point standard runs, as detailed in Table 1. The lower limits of quantification (LLOQ) ranged from 5ng to 10ng on-column, while the upper limits of quantification (ULOQ) extended from 1200 ng up to 5000 ng.

## Evaluation of Extraction Kinetics and Reproducibility

Quantification datasets collected across sequential intervals verified that the bulk emission mass of both volatile aldehydes and higher-molecular-weight phthalates desorbs during the primary phase of heating. Tables 2 and 3 outline mass values extracted during the first and second sampling intervals, respectively, utilizing 5 g dust samples DH (Table 2) and FH (Table 3).

**Table 1**: Instrumental Lower and Upper Limits of Quantification (LOQ)

| Compound Name | Lower Limit of Quantification (ng) | Upper Limit of Quantification (ng) |
|---|---|---|
| Benzene | 5 | 5000 |
| Hexanal | 5 | 4000 |
| Nonanal | 5 | 4000 |
| Diethyl Phthalate (DEP) | 5 | 4000 |
| Diisobutyl Phthalate (DIBP) | 5 | 1600 |
| Dibutyl Phthalate (DBP) | 5 | 1200 |
| Diethylhexyl Phthalate (DEHP) | 10 | 1200 |

To confirm method repeatability following optimization, five replicate of 1 g aliquots of dust (DH1) and dust (FH) were analyzed under identical parameters. The statistical summaries are shown in Table 4

The relative standard deviations (RSD) across the replicates were generally between 9.7% and 27.3%. This is within acceptable boundaries for complex trace environmental sample matrices. The higher variance observed for benzene (46.6%) is attributed to its low baseline concentrations near the detection limit. The absence of benzene in the dust (FH) samples highlight differences in indoor fuel usage, ventilation architectures, or structural material types between the sampling regions.

## Comparative Extraction Analysis: Solvent versus Thermal Micro-Chamber

The VOCs concentrations obtained via μ-CTE thermal extraction were compared directly with those from aggressive organic solvent extraction workflows, as well as the obtained commercial standard dust (SRM) sample as summarized in Table 5.

The results obtained in this study were compared with those obtained in other studies which showed similarity in the dominance of DEHP among other phthalates in house dusts. The median concentrations of the phthalates found in this study were compared with those reported from various cities from different geographical locations. DEP was found to be higher in the Nigerian dusts (this study) than those reported from other cities and the median for DEHP in this study was the least as compared to those reported from other studies (Table 6).

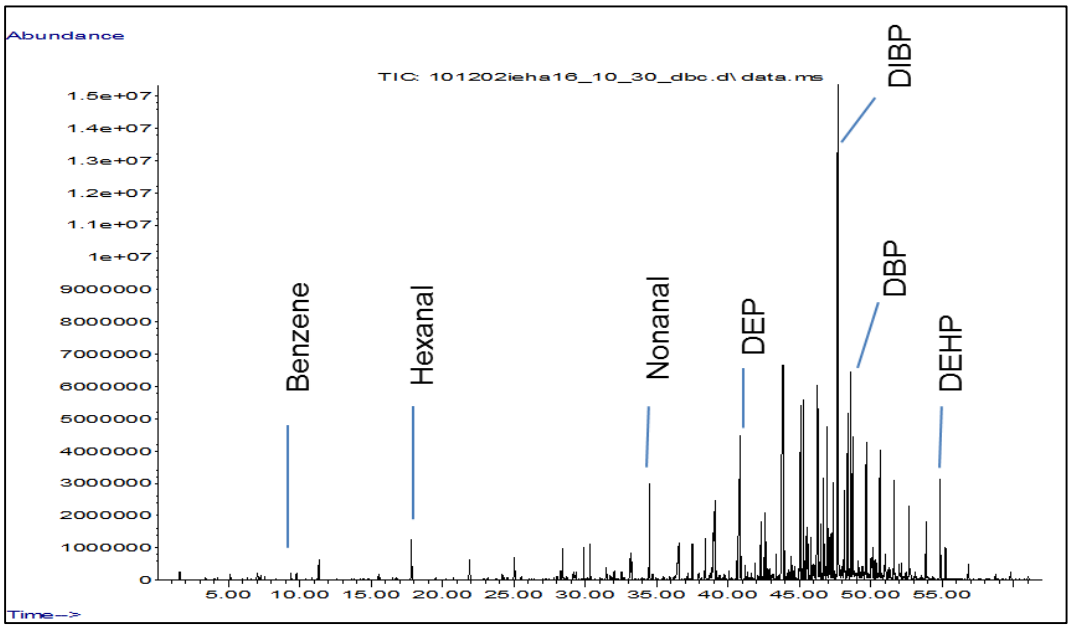


**Figure 1**: Total Ion Chromatogram (TIC) profiles showcasing characteristic volatile and semi-volatile emissions isolated from household dust beds during μ-CTE thermal processing.

**Table 2: Target Analyte Mass Recovered on Tube Assemblies (Sampling Interval 1)**

| Compound | Blank sorbent Tube (ng) | Chamber Control Pot (ng) | Dust DH1 Pot 3 (ng) | Dust DH1 Pot 4 (ng) | Dust DH2 Pot 5 (ng) | Dust DH2 Pot 6 (ng) |
|---|---|---|---|---|---|---|
| Benzene | <5 | 31 | 56 | 58 | 34 | 32 |
| Hexanal | <5 | 89 | 2474 | 2789 | 1883 | 1885 |
| Nonanal | <5 | 79 | ~8576* | ~8482* | ~6845* | ~6863* |
| DEP | <5 | 73 | 149 | 155 | 148 | 145 |
| DIBP | <5 | 130 | ~4676* | ~4654* | 1041 | 1082 |
| DBP | <5 | 11 | 494 | 500 | 404 | 356 |
| DEHP | <10 | 96 | 520 | 488 | 391 | 387 |

** Note: Concentration values are estimated approximations as integrated signal yields surpassed the calibrated upper limit of quantification (ULOQ).*

**Table 3**: Target Analyte Mass Recovered on Tube Assemblies (Sampling Interval 2)

| Compound | Blank Sorbent Tube (ng) | Chamber Control Pot(ng) | Dust FH1 Pot 1 (ng) | Dust FH1 Pot 2 (ng) | Dust FH2 Pot 3 (ng) | Dust FH2 Pot 4 (ng) |
|---|---|---|---|---|---|---|
| Benzene | <5 | 25 | 59 | 60 | 54 | 53 |
| Hexanal | <5 | <5 | 1285 | 1280 | 1140 | 1187 |
| Nonanal | <5 | 20 | 3571 | 3531 | ~4081* | ~4013* |
| DEP | <5 | 3 | 213 | 218 | 123 | 142 |
| DIBP | <5 | 145 | ~6619* | ~6581* | ~2984* | ~2874* |
| DBP | <5 | 6 | 952 | 986 | 870 | 885 |
| DEHP | <10 | 12 | ~1268* | ~1230* | ~1161* | ~1185* |

** Note: Concentration values are estimated approximations as integrated signal yields surpassed the calibrated upper limit of quantification (ULOQ).*

**Table 4**: Method Repeatability and Target Analyte Concentration Profiles

| Compound | Dust (DH) Mean (ng $g^{-1}$) | Dust (DH) SD | Dust (DH) RSD% | Dust (FH) Mean (ng $g^{-1}$) | Dust (FH) SD | Dust (FH) RSD% |
|---|---|---|---|---|---|---|
| Benzene | 14 | 7 | 46.6 | ND | — | — |
| Hexanal | 1732 | 228 | 13.1 | 1524 | 281 | 9.7 |
| Nonanal | 2892 | 281 | 9.7 | 1491 | 128 | 8.6 |
| DEP | 157 | 36 | 23.0 | 94 | 24 | 25.4 |
| DIBP | 1805 | 346 | 19.2 | 1449 | 142 | 9.8 |
| DBP | 654 | 91 | 13.8 | 457 | 125 | 27.3 |
| DEHP | 886 | 80 | 9.1 | 586 | 139 | 23.8 |

*ND = Not Detected (below method detection threshold).*

**Table 5**: Comparative Matrix concentrations between Solvent extraction and μ-CTE

Mean concentrations (ng $g^{-1}$) ratios in the Solvent Extracts and CTE Extracts

| | IDH Dusts (n=26) | | | FH Dusts (n=26) | | | SRM Dust (n=6) | | |
|---|---|---|---|---|---|---|---|---|---|
| Compound | SE | μ-CTE | ratio | SE | μ-CTE | ratio | SE | μ-CTE | ratio |
| Hexanal | ND | 1063 | - | ND | 598 | - | ND | 857 | - |
| Limonene | ND | 222 | - | ND | ND | - | ND | 23 | - |
| Nonanal | 16,651 | 5785 | 3 | 6431 | 2560 | 3 | 2993 | 194 | 15 |
| DEP | 8530 | 916 | 9 | 1806 | 355 | 5 | 1343 | 1422 | 1 |
| DIBP | 22,802 | 1707 | 13 | 2185 | 701 | 3 | 2699 | 1169 | 2 |
| DBP | 25,454 | 1703 | 15 | 2844 | 630 | 5 | 2226 | 1073 | 2 |
| DEHP | 41,768 | 4121 | 10 | 25,442 | 1951 | 13 | 4776 | 2075 | 2 |

SE= solvent extracts μ-CTE =thermal extracts ratio= SE/μ-CTE *Limonene only seen in CTE extract

**Table 6**: Concentration (μg $g^{-1}$) of organic chemicals (phthalates) in house dusts from various studies

| Location | Sampling method | Solvent type | Analytical method | DEP | DIBP | DBP | DEHP | Reference |
|---|---|---|---|---|---|---|---|---|
| Danish homes (500) | ALK dust filter | n-hexane: diethyl ether | GC/MS | GM-3.1 Med-1,7 | GM-16.6 Median-27 | NA | GM-220 Median-210 | Langer *et al.*, 2010 |
| Beijing (11) | Sweep/ wipe | Hexane : acetone | GC/MS | Med-0.4 Range-0.1-0.6 | Median 12.6 Range-7.2-84 | Median 18.9 Range-7-32 | Median 156 Range-47.6-883 | Guo and Kannan, 2011 |
| Shanghai (21) | Sweeping and wiping | Hexane : acetone | GC/MS | Med-0.4 Range nd-46 | Median 33.6 Range 7.0-86 | Median 26.9 Range 1.5-96 | Med-319 Range-117-1380 | Guo and Kannan, 2011 |
| Albany (33) | Vacuum cleaner bags | Hexane : acetone | GC/MS | Med-2.0 Range 0.7-11.8 | Median 3.8 Range 0.7-34 | Median 13 Range 4.5-94 | Med-304 Range 37.2-9650 | Guo and Kannan 2011 |
| Kuwaiti homes (21) | Vacuum cleaner bags | Hexane : acetone | GC/MS | Median 1.8 Range-0.1–16 GM-1.5 | NA | Median-45 Range-8–160 GM-51 | Median-2256 Range-380–7800 GM-1700 | Gevao *et al.*, 2012 |
| Nigerian homes (26) | Vacuum cleaner bags | Hexane : acetone | GC/MS | AM-8.5 Median-4.0 Range-2-25 GM-5.4 | AM-22.8 Median-24.8 Range-11-37 GM-21.8 | AM-25.5 Median-26.7 Range-12-46 GM-24.6 | AM-41.8 Median-43.3 Range-12.2-94 GM-36.4 | This study |

The analytical data revealed significantly higher concentration readings for phthalates and long-chain aldehydes within the organic liquid solvent extracts compared to the μ-CTE gaseous desorbed outputs. This trend is demonstrated by the high Solvent/CTE extraction ratios, which reached up to 15 for DBP in residential dust samples. This difference is consistent with the extraction mechanisms of each method: liquid solvent extraction dissolves the organic matrix, capturing the total internal chemical load. In contrast, the μ-CTE method relies on temperature-driven vaporization, releasing only the volatile, loosely bound, or surface-adsorbed chemical fractions without destroying the dust structure.

Notably, highly volatile constituents like hexanal and limonene were not detected in the solvent extracts. This is due to evaporation losses during the solvent concentration and nitrogen blow-down steps required for liquid injections. The μ-CTE system avoids these losses by channeling the emitted gas phase directly onto sorbent tubes, preventing the loss of volatile fractions. The μ-CTE approach provides a distinct analytical advantage for indoor environmental health assessments. Rather than measuring total chemical content, it quantifies the readily vaporizable and bioaccessible fractions-the portions most likely to migrate into indoor air or be absorbed via human exposure pathways.

## Conclusion

A novel method of extracting organic chemical emissions from dust was further developed that involved heating of the dusts directly in a micro chamber (μ-CTE™) and collection of emissions on sampling tubes. The sampled tubes were analysed by TD/GC/MS. This provided a relatively quick analysis to characterise the dust and provide a measure of the amounts of chemicals in the dust that are quite readily available for release.

The mean chemical emissions were higher in the solvent extracts than the thermal extracts while hexanal and limonene were detected only in the thermal extracts. The ratios of the concentrations from the solvent and thermal extracts were in the range of 3 to 15 fold.

The μ-CTE thermal extraction followed by TD/GC/MS analysis offers a relatively rapid and repeatable method to compare the most available fractions of analytes and has been found to be a good method of investigating chemicals in matrices such as dust in homes

## Recommendations

This study demonstrates the viability of using a Micro-Chamber Thermal Extractor (μ-CTE) coupled with TD/GC/MS for the direct, solventless analysis of SVOCs and VOCs in household dust.

By encapsulating dust samples between pre-conditioned glass micro-fibre filters, the modified method allows for clean thermal desorption without entraining dust particles or fouling the instrumentation.

The optimized μ-CTE protocol offers several key benefits over conventional solvent extraction workflows:

- It eliminates the need for toxic organic solvents, aligning with green chemistry principles.
- It shortens sample preparation and processing times, enabling high-throughput screening.
- It prevents the loss of highly volatile target compounds during sample concentration.
- It measures the readily available, vaporizable pollutant fraction, providing a more relevant metric for indoor inhalation and exposure modeling.

Based on these findings, this method is recommended for large-scale indoor air quality screenings and environmental health monitoring programs.

Future work should focus on expanding this method to validate a wider range of emerging indoor contaminants, including organophosphate flame retardants (OPFRs) and novel synthetic plasticizers across diverse urban and rural environments.

### Conflicts of interest

The authors declare no conflicts of interest.

### Ethical Clearance

The samples analysed were completed coded during the study for privacy.

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## Author contributions

Fatima S. Mohammed, Phd: Conception of idea, research design, collection of dust samples, and supervision of laboratory practicals, supervision of sampling.

Sakina Bello Soye: coordination of sample collection and practicals, data collection

Muhammad Lawan Kamaluddeen,Phd: Literature review, design and data analyses.

Derrick Crump, Phd: Mentorship, supervision and review of practicals/write ups

Sakina Bello Soye: Coordination/sample collection in Nigeria homes, Laboratory analysis.

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