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## Performance of prototype high-flow inhalable dust sampler in a livestock production facility

T. Renée Anthony <sup>a</sup>, Changjie Cai<sup>a</sup>, John Mehaffy<sup>b</sup>, Darrah Sleeth<sup>c</sup>, and John Volckens<sup>b</sup>

<sup>a</sup>Department of Occupational and Environmental Health, University of Iowa, Iowa City, Iowa; <sup>b</sup>Department of Mechanical Engineering, Colorado State University, Fort Collins, Colorado; <sup>c</sup>Department of Family and Preventive Medicine, University of Utah, Salt Lake City, Utah

### ABSTRACT

A high-flow inhalable sampler, designed for operational flow rates up to 10 L/min using computer simulations and examined in wind tunnel experiments, was evaluated in the field. This prototype sampler was deployed in collocation with an IOM (the benchmark standard sampler) in a swine farrowing building to examine the sampling performance for assessing concentrations of inhalable particulate mass and endotoxin. Paired samplers were deployed for 24 hr on 19 days over a 3-month period. On each sampling day, the paired samplers were deployed at three fixed locations and data were analyzed to identify agreement and to examine systematic biases between concentrations measured by these samplers. Thirty-six paired gravimetric samples were analyzed; insignificant, unsubstantial differences between concentrations were identified between the two samplers ( $p = 0.16$ ; mean difference  $0.03 \text{ mg/m}^3$ ). Forty-four paired samples were available for endotoxin analysis, and a significant ( $p = 0.001$ ) difference in endotoxin concentration was identified: the prototype sampler, on average, had  $120 \text{ EU/m}^3$  more endotoxin than did the IOM samples. Since the same gravimetric samples were analyzed for endotoxin content, the endotoxin difference is likely attributable to differences in endotoxin extraction. The prototype's disposable thin-film polycarbonate capsule was included with the filter in the 1-hr extraction procedure while the internal plastic cassette of the IOM required a rinse procedure that is susceptible to dust losses. Endotoxin concentrations measured with standard plastic IOM inserts that follow this rinsing procedure may underestimate the true endotoxin exposure concentrations. The maximum concentrations in the study ( $1.55 \text{ mg/m}^3$  gravimetric,  $2328 \text{ EU/m}^3$  endotoxin) were lower than other agricultural or industrial environments. Future work should explore the performance of the prototype sampler in dustier environments, where concentrations approach particulates not otherwise specified (PNOS) limits of  $10 \text{ mg/m}^3$ , including using the prototype as a personal sampler.

### KEYWORDS

Aerosol; agricultural exposure; endotoxin; IOM; low cost

## Introduction

Inhalable dust is defined as any dust that can penetrate into the mouth or nose of a breathing human, which then is available to deposit anywhere in the respiratory system. When exposures to inhalable dusts are associated with health outcomes, regardless of where the dust deposits in the respiratory system, occupational assessments should be performed using samplers that meet the inhalability particulate mass (IPM) criterion, adopted by the American Conference of Governmental Industrial Hygienists (ACGIH), the European Committee for Standardization (CEN), and the International Standards Organization (ISO). The IPM criterion is defined as:

$$IPM = 0.5 (1 - e^{-0.06d_{ae}}),$$

where  $d_{ae}$  is the aerodynamic diameter ( $\mu\text{m}$ ) of the particle being sampled, up to  $100 \mu\text{m}$ .<sup>[1]</sup> While the IOM and the Button samplers are available in the U.S., additional inhalable samplers are available globally (Table 1).

The performance of personal aerosol samplers relative to this criterion has been assessed in numerous studies, but adoption and use of inhalable dust monitors have been slow in the U.S. Possible reasons include the perceived difficulty of having to handle the filter, the increased cost associated with inhalable samplers compared to the inexpensive 37-mm closed-face cassette (CFC), and a lack of regulatory pressure to monitor inhalable exposures (e.g., OSHA specifies the use of the CFC for “total” dust samples). To address these limitations, this team has developed a prototype inhalable sampler, designed to be

**CONTACT** T. Renée Anthony  [rene-anthony@uiowa.edu](mailto:rene-anthony@uiowa.edu)  Department of Occupational and Environmental Health, University of Iowa, 145 Riverside Drive, Iowa City, IA 52242.

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**Table 1.** Inhalable samplers.

Sampler	Manufacturer/Distributor	Flow Rate (L min <sup>-1</sup> )	Include Wall Deposits	Region of Use
IOM	SKC Inc.	2	Yes	Europe, U.S.
Button	SKC Inc.	4	No	U.S.
GSP (Gesamtstaub-Probenahmesystem)	GSMGesellschaft für Schadstoffmesstechnik, GmbH, Neuss-Norf, Germany	3.5	No	Germany
CIS (Conical Inhalable Sampler)	Casella CEL, UK	3.5	No	UK HSE, Germany
CIP10-I	Areloco ARC, France	10	No, version 2 reduces wall losses	France (wood dust)
PAS-6 (Personal Air Sampler)	University of Wageningen, Netherlands	2	No	Netherlands
PERSPEC	Lavoro e Ambiente, No longer commercially available	2	—	Italy
Multi-orifice ("seven-hole")	Casella CEL, UK	2	No	UK HSE
37-mm closed face cassette (CFC)*	e.g., SureSeal Cassette, SKC Inc.	1-2	No <sup>a</sup>	U.S. ("total" dust standards)
Prototype	(currently under evaluation)	10	Yes	—

<sup>a</sup>Note that the CFC was not designed to be an inhalable sampler, but are still commonly used in the U.S. to assess exposures relative to "total dust" limits. The NIOSH NMAM method does not specify wall losses be included, but Chapter O (*Factors Affecting Aerosol Sampling*) in NMAM<sup>[5]</sup> recommends that internal wall losses be included in the analysis.

inexpensive and disposable, simple to use, and compatible with low-velocity IPM sampling criterion. The initial design parameters were to maintain dimensions and operation similar to the widely used 37-mm CFC but to modify the inlet cap to improve the sampling efficiency for large particles.

Details of the new prototype sampler and its assembly are provided in Figure 1. The outer dimensions resemble a two-piece CFC, with a 15-mm inlet replacing the smaller 4-mm CFC inlet. In addition, the prototype sampler includes an internal capsule to collect wall deposits for inclusion in the mass concentration measurements (Figure 1d–f). This capsule protrudes beyond the face of the prototype sampler, providing a 5-mm lip surrounding the edge of the 15-mm opening into the sampler. The initial design parameters were explored in Anthony et al.<sup>[2]</sup> using computational fluid dynamic modeling: this initial work identified the dimensions and shape of an inlet cap for the prototype of the modified 37-mm CFC. Subsequently, prototypes of this design were built and tested in the lab<sup>[3]</sup> to demonstrate the sampler stability and performance relative to the IPM criterion. These initial lab studies were conducted using 2 L/min sampling rates, but additional collection efficiency studies at 10 L/min are also underway. To accommodate the trend of decreasing exposure limits, the prototype sampler was designed to meet the IPM criterion at 10 L/min sampling rates, which should be achievable with modern high-flow personal sampling pumps.

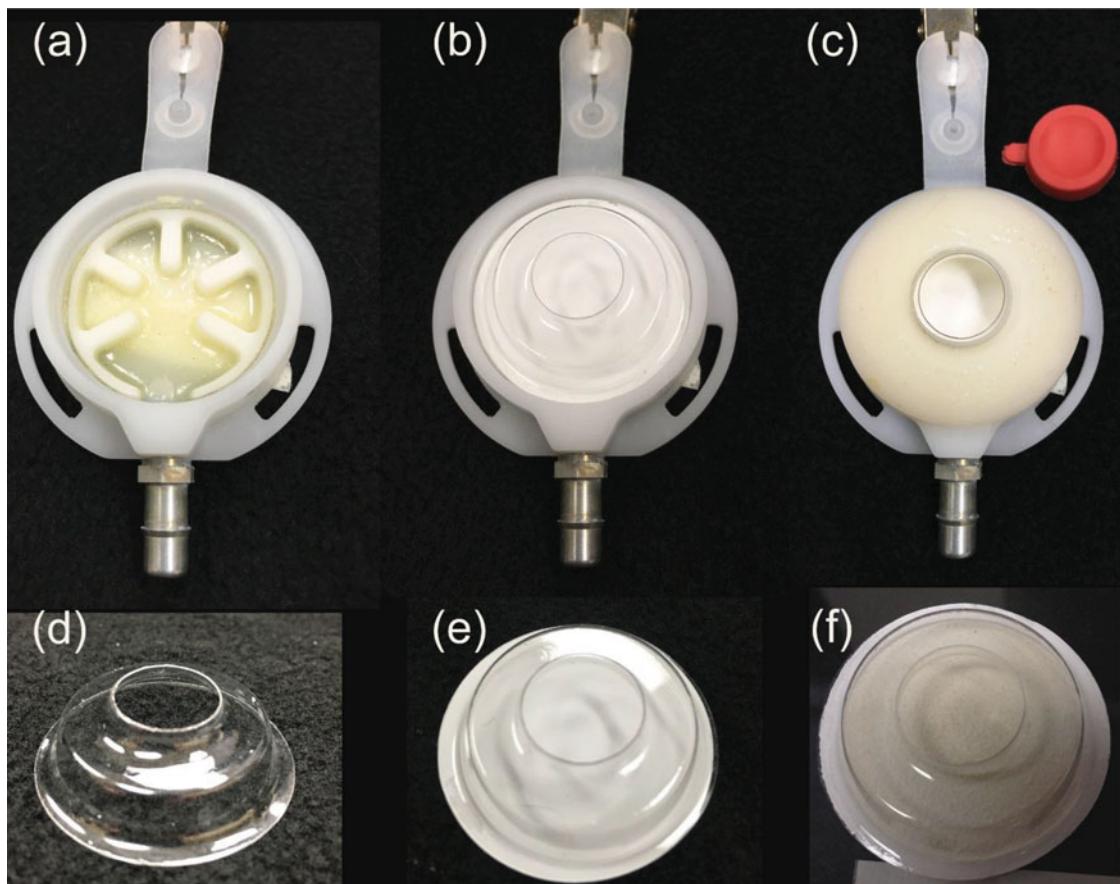
To evaluate the performance of the prototype sampler in the field, we collocated it with a standard IOM inhalable sampler, our benchmark reference sampler for this study, and collected samples of inhalable dust and inhalable endotoxin. To evaluate changes in particle size distributions that may occur during the field study, collocated respirable dust samplers were also deployed

to provide matched respirable mass concentrations throughout the study. All three samplers were deployed throughout a swine farrowing barn as part of a larger study examining ventilation improvements on indoor air quality.<sup>[4]</sup> This building had minimal air movement throughout the study period, which reflects low-velocity conditions typical of many workplaces. Concentration measurements, paired by sample location and date, were used to compare how well the new prototype sampler matched the benchmark IOM concentrations, while the collocated respirable concentrations allowed for an analysis of relative performance of the prototype sampler by a surrogate for changes in particle size. Difficulties in handling and operation of the prototype samplers in the three-month field study are discussed, along with recommendations to future users, particularly in regards to filter-capsule preparation, stability, and orientation of the high-flow sampler.

## Methods

### Test site description

This study was conducted in a swine farrowing room at the Kirkwood Community College Mansfield Swine Education Center in Cedar Rapids, IA. This is the same test area as described in Anthony et al.<sup>[2]</sup> although the data presented here were collected in a subsequent period. In short, the farrowing room measured 9.2 m by 14 m, had a capacity for 19-sows, and included 3 rows of 5 farrowing crates, each 1.5 m by 2.4 m, and one row of four 2 m by 2.4 m crates. Dust monitoring occurred on 19 days between December 2014–February 2015. Typical of Midwest swine production, air movement was minimal, where the vents that bring fresh air into the building during warmer months remained closed during



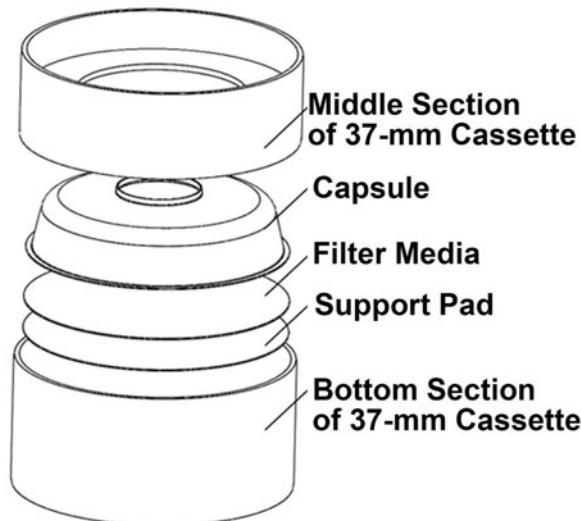
**Figure 1.** Prototype sampler (a) housing and interior, (b) housing with cellulose backup and bonded filter-capsule, (c) assembled sampler with inlet (used IOM inlet cover, to the side), with (d) unbonded capsule, (e) capsule bonded to filter, and (f) filter + capsule post-sampling.

the test period. Air was exhausted from the under-floor manure pit beginning on January 15, 2015, after which they remained on through the remaining study period. Makeup air into the test room entered primarily through two pressure louvres along the east wall (42-in long and opened 2–5 cm) and doors on this same wall, bringing in air from the heated hallway.

The dust sampling was part of a larger study examining the effectiveness of an installed recirculating ventilation system (1699 m<sup>3</sup>/h; 1000 cfm, with cyclonic dust control technology [Donaldson Inc., Model 16]). On 12 sample days, room air was exhausted at 2 locations, sufficiently far from the sampling stations, treated with an industrial cyclone, then returned to the room via fabric diffusion ducts along the ceiling. The operation of this system induced no discernable air movement near the fixed sampling stations. On seven sample days, the system was off. Investigating sampler performance over both room ventilation conditions was anticipated to allow the assessment of both high (system off) and low (system on) dust and endotoxin concentrations throughout the three-month study period (results from the ventilation/controls study will be reported separately).

#### Prototype sampler assembly and handling

The prototype sampler was designed to have an internal capsule attached to the filter media to allow for easy quantification of the dust concentration entering the sampler, similar to the Accu-CAP (SKC 225-8516GLA) capsule insert for the 37-mm cassette.<sup>[5,6]</sup> The design of the sampler is described in detail in L'Orange et al.,<sup>[3]</sup> but specific details regarding the handling in the field study are provided here. Figure 2 illustrates the procedure used to bond the capsule to the filter. First, a stack of five standard cellulose support pads were placed in a standard 37-mm cassette. Then, the entire perimeter of the capsule's base was wetted with toluene using a cotton swab. The wetted capsule was then placed onto a 37-mm air sampling filter, with careful attention paid towards aligning the edges concentrically. The capsule-filter unit was then placed onto the support pads and a middle ring of a standard 37-mm cassette was placed on top. Pressure was applied to the outside of the 37-mm cassettes to ensure contact and bonding of the capsule to the filter. After 20 sec, the cassette was disassembled and the bonded filter/capsule were placed in petri dishes in a fume hood (2–12 hr) to allow evaporation of residual toluene. These



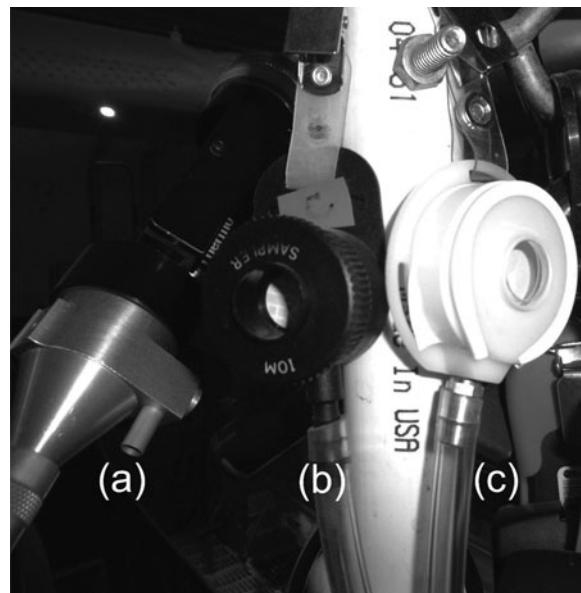
**Figure 2.** Bonding the 37-mm filter to the prototype filter capsule.

filters/capsules were located to an environmentally stable room for at least seven days, after which the pre-sampling weight was measured.

PVC filters, matching the NIOSH 0500 analytical method, were identified as incompatible with this bonding procedure, as the toluene changed the surface characteristics of the PVC filter. Since the analyses of the samples for this study required gravimetric analyses, weight-stable PTFE media was used (2  $\mu\text{m}$  PTFE filter with PMP ring, SKC 225-1709). One problem with the filters selected for this study was that the PMP ring randomly detached from the bonded unit post-sampling, which required handling of both the bonded filter-capsule and a secondary ring when conducting post-sampling weighing on samples where the ring did not stay attached to the unit upon removal from the new prototype sampler.

### **Sampling and analysis**

Dust samples were collected over 24-hr periods at three fixed locations in the test room. Inlets to the monitors were positioned at breathing zone height (1.5 m), collocated as close to one another as practical (Figure 3). Respirable dust was collected onto 5  $\mu\text{m}$  PVC filters using cyclones (BGI GK2.69) positioned on direct reading equipment (pDR-1200, Thermo-Electron Corp.), with sampling pumps (PCXR4, SKC, Inc.) pulling air through the cyclone-pDR at 4.2 L/min. Inhalable dust was collected at matched locations using both the IOM (5  $\mu\text{m}$  PVC filters, 2 L/min sampling rate using PCXR4 pumps, SKC, Inc.) and the prototype low-cost inhalable dust sampler (2  $\mu\text{m}$  PTFE filter with PMP ring, using a combination of Leland Legacy and BGI 400 pumps to achieve the high flow rate). The study attempted to obtain 57 collocated respirable-IOM inhalable-prototype inhalable samples.



**Figure 3.** Collocated samplers as positioned in the field, (a) inlet to respirable cyclone (BGI GK2.69), (b) IOM inhalable sampler, and (c) prototype inhalable sampler.

Assembled filter media were stored in an environmentally controlled laboratory for seven days prior to both pre- and post-sampling weighing.<sup>[7]</sup> All sample media (respirable filters, IOM filters + internal cassette, and prototype sampler filters + capsules) were weighed in triplicate (MT5, Mettler-Toledo, Columbus, OH) before and after sampling, with weight gains computed using the mean of pre- and post-sampling weights, adjusted by any weight change in field blanks collected on matched sampling dates.

After gravimetric analyses, the IOM and prototype samples were stored at  $-20^{\circ}\text{C}$  for accumulation prior to endotoxin analysis. Because respirable dust samples were collected after passing through the direct reading instrumentation, endotoxin contamination between sampling events was a concern for the respirable samples, which were therefore not analyzed. Each prototype filter-capsule sample was inserted into 50 mL centrifuge tubes (Falcon Tubes, Corning Inc., Corning, NY) for endotoxin analysis prior to shipping for analysis. Each IOM sample remained sealed in the transport cases for shipping.

Before sample extraction, prototype sampler capsules were pushed to the bottom of Falcon<sup>TM</sup> tubes with a sterile spatula in order to assure submergence in extraction fluid. IOM filters were removed from the transfer clips, separated from the internal plastic cassette, and placed in 50 mL Falcon tubes. The IOM internal cassettes were rinsed with 1 mL extraction solution, repeated twice, with this solution added to the tube containing the filter. A 0.05% tween solution with Tween 20 (Amresco, Solon, OH) and LAL reagent water (Lonza, Walkersville, MD) was used to perform extractions with a 20-mL volume for

prototype samples and 10-mL volume for IOMs. Samples were vortexed and shaken for 1 hr with appropriate dilutions prepared following the extraction procedure. Sample dilutions were loaded into 96-well plates along with endotoxin standards, blank extraction solution, and control spikes in triplicate, 100  $\mu$ L each.

Endotoxin analysis was performed with a Pyrogenic Recombinant Factor C assay (Lonza Group, Walkersville, MD) and Bioteck FLx800 fluorescence microplate reader with Gen5 software (Bioteck, Winooski, VT). The Pyrogenic assay operates on the principle that endotoxin activates Recombinant Factor C enzyme which then cleaves a fluorogenic substrate and fluorescence intensity is measured by a microplate reader. Analysis was performed using an endpoint method with a one hour incubation period at 37°C. Time zero readings were subtracted from post incubation readings and standard curves from *E. coli* 055:B5 standards (Lonza, lot# 0000419301) were used to calculate Endotoxin Units (EU)/mL from raw fluorescence units (RFUs) where the log net fluorescence is proportional to the log endotoxin concentration. Readings were taken with excitation/emission wavelengths of 380/440 nm.

### **Data analysis**

Both mass and endotoxin concentrations were computed for each paired sampler. The normality of concentration data, by sampler and analysis, were assessed, using raw and natural-log transformed concentration measures. Student's t-tests were conducted to analyze the agreement between sampler concentrations. For non-normally distributed data sets, non-parametric analyses (Wilcoxon two-sample tests) were also used, examining Spearman correlation coefficients. Bland-Altman plots<sup>[8]</sup> were constructed to evaluate qualitative biases between collocated measurements (IOM and prototype). Simple linear regression, using both fitted and zero intercepts, was performed to examine the strength of the relationship between the concentrations measured by these samplers. Residuals, computed as the modeled minus measured concentration, were also examined to assess bias and identify the most appropriate form of the relationship between the concentrations obtained by these two samplers. Analyses used SAS 9.4 (SAS Institute Inc., Cary, NC).

## **Results**

### **Qualitative feedback on prototype sampler**

The objective of this work was to evaluate the performance of the prototype sampler operated at 10 L/min by

comparing paired concentrations from collocated IOM samplers, operated at 2 L/min. While high-flow pumps are available from pump manufacturers, it was difficult to achieve the desired 10 L/min flow rate through the prototype sampler with 2  $\mu$ m pore size PTFE, which was identified early in the study. Neither the Leland nor the BGI pumps could maintain 10 L/min for sufficient calibration, even when fully charged or while plugged in. However, these pumps were able to maintain flow at a nominal 8 L/min flowrate (mean = 8.2 L/min, sd = 0.58) throughout the study, with pumps connected to power for the entire 24-hr sampling period. These initial field studies attempted to use the traditional cellulose backup pad behind the filter-capsule sample media, and subsequent field testing and redesign of the filter platform inside the prototype sampler has been redesigned to eliminate the need for the backup pad.<sup>[9]</sup> Subsequent to this field study, collaborators have been able to achieve 10 L/min flow rates through 5  $\mu$ m MCE filters with the removal of a backup pad. Minimal flow changes pre- and post-sampling were identified at this lower flow rate, and no sample was eliminated due to low post-calibration flow rate.

The prototype sampler was manufactured to have a press-fit seal to eliminate the need for threads or gaskets. While these samplers were designed to be disposable, we used four of the prototype exterior sampler housings throughout the 19 sample days, rotating cassettes used at the three fixed positions and the field blank throughout the study. In some cases, the bonding of the internal capsule to the PTFE filter resulted in wavy surfaces around the bonded capsule edge, which prevented the tight fit of the prototype sampler. Throughout the study, electrical tape was positioned around the edge of the seam between the inlet cover and the housing, while ensuring the tape on the face of the inlet cover remained smooth, as shown in Figure 3. No visible indication that the sampler leaked around the edge of the prototype filter-capsule unit was evident post-sampling (e.g., see Figure 3c).

### **Agreement between inhalable sampler concentrations**

Over the three-month study period, 24-hr integrated samples were collected on 19 days at three positions in the farrowing room. Five pump failures occurred (1 IOM, 1 respirable, 3 prototype), resulting in sample durations < 812 min; these samples were excluded from paired analyses. The mean sample duration for the remaining samples was 1474 min (24.5 hr). In addition, nine of the initial prototype gravimetric analyses were discarded due to problems associated with the separated ring on the PTFE filters: while the gravimetric analyses were voided, the

**Table 2.** Concentration comparisons pairing collocated data.

	Respirable Dust <sup>c</sup>	Inhalable Dust		Inhalable Endotoxin	
		IOM <sup>c</sup>	Prototype	IOM	Prototype
<i>All Concentration Measures:</i>					
N	56	53	40	54	47
Mean*	0.10	0.70	0.66	721	850
SD*	0.03	0.32	0.29	404	458
<i>Concentration Measures with Paired Data Only:</i>					
N-pairs	—	36	—	44	—
Mean <sup>a</sup>	—	0.65	0.68	720	839
SD <sup>a</sup>	—	0.31	0.28	412	427
Geometric Mean <sup>a</sup>	0.09	0.58	0.62	594	729
Geometric Standard Deviation	1.41	1.67	1.50	1.97	1.78
1-Tail, paired t-test, p	<0.001 <sup>b</sup>	—	0.16	—	0.001
Wilcoxon, one-sided, p	<0.001 <sup>b</sup>	—	0.23	—	0.079
Spearman Correlation Coefficient, estimate (95% CI)	—	0.87 (0.76–0.93)	—	0.87 (0.77–0.93)	—

<sup>a</sup>Dust concentrations in mg/m<sup>3</sup>, endotoxin concentrations in EU/m<sup>3</sup>.

<sup>b</sup>Compares respirable to IOM gravimetric concentrations.

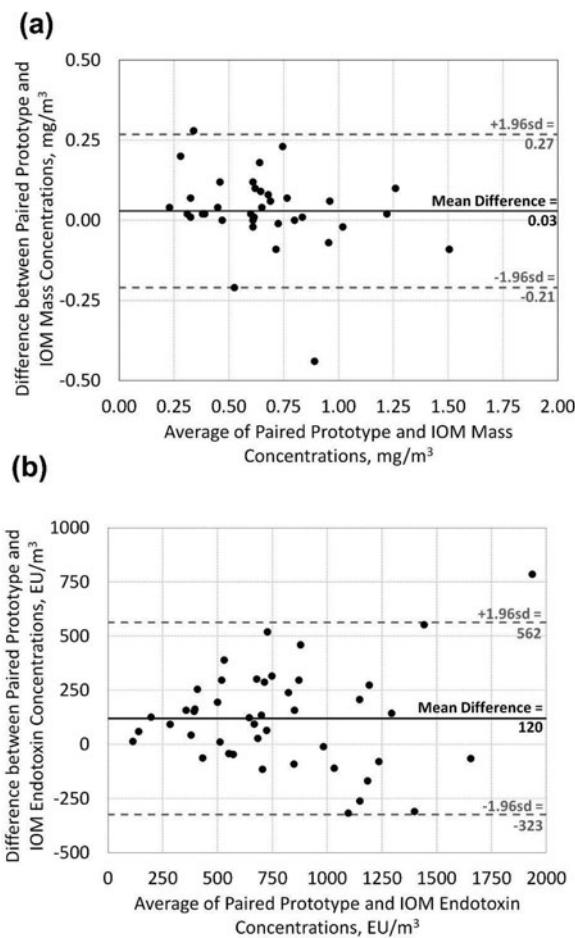
<sup>c</sup>Normally distributed (Shapiro-Wilk, [Pr < W] > 0.06).

samples were analyzed for endotoxin, as the detached ring was on the non-exposed surface of the filter-capsule. Following the third sampling day, procedures were developed to include the ring with the sample's post-weight, if separated from the bonded filter-cassette. On day 5, field blanks for the prototype samplers had significant blank weight loss, exceeding the field samples weight gains, which necessitated elimination of these gravimetric data. Finally, one of the scheduled sample events (day 6) had insufficient prototype filters available for deployment. The total number of samples collected, by sampler type and subsequent analyses, are indicated in **Table 2**, along with descriptive statistics of the dust and endotoxin concentrations.

Between-sampler comparisons of gravimetric and endotoxin concentrations are given in **Table 2**, as measured by these three samplers. The inhalable dust concentrations were approximately five times that of the respirable samplers ( $p < 0.001$ , paired t-test), indicating that much of the aerosol contained particles larger than the 10  $\mu\text{m}$  upper limit of the respirable cyclone. Furthermore, the mean mass concentration of inhalable dusts was 0.03 mg/m<sup>3</sup> higher with the prototype sampler compared to the IOM sampler when comparing paired data ( $N = 36$ ), an unsubstantial and insignificant difference ( $p = 0.16$ ). Correlation between the mass concentrations of these inhalable samplers was high (Spearman correlation coefficient = 0.85). The between-sampler difference by average paired sampler concentration are shown in **Figure 4a**, which illustrates the mean difference of the 0.03 mg/m<sup>3</sup>. Limited bias between samplers was observed over the range of concentrations measured in this agricultural building. The 95% limits of agreement bands identified that the majority of the mass concentrations in the prototype sampler ranged from 0.21 mg/m<sup>3</sup> above to

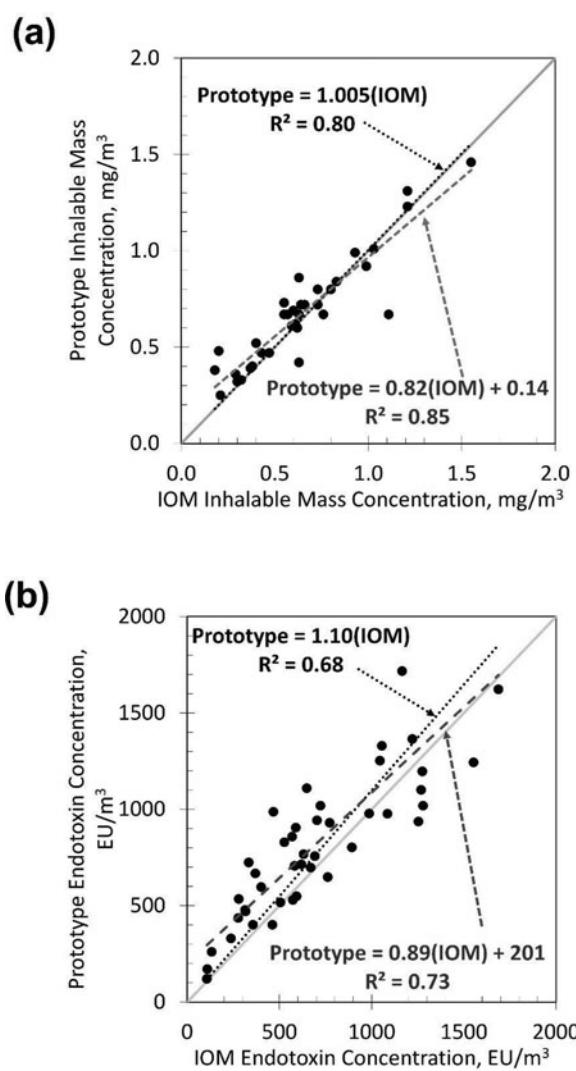
0.27 mg/m<sup>3</sup> below that of the IOM. Two pairs of gravimetric samples were outside this band, where in one case the prototype sampler exceeded twice the IOM concentration (0.48 vs. 0.2 mg/m<sup>3</sup>) and in the other case, the reverse was true (0.67 prototype vs. 1.11 mg/m<sup>3</sup> IOM). A more typical comparison of inhalable mass concentrations of the paired samples is provided in **Figure 5a**, illustrating both linear regressions with and without fitted intercepts. Increased mass sampled beyond the high end of this data set (1.5 mg/m<sup>3</sup>) is needed to ensure that the prototype does not under-sample the inhalable dust relative to the IOM. However, since the mean difference between sampler was low (0.03 mg/m<sup>3</sup>) and insignificant (t-test  $p = 0.16$ ), examination of a forced (0,0) intercept was made. This method identified <1% difference between inhalable mass concentrations between the prototype sampler and the IOM (slope = 1.005), with a substantial portion of the variance in the prototype concentration still attributable to that of the IOM concentration ( $R^2 = 0.80$ ). Residuals in both linear models identified random pattern over concentrations measured, with no clear trend. The mean residual for the fitted intercept ( $-0.006 \text{ mg/m}^3$ ) was slightly improved over the model with the zero intercept ( $-0.026 \text{ mg/m}^3$ ) for the mass concentration measurements.

While minor and insignificant differences were identified in mass concentrations between inhalable samplers, endotoxin analyzed from the two samplers differed. The prototype sampler averaged higher endotoxin concentrations over all paired samples ( $N = 44$ ), with 70% of all paired data having higher measures on the prototype sampler. Correlation between the endotoxin concentrations of these inhalable samplers was reasonable but not as strong as with mass concentration (Pearson correlation coefficient = 0.87). Using the Bland-Altman



**Figure 4.** Bland-Altman plots to show agreement between (a) mass concentration (gravimetric) and (b) endotoxin concentration, using paired data, using differences computed from (Prototype – IOM). The estimated bias is shown as the mean difference, and the 95% limits of agreement bands ( $\pm 1.96$  sd).

plot in Figure 4b, the mean difference between samples was 120 EU/m<sup>3</sup>, indicating the systematic bias for higher endotoxin in the prototype sampler. The 95% limits of agreement band identified the majority of prototype samplers differed from 562 to –323 EU/m<sup>3</sup> compared to the IOM sampler, with one outlier for which the prototype sampler collected 750 EU/m<sup>3</sup> more than the IOM. Linear regression (Figure 5b) confirmed this same trend, with a fitted intercept at 201 EU/m<sup>3</sup>. Residuals analysis confirmed an improved fit using the fitted intercept (mean residual –7.35 EU/m<sup>3</sup>) compared to the zero intercept (mean residual –58.26 EU/m<sup>3</sup>), confirming the model with the fitted intercept of 201 EU/m<sup>3</sup> is preferential, again confirming the substantial difference in endotoxin concentration between samplers. At concentrations above the range identified in this study, it is again unclear if the concentrations of the IOM would yield more than that of the prototype, particularly above approximately 1830 EU/m<sup>3</sup>, e.g., where [Prototype] = [IOM] in the regression equation. Residuals analysis identified this concern, but



**Figure 5.** Prototype versus IOM (a) inhalable mass and (b) endotoxin concentrations. Solid line indicates perfect agreement. Dashed lines indicate best fit (linear regression), using zero as intercept (rounded dash) and fitted intercept (straight dash).

with only one prototype endotoxin concentration above 1830 EU/m<sup>3</sup>, additional evaluation is needed to confirm whether the performance changes at high concentrations.

## Discussion

### Sampling method

In the field, the prototype sampler was as easy to use as the widely available IOM sampler, with less parts needed to ensure proper assembly. Preparation of the filter-capsule in the prototype sampler required the use of toluene in a laboratory fume hood for bonding, which then required waiting for seven days for the weights to stabilize prior to use in the field. This may require additional time and bench space for preparation in contract laboratories, which may add to the cost of sample preparation and

analysis, relative to the IOM. In the field, handling requirements for the prototype sampler was similar to the IOM: use of a transport cap was critical to protect the internal cassette from hands during handling, as the internal cassette protrudes through the sampler housing in both samplers, and touching that surface could contaminate the samples.

More critical was our difficulty achieving the target flow rate of 10 L/min through the prototype sampler using 2  $\mu\text{m}$  pore-size PTFE filters. The high-flow personal samplers were able to consistently provide 8.2 L/min on average, but operation at this level was noisy. Previous simulation work<sup>[2]</sup> identified minimal sampling efficiency differences over flows ranging between 8 and 10 L/min for the design similar to this sampler ("Central-5mm" simulated design), but this difference was not testable with the pressure drop and sampling pumps available.

Recommendations on handling endotoxin samples were also identified from this early field study. The bonded filter + capsule was placed in 50 mL Falcon tubes after post-sampling weighing and were stored in the freezer for accumulation. Due to the size of the capsule relative to the tube opening, bending of the capsule for insertion was required. This often resulted in detachment or tearing of the filter, which ultimately was desirable for endotoxin analysis as it facilitated adequate mixing during extraction procedures. However, care must be taken to not lose material during the capsule insertion. When inserting the prototype's filter + capsule into the Falcon tubes, we recommend pushing the capsule into the very bottom of the tube, as adjusting placement of the capsule at a later date introduces an additional potential for contamination or losses. Placement of the capsule at the bottom of the tube also ensures full submersion within the extraction media.

### Sampler performance

Limited aerosol sampling methods are available to identify the size distribution of field aerosols that include a substantial number of larger, inhalable particles, which makes identification of the "true" inhalable dust concentration in the field difficult. Hence, the IOM was used as a benchmark reference concentration measurement in this study. However, laboratory and field studies have identified low reproducibility between paired samples with the IOM, which introduces uncertainty in our reference concentration measures. For example, in controlled laboratory experiments, Aizenberg et al.<sup>[10]</sup> identified that the IOM sampler data provided the closest sampling efficiency relative to the inhalability criterion, but mass concentrations from the IOM was less reproducible than the Button sampler at test velocities of 0.55 and 2.0  $\text{m s}^{-1}$ . The

Button sampler, on the other hand, has very low sampling efficiency when measuring droplet aerosol,<sup>[11]</sup> which may affect the performance of the Button sampler in wet or highly humid conditions.

Agreement between the sampled mass concentrations between the IOM and the prototype sampler evaluated here was similar to comparison studies of other inhalable samplers on the market. Early in the study of inhalable samplers, Vaughn et al.<sup>[12]</sup> compared the performance of area samples of inhalable dust in nine industries and identified that the IOM gave 21%, on average, higher mass concentrations compared to other available inhalable samplers, including both seven-hole samplers (Casella and J.S. versions). Zugasti et al.<sup>[13]</sup> identified that the Button sampled only 90% the Gesamtstaub-Probenahmesystem (GSP) sampler and 92% of the welding mass concentration measured by an IOM in field studies.

There appears to be limited proportional bias in the gravimetric mass concentrations in this study, but note that the inhalable concentration did not exceed 1.55  $\text{mg/m}^3$  over the study period. Hence, additional analyses would be needed to compare the performance between samplers at higher concentrations to confirm this across a larger range of exposures in which inhalable sampling might reasonably occur.

Since the same physical samples were analyzed first gravimetrically then were processed for endotoxin, the different performance in endotoxin concentration without a similar difference in gravimetric analysis indicates there may be systematic differences in the samplers due to the analytical technique and not the sampling efficiency. The endotoxin extraction methods differed between the two samplers due to differences in the structure of the internal capsule, thereby necessitating different processing for endotoxin analysis. The prototype capsule was designed to be flexible and disposable and was manufactured with a thin-film of polycarbonate using thermal vacuum-forming. This structure made it easy to bend and add it directly into the 30-mm diameter centrifuge tubes, along with the bonded filter. The internal cassette of the IOM is a rigid, reusable structure that is typically rinsed and not soaked in endotoxin extraction solution. In this study, the filter + capsule of the prototype sampler was soaked, together, in 20 mL of Tween solution, whereas the internal cassette of the IOM was rinsed with 1 mL of the Tween solution, twice, prior to the 1-hour vortex/shaking process. These differences may account for these systematically higher endotoxin concentrations reported for the prototype sampler.

Room concentrations throughout this study were well below the 10  $\text{mg/m}^3$  inhalable particulates not otherwise specified (PNOS) by ACGIH,<sup>[1]</sup> which limited



our ability to evaluate any bias in the new sampler across the full range of possible exposures. The range of inhalable dust concentrations was 30% lower than what was found in the previous winter season.<sup>[4]</sup> In addition, the inhalable mass concentrations measured in this study (1.55 mg/m<sup>3</sup> maximum, 0.65 mg/m<sup>3</sup> arithmetic mean, 0.58 mg/m<sup>3</sup> geometric mean, 1.35 mg/m<sup>3</sup> 95<sup>th</sup> percentile, from the IOM) were below concentrations reported in recent field studies of Danish pig workers, where the geometric mean (GM) inhalable dust concentration was 4.0 mg/m<sup>3</sup> (GSD = 2.1).<sup>[14]</sup> The endotoxin concentrations in our US study ranged from 120–2328 EU/m<sup>3</sup>, with GM = 600 EU/m<sup>3</sup> (IOM samples); this was below the concentrations in the 2013 Danish study, where personal endotoxin exposures had GM = 1800 EU/m<sup>3</sup> and ranged up to 380,000 EU/m<sup>3</sup>.<sup>[14]</sup> Hence, additional evaluation of the sampler performance would be needed to fully evaluate the range of possible exposures in pig production and in high-exposures present in other industries. Testing the performance of the prototype sampler in these higher concentrations would provide additional insights into whether the between sampler endotoxin bias increases with increased sampled mass.

## Conclusions

This project demonstrated the general agreement in sampler performance between a new prototype inhalable dust sampler, designed to integrate the field handling advantages of the commonly used 37-mm CFC with the sampling and sample recovery performance of the widely available IOM. While high-flow personal sampling pumps that were currently available were not able to maintain the desired 10 L/min flow rate with the 2 μm pore-size PTFE filter, operation at 8.2 L/min provided an increased limit of detection for the mass concentration of the prototype sampler relative to the traditional 2 L/min IOM sampler. This initial field-based sample comparability study relied on side-by-side comparisons of fixed samplers (area measurements) rather than placing the samplers in the breathing zone of workers in order to eliminate specific sources of variability between samplers. However, the reasonable performance of the prototype sampler demonstrated in this field study warrant deploying the prototype as a personal sampler. Future work should deploy the prototype sampler alongside a benchmark inhalable sampler (e.g., the IOM) and should include monitoring exposures that approach the PNOS exposure limit to verify the performance and possible bias associated with this new sampler, particularly at higher concentrations to which workers might be exposed.

## Disclaimer

The authors declare no conflict of interest relating to the material presented in this article. Its contents, including any opinions and/or conclusions expressed, are solely those of the authors.

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## ORCID

T. Renée Anthony <http://orcid.org/0000-0002-3780-7436>

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