IJOH INTERNATIONAL JOURNAL OF OCCUPATIONAL HYGIENE
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2008-5435/14/63-1-8

ORIGINAL ARTICLE

Pressure Drop of Respirable Dust Cyclone Samplers

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Received June 17, 2019; Revised July 19, 2019; Accepted August 05, 2019

This paper is available on-line at http://ijoh.tums.ac.ir

ABSTRACT

The measure of volumetric flow rate is utilized to calculate sampling volume and airborne concentration in integrated air sampling. Consequently, calibration of the sampling train to verify volumetric flow rate is critical. The relation between sampler pressure drop and volumetric flow rate was studied in support of using the former rather than the latter for the calibration of sampling trains. Four types of respirable cyclones, two filter brands with membrane samples of the same and different lots of production, and two personal pump types were considered as components of the sampling trains under consideration. Volumetric flow rate and pressure drop were measured under controlled conditions in a cylindrical jar designed for these determinations. For all the configurations considered, the relation between sampler pressure drop and standard volumetric flow rate was linear. Intra-sample selection of cyclones of the same type and pump type did not create significant differences in sampler pressure drop. Filter selection, regardless of brand or production lot, did create linear response functions that had statistically different slopes and intercepts. When grouped by cyclone type and filter brand, the sampler pressure drop at the flow rate recommended by the cyclone's manufacturers showed variability that was not normally distributed. The recommended central tendency estimate of pressure drop is the median value, with point estimates that should be specific to a cyclone type / filter brand combination.

KEYWORDS: *Pressure Drop, Respirable Dust, Aerosol Sampling, Calibration*

INTRODUCTION

The knowledge of volumetric flow rate is fundamental for assessing sampling volume and airborne concentration in integrated sampling. In addition, size-selective sampling relies on sampling at very specific flow rate values that are established to allow size cut-points in conformity with size distribution functions defined to address a selective toxicity [1-3]. Many of the samplers currently in the

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market used for collecting samples of inhalable, thoracic, respirable and PM10 or PM2.5 fractions are thoracic, respirable and PM10 or PM2.5 fractions are typically flow rate-calibrated by using, either adapters, or the "old" jar option. These samplers may be good candidates for pressure drop calibration if a "correlated and tight" relation between volumetric flow rate and pressure drop could be established.

The relationship between air sampler type and flow rate-related pressure-drop has been previously considered for setting volumetric flow rates in air sampling trains. Noteworthy is the example of the "jarless" calibration method, initially developed by NIOSH [4] and currently recommended by OSHA [5] as a method of choice for flow rate adjustment in sampling trains with nylon cyclones and cassettes containing PVC, 5-µm pore size, membrane filters.

The jarless method relies on mechanical means to create a pressure drop equivalent to that of a clean sampler and consecutively sets the flow rate at a stated value in the air sampling pump. A second test verifies that under a pressure drop equivalent to that of a fully-loaded sampler, the initial flow rate remains relatively unchanged (less than 5 % of change). The jarless, or pressure-drop method, has multiple advantages. It removes a need for calibration adapters, it does not require a reserved additional sampler for calibration, and most importantly, it eliminates potential sources of error that result from a poor or inconsistent seal of calibration adapters or calibration jar lids [6].

The jarless calibration paradigm is an attractive approach due to the above-mentioned advantages and the simplicity of its protocol. However, in order to use pressure-drop as a reliable indicator of flow rate, the relation between the former and the latter, as they apply to specific sampler types, must be known in advance. Sampler pressure drop, or total pressure loss with contributions of dynamic and static pressure, is a function of air velocity, air acceleration and deceleration within the sampler, the degree of tortuosity of the air trajectory (created by particle size separation needs), and filtration pressure losses created by the attached sample-collecting membrane or substrate. Therefore, sampler pressure drop can be expected to be air velocity sampler design (inter- and intra-model) and filter type-dependent. Air pump selection may also be influential since the second test step of the calibration protocol requires a "limited" change in volumetric flow rate after the pump is challenged by an increased pressure drop. It is expected that pumps with diverse designs may handle differently the task of maintaining relatively unchanged flow rates as the pressure drop rises.

This research was involved two phases and was limited in scope to respirable dust cyclones and PVC membrane filters. The first phase was considered an overall, multi-variable evaluation of the relation between flow rate and pressure drop. Pressure drop at different volumetric flow rates was measured under multiple combinations of sampler type (including inter- and intra-model samples), filter brand (two manufactures, samples of three separate production lots each), and pump type. Segregated and grouped data were used to find central tendency and dispersion estimators of pressure drop at each of the flow rates specified by the sampler's manufacturers. In the second phase, a critical review of the NIOSH jarless method was focused and addressed as follow: (1) the rationale behind end point test decisions, (2) apparent protocolary gaps and, (3) the scrutiny of test accuracy in contrast to flow rate measurements in a well-controlled, jar protocol. It is expected that the second phase results will be submitted in a separate, later publication.

The contributions anticipated from this two-phase effort was to generate data that facilitates the choice of pressure drop over volumetric flow rate measurements in the calibration of specific air sampling trains. It also attempts to raise consciousness around a need for published performance data on pressure drop of air sampling equipment, so that the choices for volumetric flow rate calibration, either directly or indirectly, can be expanded.

MATERIALS AND METHODS

COMMERCIAL SAMPLER AND FILTER MEDIA SELECTION:

The sampling train configurations matrix considered in this study have been presented in Table 1. These configurations included: (1) four different types of respirable dust cyclones commonly used for air sampling in industrial hygiene, (2) two different commercial brands of 37-mm, 5-µm pore size, polyvinyl chloride (PVC) filters with samples from three separate prduction lots each, and (3) two different air sampling pump models.

EXPERIMENTAL SETUP:

The schematic diagram of experimental setup has been shown in Figure 1. An air-tight leak-proof calibration jar was built for this study to measure standard volumetric flow rates (SVFR, actual volumetric flow rate corrected to standard conditions of temperature and pressure) and related pressure drops for each cyclone-filter combination. The calibration jar, a cylinder made of acrylic, had the following dimensions: 30.5 cm of length, 20 cm of outer diameter, and 0.64 cm of wall thickness. The jar was equipped with a 25 cm x 25 cm fixed base plate and a 25 cm diameter, 1.3 cm thick removable top cover that was secured to the top flange with a built in O-ring, via bolts. Two 0.64 cm diameter threaded ports placed centrally in the top cover, 5 cm apart, were welded to 0.953 cm diameter stainless-steel tubing using standard pipe fittings. The stainless-steel ports allowed airflow into and out of the jar and allowed a means for connecting the mass flowmeter and manometer (manometer was actually connected to secondary metal ports welded in a T configuration to the main vertical ports). The secondary ports, used for pressure measurements, had a diameter of 0.953 cm and were welded at a minimum of three diameters from the air intake in the main port to allow proper pressure readings. All welds were inspected to verify no flashings were introduced that would impede airflow. The diameters of primary and secondary ports were selected to accommodate tight connection of standard Tygon® tubing regularly used in air sampling components.

PRELIMINARY TESTS:

Two preliminary tests were performed to verify jar containment and jar pressure drop. The jar containment test was assessed to confirm no air leakage into the vessel. Two tests were initially considered for containment confirmation: sustained vacuum and volumetric flow rate balance. Sustained vacuum was disregarded because of a large dead space in the jar and the stress that negative pressure could pose onto the jar's structure. For volumetric flow rate balance testing, a nominal flow rate was selected using an AirChek TOUCH sampling pump (SKC Inc., Eighty Four, PA, USA). Two Defender 510 DryCal calibrators (Mesa Labs Inc., Butler, NJ, USA) were used to measure flow rate at the entry and exit sampling ports simultaneously. Additionally, pressure was measured using a digital manometer (Model 475-2-FM, Dwyer Instruments, Michigan City, IN, USA) at the exit port. No sampler was attached during these preliminary tests.

After correcting the exit flow rate by pressure (atmospheric pressure minus exit port pressure), and comparing it to the entry flow rate, it was confirmed that the percent difference in flow rate measured was less than the reported instrumental error.

Jar pressure drop is the pressure drop across the designed calibration jar measured as differential pressure across the two secondary ports with no sampler, filter cassette, or Tygon® tubing attached (except tubing needed to connect measuring instruments). Jar pressure drop was measured using a digital manometer (Model 475-00-FM or 475-2-FM, Dwyer Instruments, Michigan City, IN, dependent on pressure drop range) for flow rates in the range of 1.0 L/min to 3.5 L/min. Flow rates were selected using an AirChek TOUCH pump and increased in 100-300 mL/min increments ensuring that critical flow rates for selected cyclone assemblies were included. SVFRs were expressed at 760 mm of Hg (101.3 kPa) and 21.1 C were measured by a mass flowmeter (Model 4140 TSI Inc. Shoreview, MN, USA). Jar pressure drop was also assessed using an SKC Universal PCXR4 sampling pump (SKC Inc., Eight Four, PA, USA). As expected, the jar pressure drop dependence on SVFR across the design vessel was linear.

The variability in sampler pressure drop (cyclone and filter) at different volumetric flow rates was measured under multiple combinations of sampler type, commercial filter brand including manufacturing lot, and pump type. The total pressure drop was measured as the differential pressure across the secondary ports, with the cyclone and filter cassette connected via tubing to the inner end of the jar exit port. The jar pressure (interpolated from the linear regression function obtained previously) was subtracted from the total pressure drop at a given volumetric flow rate to ascertain the sampler pressure drop.

SEQUENCE OF TRIALS:

The multi-variate experimental design considered the following sequence of tests:

Test 1 studied the variability (as described in the previous paragraph) due to intra-unit selection of 10-mm Dorr-Oliver nylon cyclones (Zefon International, St. Petersburg, FL). It measured the total pressure drop for flow rates in the range of 1.0 L/min to 3.5 L/min for three different cyclone units (n=3) holding the same PVC filter, 5.0 um, 37 mm (SKC Inc., Eighty Four, PA, USA) and same AirChek TOUCH pump.

Test 2 studied the variability by the PVC filter structural consistency within the same lot of production using different filters (n=3) from the same manufactured lot, attached to the same 10-mm Dorr-Oliver nylon cyclone and same AirChek TOUCH pump.

Test 3 studied the variability by the PVC filter structural consistency across different manufactured lots by measuring pressure drop at different flow rates using filters from different lots (n=3) and attached to the same 10-mm Dorr-Oliver nylon cyclone and same AirChek TOUCH pump.

Tests 1-3 were repeated for the Zefon® 10mm conductive nylon cyclone (Zefon International, St. Petersburg, FL, USA), GS-3 cyclone (SKC Inc., Eighty Four, PA, USA), and aluminum cyclone (SKC Inc., Eighty Four, PA, USA). All tests were then repeated using a different commercial brand of PVC filters 5.0 um, 37 mm (Zefon International, St. Petersburg, FL, USA).

Lastly, variability due to the air sampling pump was considered by repeating all tests utilizing an SKC Universal PCXR4 pump. Overall, a total of 112 trials were completed.

DESCRIPTION OF STATISTICAL METHODS:

Data were analyzed using Minitab®, version 18 (Minitab, Inc., State College, PA, USA) and Microsoft Excel Analysis ToolPak (Microsoft Corp., Redmond, WA, USA). For each test (n=112), a simple linear regression was conducted to assess the relationship between sampler pressure drop and SVFR. A two-way analysis of variance (ANOVA) was conducted for each cyclone to determine the effect of pump type and filter brand on sampler pressure drop variability. In some instances, the necessary assumptions of no outliers and normality for each cyclone-pump-filter brand combination were not met. Therefore, analyses were rerun with outliers removed, which typically improved the normality of the data sets but did not change the overall results of the statistical test.

Because not all underlying assumptions of the two-way ANOVA were met, further comparisons were made using the nonparametric Kruskal-Wallis test. This test allows for the comparison of the medians of different populations [7]. Based on results of the two-way ANOVA as well as visual differences observed in the data sets, data for each cyclone were subdivided based on filter brand. All results were considered significant at probability <0.05.

Finally, confidence intervals for each cyclone, again was subdivided by filter brand, were calculated. Similar to the ANOVA analyses, not all necessary assumptions for one-sample t confidence intervals for the mean were met. Thus, confidence intervals for the median using the nonparametric Mood's median test were also calculated. All reported intervals were 95% confidence intervals and include any outliers so as not to discard valuable data.

Published online: August 11, 2019

Table1: Sampling train configurations

Air sampling pump	**Cyclone assembly**	**PVC filter, 5.0-µm, 37-mm**
AirChek TOUCH  Universal PCXR4 	10-mm Dorr-Oliver nylon cyclone *(n=3)* 	Brand: SKC Lot 1 *(n=3)* Lot 2 *(n=1)* Lot 3 *(n=1)* Brand: Zefon Lot 1 *(n=3)* Lot 2 *(n=1)* Lot 3 *(n=1)*
AirChek TOUCH Universal PCXR4	Zefon® 10mm conductive nylon cyclone *(n=3)* 	Brand: SKC Lot 1 *(n=3)* Lot 2 *(n=1)* Lot 3 *(n=1)* Brand: Zefon Lot 1 *(n=3)* Lot 2 *(n=1)* Lot 3 *(n=1)*
AirChek TOUCH Universal PCXR4	GS-3 cyclone *(n=3)* 	Brand: SKC Lot 1 *(n=3)* Lot 2 *(n=1)* Lot 3 *(n=1)* Brand: Zefon Lot 1 *(n=3)* Lot 2 *(n=1)* Lot 3 *(n=1)*
AirChek TOUCH Universal PCXR4	Aluminum cyclone *(n=3)* 	Brand: SKC Lot 1 *(n=3)* Lot 2 *(n=1)* Lot 3 *(n=1)* Brand: Zefon Lot 1 *(n=3)* Lot 2 *(n=1)* Lot 3 *(n=1)*

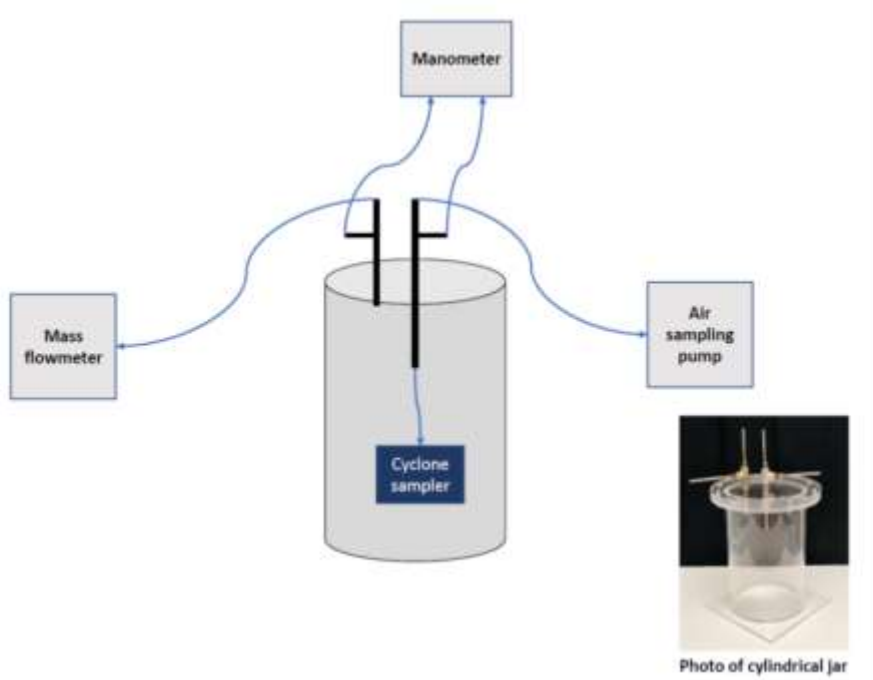

Fig. 1. *Schematic of experimental design*

RESULTS

The relation of jar pressure drop with respect to SVFR for each pump used in the study has been illustrated in Figure 2. The relation was found to be linear for both the AirChek Touch and Universal pump (R^2=0.9955, p<0.001 and R^2=0.9837, p<0.001, respectively). The relevant regression equation was used to obtain pressure drop point values, which were subsequently subtracted from the total pressure drop to calculate sampler pressure drop at different SVFRs.

The relation of sampler pressure drop and SVFR obtained for trials 1 to 3, as described in the methods section for the options of cyclone type (nylon) and filter brand (SKC) has been shown in Figure 3. The set of figures was presented in duplicate based on the personal pump type used in these trials. Figure 4 shows the same set of results now applicable to the second filter brand under consideration (Zefon). Equivalent sets of results for the Zefon, GS-3 and aluminum cyclones are available in the supplemental files.

ASSESSMENT OF INDIVIDUAL TESTS:

For each test (n=112) involving the various cyclone-filter-pump combinations, the relationship between sampler pressure drop and SVFR was linear (R^2≥0.9361, p<0.001) which was consistent with previous published studies [8-10].

Paired comparisons of regression lines for cyclone units of the same brand and with the same filter attached to them (test 1) yielded no significant differences in the values of intercept and slope. As evidenced by figures 3 and 4, regression lines were more dispersed for filters originating from the same (test 2) or different lots (test 3) when they were attached to the same cyclone unit. Paired comparisons of these cyclone-filter assemblies found linear regressions that, for most cases, had significant differences in slope and intercept.

COMPARISON OF PUMP AND FILTER BRAND:

Sampler pressure drop was calculated at the critical flow rate specified by the manufacturer for each cyclone type using the respective regression equation for each trial. These calculated point values (n=112) were segregated by pump type and filter brand (n=7 in each group) and were subsequently studied to confirm: (1) possible underlying distributions of data, (2) significant interaction of pump and filter brands on sampler pressure drop, and (3) suitable central tendency estimates of sampler pressure drop.

A two-way ANOVA was conducted for each cyclone to determine if the mean sampler pressure drop varied based on pump type and filter brand. For each cyclone, the interaction between the pump type and filter brand were not statistically significant; thus, main effects were examined individually. The effect of pump type was not statically significant for any of the cyclones. However, the effect of the filter brand was statistically significant for the Zefon (F=11.86, p=0.002) and GS-3 (F=27.06, p<0.001) cyclones and borderline significant for the nylon cyclone (F=4.04, p=0.056). The effect of the filter brand was not statistically significant for the aluminum cyclone (F=0.02, p=0.882); however, based on the results for the other cyclones, it was considered reasonable to separate the data by filter brand for this cyclone as well.

The two-way ANOVA method is fairly robust to violations of normality [7]. However, because data for the nylon, Zefon and aluminum cyclones did not meet the required assumption (normality of residuals), nonparametric statistics were also used to analyze the data. A Kruskal-Wallis test was conducted for each cyclone to determine if the median sampler pressure drop varied based on filter brand. The medians were significantly different for the Zefon (H=10.94, p=0.001) and GS-3 (H=11.25, p=0.001) cyclones. However, the difference was not statistically significant for the nylon (H=1.02, p=0.312) and aluminum (H=0.84, p=0.358) cyclones.

CENTRAL TENDENCY ESTIMATES:

For each cyclone-filter brand combination, 95% confidence intervals for the mean and median sampler pressure drop at the critical flow rate specified by the manufacturer (to provide size-selective sampling for the respirable fraction) were calculated and linear regression equations were determined (see Table 2). The intervals (both mean and median) for Zefon and GS-3 cyclones did not overlap across filter brands, corresponding with the two-way ANOVA and Kruskal-Wallis results, while there was some overlap for the nylon and aluminum cyclones.

Table 2. Sampler pressure drop at flow rates specified by cyclone manufacturers and regression equations

Cyclone Assembly	Filter Brand	Mean ΔP (in. w.g.) *(95% CI)*	Median ΔP (in. w.g.) (95% CI)	Regression Equation $Y^A = b + m(X)^B$
Nylon	SKC	1.610 (1.505, 1.714)	1.634 (1.534, 1.759)	Y = -0.4230 + 1.1990(X)
	Zefon	1.327 (1.052, 1.602)	1.447 (0.858, 1.809)	Y = -0.5356 + 1.0960(X)
Zefon	SKC	1.660 (1.465, 1.854)	1.730 (1.682, 1.809)	Y = -0.5164 + 1.2850(X)
	Zefon	2.030 (1.919, 2.141)	2.092 (1.809, 2.181)	Y = -0.6384 + 1.5700(X)
GS-3	SKC	1.710 (1.451, 1.969)	1.489 (1.409, 1.947)	Y = -0.5744 + 0.8354(X)
	Zefon	2.800 (2.404, 3.197)	3.094 (2.509, 3.304)	Y = -0.2828 + 1.2320(X)
Aluminum	SKC	2.367 (2.160, 2.574)	2.480 (2.367, 2.524)	Y = -0.3569 + 1.0930(X)
	Zefon	2.395 (2.066, 2.724)	2.625 (2.250, 2.805)	Y = -0.4381 + 1.1370(X)

[A]Pressure drop (in. w.g.)
[B]Standard volumetric flow rate (L/min)

Published online: August 11, 2019

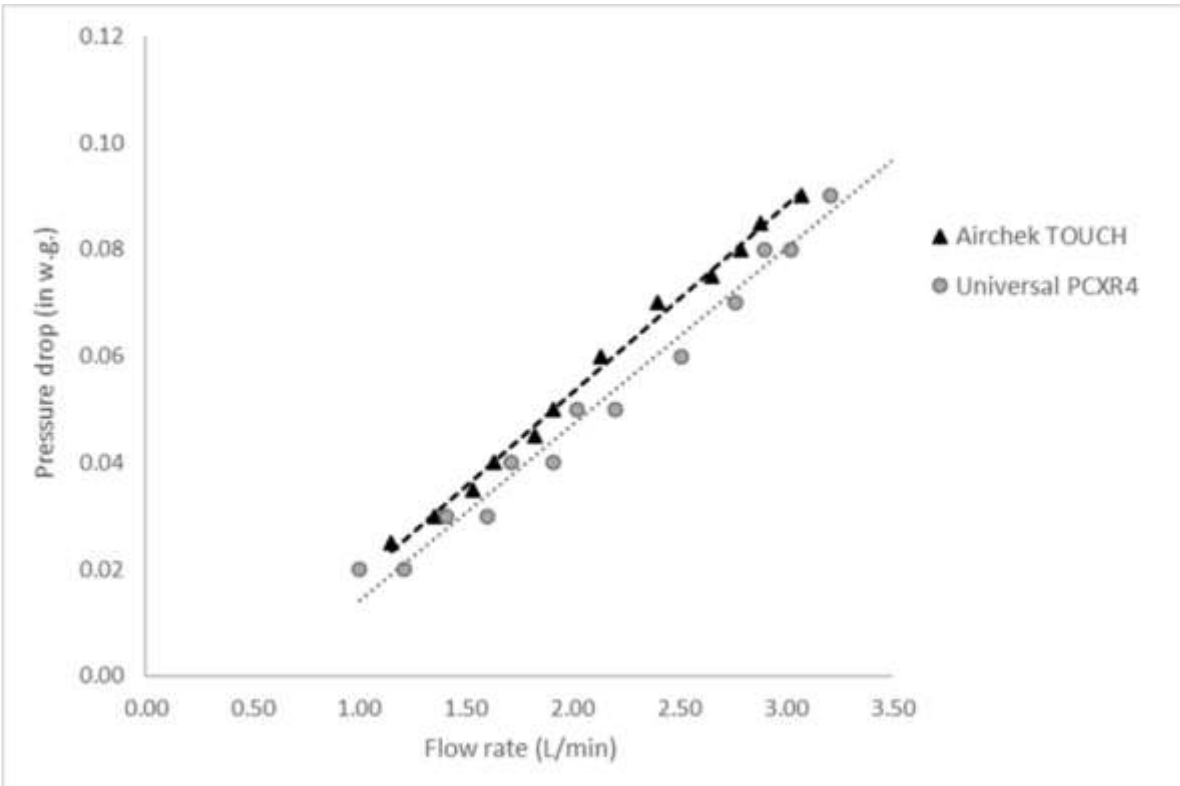

Fig. 2. Jar pressure drop as a function of standard volumetric flow rate

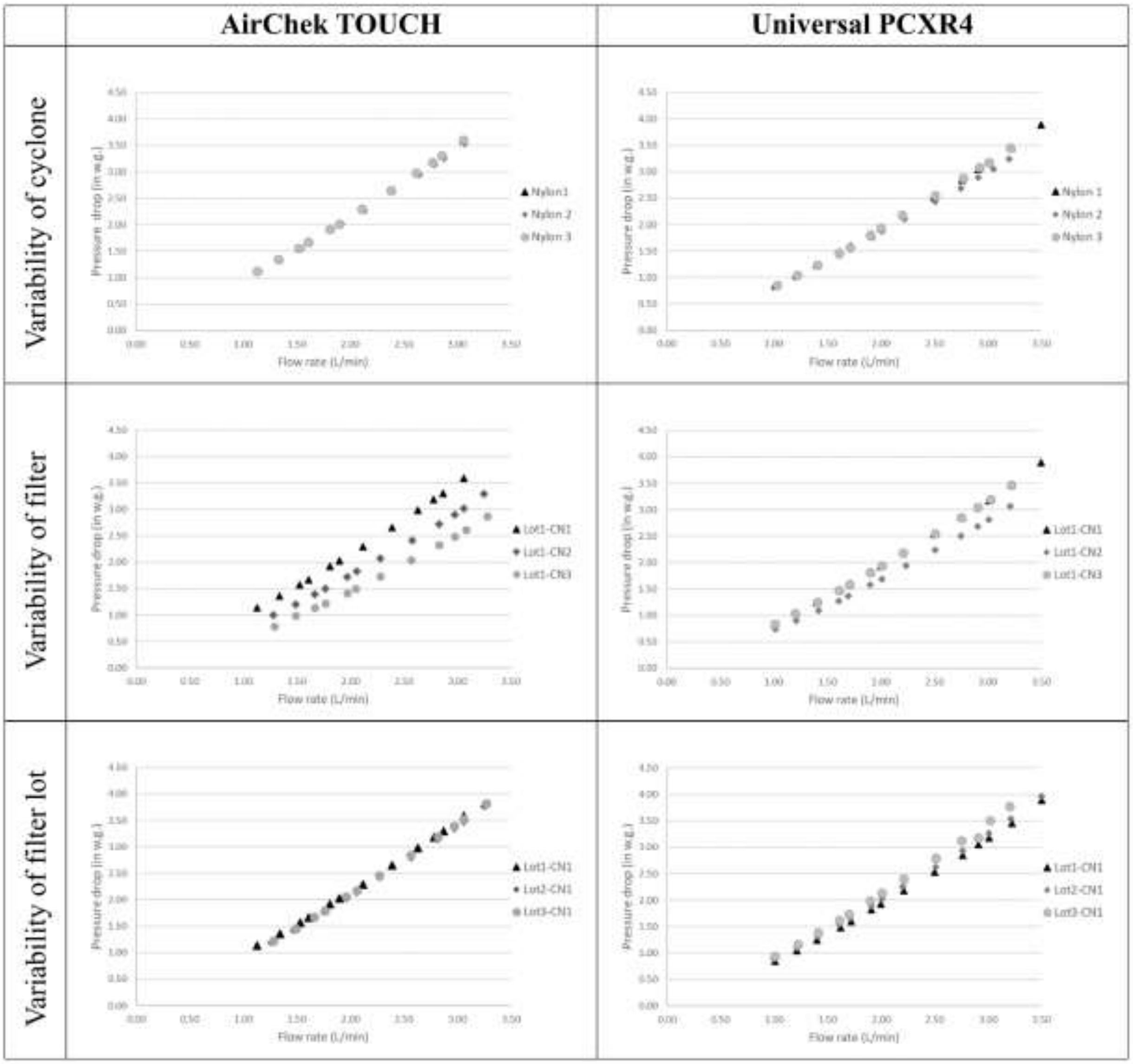

Fig. 3. Sampler pressure drop as a function of standard volumetric flow rate for nylon cyclone with SKC filters

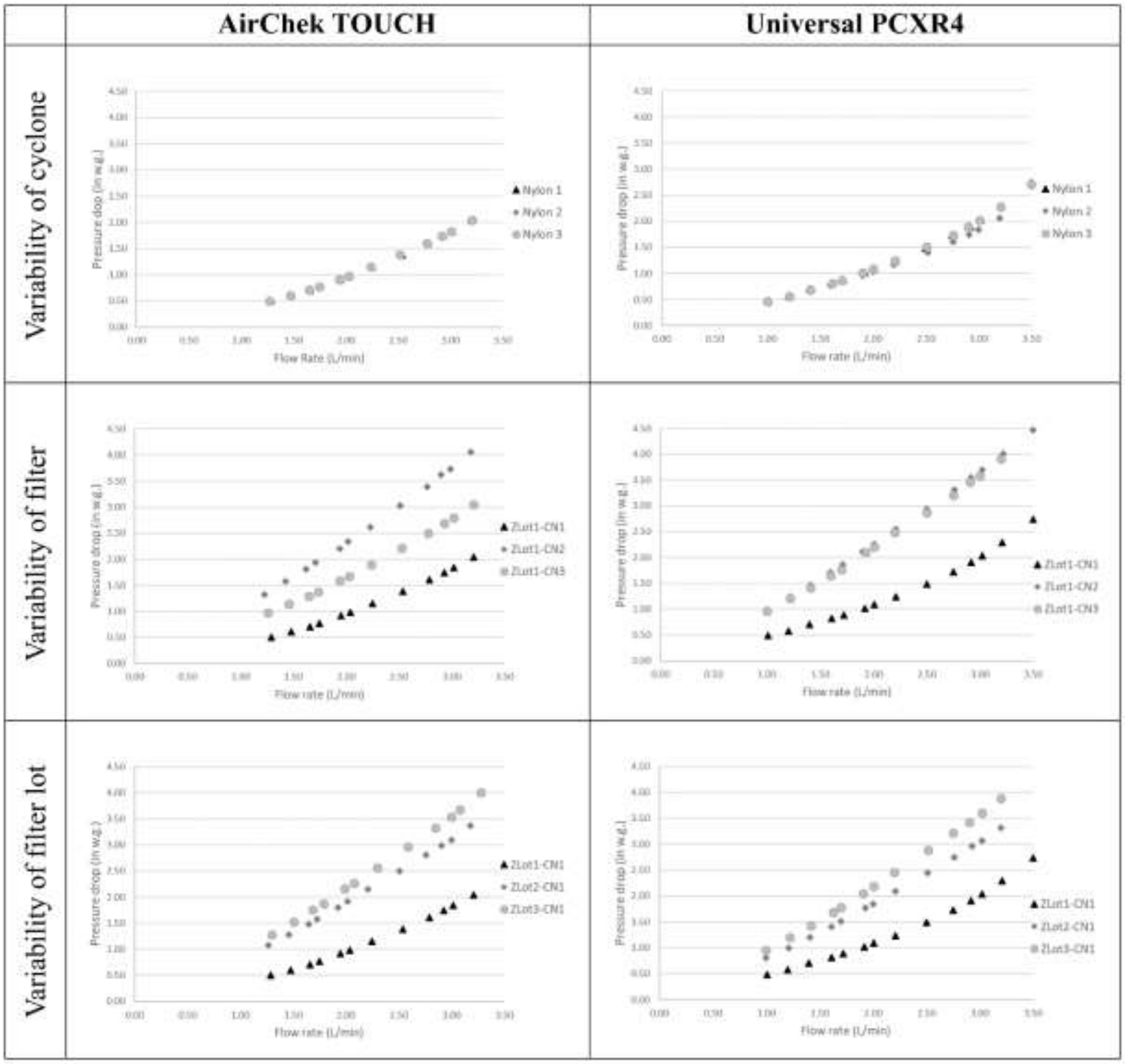

Fig. 4. Sampler pressure drop as a function of standard volumetric flow rate for nylon cyclone with Zefon filter

DISCUSSION

A unique value of sampler pressure drop at the critical flow rate was not found for any of the respirable dust cyclones under consideration. The cyclone type obviously made a difference in the sampler pressure drop value expected, along with the filter type, regardless if the filter sample came from the same or different brand or the same or different lot of production.

As shown in Figures 3 and 4, the intra-cyclone trials did not yield significant variability in the sampler pressure drop value at the critical volumetric flow rate. This finding was confirmed consistency in quality control of cyclone fabrication and was assured that selecting a given cyclone from a group of equal units should not create significant differences in the expected pressure drop at the critical flow rate. The limited intra-cyclone variability of sampler pressure drop supports the use of this parameter as a valid surrogate for volumetric flow rate.

Unlike intra-cyclone tests, the change in filters did make a difference in the sampler pressure drop value. For tests involving different filters from the same brand, the sampler pressure-drop exhibited significant differences regardless of the filter's origin (same or different lots). Moreover, when the interaction of pump type and filter brand on sampler pressure drop value were examined, the two-way ANOVA showed that significant effects on pressure drop could be attributed to the filter brand but not the pump type. This finding was consistent with the results from a study conducted by Soo et al. where the authors concluded pressure drop increased with an increased sampling flow rate but differed among filter manufacturer [11].The differences introduced by the filters to the sampler pressure drop made the one-on-one relation between sampler pressure drop and volumetric flow rate less determined, eliciting a need for statistical estimates of pressure drop, considering the variability of the data. A possible cause for the filter-pressure drop dispersion could be attributed to the variability expected in pore size distribution that is not assessed by the pore size measurement protocol [12]. Perhaps if efforts were made toward improving structural uniformity of filters, the variability on pressure drop of the filter substrate would be reduced. This advance in filter design will improve the "tightness" of the value of sampler pressure drop and critical volumetric flow rate and will facilitate even further the use of pressure drop instead of volumetric flow rate as a technique for the calibration of sampling trains.

In search for a central tendency estimate of pressure drop at the critical flow rate, possible underlying distributions of data were examined. The normal distribution hypothesis was rejected in most cases, making the mean value and the respective confidence interval around the mean not recommended as calibration guides. The lack of a single and known distribution of data across all segregated sets of data led to the selection of the median value as central tendency measure of preference. Confidence interval around the median value were found by non-parametric statistics and yielded, as expected, a wider range of variation.

The regression lines equation for each cyclone-filter brand combination was used to calculate the equivalent volumetric flow rate for the median, upper and lower confidence limits values of sampler pressure drop (based on the 95% confidence limits). For each cyclone-filter combination the volumetric flow rate variation around the median value was between 1.6% and 22.2% above and between 2.1% and 29.7% below the median value. Calibration jars are not air-tight by nature and can introduce undetectable air leaks, resulting in measurement error up to ± 30% [3]. Therefore, if the flow rate is within the 95% confidence limit after selecting the median value of pressure drop as the calibration set point, the expected error would be less than the uncertainty created by a poor seal of the calibration jar. This fact, along with the simplicity of pressure drop calibration, would favor the latter as a calibration tool.

CONCLUSIONS

This study investigated the relationship between pressure drop and volumetric flow rate for various combinations of sampler type, commercial filter brand, and pump type. Overall, pressure drop increased with increasing flow rate. Sampler pressure drop at the critical volumetric flow rate specified by the cyclone manufacturer varied based on sampler type as expected. Repeated tests involving samplers of the same type did not exhibit intra-cyclone variability on sampler pressure drop, indicating consistency in cyclone production. However, sampler pressure drop varied significantly based on a filter selection that considered intra- and inter-samples of two commercial brand of filters. Unlike filters, pump type did not affect significantly sampler pressure drop.

The recommended best estimate of central tendency of the sampler pressure drop at the critical flow rate was the median value, which must be obtained by grouping data by cyclone type and filter brand. Confidence intervals of the median values set by nonparametric statistics models yielded the expected error in volumetric flow rates when calibration of sampling trains was based on pressure drop. The expected error was in each case under consideration less than that reported for the conventional calibration jar method.

ACKNOWLEDGEMENTS

This study was supported by Grant 5T03OH008624-14 from the National Institute for Occupational Safety and Health (NIOSH). Its contents are solely the responsibility of the authors and do not necessarily represent the official views of NIOSH.

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