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Determination of paraffin oil mist penetration at high flow rates through air-purifying respirators

Agnieszka Brochocka  and Małgorzata Okrasa 

Central Institute for Labour Protection – National Research Institute, Poland

ABSTRACT

This article presents paraffin oil mist penetration tests of commercially available air-purifying respirators of different construction conducted using the method described by Standard No. ISO 16900-3:2012, which incorporates flow rates (up to 255 l/min) of test aerosol. The testing method reflects differences in work intensity during the use of respirators. Moreover, the experimental stand, designed according to the international specifications, is described. The results show that the higher the paraffin oil mist flow rate, the higher the penetration index, irrespective of the testing method used and the type of respirator investigated. While at high flow rates, filtering half masks of the first protection class (FFP1) met the requirements of their protection class according to European Standard No. EN 149:20001+A1:2009, filtering half masks of the second and the third protection class (FFP2 and FFP3) did not.

KEYWORDS

penetration index; paraffin oil mist; air-purifying respirators; high flow rates; ISO standards

1. Introduction

In the European Union, air-purifying respiratory protective devices (RPDs) are tested and classified according to relevant European standards (EN) harmonized with Regulation 2016/425 [3]. However, coming years will see the wide adoption of new, international standards for RPD assessment. This implies an entirely new classification of RPDs, more stringent requirements and different test methods that should be considered while designing new materials and products or adjusting the existing ones to market requirements.

One of the most important parameters affecting filtering media performance and protection levels of filtering RPDs is the penetration index, which indicates the proportion of particles captured by the device. The existing methodology of testing liquid aerosol penetration through RPDs, based on Standard No. EN 149:2001+A1:2009 [2] and Standard No. EN 13274-7:2007 [4], involves measuring the concentration of the test aerosol (paraffin oil mist) using a laser photometer at 95 l/min.

In the USA, the National Institute for Occupational Safety and Health (NIOSH) examines air-purifying RPDs using charge-neutralized liquid dioctyl phthalate (DOP) using an automated tester with a flow rate of 85 l/min [5]. According to the new International Organization for Standardization (ISO) standard requirements, the performance of air-purifying RPDs will be assessed using particle number-based methods and an experimental stand consisting of a photometer, aerosol generator, flow meter and test chamber. Moreover, filter media are to also be tested at a whole range of flow rates (up to 255 l/min).

In the international literature, there are few publications devoted to testing methods involving liquid aerosols. This issue is particularly relevant for electret filter media used in RPDs, as their performance quickly deteriorates due to the deposition of aerosol droplets on the fiber surface. Existing reports primarily focus on evaluating the penetration of liquid DOP aerosols through various filter media and RPD types.

Martin and Moyer [6] studied filter media and respirators in terms of penetration by DOP with a median particle size of $0.075 \pm 0.020 \mu\text{m}$ with a geometric standard deviation of less than 1.6. The tests were conducted at a flow rate of 85 l/min for particulate filters, and at half that value (42.5 l/min) for filters deployed in pairs. Samples were dipped in isopropanol for 15 s to reduce electrostatic charges. DOP penetration tests were found to be more stringent than tests involving solid aerosols, such as sodium chloride. Furthermore, reduction or elimination of electrostatic charges led to a dramatic (two-fold to three-fold) increase in aerosol penetration.

Huang et al. [7] studied the penetration of monodisperse liquid aerosols through polysulfone materials used in membrane filters. Those materials were characterized by good physicochemical properties, including thermal and chemical stability, mechanical strength and excellent oxidative resistance. The experimental system used in that study consisted of an aerosol generator, electrostatic particle classifier, neutralizer, mixing column, filter holder, aerosol electrometer, condensation particle counter, pressure gauge and flow meter. The liquid test aerosol was single-charged monodisperse DOP with a concentration of 5×10^3 particles/cm³ and a submicrometer-sized range from 0.03 to 0.05 μm with a geometric standard deviation of 1.10–1.15. Tests were conducted at different linear flow rates (5, 10 and 20 cm/s). It was found that the most penetrating particle size for polysulfone membrane filters with different morphological structures was 0.05 μm .

Furthermore, Martin et al. [8] investigated powered particulate respirator filter penetration by electrostatically charged and uncharged (neutralized) liquid DOP aerosols at higher flow rates: 83, 100, 147, 224 and 225 l/min using a laser photometer. The filters were found to be less efficient for uncharged aerosols. The same method was used by Jaspen et al. [9], who evaluated the performance of three different electret filter media and a glass non-woven material following exposure

to ethylbenzene vapor and liquid. The count median particle diameter of the DOP aerosol used in the tests was $0.20\ \mu\text{m}$ with a geometric standard deviation of less than 1.6. The aerosol flow rate was constant at $42.5\ \text{l/min}$. Direct contact of electret filter materials with ethylbenzene in the liquid phase was found to reduce their filtration performance by 40%, which is attributable to changes in the static electric field due to the deposition of liquid ethylbenzene on the fiber surface. On the other hand, the filtration performance of the glass non-woven material was unaffected by exposure to ethylbenzene, whether in gas or liquid form.

Brochocka et al. [10] studied liquid aerosol penetration through plasma-modified polycarbonate filter media according to the methodology described in Standard No. EN 13274-7:2007 [4] on determination of particle filter penetration. The experimental apparatus consisted of a laser photometer and a paraffin oil mist generator; the aerosol flow rate was $95\ \text{l/min}$. The size distribution of aerosol particles was log-normal with a median Stokes diameter of $0.4\ \mu\text{m}$ and a geometric standard deviation of 1.82. Polycarbonate filter media with fibers modified by cold plasma in argon were found to provide an efficient barrier to paraffin oil mist.

Furthermore, different methods of testing the performance of filter media and air-purifying RPDs have been evaluated by several research teams. A group of NIOSH researchers and Rengasamy et al. [11] compared the penetration of a liquid DOP aerosol and a solid NaCl aerosol through filters preloaded with various amounts of DOP. It was reported that penetration by the liquid aerosol was higher than that by the solid one irrespective of the amount of DOP loading. Moreover, filter performance declined with increased DOP loading due to the neutralization of electrical charges by ionic conduction through the oil film deposited on the fibers.

Chattopadhyay et al. [12] evaluated the filtration performance of electrospun cellulose acetate fibers using solid and liquid aerosols, i.e., NaCl and diethyl-hexyl sebacate (DEHS), respectively. The liquid aerosols exhibited a narrow particle size distribution of $0.09\text{--}0.35\ \mu\text{m}$ with a geometric standard deviation of less than 1.35. Using a particle number-based test method with an electrostatic classifier, condensation particle counter and electrostatic charge neutralizer, the authors reported higher penetration by the liquid aerosol as compared to the solid one, which was consistent with previous research. Moreover, it was found that oil particles tended to form a film on cellulose acetate fibers.

Rengasamy and Eimer [13] evaluated air-purifying particulate respirators, challenged with the same aerosols, using two methods: photometry and particle number detection. Aerosol penetration measured using a laser photometer was lower than that obtained with a particle counter. The study's objective was to better understand the effects of crucial test parameters, such as the filter medium type, aerosol size distribution and detection system. It was found that more rigorous RPD tests could be achieved using particle counters with a suitable measurement range.

Although there exist several methods for evaluation of the performance of filter media challenged by different liquid aerosols, it seems that a standardized protocol using one type of test aerosol at a predefined concentration, flow rate and particle size distribution (as specified in Standard No. ISO 16900-3:2012 [1]) should be used to assess the performance of air-purifying RPDs. This article aimed to show how filtering half masks commercially available in the EU perform when tested according to the newly proposed methodology.

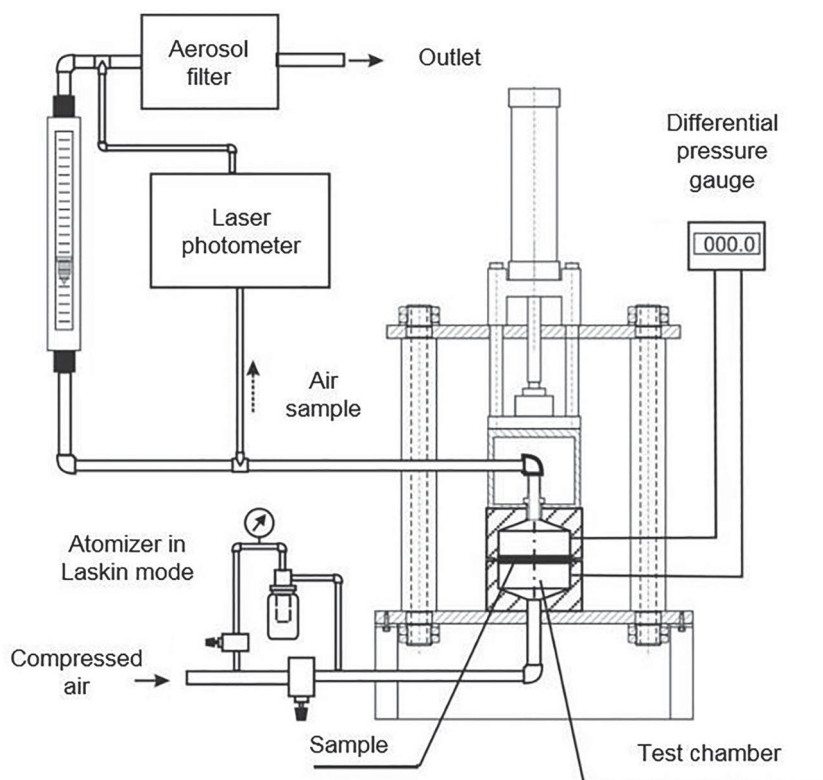
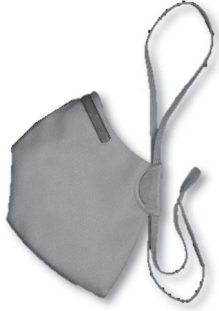



Figure 1. Diagram of the experimental stand for testing paraffin oil mist penetration according to Standard No. ISO 16900-3:2012 [1].

Table 1. Characteristics of the tested filtering half mask.

Characteristic	Type of filtering half mask	Flat-fold filtering half mask	Cup-shaped filtering half mask
Photograph of filtering half mask			
Filtering half mask construction	FFP1 ^a	<ul style="list-style-type: none"> • Polypropylene spun-bonded structural non-woven material with an area density of 40 g/m² • Polypropylene melt-blown filter non-woven material with an area density of 50 g/m² • Two layers of polypropylene spun-bonded structural non-woven material with an area density of 40 and 60 g/m² 	<ul style="list-style-type: none"> • Polyester needle-punched structural non-woven material with an area density of 160 g/m² • Polypropylene melt-blown filter non-woven material with an area density of 50 g/m² • Polyester needle-punched structural non-woven material with an area density of 200 g/m² with a prefilter
	FFP2 ^a	<ul style="list-style-type: none"> • Polypropylene spun-bonded structural non-woven material with an area density of 17 g/m²• Polypropylene melt-blown filter non-woven material with an area density of 70 g/m²• Two layers of polypropylene spun-bonded structural non-woven material with an area density of 80 and 40 g/m² 	<ul style="list-style-type: none"> • Polyester needle-punched structural non-woven material with an area density of 160 g/m² • Polypropylene melt-blown filter non-woven material with an area density of 70 g/m² • Polyester needle-punched structural non-woven material with an area density of 200 g/m² with a prefilter
	FFP3 ^a	<ul style="list-style-type: none"> • Polypropylene spun-bonded structural non-woven material with an area density of 17 g/m² • Two layers of polypropylene melt-blown filter non-woven material with an area density of 70 and 20 g/m² • Two layers of polypropylene spun-bonded structural non-woven material with an area density of 80 and 40 g/m² 	<ul style="list-style-type: none"> • Polyester needle-punched structural non-woven material with an area density of 160 g/m² • Two layers of polypropylene melt-blown filter non-woven material with an area density of 70 and 20 g/m² • Polyester needle-punched structural non-woven material with an area density of 200 g/m² with a prefilter

^aClassification according to Standard No. EN 149:2001+A1:2009 [2].

Note: FFP1 = filtering half mask of the first protection class; FFP2 = filtering half mask of the second protection class; FFP3 = filtering half mask of the first protection class.

2. Materials and methods

The experimental stand was designed according to Standard No. ISO 16900-3:2012 [1] and consisted of four modules: a DOP 3500 Touch laser photometer (DOP Solutions, UK), an ATM 221 Laskin atomizer (TOPAS, Germany), a pneumatic sample holder and an airflow control module incorporating valves and two rotameters with measurement ranges of 10–100 l/min and 30–300 l/min (Figure 1).

The laser photometer measured the aerosol concentration in the range of 0.001–150 mg/m³ with a sensitivity of 0.000001 mg/m³. The Laskin atomizer supplied with compressed air was used to generate a liquid aerosol at a concentration of 15–35 mg/m³ (not deviating from the initial concentration by more than ±10% throughout a test) at flow rates of 30–300 l/min.

An important parameter affecting paraffin oil mist penetration tests is the median particle diameter distribution, which according to Standard No. ISO 16900-3:2012 [1] should be between 0.16 and 0.21 μm with a geometric standard deviation of 1.4–1.8. The particle size distributions of the generated aerosol were determined using a condensation particle counter (model 3775; TSI, USA) connected to an electrostatic classifier (model 3080; TSI, USA). Measurements were carried out for nine selected aerosol flow rates: 35, 65, 85, 95, 120, 135, 170, 205 and 255 l/min, respectively.

To evaluate the performance of respirators, the experimental stand was used to determine the penetration index for two types (flat-fold and cup-shaped) of commercially available air-purifying respirators (SI ZGODA, Poland). Specimens representing all three protection classes (FFP1, FFP2 and FFP3) were tested for each respirator type. The construction of the respirators is presented in Table 1.

During the measurements, the Laskin atomizer generated aerosol with a concentration of 20 mg/m³, which was passed at flow rates ranging from 35 to 255 l/min through an air-purifying respirator mounted in the pneumatic holder. The aerosol concentration upstream and downstream of the sample was ascertained using a laser photometer. The penetration index for paraffin oil mist, P_{ISO} , was computed using the following equation:

$$P_{ISO} = \frac{c_1}{c_2} \times 100\%, \quad (1)$$

where c_1 = aerosol concentration downstream of the sample; c_2 = aerosol concentration upstream of the sample.

Results were read after 5 min of testing in the initial phase of filtration. Nine specimens of each type and class of air-purifying respirator were tested.

The obtained results were compared to the penetration indices determined according to the current European

Standard No. EN 13274-7:2007 [4]. The experimental apparatus consisted of an AGW-F/type BIA aerosol generator, an FH 143/14 pneumatic holder and a Lorenz AP2E laser photometer (Lorentz, Germany). Measurements were conducted at a paraffin oil mist concentration of 20 mg/m³ and flow rates of 35, 65, 85 and 95 l/min. The aerosol particle size distribution was log-normal with a median Stokes diameter of 0.4 µm and a geometric standard deviation of 1.82.

The penetration results obtained using the two methods involving two different experimental stands were analyzed statistically at a significance level of $\alpha = 0.05$. Measurement data were evaluated with the Shapiro–Wilk test for normality of distribution. In all cases, the obtained p values were higher than the adopted significance level, which meant that the null hypothesis about normal distribution was not rejected. Subsequently, the parametric Fisher–Snedecor and Student tests were conducted to determine differences between variances and mean penetration values.

3. Results and discussion

The results of aerosol size distribution measurements are presented in Table 2 with median size distributions for different flow rates shown in Figure 2.

The median values of particle size distributions for paraffin oil mist were found to comply with the Standard No. ISO 16900-3:2012 [1] specifications for all airflow rates except for the lowest (35 l/min). A comparison of the median values indicates a slight decrease in particle size with an increase in air-flow rate. The geometric standard deviation conformed to the ISO standard as well.

Figure 3 shows the paraffin oil mist penetration measurements through two types of air-purifying respirators, available in three protection classes each, which were carried out on the experimental apparatus configured according to the ISO standard. The obtained penetration indices were compared with the results determined using the experimental stand assembled according to European Standard No. EN 13274-7:2007 [4]. Only flow rates of up to 95 l/min were used due to the technical limitations of the set-up (our apparatus was calibrated in the flow range between 0 and 95 l/min).

A linear function can approximate the relationship between the penetration index and the test aerosol flow rate. The solid lines in Figure 3 represent a regression model with parameters resulting from the minimization of the sum of square errors. The corresponding coefficients of determination R^2 vary from 0.87 to 1, indicating an excellent fit to the experimental data.

For every studied respirator type and protection class, the penetration index increased with airflow. The most significant rise was observed for filtering half masks with the lowest protection class. Because aerosol penetration is one of the primary determinants of the RPD protection class, its increase directly affects equipment classification. The slopes of the regression lines illustrate the scale of penetration changes. Penetration indices decreased with increasing protection class for both test methods and both respirator types. This means that in high-performance RPDs, higher aerosol flow rates lead to a steeper decline in penetration resistance.

At low aerosol flow rates, the results obtained on the two experimental stands were essentially the same. Some slight statistical differences were only observed for cup-shaped respirators of lower protection classes for specific flow rates.

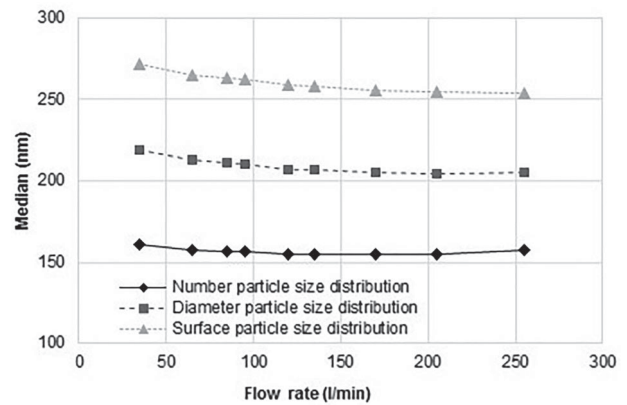


Figure 2. Medians of particle size distributions for different air flow rates.

Table 2. Particle size distributions of paraffin oil mist.

Flow rate (l/min)	Parameter	Particle size distribution		
		Number	Diameter	Surface
35	<i>Mdn</i> (nm)	160.6	218.9	272.1
	<i>M</i> (nm)	177.4	229.9	273.7
	GM (nm)	149.7	205.5	253.7
	GSD	1.87	1.66	1.51
65	<i>Mdn</i> (nm)	157.4	212.7	265.2
	<i>M</i> (nm)	174.4	225.0	268.6
	GM (nm)	148.3	201.2	248.5
	GSD	1.83	1.65	1.52
85	<i>Mdn</i> (nm)	156.8	210.9	263.1
	<i>M</i> (nm)	173.8	223.6	266.9
	GM (nm)	148.4	200.0	246.8
	GSD	1.82	1.65	1.52
95	<i>Mdn</i> (nm)	156.9	210.5	262.4
	<i>M</i> (nm)	173.8	223.3	266.5
	GM (nm)	148.6	199.9	246.4
	GSD	1.81	1.64	1.52
120	<i>Mdn</i> (nm)	154.7	207.3	259.0
	<i>M</i> (nm)	171.8	220.6	263.8
	GM (nm)	147.1	197.3	243.6
	GSD	1.80	1.64	1.52
135	<i>Mdn</i> (nm)	154.9	206.7	258.0
	<i>M</i> (nm)	171.9	220.1	263.1
	GM (nm)	147.5	197.1	243.0
	GSD	1.79	1.64	1.52
170	<i>Mdn</i> (nm)	154.7	204.9	255.5
	<i>Mv</i> (nm)	171.5	218.7	261.3
	GM (nm)	148.0	196.1	241.3
	GSD	1.77	1.63	1.52
205	<i>Mdn</i> (nm)	155.1	204.3	254.3
	<i>M</i> (nm)	172.0	218.3	260.5
	GM (nm)	149.0	196.0	240.6
	GSD	1.75	1.63	1.52
255	<i>Mdn</i> (nm)	157.3	204.9	254.0
	<i>M</i> (nm)	174.1	219.0	260.6
	GM (nm)	152.0	197.3	240.9
	GSD	1.72	1.61	1.52

Note: GM = geometrical mean; GSD = geometrical standard deviation.

Those differences may occur irrespective of the test method used and may be attributable to respirator construction.

Notwithstanding the aforementioned, extrapolation of penetration measurements conducted according to the EN

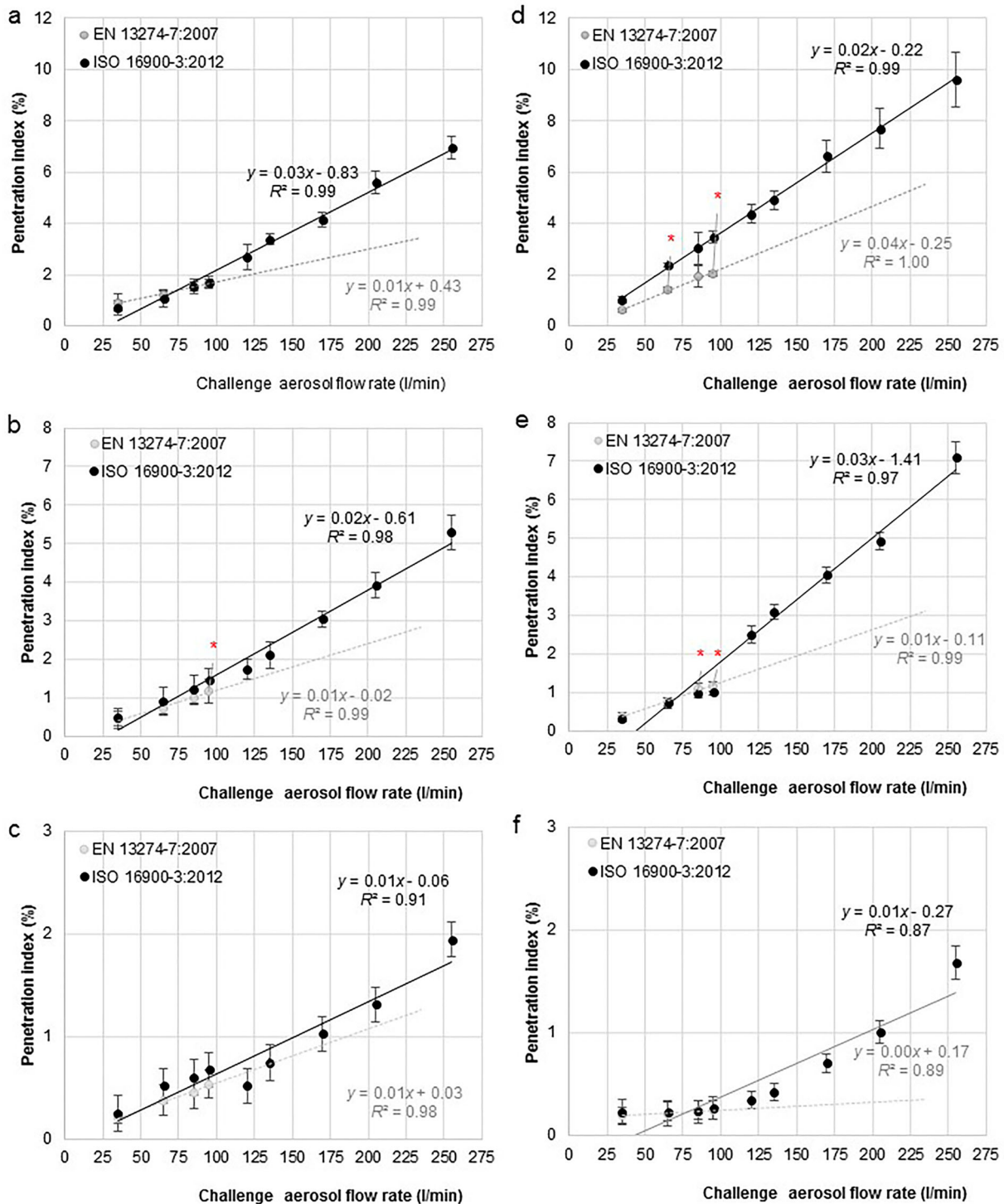


Figure 3. Penetration indices for flat-fold filtering half masks FFP1 (a), FFP2 (b) and FFP3 (c), and for cup-shaped filtering half masks FFP1 (d), FFP2 (e) and FFP3 (f) at different aerosol flow rates. *Results for which Student's *t* test revealed statistically significant differences at 0.05.

Note: FFP1 = filtering half masks of the first protection class; FFP2 = filtering half masks of the second protection class; FFP3 = filtering half masks of the third protection class.

standard shows that at higher aerosol flow rates, the two test methods may lead to divergent results, possibly affecting the protection classification of RPDs as the penetration indices obtained with the new methodology tend to be higher for all air-purifying respirator types and classes. This implies that equipment manufacturers can face more stringent requirements concerning filter medium efficiency and respirator construction.

It is also worth noting that an aerosol penetration test with a cyclic flow more closely simulates real-world breathing conditions, which feature a constantly changing flow depending on the level of the breathing frequency. However,

various studies that have addressed the effect of flow patterns on filter efficiency show that a reasonable approximation of the cyclic flow can be achieved by selecting an equivalent constant flow rate within the range of the mean and peak inhalation flow [14–17].

4. Conclusions

The presented experimental stand was successfully deployed to test air-purifying RPDs according to the requirements of Standard No. ISO 16900-3:2012 [1]. The test set-up was evaluated by determining the effect of the flow rate on the

particle size distribution of paraffin oil mist. The medians of particle diameter distributions were found to comply with the requirements of the standard. Two types of air-purifying respirators with varying efficiency were then evaluated in terms of paraffin oil penetration using a new ISO test stand and a conventional test stand complying with Standard No. EN 13274-7:2007 [4]. The results obtained on both test stands were then compared. Irrespective of the test methodology and the type of air-purifying respirator, paraffin oil mist penetration increased with the flow rate. The slopes of the linear regression lines approximating the relationship between the penetration index and the airflow rate revealed more dynamic penetration changes in the case of ISO procedures. It was also shown that the higher the protection class of the RPD, the greater the difference between the results obtained on ISO apparatus compared to the predicted results for the EU-based test stand.

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Disclosure statement

No potential conflict of interest was reported by the authors.

ORCID

Agnieszka Brochocka  <http://orcid.org/0000-0003-3386-9629>

Małgorzata Okrasa  <http://orcid.org/0000-0003-4980-0909>

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