

Breakthrough Measurements for Small-Capacity Cartridges at Elevated Flow Rates in Different Flow Modes

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ABSTRACT

The new user-oriented ISO approach in the standardization of respiratory protective devices suggests the possibility of integrating different work rate levels expressed in volumetric flows into certain respirator tests. There are fears that very small gas filter cartridges might reveal shortcomings under such forced conditions due to their shallow bed depth in relation to the required breakthrough time of certain gases for adsorption. This concern was the reason for a pre-standardization study on the dynamic behaviour of a selected small gas filter type. Testing with cyclohexane mainly at 300 ppm in continuous and sinusoidal flow mode was performed at different average flow rates ranging from 30 to 80 lpm. Even at the beginning of the test, the small cartridges showed penetration of the test gas in nearly all test conditions. Starting at different levels, the downstream concentration increased slowly over time in relation to the flow mode, flow rate and test gas concentration. By comparison, an ABEK1 used as a reference filter did not show this behaviour at all. Furthermore, it was found that the residence time is significantly lower in dynamic flow mode than in continuous mode at the same average flow rate. In view of the observed and characterised effect of instantaneous breakthrough, there is a need for further systematic investigation with even higher flow rates and other type of gases with their respective filters. This will provide the ISO standardisation committees with sufficient information for the establishment of a valid basis for integration of the findings into a residence test to improve the safety of gas filters.

Keywords: gas filter, small capacity, dynamic flow, sinusoidal breathing, breakthrough time, high flow rates, residence time

INTRODUCTION

A new approach in the worldwide standardisation (ISO) of respiratory protective devices focuses on the needs of the user. A principal concern of the approach is to ensure that RPDs are safe in use even in very different and extreme working conditions, e.g. prolonged work and high work rates. A project group under ISO/TC 94/SC15 Respiratory Protective Devices has been established to examine the human physiological aspects. In its draft technical specification (ISO approach, 2005), the group established a correlation between higher work rates and air demand expressed as average volume flow rate in lpm of inhaled air. It also ascertained the dependency of minute volume flow on peak inspiratory flow rate during a respiratory cycle for different standardised persons. A brief summary of this study is shown in the annex where the work rate is classified and correlated with the type of work, minute volume and peak flow rates. On the basis of these investigations, the responsible standardisation committees intend to establish requirements for the performance testing of RPDs. They also wish to introduce different work

rates with their corresponding flow rates in respiratory testing to make sure that RPDs remain safe even in these extreme conditions. There are certain fears that Class 1 gas filters conforming to EN 14387:2004 and particularly the smaller gas filter cartridges available in Asian countries might reveal weaknesses in terms of potential early breakthrough in such forced conditions due to their low carbon volume in relation to the required residence time of some gases for adsorption.

This study is being conducted in order to examine the dynamic performance of small gas filters and to provide the relevant ISO committees with crucial information for the establishment of a dynamic gas test – a so-called residence test – which is different from the widely used standard capacity test.

This paper describes the testing of a specially selected small gas filter for organic vapours with cyclohexane mainly at 300 ppm under continuous and sinusoidal flow conditions at different average flows ranging from 30 to 80 lpm. The results are compared to those of an ABEK1 reference filter.

METHODS AND MATERIALS

Gas Filter

All the tests focused on a gas filter for organic gases/vapours, which is referred to in the following as a “small-capacity gas filter” (CA-705/OV, Shigematsu Works Co., Ltd.). The specifications are summarized in Table I. Such filters are classified in Japanese and Australian standards (AS/NZS 1716:2003), for example. A breakthrough time of approximately 100 minutes for 300 ppm cyclohexane is stated on the filter packaging. Its capacity expressed as the breakthrough time under defined test conditions for organic vapours is less than half of what is required in the European standard EN 14387:2004 for Class A1. An ABEK1 filter (671 A1B1E1K1, Dräger Safety AG & Co. KGaA), which has been tested against EN 14387:2004 and approved under the European Directive 89/686/EEC, has been used as the reference device.

Table I. Specifications of the Small-Capacity Filter (CA-705/OV)

Activated carbon mass	23 g
Housing mass	21 g
Dimensions of carbon bed: diameter x height	7.6 cm x 1.1 cm
Resistance at 95 l/min	200 Pa

Test Gas Preparation

For continuous flow mode, a mass flow controller is used to generate the required flow, which is humidified by a temperature-controlled water bath. After this, the conditioned test air is heated up to 50 degrees to vaporize the cyclohexane, which is pumped as a liquid in small quantities into the gas stream by a diaphragm pump (BF411, Telab Technology GmbH). This test gas is passed through a number of condensers to achieve a temperature of 20 degrees and the required humidity, before arriving in the test chamber. The test gas concentration and the breakthrough concentration were measured with different flame ionization detectors (FID): Model 3006, Bernath Atomic GmbH & Co. KG, for the test gas concentration and a Beckman Model 400 A, Beckman Industrial Process Instruments Division, for the breakthrough concentration. The experimental set-up is shown in the annex.

The dynamic flow was generated by an artificial lung (Model AL92-C, Walter & Gareiss) which simulates human breathing with a sinusoidal function (Figure 1). In contrast to the set-up for continuous

mode, a flexible gas bag had to be introduced as a buffer to compensate the peak flows and prevent gas penetrating from the environment into the system. The required test gas concentration was generated continuously and adjusted to the desired average flow rate until the gas bag was full. The lung then started to draw the required test gas from the downstream side of the filter. For detection of the breakthrough concentration, the same FID detectors were used. The experimental set-up is shown in the annex.

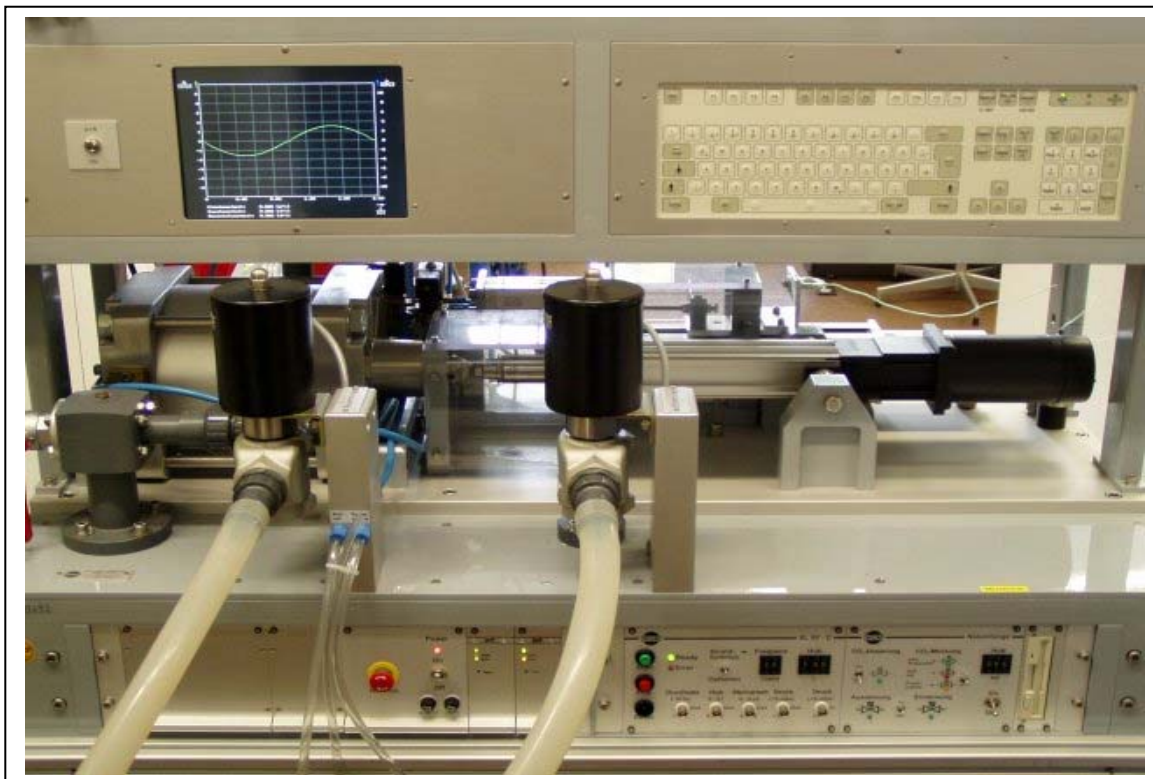


Figure 1. Breathing machine.

Filter Testing

Two sets of experiments were carried out with the small-capacity filter. The first set was with continuous flow and the second set with dynamic (sinusoidal) flow, both with cyclohexane concentrations of 300 and 1000 ppm and flow rates ranging from 30 to 80 lpm. Humidity was set at 50%, limited by the maximum achievable humidity for sinusoidal breathing mode, and the temperature was set to 20°C for all experiments.

As a reference for the small-capacity filter, most of the tests were also carried out with an ABEK1 filter. The range of average flow rates from 30 to 80 lpm (corresponding to peak flows from 90-240 lpm in sinusoidal mode) for two flow modes were selected to cover the most important work rate levels from moderate work to intensive work, as defined in current ISO draft standards.

RESULTS AND DISCUSSION

Small-Capacity Filter

Figures 2 and 3 show a phenomenon which has not yet been observed in this laboratory for the testing of Class 1 filters to European standards. A test gas emission can be detected on the downstream side of the filter even in the first minutes of the test. To make sure that this instantaneous penetration is not caused by leakage due to improper sealing of the cartridge within the test chamber, the chart recorder was switched on before the filter was fixed in the chamber. The first large peak (Fig. 3) reaching about 23 ppm, is due to the test gas effusing from the open chamber and partly being picked up by the sampling line to the detector. When the cover of the chamber was closed, the detector signal decreased sharply and increased again due to the onset of the instantaneous penetration of cyclohexane on the downstream (clean) side of the filter. If there were systemic leakage, the signal would not reach this observed low level again. The signal could not return to its initial level due to the detector's slow decay after the previous high exposure.

Instantaneous penetration can be observed in all applied conditions, its extent largely depending on the test concentration, flow mode and flow rate. At a cyclohexane concentration of 300 ppm, the emission's initial level increases with increasing flow in continuous mode. By contrast, dynamic mode does not show any clear dependence of the initial level on the flow rate. For both modes, the curves rise steadily with the flow rate and are steeper in dynamic flow mode than in continuous mode at the same average flow rates. This can be explained by the effect of the high peak flows on the residence time of the gas.

Table II shows the breakthrough times of the small-capacity filter in relation to flow mode at a test concentration of 300 ppm for a breakthrough concentration of 5 ppm. The values in Table III are based on data analysis taking 10 ppm as the breakthrough concentration. The breakthrough time is the period of time in which the downstream concentration of the test gas reaches a predefined concentration. The variation in breakthrough times between the different breathing modes found in data analysis using the 5 ppm breakthrough concentration is caused by uncertainty mainly due to the different initial levels of instantaneous penetration in dynamic flow mode. This effect was reduced by adopting the 10 ppm concentration. These variations were even higher with a 1000 ppm test concentration since breakthrough occurred in some cases immediately after the start of the test at higher flow rates. This is the reason why the results measured at 1000 ppm have not been represented.

Compared to the given breakthrough time of 100 minutes with a test concentration of 300 ppm, as stated by the manufacturer, the filter passed the continuous tests, albeit the occurrence of any earlier low level penetration.

Table II. Breakthrough Times t_b and Residence Times in Continuous and Dynamic Mode with a 300 ppm Cyclohexane Test Concentration for a Breakthrough Concentration of 5 ppm

Flow rate (lpm)	Residence time (sec)	Continuous mode t_b (min)	Dynamic mode t_b (min)	Capacity (l)	
				Contin.	Dyn.
30	0.10	114	70	1.0	0.6
50	0.06	55	27	0.8	0.4
60	0.05	44	22	0.8	0.4
80	0.04	24	19	0.6	0.4

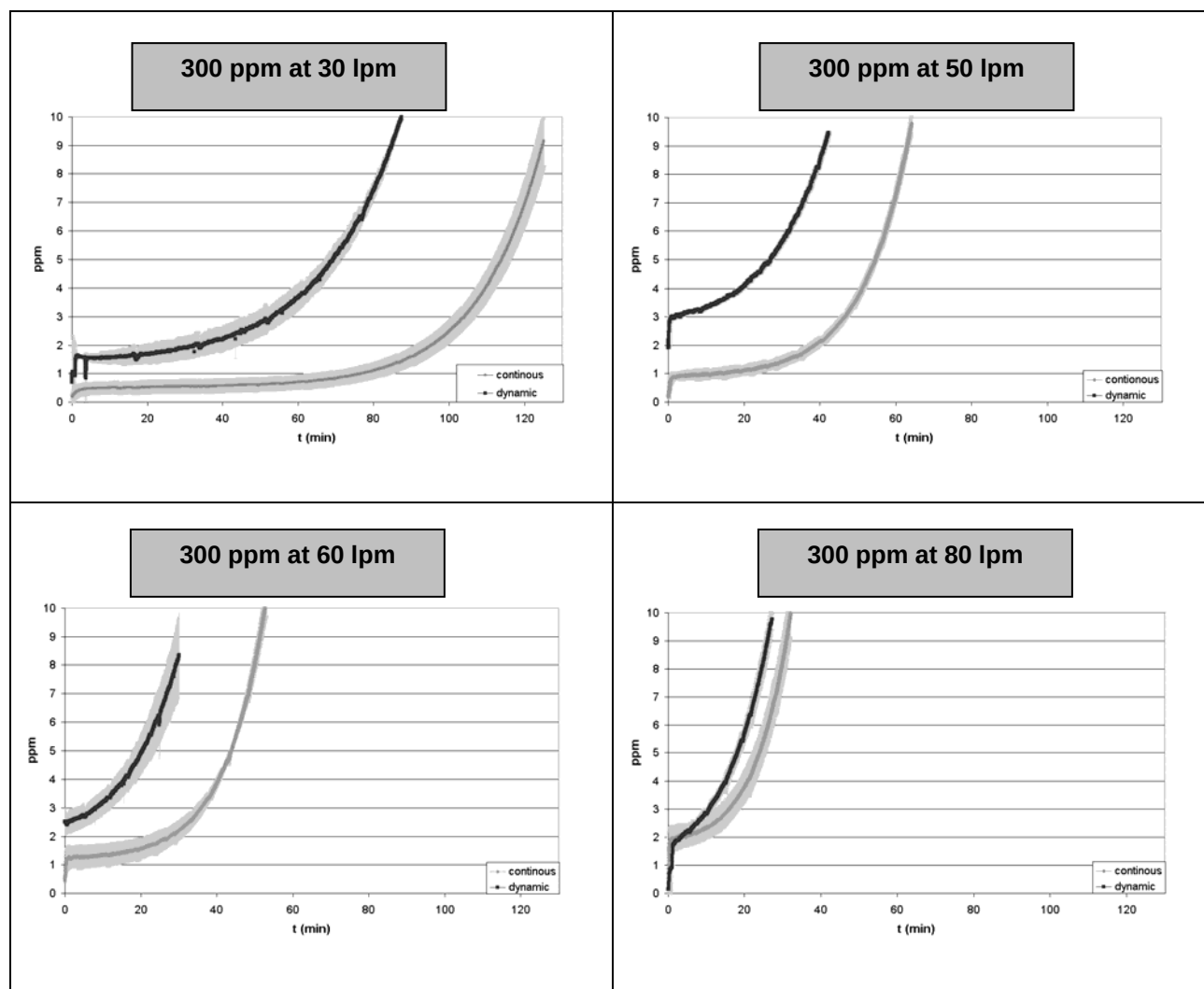


Figure 2. Breakthrough curves in continuous and dynamic mode for different flow rates at a test concentration of 300 ppm. The variation in the two measurements is indicated for each breakthrough curve with light grey bands.

Table III. Breakthrough Times t_b in Continuous and the Dynamic Mode with a 300 ppm Cyclohexane Test Concentration for a Breakthrough Concentration of 10 ppm

Flow rate (lpm)	Continuous mode t_b (min)	Dynamic mode t_b (min)	Capacity (l)	
			Contin.	Dyn.
30	127	88	1.1	0.8
50	65	44	1.0	0.7
60	53	35	1.0	0.6
80	32	28	0.8	0.7

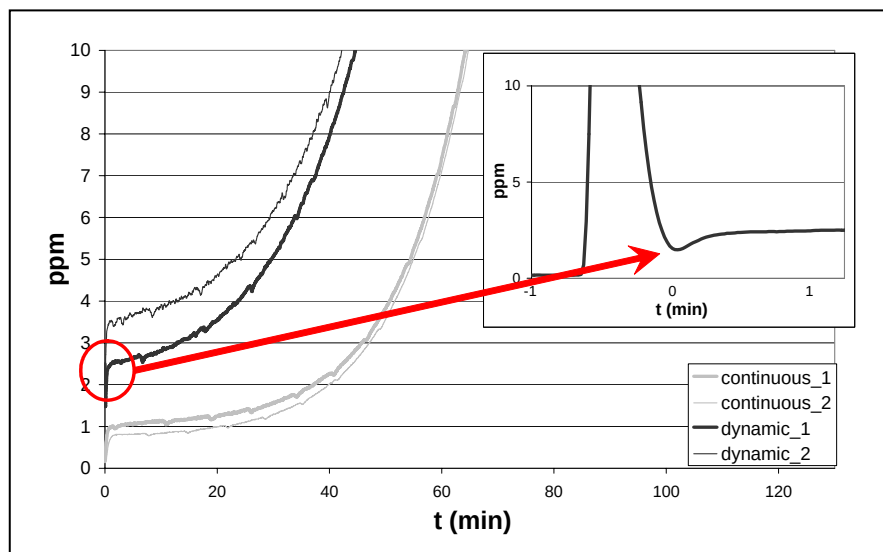


Figure 3. Measuring of instantaneous penetration. Recording starts before the filter is fixed in the test chamber (>10 ppm detected). After insertion of the filter, the signal decreases sharply and increases again rapidly due to instantaneous penetration.

It can clearly be shown (Tables II and III) that the breakthrough times measured in dynamic mode are significantly lower than those measured in continuous mode. This observation can be attributed to the shorter residence time of the gas in the filter due to the high peak flows in dynamic mode. This phenomenon has also reported by, among others, Linders et al. (2003), who investigated the adsorption of toluene on self-prepared Norit R1 activated carbon columns in dry conditions of < 5% relative humidity, 15 lpm and 1300 ppm. The authors showed the relative decrease in breakthrough time in dynamic mode compared to continuous flow as a function of bed length. For a bed depth of 0.01 m, they found a relative decrease of 59% at a breakthrough concentration of 5 mg/m³. These results concur with the present study, although the conditions are different. We found a 50% reduction with a comparable cyclohexane flow which is calculated by multiplying the test concentration with the flow rate. A clear dependence of the differences in breakthrough times on the flow rate between the two modes can be seen explicitly, even if the difference is almost negligible at the highest flow rate of 80 lpm. The fact that the calculated capacity (by multiplication of the test gas concentration with flow rate and breakthrough time) of the filter decreases with increasing flow rate for both flow modes reveals that the filter's capacity cannot be fully exploited at higher flow rates due to the decrease in residence time. The conclusion that this phenomenon is much more pronounced in continuous mode than in dynamic mode suggests a convergence of the values of the effectively useable filter capacity for the two breathing modes at the highest flow rate. This means that the effect of the difference in filter capacity between the two breathing modes seems to be weaker, the higher the flow rate. More tests will have to be carried out for confirmation of this assumption.

The small cartridges were also tested with a low continuous flow rate of 15 lpm (Figure 4). A breakthrough time of 200 minutes was obtained for a breakthrough concentration of 5 ppm. In this case, instantaneous penetration was considerably smaller compared to the results measured at flow rates above 30 lpm. Within the first minutes, a very slow increase in penetration was measured over a long period of about 100 minutes. After that, penetration increased exponentially to the breakthrough concentration of 5 ppm. This suggests that the residence time (0.2 s) in these experimental conditions is very close to critical residence time. No comparison with dynamic flow is available because the breathing machine was unable to work at such low flow rates.

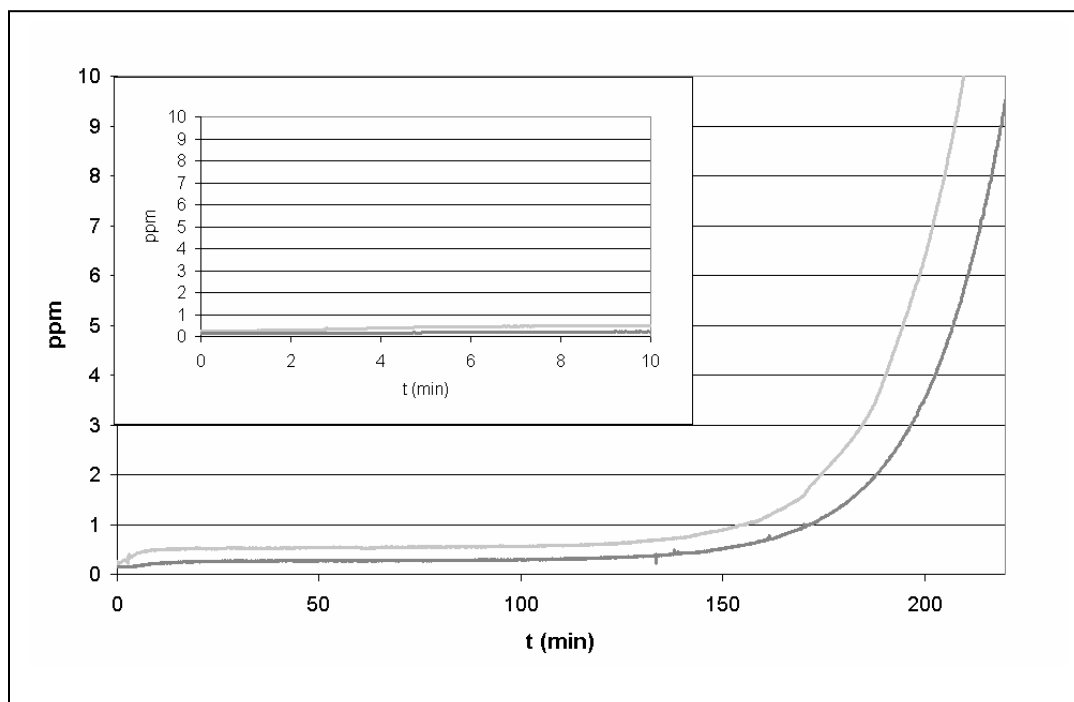


Figure 4. Breakthrough measurement of two filters at 300 ppm and 15 lpm continuous flow.

ABEK1 filter

Some capacity tests were carried out with the reference ABEK1 filter, both in dynamic flow mode and continuous mode (Figure 6 and Table IV).

For the EN standard conditions (1000 ppm, 30 lpm, 70% r.h.) for Class 1 filters conforming to EN 14387:2004, the breakthrough time of the small filter is about half of what is required for a Class 1 filter in continuous mode. The difference becomes even more pronounced with increasing dynamic flow. For example, the breakthrough time of the ABEK1 filter with a test concentration of 300 ppm and a dynamic flow rate of 80 lpm is about four times higher than that of the small-capacity filter (77 minutes for ABEK1 and 19 minutes for the small filter). The breakthrough times of the small filters are strongly affected by the flow rates due to the shallow bed depth. In addition, the effect of concentration can be seen in dynamic test conditions of 1000 ppm and 60 lpm, where immediate breakthrough was observed for the small filters (Figure 5).

The reference filter (ABEK1) has a bed depth of 2.1 cm and a diameter of 8.5 cm. For tests with 1000 ppm cyclohexane at average flow rates of 30, 50 and 60 lpm, the measured differences between the flow modes (Table IV) were lower for the reference filter compared to the small filter because of the bed depth. Filters with similar dimensions were also used by Nelsen and Harder (1972). They tested filters with similar bed depths (2.1 and 2.3 cm) by varying the flow rate from 14 to 71 lpm and could not find any difference between the flow modes. Linders et al. (2003) tested filters with different bed depths at flow modes with an average flow rate of 15 lpm by varying the concentration (1253 – 2610 ppm toluene). The differences in dynamic and continuous flow rates decrease with increasing bed depth. Because of the different test conditions (flow rate, humidity, concentration, substance), it is difficult to compare these results with the results of the present study. In most cases, only single measurements were carried out on the reference filter.

Even at high concentrations and flow rates, the instantaneous penetration manifested by the small-capacity filters in nearly all conditions could not be detected for the ABEK1 filter, as Figure 6 shows.

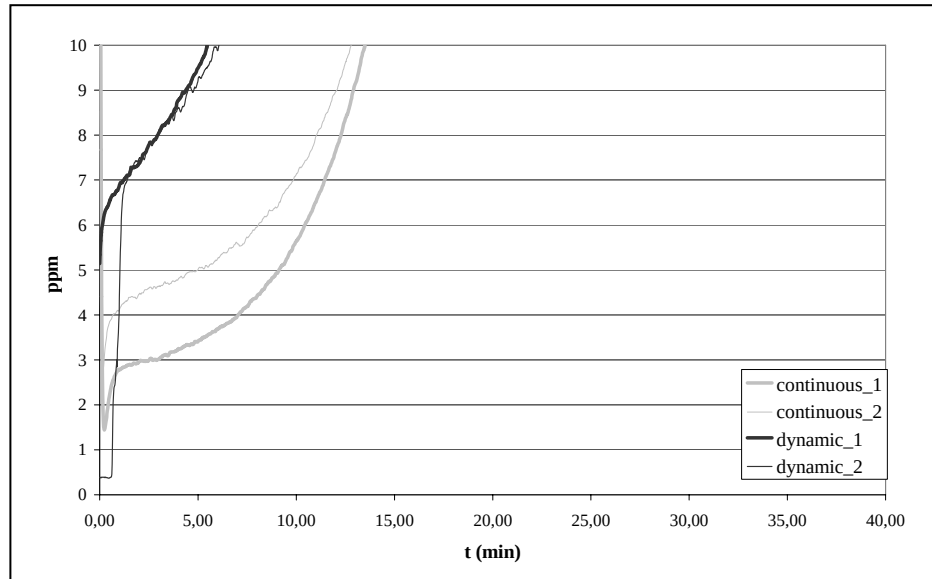


Figure 5. Breakthrough times (at 5 ppm) in continuous and dynamic mode for different flow rates (average flow 60 lpm) with a test concentration of 1000 ppm for the small filters. Two measurements for each flow mode are shown.

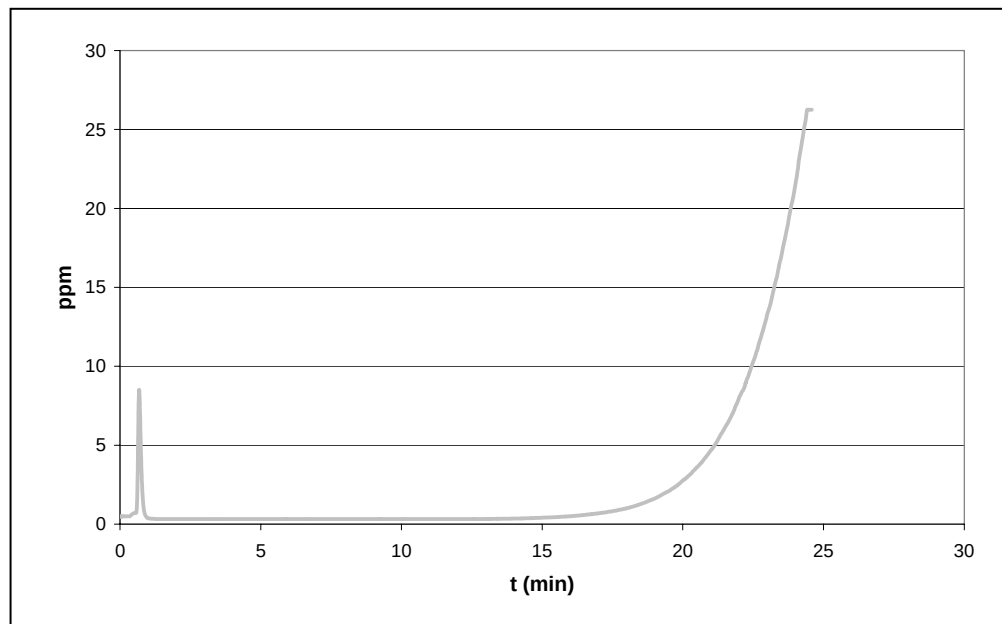


Figure 6. Breakthrough curve for ABEK1 filter at 100 lpm continuous flow with 1000 ppm cyclohexane. Recording starts before the filter is fixed in the test chamber (8 ppm detected). After insertion of the filter, the signal decreases sharply.

Table IV. Breakthrough Times and Residence Times of ABEK 1 for Continuous and Dynamic Mode with a 1000 ppm Cyclohexane Test Concentration for a Breakthrough Concentration of 5 ppm

Flow rate (lpm)	Residence time (sec)	Continuous mode b_{th} (min)	Dynamic mode b_{th} (min)	Capacity (l)	
				Contin.	Dyn.
30	0.24	102	92	3.1	2.8
50	0.14	72	53	3.6	2.7
60	0.12	58	42	3.5	2.5

CONCLUSIONS

The purpose of this investigation was to show the behaviour of absorption of small gas filter cartridges, which are smaller than the smallest class known to the European system. These were tested simulating moderate to intensive work rates with defined test concentrations of cyclohexane in continuous and dynamic test modes. Because of the shallow bed depth, the effects of early breakthrough due to critical residence time and also of potential channelling had to be considered for these small filters.

The small-capacity filters show a very early penetration of the test gas immediately after the start of the capacity test for the average flow rates of 30 – 80 lpm for both modes. That phenomenon can be attributed to the shallow bed depth of the filter in these severe test conditions. A limited effect due to a channelling mechanism could not be definitively excluded.

Low but slowly increasing penetration was detectable within the first 100 minutes even at an average continuous flow rate of 15 lpm. The small filters being tested with low continuous penetration might pass a concentration-based criterion according to existing standards, but would possibly fail a stringent dose-related criterion.

To the authors' knowledge, this penetration phenomenon has never been mentioned before in the literature. Since there is no requirement at least in European standards taking this phenomenon into account, we need to ask whether a gas filter may show such so-called instantaneous penetration at all and, if so, to what extent. All conventions concerning breakthrough concentrations in gas filter standards are technically based, but do not have any relevance to safety issues, e.g. toxicological validation. By comparison, particle filters never achieve 100% filtration. Their penetration is regarded in the context of face seal leakage of respiratory interfaces as part of the total inward leakage as a measure of the overall performance of a device providing protection from hazardous materials. This approach could also be adopted for gas filters. In the course of the recent discussion in ISO/TC 94/SC15 Respiratory Protective Devices, discussion of this issue in connection with inward leakage has already been recommended.

Another effect observed is the strong dependence of flow mode and flow rate on breakthrough time. Higher flow rates used for simulating breathing at higher work rate levels increase the level of instantaneous penetration and may therefore impair a device's performance. In addition, a clear dependence of residence time on flow mode has been shown to reduce breakthrough time. All these parameters together have to be studied in greater depth for other filter types and their respective test gases so that appropriate requirements can be set in the standardisation process.

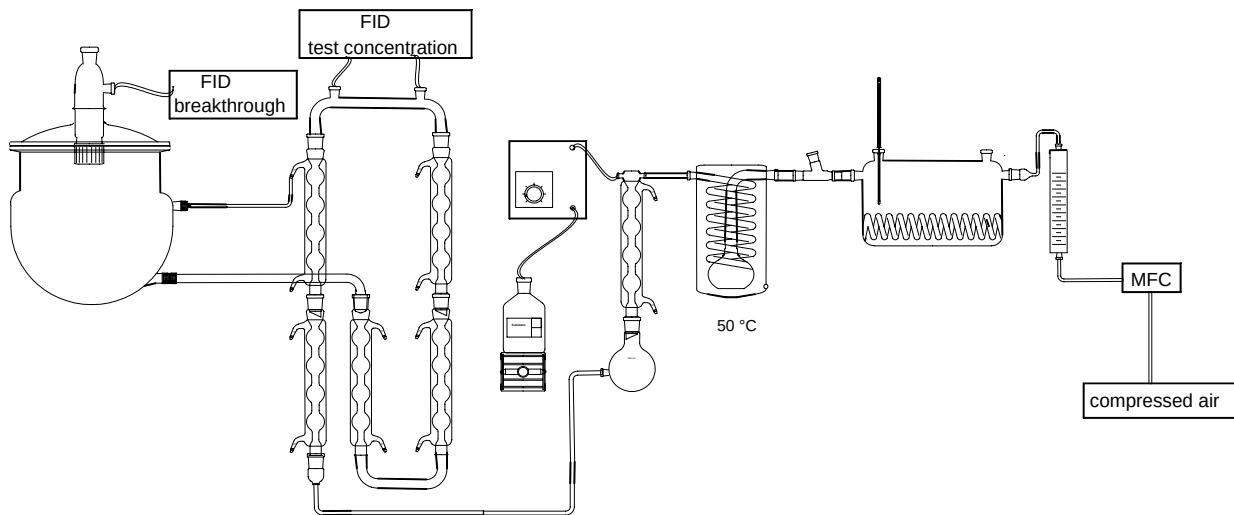
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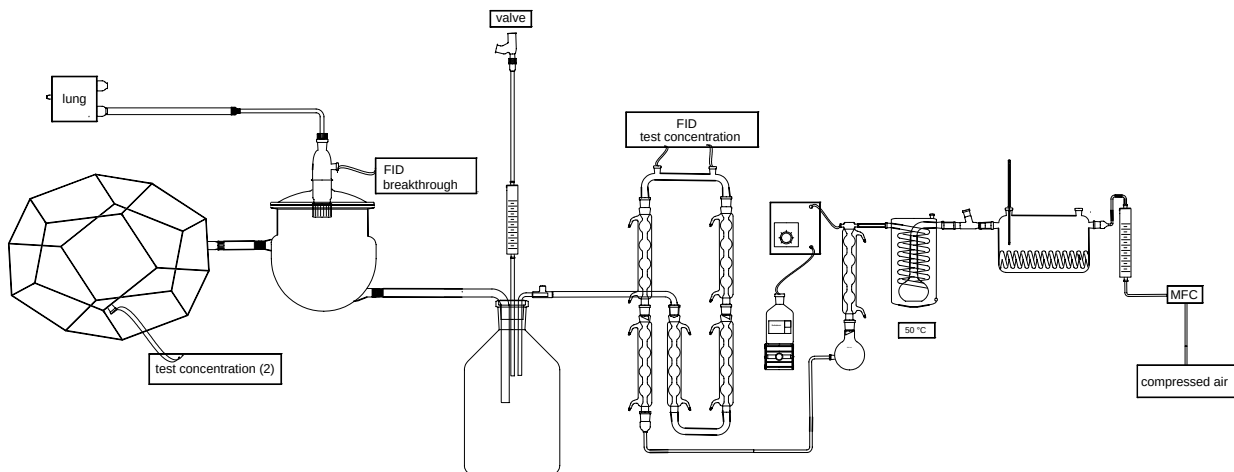
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Annex



Schematic of the experimental set-up for continuous measurement



Schematic of the experimental set-up for dynamic measurement

Work rate classification and correlation with the minute volume, peak flow rate and work description

Work rate class	Metabolic rate (W/m ²)	Minute volume (lpm)	Peak flow rate (lps)	Example
1 (Resting)	65	11	0.84	Resting
2 (Low metabolic rate)	100	16	1.20	Writing, driving a vehicle in normal conditions
3 (Moderate metabolic rate)	165	27	1.82	Plastering, driving lorries, walking at 3.5 – 5.5 km/h
4 (High metabolic rate)	230	38	2.39	Hard work like sawing, walking at 5.5 -7 km/h
5 (Very high metabolic rate)	290	48	2.90	Walking briskly with short steps, using an axe, walking at > 7 km/h
6 (Extremely high metabolic rate)	400	66	3.80	Running, 50 – 60% of maximum aerobic capacity
7 (Extreme work)	475	78	4.38	Rescue and fire-fighting assignments with high physical strain, 70-80% of maximum aerobic capacity
8 (Exhausting work)	600	99	5.32	Rescue and fire-fighting assignments working flat out