

# Scientific & Technical Report

# A Comparison of Evaluative Test Methods for Cleanable Filter Elements Used in Polymer Applications

Doug Elliott, Pall Corporation Mike Whitlock, Pall Corporation

June 30, 2010

#### Objective

The goal of this paper is to compare test data from non-destructive, evaluative test methods such as 1<sup>st</sup> bubble point determination, 10 LPM wetted air flow testing, and bypass flow testing with efficiency test results for a given set of cleanable filter elements (p/n: D116205-200-1407-LOP). Such a comparison presents scientific evidence to support the hypothesis that the bypass flow test method is a more sensitive tool for predicting the removal efficiency of this group of filter elements.

#### Summary

Filtration is applied to many applications in the polymer industry to improve the quality of the final product. These filtration applications often utilize cleanable filter elements such as candles, leaf discs, and segments to achieve this improved quality. Non-destructive tests are commonly used to evaluate the integrity of a filter element. The results are then compared to predetermined standards for each element. The 1<sup>st</sup> bubble point and 10 LPM test methods have evolved as quick methods to determine this integrity. Filter elements are normally tested by one or both of these methods after manufacturing (i.e., prior to the first use) and after subsequent cleanings.

In order to effectively evaluate the bypass flow method, a group of 13.97 cm (5.5 in) Ultipleat® Polymer Candle elements having a range of 1st bubble points both above and below the minimum specification were selected, cleaned, and evaluated for 1st bubble point, 10 LPM, and bypass flow. The filter elements were measured for baseline removal efficiency testing using a modified F-2 test and again cleaned. The "modified" F-2 test used throughout this study was a single pass oil F-2 test using a very low (10 mg/liter or 10 ppm) particle challenge and a limited differential pressure (DP) rise of 1.03 bard (15 psid) to minimize the effect of cake filtration. The elements were then subjected to a simulated use cycle by challenging them with a test mixture of polyethylene terephthalate, carbon powder, polyethylene gel powder, and copper powder. All filters were challenged to a terminal DP of 128 bard (1,850 psid). After cleaning, the elements were retested using the 1<sup>st</sup> bubble point,

10 LPM, and bypass flow procedures. The filter elements were reevaluated for F-2 efficiency and cleaned for a final time. When returned from the cleaning vendor, the filter elements underwent a final round of 1<sup>st</sup> bubble point, 10 LPM, and bypass flow testing.

Analysis of the test results revealed several interesting trends. First, the changes in the 10 LPM results were not found to vary significantly with changes in the F-2 efficiency of the filter elements. Changes in 1<sup>st</sup> bubble point results for the filter elements did not demonstrate a clear relationship when compared to the F-2 efficiency. As the minimum 1<sup>st</sup> bubble point for these filters was 5.0" wc, the test data also indicated that even though several elements were below this minimum value, the F-2 removal efficiencies remained high. Finally, bypass flow test results were found to vary with a reasonably linear relationship to even small changes in the F-2 efficiency over this same range.

For the group of elements used in this study, the results demonstrate that the bypass flow test was a sensitive and reliable tool for prediction of F-2 removal efficiency. Utilization of the bypass flow test over these test methods may provide the end-user with another accurate tool to evaluate cleanable filter elements and may prevent the premature discarding of a polymer filter element that can provide acceptable removal efficiency. The bypass flow methods and test pressures described in this paper should be evaluated for each media and filter design that differs from the group of filters evaluated in this study.

#### Background

Filtration is applied to many applications in the polymer industry to improve the quality of the final product. These polymer filtration applications often utilize cleanable filter elements such as candles, leaf discs, and segments to achieve this improved quality. Non-destructive test methods are used to evaluate the integrity of the filter element, and the results are then compared to predetermined standards. The 1<sup>st</sup> bubble point and 10 LPM test methods are two procedures that are commonly used to determine this integrity. Filter elements are normally tested by one or both of these methods after manufacturing (i.e., prior to the first use) and after subsequent cleanings.

The on-stream life of cleanable filter elements is governed by the flow resistance which increases as contaminants build upon and within the filter elements. Filter elements are removed from service for appropriate cleaning when the flow resistance reaches a value that is limited either by maximum differential pressure across the filter assembly or by minimum process flow rate.

The cleaning of elements used in polymer applications is typically carried out at a qualified cleaning vendor or in some cases in-house at the production plant. The cleaning process consists of one or more of the following cleaning methods: cleaning with abrasives or solvents, chemical treatment (with acid or caustic) followed by neutralization, molten salt baths, pyrolysis, and ultra-sonication. The selected cleaning steps are based upon the type of filter element to be cleaned, the type of contaminants to be removed, as well as cost and lead time.

Post-cleaning testing (identical to the methods used for new filters) is performed to evaluate the effectiveness of the cleaning process and to ensure that the physical integrity and removal efficiency of the filter medium in the filter elements remains within specification. The physical integrity of a filter indicates whether there are significant breaches in or general degradation of the filter medium, while removal efficiency refers to the ability of a filter element to remove particles of a particular size range. The continual monitoring of 1<sup>st</sup> bubble point and 10 LPM values after each use and subsequent cleanings provides a comparison basis for the condition of the elements.

The 1st bubble point test method and the F-2 efficiency test method are described in the paper "Validation of Integrity Test Values for Cleanable Porous Stainless Steel Polymer Filters" by Dr. Tore Lindstrom of Pall Corporation's Application Engineering Department (see Reference 1). In particular this paper references the Aerospace Recommended Practice (ARP) 901 procedure for the determination of 1st bubble point and demonstrates how to determine the minimum bubble point below which a desired F-2 removal efficiency cannot be achieved (Reference 2). First bubble point testing is used to detect damage or larger-than-typical pores in the filter medium or assembly. Filter elements are normally tested by this method after manufacturing (i.e., before the first use) and after subsequent cleanings. The continual monitoring of this parameter after each use and subsequent cleaning can provide a comparison basis for the condition of the elements.

One limitation of the 1<sup>st</sup> bubble point method is that it cannot quantify the number of leaks that are present in the filter element. Figure 1 illustrates this phenomenon. The dilemma that accompanies this limitation is whether a filter should be removed from service based upon 1<sup>st</sup> bubble point alone. In this example, both filters have the same 1<sup>st</sup> bubble point, indicating that either filter might be removed from service. However, the filter with the single large pore "leak" would most likely have better efficiency than the filter having many leaks, since the probability of a particle smaller than a pore identified as a "leakage" pore to pass through would be lower in proportion to the number of "leaks" found in the element. Another limitation is that 1st bubble point and 10 LPM limits are often arbitrarily set based upon the typical lower boundaries of test values on new elements (what can be achieved with acceptable manufacturing

yields) and are not calculated based upon a minimum desired efficiency as described in Reference 1. However, field experience (i.e., acceptable product quality over time) is considered the validation of most current 1<sup>st</sup> bubble point specifications.





Another test employed to evaluate the integrity of cleaned candle filters is the 10 LPM wetted air flow test method. In this procedure a measured flow of ten standard liters per minute of air is passed through a filter element that has been wetted in a test fluid (typically isopropyl alcohol (IPA) or denatured ethanol). The maximum pressure across the filter is measured as the surface tension of the fluid in some of the pores is overcome. This value provides a rough indication of average pore size; however, as no account of the filter area is considered, this value can only provide a baseline for future testing.

Finally, an alternative evaluative test to the 1<sup>st</sup> bubble point or 10 LPM test methods is bypass flow determination. Bypass flow is determined by measuring the collective flow from all pores and defects that are larger than the nominal pore size of the filter medium. Before bypass flow is calculated, a determination of the test pressure

and maximum bypass flow/unit area is made. The bypass test pressure corresponds to the bubble point of a pore that is no longer considered to possess any capture ability by any mechanism. A bypass flow test pressure equal to 60% of the pressure at which 0.3 m/min (1 ft/min) velocity of air flows through a wetted membrane has been determined empirically to be a good approximation of this pore size. Figure 2 illustrates flow through a porous medium showing a "filtering" pore and a "bypass pore." The bypass flow procedure requires that the flat sheet medium absolute F-2 efficiency with no defects (i.e., bypass flow=0) also be known. This is the medium efficiency prior to any forming or fabrication steps. The maximum bypass flow / unit area corresponding to a lower bound on F-2 efficiency can then be calculated for any element utilizing the same medium. The test procedure for the determination of bypass flow is described in Appendix A.

Figure 2:

Model of a "Filtering" Pore and a "Bypass" Pore



#### **Methods and Procedures**

#### 1<sup>st</sup> Bubble Point Test

Determination of the 1st bubble point is based upon the fact that, for a given fluid and pore size with constant wetting, the pressure required to force an air bubble through the pore opening is inversely proportional to the size of the pore. In this test an approximation of the largest pore (or defect) in a filter or similar permeable membrane structure can be determined by completely wetting the filter with a fluid (typically isopropyl alcohol or denatured ethanol) and measuring the pressure at which the first bubble is emitted from the filter surface after air is introduced into the filter interior. Reference 2 is a formal recognized procedure for this testing. For the filters used in this study, the minimum acceptable first bubble point specification is 12.7 cm (5.0 in) of water.

#### 10 LPM Test

The 10 LPM wetted air flow test also involves the use of the bubble point test stand that is referenced in the 1<sup>st</sup> bubble point test. In this method, the filter element is inspected for cleanliness and any gross contamination or defects prior to testing. The element is totally immersed with a fluid (usually isopropyl alcohol). The filter element is then drained and a stopper is inserted so that an attachment to an air line can be made.

The filter element is then immersed into the test tank to 1.27 cm (0.5 in) below the surface. The gas flow is then set to a rate of 10 lpm (0.35 cfm). The pressure drop is then read to the nearest .25 cm (0.1 in) of water. For the filters used in this study, the minimum acceptable 10 LPM pressure drop specification is 30% below the new element baseline value (i.e., there is no "minimum" specification for a new element). Thus, the 10 LPM test is a qualitative comparison that is primarily used to track changes over multiple cleaning cycles.

#### **Bypass Flow Test**

The test stand for this procedure has a set of very accurate flow meters with minimal restriction for accurate measuring of the air flow through the element. The bypass flow test begins with the determination of the bypass flow test pressure. This value equals 60% of the pressure at which 0.3 m/min (1 ft/min) velocity of air flows through a wetted membrane. This value corresponds to the average pore size in the medium. The filter element to be tested is placed in the tank and allowed to thoroughly wet the medium. The element is then removed carefully from the tank where the excess fluid is poured from the element. A pressure source is then connected to the end of the element, and the element is again immersed. Pressure is applied slowly until the predetermined test pressure is reached. The airflow rate is monitored until stabilization occurs. The element is then lifted from the tank and a reading of the air flow (to the nearest ml/min) is made as soon as the pressure stabilizes. For the filters used in this study, the maximum acceptable bypass flow specification is 705 ml/min (0.025 cfm). (See Appendix A)

#### **Filter Evaluation Procedure**

In order to effectively evaluate the bypass flow method and compare the results to other test values, the following steps were taken:

- First, a group of 13.97 cm (5.5 in) Ultipleat Polymer Candle elements was fabricated. These elements are rated at 20 microns nominal, but for bypass flow calculations the absolute efficiency is needed. The absolute efficiency for this media is approximately 40 microns, so all F-2 efficiencies were evaluated at this particle size.
- After fabrication, the elements were sent to an outside cleaning vendor for thorough cleaning. The elements were then baseline tested for the bubble point spectrum (1<sup>st</sup> through 10<sup>th</sup>) and 10 LPM testing.
- After the elements were returned to Pall's R&D laboratory in Cortland, NY, the elements were again baseline tested by the same procedures as the cleaning vendor, along with the addition of the bypass flow test. (See Appendix A for how this value is determined)
- In order to have a set of elements with a range of bubble point values, leaks were purposely created with a needle in some filters to show the effect of a few large leaks on F-2 efficiency.
- The elements were then measured for baseline removal efficiency testing (F-2 testing) at Pall's hydraulic laboratory.

- After F-2 testing, the elements were then returned to the outside vendor for cleaning, followed by testing for the bubble point spectrum (1<sup>st</sup> through 10<sup>th</sup>),10 LPM testing, and bypass flow testing.
- Upon return the elements were then subjected to a simulated use cycle by challenging them with a test mixture of polyethylene terephthalate (PET), carbon powder, polyethylene gel powder, and copper powder. The trials were run on a 2.54 cm (1 in) extruder at Pall's Filter Performance Laboratory in Cortland, NY. All filter elements were challenged to a terminal DP of 128 bard (1,850 psid) across the element.
- After all of the polymer testing was completed, the elements were again returned to the outside vendor where the elements were cleaned.
- The returned elements were retested by bubble point, 10 LPM, and bypass flow test procedures.
- The elements were then evaluated for F-2 efficiency once again in the hydraulic laboratory, after which the elements were returned for final cleaning.
- After the final cleaning the elements were again tested for bubble point spectrum (1<sup>st</sup> through 10<sup>th</sup>), 10 LPM testing, as well as bypass flow determination. This cleaning did not have an effect on the test results and was used to demonstrate the reproducibility of the integrity test data after the final element cleaning. (See Appendix B for how the bypass flow test efficiency prediction was calculated)

The 1<sup>st</sup> bubble point, 10 LPM, and bypass flow test results for the given set of filter elements were plotted against removal efficiency. A graph of the results is shown in Figure 3. The graph highlights the results of the 1<sup>st</sup> bubble point test (shown in blue), the 10 LPM test results (shown in green), and the bypass flow test results (shown in red) as tested just prior to the final F-2 efficiency testing.

The graph of the testing results shows the following data trends:

- The 10 LPM results did not vary significantly over the F-2 efficiency range.
- The 1<sup>st</sup> bubble point test values for the filter elements did not demonstrate a clear relationship when compared to the F-2 efficiency over the same efficiency range. As the minimum 1<sup>st</sup> bubble point for the medium used in this group of filters was 5.0°wc, the test data indicated that even though several elements were well below this minimum value, the F-2 removal efficiencies of these elements remained high.
- Finally, the graph demonstrates that changes in bypass flow results were found to vary with a reasonably linear relationship to even small changes in the F-2 efficiency over the same range.



#### Figure 3: Evaluative Test Results Versus 40 Micron Efficiency

Analysis of the test results revealed the following conclusions:

- 1. The changes in the 10 LPM results did not vary significantly with changes in the F-2 efficiency of the filter elements that were evaluated.
- 2. Changes in 1<sup>st</sup> bubble point results for the filter elements did not demonstrate a clear relationship when compared to the F-2 efficiency. As the minimum 1<sup>st</sup> bubble point for these filters was 5.0"wc, the test data also indicated that even though several elements were below this minimum value, the F-2 removal efficiencies remained high.
- 3. The bypass flow test results were found to vary with a reasonably linear relationship to even small changes in the F-2 efficiency over the same range.

The results demonstrate that for the elements evaluated in this study the bypass flow test was a more sensitive and reliable tool for non-destructive filter evaluation than either the 1<sup>st</sup> bubble point or the 10 LPM airflow tests. Utilization of the bypass flow test over the 1<sup>st</sup> bubble point and 10 LPM methods may provide the end-user with a more sensitive tool to evaluate cleanable filter elements and prevent the premature discarding of a polymer filter element that can provide acceptable removal efficiency. The bypass flow methods and test pressures described in this paper should be evaluated for each media and filter design that differs from the group of filters used in this study.

#### References

(1) Lindstrom, Tore, "Validation of Integrity Test Values for Cleanable Porous Stainless Steel Polymer Filters", Presented at the American Filtration Society Annual Meeting, March 18-22, 1990, Arlington, VA, Appendix I, pages 5-9.

(2) Aerospace Recommended Practice, ARP 901, "Bubble Point Test Method", Society of Automotive Engineers, Inc., 1998.

#### APPENDIX A

#### **Bypass Flow Determination for Polymer Filter Elements**

#### Background

The bypass flow test measures any flow through pores at or above a diameter identified as total bypass (no particle capture, i.e. leaks). The test pressure corresponding to this pore diameter (using the relationship defined in Reference 2) is determined empirically and is based on 60% of the pressure at which 0.3 m/min (1 ft/min) velocity of air flows through a wetted membrane. Since 100% of this pressure is a measure of the average pore diameter, the bypass flow pressure must equal some amount below this value. The 60% value was chosen based on previous bypass flow studies that showed increasing accuracy of predicted versus measured efficiency at test pressures up to approximately 60% of the 0.3 m/min (1 ft/min) value.

If the percentage of total flow through the bypass pores is known, the calculations can be made based upon the relationship of pressure drop to flow through an orifice (pore). This results in a percentage of total flow through the element that is not being filtered. This value can be used along with parameters defined in the F-2 particle challenge test to predict the impact of the leaks on the filtration efficiency of the element. Using this methodology it was shown that the filter elements in this study met a predetermined minimum F-2 efficiency if the bypass flow did not exceed a certain flow rate, even if the bubble point is much lower than the specified "failure" value. For purposes of this study, the minimum F-2 efficiency was set at 99.5%.

#### **Test Procedure**

A test stand set-up similar to that shown in Figure 4 is required and is somewhat similar to that of a bubble point test stand. The main difference is the addition of a set of low-flow precision flow meters that are placed in-line upstream of the filter to measure the flow of air emerging from the leaks. The use of a range of flow meters is required to maintain sufficient accuracy in the lower air flow ranges.

s 1 MICRON FILTERED DRY, OL - FREE AIR 0-20 PSIG REG. 0-2 NUMPTER COMMETTER COMM

#### Figure 4:

Schematic of Bypass Flow Test Stand with a Candle Element The bypass flow test procedure is performed as follows:

- The filter element is first immersed in clean isopropyl alcohol (filtered to ≤ 1.0 micron) to thoroughly wet the medium.
- Then the filter element is removed from the filtered IPA and allowed to drain until a steady stream of alcohol no longer runs from the element.
- The element is then connected to the pressure source and reimmersed in the IPA.
- After immersing the element, the 0-0.14 bard (0-2 psig) regulator is used to slowly apply pressure until the required test pressure is reached.
- Sufficient time (typically 15-30 seconds) is then allowed for the flow reading on the appropriate flow meter to stabilize.

- After the waiting period, the element is carefully lifted from the tank (free of the IPA) and the reading in ml/min is recorded by the appropriate filter serial # as soon as the pressure stabilizes.
- The bypass flow result is then compared to the predetermined maximum bypass flow rate, and the element is accepted if it is equal to or less than the maximum value.

It is important that the connection for the manometer be placed as close as possible to the filter element and that the test lines are sufficiently large so that inaccurate test pressures due to pressure losses do not occur.

### APPENDIX B Maximum Bypass Flow Efficiency Determination

The computation of estimated efficiency with bypass flow requires a three-step calculation. Examples of the calculation steps are listed below.

# a) Calculation of Bypass Flow Ratio (alpha)

First, the bypass flow ratio or alpha must be calculated. This value corresponds to the ratio of total flow through the element at efficiency test conditions.

$Q_{bp}$ ' = $Q_{bp}$ *	14.7 ((PT*.036) + 14.7)	Where	$Q_{bp}$	=	Bypass flow at standard conditions (SCFM)
			Q <sub>bp</sub> '	=	Bypass flow at bypass flow test pressure (ACFM)
			РТ	=	Bypass flow test pressure
Qbp" = Qbp' * 7.48	$3 * \frac{DP}{(PT^*.036)} * \frac{\mu_t}{\mu_{et}}$		Q <sub>bp</sub> "	=	Bypass flow at efficiency test conditions (ACFM)
			$Q_{\text{T}}$	=	Total flow through element (ACFM) at efficiency test conditions
			$\mu_t$	=	Bypass flow test fluid viscosity
			$\mu_{et}$	=	F-2 efficiency test fluid viscosity

Therefore the bypass flow ratio or alpha =  $Q_{bp}$ "/ $Q_T$ 

## b) Calculation of Fractional Penetration with Bypass (FPd')

The second portion of the bypass flow calculation involves the flat sheet penetration of particles of size d ( $FP_d$ ) that have been modified to account for bypass.

$FP_{d}' = (alpha * (1 - FP_{d})) + FP_{d}$	$FP_d$	=	Flat sheet fractional penetration
			of particle size d
		=	1/β
	β	=	# upstream "d" diameter particles
			# downstream "d" diameter particles

## c) Calculation of Predicted Efficiency with Bypass

The final portion involves the calculation of predicted particle removal efficiency based upon the bypass flow measurement.

Predicted efficiency =  $100 - (FP_d, *100)$ 

The **maximum** bypass flow is the flow corresponding to the lowest acceptable predicted efficiency with bypass. For cleaned elements this value is chosen by the end-user, but for new elements Pall Corporation uses a minimum predicted F-2 efficiency with bypass of 99.5%

Sample calculations using the bypass flow test results and the F-2 removal efficiency results are shown in Table 1 below.

Table 1:   Bypass Flow Test   Efficiency   Prediction	F-2 test flux rate Flat sheet fract. pen.@ Qt	= Qt = = Fp =	24 0.000034	(lpm/ft²)		
	Bypass flow test pressure	= Pt =	0.184	(psi)		
	F-2 test pressure drop	= DP =	15	(psid)	(Time averaged DP)	
	F-2 test fluid viscosity	= mu =	14.8	(cp)		
	Test Element area	= A =	0.6	(sq.ft.)		

	(ml/min)	(ml/min)	(ml/min)	(lpm)			Predicted	Actual Efficiency	
Serial #	Qbpi	Qbp	Qbp'	Qbp''	Alpha	Fp*	F-2 Efficiency	@ 40 micron	% difference
zero bypass (serial #4)	0	0.0	0.0	0.0000	0.00000	0.000034	100.00	100.00	0.000
14	185	182.4	187.7	0.0186	0.00129	0.001326	99.87	99.81	0.062
2	16.2	16.0	16.4	0.0016	0.00011	0.000147	99.99	100.00	-0.011
6	112	110.4	113.6	0.0113	0.00078	0.000816	99.92	99.95	-0.031
8	26.2	25.8	26.6	0.0026	0.00018	0.000217	99.98	99.99	-0.013
9	6.84	6.7	6.9	0.0007	0.00005	0.000082	99.99	99.99	0.002
10	62.62	61.7	63.5	0.0063	0.00044	0.000471	99.95	99.99	-0.034
11	411	405.1	416.9	0.0413	0.00287	0.002905	99.71	99.65	0.061
12	93.9	92.6	95.3	0.0094	0.00066	0.000690	99.93	99.96	-0.029
maximum	705	695.0	715.2	0.0709	0.00492	0.004958	99.50		



25 Harbor Park Drive Port Washington, NY 11050 +1 516 484 3600 telephone +1 800 289 7255 toll free US

Portsmouth - UK +44 (0)23 9230 3303 telephone +44 (0)23 9230 2507 fax processuk@pall.com

#### Visit us on the Web at www.pall.com.

Pall Corporation has offices and plants throughout the world. For Pall representatives in your area, please go to www.pall.com/contact

Because of technological developments related to the products, systems, and/or services described herein, the data and procedures are subject to change without notice. Please consult your Pall representative or visit www.pall.com to verify that this information remains valid. Products in this document may be covered by one or more of the following patent numbers: EP 0 667 800; EP 0 982 061; EP 1 380 331; US 5,543,047; US 5,690,765; US 5,725,784; US 6,113,784; US 7,083,564; US 7,318,800.

© Copyright 2010, Pall Corporation. Pall, (A. ), and Uthipleat are trademarks of Pall Corporation. ® Indicates a trademark registered in the USA. *Filtration. Separation. Solution.sw* is a service mark of Pall Corporation.

FCPOLYTESTEN