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AN ANALYSIS OF WEIGHT CHANGE IN FILTERS

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14. ABSTRACT
Due to recent repeatability problems with the gravimetric analysis procedure used for military conformance testing of military specification hydraulic fluids, an in-depth investigation was undertaken to evaluate possible sources of error. Many sources of weight change exist, including particulate and water accumulation from air, buildup of static electricity causing interference with the metal microbalance pan, and removal of extractables from filters during testing. Various procedures and filter types were investigated. Results show that the current testing procedures have several key shortcomings, and that the filters allowed for use in military conformance testing are incapable of delivering the required accuracy. By combining the application of slightly modified existing procedures with different filter materials, the required accuracy was achieved.

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An Analysis of Weight Change in Filters

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Abstract

Due to recent repeatability problems with the gravimetric analysis procedure used for military conformance testing of military specification hydraulic fluids, an in-depth investigation was undertaken to evaluate possible sources of error. Many sources of weight change exist, including particulate and water accumulation from air, buildup of static electricity causing interference with the metal microbalance pan, and removal of extractables from filters during testing. Various procedures and filter types were investigated. Results show that the current testing procedures have several key shortcomings, and that the filters allowed for use in military conformance testing are incapable of delivering the required accuracy. By combining the application of slightly modified existing procedures with different filter materials, the required accuracy was achieved.

Introduction

Gravimetric analysis is used to detect and measure the presence of insoluble particulate contaminants in hydraulic fluid. This is essential for the performance and longevity of equipment because particulate contaminants can cause filter failure and damage other components of the hydraulic system, resulting in excessive wear and even system failure. For this reason, accuracy is of the utmost importance.

United States military hydraulic fluids must pass conformance tests on each batch purchased, as described in the military specifications (MIL-PRF-5606, MIL-PRF-83282, MIL-PRF-87257). Fluids exceeding the specified gravimetric increase in weight, after a fluid is filtered, are rejected.

Because of discrepancies in experimental results using the military's currently prescribed methodology for gravimetric analysis of hydraulic fluids, a more reliable, accurate, and reproducible method is needed to ensure proper acceptance of hydraulic fluids. Based on this work MIL-PRF-5606 and MIL-PRF-87257 specifications have been changed to require two stacked polypropylene filters. In this investigation, a step-by-step approach was taken to isolate possible sources of error and develop alternate methods to eliminate them. The response of filters to various environmental conditions and solvent interaction has been examined in detail.

Experiments were devised to better understand the lack of repeatability of the method. This would allow the military hydraulic fluid specifications to require specific practices so that various fluid suppliers and the military laboratories can improve reproducibility. The primary purpose of these experiments was to develop accurate, precise methods for gravimetric analysis of hydraulic fluids. Therefore, great care was taken to ensure that outside sources of contamination were avoided throughout the procedure.

During the investigation in Part I of the effects of various treatments on filter weight changes in the gravimetric analysis procedure, the filter weights were changing as time passed, even when not subjected to testing. The causes of these independent weight changes were investigated.

The unexpected weight changes were first noticed when filters had been placed in a desiccator for 24 hours prior to testing. When the filters were weighed after the desiccation period, all had gained weight. Since the Petri dishes containing the filters had their lids ajar during desiccation, as prescribed in ASTM D4898, there was one obvious possibility for the observed weight gain: the accumulation of particulate matter from the air on the filters. Experiments were devised to test the effects of this particulate accumulation. It was determined that the position of the Petri dish lid did indeed affect the weight change of the filter, but that there was another cause that had a much greater impact. Since some of the filter types being examined were hydrophilic (readily absorbing water from the environment), it was hypothesized that the additional weight gain was caused by increases in relative humidity of the laboratory. When laboratory relative humidity data was compared to the weight changes in filters, a rough trend of filter weight increasing with relative humidity was observed. Experiments were then devised to test whether placing the filters in a desiccator had any benefits at all. Detailed analyses of procedures and data follow.

Similarly, problems were encountered in weighing filters of all types prior to the acquisition of an air ionizing unit. Ionization of filters prior to each weighing removes static charges that may build up in filters during treatments and storage. This static charge did interact with the balance pan during weighing and gave inaccurate weights. Data and observations are presented to reinforce the necessity of such equipment for accurate gravimetric analysis testing.

In Part II, several filter types were treated in various ways, both before and after solvent filtration. Table 1 details the filters tested in this investigation. Mixed cellulose ester (MCE), polytetrafluoroethylene (PTFE), and polypropylene filters were examined. If the weight change resulting from these treatments was significant, the filter type was unsuitable for testing purposes. Reagent grade hexane was filtered through the type of filter to be used and was used for all tests. Note that hexane was used in these experiments because it is specifically called for in ASTM-D4898 as the solvent to be used to wash the residual hydraulic fluid from the filter after the sample of hydraulic fluid has been filtered. Therefore, the compatibility of the filter media with hexane is very critical to obtaining accurate results.

Designation	Filter	Vendor	Pore Size (μm)	Composition
Filter A	MF-Millipore	Millipore	0.45	MCE
Filter B	GN6-Metricel	Pall-Gelman	0.45	MCE
Filter C	FHUP-Fluoropore	Millipore	0.50	PTFE
-	FHLP-Fluoropore	Millipore	0.50	PTFE/polyethylene
Filter D	GH-Polypro	Pall-Gelman	0.45	polypropylene

Table 1: Details of filters used in this investigation.

Part I: Environmental Effects

Experimental

Ionization

To evaluate the necessity of an air ionization unit, the prescribed procedure for gravimetric analysis of hydraulic fluids (ASTM D4898) was performed with numerous polytetrafluoroethylene (PTFE) filters. The method was modified to test the filter using only filtered hexane as a test fluid. Some filters were ionized using the StaticMaster Ionizing Unit, while others were not. Ionization was performed by passing the filter between the parallel plates of the ionizer for approximately ten seconds. These filters were subjected to a variety of treatments, both before and after the filtration of solvent. See Tables 2 and 3.

Lid Position

To evaluate what, if any, the position of the Petri dish lid has on the weight change of filters during treatments, hydrophilic mixed cellulose ester (MCE) membrane filters were ionized, weighed, and placed in a numbered Petri dish. Four of these dishes were placed in a desiccator with lids ajar, as currently called for in prescribed procedures for gravimetric analysis of hydraulic fluids (ASTM D4898). Four more dishes were placed in a desiccator with the lid completely removed. Finally, eight more dishes had the lids completely covering the dish. Half of these eight were placed in a desiccator, while the other half were placed on the bench next to the desiccator as a control. All filters were ionized and weighed at 24, 96, 168, 192, and 220 hours after original weighing. Samples in the desiccator were removed and quickly weighed to avoid possible absorption of water, then were replaced in the desiccator as quickly as possible. All Petri dishes used were 50 mm diameter plastic dishes. This contrasts with the 150 mm glass dishes called for in the ASTM method. However, for the quantity of evaluations to be performed, cleaning large glass dishes between each was impractical. This variation was not expected to affect the results.

When it was observed that relative humidity had a large effect on filter weight changes, a new procedure was explored. Matched pairs of MCE filters (filters paired and packaged together by the manufacturer based on similar weight) were used in this procedure to ensure that filter pairs would be affected identically by atmospheric conditions. Each pair was ionized and weighed. Each filter was placed in its own individually numbered Petri dish. All Petri dish lids were fully closed. Three such pairs were placed in a desiccator. Three more pairs were placed on the bench beside the desiccator. Two further pairs were divided such that one filter from each pair was in the desiccator and the other on the bench. All filters were ionized and weighed at 24, 96, 168, 192, and 220 hours after the original weighing. Samples in the desiccator were removed and quickly weighed to avoid possible absorption of water, then were replaced in the desiccator as quickly as possible. Relative humidity was recorded at the time that each filter was weighed. It is also noteworthy that this evaluation took place during the winter months when the relative humidity was low.

Data Summary and Analysis

Ionization

Table 2 gives test conditions and weight changes for PTFE filters when filters were weighed without the use of an air ionization unit. Table 3 gives test conditions and weight changes for PTFE filters when filters were weighed with the use of an air ionization unit. All oven treatments have duration of 15 minutes. All desiccator treatments have duration of 24 hours. The desiccator used was filled with Drierite® color indicating desiccant.

Treatment	Weight Gain (mg)	Average (mg)
Convection Oven, 70°C	0.194	0.177
Convection Oven, 70°C	0.160	
Convection Oven, 75°C	0.050	0.039
Convection Oven, 75°C	0.028	
Convection Oven, 80°C	0.604	0.328
Convection Oven, 80°C	0.052	
Vacuum Oven, 60°C	0.012	-0.024
Vacuum Oven, 60°C	-0.060	
Vacuum Oven, 70°C	0.032	0.144
Vacuum Oven, 70°C	0.256	
Desiccator	0.374	0.207
Desiccator	0.040	

Table 2: Weight changes for PTFE filters after filtering 300 milliliters hexane when subjected to the stated treatment and weighed without the use of an air ionization unit.

Treatment	Weight Gain (mg)	Average (mg)
Convection Oven, 70°C	0.060	0.059
Convection Oven, 70°C	0.058	
Convection Oven, 75°C	0.052	0.087
Convection Oven, 75°C	0.122	
Convection Oven, 80°C	0.054	0.065
Convection Oven, 80°C	0.076	
Vacuum Oven, 60°C	0.062	0.061
Vacuum Oven, 60°C	0.060	
Vacuum Oven, 70°C	0.056	0.053
Vacuum Oven, 70°C	0.050	
Desiccator	0.058	0.059
Desiccator	0.060	

Table 3: Weight changes for PTFE filters after filtering 300 milliliters hexane when subjected to the stated treatment and weighed with the use of an air ionization unit.

This data makes it readily apparent that the air ionization unit greatly increases the precision of testing. Use of the ionization unit decreased the standard deviation in filter weights by an order of magnitude: 0.19 mg without ionization, 0.019 mg with ionization. This is particularly significant in light of the 0.30 mg weight change limit for this evaluation procedure in some military specifications.

Similar experiments were attempted with MCE filters. However, these filters could not be successfully weighed without ionization. Weights did not stabilize, even after hours of resting on the microbalance pan. This is likely due to the interaction between static electricity within the filter and the balance pan. This same buildup of static electricity is likely the cause for the irregularities observed in weight changes when PTFE filters were weighed without the use of an air ionization unit.

Lid Position Effects

Figure 1 gives weight change data for MCE filters placed in Petri dishes placed in a desiccator with each of the lid positions (on, off, and ajar), as well as the control filters placed outside the desiccator. This data makes it clear that the currently prescribed method, keeping Petri dish lids ajar during treatments, gave unsatisfactory results. At all weighings, filters in Petri dishes with lids on had lower weight gain or greater weight loss than those with lids ajar. Those with lids completely off had still higher relative weights. It appears that the desiccation period removed some water from the filters, as observed in the differences in weight changes between the filters in Petri dishes with lids on and the control filters placed outside the desiccator, also with lids on. However, this data also shows that there was another source of weight change affecting the filters. Since these MCE filters are hydrophilic, the obvious reason for weight change is the sorption of water. The only source for water absorption was the surrounding air, so the relative humidity was recorded at each weighing. Table 4 gives the relative humidity and relative humidity change in the laboratory at the time of each filter weighing. There is a clear correlation between the weight changes of the filters and the relative humidity at the time of the weighing. Filter weights increase and decrease with relative humidity. However, there appears to be a small amount of weight loss that occurred after the filter is first removed from the package and weighed. This weight loss was possibly due to the evaporation of another species, possibly plasticizer, but this has not been confirmed. The observed deviations in weight changes indicated that the interactions between water and filter may have been inconsistent from filter to filter. Some filters lost more weight within the desiccator than others, even when evaluated with similar Petri dish lid positions.

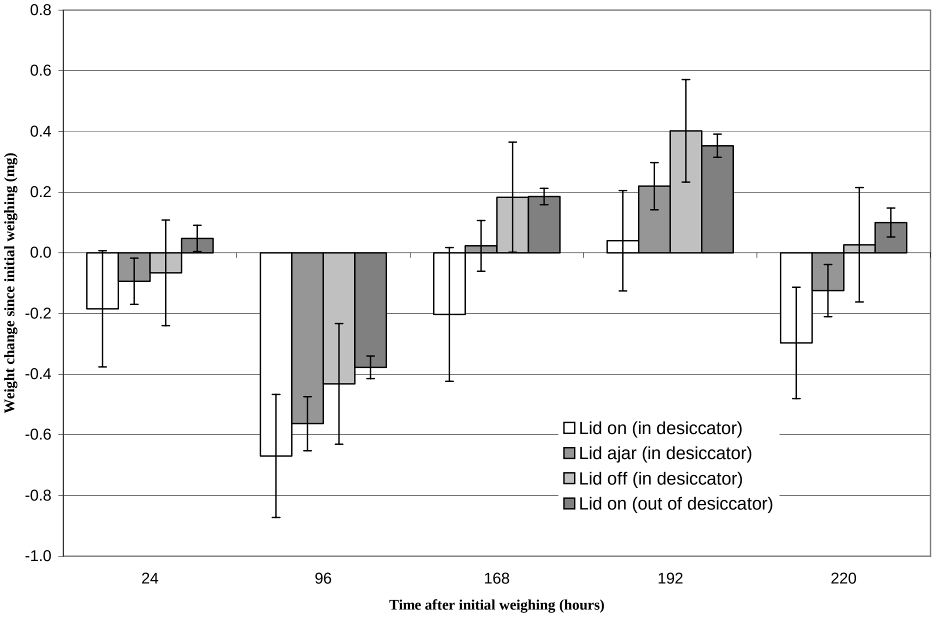

Figure 1: Filter weight changes for varying lid positions in and out of desiccator.

Time (hr)	RH (%)	RH Change (%)
0	24	N/A
24	24	0
96	9	-15
168	30	6
192	38	14
220	28	4

Table 4: Relative humidity and relative humidity change at the time of each filter weighing.

Since filter weights changed so much with relative humidity, it was decided that the effects of humidity needed to be further characterized. Figures 2 and 3 gives filter weight changes as a function of relative humidity change for filters stored in a desiccator and in air, respectively. Note that nine percent relative humidity is used as the reference because it was the lowest observed relative humidity. Sixteen MCE filters were placed in Petri dishes. Half of these were placed in an ambient pressure desiccator, while the other half were placed on the bench. This data makes it clear that changes in relative humidity can skew the results of gravimetric analysis testing. It is also apparent that time spent in the desiccator is relatively unimportant, as weight change appeared a function of relative humidity and not time. This is likely because the sorption of water by the filter occurred rapidly enough that the water achieved equilibrium with the surrounding air in the time required between removing the filter from the desiccator and weighing. This explanation is plausible since the quantities of water in question are so small relative to the amount of surface area interacting with the surrounding air.

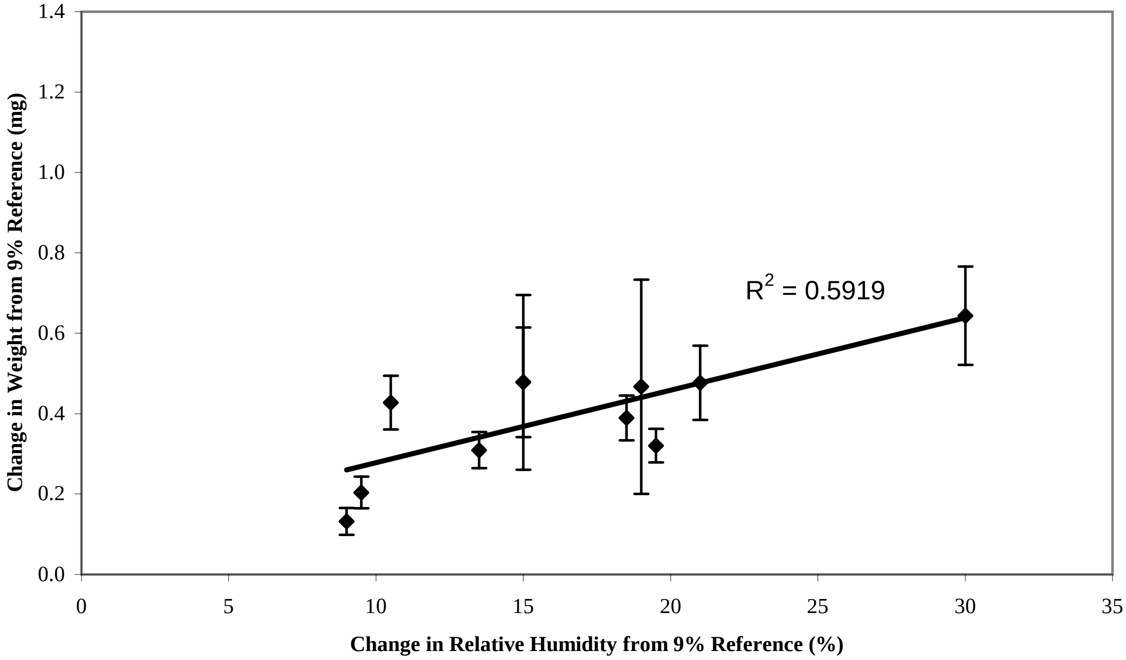

Fig. 2: Filter weight change as a function of relative humidity for filters stored in a desiccator. Filter weights recorded at 9% relative humidity are used as reference weights for each filter. Error bars indicate standard deviation in observed weight changes.

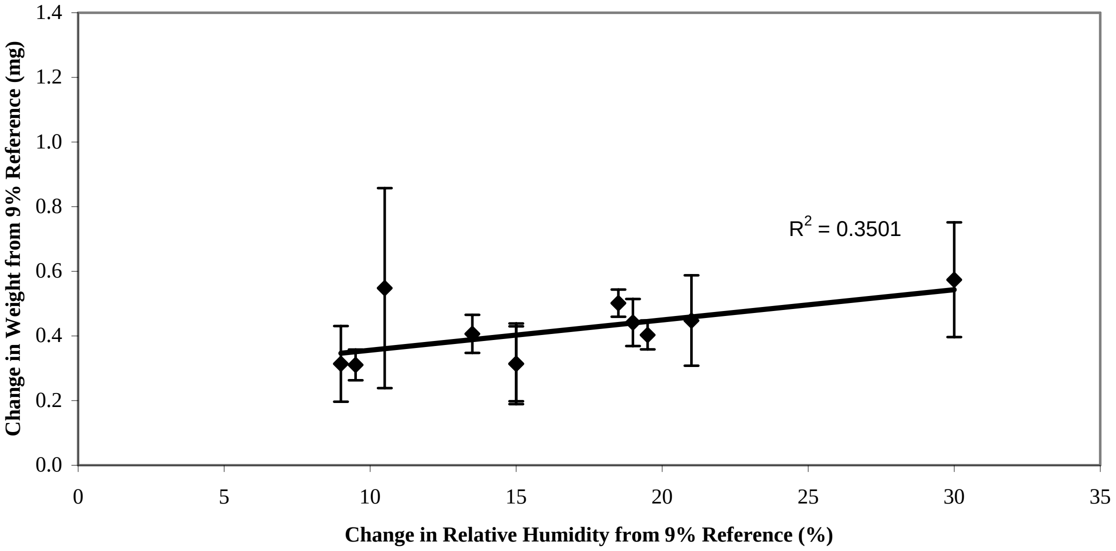

Fig. 3: Filter weight change as a function of relative humidity for filters stored in air. Filter weights recorded at 9% relative humidity are used as reference weights for each filter. Error bars indicate standard deviation in observed weight changes.

It is worth noting that the standard deviations of these measurements were smaller for the desiccator samples, particularly at low relative humidity changes. These low changes are what one might expect over the short course of a single gravimetric analysis procedure and it is important to minimize these variations.

Part II: Solvent and Treatment Effects

Experimental

One possible source of error is from glassware contamination. To eliminate this possibility, a thorough cleaning procedure was instituted. All glassware was first washed thoroughly using hot water and surfactant-free soap. The glassware was then rinsed twice with double-distilled water, followed by reagent grade 2-propanol, hexane, then filtered hexane. Solvents evaporated rapidly in a fume hood to dry the glassware. All open areas of glassware were then carefully wrapped with clean aluminum foil to prevent contamination prior to use.

Hexane used during all testing was filtered prior to use by passing it through a filter of the type to be used in the analysis. Only 300 milliliters of hexane was filtered prior to filter replacement to ensure that no degradation of filter efficiency would occur, outside of that which would occur during normal testing. It is anticipated that filtered hexane would remove any particulate that might cause a weight change in filters during evaluation. Filtered hexane was stored in a glass solvent container that had been previously cleaned using the above glassware cleaning procedure. This container was sealed with a polytetrafluoroethylene-lined cap to prevent contamination.

To determine the cause of sporadic weight changes observed in the MCE filters tested, the filter manufacturers were consulted. Information provided by the filter vendors indicated that some filters were plasticized to prevent the filter material from being too brittle. Therefore, a simple test was conducted to evaluate the weight loss due to extraction of the plasticizer by the hexane. Each filter type was evaluated for weight change by varying amounts of solvent and using a specific drying method. Filters were first ionized with a StaticMaster Ionizing Unit to reduce static interference with the metal balance pan. Ionization was performed by passing the filter between the parallel plates of the ionizer for approximately ten seconds. The filter was weighed and placed in the filtration apparatus (Fig. 4). A light vacuum was initiated and the desired amount of filtered hexane was passed through the filter. The flask pressure was maintained at 15 inches of mercury (~50000 Pascals) until all hexane had passed through the filter. The filtration funnel was then removed and the pressure increased to approximately 25 inches of mercury (~84400 Pascals). The vacuum was released and the filter removed after visibly dry (typically one minute). The filter was then placed in a closed plastic Petri dish, which was placed in a convection oven, maintained at 75°C for 15 minutes. The Petri dish was then removed and placed in a desiccator for 15 minutes to allow the filter to reach room temperature. The filter was then removed from the dish, ionized, and weighed.

Figure 4: Filtration apparatus detail.

A similar approach was used to evaluate filter weight changes for varying treatments. The test filter was ionized and weighed. Any desired pretreatment of the filter was then applied. The filter was then placed in the filtration apparatus. A light vacuum was initiated, and then 100 mL of filtered hexane were decanted through the filter. The interior of the filtration funnel was then thoroughly rinsed with filtered hexane. The filtration funnel was then removed. The rubber seal on the bottom of the filtration funnel was rinsed with filtered hexane, taking care to ensure that all hexane passed through the test filter. The filter itself was then rinsed with filtered hexane until the total amount of hexane used was 300 mL. The flask pressure was maintained at 15 inches of mercury (~50000 Pascals) until all hexane had passed through the filter. Care was taken while rinsing the filter to ensure that the solvent spray was always towards the center of the filter to avoid removing any particulate contaminants. The flask pressure was increased to 25 inches of mercury (~84400 Pascals). The vacuum was then released and the filter removed after visibly dry (typically one minute). The filter was carefully placed into a plastic Petri dish. The Petri dish was then subjected to a drying treatment, as detailed in Table 5, after which it was equilibrated to room temperature in a desiccator. The filter was then removed from the dish, ionized, and weighed.

Test #	When Applied	Apparatus	Temperature	Duration
1	Prior to Filtration	Desiccator	Ambient	24 hours
2	Prior to Filtration	Convection Oven	70°C	15 mins
3	Prior to Filtration	Convection Oven	80°C	15 mins
4	After Filtration	Convection Oven	70°C	15 mins
5	After Filtration	Convection Oven	75°C	15 mins
6	After Filtration	Convection Oven	80°C	15 mins
7	After Filtration	Vacuum Oven	60°C	15 mins
8	After Filtration	Vacuum Oven	70°C	15 mins
9	After Filtration	Desiccator	Ambient	6 hours
10	After Filtration	Vacuum Desiccator	Ambient	2 hours

Table 5: Explicit definitions of test conditions and test reference numbers.

One approach to allow for test oil or solvent extraction of plasticizer, or other filter-related material, from the filter is to use two filters stacked on top of each other. The weight change of the bottom filter is subtracted from the weight change of the top filter to theoretically cancel out weight change due to the extraction of filter materials by solvent or test oil. This method should also account for the change in filter weight due to humidity and other environmental effects, and is therefore recommended by the American Society for Testing and Materials (ASTM-D4898) for hydraulic fluid gravimetric analysis. The two-filter evaluation was carried out in an identical manner as the single filter testing. The plastic Petri dishes were clearly marked to distinguish which contained the top or bottom filters. Reported weight changes in filter pairs are given as the difference in weight change in the test (top) filter and control (bottom) filter. This assumes that the bottom filter undergoes all environmental and external contamination changes that the test filter does, but does not accumulate any particulate from the test fluid sample. Therefore, the reported pair difference is the filter weight change due solely to sample contamination.

Four different treatments were devised for removal of solvent and other volatile material from filters to ensure that any weight changes observed were due to fluid contaminant during testing, when a convection oven was used at temperatures of 70, 75, and 80°C. A vacuum oven was used at 60 and 70°C with a constant pressure of 5 inches of mercury (~17000 Pascals). All oven exposures lasted 15 minutes. In addition, both ambient pressure and vacuum desiccators were used. Samples were placed in an ambient pressure desiccator for 6 hours and in a vacuum desiccator for 2 hours.

It is important to note that exposure of the filter to open air should be minimized. While in the Petri dish, the lid should remain on at all times. The lid should be set lightly in place when the dish is placed in an oven or desiccator for treatment to allow the escape of volatile species.

Results and Discussion

Due to the high accuracy requirement of this test, the analysis of test results is based on very strict criteria. A successful test shall be one in which the average filter weight change, or apparent weight change of the filter pair, is less than 0.05 milligrams with less than 0.02 milligrams of deviation in testing. A mediocre result shall be a procedure in which these values are 0.10 milligrams and 0.04 milligrams respectively. Any other results will be considered failing. These established limits are based on experimental data and the existing limit for military acceptance evaluations (0.30 mg). All filter weight change evaluations were conducted in duplicate.

Single Filter Tests

Initially, the examination of filter weight change focused on single filter techniques as called for in MIL-PRF-87257 and MIL-PRF-83282 military specifications. Two different brands of MCE filters were evaluated using a predetermined test matrix. Tests are referred to by reference number, given in Table 5. Not all filters were subjected to all tests.

Two MCE filters in common use in military laboratories for the examination of hydraulic fluid contamination, filters A and B, were evaluated. Note that these filters are intended for use in biological testing of aqueous solutions, so the use of plasticizers in their manufacture should not be surprising. Fig. 5 gives observed weight changes for filter A, while Fig. 6 gives observed weight changes for filter B. Test condition 10 was not performed for filters A, B, or C at this point because vacuum desiccation was only considered as a treatment option when it was learned that same-day test results were often required for this analysis.

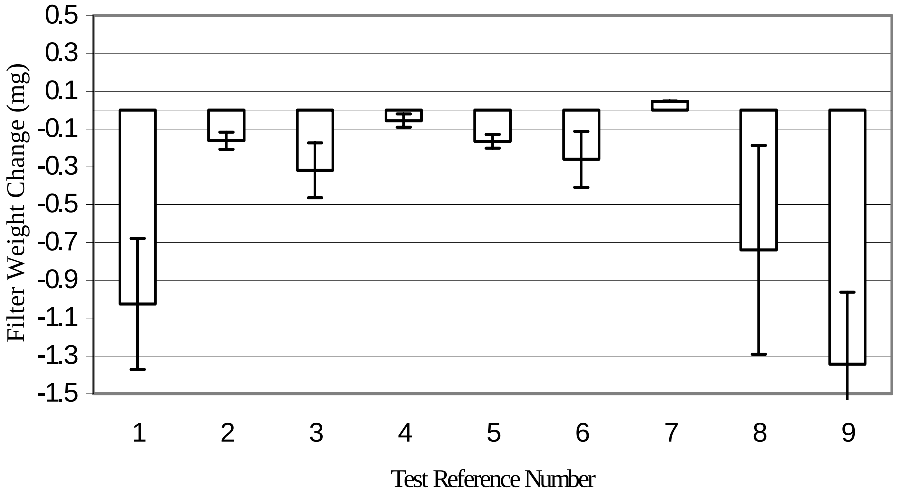

Fig. 5: Weight changes for various treatments of filter A. Test reference numbers given in Table 2. Error bars indicate experimental differences in filter weight changes.

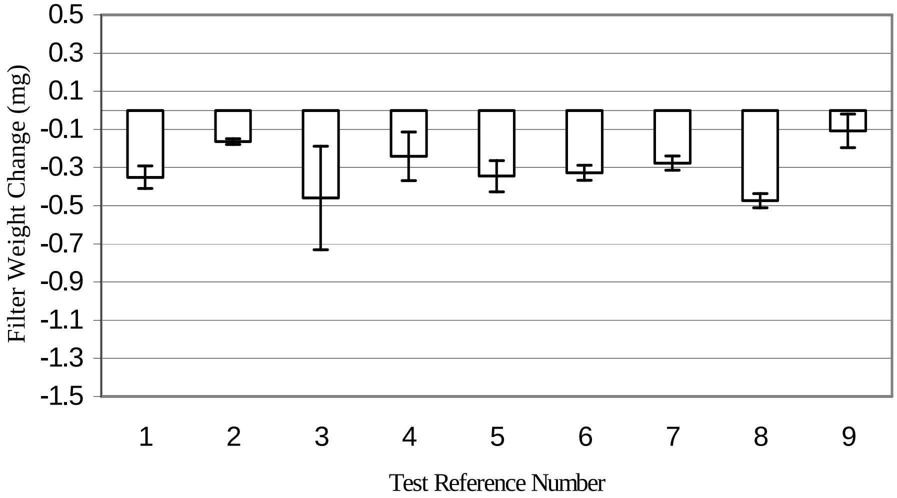

Fig. 6: Weight changes for various treatments of filter B. Test reference numbers given in Table 2. Error bars indicate experimental differences in filter weight changes.

When the weight changes from these filter types were evaluated, it was observed that they were both very erratic and large relative to the weight change limit of 0.30 milligrams outlined in relevant military specifications. The only successful analysis was that of filter A evaluated using procedure 7, which is likely due to the low temperature (60°C) applied in the vacuum oven. This low temperature may decrease the volatilization of plasticizers, as further discussed below. The evaluation of filter A using procedure 4 is mediocre, again giving fairly good results at the lowest temperature tested (70°C). All other tests failed based on the large observed weight losses. In an effort to find a more suitable filter, a PTFE filter (filter C) was examined in the same manner as the MCE filters. The choice of a PTFE filter was based on its ability to withstand high temperatures and its hydrophobic nature, and inert behavior. PTFE filters do not have plasticizers. Figure 7 gives observed weight changes for filter C.

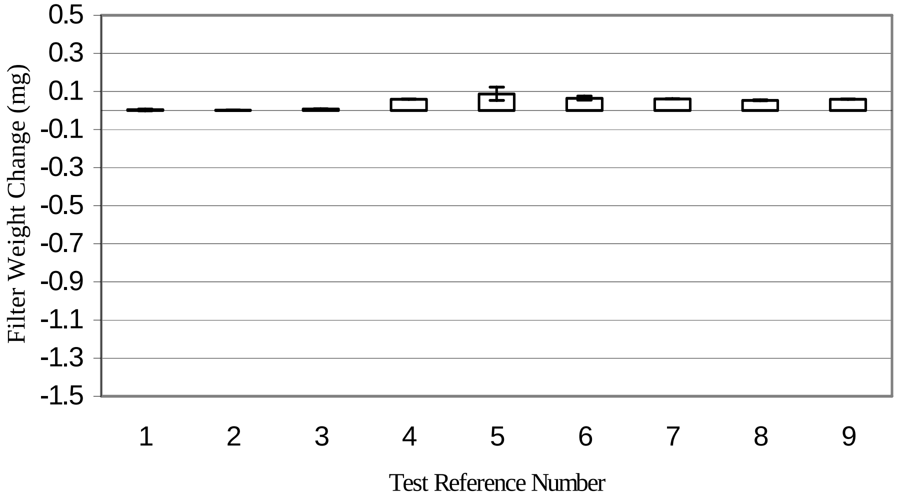

Fig. 7: Weight changes for various treatments of filter C. Test reference numbers given in Table 2. Error bars indicate experimental differences in filter weight changes.

The results from testing filter C are nearly ideal, indicating that this material is suitable for gravimetric analysis testing. However, the filter itself is extremely difficult to handle. Its inherent flexibility at ambient conditions causes it to sag under its own weight to the extent that contaminants would likely be lost. Therefore, another PTFE filter supported with polyethylene backing was briefly examined. Unfortunately, this filter proved unsuitable for testing purposes due to constriction of the polyethylene material during various treatments. This constriction deformed the filter in such a way as to make handling, weighing, and storage impossible with the Petri dishes and microbalance used. Still searching for a more suitable material, a polypropylene filter, filter D, was examined. Figure 8 gives observed weight changes for filter D.

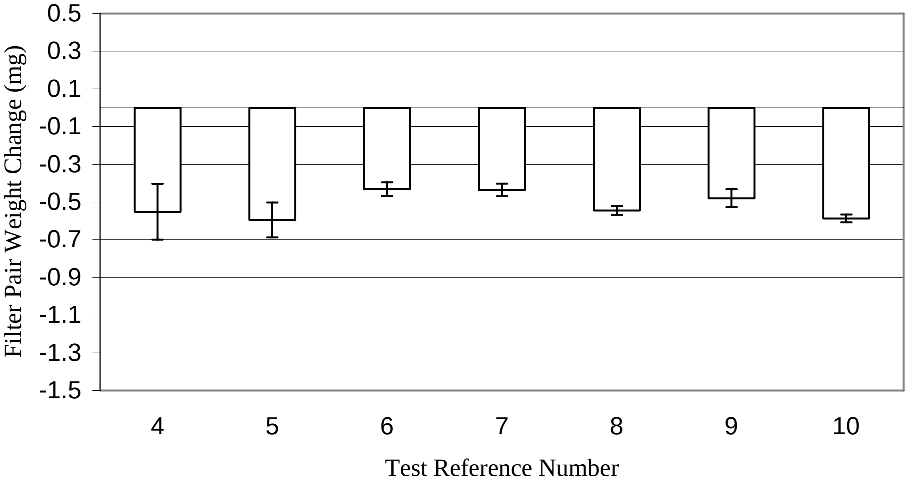

Fig. 8: Weight changes for various treatments of filter D. Test reference numbers given in Table 2. Error bars indicate experimental differences in filter weight changes.

Filter D failed under all evaluation procedures. However, the typical precision of the results was greater than that of the MCE filters A and B, with all analyses resulting in similar weight losses. (Test scenarios 1, 2, and 3 were not performed on this filter because it was shown, as detailed in Part I, that humidity effects would negate any drying of the filters by such treatments.)

Since filters A, B, and D all lost weight under almost all drying conditions, it was hypothesized that a volatile species was evaporating from the filters. The obvious source of weight loss was due to the removal of water from the filters. While this likely accounts for some of the weight loss, the lack of precision in the test results is likely caused by variations in relative humidity, as detailed in Part I. Several filter manufacturers agreed that the most likely cause of weight loss in both MCE and polypropylene filters is the loss of plasticizer, either to the solvent during filtration or to evaporation during drying. Therefore, each of these filters was evaluated with test method 5 (after filtration, convection oven at 75°C for 15 minutes) applied after solvent filtration. Filters were also examined for stiffness, a typical sign of plasticizer loss, after treatment. Filter C was tested as a control filter to examine the weight loss due to the evaluation procedure itself, as PTFE does not require the use of plasticizers for flexibility. Table 6 gives data for plasticizer removal testing.

	100 mL solvent		300 mL solvent	
Filter Type	**Weight Change (mg)**	**Stiff?**	**Weight Change (mg)**	**Stiff?**
Filter A	-0.206	No	-0.198	No
	-0.224	No	-0.266	No
Filter B	-0.030	No	-0.192	No
	0.178	No	0.034	No
Filter D	-0.284	No	-0.272	Yes
	-0.284	No	-0.276	Yes
Filter C (control)	0.020	No	0.006	No
	-0.014	No	0.000	No

Table 6: Data for plasticizer removal testing with drying procedure 5.

From the plasticizer removal testing, evaluation of the poor handling qualities of PTFE filters, and other evaluations, it became clear that none of these filters was suitable for use in single filter procedures requiring a high degree of accuracy. Recalling other testing methods used in specifications, it was decided to examine the use of two filters simultaneously in the filter apparatus. Ideally, the presence of the second control filter, placed underneath the test filter during testing, would make an allowance for any filter weight changes caused by ambient conditions, test procedures, or other outside sources. This is the current procedure called for by ASTM.

Two Filter Tests

From test results, it was observed that both filters A and B exhibited sporadic weight changes under standard testing conditions, again indicating that they are unsuitable for both single and two filter test procedures. However, since this evaluation was so limited in scope, these filters were still evaluated using the two filter method. Filter D lost a substantial amount of weight, but was very consistent in amount of weight loss for a set amount of solvent. This made filter D unsuitable for single filter test procedures, but a good candidate for analysis methods using two filters. Figures 9, 10, and 11 give weight change data for filters A, B, and D, respectively, for two filter evaluation. These tests were performed using conditions 4, 5, and 6 (Table 5). Filter pair weight changes were calculated as the weight change of the top (test) filter minus the weight change in the bottom (control) filter.

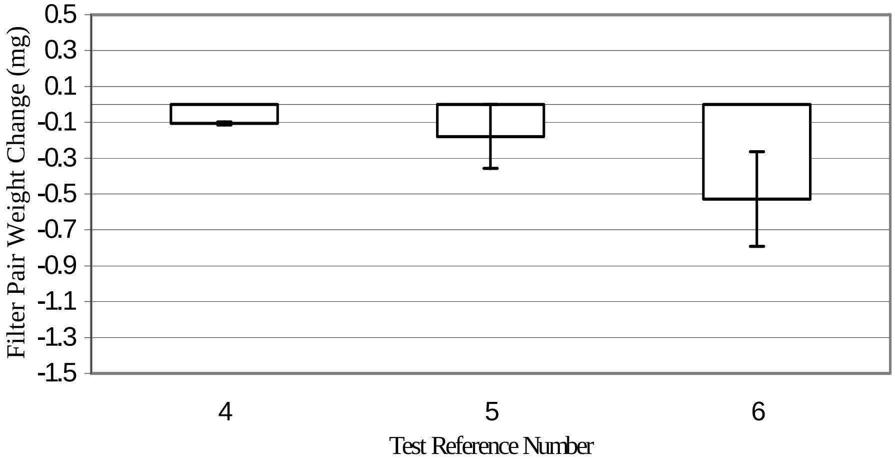

Fig. 9: Filter pair weight changes for various treatments of filter A using the two filter method. Test reference numbers given in Table 2. Error bars indicate experimental differences in filter pair weight changes.

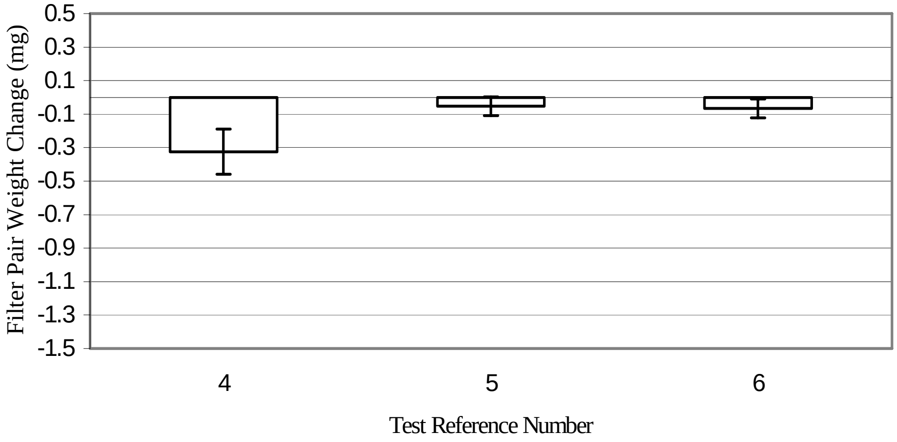

Fig. 10: Filter pair weight changes for various treatments of filter B using the two filter method. Test reference numbers given in Table 2. Error bars indicate experimental differences in filter pair weight changes.

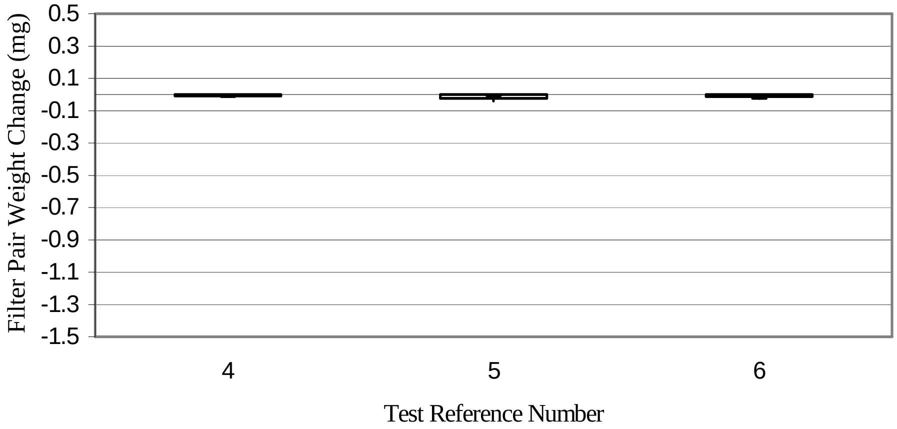

Fig. 11: Filter pair weight changes for various treatments of filter D using the two filter method. Test reference numbers given in Table 2. Error bars indicate experimental differences in filter pair weight changes.

Filters A and B both showed erratic results at all three temperatures and so these filters are not considered acceptable for two filter procedures. However, filter D showed precise, accurate test results, indicating that this filter is suitable for testing using two filter procedures.

Conclusions and Recommendations

Based on these findings, several procedural changes are recommended for accurate, precise gravimetric analysis testing. Some of these recommendations coincide with current testing methodology, while others point out shortcomings of current procedures.

1) It is clear that an air ionizer is required to perform testing using the filter types tested. This is already noted in relevant testing methods.
2) The Petri dish lid must remain on the dish at all times while the filter is inside. The placement of Petri dish lids throughout the course of testing plays a small role in preventing outside sources of contamination. While the exact extent of this has yet to be determined, it is clear that placing the lid completely over the bottom of

the Petri dish can prevent the accumulation of particulate on test filters during storage and treatment.

3) Filters should be stored in Petri dishes in a desiccator prior to use to increase repeatability.
4) Single filter test methods do not provide the necessary accuracy for this evaluation.
5) Filters A and B (MCE filters) both performed poorly in both single and two filter procedures. Filter A may produce acceptable results when dried at lower temperatures when using single filter evaluation methods, but does not appear suitable for two filter testing. Filter B failed all single filter evaluation methods, but showed mediocre performance for some two filter tests, tending to better results at higher temperatures.
6) Filter C (a PTFE filter) exhibited very good accuracy and precision in almost all evaluations conducted. However, the filter was unsuitable for gravimetric analysis using this procedure due to the inevitable loss of particulate matter during handling. If a way could be found to support this filter material during handling and transportation, PTFE filters may provide the best results of any filter material, even when using only a single filter for analysis.
7) Filter D (a polypropylene filter) exhibited large but consistent weight losses under all drying conditions. It is therefore unfit for gravimetric analysis using single filter techniques. Results using two filter methods are excellent. Of the filters tested, filter D showed the most accurate and repeatable weight changes when the two filter procedure was implemented.
8) Based on these results hydraulic fluid military specifications have been changed to require two D filters.

References

MIL-PRF-5606, 7 June 2002, Military Specification Hydraulic Fluid, Petroleum Base; Aircraft, Missile and Ordnance

MIL-PRF-83282, 30 Sept 1997, Performance Specification Hydraulic Fluid Fire Resistant, Synthetic Hydrocarbon Base, Metric, NATO Code Number H-537

MIL-PRF-87257, 22 April 2004, Military Specification, Hydraulic Fluid, Fire Resistant; Low Temperature, Synthetic Hydrocarbon Base, Aircraft and Missile

ASTM-D4898-90, *Standard Test Method for Insoluble Contamination of Hydraulic Fluids by Gravimetric Analysis* (reapproved 2005)