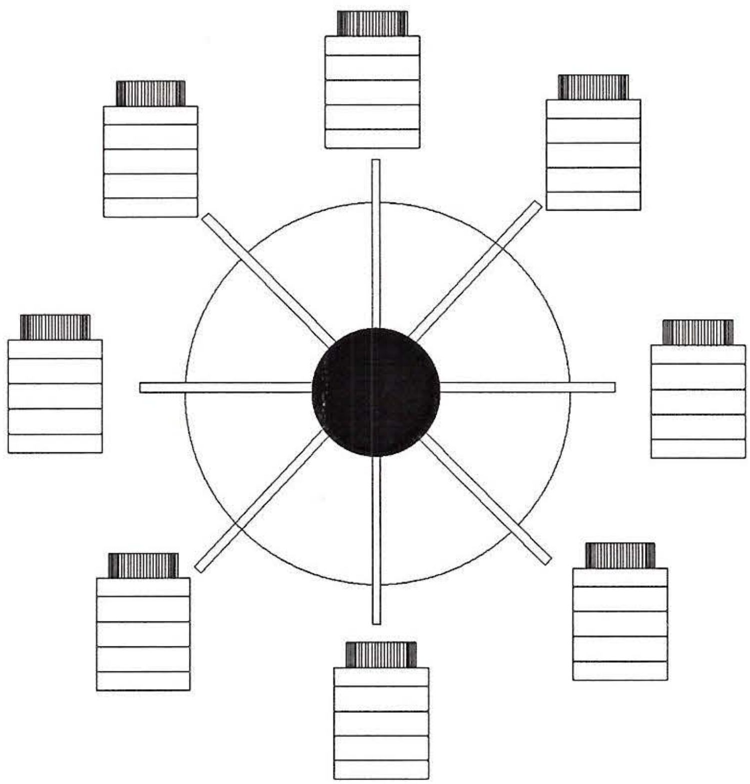
DIAGNETICS

TECHNICAL APPLICATION ARTICLE

8

Comparison of Particle Counts Between Eight Commercial Oil Analysis Laboratories



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INTRODUCTION

The inclusion of particle counting in the periodic analysis of hydraulic and lubricating fluids has provided an important new advancement to machine diagnostics. With particle counting, machinery users can monitor the principal cause of failure, not just the symptoms, or results, of failure. The benefit, when particle levels are controlled, is extended machine life and reduced failure frequency. This is the objective of the growing practice of proactive maintenance.

Along with this important trend has come the practical questioning of particle counter accuracy. It is estimated that by the year 2000, as many as 50 million particle counts will be performed on fluid samples each year. Hence, a failure to do particle counting with reasonable accuracy could effectively undermine user confidence and erode this incredible rate of growth.

While calibration techniques are available for most types of particle counters, the frequency and proper use of these techniques is not well understood. Likewise, it can be questioned whether the type of fluid and test particles (calibration fluid) used in calibration of particle counters is sufficiently close to field oils and field contaminants. Additionally, accuracy is also influenced by bottle cleanliness, fluid agitation, deaeration, dilution, dilution fluid cleanliness, and operator error.

This report attempts to assess the general accuracy of particle counting as performed by commercial fluid analysis labs. Commercial labs were used as it was felt that they would be the most sophisticated in terms of technique, calibration, instrumentation, and technician skill. Eight of the largest and most respected labs were included in this study. Eight identical sets of four different samples were sent to these labs. Every effort was made to control variables not related to the lab itself. The results were analyzed in terms of lab-to-lab variability, since a scientific absolute particle count was not known.

OBJECTIVES OF STUDY

The principal objective of this study was to use an experimental approach to assign some level of accuracy to particle counters in commercial oil analysis applications. At the same time, the study was able to measure and compare other aspects, such as:

1. Lead time to receive results
2. Cost of particle counting
3. Instrumentation used
4. Sample preparation methods
5. Instrument operating procedures

It is hoped that the results of this study will help both the users and the labs to better understand these important issues. Likewise, it is hoped that this information will lead to distinct improvements by commercial labs, leading to higher confidence of users.

DESCRIPTION OF LABS

The labs included in this study were carefully selected based on reputation and perceived volume of commercial fluid analysis business. Five of the eight labs are considered to be among the largest commercial labs in the United States; a few with multiple locations. The other three labs offer fluid analysis as a support service to other products and services they provide. For example, one of these labs is operated by a very large filter company, another lab is operated by a major producer of lubricants and hydraulic fluids, and the third lab is a part of an organization providing a wide range of scientific, research, and testing services.

To avoid the possibility of alerting the labs to this study, the analysis services of six of the labs were purchased through a third-party intermediary. The other two labs were already providing oil analysis services for us on an occasional basis. All services were purchased, invoiced, and paid in the traditional manner. No favors of free services were requested or provided. Therefore, it is believed that the samples were handled and analyzed in the routine manner.

Most labs asked for questionnaires to be completed for each sample. The same information, data, and specifications for each sample was given to each individual lab. Only one of the eight labs called to ask for more information about the samples.

Separately, a telephone interview was conducted with the manager and/or lab technician of each lab. Each of these lab managers were asked the same questions and a transcript of their answers was recorded. A summary of the questions and answers is given in Table 1 and discussed below:

<u>Type of Particle Counter</u>. Seven of the eight labs used optical particle counters of the same brand.

<u>Calibration Frequency</u>. The frequency of calibration greatly affects accuracy. The frequency varied from once every day to once every six months.

<u>Method of Agitation</u>. Tests have shown that vigorous agitation is often needed to resuspend particles that have settled to the bottom of sample containers. As such, hand shaking and rocker shakers may leave particles on container surfaces.

TABLE 1
Summary of Phone Interviews with Labs

LABS	A	B	C	D	E	F	G	H
Type of Particle Counter	Optical	Optical	Optical	Optical	Optical	Optical	Optical	Optical
How Often it is Calibrated	every 6 months	every 5 samples	regularly	every 6 months	each week	each day	each month	every 2 months
Method of Agitation	rocker type agitator	mixing machine	paint shaker	don't do it	paint shaker	by hand	rocker typer agitator	by hand
How Long it is Agitated	15 min.	20 min.	15 min.	don't do it	15 min.	1 min.	5 min.	1 min.
Method of Deaeration	vacuum only	ultra-sonic only	vacuum & ultra-sonic	vacuum & ultra-sonic	vacuum & ultra-sonic	ultra-sonic only	vacuum & ultra-sonic	vacuum & ultra-sonic
Use of Dilution	only if dark	sometimes, TCE	yes, MIL-H-5606	no	yes, MIL-H-5606	yes, TCE	no, unless not enough sample	yes, MIL-H-5606
Assessment of Moisture	visible	yes	yes	no	yes	yes	visible & spat test	spat test

Agitation Period. An adequate amount of agitation is 15 minutes in a paint shaker for precision particle counts. Poor agitation is one of the biggest causes of particle counting errors.

Method of Deaeration. The best labs use both ultrasonic and vacuum degas to remove small air bubbles from the sample. The use of ultrasonic or vacuum degas only, may leave bubbles in the more viscous fluids.

Use of Dilution. With optical particle counters, dilution is generally needed to avoid the risk of particle counter sensors becoming saturated. Ultraclean MIL-H-5606 is the preferred dilution fluid. Lighter dilution fluid, such as trichloralethane can cause particle settling.

Assessment of Moisture. Emulsified water particles are seen by the optical particle counter (OPC) as solid particles. Trace amounts of moisture can be very difficult to detect. The spat test (crackle test) is generally considered the most reliable indicator of moisture. Most labs were not clear on how they check for moisture before doing a particle count.

DESCRIPTION OF SAMPLES

Seven of the eight labs provided their own sample bottles. For the other lab, a certified super clean bottle was used. No attempt was made to clean the bottles provided by the labs. Likewise, care was taken to not remove the caps from these bottles until the sample was ready to be introduced.

Each of the four samples was prepared in batches and dispensed to the eight bottles all at one time. A description of the four samples and their preparation is given below:

Sample One. This sample was prepared in the test reservoir of an ISO-validated multipass filter test stand. Two inline optical particle counters, calibrated to ISO 4402, were installed. After the fluid was cleaned rigorously, AC Fine Test Dust was introduced to the test reservoir containing MIL-H-5606 fluid. After several minutes of circulation, the particle counts stabilized at approximately 530 greater than 10 microns per ml. At that time, the eight sample bottles were filled from the bottle sampling tap and capped immediately. Each bottle was filled no more than 3/4 full. (Note: none of the sample bottles instructed users not to fill the bottle more than 3/4 full. This is necessary for adequate agitation.) The slope of particle size distribution of AC Fine Test Dust is considered very predictable. This slope will be compared to actual particle counts from the commercial labs. This sample is considered very similar to ISO calibration fluid used for optical particle counters.

Sample Two. This sample was prepared in a liter size plastic bottle. Approximately 750 ml of 10-weight motor oil was placed in the bottle. The fluid was contaminated with several grams of metal filings sifted through a 125 micron sieve. After the filings were introduced, the bottle was agitated on a paint shaker for more than 15 minutes. The eight sample bottles were then filled one at a time. Between each fill, the liter bottle was reagitated to guard against particle settling. Successful particle counting of this highly contaminated fluid requires good agitation and extensive dilution.

Sample Three. This sample was prepared with a multipass filter test stand, like sample number one. The MIL-H-5606 fluid was rigorously cleaned with a one-micron filter until the inline particle counters showed zero particles greater than 10 microns per ml. At that point, each of the eight bottles was quickly filled in the same manner as previously described under sample one. This sample will assess the influence of poorly cleaned bottles, dirty dilution fluid, and ineffective deaeration.

Sample Four. This sample was prepared in a one liter bottle using 10-weight motor oil. The one liter bottle was new but uncleaned. The motor oil was new but unfiltered. No additional particle contamination was introduced to the fluid. Instead, ten drops of water were added.

Afterwards, the liter bottle was vigorously agitated on a paint shaker for more than 15 minutes. The moisture was noticeable as a light haze in the fluid. The eight sample bottles were filled in the manner described above for sample two. Accurate particle counting requires the removal of emulsified water first.

After a week, all eight sets of four different samples were packaged and sent by UPS to the respective labs. All of the labs are located in the United States.

INTERPRETATION OF RESULTS

Cost Comparison. The comparison of the labs on the basis of cost is presented in Table 2 below:

TABLE 2
Cost Comparison

Lab	$/Sample	Lab	$/Sample
A	31.50	E	65.00
B	24.75	F	25.00
C	12.76	G	29.00
D	31.75	H	25.00

Average - $30.60/Sample

Lab E, which is the scientific testing lab, is considerably higher than the others due to the presumed lower volume of samples that they analyze. However, several of the labs performed other tests in addition to particle counting, such as elemental analysis, viscosity analysis, moisture analysis, and total acid number (TAN). They declined our request to price their services for particle counting only.

Turnaround Time Comparison. A quick turnaround time by labs in doing the analysis and sending back test results is very important. Late results can seriously undermine the effectiveness of a proactive maintenance program. In the study, all eight samples were sent out the same day by UPS. The total number of days from that time was counted and is summarized in Table 3 below:

TABLE 3
Turnaround Time

Lab	No. of Days	Method
A	7	Mail
B	8	Fax
C	3	Fax
D	8	Mail
E	9	Fax
F	12	Mail
G	12	Fax
H	81	Fax
Average	17.5	
Average without Lab H	8.4	

Lab H took an extraordinarily long time to analyze the four samples. They claimed that the problem was moisture. After several calls to this lab, we were finally able to get our results. The two labs that took 12 days are high volume oil analysis labs. The fastest lab analyzed and faxed us results the same day they received it, three days in all.

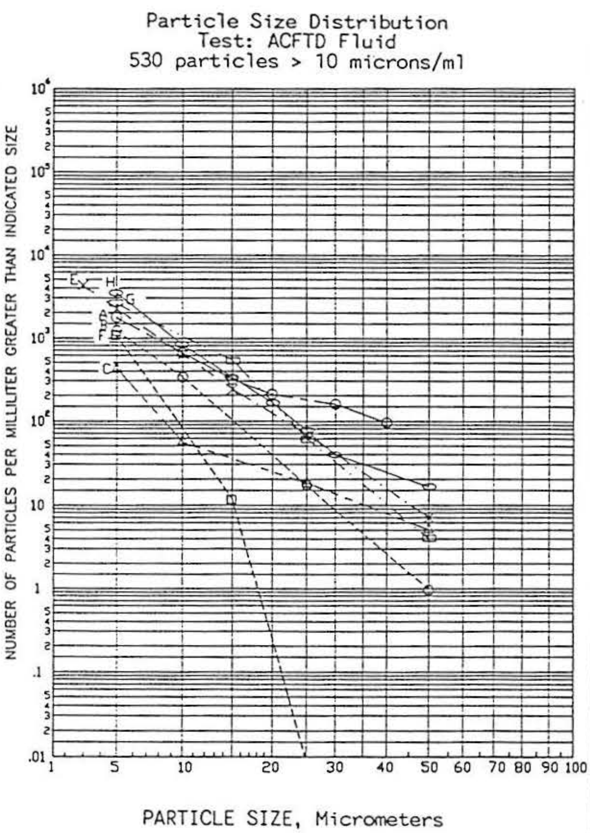
Analysis of Sample One Results. (See Figure 1 and Table 4). The use of AC Fine Test Dust in sample one allows us to more precisely examine accuracy in terms of both particle concentration and distribution. As a group, the labs performed much better with this sample than the other three. The reason might be that AC Fine Test Dust (ACFTD) and MIL-H-5606 fluid is the calibration medium used for optical particle counters.

The 10 micron counts averaged 531 particles per ml. When the sample was prepared, the inline counter registered 538 particles greater than 10 microns per ml.

TABLE 4 Comparison of Particle Counts - Sample One									
Labs	A	B	C	D	E	F	G	H	Average
>5 microns/ml	1,555	1,285	422	N/A	2,313	1,173	2,635	3,523	1,843.71
>10 microns/ml	633	343	57	N/A	624	*80	*1,100	883	531.43
>15 microns/ml	311	*100	*35	N/A	243	13	506	304	216.00
>20 microns/ml	207	*40	*25	N/A	*104	*0	*160	*150	98.00
>25 microns/ml	*180	19	19	N/A	68	0	62	78	60.86
>30 microns/ml	132	*9	*13	N/A	*25	*0	*32	*40	35.86
>50 microns/ml	*60	1	5	N/A	7	0	4	*16	13.28
ISO	18/15	17/14	16/12	N/A	18/15	17/11	19/16	19/15	18/15
Slope 5/15	.6	.8	.7	N/A	.7	1.4	.5	.8	.8

Note: Numbers marked with "" are straight line extrapolations, not reported counts.

Figure 1
Sample 1



However, the spread of 10 micron counts ranged from as low as 57 particles per ml at one lab, to as high as 1100 particles at another. Likewise, the ISO codes ranged from 15/12 to 19/16.

The correct particle size distribution slope for ACFTD is about 0.7. The slope average for the group was 0.8, ranging from 0.5 to 1.4. Five of the seven labs reported slopes in the narrow band from 0.6 to 0.8. Lab D claimed the sample size (70 ml) was insufficient to perform particle counts. Although none of the other labs had this problem, they could have easily added a measured amount of superclean dilution fluid. We were still charged for the analysis that was not performed. Likewise, the sample was not returned to us.

Analysis of Sample Two Results. (See Figure 2 and Table 5). The gross level of contamination of sample two presents a difficult challenge for optical particle counters. However, the concentration of particles in this sample is not unlike those typically found in unfiltered gear and bearing lubricants. In order to count particles at high concentrations with optical particle counters, considerable dilution is

TABLE 5 Comparison of Particle Counts - Sample Two									
Labs	A	B	C	D	E	F	G	H	Average
>5 microns/ml	16,896	79,865	1,041,773	N/A	1,852,183	3,486	18,165	N/A	502,061.33
>10 microns/ml	15,434	18,888	170,817	N/A	240,183	*250	*10,480	N/A	76,008.66
>15 microns/ml	13,168	*7,800	*8,200	N/A	35,232	34	12,624	N/A	12,843.00
>20 microns/ml	10,304	*3,900	*5,000	N/A	*10,500	*.42	*7,000	N/A	6,117.40
>25 microns/ml	*5,900	2,008	3,320	N/A	5,883	0	4,762	N/A	3,645.50
>30 microns/ml	4,280	*720	*2,400	N/A	*4,600	*0	*3,400	N/A	2,566.67
>50 microns/ml	*500	25	910	N/A	1,633	0	784	N/A	642.00
ISO	21/21	23/20	27/24	N/A	28/22	19/12	21/21	N/A	26/21
Slope 5/15	.1	.8	1.5	N/A	1.2	1.4	.3	N/A	.88

Note: Numbers marked with "" are straight line extrapolations, not reported counts.

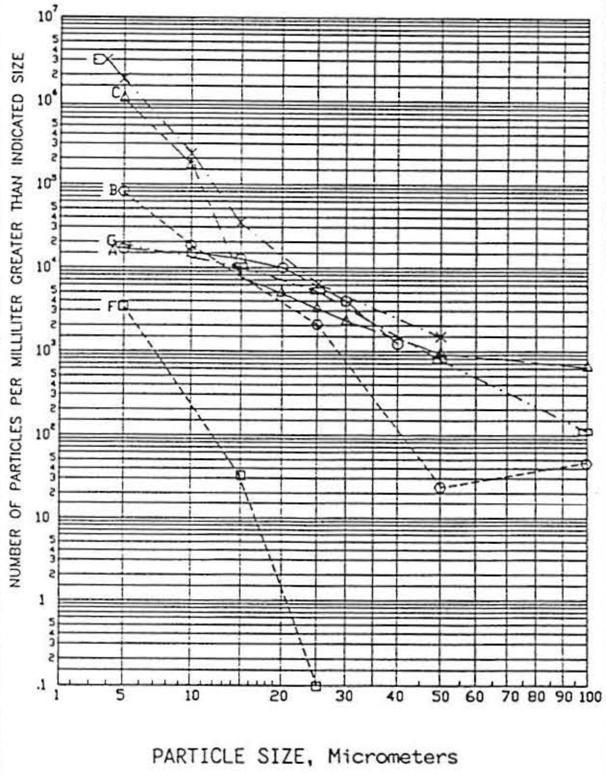
required. Only two labs reported that they diluted the sample. Lab C reported a dilution ratio of 100:1, while Lab E reported a dilution ratio of 1000:1. From the results, it is seen that the higher the dilution ratio, the higher the particle count.

Based on the estimated gravimetric level of the contaminant introduced into sample two, the particle count of Lab E would be close to the true particle count. These two labs (C & E) are also the only ones that use paint shakers for agitation. It is incredible that Lab F understated the concentration by approximately three orders of magnitude (1000X). This is roughly 10 ISO range numbers.

There were also striking differences between particle size distribution slopes among the labs. While Lab A reported a flat slope of 0.1 (between 5 & 15 microns), Lab C reported a steep 1.5 slope. The average for the group is 0.88. The two labs that did dilution reported steep slopes of 1.5 and 1.2 respectively. It would appear that the lack of appropriate dilution masks the presence of high concentrations of small particles. For instance, the variance of particle counts at 15 microns ranged only from 1008 to 5883, when Lab F is not considered.

Figure 2
Sample 2

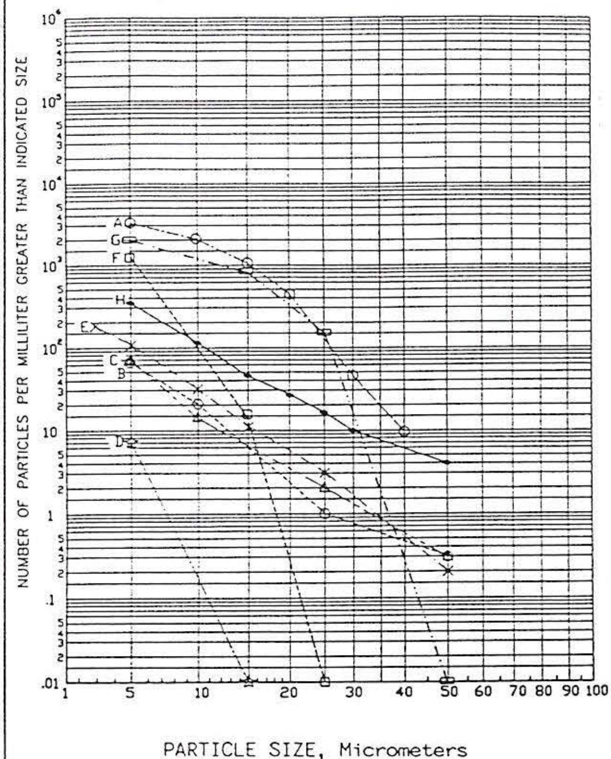
Particle Size Distribution
Test: Gross Metallic Contamination



Labs D and H claimed to be unable to analyze the sample and hence reported nothing.

Figure 3
Sample 3

Particle Size Distribution
Test: 0 > 10 Micron



Analysis of Sample Three Results. (See Figure 3 and Table 6). The comparison of particle counts for sample three was the most puzzling. When this sample was prepared, the inline particle counter showed zero particles greater than 10 microns per ml. However, the data reported by the labs showed a spread of roughly three orders of magnitude (1000X). Only one lab reported the sample to be as clean as our inline counter had indicated. At 10 microns, particle counts ranged from less than one to over 2000 per ml. This variation is equal to about 10 ISO range numbers at 15 microns.

The slopes (between 5 and 15 microns) also varied widely, from 0.3 to 1.3. Insufficient bottle cleanliness may have contributed to the apparent errors. Careless handling of the samples could have also contributed to the problem. Unlike the other three samples, all eight labs reported particle counts on sample three.

TABLE 6
Comparison of Particle Counts - Sample Three

Labs	A	B	C	D	E	F	G	H	Average
>5 microns/ml	3,189	65	63	7	103	1,300	2,020	386	891.62
>10 microns/ml	2,021	21	14	*.16	31	*98	*1,300	111	449.52
>15 microns/ml	1,085	*6	*6	0	12	15	825	46	249.37
>20 microns/ml	448	*2.4	*3.5	0	*5.5	*2.7	*320	*28	101.26
>25 microns/ml	*180	1	2	0	3	0	157	17	45.00
>30 microns/ml	46	*.72	*1.3	0	*1.6	0	*16	*10	9.45
>50 microns/ml	*2.6	.3	.3	0	.2	0	0	*4	.92
ISO	19/17	13/10	13/10	10/7	14/11	17/11	18/17	16/13	17/15
Slope 5/15	.3	.8	.8	2	.7	1.3	1.1	.7	.96

Note: Numbers marked with "" are straight line extrapolations, not reported counts.

Analysis of Sample Four Results. (See Figure 4 and Table 7). Because of the trace amount of moisture present in sample four, Labs A and G declined to perform particle counts. Like samples two and three, extreme variability of the reported particle counts is apparent. Ten micron counts ranged from 88 to 17,000. Twenty micron counts ranged from 0.3 to 8,500; a variance of more than 10 ISO range numbers.

The exact influence of the moisture on this variability can't be assessed. Lab D is the most suspect in terms of counting moisture particles instead of hard particles. This is evidenced from the flat distribution between 5 and 10 microns (Slope of 0.08). Labs B, C, E, and H had typical ACFTD slopes which suggests that maybe they were able to dehydrate the sample before particle counting. This can sometimes be done by simply diluting the sample with clean dry MIL-H-5606. Other techniques are available. Four labs reported that moisture was present in the sample (See Table 8).

In sample four, the moisture level reported ranged from 351 ppm (0.0351%) to 5,000 ppm (10.5%). The calculated amount of moisture added was less than 1,000 ppm (0.1%).

Figure 4
Sample 4

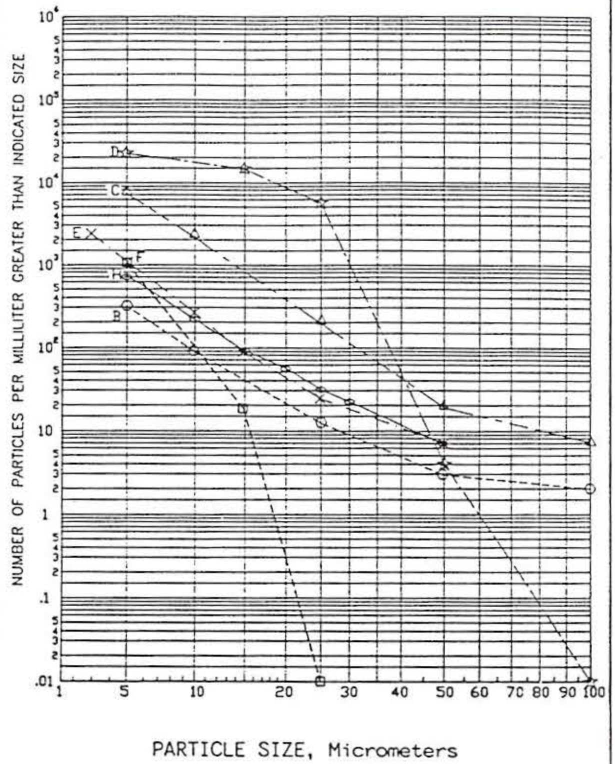


TABLE 7 Comparison of Particle Counts - Sample Four									
Labs	A	B	C	D	E	F	G	H	Average
>5 microns/ml	N/A	318	7,697	21,731	1,236	1,298	N/A	710	5,498.33
>10 microns/ml	N/A	88	2,449	*17,000	276	*100	N/A	206	3,353.17
>15 microns/ml	N/A	*40	*850	15,269	87	18	N/A	98	2,727.00
>20 microns/ml	N/A	*21	*390	*8,500	*42	*.3	N/A	*55	1,501.38
>25 microns/ml	N/A	13	208	5,402	25	0	N/A	34	947.00
>30 microns/ml	N/A	*9	*120	*1,000	*18	0	N/A	*24	195.17
>50 microns/ml	N/A	3	19	4	7	0	N/A	7	6.67
ISO	N/A	15/12	20/16	22/21	17/14	17/11	20/17	17/14	20/19
Slope 5/15	N/A	.7	.7	.08	.7	1.3	N/A	.6	.68

Note: Numbers marked with "" are straight line extrapolations, not reported counts.

TABLE 8
Reported Moisture Levels

Lab	Sample 1	Sample 2	Sample 3	Sample 4
A	--	0.009%	--	0.0568%
D	--	--	--	detected
G	<.03%	<.03%	<.03%	<0.5%
H	0.0086%	0.006%	0.007%	0.0351%

CONCLUSIONS

From the results of this study, it is increasingly difficult to gain confidence in the use of optical particle counters in non-scientific labs. The source of the problem seems to be a combination of the complexity of the instrument, the complexity of the sample preparation procedure, and the non-standard characteristics of field-oil contaminants.

This point is further evidenced by a National Fluid Power Association (NFPA) study involving a round robin study among seven T2.9.6 working group members. The study found a 35% coefficient of variation despite the use of common instruments, calibration method (ISO 4402), and sample materials. Analysis procedures were also controlled. This equated to observed particle concentration differences of nearly a factor of three. Round robins conducted in a German study concluded variability of plus/minus one ISO range number. Unlike the German and NFPA round robins, the labs in this study were unaware of the purpose of their analysis.

Considering the high cost of particle counting, alternate non-optical methods need to be investigated for application with field fluids. Without rapid adoption of improved particle counting methods, progress in the fields of proactive maintenance and contamination control will be hampered.

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