ISO 4406 Testing: Contamination Particles in Oil

AccuSizer® and A2000 CMS

Particulate contamination in various oils (hydraulic, transmission, and other fluid power systems) must be monitored to ensure proper performance and avoid damage to valuable equipment. These measurements are typically made using a liquid particle counter, such as the Entegris AccuSizer®, following the ISO 4406 standard reporting format. This application note describes how the AccuSizer automates these measurements and post-analysis reporting.

INTRODUCTION

Contamination is the single greatest cause of oil degradation. If not controlled or prevented, contamination can cause systems to fail catastrophically. Quantifying fluid cleanliness is often performed using a liquid particle counter, such as the AccuSizer. The sample is analyzed using an optical sensor. Various industry standards[1,2,3] provide means to report fluid cleanliness using code (scale) values associated with concentration at chosen particle sizes. The most commonly used standard is ISO 4406 "Hydraulic fluid power-fluids-method for coding the level of contamination by solid particles".

ISO 4406

Table 1 in ISO 4406 provides scale values as a function of particle concentration (particles/mL). A three number code defines the amount of contamination at 4, 6, and 14 µm. Each time a scale number increases the quantity of particles is doubled. This table and an example of assigning scale numbers is shown in Table 1.

Example:

- 12,000 particles/mL >4 µm
- 2600 particles/mL >6 µm
- 1100 particles/mL >14 µm

ISO code = 21/19/17

More than	Up to/including	Scale number
2 500 000		28
1 300 000	2 500 000	28
640 000	1 300 000	27
320 000	640 000	26
160 000	320 000	25
80 000	160 000	24
40 000	80 000	23
20 000	40 000	22
10 000	20 000	21
5 000	10 000	20
2 500	5 000	19
1 300	2 500	18
640	1 300	17
320	640	16
160	320	15
80	160	14
40	80	13
20	40	12
10	20	11
5	10	10
2,5	5	9
1,3	2,5	8
0,64	1,3	7
0,32	0,64	6
0,16	0,32	5
0,08	0,16	4
0,04	0,08	3
0,02	0,04	2
0,01	0,02	1
0,00	0,01	0

Table 1. ISO 4406 table.

ACCUSIZER

The AccuSizer is an advanced liquid particle counter used for a variety of applications, including ISO 4406 testing. The sensor can be used solely in light extinction mode, or as a combination of extinction + scattering, in order to measure smaller sizes down to 0.5 µm. Pulses from the sensor are sent to a pulse height analyzer (counter) that converts pulses to particle size through the use of a calibration curve. When performing ISO 4406 testing, the sensors are calibrated following the ISO 11171[4] procedure using a secondary reference standard. Samples are transported through the sensor using a variety of sampler fluidics, including an autosampler as part of the A2000 CMS system (Figure 1).

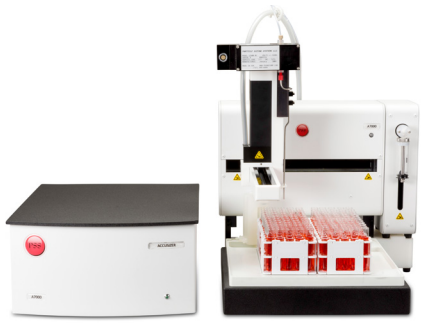

Figure 1. AccuSizer 2000 CMS.

CALIBRATION

The LE-400 sensor is calibrated following the ISO 11171 procedure using a secondary standard based on the NIST standard reference material (SRM) 2806-a traceable particle count standard made from medium mineral test dust suspended in hydraulic oil. An automated calibration procedure within the AccuSizer software facilitates checking, and adjusting the calibration curve, based on the certificate of analysis provided with the secondary standard.

SAMPLE PREPARATION

Most samples are prediluted (often 9:1). The dilution step assures all measurements are below the coincidence error level, as well as lowering the viscosity of samples that might otherwise be over the limit of the system (100 cP). The software uses the predilution factor to perform automatic calculations back to the actual concentration in the sample. The A2000 CMS can accept trays up to 60 samples depending on beaker/tube size, and up to two trays can be loaded onto the system.

MEASUREMENT PROCEDURE

The measurement and tray sampling protocols are defined in the software using the dialog boxes shown in Figures 2 and 3. Each sample can have a unique measurement protocol but typically the entire tray is analyzed using a single protocol. The tray sampling can be organized in any order desired. Both internal tests and evaluations at customer sites have proven that there is no detectable variation comparing the same sample at the beginning and the end of a tray run.

The sample needle lowers into a sample tube. Three replicates (or more) can be performed on each sample tube. Upon completion, the needle and fluidics are cleaned using a two-stage process. The first cleaning reservoir cleans the outside of the needle, while the second reservoir cleans the inside of the needle and fluidics. Clean fluid is flushed through the system and sensor until an acceptable low background count is achieved.

The process is then repeated for the rest of the tray. An individual sample is processed in less than 2 minutes, allowing for hundreds of samples/day throughput.

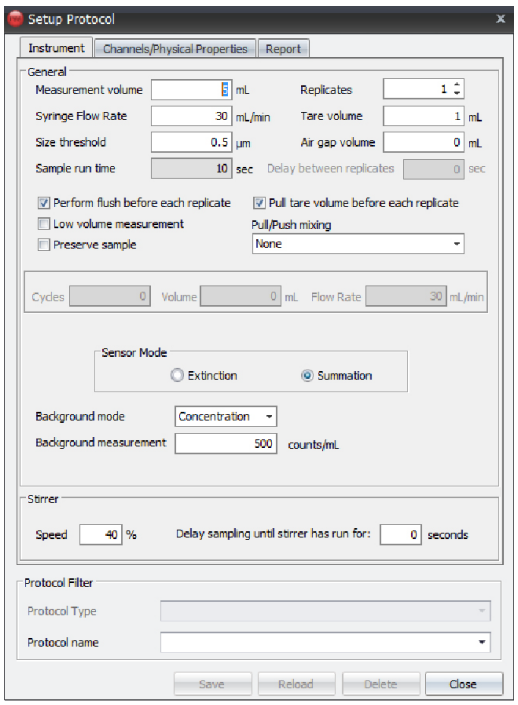

Figure 2. Measurement protocol.

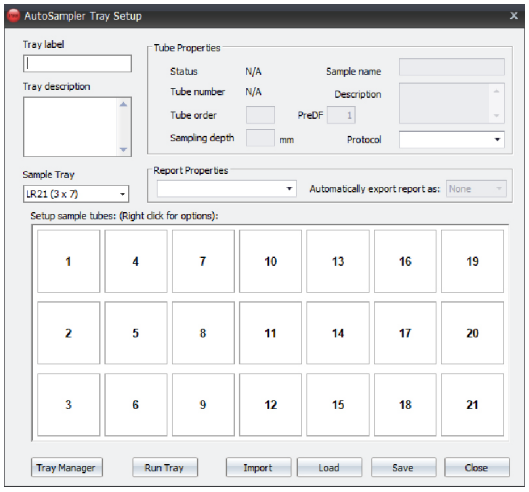

Figure 3. Autosampler tray protocol.

RESULTS

Automated reports providing particle concentration and ISO 4406 scale numbers are printed or sent to a LIMS system. Examples of generated reports are shown in Figures 4 and 5. Figure 4 shows a standard ISO 4406 report for a single sample tube measured three times. The data from Figure 5 shows the particle counts vs. size data for one sample split into four sample tubes, each analyzed three times. The lower two curves show the background counts after clean up between bottles.

Sample	Run Date/Time	> 4 um (#/mL)	> 6 um (#/mL)	> 14 um (#/mL)
Tube 20 Rep. 1	12/13/2016 17:03	671	294	39
Tube 20 Rep. 2	12/13/2016 17:04	764	325	39
Tube 20 Rep. 3	12/13/2016 17:05	750	308	38
	Mean (#/mL)	728	309	39
	Standard Deviation (#/mL)	40.942	12.675	0.471

Classification
17/15/12

Figure 4. ISO 4406 Report format.

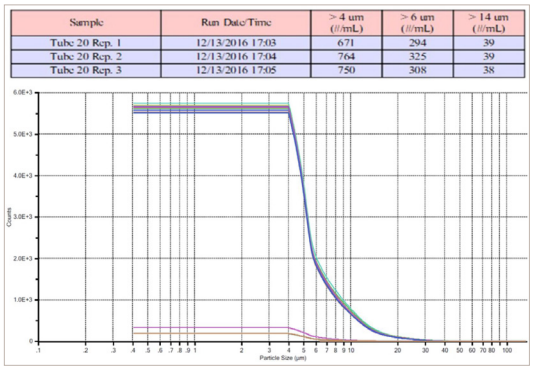

Figure 5. Repeat results from one sample split into four tubes, each measured three times. The lower curves show the background during clean up between tubes.

CONCLUSIONS

The AccuSizer can be used to analyze a wide range of oils to determine the particulate contamination and automatically report in ISO 4406 format. Samples can be analyzed one at a time, in batch mode, or loaded onto a tray for automated analysis of 200 samples/day. The calibration, measurement functions, and reporting is all automated using the AccuSizer software. The software also provides automated reporting according to NAS 1638, NAVAIR, and other contamination standards.

References

[1] ISO 4406 "Hydraulic fluid power-fluids-method for coding the level of contamination by solid particles

[2] NAS 1638 "Cleanliness requirements of parts used in Hydraulic systems"

[3] NAVAIR 17-15E-52

[4] ISO 11171 Hydraulic fluid power-Calibration of automatic particle counters for liquids

FOR MORE INFORMATION

Please call your Regional Customer Service Center today to learn what Entegris can do for you. Visit entegris.com and select the Contact Us link to find the customer service center nearest you.

TERMS AND CONDITIONS OF SALE

All purchases are subject to Entegris' Terms and Conditions of Sale. To view and print this information, visit entegris.com and select the Terms & Conditions link in the footer.

Entegris

Corporate Headquarters
129 Concord Road
Billerica, MA 01821
USA

Customer Service
Tel +1 952 556 4181
Fax +1 952 556 8022
Toll Free 800 394 4083