

# Measuring Gross Water Contamination in Turbine and Industrial Oils with FluidScan and Homogenizer Preparation

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## Introduction

Water contamination in turbine and other industrial oils can be a very serious issue and water testing is always a part of any lubricant condition monitoring program. Turbine oils typically are formulated to have high thermal stability, oxidation resistance, and excellent water separation. Lubricants available specifically for gas turbines or steam turbines are designed with specific additive formulations, but there are also many oils that can work with all different types of turbines. Gas turbines have the tendency to build up sludge and varnish whereas steam turbines may experience oxidation, foaming, and sludge. However, a concern of all turbine systems is water contamination. Severe water contamination can cause changes in the oil's viscosity, accelerated oxidation, additive depletion, and decreased bearing life. Turbine manufacturers typically recommend a warning alarm limit of <1000 ppm.

The most widely accepted method for detecting water in oil is by Karl Fischer (KF) coulometric titration (ASTM D6304)¹. This titration method is somewhat cumbersome, as it requires hazardous reagents, careful sample preparation, expensive equipment, and at least several minutes per analysis. However, Karl Fischer analysis for water can yield highly accurate and repeatable results when executed by a skilled operator and is the comparative method for other analytical techniques for water determination. Also, the water does not have to be fully dissolved in the oil.

The FluidScan can detect the light scattering of water droplets present in oil by a lift in the baseline of the infrared absorbance spectrum. Figure 1 shows several FluidScan spectra of used turbine oil samples with high levels of water contamination.

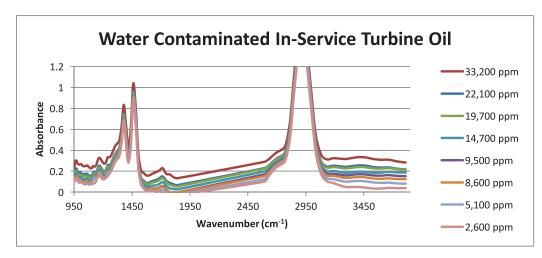


Figure 1. FluidScan spectra of used turbine oil heavily contaminated with water used to monitor a vacuum dehydration process at a power generation plant.

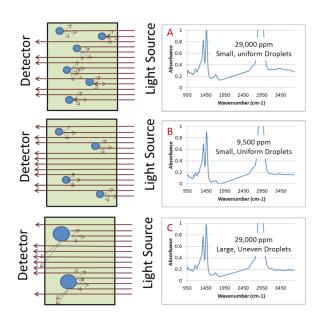


Figure 2. Graphical representation of light scattering in used turbine oil due to varied water droplets. Spectrum A is a used turbine oil with 29,000 ppm water contamination immediately analyzed after homogenization. Spectrum B is a used turbine oil with 9,500 ppm water contamination immediately analyzed after homogenization. Spectrum C is the same sample as in A (29,000 ppm) but has been allowed to sit for 45 minutes after homogenization. The change in concentration and water droplet size is apparent in the degree of baseline lift.

The degree of light scattering caused by a water-in-oil mixture indeed depends on the concentration of water present, but it also is strongly influenced by how the water is physically dispersed in the oil: the number and size of discrete water droplets present in the oil (Figure 2).

For this reason, it is important to have representative, homogeneous sampling. A portable instrument such as the FluidScan can be used at the sampling site for immediate results where the oil and water will be homogeneous due to the turbulent motion inside the instrument. If the samples are left to settle, perhaps during transit to a designated oil analysis site or laboratory, the water will eventually separate (Figure 3). After the water has completely separated from the oil, it is difficult to get accurate measurement of the water content.

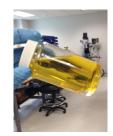


Figure 3. Sample of used Chevron GST 32, as received after shipment from a power generation plant.

### Method

A new water calibration which measures light scattering due to the presence of water droplets is available on the FluidScan for the Industrial Library. The method was developed with water-contaminated samples of several popular brands of turbine and gear/bearing



Figure 4. Homogenizing a sample of watercontaminated oil.

oils for a robust universal calibration of industrial fluids ranging from 1,000 ppm up to 65,000 ppm water. An important component of the method is the use of a homogenizer. The samples were homogenized with a commercially available mechanical homogenizer and allowed to sit at room temperature for two minutes (no more than 30 minutes) prior to measurement on the FluidScan (Figure 4).

#### Results

Sixteen samples between the range of 500 ppm and 10,000 ppm water contamination were used to test the Total Water FluidScan measurement against Karl Fischer D6304. Each sample was prepared by homogenizing them for 30 seconds on high prior to analysis. They were measured simultaneously on three FluidScans and by Karl Fischer to minimize the effects of sampling errors. The results are shown in Figure 5.

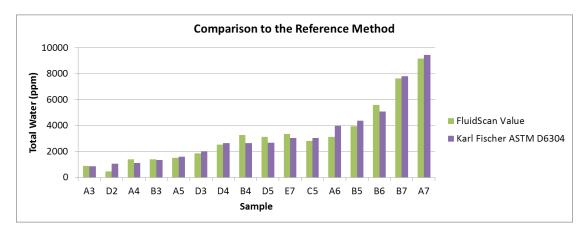


Figure 5. Comparison of the new total water measurement on the FluidScan to ASTM D6304 Karl Fischer titration method.



Figure 6. Samples shown after being homogenized for 30 seconds on high.

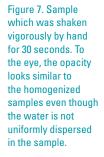
To demonstrate the importance of the homogenizer in the determination of industrial fluids which are designed for excellent water separability, a test set comprised of 13 in-service Chevron GST 32 oil samples from a power generation plant were analyzed with and without proper homogenization.

GROUP A: Samples were homogenized for 30 seconds on high (Figure 6). Before analysis, the sample bottles were gently inverted 20 times to mix.

**GROUP B:** Samples were shaken vigorously by hand for 30 seconds (Figure 7) and then left to sit for several minutes to allow air bubbles to dissipate. Before analysis, the sample bottles were gently inverted 20 times to mix.

A plastic disposable pipette was filled from the middle of the bottle, and the same aliquot was used to dispense fluid into KF vials and onto the FluidScan flip-top cell. The results are shown in Figure 8.

Clearly, the sample preparation method has a large effect on the results. All samples prepared only with vigorous hand-shaking (Method B) had unacceptably large errors, and in fact, never measured higher than 6,000 ppm water on the FluidScan. Even though the hand-shaken sample appeared opaque, similar to the homogenized samples, a handshaken mixture of oil with water is not truly homogenous. For at site analysis, a fresh oil sample measured immediately at the sampling site should be homogeneous with uniform water droplet size from the turbulence and shearing inside the machine.



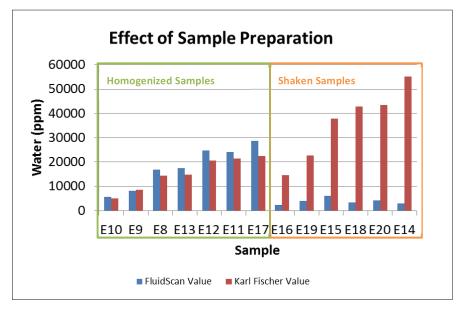


Figure 8. The samples prepared with a homogenizer showed great agreement between the calculated water concentration on the FluidScan and Karl Fischer result. The samples that were shaken by hand were not accurate.

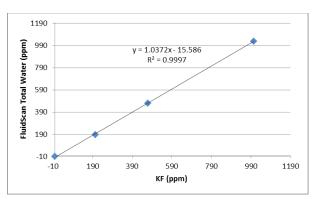


Figure 9. Average water result for each Mobil DTE 732 sample over 4 days obtained on Unit 1.

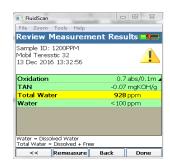
In turbine oils specifically, the critical limit for water contamination is in the range ~ 500 ppm. Because the measurement is sensitive to baseline drift, in order to provide the needed precision in this range there must be great care taken in backgrounding the instrument prior to measuring each sample. At a minimum, a fresh background should be taken before every measurement. Also, some cleanliness thresholds that look for the presence of residual oil in the cell should be implemented. It may also be beneficial to take an "air" background as well as an "empty cell" background.

Measurements of Mobil DTE 732 samples with increasing amounts of water contamination present were taken on multiple spectrometers

over several days. Samples were measured in randomized duplicates

on each unit on each device, homogenizing the sample for 60 seconds each time prior to measurement on the FluidScan and by KF. The average results obtained over the 4 days of testing for one spectrometer, Unit 1, demonstrates the excellent linearity of response for the method (Figure 9).

Detailed results for all measurements taken on all five FluidScan units are shown in Table 1. On each individual unit, the repeatability over the several days of testing was excellent, on average 100 ppm (1\*STD). The method performed well on all five units with reproducibilities of <150 ppm (1\*STD) for each measurement. It's clear to see that actionable information can be obtained in the range of 500 to 1000 ppm.



FluidScan measurement result for a turbine oil with severe water contamination.

Dr	y Measuremen	. 9	Sample A, KF = 0 ppm					Sample B, KF = 205 ppm					Sample C, KF = 470 ppm					Sample D, KF = 1005 ppm				
De	y ivieasuremen	Unit 1	Unit 2	Unit 3	Unit 4	Unit 5	Unit 1	Unit 2	Unit 3	Unit 4	Unit 5	Unit 1	Unit 2	Unit 3	Unit 4	Unit 5	Unit 1	Unit 2	Unit 3	Unit 4	Unit 5	
1	1	0	158	0	0	94	244	244	297	270	240	343	428	506	390	536	1139	1038	1164	1223	1061	
	2	0	0	0	78	76	136	167	324	297	163	481	470	437	400	103	1060	1081	1204	1299	1117	
,	1	9	239	85	156	66	183	201	286	122	233	494	404	561	601	423	915	909	1281	1134	1098	
	2	71	27	207	213	0	160	264	183	373	200	705	496	808	675	657	942	962	1075	1092	1156	
-	1	101	0	82	85	23	170	0	279	276	91	238	110	409	353	394	988	823	1020	1212	857	
3	2	0	0	50	67	0	234	141	83	312	298	428	387	621	603	354	1132	1079	1056	1253	1143	
	1	0	0	0	0	80	36	0	130	115	0	309	348	436	457	453	941	655	1007	906	749	
	2	78	54	131	195	127	61	33	339	107	109	505	345	416	536	466	841	617	1059	1023	894	
	AVE	33	60	69	99	59	153	131	240	234	167	438	373	524	502	424	995	895	1108	1143	1010	
	STDEV	41	84	69	77	43	74	107	96	104	96	145	119	137	118	160	107	183	97	131	154	

Table 1. Results from multiple days of testing samples of Mobil DTE 732 with varying water contamination levels are shown here.

### Conclusion

The new FluidScan method for analysis of water contamination in turbine oils is a robust, reliable method capable of providing immediate alert of severe water contamination. The largest contributor to the variation is the sampling. Hand-shaking is not sufficient for obtaining a homogeneous sample and reliable results for water measurement on the FluidScan. Immediate analysis at-site or the preparation of samples prior to analysis with a commercially available homogenizer is recommended for the best results. With best practice sampling technique, results correlating within 20% to Karl Fischer can be achieved. The new FluidScan water calibration provides accurate determination of the total water contamination in 90% of the industrial library for >300 ppm water in turbine oils and >1000 ppm water in other oils.

