DETERMONATION OF PARTICLE CONTAMINATION IN OIL BY MICROSCOPE – A SIMPLIFIED APPROACH

By Shankar Periketi, *Officer (Chemistry), NTPC, RGPPL Site.*

ABSTRACT:
Analysis procedure for the Determination of Particulate Contamination by the Microscopic Particle Count method is simplified by introducing a Web Cam at the eye-piece of the microscope and observing particles on computer display with magnification. This is a simple technique for observing particle contamination without having eye strain. Such counting method increases accuracy in analysis and saves analysis time significantly. The image can be saved and retrieved/ reproduced at any point of time. The method is compared with automatic particle count analyzer also.

INTRODUCTION:
At RGPPL, Microscopic particle counting method used for EHC (Electro hydraulic control) fluids as per guideline of GE (GEK 46357E[1]). The high pressure hydraulic control system provides accurate steam valve control, over speed protection, and safety against fires through the use of a synthetic phosphate ester fluid as a working medium.

IMPORTANCE OF PARTICLE CONTAMINATION DETERMINATION:
Electro Hydraulic Controls (EHC) Valves are responsible for governing steam supply to turbine generators. Minimizing particulate contamination in EHC Oil is important at all times for control valves. Control valves are particularly sensitive to abrasive wear and erosion from particulates; leading to loss of system sensitivity and control. Particulate contamination may also impact the resistivity of the fluid causing further erosion of the valve components. Since particle contamination is detrimental in smooth operation of valves, it is imperative to determine particle contamination in the fluid, periodically.

As per GE, particle count limit w.r.t different sizes for operating EHC fluid, in 100 ml sample are given as, 5-10 micron size -24000, 10-25 micron size- 5360, 25-50 micron size-780, 50-100 micron size-110 and greater than 100 micron size- 11.

For Turbine Oil, the cleanliness of oil plays an important role for proper functioning of cooling, movement, lubrication and cleaning. The clearance in the bearing is too low in the tune of 5 micron or so. So the oil in the system should be clean enough not to damage this clearance and to disturb the bearing performance. NAS/ISO values indicate the cleanliness level of oil.

PAERTICLE COUNT ESTIMATION:
A brief outline of particle count/contamination estimation of hydraulic fluids, as per GEK 46357E is as follows:

1. Each item of filtration apparatus will be cleaned before each run of samples and ensure no other contamination from any other source should not incorporate throughout entire filtration.
2. Thoroughly agitate the oil sample (100ml) bottle to ensure that all solid particles are in suspension.
3. Remove the filter cap and pour oil sample into the filter funnel. Replace the filter cap. Pour 100 ml of pre filtered petroleum ether (0.45 micron) into the sample bottle, agitate it and pour it in filter funnel.
4. Rinse the funnel walls with 50 ml filtered petroleum ether and filter this sample by applying vacuum through 0.45 micron grid membrane filter paper.

5. After one half of filtration complete, carefully wash down funnel wall with filtered petroleum ether (50 ml) and apply vacuum until filter paper is complete dry.
6. Place this filter paper carefully under the microscope for counting particles according to procedure given below. Each grid square of filter paper is equal to 1/100th of the total effective filtering area.
7. A blank analysis is to be performed on 200 ml of filtered petroleum ether prior to each sample analysis (The maximum particle count value of a blank analysis shall be no greater than 10% of the count of an acceptable sample for a specific laboratory).
8. Fibers are counted as particles and not differentiated unless their length exceeds 100 microns [100µm].
9. Particles on each grid are to be counted & tabulated as particles >100µm, 50-100µm, 25-50µm, 10-25µm and 5- 10µm.

For obtaining the number of particles of a given particle size range, the number of particles on a representative number of grid squares on the filter disc are counted manually through the eye-piece. From this count, the total number of particles, which would be present statistically on the total effective filtration area of 100 imprinted grid squares, is calculated as detailed in Table-2.

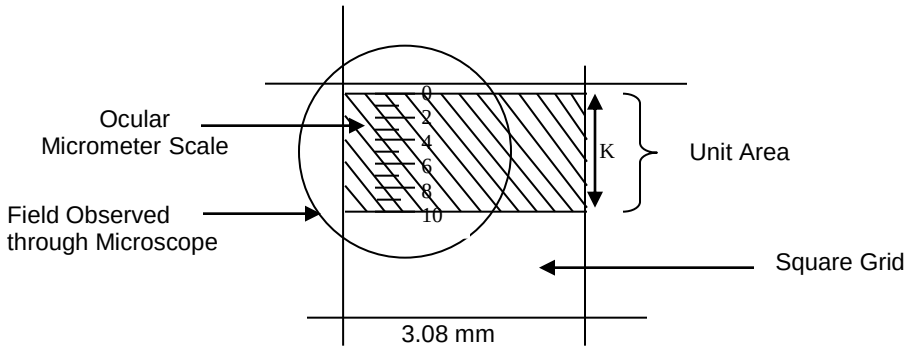
No. of particle on filter paper of given size range estimated between	No. of Unit Area/grid to be counted	Multiplication factor for total statistical particle count of entire effective filtration area
1-50	Entire filtration area	1
50-1000	20 randomly chosen square grids	5
1000-5000	10 randomly chosen square grids	10
>5000	At least 10 randomly chosen Unit areas	Calibration factor 'F' calculated as below

Table-1: Particle count estimation

$$F = \text{Total effective filtration area} \quad \frac{(960)\ mm2}{3.08\ mm \times N \times L\ (mm)} = \frac{312}{N \times L}$$

N = no. of basic unit areas counted

L = The calibrated length of the ocular micrometer scale or portion of scale used in defining unit area in mm (K/magnification).

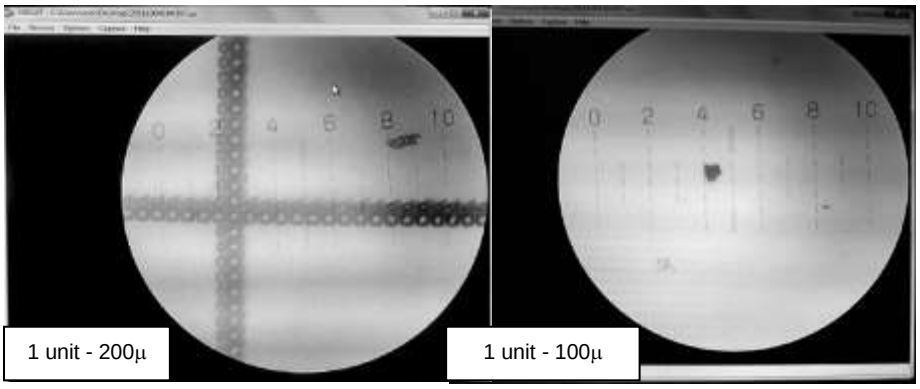


Pic-1: Unit area

RGPPL Chemistry lab is equipped with Olympus Microscope (model BX 60) with mechanical Stage, 5X, 10X, 20X and 50X magnifications and ocular micrometer with 10 division scales. The Ocular micro meter accompanied with thumbscrew (divided into 100 equal divisions) for accurate particle measurements.

COUNTING PROCEDURE OF PARTICLE:

1. Carefully Place the filter paper on stage of microscope and check for uniform distribution of particles over filter paper by observing at 5X magnification.
2. Keep the microscope at 5X magnification. Check for >100µm particles. The total number of square grids/unit areas to be counted for this size range is obtained from Table-1 and the results are multiplied by respective multiplication/Calibration factor (Table-1) to get total particle count of >100µm particle size range over entire effective filtration area. At 5X magnification 1 unit of ocular scale corresponds to 200µm and it is shown in Pic-2 left side.



Pic-2: At 5X (Left) and 10X (Right) magnifications

3. After counting >100µm particles, change to the 10X magnification and observe the 50-100 micron particles (Pic-2 right side). Follow the same procedure as given above to get total particle count of 50-100µm range. At 10X magnification, 1 unit of ocular scale corresponds to 100µm.
4. Now change to 20X magnification mode and observe the 25-50µm particle. Follow the same procedure as given above to obtain total particle count of 25-50 and 10-25 micron range. At 20X magnification, 1 unit of ocular scale corresponds to 50µm.
5. The particles of 5-10µm range are counted at 50X magnification as above. At 50X magnification, 1 unit of ocular scale corresponds to 20µm.
6. Finally, the results are tabulated as in Table 2.

MODIFICATION AND EXPERIMENTING WITH LUBE OILS:

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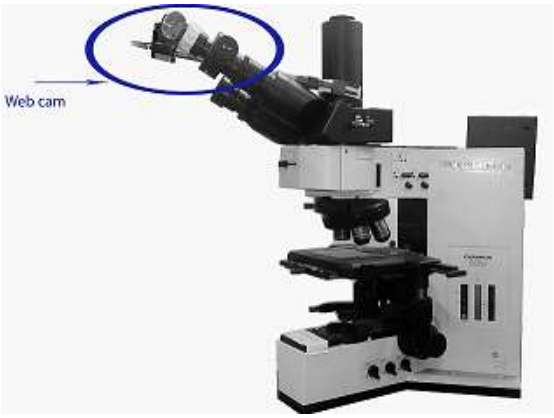
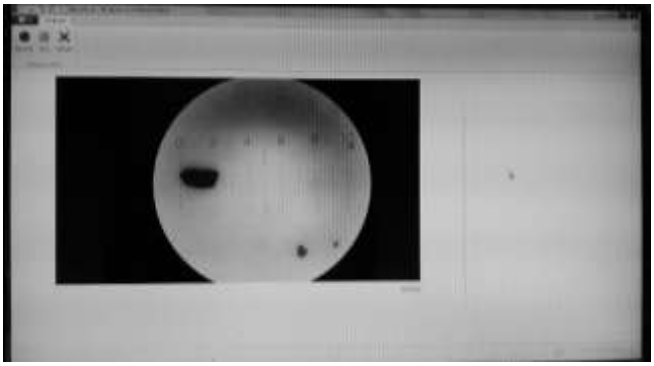


Pic-3: Microscope with Web Cam

A HD Web Cam of 2.1MP (Video) was fixed at the eyepiece of microscope (Pic-3) and connected to the computer display. The magnified filter paper area with ocular micrometer scale is displayed on the computer screen. This enables to determine the size of particle on computer. Now complete counting can be done using image on computer screen by adjusting stage of microscope.Pic-4 showed the magnified display of membrane filter paper with contamination on computer screen, when observed at 10x magnification of microscope.



Pic-4: 10X Magnified view of particle on Computer Screen

The physical observance of particles in oil can also distinguish the metallic impurities, which can be due to the wear and tear of bearings through their shininess.

This method was extended for the determination of contamination in lube oil samples and found useful in arriving the NAS values. Two different samples of turbine oils (one of ST and another of GT) were taken. Each sample was analyzed three times and the report is tabulated as A,B &C. Good consistency was reported. Below data shows Particle Count in two different lube oil samples with three sets per each sample.

	Sample-1			Sample-2		
Size	**A**	**B**	**C**	**A**	**B**	**C**
>100μm	3	5	5	7	4	5
50-100μm	70	50	51	85	76	80
25-50μm	650	675	620	635	620	650
15-25μm	690	680	660	700	685	690
5-15μm	16692	15600	14976	16848	16536	17004
NAS	7	7	7	7	7	7

Table-2: Particle count in lube oil samples1 & 2

The values so obtained were compared with that from Parker Make Laser CM20 automatic particle count analyzer. The values for 5-15μm range found to differ heavily with CM20 analyzer showing lower counts. The results for Sample-1 and Sample-2 above are as below in Table-3:

	Sample-1	Sample-2
Size		
>100μm	2	3
50-100μm	33	61
25-50μm	250	250
15-25μm	465	593
5-15μm	3100	3521
NAS	5	6

Table-3: Analysis result with Laser CM20

The microscopic method above, however, is time consuming as care should be taken during filtration to avoid any contamination and ensure uniform distribution over the filter paper. The filtration takes around 2 hours for one sample and counting takes around 5 hours. Also if the oil contains varnish or is opaque, this method cannot be used as the particles will not be visible clearly.

ADVANTAGES OF USE OF WEB CAM:

1. The use of Web Cam reduces the analysis time significantly compared to counting through eye-piece.
2. With Web Cam, Counting analysis becomes more accurate
3. One can capture picture of particle at magnification while counting, can be stored in soft form for future reference.
4. One can do the counting without any eye strain.

Use of manual counting can point to the wear and tear of bearings by observing the metal particles as against the automatic analyzer.

CONCLUSIONS:

Though slightly time consuming, the microscopic method is found to give a good idea of the particle contamination in hydraulic and lube oils. Estimation can be done as per NAS and ISO methods. Type of particles especially wear debris can also be distinguished from their shininess. Introducing the web cam adds to the advantage of easy counting, less eye-strain and record keeping. When oil sample contain high level of moisture content and entrapped air bubble, automatic particle counter may not give the accurate results but manual counting gives accurate results.

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REFERENCE [1]: GEK 46357 E, Revision E Oct, 1990, Steam Turbine–Generator EHC Fluid Specifications and Maintenance.