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Determination of Size and Concentration of Particles in Oils

Beckman Coulter Application Bulletin

Multisizer™ 3 COULTER COUNTER®

The concentration and size distribution of particles in an oil sample may be measured using the Coulter Principle also known as the Electrical Sensing Zone (ESZ) method. A suitable organic electrolyte solution is chosen to perform the analysis. The sample is prepared by dissolving a certain volume of oil in the electrolyte and analyzing it using a Beckman Coulter Multisizer 3 to determine the size distribution and concentration for the particles present in the oil. If the oil being tested is not soluble in the electrolyte, it is necessary an intermediate step involving the use of a solvent capable of dissolving the oil and then from this solution a sample is taken and dispersed into the electrolyte. A blank run is subtracted from the sample. The results are then reported as number of particles per milliliter for the desired size range.



DETERMINATION OF SIZE AND CONCENTRATION OF PARTICLES

SIGNIFICANCE

The International Standard ISO 4406 defining the Solid Contaminant Code for Hydraulic Fluids and Lubricants classifies the oils according to their particle content in the following ranges:

Particles /ml	≥ 4 μm
	≥ 6 μm
	≥ 14 μm

This is the most widely used range to analyze the particle content in oils. A 100 μm aperture tube (2 μm to 60 μm range) will be suitable. For a different analysis range other aperture tubes may be used.

3. Instrument Set up and Calibration

Select the size of the aperture tube appropriate for the analysis size range. The linear dynamic range for any aperture is 2% to 60% of its size, i.e. a 100 μm aperture tube will be capable of analyze the particle concentration and size distribution from 2 μm to 60 μm. Set up and calibrate the instrument according to the Multisizer 3 Operator's Manual. For determining particle concentration the control mode for the instrument must be Volumetric Mode. Select any volume up to 2000 μL; the volume required for the analysis has to be determined experimentally depending upon the state of the oil and the length of the analysis.

PROCEDURE

4.1 Preparation of electrolyte solution

A 2% solution of NH₄SCN (Ammonium Thiocyanate) in Isopropyl alcohol (IPA) is suitable for the majority of oils. Prepare the electrolyte by dissolving 20 g NH₄SCN in 1.0 L of Isopropyl alcohol. Filter the solution using a 0.45 μm alcohol-compatible membrane filter.

4.2 Sample preparation

If the oil being tested is not soluble in the electrolyte, it is necessary an intermediate step involving the use of a solvent capable of dissolving the oil and then from this solution a sample is taken and dispersed into the electrolyte.

4.2.1 Oils soluble in Isopropyl alcohol

Measure exactly 20 mL of electrolyte solution into a 20 mL Accuvette® II. Pipette 2.0 mL of oil into the electrolyte, this quantity may be different according to

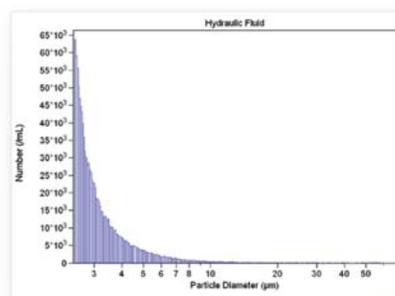
the state of the oil. Stir gently to dissolve thoroughly without creating bubbles.

4.2.2 Oils not soluble in Isopropyl alcohol

Measure exactly 10 mL of Methyl Isobutyl Ketone (MIK) into an appropriate size glass flask. Pipette 2.0 mL of oil into the flask containing the MIK, this quantity may be different according to the state of the oil. Stir gently to dissolve thoroughly without creating bubbles. Measure exactly 20 mL of electrolyte solution into a 20 mL Accuvette® II. Pipette 2.0 mL of the MIK-oil mixture into the electrolyte. Stir gently to dissolve thoroughly without creating bubbles.

4.3 Entering the sample information in the Multisizer 3 Software

Enter the required sample information in the software, i.e. dilution factor, analytical volume and volume of oil used to prepare the sample. By entering the sample information, the software calculates the concentration of particles in the original oil.



Analysis of a Hydraulic fluid sample

Particle Diameter (µm)	Number /ml >
4	103509
6	12333
14	13745

4.4 Sample Analysis

Prepare a blank by following the same procedure used according to the kind of oil to be analyzed but without adding the oil. Run the blank and set the software to automatically subtract it from all the subsequent runs. Run the oil samples.

REPORTING THE RESULTS

Results can be represented as the entire size distribution, total number of particles or number of particles above, below or within selected size categories in the range of the size distribution by using the Interpolation feature in the Multisizer 3 Software.

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