AEROSPACE RECOMMENDED PRACTICE

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PROCEDURE FOR CALIBRATION AND VERIFICATION OF LIQUID-BORNE PARTICLE COUNTER: AN ABSOLUTE STANDARD

1. SCOPE

This ARP describes procedure for determining the counting and sizing accuracy for an automatic liquid-borne particle counter (APC). This procedure is used in conjunction with a suspension of precisely sized spherical particles.

2. PURPOSE

2.1 Reliable and accurate performance of many devices requires that lubricating, cooling or power transmission be accomplished by liquid flow. If the liquid contains particulate material whose size is so large or in concentration sufficient to interfere with operation of the device, then performance is degraded. Accurate measurement of the number and size of particles in such a liquid stream must be carried out. The interpretation of data from an automatic particle counter is based on the reliability of the instrument for both counting and sizing the particles that may be passed through it.

2.2 In applying this ARP, care is required to make sure that good sampling is accomplished from a well mixed volume of precision sample suspension and that no contaminants are introduced in the sample acquisition, storage or handling processes; these precautions are necessary both for the test and operation of the automatic particle counter.

CAUTION: Introduction of miscellaneous contaminants during the test will cause errors. It is necessary to ensure that this does not occur. Calibrations should be carried out in a clean room (FED-STD-209B Class 10,000 or better); a horizontal or vertical flow bench of adequate size may be used.

3. INTRODUCTION

3.1 The particle size parameter that is reported here is the equivalent optical diameter based on the projected area of a spherical particle of refractive index different from that of the liquid. The maximum diameter that can be measured is determined by the dynamic range of the instrument being used and by the smallest aperture in the liquid flow path.

3.2 The concentration and size distribution of particles vary widely. Furthermore, the difference in optical, electronic and sampling systems of the various counters are reasons to do all calibration within the operating specifications set down by the manufacturer.

4. DEFINITIONS

4.1 Equivalent Optical Diameter: The particle diameter reported by an optical particle counter is based on calibration data for the particular instrument. Normally the diameter reported is that of a sphere of equal projected area and shape factor as well as optical properties.

4.2 Agglomerate: Two or more particles which are in intimate contact and cannot be separated by gentle stirring and the small shear forces thus generated.

4.3 Particle Concentration: This is the number of individual particles per unit volume of liquid.

4.4 Primary Calibration: Primary calibration is calibration performed using reference particles of known size and physical properties.

4.5 Secondary Calibration: (Standardization) Secondary calibration is an electronic calibration performed using the reference system built into the instrument.

4.6 Referee Particles: Referee particles must be styrene polymer of uniform diameter, with no more than 5% standard deviation in any size range. The specific gravity should be 1.05 @ 25°C and a refractive index of 1.59 @ 25°C. Particles must be the same color in all size ranges for any calibration, and must be traceable to the National Bureau of Standards.

4.7 Minimum Diameter: The minimum diameter that can be effectively measured will be stated in the instrument's specifications.

4.8 Coincidence Error: Coincidence error is a statistical function of particle concentration and of the sensing zone volume. A typical coincidence error is 5% for a specified maximum particle concentration introduced into the particle counter. Coincidence is defined as the probability that one or more particles will be present in the optical sensing zone at any one time, and one may block the other.

4.9 Maximum Flow: The maximum flow rate is given in instrument specifications and the accuracy of calibration depends on maintaining flow rate within specification.

4.10 Saturation Level: The saturation level or maximum counting rate of the electronic counting circuitry will be specified by the manufacturer and should always be higher than the counting rate at the specified maximum concentration, and maximum flow rate of section 4.9.

4.11 Precision Clean Liquid: Precision clean liquid is defined for this calibration as liquid having a maximum of 10 particles in the 5-15 micrometre range and 0 particles in all larger size ranges per 100 mL sample.

4.12 Dynamic Range: The dynamic range of an instrument is that dimension of smallest size particle compared to largest size particle that can be counted in the individual channels or total instrument; e.g., a dynamic range of 2.0 to 1200 micrometres would yield a ratio of 60:1.

5. APPARATUS

5.1 Automatic Liquid-Borne Particle Counter (APC): This instrument should be in good operating order in accordance with manufacturer's specifications. A procedure for passing a controlled volume liquid sample at a rate specified by the manufacturer should be included. Sample system should be fully charged with precision cleaned liquid before calibration count begins.

5.2 Sample Containers: Sample containers should be glass bottles with screw caps, gasketed with compatible polymer liners. Nylon-6, mylar or polypropylene have been found suitable.

5.3 Dispersing System: An ultrasonic cleaner rated at 2-1/2 - 3 watts per square inch is recommended to disperse and de-agglomerate particles to be suspended.

5.4 Vacuum Source: A suitable vacuum source is required for reducing pressure on samples to remove bubbles.

5.5 Clean Containers: Same level as noted in paragraph 4.11.

6. PREPARATION OF CALIBRATION SAMPLES

CAUTION: Particular care should be taken to ensure that the calibration sample is not affected by either introduction of miscellaneous debris from the environment, or malfunctions of the APC system itself. All operations should be carried out in a clean environment. A Class 10,000 clean room is ideal, but a Class 100 laminar flow bench of adequate size is acceptable.

6.1 Samples are of two basic forms:

a) Sizing
b) Counting

6.2 a) Sizing samples must be referee particles as defined in 4.6.
b) Counting samples must be a mixture of precisely counted referee particles (4.6) in precision clean liquid (4.11). Use of "microbeads" mounted on a glass slide with a soluble (calibration liquid) carrier strip facilitates this operation.

6.3 Appropriate samples should be introduced into approximately 100 mL of precision clean liquid (4.11) without adding any contamination.

6.4 Mix the sample well to ensure uniform distribution of particles. It is recommended that the dispersing system defined in 5.3 be used.

6.5 Air bubbles, which may be counted as particles, should be removed by exposing the sample to reduced pressure for two minutes immediately before passing through the APC. The recommended pressure should be saturation vapor pressure for the liquid + 20% in absolute pressure units.

6.6 Limit the concentration of particles in the liquid to a level where coincidence errors are insignificant. Refer to APC manufacturer's specifications for coincidence error and particle concentration limits as well as flow rate.

7. PROCEDURE

7.1 Sizing Accuracy:

7.1.1 Select the referee particle sample of interest and mix with precision clean liquid to make suspension within concentration defined in 6.6.

7.1.2 Perform the manufacturer's recommended pre-operation warm-up and field standardization procedures on the particle counter under test.

7.1.3 Using a series of 100 mL batches of precision clean liquid, flush the particle counter and its feeder system 5 times. (The flush liquid may be recovered and cleaned for later use). A particle count as defined in 4.11 should be obtained at the last flush.

7.1.4 Pass a sample of at least 100 mL of the suspension prepared in 7.1.1 through the automatic particle counter.

7.1.5 As the sample is passing through the counter, observe the counting rate. Then adjust the lower threshold setting upward until the counting rate approaches zero. At this point adjust downward until the initial counting is reestablished.

7.1.6 Record the MV level or calibration number depending upon the APC being calibrated.

7.1.7 Repeat 7.1.5 and 7.1.6 for each particle size of interest.