Institute of Environmental Sciences and Technology

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Contamination Control Division
Recommended Practice 042.1

Sizing and Counting of Submicrometer Liquid-borne Particles Using Optical Discrete-particle Counters

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1 SCOPE AND LIMITATIONS

1.1 Scope

This Recommended Practice (RP) addresses the sizing and counting of submicrometer liquid-borne particles using optical discrete-particle counters with a focus on applications in the semiconductor, flat-panel display, and data storage industries. Topics covered include the following:

- Overview of light-scattering technology
- Liquid particle counter (LPC) instrument types
- Data interpretation
- Coincidence level or maximum concentration limit
- Particle-size detection limit
- Bubble issue
- Refractive index effect
- Flow rate
- Calibration verification
- Instrument noise and false counts
- Practice of minimizing sensor contamination
- Sensor correlation
- Particle counting statistics

1.2 Limitations

This RP does not address the measurement and identification of living organisms, e.g., bacteria.

This RP does not address the measurement of extremely high particle concentrations, e.g., chemical-mechanical polishing (CMP) slurry.

This RP does not address the calibration of LPCs.

2 REFERENCES

The cited editions of the following documents are incorporated into this Recommended Practice to the extent specified herein. Users are encouraged to investigate the possibility of applying the most recent editions of the references.

2.1 Reference Documents

IEST-RD-CC011.2: A Glossary of Terms and Definitions Related to Contamination Control

ISO 21501-2:2007 Determination of particle size distribution—Single particle light interaction methods—Part 2: Light scattering liquid-borne particle counter

2.2 Sources and Addresses

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3 TERMS AND DEFINITIONS

The following terms have special meaning in the context of this RP. See also *IEST-RD-CC011*.

capillary (also flow cell)
The conduit through which sample fluid passes through the illuminating path. NOTE: The entire flow path may not be illuminated. See *LPC, volumetric*, and *LPC, non-volumetric*.

channel (also size bin)
A bin for recording counts of particles within a specified size range. Channels are established by means of voltage thresholds that match particle light-scattering intensity to particle size.

coincidence
The simultaneous presence of two or more particles in the sensing volume of the instrument, causing the instrument to interpret the combined signal erroneously as resulting from one larger particle.

coincidence level (also maximum particle number concentration)
The particle concentration — specified by the manufacturer of the LPC — at which the error due to coincidence is 10% or less. (Manufacturers may specify concentration limits at error levels other than 10%.)

counter
A type of LPC typically having several channels (see section 5.2.1). This term is also used generically to refer to particle measurement instruments, as in "liquid-borne particle counter."

counting efficiency
The ratio of the measured result of a light-scattering liquid-borne particle counter to the measured result of a reference instrument using the same sample (see ISO 21501-2: 2007).

false count rate (also false count, noise, noise rate, zero count rate)
The maximum particle count indicated by a particle counter, in a given volume, that is sampling particle-free liquid. This value is specified by the LPC manufacturer.

flow rate, effective (also actual measured sample flow rate)
The flow rate of the introduced sample that is illuminated within the viewing volume region. Only particles passing through this region will be detected. Effective flow rate is used to determine particle concentration.

flow rate, sampling
The volumetric rate at which a sample liquid passes through the particle sensor. Typically, particle sensors are calibrated at specific sampling flow rates. For liquid particle counters, units are typically expressed in milliliters per minute (mL/min).

liquid-borne particle counter (LPC)
A type of optical particle counter that uses light to count and size particles suspended in a liquid medium by detecting light scattered by discrete particles.

LPC, volumetric
A type of LPC in which the laser beam illuminates the entire sample volume to measure particles using a uniform intensity of laser beam shape.

LPC, non-volumetric
A type of LPC in which the laser beam illuminates only a portion of the sample volume to measure particles.

monitor
A type of LPC that typically has poor resolution and few channels but high sensitivity.

optical equivalent size
The equivalent size of particles, as reported by an LPC, that would scatter the same amount of light into a defined solid angle element as the spherical reference particle.

particle-size detection limit
The smallest size at which the counting efficiency is 50%. This definition only applies to spectrometers and counters.

particle size distribution (PSD)
A list of values (typically a number of counts) or a mathematical function that defines the relative numbers of particles present, sorted according to particle sizes or size ranges.

refractive index contrast
The ratio of the refractive index of a particle to the refractive index of the suspending medium.

sample time
Typically, a selectable period during which particles are counted and sized.

size resolution
A measure of the ability of an instrument to distinguish between particles of different sizes, typically expressed as a percentage at a specified particle size, referring to the ratio of size variance at that size to the mean size.

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spectrometer
A type of LPC used to measure PSD; spectrometers provide high resolution and many channels.

volume, effective sampling
The portion of the sample that has passed through the sensing zone of the particle sensor. Effective sampling volume = sampling volume × viewing volume %.

volume, sampling
The total volume of liquid that has passed through the particle sensor constituting a discrete sample measurement.

volume, viewing (%)
The portion of the sample flow path, expressed as a percentage, that is illuminated by the incident illumination of the particle sensor.

4 BACKGROUND AND PURPOSE

Liquid-borne particle measurement in the semiconductor industry involves many technical challenges, which, if not handled properly, can substantially affect the measurement results in both sizing and counting of particles. Research work and technical papers have addressed the technical challenges individually. What is lacking is a single source that covers a broad range of those challenges and solutions and provides a handy reference for professionals in this field. This RP is intended to fill that gap.

5 LIQUID-BORNE PARTICLE MEASUREMENT—CHALLENGES AND RECOMMENDED PRACTICES

5.1 Overview of light-scattering technology

A liquid-borne particle counter (LPC) is an instrument that uses light to detect particles suspended in a liquid medium. Particle detection technology using light sources involves measurement by either light-scattering or light-extinction method. *Light-scattering technology* directly measures the amount of light scattered by particles to obtain information on the concentration and the size distribution of those particles. LPCs employing light-scattering technology are predominantly used to detect low concentrations of particles — ranging in diameter from 0.05 µm to 20 µm — in industries such as the semiconductor industry. *Light-extinction technology* measures the overall loss of light intensity from the light source due to scattering by the particles collectively, and is typically used to measure high concentrations of particles larger than 2 µm in diameter. This RP focuses on the LPCs based on light-scattering technology.

Light scattering and extinction by particles are described by the Lorenz-Mie theory (Bohren & Huffman, 1998) as applied to spherical particles of known size and refractive index. The theory relates the scattering efficiency to the cross-sectional area of a particle. The scattering of light by particles is defined by the optical size parameter α, termed as:

$$\alpha = \frac{\pi d_p}{\lambda}$$

where d_p is the particle characteristic, usually taken as the geometrical diameter of a spherical particle, and λ is the wavelength of light illuminating the particles. Depending on the optical size parameter range, particle size scattering power can be divided into three scattering regimes, as follows:

Rayleigh scattering ($\alpha << 0.1$): This type of scattering is applicable to small particles with diameters less than that of the wavelength of the illuminating light. The intensity of the scattered light follows the sixth power of the particle diameter, given by

$$\frac{d_p^6}{\lambda^4}.$$

Mie scattering ($0.1 < \alpha < 10$): This type of scattering is dominant when the particle sizes are comparable to the light wavelength. This is the transition regime in which the detection limits of most of the LPCs are determined. For example, the wavelength of helium-neon (He-Ne) red light is about 633 nm. When this kind of laser is used, particles between 20 nm and 2000 nm in diameter fall within the Mie scattering range.

Geometric scattering ($\alpha >> 10$): This type of scattering is dominant when the particle sizes are larger than the light wavelength. Usually, geometric scattering is used to explain the scattering of particles larger than 10 µm.

In an LPC, laser light is focused on a sample cell or capillary through which liquid is flowing, as schematically shown in Figure 1. If the refractive index of the particle is different from that of the liquid, light is scattered by each particle that traverses the laser beam. An optical assembly of lenses and mirrors is used to collect scattered light and focus the light onto the array of photodetectors. The photodetectors then convert the scattered light into electrical signals. Each electrical signal or pulse correlates to a particle,

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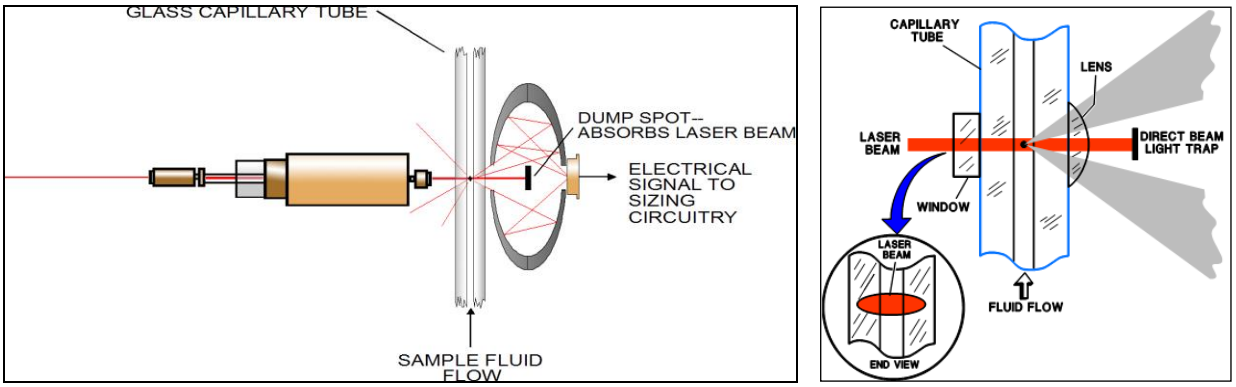

Figure 1—Schematics of the optical system of a liquid-borne particle counter based on light scattering. (Left: part of the flow cross-section is illuminated; right: 100% of the flow cross-section is illuminated.)

since the magnitude of the voltage pulse is correlated to the amount of light scattered by a particle of a specific diameter. During sampling, individual pulses are converted into the numerical data that represent particle counts.

The optical response R for a single-particle light-scattering instrument is

$$R = \iint G \frac{dC_{sca}}{d\Omega} f d\lambda d\Omega ,$$

where G describes the illumination and collection geometry, f includes the source spectrum and spectral sensitivity of the photodetector, and the differential scattering cross-section

$$\frac{dC_{sca}}{d\Omega}$$

is calculated from the Lorenz-Mie theory, in which the intensity I of the light scattered by the particles depends on the incident light intensity I_0, the polarization angle Φ, the detection angle of the scattered light Θ, the refractive index n, the wavelength λ, and the optical size parameter α : $I = I_0 f\left(\Phi, \Theta, n, \alpha\right)$.

5.2 LPC instrument types

5.2.1 Volumetric vs. non-volumetric and monitor vs. spectrometer/counter

From a capability point of view, depending on the size resolution and the number of size channels, LPC instruments are categorized as spectrometer, counter, or monitor. Spectrometers have the highest resolution and the greatest number of size channels. Counters may have high intrinsic optical resolution, but that resolution is reduced because fewer size channels are provided in the design of electronics to reduce cost. Monitors do not provide uniform sample volume illumination, thus they offer poor resolution. However, one of the advantages of monitors is that they allow larger effective flow rates for the same sensitivity relative to spectrometers; hence, monitors yield more statistically reliable data in high-purity applications. Monitors are also simpler to operate and less expensive than spectrometers and counters.

From a design point of view, depending upon the effective sampling volume, LPC instruments are categorized as volumetric or non-volumetric. Volumetric LPCs are designed to view the entire sample flow under a uniform laser beam intensity. The uniform beam shines on the particles flowing in the liquid with the same light energy, regardless of each particle's location in the flow path, and therefore offers high resolution. The drawback to this design is that the capillary walls are constantly illuminated with a high-intensity laser beam, resulting in unwanted scatter that will limit the sensitivity of the instrument. Volumetric LPCs are generally used in both batch sampling and online applications. Liquid spectrometers and counters are typically volumetric LPCs.

Non-volumetric LPCs are instruments that allow only a small fraction of the sampling volume to be illuminated by a laser light. With the exception of designs using special and often expensive optical systems, this design typically offers poor resolution, as the light intensity is not uniform across the laser beam. Poor size resolution results in less accurate size calibrations. However, capillary walls can be placed far apart to accommodate a wider range of laser beam profiles, resulting in less background light scatter and greater sensitivity. Non-volumetric LPCs are well suited for continuous monitoring of supply lines. Most liquid monitors are non-volumetric LPCs.

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