CORDOUAN Technologies

VASCO™ Application Note

>> Select the right DLS system for your application

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an overview of performances/sensitivity comparison between Avalanche Photodiode (APD) vs Photo Multiplier Tube (PMT) configurations

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Abstract

This note aims to give some guide lines in order to select the right **VASCO™** Dynamic Light Scattering (DLS) instrument version in regards to the targeted applications. After a general presentation of the **VASCO™** system principle, we show how the choice of the detector (PMT vs APD) impacts the sensitivity of the instrument and allows to cover an unprecedented broad range of application from very diluted / low scattering colloids to non-transparent samples.

Keywords : nanoparticle sizing, dynamic light scatterring (DLS), colloidal suspensions, APD, PMT.

VASCO™ an innovative and versatile tool for nano-particle sizing

Dynamic Light Scattering (DLS) is a powerful and versatile technique used to characterize dispersions of particles in liquids [1]. Based on the analysis of scattered light fluctuations caused by the Brownian motion of particles, it provides sizes measurements from the nanometer range up to a few microns. Nevertheless, when analyzing dark and concentrated dispersions, conventional DLS techniques have to deal with significant limitations, namely laser absorption and multiple scattering which strongly impacts measurement capabilities.

Thanks to an original and patented of its sample cell design (see figure 1), the VASCO™ instrument overcomes these limitations by combining the following major improvements:

- Back-scattered light detection configuration which provides higher detection efficiency for small particles and opaque/dark sample.
- Measurement performed on liquid thin film with controllable sample thickness from one millimeter down to few tenths microns. This configuration allows cancelling measurement artefacts of multiple scattering and strongly reduces laser induced thermal effects due to light absorption.

An innovative patented geometry

The bottom of the measurement cell is made by the upper surface of a glass prism which is also used to guide the incident laser beam to the sample. This configuration allows also the photo-detector to collect the back-scattered light signal at a scattering angle of 135° with respect to the incident laser beam. In addition, the cell is hermetically closed by a mechanical system named DTC™ (Dual Thickness Controller) that includes a mobile glass rod with a photon trap. This rod can both control the sample thickness (down to few tenths of microns) and absorb the excess of transmitted light

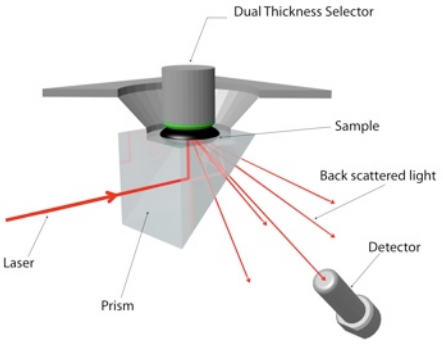

Figure 1: Illustration of the VASCO™ measurement cell principle.

These innovations make the **VASCO™** instrument the perfect tool for the characterization of ink, paint or concentrated emulsion analysis, for example. Moreover, the **VASCO™** takes full advantage of the latest technology advances in opto-electronic components (detector, correlator, laser) to provide high performance results for diluted and/ or very low scattering level of samples like micelles, liposomes, virus, proteins, etc. In this application note, we first describe the specification of these key components before illustrating experimental results their benefits on the measurement sensitivity of the apparatus.

Keys components

sensitivity and accuracy performances of the VASCO™ series

The VASCO series is currently made of three different versions which integrate dedicated and optimized key components:

>> Versatile measurement cell for concentrated and diluted samples

With its liquid thin film measurement configuration, the patented geometry of the VASCO™ measurement cell is unique in DLS instrumentation. In addition to the analysis of dark /concentrated and diluted samples, this original design allows on line measurement without stop flow.

>> High reliability fiber pigtailed laser diode

Using Peltier thermo elements for temperature stabilization, our semi-conductor laser can provide a maximum optical power of up to 65 mW @ 658 nm with a very low noise level. Thanks to its specialty fiber pigtailed output, the laser beam is easy to transport with a purely Gaussian spot and with an ultra-stable polarization. With a technology derived from the telecom industry, the high reliability laser has an operating lifetime > 20 000 hours. Its power stability is guaranteed by optimized integrated electronics drivers. With such a large power range and high stability, the VASCO™ product line is able to measure a very broad range of particle types and sizes.

>> High resolution correlator

With up to 1000 linear channels (DSP 16bits), a channel time selectable from 125 ns up to 30 ms (figure 2), and a count bandwidth of 8 Mcounts/sec, our customed designed correlator provides high accuracy and excellent time resolution even for fast events analysis. This capability is particularly valuable for measurement of very small particles (ie. in the nanometer range) up to a few microns.

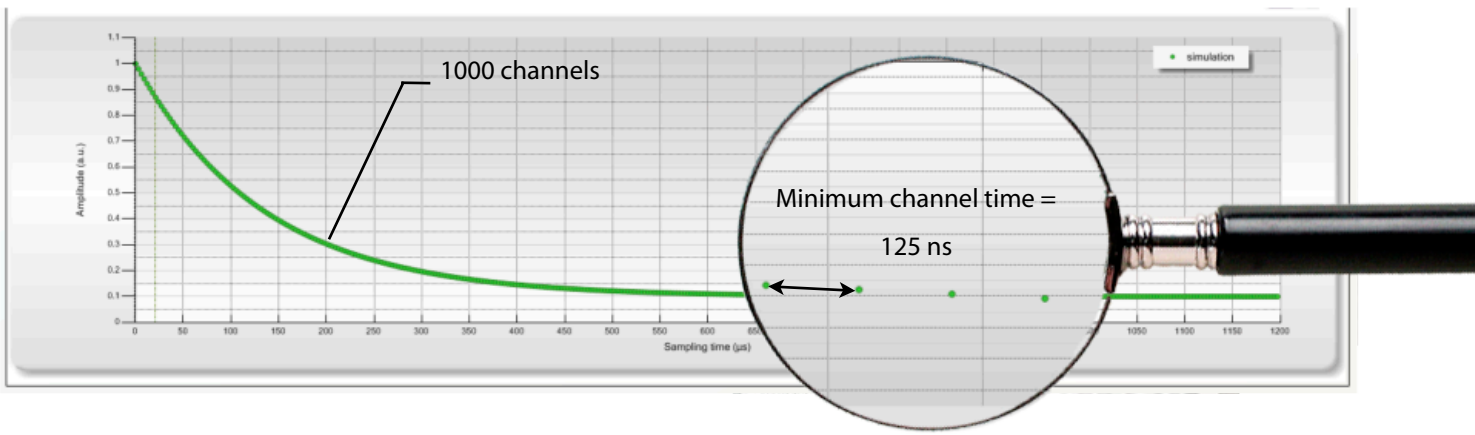

Figure 2 - High resolution correlogram

Detector

In order to cover the broadest application range, the VASCO product line is currently proposed in three versions namely VASCO™1, VASCO™2 & VASCO™3 with different detector types :

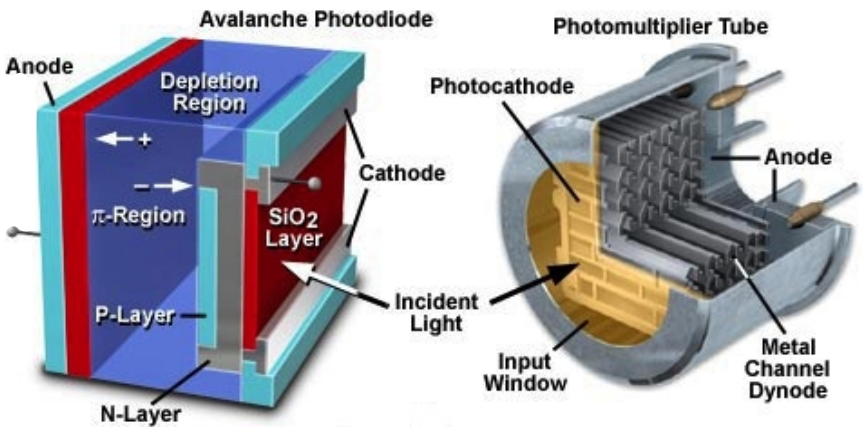

Figure 3 - Design of an avalanche photodiode (APD) and photomultiplier tube (PMT)

>> Photo-Multiplier Tube (PMT)- VASCO™1 & 2

Our PMT is a robust technology and of the highest quality. It provides a very low noise (dark count ≈ 150 count/sec), high sensitivity (Gain > 10^5) and an ultra-fast response time (<10 nsec). This level of performance is comparable to most of APD's currently used in similar instrumentation. This makes the PMT a very reliable and versatile tool for efficient photon counting measurements under low light level or short light pulses.

>> Single Photon Avalanche Photo-Diode (APD) - VASCO™3

In order to further extend the measurement capabilities of the VASCO™ system for extreme detection applications (very diluted samples, very small size and/or particles with poor refractive index contrast) we have developed the VASCO™3 system which integrates a new generation of APD named SP-APD for "Single Photon counting" APD. This new generation of photon detector offers an advanced quenching active mode where the output is directly a digital pulse exactly synchronized with the photon arrival time. Just after the photon detection, the detector voltage is immediately reduced which makes it blind for a very short period of dead-time (< few tenths of nsec). As a result, after-pulse effect becomes negligible, the detector is never saturated, and no external gating is necessary. This new technology is also very robust to light over-exposure and completely immune to surrounding magnetic fields. Finally, with its Peltier thermo element for temperature stabilization, this unique technology reduces the dark count noise down to less than tenths of count/sec which makes it the perfect detector for very low level signal detection. This APD combined with our high power laser diode, makes the VASCO™3 system the ultimate DLS tools of our product line for particle size measurement.

Experimental comparison between PMT and APD detector sensitivity

The capability for a DLS instrument to detect nano-particles is largely dependent on the nature and structure of the sample. Actually, the scattering properties of a colloid are intimately related to the size of particles, their concentration, and the refractive index contrast between the particles and the solvent. Thus, mineral or metallic particles, for instance, scatter much more light intensity in water and can be easily characterized at very low concentration and/or with small size. On the contrary, surfactant micelles, polymers in solution or proteins are much difficult to detect. The following table presents experimental evaluation of the detection sensitivity of the VASCO™2 and VASCO™3 instruments for various representative colloid samples. For each sample, we compare minimum concentration (wt %) values measurable with the apparatus. All the measurements are performed at a room temperature of 25°C, and scaled with the same signal to Noise ratio and for a defined acquisition time.

Particle type and hydrodynamic diameter	***PMT (VASCO™ 1&2)***	***SP-APD (VASCO™ 3)***	***APD vs PMT sensitivity gain***
Standard latex (20nm polystyrene particles in water)	5.10^{-3}wt%	5.10^{-4}wt%	10
Standard latex (100nm polystyrene particles in water)	10^{-4}wt%	10^{-6}wt%	100
Standard latex (1µm polystyrene particles in water)	10^{-5}wt%	3.10^{-7}wt%	33
Metallic & Metallic oxide (6nm cerium oxide in water)	5.10^{-3}wt%	2.10^{-4}wt%	25
Silica (Ludox 20nm)	10^{-2}wt%	10^{-3}wt%	10
Micelles (Triton X-100, 8nm)	0.5wt%	2.10^{-2}wt% ≈ cmc	25
Protein (Lysozyme, 5nm)	0.5wt%	5.10^{-2}wt%	10
Solubilized Polymer (polystyrene in THF, 33500M, 10nm)	0.1wt%	10^{-3}wt%	100

These results show that, depending on the nano-particle type to be characterized, the APD detector brings an improvement to the detection sensitivity of the apparatus by a factor 10 to 100.

Conclusion

The VASCO™ particle size analyzer series offers an innovative solution to perform particle size measurements in a very broad range of applications from very concentrated samples (up to few tenths %) down to very diluted ones (concentration less than few parts per million) and very small particles. The proper choice of VASCO™ version should be mainly driven by your application measurement conditions and in particular by the concentration and the size of the particles to be characterized.

With a PMT detector, the **VASCO™1 & 2 solutions** are perfectly adapted for the characterization of highly concentrated sample (few % wt) down to hundredths of % wt concentration. Coupled to the innovative measurement cell and our unique in line measurement option, these versions are the ideal tool for industrial applications and operation in harsh environment. VASCO™2 is a very good alternative in comparison to "Classical DLS" instrument using disposable cells, with the best price to performance trade off in the market.

For characterization of nano-particles with a hydrodynamic diameter below 10nm and concentration in the part per million (ppm) range, and more generally, when scattered light intensity is very weak (less than 1000 kcounts/sec), we recommend to use the **VASCO™3 solution**. Thus, with its APD detector combined with our high power high stability laser diode, the VASCO™3 system is the ultimate DLS tools in the CORDOUAN product line for particle size measurement.

Since the introduction of the VASCO™ series has been launch into the market, CORDOUAN technologies has received a very positive feedback from all its customers and users, from the scientific community as well as from industrial labs where the VASCO instruments have brought an efficient and very cost effective solution for advanced nano-particles size characterization.

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References:

[1]: R. Pecora, editor, Dynamic Light Scattering: Application of Photon Correlation Spectroscopy, (Plenum Press, NewYork, 1985).

Specifications

Description	VASCO-1	VASCO-2	VASCO-3
Particle size range (nm)	10 to 6000	2 to 6000	1 to 6000
Sample concentration range (% volume)[1]	0.01% to 40%	0.001% to 40%	0.0001% to 40%
Photon counting unit	PMT[2]	PMT[2]	APD[3]
Sample setting temperature	Fixed at 20 °C Temperature stabilized	+ 15 to + 90 °C Temperature stabilized	+ 15 to + 90 °C Temperature stabilized
Laser diode characteristics	658 nm/15 mW option : 532 nm/30 mW	658 nm/65 mW option : 532 nm/30 mW	658 nm/65 mW option : 532 nm/30 mW
Applications	Ink, bitumen, metallic oxide dispersions, emulsions, etc...	Ink, polymers, metallic oxide nanoparticles, emulsions, etc...	Ink, polymers, metallic oxide nanoparticles, micellar dispersions, etc...
On-line measurement option	No	Yes	Yes

General characteristics

Reproducibility / Repeatability	Better than 5%
Analysis Software	nanoQ® performs multi-acquisitions, size distribution simulation, kinetic size of aggregation monitoring, features Cumulants, Contin and unique **Padé-Laplace** inversion algorithms.
Measurement time (typical) (programmable)	30 sec to 5 min depending on sample and measurement settings
Sample volume	< 50 µL
Ambient temperature	+15 to +30 °C
Warm up time (cold start)	< 5 min
Solvent[4]	Aqueous and organic solvents
Dimensions (HxWxD)	30 x 33 x 28 cm
Power supply	110/220 V in standard
Power consumption	< 50 W
Instrument configuration	Bench top (< 12 Kg)
Operating System	Windows 2000, XP, Vista, 7
Normalisation	CE marked product, CFR 21 part 11, ISO 13321 compliant
Laser safety classification	Class I compliant EN 60825-1/A2
Computer configuration[5]	Pentium III or equivalent, RAM 512 Mo
Accessories	Power supply and USB cable, nanoQ® installation CD ROM, Pelicase™ transport case
Computer Interface	USB 2

(1): maximum concentration value is limited by particle interactions

(2): photomultiplier tube (3): avalanche photodiode (4): solvent proof cell

(5): minimal expected configuration for optimal operation

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