MICROTRAC MRB

PARTICLE CHARACTERIZATION

DYNAMIC LIGHT SCATTERING

Nanoparticle Size, Charge & Molecular Weight

Size distribution and Zeta potential | Sample introduction flexibilty | 0.8 to 6,500 nm

PHARMACEUTICALS
FOOD / BEVERAGES
LIFE SCIENCES
CHEMICALS
ENVIRONMENTAL
POLYMERS
MICROEMULSIONS
COLLOIDS
INKS / PIGMENTS
GLUES
METALS
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Microtrac's 180° Flex Probe Technology

Microtrac probe technology – unique design, superior results

Microtrac has taken an innovative approach to dynamic light scattering (DLS) by using a proprietary probe design to deliver and collect light. By focusing the laser probe at the material interface, Microtrac combines the benefits of a short path length with reference beating and 180° backscatter, delivering the best accuracy, resolution and sensitivity.

180º backscatter and GRIN lens focusing for accuracy at highest concentrations

Microtrac's probe focuses the laser at the interface between probe and particle suspension. Light penetrates the suspension and scattering takes place with the encountered particles and 180° backscattered light. Mixed with the incident light it returns to the photodetector. The total path length is minimized, while the collected scattered light is maximized. Result: accurate measurements at the highest particle concentrations.

Reference beating technology – strongest optical signal and accuracy at lowest concentrations

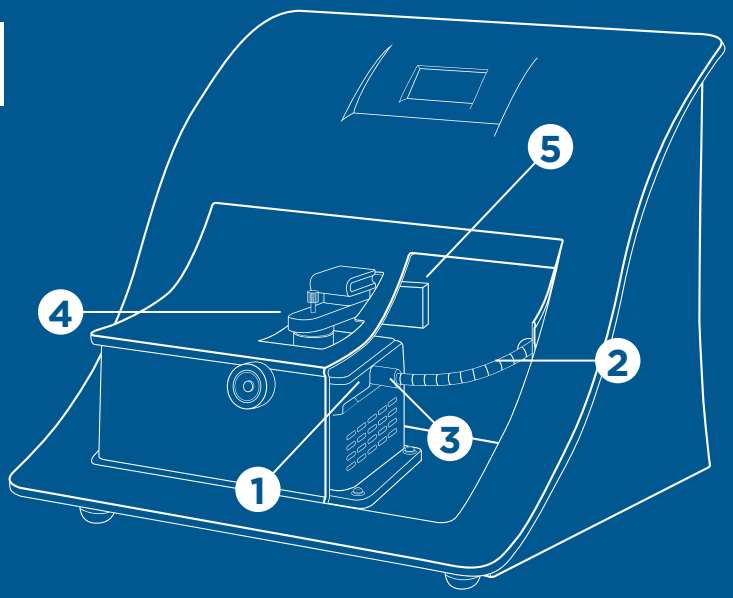
All dynamic light scattering measurements use a form of 'beating' to strip away the high optical frequency from the scattered light, leaving the particle motion-induced lower frequencies required for size analysis. Microtrac's heterodyne detection principle uses the probe to collect 180° backscattered light mixed with incident light. The geometry of the components enables light to reflect from the interface and combines it with collected scattered light. The reflected light enables reference beating. The total optical signal is amplified by the high intensity of the reflected component. The result is the highest possible optical signal providing accurate measurements in the lowest possible concentrations.

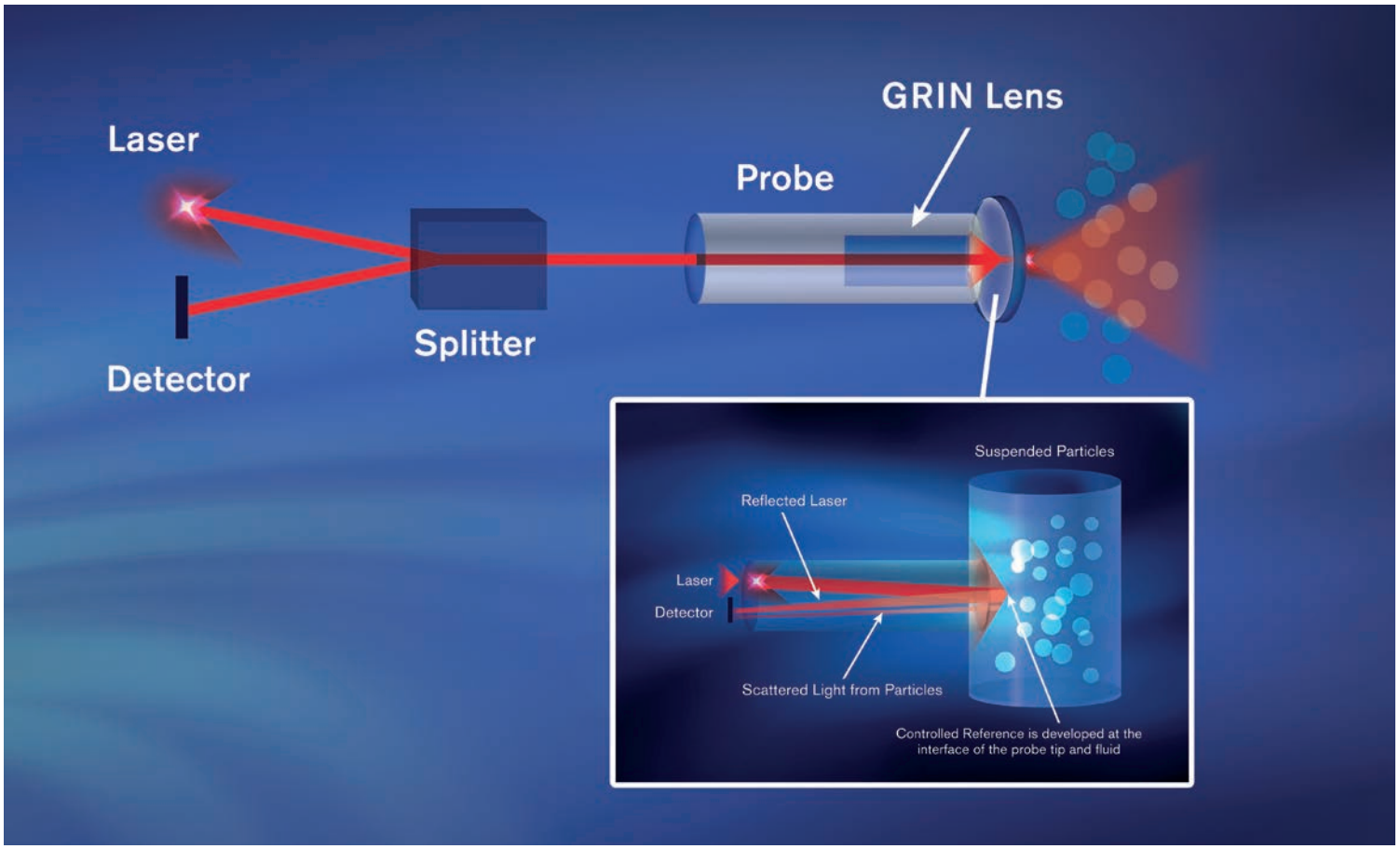
The heterodyne measurement principle with reference beating also allows for sizing fluorescent particles.

Microtrac's probes depicted

Nanotrac Wave II / II Q

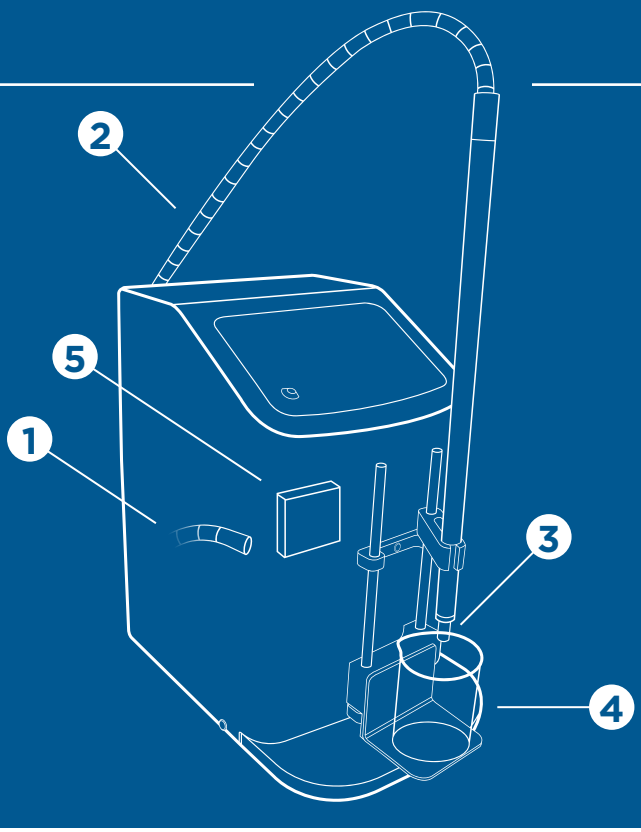
1. Laser
2. Light fiber
3. Probe
4. Sample chamber (II) / cuvette (II Q)
5. Detector





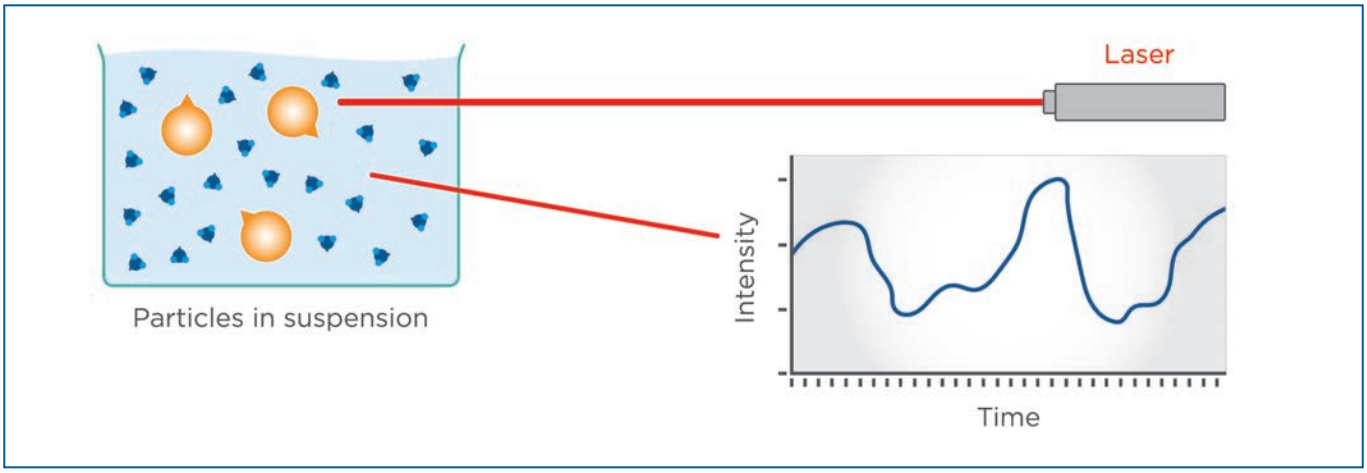
Nanotrac Flex

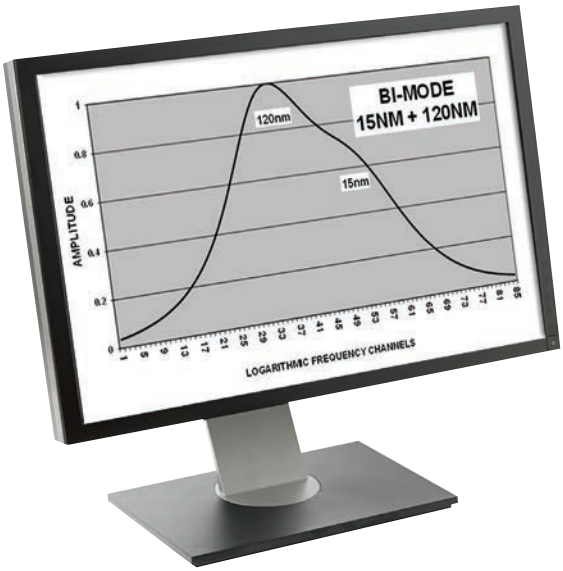
1. Laser
2. Fiber
3. Probe
4. Sample vessel
5. Detector



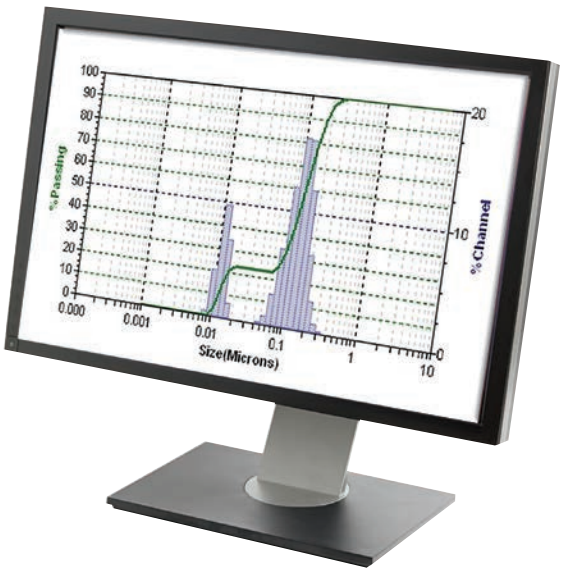
DLS: Frequency Power Spectrum

Dynamic Light Scattering is based on the Brownian motion of particles in suspension. Smaller particles move faster, larger particles move slower. Light scattered by moving particles carries information about the size distribution. The conventional method to analyze scattered light in DLS is called Photon Correlation Spectroscopy (PCS). It requires an autocorrelator and provides just a mean size, or needs vendor-specific curve-fit algorithms to estimate a size distribution. The Frequency Power Spectrum (FPS) method is different – the intensity signal of the photo detector is transformed mathematically by Fast Fourier Transformation into a Frequency Power Spectrum and directly provides a size distribution by iterative error minimization.





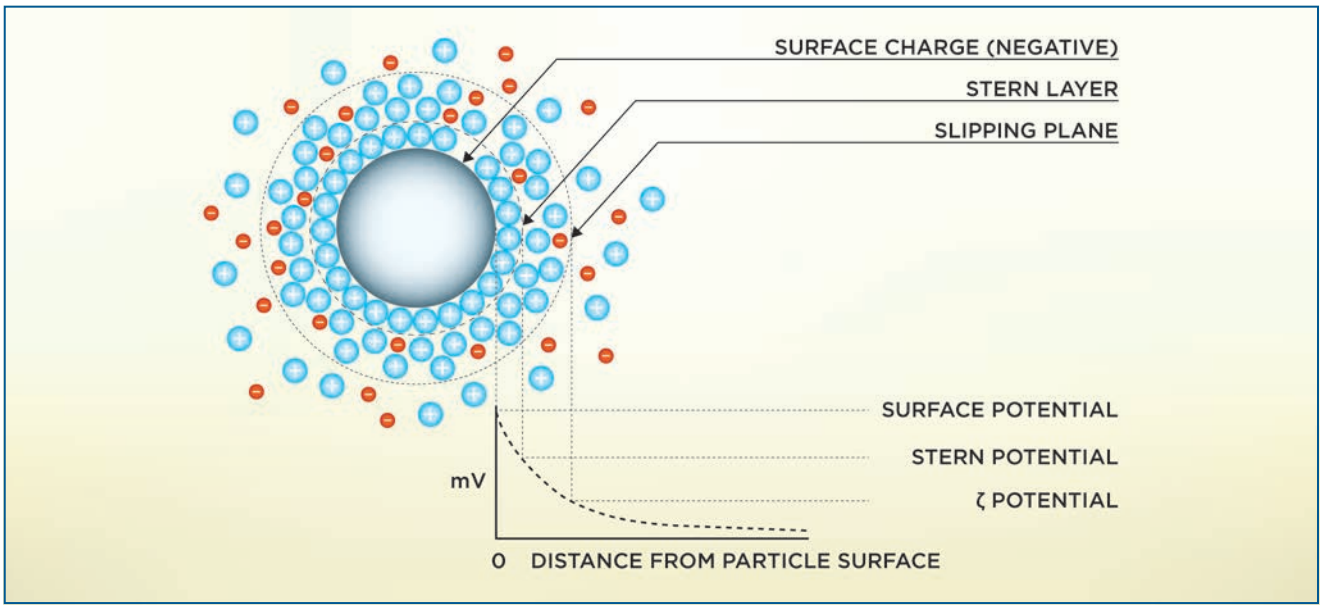
Frequency Power Spectrum



15 and 120 nm bimodal size distribution

Full size distribution and peak analysis

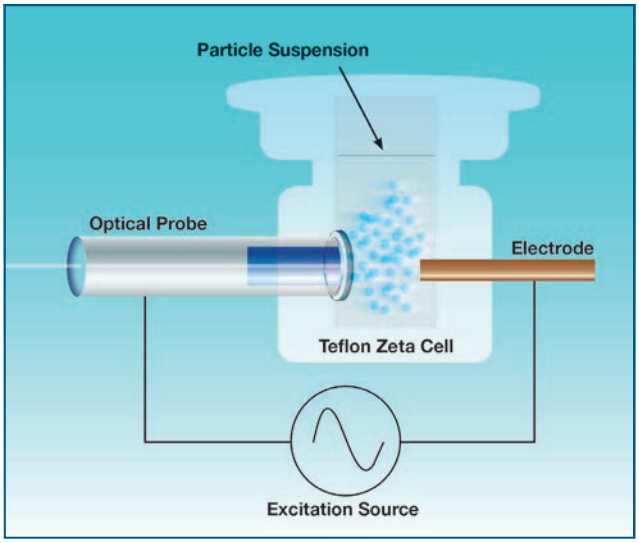
- **Distribution analysis** – For full featured, accurate particle size distributions from nm to µm
- **Mode analysis** – Quick results with specified size and volume concentration; this approach enables you to resolve and report accurate multi-mode distributions
- **Legacy calculations** – Enables measurement of historical specifications for data consistency with legacy instrumentation



Zeta potential is the electrokinetic potential in colloidal dispersions. Theoretically, the Zeta potential of a particle is the electric potential in the interfacial doublelayer at the location of the slipping plane relative to a point in the bulk fluid away from the interface. In other words, Zeta potential is the potential difference between the dispersion medium and the stationary layer of fluid attached to the dispersed particle.

Suspended charged particles are in constant Brownian motion. Applying an AC electric field creates an electrophoretic mobility that combines with Brownian motion, resulting in a modulation of the Brownian motion power spectrum called the **Modulated Power Spectrum (MPS)** signal. The MPS signal is proportional to the zeta potential of the particles:

The 180° backscattering probe by Microtrac allows for measuring the highest concentration of any optical Zeta potential and enables you to operate at or closer to your suspension's designed concentration.



- **High concentration** – The Microtrac probe is paired with an electrode. A voltage is applied, establishing an electric field between those two points, enabling accurate Zeta potential analysis across a broad concentration range.
- **High accuracy** – The Microtrac zeta probe measures the modulated power spectrum signal (MPS) using high frequency modulation, eliminating the need to correct the electro-osmotic flow
- **High precision** – Achieved by inclusion of Brownian motion frequency spectrum
- **Low sample volume** – Compact probe / sample interface allows for lowest volume available: 150 µL

Nanotrac Wave II

The **Nanotrac Wave II** is a measurement system for direct, automatic measurement of the electrophoretic movability and Brownian motion, as well as the resulting zeta potential and particle size. The analyzer's innovative design offers faster analyses with reliable technology, particle size measuring to below 0.8 nm and higher precision and accuracy – all of this combined in a compact dynamic light scattering analyzer without moving optical components.

The Wave II employs **Reference Beating,** which increases the optical signal anywhere from 100 to 1,000,000 times compared to traditional DLS. The increased optical signal enables users to accurately measure single and multi-mode distributions across the widest concentration range in the market.

- Enhanced optical signal for superior accuracy through Reference Beating
- Removable sample cell – available in Teflon or stainless steel in various sizes
- Able to measure Zeta potential closer to iso-electric point by eliminating errors caused by electro-osmotic flow (ZP measurement ranges from -200 to +200mV)
- Flow cell option & ability to connect a titrator
- No "A priori" or advanced knowledge of the particle size distribution is required
- 150 µl sample volume required – ideal for expensive materials
- User-selectable data presentation modes & Peltier temperature control device
- Unique probe design, fixed optics & 180° backscatter enable fast and precise measurement across widest concentration range – from ppm to near solids (40 % w/v)

Sample cells used with Microtrac's Nanotrac Wave II:

Micro: PTFE
50 µl – 100 µl

Nano: PTFE
200 µl – 2,000 µl

Nano: Stainless steel
200 µl – 2,000 µl

Large: Stainless steel
1,700 µl – 8,000 µl

The **Nanotrac Wave II Q** combines the best of both worlds for nanoparticle size analysis; Microtrac's proprietary probe technology, adapted for a cuvette interface, facilitating fast measurements and the ability to dispose of the sample cell after use.

The Wave II Q takes full advantage of the amplified optical signal via Reference Beating, probe technology fixed at the cuvette interface, and advanced algorithms. From ppm to near finished product, this DLS analyzer determines fast, repeatable, and sensitive measurements of particles ranging from sub-nanometer to several microns.

Even with a cuvette, Reference Beating increases the optical signal anywhere from 100 to 1,000,000 times compared to traditional DLS.

- Simultaneously measure size & concentration
- Cuvette error detection alerts
- No blank measurement or prior knowledge of size distribution required – simply load cuvette and measure
- Widest concentration range on the market from ppm to 40%
- Peltier temperature control device
- Compatible with 5 cuvette volumes:
  - Macro volume
  - Glass
  - Semi-micro volume
  - Micro volume
  - Stainless steel / Sapphire

Cuvettes used with Microtrac's Nanotrac Wave II Q:

Micro: Plastic
50 µl – 1,000 µl

Semi: Polystyrene
300 µl – 2,000 µl

Stainless: Sapphire
750 µl – 2,500 µl

Glass
1,000 µl – 3,000 µl

Macro: Polystyrene
1,000 µl – 3,000 µl

Zetrator & FLEX software

Microtrac Zetrator

Add titration to your size & Zeta potential analysis

- Define optimal process and storage conditions by identifying isoelectric points (IEP)
- Alter the electro-chemical conditions of your sample to determine points of stability
- Design elements eliminate bubbles interfering with results
- Available with one, three or five pumps
- Auto-clean mechanism ensures measurement repeatability & accuracy

Zeta cell: PTFE / Gold, 150 µl – 2,000 µl

Flow Cap for titration

FLEX software

Quickly identify if your material meets size specifications and take a detailed dive into your data – the FLEX software provides you with the tools you need.

- No “A priori” knowledge of particle size distribution needed – simply load your material and hit RUN
- Easy SOP set-up and administration, ideal for managing users across multiple shifts
- Cleanliness of cuvettes and sample cells displayed, including alarms and error messages
- Available in several translated languages
- Extensive database
- Compliant with FDA 21 CFR Part 11
- Statistical analysis
- Manually select dynamic viscosity value of material – ensures accuracy and consistency (according to Stokes-Einstein)
- No set zero required – the blank measurement is built into the software
- User gets notifications about the passing or failing of specifications
- Live or recalled database trending plots
- User-defined data reports and calculations
- Multiple data export options

Nanotrac Flex

- External probe with 180° backscatter
- Turn any vessel into a sample cell – the external probe allows you to dip and measure
- Small sample volume required, as low as 5 µl
- Amplified optical signal for high accuracy and sensitivity through Reference Beating
- Compliant to ISO 22412 & CFR21 Part 11
- Numerous probe lengths available from 1.0 m to 3.5 m
- Universal solvent compatibility
- Molecular weight calculation

Powered by Microtrac's enhanced dynamic light scattering technology, the Nanotrac Flex is ideal for measuring particles across wide concentration ranges, using minimal sample volume. Named appropriately, the external probe grants the flexibility to measure suspensions from sub-nanometer to several microns, in-situ or on a lab bench.

Based on 180° heterodyne dynamic light scattering the Nanotrac Flex can measure up to 40% w/v material concentration. With this setup, a part of the laser beam is added to the scattered light, which works like an optical enhancement. In addition to particle size, the molecular weight according to Debye can also be measured without having to add the needed dn/dc value manually. The dip-in probe enables direct measurement via in-situ analysis. Advantages of this are that no cuvettes are needed, the direct measurement in the product and the in-situ option. The results are ready after a measurement time of 10 to 360 seconds, with parameters like refractive index, particle absorption and viscosity being available in a database. By utilizing the proprietary Mie-calculation, the user is able to measure transparent or light-absorbing samples as well as spherical, non-spherical and irregular particles.

The probe as the catalyst for Reference Beating and Microtrac's enhancement to traditional DLS, increases the optical signal back to the detector anywhere from 100 to 1,000,000 times more than systems that use "self-beating." The enhanced signal produces superior analysis results when measuring single modes or multi-modes across the widest concentration range.

At-Line / On-Line

The Nanotrac Flex can be connected to liquid handling devices at customers‘ sites.

The FLEX software controlling the Nanotrac Flex has an interface to connect to external process control units, to initiate measurements on command, and export data to other computers or network drives.

The external probe has been integrated into high-throughput workstations to measure particle size automatically in formulation vessels and synthesis reactors. An automated dilution and flushing can be added, if necessary.

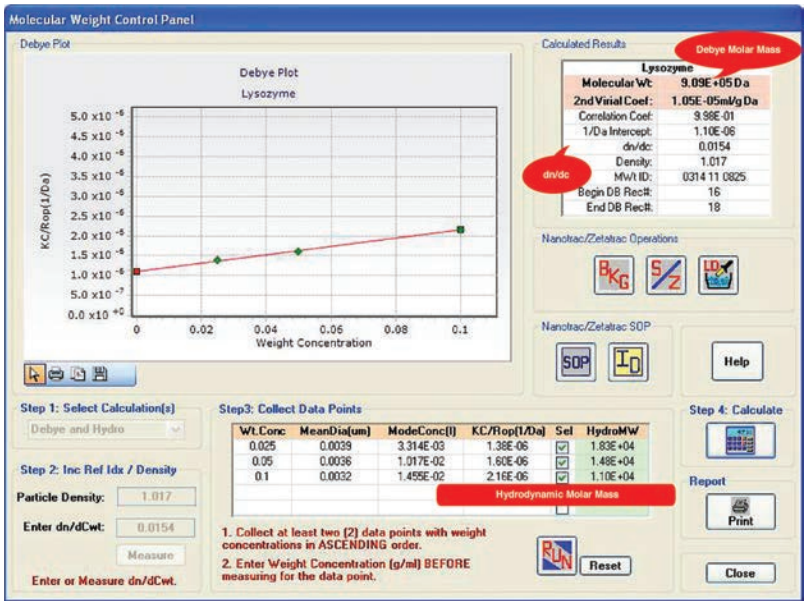
A flow cell is available for on-line bypass measurements without dilution, under pressure up to 6 bar.

Flex probe utilised with a reactor

Beyond Size & Zeta Potential

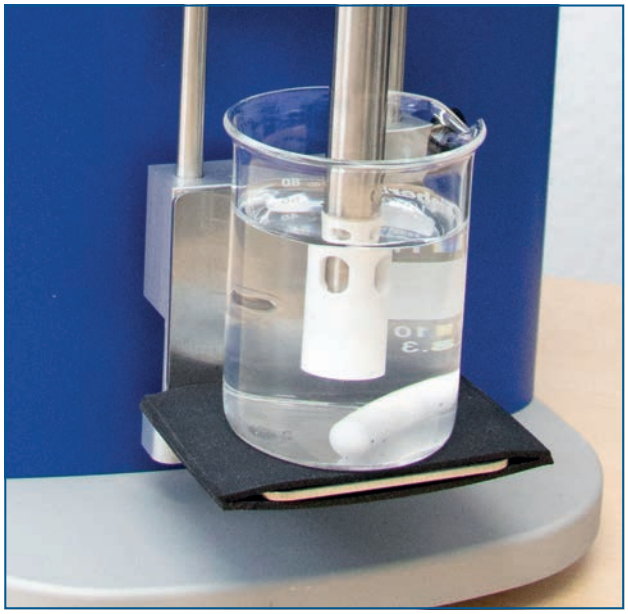
Molecular weight

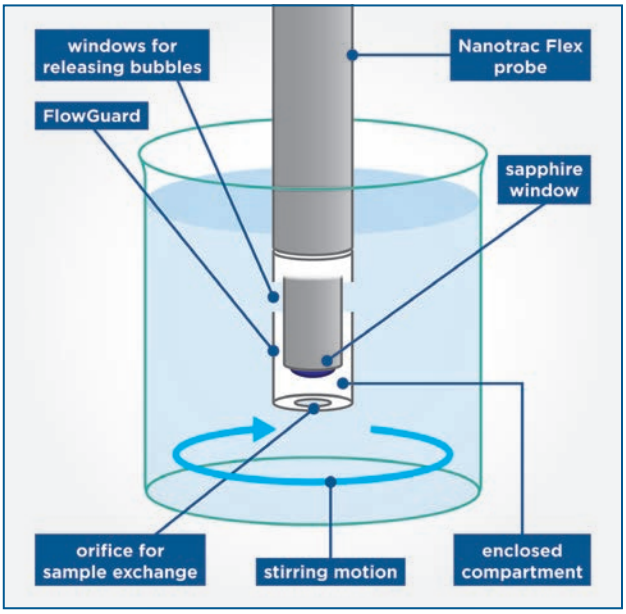
By using the Frequency Power Spectrum method, the molecules’ scattered light intensity – which goes into Debye plot calculation as a prime input – can be accurately measured with the prime input. The molecule index of refraction, a constant required for the Debye plot technique, can be determined with the Microtrac probe through the built-in index calculator.



Volume concentration

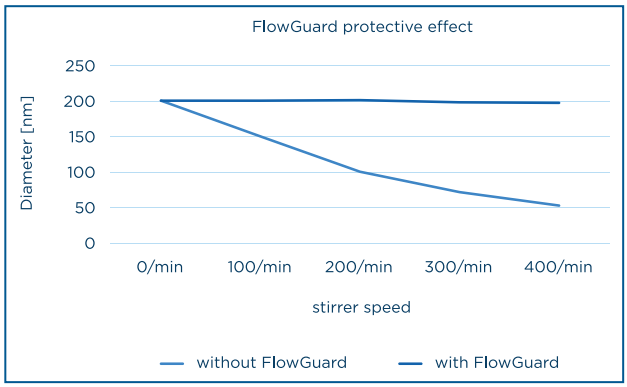
Mode analysis determines not only the size, but also the volume concentration (in cc/mL units) of each mode of the sample through the power spectrum magnitude. This method is ideal for monitoring reactor processes and nano attrition milling.



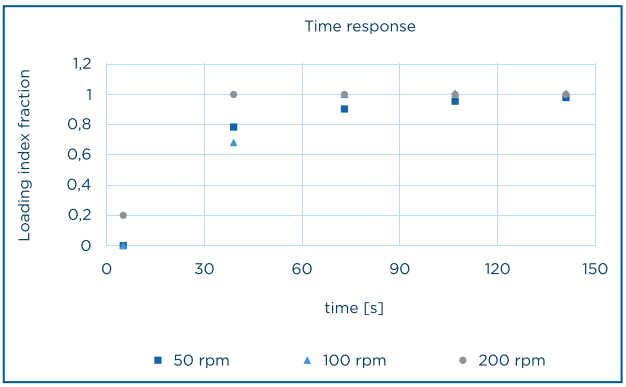


The **FlowGuard** creates an enclosure around the probe, which shields the measurement surface from turbulent flow. An orifice ensures constant exchange of sample, while slowing down the stirring movement at the probe interface. This design ensures an accurate particle size distribution that is representative of the suspension outside the enclosure.

- Apparent particle size decreases with increased external motion like stirring
- FlowGuard efficiently blocks the external movement in volumes a low as 20 ml
- Applicable to a wide range of nanoparticle sizes
- It works in a wide range of stirring speeds in laminar flow, up to critical Reynolds number
- Sample exchange is still ensured in a short time
- Get a real-time size measurement every few minutes
- Get control of your nanoparticle production process by connecting automated in-situ measurement to a process control unit



Impact of various stirring speeds on 200 nm polystyrene particles



Time response of exchange of 200 nm particles with various stirring speeds

Specifications

Microtrac's dynamic light scattering systems can measure particle size from 0.8 to 6,500 nanometers. The systems are compliant to ISO 22412 and produced under ISO 9001:2015. They meet the safety requirements according to CFR21 Part 11.

	Nanotrac Wave II	Nanotrac Flex	Nanotrac Wave II Q
			
Sample cell	Removable Teflon cell / stainless steel / ext. probe	External probe (in-situ)	Cuvette
Sample volume	▪ PTFE: 0.2 – 2 ml ▪ PTFE: 0.05 – 0.1 ml ▪ PTFE / Gold: 0.15 – 2 ml ▪ Stainl. steel: 0.2 – 2 ml ▪ Stainl. steel: 1.7 – 8 ml	Min. 0.05 ml	▪ Micro: 0.05 – 1 ml ▪ Semi-micro: 0.3 – 2 ml ▪ Sapphire: 0.75 – 2.5 ml ▪ Glass: 1 – 3 ml ▪ Macro: 1 – 3 ml
Peltier temperature control	√	–	√
Zeta potential	√	–	–
Molecular weight	√	√	√
Concentration range	Min. 100 ppb, max. 40% w/v*	Min. 100 ppb, max. 40% w/v*	Min. 100 ppb, max. 40% w/v*
Concentration determination	√	√	√
Measurement angle	180°	180°	180°
pH range	2 - 12	2 - 12	2 - 12
Chemical compatibility	Compatible with a wide choice of organic and inorganic dispersants	Compatible with a wide choice of organic and inorganic dispersants	Compatible with a wide choice of organic and inorganic dispersants

*sample-dependent

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