Unlocking Aerosol Insights Through Particle Size Analysis

Olga MATVIICHUK, Jean-Baptiste MATHIEU

Introduction to the LEREM

- **Established History:** Founded in 1961, the LEREM has over six decades of expertise in metal packaging research and testing. It operates as a non-profit association.

- **Core Specialization:**
  - *Dangerous Goods Transport:* Certified by the French Ministry of Transport for testing and approving packaging for dangerous goods. Approved for the performance of manufacturing controls.
  - *Aerosol Safety:* Experts in testing mechanical strength, flammability, and content-container compatibility for aerosol products and other metal packaging.

- **Industry Partnerships:** Actively collaborates with regulatory bodies (MPE, SNFBM, EIPA, CFA, FEA, AD) and contributes to technical committees in France and Europe.

- **Quality Assurance:** ISO 9001 certified since 1997, ensuring reliable and high-quality service.

Introduction to the LEREM: Unique Value

- **Highly Qualified Experts:** Our team of seasoned professionals brings decades of experience, ensuring precise analysis, tailored advice, and reliable solutions for even the most complex challenges.

- **A One-of-a-Kind Laboratory:** The LEREM stands alone as a specialized and independent lab dedicated to the metal packaging industry, offering unparalleled expertise and services.

- **Comprehensive and Customizable Testing:**
  - A wide range of mechanical tests, flammability tests, electrochemical (EIS) and aging studies…., designed to meet diverse client needs.
  - Custom protocols are developed to address unique problems, ensuring a personalized approach to each project.

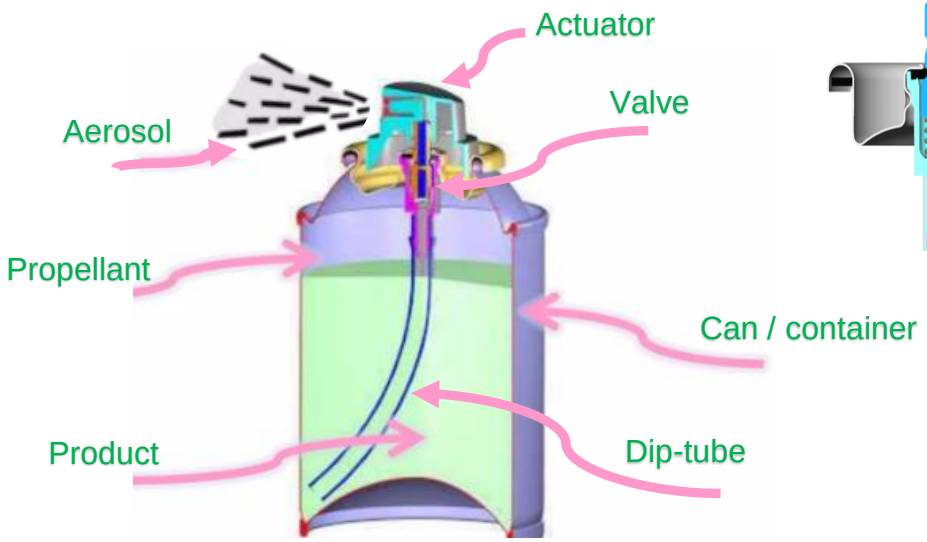
- **Problem-Solving Partner:** Whether solving quality or compliance problems or analyzing the root causes of persistent problems, the LEREM is an essential partner for customers facing critical packaging issues.

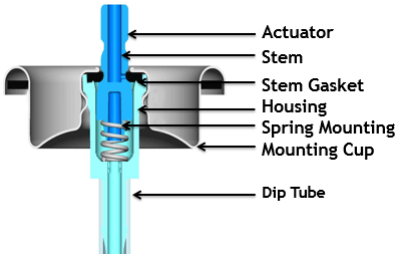
Basics of Particle Size Analysis and Spray Measurements

Spray definition and its relevant parameters

*A **spray/aerosol is a dispersion of liquid droplets in a surrounding gas**, typically formed by forcing a **liquid** through a **nozzle** (actuator) under **pressure**.*

- **Aerosols consist of:**
  - Can / container
  - Actuator
  - Valve
  - Product
  - Propellant
    - Liquefied gases
    - Compressed gases





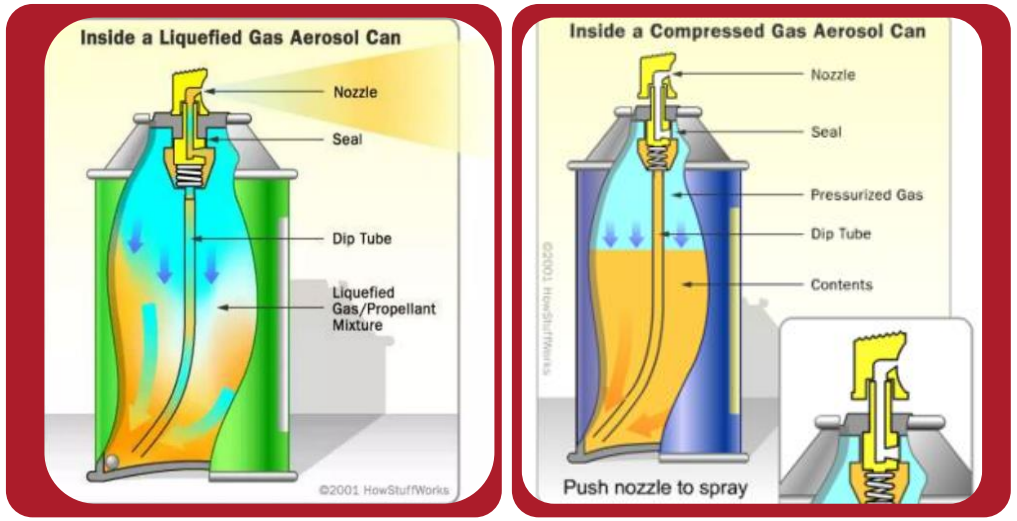
Propellants: a) Responsible for developing proper pressure withing the container to b) Provide a driving force to expel the product out of it.

Basics of Particle Size Analysis and Spray Measurements

Spray definition and its relevant parameters

- ***Liquefied Gases:*** *Stored as liquids under pressure with some gas in the headspace;* expand into gas on release, ensuring consistent atomization.
- ***Compressed Gases:*** *Stored entirely as gas under high pressure;* expansion propels the product, but pressure drops with use, reducing consistency.

- **Liquefied Gases:**
  - Fluorinated hydrocarbons (HFC)
  - HFO 1234ze
  - DME
  - Hydrocarbons (HC)



- **Compressed Gases:**
  - Nitrogen
  - Carbon di-oxide
  - Nitrous oxide

Examples:

- Personal care products (hairsprays, deodorants)
- Household products (air fresheners, etc….)
- Technical products (paint spray, cleaners…)

Examples:

- Personal care products (some shaving creams, deodorants…)
- Household products (air dusters, etc...)
- Technical products (some cleaning and lubrication products, as well as paints...)

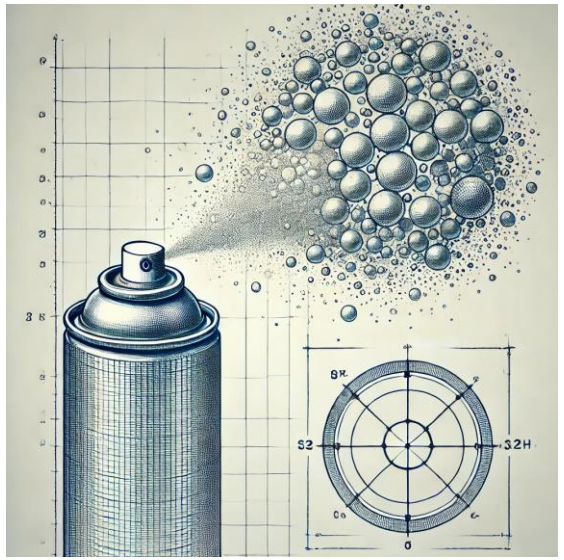
Basics of Particle Size Analysis and Spray Measurements

Spray definition and its relevant parameters

Several parameters are critical for evaluation of aerosols, characterizing and optimizing their performance:

- **Flammability and combustibility**
  - Flame projection
  - Ignition distance
  - Enclosed space test

- **Performance testing**
  - Internal pressure
  - Discharge rate
  - Droplets velocity
  - Leakage test
  - Formula stability
  - Spray patten and spray angles
  - Particle size determination
  - Droplet impact force

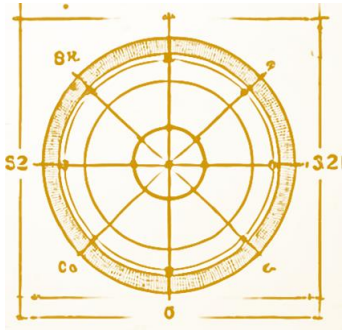


- **Physicochemical characteristics**
  - Propellant identification (IRTF)
  - Density
  - Moisture content

- **Biological testing**
  - Toxicity
  - Inhalation studies, etc....

Measurement Techniques for Particle and Spray Analysis

- **Spray Measurement:**
  - **Malvern Spraytec:** Uses laser diffraction to measure real-time size distribution of droplets in a spray.
  - **Phase Doppler Anemometry (PDA):** Measures droplet size of emulsions and velocity by analyzing the Doppler shift of laser light scattered by moving droplets.
  - **High-Speed Imaging:** Captures high-resolution images of spray droplets or jets at high frame rates for visualization and size estimation.
  - **Patternation Techniques:** Analyzes the spatial distribution of spray droplets by collecting and measuring their deposition on a surface or in a collector array.



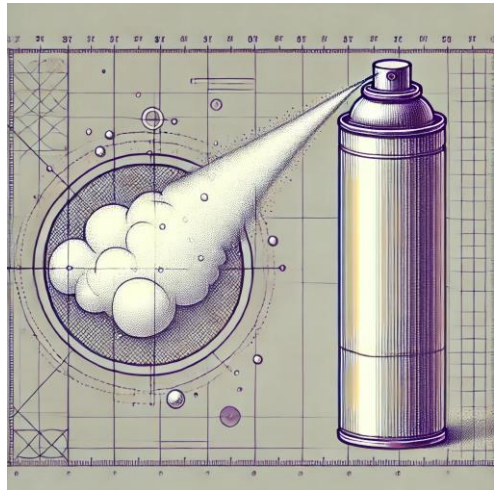
- **Particle Size Measurement:**
  - **Laser Diffraction:** Measures particle size distribution by analyzing laser light diffraction angles caused by particle interactions.
  - **Dynamic Light Scattering (DLS):** Analyzes the Brownian motion of particles in a liquid suspension, calculating size based on the fluctuations in scattered light intensity.
  - **Microscopy:** Manual and automated image analysis to visually inspect particle size and shape.
  - **Sieving:** Particles pass through a series of mesh screens with decreasing pore sizes.

Basics of Particle Size Analysis and Spray Measurements

Particle Size Measurement

Particle size refers to the physical dimension of individual particles in a material or spray.

- **Particle Size: Impact factors**
  - Product formulation
  - Type of gas propellant
  - Pressure
  - Valve & Actuator system
  - Agitation before use
    - Multi-phase formulation
    - Dissolved gas

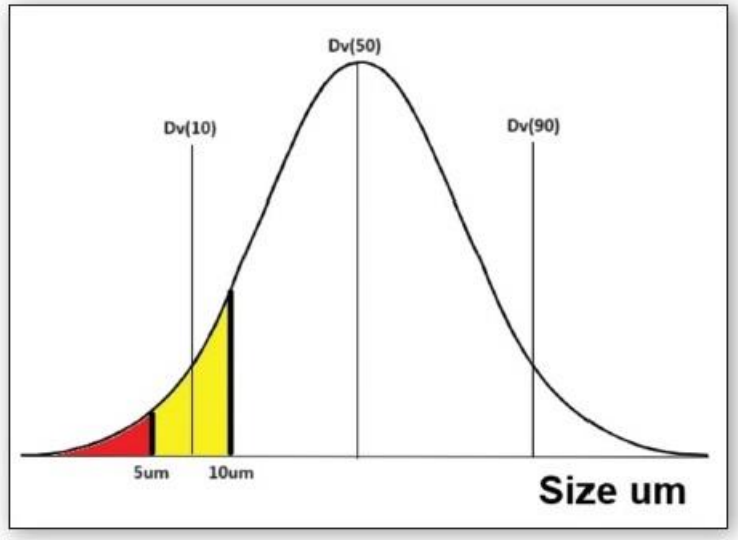


- **Particle Size Distribution Main Parameters:**
  - **Median Particle Size Dv50:** The size below which 50% of particles fall.
  - **Percentile Particle Size Dv10, 90:** Percentiles values of the particles size distribution.
  - **Mean volumic Particle Size D[4][3]:** The average particle size (e.g., arithmetic mean, volume-weighted mean)
  - **Respirable fraction %V < 10 µm**: The volumic percentage of the inhaled particles
  - **Standard Deviation:**
    - Indicates the spread of particle sizes
    - The product spraying quality
    - The measurement quality (R&R)

A device dedicated to control and safety

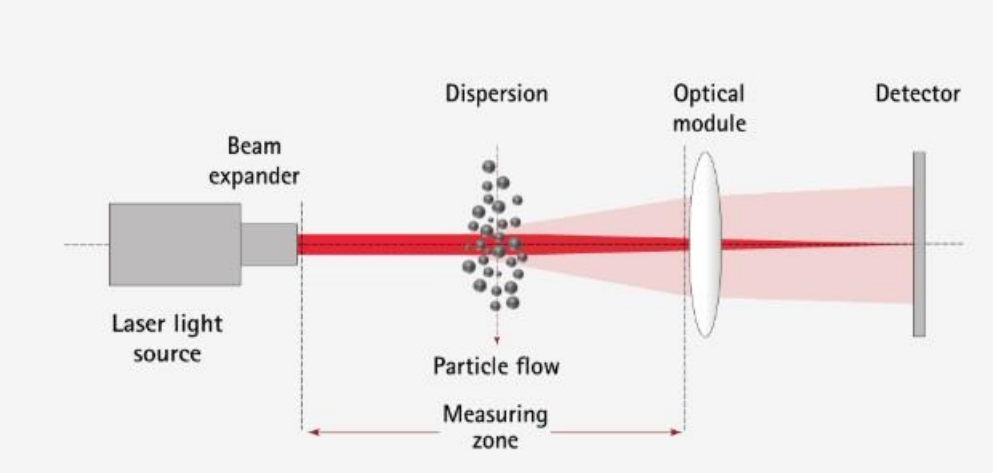
- Product particles size control
  - Product quality & efficiency assessments
  - Match the droplet size distribution with the expected product performance
  - Reliability of the results – repeatability between samples

- Safety issues
  - Inhalation fraction: the limit of 10 µm
  - Health assessments
    - CMR products
    - Toxicological validation for substances at risk



Laser diffraction technique

- Particle size analysis Standard
  - ISO 13320: 2020 : general requirements for instrument qualification and size distribution measurement
  - FEA guide (2009): specific to Aerosol Products



- Malvern Spraytec apparatus
  - Dedicated to aerosol products
  - Air as dispersing medium
  - Range of measurement from 0.1 µm to 2500 µm
  - Measurement calculation following the Fraunhofer Model
  - Datas processing for correction of the particle size distribution

Basics of Particle Size Analysis and Spray Measurements

LEREM's Measurement Technique

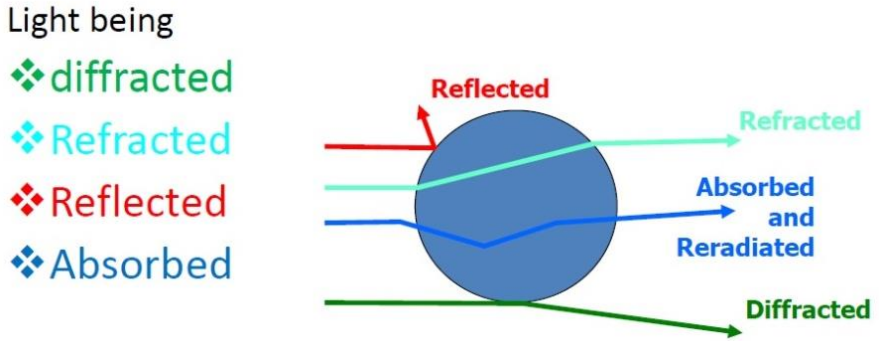
Basics of Particle Size Analysis and Spray Measurements

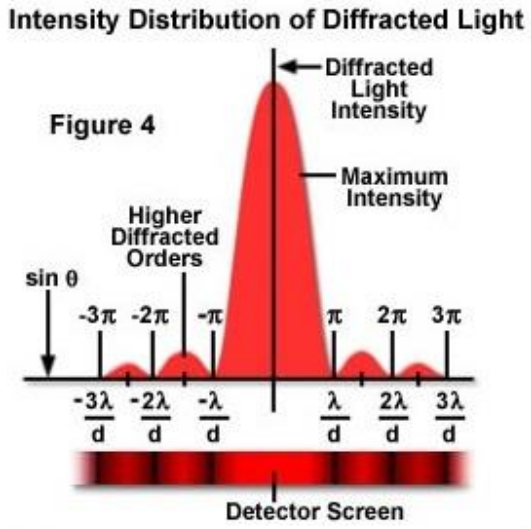
LEREM's Measurement Technique

Malvern Spraytec

- Initial settings
  - Average Refraction Index of the formulation
  - Background (electronic and physic)
  - Lenses cleanliness
  - Acquisition rate (frequency)
  - Acquisition duration
  - Spray length
  - Minimum detectable intensity threshold

- Spraying product
  - Product shaking and purge before measurement
  - Spray orientation
  - Measurement distance
  - Spray activation strength

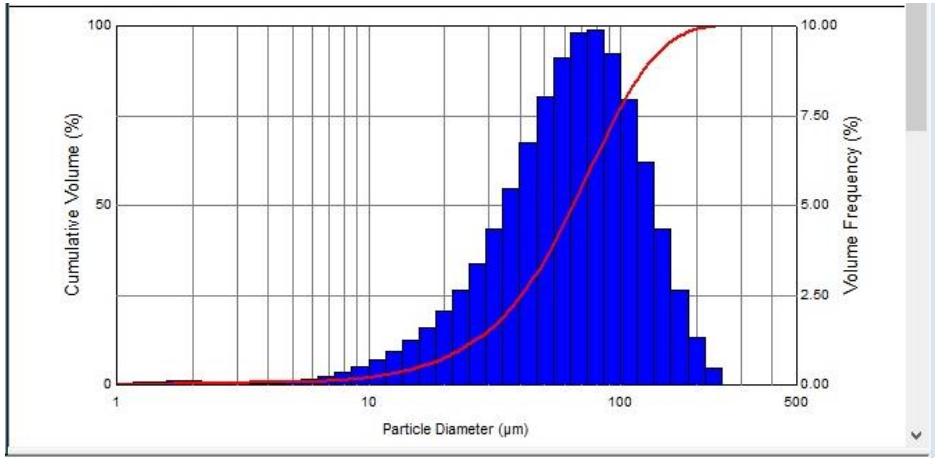




Basics of Particle Size Analysis and Spray Measurements

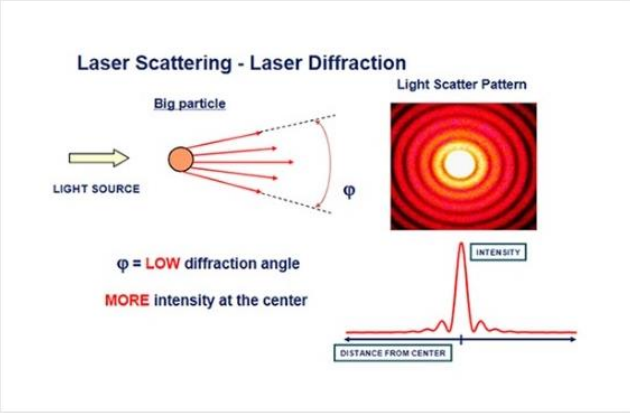
Study examples

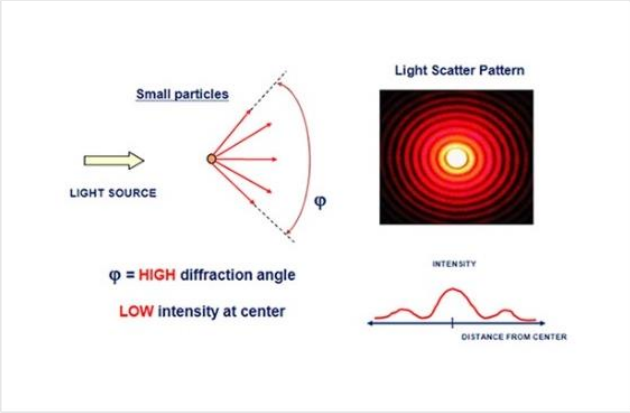
Particle size distribution: spray with compressed gas propellant



Title	Average	σ	Min	Max
Dv(10) (µm)	28.63	2.467	23.57	33.14
Dv(50) (µm)	64.25	3.033	57.63	71.8
Dv(90) (µm)	123.4	5.564	113.4	133.6
D[4][3] (µm)	70.87	2.735	65.33	77.67
D[3][2] (µm)	45.38	2.933	40.2	53.58
Cv (PPM)	50.15	8.683	33.46	74.77
%V < 5µ (%)	0.4693	0.144	0.04253	0.7523
%V < 10µ (%)	1.565	0.209	1.319	2.076
%V < 50µ (%)	33.31	3.053	25.68	40.33
%V < 15µ (%)	2.804	0.7153	1.692	4.44
Trans (%)	70.1	3.233	62.1	76.1

- Classical gaussian distribution
- No datas correction to apply
- Selection of the range for calculation
- Volumic averaging for relevant results

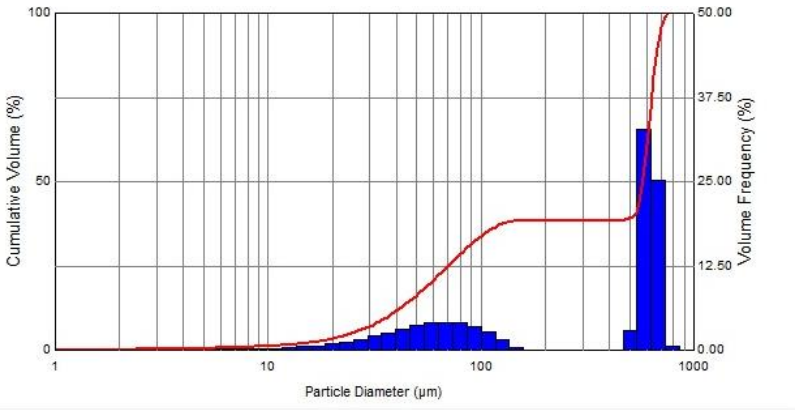




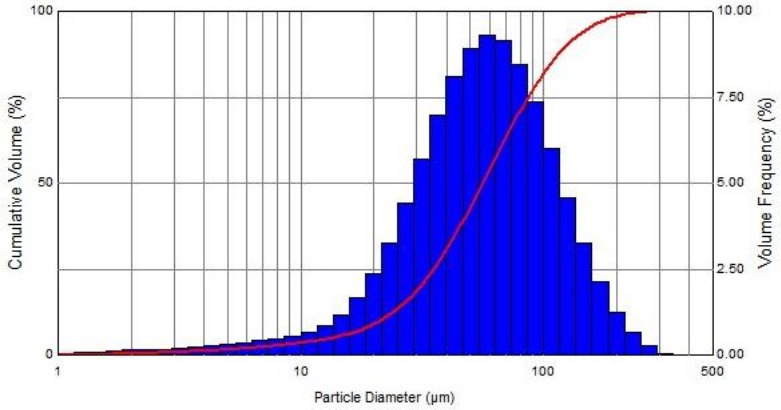
Basics of Particle Size Analysis and Spray Measurements

Study examples

Particle size distribution: spray with liquefied gas propellant



- « Beam steering » artifact
- Mix of air and gas as dispersing medium
- Idea of the expected particle sizes
- Datas correction necessary
- Propellant nature dependent



Title	Average	σ	Min	Max
Dv(10) (µm)	499.1	270.3	28.25	604.3
Dv(50) (µm)	631.5	74.39	123.3	666.9
Dv(90) (µm)	779	17.66	667	735.2
D[4][3] (µm)	636.2	107.2	332.9	662.1
D[3][2] (µm)	616.5	278.4	59.06	658.4
Cv (PPM)	351.6	232.3	19.12	634
%V < 5µ (%)	0	0.3717	0	0.9766
%V < 10µ (%)	0	0.8728	0	2.447
%V < 50µ (%)	0	8.335	0	24.75
%V < 15µ (%)	0	1.526	0	4.962
Trans (%)	84.2	4.801	74.5	95.3

Datas correction

Title	Average	σ	Min	Max
Dv(10) (µm)	9.728	6.819	4.296	28.01
Dv(50) (µm)	38.79	16.85	13.3	61.4
Dv(90) (µm)	98.07	34.13	35.59	127.5
D[4][3] (µm)	47.78	17.36	19.63	69.77
D[3][2] (µm)	19.76	9.845	8.843	42.79
Cv (PPM)	9.952	3.019	4.511	14.87
%V < 5µ (%)	3.914	3.521	0.7532	12.76
%V < 10µ (%)	10.38	11.01	2.258	36.06
%V < 50µ (%)	62.26	21.99	36.07	96.33
%V < 15µ (%)	17.48	18.42	3.282	56.05
Trans (%)	84.2	4.49	75.6	90.9

Basics of Particle Size Analysis and Spray Measurements

Applications and Challenges

- **Applications:**

  - **Cosmetics:** Optimizing aerosol spray characteristics.
  - **Pharmaceuticals:** Drug delivery efficiency.
  - **Agriculture:** Pesticides application precision and safety.
  - **Automotive & Coatings:** Achieving uniform spray patterns in paint and protective coatings.
  - **Cleaning & Disinfection:** Enhancing surface coverage and efficacy in cleaning sprays and disinfectants.

- **Challenges:**

  - **Test condition consistency:** Variability in environmental factors (humidity, temperature) affecting spray behavior.
  - **Sampling repeatability**
  - **Very wide spray pattern:** Contamination on lenses or nozzles affecting accuracy in capturing spray distribution.
  - **Correct Machine Settings**: Adjusting for specific products, new gases, and varying formulations.
  - **Results Interpretation**: Accurate analysis of spray data requires expertise, as many parameters interact and must be considered to make reliable conclusions.

thank you!

+33(0)3 44 72 82 77

laboratoire@lerem.org