Introduction to Particle Size Analysis

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What we’ll talk about

- Featured technologies
- Defining particle size
- Understanding size metrics
- Differences between techniques
- Q&A
Featured technologies

- **LA-950**
  Laser Diffraction

- **SZ-100**
  Dynamic Light Scattering & Zeta Potential

- **CAMSIZER & CAMSIZER XT**
  Dynamic Image Analysis

- **PSA300**
  Static Image Analysis

- **SA-9600**
  Flowing Gas BET Surface Area
LA-950: Laser Diffraction

- Particle size performance leader
- Ninth generation
- Ultra durable
- Lowest total cost of ownership
- Suspension, emulsion, powder, paste, gel
- 10 nanometer – 3 mm
SZ-100: Dynamic Light Scattering

- Particle size: 0.3 nm – 8 µm
- Zeta potential: -200 – +200 mV
- Molecular weight: $1 \times 10^3$ – $2 \times 10^7$ Da
- Patented ultra long-life graphite electrodes
- Lowest total cost of ownership
- Optional autotitrator

NEW!
Image Analysis

Dynamic:
particles flow past camera

Static:
particles fixed on slide,
stage moves slide
CAMSIZER Series

- High resolution size & shape
- Intelligent sieve correlation
- Patented dual capture
- CAMSIZER
  - 30 µm – 30 mm
  - Free-flowing powders
- CAMSIZER XT
  - 1 µm – 3 mm
  - Cohesive or free flowing
PSA300

- High resolution size & shape
- Referee technique for micronized powders
- Turnkey, automated image analysis
- 1 µm – 1,000 µm
- Cohesive or free flowing powders
- Optional Powder Disperser accessory
More Information

- Best Practices/Training
  - Understanding Laser Diffraction PSA Results TR008
  - Troubleshooting Laser Diffraction Data TR010
  - Understanding Dynamic Light Scattering Results TR012
  - Help! How Can I Trust My Size Results? TR015
  - Refractive index selection, sampling, dispersion, system verification, method development and more

- Technology
  - Intro to CAMSIZER TE002
  - Find the Best Analyzer for Your Application TE006
  - Intro to Static Image Analysis TE008
  - Intro to Dynamic Image Analysis TE009
  - Intro to Laser Diffraction TE010
  - Intro to Dynamic Light Scattering TE012
  - Intro to CAMSIZER XT TE015
Who Cares About Particle Size?

- Minerals Grinding
- Portland Cement
- Glass Beads
- Abrasives
- Foods
- Cosmetics
- Pharmaceuticals
- Paint and Coatings
- Metals and Ceramics
- Explosives and Fireworks
What we’ll talk about

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The most common designation is micrometers or microns. When very small, in colloid region, measured in nanometers, with electron microscopes or by dynamic light scattering.
Poll!

Which size ranges do you measure?
Particle Diameter (μm)

<table>
<thead>
<tr>
<th>Size Ranges</th>
<th>Ultrafine</th>
<th>Fine</th>
<th>Coarse</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Inhalable</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Relative Size of Common Material</th>
<th>Tobacco Smoke</th>
<th>Spores</th>
<th>Beach Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viruses</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bacteria</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carbon Black</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Paint Pigment</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Human Hair</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Common Methods for Particle Size Analysis</th>
<th>Electron Microscope</th>
<th>Optical Microscope</th>
<th>Static Angular Light Scattering</th>
<th>Dynamic Light Scattering</th>
<th>Electrozone Sensing</th>
<th>Sedimentation Methods</th>
<th>Field Flow Fractionation</th>
<th>Light Obscuration</th>
<th>Ultrasonic Spectroscopy</th>
</tr>
</thead>
</table>
The Basics

Which is the most meaningful size?

different size definitions \rightarrow different results
The Basics

What sizes can be measured?
Size Definitions

- **Martins’s Diameter**: The distance between opposite sides of a particle measured on a line bisecting the projected area. To ensure statistical significance all measurements are made in the same direction regardless of particle orientation.

- **Feret’s Diameter**: The distance between parallel tangents on opposite sides of the particle profile. Again to insure statistical significance, all measurements are made in the same direction regardless of particle orientation.

- **Note**: Both Martin’s and Feret’s diameters are generally used for particle size analysis by optical and electron microscopy.

- **Equivalent Circle Diameter**: The diameter of a circle having an area equal to the projected area of the particle in random orientation. This diameter is usually determined subjectively and measured by oracular micrometers called graticules.

- **Equivalent Spherical Diameter**: The diameter of a sphere that has the same volume as the irregular particle being examined.
Particle Orientation

- Martin’s and Feret’s Diameter’s will vary as particles are viewed in different orientations. The result will be a DISTRIBUTION from smallest to largest.
The Basics

Particle Distribution
The Basics

Particle Size  Particle Size Distribution

4 µm
Monodisperse vs. Polydisperse

- **Monodisperse Distribution:**
  - All particles are the same size
  - Latex standards

- **Polydisperse Distribution:**
  - Particles of Many Sizes
  - Everything else
Logarithmic vs. Linear Scale

- Logarithmic X-Axis Distribution
- Linear X-Axis Distribution
Distribution Display

- Represented by series of segments or channels known as histogram.
- Number of channels based on design, practicality and aesthetics
Your Analyzer’s Displays

- Frequency
- Frequency + cumulative (undersize)
- Histogram
- Multiple frequency + cumulative (undersize)
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Central Values

Mean
- Weighted Average
- Center of Gravity

Median
- 50% Point

Mode
- Peak of the distribution
- Most common value
What does “Mean” mean?

Three spheres of diameters 1, 2, 3 units

What is the average size of these spheres?

\[
\text{Average size} = \frac{(1+2+3)}{3} = 2.00
\]

This is called the D[1,0] - the number mean
Many possible Mean values

\[ X_{nl} = D[1,0] = \frac{1+2+3}{3} = 2.00 \]

\[ X_{ns} = D[2,0] = \sqrt{\frac{1+4+9}{3}} = 2.16 \]

\[ X_{nv} = D[3,0] = \sqrt[3]{\frac{1+8+27}{3}} = 2.29 \]

\[ X_{sv} = D[3,2] = \frac{1+8+27}{1+2+3} = 2.57 \]

\[ X_{vm} = D[4,3] = \frac{1+16+81}{1+8+27} = 2.72 \]

None of the answers are wrong they have just been calculated using different techniques.
Volume-based Mean diameter

D[4,3] which is often referred to as the Volume Mean Diameter [VMD]

\[ D \text{[4,3]} = \frac{\sum D_i n_i}{\sum D^3_i n_i} \]

Monitoring the D[4,3] value in your specification will emphasize the detection of large particles

Mean Size
The frequency distribution is found using the arithmetical mean diameter, as shown in the formula below:

\[ \text{Mean Diameter} = \frac{\Sigma q(J) \times X(J)}{\Sigma q(J)} \]

- \(J\) : Particle Diameter Division Number
- \(q(J)\) : Frequency Distribution Value (%)
- \(X(J)\) : Jth Particle Diameter Range’s Representative Diameter (μm)
Central Values revisited

Mean
- Weighted Average
- Center of Gravity

Median
- 50% Point

Mode
- Peak of the distribution
- Most common value

Remember: $D[4,3]$ is sensitive to large particles
Most Common Statistics

half are smaller than this diameter  
half are larger than this diameter

10% of the particles lie below this diameter

90% of the particles lie below this diameter

D(3,2) sensitive to small particles

D(4,3) sensitive to large particles

D(v,0.1) median

Never use the D100!
Standard Deviation

- Normal (Gaussian) Distribution Curve
- \( \mu = \) distribution mean
- \( \sigma = \) standard deviation
- \( \text{Exp} = \) base of natural logarithms

\[ Y = \frac{1}{\sigma \sqrt{2\pi}} \exp \left[ -\frac{(x - \mu)^2}{2\sigma^2} \right] \]
Distribution Width

- Polydispersity Index (PI, PDI)
- Span
- Geometric Std. Dev.
- Variance
- Etc…
For Your Reference

Variance
The value for the expanded distribution condition is found using the formula below.

\[
\text{Variance} = \sum \left[ \frac{(X(J) - \text{Mean})^2 \times q(J)}{100} \right]
\]

- J: Particle Diameter Division Number
- q(J): Distribution Graph Value (%)
- X(J): Jth Particle Diameter Range’s Representative Diameter (μm)
- Mean: Arithmetic Mean Diameter (μm)

Std. Dev.
Value taken from variance value’s square root.

Coefficient of Variation (CV)
This result of dividing the arithmetic standard deviation (Std. Dev.) by the mean diameter.

Mode Size
Frequency distribution value’s largest values that become particle diameters of the frequency distribution graph’s peak.

Span
Value that becomes the criteria for widening the distribution, as shown below.
Not displayed if both of the diameter on cumulative % are not set.

\[
\text{Span Value} = \frac{\text{Diameter on cumulative % A} - \text{Diameter on cumulative % B}}{\text{Median diameter}}
\]

Diameter on cumulative % A: the first value set in the display conditions.
Diameter on cumulative % B: the second value set in the display conditions.

Note: Span typically = (d90 – d10)/ d50
For Your Reference

Geometric Mean Size
The frequency distribution is found using the geometric mean value, as shown in the formula below.

\[
\text{Geometric Mean Diameter} = 10 \frac{\sum (\log X(J) \times q(J))}{\sum q(J)}
\]

J : Particle Diameter Division Number
q(J) : Frequency Distribution Value (%)
X(J) : Jth Particle Diameter Range’s Representative Diameter (µm)

Geometric Variance
The value for the expanded distribution condition is found using the formula below.

\[
\text{Geometric Variance} = 10 \sum (\log X(J) - \log (\text{Mean}))^2 \times \frac{q(J)}{100}
\]

J : Particle Diameter Division Number
q(J) : Frequency Distribution Value (%)
X(J) : Jth Particle Diameter Range’s Representative Diameter (µm)
Mean : Geometric Mean Diameter (µm)

Geometric Standard Deviation

\[
\text{Geometric Distribution Deviation} = 10 \sqrt{\sum (\log X(J) - \log (\text{Mean}))^2 \times \frac{q(J)}{100}}
\]

J : Particle Diameter Division Number
q(J) : Frequency Distribution Value (%)
X(J) : Jth Particle Diameter Range’s Representative Diameter (µm)
Mean : Geometric Mean Diameter (µm)
For Your Reference

Error Calculations in LA-950 only

Chi Square
Indicates the degree of similarity between the refractive index used to produce the particle size distribution calculation result and the actual scattering data. The closer to "0", the greater the similarity. Becomes the selection’s criterion when the refractive index is not known for the sample being measured. Chi Square($\chi^2$) is found using the following formula:

$$
\chi^2 = \sum \left\{ \frac{1}{\sigma_i^2} \left[ y_i - y(x_i) \right]^2 \right\}
$$

- $y_i$: Actual scattering measurement data
- $y(x_i)$: Scattering data found using refractive index file data and displayed particle size distributions
- $\sigma_i$: Scattering data standard deviation

R Parameter
Indicates the degree of similarity between the refractive index used to produce the particle size distribution calculation result and the actual scattering data. The closer to "0", the greater the similarity. Becomes the selection’s criterion when the refractive index is not known for the sample being measured. The Residual R Parameter is found using the following formula:

$$
R = \frac{1}{N} \sum_{i=1}^{N} \left\{ \frac{1}{y(x_i)} \left| y_i - y(x_i) \right| \right\}
$$

- $y_i$: $i$-th detector channel’s actual scattering measurement data
- $y(x_i)$: Scattering data calculated by calculating backwards from the refractive index data and the displayed particle size distribution
- $N$: Number of light detector channels
**Skewness**

1. **positive skew**: The right tail is longer; the mass of the distribution is concentrated on the left of the figure. The distribution is said to be **right-skewed**.

2. **negative skew**: The left tail is longer; the mass of the distribution is concentrated on the right of the figure. The distribution is said to be **left-skewed**.

---

**Negative Skew**
- Elongated tail at the **left**
- More data in the left tail than would be expected in a normal distribution

**Positive Skew**
- Elongated tail at the **right**
- More data in the right tail than would be expected in a normal distribution
Kurtosis (Peakedness)

From highest to lowest peak:
red, kurtosis 3
orange, kurtosis 2
green, kurtosis 1.2
black, kurtosis 0,
cyan, kurtosis −0.593762…
blue, kurtosis −1
magenta, kurtosis −1.2
Number vs. Volume Distributions

\[ V = \frac{4}{3} \pi r^3 \]

\[
\begin{align*}
V*3 &= 12 \\
12/432 &= 2.8% \\
96/432 &= 22.2% \\
324/432 &= 75%
\end{align*}
\]

\[
\begin{align*}
r = 1 \mu m & \quad v = 4 \\
r = 2 \mu m & \quad v = 32 \\
r = 3 \mu m & \quad v = 108
\end{align*}
\]

Total = 12 + 96 + 324 = 432

Number

Volume

Explore the future
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Beans!
Equivalent Volume Distributions
Equivalent Volume Distributions
Equivalent Volume Distributions
Equivalent Volume Distributions
Comparing Distribution Bases

- Same material shown as volume, number and area distribution

<table>
<thead>
<tr>
<th>Particle Size</th>
<th>Number Distribution</th>
<th>Volume Distribution</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean = 0.38µm</td>
<td>Mean = 12.65µm</td>
</tr>
<tr>
<td></td>
<td>Median=0.30 µm</td>
<td>Median=11.58 µm</td>
</tr>
<tr>
<td></td>
<td>SA=13467 cm²/cm³</td>
<td>Std Dev.=8.29</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.34 0.58 1.15</td>
<td>11.58 µm</td>
<td>Std Dev.=0.40</td>
</tr>
<tr>
<td>2.27 4.47 8.82</td>
<td></td>
<td></td>
</tr>
<tr>
<td>17.38 34.25</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Volume Distribution
Mean = 12.65µm
Median=11.58 µm
SA=13467 cm²/cm³
Std Dev.=8.29

Number Distribution
Mean = 0.38µm
Median=0.30 µm
SA=13467 cm²/cm³
Std Dev.=0.40
Statistical Issues with Distributions

- L Neumann, E T White, T Howes (Univ. Queensland) “What does a mean size mean?” 2003 AIChE presentation at Session 39 Characterization of Engineered particles November 16 - 21 San Francisco

Other references:
Does the Mean Match the Process?

- Particle size measurements often made to monitor a process
  - Size reduction (milling)
  - Size growth (agglomeration)
- Does the measured/calculated mean diameter describe the change due to the process?
- It depends on which mean used…
Size Reduction Scenario

10 x 1 μm

breaks into two smaller particles

100 μm

79.4 μm

79.4 μm
Size Reduction: Number Mean

Ten particles of size 1; one of size 100 units

Number mean = \( D[1, 0] = \frac{10 \times 1 + 1 \times 100}{11} = 10 \) units

Largest particle (100) breaks into two of 79.37
(conserves volume/mass: \( 2 \times 79.37^3 = 1 \times 100^3 \))

Have broken one

What happens to the number mean?

Mean = \( \frac{10 \times 1 + 2 \times 79.37}{12} = 14.06 \) units

Surprise, surprise a 40.6% increase!
Size Reduction: Volume Mean

Ten particles of size 1; one of size 100 units

Volume Moment Mean

\[ D[4, 3] = \frac{10 \times 1^4 + 1 \times 100^4}{10 \times 1^3 + 1 \times 100^3} \approx 100 \text{ units} \]

\[ \text{..} \]

Largest particle (100) breaks into two of 79.37
(conserves volume/mass: \( 2 \times 79.37^3 = 1 \times 100^3 \))

Have broken one
What happens to the \( D[4, 3] \)?

New \( D[4, 3] = \frac{10 \times 1^4 + 2 \times 79.37^4}{10 \times 1^3 + 2 \times 79.37^3} \approx 79.37 \text{ units} \)

This shows the expected behavior
Can You See the Problem?

10 x 1 μm breaks into two smaller particles

Number mean = 10
Volume mean = 100

Number mean = 14
Volume mean = 79
Size Growth Scenario

Ten 46.4 μm particles agglomerate into one 100 μm particle
Growth: Number Mean

Ten particles of size 1; ten of size 46.42
\[
D[1, 0] = \frac{10 \times 1 + 10 \times 46.42}{20} = 23.71 \text{ units}
\]

Ten of 46.42 agglomerate into one of 100
(conserves volume/mass: \(10 \times 46.42^3 = 1 \times 100^3\))
Have agglomerated half; does mean increase?

Mean = \(\frac{10 \times 1 + 1 \times 100}{11} = 10\) units
Over a 50% decrease!
Growth: Volume Mean

Ten particles of size 1; ten of size 46.42

\[ D[4, 3] = \frac{10 \times 1^4 + 10 \times 46.42^4}{10 \times 1^3 + 10 \times 46.42^3} \]
~ 46.4 units

(Note again the volume moment mean is dominated by the large particles)

Ten of 46.42 agglomerate into one of 100
(conserves volume/mass: 10 @ 46.42^3 = 1 @ 100^3)
Have agglomerated half; does mean increase?

\[ D[4, 3] = \frac{10 \times 1^4 + 1 \times 100^4}{10 \times 1^3 + 1 \times 100^3} \sim 100 \text{ units} \]
This shows the expected behavior
Can You See the Problem?

Ten 46.4 μm particles agglomerate into one 100 μm particle

Number mean = 24  Number mean = 10
Volume mean = 46  Volume mean = 100
Practical Implications

- Not just a “party trick” topic!
  “Do you know you can break particles and the mean will increase?”

- Serious. “Did an experiment. I thought I broke particles but the mean has increased”
  (REAL experience)

- Should be aware it can happen!

- Analyse whole size distribution, not mean alone.
What we’ll talk about

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- Understanding size metrics
- Differences between techniques
- Q&A
Why should one consider various methods of particle size analysis?

- Material suppliers and users employ many different types of instruments
- Use a different technique = get a different answer
- It is important to understand how analysis methods differ in order to know how to compare data
Size Range by Technique

- Electroformed Mesh Sieves
- Centrifugal Sedimentation
- Acoustic Spectroscopy
- Optical Microscopy / Image Analysis
- Electrical Conductivity
- Light Obscuration / Electrical Sensing Zone
- Dynamic Light Scattering
- Laser Diffraction

Size Range:
- 10 nm
- 100 nm
- 1 µm
- 10 µm
- 100 µm
- 1 mm
What Size is Measured?

Laser Diffraction
   Equivalent Spherical Diameter

Dynamic Light Scattering
   Hydrodynamic Radius

Image Analysis
   Lengths, Widths, Equivalent Spherical

Acoustic Spectroscopy
   Equivalent Spherical Diameter
Particle Shape Definitions

- **Acicular:** Needle-shaped, rigid
- **Angular:** Edgy, hard angles
- **Fibrous:** Thread-like, non-rigid
- **Granular/Blocky:** Irregular-shaped, low aspect-ratio
- **Spherical:** Regular-shaped, unity aspect ratio

**Aspect ratio:** Breadth / length OR Length / breadth

**Sphericity:** How spherical is the particle?

**Roundness:** How round is the particle?
Poll!

What are the shapes of your particles?
Poll!

Do you use multiple sizing techniques?
Hegman Gauge

- Used in paint and coatings industry
  - Device has tapered center channel
  - Slurry is placed in channel, then straight edge is drawn across it
  - “Hegman Number” is where particles disturb smooth surface of slurry
  - Information from largest particles only – no distribution
Sieves

Weigh % sample caught on known screen sizes

Solid particles 30 μm – 30 mm (and larger)

Advantages:

- Low equipment cost
- Direct measurement method
- No practical upper limit

Disadvantages:

- Limited lower range
- Time Consuming
- High Labor Cost
- Need Large Sample

Available through www.retsch.com
Electrical Sensing Zone

- **Coulter Principle**
  - Based on change in conductivity of aperture as particle traverses.
  - Requires conducting liquid.
  - Directly measures particle volume and counts.
  - High resolution
  - Used for blood cell counting more than industrial applications
Light Obscuration

Advantages:
- Particle count available
- USP<788> testing
- High resolution histogram

Disadvantages:
- Dilution required for particle size analysis
- Prone to cell clogging
Sedimentation

**Stokes Law**

\[
D = \sqrt{\frac{18\ \mu\ V_p}{(A - B)\ G}}
\]

- \(V_p\) = Settling velocity of discrete particle
- \(G\) = Gravity constant
- \(A\) = Density of Particle
- \(B\) = Density of Carrier Fluid
- \(D\) = Diameter of discrete particle
- \(\mu\) = Viscosity of Carrier Fluid

Note: assumes settling of spherical particle
Under-sizes compared to other techniques if non-spherical
Sedimentation Issues

Comparison of Brownian Motion and Gravitational Settling

(Movement in 1 second; Particle density of 2.0 grams/cc)

<table>
<thead>
<tr>
<th>Particle Diameter (In micrometers)</th>
<th>Movement due to Brownian Motion</th>
<th>Movement due to Gravitational Settling</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>2.36</td>
<td>&gt;&gt;</td>
</tr>
<tr>
<td>0.25</td>
<td>1.49</td>
<td>&gt;</td>
</tr>
<tr>
<td>0.50</td>
<td>1.052</td>
<td>&gt;</td>
</tr>
<tr>
<td>1.0</td>
<td>0.745</td>
<td>~</td>
</tr>
<tr>
<td>2.5</td>
<td>0.334</td>
<td>&lt;</td>
</tr>
<tr>
<td>10.0</td>
<td>0.236</td>
<td>&lt;&lt;</td>
</tr>
</tbody>
</table>

Below 1 micrometer, Brownian motion becomes an appreciable factor in particle dynamics. Gravity sedimentation may not be an appropriate measurement technique for very small particles.
Dynamic Light Scattering

Most common technique for sub-micron sizing
Range: 1 nm – 1 μm*

Particles in suspension undergo Brownian motion due to bombardment by solvent molecules in random thermal motion.

* Density dependent, when does settling become prominent motion?

Stokes-Einstein

\[ R_H = \frac{kT}{6\pi\eta D} \]
Manual Microscopy

- Count particles in a given field of view
- Use graticule to obtain size
- Repeat this process for a number of fields
- At least hundreds of particles must be sized

**Advantages:**
- Simple
- Inexpensive
- Can see shape

**Disadvantages:**
- Slow
- Measures very few particles
- Very tedious
Automated Microscopy

Static:
Particles fixed on slide, stage moves slide

Dynamic:
Particles flow past camera(s)
Automated Microscopy

Objective & camera

Image Acquisition and enhancement

Subjective or automatic

Thresholding

Decisions or black box

Image Processing

Measurements

- Advantages:
  - Quick size + shape info
  - Statistically valid
  - High resolution
  - Particle images

- Disadvantages:
  - Expense
  - Knowing which numbers are important
Acoustic Spectroscopy

- Acoustic signal sent into concentrated sample
- Detector measures attenuation \( f \) (frequency, distance from source)

Advantages:
- Can accommodate high sample concentrations (no dilution)
- Rheological properties
- Also measure zeta potential

Disadvantages:
- Need at least 1 wt% particles
- Need to know wt%
- Minimum sample = 15 ml
Laser Diffraction

- Converts scattered light to particle size distribution
- Quick, repeatable
- Powders, suspensions
- Most common technique
Poll!

Which techniques do you use?
What we’ll talk about

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- Q&A
Thank you

Gracias

Danke

Merci

おおかく

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