Producing a Calibrated Leak Standard With Laser Technology

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What is a “Calibrated Leak”?

Definition for CCIT case:

Calibrated leak is the opening in the wall of a container in a shape of an orifice or a crack that gives a specific flow rate for a given gas or liquid at given pressures applied to both sides of the material wall where the leak is made.

Container Varieties, Elements, and Constituent Materials

Containers Varieties

- **Vials:** Stable for samples/reagents.
- **Syringes:** For accurate dosing in healthcare.
- **Pouches:** Light, flexible for various products.
- **Bags:** Versatile for fluids/goods.

Container Elements

- **Tubing:** Fluid transfer within/between units.
- **Caps:** Secure seals for content integrity.
- **Lids:** Accessible seals for containers.
- **Plungers:** Precision dispensing in syringes.

Constituent Materials

- **Metals/Alloys:** Durable and sterilizable.
- **Glass:** Inert and clear for labs/medicine.
- **Plastics:** Flexible design for multiple uses.
- **Composites:** Tailored for specific needs.

PDA Parenteral Drug Association

Container Conditions

Open vs. Closed

- Open containers are directly accessible for drilling and measurement.
- Closed containers require specific approaches to access without compromising seal integrity.

Filling Conditions

- **Filled Containers (Non-Medical Contents):** Can contain various substances; processes and procedures must account for potential interactions with laser drilling.
- **Pre-filled Containers**
  - **With Head Space:** Allows for pressure adjustments and gas expansion, affecting drilling accuracy and calibration.
  - **Minimal Head Space:** Techniques must ensure no distortion of container shape or compromise of the seal during processing.

Sealing Methods

- Capped, crimped, welded, or temperature-sealed methods influence the selection of laser drilling and calibration techniques.

Contents State

- **Liquid:** Requires careful handling to avoid contamination during drilling. Limited ability for precise drilling and measurements
- **Empty:** Easier to drill but may still need precision to maintain container structure.

Guidelines for Laser-Drilled Leak Placement

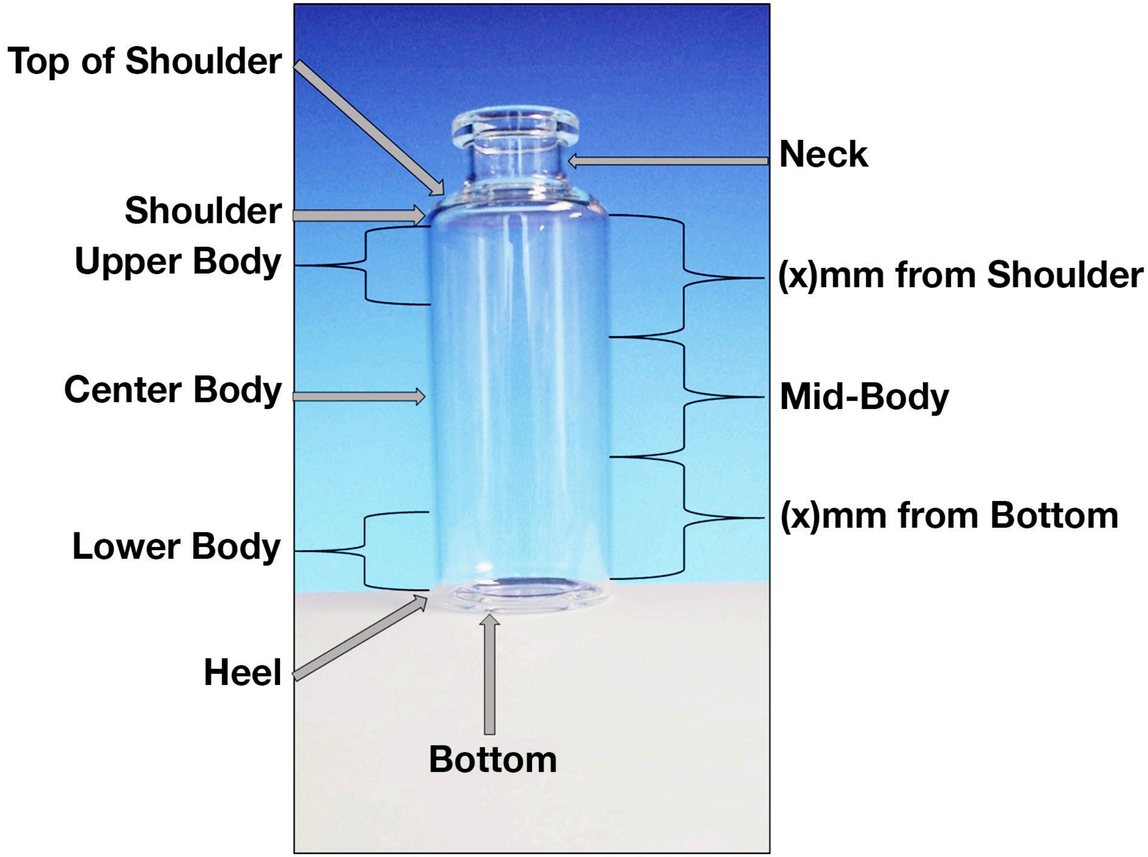
Recommended Locations

- **Thermal Seal Vicinity:**
  To assess seal integrity.
- **Closure System Intersections:**
  Where closure elements meet.
- **High Stress Areas:**
  For testing durability.
- **Critical Contamination Points:**
  To safeguard product safety.

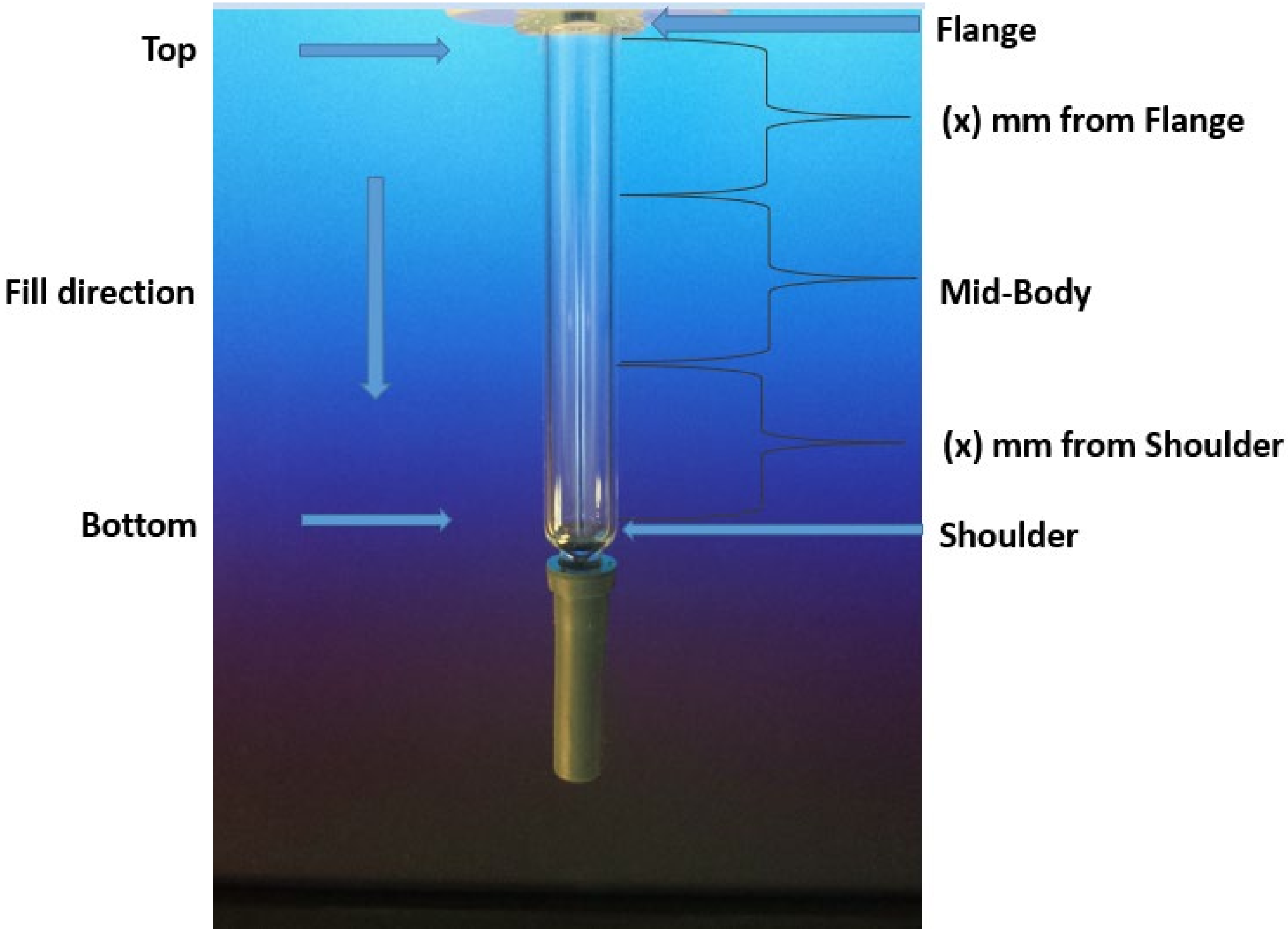
Placement Considerations

- **Accessibility:**
  Must be reachable for laser drilling and calibration.
- **Container Integrity:**
  Should remain intact, with no content compromise during procedures.

Common Locations for Laser Drilled Leak



Common Sites for Syringe Laser-Drilled Leak (Cont.)



Methods of Creating Laser Drilled Leaks

Standard Process (SP)

Emulates common real-world defects with potential for cracks.

Lower Aspect Ratio for standard damage replication.

- Aspect Ratio (AR), calculated as the length of the channel divided by its diameter, which is indicative of the process's accuracy and finesse.

Controlled / Cold Ablation (CA)

Precise drilling for clean, round channels without cracks or thermal affects.

High Aspect Ratio and excellent dimensional control.

Combined Process (CP)

Hybrid of SP's realism with CA's precision and controlled geometry.

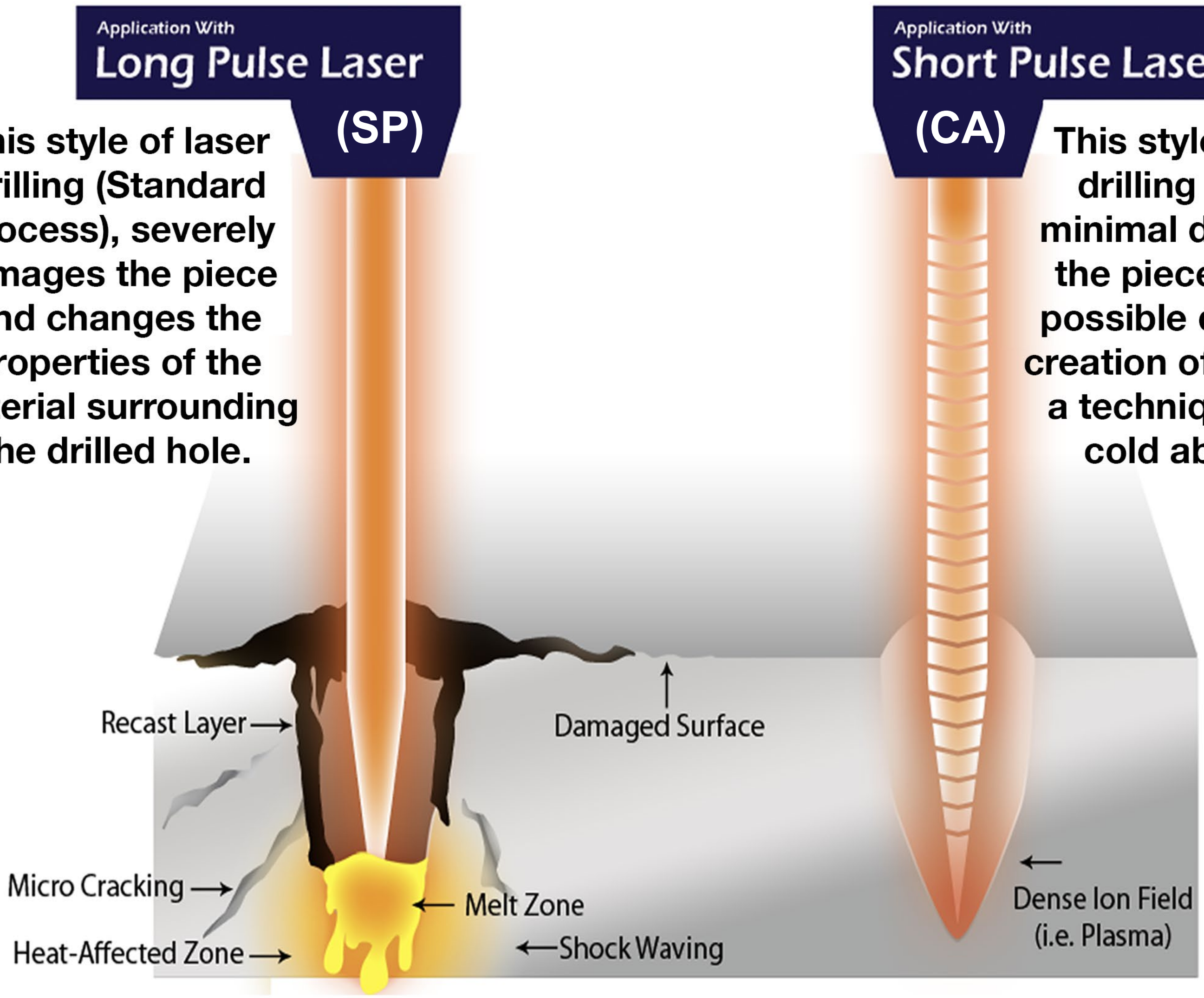
Offers a balanced outcome, integrating benefits of both methods.

Standard Process

Cold/Controlled Ablation

This style of laser drilling (Standard Process), severely damages the piece and changes the properties of the material surrounding the drilled hole.

This style of laser drilling creates minimal damage to the piece. This is possible due to the creation of plasma in a technique called cold ablation.



Standard Process (SP)

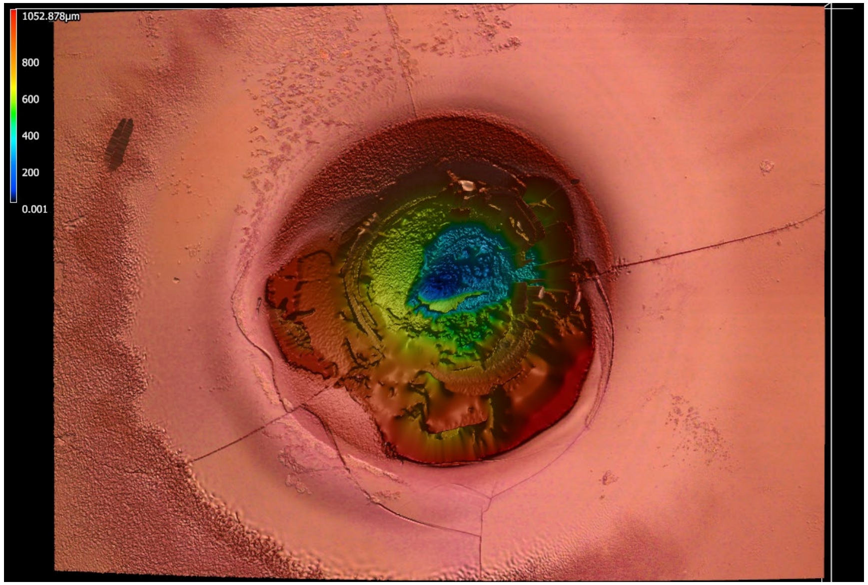
Overview
Imitates defects found in typical real-world environments.
Tends to produce channels with imperfections like cracks.

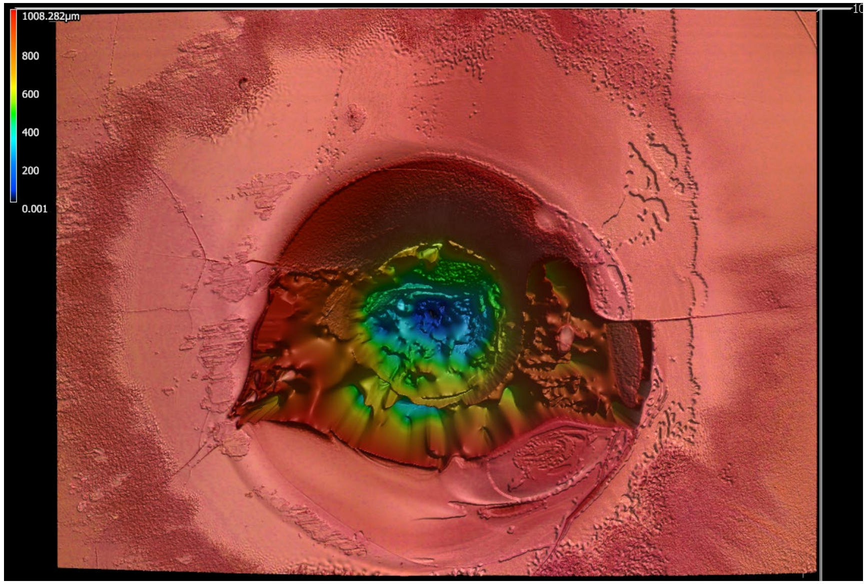
Aspect Ratio (AR) Considerations
AR is lower compared to other methods, which can be preferable for certain applications where standard damage replication is desired.
AR is a key metric, derived from channel length to diameter, serving as an indicator of the channel's proportionality and precision.

Process Suitability
Ideal for applications requiring a model of common defect scenarios.
Often used in testing environments that simulate everyday use and wear.
Process is only suitable for glass containers

Standard Process (SP)

Top View





Controlled Ablation (CA)

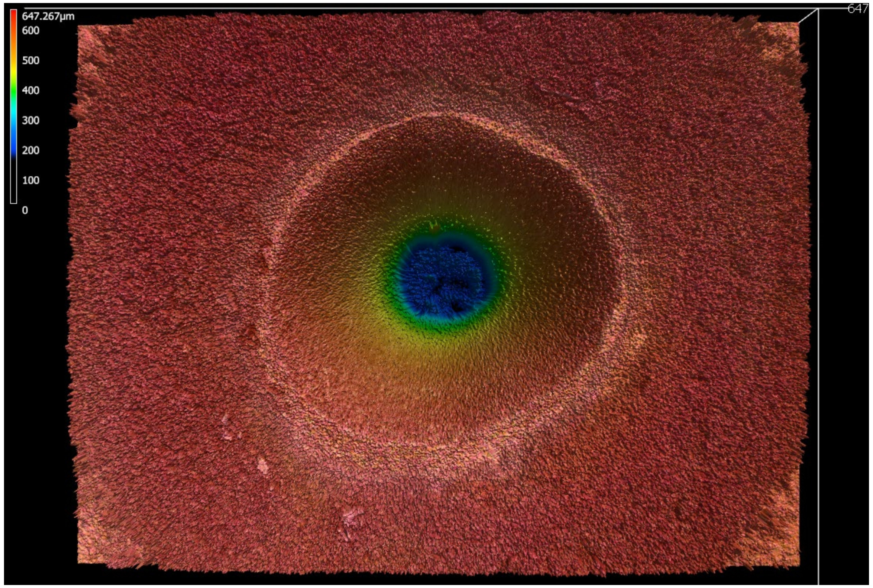
Precision Drilling
Delivers high-precision channels that are consistently clean and crack-free.
Utilizes a controlled laser to avoid heat-induced damage or stress to the material.

High Aspect Ratio
Achieves a higher AR, signaling a channel that is longer relative to its diameter.
High degree of control and precision of hole geometry.
Ideal for scenarios requiring high-precision leakage paths without the risk of residual stress.

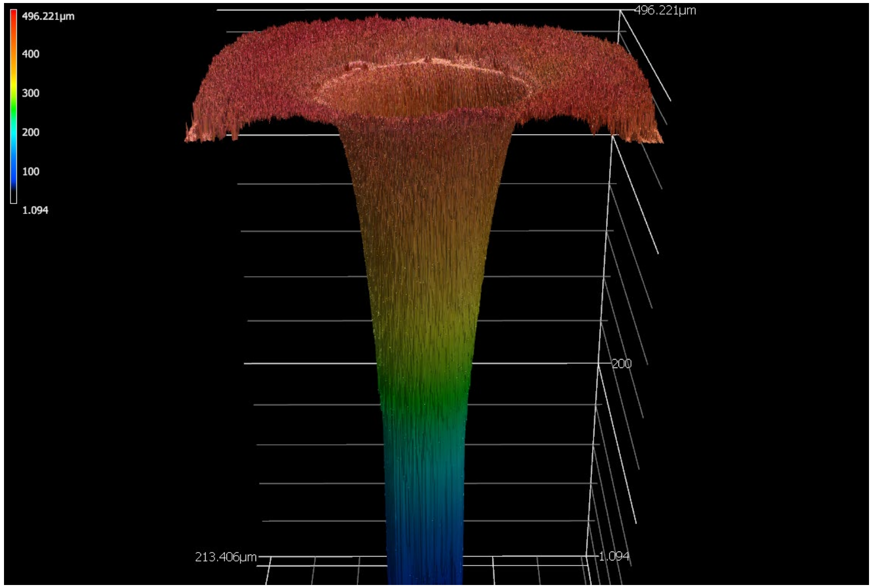
Application Scope
Suitable for all material types including multi-layer materials or when integrity of the surrounding structure is paramount.
Also employed in high-tech industries or in medical applications where precision is non-negotiable.
Smaller feature sizes and tighter tolerances

Controlled Ablation (CA)

Top View



Profile View



Combined Process (CP)

Hybrid Technique

Merges some of the geometry of the SP Process(lower AR) with the precision and control of the CA Process.

Designed to benefit from the strengths of both SP and CA, providing a versatile solution.

Balanced Outcomes

Provides a balance between realistic defect emulation and the need for precision.

The combined approach can tailor the drilling process to specific requirements of the package

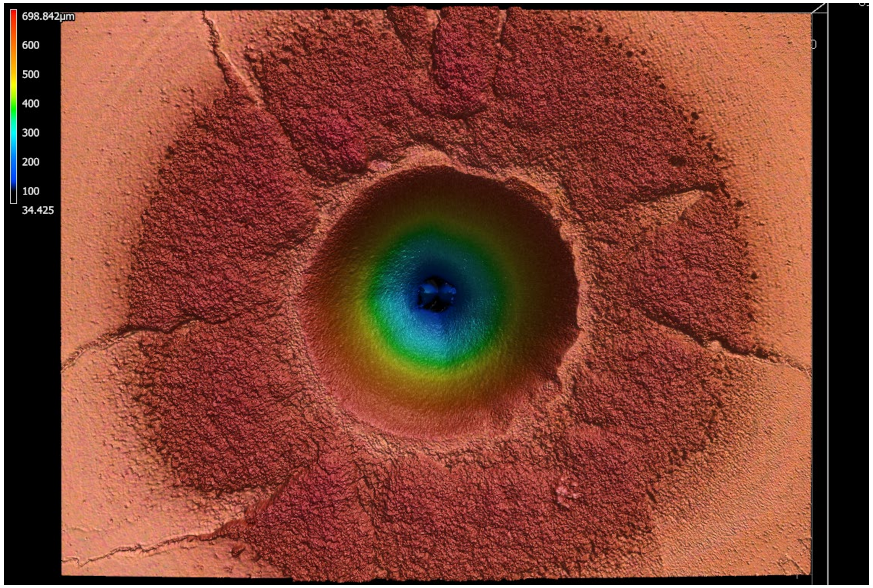
When to Use CP

When a balance is needed between creating desired profile and maintaining clean, precise channels in virtually any material type while also maintaining integrity of surrounding material.

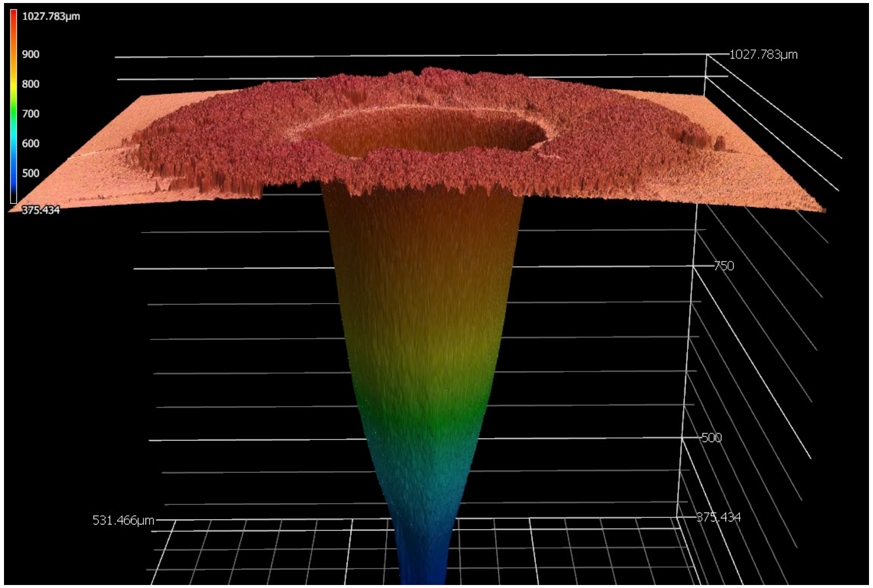
Optimal for complex applications where reduced aspect ratio and accuracy are desired. May also achieve smaller

Combined Process (CP)

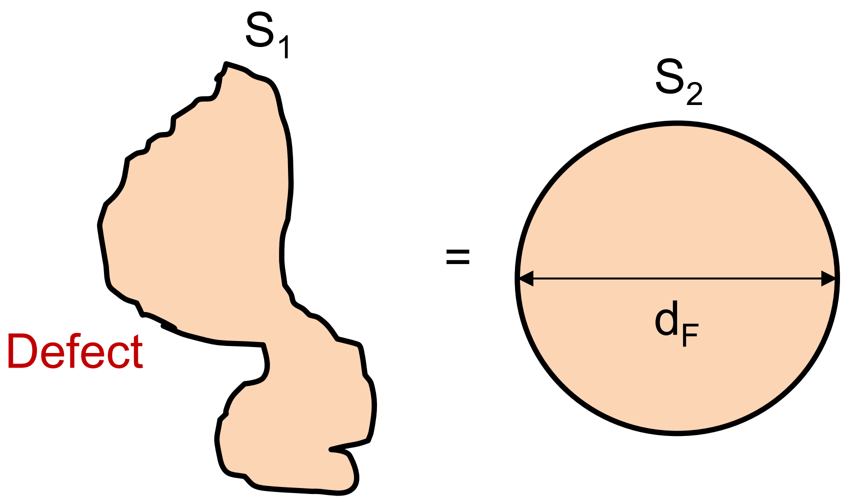
Top View



Profile View

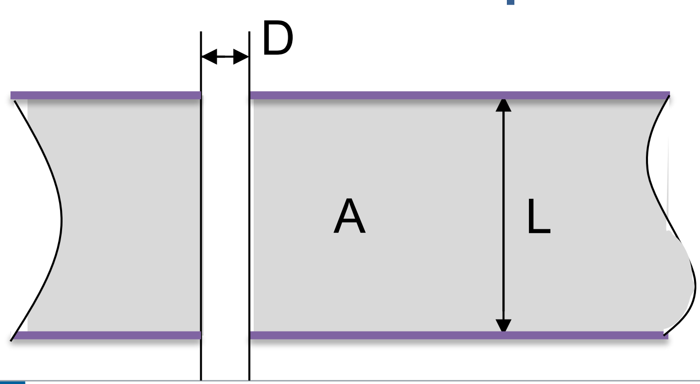


Formula: Flow Effective Diameter (FED)



$$S_1 = S_2 = \pi \frac{(d_F)^2}{4}$$

Aspect Ratio



L = (mm) length of a channel
D = (mm) diameter of a channel
A = L/D (n*10, n*100, n*1000)

Flow Effective Diameter (FED)

Definition

Flow Effective Diameter (FED) refers to the diameter of a circular channel which would enable a flow rate equivalent to that through a leak of any arbitrary shape and size.

Application in Flow Dynamics

Concept of "Flow Effective Diameter" is crucial for calculating flow rates. It quantifies the effective area available for fluid passage, impacting the volume of fluid that can be transported within a given time frame at a specific velocity.

Key Characteristic of FED

An essential quality of the Flow Effective Diameter (FED) is its uniformity (invariant) across various fluid mediums.

Note: The FED measurement is not affected by the nature of the fluid—whether gas or liquid—flowing through the system, remaining unchanged for different substances.

Principal Techniques for Calibrating a Laser-Drilled Leak

In-Process Calibration

Advantage: 100% non-destructive testing, every leak directly measured and calibrated.

Ideal for: High-value or critical components requiring absolute leak integrity.

Cross Calibration

Method: Combines non-destructive and potentially destructive phases.

Benefits: 100% direct measurement and calibration of a smaller sample.
Provides reference data for subsequent non-destructive testing.

Suitable for: Applications where some destructive testing is acceptable for establishing calibration standards.

Statistical Calibration

Method: Non-destructive testing of a sample, final certification based on statistical analysis.

Note: In case of sealed containers, destructive testing/sampling may be required

Advantage: Limits the amount of testing required. Preserves most containers non-destructively while ensuring overall quality.

Limitations: Relies on accurate sampling and statistical models.
May not be suitable for critical applications requiring absolute certainty.

Ideal for: Sealed containers or Large-scale production runs where 100% measurement is not required

Matching Metrology Methods to Container Orifice/Leak Characteristics

Introduction:

Container condition(empty/filled, Open/sealed) may determine possible laser methods/process types and locations that are available.

Selecting the appropriate metrology method is crucial for accurate and reliable orifice characterization.

Key Metrology Techniques

1. Mass Flow & Volumetric

Measures flow or volume change.

Best for open, unfilled containers at room pressure.

2. Optical Microscope

Assesses size and shape at a micro level.

Best for thin wall, open, unfilled containers at room pressure.

3. Scanning Electron Microscope (SEM)

Provides detailed size and shape at a nano level.

Best for thin conductive material types

4. Bubble Test

Detects orifice existence and estimates size.

Works with any container, but less precise.

5. Laser Scanning Microscope (LSM)

Non-invasively maps orifice in 3D.

Flexible for all container conditions.

Sample Process Considerations and Documentation

Laser Process Considerations

Specialized techniques are used to prevent clogging of small orifices during drilling.

Assist gases or purified air may be employed to eject debris from the drilling site.

Methods are in place to ensure that particles created by the laser do not remain on the sample.

Protocols for Filled Containers

Protect the content from contamination.

Prevent obstruction of the drilled channel.

Sample Documentation and Identification

Each drilled sample is accompanied by a detailed report featuring either the effective flow diameter, optical diameter or flow rate, and testing parameters/conditions.

Proper identification of each container is critical. Parts are individually packaged and labeled with unique part ID or s/n.

Although marking directly on the sample is typically prohibited, this is possible if desired

Recommended Handling/Storage Practices

Part preparation for shipment

- Use container Mfr. supplied packaging(tubs/trays) where possible
- Reduce risk of breakage(glass)by carefully packaging containers.
- Limit movement within package.

Handling/Storage of laser drilled sample

- Handle laser drilled samples with powder free Nitrile gloves
- Avoid touching or handling part at or around laser drilled area
- Keep part in dust-free and moisture-free environment
- Place sample into supplied packaging after use

Alternative laser drilled solutions for CCIT

Laser drilled Needle Orifice

- Known calibrated leak with flow effective diameter(FD)
- Allows for insertion into elastomer stoppers without additional sealant
- Ideal for sealed/filled containers
- Stainless steel material. Can be re-used.

Laser drilled Disc Orifice

- Known calibrated leak with flow effective diameter(FD)
- Simple design allows for application on various container types
- Ideal for flexible, sealed/filled containers
- Stainless steel material. Can be re-used.
- Requires additional sealant at application site
- *Product in development for a self-adhesive option

Thank You!

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