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Producing a Calibrated Leak Standard With Laser Assisted Technology

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What is a “Calibrated Leak”?

Definition for CCIT case:

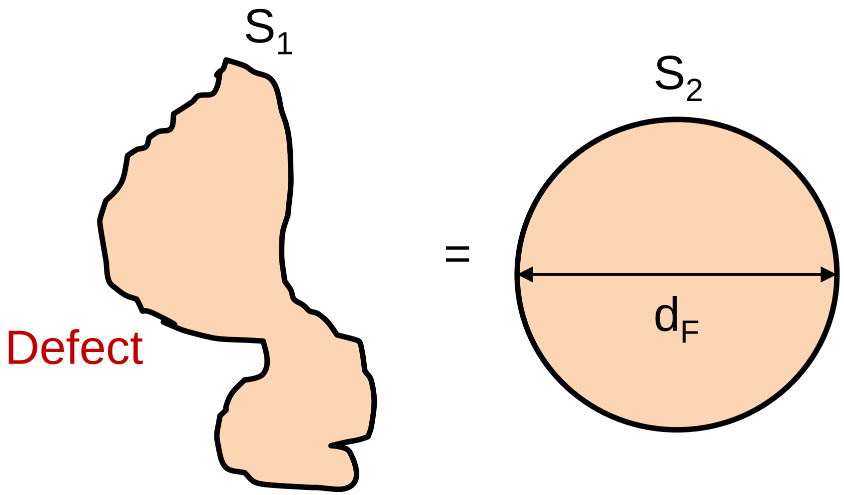
Calibrated leak is the opening in wall of a container in a shape of orifice or crack that gives a specific flow rate for a given gas or liquid at given pressures applied to both sides of the material wall where the leak is made.

Geometrical Properties of a "Calibrated Leak"

Method: Optical measurement for characterization of the different geometrical shapes

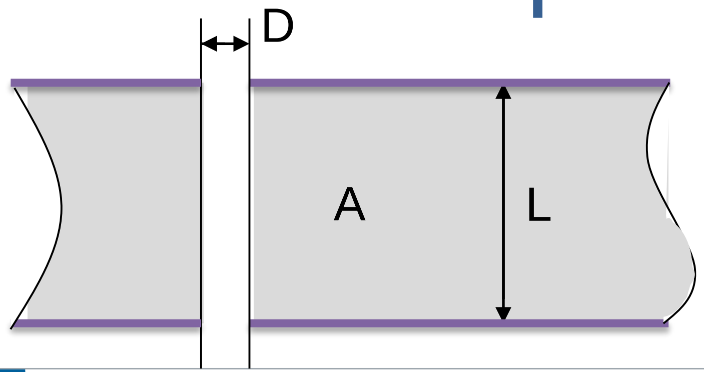
- Round orifice: The diameter and the depth of a channel
- Rectangular Slit: The slit width, length and depth of the channel
- Tortuous shape:
  The surface area and depth of the channel*

Flow Effective Diameter



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

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**

  FED is the diameter of the channel with the round cross section area, that causes the flow rate equivalent to the leak with arbitrary shape

- **Main property of FED**

  An invariant with respect to types of gasses or liquids used in the flow measurement.

Most common container types, elements, and materials

- **Container types**: Vials, Syringes, Pouches, Bags of any shape.

- **Elements of containers and systems:** tubing, caps, lids, plungers etc.

- **Container materials:** All types of metals or metal alloys, glass, plastics or composites

Open Containers

Most common container conditions

- **Open or closed** (capped, cramped, welded, temperature sealed)

- **Empty**
- Pre- filled with head space or almost no head space.
- Contains: liquid or dry.

Sealed and filled containers

What is laser drilled Calibrated Leak?

Definition:

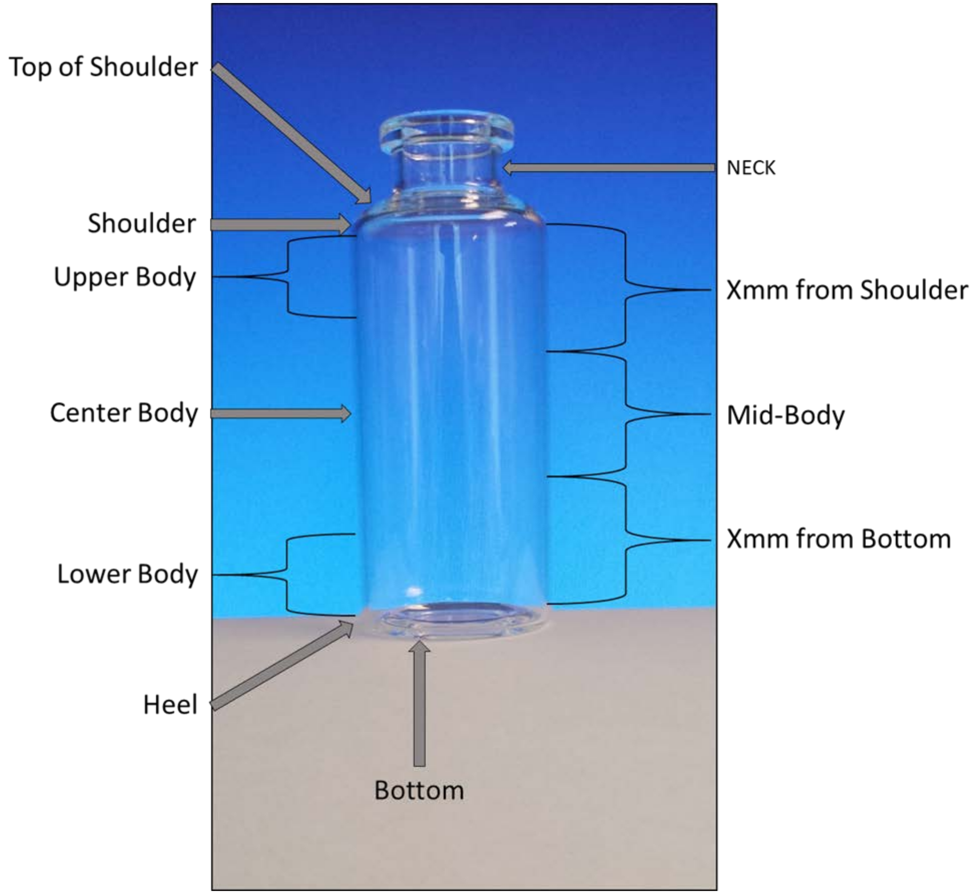
- Laser Drilled Calibrated Leak-
  the laser drilled channel through material wall that gives a specific flow rate for a given gas or liquid at given pressures applied to both sides of the material wall where the leak is made or has known geometrical shape and dimensions.

Most common locations for the laser drilled leak

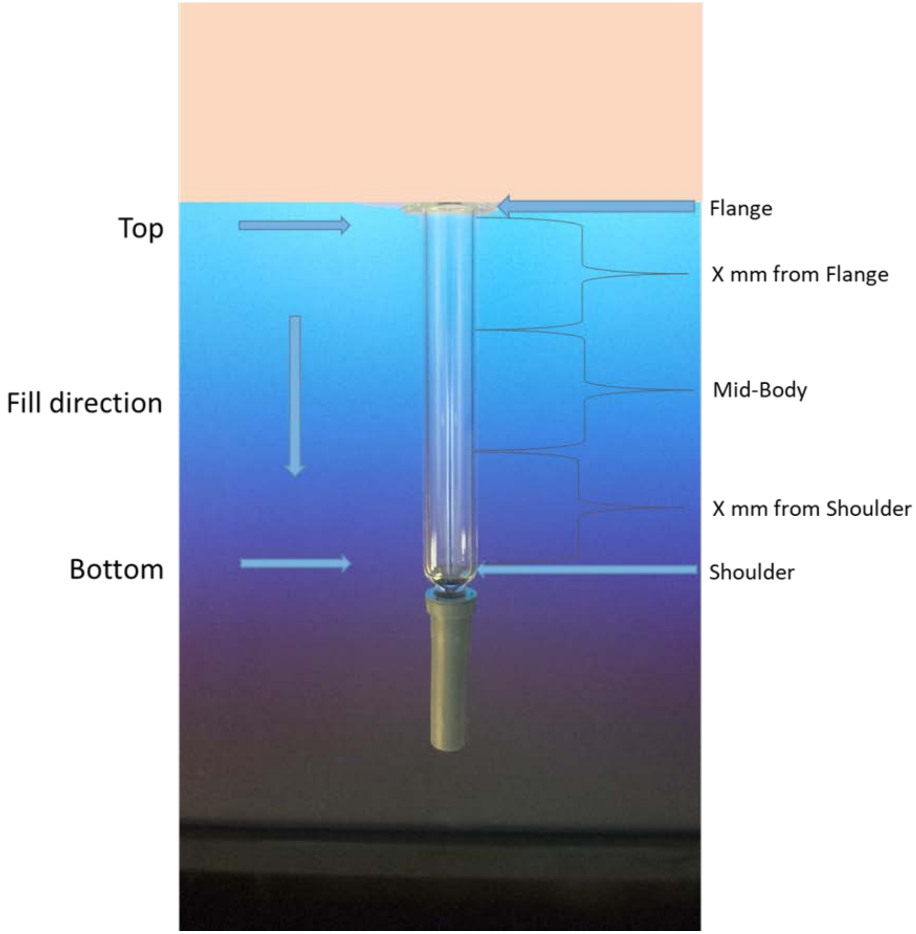
- **Recommendations for location:**
  Closer to a thermal seal; connection interface of different parts of the closure system; area that suffers the most use or stress; most critical area for contamination.

- **Limitations:**
  It has to be reachable to a laser beam or measurement instrument for calibration without damaging or modifying the other parts of the container closure or its content.

Common Hole Locations in Glass Vials



Common Hole Locations in Syringes



Major methods for creating a Laser Drilled Leak

- **Controlled Ablation (CA)**

Very clean, round, no cracks channel with high (AR) aspect ratio AR= length/ diameter

- **Standard laser drilling process (SA)**

Simulates the real defect with cracks. AR is small

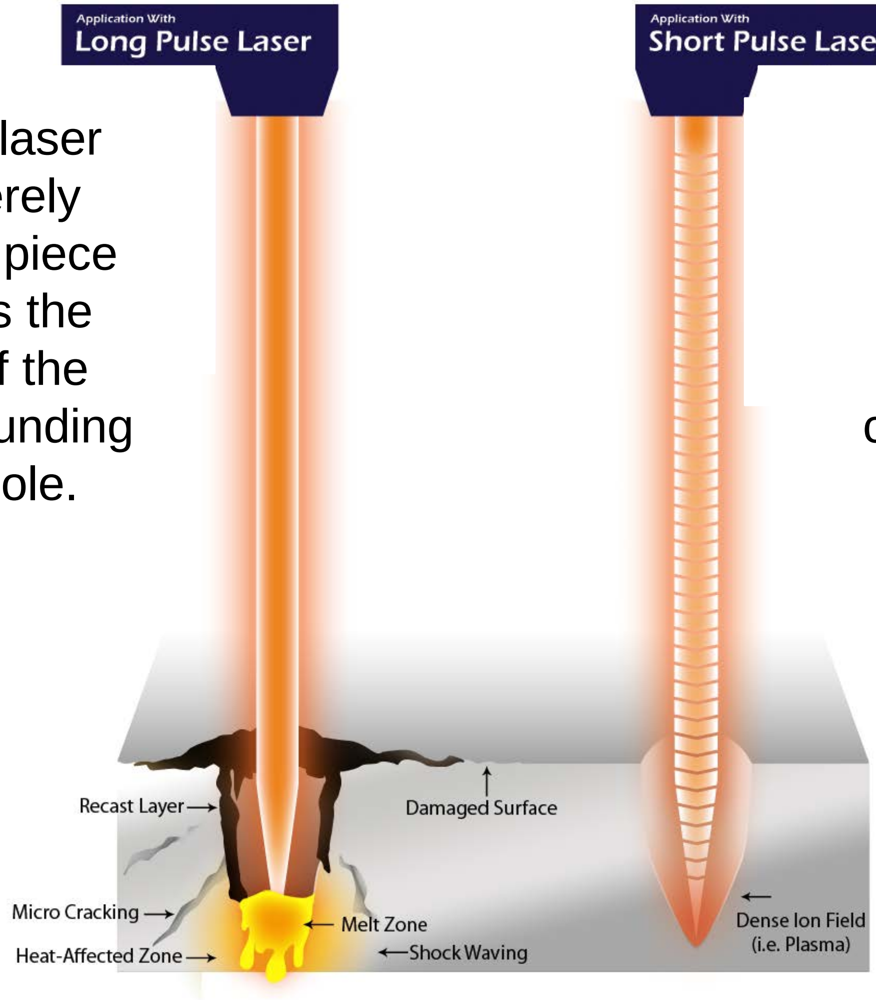
- **Combined process (CP)**

Controlled Ablation by a Laser Beam (CA)

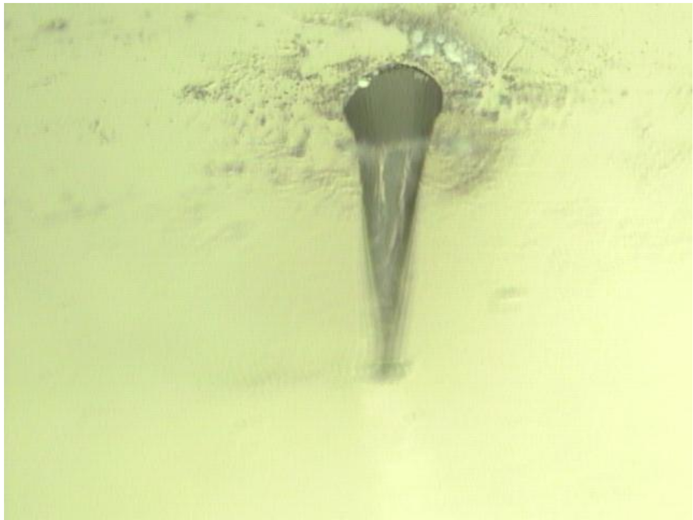
- **Definition**

CA is the process of the removing material from a solid surface by irradiating it with an ultrashort laser pulse.
The dominating process here is ablation (evaporation) of the material by converting it to a plasma. The surface melting and heat transfer is very minimal or none.

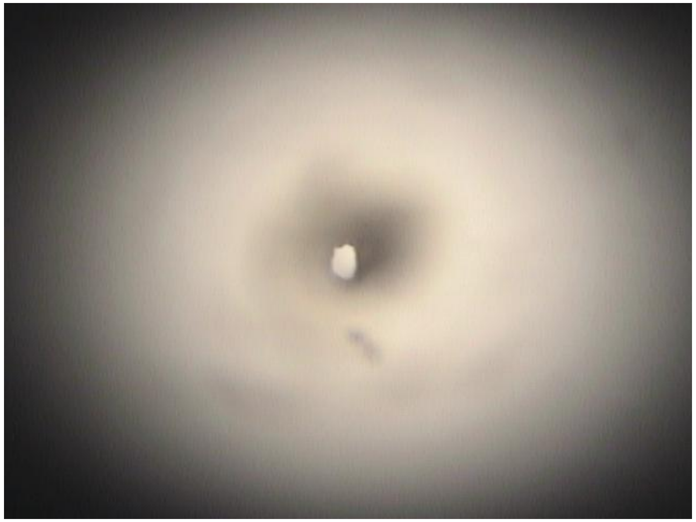
This style of laser drilling severely damages the piece and changes the properties of the material surrounding the drilled hole.



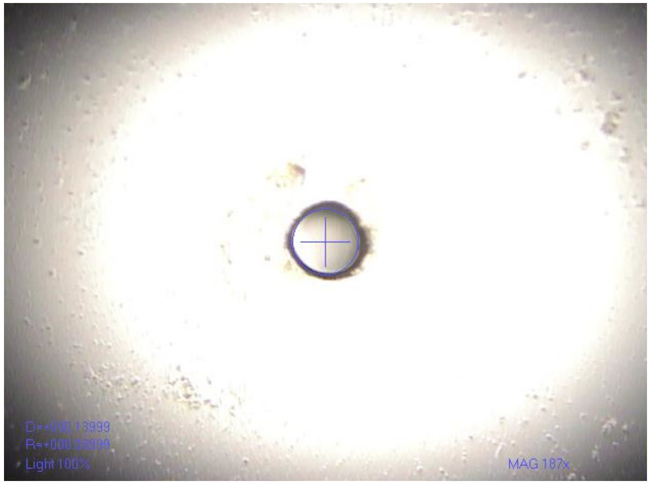
This style of laser drilling with minimal damage to the piece. This is possible due to the creation of plasma in a technique called cold ablation.



Cross Section

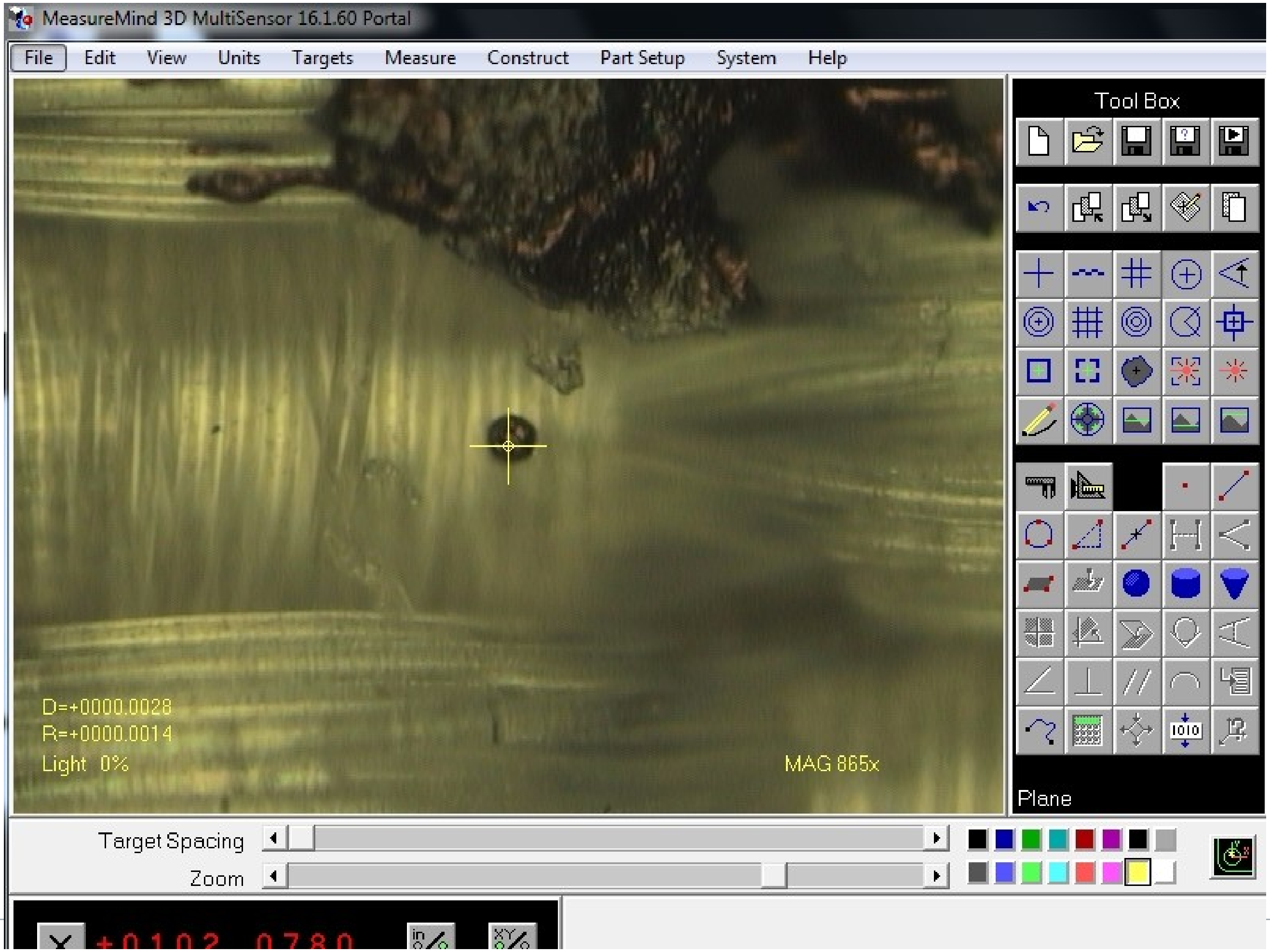


Bottom View

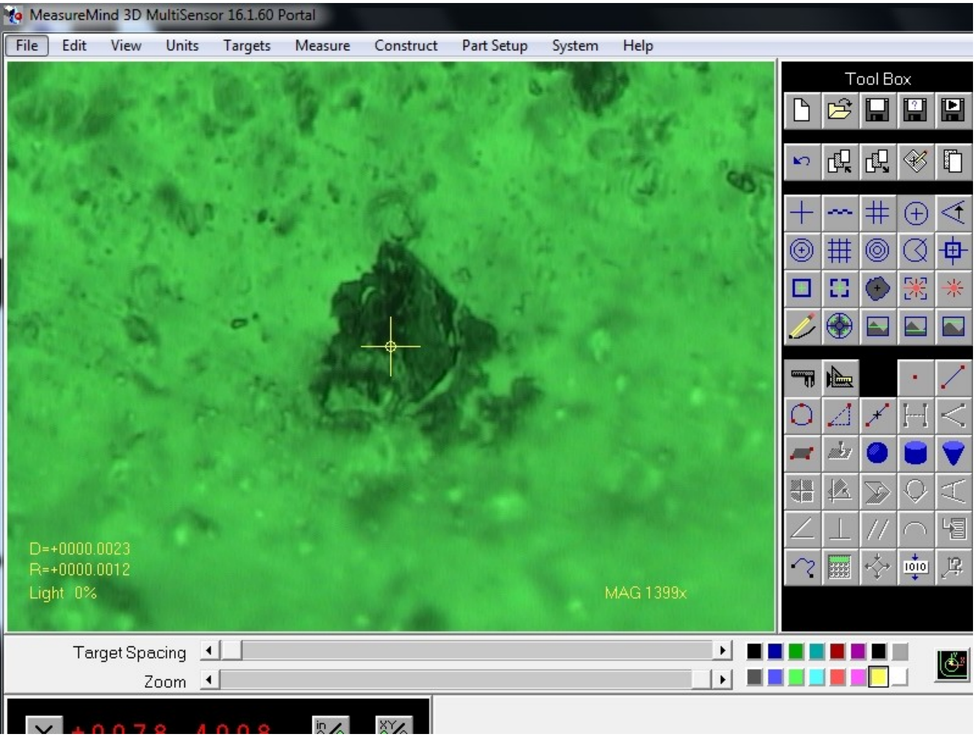


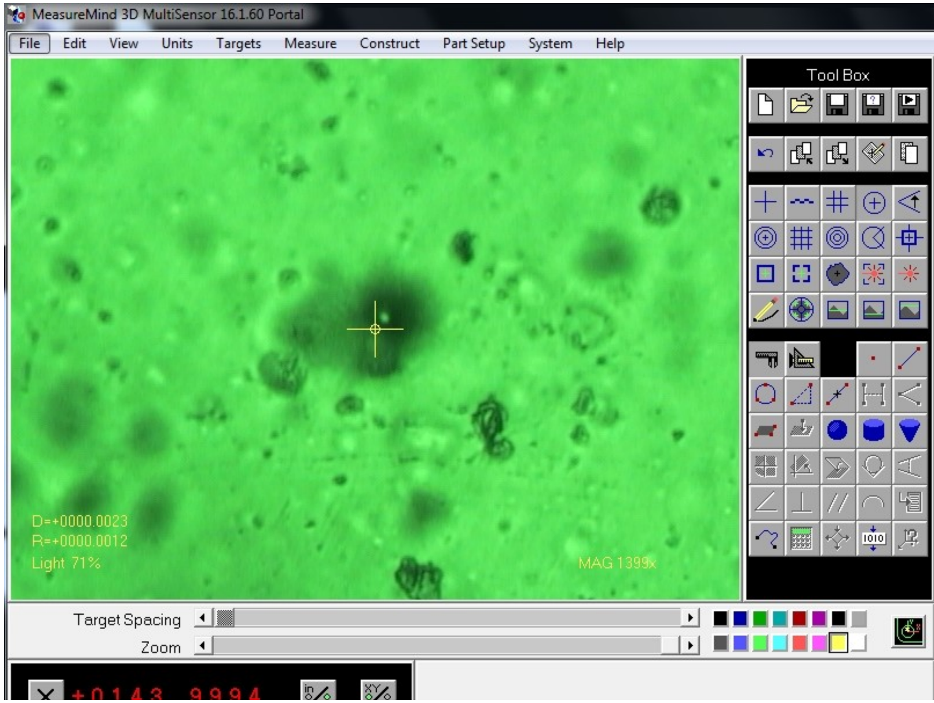
Top View

Interwoven Plastic Bag

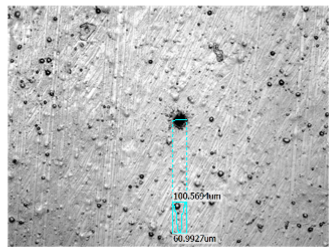


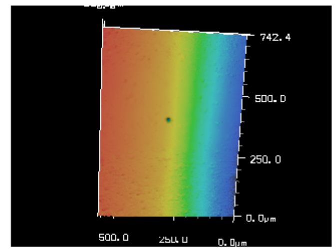
Plastic Bag (20µm Thick)

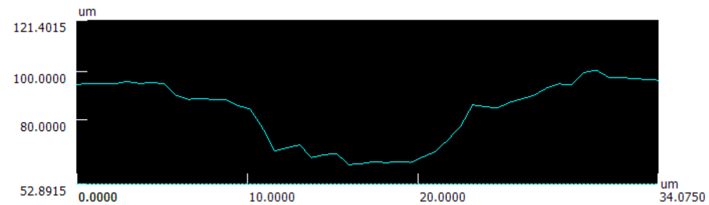




Surface Profile of Laser Drilled Orifice







Profile1	Horz. dist.	Hght. diff.	Hght. ave.	Angle	C.S. length	C.S. area	R	Comment
All	34.0750um	1.7475um	83.0830um	2.9358°	107.9391um	1050.6640u...		
Seg.1								
Seg.2								
Seg.3								
Seg.4								
Seg.5								
Seg.6								
Seg.7								
Seg.8								
Seg.9								
Seg.10								
Total								
Max.								
Min.								
Ave.								
Std. DV								
3sigma								

Profile1
Line type : Set 2pt.
Ave: None
Correction : Smooth intensity None, DCL/BCL None, Smooth height None, Correct tilt None
Ref. value1 : 121.4015um
Ref. value2 : 52.8915um
Step : 68.5100um

Properties of a “Calibrated Leak” 2

- **Flow characterization of the crack:**
  Measuring the flow rate through the opening

- **Cross calibration between Optical and Flow measurement**

  If the correlation between the Optical and Flow measurements is established then it becomes possible to introduce the **Flow Effective Diameter**.

Major approaches for calibration of a Laser Drilled Leak

- **In-process calibration**
  Absolutely non- destructive. In this case 100% of the leaks are directly measured and calibrated.

- **Cross calibration**
  May include the destructive phase. In this case 100% of the leaks are directly measured and calibrated.

Major approaches for calibration of a Laser Drilled Leak (cont.)

- **Statistical calibration**

Indirect measurement, when the final certification is done based on sampling of drilled containers. This method is destructive for the sampled containers and non- destructive for the main batch of containers.

Understanding the geometrical metrology for Calibrated Leak

- **Optical instruments:** laser based microscopes

- **Goals for cross calibration**
- Deriving the correction multiplies between optical and flow methods, based on the precisely measured geometrical dimensions of the channel and its flow rate.
- Creating the cross calibration metrology standards.


FLASH
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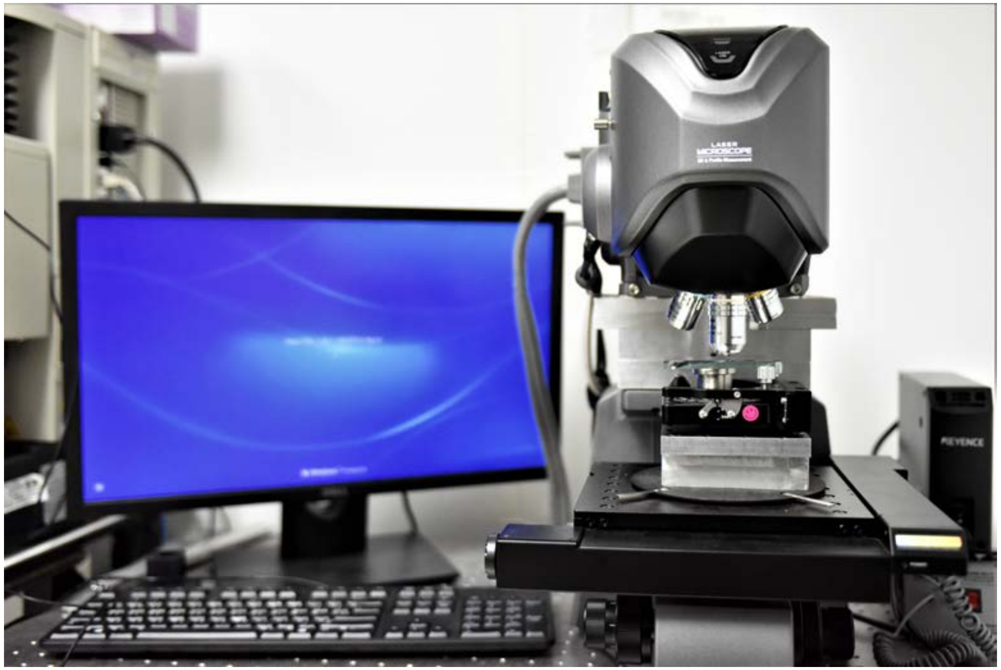
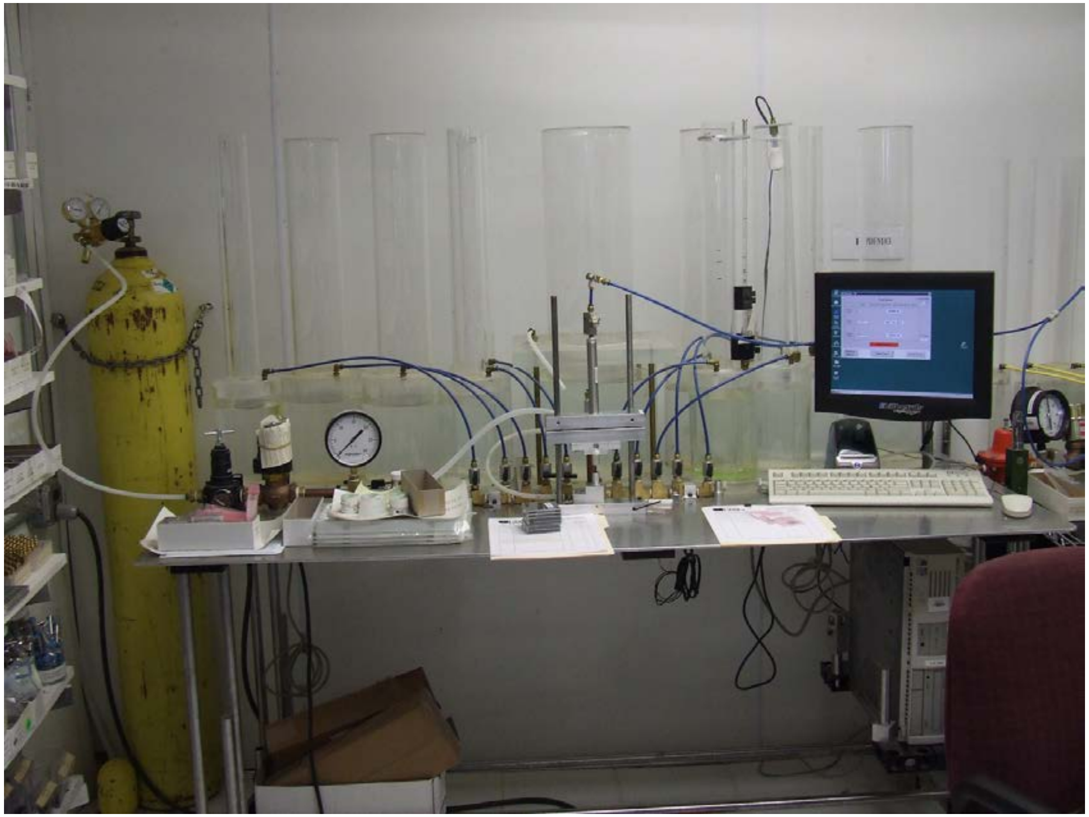


Understanding the main flow metrology for Calibrated Leak

- **Volumetric flow** main relations, metrology and instruments.

- **Mass flow** main relations, metrology and instruments.

Flow measuring method by water displacement



Standard vs Actual Volumetric Flow (cont.)

- Standard volumetric flow: $Q_{std} = m/\rho_{std}$
  - The volumetric flow that would exist at the standard pressure (P_{std}) and temperature (T_{std}) for the same mass flow
  - The fluid density is determined at the standard temperature and pressure conditions: $\rho_{std} = \rho(P_{std}\ T_{std})$

Understanding Standard Volumetric Flow

- The Relationship between Q, Qstd, and m

$$Q_{std} = \frac{m}{\rho_{std}} = \frac{\rho_a Q_a}{\rho_{std}} \qquad \rho_{std} = \rho(P_{std} T_{std})$$

- For a fixed choice of standard conditions

- Qstd is directly proportional to mass flow
- Qstd is essentially a mass flow in volumetric units

Understanding Standard Volumetric Flow (cont.)

- To distinguish between Qa and Qstd the following notation is sometimes used
  - Actual volumetric flow: an "a" is sometimes placed before units
    Example: acfh, accm
  - Standard volumetric flow: an "s" is sometimes placed before units
    Example: scfh, sccm

Standard vs Actual Volumetric Flow

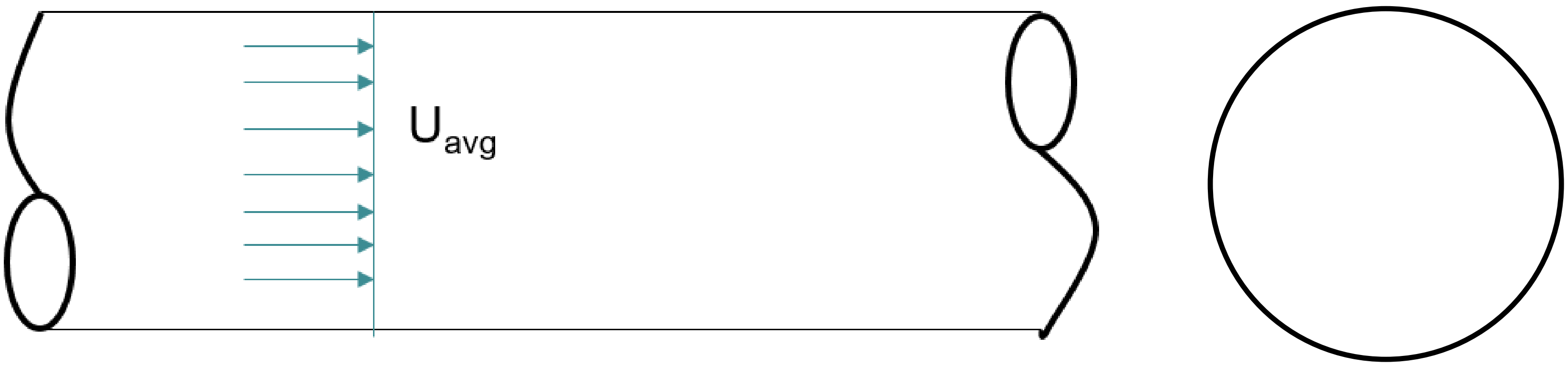
- Volumetric Flow: Q (or Qa)
  - The volumetric flow at the actual pressure (Pa) and temperature (Ta) conditions that the flow was measured
- Mass Flow: m = ρQ (or m = ρaQa)
  - The fluid density is determined at the actual temperature and pressure conditions: ρa = ρ(PaTa)

Mass Flow (Simplified)

$$\dot{m} = \rho(U_{avg} A) = \rho Q$$

U_{avg} = Average Fluid Velocity
A = Cross Sectional Area
ρ = Fluid Density

Pipe Flow Example



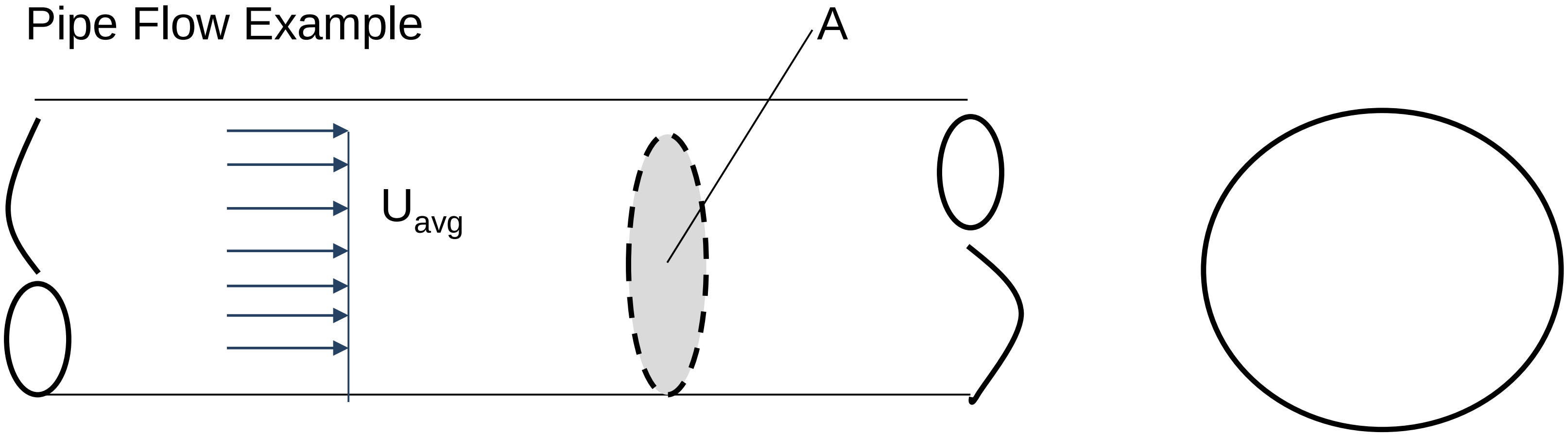
SI unit for mass flow: kg/s, kg/min, kg/hr, g/s, g/min, g/hr
Other units: lb/s. lb/min, lb/hr

Volumetric Flow (Simplified)

$$Q = U_{avg} A$$

U_{avg} = Average Fluid Velocity
A = Cross Sectional Area

Pipe Flow Example



SI unit: m^3/s, m^3/min, m^3/hr
Other units: Lpm (liters/min), gal/min, cm^3/min (ccm), ft^3/hr (acfh) …

Fluid Density

- Most flowmeters measure Q (or Uavg)
- Mass flow is usually the desired measurand
- Density is needed to convert to mass flow
  - –Densitometer – Liquid or high pressured gas
  - –Equation of State: $\rho = \rho(T, P, x_k)$

T = Temperature Measurements
P = Pressure Measurements
x_k = Composition Measurements

Fluid Density of Gases

- Equation of State: $$\rho = \frac{PM}{ZR_U T}$$
- RU = 8.314172 J/mol*K (universal gas constant)
- M = Molar Mass (Molecular Weight)
  - Pure gas: M determined using reliable reference
  - Gas Mixture:
- T = Temperature in absolute units (K or °R)
  - T(K) = T(°C) + 273.15
  - T(°C) = (T(°F) -32) / 1.8
  - T(°R) = T(°F) + 459.67

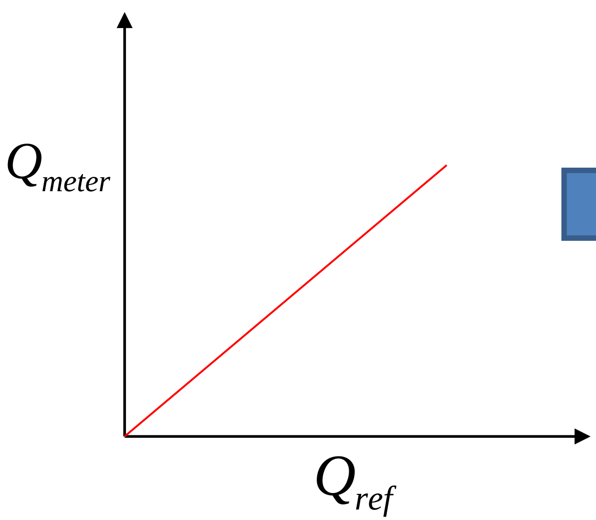
Flow Meter Measurements

Depending on the flow meter method of operation the fundamental measurand can be either

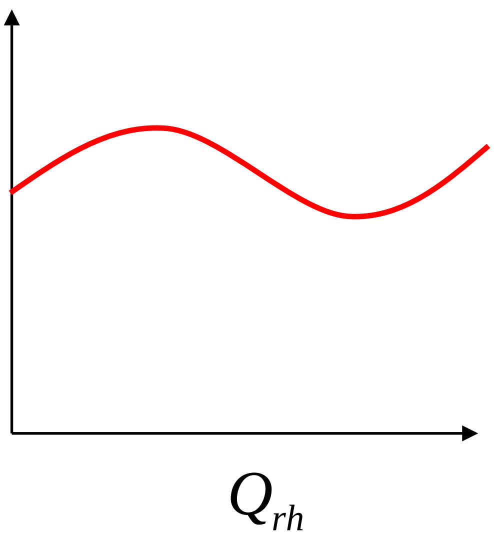
- Mass Flow
- Volumetric Flow
- Velocity (cross sectional area must be measured)

Reynolds Number and Discharge Coefficient

$$\mathrm{Re} = \frac{\rho D \bar{u}}{\mu} = \frac{4Q\rho}{\pi D \mu} = \frac{inertial}{viscous}$$

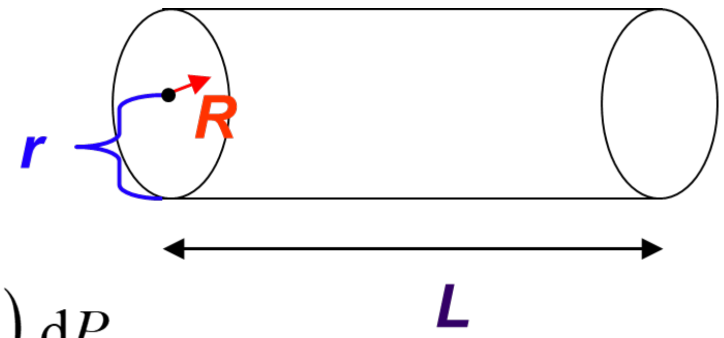


$$C_d = \frac{Q_{ref}}{Q_{rh}}$$



Laminar Flow Meter: Physical Model

$$u(R) = \frac{-(R^2 - r^2)}{4\mu}\frac{dP}{dx}$$

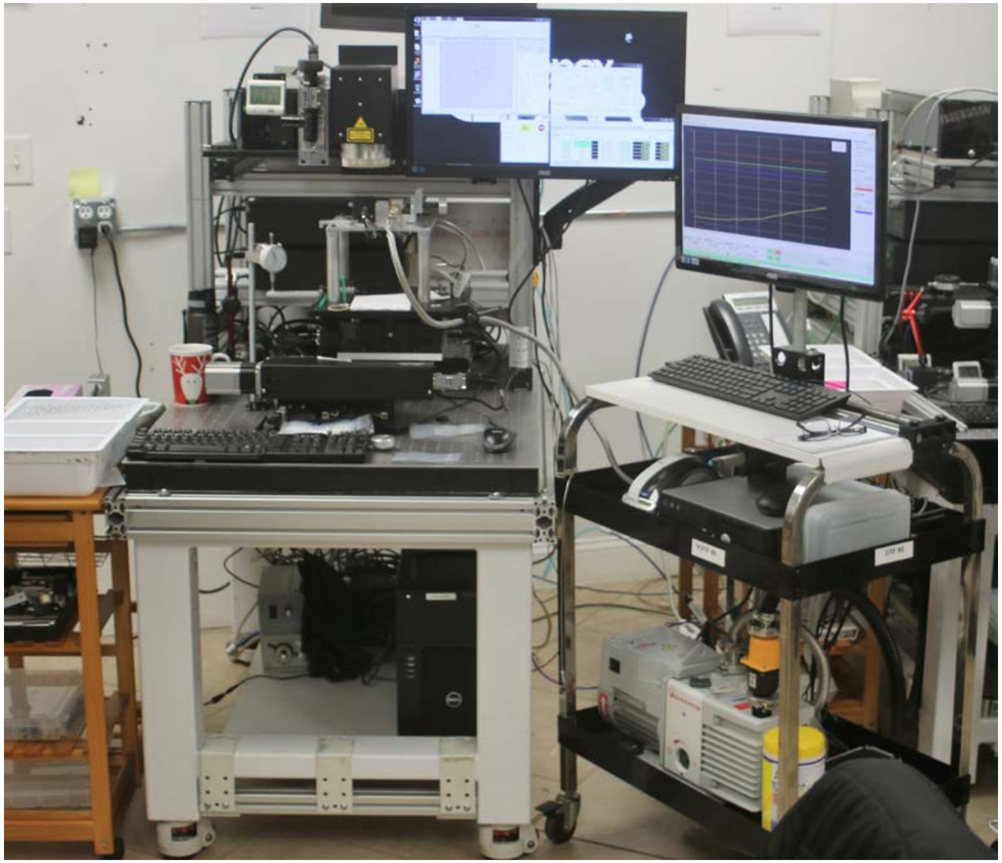


$$Q = \int u\,dA = \int \frac{-(R^2 - r^2)}{4\mu}\frac{dP}{dx} 2\pi\, R dR$$

$$Q = \frac{\pi r^4}{8\mu}\frac{(P_1 - P_2)}{L} = \frac{\pi r^4}{8\mu}\frac{\Delta P}{L}$$

Hagen-Poiseuille (H-P) Equation

Container Laser Drilling Station



Selecting the correct methods for shipping and handling

- Due to a small diameter of the orifice a specific methods preventing the drilled channel from being clogged are applied.
- During processing it may mean a presents of the assist gas or clean air and some method assuring the transport of the laser created material particles away from a drilled sample.
- The samples have individual reports indicating the effective flow diameter, or optical diameter, flow rate and test conditions. So correct identification of each sample container becomes very important. No marking on a sample itself is usually allowed.
- In case the containers are filled, Lenox Laser with a customer develops a specific method of packing to preserve the content from contamination and the drilled channel from clogging.