Producing a Calibrated Leak Standard With Laser Technology

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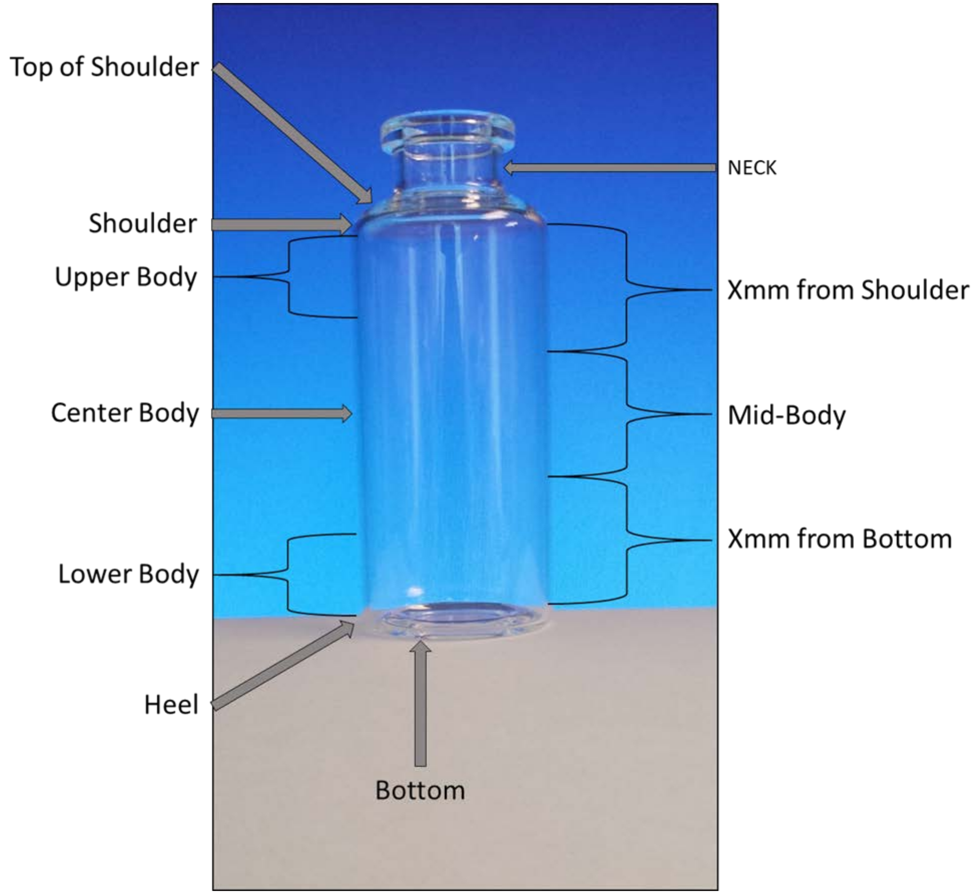
Three parts of integrity testing by calibrated orifice

- Manufacturing the leak
- Calibrating the leak
- Cross calibrating the leak test methods

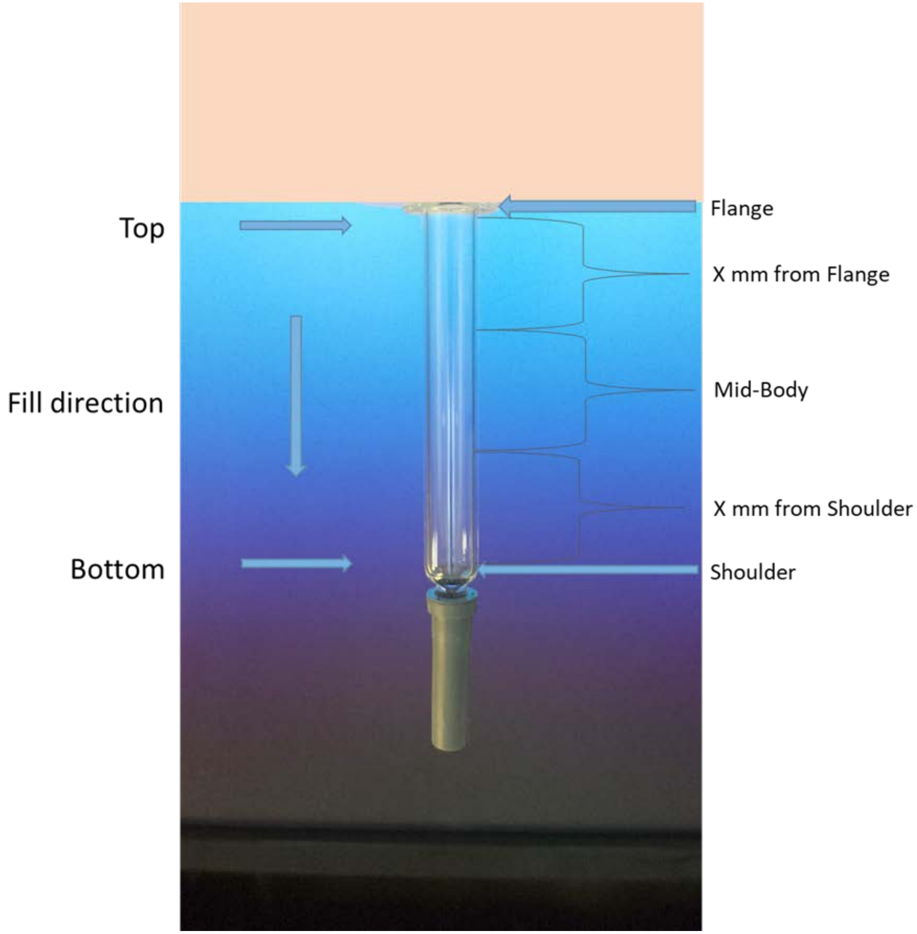
Open Containers

Sealed and filled containers

Common Hole Locations in Glass Vials



Common Hole Locations in Syringes



Glossary

- Calibrating the leak – measuring the flow and flow effective diameter
- Flow effective diameter – the diameter of the round orifice with the area equivalent to a leak
- Positive, negative, volumetric flow meters
- Aspect ratio – length of the channel divided by its diameter
- Laser parameters: wave length, energy per pulse, repetition rate, pulse width
- SEM (Electron Scanning Microscope)

Two major parts of manufacturing the leak

- Laser drill the orifice with post measurement
(non destructive and destructive)

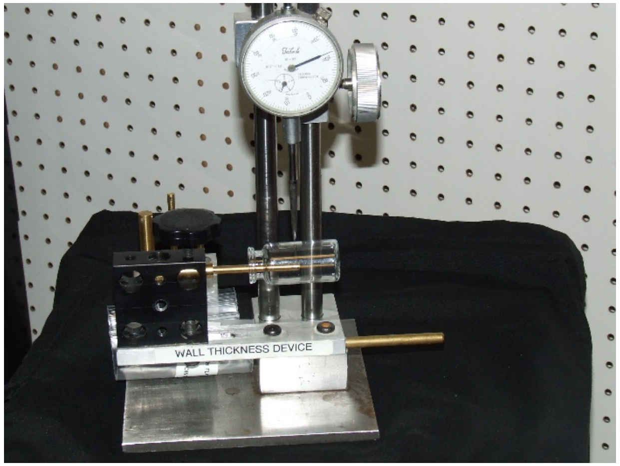
- Laser drill the orifice with in-process test

Prequalification of containers: syringes, ampoules and vials for laser drilling

- Measurement of the wall thickness deviation
- Selecting the methods of preprocessing (mechanical, laser beam)
- Optimizing the laser parameters for material composition
- Selecting the metrology methods for different contents of the containers (developing the repeatability, nondestructive and destructive methods)
- Selecting the correct methods and ways of handling: mechanical fixtures for drilling, marking, testing, storing, shipping

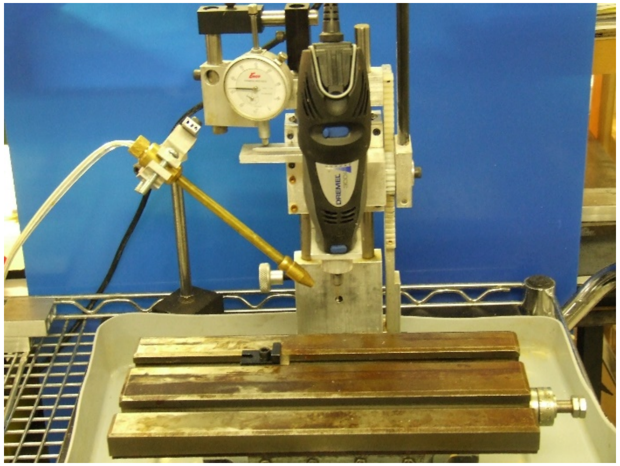
Measurement of the wall thickness deviation (Fig.1)

- The thickness of the material (glass, plastic) varies in different parts of the given container such as ampoule, syringe and bottle.
- Although this thickness is defined on the drawings of the manufacture of the given containers, it may vary by 50%.
- The diameter of the microhole is effected by this thickness variation.
- Therefore Lenox Laser performs its own study of the thickness in the areas selected for a drilling by customer. Sometimes these methods are destructive.

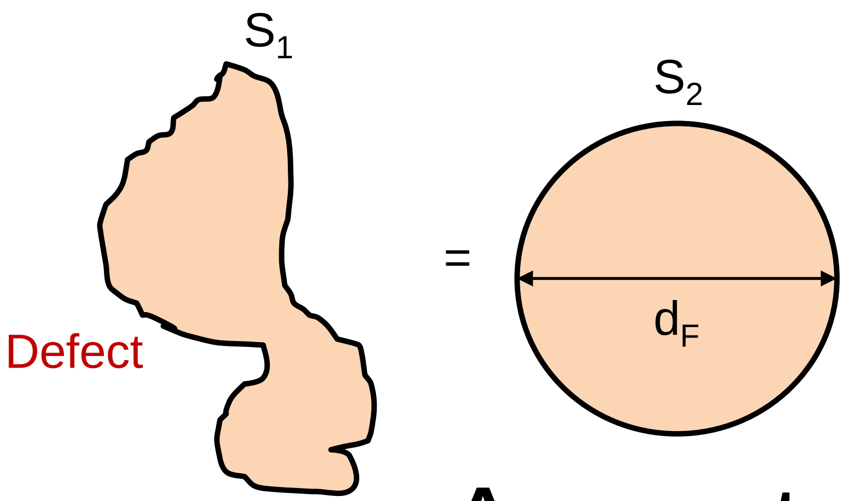


Selecting the methods of preprocessing

- Due to high precision and aspect ratios (ratio of a wall thickness to a orifice diameter) application of more then one laser beam is required. This step may include also some mechanical drilling before the application of the final laser beam for the calibrated orifice is done.

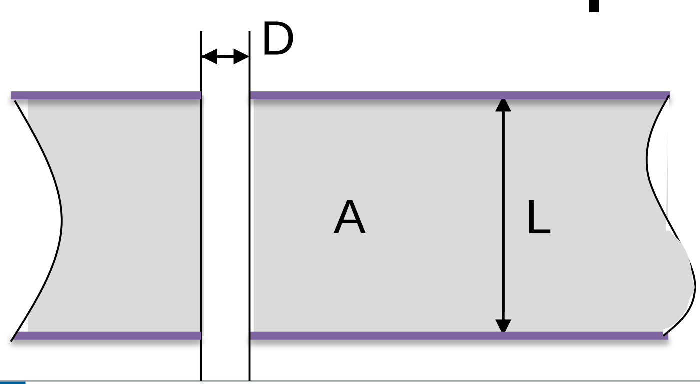


Flow Effective Diameter



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

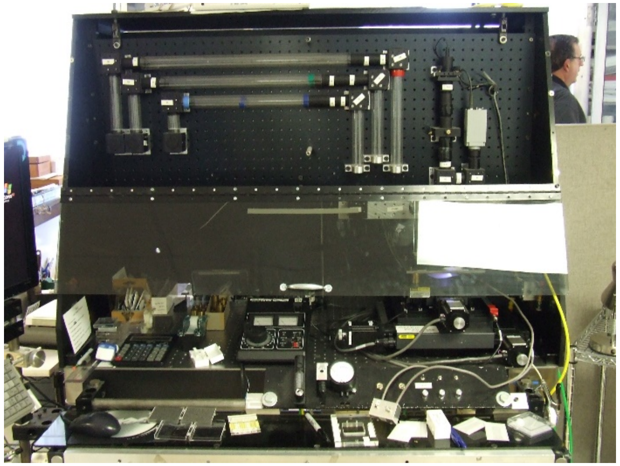
Aspect Ratio



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

Optimizing the laser parameters for material composition

- Different types of glass or plastic have different thermal, mechanicals and optical properties. Some of those types are very sensitive to changes of the laser parameters such as wave length, energy pre pulse, repetition rate, pulse width etc.
- Choice of the correct combination of the laser parameters may lead to the absence of the micro cracking, melting, carbonization, separation of the composite materials, presents of micro particles in a channel.



Selecting the metrology methods for different contents of the containers

- The containers supplied for the orifice manufacturing may be empty or filled with liquid. The containers also may be open to the ambient atmosphere or sealed, having the internal pressure above or lower then ambient.
- In each particular case one or more hole calibration methods may be used.

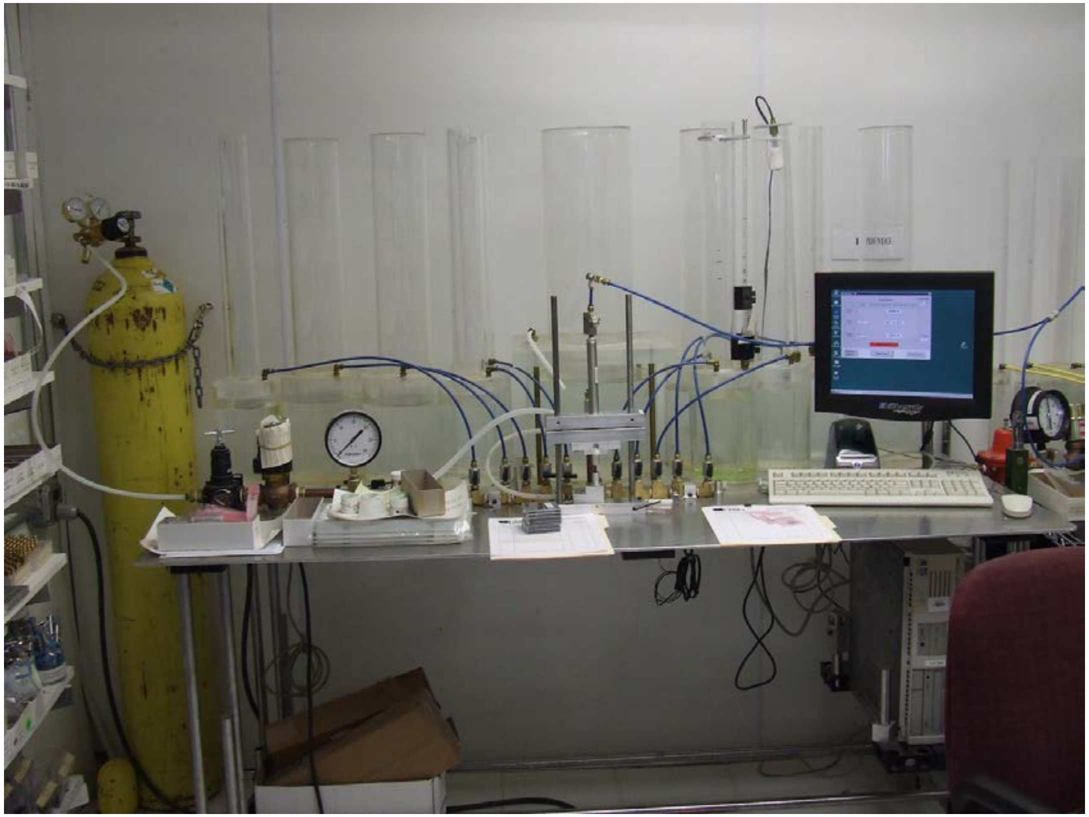
- The methods include:
- 1. Mass flow, volumetric methods (volume vrc. time).
- Standard: leak standard.
- 2. Optical microscope method (geometrical size and shape).
- Standard: optical standard.
- 3. SEM method (geometrical size and shape on nano scale). Standard: nano sphere.
- 4. Bubble detection. Qualitative method.

Measurement with OGP

Measurement by SEM

Mass flow methods

Flow measuring method by water displacement



Flow measurement by vacuum gages

Selecting the correct methods and ways of 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 contentce from contamination and the drilled channel from clogging.

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: $m = \rho Q$ (or $m = \rho a Q a$)
  - –The fluid density is determined at the actual temperature and pressure conditions: $\rho a = \rho(PaTa)$

Standard vs Actual Volumetric Flow (cont.)

- Standard volumetric flow: Qstd = m/ρstd
  - The volumetric flow that would exist at the standard pressure (Pstd) and temperature (Tstd) for the same mass flow
  - The fluid density is determined at the standard temperature and pressure conditions: ρstd = ρ(Pstd Tstd)

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.)

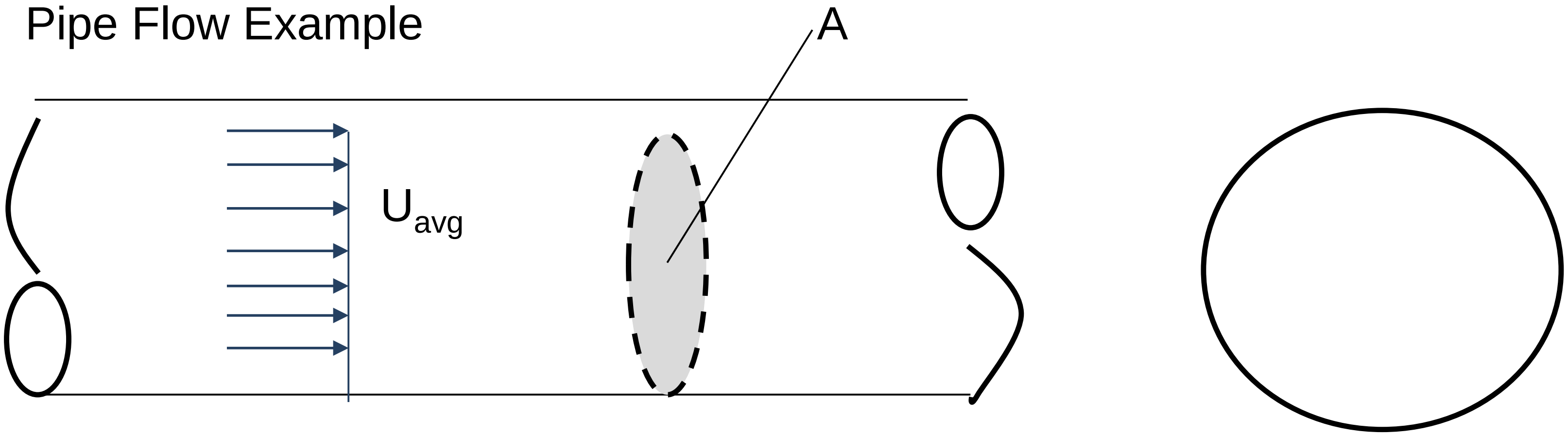
- To distinguish between Qa and Qstd the following notation is sometimes used
  - Actual volumetric flow: an “a” is sometimes placed before units
    Example: acfh, accm
  - Standard volumetric flow: an “s” is sometimes placed before units
    Example: scfh, sccm

Volumetric Flow (Simplified)

$$Q = U_{avg} A$$

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

Pipe Flow Example



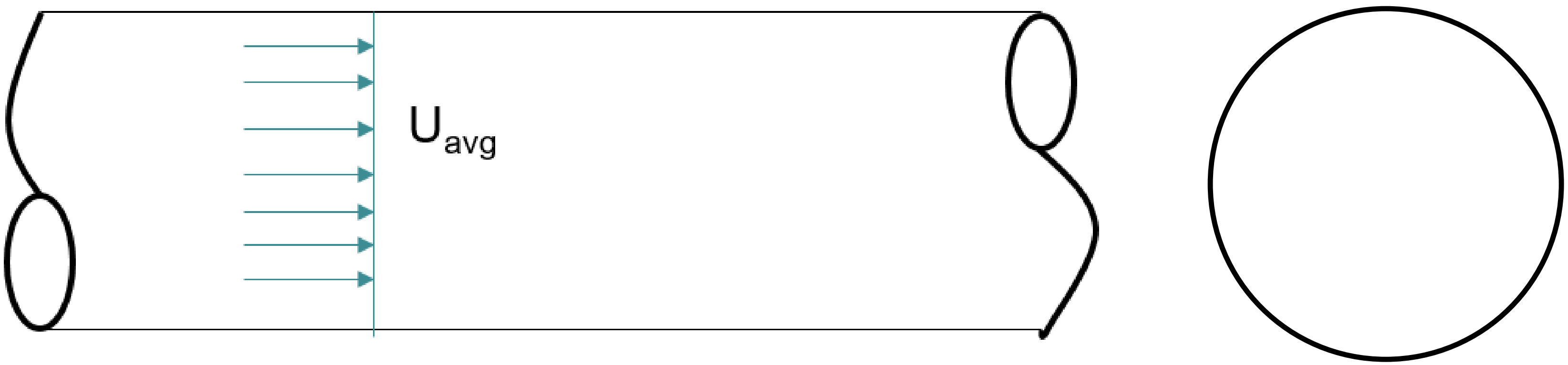
SI unit: m^3/s, m^3/min, m^3/hr
Other units: Lpm (liters/min), gal/min, cm^3/min (ccm), ft^3/hr (acfh) …

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

Fluid Density

- Most flowmeters measure Q (or Uavg)
- Mass flow is usually the desired measurand
- Density is needed to convert to mass flow
  - Densimeter – Liquid and high pressure 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

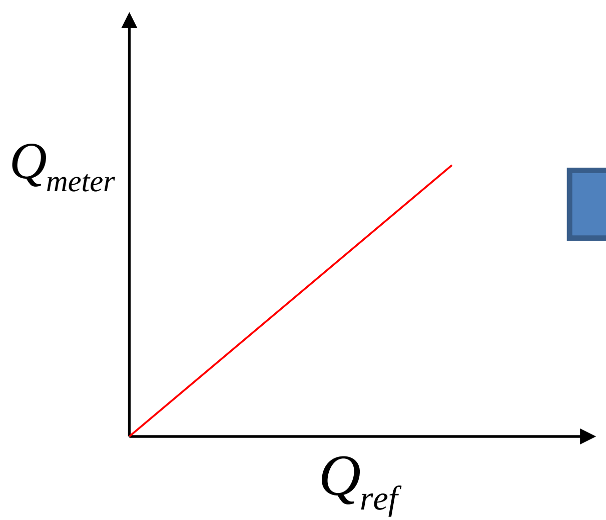
Flow Meter Measurements

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

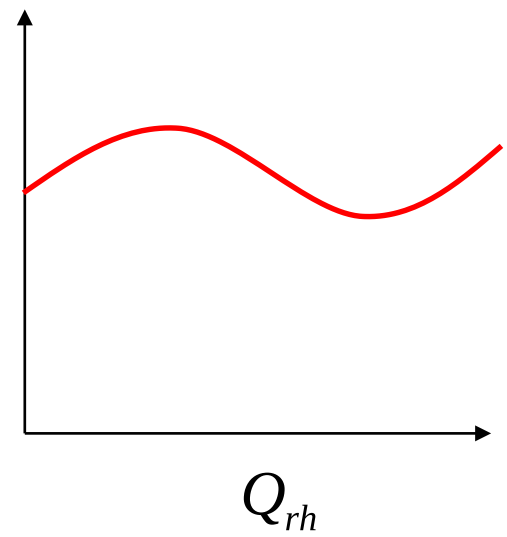
- Mass Flow
- Volumetric Flow
- Velocity (cross sectional area must be measured)

Reynolds Number and Discharge Coefficient

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

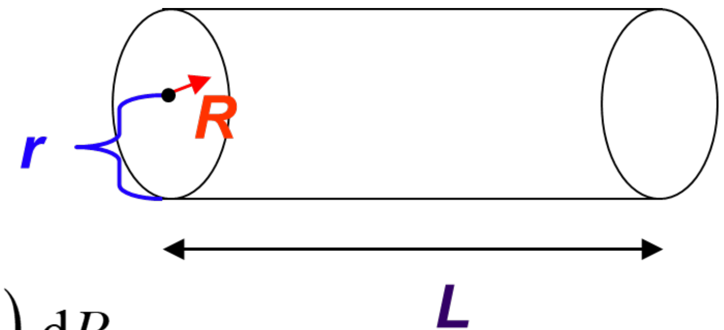


$$C_d = \frac{Q_{ref}}{Q_{rh}}$$



Laminar Flow Meter: Physical Model

$$u(R) = \frac{-(R^2 - r^2)}{4\mu}\frac{\mathrm{d}P}{\mathrm{d}x}$$



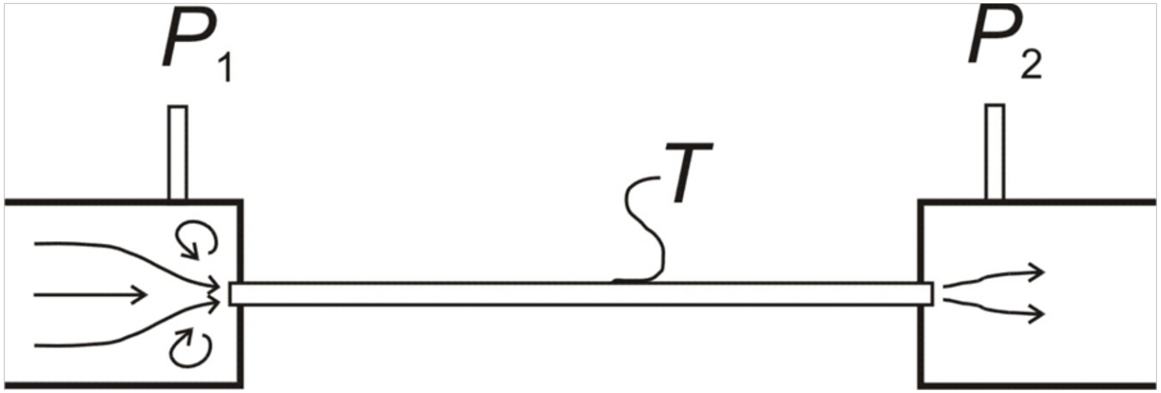
$$Q = \int u\mathrm{d}A = \int \frac{-(R^2 - r^2)}{4\mu}\frac{\mathrm{d}P}{\mathrm{d}x} 2\pi \; R\mathrm{d}R$$

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

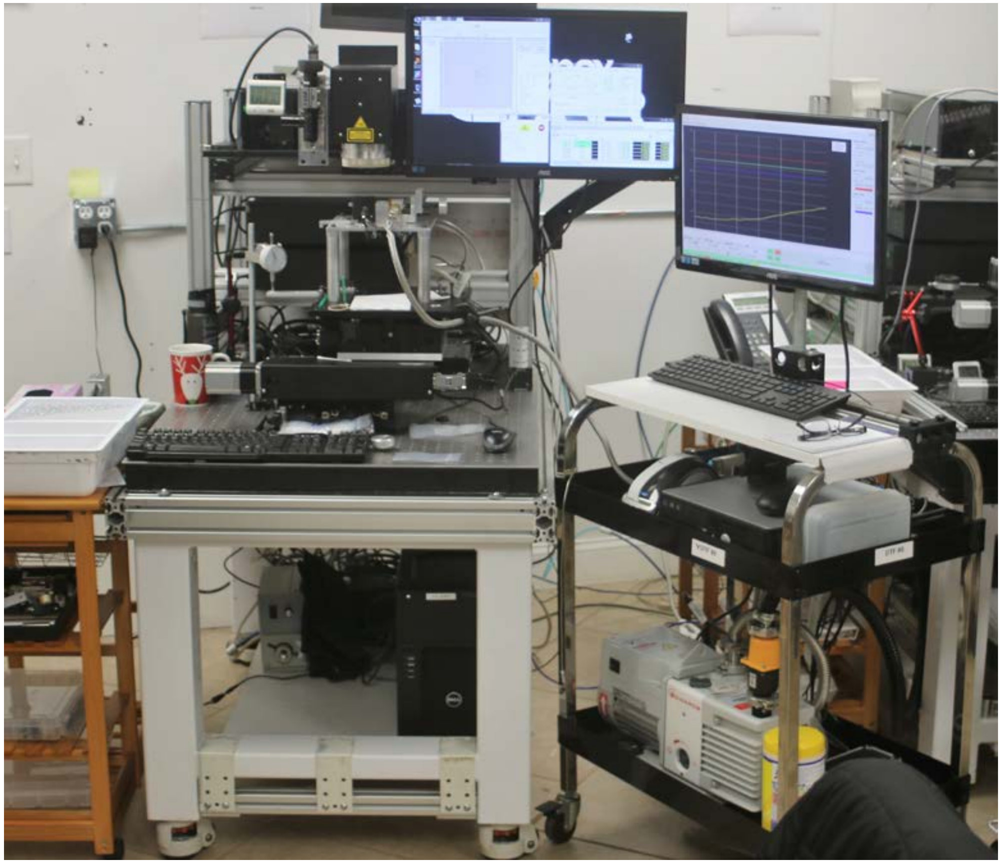
Hagen-Poiseuille (H-P) Equation

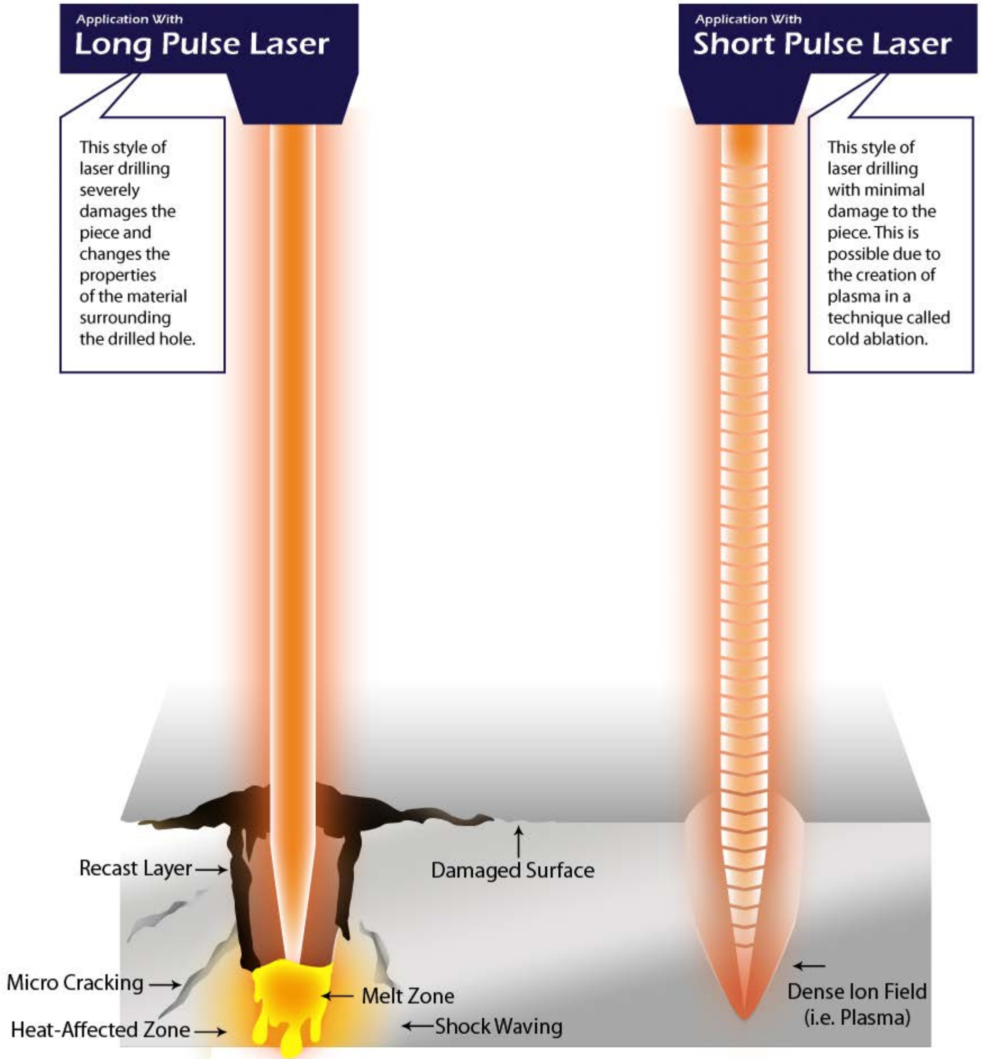
Entrance and Exit Corrections

- Before flowing into the tube, the flow must converge and accelerate. Flow exiting the tube creates a jet into the exit chamber. Both effects contribute to the measured pressure drop in a manner not included in the Hagen-Poiseuille equation.



Container Laser Drilling Station




Application With
Long Pulse Laser
This style of laser drilling severely damages the piece and changes the properties of the material surrounding the drilled hole.
Application With
Short Pulse Laser
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.
Recast Layer
Damaged Surface
Micro Cracking
Melt Zone
Heat-Affected Zone
Shock Waving
Dense Ion Field
(i.e. Plasma)

Controlled Ablation by Laser

- Definition:
  - Process of removing material from a solid surface by irradiating it with a very short, high energy laser pulse. During this process the material is converted to a plasma from a solid state directly. The absorbed laser energy is not converted to a heat. Laser pulses have to be of the order of hundred(s) of femtoseconds (fs) 10-16 [s] to a few picoseconds (ps) 10-12 [s] long.

Goals and Vision

- Zero defects practice (aerospace philosophy)
- Use of the simplest possible methods and “transparent” physics for manufacturing.
- Use “optical signature” methods such as photography and videography on macro and micro scale for quality control and manufacturing.
- Develop new “Object oriented” methods and techniques for metrology