

# A leak or not a leak?

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In 1810 package integrity becomes an issue



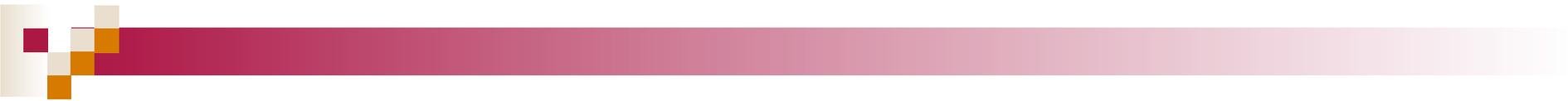
For me it all started with canned peppers.



## A leak or not a leak?

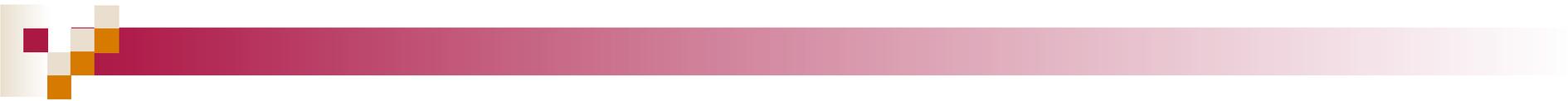
- Package Integrity Conference, Slough England
- If cooling water was inoculated, then 3% of metal cans would be spoiled.





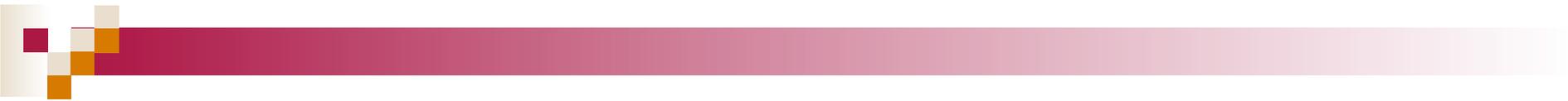
# Closure integrity becomes an important issue to new packaging





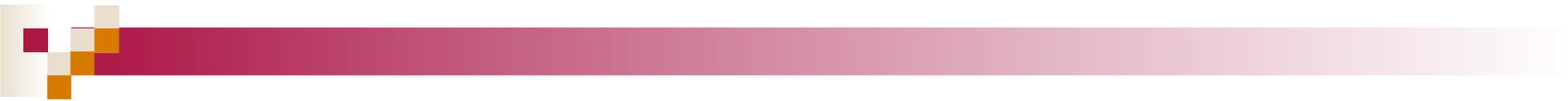
## What is a Leak?

- **ASTM Definition** - “a hole or void in the wall of an enclosure, capable of passing liquid or gas from one side of the wall to the other under action of pressure or concentration differential existing across the wall, independent of the quantity of fluid flowing.”



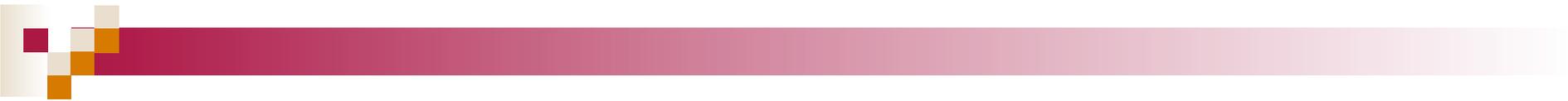
## Leak-Free Package

- Leakage is a continuous variable.
- Leak-free means that the leak rate is so small that it has no practical significance.
- Must establish leak rate specifications.



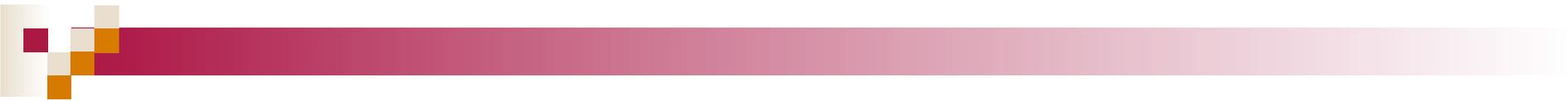
## Leakage Definitions

- **Microhole** - a hole with diameter that falls below the threshold of human visual inspection.
- **Microleak** - a leak occurring through a microhole.



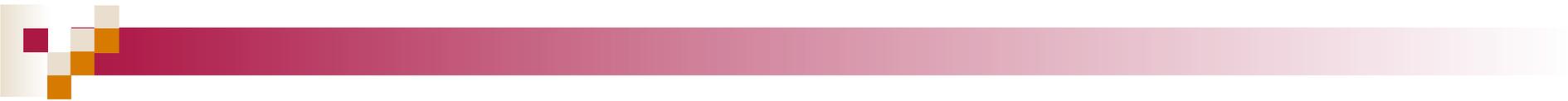
## Leakage Definitions

- **Threshold Leak Size** - The lowest size microhole at the Threshold Leak Pressure that allows initiation of liquid flow into the defect.
- **Critical Leak Size** - The lowest size microhole that allows microorganisms to traverse the defect and enter the package.



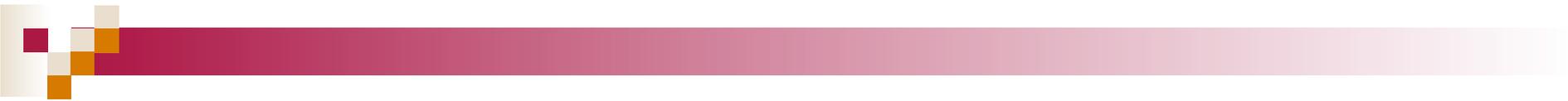
## Previous Leakage Study Problems

- Flexible packaging allows for deformation of manufactured defects.
- Difficulty in manufacturing micron sized defects less than 5  $\mu\text{m}$ .
- Immersion biotest too extreme.



## Previous Leakage Studies

- Bacteria travel through liquid in the defect via pressure.
- Correlated post-process contamination with handling and storage conditions.
- Biotesting is dependent on bacterial concentration and viscosity of medium.
- Long, curved defects lower probability of microbial penetration.



# Initiation of Leakage

## ■ Requirements

□ Imposed pressure must overcome external forces.

■ Surface Tension

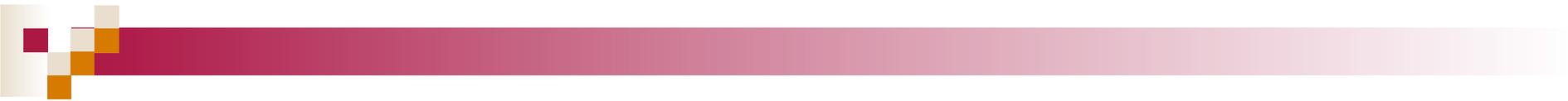
■ Hydraulic Diameter of Defect

■ Atmospheric Pressure

□ Imposed pressure is considered the pressure on the inside of the package.

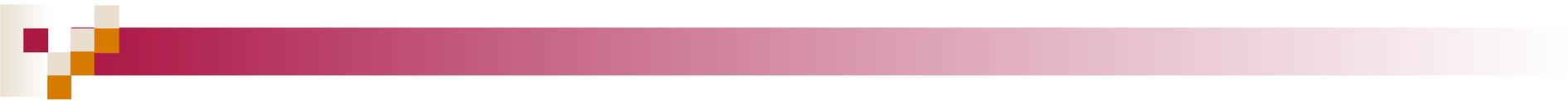
■ Liquid Static Head

■ Pressure Generated Internally



## Leakage-Mathematically

- Rate at which a volume of fluid flows into or out of the leak under specific conditions of temperature and pressure.
- Common units used to express leakage:
  - std cm<sup>3</sup>/s (std means at 20°C & 1 atm)
  - Pa·m<sup>3</sup>/s-- typically for gas leakage rates

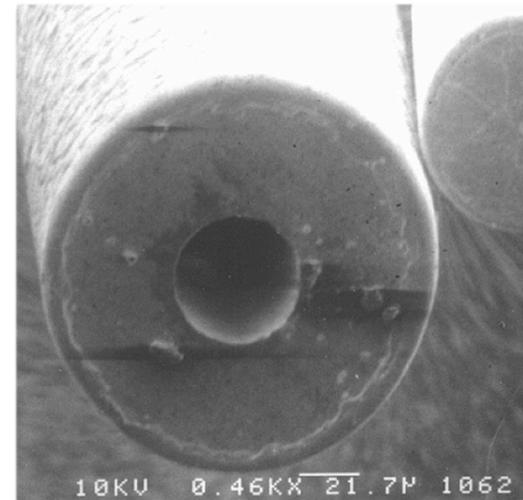
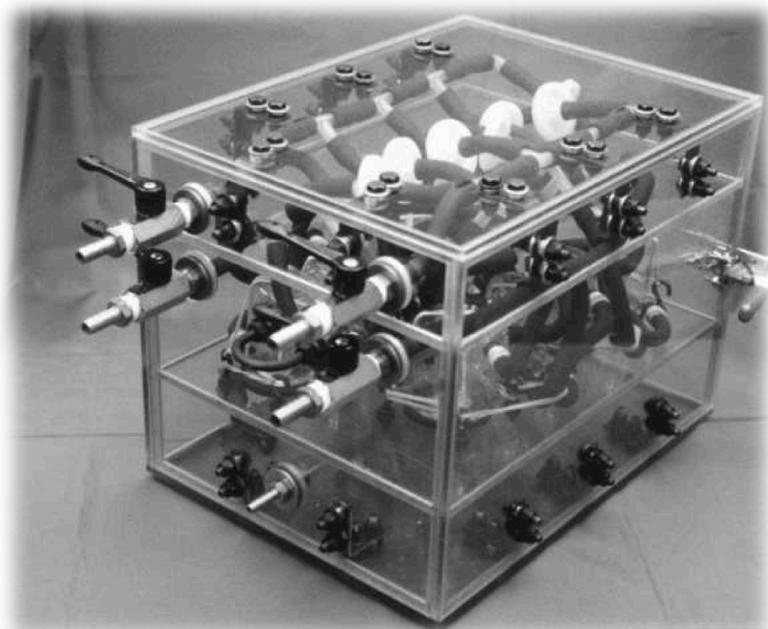


## Immersion versus bioaerosol testing

- Produced pouches with 20  $\mu\text{m}$  channel leaks
- Ingress significant for motile organisms @ 6 log CFU/ml for both methods (44% were positive)
- Testing at 2 log CFU/ml was not positive
- Bioaerosol method provides a means of package integrity testing that more closely approximates non-retorted package environments

# Bioaerosol challenge.

Unique opportunity to control pressure,  
diameter and length of defect.



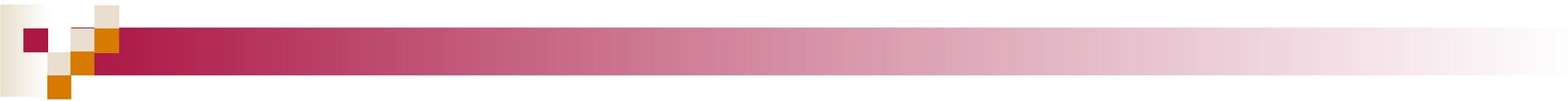
# Bioaerosol Exposure Chamber

- Developed by Keller, et. al, 1998



TABLE 1. *Microbial ingress into test cells as a result of bioaerosol exposure and imposed pressures at 25 °C.*

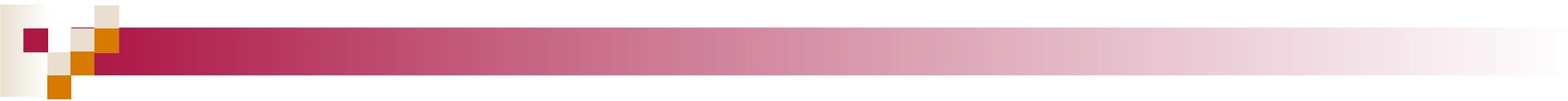
Microtube ID Size ( $\mu\text{m}$ )	Imposed Pressure (kPa)							Total Positives
	-20.7	-13.8	-6.9	0	6.9	13.8	20.7	
50	4/9	8/9	1/9	2/9	3/9	1/9	3/9	22/63
20	6/9	4/9	0/9	0/9	1/9	6/9	6/9	23/63
10	1/9	4/9	0/9	0/9	0/9	3/9	3/9	11/63
7	0/9	1/9	0/9	0/9	0/9	3/9	1/9	5/63
5	1/9	2/9	0/9	0/9	0/9	3/9	1/9	7/63
2	0/9	0/9	0/9	0/9	0/9	0/9	0/9	0/63
Total Positives	12/54	19/54	1/54	2/54	4/54	16/54	14/54	68/378



# Initiation of Leakage

## ■ Requirements

- Imposed pressure must overcome external forces.
  - Surface Tension
  - Hydraulic Diameter of Defect
  - Atmospheric Pressure
- Imposed pressure is considered the pressure on the inside of the package.
  - Liquid Static Head
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## Materials and Methods

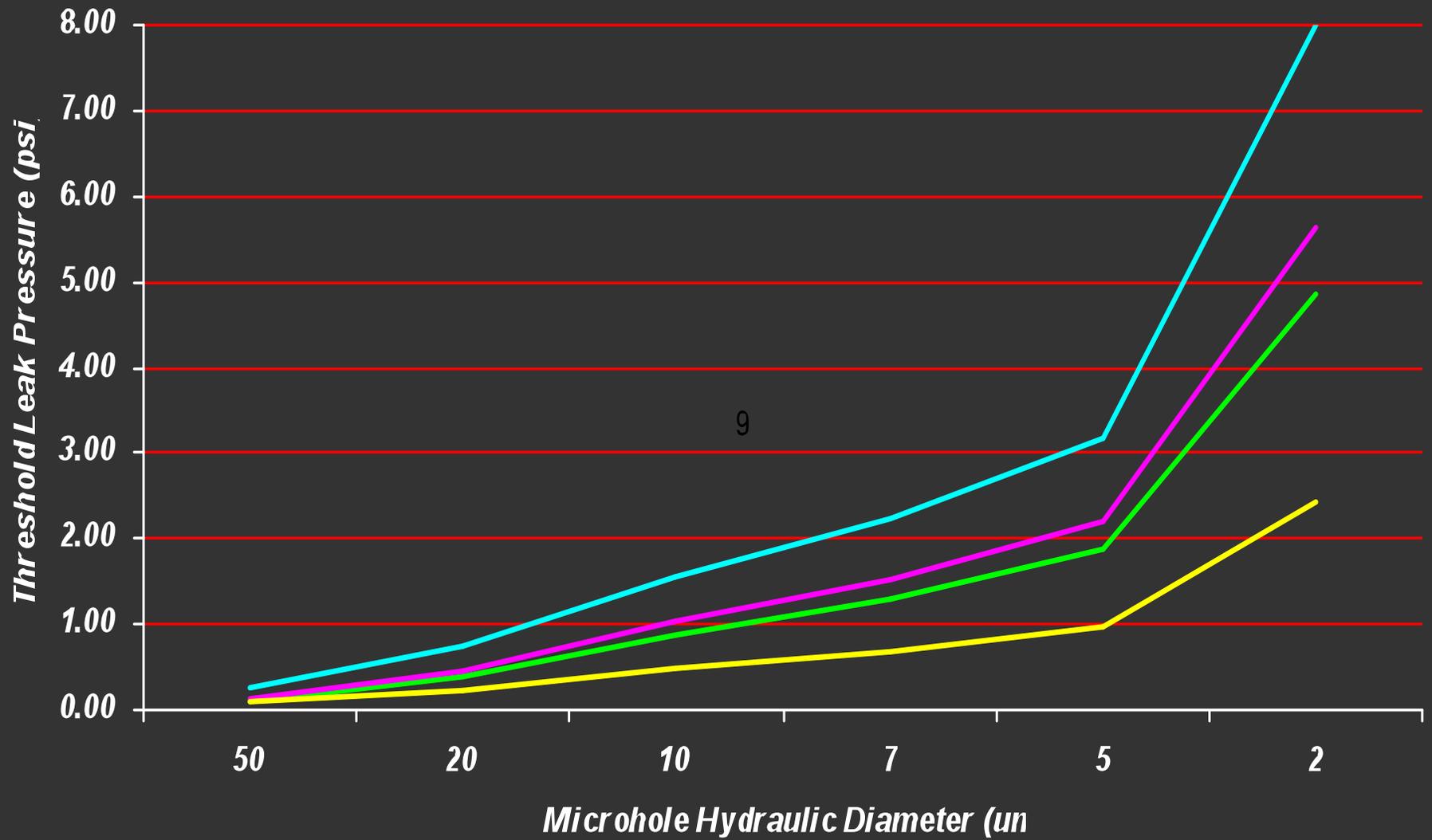
- Objective 1- Measured the:
  - density
  - viscosity (Brookfield rotational viscometer)
  - surface tension (du Nouy ring method with KSV Instruments Sigma 70 automated surface tension reader)

# Products Used

- Liquid products that exhibit a wide range of surface tension and viscosity values.



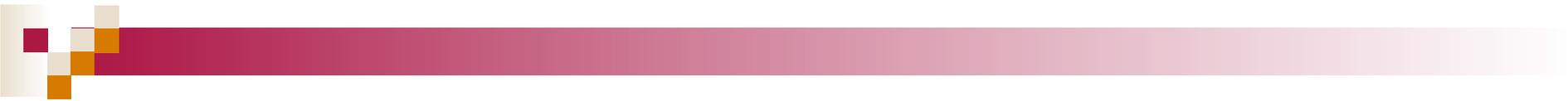
# Threshold Leak Pressures vs. Microhole Hydraulic Diameter



— milk, 2% — wine — safranin red — DI water

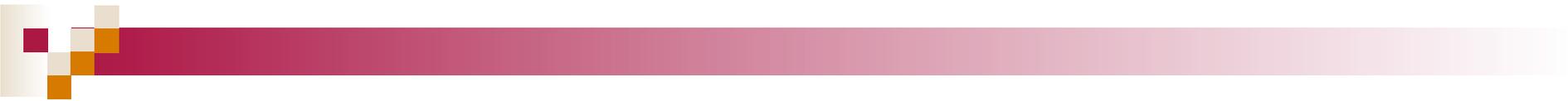
- Critical leak size of liquid filled defects correspond to the threshold leak size.

Hydraulic Diameter ( $\mu\text{m}$ )	IMPOSED PRESSURES						
	20.7 KPa	13.8 KPa	6.9 KPa	0 KPa	-6.9 KPa	-13.8 KPa	-20.7 KPa
0	-	-	-	-	-	-	-
2	-	-	-	-	-	-	-
5	X	X	-	-	-	X	X
7	X	X	-	-	-	X	-
10	X	X	-	-	-	X	X
20	X	X	X	-	-	X	X
50	X	X	X	X	X	X	X



## Leak Rates

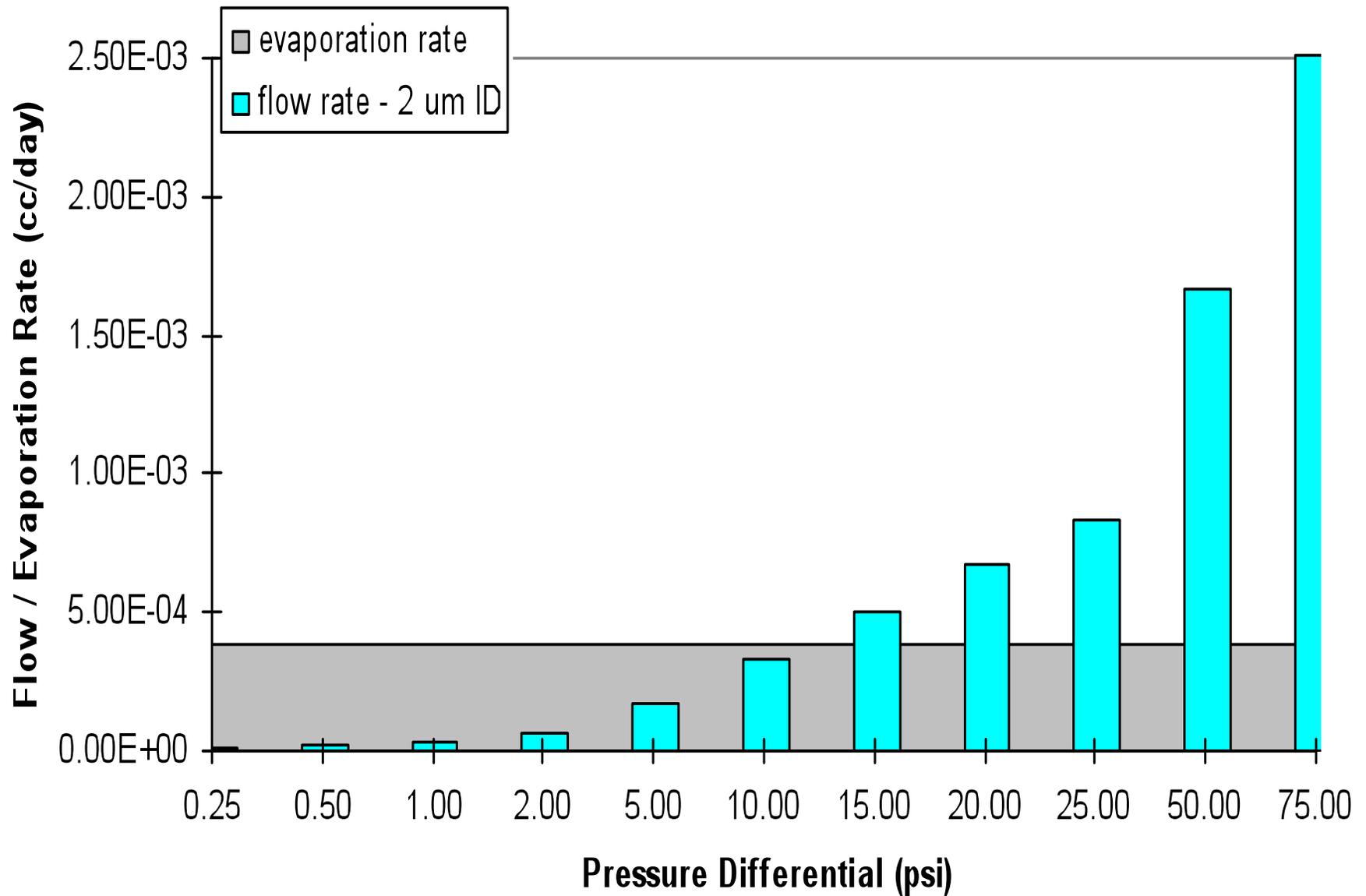
- If a leak is initiated, then will it flow fast enough through the defect to make contact with the outside of the package?
- Leak rates calculated with Hagen-Poiseuille volumetric flow rate equation



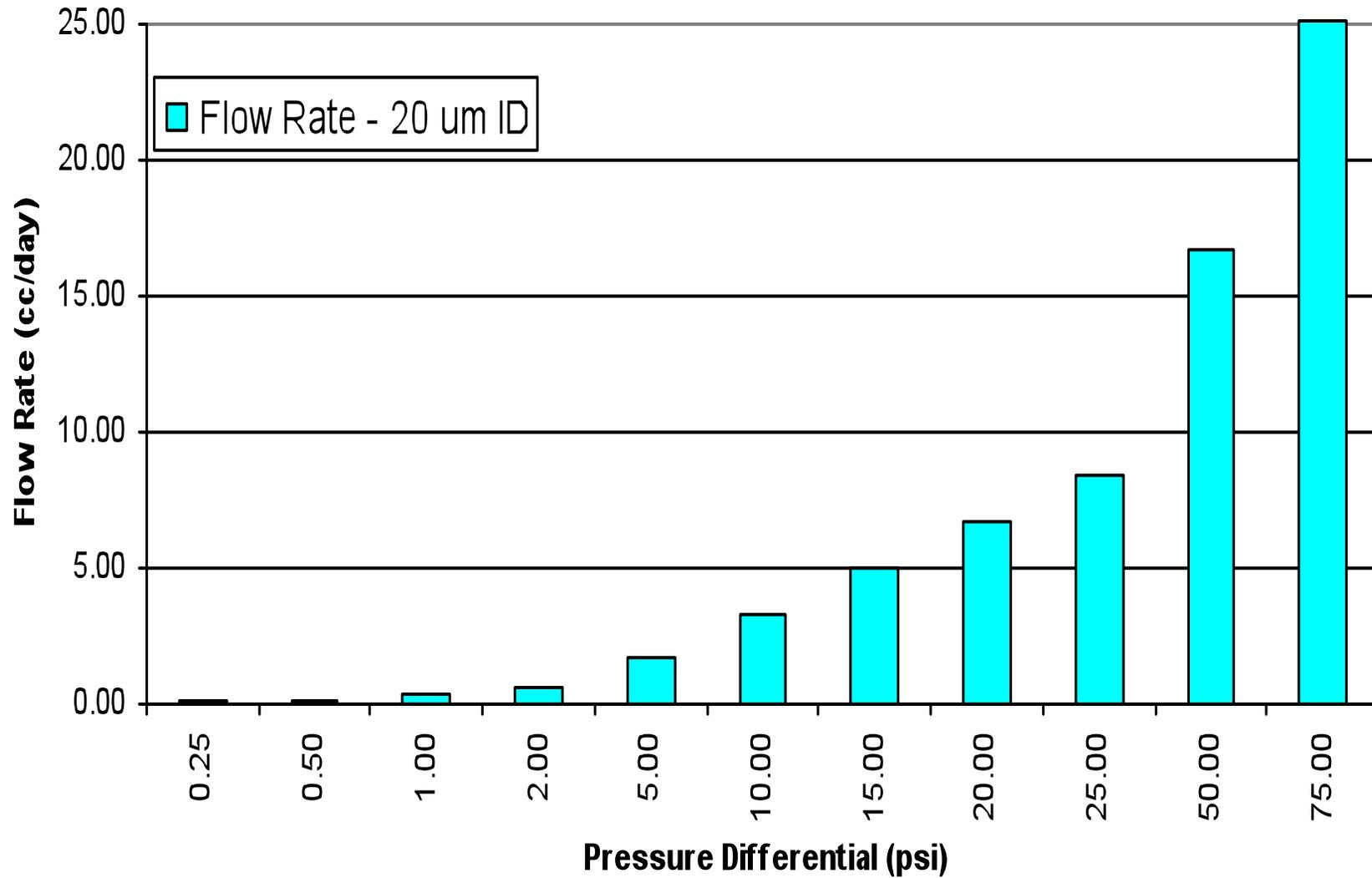
# Evaporation Rate

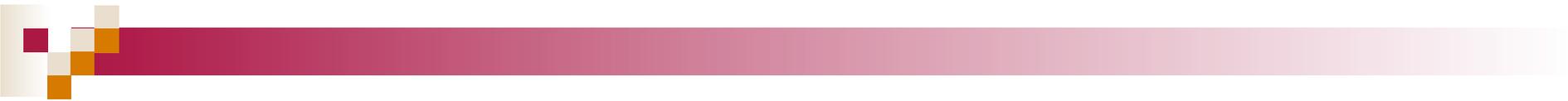
- If the leak flows fast enough to get through the defect, does it flow faster than the evaporation rate?
- Solids may reseal the package

# Flow and Evaporation Rate of Water at 2 $\mu\text{m}$ ID Microhole



# Flow Rate of Water at 20 $\mu$ m ID





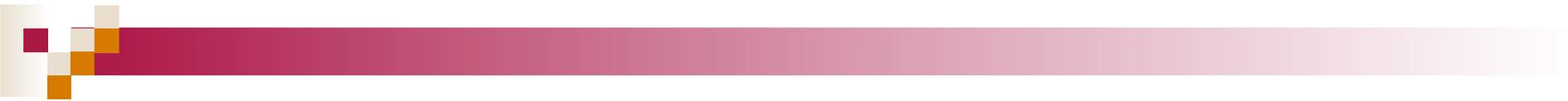
## Air-filled defects

- Air-filled defect with vacuum draws bioaerosol particle to microtube opening
  - Smaller microtubes exhibit leak initiation scenario, the bioaerosol droplet size (2.68  $\mu\text{m}$ ) covers the opening and the vacuum must overcome the threshold leak pressure
  - Larger microtubes may not require a leak initiation due to difference in size of bioaerosol

# Critical Leak Size of Air-Filled Micro-Defects

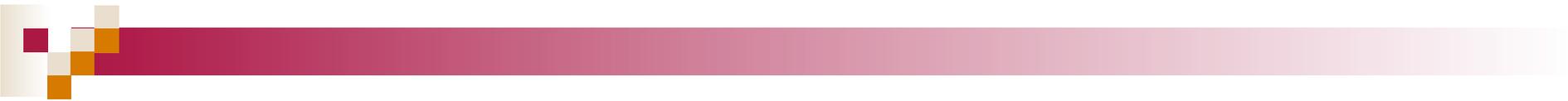
- Between 5 and 7  $\mu\text{m}$  regardless of pressure differential

Hydraulic Diameter ( $\mu\text{m}$ )	IMPOSED PRESSURES			
	0 KPa	-6.9 KPa	-13.8 KPa	-34.5 KPa
0	-	-	-	-
2	-	-	-	-
5	-	-	-	-
7	X	-	X	X
10	X	X	X	X
20	X	-	X	X
50	X	X	X	X



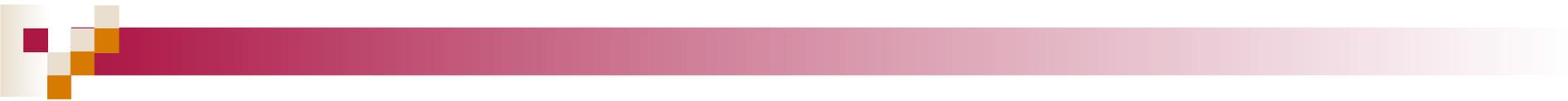
## Leakage- Is a three step problem

- Flow Initiation
  - Surface tension, radius, pressure differential
- Flow
  - According to Hagen Poiseulle
- Flow rate must be greater than evaporation rate or no flow.



## What did we learn?

- In 1988, Virginia Chamberlain, then a sterility expert in the compliance division of FDA's Center for Devices and Radiological Health, said, "If something fails a certain physical test, what does that mean in terms of compromising the product's sterility?"



## What did we learn?

- Length of defect was not significant
- A three different shapes of microorganism were tested and shape was not significant
- Surface tension and diameter play an important role in leak initiation
- Leak sizes critical to sterility are based on the relationship of surface tension and imposed pressure
- Choose your dye for leak testing carefully.

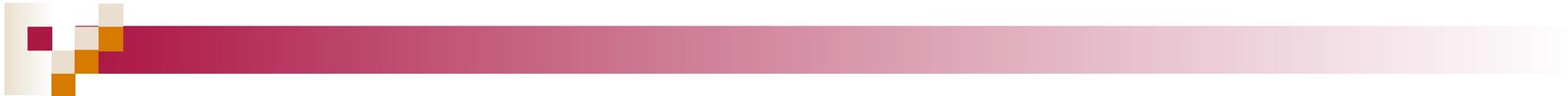
# Closure integrity is still an issue, but....



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351570, ZHEJIANG, CHINA

NINGBO HUI FOODS CO., LTD.





# Thank you

Special thanks to:  
Barbara Blakistone  
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