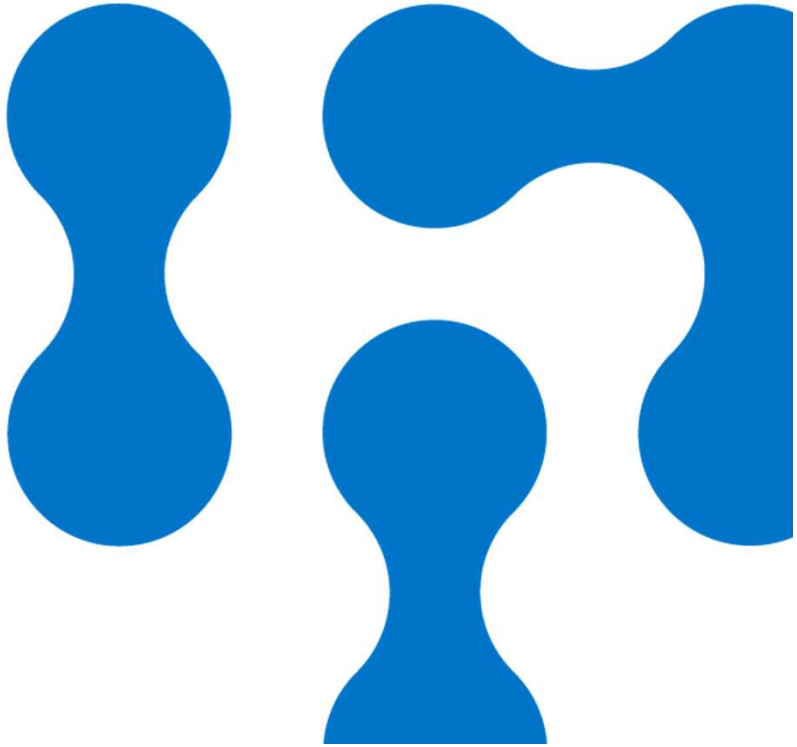
PHYSICAL CHARACTERIZATION

Density
Surface Area
Porosimetry



BIOMATERIALS,
BIOMECHANICS &
TISSUE ENGINEERING

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DENSITY

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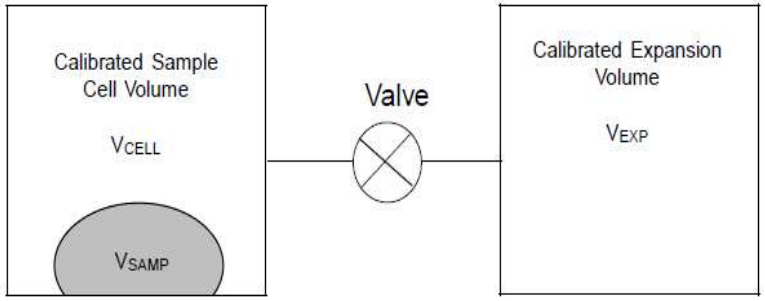

Accupyc 1330

$$P \cdot V = n \cdot R \cdot T$$

$$V_{SAMP} = V_{CELL} - \frac{V_{EXP}}{\frac{P_{1g}}{P_{2g}} - 1}$$

- This equipment consists of two chambers of known volume separated by a valve.
- The sample displaces a certain volume equivalent to the volume of the studied sample.
- Knowing the mass or weight of the sample, its density can be calculated.

$$\rho = \frac{M}{V}$$

DENSITY

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- The range that this equipment can measure is between 1cm^3 and 10cm^3.
- Helium can only penetrate open pores.
- Non destructive technique.
- Time of experiment = 30 - 40min.
- Common errors:
  - DTA_WRN: volume 10% full-scale: the amount of sample placed is too small.
  - ANSERR: overrange /underrange: adjust the pressure of the helium.

SURFACE AREA

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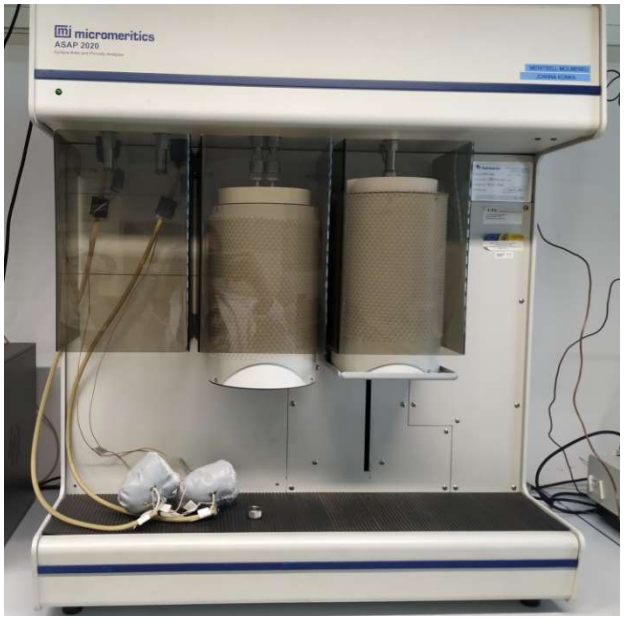
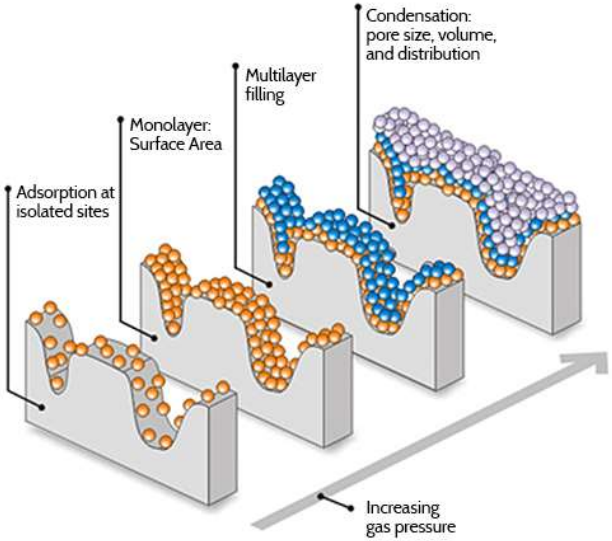
- This technique can measure specific surface area and porosity distribution.
- BET theory (Brunauer-Emmett-Teller): to calculate the specific area. This theory assumes that a gas, such as nitrogen, when is liquefied and absorbed on clean solid surfaces, will fill the entire available surface into multiple layers. At low pressure a monolayer is formed (between 0.05 and 0.3 relative pressure).
- Smaller pores fill at low pressures and larger pores at larger pressures





SURFACE AREA

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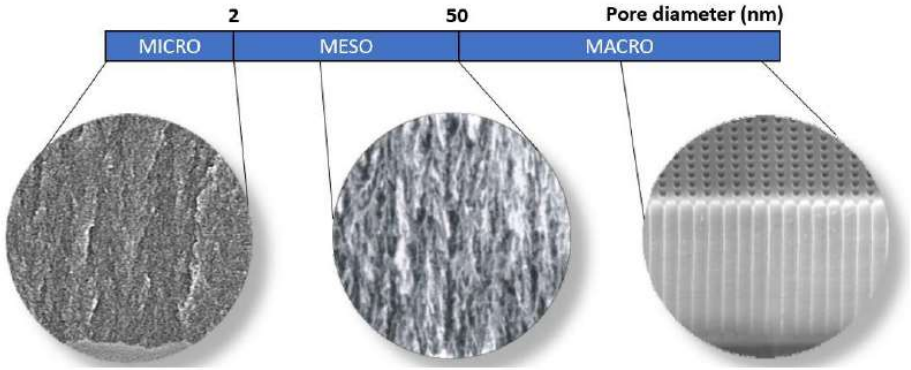
- The solid is degassed, and cooled to -196ºC. Nitrogen gas is introduced and adsorbed.
- Progressive nitrogen P/P_0 ratios are used
- If we measure with nitrogen, the total area must be greater than $1m^2$.
- If we have smaller surface area Krypton can be used.
- The BJH (Barret Joyner Halenda) theory is used to analyze the distribution of mesoporosity
- HK theory is used to analyze the distribution of microporosity.
- At the degassing stage, the sample can be heated to 350°C.
- Non destructive technique
- Time of anaylsis:
  - Sample degasification : 24h
  - Sample analysis:
    - Surface area= 5-6h
    - BJH=24h
    - HK=72h

Clasificación de la porosidad según el tamaño de poros por la IUPAC (Unión Internacional de Química Pura y Aplicada)

- Microporos: tienen un tamaño inferior a 2 nm.
- Mesoporos: tienen un tamaño de poros comprendido entre 2 y 50 nm.
- Macroporos: tienen un tamaño mayor a 50 nm.



SURFACE AREA

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Measuring range: From 0.3nm to 300nm

BET area results
- The correlation coefficient must have at least 4 nines.
- The coefficient C must be positive. Indicates when a monolayer is no longer available.

Sample type:
- Solids: The sample holder has a diameter of 7mm. The bottom sphere of the sample holder must be filled.
- Powder.

How much sample?
- Enter as many samples as you can to get at least $1m^2$ in total.

Error:

Total Surface Area in Sample Tube, m^2								
0.2	0.5	1	2	5	10	20	50	100
190%	76%	38%	19%	7.6%	3.8%	1.9%	0.76%	0.38%

POROSIMETER

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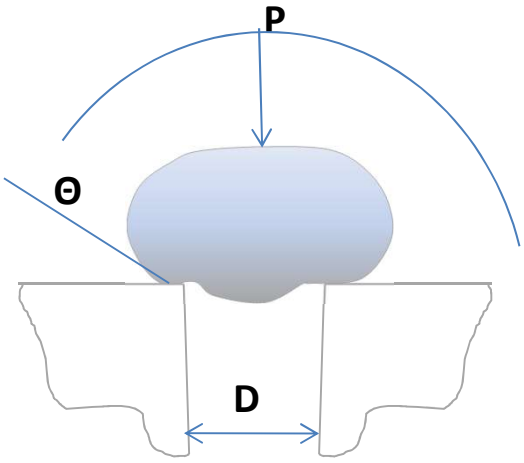
Low pressure port
High pressure port

- Mercury intrusion porosimetry is a technique that, by applying pressure, forces the entry of mercury into the pores of a solid or powder.
- Applied pressure and pore inlet diameter are related to the Wasburn equation.
- Wasburn described the behavior of a non-wettable liquid.
- Mercury is the only liquid that does not wet and therefore cannot enter the pores by capillary action. It must be forced.
- The force done to enter the pores is inversely proportional to the pore inlet size.



$$D = \frac{-4\gamma \cos\theta}{P}$$

ɣ = superficial tension (constant)
Θ= contact angle (constant)

POROSIMETRY

BIOMATERIALS, BIOMECHANICS & TISSUE ENGINEERING

- The equipment used is the AUTOPORE IV 9500 (micromeritics).
- The material must be rigid enough so that it does not bend when the mercury enters.
- Only by open and interconnected pores
- Destructive and toxic method.
- This technique measures the inlet diameter of the pores.
- The sample must be dry.
- An approximation is made: cylindrical pores are assumed.
- Samples can be analyzed in powder and solid format.
- The sample holder has a size of: diameter = 15mm and height = 10mm.
- How much sample? Enter as much sample as possible so that the percentage of mercury entering the sample is between 25% and 90% of the volumen of the stem.

POROSIMETRY

BIOMATERIALS, BIOMECHANICS & TISSUE ENGINEERING

- Measuring range: From 0.006um to 360um

- Pressure range:

Low pressure: up to 30psi - 14um-360um

High pressure: up to 30000psi - 6nm-14um

- Time analysis:
  - Low pressure = 1h
  - High pressure = 2h

- The sample must be dry, non compressible.

THANK YOU FOR YOUR ATTENTION

https://biomaterials.upc.edu/ca

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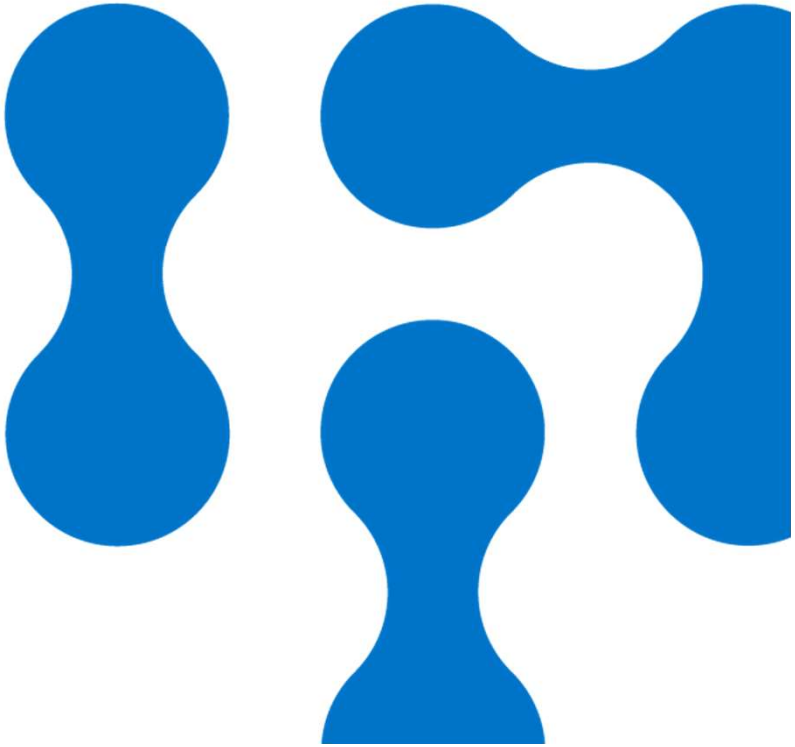
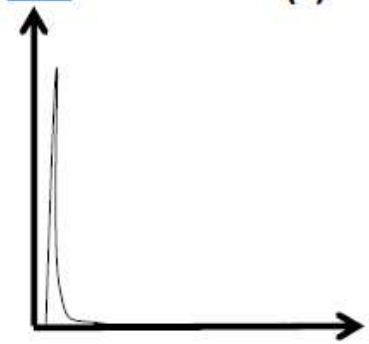


Cómo representar la distribución de tamaño?

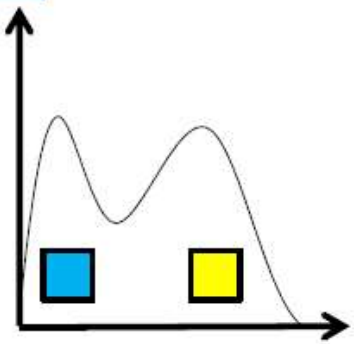
1 **dV/dr = f(r)**



r (linear scale)

El uso de una escala lineal no es adecuado ya que una PSD generalmente cubre **varios órdenes de magnitud**

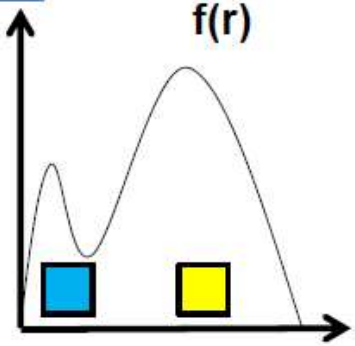
2 **dV/dr = f(r)**



r (log10 scale)

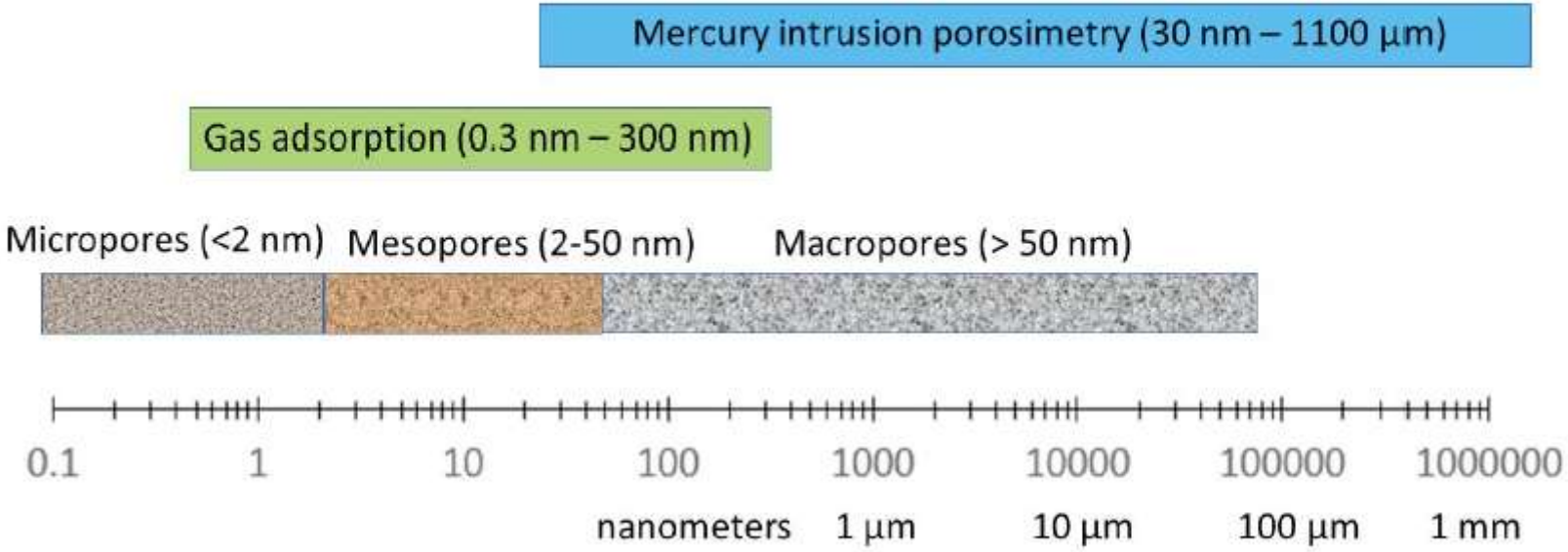
Una escala logarítmica para r permite una mejor visualización de la PSD. Sin embargo, los dos cuadrados **no representan el mismo volumen** y pueden dar lugar a una **mala interpretación** de los resultados.

3 **dV/dlog(r) = f(r)**



r (log10 scale)

Representar **dV / dlog (r)** como el eje de ordenadas asegura que los cuadrados representen el **mismo volumen**, y así permitir una **interpretación correcta de la PSD**.


Mercury intrusion porosimetry (30 nm – 1100 µm)
Gas adsorption (0.3 nm – 300 nm)
Micropores (<2 nm)
Mesopores (2-50 nm)
Macropores (> 50 nm)
0.1
1
10
100
1000
10000
100000
1000000
nanometers
1 µm
10 µm
100 µm
1 mm