Vacuum and Pressure decay leak testing

Basel, 27. February 2020

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Overview

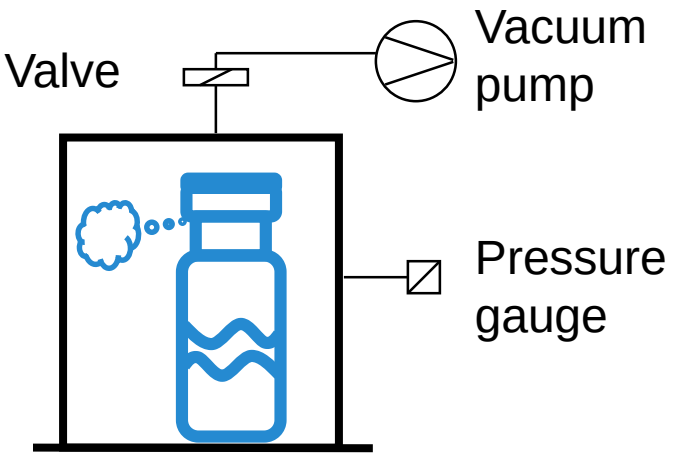
- Fundamentals of DP method
  - LFC, P, V (different types)
  - pressure curve / course
  - steps of the differential pressure test
- Method selection
- Equipment in process control
- Applications of differential pressure
  - inline vs. lab scale systems
  - inline systems

Fundamentals of DP

LFC, P, V

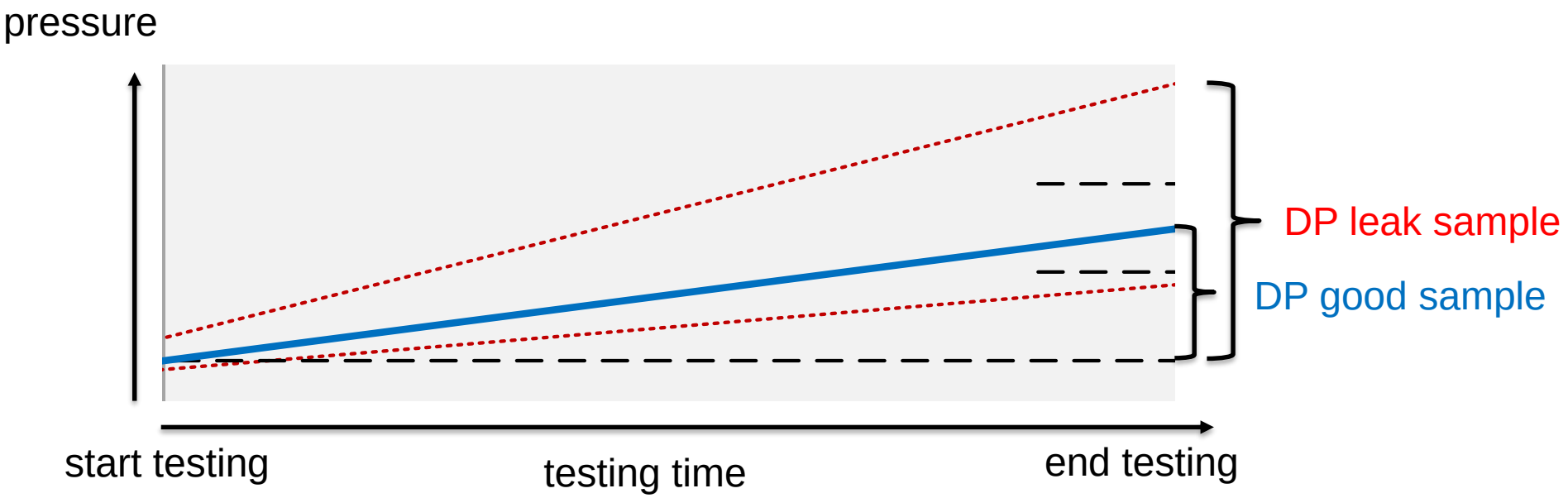
Working principle – general

1) Place sample in a hermetically sealed test chamber
2) Apply either under- or overpressure to the test chamber
3) Lock vacuum / pressure supply from the inside of the chamber
4) Monitor the pressure change in the chamber by highly sensitive sensors
5) If a mass transfer is possible between the inside of the sample and the volume in the chamber (either way) the change of the pressure in the chamber will be different from the one of a tight sample

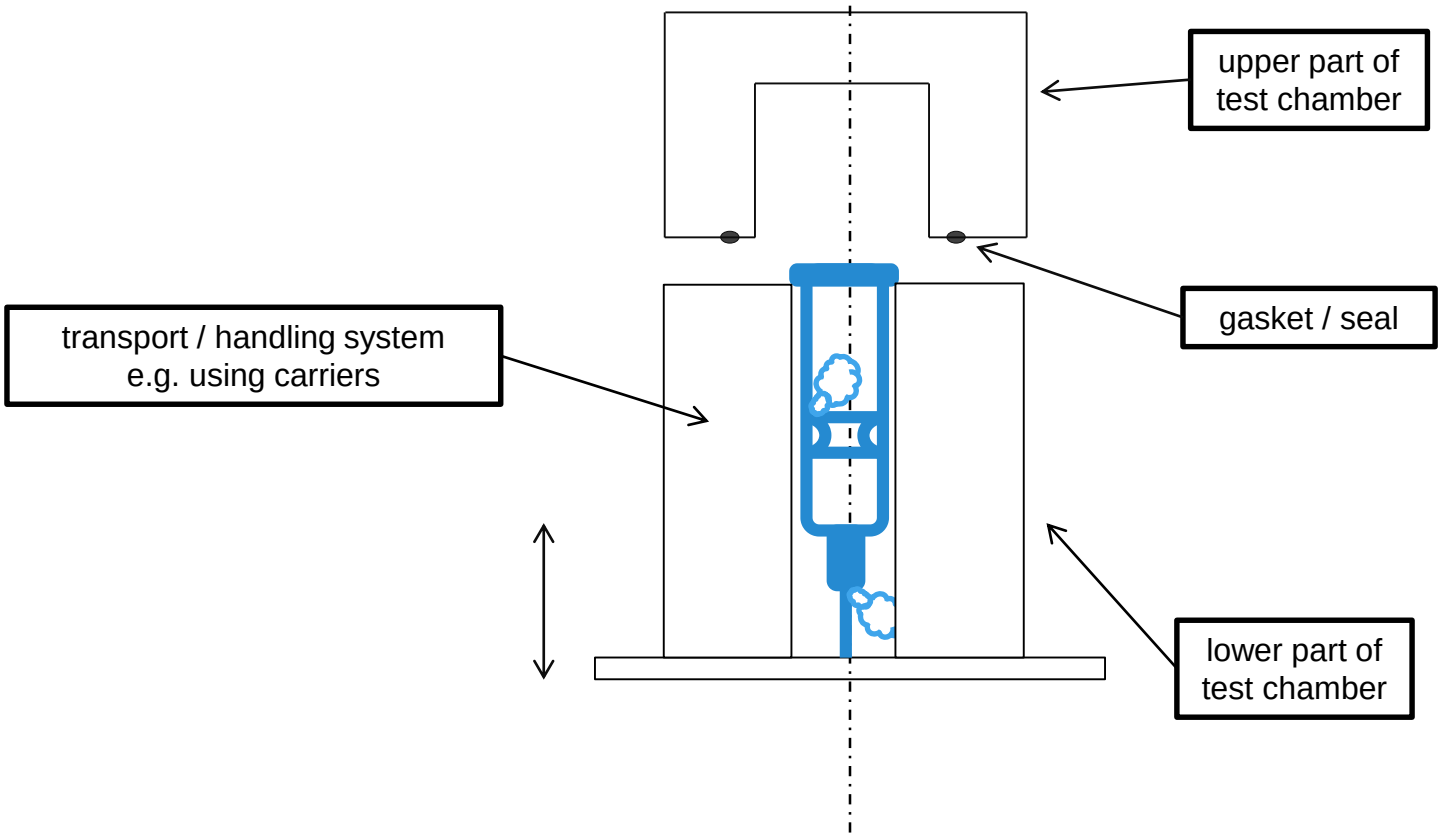


Working principle – DP test

- example of an expected differential pressure change during a vacuum test over time of a good and leaking sample

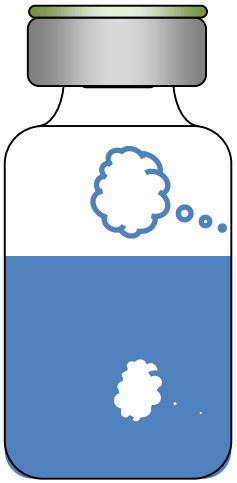


Lower / upper test chamber



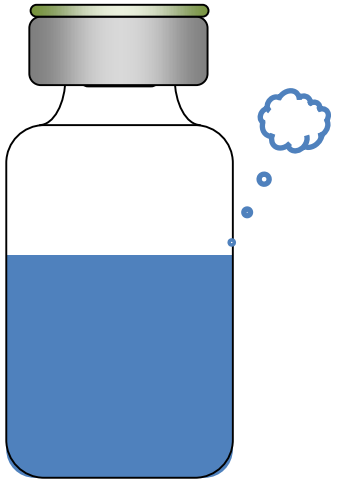
DP – variations

Pressure decay (P)

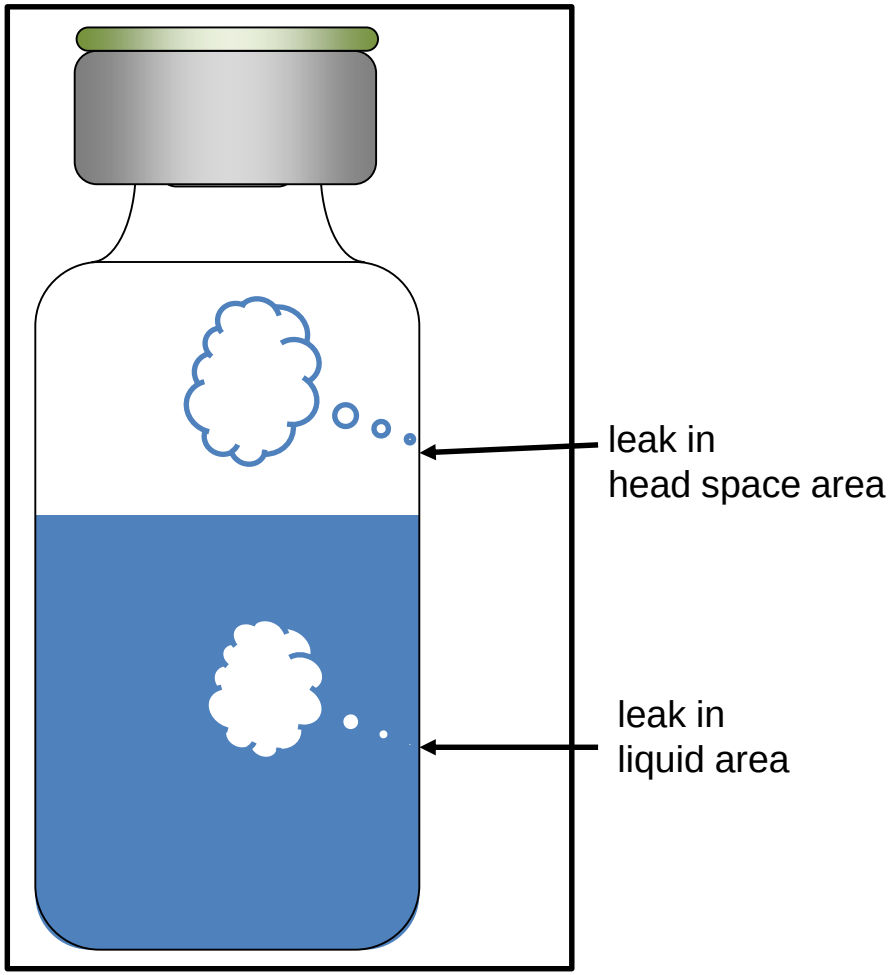


Vacuum decay (V)

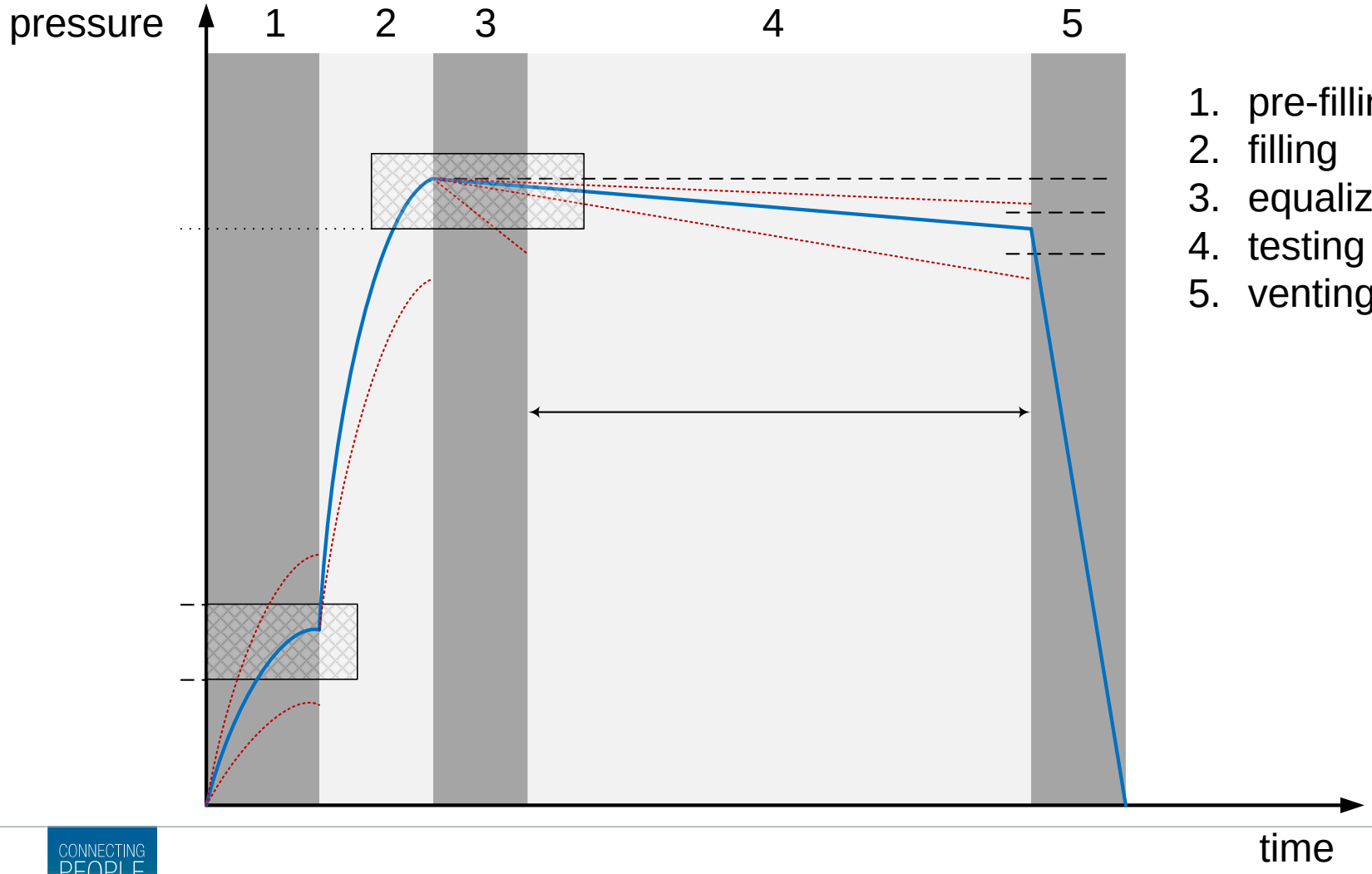
Deep vacuum vaporization (LFC)



DP – pressure decay

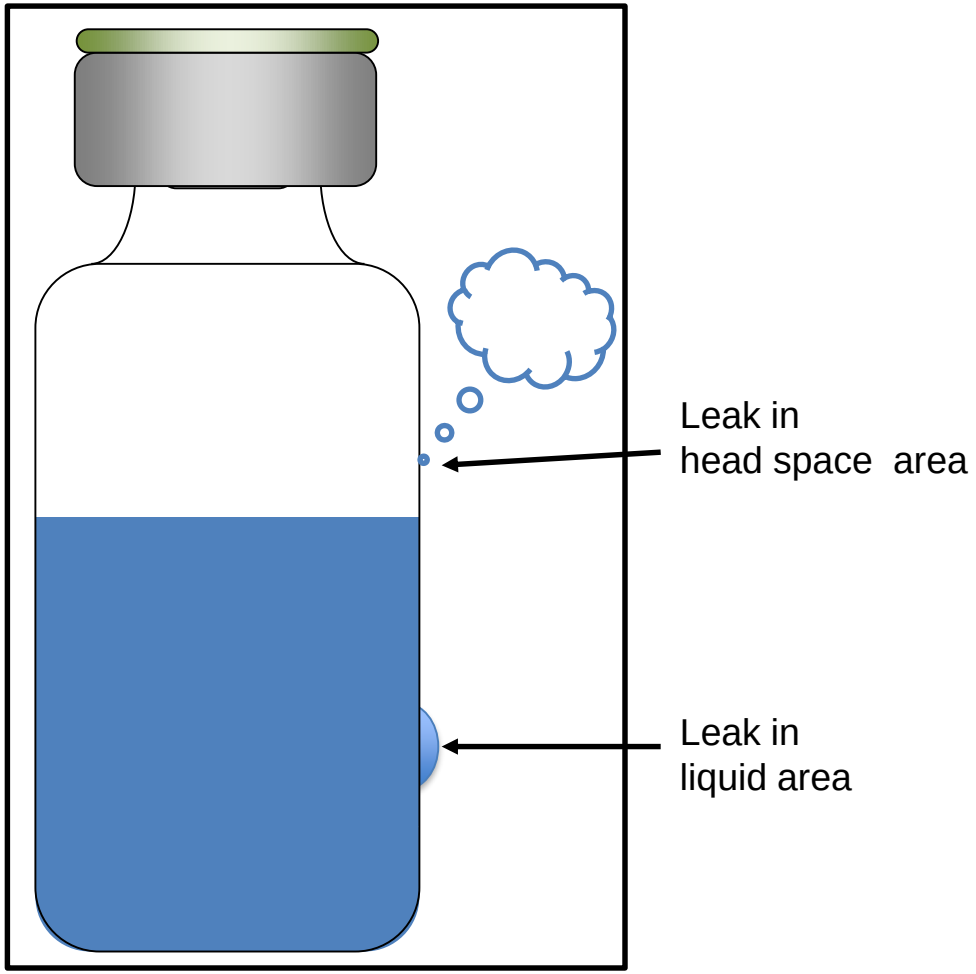


P - pressure graph

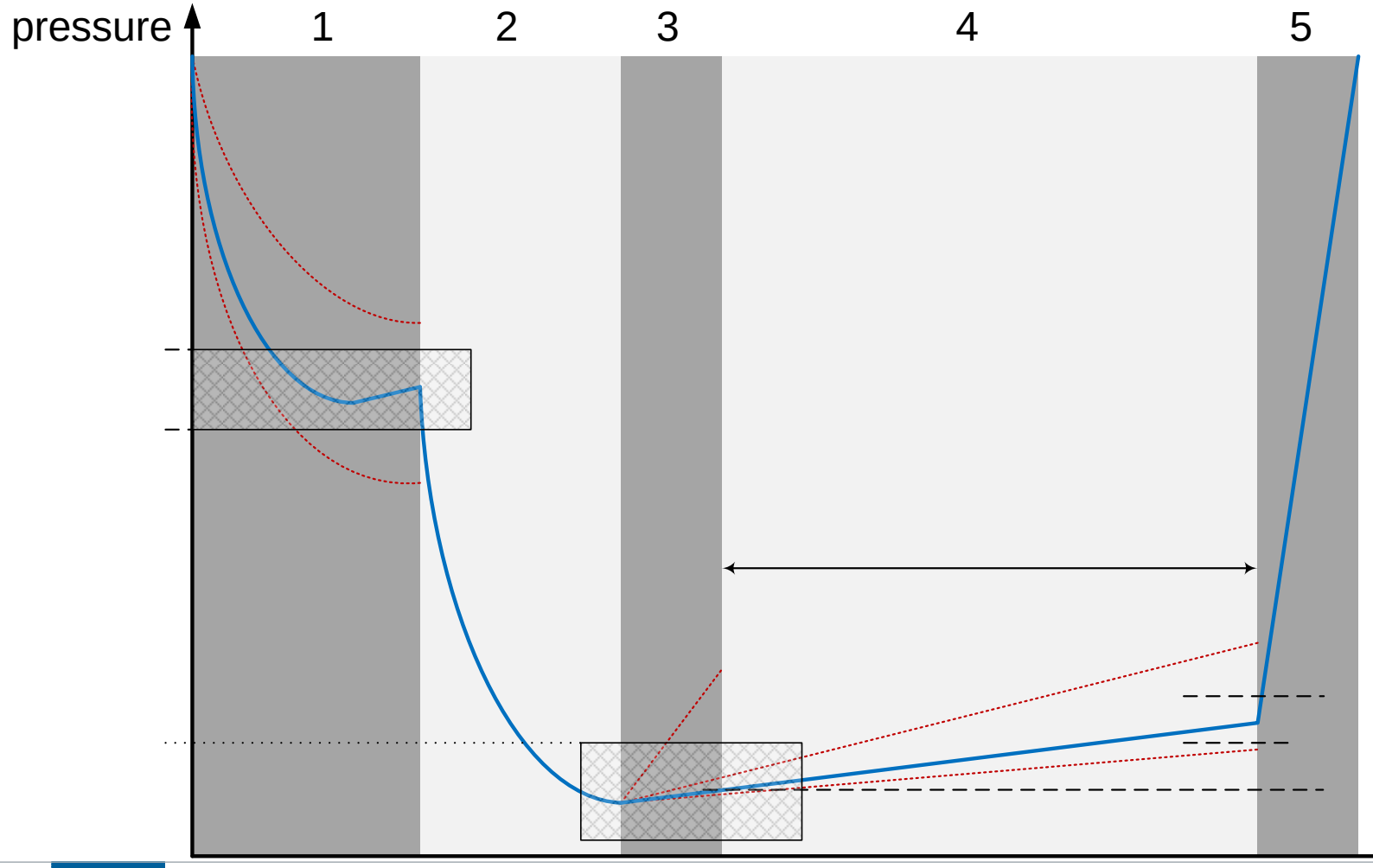


1. pre-filling
2. filling
3. equalizing
4. testing
5. venting

DP – vacuum decay

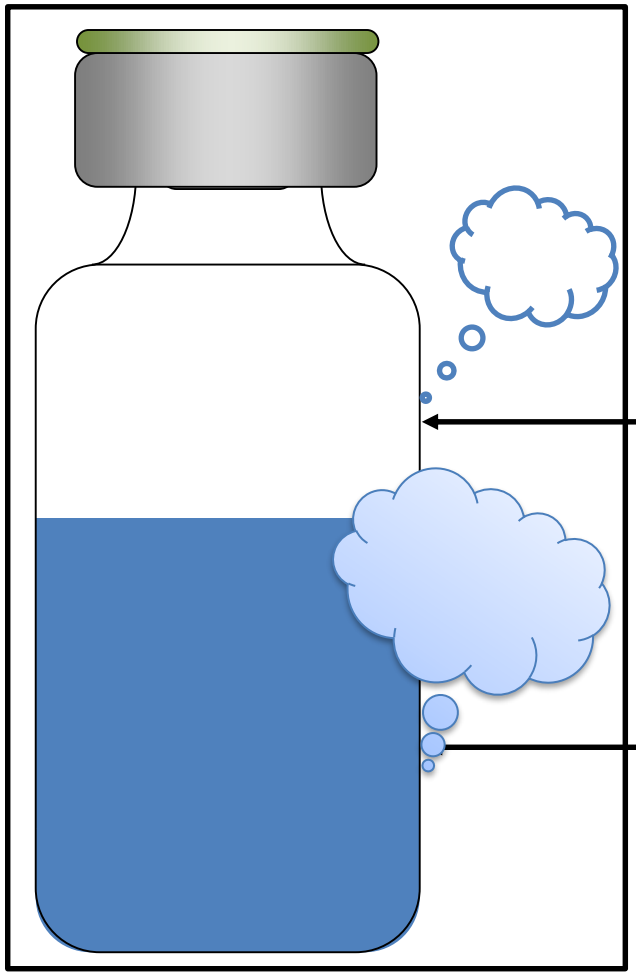


V – pressure graph



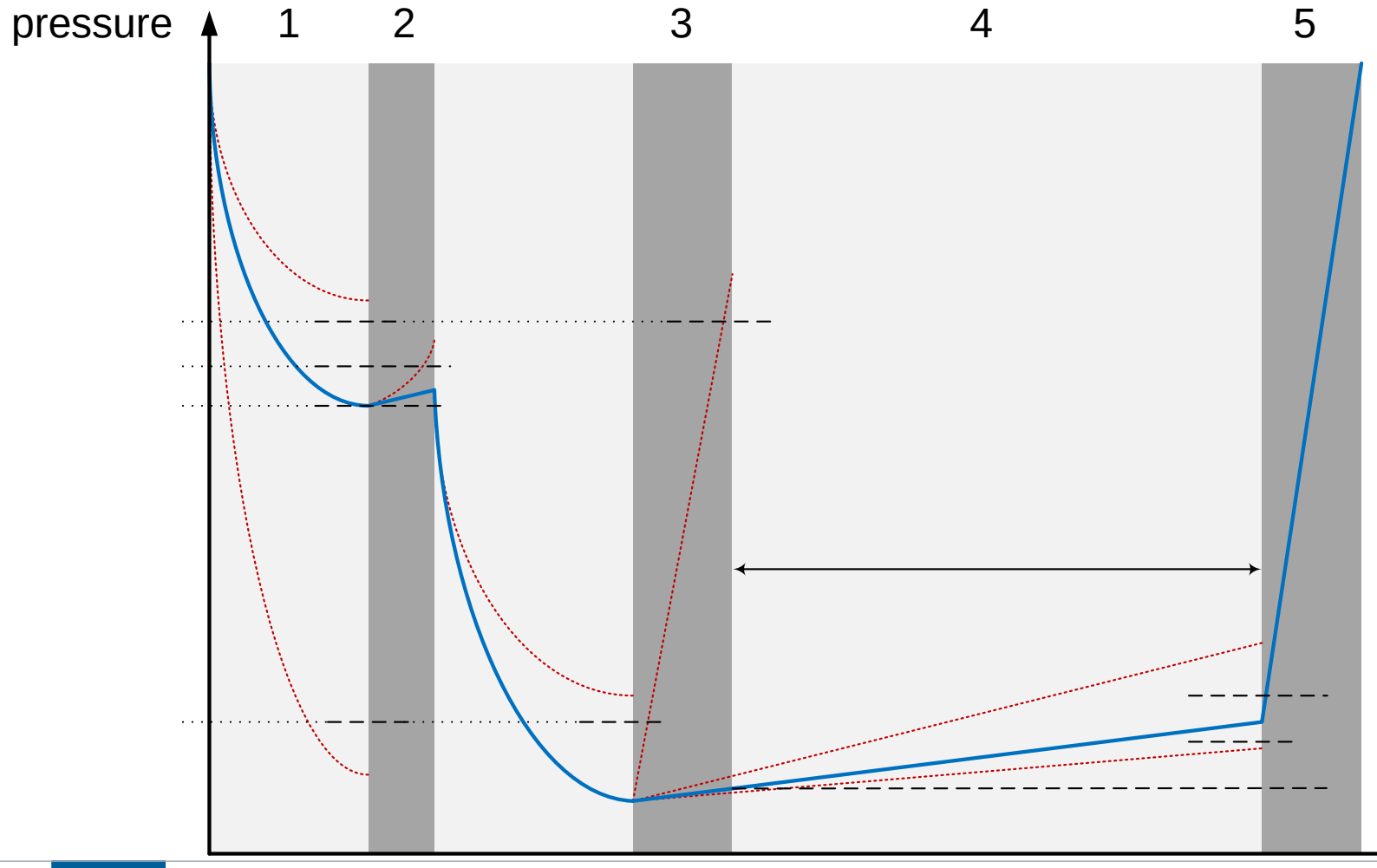
1. pre-filling
2. filling
3. equalizing
4. testing
5. venting

DP – LFC® method



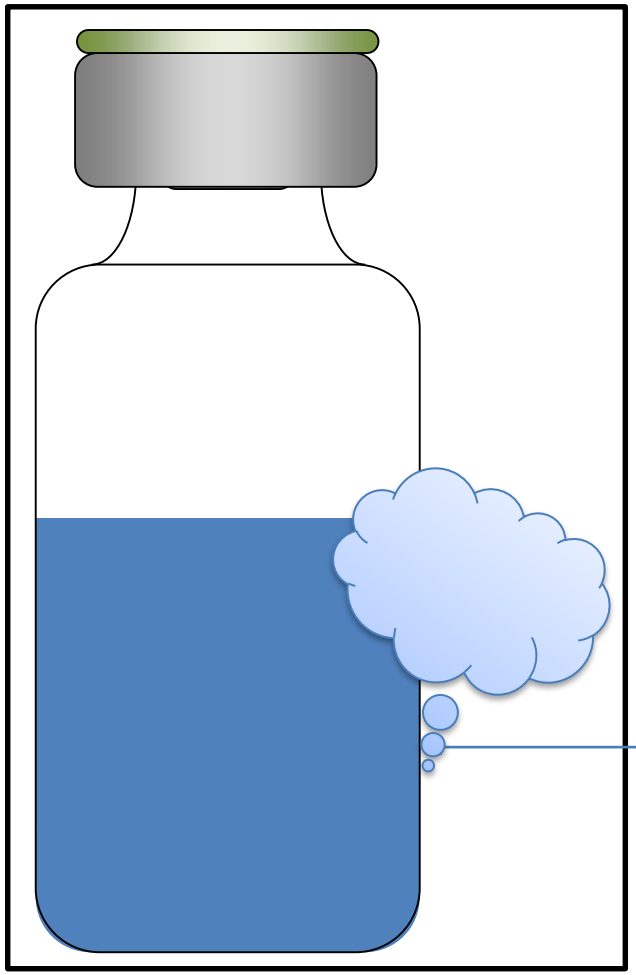
**Test chamber is evacuated below the
water vapor pressure
(e.g. 23.4 mbar absolute @ 20 °C)**

LFC® method – pressure graph



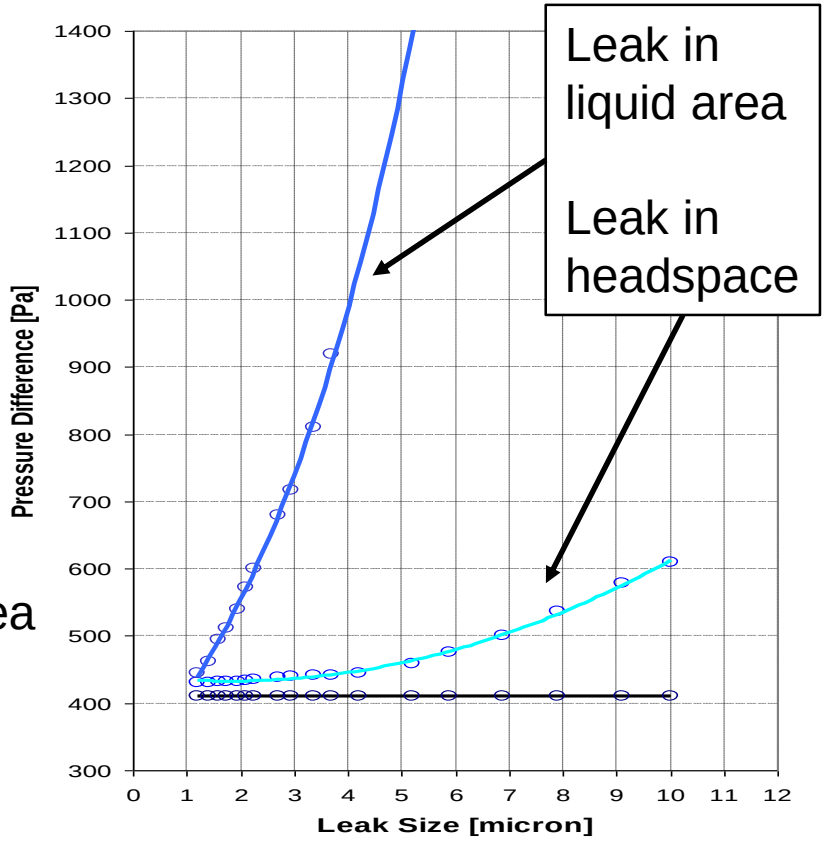
1. system protection
2. filling
3. equalizing
4. testing
5. venting

DP – LFC® method



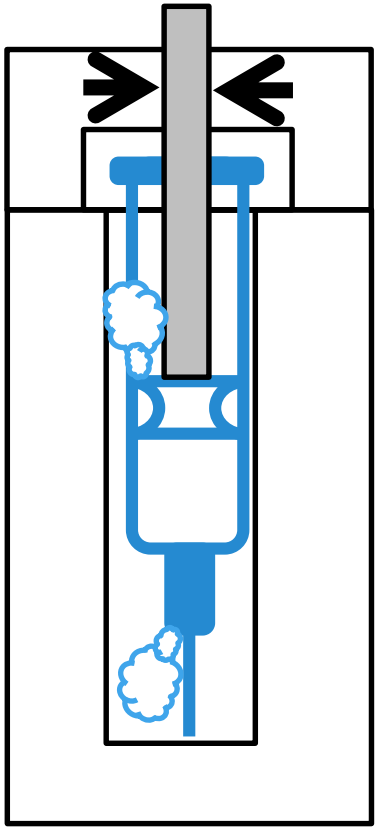
Leak in liquid area

most sensitive in liquid area due to vaporization effect of liquid



DP – on syringes

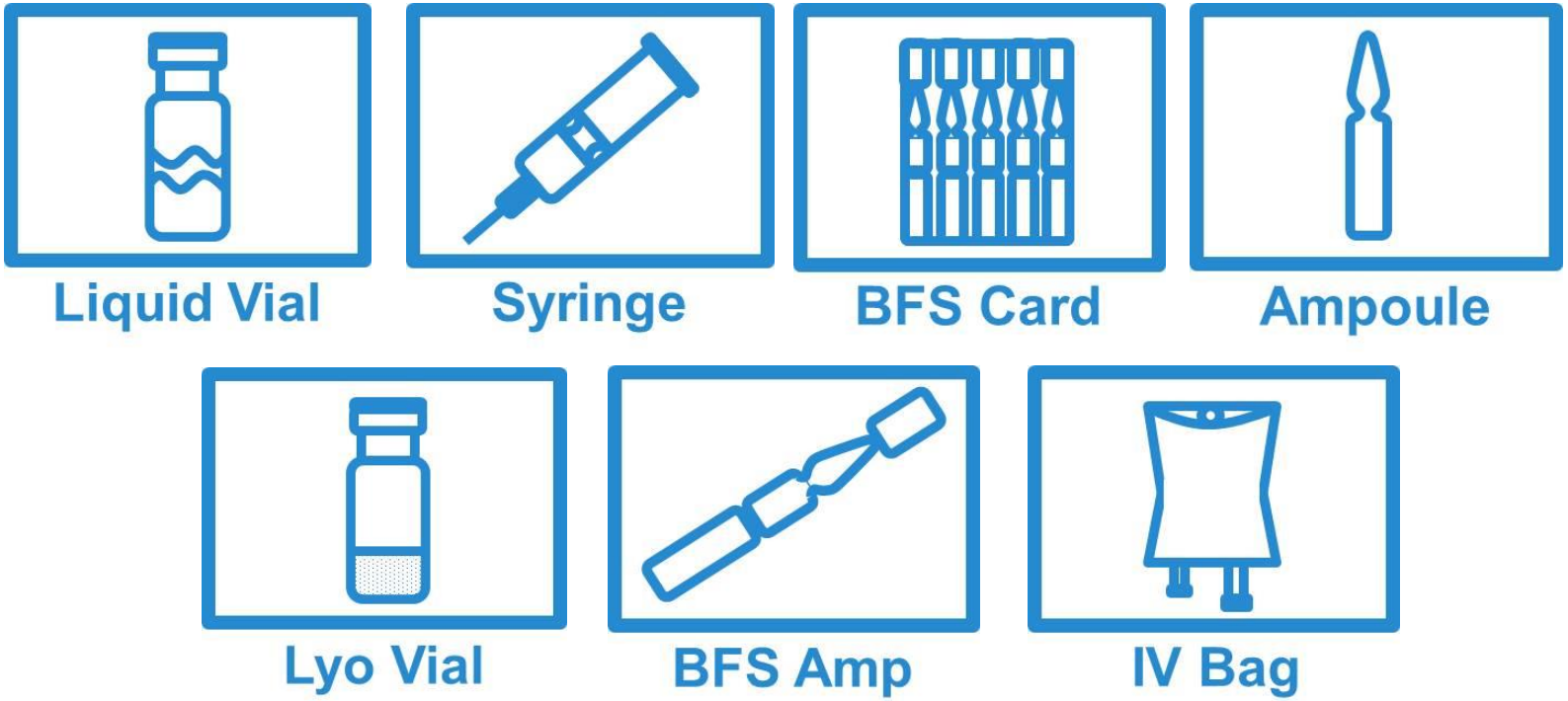
- prevention of stopper movement
- floating pin design
- test entire syringe
- no manipulation of sample



Method selection

LFC, P, V

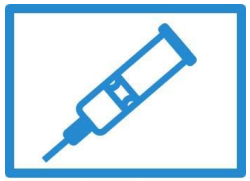
DP – applicable containers

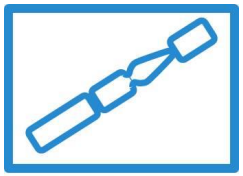


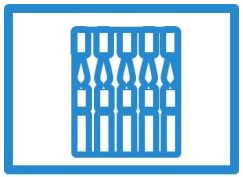
DP methods – containers / contents

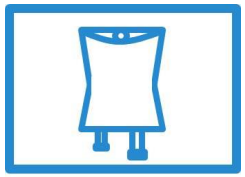
LFC


Liquid Vial

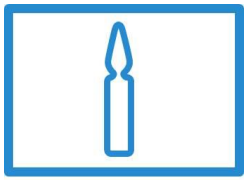

Syringe


BFS Amp

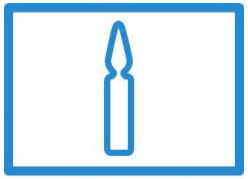

BFS Card


IV Bag


Lyo Vial


Ampoule

P


Ampoule


Liquid Vial

V


Lyo Vial

In process control

LFC, P, V

DP - in process control

Using positive samples (capillaries, laser drilled holes, cracks)

- ☹ costly
- ☹ time consuming
- ☺ most similar to negative samples (used for method development)

Determining the measuring capability of a machine: needle valve

- ☺ adjustable orifice
- ☺ set-up for a defined leak rate
- ☺ installation on every chamber
- ☺ manual or automated activation for process control

Advantages of a needle valve

- ☺ leak rate reproducible, quickly qualified by flowmeter
- ☺ no big amounts of prepared samples needed
- ☺ automated testing to challenge functionality of stations

Comparing Physical Container Closure Integrity Test Methods and Artificial Leak Methodologies; S. Pelaez, M. Kahl, R. Mattes et al.; PDA Journal of Pharmaceutical Science and Technology; 2019

Applications

from lab scale to inline systems

Lab scale – NEO DPX

Key Advantages

- multiple presentations: vials, syringes, ampules
- all three DP test methods included
- improved sensitivity of the DP measurement technology, down to 1.1 µm
- recipe management system

Trends

- many presentations and size range
- enhanced sensitivity
- simple changeover
- development of recipes by end-user

NEO DPX – changeover

NEO DPX – changeover

R 36 MC/LFC @ 400 / min

Thank you!