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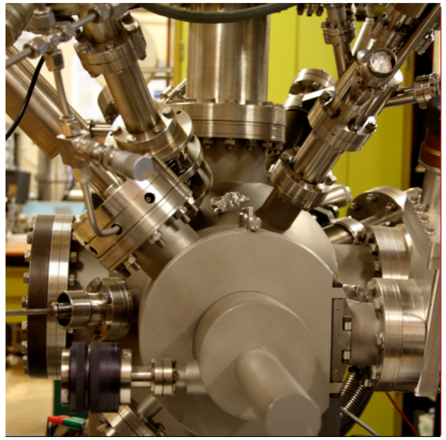

Calibration of reference leaks in a wide pressure range

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Outline

- Motivation
  - Reference leaks for sniffers, R134a leaks
- Calibration set-up
  - Syringe and stepper motor
  - User interface
  - Measuring range
  - Temperature effects
  - Repeatability
  - Time needed for calibration
- Typical uncertainty
- Method validation
- Typical results
  - R134a and He leaks

Motivation: reference leaks for sniffers

- Inside-out He leak detection technique (sniffer)
  - He reference leaks in the range 10^{-6} to 10^{-5} mbar.L/s
- 842/2006 regulation and subsequent statements
  - R134a reference leaks of 5 g/year

REGULATION (EC) No 842/2006 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL

of 17 May 2006

on certain fluorinated greenhouse gases

COMMISSION REGULATION (EC) No 1516/2007

of 19 December 2007

establishing, pursuant to Regulation (EC) No 842/2006 of the European Parliament and of the Council, standard leakage checking requirements for stationary refrigeration, air conditioning and heat pump equipment containing certain fluorinated greenhouse gases

2. Gas detection devices referred to in paragraph 1(a) shall be checked every 12 months to ensure their proper functioning. The sensitivity of portable gas detection devices shall be at least five grams per year.

+ Motivation

- Calibration by comparison using a MS leak detector is not suitable
  - Calibration pressure should be atmosphere, not vacuum
- EN 13192 method C: calibration of leaks from overpressure to atmosphere
  - Capillary with a moving slug
  - Although vey simple, is of limited application (slug friction, pressure control, etc.)
- However, $p\Delta V$ method is well suited

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

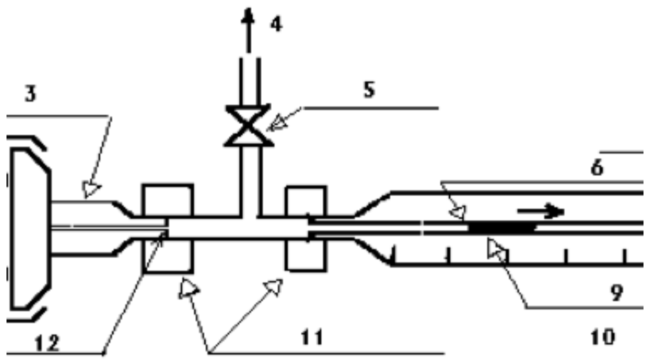
EN 13192

November 2001

ICS 19.100

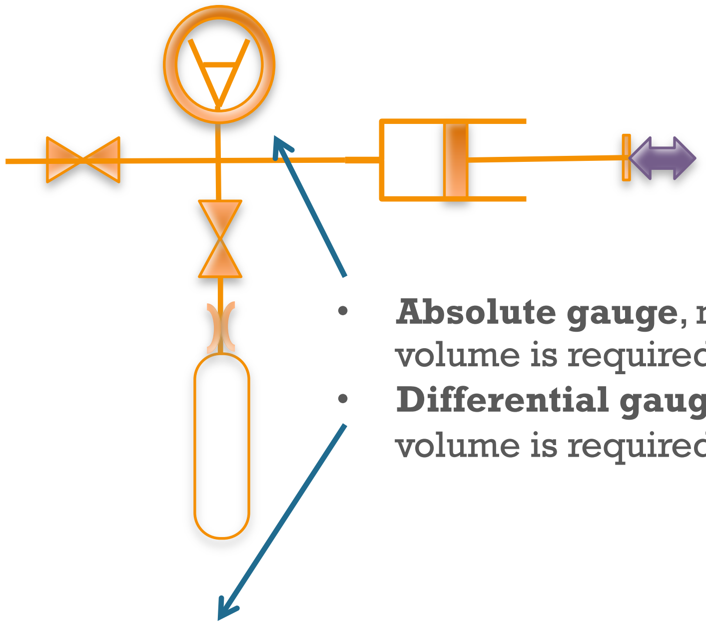
English version

Non destructive testing - Leak testing - Calibration of reference leaks for gases

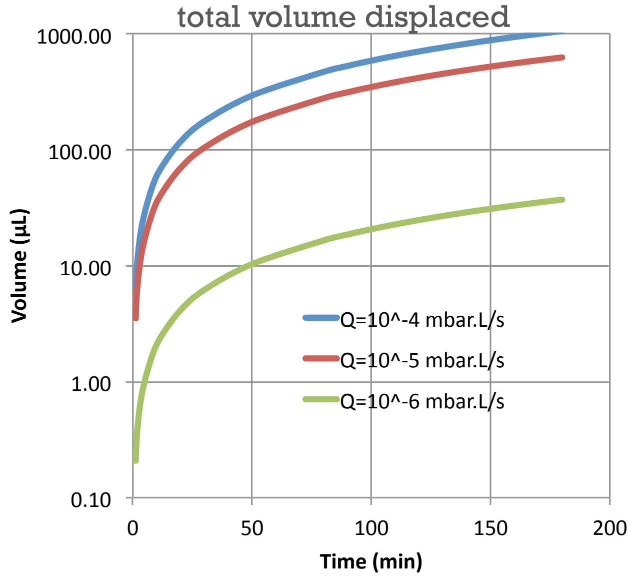


+ Calibration method: $p\Delta V$

- The leak is connected to a closed volume
- A piston is actuated every time a pressure increase is detected to keep the pressure constant
  - **Volume ≈ 1 mL, step < 1μL (resolution)**

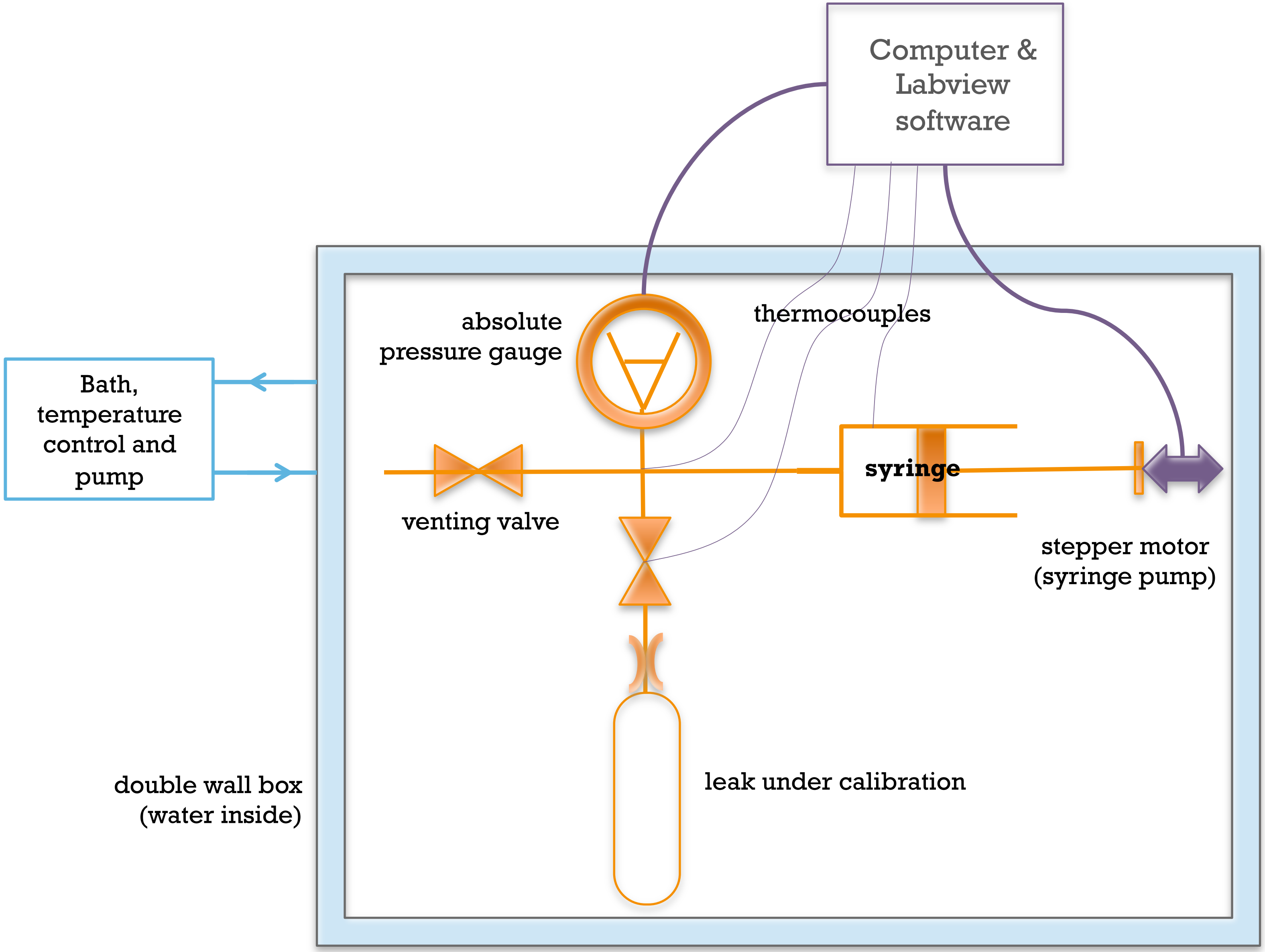


- **Absolute gauge**, no further volume is required
- **Differential gauge**, reference volume is required



1. M. Bergoglio e D. Mari, "INRIM primary standard for microgas-flow measurements with reference to atmospheric pressure" *Measurement 45* (2012) 2459
2. K. Jousten e U. Becker, "A primary standard for the calibration of sniffer test leak devices," *Metrologia,* 46 (2009) 560-568
3. F. Boineau, "Characterization of the LNE constant pressure flowmeter for the leak flow rates measurements with reference to vacuum and atmospheric pressure," *PTB-Mitteilungen,* 121 (2011) 313

+ Calibration set-up

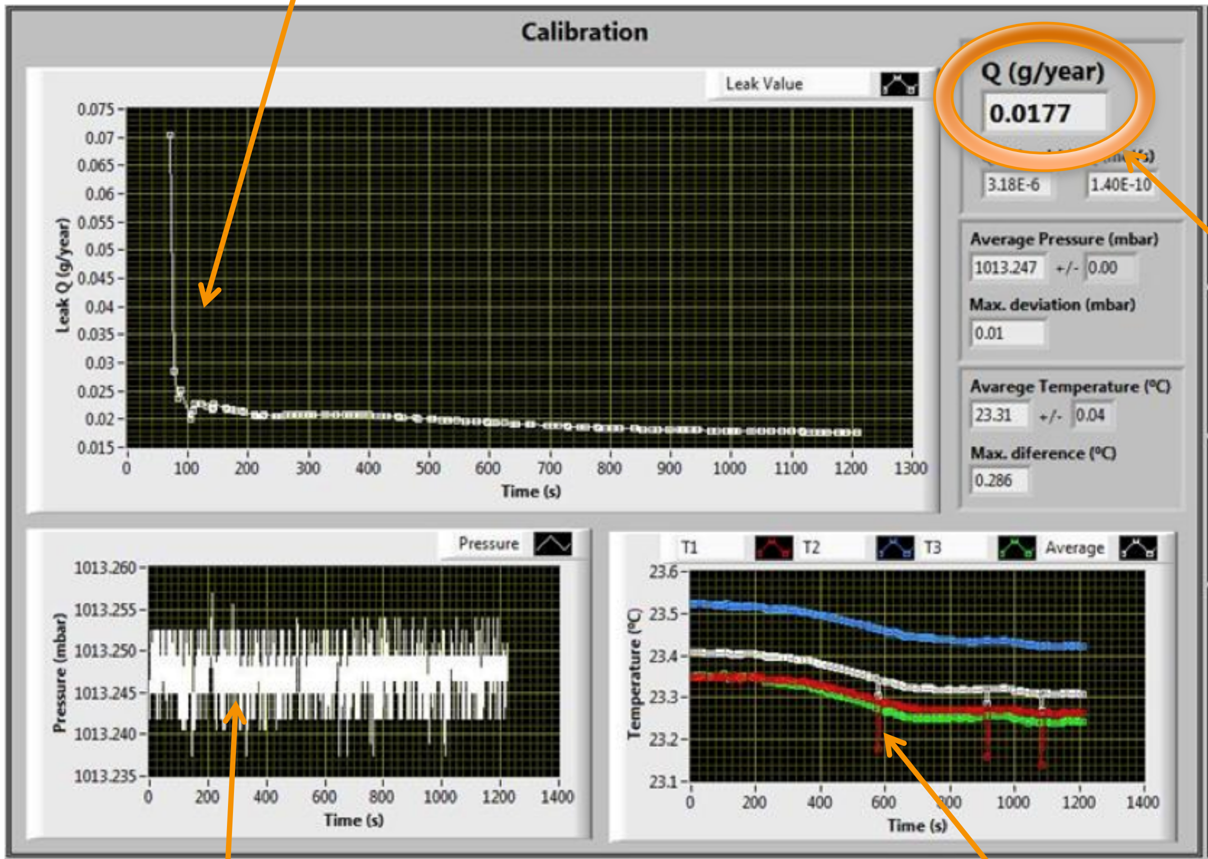


Calibration set-up

- Piston: **1 mL** SGE gas tight syringe with PTFE plunger tip
- Piston actuator: syringe pump NE-500
  - 800 steps/revolution; 1 step≈ 1.7 µm
- Gauge: Druck DPI 142 precision Barometer
  - Resolution ≈ 3×10^{-3} mbar; piezo sensor
- Software: Labview platform
- Tubing: 1/8'' swagelok fittings and valves
- Stainless stell double wall box with 2 computer fans inside to increase temperature homogeneity
- Water circulator Julabo F25-ED

User interface

Flow rate (integrated measurement), converges with time



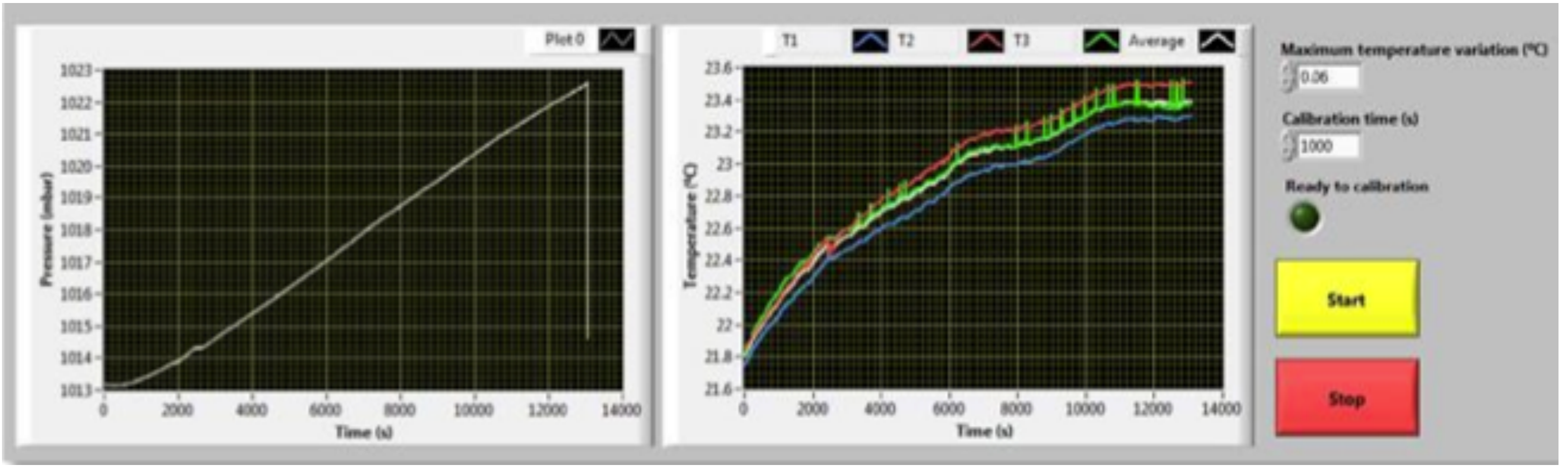
Actual measurement

Pressure (≈10^{-2} mbar maximum variation)

Temperature drift (3 thermocouples), critical to achieve low uncertainty

Before calibration

- ΔpV method, for preliminary evaluation
- temperature stability confirmation



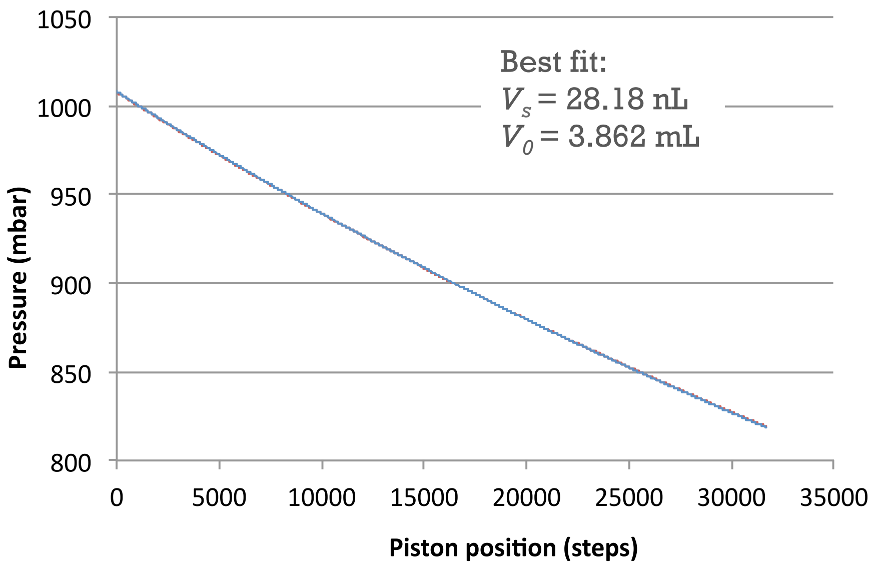
Syringe characterization

- Gravimetric volume calibration
  - Step volume V_s=28.0 ± 0.22 nL
- Syringe expansion without leak
  - Boyle-Mariotte relation
  - Full volume range ≈ 32 000 steps
- Step volume V_s and dead volume V_0 can be calculated by fitting these parameters in the experimental data

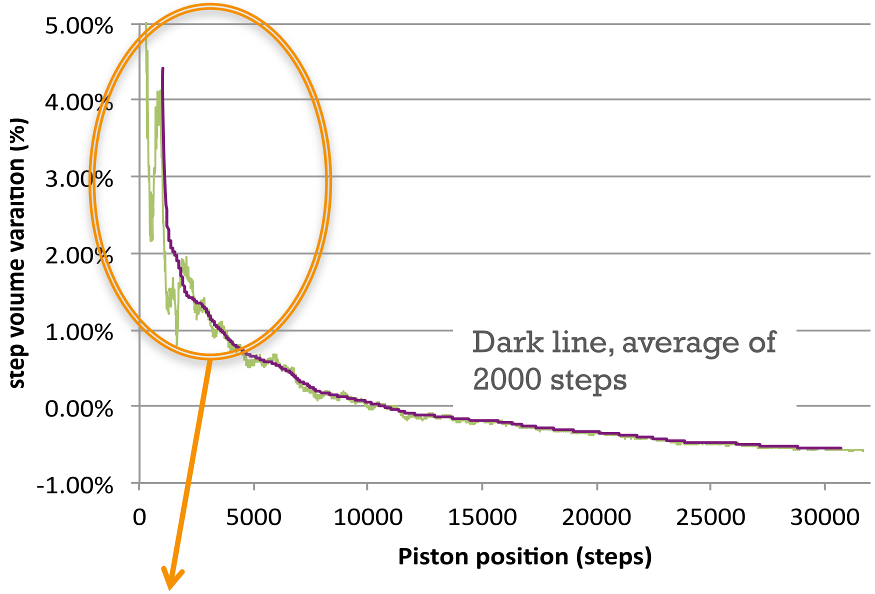
$$p = \frac{p_0 V_0}{n V_s + V_0}$$

fitting parameters

$$V_s = \frac{V_0}{n}\left(\frac{p_0}{p} - 1\right)$$



Step volume as function of position



most used position = larger diameter

Measuring range, time needed for calibration

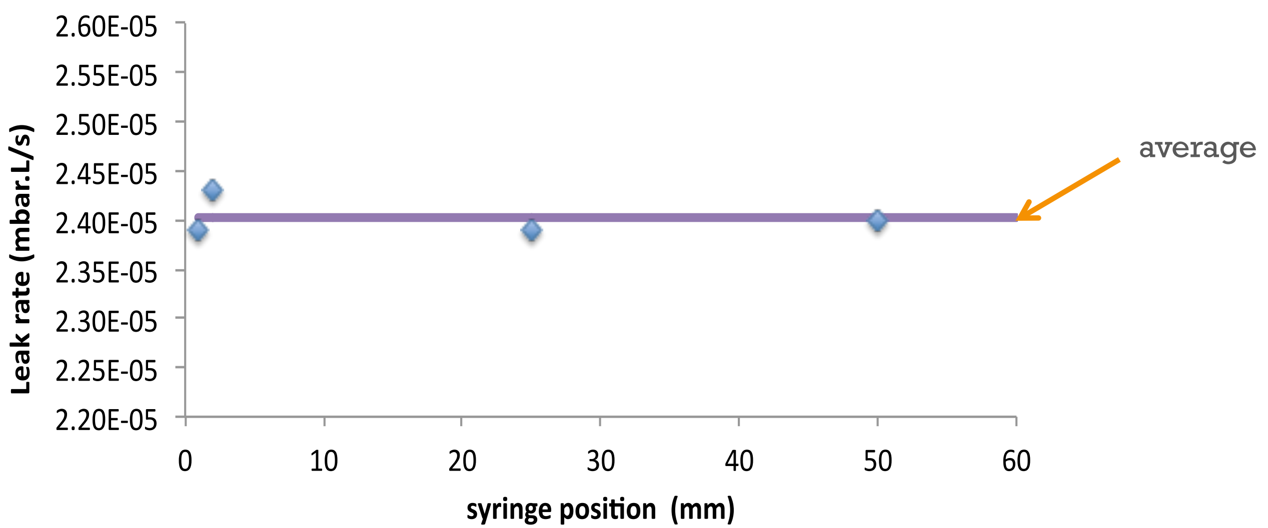
- 2000 steps averages thread defects

total steps	Q	time		
		s	min	h
2000	10^{-4} mbar.L/s	560	9.3	
	10^{-5} mbar.L/s	5600	93.3	1.6
	10^{-6} mbar.L/s	56000	933.3	15.6
	5 g/year R134 a	1474	24.6	

Repeatibility

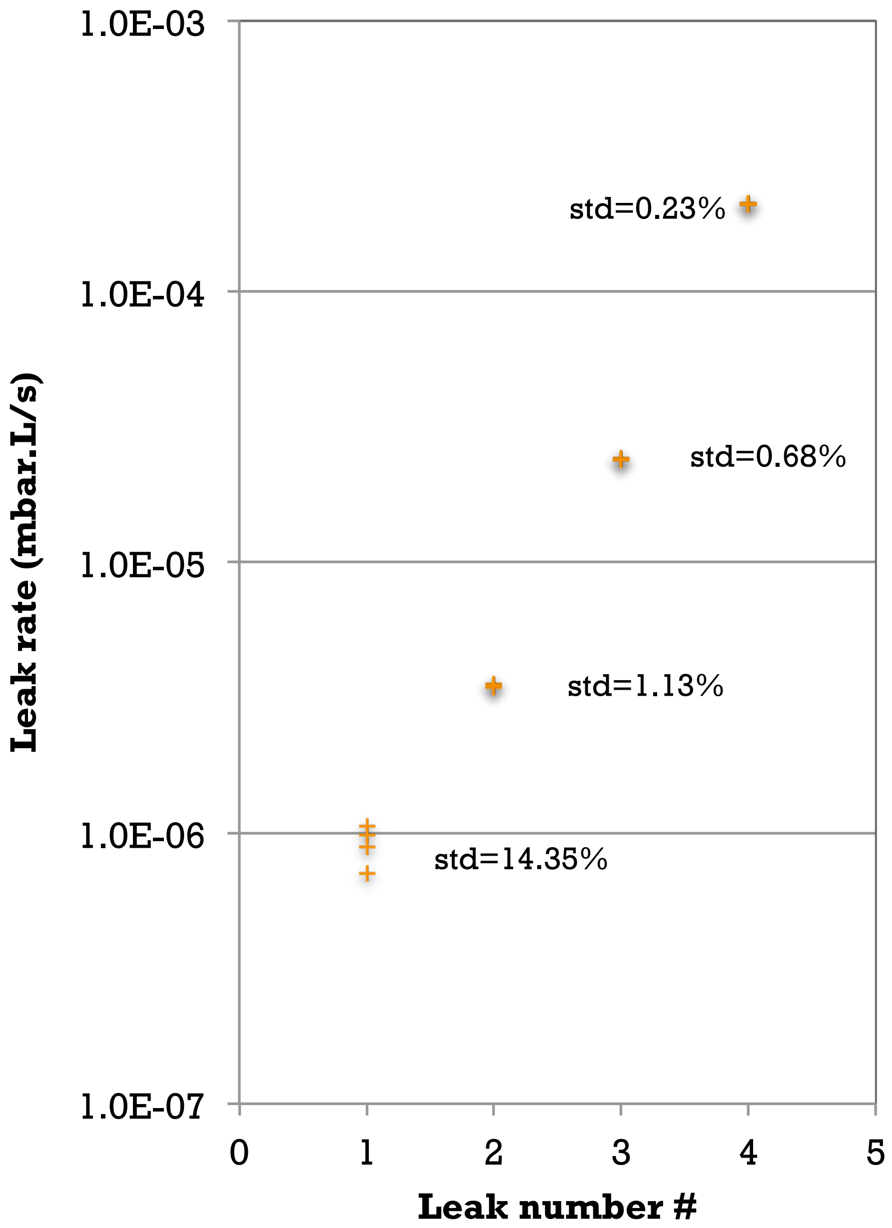
- Effect of the position of the syringe plunger tip (piston)

He leak, nominal leak rate = 2.60 × 10^{-5} mbar.L/s calibrated to vacuum



+ Repeatability

- 4 different leaks were measured 4 times each
- Repeatability depends on:
  1. Number of steps (acquisition time)
  2. Temperature drift has a major impact
  3. Leak stability
- On the graph 1000 to 10000 steps were used.



Typical uncertainty

- R134a leak, Q=3.48 g/year

Quantity, x_i	Estimate	u(x_i)	c_i u(x_i)/d (g/year)	relative contribution %
pressure	1000 mbar	0.015 mbar	1.54E-05	0.027
volume	22 µL	0.17 µL	7.79E-03	13.7
temperature	296.9 K	0.06 K	5.38E-04	0.4
time	827 s	9.6×10^{-3} s	1.16E-05	0.02
repeatability (ΔT≈0.1K)		0.17 g/year	0.049	85.9
Total (k=2)			**0.098** **2.82%**	100

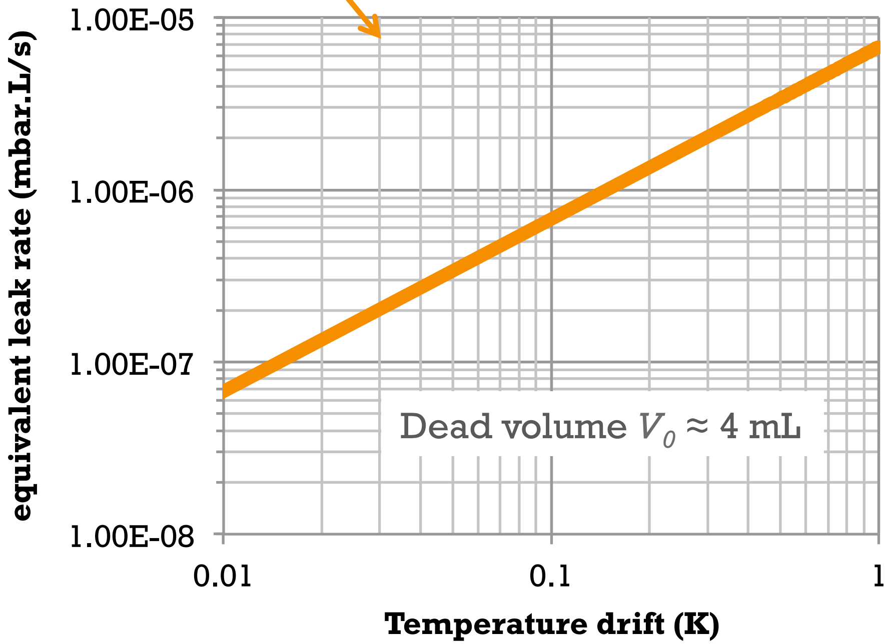
Q = 3.48 ± 0.10 g/year

Temperature effects

- The use of an absolute gauge turns this calibration set-up more sensitive to temperature drifts
  - A drift in the temperature is equivalent to a **temporary virtual leak** (positive or negative)
  - This effect is slightly cancelled by tubing dilatation

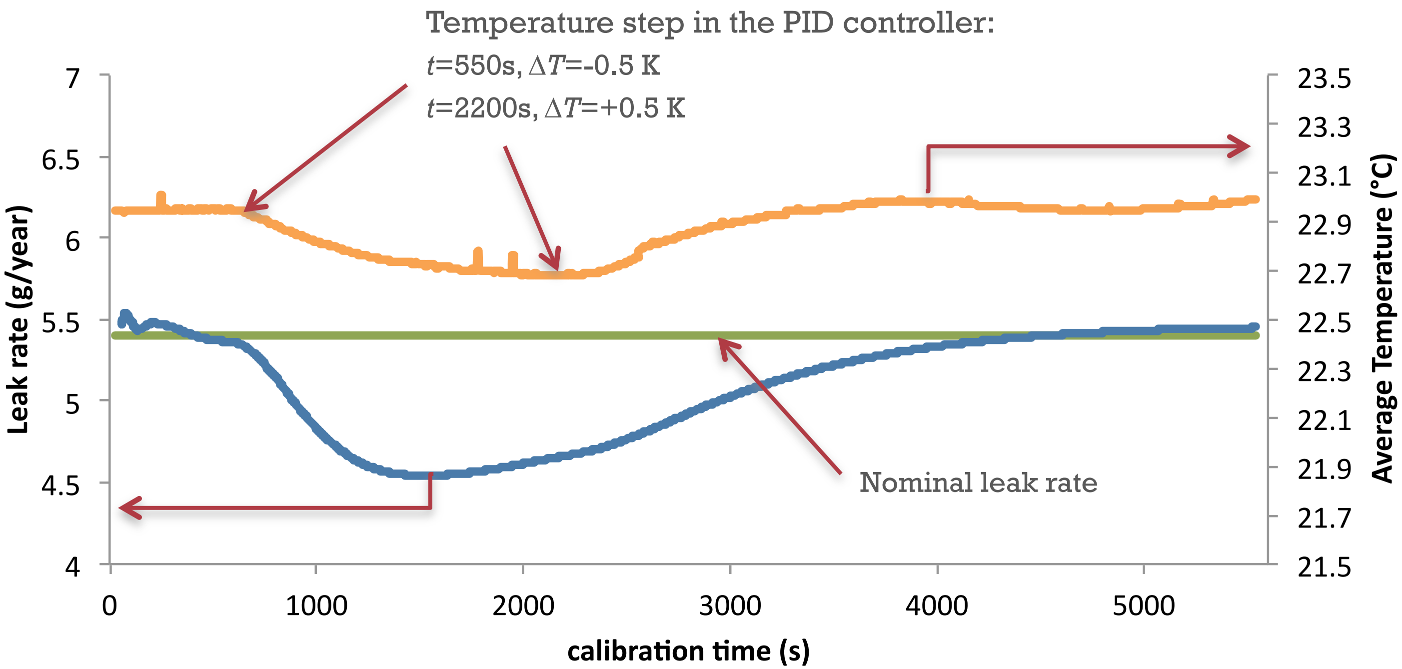
Gay-Lussac law

$$Q_{eq} = \frac{p_0 V_0}{t}\left(\frac{T_0 + \Delta T}{T_0} - 1\right)$$



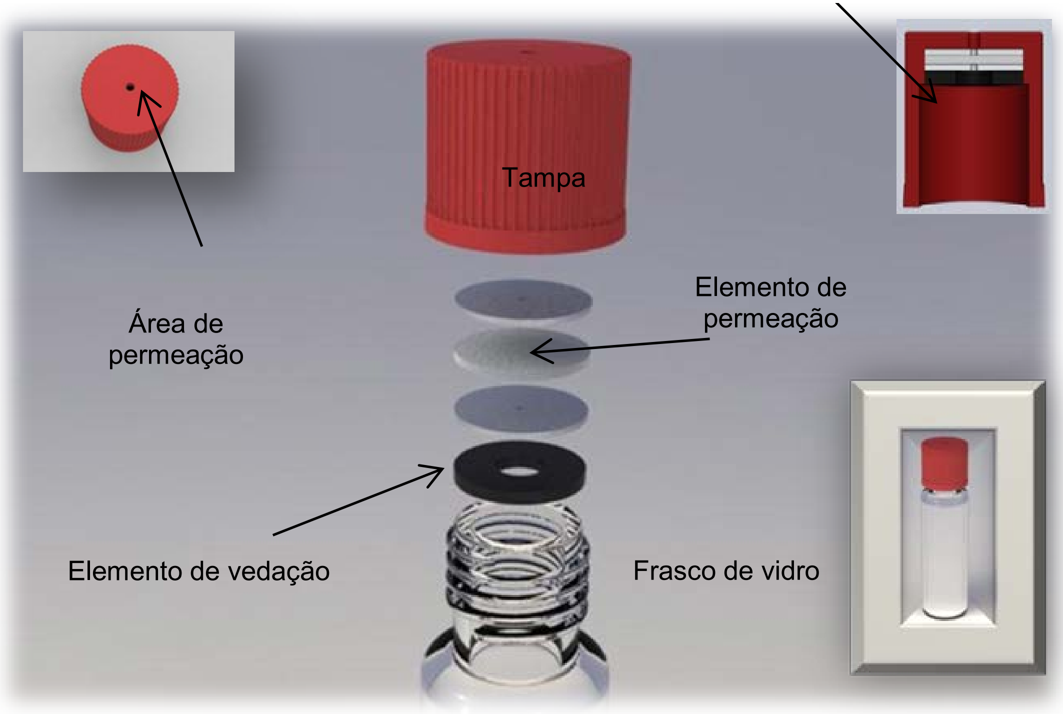
Temperature effects

- Total temperature drift is more important than temperature uniformity.
  - If temperature changes slowly, what matters is the difference between the starting temperature and the final temperature
- Experiment: induced temperature step
  - Temperature effects may be cancelled by positive and negative drifts



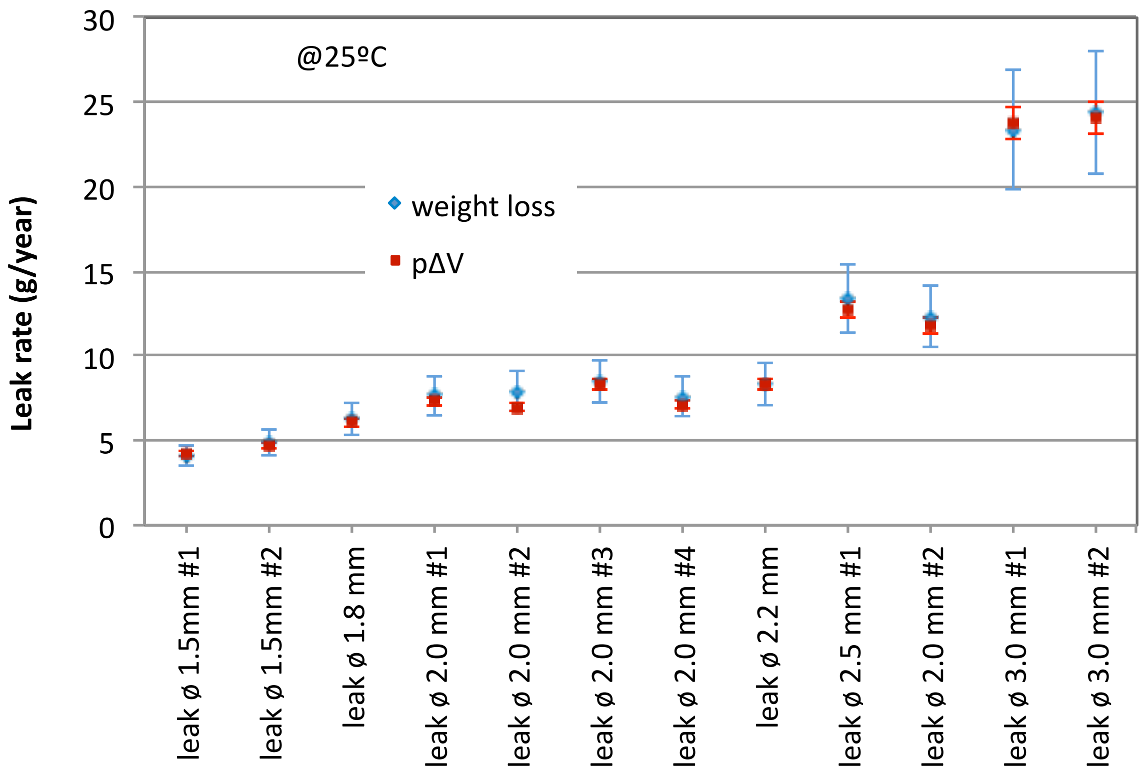
Method validation

- A series of R134a leaks were built with leak rates ranging 4 to 24 g/year



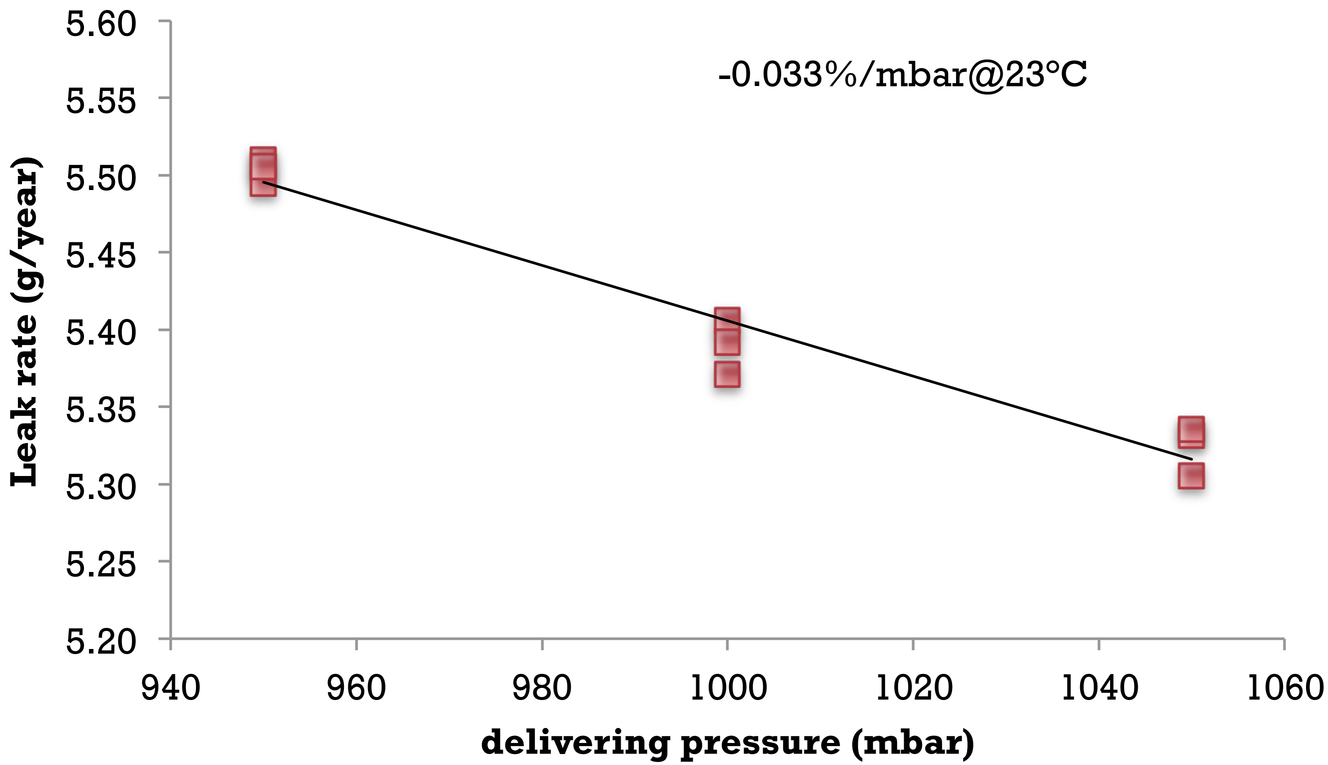
Method validation

- Leaks were weighted over several days (U=± 15%) and leak rate calculated.
- Then, all leaks were calibrated in the *p*Δ*V* set-up and compared



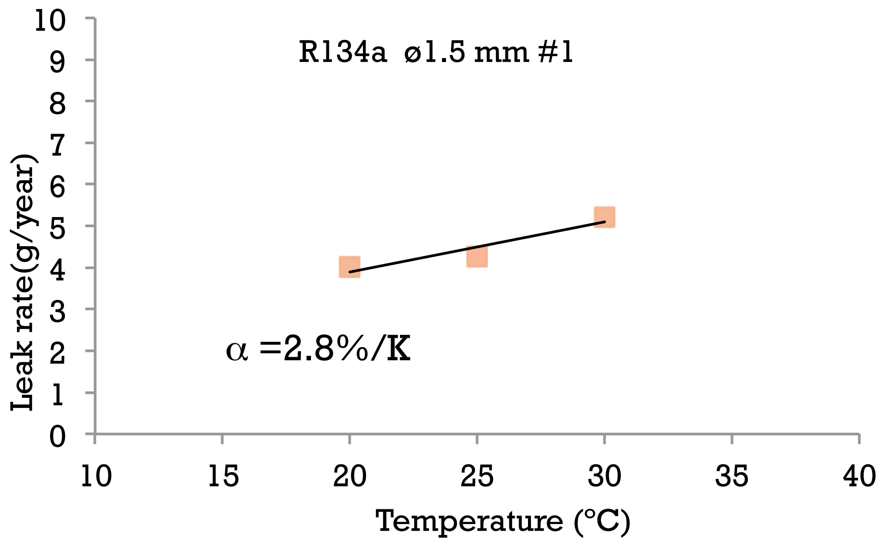
R134a reference leaks

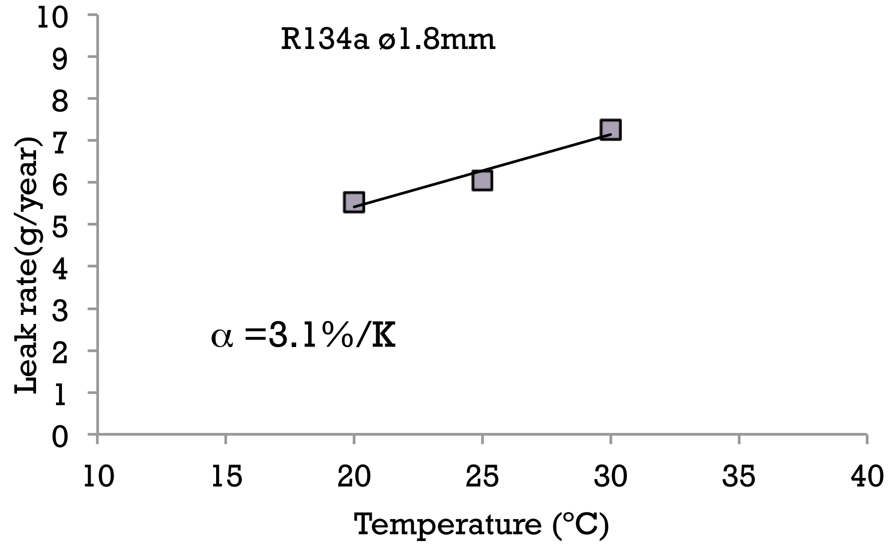
- Dependence on the delivering pressure
  - 2000 steps measurements ≈ 25 min



R134a reference leaks

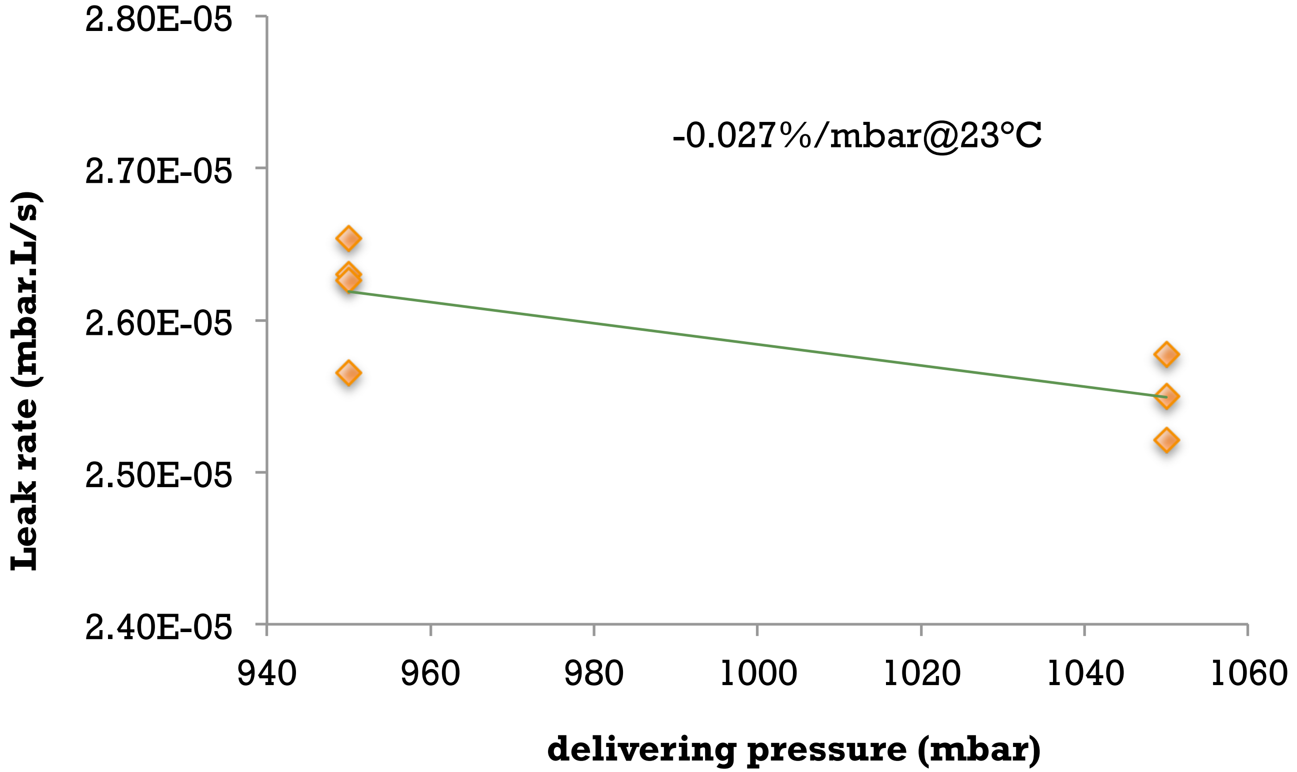
- Dependence on the temperature





+ He reference leaks (permeation)

- Dependence on the delivering pressure
  - 2000 steps measurements ≈ 35 min



+ Conclusions

- $p\Delta V$ calibration method was assembled with a syringe pump and is properly working at Metrovac laboratory in Lisbon.
- One absolute pressure gauge is used
  - This gauge is suitable to measure both the calibration pressure and the pressure change
- Temperature stability is critical to achieve good uncertainty
- Uncertainties ≈3% are easily achieved for the range of 10^{-5} mbar.L/s or 5g/year
- R134a reference leaks were developed and fully characterized.
  - This leaks were used to validate the method

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Thanks for your attention

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