Federal Department of Justice and Police FDJP
Federal Institut of Metrologie METAS

NO_2 portable permeation and dilution system :PoPS (or TMPG, Traceable mobile permeation generator)

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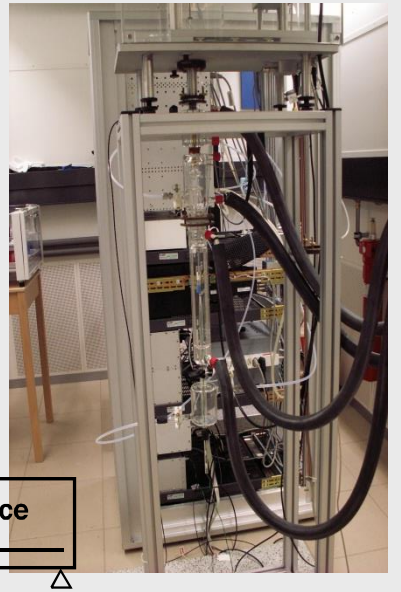
Introduction

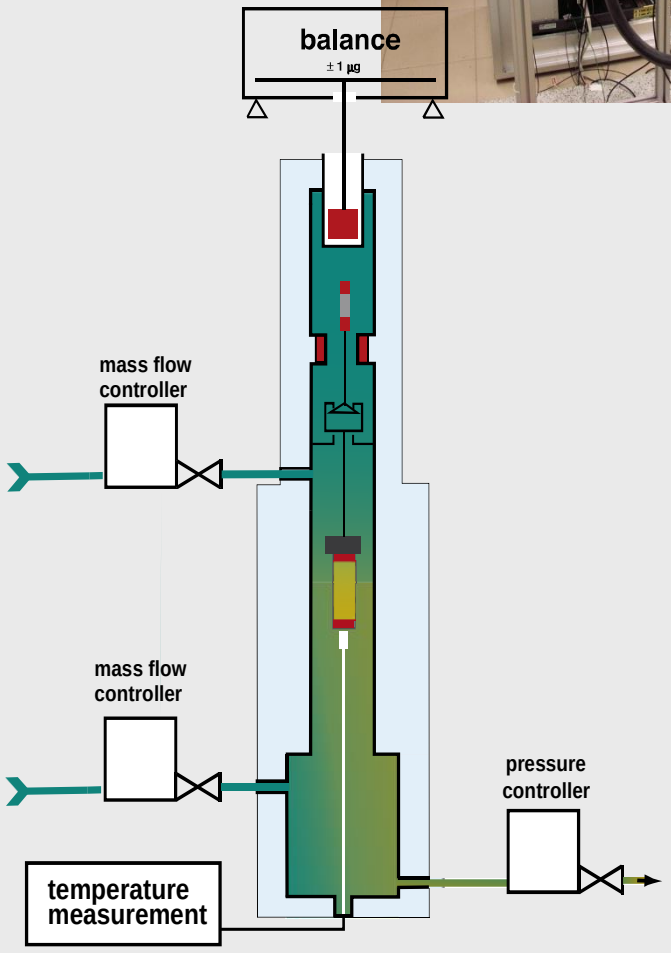
Production of NO_2 mixtures by permeation:
Well-known and standardized method (ISO 6145-10)

METAS:
Calibration of the permeation unit with the microgravimetric methode based on a magnetic suspension balance (**MSB**) from Rubotherm

→ Building up of a transfer standard: The **PoPS** (or TMPG, traceable mobile permeation generator)

Transfer of the permeation unit in the PoPS

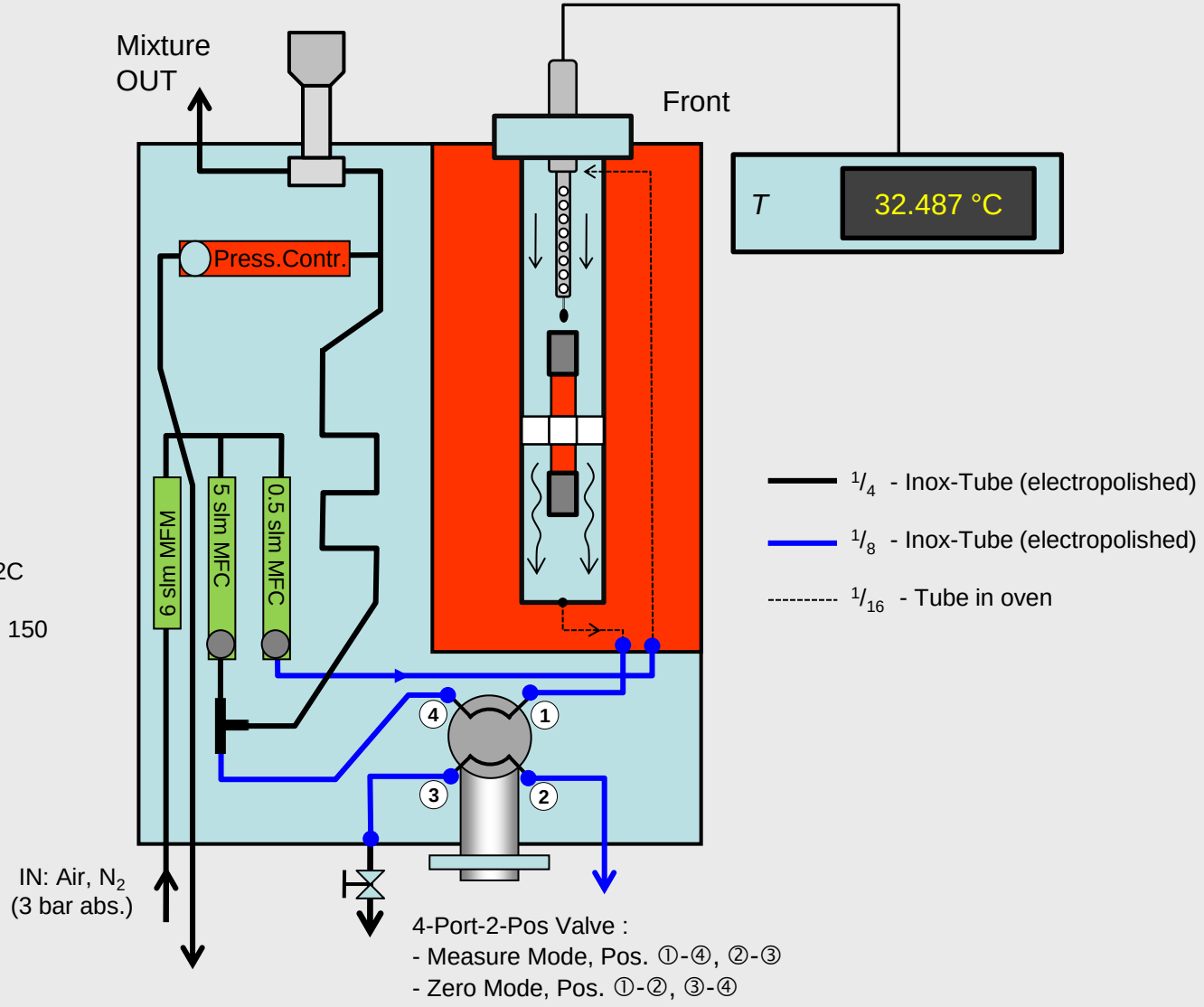




Picture of PoPS

Schematics of POPS

Components:
- Hart 1504 Tweener
- red-y smart controller GSC-A5TT-BB22 0.5 slm
- red-y smart controller GSC-B5TT-BB23 5 slm
- red-y smart meter GSM-B9TT-BN00 6 slm
- Bronkhorst EL-Press P-702C Pressure Controller
- VICI Dynacalibrator, Model 150

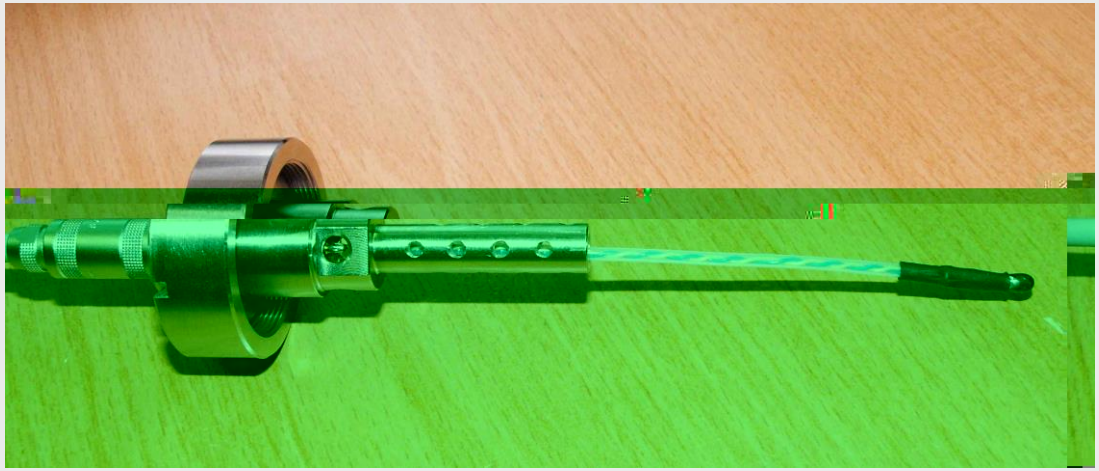


Components (I)

Oven: Vici Dynacal Mod. 150 OEM Oven. Inner surface is silanised (glass coated) stainless steel:

- Excellent stability (< 3 mK over several days)
- Best possible surface material for NO_2
- Offset of temperature setting (~ 0.2 K)
- Difficult accessibility for independent temperature probe

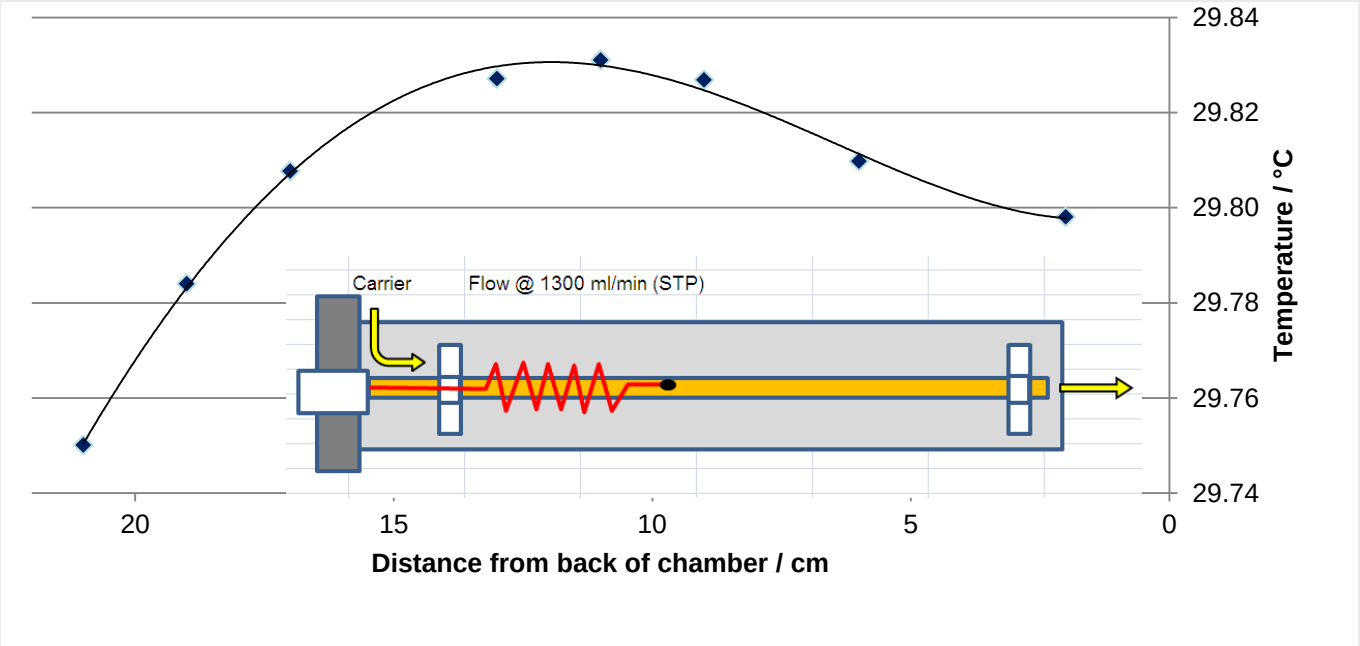
Action: Reconstruction of sealing cap and incorporation of leak tight flexible temperature probe (thermistor)

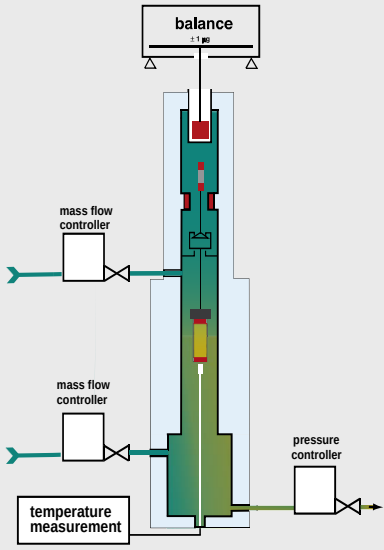


Components (II)

Temperature Probe: NTC Thermistor (YSI 46000 series) with FLUKE Hart 1504 Tweener

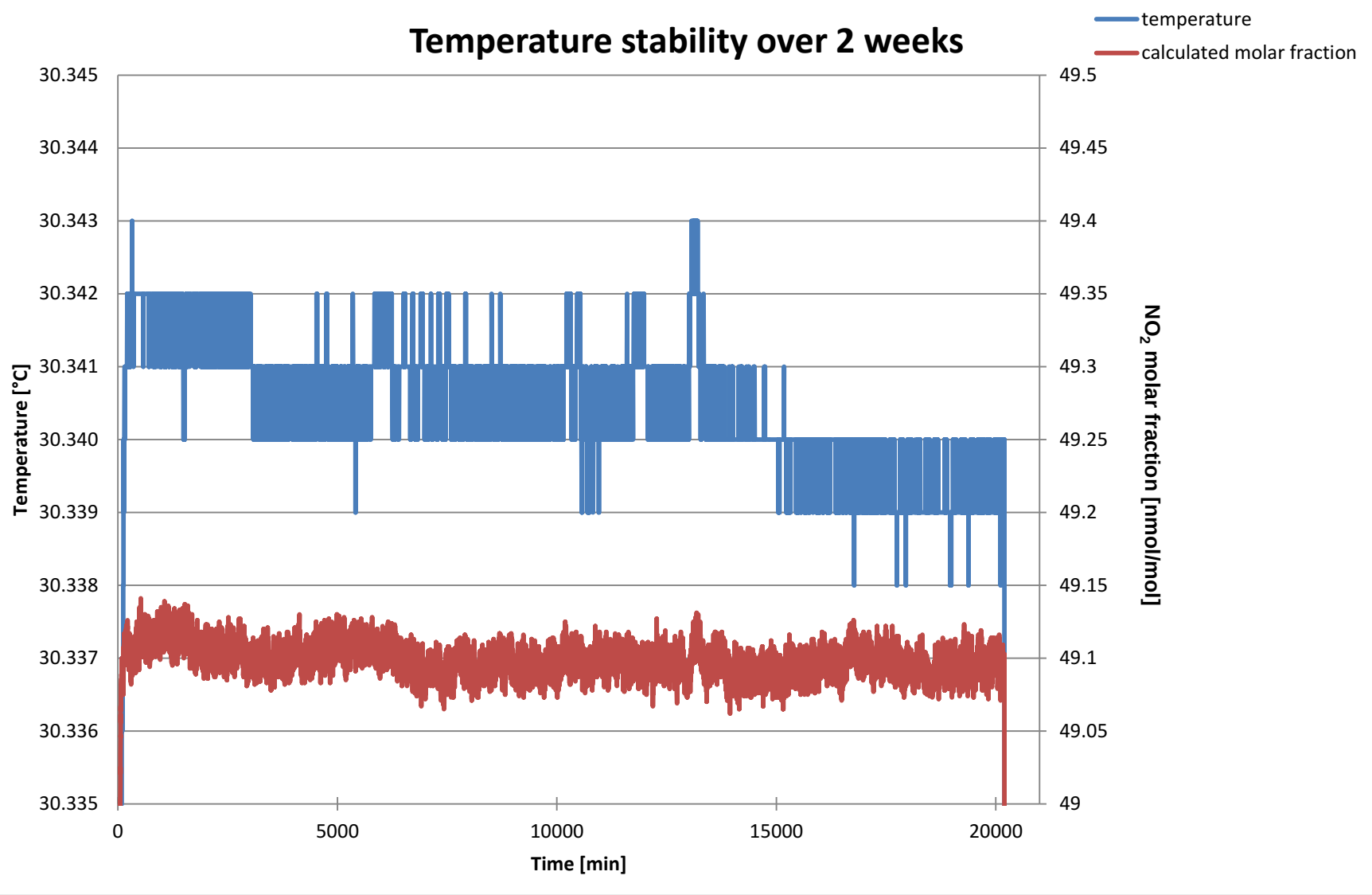
- Excellent stability and resolution
  - specs: probe < 0.01°C in 100 month ! when used < 50°C; < 0.002°C with resolution of 0.001°C
- Representative temperature measurement:
- Almost identical to the situation in Rubotherm balance





Mid term stability of temperature

Temperature stability over 2 weeks NO2-generation



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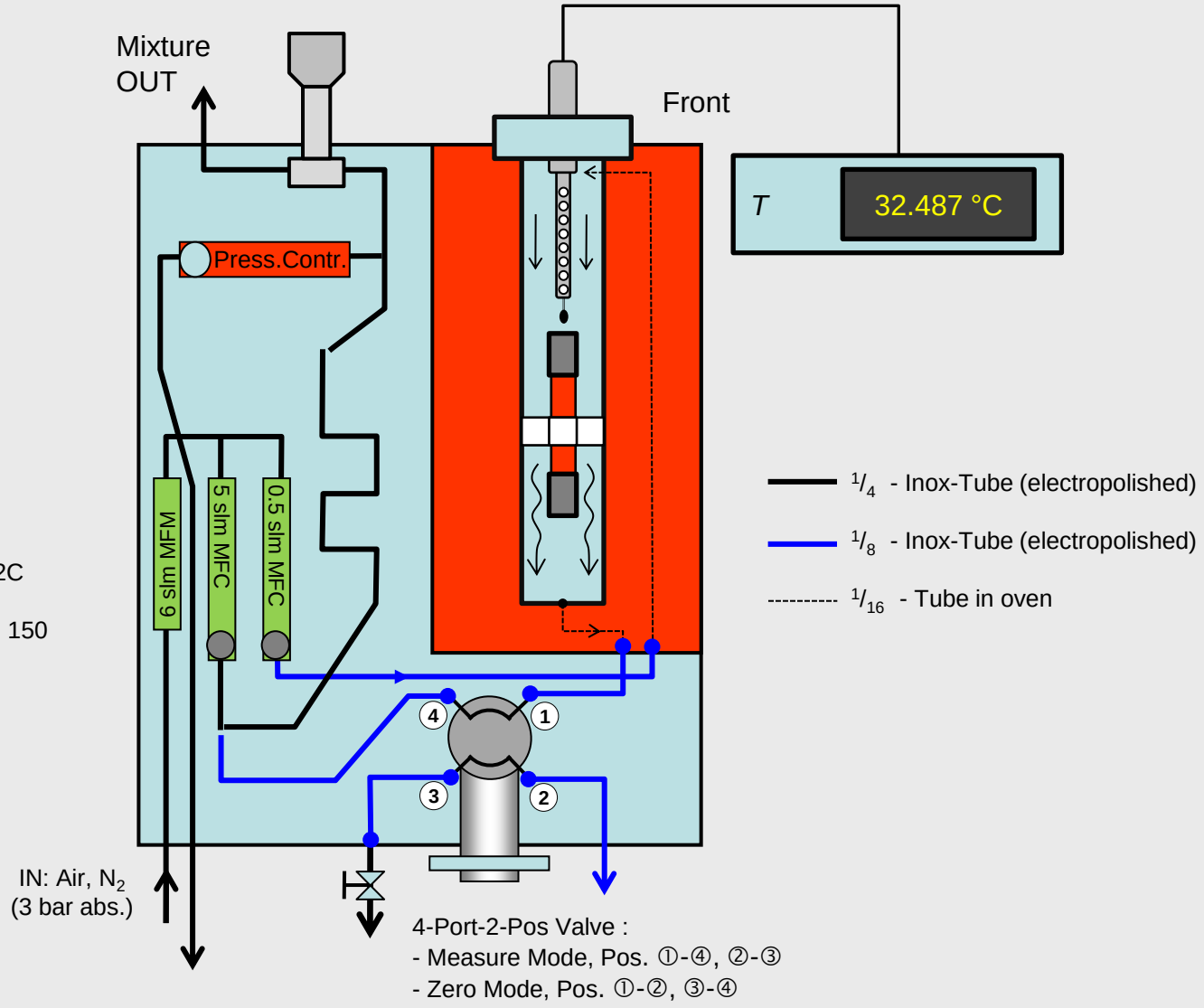


Components (III)

Flow devices: Vögtlin red-y smart series based on CMOSens by Sensirion:

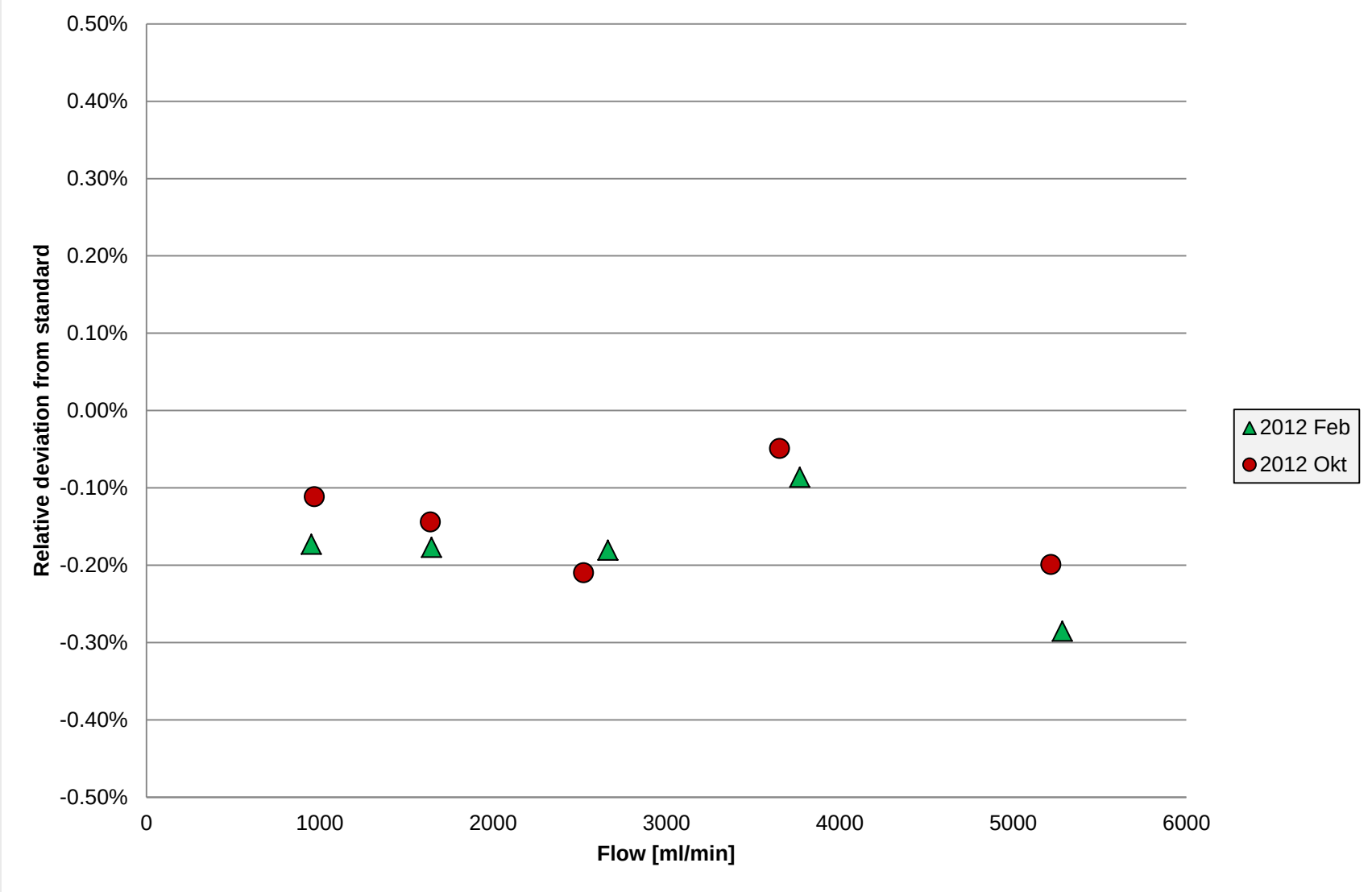
- 1 meter for total flow measurement (600 to 6000 ml/min)
- 1 controller for chamber flush flow (normally 300 ml/min)
- 1 controller for dilution flow (500 to 5000 ml/min)
- 1 back pressure controller Bronkhorst for isolation from atmospheric pressure

Individual calibration for purified air or nitrogen

Stability of total flow <0.05 %

Reproducibility <0.1 % over 0.5 year

Long-term flow reproducibility



Components (IV)

Permeation devices:

Choice: VICI wafer with permeated mass flow of ~ 200 ng/min

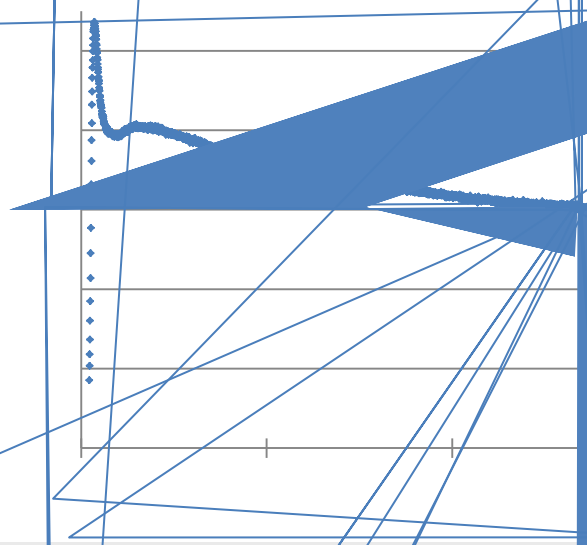
Same producer as for CCQM-K74 (purity issue could be similar)

Purity declared by manufacturer 99.8 %

Ratio between permeation surface and reservoir volume is ideal for long term use

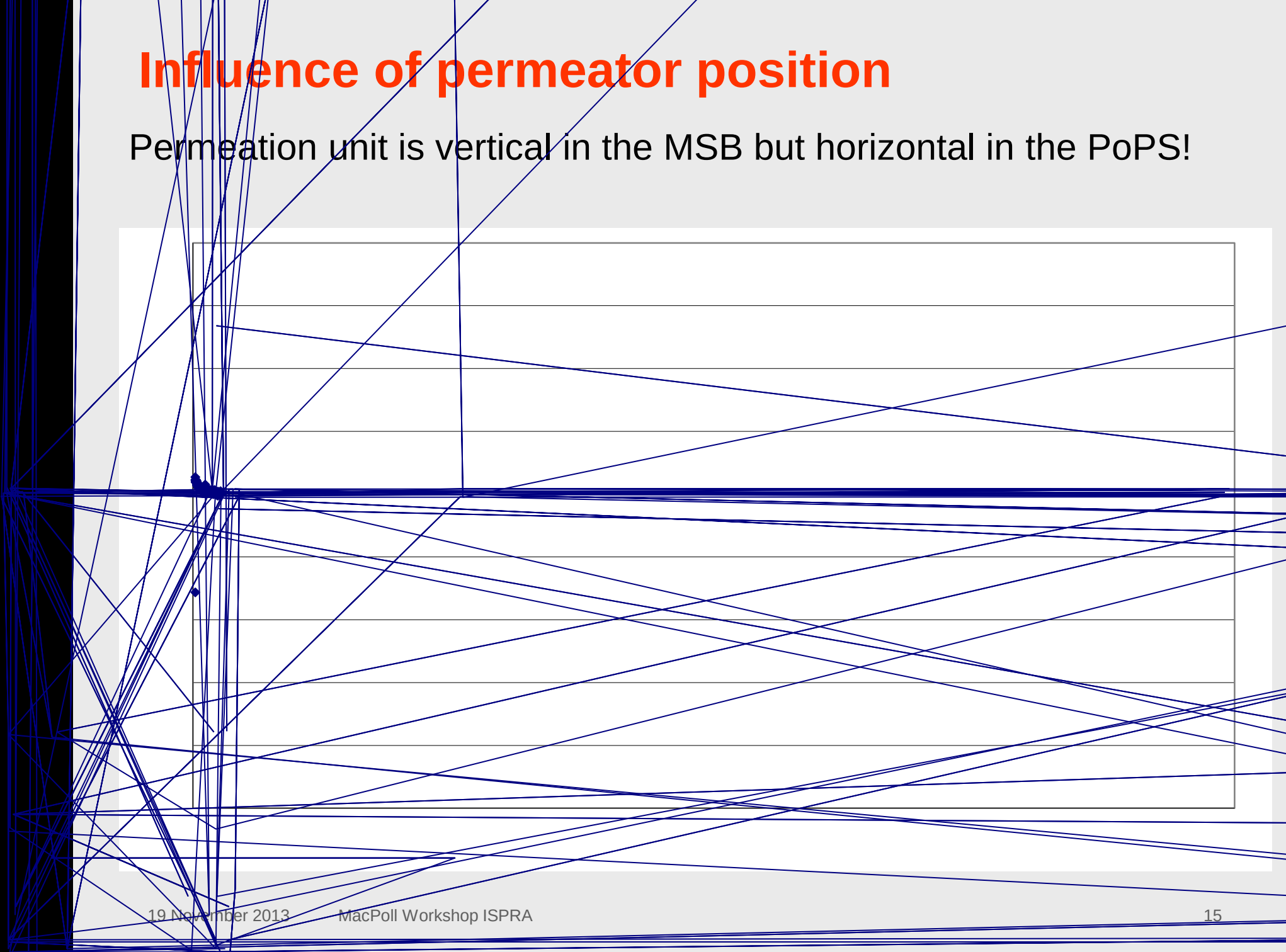
Optimisation of equlibration and calibration procedure.

Permeation unit stabilisation



Influence of permeator position

Permeation unit is vertical in the MSB but horizontal in the PoPS!



Components (V)

Additional Features:

Software on Linux PC

- Fully transparent calibration parameters for input quantities
- Documentation of generation by saving ASCII-file via USB
- Automatic calculation and setting of flows as a function of chosen amount of substance fraction.
- Instant calculation of permeated mass flow as a function of measured temperature.

2-way valve for mixture mode and zero mode

External nitrogen supply for short transportation

Traceability issues

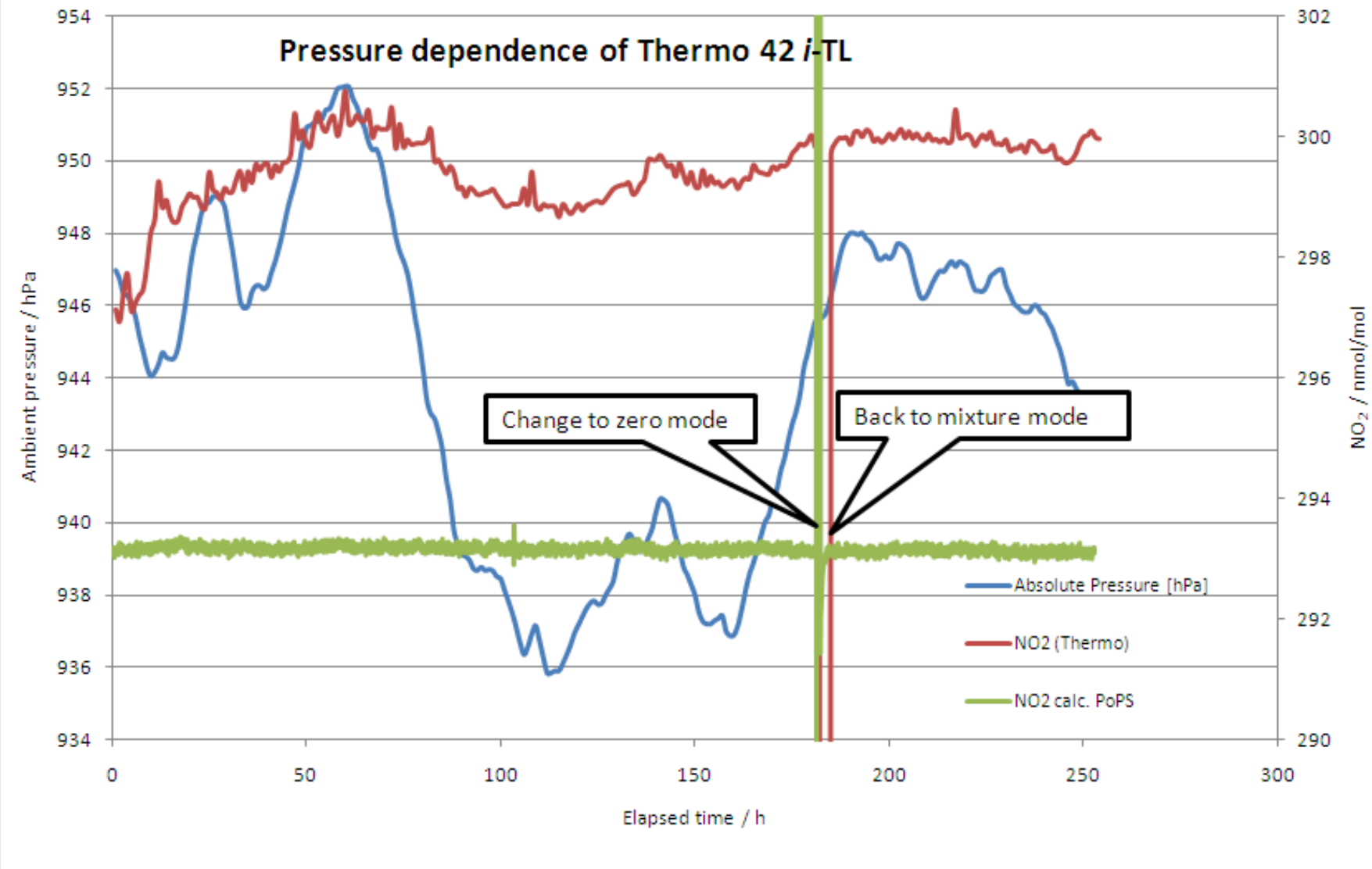
Traceability of input quantities:

Permeated mass flow:	METAS Mass lab (CMCs) METAS time server ntp
Dilution volume flow:	METAS Gas analytical lab
Temperature:	METAS Thermometry lab
Purity of dilution gas:	METAS gas analytical lab without CMCs (DL of Thermo 42 i-TL)
Purity of NO_2 in wafer:	Declaration of manufacturer. Experience of BIPM-K74 with same manufacturer (different permeation device).

Stability of NO_2 (reaction with H_2O, other reactants ???)

Mid term stability of Thermo 42*i*-TL

with ambient pressure change



21 stations comparison

Long term stability from Cercl'air comparison:

21 stations during summer 2011 visited for 1 week each
Travelling standard PoPS for NO_2 comparison.
Monitoring of PoPS with Horiba APNA 370 (adjusted with NO-diluted and GPT)
Horiba readings when using PoPS: 46.96 ± 0.32 nmol/mol
or σ = 0.68 % rel.
Ref.value PoPS (x ± U): 48.33 ± 0.72 nmol/mol (1.5 % rel.)

Next step: The MacPoll Project

Generation of NO2-mixtures at ppb-level

Travelling of PoPS in 4 countries during 8 months

Measurement of generated mixtures by all participating institutes

Results coming in the next presentation