Metrology for Climate Relevant VOCs

Generation of dynamic reference gas mixtures – traceability and uncertainty

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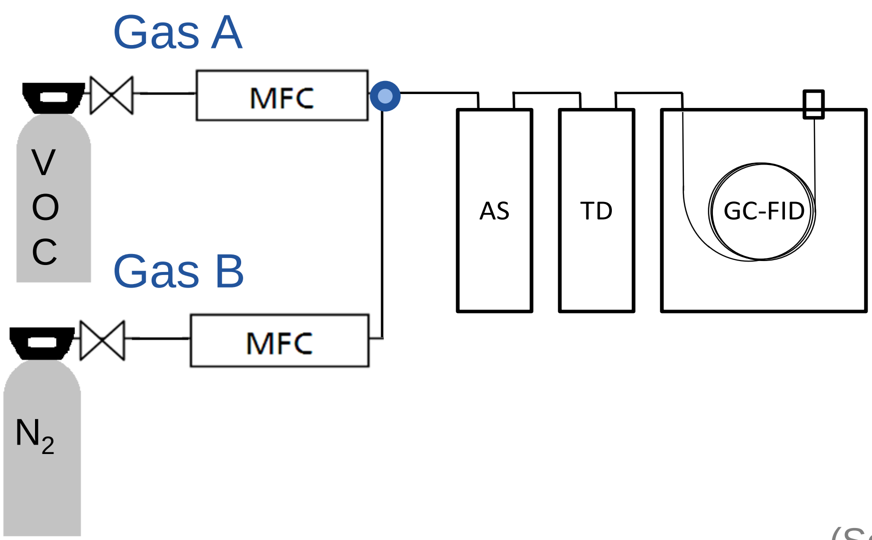
The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

ISO 6145 – dynamic methods

Preparation of calibration gas mixtures using dynamic methods, which relay in flow rates (gas A introduced at a known constant volume or mass flow rate into a known constant flow rate of gas B).

Dynamic dilution of a high fraction reference gas mixture with mass-flow controllers (MFCs)



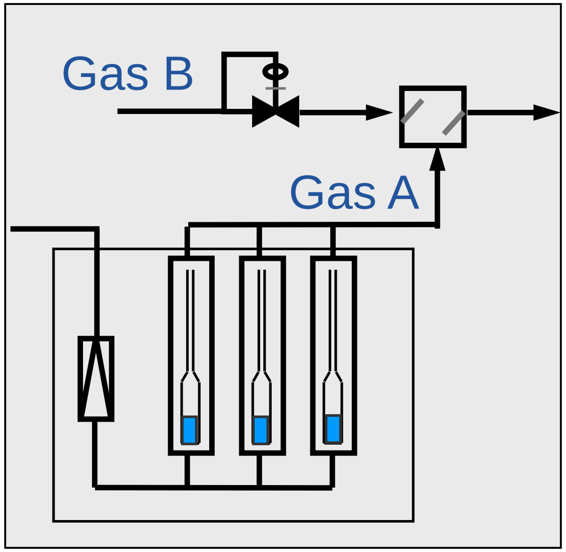
(Source: courtesy of VSL)

ISO 6145 – dynamic methods

Main dynamic methods

- Piston pumps
- Continuous injection
- Capillary
- Critical orifices
- Thermal mass-flow controllers
- Diffusion
- Saturation
- Permeation
- Electrochemical generation

Diffusion



(Source: courtesy of VSL)

Permeation dynamic method: MSB

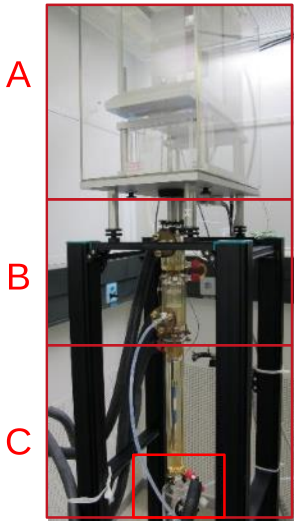

Magnetic suspension balance (MSB):

glass (left), metal (right)

- TA Instruments (former Rubotherm)
- Glass/metal (SilcoNert 2000 coated SS)

Permeation dynamic method: MSB

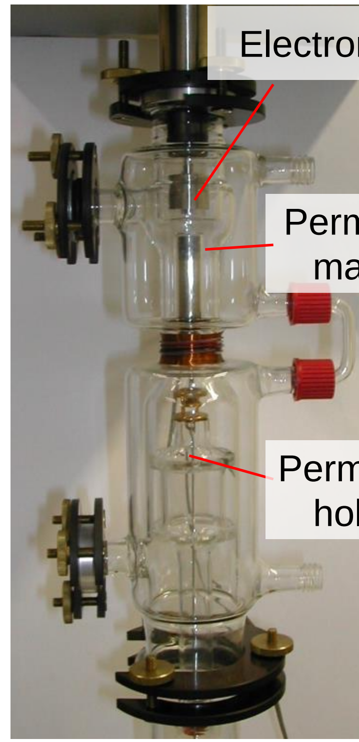
MSB elements



A

Reference weights

Electromagnet hanger

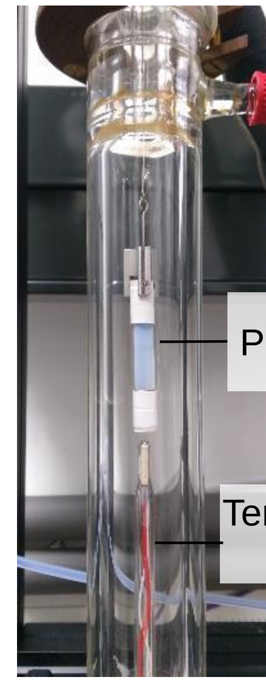


B

Electromagnet

Permanent magnet

Permeator holder



C

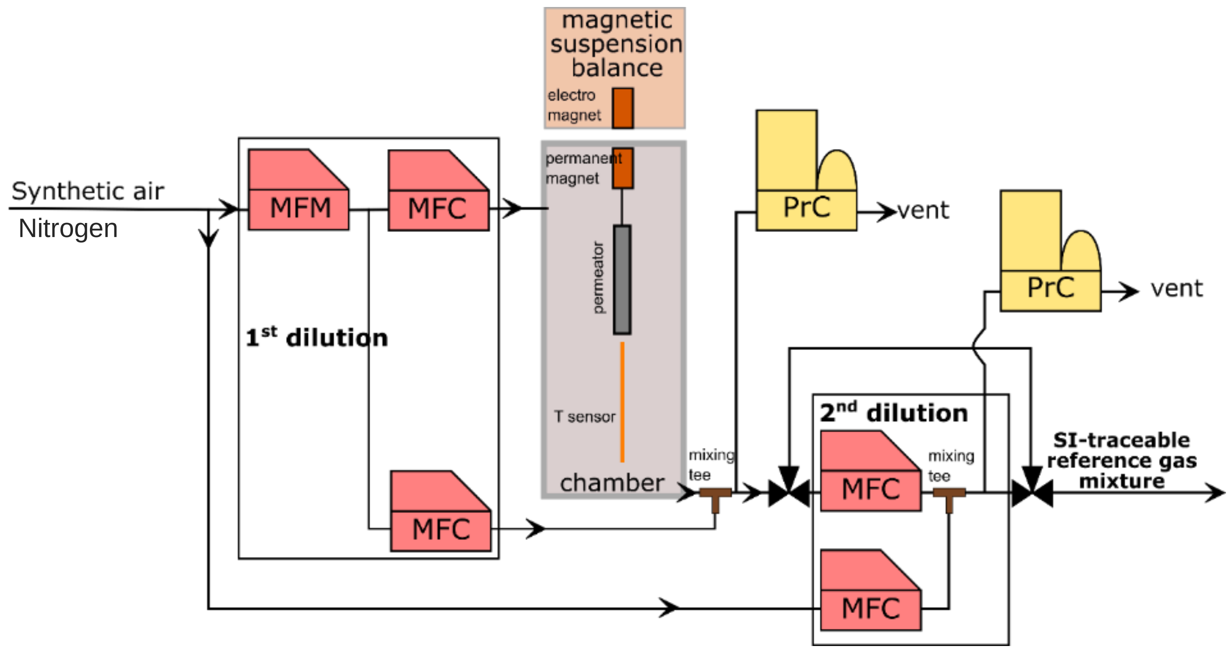
Permeator

Temperature probe

Mixing chamber

Permeation dynamic method: generation

Primary reference gas mixtures using a MSB (magnetic suspension balance)



Permeation dynamic method: generation

Step 1: Permeation unit calibration

VICI Metronics, FineMetrology, KIN-TEK,…

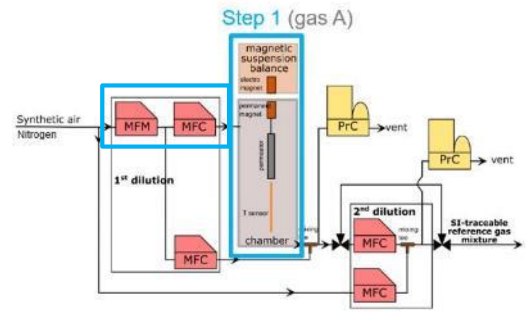
Main types: wafer, tube

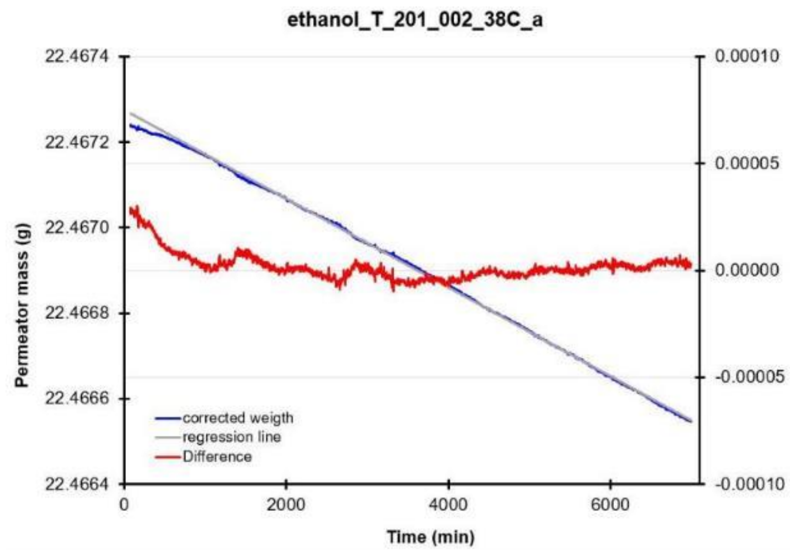
Controlled conditions (T, P, flow)

Calibration at different temperatures

> 5 days (1st calibration point)

> 3 days (for the rest; case-by-case)

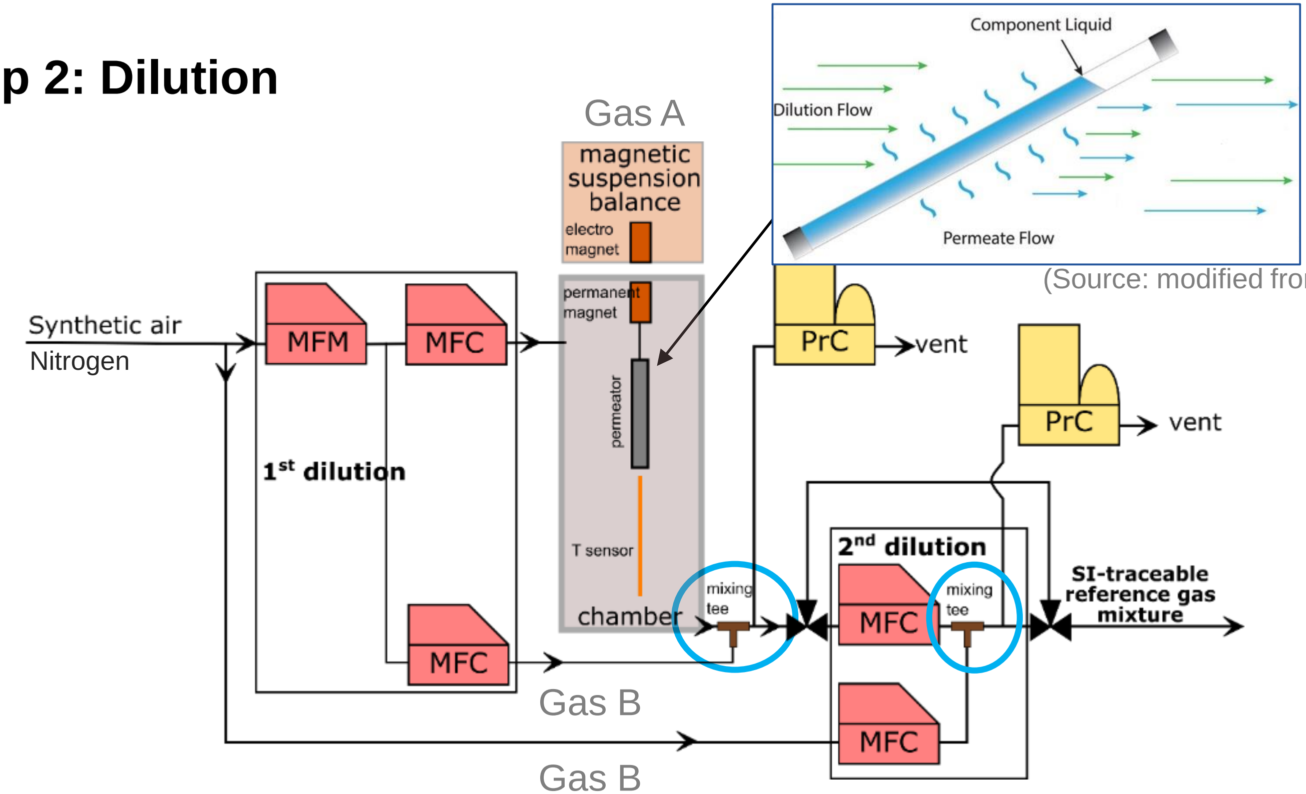




Permeation rate: $q_m = \frac{\Delta m}{\Delta t} = \frac{m_2 - m_1}{t_2 - t_1}$

Permeation dynamic method: generation

Step 2: Dilution

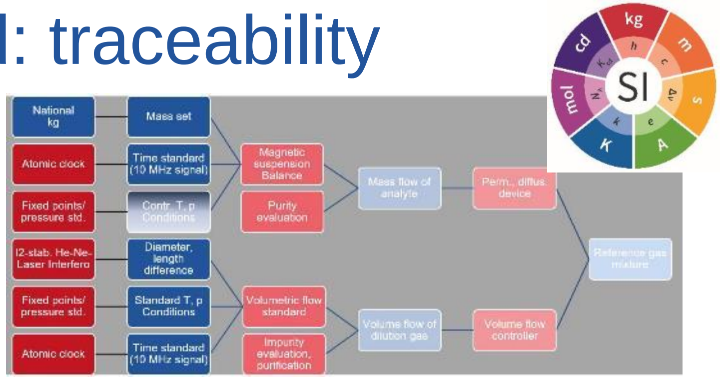


(Source: modified from KIN-TEK)

1st dilution: $X_{compound} = q_m \cdot purity \cdot \left(\frac{V_{m\,gas}}{M_{m\,comp}}\right) \cdot f_{dil1} + X_{res}$

2nd dilution: $X_{compound} = \left(q_m \cdot purity \cdot \left(\frac{V_{m\,gas}}{M_{m\,comp}}\right) \cdot f_{dil1} + X_{res}\right) \cdot f_{dil2} + X_{res}$

Permeation dynamic method: traceability

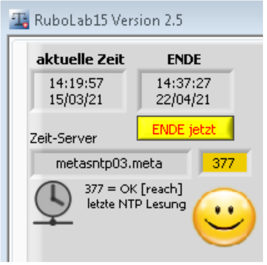


SI-traceable RGMs

Step 1: Permeation unit calibration

Permeation rate: $q_m = \frac{\Delta m}{\Delta t}$

Reference weights calibrated at METAS mass lab (→ **kg**)



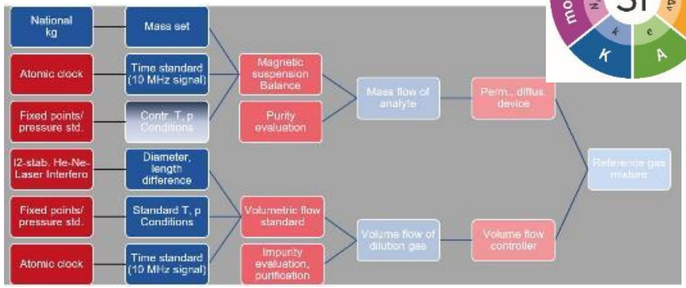
PC time synchronized through a Network-Time-Protocol (NTP) server with Swiss official time given by atomic clocks at METAS photonic, time and frequency lab (→ **s**)

Conditions in MSB chamber

Temperature probe calibrated at METAS thermometry lab based on International Temperature Scale of 1990 (→ **K**)

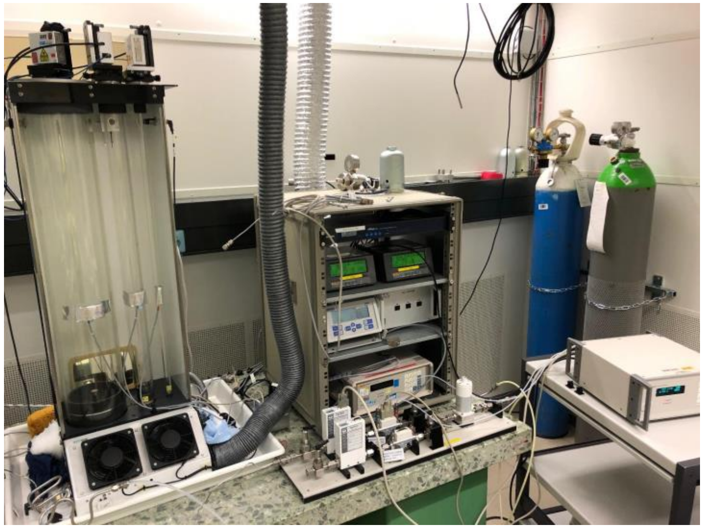
Pressure regulators calibrated at METAS force and pressure lab according norm ISO 17025 (Pa → **kg, m, s**)

Permeation dynamic method: traceability



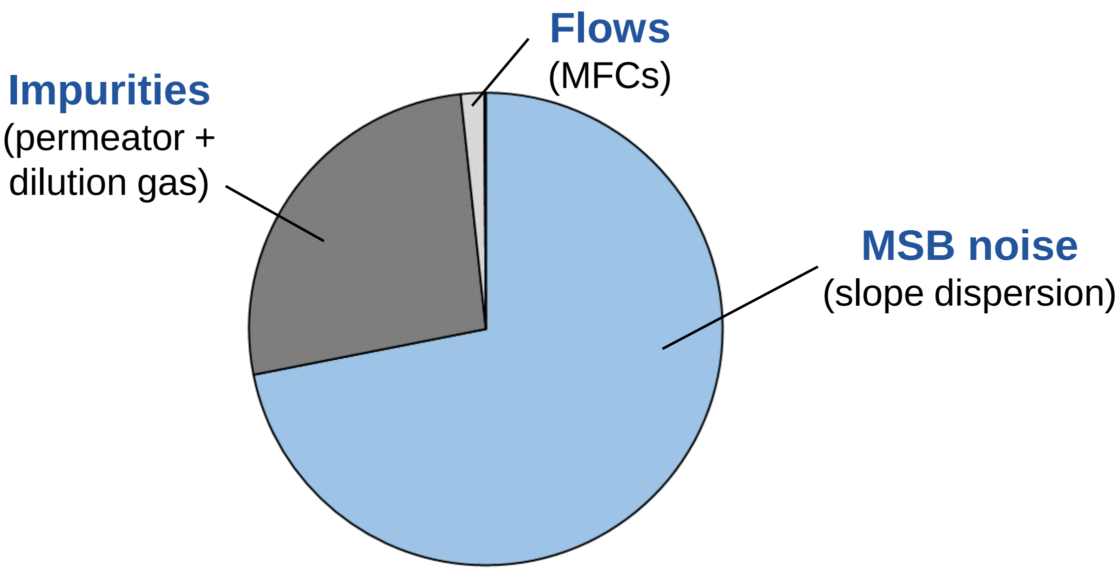
Step 2: Dilution

Mass-flow controllers ⇒ calibrated against METAS volumetric standard (→ **m, s**)

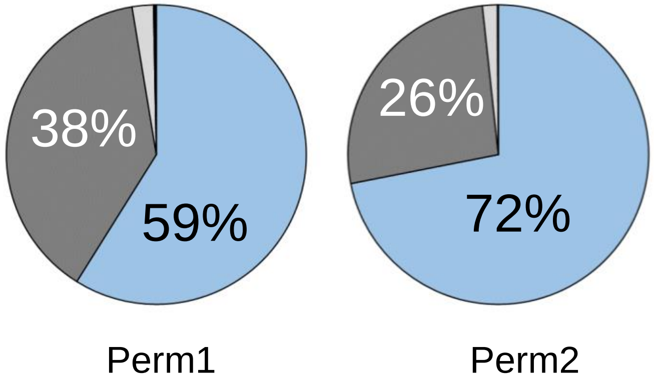


Permeation dynamic method: uncertainty

Main uncertainty sources of the generation of primary RGMs

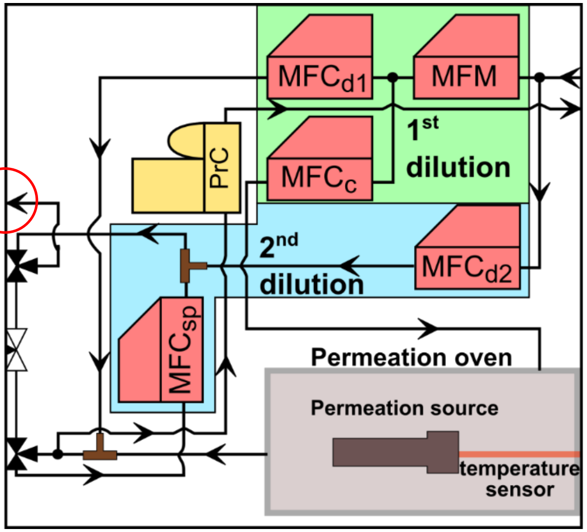


Permeator	Dilution steps	Amount fraction (nmol/mol)	Uncertainty (k = 2)
Perm1	2	31.3	1.9%
Perm2	1	345.9	1.6%

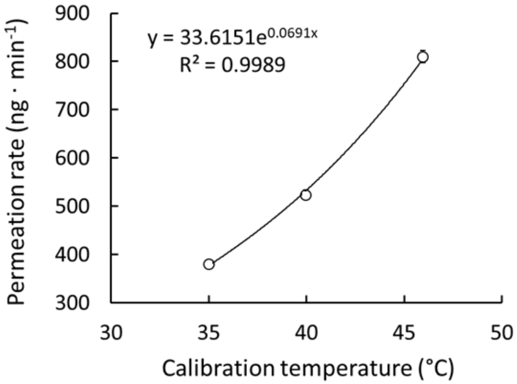


Portable RGM generators

Secondary reference gas mixtures using a portable generator (ReGaS)



$$q_m = a \cdot e^{b \cdot \mathrm{Temperature}}$$



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For more information, visit

www.metclimvoc.eu

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