PROMETH$_2$O

20IND06 PROMETH2O

Metrology for trace water in ultra-pure process gases

Training course

Morning - Golf Club Settimo, strada Cebrosa 166 Settimo Torinese (TO) - Italy

Afternoon - Nippon Gases, via Marie Curie 134/D Chivasso (TO) - Italy

Thursday 7th of March 2024

EMPIR EURAMET

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

Tips and tricks for traceable humidity measurements

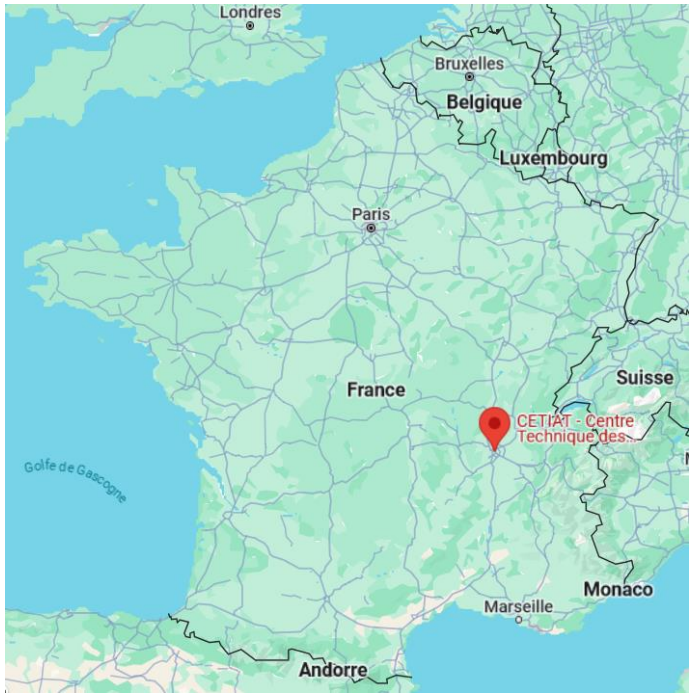
E. Georgin – eric.georgin@cetiat.fr

CETIAT

CETIAT

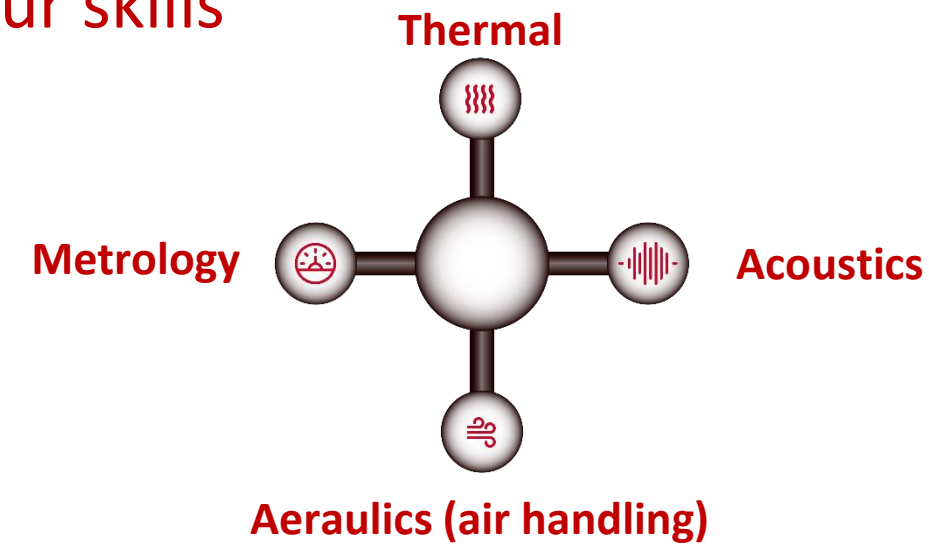
Technical centre for thermal and air handling industries

- Our legal structure
  - A technical industrial centre, created in 1960 on request from industrial firms, air handling and thermal system manufacturers dealing with the building sector and with industry in general (our members).
  - A public interest organization in charge of general interest missions.
  - An independent, impartial entity of private Law.



Technical centre for thermal and air handling industries

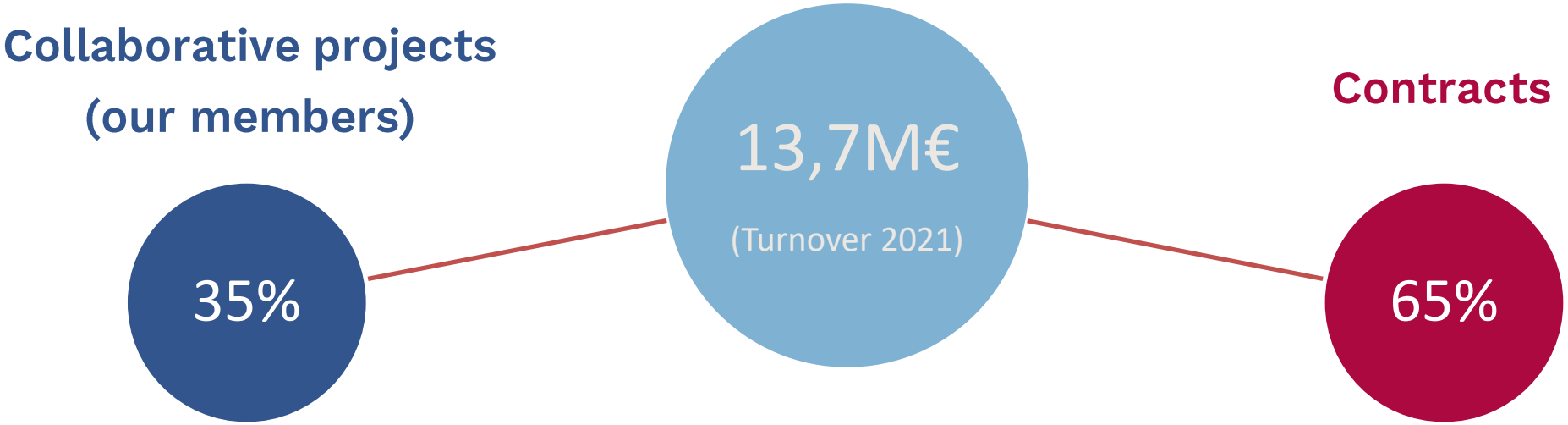
- Our skills



- Our fields of expertise
  - Ecological transition and energy efficiency
  - Decarbonization

- Our trades
  - Studies
  - Tests
  - Calibrations
  - Technical watch and dissemination of information
  - Training
  - Standardization
  - Enhancing indoor environments quality
  - Energy metrology

- Key figures



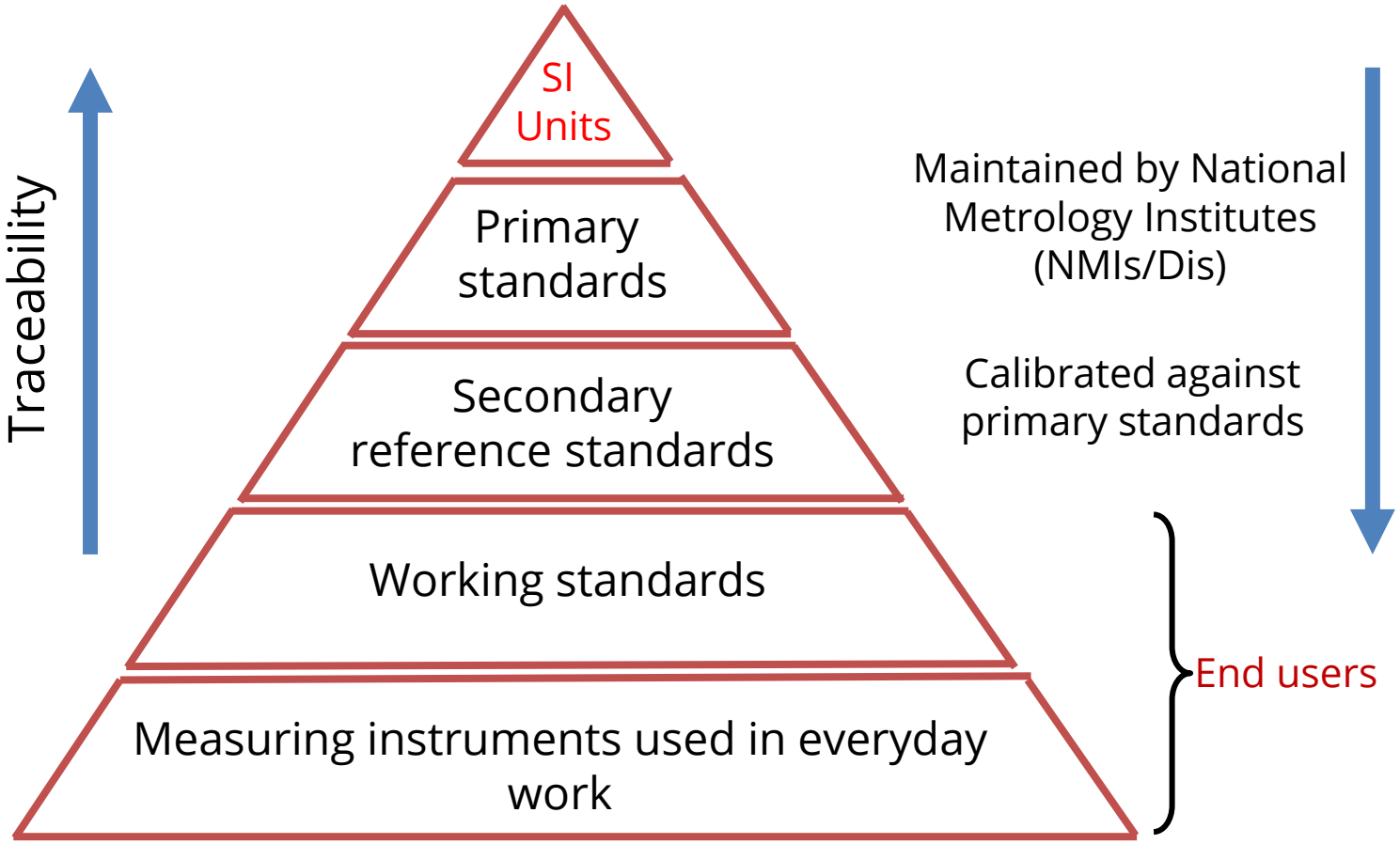
141 persons – 10 000 m² of labs
800 k€ invested – 50 test platforms – 6 calibration laboratories

- Symbols in humidity
  - No specific international standard for humidity measurement, nevertheless you may see within this presentation

- p : total pressure of a gas or gas mixture (Pa)
- e_s : saturation water vapour pressure (Pa)
- $e'(p, \theta_{d/f})$: water vapour partial pressure (Pa)
- θ_{dry} : dry (bulb) temperature (°C)
- $\theta_{d/f}$: dew/frost point temperature (°C)
- $U_{w/i}$: relative humidity (%rh)
- r : mass ratio ($g_{v.}/kg_{d.g.}$)
- $x_{v.}$: mole fraction (-)
- $f(p, \theta_{d/f})$: enhancement factor (-)
- $\emptyset$: mass flow (kg/s) or volumetric flow rate (m^3/s)
- M_x : molecular mass (g/mol)

Traceability

- General overview



International vocabulary of metrology – Basic and general concepts and associated terms (VIM) – JCGM 200/2012

2.41 (6.10)
metrological traceability

property of a **measurement result** whereby the result can be related to a reference through a documented unbroken chain of **calibrations**, each contributing to the **measurement uncertainty**

2.42 (6.10 Note 2)
metrological traceability chain
traceability chain

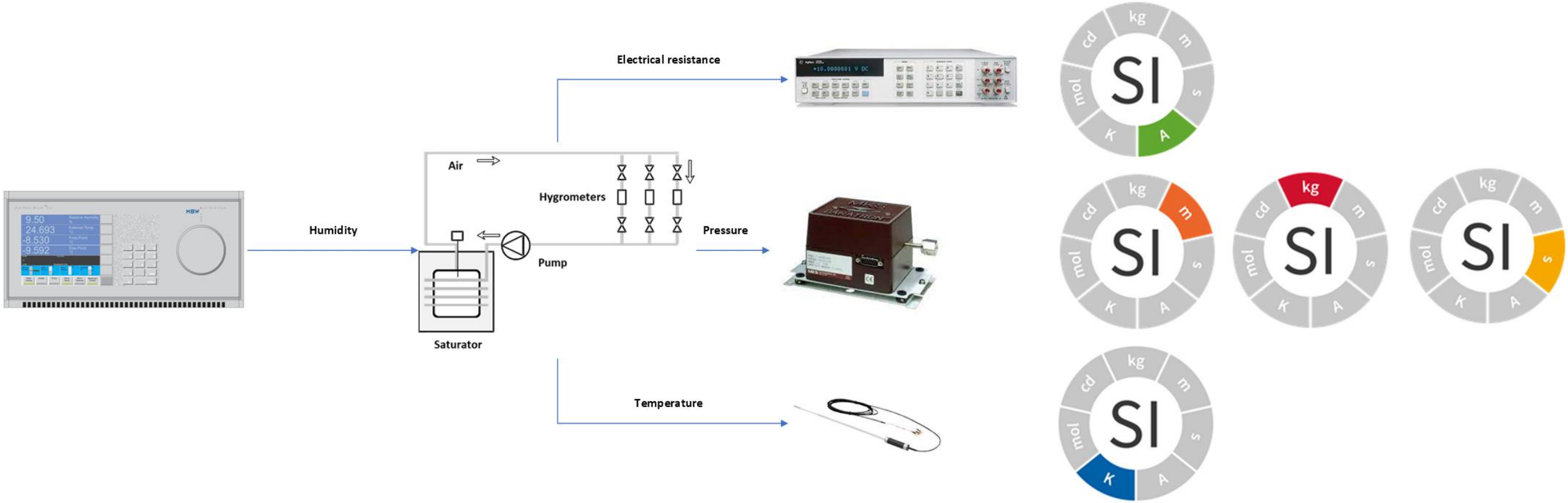
sequence of **measurement standards** and **calibrations** that is used to relate a **measurement result** to a reference

2.43
metrological traceability to a measurement unit
metrological traceability to a unit

metrological traceability where the reference is the definition of a **measurement unit** through its practical realization

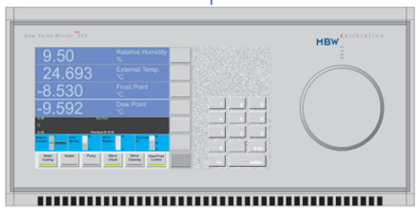
Traceability

- Traceability routes



- Traceability routes

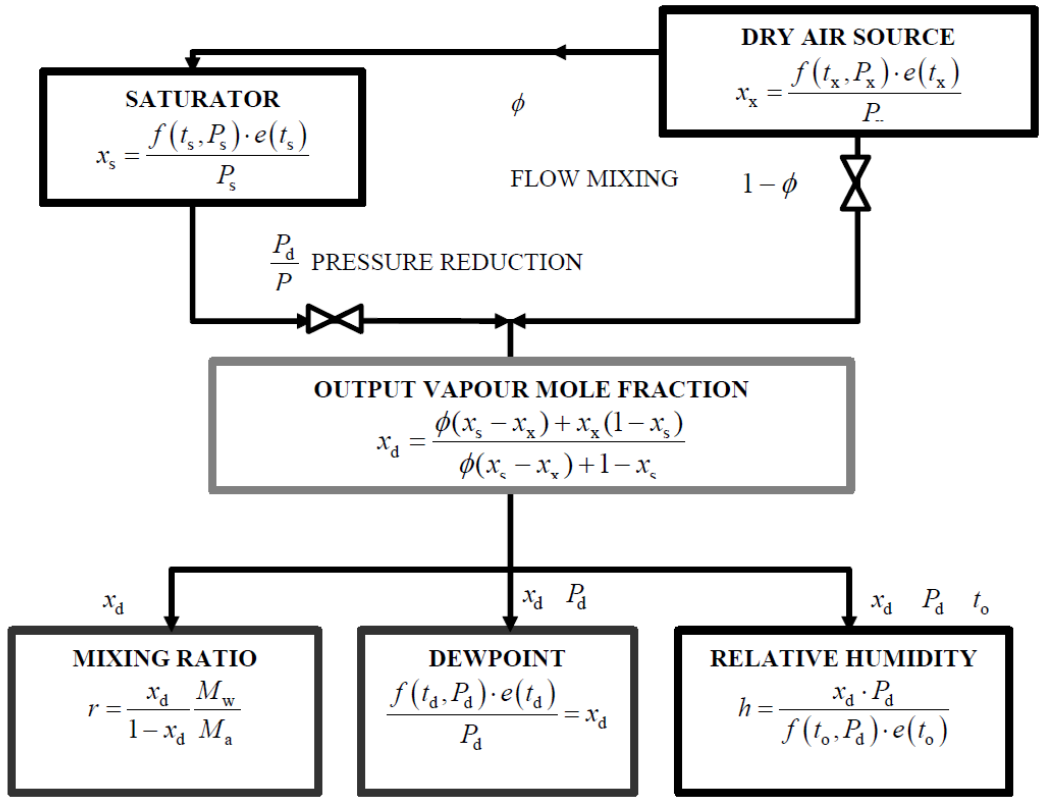
Electrical resistance



Pressure

Humidity realizations

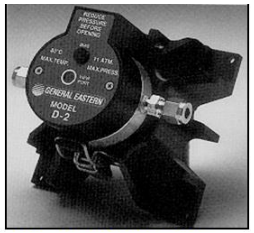
- Dew/frost point realizations
  - Mass and volume based
    - Permeation/diffusion tubes
    - 2 F or divided flow generator
  - Saturation based
    - 1 P, 1 T humidity generator
    - 2 P, 1 T humidity generator
  - “Hybrid ” (1 P , 2 P, 2 F)
  - Hygrometer + stable humid gas source



J. Lovell-Smith, Metrologia (2009)

Chilled mirror hygrometer

- Dew (frost) point temperature measurement : chilled mirror
- Additional measurements : dry temperature; pressure
  - Derived quantities could be calculated and displayed : relative humidity, water vapour partial pressure, mixing ratio, … etc …



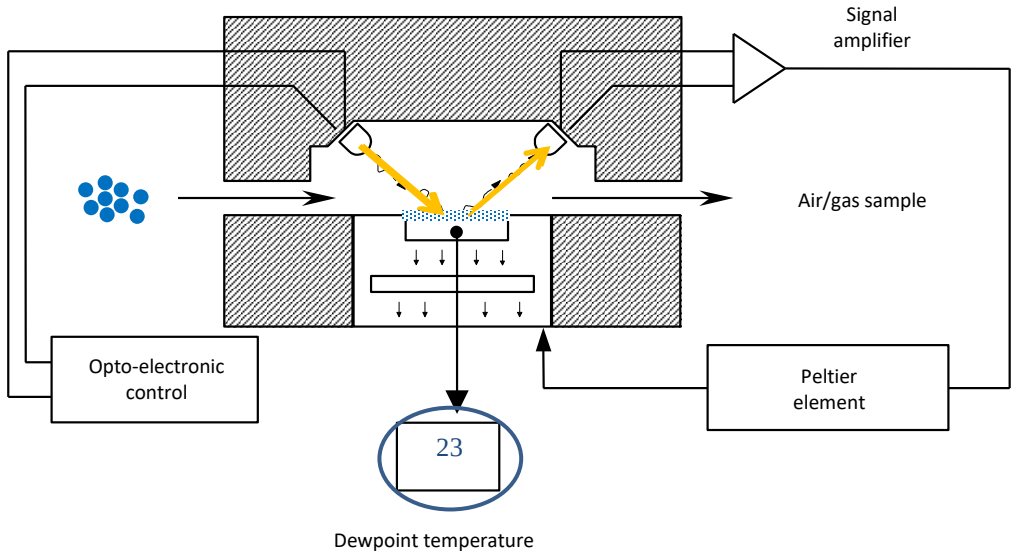
GE user's manual

MICHELL Instruments user's manual

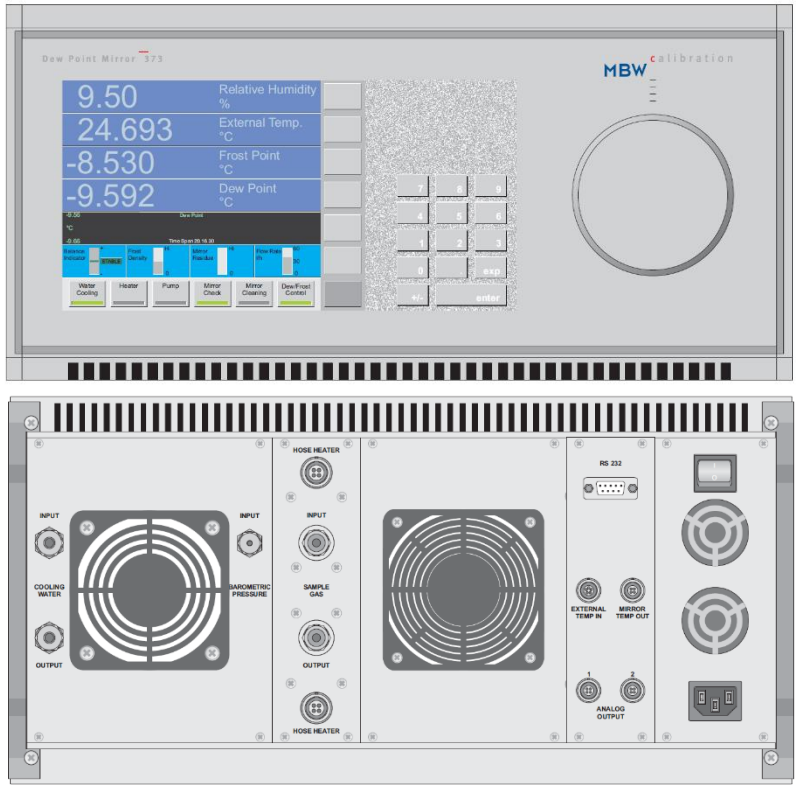
MBW user's manual

Chilled mirror hygrometer

- Dew (frost) point temperature measurement: chilled mirror
- Principle: detection and measurement at which condensation in the form of dew or frost is induced on a small mirror



- Measuring head
  - Integrated head with sampling



External cooling possible

MBW 373 user's manual

Michell Instruments S8000 integral user's manual

Chilled mirror hygrometer

- Measuring head
  - Integrated head with sampling

External cooling possible

GE D2
OPTISONDE user's manual

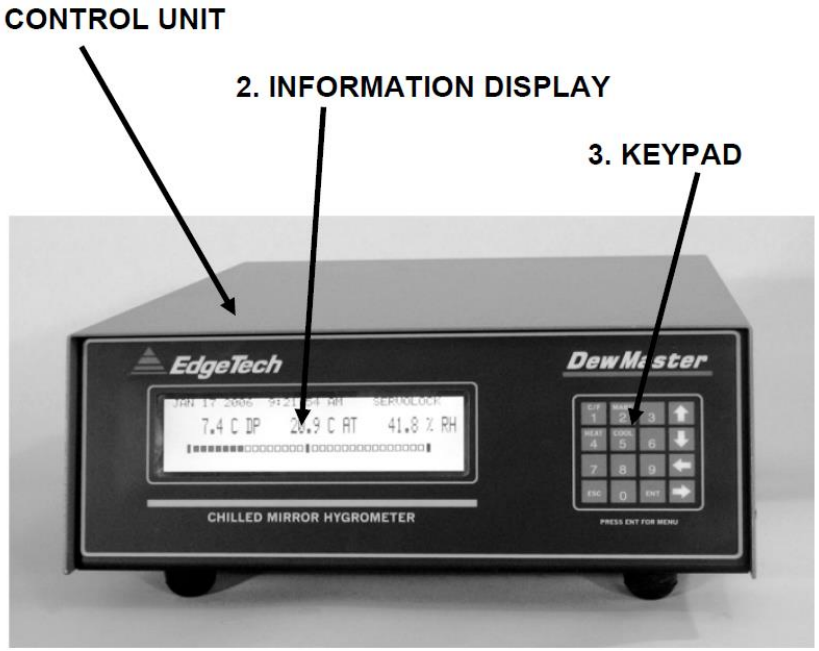
GE 1211H
OPTISONDE user's manual

GE 1311DR
OPTISONDE user's manual

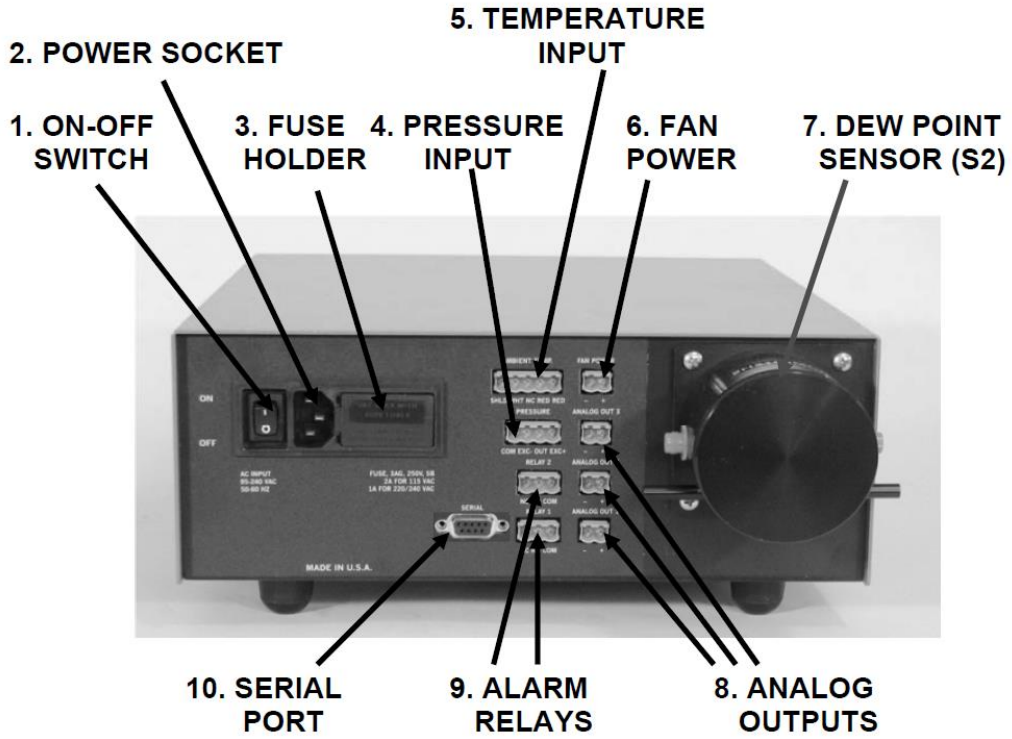
Chilled mirror hygrometer

- Measuring head
  - Integrated head with sampling

External cooling possible

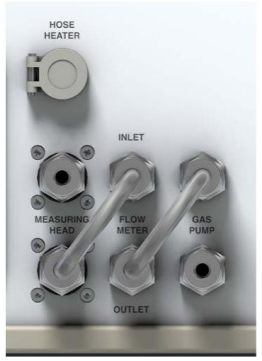


EDGETECH DEWMASTER user's manual

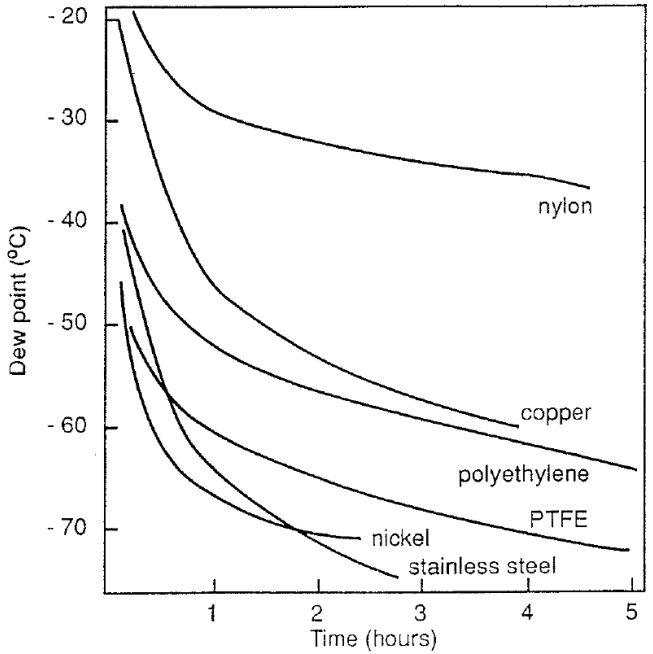


MBW DP30 user's manual

MBW 573 user's manual

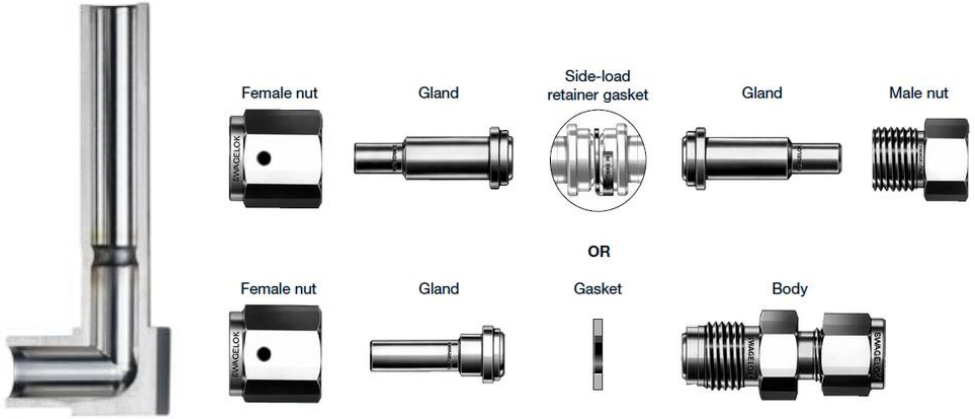


- Sampling
  - Internal vs external pump/pressure source
  - Heated hose / water trap
  - Leaks and dead volumes
  - Tubing materials



S. Bell, "A guide to the measurement of humidity", NPL, 1996

https://www.process-insights.com/products-3/products-industrial/chilled-mirror-hygrometers/573-model/



https://edmontonvalve.swagelok.com/blog/bid/325525/the-advantages-of-swagelok-vcr-and-vco-fittings

Chilled mirror hygrometer

- Measuring head
  - Remote head / immersion head

Michell Instruments
S8000 remote

GE 1111H
OPTISONDE user's man

MBW 473 user's manual

EDGETECH DewTrack II with remote sensor product overview

Chilled mirror hygrometer

- Measuring head
  - Hybrid sampling / immersion head

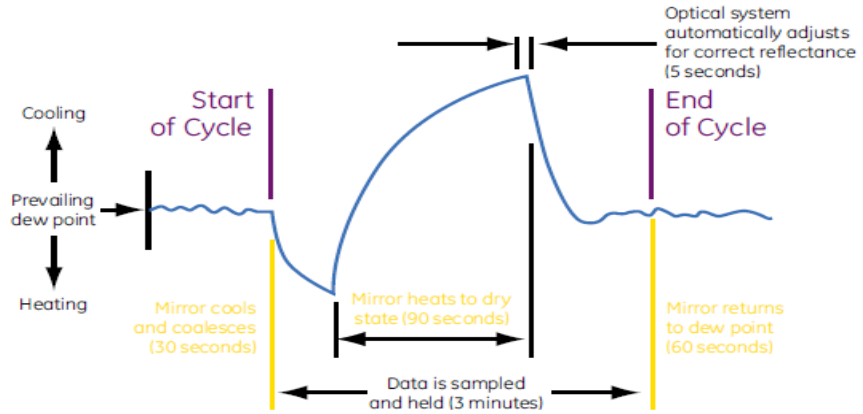
GE 1111H
OPTISONDE user's manual

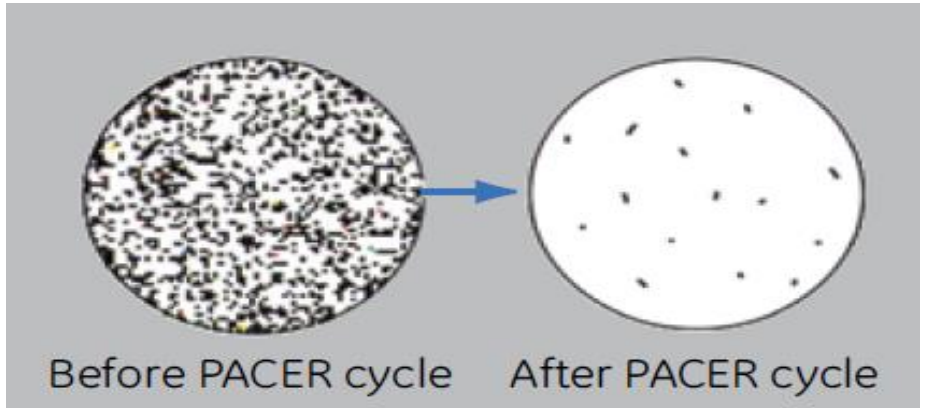
MBW 473 user's manual

Chilled mirror hygrometer … tips and traps

- Mirror contamination and cleaning
  - Manual
  - Cleaning function : PACER (Program Automatic Error Reduction), ABC (Automatic Balance Compensation), DCC (Dynamic Contamination Control), MCC (Mirror Check Control)

MBW 373 user's manual

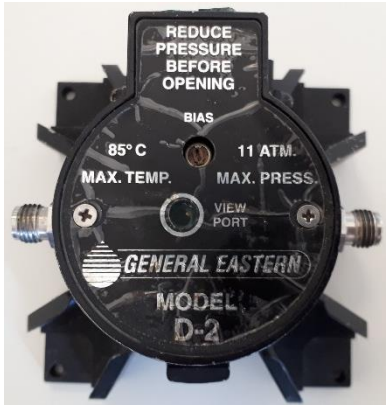




GE developed a patented contamination compensation scheme called PACER (Program Automatic Error Reduction)

Chilled mirror hygrometer … tips and traps

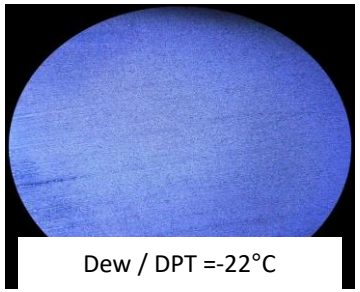
- Mirror adjustment and optical balance
  - Manual

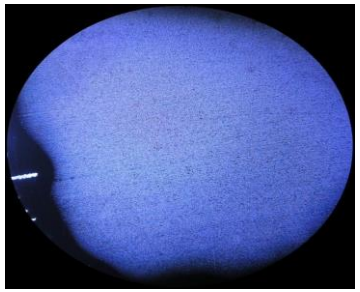


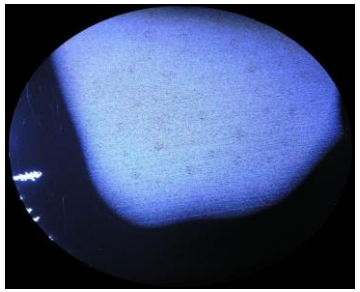
  - Function : PACER (Program Automatic Error Reduction), ABC (Automatic Balance Compensation), DCC (Dynamic Contamination Control), MCC (Mirror Check Control)

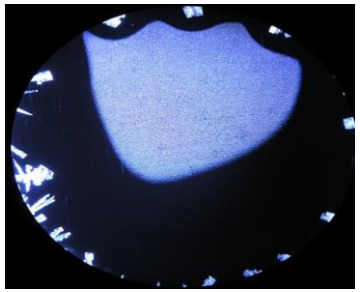
Chilled mirror hygrometer … tips and traps

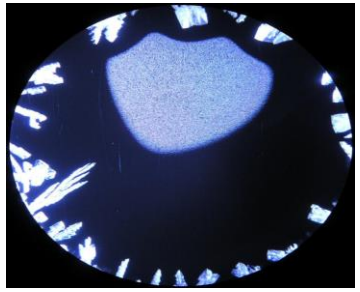
- Condensate formation : ice vs subcooled water

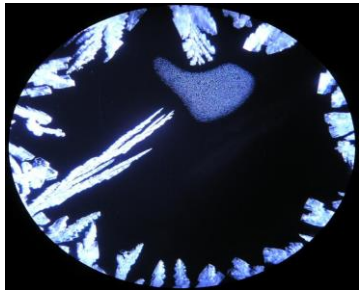

Dew / DPT =-22°C

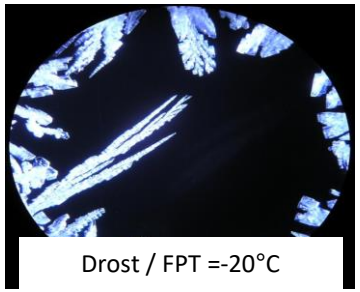


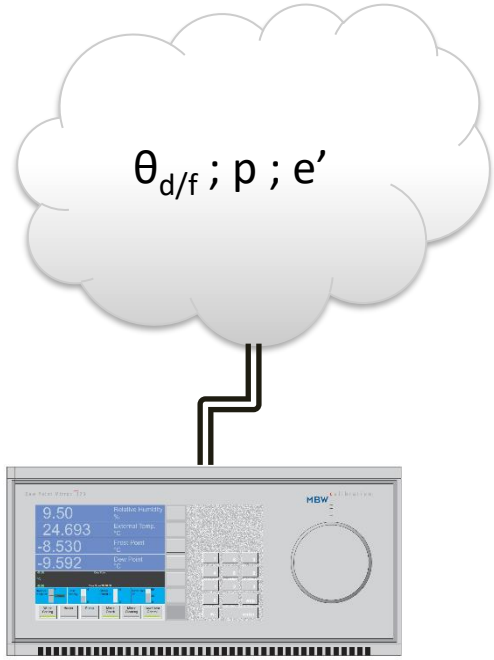









Drost / FPT =-20°C

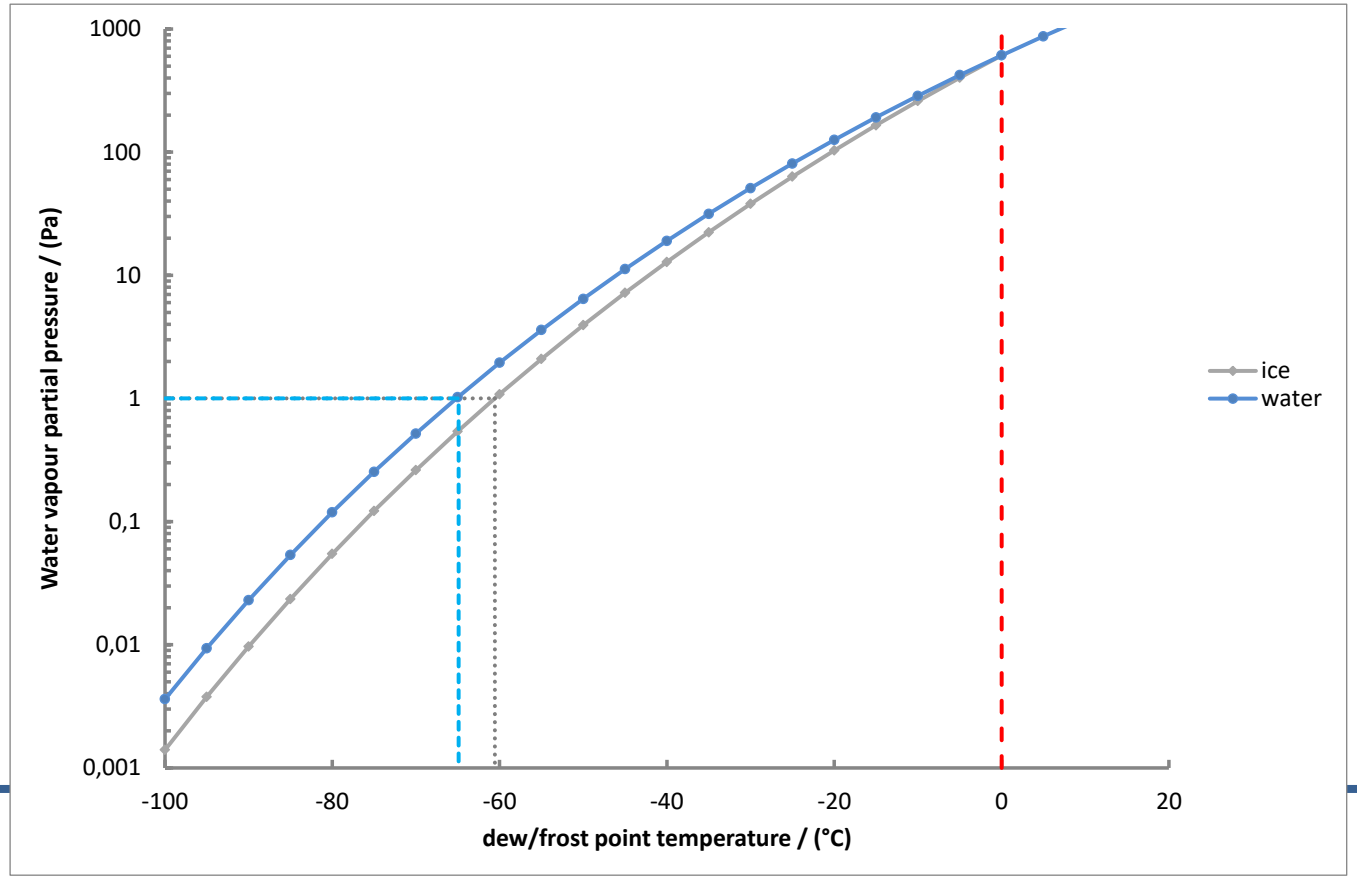


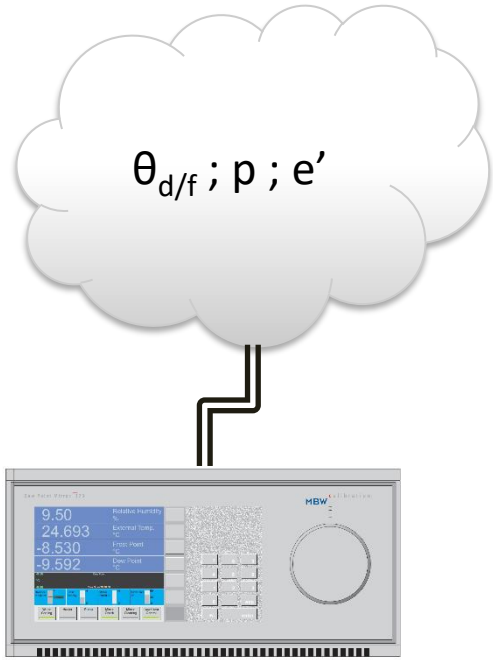
With the courtesy of Daniel MUTTER - MBW

Chilled mirror hygrometer … tips and traps

Mixing ratio r (kg/kg)	Dew point temperature θ_d (°C)	Frost point temperature θ_f (°C)	*Deviation* $\theta_f - \theta_d$ (°C)
$3{,}0 .10^{-3}$	-2,68	-2,37	0,31
$2{,}0 .10^{-3}$	-7,99	-7,10	0,89
$1{,}0 .10^{-3}$	-16,58	-14,84	1,74
$0{,}5 .10^{-3}$	-24,57	-22,14	2,43

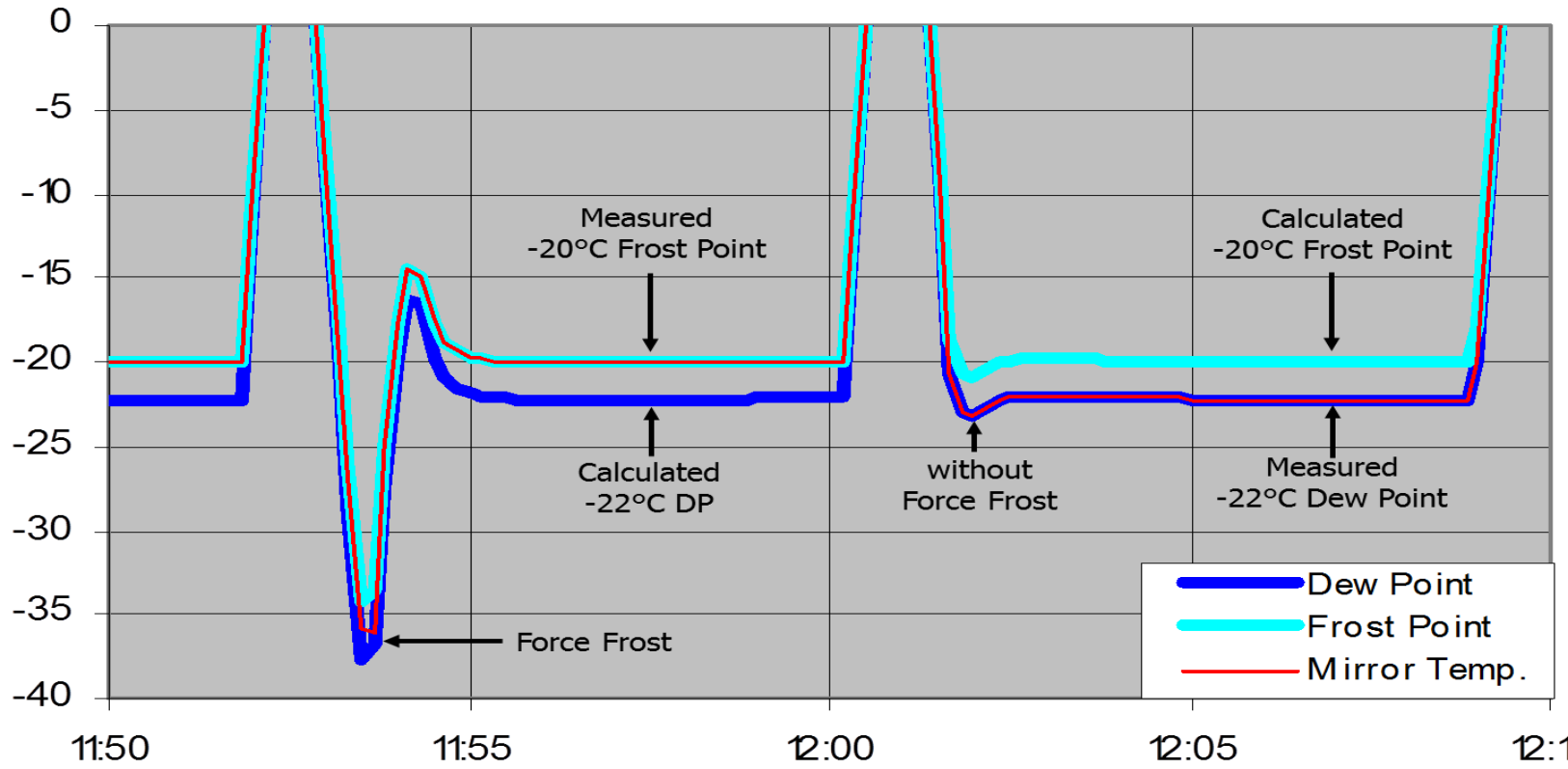
- Condensate formation : ice vs subcooled wate





Chilled mirror hygrometer … tips and traps

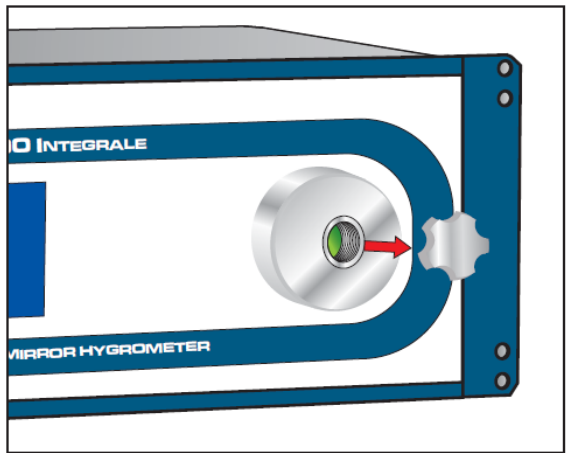
- Condensate formation : ice vs subcooled water
  - FORCE FROST function (MBW) / FAST (Michell Instrument)

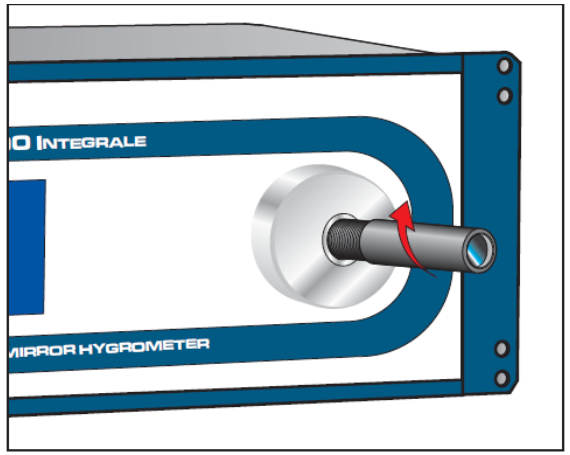


With the courtesy of Daniel MUTTER - MBW

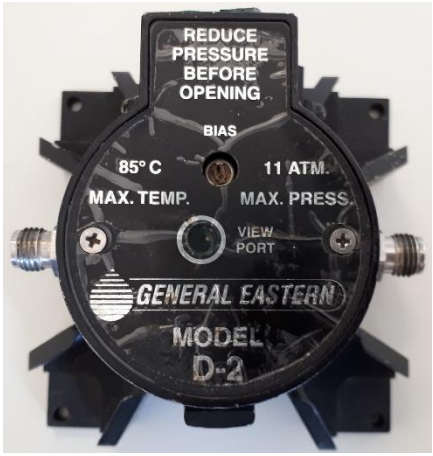
Chilled mirror hygrometer … tips and traps

- Condensate formation : ice vs subcooled water
  - Direct observation of the mirror





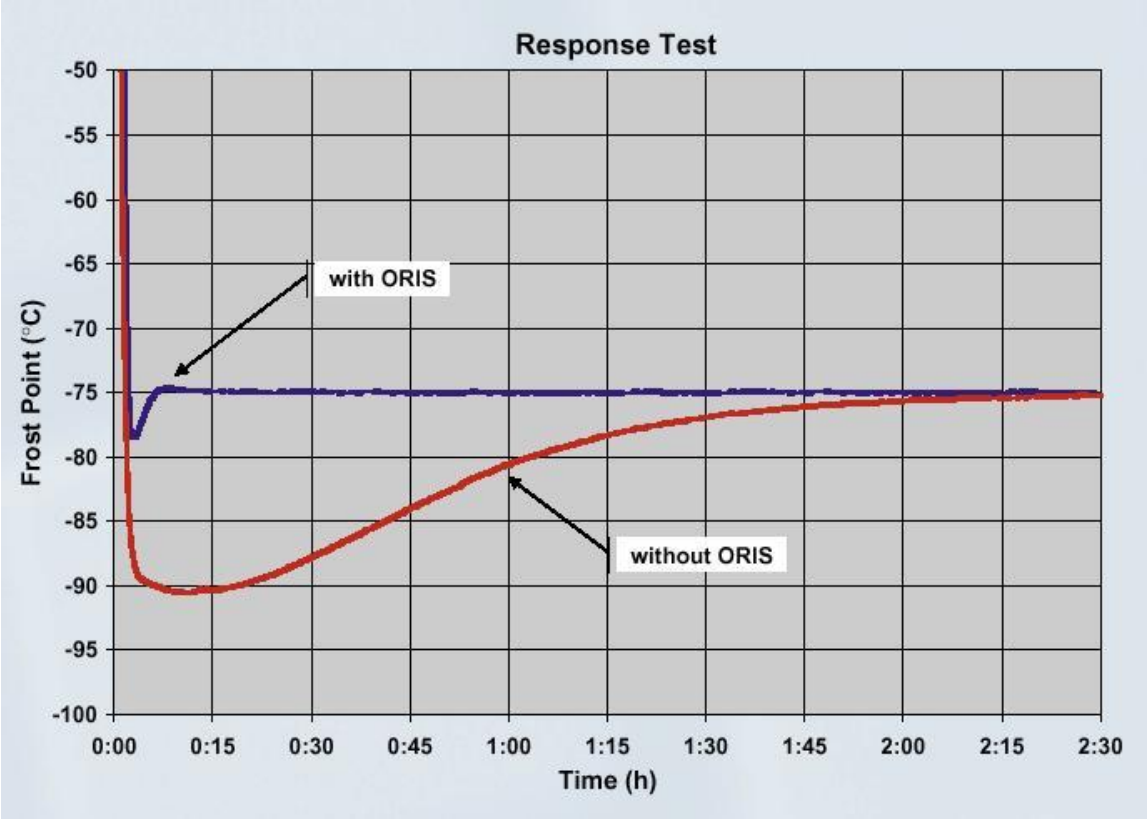
Michell Instruments S8000 integral user's manual



Chilled mirror hygrometer … tips and traps

With the courtesy of Daniel MUTTER - MBW

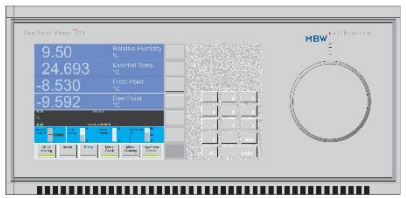
- Condensate formation : ice
  - ORIS - Optimal Response Injection System
    - ORIS allows faster measurements at mirror temperatures below -60°C
    - Accelerates formation of the thin layer of frost on mirror surface



Chilled mirror hygrometer … tips and traps

- Sampling, pressure drop and dew point temperature measurement

Measuring head			Medium			Pressure drop	Correction dew point temperature
Total pressure p_1 (Pa)	Dew point temperature θ_{d1} (°C)	Partial pressure e'_1 (Pa)	Total pressure p_2 (Pa)	Partial pressure e'_2 (Pa)	Dew point temperature θ_{d2} (°C)	p_2-p_1 (Pa)	θ_{d2}-θ_{d1} (°C)
97 000	14,5	1 662	100 000	1 713	15,0	3 000	0,5
99 700	14,95	1 708	100 000	1 713	15,00	300	0,05



θ_{d1} ; p_1 ; e'_1

$\Delta p = p_2 - p_1 = 3000$ Pa

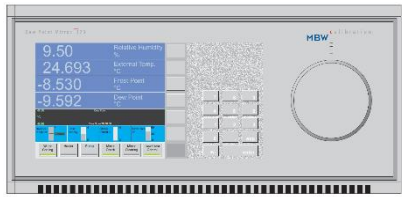
θ_{d2} ; p_2 ; e'_2

Chilled mirror hygrometer … tips and traps

- Sampling, pressure drop and dew point temperature measurement

Measuring head			Medium			Pressure drop	Correction dew point temperature
Total pressure p_1 (Pa)	Dew point temperature θ_{d1} (°C)	Partial pressure e'_1 (Pa)	Total pressure p_2 (Pa)	Partial pressure e'_2 (Pa)	Dew point temperature θ_{d2} (°C)	p_2-p_1 (Pa)	θ_{d2}-θ_{d1} (°C)
97 000	14,5	1 662	100 000	1 713	15,0	3 000	0,5
99 700	14,95	1 708	100 000	1 713	15,00	300	0,05

$$\frac{e'_2(p_2,\theta_{d,2})}{p_2} = \frac{e'_1(p_1,\theta_{d1})}{p_1}$$



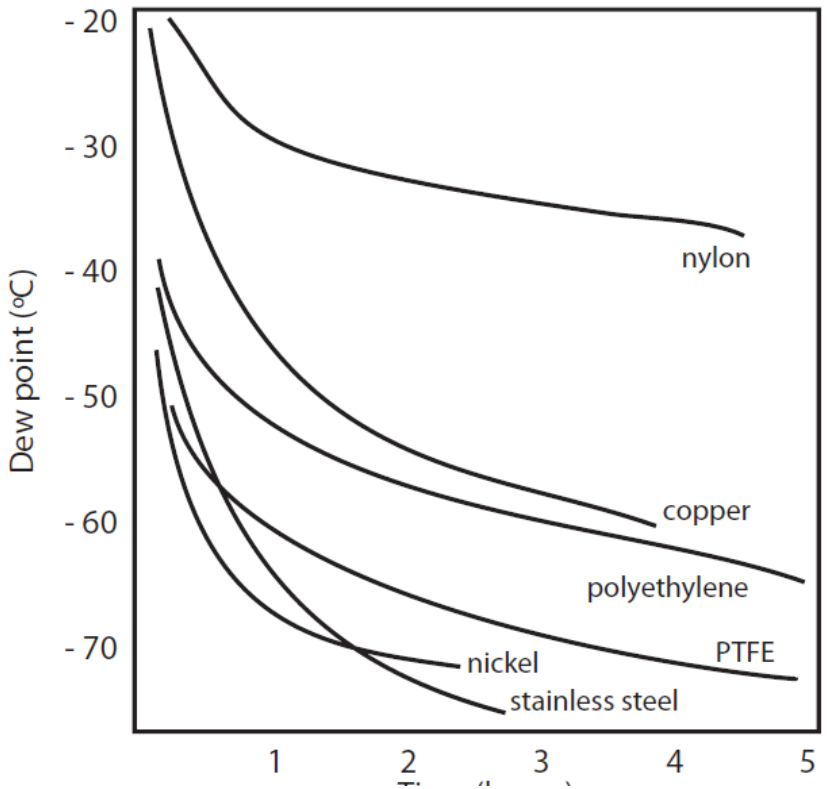
θ_{d1} ; p_1 ; e'_1

$\Delta p = p_2 - p_1 = 300$ Pa

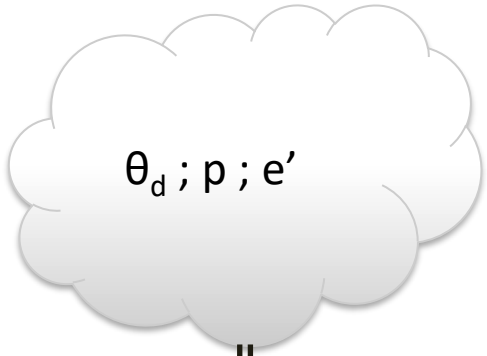
θ_{d2} ; p_2 ; e'_2

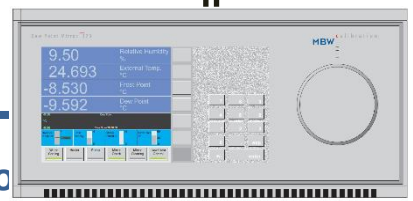
Chilled mirror hygrometer … tips and traps

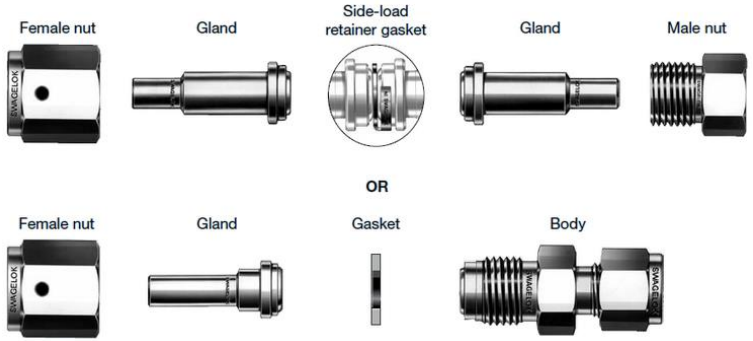
- Sampling : sources and sinks of water vapour should be avoided
  - Leaks, droplets, condensation and hygroscopic materials



S. Bell, "A guide to the measurement of humidity", NPL, 1996



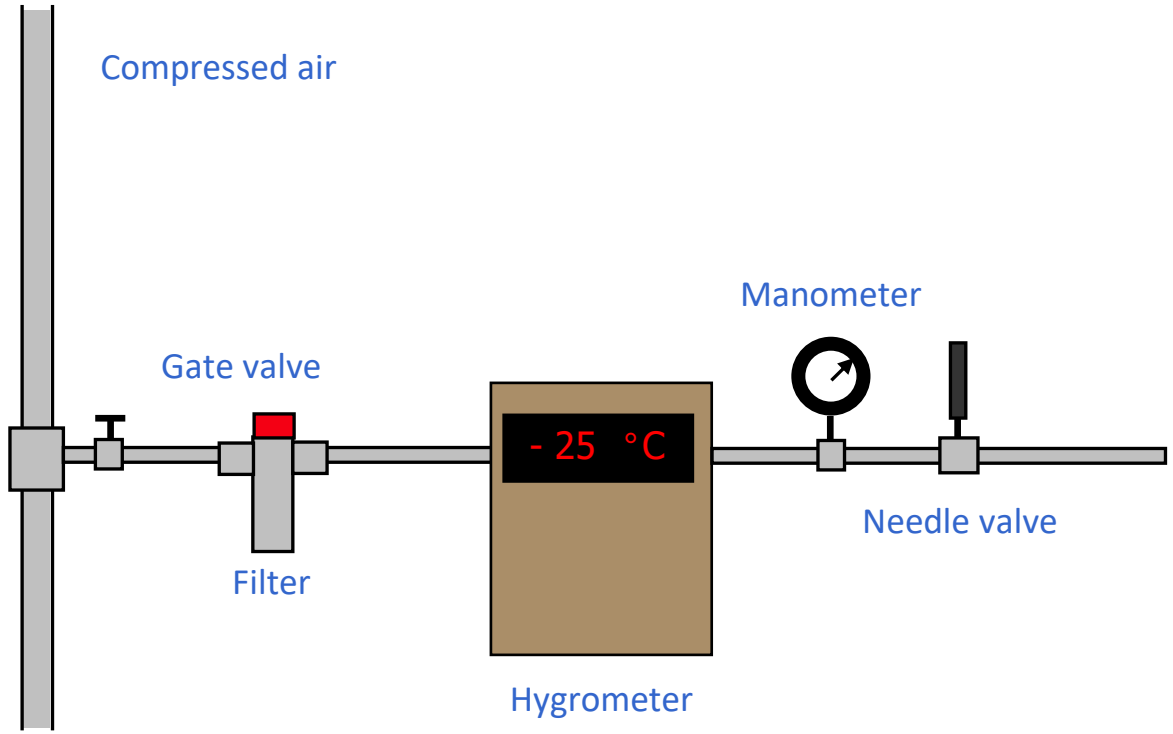




https://edmontonvalve.swagelok.com/blog/bid/325525/the-advantages-of-swagelok-vcr-and-vco-fittings

Chilled mirror hygrometer … tips and traps

- Sampling : low frost point temperature measurement ($\theta_{d/f} \leq \theta_{d/f\ ambient}$)



PROMETH$_2$O

Thank you for your attention

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