European Commission

Calibration procedures and challenges in the automotive field

CCQM Workshop on Particle Metrology

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Joint Research Centre

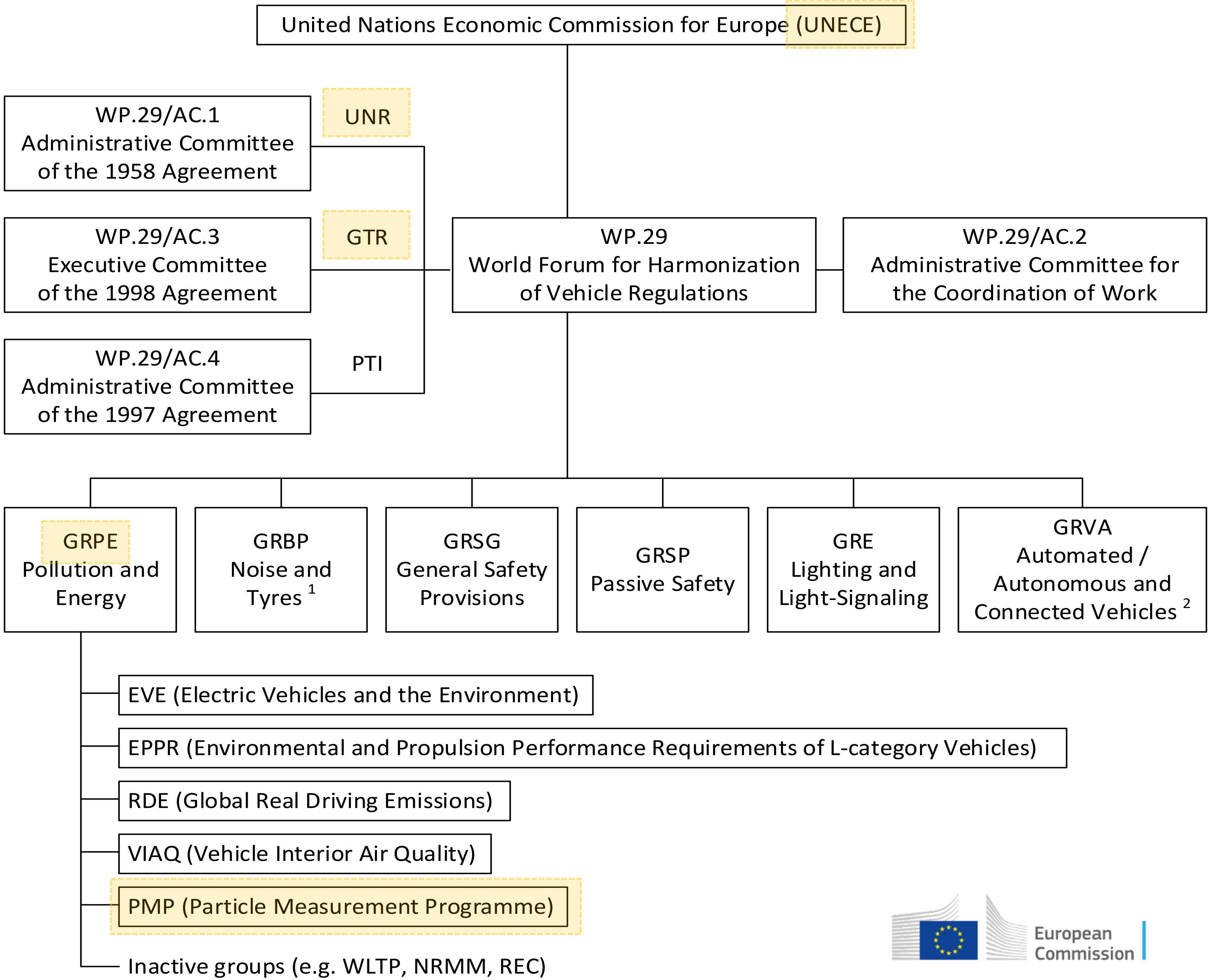
Overview

- Introduction
- PMP (Particle Measurement Programme) and UNECE
- Regulations
- LABS (Laboratory systems)
- PEMS (Portable Emission Measurement Systems)
- PTI (Periodic technical inspection)
- Outlook

GTR: Global Technical Regulations: Contracting parties have to apply their procedures and limits

UNR: United Nations Regulations: Mutual recognition between Contracting Parties

EU: EU Regulations apply across EU



European Commission

PMP history

The Particle Measurement Program (**PMP**) group was launched in 2001, and since then, it has evolved into an international group.

2001-2003: Assessment of sampling systems and measurement techniques. Definition of particle number methodology (solids >23 nm)

2004-2006 (light-duty), 2007-2009 (heavy-duty) inter-lab exercises

2010-2011: Regulation refinements

2013-today: Extension to lower particle size (>10 nm), other technologies and non-exhaust emissions

AST 42, 719-749, 2012

Regulations

Light-duty

Location	GTR	UNR	EU	SPN_{23} (#/km)	PM (mg/km)
$LABS_{23}$	15	83 or 154	2017/1151	6×10^{11}	4.5
$PEMS_{23}$	- [1]	- [2]	2017/1151	CF=1.5 [3]	-

[1] informal document; [2] working document; [3] to be reduced to 1.34.

Heavy-duty

Location	GTR	UNR	EU	SPN_{23} (#/kWh)	PM (mg/kWh)
$LABS_{23}$	4 (no PN)	49	582/2011	6×10^{11} [1]	10
$PEMS_{23}$	-	-	582/2011	CF=1.63	-

[1] 8×10^{11} #/kWh for the steady cycle (only diesel).

NRMM (non-road mobile machinery

Location	GTR	UNR	EU	SPN_{23} (#/kWh)	PM (mg/kWh)
$LABS_{23}$	11 (no PN)	96	2017/654	1×10^{12}	15
$PEMS_{23}$	-	-	2016/1628	-	-

Worldwide regulations

Table 7. Summary of regulations including particle number limits. The table gives the first emission stage including a particle number limit. In brackets the relative technology and the

Country	System	Light-duty	Heavy-duty	NRMM
Korea	LABS23	Euro 6 (D, 2014)	Euro VI (D, 2015)	no
Singapore	LABS23	Euro 6 (D, GDI 2018)	Euro VI (all, 2018)	no
India	LABS23	BS VI (D, GDI, 2020)	BS VI (all, 2020) [1]	BS V (2024)
	PEMS23	CF=tbd (2023)	CF=tbd (2023)	
China	LABS23	CN5 (D, 2016) [2]		
	LABS23	CN 6a (all, 2020)	CN VIa (all, 2019)	Stage IV (2023) [1]
	PEMS23	CN 6b (CF=2.1, 2023)	CN VIb (CF=2.0, 2021)	CF=2.5 (not PN)

[1] 2025 for positive ignition; [2] earlier in Beijing (2013) and Shanghai (2014); limit 5×10^{12} p/kWh.
BS=Bharat stage; CN=China; D=diesel; GDI=gasoline direct injection; NRMM=non-road mobile machinery

Sampling possibilities

LABS=Laboratory system

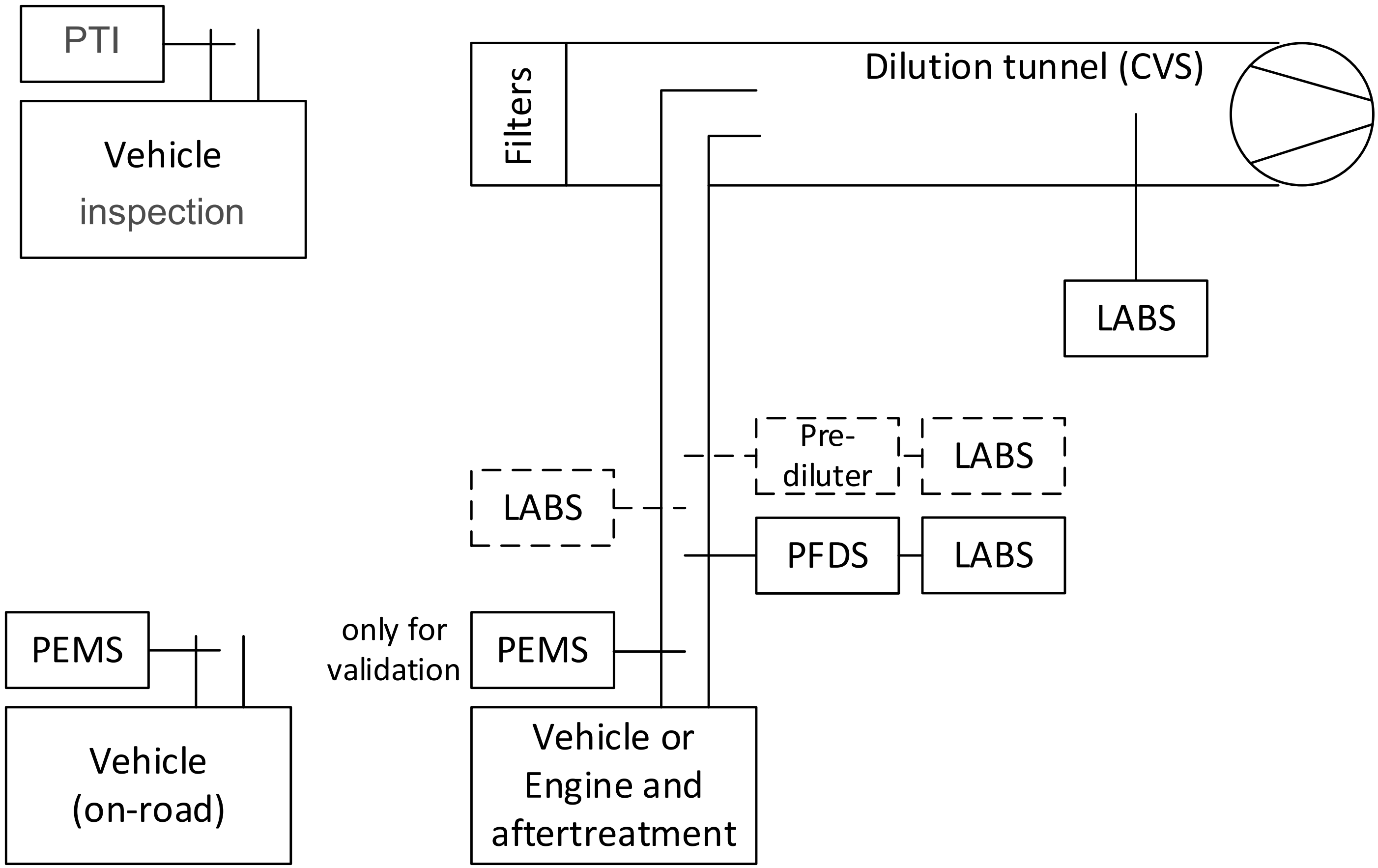
Full dilution tunnel with constant volume sampling (**CVS**)

Proportional partial flow dilution system (**PFDS**) for heavy duty

Direct tailpipe sampling with optional pre-diluter (heavy-duty resolution)

PEMS=Portable emissions measurement system

PTI=Periodic technical inspection



LABS

Technical specifications of laboratory systems

Black font: Current 23nm

Green font: Improvements

Blue italics font in dotted square: Future 10nm

Red: Heated

Dashed lines: optional parts

Main calibration requirements

VPR

$P_{100nm} \geq 70\%$

$PCRF_{50} / PCRF_{100} \leq 1.2$

$PCRF_{30} / PCRF_{100} \leq 1.3$

$VRE_{C40,30nm,>10^4\ \#/cm^3} > 99.0\%$

$PCRF_{15} / PCRF_{100} \leq 2.0$

$VRE_{C40,50nm,>1\ mg/m^3} > 99.9\%$

PNC

Linearity ±5% from slope

$CE_{23} = 50\%\ (\pm 12\%)$

$CE_{41} > 90\%$

$CE_{10} = 65\%\ (\pm 15\%)$

$CE_{15} > 90\%$

Recommended system:

T_{ET} =350 °C (may be CS)

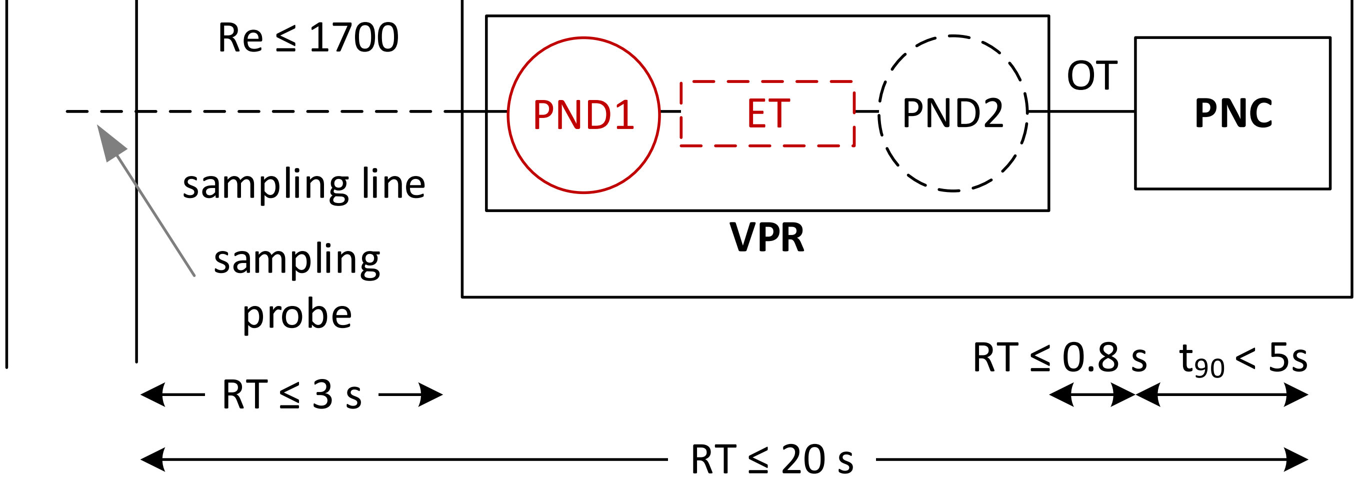
T_{ET} =350 °C (must be CS)

Full flow

Single counting mode

DF ≥ 10:1 150 ≤ T ≤ 400 °C

$d_{in} \geq 4mm$



CE=counting efficiency
PNC=particle number counter
VPR=volatile particle remover
VRE=volatile removal efficiency
RT=residence time
ET=evaporation tube
CS=catalytic stripper
PCRF=particle concertation reduction factor
DF=dilution factor
P=penetration

Calibration of LABS (PNC+VPR)

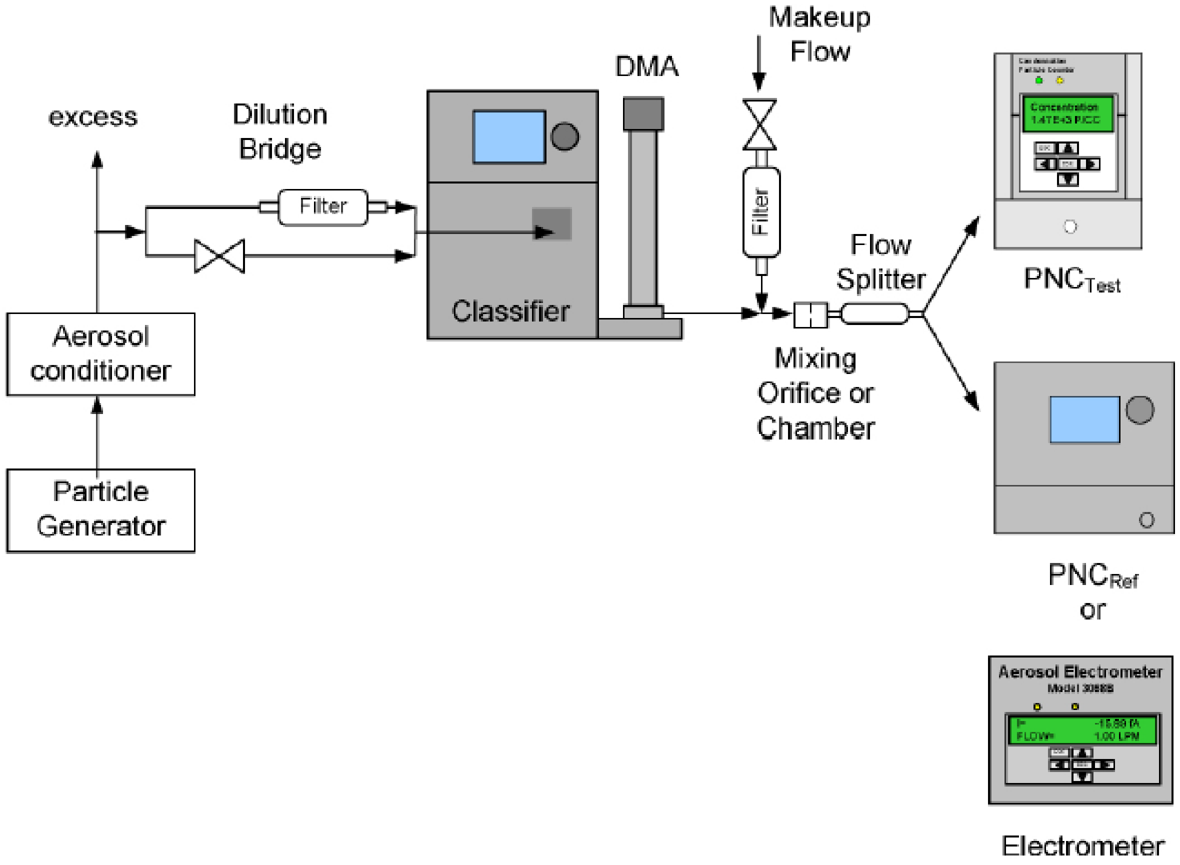


Figure 1. Example of an experimental setup for the calibration of a PNC.

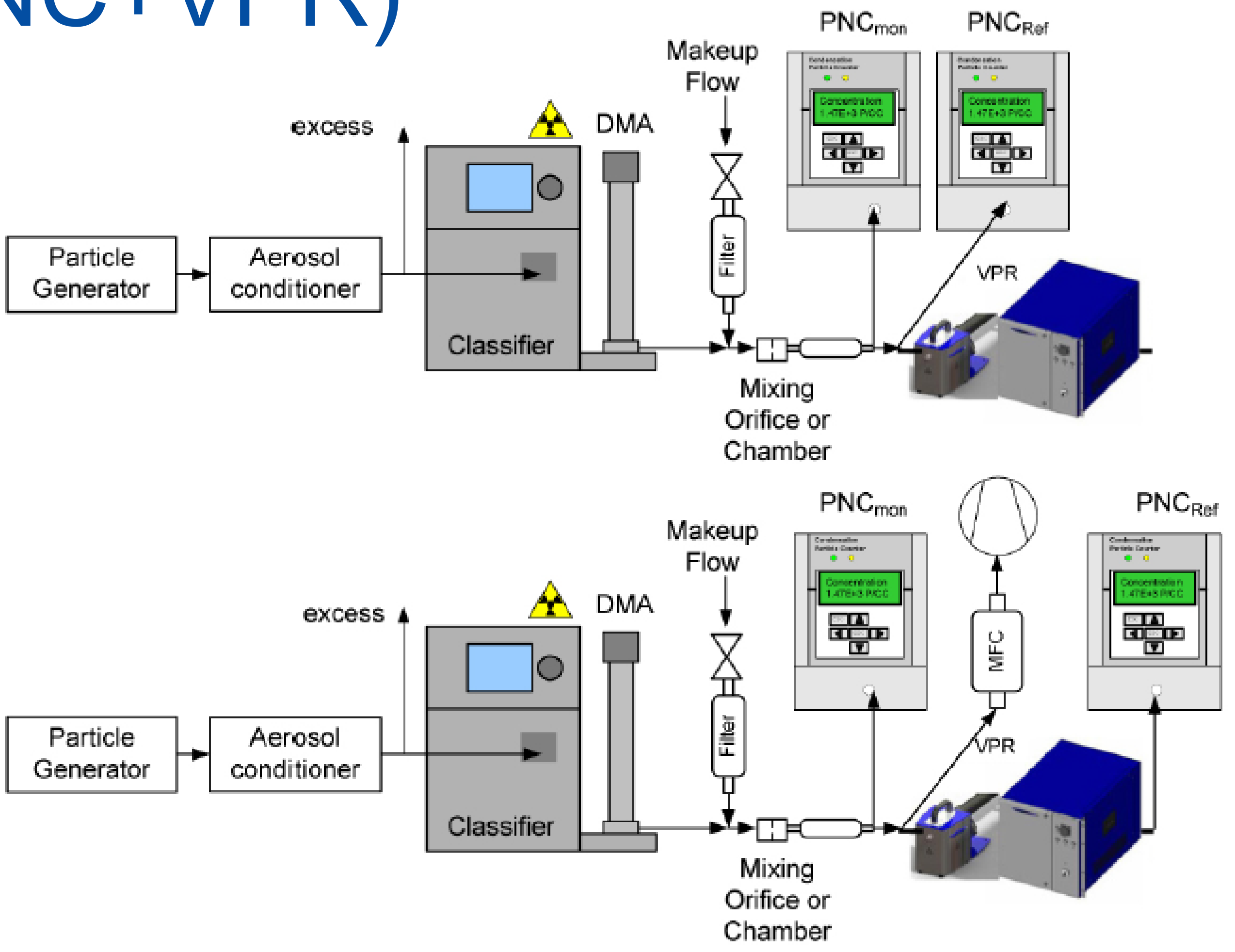


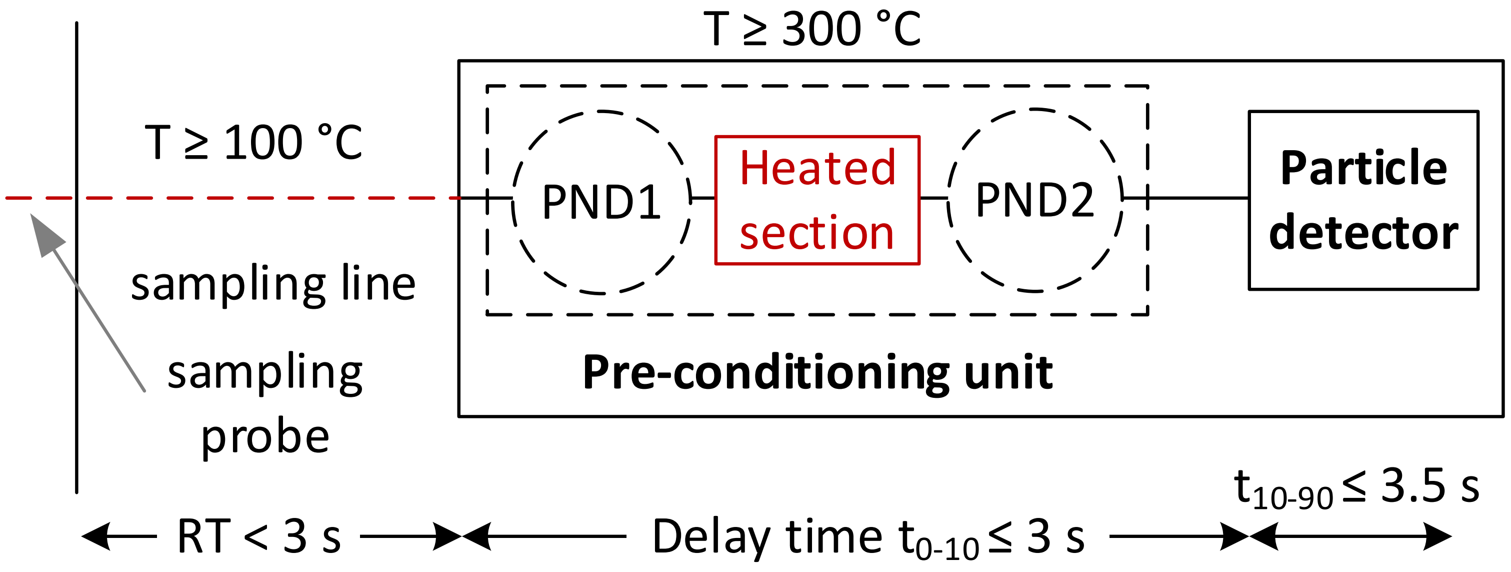
Figure 10. Example of the experimental setup for the calibration of a VPR a) upstream b) downstream measurement.

Portable Emission Measur. System (PEMS)

PEMS was not developed by PMP group, but from EU light-duty RDE group.

Only a few requirements:

- Heated section at 300°C
- Linearity
- Monodisperse efficiency
- Volatile removal efficiency



PEMS technical requirements

Linearity: slope ±15% (and differences within 15%)

Volatile removal efficiency >1 mg/m^3 tetracontane

Strict monodisperse efficiencies

Measurement parameter/instrument	$\lvert x_{min} \times (a_1 - 1) + a_0 \rvert$	Slope a_1	Standard error of the estimate *SEE*	Coefficient of determination r^2
PN analysers[14]	$\leq$ 5 % x_{max}	0.85 – 1.15[15]	$\leq$ 10 % of x_{max}	$\geq$ 0.950

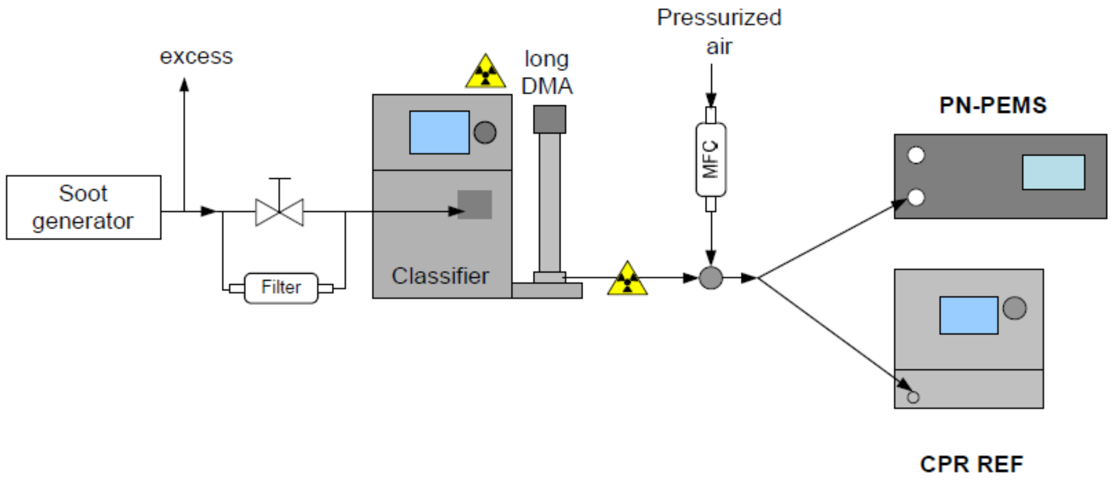
[14] The linearity check shall be verified with soot-like particles, as these are defined in paragraph 6.2. of this annex.

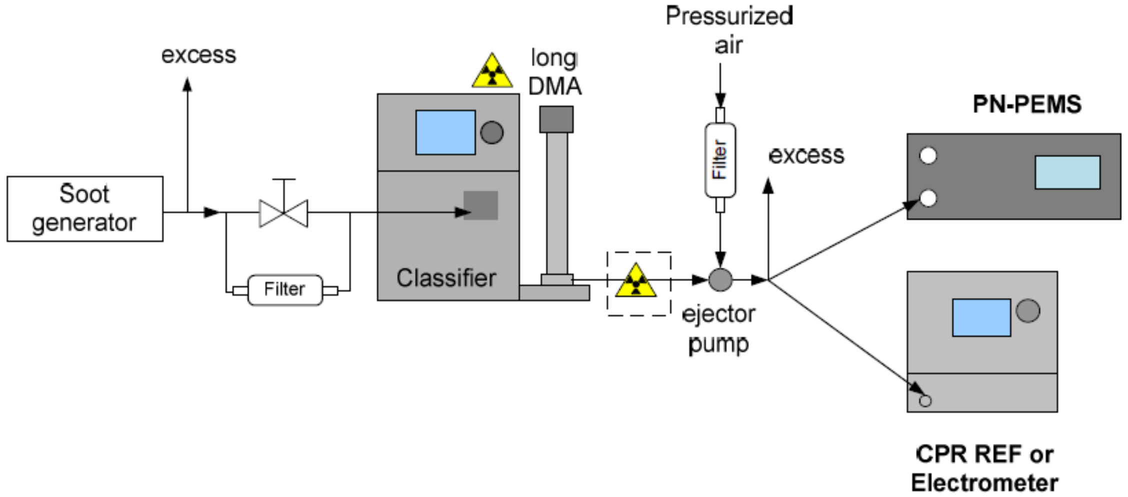
[15] To be updated based on error propagation and traceability charts.

Table 3. Efficiencies of particle number portable emissions measurement systems PEMS.

System	10 nm	15 nm	23 nm	30 nm	50 nm	70 nm	100 nm	200 nm
$PEMS_{23}$			0.20 – 0.60	0.30 – 1.20	0.60 – 1.30	0.70 – 1.30	0.70 – 1.30	0.50 – 2.00
$PEMS_{10}$	0.10 – 0.50	0.30 – 0.70		0.75 – 1.05	0.85 – 1.15	0.85 – 1.15	0.80 – 1.20	0.80 – 2.00

Calibration of PEMS (efficiency, linearity)





Linearity challenge mainly for DC based systems: how to reach 10^8 #/cm^3 (reference instrument, coagulation)

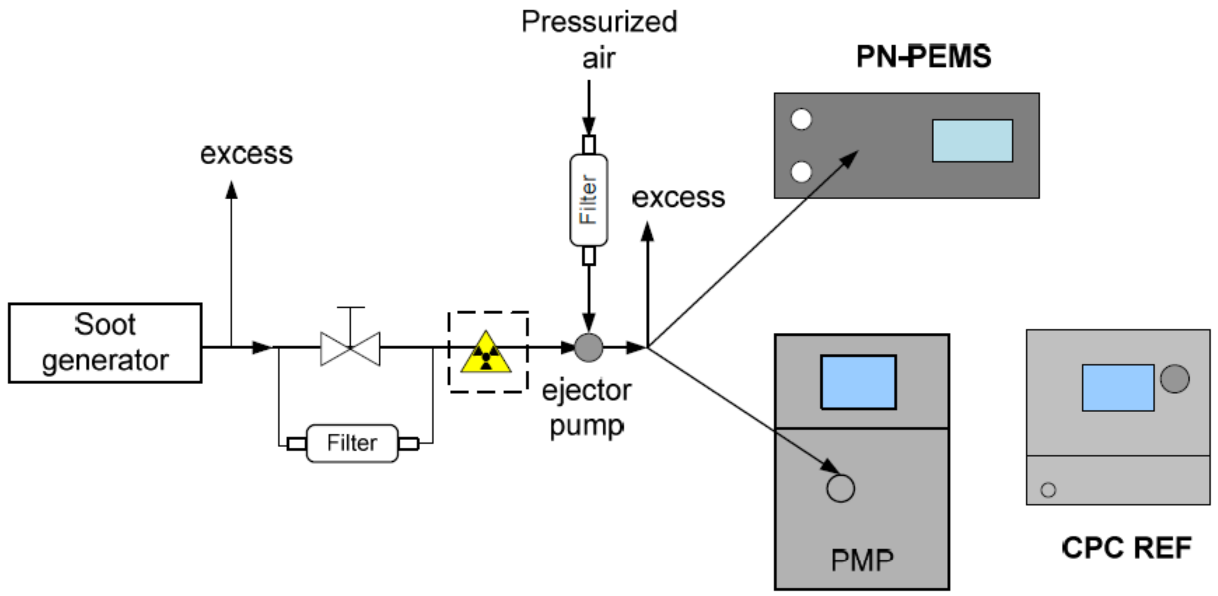
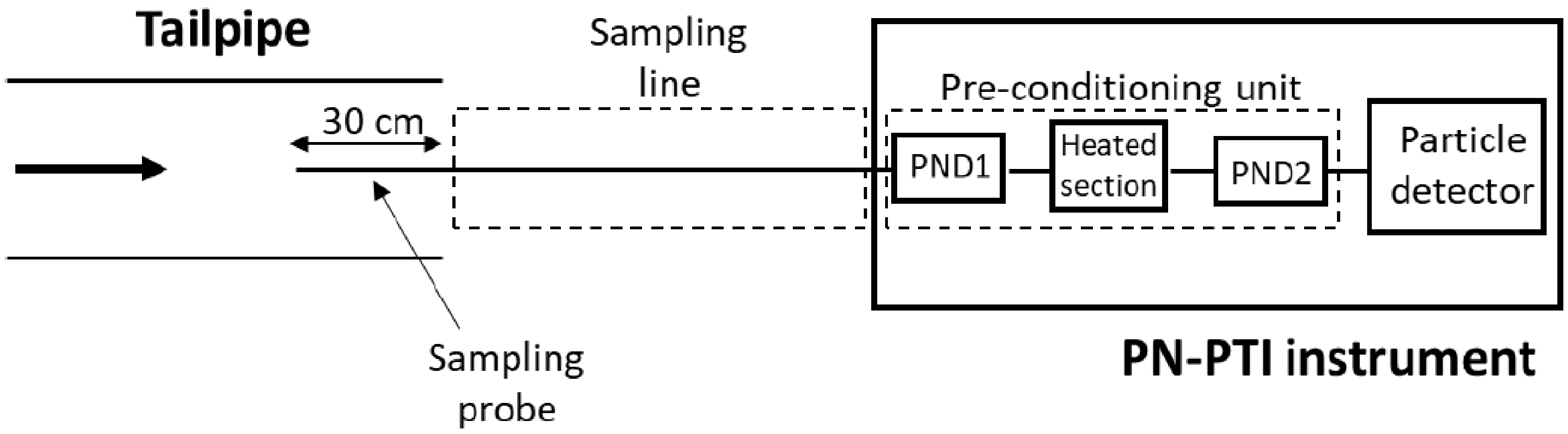


Figure 4.1: Example of setup for polydisperse linearity check.

Periodical Technical Inspection (PTI)

The PN PTI instrument should be comprised of a sampling probe, a sampling line (optional), a device/technique to avoid water condensation, a pre conditioning unit for removing volatiles (optional), and a particle detector



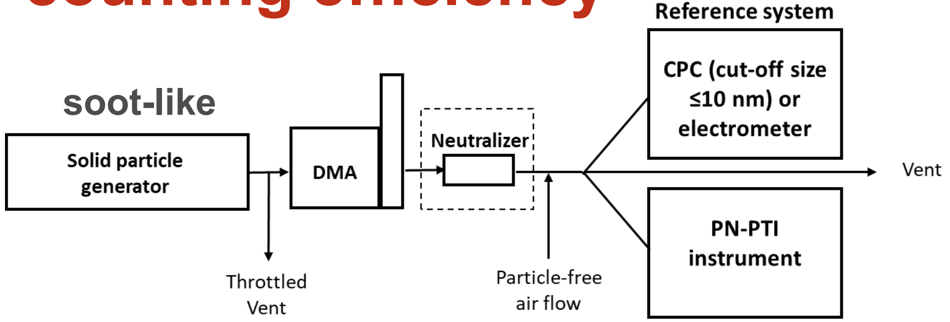
With dash lines the optional parts

PTI calibration setups

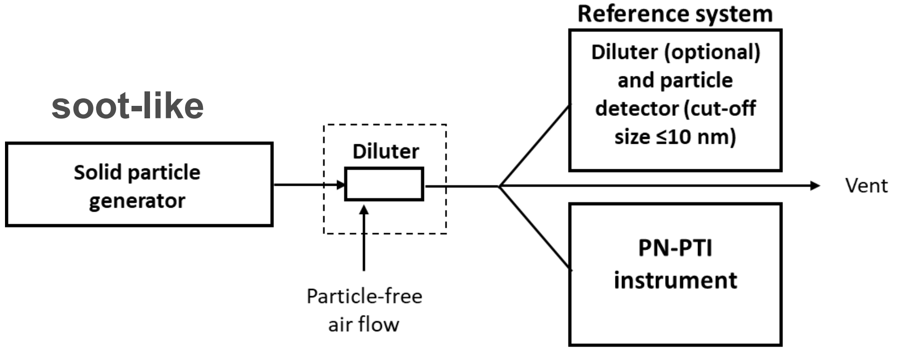
- Type examination of a PN-PTI instrument (NMI): thermally stable soot-like particles: efficiency and linearity (and other controls)
- Verification (only linearity) (manufacturer or authorised centres): any but with a correlation factor with soot-like particles. Also the reference system efficiency should be correlated to soot-like

NMI: counting efficiency

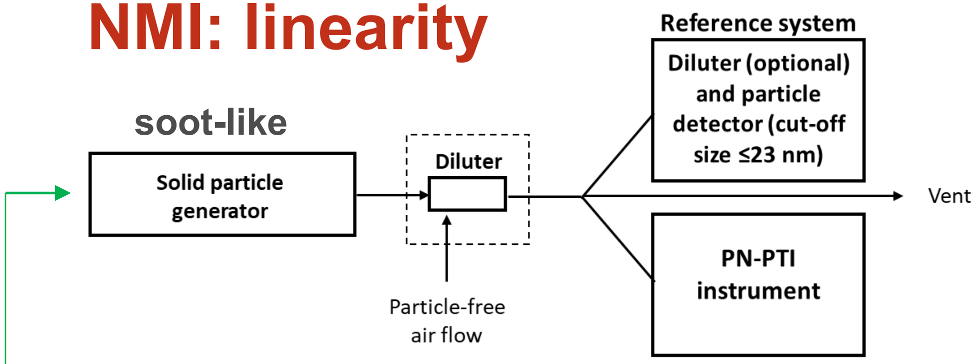
Mono-disperse



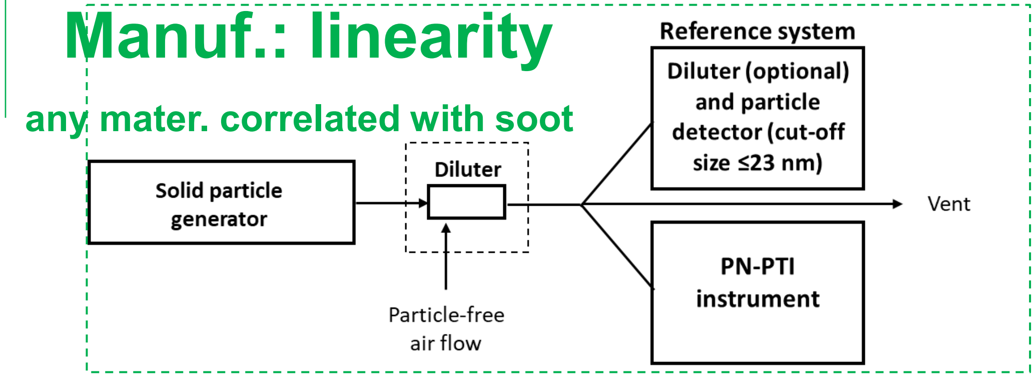
Poly-disperse



NMI: linearity



Manuf.: linearity



Outlook

Brakes: In addition to the mass (filter method), methodology for TOTAL (or SOLID) particles. Material: soot-like, emery oil, and silver. Emphasis should also be given to micrometer range.

Calibration improvements: Either “Simple” improvements in the calibration procedures or more complicated more common requirements and efficiencies between LABS and PEMS

- Uncertainty of systems calibrated in parts vs complete system

Daily checks: High response check not available

Outlook

Removal of volatiles

- Evaporation tubes: Volatile artefacts mainly <23 nm noted. Risk for PTI
- Catalytic stripper: oxidation efficiency check (e.g.. with a gas like propane) or degradation over time. Is there any other material more suitable (e.g. SO_2)?

Linearity

- Monodisperse: CPCs can go up to 10^5 #/cm^3 in single counting mode. Can DMAs / neutralizers handle the respective inlet concentrations?
- Polydisperse: Linearity at 10^8 #/cm^3 concentrations. Is the 15% limit for PEMS feasible?

Outlook

Materials

- PNC: emery oil or soot-like
- VRP: thermally stable
- PEMS: soot like
- PTI: soot-like but any at verification (mainly salt) with correlation to soot

<u>Concentration</u>: Can soot-like 10 nm particles be produced at high enough concentrations for calibrations?

<u>Uncertainty budget</u>: Soot-like (and other) materials can have high multiply charged particles fraction. Is this correctly taken into account?

<u>Traceability</u>: Is the selected material(s) creating traceability issues?

Thank you for your attention!

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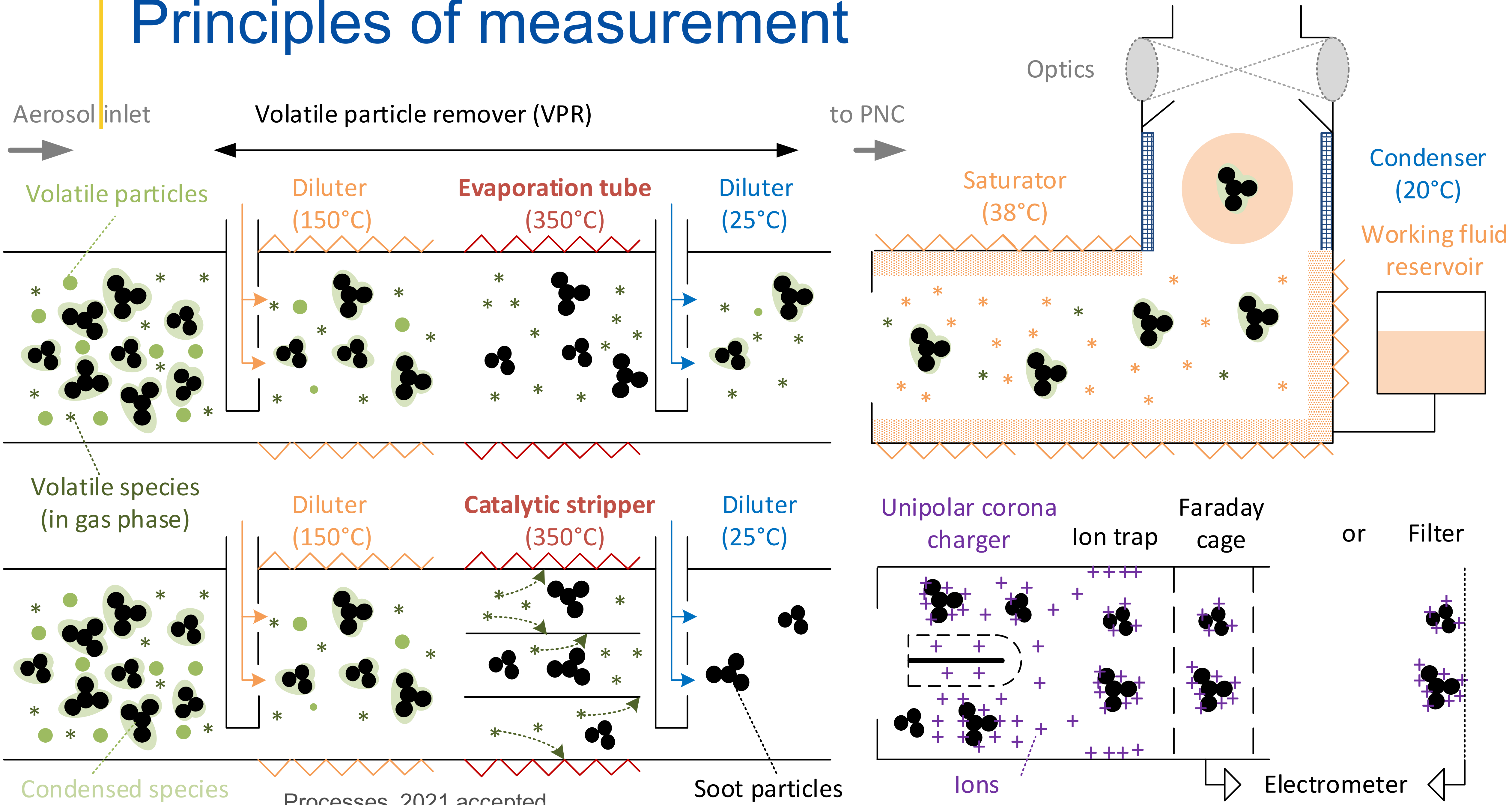
Terminology

The term "particle" is used for the matter being characterized (measured) in the airborne phase (suspended matter), and the term "particulate" for the deposited matter. In regulations particle number (PN) emissions mean the total number of solid particles emitted from the vehicle exhaust quantified according to the dilution, sampling and measurement methods as specified in the regulation.

Current particle methodology (laboratory): Sampling from the dilution tunnel. Hot dilution of 150°C, evaporation tube at 350°C, counting of particles >23 nm

Typically the abbreviation **PN** (particle number) is used, but recently **SPN** (solid particle number) has been introduced.

Principles of measurement



Processes, 2021 accepted

Table 1. Comparison of original $LABS_{23}$, improved $LABS_{23}$ and new (improved) $LABS_{10}$ technical specifications and calibration requirements. Dashed line separate the calibration requirements. In green improvements compared to the original regulation. In blue and italics specifications for the $LABS_{10}$ system.

Part	$LABS_{23}$ original	$LABS_{23}$ improved	$LABS_{10}$ (improved)
Sampling line	optional RT ≤ 3 s Re ≤ 1700	optional RT ≤ 3 s Re ≤ 1700	optional RT ≤ 3 s Re ≤ 1700
Volatile Particle Remover (VPR)	PND1 (DF ≥ 10:1) 150 ≤ T ≤ 400 (±10) °C 300 – 400 °C (recommended) No CS $T_{in,PNC}$ < 35°C	PND1 (DF ≥ 10:1) 150 ≤ T ≤ 400 (±10) °C 350 °C (recommended) May be CS $T_{in,PNC}$ < PNC specs	PND1 (DF ≥ 10:1) 150 ≤ T ≤ 400 (±10) °C 350 °C (recommended) With CS (recommended) $T_{in,PNC}$ < PNC specs
	P_{100} ≥ 70% (each instrument) $PCRF_{50}$ / $PCRF_{100}$ ≤ 1.2 $PCRF_{30}$ / $PCRF_{100}$ ≤ 1.3 - $VRE_{C40,30nm,≥10^4\ \#/cm^3}$ > 99.0% VRE yearly Thermally stable material Calibration 12 months PCRF validation 30, 50, 100 nm or polydisperse (50 nm): ±10%	P_{100} ≥ 70% (once for family) $PCRF_{50}$ / $PCRF_{100}$ ≤ 1.2 $PCRF_{30}$ / $PCRF_{100}$ ≤ 1.3 - $VRE_{C40,30nm,10^4\ \#/cm^3}$ > 99.0% VRE according to manuf. Thermally stable material Calibration 13 months PCRF validation 30, 50, 100 nm or polydisperse (50 nm): ±10%	P_{100} ≥ 70% (once for family) $PCRF_{50}$ / $PCRF_{100}$ ≤ 1.2 $PCRF_{30}$ / $PCRF_{100}$ ≤ 1.3 $PCRF_{15}$ / $PCRF_{100}$ ≤ 2.0 $VRE_{C40,≥50nm,1\ mg/m^3}$ > 99.9% VRE according to manuf. Thermally stable material Calibration 13 months PCRF validation 30, 50, 100 nm: ±10%

Table 1. Comparison of original $LABS_{23}$, improved $LABS_{23}$ and new (improved) $LABS_{10}$ technical specifications and calibration requirements. Dashed line separate the calibration requirements. In green improvements compared to the original regulation. In blue and italics specifications for the $LABS_{10}$ system.

Part	$LABS_{23}$ original	$LABS_{23}$ improved	$LABS_{10}$ (improved)
Particle Number Counter (PNC)	$t_{90} < 5$ s	$t_{90} < 5$ s	$t_{90} < 5$ s
	Single counting mode	Single counting mode	Single counting mode
	Flow ±5% nominal	Flow ±5% last certificate	Flow ±5% last certificate
	Any material	Soot or PAO	Soot or PAO
	$0.9 < k_{slope} < 1.1$, $R^2 > 0.97$	$0.9 < slope < 1.1$, $R^2 > 0.97$	$0.9 < slope < 1.1$, $R^2 > 0.97$
	Linearity ±10%	Linearity ±5% from slope	Linearity ±5% from slope
	$CE_{23} = 50\%$ (±12%)	$CE_{23} = 50\%$ (±12%)	$CE_{10} = 65\%$ (±15%)
	$CE_{41} > 90\%$	$CE_{41} > 90\%$	$CE_{15} > 90\%$
	k_{slope} may be included in CE	k_{slope} included in CE	k_{slope} included in CE
	Coincidence correction <10%	Any internal correction	Any internal correction
	6 mo monitor or wick exchange or ±10% of PNC_{Ref} or ≥ 2 PNCs	6 mo monitor or wick exchange or ±10% of PNC_{Ref} or ≥ 2 PNCs	6 mo monitor or wick exchange or ±10% of PNC_{Ref} or ≥ 2 PNCs
	Certificate 12 months	Certificate 13 months	Certificate 13 months
Combined	$PCRF_{ave}$ of 30, 50, 100 nm	$PCRF_{ave}$ of 30, 50, 100 nm	$PCRF_{ave}$ of 30, 50, 100 nm
	Total RT ≤ 20 s	Total RT ≤ 20 s	Total RT ≤ 20 s
	Ambient air: > 100 #/cm³	No error	No error

CE = counting efficiency; CS = catalytic stripper; DF = dilution factor; ET = evaporation tube; P = penetration; PCRF = particle number concentration reduction factor; PNC = particle number counter; RT = residence time; T = temperature; VRE = volatile removal efficiency; VPR = volatile particle remover.

Pre-diluter

Proposal for heavy-duty engines type approval: Sampling directly from the tailpipe with fixed dilution, optionally adding a pre-diluter.

Table 2. Draft technical specifications of the pre-diluter.

Specification	Description
Location	A cold or hot pre-diluter may be located at the end of the particle sampling probe and in front of the particle transfer tube (PTT). [1]
Dilution	A fixed dilution ration >5:1 shall be applied to the cold or hot dilution stage. Cold dilution is defined as a dilution with (unheated) dilution air and/or diluter temperature ≥20 °C
Penetration	The complete system (pre-diluter, PTT, and VPR) penetration shall not decrease more than ~~20%~~ 10% the penetration requirements specified for the VPR.
PCRF	The complete system (pre-diluter, PTT, and VPR) PCRFs shall not exceed ~~10%~~ 0% for 50 nm, ~~20%~~ 10% for 30 nm, and ~~30%~~ 25% for 15 nm (if applicable) the PCRF requirements specified for the VPR.

[1] The residence time until the pre-diluter shall be ≤1 s. The tubing shall be heated at ≥150 °C if >10 cm, or insulated if ≤10 cm.

In green final agreed values in last PMP meeting