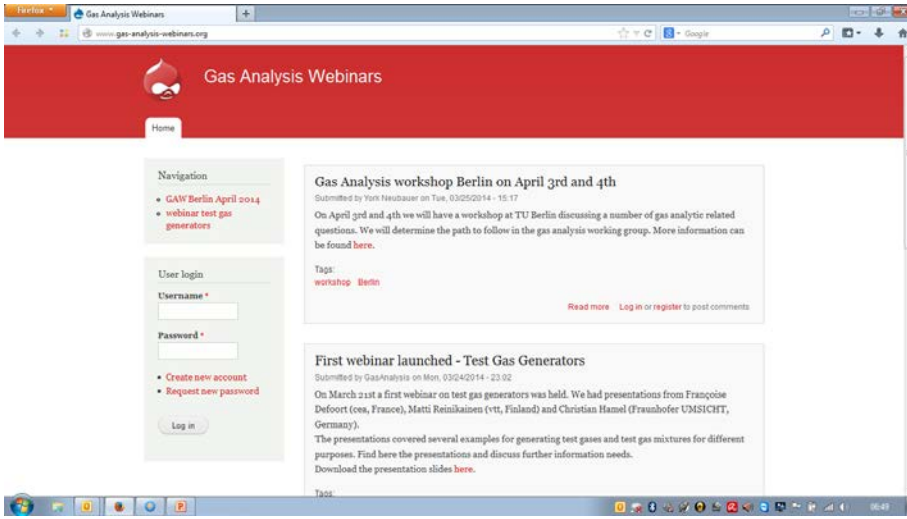
bioenergy2020+

Water measurement; an introduction



Markus Kleinhappl Bioenergy2020+

→ http://www.gas-analysis-webinars.org/

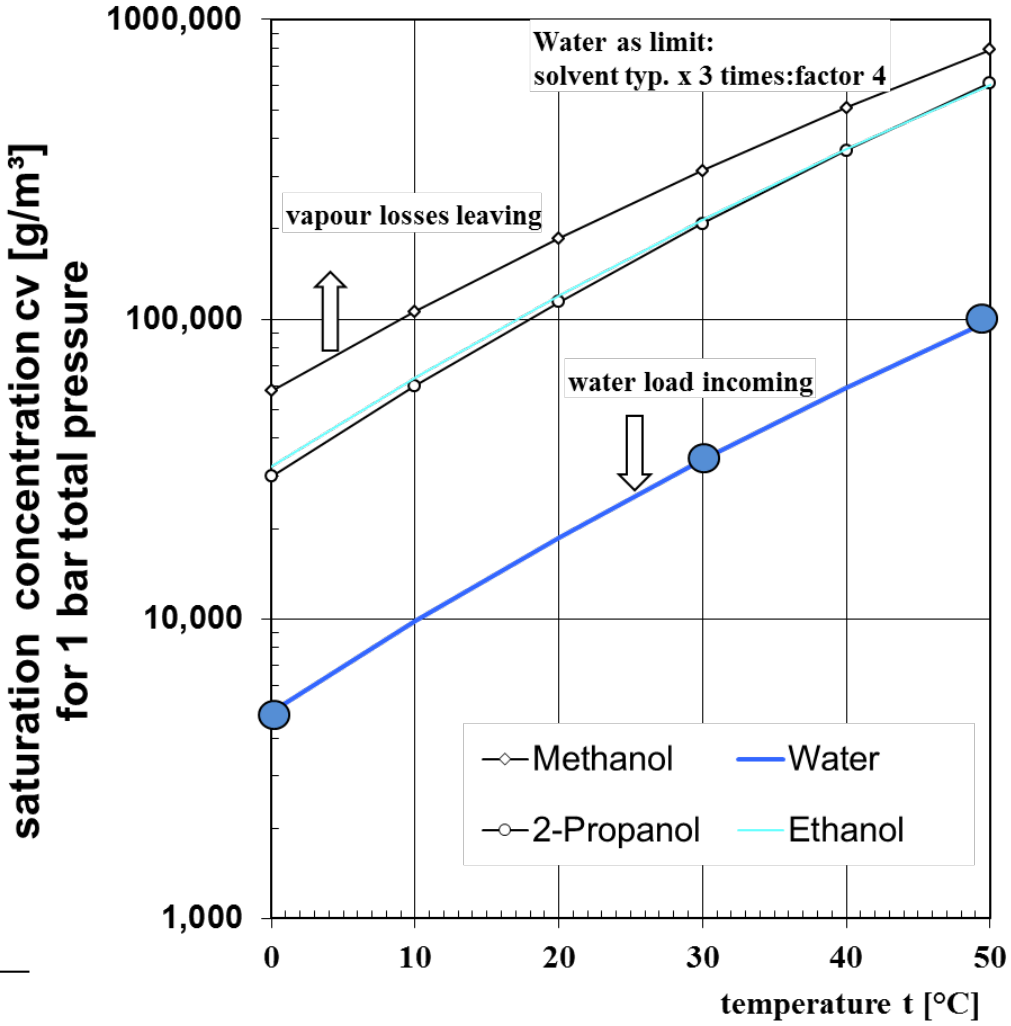
Workshop Berlin
3. and 4. th April Berlin

innovations kompetenz

Content

- Water content (Gas → sampling liquid)
- Water quantification methods
- BE approach for ‘standard’ applications
- Discussion

Saturation with water =humidity at ambient pressure (compared to other solvents)

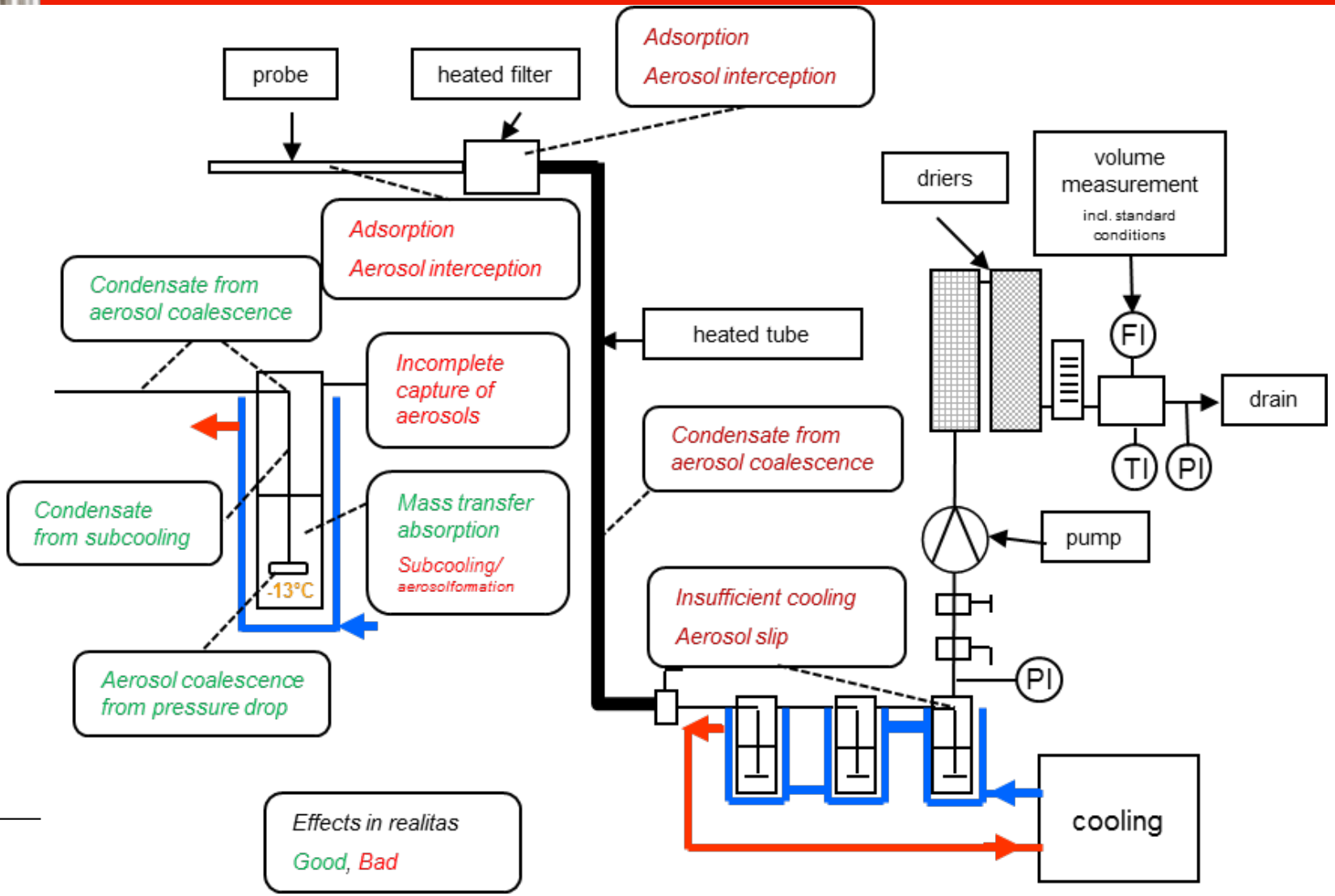


Consider:
Sample gas with dewing point of 50°C is introducing more water, than the loss of solvent of 2-Propanol is:

→ Sample 1 m³ gas in 100 ml liquid (initial)
→ Status at the end of sampling:
Solvent: -25 g (~30 ml) Propanol
Water: + 100g=100 ml

Final water content (mixed): 100/(100+100-30)>58%vol!

Effects all over the sampling line



Negative effects

Water and steam is very important during gasification, char gasification, tar reforming, but:

- **Dilution** of solvents
- **Phase separation** of inpolar solvents
- **Increased slip** of unpolar fractions during sampling
- **Layering on polar SPA-substrates** (reduced capture, time dependent result)
- **Ice formation,** sludge, rocks in liquid samplers

- **Baseline instabilities** (steam-breathing of column, FID-blowers)
- **Discrimination during evaporation** in GC injectors, early peaks blow off
- (compared low interaction HPLC-Systems: AcN:MeOH eluent)
- **Discrimination and delay in gravimetric** procedure, hygroscopic phenolics

Lab crew can do this:

- Do not sampling,
- Apply mixtures of solvents

- Take a laggard /ger: Schlepper/ like THF into liquid for gravimetric determination
- Separate majority of humidity during sampling with a pre-condensor
- (but treat then also this condensate and the glass equipment there)

- Separate the water from the sample
- Azeotropic distillation, like EN1948-1 (xylene)
- Diluter against water condensation (only for SPA-cartridges), like VDI 3872.

- Stop early enough the sampling at low water content, when You want to sample Benzene,…
- Dry the liquid with Na_2SO_4 x anhydro
- Extract the liquid sample with DCM or Hexane.

Degree of difficulties; scale

Temperature at sampling point	*Content of DUST*	*Content of WATER*	*Content of organics ('tars')*	*Compounds organic*	*Compounds in-organic*	*total*
1,000 → 4	>10,000 → 5	>80°C → 5	>10,000 → 5	**Manual**: Multiply x 1	**Manual**: Multiply x 1	
800 → 4	<10,000 → 5	<80°C → 3	<10,000 → 3			
600 → 4	<1000 → 3	<60 → 2	<1,000 → 2	**online**: Multiply x 10	**online**: Multiply x 5	
400 →3	<100 →2	<40 →1	<100 →1			
200 → 2	<10 → 1	<10	<10	**Pressure**:*) Multiply x p[bar]/10	**Pressure**:: Multiply x p/10	
ambient	<1	<0°C	<1			
°C	mg/m³	dewpoint local pressure	mg/m³ dewing			

Pressure:*) *factor for extractive sample preparation*

TF	+	DF	+	WF	+	OF	=	DEGREE
2+		2+		1+		2	=	normal application 7 (=medium)
4+		5+		3+		2	=	e.g. high dust fluidised bed sampling 14 high

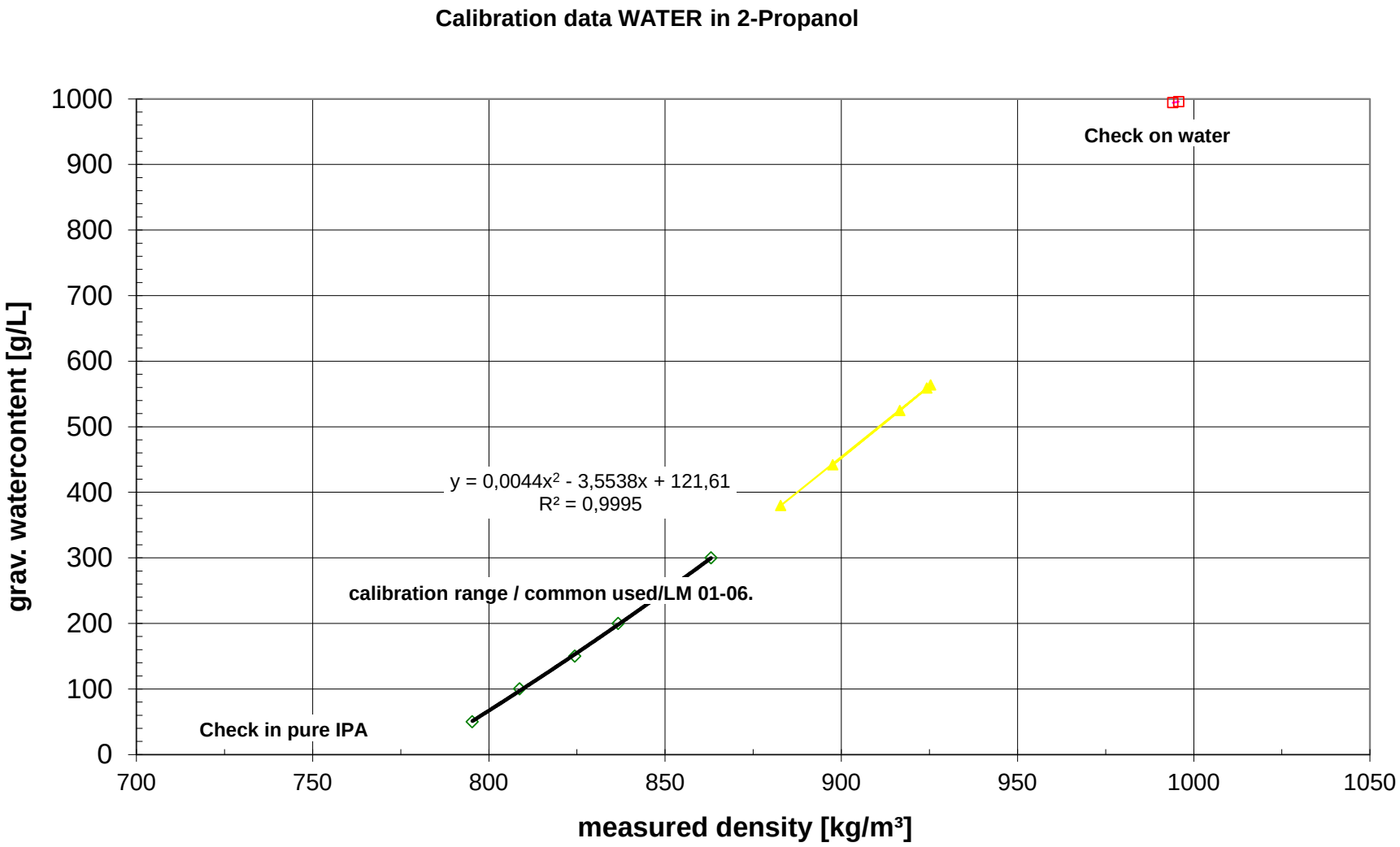
Example of water effects two chromatograms with/without water

Water quantification: toolboxes

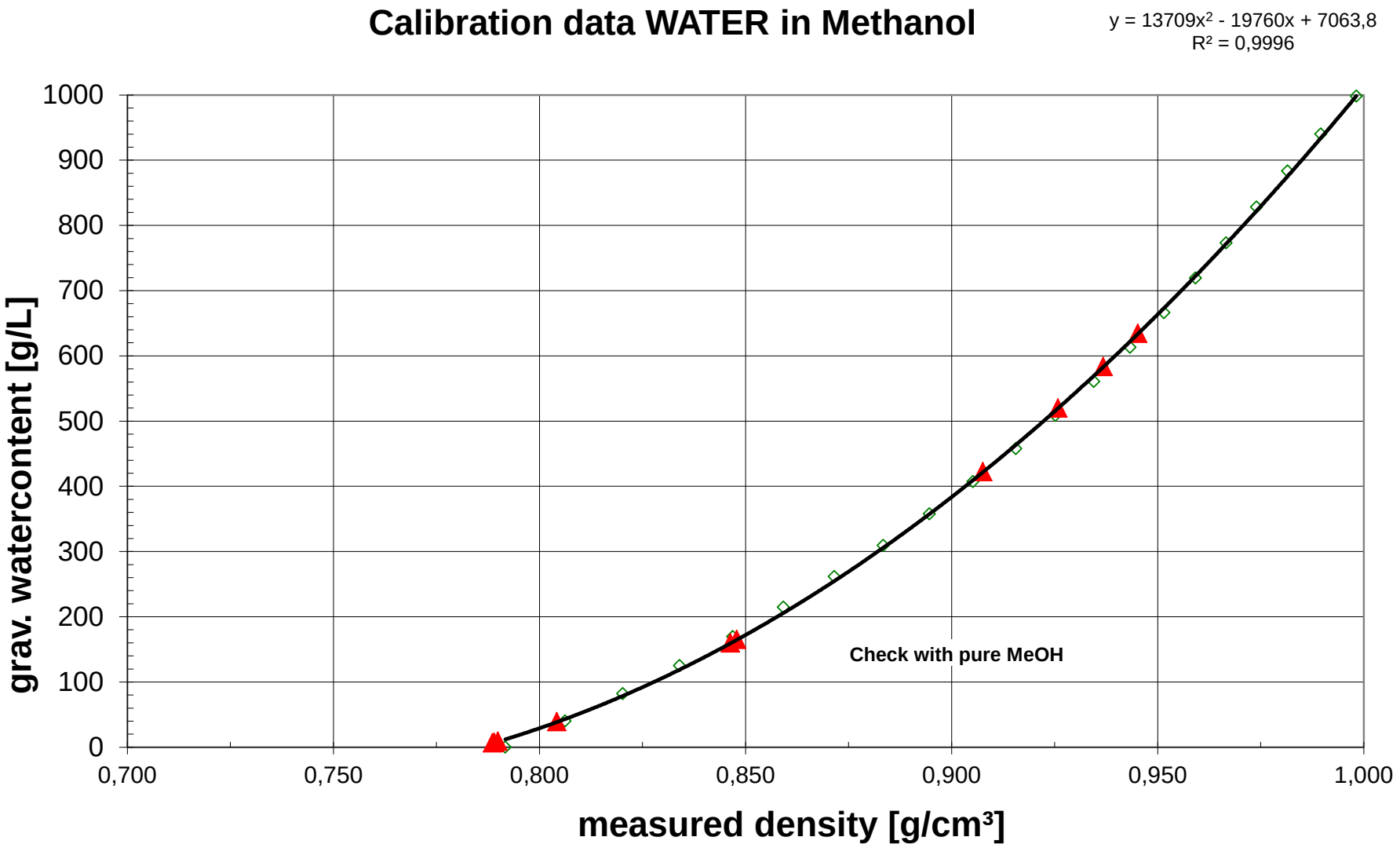
	Offline sampling & later analysis		**online sampling & quasi cont. sampling**	**Real time sample extraction and/or detection/quantification**
	Accumulation on solid phase	Accumulation in liquids (cooled)	Dosage, fractioning chromatographic separation in retention	Dosage or dilution, selective detection & quantification
Materials gadgets	Silicagel Secca-pent	Co-sampling with organics (tar)	Plot/mole-sieve Hayesep TCD-detector	Wet saturation temp. (wet bulb) Bartec [R] Acoustic, 'ultra-acust' NDIR-NH_3-detector and FTIR
Problems	Manually procedure Co-adsorption Heavy PAH-deposits Pyrolytic tar reactions	Sampling dilution liquid Slip of inpolars	Maintenance Co-adsorption on columns Conditioning of transferline & columns	Interference form dust, tar-deposits Dependency on main gas components (speed of sound) IR-band overlapping
COSTS	**Very low**	**Medium**	**Medium-High**	**High**

Water quantification

Water quantification via ‘tar-solvent’ from sampling IPA



Water quantification via ‘tar-solvent’ from sampling MeOH

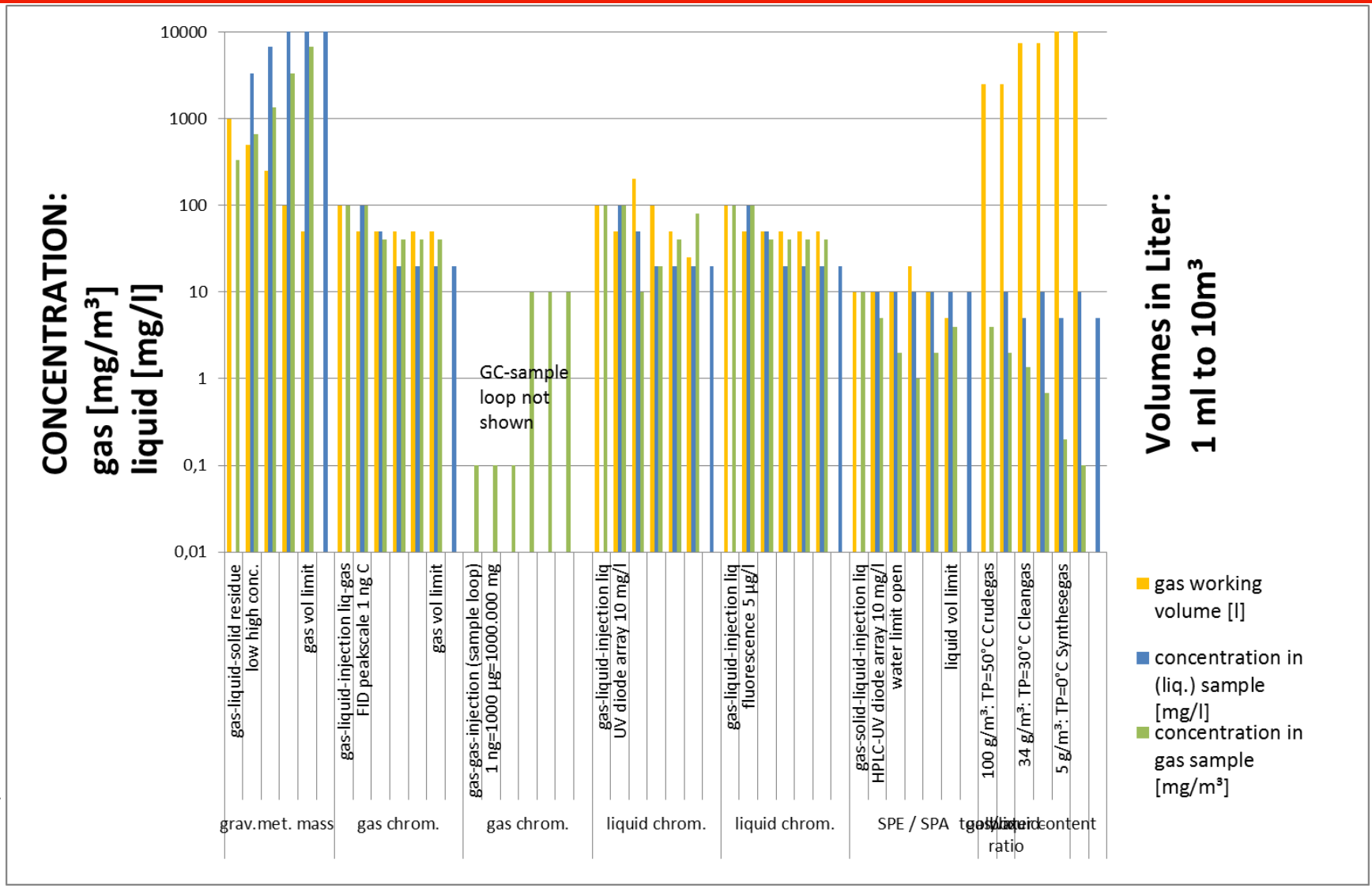


discussion

- Protocol of main facts:

- 1.
- 2.
- 3.
- 4.
- 5.

The gas/liquid ratio



H. Bosch *1450-1516*; *der Gauckler, the faker, il falsario*