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ifk

Current status of the further development of IFKs' FID based online tar measurement device

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Gas Analysis Workshop

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Tar analyzer history

~ 1994
- Idea of tar measurement with an FID based online device at IFK
- Development of 1st prototype at IFK lab for the verification of the general measurement system

1998
- 2nd (improved) prototype

1999
- 3rd and final IFK prototype available

2000
- License agreement to “Ratfisch Analysensysteme GmbH”
- 1st Ratfisch prototype available
- Start commercialization 1st generation measurement device

2001
- Different research projects in the field of gasification and (online) tar measurement at IFK with Ratfisch prototype

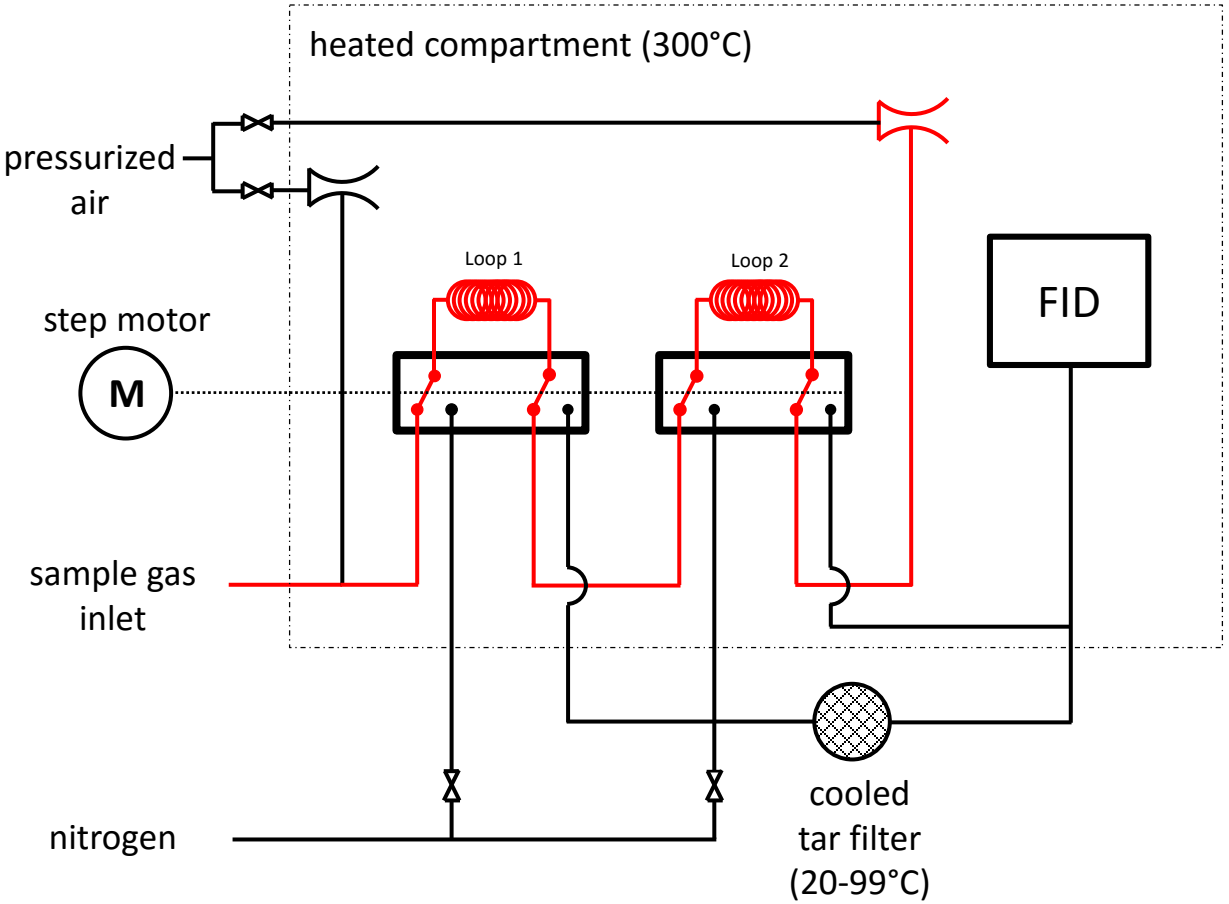
2011
- Start KIC DEMITAR project
  - Development of 2nd generation FID based online tar analyzer

2014
- **Laboratory and field testing**
  - Selection of filter material for the underlying difference measurement
  - Investigation of the impact of disturbances on the measurement principle and its accuracy
  - Comparison measurements with DIN CEN/TS 15439 and SPA at different gasifiers and gasification systems for validation

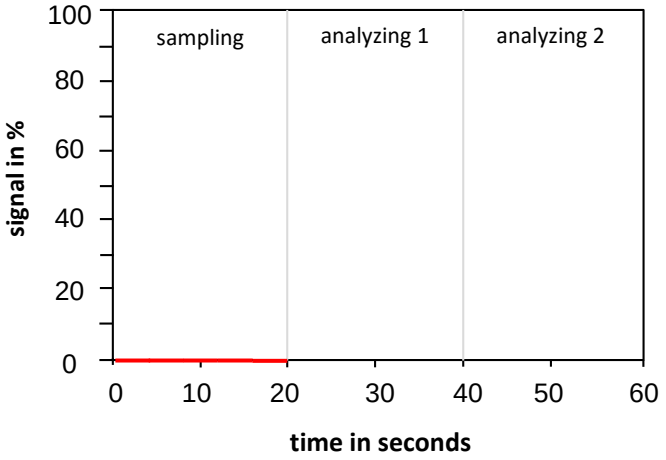
2016
- **Start commercialization 2nd generation measurement device**

Final device

Measurement principle

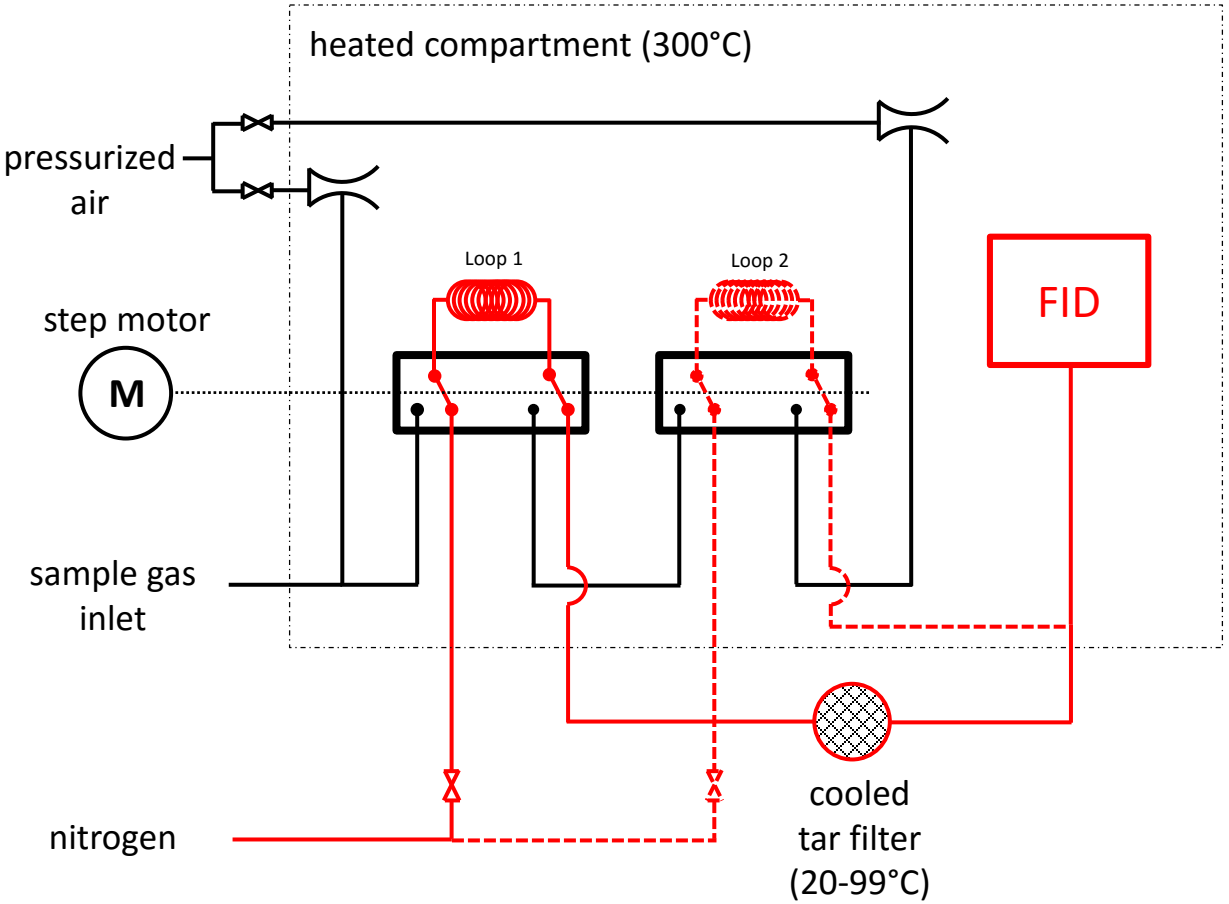


sampling phase

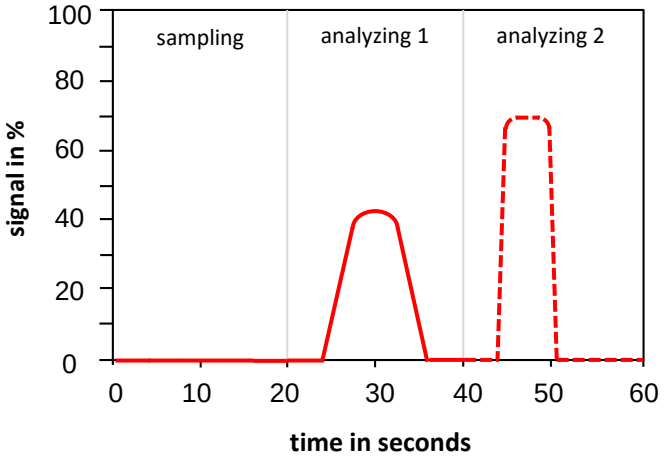


- Sample gas is sucked through sample loops with venturi nozzle.
- Both sample loops are filled consecutively.
- Detector signal is zero during loading phase.

Measurement principle



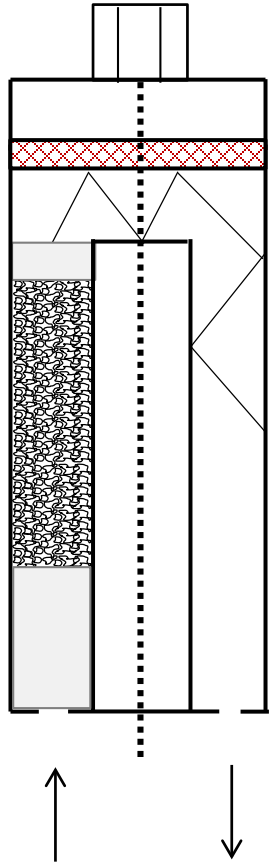
analyzing phase



- Gas in sample loops is flushed to the detector consecutively.
- Tars of sample loop 1 are condensed/separated on a cooled filter.
- Measured components:
  - Total hydrocarbon
  - Non-condensable HC
  - Tars (condensable HC)

Selection of tar filter material for the diff. measurement

- Optimal: Mix of cellulose acetate and “bentonite”
  - Cellulose acetate for “heavy tars”
    - Commercial cigarette filter (brand Zig-Zag)
  - „Bentonite“ for “light tars”
    - Different clay minerals
    - Principal component: Montmorillonite
    - Grain size: 0.3-0.4 mm

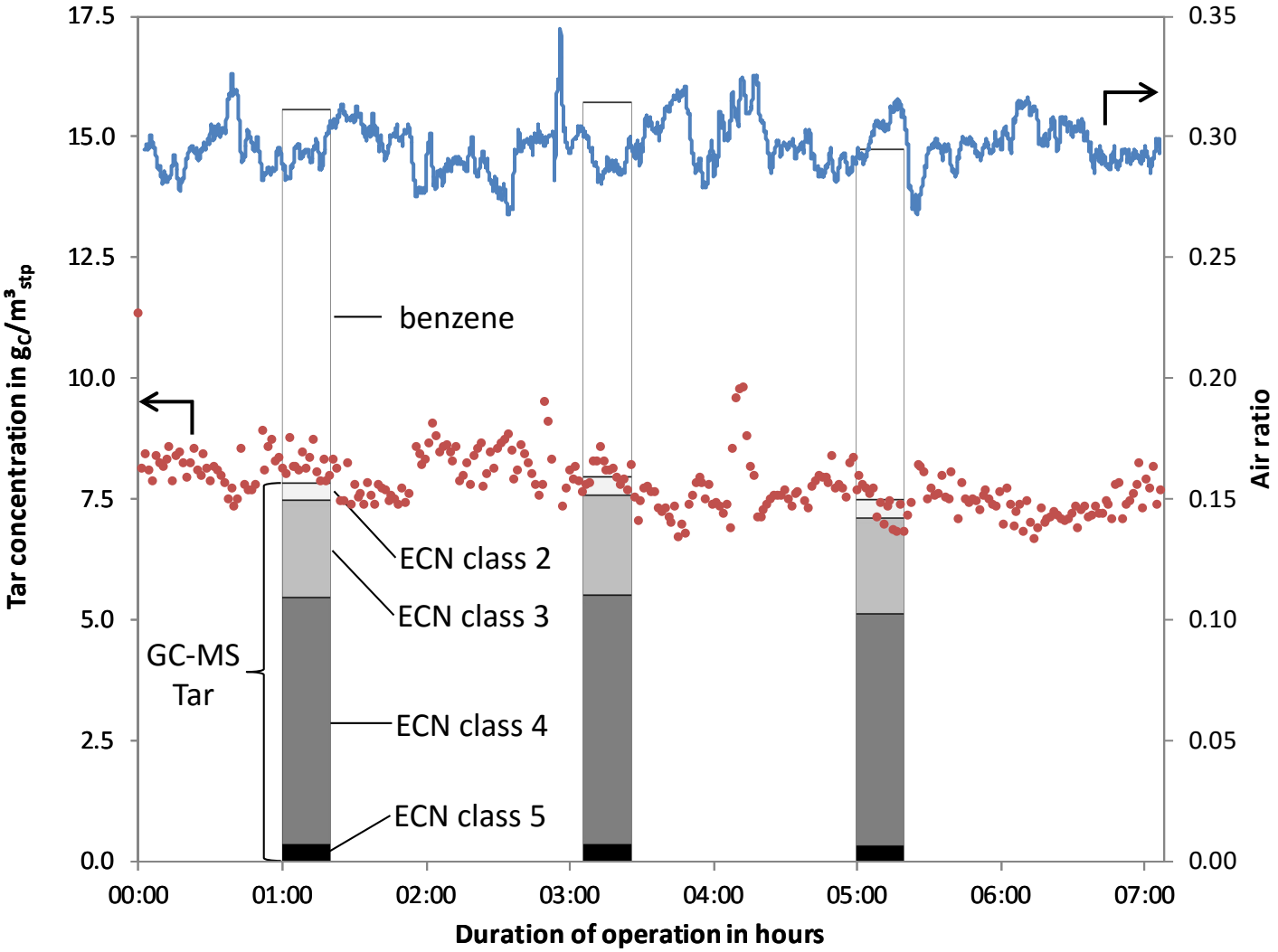


Producer gas

Height: 30mm
Diameter: 6mm
Volume: ca. 850mm³

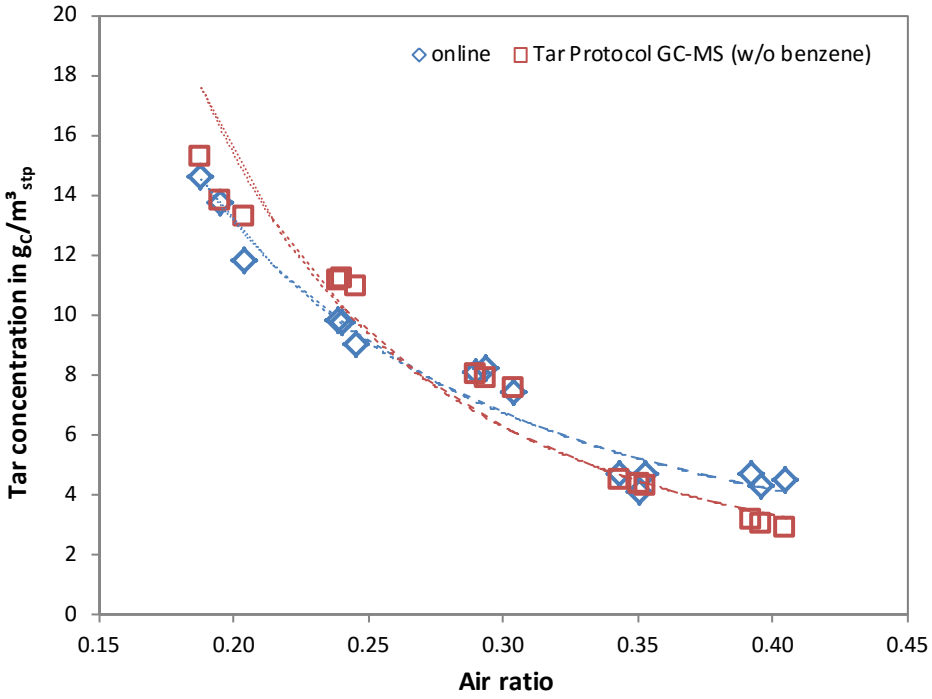
Comparative measurements - Results base case

Base case at 800 °C and an air ratio of 0.3



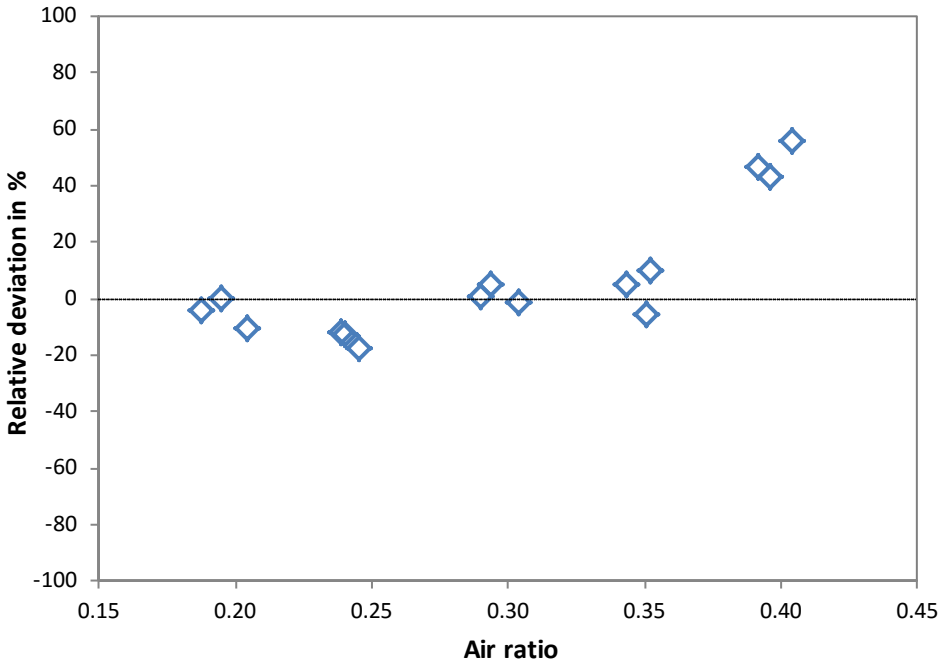
- Time of operation of more than 7 hours.
- 3 comparative measurements.
- One online measurement cycle had a duration of around 84 seconds (300 cycles).
- Online measured tar concentrations follow the wet chemical comparative measurements very close over the total time of operation.
- Air ratio fluctuates little because of the volumetric dosing system.
- Heavy fluctuations in air ratio result from refill of dosing system.
- If heavy fluctuations of air ratio present, online measurements increase/decrease immediately.

Comparative measurements - Results of deviations



- Relative deviations between both measurement methods for all experimental points max. 20%.
- Exception at 900 °C, λ = 0.40
  (low total tar concentration, but high percentage share of benzene).

- Online measured tar concentrations have only little absolute deviation compared with wet chemical (GC-MS) measurements .
- GC-MS tar concentrations at low gasification temperatures are slightly higher compared to online measured values (more phenols, less benzene (relatively)).



Possible applications

- General (industrial) gasifier monitoring
- Surveillance / Monitoring of gas cleaning / gasifier downstream equipment
- Simplified detection of optimal gasifier / gas cleaning equipment operation point

Thank you for your attention !

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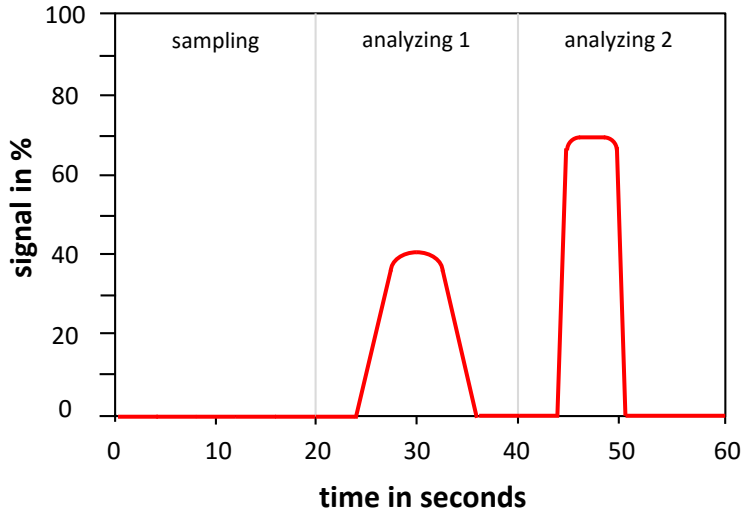
Analyzer manufacturer:
www.ratfisch.de

Ratfisch®
ANALYSENSYSTEME

FID: Signal evaluation and calibration



tar concentration = peak area loop 2 * calibration factor 2 – peak area loop 1 * calibration factor 1

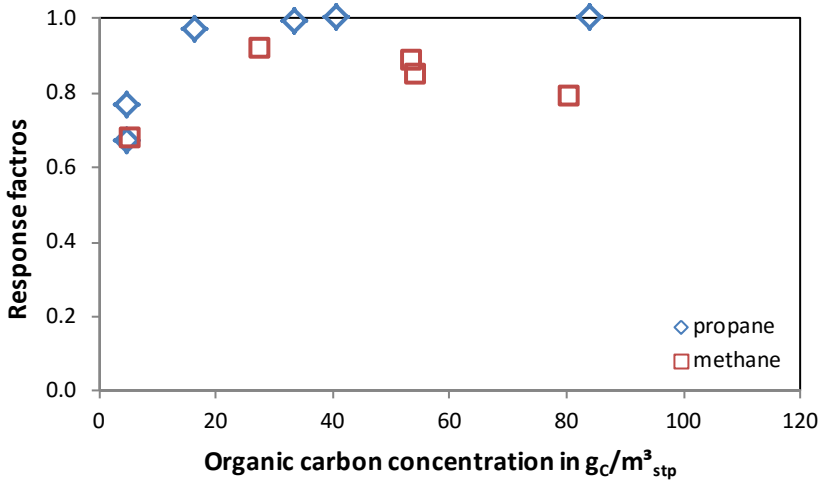
- peak area loop 1/2 = accumulated FID Signal of respective peak area 1/2 (integral)
- calibration factor 1/2 = $\frac{c\text{–}concentration\ of\ calibration\ gas\ in\ \frac{mg_C}{m^3_{stp}}}{peak\ area\ loop\ 1/2}$

FID: Response factors, sensibility and accuracy

Substance	This prototype	Gans/ Baumbach[1]	Wandinger[2]
Propane	1.00	1.00	1.00
Methane	0.90		1.26
Benzene	1.05		1.14
Toluene	1.01	0.86 - 0.99	1.08
Phenol	0.94		
Xylene	1.08	1.04	1.08
Indene	1.07		

- Response factor of common (light) tar substances compared with propane (calibration gas) close to 1.
- Response factor of methane around 0.9
  - ➢ Not relevant for tar measurements because of difference measurement!

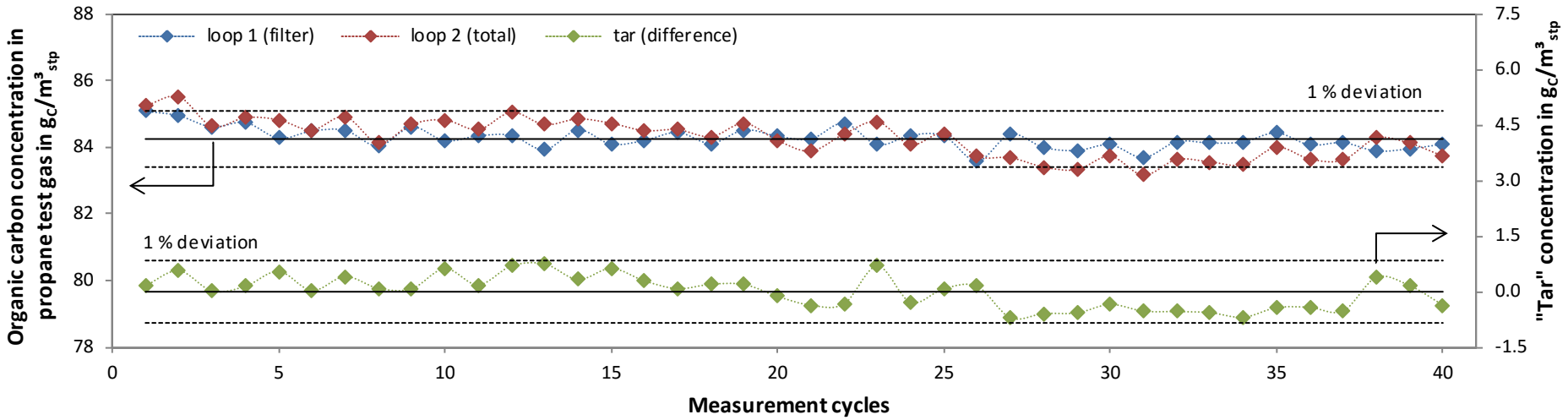
- Propane response factor linear for most of the measurement range.
- Only nonlinear in the lowest sixth of the measurement range.
- Methane response factor not fully linear.
  - ➢ Only crucial for total hydrocarbon concentration (loop 2).



[1] Gans, W.; Baumbach, G.: Kalibrierverfahren zur quantitativen Bestimmung flüchtiger, organischer Substanzen in Abluft und Abgasen mit dem Flammenionisationsdetektor, Fortschrittsberichte VDI Reihe 15, Nr. 32, VDI-Verlag GmbH, Düsseldorf, 1985

[2] Wandinger, H.: Emissionsmesstechnik in gasbetriebenen Geräten und Anlagen, Kohlenwasserstoff-Messtechnik, interner Bericht, Ratfisch GmbH

FID: Response factors, sensibility and accuracy



- Detector test with non-condensable hydrocarbon (propane) shows good repeatability over time.
- Deviation of measurements of both loops within 1 % of total hydrocarbon concentration.
- Difference measurement of both loops shows deviation within a band of 1 % of total hydrocarbon concentration.

Definition of “tar”

- Many / various definitions of “tar” existent.
- Most expedient, most practical and commonly most used definition in the field of biomass gasification (from the European tar measurement standard CEN/TS 15439):

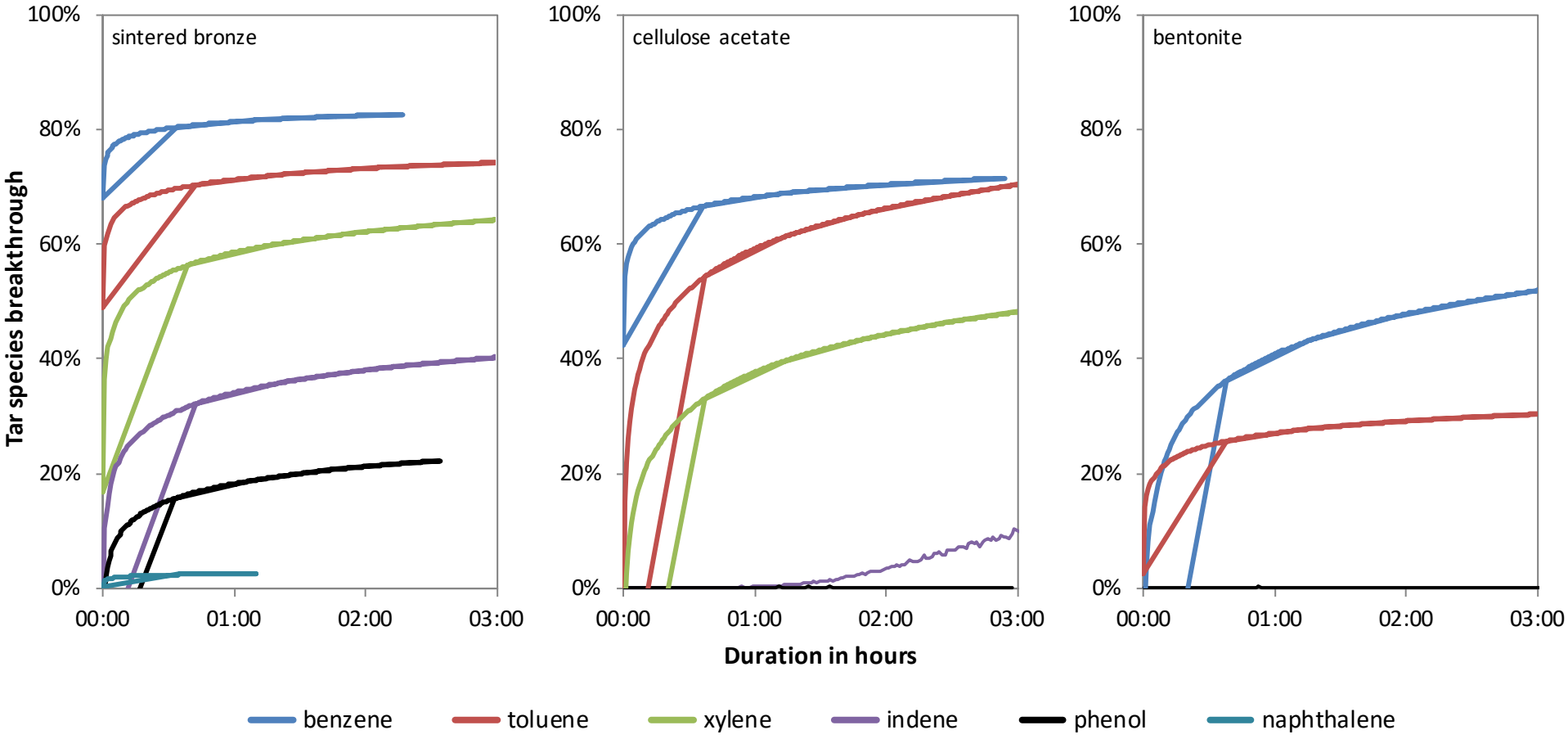
> ***“Generic (unspecific) term for entity of all organic compounds present in the producer gas excluding gaseous hydrocarbons (C_1 to C_6). Benzene is not included in tar.”***

➢ This definition is used for the further development of the IFK online tar measurement technique and therefore for the choice of the tar filter material.

Results of tar filter material selection

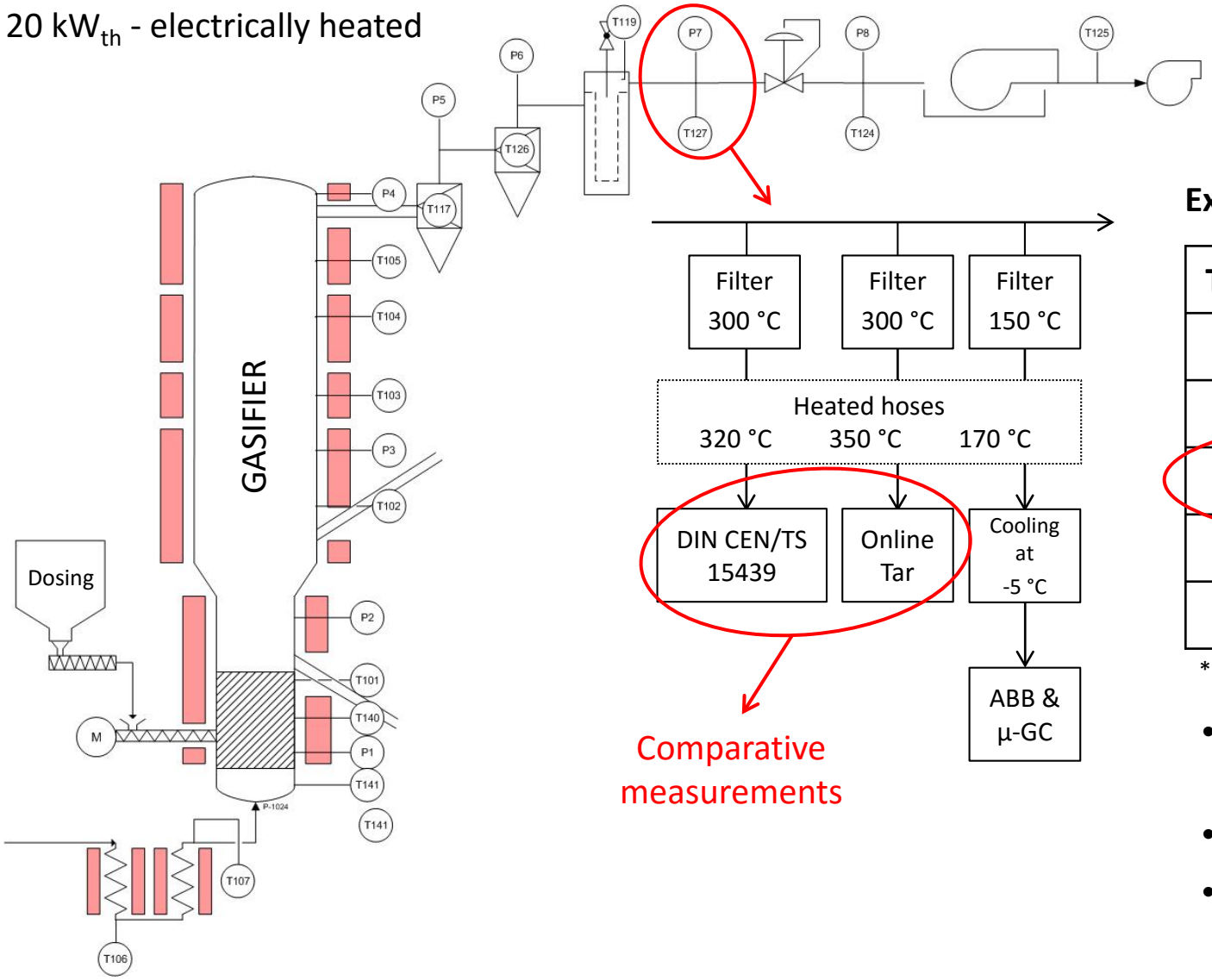
Comparison of sintered bronze, cellulose acetate and "bentonite"

- Laboratory tests with $10 g_C/m^3_{stp}$ of each tar species in Nitrogen (N_2) (carrier gas)
- A filter temperature of 20 °C
- Each with a filter volume of ca. 850mm³



Comparative measurements - Testing facility ELWIRA

20 kW_{th} - electrically heated



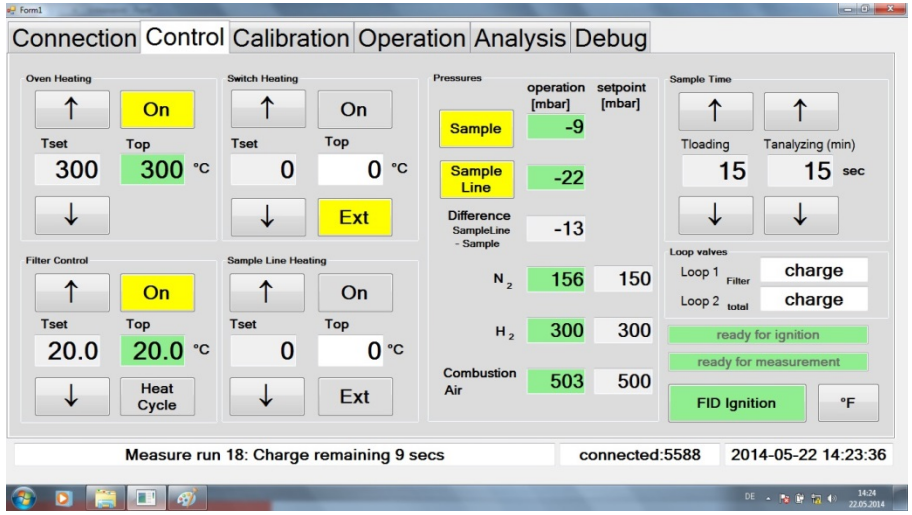
Experimental Set-up

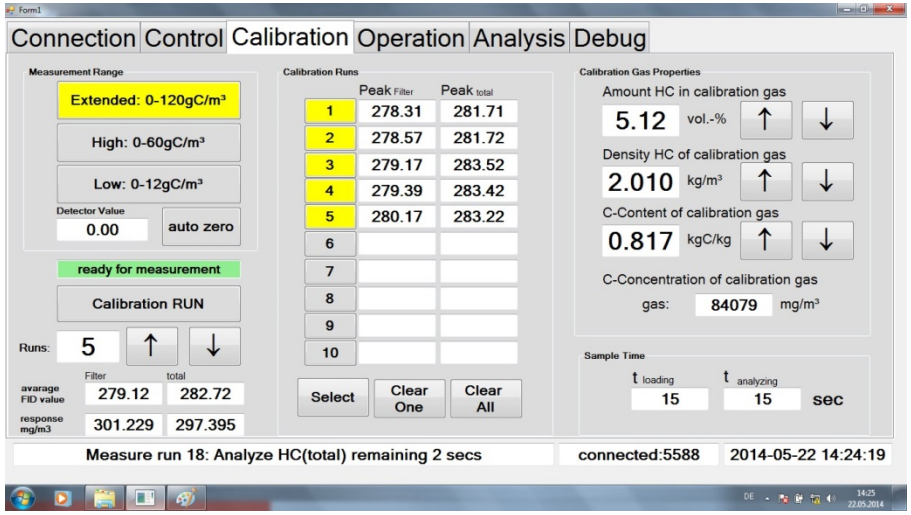
Temperature	Air Ratio
700 °C	0.20
750 °C	0.25
800 °C *	0.30
850 °C	0.35
900 °C	0.40

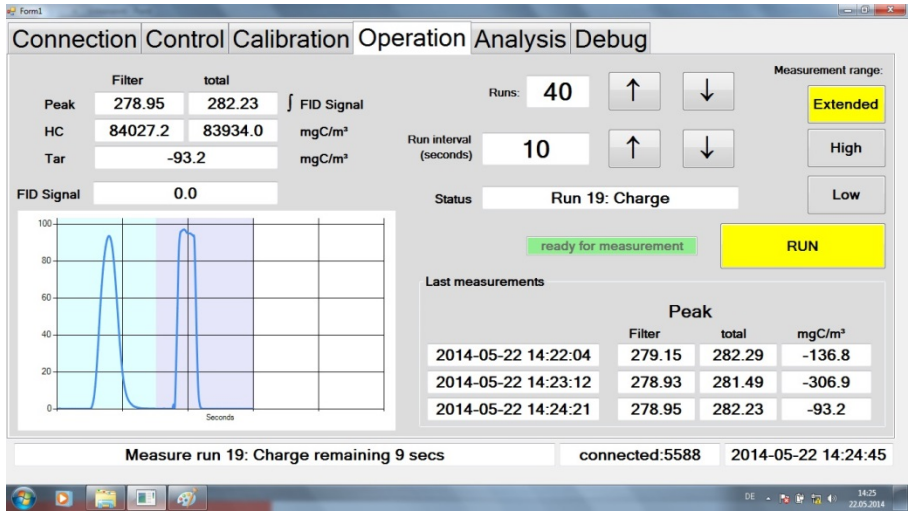
* Base Case

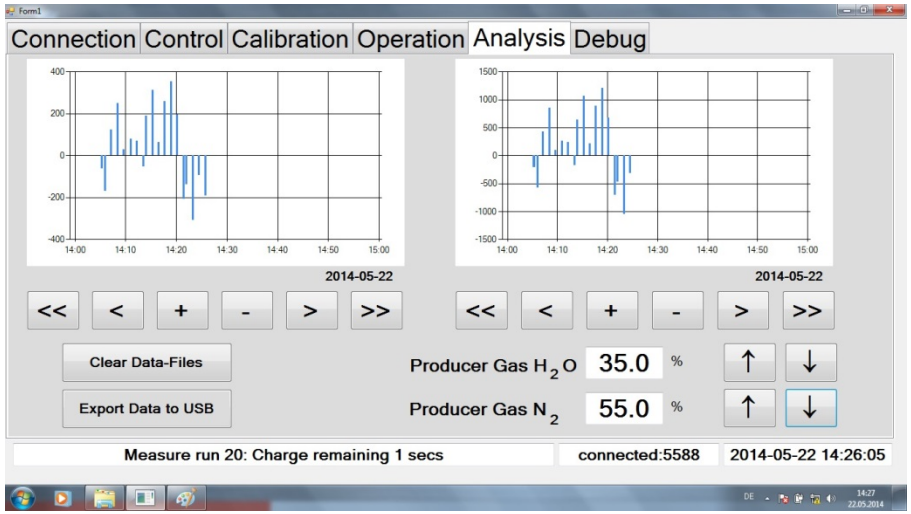
- Fuel: Commercial wood pellets
- Gasification agent: Air
- Duration of at least 6 hours

Operation and control software









Summary and outlook

- New, **further developed prototype** of tar measurement device is successfully manufactured and commissioned.
- **Choice of tar filter material** is fundamental for the accuracy of the results.
  - Bentonite (mineral gas adsorber, a phyllosilicate) shows best hydrocarbon separation behavior according to DIN CEN/TS 15439 so far.
  - Cellulose acetate could achieve realistic results at very low temperatures and limited measurement time.
  - Non-active materials show tar breakthrough already during first measurement cycles.
- **Comparison measurements** with wet chemical method (DIN CEN/TS 15439) for validation of online measurement method and the lab experiments **successfully conducted**.
  - Trends in change of tar production during gasification immediately visible.
  - Absolute deviations within the range of around ±1.5 g_C/m^3_{stp}.
  - Relative deviations within the range of ±20 %. (With the exception of low concentrations; possibly other filter material required.)
- **Further field tests** at different gasifiers **planned**. **Further** possibilities of **comparative measurements** with other methods **are welcomed**.
- Last **improvements** of prototype **underway**.
- Measurement device **commercially available in second half of 2016**.