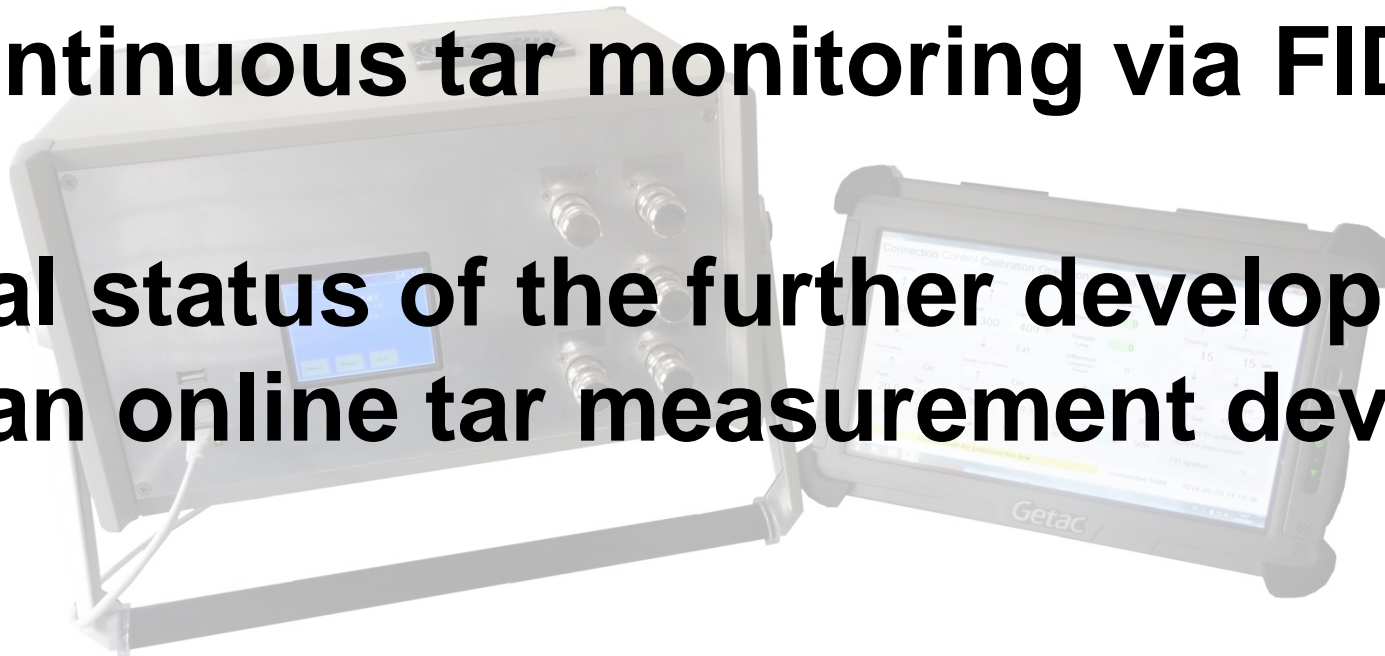
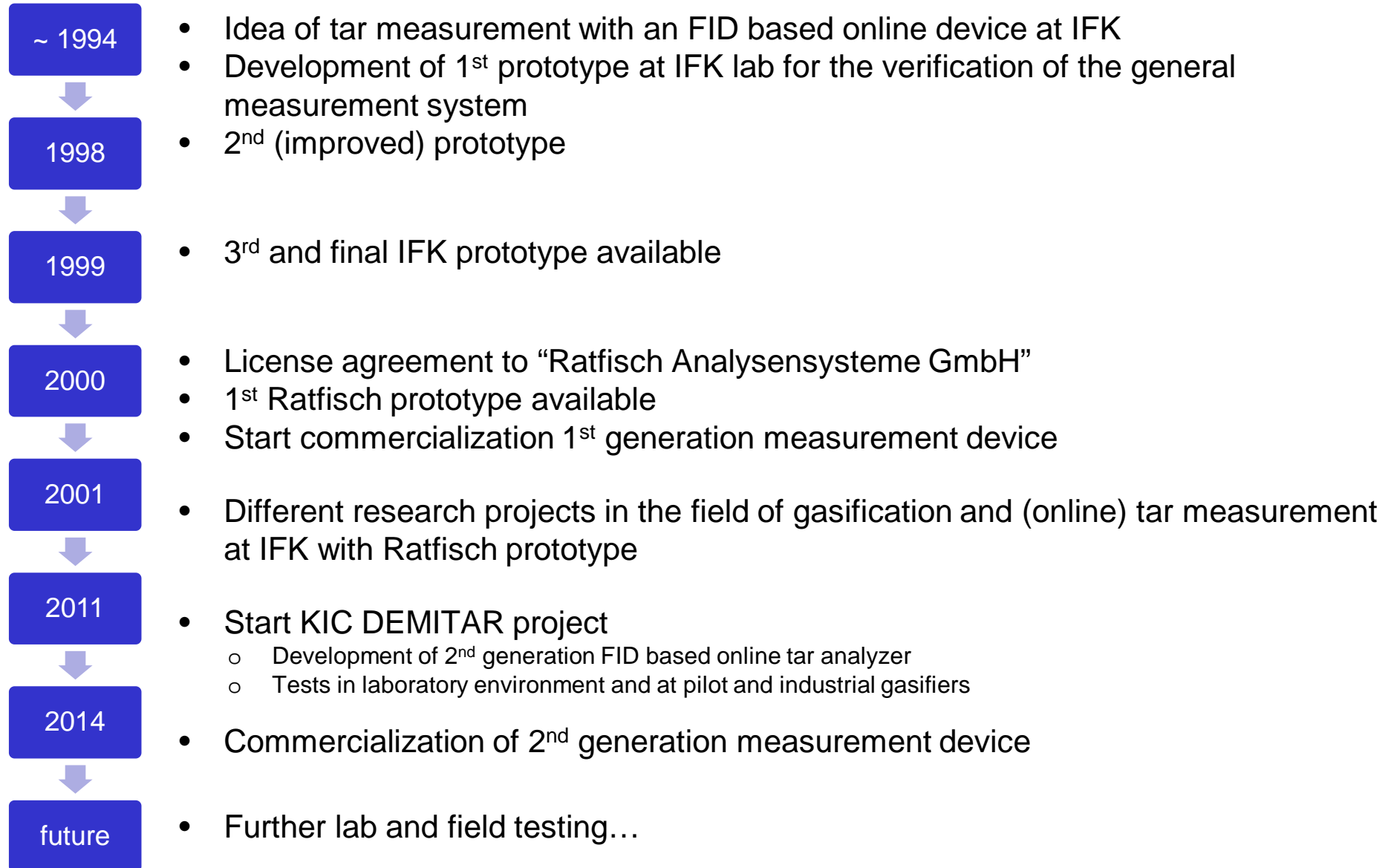


Continuous tar monitoring via FID – Actual status of the further development of an online tar measurement device

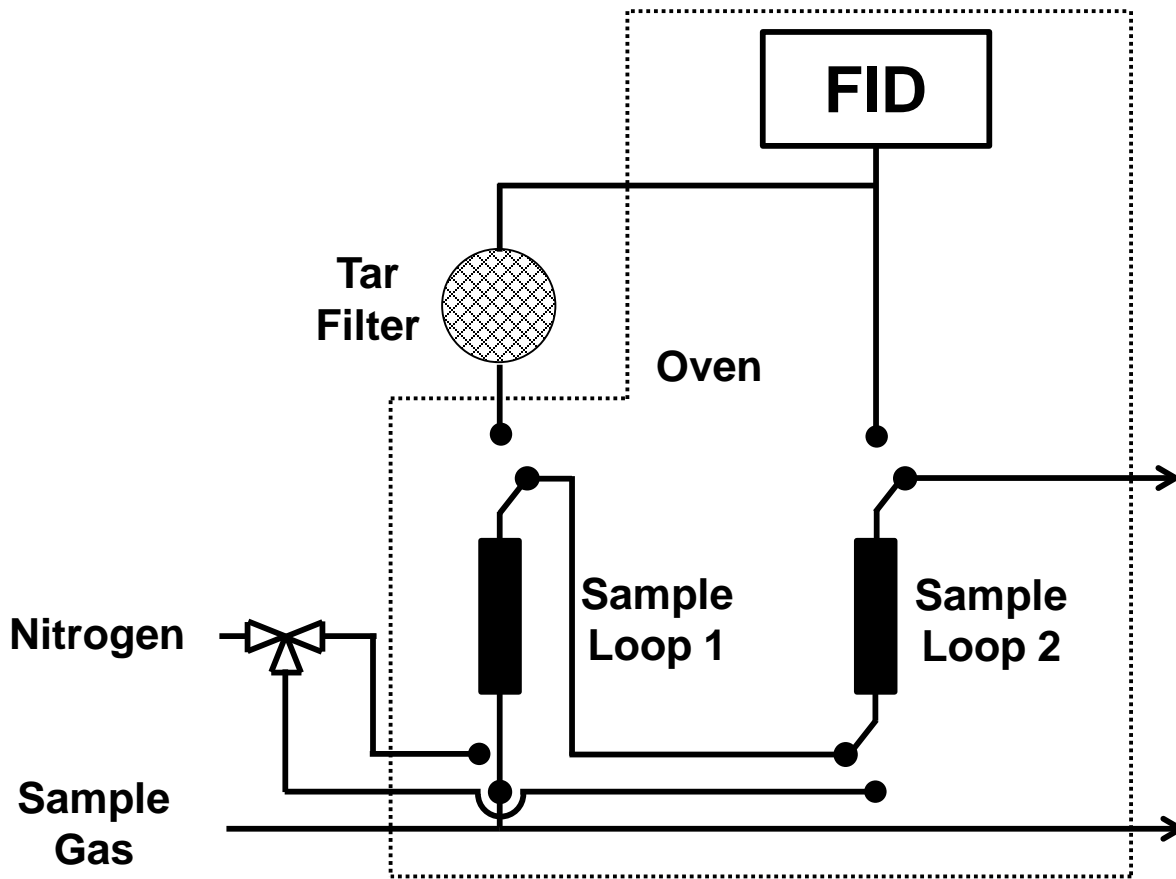


Andreas Gredinger

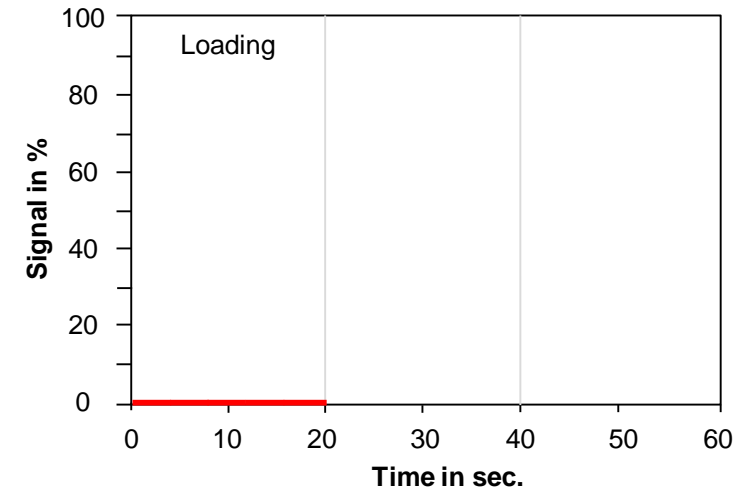
Tar analyzer history



Measurement principle

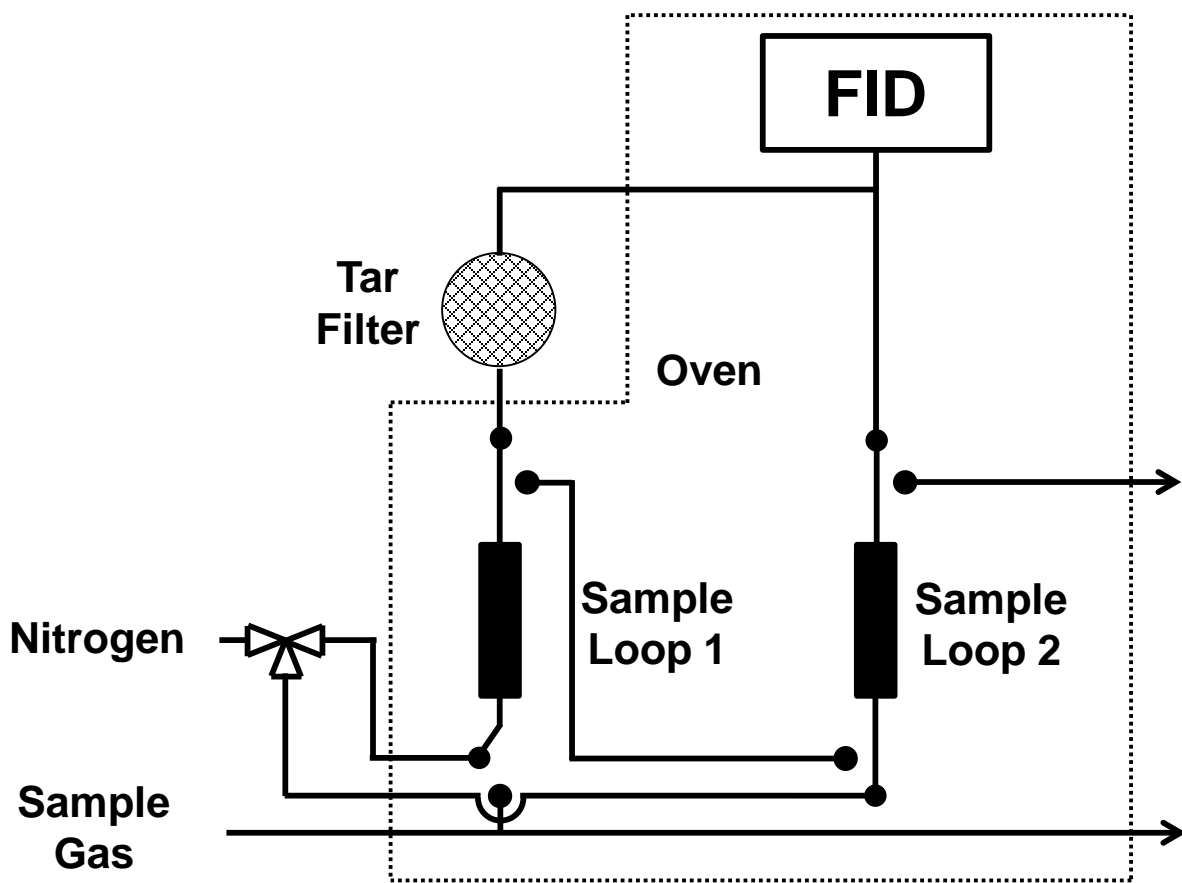


Loading phase

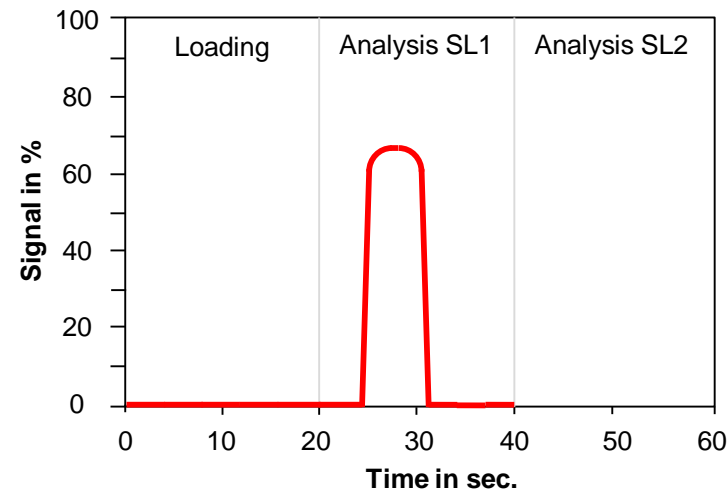


- Sample gas is sucked into the analyzer by venturi nozzles
- Both sample loops are loaded consecutively
- Measurement signal is zero

Measurement principle

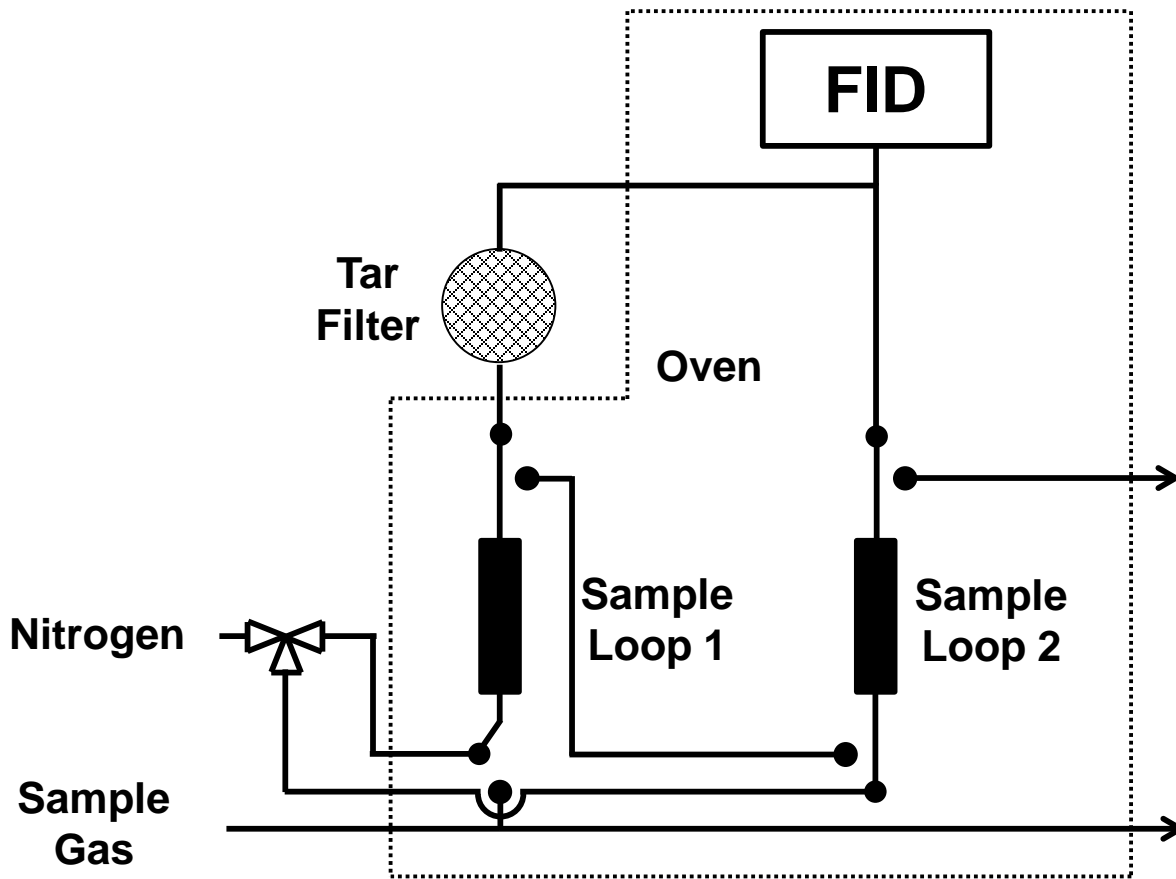


Analyzing phase

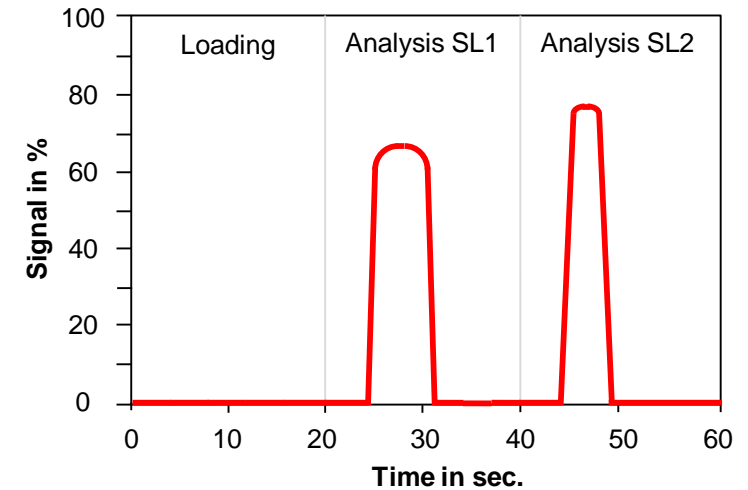


- Sample loops are flushed subsequently to the FID
- Difference between loop 2 and 1 determines the tar content
- Measurable values:
 - Total hydrocarbon content
 - Total non-condensable hydrocarbons
 - Total condensable hydrocarbons

Measurement principle



Analyzing phase



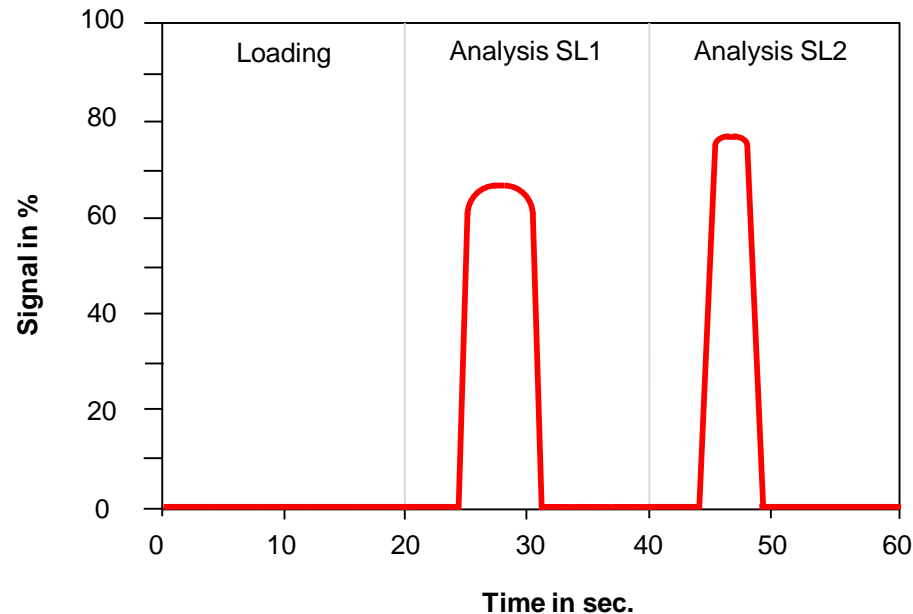
- Sample loops are flushed subsequently to the FID
- Difference between loop 2 and 1 determines the tar content
- Measurable values:
 - Total hydrocarbon content
 - Total non-condensable hydrocarbons
 - Total condensable hydrocarbons

Analyzer specifications



- Measurement principle: Differential measurement of sample gas with and without tars (tars = hydrocarbon condensate on filter!)
- Oven temperature: 300°C
- Tar filter temperature: Adjustable between 20 and 99°C
- Tar filter material: Sintered bronze
- Effective measurement range: 3 zones up to 120g_{Carbon}/m³_{stp} (0-12, 0-60, 0-120)
- Measurement limit: Around 200mg_{Carbon} (Still to be validated!)
- Reproducibility: < 0.5% of the measured value
- Time for sampling and analysis: Minimum 50-60 seconds (per measurement)
- Sample gas flow rate: ~1l/min
- Ambient temperature: 0 to 50°C
- Heating time: Minimum 60 minutes
- Sample gas pressure: -10 to 50mbar

Calculation of tar concentration



$$\text{Concentration}_{\text{tar}} = \text{Peakarea}_{\text{Sample Loop 2}} \cdot \text{Calibration factor}_2 - \text{Peakarea}_{\text{Sample Loop 1}} \cdot \text{Calibration factor}_1$$

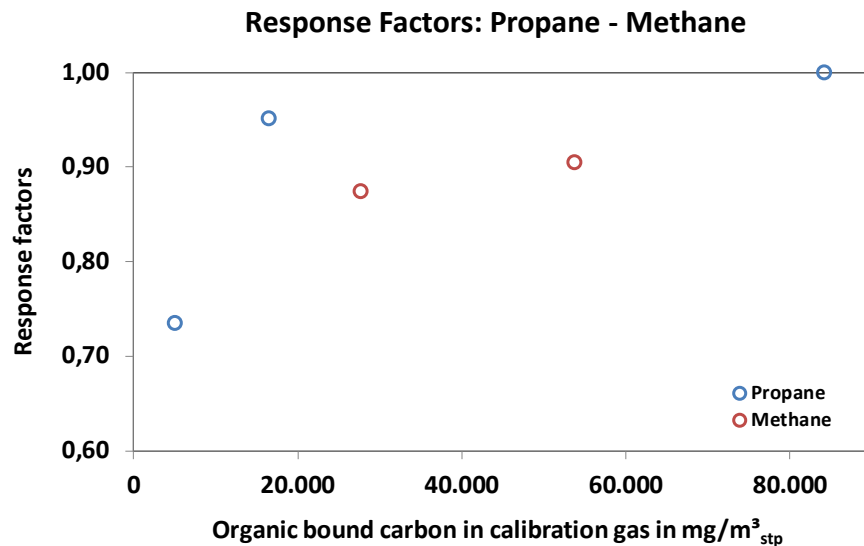
- $\text{Peakarea}_{\text{Sample Loop } 1/2} = \text{Accumulated FID Signal of respective peak area (integral)}$

- $$\text{Calibration factor}_{1/2} = \frac{C - \text{concentration of calibration gas} \left(\frac{\text{mg}_{\text{Carbon}}}{\text{m}^3 \text{stp}} \right)}{\text{Peakarea}_{\text{Sample Loop } 1/2}}$$

Calibration and response factors

From theory:

- The signal of an FID is almost directly proportional to the amount of organically bound carbon.
- Propane is commonly used as calibration gas for FIDs because of the linear detection of different concentrations.



| Substance | <i>Gredinger</i> | <i>Dobson</i> | <i>Wandinger</i> |
|-----------|------------------|----------------------|------------------|
| Propane | 1.00 | 1.00 | 1.00 |
| Methane | ~0.90 | 1.00 | 1.26 |
| Butane | | 0.95 | 1.01 |
| Benzene | | 0.97 | 1.14 |
| Toluene | | 1.00 | 1.08 |
| Xylene | | 0.93 | 1.08 |
| Phenol | | expected to be < 0.8 | |

Use of 5.12% propane in N₂ as reference gas.

Decreasing response factors for propane with decreasing concentrations → Choice of measurement range!

Response factors for methane ~0.9 compared to propane

Operation and control software



Form1

Connection Control Calibration Operation Analysis Debug

Oven Heating: On, Tset: 300, Top: 300 °C

Switch Heating: On, Tset: 0, Top: 0 °C, Ext

Pressures: operation [mbar] -9, setpoint [mbar] -22, Sample Line -13

Sample Time: 15, 15 sec

Filter Control: On, Tset: 20.0, Top: 20.0 °C

Sample Line Heating: On, Tset: 0, Top: 0 °C, Ext

Loop valves: Loop 1 Filter charge, Loop 2 total charge

ready for ignition, ready for measurement

FID Ignition

Measure run 18: Charge remaining 9 secs, connected:5588, 2014-05-22 14:23:36

Form1

Connection Control Calibration Operation Analysis Debug

Measurement Range: Extended: 0-120gC/m³, High: 0-60gC/m³, Low: 0-12gC/m³

Detector Value: 0.00, auto zero

ready for measurement

Calibration RUN

Runs: 5

| Peak | Filter | Peak total |
|------|--------|------------|
| 1 | 278.31 | 281.71 |
| 2 | 278.57 | 281.72 |
| 3 | 279.17 | 283.52 |
| 4 | 279.39 | 283.42 |
| 5 | 280.17 | 283.22 |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |

Calibration Gas Properties: Amount HC in calibration gas: 5.12 vol.-%, Density HC of calibration gas: 2.010 kg/m³, C-Content of calibration gas: 0.817 kgC/kg, C-Concentration of calibration gas gas: 84079 mg/m³

Sample Time: t loading: 15, t analyzing: 15 sec

average FID value: 279.12, 282.72, response mg/m3: 301.229, 297.395

Measure run 18: Analyze HC(total) remaining 2 secs, connected:5588, 2014-05-22 14:24:19

Form1

Connection Control Calibration Operation Analysis Debug

Peak: Filter 278.95, total 282.23, HC 84027.2, 83934.0 mgC/m³, Tar -93.2 mgC/m³

FID Signal: 0.0

Runs: 40, Run interval (seconds): 10

Measurement range: Extended, High, Low

Status: Run 19: Charge, ready for measurement, RUN

Last measurements:

| | Filter | Peak total | mgC/m ³ |
|---------------------|--------|------------|--------------------|
| 2014-05-22 14:22:04 | 279.15 | 282.29 | -136.8 |
| 2014-05-22 14:23:12 | 278.93 | 281.49 | -306.9 |
| 2014-05-22 14:24:21 | 278.95 | 282.23 | -93.2 |

Measure run 19: Charge remaining 9 secs, connected:5588, 2014-05-22 14:24:45

Form1

Connection Control Calibration Operation Analysis Debug

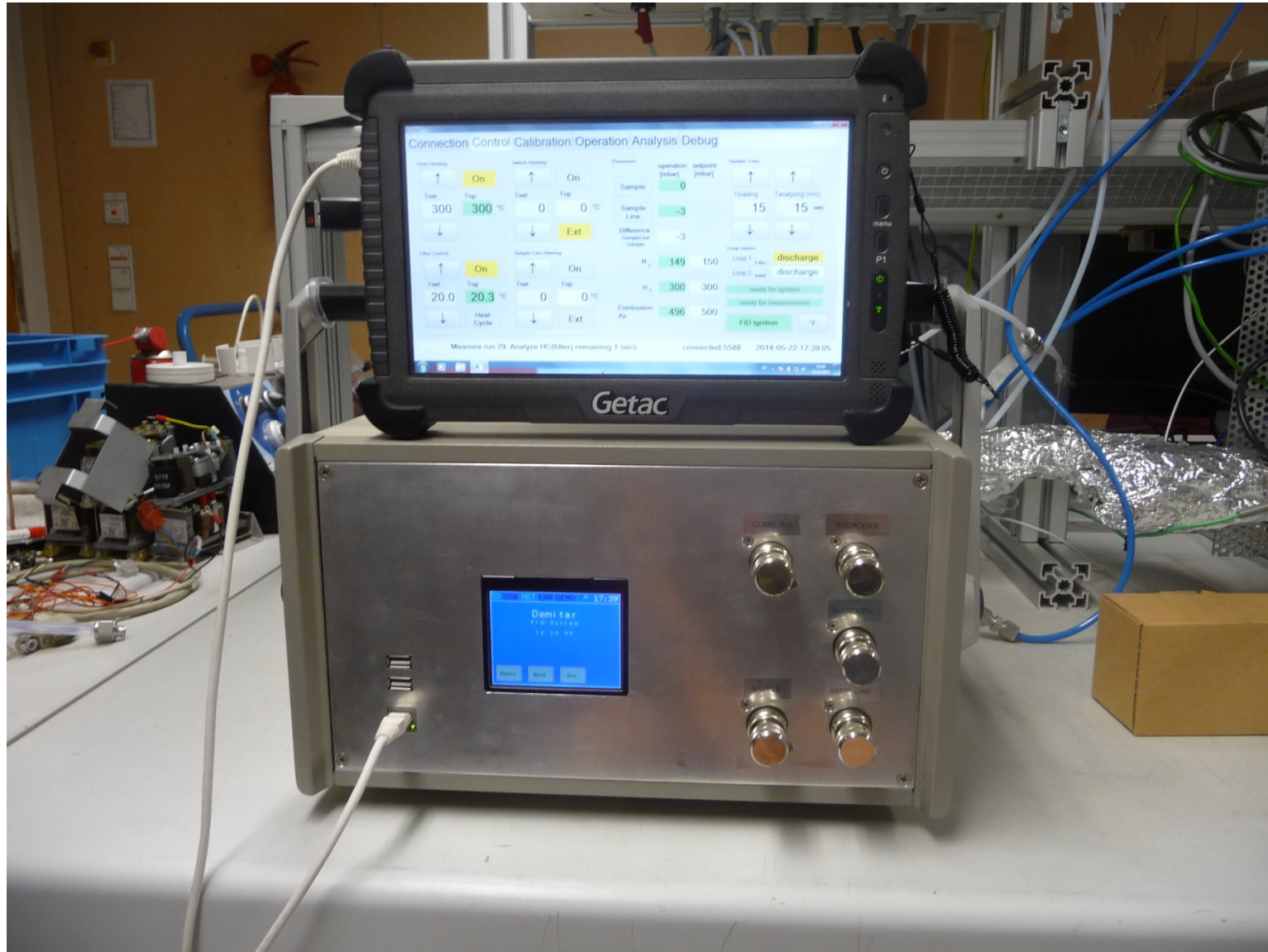
2014-05-22

2014-05-22

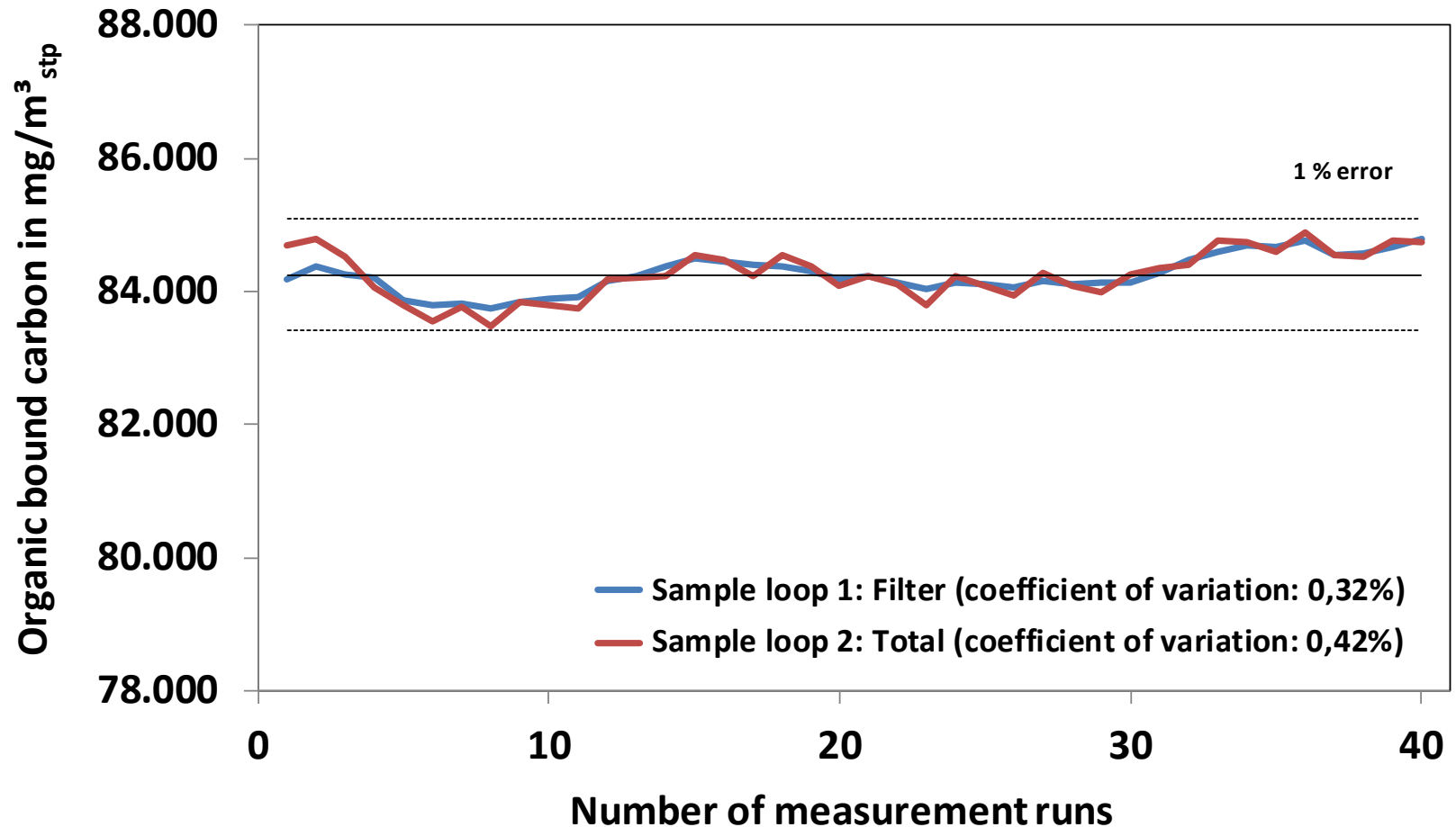
Producer Gas H₂O: 35.0 %, Producer Gas N₂: 55.0 %

Measure run 20: Charge remaining 1 secs, connected:5588, 2014-05-22 14:26:05

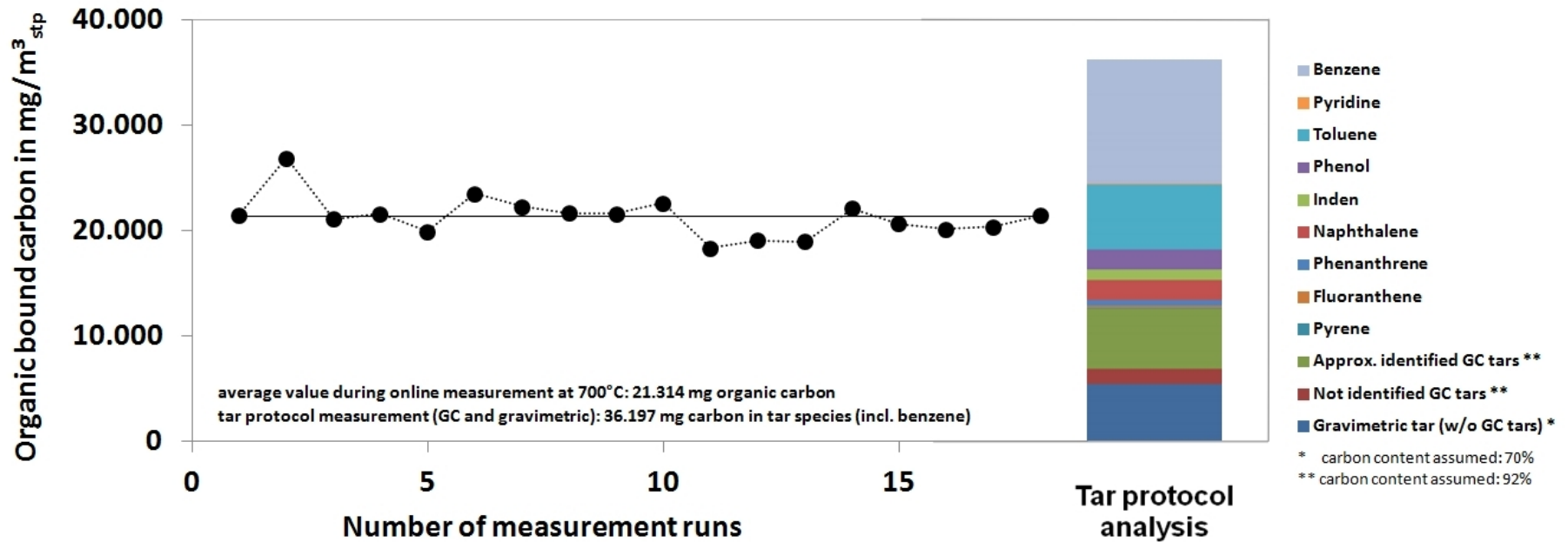
2nd gen. online tar measurement device



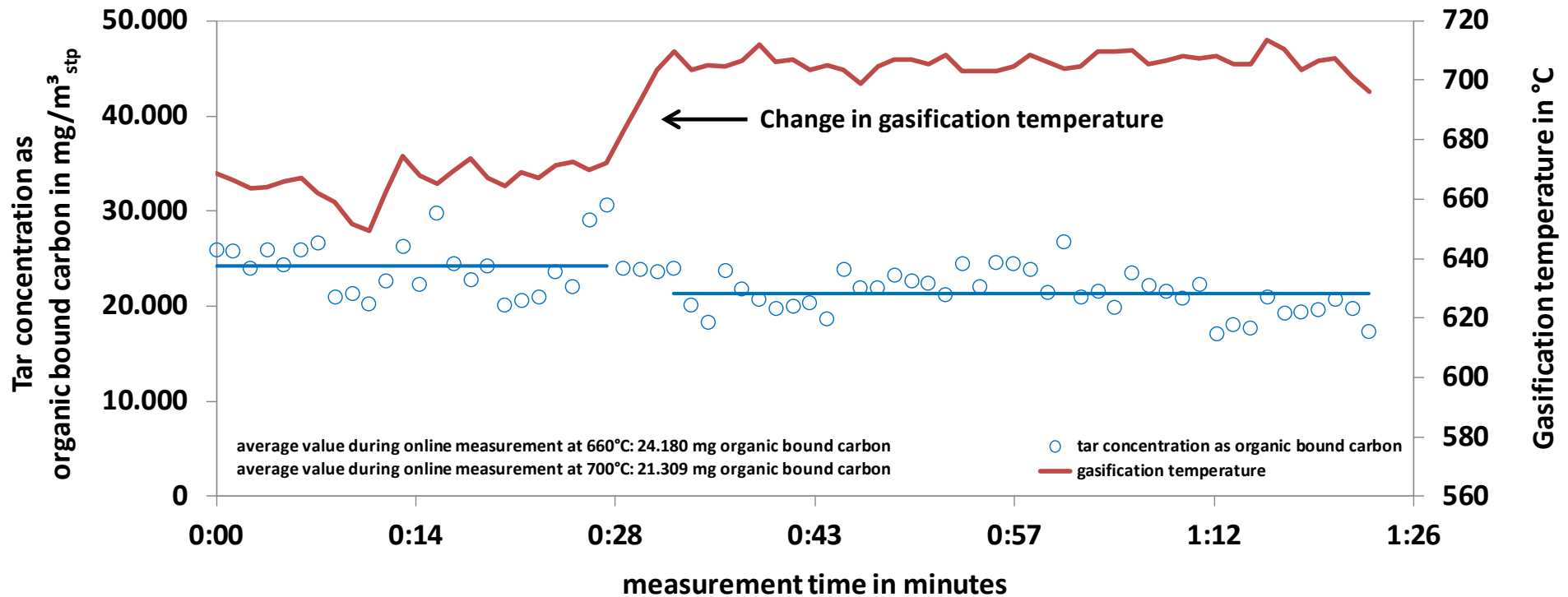
Measurement Accuracy and Repeatability



Comparison with results from tar protocol



Tar monitoring - Change in gasification temp.



Possible applications

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

Future experiments and analyzer validation



- Further tests with calibration gas for general analyzer validation
 - Influence of general analyzer settings to measurement signal
- Tests with single tar species (generated with own „tar generator“):
 - Identification of single response factors
 - Identification of filter breakthrough at different concentrations
- Tests with hydrogen in sample gas to detect influence on analyzer signal
- Tests with steam in sample gas to detect influence on analyzer signal and filter behavior
- Long term tests at IFK pilot plant to see time of filter breakthrough
- Tests at IFK pilot plants to compare measurements with tar protocol and SPA at different gasification processes
- Tests at CIUDEN gasifier (Ponferrada/Spain) in September

Interested parties are always welcome to visit IFK during test time!

Thank you for your attention !

Contact person: Andreas Gredinger

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Analyzer manufacturer:
www.ratfisch.de

