



**University of Stuttgart**

Institute of Combustion and Power Plant Technology

Prof. Dr. techn. G. Scheffknecht

# Syngas measurement at IFK

**Whats our status?  
What are we interested in?**

Gas Analysis Workshop, 16 June 2017, Stockholm

**M. Schmid, G. Waizmann, A. Gredinger,  
D. Schweitzer, R. Spörl,  
G. Scheffknecht**



**University of Stuttgart**

Institute of Combustion and Power Plant Technology

Prof. Dr. techn. G. Scheffknecht

## Department of Decentralized Energy Conversion

### **Fluidized Bed Processes**

- ✓ Gasification
- ✓ Combustion
- ✓ Calcium Looping (CaL)
- ✓ Chemical Looping (CLC)

### **Fuels**

- ✓ Biomass, Residues
- ✓ Coal

### **Gas measurement**

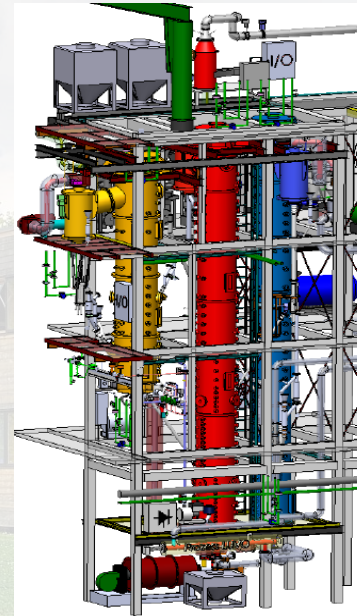
- ✓ Permanent gases
- ✓  $H_2S$ ,  $NH_3$ ,  $HCl$
- ✓ tar

20 kW<sub>th</sub> electrically heated DFB System



**Tar generator test rig**

200 kW<sub>th</sub> DFB Pilot Facility



### **Flameless oxidation**

- ✓ Liquid fuels
- ✓ Producer gas

### **Gas/ Tar Analysis**

### **Cold Model Analyses**

### **Modelling and Simulation**

# Fuels used in the experimental work

Wood pellets



Straw pellets



Dried sewage sludge

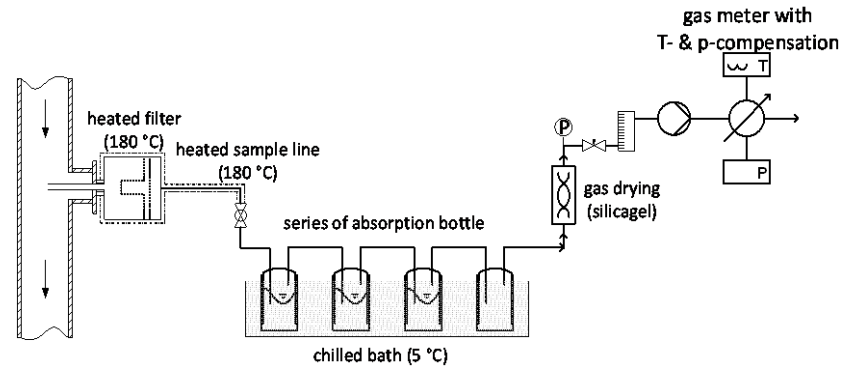


in wt-%	proximate analysis		elemental analysis waf					
	moisture	ash wf	C	H	O	N	S	Cl
<b>wood pellets</b>	9.8	0.1	50.8	6.3	42.9	-	-	-
<b>straw pellets</b>	10.3	5.7	49.3	6.4	42.8	0.8	0.2	0.5
<b>dried sewage sludge</b>	6.5	47.6	51.0	6.9	32.1	7.5	2.4	0.2

# Measurement techniques at IFK



- IFKs Online FID Tar Analyzer



- Wet chemical  $H_2S$ ,  $NH_3$ ,  $HCl$
- Tar protocol

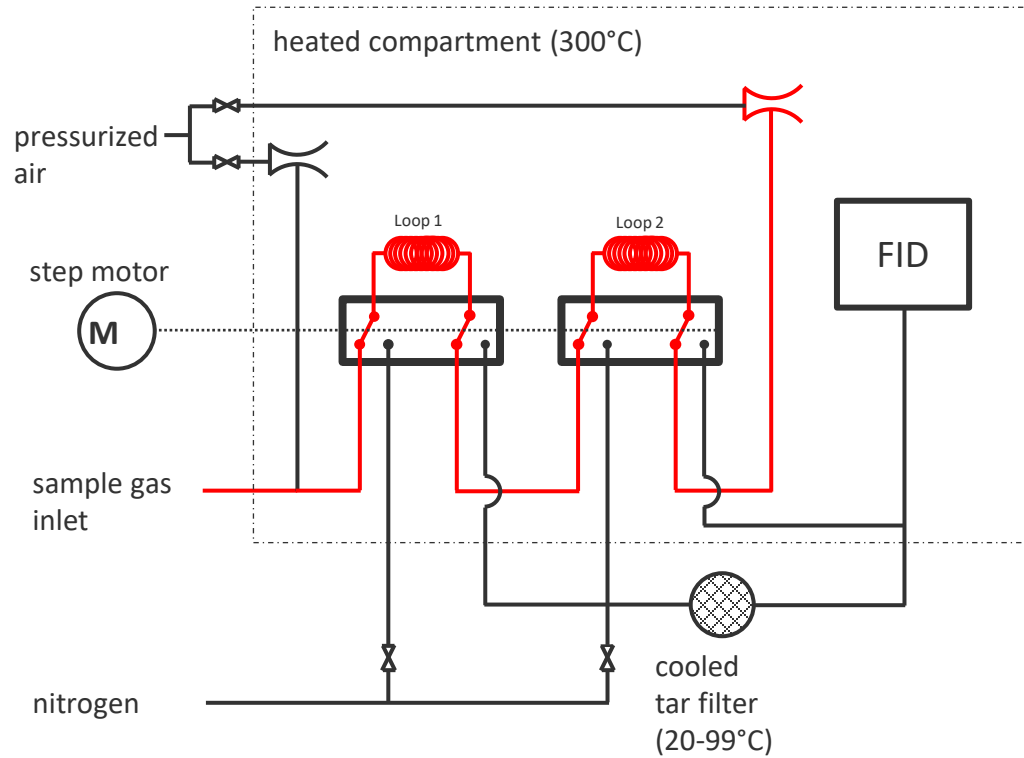


- Gaset FTIR Analyzer

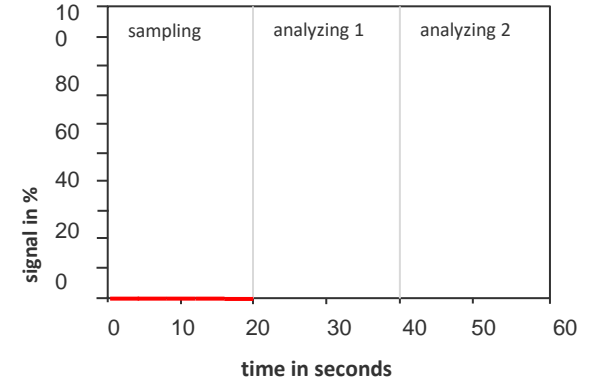
# Final device



# Measurement principle

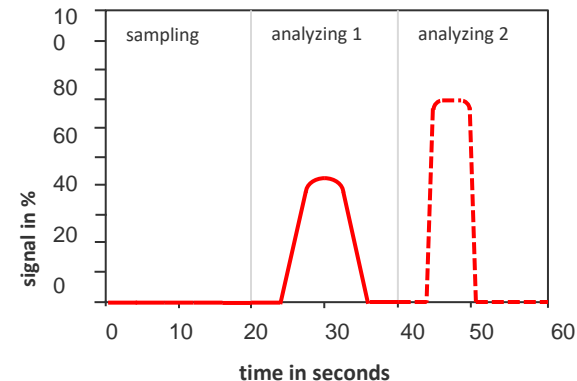
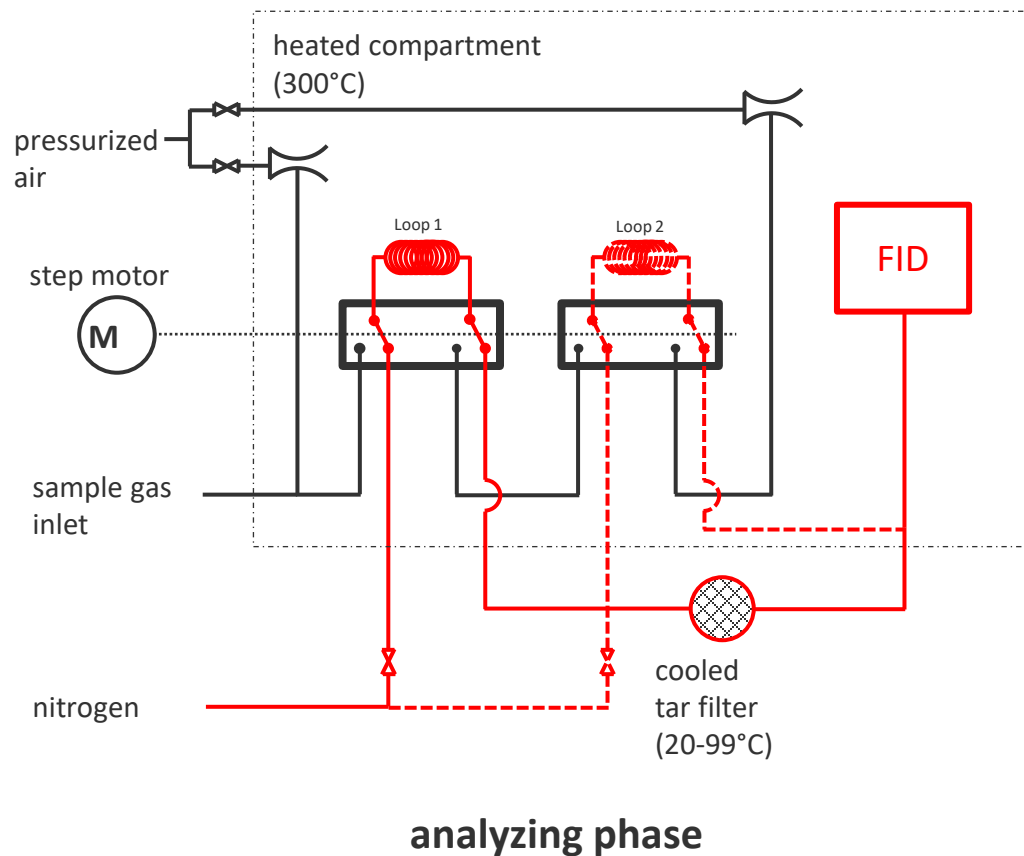


sampling phase



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

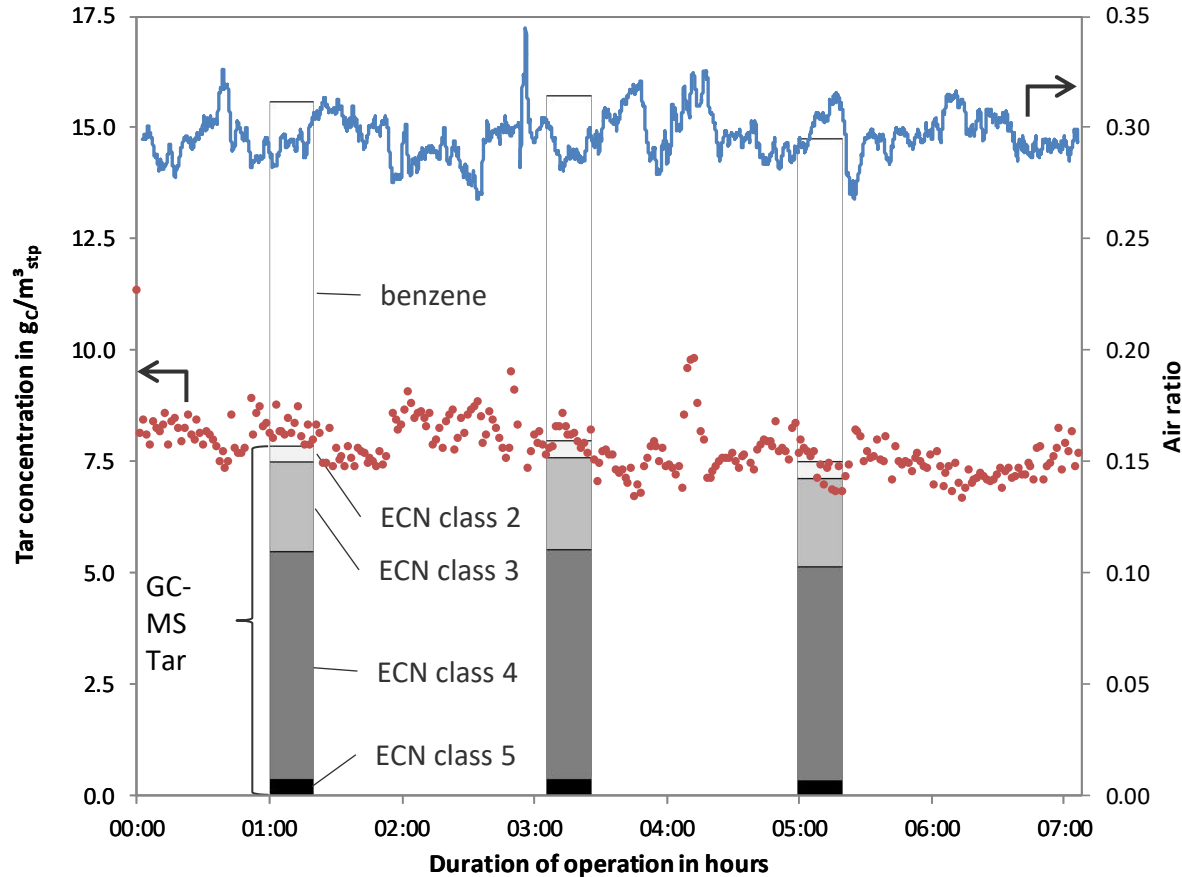
## Measurement principle



- 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)

## Comparative measurements - Results base case

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

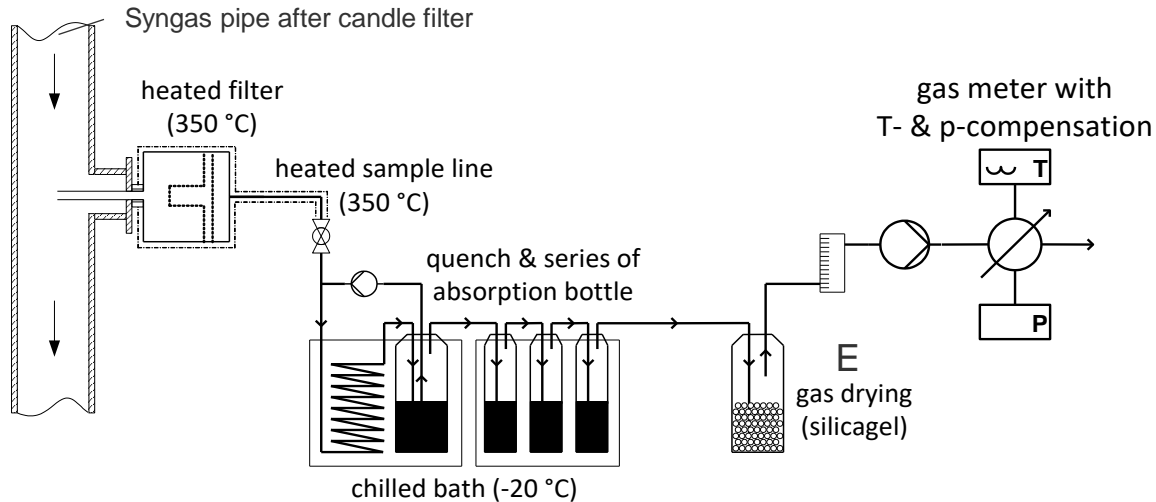


- Time of operation of more than 7 hours.
- 3 comparative measurements.
- One online measurement cycle had a duration of around 84 seconds (300 cycles).
- Online measured tar concentrations follow the wet chemical comparative measurements very close over the total time of operation.
- Air ratio fluctuates 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.



# Methods – wet chemical impurity and tar measurement

## Tar measurement arrangement



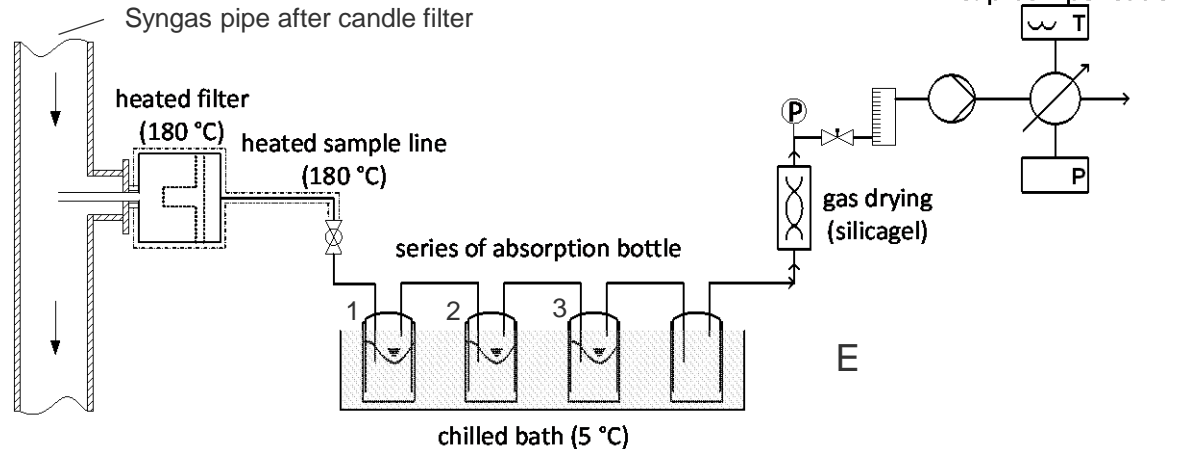
DIN CEN/TS15439 (tar protocol)

- Absorption liquid: Isopropanol
- Liquid is sampled and analysed Gravimetrically (GC-MS also possible)

Further explanations:  
**Visual Presentation 2CV.3.31**  
D. Schweitzer, M. Schmid, A. Gredinger, R. Spörl., G. Scheffknecht:  
Gasification of waste biomasses:  
Measurement of pollutants in product gas, EUBCE 2017

# Methods – wet chemical impurity and tar measurement

## H<sub>2</sub>S, NH<sub>3</sub>, HCl measurement for gasification



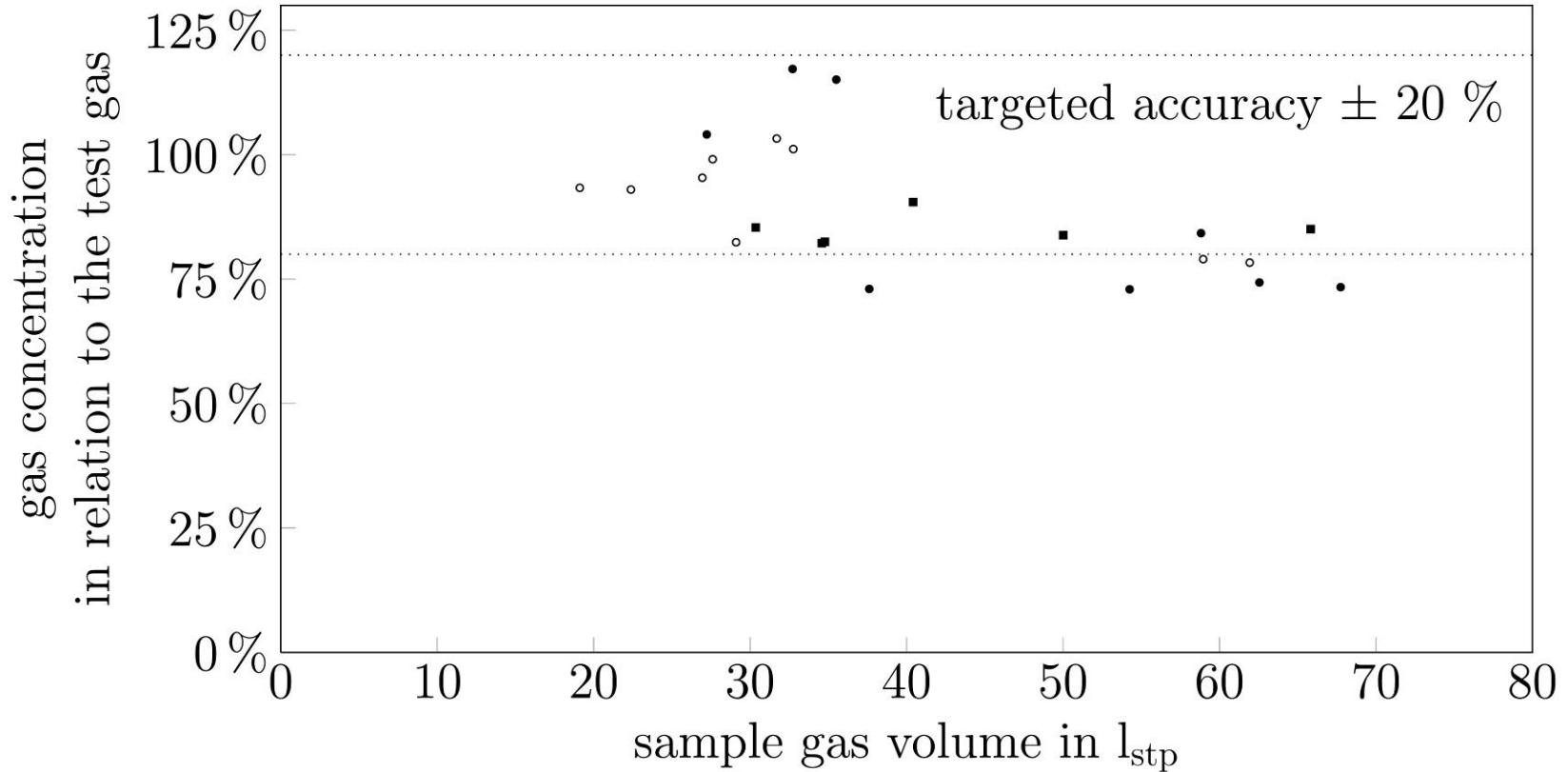
Further explanations:  
**Visual Presentation 2CV.3.31**  
 D. Schweitzer, M. Schmid, A. Gredinger, R. Spörl., G. Scheffknecht:  
 Gasification of waste biomasses:  
 Measurement of pollutants in product gas, EUBCE 2017

- H<sub>2</sub>S: DIN 51855-4 (iodometric titration)
- NH<sub>3</sub>: DIN EN ISO 11732 (indophenol method)
- HCl: Coulometric analysis

	H <sub>2</sub> S	NH <sub>3</sub>	HCl
<b>Tar removal solution</b> <i>bottle 1</i>	Isopropanol, H <sub>2</sub> SO <sub>4</sub>	Isopropanol, NaOH	-
<b>absorption solution</b> <i>bottle 2+3</i>	Zinc acetate	1 mol/l H <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> O

# Accuracy of wet chemical measurement techniques

◦  $y_{H_2S}$  •  $y_{NH_3}$  ▪  $y_{HCl}$



# FTIR experience for syngas/offgas analysis

## Gasmet DX4000



Gas	Measured species	Result quality
Synthetic flue gas	HCl, N <sub>2</sub> O, standard gases	Very good
Flue gas	HCl, N <sub>2</sub> O, standard gases	good in accordation to other analyzers
H <sub>2</sub> O, tar model compounds, N <sub>2</sub> from test gas generator	H <sub>2</sub> O, phenole, xylene	Very good
Catalytic reforming of test gas from test gas generator	H <sub>2</sub> O, phenole, xylene All possible reforming products	promising / ? Values make sense, but higher residual values, some hydrocarbons have odd values (to high)
raw syngas from fluidized bed gasification	H <sub>2</sub> O, permanent gases, hydrocarbons, tars, pollutants	Not so good / ? Permanent gases do not fully match with other analyzers, some spezies have odd values, high residual values <i>Positive: device was not damaged!</i>

# What is IFK interested in?



- Tar measurement
  - Joined comparison measurements
  - Improving the handling of the tar protocol
  - How to deal with heteroatoms (S, N, Cl) in tar analysis
    - Gravimetric: What about the salts?
    - GC-MS: how to detect S, N, Cl containing tar species?
- FTIR
  - Experience exchange
  - How to evaluate data from raw syngas measurement



**University of Stuttgart**  
Germany

**Thank you!**



**M.Sc. Max Schmid**

e-mail [max.schmid@ifk.uni-stuttgart.de](mailto:max.schmid@ifk.uni-stuttgart.de)

phone +49 711 685-63394

fax +49 711 685-63491

University of Stuttgart  
Institute of Combustion and Power Plant Technology  
Pfaffenwaldring 23 • 70569 Stuttgart • Germany

