

# Gas Analysis Workshop 2017 Berlin

## test gas generators

*Why do we need test gases?*

- *Test on-line tools under known and stable conditions*
- *Calibrate on-line tools*
- *Test adsorbers and (catalytic) reformers*

CHREV. 171

## GENERATION OF STANDARD GASEOUS MIXTURES

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Sources:

J. Namieśnik, “Generation of standard gaseous mixtures,” *Journal of Chromatography A*, vol. 300, pp. 79–108, 1984

See also:

Yue Li, Tim Täffner, Michael Bischoff, and Bernd Niemeyer, “Test Gas Generation from Pure Liquids: An Application-Oriented Overview of Methods in a Nutshell,” *International Journal of Chemical Engineering*, vol. 2012, Article ID 417029, 6 pages, 2012. doi:10.1155/2012/417029

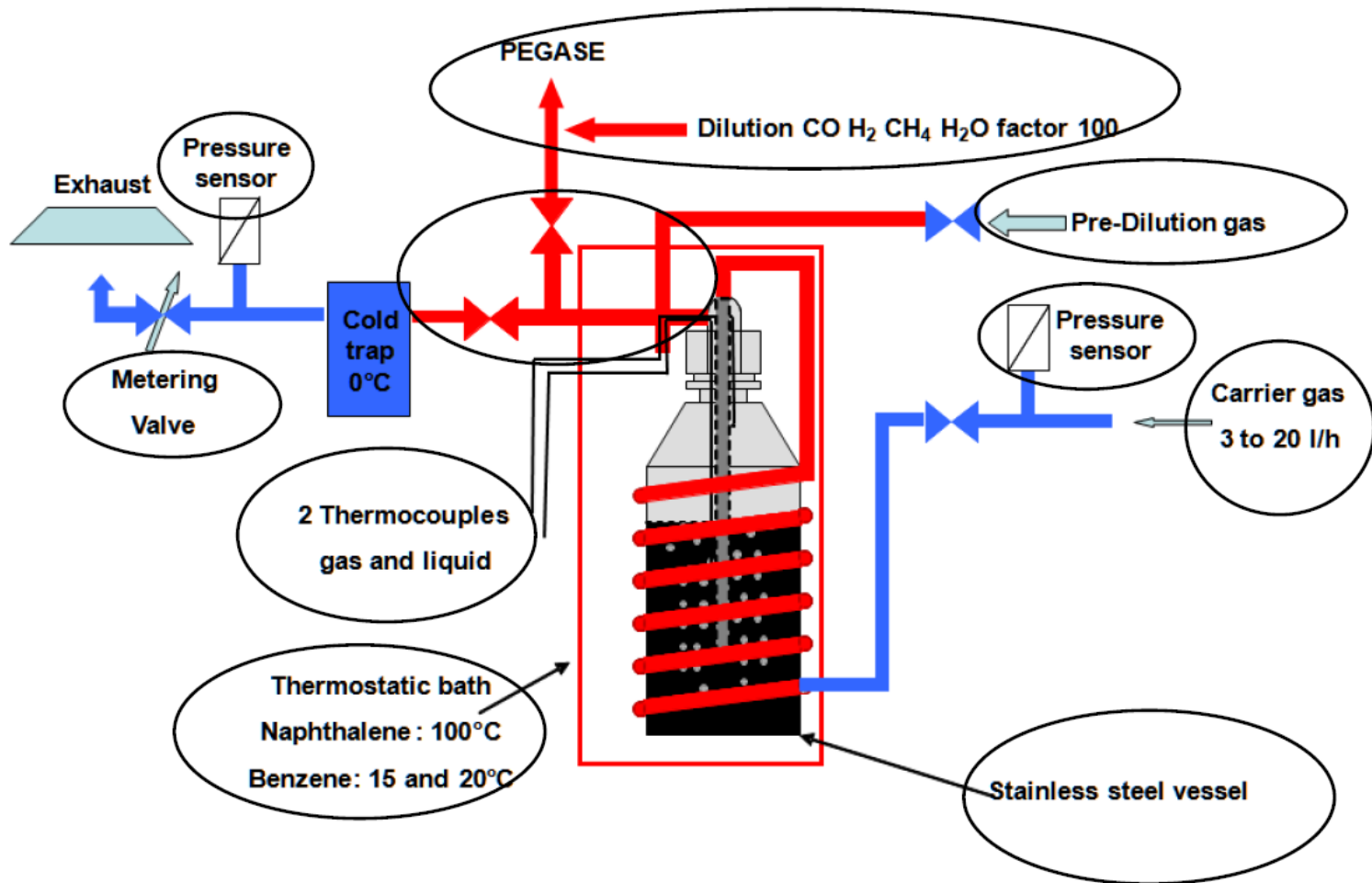
# Gas Analysis Webinars

## test gas generators

March 21<sup>st</sup> 2014

- *Y. Neubauer, TU Berlin, Germany*  
*Brief introduction and scope of this webinar*
- F. Defoort, cea, Grenoble, France  
Two types of test gas generator systems based on entrainment of saturated gas and on syringe into vacuum vessel
- M. Reinikainen, vtt, Espoo, Finland  
Tar generation by ethene pyrolysis
- C. Hamel, C. Unger, Fraunhofer UMSICHT, Oberhausen, Germany  
Model gas made by Fraunhofer UMSICHT –  
Test gas for catalyst examinations including steam and tar compounds

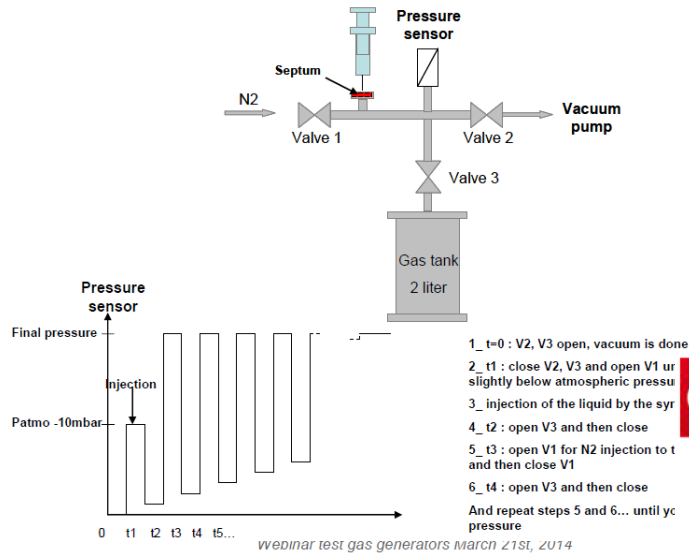
## 2-System based on entrainment of saturated gas



### 3- System based on syringe into vacuum vessel

- Need a gas generator
  - For pure component (gas not polluted by solvent)
  - With about 6 liters of calibrated gas

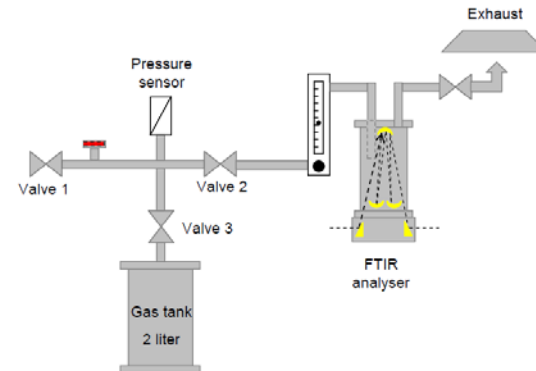
Filling the Gas Tank



liten

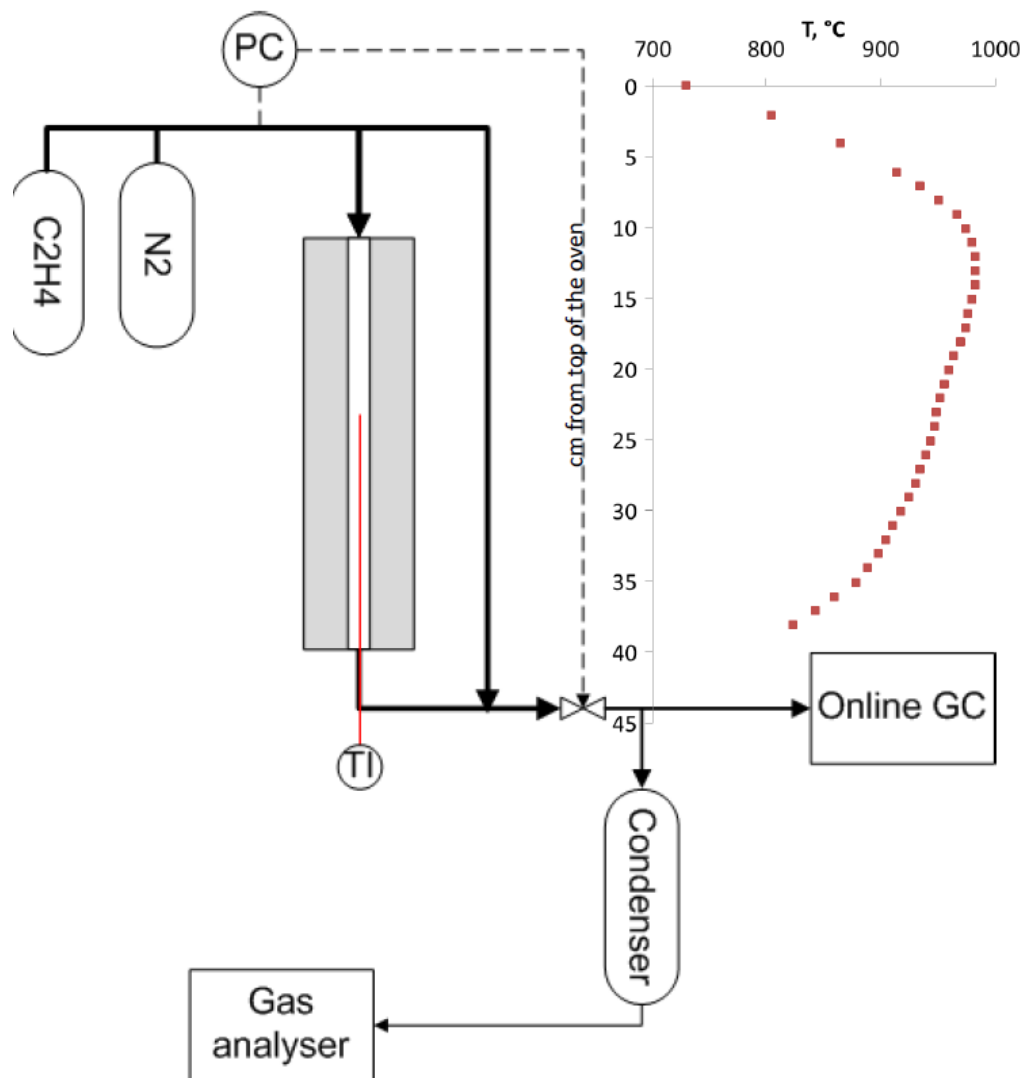
### 3- System based on syringe into vacuum vessel

FTIR calibration



- Syringe + vacuum system validated by comparing with:
  - Other gas generator (bubbling) for steam, acetaldehyde, methanol
  - FTIR integrated values with cold trap residue + GC-MS
- Problem with formaldehyde (adsorption on the surface of the vessel in stainless steel)

# Laboratory set-up and conditions

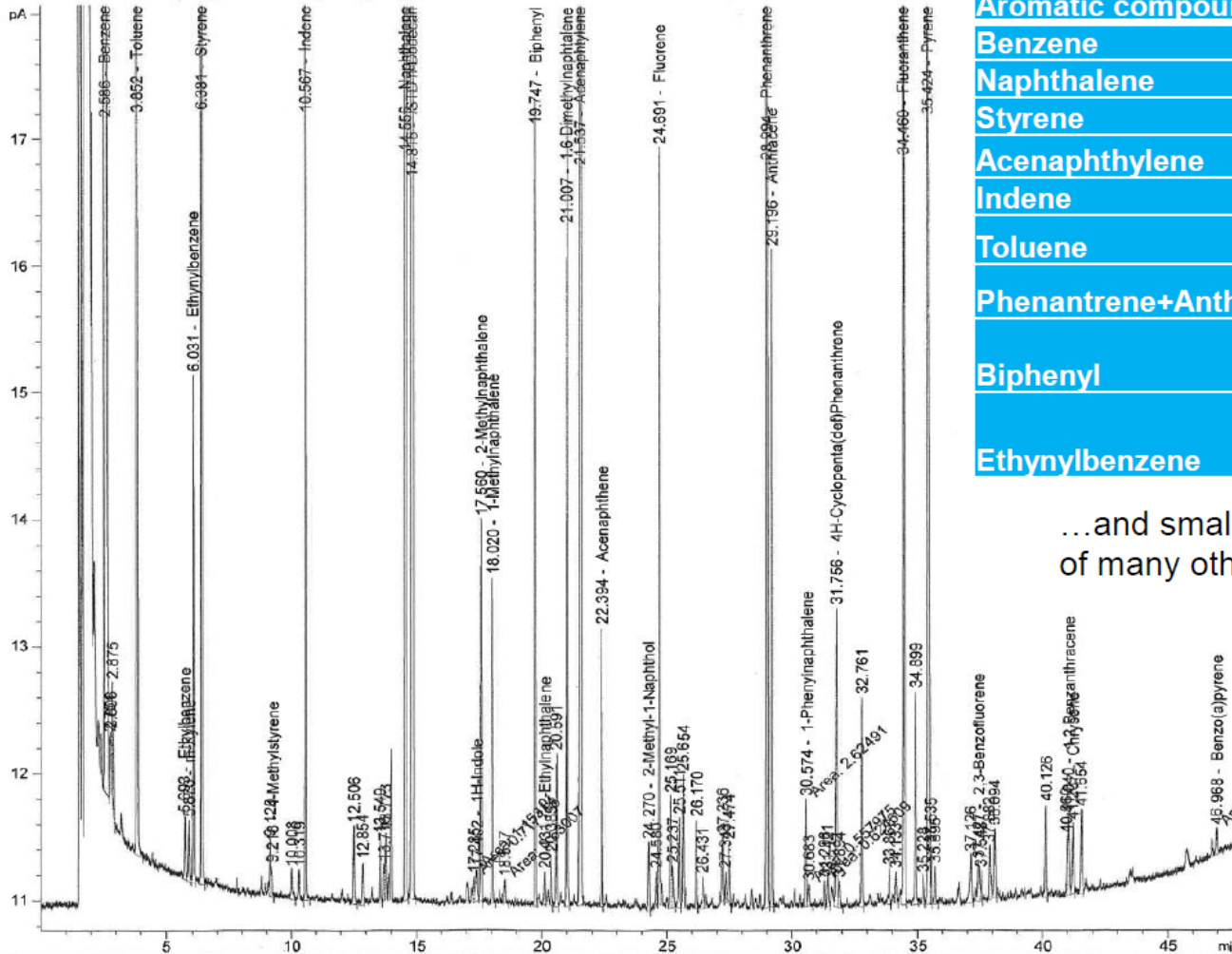


- Feed: 5 vol-% ethene in N<sub>2</sub>
- Conditions tested
  - Pressure: 1-6 bar(a)
  - Temperature: 800 – 975 °C
  - Residence time: 0.09-3.34 s (calculated for the whole reactor length)

# Example of most abundant tar compounds



C<sub>2</sub>H<sub>4</sub>, 961 °C, 6 bar, 3.34 s



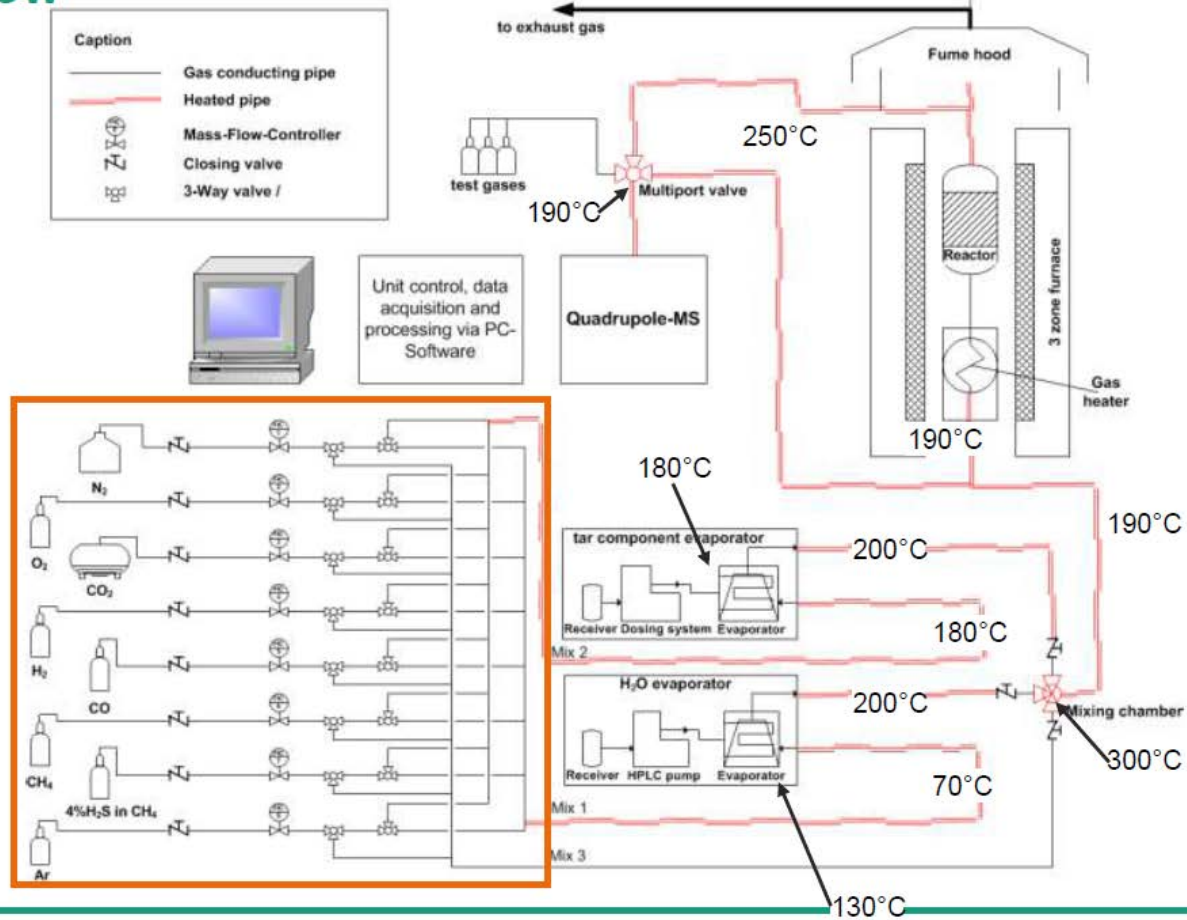
Aromatic compound	Amount, ppm
Benzene	2362
Naphthalene	535
Styrene	129
Acenaphthylene	91
Indene	60
Toluene	42
Phenantrene+Anthracene	29
Biphenyl	19
Ethynylbenzene	18

...and smaller amounts of many other compounds



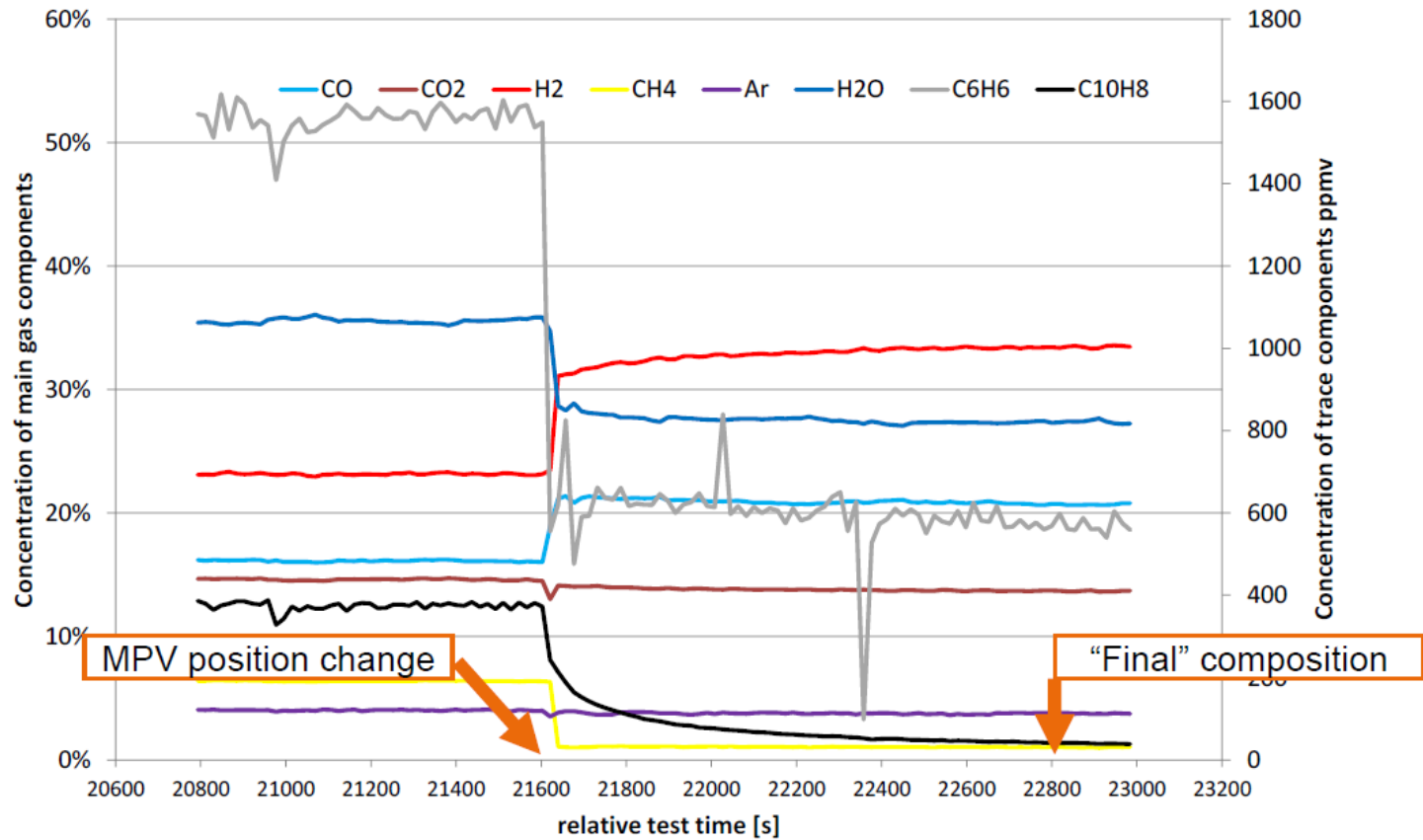
# Test system

## Overview



# Gas composition

## Example for results



# GAW Hamburg 2014;

<http://www.gas-analysis-webinars.org/?q=node/14>

	V. Dragan O. Akpolat	Des Plaines, USA
Concept of round robins and host site activities	M. Kleinhappl	Bioenergy2020+ Graz, Austria
Method comparison for sampling and analysing syngas pollutants	E. Basset	GDF-Suez Paris, France
Comparison of different methods for tar analysis based on SPA and wet-chemical tar sampling for thermochemical gasification of biomass	<u>S. Reil</u> J. Karl H. Stork S. Beer	Fraunhofer UMSICHT Sulzbach-Rosenberg, FAU Erlangen-Nürnberg, OTH Amberg-Weiden, Germany
Method catalogue	M. Kleinhappl	Bioenergy2020+ Graz, Austria
A Test gas generation system from Bioenergy 2020+	M. Kleinhappl	Bioenergy2020+ Graz, Austria
Supplementary tools for gasification gas analysis	<u>M. Reinikainen</u> N. Kaisalo S. Tuomi P. Simell	VTT Technical Research Centre of Finland Espoo, Finland
Optical absorption spectroscopy for gas analysis in biomass gasification	<u>H. Grosch</u> A. Fateev S. Clausen K. L. Nielsen	DTU Technical University of Denmark Roskilde, Denmark
Continuous tar monitoring via FID –Actual status of the further development of an online tar measurement device	A. Gredinger	University of Stuttgart - IFK Stuttgart, Germany
Recent work on on-line tar analysis at TU Berlin	Y. Neubauer	TU Berlin Berlin, Germany

GAW Hamburg 2014;

<http://www.gas-analysis-webinars.org/?q=node/14>

## Barbara system for test gases in cell-based toxicology: reality



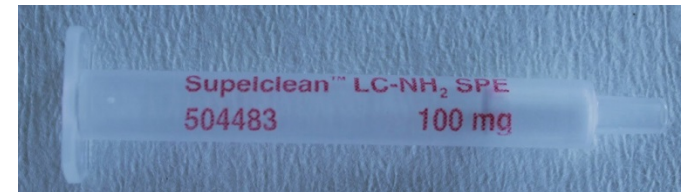
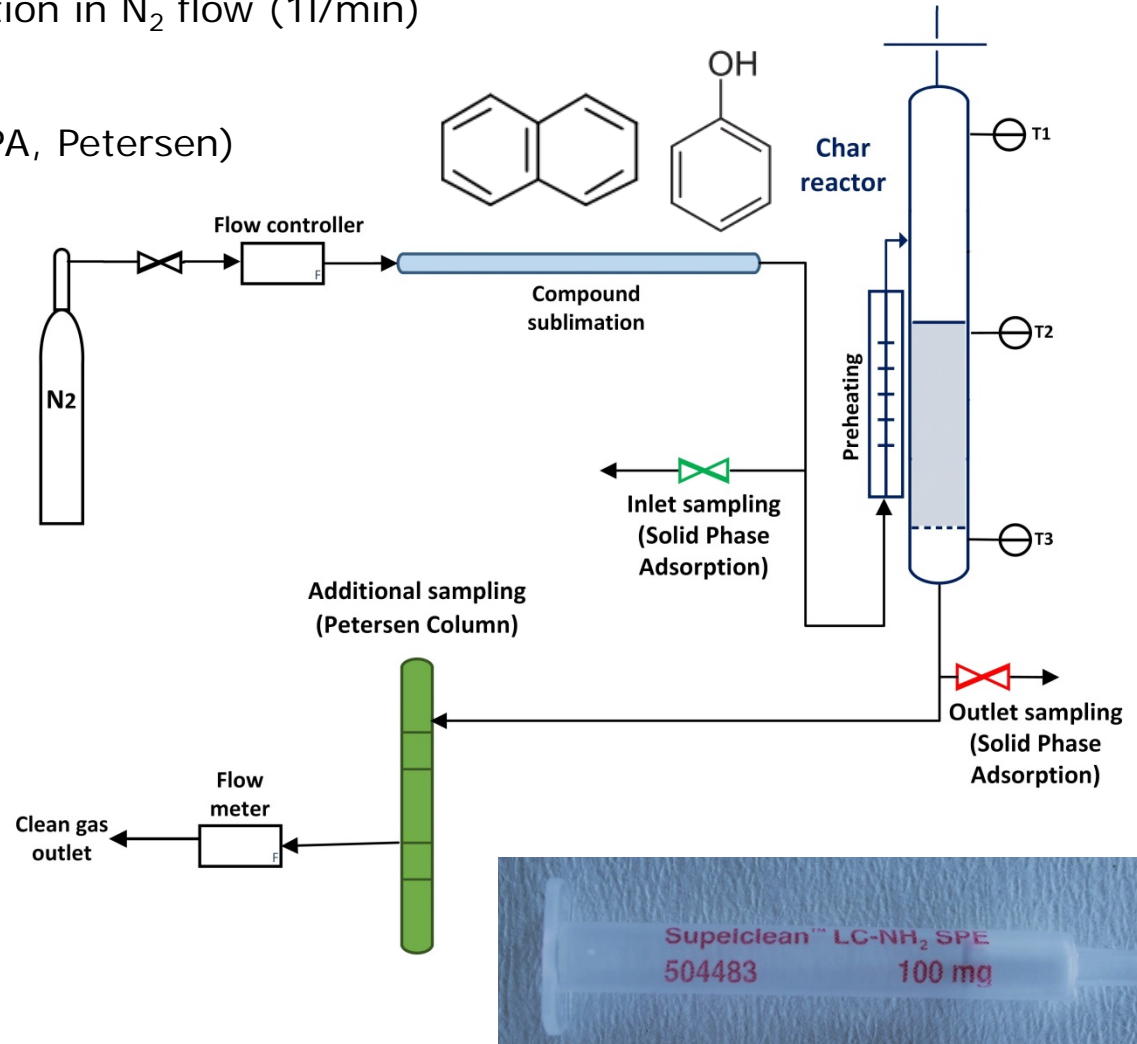
MEDIZINISCHE  
UNIVERSITÄT  
INNSBRUCK



Information already given in this workshop

# Experimental set up

- Phenol, naphthalene: sublimation in N<sub>2</sub> flow (1l/min)
- Contact with hot biochar bed
- Inlet - outlet gas sampling (SPA, Petersen)





# PAH test-gas generator



Figure 3.9: Photo of solid PAH substances reservoir

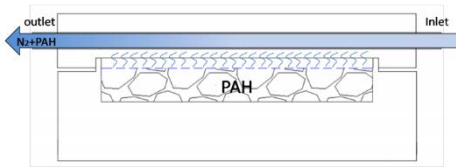
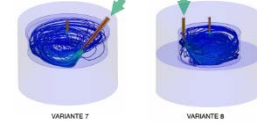
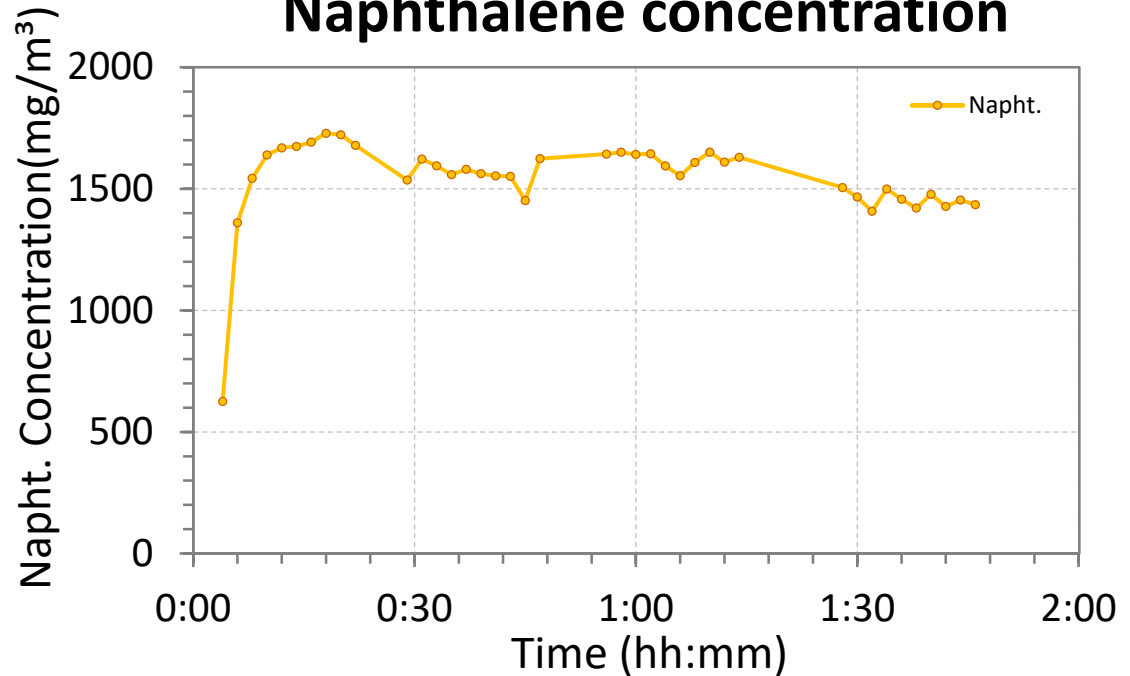


Figure 3.10: Schematic diagram of solid PAH substances reservoir

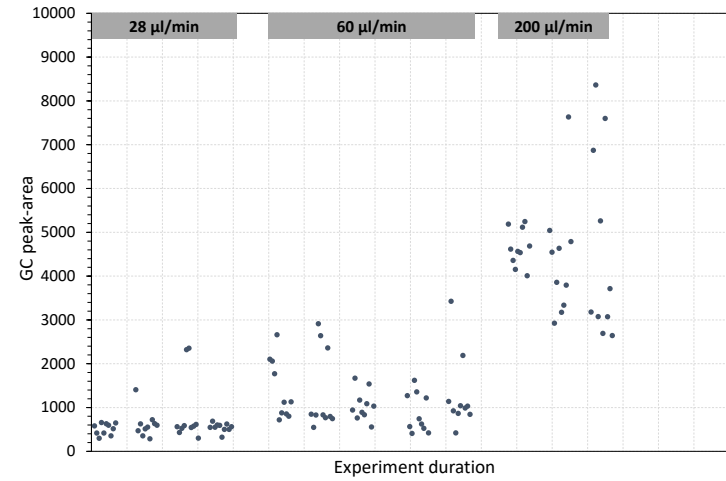
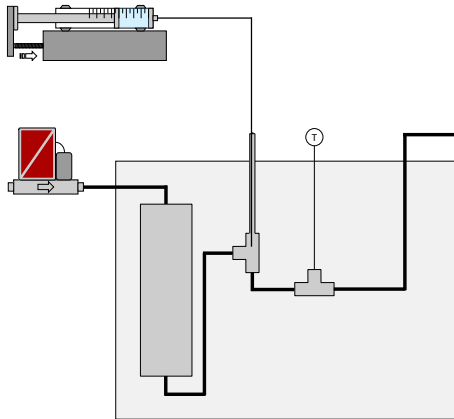


## Naphthalene concentration

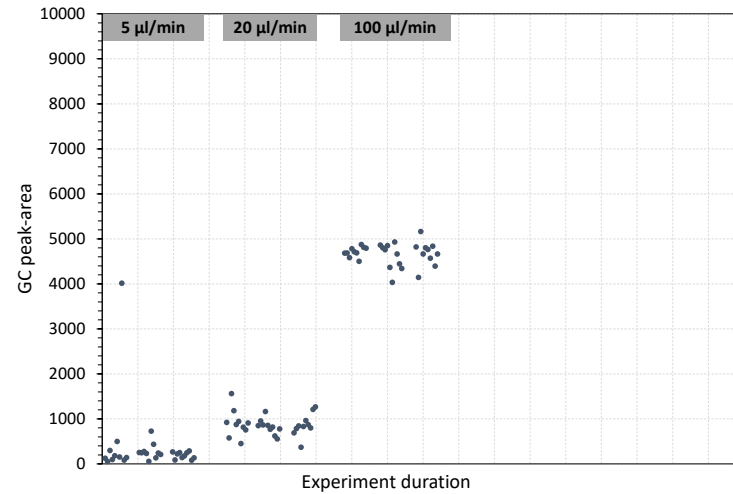
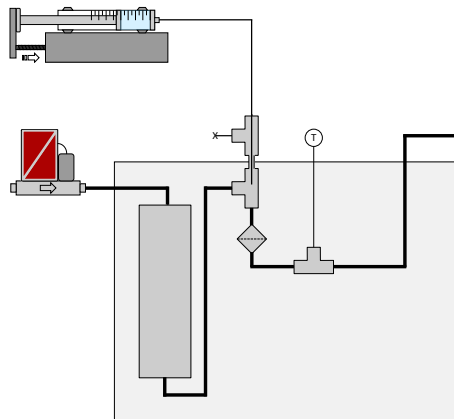


# PAH test-gas generator

- Version 1



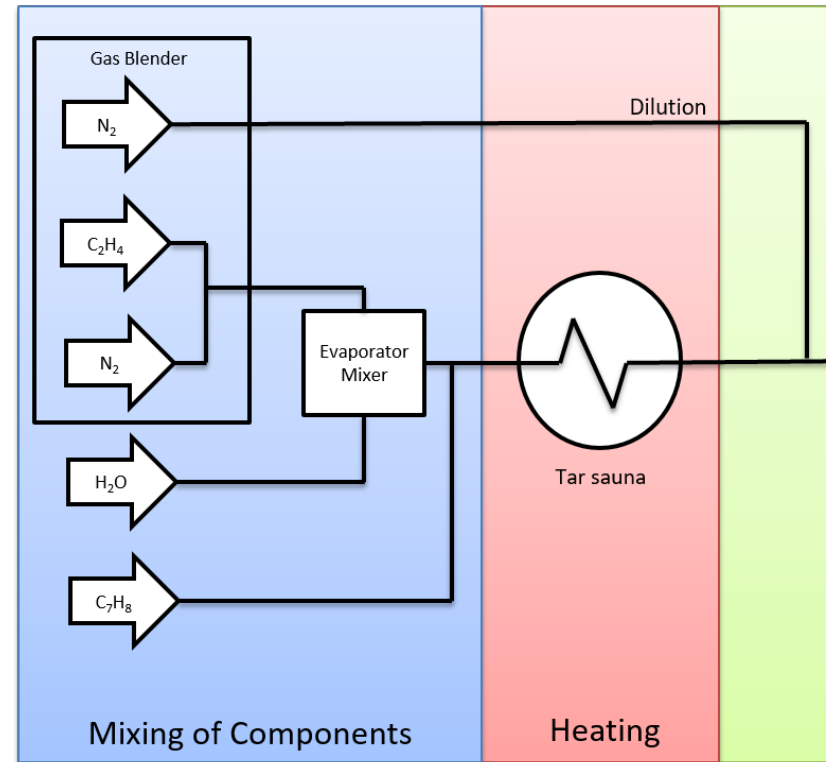
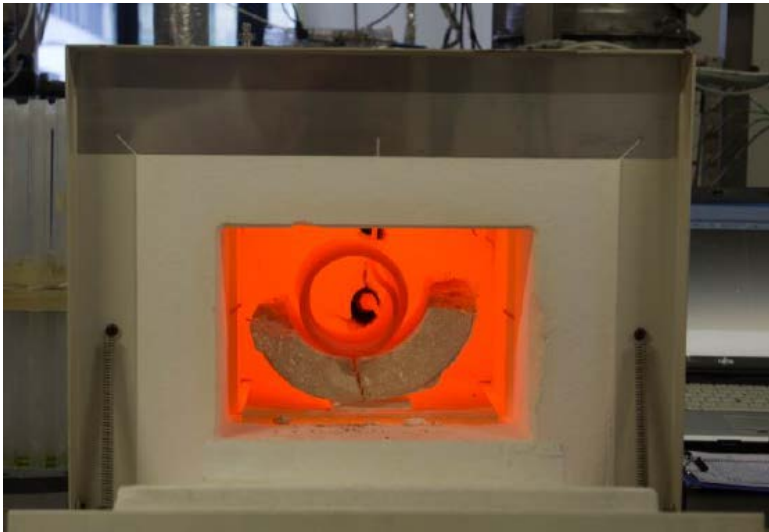
- Version 2





# Test gas generation with the Finnish 'tar sauna'

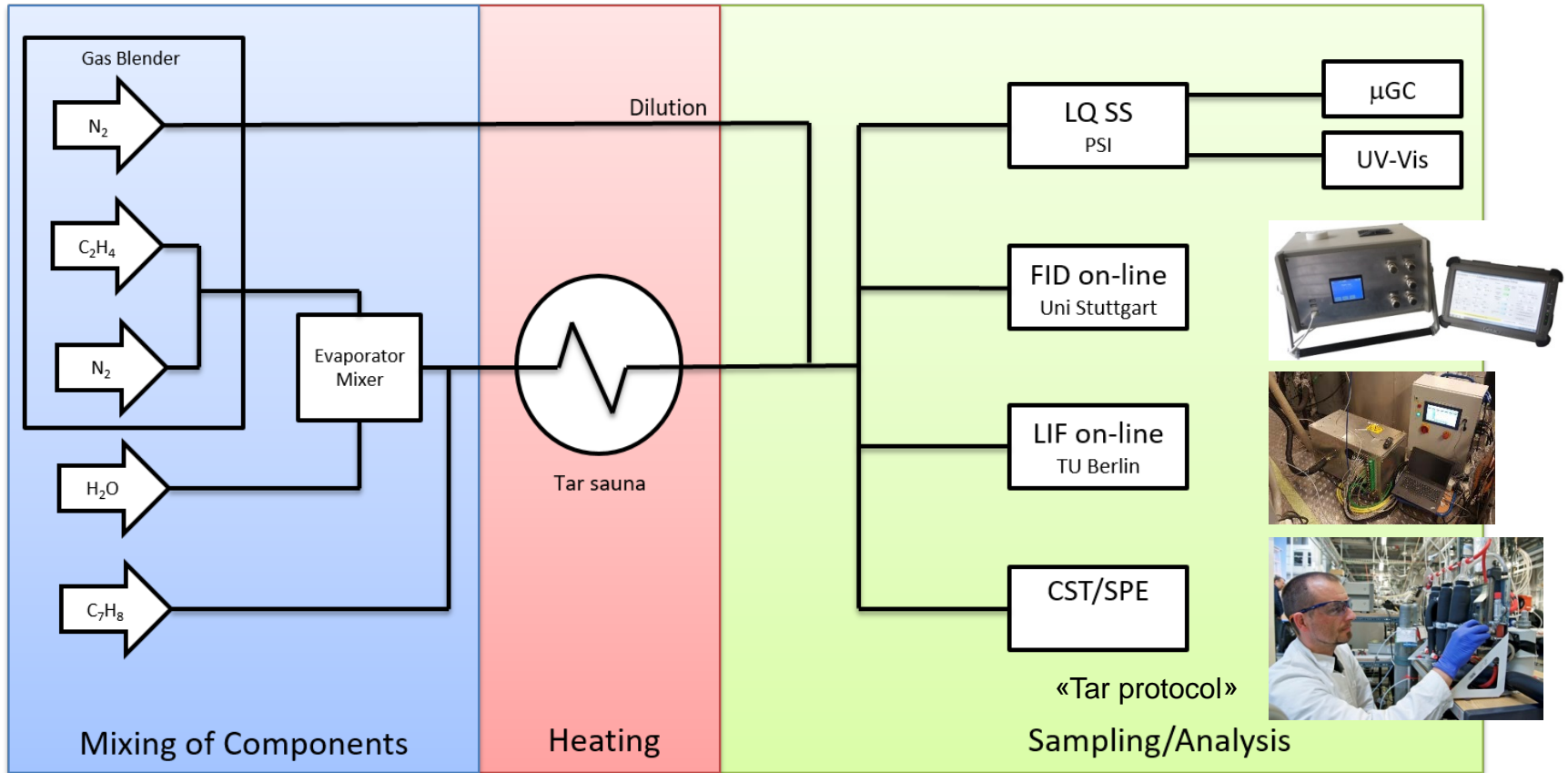
Principal idea for this approach  
from VTT



Reinikainen, M., Kaisalo, N., Tuomi, S., Simell, P.: Tar generation by ethene pyrolysis. Gas Analysis Webinar 21.03.2014 - <http://www.gas-analysis-webinars.org/?q=node/2>

Kaisalo, N. K., Koskinen-Soivia, M.-L., Simell, P. A., Lehtonen, J.: Effect of process conditions on tar formation from thermal reactions of ethylene. Fuel, 153 (2015) 118–127 DOI: 10.1016/j.fuel.2015.02.085

# Measurement setup - combination of test-gas generator with analytical tools



Further updates:

- New System @ Fraunhofer UMSICHT built in 2016/17
- Reactive system @ PSI used also with Sulphur addition
- „Unshown“ presentation from PSI from webinar 2014
  
- New Webinar with further updates and needs ?

Note:

Reactive systems suitable for delivering larger PAH with low vapour pressure and high boiling points- species hard to evaporate without changing chemical structure

In General: Little information published - just tools !

Thank you for your interest!