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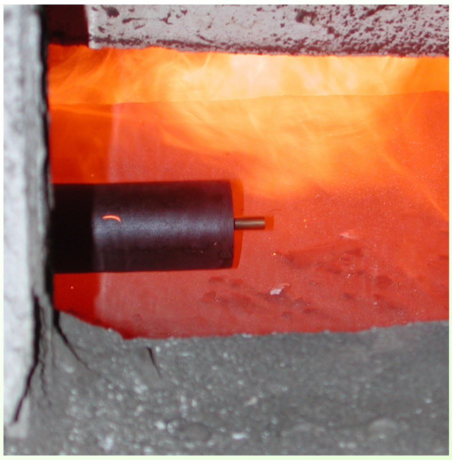
Your partner for energy utilisation from biomass and energy efficiency
Research • Development • Engineering

Measurements, Test runs and Analyses



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BIOS BIOENERGIESYSTEME GmbH
Key information

- Founded in 1995 as a spin-off of the Graz University of Technology
  Re-organisation to a limited liability company in 2001
- 2015 opening of the BIOS Innovation Centre
- General manager: Prof. Dr. Ingwald Obernberger
- Present staff: 25 (21 graduated engineers)
- Annual turnover in 2020: approx. 5.0 Mio €
- Markets: Austria, Germany, Italy, Switzerland but also Belgium, Denmark, Estonia, France, Greece, United Kingdom, Ireland, Croatia, Montenegro, The Netherlands, Norway, Serbia, Slovakia, Spain, Czech Republic, Hungary, Bangladesh, Barbados, Belarus, Chile, Honduras, Canada, Russia, South Africa, Taiwan, USA

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Mission

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Measurements at combustion, gasification and pyrolysis plants

- **BIOS BIOENERGIESYSTEME GmbH has an extensive range of equipment for the performance of test stand and field tests at combustion, gasification and pyrolysis plants available.**
- **The equipment used at BIOS includes**
  - conventional gas analysers and particle measurement techniques
  - specially designed innovative methods and devices
    for fly ash, aerosol and deposit sampling, for hot gas measurements (measurement and sampling in reactors at temperatures up to 1,200°C) and low-temperature corrosion measurements.
- **Due to the modular structure of the data logger system it is possible to collect measurement data at several distant locations in a plant as well as to visualise and store them on a central computer. Therefore, a high flexibility concerning the solution of different measurement problems can be gained. For data evaluation in-house developed software is available.**

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Measurements and methods applied – overview (I)

Measurement	Methods
Conventional emission measurements at combustion units: O_2, CO, CO_2, NO_x, NO_2, OGC, dust, HCl, SO_x, NH_3, gaseous heavy metals	• conventional gas analysis continuous methods • conventional gas analysis discontinuous methods
Product gas measurements at biomass gasification and pyrolysis reactors: CO, CO_2, H_2, CH_4, C_xH_y, tars (including tar analyses)	• conventional gas analysis continuous methods • conventional gas analysis discontinuous methods
Measurement of the gas temperatures in gasification and pyrolysis reactors, furnaces and boilers using suction pyrometers	• conventional continuous methods

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Measurements and methods applied – overview (II)

Measurement	Processes
Determination of the particle size distribution and concentration of aerosols and fly ashes in the gas	• discontinuous particle measurement techniques • continuous methods electric low-pressure impactor
Ash, fly ash, and aerosol sampling in biomass conversion processes for subsequent wet chemical analyses and analyses by electron microscopy	• particle sampling and analyses
Hot gas sampling of particles in the conversion reactor	• high temperature particle sampling
Deposit sampling and analyses in biomass furnaces and boilers	• deposit sampling
Determination of the potential of flue gases for low-temperature corrosion	• continuous methods low-temperature corrosion probe

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Equipment – conventional gas analysers and measurement techniques (I)

- **Flue gas, product gas and pyrolysis gas analyses and measurements - continuous methods**

Parameter	Principle	Device
O_2	Paramagnetic	Emerson NGA 2000
CO/CO2	NDIR	Emerson NGA 2000 ABB URAS26
OGC (organic gaseous carbon)	Flame Ionisation Detection	ErsaTec SmartFID Environment S.A. Graphite 52M
H_2	Heat conductivity	ABB CALDOS27
$NO/NO_x/NO_2$	Chemielumineszenz ND-IR	ECO Physics CLD 700 EL ht Emerson NGA 2000
CO, NO, NO_2, N_2O, HCN, HCl, SO_2, NH_3, CH_4, several hydrocarbons	FT-IR	Sicom DX-4000
Pressure, differential pressure	Two wire technique pressure gauge	various devices
Temperature	Resistance, thermos couples	various devices
Gas humidity	Thermo-Hygrometer	various devices
Gas velocity	Prandtl-tubes, calorimetric	various devices

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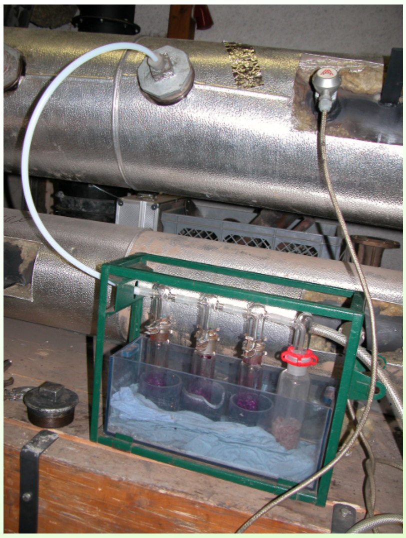
Equipment – conventional gas analysers and measurement techniques (II)

- **Flue gas analyses in combustion plants - discontinuous methods**

Parameter	Method
SO_x- and HCl-concentration in the flue gas	Method according to VDI 3480; Gas sampling with heated probe, dust removal and collection of acid compounds in distilled water with H_2O_2, NaOH. Detection by HPLC.
NH_3-concentration in the flue gas	Gas sampling with heated probe, dust removal and collection in H_2SO_4. Detection according to Kjeldahl.
Hg-concentration in the flue gas	Gas sampling with heated probe, dust removal and collection of Hg in cooled H_2SO_4 and diluted nitric acid. Detection by HGAAS or ICP-MS.
Heavy metal vapours in the flue gas	Gas sampling with heated probe, dust removal and collection of heavy metals in cooled diluted nitric acid. Detection by absorption spectrometry (FAAS, ICP-OES or GFAAS) or mass spectrometry (ICP-MS).



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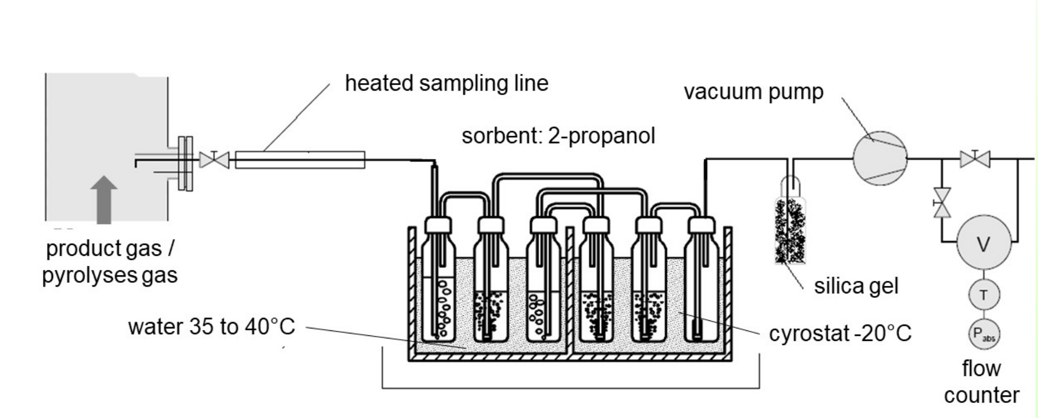
Equipment – conventional gas analysers and measurement techniques (III)

▪ Product gas analyses in gasification and pyrolysis plants – discontinuous methods

Parameter	Method
Tar sampling and tar analyses	Gravimetric method according to CEN/TS 15439:2006, Biomass gasification – Tar and particles in product gases – Sampling and analysis Adsorption of tars in 5 bottles filled with propanol at 40°C resp. -20°C; evaporation of the solvent in a vacuum dryer and subsequent gravimetric determination of the tar content as well as tar analyses regarding C, H and N.
Gas sampling tube	Discontinuous product gas sampling in a sampling tube and subsequent analyses by means of gas chromatography and mass spectrometry (GC-MS) regarding CO, CO_2, H_2, CH_4, O_2 and N_2.



Schematic drawing of the tar sampling train

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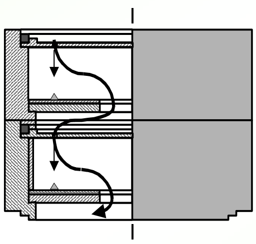
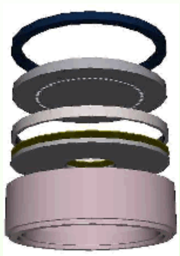
Equipment – conventional gas analysers and measurement techniques (IV)

▪ Particle measurement techniques - discontinuous methods

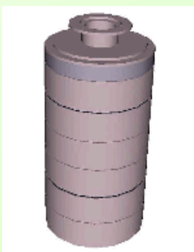
Parameter	Method
Total dust concentration in gases	Method: Gravimetric method according to VDI 2066. Principle: For a certain period a slipstream of the gas is sucked through a quartz filter, where the dust particles are precipitated. The dust concentration in the gas is calculated by the mass change of the filter divided by the gas volume sucked through the filter. To achieve representative sampling, the slip stream must be taken with the same velocity as the gas has at the sampling point (isokinetic sampling).
Particle size distribution and concentration of aerosols in the gas	Method: Low-pressure cascade impactor Devices: Hauke LPI30, cut diameters: 8/4/2/1/0.5/0.25/ 0.125/0.0625 µm Principle: A slip stream is isokinetically sampled from the gas channel and sucked through the impactor. The impactor consists of several stages. In each stage the gas changes its flow direction and particles which are too big to follow the streamlines of the flue gas, are precipitated.



particle separation in the impactor



scheme of one impactor stage

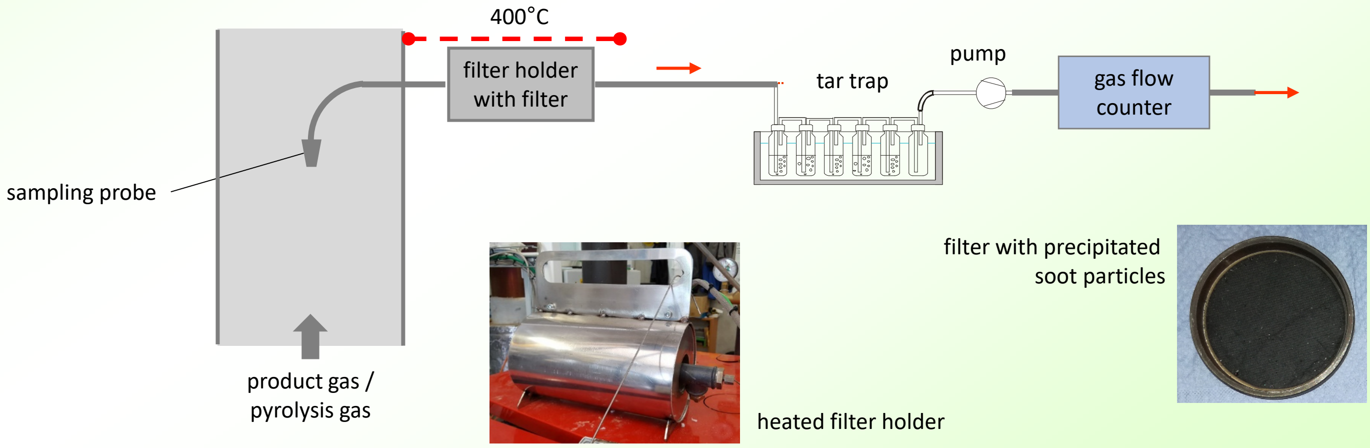


assembled cascade impactor

Equipment – conventional gas analysers and measurement techniques (V)

▪ Particle measurement techniques - discontinuous methods

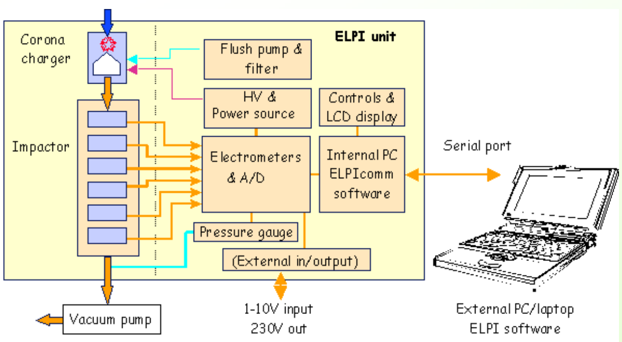
Parameter	Method
High-temperature dust and soot measurements in gases from gasifiers and pyrolysers	Method: Gravimetric method (filter measurement). Principle: For a certain period a slipstream of the gas is sucked through a quartz filter, where the dust and soot particles are precipitated. The dust/soot concentration in the gas is calculated by the mass change of the filter divided by the gas volume sucked through the filter. To avoid tar condensation, the sampling probe and the filter holder including the filter are pre-heated and then heated during the measurement to 400°C. Therefore, appropriate high-temperature seali8ng materials are applied.



Equipment – conventional gas analysers and measurement techniques (VI)

▪ Particle measurement techniques – continuous method

Parameter	Method
Particle size distribution and concentration of aerosols in the gas (continuous method)	Method: Electrical low-pressure cascade impactor Device: Dekati, 10 lpm Principle: At the ELPI inlet particles are charged and then pass several impactor stages. As soon as a particle is separated from the gas, it looses its electrical charge. The resulting current is measured for each impactor stage and from these data, the particle size distribution is determined (as a number size distribution). Therefore, the ELPI provides the possibility of quasi-continuously detecting the particle size distribution and concentration of aerosols and fly ashes in the gas within a size range between 0.03 and 8.97 µm in intervals down to 1 second. The ELPI is especially applicable for basic research concerning influencing parameters on aerosol (fine particle) formation in biomass conversion plants as well as for the determination of separation efficiencies of fine particle precipitation devices.



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Equipment – conventional gas analysers and measurement techniques (VI)

▪ Deposit probe sampling

Parameter	Method
Deposit sampling and analyses	Method: Sampling with an air cooled deposit probe and subsequent analyses by SEM/EDX Device: in-house development Principle: • A deposit probe consists of an air cooled tube at which a sampling ring is mounted. • The deposit probe is introduced into the conversion reactor for a certain period. • The surface temperature of the sampling ring is controlled by the cooling air. Thereby it is possible to simulate the surface of a boiler tube. • The sampling ring is weighed before and after exposure to the gas and the ash deposit build-up on the ring is determined in g/m³/h. • Fly ash and aerosol deposits, which have been formed on the sampling ring, are afterwards analysed concerning their structure and chemical composition by using electron microscopy (SEM/EDX). These results represent a basis for the definition of guidelines concerning the melting behaviour of deposits and corrosion risks. • By these measures an evaluation of the risk for deposit build-up during the conversion of a certain fuel in a certain reactor and at a certain surface temperature is possible.


Deposit probe in operation


Sampling ring with deposit

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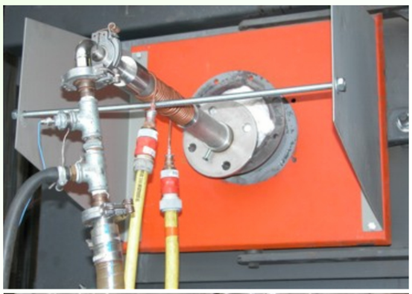
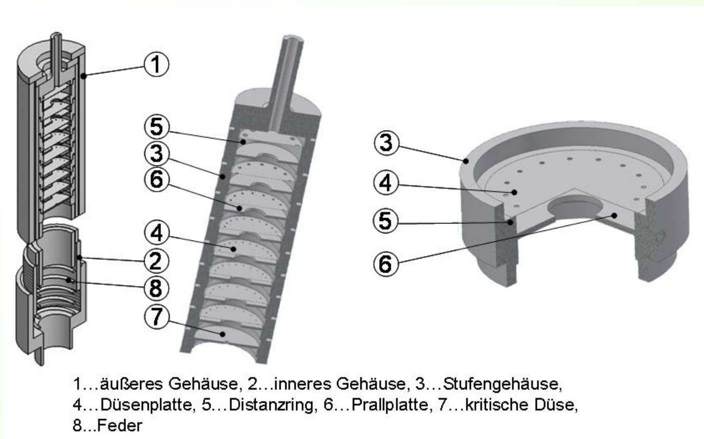
Innovative methods (I)

▪ High-temperature particle sampling

- To facilitate particle sampling for subsequent analyses by wet chemical measures and electron microscopy even from a high-temperature environment (e.g.: from the hot reactor), a special high-temperature low-pressure impactor has been developed.
- This device can be applied for in-situ particle sampling at temperatures up to 1,100°C.


High temperature impactor in operation


High temperature impactor immediately after sampling



Scheme of the high temperature impactor

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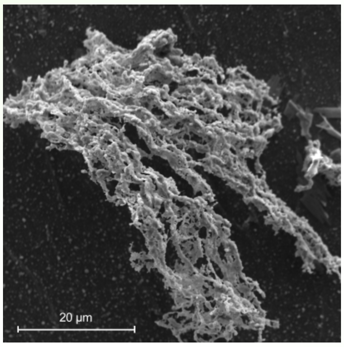
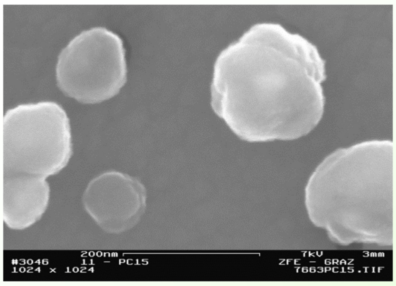
Innovative methods (II)

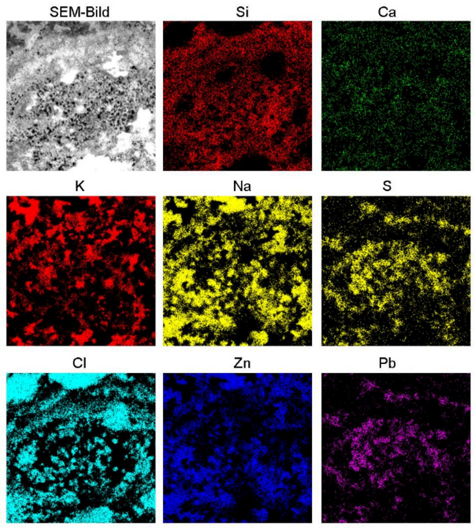
- **Particle sampling for subsequent analyses by wet chemical measures and electron microscopy**
  - Wet chemical analyses of total dust and impactor samples
  - Analyses if fly ash, aerosol and deposit samples by scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDX).



SEM-image of a coarse fly ash particle sampled during beech combustion



SEM-image of aerosols from bark combustion



SEM-image and element mapping of aerosols sampled with the High-temperature impactor in the hot flue gas at superheater inlet of a waste fired combustion plant (higher colour intensities indicate higher element concentration levels)

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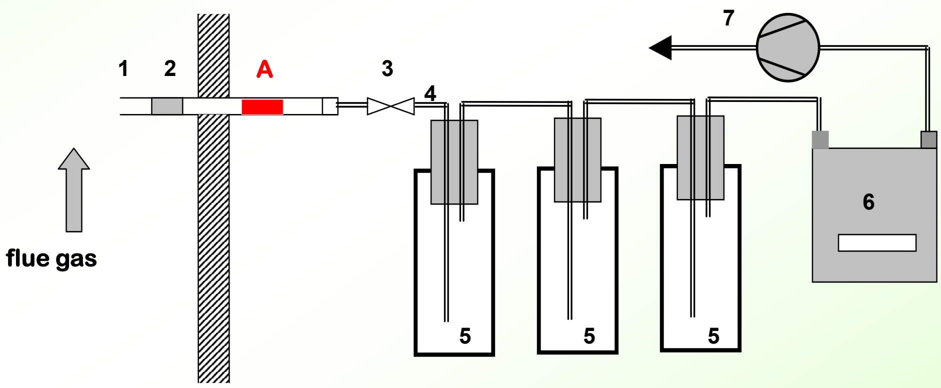
Innovative methods (III)

- **Determination of SO_2 and SO_3 concentrations in the gas**
  - The SO_3 content is a relevant parameter regarding low-temperature corrosion
  - Therefore, a SO_2/SO_3 measurement, the so-called "salt method" has been further developed by BIOS
  - Determination of SO_3 (as S) trapped in a salt bed (chloride) by ICP-OES
  - Determination of SO_2-ions precipitated in washing flasks with distilled water and diluted sodium hydroxide solution (enriched with H_2O_2) by HPLC



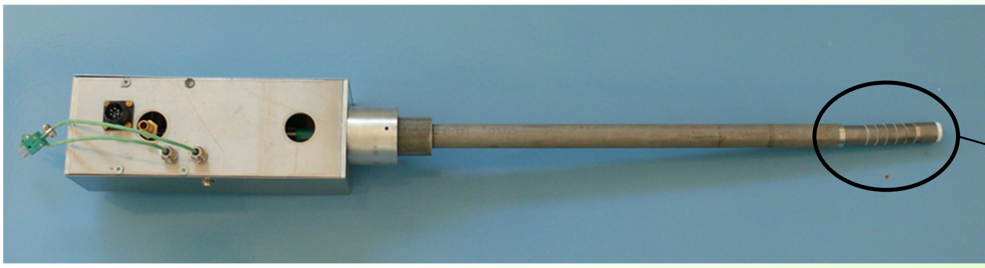
1 … extraction probe (glass/teflon)
2 … filter (quartz wool)
A … packed salt bed
3 … lock valve
4 … extraction line (silicone)
5 … washing flasks (glass)
6 … gas meter
7 … vacuum pump

Innovative methods (IV)

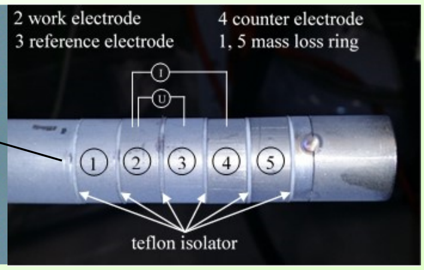
▪ On-line low-temperature corrosion probe

Parameter	Method
Low-temperature corrosion potential	Application: Determination of the acid dew point and of the temperature at which corrosion due to hygroscopic salt deposits starts (indicated by the increase of the sensor signal) Manufacturer: Babcock Borsig Steinmüller GmbH, DE Principle: • Sensor head with steal rings separated by teflon isolators • Systematic variation of the sensor surface temperature by cooling the sensor with pressurised air • Liquid phases (due to hygroscopic salts) or the condensation of acids on the sensor surface lead to the formation of an electrolyte. • By applying a voltage between the working and the counter electrode a current can be detected ➔ corrosion signal.

low-temperature corrosion probe



sensor



Lab-scale reactors for fuel characterisation and conversion tests (I)

▪ Thermal analysis

- Sample intake: up to 5 g
- Temperature range: 25 - 1,050°C
- With this device
  - biomass fuels
  - torrefied biomass
  - biochar

  can be investigated regarding their thermal decomposition behaviour at different gas phase conditions.

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Lab-scale reactors for fuel characterisation and conversion tests (II)

Lab-scale reactor (I)

- Lab-scale reactor applied to determine relevant basic parameters concerning the thermal decomposition of different biomass fuels (in-house development of BIOS).
- Fields of application
  - Determination of the combustion behaviour of biomass fuels.
  - Determination of the release of gaseous compounds from the fuel during biomass pyrolysis, gasification and combustion.
  - Determination of the release behaviour of volatile and semi-volatile ash forming elements.
  - Evaluate the ash melting behaviour.
- Reactor design
  - The reactor consists of an electrically heated retort made of Alsint 99.7, which is externally heated. Alsint 99.7 is at reducing and oxidizing conditions inert and was selected to prevent for possible reactions between the retort and the gases.
  - The retort is cylindrical (inner diameter: 12 cm).
  - The sample holder and the border of the fuel bed (diameter 9.5 cm and height 10 cm) are also made from Alsint 99.7 and are placed on a scale. Reaction gas (e.g. air, N2) is supplied from below the fuel bed. The scale is mechanically decoupled from the retort by a liquid seal. A synthetic thermal oil (Therminol 66) is used as sealant.
  - The sample holder is placed on a balance in order to be able to determine the weight loss of the sample during the pyrolysis/gasification/combustion process. Conventional gas analysers are used to determine the gas composition in the reactor and of the exhaust gas from the reactor.

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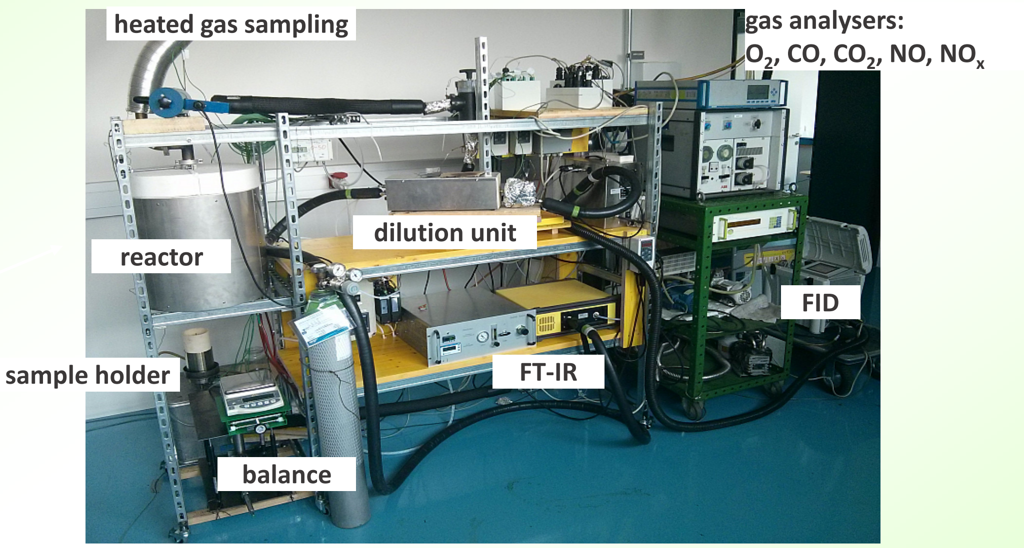
Lab-scale reactors for fuel characterisation and conversion tests (III)

Lab-scale reactor (II)

- The sample is introduced into the pre-heated reactor
- Therefore, a rapid heating, which is well comparable with the one in real thermal conversion processes prevails
- The initial sample as well as the residues are analysed and therefore, also detailed information about the release of inorganic species from the fuel to the gas phase is obtained.




Fuel and residues (ashes) from a test run with beech wood chips


Fuel and residues (ashes) from a test run with straw pellets

Tests at test stands and pilot-scale plants (I)

- **Performance of test runs at combustion, gasification and pyrolysis plants for technology development, evaluation and optimisation**
- **Within dedicated test runs plant operation data collected by the process control system over several hours are evaluated and additionally, samples from all relevant in-going and out-going streams are taken. Usually the following parameters are determined and samples are taken:**
  - flue gas composition (in combustion plants) resp. gas compositions (in gasification and pyrolysis plants) along the gas pathway through the plant
  - temperatures and volume flows of the combustion resp. gasification air as well as gas flows in different plant sections
  - gas temperatures in the conversion reactors and at the inlet and outlet of subsequent heat exchangers resp. gas coolers and gas cleaning reactors
  - deposit formation in conversion reactors and heat exchangers/boilers (applying deposit probes)
  - particle size distributions, concentrations and chemical compositions of fly ashes and aerosols (fine particulates) in different sections
  - tar and soot contents of the gases in different sections of gasification and pyrolysis plants
  - fuel and ash sampling and subsequent chemical analyses of the samples taken

Tests at test stands and pilot-scale plants (II)

- **In the subsequent evaluation, at first mass, element and energy balances are calculated over the plant on the basis of the measurement and analysis results. These balances serve on the one hand to ensure the plausibility of the measurement and analysis results and on the other hand to perform element flow analyses.**
- **In the following the results of the test runs are evaluated with special respect to:**
  - process stability and conversion behaviour of the fuel investigated
  - slagging tendencies in the fuel bed and in the conversion reactor
  - regarding combustion plants:
    - formation and reduction of gaseous emissions (CO, OGC, NO_x, HCl, SO_x, heavy metals)
    - formation and reduction aerosol and fly ash emissions
    - formation and chemical characterisation of ash deposits in the boiler as well as evaluation of their potential for corrosion
  - regarding gasification and pyrolysis plants
    - assessment of the gas quality (heating value; tar, soot, HCl and H_2S contents)
    - assessment of the efficiencies of product gas cleaning reactors
    - assessment of the burnout quality of the ashes (gasification plants) resp. of the quality of the charcoal (bio-char) produced (pyrolysis plants)

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Tests at test stands and pilot-scale plants (III)

- **The data obtained in this way form an important basis for**
  - development and design of new biomass conversion technologies and systems (based on combustion, gasification and pyrolysis)
  - development and conception of corresponding flue gas or product gas cleaning systems
  - evaluation and optimization of existing systems
  - evaluation and, if necessary, adaptation of existing systems with regard to the utilisation of new biomass fuel assortments

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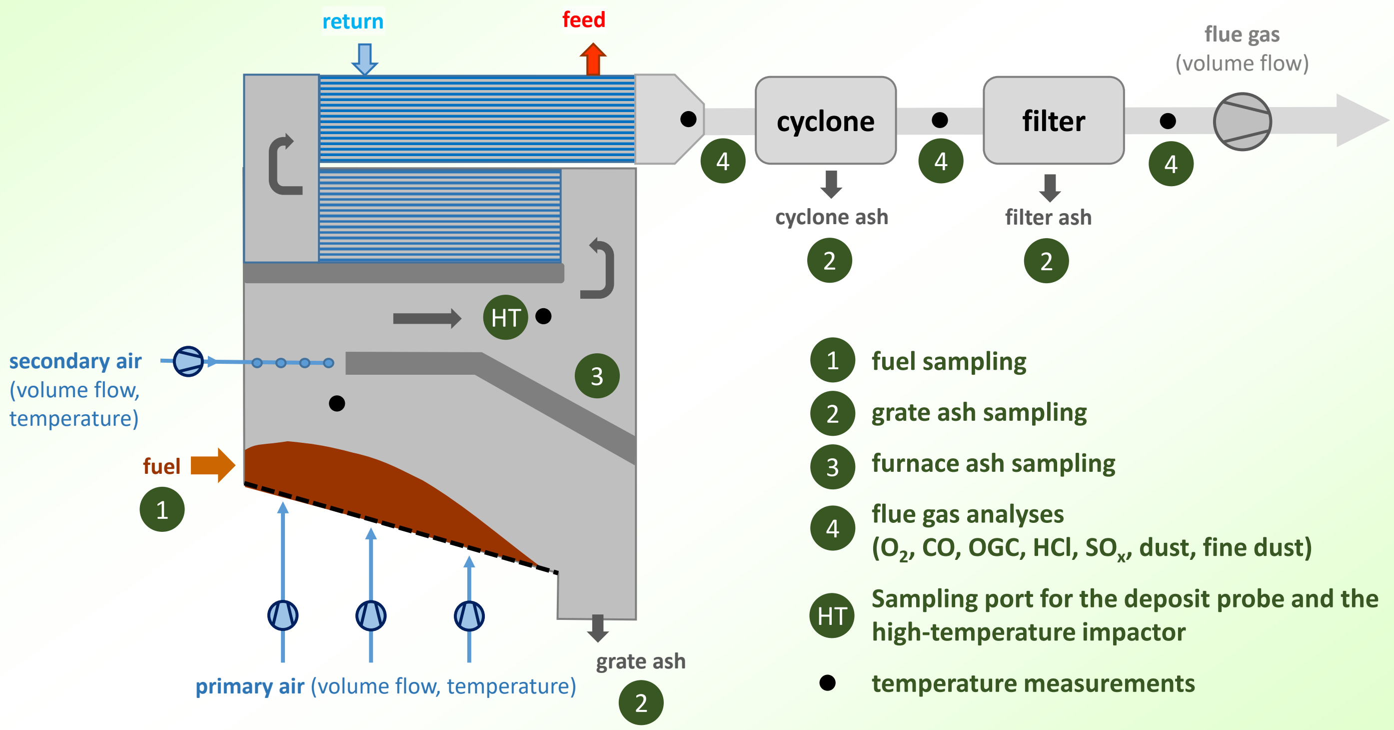
Tests at test stands and pilot-scale plants (IV)

Typical set up for a test run at a combustion plant



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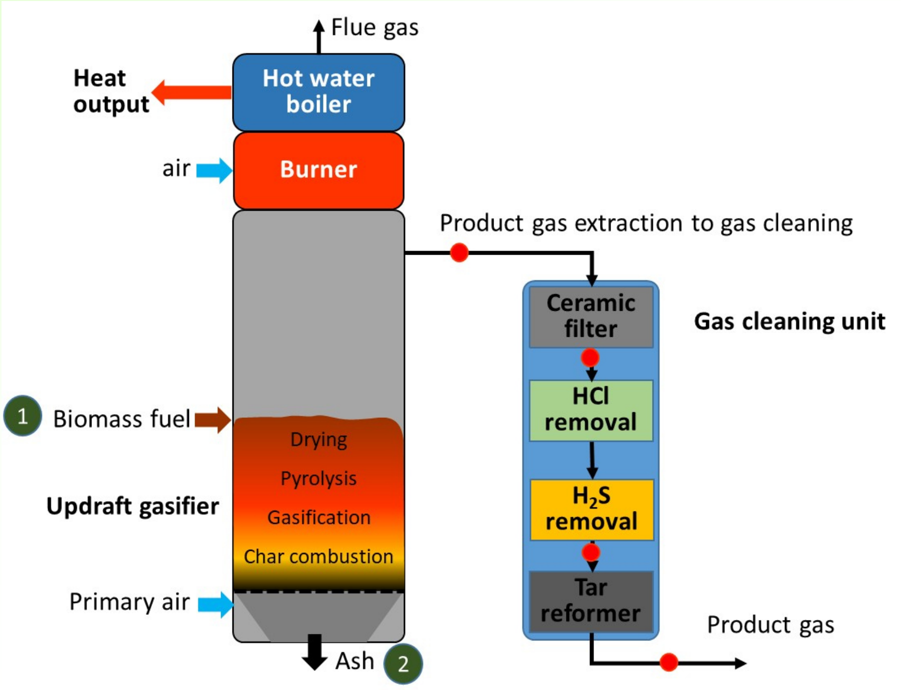
Set up for a test run at a gasification plant with a side stream gas cleaning unit



1 …fuel sampling

2 …bottom ash sampling

● sampling points for measurements of
- gas composition
- HCl contents
- H2S contents
- soot and tar contents

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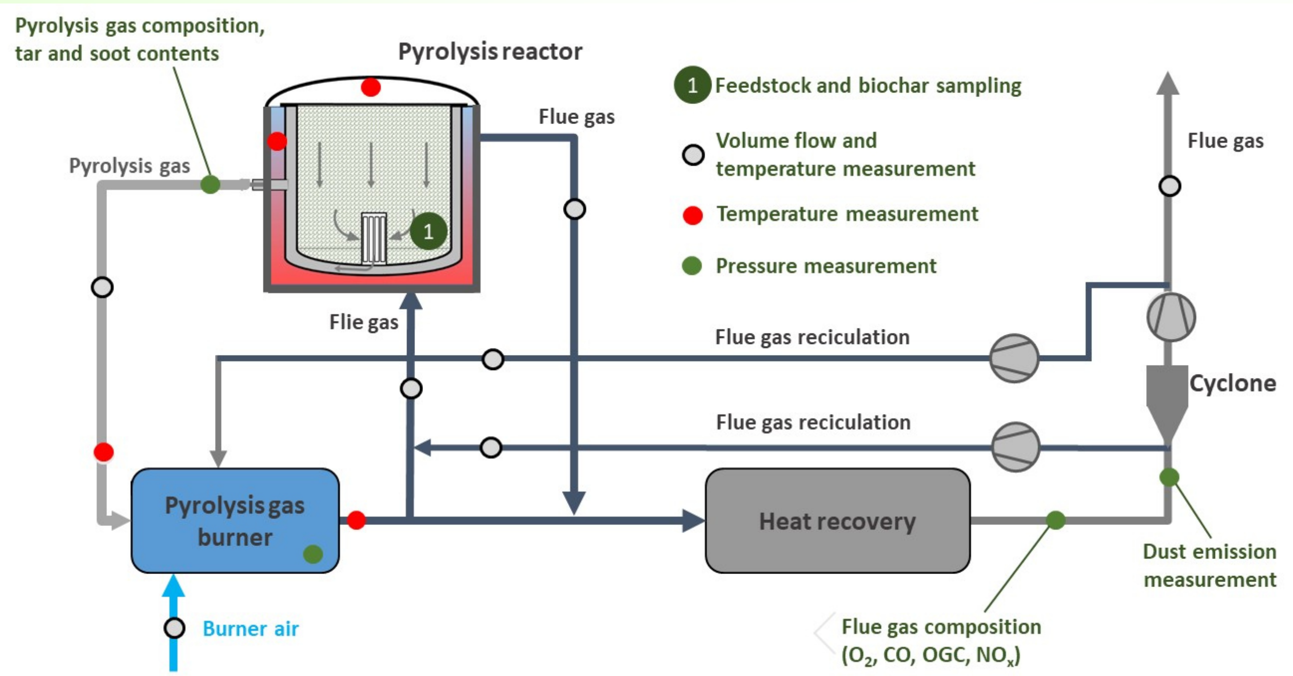
Set up for a test run at a batch-wise working pyrolysis reactor for biochar production coupled with a pyrolysis gas burner and a subsequent heat recovery



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Analyses (I)

- **BIOS offers a wide range of chemical and electron microscopic analyses.**
- **Regarding electron microscopic analyses, a close co-operation with the Research Institute for Electron Microscopy and Fine Structure Research at the Graz University of Technology exists.**
- **Therefore, a high flexibility concerning the solution of different analytical problems in the field of thermochemical biomass conversion can be offered.**

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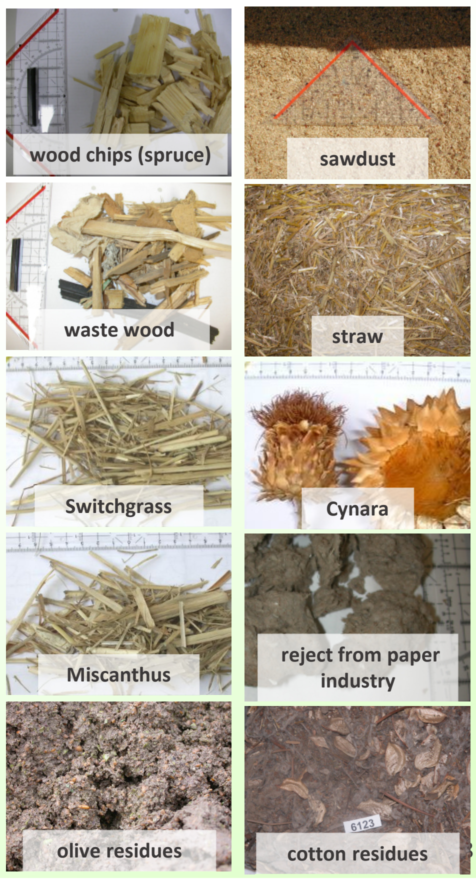
Analyses (II)

- **Major areas in chemical analytics**
  - Wet chemical analyses of
    - fuels
    - substrates
    - ashes and aerosols (fine particulates)
    - tars
    - deposits and slags
    - condensates
    - boiler water
  - regarding
    - major elements (C, H, N, S, Cl)
    - minor elements (e.g. Si, Ca, Mg, K, Na, Fe, P)
    - trace elements (heavy metals)
    - sample specific parameters such as pH-value, electric conductivity, calorific value, moisture content, ash content, organic and inorganic carbon, organic dry substance (ODS), chemical oxygen demand (COD), SO_3, SO_4, NH_3, NH_4, NO_3, NO_2, etc.



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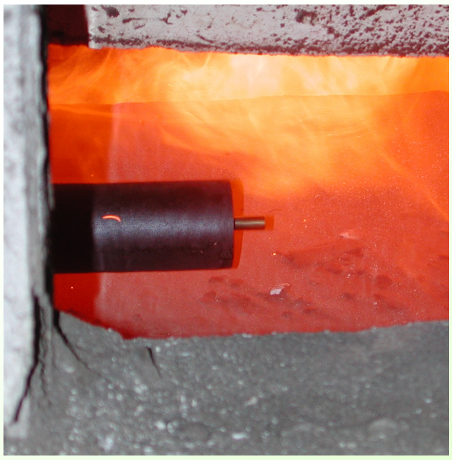

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