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Guideline for Sampling and Analysis of Tars Condensates and Particulates from Biomass Gasifiers

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1 Introduction

Producer gases from gasification reactors contain particulates and organic contaminants (tars) which, if not removed, can cause operational problems. For fixed bed gasifier / IC engine applications, too high levels of particulates and tars would damage the engine or incur an unacceptable level of maintenance. Referring to [Stassen 1993], an IC engine can be operated successfully when the particulates content is below 50 mg/Nm³ and the tar content is less than 100 mg/Nm³. A successful engine operation also includes minimal expenses for maintenance and wear.

A number of different sampling and analysis methods are used to determine the level of these contaminants in the gas. This diversity of methods makes the comparison of operating data from different sources very difficult. This represents a significant barrier to the further development and commercialisation of the technology. All methods in use have one or more disadvantages which lead to more or less severe limitations of the methods. One method which is used quite successfully in cocurrent fixed bed biomass gasifiers and in various gas cleaning systems is the ETH/Verenum P&T sampling method presented in this guideline. It is a classical impingement train which uses anisole as a tar absorption solvent.

This guideline presents the procedures for the determination of heavy tars, polyaromatic hydrocarbons, condensates and particulates from atmospheric biomass gasifiers. Except for the determination of the light tars (boiling point < 200°C), all these procedures were verified and optimized by attentive experimentation during test runs at the Indian Institute of Science in Bangalore, India as well as at gasifier test runs in Europe conducted by the ETH Zurich and/or Verenum Zurich [Mukunda et al. 1994], [Sharan et al. 1996]. The laboratory treatment and upgrading procedures have been jointly worked out and verified with the Swiss Federal Laboratories for Materials Testing and Research in Dübendorf.

The particulate and tar sampling procedure (designated as P&T unit) has been adapted from known methods for either particulate or hydrocarbon sampling methods (e.g. US EPA Method 5 for isokinetic particulate sampling from stack gases; Canadian protocol developed in the ENFOR project; German VDI Richtlinie 2499, Blatt 3 – Methode A for isokinetic sampling of polychlorinated dioxins and furans). Some modifications have been made with respect to the solvent used and the method of particulate sampling. Furthermore, isokinetic sampling is not controlled electronically but is adjusted to the operating condition manually (quasi isokinetic sampling). According to [Kurkela 1995], the best and most often used method for tar sampling from biomass gasifiers is based on the use of impinger trains with an organic solvent.

With the presented sampling system, the absorption of particulates and heavy tar components as well as for polyaromatic hydrocarbons (PAH) is very efficient. Naphthalene as the PAH component with the highest volatility can be retained with an efficiency of >99%. The absorption efficiencies for toluene and benzene are app. 90% and app. 40% respectively.

It is assumed, that the parameters for the characterisation of the gasifier performance (energy and mass balance, temperatures etc.) are registered without further mentioning in this guideline. In cases where two sampling units (before and after the gas cleaning unit) are used, all the following procedures and analysis have to be done for each sampling unit separately.

2 Definitions

For the assessment of the gasifier, the gas cleaning system and/or the IC engine, comparable sampling and analytical procedures for particulates and tars are necessary. Today, there are at least a dozen of different sampling procedures known in the literature. Tar is often determined as a gravimetric value which contains a mixture of heavy and light tars, PAH's as well as some phenolic compounds. There is no common or widely accepted method which compounds should be considered as tars or not and how the tar components are sampled and analyzed. Moreover, the sampling quality of the methods used is not known. Up to date, no assessment of the different sampling and analytical methods has been made. It must be assumed that most of the methods used give non comparable results. Hence, the tar values given in the literature must be considered as a *qualitative indication* of the tar amount in the producer gas.

In this guideline, the following definitions of producer gas contaminants are used:

Heavy tars: Heavy tars generally are considered as the sum of high molecular organic compounds with high boiling points, typically above 200°C. The chemical composition is generally unknown. For this guideline, heavy tars are determined gravimetrically by evaporating the tar absorbing solvent anisole (=methoxy benzene; $t_b = 153^\circ\text{C}$). At least, all components with boiling point below 153°C will quantitatively evaporate.

PAH: Polycyclic aromatic hydrocarbons. These are organic compounds with more than two aromatic rings. Today, several hundreds PAH compounds have been identified and analyzed. However most of them are found in very low concentration (ppb level) and therefore have no relevance in typical biomass producer gases¹. However, some of the lower molecular weight PAH's are present in biomass producer gas in considerable concentration. Typical representatives of these PAH's are: naphthalene, phenanthrene. A list of 16 environmentally relevant PAH compounds according to the US EPA is given in the appendix 6.9. In this guideline, only the 16 US EPA PAH's are considered (some of them are also major tar components in biomass producer gas). The determination of the US EPA PAH's is generally made using GC/MS.

¹ Moreover, very few laboratories are able to determine all the PAH compounds.

- Light tars:** Aromatic hydrocarbons with a medium volatility. The boiling point range typically is between approx. 80°C and 200°C. Representatives are: benzene, toluene, xylene.
- Light hydrocarbons:** Nonaromatic hydrocarbons (up to approx. C6 hydrocarbons) with a high volatility at ambient conditions. The components are gaseous at room temperature. Typical representatives are: methane, ethylen, propane.
- Phenols:** Aromatic organic compounds with at least one –OH group (most of the relevant phenols have only one –OH group). Typical representatives are „phenol“ (C₆H₅OH) and cresols (= hydroxy-toluene; 3 different chemical structures possible). Phenol and cresols are considered as corrosive components for fuel gas applications in IC engines.
- Water soluble organic residue:** The amount of solid matter found as evaporation residue from the aqueous phase after particle separation and anisole extraction. In cocurrent gasifiers, it has been found that this solid residue is of purely organic nature with native wood fuels (see chapter 6.5). With other fuels, the residue can also contain salt components (such as ammonium chloride) or other gaseous inorganic components (e.g. heavy metals).
- Particulates:** The amount of solid matter which is filtered from liquids and which is deposited on microfibre filters. „Tars“ adsorbed on the particles are extracted using an appropriate solvent. The determination method is gravimetric.

The gravimetric determination of the heavy tars as an overall sum value can lead to misinterpretation and other conclusions. The evaporation temperature depends on the type solvent used and vacuum evaporation possibly leads to other results than ambient pressure evaporation. Therefore in this guideline, anisole is used as a solvent and evaporation is made under vacuum. Nevertheless, the gravimetric heavy tar determination is a useful parameter since not all heavy tar components can be analytically quantified e.g by GC/MS. These so called *non chromatographable hydrocarbons*² are considered as an important fraction of the heavy tars.

As an additional and more reproducible value than the heavy tars, some of the PAH's determined analytically by standard methods. Each of the 16 PAH's determined is known precisely. Some of these PAH are also present in the heavy tar value.

Particulate sampling requires a (quasi) isokinetic sampling method. Depending on the sampling temperature and the chemical nature of the particulates and tars, some tar components may adsorb on

² Some hydrocarbons especially high molecular compounds are very difficult to analyze e.g. by gas chromatography. The determination method for these compounds is not a standard method and requires particular skills of the operator.

the (carbonaceous) particulates or may be present as liquid droplets (e.g. after wet gas cleaning systems). Representative sampling of particulates or liquid droplets with diameters above app. 1 μm requires isokinetic sampling. Therefore, tar sampling is preferably also done under quasi isokinetic sampling conditions.

At the gasifier outlet, most of the organic compounds such as the phenols or the PAH's will be present as aerosols. Generally, aerosols can not be quantitatively deposited e.g. on glass fibre filters even at moderate low temperature. Also the vapour pressure of these compounds is considerable at typical sampling temperatures. For a total condensation of these substances, the compounds either must be absorbed in an appropriate liquid (e.g. an organic solvent such as acetone or anisole) or must be adsorbed on solid surfaces such as modified polymers. For this guideline, the solvent absorption with impinger trains is used. Solvent absorption with impingers is a widely used method for tar sampling from biomass gasifiers [Kurkela et al. 1995]. However, the solid polymer adsorption (SPA) technique has also been tested for biomass derived tars [Brage et al. 1997] and can be an attractive alternative to solvent absorption.

3 Description of the P&T sampling unit

3.1 Preliminary remarks

The development of the sampling parameters has been made with the assumption that the gasifier producer gas has a particulates content $< 50 \text{ mg/Nm}^3$ and tar contents are $< 100 \text{ mg/Nm}^3$. From this, a satisfactory amount of tar and particulates for the analytical determination in the laboratory will require sampling durations in the range of 3 hours and sample flow rates of approximately $0.6 \text{ Nm}^3/\text{h}$. However, the sampling unit will equally well operate with producer gases of much higher particulate and tar levels. In this case, sampling duration and/or flow rates can be reduced.

3.2 Quasi isokinetic sampling

To ensure correct particulate sampling, the sample gas stream must be taken under isokinetic conditions. Most presumably, some of the heavy tars will absorb on carbonaceous particulates and hence, isokinetic sampling conditions is also preferable for the sampling heavy tar components.

Isokinetic sampling means that the sample gas velocity must be identical to the main gas velocity in the duct. Since under isokinetic sampling leads to higher sampling errors than over isokinetic sampling, it is recommended to use a 5 to 10% over isokinetic sampling flow rate. For sampling flow rates of approximately $0.6 \text{ Nm}^3/\text{h}$, different sampling nozzles are since the gas flow rates vary depending both on the gasifier load and the sampling temperature (hot or cold end). During test runs with varying gasifier load, sampling nozzles generally cannot be changed for practical reasons. In such cases, the sampling flow rate must be reduced to ensure isokinetic sampling conditions.

To ensure proper isokinetic sampling and avoid turbulences in the main gas stream, the sampling nozzle must not be placed directly before and after duct bends. As a rule, a straight duct length of 5 and 3 times the duct diameter is necessary before and after the sampling nozzle to ensure uniform and stable velocity profiles (figure 3-1).

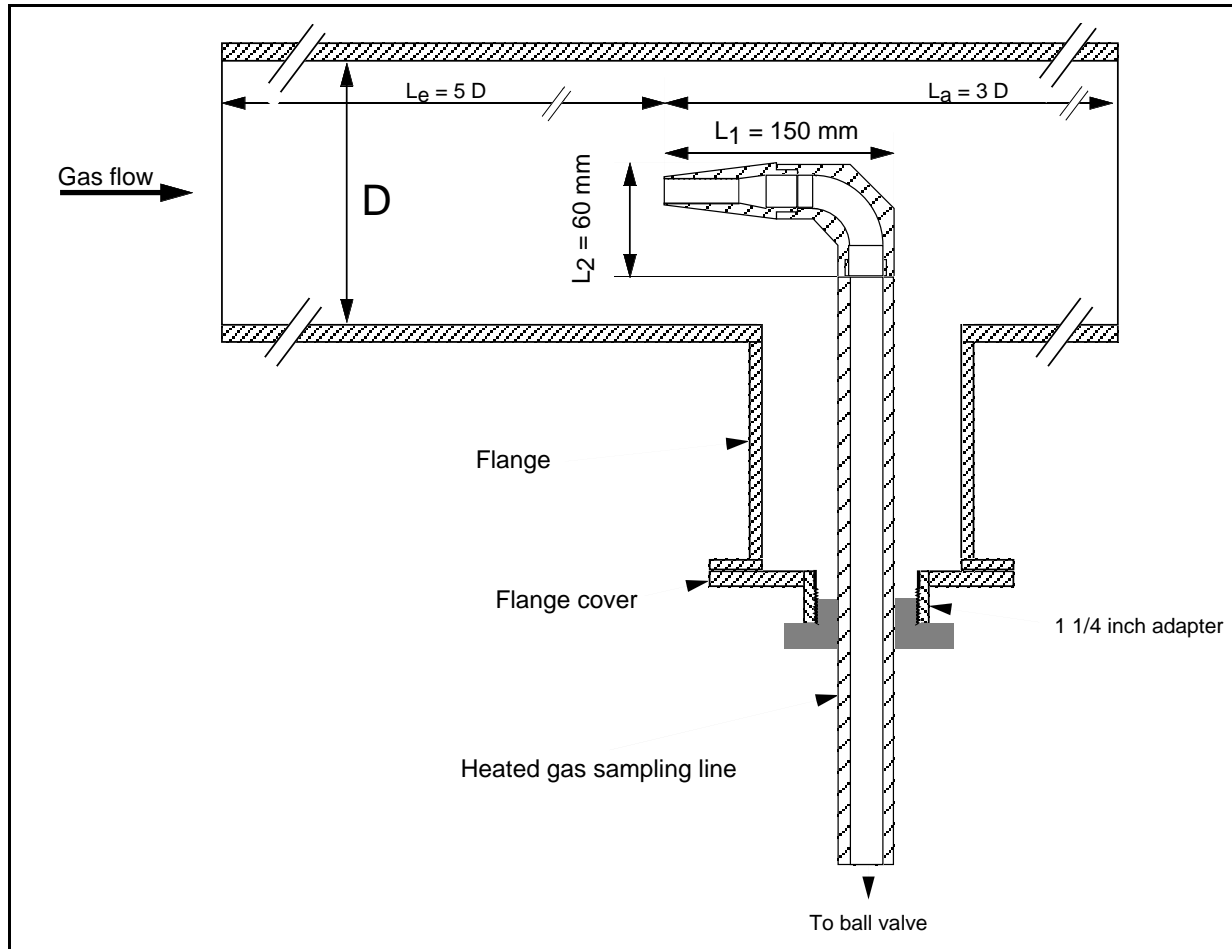


figure 3-1: Schematic arrangement of sampling nozzle for isokinetic sampling

Up to date, the gas velocity in the duct is not measured within this guideline. Hence the average velocity must be calculated from the duct diameter, the gas temperature, the producer gas composition and the gasifier load. The following assumptions must be made for this calculation: gasifier load, gas temperature and gas composition are known and do not change significantly during the test run. The actual sampling flow rate is then manually adjusted to the operating conditions. This procedure is designated as quasi isokinetic sampling.

Isokinetic sampling with automatic flow adjustment is preferable. However, flow measurement e.g. with a Prandtl tube will possibly not work properly for longer sampling periods and/or all kind of gas qualities. In this guideline, quasi isokinetic sampling is used for simplicity reasons.

3.3 Working principle of the P&T sampling unit

The sampling for tar and particulate analysis can be made both at the hot and the cold end of the gasifier system. At least the hot end sampling unit contains an additional cooling heat exchanger before the sample gas is lead to the condensation bottle (figure 3-2). The cooling heat exchanger is used to cool the gas and partially condense the producer gas moisture.

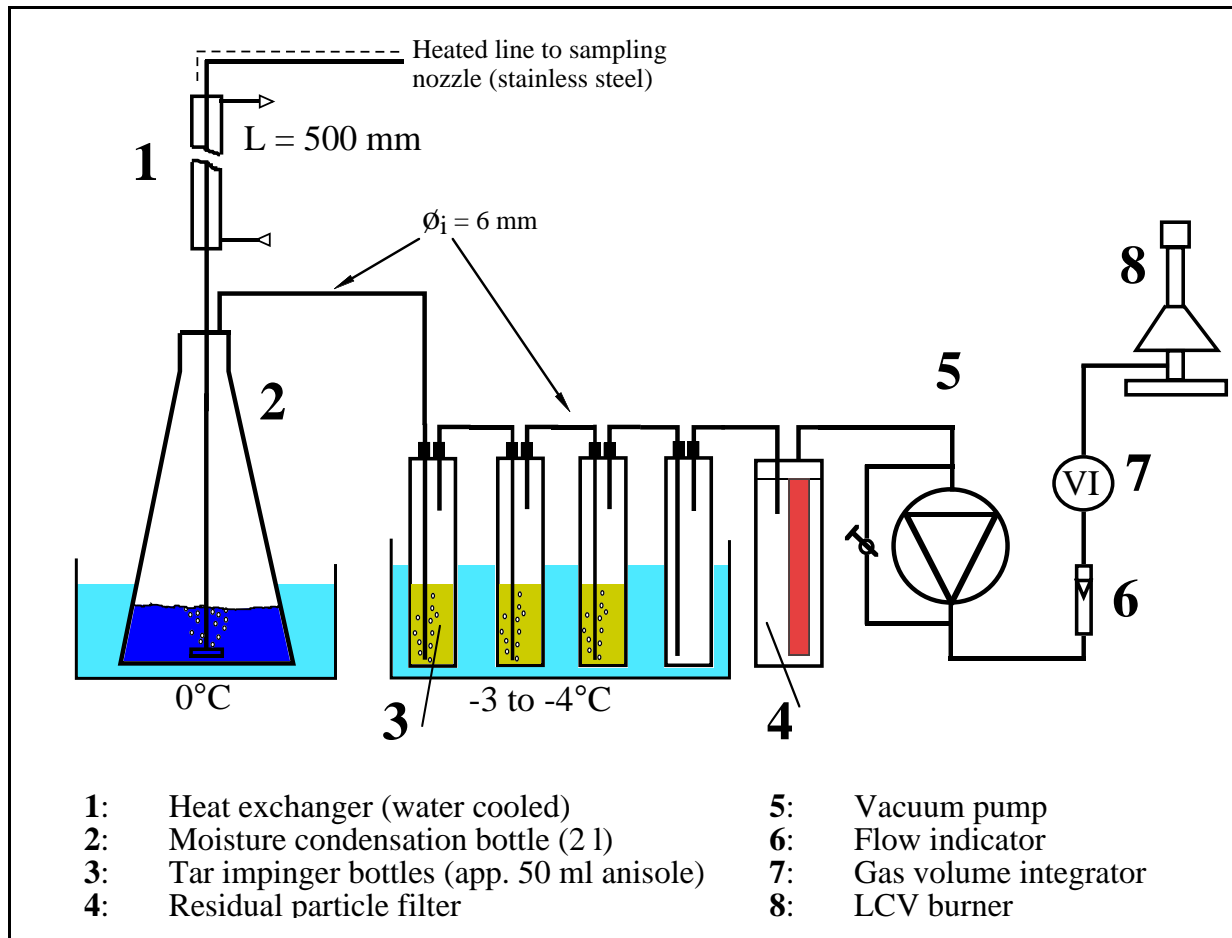


figure 3-2: Schematic of the particulate and tar (P&T) sampling train

The particulates are found in the aqueous condensate, in the solvent washing solution as well as in the residual particulate filter at the end of the sampling train.

In the following, a brief description of the hardware and the sampling procedure is given:

- **Gas nozzle / Connection to the gas pipe**

To conduct the gas from the gas pipe to the sampling equipment, an L-shaped tube is fitted by a flange to the gas pipe (figure 3-1). At the top of this tube, nozzles with different diameters are mounted. Various nozzle shapes are given in appendix 6.1. Leakages between the L-shaped tube and the metall flange are prevented by using a flexible graphite cord as a sealing media. The L-shaped tube and the sampling nozzles are preferably made of stainless steel ³. The gas velocities in the sampling tube vary between 1.7 m/s (gas temp of 20°C) and 3.3 m/s (gas temp of 300°C) for 3/8" tube diameters and a sampling flow rate of 0.6 Nm³/h. Between the tube and the following sampling equipment, a high temperature ball valve is used which allows starting, shutdown and/or interrupting of the gas sampling without affecting the gasifier operation. The total length from the tube to the valve is less than 500 mm. Heating of the tube and the valve to at least 200°C is recommended to avoid tar condensation. A sampling train leakage test must be made before every test run.

- **Condensation**

After the gas nozzle, a 0.5 m tube heat exchanger (1) cools down the temperature of the sampled gas stream. The heat exchanger is made of glass, the cooling medium generally is water at $\approx 10^{\circ}\text{C}$. The condensing water and tars flow directly through a teflon tube into the moisture condensation bottle (2).

- **Absorption in water**

In the moisture condensation bottle (2) mainly water will condense. Some of the particulates, phenols, PAH and tars will also condense in this bottle. Before starting the sampling, 800 ml of distilled water is put in the bottle. The gas entering the water is dispersed into small bubbles by using a glass bowl with approx. 20 small holes. To prevent dust and tar from plugging the outlet of the diving-pipe the hole diameter is approx. 1 mm. The diving pipe must be at least 15 mm below the water surface. The water condensing bottle is cooled to 0°C to -2°C with an ice bath or brine.

- **Absorption in anisole (methoxy benzene)**

After the condensor, the gas enters a series of 4 impinger bottles (3). Three of these bottles contain 50 ml of anisole as an absorbing liquid for organic contaminants. After the third tar impinger, a drop separator is installed. Possible dimensions of the absorption bottles are given in Appendix 6.2. The tar condensing bottles and the drop separator (same dimension as the absorption bottle) are cooled with either brine, isopropanol/dry ice (solid CO₂) or cryostatic cooler to -3°C to -5°C. At lower temperature, water freezing can occur in the diving-pipe of the first bottle. The inner diameter of

³ Stainless steel tubes, faucets etc. are mainly used as a safety measure. For short term gasifier test runs, L-shaped tubes, sampling nozzles and faucets made of glass can be used.

the pipe connections and the diving pipe is 4 mm. The diving pipe distance from bottle bottom should be 5 mm. Teflon (PTFE) is used to connect the glass tubes.

- **Residual particulate filter**

After the tar absorption bottles and the drop separator, the gas passes a glass fibre thimble filter (thimbles supplied by Schleicher&Schüll; prod. no. 371036) for residual removal of particulates (5). Totally 4 glass fibre tubes are used. With a total filtration area of approximately 500 cm² and a sampling flow rate of 0.6 Nm³/h, no significant pressure drop increase was observed over a sampling period of more than 4 hours. The residual dust separator proved to be necessary to collect submicron particles.

- **Vacuum pump and instruments**

The gas is sampled with a vacuum pump (6). The vacuum pump must be able to suck a gas flow of at least 1 Nm³/h at a pressure drop of 500 mbar. At normal pressure, the nominal flow rate of the pump should be at least 4 m³/h. The sampling train includes a rotameter (0-25 l/min) (7) and a gasmeter (8), used for volume integration over the sampling time. The vacuum pump flow rate is adjusted by using a bypass valve. To calculate the sampled gas flow rate, the absolute pressure and the gas temperature must be measured. The sampled gas is burned in a swirl burner (9) at the end of the sampling train.

Additional experimental hints:

- In the heated part of the sampling nozzle and the tube before the valve, deposition of particulates and/or tars can occur. Therefore it is strongly recommended to backflush the sampling nozzle with N₂ (or any other oxygen free gas) to blow off particulates and (if possible) tars which may have deposited before every start of a P&T sampling run.
- Turn (if possible) the nozzle opposite the flow direction if the nozzles are left in the gas duct over longer periods of time (particulate deposition can be minimized).

4 Description of the gas analysis sampling unit

The determination of the gas composition (H_2 , CO , CO_2 , CH_4 , O_2 , N_2) is important for the assessment of a gasifier system since both the gasifier cold gas efficiency and the lower heating value of the gas is based on the gas composition. Furthermore, the gas composition is needed for the calculation of the producer gas velocity in the duct. If possible, the gas composition should be measured continuously e.g. by using IR spectroscopical devices. A possible sampling and analysis train is shown in figure 4-1. Whenever possible, gas composition sampling should be made at the cold end of the gasifier system (e.g. after the gas washers).

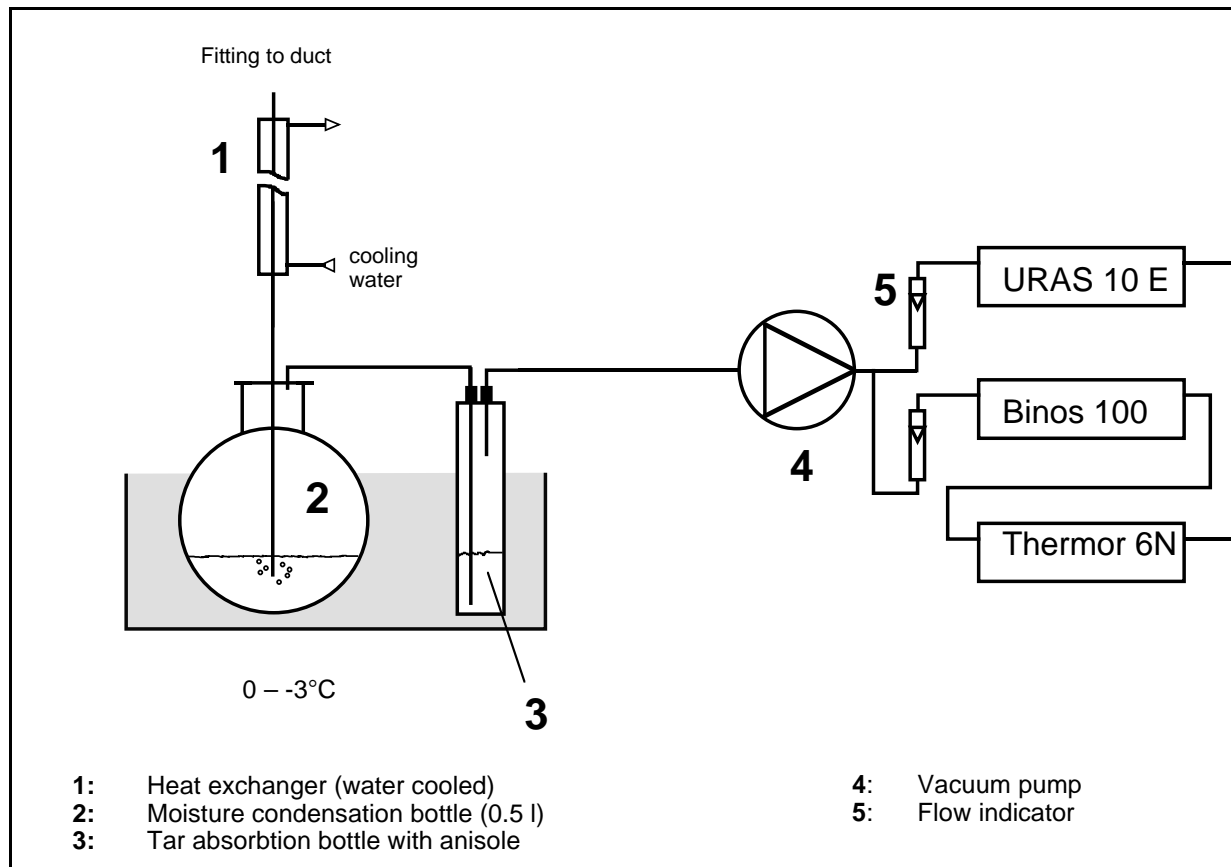


figure 4-1: Schematic of the gas analysis sampling train

Possible instruments suppliers are:

URAS 10 E (Hartmann&Braun) for CO , CH_4 , O_2

Binos 100 (Fisher Rosemount) for CO_2

Thermor 6N (MBE Electronics) for H_2

The measurement of CO, CH₄ and CO₂ is based on NDIR spectroscopy, H₂ is measured using a thermal conductivity detector (TCD) and O₂ is measured with a paramagnetic device. Using a serial arrangement for the Binos and Thermor unit, a total sample gas flow of approximately 200 l/h is required.

Nitrogen (N₂) is calculated as the difference of [CO+CO₂+H₂+CH₄+O₂] to 100%. All units must be carefully calibrated with standard gases preferably with a gas mixture of known composition. The composition of the calibration gas mixture should be similar to the fuel gas composition. The calibration must be made at least at the beginning and the end of the gasifier test run. It is highly recommended to recalibrate all devices every 48 hours.

Moisture condensation generates approximately 20 ml/h of condensate in the moisture condensation bottle (2). In cases where continuous gas analysis is desired during long term operation (e.g. for the 150 hours test), the condensate must be withdrawn. This can be done continuously by using a pump which removes the liquid from the bottom of the condensation bottle. Care must be taken to avoid gas leakages. More simple way is the discontinuous discharge of the condensate by interrupting the sampling and opening the condensation bottle. This procedure does not take more than 1 minute and does not affect the sampling quality.

As an alternative to the continuous gas analysis described above, on-line gas chromatographic (GC) analysis or discontinuous sampling in glass containers followed by off-line GC analysis can be made. On-line GC analysis requires a previous particulate separation and drying of the gas whereas sampling in glass containers does not need special pretreatment. With glass container sampling, care must be taken with respect to residual air in the containers and H₂ diffusion losses. It is highly recommended to use glass containers which are initially filled with water (can be coloured and/or contain salt if desired). Gas sampling is done by slowly discharging the water from the glass container. The recommended time for the water discharging is approximately 5 minutes. The GC must be calibrated with appropriate gas mixtures. When using molecular sieve columns, care must be taken with respect to residual gas moisture.

5 Experimental section

5.1 Presampling procedures

Before the sampling can be started, the following procedures must be made:

- The gas nozzle with the appropriate diameter and the L-shaped tube must be fitted to the duct before starting the gasifier.
- Calibration of gas analyzers and/or GC's must be made with calibration gas mixture. The calibration gas mixtures should have a comparable composition as the producer gas.
- The initial amount of the water added to the moisture condensation bottle (see figure 3-2) must be known.
- The initial weight (dried to constant mass in a given atmosphere) of the glass fibre filter thimbles (figure 3-2) must be determined.
- A sampling unit air leakage test must be made. For this, the gas sampling tube before the moisture condensation bottle is closed and the vacuum pump is carefully turned on. When bubbling in the last tar absorption bottle disappears, the sampling unit is leak tight. The leakage test should be made with a differential pressure of at least 400 mbar. After the leakage test, the locked sampling tube must be opened very slowly and carefully. As soon as bubbles pass through the sampling bottles, the volumetric pump flow is slowly reduced. Backflushing the bottles must be avoided. The depressurization of the sampling train generally takes several minutes.
- After the leakage test, the volumetric sampling flow rate is adjusted to the experimental value of 0.6 Nm³/h. Afterwards, the value on volumetric gas reading device must be read (initial value).
- The barometric pressure is measured. The absolute pressure is used for the calculation of the nominal sample flow rate and the contaminants levels (all values are given in mg/Nm³).

5.2 Sampling procedures

5.2.1 Tar and particulate sampling

During the gasification test run, one P&T sampling unit will be operated for the determination of particulates, tars and condensates in the the raw gas stream directly after the gasifier. A second unit must be used in the clean gas stream after the gas cleaning system if such a system is installed.

To get reliable data, it is most important that the sampling conditions can be maintained at defined values during the experiment. The sample gas flow rate can decrease because of plugging in the condensation/absorption bottles (ice formation) or in the thimble filter (particulate deposition). Ice formation should be monitored carefully. During normal operation (and sampling periods of less than 4 hours), the total pressure drop over the sampling train is less than 200 mbar. In cases where ice formation is observed, the pressure drop increases quickly within some minutes until the diving pipe of the impinger is completely blocked.

The following parameters should be checked, registrated and adjusted regularly:

Parameter	Check every...	Check for...
Gas metering device	10 min	Volume
Pressure drop over P&T sampling unit	10 min	<100 mbar
Sample gas temperature after vac. pump	10 min	max. 10 °C
Sample gas flow rate	10 min	... Nm ³ /h
Gas temperature at the sampling nozzle	30 min.	temperature
Gas temperature after condensation	30 min.	temperature
Ice bath temperature (condensation bottle)	30 min	0°C
Brine temperature (tar absorption bottles)	30 min	-4°C ... -3°C

The listed parameters should be monitored and documented by attentive observation and control of the corresponding instruments. These procedures should be done for each sampling units separately. An example of a test protocol is given in the appendix 6.7.

5.2.2 Sampling for producer gas analysis

If continuous gas analysis is done, the instrument reading should be carefully monitored. Recalibration should be done every 48 hours approximately, but not during a P&T sampling period. The gas preconditioner (see figure 4-1) needs adaptation in cases where the gas sampling temperature is above the gas dew point and tar concentrations are high. High gas temperatures and/or tar contents require additional condensation and absorption bottles or tar adsorbers.

For on- and off-line GC gas analysis, the chromatograms must be carefully observed. Moisture and/or excessive tar adsorption on the columns can lead to shifts in the residence time and/or response factors. Recalibration should be done every 10 hours of continuous operation (but not during a P&T sampling period) or after 10 off-line analysis.

Discontinuous gas sampling should always be made parallel with two glass containers. In cases where leakages occurred or unexpected gas compositions have been measured (e.g. the GC calibration is not valid any more), a backup sample is available.

5.2.3 Gasifier performance protocol

Beside the registration of the sampling conditions, the gasifier operating conditions should also be regularly monitored and registered. The following actions have to be included (list not completed):

- Startup time of gasifier operation
- Fuel feeding rate
- Pressure drops in the overall system (gasifier, gas cleaning unit)
- Temperatures (gasifier wall temperatures, gas temperatures,...)
- Start and stop of sample collection
- Time of irregular interrupts or shutdowns

For the gasifier performance, separate test protocols must be used. It is beyond the scope of this guideline to discuss these test protocols.

5.3 Postsampling procedures at the sampling site

An overview on the on site procedures after the P&T sampling is given in figure 5-1. The cleaning of the tubes and the gas lines should be made as quickly as possible after the end of the P&T sampling. Totally, three washing procedures are made:

a) Washing procedure with distilled water

1. Disconnect all the sampling lines from the heated ball valve to the first tar absorption bottle (figure 3-2).
2. Determine the total mass of condensate generated during the sampling period. The producer gas moisture is determined using the condensate mass and the sampled producer gas volume (see chapter 3.3). Eventually, the residual gas moisture after condensation is also included. Pour the aqueous condensate in a glass storing bottle.
3. Wash the sampling lines, the water cooler and the diving pipe with as little distilled water as possible. Add the wash solution to the aqueous condensate. Determine the total mass of aqueous solution (designated as solution AP).

Attention: The nozzle can not be removed before the shut down of the gasifier because no oxygen is allowed to get in the producer gas duct system.

4. Until further analysis, the glass bottle should be stored in the refrigerator.

b) Washing procedure with anisole

1. After the water washing procedure, wash the sampling lines, the water cooler (if present) and the diving pipe with as little anisole as possible. Pour the solution into the moisture condensation bottle.
2. Disconnect the piping between the tar absorption bottles. Pour the content of the first and second tar absorption bottle into the anisole storing bottle. Use the anisole solution of the last washing bottle to preclean the first and second tar condensing bottle.
3. Use fresh anisole to wash all the tar absorbing and the drop separator bottles as well as all the piping connections. Use as few anisole as possible.
4. Pour the remaining anisole solutions into the moisture condensation bottle and shake well.
5. Pour the organic solution from the moisture condensation bottle into the organic sample storing bottle.
6. Use fresh anisole to wash the moisture condensation bottle and add it to organic sample storing bottle. Use as few anisole as possible.
7. Determine the total mass of the anisole solution (designated a solution OP1).

Droplets of remaining water and/or tar and some particulates remain on the surfaces after the anisole washing procedure. An almost overall cleaning can be achieved with an additional acetone washing.

c) Washing procedure with acetone

1. After the anisole washing procedure, all sampling lines and bottles are carefully washed with as few acetone as possible.
2. Pour the acetone solution into into a separate sample storing bottle (designated as solution OP2).
3. Determine the total mass of the acetone solution OP2.

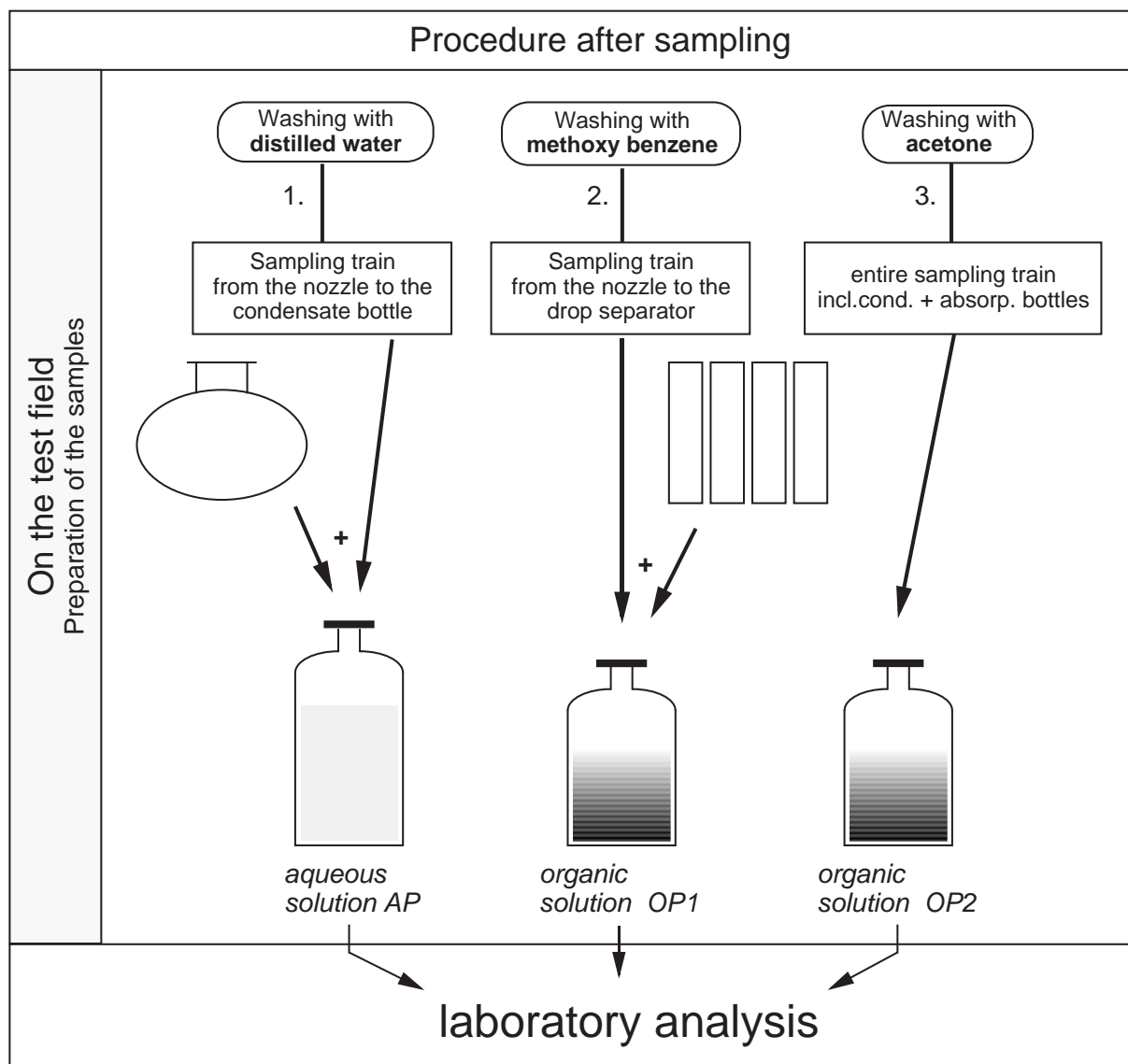


figure 5-1: Overview on the procedures after the P&T sampling

The result of the P&T sampling and the post sampling washing procedures are three bottles, one containing an aqueous solution (AP) and two containing an organic solutions (OP1 and OP2), and a glass fibre thimble filter with residual particulates.

Remark:

- All storing bottles must be made of glass and the lids must be gas tight. At least the covers for the organic solutions must have a PTFE seal (supplier: e.g. E. Merck AG; prod. no. for lid: 30 355 02) to avoid liquid contamination.

5.4 Sample preparation in the laboratory

The analysis train in the laboratory is shown in **Fehler! Verweisquelle konnte nicht gefunden werden.** In the laboratory, mostly preparatory steps as well as the quantitative determination of solid residues are made. Most of the procedures and methods are standard methods in chemical laboratories. At the end of the laboratory preparation, the total amount of solid residues is known and a number of liquids are generated for the quantitative analysis of the heavy tars, PAH, phenols and condensate components.

In the following, a summarized description of the sample treatment is given. Preliminary remarks:

- For the laboratory analysis, anisole from the same batch should be used if possible.
- From the original anisole stock, an extra analysis should be made to determine the amount of heavy tars and the PAH in the pure solvent.
- It is advantageous to treat a series of samples simultaneously. By doing this, work is more effective and costs can be reduced.
- Particle filtration from the sampling liquids is made with a slow filtration cellulosic filter (supplier: Schleicher&Schuell; prod. 589/3)
- From the organic solution OP2 (acetone washing solution from P&T sampling unit), the major part of the solvent is carefully evaporated at approx. 56°C. The remaining liquid is joined with the organic solution OP1 to give one organic solution (designated as OP) for further treatment.
- The amount and the fractions of all solids and liquids must be determined and carefully noted. These values are needed to calculate the absolute amount of the components in the gas.

Example for the determination of phenols: if 17% of the aqueous filtrate are used, also 17% of the organic phase filtrate must be used; the absolute amount of phenols determined in the neutralized aqueous extract accounts to 17% of the total amount in the gas.

- The extraction of aqueous liquids with anisole or vice versa is time consuming since the liquid densities are comparable ($\rho = 0.998$ for water vs $\rho = 0.996$ for anisole at 20°C). Generally, the phase separation takes several hours.
- All analysis and backup samples should be kept in gas tight bottles in the refrigerator until further analysis.
- For the determination of the solid residues *Solid A*, *Solid B* and *Solid C* (see **Fehler! Verweisquelle konnte nicht gefunden werden.**), the samples have to be dried until weight constancy.
- The amount of the organic phase mixture OPM (**Fehler! Verweisquelle konnte nicht gefunden werden.**) should be as large as possible. The determination of PAH's requires at least 10 ml and

the determination of heavy tars should be made with at least 50 ml. The organic backup sample should be at least 10 ml.

- For all results, SI units (kg, s, m) must be used. Volumes must always be given on a moisture free basis and at standard conditions (0°C, 1013 mbar).

a) Particulate filtration

1. For the separation of solid particulates in the sample solutions AP and OP, a glass microfibre membrane filter is used (supplier Whatman; Type GF/C). Before filtration, the membrane is dried at 105°C and weighed in a defined atmosphere.
2. The aqueous solution is first passed through the membrane filter using vacuum. Eventually, the filtering must be repeated if some of the particulates pass through the membrane at the beginning of the filtration process. After the filtration of the aqueous solution, filtrate the anisole phase OP through the same membrane filter.
3. The membrane filter with the (solid) filtration residue is extracted with 100 ml methylen chloride (CH₂Cl₂) in a Soxhlet apparatus at 40°C for at least 8 hours. For the extraction, preferably Soxhlet tubes made of glass fibres are used. From a defined fraction of the Soxhlet extract, the methylen chloride is carefully evaporated at approx. 40°C. The residual liquid is added to the organic phase mixture OPM. The Soxhlet residue (Solid A) is a fraction of the total particulates.

Remark:

- Due to the high amount of very small particulates, the filtration of the aqueous and the organic phase through the glass fibre filter can be time consuming.

b) Treatment of the liquid filtrates

1. From the filtrate of the aqueous phase AP, a sample for the condensate analysis (e.g. 200 ml) may be taken. Condensate analysis is optional.
2. Preparation of the *organic phase mixture OPM*:
 - A (defined) fraction of the organic phase filtrate OP (anisole as solvent) is used for the extraction of organic components from the aqueous phase filtrate AP. If the amount of the organic solution is too small for the ratio aq:org = 2:1, use fresh anisole to make up.
 - The aqueous phase from the extraction is extracted with fresh methoxy benzene, the organic phase is extracted with 100 ml of distilled water.
 - The two aqueous phases are joined and used for the determination of the water soluble organic residue (Solid B). Solid B is the evaporation residue of the extracted aqueous phases.
 - The two organic phases from the extraction is used for the preparation of the organic phase mixture OPM (the OPM additionally contains the evaporation residue from the Soxhlet extraction).

3. Preparation of the *sample for phenol analysis*:

- A (defined) fraction of the alkalized aqueous phase filtrate AP (e.g. 10 M KOH added to AP until pH = 13) is used to extract acidic organic components from a defined fraction of the organic phase filtrate OP.
- After the alkaline extraction process, the aqueous solution is neutralized. The organic extract is not used anymore.
- The sample for the phenol analysis must be free of organic solvent, the pH should be neutral. Phenol analysis should be done within a few hours.
- The amount of the sample for phenol analysis should be approx. 100 ml.

Remark:

- For the condensate analysis, parameters such as pH, DOC, NH₃, phenols etc can be determined. These parameters can be used to characterize condensates from a wet gas cleaning system.
- The condensate analysis is made using standard water analysis procedures.

c) Determination of heavy tar content

- The amount of heavy tars is determined as a gravimetric value from the heavy tar sample (see **Fehler! Verweisquelle konnte nicht gefunden werden.**) by carefully distilling off the solvent anisole ($t_b = 153^\circ\text{C}$). The evaporation is made in vacuum (10 to 20 mbar absolute pressure) over a heated water bath at $\approx 85^\circ\text{C}$. The evaporation is made until completion of solvent evaporation. As soon as all the solvent is evaporated, tar evolution begins with a weakly visible smoke formation. At this point, the evaporation is stopped. The sample is cooled and weighed on a micro balance.
- The heavy tar content in the gas is given by the absolute amount found in the evaporation process, the mass fraction of aqueous and organic phase used for the preparation of the tar analysis sample and the amount of gas sampled.

d) Determination of PAH content

- The amount of PAH is determined quantitatively from the organic phase mixture OPM using approved GC/MS procedures.
- Determination should be made for the 16 PAH according to the EPA list (see appendix 6.9). The absolute amount of each PAH must be documented.
- The PAH content in the gas (16 individual PAH's) is given by the absolute amount found in the GC/MS analysis, the appropriate mass fraction of the OPM and the amount of gas sampled.

e) Determination of total phenol content

- The amount of phenols is determined quantitatively from the prepared sample for the phenol analysis using either photometric or GC/MS procedures. The photometric method (e.g. according to ASTM D 1783-91) gives a sum of the most prominent phenols, whereas with GC/MS single phenolic components can be quantified.
- The phenol content in the gas is given by the absolute amount found in the analysis, the mass fraction used for the preparation of the analysis sample and the amount of gas sampled.

f) Determination of the content of the water soluble organic residue

- The solid residue B found during the laboratory processing of the extracted aqueous phase is composed of purely organic matter (see chapter 6.5). The liquid fraction used to prepare solid B must be integrated in the calculation.
- The water soluble organic content in the gas is given by the absolute amounts of solid B found and the amount of gas sampled.

g) Determination of particulate content

- The total amount of solid is the weighed sum of solids A and C determined during the laboratory processing. The mass fraction used to prepare the solid samples must be carefully integrated in the calculation.
- The particulate content in the gas is given by the absolute amounts of solid A and C found and the amount of gas sampled.

Remark:

- The particulates (Solid C) on the glass fibre thimble filter is free of heavy tars and does not necessarily need to be treated by Soxhlet extraction. However some light tars, anisole and PAH's can condense on the filters so that the drying temperature of the thimble filters should be approx. 150°C. Anisole will completely evaporate at 150°C within less than 1 hours. The filter thimbles must be made of glass fibres since cellulosic filter thimbles will partially oxidize.
- The amount of PAH compounds found on the (untreated) thimble filters is less than 5% of the total PAH concentration in the gas. The correction factors for the PAH's are given in appendix 6.3.

6 Appendix

6.1 Sampling nozzle shapes and dimensions

The parameters for the dimensioning of the sampling nozzle are given in figure 6-1. The nozzle are made of either stainless steel. Please note the following constructional instructions:

$$l_1 \geq \frac{d_{\text{wirk}}}{2} \qquad s \leq \frac{d_{\text{wirk}}}{12}$$

The dimensions of d_{wirk} can vary between 5 and 30 mm.

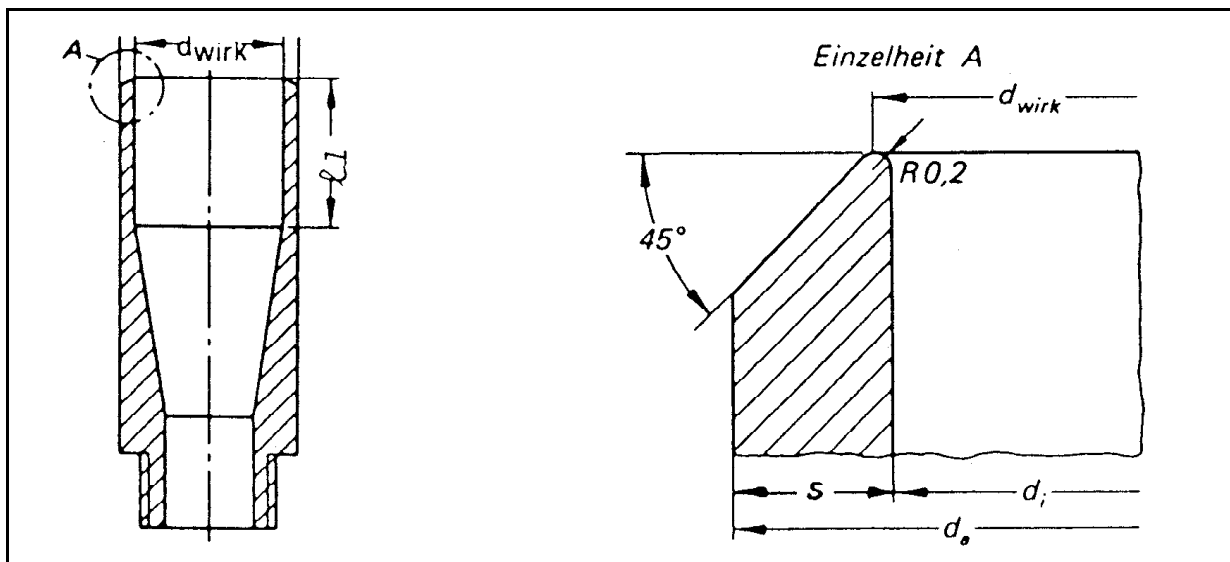


figure 6-1: Dimension parameters for nozzles suitable for isokinetic gas sampling

Possible shapes of the sampling nozzles are given in figure 6-2. We recommend to use nozzle shapes of type A.

The nozzles dimensions and shapes displayed in figure 6-1 and figure 6-2 are used in Switzerland for particulate emission measurements from incineration processes [BUS 1987]. The nozzle shapes are not strictly identical to the german VDI Richtlinie 2066. But it has been shown by several accredited companies that using this nozzles give identical results.

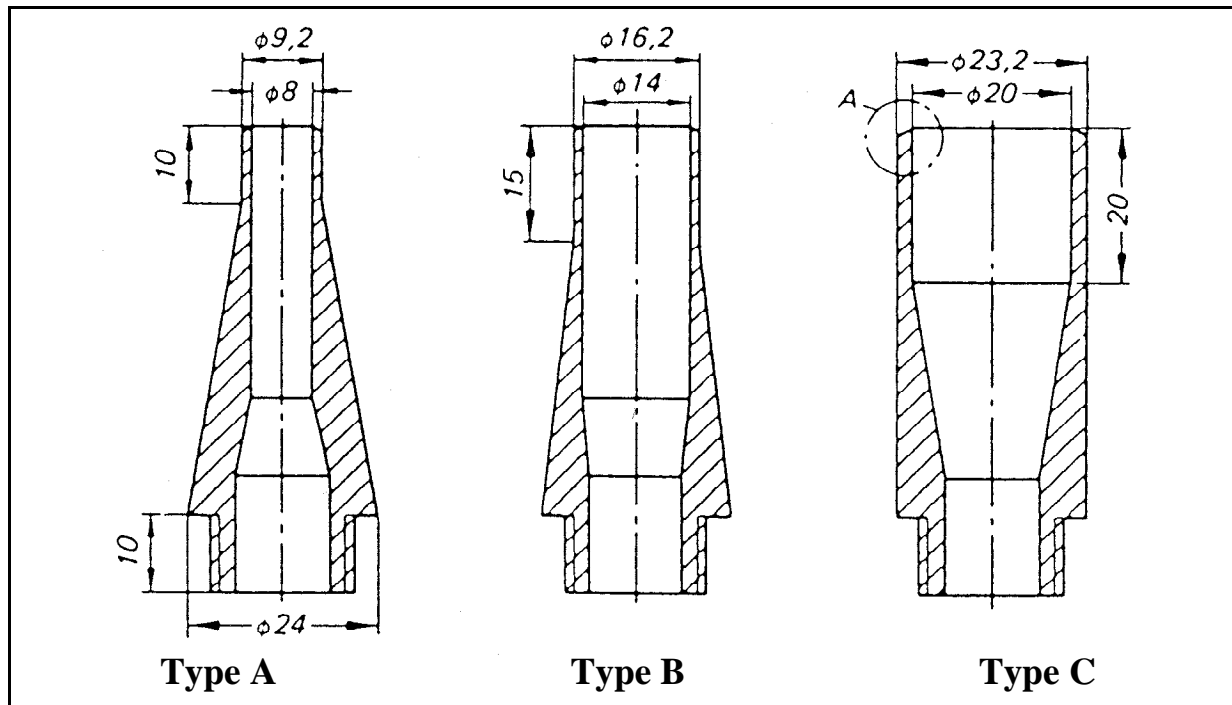


figure 6-2: Examples of different sampling nozzles shapes

6.2 Dimensions of tar absorption bottles

The dimensions of the tar absorption bottles using anisole as a solvent (see also figure 3-2) are given in figure 6-3. Although we think that the dimensions of the absorption bottles are not critical with respect to comparability of test equipment, we recommend to use comparable dimensions.

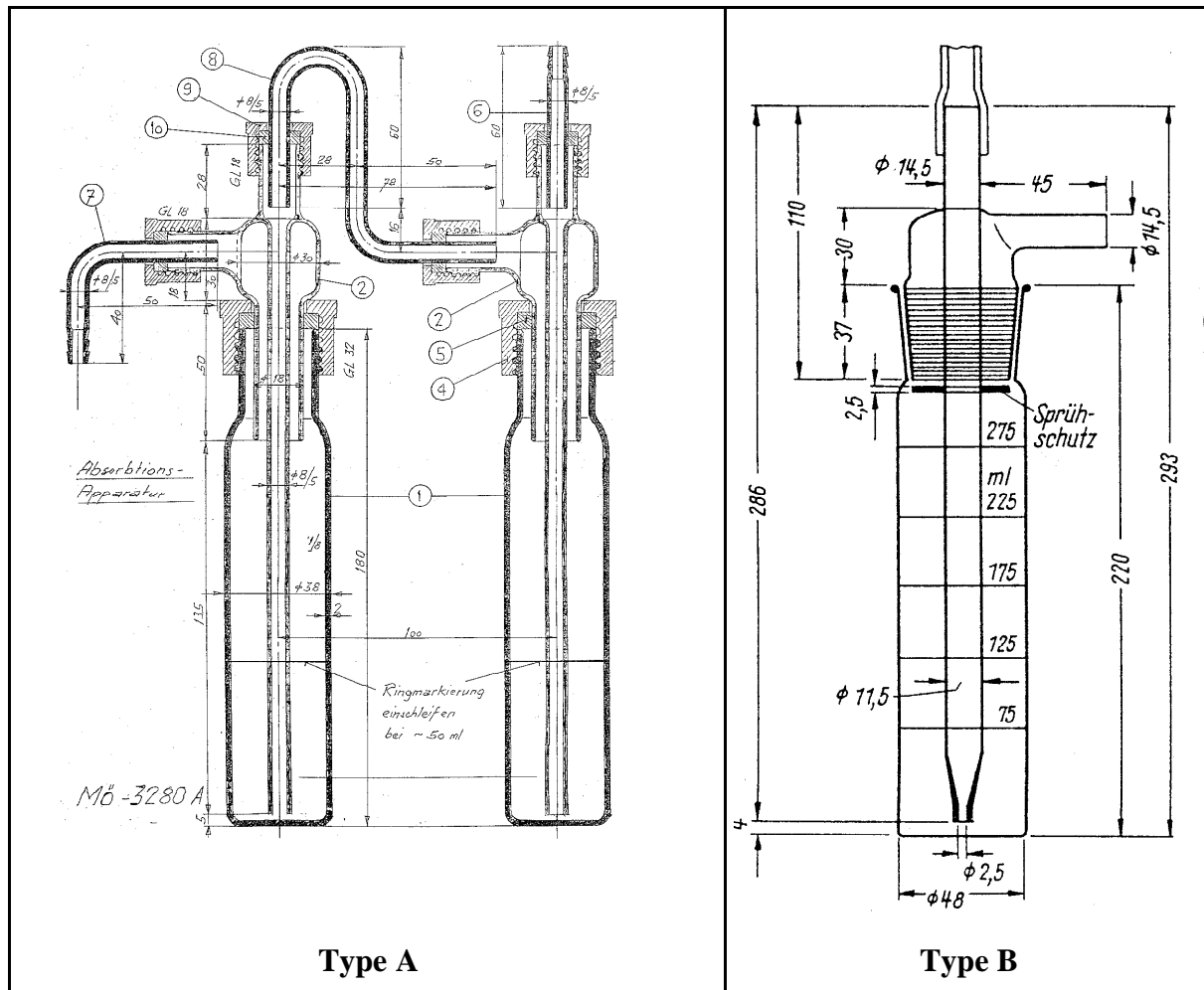


figure 6-3: Dimensions of the tar absorption bottles, type A and type B

Remarks: Absorption bottle type A is used for the P&T unit in this guideline; Type B is a standard gas impinger; Description of standard impinger can be found e.g. in VDI 2452 Blatt 1; With flow rates $>1.8 \text{ m}^3/\text{h}$, standard impingers can be used also for particulate separation.

As an alternative, standard impingers can be used. The inner diameter of the diving pipe in the first tar absorption bottle must be 4 to 5 mm. Smaller diameters can lead to tube blocking due to ice formation from residual moisture.

6.3 Correction factors for the determination of the PAH concentrations

The treatment of the glass fibre thimble filters (see chapter 5.4) generally does not include soxhlet extraction. On the thimble filters, mainly residual particulates (Solid C) are found. However some light tars, anisole and PAH's can condense on the filters and hence these compounds are lost during thimble filter drying at app. 155°C. The loss of PAH due to this drying process has been experimentally determined both for raw and clean producer gas. The correction factors are given in the following table. The PAH values found from the absorption/extraction train can be multiplied by these correction factors. For naphthalene which is the dominant PAH found, 5% of the total naphthalene present in the gas are found on the thimble filter. It should be kept in mind that the sum of the concentrations from chrysene to benzo(g,h,i) perylene generally amounts to less than 3% of the total PAH concentration in the producer gas.

P&T sampling site	Amount of PAH found on thimble filter			Correction factor
	Raw gas %	Clean gas %	Mean value %	
Naphthalene	5.6	4.7	5.1	1.051
Acenaphthylene	8.4	12.4	10.4	1.104
Acenaphthalene	13.4	16.9	15.1	1.151
Fluorene	10.8	13.9	12.3	1.123
Phenanthrene	8.7	14.7	11.7	1.117
Anthracene	14.9	14.3	14.6	1.146
Fluoranthene	12.8	14.0	13.4	1.134
Pyrene	14.7	15.5	15.1	1.151
Chrysene	11.2	15.0	13.1	1.131
Benzo(a)anthracene	15.6	16.5	16.1	1.161
Benzo(b)fluoranthene	11.3	11.7	11.5	1.115
Benzo(k)fluoranthene	13.0	12.2	12.6	1.126
Benzo(a)pyrene	17.1	11.5	14.3	1.143
Indeno(1,2,3-cd)pyrene	12.6	4.7	8.7	1.087
Dibenzo(a,h)anthracene	3.2	0.7	2.0	1.020
Benzo(g,h,i)perylene	9.6	5.4	7.5	1.075

6.4 Chemical composition of the heavy tar residue

The determination of the heavy tar concentration is done is a gravimetric value with this sampling method. With the gravimetric method, no information about the chemical nature of the heavy tars can be derived. Hence a chemical analysis has been made with a heavy tar residue to get some insight view of the heavy tar composition. The heavy tar residues analyzed originate from the producer gas used for fabric filter cleaning test runs. The fabric filter test rig was fed with a slipstream of the raw producer gas from the IISc/Dasag open top gasifier in Châtel-St-Denis in Switzerland.

The most relevant organic compounds found in the heavy tar residue are the PAH (according to the US EPA list) with approximately 20 weight% (table 6-1; figure 6-4).

Test run Sampling site	GF2 raw [g/kg]	GF2 clean [g/kg]	GF3 raw [g/kg]	GF3 clean [g/kg]	GF4 raw [g/kg]	GF4 clean [g/kg]
Anisole			5.0	0.4	6.0	4.0
Sum of phenols	65.5	64.2				
Naphthalene		100.0				
Acenaphthylene		49.0				
Acenaphthalene		3.10				
Fluorene		15.0				
Phenanthrene		29.0				
Anthracene		6.30				
Fluoranthene		6.90				
Pyrene		5.90				
Chrysene		0.85				
Benzo(a)anthracene		1.50				
Benzo(b)fluoranthene		0.59				
Benzo(k)fluoranthene		0.29				
Benzo(a)pyrene		0.42				
Indeno(1,2,3-cd)pyrene		0.11				
Dibenzo(a,h)anthracene		0.11				
Benzo(g,h,i)perylene		0.06				
Sum of PAH		219.1				

table 6-1: Chemical composition of heavy tar (HT) residues

Remarks: The phenols were determined as a photometric value acc. to ASTM D 1783-91; The amount of residual anisole in the HT was determined by GC/FID.

The dominant compound found in the PAH present in the heavy tars residue is naphthalene (figure 6-5).

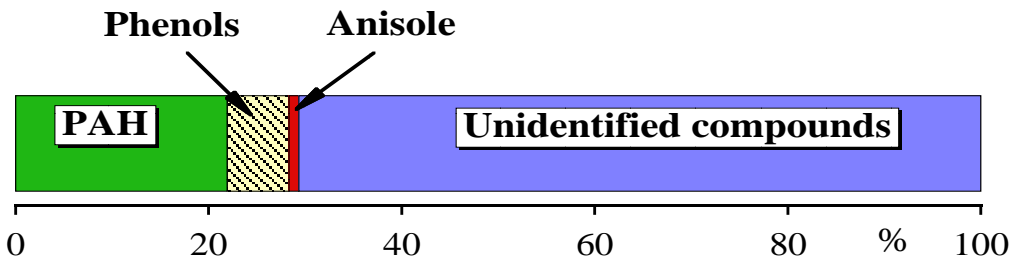


figure 6-4: Chemical composition of the heavy tar residue

Approximately 6 weight% of the heavy tars residue is found to be of phenolic nature and only negligible amount of the solvent anisole remain in the heavy tar residue. More than 70 weight% of the heavy tars still remain unidentified.

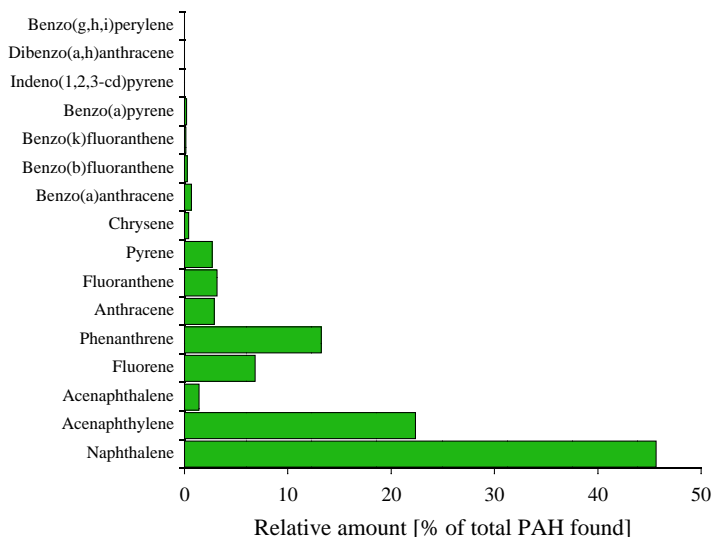


figure 6-5: Relative amount of individual PAH compounds found in the PAH fraction of the heavy tar residue

6.5 Composition of the solid residue from the water phase

As with the heavy tars, the determination of the water soluble residue is determined as a gravimetric value from the aqueous phase extracted with anisole. With gravimetric methods, no information about the chemical nature of the solid residue can be derived. The solid residue from the aqueous phase can be both inorganic or organic.

The aqueous phase residues analyzed originate from the clean producer gas after a fabric filter test rig. The fabric filter test rig was fed with a slipstream of the raw producer gas from the IISc/Dasag open top gasifier in Châtel-St-Denis in Switzerland.

The aqueous phase residue contains between 1 and 1.4 wt% of phenols (table 6-2). The mass of phenols found in this residue is 0.7 to 1.4% of the total mass of phenols present in the gas. The water soluble solid residue contains more than 47 wt% of carbon and 7 wt% of hydrogen which indicates that the residue is mainly of organic nature.

Test run Sampling site	GF2 raw [g/kg]	GF2 clean [g/kg]	GF3 raw [g/kg]
Sum of phenols	1.06	1.40	
Elemental composition			
Carbon			467.9
Hydrogen			70.3

table 6-2: Chemical composition of water soluble solid residues

Additionally, a thermogravimetric analysis (TGA) of one aqueous phase residue has been made (figure 6-6). The sample is heated from room temperature to 600°C in nitrogen atmosphere. The weight decreases steadily with increasing temperature. At 600°C, 70 wt-% of the original mass is evaporated or pyrolyzed. Changing the atmosphere from nitrogen to air at 600°C results in an almost complete oxidation of the residue. The non oxidated mass amounts to 0.3 wt-%.

From the TGA experiment, it can be clearly seen that the solid residue from the aqueous phase is undoubtedly of organic nature although the aqueous phase has been extracted with anisole (see chapter 5.4). Most presumably, the solid residue from the aqueous phase consists of highly oxygenated products from wood pyrolysis such as polysaccharides.

Sample: 23441, VERENUM
Size: 32.8360 mg
Method: Verenum, HR=5°C/min
Comment: Balance 20ml/min, Purge 80ml/min, N2/Luft

TGA

File: C: 23441.002
Operator: ZA
Run Date: 10-Aug-98 14: 26

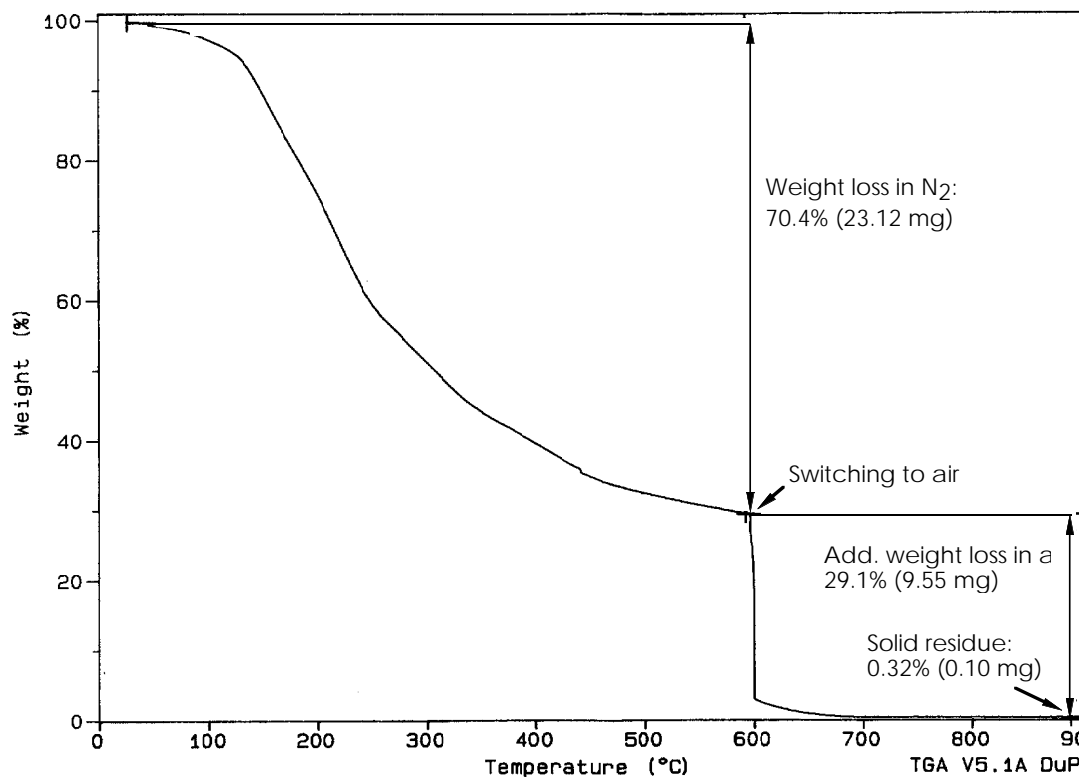


figure 6-6: Thermogravimetric analysis of a solid residue from the aqueous phase

Remarks: The gas sample originates from a slip stream of the raw producer gas of the IISc/Dasag gasifier in Châtel-St-Denis (Switzerland); Test run identification is GF3, raw.

6.6 Tar deposits found in IC engines

Power production in IC engines is one of the most promising application of cocurrent fixed bed gasifiers. Depending on the type of gasifier, gas cleaning, air mixing and engine used, some deposits are normally found in various parts of the installation after longer periods of operation.

For the assessment of the „tar“ values used within this guideline, two type of deposits found in the IISc/Dasag installation in Châtel-St-Denis (Switzerland) were analyzed. One deposit was found in the engine manifold just before the air/fuel mixture enters the combustion chamber. Another small amount of deposit was found on the cylinder wall of the engine after several hundreds of operating hours. In both deposits, the amount of PAH (sum of 16 compounds according to the US EPA list) and heavy tars were determined, and in the cylinder deposit also the amount of solids.

The ratio of heavy tars / PAH is much higher in the two engine deposits than in the clean producer gas (figure 6-7). In the cylinder deposit, approximately 200 times more heavy tars are found than PAH. T compounds are deposited more preferentially than the PAH compounds both in the engine manifold and in the cylinder.

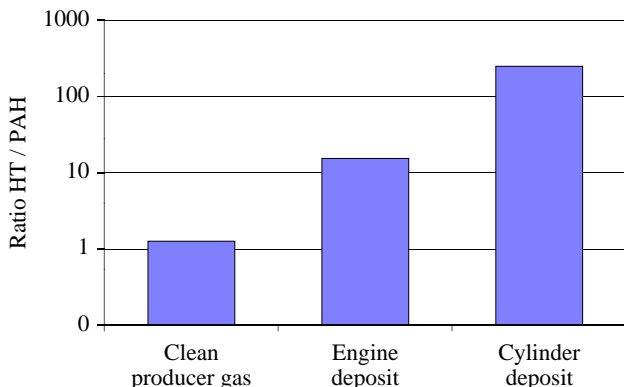


figure 6-7: Ratios of Heavy tars / PAH found in the clean producer gas, in the engine manifold and in the cylinder from the IISc/Dasag gasifier installation in Châtel-St-Denis (Switzerland)

The cylinder deposit contains more than 85 wt% of heavy tars and minor amounts of PAH compounds (figure 6-8). Approximately 12 wt% of the cylinder deposit is found as solid matter. The ratio of particles / heavy tars in the clean gas is comparable to the ratio found in the cylinder deposit.

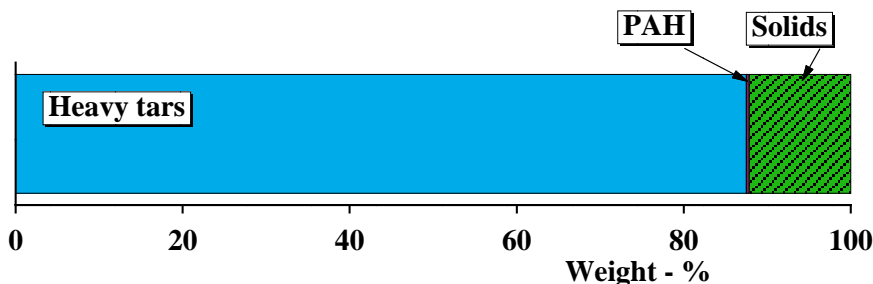


figure 6-8: Composition of deposit in the engine cylinder [Giordano 1988]

The high amount of heavy tars found in the engine manifold and on the cylinder wall indicate that the heavy tar as defined in this guideline are good indicator of the producer gas quality.

6.7 P&T sampling protocol

On the following pages, an example of a test protocol during sampling of particulates and tars is given. The documentation of the actual sampling conditions and operational events is considered as an important part of the total sampling procedure.

P+T - Measurement for Biomass Gasifiers

Examinators: _____

Gasifier Type/Site: _____ Date: _____

Test No.: _____

Gasifier parameter

Actual power output: _____ kW (max. kW)
(based on fuel fuel input)Raw gas flow rate (dry): _____ [Nm³/h] (if calc., spec. gas rate: _____ Nm³/kg wet

Gasifier start *: _____ stationary since *:

Fuel type *: _____

Fuel moisture *: _____ [%, dry basis]

Actual fuel feeding rate *: _____ [kg wet/h]

Sampling parameters

Sampling site (hot/cold end): _____

Ambient temperature: _____ [°C]

Ambient pressure: _____ [mbar]

Duct diameter: _____ cm

Diameter of sampling nozzle: _____ mm

Sampling tube temp. before cooler: _____ [°C]

Water condensation bottle: _____ [g]

Condensate storing bottle: _____ [g]

Anisole storing bottle: _____ [g]

Acetone storing bottle: _____ [g]

Filter tubes: _____ [g]

tara weight	netto weight	final weight 1	final weight 2
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Netto weight: Mass of water initially filled in the condensation bottle*Final weight 1:* Total mass of bottle plus liquid after sampling, **excl.** washing solutions*Final weight 2:* Mass after sampling, **incl.** washing solutions*Filter tube pretreatment:* hours at°C*Filter tube posttreatment:* hours at°C

Summary protocol

Generated condensate mass: _____ [g]

Total mass of water: _____ [g]

Total mass of anisole: _____ [g]

Total mass of acetone: _____ [g] (acetone: cleaning solvent)

Total particle mass in filter tubes: _____ [mg]

Normalized sampled gas flow rate: _____ [Nm³/h]

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: _____

Sampling site (hot/cold end): _____

Date: _____

Test No.: _____

Rated sampling gas flow rate (dry): _____

Nm³/h (calculated set point; 10% over isokinetic)

P+T sampling start: _____

P+T sampling end: _____

Gas meter protocol					
Time [hh:mm]	Gas temperature in duct [°C]	Reading gas meter [m ³]	Temperature in gas meter [°C]	Δp pump [mbar]	Sampling Flow rate [Nm ³ /h]

Remarks: - Condensation train: cooler (-4°C), condensator (0°C), drop separator.
 - Tar absorption train: 3 washbottles with anisole (bottles of type Drechsel, -4°C) and drop separator.
 - Before test run, heated sampling nozzle was cleaned by N₂ flushing.

Averaged sample flow rate: _____ Nm³/h
 Total amount of gas sampled: _____ Nm³
 Total sampling duration: _____ hours
 Mean gas meter temperature: _____ °C

Calculation of the gas dew point

Total amount of condensed moisture: _____ g
 water vapour equivalent: _____ Nm³
 water vapour pressure: _____ bar
 Calculated dew point of the gas: _____ °C

6.8 150 hours duration test program

For the assessment and comparison of different gasifier types, a 150 hours continuous and noninterrupted gasifier operation is considered as a good performance indicator of the integrated gasifier / gas cleaning and where existing of the IC engine system. In general this means that the gasifier is operated automatically and adequate safety precautions are taken. Today, only short term experiments of several hours are available from fixed bed gasifiers. Within the 150 hours, the gasifier will reach an „equilibrium“ state which represents almost commercial operation conditions. Possibly, the gas quality is also different in a equilibrium state compared to a short term test run. Only well developed gasifiers will be able to run for 150 hours uninterrupted.

However, the credibility of the gasifier performance is only given if the event history is well documented and a certain standard for the test program is passed. Therefore, a uniform test program has been accepted within the EU project group „Fixed bed gasification of SGF“ (contract JOR3-CT95-0084). The 150 hours test program is as follows:

1. Sampling equipment and analysis procedures must be uniform and the procedures must be made according to this guideline.
2. Operation test profile is:
 - 130 hour test period with full load
 - 20 hours test period with reduced load (e.g. at 50%)
3. Actions and parameters that must to be determined:

A total of 4 particulate and tar (P&T) samplings is made during the 150 hours test.

- The first sampling is made 8 to 12 hours after gasifier ignition
 - The second sampling is made 70 to 80 hours after ignition
 - The 3rd sampling is made 110 to 125 hours after ignition
 - The 4th sampling is made 8 to 12 hours after load reduction
4. Sampling must be done at least at the hot end (after the gasifier outlet). In cases where a gas cleaning system is installed, at least 1 additional sampling must be done after the gas cleaning parallel to one of the hot end samplings.
 5. Sampling duration is at least 4 hours.
 6. From each sample, the following parameters are determined in the laboratory:
 - Particulate content in the gas
 - Content of the water soluble organic residue in the gas
 - Content of heavy tars in the gas (heavy tars as defined in this guideline)
 - Content of phenol (as the sum of phenols; photometric method) in the gas
 - Content of 16 PAH (acc. to EPA list; appendix 6.9) in the gas
 7. A continuous gas analysis during the 150 hours test is preferable. Gas analysis starts at the same time as the gasifier is ignited (cold start). If discontinuous gas sampling is done, a sampling must be made at least every 10 hours, the first sample to be taken not later than 1 hour after gasifier

ignition. The discontinuous gas sampling should represent an average gas composition of approx. 5 minutes (i.e. the water in the glass should be displaced within 5 min).

6.9 US EPA list of polyaromatic hydrocarbons (PAH)

Chemical compound	Formula	mol. weight	m.p. °C	b.p. °C
Naphthalene	C ₁₀ H ₈	128.19	81	218
Acenaphthylene	C ₁₂ H ₈	152.21	92	270
Acenaphthene	C ₁₂ H ₁₀	154.21	93	279
Fluorene	C ₁₃ H ₁₀	166.23	114	298
Phenanthrene	C ₁₄ H ₁₀	178.24	97	340
Anthracene	C ₁₄ H ₁₀	178.24	216	340
Fluoranthene	C ₁₆ H ₁₀	202.26	110	375
Pyrene	C ₁₆ H ₁₀	202.26	150	393
Chrysene	C ₁₈ H ₁₂	228.30	252	448
Benz(a)anthracene	C ₁₈ H ₁₂	228.30	152	435
Benzo(b)fluoranthene	C ₂₀ H ₁₂	252.32	168	-
Benzo(k)fluoranthene	C ₂₀ H ₁₂	252.32	217	480
Benzo(a)pyrene	C ₂₀ H ₁₂	252.32	179	495
Indeno(1,2,3-cd)pyrene	C ₂₂ H ₁₂	276.34	163	-
Dibenzo(a,h)anthracene	C ₂₂ H ₁₄	278.35	267	524
Benzo(g,h,i)perylene	C ₂₂ H ₁₂	276.34	278	>500

US EPA's „Priority pollutants list“ for PAH

6.10 Addresses of contact persons

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6.11 Addresses of contact persons

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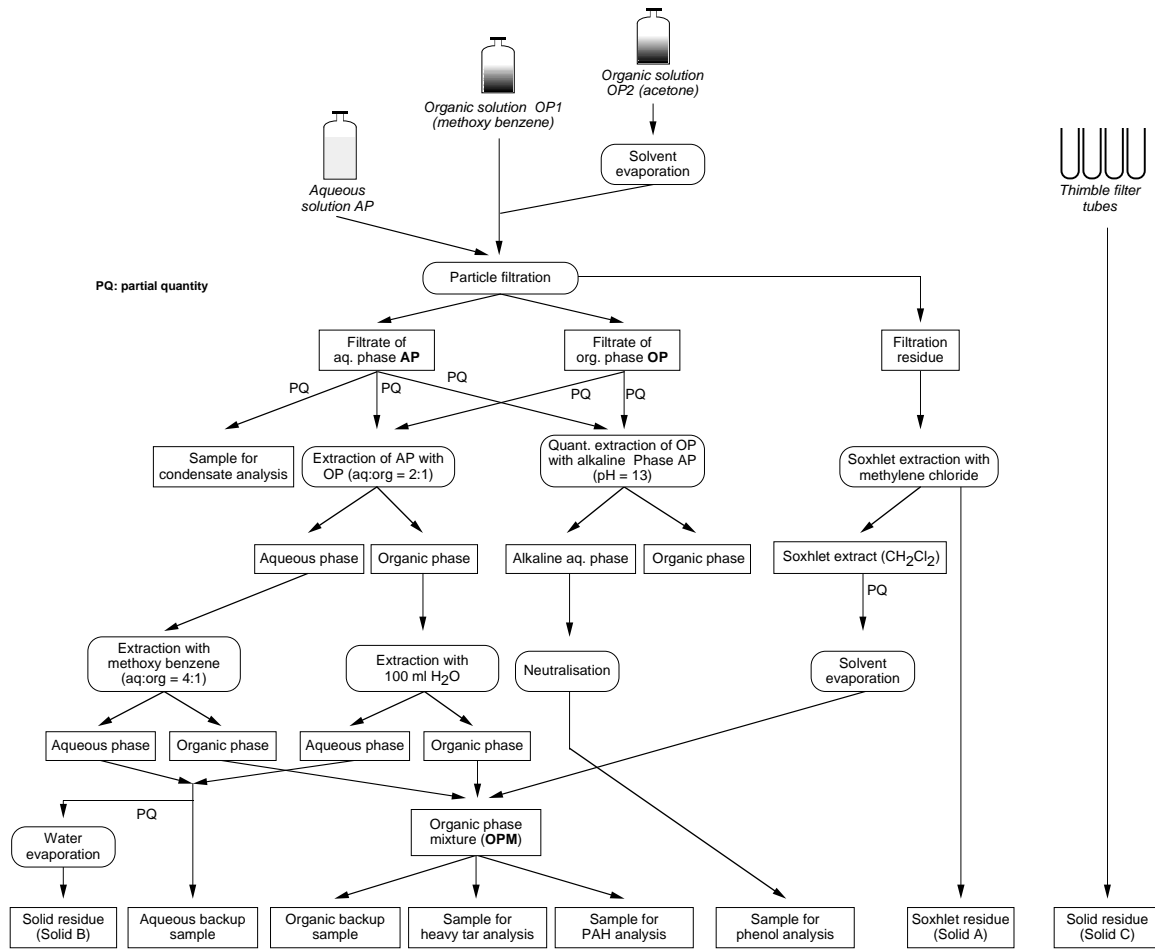
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CH-8006 Zürich Fax.: +41 1 364 14 21

☞ Copies of this guidelines can be ordered from Verenum, Zurich

7 Literature

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Schematic of the laboratory analysis