# Guideline for sampling and analysis of tars and particles in biomass producer gases

J.P.A. Neeft<sup>1</sup>, H.A.M. Knoef<sup>2</sup>, G.J. Buffinga<sup>2</sup>, U. Zielke<sup>3</sup>, K. Sjöström<sup>4</sup>, C. Brage<sup>4</sup>, P. Hasler<sup>5</sup>, P.A. Simell<sup>6</sup>, M. Suomalainen<sup>6</sup>, M.A. Dorrington<sup>7</sup>, C.Greil<sup>8</sup>

- <sup>1</sup>: ECN, P.O. Box 1, 1755 ZG Petten, The Netherlands
- <sup>2</sup>: BTG, P.O. Box 217, 7500 AE Enschede, The Netherlands
- <sup>3</sup>: DTI, Kongsvang Allé 29, DK-8000 Aarhus, Denmark
- <sup>4</sup>: KTH, Teknikringen 42, 10044 Stockholm, Sweden
- <sup>5</sup>: Verenum, Langmauerstrasse 109, 8006 Zürich, Switzerland
- <sup>6</sup>: VTT, P.O. Box 1601, 02044 VTT Espoo, Finland
- <sup>7</sup>: CRE Group, Stoke Orchard, GL52 4RZ Cheltenham, UK
- <sup>8</sup>: Lurgi, Lurgiallee 5, D-60295 Frankfurt am Main, Germany

**ABSTRACT** This paper reports the further development of a Guideline (formerly Protocol) for sampling and analysis of tars from biomass producer gases. This Guideline is being developed in a project in the European Fifth Framework Programme with additional partners from Switzerland and North America.

This paper gives the outline and principle of the Guideline. The Guideline is based on isokinetic sampling of particles and tar from the main producer gas duct, particle filtration at high temperature, gas cooling in a liquid quench, tar absorption in a solvent at low temperatures, an optional backup adsorber, and flow measurement and control. The Guideline gives a definition for Gravimetric tar which is the tar number to be determined by the Guideline. Besides, the Guideline gives procedures for compound analysis by GC-MS or GC-FID.

Moreover, in this paper the major choices that were made to come to the first version of the Guideline are explained. Finally, at the end of the paper it is described how and on what time scale the development of the Guideline will be completed.

This paper does not contain the full text of the Guideline. This full text will be available on the Internet at www.tarweb.net.

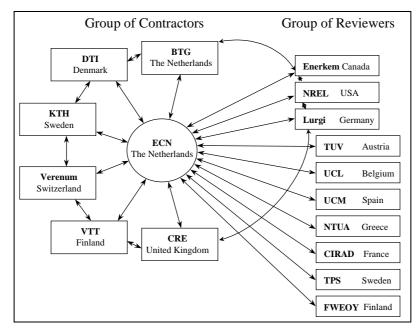
# **1. INTRODUCTION**

The main contaminants in the product gases of biomass gasification are dust and soot particles, organic contaminants (often being referred to as tars), alkali metals, acid

gases and alkaline gases. Measuring techniques for these contaminants allow to determine the functioning of the gas cleaning and to assess the quality of the cleaned gas to be used in the engine or turbine. For most contaminants, well-developed measurement techniques exist which are similar to techniques used for related technologies, such as coal combustion and coal gasification.

For organic contaminants, however, no well-developed measurement technique exists in related technology fields. As some of the organic contaminants are seen as the major problem-causing contaminants in biomass gasification, manufacturers and other workers in this field have used a number of different sampling and analysis methods to determine the level of organic contaminants. In these methods, also the definition of organic contaminants or tars has been rather diverse.

Recently, a project in the European Fifth Framework Programme has started which is aimed at the development of a standard method (Protocol) for the sampling and analysis of organic contaminants (tars) in biomass producer gases. The partners in this project are named in Figure 1. This project has a history dating back to a joint meeting in Brussels (1998) by members of the Gasification Task of the IEA Bioenergy Agreement, US DoE and DGXVII of the European Commission. This meeting led to the development of two draft Protocols for tar measurement, which were presented at the 10<sup>th</sup> European biomass conference in Würzburg, Germany and were recently published [1-3]. The current project is a continuation of this early work.



*Figure 1* Project partners (contractors and reviewers) of the EU project with acronym 'Tar Protocol'

The history, objectives and structure of this EU project were presented in a previous paper [4]. The current paper will outline the contents and principle of the first version of the Protocol, which is now called Guideline for reasons described later.

In the next Chapter 2 the principle of the Guideline will be described. Then, in Chapter 3 attention is paid to choices that were made so far to come to version 1 of the Guideline. Examples of such choices are the name Guideline instead of Protocol, and

the choice for not defining tar whereas Gravimetric tar is defined in the Guideline. In the final Chapter 4 it is shortly described how and on what time scale the development of the Guideline will be completed and which R&D activities are to be performed before completion and field testing of the Guideline.

# 2. PRINCIPLE OF THE GUIDELINE

# **INTRODUCTION**

This chapter contains a concise description of the working principle and the structure of the Guideline for sampling and analysis of tars and particles from biomass producer gases. This description includes the results of many choices, which were made during the development of the Guideline. The rationale behind these choices is not described in this chapter, as this would draw the attention from the working principle and structure of the Guideline. Instead, by using numbers in superscript, reference is made to the next Chapter 3 in which this rationale is explained.

One of the most fundamental questions that rises when discussing a Guideline or Protocol or Standard for tar measurement is "what is it's use?", in other words "is it really needed?". This is the first question answered in the next chapter<sup>1</sup>. The second question will address the word "Guideline" which is now used instead of "Protocol"<sup>2</sup>.

#### **WORKING PRINCIPLE**

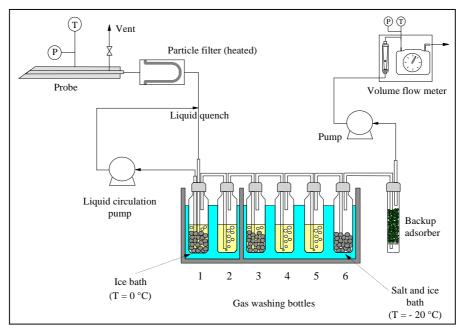
The measurement principle of the Guideline is based on the discontinuous sampling of a gas stream containing particles and condensable organic compounds. The principle is set-up in such a way that also particles can be measured quantitatively<sup>3</sup>. Condensable organic compounds will be further referred to as tars although this word tars is not defined in detail<sup>4</sup>.

The measuring principle has been kept as simple as possible because the measuring conditions can vary from very comfortable laboratory rooms to an executive plant gasifier where there is no customised room for measurements or measurement apparatus. Also the weather conditions can be challenging, for instance in northern Europe measurements might have to be performed at temperatures under  $0^{\circ}$ C.

Sampling is performed under isokinetic conditions<sup>5</sup>. The tar and particle sampling system consists of a heated probe, a heated particle filter, a condenser and a series of impinger bottles containing a tar absorbing solvent. A backup adsorber for volatile organic compounds can optionally be placed after the impinger bottles. The solvent containing bottles are placed in a cold bath so that gradual cooling of the sampled gas from about 0°C to the final temperature - 20°C takes place in them. The sampling train is shown schematically in Figure 2.

The gas sample is led to flow a sufficiently long time through the sampling line and the filter either with the aid of process pressure or a pump included in the sampling line to the gas impingers. A continuous gas flow and heating is necessary in the sampling systems. The flow rate of the sample gas is monitored and controlled during sampling.

The sampling lines including the filter should be heated in order to prevent tar compounds from condensing. However, to avoid thermal decomposition of compounds, the temperature should not be too high. In updraft gasification the temperature should be below 400°C, while in downdraft and fluidised-bed gasification it should be below 700°C. According to experience in practice, 200 - 300°C is suitable gas temperature in the sampling lines.



*Figure 2* Sampling train of the Guideline for sampling and analysis of tars and particles in biomass producer gas

Particles are collected in an externally heated quartz filter<sup>6</sup> which is heated to 250°C or 100°C depending on the gasifier type from which producer gases are sampled<sup>7</sup>. Then, the producer gas is cooled, either on a heat exchanger surface or in an internal circulating liquid quench system in a liquid<sup>8</sup>. Gas quenching by an externally cooled liquid is done immediately after the particle filter and facilitates sampling from producer gas with high tar loadings or with reactive tar species. The condensate is collected in a glass bottle. After the condensor, the gas passes an impinger filled with solvent before further temperature letdown.

In the following series of impingers<sup>9</sup>, tars are absorbed in the solvent<sup>10</sup> at a temperature of -20°C. A backup residual tar adsorber<sup>11</sup> can optionally be used as a safety filter after the impinger train. The volume, temperature, pressure, and gas flow rate through the equipment are measured after the impinger bottles.

Immediately after sampling the contents of the impinger bottles, including the glass beads, are decanted into a storage bottle. All surfaces (including metal surfaces) becoming in contact with the gas at a lower than the process temperature are washed with the solvent, and the washes are combined with the actual sample. This is quite easy to arrange in atmospheric processes, but very difficult in pressurised systems. The storage bottle is stored tightly closed at cool,  $< 5^{\circ}$ C, temperature for later analysis.

The sampling of tars and particles can also be performed separately. For large scale gasifiers where high temperature tar is formed, the generally used sampling strategy is based on separate sampling of particles and gaseous effluents. In case of measuring only tars, isokinetic sampling<sup>5</sup> is only necessary when the temperature of the gas under study at the sampling site is so low that the organic compounds are condensed and form aerosols and/or drops.

# BASIC CONCEPT OF THE SAMPLING TRAIN

The modular sampling train consists of 4 main modules and their submodules. The main modules are gas preconditioning, particle collection, tar collection and volume sampling. The modules are shown in *Figure 3* and the purposes of the modules and needed equipment are aggregated in Table 1.

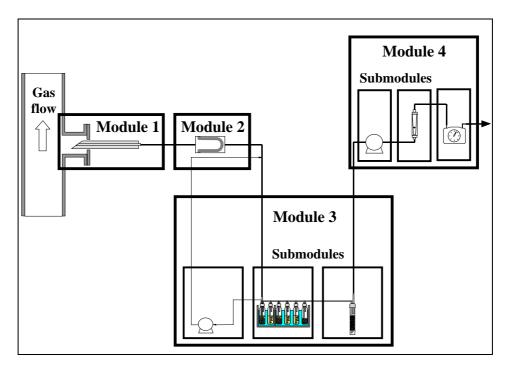


Figure 3 Basic concept of the sampling train

In the preconditioning module (Module 1) the gas is led to cool or is heated from the process temperature to a constant temperature, 250 °C, in a heated probe. In case of pressurised gasification the pressure is diminished to atmospheric pressure. The equipment needed in the module is a sampling probe including a nozzle and necessary valves, at least one shut-off valve and one control valve, and in pressurised gasification also a pressure relief valve. Also the valves are heated to constant temperature.

In the particle collection module (Module 2) a heated thimble filter<sup>6</sup> at the probe temperature collects solids from the gas.

The tar collection module (Module 3) consists of three submodules. In the submodule 3.1, the gas is cooled and moisture and heavy tars are collected in a condenser<sup>8</sup> at the temperature 0°C. In the simplest version, the condenser is an empty impinger bottle in an external ice water bath. More effective versions use either an additional externally cooled heat exchanger before the condenser or an internal liquid quench system. In the submodule 3.2 tars and volatile organic compounds are absorbed into the solvent at -20°C in a series of impinger bottles<sup>9</sup>. The optional submodule 3.3 is a backup adsorber<sup>11</sup>, which collects residual volatile organic compounds that may have penetrated the impinger train. The backup adsorber is not necessary when enough impinger bottles, appropriate solvents<sup>10</sup> and collection temperatures are used. In a later

version of the Guideline, the low temperature impingers may be replaced by an adsorber tower filled with a solid sorbent.

The volume-sampling module (Module 4) consists of three submodules. The purpose of submodule 4.1 is the gas suction or pump (not needed in pressurised gasification). In the submodule 4.2 a gas meter measures the volume of gas flow. Additionally a flow meter, a pressure indicator and a temperature indicator are needed.

 Table 1
 General description of modules and submodules with purpose and equipment used

Module	Purpose	Equipment
Module 1 (Gas preconditioning)		
	Gas cooling, pressure letdown	Nozzle, valves, sampling lines
Module 2 (Particle collection)		
	Collection of solids	Heated filter (high temperature)
Module 3 (tar collection)		
Submodule 3.1	Moisture and tar condensation	Condenser at 0°C
Submodule 3.2	Tar/hydrocarbon collection	Impingers with solvent at -20°C
Submodule 3.3	Backup sampler	Adsorber tower at T <sub>amb</sub> or lower
Module 4 (Volume sampling)		
Submodule 4.1	Gas suction	Pump
Submodule 4.2	Gas volume integration	Gas meter, P and T indicators
Submodule 4.3	Vent/exhaust gas handling	Outdoor ventilation

# **DESIGN OPERATING CONDITIONS**

The sampling train is designed for sample flow rates ranging from  $0, 1 - 0, 6 \text{ m}^3_{\text{n}}/\text{h}$ . The volume of the gas drawn to the tar sample is dependent on the tar content of the gas. A suitable sample volume in (pressurised) fluidised bed gasification (at a tar concentration  $< 20 \text{ g/m}^3_{\text{n}}$ ) is about  $0, 1 \text{ m}^3_{\text{n}}$  and in updraft gasification (tar concentration  $100 - 200 \text{ g/m}^3_{\text{n}}$ )  $0, 05 - 0, 1 \text{ m}^3_{\text{n}}$ . The total content of tar in the solvent should be about  $10 \text{ g/dm}^3$  for gravimetric determination and about  $2 \text{ g/dm}^3$  for gas chromatographic analysis. However, the detection limit is significantly lower for single components (0.25 ppm).

The temperature in the sampling line of the product gas of the updraft gasifier is kept at 100-250°C (to be examined) and in downdraft and fluidised-bed gasification at  $250^{\circ}$ C.

#### SITE SPECIFIC SAMPLING TRAIN SET-UPS

Applying the sampling principle depends additionally on the gasifier type. Most of the gasifiers fall in four categories based on the contact type of the feedstock and on the gasification agent. The four main types are: 1) fixed-bed updraft, 2) fixed-bed downdraft, 3) fluidised-bed and 4) entrained-flow. The yields of tars and particles depend greatly on the gasifier type. The tar concentration in countercurrent (updraft) gasifier is remarkable high (typically  $100 - 200 \text{ g/m}_n^3$ ) comparing to the other gasification applications (tar content usually below  $20 \text{ g/m}_n^3$ ). Also the composition of the tars varies depending on the gasifier type. The particle concentration in the product gas is the largest in the fluidised bed gasifiers.

The amount of particles and amount and composition of tars determine the specific sample train arrangements. Therefore, this arrangement depends on:

- Gasifier type (as described above);
- Gasifier operation condition (specific pre-conditioning steps are foreseen for sampling from pressurised gasifiers or at low temperatures);
- Gasifier scale (sampling at large scale gasifiers at high temperature might be performed non-isokinetically);
- Sample location: before or after gas cleaning (when sampling after the gas cleaning, use of a backup tar adsorber might not be necessary).

The Guideline will give examples of such site-specific sample trains.

## TYPE OF SOLVENT USED

The condensate should preferably be collected in a solvent, not in a cold trap in which there is no "diluting surroundings"<sup>12</sup>. The modular sampling train set-up displayed in this chapter can be used for both water miscible and non-water miscible (non-polar) solvents.

For the water miscible solvents, 1-methoxy-2-propanol has been selected as the most promising candidate. In a later stage, only minor modifications will arise in the Guideline when a non-polar solvent will be used.

The boiling point (evaporation rate) and the water solubility are the main two parameters, which influence the applicability of the liquid quench system in the sampling train set-up.

#### POST-SAMPLING AND ANALYSIS

The Guideline will provide procedures for post-sampling and analysis of the tar samples. Basically, tars are analysed in two ways: gravimetrically<sup>13</sup> and by gas chromatography<sup>14</sup>. The gravimetrical analysis will result in one concentration of gravimetric tars (see note <sup>4</sup> in the next chapter for a definition) whereas the chromatographic analysis will result in concentrations of individual compounds.

At this point the procedures for post-sampling and analysis are not given, as they will to some extent depend on the solvent being chosen. The time frame in which this choice will be made and in which the full Guideline will be available, is given in Chapter 4.

# 3. RATIONALE BEHIND CHOICES MADE DURING DEVELOPMENT OF THE GUIDELINE

During the development of the Guideline many choices were made. Some of these choices are practical choices to move along with the development of the Guideline. Other choices, however, are fundamental ones that were only made after long discussions in the Group of Contractors. These choices and their background discussions are to some extent explained in this chapter. The choices are numbered: the numbers refer to the number in superscript in the previous chapter "principle of the Guideline".

1. Why do we need a Protocol or Guideline or Standard for sampling and analysis of tars? In the past, several tar measurement methods have been developed in the field of biomass gasification. Many of these methods gave and give reliable data on tar concentration, tar being defined as part of the measurement method. Therefore, for tar measurements on single biomass gasification installations the Guideline is not needed and current practice is fine.

Current practice becomes less obvious when tar data are to be compared, for instance when comparing the performance of biomass gasifiers or apparatuses for gas cleaning, or when setting or working with tolerances (maximum allowable concentrations) for tar in gas cleaning devices or prime movers. The value of the comparison will depend on definition of tar and on the ability to measure tar according to the definition. A recent report on parallel measurements with different tar measurement methods at the same location [5] shows that comparison of tar data can be a difficult task.

So the expected value of the Guideline is in the field of comparison. Two types of comparison can be distinguished:

- 1. Comparison of tar concentrations from different locations. To do so, the Guideline has to be used at the two sites.
- 2. Comparison of existing methods for tar measurement. A requisite for reliable comparison of tar concentrations with different measurement methods is that these methods give good results in the ranges of conditions applied (temperatures, pressures, and concentrations and compositions of tars). The Guideline, which will cover large ranges of conditions, can be used to check the applicability of these methods.

The comparison mentioned under 2. will result in knowledge on which method can reliably be used under which conditions. As other methods might be simpler (in terms of man-hours and equipment needed to perform the measurement) compared to the Guideline or produce online results on-site, we have named them "shortcut methods"<sup>15</sup>. Examples of these methods are a) the solid phase adsorption (SPA) method developed by KTH [6], b) a number of solvent-free tar collection systems used by BTG, BEF, IGT [7-9] and c) the FID online tar analysing method under development at the University of Stuttgart [10,11]. Currently it is unknown under which conditions these methods give reliable results, for example it is unknown whether the SPA method can be used for updraft gasifier tars and at which conditions the solvent-free methods fail to collect all tars, for instance as a result of aerosol formation.

In conclusion it can be said that the Guideline will not stop other methods from being used, which is also not the intention of the Guideline as it will be a method that is elaborate in comparison with many existing methods. The Guideline will provide a common method –with which other methods will be compared– that applies for the whole range of measurement conditions.

2. **Protocol versus Guideline**. It was decided to not longer use the word 'Protocol' as it is rather formal and has a broad meaning. Instead, the word 'Guideline' will be used. This word better represents the current status of the method for sampling and analysis than the word 'Protocol'. It should be kept in mind that the Guideline has a temporary status; the aim is to have a Standard. To reach this goal a Standardisation trajectory at CEN will be started during the execution of the EU project.

The full title of the Guideline is 'Guideline for sampling and analysis of tars and particles in biomass producer gases'.

3. **Measurement of tar** *and* **particles**. Among the members of the Group of Contractors in the EU project 'Tar Protocol', it is recognised that the interaction between carbon-rich particles and tar can strongly influence measured tar concentrations. For example, measuring tars in a producer gas with relatively high carbon-rich particle concentrations and relatively low tar concentrations can be a difficult task because of these interactions. In the Guideline, the post-sampling procedures of the particle filter will receive thorough attention in order to produce reproducible results when measuring the concentration of Gravimetric tar (see the next point 4). Likewise, the concentration of particles is also influenced by these post-sampling procedures (e.g. during parallel measurements it was observed that the low particle concentrations in updraft gasifiers are difficult to measure as a result of tar condensation on the particle filter).

Because of the attention paid to sampling and post-sampling procedures for particles, the Guideline will also give a number for particle concentrations and, therefore, is a Guideline for tar *and* particles.

4. **Definition of tars**. The word tar is not defined in the Guideline because a definition (on top of the many definitions that have already been given) will likely cause (further) confusion. The authors of this paper feel that the word tar is an ambiguous term that has no defined meaning and is used in several contexts with at least slightly different meanings. Even the definitions "compounds that condense" or "compounds that contaminate linings or compressors or moving parts in the prime mover" are not unambiguous as condensation or contamination varies with temperature of, and pressure in, the linings/compressors/moving parts as well as with concentration of tar itself.

Besides, the Guideline is not developed for determination of a tar concentration. With the Guideline, two concentrations can be determined:

- The concentration of Gravimetric tar;

- The concentration of individual organic compounds;

**Gravimetric tar** is defined as the evaporation residue at given standard conditions (temperature, pressure, duration). These conditions will be set later when the solvent has been chosen.

In the Guideline, a compound list will be added with **individual organic compounds** including chemical abstract service (CAS) registry numbers. This list is compiled from experimental data on the presence of compounds found in biomass producer gases from updraft, downdraft and fluidised bed gasification.

5. **Isokinetic conditions**. Because particles are sampled and analysed as part of the Guideline, the sampling is performed isokinetically. Instructions of isokinetic sampling of flue gases are given in the standards ISO 9096 or VDI 2066.

Isokinetic sampling with realistic probe dimensions requires, even at ambient pressures, high gas velocities up to 20 m/s with corresponding gas flow rates between

0.1 and 0.6  $m_n^3/h$ . Especially high flow rates are needed in pressurised systems to maintain isokinetic conditions and this is unpractical for impinger sampling using liquids. Therefore, a non-isokinetic sampling arrangement is practical for condensable organic compounds from pressurised systems at temperatures above their condensation point. Non-isokinetic tar sampling is also practical in large-scale atmospheric gasification applications where the pipe diameter is large causing the isokinetic sampling according to the standard to be very difficult and time consuming. The gas temperature above 350°C at the sampling point assures good accuracy in measuring tars also non-isokinetically. In non-isokinetic sampling the alignment of the probe in relation to the gas flow as well as the shape of the probe nozzle can be designed more freely to prevent the nozzle from blocking. This is important especially in pressurised gasification since the probe cannot be removed from the gas line during operation.

- 6. **Particle filters**. Particle filters to be used in the Guideline are thimble filters because of the higher capacity and lower pressure drop of thimble filter compared to flat filters. Only for low particle concentrations (<  $20 \text{ mg/m}^3_n$ ) flat filters will be used as these have a lower weight so that the accuracy of particle weighting increases. The filter material is quartz.
- 7. **Temperatures of particle filters**. The temperature of the particle filter varies with the gasifier type. When sampling high temperature tars (downdraft and fluidised bed gasifiers) the preferable filter temperature is 250°C or higher in order to avoid condensation of tars on carbon rich particles. When sampling low temperature tars (updraft gasifiers) the filter temperature is 100°C to avoid polymerisation of the tars. These are draft temperatures to be evaluated.
- 8. Cooling and/or quenching of the producer gas before the impinger. After the particle filter, the gas must be cooled to condense the moisture and collect the tar in the impingers. Cooling can be done either by using conventional heat exchangers or by using a liquid quench system. In both cases, the condensate is collected in a condenser bottle. A liquid quench system is optional but facilitates post-sampling cleaning procedures because sampling lines are kept clean and polymerisation of reactive tar species is minimised.

The use of a liquid quench strongly depends on the tar impingement solvent chosen. With a water miscible solvent exhibiting a medium volatility (like methoxypropanol) a liquid quench can be used using the same solvent. With water miscible solvents exhibiting a high volatility (like acetone) a liquid quench with the solvent cannot be used. Instead conventional cooling (with heat exchangers) or a liquid quench with water can be used. With non-polar solvents (like iso-octane) a liquid quench with the solvent cannot be used. With non-polar solvents (like iso-octane) a liquid quench with the solvent cannot be used as it would lead to two-phase liquids or even formation of emulsions. Instead conventional cooling (with heat exchangers) or a liquid quench with water can be used.

When a liquid quench with a water miscible solvent is used, obstruction of the gas flow through ice formation can be safely avoided.

- 9. **Impinger bottles**. One of contractors has very good experience with custom-made impinger bottles. During the development of the Guideline, the performance of standard impinger bottles (which are commonly available) will be compared with the use of modified impinger bottles.
- 10. **Solvent**. At the time of writing of this paper, a final choice for a solvent has not been made. In the Group of Contractors it is clear that dichloromethane (DCM) cannot longer be used for various reasons (toxicity, volatility, safety). During the first year of the development of the Guideline, a number of alternative solvents will be evaluated. Criteria for this evaluation are set (see below in Chapter 4). The

choice of the solvent will determine the sampling temperature. A temperature of  $-20^{\circ}$ C is used as a start.

- 11. **Backup adsorber**. First tests with tar measurement following the Guideline will be performed with a backup adsorber as a safety device and to learn which volatile organic compounds and/or tar compounds pass the impinger bottles. The backup adsorbed is currently an optional submodule in the Guideline. In the parallel R&D program (see Chapter 4) a number of potential adsorbing materials will be evaluated, such as activated carbon, XAD, and SPA material. In a later version of the Guideline, the solvent impingers can possibly be replaced by an adsorber tower filled with an appropriate sorbent.
- 12. **Solvent or no solvent**. For tar sampling from updraft gasifier producer gases, a solvent is needed because direct condensation of the tars without diluting media can result in further reactions (polymerisation). The reactivity is suppressed by using a diluent like an absorbing liquid or an adsorbent resin.

For tars sampled from downdraft and fluidised bed gasifiers, it has to be shown whether "no solvent" can be as effective as sampling with a solvent. For the first version of the Guideline, the Group of Contractors has chosen to use a solvent. This choice will be evaluated during the running EU project.

- 13. **Gravimetric analysis**. The weight of tar is determined gravimetrically by means of solvent distillation and evaporation according to a defined procedure (temperature, pressure and duration of distillation and evaporation). The resulting number is the amount of Gravimetric tar.
- 14. **Compound analysis by GC**. Individual compounds are determined by gas chromatography (GC). GC in combination with a Mass-Spectrometer (MS) is preferred, GC in combination with a Flame Ionisation Detector (FID) with internal standard calibration is an alternative.
- 15. **Shortcut methods** are not described in the Guideline. However, reference is made to a web page where these methods will be described and where reference to open literature is made.

#### 4. FUTHER DEVELOPMENT OF THE GUIDELINE

The Guideline is being developed in a two year EU project in which also two partners from North America participate. The participants were named in Figure 1.

The time frame of further development of the Guideline and the anticipated R&D work are described in this chapter. More information, including the inventory of required R&D and the most recent version of the Guideline, can be found on a web page (www.tarweb.net).

# TIME FRAME OF FURTHER DEVELOPMENT

Figure 4 gives a rough time frame of the main activities performed, and results obtained, in this project. By the time this paper appears, a first version of the Guideline will be ready. This version will be updated by the results of critical and short-term R&D needs, amongst which is the selection of the solvent. The resulting second version of the Guideline will be reviewed. The input from the Reviewers will be used together with the results of ongoing R&D activities (for example towards the interaction between particles and tar) to prepare a final version of the Guideline. Early

2002 a workshop will then be organised on tar sampling and analysis in general and the new Guideline in particular.

# **R&D ACTIVITIES TO BE PERFORMED**

Because the EU project "Tar Protocol" is a Concerted Action project, no R&D will be performed in the project. However, it is recognised that some technical details have to be found out before the Guideline will perform satisfactory. Therefore, included in the project is the co-ordination of R&D projects executed under national funds.

One of the major R&D questions to be answered is "which is the preferable solvent to be used in the Guideline?" as DCM is no longer an acceptable option.

The Group of contractors has proposed a number of candidate solvents: methanol, isobutyl methyl ketone and 1-methoxy-2-propanol. In relatively short R&D projects these candidates will be evaluated using a number of selection criteria that were proposed by the group of contractors. These selection criteria are:

# Selection criteria by exclusion

- Solvent is not toxic or carcinogenic
- Solvent is non halogenated
- Solvent or impurities in solvent are not present in relevant concentrations in the tar matrix
- No additional solvent is needed for equipment cleaning

#### Quantitative solvent selection criteria

- tar solubility (e.g. oxygenated organics, PAH, Gravimetric tar)
- Collection efficiency for individual compounds
- Suitability for standard analytical procedures
- Evaporation rate at 0°C and -25°C
- Ice formation tendency
- Freezing point of solvent
- Occupational exposure risk factor (e.g. as 10hr TWA/vapor press.)
- Time requirement for sampling and post-sampling procedures
- Availability / cost

Other R&D questions that are recognised from within the project "Tar Protocol" are amongst others the effectiveness of tar adsorbents, whether tar polymerisation occurs at the proposed sampling and post-sampling conditions, and the interaction between particles and tar during sampling and post-sampling. The complete R&D inventory can be found on the web site (www.tarweb.net). Any institution or company having knowledge, performing R&D or planning to perform R&D is invited to contact the authors of this paper to exchange knowledge and/or to co-ordinate or perform R&D activities.

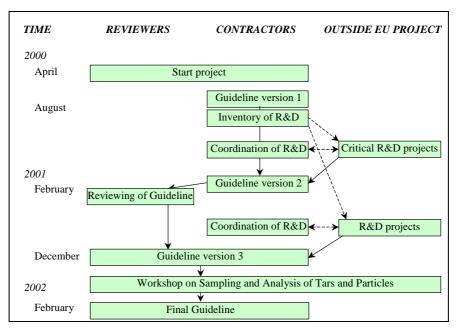


Figure 4 Time frame of main activities of the EU project 'Tar Protocol'

#### **5. REFERENCES**

- 1. K.Maniatis and A.A.C.M.Beenackers: *Introduction. Tar Protocols. The IEA Bioenergy gasification task.* Biomass and Bioenergy **18** (1) 1-4 (2000).
- 2. N.Abatzoglou, N.Barker, P.Hasler and H.Knoef: *The development of a draft protocol for the sampling and analysis of particulate and organic contaminants in the gas from small biomass gasifiers*. Biomass and Bioenergy **18** (1) 5-17 (2000).
- P.Simell, P.Stahlberg, E.Kurkela, J.Albrecht, S.Deutsch and K.Sjöström: *Provisional protocol for the sampling and anlaysis of tar and particulates in the gas from large-scale biomass gasifiers. Version 1998.* Biomass and Bioenergy 18 (1) 19-38 (2000).
- 4. J.P.A.Neeft, H.A.M.Knoef, U.Zielke, K.Sjöström, P.Hasler, P.A.Simell, M.A.Dorrington and C.Greil: *Tar protocol. Development of a standard method for the measurement of organic contaminants (tar) in biomass producer gases.* In: Biomass for energy and industry. Proc. 1st World conference and exhibition, held in Sevilla (Spain), 5-9 June 2000 (Eds. S.Kyritsis et al.), EnergiaTA, Florence (Italy) (2000).
- U.Zielke: Tar and solid particles in LCV-gas from fixed bed gasifiers. Danish measurement results. In: IC engines for LCV gas from biomass gasifiers. Proc. IEA Thermal gasification seminar, Zürich, October 28, 1997 (Ed. R.Bühler), pp. 31-38, Unwelt + Energie, Maschwanden (Switzerland) (1998).
- 6. C.Brage, Qizhuang Yu, Guanxing Chen and K.Sjöström: Use of amino phase adsorbent for biomass tar sampling and separation. Fuel **76** (2) 137-142 (1997).

- 7. H.A.M.Knoef: The UNDP/World Bank monitoring program on small scale biomass gasifiers (BTG's experience on tar measurements). Biomass and Bioenergy 18 (1) 39-54 (2000).
- 8. A.Das: Contaminant testing method for biomass gasifier engine systems, Golden (USA), Colorado School of Mines (1985).
- 9. R.A.Knight: *Experience with raw gas analysis from pressurized gasification of biomass*. Biomass and Bioenergy **18** (1) 67-77 (2000).
- 10. P.Hasler and T.Nussbaumer: *Kontinuierliche Teermessung mittels FID bei einem Holzvergaser*, Zürich (Switerland), Verenum, 63 p. (2000).
- 11. O.Moersch, H.Spliethoff and K.R.G.Hein: *Tar quantification with a new online analyzing method*. Biomass and Bioenergy **18** (1) 79-86 (2000).

# 6. ACKNOWLEDGEMENTS

The European Commission, The Netherlands Agency for Energy and the Environment (NOVEM) the Swiss Federal Office of Education and Science, the US Department of Energy and National Resources Canada are greatly acknowledged for their (financial) contribution to the work presented in this paper.