# **Rationale for setup of impinger train**

# as used in the Technical Specification of Sampling and Analysis of Tar and Particles in the Product Gases of Biomass Gasification.

# **Technical background document**

Prepared under

CEN BT/TF 143 "Organic contaminants ("tar") in biomass producer gases"

by J.P.A. Neeft, SenterNovem (The Netherlands), convenor of CEN BT/TF 143

January 2005



# Table of Contents

1.	Introduction	3
2.	Main results on R&D towards the efficiency of tar collection	4
2.1.	Available data	4
2.2.	Results of ECN	4
2.3.	Results of VTT	6
3.	Arguments used in the discussion on optimised sampling conditions	8
3.1.	General arguments	8
3.2.	Specific arguments	8
4.	Final conditions of Technical Specification	11
5.	References	12

## 1. Introduction

This document is a background document to the development of a method for sampling and analysis of tar and particles in the product gases of biomass gasification. This method was developed in two subsequent EU projects under the EU fifth framework programme. The first project was aimed at the development of the "Tar Guideline", the second project was aimed at standardisation of this Guideline into a CEN Technical Specification. The second project was performed under the umbrella of a Technical Committee under CEN, called CEN/TC BT/TF 143 "Measurement of organic contaminants (tar) in biomass producer gases".

In the method, the tars are sampled from biomass gasification product gases by impinger bottles containing isopropanol. The sum of the six individual impinger bottles is referred to as the "impinger train". During the development of the Guideline and the Technical Standard, the actual conditions of the impinger train proved to be of large importance for the efficiency of collection of tars. In the project group, long discussions were held in order to optimise these conditions. In a meeting in Rome, June 2004, the final conditions were agreed upon. This document describes the rationale behind the choice for these final conditions.

This report focuses on the temperature of the impinger bottles and the use of glass frits and glass beads. Other topics that were researched as part of the development of a method for sampling and analyses of tars, are described in an R&D report that was made at the end of the first EU project [Brage, 2002]. In this 2002 report, for instance, the rationale for the choice of isopropanol as a solvent is given. As the discussion on the collection efficiency has continued after issuing the 2002 report, this part is updated by the current document.

In the current document, the following topics are covered:

- The R&D that has been performed on the efficiency of collection of tars in the impinger train is presented in Chapter 2;
- The arguments that were used in discussions on the optimised sampling conditions are given in Chapter 3;
- The final conditions of the Technical specification are given in Chapter 4;
- Finally, chapter 5 gives reference to the full R&D reports.

## 2.1. Available data

To the author's knowledge, so far only two institutions have performed R&D work on the efficiency of tar collection in which the collection efficiency of tar compounds was quantitatively determined. The results of this R&D work by ECN and VTT will be described in this chapter. Other institutes, like BTG, DTI, TU-Graz, Umsicht and Verenum, have used the Guideline or the draft Technical Specification<sup>1</sup> over the last years. They did not, however, check the concentrations of tars after the impinger train to determine the collection efficiency. BTG did report on the collection efficiency [Gansekoele, 2002], however, the collection efficiency was determined qualitatively by the colour of a back-up filter. Therefore, these BTG results will not be considered here.

The discussion in this chapter is focused on the efficiency of tar collection by the impinger train. Other R&D topics are not covered. For instance, BTG and ECN have performed a joint R&D programme in which R&D topics like the temperature of the particle filter, the solvent in the impinger bottles and the method for analysis of gravimetric tars were evaluated. The overall report [Gansekoele, 2002] contains R&D results on these topics, which are summarised in [Brage, 2002].

### 2.2. Results of ECN

The results given in this paragraph are copied from [Neeft, 2001], unless otherwise stated. At ECN, in 2001 three series of tests with the Guideline were performed:

Samples taken:	Gasifier conditions	Aim of sampling
March 16, 2001	CFB, 850°C	Check methoxypropanol as a solvent, compare Guideline with SPA
July 20, 2001	BFB, 825°C	Compare ethanol and isopropanol as solvents, compare Guideline with SPA
September 26, 2001	CFB, 880°C	Parallel testing BTG and ECN, compare Guideline and SPA

The March 2001 samples will not be considered as their main aim was to compare the Guideline with the SPA method. The September 2001 samples are only considered qualitatively as the collection efficiency in these tests was not determined (SPA samples were only taken upstream from the impinger train). Results from the July tests are described in detail. In the July and September tests two types of frits were used: a standard G1 frit and a denser G3 frit. Conditions in the impinger bottles and the type of frits are shown in the table below.

		Liquio	l Solvent	Impingers (temperature, type of frit)					
		Quenc	h	1	2	3	4	5	6
Date	Test			(150 ml)	(75 ml)	(75 ml)	(75 ml)	(75 ml)	Empty
July 2001	1	Yes	Isopropanol	0°C, no frit	0°C, G1	-20°C, G1	-20°C, G3	-20°C, G1	-20°C, G3
July 2001	2	Yes	Isopropanol	40°C, no frit	40°C, G1	-20°C, G3	-20°C, G3	-20°C, G1	-20°C, G3
July 2001	3	Yes	Isopropanol	40°C, no frit	40°C, G1	-20°C, G3	40°C, G3	-20°C, G1	-20°C, G3
July 2001	4	Yes	Ethanol	40°C, no frit	40°C, G1	-20°C, G3	40°C, G3	-20°C, G1	-20°C, G3
July 2001	5	No	Ethanol	40°C, no frit	40°C, G1	-20°C, G3	40°C, G3	-20°C, G1	-20°C, G3
September 2001	1	Yes	Isopropanol	40°C, G1	40°C, G3	-20°C, G1	-20°C, G3	-20°C, G3	-20°C, G3
September 2001	2	Yes	Isopropanol	40°C, G1	-20°C, G3	40°C, G1	-20°C, G3	-20°C, G3	-20°C, G3

<sup>&</sup>lt;sup>1</sup>: During the first EU project (2000-2002) the method for sampling and analysis of particles and tars from biomass producer gases was named the "Tar Guideline". During the start of the second project (2002-2005) the name was changed into "draft Tar Standard" which was changed again into "draft Technical Specification (on sampling and analysis of particles and tars)" once it became clear that the CEN standardisation will result in a CEN Technical Specification (CEN TS) and, for the moment being, not in a CEN standard. This CEN TS will later (after 1 or 2 years) be changed into a CEN EN Standard. All these names refer to the same method for sampling and analysis of tars. Of course, the method was improved by small changes over the years 2000 – 2004. At the end of 2004, the final method was established.

In these tests, the sample flow rate was set to 150 l/h, sampling was performed for about 30 min. Downstream of impinger 6, a  $\mu$ m-filter was placed in all five tests. The pressure drop over the sampling train incl.  $\mu$ m-filter was about 50-100 mbar. During the first test, the pressure difference increased to about 400 mbar. In all other tests, the pressure difference rose slowly to 150-200 mbar pressure difference. Before and after the sampling train (and after the  $\mu$ m-filter), tars were also sampled with the SPA method. SPA is an abbreviation for "Solid Phase Adsorption", the method was developed by KTH, details can be found in [Brage, 1997].

The last test (September 2001, test 2) was performed for only 24 minutes and was then stopped because of a problem with the gasifier (fuel feed was temporarily stopped). During the last four minutes of this test the tar concentration was probably strongly reduced due to an increased air-to-fuel ratio.

During sampling, some qualitative observations were made:

#### Mist and/or tar aerosols are visible during sampling

1. A thick white mist appears in the first impinger trains. The first impinger with a G3 frit catches most of the mist, however, a close look learns that a fraction of the mist escapes from the impinger and is only removed in subsequent impingers.

#### Solvent in impingers and filters after impingers obtain a yellow to orange colour

- 2. The solvent colours yellow to orange-brown. The colour of the first impinger bottle seems to be more orange/brown than the colour of the 4 subsequent impingers filled with liquid which also have a lighter colour.
- 3. The µm-filters after the sampling train (samples July) get a similar yellow colour. Also BTG observed this yellow colour in a backup-filter after their sampling train during the September tests. This yellow colour is, in the eyes of ECN, a good indicator of the performance of the sampling train. When the filter after the sampling train colours yellow, then also the frit in the last (empty) impinger bottle turns yellow. Apparently, still a small part of the aerosols pass the sampling train, which will further be denoted to as "aerosol slip".

#### A temperature gradient in the impinger train reduces the "aerosol slip"

4. It was observed that when the temperature of the first impinger is increased (from 0°C to 40°C) or when two temperature gradients are used (in tests 3, 4, and 5 in July and in test 2 in September), then the aerosol slip is decreased. This is most obviously visible from the colours of the five µm-filters from the July tests, see Figure 1. This shows that a temperature increase from 0 to 40°C of the first impingers results in a lighter yellow colour of the µm filter. The filter remains colourless when also impinger bottle 4 is kept at 40°C so that a second temperature gradient is introduced. In the September tests the two filters stayed colourless independent of the use of one or two temperature gradients. The colour of the isopropanol in impinger 5 was, however, very lightly yellow in test 1 which had only one temperature gradient from +40 to -20°C whereas this colour was almost transparent (very, very lightly yellow) in test 2 with two such temperature gradients. The µm-filters in the September tests.



Figure 1 The five µm-filters for tests 1 to 5 performed July 20, 2001. For black-white prints: the filters 1 and 2 have a clear yellow colour whereas filters 3, 4 and 5 are white.

The collection efficiency in the impinger trains was calculated from the SPA samples before and after the impinger trains: collection efficiency = 100%\*(1 - [SPA,after]/[SPA,before]). These data were not published before (*i.e.* they are not part of the [Neeft, 2001] report). For all compounds reported except for the lightest compounds (benzene, toluene, xylenes) the concentrations as measured by SPA correspond very well to the

concentrations measured by the Guideline. The collection efficiency for the July tests are shown in the table below.

	Removal efficiency in July tests				
Compound	Test 1	Test 2	Test 3	Test 4	Test 5
Benzene	75,5%	95,6%	97,1%	98,9%	99,1%
Pyridine	100,0%	100,0%	100,0%	100,0%	100,0%
Toluene	89,8%	93,5%	94,8%	98,0%	97,9%
m/p-Xylene	100,0%	100,0%	100,0%	100,0%	100,0%
o-Xylene + Styrene	100,0%	100,0%	100,0%	100,0%	100,0%
Phenol	99,0%	99,2%	99,5%	97,1%	100,0%
Indene	100,0%	100,0%	99,8%	100,0%	100,0%
Naphthalene	100,0%	100,0%	100,0%	100,0%	100,0%
2-methylnaphthalene	100,0%	100,0%	100,0%	100,0%	100,0%
1-methylnaphthalene	100,0%	100,0%	100,0%	100,0%	100,0%
Biphenyl	100,0%	100,0%	100,0%	100,0%	100,0%
Ethenylnaphthalene	100,0%	100,0%	100,0%	100,0%	100,0%
Acenaphthylene	100,0%	100,0%	100,0%	100,0%	100,0%
Fluorene	100,0%	100,0%	100,0%	100,0%	100,0%
Phenanthrene	100,0%	100,0%	100,0%	100,0%	100,0%
Anthracene	100,0%	100,0%	100,0%	100,0%	100,0%
Fluoranthene	100,0%	100,0%	100,0%	100,0%	100,0%
Pyrene	100,0%	100,0%	100,0%	100,0%	100,0%
Chrysene	100,0%	100,0%	100,0%	100,0%	100,0%
Benzo(b)fluoranthene	100,0%	100,0%	100,0%	100,0%	100,0%
Benzo(k)fluoranthene	100,0%	100,0%	100,0%	100,0%	100,0%
Benzo(a)pyrene	100,0%	100,0%	100,0%	100,0%	100,0%
Perylene	100,0%	100,0%	100,0%	100,0%	100,0%
Total GC tars (excl. Benzene)	99,3%	99,4%	99,6%	99,7%	99,8%
Tars from acenaphthylene to pyrene	100,0%	100,0%	100,0%	100,0%	100,0%

### 2.3. Results of VTT

VTT has performed several tests after bubbling and circulating fluidised bed gasifiers. A number of different solvents were used in the impinger trains (DCM, ethanol, isopropanol, methoxypropanol) as part of the work was aimed at choosing the solvent and optimising the impinger train design. In this report, only the isopropanol results of VTT will be reported. In the VTT tests with isopropanol, the temperature of the impinger bottles was varied (see table beneath). In order to determine the collection efficiency, a second impinger train was added. VTT did not use glass frits, but used glass beads (outer diameter 6 mm) in impinger bottles 1, 5 and 6.

More details on these tests can be found in the Appendix.

The results are shown in the two tables below. Tests were performed at three temperature levels of the impingers, for each level two tests were performed and an average removal efficiency was calculated. The removal efficiency was calculated as the percentage of the tar found in the first sampling train, compared to the total amount of tar found (first and second sampling trains). For the 6 tests, the removal efficiency is shown in the first table:

Sample	Cooling	Removal efficiency			
		Total GC tars	Average	Tars from acenaphtylene to pyrene	Average
		%	%	%	%
1	4 x +40°C / 2 x –20°C	99	100	95	07
2	Idem	100	100	98	97
3	4 x +20°C / 2 x –20°C	98	00	87	02
4	Idem	100	99	96	92
5	2 x +20°C / 4 x –20°C	97	00	82	ог
6	Idem	98	98	87	ŏ5

Compounds specific removal efficiencies for tests 1, 2, 5 and 6 are listed in the following table:

	Compound	l specific remo	val efficiency i	n VTT tests
Compound	Test 1	Test 2	Test 5	Test 6
Benzene	99,4 %	100 %	100 %	100 %
Toluene	100,0 %	100 %	100 %	100 %
Ethynylbenzene	100,0 %	100 %	100 %	100 %
Styrene	100,0 %	100 %	100 %	100 %
4-Methylstyrene	100,0 %	100 %	100 %	100 %
Indene	100,0 %	100 %	100 %	100 %
Naphthalene	100,0 %	100 %	100 %	100 %
2-Methylnaphthalene	100,0 %	100 %	100 %	100 %
1-Methylnaphthalene	100,0 %	100 %	100 %	100 %
Biphenyl	100,0 %	100 %	100 %	100 %
2-Ethylnaphthalene	100,0 %	100 %	100 %	100 %
Acenaphthylene	100,0 %	100 %	100 %	100 %
Acenaphthene	100,0 %	100 %	100 %	100 %
Fluorene	100,0 %	100 %	92 %	<b>94</b> %
Phenanthrene	100,0 %	100 %	73 %	77 %
Anthracene	100,0 %	100 %	<b>70</b> %	74 %
4H-Cyclopenta[def]phenanthrene	100,0 %	100 %	65 %	71 %
Fluoranthene	100,0 %	100 %	<b>70</b> %	77 %
Benz[e]acenaphthylene	100,0 %	100 %	67 %	71 %
Pyrene	72,7 %	86 %	63 %	72 %
Total GC tars (excl. Benzene)	99,1 %	99,6 %	96,8 %	97,8 %
Tars from acenaphthylene to pyrene	95,9 %	97,7 %	82,4 %	86,8 %

# 3. Arguments used in the discussion on optimised sampling conditions

### 3.1. General arguments

The number of parameters, that have an influence on the collection efficiency of the impinger train, is large. At least the following parameters were discussed:

- Solvent in the impinger bottles;
- Temperatures of the impinger bottles;
- Flow rate of the producer gas through the impinger bottles;
- Actual design of the impinger bottles
- (specifically the height and the diameter of the bottle and the standpipe);
- Presence of glass frits or glass beads in the impinger bottles;
- Pressure drop over the sampling train as a result of the presence of glass frits.
- Safety of the use of the method "on-site" (industrial plant facility, north-European weather conditions)
- Actual composition of the tar

(optimised conditions could be different for low temperature tar compared to high temperature tar);

It was acknowledged that much R&D work could be performed in order to optimise these parameters towards high collection efficiency of the tars. It was decided to follow a practical approach, basically because of the following two arguments:

- 1. Limited time and budget to perform R&D
- 2. Focus on the development of a practical method, which should be **<u>good enough</u>**, and not necessarily be the **<u>best one possible</u>**.

Therefore, it was decided to use the information that was available and to choose for the best method **on the basis of this information**. This method should be good enough to give satisfactory repeatability and reproducibility date in Round Robin analysis and parallel tests.<sup>2</sup>

### 3.2. Specific arguments

The following specific arguments led to the set-up in the current Technical Specification on sampling and analysis of particles and tars in biomass producer gases:

#### **3.2.1.** Poor tar sampling is (partly) a result of formation of tar aerosols

A number of observations led to the conclusion that the formation of aerosols is at least partly responsible for the fact that a collection efficiency of 100% is difficult to reach with the impinger train. These observations are:

- 1. At the entrance of the impinger train, almost in all tests a white fog can be seen. This white fog disappears in downstream impinger bottles (observation 1 of paragraph 2.2).
- 2. In some tests, both glass frits in the empty 6<sup>th</sup> impinger bottle and backup µm-filters coloured yellow. This colour is ascribed to the collection of yellow coloured tar aerosols.
- 3. When analysing the tar content in the 6 individual impinger bottles (in a test without frits), tars were found in all bottles and the tar concentrations in impingers 2 6 were more or less constant (figure 3 in [Brage 2002]). This observation was ascribed to aerosols passing the impingers [Brage, 2002].

It was tried to analyse the tar compounds in the yellow deposit on the backup  $\mu$ m-filters. No tar compounds could be detected by GC-FID or GC-MS. Apparently, the deposit contains other (high molecular) compounds. The same observations had earlier been made for the extracts of the 325°C particle filter, which was yellow but which did not or hardly contain GC-FID or GC-MS detectable tar compounds (see [Neeft, 2001]). After discussion, the members of the CEN BT/TF 143 still believe that the yellow deposit is likely to be tar as the

<sup>&</sup>lt;sup>2</sup>: As sampling is part of the method, it is not possible to obtain full data on repeatability and reproducibility by sending round samples for Round Robin analysis. Apart from Round Robin analysis on the analysis part of the method, also two parallel tests were performed. In a parallel test, several institutions are present at a running gasifier to sample the product gases simultaneously in order to obtain data on repeatability and reproducibility of the overall method.

colour is the colour of high temperature biomass gasification tar and the colour of the isopropanol after collection of tars.

#### 3.2.2. A temperature of 40°C in the first impingers gives a better performance than 20°C

The results of VTT tests 1-4 indicate that the method performs better when the temperature of the first impinger bottles is 40°C instead of 20°C. A plausible explanation is that the dissolution of tars at higher temperature is faster than at lower temperature. A second possible advantage of a higher temperature is that at a higher temperature more isopropanol will evaporate, leading to a higher concentration of aerosols and/or to larger aerosol droplets when the gas is cooled to -20°C and, therefore, to a more efficient collection of the tars which are part of the aerosols.

A temperature of 40°C is the highest temperature possible for sampling with isopropanol, as at higher temperatures the evaporation of isopropanol is that high that the isopropanol might be totally evaporated before the sampling is completed, in particular when sampling times have to be long due to low tar concentrations. At long sampling times (longer than one hour), it is recommended to have a first impinger bottle of 250 ml with 150 ml of solvent in order to avoid evaporation of all solvent during sampling.

#### **3.2.3.** Fine glass frits improve the efficiency of sampling of tar aerosols

When comparing the results of the VTT tests with the ECN tests, it can be concluded that the collection efficiency of the ECN tests is higher (100% for tars from acenaphthylene to pyrene) compared to the VTT tests (up to 97% for tars from acenaphthylene to pyrene). The major difference between the ECN and the VTT set-up was the use of glass beads in the VTT set-up and the use of glass frits in the ECN set-up.

After discussion, the hypothesis is that G3 frits capture part of the aerosols and, therefore, increase the collection efficiency. This hypothesis is supported by the visual observations when performing a test with and without a G3 frit. Without a frit a white fog is witnessed in three, four or even five impinger bottles whereas a G3 frit in the second impinger bottle removed all or virtually all visible droplets [Brage, 2002]. Visually, there is a large difference between G1 frits / glass beads and G3 frits. The G1 frits and the glass beads cause relatively large gas bubbles, the frits / beads might therefore not be capable of removing small aerosols. They do, however, have a function in the tar collection as the gas bubbles in the impingers are smaller than bubbles without frit or glass beads. The smaller bubbles enhance the dissolution of tars in the isopropanol (they increase the mass transfer rate by decreasing the transfer distance). It was concluded, therefore, that G3 frits are preferred.

The above hypothesis was also supported by the more detailed analysis on a compound basis. In the ECN tests, the only compounds that were not collected with an efficiency of 100% were the small compounds (benzene, toluene, xylene, phenol and in one test indene). In the VTT test, on the contrary, the smaller compounds were collected quantitatively (on behalf of one number for benzene of 99,4 %) whereas the higher tar compounds were not collected quantitatively. This indicates that in the VTT test, slip of aerosols does occur whereas the dissolution of smaller compounds is somewhat more efficient which is possibly due to the four impinger bottles at +40°C in comparison with three such bottles in the ECN tests. The most logical explanation that was found in the discussions in the CEN BT/TF 143, was that G3 frits enhance the capturing of aerosols.

#### 3.2.4. A second temperature drop (+40 to -20°C) improves the efficiency of sampling of aerosols

It was also concluded to be likely that the temperature gradient (from +20 or +40°C to -20°C) enhances the formation of aerosols and, therefore, the capturing of tars in combination with G3 frits. The rationale for this is that at 40°C isopropanol evaporates and the gas becomes saturated or almost saturated with isopropanol. When the temperature of the gas is decreased to -20°C, the isopropanol vapour will partly condense forming more or larger aerosols compared to the amount or size of aerosols in a gas that stays at one temperature.

In ECN tests 3, 4 and 5 a second temperature gradient was introduced (*i.e.* impingers 1 and 2 at +40°C, impinger 3 at -20°C, impinger 4 at +40°C and impingers 5 and 6 at -20°C). In these tests the µm backup filters did not colour yellow whereas they did in the tests with only one temperature gradient. Although it could not be shown that this yellow colour is indeed caused by tar aerosols (tar compounds could not be detected in the yellow deposit), is seems likely that the yellow colour is caused by high-molecular tar compounds and that the yellow colour, therefore, is a marker for the presence of tar aerosols after the impinger train.

The final conclusion was that G3 frits are preferred, in combination with a temperature of +40°C and two temperature gradients from +40°C to -20°C. As the G3 frits remove the aerosols after a temperature decrease (including the one after the particle filter to the impinger train), and as the first impinger should preferably not contain a frit due to former experiences with blockage of this impinger, it was decided to place G3 frits in

impinger bottles 2, 3 and 5. Besides, a G3 frit is placed in impinger bottle 6 as an indicator for passing aerosols (when it turns yellow, this means that some aerosols pass the impinger train).

#### **3.2.5.** Fine glass frits cause a risk of high pressure drops over the sampling train

Using fine G3 frits introduces the risk that the pressure drop over the sampling train increases during the tar sampling. The pressure drop can become that high that a good tar sampling is no longer guaranteed. When this happens, it is advised to use courser G2 frits. So far (among others: ECN tests reported here, parallel tests in Oberhausen in November 2004), too high pressure drops have not occurred.

At some sampling locations, the risk of higher pressure drops over the sampling trains should be avoided at any time. This is for instance the case when tars are to be sampled at a commercial gasifier where the risk of a gas leakage should be kept to an absolute minimum. For these circumstances, an alternative set-up of the impinger train is given in the Technical Specification. This set-up is similar to the set-up that VTT did use in the tests that are described in this document: 4 impinger bottles at  $+40^{\circ}$ C, two impinger bottles at  $-20^{\circ}$ C, no frits but glass beads with outer diameter of 6 mm in impinger bottles 1, 5 and 6. When using this alternative set-up, the user should check the collection efficiency of the impinger train as it can be low for the high molecular tar compounds, especially when compounds with a larger molecular weight than pyrene will be reported (see VTT results).

#### **3.2.6.** A liquid quench does not seem to have an effect on collection efficiency

In older versions of the Tar Guideline, a liquid quench was part of the set-up. The rationale behind this liquid quench was that the introduction of the solvent isopropanol directly after the hot particle filter would lead to an improved collection efficiency due to:

- (a) dissolving tars into the isopropanol already before the impinger train; and
- (b) evaporation of isopropanol at increased temperatures directly after the particle filter, resulting in more aerosols in the first impinger bottles and a better collection of tars.

As a better collection efficiency was not found (e.g. compare ECN July 2001 tests 4 and 5), it was decided that a liquid quench is not a required part of the method. A liquid quench can be installed, it still has the advantage that the tubes from the particle filter up to and including the standpipe of the first impinger are washed with isopropanol, avoiding the deposition of tars. Without a liquid quench, washing of these tubings and of the first impinger after the sampling takes more time.

# 4. Final conditions of Technical Specification

The arguments that were given in the previous chapter have led to the next two requirements for the impinger train in the Technical Specification:

- 1. Tar collection shall be performed with 6 impinger bottles of which the first acts as a condenser for water. Standard impinger bottles (NS 29/32) of 100 or 250 ml can be used. Use impinger bottles of 250 ml for gas flow rates >  $0,3 \text{ m}^3_{n}/h$ . Impinger bottles 1, 2, 3, 4 and 5 contain approximately 50 ml of solvent, impinger bottle 6 is empty. With high moist gas, a large amount of condensate will be generated thus requiring a large condenser.
- 2. One of the following two setups for the 6 impinger bottles shall be chosen. The alternative setup shall only be used when the pressure drop over the impinger bottles is too high or when, for safety reasons, it may never become high. When using the alternative setup, the user shall verify the collection efficiency over the impinger bottles.

#### Standard setup

- Impinger bottles 2, 3, 5 and 6 contain glass frits. These frits are placed either as a filter at the end of the inner tube, or are placed around the inner tube covering the rest of the diameter of the impinger bottle. G3 frits shall be used, if this results in a too high pressure drop over module 3, G2 frits shall be used.
- The temperature of impinger bottles 1, 2, and 4 shall be between 35 and 40°C, the temperature of impinger bottles 3, 5 and 6 shall be between -15 and -20°C.

#### **Alternative setup**

- Impinger bottles 1, 5, and 6 contain glass beads. The outside diameter of the glass bead is 6 mm.
- The temperature of impinger bottles 1, 2, 3 and 4 shall be between 35 and 40°C, the temperature of impinger bottles 5 and 6 shall be between -15 and -20°C.



The next figure gives a schematic representation of the impinger train in the Standard Setup.

### 5. References

- [Brage, 1997] 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
- [Brage, 2002] C. Brage and K. Sjöström (with contributions of J. Neeft, S. van Paasen, M. Suomalainen, U. Zielke, G.-J. Buffinga, Ph. Hasler and J. Good), "An outline of R&D work supporting the Tar Guideline", June 2002
- [Gansekoele, 2002] E. Gansekoele and J.P.A. Neeft, "R&D work supporting the development of a Guideline for sampling and analysis of tars in biomass producer gases. Summary of results as input for R&D report on Tar Guideline", 2DEN-02.32, January 2002
- [Neeft, 2001] J.P.A. Neeft, E. Zuiver and B.F. van Egmond, "ECN contribution to R&D work supporting the development of a Guideline for sampling and analysis of tars in biomass producer gases. Sub-report to a joint BTG-ECN report to Novem". ECN-CX-001-048, October 2001

# Appendix – Results of VTT

Data reported by Marjut Suomalainen to the project partners, april 2004.

#### Set-up

A **check-up** sampling train (a VTT method sampling train, 6 impinger bottles, solvent DCM) was connected between the **actual** sampling train and the pump (picture 1) in order to determine the amount of tar passing through the actual sampling train.



Picture 1. The set-up.

The cooling of impinger bottles in different experiments are given in the table 1. No frits were used.

Table 1. The cooling of impinger bottles

Measurement	1,2	3,4	5,6	Check-up
Solvent	IP	IP	IP	DCM
Filling of the impinge				
Bottle 1	100 ml	100 ml	100 ml	0 ml
	+ glass beads	+ glass beads	+ glass beads	+ glass beads
Bottle 2	100 ml	100 ml	100 ml	70 ml
Bottle 3	100 ml	100 ml	100 ml	70 ml
Bottle 4	100 ml	100 ml	100 ml	70 ml
Bottle 5	100 ml	100 ml	100 ml	30 ml
	+ glass beads	+ glass beads	+ glass beads	+ glass beads
Bottle 6	50 ml	50 ml	50 ml	0 ml
	+ glass beads	+ glass beads	+ glass beads	+ glass beads
Cooling of the impinge	er bottles:			
Bottle 1	+40 °C 1	+20 °C	+20 °C	0 °C
Bottle 2	+40 °C	+20 °C	+20 °C	0 °C
Bottle 3	+40 °C	+20 °C	-20 °C	0 °C
Bottle 4	+40 °C	+20 °C	-20 °C	0 °C
Bottle 5	-20 °C	-20 °C	-20 °C	-70 °C
Bottle 6	-20 °C	-20 °C	-20 °C	-70 °C

### Results

Results are in the table 2. Removal efficiency is calculated as follows: tar concentration of actual sampling train, per cent of total tar concentration (actual + check-up sampling trains).

Table 2. Results.

Sample	Cooling	Removal efficiency					
		Total GC tars	Total GC tars Average Tars from acenaphtylene to pyrene				
		%	%	%	%		
1	4 x +40C / 2 x -20C	99	100	95	07		
2	4 x +40C / 2 x -20C	100	100	98	9/		
3	4 x +20C / 2 x -20C	98	00	87	02		
4	4 x +20C / 2 x -20C	100	99	96	92		
5	2 x +20C / 4 x -20C	97	00	82	05		
6	2 x +20C / 4 x -20C	98	98	87	65		