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PARALLEL MEASUREMENTS OF TAR AND PARTICULATES

Prepared by

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Contributions by

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Pekka Simell, VTT, Finland

for

Danish Energy Agency
NOVEM
Swiss Federal Office of Energy

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Test Report
Danish Energy Agency

DEA Journal No. 51166/99-0004

September 1999

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Publication and printing:
© Danish Technological Institute, Energy Division
Kongsvang Allé 29, DK-8000 Aarhus C
Tel.: +45 89 43 85 56

EH-Tryk, Aarhus, Denmark

ISBN No.: 87-7756-558-4

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Acknowledgements

On the initiative of Mr. Henrik Flyver Christiansen, the Danish Energy Agency, it was agreed to carry out parallel measurements of particulates and tar on different gasifiers in Denmark. Initially, the object was to compare the Finnish and the Danish tar measuring method. Since then, the test has been extended to include participants from the Netherlands and Switzerland.

The participation of DTI, Denmark and VTT, Finland, was rendered possible by the Danish Energy Agency (DEA).

The participation of BTG, the Netherlands, was rendered possible by Novem.

The participation of Verenum Research was rendered possible by the Swiss Federal Office of Energy (BFE).

The participating laboratories would like to express their gratitude to the institutions that have supported the project financially.

Appendices

Parallel testing of tar and dust, contribution of BTG, November 1998, Harrie Knoef, BTG, Enschede, the Netherlands

Measurement report "Parallel measurement of dust and tar in connection with updraft and downdraft gasification of wood", ELAB-0362, January 1999, Uwe Zielke, DTI Energy, Aarhus, Denmark

"Report on Tar and Particulate Measurements in Biomass Producer Gases using the modified ETH/Verenum Sampling Method", January 1999, Philipp Hasler, Verenum Research, Zürich, Switzerland

Report on tar and particulate measurements in Denmark on 1-6 November 1998, December 1998, Pekka Simell, VTT Energy, Espoo, Finland

Summary

In November 1998, parallel tests within tar and particulate measurements were carried out at two gasification plants in Denmark at the request of the Danish Energy Agency.

The following laboratories participated in the test: BTG, the Netherlands; Danish Technological Institute, Denmark; Verenum Research, Switzerland, and VTT, Finland.

Four different measuring methods were applied in connection with a number of simultaneous samplings. A description of the utilized sampling trains, sampling procedures and analysis methods appear from chapter 1 in the report. Chapter 2 briefly describes the various plants and states operational related problems influencing the measurements. In addition, problems that have been observed in connection with sampling and post sampling procedures have been mentioned.

Measurement results with related comments concerning differences and possible measuring errors are discussed in chapter 3. Particle measurement in the updraft gas results in two different perceptions of the measurement results that are one order of magnitude from each other. The reason is obviously that particulates and tar are mixed up. This measuring error does not appear in connection with measurements in the downdraft gas, as the smaller tar content of the downdraft gasifier makes it impossible to make such serious measuring errors as seen on the updraft gasifier. In general, the gravimetric measuring methods result in the greatest differences between the four laboratories. The concepts "heavy tar" and "light tar" require a more exact definition if a distinction is to be made in future. In any case, the two fractions must not overlap and, of course, there has to be agreement about the evaporation conditions by determination of heavy tar, which unfortunately has not been the case for this parallel test.

In general, the gas chromatographical analyses show rather good agreement in cases, where attention is given to specific substances. In connection with volatile tar components such as benzene and toluene the use of chilled solvent has turned out to be more efficient than the use of solid adsorbents (XAD-2).

If attention is given to the results of the individual participants, then it can be ascertained that most deviations have a reasonable explanation causing some results to be vitiated with systematic deviations. They can be eliminated by standardizing the measurement method.

A comparison of the financial aspects of the applied measuring methods appears in chapter 4. Expenses for equipment, sampling and analyses have been stated. The cost of the methods used show some variation, which also reflects the type of results generated. More detailed information generally increases the expenses for sampling and analysis. All methods are rather time-consuming in connection with installation of equipment, sampling and postsampling procedures on site. None of the methods exhibit significant advantages in this respect and therefore the costs connected with sampling and postsampling of the methods are comparable.

Please refer to the conclusion on page 43 and 46 for a general evaluation of the results of the parallel test.

1. Scope and objective of the parallel test

The measurements document the results of 4 different methods of measuring tar and particulates in producer gas from wood gasification. As far as possible, the measurements have been carried out simultaneously on a representative gas flow before and after the gas purification aggregate of the plant. Measurements were performed on two anonymous Danish gasification plants. The first plant, called "plant A", has an updraft gasifier, operating on wood chips and the second plant, called "plant B", has a downdraft gasifier, operating on wood offcuts. The measurements were carried out in week 45/1998 by:

G.J. Buffinga and Harrie Knoef
BTG Biomass Technology Group B.V., University of Twente, Enschede, the Netherlands

Poul D. Kellberg, Finn Petersen and Uwe Zielke
Danish Technological Institute, Energy Division, Aarhus, Denmark

Philipp Hasler
Verenum Research, Zürich, Switzerland

Pekka Simell, Mikko Grommi and Kari Koskela
VTT Energy, Espoo, Finland

Uwe Zielke, Danish Technological Institute, coordinated the implementation of the measurements and the discussions of the results once the measurement reports of the four laboratories were available. Mr. Elfin Larsen from the Risoe National Laboratory, Denmark, participated in the subsequent discussions.

The following measurements have been carried out:

A number of tar and particulate measurements on updraft gasifier in raw gas (bypass gas stream)
A number of tar and particulate measurements on updraft gasifier in cleaned bypass gas stream after scrubber
A number of tar and particulate measurements on downdraft gasifier in raw gas
A number of tar and particulate measurements on downdraft gasifier in cleaned gas after scrubber

In addition, the composition of the producer gas is continuously measured and operating parameters are registered for the test period.

The results are compared with the various sampling and analysis methods for particulates and tar:

- Raw gas with high tar concentration
- Scrubbed gas with low tar concentration
- Raw gas with high dust concentration
- Scrubbed gas with low dust concentration

Possible reasons for discrepancies are discussed.

2. How the measurements were carried out

2.1 Applied sampling and analysis methods

Sampling has been carried out according to the methods of the four laboratories for tar and particulate measurement. The different sampling trains appear from table 1. A description of the used analysis methods appears from table 2 – 4. For more detailed information on the used measurement methods please refer to the attached measurement reports from the four participants.

2.1.1 Comparison of sampling trains

	BTG	DTI	Verenum	VTT
1. Sampling train				
Related standards		Particulates: VDI 2066 Tar: VDI 3499 Bl.2		
Particle filter	Glass fibre filter thimble 75 x 250	Glass frit G3 i.d. 90 or Strëhlein filter cartridge 26 x 65 with quartz wool	Glass fibre filter thimble 33 x 205	Quartz fibre thimble 30 x 77
Filter temperature	A 120 °C B 120 °C	A 105 °C B 250 °C	A 200 °C raw gas 120-150 °C clean gas B 150 °C	A 250 °C B 250 °C
Condenser	RVS Vessel 118 x 300 with 6 m spiral tube 2 °C	Glass cooler 70 x 500 8 °C	Glass cooler 20 x 500 8 °C	1 bottle with glass beads 0 °C
Gas washing system	None	None	3 impinger bottles with 50 ml anisole 1 drop separator -3 °C	3 bottles with DCM 0 °C 1 bottle with DCM 1 bottle with glass beads -70 °C
Adsorption system	Cotton wool in glass tube for visual inspection	Glass frit G3 i.d. 90 with 100 ml XAD2	None	None
Vacuum system	Sliding vane gas pump 22 m^3/h Volume meter	Membrane pump 11 m^3/h Orifice gauge 1.5-7 m^3/h Electronic volume meter with flow indicator	Membrane pump 14 m^3/h Rotameter 0-25 l/min. Volume meter	Membrane pump Rotameter 0-20 l/min. Volume meter

Table 1: Description of sampling trains

2.1.2 Comparison of methods for analysing the particulate content in producer gas

BTG	DTI		Verenum	VTT
2. Gravimetric determination of particulates (dust)				
Dry the filter thimble at 105 °C, exsiccator, 1st weighing. Filter exposure, extract the filter in Soxleth (DCM), dry the filter at 105 °C, exsiccator, 2nd weighing.	A: With weighed membrane filters carry out filtration of extracts from frit, PTFE tubing, glass cooler washings and condensate. The filters are dried at 105 °C, exsiccator, 2nd weighing.	B: Dry, weighed Ströhlein filter is exposed. Dry the filter at 105 °C, exsiccator, 2nd weighing.	Dry the filter thimble at 105 °C, exsiccator, 1st weighing. Filter exposure, extract the filter in Soxleth (anisole and methanol), dry the filter at 105 °C, exsiccator 2nd weighing.	Dry the filter thimble at 105 °C, exsiccator, 1st weighing. Filter exposure, extract the filter in Soxleth (DCM), dry the filter at 105 °C, exsiccator, 2nd weighing.
	Heat the filters to 450 °C in N_2 for 30 min., exsiccator, 3rd weighing.	Heat the filter to 450 °C in N_2 for 30 min., exsiccator, 3rd weighing.	The difference between 1st and 2nd weighing is "Solid C". Filtration of sampled liquids, Remanence is "Solid A".	Non-extracted matter is particulates. Filtration of solvents.
The difference between 1st and 2nd weighing is the amount of particulates.	The difference between 1st and 3rd weighing is the amount of particulates.	The difference between 1st and 3rd weighing is the amount of particulates.	The amount of particulates is the sum of solids A and C.	The amount of particulates is the sum of non-extracted matter from the filter and the remanence from the solvent filtration.

Table 2: Gravimetric determination of particulates (dust)

2.1.3 Comparison of methods for analysing the heavy tar content in producer gas

	BTG	**DTI**	**Verenum**	**VTT**
3. Heavy tar analyses				
Definition of heavy tar	Residue of evaporation at room temperature (= total tar) Residue of evaporation of the samples at 105 °C	Residue of evaporation according to SP-Method 1071	Residue of evaporation of (worked up) anisole solution at boiling point of solvent (153 °C)	Organic compounds with molar mass > 200 g/mol
Preparation of extraction samples	Filter thimbles: Soxhlet until the DCM is colourless	Frits: Shaken 10 min. with acetone on each side and 10 min. with DCM on each side XAD 2: Soxhlet with acetone for 7 hours	Filter thimbles: A: Soxhlet with anisole and methanol B: Soxhlet with anisole	
Sample fractions	1. Extract from hot filter thimble 2. Washings from tubing 3. DCM fraction from condenser 4. Extract from 2^{nd} filter if such has been used	A: 1. Extract from frit 2. Washings from probe + tubing 3. DCM + acetone fraction from glass cooler 4. Extract from XAD 2 and 2^{nd} frit B: 1. Washings from probe + tubing 2. VOC on filter (450 °C) 3. DCM + acetone fraction from glass cooler 4. Extract from XAD 2 and 2^{nd} frit	1. Extract from filter thimble 2. Extract from aqueous solution incl. washings 3. Organic solution from tar impingers and washing with anisol 4. Organic solution from washing with acetone	1. Extract from filter 2. Organic solution from tar impingers and washings from tubing
Evaporation conditions	105 °C until the change of weight gets small. The trend line's crossing point with y-axis.	Rotating evaporator at 25 °C and vacuum 200 mbar abs., until the solvents are evaporated. Continue for another 10 min. and flush with N_2 for 30 min.	Heated water bath 85 °C. Vacuum 10 to 20 mbar abs. Until visible smoke formation.	105 °C for 1 hour

Table 3: Description of heavy tar measurement methods

2.1.4 Comparison of methods for analysing the light tar content in producer gas

	BTG	DTI	Verenum	VTT
4. Light tar analyses				
Definition of light tar	Difference between total tar and heavy tar	1. Organic compounds in aqueous solution with molar mass from 78 to 202 and 2. Organic compounds in solvents with molar mass from 78 to 202		Organic compounds with molar mass from 78 (79) to 202
Principle of analysis	Calculation of difference	Aqueous solution: GC/FID Solvent extracts: GC/MS	Phenols: Photometric method PAH: GC/MS	
Chromatogr. conditions		GC/MS Column: CP-SIL 5 CB 50 m x 0.25 mm i.d. phase 0.25 µm Oven: 35 °C for 1 min. 35-280 °C 25 °C/min. 280 °C for 27 min.		GC/MS Column: 50 m x 0.32 mm i.d. phase 0.52 µm Oven: 30-300 °C 3 °C/min. 300 °C for 60 min.

Table 4: Description of light tar measurement methods

2.2 Measurement environment and remarks to the samples

2.2.1 "Plant A"

"Plant A" is a 5 MW updraft gasifier utilizing wood chips with an average water content of 45%. The samples are taken in a bypass gas line where a gas flow of 350 Nm^3/h is led to a pilot gas scrubber. Measurements are carried out before as well as after the scrubber. The gas line internal diameter before the scrubber is 213 mm. The samples are taken at one point, centrally in the gas line. The gas temperature and pressure upstream the scrubber are 80 °C and –150 Pa, respectively, and the average flow velocity is calculated to be 3.5 m/s.

As the sampling port was too long, Verenum was not able to mount a 90° nozzle on the probe. Therefore, sampling was carried out perpendicularly in relation to the flow direction of the gas, which, of course, contributed to potential measuring errors.

Visible smoke formation appeared in Verenum's as well as VTT's impinger bottles and as a consequence Verenum found tar in the active carbon protection system in front of the pump. In order to improve the sampling efficiency VTT added dichloromethane to the empty bottle in their impinger train.

Because of considerable tar concentrations, particularly BTG had problems with tar penetration in the sample train. Visible smoke was noted from the exhaust pipe of the sampling train. At 9:45 a.m. BTG interrupted the measurement as the pump of the sample train was clogged with tar. Furthermore, BTG and Verenum had certain problems with the filter thimbles as they partly went to pieces during handling.

As far as DTI is concerned, a minor browning of the silica gel in the sample train was noted. That might indicate that the DTI method cannot hold back tar completely either.

Downstream of the scrubber, the gas line internal diameter is 108 mm. The samples are taken at one point, centrally in the gas line. The gas temperature and pressure after the scrubber are 42 °C and 1000 Pa, respectively. After the scrubber the average flow velocity is calculated to be 12.1 m/s. The gas scrubber was started just before sampling.

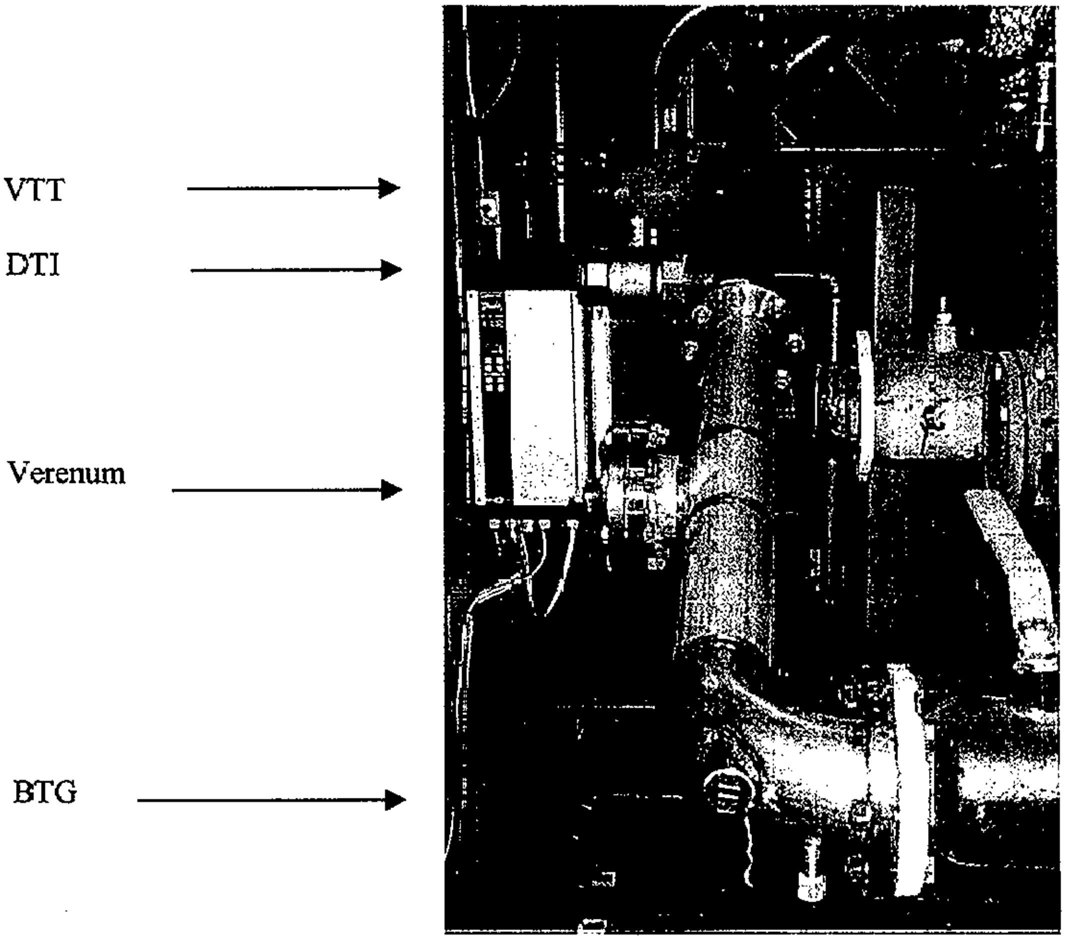

Photo 1: Sampling ports on clean gas line

At app. 10:00 a.m. BTG carried out the sampling without a pump as there were clogging problems in their pump. Sampling was solely carried out by means of the overpressure in the gas pipe. That may have resulted in measuring errors.

2.2.2 "Plant B"

"Plant B" is a 1 MW downdraft gasifier; wooden offcuts with an average water content of 12% are used. The samples are taken in the main gas line before and after the scrubber. The gas flow is app. 240 Nm^3/h. The gas line internal diameter upstream the scrubber is 150 mm. The samples are taken at one point, centrally in the pipe. The gas temperature and pressure before the scrubber are 595 °C and –150 Pa, respectively. The average flow velocity upstream the scrubber is calculated to be 12.3 m/s.

Due to water return from the washing system to the gasifier, the gasifier was idle on November 4th 1998. In the course of the day, the gasifier was emptied of water and ashes. The plant was started at 4:00 p.m. on November 4th 1998. At 10.15 a.m. on November 5th 1998, the first sampling took place. At that point, normal operational conditions could be expected.

As the gas temperature is very high (600-700 °C), BTG's measurements were carried out with modified sampling conditions as a cooling pipe, app. 1 m long, was inserted between the sample port and the particle filter. That cooling distance might influence the measurement results.

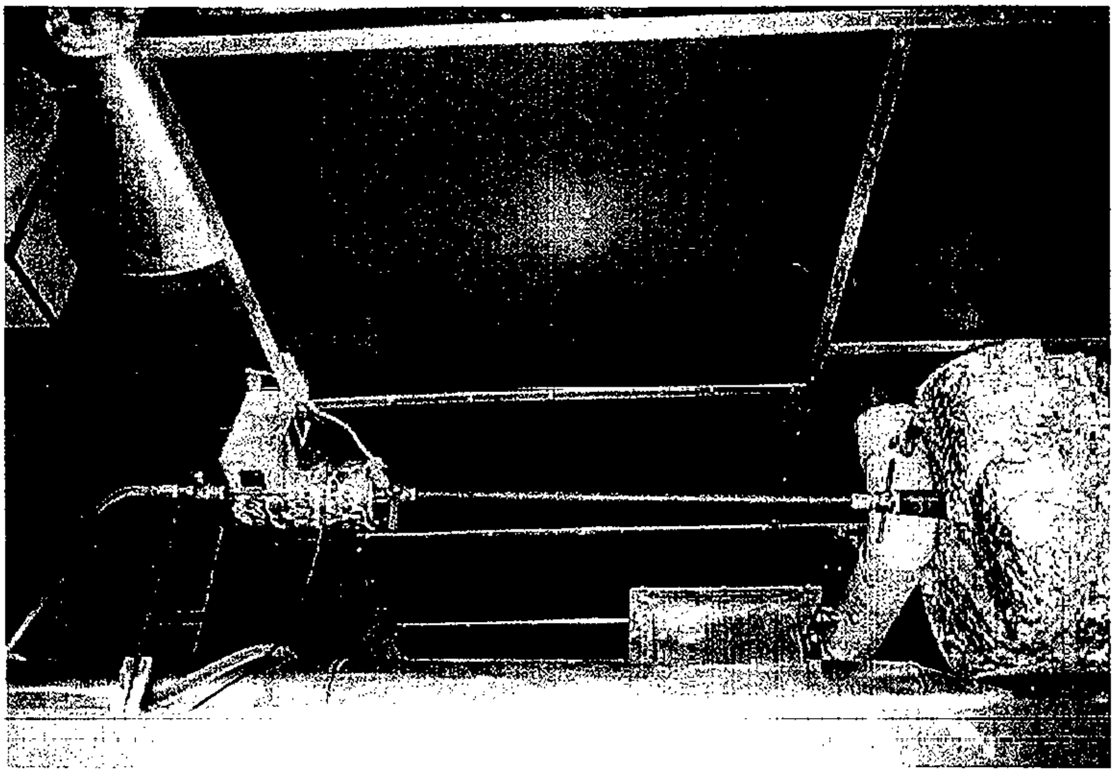

Photo 2: BTG's sampling system with cooling pipe

Downstream the scrubber the gas line internal diameter is 110 mm. The samples are taken at one point, centrally in the pipe. The gas temperature and pressure after the scrubber are 45 °C and –1000 Pa, respectively. The average flow velocity downstream the scrubber is calculated to be 9.1 m/s.

Ash transport stopped at 3:00 a.m. on November 6th 1998 due to clogging in the ash screw conveyor (caused by the mentioned accident with water penetration). The plant went down at 5:00 a.m. From 7 a.m. till 8:00 a.m. attempts were made to start the engine, but without luck. Then it was decided to continue the operation of the gasifier and to direct the gas to the emergency flare of the plant. Because of the recent stop, larger tar concentrations were to be expected shortly after starting-up and declining concentrations were to be expected in the course of the day.

2.2.3 General remarks

The intention was to carry out 3 simultaneous measurements at each sampling position, but due to various reasons that could not be realised in all cases. In general, sampling was interrupted when the pressure drop in the sampling system had reached a certain level. In other cases, there were some problems, which led to the participants having to skip a test. That could for instance be due to ice formation on the impingers, a quickly increasing pressure drop as the particle filter clogs or pump repair and cleaning of sample train due to tar penetration.

Therefore, in most cases BTG only carried out 2 measurements. As it appears from table 7 and 18, BTG takes the largest samples (up to 2470 liter). For accuracy reasons BTG needs sufficient sample volume of gas for the gravimetric determination of dust and tar.

Verenum only planned 2 measurements for each sampling position while VTT in general made more than 3 tests, but of shorter duration. VTT test duration is based on the required mass of tar in the sample, for GC-analysis. Usually, a sample volume of 40 to 50 liters is enough for VTT.

DTI carried out 3 measurements in each measuring position, as planned. In connection with all 4 sampling methods, penetration of tar aerosols in the sampling train was noted when measurements were carried out in high concentrations in the raw gas. Visible smoke penetration was observed in the impinger bottles; Verenum and DTI noted smaller tar amounts on the absorber in front of their respective pumps, and BTG had tar in the entire testing system which i.a. resulted in clogging of the pump.

In the following, when reference is made to standard volume (Nm^3), it is defined as dry m^3 gas at 0 °C and 1013 mbar, respectively.

3. Results

3.1 Measurements on updraft gasifier (Plant A)

3.1.1 Comparison of sampling conditions – period, volume, isokinetic etc.

Start and stop periods as well as sampling duration appear from the subsequent table 5 and 6. The bars in the diagram illustrate the sampling periods of the laboratories. Sample number-ing is in accordance with the designations used by the laboratories in the enclosed measure-ment reports.

Laboratory	Sample No.	Start	Stop	Duaration
BTG	Sample 1	10:05	10:33	28 Min
BTG	Sample 2	11:44	12:18	34 Min
BTG	Sample 3	14:17	14:48	31 Min
DTI	Sample 1	09:32	10:02	30 Min
DTI	Sample 2	11:43	12:13	30 Min
DTI	Sample 3	14:15	14:48	33 Min
Verenum	Sample 1	09:33	10:03	30 Min
Verenum	Sample 2	11:43	12:14	31 Min
VTT	Sample 1	09:35	10:05	30 Min
VTT	Sample 2	10:20	10:43	23 Min
VTT	Sample 3	11:45	12:13	28 Min
VTT	Sample 4	12:33	12:57	24 Min
VTT	Sample 5	14.15	14.40	25 Min
VTT	Sample 6	14:55	15:18	23 Min

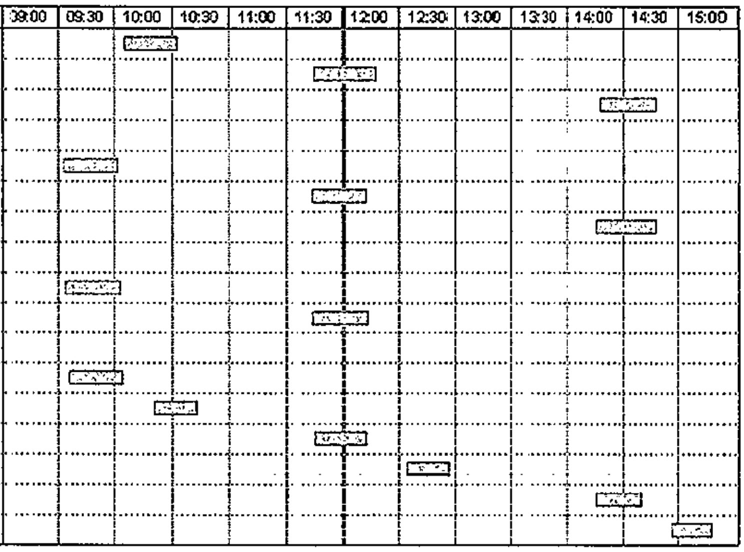

Table 5: Sampling time and duration - raw gas (Plant A)

From table 5 it appears that VTT carries out 2 measurements with and without a particulate filter each time the others carry out one. Verenum has planned to carry out only 2 measurements per sample position. BTG started at the same time as the others, but interrupted the measurement at 9:45 a.m. as there was a leak in their sample train. The system was made leak tight and a new measurement was started at 10.05 a.m. Besides, the simultaneous time phases of the measurements were satisfactory.

Laboratory	Sample No.	Start	Stop	Duaration	09:00	09:30	10:00	10:30	11:00	11:30	12:00	12:30	13:00	13:30	14:00	14:30	15:00
BTG	Sample 4	09:24	10:17	53 Min	▨	▨	▨										
BTG	Sample 5	11:47	12:38	51 Min						▨	▨	▨					
DTI	Sample 4	09:15	10:15	60 Min	▨	▨	▨										
DTI	Sample 5	11:15	12:18	63 Min					▨	▨	▨						
DTI	Sample 6	13:47	14:47	60 Min										▨	▨	▨	
Verenum	Sample 3	09:15	10:17	62 Min	▨	▨	▨										
Verenum	Sample 4	11:14	12:18	64 Min					▨	▨	▨						
VTT	Sample 1	09:20	09:45	25 Min	▨	▨											
VTT	Sample 2	09:50	10:12	22 Min		▨	▨										
VTT	Sample 3	11:15	11:35	20 Min					▨	▨							
VTT	Sample 4	11:40	12:02	22 Min						▨	▨						
VTT	Sample 5	13:50	14:09	19 Min										▨	▨		
VTT	Sample 6	14:15	14:35	20 Min											▨	▨	

Table 6: Sampling time and duration - clean gas (Plant A)

A somewhat longer sampling period was chosen in the clean gas due to the expected lower concentrations of tar and particulates. Again, VTT carried out 2 measurements of shorter duration each time the others carried out one sampling.

There was good simultaneousness during the first series of measurements. During the second series of measurements, BTG started a bit later as there was tar in the pump. The third measurement was only carried out by DTI and VTT.

The major results of the sampling appear from the subsequent table 7. Closer analyses of the figures show that deviations in isokinetic sampling do not consistently lead to the expected diviation in the measuring results. In the case of too low suction speed too much dust is normally measured and in connection with too high suction speed too little dust is normally measured. However, in connection with positive suction error, the measuring error is much smaller than in connection with negative suction error. That condition is not confirmed by what is written overleaf and therefore it is anticipated that the results are outshadowed by other sources of errors.

Lab.	Sample No.	Sample vol. Nm³	Sample flow Nm³/h	Nozzle size mm	v_o/v_t	Pressure drop mbar	Sampled condensate		Sampled matter, total	
					-		g	g/Nm³	g	g/Nm³
BTG	1	1.56	2.47	16	1.69	0-10	810	518	207	133
	2	1.63	2.88	16	1.97	0-10	681	418	-	-
	3	1.14	2.22	16	1.52	0-10	456	398	144	126
DTI	1	0.3007	0.60	15	0.56	50-100	146.1	486	23.9	79
	2	0.3185	0.64	15	0.65	50-100	191.9	603	29.7	93
	3	0.366	0.67	15	0.67	50-100	211.1	578	35.8	98
Verenum	1	0.335	0.68	10	1.42	100	203	606	22	65
	2	0.328	0.64	10	1.33	70	195	595	25	76
VTT	1	0.088	0.26	5	0.73	50-100	n.m.	n.m.	3.9	44
	2	0.074	0.19	5	1.00	50-100	n.m.	n.m.	3.4	46
	3	0.083	0.18	5	1.08	50-100	n.m.	n.m.	3.2	39
	4	0.074	0.19	5	1.04	50-100	n.m.	n.m.	3.3	44
	5	0.074	0.18	5	1.09	50-100	n.m.	n.m.	3.4	46
	6	0.073	0.19	5	1.01	50-100	n.m.	n.m.	2.9	40
BTG	4	0.9	1.02	16	0.14	0-10	61	67	15	16.5
	5	2.01	2.36	16	0.33	0-10	214	106	13.6	6.8
DTI	4	1.0384	1.04	6	1.21	50-100	100	96	5.3	5.1
	5	1.0581	1.00	6	1.17	50-100	92.1	87	4.9	4.6
	6	0.9852	0.99	6	1.14	50-100	91.9	93	4.1	4.1
Verenum	3	0.781	0.755	6	0.77	100	70	90	3.6	4.6
	4	0.808	0.758	6	0.78	50	78	97	3.8	4.7
VTT	1	0.142	0.34	3.5	1.08	50-100	n.m.	n.m.	0.61	4.3
	2	0.141	0.38	3.5	0.96	50-100	n.m.	n.m.	0.34	2.4
	3	0.132	0.40	3.5	0.93	50-100	n.m.	n.m.	0.30	2.2
	4	0.131	0.36	3.5	1.03	50-100	n.m.	n.m.	0.28	2.1
	5	0.132	0.42	3.5	0.88	50-100	n.m.	n.m.	0.27	2.1
	6	0.131	0.39	3.5	0.94	50-100	n.m.	n.m.	0.32	2.4

Table 7: Comparison of specific sampling values (Plant A)
n.m. = not measured

The values above the thick line in table 7 are from measurements in raw gas and the values below the thick line are from measurements in clean gas. In the following, comments will be made to the values in table 7:

Sample volume: There are rather large differences in the sampled volumes. The general trend is that the total sampled matter decreases with the lower volume sampled.

Isokinetic sampling: The deviation in isokinetic sampling is given by the ratio between obtained velocity and target velocity (v_o/v_t). The calculated average target velocity was given to the participants by DTI before sampling. It appears from the table that the samples were taken under-isokinetic as well as over-isokinetic. This could lead to possible errors, especially in larger particle sampling. In tar sampling it is normally of minor importance, but as tar can condense on the particles, isokinetic sampling should be aimed at in all cases.

Pressure drop: These figures show the maximum pressure drop increase in the sampling train. That could be effected by clogging filters or by ice formation in the impingers. The values reveal no problems of any kind.

Sampled condensate: In average, the four laboratories have measured 445 g/Nm^3 and 87 g/Nm^3 (BTG); 556 g/Nm^3 and 92 g/Nm^3 (DTI); 600 g/Nm^3 and 94 g/Nm^3 (Verenum). VTT has not determined the amount of aqueous condensate. The first value refers to raw gas and the second to clean gas. There is quite good agreement on the level of condensate in the gas. However, BTG's raw gas value is rather low and in the clean gas BTG has considerable deviation that cannot be accounted for by the operation of the plant. The reason could be that no gas pump was used during sample 4. DTI and Verenum's measurements show greater constancy.

Sampled matter, total: "Sampled matter, total" is dust and tar that has been gravimetrically analysed. Verenum's values also include the water-soluble organic residue. In average, the four laboratories have measured 130 g/Nm^3 and 12 g/Nm^3 (BTG); 90 g/Nm^3 and 4.6 g/Nm^3 (DTI); 71 g/Nm^3 and 4.7 g/Nm^3 (Verenum); 43 g/Nm^3 and 2.6 g/Nm^3 (VTT). VTT's figures are smaller because they only include heavy tar; total tar has not been measured by VTT. If it is assumed that heavy tar forms app. 50 % of total tar, then there is accordance between VTT, DTI and Verenum. BTG's measurements deviate from the others and that is probably due to the same reasons as mentioned previously.

3.1.2 Gravimetric measurement of particulates in raw gas (Plant A)

Sampling period 02. 11. 98	BTG mg/Nm^3	DTI mg/Nm^3	Verenum mg/Nm^3	VTT mg/Nm^3
09:32-10:05		259	3440	0
10:05-10:33	5138			
10:20-10:43				300
11:43-12:13	-	414	8260	0
12:33-12:57				200
14:15-14:48	4070	150		200
14:55-15:18				400

Table 8: Comparison of particle measurements in raw gas (Plant A)

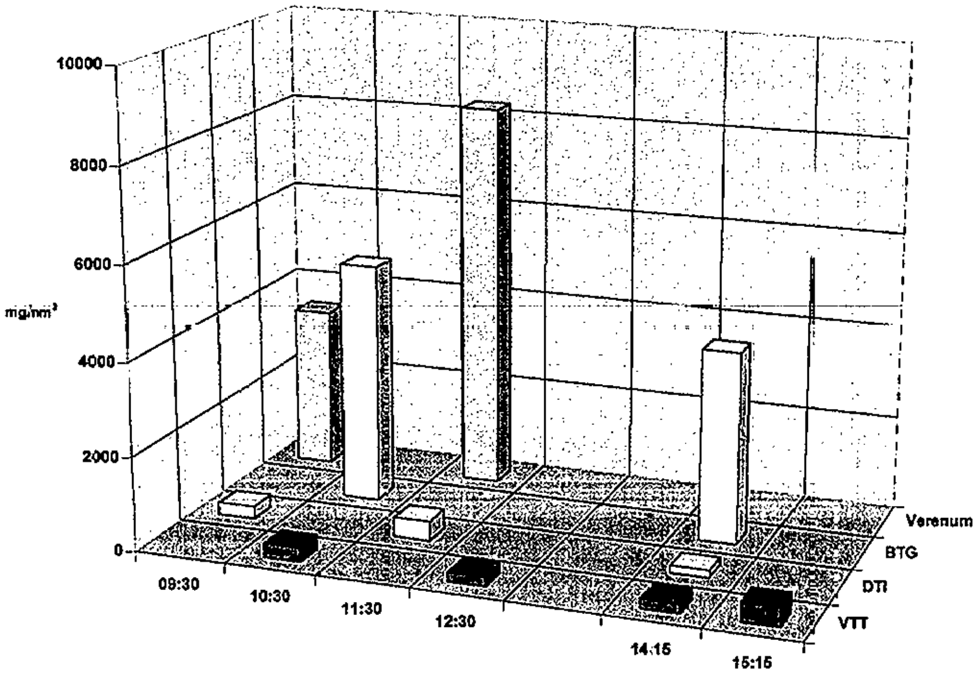

Figure 1: Particulates in raw gas (Plant A)

As it appears from figure 1, there is considerable disagreement on the dust content in the raw gas. VTT's and DTI's results are in the same order of magnitude, but BTG's and Verenum's results are one order of magnitude higher. The following result analysis has led to the conclusion that BTG's and Verenum's particle measurements contain considerable tar residue that cannot be extracted from the filter thimbles. It is not possible to say why VTT can extract the filters to 100% cleanness by using DCM, and why BTG apparently cannot.

In order to remove the tar residue from the particles DTI heats the filters to 450 °C in an inert asmosphere. In that way, the filters lose app. 50% in weight. That does not fit in with the much higher differences illustrated in figure 1.

3.1.3 Gravimetric measurement of particulates in gas after gas scrubber (Plant A)

Sampling period 03.11.98	BTG mg/Nm³	DTI mg/Nm³	Verenum mg/Nm³	VTT mg/Nm³
09:15-10:15	172	44	200	-
11:15-12:18		23	210	-
11:47-12:38	316			-
13:47-14:47		16		-

Table 9: Comparison of particulate measurements in gas after scrubber (Plant A)

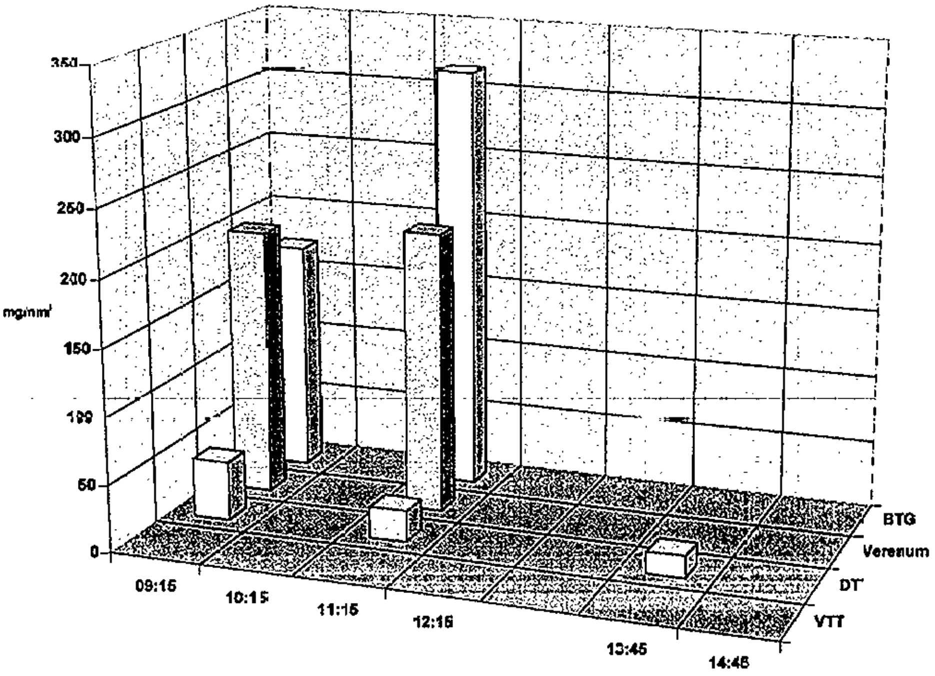

Figure 2: Particulates in clean gas (Plant A)

As it appears from figure 2, there is also disagreement on the dust content in the clean gas. VTT has not measured dust there as the particulate measurement was not in their quotation to DEA. BTG's and Verenum's results are one order of magnitude higher than DTI's results. Once again, we assume that the high values are due to tar residue that could not be extracted from the filter thimbles probably because part of the collected tar polymerises.

It is Verenum's opinion that the high values could be correct due to possible loss of submicrone particles in the DTI system. However, it is DTI's experience that the submicrone particles (because of small mass) contribute inessentially to the result of the gravimetrical particle measurement. Furthermore, the particle content of the sample train after the filter has been examined and only 19% of the total particle mass after the filter has been found.

3.1.4 Gravimetric measurement of heavy tar in raw gas (Plant A)

Table 10 and figure 3 illustrate the content of heavy tar in the raw gas, determined by weighing the residue of evaporation.

Sampling period 02.11.98	BTG g/Nm^3	DTI g/Nm^3	Verenum g/Nm^3	VTT g/Nm^3
09:32-10:05		79	41.2	44.2
10:05-10:33	51.4			
10:20-10:43				45.6
11:43-12:13	38.6	93	44.2	38.6
12:33-12:57				43.8
14:15-14:48	46.9	97		45.7
14:55-15:18				39.7

Table 10: Comparison of heavy tar measurements in raw gas (Plant A)

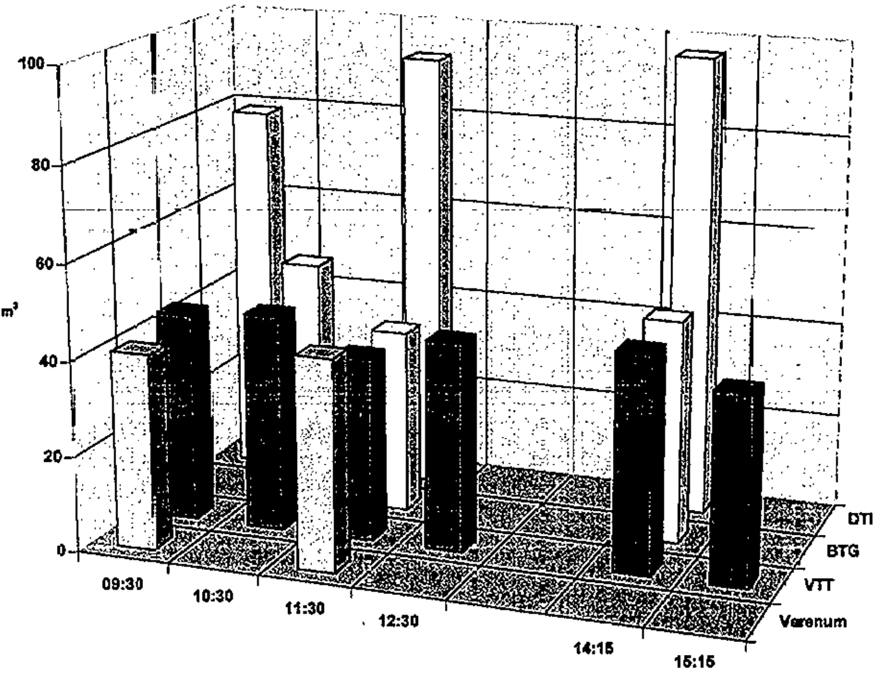

Figure 3: Heavy tar in raw gas (Plant A)

From figure 3 it appears that there is good agreement between BTG, Verenum and VTT. DTI's measurements are app. one factor 2 above the others. The reason is that DTI evaporates at 25 °C and 200 mbar abs. The other three laboratories evaporate at higher temperatures and varying pressures (see table 3). It is also possible that the DTI values contain a surplus of moisture.

3.1.5 Measurement of light tar in raw gas (Plant A)

Table 11 illustrates the content of light tar in the raw gas. BTG's results have been calculated as the difference between totall tar (residue of evaporation at room temperature) and heavy tar (residue of evaporation at 105 °C). The remaining results are based on GC analyses (total area incl. non-identified peaks). The values of DTI and VTT show tar components with a molar mass from 78 to 202. Verenum's values only include PAH and water soluble organic residue, phenols. The value marked with * was informed by VTT in connection with subsequent discussions as VTT did not officially measure light tar on the updraft gasifier.

Sampling period 02.11.98	BTG g/Nm^3	DTI g/Nm^3	Verenum g/Nm^3	VTT g/Nm^3
09:32-10:05		33.1	20.1	-
10:05-10:33	76.6			-
10:20-10:43				-
11:43-12:13	52.6	35.3	23.6	-
12:33-12:57				-
14:15-14:48	75.1	38.1		25.9 *

Table 11: Comparison of light tar measurements in raw gas (Plant A)

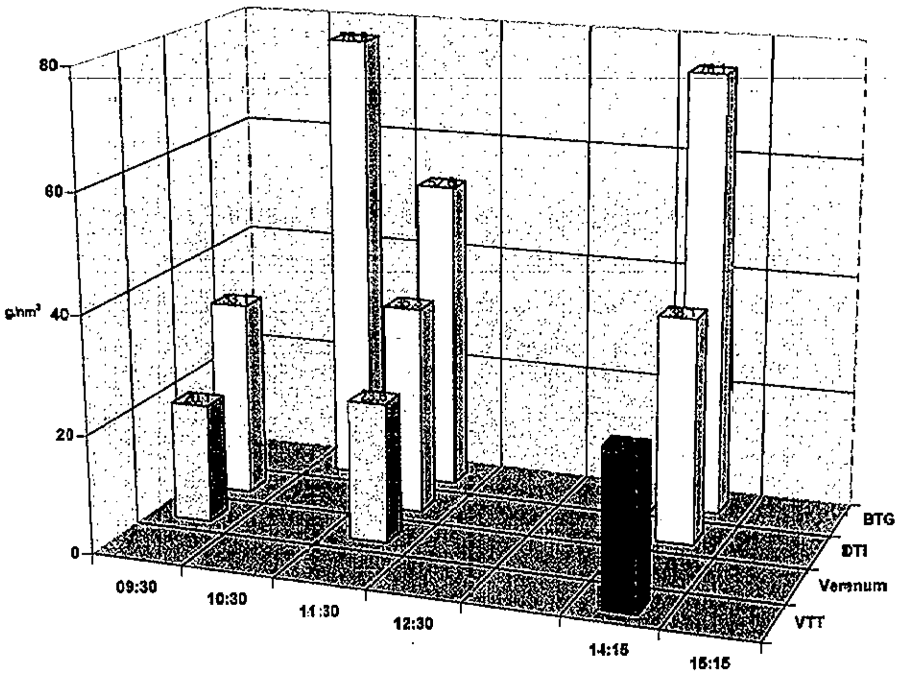

Figure 4: Light tar in raw gas (Plant A)

In this case, differential weighing of the residue of evaporation leads to larger results than in connection with the GC analysis. The GC results are apparently in rather good agreement. Verenum's figures are smallest as they only include part of the tar components. Since all participants have used different definitions and analysis methods it is not possible to compare the results from light tar measurements. A closer examination of the chromatograms shows that considerable deviations appear on single components but most of the results are in good agreement, as it appears from the next section.

3.1.6 Comparison of specific tar components in raw gas (Plant A)

In the following, results will be shown for selected individual components determined by a GC analysis carried out by 3 of the participating laboratories. DTI's and VTT's measurements were carried out simultaneously, and in relation to time Verenum's measurement No. 2 comes closest to the other measurements. The thick line in table 12 indicates the separation line between light tar and heavy tar according to VTT's definition. Components below the broken line are PAHs.

Tar component	DTI No. 3 mg/Nm^3	Verenum No. 2 mg/Nm^3	VTT No. 5 ** mg/Nm^3
Benzene	136		1452
Toluene	276		540
Phenol	1053	2530 *	1292
Xylene	153		198
Indene	39		48
Naphthalene	65	0.4	121
Acenaphthylene	9.3	0.4	38
Acenaphthene	3.6	0.4	34
Fluorene	13	10.4	33
Phenanthrene	23	19.8	35
Anthracene	6.6	4.6	0
Fluoranthene	3.4	5.0	132
Pyrene	5.1	3.3	0
Benz(a)anthracene	2.3	1.0	
Chrysene	2.4	1.6	
Benz(b+k)fluoranthene	1.4	0.8	
Benz(a)pyrene	1.2	2.4	
Indeno(1,2,3)pyrene	0.4	0.4	
Benz(ghi)perylene	0.3	0.4	
Dibenz(ah)anthracene	0	0.4	
Total PAH	137	51.3	393

Table 12: Comparison of specific tar components in raw gas (Plant A)

** *The shown components should be verified by GC-MS, because the GC chromatogram can include some unidentified components with the same retention time as the one that has been indicated.*

From table 12 it appears that the DTI method has a shortcoming in relation to adsorption of the most volatile components by the adsorbent XAD-2. The reason can be insuficient contact time, channelling in XAD-2 or losses during post sampling activities. Therefore, DTI only finds app. 10% of VTT's value. From phenol and higher the method is equal to the others. DTI's measurement values originate from DCM and acetone extract analysed by GC-MS. As it appears from the attached DTI report, light tar has also been found in the aquous condensate, which, presumably, mainly consists of the easily soluble phenols in water.

Verenum measures the total of phenolic compounds (*) by applying a photometric method according to ASTM D 1783-91. The value is of course greater than that of the other two laboratories as their values only include phenol. In addition, it is noted that Verenum's GC analysis gives faulty results in connection with the lower boiling PAH compounds. The reason is presumably that the naphthalene peak drowns in methyl-guaiacol, which has almost the same retention time. From the enclosed DTI measuring report it appears that guaiacol and 4-methyl guaiacol are two of the main components of "light tar". However, these substances are not analysed specifically by the other participants.

VTT's method is effective when confronted with tar components with low boiling points. Compared with DTI's and Verenum's PAH values, the results of VTT deviate and in connection with fluoranthene, there is apparently an error. The reason is apparently interference with other components.

3.1.7 Gravimetric measurement of heavy tar in gas after gas scrubber (Plant A)

Sampling period 03.11.98	BTG g/Nm³	DTI g/Nm³	Verenum g/Nm³	VTT g/Nm³
09:15-10:15	3.12	5.0	2.54	4.3 and 2.4
11:15-12:18	2.4	4.6	2.74	2.2 and 2.1
13:47-14:47		4.1		2.1 and 2.4

Table 13: Comparison of heavy tar measurements in gas after scrubber

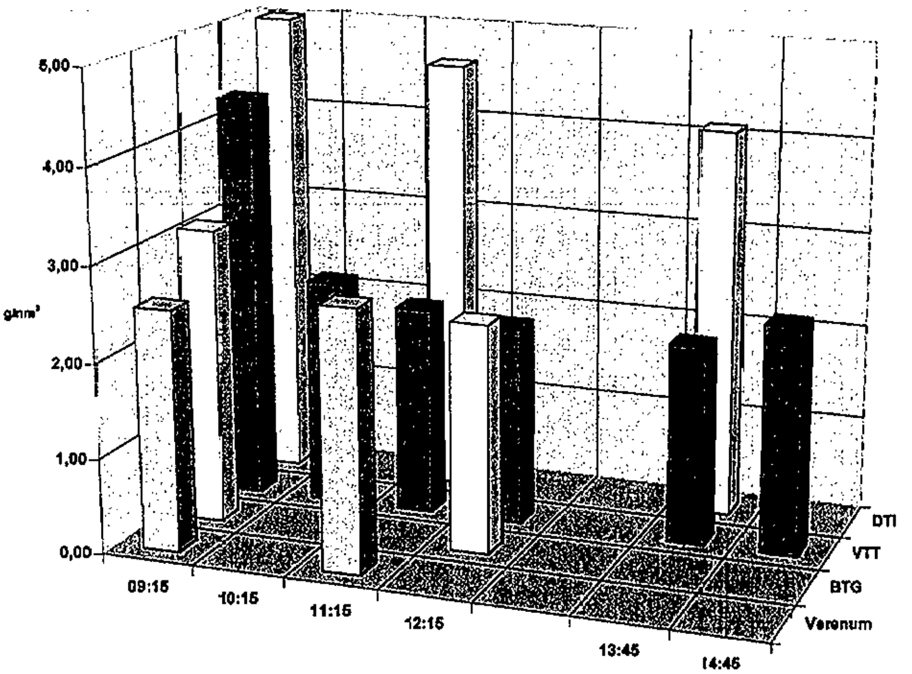

Figure 5: Heavy tar in clean gas (Plant A)

Again, there is relatively good agreement between BTG's, Verenum's and VTT's results and DTI is once again app. factor 2 above the others due to a difference in temperature of evaporation and differing pressure. VTT's first measurement is apparently a measuring error.

3.1.8 Measurements of light tar in gas after gas scrubber (Plant A)

Sampling period 03.11.98	BTG mg/Nm³	DTI mg/Nm³	Verenum mg/Nm³	VTT mg/Nm³
09:15-10:15	13.2	4.9	1.84	-
11:15-12:18	4.1	4.2	1.82	-
11:47-12:38				-
13:47-14:47		3.8		4.5 *

Table 14: Comparison of light tar measurements in gas after scrubber (Plant A)

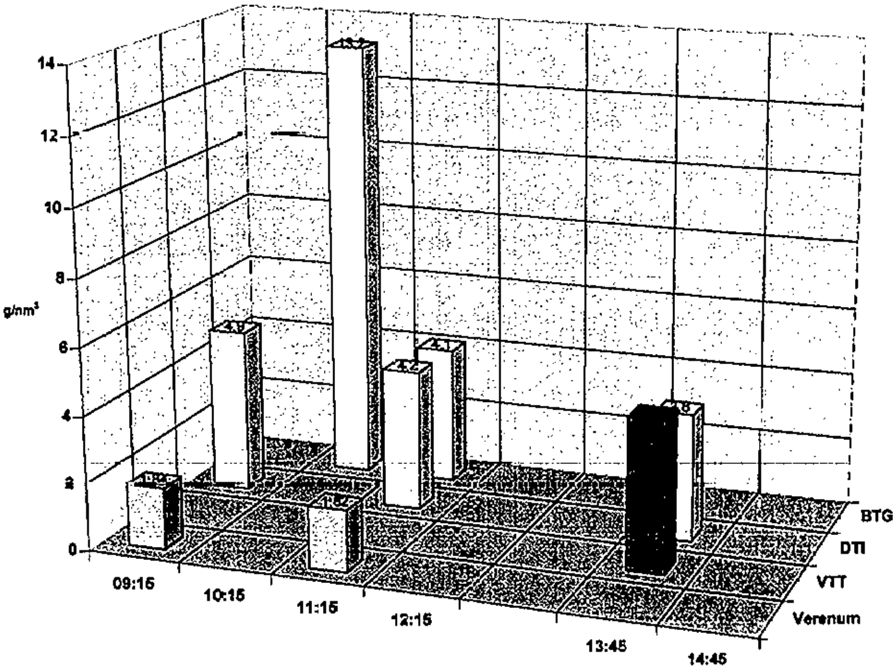

Figure 6: Light tar in clean gas (Plant A)

As previously, BTG's results are calculated as the difference between total tar and heavy tar. The other results are based on GC analyses. The values of DTI and VTT show tar components with molar mass from 78 to 202. Verenum's values only include water-soluble organic residue, phenols and PAH. The value marked with * was informed by VTT in connection with subsequent discussions as VTT unofficially measured light tar on the updraft gasifier.

Of the same reason as mentioned earlier, Verenum's results are lower than those of the other laboratories, as they only include part of the tar content.

As previously mentioned BTG's initial measurement was sampled without a pump. That might also be the reason for the considerable deviation. As all participants have used different definitions and analysis methods, it is not possible to compare the results from light tar measurement.

3.1.9 Comparison of specific tar components in clean gas (Plant A)

In the following, results are shown for selected individual components determined by a GC analysis carried out by the 3 participating laboratories. The three selected measurements were carried out simultaneously. The thick line i table 15 indicates the separation line between light tar and heavy tar according to VTT's definition. Components below the broken line are PAHs.

Tar component	DTI No. 5 mg/Nm^3	Verenum No. 4 mg/Nm^3	VTT No. 5 ** mg/Nm^3
Benzene	30		294
Toluene	123		310
Phenol	86	490 *	177
Xylene	104		120
Indene	23		22
Naphthalene	27	7.3	26
Acenaphthylene	0.7	0.33	0
Acenaphthene	0.2	0.41	0
Fluorene	0.7	0.47	0
Phenanthrene	0.8	0.61	0
Anthracene	0.3	0.17	0
Fluoranthene	0.2	0.33	0
Pyrene	0.2	0.35	0
Benz(a)anthracene	0.1	0.16	
Chrysene	0.1	0.11	
Benz(b+k)fluoranthene	0.1	0.42	
Benz(a)pyrene	0.1	0.26	
Indeno(1,2,3)pyrene	0	0.13	
Benz(ghi)perylene	0	0.12	
Dibenz(ah)anthracene	0	0.09	
Sum PAH	30.5	11.26	26

Table 15: Comparison of specific tar components in clean gas (Plant A)

** *The shown components should be verified by GC-MS, because the GC chromatogram can include some unidentified components with the same retention time as the one that has been indicated.*

As it appears from table 15, DTI again found 10% of VTT's value for benzene.

Of the same reason as mentioned earlier, Verenum's value for phenol is larger than that of the other two laboratories and again too low an amount of naphtalene was found.

3.2 Measurement on downdraft gasifier (Plant B)

3.2.1 Comparison of sampling conditions – period, volume, isokinetic etc.

Laboratory	Sample No.	Start	Stop	Duaration	09:00	09:30	10:00	10:30	11:00	11:30	12:00	12:30	13:00	13:30	14:00	14:30	15:00
BTG	Sample 6	10:17	11:33	76 Min			▭	▭	▭	▭							
BTG	Sample 7	14:33	15:07	34 Min												▭	▭
DTI	Sample 7	10:15	10:31	16 Min			▭	▭									
DTI	Sample 8	11:24	11:55	31 Min					▭	▭							
DTI	Sample 9	14:30	15:06	36 Min												▭	▭
Verenum	Sample 5	10:14	11:32	78 Min			▭	▭	▭	▭							
Verenum	Sample 6	14:32	15:06	34 Min												▭	▭
VTT	Sample 1	10:15	10:27	12 Min			▭										
VTT	Sample 2	10:55	11:05	10 Min				▭	▭								
VTT	Sample 3	11:42	11:53	11 Min						▭							
VTT	Sample 4	12:13	12:24	11 Min							▭						
VTT	Sample 5	14:30	14:42	12 Min												▭	
VTT	Sample 6	14:57	15:08	11 Min												▭	▭

Table 16: Sampling time and duration - raw gas (Plant B)

BTG and Verenum have larger filter thimbles and therefore they can sample over a longer period of time than DTI and VTT. Especially in connection with sample No. 7 DTI had to stop ahead of time as the high dust concentration resulted in a rapid pressure increase in the sampling train.

Laboratory	Sample No.	Start	Stop	Duaration	09:00	09:30	10:00	10:30	11:00	11:30	12:00	12:30	13:00	13:30	14:00	14:30	15:00
BTG	Sample 8	09:02	09:22	20 Min	▭												
BTG	Sample 9	10:18	10:46	28 Min			▭	▭									
DTI	Sample 10	09:00	09:20	20 Min	▭												
DTI	Sample 11	10:15	10:43	28 Min			▭	▭									
DTI	Sample 12	11:41	12:05	24 Min						▭	▭						
VTT	Sample 1	09:03	09:14	11 Min	▭												
VTT	Sample 2	09:32	09:44	12 Min		▭											
VTT	Sample 3	10:19	10:33	14 Min			▭	▭									
VTT	Sample 4	11:44	11:57	13 Min						▭							
VTT	Sample 5	12:09	12:20	11 Min							▭						

Table 17: Sampling time and duration - clean gas (Plant B)

Of reasons related to lack of time Verenum does not measure in the clean gas at plant B. In addition, the measurements are carried out simultaneously.

Lab.	Sample No.	Sample vol.	Sample flow	Nozzle size	v_o/v_t	Max. pressure drop	Sampled condensate		Sampled matter, total	
		Nm^3	Nm^3/h	mm	-	mbar	g	g/Nm^3	g	g/Nm^3
BTG	6	1.62	1.28	12	0.90	0-10	-	-	13.2	8.2
	7	0.78	1.37	12	0.96	0-10	12	15.9	8.9	11.5
DTI	7	0.1447	0.54	8	0.89	100-250	15.7	109	0.443	3.06
	8	0.3425	0.66	8	1.10	100-150	42.1	123	0.596	1.74
	9	0.2374	0.40	6	1.15	100-150	24.9	105	0.32	1.35
Verenum	5	0.81	0.623	8	1.02	500	118	146	2.8	3.44
	6	0.233	0.403	8	0.66	150	-	-	0.53	2.27
VTT	1	0.070	0.35	5	0.78	50-100	n.m.	n.m.	0.061	0.87
	2	0.047	0.28	5	0.98	50-100	n.m.	n.m.	0.008	0.17
	3	0.056	0.30	5	0.90	50-100	n.m.	n.m.	0.041	0.73
	4	0.055	0.30	5	0.90	50-100	n.m.	n.m.	0.041	0.75
	5	0.056	0.28	5	0.98	50-100	n.m.	n.m.	0.041	0.74
	6	0.055	0.30	5	0.91	50-100	n.m.	n.m.	0.042	0.77
BTG	8	0.82	2.47	12	0.88	0-10	109	132	9.6	11.7
	9	1.04	2.22	12	0.98	0-10	138	133	2.0	1.9
DTI	10	0.2699	0.81	8	0.82	100	37.8	140	0.52	1.94
	11	0.3630	0.78	8	0.80	100	51.9	143	0.48	1.33
	12	0.3528	0.88	8	0.90	100	47.6	135	0.44	1.26
VTT	1	0.054	0.29	3.5	0.82	50-100	n.m.	n.m.	0.037	0.68
	2	0.056	0.28	3.5	0.86	50-100	n.m.	n.m.	0.109	1.96
	3	0.069	0.30	3.5	0.81	50-100	n.m.	n.m.	0.045	0.65
	4	0.065	0.30	3.5	0.81	50-100	n.m.	n.m.	0.023	0.35
	5	0.064	0.35	3.5	0.69	50-100	n.m.	n.m.	0.029	0.45

Table 18: Comparison of specific sampling values on "plant B"

The values above the thick line in table 18 are from measurements in raw gas and the values below the thick line are from measurements in clean gas. In the following, comments will be given to the values stated in table 18:

Sample volume: Also on plant B there were relatively large differences in the sampled volumes. It was noted that especially VTT collects very small samples, which makes greater demands to subsequent handling and to weighing accuracy. Again, the total sampled matter decreases with lower volume sampled, except BTG's sample No. 9.

Isokinetic sampling: The deviation in isokinetic sampling is given by the ratio between obtained velocity and target velocity (v_o/v_t). DTI gave the calculated average target velocity to the participants before sampling. It appears from the table that most of the samples were taken slightly under-isokinetic. That leads to possible errors, especially in larger particle sampling. In tar sampling it is of minor importance. Due to other sources of errors/ uncertainties it is not very likely, as mentioned previously, that the deviation in isokinetic sampling is of substantial importance to particle determination.

Pressure drop: These figures show the maximum increase in pressure drop in the sampling train. This could be affected by clogging filters or by ice formation in the impingers. The values reveal no problems of any kind.

Sampled condensate: In average, the four laboratories have measured 15.9 g/Nm^3 and 133 g/Nm^3 (BTG), 112 g/Nm^3 and 139 g/Nm^3 (DTI); 146 g/Nm^3 (Verenum). VTT has not determined the amount of aqueous condensate. The first value refers to raw gas and the second to clean gas. There is quite good agreement in the level of condensate in the gas, but it is obvious that BTG has a measuring error in raw gas (15.9 g/Nm^3). The mounted cooling pipe (photo 2) might be the reason for the low value.

Sampled matter, total: "Sampled matter, total" is dust and tar that have been gravimetrically analysed. Verenum's values also include the water-soluble organic residue. In average, the four laboratories have measured 9.9 g/Nm^3 and 6.8 g/Nm^3 (BTG); 2.1 g/Nm^3 and 1.5 g/Nm^3 (DTI); 1.7 g/Nm^3 (Verenum); 0.7 g/Nm^3 and 0.8 g/Nm^3 (VTT). The first value refers to raw gas and the second to clean gas. It is noted that BTG's results differ from the others and in spite of a small amount of condensate a high tar content is found. This does not seem to indicate that the cooling pipe is the reason for the deviation.

3.2.2 Gravimetric measurement of particulate in raw gas (Plant B)

Measurement period 05.11.98	BTG mg/Nm^3	DTI mg/Nm^3	Verenum mg/Nm^3	VTT mg/Nm^3
10:15-10:31		1146		550
10:15-11:33	818		830	90
11:24-11:55		963		
11:42-11:53				560
12:13-12:24				510
14:30-15:06	864	773	660	570 and 630

Table 19: Comparison of particulate measurements in raw gas (Plant B)

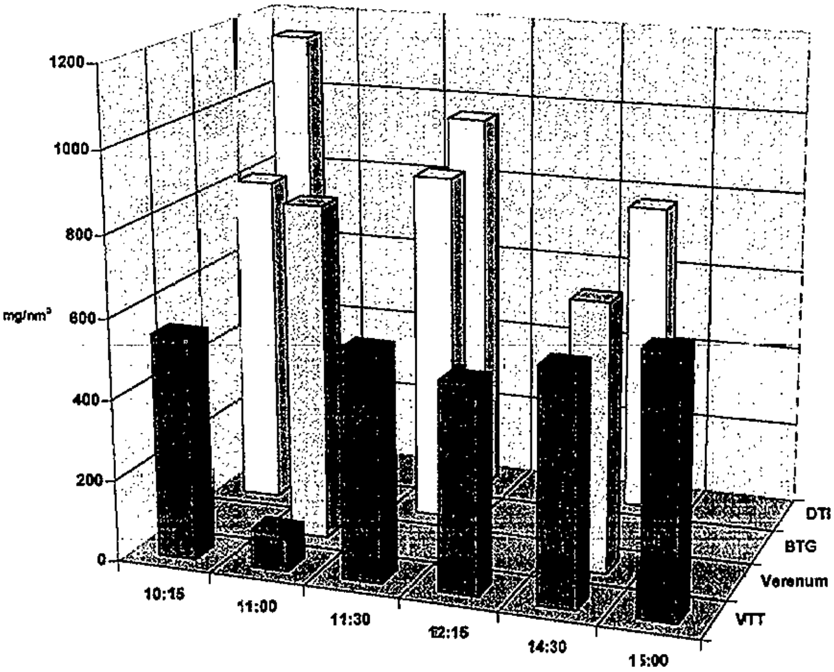

Figure 7: Particulate in raw gas (Plant B)

In general, there is greater agreement on the dust content than in connection with plant A.. That is because the smaller tar content of the downdraft gas makes it impossible to make such serious measuring errors as seen on the updraft gasifier.

As it could be expected, some measurements show a decreasing tendency in the course of the day, which is related to the recent start-up of the plant. In general, VVT's measurements are below average and measurement No. 2 is apparently faulty.

3.2.3 Gravimetric measurement of particulates in gas after gas scrubber (Plant B)

Measurement period 06.11.98	BTG mg/Nm^3	DTI mg/Nm^3	Verenum mg/Nm^3	VTT mg/Nm^3
09:00-09:20	510	604	-	470
09:32-09:44			-	1700
10:15-10:43	527	426	-	460
11:41-12:05		406	-	210
12:09-12:20			-	250

Table 20: Comparison of particulate measurements in gas after scrubber (Plant B)

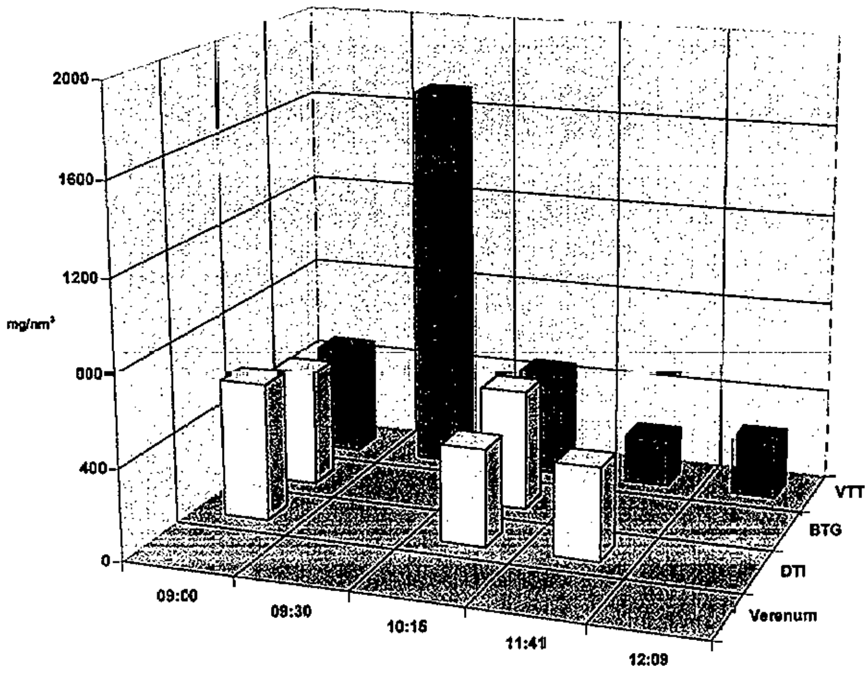

Figure 8: Particulate in clean gas (Plant B)

In this case, there is also greater agreement on the dust content than was the case with plant A. Apart from VTT's measurement No. 2 there is good agreement between the measurement values. In the course of the day, a decreasing tendency was again noted.

3.2.4 Gravimetric measurement of heavy tar in raw gas (Plant B)

Measurement period 05.11.98	BTG mg/Nm^3	DTI mg/Nm^3	Verenum mg/Nm^3	VTT mg/Nm^3
10:15-10:31		1913		-
10:15-11:33	6310		980	-
11:24-11:55		744		-
11:42-11:53				-
12:13-12:24				-
14:30-15:06	9880	561	560	-

Table 21: Comparison of heavy tar measurements in raw gas (Plant B)

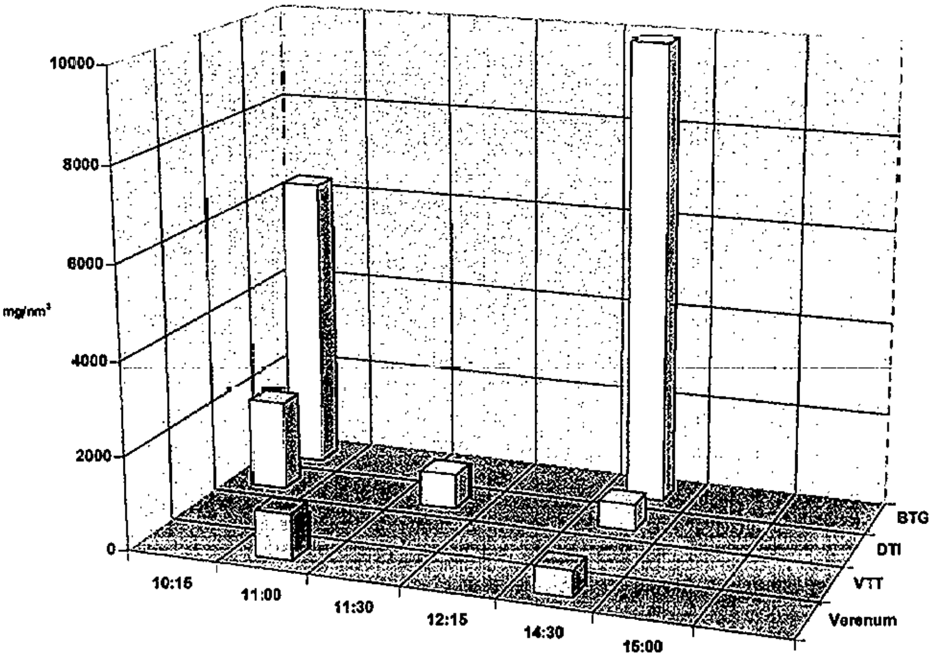

Figure 9: Heavy tar in raw gas (Plant B)

VTT has not measured heavy tar on the downdraft gasifier as that is not included in their quotation to DEA.

In downdraft gas the higher boiling tar components dominate. That, in combination with the fact that light tar contains a high amount of lower boiling components such as benzene, toluene etc., explains why there is reasonable agreement between DTI and Verenum in spite of various temperatures of evaporation at the gravimetric determination of heavy tar.

Another reason for the agreement between DTI and Verenum could be that the tar at plant B is more hydrophobic which facilitates moisture evaporation. Please see the next page for comments on BTG's results.

3.2.5 Measurement of light tar in raw gas (Plant B)

Measurement period 05.11.98	BTG mg/Nm³	DTI mg/Nm³	Verenum mg/Nm³	VTT mg/Nm³
10:15-10:31		4050	1633	6439
10:17-11:33	1050			2297
11:24-11:55		2717		3981
11:42-11:53				
12:13-12:24				5300
14:30-15:06	730	2238	1051	3880 and 3408

Table 22: Comparison of light tar measurements in raw gas (Plant B)

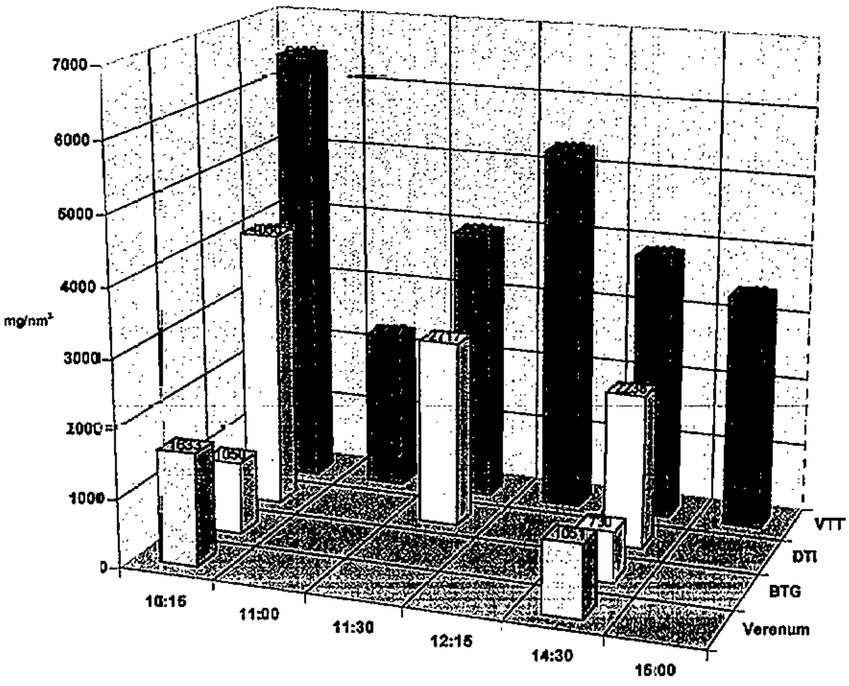

Figure 10: Light tar in raw gas (Plant B)

As previously, BTG's results are calculated as the difference between total tar and heavy tar. The other results are based on GC analyses. The values of DTI and VTT show tar components with a molar mass from 78 till 202. Verenum's values only include water-soluble organic residue, phenols and PAH.

Because of the stated reasons Verenum's measurement values are lower that those of DTI and VTT.
DTI's results are too small and that is due to lack of benzene and partly to lack of toluene.
VTT's results vary a bit and therefore measurement No. 2 seems to be too low.

BTG's results are completely different from those of the others. The heavy tar results are higher and the light tar results are lower than the results of the others. In a note to these results BTG claims that their results are correct. Because of the high boiling, tar components dominate in downdraft gas. In consequence, the results of the other participants are wrong. (BTG statement). Since all participants have used different definitions and analysis methods it is not possible to compare the results from light tar measurements.

3.2.6 Comparison of specific tar components in raw gas (Plant B)

The following specifies the results of for selected individual components determined by a GC analysis carried out by several laboratories. The chosen measurements were carried out simultaneously. The thick line in table 23 indicates the separation line between light tar and heavy tar according to VTT's definition. Components below the broken line are PAHs.

Tar component	DTI No. 7 mg/Nm^3	Verenum No. 5 mg/Nm^3	VTT No. 1+2 mg/Nm^3
Benzene	625		2380
Toluene	208		338
Phenol	95	20 *	57
Xylene	42		38
Indene	310		164
Naphthalene	624	834	624
Acenaphthylene	279	223	205
Acenaphthene	13	10.6	13
Fluorene	48	40	10
Phenanthrene	95	108	131
Anthracene	29	32	33
Fluoranthene	34	46	52
Pyrene	30	47	53
Benz(a)anthracene	5.3	8.7	
Chrysene	5.6	6.9	
Benz(b+k)fluoranthene	7.9	11	
Benz(a)pyrene	5.0	6.2	
Indeno(1,2,3)pyrene	2.8	3.9	
Benz(ghi)perylene	2.8	4.4	
Dibenz(ah)anthracene	0.2	0.9	
Total PAH	1182	1383	1121

Table 23: Comparison of specific tar components in raw gas (Plant B)

From table 23 it appears that DTI again found too little benzene.

Contrary to expectations Verenum found least phenol and most naphtahalene – actually, the opposite tendency of the values from the updraft gasifier. However, concerning the downdraft gasifier Verenum should have the same value of phenol as DTI and VTT, as no higher phenols are present.

In addition, there is good agreement in connection with PAH.

Concordantly, the laboratories found a PAH content in the downdraft gasifier that was one order of magnitude larger than in the updraft gasifier.

3.2.7 Gravimetric measurement of heavy tar in gas after gas scrubber (Plant B)

Measurement period 06.11.98	BTG mg/Nm^3	DTI mg/Nm^3	Verenum mg/Nm^3	VTT mg/Nm^3
09:00-09:20	9660	1336	-	-
10:15-10:43	1160	904	-	-
11:41-12:05		855	-	-

Table 24: Comparison of heavy tar measurements in gas after scrubber (Plant B)

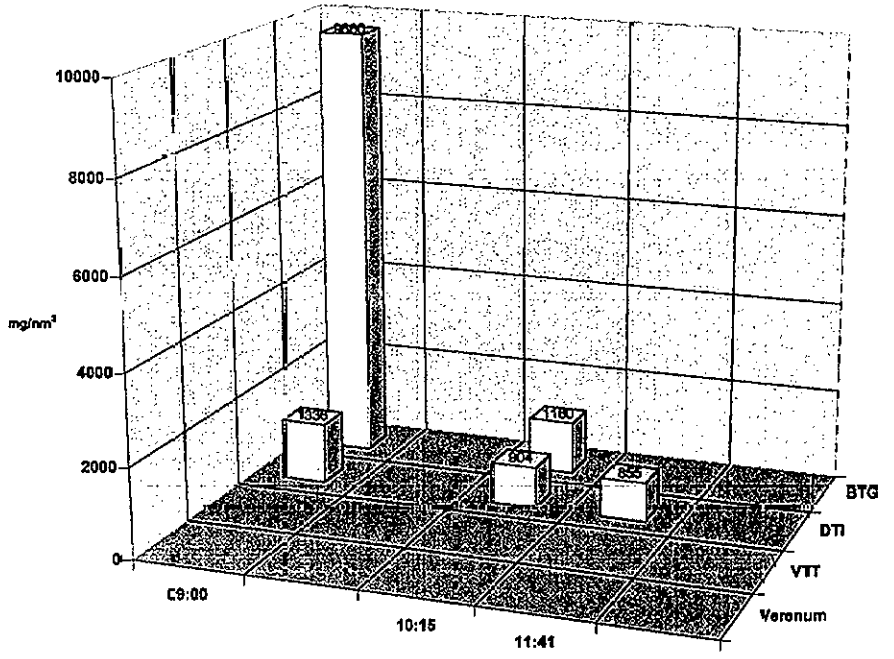

Figure 11: Heavy tar in clean gas (Plant B)

VTT did not measure heavy tar as it was not in their quotation to DEA. Verenum did not carry out measurements due to lack of time. Therefore, the basis of comparison is rather weak. However, it is obvious that the diversity of BTG's measurement results is too great although an increased CH_4 concentration in gas has been observed between 9.15 and 9.30 (see figure 16). An increased CH_4 concentration indicates an increased tar content.

3.2.8 Measurements of light tar in gas after gas scrubber (Plant B)

Measurement period 06.11.98	BTG mg/Nm³	DTI mg/Nm³	Verenum mg/Nm³	VTT mg/Nm³
09:00-09:20	1510	3572	-	4714
09:32-09:44			-	5917
10:15-10:43	220	2679	-	4290
11:41-12:05		2725	-	3482
12:09-12:20			-	4628

Table 25: Comparison of light tar measurements in gas after scrubber (Plant B)

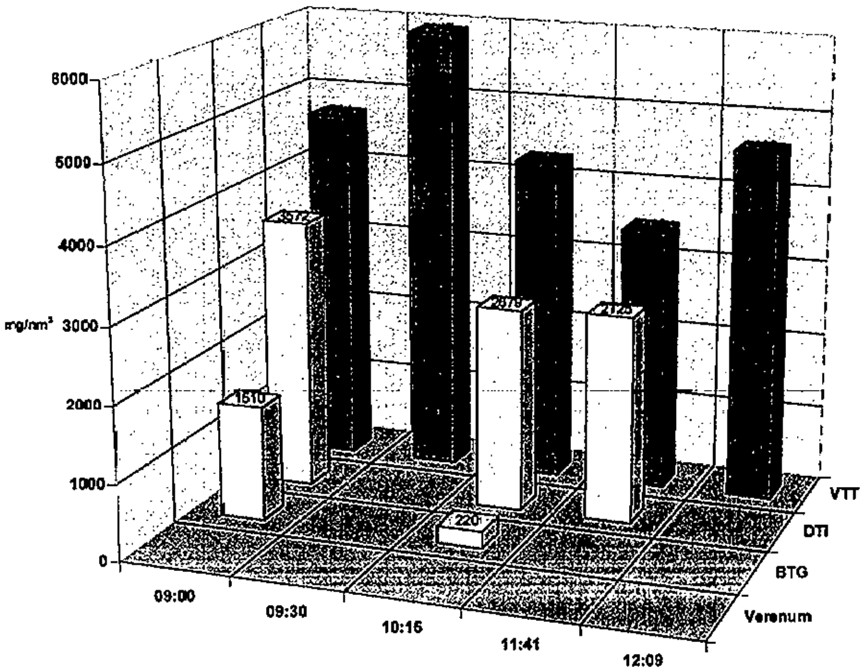

Figure 12: Light tar in clean gas (Plant B)

As previously, BTG's results are calculated as the difference between total tar and heavy tar. The other results are based on GC analyses. The values of DTI and VTT show tar components with a molar mass from 78 till 202. Verenum does not measure in the clean gas.

DTI's results are again too small due to lack of benzene.

BTG's results are very different from the others and once again there is considerable diversification. Since all participants have used different definitions and analysis methods it is not possible to compare the results from light tar measurements.

3.2.9 Comparison of specific tar components in clean gas (Plant B)

The following specifies the results of selected individual components determined by a GC analysis carried out by 2 of the participating laboratories. The selected measurements were carried out simultaneously. The thick line in table 26 indicates the separation line between light tar and heavy tar according to VTT's definition. Components below the broken line are PAHs.

Tar component	DTI No. 10 mg/Nm^3	BTG No. 8 aromatics and phenols mg/sample	VTT No. 1 mg/Nm^3
Benzene	448	1.88	2598
Toluene	108	3.24	372
Phenol	15	19.06	12
Xylene	48	5.06	41
Indene	379	32.47	190
Naphthalene	546	114.17	727
Acenaphthylene	198	36.47	245
Acenaphthene	9	1.09	9
Fluorene	41	5.04	38
Phenanthrene	63	10.05	72
Anthracene	20	2.45	16
Fluoranthene	22	5.71	34
Pyrene	21	10.87	43
Benz(a)anthracene	4.6		
Chrysene	4.9		
Benz(b+k)fluoranthene	7.4		
Benz(a)pyrene	5.0		
Indeno(1,2,3)pyrene	3.2		
Benz(ghi)perylene	2.9		
Dibenz(ah)anthracene	0.2		
Total PAH	948		1184

Table 26: Comparison of specific tar components in clean gas (Plant B)

From table 26 it appears that DTI again found too little benzene. In addition, there is good agreement in connection with PAH.

BTG has reported values of aromatics and phenols as concentration in various extracts. These values can be compared relatively with the results from the other laboratories. Comparison takes place for BTG measurement No. 8, which is carried out simultaneously with the two other measurements in table 26.

BTG's values stem from the sum of extracts from test run 8 and cotton wool 8. The tendency in the values can be compared with the other results (app. factor 10). However, the deviation in connection with benzene, toluene and phenol is pronounced.

3.3 Gas composition during measurement periods

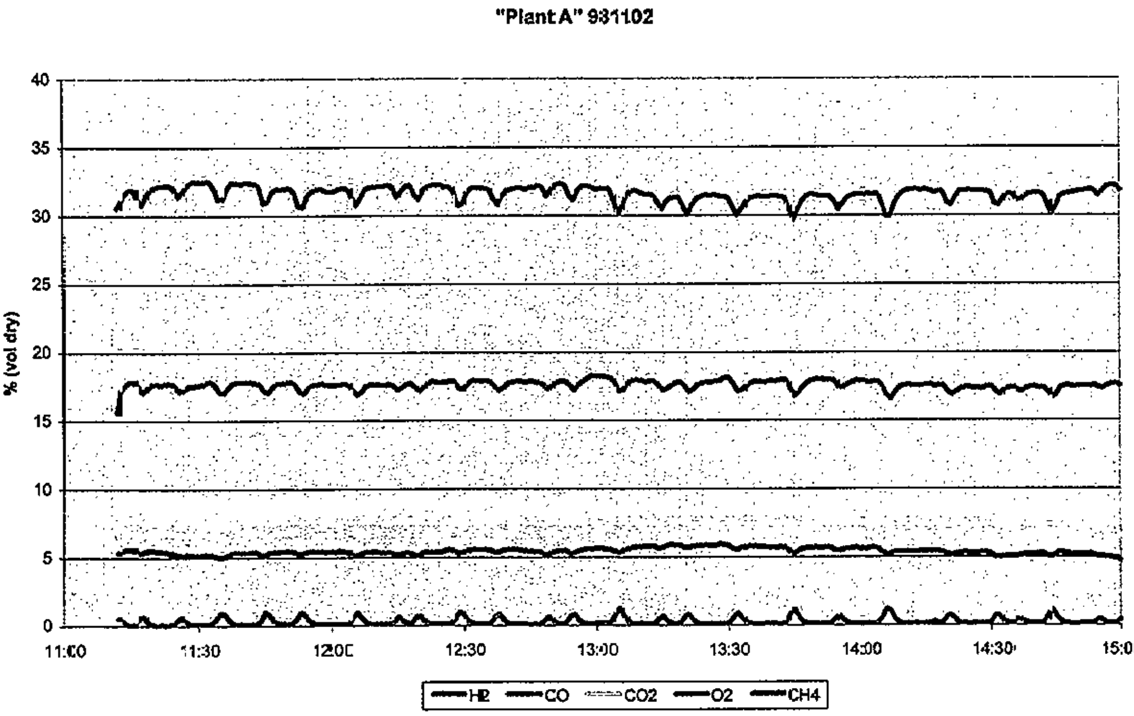

Figure 13: Gas composition, 2nd Nov. 1998, raw gas, "Plant A"

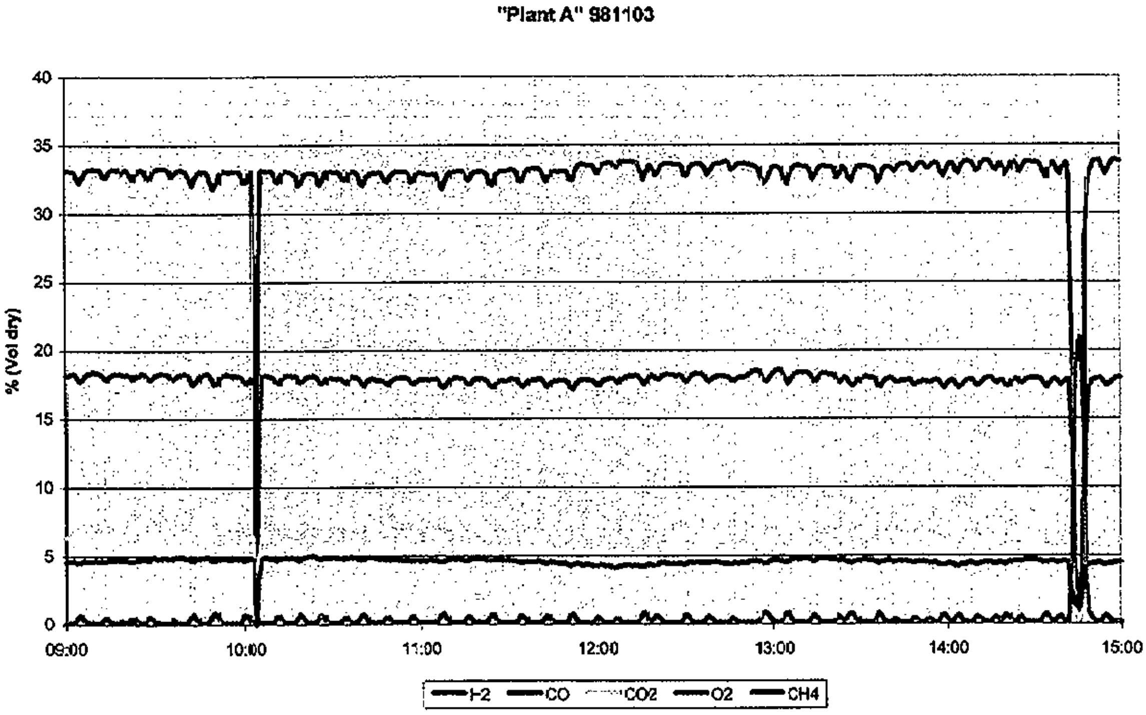

Figure 14: Gas composition, 3rd Nov. 1998, clean gas, "Plant A"

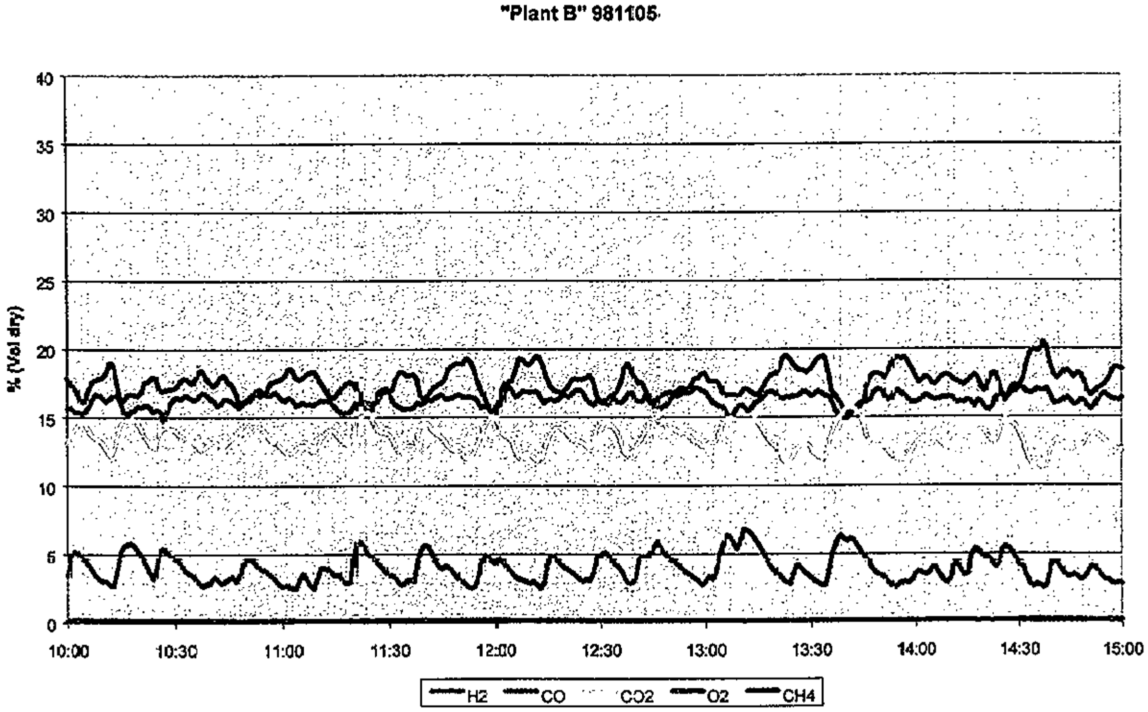

Figure 15: Gas composition, 5th Nov. 1998, raw gas, "Plant B"

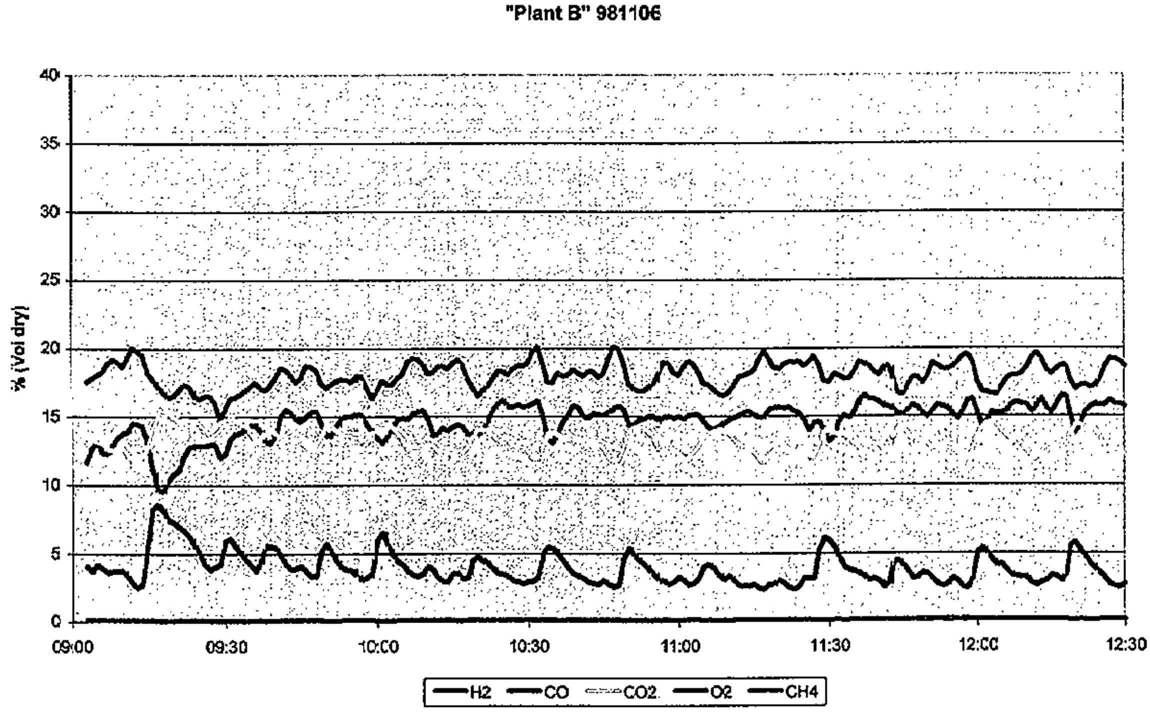

Figure 16: Gas composition, 6th Nov. 1998, clean gas, "Plant B"

4. Financial aspects of the sampling methods

In the following, the financial aspects of the sampling methods are compared. The following parameters are included in the cost analysis:

- Investment costs for sampling equipment
- Operating costs – preparations and sampling
- Cost of preparation of samples and chemical analyses
- Cost of consumables

4.1 Sampling equipment

	BTG Euro	DTI Euro	Verenum Euro	VTT Euro
Heated probe with nozzles, filter housing and valve	2000	1155	1041	1000
Temperature regulator	100	538		300
Condenser, impinger frits and glass equipment – rack	700	525	191	1000
Pump protection equipment		140	53	
Vacuum pump	680	807	1787	500
Flow meter		100	188	100
Volume meter	100	215	188	200
Attached equipment	725	1670	17	200
Total	4305	5150	3465	3300

Table 27: Investment costs for sampling train

The DTI prices are from 1998.
The BTG prices are app. 5 years old.

4.2 Sampling costs

	BTG ** Man-hours	DTI ** Man-hours	Verenum * Man-hours	VTT ** Man-hours
Preliminary preparations		2		1
Installation - plant A	4	3	3.5	1
Installation and calibration of gas analysers		3		
P&T sampling – plant A	2	15	14.5	15
Dismantling – plant A	3	4	3	1
Installation – plant B	4	2.5	2.5	1
Installation and calibration of gas analysers		3		
P&T sampling – plant B	10	12	9	10
Dismantling – plant B	3	4	2.5	1
Postsampling activities	36	1.5	8.5	4
Total man-hours	66	50	43.5	34
Man-hours per sample	7.3	4.2	7.25	1.5

Table 28: Sampling costs

* Samples taken by 1 person (scientist)
** Samples taken by 2 persons (1 scientist and 1 technician)

The hours shown in table 28 are based on the effective time used by the participants during the measurement on both sites. That means that possible waiting time between the measurements has not been included in the time consumption.

4.3 Analysis costs

	BTG Euro/sample	DTI Euro/sample	Verenum Euro/sample	VTT Euro/sample
Preparation of samples, soxhlet, filtration etc.	250	280		Included in the analysis cost
Gravimetic analysis of particulates and heavy tar	100	242	375	200
Analysis of light tar		537	228	140
Total analysis costs per sample	350	1059	603	340

Table 29: Analysis costs

4.4 Cost of consumables

	BTG Euro/sample	DTI Euro/sample	Verenum Euro/sample	VTT Euro/sample
Filter materials	10	3	5	3
Solvents	5	25	26	4
Others		2		5
Total cost of consumables	15	30	31	12

Table 30: Cost of consumables

It is not immediately possible to compare the measurement costs – especially as there are great differences in the field of analysis. DTI's analysis price is a little on the large side of the normal costs as extra tests have been carried out.

Besides the mentioned costs a normal task also includes travelling time, board & lodging and time spent on reporting of measurements.

For all methods there is a need to reduce the man-hours and costs for sampling, postsampling and analysis. In that connection, a further development of a reliable "short cut" method might be a step in the right direction. Several laboratories are working at such a development.

5. Conclusion

5.1 Conclusion relating to sampling methods

The applied sampling methods vary a lot and the parallel test has revealed pros and cons of the methods. In general, it can be concluded that no problems have been observed when using glass equipment on the sites. In spite of limited space the samples have been taken without breaking any glass equipment. Due to transparancy and good cleaning properties the application of glass equipment can be recommended.

BTG's sampling train is very robust but it is not transparent except for the glass tube filled with cotton wool. BTG makes the largest samples compared with the other participants. In updraft tar sampling BTG's method had evident problems with tar penetration. According to BTG that was due to their lack of experience with updraft gasifiers. The problem could be solved by increasing the number of condensers in the train. BTG does not use any solvent during sampling. In post sampling procedures on site, BTG uses DCM for rinsing the flexible hoses and for soxhlet extraction. More attention should be given to personal protective measures such as gloves and respiratory protection.

DTI's sampling train mainly consists of glass components built into a rack, which is very easy to use. However, the stainless steel probe is too long and too heavy in use and should be further developed so it becomes more user friendly. There was no visible aerosol penetration in DTI's sampling train, but nevertheless a small loss of tar has been ascertained. DTI does not use solvents during sampling. For instance, the DTI method is the only method which uses solid adsorbent XAD2. After sampling the sampling train is rinsed with DCM and acetone. The vacuum pump is in operation during rinsing so the operator does not come in contact with solvent fumes. The fumes are led out of the building by means of an exhaust hose. Handling DCM then solely takes place in the laboratory under rigidly controlled conditions.

VTT's use of chilled DCM has turned out to be more effective for sampling volatile tar compounds than solid adsorbents (XAD2). If solid adsorbents have to be used in future, then a combination of e.g. XAD2 and active carbon should be chosen. It is not recommended to use solvents that are hazardous to the health of humans and animals (toxicity etc.), to safety (explosion risk etc.) or to the environment (ODP, smell etc.). If solvents are to be used in future then DCM should be replaced by a substance that is environmentally acceptable. VTT's use of DCM on site takes place under reassuring conditions, as respiratory protection (activ carbon) and rubber gloves are used. In connection with all measurements VTT has taken the smallest samples. In general, the total sampled matter increases the higher the sampled volume is.

The parallel measurements have also shown that there is a need for an unambiguous definition of the notions "tar", "heavy tar", "light tar" and "particulates". Furthermore, a uniform preparation of the samples has to be used and so do uniform conditions of evaporation within the different sampling procedures. Gravimetrical determination of "light tar" as the difference between "total tar" and "heavy tar" does not give results that can be compared with "light tar" determination on the basis of a GC analysis. The test has shown that evaporation procedures for tar determination have to be used very carefully as many parameters influence the result (temperature, pressure, time, moisture content etc.).

The residue of evaporation is influenced by a great number of parameters:

- There is a risk of chemical changes (i.e. polimerisation) during evaporation
- Tar losses are inevitable
- There is an overlap of "light tars" determined by GC

From table 31 the pros and cons of the methods used during parallel test runs in Denmark can be seen.

Evaluation parameter	BTG	DTI	Verenum	VTT
Measurements on updraft gasifier				
Probe size, space requirements	++	– –	++	–
Possibility to mount probe during gasifier operation	– –	++	– –	++
Method used for particulate sampling and analysis	–	+	–	–
Method used for heavy tar sampling and analysis	–	+	–+	++
Method used for PAH sampling and analysis	?	++	+	+
Comparability of particulate concentration	–	+	–	+
Comparability of heavy tar concentration	++	+	++	++
Comparability of PAH concentration	?	++	–	–
Consistency of particulate, heavy tars and PAHdata	+	+	+	+
Measurements on downdraft gasifier				
Method used for particulate sampling and analysis	–	++	–	++
Method used for heavy tar sampling and analysis	+	+	++	++
Method used for PAH sampling and analysis	?	++	++	++
Comparability of particulate concentration	++	++	++	+
Comparability of heavy tar concentration	++	++	++	+
Comparability of PAH concentration	?	++	++	++
Consistency of particulate, heavy tars and PAH data	+	++	++	+
General parameters				
Time requirement for equipment installation/dismantling	–	–	– –	++
Time requirement for on site post sampling	– –	++	– –	+
Use of "good laboratory practices" on site	–	+	+	++
Results reporting (structure, content, discussion, documentation etc.)	+	++	++	+

Table 31: Evaluation of the used methods

Qualitative assessment parameters:

++	good
+	acceptable (minor improvements appreciated)
?	no assessment possible (i.e. not measured, not offered)
–	minor improvement/modification recommended
– –	improvement/modification required

Remarks:

- When viewed for each participant separately, the method used for particulate, heavy tar and PAH sampling is identical.
- Comparable assessment is made with respect to the data of the other methods used (no strong deviations from the others results) or with respect to the "expected" concentration (i.e. as known from literature).
- No assessment for the "light tars" (as discussed in draft final report) is made.
- No assessment of the reproducibilities of the methods is possible (not a target of the investigation)

5.2 Conclusion relating to measurement results

The main result of the particulate measurement is that it to some degree is difficult to distinguish between tar and particulates. A high tar content in the gas often implies measuring errors in particle measurement results. BTG and Verenum measure particulates in updraft gas, which are one order of magnitude higher than DTI and VTT. The results are more uniform in downdraft gas (max. diviation by factor 2).

Various conditions of evaporation have resulted in various data for heavy tar. DTI has measured app. twice as much heavy tar as the other laboratories as evaporation has taken place at room temperature. The other laboratories evaporate at 105 °C. In some cases, especially in updraft gas, comparable heavy tar values were found although various sampling methods, conditions, solvents and analytical procedures were used.

When determining the light tar content of the gas there is generally good agreement in connection with the higher boiling compounds and with PAH. On the other hand, greater deviations are noted with benzene and to a certain extent with toluene. VTT's sampling method is more effective as regards components with low boiling points than DTI's methods. Verenum has not determined the components with low boiling points.

5.3 Recommendations to the future work on a tar protocol

BTG has problems with tar penetration when measurements are carried out in high concentrations. One condenser is obviously not as efficient as required. There were several irregularities during sampling, e.g. leaks from the sampling train, blockage of the pump because of tar and a broken filter cartridge. Furthermore, BTG's results vary a lot and for instance a factor 8 between two successive measurements has been observed. In one case, 7-9 times less condensate was measured than what was found by other participants. Determination of light tar, by means of differential weighing between total tar and heavy tar apparently gives no uniform results. As it appears from table 31 the sampling and analytical procedures have to be improved. After this improvement the BTG measuring method can form part of the future work on the tar protocol.

DTI consistently finds twice as much heavy tar as the other participants. That is because tar evaporation took place at 25 °C, whereas the other participants evaporate at 105 °C. The future measuring method must not overlap between heavy tar and light tar. Therefore, evaporation has to take place at a temperature where overlap is avoided, e.g. 105 °C. In connection with light tar determination, DTI found too little benzene and to some extent too little toluene. That is because XAD-2 is not especially suited for adsorption of volatile components. DTI's measuring method is applicable if the problem with penetration of benzene is solved. That can be achieved by e.g. using active carbon. After minor modifications DTI's measuring method can form part of the future work on the tar protocol.

Verenum's measuring method has a minor shortcoming in connection with separation of particulates and tar. In the impinger train, penetration of tar aerosols has also been observed. The problem can be solved e.g. by analysing the connected active carbon. Verenum's tar analysis only includes part of the light tar components and therefore the results are in general lower than the results of DTI and VTT. In addition, there were problems connected with finding naphthalene, when there are high concentrations of tar substances with corresponding retention time. One advantage of Verenum's measuring method is that DCM is not used.

After minor modifications the Verenum measurement method can form part of the future work on the tar protocol.

The measuring method used by VTT is not sensitive enough to determine particulates. In updraft gas either 0 or 200-400 mg/Nm^3 has been found which is too inaccurate. According to VTT they did not use their standard particulate measurement method, because it was not in their quotation to DEA. Normally, VTT uses a membrane filter for particulate sampling. Penetration of tar aerosols has also been observed in some situations. That should be avoided in the final measuring method by adding more solvent to the impingers or by adjusting the sample rate. In the downdraft gas there are some results that appear unlikely. After minor modifications VTT's measuring method is recommended to form part of the future work on the tar protocol.

In connection with the future measuring method to be described in the tar protocol, it is generally recommended to avoid using substances that are hazardous to health or harmful to the environment.

Other recommendations for the future measuring method:

- Calibrated measuring equipment should be used – which is normal in connection with accreditated measurements.

- A uniform protocol for sampling, postsampling and analysis should be used/developed and appended in the measurement report (such as the Verenum protocols). The aim is:

    1. to ensure a quality standard
    2. to use a comparable experimental basis
    3. to avoid errors/mistakes/misinterpretations of results

APPENDIX 1

Parallel Testing of Tar and Dust Contribution of BTG

BTG, Enschede, the Netherlands

by Harrie Knoef
November 1998

Project No. / *Projectnr.*

355298/2170

Title / *Titel*

Parallell testing of Tar and Dust

Contribution of BTG

Test report
Confidential

Date / *Datum*

July 1999

Prepared for / *Voor*

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TABLE OF CONTENTS

1 INTRODUCTION

Comparable and simultaneous tar and dust measurements have been carried out at two plants in Denmark by four European laboratories using their own method of measurement: BTG (The Netherlands), DTI (Denmark), Verenum (Switzerland) and VTT (Finland).

These parallel measurements were conducted to:
- compare the tar and dust measurement results of the four methods;
- get insight in the applicability of the methods under varying conditions;
- get insight in the costs of the different methods;
- reach consensus on one measuring protocol for tar and dust determination in producer gas to be used in future projects and the development of an international standard.

This report contains the results of the tar and particulate determination at two wood gasifier installations according to the BTG method developed in the early 80s in the framework of the UNDP/World Bank monitoring program on small scale biomass gasifier installations. The experiments were carried out by BTG. In Chapter 2 the main features of the BTG method are summarized. In Chapter 3 the results are given. The results are discussed in Chapter 4 and conclusions are given in Chapter 5.

2 CHARACTERISTICS OF THE BTG SAMPLING METHOD

A summary of the main features characterizing the sampling method is described below. Distinction is made between on-site sampling and laboratory analysis.

On site sampling:

- Isokinetic sampling;
- Simple, robust and low cost system;
- Particulate separation by glass fibre filter cartridges at temperatures above 100 °C to avoid water condensation;
- tar separation through condensation at approx. 2 °C without organic solvents in a 5 liter RVS condenser. This temperature is selected to simulate the real situation where the producer gas / air mixture enters the engine at nearly ambient conditions;
- Not sensitive to sampling duration and/or contaminant level in the product gas;
- On site preparation of the samples that will be transported to a laboratory;

Laboratory analyses:

- gravimetric determination of particulate matter in the filter;
- gravimetric determination of total tar amount in the sample by drying at room temperature;
- Optional: gravimetric determination of heavy tar by drying at 105 °C;
- Optional: GC analyses determining the tar components.

3 RESULTS

3.1 Experimental program

The experiments are done at two plants, to be named plant 1 and plant 2. An overview of the experiments conducted is given below.

Plant 1: Raw gas side

At plant 1 gas was sampled from a bypass of the main producer gas line. The raw gas sample point, used by BTG, was located at a vertical bend of the duct where the producer gas flow was downstream towards the wet venturi scrubber. Temperature at this sample point was about 75 °C.

Plant 1: Clean gas side

At the clean gas side the gas temperature was about 45 °C. This time the sampling nozzle of BTG was located in a horizontal bend of the clean gas part of the duct.

Plant 2: Raw gas side

At plant 2 the experiments were carried out at an horizontal bend in the main producer gas line. Because of the high temperatures (up to 600 °C according to the plant manager) at the raw gas side, some adjustments had to be made for the equipment. The major adjustment was the introduction of a 1 m cooling tube with a diameter of 0.5" directly behind the sampling nozzle. The adjustment made it possible to cool the gas to a temperature of about 120 °C and worked as a safeguard for the teflon lined valve of the filter house.

During the measurements at plant 2, a second condenser vessel was used, to prevent the entrainment of tar. Additionally, a glass tube with cotton wool was placed to visualize the entrainment of dust or tar (smoke). This cotton wool filter, which does not belong to the standard BTG-tar and dust sampling equipment was not used during the measurements at plant 1. The tar left on the cotton wool was included in the measurement by refluxing it with DCM in the Soxhlet apparatus. Moreover, before and after the measurements the weight of the cotton wool was measured.

Plant 2: Clean gas side

At the clean gas side, the sample point was located at a vertical upstream part of the clean producer gas line. Before the clean gas measurement, the filter house vessel was heated up before the experiment to a temperature of 120 °C.

It should be mentioned that in the morning of 6 November, a disturbance in the ash removal system of the cyclone caused a shut down of the engine. The measurement of tar and dust particles was enabled by operating the gasifier at the blower and the flare. Higher dust content could therefore be expected.

Overview of measurements

In Table 1 an overview is given of the measurements carried out at plant 1 and plant 2.

Table 1: Overview of tar and dust measurements carried out by BTG in Denmark.

No.	Site	Sampling	Date	Start	Stop	Sampled volume [Nm³]	Flow [Nm³/hr]
1	1	raw gas	2-11-'98	10:05	10:33	1.56	2.47
2	1	raw gas	2-11-'98	11:44	12:18	1.63	2.88
3	1	raw gas	2-11-'98	14:17	14:48	1.14	2.22
4	1	clean gas	3-11-'98	9:24	10:17	0.9	1.02
5	1	clean gas	3-11-'98	11:47	12:38	2.01	2.36
6	2	raw gas	5-11-'98	10:17	11:33	1.62	1.28
7	2	raw gas	5-11-'98	14:33	15:07	0.78	1.37
8	2	clean gas	6-11-'98	9:02	9:22	0.82	2.47
9	2	clean gas	6-11-'98	10:18	10:46	1.04	2.22

Notes:

1. the gasflow is adjusted to isokinetic conditions as closely as possible. However, during some runs, the flow was decreased due to extremely high tar levels.
2. the nozzle size used at plant 1 was 16 mm and 12 mm aty plant 2.

3.2 Dust measurement

In Table 2, the measuring results are tabulated for the 9 experiments. The dust content at the raw gas side of plant 1 is very high.

If dust collection efficiency is based on mass, the dust collection efficiency of the wet scrubber of plant 1 seems to be good (> 93 %). For plant two the collection efficiency is rather low due to operational problems with the cyclone ash removal system.

Table 2: Results of dust measurement.

	weight of filter cartridge			
No.	before [g]	after [g]	dust [g]	dust content [mg/Nm^3]
1	20.1246	28.152	8.0274	5138
2	23.1715	-	-	-
3	22.4833	27.142	4.6587	4070
4	23.2602	23.4154	0.1552	172
5	21.0924	21.7259	0.6335	316
6	22.0364	23.3594	1.323	818
7	22.0251	22.6972	0.6721	864
8	19.5245	19.9449	0.4204	510
9	18.263	18.8089	0.5459	527

The 'duplo" measurements 6 and 7 show quite similar results. The same holds for measurement 8 and 9.

Note: during experiment no. 2, the filter cartridge was broken.

3.3 Tar measurement

The determination of the total amount of tar was done by the removal of solvent (DCM) through distillation and evaporation (of the DCM traces) to the air at room temperature. The amount of heavy tar was determined by further evaporation of the samples at 105 °C.

Table 3 shows the tar measurement results for the 9 measurements conducted at plant 1 and 2 (see also Table 1). The total amount of tar, heavy tar is given and micrograms per normal cubic meters. Through the condensation method, the water content in the product gas can also be determined. The results in grams per normal cubic meter are also given in Table 3.

Table 3: Tar measurement results

No.	Side	Tar		Heavy tar		Condensate		
		[g]	[mg/Nm^3]	[g]	[mg/Nm^3]	[g]	[g/Nm^3]	[vol%]
1	raw	199	$128*10^3$	80	$51.4*10^3$	810	518	64.2
2	raw	149	$91.2*10^3$	63	$38.6*10^3$	681	418	51.7
3	raw	139	$122*10^3$	54	$46.9*10^3$	456	398	49.3
4	clean	14.8	$16.35*10^3$	2.8	$3.12*10^3$	61	67.1	8.3
5	clean	13	$6.46*10^3$	4.8	$2.40*10^3$	214	106	13.2
6	raw	11.9	$7.36*10^3$	10	$6.31*10^3$	*	*	*
7	raw	8.26	$10.61*10^3$	7.7	$9.88*10^3$	12	15.9	2
8	clean	9.2	$11.17*10^3$	8	$9.66*10^3$	109	132	16.4
9	clean	1.43	$1.38*10^3$	1.2	$1.16*10^3$	138	133	16.5

* The condensate of experiment 6 was left behind at plant 2

During test no. 4 the pump was switched off. It was expected that due to the overpressure of the gasifier installation, iso-kinetic conditions were obtained automatically.
The quantity of condensate during test no. 7 was found to be very low. No explaination could be found for this finding.

As expected, the raw gas of the updraft gasifier contained large amounts of tars. The reproducability is quite acceptable. The raw gas of the downdraft gasifier contains about 10 g/Nm^3. Due to the operational problems, strange and unreliable data were found at the clean gas during the last testrun.
Comparing the relative figures of 'tars' and 'heavy tars' it can be concluded that the downdraft gasifier (exp. 6-9) contains relatively more heavy tars than the updraft gasifier (exp. 1-5). This can be expected from the higher gasification temperatures.

3.4 Remarks on gravimetric tar determination.

A remarkable difference was found in the gravimetric tar determination and in particularly on the evaporation of DCM between tar samples of plant 1 and plant 2.
In the first part of this paragraph the determination of the total amount of tar and the heavy tars in sample 5 while in the second part the determination in sample 1-3 is analysed in more detail. For more detailed tar measurement results one should consider Appendix B.

Determination of total amount of tar at room temperature: sample 5, (plant 1, clean gas)

As an example, the measured weight of sample 5 is given in Figure 1 as function of time during the evaporation of the remaining DCM after distillation. It can be seen that initially DCM evaporates rapidly. Later on the weight loss as a function of time decreases and the evaporation rate of light aromatics drops to a very low level. The total amount of tar present in the sample at room temperature can be calculated from the intersection of the trend lines shown in Figure 1.

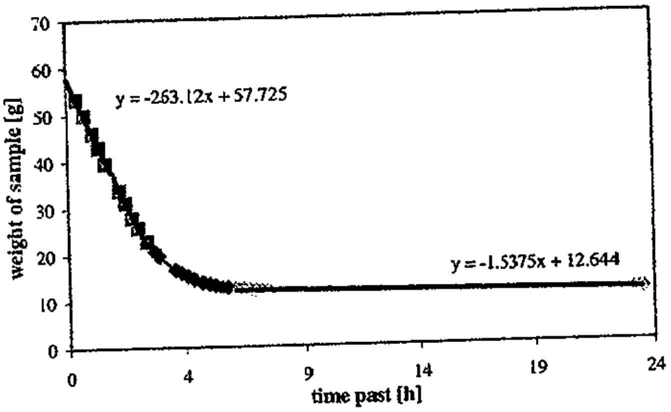

Figure 1: Weight loss of sample 5 as a function of time at room temperature.

Determination of total amount of heavy tar at 105 °C: sample 5, (plant 1, clean gas)

After determination of the total amount of tar at room temperature, the sample is placed in an oven at 105 °C and its weight loss is monitored in time. The total amount of heavy tar can be calculated from the intersection of the trend line and the y-axis, see Figure 2. Figure 2 shows that at the start of the heavy tar determination, the light aromatics evaporate rapidly.

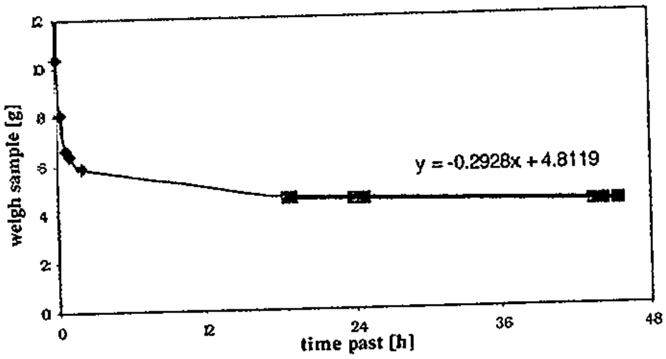

Figure 2: Weight loss of sample 5 as function of time at T = 105 °C.

Determination of total amount of tar at room temperature: sample 1-3 (plant 1, raw gas)

Figure 3 shows the weight loss for sample 1-3 in comparison with sample 5. [Note: the amount of tars for sample 1-3 are only part of the total!, see table 3 for the total]. This illustration shows that the weight loss of sample 1-3 is extremely small. A small film on top of the sample could be observed which might have caused the low evaporation rate of DCM. Therefore, it can be concluded that the determination of the exact amount of tar at ambient temperature present in these particular tar samples is cumbersome, see Appendix B.

The other conclusion is that the method of tar determination depends on the type of tar and thus gasifier design, although this conclusion should be treated carefully while this is the first time that the method was used at this type of gasifier (updraft).

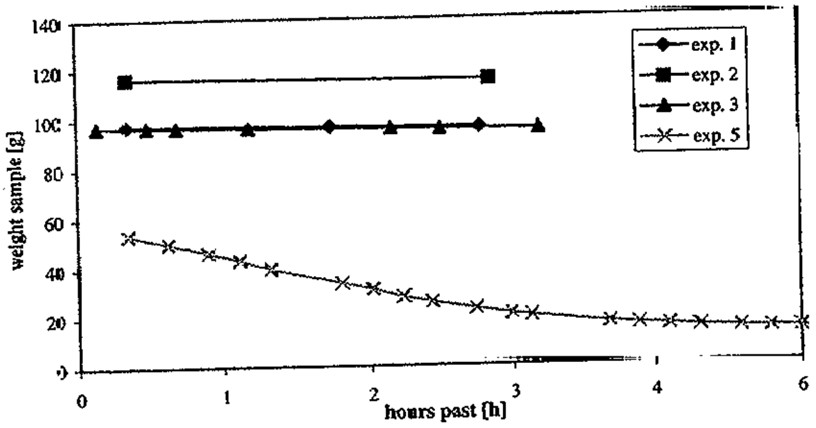

Figure 3: *Weight loss of sample 1-3 as function of time at room temperature.*

3.5 GC measurement of total tars and heavy tars

GC analysis were made of the total tars at ambient conditions and heavy tars after evaporation at 105 °C by using the SPA-method (solid phase adsorption) of some of the experiments. The results are shown in Appendix C which clearly shows that all light aromatics are evaporated after placing the sample in an oven at 105 °C..

4 DISCUSSION

4.1 Gas composition as indication for tar production

To validate the amount of tar measured during a certain period of time, a comparison with gas composition is useful. From the average methane content of the producer gas it can be predicted more or less whether the tar content will be higher or lower compared to a similar measurement. In general, higher tar contents can be validated by higher methane levels.

The Figures A1, A2, A3 and A4 in the Appendices A1 and A2 give an indication of the stability of the gas composition during tar and dust sampling. From these figures it follows, although the updraft gasifier shows a more stable gas composition then the downdraft gasifier, that the gas composition was fairly constant during the time of tar and dust sampling. In fact, only one deviation from the normal gas composition occurred in the morning of 6 November in plant 2 (see Figure A4). At around 9 o'clock that morning, the methane level was almost twice as high as normal. Therefore, tar production must have been higher during measurement no. 8.
The expected higher tar content is confirmed by the results shown in Table 3. The tar level in the clean gas during experiment 8 is almost equal to the level of the raw gas during experiments 6 and 7. Comparing Figure A3 and A4 it is can be assumed that during experiment 9 (taken between 10:18 and 10:46, see table 1) the gas composition was stable again.

From the high methane content and the high tar level during the morning (slightly after 9), it can be concluded that the installation did not function properly. It is not sure whether this was caused by a failure of the ash removal section only or whether some other disturbances in the bed occured. More detailed information is needed on the performance (pressure drops, temperature levels) to analyse the exact reason. For sure is that the engine was shut-down in the night due to malfunctioning of the ash removal system.

4.2 Influence gasifier type and sampling location

Gasification plant 1 is an updraft type gasifier operating on wood chips. In this type of gasifier the wood is dried by the producer gas leaving at the top. The water stripped of from the wood easily condenses in the overhead line, where a three phase flow of solids, water and producer gas is formed. It should be noted that the existence of a changing multi phase regime in the producer gas line may have a strong influence on the amount of tar found by the different methods.

The effect of liquid water is that it will entrain a lot of tar and dust. In process technical point of view the presence of liquid water is an advantage, because it keeps the inner walls of the tube clean by preventing sticking of tars on the wall. However, from tar sampling point of view it is not an advantage, because it complicates the comparison of the amounts of tar measured by the four different methods. In fact, at plant 1 the amount depends on the location of the sample point. For example, it will make difference if the sample point was at the begin or the end of the pipeline and there will be a difference between sampling tar in a vertical or horizontal bend. This should be considered when the absolute figures are compared with eachother.

4.3 Tar and condensate production

Updraft gasifier

The first three experiments at the raw gas side showed excessively high amount of tars and condensates. The amount of tar and condensate at the clean gas side is considerably lower, see experiment 4 and 5.

Downdraft gasifier

This gasifier produces considerably lower amounts of tar and condensates compared to the updraft gasifier, as expected. The heavy tar amount is relatively higher for this gasifier compared to the updraft gasifier. The amount of condensate increases at the clean gas side due to the wet gas cleaning.

4.4 Comparison with other methods

Table 2 and 3 of chapter 3 show the absolute amount of tar and dust sampled during the sample period. It may be useful to compare these amounts with the amounts of tar and dust sampled by the other methods. A first estimate is that absolute amounts sampled with this method are much higher compared to the other methods, resulting in higher accuracies.

One of the items, when comparing the different methods, is the carry over of smoke in the gas leaving the condensor. To visualize smoke BTG uses a glass tube containing a cotton wool filter. Because large amounts of gas are sampled by this method the loss of tar by the carry over of a small amount of smoke will not influence the result significant. Of course, this error should be avoided, for example by installing an additional condensate vessel.

The main difference with the other methods is the fact that no organic solvent is used during sampling which makes it possible to sample over long periods.

4.5 Economics of the sampling method

The economics can be divided in three parts: equipment cost, the cost for one experiment and the labour time. The measurements conducted showed that three complete experiments can be conducted every day on site by two experienced persons, if the equipment is installed.

However, this depends on the sampling duration and hence the tar/dust content. The installation time of the sampling equipment depends on the circumstances at the plant site. In most cases about four hours are sufficient. For post-sampling at the laboratory one experienced person will be busy for one day working on 9 different samples at the same time. The lab assistant can do also work on other assignments parallel to the weighing of the samples. A summary of the economics is given in Table 4.

Table 4: Economical aspects of the tar sampling method.

	Equipment costs (Hfl.)
Metal parts for samples, condenser (RVS)	6000
gas pump	1500
gas meter	250
glassware for Soxtlet extraction	1000
inclined manometer	500
auxiliaries	100
Total equipment costs	**9350**
	Variable costs (Hfl.)
Glass fiber filter per unit	25
Dichloromethane per liter	11
Total variable costs per experiment	**36**
	Labour costs (days work)
On site: six samples per day per two persons	≈1/3
Laboratory: 9 samples per day per person	≈1/9
Labor cost per experiment	1 day

5 CONCLUSIONS

In general it can be concluded that the dust and tar measurement results show the right trend, meaning that higher values were measured at the raw gas side and that the updraft gasifier produces much more tar and dust compared to the downdraft gasifier.

The method can be used for downdraft and updraft gasifiers. Practical problems may arise in case the amount of tar is extremely high, as was measured at the raw gas side of plant 1. In such cases, an additional condenser is required.

The reproducability is quite good and any deviation can be explained and/or correlated to the gas composition, malfunctioning of the gasifier installation, etc.

The determination of the total amount of tar at atmospheric conditions for updraft gasifiers is difficult, because the last DCM present hardly evaporates. In such case, only the heavy tar content determination is reliable.

APPENDIX A1: GC analysis of producer gas at plant 1

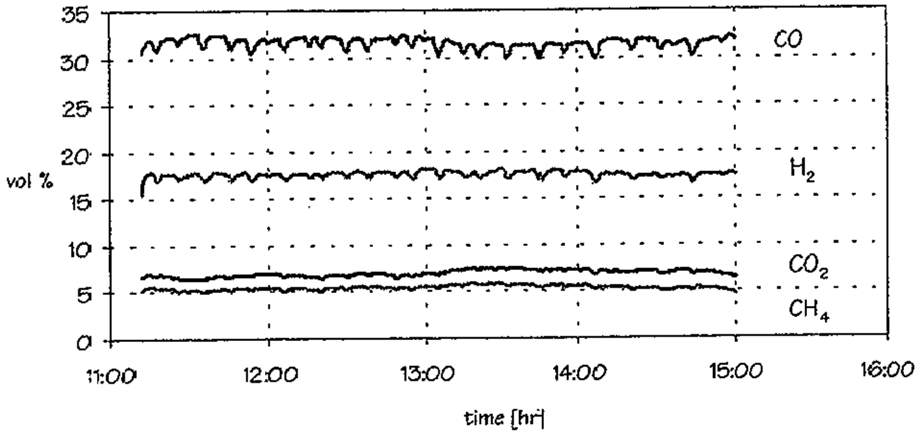

Figure A1: *Producer gas composition of the updraft gasifier at plant 1 on 2 November 1998. Experiment 1-3.*

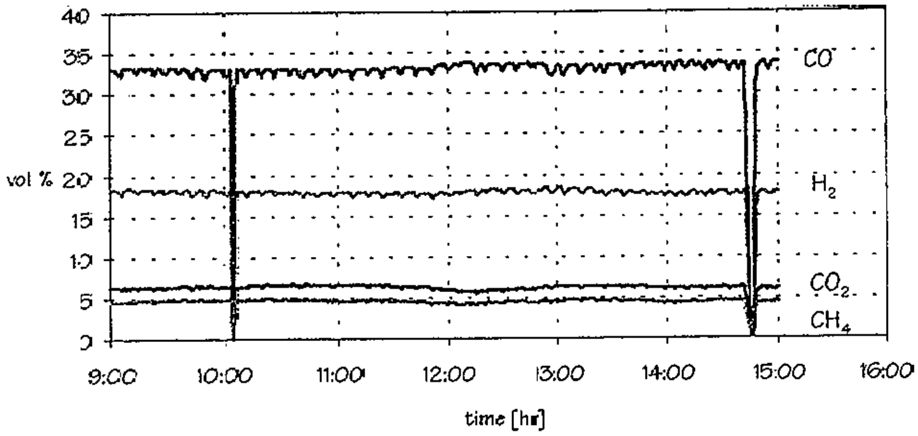

Figure A2: *Producer gas composition of the updraft gasifier at plant 1 on 3 November 1998. Experiment 4 and 5.*

APPENDIX A2: GC analysis of producer gas at plant 2

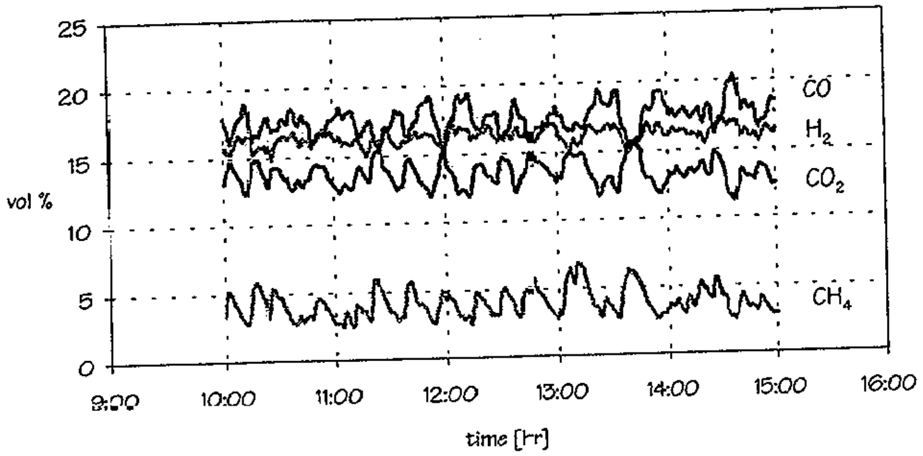

Figure A3: *Producer gas composition of the downdraft gasifier at plant 2 on 5 November 1998. Experiment 6 and 7.*

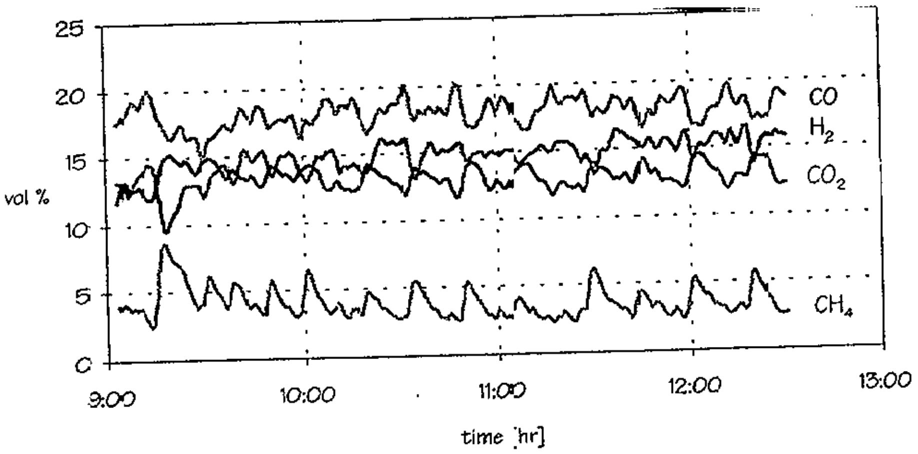

Figure A4: *Producer gas composition of the downdraft gasifier at plant 2 on 6 November 1998. Experiment 8 and 9.*

APPENDIX B: Determination of tar content in producer gas

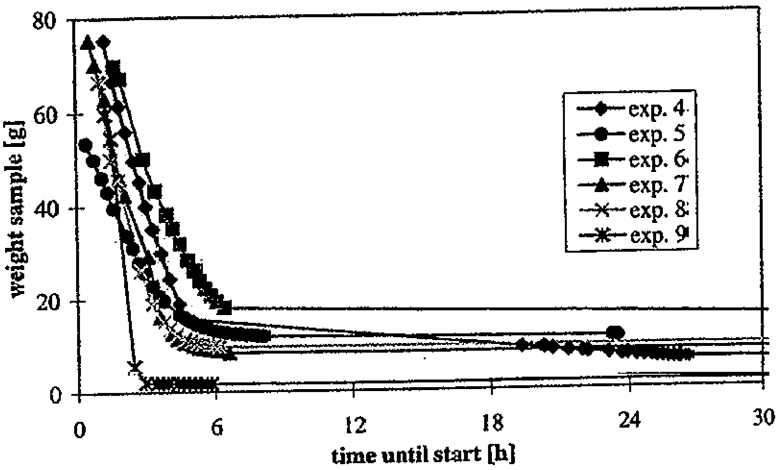

Figure B1: *The decrease in weight of experiment 4 until 9 as function of time at room temperature.*

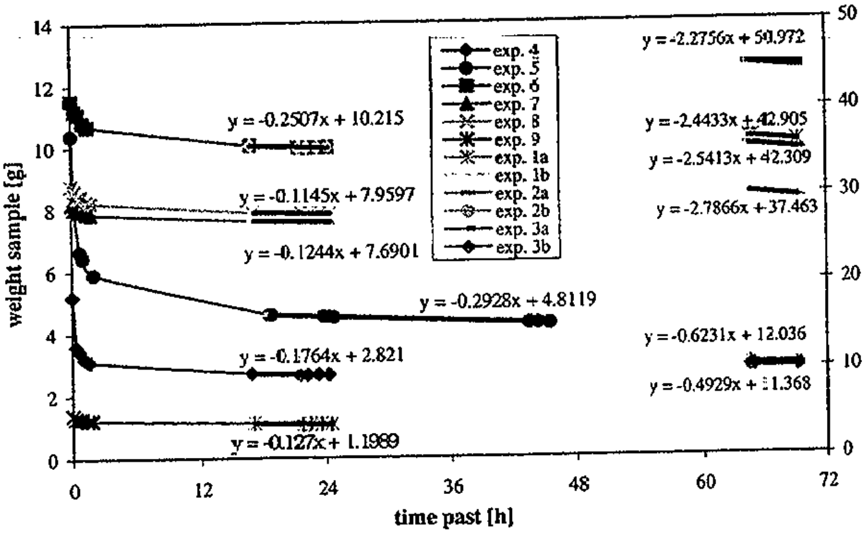

Figure B2: *The decrease in weight of all experiments as function of time at T = 105 °C.*

Figure B.1 shows the evaporation rate of samples 4-9 at ambient conditions. Figure B.2 shows the results of the heavy tars determination. The sample of experiment 1 to 3 is divided over two Petri dishes to increase the amount of surface area for evaporation. The Petri dishes are placed in the oven during one weekend.

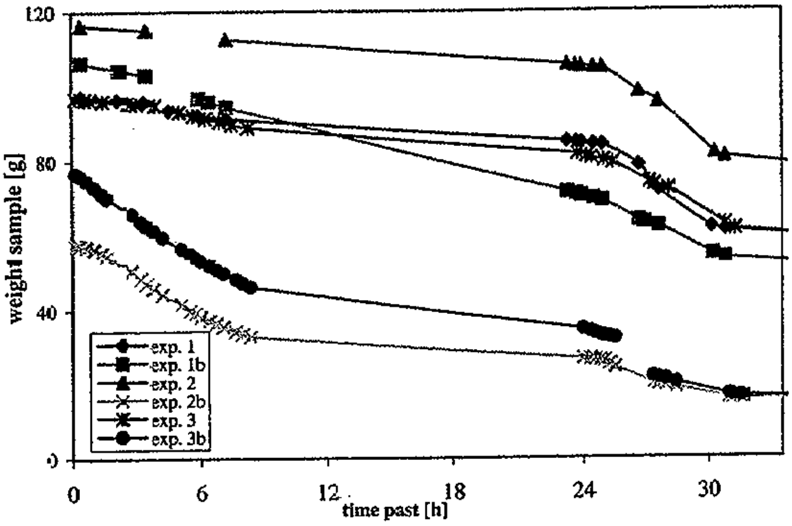

Figure B.3 *The weight changes of the samples of experiment 1 to 3*

The evaporation of DCM of samples 1-3 was extremely slow and therefore the sample was placed in the oven. That particular moment is illustrated by a gap in the line of (for experiment 2b the gap is after 24 hours.) The temperature of the oven was 40 °C between 6 and 24 hours and afterwards 70 °C because at 40 °C the evaporation rate of DCM was still quite low. The fast increase at the start of experiment 2b and 3b is due to the extra amount of DCM that was added due to rinsing of the flask were the sample was transported in.

APPENDIX C: RESULTS OF QUALITATIVE DATA OF TARS

The fraction 'total tars.' were qualitative analysed for their composition on aromatic hydrocarbons and phenols. Some samples were analysed for the aromatic hydrocarbons of the heavy tar samples. Also the cotton wool and condensate were analysed for the aromatics. In the following table, the listing is presented of the samples with the experiments. The reader should check the vial number with the testrun numbers of table 1, 2 and 3.

Vial no.	Analyses	Testrun no.
1	aromatics	6
2	aromatics	7
3	aromatics	8
4	aromatics	9
5	aromatics	3
6	aromatics	4
7	aromatics	5
8	aromatics	8, cotton wool
9	aromatics	4, condensate
10	phenols	6
11	phenols	7
12	phenols	8
13	phenols	9
18	phenols	3
19	phenols	4
20	phenols	5
21	phenols	8, cotton wool
22	phenols	4, condensate
3	aromatics	4, after placing at 105 oC
4	aromatics	5, after placing at 105 oC
5	aromatics	6, after placing at 105 oC
6	aromatics	7, after placing at 105 oC
7	aromatics	8, after placing at 105 oC

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

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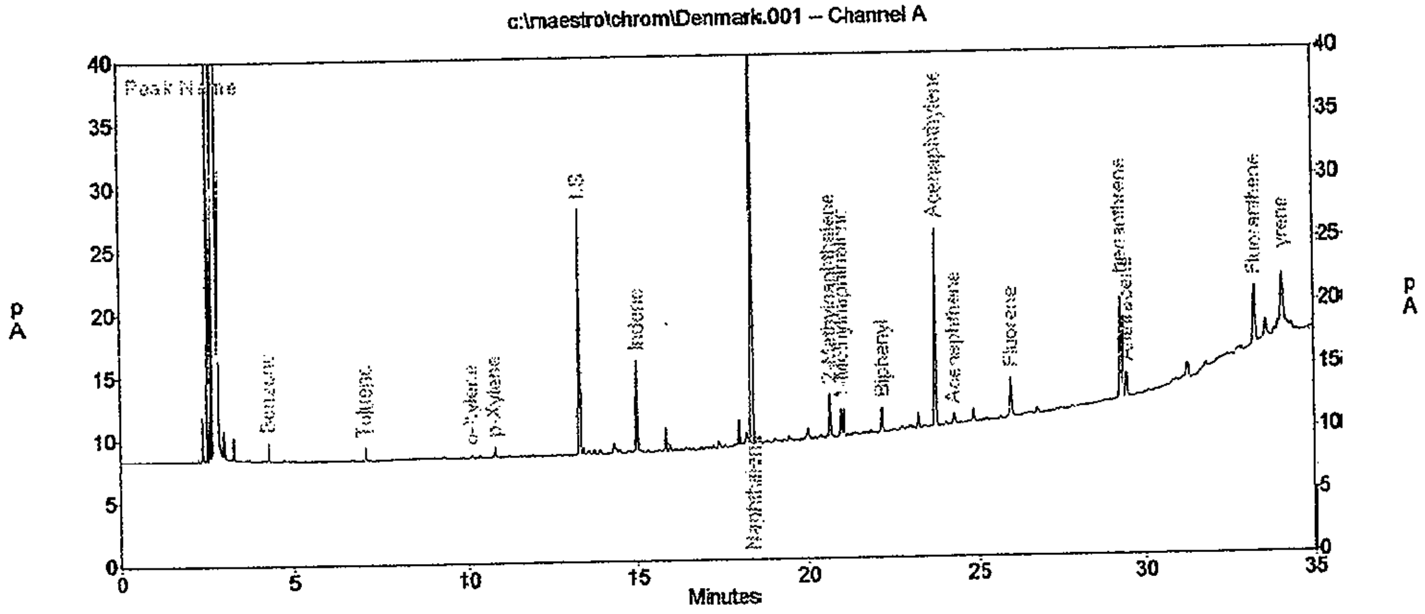
Printed : Dec 03, 1998 16:00:44
User : BertB



Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
6	Benzene	4.20	3398	0.004	1.660
7	Toluene	7.01	2561	0.003	2.149
8	o-Xylene	10.12	651	0.001	0.815
10	p-Xylene	10.81	2219	0.003	1.646
11	I.S.	13.34	50865	0.063	44.065
18	Indene	15.02	20297	0.025	19.748
25	Naphthalene	18.40	181894	0.224	140.257
30	2-Methylnaphthalene	20.67	10818	0.013	7.900
31	1-Methylnaphthalene	21.01	7244	0.009	7.060
32	Biphenyl	22.21	6150	0.008	5.262
36	Acenaphthylene	23.74	54491	0.067	47.912
38	Acenaphthene	24.29	3184	0.004	2.399
40	Fluorene	26.02	10907	0.013	9.066
42	Phenanthrene	29.29	30800	0.038	24.812
43	Anthracene	29.45	7536	0.009	6.423
47	Fluoranthene	33.33	22675	0.028	17.965
49	Pyrene	34.15	45240	0.056	35.894
Totals :			460930	0.569	375.031

TOTAL

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)
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Printed : Dec 03, 1998 16:01:21
User : BertB

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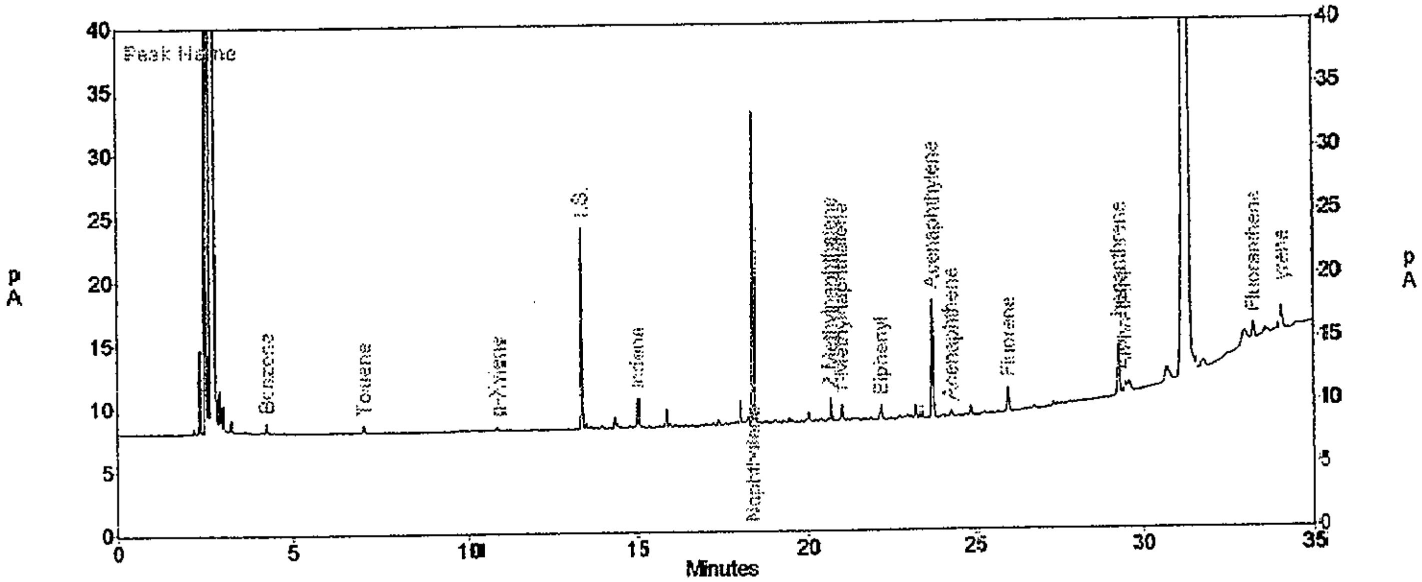

Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
8	Benzene	4.30	1839	0.002	1.029
9	Toluene	7.04	1394	0.002	1.509
--	o-Xylene	10.13	0	0.000	0.000
10	p-Xylene	10.85	519	0.001	0.614
11	I.S.	13.38	39698	0.052	44.065
14	Indene	15.05	5772	0.008	7.713
20	Naphthalene	18.43	74998	0.099	73.923
25	2-Methylnaphthalene	20.68	5240	0.007	5.014
26	1-Methylnaphthalene	21.02	3441	0.005	4.497
27	Biphenyl	22.22	4654	0.006	5.115
29	Acenaphthylene	23.74	33120	0.044	37.341
30	Acenaphthene	24.31	1349	0.002	1.302
32	Fluorene	26.03	6626	0.009	7.057
34	Phenanthrene	29.29	16512	0.022	17.220
35	Anthracene	29.47	4071	0.005	4.417
41	Fluoranthene	33.34	4981	0.007	5.307
42	Pyrene	34.14	9469	0.012	9.782
Totals :			213683	0.281	225.906

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

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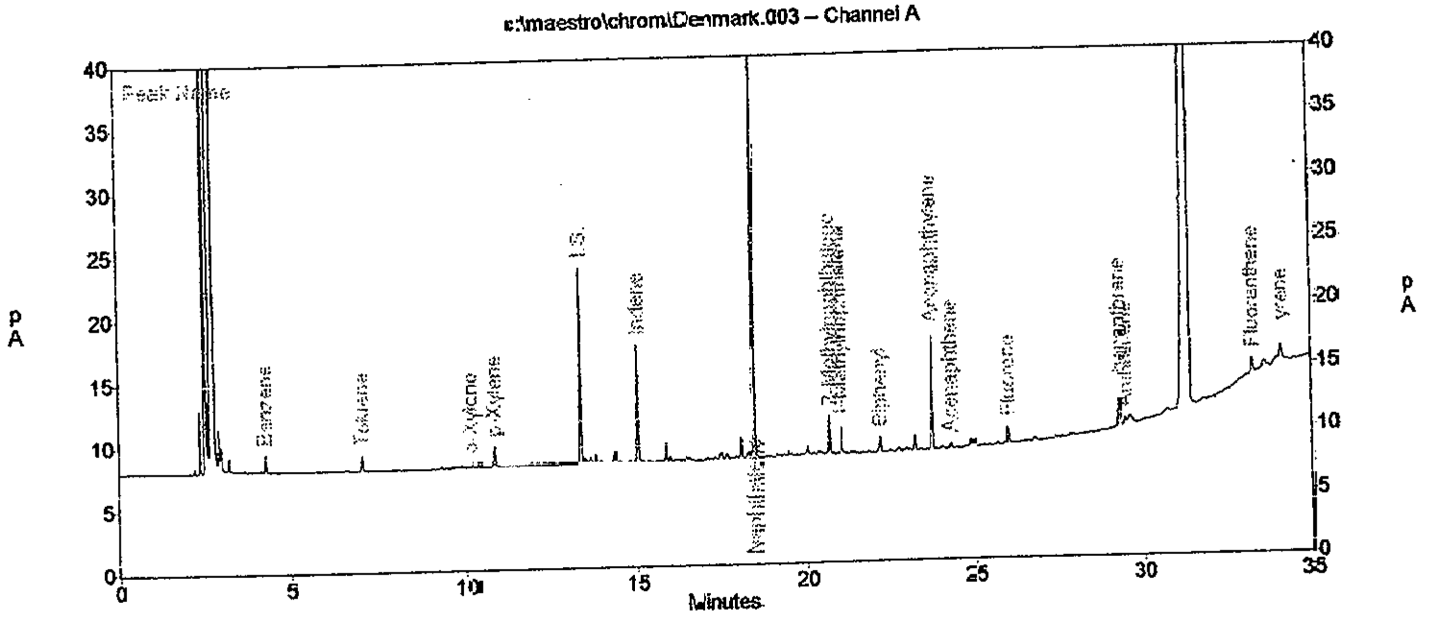
User : BertB



Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
8	Benzene	4.31	2929	0.004	1.880
9	Toluene	7.06	3019	0.004	3.238
10	o-Xylene	10.17	973	0.001	1.227
12	p-Xylene	10.86	4289	0.006	3.833
13	I.S.	13.39	39591	0.057	44.065
19	Indene	15.06	25736	0.037	31.658
28	Naphthalene	18.44	115309	0.167	114.165
31	2-Methylnaphthalene	20.69	9179	0.013	8.585
32	1-Methylnaphthalene	21.04	7677	0.011	9.427
33	Biphenyl	22.22	4376	0.006	4.847
37	Acenaphthylene	23.75	32144	0.047	36.342
38	Acenaphthene	24.31	1125	0.002	1.089
40	Fluorene	26.03	4719	0.007	5.039
42	Phenanthrene	29.29	8826	0.013	9.491
43	Anthracene	29.46	2291	0.003	2.451
46	Fluoranthene	33.32	5368	0.008	5.707
48	Pyrene	34.15	10305	0.015	10.655
Totals :			277856	0.402	293.699

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

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User	: BertB

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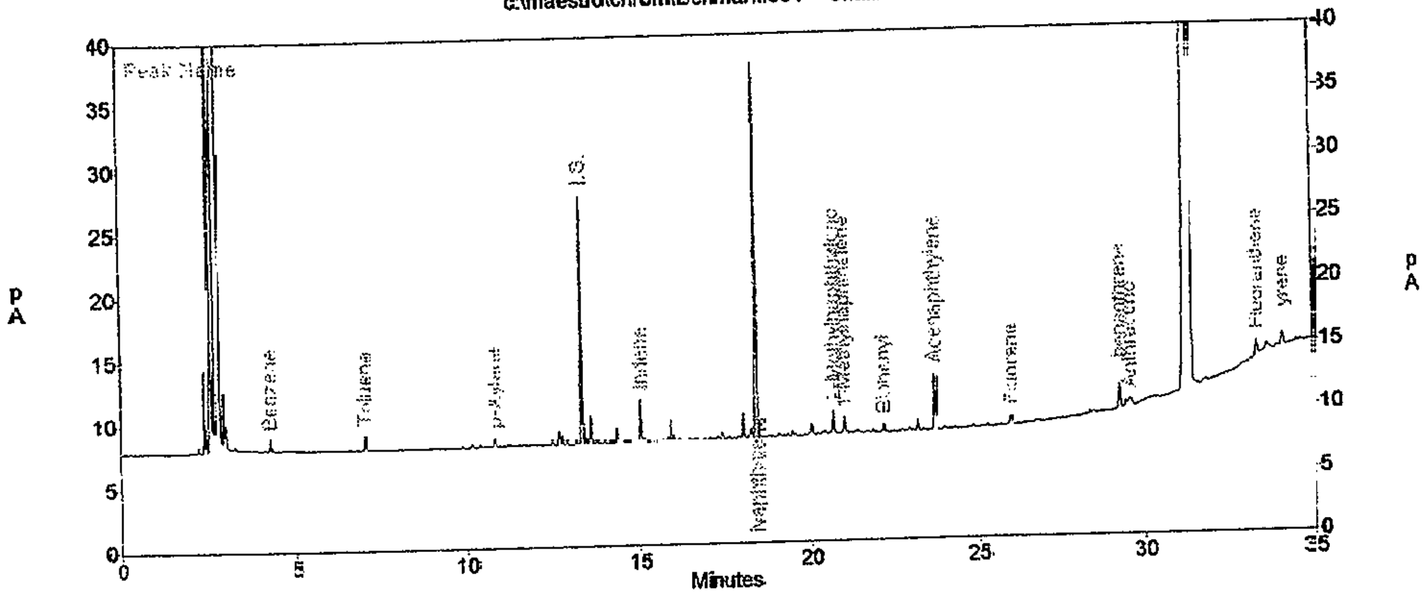

Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
7	Benzene	4.31	1942	0.003	0.789
8	Toluene	7.06	2969	0.004	2.508
--	o-Xylene	10.13	0	0.000	0.000
9	p-Xylene	10.87	1680	0.002	1.298
15	I.S.	13.39	50420	0.070	44.065
21	Indene	15.07	8714	0.012	9.015
30	Naphthalene	18.43	90899	0.127	70.526
36	2-Methylnaphthalene	20.69	5535	0.008	4.220
37	1-Methylnaphthalene	21.03	3242	0.005	3.468
38	Biphenyl	22.22	1890	0.003	1.928
40	Acenaphthylene	23.74	15334	0.021	13.694
--	Acenaphthene	24.25	0	0.000	0.000
42	Fluorene	26.03	2375	0.003	1.991
43	Phenanthrene	29.29	7395	0.010	6.437
44	Anthracene	29.57	6670	0.009	5.725
46	Fluoranthene	33.32	5318	0.007	4.517
48	Pyrene	34.14	4085	0.006	3.464
Totals :			208468	0.291	173.644

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

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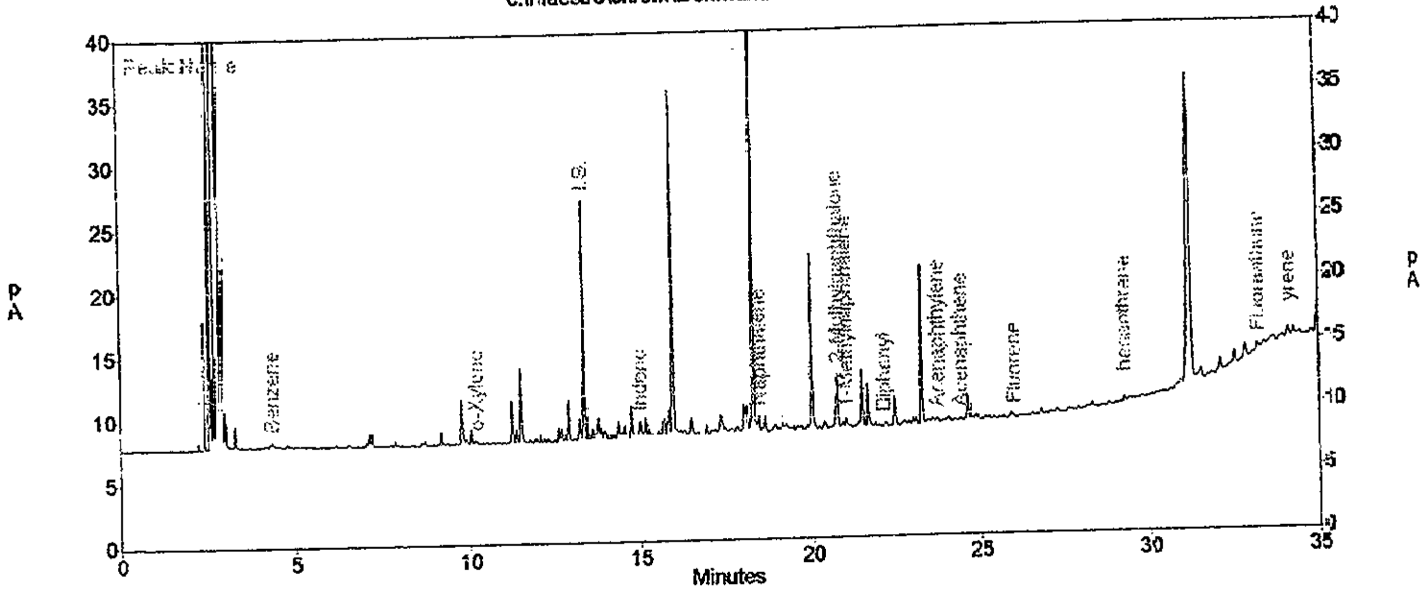
Method	: c:\maestro\methods\8aroma5.met
Sample ID	: Harrie / Denemarke
Vial	: 5
Volume	: 1
Acquired	: Nov 18 1998 17:36:53
Printed	: Dec 03 1998 16:03:13
User	: BertB

c:\maestro\chrom\Denmark.C05 -- Channel A



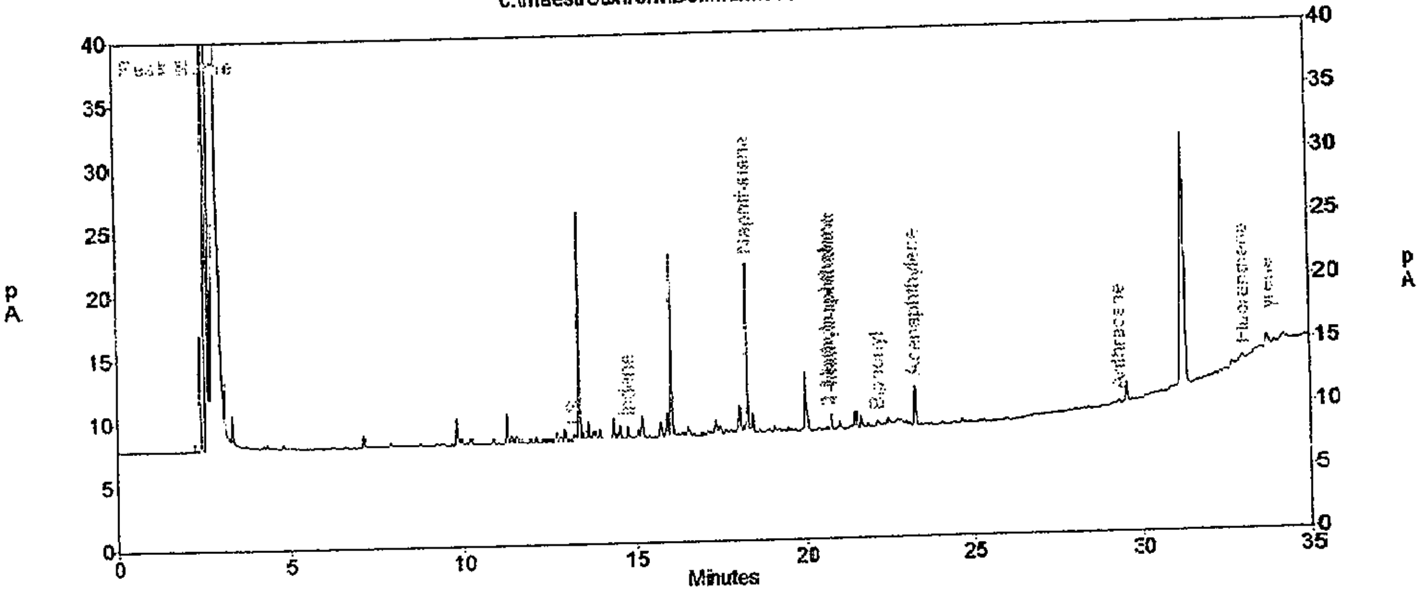
Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
8	Benzene	4.36	1380	0.002	0.448
--	Toluene	6.96	0	0.000	0.000
16	o-Xylene	10.21	1027	0.001	1.081
--	p-Xylene	10.80	0	0.000	0.000
29	I.S.	13.40	50309	0.070	44.065
40	Indene	15.02	4700	0.007	5.247
59	Naphthalene	18.44	6900	0.010	5.023
70	2-Methylnaphthalene	20.76	18823	0.026	13.674
72	1-Methylnaphthalene	21.04	1770	0.002	2.129
76	Biphenyl	22.17	1554	0.002	1.664
83	Acenaphthylene	23.74	808	0.001	0.846
84	Acenaphthene	24.42	517	0.001	0.394
88	Fluorene	26.02	621	0.001	0.522
90	Phenanthrene	29.28	1345	0.002	1.634
--	Anthracene	29.41	0	0.000	0.000
97	Fluoranthene	33.24	2072	0.003	1.976
100	Pyrene	34.19	5527	0.008	4.621
Totals :			97353	0.136	83.324

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

File	: c:\maestro\chrom\Denmark.006
Method	: c:\maestro\methods\3aroma5.met
Sample ID	: Harris / Denemarke
Vial	: 6
Volume	: 1
Acquired	: Nov 18, 1998 18:22:20
Printed	: Dec 03, 1998 16:25:50
User	: BertB

c:\maestro\chrom\Denmark.006 -- Channel A

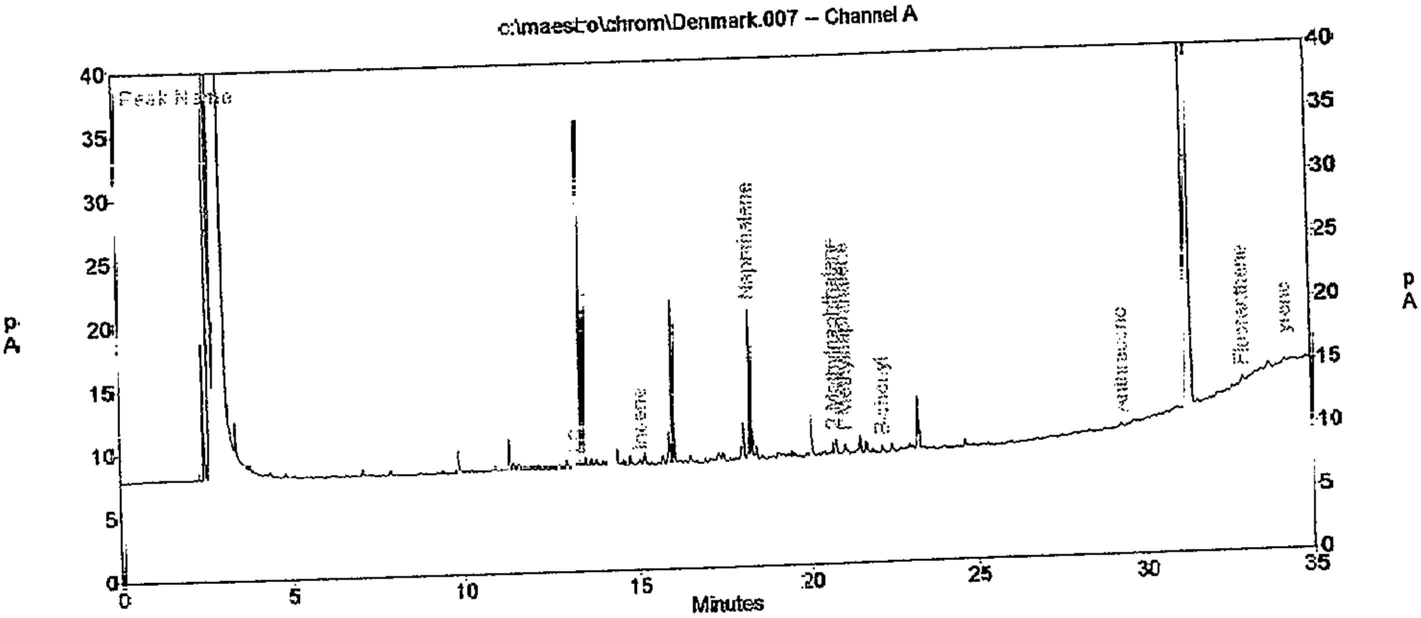


Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample
--	Benzene	4.23	0	0.000	0.000
--	Toluene	6.96	0	0.000	0.000
--	o-Xylene	10.13	0	0.000	0.000
—	p-Xylene	10.80	0	0.000	0.000
24	I.S.	13.28	3939	0.005	44.055
33	Indene	14.81	3246	0.004	39.915
51	Naphthalene	18.29	44038	0.052	435.287
61	2-Methylnaphthalene	20.72	3054	0.004	26.019
62	1-Methylnaphthalene	20.78	3841	0.005	45.344
69	Biphenyl	22.20	917	0.001	9.734
72	Acenaphthylene	23.29	10485	0.012	118.854
--	Acenaphthene	24.25	0	0.000	0.000
--	Fluorene	25.98	0	0.000	0.000
--	Phenanthrene	29.25	0	0.000	0.000
74	Anthracene	29.33	773	0.001	8.538
78	Fluoranthene	32.95	2835	0.003	28.790
79	Pyrene	33.73	4772	0.006	43.815
Totals :			77900	0.092	811.361

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

File	: c:\maestro\chrom\Denmark.007
Method	: c:\maestro\methods\aroma5.met
Sample ID	Harrie / Denemarke
Vial	7
Volume	1
Acquired	: Nov 18, 1998 19:07:40
Printed	: Dec 03, 1998 16:04:28
User	: BertB



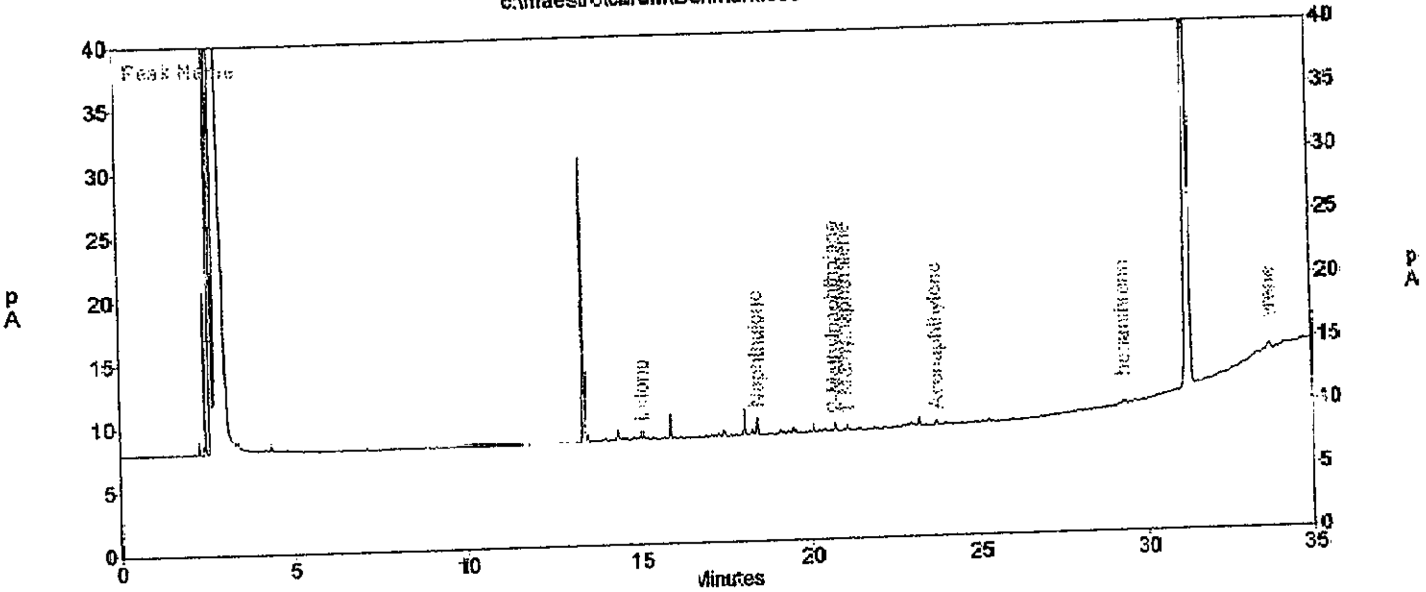
Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
--	Benzene	4.23	0	0.000	0.000
--	Toluene	5.96	0	0.000	0.000
--	o-Xylene	10.13	0	0.000	0.000
--	p-Xylene	10.80	0	0.000	0.000
18	I.S.	13.31	1358	0.001	44.065
27	Indene	15.10	628	0.001	22.756
41	Naphthalene	18.29	39420	0.038	1141.166
51	2-Methylnaphthalene	20.72	5580	0.005	147.229
53	1-Methylnaphthalene	21.06	4376	0.004	148.664
58	Biphenyl	22.19	1918	0.002	56.877
--	Acenaphthylene	23.68	0	0.000	0.000
--	Acenaphthene	24.25	0	0.000	0.000
--	Fluorene	25.98	0	0.000	0.000
--	Phenanthrene	29.25	0	0.000	0.000
64	Anthracene	29.31	703	0.001	22.838
67	Fluoranthene	32.92	1704	0.002	49.934
69	Pyrene	34.25	3139	0.003	94.422
Totals :			58881	0.056	1727.952

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

File	: c:\maestro\chrom\Denmark.008
Method	: c:\maestro\methods\8aroma5.met
Sample ID	: Harrie / Denemarke
Vial	: 8
Volume	: 1
Acquired	: Nov 18, 1998 19:52:51
Printed	: Dec 03, 1998 16:05:04
User	: BertB

c:\maestro\chrom\Denmark.008 -- Channel A



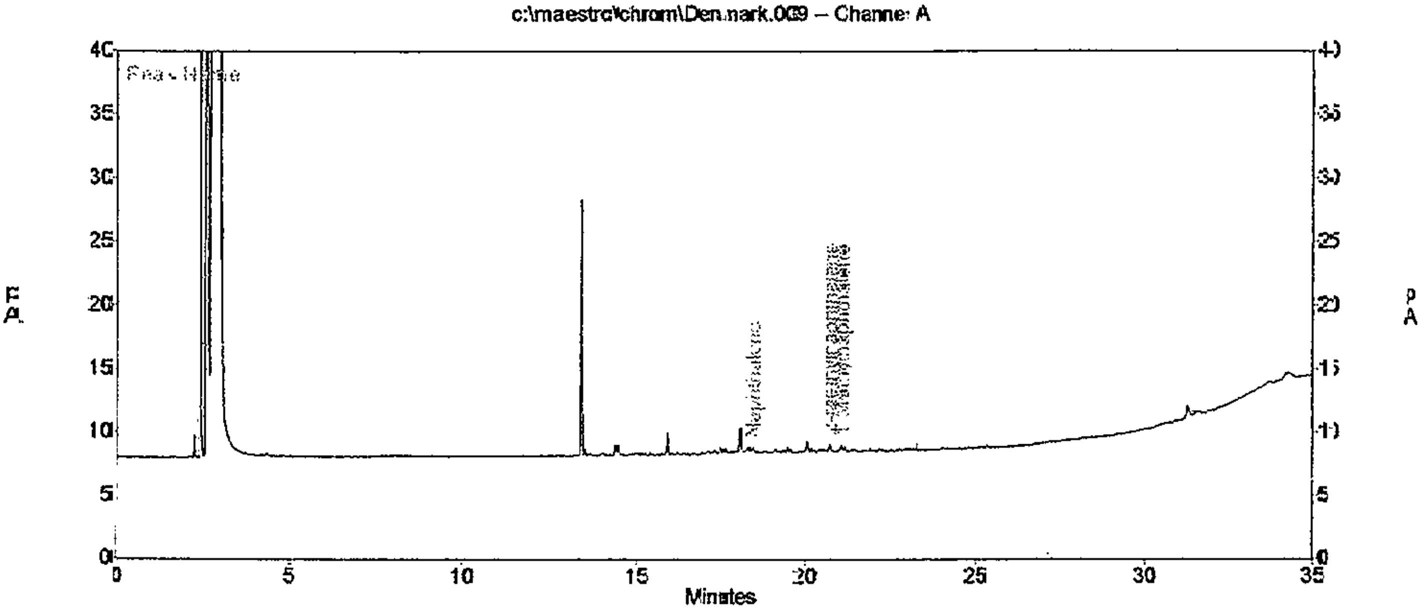
Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
--	Benzene	4.23	0	0.000	0.000
--	Toluene	6.96	0	0.000	0.000
--	o-Xylene	10.13	0	0.000	0.000
--	p-Xylene	10.80	0	0.000	0.000
--	I.S.	13.34	0	0.000	0.000
9	Indene	15.08	1274	0.001	0.814
16	Naphthalene	18.46	4097	0.004	0.000
23	2-Methylnaphthalene	20.71	1974	0.002	0.294
24	1-Methylnaphthalene	21.05	1446	0.001	0.512
--	Biphenyl	22.18	0	0.000	0.000
26	Acenaphthylene	23.76	1168	0.001	0.129
--	Acenaphthene	24.25	0	0.000	0.000
--	Fluorene	25.98	0	0.000	0.000
27	Phenanthrene	29.32	785	0.001	0.563
--	Anthracene	29.11	0	0.000	0.000
--	Fluoranthene	33.29	0	0.000	0.000
29	Pyrene	33.73	3049	0.003	0.213
Totals :			13793	0.014	2.526

BTG Biomass Technology Group BV

Teer monster analyse (aromaten)

```
File       : c:\maestro\chrom\Denmark.009
Method     : c:\maestro\methods\8aroma5.met
Sample ID  : Harrie / Denemarke
Vial       : 9
Volume     : 1
Acquired   : Nov 18, 1998  20:37:54
Printed    : Dec 03, 1998  16:05:39
User       : BertB
```



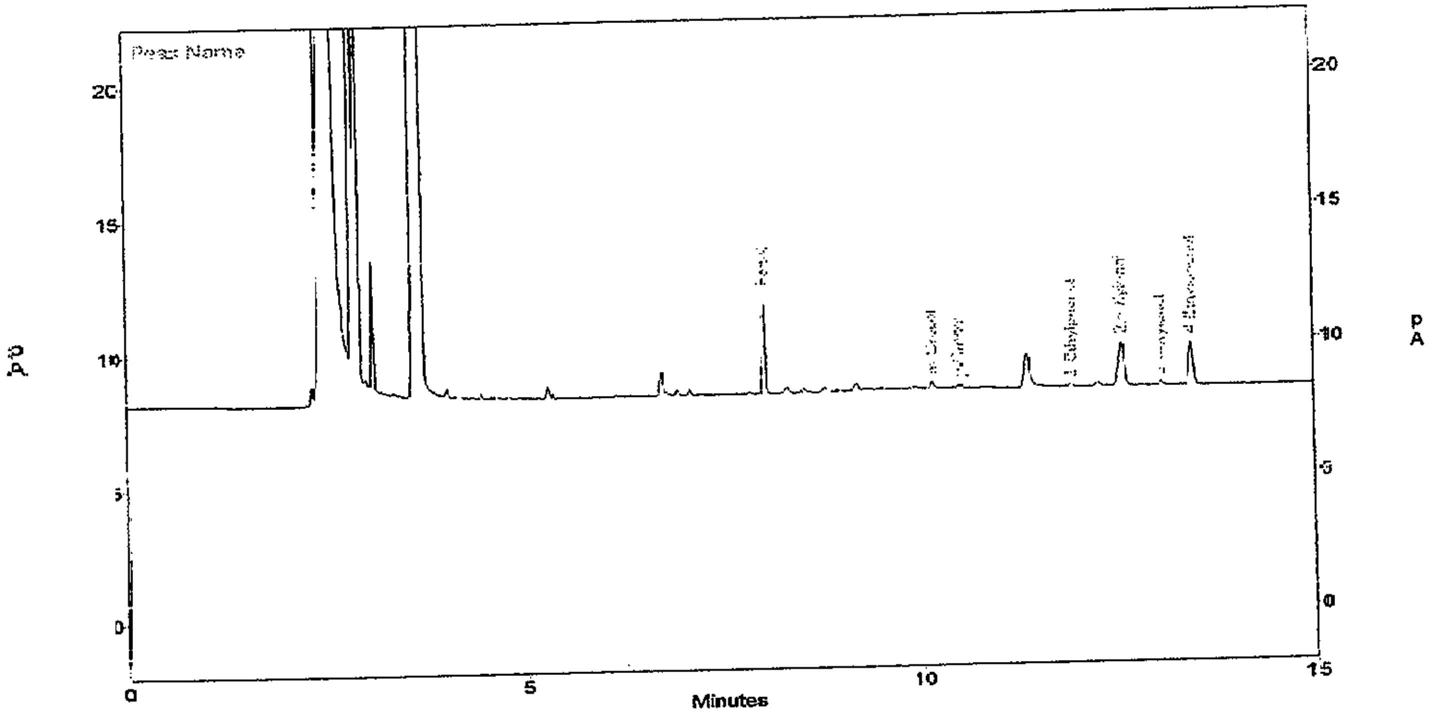
Channel A Results

```
 Peak    Peak Name               Time     Area      Area %     Conc (ug/sample)
 -----   -------------------    ------   --------   --------   ---------------
  --     Benzene                 4.23         0      0.000               0.000
  --     Toluene                 6.96         0      0.000               0.000
  --     o-Xylene               10.13         0      0.000               0.000
  --     p-Xylene               10.80         0      0.000               0.000
  --     I.S.                   13.34         0      0.000               0.000
  --     Indene                 14.99         0      0.000               0.000
  13     Naphthalene            18.45      1039      0.001               0.000
  19     2-Methylnaphthalene    20.71      1852      0.002               0.294
  20     1-Methylnaphthalene    21.05       855      0.001               0.512
  --     Biphenyl               22.18         0      0.000               0.000
  --     Acenaphthylene         23.68         0      0.000               0.000
  --     Acenaphthene           24.25         0      0.000               0.000
  --     Fluorene               25.98         0      0.000               0.000
  --     Phenanthrene           29.25         0      0.000               0.000
  --     Anthracene             29.41         0      0.000               0.000
  --     Fluoranthene           33.29         0      0.000               0.000
  --     Pyrene                 34.10         0      0.000               0.000

Totals :
                                           3746      0.003               0.806
```

BTG Biomass Technology Group BV
Teer monster analyse (fenolen)

File : c:\maestro\chrom\Denmark.010
Method : c:\maestro\methods\10fenol7.met
Sample ID : Harrie / Denemarke
Vial : 10
Volume : 1
Acquired : Nov 18, 1998 21:24:45
Printed : Dec 03, 1998 16:06:15
User : EertB

c:\maestro\chrom\Denmark.010 -- Channel A



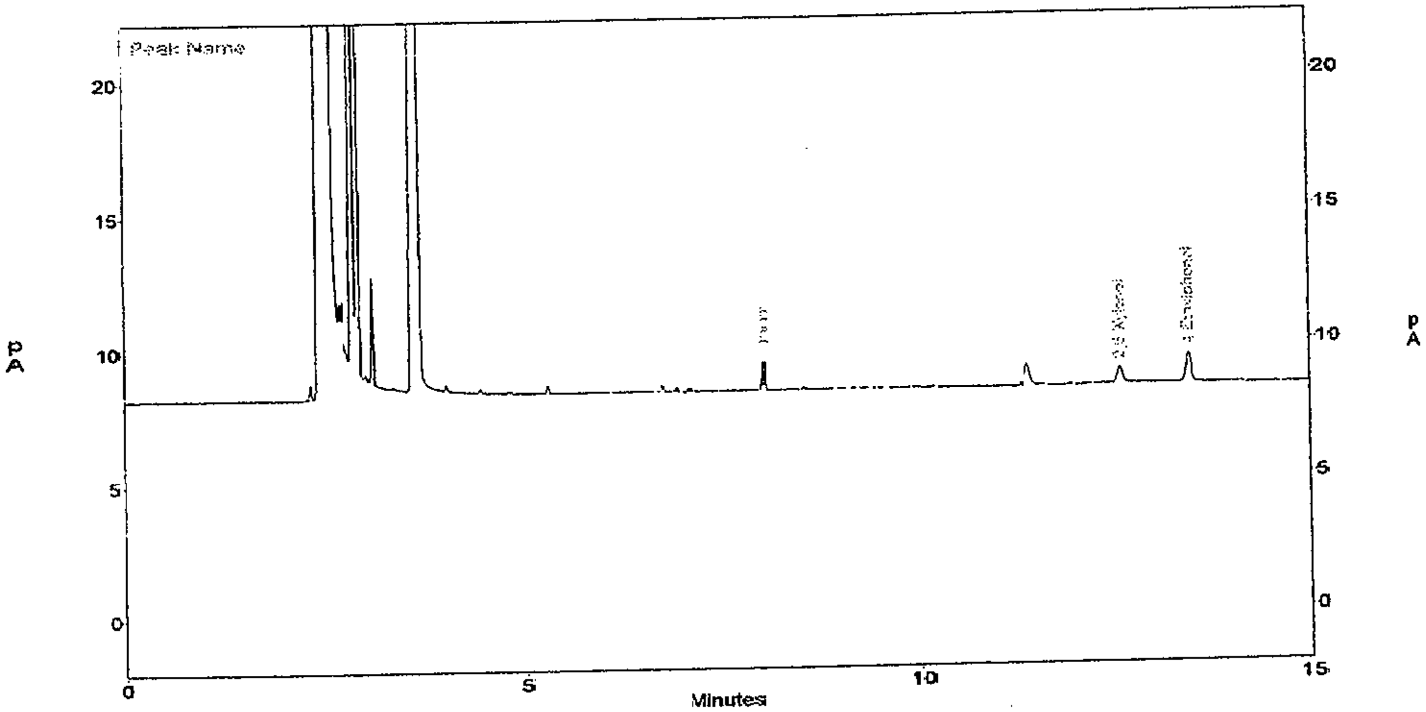
Channel A Results

Peak	Peak name	Time	Area	Area %	Conc. (ug/sample)
5	Phenol	7.99	8479	22.07	38.543
--	o-Cresol	9.85	0	0.00	0.000
11	m-Cresol	10.15	711	1.85	3.962
12	p-Cresol	10.47	313	0.81	1.473
14	2-Ethylphenol	11.89	220	0.57	1.534
16	2,5-Xylenol	12.53	7679	19.99	38.110
17	2,4-Xylenol	13.02	438	1.14	2.859
18	4-Ethylphenol	13.42	6753	17.58	34.390
--	2,6-Xylenol	13.55	0	0.00	0.000
--	2,3-Xylenol	13.91	0	0.00	0.000
--	3,4-Xylenol	14.26	0	0.00	0.000
Totals :			24593	64.02	120.869

BTG Biomass Technology Group BV
Teer monster analyse (fenolen)

```
File       : c:\maestro\chrom\Denmark.011
Method     : c:\maestro\methods\~0fenol7.met
Sample ID  : Harrie / Denemarke
Vial       : 11
Volume     : 1
Acquired   : Nov 18, 1998  22:00:27
Printed    : Dec 03, 1998  16:06 45
User       : BertB
```

c:\maestro\chrom\Denmark.011 -- Channel A

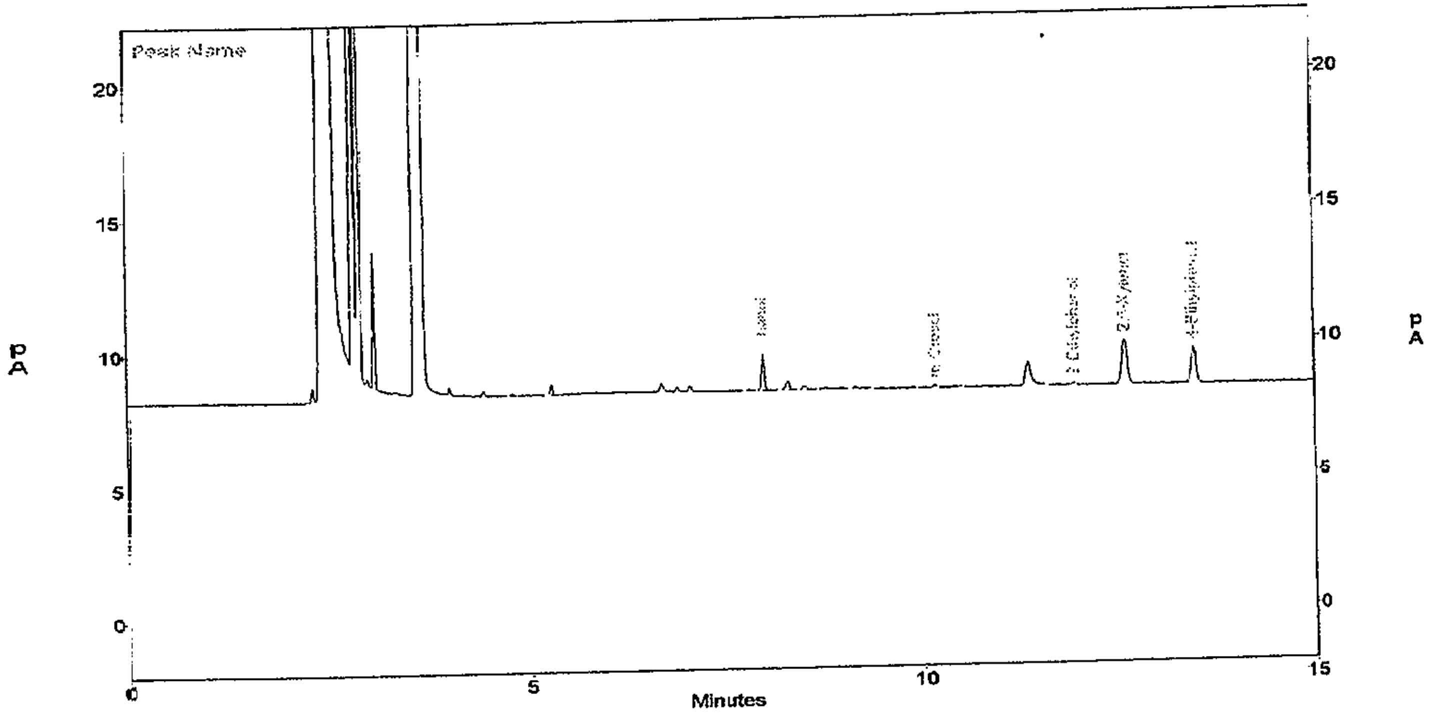


```
Channel A Results

Peak   Peak name                   Time        Area      Area %      Conc. (ug/sample)
-----  ------------------------    ---   --------------------   ----------------
    4  Phenol                       8.01        2750      16.96            19.281
   --  o-Cresol                     9.85           0       0.00             0.000
   --  m-Cresol                    10.05           0       0.00             0.000
   --  p-Cresol                    10.40           0       0.00             0.000
   --  2-Ethylphenol               11.77           0       0.00             0.000
    8  2,5-Xylenol                 12.56        2655      16.37            20.263
   --  2,4-Xylenol                 12.88           0       0.00             0.000
    9  4-Ethylphenol               13.45        4428      27.31            34.390
   --  2,6-Xylenol                 13.55           0       0.00             0.000
   --  2,3-Xylenol                 13.91           0       0.00             0.000
   --  3,4-Xylenol                 14.26           0       0.00             0.000

Totals :
                                                9833      60.64            73.933
```

BTG Biomass Technology Group BV
Teer monster analyse (fenolen)

File : c:\maestro\chrom\Denmark.012
Method : c:\maestro\methods\10fenol7.met
Sample ID : Harrie / Denemarke
Vial : 12
Volume : 1
Acquired : Nov 18, 1998 22:36:20
Printed : Dec 03, 1998 16:07:14
User : BertB

c:\maestro\chrom\Denmark.012 – Channel A

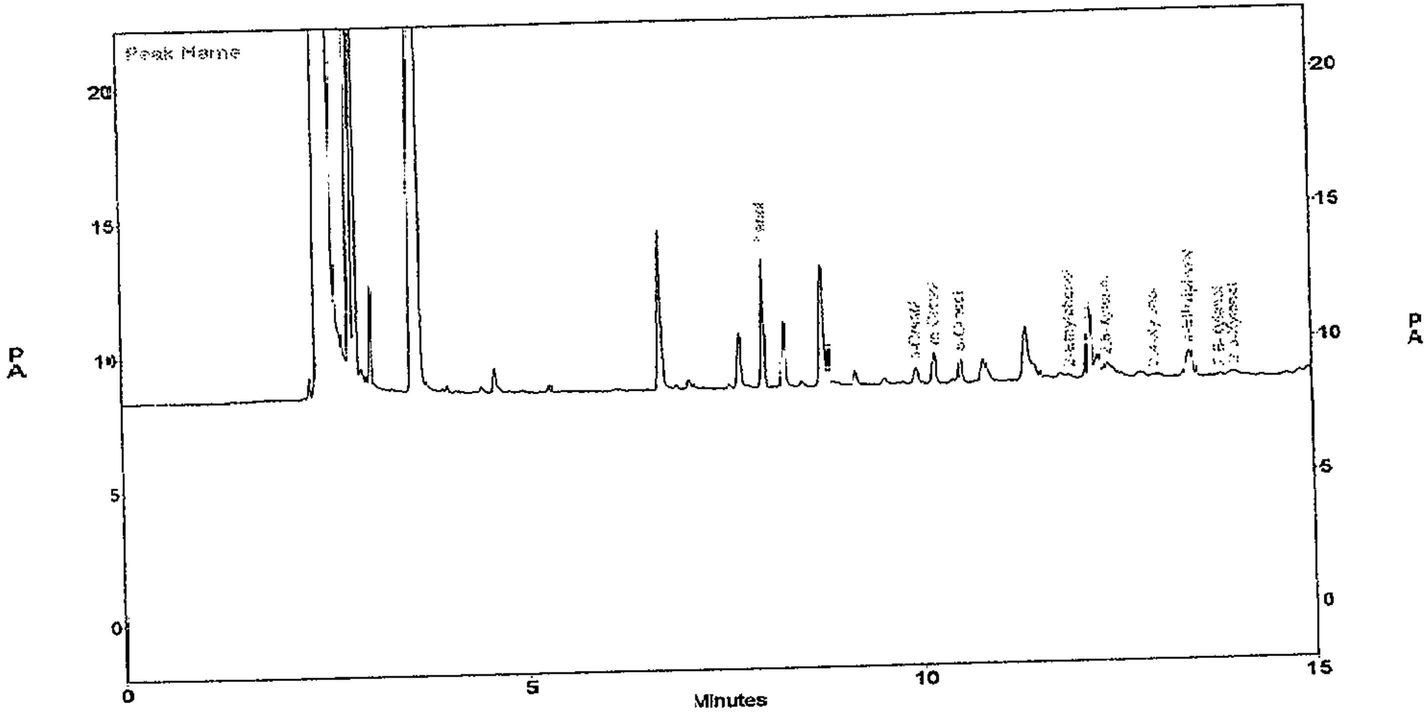


```
Channel A Results

Peak   Peak name                Time         Area       Area %     Conc. (ug/sample)
-----  ---------------------    ----------   --------------------  -----------------
   4   Phenol                        8.00        3511      13.62            19.058
  --   o-Cresol                      9.85           0       0.00             0.000
   8   m-Cresol                     10.16         282       1.09             1.993
  --   p-Cresol                     10.40           0       0.00             0.000
  10   2-Ethylphenol                11.91         148       0.57             1.263
  11   2,5-Xylenol                  12.55        7694      29.85            45.007
  --   2,4-Xylenol                  12.88           0       0.00             0.000
  12   4-Ethylphenol                13.44        5721      22.20            34.390
  --   2,6-Xylenol                  13.55           0       0.00             0.000
  --   2,3-Xylenol                  13.91           0       0.00             0.000
  --   3,4-Xylenol                  14.26           0       0.00             0.000

Totals :                                        17356      67.34           101.711
```

BTG Biomass Technology Group BV
Teer monster analyse (fenolen)

```
File        : c:\maestro\chrom\Denmark.014
Method      : c:\maestro\methods\10fenol7.met
Sample ID   : Harrie / Denemarke
Vial        : 14
Volume      : 1
Acquired    : Nov 18, 1998 2 :48:00
Printed     : Dec 03, 1998 1 :08:15
User        : Ber[illegible]B
```

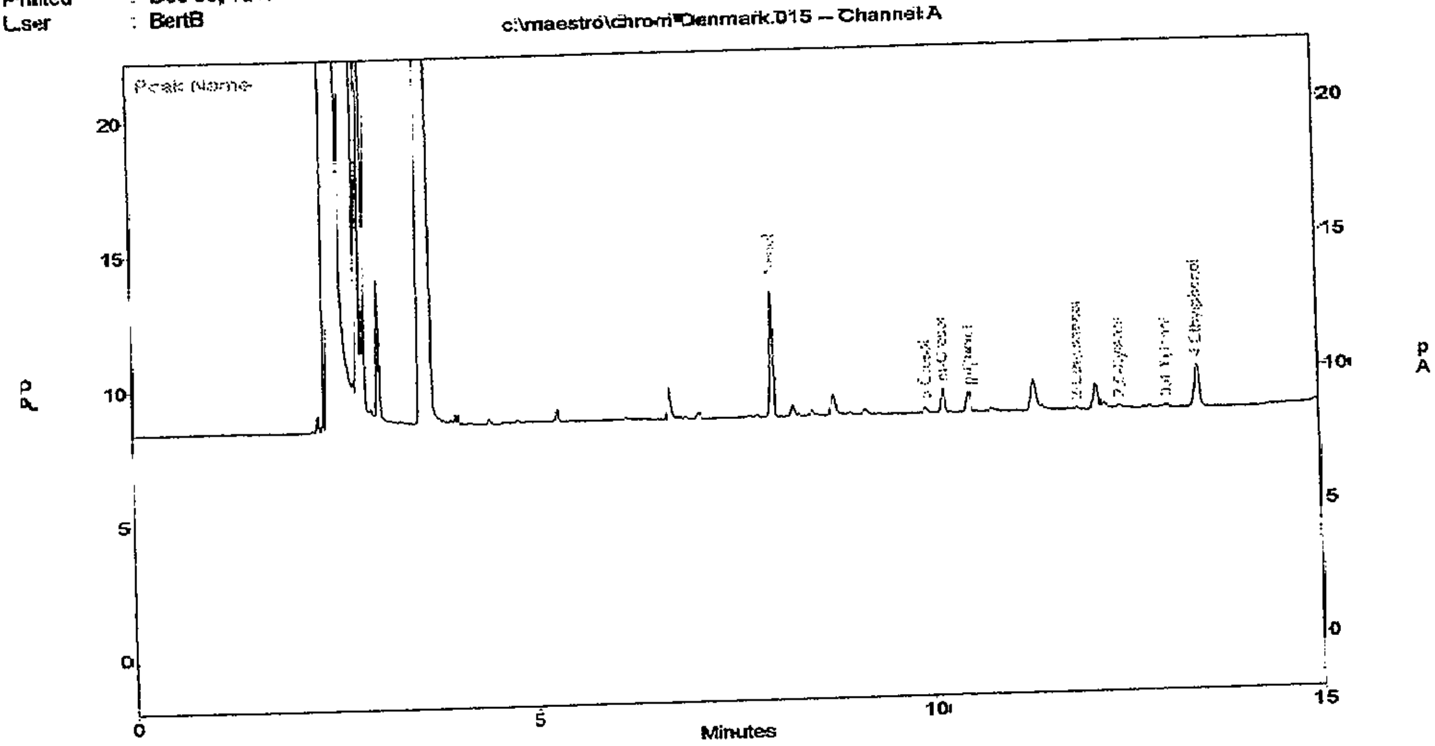
c:\maestro\chrom\Denmark.014 -- Channel A



```
Channel A Results

Peak  Peak name                    Time        Area      Area %   Conc. (ug/sample)
----- ----------------------   ----------  ----------   ------  -----------------
   7  Phenol                         8.00       12086     9.47             65.897
  15  o-Cresol                       9.94        2325     1.82             14.630
  16  m-Cresol                      10.16        3756     2.94             23.822
  17  p-Cresol                      10.49        3802     2.98             21.685
  21  2-Ethylphenol                 11.89         378     0.30              2.941
  24  2,5-Xylenol                   12.37        4454     3.49             26.744
  27  2,4-Xylenol                   12.97         720     0.56              5.182
  28  4-Ethylphenol                 13.43        5604     4.39             34.390
  29  2,6-Xylenol                   13.81         496     0.39              2.919
  30  2,3-Xylenol                   13.99        1208     0.95              7.147
  --  3,4-Xylenol                   14.26           0     0.00              0.000

Totals :                                        34829    27.30            205.355
```

BTG Biomass Technology Group BV
Teer monster analyse (fenolen)

File : c:\maestro\chrom\Denmark.015
Method : c:\maestro\methods\10fenol7.met
Sample ID : Harrie / Denemarke
Vial : 15
Volume : 1
Acquired : Nov 19, 1998 00:23:48
Printed : Dec 03, 1998 16:08:45
User : BertB

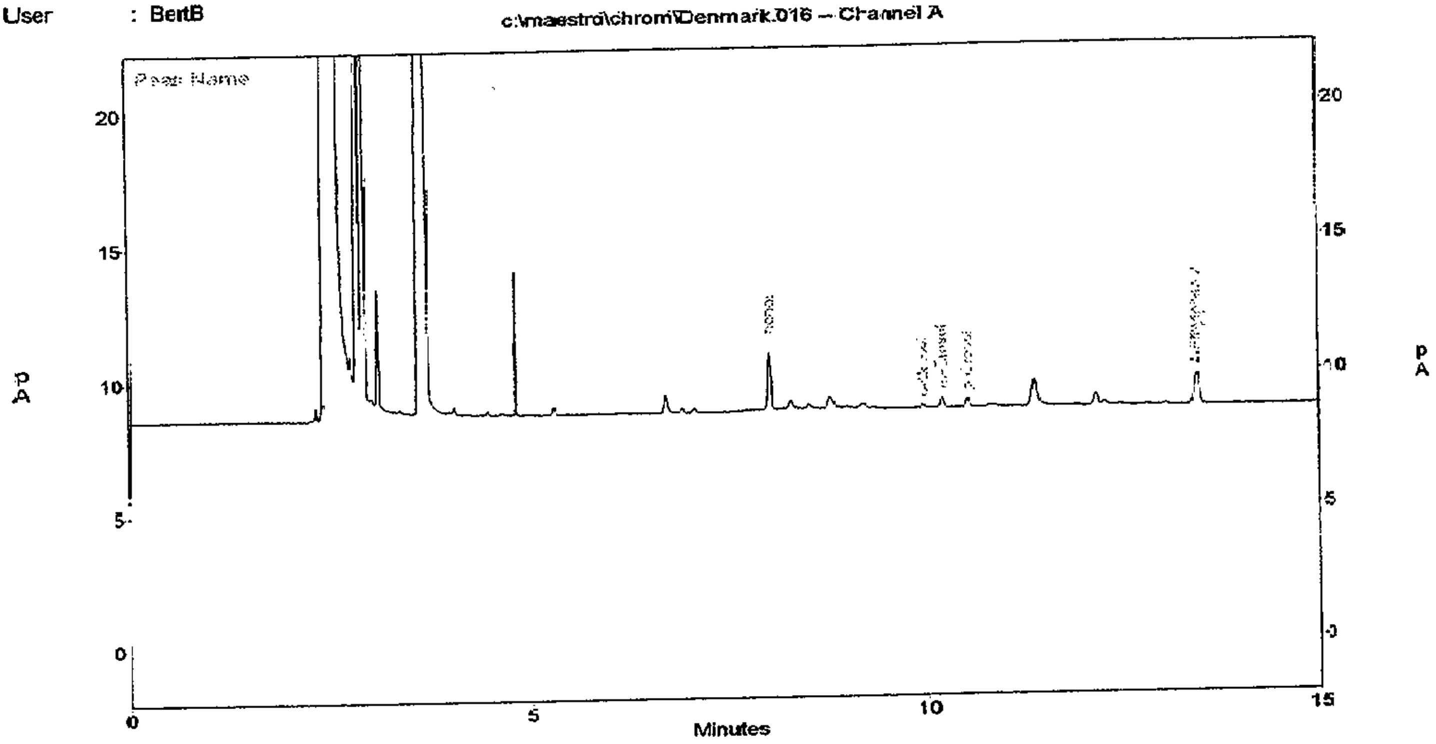


Channel A Results

Peak	Peak name	Time	Area	Area %	Conc. (ug/sample)
6	Phenol	8.01	11929	24.60	54.622
12	o-Cresol	9.95	666	1.37	4.150
13	m-Cresol	10.17	2801	5.78	14.996
14	p-Cresol	10.51	2277	4.70	10.887
16	2-Ethylphenol	11.91	148	0.31	1.113
19	2,5-Xylenol	12.46	414	0.85	2.412
21	2,4-Xylenol	13.05	629	1.30	3.928
22	4-Ethylphenol	13.45	6682	13.78	34.390
--	2,6-Xylenol	13.55	0	0.00	0.000
--	2,3-Xylenol	13.91	0	0.00	0.000
--	3,4-Xylenol	14.26	0	0.00	0.000
Totals :			25546	52.68	126.498

BTG Biomass Technology Group BV
Teer monster analyse (fenolen)

File : c:\maestro\chrom\Denmark.016
Method : c:\maestro\methods\10fenol7.met
Sample ID : Harrie / Denemarke
Vial : 16
Volume : 1
Acquired : Nov 19, 1998 00:59:39
Printed : Dec 03, 1998 16:09:15
User : BertB

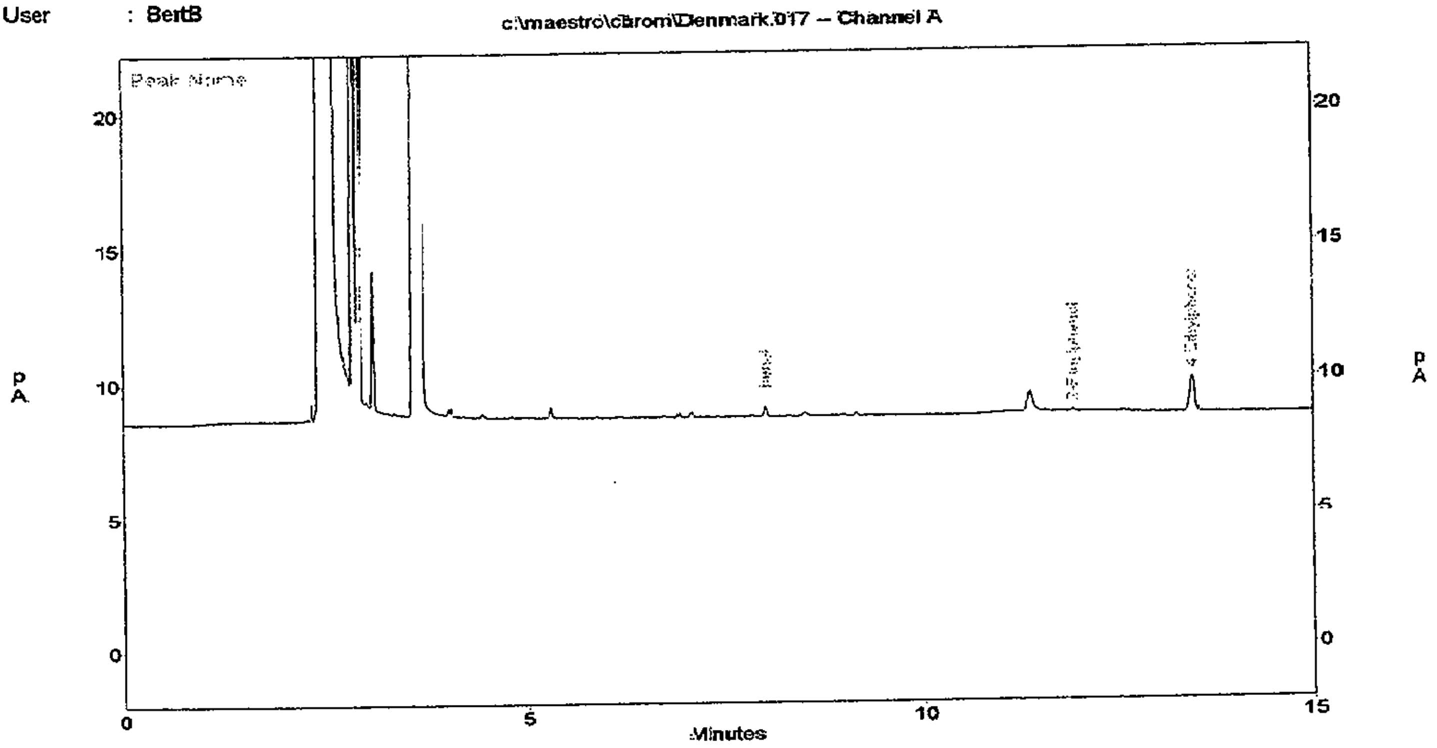

c:\maestro\chrom\Denmark.016 -- Channel A

Channel A Results

Peak	Peak name	Time	Area	Area %	Conc. (ug/sample)
4	Phenol	8.02	5483	19.17	34.114
10	o-Cresol	9.96	400	1.40	3.528
11	m-Cresol	10.18	1279	4.47	9.360
12	p-Cresol	10.51	1082	3.78	6.993
--	2-Ethylphenol	11.77	0	0.00	0.000
--	2,5-Xylenol	12.31	0	0.00	0.000
--	2,4-Xylenol	12.88	0	0.00	0.000
16	4-Ethylphenol	13.46	4941	17.28	34.390
--	2,6-Xylenol	13.55	0	0.00	0.000
--	2,3-Xylenol	13.91	0	0.00	0.000
--	3,4-Xylenol	14.26	0	0.00	0.000
Totals :			13185	46.10	88.385

BTG Biomass Technology Group BV
Teer monster analyse (fenolen)

File : c:\maestro\chrom\Denmark.C17
Method : c:\maestro\methods\10fenol7.met
Sample ID : Harrie / Denemarke
Vial : 17
Volume : 1
Acquired : Nov 19, 1998 01:35:21
Printed : Dec 03, 1998 16:09:45
User : BertB

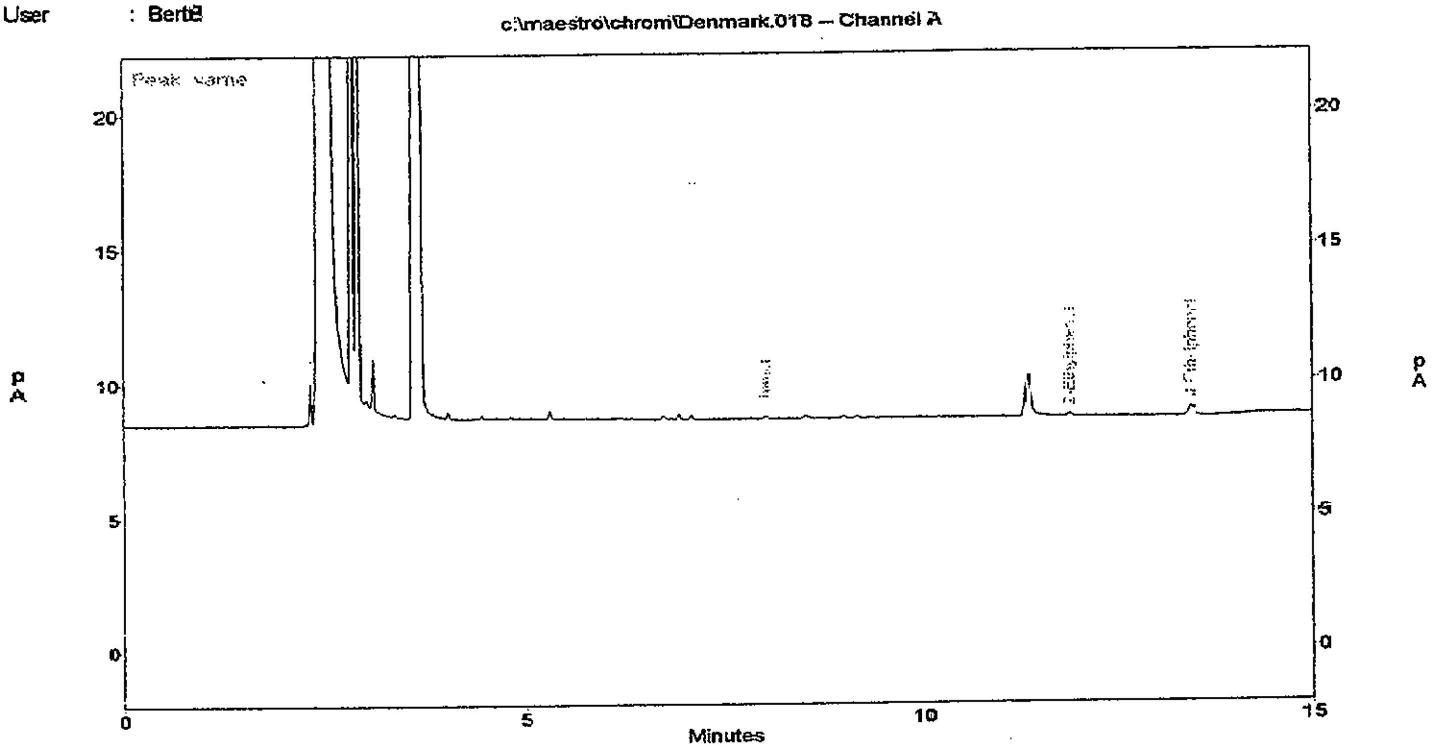


Channel A Results

Peak	Peak name	Time	Area	Area %	Conc. (ug/sample)
3	Phenol	8.01	915	7.57	5.454
--	o-Cresol	9.85	0	0.00	0.000
--	m-Cresol	10.05	0	0.00	0.000
--	p-Cresol	10.40	0	0.00	0.000
8	2-Ethylphenol	11.90	160	1.32	1.387
--	2,5-Xylenol	12.31	0	0.00	0.000
--	2,4-Xylenol	12.38	0	0.00	0.000
9	4-Ethylphenol	13.45	5526	45.70	34.390
--	2,6-Xylenol	13.55	0	0.00	0.000
--	2,3-Xylenol	13.91	0	0.00	0.000
--	3,4-Xylenol	14.26	0	0.00	0.000
Totals :			6601	54.59	41.231

BTG Biomass Technology Group BV
Teer monster analyse (Fenolen)

File : c:\maestro\chrom\Denmark.018
Method : c:\maestro\methods\10fenol7.met
Sample ID : Harre / Denemarke
Vial : 18
Volume : 1
Acquired : Nov 19, 1998 02:10:54
Printed : Dec 03, 1998 16:10:15
User : BertB



Channel A Results

Peak	Peak name	Time	Area	Area %	Conc. (ug/sample)
4	Phenol	8.01	211	1.84	4.579
--	o-Cresol	9.85	0	0.00	0.000
--	m-Cresol	10.05	0	0.00	0.000
--	p-Cresol	10.40	0	0.00	0.000
9	2-Ethylphenol	11.90	252	2.19	6.809
--	2,5-Xylenol	12.31	0	0.00	0.000
--	2,4-Xylenol	12.88	0	0.00	0.000
10	4-Ethylphenol	13.45	1543	13.43	34.390
--	2,6-Xylenol	13.55	0	0.00	0.000
--	2,3-Xylenol	13.91	0	0.00	0.000
--	3,4-Xylenol	14.26	0	0.00	0.000
Totals :			2006	17.46	45.778

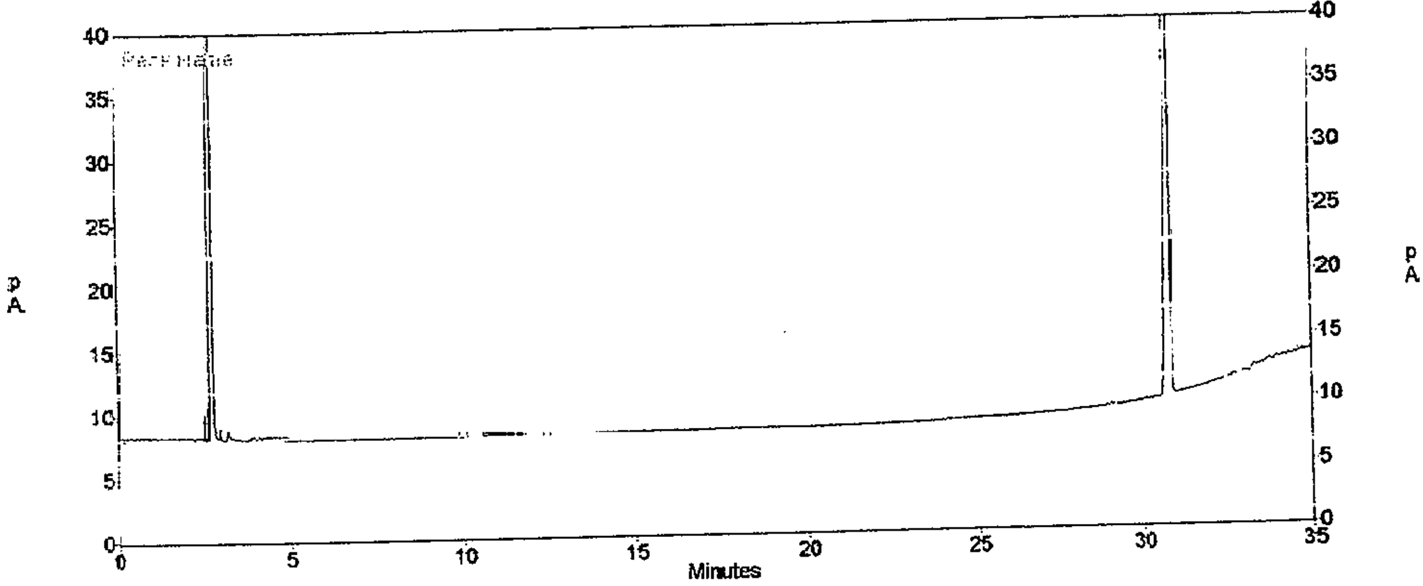
BTG Biomass Technology Group BV
Teer monsteranalyse (aromaten)

NA Oven T>105°C

File : c:\maestro\chrom\avhex2.001
Method : c:\maestro\methods\8aroma5.met
Sample ID : testen teer
Vial : 3
Volume : 1
Acquired : Nov 27, 1998 10:25:16
Printed : Dec 03, 1998 15:29:12
User : BertB

Exp. 4

c:\maestro\chrom\avhex2.001 -- Channel A



Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
--	Benzene	4.23	0	0.000	0.000
--	Toluene	6.96	0	0.000	0.000
--	o-Xylene	10.13	0	0.000	0.000
--	p-Xylene	10.80	0	0.000	0.000
--	I.S.	13.34	0	0.000	0.000
--	Indene	14.99	0	0.000	0.000
--	Naphthalene	18.39	0	0.000	0.000
--	2-Methylnaphthalene	20.65	0	0.000	0.000
--	1-Methylnaphthalene	20.99	0	0.000	0.000
--	Biphenyl	22.18	0	0.000	0.000
--	Acenaphthylene	23.68	0	0.000	0.000
--	Acenaphthene	24.25	0	0.000	0.000
--	Fluorene	25.98	0	0.000	0.000
--	Phenanthrene	29.25	0	0.000	0.000
--	Anthracene	29.41	0	0.000	0.000
--	Fluoranthene	33.29	0	0.000	0.000
--	Pyrene	34.10	0	0.000	0.000
Totals			0	0.000	0.000

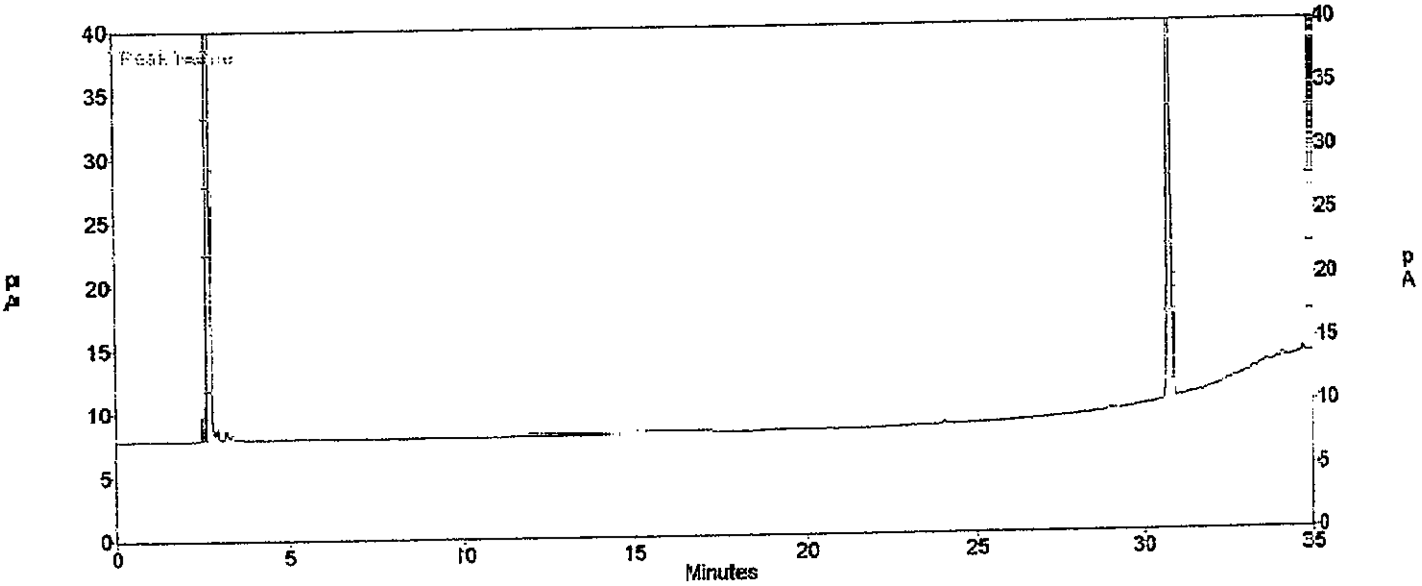
Na oven T>105 °C

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

File : c:\maestro\chrom\avhex2.002
Method : c:\maestro\methods\8aroma5.met
Sample ID : testen teer
Vial : 4
Volume : 1
Acquired : Nov 27, 1998 11:11:37
Printed : Dec 03, 1998 15:29:46
User : BertB

Exp 5

c:\maestro\chrom\avhex2.002 – Channel A



Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
--	Benzene	4.23	0	0.000	0.000
--	Toluene	6.96	0	0.000	0.000
--	o-Xylene	10.13	0	0.000	0.000
--	p-Xylene	10.80	0	0.000	0.000
--	I.S.	13.34	0	0.000	0.000
--	Indene	14.99	0	0.000	0.000
--	Naphthalene	18.39	0	0.000	0.000
--	2-Methylnaphthalene	20.65	0	0.000	0.000
--	1-Methylnaphthalene	20.99	0	0.000	0.000
--	Biphenyl	22.18	0	0.000	0.000
--	Acenaphthylene	23.68	0	0.000	0.000
--	Acenaphthene	24.25	0	0.000	0.000
--	Fluorene	25.98	0	0.000	0.000
--	Phenanthrene	29.25	0	0.000	0.000
--	Anthracene	29.41	0	0.000	0.000
--	Fluoranthene	33.29	0	0.000	0.000
--	Pyrene	34.10	0	0.000	0.000
Totals :			0	0.000	0.000

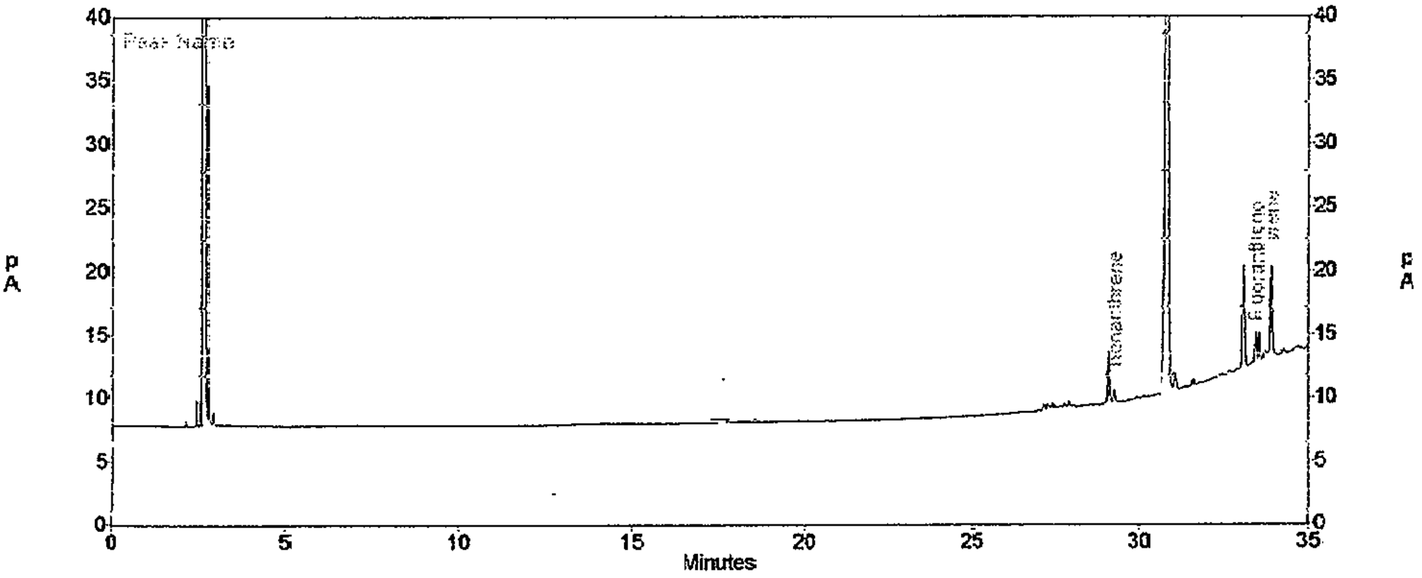
BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

N₂ oven T>105°C

File : c:\maestro\chrom\avhex2.003
Method : c:\maestro\methods\8aroma5.met
Sample ID : testen teer
Vial : 5
Volume : 1
Acquired : Nov 27, 1998 11:58:04
Printed : Dec 03, 1998 15:30:21
User : BertB

Exp.

c:\maestro\chrom\avhex2.003 -- Channel A



Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
--	Benzene	4.23	0	0.000	0.000
--	Toluene	6.96	0	0.000	0.000
--	o-Xylene	10.13	0	0.000	0.000
--	p-Xylene	10.80	0	0.000	0.000
--	I.S.	13.34	0	0.000	0.000
--	Indene	14.99	0	0.000	0.000
--	Naphthalene	18.39	0	0.000	0.000
--	2-Methylnaphthalene	20.65	0	0.000	0.000
--	1-Methylnaphthalene	20.99	0	0.000	0.000
--	Biphenyl	22.18	0	0.000	0.000
--	Acenaphthylene	23.68	0	0.000	0.000
--	Acenaphthene	24.25	0	0.000	0.000
--	Fluorene	25.98	0	0.000	0.000
12	Phenanthrene	29.26	3457	0.009	0.013
--	Anthracene	29.41	0	0.000	0.000
17	*Fluoranthene*	33.44	10302	0.028	0.008
18	Pyrene	33.92	29569	0.081	0.005
Totals :			43328	0.118	0.026

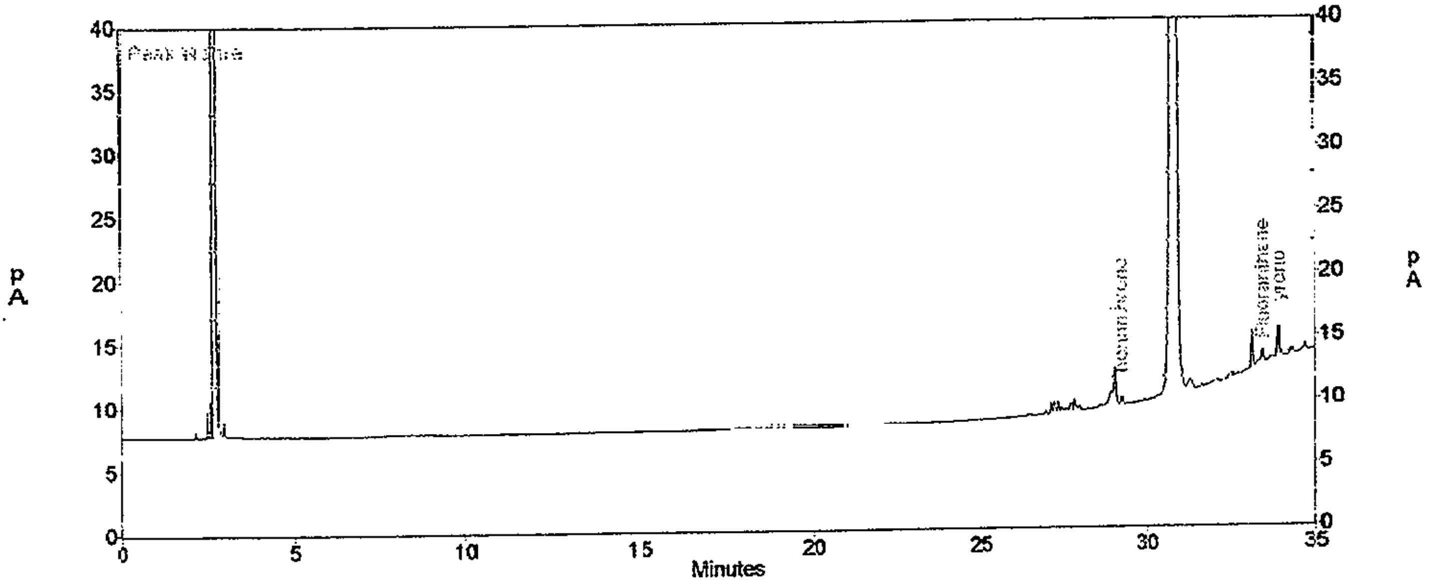
NA oven T>105°C

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

File : c:\maestro\chrom\avhex2.004
Method : c:\maestro\methods\8aroma5.met
Sample ID : testen teer
Vial : 6
Volume : 1
Acquired : Nov 27, 1998 12:44:26
Printed : Dec 03, 1998 15:30:56
User : BertB

bxp-7

c:\maestro\chrom\avhex2.004 -- Channel A



Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
--	Benzene	4.23	0	0.000	0.000
--	Toluene	6.96	0	0.000	0.000
--	o-Xylene	10.13	0	0.000	0.000
--	p-Xylene	10.80	0	0.000	0.000
--	I.S.	13.34	0	0.000	0.000
--	Indene	14.99	0	0.000	0.000
--	Naphthalene	18.39	0	0.000	0.000
--	2-Methylnaphthalene	20.65	0	0.000	0.000
--	1-Methylnaphthalene	20.99	0	0.000	0.000
--	Biphenyl	22.18	0	0.000	0.000
--	Acenaphthylene	23.68	0	0.000	0.000
--	Acenaphthene	24.25	0	0.000	0.000
--	Fluorene	25.98	0	0.000	0.000
14	Phenanthrene	29.26	2201	0.006	0.013
--	Anthracene	29.41	0	0.000	0.000
19	Fluoranthene	33.44	6413	0.017	0.003
20	Pyrene	33.92	9777	0.025	0.005

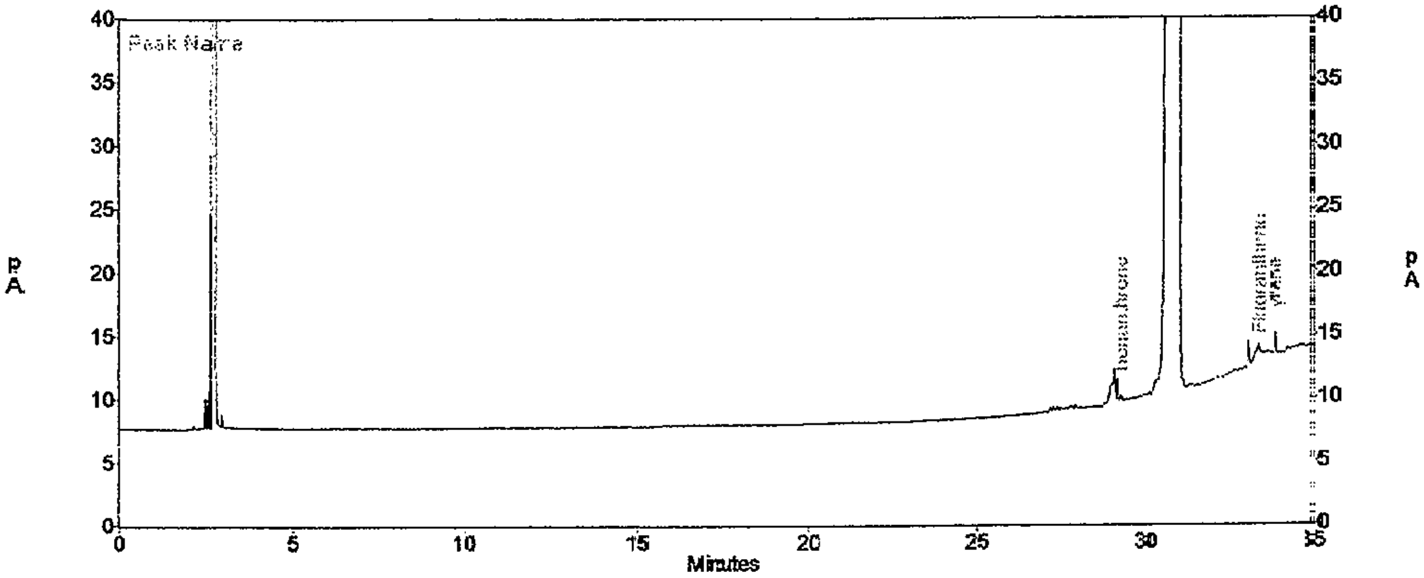
Totals :			18391	0.048	0.026

BTG Biomass Technology Group BV
Teer monster analyse (aromaten)

Na. Cvbr T>105°C

File : c:\maestro\chrom\avhex2.005
Method : c:\maestro\methods\aroma5.met
Sample ID : testen teer Exp 8
Vial : 7
Volume : 1
Acquired : Nov 27, 1998 13:30:32
Printed : Dec 03, 1998 15:40:56
User : BertB

c:\maestro\chrom\avhex2.005 – Channel A



Channel A Results

Peak	Peak Name	Time	Area	Area %	Conc (ug/sample)
--	Benzene	4.23	0	0.000	0.000
--	Toluene	6.96	0	0.000	0.000
--	o-Xylene	10.13	0	0.000	0.000
--	p-Xylene	10.80	0	0.000	0.000
--	I.S.	13.34	0	0.000	0.000
--	Indene	14.99	0	0.000	0.000
--	Naphthalene	18.39	0	0.000	0.000
--	2-Methylnaphthalene	20.65	0	0.000	0.000
--	1-Methylnaphthalene	20.99	0	0.000	0.000
--	Biphenyl	22.18	0	0.000	0.000
--	Acenaphthylene	23.68	0	0.000	0.000
--	Acenaphthene	24.25	0	0.000	0.000
--	Fluorene	25.98	0	0.000	0.000
11	Phenanthrene	29.25	1670	0.004	0.013
--	Anthracene	29.41	0	0.000	0.000
15	Fluoranthene	33.43	7372	0.017	0.008
16	Pyrene	33.90	6562	0.015	0.005
Totals :			15604	0.036	0.026

APPENDIX 2

Parallel Measurement of Dust and Tar in Connection with Updraft and Downdraft Gasification of Wood

DTI Energy
Denmark

by Uwe Zielke
January 1999

MEASUREMENT REPORT

Date: 1999.01.29
Report No.: ELAB-0362
Page: 1 of 32

Ref.: UZI/FDK/HAC
File No.: 270-8-0093
Enclosures: 3

Client:

Contact person: Henrik Flyver Christiansen
Company: Energistyrelsen (Danish Energy Agency)
Address: Amaliegade 44
City: 1256 Copenhagen K
Country: Denmark
Tel.: 3392 6700
Fax: 3311 4743

Subject: Parallel measurement of dust and tar in connection with updraft and downdraft gasification of wood.

Date: The measurements were carried out in week 45, 1998.

Procedure: Measurements were carried out according to the internal DTI standard.

Result: Please refer to chapter 3 of this report and the conclusion on page 32.

Remarks: The measurements have been carried out simultaneously by VTT, BTG and Verenum.

Terms: The measurements have been carried out according to the general terms and conditions regarding commissioned work accepted by the Danish Technological Institute. The measurement results apply to the tested components only. This measurement report may be reproduced in extract only if the laboratory in writing has approved the extract.

Location: Energy, DK-Aarhus

Date: 29-1-1999

Signature:

Uwe Zielke
B.Sc. (mech. eng.)

1. Scope and objective of the parallel test

The measurements document the results of 4 different methods of measuring dust and tar in producer gas from wood gasification. As far as possible, the measurements have been carried out simultaneously on a representative gas flow before and after the gas purification aggregate of the plant. Measurements were performed on an updraft gasification plant and on a downdraft gasification plant. The plants are anonymous and in the following they are called "plant A" and "plant "B". The measurements were carried out in week 45/1998 by Poul D. Kellberg, chemical engineer, and Finn Petersen, chemical process engineer. Uwe Zielke, B.Sc. (mech. eng.), co-ordinated the implementation of the measurements.

The measurements show the following relations:

- Comparison of measurement results when sampling raw gas with high tar concentrations
- Comparison of measurement results when sampling gas with minor tar concentrations
- Comparison of measurement results when sampling raw gas with high dust concentrations
- Comparison of measurement results when sampling gas with minor dust concentrations
- The samples will be analysed for heavy tar, light tar and particles
- The costs of the measurement methods in connection with sampling, analysis and reporting

In addition, the composition of the producer gas is continuously measured and operating parameters are registered for the test period.

The following measurements have been carried out by the Danish Technological Institute:

- 3 dust and tar measurements on updraft gasifier in raw gas
- 3 dust and tar measurements on updraft gasifier in cleaned bypass gas stream after scrubber
- 3 dust and tar measurements on downdraft gasifier in raw gas
- 3 dust and tar measurements on downdraft gasifier in cleaned gas after scrubber

The measurements were carried out simultaneously with three other European laboratories – namely, VVT from Finland, BTG from the Netherlands and Verenum from Switzerland. This report solely deals with the measurement results of the Danish Technological Institute.

2. How the measurements were carried out

2.1 Applied sampling and analysis methods

Sampling has been carried out according to the DTI method for dust and tar measurement and the sample train appears from figure 1. Analysis standards and regulations appear from the laboratory reports.

Sampling is carried out via a 3" sampling port equipped with a ball valve and a gas-proof gate. The sampling probe can be inserted or removed during operation and no gas will be released from the system. Isokinetic sampling is carried out centrally in the gas line or by traversing in several test points depending on the gas line dimension. Isokinetic sampling is ensured by suction nozzle choice and sampling flow adjustment. The gas flows through a heated filter where dust and tar are sampled. Depending on the expected dust and tar concentrations differing filter medium and filter temperatures are chosen.

- In connection with updraft gasification with high tar concentrations a G3 glass frit is chosen and it is heated to 105 °C.
- In connection with downdraft gasification with high dust concentrations a Ströhlein – quartz wool cartridge (2-12 micron) is chosen and it is heated to 250-350 °C.

After filtration the gas flows through a glass pipe cooler where the gas temperature is lowered to less than 8 °C. This is where the water content of the gas and pyrolysis products are condensed when they have passed the filter.

From the cooler the gas flows through an adsorbent (XAD 2, PUF, active coal, etc.). This is where volatile components and aerosols are adsorbed when they have passed the cooler. Previous examinations have shown that XAD 2 has the best adsorption property.

After sampling, the equipment is rinsed with acetone and dichlormetane (DCM). Extraction solvents and condensate are filtered and analysed for dust and tar. In order to identify where in the sampling system dust and tar are retained the various remanences and filtrates have been analysed individually. The method can be simplified by pouring the extracts together so only one remanence and one filtrate has to be examined.

2.2 Measuring instruments

Instruments for dust and tar measurement

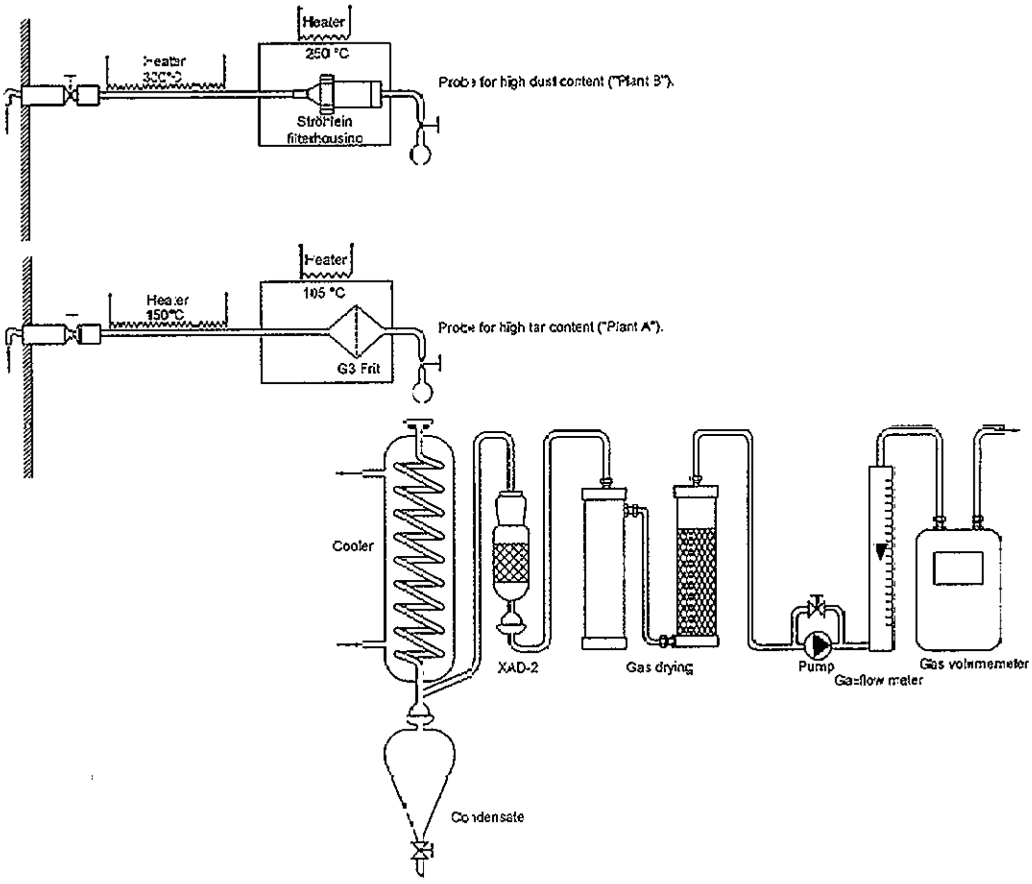


Figure 1: The DTI sample train for dust and tar measurements

Instruments for gas analysis

Gas analysers for producer gas measurements (LCV gas)

CO concentration:	NDIR gas analyser, brand Hartmann & Braun, type URAS 4 with cabinet purge
Measurement range 2:	0-40 vol% CO
H_2 concentration:	Thermal conductivity gas analyser, brand Hartmann & Braun, type d with cabinet purge
Measurement range 1:	0-10 vol% H_2
Measurement range 2:	0-40 vol% H_2

CH_4 concentration: Flame ionization demodulator, brand Hartmann & Braun, type Micro FID 100 with 3 measurement ranges

CO_2 content: NDIR gas analyser, brand Hartmann & Braun, type URAS 4 with cabinet purge

Measurement range: 0-20 vol% CO_2

O_2 content: Paramagnetic gas analyser, brand Hartmann & Braun, type MAGNOS 6 with cabinet purge

Measurement range 1: 0-10 vol% O_2
Measurement range 2: 0-25 vol% O_2

The gas analysers are intended for measuring explosive gas mixtures. They are incorporated in gas-proof cabinets that are constantly purged with nitrogen.
The gas analysers are checked in the 0-point with 100% N_2 and at app. 80% of full scale by means of certified reference gas (calibration gas).

2.3 Measurement environment and remarks to the measurements

Dust and tar measurements have been carried out at two Danish gasification plants. "Plant A" is a 5 MW updraft gasifier utilizing wood chips with average water contents of 45%. The samples are taken in a bypass gas line where a gas flow of 350 Nm^3/h is led to a pilot gas scrubber. Measurements are carried out before as well as after the scrubber. The gas line internal diameter before the scrubber is 213 mm and after the scrubber it is 108 mm. The samples are taken at one point, centrally in the gas line. The gas temperature and pressure before the scrubber is 80 °C and –150 Pa and after the scrubber the temperature is 42 °C and the pressure is 1000 Pa. The flow velocity of the gas is 3.5 m/s before the scrubber and 12.1 m/s after the scrubber. In order to obtain isokinetic sampling a 15 mm nozzle is applied before the scrubber requiring a sample flow of 21 litres/min. After the scrubber a 6 mm nozzle is applied requiring a sample flow of 17 litres/min. The obtained sample velocities are 2.2 m/s and 14.3 m/s, respectively, before and after the scrubber. The sub-isokinetic sampling before the scrubber is due to a considerable system pressure drop in the sample train. The measurements were carried out indoors.

At "plant B", which is a 1 MW downdraft gasifier, wooden blocks with an average water content of 12% are used. The samples are taken in the main gas line before and after the scrubber. The gas flow is app. 240 Nm^3/h. The gas line internal diameter before the scrubber is 150 mm and after the scrubber it is 110 mm. The samples are taken at one point, centrally in the pipe. The gas temperature and pressure before the scrubber is 595 °C and –150 Pa and after the scrubber the temperature is 45 °C and the pressure is –1000 Pa. The flow velocity of the gas is 12.3 m/s before the scrubber and 9.1 m/s after the scrubber. In order to obtain isokinetic sampling a 8 mm nozzle is applied before the scrubber requiring a sample flow of 11 litres/min. After the scrubber a 8 mm nozzle is applied requiring a sample flow of 19 litres/min. The obtained sample velocities are 12.8 m/s and 7.7 m/s, respectively, before and after the scrubber. Primarily small particles were obtained after the scrubber and therefore the error owing to deviation in isokinetic sampling can be ignored. The measurements were carried out indoors.

3. Results

3.1 Measurements on updraft gasifier "plant A"

3.1.1 Gravimetric measurement of dust in raw gas

Dust and tar are sampled on a G3 frit (16-40 micron) that has been heated to 105 °C so water can pass through the frit. In order to obtain an initial indication of the pollution level of the gas the glass frit was weighed on-site. Besides the frits, the sampling probe (from the inlet nozzle to the frit) was check-weighed after each measurement. The weight increase, in average 0.2 g per measurement, was minimal and might owe to differences in use of sealing material on the thread of the probe. This insignificant weight difference has been ignored. The initial weighing result after frit cooling appears from table 1.

Measurement No.	Date	Measurement period	Frit temperature °C	Mass of dust and tar on the frit g	Amount of dust and tar on the frit g/Nm^3
1	02.11.98	09:32-10:02	120-130	15.9	47.6
2	02.11.98	11:43-12:13	95-105	21.9	81.6
3	02.11.98	14:15-14:48	102-108	29.4	80.3

Table 1: Dust and tar on the frit, initial weighing immediately after sampling

Measurement 1 differs from the other measurements, as the result is lower than the result of measurement 2 and 3. That is presumably because the increased frit temperature made an increased amount of tar pass through the frit during measurement 1.

The dust content in the raw gas is measured by addition of 4 contributions originating from the following:

- Dust in probe before the glass frit
- Extractable dust on the glass frit
- Residual dust in the glass frit that cannot be shaken out
- Dust in sample train downstream the frit (especially cooler)

When measurements 1 + 2 + 3 have been carried out the sampling probe is rinsed with acetone and dichlormethane (DCM). The extraction solvent is filtrated and the dust and tar content is distributed according to the exhausted gas volumes of the 3 measurements.

After the above-mentioned initial weighing of the frits they are rinsed on-site with acetone and (DCM). At the laboratory, the frits are shaken up with acetone and DCM in order to extract possible dust and tar residuals. Furthermore, it is examined if dust has penetrated the frit as the solvents from the cooler and the connection pipes are filtrated. The tar content of the filtrate is determined and the remanence is dried at 105°C and analysed for dust. The amount of dried remanences equals the solid substance content of the raw gas (incl. the residual volatile components), see table 2.

Measurement No.	Date	Measurement period	Remanence from filtration of frit extract mg	Residue on cleaned frit and probe mg	Remanence from filtration of condensate mg	Remanence from filtration of DCM and acetone mg	Solid substance in raw gas mg/Nm^3
1	02.11.98	09:32-10:02	59.3	68.9	7.3	22.7	526
2	02.11.98	11:43-12:13	70.8	102.5	6.5	87.4	839
3	02.11.98	14:15-14:48	52.8	21.9	8.6	28.5	305

Table 2: Solid substance in raw gas (drying at 105 °C)

Table 2 shows that in average 30% of the total solid substance of the raw gas penetrates the frit. A filter efficiency of 70% is not satisfactory. In connection with measurements requiring precision it is necessary to determine the remanences from filtration of condensate and extraction solvents. Alternatively, a G4 frit (10-16 micron) can be used but that results in an unacceptably large system pressure drop.

In order to examine if the solid substance contains volatile tar components the matter is heated to 450 °C in N_2 for 30 minutes and afterwards the remanence is weighed once again. The weighing results indicate the dust content in the raw gas. By means of heating, the remanence mass is in average reduced to 49.3%. If the values from table 2 are corrected for 50.7% of volatile components, then the following dust concentrations in the raw gas are obtained.

Measurement No.	Date	Measurement period	Dust amount after heating mg	Dust in raw gas mg/Nm^3
1	02.11.98	09:32-10:02	78	259
2	02.11.98	11:43-12:13	132	414
3	02.11.98	14:15-14:48	55	150

Table 3: Dust in raw gas (heating to 450 °C in nitrogen (30 min.))

In order to determine the dust content of flammable material (C) and the dust content of inorganic material (ashes) the remanence is reheated to 550°C with air access. This examination shows that dust in raw gas consists of 93% flammable material and 7% ashes.

If the laboratory results are compared with the on-site weighing results (table 1), then it is demonstrated that the main part of the frit volume increase is due to tar as the dust on the frits in average only amounts to app. 0.3% of the collected mass in the frit. Chapter 3.1.4 illustrates if on-site frit weighing is an applicable alternative to the extensive analyses at the laboratory.

3.1.2 Gravimetric measurement of dust in gas after gas scrubber

In connection with the dust measurements after the plant's gas scrubber the same procedure is used as described above. The weighing results of the solid substance fractions after drying at 105 °C appear from table 4.

Measurement No.	Date	Measurement period	Remanence from filtration of frit extract mg	Residue on cleaned frit mg	Remanence from filtration of condensate mg	Remanence from filtration of DCM and acetone mg	Solid substance in clean gas mg/Nm³
4	03.11.98	09:15-10:15	27.5	34	5.0	9.1	72.8
5	03.11.98	11:15-12:18	17.2	14	6.6	1.5	37.1
6	03.11.98	13:47-14:47	15.2	7	2.1	2.4	27.1

Table 4: Solid substance in clean gas (drying at 105 °C)

When measuring in the somewhat cleaner gas after the gas scrubber, filter efficiency in average increases to 82%. However, that is not enough to ignore the remanence from filtration of condensate and extraction solvent.

As mentioned before, the solid substance is heated to 450 °C in N_2 and afterwards the dust content is determined. By means of heating the remanence mass in average is reduced to 60.7%. Consequently, 39.3% volatile components were present in the solid substance after the scrubber.

Measurement No.	Date	Measurement period	Amount of dust after heating mg	Dust in clean gas mg/Nm³
4	03.11.98	09:15-10:15	46	44
5	03.11.98	11:15-12:18	24	23
6	03.11.98	13:47-14:47	16	16

Table 5: Dust in clean gas (heating to 450 °C in nitrogen (30 min.))

In order to determine the dust's content of flammable material (C) and the dust's content of inorganic material (ashes) the remanence is reheated to 550 °C with air access. This examination shows that dust in the clean gas consists of 91% flammable material and 9% ashes.

3.1.3 Gravimetric measurement of heavy tar in raw gas

The various parts of the sample train are rinsed and extracted with acetone and DCM and afterwards the extracts are filtered. The filtrate is then examined for tar. In principal that takes place in two ways, partly by evaporation of the filtrates and partly by a GC analysis.

- In connection with evaporation the tar fraction called "heavy tar" is determined
- The "light tar" is determined by a GC analysis of organic substances with a molar mass between 78 and 202.

A tar measurement carried out according to the DTI method results in 3 extracts, originating from:

- G3 frit
- Glass pipe cooler (incl. rinsing)
- Adsorbent which in this case is XAD 2

In connection with measurements 1 + 2 + 3, the rinsing of the probe has been described in chapter 3.1.1.

Volatile components, measured when heating the remanences, are added to the "Residue of evaporation from frit and probe" in table 6.

Measure-ment No.	Date	Measurement period	Residue of evaporation from frit and probe mg	Residue of evaporation from cooler mg	Residue of evaporation from XAD 2 mg	Amount of heavy tar in raw gas g/Nm^3
1	02.11.98	09:32-10:02	13150	9740	830	79
2	02.11.98	11:43-12:13	21920	6190	1360	93
3	02.11.98	14:15-14:48	27860	4860	2950	97

Table 6: Heavy tar in raw gas at "plant A"

The previously mentioned penetration of tar through the frit (owing to increased temperature during measurement 1) is confirmed by the above-stated results. Although the lowest tar concentration was registered during measurement 1, the residue of evaporation from the cooler has the greatest value. That is due to the increased frit temperature.

3.1.4 Comparison between on-site frit weighing and evaporation results

In order to examine if field weighing of the frit gives a usable measuring result for easy and quick determination of the "heavy tar" content in gas with high tar concentration a comparison of the results from the direct weighing and the results of laboratory evaporation is carried out.

Measurement No.	Date	Measurement period	Heavy tar in raw gas g/Nm³	On-site frit weighing g/Nm³	Deviation %
1	02.11.98	09:32-10:02	79	47.6	40
2	02.11.98	11:43-12:13	93	81.6	12
3	02.11.98	14:15-14:48	97	80.3	17

Table 7: On-site weighing compared with residue of evaporation

The deviation in measurement No. 1 (table 7) is due to the previously mentioned frit temperature increase. The two other measurements indicate that on-site frit weighing gives a usable result for quick evaluation of the tar production of the updraft gasifier.

3.1.5 GC measurement of light tar in raw gas

Before evaporation of the above-mentioned extracts samples were taken and they were examined by means of a GC/FID analysis.

3.1.5.1 Light tar in aqueous condensate

The water phase solely originates from the glass pipe cooler. The GC/FID analysis of the aqueous condensate from the raw gas appears from table 8.

Measurement No. Date Measurement period	Molar mass / boiling point	1 02.11.98 09:32-10:02 mg/Nm³	2 02.11.98 11:43-12:13 mg/Nm³	3 02.11.98 14:15-14:48 mg/Nm³
Methanol	32 / 65	1830	254	2080
Ethanol	46 / 78	5.0	6.6	5.8
Acetone	58 / 56	17	11	15
Acetic acid	60 / 118	8310	7850	2186
MEK	72 / 80	1.2	0.9	1.6
Propionic acid	74 / 141	1360	534	120
Light tar *)	78 – 202	700	1040	13

Table 8: GC analysis of light tar in aqueous condensate

*) Light tar with molar mass from 78 to 202 is calculated as isobutanol (74 g/mol)

The reason why the light tar total during measurement 3 sharply deviates from measurement 1 and 2 is presumably due to the extraordinarily large heterogeneity of the aqueous condensate. Extraction of the organic phase in the condensate by means of DCM would presumably have given a more uniform result. The degree of accuracy on this contribution is generally speaking of no importance to the result of light tar measurement which appears from chapter 3.1.6.

3.1.5.2 Light tar in acetone and DCM extracts

After sampling and field weighing the frits are rinsed with acetone and DCM. The resulting extract is called "fraction A". The frits are shaken at the laboratory in order to extract possible tar residues, resulting in "fraction B". DCM washed out from the cooler results in "fraction C" and the washed out acetone is called "fraction D". In addition, XAD 2 is extracted, resulting in "fraction E". The following extracts are analysed:

- GC analysis of the fractions A and B together constitute light tar on the frit
- GC analysis of the fractions C and D are light tar in the cooler (excl. water-phase)
- GC analysis of fraction E is light tar in XAD 2

Light tar residues found in the wash out from the measurement probe are added to fraction A and B. The analysis results and the calculation of the light tar concentration in the raw gas appear from table 9.

Measurement No. Date Measurement period	Molar mass/ boiling point	1 02.11.98 09:32-10:02 mg/Nm³	2 02.11.98 11:43-12:13 mg/Nm³	3 02.11.98 14:15-14:48 mg/Nm³
Benzene	78 / 80	157	153	136
Toluene	92 / 111	271	294	276
Phenol	94 / 182	1022	711	1053
Ethylbenzene	106 / 136	47	58	57
Xylene	106 / 139	140	159	153
Indene	116 / 182	34	35	39
Guaiacol	124 / 205	2194	1603	2106
Naphthalene	128 / 218	50	69	65
4-Methyl-Guaiacol	138 / 220	2820	2323	3060
Acenaphthylene	152 / 270	13	16	9.3
Acenaphthene	154 / 278	3.2	2.6	3.6
Fluorene	166 / 298	12	10	13
Phenanthrene	178 / 340	20	18	23
Anthracene	178 / 340	5.9	5.5	6.6
Fluoranthene	202 / 367	3.0	3.1	3.4
Pyrene	202 / 393	3.8	3.9	5.1
Benz(a)anthracene	228 / 400	1.8	2.0	2.3
Chrysene	228 / 410	2.0	2.1	2.4
Benz(b+k)fluoranthene	252 / 480	1.2	1.1	1.4
Benz(a)pyrene	252 / 495	1.0	1.2	1.2
Indeno(1,2,3)pyrene	276 / 500	0.3	0.8	0.4
Benz(ghi)perylene	276 / 510	0.5	0.4	0.3
Dibenz(ah)anthracene	278 / 520	0	0	0
Light tar (78-202) *)		32380	34345	38102

Table 9: GC analysis of light tar in acetone and DCM

*) Light tar with molar mass from 78 to 202 is calculated as anthracene (178 g/mol)

3.1.6 Total light tar in raw gas

As it appears from table 10, total light tar is found by adding light tar to the aqueous condensate and to acetone and the DCM extracts.

Measurement No.	Date	Measurement period	Light tar in condensate g/Nm³	Light tar in acetone and DCM g/Nm³	Light tar in raw gas g/Nm³
1	02.11.98	09:32-10:02	0.7	32.4	33.1
2	02.11.98	11:43-12:13	1.0	34.3	35.3
3	02.11.98	14:15-14:48	0	38.1	38.1

Table 10: Total light tar in raw gas

3.1.7 Gravimetric measurement of heavy tar in gas after gas scrubber

As mentioned in chapter 2.1.3, tar measurements were carried out in the clean gas after the gas scrubber. The results of the evaporation of the tar fractions appear from table 11.

Measurement No.	Date	Measurement period	Residue of evaporation from frit mg	Residue of evaporation from cooler mg	Residue of evaporation from XAD 2 mg	Amount of heavy tar in clean gas g/Nm³
4	03.11.98	09:15-10:15	2860	1550	820	5.0
5	03.11.98	11:15-12:18	2690	500	1660	4.5
6	03.11.98	13:47-14:47	2410	1060	570	4.1

Table 11: Heavy tar in clean gas at "plant A"

3.1.8 GC measurements of light tar in gas after gas scrubber

As mentioned in chapter 3.1.5, concerning GC measurements in the raw gas, the measurements are carried out in the clean gas.

3.1.8.1 Light tar in aqueous condensate

The water phase solely originates from the glass pipe cooler. The GC/FID analysis of the aqueous condensate from the raw gas appears from table 12.

Measurement No. Date Measurement period	Molar mass / boiling point	4 03.11.98 09:15-10:15 mg/Nm³	5 03.11.98 11:15-12:18 mg/Nm³	6 03.11.98 13:47-14:47 mg/Nm³
Methanol	32 / 65	828	436	782
Ethanol	46 / 78	2.5	1.4	3.0
Acetone	58 / 56	13	0.7	0
Acetic acid	60 / 118	828	458	7714
MEK	72 / 80	0.4	0	0.5
Propionic acid	74 / 141	55	56	2030
Light tar *)	78 – 202	23	29	142

Table 12: GC analysis of light tar in aqueous condensate

*) Light tar with molar mass from 78 to 202 is calculated as isobutanol (74 g/mol)

The reason why the light tar total during measurement 3 deviates sharply from measurement 1 and 2 is probably due to the considerable heterogeneity of the aqueous condensate. Also in this case, the results from the aqueous condensate can be ignored, which appears from chapter 3.1.9.

3.1.8.2 Light tar in acetone and DCM extracts

As described in chapter 3.1.5.2, concerning raw gas measurements, the GC analysis is carried out to determine the light tar in the clean gas, which appears from table 13.

Measurement No. Date Measurement period	Molar mass/ boiling point	4 03.11.98 09:15-10:15 mg/Nm^3	5 03.11.98 11:15-12:18 mg/Nm^3	6 03.11.98 13:47-14:47 mg/Nm^3
Benzene	78 / 80	32	30	5.1
Toluene	92 / 111	116	123	64
Phenol	94 / 182	86	86	77
Ethylbenzene	106 / 136	39	37	28
Xylene	106 / 139	116	104	85
Indene	116 / 182	24	23	19
Guaiacol	124 / 205	279	287	247
Naphthalene	128 / 218	27	27	22.2
4-Methyl-Guaiacol	138 / 220	278	306	248
Acenaphthylene	152 / 270	0.8	0.7	0.6
Acenaphthene	154 / 278	0.3	0.2	0.6
Fluorene	166 / 298	0.7	0.7	0.5
Phenanthrene	178 / 340	0.8	0.8	0.6
Anthracene	178 / 340	0.3	0.3	0.3
Fluoranthene	202 / 367	0.2	0.2	0.1
Pyrene	202 / 393	0.2	0.2	0.2
Benz(a)anthracene	228 / 400	0.1	0.1	0.1
Chrysene	228 / 410	0.1	0.1	0.1
Benz(b+k)fluoranthene	252 / 480	0.1	0.1	0.1
Benz(a)pyrene	252 / 495	0.1	0.1	0.1
Indeno(1,2,3)pyrene	276 / 500	0.1	0	0
Benz(ghi)perylene	276 / 510	0	0	0
Dibenz(ah)anthracene	278 / 520	0	0	0
Light tar (78-202) *)		4889	4186	3674

Table 13: GC analysis of light tar in acetone and DCM

*) Light tar with molar mass from 78 to 202 is calculated as anthracene (178 g/mol)

3.1.9 Total light tar in gas after gas scrubber

As it appears from table 14, the light tar total is found by adding light tar to the aqueous condensate and to acetone and the DCM extracts.

Measurement No.	Date	Measurement period	Light tar in condensate g/Nm³	Light tar in acetone and DCM g/Nm³	Light tar in raw gas g/Nm³
4	03.11.98	09:15-10:15	0	4.9	4.9
5	03.11.98	11:15-12:18	0	4.2	4.2
6	03.11.98	13:47-14:47	0.1	3.7	3.8

Table 14: Total light tar in clean gas

3.2 Measurement on downdraft gasifier "Plant B"

3.2.1 Gravimetric measurement of dust in raw gas

Unlike the measurements on "plant A", where a glass frit was used, quartz filter cartridges were used, as the main pollutant in the gas of "plant B" is dust and not tar. Dust is mainly separated in a Ströhlein filter cartridge filled with quartz wool (2-12 micron). The filter and sample probe are heated to 250 °C.

In order to obtain an initial indication of the dust content of the gas, the filters are weighed on-site after cooling. The results from the initial weighing appear from table 15.

Measurement No.	Date	Measurement period	Filter temperature °C	On-site filter weighing g	Dust concentration filter only mg/Nm³
7	05.11.98	10:15-10:31	250	0.14	968
8	05.11.98	11:24-11:55	250	0.22	642
9	05.11.98	14:30-15:06	250	-	-

Table 15: Weighing of Ströhlein filters immediately after sampling

The dust content in the raw gas is measured by adding 4 dust fractions, originating from the following parts of the sampling equipment:

- Probe in front of filter
- Ströhlein filter
- Probe after filter
- Cooler

The Ströhlein filters are treated as usual at the laboratory as they are dried and given a climatic condition before return weighing takes place in the climatic chamber. The sampling probe (from the inlet nozzle to the filter) is rinsed with acetone and DCM after each measurement. The extraction solvent is filtered and the remanence is dried and weighed. In connection with measurements 7 + 8 + 9 + 10 the probe **after** the filter is rinsed in the same way. At the laboratory, the remanence mass is defined as 21.2 mg. This mass is distributed according to the exhausted gas volume at each measurement. Furthermore, the condensate from the cooler is filtered and the remanence is dried and weighed. The mass of the four remanences is included in the column "Solid substance in other sampling equipment".

Measurement No.	Date	Measurement period	Solid substance on filter mg	Solid substance in other sampling equipment mg	Solid substance in raw gas mg/Nm^3
7	05.11.98	10:15-10:31	137.5	35.2	1194
8	05.11.98	11:24-11:55	236.3	104.2	994
9	05.11.98	14:30-15:06	157.8	29.7	790

Table 16: Solid substance in raw gas (drying at 105 °C)

In order to examine if tar is connected to the filters and the remanences they are heated to 450 °C in N_2 for 30 minutes and then they are weighed once again. The weighing results show the actual dust content in the raw gas.

Measurement No.	Date	Measurement period	Dust on filter mg	Dust in other sampling equipment g	Dust in raw gas mg/Nm^3
7	05.11.98	10:15-10:31	131.4	34.4	1146
8	05.11.98	11:24-11:55	228.1	101.8	963
9	05.11.98	14:30-15:06	154.6	29.0	773

Table 17: Dust in raw gas (heating to 400 °C in nitrogen (30 min))

From table 17 it appears that the main part of pollution on the filters, not surprisingly, consists of "real" dust as only app. 3% of the mass is lost during heating. Furthermore, it appears from the table that the filter efficiency is between 81 and 55% and it declines as the dust load increases. Therefore, it is necessary also in future to pay regard to the penetration of the filter.

In order to determine the content of flammable material (C) and the dust content of inorganic material (ashes) the filters and the remanence are reheated to 550 °C with air access. This examination shows that the dust in the raw gas consists of 90% flammable material and 10% ashes.

3.2.2 Comparison of on-site filter weighing with laboratory weighing

Measurement No.	Date	Measurement period	Dust on filter (laboratory) mg	Dust on filter (field weighing) g	Deviation %
7	05.11.98	10:15-10:31	131.4	0.14	+ 6.5
8	05.11.98	11:24-11:55	228.1	0.22	- 3.6
9	05.11.98	14:30-15:06	154.6	-	

Table 18: Comparison of field weighing

Just as heavy tar field measurements, dust field measurements give acceptable results.

3.2.3 Gravimetric measurement of dust in gas after gas scrubber

In connection with dust measurements after the plant's gas scrubber the same procedure is used as described above. Table 19 shows laboratory weighing after the samples have been dried at 105 °C.

Measurement No.	Date	Measurement period	Solid substance on filter mg	Solid substance in other sampling equipment mg	Solid substance in clean gas mg/Nm³
10	06.11.98	09:00-09:20	166.0	13.4	665
11	06.11.98	10:15-10:43	141.8	21.0	449
12	06.11.98	11:41-12:05	136.8	14.3	428

Table 19: Solid substance in clean gas (drying at 105 °C)

As previously mentioned, it has been examined if tar is connected to the filters and the remanences. Therefore, they are heated to 450 °C in N_2 for 30 minutes and then they are weighed once again. These weighing results present the actual dust content of the clean gas.

Measurement No.	Date	Measurement period	Dust on filter mg	Dust in other sampling equipment mg	Dust in clean gas mg/Nm³
10	06.11.98	09:00-09:20	151.2	11.7	604
11	06.11.98	10:15-10:43	136.5	18.3	426
12	06.11.98	11:41-12:05	130.7	12.4	406

Table 20: Dust in clean gas (heating to 400 °C in nitrogen (30 min.))

As it appears from table 20, the solid substance after the scrubber has a somewhat larger amount of volatile components that the solid substance before the scrubber as app. 7% of the mass is lost during heating. In connection with the rather small dust load in the clean gas and the lower gas temperature, the Ströhlein filter is more satisfactory as filter efficiency increases to app. 90%.

In order to determine the dust's content of flammable material (C) and the dust's content of inorganic material (ashes) the filters and the remanence are reheated to 550 °C with air access. This examination shows that the dust in the clean gas consists of 96% flammable material and 4% ashes.

3.2.4 Gravimetric measurement of heavy tar in raw gas

The content of heavy tar in the raw gas is measured by adding 4 contributions, originating from:

- Washing out of probe before filter
- Volatile substances on filter
- Condensate from cooler incl. wash out
- Extraction of XAD 2

In table 21, the volatile components measured during heating of the filter have been added to "Residue of evaporation from probe".

Measurement No.	Date	Measurement period	Residue of evaporation from probe mg	Residue of evaporation from cooler mg	Residue of evaporation from XAD 2 mg	Amount of heavy tar in raw gas mg/Nm^3
7	05.11.98	10:15-10:31	35.4	167.0	74.4	1913
8	05.11.98	11:24-11:55	78.1	74.3	102.4	744
9	05.11.98	14:30-15:06	50.7	24.0	58.4	561

Table 21: Heavy tar in raw gas at "plant B"

3.2.5 GC measurement of light tar in raw gas

Before evaporation of the above-mentioned extracts a sample was taken and they were examined by means of a GC/MS analysis. The aqueous condensate is separated from the DCM phase and has been examined through a GC/FID analysis.

3.2.5.1 Light tar in aqueous condensate

The water phase solely originates from the glass pipe cooler. The GC/FID analysis of the aqueous condensate from the raw gas appears from table 22.

Measurement No. Date Measurement period	Molar mass/ boiling point	7 05.11.98 10:15-10:31 mg/Nm³	8 05.11.98 11:24-11:55 mg/Nm³	9 05.11.98 14:30-15:06 mg/Nm³
Methanol	32 / 65	256	158	194
Ethanol	46 / 78	0.2	0.3	0.2
Acetone	58 / 56	0.8	0.7	3.2
Acetic acid	60 / 118	< 35	< 35	< 35
MEK	72 / 80	0	0	0
Propionic acid	74 / 141	0	0	0
Light tar *)	78 – 202	0	0	0

Table 22: GC analysis of light tar in aqueous condensate

*) Light tar with molar mass from 78 to 202 is calculated as isobutanol (74 g/mol)

3.2.5.2 Light tar in acetone and DCM extracts

As described in chapter 3.1.5.2, concerning measurements in raw gas, a GC analysis for determination of light tar in the clean gas was carried out, which appears from table 13.

Measurement No. Date Measurement period	Molar mass/ boiling point	7 05.11.98 10:15-10:31 mg/Nm^3	8 05.11.98 11:24-11:55 mg/Nm^3	9 05.11.98 14:30-15:06 mg/Nm^3
Benzene	78 / 80	625	410	466
Toluene	92 / 111	208	111	63
Phenol	94 / 182	95	32	9.5
Ethylbenzene	106 / 136	4.3	1.6	0.5
Xylene	106 / 139	42	19	7.6
Indene	116 / 182	310	165	103
Guaiacol	124 / 205	0	0	0
Naphthalene	128 / 218	624	486	313
4-Methyl-Guaiacol	138 / 220	0	0	0
Acenaphthylene	152 / 270	279	196	149
Acenaphthene	154 / 278	13	8.2	5.2
Fluorene	166 / 298	48	44	24
Phenanthrene	178 / 340	95	94	75
Anthracene	178 / 340	29	26	18
Fluoranthene	202 / 367	34	37	27
Pyrene	202 / 393	30	33	22
Benz(a)anthracene	228 / 400	5.3	5.4	4.0
Chrysene	228 / 410	5.6	5.8	4.4
Benz(b+k)fluoranthene	252 / 480	7.9	8.0	6.4
Benz(a)pyrene	252 / 495	5.0	5.0	4.0
Indeno(1,2,3)pyrene	276 / 500	2.8	3.3	2.6
Benz(ghi)perylene	276 / 510	2.8	3.2	2.5
Dibenz(ah)anthracene	278 / 520	0.2	0.2	0.2
Light tar (78-202)*)		4050	2717	2238

Table 23: GC analysis of light tar in acetone and DCM

*) Light tar with molar mass from 78 to 202 is calculated as anthracene (178 g/mol)

3.2.6 Total light tar in raw gas

As it appears from table 24, the light tar total is found by adding light tar to the aqueous condensate and to acetone and the DCM extracts.

Measurement No.	Date	Measurement period	Light tar in condensate mg/Nm^3	Light tar in acetone and DCM mg/Nm^3	Light tar in raw gas mg/Nm^3
7	05.11.98	10:15-10:31	0	4050	4050
8	05.11.98	11:24-11:55	0	2717	2717
9	05.11.98	14:30-15:06	0	2238	2238

Table 24: Total light tar in raw gas

3.2.7 Gravimetric measurement of heavy tar in gas after gas scrubber

As mentioned in chapter 3.2.4, tar measurements were carried out in the clean gas after the gas scrubber. The results from the evaporation of the tar fractions appear from table 25.

Measurement No.	Date	Measurement period	Residue evaporation from probe mg	Residue evaporation from cooler mg	Residue evaporation from XAD 2 mg	Amount of heavy tar in raw gas mg/Nm^3
10	06.11.98	09:00-09:20	69.7	114.3	176.6	1336
11	06.11.98	10:15-10:43	79.5	118.9	129.8	904
12	06.11.98	11:41-12:05	77.5	121.2	103.1	855

Table 25: Heavy tar in clean gas at "plant B"

3.2.8 GC measurements of light tar in gas after gas scrubber

Before evaporation of the above-mentioned extracts samples were taken and they were examined by means of a GC/MS analysis. The aqueous condensate is sampled from the DCM phase and examined by means of a GC/FID analysis.

3.2.8.1 Light tar in aqueous condensate

The water phase solely originates from the glass pipe cooler. The GC-FID analysis of the aqueous condensate from the raw gas appears from table 22.

Measurement No. Date Measurement period	Molar mass/ boiling point	10 06.11.98 09:00-09:20 mg/Nm^3	11 06. 1.98 10:15-10:43 mg/Nm^3	12 06.11.998 11:41-12:05 mg/Nm^3
Methanol	32 / 65	207	149	162
Ethanol	46 / 78	0.6	0.4	0.5
Acetone	58 / 56	0.3	0.4	0.5
Acetic acid	60 / 118	< 35	<35	< 35
MEK	72 / 80	0	0	0
Propionic acid	74 / 141	0	0	0
Light tar *)	78 – 202	0	0	0

Table 26: GC analysis of light tar in aqueous condensate

*) Light tar with molar mass from 78 to 202 is calculated as isobutanol (74 g/mol).

3.2.8.2 Light tar in acetone and DCM extracts

As described in chapter 3.1.5.2, concerning raw gas measurements, the GC analysis was carried out in order to determine the light tar in the clean gas, which appears from table 27.

Measurement No. Date Measurement period	Molar mass/ boiling point	10 06.11.98 09:00-09:20 mg/Nm³	11 06.11.98 10:15-10:43 mg/Nm³	12 06.11.98 11:41-12:05 mg/Nm³
Benzene	78 / 80	448	471	599
Toluene	92 / 111	108	97	91
Phenole	94 / 182	15	15	9.0
Ethylbenzene	106 / 136	17	1.9	1.1
Xylene	106 / 139	48	20	15
Indene	116 / 182	319	138	158
Guaiacol	124 / 205	0.0	0.0	0.0
Naphthalene	128 / 218	546	495	492
4-Methyl-Guaiacol	138 / 220	0.0	0.0	0.0
Acenaphthylene	152 / 270	198	188	181
Acenaphthene	154 / 278	9.2	7.3	8.0
Fluorene	166 / 298	41	43	35
Phenanthrene	178 / 340	63	58	57
Anthracene	178 / 340	20	96	19
Fluoranthene	202 / 367	22	23	22
Pyrene	202 / 393	21	115	21
Benz(a)anthracene	228 / 400	4.6	4.7	4.4
Chrysene	228 / 410	4.9	4.9	4.7
Benz(b+k)fluoranthene	252 / 480	7.4	7.8	7.3
Benz(a)pyrene	252 / 495	5.0	4.8	4.4
Indeno(1,2,3)pyrene	276 / 500	3.2	3.2	3.0
Benz(ghi)perylene	276 / 510	2.9	3.3	2.9
Dibenz(ah)anthracene	278 / 520	0.2	0.2	0.3
Light tar (78-202)*)		3572	2679	2555

Table 27: GC analysis of light tar in acetone and DCM

*) Light tar with molar mass from 78 to 202 is calculated as anthracene (178 g/mol)

3.2.9 Total light tar in gas after gas scrubber

As it appears from table 28, the light tar total is found by adding light tar to the aqueous condensate and to acetone and DCM extracts.

Measurement No.	Date	Measurement period	Light tar in condensate mg/Nm³	Light tar in acetone and DCM mg/Nm³	Light tar in raw gas mg/Nm³
10	06.11.98	09:00-09:20	0	3572	3572
11	06.11.98	10:15-10:43	0	2679	2679
12	06.11.98	11:41-12:05	0	2555	2555

Table 28: Total light tar in clean gas

4. Plant description

Out of consideration for the anonymity of the plants no plant description forms part of this report.

5. Operational conditions during the measurement period

In connection with measurements carried out on "plant A" on 2nd and 3rd November the output varies from 1.7 to 2.3 MW depending on the heat consumption in town. Typically, the plant starts with the greatest output in the morning and towards noon it declines to the minimum value. However, that does not influence sampling as the gas flow is constant in the bypass line.

On 3rd November, the moisture content of the fuel was increased from app. 12.00 noon and that corresponds to the declining dust content of the gas during that period. There are no comments to the operation. Figure 2 and 3 (overleaf) show the boiler output on the days of measurement and the gasification airflow and they are direct indications of the gasifier load. The numbered columns show the sampling time of the 6 measurements.

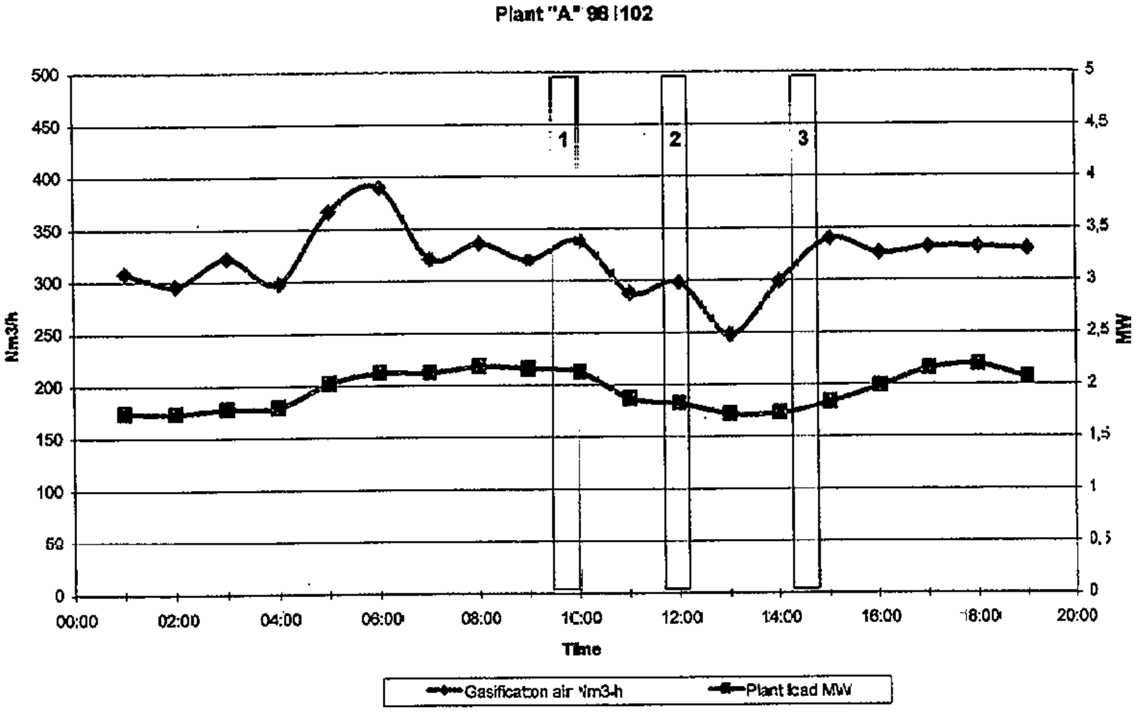


Figure 2: Operational conditions – boiler output, 2nd Nov. 1998

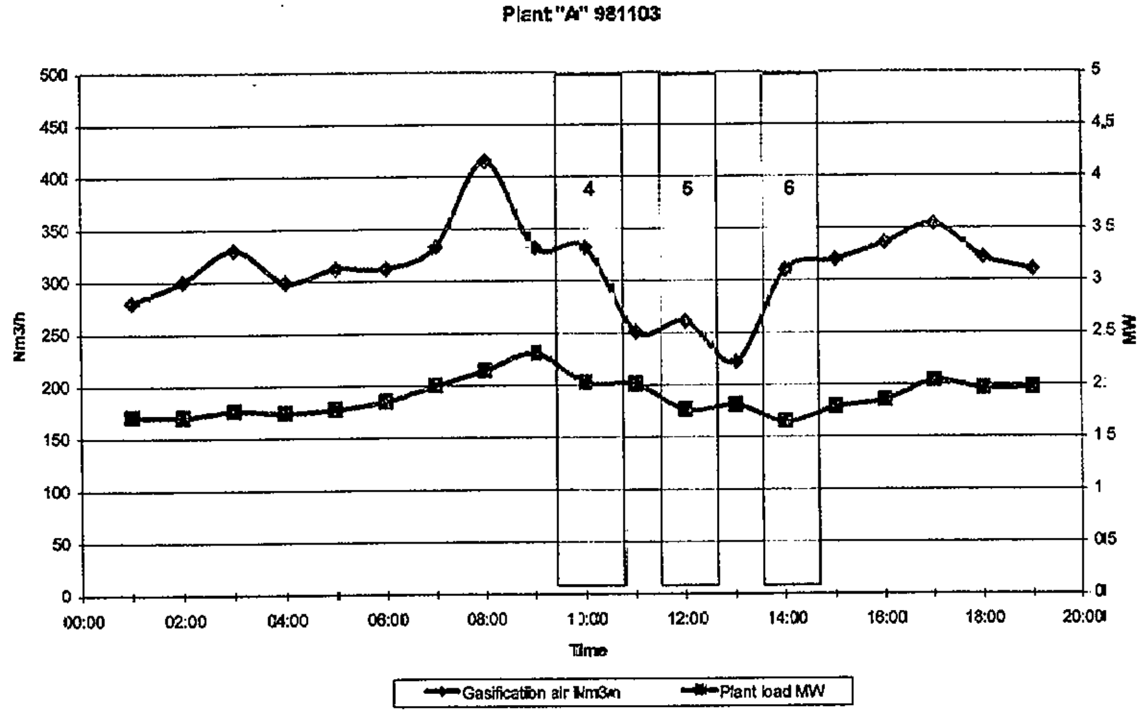


Figure 3: Operational conditions – boiler output, 3rd Nov. 1998

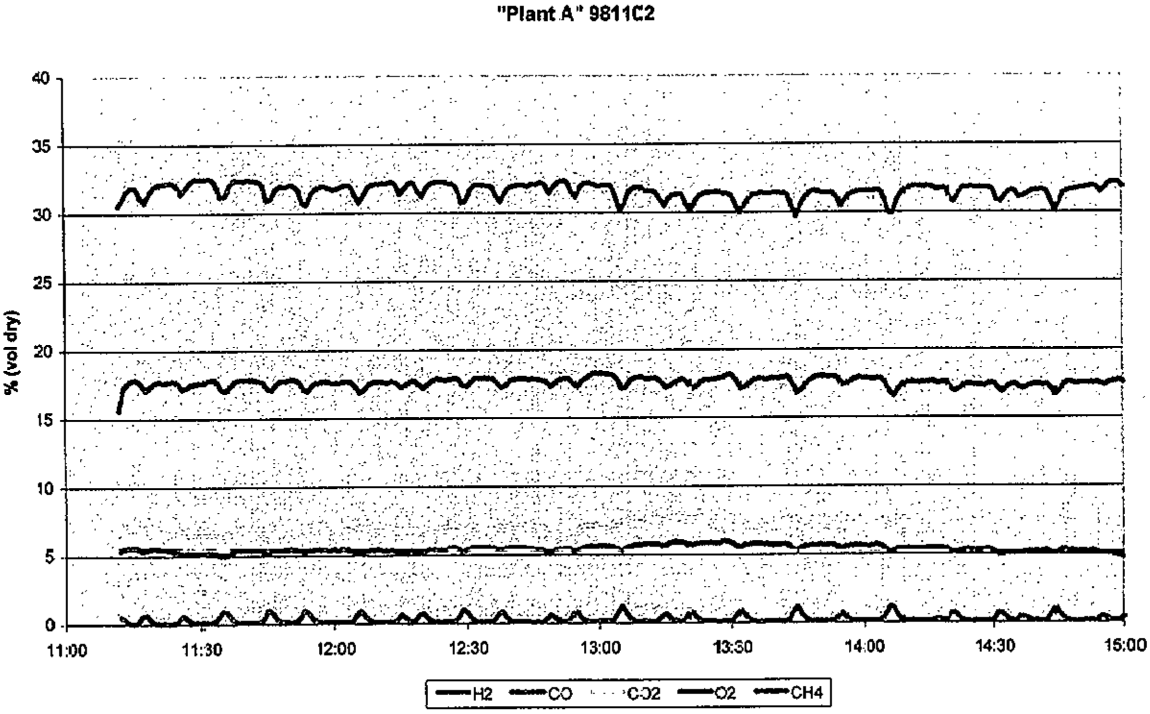
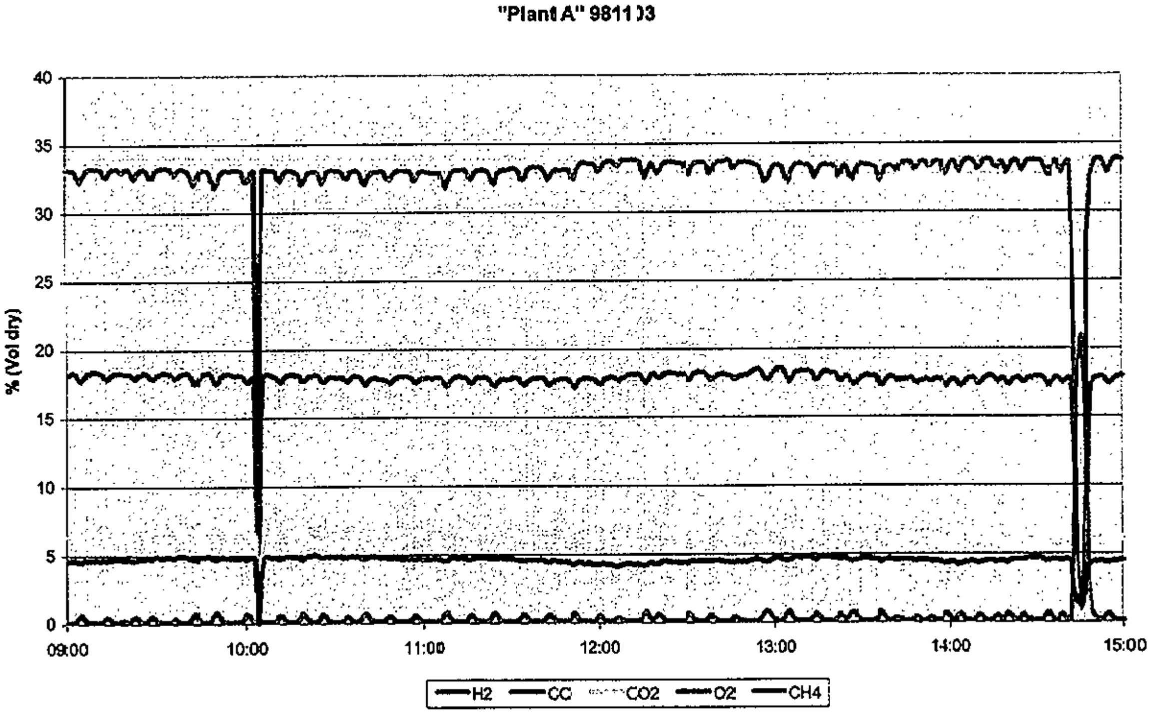


Figure 4: Gas composition, 2nd Nov. 1998



Figur 5: Gas composition, 3rd Nov. 1998

Information about the operation of "plant B" has not been stored in a computer as it was in connection with "plant A". However, the following comments have been noted in the field book.

4. 11. 1998	On arrival at "plant B" the gasifier was out of order. By accident, water had been filled on the gasifier from the washing plant. The gasifier was emptied of coal, water and ashes. The measuring equipment is set up.
5. 11. 1998	The gasifier was started during the night and operation seems normal. The relatively high tar concentration during the initial measurement at 10:15 and the declining concentration in the course of the day can presumably be traced back to the start up.
6. 11. 1998	The ash conveyor of the ash cyclone stopped 3 o'clock in the morning and the plant stopped completely at 5 o'clock in the morning. The motor cannot be restarted and at 8:20 the gasifier is started by means of the booster fan and the gas is flared. The first measurement started at 9:00 a.m. Today, there is also a clear tendency to higher tar concentrations at the beginning of the day and there is a declining tendency as the morning passes. There is no doubt that this has to do with the recent start. From the gas composition in figure 7 it appears that there is considerable methane content in the same period which also indicates an increased content of pyrolysis products in the gas.

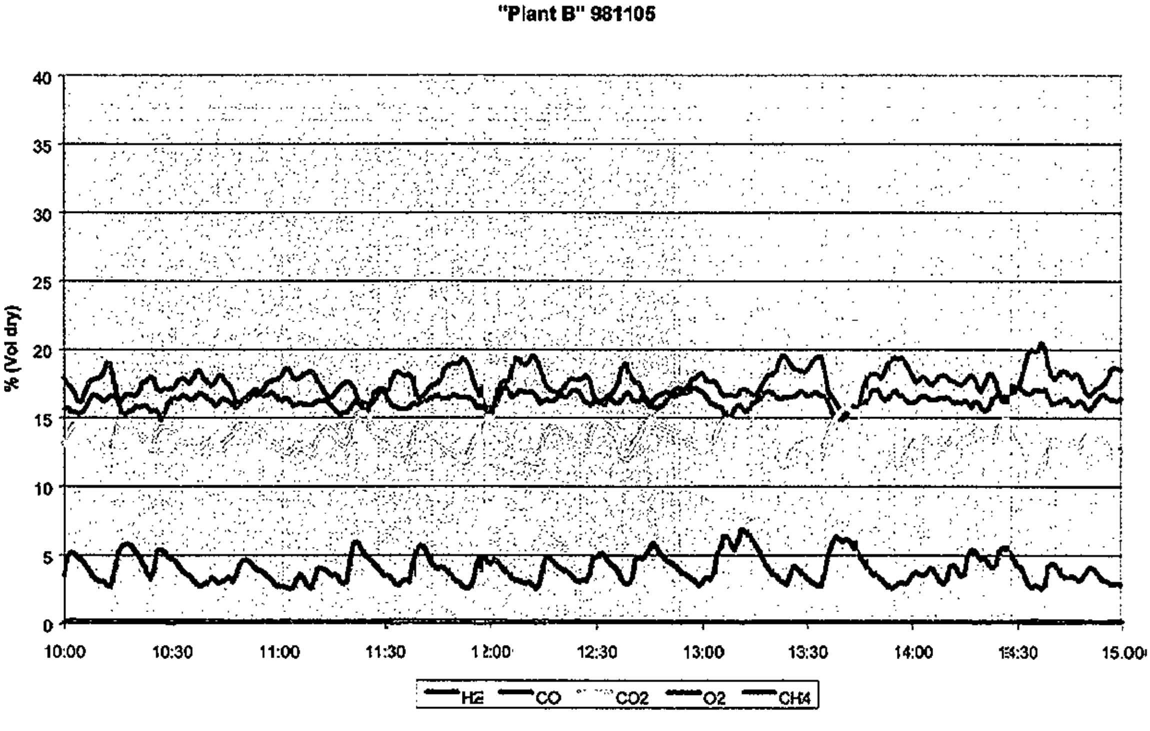


Figure 6: Gas composition, 5th Nov. 1998

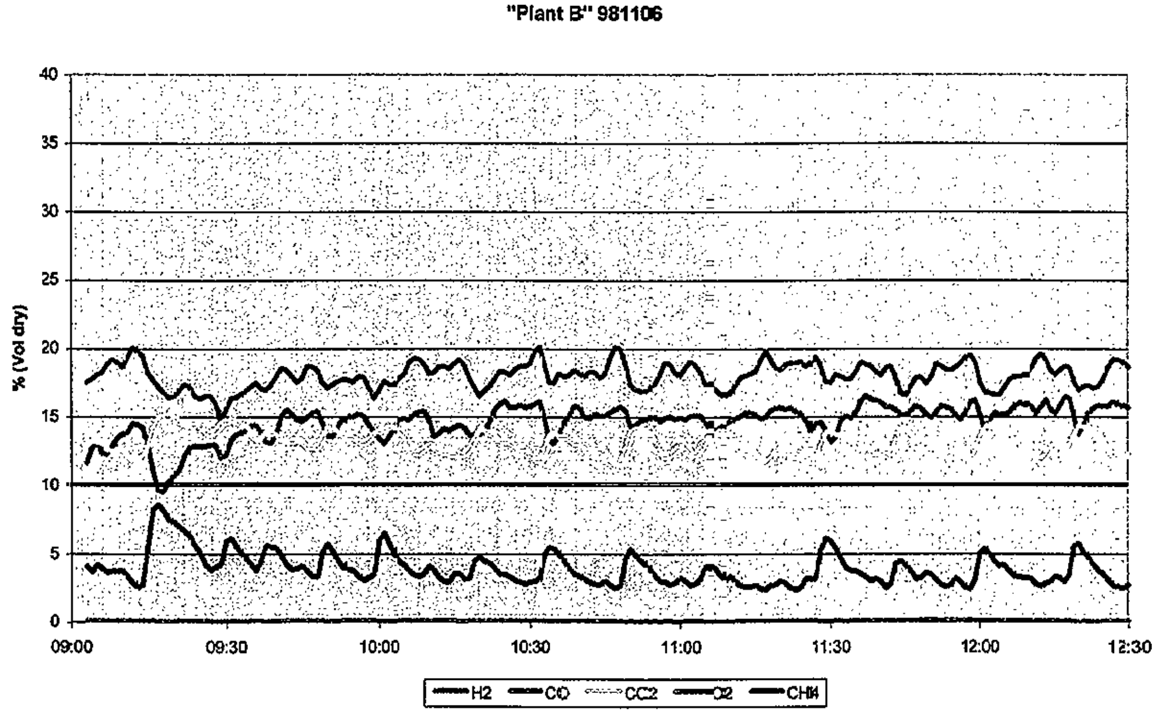


Figure 7: Gas composition, 6th Nov. 1998

6. Conclusion

DTI has carried out the 12 planned measurements and no important remarks have been written in the field book. The results reflect the normal standard of the DTI dust and tar measurements. However, the report contains more information than usual about the dust and tar measurements as we wanted to prove how much dust and tar is separated various places in the sampling system. Table 29 gives a summary of the main results of dust, heavy tar and light tar.

"Plant A"

Measurement No.	Dust mg/Nm^3	Heavy tar g/Nm^3	Light tar g/Nm^3
1	259	79	33.1
2	414	93	35.3
3	150	97	38.1
4	44	5.0	4.9
5	23	4.6	4.2
6	16	4.1	3.8

Table 29: Measurement results from "plant A"

"Plant B"

Measurement No.	Dust mg/Nm^3	Heavy tar mg/Nm^3	Light tar mg/Nm^3
7	1146	1913	4050
8	994	744	2717
9	790	561	2238
10	604	1336	3572
11	426	904	2679
12	406	855	2555

Table 30: Measurement results from "plant B"

APPENDIX 3

Report on Tar and Particulate Measurements in Biomass Producer Gases Using the modified ETH-Verenum Sampling Method

Verenum Research
Zürich, Switzerland

by Philipp Hasler
January 1999

Bundesamt für Energie
Office fédéral de l'énergie
Ufficio federale dell'energia
Uffizi federal d'energia

Biomass Programme

Report on Tar and Particulate Measurements in Biomass Producer Gases using the modified ETH / Verenum Sampling Method

Test report from the parallel sampling test runs in Denmark, November 1998

prepared by
Philipp Hasler, Verenum Research, Zürich

Zurich February 1999

Swiss Federal Office of Energy

Report on Tar and Particulate Measurements in Biomass Producer Gases using the modified ETH / Verenum Sampling Method

Test report from the parallel sampling test runs in Denmark, November 1998

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Content

1 Introduction

Producer gases from gasification reactors contain particulates and organic contaminants (tars) which, if not removed, would damage the engine or incur an unacceptable level of maintenance.

A number of different sampling and analysis methods have been used by manufacturers and other workers in this field to determine the level of these contaminants in the gas. The 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.

To overcome this barrier and to make a contribution to the future development of an international standard for sampling particulates and tars from biomass gasifiers, a series of parallel test runs have been carried out with four different sampling methods at two biomass gasification reactors in Denmark in November 1998. The four institutes/companies participating in these parallel test runs are (in alphabetical order):

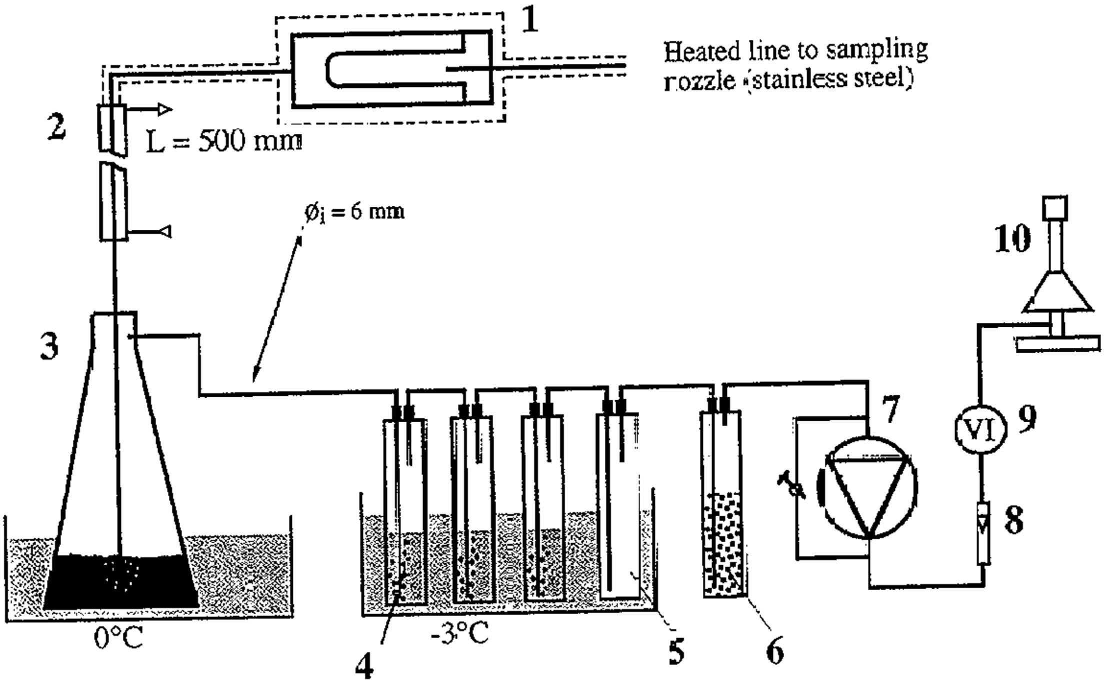
- Biomass Technology Group (BTG; Netherlands)
- Danish Technical Institute (DTI, Denmark)
- Verenum Research (Switzerland)
- VTT Energy (Finland)

This report presents the results from the test runs which have been carried out at the two Danish gasifiers using the ETH/Verenum sampling train. The method is a classical impingement train which uses anisole as a tar absorption solvent [Hasler et al. 1998a] and has been used at more than 8 different fixed bed biomass gasifiers and various gas cleaning units so far. For the test runs in Denmark, the sampling train has been slightly modified by using a particle collection in a heated filter before the onset of moisture condensation.

2 Description of the P&T sampling train

2.1 Working principle of the P&T sampling train

The sampling train used for the test runs in Denmark is a slightly modified version of the conventional ETH/Verenum particulate and tar (P&T) sampling train. A detailed description of the conventional sampling train is given in [Hasler et al. 1998a]. Except for the type of solvent used, the sampling train used in Denmark is in agreement of the proposed Version98 tar protocol of the EC / IEA / US DoE working group for small scale biomass gasifiers [Barker et al. 1998]. The sampling train is shown in figure 2-1.



1: Particle filter (heated)
2: Heat exchanger
3: Moisture condensation bottle (1 l)
4: Tar impinger bottles (app. 50 ml solvent)
5: Drop separator
6: Coke adsorber (pump protection)
7: Vacuum pump
8: Gas flow indicator
9: Gas volume integrator
10: LCV burner

figure 2-1: Schematic of the P&T sampling train used in Denmark by Verenum Research

To ensure correct particulate sampling, the sample gas stream is taken as close to isokinetic sampling conditions as possible. Since under isokinetic sampling leads to higher sampling errors than over isokinetic sampling, a 5 to 10% over isokinetic sampling flow rate is recommended. With the present sampling arrangement, flow rates up to 0.8 Nm^3/h can be sampled. The isokinetic sample flow rate is normally calculated based on the fuel composition, the gasifier load, the duct dimension and producer gas composition. For the test runs in Denmark, the sample flow rate has been given by the Danish Technical Institute (DTI).

Particles are collected in a heated filter at temperatures between 120°C and 150°C. As a filter material, glass fibre thimbles (supplied by Schleicher&Schuell; type 603g; 33 x 205 mm) are used. The total surface area of the thimble is approximately 200 cm^2. The particle filter used is a prototype design and has been tested so far for a limited number of test runs.

After the particle filter, a glass cooler of 500 mm lenght and an inner diameter of 20 mm is used. Cooling media is either ethylene glycol (-3°C) or cooling water (approx. 10°C). The condensed moisture and tars flow through a teflon tube into the moisture condensation bottle. The tube lenght between the cooler and the moisture condensation bottle is given by the actual sampling situation. For the test runs at the gasifier site 1, the tube length was approx. 0.5 m whereas at site 2 the tube lenght was approx. 5 m.

In the moisture condensation bottle additional moisture and some of the organic contaminants („tars“) will condense. Before start of the sampling, approximately 200 ml of distilled water is put in the bottle. The gas entering the water is dispersed into small bubbles by a diving pipe with an inner diameter of 4 mm. The diving pipe is at any time below the water level. The water condensing bottle is cooled to 0°C with an ice bath.

After the condensor, the gas enters a series of three tar impingers with a volume fo 250 ml (impingers of type Drechsel supplied by Merck). The impingers contain 50 ml of anisole as an absorbing liquid for organic contaminants. The diving pipe is placed not more than 5 mm above the bottle bottom and has an inner diameter of 4 mm. After the third tar impinger, a drop separator is installed. The tar impingers and the drop separator (same dimension as the impingers) are cooled to -3°C using a ethylene glycol / water mixture and a cryostatic cooling unit. At lower temperature. water freezing occurs in the diving-pipe of the first impinger.

After the drop separator, a savety coke adsorber is used as a pump protection. The adsorber is filled with approximately 200 grams lignite coke as an adsorbent.

The gas is sampled with a vaccum pump which has a nominal capacity of approx 14 m^3/h at normal pressure. The gas passes a rotameter (0-25 l/min) and a gasmeter used for volume integration over the sampling time. The gas flow rate through the pump is adjusted with a manually operated bypass ball valve. The sampled gas is combusted in a modified Bunsen burner at the end of the sampling train.

2.2 Definitions of producer gas impurities

The definitions of the contaminants in the producer gas used for the test runs made in Denmark are identical as for the conventional ETH/Verenum sampling train. The definitions of compounds which are analyzed for this report are as follows:

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 unknown. The heavy tars are determined gravimetrically by carefully evaporating the worked up solution of anisole (=methoxy benzene; t_b = 153°C) at ambient pressure.
PAH:	Polyaromatic hydrocarbons. These are organic compounds with more than two aromatic rings. Typical representatives of PAH's found in (cocurrent) biomass producer gases are: naphthalene, phenanthrene. Here, 16 PAH compounds according the the US EPA PAH list are analyzed. The determination of the PAH's is made using GC/MS
Phenols:	Aromatic organic compounds with at least one –OH group. Typical representatives are „phenol" (C_6H_5OH) 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. It has been found that this solid residue is of purely organic nature with uncontaminated wood fuels used in cocurrent gasifiers. As this organic compounds remain in the highly polar aqueous phase and are not extractable with less polar anisole, this residue is composed of highly polar tar compounds.
Particulates:	The amount of solid matter which is deposited on the glass fibre thimble and which is filtered from the liquids. „Tars" adsorbed on particulates are extracted using appropriate solvents. The determination method is gravimetric.

2.3 Postsampling and analytical procedures

The postsampling and analytical procedures are shown in figure 2-2 and figure 2-3. With minor modifications, the procedures are identical as for the conventional ETH/Verenum P&T sampling train.

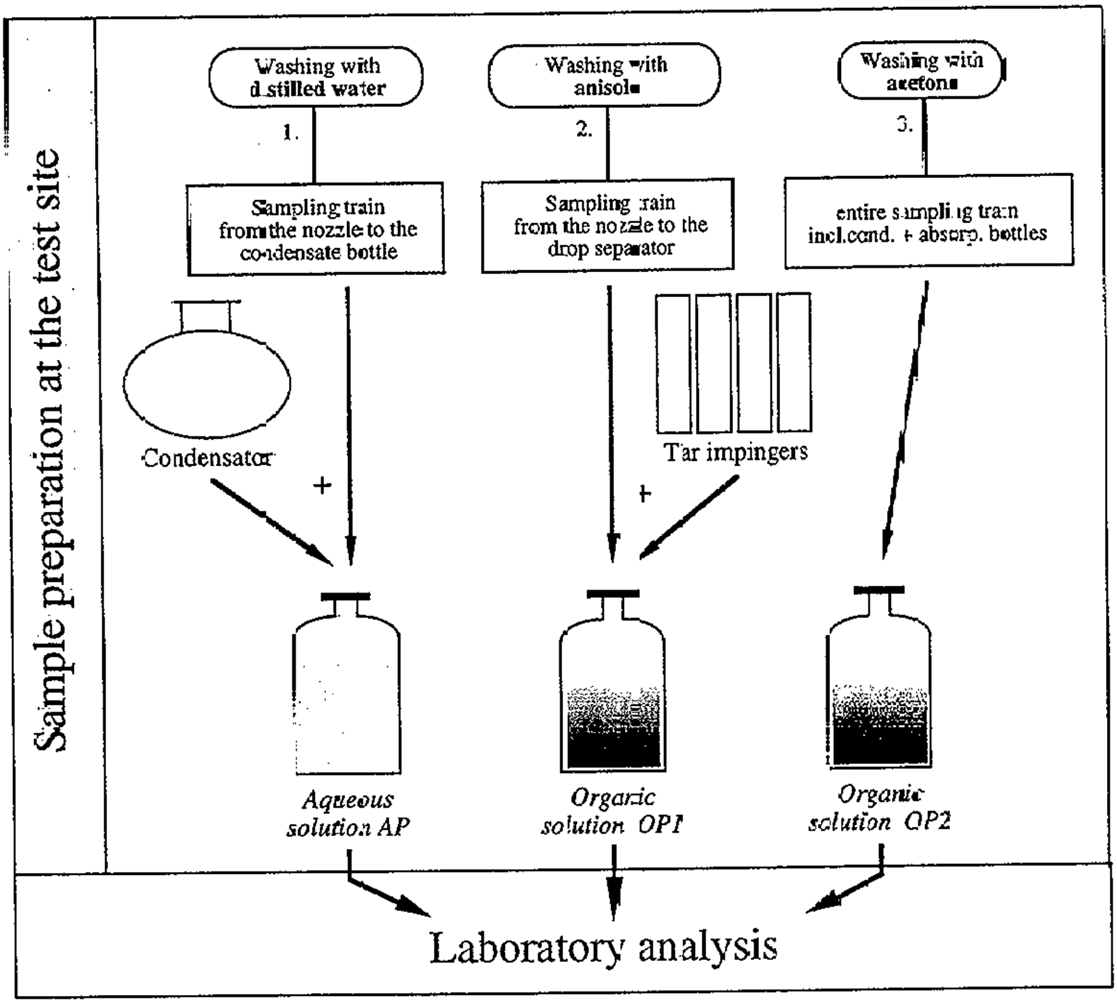


figure 2-2: Postsampling procedures for the P&T sampling train used by Verenum Research.

The cleaning of the tubes and the gas lines is made immediately after the end of the sampling. Totally, three washing procedures are made (figure 2-2). The total mass of condensate generated during the sampling period is also measured to determine the moisture content of the producer gas. The washing procedure with anisole is made after the water washing. The cleaning is made with fresh anisole. A final washing is made with acetone to remove droplets of water and anisole in the sampling train.

The result of the P&T sampling and the post sampling washing procedures are three bottles, one containing an aqeuous solution (AP) and two containing an organic solutions (OP1 and OP2), and a glass fibre thimble from the particle filter. The particle filter thimble is wrapped in aluminium foil and stored for further treatment. At sampling site 1, some thimbles were rinsed with solvent at the site. However, it turned out that tar removal is insufficient when rinsing with solvent is applied.

The analysis train in the laboratory is shown in figure 2-3. A detailed description of the sample treatment is given in [Hasler et al. 1998a].

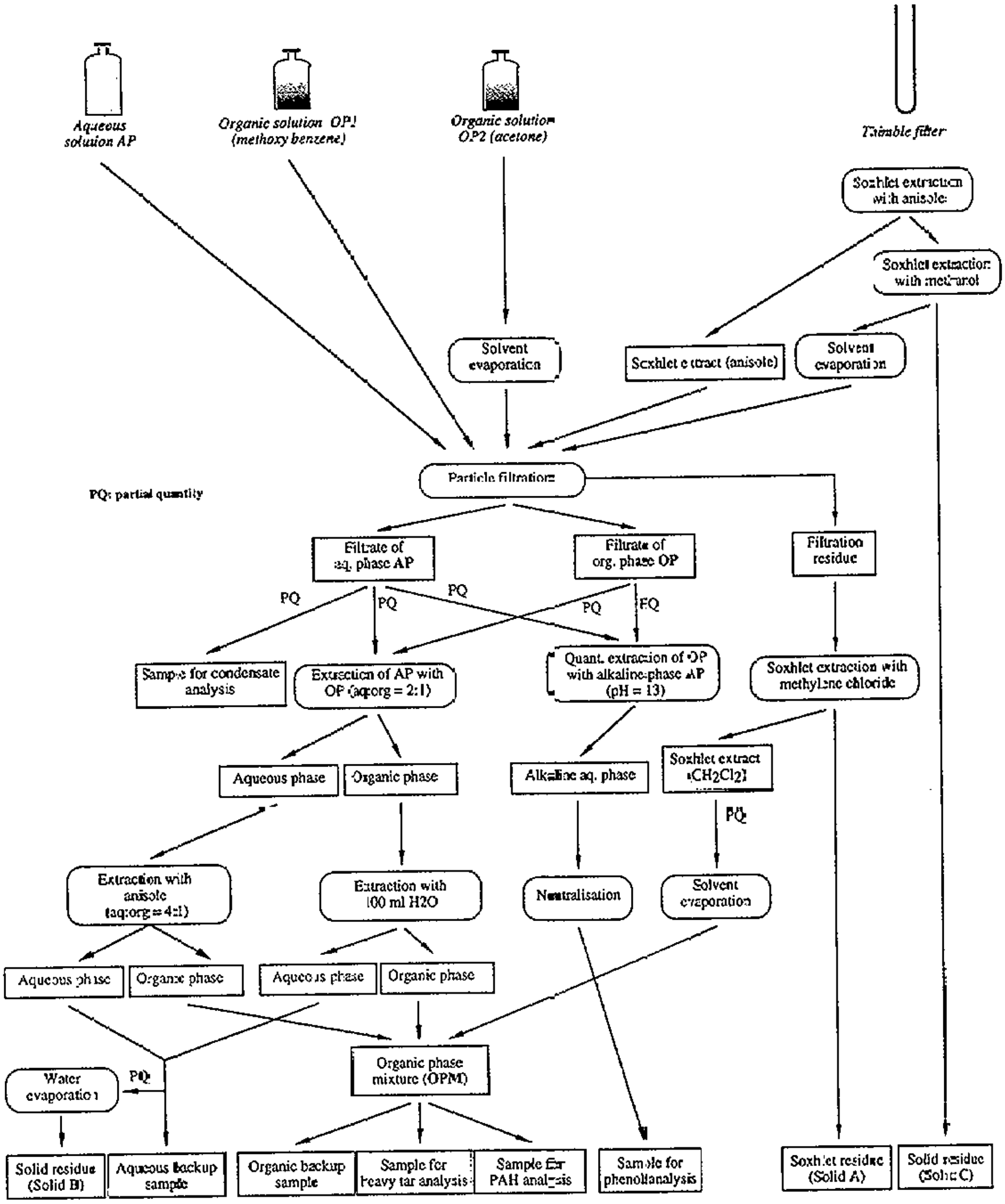


figure 2-3: Schematic of the sample treatment in the laboratory for the P&T sampling train used by Verenum Research

For the sampling train used in Denmark, additional Soxhlet extractions are made with the particle filter thimble. For the samples from site 1, Soxhlet extractions are made with anisole and methanol. For the samples from site 2, only Soxhlet extractions with anisole were made.

2.4 Methods for the determination of impurities

a) Heavy tar content

- The amount of heavy tars is determined as a gravimetric value from the heavy tar sample (see figure 2-3) by carefully distilling off the solvent anisole (t_b = 153°C). The evaporation is made in vaccum (10 to 20 mbar absolute pressure) over a heated water bath at ≅85°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.

b) PAH content

- The amount of PAH is determined quantitatively from the organic phase mixture OPM using GC/MS. The determination is made for the 16 PAH according to the EPA list. A correction factor is used for the individual PAH compounds. This correction compensates the incomplete collection of the PAH compounds in the sampling train (see also [Hasler et al .1998a]. The individual correction factors vary in the range from 1.02 to 1.17.

c) 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. In this report, the phenolic compounds are determined using the photometric method.

d) Content of the water soluble organic residue

- The solid residue B found as gravimetric value from the laboratory processing of the extracted aqueous phase. It has been found that this residue is composed of purely organic matter.

e) Particulate content

- The total amount of particulates is the sum of solids A and C determined during the laboratory processing. Particle filtration from the sampling and postsampling liquids (see figure 2-3) is made through cellulosic filters (supplier: Schleicher&Schuell; prod. no. 589/3; slow filtration velocity filter paper).

3 Experimental section

3.1 Results from the test runs at site 1 (countercurrent gasifier)

3.1.1 Tar and particulate concentrations

The summary of all data measured with the ETH/Verenum sampling method at the site of the countercurrent gasifier is given in table 3-1. Measurements have been made both in the raw and in the clean gas.

Test run		1	2	3	4
Date		2.11.1998	2.11.1998	3.11.1998	3.11.1998
Gasifier type		Countercurrent	Countercurrent	Countercurrent	Countercurrent
Sampling site		Raw gas	Raw gas	Clean gas after scrubber	Clean gas after scrubber
Start of P&T sampling	hh:mm	9:33	11:43	9:15	11:14
End of P&T sampling	hh:mm	10:03	12:14	10:17	12:18
Nozzle diameter	mm	10	10	6	6
Temperature of probe	°C	200	200	200	200
Temperature of particle filter	°C	200	200	200	120
Amount of sampled gas	Nm^3	0.335	0.328	0.781	0.808
P&T sampling duration	h	0.50	0.51	1.03	1.07
Sampling flow rate	Nm^3/h	0.675	0.640	0.755	0.758
Max. pressure drop increase in sampling train	mbar	100	70	100	50
Amount of condensate in producer gas sampled	g	203	195	70	78
Total amount of aqeous phase (EMPA)	g	604	360	320	327
Amount of anisole solution (EMPA)	g	855.2	801.8	800.7	774.4
Amount of particles found on thimble filters	mg	131	196	0	0
Amount of particles found in sampling train	mg	1022	2512	157	168
Result summary					
Moisture content of producer gas	vol%	20.1	19.5	8.0	8.8
Producer gas temperature *	°C	30	80	40	40
Velocity of sampled gas at nozzle **	m/s	4.96	4.67	9.27	9.35
Particles	mg/Nm^3	3440	8260	200	210
Water soluble organic residue	mg/Nm^3	15900	21030	1470	1320
Heavy tars	mg/Nm^3	41160	44210	2540	2740
Sum of phenols	mg/Nm^3	3130	2530	360	490
Sum of PAH	mg/Nm^3	100	51	14	11
Naphthalene	mg/Nm^3	< 0.4	< 0.4	9.1	7.3
Benzo(a)pyrene	mg/Nm^3	3.5	2.4	0.22	0.26

Remarks:
Nm^3 at 1013 mbar, 0°C, dry
* Values given by DTI
** calculated for 1013 mbar absolute pressure

table 3-1: Summary of the results from the tar and particulate measurements at gasifier site 1
Remarks: The individual sampling protocols are given in appendix 5.1; For definitions, sampling and analysis procedures see chapter 2.2

The particulate concentration in the raw gas has been found to vary between 3400 and 8300 mg/Nm^3 and in the gas 200 mg/Nm^3 of particulates have been determined. The amount of particulates found in the sampling train after the particle filtration is much higher than expected (see also chapter 3.3).

The total concentration of organic compounds (as the sum of the water soluble organic residue, the heavy tars, the sum of phenols and the PAH 1)) has been found to be approximately 60'000 mg/Nm^3 in the raw gas and approximately 4500 mg/Nm^3 in the clean gas. Compared to the heavy tar level, much lower concentrations of PAH compounds have been found. The amount of the water soluble organic residue is approximately half of the heavy tar concentration both in the raw and in the clean gas.

The distribution of the various tar values in the raw gas and in the clean gas is shown in figure 3-1. The relative distribution remains almost identical although the absolute amount is much lower in the clean gas than in the raw gas.

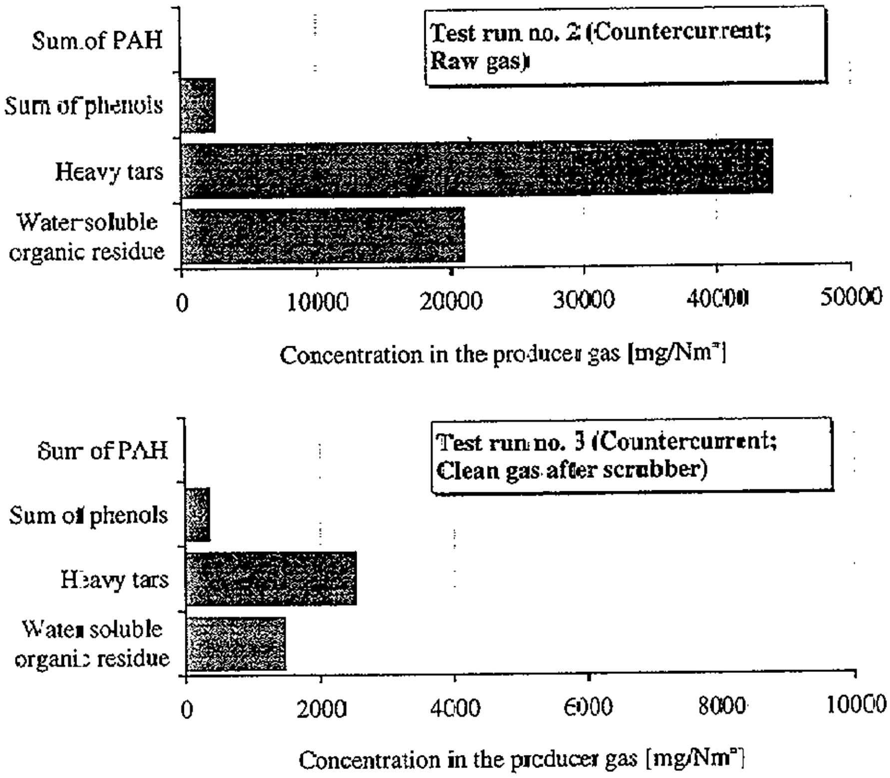


figure 3-1: Distribution of the tar values in the raw gas (top) and in the clean gas (bottom) of the countercurrent gasifier

Remarks: Definitions of tar values according to chapter 2.2

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[1] The water soluble organic residue and the heavy tars are purely additive values. For cocurrent gasifiers, 0.7 to 1.5 mass% of the total amount of phenolic compounds present in the gas was found in the water soluble organic residue and 8 to 17 mass% was found in the heavy tar residue. Hence the mass of phenolic compounds can be used as an additive value with little error. For the PAH compounds, approximately 30 weight% of the total amount present in the gas was found in the heavy tars (cocurrent gasifier).

3.1.2 PAH profiles in the countercurrent producer gas

The concentration profiles of the 16 US EPA PAH components is shown in figure 3-2 for the raw and the clean gas from the countercurrent gasifier. A table with the concentration numbers from all test runs is given in the appendix 5.3.

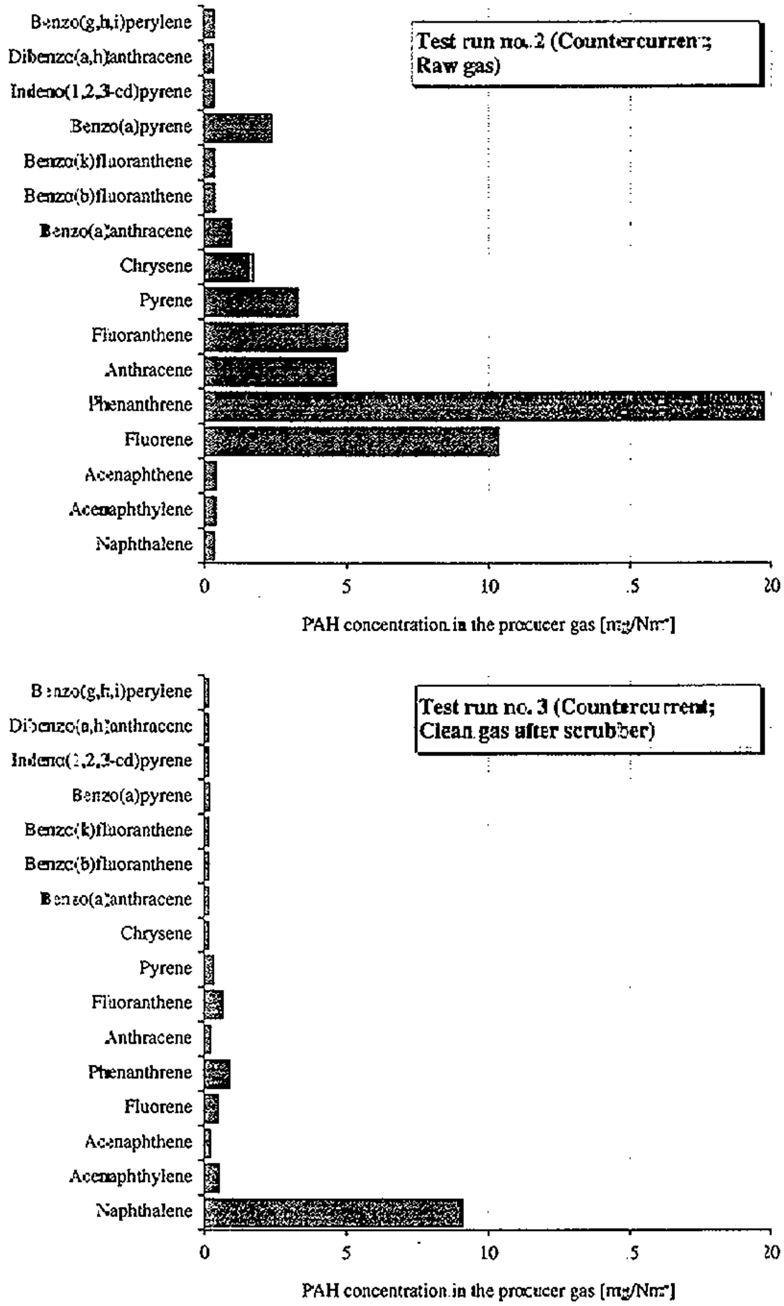

figure 3-2: PAH profiles in the raw gas (top) and the clean gas (bottom) from the countercurrent gasifier

The total concentration of the PAH compounds in the raw gas from the countercurrent gasifier amounts to 51 mg/Nm^3 (test run #2) and 100 mg/Nm^3 (test run #1) whereas in the clean gas 11 and 14 mg/Nm^3 of PAH compounds were found (test run #4 and #3).

The figures clearly show the effect of the scrubber which primarily reduces the PAH compounds with high boiling points. In the clean gas, naphthalene is the dominant PAH compound found whereas in the raw gas, the highest concentration has been observed for phenanthrene.

Surprisingly, the naphthalene concentration in the clean gas after the scrubber is found to be significantly higher than in the raw gas. Although the samplings in the raw and clean gas have not been measured simultaneously and hence some variation in the PAH composition is possible, such large differences are unlikely since all other measured organic species (heavy tars, water soluble organic residue, phenols, other PAH compounds except naphthalene) were found in the expected concentration ranges. Possibly, the low naphthalene concentration in the raw gas can be attributed to an analytical error.

3.2 Results from the test runs at site 2 (cocurrent gasifier)

3.2.1 Tar and particulate concentrations

The summary of all data measured with the ETH/Verenum sampling method at the site of the cocurrent gasifier is given in table 3-2. Measurements have been made only in the raw gas.

Test run		5	6
Date		5.11.1998	5.11.1998
Gasifier type		Cocurrent	Cocurrent
Sampling site		Raw gas	Raw gas
Start of P&T sampling	hh:mm	10:14	14:32
End of P&T sampling	hh:mm	11:32	15:06
Nozzle diameter	mm	8	8
Temperature of probe	°C	200	200
Temperature of particle filter	°C	150	150
Amount of sampled gas	Nm^3	0.810	0.233
P&T sampling duration	h	1.30	0.58
Sampling flow rate	Nm^3/h	0.623	0.403
Max. pressure drop increase in sampling train	mbar	500	150
Amount of condensate in producer gas sampled	g	118	n.d.
Total amount of aqeous phase (EMPA)	g	372	222
Amount of anisole solution (EMPA)	g	838.1	817.5
Amount of particles found on thimble filters	mg	535	62
Amount of particles found in sampling train	mg	134	91
Result summary			
Moisture content of producer gas	vol%	12.8	n.d.
Producer gas temperature *	°C	595	595
Velocity of sampled gas at nozzle **	m/s	12.54	8.12 1)
Particles	mg/Nm^3	830	660
Water soluble organic residue	mg/Nm^3	230	90
Heavy tars	mg/Nm^3	980	560
Sum of phenols	mg/Nm^3	20	6
Sum of PAH	mg/Nm^3	1383	955
Naphthalene	mg/Nm^3	834	582
Benzo(a)pyrene	mg/Nm^3	5.2	6.9

Remarks:	Nm^3	at 1013 mbar, 0°C, dry
	*	Values given by DTI
	**	calculated for 1013 mbar absolute pressure
	1)	Velocity is calculated for a gas moisture of 12.8 vol% (see test run no. 5)

table 3-2: Summary of the results from the tar and particulate measurements at gasifier site 2
Remarks: The individual sampling protocols are given in appendix 5.2; For definitions, sampling and analysis procedures see chapter 1

The particulate concentration in the raw gas has been found to vary between 670 and 830 mg/Nm^3. As with the countercurrent gasifier, more particles were found in the sampling train after the particle filtration than in the particle filter (discussion in chapter 3.3).

The distribution of the various tar values in the raw gas of the cocurrent gasifier is given in figure 3-3. The dominant tar group found are the PAH components followed by the heavy tars.

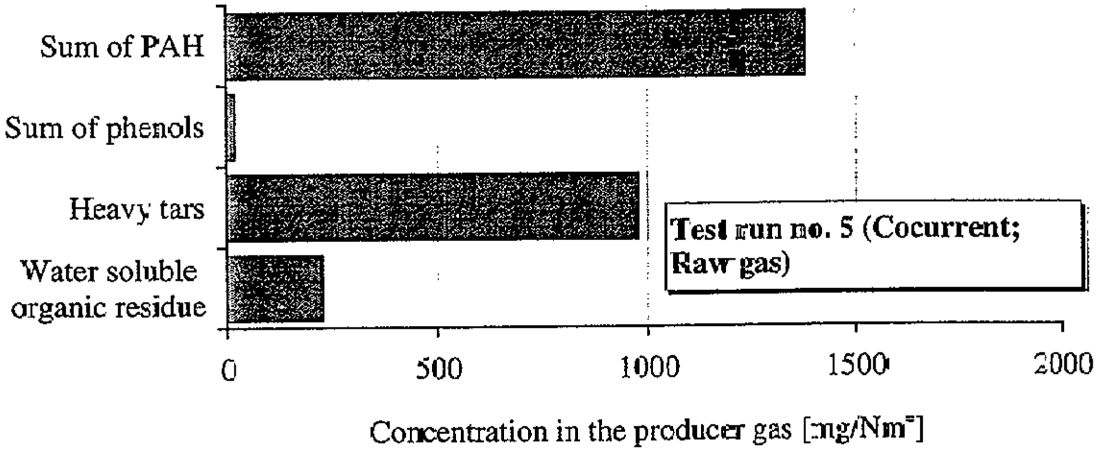

figure 3-3: Distribution of the tar values in the raw gas of the cocurrent gasifier

Remarks: Definitions of tar values according to chapter 2.2

The total concentration of organic compounds (as the sum of the water soluble organic residue, the heavy tars, the sum of phenols and the PAH; see footnote 1) has been found as 1600 mg/Nm^3 (test run #6) and 2600 mg/Nm^3 (test run #5). Between 50 wt% and 60 wt% of the total concentration of organic compounds is composed of PAH components. Rather little concentrations of phenolic compounds were detected.

3.2.2 PAH profile in the cocurrent producer gas

The concentration profile of the 16 US EPA PAH components is shown in figure 3-4 for the raw gas from the cocurrent gasifier. A table with the concentration numbers from all test runs is given in appendix 5.3. The total concentration of the PAH compounds in the raw gas from the cocurrent gasifier have been found as 950 mg/Nm^3 (test run #6) and 1380 mg/Nm^3 (test run #5). The PAH level is at least one order of magnitude higher than in the raw gas from the countercurrent gasifier.

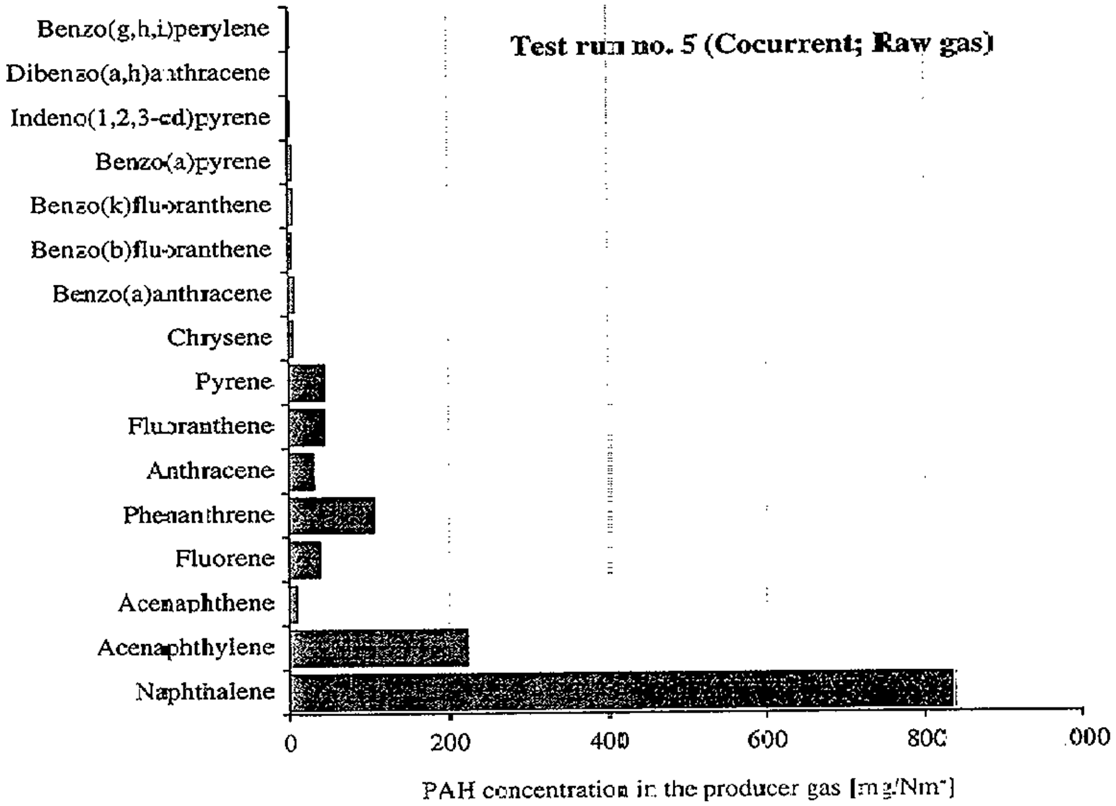

figure 3-4: PAH profile in the raw producer gas from the cocurrent gasifier

Remark: In the clean gas, no P&T sampling has been made.

3.3 Interpretation of the data

3.3.1 Particle collection in the sampling train

For the test runs in Denmark, a heated particle filter has been used prior to the condensation train (see figure 2-1). The prototype filter unit uses glass fibre thimbles which turned out to be delicate during handling. The filter has not been tested before extensively for gasification applications. It has been observed during the sampling test runs that particulates penetrated the particle filter and hence all liquids were filtered. The filter residue is then extracted with methylene chloride and the remaining solid residue is considered as particulate matter. The mass of this particulate matter is included in the determination of the particulate concentration in the gas.

With the present experience it is not clear whether the particle penetration through the thimble filter is attributed to an unproper design of the thimble holder or to unsufficient collection of aerosol particles. Also a postsampling polymerisation of organic compounds can not be excluded.

3.3.2 Tar collection in the sampling train

The collection efficiency of the sampling train has been tested for individual tar components in previous test runs [Hasler et al. 1998a, 1998b]. It has been found that 30% to 40% of the benzene present in the gas and 90% of the toluene is collected at comparable sampling conditions. The sum of the 16 US PAH compounds are collected by more than 99%. For the heavy tars and the organic residue from the aqueous phase, the collection efficiency has not been determined yet.

During the test runs in Denmark, an activated carbon filter has been used after the sampling train as a pump protection unit. This filter is solely used as a pump protection device and tar components adsorbed on this filter are not analyzed. After approximately 3 m^3 of producer gas has passed the activated carbon filter, small amounts of a sticky tar deposit has been observed at the entrance of the adsorber. The total amount of this tar deposit is roughly estimated as less than 500 mg. Hence it can be concluded that the collection efficiency of the sampling train for the heavy tars and the organic residue from the aqueous phase is not complete.

3.3.3 Comparison of counter- and cocurrent gasifier data

As expected, the countercurrent gasifier generates much higher levels of „tar" than the cocurrent gasifier. The „tar" level (as the sum of the water soluble organic residue, the heavy tars, the sum of phenols and the PAH) in the raw gas is approximately one order of magnitude higher in the countercurrent than in the cocurrent gasifier. However, a closer look reveals that the PAH concentrations are higher in the cocurrent gasifier which is a typical observation made with „matured" or tertiary tars from these type of reactor design [Milne et al. 1998].

The pattern of the PAH compounds distribution in the raw gas is quite different for the countercurrent and the cocurrent gasifier. In the cocurrent gasifier, naphthalene is the dominant PAH compound whereas in the countercurrent gasifier, a number of PAH compounds have been found in comparable concentrations (see figure 3-2).

3.4 Economics of the sampling method

The economics of a sampling method are determined by a number of parameters which can be divided into investment, operating and analytical costs. The investment cost cover the expenses for the sampling equipment. The operating cost are given by the hours of manpower used for the installation of the equipment, the time for the sampling and the dismanteling of the equipment. Furthermore, consumables used for the sampling and postsampling procedures are also included. The analytical cost cover the chemical analysis of the tar and particulate values done in the laboratories. For the method used by Verenum Research the costs are given in the following tables. All this values are solely based on the actual data from the parallel test runs in Denmark in November 1998.

The investment cost of the sampling equipment is shown in table 3-3. The total equipment cost excluding the cryostatc unit is approximatley 5500 CHF. The most costly item is the vacuum pump. For a sampling flow rate of less than 1 Nm^3/h and a pressure drop of less than 100 mbar, cheaper pumps can equally well be used.

Investment cost for sampling equipment	no. of items	Item cost
Stainless steel tubes	1	60 CHF
Nozzle	5	100 CHF
Particle filter (stainless steel)	1	600 CHF
High temperature ball valve, stainless steel	1	500 CHF
Glass heat exchanger	1	250 CHF
Condensation bottle, 1000 ml	1	26 CHF
Drop separator, 250 ml	1	28 CHF
Tar absorption bottle, 250 ml	3	28 CHF
Gast vacuum pump, model 1023 (~14 m^3/h free flowing)	1	2850 CHF
Rotameter	1	300 CHF
Gas flow integrator	1	300 CHF
Fuel gas burner	1	27 CHF
Total investment cost for sampling equipment		**5525 CHF**

table 3-3: Investment cost for the P&T sampling equipment used by Verenum Research

The time needed for the P&T sampling is presented in table 3-4 based on the actual time used during the measurements in Denmark. Travelling, transport and reporting is not included in the table. For one sample, the total hours of labour is slightly more than 7 hours when equipment installation and dismanteling is included. The average sampling duration in all of the 6 test runs is 50 minutes. The rest of the time is needed for the test preparation, onsite sample treatment and protocol reporting.

Sampling cost	no. of runs		
Installation Site 1		3.5	manhours
P&T sampling Site 1	4	14.5	manhours
Dismanteling Site 1		3	manhours
Installation Site 2		2.5	manhours
P&T sampling Site 2	2	9	manhours
Dismanteling Site 2		2.5	manhours
Sampling preparation for lab analysis (Soxhlet)	6	8.5	manhours
Total hours of labour		43.5	manhours
Total manhours per test run		**7.25**	**hours / sample**

table 3-4: Manhours used for P&T sampling during the test runs in Denmark

Remark: The hours given in this table are based on the effective time used by Verenum Research during the parallel sampling in Denmark in November 1998.

For the P&T sampling and the preliminary working up of the thimble filters (Soxhlet extractions), a glass fibre thimble and various solvents are used (table 3-5). The Soxhlet extraction with methanol is made solely for the samples from site 1 (countercurrent gasifier). A detailed description of the sampling and postsampling procedures is given in the appendix 5.1 and 5.2.

Cost of consumables	Cost	
Anisol (0.25 + 0.5 l; Tar absorption and Soxhlet extraction)	30	CHF/sample
Aceton (0.25 l rinsing)	7	CHF/sample
Methanol (0.3 l; Soxhlet extraction)	5	CHF/sample
Glass thimble filter (particle collection)	8	CHF/sample
Total consumables per test run	**50**	**CHF/sample**

table 3-5: Cost of consumables for the P&T method used by Verenum Research

All the chemical analysis of the samples generated from the P&T sampling and postsampling procedures is done in external laboratories. The laboratories involved are approved and follow the regulations set by the Swiss Testing Service STS. The costs are given in table 3-6. The upgrading of the samples to determine the concentrations of the producer gas impurities follow the scheme displayed in figure 2-3 and include various extraction procedures.

Chemical analysis cost	Cost	
Cost of laboratory analysis (particles, heavy tars, water soluble organics)	600	CHF/sample
Cost of photometric phenol analysis:	100	CHF/sample
Cost of PAH analysis:	266	CHF/sample
Total analysis cost	**966**	**CHF/sample**

table 3-6: Cost of the chemical analysis of the samples generated during P&T sampling

4 Conclusions

The concentrations of the producer gas contaminants which have been determined with the slightly modified ETH /Verenum sampling train and the analytical treatment developped thereof showed no unexpected results both for the test runs made at the countercurrent and the cocurrent gasifier.

Although the sampling durations were in the range of less than 1 hour, no observations were made which indicated sampling problems for longer sampling periods even for the countercurrent gasifier.

The preseparation of particles in a heated filter was possible and caused no prohibitive pressure drop. However, a significant amount of particulates (nonextractable solid mass) was found after the particle filter in all test runs.

The working up of the particle filter thimbles from the countercurrent gasifier turned out to be rather delicate and asked for an extra extraction procedure. Onsite Soxhlet extraction of the filter tubes is considered as non feasible due to the time required for complete extraction.

The relatively high analysis cost of this method is caused by the time required for the sample preparation in the lab and by the large number of parameters determined.

5 Appendix

5.1 Protocols from site 1 (countercurrent gasifier)

5.1.1 Sampling protocol of test run #1 (V1 raw)

P+T prot., V1 raw (DK)

P+T - Measurement for Biomass Gasifiers

Examinators: Fh.Hasl
Gasifier Type/Site: Countercurrent gasifier (updraft) Date: 2.11.1998
Test No.: 1

Gasifier parameter

Actual power output: ______ KW
(based on fuel fuel input)
Raw gas flow rate (dry): 0 [Nm³/h] (if calc., spec. gas rate: 2.60 Nm³/kg wet
Gasifier start *: permanent stationary since *: ______
Fuel type *: Native wood chips
Fuel moisture *: 100 [%, dry basis]
Actual fuel feeding rate *: ______ [kg wet/h]

Sampling parameters

Sampling site (hot/cold end): Raw gas
Ambient temperature: 10 [°C]
Ambient pressure: 1014 [mbar]
Duct diameter: 21.3 cm
Diameter of sampling nozzle: 10 mm
Sampling tube temp. before filter: 200 [°C] Heating defect after 10 min (T at end app.140°C).
Particle filter 200 [°C]

	tara weight	netto weight	final weight 1	final weight 2	
Water condensation bottle:	452.2	236.6	891.5	———	[g]
Condensate storing bottle:	992.5	———	———	1578.9	[g]
Anisole (impinger) storing bottle:	368.6	———	———	676.5	[g]
Acetone storing bottle:	372.1	———	———	458.2	[g]
Filter tube (no 1):	5.4260	———	5.5573	———	[g]

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*

Summary protocol

Generated condensate mass: 202.7 [g]
Total mass of water: 586.4 [g]
Total mass of anisole: 307.9 [g]
Total mass of acetone: 86.1 [g] (acetone: cleaning solvent)
Total particle mass in filter tubes: 131 [mg]
Normalized sampled gas flow rate 0.675 [Nm³/h] (set point: 0 Nm3/h; 10% over isokinetic)

Remarks:
-Values given by the gasifier operators are marked with *.
- Sampling nozzle was mounted perpendicular to the gas flow
(hence particulate sampling will not be correct)

protocol 1-3 *Verenum, Zürich (16.6.1999)*

P+T prot., V1 raw (DK)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Countercurrent gasifier (updraft)

Sampling site (hot/cold end): Raw gas

Date: 2.11.1998

Test No.: 1

Rated sampling gas flow rate (dry): 0.000 Nm³/h (calculated set point; 10% over isokinetic)

P+T sampling start: 9:33 P+T sampling end: 10:03

Gas meter protocol

Time [hh:mm]	Gas temperature in duct [°C]	Reading gas meter [m³]	Temperature in gas meter [°C]	Δp pump [mbar]	Sampling Flow rate [Nm³/h]
9:33:45	80	73.666	18.5	100	--------
9:37:45		73.717	18.6	100	0.717
9:47:15		73.840	19.1	100	0.727
9:54:15		73.921	19.6	50	0.648
10:03:30		74.024	20.6	50	0.622

Remarks:

- Particle filter heated to 200°C.
- Condensation train: cooler (-3°C), condensator (0°C), no drop separator.
- Tar absorption train: 3 washbottles with anisole (bottles of type Drechsel, -3°C), drop separator
- Before test run, heated sampling nozzle was cleaned by N_2 flushing
- In the cooler, a black deposit is found (most probably tar)

Averaged sample flow rate:	0.675 Nm³/h	#DIV/0
Total amount of gas sampled:	0.335 Nm³	
Total sampling duration:	0.50 hours	
Mean gas meter temperature:	19.3 °C	

Calculation of the gas dew point

Total amount of condensed moisture:	202.7 g	
water vapour equivalent	0.252 Nm³	20.1 Vol.-%
water content in producer gas	606 g/Nm³ dry	
water vapour pressure	0.430 bar	
Calculated dew point of the gas:	77.7 °C	

P+T prot., V'l raw (DK)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Countercurrent gasifier (updraft) Date: 2.11.1998
Sampling site (hot/cold end): Raw gas
Test No.: 1

Particle filter pretreatment

Filter tube no.: 1
Preconditioning temperature: 270 °C
Preconditioning duration: 3 hours
Initial tube weight after preconditioning: 5.4260 g

Particle filter tar extraction procedure

Cold extraction with anisole

Start: 11:15:00 hh mm
End: 12:45:00 hh mm
Duration: 1.50 hours

	tara weight	brutto weight	netto weight	
Anisole cold extract bottle:	369.2	566.2	197.0	[g]
Acetone rinsing bottle*	368.3	490.4	122.1	[g]

Remarks: * Aceton rinsingsolution originates from particle filter housing and cold anisol extraction process.
After the anisol extraction, the thimble remains black (insufficient tar removal).
Thimble is packed in alufoil for further treatment.

Soxhlet extraction with anisole

Start: 11:30:00 hh:mm
End: 9:15:00 hh:mm
Duration: 21.8 hours

	tara weight	brutto weight	netto weight	
Anisole extract bottle	369.2	902.3	533.1	[g]

Remarks: The Soxhlet extraction is made with the anisole cold extraction solution with additional fresh anisole to makeup. The Soxhlet is made at ambient pressure.
The acetone rinsing solution is slowly evaporated and transferred to the final storage bottle.
Unfortunately, the acetone solution is lost during the evaporation process.
The anisole in the Soxhlet extractor is almost colourless after 22 hours of extraction.
However, the filter tube remains black at the outer surface. The thimble has an intense sweet smell (sugar like). Hence some tar components may not have been extracted.

Soxhlet extraction with methanol

Start: 8:30:00 hh:mm
End: 9:00:00 hh:mm
Duration: 24.5 hours

	tara weight	brutto weight	netto weight	
Methanol extract bottle	372.6	723.9	351.3	[g]

Remarks: Already the cold methanol solution at the beginning has an intense brown/reddish colour.
By the extraction process, the thimble colour turns from dark black to light brown.
The methanol solution in the Soxhlet extractor is colourless after 24 hours of extraction.

Particle filter posttreatment

Postconditioning temperature: 175 °C
Postconditioning duration: 1.5 hours
Final tube weight after postconditioning: 5.5573 g

Remarks:

5.1.2 Sampling protocol of test run #2 (V2 raw)

P+T prot., V2 raw (DK)

P+T - Measurement for Biomass Gasifiers

Examinators: Ph.Hasler

Gasifier Type/Site: Countercurrent gasifier (updraft) Date: 2.11.1998

Test No.: 2

Gasifier parameter

Actual power output: kW
(based on fuel fuel input)

Raw gas flow rate (dry) 0 [Nm³/h] (if calc., spec. gas rate: 2.63 Nm³/kg wet

Gasifier start *: permanent operation stationary since *:

Fuel type *: Native wood chips

Fuel moisture *: 100 [%, dry basis]

Actual fuel feeding rate *: [kg wet/h] (side stream of gasifier outlet)

Sampling parameters

Sampling site (hot/cold end): Raw gas

Ambient temperature: 10 [°C]

Ambient pressure: 1014 [mbar]

Duct diameter: 21.3 cm

Diameter of sampling nozzle: 10 mm

Sampling tube temperature before filter: 200 [°C]

Particle filter temperature: 200 [°C]

	tara weight	netto weight	final weight 1	final weight 2	
Water condensation bottle:	467.8	77.9	741	—	[g]
Condensate storing bottle:	994	—	—	1360.7	[g]
Anisole (impinger) storing bottle:	367.2	—	—	623.8	[g]
Acetone storing bottle:	368.1	—	—	429.5	[g]
Filter tube (no 4):	4.6882	—	4.884	—	[g]

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*

Summary protocol

Generated condensate mass: 195.3 [g]

Total mass of water: 366.7 [g]

Total mass of anisole: 256.6 [g]

Total mass of acetone: 61.4 [g] (acetone: cleaning solvent)

Total particle mass in filter tube: 196 [mg]

Normalized sampled gas flow rate: 0.640 [Nm³/h] (set point: 0 Nm3/h; 10% over isokinetic)

Remarks:
- Values given by the gasifier operators are marked with *.
- Sampling nozzle was mounted perpendicular to gas flow

P+T prot., 1/2 raw (DH)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Countercurrent gasifier (updraft)

Sampling site (hot/cold end): Raw gas Date: 2.11.1998

Test No.: 2

Rated sampling gas flow rate (dry): 0.000 Nm³/h (calculated set point; 10% over isokinetic)

P+T sampling start: 11:43 P+T sampling end: 12:14

Gas meter protocol

Time [hh:mm]	Gas temperature in duct [°C]	Reading gas meter [m³]	Temperature in gas meter [°C]	Δp pump [mbar]	Sampling Flow rate [Nm³/h]
11:43:30	80	74.025	18.6		--------
11:48:30		74.038	18	70	0.704
12:04:45		74.276	18.3		0.653
12:14:15		74.375	18.5	70	0.583

Remarks:

- Preseparation of particles in filter (glass fibre thimbles) heated to 200°C
- Condensation train: cooler (-3°C), condensator (0°C), no drop separator.
- Tar absorption train: 3 washbottles with anisole (bottles of type Drechsel, -3°C), drop separator and safety tar absorber filled with lignite coke
- Before test run, heated sampling nozzle was cleaned by N_2 flushing.

Averaged sample flow rate:	0.640 Nm³/h	#DIV/0!
Total amount of gas sampled:	0.328 Nm³	
Total sampling duration:	0.51 hours	
Mean gas meter temperature:	18.4 °C	

Calculation of the gas dew point

Total amount of condensed moisture:	195.3 g	
water vapour equivalent	0.243 Nm³	19.5 Vol.-%
water content in producer gas	596 g/Nm³ dry	
water vapour pressure	0.426 bar	
Calculated dew point of the gas:	77.5 °C	

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Countercurrent gasifier (updraft) Date: 2.11.1998
Sampling site (hot/cold end): Raw gas
Test No.: 2

Particle filter pretreatment

Filter tube no.: 4
Preconditioning temperature: 270 °C
Preconditioning duration: 3 hours
Initial tube weight after preconditioning: 4.6882 g

Particle filter tar extraction procedure

Cold extraction with anisole

Start: 14:00:00 hh:mm
End: 14:45:00 hh:mm
Duration: 0.75 hours

	tara weight	brutto weight	netto weight	
Anisole cold extract bottle:	370.5	421.2	50.7	[g]
Acetone rinsing bottle *:	360.4	408.2	[illegible]	[g]

Remarks: * Aceton rinsingsolution originates from particle filter housing and cold anisol extraction process.
After the anisol extraction, the thimble remains black (insufficient tar removal).
Thimble is packed in alu foil for further treatment.

Soxhlet extraction with anisole

Start: 10:45:00 hh:mm
End: 12:00:00 hh:mm
Duration: 25.3 hours

	tara weight	brutto weight	netto-weight	
Anisole extract bottle	370.5	892.4	521.9	[g]

Remarks: The Soxhlet extraction is made with fresh anisole and at ambient pressure.
The acetone rinsing solution is slowly evaporated and transferred to the final storage bottle.
The anisol solution in the extractor housing is colourless after 25.3 hours of extraction.
However, the filter tube remains black at the outer surface. The thimble has an intense
sweet smell (sugar like). Hence some tar components may not have been extracted.
Thimble is packed in alu foil for further treatment.

Soxhlet extraction with methanol

Start: 9:30:00 hh:mm
End: 9:45:00 hh:mm
Duration: 24.3 hours

	tara weight	brutto weight	netto weight	
Methanol extract bottle	367.1	672.9	305.8	[g]

Remarks: The Soxhlet extraction is made at ambient pressure.
The methanol solution in the extractor housing is colourless after 24.3 hours of extraction.
The thimble has a brownish colour after the extraction.

Particle filter posttreatment

Postconditioning temperature: 175 °C
Postconditioning duration: 1.5 hours
Final tube weight after postconditioning: 4.884 g

Remarks:

5.1.3 Sampling protocol of test run #3 (V3 clean)

P+T prot. 1-3 clean (XX)

P+T - Measurement for Biomass Gasifiers

Examinators: Ph.Hasler

Gasifier Type/Site: Countercurrent gasifier (updraft) Date: 3.11.1993

Test No.: 3

Gasifier parameter

Actual power output: ______ kW
(based on fuel fuel input)

Raw gas flow rate (dry): 0 [Nm³/h] (if calc., spec. gas rate: 2.63 Nm³/kg wet

Gasifier start *: permanent operation stationary since *:

Fuel type *:

Fuel moisture *: 100 [%, dry basis]

Actual fuel feeding rate *: ______ [kg wet/h] (side stream of gasifier outlet)

Sampling parameters

Sampling site (hot/cold end): Clean gas after scrubber

Ambient temperature: 5 [°C]

Ambient pressure: 1002 [mbar]

Duct diameter: 10.8 cm

Diameter of sampling nozzle: 6 mm

Sampling tube temperature before filter: 200 [°C]

Particle filter temperature: 200 [°C]

	tara weight	netto weight	final weight 1	final weight 2		
Water condensation bottle:	465.9	145	681.1	——	[g]	
Concensate storing bottle:	994.2	——	——	1317.3	[g]	
Anisole storing bottle:	366.6	——	——	676.4	[g]	
Acetone storing bottle:	364.3	——	——	462	[g]	4[illegible].8 incl. acetone wash from
Filter tube (no 6):	4.3754	——	4.2792	——	[g]	Soxhlet extractor

Netto weight: *Mass of water initially filled in the condensator bottle*
Final weight 1: *Total mass of bottle plus liquid after sampling, excl. washing solutions*
Final weight 2: *Mass after sampling, incl. washing solutions*

Summary protocol

Generated condensate mass: 70.2 [g]

Total mass of water: 323.1 [g]

Total mass of anisole: 309.8 [g]

Total mass of acetone: 97.7 [g] (acetone: cleaning solvent)

Total particle mass in filter tube: 0 (mg)

Normalized sampled gas flow rate: 0.755 [Nm³/h] (set point: 0 Nm3/h 10% over isokinetic)

Remarks: -Values given by the gasifier operators are marked with *.

P+T prot., V3 clean (D.S)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Countercurrent gasifier (updraft)

Sampling site (hot/cold end): Clean gas after scrubber

Date: 3.11.1998

Test No.: 3

Rated sampling gas flow rate (dry): 0.000 Nm³/h (calculated set point: 10% over isokinetic)

P+T sampling start: 9:15 P+T sampling end: 10:17

Gas meter protocol

Time [hh:mm]	Gas-temperature in duct [°C]	Reading gas meter [m³]	Temperature in gas meter [°C]	Δp pump [mbar]	Sampling Flow rate [Nm³/h]
9:15:00	40	74.374	11.4		--------
9:20:30		74.440	11.1	70	0.679
9:30:30		74.558	11.4	70	0.675
9:52:00		74.832	12	70	0.725
10:10:45		75.109	12.1	100	0.840
10:17:00		75.197	12.3	100	0.800

Remarks:

- Preseparation of particles in filter (glass fibre thimbles) heated to 200°C
- Condensation train: cooler (-3°C), condensator (0°C), no drop separator.
- Tar absorption train: 3 washbottles with anisole (bottles of type Drechsel, -3°C), drop separator and savety tar adsorber filled with lignite coke
- Before test run, heated sampling nozzle was cleaned by N_2 flushing.
- In the washing bttle, a white mist builds up (tar aerosols formed in scrubber ?).

Averaged sample flow rate:	0.755 Nm³/h	#DIV/0!
Total amount of gas sampled:	0.781 Nm³	
Total sampling duration:	1.03 hours	
Mean gas meter temperature:	11.7 °C	

Calculation of the gas dew point

Total amount of condensed moisture:	70.2 g	
water vapour equivalent	0.087 Nm³	8.0 %moisture in gas
water content in producer gas	90 g/Nm³ dry	
water vapour pressure	0.101 bar	
Calculated dew point of the gas:	46.2 °C	

P+T prot., V3 clean (DK)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Countercurrent gasifier (updraft) Date: 3.11.1998

Sampling site (hot/cold end): Clean gas after scrubber

Test No.: 3

Particle filter pretreatment

Filter tube no.: 6

Preconditioning temperature: 270 °C

Preconditioning duration: 3 hours

Initial tube weight after preconditioning: 4.3754 g

Particle filter tar extraction procedure

Cold extraction with anisole

Start: ________ hh:mm

End: ________ hh:mm

Duration: ________ hours

	tara weight	brutto weight	netto weight	
Anisole cold extract bottle *:	371.9	454.8	82.9	[g]
Aceton rinsing bottle **:	304.7	450.0	86.2	[g]

Remarks:

* Anisole is used only for the cleaning of the particle filter housing.

** Aceton rinsing solution originates from particle filter housing cleaning process.

Thimble is packed in alu foil for further treatment.

Soxhlet extraction with anisole

Start: 13:00:00 hh:mm

End: 12:00:00 hh:mm

Duration: 47.0 hours

	tara weight	brutto weight	netto weight	
Anisole extract bottle	371.9	848.3	476.4	[g]

Remarks:

The Soxhlet extraction is made with fresh anisole and at ambient pressure.

The acetone rinsing solution is slowly evaporated and transferred to the final storage bottle.

The anisole in the Soxhlet thimble housing is almost colourless after 47 hours of extraction.

The filter tube thimble has a light brown colour at the outer surface.

Thimble is packed in alu foil for further treatment.

Soxhlet extraction with methanol

Start: 10:45:00 hh:mm

End: 15:45:00 hh:mm

Duration: 5.0 hours

	tara weight	brutto weight	netto weight	
Methanol extract bottle	360.4	699.9	339.5	[g]

Remarks:

The Soxhlet extraction is made at ambient pressure.

The thimble immediately turns completely white when fresh methanol is filled in the extractor.

The methanol in the Soxhlet thimble housing is colourless after 5 hours of extraction.

Particle filter posttreatment

Postconditioning temperature: 175 °C

Postconditioning duration: 1.5 hours

Final tube weight after postconditioning: 4.2792 g

Remarks: ________

5.1.4 Sampling protocol of test run #4 (V4 clean)

P+T prot., V4 clean (OK)

P+T - Measurement for Biomass Gasifiers

Examinators: Ph. Hasler

Gasifier Type/Site: Countercurrent gasifier (updraft) Date: 8.12.1998

Test No.: 4

Gasifier parameter

Actual power output: ______ kW (max. 10000 kW)
(based on fuel fuel input)

Raw gas flow rate (dry): 0 [Nm³/h] (if calc., spec. gas rate: 2.63 Nm³/kg wet

Gasifier start *: permanent operation stationary since *: ______

Fuel type *: ______

Fuel moisture *: 100 [%, dry basis]

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

Sampling parameters

Sampling site (hot/cold end): Clean gas after scrubber

Ambient temperature: 5 [°C]

Ambient pressure: 1002 [mbar]

Duct diameter: 10.8 cm

Diameter of sampling nozzle: 6 mm

Sampling tube temperature before Filter: 200 [°C]

Particle filter temperature: 120 [°C]

	tara weight	netto weight	final weight 1	final weight 2	
Water condensation bottle:	452.8	108.3	638.6	______	[g]
Condensate storing bottle:	994.6	______	______	1325.6	[g]
Anisole storing bottle:	368.8	______	______	664.7	[g]
Acetone storing bottle:	257.2	______	______	294.3	[g]
Filter tube (no 5):	4.5831	______	4.583	______	[g]

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*

Summary protocol

Generated condensate mass: 77.5 [g]

Total mass of water: 331 [g]

Total mass of anisole: 295.9 [g]

Total mass of acetone: 37.1 [g] (acetone: cleaning solvent)

Total particle mass in filter tube: 0 [mg]

Normalized sampled gas flow rate: 0.758 [Nm³/h] (set point 0 Nm3/h; 10% over isokinetic)

Remarks: -Values given by the gasifier operators are marked with *.

P+T prot. /4 clean (DX)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site:	Countercurrent gasifier (updraft)		
Sampling site (hot/cold end):	Clean gas after scrubber	Date:	3.11.1998
Test No.:	4		

Rated sampling gas flow rate (dry): 0.000 Nm³/h (calculated set point; 10% over isokinetic)

P+T sampling start: 11:14 P+T sampling end: 12:18

Gas meter protocol

Time [hh:mm]	Gas temperature in duct [°C]	Reading gas meter [m³]	Temperature in gas meter [°C]	Δp pump [mbar]	Sampling Flow rate [Nm³/h]
11:14:45	40	75.198	12.4		-------
11:28:00		75.382	12.4	50	0.788
11:49:15		75.666	12.7	50	0.758
12:05:45		75.883	12.6	50	0.746
12:18:45		76.053	12.5		0.740

Remarks:

- Preseparation of particles in filter (glass fibre thimbles) heated to 120°C
- Condensation train: cooler (-3°C), condensator (0°C), no drop separator.
- Tar absorption train: 3 washbottles with anisole (bottles of type Drechsel, -3°C), drop separator and savety tar adsorber filled with lignite coke (already used in previous test runs).
- Before test run, heated sampling nozzle was cleaned by N_2 flushing.
  In the impingers, no mist can be seen (hence lower particle filter temp. collects aerosols)

Averaged sample flow rate:	0.758 Nm³/h	#DIV/0!
Total amount of gas sampled:	0.808 Nm³	
Total sampling duration:	1.07 hours	
Mean gas meter temperature:	12.5 °C	

Calculation of the gas dew point

Total amount of condensed moisture:	77.5 g	
water vapour equivalent	0.096 Nm³	8.8 Vol.-% moisture in gas
water content in producer gas	96 g/Nm³ dry	
water vapour pressure	0.107 bar	
Calculated dew point of the gas:	47.4 °C	

P+T prot., V#clean (DK)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Countercurrent gasifier (updraft) Date: 3.11.1998
Sampling site (hot/cold end): Clean gas after scrubber
Test No.: 4

Particle filter pretreatment

Filter tube no.: 5
Preconditioning temperature: 270 °C
Preconditioning duration: 3 hours
Initial tube weight after preconditioning: 4.5831 g

Particle filter tar extraction procedure

Cold extraction with anisole

Start: ____ hh:mm
End: ____ hh:mm
Duration: ____ hours

	tara weight	brutto weight	netto weight	
Anisole cold extract bottle *:	371.4	484.2	112.8	[g]
Acetone rinsing bottle **:	364.7	429.6	64.9	[g]

Remarks:
* Anisole is used only for the cleaning of the particle filter housing.
** Aceton rinsing solution originates from particle filter housing cleaning process.
Thimble is packed in alu foil for further treatment.

Soxhlet extraction with anisole

Start: 9:30:00 hh:mm
End: 9:00:00 hh:mm
Duration: 23.5 hours

	tara weight	brutto weight	netto weight	
Anisole extract bottle	371.4	844.6	473.2	[g]
Acetone-rinsing bottle ***	364.7	448.0	83.3	[g]

*** Sum of acetone liquids from filter house and Soxhlett apparatus cleaning

Remarks:
The Soxhlet extraction is made with fresh anisole and at ambient pressure. Acetone is used to clean the Soxhlett apparatus. The acetone rinsing solution is not evaporated.
The anisole in the Soxhlet thimble housing is almost colourless after 23.5 hours of extraction.
The filter tube becomes a reddish colour at the outer surface.
Thimble is packed in alu foil for further treatment.

Soxhlet extraction with methanol

Start: 17:00:00 hh:mm
End: 23:00:00 hh:mm
Duration: 6.0 hours

	tara weight	brutto weight	netto weight	
Methanol extract bottle	368.4	668.9	300.5	[g]

Remarks:
The Soxhlet extraction is made at ambient pressure.
The thimble immediately turns completely white when fresh methanol is filled in the extractor.
The methanol in the Soxhlet thimble housing is colourless after 6 hours of extraction.

Particle filter posttreatment

Postconditioning temperature: 175 °C
Postconditioning duration: 1.5 hours
Final tube weight after postconditioning: 4.5903 g

Remarks: ____

5.2 Protocols from site 2 (cocurrent gasifier)

5.2.1 Sampling protocol of test run #5 (V5 raw)

P+T prot., V5 raw (DK)

P+T - Measurement for Biomass Gasifiers

Examinators: Ph.Hasler

Gasifier Type/Site: Cocurrent gasifier (downdraft)

Date: 5.11.1998

Test No.: 5

Gasifier parameter

Actual power output: 530 KW (max. * KW)
(based on fuel fuel input)

Raw gas flow rate (dry): 316 [Nm³/h] (if calc., spec. gas rate: 2.63 Nm³/kg wet

Gasifier start *: *

stationary since *: *

Fuel type *: Hard wood blocks (approx. 5 x 10 x 10 cm, no dust)

Fuel moisture *: 15 [%, dry basis]

Actual fuel feeding rate *: 120 [kg wet/h]

Sampling parameters

Sampling site (hot/cold end): Raw gas

Ambient temperature: 5 [°C]

Ambient pressure: 1003 [mbar]

Duct diameter: 15 cm

Diameter of sampling nozzle: 8 mm

Sampling tube temperature before filter: 250 [°C]

Particle filter temperature: 150 [°C]

	tare weight	netto weight	final weight 1	final weight 2	
Water condensation bottle:	454.2	114	685.7	———	[g]
Condensate storing bottle:	996.1	———	———	1374.6	[g]
Anisole storing bottle:	374.1	———	———	720	[g]
Acetone storing bottle:	370.1	———	———	449.9	[g]
Filter tube (no 9):	4.6978	———	5.2326	———	[g]

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*

Summary protocol

Generated condensate mass: 117.5 [g]

Total mass of water: 378.5 [g]

Total mass of anisole: 345.9 [g]

Total mass of acetone: 79.8 [g] (acetone: cleaning solvent)

Total particle mass in filter tube: 535 [mg]

Normalized sampled gas flow rate: 0.623 [Nm³/h] (set point: 0.989 Nm3/h; 10% over isokinetic)

Remarks: -Values given by the gasifier operators are marked with *.

P+T prot., V5 mw (DK)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: #BEZUG!

Sampling site (hot/cold end): Raw gas

Date: 5.11.1998

Test No.: 5

Rated sampling gas flow rate (dry): 0.989 Nm³/h (calculated set point; 10% over isokinetic)

P+T sampling start: 10:14 P+T sampling end: 11:32

Gas meter protocol

Time [hh:mm]	Gas temperature in duct [°C]	Reading gas meter [m³]	Temperature in gas meter [°C]	Δp pump [mbar]	Sampling Flow rate [Nm³/h]
10:14:45	595	76.053	16	70	--------
10:23:15		76.164	15.6	70	0.734
10:35:00		76.302	15.9	70	0.660
10:48:45		76.485	16.2	150	0.746
11:10:30		76.728	16.5	120	0.626
11:19:30		76.795	16.8	300	0.417
11:32:45		76.920	17	500	0.528

Remarks:

- Preseparation of particles in filter (glass fibre thimbles) heated to 150°C
- Condensation train: cooler (app. 10°C), condensator (0°C), no drop separator.
- Tar absorption train: 3 washbottles with anisole (bottles of type Drechsel, -3°C), drop separator and savety tar adsorber filled with lignite coke (already used in previous test runs).
- Before test run, heated sampling nozzle was cleaned by N_2 flushing.
- Thimble filter is brocken near the fixation and hence particles are found in the sampling train.
- White mist in the impinger bottles.
- In tar adsorber, a sticky product (tar and carbon) blocked the tube (pressure drop is most probably caused by this cake).

Averaged sample flow rate:	0.623 Nm³/h	37 % under isokinetic
Total amount of gas sampled:	0.810 Nm³	
Total sampling duration:	1.30 hours	
Mean gas meter temperature:	16.3 °C	

Calculation of the gas dew point

Total amount of condensed moisture:	117.5 g	
water vapour equivalent	0.146 Nm³	12.8 Vol.-%
water content in producer gas	145 g/Nm³ dry	
water vapour pressure	0.153 bar	
Calculated dew point of the gas:	54.7 °C	

P+T prot., V5 raw (DK)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Cocurrent gasifier (downdraft) Date: 5.11.1998

Sampling site (hot/cold end): Raw gas

Test No.: 5

Particle filter pretreatment

Filter tube no.: 9

Preconditioning temperature: 270 °C

Preconditioning duration: 3 hours

Initial tube weight after preconditioning: 4.6978 g

Particle filter tar extraction procedure

Cold extraction with anisole

Start: ______ hh:mm

End: ______ hh:mm

Duration: ______ hours

	tara weight	brutto weight	netto weight	
Anisole cold extract bottle *:	363.8	481.2	117.4	[g]
Acetone rinsing bottle **:	370.2	416.2	46.0	[g]

Remarks: * Anisole is used only for the cleaning of the particle filter housing.

** Aceton rinsing solution originates from particle filter housing cleaning process.

Thimble is packed in alu foil for further treatment. Outside colour of thimble is white.

Soxhlet extraction with anisole

Start: 9:30:00 hh:mm

End: 16:50:00 hh:mm

Duration: 7.3 hours

	tara weight	brutto weight	netto weight	
Anisole extract bottle	363.8	856.3	492.5	[g]
Acetone rinsing bottle ***	370.2	450.7	80.5	[g]

*** Sum of acetone liquids from filter house and Soxhlet apparatus cleaning

Remarks: The Soxhlet extraction is made with fresh anisole and at ambient pressure. Aceton is used also for the final cleaning of the Soxhlet apparatus. The acetone rinsing solution is not evaporated. The anisole in the Soxhlet thimble housing is colourless after 7 hours of extraction. However, the filter tube remains black at the outer surface.

Particle filter posttreatment

Postconditioning temperature: 175 °C

Postconditioning duration: 1.5 hours

Final tube weight after postconditioning: 5.2326 g

Remarks: ______

protocol 3-3

Verenum, Zürich (16.6.1999)

5.2.2 Sampling protocol of test run #6 (V6 raw)

P+T prot., V6 raw (DK)

P+T - Measurement for Biomass Gasifiers

Examinators: Ph.Hasler

Gasifier Type/Site: Cocurrent gasifier (downdraft)

Date: 5.11.1998

Test No.: 6

Gasifier parameter

Actual power output: (based on fuel fuel input) 530 kW (max. [illegible] kW)

Raw gas flow rate (dry): 316 [Nm³/h] (if calc., spec. gas rate: 2.63 Nm³/kg wet

Gasifier start *: 3.11.1998,app. 16:00h stationary since *:

Fuel type *: Hard wood blocks (approx. 5 x 10 x 10 cm, no dust)

Fuel moisture *: 15 [%, dry basis]

Actual fuel feeding rate *: 120 [kg wet/h]

Sampling parameters

Sampling site (hot/cold end): Raw gas

Ambient temperature: 5 [°C]

Ambient pressure: 1003 [mbar]

Duct diameter: 15 cm

Diameter of sampling nozzle: 8 mm

Sampling tube temperature before filter: 200 [°C]

Particle filter temperature: 150 [°C]

	tara weight	netto weight	final weight 1	final weight 2	
Water condensation bottle:	465.8	123	664.3	——	[g]
Condensate storing bottle:	993.3	——	——	1264.5	[g]
Anisole storing bottle:	370.2	——	——	598	[g]
Acetone storing bottle:	368.2	——	——	471.5	[g]
Filter tube (no 8):	4.7650	——	4.8268	——	[g]

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*

Summary protocol

Generated condensate mass: 75.5 [g] In water phase some anisol is found

Total mass of water: 271.2 [g]

Total mass of anisole: 227.8 [g]

Total mass of acetone: 103.3 [g] (acetone: cleaning solvent)

Total particle mass in filter tube: 62 [mg]

Normalized sampled gas flow rate: 0.403 [Nm³/h] (set point: 0.989 Nm3/h; 10% over isokinetic)

Remarks: *Values given by the gasifier operators are marked with *.

P+T prot., V6 raw (CK)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Cocurrent gasifier (downdraft)

Sampling site (hot/cold end): Raw gas

Date: 5.11.1998

Test No.: 6

Rated sampling gas flow rate (dry): 0.989 Nm^3/h (calculated set point; 10% over isokinetic)

P+T sampling start: 14:32 P+T sampling end: 15:06

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]
14:32:00	595	76.945	16.9	70	-------
14:45:30		77.036	16.9	150	0.385
15:06:40		77.195	16.9	50	0.415

Remarks:

- Pre-separation of particles in filter (glass fibre thimbles) heated to 150°C
- Condensation train: cooler (app. 10°C), condensator (0°C), no drop separator.
- Tar absorption train: 3 washbottles with anisole (bottles of type Drechsel, -3°C), drop separator and safety tar adsorber filled with lignite coke (already used in previous test runs).
- Before test run, heated sampling nozzle was cleaned by N_2 flushing.
- At 14:54, the tar adsorber blocked up with oily tars (!) and sampling was stopped for 15 sec
- No mist can be seen in the impingers
- Particles are found in the entire sampling train (thimble has partially broken).

Averaged sample flow rate:	0.403 Nm^3/h	55 % under isokinetic
Total amount of gas sampled:	0.233 Nm^3	
Total sampling duration:	0.58 hours	
Mean gas meter temperature:	16.9 °C	

Calculation of the gas dew point

Total amount of condensed moisture:	75.5 g		
water vapour equivalent	0.094 Nm^3	n.d.	Vol.-%
water content in producer gas	324 g/Nm^3 dry		
water vapour pressure	0.287 bar		
Calculated dew point of the gas:	68.4 °C		

P+T prot., V6 raw (DK)

P+T - Measurement for Biomass Gasifiers

Gasifier Type/Site: Cocurrent gasifier (downdraft) Date: 5.11.1998

Sampling site (hot/cold end): Raw gas

Test No.: 6

Particle filter pretreatment

Filter tube no.: 8

Preconditioning temperature: 270 °C

Preconditioning duration: 3 hours

Initial tube weight after preconditioning: 4.7650 g

Particle filter tar extraction procedure

Cold extraction with anisole

Start: ____ hh:mm

End: ____ hh:mm

Duration: ____ hours

	tara weight	brutto weight	netto weight	
Anisole cold extract bottle *:	367.5	514.9	147.4	[g]
Acetone rinsing bottle **:	366.2	416.6	50.4	[g]

Remarks:

* Anisole is used only for the cleaning of the particle filter housing.

** Aceton rinsing solution originates from particle filter housing cleaning process.

Thimble is packed in alu foil for further treatment.

Soxhlet extraction with anisole

Start: 17:30:00 hh:mm

End: 8:00:00 hh:mm

Duration: 14.5 hours

	tara weight	brutto weight	netto weight	
Anisole extract bottle	367.5	914.3	546.8	[g]
Acetone rinsing bottle ***	366.2	431.7	65.5	[g]

*** Acetone liquids from filter housing and Soxhlet apparatus cleaning.

Remarks: The Soxhlet extraction is made with fresh anisole and at ambient pressure. Acetone is used for cleaning of the Soxhlet apparatus. The acetone rinsing solution is not evaporated. The anisole in the Soxhlet thimble housing is almost colourless after 14 hours of extraction. The filter tube remains dark at the outer surface.

Particle filter posttreatment

Postconditioning temperature: 175 °C

Postconditioning duration: 1.5 hours

Final tube weight after postconditioning: 4.8268 g

Remarks:

5.3 PAH concentrations in the producer gases

Test run		1	2	3	4	5	6
Date		2.11.1998	2.11.1998	3.11.1998	3.11.1998	5.11.1998	5.11.1998
Gasifier type		Countercurrent	Countercurrent	Countercurrent	Countercurrent	Cocurrent	Cocurrent
Sampling site		Raw gas	Raw gas	Clean gas after scrubber	Clean gas after scrubber	Raw gas	Raw gas
Start of P&T sampling	hh:mm	9:33	11:48	9:15	11:14	10:14	14:32
End of P&T sampling	hh:mm	10:03	12:14	10:17	12:18	11:32	15:06
Naphthalene	mg/Nm^3	< 0.40	< 0.39	9.1	7.3	834	582
Acenaphthylene	mg/Nm^3	22.5	< 0.41	0.56	0.33	223	95
Acenaphthene	mg/Nm^3	< 0.44	< 0.42	0.25	0.41	10.6	79
Fluorene	mg/Nm^3	22.5	10.4	0.54	0.47	40	21
Phenanthrene	mg/Nm^3	24.5	19.8	0.93	0.61	108	69
Anthracene	mg/Nm^3	4.9	4.6	0.28	0.17	32	17.5
Fluoranthene	mg/Nm^3	4.1	5.0	0.68	0.33	46	26.4
Pyrene	mg/Nm^3	6.2	3.3	0.37	0.35	47	23.3
Chrysene	mg/Nm^3	3.8	1.6	< 0.17	0.11	6.9	5.3
Benzo(a)anthracene	mg/Nm^3	1.3	1.0	0.21	0.16	8.7	6.3
Benzo(b)fluoranthene	mg/Nm^3	2.0	< 0.41	0.21	0.19	4.7	4.4
Benzo(k)fluoranthene	mg/Nm^3	2.0	< 0.42	< 0.17	0.23	6.5	5.8
Benzo(a)pyrene	mg/Nm^3	3.5	2.4	0.23	0.26	6.2	6.9
Indeno(1,2,3-cd)pyrene	mg/Nm^3	0.46	< 0.40	< 0.17	0.13	3.9	5.8
Dibenzo(a,h)anthracene	mg/Nm^3	< 0.39	< 0.38	< 0.16	0.09	0.9	2.9
Benzo(g,h,i)perylene	mg/Nm^3	1.08	< 0.40	0.18	0.12	4.4	5.0
Sum of PAH analyzed	mg/Nm^3	100	51	14	11	1383	955

Remarks: Nm^3 at 1013 mbar, 0°C, dry

6 Literature

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Hasler, Ph.; Nussbaumer, Th.: Guideline for sampling and analysis of tars and particulates from biomass gasifiers, Bundesamt für Energie (BfE), 3003 Bern, updated version, August 1998a

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Milne, T.A.; Abatzoglou, N.; Evans, R.J.: Biomass Gasifier „Tars": Their Nature, Formation, and Conversion, National Renewable Energy Laboratory (NREL), Golden, Colorado, report NREL/TP-570-25357, November 1998

Zurich, January 26, 1999

Dr. Ph. Hasler, Verenum Research

APPENDIX 4

Report on Tar and Particulate Measurements in Denmark on 1-6 November 1998

VTT Energy
Espoo, Finland

by Pekka Simell
December 1998

Research Report
ENE/33/98

REPORT ON TAR AND PARTICULATE MEASUREMENTS IN DENMARK on 1 - 6 November1998

Confidential

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Espoo, 8 December 1998

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1 GENERAL

VTT Energy carried out tar and particulate content measurements from gasifier product gas at plant A on 2 - 3 November 1998 and at plant B on 5 - 6 November 1998. The plant A produced district heat and it had an 5 MW_{th} updraft gasifier using forest residues as feedstock. The plant B had a 1 MW_{th} downdraft gasifier and had a gas engine (160 kW_e) conneceted to the gasifier. The product gas of the gasifier was cleaned in a water scrubber and a sawdust filter before feeding it to the engine. The feedstock used in plant B consisted of wood blocks obtained as residue from nearby furniture factories.

The sampling was carried out simultaneously with three other measuring groups, which were from the Dansk Teknologisk Insitute (DTI), Biomass Technology Group (BTG, the Netherlands) and Verenum (Switzerland). This report presents the results of VTT Energy.

The samples were taken at the plant A from a bypass line equipped with a water scrubber. On 2 November, six samples were taken before the scrubber. On the following day six samples were taken from the clean gas after the scrubber. At the plant B six samples were taken from the product gas line before the gas scrubbers on 5 November. On the following day five samples were taken after the water scrubber system, but before the saw-dust filters.

2 SAMPLING AND ANALYTICAL METHODS

Gas flow rates and temperatures in the sampled gas pipes were provided by DTI. The sampling gas flow rates and probe tip diameters were chosen according to this information so that the sampling took place isokinetically.

The samples from the plant A were taken by applying two methods. On 2 November the samples were taken with and without a particulate filter from the bypass-line before the gas scrubbers. This was done in order to find out the particulate content of the gas and, on the other hand, the amount of condensing tars at the gas filtration temperature. The gas filter was held at about 250 °C (set-up temperature of the filter box was 350 °C). After the filter, the tars were absorbed into dichlormethane. The sample solvent obtained without the gas filter was filtered in the laboratory before the tar analysis and the gas solids content was determined according to this procedure. On 3 November the samples were taken only without the particulate filter, because the particulate content was assumed to be negligible after the water scrubber. To obtain the total amount of tars, the samples were analysed gravimetrically by evaporating the residues at 105 °C for one hour. The sampling and analytical methods employed are described in detail in reference [1].

The samples from the plant B were taken with the particulate filter. The gas filter was held at about 250 °C (set-up temperature of the filter box was 350 °C). The tar samples were analysed for the light tar compounds by HP 5890 A series II gas chromatograph equipped with an FID detector and HP Ultra 2 column (0.32 i.d. x 50 m). The particulate filters were dried at 105 °C for 1 h and weighed. The sampling and analysing methods are described in detail in [1].

The particulate filters were weighed after drying at 105 °C for 1 h. After this the filters were extracted with dichlormethane in a Soxhlet apparatus. The obtained extracts were then analysed gravimetrically in similar manner as the tar samples by evaporating them at 105 °C for 1 h.

3 RESULTS

The measured tar and particulate contents of the plant A are presented in Appendix 1. The evaporation residue at 105 °C corresponds to the content of compounds with a boiling point higher than ca. 350 °C [2]. Hence, this residue can be considered to represent the content of the heavy tars.

The 'solids' in the three particulate filters obtained from the plant A seemed to be condensed tars as they were extracted completely to dichloromethane. They were also recovered completely during the evaporation of the solvent and thus the total tar from samples 1, 3 and 5 was calculated as the sum of the tars on the filter and the tars obtained from the solvent. Hence, it seemed that solids were present in the gas in concentrations that were below our normal detection limit with this kind of sampling procedure. However, the solids measurement for the sample solvent taken without the filter (samples 2, 4 and 6) gave results in the range of 0.2 - 0.4 g/m^{3}n.

The measured light tar contents of the plant B are presented in Appendix 2 and the particulate contents in Appendix 3. The obtained particulate filters were extracted with dichloromethane similarly to the filters from the plant A in order to determine the amount of condensed tars. The weight loss of the filters was about 3%.

4 REFERENCES

1. Ståhlberg, P., Lappi, M., Kurkela, E., Simell, P., Oesch, P & Nieminen, M. Sampling of contaminants from product gases of biomass gasifiers. Espoo: Technical Research Centre of Finland, 1998. 49 p. + app. 46 p. (VTT Research Notes 1903).

2. Simell, P. Kotimaisten polttoaineiden kaasutuksessa syntyvät tervamaiset epäpuhtaudet. Espoo: Technical Research Centre of Finland, 1988. 53 p. + app. 22 p. (VTT Research Reports 531).

APPENDIX 1

TAR CONTENT OF THE GAS AT PLANT A

Tar analysed gravimetrically as an evaporation residue at 105 °C (1 h).

A) Sampling before the scrubber. Sampling date 2.11.1998

Table 1. Particulate content of gas as measured with particulate filter and by filtering tar absorbtion solvent. The amount of extractibles (tars) on the gas filter.

Sample	Time	Particulates measured from the gas filter g/m^3_n	Extractibles wt%	Particulates measured from the solvent g/m^3_n	Particulates g/m^3_n
1	9:35 - 10:05	17.7	100	-	0
2	10:20 - 10:43	-	-	0.3	0.3
3	11:45 - 12:13	7.8	100	-	0
4	12:33 - 12:57	-	-	0.2	0.2
5	14:15 - 14:40	16.7	99	-	0.2
6	14:55 - 15:18	-	-	0.4	0.4

Table 2. Tar content of gas.

Sample	Time	Extractibles on particulate filter g/m^3_n	Tar g/m^3_n	Sum of tar and extractibles g/m^3_n
1	9:35 - 10:05	17.7	26.5	44.2
2	10:20 - 10:43	-	45.6	45.6
3	11:45 - 12:13	7.8	30.8	38.6
4	12:33 - 12:57	-	43.8	43.8
5	14:15 - 14:40	16.5	29.2	45.7
6	14:55 - 15:18	-	39.7	39.7

B) Sampling after the scrubber. Sampling date 3.11.1998

Table 3. Tar content of gas.

Sample	Time	Tar g/m^3_n
1	9:20 - 9:45	4.3
2	9:50 - 10:12	2.4
3	11:15 - 11:35	2.2
4	11:40 - 12:02	2.1
5	13:50 - 14:09	2.1
6	14:15 - 14:35	2.4

APPENDIX 2

LIGHT TAR CONTENT OF THE GAS AT THE PLANT B

Tars analysed by GC.

Sampling before the gas scrubbers.

Sample Point Date Time	1 Before scrub. 5.11.1998 10.15-10.27	2 Before scrub. 5.11.1998 10.55-11.05	3 Before scrub. 5.11.1998 11.42-11.53	4 Before scrub. 5.11.1998 12.13-12.24	5 Before scrub. 5.11.1998 14.30-14.42	6 Before scrub. 5.11.1998 14.57-15.08
CONCENTRATIONS mg/m^3_n						
Benzene	3325	1436	2278	2930	2193	2001
Tars:						
Pyridine	0	0	0	0	0	0
Toluene	574	101	272	383	265	214
m-Xylene	57	5	20	26	19	12
Ethynylbenzene	39	6	17	28	20	18
Styrene	143	12	45	72	52	36
o-Xylene	13	0	4	5	4	0
Phenol	108	5	16	23	16	0
4-Methylstyrene	34	0	7	11	10	0
Indene	278	50	135	191	116	99
Naphthalene	890	357	583	765	565	498
Quinazoline	0	0	0	0	0	0
Isoquinazoline	8	0	0	0	0	0
2-Methylnaphthalene	69	10	29	42	29	22
1-Methylnaphthalene	43	5	16	24	17	12
Biphenyl	41	8	20	30	22	17
2-Ethylnaphthalene	8	0	0	4	0	0
Acenahthylene	307	103	187	253	182	163
Acenaphthene	18	7	10	11	8	7
Dibenzofurane	27	10	15	21	17	15
Fluorene	5	15	44	64	36	32
Phenantrene	198	64	115	173	126	104
Anthracene	52	14	27	42	28	23
4H-Cyclopenta(def)-phenanthrene	31	12	19	26	18	16
Fluoranthene	69	34	51	70	61	50
Benz(e)acenaphthylene	25	13	19	28	21	19
Pyrene	76	30	52	76	58	49
TARS (mp.> 79 g/mol)	3115	861	1703	2370	1688	1406
TARS + BENZENE	6439	2297	3981	5300	3880	3408

APPENDIX 3

LIGHT TAR CONTENT OF THE GAS AT THE PLANT B

Tars analysed by GC.

Sampling after the water scrubber.

Sample	1	2	3	4	5
Point	After scrub.	After scrub.	After scrub.	After scrub.	After scrub.
Date	6.11.1998	6.11.1998	6.11.1998	6.11.1998	6.11.1998
Time	9.03-9.14	9.32-9.44	10.19-10.33	11.44-11.57	12.09-12.20
CONCENTRATIONS [mg/m^3_n]:					
Benzene	2598	3279	2446	2091	2687
Tars:					
Pyridine	0	0	0	0	0
Toluene	372	466	351	194	364
M-Xylene	33	49	28	11	30
Ethynylbenzene	47	52	30	20	36
Styrene	93	110	75	34	90
O-Xylene	3	11	6	0	7
Phenol	12	25	15	10	10
4-Methylstyrene	21	26	16	5	20
Indene	190	305	172	108	183
Naphthalene	727	918	551	577	684
Quinazoline	0	0	0	0	0
Isoquinazoline	0	0	0	0	0
2-Methylnaphthalene	45	50	41	26	39
1-Methylnaphthalene	29	36	24	14	23
Biphenyl	28	32	24	18	24
2-Ethylnaphthalene	7	8	5	0	5
Acenahthylene	245	291	212	190	211
Acenaphthene	9	12	3	6	8
Dibenzofurane	19	21	16	15	17
Fluorene	38	51	35	29	37
Phenantrene	72	76	54	53	64
Anthracene	16	19	13	12	15
4H-Cyclopenta(def)phenanthrene	11	13	3	7	9
Fluoranthene	34	31	25	24	27
Benz(e)acenaphthylene	17	9	11	11	12
Pyrene	43	18	22	26	27
TARS (mp.> 79 g/mol)	2115	2638	1844	1391	1941
TARS + BENZENE	4714	5917	4290	3482	4628

APPENDIX 4

PARTICULATE CONTENT OF THE GAS AT THE PLANT B

Sample	Date	Time	Particulates mg/m³	Extractibles wt%
1	5.11.1998	10 15 - 10:27	550	2.8
2		10 55 - 11:05	90	24.0
3		11 42 - 11:53	560	3.8
4		12 13 - 12:24	510	2.5
5		14 30 - 14:42	570	3.2
6		14 57 - 15:08	630	1.4
1	6.11.1998	9 03 - 9:14	470	2.8
2		9 32 - 9:44	1 700	1.3
3		10 19 - 10:33	460	1.6
4		11 44 - 11:57	210	8.0
5		12 09 - 12:20	250	2.5