

MANUAL TAR MEASURING METHOD

Phase 2

ROUND ROBIN TEST 02 and 03

*Energy Research Programme EFP 2002
Environmentally friendly production of power and heat*

*Danish Energy Agency
File No.: 1373/02-0009*

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September 2004

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1. Introduction

In connection with thermal conversion (gasification) of solid biomass, measurements of pollution in the producer gas are required. Gas pollution is very complex as it may consist of both solid substances (particulate, soot), drops (aerosol, fog) and a large number of organic compounds (alcohols, organic acids, "tar", PAHs). For a number of years, Danish Technological Institute, Centre for Combustion and Engine Technology, has worked on developing a suitable measuring method which can quantify the total spectrum of pollution. In connection with the Danish Energy Research Programme EFP 2000 the report "Verification and Validation of Manual Tar Measurement Method" \1\ was published.

At European level different initiatives were taken in order to solve the same problem. The previously published reports "Provisional Protocol for the Sampling and Analysis of Tar and Particulates in the Gas from Large-Scale Biomass Gasifiers" \2\ and "Protocol for the Sampling and Analysis of Particulate and Organic Contaminants in the Gas from Small-Scale Biomass Gasifiers" \3\ have - together with the results of parallel measurements \4\, which were carried out in Denmark by four European laboratories - resulted in an EU project (ERK6-CT-1999-20002) with the purpose of creating one protocol covering the whole field. In the following this project is called *Tar Guideline* \5\.

In 2002, the EU Commission decided to subsidise a project (ENK5-CT-2002-80648) "Tar Measurement Standard" with the primary objective to standardise the Tar Guideline, in order to reduce the technical and non-technical risks of implementation of biomass based CHP systems in the future. Work package WP2 is critical in this project and it shall provide data on

accuracy and reproducibility

of the draft Standard according to requirements for CEN. These data will be provided by the group of technical experts (ECN, BTG, CIRAD, DTI, NREL, TU Graz, UMSICHT, Unizar, and VTT) who have collected and will include these data in their current national R&D projects plus in additional national projects aimed to provide these data. The necessity of national R&D effort is based on the fact, that the EU project only supports the standardisation activity. DTI and VTT are responsible for collection and interpretation of data. The data will be used in WP1 by the CEN working group in order to standardise the Tar Guideline.

One of the objectives of the present EFP 2002 project is to contribute to the procurement of data. In this context DTI organised a Round Robin Test consisting of three rounds (WP2, Task 2.3, Round Robin on gas chromatographic and gravimetric methods). In the first round synthetic samples with about 10 tar compounds representing updraft and downdraft or fluidised bed gasifiers were analysed gas chromatographically by the participating laboratories. On the basis of the results of round 01, real samples from updraft and downdraft or fluidised bed gasifiers were analysed gas chromatographically and gravimetrically (residue of evaporation) in round 02. This round showed that it is necessary to tighten up the description of the gravimetric analysis method due to varying results. It was decided to carry out a third round only for gravimetric analysis. The present report concerns the results of the second and third round of the Round Robin Test.

2. Conclusion and Recommendations

The results of these Round Robin tests were used to determine the performance of the GC and gravimetric methods of the draft standard *Sampling and Analysis of Tar and Particles in Biomass Producer Gases* \6\.

Round 1 (published in a separate report \7\) as a preliminary test of GC analysis method proved to be important for the successful round 02. The data collected in round 02 were used to evaluate the performance of the GC method. The precision data of gravimetric method were originally planned to be collected in the round 02. However, due to differences in the evaporation procedure used by different laboratories, the results were discarded and an additional round was carried out. Thus, the precision data describing the performance of the gravimetric method were collected from round 03.

Gas chromatographable tars

The evaluation of the data was divided into two parts: analysis of individual compounds and analysis of total gas chromatographable tar (calculated as naphthalene). In addition, based on differences in the results, individual compounds were divided to represent two types of tar:

- **High temperature tar** is formed in high temperature processes like fluidised bed gasifier or downdraft gasifier and consists mostly of non-polar aromatic compounds.
- **Low temperature tar** is formed in lower temperature, for example in updraft gasifier, and contains a high amount of polar compounds. Also the number of individual tar compounds in low temperature tar is very high (that makes the matrix of low temperature tar very complex).

The overall observation was that the precision values depended on the concentration. In addition, the percentage values of the precision data increased with decreasing concentration level. This was very obvious for high temperature tar and for total gas chromatographable tar. It was also indicated from the results of updraft tars, however not so clearly. The reason for the different and somewhat confusing values of low temperature tars may be due to difficult compound matrix of tar.

The main results of the Round Robin test of the GC method are presented in the following table, containing the suggested repeatability and reproducibility values for the performance of the method for the draft standard *Sampling and Analysis of Tar and Particles in Biomass Producer Gases*. Precision values are presented as mean values for different concentration ranges with one exception: it is not recommended to include the precision values of low temperature tar below 1 mg/dm³ to the performance table of the draft standard because its values differed significantly from the general trend.

The precision values of lowest concentration level of both high temperature tar and total chromatographable tar are very high (i.e. are very poor), especially reproducibility values. Thus, it should be recommended in the draft standard that the concentration of total

chromatographable tars (if determined in the same way as in this test) should be over 100 mg/dm³ in solution.

In general, round 01 and round 02 indicate that in order to attain reliable results, it is required that a professional GC analyst carries out the analysis. In addition, a GC program to suit the sample and the correct identification are considered very important.

Repeatability and reproducibility for the gas chromatographic analysis of tar.

Tar type	Concentration range mg/dm ³	Repeatability standard deviation s _r %	Repeatability limit ² r %	Reproducibility standard deviation s _R %	Reproducibility limit ³ R %
HIGH TEMPERATURE TAR					
Individual compounds	20 – 140	2.5	7.1	7.3	20
Individual compounds	1– 10	4.8	13	13	35
Individual compounds	0.2 – 0.3	17	47	28	78
LOW TEMPERATURE TAR					
Individual compounds	30 – 560	4.8	13	19	54
Individual compounds	2 – 8	8.2	23	28	80
TOTAL GC-DETECTABLE TAR¹					
	300 – 6000	2.9	8.2	13	37
	5 – 50	10	28	74	210

¹ Calculated as naphthalene.

² The difference between two test results found on the same sample by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit on average not more than once in 20 cases in the normal and correct operation of the method.

³ Test results on the same sample reported by two laboratories will differ by more than the reproducibility limit on average no more than once in 20 cases in the normal and correct operation of the method.

Gravimetric tars

A gravimetric method was developed during the EU project Tar Guideline (ERK6-CT-1999-20002). The new gravimetric method was required because of the new solvent chosen as absorption solvent in the sampling method (iso-propanol).

The results of the Round Robin test showed that there is a systematic error in the results between laboratories. This indicates that the laboratories have some differences in their procedure of gravimetric analysis which affect the results. The problem is caused by the light tar fraction, which evaporates partly during the gravimetric analysis. Also the moisture content of the sample may have an effect. Until now, the reason for the difference has not been detected although several possibilities have been excluded. One reason may be the efficiency with which the evaporation is started.

The results of the Round Robin test of gravimetric method are presented in the table below, containing the suggested repeatability and reproducibility values for the performance of the method for the draft standard *Sampling and Analysis of Tar and Particles in Biomass Producer Gases*. The average reproducibility value was high, 71%. The results of the gravimetric method indicated very clearly the same trend which was observed from the results

of the GC method: the percentages of precision values increased with decreasing concentration.

The reproducibility clearly increased when the concentration decreased, and this indicates that the reproducibility would be even higher for concentration below tested. Thus, it should be recommended in the draft standard that the lower limit for gravimetric concentration in solvent should be 1000 mg/dm³.

Based on the above, the gravimetric method is suitable mainly for estimation of very high tar concentration in producer gas (typically present in updraft gasifier). The concentration of gravimetric tar in producer gas of for example fluidised bed gasifier is often so low that the gravimetric analysis would require an extremely high sampling volume to attain the concentration level 1000 mg/dm³ in solution.

It should be emphasized to the users of the standard that the value of gravimetric tar is suitable for estimation of producer gas containing high tar concentration whereas the GC method provides informative and comparable data in case producer gas contains lower tar concentration (like typically fluidised bed or downdraft gasifiers).

The gravimetric method requires development before the gravimetric tar concentration can be regarded as more than an estimate for lower tar concentration. However, the systematic error between the laboratories indicates that focusing on the method description and discovering the reason for the repetitive difference between the laboratories may improve the performance of the method. In addition, it would be necessary that lower concentration levels would be covered by Round Robin test.

Repeatability and reproducibility for the gravimetric analysis of tar.

Tar type	Concentration range g/dm ³	Repeatability standard deviation	Repeatability limit ¹	Reproducibility standard deviation	Reproducibility limit ²
		s _r %	r %	s _R %	R %
GRAVIMETRIC TAR	5 – 60	6.5	18	26	71

¹ The difference between two test results found on the same sample by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit on average not more than once in 20 cases in the normal and correct operation of the method.

² Test results on the same sample reported by two laboratories will differ by more than the reproducibility limit on average no more than once in 20 cases in the normal and correct operation of the method.

3. Participating Laboratories

In August 2003, in connection with round 02, nine laboratories received five real tar samples including one blind sample. The lab that withdrew in round 01 was excluded and instead a new laboratory participated in the test. Again one lab withdrew and the same two laboratories as in round 01 did not report their results.

Consequently round 02 includes results from six laboratories.

Laboratories involved in this Round Robin Test are listed below in alphabetical order:

BTG Biomass Technology Group B.V. - Enschede, The Netherlands

CIRARD Forêt - Montpellier, France

DTI - Danish Technological Institute - Aarhus, Denmark

ECN Biomass - Energy Research Center of the Netherlands - Petten, The Netherlands

Fraunhofer UMSICHT - Oberhausen, Germany

NREL - National Renewable Energy Laboratory - Golden, CO, USA

University of Zaragoza, Spain

VTT Energy - Espoo, Finland

The participating laboratories were given arbitrarily code numbers so the results would be anonymous.

In round 03 the same laboratories were asked to participate in the test. This time there was one withdrawal, but a new laboratory expressed interest in participating in the gravimetric test. The new participant was:

Technische Universität Graz, Austria

The other participants in round 03 were:

BTG Biomass Technology Group B.V. - Enschede, The Netherlands

DTI - Danish Technological Institute - Aarhus, Denmark

ECN Biomass - Energy Research Center of the Netherlands - Petten, The Netherlands

Fraunhofer UMSICHT - Oberhausen, Germany

NREL - National Renewable Energy Laboratory - Golden, CO, USA

VTT Energy - Espoo, Finland

In March 2004, these laboratories received five samples of updraft tar dissolved in isopropanol. One laboratory did not report its results.

Consequently round 03 includes results from six laboratories.

4. The Tar Samples

4.1. Round 02 - Real Tar

Round 02 is based on real tar samples taken from producer gas in July 2003. The origin and specifications of the tar samples are as follows:

TAR 01:

Gasifier: Countercurrent (updraft)
Plant: Harboøre, 4.5 MW fuel
Fuel: Wood chips
Sample position: Outlet gasifier (raw gas)
Sampling duration: 4.5 hours
Sampling temp.: 70-100 °C
Sample volume: 1.022 m³_n sampled in 5 litre isopropanol

TAR 02:

Gasifier: Countercurrent (updraft)
Plant: Harboøre, 4.5 MW fuel
Fuel: Wood chips
Sample position: Outlet ESP (clean gas)
Sampling duration: 18 hours
Sampling temp.: 40 °C
Sample volume: 6.907 m³_n sampled in 5 litre isopropanol

TAR 03:

Gasifier: Atmospheric airblown CFB
Plant: Fraunhofer UMSICHT ZWS, 0.5 MW fuel
Fuel:
Sample position: Raw gas
Sampling duration:
Sampling temp.: 430 °C
Sample volume: 0.480 m³_n sampled in 5 litre isopropanol

TAR 04:

Gasifier: Atmospheric airblown CFB
Plant: Fraunhofer UMSICHT ZWS, 0.5 MW fuel
Fuel:
Sample position: Hot clean gas
Sampling duration:
Sampling temp.: 350 °C
Sample volume: 0.480 m³_n sampled in 5 litre isopropanol

In updraft raw gas DTI took 10 samples in about 500 ml isopropanol each. In updraft clean gas 12 samples were taken two by two at the same time to minimize the time consumption. The raw gas samples from updraft gas were filtrated in order to remove particulate matter before they were divided into test samples at DTI. The samples from the CFB gasifier were mixed from eight samples in raw and clean gas. The solutions did not contain particulate impurities, they were divided without any pre-treatment.

For GC-analysis 20 ml from each of the four samples were transferred into capet vials for shipment. In addition, one blind sample containing pure isopropanol was prepared. For gravimetric tar analysis 300 ml of each sample was forwarded to the participating laboratories in August 2003. Eight laboratories agreed to participate in the test. Results from six laboratories are received at the present time.

The laboratories have divided these samples into 6 sub samples for repetition of the analysis.

4.2. Round 03 - Gravimetric Tar

From the updraft gasifier in Harboøre two tar samples from the heavy tar and from the aqueous tar storage tank were taken. The two tar fractions were mixed in different ratios and dissolved in isopropanol. Five different solutions were produced as follows:

	Aimed conditions		Weighed tar fractions		Iso-propanol ml	Total ml
	Concentration g/m^3_n	Water content vol %	Heavy tar g	Aqueous tar g		
Solution 1	55	40	220	1286	2494	4000
Solution 2	1.6	10	6.4	321	3672	4000
Solution 3	1.6	40	6.4	1286	2708	4000
Solution 4	6.5	15	26	482	3492	4000
Solution 5	22.5	15	90	482	3428	4000

The solutions were divided into eight equal samples of 500 ml each. These samples, marked GRAVTAR 01, GRAVTAR 02, GRAVTAR 03, GRAVTAR 04 and GRAVTAR 05, were sent to the participating laboratories on 3 March 2004.

5. The Results of the Laboratories

5.1. Round 02

The laboratories individual results are presented in a table like the paragon shown below:

- 1) Round Robin Test on Tar, September 2003
- 2) Round 02 - Synthetic tar

3) Laboratory code No. x

4) Table 1

Sample identification	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine									
Toluene									
Phenol									
Indene									
Guaiacol									
Naphthalene									
Acenaphthylene									
4-methylguaiacol									
Phenanthrene									
Fluoranthene									
Pyrene									

5)

Number of comp.							Mean of CV's %	
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6)

Total chrom. tar*									
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7

Gravimetric tar									
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8) Table 2

Used apparatus, sample preparation and analysis condition

The table shall be read as follows:

- 1) Title of the test and months of completion.
- 2) Number of round and type of sample.
- 3) Code No.of the laboratory
- 4) Table 1: Column 1 contains the sample identity and the compounds in the performance test. Column 2-7 contain the analysis results of the laboratory in question for the sub divided sample. Column 8 contains the mean value of the 6 analysis results. Column 9 contains the mean values deviation from the nominal value. Column 10 contains the standard deviation of the repeated analysis results and column 11 contains the coefficient of variation.

$$CV = \frac{STDev \cdot 100}{Mean Value} \quad [\%]$$

- 5) The number of compounds reported by the laboratory and the average CV.
- 6) The numerically mean deviation from the nominal value (which means, the deviations are added without sign).
- 7) The results of the gravimetric tar analysis (residue of evaporation).
- 8) Table 2: Contains information about used apparatus, sample preparation and analysis condition. The level of information is voluntary.

In the following the results of the participating laboratories are presented.

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO.: Coordinator

TAR 01	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	9.85	7.11	8.09				8.35	1.39	16.6
Toluene	27.3	26.2	22.2				25.2	2.68	10.6
Phenol	112	110	110				111	1.15	1.04
Indene	3.67	2.29	2.68				2.88	0.711	24.7
Guaiacol	258	261	262				260	2.08	0.800
Naphthalene	5.78	4.55	4.62				4.98	0.691	13.9
Acenaphthylene	1.21	1.23	1.25				1.23	0.020	1.63
4-methylguaiacol	239	260	264				254	13.4	5.28
Phenanthrene	1.84	1.81	1.81				1.82	0.017	0.95
Fluoranthene	0.412	0.397	0.408				0.406	0.008	1.91
Pyrene	0.429	0.41	0.427				0.422	0.010	2.47
Number of comp.	11	11	11				Mean of CV's %		7.27
Total chrom. tars *	5040	6050	5760	4840	5130	5830	5442	499	9.16
Gravimetric tar	13744	14116	13974	14272	13842	14040	13998	190	1.36

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Comment on the coordinators results:

Due to insufficient internal communication, the coordinator carried out three repetitions instead of six. This applies for all samples.

Used apparatus, sample preparation and analysis condition
GC/MS-SIM, HP 6890/5973 Internal standard added. Analysed direct by GC/MS full scan.

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 5

TAR 01	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	NA	NA	NA	NA	NA	NA			
Toluene	52.4	52.7	51.6	51.1	52.1	52.9	52.1	0.677	1.30
Phenol	157	158	157	157	159	157	157	0.940	0.597
Indene	14.5	14.0	7.31	13.1	7.66	12.9	11.6	3.22	27.8
Guaiacol	352	352	350	350	356	351	352	2.24	0.636
Naphthalene	20.1	19.9	19.9	20.1	39.5	19.6	23.2	8.02	34.6
Acenaphthylene	8.22	8.09	7.18	7.92	8.70	5.53	7.61	1.13	14.9
4-methylguaiacol	389	388	384	386	391	385	387	2.44	0.631
Phenanthrene	3.03	3.29	3.12	3.29	4.06	3.54	3.39	0.371	11.0
Fluoranthene	1.17	2.09	2.58	3.10	3.73	2.06	2.46	0.893	36.4
Pyrene	3.82	0.00	3.05	5.75	3.26	2.88	3.12	1.86	59.4
Number of comp.	10	10	10	10	10	10	Mean of CV's %		18.7
Total chrom. tars *	4764	4860	4801	4956	5076	4743	4867	128	2.63
Gravimetric tar	19472	20354	20254	19176	21708	20074	20173	882	4.37

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Comment on Lab 5's results:

Lab 5 informed that it wanted to withdraw the results for individual compounds in updraft gas because of problems with compound identification in the peak jungle of updraft tars by GC-FID. The statistic analysis for individual compounds was carried out without Lab 5's results.

Used apparatus, sample preparation and analysis condition
GC/FID, Shimadzu GC 17A, split injector GC-column: J&W DB 5.625, 30 m x 0.25 mm, df = 0.25 µm Oven temp.: 50 °C 4 min, 25 °C/min 150 °C 0 min, 5 °C/min 300 °C 10 min
Analysis of pyridine GC-column: ZB-WAX, 30 m x 0.25 mm, df = 0.25 µm (Phenomenex) Oven temp.: 50 °C 5 min, 10 °C/min 240 °C

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 6

TAR 01	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	8.89	8.31	7.78	9.78	9.13	12.3	9.37	1.59	17.0
Toluene	101	98.6	97.8	100	98.1	107	100	3.42	3.40
Phenol	148	142	142	147	140	147	144	3.26	2.26
Indene	12.9	13.3	13.3	13.5	12.5	13.9	13.2	0.486	3.68
Guaiacol	311	308	306	310	305	314	309	3.27	1.06
Naphthalene	10.8	10.5	10.7	10.2	10.6	10.3	10.5	0.236	2.24
Acenaphthylene	4.50	4.44	4.50	4.45	4.20	4.57	4.44	0.128	2.88
4-methylguaiacol									
Phenanthrene	4.66	4.40	4.50	4.51	5.14	4.39	4.60	0.282	6.13
Fluoranthene	10.1	9.86	8.46	8.93	8.62	9.43	9.23	0.663	7.18
Pyrene	1.55	4.01	6.04	5.95	3.43	4.13	4.19	1.68	40.1
Number of comp.	10	10	10	10	10	10	Mean of CV's %		8.6
Total chrom. tars *	3966	3974	3956	3966	3871	3858	3932	52.7	1.34
Gravimetric tar	9888	9956	9774	9415	9095	8835	9494	458	4.82

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Comments on Lab 6's results:

There was some uncertainty in compound identification because of use of GC-FID. It was decided to carry out the statistic analysis for individual compounds without Lab 6's results.

Used apparatus, sample preparation and analysis condition
GC/FID, HP 5890, HP 3396A integrator Column: HP Ultra 2 (crosslinked 5% Ph Me silicone) 50 m x 0.32 mm, film thickness 0.52 µm Injector: Split/splitless injector 290 °C Carrier gas: Helium, injector pressure about 120 kPa, total flow about 20 ml/min Oven temp.: 50 °C 5 min, heating rate 3 °C /min to 160 °C, then 10 °C/min to 290 °C ISTD = n-dodecane

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 7

TAR 01	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	2.10	2.20	1.60	1.10	2.30	1.80	1.85	0.451	24.4
Toluene	55.5	54.0	52.9	54.5	54.9	54.0	54.3	0.892	1.64
Phenol	115	117	112	111	115	114	114	2.18	1.91
Indene									
Guaiacol	296	297	286	285	293	295	292	5.13	1.76
Naphthalene	6.80	6.80	6.50	6.60	6.60	6.70	6.67	0.121	1.82
Acenaphthylene									
4-methylguaiacol	334	319	314	314	318	315	319	7.73	2.42
Phenanthrene	2.50	2.40	2.40	2.40	2.40	2.50	2.43	0.052	2.12
Fluoranthene	< 2	< 2	< 2	< 2	< 2	< 2			
Pyrene	0.400	0.400	0.400	0.400	0.400	0.400	0.400	0.000	0.000
Number of comp.	8	8	8	8	8	8	Mean of CV's %		4.50
Total chrom. tars *	3875	4008	4140	4286	4277	4287	4146	173	4.17
Gravimetric tar	18940								

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/MS, Agilent 6890 GC with FID for total tar, Agilent 5973 MSD, Agilent 7673 Autosampler Column: J&W Scientific DB-5MS, 30 m x 0.25 mm, 0,25 µm film Split injector 275 °C MSD transfer line 300 °C</p> <p>Sample was spiked with 100 µl of internal standard for MS analysis, no internal standard for FID/total GC tar. Column flow 1 ml/min, split ratio 30:1 Oven temp. 50 °C 5 min, ramp 8 °C/min to 250 °C, hold 0 min, ramp 10 °C/min to 300 °C hold 2,5 min</p> <p>Gravimetric tar: Roto Vap - Büchi Model 142 with temperature and vacuum control</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 16

TAR 01	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	3.99	3.81	3.85	3.63	3.80	3.71	3.80	0.12	3.24
Toluene	45.4	42.7	43.3	40.7	42.3	39.9	42.4	1.94	4.58
Phenol	117	118	119	118	122	117	118	1.83	1.54
Indene	4.53	4.39	4.49	4.15	4.35	4.28	4.37	0.139	3.19
Guaiacol	301	310	309	310	319	304	309	6.29	2.04
Naphthalene	9.28	9.08	9.16	8.79	8.96	8.91	9.03	0.178	1.97
Acenaphthylene									
4-methylguaiacol	372	372	375	374	383	367	374	5.35	1.43
Phenanthrene	1.77	1.79	1.79	1.82	1.80	1.78	1.79	0.017	0.961
Fluoranthene	0.43	0.43	0.43	0.42	0.43	0.41	0.425	0.008	1.97
Pyrene	0.44	0.46	0.46	0.44	0.46	0.45	0.452	0.010	2.18
Number of comp.	10	10	10	10	10	10	Mean of CV's %		2.31
Total chrom. tars *	3385	3601					3493	153	4.37
Gravimetric tar	17020	19160	18850				18343	1156	6.30

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/MS, HP 5890/5971 Column: Ultra 2; 25 m, 0,2 mm ID, 0,33 µm film (Agilent)</p> <p>ISTD: Phenol-d5, phenanthrene-d10, benzo[a]pyrene-d12 Oven: 60 °C ->300 °C, 8 °C/min Injectionvolume: 1 µl Split: 10ml/min</p> <p>Date of analysis : 30 August 2003</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 18

TAR 01	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	/	/	/	/	/	/			
Toluene	73.0	66.0	66.6	67.9	65.7	66.7	67.7	2.73	4.03
Phenol	125	98.6	102	97.7	101	113	106	10.7	10.1
Indene	<1	<1	<1	<1	<1	<1			
Guaiacol	266	283	295	297	300	280	287	13.0	4.52
Naphthalene	9.77	6.96	7.35	7.22	7.29	9.90	8.08	1.37	16.9
Acenaphthylene	<1	<1	<1	<1	<1	<1			
4-methylguaiacol	186	177	182	175	180	129	172	21.2	12.3
Phenanthrene	1.23	0.950	0.960	1.08	0.950	1.40	1.10	0.185	16.93
Fluoranthene	<1	<1	<1	<1	<1	<1			
Pyrene	<1	<1	<1	<1	<1	<1			
Number of comp.	6	6	6	6	6	6	Mean of CV's %	10.8	
Total chrom. tars *	/	/	/	/	/	/			
Gravimetric tar	27060	36850	37715				33875	5918	17.5

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>HPLC with UV-DAD detector, KONTRON/BIO-TEK Column: UP 5 ODB-25k (C18 type)</p> <p>No sample preparation</p> <p>Oven temp.: 20 °C</p> <p>Eluent: Methanol/acetonitrile/water Injection volume: 10 µl</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO.: Coordinator

TAR 02	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean Value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	61.9	70.0	55.8				62.6	7.12	11.4
Toluene	574	534	462				523	56.8	10.8
Phenol	43.7	44.6	41.3				43.2	1.71	3.95
Indene	37.4	43.1	37.8				39.4	3.18	8.07
Guaiacol	110	109	104				108	3.21	2.99
Naphthalene	27.6	30.6	27.8				28.7	1.68	5.85
Acenaphthylene	0.238	0.248	0.223				0.236	0.013	5.32
4-methylguaiacol	47.0	45.1	43.4				45.2	1.80	3.99
Phenanthrene	0.052	0.042	0.035				0.043	0.009	20.1
Fluoranthene	0.010	0.008	0.006				0.008	0.002	27.3
Pyrene	0.009	0.007	0.006				0.007	0.002	25.1

Number of comp.	11	11	11				Mean of CV's %	11.4
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Total chrom. tars *	13100	11700	12700	16000	12400	12200	13017	1535	11.8
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Gravimetric tar	1252	1288	1682	1388	1564	1475	1442	165	11.5
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* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
GC/MS-SIM, HP 6890/5973 Internal standard added. Analysed direct by GC/MS full scan.

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 5

TAR 02	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	NA	NA	NA	NA	NA	NA			
Toluene	648	650	649	647	644	645	647	2.30	0.355
Phenol	83.7	83.8	83.5	83.2	82.7	82.7	83.2	0.494	0.593
Indene	66.8	67.1	67.6	67.3	67.0	67.1	67.2	0.274	0.408
Guaiacol	252	252	251	293	249	251	258	17.1	6.62
Naphthalene	47.9	48.5	48.5	48.1	48.2	48.3	48.2	0.253	0.525
Acenaphthylene	1.20	1.35	1.29	1.35	1.25	1.26	1.28	0.058	4.54
4-methylguaiacol	106	105	106	106	105	105	105	0.530	0.503
Phenanthrene	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Fluoranthene	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Pyrene	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			

Number of comp.	7	7	7	7	7	7	Mean of CV's %	1.94
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Total chrom. tars *	6912	6957	6946	6915	6882	6896	6918	28.6	0.41
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Gravimetric tar	9694	8004	9186	7568	5896	9628	8329	1476	17.7
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* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/FID, Shimadzu GC 17A, split injector GC-column: J&W DB 5.625, 30 m x 0.25 mm, df = 0.25 µm Oven temp.: 50 °C 4 min, 25 °C/min 150 °C 0 min, 5 °C/min 300 °C 10 min</p> <p>Analysis of pyridine GC-column: ZB-WAX, 30 m x 0.25 mm, df = 0.25 µm (Phenomenex) Oven temp.: 50 °C 5 min, 10 °C/min 240 °C</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 6

TAR 02	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	28.1	28.0	27.2	27.1	27.4	27.7	27.6	0.435	1.58
Toluene	553	565	556	565	555	549	557	6.60	1.18
Phenol	183	188	178	178	191	179	183	5.52	3.01
Indene	41.4	41.8	40.6	40.7	41.2	41.3	41.1	0.461	1.12
Guaiacol	274	263	254	266	265	264	264	6.42	2.43
Naphthalene	39.1	39.0	32.6	38.4	32.7	38.3	36.7	3.14	8.57
Acenaphthylene	-	-	-	-	-	-			
4-methylguaiacol									
Phenanthrene	-	-	-	-	-	-			
Fluoranthene	-	-	-	-	-	-			
Pyrene	-	-	-	-	-	-			

Number of comp.	6	6	6	6	6	6	Mean of CV's %	2.98
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Total chrom. tars *	6843	6790	6696	6853	6750	6886	6803	71.3	1.05
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Gravimetric tar	852	720	752	960	574	648	751	139	18.5
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* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
GC/FID, HP 5890, HP 3396A integrator Column: HP Ultra 2 (crosslinked 5% Ph Me silicone) 50 m x 0.32 mm, film thickness 0.52 µm Injector: Split/splitless injector 290 °C Carrier gas: Helium, injector pressure about 120 kPa, total flow about 20 ml/min Oven temp.: 50 °C 5 min, heating rate 3 °C /min to 160 °C, then 10 °C/min to 290 °C ISTD = n-dodecane

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 7

TAR 02	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine									
Toluene	532	540	535	536	543	539	537	3.65	0.679
Phenol	34.7	34.5	34.0	34.6	34.3	33.9	34.3	0.327	0.951
Indene									
Guaiacol	133	114	133	134	134	132	130	7.97	6.14
Naphthalene	25.0	25.0	25.0	25.0	25.2	24.9	25.0	0.098	0.393
Acenaphthylene									
4-methylguaiacol	59.5	58.7	57.5	58.4	58.1	56.7	58.2	0.971	1.67
Phenanthrene	< 2	< 2	< 2	< 2	< 2	< 2			
Fluoranthene	< 2	< 2	< 2	< 2	< 2	< 2			
Pyrene	< 2	< 2	< 2	< 2	< 2	< 2			
Number of comp.	5	5	5	5	5	5	Mean of CV's %	1.97	
Total chrom. tars *	5846	5818	5950	6021	5846	5746	5871	98.4	1.68
Gravimetric tar	3330								

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/MS, Agilent 6890 GC with FID for total tar, Agilent 5973 MSD, Agilent 7673 Autosampler Column: J&W Scientific DB-5MS, 30 m x 0.25 mm, 0,25 µm film Split injector 275 °C MSD transfer line 300 °C</p> <p>Sample was spiked with 100 µl of internal standard for MS analysis, no internal standard for FID/total GC tar. Column flow 1 ml/min, split ratio 30:1 Oven temp. 50 °C 5 min, ramp 8 °C/min to 250 °C, hold 0 min, ramp 10 °C/min to 300 °C hold 2,5 min</p> <p>Gravimetric tar: Roto Vap - Büchi Model 142 with temperature and vacuum control</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 16

TAR 02	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	0.59	0.63	0.65	0.69	0.69	0.66	0.652	0.038	5.86
Toluene	561	584	587	522	576	563	565	24.03	4.25
Phenol	51.5	50.8	51.6	50.9	51.6	50.2	51.1	0.564	1.10
Indene	36.4	37.5	39.4	37.9	39.4	36.6	37.9	1.33	3.51
Guaiacol	137	137	138	144	137	140	139	2.92	2.10
Naphthalene	28.5	29.0	29.8	29.3	30.0	28.8	29.2	0.579	1.98
Acenaphthylene									
4-methylguaiacol	60.3	57.7	58.3	57.2	58.5	57.7	58.3	1.11	1.90
Phenanthrene	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			
Fluoranthene	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			
Pyrene	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			
Number of comp.	7	7	7	7	7	7	Mean of CV's %		2.96
Total chrom. tars *	5174	5034					5104	99.0	1.94
Gravimetric tar	8086	4996	5992				6358	1577	24.8

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/MS, HP 5890/5971 Column: Ultra 2; 25 m, 0,2 mm ID, 0,33 µm film (Agilent)</p> <p>ISTD: Phenol-d5, phenanthrene-d10, benzo[a]pyrene-d12 Oven: 60 °C ->300 °C, 8 °C/min Injectionvolume: 1 µl Split: 10ml/min</p> <p>Date of analysis : 30 August 2003</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 18

TAR 02	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	/	/	/	/	/	/			
Toluene	640	600	599	601	597	494	589	49.1	8.34
Phenol	71.2	54.3	54.4	60.8	54.4	59.9	59	6.59	11.14
Indene	62.2	55.6	57.5	57.7	56.7	54.8	57.4	2.59	4.52
Guaiacol	145	149	149	150	150	143	148	2.94	1.99
Naphthalene	48.6	45.5	45.3	46.4	45.3	44.7	46.0	1.40	3.05
Acenaphthylene	<1	<1	<1	<1	<1	<1			
4-methylguaiacol	63.8	46.5	46.1	46.1	46.9	54	50.6	7.17	14.2
Phenanthrene	<1	<1	<1	<1	<1	<1			
Fluoranthene	<1	<1	<1	<1	<1	<1			
Pyrene	10.1	8.63	8.6	8.61	8.57	8.4	8.82	0.633	7.18
Number of comp.	7	7	7	7	7	7	Mean of CV's %		7.20
Total chrom. tars *	/	/	/	/	/	/			
Gravimetric tar	14298	23982	26494				21591	6440	29.8

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>HPLC with UV-DAD detector, KONTRON/BIO-TEK Column: UP 5 ODB-25k (C18 type)</p> <p>No sample preparation</p> <p>Oven temp.: 20 °C</p> <p>Eluent: Methanol/acetonitrile/water Injection volume: 10 µl</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO.: Coordinator

TAR 03	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	1.10	1.05	1.09				1.08	0.03	2.45
Toluene	41.5	31.3	44.4				39.1	6.88	17.6
Phenol	0.285	0.292	0.288				0.29	0.00	1.22
Indene	12.8	11.2	12.4				12.1	0.83	6.86
Guaiacol	<0.05	<0.05	<0.05						
Naphthalene	141	128	138				136	6.81	5.02
Acenaphthylene	18.1	16.8	17.7				17.5	0.67	3.80
4-methylguaiacol	<0.05	<0.05	<0.05						
Phenanthrene	19.6	19	19.8				19.5	0.42	2.14
Fluoranthene	5.28	5.49	5.16				5.31	0.17	3.15
Pyrene	5.26	5.54	5.09				5.30	0.23	4.29
Number of comp.	9	9	9				Mean of CV's %		5.17
Total chrom. tars *	193	171	237	205	243	258	218	33.4	15.3
Gravimetric tar	102	68	78	88	76	92	84.0	12.3	14.7

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
GC/MS-SIM, HP 6890/5973 Internal standard added. Analysed direct by GC/MS full scan

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 5

TAR 03	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	1.11	1.12	1.08	1.12	1.15	1.09	1.11	0.024	2.19
Toluene	30.7	30.6	30.9	30.8	30.6	30.8	30.7	0.109	0.356
Phenol	0.231	0.231	0.231	0.211	0.231	0.221	0.226	0.008	3.70
Indene	10.8	10.8	10.9	10.9	10.8	10.9	10.9	0.048	0.437
Guaiacol	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Naphthalene	117	117	118	117	117	118	117	0.503	0.429
Acenaphthylene	18.2	18.2	18.5	18.3	18.2	18.3	18.3	0.104	0.571
4-methylguaiacol	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Phenanthrene	21.9	22.0	22.2	22.0	22.0	22.1	22.1	0.082	0.372
Fluoranthene	7.02	7.15	7.20	7.14	7.12	7.08	7.12	0.061	0.857
Pyrene	7.90	7.92	7.97	7.93	7.90	7.93	7.92	0.027	0.338
Number of comp.	9	9	9	9	9	9	Mean of CV's %		1.03
Total chrom. tars *	289	289	292	290	289	290	290	1.32	0.456
Gravimetric tar	154	122	134	102	156	142	135	20.5	15.2

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/FID, Shimadzu GC 17A, split injector GC-column: J&W DB 5.625, 30m x 0.25 mm, df = 0.25 µm Oven temp.: 50 °C 4 min, 25 °C/min 150 °C 0 min, 5 °C/min 300 °C 10 min</p> <p>Analysis of pyridine GC-column: ZB-WAX, 30 m x 0.25 mm, df = 0.25 µm (Phenomenex) Oven temp.: 50 °C 5 min, 10 °C/min 240 °C</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 6

TAR 03	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	-	-	-	-	-	-			
Toluene	29.0	30.5	30.4	29.2	29.2	28.9	29.5	0.714	2.42
Phenol	-	-	-	-	-	-			
Indene	11.5	11.5	11.5	11.6	11.4	11.5	11.5	0.049	0.426
Guaiacol	-	-	-	-	-	-			
Naphthalene	116	116	116	117	116	116	116	0.375	0.324
Acenaphthylene	17.1	17.2	17.3	17.2	17.0	17.2	17.2	0.088	0.510
4-methylguaiacol									
Phenanthrene	21.8	21.7	21.8	21.5	21.5	21.8	21.7	0.142	0.655
Fluoranthene	6.93	6.97	6.92	6.68	6.62	7.16	6.88	0.199	2.89
Pyrene	8.15	7.63	7.72	7.63	7.48	6.36	7.50	0.601	8.02
Number of comp.	7	7	7	7	7	7	Mean of CV's %		2.18
Total chrom. tars *	243	250	251	250	241	245	247	4.22	1.71
Gravimetric tar	90	86	48	64	110	45	73.8	25.7	34.9

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition

GC/FID, HP 5890, HP 3396A integrator
 Column: HP Ultra 2 (crosslinked 5% Ph Me silicone) 50 m x 0.32 mm, film thickness 0.52 µm
 Injector: Split/splitless injector 290 °C
 Carrier gas: Helium, injector pressure about 120 kPa, total flow about 20 ml/min
 Oven temp.: 50 °C 5 min, heating rate 3 °C /min to 160 °C, then 10 °C/min to 290 °C
 ISTD = n-dodecane

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 7

TAR 03	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	< 4	< 4	< 4	< 4	< 4	< 4			
Toluene	29.8	29.4	29.4	29.8	29.8	29.5	29.6	0.204	0.689
Phenol	< 20	< 20	< 20	< 20	< 20	< 20			
Indene									
Guaiacol	< 5	< 5	< 5	< 5	< 5	< 5			
Naphthalene	108	106	105	107	105	105	106	1.49	1.41
Acenaphthylene									
4-methylguaiacol	< 5	< 5	< 5	< 5	< 5	< 5			
Phenanthrene	21.9	19.8	19.9	19.5	19.3	19.0	19.9	1.03	5.19
Fluoranthene	6.9	6.2	6.0	6.0	6.0	5.8	6.15	0.389	6.32
Pyrene	6.2	5.6	5.4	5.8	5.7	5.6	5.72	0.271	4.75
Number of comp.	5	5	5	5	5	5	Mean of CV's %		3.67
Total chrom. tars *	230	236	233	239	244	242	237	5.35	2.26
Gravimetric tar	180								

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/MS, Agilent 6890 GC with FID for total tar, Agilent 5973 MSD, Agilent 7673 Autosampler Column: J&W Scientific DB-5MS, 30 m x 0.25 mm, 0,25 µm film Split injector 275 °C MSD transfer line 300 °C</p> <p>Sample was spiked with 100 µl of internal standard for MS analysis, no internal standard for FID/total GC tar. Column flow 1 ml/min, split ratio 30:1 Oven temp. 50 °C 5 min, ramp 8 °C/min to 250 °C, hold 0 min, ramp 10 °C/min to 300 °C hold 2,5 min</p> <p>Gravimetric tar: Roto Vap - Büchi Model 142 with temperature and vacuum control</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 16

TAR 03	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	0.75	0.80	0.75	0.84	0.80	0.83	0.795	0.038	4.82
Toluene	28.7	30.1	27.1	28.6	26.6	26.5	27.9	1.42	5.07
Phenol	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00			
Indene	11.1	11.2	11.0	11.0	10.8	10.7	11.0	0.203	1.85
Guaiacol	< 2.5	< 2.5	< 2.5	< 2.5	< 2.5	< 2.5			
Naphthalene	114	115	115	115	112	112	114	1.38	1.21
Acenaphthylene									
4-methylguaiacol	3.15	3.18	3.16	3.19	3.16	3.13	3	0.0	0.676
Phenanthrene	20.3	20.4	20.4	20.5	20.5	20.6	20.4	0.129	0.630
Fluoranthene	6.72	7.11	7.11	7.08	7.35	7.39	7.13	0.240	3.36
Pyrene	6.64	6.91	7.02	7.34	7.25	7.28	7.07	0.269	3.81
Number of comp.	8	8	8	8	8	8	Mean of CV's %		2.68
Total chrom. tars *	305	294					300	7.78	2.60
Gravimetric tar	226	161	89				159	68.4	43.1

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/MS, HP 5890/5971 Column: Ultra 2; 25 m, 0,2 mm ID, 0,33 µm film (Agilent)</p> <p>ISTD: Phenol-d5, phenanthrene-d10, benzo[a]pyrene-d12 Oven: 60 °C ->300 °C, 8 °C/min Injectionvolume: 1 µl Split: 10ml/min</p> <p>Date of analysis: 30 August 2003</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 18

TAR 03	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	<5	<5	<5	<5	<5	<5			
Toluene	28.8	30.1	28.3	26.8	29.9	26.5	28.4	1.52	5.34
Phenol	<1	<1	<1	<1	<1	<1			
Indene	12.1	13.7	12.7	13.9	13.8	12	13.0	0.876	6.72
Guaiacol	/	/	/	/	/	/			
Naphthalene	139	142	141	141	141	134	140	2.94	2.11
Acenaphthylene	20.3	20.2	19.1	20.2	19.9	18.5	19.70	0.735	3.73
4-methylguaiacol	/	/	/	/	/	/			
Phenanthrene	22.8	23.1	22.6	22.8	23.1	23	22.90	0.200	0.873
Fluoranthene	4.89	5.92	5.99	6.59	6.24	6.1	5.955	0.573	9.62
Pyrene	6.92	6.98	6.68	7.07	6.72	6.56	6.822	0.198	2.90
Number of comp.	7	7	7	7	7	7	Mean of CV's %		4.47
Total chrom. tars *	/	/	/	/	/	/			
Gravimetric tar	10612	8334	11826				10257	1773	17.3

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>HPLC with UV-DAD detector, KONTRON/BIO-TEK Column: UP 5 ODB-25k (C18 type)</p> <p>No sample preparation</p> <p>Oven temp.: 20 °C</p> <p>Eluent: Methanol/acetonitrile/water Injection volume: 10 µl</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO.: Coordinator

TAR 04	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	<0.1	<0.1	<0.1						
Toluene	0.179	0.213	0.413				0.268	0.126	47.1
Phenol	<0.05	<0.05	<0.05						
Indene	0.045	0.051	0.073				0.056	0.014	25.6
Guaiacol	<0.05	<0.05	<0.05						
Naphthalene	1.44	1.54	1.98				1.653	0.287	17.4
Acenaphthylene	0.079	0.083	0.098				0.087	0.010	11.5
4-methylguaiacol	<0.05	<0.05	<0.05						
Phenanthrene	0.205	0.213	0.253				0.224	0.026	11.5
Fluoranthene	0.046	0.049	0.069				0.055	0.013	23.4
Pyrene	0.047	0.049	0.067				0.055	0.011	20.1
Number of comp.	7	7	7				Mean of CV's %		22.4
Total chrom. tars *	45.5	48.3	43.9	51.8	51.3	40.6	46.9	4.38	9.34
Gravimetric tar	30	26	24	34	26	38	29.7	5.43	18.3

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition

GC/MS-SIM, HP 6890/5973
Internal standard added. Analysed direct by GC/MS full scan

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 5

TAR 04	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Toluene	0.201	0.255	0.232	0.263	0.255	0.263	0.245	0.024	9.93
Phenol	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Indene	0.050	0.058	0.050	0.043	0.050	0.036	0.048	0.007	15.7
Guaiacol	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Naphthalene	1.60	1.56	1.55	1.55	1.53	1.53	1.55	0.026	1.65
Acenaphthylene	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
4-methylguaiacol	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Phenanthrene	0.335	0.349	0.371	0.306	0.321	0.314	0.333	0.024	7.29
Fluoranthene	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Pyrene	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Number of comp.	4	4	4	4	4	4	Mean of CV's %		8.63
Total chrom. tars *	18.3	18.1	17.9	17.5	17.2	17.2	17.7	0.466	2.63
Gravimetric tar	10.0	-2.0	4.0	14.0	26.0	13.6	10.9	9.59	87.7

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition

GC/FID, Shimadzu GC 17A, split injector
 GC-column: J&W DB 5.625, 30m x 0.25 mm, df = 0.25 µm
 Oven temp.: 50 °C 4 min, 25 °C/min 150 °C 0 min, 5 °C/min 300 °C 10 min

Analysis of pyridine
 GC-column: ZB-WAX, 30 m x 0.25 mm, df = 0.25 µm (Phenomenex)
 Oven temp.: 50 °C 5 min, 10 °C/min 240 °C

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 6

TAR 04	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	-	-	-	-	-	-			
Toluene	-	-	-	-	-	-			
Phenol	-	-	-	-	-	-			
Indene	-	-	-	-	-	-			
Guaiacol	-	-	-	-	-	-			
Naphthalene	1.68	1.65	1.78	1.66	1.66	1.67	1.68	0.048	2.88
Acenaphthylene	-	-	-	-	-	-			
4-methylguaiacol									
Phenanthrene	-	-	-	-	-	-			
Fluoranthene	-	-	-	-	-	-			
Pyrene	-	-	-	-	-	-			

Number of comp.	1	1	1	1	1	1	Mean of CV's %	2.88
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Total chrom. tars *	19.1	19.4	20.0	19.0	19.2	19.6	19.4	0.358	1.85
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Gravimetric tar	0	0	0	0			0	0.00	
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* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/FID, HP 5890, HP 3396A integrator Column: HP Ultra 2 (crosslinked 5% Ph Me silicone) 50 m x 0.32 mm, film thickness 0.52 µm Injector: Split/splitless injector 290 °C Carrier gas: Helium, injector pressure about 120 kPa, total flow about 20 ml/min Oven temp.: 50 °C 5 min, heating rate 3 °C /min to 160 °C, then 10 °C/min to 290 °C ISTD = n-dodecane</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 7

TAR 04	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	< 4	< 4	< 4	< 4	< 4	< 4			
Toluene	< 2	< 2	< 2	< 2	< 2	< 2			
Phenol	< 20	< 20	< 20	< 20	< 20	< 20			
Indene									
Guaiacol	< 5	< 5	< 5	< 5	< 5	< 5			
Naphthalene									
Acenaphthylene									
4-methylguaiacol	< 8	< 8	< 8	< 8	< 8	< 8			
Phenanthrene	< 5	< 5	< 5	< 5	< 5	< 5			
Fluoranthene	< 2	< 2	< 2	< 2	< 2	< 2			
Pyrene	< 2	< 2	< 2	< 2	< 2	< 2			
Number of comp.	0	0	0	0	0	0	Mean of CV's %		
Total chrom. tars *	5	8	8	8	8	9	8	1.37	17.82
Gravimetric tar	-90								

* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/MS, Agilent 6890 GC with FID for total tar, Agilent 5973 MSD, Agilent 7673 Autosampler Column: J&W Scientific DB-5MS, 30 m x 0.25 mm, 0,25 µm film Split injector 275 °C MSD transfer line 300 °C</p> <p>Sample was spiked with 100 µl of internal standard for MS analysis, no internal standard for FID/total GC tar. Column flow 1 ml/min, split ratio 30:1 Oven temp. 50 °C 5 min, ramp 8 °C/min to 250 °C, hold 0 min, ramp 10 °C/min to 300 °C hold 2,5 min</p> <p>Gravimetric tar: Roto Vap - Büchi Model 142 with temperature and vacuum control</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 16

TAR 04	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Pyridine	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Toluene	0.23	0.24	0.25	0.23	0.24	0.21	0.2	0.01	5.9
Phenol	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00			
Indene	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Guaiacol	< 2.50	< 2.50	< 2.50	< 2.50	< 2.50	< 2.50			
Naphthalene	1.45	1.45	1.48	1.40	1.49	1.44	1.45	0.032	2.2
Acenaphthylene									
4-methylguaiacol	< 2.50	< 2.50	< 2.50	< 2.50	< 2.50	< 2.50			
Phenanthrene	0.21	0.20	0.21	0.21	0.21	0.20	0.21	0.005	2.50
Fluoranthene	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			
Pyrene	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			

Number of comp.	3	3	3	3	3	3	Mean of CV's %	3.52
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Total chrom. tars *	< 5	< 5						
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Gravimetric tar	< 50	< 50	< 50					
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* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>GC/MS, HP 5890/5971 Column: Ultra 2; 25 m, 0,2 mm ID, 0,33 µm film (Agilent)</p> <p>ISTD: Phenol-d5, phenanthrene-d10, benzo[a]pyrene-d12 Oven: 60 °C ->300 °C, 8 °C/min Injectionvolume: 1 µl Split: 10ml/min</p> <p>Date of analysis : 30 August 2003</p>

Round Robin Test on Tar, September 2003

Round 02 - Real tar

LABORATORY CODE NO. 18

TAR 04	Measured value [µg/ml]						Statistics		
	1	2	3	4	5	6	Mean value [µg/ml]	STDev [µg/ml]	CV %
Sub sample No.									
Pyridine	<5	<5	<5	<5	<5	<5			
Toluene	<1	<1	<1	<1	<1	<1			
Phenol	<1	<1	<1	<1	<1	<1			
Indene	<1	<1	<1	<1	<1	<1			
Guaiacol	/	/	/	/	/	/			
Naphthalene	<1	<1	<1	<1	<1	<1			
Acenaphthylene	<1	<1	<1	<1	<1	<1			
4-methylguaiacol	/	/	/	/	/	/			
Phenanthrene	0.41	0.39	0.32	0.29	0.32	0.38	0.35	0.048	13.6
Fluoranthene	<1	<1	<1	<1	<1	<1			
Pyrene	<1	<1	<1	<1	<1	<1			

Number of comp.	1	1	1	1	1	1	Mean of CV's %	13.6
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Total chrom. tars *	/	/	/	/	/	/		
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Gravimetric tar	10586	10428	5786				8933	2726	30.5
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* Total chromatographable tars with molecule weight mw. > 79 g/mol (> benzene) calculated as naphthalene.

Used apparatus, sample preparation and analysis condition
<p>HPLC with UV-DAD detector, KONTRON/BIO-TEK Column: UP 5 ODB-25k (C18 type)</p> <p>No sample preparation</p> <p>Oven temp.: 20 °C</p> <p>Eluent: Methanol/acetonitrile/water Injection volume: 10 µl</p>

5.2. Evaluation of results from round 02

The aim of the evaluation is to show an illustrative picture of the individual laboratories' performance compared to each other. In addition to this, the primary aim of the evaluation at single round level is to identify outliers in the reported results. Primarily, focus is on comparison of accuracy and repeatability of the individual labs and the reproducibility between the labs. This includes a control of the presence of systematic errors. Outliers in the reported data should be eliminated from the data set that qualifies for an estimate of the accuracy and reproducibility of the tar measuring method.

The following graphs show the results of the laboratories' as the range of observations (the difference between the largest observation and the smallest observation), where the data mark indicates the mean value (mean of lab).

- The bold, red line in the graph represents the mean value of the participating laboratories' results (mean of lab means).
- The two thin red lines represent the limits of the 95% confidence interval (CI) of the mean of lab means. The 95% CI is a range of values the mean of lab means takes with probability 95%.

The statistic analysis of the reported data is carried out in accordance with ISO 5725-2:1994. The statistic analysis includes testing for outlying lab means and an analysis of variances. Variances are examined for outliers according to Cochran's test, the homogeneity of variances is tested and, finally, the statistic significance of differences between labs is tested. Outlying lab means are tested by means of three different methods: Dixon's test, Nalimov t-test and Grubb's test.

According to ISO 5725 the definition of outlier depends on the probability level (P) of the Cochran's and Dixon's test. If $P \leq 1\%$, i.e. the test statistic is larger than its 1% critical value. In this case, the item is called a statistic outlier which can be discarded after thoroughly investigation.

The received results were examined by the coordinator. After this it was investigated if errors could be explained and corrected. The outcome of this investigation appears from comments on the individual laboratories' results in paragraph 4.2. Remaining outliers which cannot be explained were discarded as real outliers not belonging to the experiment. Particularly in cases where several unexplained outliers occurred at different levels within the same lab, it was decided to discard the data from such an outlying laboratory. In some cases laboratories have withdrawn their results. In the following, all outliers and withdrawn or suspect results are printed in red colour.

Finally, the methods precision is calculated. Within labs the analysis precision is expressed by the repeatability value "r" - the value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie with a probability of 95%.

The repeatability conditions are the conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

Between labs the analysis precision is expressed by the reproducibility value "R" - the value below which the absolute difference between two single test results obtained under reproducibility conditions may be expected to lie with a probability of 95%. The reproducibility conditions are the conditions where test results are obtained with the same method on identical test material in different laboratories with different operators using different equipment. These values "r" and "R" appear from paragraph 4.3.

5.2.1. TAR 01, Pyridine

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	9.85	7.11	8.09				8.35	1.388	3.449
Lab 5	NA	NA	NA	NA	NA	NA			
Lab 6	8.89	8.31	7.78	9.78	9.13	12.3			
Lab 7	2.10	2.20	1.60	1.10	2.30	1.80	1.85	0.451	0.473
Lab 16	3.99	3.81	3.85	3.63	3.80	3.71	3.798	0.123	0.129
Lab 18	NA	NA	NA	NA	NA	NA			

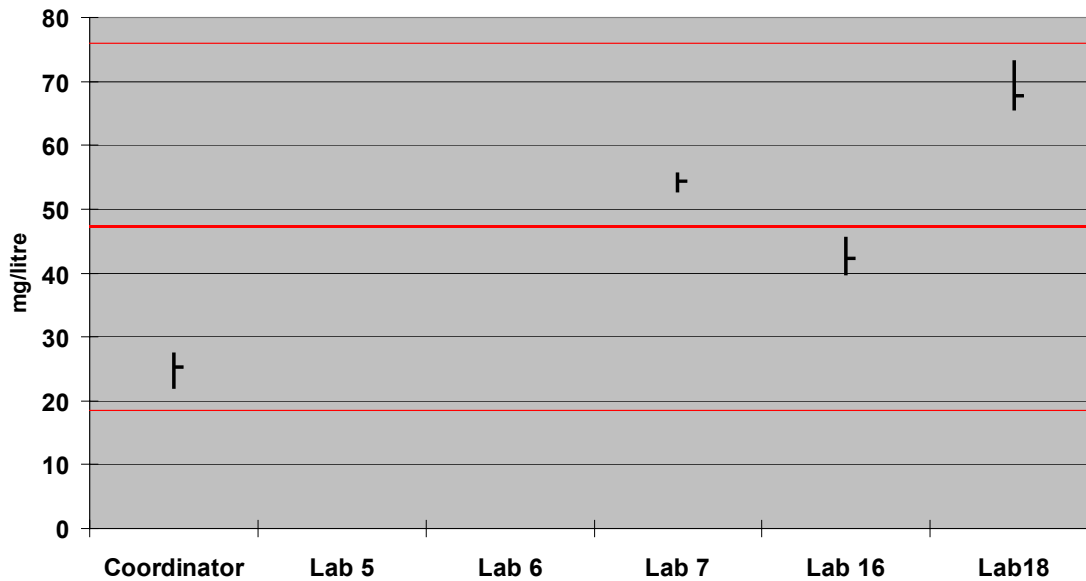
The table above shows the results from all six labs. In this case, the statistic analysis detected no outliers, but it was decided not to use Lab 6's results due to uncertainty in compound identification, as mentioned in paragraph 3.3. After this, the coordinator was detected as an outlier. As a consequence of that, results from only two labs remained and it was decided not to carry out further analysis on the pyridine results.

5.2.2. TAR 01, Toluene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	27.3	26.2	22.2				25.233	2.684	6.667
Lab 5	52.4	52.7	51.6	51.1	52.1	52.9			
Lab 6	101	98.6	97.8	100	98.1	107			
Lab 7	55.5	54.0	52.9	54.5	54.9	54.0	54.3	0.892	0.936
Lab 16	45.4	42.7	43.3	40.7	42.3	39.9	42.383	1.952	2.048
Lab 18	73.0	66.0	66.6	67.9	65.7	66.7	67.65	2.728	2.863
Mean of lab means							47.39	18.02	28.67

The table above shows the results from all six labs, but as Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 01 - Toluene



TESTING OF OUTLYING LAB MEANS

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

TESTING OF VARIANCES

Cochran test: Pass # 1: No outlier detected
 Bartlett test: Lab variances homogeneous?
 Yes (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	47.39 mg/l	Within labs STDev:	2.096 mg/l
StDev of lab means:	18.02 mg/l	Between labs STDev:	16.40 mg/l
Half width 95% CI:	28.67 mg/l		
Upper limit of 95% CI	76.066 mg/l	Lower limit of 95% CI	18.717 mg/l

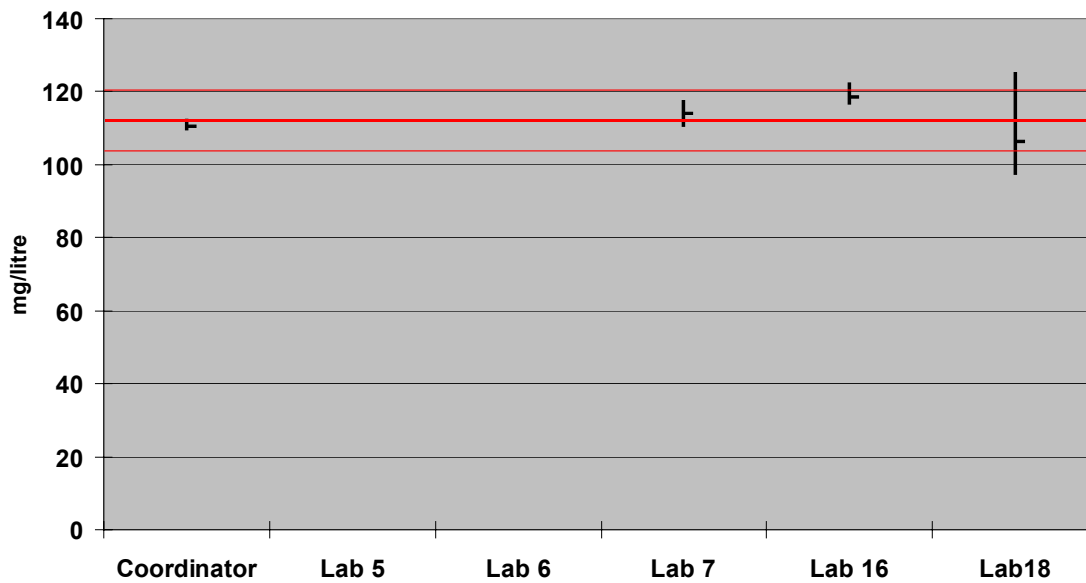
Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
50	2.096	4.2	5.87	11.7	16.53	33.1	46.28	92.6

5.2.3. TAR 01, Phenol

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	112	110	110				110.67	1.155	2.868
Lab 5	157	158	157	157	159	157			
Lab 6	148	142	142	147	140	147			
Lab 7	115	117	112	111	115	114	114.08	2.179	2.287
Lab 16	117	118	119	118	122	117	118.5	1.871	1.963
Lab 18	125	98.6	102	97.7	101	113	106.22	10.718	11.248
Mean of lab means							112.37	5.205	8.282

The table above shows the results from all six labs, but as Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 01 - Phenol



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means: 112.37 mg/l Within labs STDev: 6.031 mg/l
 StDev of lab means: 5.205 mg/l Between labs STDev: 4.888 mg/l
 Half width 95% CI: 8.282 mg/l
 Upper limit of 95% CI 120.65 mg/l Lower limit of 95% CI 104.08 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
110	6.031	5.5	16.89	15.4	7.763	7.1	21.74	19.8

5.2.4. TAR 01, Indene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	3.67	2.29	2.68						
Lab 5	14.5	14.0	7.31	13.1	7.66	12.9			
Lab 6	12.9	13.3	13.3	13.5	12.5	13.9			
Lab 7									
Lab 16	4.53	4.39	4.49	4.15	4.35	4.28			
Lab 18	< 1	< 1	< 1	< 1	< 1	< 1			

The coordinator, Lab 7 and Lab 16 use GC/MS for analysis. Lab 5 and Lab 6 use GC/FID. Lab 18 uses HPLC-UV.

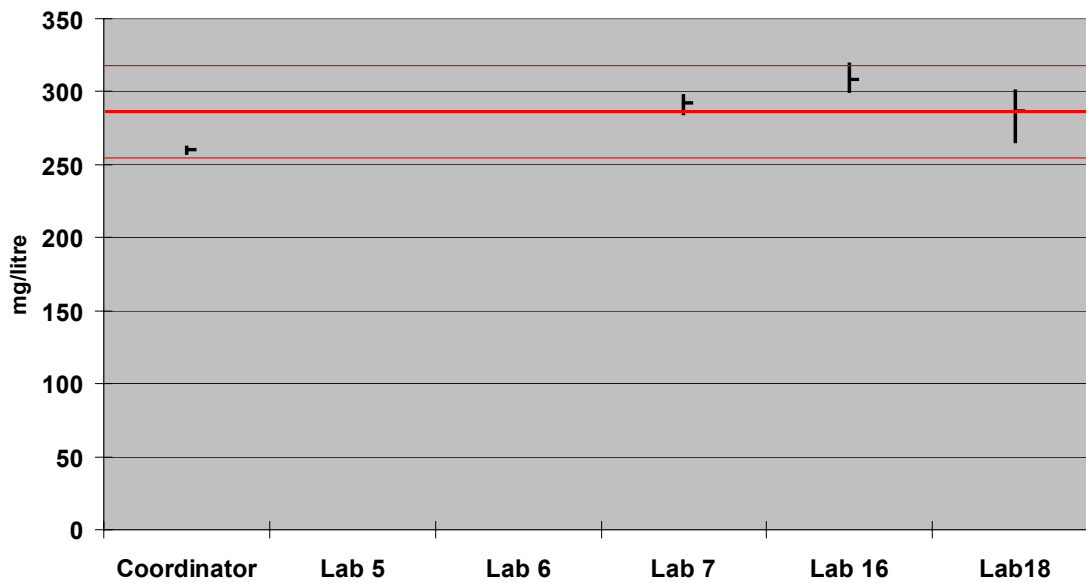
As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, only results from two labs remained for analysis. Due to the few numbers it was decided not to carry out further analysis on the indene results.

5.2.5. TAR 01, Guaiacol

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	258	261	262				260	2.082	5.171
Lab 5	352	352	350	350	356	351			
Lab 6	311	308	306	310	305	314			
Lab 7	296	297	286	285	293	295	292	5.133	5.387
Lab 16	301	310	309	310	319	304	309	6.178	6.483
Lab 18	266	283	295	297	300	280	287	12.95	13.59
Mean of lab means							287	20.1	32.0

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 01 - Guaiacol



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	287 mg/l	Within labs STDev:	8.296 mg/l
StDev of lab means:	20.1 mg/l	Between labs STDev:	17.326 mg/l
Half width 95% CI:	32 mg/l		
Upper limit of 95% CI	319 mg/l	Lower limit of 95% CI	255 mg/l

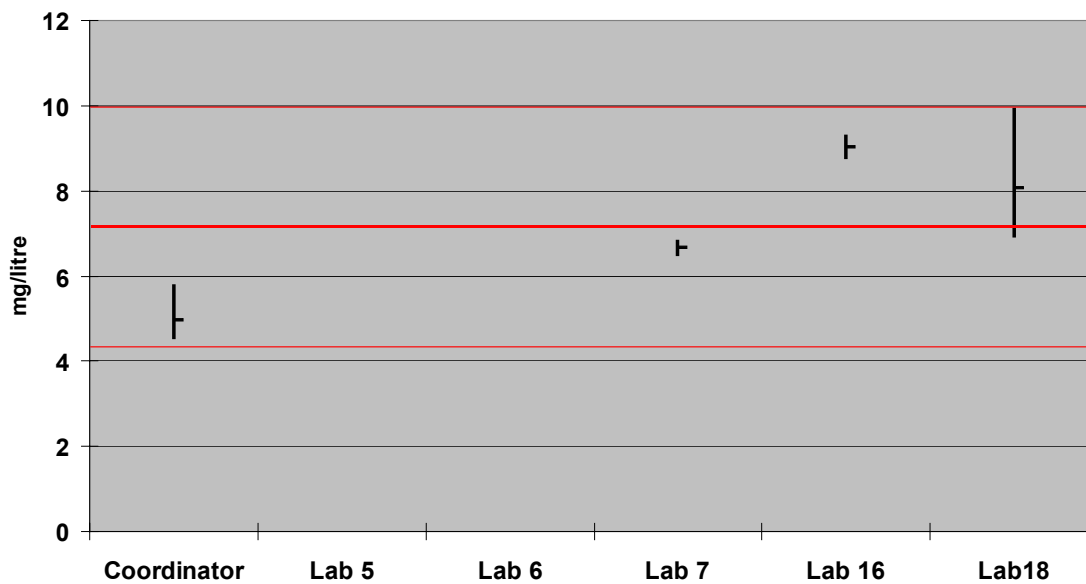
Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
290	8.296	2.9	23.23	8.0	19.21	6.6	53.79	18.5

5.2.6. TAR 01, Naphthalene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	5.78	4.55	4.62				4.983	0.691	1.716
Lab 5	20.1	19.9	19.9	20.1	39.5	19.6			
Lab 6	10.8	10.5	10.7	10.2	10.6	10.3			
Lab 7	6.80	6.80	6.50	6.60	6.60	6.70	6.667	0.121	0.127
Lab 16	9.28	9.08	9.16	8.79	8.96	8.91	9.030	0.178	0.187
Lab 18	9.77	6.96	7.35	7.22	7.29	9.90	8.082	1.365	1.433
Mean of lab means							7.19	1.763	2.805

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 01 - Naphthalene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Pass # 2: Coordinator is an outlier
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	7.190 mg/l	Within labs STDev:	0.786 mg/l
StDev of lab means:	1.763 mg/l	Between labs STDev:	1.557 mg/l
Half width 95% CI:	2.805 mg/l		
Upper limit of 95% CI	9.996 mg/l	Lower limit of 95% CI	4.385 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
7	0.786	11.2	2.2	31.4	1.744	24.9	4.884	69.8

5.2.7. TAR 01, Acenaphthylene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	1.21	1.23	1.25						
Lab 5	8.22	8.09	7.18	7.92	8.70	5.53			
Lab 6	4.50	4.44	4.50	4.45	4.20	4.57			
Lab 7									
Lab 16									
Lab 18	<1	<1	<1	<1	<1	<1			

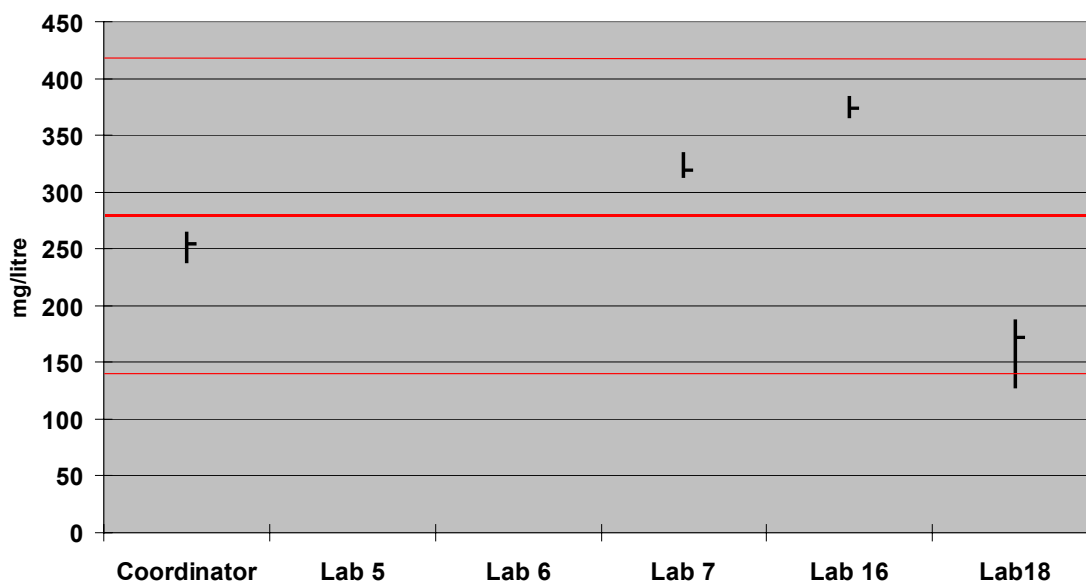
As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, only results from two labs remained for analysis. Due to the few numbers it was decided not to carry out further analysis on the acenaphthylene results.

5.2.8. TAR 01, 4-methylguaiacol (creosol)

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	239	260	264				254	13.43	33.36
Lab 5	389	388	384	386	391	385			
Lab 6									
Lab 7	334	319	314	314	318	315	319	7.73	8.11
Lab 16	372	372	375	374	383	367	374	5.27	5.53
Lab 18	186	177	182	175	180	129	172	21.17	22.22
Mean of lab means							279.6	87.1	138.6

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 01 - 4-methylguaiacol



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	279.63 mg/l	Within labs STDev:	13.372 mg/l
StDev of lab means:	87.072 mg/l	Between labs STDev:	93.103 mg/l
Half width 95% CI:	138.551 mg/l		
Upper limit of 95% CI	418.18 mg/l	Lower limit of 95% CI	141.078 mg/l

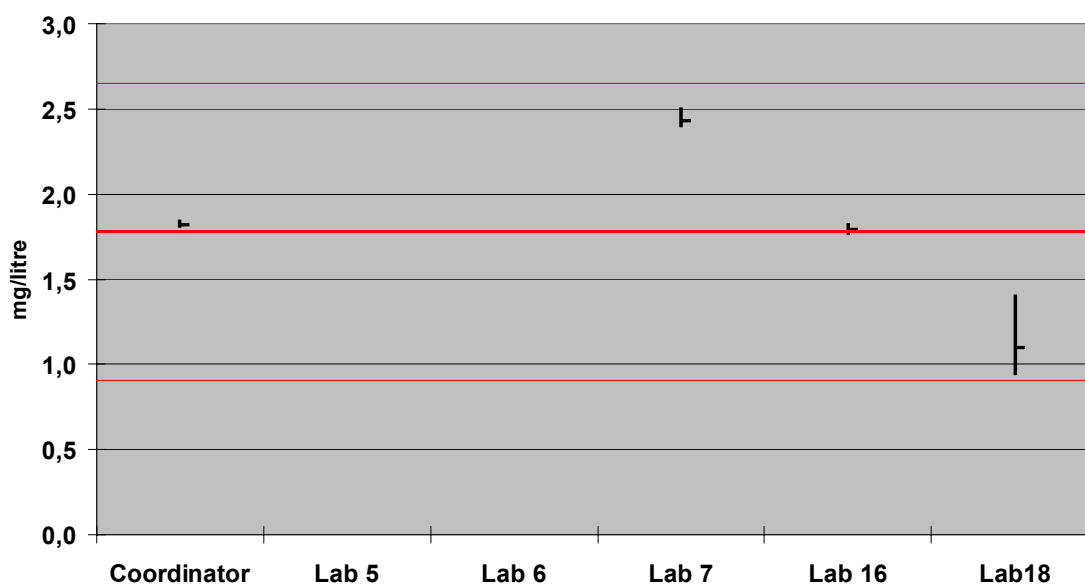
Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
280	13.372	4.8	37.44	13.4	94.06	33.6	263.4	94

5.2.9. TAR 01, Phenanthrene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	1.84	1.81	1.81				1.82	0.017	0.043
Lab 5	3.03	3.29	3.12	3.29	4.06	3.54			
Lab 6	4.66	4.40	4.50	4.51	5.14	4.39			
Lab 7	2.50	2.40	2.40	2.40	2.40	2.50	2.433	0.052	0.054
Lab 16	1.77	1.79	1.79	1.82	1.80	1.78	1.792	0.017	0.018
Lab 18	1.23	0.950	0.960	1.08	0.950	1.40	1.095	0.185	0.194
Mean of lab means							1.785	0.547	0.87

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 01 - Phenanthrene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Pass # 2: Lab 7 is an outlier
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	1.785 mg/l	Within labs STDev:	0.105 mg/l
StDev of lab means:	0.547 mg/l	Between labs STDev:	0.589 mg/l
Half width 95% CI:	0.870 mg/l		
Upper limit of 95% CI	2.655 mg/l	Lower limit of 95% CI	0.915 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
2	0.105	5.3	0.294	14.7	0.598	29.9	1.675	83.8

5.2.10. TAR 01, Fluoranthene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.412	0.397	0.408						
Lab 5	1.17	2.09	2.58	3.10	3.73	2.06			
Lab 6	10.1	9.86	8.46	8.93	8.62	9.43			
Lab 7	< 2	< 2	< 2	< 2	< 2	< 2			
Lab 16	0.43	0.43	0.43	0.42	0.43	0.41			
Lab 18	<1	<1	<1	<1	<1	<1			

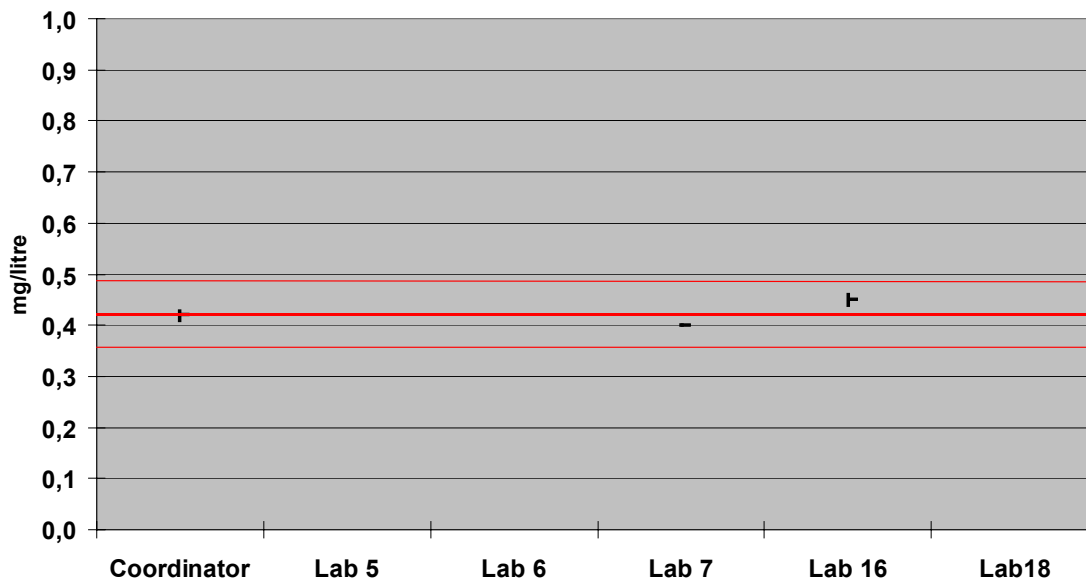
As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, only results from two labs remained for analysis. Due to the few numbers it was decided not to carry out further analysis on the fluoranthene results.

5.2.11. TAR 01, Pyrene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.429	0.41	0.427				0.422	0.010	0.026
Lab 5	3.82	0.00	3.05	5.75	3.26	2.88			
Lab 6	1.55	4.01	6.04	5.95	3.43	4.13			
Lab 7	0.400	0.400	0.400	0.400	0.400	0.400	0.4	0	0
Lab 16	0.44	0.46	0.46	0.44	0.46	0.45	0.452	0.010	0.010
Lab 18	<1	<1	<1	<1	<1	<1			
Mean of lab means							0.425	0.026	0.064

As Lab 5 withdrew its individual results on updraft tar analysis on, and as the results from Lab 6 were not used as previously mentioned, the statistic analysis is based on the results from the three remaining labs.

TAR 01 - Pyrene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

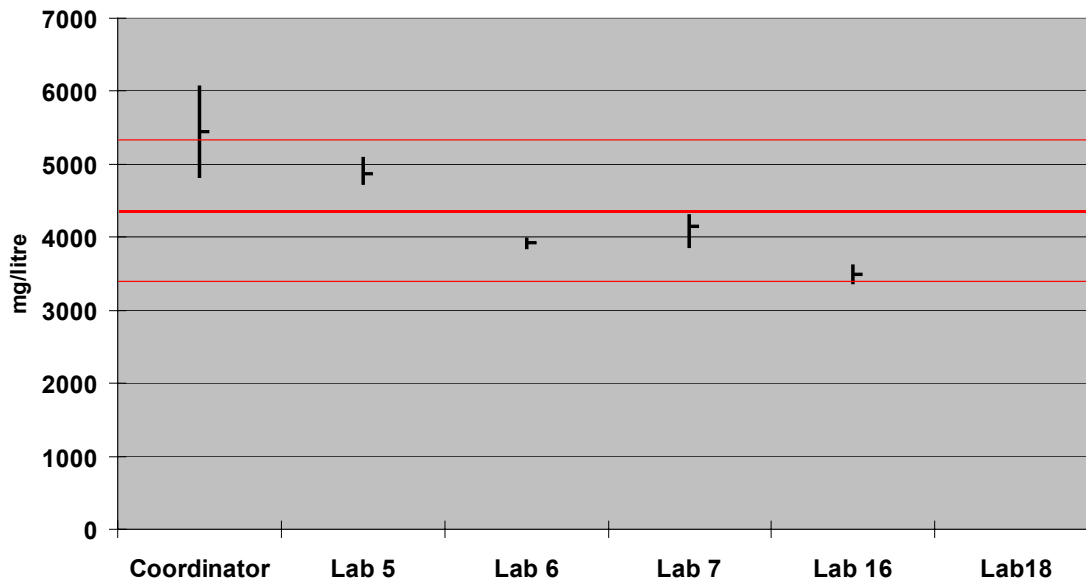
Mean of lab means: 0.425 mg/l Within labs STDev: 0.008 mg/l
 StDev of lab means: 0.026 mg/l Between labs STDev: 0.029 mg/l
 Half width 95% CI: 0.064 mg/l
 Upper limit of 95% CI 0.489 mg/l Lower limit of 95% CI 0.360 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
0.5	0.008	1.6	0.022	4.4	0.030	6.0	0.084	16.8

5.2.12. TAR 01, Total chromatographable tars calculated as naphthalene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	5040	6050	5760	4840	5130	5830	5442	498.5	523.2
Lab 5	4764	4860	4801	4956	5076	4743	4867	128.1	134.4
Lab 6	3966	3974	3956	3966	3871	3858	3932	52.63	55.23
Lab 7	3875	4008	4140	4286	4277	4287	4146	172.7	181.3
Lab 16	3385	3601					3493	152.7	1372
Lab 18	/	/	/	/	/	/			
Mean of lab means							4376	776	963

TAR 01 - Total GC tar



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test:
 Pass # 1: Coord. is an outlier at $\alpha = 0.01$ and 0.05
 Pass # 2: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No ($\alpha = 0.05$)
 No ($\alpha = 0.01$)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes ($\alpha = 0.05$), Yes ($\alpha = 0.01$)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	4375.7 mg/l	Within labs STDev:	268.24 mg/l
StDev of lab means:	775.6 mg/l	Between labs STDev:	720.93 mg/l
Half width 95% CI:	963.1 mg/l		
Upper limit of 95% CI	5338.8 mg/l	Lower limit of 95% CI	3412.6 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s_r		r		s_R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
4400	268.24	6.1	751.07	17.1	769.22	17.5	2154	49

5.2.13. TAR 01, Gravimetric tar

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	CV %
Coordin.	13744	14116	13974	14272	13842	14040	13998	190	1.36
Lab 5	19472	20354	20254	19176	21708	20074	20173	882	4.37
Lab 6	9888	9956	9774	9415	9095	8835	9494	458	4.82
Lab 7	18940								
Lab 16	17020	19160	18850				18343	1156	6.30
Lab 18	27060	36850	37715				33875	5918	17.5

The deviation in results within labs is satisfactory except for Lab 18, but between the labs the deviations are much too large ranging from about 10 to 34 g/l. Therefore, the individual labs were asked to describe the exact evaporation procedure they had used. It was evident that there were differences particularly in the vacuum control. At the Amsterdam meeting on 27 November 2003 it was decided to reject the results for gravimetric tar measurement and arrange a new Round Robin Test for this subject after the correct procedure was emphasized for the participants. Round 03 on gravimetric tar was only carried out from March to April 2004 and the results appear from paragraph 4.4 in this report.

5.2.14. TAR 02, Pyridine

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	61.9	70.0	55.8				62.6	7.12	
Lab 5	NA	NA	NA	NA	NA	NA			
Lab 6	28.1	28.0	27.2	27.1	27.4	27.7			
Lab 7									
Lab 16	0.59	0.63	0.65	0.69	0.69	0.66	0.652	0.038	
Lab 18	/	/	/	/	/	/			

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, only results from two labs remained for analysis. Due to the few numbers it was decided not to carry out further analysis on the fluoranthene results.

5.2.15. TAR 02, Toluene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	574	534	462				523	56.8	141
Lab 5	648	650	649	647	644	645			
Lab 6	553	565	556	565	555	549			
Lab 7	532	540	535	536	543	539	537	3.65	3.83
Lab 16	561	584	587	522	576	563	565	23.8	25.0
Lab 18	640	600	599	601	597	494	589	49.1	51.5
Mean of lab means							554	29.1	46.3

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 02 - Toluene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	553.7 mg/l	Within labs STDev:	23.158 mg/l
StDev of lab means:	29.071 mg/l	Between labs STDev:	35.477 mg/l
Half width 95% CI:	46.259 mg/l		
Upper limit of 95% CI	599.96 mg/l	Lower limit of 95% CI	507.45 mg/l

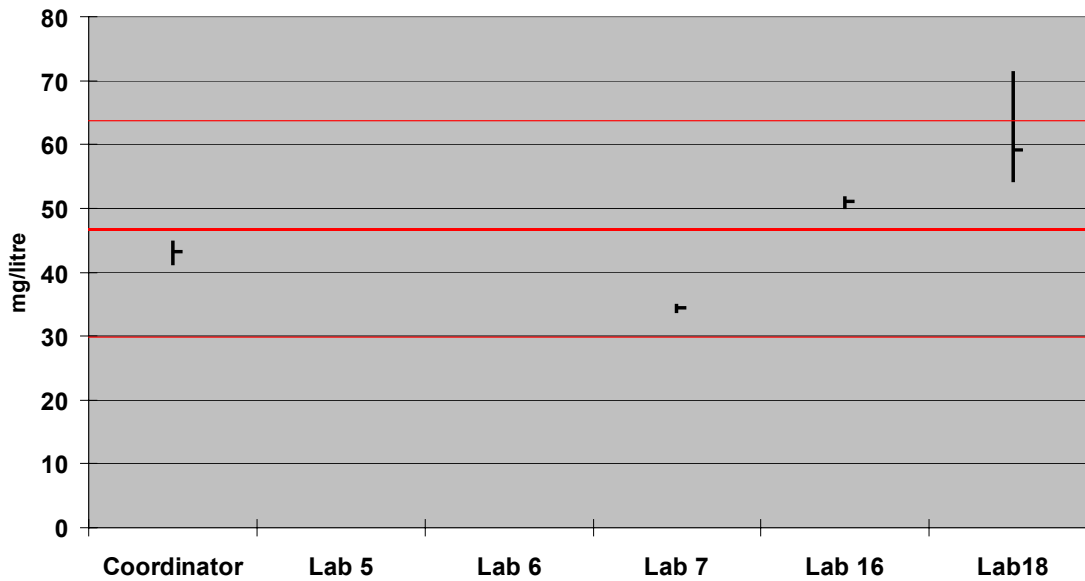
Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
550	23.158	4.2	64.84	11.8	42.37	7.7	118.6	21.6

5.2.16. TAR 02, Phenol

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	43.7	44.6	41.3				43.2	1.71	4.238
Lab 5	83.7	83.8	83.5	83.2	82.7	82.7			
Lab 6	183	188	178	178	191	179			
Lab 7	34.7	34.5	34.0	34.6	34.3	33.9	34.3	0.327	0.343
Lab 16	51.5	50.8	51.6	50.9	51.6	50.2	51.1	0.564	0.594
Lab 18	71.2	54.3	54.4	60.8	54.4	59.9	59.2	6.59	6.916
Mean of lab means							46.95	10.64	16.93

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 02 - Phenol



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Pass # 2: Coordinator is an outlier
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	46.95 mg/l	Within labs STDev:	3.639 mg/l
StDev of lab means:	10.641 mg/l	Between labs STDev:	11.243 mg/l
Half width 95% CI:	16.933 mg/l		
Upper limit of 95% CI	63.883 mg/l	Lower limit of 95% CI	30.017 mg/l

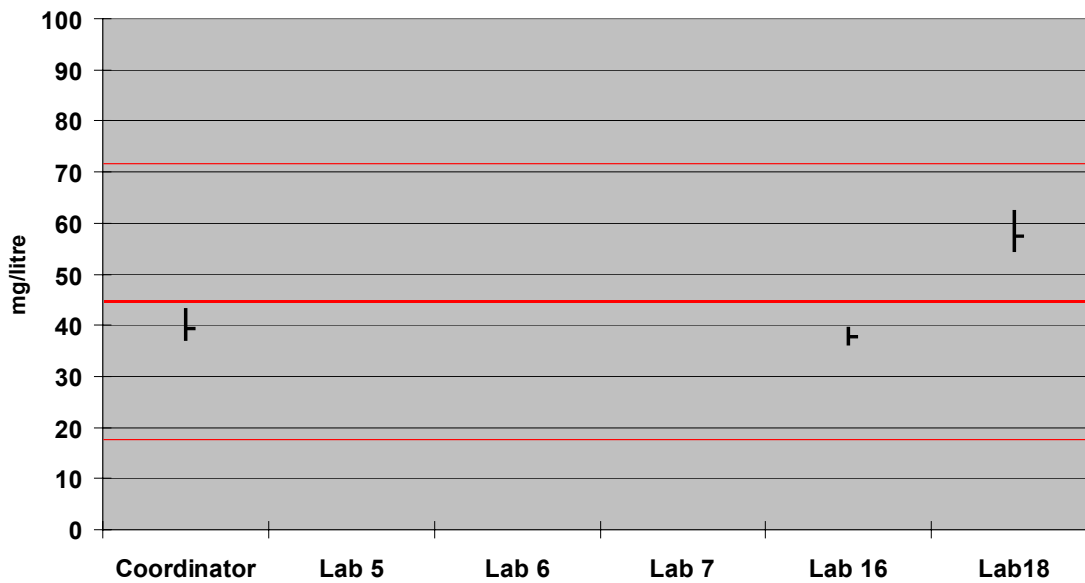
Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
50	3.639	7.3	10.19	20.4	11.82	23.6	33.09	66.2

5.2.17. TAR 02, Indene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	37.4	43.1	37.8				39.4	3.18	7.90
Lab 5	66.8	67.1	67.6	67.3	67.0	67.1			
Lab 6	41.4	41.8	40.6	40.7	41.2	41.3			
Lab 7									
Lab 16	36.4	37.5	39.4	37.9	39.4	36.6	37.9	1.33	1.38
Lab 18	62.2	55.6	57.5	57.7	56.7	54.8	57.4	2.59	2.72
Mean of lab means							44.91	10.86	26.99

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used, as previously mentioned, and as Lab 7 did not report on indene, the statistic analysis is based on the results from the three remaining labs.

TAR 02 - Indene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means: 44.906 mg/l Within labs STDev: 2.282 mg/l
 StDev of lab means: 10.863 mg/l Between labs STDev: 11.627 mg/l
 Half width 95% CI: 26.986 mg/l
 Upper limit of 95% CI 71.891 mg/l Lower limit of 95% CI 17.920 mg/l

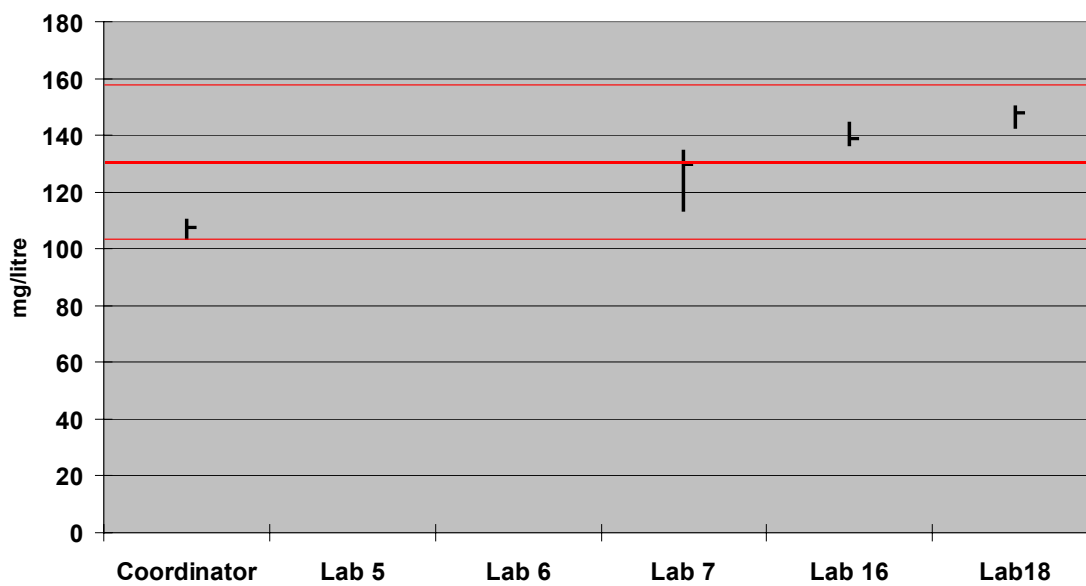
Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
50	2.282	4.6	6.39	12.8	11.85	23.7	33.18	66.4

5.2.18. TAR 02, Guaiacol

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	110	109	104				108	3.21	7.99
Lab 5	252	252	251	293	249	251			
Lab 6	274	263	254	266	265	264			
Lab 7	133	114	133	134	134	132	130	7.97	8.36
Lab 16	137	137	138	144	137	140	139	2.79	2.93
Lab 18	145	149	149	150	150	143	148	2.94	3.09
Mean of lab means							131	17.2	27.3

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used, as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 02 - Guaiacol



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 7 is an outlier
 Bartlett test: Lab variances homogeneous?
 Yes (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means: 130.983 mg/l Within labs STDev: 4.972 mg/l
 StDev of lab means: 17.177 mg/l Between labs STDev: 14.785 mg/l
 Half width 95% CI: 27.332 mg/l
 Upper limit of 95% CI 158.315 mg/l Lower limit of 95% CI 103.652 mg/l

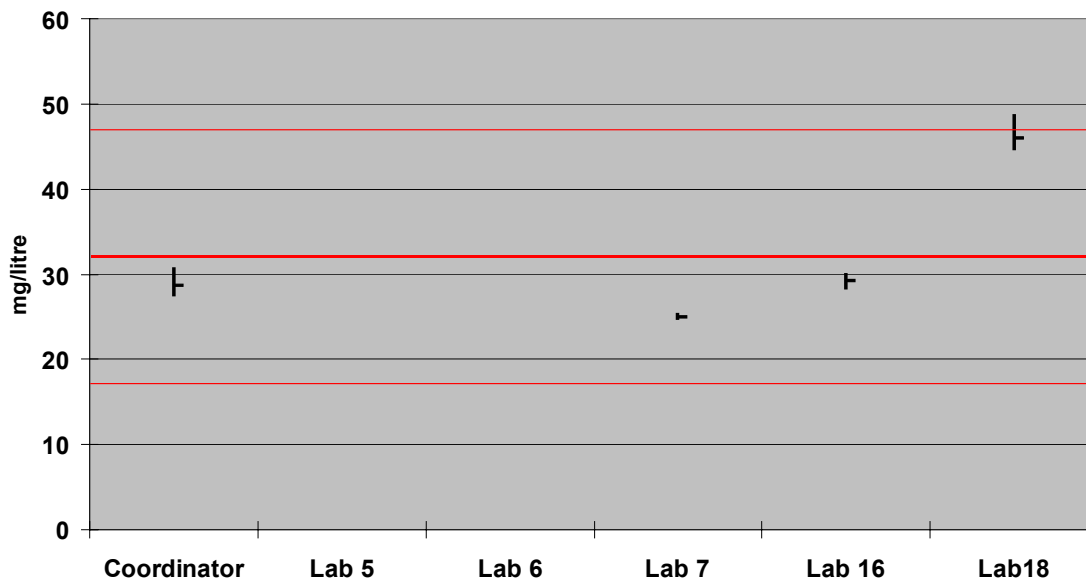
Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
130	4.972	3.8	13.92	10.7	15.6	12.0	43.68	33.6

5.2.19. TAR 02, Naphthalene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	27.6	30.6	27.8				28.7	1.68	4.17
Lab 5	47.9	48.5	48.5	48.1	48.2	48.3			
Lab 6	39.1	39.0	32.6	38.4	32.7	38.3			
Lab 7	25.0	25.0	25.0	25.0	25.2	24.9	25.0	0.098	0.103
Lab 16	28.5	29.0	29.8	29.3	30.0	28.8	29.2	0.582	0.611
Lab 18	48.6	45.5	45.3	46.4	45.3	44.7	46.0	1.47	1.472
Mean of lab means							32.2	9.35	14.9

As Lab 5 withdrew its individual results on updraft tar analysis, and as the results from Lab 6 were not used, as previously mentioned, the statistic analysis is based on the results from the four remaining labs.

TAR 02 - Naphthalene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: Lab 18 is an outlier
 Grubb's test: No outlier detected

Cochran test: Pass # 1: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	32.221 mg/l	Within labs STDev:	1.006 mg/l
StDev of lab means:	9.352 mg/l	Between labs STDev:	9.952 mg/l
Half width 95% CI:	14.882 mg/l		
Upper limit of 95% CI	47.103 mg/l	Lower limit of 95% CI	17.339 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
30	1.006	3.4	2.82	9.4	10.00	33.3	28.01	93.4

5.2.20. TAR 02, Acenaphthylene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.238	0.248	0.223						
Lab 5	1.20	1.35	1.29	1.35	1.25	1.26			
Lab 6									
Lab 7									
Lab 16									
Lab 18	< 1	< 1	< 1	< 1	< 1	< 1			

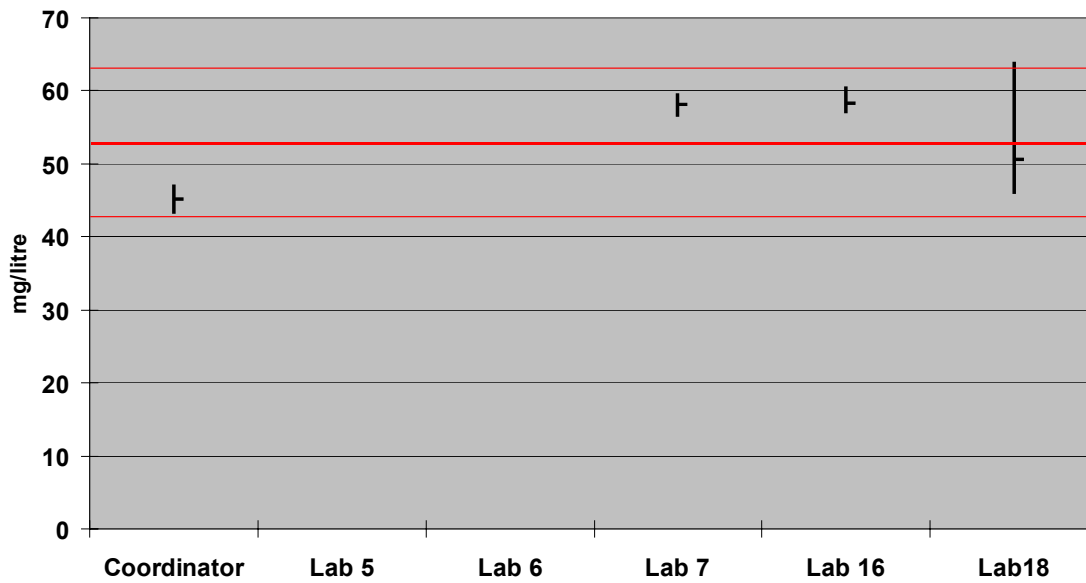
Statistic analysis was not carried out. Only two laboratory reported analysis results. The other laboratories reported values either below detection limit or no information.

5.2.21. TAR 02, 4-methylguaiacol (creosol)

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	47.0	45.1	43.4				45.2	1.80	4.474
Lab 5	106	105	106	106	105	105			
Lab 6									
Lab 7	59.5	58.7	57.5	58.4	58.1	56.7	58.2	0.971	1.019
Lab 16	60.3	57.7	58.3	57.2	58.5	57.7	58.3	1.093	1.147
Lab 18	63.8	46.5	46.1	46.1	46.9	54	50.6	7.17	7.521
Mean of lab means							53.04	6.37	10.14

As Lab 5 withdrew its individual results on updraft tar analysis, and as Lab 6 did not report results on creosol, the statistic analysis is based on the results from the four remaining labs.

TAR 02 - 4-methylguaiacol



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Bartlett test: Lab variances homogeneous?
 Yes (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means: 53.042 mg/l Within labs STDev: 4.014 mg/l
 StDev of lab means: 6.369 mg/l Between labs STDev: 5.515 mg/l
 Half width 95% CI: 10.135 mg/l
 Upper limit of 95% CI 63.177 mg/l Lower limit of 95% CI 42.906 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
50	4.014	8.0	11.24	22.5	6.82	13.6	19.1	38.2

5.2.22. TAR 02, Phenanthrene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.052	0.042	0.035						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 2	< 2	< 2	< 2	< 2	< 2			
Lab 16	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			
Lab 18	< 1	< 1	< 1	< 1	< 1	< 1			

Statistic analysis was not carried out. Only one laboratory reported analysis results.

5.2.23. TAR 02, Fluoranthene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.010	0.008	0.006						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 2	< 2	< 2	< 2	< 2	< 2			
Lab 16	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			
Lab 18	< 1	< 1	< 1	< 1	< 1	< 1			

Statistic analysis was not carried out. Only one laboratory reported analysis results.

5.2.24. TAR 02, Pyrene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.009	0.007	0.006						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 2	< 2	< 2	< 2	< 2	< 2			
Lab 16	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			
Lab 18	10.1	8.63	8.6	8.61	8.57	8.4			

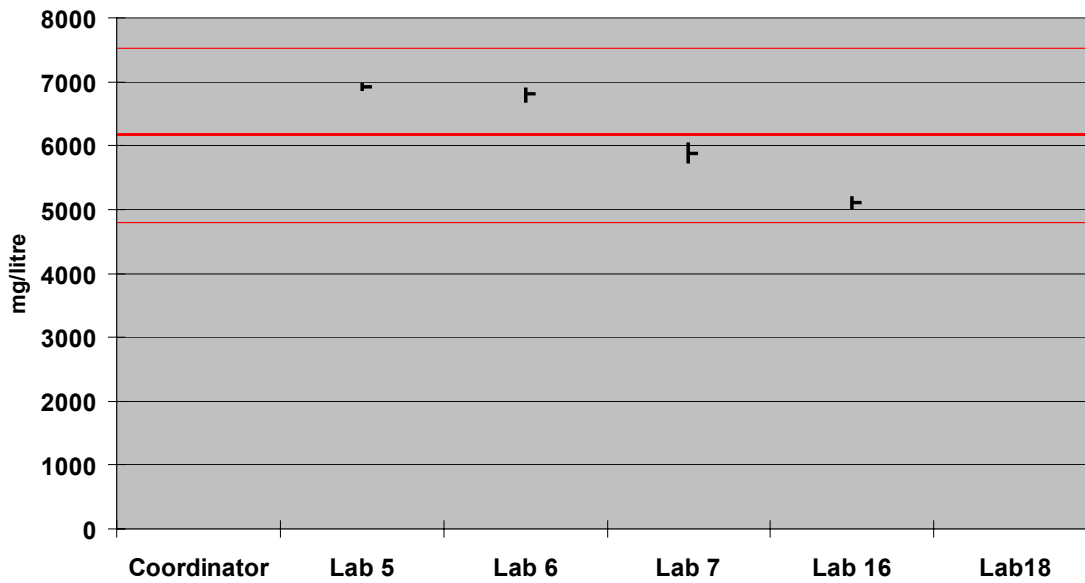
Statistic analysis was not carried out. Only two laboratory reported analysis results.

5.2.25. TAR 02, Total chromatographable tars calculated as naphthalene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	13100	11700	12700	16000	12400	12200			
Lab 5	6912	6957	6946	6915	6882	6896	6918	28.74	30.16
Lab 6	6843	6790	6696	6853	6750	6886	6803	71.30	74.82
Lab 7	5846	5818	5950	6021	5846	5746	5871	98.41	103.28
Lab 16	5174	5034					5104	99.0	889.4
Lab 18	/	/	/	/	/	/			
Mean of lab means							6174	854	1358

The coordinator was recognized as an outlier. Therefore, the results were discarded and a new statistic analysis was carried out on the results of the remaining four labs.

TAR 02 - Total GC tar



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: No outlier detected
 Bartlett test: Lab variances homogeneous?
 Yes (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	6174 mg/l	Within labs STDev:	74.065 mg/l
StDev of lab means:	853.6 mg/l	Between labs STDev:	726.59 mg/l
Half width 95% CI:	1358 mg/l		
Upper limit of 95% CI	7532 mg/l	Lower limit of 95% CI	4816 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
6200	74.065	1.2	207.4	3.3	730.4	11.8	2045	33

5.2.26. TAR 02, Gravimetric tar

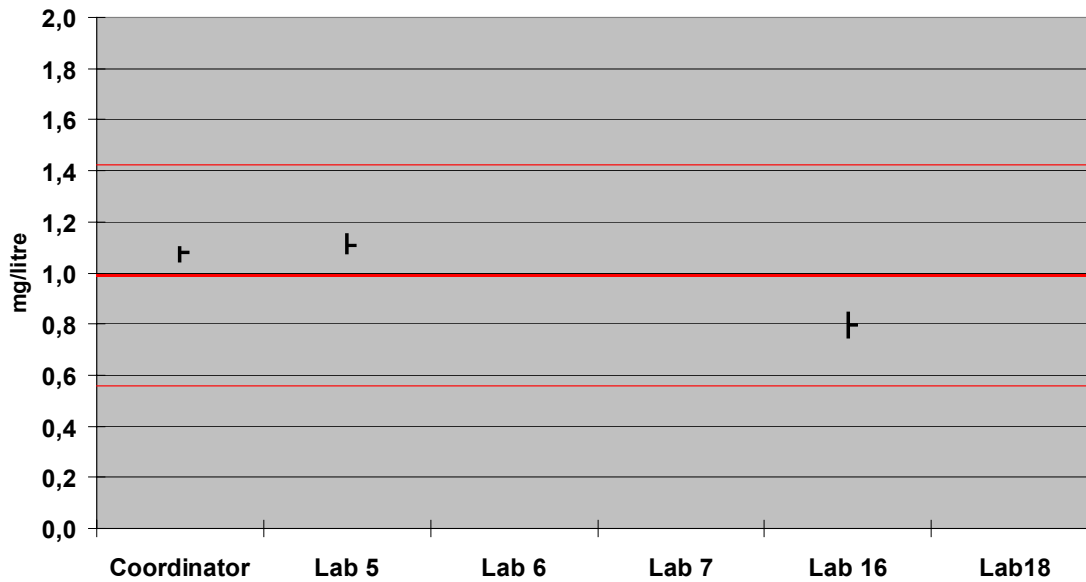
Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	CV %
Coordin.	1252	1288	1682	1388	1564	1475	1442	165	11.5
Lab 5	9694	8004	9186	7568	5896	9628	8329	1476	17.7
Lab 6	852	720	752	960	574	648	751	139	18.5
Lab 7	3330								
Lab 16	8086	4996	5992				6358	1577	24.8
Lab 18	14298	23982	26494				21591	6440	29.8

The deviations in results within labs are not satisfactory and between labs the deviations are much too large ranging from about 0.75 to 22 g/l. Therefore, the individual labs were asked to describe the exact evaporation procedure they had used. It was evident that there were differences particularly in the vacuum control. At the Amsterdam meeting on 27 November 2003 it was decided to reject the results for gravimetric tar measurement and arrange a new Round Robin Test for this subject after the correct procedure was emphasized for the participants. Round 03 on gravimetric tar was only carried out from March to April 2004 and the results appear from paragraph 4.4 in this report.

5.2.27. TAR 03, Pyridine

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	1.10	1.05	1.09				1.080	0.026	0.066
Lab 5	1.11	1.12	1.08	1.12	1.15	1.09	1.112	0.025	0.026
Lab 6									
Lab 7	< 4	< 4	< 4	< 4	< 4	< 4			
Lab 16	0.75	0.80	0.75	0.84	0.80	0.83	0.795	0.038	0.040
Lab 18	<5	<5	<5	<5	<5	<5			
Mean of lab means							0.996	0.174	0.433

TAR 03 - Pyridine



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: No outlier detected
 Bartlett test: Lab variances homogeneous?
 Yes (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	0.996 mg/l	Within labs STDev:	0.031 mg/l
StDev of lab means:	0.174 mg/l	Between labs STDev:	0.187 mg/l
Half width 95% CI:	0.433 mg/l		
Upper limit of 95% CI	1.429 mg/l	Lower limit of 95% CI	0.562 mg/l

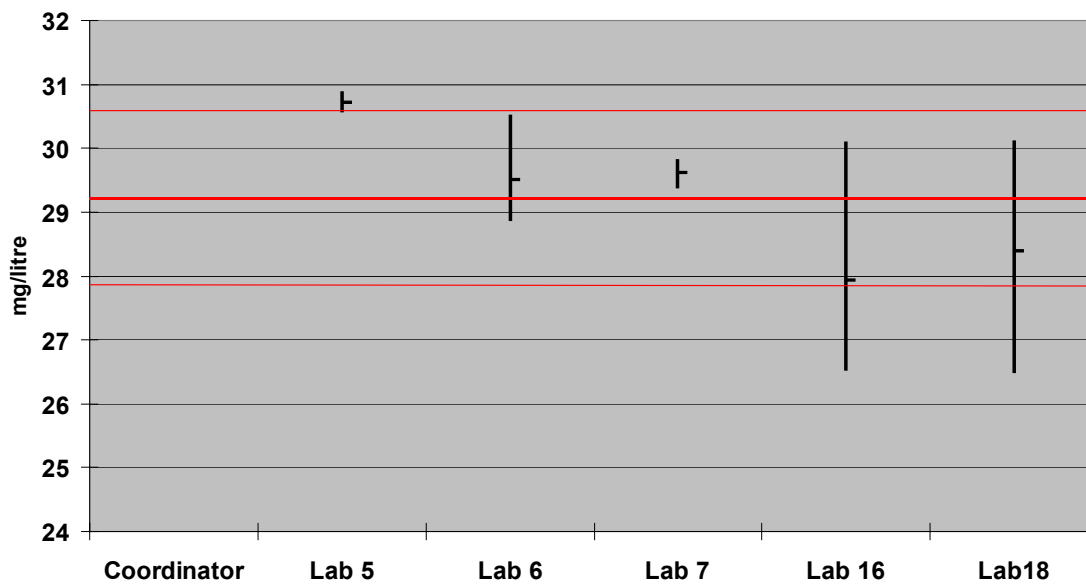
Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
1	0.031	3.1	0.087	8.7	0.190	19	0.531	53.1

5.2.28. TAR 03, Toluene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	41.5	31.3	44.4				39.1		
Lab 5	30.7	30.6	30.9	30.8	30.6	30.8	30.7	0.109	0.113
Lab 6	29.0	30.5	30.4	29.2	29.2	28.9	29.5	0.714	0.749
Lab 7	29.8	29.4	29.4	29.8	29.8	29.5	29.6	0.204	0.214
Lab 16	28.7	30.1	27.1	28.6	26.6	26.5	27.9	1.42	1.503
Lab 18	28.8	30.1	28.3	26.8	29.9	26.5	28.4	1.52	1.590
Mean of lab means							29.2	1.10	1.36

The table above shows the results from all six labs. The first statistic analysis showed that the coordinator was an outlier. The deviation could not be explained which is why the coordinator's results were rejected and a new analysis was carried out based on the results from the five remaining labs.

TAR 03 - Toluene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: No outlier detected
 Bartlett test: Lab variances homogeneous?
 Yes (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means: 29.237 mg/l Within labs STDev: 0.991 mg/l
 StDev of lab means: 1.098 mg/l Between labs STDev: 1.020 mg/l
 Half width 95% CI: 1.363 mg/l
 Upper limit of 95% CI 30.600 mg/l Lower limit of 95% CI 27.874 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
30	0.991	3.3	2.77	9.2	1.42	4.7	3.98	13.3

5.2.29. TAR 03, Phenol

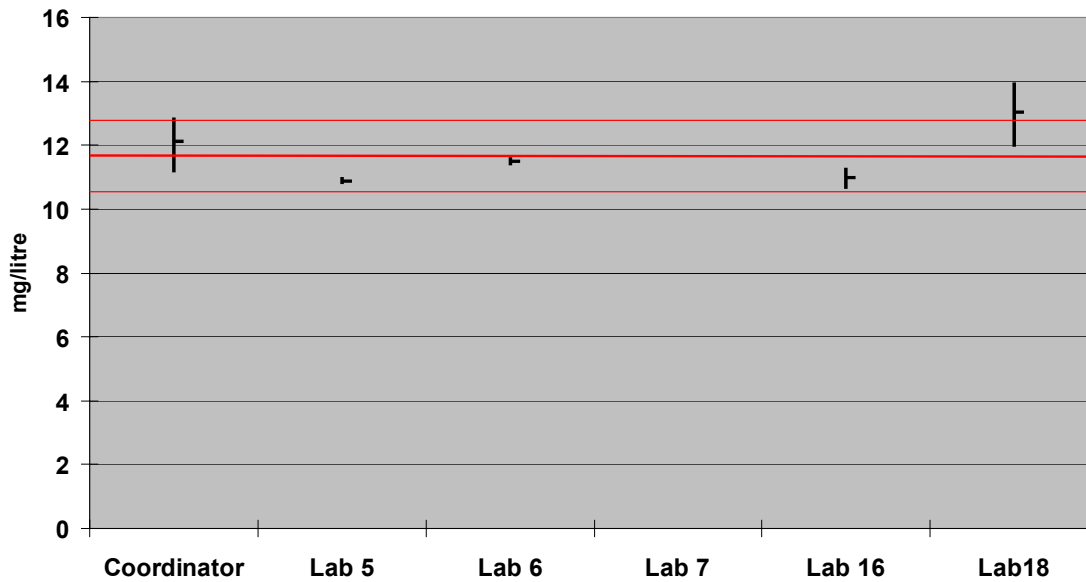
Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.285	0.292	0.288						
Lab 5	0.231	0.231	0.231	0.211	0.231	0.221			
Lab 6									
Lab 7	< 20	< 20	< 20	< 20	< 20	< 20			
Lab 16	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00			
Lab 18	<1	<1	<1	<1	<1	<1			

Statistic analysis was not carried out. Only two laboratories reported analysis results.

5.2.30. TAR 03, Indene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	12.8	11.2	12.4				12.1	0.83	2.068
Lab 5	10.8	10.8	10.9	10.9	10.8	10.9	10.9	0.048	0.050
Lab 6	11.5	11.5	11.5	11.6	11.4	11.5	11.5	0.049	0.051
Lab 7									
Lab 16	11.1	11.2	11.0	11.0	10.8	10.7	11.0	0.186	0.195
Lab 18	12.1	13.7	12.7	13.9	13.8	12	13.0	0.876	0.919
Mean of lab means							11.7	0.896	1.11

TAR 03 - Indene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Pass # 2: Coordinator is an outlier
 Pass # 3: Lab 16 is an outlier
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means: 11.701 mg/l Within labs STDev: 0.496 mg/l
 StDev of lab means: 0.896 mg/l Between labs STDev: 0.910 mg/l
 Half width 95% CI: 1.113 mg/l
 Upper limit of 95% CI 12.814 mg/l Lower limit of 95% CI 10.588 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
10	0.496	5	1.39	13.9	1.04	10.4	2.9	29

5.2.31. TAR 03, Guaiacol

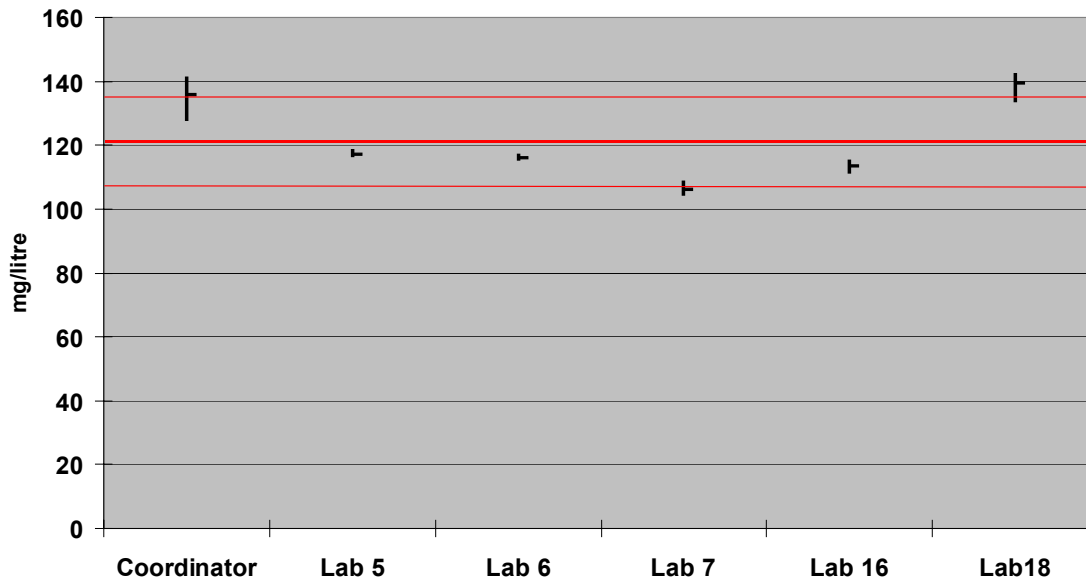
Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	<0.05	<0.05	<0.05						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 5	< 5	< 5	< 5	< 5	< 5			
Lab 16	< 2.5	< 2.5	< 2.5	< 2.5	< 2.5	< 2.5			
Lab 18									

Statistic analysis was not carried out. Laboratories reported values either below detection limit or no information (blank cells in the report).

5.2.32. TAR 03, Naphthalene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	141	128	138				136	6.81	16.91
Lab 5	117	117	118	117	117	118	117	0.503	0.527
Lab 6	116	116	116	117	116	116	116	0.375	0.394
Lab 7	108	106	105	107	105	105	106	1.49	1.565
Lab 16	114	115	115	115	112	112	114	1.47	1.545
Lab 18	139	142	141	141	141	134	140	2.94	3.089
Mean of lab means							121.4	13.3	13.9

TAR 03 - Naphthalene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Coordinator is an outlier
 Pass # 2: Lab 18 is an outlier
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

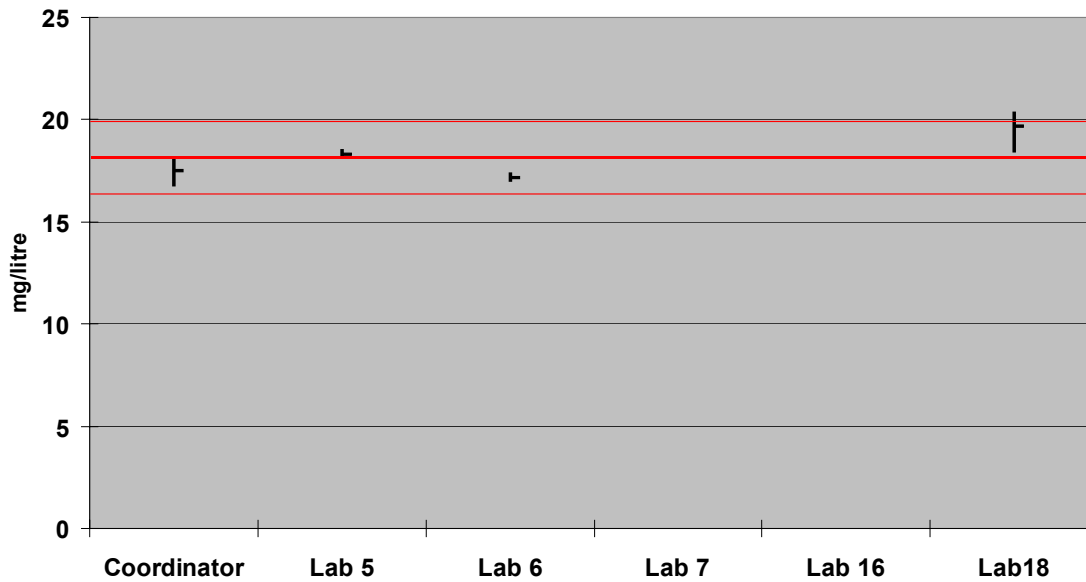
Mean of lab means: 121.396 mg/l Within labs STDev: 2.434 mg/l
 StDev of lab means: 13.256 mg/l Between labs STDev: 12.952 mg/l
 Half width 95% CI: 13.911 mg/l
 Upper limit of 95% CI 135.306 mg/l Lower limit of 95% CI 107.485 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
120	2.434	2	6.815	5.7	13.179	11	36.9	30.8

5.2.33. TAR 03, Acenaphthylene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	18.1	16.8	17.7				17.5	0.67	1.654
Lab 5	18.2	18.2	18.5	18.3	18.2	18.3	18.3	0.104	0.111
Lab 6	17.1	17.2	17.3	17.2	17.0	17.2	17.2	0.088	0.092
Lab 7									
Lab 16									
Lab 18	20.3	20.2	19.1	20.2	19.9	18.5	19.70	0.735	0.771
Mean of lab means							18.17	1.122	1.786

TAR 03 - Acenaphthylene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means: 18.172 mg/l Within labs STDev: 0.465 mg/l
 StDev of lab means: 1.122 mg/l Between labs STDev: 1.156 mg/l
 Half width 95% CI: 1.786 mg/l
 Upper limit of 95% CI 19.957 mg/l Lower limit of 95% CI 16.386 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
20	0.465	2.3	1.30	6.5	1.25	6.3	3.49	17.4

5.2.34. TAR 03, 4-methylguaiacol (creosol)

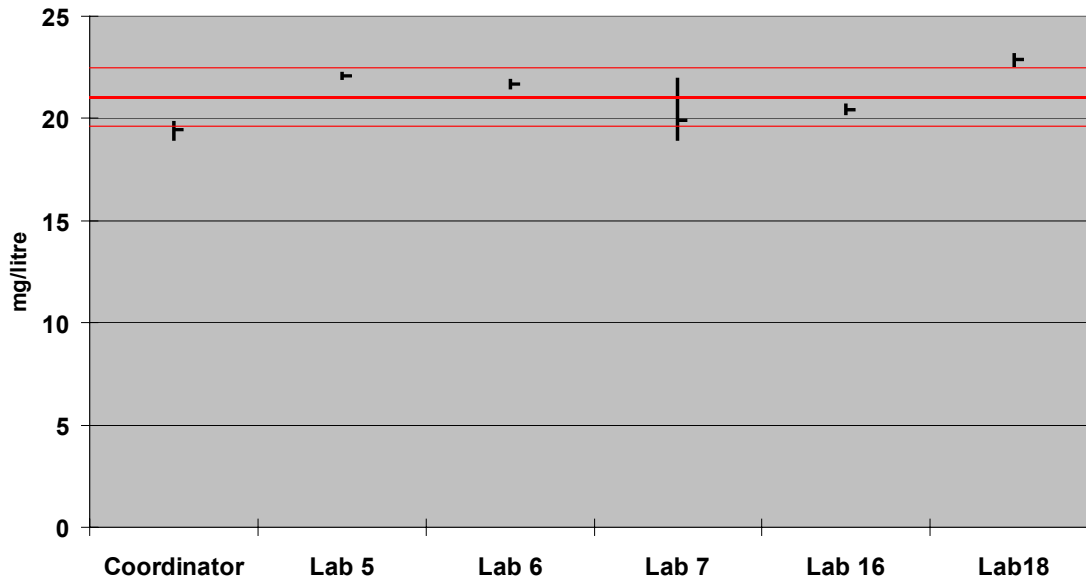
Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	<0.05	<0.05	<0.05						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 5	< 5	< 5	< 5	< 5	< 5			
Lab 16	3.15	3.18	3.16	3.19	3.16	3.13			
Lab 18									

Statistic analysis was not carried out. Only one laboratory reported analysis results.

5.2.35. TAR 03, Phenanthrene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	19.6	19	19.8				19.5	0.416	1.034
Lab 5	21.9	22.0	22.2	22.0	22.0	22.1	22.1	0.082	0.087
Lab 6	21.8	21.7	21.8	21.5	21.5	21.8	21.7	0.142	0.149
Lab 7	21.9	19.8	19.9	19.5	19.3	19.0	19.9	1.033	1.085
Lab 16	20.3	20.4	20.4	20.5	20.5	20.6	20.4	0.105	0.110
Lab 18	22.8	23.1	22.6	22.8	23.1	23	22.9	0.200	0.210
Mean of lab means							21.08	1.364	1.412

TAR 03 - Phenanthrene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 7 is an outlier
 Pass # 2: Coordinator is an outlier
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

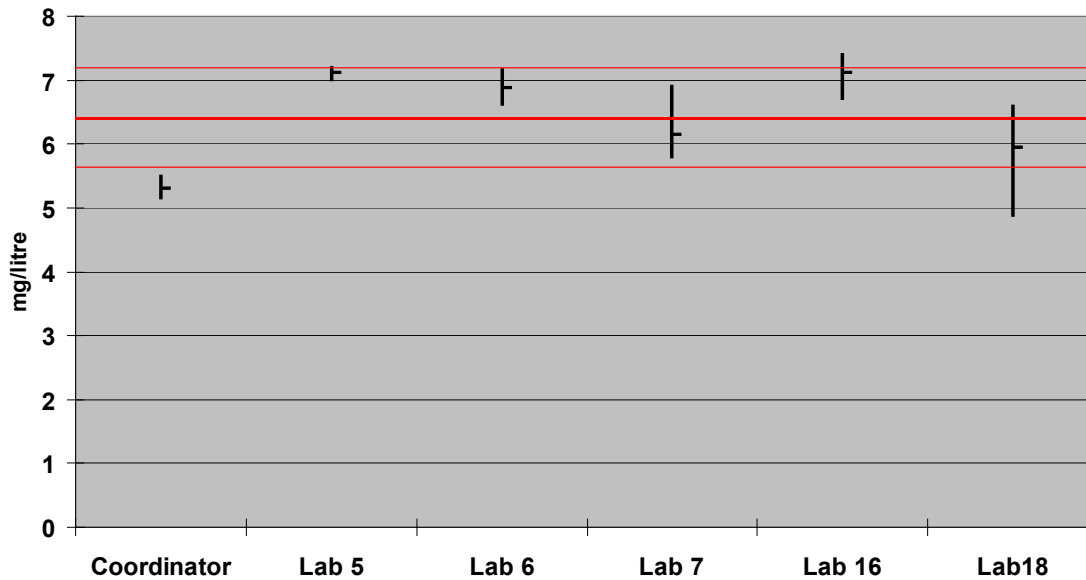
Mean of lab means:	21.08 mg/l	Within labs STDev:	0.474 mg/l
StDev of lab means:	1.364 mg/l	Between labs STDev:	1.280 mg/l
Half width 95% CI:	1.412 mg/l		
Upper limit of 95% CI	22.493 mg/l	Lower limit of 95% CI	19.668 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
20	0.474	2.4	1.327	6.6	1.365	6.8	3.821	19.1

5.2.36. TAR 03, Fluoranthene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	5.28	5.49	5.16				5.31	0.17	0.415
Lab 5	7.02	7.15	7.20	7.14	7.12	7.08	7.12	0.061	0.065
Lab 6	6.93	6.97	6.92	6.68	6.62	7.16	6.88	0.199	0.209
Lab 7	6.9	6.2	6.0	6.0	6.0	5.8	6.15	0.389	0-408
Lab 16	6.72	7.11	7.11	7.08	7.35	7.39	7.13	0.240	0.252
Lab 18	4.89	5.92	5.99	6.59	6.24	6.1	5.955	0.573	0.601
Mean of lab means							6.423	0.738	0.774

TAR 03 - Fluoranthene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Pass # 2: Lab 7 is an outlier
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

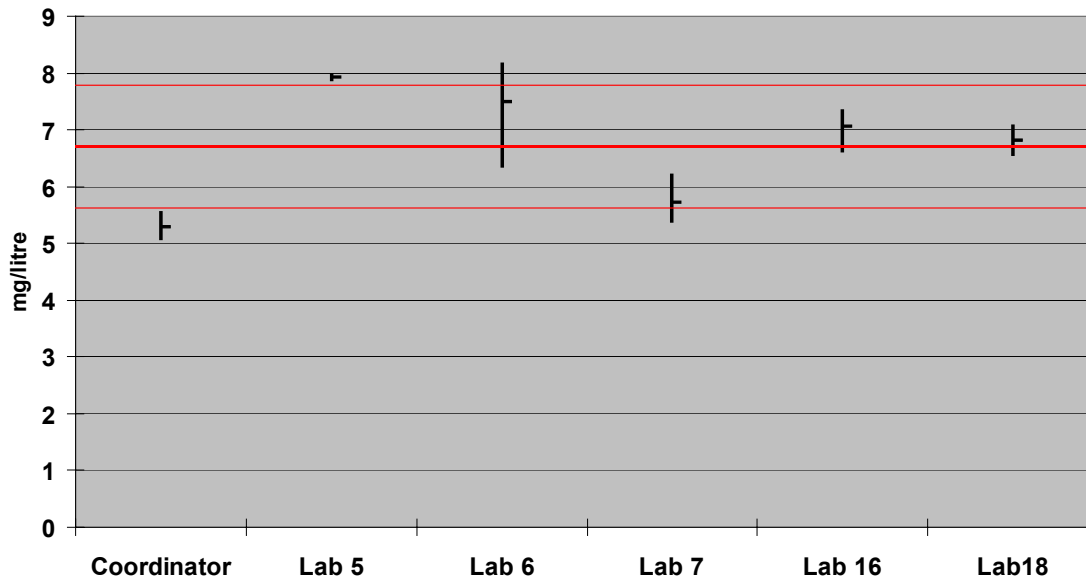
Mean of lab means:	6.423 mg/l	Within labs STDev:	0.331 mg/l
StDev of lab means:	0.738 mg/l	Between labs STDev:	0.655 mg/l
Half width 95% CI:	0.774 mg/l		
Upper limit of 95% CI	7.197 mg/l	Lower limit of 95% CI	5.649 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
6	0.331	5.5	0.927	15.5	0.734	12.2	2.055	34.2

5.2.37. TAR 03, Pyrene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	5.26	5.54	5.09				5.30	0.23	0.564
Lab 5	7.90	7.92	7.97	7.93	7.90	7.93	7.92	0.027	0.027
Lab 6	8.15	7.63	7.72	7.63	7.48	6.36	7.50	0.601	0.630
Lab 7	6.2	5.6	5.4	5.8	5.7	5.6	5.72	0.271	0.285
Lab 16	6.64	6.91	7.02	7.34	7.25	7.28	7.07	0.269	0.283
Lab 18	6.92	6.98	6.68	7.07	6.72	6.56	6.822	0.198	0.207
Mean of lab means							6.721	1.022	1.072

TAR 03 - Pyrene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 6 is an outlier
 Bartlett test: Lab variances homogeneous?
 Yes (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	6.721 mg/l	Within labs STDev:	0.324 mg/l
StDev of lab means:	1.022 mg/l	Between labs STDev:	0.941 mg/l
Half width 95% CI:	1.072 mg/l		
Upper limit of 95% CI	7.794 mg/l	Lower limit of 95% CI	5.649 mg/l

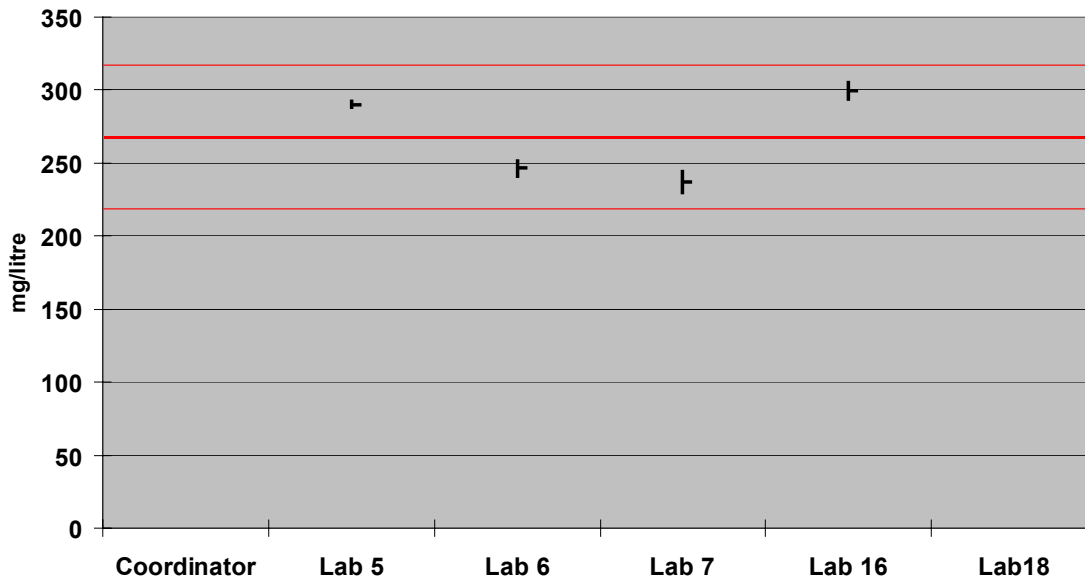
Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
6	0.324	5.4	0.907	15.1	0.995	16.6	2.787	46.4

5.2.38. TAR 03, Total chromatographable tars calculated as naphthalene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	193	171	237	205	243	258			
Lab 5	289	289	292	290	289	290	290	1.32	1.386
Lab 6	243	250	251	250	241	245	247	4.22	4.426
Lab 7	230	236	233	239	244	242	237	5.35	5.619
Lab 16	305	294					300	7.78	69.88
Lab 18									
Mean of lab means							268	30.86	49.1

The table above shows the results from all six labs. The first statistic analysis showed that the coordinator was an outlier. The deviation could not be explained which is why the coordinator's results were rejected and a new analysis was carried out based on the results from the four remaining labs.

TAR 03 - Total GC tar



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: No outlier detected
 Bartlett test: Lab variances homogeneous?
 Yes (a = 0.05)
 Yes (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means: 268.4 mg/l Within labs STDev: 4.341 mg/l
 StDev of lab means: 30.856 mg/l Between labs STDev: 29.392 mg/l
 Half width 95% CI: 49.099 mg/l
 Upper limit of 95% CI 317.5 mg/l Lower limit of 95% CI 219.3 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
300	4.341	1.5	12.155	4.1	29.711	9.9	83.19	27.7

5.2.39. TAR 03, Gravimetric tar

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	CV %
Coordin.	102	68	78	88	76	92	84.0	12.3	14.7
Lab 5	154	122	134	102	156	142	135	20.5	15.2
Lab 6	90	86	48	64	110	45	73.8	25.7	34.9
Lab 7	180								
Lab 16	226	161	89				159	68.4	43.1
Lab 18	10612	8334	11826				10257	1773	17.3

The deviations in results within labs are not satisfactory and between labs the deviations are much too large ranging from about 0.075 to 10 g/l. Therefore, the individual labs were asked to describe the exact evaporation procedure they had used. It was evident that there were differences particularly in the vacuum control. At the Amsterdam meeting on 27 November 2003 it was decided to reject the results for gravimetric tar measurement and arrange a new Round Robin Test for this subject after the correct procedure was emphasized for the participants. Round 03 on gravimetric tar was only carried out from March to April 2004 and the results appear from paragraph 4.4 in this report.

5.2.40. TAR 04, Pyridine

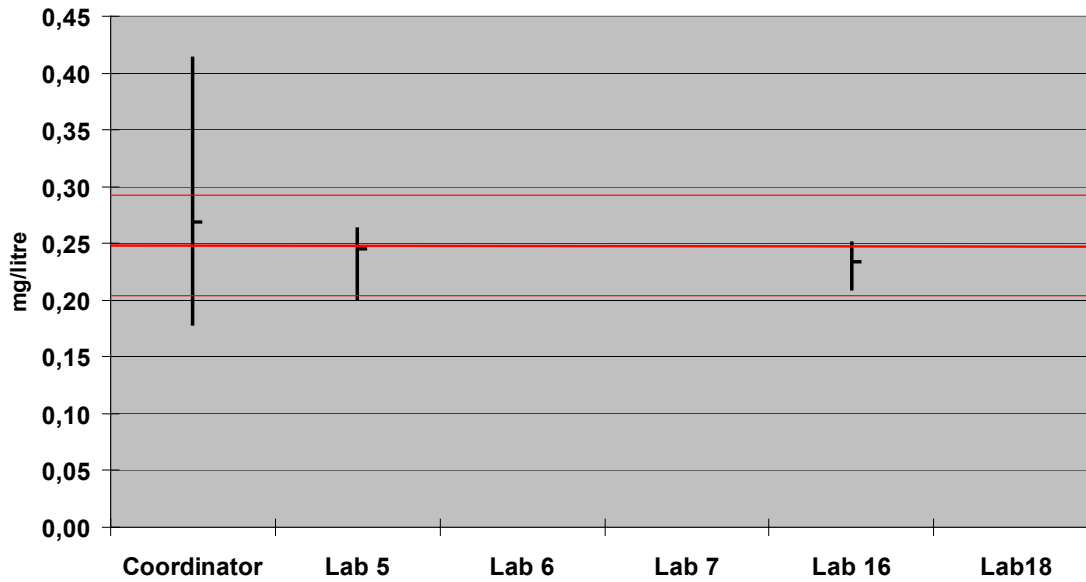
Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	<0.1	<0.1	<0.1						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 4	< 4	< 4	< 4	< 4	< 4			
Lab 16	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 18	<5	<5	<5	<5	<5	<5			

Statistic analysis was not carried out. Laboratories reported values either below detection limit or no information.

5.2.41. TAR 04, Toluene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.179	0.213	0.413				0.268	0.126	0.314
Lab 5	0.201	0.255	0.232	0.263	0.255	0.263	0.245	0.024	0.026
Lab 6									
Lab 7	< 2	< 2	< 2	< 2	< 2	< 2			
Lab 16	0.23	0.24	0.25	0.23	0.24	0.21	0.233	0.014	0.230
Lab 18	<1	<1	<1	<1	<1	<1			
Mean of lab means							0.249	0.018	0.044

TAR 04 - Toluene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Coord. is an outlier
 Bartlett test: Lab variances homogeneous?
 No ($\alpha = 0.05$)
 No ($\alpha = 0.01$)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 No ($\alpha = 0.05$), No ($\alpha = 0.01$)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	0.249 mg/l	Within labs STDev:	0.055 mg/l
StDev of lab means:	0.018 mg/l	Between labs STDev:	MSB < MSW mg/l
Half width 95% CI:	0.044 mg/l		
Upper limit of 95% CI	0.293 mg/l	Lower limit of 95% CI	0.205 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s_r		r		s_R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
0.25	0.055	22	0.154	61.6	-	-	-	-

5.2.42. TAR 04, Phenol

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	<0.05	<0.05	<0.05						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 20	< 20	< 20	< 20	< 20	< 20			
Lab 16	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00			
Lab 18	<1	<1	<1	<1	<1	<1			

Statistic analysis was not carried out. Laboratories reported values either below detection limit or no information.

5.2.43. TAR 04, Indene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.045	0.051	0.073						
Lab 5	0.050	0.058	0.050	0.043	0.050	0.036			
Lab 6									
Lab 7									
Lab 16	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 18	<1	<1	<1	<1	<1	<1			

Statistic analysis was not carried out. Only two laboratories reported analysis results. The other laboratories reported values either below detection limit or no information.

5.2.44. TAR 04, Guaiacol

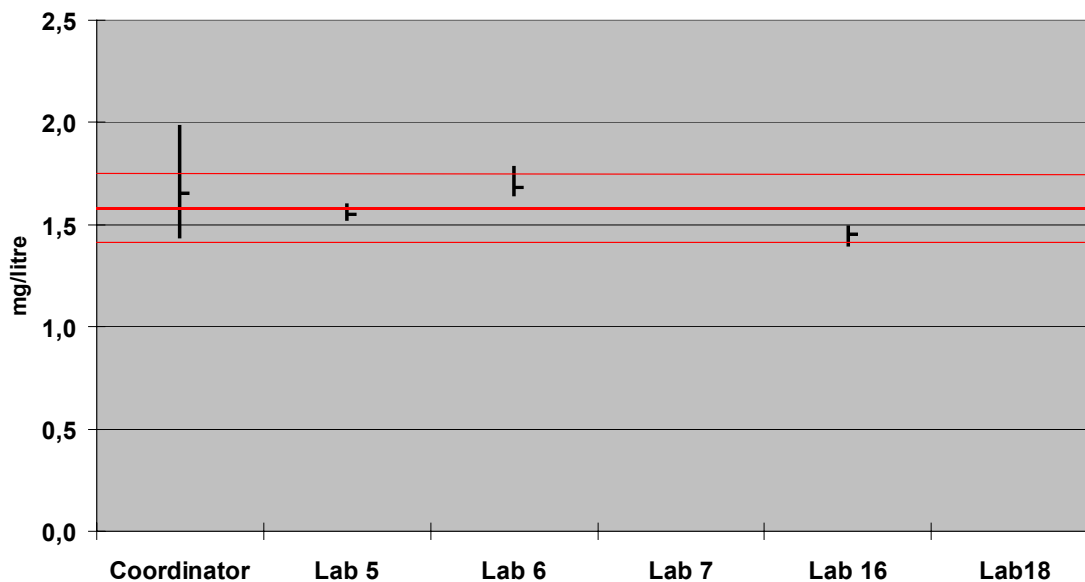
Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	<0.05	<0.05	<0.05						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 5	< 5	< 5	< 5	< 5	< 5			
Lab 16	< 2.50	< 2.50	< 2.50	< 2.50	< 2.50	< 2.50			
Lab 18									

Statistic analysis was not carried out. Laboratories reported values either below detection limit or no information.

5.2.45. TAR 04, Naphthalene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	1.44	1.54	1.98				1.653	0.287	0.714
Lab 5	1.60	1.56	1.55	1.55	1.53	1.53	1.553	0.026	0.027
Lab 6	1.68	1.65	1.78	1.66	1.66	1.67	1.683	0.048	0.051
Lab 7									
Lab 16	1.45	1.45	1.48	1.40	1.49	1.44	1.452	0.032	0.033
Lab 18	<1	<1	<1	<1	<1	<1			
Mean of lab means							1.585	0.105	0.167

TAR 04 - Naphthalene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Coord. is an outlier
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	1.585 mg/l	Within labs STDev:	0.104 mg/l
StDev of lab means:	0.105 mg/l	Between labs STDev:	0.099 mg/l
Half width 95% CI:	0.167 mg/l		
Upper limit of 95% CI	1.753 mg/l	Lower limit of 95% CI	1.418 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
1.6	0.104	6.5	0.291	18.2	0.144	9	0.402	25.1

5.2.46. TAR 04, Acenaphthylene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.079	0.083	0.098						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7									
Lab 16									
Lab 18	<1	<1	<1	<1	<1	<1			

Statistic analysis was not carried out. Only one laboratory reported analysis results. The other laboratories reported values either below detection limit or no information.

5.2.47. TAR 04, 4-methylguaiacol (creosol)

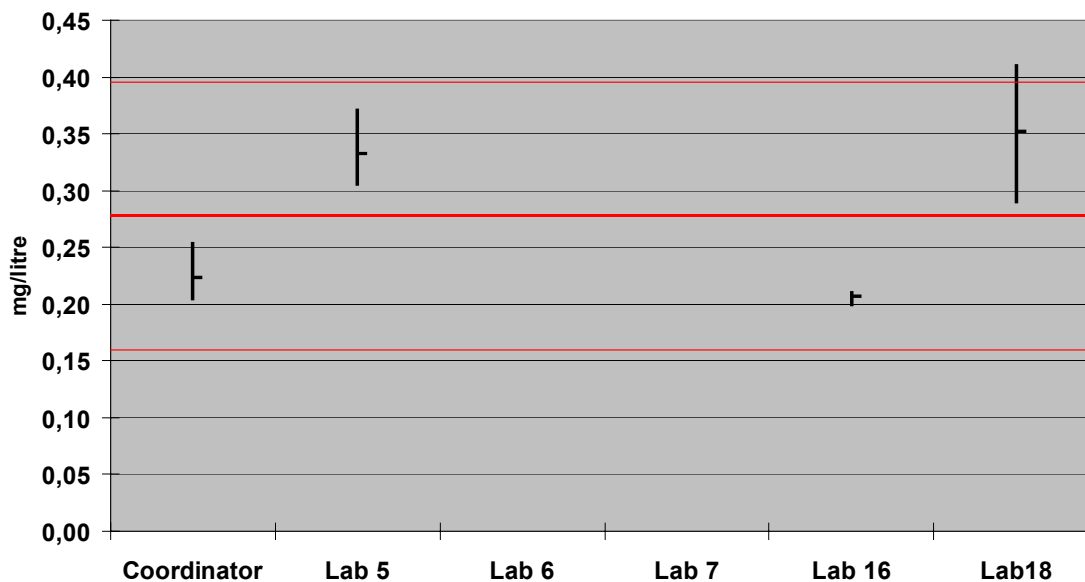
Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	<0.05	<0.05	<0.05						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 8	< 8	< 8	< 8	< 8	< 8			
Lab 16	< 2.50	< 2.50	< 2.50	< 2.50	< 2.50	< 2.50			
Lab 18									

Statistic analysis was not carried out. Laboratories reported values either below detection limit or no information.

5.2.48. TAR 04, Phenanthrene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.205	0.213	0.253				0.224	0.026	0.064
Lab 5	0.335	0.349	0.371	0.306	0.321	0.314	0.333	0.024	0.025
Lab 6									
Lab 7	< 5	< 5	< 5	< 5	< 5	< 5			
Lab 16	0.21	0.20	0.21	0.21	0.21	0.20	0.207	0.005	0.005
Lab 18	0.41	0.39	0.32	0.29	0.32	0.38	0.352	0.048	0.050
Mean of lab means							0.279	0.074	0.118

TAR 04 - Phenanthrene



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test: Pass # 1: Lab 18 is an outlier
 Bartlett test: Lab variances homogeneous?
 No (a = 0.05)
 No (a = 0.01)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes (a = 0.05), Yes (a = 0.01)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means: 0.279 mg/l Within labs STDev: 0.031 mg/l
 StDev of lab means: 0.074 mg/l Between labs STDev: 0.074 mg/l
 Half width 95% CI: 0.118 mg/l
 Upper limit of 95% CI 0.397 mg/l Lower limit of 95% CI 0.161 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
0.28	0.031	11.1	0.087	31.1	0.080	28.6	0.225	80.2

5.2.49. TAR 04, Fluoranthene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.046	0.049	0.069						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 2	< 2	< 2	< 2	< 2	< 2			
Lab 16	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			
Lab 18	<1	<1	<1	<1	<1	<1			

Statistic analysis was not carried out. Only one laboratory reported analysis results. The other laboratories reported values either below detection limit or no information.

5.2.50. TAR 04, Pyrene

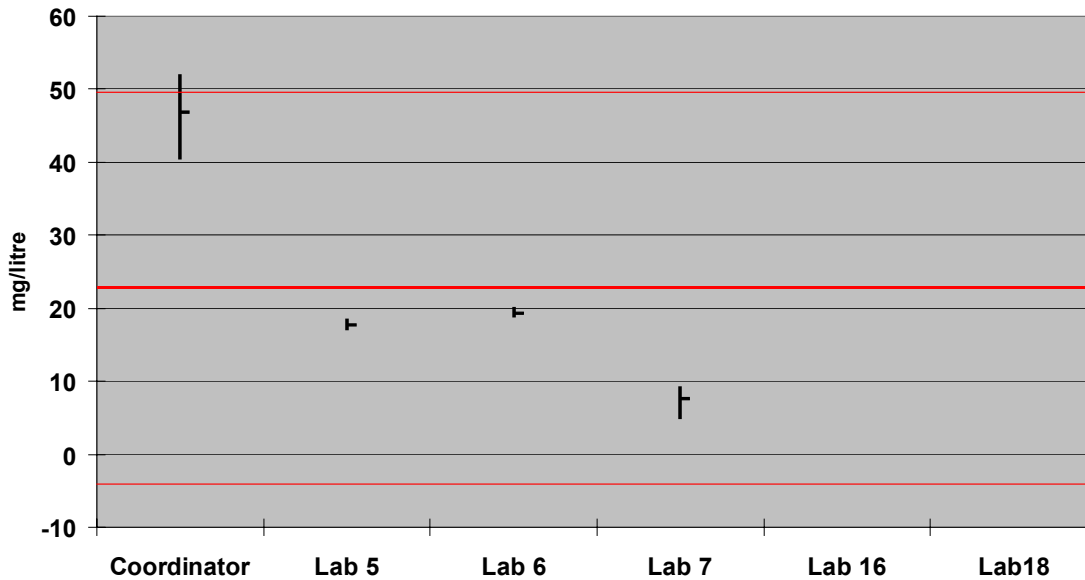
Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	0.047	0.049	0.067						
Lab 5	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25			
Lab 6									
Lab 7	< 2	< 2	< 2	< 2	< 2	< 2			
Lab 16	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20			
Lab 18	<1	<1	<1	<1	<1	<1			

Statistic analysis was not carried out. Only one laboratory reported analysis results. The other laboratories reported values either below detection limit or no information.

5.2.51. TAR 04, Total chromatographable tars calculated as naphthalene

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	H.W. CI (95%) mg/l
Coordin.	45.5	48.3	43.9	51.8	51.3	40.6	46.9	4.381	4.598
Lab 5	18.3	18.1	17.9	17.5	17.2	17.2	17.7	0.469	0.492
Lab 6	19.1	19.4	20.0	19.0	19.2	19.6	19.4	0.371	0.389
Lab 7	5	8	8	8	8	9	7.667	1.366	1.434
Lab 16	< 5	< 5							
Lab 18									
Mean of lab means							22.913	16.807	26.744

TAR 04 - Total GC tar



TESTING OF OUTLYING LAB MEANS

Dixon's test: No outlier detected
 Nalimov t-test: Coord. is an outlier at $\alpha = 0.05$
 Grubb's test: No outlier detected

TESTING OF VARIANCES

Cochran test:
 Pass # 1: Coord. is an outlier at $\alpha = 0.01$ and 0.05
 Pass # 2: Lab 7 is an outlier at $\alpha = 0.01$ and 0.05
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 Yes ($\alpha = 0.05$)
 Yes ($\alpha = 0.01$)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes ($\alpha = 0.05$), Yes ($\alpha = 0.01$)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	22.913mg/l	Within labs STDev:	2.314 mg/l
StDev of lab means:	16.807 mg/l	Between labs STDev:	16.781 mg/l
Half width 95% CI:	26.744 mg/l		
Upper limit of 95% CI	49.657 mg/l	Lower limit of 95% CI	-3.832 mg/l

Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	mg/l	%	mg/l	%	mg/l	%	mg/l	%
23	2.314	10.1	6.479	28.2	16.94	73.7	47.43	206

5.2.52. TAR 04, Gravimetric tar

Lab	Sample # 1 mg/l	Sample # 2 mg/l	Sample # 3 mg/l	Sample # 4 mg/l	Sample # 5 mg/l	Sample # 6 mg/l	Mean mg/l	STDev mg/l	CV %
Coordin.	30	26	24	34	26	38			
Lab 5	10.0	-2.0	4.0	14.0	26.0	13.6			
Lab 6	0	0	0	0					
Lab 7	-90								
Lab 16	< 50	<50	<50						
Lab 18	10586	10428	5786						

The deviations in results within labs are not satisfactory and between labs the deviations are much too large ranging from about -0.09 to 10 g/l. Therefore, the individual labs were asked to describe the exact evaporation procedure they had used. It was evident that there were differences particularly in the vacuum control. At the Amsterdam meeting on 27 November 2003 it was decided to reject the results for gravimetric tar measurement and arrange a new Round Robin Test for this subject after the correct procedure was emphasized for the participants. Round 03 on gravimetric tar was only carried out from March to April 2004 and the results appear from paragraph 4.4 in this report.

5.2.53. TAR 05, Blind sample

Laboratories reported mainly values either below detection limit or no information (blank cells in the report). However, some small quantities of compounds were found.

Lab mean [mg/l]	Coordinator	Lab 5	Lab 16
Toluene		0.234	0.260
Guaiacol	0.052		
4-methylguaiacol	0.074		

5.3. GC-analysis precision

The tables below shows the repeatability and reproducibility values for typical test results (levels). The repeatability appears both as a standard deviation and as the value "r" which is explained in paragraph 5.2. The reproducibility again is reported as a standard deviation and as the value "R", see also paragraph 5.2. All four values are also expressed as percentage of the measured concentration. The concentrations are divided into ranges (levels) and the average values for those ranges appear from the table.

Compound	Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
		s _r		r		s _R		R	
		mg/l	%	mg/l	%	mg/l	%	mg/l	%
Updraft tars, range < 1 mg/l (TAR 01 and TAR 02)									
Pyrene	0.5	0.008	1.9	0.022	5.2	0.03	7.1	0.084	19.8
Mean value	0.5	0.008	1.9	0.022	5.2	0.03	7.1	0.084	19.8
Updraft tars, range 1 - 10 mg/l (TAR 01 and TAR 02)									
Naphthalene	7	0.79	10.9	2.20	30.6	1.74	24.3	4.88	67.9
Phenanthrene	2	0.11	5.9	0.29	16.5	0.60	33.5	1.68	93.8
Mean value	4	0.45	8.4	1.25	23.5	1.17	28.9	3.28	80.9
Updraft tars, range > 10 mg/l (TAR 01 and TAR 02)									
Toluene	550	23.16	4.2	64.84	11.7	42.37	7.7	118.6	21.4
Guaiacol	290	8.30	2.9	23.23	8.1	19.21	6.7	53.8	18.7
Creosol	280	13.37	4.8	37.44	13.4	94.06	33.6	263.4	94.2
Guaiacol	130	4.97	3.8	13.92	10.6	15.60	11.9	43.7	33.3
Phenol	110	6.03	5.4	16.89	15.0	7.76	6.9	21.7	19.3
Toluene	50	2.10	4.4	5.87	12.4	16.53	34.9	46.3	97.7
Phenol	50	3.64	7.8	10.19	21.7	11.82	25.2	33.1	70.5
Indene	50	2.28	5.1	6.39	14.2	11.85	26.4	33.2	73.9
Creosol	50	4.01	7.6	11.24	21.2	6.82	12.9	19.1	3.0
Naphthalene	30	1.01	3.1	2.82	8.8	10.00	31.0	28.0	86.9
Mean value	160	6.89	4.9	19.28	13.7	23.60	19.7	66.1	55.2

For updraft tars applies:

Repeatability value "r"

For single compounds in the range < 1 mg/l the difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value $r = 0.02$ mg/l corresponding to 5.2% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

For single compounds in the range from 1 to 10 mg/l the difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value $r = 1.25$ mg/l corresponding to 23.5% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

For single compounds in the range from 10 to 600 mg/l the difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value $r = 19.3$ mg/l corresponding to 13.7% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

Reproducibility value "R"

For single compounds in the range < 1 mg/l single results on identical test material reported by two laboratories will differ by more than the reproducibility value $R = 0.08$ mg/l corresponding to 19.8% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

For single compounds in the range from 1 to 10 mg/l single results on identical test material reported by two laboratories will differ by more than the reproducibility value $R = 3.3$ mg/l corresponding to 80.9% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

For single compounds in the range from 10 to 600 mg/l single results on identical test material reported by two laboratories will differ by more than the reproducibility value $R = 66.1$ mg/l corresponding to 55.2% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

Compound	Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
		s _r		r		S _R		R	
		mg/l	%	mg/l	%	mg/l	%	mg/l	%
CFB tar, range < 1 mg/l (TAR 03 and TAR 04)									
Toluene	0.3	0.055	22.1	0.154	61.8	-	-	-	-
Phenanthrene	0.3	0.031	11.1	0.087	31.2	0.080	28.7	0.23	80.6
Mean Value	0.3	0.043	16.6	0.12	46.5	0.080	28.7	0.23	80.6
CFB tar, range 1 - 10 mg/l (TAR 03 and TAR 04)									
Indene	10	0.50	4.2	1.39	11.9	1.04	8.9	2.90	24.8
Fluoranthene	6	0.33	5.2	0.93	14.4	0.73	11.4	2.06	32.0
Pyrene	6	0.32	4.8	0.91	13.5	1.00	14.8	2.79	41.5
Naphthalene	2	0.10	6.6	0.29	18.4	0.14	9.1	0.40	25.4
Pyridine	1	0.03	3.1	0.09	8.7	0.19	19.1	0.53	53.3
Mean Value	5	0.26	4.8	0.72	13.4	0.62	12.7	1.74	35.4
CFB tar, range > 10 mg/l (TAR 03 and TAR 04)									
Naphthalene	120	2.43	2.0	6.82	5.6	13.18	10.9	36.90	30.4
Toluene	30	0.99	3.4	2.77	9.5	1.42	4.9	3.98	13.6
Acenaphthylen	20	0.47	2.6	1.30	7.2	1.25	6.9	3.49	19.2
Phenanthrene	20	0.47	2.2	1.33	6.3	1.37	6.5	3.82	18.1
Mean value	48	1.09	2.6	3.05	7.1	4.30	7.3	12.05	20.3

For CFB tars applies:

Repeatability value "r"

For single compounds in the range < 1 mg/l the difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value $r = 0.12$ mg/l corresponding to 46.5% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

For single compounds in the range from 1 to 10 mg/l the difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value $r = 0.72$ mg/l corresponding to 13.4% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

For single compounds in the range from 10 to 150 mg/l the difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value $r = 3.1$ mg/l corresponding to 7.1% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

Reproducibility value "R"

For single compounds in the range < 1 mg/l single results on identical test material reported by two laboratories will differ by more than the reproducibility value $R = 0.23$ mg/l corresponding to 80.6% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

For single compounds in the range from 1 to 10 mg/l single results on identical test material reported by two laboratories will differ by more than the reproducibility value $R = 1.74$ mg/l corresponding to 35.4% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

For single compounds in the range from 10 to 150 mg/l single results on identical test material reported by two laboratories will differ by more than the reproducibility value $R = 12.1$ mg/l corresponding to 20.3% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

	Level mg/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
		s_r		r		s_R		R	
		mg/l	%	mg/l	%	mg/l	%	mg/l	%
Total chromatographable tars range 10 to 6200 mg/l									
Tar 01	4400	268.2	6.1	751.1	17.1	769.2	17.5	2154	49.0
Tar 02	6200	74.1	1.2	207.4	3.3	730.4	11.8	2045	33.0
Tar 03	300	4.3	1.5	12.2	4.1	29.7	9.9	83.2	27.7
Tar 04	23	2.3	10.1	6.5	28.2	16.9	73.7	47.4	206
Mean value	2731	87.2	4.7	244.3	13.2	386.6	28.2	1082	78.9

For total chromatographable tars applies:

Repeatability value "r"

In the range from 20 to 6200 mg/l the difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value $r = 244$ mg/l corresponding to 13.2% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

Reproducibility value "R"

For total chromatographable tars in the range from 20 to 6200 mg/l single results on identical test material reported by two laboratories will differ by more than the reproducibility value $R = 1082$ mg/l corresponding to 78.9% of the measured concentration on average not more than once in 20 cases in the normal and correct operation of the method.

The table above resulted in an overall conclusion, that there are differences in analysis precision as regards updraft tar, total chromatographable tar and CFB tar. The last-mentioned has reproducibility values a little better than the other tars, particularly at concentration levels > 10 mg/l where the average reproducibility value corresponds to about 20% of the measured concentration level. Otherwise, there are only similarly good results at single compound level. The better values for CFB tar vs updraft tar are presumably caused in updraft tars considerable complexity and some laboratories lacking experience with updraft tars. Worst is the reproducibility value for total chromatographable tars with an average reproducibility value corresponding to about 80% of the measured concentration level. This emphasizes the necessity of producing a better and more precise description of the analysis method for total chromatographable tar.

Furthermore, it can be concluded that high concentration levels result in better reproducibility value than low levels. This is most distinct on CFB tar where the average reproducibility value corresponds to about 45% of the measured concentration level when measuring below 10 mg/l and as mentioned to about 20% at concentration levels > 10 mg/l. This is not the case with updraft tar: here the average reproducibility value corresponds to 57% and 54% respectively, thus it is a much more confusing result.

It also can be concluded that in general the laboratories perform really good repeatability values with an over all average of about 15% of the measured concentration level.

For total chromatographable tars applies that it is not possible to make satisfactory determinations in case of total concentrations below 100 mg/l.

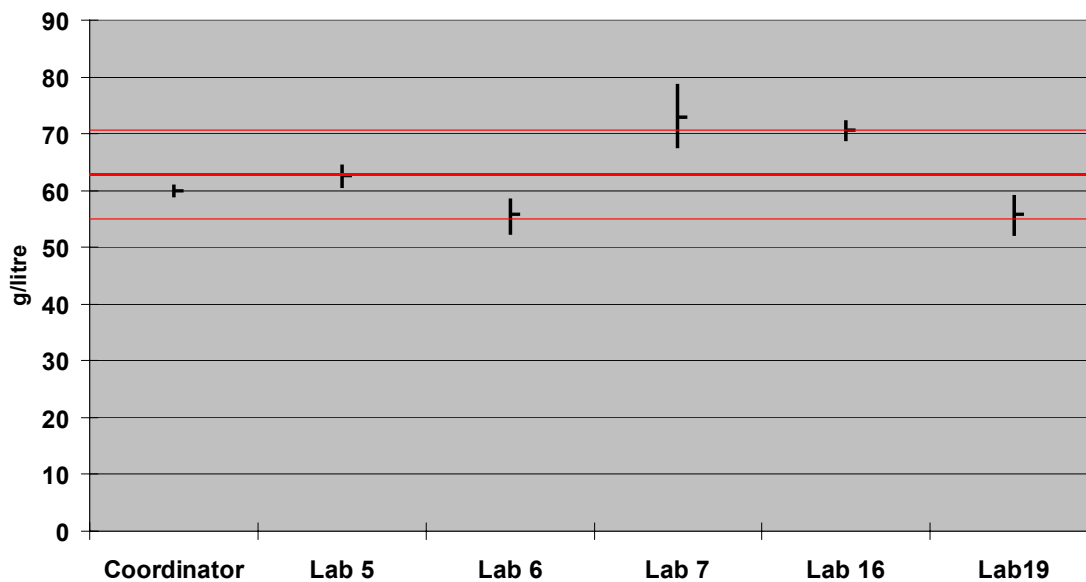
5.4. Round 03

Round Robin Test on Tar, March 2004

Round 03 - Gravimetric tar

GRAVTAR 01	Measured value [mg/ml]						Statistics		
	1	2	3	4	5	6	Mean value mg/ml	STDev mg/ml	H.W. CI (95%) mg/ml
Coordinator	59.4	59.1	60.7	60.2	60.8	59.4	59.9	0.731	0.767
Lab 5	63.3	64.23	62.09	63.02	61.97	60.76	62.6	1.213	1.273
Lab 6	55	52.5	55.1	58.3	55.4	58.3	55.8	2.221	2.331
Lab 7	73.55	78.51	71.94	70.26	67.84	75.90	73.0	3.858	4.049
Lab 16	69	71	72				70.7	1.528	3.795
Lab 19	55.3	58.9	58.4	54.2	55.6	52.4	55.8	2.481	2.604
Mean of lab means							62.96	7.383	7.747

TAR 01 - GravTar



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test:
 Pass # 1: Lab 7 is an outlier at $\alpha = 0.05$
 Bartlett test: Lab variances homogeneous?
 No ($\alpha = 0.05$)
 Yes ($\alpha = 0.01$)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes ($\alpha = 0.05$), Yes ($\alpha = 0.01$)

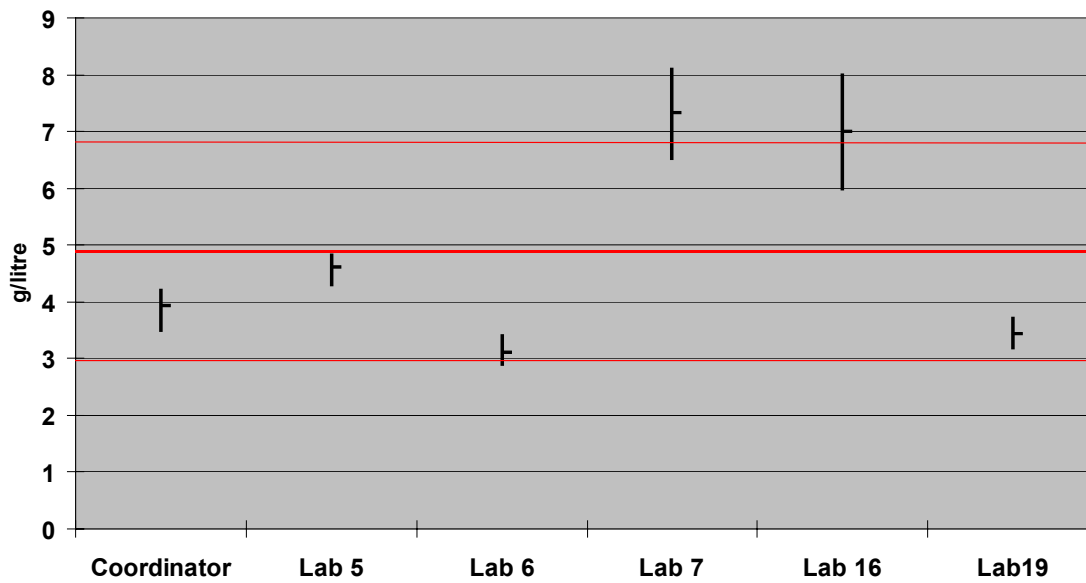
Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	62.955 g/l	Within labs STDev:	2.314 g/l
StDev of lab means:	7.383 g/l	Between labs STDev:	7.200 g/l
Half width 95% CI:	7.747 g/l		
Upper limit of 95% CI	70.702 g/l	Lower limit of 95% CI	55.207 g/l

Level [g/l]	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s_r		r		s_R		R	
	g/l	%	g/l	%	g/l	%	g/l	%
63	2.314	3.7	6.479	10.3	7.563	12.0	21.176	33.6

GRAVTAR 02	Measured value [mg/ml]						Statistics		
	1	2	3	4	5	6	Mean value mg/ml	STDev mg/ml	H.W. CI (95%) mg/ml
Coordinator	4.21	4.07	4.13	3.92	3.77	3.50	3.9	0.264	0.277
Lab 5	4.51	4.31	4.64	4.82	4.69	4.68	4.6	0.177	0.186
Lab 6	3.1	3	3.2	3.1	3.4	2.9	3.1	0.172	0.181
Lab 7	7.85	7.43	6.54	8.10	6.61	7.52	7.3	0.641	0.673
Lab 16	8	7	6				7.0	1.000	2.484
Lab 19	3.5	3.5	3.7	3.3	3.4	3.2	3.4	0.175	0.184
Mean of lab means							4.9	1.828	1.919

TAR 02 - GravTar



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test:
 Pass # 1: Lab16 is an outlier at $\alpha = 0.01$ and 0.05
 Pass # 2: Lab 7 is an outlier at $\alpha = 0.01$ and 0.05
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No ($\alpha = 0.05$)
 No ($\alpha = 0.01$)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes ($\alpha = 0.05$), Yes ($\alpha = 0.01$)

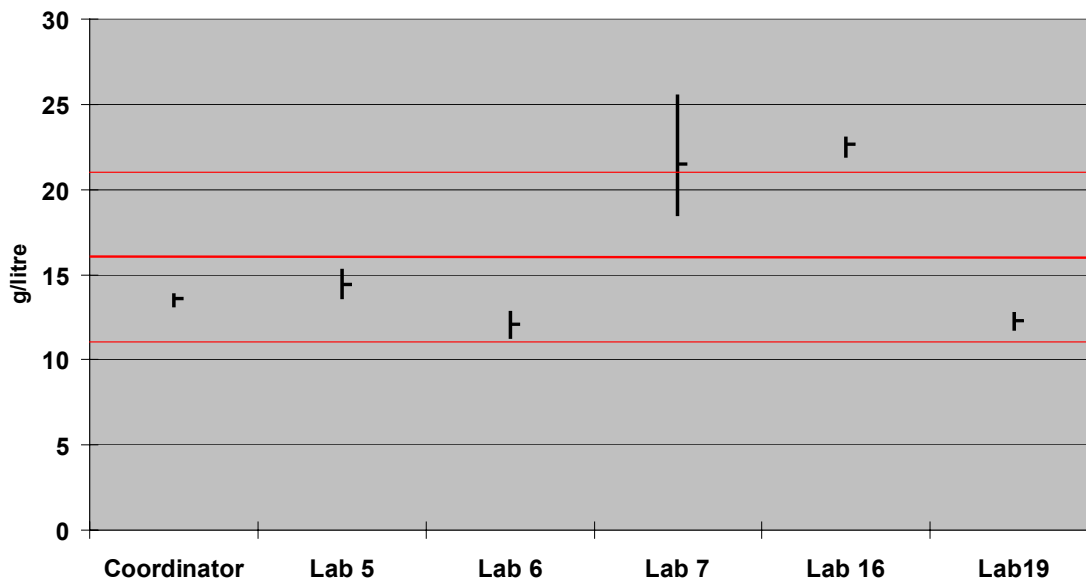
Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	4.905 g/l	Within labs STDev:	0.424 g/l
StDev of lab means:	1.828 g/l	Between labs STDev:	1.766 g/l
Half width 95% CI:	1.919 g/l		
Upper limit of 95% CI	6.824 g/l	Lower limit of 95% CI	2.987 g/l

Level [g/l]	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	g/l	%	g/l	%	g/l	%	g/l	%
5	0.424	8.6	1.145	23.3	2.105	42.9	5.683	115.9

GRAVTAR 03	Measured value [mg/ml]						Statistics		
	1	2	3	4	5	6	Mean value mg/ml	STDev mg/ml	H.W. CI (95%) mg/ml
Coordinator	13.6	13.8	13.7	13.8	13.5	13.2	13.6	0.228	0.239
Lab 5	14.76	15.22	14.12		13.65	14.3	14.4	0.603	0.748
Lab 6	12.5	11.3	12.1	12.1	11.5	12.8	12.1	0.572	0.600
Lab 7	25.49	23.58	21.39	20.98	18.52	19.09	21.5	2.655	2.786
Lab 16	23	23	22				22.7	0.577	1.434
Lab 19	12.2	12	12.7	12.6	11.8	12.4	12.3	0.349	0.366
Mean of lab means							16.1	4.742	4.977

TAR 03 - GravTar



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test:
 Pass # 1: Lab 7 is an outlier at $\alpha = 0.01$ and 0.05
 Bartlett test: Lab variances homogeneous?
 No ($\alpha = 0.05$)
 No ($\alpha = 0.01$)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes ($\alpha = 0.05$), Yes ($\alpha = 0.01$)

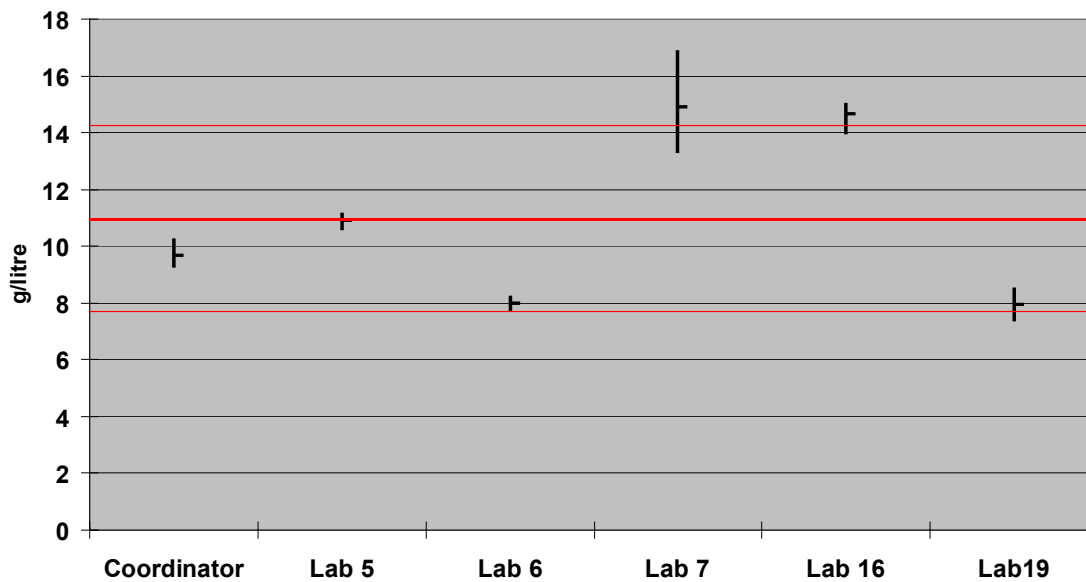
Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	16.086 g/l	Within labs STDev:	1.238 g/l
StDev of lab means:	4.742 g/l	Between labs STDev:	4.452 g/l
Half width 95% CI:	4.977 g/l		
Upper limit of 95% CI	21.063 g/l	Lower limit of 95% CI	11.110 g/l

Level [g/l]	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s_r		r		s_R		R	
	g/l	%	g/l	%	g/l	%	g/l	%
16	1.238	7.7	3.466	21.7	4.621	28.9	12.939	80.9

GRAVTAR 04	Measured value [mg/ml]						Statistics		
	1	2	3	4	5	6	Mean value mg/ml	STDev mg/ml	H.W. CI (95%) mg/ml
Coordinator	9.77	9.57	9.54	9.59	10.2	9.31	9.7	0.301	0.316
Lab 5	10.85	11.03	10.97	10.92	10.63	11.13	10.9	0.172	0.180
Lab 6	7.8	7.9	8.2	8	8.1	7.9	8.0	0.147	0.154
Lab 7	16.85	16.30	15.58	13.86	13.45	13.34	14.9	1.539	1.615
Lab 16	15	15	14				14.7	0.577	1.434
Lab 19	8.3	7.4	7.9	8.5	7.9	7.7	8.0	0.399	0.418
Mean of lab means							11.0	3.125	3.280

TAR 04 - GravTar



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test:
 Pass # 1: Lab7 is an outlier at $\alpha = 0.01$ and 0.05
 Pass # 2: Lab 16 is an outlier at $\alpha = 0.05$
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No ($\alpha = 0.05$)
 No ($\alpha = 0.01$)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes ($\alpha = 0.05$), Yes ($\alpha = 0.01$)

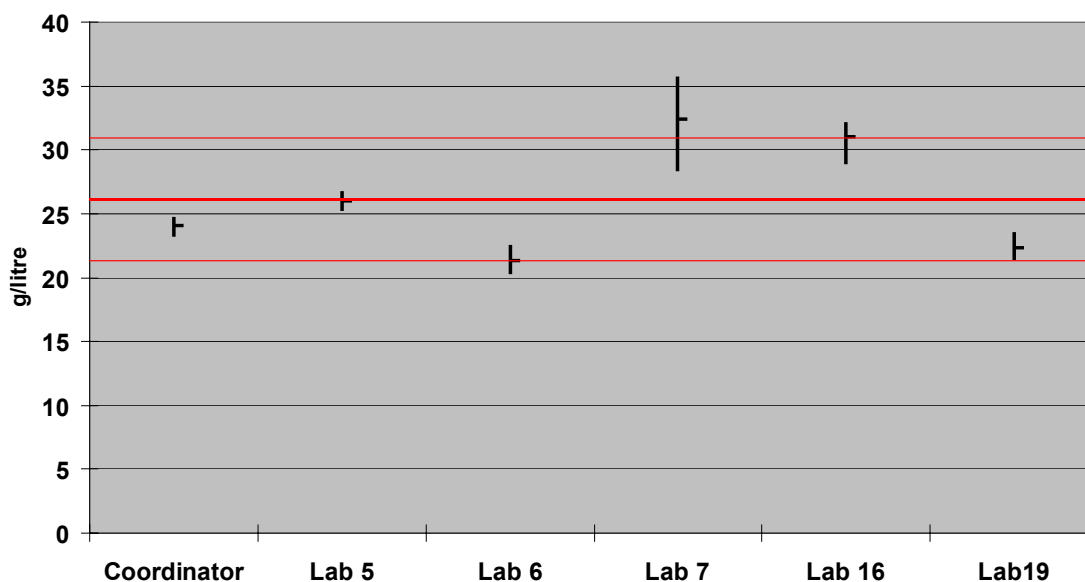
Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	11.014 g/l	Within labs STDev:	0.720 g/l
StDev of lab means:	3.125 g/l	Between labs STDev:	3.008 g/l
Half width 95% CI:	3.280 g/l		
Upper limit of 95% CI	14.294 g/l	Lower limit of 95% CI	7.734 g/l

Level [g/l]	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	g/l	%	g/l	%	g/l	%	g/l	%
11	0.720	6.5	2.016	18.3	3.093	28.1	8.660	78.7

GRAVTAR 05	Measured value [mg/ml]						Statistics		
	1	2	3	4	5	6	Mean value mg/ml	STDev mg/ml	H.W. CI (95%) mg/ml
Coordinator	24	23.3	23.4	24.5	24.6	24.6	24.1	0.599	0.628
Lab 5	25.87	25.71	26.06	25.33	26.63	26.47	26.0	0.484	0.508
Lab 6	22.4	22.4	20.8	20.4	20.8	21.3	21.4	0.862	0.905
Lab 7	35.59	33.04	34.80	28.45	31.25	31.20	32.4	2.634	2.764
Lab 16	32	29	32				31.0	1.732	4.303
Lab 19	21.5	22.7	22.2	22.5	23.4	21.7	22.3	0.695	0.729
Mean of lab means							26.2	4.569	4.795

TAR 05 - GravTar



TESTING OF OUTLYING LAB MEANS TESTING OF VARIANCES

Dixon's test: No outlier detected
 Nalimov t-test: No outlier detected
 Grubb's test: No outlier detected

Cochran test:
 Pass # 1: Lab7 is an outlier at $\alpha = 0.01$ and 0.05
 Pass # 2: Lab 16 is an outlier at $\alpha = 0.01$ and 0.05
 Pass # 3: No outlier detected
 Bartlett test: Lab variances homogeneous?
 No ($\alpha = 0.05$)
 No ($\alpha = 0.01$)

ANOVA

Snedecor F-test: Difference between labs statistically significant?
 Yes ($\alpha = 0.05$), Yes ($\alpha = 0.01$)

Snedecor F-test and Bartlett test show that pooling is: NOT ALLOWED

Mean of lab means:	26.192 g/l	Within labs STDev:	1.358 g/l
StDev of lab means:	4.569 g/l	Between labs STDev:	4.456 g/l
Half width 95% CI:	4.795 g/l		
Upper limit of 95% CI	30.987 g/l	Lower limit of 95% CI	21.396 g/l

Level [g/l]	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
	s _r		r		s _R		R	
	g/l	%	g/l	%	g/l	%	g/l	%
26	1.358	5.2	3.802	14.6	4.658	17.9	13.043	50.2

5.5. Evaluation of results from round 03

	Level g/l	Repeatability conditions (within labs)				Reproducibility conditions (between labs)			
		s _r		r		S _R		R	
		g/l	%	g/l	%	g/l	%	g/l	%
Gravimetric tars range 1 to 100 g/l									
Gravtar 01	63	2.31	3.7	6.48	10.3	7.56	12.0	21.18	33.6
Gravtar 02	5	0.42	8.6	1.15	23.3	2.11	42.9	5.68	115.9
Gravtar 03	16	1.24	7.7	3.47	21.7	4.62	28.9	12.94	80.9
Gravtar 04	11	0.72	6.5	2.02	18.3	3.09	28.1	8.66	78.7
Gravtar 05	26	1.36	5.2	3.80	14.6	4.66	17.9	13.04	50.2
Mean value	24.2	1.21	6.3	3.38	17.6	4.41	26.0	12.30	71.9

The results of the gravimetric tar measurements are still not fully acceptable with an average reproducibility value corresponding to about 72% of the measured concentration. The reason can probably be found in the results from lab 7 and 16 which results in average are more than 50% higher than the other laboratories results as it appears from the following table.

	Gravtar 01 g/l	Gravtar 02 g/l	Gravtar 03 g/l	Gravtar 04 g/l	Gravtar 05 g/l
Coord.	59.9	3.9	13.6	9.7	24.1
Lab 5	62.6	4.6	14.4	10.9	26.0
Lab 6	55.8	3.1	12.1	8.0	21.4
Lab 19	55.8	3.4	12.3	8.0	22.3
Mean value	58.5	3.8	13.1	9.2	23.5
Lab 7	73.0	7.3	21.5	14.9	32.4
Lab 16	70.7	7.0	22.7	14.7	31.0
Mean value	71.9	7.2	22.1	14.8	31.7
Deviation %	22.8	90.7	68.7	61.7	35.2

The deviations are increasing with decreasing tar concentrations. This might suggest that the influence from the water content in the samples is more clear in thin solutions. It should be borne in mind that the samples are mixed from the following three fractions:

	Solution 1 g/l	Solution 2 g/l	Solution 3 g/l	Solution 4 g/l	Solution 5 g/l
"Heavy tar"	55.0	1.6	1.6	6.5	22.5
Aqueous tar	320	80	320	120	120
Isopropanol	490	722	408	686	674

The "heavy tar" fraction should be recovered more or less 100% after evaporation and the aqueous tar fraction presumes to contribute to the gravimetric tar value only with a small amount. The four laboratories with the lower values find that the aqueous tar in average contribute with 4 g/l to the gravimetric tar value, and lab 7 and 16 find that the contribution is 12 g/l in average. For both values apply that there are considerably deviations, but it is obvious that the differens are caused by the water content from the aqueous tar fraction.

It can be concluded that the current method for determination of gravimetric tar only gives a very rough estimate of the gravimetric tar content in producer gas. This lead to the recommendation that further investigations of the evaporation method and a more precise description of the method should be carried out.

6. References

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- \6\ Draft Standard, CEN/TC BT/TF 143 "Sampling and Analysis of Tar and Particles in Biomass Producer Gas" Working dokument
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