

3 September 2013

**Participants in the proficiency test Proftest SYKE 6/2013**

Ref. 17 June 2013

Subject **Samples****Proficiency test Proftest SYKE 6/2013**

Enclosed please find the coal-, peat- and wood pellet samples for the Proficiency Test Proftest SYKE 6/2013 – Gross and net calorific value of fuels. In this proficiency test, 42 laboratories take part.

**1 General information**

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Subcontracting: Ramboll Finland, homogeneity testing of samples (Vantaa, Finland)  
Water Protection Association of the Kokemäenjoki River, sieving and dividing of peat and coal samples (Tampere, Finland)

Samples: Vapo Group, wood pellet (Turenki, Finland)  
Enas Ltd, preparation of peat and wood pellet sample (Jyväskylä, Finland)  
Helsinki Energia, preparation of coal sample (Helsinki, Finland)

**The participant has to read the all the related instructions before the measurements!**

For more detail information of the proficiency test protocol *Guide for participating laboratories in Proftest proficiency testing schemes* is available on our web site [www.syke.fi/proftest/en](http://www.syke.fi/proftest/en) > Running proficiency test, guide for participants.

Finnish Environment Institute (SYKE), Laboratory Centre

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## 2 Samples and measurements

This test includes three **air dried** fuel samples of peat, wood pellet and coal (about 40 g of each). Measurements are as follows using the normal analytical protocols of participating laboratory:

Sample	Sample type	Measurements
B1	Peat	Gross and net calorific value, C, S, H, N, moisture content of the analysis sample ( $M_{ad}$ ), ash content, volatile matter ( $V_{db}$ )
B2	Wood Pellet	Gross and net calorific value, C, H, N, moisture content of the analysis sample ( $M_{ad}$ ), ash content, volatile matter ( $V_{db}$ )
K1	Coal	Gross and net calorific value, C, S, H, N, moisture content of the analysis sample ( $M_{ad}$ ), ash content, volatile matter ( $V_{db}$ )

Additionally, participants shall be estimated/calculated the **emission factor EF (as received)** for peat and coal samples. For this estimation/calculation the total moisture contents as received ( $M_{ar}$ ) are:

- peat **B1** 47,8 %,
- coal **K1** 8,7 %.

## 3 Receiving of the samples

Please, check your sample package immediately after its arrival. If samples have not arrived on 10 September 2013 or samples are broken/missing, please contact to the provider by e-mail to [proftest@environment.fi](mailto:proftest@environment.fi).

Appendix 1 for notification of sample receiving is available for downloading on our website: [www.syke.fi/proftest/en](http://www.syke.fi/proftest/en) > *Running proficiency test* > *6/2013 Gross and net calorific value of fuels*. Return Appendix 1 by e-mail ([proftest@environment.fi](mailto:proftest@environment.fi)) or by fax **not later than 10 September 2013 or immediately after receiving samples**.

## 4 Storing and analysing of the samples

The delivered samples are room dried samples and they should be kept dry and at the room temperature before measurements. Samples have to be homogenated before measurements. **Moisture content of the analysis sample should be measured only after storing samples for one day in the measuring laboratory (= the reported moisture content of the analysis sample  $M_{ad}$ ).** In addition, moisture content of the analysis should be measured daily during every measuring day. This is important to eliminate the influence of humidity to the measurements.

**Analysis shall be performed as two replicate determinations (as dry basis)** according to the normal procedures of the participating laboratories. Both results are required to be reported as J/g (calorific values) and w-% (other parameters). The results will be reported on dry weight basis. Emission factor will be reported as t CO<sub>2</sub>/TJ **as received**.

## 5 Results and reporting

The results should be reported to Proftest **not later than 26 September 2013** using the electric result sheet, which is available on our website: [www.syke.fi/proftest/en](http://www.syke.fi/proftest/en) > *Running proficiency test* > *6/2013 Gross and net calorific value of fuels* or using Appendix 2. The electric result sheet should be saved as excel-file on your computer hard disk before opening. On request, the electric results sheet will be sent by e-mail ([proftest@environment.fi](mailto:proftest@environment.fi)).

**It is very important, that the reporting of the results is made in the requested unit. The provider will generally not correct the result if it has been reported wrongly.**

Also the measurement uncertainties, the approach used for the estimation of the measurement uncertainties and information about accreditation should be reported. Appendix 3 contains instructions for how to fill in the results sheet (Appendix 2). Appendix 4 explains the method codes for the different measurements. **Additionally, there will be electronic questionnaire (Webropol) of used analytical methods. The internet link to the questionnaire will be send by e-mail to participants.**

With the analytical methods also the relative humidity (%) of the measuring room should be reported as an average of the measuring dates.

The preliminary result lists will be sent to the participants on week 41 (2013) and the final report will be published on the ProfTest SYKE website ([www.syke.fi/proftest/en](http://www.syke.fi/proftest/en)) by the end of February 2014. The participants will be informed when the report is available.

## 6 Assigned value

The robust mean value of the results will be used as the assigned value in measurement of the samples. If there will be few results, the mean value of the results will be used as the assigned value. In special case data of expert laboratories, might be used in the calculation of the assigned value. The data produced according to the given instructions will be used for the calculation of the assigned value.

## 7 Performance evaluation

The results will be treated using the variance analysis (ANOVA). Therefore reporting of two replicates is necessary. In preliminary performance evaluation the requirements presented in the international standards will be used as follows:

Sample	Measurement	Repeatability	Reproducibility	Reference
<b>B1 Peat B2 WoodPellet</b>	Calorific value	± 120 J/g	± 300 J/g	EN 14918 (2010)
	C	± 0,5 %	± 0,5 %	EN 15104 (2011)
	H	± 0,25 %	± 0,5 %	EN 15104 (2011)
	N	± 10 % relative	± 20 % relative	EN 15104 (2011)
	S	± 0,02+0,03*X % <sup>1</sup>	± 0,02+0,09*X % <sup>1</sup>	ASTM D 4239 (2012)
	Ash content	± 0,2 %	± 0,3 %	EN 14775 (2010)
	Analytical moisture	± 0,2 %		EN 14774-3 (2010)
	Volatile matter	± 2 %*X	± 4 %*X	EN 15148 (2010)
<b>K1 Coal</b>	Calorific value	± 120 J/g	± 300 J/g	ISO 1928 (2009)
	C	± 0,3 %	± 1,0 %	ISO 29541 (2010)
	H	± 0,06 %	± 0,25 %	ISO 29541 (2010)
	N	± 0,03 %	± 0,15 %	ISO 29541 (2010)
	S	± 0,02+0,03*X % <sup>1</sup>	± 0,02+ 0,09*X % <sup>1</sup>	ASTM D 4239 (2012)
	Ash content when <10 % when >10 %	± 0,2 %*X % <sup>1</sup> ± 2 %*X % <sup>1</sup>	± 0,3 %*X % <sup>1</sup> ± 3 %*X % <sup>1</sup>	ISO 1171 (2010)
	Analytical moisture	± 0,2 %		DIN 51718 (2002)
	Volatile matter	± 3 %*X	0,5 % absolute or ± 4 %*X , whichever is greater	ISO 562 (2010)

<sup>1</sup>X = mean value

Evaluation of a laboratory performance will be based on z score. The preliminary target values of the total standard deviation for proficiency assessment are the reproducibility requirements presented above. Based on the standard deviation of C, H, N, S data the total standard deviation for proficiency assessment will be compared with the reproducibility given in the international standards.

## 8 Confidentiality

The results of the participants will be treated anonymous.

## 9 Costs

The costs depend on the number of the ordered samples. The participation to the proficiency test will be charged totally maximum **730 €** plus VAT (23%), where applicable. The paper copy of final report will cost 30 €.

The basic cost is 280 € and the costs of each sample are as follows:

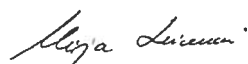
Sample type	Cost/sample
Peat, B1	150 €
Wood Pellet, B2	150 €
Coal, K1	150 €

The participant is responsible for possible clearance or customs fee of the samples.

We will send out the invoice after delivering the lists of the preliminary results. If the invoicing address or any other additional information has to be corrected after the invoicing, the extra handling cost will be charged.

Do not hesitate to contact the organiser, if you need further information.

Sincerely yours,



Mirja Leivuori  
PT- coordinator

Appendix 1	Notification of sample receiving
Appendix 2	Result sheet
Appendix 3	Information for the result sheet and estimation procedure of uncertainty
Appendix 4	Method descriptions

## Appendix 1

### NOTIFICATION OF SAMPLE RECEIVING

**The samples arrived**

Date\_\_\_\_\_ Time\_\_\_\_\_

The samples were OK? \_\_\_\_\_ (Yes or No )

Container\_\_\_\_\_were missing.

**Notes:** \_\_\_\_\_

\_\_\_\_\_

Laboratory: \_\_\_\_\_

Contact person: \_\_\_\_\_

Address: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

Phone: \_\_\_\_\_ Fax: \_\_\_\_\_

E-mail: \_\_\_\_\_

**Please, return this form not later than 10 September 2013 or immediately after receiving of samples. This form is also available on our website: [www.syke.fi/proftest/en](http://www.syke.fi/proftest/en) > Running proficiency test > 6/2013 Gross and net calorific value of fuels.**

**Return this form by e-mail to: [proftest@environment.fi](mailto:proftest@environment.fi) or by fax to the number: +358 9 448 320**

## RESULT SHEET

Results, measurement uncertainties and the method of uncertainty evaluation, number of sample container, method accredited or not and quality system (see Appendix 3).

Please report the results of two replicates.

### Result codes to calorific value:

- Gross calorific value  
 $q_{V,gr,d}$ :  $q-V,gr,d$  (as dry basis after corrections)
- Net calorific value  
 $q_{p,net,d}$ :  $q-p,net,d$  (as dry basis after corrections)

### Reporting of the results (dry weight basis):

- |  |  |
|--|--|
| • Calorific values   | to the nearest 1 J/g                     |
| • H, S, N  | to the nearest 0,001 w-%                 |
| • C  | to the nearest 0,01 w-%                  |
| • Moisture content ( $M_{ad}$ ), w-%                       | to the nearest 0,01 w-%                  |
| • Ash content, w-%   | to the nearest 0,01 w-%                  |
| • Emission factor (EF, as received), t CO <sub>2</sub> /TJ | to the nearest 0,1 t CO <sub>2</sub> /TJ |
| • Volatile matter, w-%                                     | to the nearest 0,01 w-%                  |

Please, return this result sheet not later than 26 September 2013. Sheet is also available on our website [www.syke.fi/proftest/en](http://www.syke.fi/proftest/en) > Running proficiency test > 6/2013 Gross and net calorific value of fuels.

Return by e-mail to: [proftest@environment.fi](mailto:proftest@environment.fi) or by fax to the number: +358 9 448 320 or by post to the address:

Proftest SYKE  
 Finnish Environment Institute  
 Laboratory Centre  
 Hakuninmaantie 6  
 FI 00430 Helsinki  
 Finland

## RESULT SHEET

NOTE! Please, check that your results are in the requested unit and in the correct line!

INPUT FORM VERSION 2.1											
Comparison		6/2013									
Laboratory											
Contact person		E-mail									
Group		Quality system code (0 = none)									
Lab code		Lab's data is reported to the environment authorities? (y/l/n)									
Analyte	Unit	Sample	Method		Bottle No.	Replicates				Uncertainty	
			Mth No.	Accre-dited		Result 1	Result 2	Result 3	Result 4	UC No.	UC%
q-V,gr,d	J/g	B1									
q-p,net,d	J/g	B1									
C,d	w%	B1									
H,d	w%	B1									
N,d	w%	B1									
S,d	w%	B1									
M <sub>ad</sub> ,d	w%	B1									
Ash,d	w%	B1									
EF	t CO <sub>2</sub> /TJ	B1									
V <sub>db</sub>	w%	B1									
q-V,gr,d	J/g	B2									
q-p,net,d	J/g	B2									
C,d	w%	B2									
H,d	w%	B2									
N,d	w%	B2									
M <sub>ad</sub> ,d	w%	B2									
Ash,d	w%	B2									
V <sub>db</sub>	w%	B2									
q-V,gr,d	J/g	K1									
q-p,net,d	J/g	K1									
C,d	w%	K1									
H,d	w%	K1									
N,d	w%	K1									
S,d	w%	K1									
M <sub>ad</sub> ,d	w%	K1									
Ash,d	w%	K1									
EF	t CO <sub>2</sub> /TJ	K1									
V <sub>db</sub>	w%	K1									

## INFORMATION FOR THE RESULT SHEET AND ESTIMATION PROCEDURE OF UNCERTAINTY

**"Lab code"**: permanent code of the laboratory, available on request (profest@environment.fi)

**"Group"**: group code of the laboratory = the first digit in the lab code

**"Quality system code"**:

- 0 = No quality system
- 1 = ISO 17025
- 2 = ISO 9000- series

**"Lab's data is reported to the Finnish environment authorities"**:- mark N for foreign participants

**"Mth. No."**: choose code from the appendix 4

**"Accredited"**: choose Yes (Y) or No (N).

**"Bottle No"**: the bottle number (showing filling order) of the sample bottle.

### ESTIMATION PROCEDURE OF UNCERTAINTY:

**"UC No:"** the procedure used for the estimation of the expanded measurement uncertainty \*) at 95 % confidence level (IQC = internal quality control)

1. Using the IQC data only from synthetic control sample and/or CRM (X-chart), see e.g. NORDTEST TR 537<sup>1)</sup>
2. Using the IQC data from synthetic sample (X-chart) together with the IQC data from routine sample replicates (R-chart or r%-chart), see e.g. NORDTEST TR 537<sup>1)</sup>
3. Using the IQC data and the results obtained in proficiency tests, see e.g. NORDTEST TR 537<sup>1)</sup>
4. Using the data obtained in method validation
5. Using the "modeling approach" (GUM Guide or EURACHEM Guide Quantifying Uncertainty in Analytical Measurement)<sup>2)</sup>
6. Other procedure, please specify
7. No uncertainty estimation

**"UC%:"** Report measurement uncertainty percent (%)

Add the equivalent code about your estimation method of uncertainty on the result sheet (Appendix 4 or on the electronic result sheet).

**\*) Please, check that you report the measurement uncertainty as the expanded uncertainty (k=2).**

<sup>1)</sup> <http://www.nordtest.info>

<sup>2)</sup> <http://www.eurachem.org>



## METHOD DESCRIPTIONS

Add the method code in the electronic result sheet or in the result sheet (Appendix 2).

Return also this sheet if you used option "Other method" send this Appendix 4 with the result form to the organizer or write a short method description to the page "METHOD" in the electric result sheet.

Analyte	Code	Method
<b>q-V,gr,d q-p,net,d</b>	<b>1</b>	EN 14918
	<b>2</b>	ISO 1928
	<b>3</b>	DIN 51900
	<b>4</b>	ASTM D 5865
	<b>5</b>	Other, what:
<b>Ash</b>	<b>1</b>	EN 14775
	<b>2</b>	ISO 1171
	<b>3</b>	DIN 51719
	<b>4</b>	ASTM D 7582
	<b>5</b>	Other, what:
<b>C, H, N</b>	<b>1</b>	EN 15104
	<b>2</b>	ISO 29541
	<b>3</b>	ASTM D 5373
	<b>4</b>	Other, what:
<b>S</b>	<b>1</b>	EN 15289
	<b>2</b>	ISO 334
	<b>3</b>	ASTM D 4239
	<b>4</b>	Other, what:
<b>V<sub>db</sub></b>	<b>1</b>	EN 15148
	<b>2</b>	ISO 562
	<b>3</b>	Other, what:
<b>M<sub>ad</sub></b>	<b>1</b>	EN 14774
	<b>2</b>	ISO 589
	<b>3</b>	DIN 51718
	<b>4</b>	ASTM D 7582
	<b>5</b>	Other, what:

