



**Swedish University of Agricultural Sciences**  
**Dept of Forest Biomaterials and Technology**

# **Certificate**

## **Wood Fuel Reference Material NJV 94-5**

**This Reference Material is intended for use in the control of instruments and methods in energy parameter and elemental analysis of wood fuel.**

### **Description of material**

The reference material consists of waste from a saw mill, Bygdsiljum, Västerbotten, Sweden. The wood species is pine and the approximate composition of the wood fuel is bark : chip (70 : 30).

The wood fuel has been dried at 60 °C during 10 days. The fuel was then ground in a hammer mill (Kamas Industri AB) with a 2 mm sieve and finally in a knife mill (Retsch) with a 1 mm sieve. The dry matter content is approximately 94 %.

### **Homogenization and test of homogeneity**

The reference material was homogenized by thorough mixing in a large sack. It was then packed in dark 200 mL glass pots with sqrew lids, using a sample divider (Retsch PTZ).

The reference material was tested for homogeneity by duplicate analyses of 30 randomly chosen samples with respect to the content of ash, carbon, hydrogen and nitrogen. The results were analysed by "Analysis of variation (ANOVA)". The material was considered homogeneous if the variation between units, including inhomogeneity and analytical error, was not significantly larger than the analytical error.

No inhomogeneity was observed with respect to the analysed parameters.

### **Storage**

The reference material should be stored in a dark and dry place in closed containers, preferably below room temperature. A similar material has been stored for over 5 years without any change in composition. Control of this material with respect to degradation will be carried out continuously.

## Certifying procedure

The composition of this reference material has been determined in a proficiency test where 13 laboratories from Sweden, Finland and Denmark participated. The laboratories are listed in alphabetic order on page 4. Two randomly chosen samples of the wood fuel material were distributed to the laboratories, together with instructions and report forms. The participants were requested to make duplicate determinations on each sample of the wood fuel material and report the results on a dry matter basis.

The statistical evaluation procedure was as follows:

1. Laboratory means were calculated on the basis of the reported individual determinations. All laboratories that reported one or more "Less than results" for a parameter were excluded from the statistical treatment of that parameter.
2. The set of data was analysed for technical outliers, which were rejected before the statistical evaluation.
3. The set of data was tested for statistical outliers by using the Dixon outlier test and the outliers were rejected. If several results were suspected as outliers, a consecutive Dixon outlier test was used.
4. The consensus value and its confidence interval at a significance level of 0.05 were calculated using a one-stage nested design according to "Certification of reference materials - General and statistical principles", **ISO GUIDE 35 : 1989**.

## Analytical methods

| Parameter       | Methods of final determination                                |
|-----------------|---|
| Ash             | SS 18 71 71; ISO 1171 (500 °C); Leco-MAC 400 (550 °C)         |
| Volatile matter | SS-ISO 562; ISO 562; DIN 51720                                |
| Calorific value | SS 18 71 82; ISO 1928; DIN 51900; ASTM D 3286                 |
| Carbon          | CHN-analyzer  |
| Hydrogen        | CHN-analyzer  |
| Nitrogen        | CHN-analyzer; Kjeldahl  |
| Sulfur          | SS 18 71 76; SS 18 71 77; ASTM D 4239; Nefelometry            |
| Chlorine        | SS 18 71 54; ASTM D 2361; ASTM D 4208 with Ion Chromatography |
| Phosphorous     | Colorimetry; ICP-AES; XRF                                     |
| Aluminium       | FAAS; ICP-AES; XRF  |
| Calcium         | FAAS; ICP-AES; XRF  |
| Iron            | FAAS; ICP-AES; XRF  |
| Magnesium       | FAAS; ICP-AES; XRF  |
| Manganese       | FAAS; ICP-AES; XRF  |
| Potassium       | FAAS; FAES; ICP-AES; XRF                                      |
| Silicon         | FAAS; ICP-AES; XRF  |
| Sodium          | FAAS; FAES; ICP-AES; XRF                                      |
| Arsenic         | AAS-hydride generation; GFAAS; ICP-MS                         |
| Cadmium         | GFAAS; ICP-MS   |
| Chromium        | FAAS; GFAAS; ICP-MS   |
| Copper          | FAAS; GFAAS; ICP-MS   |
| Lead            | GFAAS; ICP-MS   |
| Titanium        | GFAAS; ICP-AES; XRF   |
| Zink            | FAAS; ICP-AES   |

## Instructions for use

Rehomogenisation of the bottle contents prior to sampling is necessary. For the analysis of e.g., calorific value, carbon, hydrogen, nitrogen and sulphur by using high temperature combustion methods, the sample should be pelleted before analysis. For the other methods the sample should be taken as it is. The correction to dry mass should be obtained on a separate portion from the bottle by drying in an oven at  $105 \pm 2$  °C to constant mass.

## Certified values

**Table** Certified values of the composition of Wood Fuel Reference Material NJV 94-5

| Parameter                | Concen- <sup>1</sup><br>tration | Confidence <sup>2</sup><br>interval | No of <sup>3</sup><br>labs | No of <sup>4</sup><br>results |
|--------------------------|---------------------------------|-------------------------------------|----------------------------|-------------------------------|
| Ash, (%)                 | 1,22                            | ± 0,040                             | 12 (0)                     | 43 (0)                        |
| Calorific value, (MJ/kg) | 20,6                            | ± 0,15                              | 9 (1)                      | 32 (4)                        |
| Sulphur, (%)             | 0,018                           | ± 0,0039                            | 6 (0)                      | 28 (0)                        |
| Phosphorous, (%)         | 0,021                           | ± 0,0028                            | 5 (1)                      | 14 (2)                        |
| Aluminium, (%)           | 0,026                           | ± 0,0032                            | 7 (1)                      | 22 (2)                        |
| Calcium, (%)             | 0,35                            | ± 0,022                             | 8 (1)                      | 26 (2)                        |
| Iron, (%)                | 0,007                           | ± 0,0016                            | 9 (0)                      | 27 (0)                        |
| Magnesium, (%)           | 0,030                           | ± 0,0018                            | 8 (1)                      | 26 (2)                        |
| Manganese, (%)           | 0,021                           | ± 0,0038                            | 8 (0)                      | 26 (0)                        |
| Potassium, (%)           | 0,09                            | ± 0,017                             | 8 (1)                      | 26 (2)                        |
| Sodium, (%)              | 0,004                           | ± 0,0012                            | 8 (0)                      | 26 (0)                        |
| Arsenic, (mg/kg)         | 0,8                             | ± 0,24                              | 5 (0)                      | 18 (0)                        |
| Cadmium, (mg/kg)         | 0,27                            | ± 0,028                             | 6 (1)                      | 23 (2)                        |
| Chromium, (mg/kg)        | 0,8                             | ± 0,30                              | 5 (0)                      | 22 (0)                        |
| Copper, (mg/kg)          | 2,2                             | ± 0,30                              | 7 (0)                      | 26 (0)                        |
| Lead, (mg/kg)            | 0,68                            | ± 0,025                             | 5 (1)                      | 20 (2)                        |
| Zinc, (mg/kg)            | 38                              | ± 8,5                               | 7 (0)                      | 26 (0)                        |

<sup>1</sup> expressed on dry matter basis

<sup>2</sup> significance level of 0,05

<sup>3</sup> accepted laboratories (eliminated laboratories)

<sup>4</sup> accepted results (eliminated results)

## Information values

**Table** Information values of the composition of Wood Fuel Reference Material NJV 94-5

| Parameter                                 | Concen- <sup>1</sup><br>tration | Confidence <sup>2</sup><br>interval | No of <sup>3</sup><br>labs | No of <sup>4</sup><br>results |
|---|---------------------------------|-------------------------------------|----------------------------|-------------------------------|
| <b>Volatile matter</b> <sup>5</sup> , (%) | 79,2                            | ± 0,60                              | 9 (0)                      | 32 (0)                        |
| <b>Carbon</b> , (%)                       | 51,7                            | ± 0,33                              | 3 (1)                      | 17 (2)                        |
| <b>Hydrogen</b> , (%)                     | 6,04                            | ± 0,082                             | 4 (0)                      | 19 (0)                        |
| <b>Nitrogen</b> , (%)                     | 0,17                            | ± 0,050                             | 6 (0)                      | 28 (0)                        |
| <b>Chlorine</b> , (%)                     | 0,010                           | ± 0,0036                            | 6 (2)                      | 20 (8)                        |
| <b>Silicon</b> , (%)                      | 0,023                           | ± 0,0037                            | 4 (1)                      | 10 (2)                        |
| <b>Titanium</b> , (mg/kg)                 | 2,4                             | ± 0,44                              | 3 (0)                      | 8 (0)                         |

<sup>1</sup> expressed on dry matter basis

<sup>2</sup> significance level of 0,05

<sup>3</sup> accepted laboratories (eliminated laboratories)

<sup>4</sup> accepted results (eliminated results)

<sup>5</sup> reported as an information value due to the high dependency of experimental conditions on the result for this operationally defined parameter

## Participating laboratories

ANALYTICA AB, Täby, Sweden

Central Laboratory Agricultural Research Centre of Finland, Jokioinen, Finland

dk-TEKNIK, Söborg, Denmark

Graf-Analys, Jönåker, Sweden

Helsingborg Energi AB, Helsingborg, Sweden

Hjortens Lab AB, Östersund, Sweden

Dept. of Agricultural Research for Northern Sweden, Swedish University of Agricultural Sciences, Umeå, Sweden

Ljunga Lab AB, Ljungaverk, Sweden

OY Keskuslaboratorio - Centrallaboratorium, Espoo, Finland

SGAB-analys, Luleå, Sweden

SP, Swedish National Testing and Research Institute, Borås, Sweden

VAPO OY, Jyväskylä, Finland

VTT, Combustion and thermal engineering Laboratory, Jyväskylä, Finland

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