Sampling and sample reduction of solid biofuels

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CEN 335 - Technical Specifications

**Currently**
- CEN/TS 14778-1:2006 Solid biofuels – Sampling
  Part 1: Methods for sampling
- CEN/TS 14778-2:2006 Solid biofuels – Sampling
  Part 2: Methods for sampling of particulate material transported in lorries
- CEN/TS 14779:2006 Solid biofuels – Sampling – Methods for preparing sampling plans and sampling certificates

**In preparation**
- EN 14778 Solid biofuels – Sampling
- EN 14780 Solid biofuels – Methods for sample preparation
Important of sampling

Deviate analyses results from “true” value depending on:

- Sampling: 80%
- Sample preparation: 15%
- Analyses: 5%

National Coal Board (GB) also verified for other bulk materials as farm products, industrial minerals etc.
Every particle in the lot should have an equal probability of being included in the sample. When this principle cannot be applied in practice, the sampler shall note the limitations in the sampling plan.
Sampling plan

1. What kind of job?
   - New job
   - Routine job

2. Full sampling plan (6.2)

3. Brief sampling plan (6.3)

4. Calculate the number of increments per lot (7)

5. Calculate the increment size (8)

6. Calculate the required volume (mass) needed for the required determinations (9)

7. Visually inspect the lot (10)

8. Determine the sampling method (12)

   - Fuels in motion (12.2)
   - Stationary materials (12.3)
   - Specific materials (12.4)
<table>
<thead>
<tr>
<th>Sampling plan reference number</th>
<th>date</th>
</tr>
</thead>
<tbody>
<tr>
<td>unique sample identification number</td>
<td></td>
</tr>
<tr>
<td>name of sampler</td>
<td>time</td>
</tr>
<tr>
<td>E-mail</td>
<td>telephone</td>
</tr>
<tr>
<td>lot or sub-lot identification number</td>
<td>packaging of the laboratory sample</td>
</tr>
<tr>
<td>product</td>
<td>airtight plastic container</td>
</tr>
<tr>
<td>trade name</td>
<td>other:</td>
</tr>
<tr>
<td>biofuel supplier</td>
<td>Comments</td>
</tr>
<tr>
<td>approximate nominal top size</td>
<td>mm</td>
</tr>
<tr>
<td>mass or volume of sub-lot</td>
<td>kg, ton, or m³</td>
</tr>
<tr>
<td>mass of laboratory sample and container</td>
<td>kg</td>
</tr>
<tr>
<td>type of sub-lot</td>
<td>other</td>
</tr>
<tr>
<td>moving:</td>
<td>conveyor, silo, other</td>
</tr>
<tr>
<td>address of supplier</td>
<td></td>
</tr>
<tr>
<td>address of carrier</td>
<td></td>
</tr>
<tr>
<td>address of sampler</td>
<td></td>
</tr>
<tr>
<td>address of laboratory</td>
<td></td>
</tr>
</tbody>
</table>
Sampling plan

- **Type of fuel:**
  - Homogeneous: e.g. sawdust, nutshells, pellets, woodchips
  - Inhomogeneous: e.g. bark, used wood

- **number of increments stationary:**
  - \( n = 5 + 0,025 \cdot m_{\text{LOT}} \) for homogeneous
  - \( n = 10 + 0,040 \cdot m_{\text{LOT}} \) for homogeneous

- **number of increments moving:**
  - \( n = 3 + 0,025 \cdot m_{\text{LOT}} \) for homogeneous
  - \( n = 5 + 0,040 \cdot m_{\text{LOT}} \) for homogeneous

- **Sample amount required**
- **Determine minimum amount per increment (depending on particle size)**
Number of increments

![Graph showing the number of increments vs. mass of the lot or sub-lot in tonnes, $m_{lot}$.

The graph compares two groups: Group 1 (solid line) and Group 2 (dashed line).

- For Group 1, the number of sampling increments starts at around 10 and increases slowly as the mass of the lot or sub-lot increases.
- For Group 2, the number of sampling increments starts at a higher value and increases more rapidly as the mass increases.

Key:
- Solid line: Group 1
- Dashed line: Group 2

Y-axis: Number of sampling increments, $n$
X-axis: Mass of the lot or sub-lot in tonnes, $m_{lot}$.
<table>
<thead>
<tr>
<th>Sampling Plan Reference Number</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unique Sample Identification Number</td>
<td>Sample Equipment</td>
</tr>
<tr>
<td>Aim of Sampling</td>
<td>Manual</td>
</tr>
<tr>
<td>Property</td>
<td>CEN Method</td>
</tr>
<tr>
<td>Moisture</td>
<td>kg</td>
</tr>
<tr>
<td>Particle Size Distribution</td>
<td>kg</td>
</tr>
<tr>
<td>Bulk Density</td>
<td>kg</td>
</tr>
<tr>
<td>Particle Density</td>
<td>kg</td>
</tr>
<tr>
<td>Mechanical Durability</td>
<td>kg</td>
</tr>
<tr>
<td>Ash</td>
<td>kg</td>
</tr>
<tr>
<td>Calorific Value</td>
<td>kg</td>
</tr>
<tr>
<td>Sulphur</td>
<td>kg</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>kg</td>
</tr>
<tr>
<td>Chlorine</td>
<td>kg</td>
</tr>
<tr>
<td>Others</td>
<td>kg</td>
</tr>
<tr>
<td>Total Mass Required for Tests</td>
<td>kg</td>
</tr>
<tr>
<td>Bulk Density</td>
<td>kg/litre</td>
</tr>
<tr>
<td>Total Volume Required for Tests ($V_{req}$)</td>
<td>litre</td>
</tr>
</tbody>
</table>

If total volume required ($V_{req}$) exceeds the calculated volume of combined sample ($V_{com}$), then increase the number of increments:

- Actual number of one increments ($n_{act}$), larger than $V_{req}/V_{inc}$,

- Actual volume of the combined sample ($n_{act} \times V_{inc}$).

Method of preparing the laboratory sample from the combined sample:

- Volume of the laboratory sample.
## Calculation of volume

### Typical bulk densities of biofuels

<table>
<thead>
<tr>
<th>Biofuel</th>
<th>Bulk density kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>pellets</td>
<td>550 – 650</td>
</tr>
<tr>
<td>briquettes</td>
<td>500 – 650</td>
</tr>
<tr>
<td>fuel powder</td>
<td>200 – 250</td>
</tr>
<tr>
<td>sawdust</td>
<td>290 – 380</td>
</tr>
<tr>
<td>shavings</td>
<td>140 – 170</td>
</tr>
<tr>
<td>wood chips</td>
<td>280 – 350</td>
</tr>
<tr>
<td>straw bale</td>
<td>130 – 180</td>
</tr>
<tr>
<td>chopped straw</td>
<td>80 – 120</td>
</tr>
</tbody>
</table>
Lot 10 t

Sample

Pretreated sample

Analysis sample ~5 g

Sample should present the whole lot
Sampling from a heap
d = nominal top size of sample (95% of material pass the screen)
Contaminants causing errors
Segregation of fines during transport

Original

During transport
Scoops - 2.5 x nominal top size of solid biofuels
Sampling from tipped lorry load
Sampling of pellets from storage rooms
Coring machine
Sampling from moving streams
Sampling from conveyor belt
Sampling from conveyor belt
Combined sample

- Take increments
- Mix to get combined sample (s)
- Perform sample reduction if necessary
To preserve physical and chemical properties of samples, packaging is an important issue.

- Airtight plastic packaging (e.g.: plastic bags)
- Protection against direct sunlight
- Protection against adulteration if necessary: sealing of container
- Cooling (5°C) to avoid biological activity if necessary (especially for moisture contents above 20% and longer storage.)
Packaging and shipment

BAD EXAMPLES !!!

Information of customers and clients is necessary to avoid these mistakes!
Packaging and shipment

MORE BAD EXAMPLES !!!
Stages in the lab

Clause 8
Sample division. Combined sample

Clause 9.1
Initial sample division

Clause 9.2
Weigh the sample

Clause 9.3
Dry the sample if necessary, and allow it to rest in the laboratory for at least 24 hours

Clause 9.4
Size-reduction to <30mm by coarse cutting

Clause 9.5
Mass-reduction by a suitable method for the material

Clause 9.6
Size-reduction to <1mm using a cutting mill

Clause 9.7
Take out analysis samples

Clause 9.8
Size-reduction to <0.25mm using a cutting mill

Combined sample.

Sub-samples for determination of bulk density, durability of pellets, etc

Sub-samples for determination of particle size distribution, abrasion resistance, etc.

Sub-samples for determination of moisture content, etc

Sub-samples for determination of ash, calorific value, chemical analysis, etc.

Sub-samples for analysis where <0.25mm is required
During a mass-reduction stage, every particle in the sample before mass-reduction should have an equal probability of being included in the sub-sample retained after mass-reduction.

Care is needed to avoid loss of fine particles during milling and other operations.
**Riffle boxes**
- A riffle box shall have at least 16 slots, with adjacent slots directing material into different sub-samples, and the width of the slots shall be at least 2.5 times the nominal top size of the material to be riffled.

**Rotary sample dividers**
- A rotary sample divider shall have a feeder device adjusted so that the divider rotates at least 20 times while the sample is being divided.

**Shovels and scoops**
- A shovel or scoop used for manual mass-reduction shall have a flat bottom, edges raised high enough to prevent particles rolling off, and shall be at least 2.5 times as wide as the nominal top size of the material to be processed.

**Apparatus for size-reduction**
- Coarse cutting mill
- Cutting mill
- Sieves
- Balance
Apparatus for mass reduction

Note.: 2.5 x d (nominal top size)
Riffle box

Minimum 2.5 x nominal top size of solid biofuel materials
Quartering
Strip mix
Strip mix
Particle size reduction of straw
Grinder for size reduction

- Low self heating
- Different sieves
- Easy to clean
- Resistant to impurities
## Minimum masses to be retained during mass-reduction stages

<table>
<thead>
<tr>
<th>Nominal to size (mm)</th>
<th>Bulk density &lt; 200 kg/m³</th>
<th>Bulk density 200–500 kg/m³</th>
<th>Bulk density &gt; 500 kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>≥100</td>
<td>10 000</td>
<td>15 000</td>
<td>20 000</td>
</tr>
<tr>
<td>50</td>
<td>1 000</td>
<td>2 000</td>
<td>3 000</td>
</tr>
<tr>
<td>30</td>
<td>300</td>
<td>500</td>
<td>1 000</td>
</tr>
<tr>
<td>10</td>
<td>150</td>
<td>250</td>
<td>500</td>
</tr>
<tr>
<td>5</td>
<td>50</td>
<td>100</td>
<td>200</td>
</tr>
<tr>
<td>≤2</td>
<td>20</td>
<td>50</td>
<td>100</td>
</tr>
</tbody>
</table>
Thank you for the kind attention!

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