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Lignin and Optional Sugars Analysis for Woodchips

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PDI Test

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ABSTRACT

Lignin and Optional Sugars Analysis for Woodchips

SOP 005-S-GF-19

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GUIDELINES

$$\text{Klason lignin \%} = (\text{mass paper and tin} + \text{lignin} - \text{mass paper and tin}) / \text{mass ODW sample}$$
$$\% \text{ Total Solids} = [\text{mass dry pan} + \text{sample} - \text{mass dry pan}] / \text{mass of wet sample} \times 100$$
$$\text{Mass ODW sample} = (\text{mass air dry sample} \times \% \text{ Total solids}) / 100$$
$$\% \text{ Ash} = [(\text{mass crucible} + \text{ash} - \text{mass crucible}) / \text{ODW sample}] \times 100$$

Frozen and labeled sample filtrate should be given to Dr. Tirschner for HPLC sugars analysis

DISCLAIMER:

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BEFORE STARTING

Required Safety Supplies: Lab Coat, Nitrile Gloves, Eye Goggles, High Temperature Gloves

- 1 Prepare washed filter papers (VWR part # 28297-984)
- 2 Rinse the filter papers on a vacuum apparatus by flushing 3x with a 20 mL aliquot of nano-pure water

- 3 Place the rinsed filter paper onto an aluminum tray, up to 20 filter papers may be places onto one tray
- 4 Put the aluminum tray with rinsed filter papers into the muffle furnace at 550o C for 30 minutes
- 5 Take out the filter papers after 30 minutes, using heat resistant gloves and the tongs
- 6 Allow the aluminum tray and filter papers to cool for 1 hour at room temperature
- 7 Rinse the filter papers again on a filtration apparatus with 3 aliquots of nano-pure water
- 8 Place the filter papers onto another aluminum tray, and into an oven at 105o C overnight
- 9 Weigh the mass of the washed filter paper, and the mass of the filter paper plus aluminum tray
- 10 Weigh 250 – 300 mg of dried woodchips into a clean 250 ml beaker, add a clean glass rod to the beaker for later steps
- 11 Add 3 mL of 72% H₂SO₄ to the 250 mL beaker with pipettor while stirring the sample well, wear safety glasses, lab coat, and nitrile gloves while handling H₂SO₄
- 12 Place the beaker, sample, and glass rod into the vacuum desiccator and put under vacuum for 15 minutes
- 13 Take the beakers out of the desiccator and cover with aluminum foil
- 14 Place the covered beaker into a water bath at 30o C
- 15 Stir the contents of the beaker frequently over the 60 minutes with the glass rod
- 16 After 60 minutes, remove the beaker from the water bath

- 17 Add 84 mL of nano-pure water to the beaker, stir, and remove the glass rod free of any particles
- 18 Cover samples with aluminum foil and autoclave for 60 minutes at 120o C
- 19 Allow the beakers to cool to room temperature
- 20 Samples can sit here overnight at room temperature
- 21 If doing sugars, add 5 mL of 500 mg cellobiose solution (Internal Standard Solution)
- 22 Weigh a washed filter paper and labeled aluminum tin to the nearest 0.1 mg
- 23 Place the filter paper onto a filtration apparatus with a CLEAN Erlenmeyer vacuum flask
- 24 Pour the beaker through the filter paper and wash materials with 5 mL of nano-pure water
- 25 Transfer the filtrate to a 100 mL volumetric flask and dilute to the mark with nano-pure water.
- 26 Invert the volumetric flask 3 times to mix, and store 15 mL of filtrate into a clean, labeled scintillation vial
- 27 Scintillation vials can be frozen after the filtrate is diluted and collected
- 28 Wash the materials on the filter paper with nano-pure DI until the filtrate pH is neutral
- 29 Place the filter paper into the labeled tin and into the oven at 105o C to dry for 24-48 hours

- 30 Weigh 2-3 g of bulk material into a weighed and labeled aluminum tin for moisture determination

- 31 Ash the glass fiber filter paper/lignin sample (filter paper only, not the aluminum tray) in the muffle furnace (follow ashing SOP)