

Measuring inorganics in biomass fast pyrolysis oils

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Appendix 1: analytical protocols

Wondimu protocol

Weigh precisely 0.1 g of samples in a Teflon reactor, add 8 mL of HNO₃ and 2 mL of H₂O₂. Apply the following program and when the mineralization is completed, flush the Teflon reactor with ultrapure water and adjust to 25 mL:

Table 1. Experimental conditions (i.e. power, residence time and temperature) for the Wondimu protocol

Power (W)	Time	Temperature (°C)
800	2 min (ramp)	100
800	30 sec (ramp)	75
800	10 min (ramp)	200
800	30 sec (hold)	200
800	10 min (ramp)	220
800	30 sec (hold)	220
800	9 min 30sec (ramp)	200
800	30 sec (hold)	200
800	3 min (ramp)	230

Ashes mineralization protocol

Weigh precisely 0.1 g of samples in a Teflon reactor, add 8 mL of HNO₃. Apply the following program:

Table 2. Experimental conditions (i.e. power, residence time and temperature) for the ashes mineralization protocol (first step)

Power (W)	Time	Temperature (°C)
800	5 min (ramp)	180
800	10 min (hold)	180

Open carefully the Teflon reactor and add 2 ml of HF, apply the second program:

Table 3. Experimental conditions (i.e. power, residence time and temperature) for the ashes mineralization protocol (second step)

Power (W)	Time	Temperature (°C)
800	5 min (ramp)	220
800	10 min (hold)	220

If possible, evaporate the HF and dilute the residue with 1 ml of HNO₃. Flush the Teflon tubes with ultrapure water and adjust to 25 mL.

ISO 16967 Method

Weigh precisely 0.1 g of samples in a Teflon tubes, add 1.6 mL of HNO₃, 0.2 mL of HF and 0.6 mL of H₂O₂. Apply the following program:

Table 4. Experimental conditions (i.e. power, residence time and temperature) for the ISO 16967 method protocol (first step)

Power (W)	Time	Temperature (°C)
1500	15 min (ramp)	190
1500	10 min (hold)	190

Neutralize with 2mL of H₃BO₃ and apply the following program:

Table 5. Experimental conditions (i.e. power, residence time and temperature) for the ISO 16967 method protocol (second step)

Power(W)	Time	Temperature (°C)
1500	2 min (ramp)	150
1500	15 min (hold)	150

When the neutralization is completed, flush the Teflon tubes with ultrapure water and adjust to 25 mL.

Appendix 2: validation of the ISO 16967 a method applied for (a) the bush branches and leaves reference material and (b) the wood fuel material reference material. Expected and measured contents are represented respectively in grey and white bars. Confidence interval is given.

Figure 1. Expected elements contents and measured elements contents using the ISO 16967 method for (a) the bush branches and leaves reference material and (b) the wood fuel material reference material.

