Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
National Metrology Institute

An enhanced metrological protocol for the determination of biofuels calorific Value

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The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

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Calorific value - Challenges

1. Lack of technical protocol addresses the difficulties during samples measurements (e.g.. samples heterogeneity)
2. Gaps in the calorific value standards
3. Lack of a defined uncertainty budget

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Why is it important?

Financial prospective

Source: https://www.economiaefinanzaverde.it/tag/euro/

Operational and process design prospective

Source: https://dcadtechnologies.com/sectors/

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WP1 – Task 1.2

1. Developing an improved traceable metrological methodology to address the challenges associated with determination of the calorific value of biofuels
2. Providing a detailed uncertainty budget to harmonize the measurements uncertainty
3. Assuring fair biomass pricing

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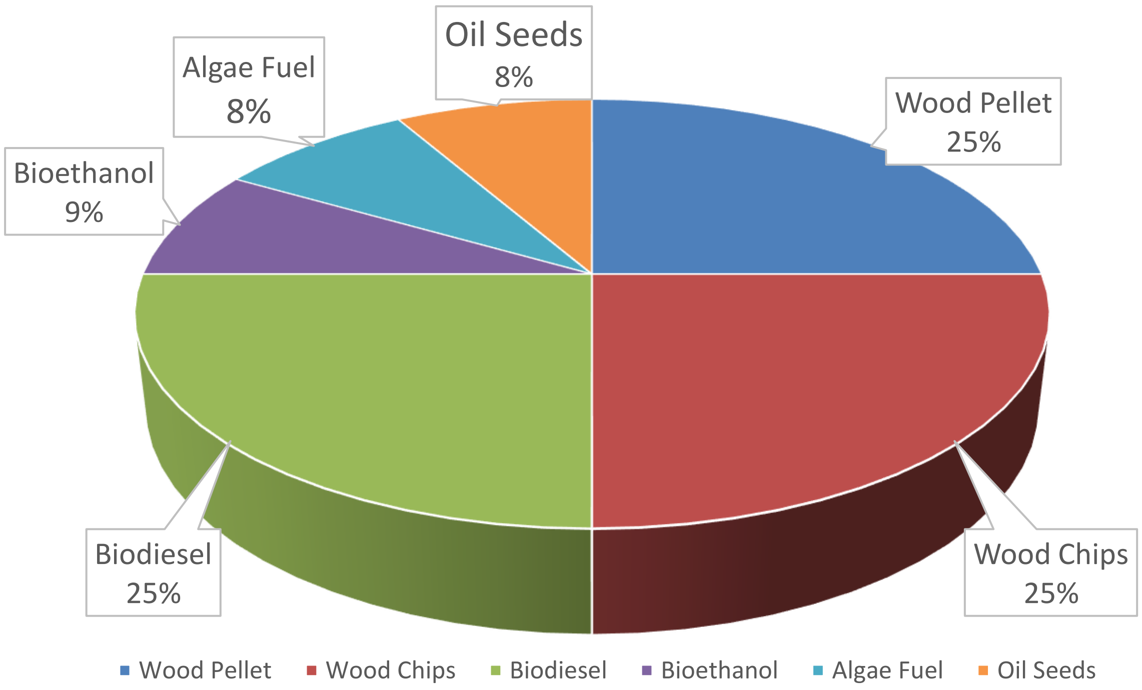
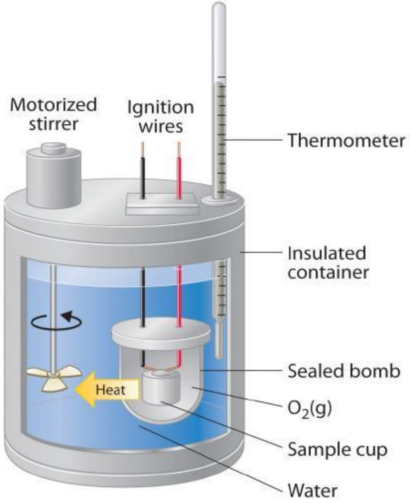
Fuel selection - Survey



Experimental setup – Bomb calorimeter



Source: https://saylordotorg.github.io/text_general-chemistry-principles-patterns-and-applications-v1.0/s09-03-calorimetry.html

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Experimental setup – Ion chromatography

Ion exchange chromatography with chemical suppression unit and Autosampler

Analyzing the anions. **F-. Cl^-.**
Br-. NO_3^-. SO_4^{2-}

Separation concept based on their affinity to the column

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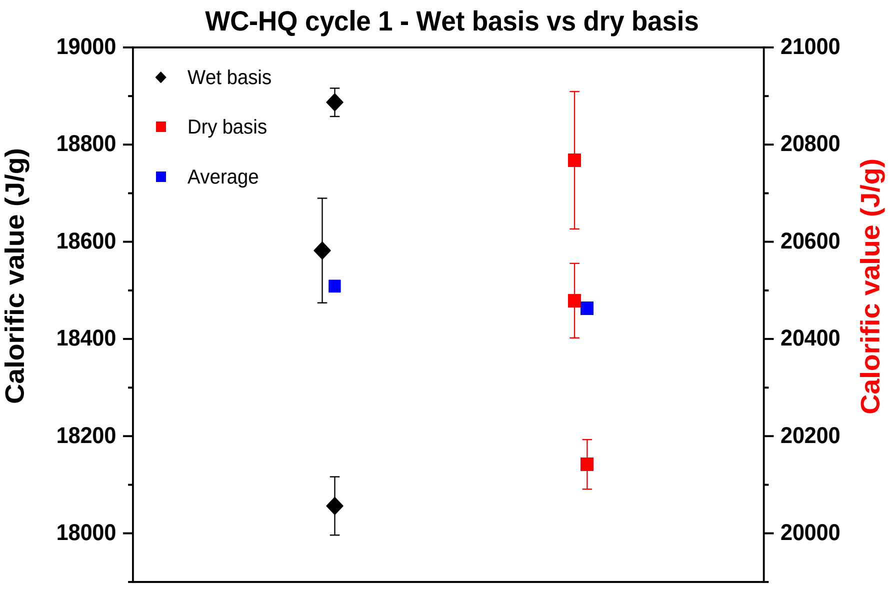
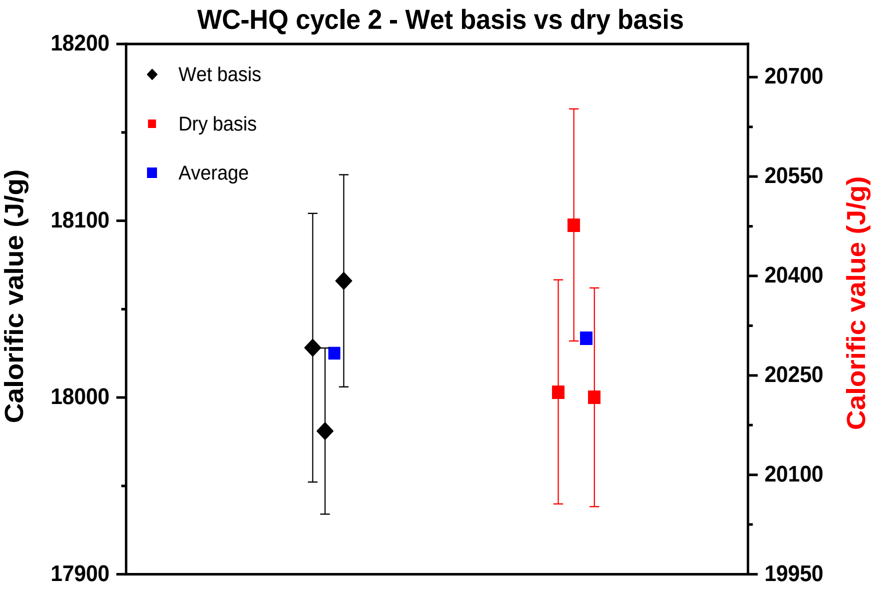
Results – Wood chips high quality (WC-HQ)



ISO 18125:2017



Improved practice

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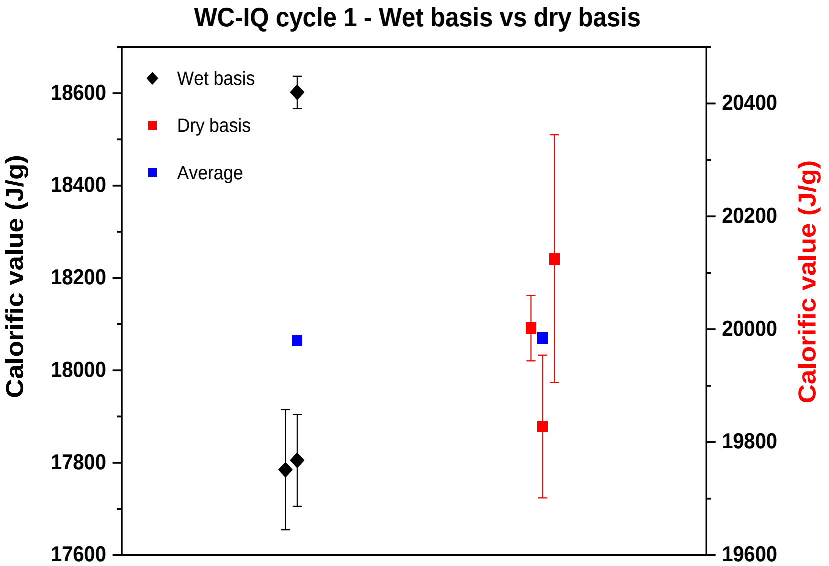
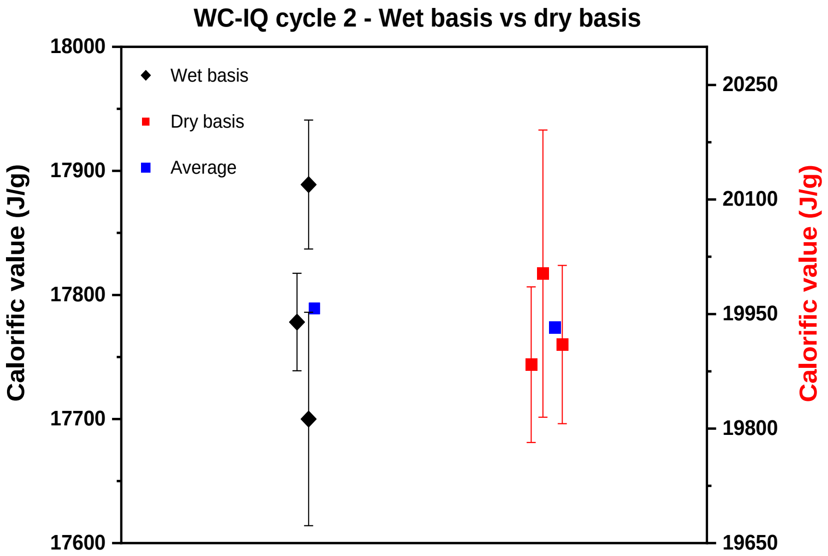
Results – Wood chips Industrial quality (WC-IQ)



ISO 18125:2017



Improved practice

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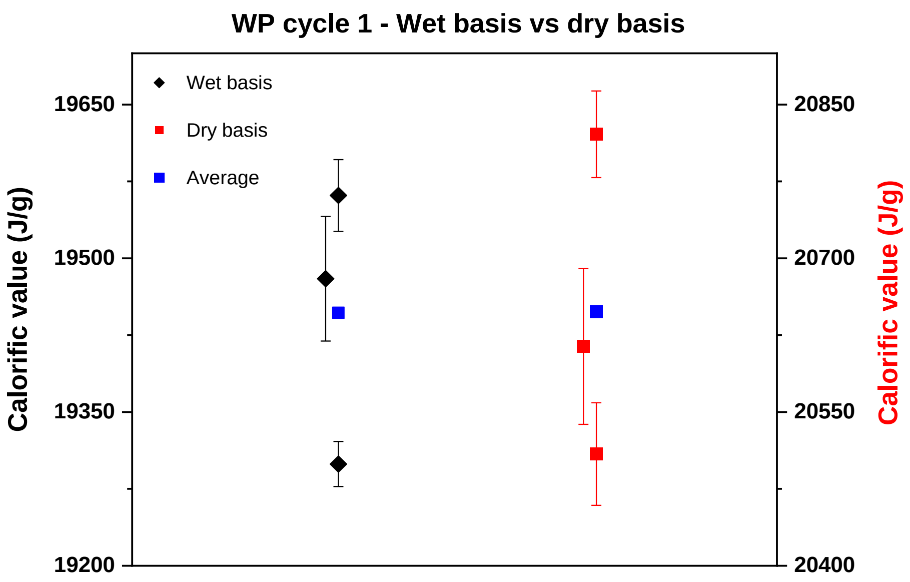
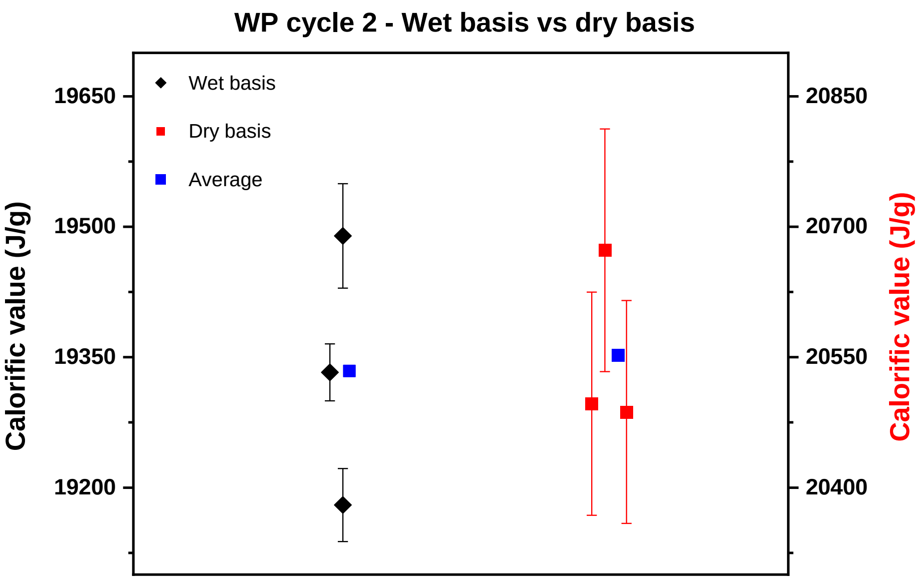
Results – Wood pellet (WP)



ISO 18125:2017



Improved practice

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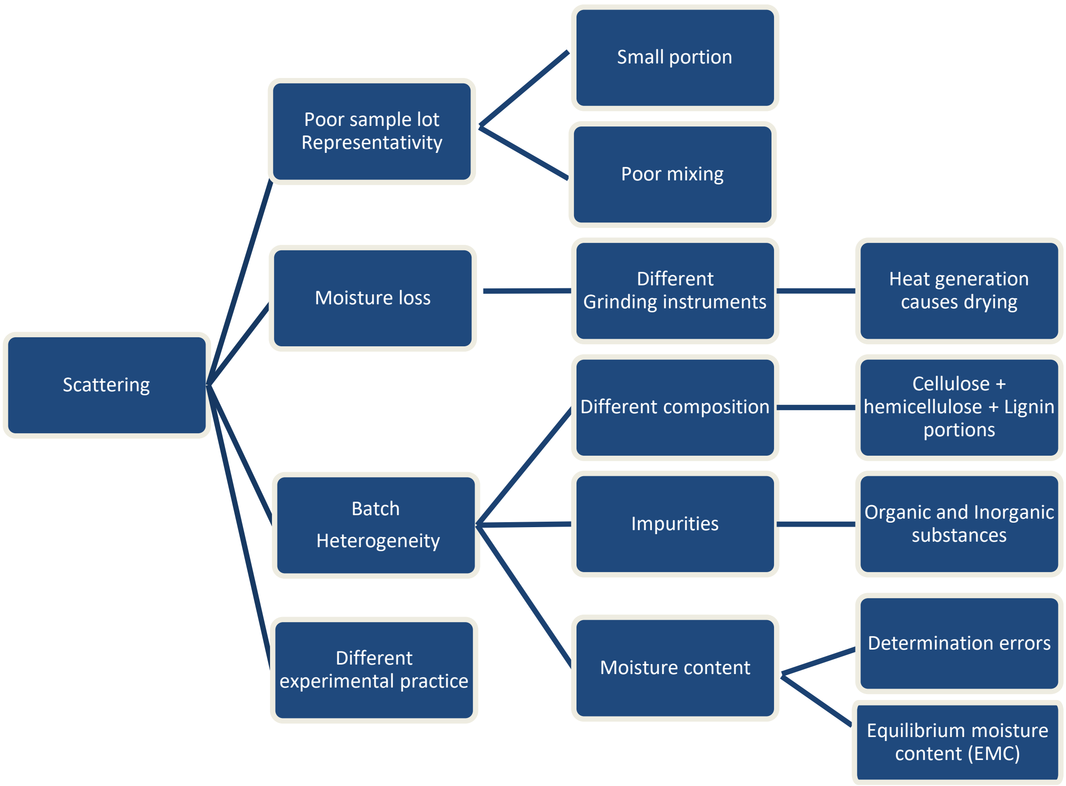
Root cause analysis

TUBITAK provided a well mixed grinded and unified samples



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Noticeable observations - Explosion


- Difficulty in burning 1 g of the sample
- Contribution from different set of crucibles, oxygen pressures, bomb sizes, ignition wires, oxygen flushing, sample mass were tested
- Lowering the sample mass for complete combustion

Why not 1 gram?
The applied pressure to form the pellet (pelletizer)

Over pressurized pellet will develop segments/breaking points

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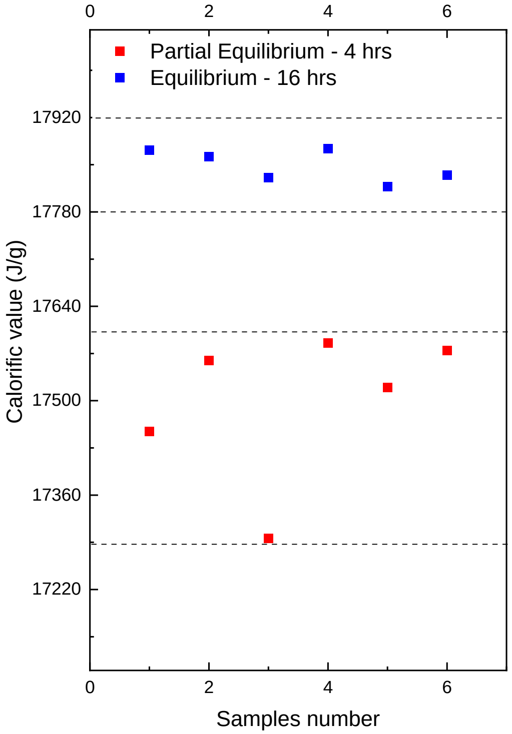
Equilibrium moisture content (EMC)

EMC Reached	Time (h)	Original Mass: 1.1914 (g)	Moisture Loss (%)
90.5%	2:30	1.1170	6.24
92.1%	4	1.1154	6.38
93.2%	5:30	1.1142	6.48
≈99%	16	1.1078	7.01

- Less scattering with longer equilibrium time

- Significant improvement in the repeatability up to 50 – 80 %

- The conditions of equilibrium depends on each laboratory's atmosphere



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Improved criteria

Parameter	BIOFMET	ISO 18125-2017
Mass of sample (g)	0.3 – 0.7 (≈ **0.5**)	0.8 – 1.2 (≈ **1**)
Pellet-applied pressure (t)	**0.5** – 3 t	None
Uncertainty budget	Detailed ≈ 1%	None
Ignition corrections	With flushing 2 – 8 J Without flushing 20–40 J	None
Grinder RPM	Up to 3400 rpm	None
EMC	Criteria provided – 16 hrs	4 hrs
Improvements	50-80% in repeatability 15-30% in the final uncertainty	None

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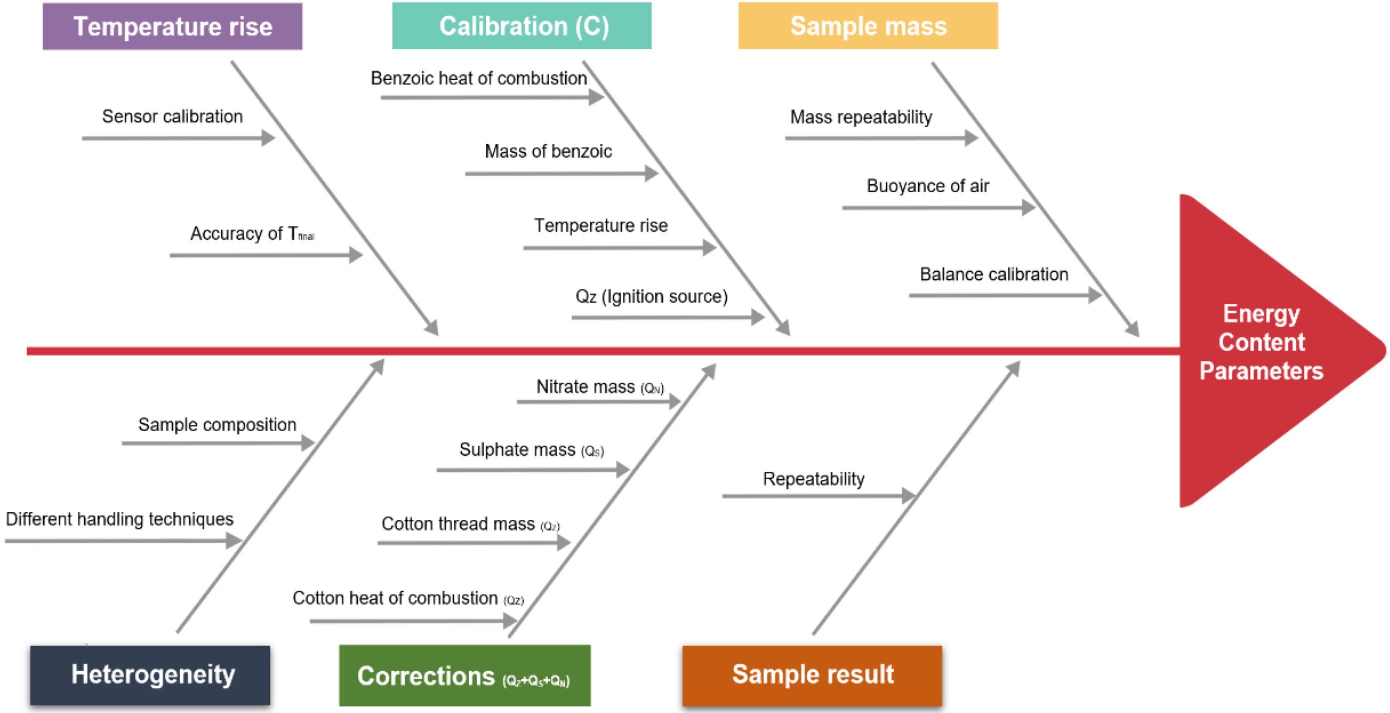
Uncertainty sources



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Unified uncertainty

The coal comparison and the results of cycle 1 from the BIOFMET project proved the need for a unified uncertainty budget. The uncertainty of cycle 2 shows a significant consistency thanks to the unified practice.

	Coal Type	Coal uncertainty %		Sample Type	Cycle 1 Uncertainty %	Cycle 2 Uncertainty %	Combined uncertainty %
COOMET Project - Coal key comparison	**AL-RU**	0.13	BIOFMET project	**WC-HQ**	0.76	0.38	**0.85**
	AH-RU	0.14		**WC-IQ**	0.71	0.38	**0.51**
	LC-RU	0.14		**WP**	0.39	0.34	**0.63**
	AL-RU	0.02		**WC-HQ**	0.254	0.35	**0.82**
	AH-RU	0.02		**WC-IQ**	0.29	0.39	**0.52**
	LC-RU	0.02		**WP**	0.24	0.34	**0.63**
	AL-RU	0.48		**WC-HQ**	0.68	0.43	**0.85**
	AH-RU	0.41		**WC-IQ**	4	1.28	**1.33**
	LC-RU	0.42		**WP**	0.67	0.43	**0.67**

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A comprehensive Analysis of the Risks Associated with the Determination of Biofuels' Calorific Value by Bomb Calorimetry

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ACCEPTED

Improved metrological methodology to address the challenges associated with the determination of biofuels calorific value by bomb calorimeter

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Thank you!

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