

Rapid and automated determination of EC and OC in Air Filter samples

Jess Gantt



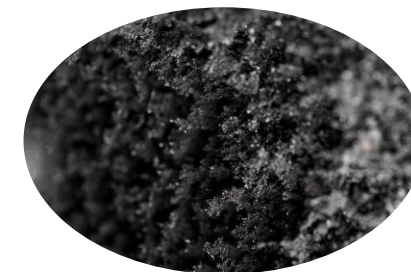
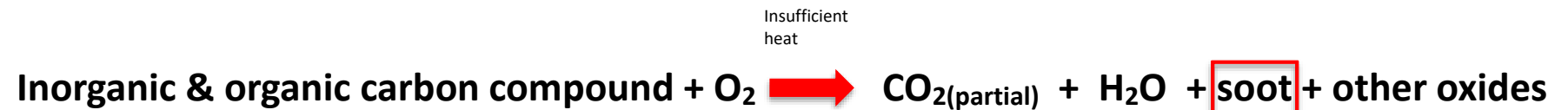
Soot formation

- Soot is a mixture of elemental carbon and higher condensed organic hydrocarbons

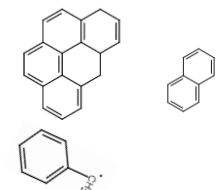
Complete
Combustion



Incomplete
Combustion

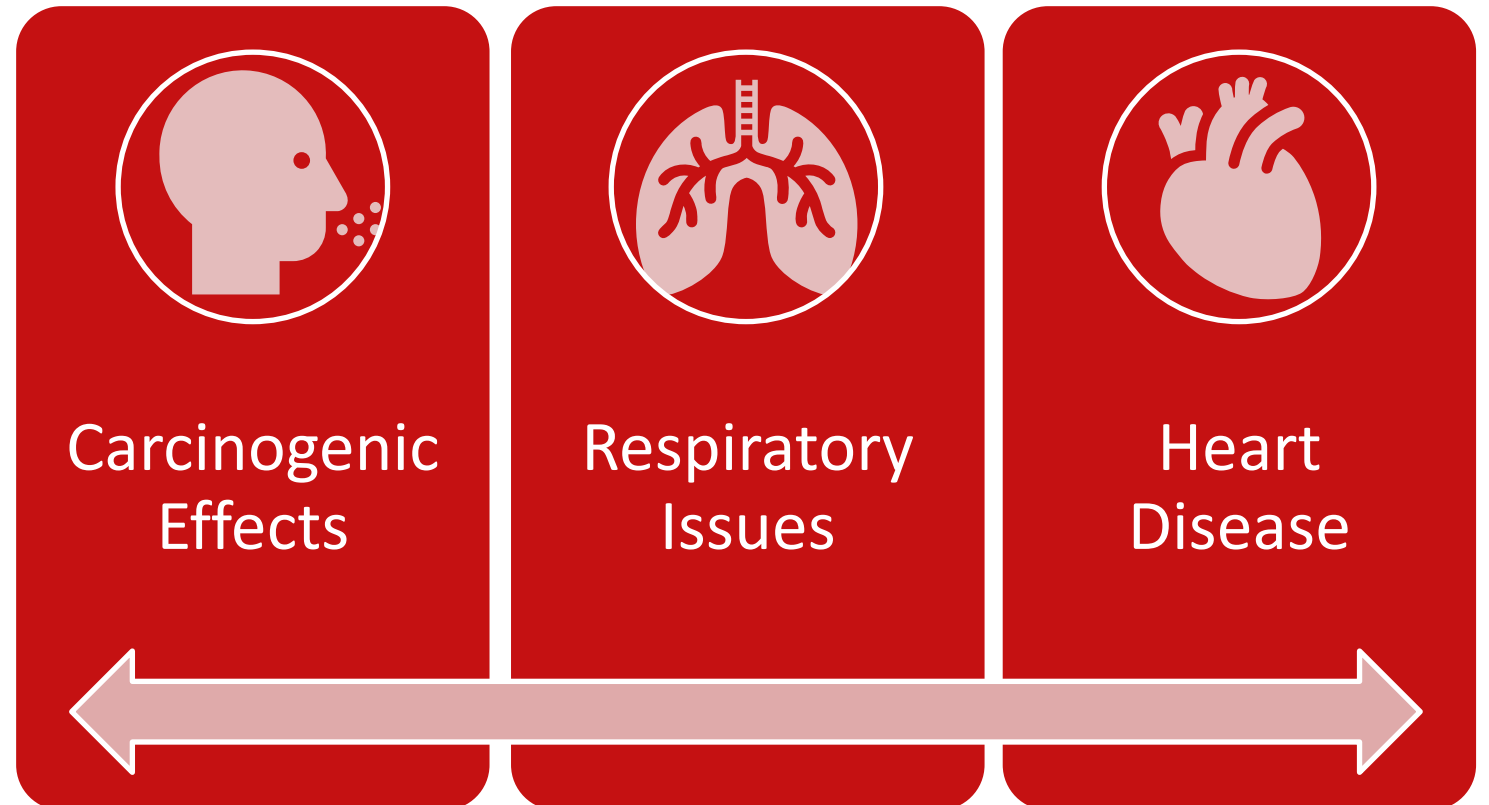


[1]



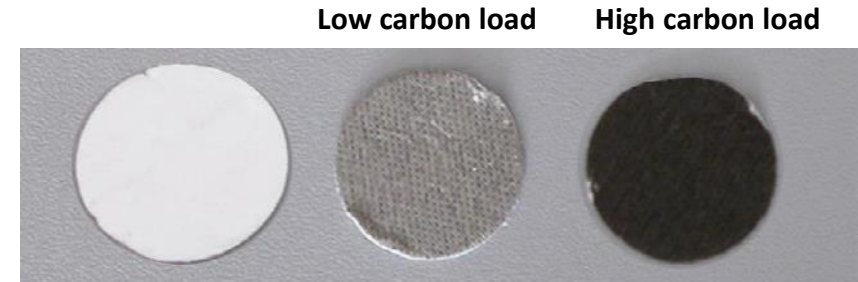
Health Effects OF Carbonaceous Aerosols

- Carbonaceous Aerosols linked to long-term health issues and present a public safety risk
- Air pollution caused by soot is a growing issue
- As of January 2023, EPA has been discussing additional regulations to enact a lower LOD for soot in air [2]



Challenges in Routine Analysis

- Wide concentration Range
- High throughput sampling needs for larger labs
- Prevent pyrolysis of organic samples in early stages of analysis
- Multiple operators on a single instrument
- Ensuring Sequential measurement of EC/OC



Solution

Self-Check System

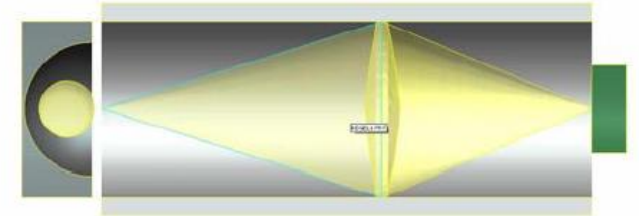
Boat Sensor

Auto-protection Valve

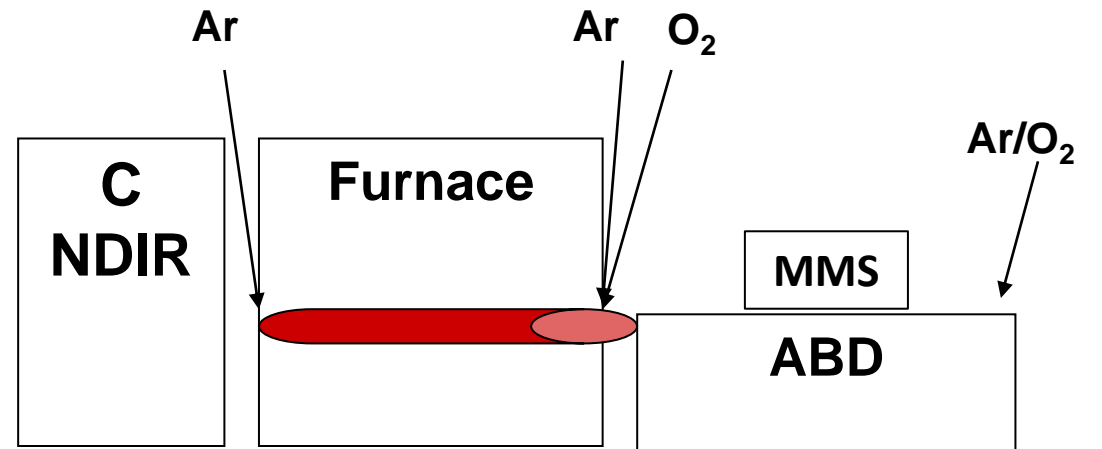
Flame Sensor Technology

Wide Range NDIR

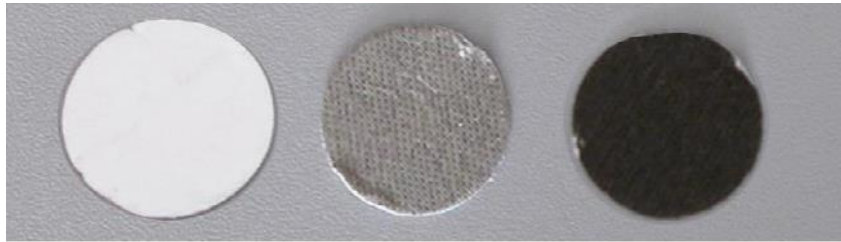
Fully Automated Carbon analysis



Instrument Configuration



Sample Supply



Multi Matrix Sampler(MMS) and Automatic Boat Drive

- Fully automated sampling system to ensure sample makes it into furnace
- High-through put analysis with 35 sample tray
- Boats with downholder to prevent loss of material

Thermal Desorption/ Combustion Digestion

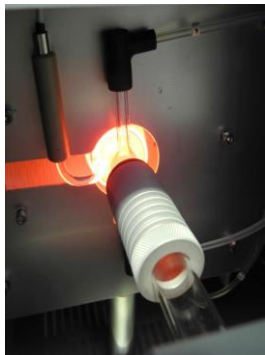
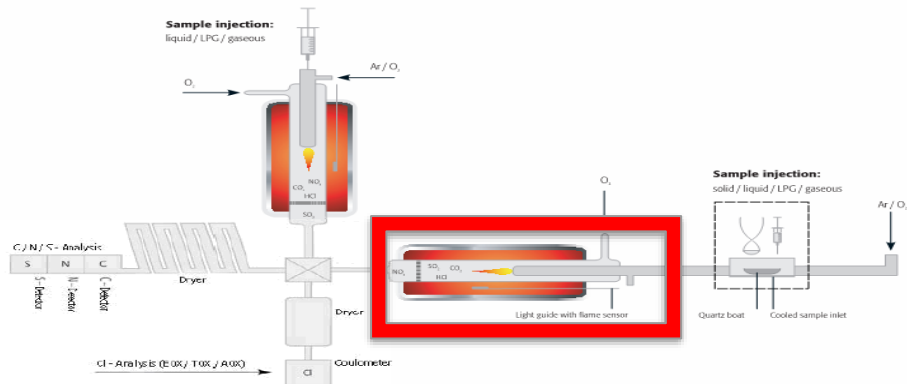
Sample Supply

**TD/
Combustion
Digestion**

Purification

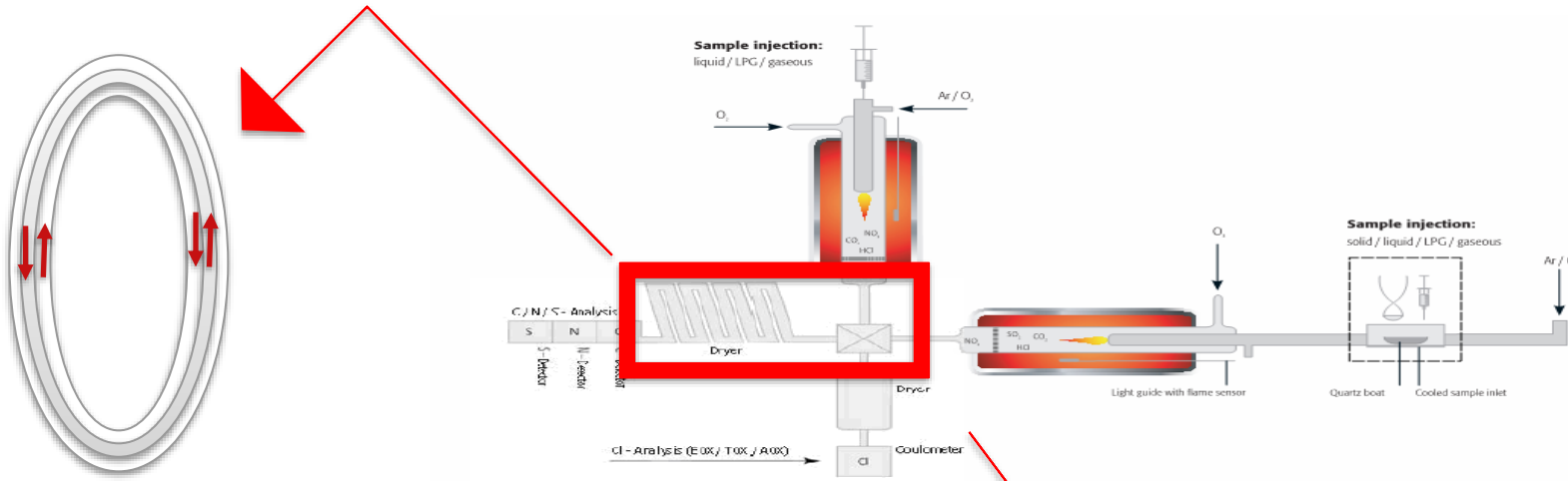
Detection

Evaluation

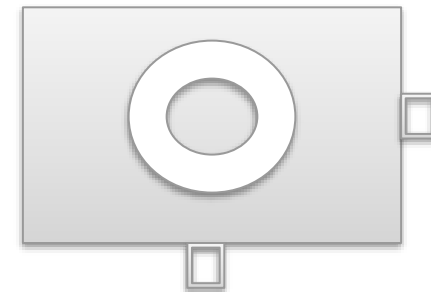


Parameter	Specification
Furnace Temp	1000°C
Thermodesorption Temp	750°C
Purge Time	60 s
Ar flow(first phase)	200 mL/min
O2 main flow	200 mL/min
O2 flow (Second Phase)	200 mL/min

Purification

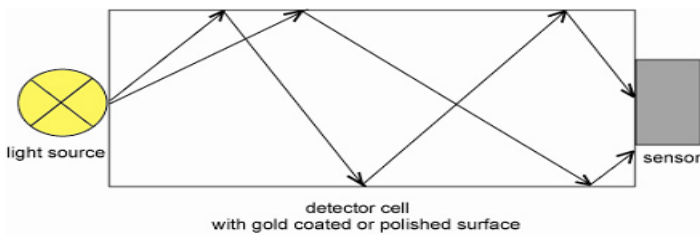


Internal Membrane dryer purifies sample using alternating gas streams



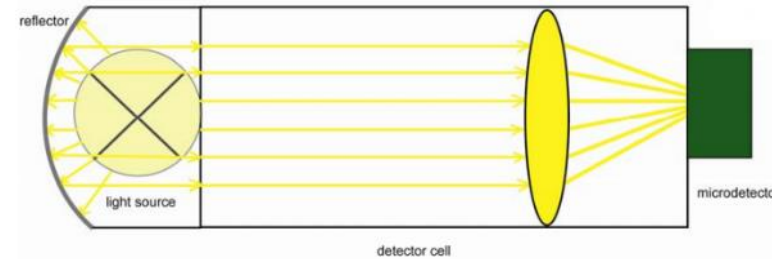
Auto-Protection valve allows for the filtering and drying of gas

Focus Radiation NDIR Detection



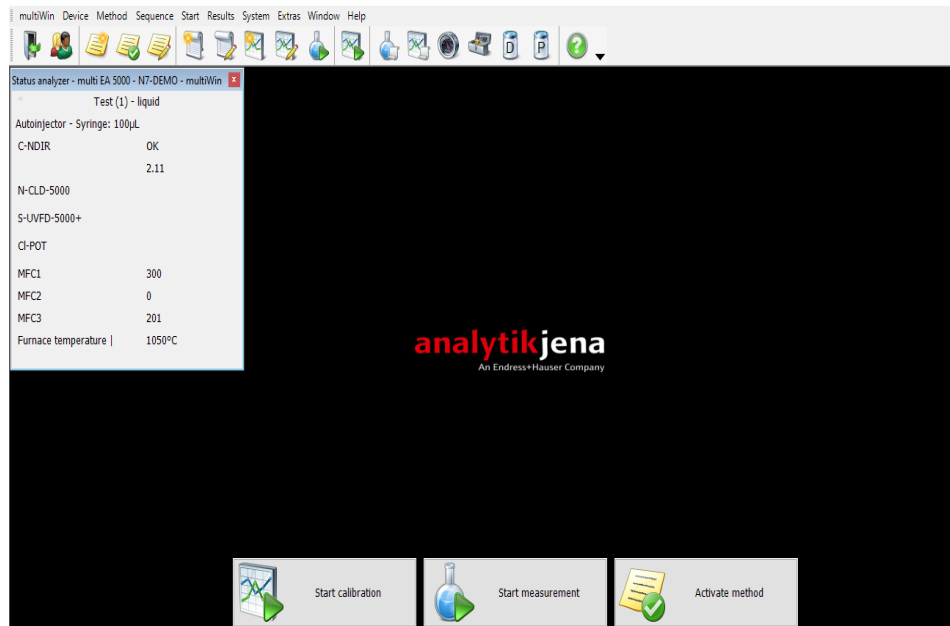
Common NDIR detector

- Reflection on cell wall
- Corrosive gases effect wall surface
- Loss of sensitivity and energy
- Cleaning or exchange of cell necessary
- Higher downtime and maintenance cost



Focus Radiation NDIR detector

- No light reflection
- No corrosion
- No maintenance
- Real wide-range detector: 0 – 30,000 mg/L without dilution



Software

- Method Library with time optimized analysis parameters
- Continuous monitoring of instrument State and system stability
- Automatic identification of Correct Components and Accessories
- Monitoring of changes made in the system

Combustion Parameters for Multi EA 5100

Parameter	Specification
Furnace Temp	1000°C
Thermodesorption Temp	750°C
Purge Time	60 s
Ar flow(first phase)	200 mL/min
O2 main flow	200 mL/min
O2 flow (Second Phase)	200 mL/min

Table 1: Process parameters multi EA 5100 in horizontal mode

Detection Parameters for EC/OC with NDIR detection

Parameter	Specification OC	Specification EC
Max Integration Time	600 s	180 s
Start	0.12 ppm	0.12 ppm
Stability	3	3
Threshold	5 ppm	0 ppm

Table 2: Detection parameters multi EA 5100 in horizontal mode

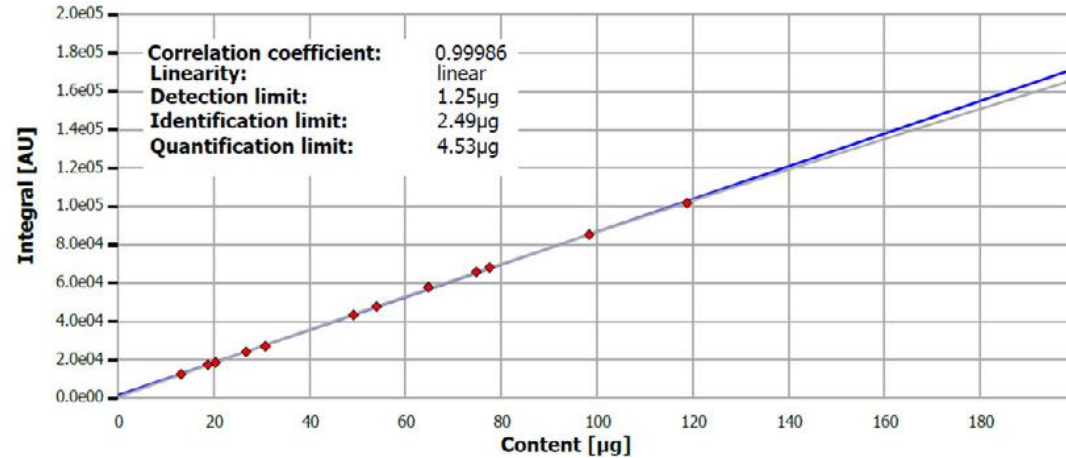


Figure 2: Example of an EC calibration for the trace range, curve including statistics

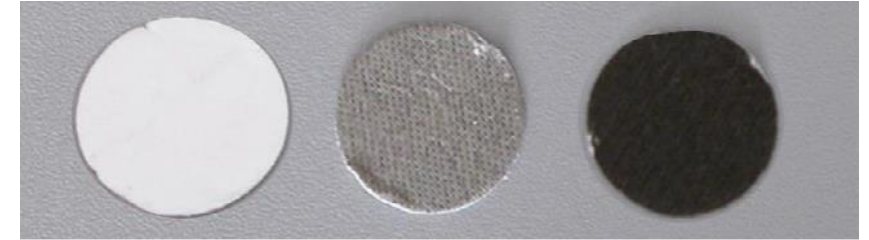
- Calibration Standards were made of soot standard of a known µg content.
- Different quantities(5-100 mg) of the EC standards were used and placed onto a quartz powder material
- Calibration shows good linearity ($r^2 = 0.999$)



Utilizing the Easy Cal and Focus Radiation NDIR features, it became possible to create a simple calibration with a large working range

Results- Samples

- Homogeneous samples were collected on quartz filters(d=15 mm)
- Filters were then cut into fourths for better desorption
- Filters were placed into boats with downholders



Measurement	EC	OC
Filter 1	875 µg	656 µg
Filter 2	124 µg	118 µg
Filter 3	32.8 µg	112.1 µg
Test standard EC= 1.01 g/kg	1.0 g/kg	

Table 3 showcases the results of 3 filters prepared using the steps above, post thermodesorption and combustion. QC Recovery was within 1% of expected value

Table 3: Results of 3

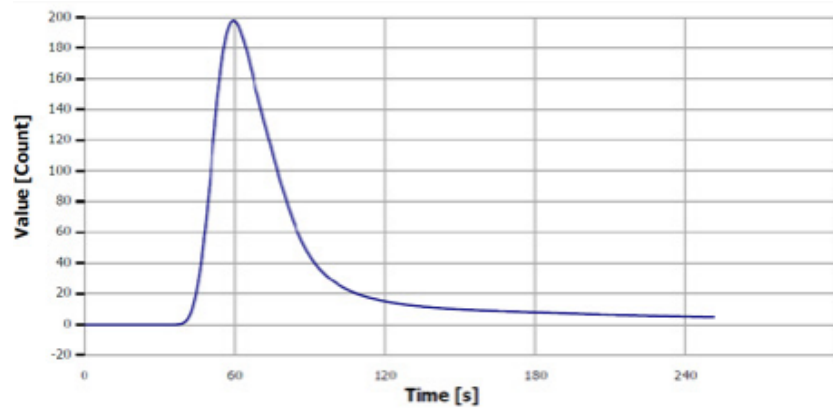


Figure 4: OC analysis curve of "filter 1"

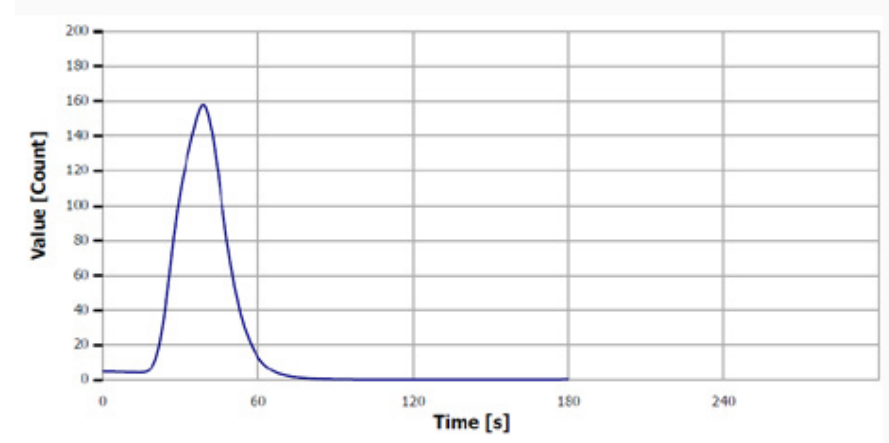


Figure 5: EC analysis curve of "filter 1"

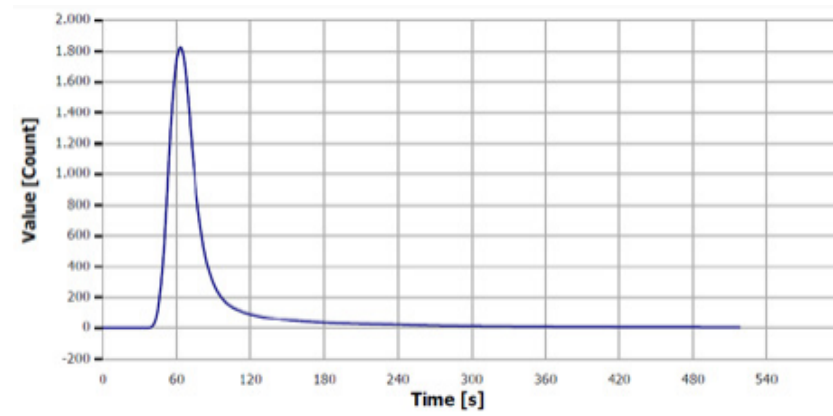


Figure 6: OC analysis curve of "filter 2"

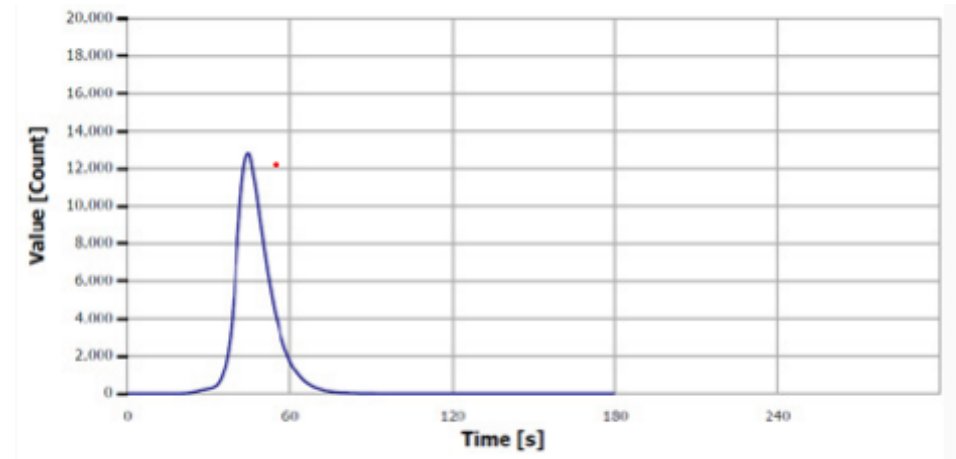
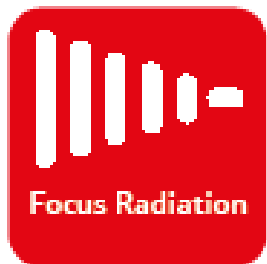


Figure 7: EC analysis curve of "filter 2"

Conclusion

- Multi EA 5100 horizontal configuration allows for automated high-throughput analysis
- Pre-configured method library allows for selection of optimized EC/OC parameters
- Self Check System makes it possible to ensure a closed, leak free environment for combustion
- Strong linear calibration was achieved due to easy calibration functionality
- Test of a known QC showcased recoveries within 1% of expected value, validating the method and calibration





Thank you for your attention!

References

1) *What are the different types of coal?*. American Geosciences Institute. (2019, January 4).
<https://www.americangeosciences.org/critical-issues/faq/what-are-the-different-types-of-coal>

2) Environmental Protection Agency. (n.d.). *EPA Proposes to Strengthen Air Quality Standards to Protect the Public from Harmful Effects of Soot*. EPA. <https://www.epa.gov/newsreleases/epa-proposes-strengthen-air-quality-standards-protect-public-harmful-effects-soot>