



# ASTM D874 Sulfated Ash Analysis Using CEM Microwave System (Phoenix Black)

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# ASTM D874 Analysis With CEM Microwave System



During Q4 2023 a set of four D874 TMC reference oils were released by the D874 SP to evaluate D874 results generated by the CEM Phoenix Black Microwave Combustion System.

- Instead of open burning of the sample on a hotplate or gas burner, multi-stage heating in a microwave oven is used for initial combustion.
- After acid treatment the sample is cycled in the microwave oven set at 775°C, cooled and weighed until a constant weight is obtained.

# Why Investigate Microwave Assisted Combustion?



Open combustion is a potentially hazardous operation and requires constant attention to prevent overheating and out-of-control combustion.

Several related safety incidents at Lubrizol have made finding an alternate combustion method a priority.

Microwave combustion is automated, and the test operator may attend to other duties during sample combustion.

Testing was performed by CEM in their Applications Lab:

Lab Seq	RO ID	%Ash	Result	Status	Crucible	Note
LZ199	820-2	1.42	Pass	Op Invalid	Porcelain	Spatter
LZ199	820-2	1.46	Pass	Valid	QFC	Low end of acceptable range
LZ200	90	0.62	Fail	Op Invalid	Porcelain	Spatter
LZ200	90	1.07	Pass	Valid	QFC	
LZ200	90	1.03	Pass	Valid	Porcelain	7g S.W. Lower than optimal S.W.
LZ201	91	0.72	Fail	Op Invalid	Porcelain	Spatter
LZ201	91	0.88	Pass	Valid	QFC	
LZ201	91	0.86	Pass	Op Invalid	QFC	5g S.W. Below required S.W.
LZ202	92	0.85	?	Op Invalid	Porcelain	Spatter
LZ202	92	1.09	?	Valid	QFC	
LZ202	92	1.14	?	Valid	QFC	5g S.W. Lower than optimal S.W.

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LZ201	91	0.88	Pass	Valid	QFC	
LZ202	92	1.09	?	Valid	QFC	
LZ202	92	1.14	?	Valid	QFC	5g S.W. Lower Than Optimal S.W.

Passing results were obtained on all oils though sample spattering caused multiple operationally invalid results when using porcelain crucibles.

- Optimization of the heating program is expected to prevent spattering in the future.
- Samples analyzed in Quartz Fiber Crucibles (QFC) did not exhibit spattering.
- Fused Silica Crucibles were not evaluated.
- Platinum Crucibles are not compatible with microwave heating.

# Next Steps



- Optimize combustion conditions for Porcelain and Fused Silica crucibles to prevent sample spattering and repeat testing.
- Investigate properties of QFC crucibles for potential inclusion into D874 test method.
- Along with the microwave study, the D874 SP released TMC Reference Oils to run ASTM E2403: Standard Test Method for Sulfated Ash of Organic Materials by Thermogravimetry. This work is planned to be completed during Q2 2024.