

# **Method Development and Validation for Microscopic Measurement of the Fluorescence Color of Sedimentary Organic Matter in Geologic Samples**

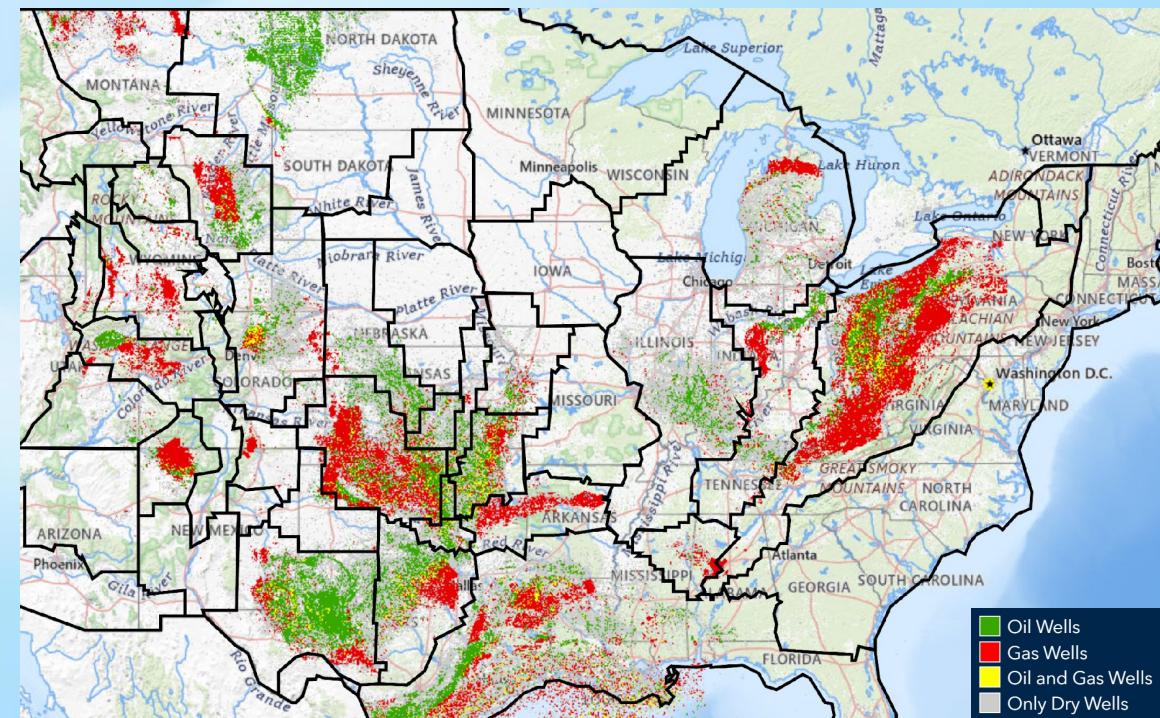
Kavin Siaw

Dr. Paul C. Hackley

This research is supported by funding provided by the American Institute of Physics (AIP).

# BACKGROUND

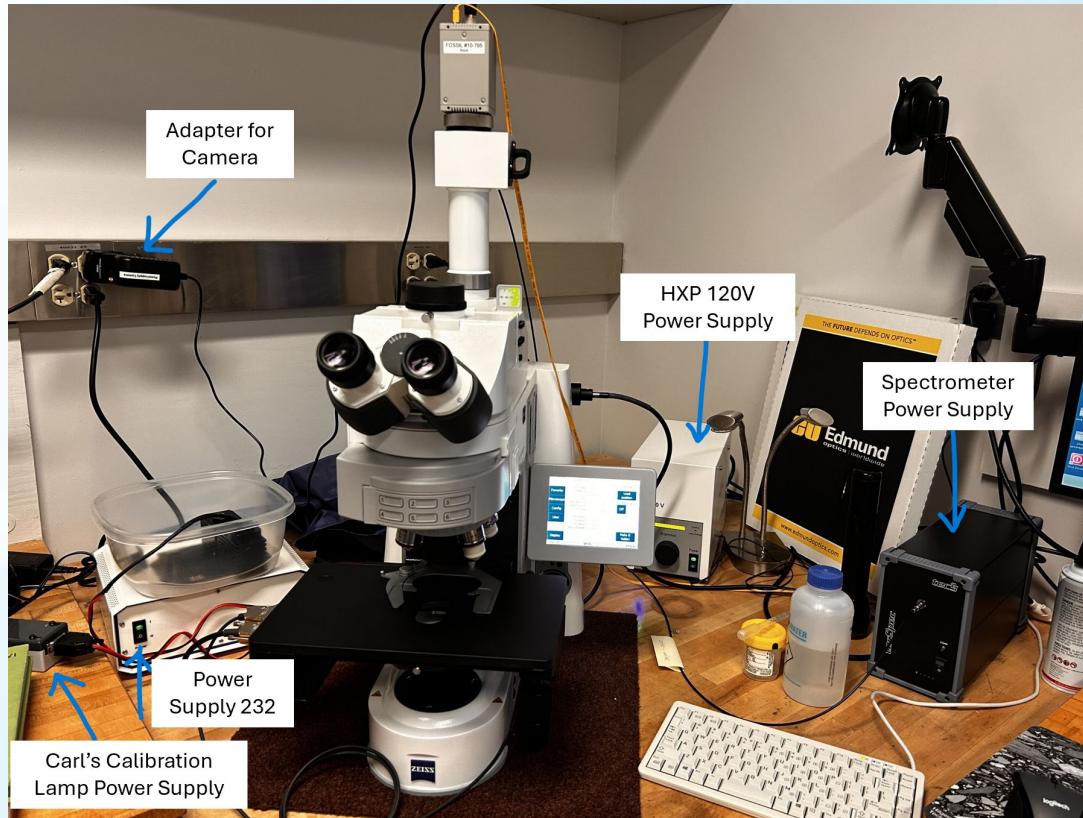
- The U.S. Geological Survey (USGS) provide periodic assessments of the oil and natural gas endowment of the United States and globally.
- The Energy Resources Program (ERP) evaluates the estimated volumes and extents of undiscovered oil and gas resources in priority geologic provinces.



Credit: <https://certmapper.cr.usgs.gov/data/apps/noga-drupal/>

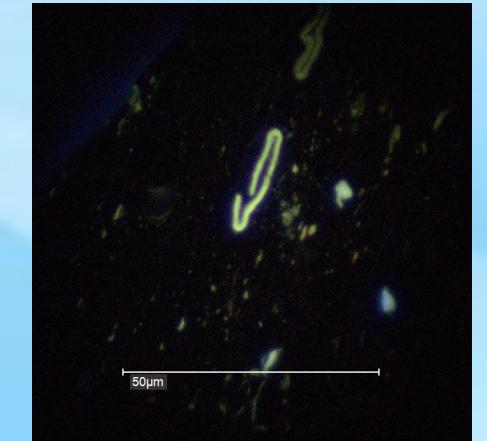
# OBJECTIVE

The fluorescence color of sedimentary organic matter is a proxy for thermal maturity of rocks in sedimentary basins and can be used to understand where rocks are immature or mature for making oil and gas.



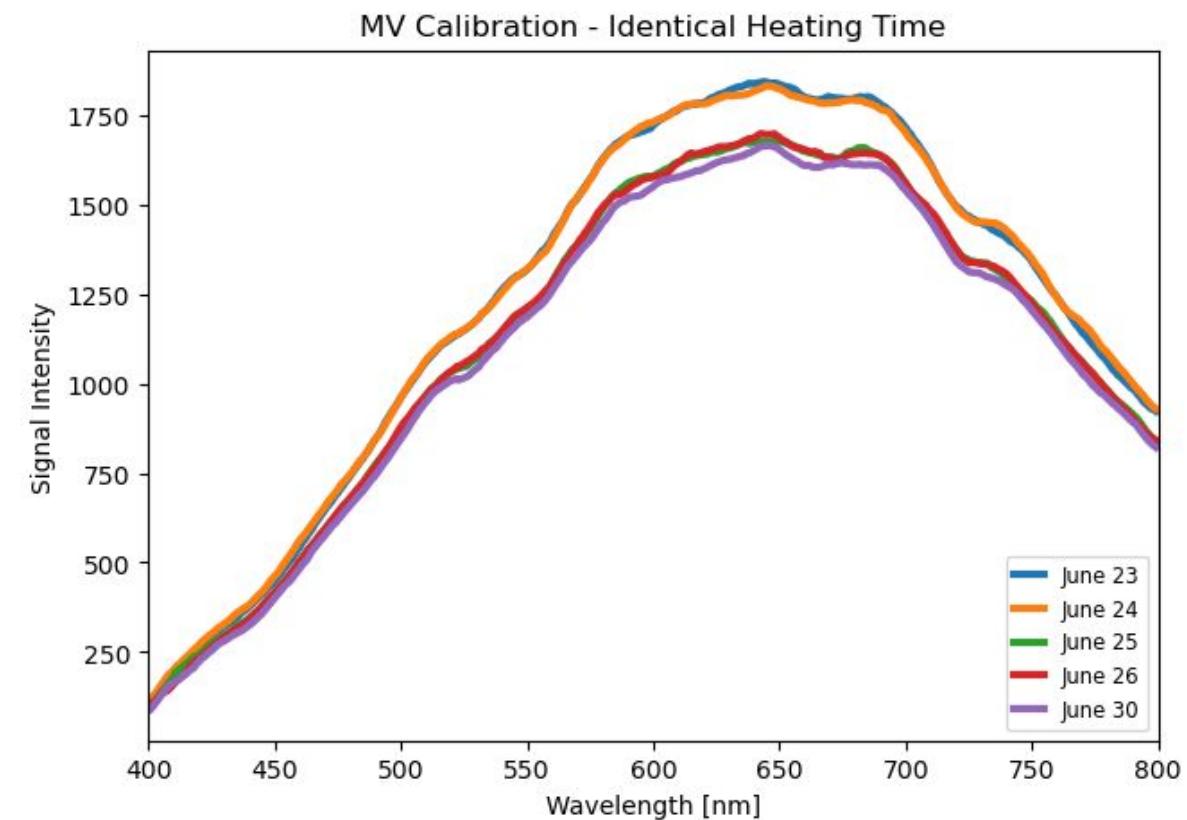
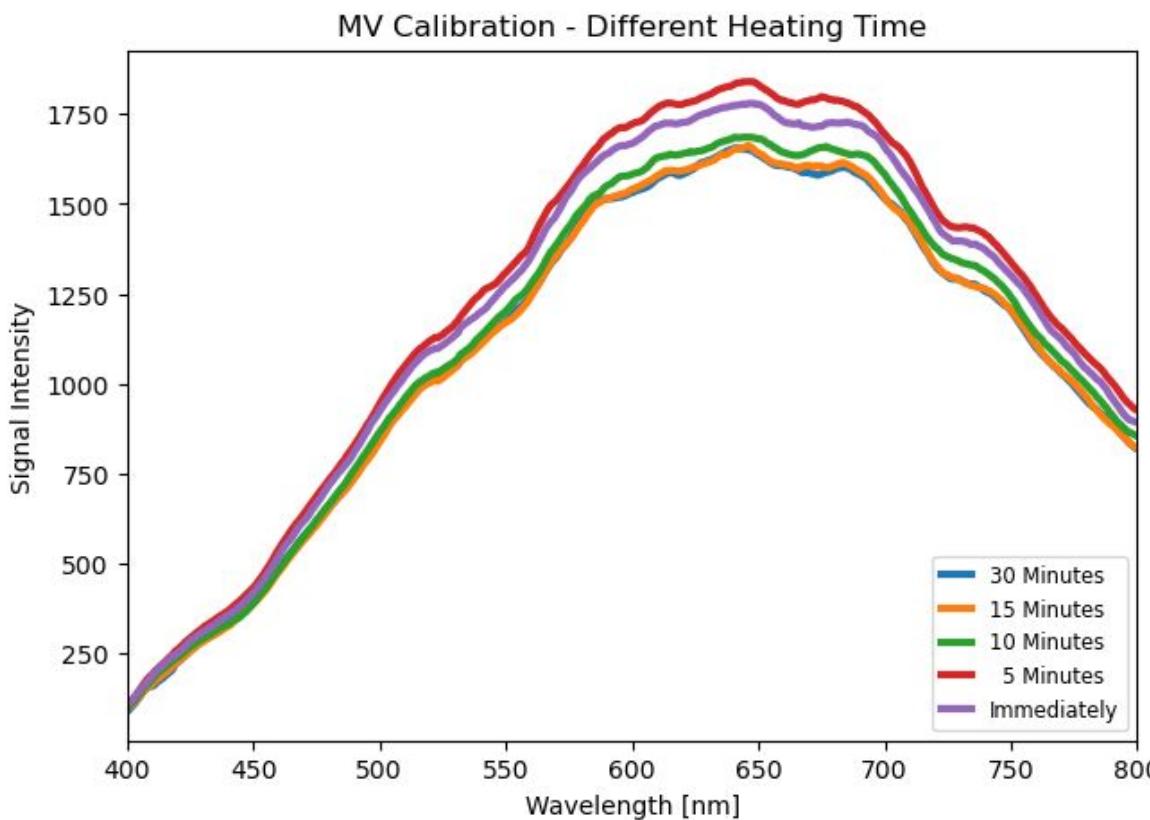
Develop and validate a standardized test method for evaluating the fluorescence color of sedimentary organic matter.

- Instrument Preparation Time
- Calibration Conditions
- Number of Measurements



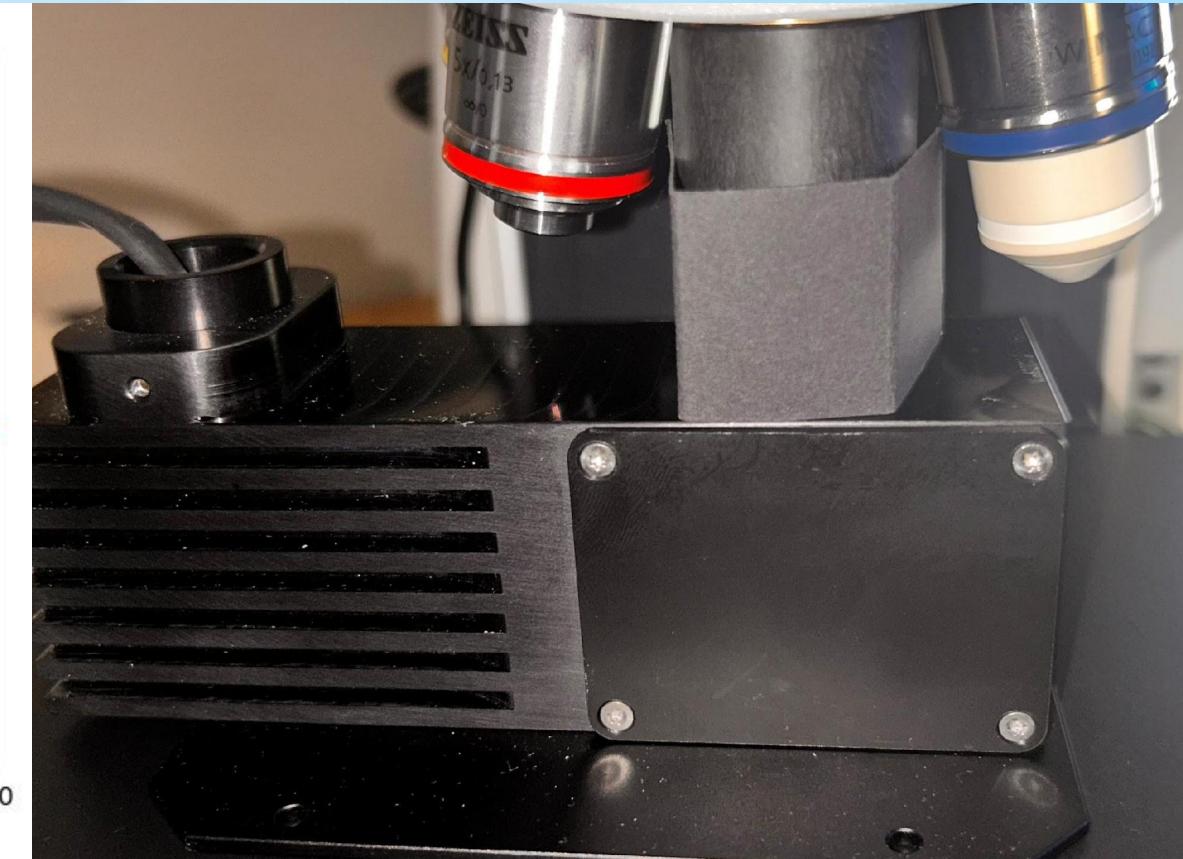
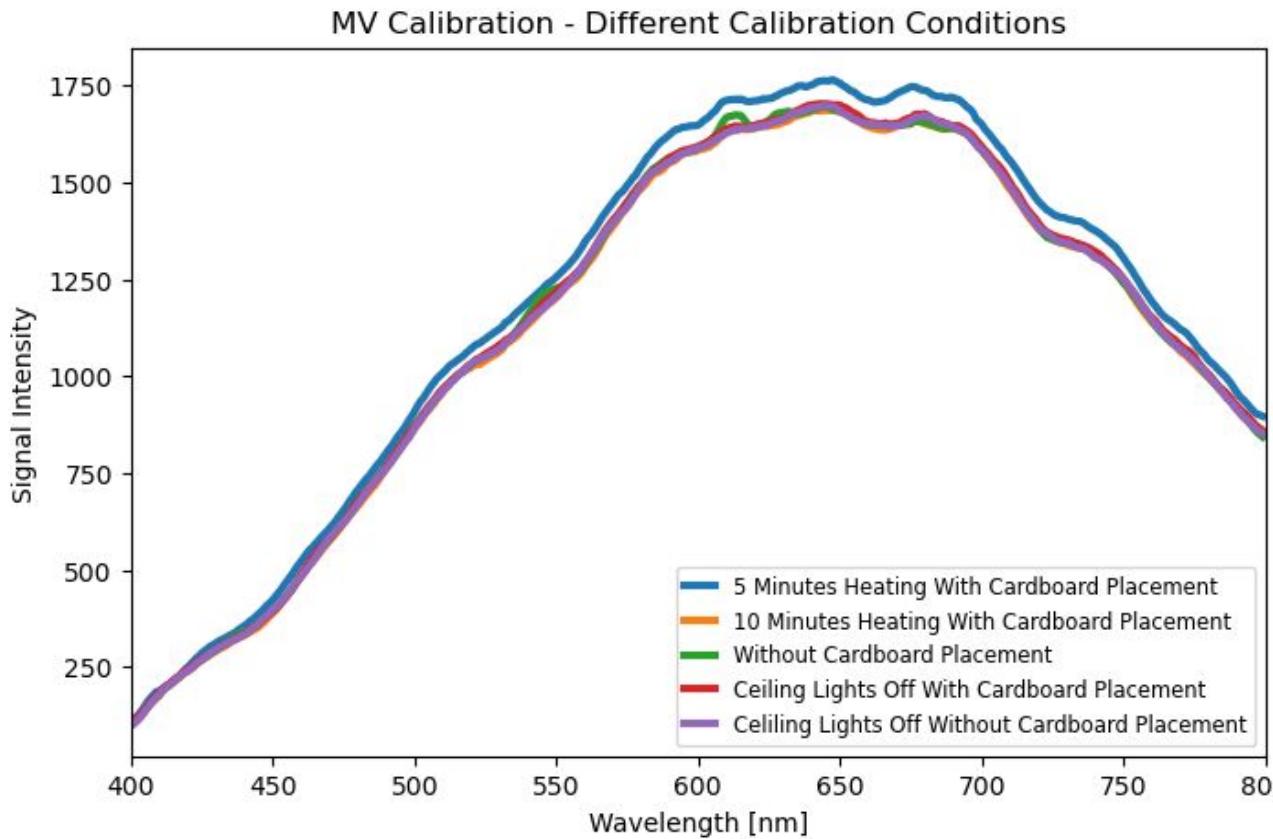
# INSTRUMENT PREPARATION TIME

The study evaluated the effect of various heating durations for the calibration lamp (i.e., 0, 5, 10, 15, and 30 minutes) prior to performing calibration measurements.



The heating time alone does not have a direct effect on the calibration quality for the fluorescence microscope. As suggested by the manual, a five-minutes heating period is recommended as a standard protocol.

# CALIBRATION CONDITIONS



Calibration and data collection should be performed when ceiling lights turned off.

# NUMBER OF MEASUREMENTS

A use of coefficient of variation (CV) to assist the determination of the best number of measurement as defined by Everitt (1998).

$$CV = \frac{\sigma}{\mu}$$

Where  $\sigma$  and  $\mu$  are standard deviation and mean, respectively.

Coefficient of Variation		Definition	
< 0.1		Very low variability (high stability)	
0.1 – 0.2		Low variability	
0.2 – 0.3		Moderate variability	
0.3 – 0.5		High variability	
> 0.5		Very high variability (unstable)	

Measurements	$\lambda_{max}$ mean	$\lambda_{max}$ std	R/G mean	R/G std	Measurements	$\lambda_{max}$ mean	$\lambda_{max}$ std	R/G mean	R/G std	
5	0.093	0.061	0.165	0.118	5	0.046	0.023	0.086	0.051	
10	0.053	0.053	0.181	0.110	10	0.071	0.011	0.104	0.047	
15	0.093	0.053	0.285	0.331	15	0.055	0.011	0.100	0.044	
20	0.101	0.044	1.149	2.483	20	0.056	0.032	0.110	0.041	
<b>A</b>	25	0.105	0.049	-0.422	1.623	<b>B</b>	0.059	0.025	0.133	0.048
Measurements	$\lambda_{max}$ mean	$\lambda_{max}$ std	R/G mean	R/G std	Measurements	$\lambda_{max}$ mean	$\lambda_{max}$ std	R/G mean	R/G std	
5	0.004	0.003	0.045	0.021	5	0.045	0.036	0.025	0.013	
10	0.010	0.004	0.061	0.028	10	0.087	0.038	0.032	0.011	
<b>C</b>	15	0.012	0.004	0.059	0.022	<b>D</b>	0.057	0.038	0.045	0.030

10 measurements are recommended as the ideal number because of the relatively low standard deviation and CV values remaining within 0.1.

# OUTCOME

A standard operating procedure and method development paper are in preparation based on the study on the spectral fluorescence microscope with different analysis to standardize the method on measuring organic material.

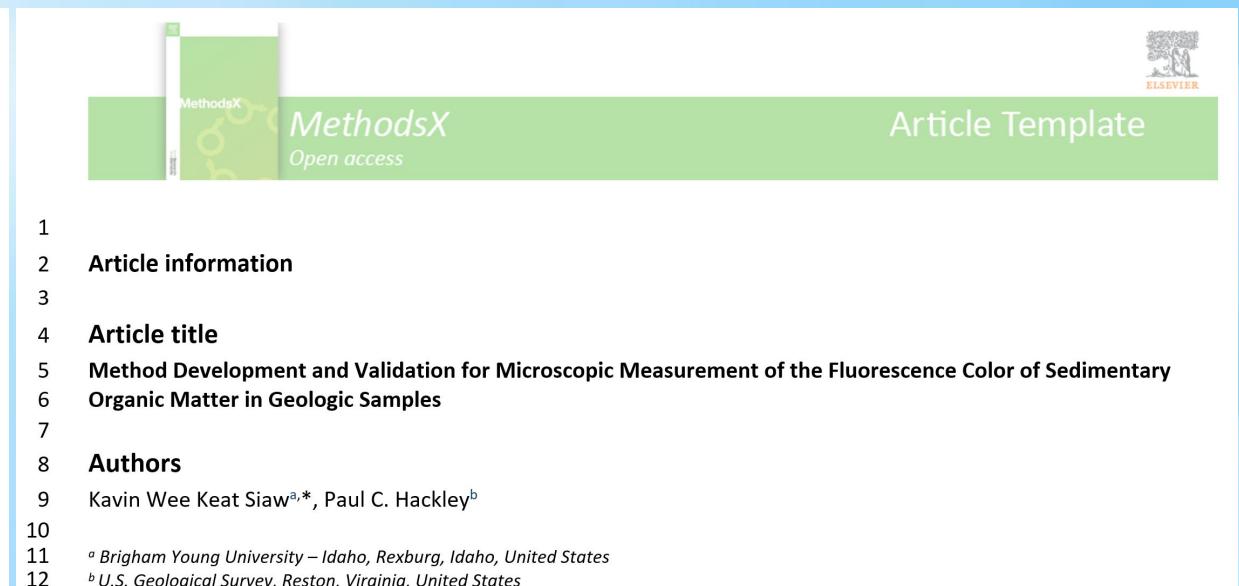


U.S. Geological Survey  
Energy and Mineral Resources Mission Area  
Quality Management System

Technical Standard Operating Procedure  
[PGM-LAB-SOP-XX.YY](#)

**SPECTRAL FLUORESCENCE MEASUREMENT WITH ZEISS  
AXIOLMAGER FOSSIL SYSTEM**

USGS Standard Operating Procedure Drafted – Peer Review  
in process.



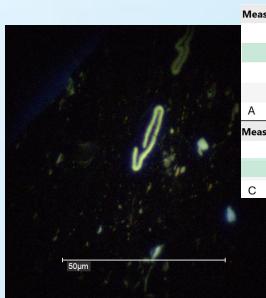
The image shows a digital interface for a MethodsX article template. At the top, there is a logo for Elsevier and the text "Article Template". Below this, there is a sidebar with a green background and white text, listing steps 1 through 12. The main content area has a white background and displays the article information, title, authors, and their institutions.

1  
2 **Article information**  
3  
4 **Article title**  
5 **Method Development and Validation for Microscopic Measurement of the Fluorescence Color of Sedimentary**  
6 **Organic Matter in Geologic Samples**  
7  
8 **Authors**  
9 Kavin Wee Keat Siaw<sup>a,\*</sup>, Paul C. Hackley<sup>b</sup>  
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11 <sup>a</sup> Brigham Young University – Idaho, Rexburg, Idaho, United States  
12 <sup>b</sup> U.S. Geological Survey, Reston, Virginia, United States

MethodsX paper rough draft initiated for peer review in the future for publication.

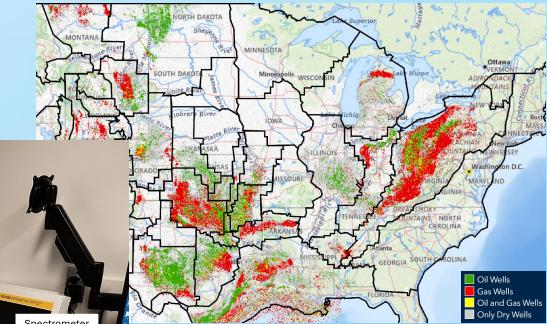
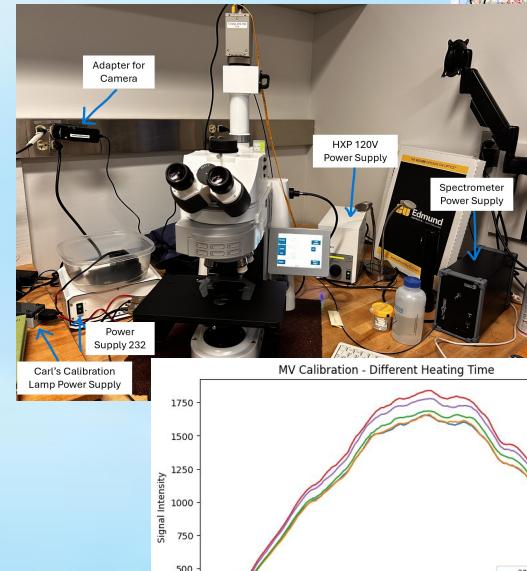
# SUMMARY

- The USGS provide periodic assessments of the oil and natural gas endowment of the United States and globally.
- Develop and validate a standardized test method for evaluating the fluorescence color of sedimentary organic matter.
- Recommended procedures:
  - Instrument warm up for 5 minutes
  - Dark current collection once per month (30 days)
  - Collect 10 measurements for each sample
  - Interpolation has minimal effect on the analysis but use with cautious
- A SOP and method development is in preparation.

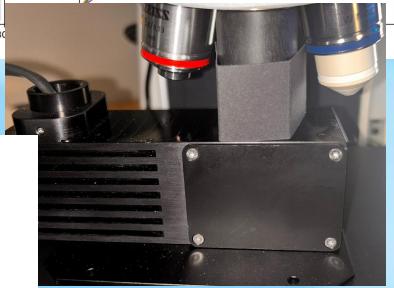
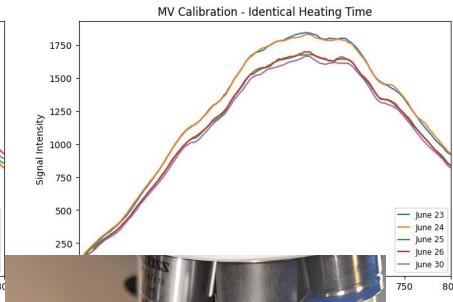
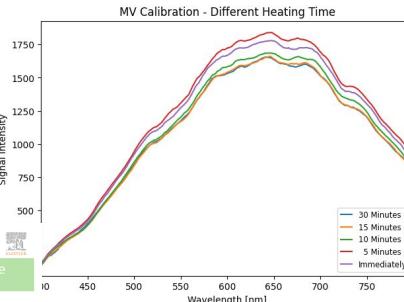


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U.S. Geological Survey  
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Quality Management System

Technical Standard Operating Procedure  
PGM-LAB-SOP-XXYY

SPECTRAL FLUORESCENCE MEASUREMENT WITH ZEISS  
AXIOLMAGER FOSSIL SYSTEM

# REFERENCE

- Hilgers Technisches Buero, 2025. Instrument manual for Fossil spectral reflectance and Fossil spectral fluorescence, 48 p.
- Everitt, Brian (1998). The Cambridge Dictionary of Statistics, Cambridge, UK New York: Cambridge University Press. ISBN 978-0521593465

# DARK CURRENT COLLECTION

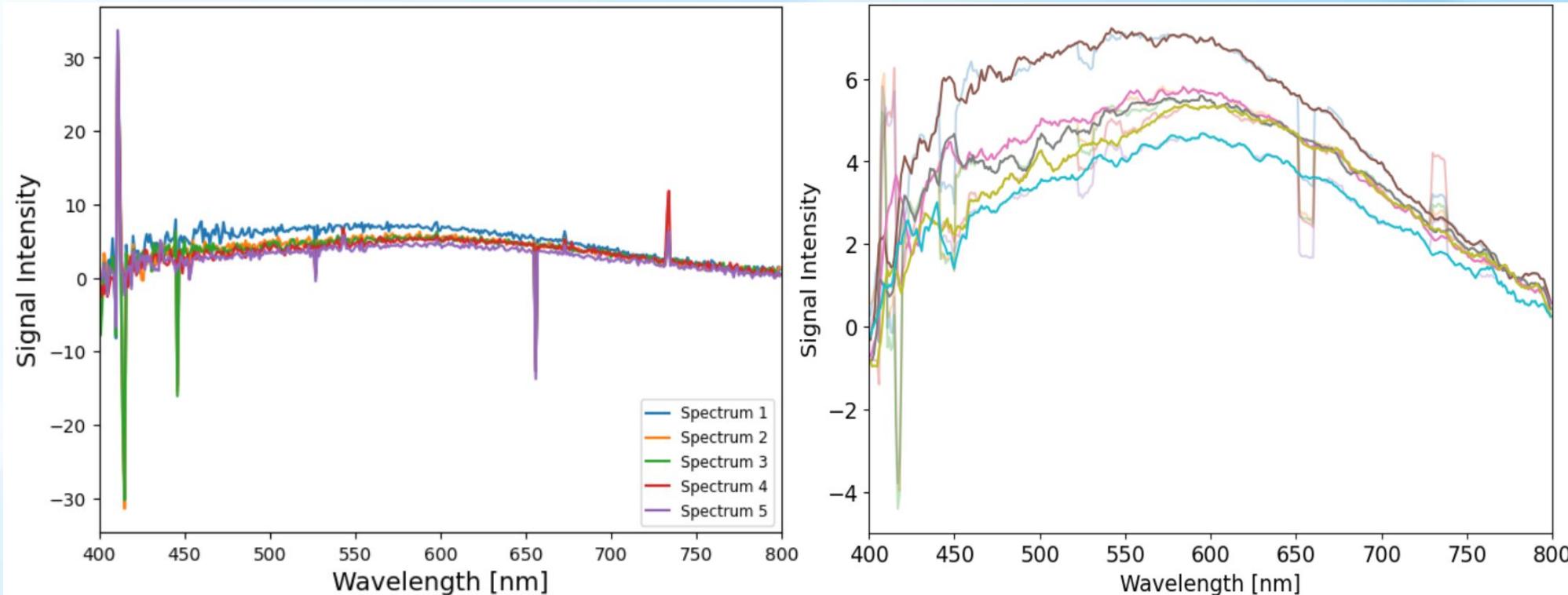
Dark current collection is essential for isolating the inherent electrical signal (or noise) generated by the CCD (Charged Couple Device) camera of the spectrometer in the absence of incident light.

Conditions	B/G Quotient	B/G std	R/G Quotient	R/G std	$\lambda_{max}$	$\lambda_{max std}$
Immediately with Cardboard	2.29	0.01	3.19	0.02	647	1.58
After 10 Minutes Cardboard	2.29	0.02	3.17	0.03	646	2.07
Without Cardboard	2.30	0.02	3.13	0.02	644	2.35
Dark Room Cardboard	2.28	0.03	3.15	0.05	644	2.30
Dark Room Without Cardboard	2.27	0.02	3.15	0.04	644	1.14
15 Minutes with Cardboard	2.27	0.02	3.16	0.04	646	0.55

- Frequent or immediate dark current collection does not significantly influence the calibration spectra.
- As suggested by the manual, dark current collection should be performed once per month (30 days).
- Whenever unexpected spectral behavior is observed, dark current collection should be performed.

# INTERPOLATION

The fluorescence spectra measured from the spectrometer on organic material in both shale and coal samples occasionally produces artifacts characterized by extreme peaks and troughs.



The variation in RGB quotients between pre- and post-interpolated data is minimal, typically within 0.01 – 0.1 range.

Interpolation should only be applied uniformly across all data in a study to maintain comparability. It is not suitable for mixing with raw datasets unless explicitly acknowledged.