100 µm

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Application of excitation-emission fluorescence microscopy to thermal maturity of geological samples

Paul C. Hackley[1], Robert C. Burruss[1], Jonathan Boyd[2]

[1]U.S. Geological Survey, MS 956 National Center, Reston VA 20192
[2]Leica Microsystems, Inc., 1700 Leider Lane, Buffalo Grove IL 60089

Acknowledgments

Bob Ryder – USGS, retired
Maria Mastalerz – IGS
Carla Araujo – Petrobras
Jolanta Kus – BGR
ICCP Commission II – Thermal Indices Working Group
USGS Energy Resources Program

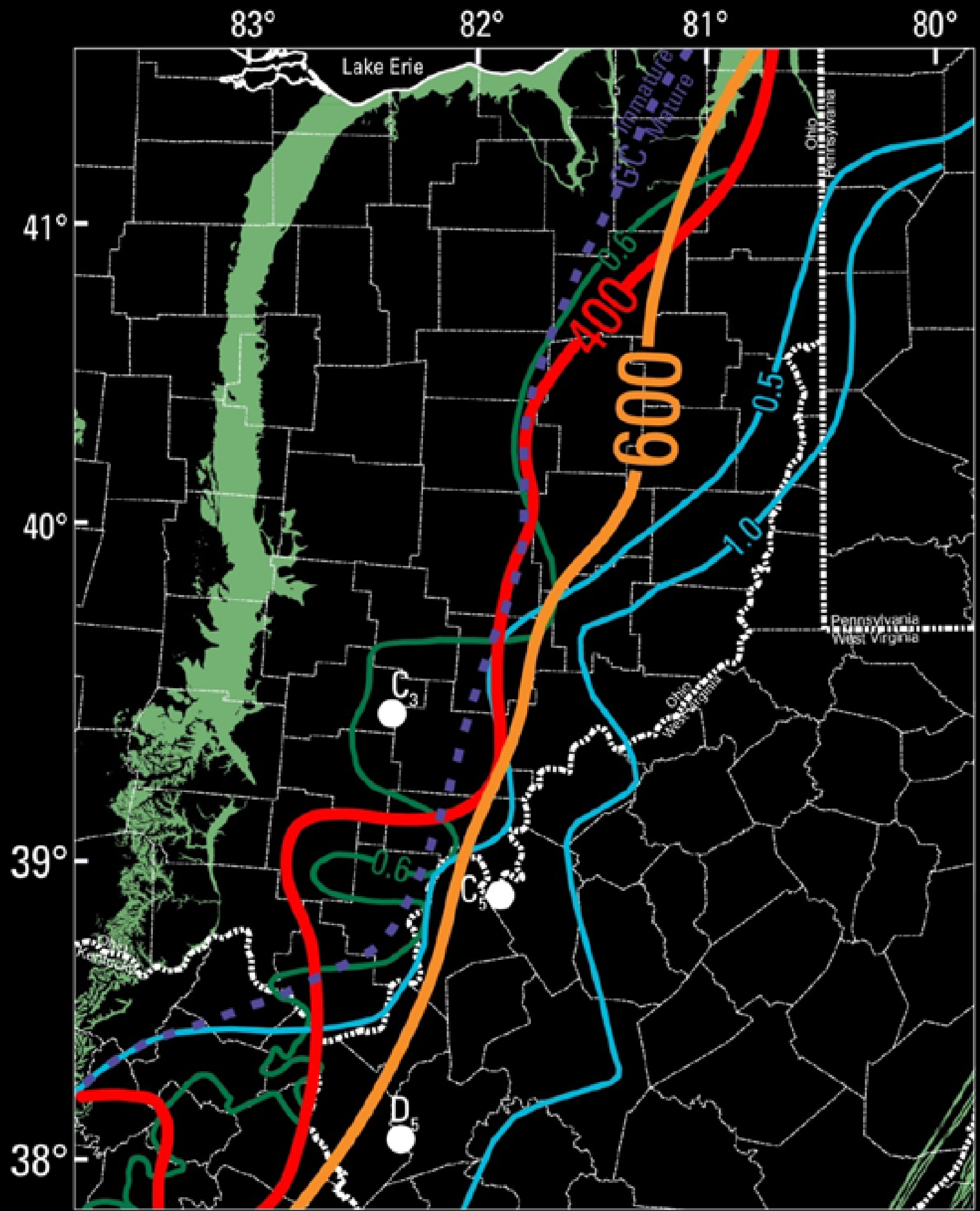
Outline

Objective and Geologic Background – the why

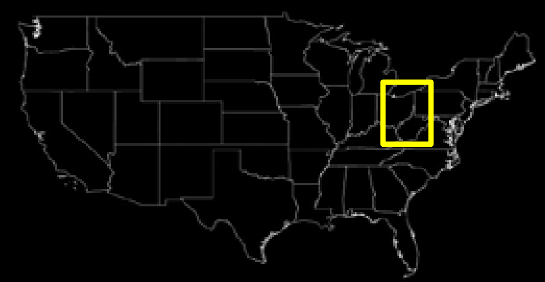
Petrographic Thermal Indices and Minutia – the how

Excitation-Emission Fluorescence Microscopy – the how?

Summary and Future Directions – the next thing



Objective: what is the western limit of thermally mature Devonian shale source rocks in the Appalachian Basin?



Explanation

- C_3 Sample Location
- Devonian shale R_o isograd
- Pennsylvanian coal $R_{o(max)}$ isograd
- GC Mature/Immature boundary line
- Spectral Fluorescence 600 isograd
- HI 400 isograd
- Outcrop areas of Devonian black shales

50 25 0 50 100 Miles

Ryder et al. 2013, AAPG S&D; Hackley et al. 2013, Fuel; Araujo et al. ms

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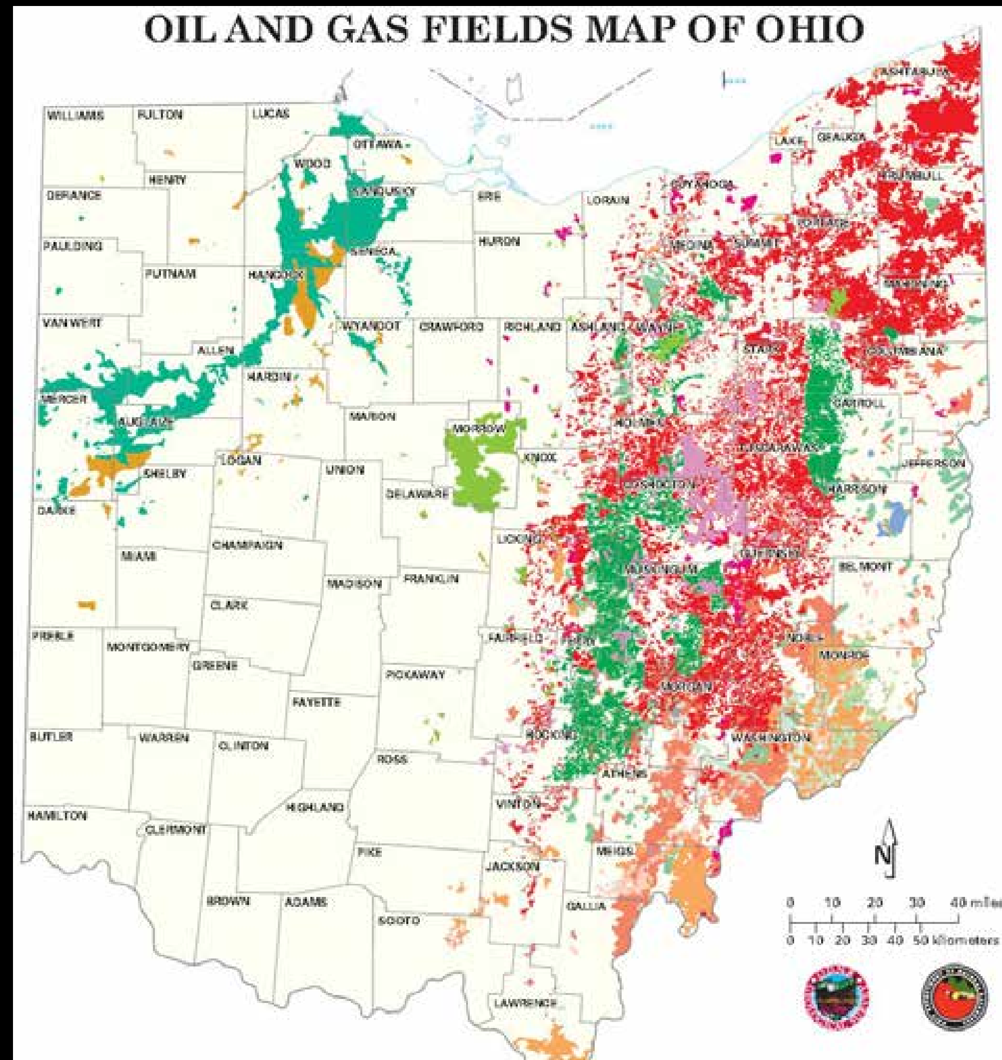
Why Important: What is the source rock?

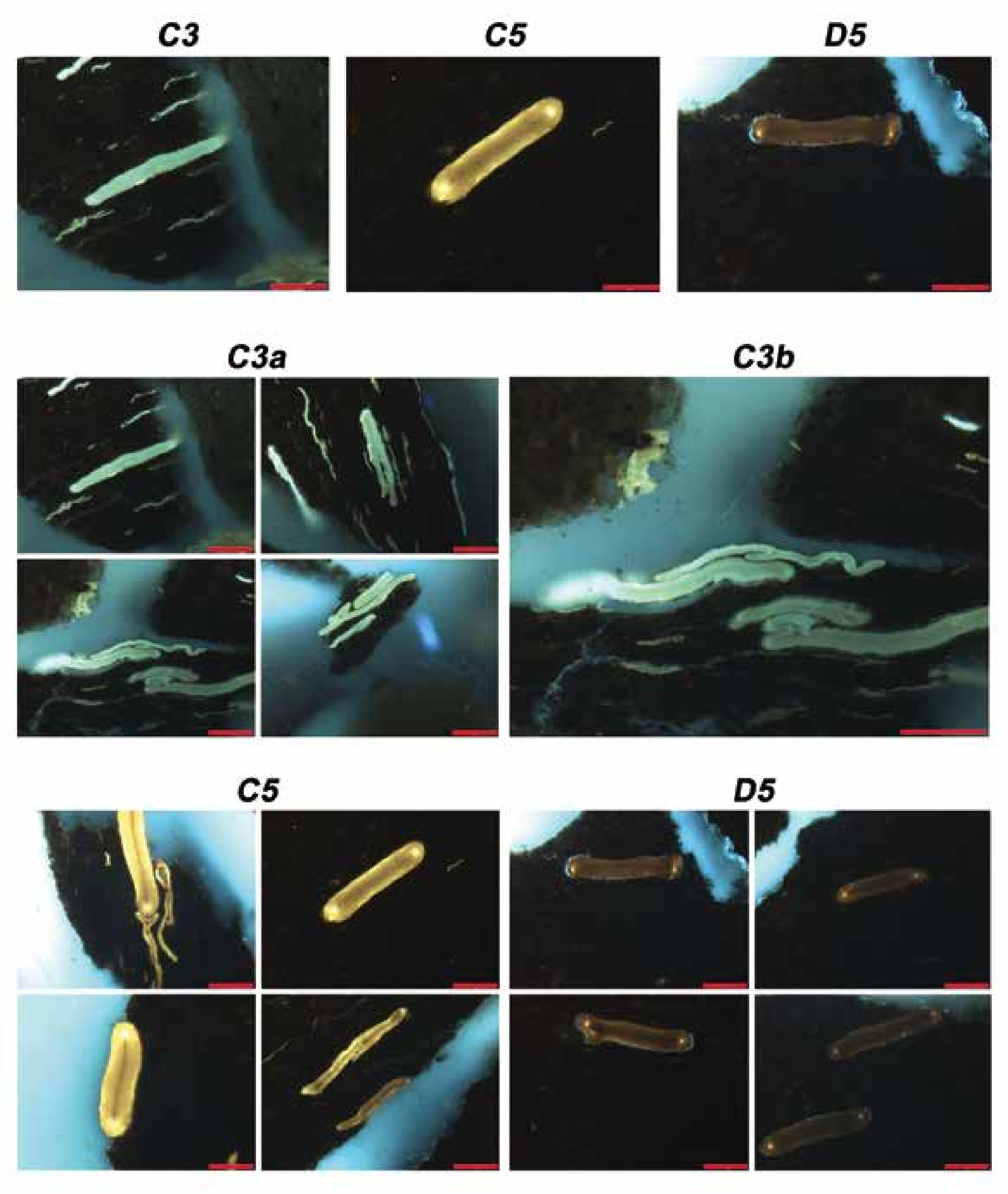
Why is thermogenic gas the dominant hydrocarbon reservoired in the Devonian?

Where is the oil?

Where do we draw boundaries for unconventional resource assessment?



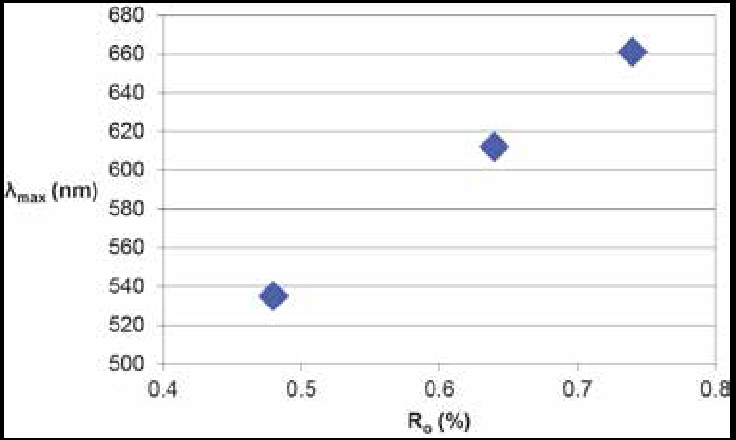
Ohio Geological Survey, 2004



Tasmanites marine algae under ultraviolet illumination

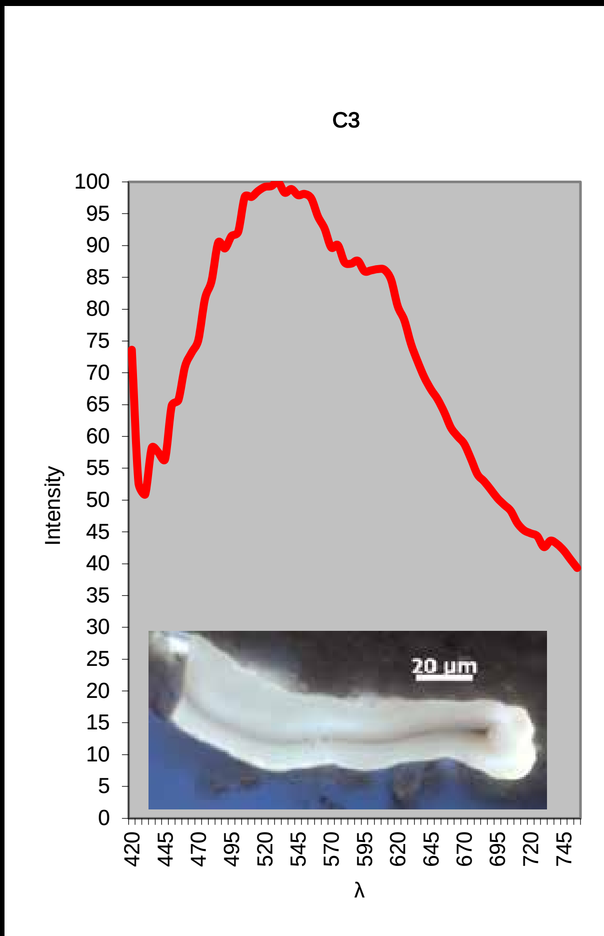
With increasing thermal maturity:

- spectra are red-shifted
- fluorescence intensity increases then extinguishes
- shrinking and cracking develop
- regions of intense fluorescence persist at points of greatest structural deformation(?)

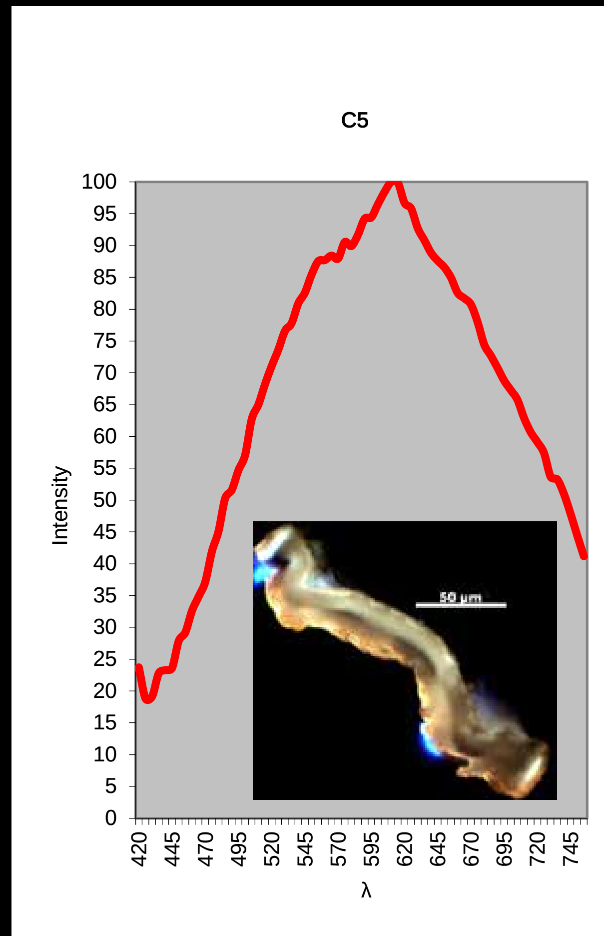


Araujo et al. ms

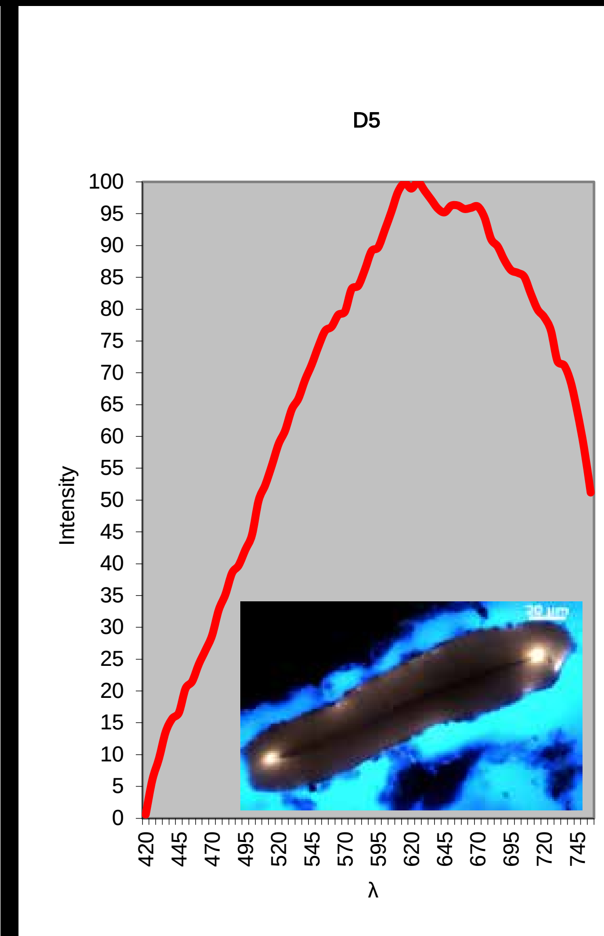
R_o = 0.45%



R_o = 0.54%



R_o = 0.66%



Conventional fluorescence microscopy; calibrated to Baranger lamp; reproducible

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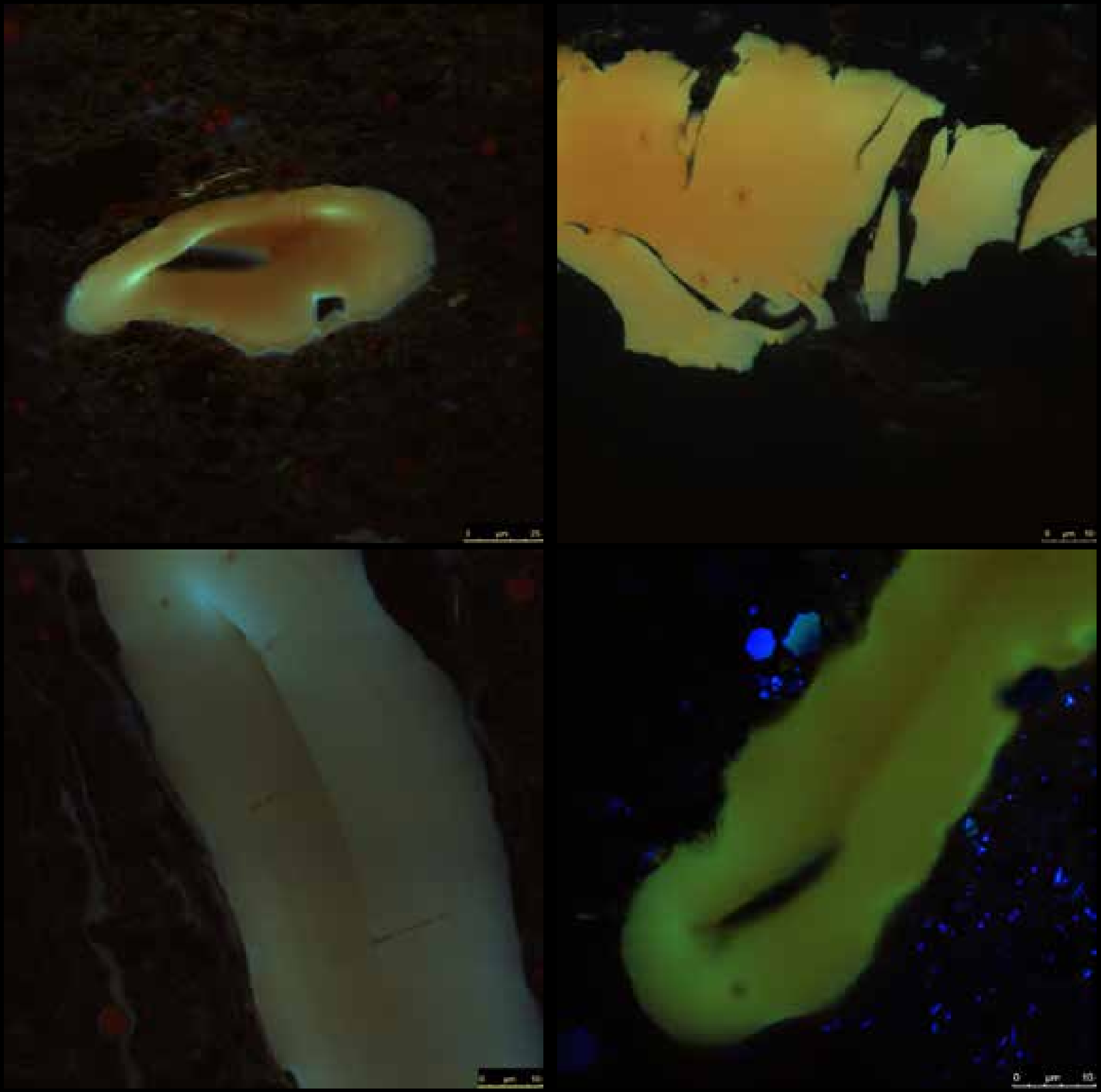
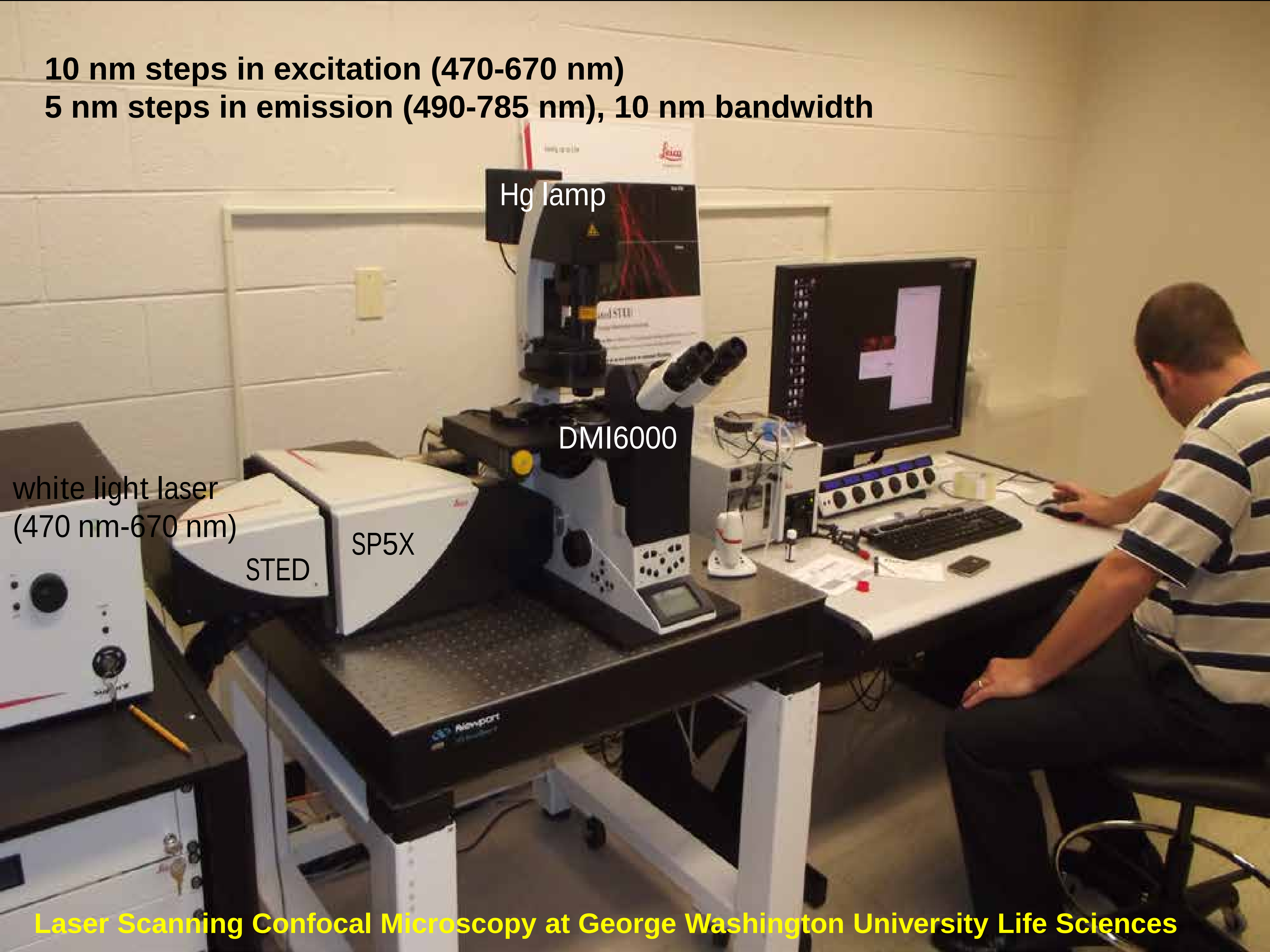


False color laser confocal images, Jolanta Kus BGR

Laser Confocal Microscopy

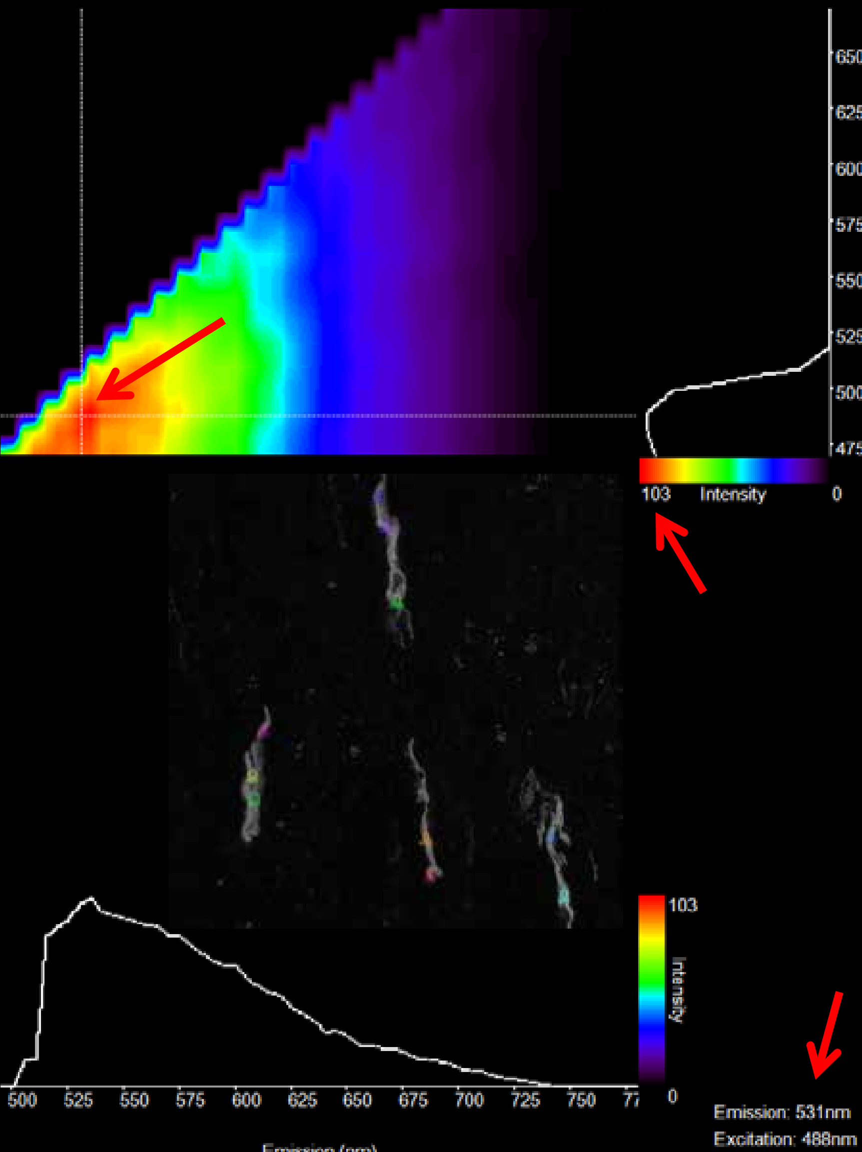
- Argon laser, 458 nm excitation @ BGR
- Powerful imaging tool
- High resolution
- Extends observations of organic fluorescence to higher maturity
- Images are false color
- Spectral data are reasonably consistent with conventional spectra from Hg illumination and equiv.

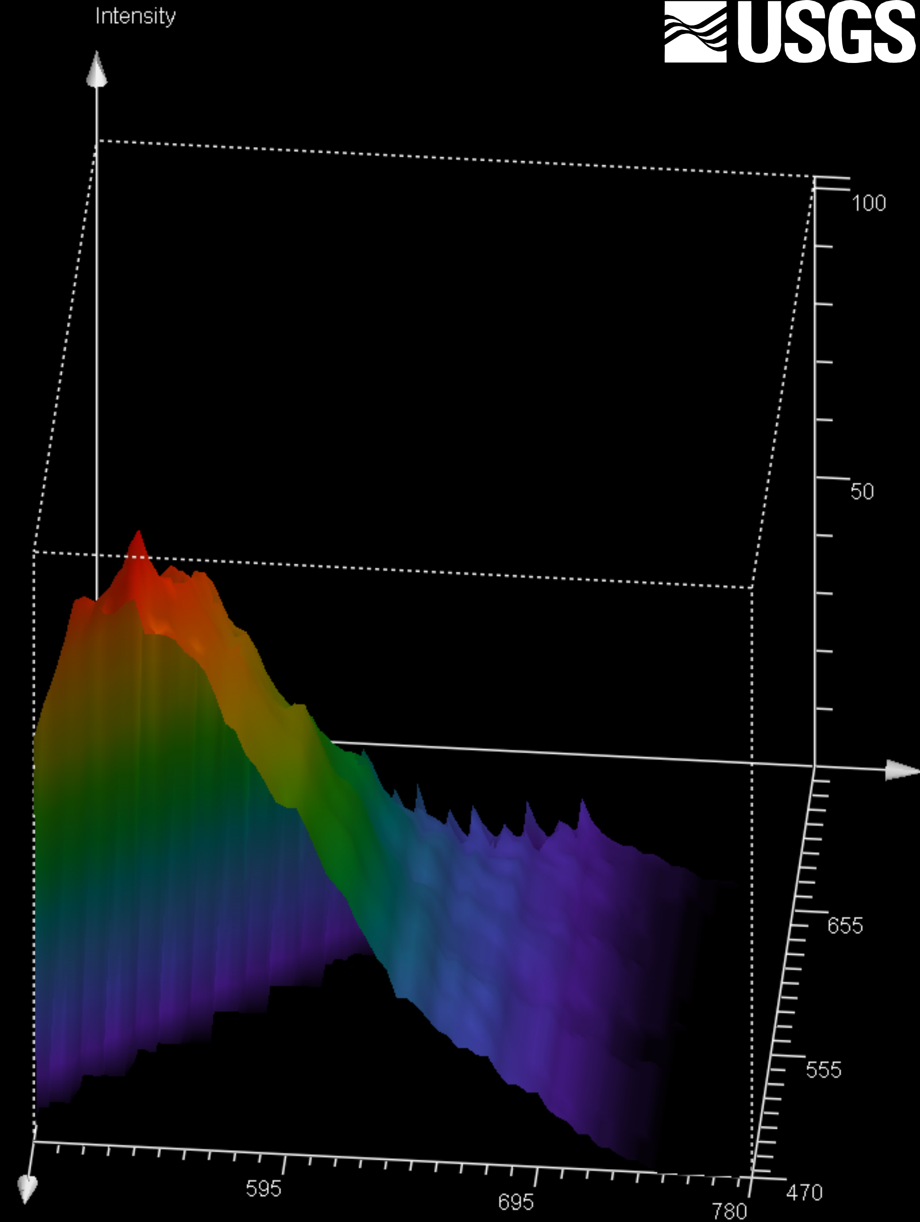
10 nm steps in excitation (470-670 nm)
5 nm steps in emission (490-785 nm), 10 nm bandwidth



Laser Scanning Confocal Microscopy at George Washington University Life Sciences

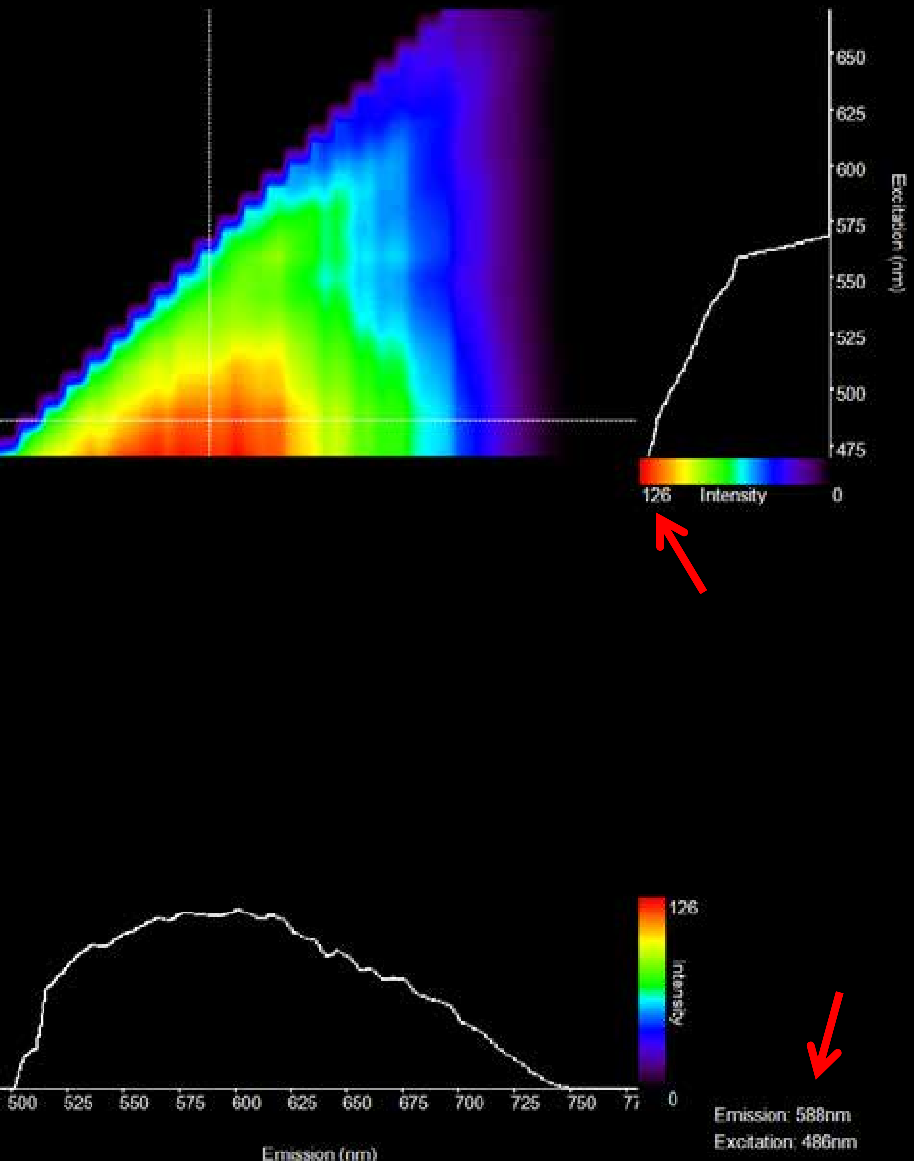
Huron 1 – lowest maturity; R_o 0.45, λ_{max} 519, T_{max} 439 (RE2)

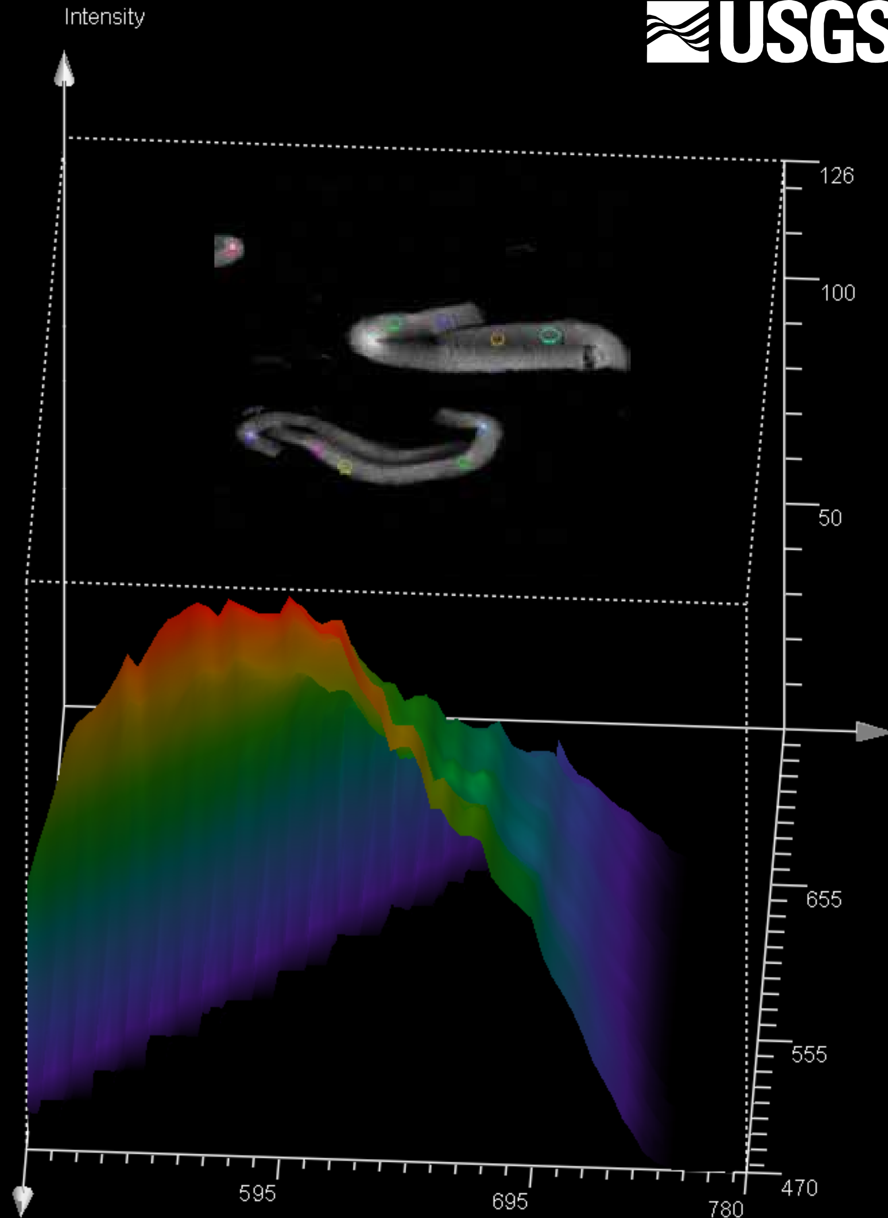




Conventional fluorescence microscopy: excitation @ 365 nm; emission @ LP 420 nm

Huron 4 – intermediate maturity; R_o 0.53, λ_{max} 611, T_{max} 448 (RE2)





Huron 3 – highest maturity; R_o 0.62, λ_{max} 610, T_{max} 440 (RE2)

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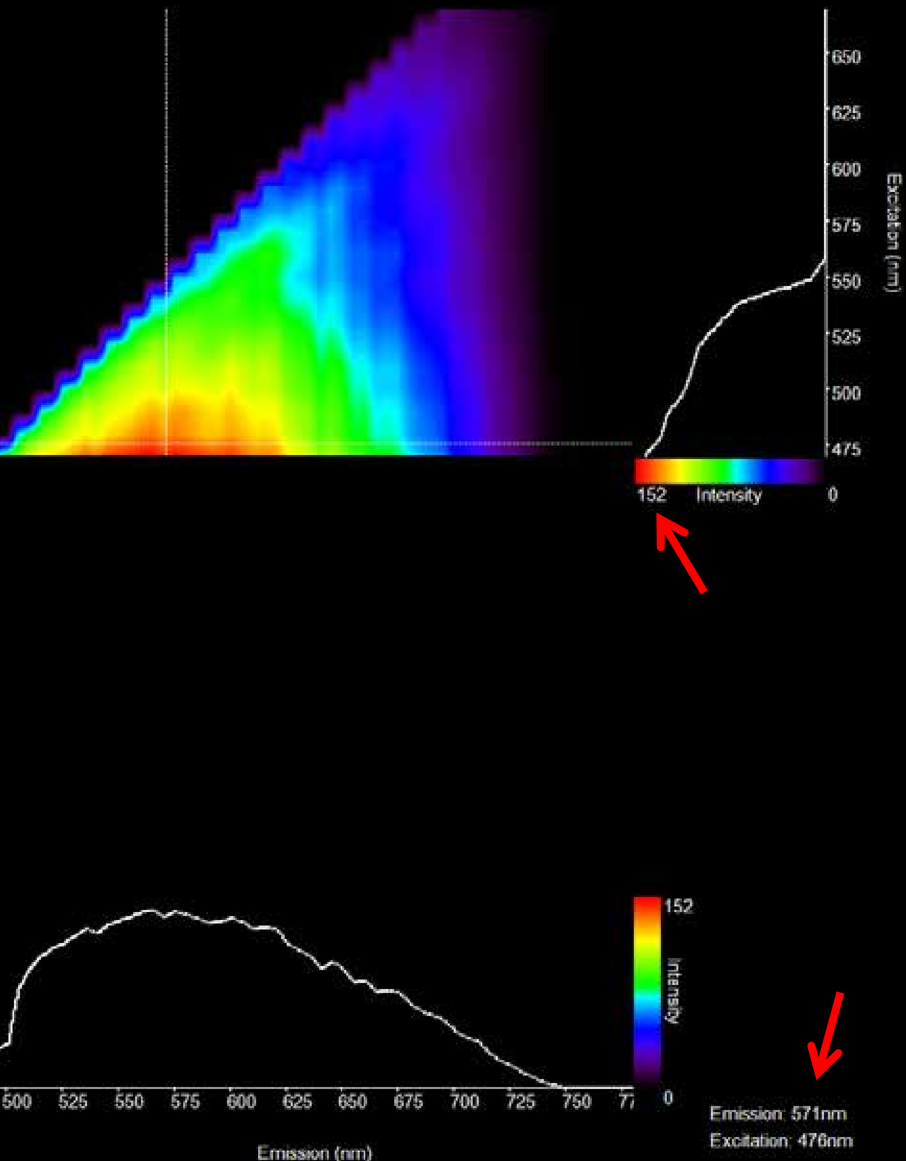
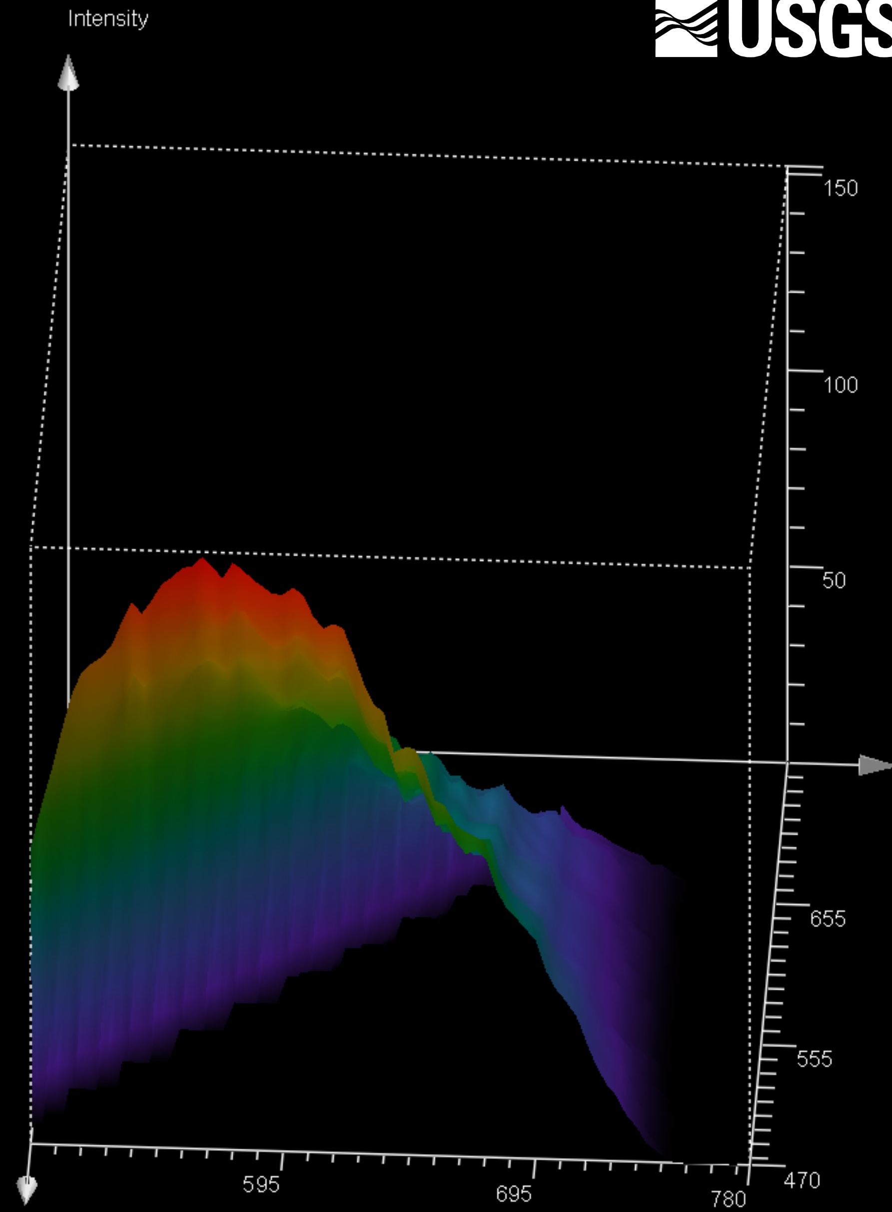
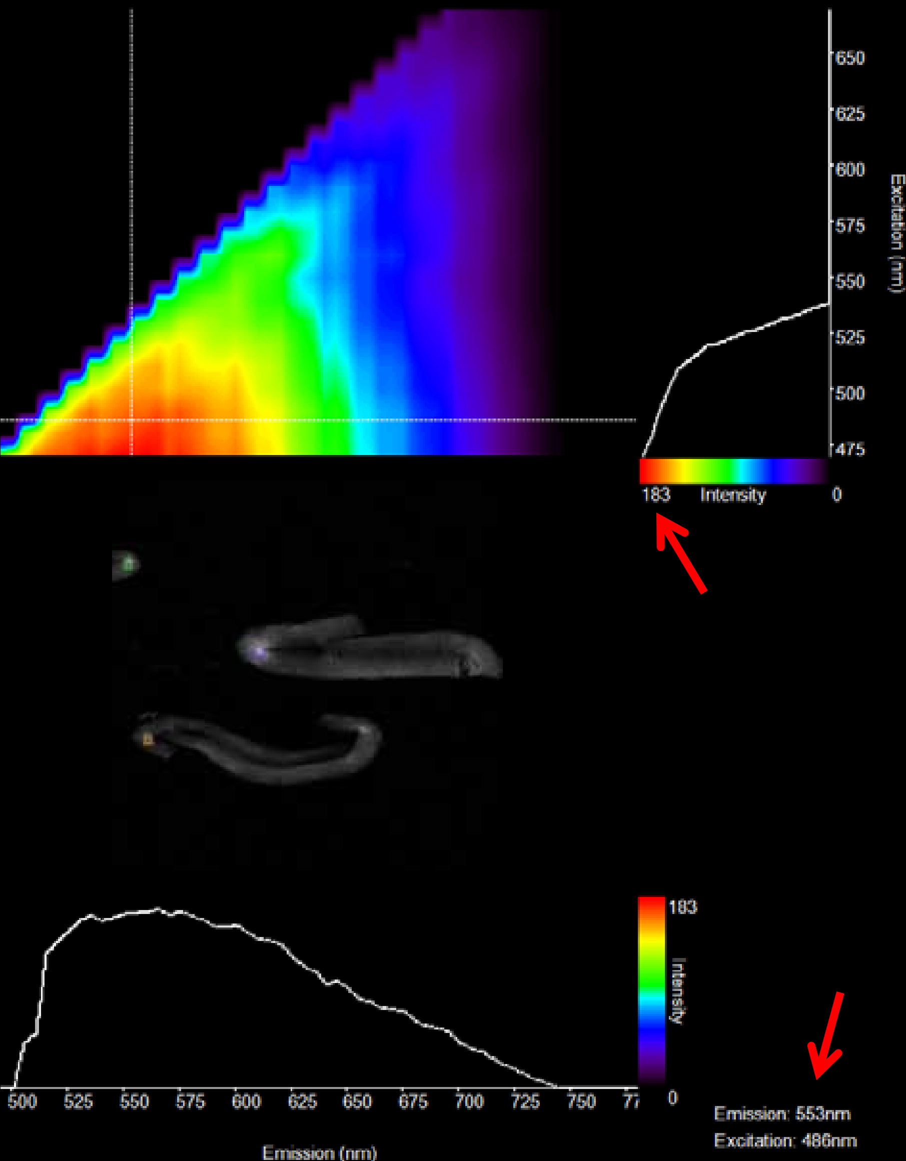


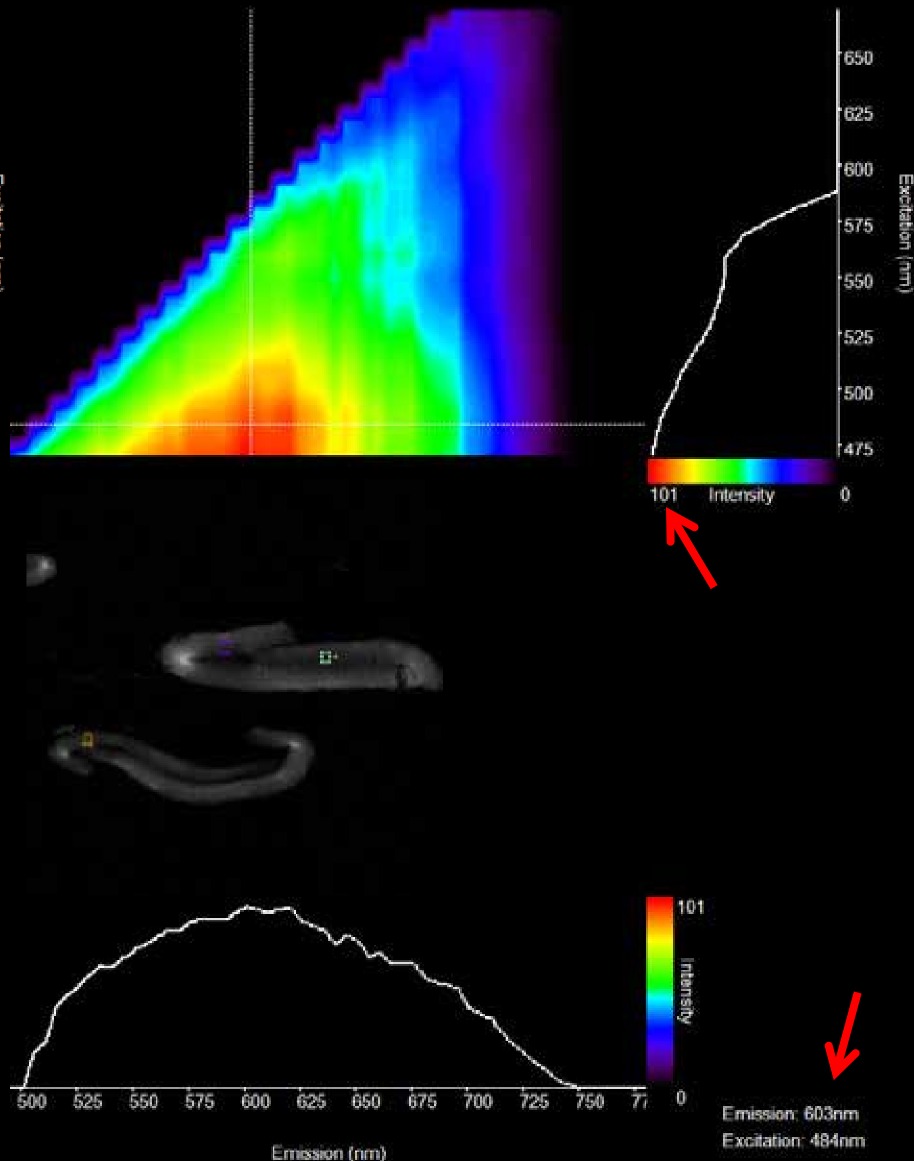


Huron 4 – intermediate maturity; R_o 0.53, λ_{max} 611, T_{max} 448 (RE2)

brighter regions, blue shifted



dimmer regions, red shifted



Summary

Laser scanning confocal microscopy applied to geological materials:

<u>So what?</u>

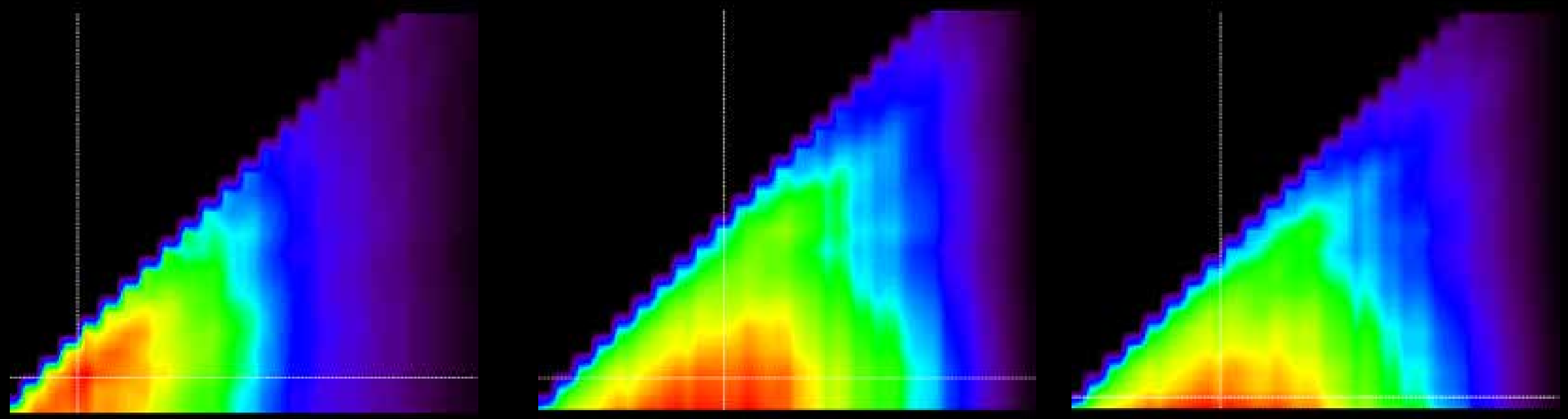
- **Improved imaging – high resolution**
- **Comparable spectra to conventional fluorescence microscopy**
- **Characterization of thermal maturity**
- **White light laser allows collection of broad ‘spectrum’ of spectral data – the EEM**

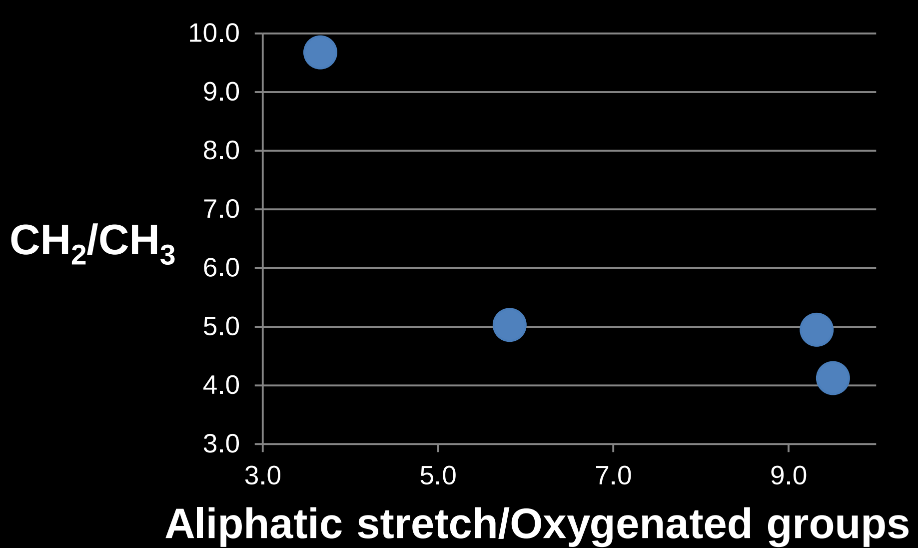
<u>Yeah .... but,</u>

- **Comparable spectra to conventional fluorescence microscopy – high instrument costs, long scanning times**
- **What do the EEMs tell us?**

Future Directions

- **What do the EEMs tell us?**





µ-FTIR data courtesy Maria Mastalerz, IGS

- Preliminary µ-FTIR data indicate aliphatic chains become shorter & more branched, oxygenated groups decrease
- What will XPS tell us about CNOS abundance & speciation? ^{13}C NMR? Kerogen concentration is challenge!
- Are the EEM data reproducible?
- Can the molecular data be tied to the EEM?

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Thank You!

image courtesy of Jolanta Kus, BGR