

**Title: Quantification of organic nano-pores using a helium ion microscope**

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Scanning electron microscopy of ion polished samples has become a common way to estimate porosity and organic matter content within shale resource rocks. Since quantitative SEM analysis has emerged as a means for assessing the porosity of shale rock, a common goal has been to image polished samples at extremely high resolutions. Since nanopores are visible at pixel resolutions ranging from 5-10 nm, it is natural to consider the possibility of a pore regime below 5 nm which could contribute a significant amount to the total porosity of the system. When considering that a molecule of methane gas is on the order of 0.4 nm diameter, these 5 nm pores could be significant transport pathways in a reservoir. These nanopores are a significant source of porosity within certain organic matter bodies, where total detectable pores using SEM (i.e., ~10 nm pore body diameter and up) can comprise up to 50 percent or more of the original volume of organic matter present. With the potential to examine the population of pores below ~10nm in diameter using the helium ion microscope, it is possible to construct a rock model that is more representative of the varied pore size regimes present.

In this study, 12 organic shale samples were selected for systematic imaging using the Carl Zeiss Orion helium ion microscope. These samples were chosen based on examination of previously completed imaging using Carl Zeiss Auriga FESEM, and were selected due to the presence of porous and non-porous organic matter. Prior to SEM imaging, the samples had been ion-polished using a Gatan argon ion polishing system. The previously completed set of SEM images were acquired with a pixel resolution of 10 nm. Samples were imaged in the helium ion system using varying parameters in order to optimize image quality. Field of view and resolution were selected and increased as appropriate, with each acquired image matching a subset of an extant SEM image to allow for a direct comparison of grayscale, resolution, and volume percentage of various materials. The smallest pixel size of these images was 0.5nm.

After careful and consistent segmentation, it was concluded that most samples had no significant pore fraction below the detection threshold of conventional FESEM imaging. The advanced resolution capabilities of the helium ion beam provide much sharper definition of pore boundaries but the total volume of these <10nm diameter pores in most samples was negligible.