U-Th dating of ostrich eggshell:

Understanding the relationship between eggshell microstructure and diffusion and implications for sample preparation

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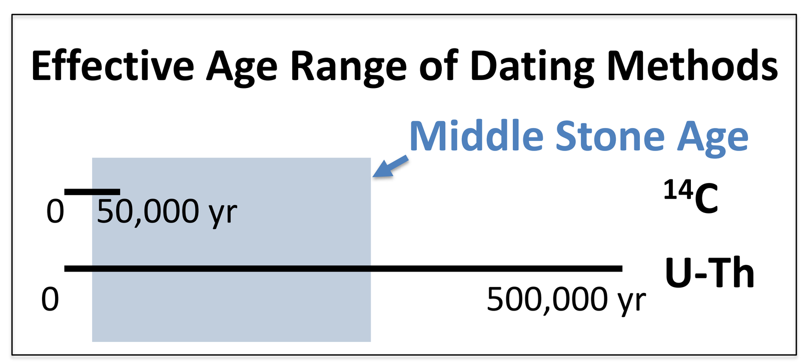
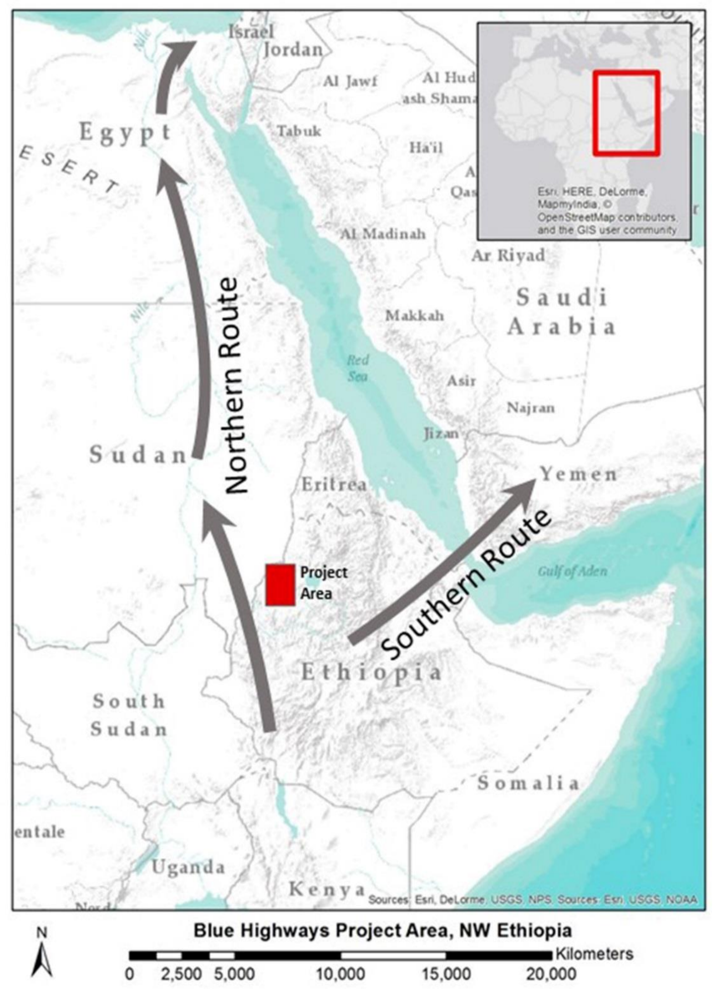
Background

Abstract: Dating the Middle Stone Age (~250-30 ka) is of great importance to studies of human evolution because this time period witnessed the origin of modern Homo sapiens in Africa, its migration out of the continent and across the rest of the world, advances in stone tool manufacture, and changes in foraging strategies. Techniques such as AMS 14C can only be applied to the youngest end of this interval. Because many human groups collected ostrich eggs, U-Th dating of ostrich eggshell (OES) has often been used to establish the age of archaeological sites. Uranium from soil diffuses into the OES crystal structure after burial, and measurement of U and its Th decay product, along with an estimation of U diffusion rates (Sharp et al., 2015) into the OES enables a determination of the time of burial. However, age determinations can be complicated by the incorporation of detrital Th from soil into the OES. Sharp et al. (2014) showed that the external OES layers contain high detrital Th, and Loewy et al. (2016) demonstrated that the pores in the internal palisade layer also contain high detrital Th. Pores provide a critical avenue between the external and internal surfaces of the OES for Th to infiltrate the eggshell. We completed high-resolution CT scans of OES to map the number, 3D pattern, and volume of the pores. Rather than a single pore, the structure consists of numerous very small "satellite" pores oriented circumferentially around a large central pore. Total pore volume can be significant. In order to minimize the effect of detrital Th, we recommend that sample preparation combine the removal of the two outside layers of the eggshell with drilling out the area around each pore. Minimizing detrital Th diminishes the impact of the correction calculation, which is critical for precise age determinations.

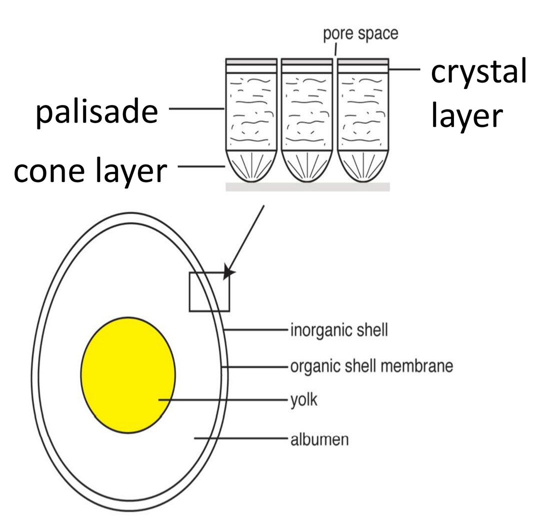
Dating the Middle Stone Age (MSA) is of great importance to studies of human evolution because this time period witnessed the origin of modern *Homo sapiens* in Africa, its migration out of the continent (see map at right) and across the rest of the world, advances in stone tool manufacture, and changes in foraging strategies.



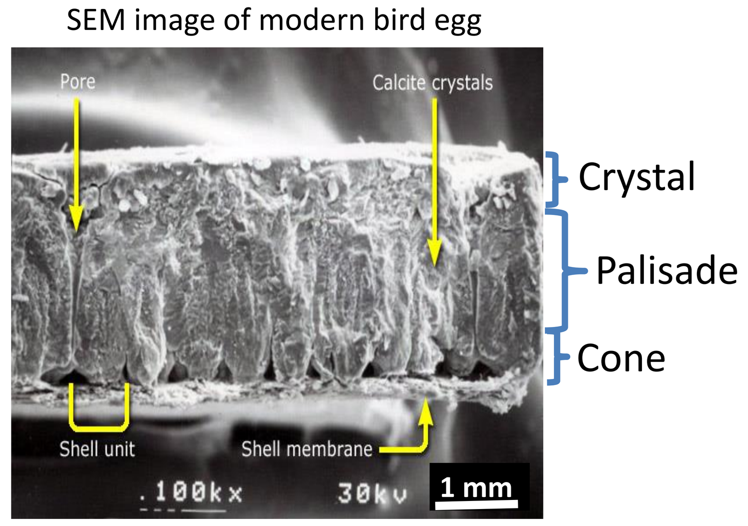


Commonly-used radiocarbon dating can only be applied to the youngest end of this time interval. Many human groups collected ostrich eggs for food, and used the empty shells as containers. Therefore, many sites contain ostrich eggshell fragments. U-Th dating of ostrich eggshell (OES) may be useful to establish the age of archaeological sites.

Uranium from soil diffuses into the OES crystal structure after burial and decays to Th. Measurement of U and its Th decay product enables determination of the time since burial. Two issues limit precise age determination: 1) the lack of constraint of the rate and timing of U diffusion into the OES after burial; and 2) incorporation of Th into the OES from infiltration of Th-rich soil particles into pores and cracks of the OES.



SEM image of modern bird egg



http://www.ucmp.berkeley.edu/science/eggshell/eggshell1.php

__U-diffusion__: In an ideal situation, U would diffuse into the OES quickly after burial and then diffusion would stop so that the OES structure would act as a closed box. The U would decay to Th inside the OES, and subsequent measurement of U and Th would reflect the timing of burial of the OES. If diffusion was slow and occurred continuously while the OES was buried, then the calculated U-Th age would be younger than the actual burial age. Ongoing work attempts to understand diffusion (Sharp et al., 2014, 2015; and see discussion of our future work at left).

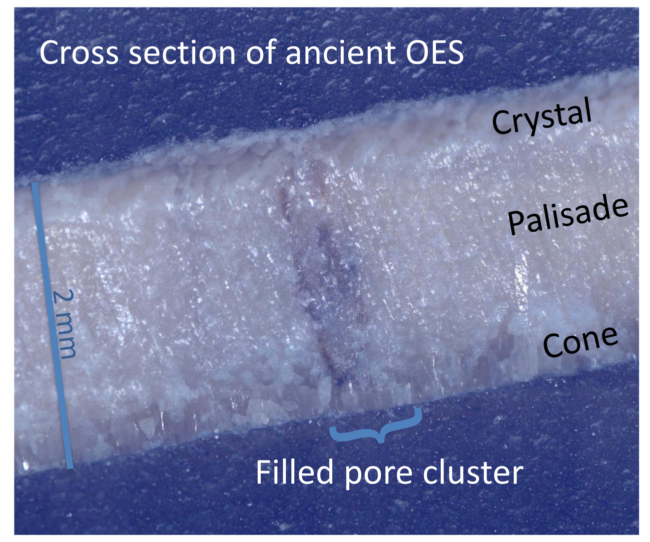
__Soil Th__: In an ideal situation, all of the Th in an ancient OES would come from the decay of U. However much of the Th in OES comes from soil particles that also fill the natural pores of the OES (Loewy et al., 2016) and the cracks in the exterior crystal and interior cone layers (Sharp et al., 2014). The isotope of Th from the decay of U is ^{230}Th. The soil Th is mostly ^{232}Th, but includes a little ^{230}Th. To determine the amount of ^{230}Th from decay of U, we must subtract the ^{230}Th from the soil-derived Th. Minimizing the quantity of soil-derived Th in the analyses will decrease the necessary correction and greatly improve precision. Work presented here focuses on a new method for eliminating soil-derived Th from analyses.

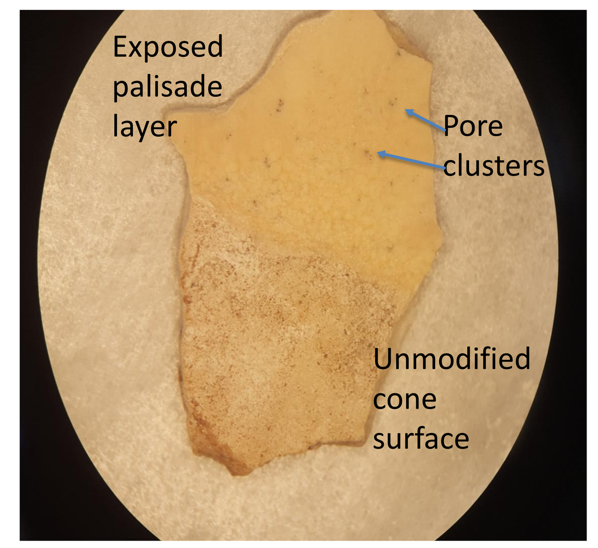
Methods

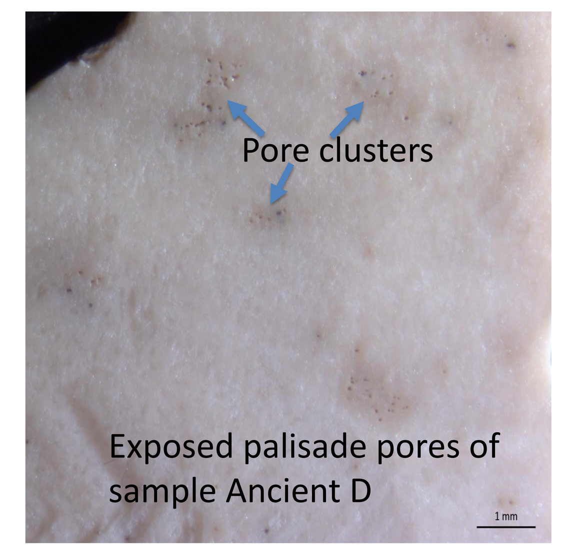
Field work in NW Ethiopia by Americans and Ethiopians excavating the MSA human site SM-1.

OES collected from the excavation site.

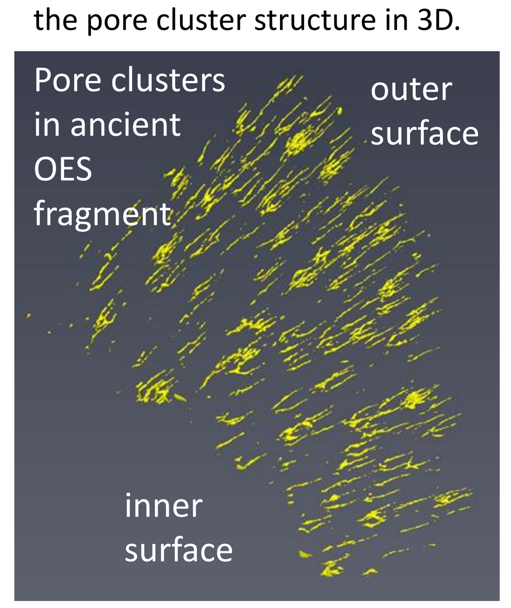
The Th-rich cone and crystal layers were removed with a dental drill, thereby exposing the pore clusters.





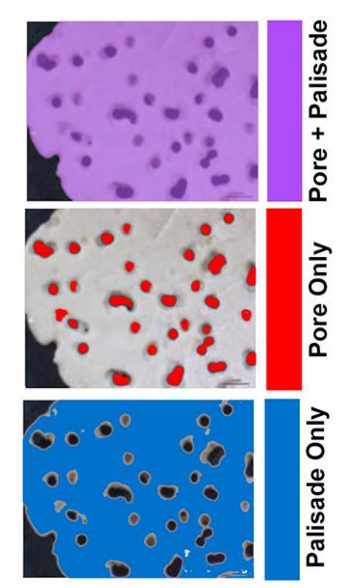


High-resolution CT used to image the pore cluster structure in 3D.



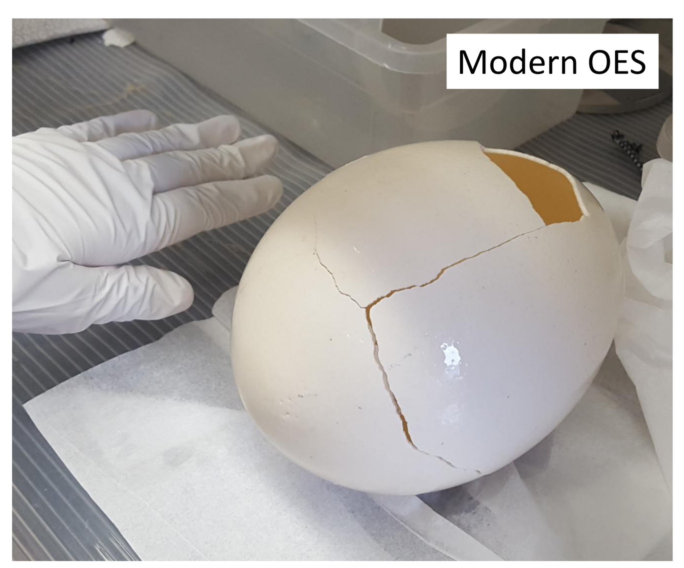
Pore cluster fill was removed with a dental drill.

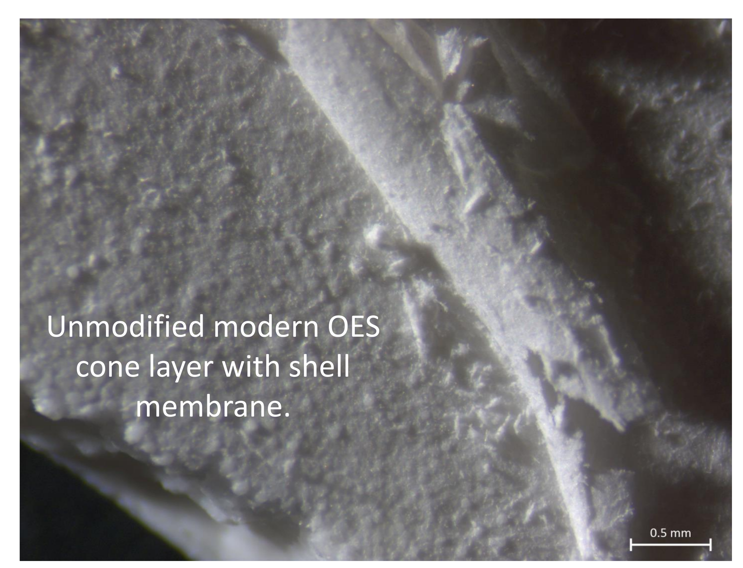
SAMPLE TYPE

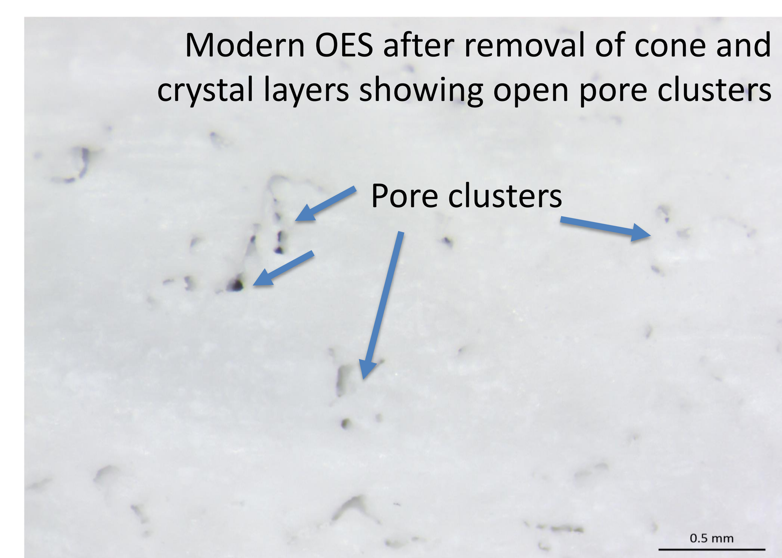


U-Th analyses were conducted on the remaining palisade layers. Pore-fills were analyzed for comparison.

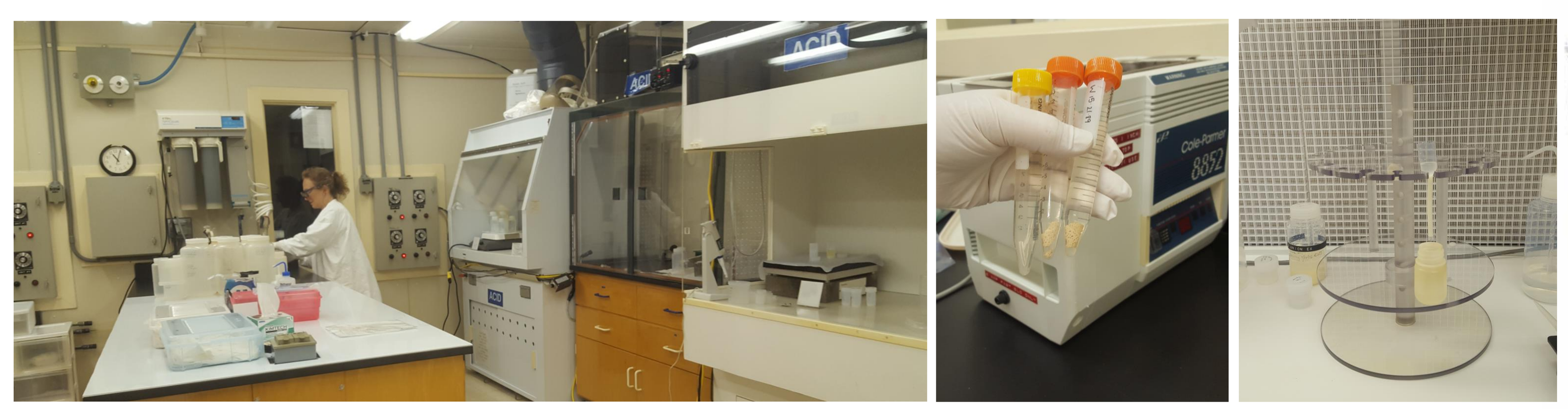
Modern OES palisade layer was analyzed in order to determine U and Th concentrations when OES forms and before burial.





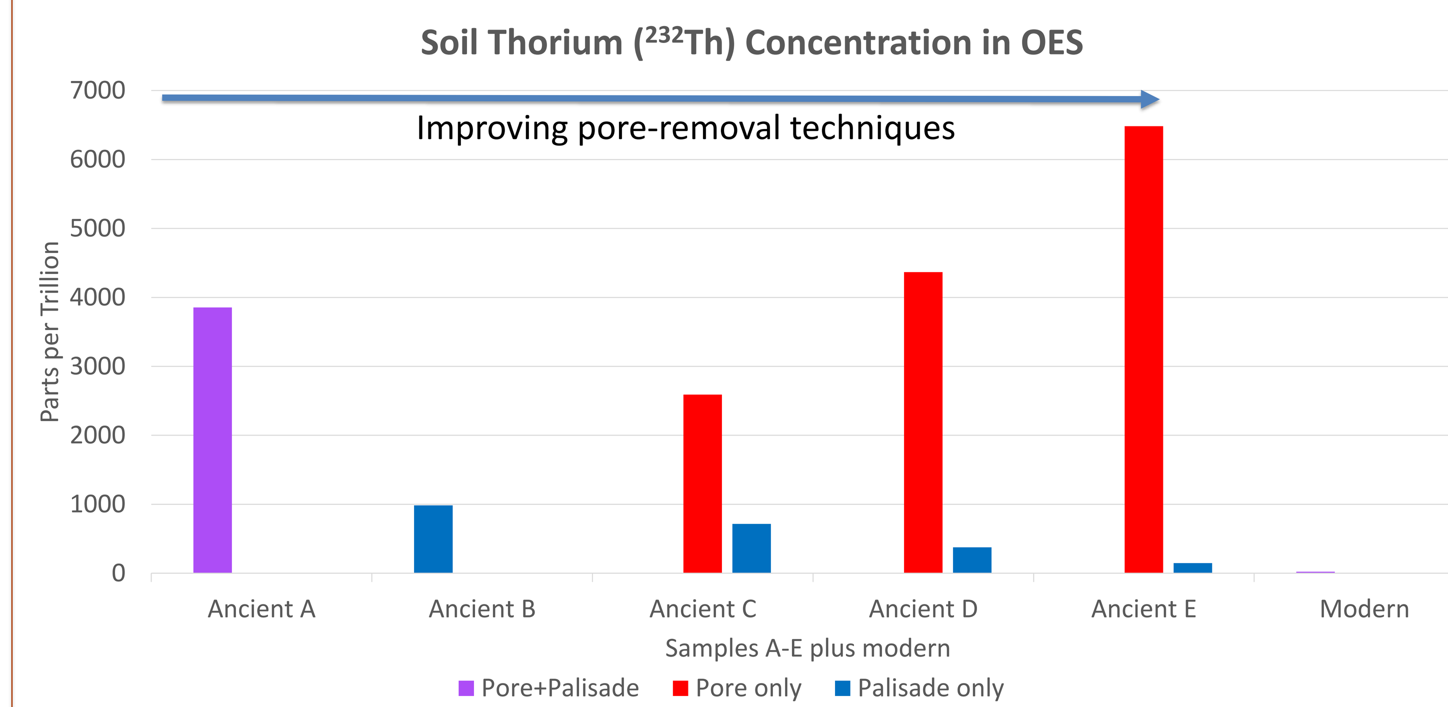


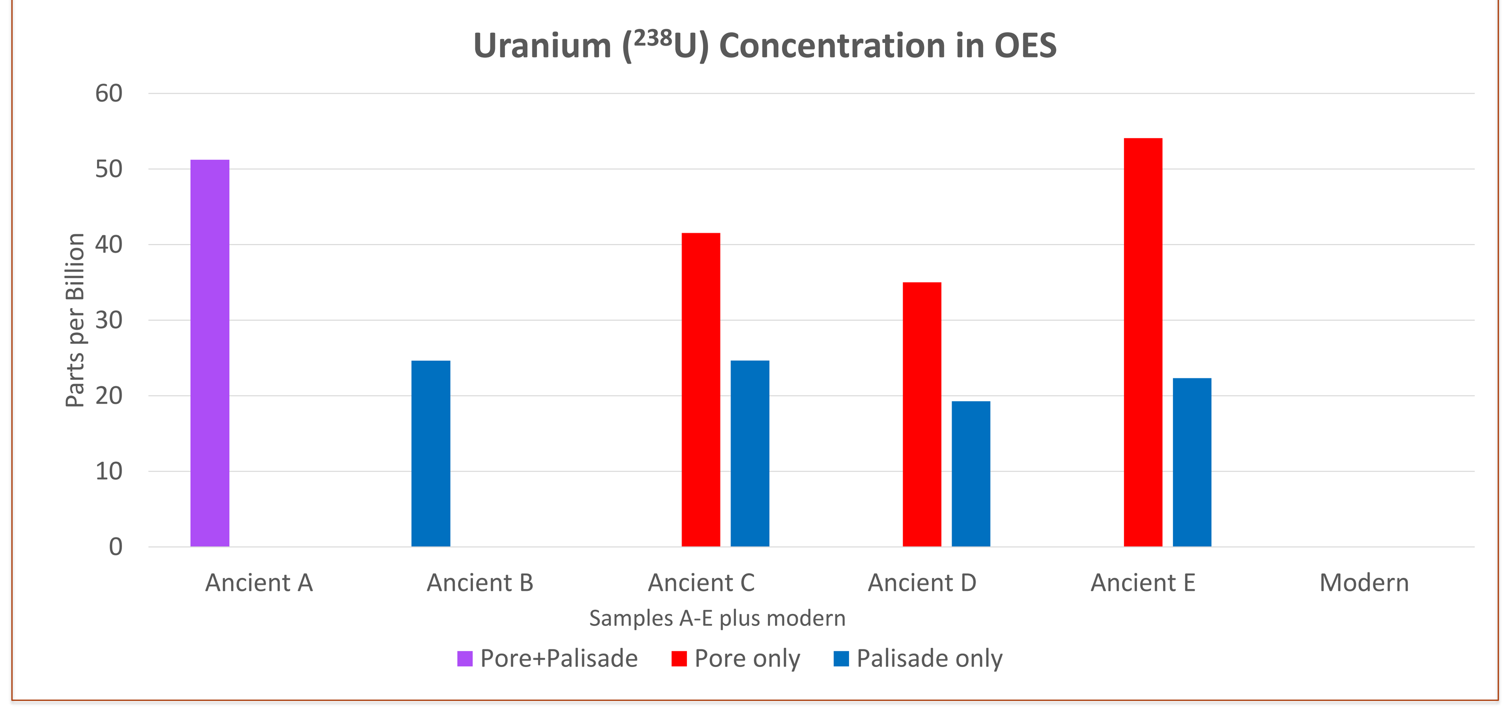
OES samples were processed in a class 100 clean lab using standard U-Th analytical techniques, and analyzed using a ThermoScientific Triton Thermal Ionization Mass Spectrometer (TIMS) at The University of Texas at Austin.



Triton TIMS

Results





Conclusions

- Soil-Th is clearly concentrated in the pore-fill. The cleaned palisade layer has almost no soil-Th.
- Our new multi-step preparation method effectively removes soil-Th from the OES.
- U is somewhat more concentrated in the pore-fill but is also present in clean palisade layer.
- Modern OES has no U (below detection level) and very little soil-Th (20 ppt), thus supporting previous conclusions that U and Th enter OES after burial.

Ongoing Work

We are testing rates of U diffusion and Th-rich particle infiltration. Six fragments of the modern OES were placed in teflon jars, 3 with soil (TX) and 3 with basalt powder (local SM-1), and distilled water and heated to 40°C, the average soil temperature at SM-1. After 6 months, 1 year, and 2 years, we will measure distribution of U and Th across OES using laser ablation ICP, image visible changes in OES with high resolution microscopy, and analyze U and Th concentrations using TIMS.

Acknowledgments

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Special thanks to our friends who live near SM-1 for assisting with field logistics.

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