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A Transdisciplinary Approach to Determining the Provenience of a Distorted, Pre-Columbian Skull Recovered in Rural Idaho

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Abstract

Transdisciplinary research involves cooperation, exchange of information, sharing of resources and integration of disciplines to achieve a common scientific goal. In this study, collaborators utilized tools and knowledge of materials science, anthropology, archaeology, geosciences and biology in an attempt to determine the provenience of skeletal remains of unknown origin. The exchange of ideas and skills along with the crossing of disciplines in this study successfully allowed the incorporation of expertise from many team members. This transdisciplinary approach to research provided a more comprehensive and detailed analysis than any one field alone could provide. An archaeological assessment of a human skull recovered in rural Idaho recognized cranial deformation and post-mortem application of a red pigment. A combination of scanning electron microscopy (SEM), x-ray fluorescence (XRF) and energy-dispersive x-ray spectroscopy (EDS) identified the major and trace elements present in the red post-mortem pigment as cinnabar and rare earth metals. Analysis via carbon and oxygen stable isotopes from teeth and bone provided insight into the diet and habitat for distinct segments of the individual's life, indicating a regional separation in early life versus late adulthood. Radiocarbon dating determined the approximate age of the skull to be between 600-700 years old and a forensic mtDNA assessment categorized a mitochondrial haplogroup for the remains as originating from the East African or Arabian Peninsula.

Keywords

provenience, radiocarbon dated, trace element analysis, DNA analysis

Disciplines

Materials Science and Engineering

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Introduction and Background

Provenance, a history of ownership, helps provide contextual evidence of an item's origin, or **provenience**. A transdisciplinary collaboration utilized a variety of techniques including archaeological and anthropological assessments, scanning electron microscopy (SEM), energy dispersive x-ray spectrometry (EDS), x-ray fluorescence (XRF), laser ablation inductively coupled plasma quadrupole mass spectrometry (LA-ICP-QMS), stable isotope analysis, radiocarbon dating and DNA analysis to yield information about the skull's background. Researchers hope to obtain enough information in order to repatriate the individual's remains.



Frontal view of the distorted skull



Left side view of skull

The skull is thought to be that of a Native American due to

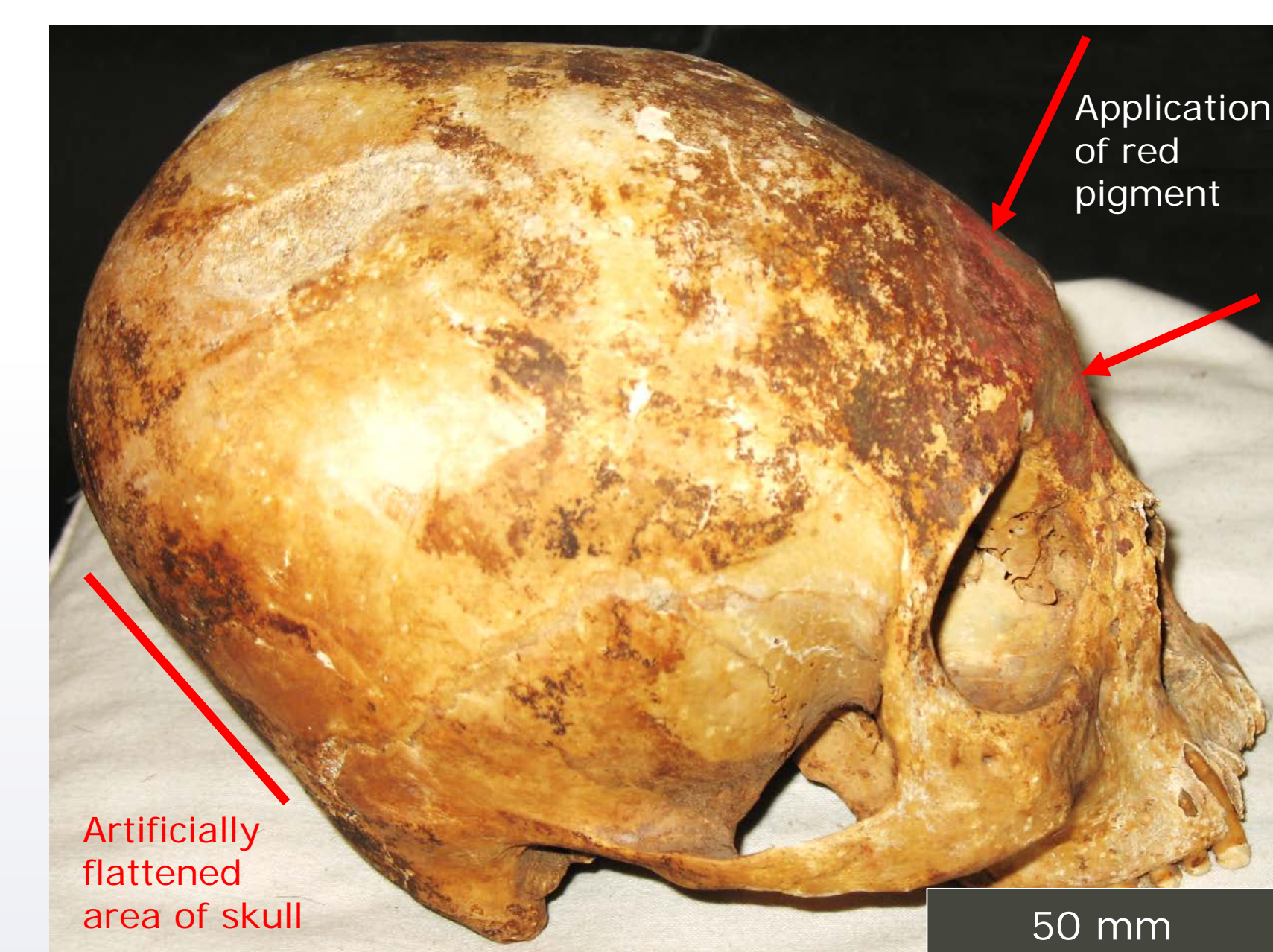
- Post-mortem application of red pigment embedded fibers
- Artificial cranial deformation (flattening at back of skull)

Analysis of pigment and fibers was completed using

- Anthropological assessment
- SEM/EDS
- LA-ICP-MS and XRF

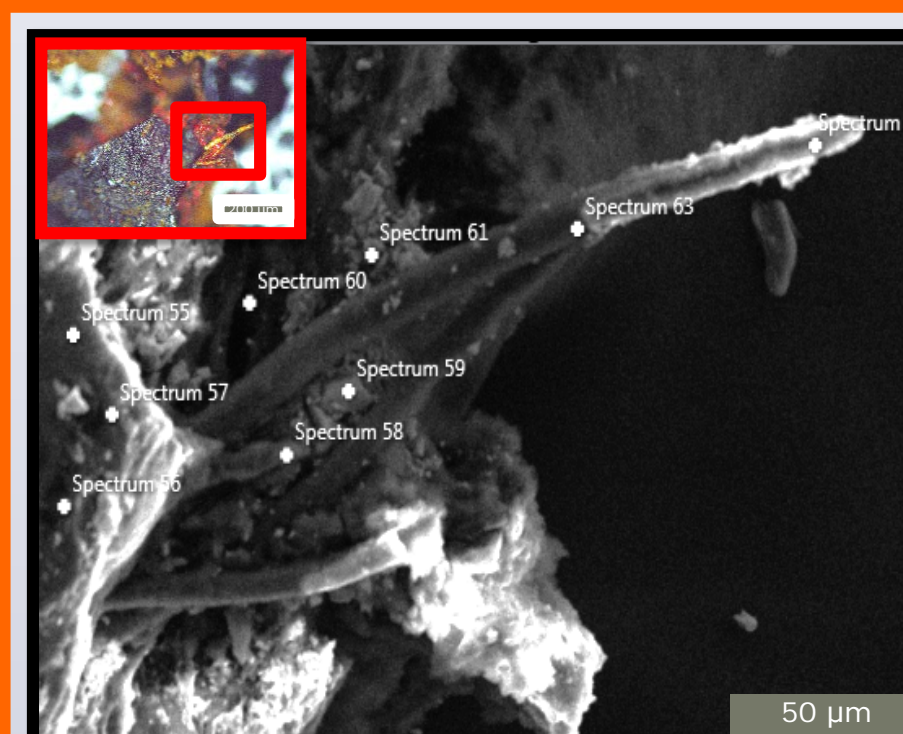
Age, habitat information, and lineage obtained by

- Radiocarbon dating and stable isotope analysis
- Forensic mitochondrial DNA for comparison to known Native American populations



Artificial cranial deformation evident at back of skull where flattening is seen

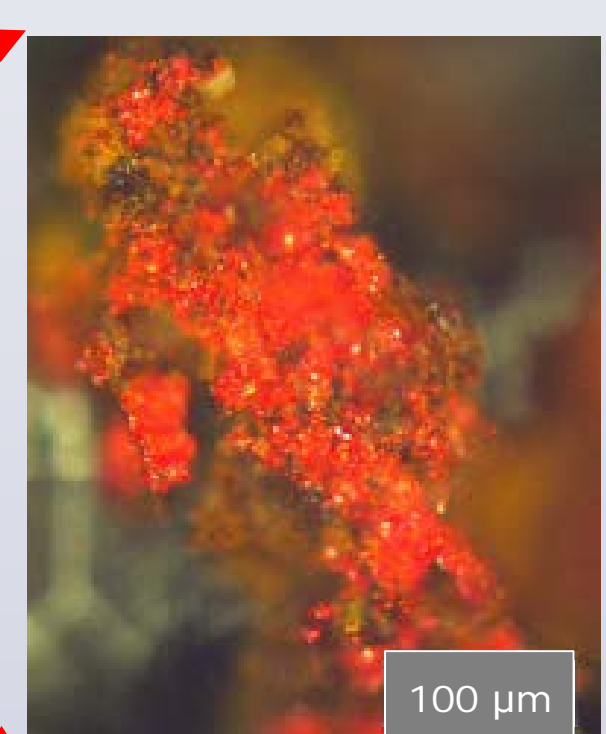
Experimental Techniques



EDS electron image of elemental analysis locations. Inset: optical photo of same location



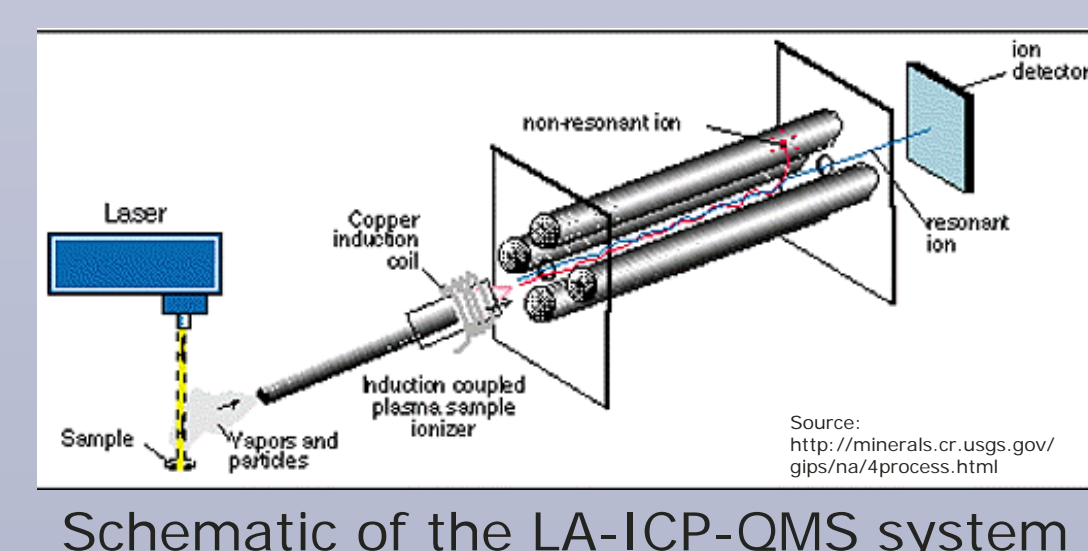
Close-up of skull where pigment samples were taken from



Optical photograph of red pigment

Laser Ablation Inductively Coupled Plasma Quadrupole Mass Spectrometry (LA-ICP-QMS)

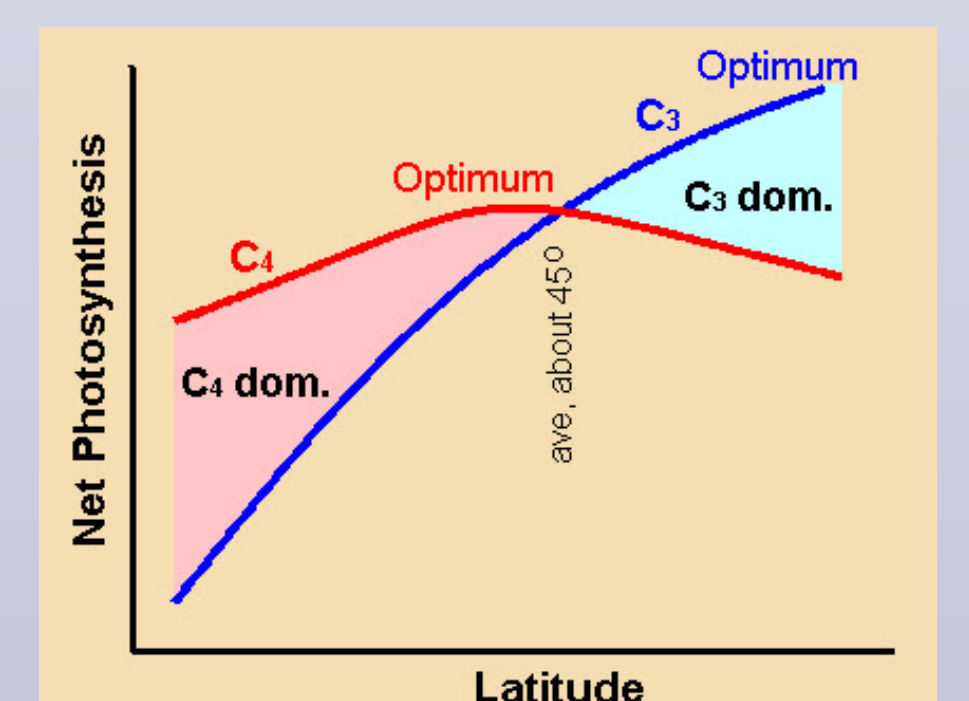
- Sample is vaporized and ionized in the plasma
- The quadrupole rod charge determines which ions will be carried to the detector for counting
- Samples were analyzed using a ThermoElectron X-Series II and New Wave Research UP-213 ND:YAG UV (213 nm) laser ablation system



Schematic of the LA-ICP-QMS system

Optical Microscopy, Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS)

- Similar to the beam of light used in an optical microscope, the SEM uses a beam of electrons to image the pigment
- EDS analysis was used to identify the elemental composition of the pigment



Net photosynthesis vs. Latitude for C₃ and C₄ plants. C₃ plants dominate at higher latitudes. [1]

Stable Isotope Analysis

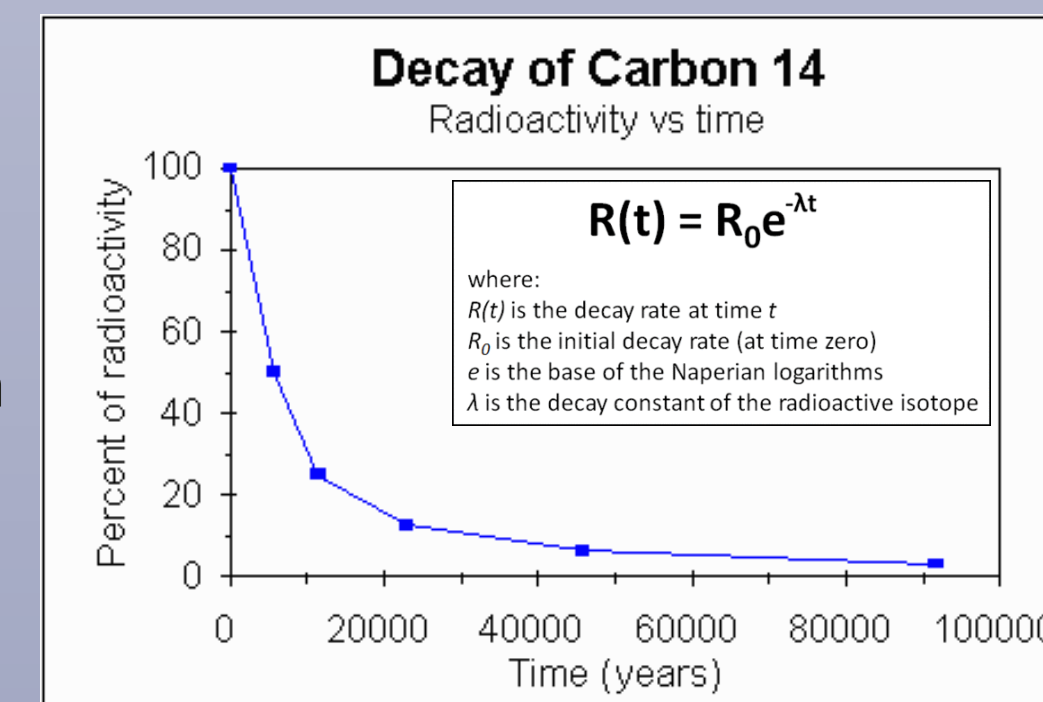
- Ratio of C₃ to C₄ in plants varies in different geographical locations
- This ratio, when measured in an animal, can identify the type of climate the animal lived in based on the kinds of plants it ate

X-Ray Fluorescence (XRF)

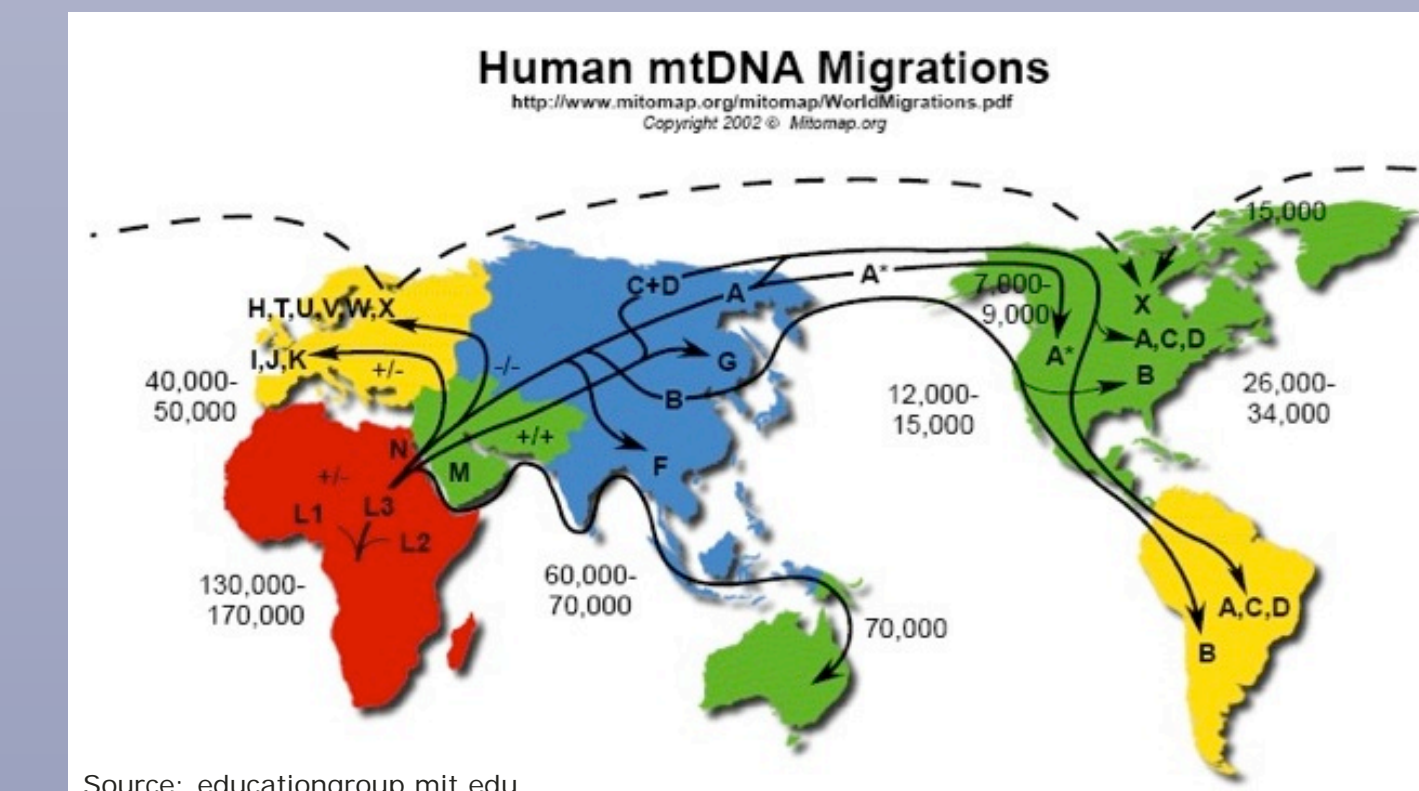
- Uses emission of secondary x-rays from samples excited with high energy x-ray or gamma rays to identify elemental composition
- Samples analyzed using a Horiba XGT-7200 XRF Spectrometer

Forensic mtDNA Analysis

ISU's Ancient DNA Extraction Lab and Molecular Research Core Facility specialize in extraction, amplification, sequencing and analysis of mitochondrial and genomic DNA from old, degraded, or fragmented samples.

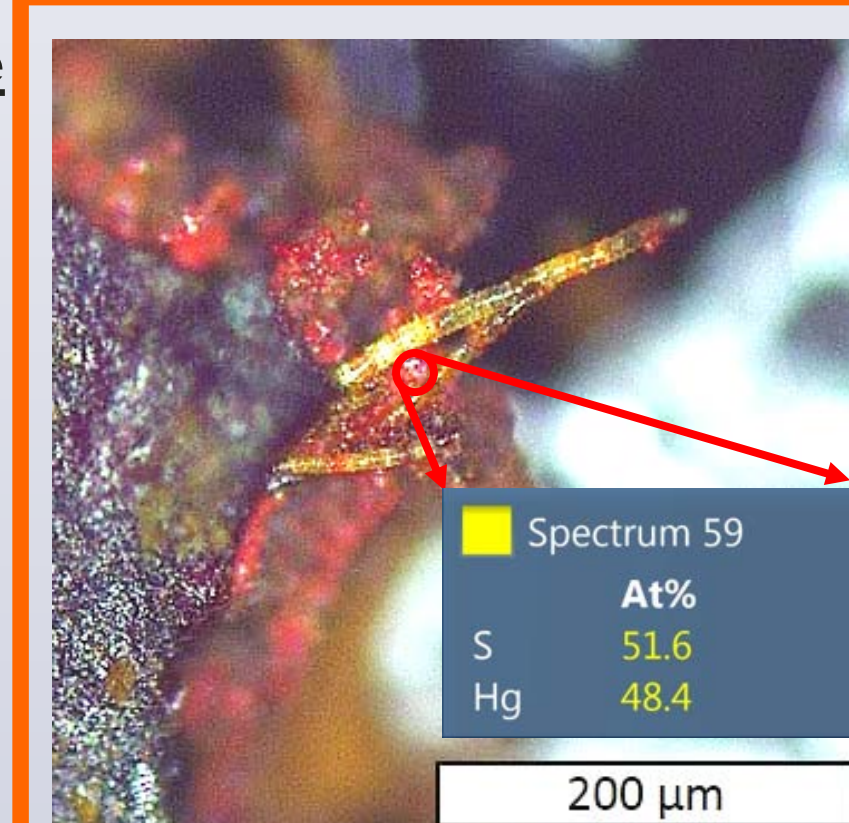


Radioactivity vs. time of Carbon-14
<http://www.chem.csustan.edu/chem3070/images/c14decay.gif>
<http://littlesbits.cc/browse-lessons/geiger-counter-and-radioactive-decay-half-life>



Source: educationgroup.mit.edu

Results and Discussion



Optical image of red pigment with EDS inset showing elemental atomic %

Red pigment identified as mercury sulfide (HgS)

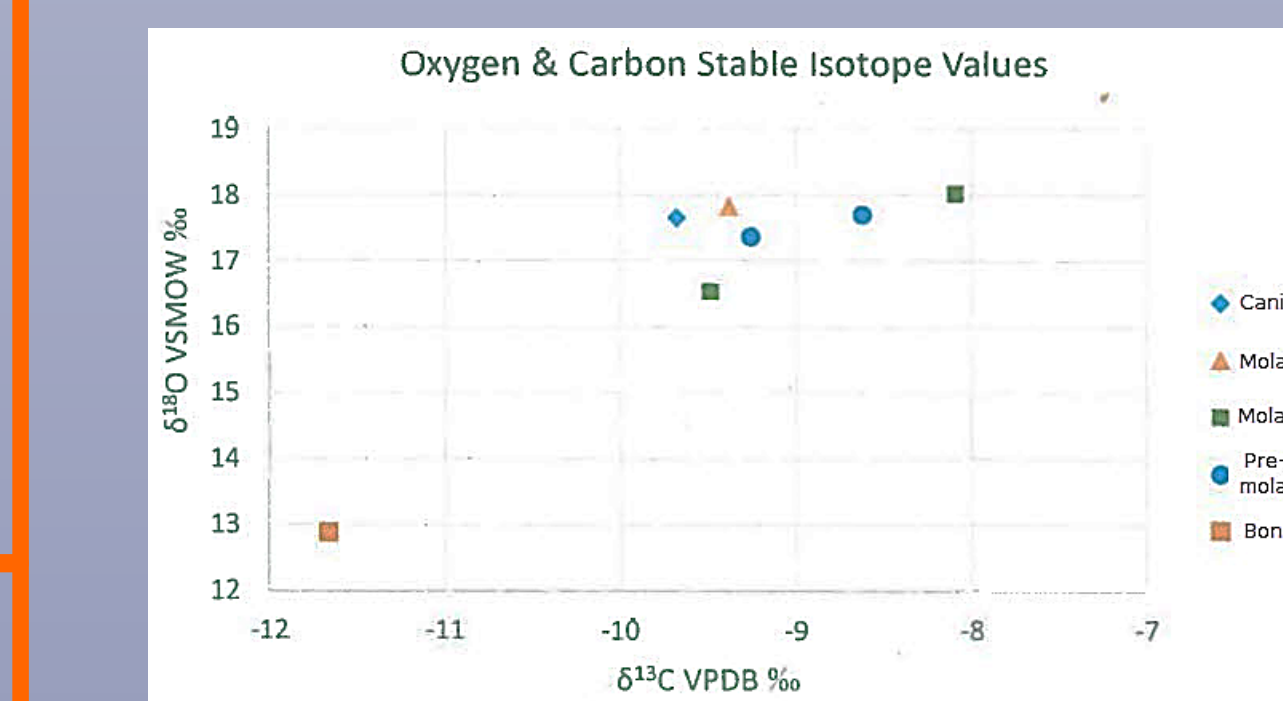
- Expected ratio of Hg to S would be 1:1
- Average ratio for all spectra was 1.12:1, possibly indicative of loss of sulfur due to microbial activity

| Major Trace Elements (mass %) | Minor Trace Elements (ppm) |
|-------------------------------|----------------------------|
| Silicon 12.7 | Phosphorus 8300 |
| Calcium 9.7 | Titanium 6000 |
| Iron 3.3 | Zirconium 5400 |
| Potassium 2.2 | Bromine 1400 |
| Dysprosium 1.1 | Nickel 1300 |
| Aluminum 0.9 | Manganese 1200 |
| | Europium 1200 |
| | Neodymium 800 |

| LA-ICP-QMS Red Pigment Trace Elements (ppb) | |
|---|---------------|
| Phosphorus 189,000 | Neodymium 180 |
| Titanium 57,000 | Lanthanum 90 |
| Potassium 47,000 | Dysprosium 75 |
| Cerium 330 | Europium 5 |

Stable Isotope analysis identified different values for teeth and bone

- Indicates individual lived in one area during youth and another area closer to death
- Bone value suggests C₃ type plants while tooth enamel is indicative of a mixed C₃/C₄ environment

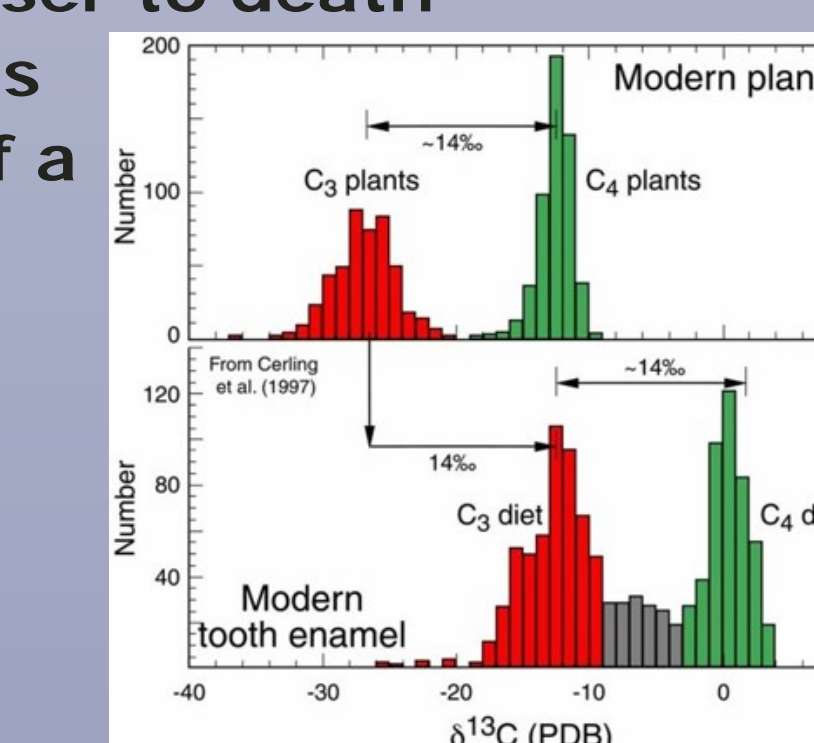


O and C stable isotope values from carbonate component of bone and teeth

- XRF identified major and minor trace elements also present in the HgS
- Suggests the cinnabar is of a silica/calcium or silicate mineral origin and not a refined mineral as would be found in artworks

LA-ICP-QMS is a more sensitive technique than XRF

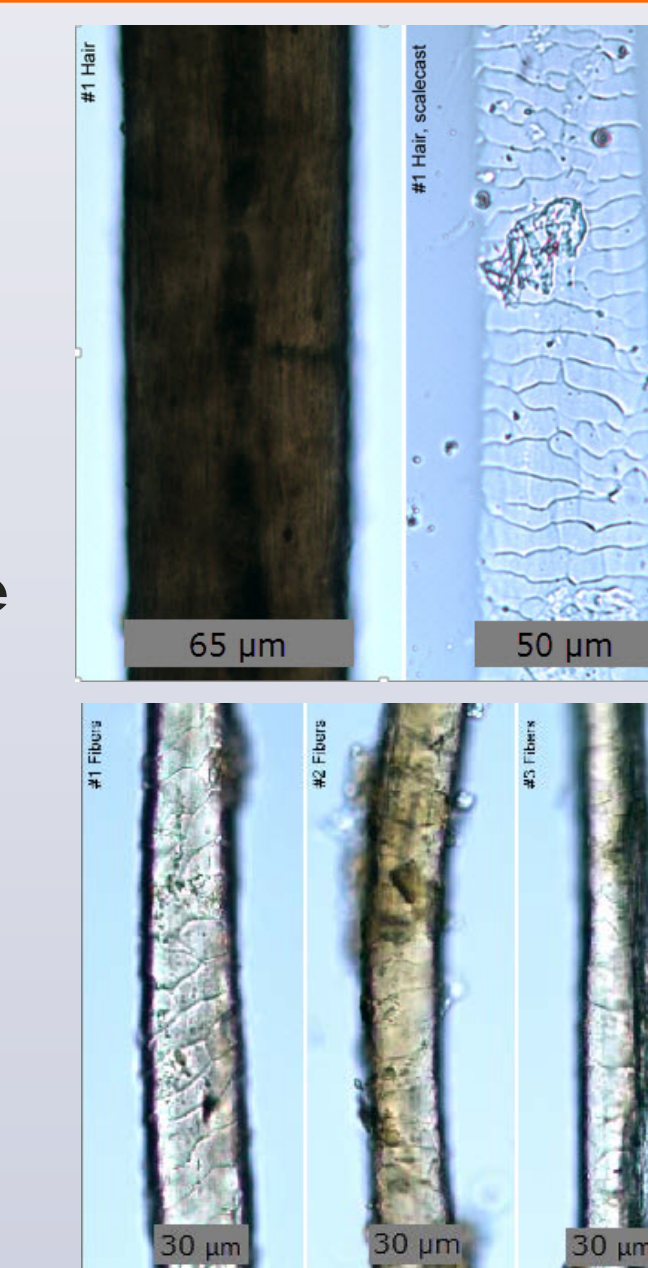
- Identified additional trace elements in red pigment
- Data can potentially be used to identify a geological cinnabar source



Relationship between carbon isotope values of C₃ and C₄ in modern plants and enamel

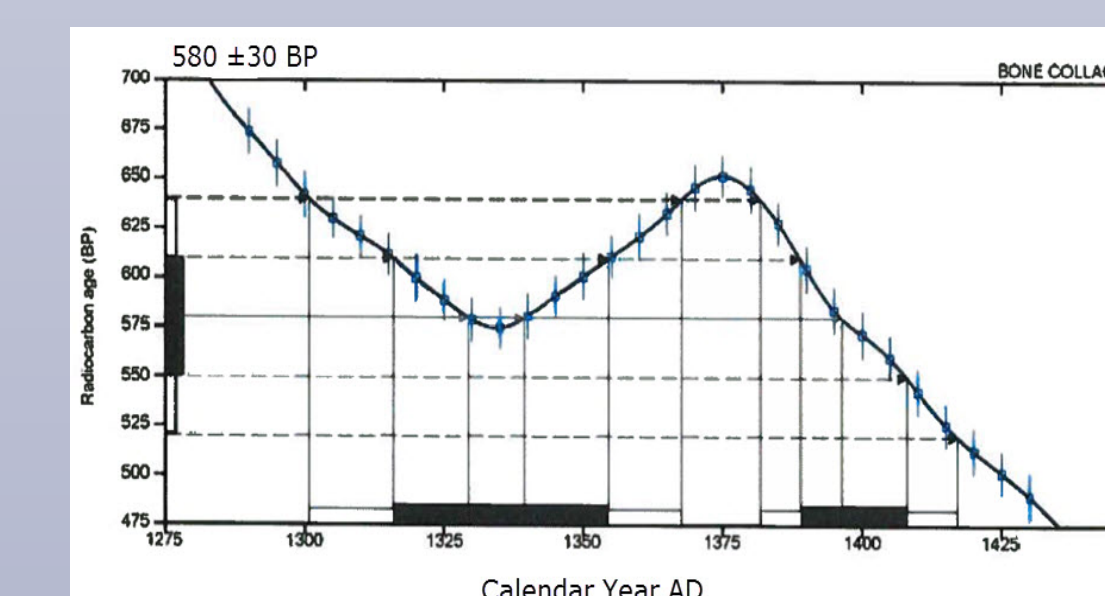
Hair and fiber samples sent for anthropological analysis to the University of Delaware Department of Art Conservation

- Hair samples (top) match the consistency of human hair
- Animal fibers (bottom) most likely sheep's wool or fine strands of cattle, deer, dog, goat, marmot or musk ox



Radiocarbon Dating

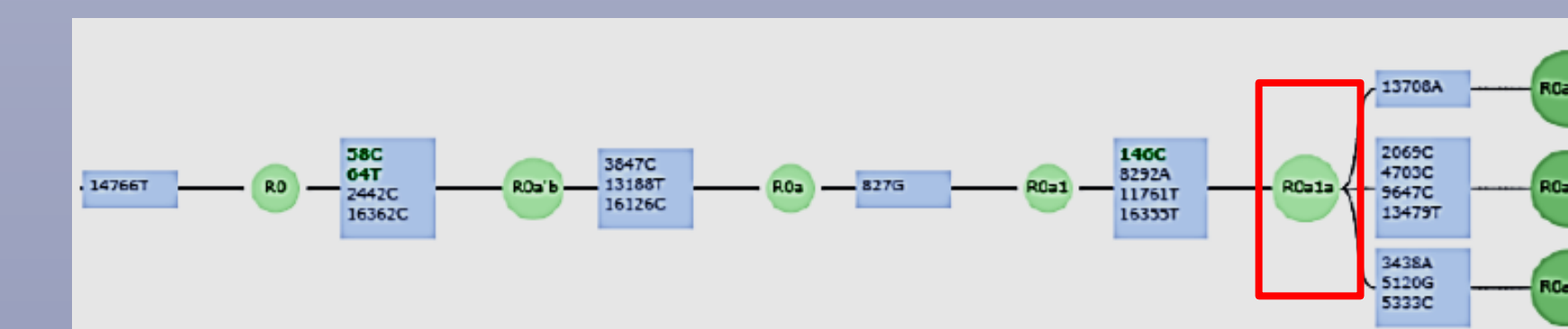
- Skull is ~550-620 years old
- Pre-Columbian (i.e. pre-European contact)
- Possibly of Chinook or Fremont culture, late pre-historic agricultural groups that extended into other states



Calibration of radiocarbon age to calendar years from Beta Analytic (Beta-393782) dating the skull to 1330-1400 AD

Forensic Mitochondrial DNA

- Haplogroup R0a1a identified, although it is not associated with known Native American populations
- Area where Haplogroup R0a1a originated is from the Arabian Peninsula or western Eurasia



Haplogroup sequence identifying the skull's lineage from a maternal contribution as being from the Arabian Peninsula or western Eurasia

Conclusions and Future Work

- Skull possibly of Chinook or Fremont culture
 - Cranial deformation and post-mortem application of HgS with adhered animal hair
- Trace elements in the HgS may potentially identify a geological source for the cinnabar
- Radiocarbon dated age ~ 550-620 years old
- Stable isotope analysis suggests habitation in different locations as a child and as an adult; locale characterized as cooler than would have been found in Idaho

- Forensic mitochondrial DNA identified haplogroup R0a1a which originated on the Arabian Peninsula; this is not consistent with the rest of the data in the study
- Future work will include
 - Additional trace element analysis from the bone to provide constraints to the duration of burial independent of C-14 dating
 - A secondary ancient DNA analysis to confirm or disprove the initial findings

Acknowledgments

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- [1] "Patterns of Distribution." *Patterns of Distribution*. Web. 6 Apr. 2015. <<http://www.geo.arizona.edu/Antevs/ecol438/lect04.html>>.