

Export/sampling permits

Please note an export permit must be linked to an object that has to be created on SAHRIS! If the object you want to work on has not been created yet, you would need to **create an ObjectID**.

Required documents:

- For export of material from KZN, Eastern Cape or Western Cape that involves destructive analysis, the **destructive sampling permit** from the respective Heritage Authority must be submitted;
- A consent letter from the accessioning institution.

The proposal should include (you can fill these in below):

- a list of participants (name, affiliation, phone no, email addresses) and how they are involved;
- the name and address of the facility, including address, it is being scanned at;
- name and address of the museum/university department that currently hosts the object;
- names of the responsible person(s) during transport and while the fossil is at the facility;
- the period/time frame during which the fossil(s) will be outside the country;
- detailed information on the fossil(s), especially as it is a "unique" specimen;
- detailed information on the research project behind it & methodology including expected outcomes (i.e., the reason for export);
- the written confirmation of the institution that currently hosts the object that the object may be used as proposed and be returned in good condition;
- should there be any damage/destructive analysis (e.g., coating for higher resolution) undertaken, this needs to be stated in detail;
- Statement why this study cannot be done in South Africa.

Applicant (name and affiliation): this is usually the museum curator!

Dr. Erich C. Fisher, Arizona State University

Applied for (principal researcher): Dr. Erich C. Fisher

Participants with affiliations, email addresses, phone numbers (& their role):

- 1) Dr. Zenobia Jacobs, Zenobia.jacobas@uow.edu.au; University of Wollongong, +61 2 4221 3663
Role: OSL Dating Specialist
- 2) Dr. Marion Bamford, marion.bamford@wits.ac.za; University of the Witwatersrand, 011 7176682
Role: Pollen sample preparation
- 3) Dr. Irene Esteban, irene.esteban.alama@gmail.com;
University of Witwatersrand, 011 7176682
Role: Phytolith specialist
- 4) Dr. Frank Neumann, fneumann1971@googlemail.com;
University of KwaZulu Natal, +49(0) 251 832 3934
Role: Pollen specialist
- 5) Dr. Enno Schefuss, eschefuss@marum.de, University of Bremen, +42(0) 421 218 65526
Role: Plant leaf waxes

The radiocarbon samples will be **couriered** to Arizona State University (facility/institution) in

February 2017 by FedEx to Arizona State University. The OSL samples will be couriered to the University of Wollongong using Fedex in February 2017 and received by Dr. Jacobs. The Pollen and phytolith samples will be couriered via Fedex in February 2017 to the University of the Witwatersrand in February 2017 and received by Dr. Bamford. The geochemistry samples will be couriered via FedEx to Dr. Schefuss in Peitermaritzburg and he will hand carry the samples to his laboratory in University of Bremen in April 2017. All samples will be shipped with a tracking reference number and signed upon receipt by the specialist mentioned in this permit. Drs. Jacobs, Bamford, Esteban, Schefuss, and Neumann (name) will be involved with the analysis (e.g., transport/scanning) of objects and sample preparation and analysis (whatever else).

Institution incl. address that currently hosts the object:

East London Museum
319 Oxford St.
East London, South Africa
5201
+27 43 743 0686

Facility incl. address at which the experiment will be done:

OSL: Centre for Archaeological Science, University of Wollongong. Bldg. 41, Northfields Ave, Wollongong, NSW, 2522 Australia

Pollen and Phytolith analysis: ESI, Paleosciences Centre, East Campus, University of the Witwatersrand, 1 Jan Smuts Ave, Braamfontein 2001, Johannesburg, South Africa

Radiocarbon Analysis: Beta Analytic Inc., 985 SW 74th Ct, Miami, FL 33155, United States, +1 305 667 5167

Geochemistry: MARUM – Centre for Marine Environment Sciences, University of Bremen, Leobener Str. 8, D-28359 Bremen, Germany

Table of objects or upload file: File has been uploaded

Site including age at which object was found:

Site A2SE-1, Lambasi District, Eastern Pondoland

Current radiocarbon analysis at Waterfall Bluff shows that deposits at the site date from 6-8 ka and from 11 ka.

Time frame:

Transport to ESI, ASU, Univ Bremen (facility): February 2017 (date) Return date: _____ (date)

Aim/rationale: The purpose of this application is to receive a permit to export scientific samples for dating and paleoenvironmental analysis. The samples were collected in 2016 during fieldwork specified

in ECPHRA permit No. 2/2/APM-PERMIT/15/03/001- and the samples are currently located at the P5 Project laboratory within the East London Museum. The requested samples include charcoal for radiocarbon dating and sediment samples for either Optically Stimulated Luminescence (OSL) dating or pollen and phytolith analysis. The samples were collected from the Lambasi district in Eastern Pondoland, Eastern Cape Province.

Methodology (short): The purpose of this application is to receive a permit to export scientific samples from the East London Museum (Eastern Cape Province) for dating and paleoenvironmental analysis, as per the research agreement specified in permit No. 2/2/APM-PERMIT/15/03/001-.

The samples were collected in 2016 during fieldwork specified in permit No. 2/2/APM-PERMIT/15/03/001-. No archaeological materials (fauna, stone artifacts, etc) were observed from any samples collected from archaeological deposits. The fieldwork was conducted at site A2SE-1 in eastern Pondoland (Lambasi District, Eastern Cape Province), which is described in Fisher (2016) and Fisher et al. (2015).

The bulk sediment samples that were taken from archaeological sediments were collected using a hand trowel and most samples were plotted with a digital total station. Approximately 100 g of sediment was collected per sample and the sediment was subsequently divided in our laboratory into samples that will be processed for phytoliths, pollen, and sediment geochemistry / isotopes / plant leaf waxes. The pollen and phytolith samples will be sent to University of the Witwatersrand, Johannesburg to be processed by Dr. Marion Bamford in her laboratory. Once the samples are mounted to microscope slides they will then be sent to Dr. Frank Neumann at the University of KwaZulu-Natal who is involved in archaeo-palynological studies across the Eastern Cape and Kwa-Zulu Natal. The phytolith samples will be kept at University of the Witwatersrand where they will be studied by Dr. Irene Esteban. Details of this method are described in (Katz et al. 2010). The geochemistry samples will be collected by Dr. Enno Schefuss and hand carried to his laboratory at the University of Bremen. There, the samples will be analyzed for plant leaf waxes, plant isotopes, soil pH, and ICP-MS to determine the elemental composition of the sediments.

Charcoal samples for radiocarbon dating were identified during archaeological excavations. The 3D location of the samples was plotted with a total station and then collected with a sterile trowel and placed into a sterile plastic cube. All of the samples that have been submitted for analysis in this permit are from areas and deposits that have sister samples in our archives. The radiocarbon samples will be couriered to Dr. Erich Fisher in the United States where they will then be couriered to Beta Analytic Co. for AMS dating. Beta Analytic has a rapid turnaround time and excellent track record for AMS dating.

OSL samples were collected by inserting a 5 cm diameter PVC tube into the sediments approximately 20 cm deep. The samples were removed directly into light-impenetrable black plastic bags and sealed tightly with duct tape. The top, middle and bottom of each sample was plotted with a total station which allows us to project the 3D location of the sample in the archaeological sections. OSL samples will be shipped to Dr. Zenobia Jacobs at the University of Wollongong. The OSL Laboratory at the Centre for Archaeological Science at the University of Wollongong is a world-leader in OSL dating. Dr. Jacobs is also actively involved in dating numerous stone age sites across South Africa.

Confirmation/permit by museum (Attached?): YES

Damage/destructive analysis? (if yes, explain in detail)

PHYTOLITH METHODS

Between 30 and 50 mg of the sediment will be placed into a 0.5 ml Eppendorf plastic centrifuge tube. Carbonate minerals and carbonated hydroxylapatite will be dissolved in 50 microliters of 6 N HCl using a micropipette (Finnpipette). 450 ml 2.4 g/ml sodium polytungstate solution $[\text{Na}_6(\text{H}_2\text{W}_{12}\text{O}_{40})\cdot \text{vH}_2\text{O}]$ will then be added after the bubbling has ceased. The tube will be vortexed and sonicated for ~10 min (Ultrasons, Selecta), vortexed again, and then centrifuged finally for 5 min at 5000 rpm (MiniSpin plus, Eppendorf). The supernatant (phytoliths and charred organic material) will then be removed to a new 0.5 ml centrifuge tube and vortexed one final time. Immediately after vortexing, a 50 ml aliquot of the supernatant will be placed on a microscope slide and covered with a 24 mm x 24 mm cover-slip. This slide will then be examined under the optical microscope.

PALYNOLOGICAL ANALYSIS

Palynomorphs will be concentrated within the sediment samples using 30% HCl, KOH, 48% HF and acetolysis. Sodium polytungstate will be used for heavy liquid separation. The residues will then be sieved over 10 μm and 250 μm mesh screens. The pollen samples will then be mounted in glycerol jelly. Lycopodium spores will then be added to all samples in order to calculate pollen and charred particle concentrations, which will be expressed as the number of grains and fragments per gram sample. Identification and counting will be performed with a light microscope under 400x and 1000x magnification.

RADIOCARBON ANALYSIS

The sediments will first be floated and agitated in deionized H₂O to disperse the sediment. They will then be progressively sieved through 250 and 180 micron sieves to remove any plant macrofossils or rootlet material prior to the pretreatments. The < 180 μ size fraction will then be subjected to a series of hot (near boiling) 0.5N HCl leaches to remove any carbonate presence. The samples will then be rinsed with deionized H₂O until neutral and dried in a 70C oven overnight. The pretreated sample material will then be homogenized and inspected under a 45x microscope to inspect for any rootlet hairs or fragments and a portion will be tested with acid again to ensure that all carbonate has been removed. The samples will then be combusted to CO₂ and graphitized for AMS counting. Quality control results will be corrected for isotopic fractionation and measured against the NIST SRM-4990B standard.

OSL DATING

A combination of single aliquot and single grain OSL dating will be used to obtain ages. Single aliquots will be measured in the first place, followed by a more detailed investigation using single grains. Using

single grains, it is possible to explicitly investigate the potential causes of the observed overdispersion (e.g. Jacobs 2005; Jacobs et al. 2006b; Jacobs and Roberts 2007). A single grain of sand is the smallest meaningful unit of measurement in OSL dating—which provides an estimate of the time elapsed since a grain was last exposed to sunlight—because each grain has experienced its own history of erosion, transport and deposition.

The sample tubes will be opened under dim red light. Sediment at both ends of each tube will be discarded (as it would have contained grains exposed to sunlight at the time of sample collection), and quartz grains will then be extracted from the light-safe portions using standard preparation procedures (Aitken 1998; Wintle 1997). First, all samples will be wet sieved to isolate grains of 180-212 μm in diameter. Second, carbonates still present in the fraction will be dissolved in 10% hydrochloric acid and then organic matter will be oxidized in 30% hydrogen peroxide solution. The remaining sample will be dried and feldspar, quartz and heavy minerals will be separated by density separation using sodium polytungstate solutions of 2.62 and 2.70 specific gravities, respectively. The separated quartz grains will be etched with 48% hydrofluoric acid for 40 min to remove the alpha-irradiated rind of each quartz grain and to destroy any remaining feldspars, and then rinsed in hydrochloric acid to remove any precipitated fluorides, dried and sieved again. Grains retained on the 180 μm diameter mesh will then be used for dating. We prefer the 180-212 μm in diameter grain-size fraction for measurement of the equivalent dose to exploit the advantages and ease of single-grain measurements using standard equipment.

Geochemistry / Plant Leaf Waxes

Plant leaf waxes will be recovered from filter samples at the University of Bremen, Germany by accelerated solvent extraction (Dionex ASE 200) using dichloromethane (DCM): methanol (MeOH) 9:1 solution at 1000 psi and 100 °C and three 5-min static phases. Extracts will be dried under nitrogen and hydrocarbons will be separated from leaf waxes using pipette columns filled with ~1.5 g silica gel (0.063–0.200 mm, 1%-deactivated by weight with water) and eluting with 4 mL of hexane. Elemental analysis will be conducted using the Total Digestion method and Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). Soil pH will be determined by mixing sediment in water and measuring the pH of the water slurry.

Statement why this study cannot be done in South Africa: The analyses in question rely on as many South African institutions as possible in the preparation of the samples. The researchers involved with the study are also either South Africa nationals or have long-standing involvement with scientific research in South Africa. The OSL analysis is being conducted at the University of Wollongong because it has state-of-the-art facilities for OSL dating that will ensure accurate and reliable age results. The pollen and phytolith samples will be processed and analyzed at South African institutions. The geochemistry / plant leaf wax analysis will be conducted at the University of Bremen, which has state-of-the-art facilities for these purposes and it is also the host of the RAIN project, which is another major research project focused on South African climates and environments. The radiocarbon analysis will be conducted in the US because Beta Analytic Inc. has a rapid turnaround for AMS dating and excellent AMS results.