

STANDARD OPERATING PROCEDURE

SOP NUMBER	SOP TITLE
CAR-016-01	WET SIEVING OF CLAY SAMPLES FOR ARCHAEOLOGICAL
	RESEARCH

PART 1

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PART 3

Authoriser	Date of Issue: 20/12/2021
Mr Simon Sammut University Secretary – Office of the Secretary Rectorate	Date of next revision: 20/12/2025

PART 4 (To be filled in by OOS, QSU or RSSD)

 This procedure has been revised and is no longer valid as from: 	Date of NEXT REVISION is extended until:	□ SOP rendered obsolete on:
(Write date)	(Max. 4 years)	(Write date)

1. Reason for revision

1.1. New SOP

2. Purpose and scope

2.1. To describe the procedure for the wet-sieving of clay samples for the scope of archaeological research including experimental archaeology and characterization.

3. Definitions

- 3.1. **SOP**: Standard Operating Procedure.
- 3.2. Clay samples: The term clay is here used to describe clay-rich sediments. This SOP adopts an archaeological perspective of clay defined as the raw material for pottery (Cuomo Di Caprio 2017, 37). These sediments therefore include sand and silt size particles which are non-clay minerals as well as other incidental materials (e.g. microfossils, organic material etc.). This is different from a mineralogist perspective which only considers clay as specific silicate minerals.
- 3.3. Wet-sieving: Wet-sieving is different from dry sieving as it uses water to wash the sample through the sieves without any crushing or other preparation of the sample. Both approaches can be used when studying raw material provenance (Hein and Kilikoglou 2020, 180).
- 3.4. University of Malta (UoM) abbreviations: CAR: Department of Classics and Archaeology; CHE: Department of Chemistry; MME: Department of Metallurgy and Materials Engineering

4. Responsibilities

- 4.1. It is the responsibility of staff and students carrying out sieving with the Department of Classics and Archaeology of the University of Malta to read and follow this SOP.
- 4.2. It is the responsibility of the staff and students using the equipment to wash and store it properly after use according to the specific regulations of each department owning the equipment.
- 4.3. It is the responsibility of staff and students carrying out wet-sieving to ask the appropriate authorisation of the Department of Classics and Archaeology, the Department of Chemistry and Department of Metallurgy and Materials Engineering for borrowing and using the appropriate equipment, as well as following training on the use of equipment if deemed necessary.
- 4.4. It is the responsibility of staff and students carrying out wet-sieving to minimize their environmental impact of the process, such as waste of water.
- 4.5. It is the responsibility of the Department to make sure that the students have undergone the safety briefing session.

5. Health and Safety Requirements

5.1. During sieving:

- 5.1.1. Sieving requires lifting heavy buckets and samples. Students and staff should care for their backs and lift following lifting recommendations such as bending their knees and keeping the back straight as well as not lifting heavier than one feels comfortable lifting.
- 5.1.2. Loads weighing more than 25 kgs should be handled by 2 persons or more.
- 5.1.3. Care has to be taken not to trap fingers between the sieves and buckets, especially when working as a group.

5.2. During filtration:

- 5.2.1. Filtration is carried out in a laboratory environment. Usual health and safety rules are to be followed including: wearing safety goggles and laboratory coat, wearing long trousers and stable closed shoes, not eating/drinking in the laboratory, not leaving unattended equipment on (e.g. the pump). No food and drink should be taken inside the laboratory as they would have to be thrown away.
- 5.2.2. Care should be taken to allow the pump to cool down when it is used for a long time as it could overheat and get damaged.
- 5.2.3. Staff and students should not touch or open any chemicals in the laboratory.
- 5.2.4. Care should be taken that the glassware is intact (no cracks) when being used particularly during vacuum and that the set-up is correct (e.g. no leaks) before using it.
- 5.2.5. Care should be taken that the whole set up is stable before using it.
- 5.2.6. The researchers should consider taking sufficient breaks outside the lab to drink water and to interrupt exposure to the continuous noise of the pump. Ear protectors may be recommended if the noise level is too high or exposure too long.
- 5.2.7. Additional health and safety measures indicated by the Department of Chemistry should be consulted, and training should be carried if necessary.

5.3. During drying:

- 5.3.1. When using the drying oven, glassware will get hot, and one should ensure that they are wearing suitable heat resistant protective equipment (e.g. gloves) to handle the glassware, or allow the glassware to cool down in the open oven.
- 5.3.2. The oven plates also get hot and one should be careful not to burn themselves or leave the oven cool down before getting the samples out.

5.4. During fraction collection:

5.4.1. If using a drying oven or a hot plate to dry the fractions collected on the sieves, care should be taken to wear suitable heat protection.

5.5. During sieve cleaning with an Ultrasonic bath:

- 5.5.1. The drying basin of the Ultrasonic bath heats the sieves and trays to a very high temperature. One should not touch the metal when opening the dryer and should wear heat protection, or wait until the tray cools down.
- 5.5.2. Care should be taken when using the pressure gas nitrogen in case of overpressure. Nitrogen could cause suffocation. In more general terms, the SDS (Safety Data Sheet; see Merck 2019) of nitrogen should be followed.
- 5.5.3. Health and safety measures indicated by the Department of Metallurgy and Material engineering should be consulted, training should be carried if necessary.

5.6. First Aid Procedure

- 5.6.1. In case of clay particles in the eye, do not attempt to take it out but rinse the eye with plenty of fresh clean water, and contact a first aider for further assistance.
- 5.6.2. In case of a medical emergency, if the person involved suffers a minor injury summon a First Aider to assist, in case of grievous injuries phone 112 and request an ambulance to be dispatched.

6. Procedure

- 6.1. The procedure is detailed by stage, including equipment list. Different stages of this procedure will occur in different location on campus.
- 6.2. Before starting the procedure, any collected clay samples should be documented appropriately as specified in the SOP "Clay and soil sampling for archaeological research using a handheld auger" written by the Department of Classics and Archaeology.

6.3. Considerations:

- 6.3.1. This procedure was partly inspired by Cordell et al. (2017, 99-100) sieving method for grain size analysis.
- 6.3.2. Time-keeping: The procedure takes a considerable amount of time, although some of this time is passive (e.g. when the samples are drying). Table 1 presents the processing time for 1.5kg of a compact sample based on the equipment described in this SOP. This time might be reduced by changing the equipment (e.g. more trays for the drying oven).

 Table 1: Example of timetable created when planning the sieving procedure for 1.5kg of sample. *The equipment might also be
 loaned out.

Procedure step	<u>Soaking</u> <u>samples:</u> Step A	<u>Sieving:</u> Step B	<u>Collection of</u> <u>fractions:</u> <u>S</u> tep C	<u>Filtration:</u> Step D	<u>Drying:</u> Step E	<u>Cleaning:</u> Step F
Time consideration	1 hour then 3 days to soak with no further action	5 hours for 5 sieves	If sieves are dry, 2 hours	3 days	30 hours total, mostly unattended, using 2 trays	2 hours for 5 sieve sizes, mostly waiting time
Location	CAR	CHE	CAR	CHE	MME	MME

6.4. Step A: Soaking the samples

6.4.1. Equipment:

- 6.4.1.1. Clean containers that can be closed: the containers should be large enough for the sample to be completely covered in water. One or several containers can be used according to the sizes of containers. A marker is required to label the containers.
- 6.4.1.2. Calibrated electronic scale (at least 0.01g precision), calibration weight adapted to the electronic scale and a measuring cylinder (1L).
- 6.4.1.3. If using only a portion of the sample: a large low tray and a plastic scraper.

6.4.2. Procedure for the soaking of the samples:

6.4.2.1. Samples should be soaked for at least three days so that it will wash through the sieves. The empty containers are first rinsed with water. Deionised water would

ideally be used but the procedure has been, in the past, carried out with tap water.

- 6.4.2.2. The sample, or the part of the sample which will be sieved, should be weighed (unsieved weight = uw), placed in a labelled container.
- 6.4.2.3. The samples are then covered fully with tap water.
 - 6.4.2.3.1. Minimum: Ration clay:water (1:1) in weight
- 6.4.2.4. The containers are then closed and left aside for three days.
- 6.4.2.5. If only part of the sample is sieved, then a portion of the sample is selected. For this, the sampling methodology described in Pirone (2017) can be followed. This method aims at selecting a portion of the sample which is representative of the whole sample.
 - 6.4.2.5.1. The complete sample is first put in a low and clean tray.
 - 6.4.2.5.2. Using a plastic scraper, the sample is mixed for at least a few minutes.
 - 6.4.2.5.3. The sample is then flattened and divided into four quadrants (Figure 2 and 3), tracing the quadrant boundaries with the scraper.
 - 6.4.2.5.4. Two opposite quadrants (A and B in Figure 3) are put back in the original sample bag.



Figure 1: The sample is divided in quadrant and A and B are removed and placed back in their sample bags



Figure 2: One of the quadrants is weighed

6.4.2.5.5. The two quadrants left in the clay are mixed together and the procedure (mixing, diving in quadrant and removing) is repeated until one resulting quadrant is of the desired weight. This is confirmed using the calibrated electronic scale (Figure 3).

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6.4.3. **Recording of procedure**: The weight and the amount of water added as well as the sample details (unique sample name, location...) should be recorded in a spreadsheet/database. The sample details should also be written down on the container.



Figure 3: The soaking equipment. 1: initial sample; 2: electronic scale; 3: written record; 4: soaked samples in labelled plastic boxes

6.5. Step B: Wet-Sieving

6.5.1. Equipment:

- 6.5.1.1. Sieves of size mesh: 4 mm, 2 mm, 1 mm, 500 μm, 250 μm, 125 μm, 63 μm, 45 μm. The department of Classics and Archaeology owns two sets of sieves of different sizes. The set used for wet sieving are of brand Cisa (200 mm Ø, 50 mm H), stainless steel frame.
 - 6.5.1.1.1. Researchers should consider the meshes size they need according to their research questions. If partial grain size analysis would be performed, then the sizes of sieves should be equated with the Wentworth scale (see Rice 2015, 41-42) although the current set only allows sieving down to the fine sand-size particles (63 μm).
 - 6.5.1.1.1.1. Some samples might be fine enough to not be sieved at 4 mm or 2 mm for example.
- 6.5.1.2. Tap water (or ideally DI water) and a clean pipe linked to a tap.
- 6.5.1.3. At least two clean buckets with a diameter larger than the diameter of the sieves. The person holding the sieves in the bucket should be able to hold it comfortably with two hands inside the bucket. The volume of the buckets should be of a minimum 10L.
- 6.5.1.4. A toothbrush and a plastic scraper to help collect the clay.
- 6.5.1.5. A basin larger than the diameter of the bucket to place the bucket inside and minimize loss.
- 6.5.1.6. Recording equipment: A phone or camera, notebook and pen, markers to write on the buckets.

6.5.2. Considerations:

6.5.2.1. This step can be messy, and sediment might damage notebooks or cameras.

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- 6.5.2.2. At least two people (Person 1 and Person 2) are required for this step although a third person (Person 3) might be required to provide clean water and record information and photographs.
- 6.5.2.3. The sieves need to be used from the larger mesh size to the smallest mesh size chosen.

6.5.3. Procedure for the wet-sieving:

- 6.5.3.1. **Setup**: (see Figure 5) A bucket should be placed in a larger basin to collect any water loss. The larger mesh size is then chosen and Person 1 holds the sieve inside the bucket at the top.
- 6.5.3.2. Person 2 empties the container with the clay inside the sieve. The clay might not wash through easily so fresh water can be added at low pressure to help wash the clay through the mesh. Using too much fresh water, however, will slow down the next steps of the process, and may require the use of several buckets.
 - 6.5.3.2.1. Person 2, using their clean hands, can spread the clay within the sieve applying little pressure to help the clay wash through.
 - 6.5.3.2.2. Person 1 can hold the sieve on the surface of the water contained in the bucket to unclog the sieve and help the clay wash through. Slow lateral and vertical movements of the sieve on the surface of the water can be performed.
 - 6.5.3.2.3. From time to time, the bottom side of the sieve should be unclogged using the hand or a toothbrush and the clay should be left falling in the bucket.
- 6.5.3.3. This procedure needs to be repeated as long as necessary in order for the remaining clay to wash through completely. This can be observed from the colour of the water going through the sieve which should be clear. The natural inclusions left on the sieve should be distinguishable in colour and shape with no adhering sediments.
- 6.5.3.4. This whole procedure needs to be repeated with each sieve chosen by the researcher. The sieves need to be used in decreasing mesh size.
- 6.5.3.5. The buckets containing the clay that washed through all chosen sieves should be closed and labelled (sample ID and information, particle size). The particle size written own is: < last mesh-size used (e.g. <45 μm).</p>
- 6.5.3.6. The bucket(s) should be left to sediment at least overnight. This will allow the syphoning of the water and filtration.
- 6.5.3.7. The sieves should be left to dry, ideally inside a lab and covered, for a few days (see Step C).



Figure 4: Sieving procedure and equipment. A bucket (2) is placed in a tray (1) whilst the sieve (3) is held on top of the bucket by Person 1 (4). Person 2 (5) is breaking the lumps of clay.

6.5.4. Recording:

- 6.5.4.1. Written record: the mesh-sizes used for each sample sieved should be recorded in a spreadsheet/database. Information about the sample (colour, inclusions) should be recorded as well as: possible contamination (e.g. dust from construction) and water/clay loss (e.g. water fell outside the bucket). This information should be recorded immediately after sieving.
- 6.5.4.2. The buckets should be labelled with the name of the sample, the size range, the date and initials of researcher (see Figure 6).
- 6.5.4.3. The photographic record should show: any specific features that have been noted (e.g. colour of inclusions) or problems encountered.



Figure 5: Labelled bucket with minimum information: sample name (QL.B), particle size (<last mesh size), initials.

6.6. Step C: Collecting the fractions.

The sieving process would have left natural inclusions on the sieves which need collecting.

6.6.1. Equipment:

- 6.6.1.1. An aluminium tray bigger than the diameter of the sieves, an A3 sheet of white paper.
- 6.6.1.2. A digital scale (0.01g precision at least).
- 6.6.1.3. Tweezers, a soft tooth brush and fine paint brushes can be used to collect the inclusions that are wedged in the sieve.
- 6.6.1.4. Sample bags and markers, a notebook and pen.
- 6.6.1.5. Good lighting and a clean working space.
- 6.6.1.6. A magnifying glass can be used to look at the inclusions.
- 6.6.1.7. A hot plate can be used to assist drying.

6.6.2. **Procedure for the collection of fractions**:

- 6.6.2.1. The sieves need to be left to dry and contamination should be avoided (e.g. by covering the sieves). A hot plate can be used to slowly heat the sieve to assist drying of the fraction.
- 6.6.2.2. Sample bags should be labelled following the data management plan of the research project. Examples of necessary information can be found in section 6.6.3. The empty labelled sample bags should be weighed (empty weight = ew) individually and the weight should be recorded.
- 6.6.2.3. **Set up**: The aluminium tray should be placed on a clean working surface. An A3 paper sheet (pre-folded in the middle) should be placed above the tray.
- 6.6.2.4. The sieve should then be placed upside down on this set up (Figure 6).
- 6.6.2.5. To collect as much of the fraction as possible, the following actions can be repeated.

- 6.6.2.5.1. Tapping gently on the sides of the sieves
- 6.6.2.5.2. Tapping the sieves gently against the tray.
- 6.6.2.5.3. Applying tooth brush or paint brush without excessive pressure, to avoid damaging the mesh. On the bigger meshes (>500 μ m), a slightly harder brush can be used if needed.
- 6.6.2.5.4. Tweezers can be used for the larger size meshes.
- 6.6.2.6. When this process is finished, the A3 paper can be folded in the middle, tilted and emptied in the appropriate sample bag.
- 6.6.2.7. The bag containing the fraction should then be weighed (total weight: tw) and the weight of the empty bag (ew) should be deducted. The weight should be recorded on the sample bag as well as in the written record. The proportion of the fraction over the full sample can then be calculated.
 - 6.6.2.7.1. Fraction weight (fw) = tw ew
 - 6.6.2.7.2. Proportion = fw/uw*100
 - 6.6.2.7.3. **Key**: tw: total weight (bag filled with fraction); ew: empty weight of the sample bag; fw: fraction weight as calculated in 6.6.2.7.1; uw: unsieved weight.
 - 6.6.2.7.4. An example is given in Table 2.
- 6.6.2.8. This procedure is repeated with each sieve.
- 6.6.2.9. At this stage, some inclusions might be selected for further analysis or characterization. After weighing the bag, they can be extracted and places in smaller sample bags with the same information written down on the latter.

6.6.3. Recording:

- 6.6.3.1. The database/spreadsheet created previously should be updated with the weight of each fraction. Description of the inclusions as well as the general sample colour can also be recorded.
- 6.6.3.2. The sample bags should be carefully labelled. Information to record: the sample name, initials, date, weight of the fraction, sieve size range.

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Figure 6: A pre-folded A3 sheet of paper is placed on a tray. The sieve is then placed upside down for the inclusions to fall on the paper.



Figure 7: A paint brush can be used to detach the inclusions from the sieve.

Table 2: Example of record for all fractions from one sample (QLA1). Produced by A. Humann and slightly				
modified for this SOP.				

	QL.A1		
fractions in μm	weight in g (fw)	proportion in %	
Sample weight before soaking (uw)	104.16	100	
> 1000	6.97	6.69	
1000 - 500	3.87	3.72	
500 - 250	3.45	3.31	
250 - 125	2.88	2.76	
125 - 63	4.53	4.35	

63 - 45	1.63	1.56
< 45	67.2	64.52
Total weight after sieving procedure	90.53	86.91

6.7. Step D: Filtration

6.7.1. Considerations:

- 6.7.1.1. This process is used to reduce the loss of finest particles after sedimentation. The researcher should assess if this step is necessary to their research project as well as the potential loss of information if not carried out. As this process is time consuming, the research team should balance the loss of information and the research time-frame.
- 6.7.1.2. If the process of filtration is not performed, the water is removed after sedimentation using pipettes and then discarded. After a first removal of water, the sample can be left to sediment an extra few days. This will allow more water to be siphoned decreasing the drying time (Step E). Some very light particles will, however, be lost.
- 6.7.1.3. This process has been performed using equipment loaned out by the Department of Chemistry. Authorisation has to be obtained before using the set-up. Training on the equipment would have to be provided.

6.7.2. Equipment:

- 6.7.2.1. *Filtration set-up*: Sealing gaskets (2 sizes to adapt to the funnels), two sizes of Büchner funnel (2 sizes to adapt to the filter, one big and one small), two sizes of Büchner flask (500ml and 2L), Vacuum pump (Portable Vacuum pump oil free). The Chemistry Department owns a BIOSBAS BIOUSRTY (SHANDONG) CO. LTD Model: GM 0.20 which has been used in the past. A bigger and more powerful pump would, however, be more efficient. Vacuum pipe, one-neck bottle with a tap (ideal 3-way tap), 2 Stands with muffles and clip. Forced ventilation directed at the pump can be used to cool the pump down.
- 6.7.2.2. *Filters*: ideally, the filters used would go down in size below 1µm to catch all clay particles. 3 3 sizes of filters can be used in a decreasing order: Filter 11µm (Whatman filter papers, 90 mm diameter, Cat No 1001 090), Filter 2.7 µm (Whatman filter papers, type 542, hardened Ashless), <u>Filter 0.45 µm (SUPLECO Analytical Nylon 66 Membranes 0.45 µm and 47 mm diameter</u>).
- 6.7.2.3. *Other equipment*: beakers, clock glasses, Full pipette and rubber bulb, Pasteur pipette and bulb, baking glass trays (at least 3.5L), chemistry metal spatula, tray to collect and dry the filters (aluminium tray for example).
- 6.7.2.4. *Safety equipment*: lab coats and safety goggles. Ear-protections might be considered if the pump is used for a long time.

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6.7.3. **Procedure for the Filtration**:

- 6.7.3.1. Set up (Figure 8):
 - 6.7.3.1.1. Care is to be taken that the set-up, especially the Büchner flask, is fixed to the stand and the pipes are strong enough to resist pressure of the vacuum.
 - 6.7.3.1.2. Filter papers of the biggest size (11 μ m) should be put in the funnel to cover all holes. The filter will be used in decreasing size (11 μ m then 2.7 μ m then 0.45 μ m).
 - 6.7.3.1.3. To ensure that the set-up works, there should be no holes in the system, and this can be verified by putting a hand on top of the Büchner funnel covering the funnel completely: suction should be felt and the pump will make a slightly different noise. Water is added in the funnel such that the filter is covered with a thin layer of water (this is also necessary to wet the filter) and the pump is then switched on.



Figure 8: Diagram of the filtration set-up; Image modified and translated from Hüniget al. (2006, 133).

- 6.7.3.2. The first step is to extract as much water as possible from the settled sample without disturbing the sedimentation. Beakers and pipettes can be used to scoop the water which should be collected in a separate bucket. This water will be filtered first (Figure 9).
- 6.7.3.3. This clearest water, containing the least soil, should be filtered first as too many particles will clog the filter.
- 6.7.3.4. The water is put in the funnel. The filter then separates the particles and the water.
- 6.7.3.5. This process is to be repeated with all the water that can be collected from the bucket. The whole cannot be filtered completely as the clay will eventually clog all filters. The point here is to filter as much top water as possible to minimize the loss of particles and collect them. The remaining sample will be dried as such.



Figure 9: The water with the least soil particle (identified by its clear colour) should be filtered first to avoid clogging the filter too fast.

- 6.7.3.6. Every time the Büchner flask is ¾ full, it is to be emptied in a bucket/container and put aside. This water will then be filtered with the next filter size (2.7μm).
- 6.7.3.7. The whole procedure is to be repeated with the three filter sizes to collect all particles above 0.45 μ m.
 - 6.7.3.7.1. Filtrating all the water with 0.45μ m is only recommended if the pump used is powerful enough. For the pump cited in this SOP, it is time consuming and might break the pump. Alternatively, after the filtration process with the 2.7 μ m filter, the water will still contain small particles which are visible to the eye. These can be left to settle for at least two days. The top water can then be discarded in the sink and the bottom water where the particles have settled can be filtered with a filter of size 0.45 μ m. If this alternative is chosen, it is to be acknowledged in the methodology.
- 6.7.3.8. Each filter collected needs to be dried on a tray.
- 6.7.3.9. If any holes appear in a filter, it signifies that the filter needs changing.
- 6.7.3.10. When the filters are dry, the clay should be collected in labelled sample bags by scraping the filter paper with a plastic spatula making sure that no fragments of papers contaminate the samples.
- 6.7.3.11. After the full filtration using the smallest filter (0.45μm), the water can be discarded in a drain or for watering plants. Both options are only allowed, if there is no contamination by chemicals.

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- 6.7.4. **Recording**: The procedure itself can be recorded in pictures. Track should be kept of the buckets/beakers containing the filtered water (and the size of filter used) as well as the bucket containing the remaining sediment to be dried as such.
- 6.8. **Step E: Drying the clay samples** (Department of Metallurgy and Materials Engineering) and collecting the clay samples
 - 6.8.1. Considerations:
 - 6.8.1.1. The drying oven needs to be booked following the requirement of the Department of Metallurgy and Materials engineering.
 - 6.8.1.2. Drying the samples takes a considerable amount of time (about two days for 1.5kg of sieved sample). An effort should be made to remove as much water from the sample as possible. This can be performed by leaving the sample settle for several days and removing the top water which would then need to be filtered following Step D.
 - 6.8.1.3. The samples can be left in the drying oven unattended.
 - 6.8.2. **Equipment**: Baking glass trays minimum 3.5 L (2 of them were used for 1.5kg sample and the procedure had to be repeated twice) capable of withstanding at least 200 °C, a drying oven (Memmert Type UF 160), heat-resistant protection for the hands and protection for the surface on which the trays are going to be placed, plastic scrapers and wood sticks, sample bags and markers, scale (electronic scale, precision 0.01g minimum), aluminium tray and A3 paper, foil.

6.8.3. **Procedure for the drying of the clay samples**:

- 6.8.3.1. The remaining unfiltered sample, which represents the biggest portion of the sample, left in the bucket should then be poured into glass trays. Ideally this step is done just before putting them in the drying oven.
- 6.8.3.2. The trays are placed in the drying oven which is turned on following the manual (Memmert, 2016. See reference list for hyperlink). If some beakers have been left sedimenting from the filtration process, they can also be placed in the drying oven.
- 6.8.3.3. The settings are then:
 - 6.8.3.3.1. Temperature: 105 °C.
 - 6.8.3.3.2. Fan: 50%.
 - 6.8.3.3.3. *Flap*: 10%
 - 6.8.3.3.4. *Timer*: For processing a sample of 1.5kg (pre-sieving weight), the minimum drying time was 15 hours (two sessions of 7.5 hours) when distributed in two flat glass baking trays. The larger the area of the base of the tray (the larger and longer the container), the least time it will take to dry the sample. Similarly, the drying oven of the Faculty of Engineering can accommodate several trays inside. The sample should be poured in as many trays as the oven can accommodate to reduce drying time. Similarly, drying the sample in small batches in large trays will take less time.
- 6.8.3.4. The trays should be removed with care because of their temperature and left to cool down on a flat surface protected with a tray or a towel.

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6.8.3	5. The trays should then be taken to the Archaeology Laboratory or a room where	
	they can be emptied. It is best to cover the trays with foil during transport to	
	prevent contamination.	
6.8.3	6. The dry clay should be collected in a weighed labelled sample bag. Some clay	
	might have adhered to the trays, plastic scrapers or wood picks can then be used	
	to collect what is left on the trays.	
6.8.3	7. An aluminium tray covered with A3 paper can also be used in the similar way as in	
	Step C.	
6.8.3	8. The sample should be weighed and the weight of the bag deducted from the	
	total.	
6.8.4 Recording . The weight of the sample should be recorded in the same		
6.8.4. R	ecording: The weight of the sample should be recorded in the same	

6.8.4. Recording: The weight of the sample should be recorded in the same database/spreadsheet than the other information as well as written down on the bag. The procedure as well as any change in settings should also be recorded.



Figure 10: The clay-water mix is poured in glass trays which are placed in the drying oven.

6.9. Step F: Cleaning (Department of Metallurgy and Materials Engineering)

6.9.1. Considerations:

- 6.9.1.1. The Ultrasonic (US) Bath (Kerry Ultrasonics Ltd., Type: COM MK2 300 A/S) needs to be booked following the requirement of the Department of Metallurgy and Materials engineering.
- 6.9.1.2. Training should be followed to ensure that the researcher knows how to use all the baths.
- 6.9.1.3. The Scientific officer should be consulted if any doubt concerning cleaning or storing of the equipment arise.
- 6.9.1.4. The sieves should be washed as quickly as possible after the sieving process.

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- 6.9.2. **Equipment**: Ultrasonic bath, heat resistant gloves (or a cloth), the set of sieves to be cleaned and their clean storage boxes.
- 6.9.3. **Procedure for the Cleaning**: All sieves should be washed with the Ultrasonic bath. The sieves can be prewashed in a sink or with a soft brush for the biggest meshes. Two sieves can be put in one of the trays provided.
 - 6.9.3.1. The cycle is as follow (refer to Figure 11 for the Baths numbers):
 - 6.9.3.1.1. *Bath 1* (washing): The sieves should be left for 15 minutes in the first bath. Bath 1 is activated pressing A.
 - 6.9.3.1.2. *Bath 2* (washing): The sieves should be left for the whole cycle. The end of the cycle is shown by a signal tone. Bath 2 is activated pressing B.
 - 6.9.3.1.3. *Bath 3 and 4* (rinsing): The tray should be then dipped in bath 3 (tap water) and Bath 4 (deionized water). Each for a few seconds.
 - 6.9.3.1.4. The sieves should then be sprayed with nitrogen gas.
 - 6.9.3.1.5. The tray should then be placed in the drying bath (*Bath 5*). This bath starts when the lid is closed. The sieves can be left in the bath for 5 minutes. When opening the drying bath, the US tray should be left to cool down before touching it.
 - 6.9.3.1.5.1. Some sieves might not be safe to put in the drying bath. For example, if the sieve has traces of glue, such as the sieve 63µm, it should not be dried using the bath but rather air-dry.
 - 6.9.3.1.6. If the sieves are not clean after Bath 3 and 4 (some inclusions might still be wedged in the mesh), then the cycle should be repeated (Bath 1, 2,3 and 4).
 - 6.9.3.1.7. The sieves are then to be stored in their clean and closed boxes and stored in the Classics and Archaeology Department store according to the storing system established by the Scientific officer.



Figure 11: The US bath is composed of different baths as mentioned in text above.

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- 6.10. Storing and Archiving of clay samples: The following should be stored appropriately according to the storing and archiving system implemented by the Scientific Officer and any future SOP relating to archiving and storing.
 - 6.10.1. A sample of un-sieved material should be kept either in a labelled bag in a non-humid space or in a vial.
 - 6.10.2. A sample of sieved material should also be kept or stored for reference if the material will be further used.
 - 6.10.3. The fraction collected from the sieves should be stored in zip-locked bag labelled with the appropriate information (weight, sample number, sieving range, project).
 - 6.10.4. A record database should be stored and backed up on a secure hard drive.

7. References

- 7.1. Cordell, A.S., Wallis, N.J. and Kidder, G., 2017. Comparative clay analysis and curation for archaeological pottery studies. *Advances in Archaeological Practice*, *5*(1), pp.93-106.
- 7.2. Cuomo Di Caprio, N., 2017. *Ceramics in archaeology: from prehistoric to medieval times in Europe and the Mediterranean: ancient craftsmanship and modern laboratory techniques: vol I*. Rome: L'Erma di Bretschneider.
- **7.3.** Hein, A., & Kilikoglou, V., 2020. Ceramic raw materials: how to recognize them and locate the supply basins: chemistry. *Archaeological and Anthropological Sciences*, *12*(8), pp. 1-17.
- 7.4. Hünig, S., Kreitmeier, P., Märkl, G. and Sauer, J., 2006. *Arbeitsmethoden in der organischen Chemie.* Berlin: Verlag Lehmann.
- 7.5. Memmert., 2016. "UNxx^{PLUS} Ufxx^{PLUS} INxx^{PLUS} Ifxx^{PLUS} : Operating instructions". Available at: <u>https://www.memmert.com/index.php?eID=dumpFile&t=f&f=3917&token=9b8a116d954b7</u> <u>aabab82ab9d1ea4582d4a701bd9</u> [Accessed: 15/10/2020].
- 7.6. Merck., 2019. Safety Data Sheet: Nitrogen. Available at: <u>https://www.sigmaaldrich.com/MSDS/MSDS/DisplayMSDSPage.do?country=MT&language=</u> <u>EN generic&productNumber=295574&brand=ALDRICH&PageToGoToURL=https%3A%2F</u> <u>%2Fwww.sigmaaldrich.com%2Fcatalog%2Fproduct%2Faldrich%2F295574%3Flang%3Dde</u> [Accessed: 15/10/2020].
- 7.7. Pirone, F.S., 2017. Trade, Interaction and Change: Trace Elemental Characterization of Maltese Neolithic to Middle Bronze Age Ceramics Using a Portable X-ray Fluorescence Spectrometer. Florida: University of South Florida. [Unpublished PhD].
- 7.8. Rice, P.M., 2015. *Pottery analysis: a sourcebook*. University of Chicago Press.
- 7.9. General Laboratory Practice for the Department of Classics and Archaeology SOP CAR-015
- 7.10. Occupational Health & Safety Policy, 2020, [Online], Available at https://www.um.edu.mt/hrmd/secure/policies/OccupationalHealthandSafetyPolicy.pdf [Accessed 19th April, 2021].
- 7.11. Health and Safety Risk Assessment Department of Classics and Archaeology Faculty of Arts.

8. List of Appendices/Worksheets

8.1. N/A