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Methods Note: The extended minimum extraction technique: an update on sampling protocols

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Abstract

This paper communicates an update to the minimum extraction technique (MET) sampling protocol (a means by which to extract and subsequently measure a minute sample from museum objects for luminescence dating), designated the extended MET protocol. The new protocol facilitates a sample yield of approximately double that of the original MET protocol without altering the outward appearance of the sampling mark on archaeological materials. This development is useful when working with museum materials where the visual integrity of artefacts often takes precedence over access to sampling for luminescence analysis.

Keywords: minimum extraction technique, ceramics, museum artefacts, optically stimulated luminescence

1. Introduction

The minimum extraction technique (MET) sampling protocol was first presented by Hood & Schwenninger (2015) as a technique which enabled sampling for absolute optically stimulated luminescence (OSL) dating while at the same time minimising the quantity of sample required for extraction from ceramic (and similar artefacts such as mud seals) housed in museum collections. While it can of course be argued that OSL dating of non-museum ceramics (i.e. from recent excavations) can yield more robust and routine data, working with museum materials is often a necessity, e.g. when access to OSL dating is not possible in certain regions or local laws prevent analysis of recently excavated material.

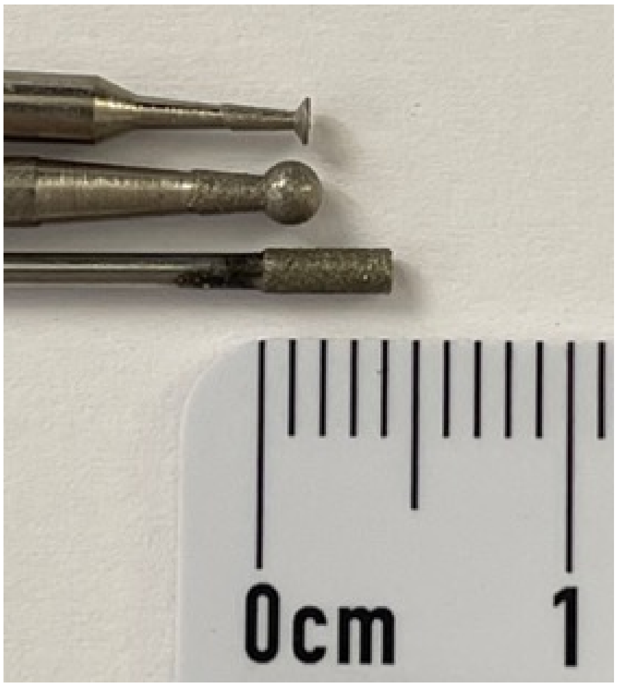

Figure 1: A (top): 1.5 mm diamond disc burr drill bit; B (middle): 2 mm diamond ball burr drill bit; C (bottom): 1.5 mm diamond core drill bit; all three drill bits are used in the extended MET sampling protocol.

2. The extended MET drilling method

Initially, MET sampling saw the removal of a sample from an artefact measuring *c.*2 mm x 4mm in volume using a hand drill. The first 2 mm x 2 mm of removed material was recovered using a 2 mm diamond ball burr drill bit (Figure 1B) and was used for internal dose rate ($\dot{D}_{int}$) measurement. The subsequent (i.e. below) 2 mm x 2 mm sample was then removed using a 1.5 mm diamond core drill bit (Figure 1C) and it was

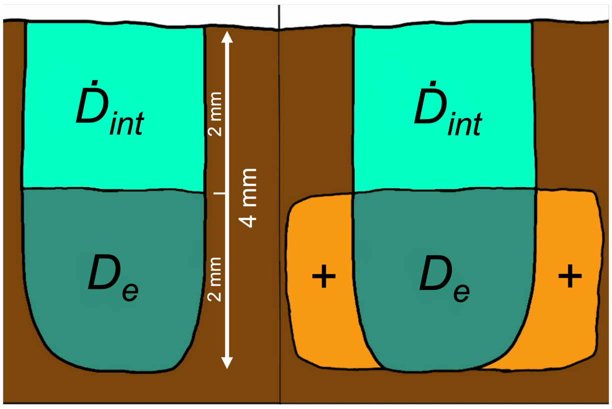

Figure 2: A schematic of original (left) and new (right) MET sampling; the '+' illustrates the laterally removed additional sample that improves MET "sample" yield.

this sample that was previously used for equivalent dose (D_e) measurement.

In the extended MET protocol, the removal of the $\dot{D}_{int}$ sample remains unchanged. The removal of the D_e sample also remains the same at first, however it is extended by an additional step. This new, final step uses a 1.5 mm diamond disc burr drill bit (Figure 1A) to carve out additional material laterally around the initial D_e sample; this lateral drilling is achieved by moving the flat drilling head both circularly and vertically between 2 mm and 4 mm in depth below the surface of the vessel. Because only the smooth shaft of the drill bit is at the surface of the vessel, no additional erosion of the top 2 mm occurs. The differences between the two methods are presented schematically in Figure 2. Figure 2 also illustrates that the additional material is suitable for D_e measurement as it comes from 2 mm below the surface of the vessel and thus avoids potential sample contamination from either light or external beta particles (cf. Feathers 2009).

3. Results

This updated, extended MET sampling method yields a significantly increased sample size, resulting in more material being available for D_e measurement without affecting the visible mark of the surface of the artefact where the sample is taken (Figure 3). Table 1 illustrates how for a test sherd, the increase in yield from MET sampling to extended MET sampling across 10 samples was just over 100%.

4. Discussion

As the density, and thus mass, of a ceramic is highly variable from vessel to vessel (or sherd to sherd), it is not possible to quantify the yield increase according to mass in absolute terms for all potential samples. As such, extended MET protocol samples carried out on other artefacts may produce different masses to those seen in Table 1, which are specific to the sherd used to demonstrate the inceased sample yield

Figure 3: A ceramic sherd (sub-sambled from MM 34209; details available here) from the Medelhavsmuseet (Museum of Mediterranean and Near Eastern Antiquities), Stockholm, displaying 10 individual sample holes on its surface; holes 1 – 5 result from the extended MET protocol, and holes 6 – 10 are those made by the original MET protocol. All visible surface holes remain at 2 mm in diameter.

resulting from the extended MET protocol presented here.

Volumetrically the increase is also difficult to quantify in absolute terms as we are not able to see beneath the surface of the sherd, but it is expected that, as with the increase in mass, the increase in volume is approximately double, as illustrated in Figure 2. It should also be noted that both the MET and extended MET sampling protocols are carried out by hand, often in make-shift laboratory conditions in museums where sampling takes place. As such, the visual presented in Figure 2 is a guide only and natural variation owing to the nature of hand drilling may see the cavity made by extended MET drilling go slightly wider and/or deeper than in Figure 2. However, it will not change the visual appearance of the sampling location on the surface of the vessel.

While each individual ceramic sherd is unique (and thus

	MET	Extended MET
	8 mg	16 mg
	9 mg	17 mg
	10 mg	19 mg
	8 mg	19 mg
	7 mg	19 mg
	7 mg	17 mg
	8 mg	20 mg
	6 mg	13 mg
	5 mg	12 mg
	7 mg	19 mg
Mean	7.5 mg	17.1 mg
Std dev	1.43 mg	2.73 mg

Table 1: Difference in sample mass yield between the MET and the Extended MET sampling protocols. NB variations in sample weight are likely resulting from the heterogeneity of the ceramic fabric, coupled with voids within the ceramic matrix, caused, e.g., by organic inclusions that have been burnt away. A portable digital scale, reading to three decimal places was used to weigh each sample.

each sampling action is unique) it has been my experience that the new extended MET protocol has an identical visible surface footprint to the original MET protocol, because the surface above the expanded sample remains stable. I therefore expect that this new technique is suitable for both intact and fragmentary objects. To date, this extended MET protocol (and indeed the MET protocol before it) has not caused structural issues for sampled objects and can be, in general, considered a suitable sampling protocol for museum ceramics. However, caution should always be exercised by the luminescence practitioner when dealing with new, unfamiliar material as the uniqueness of each individual piece could render destructive sampling difficult for certain artefacts, particularly if their matrix is of a particularly friable nature.

An additional benefit of the extended MET protocol is that is also permits sampling with increased yield of ceramics with thinner profiles. In general, if working with complete vessels, the ideal sample location is the base of the vessel (as it is usually both the thickest part of the vessel, and the most sturdy and the most inconspicious place to sample). However, to ensure that the required 2 mm of surface material remains in place to ensure no contamination from light or beta particles, an artefact thickness of at least 6 mm was required for the original MET protocol. This depth was necessary to allow 2 mm to be remaining between the sample location and the interior or back surface even after the 4mm sample was removed from the exterior surface (2 mm removed for $\dot{D}_{int}$ determination, plus the 2 mm sample for D_e measurement). As such, a 6 mm vessel profile was required for successful MET samping. A profile width of 5 mm could be worked with if necessary, however with the original MET sampling, this meant a significantly reduced D_e sample could be taken. However with the extended MET protocol, even when working with a thinner ceramic profile of 5 mm, it is in theory possible to achieve a D_e sample yield which is approximately equal to the sample volume achievable with the standard MET protocol, as it is possible to remove additional sample laterally within the central 1 mm of suitable sample at the middle of the vessel wall.

5. Conclusion

This paper has presented an update to MET sampling, termed the extended MET, which sees an increase of $\sim$100% yield for the D_e sample compared to the sample yield that the original MET sampling protocol delivered. This is a significant improvement in the sample sizes that can be taken from museum objects whilst still ensuring that a minimum amount of damage is caused and that the aesthetic integrity of the artefact is upheld. The extended MET protocol is thus recommended for use by those luminescence practitioners who work with museum objects.

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Reviewer

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