Ancient TL

www.ancienttl.org · ISSN: 2693-0935

Weida, W. and Junding, X., 1989. *A convenient method for preparation of fine-grain samples.* Ancient TL 7(3): 47. https://doi.org/10.26034/la.atl.1989.151

A convenient method for preparation of fine-grain samples

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In the TL dating of pottery, the fine-grain samples are usually separated by suspending in acetone, and the acetone is eventually lost by evaporation (Zimmerman, 1971, Aitken, 1985). We have developed a separation method for fine-grains using a water flotation process. This method is more convenient and quicker than the acetone method, and it has been used in our laboratory for the past few years.

The operational procedures for the method are as follows:

1. The pottery fragment is crushed by squeezing in a vice; the rubble produced by the "vicing" is then gently crushed further in an agate pestle and mortar, and the grains in the size of less than 60 μm are selected by sieving.
2. Put the selected grains, about 400 mg, into a 150 ml beaker; the grains are then washed in the acetone-alcohol-distilled water sequence. In order to avoid the coagulation of grains after the acetone evaporation, alcohol must be used as a transition material.
3. Pour more distilled water into the beaker until the height is 70 mm. After stirring, the beaker is kept still for 10 mins, so that the grains of size greater than 8 μm deposit at the bottom of the beaker.
4. Slowly pour the suspension into another beaker. This is then kept still for 60 mins; at the end of this period grains having a size of less than 3 μm are still in suspension.
5. Finally, this suspension is tipped into another beaker thereby obtaining grains in the size range of 3 to 8 microns. The settling times of 10 mins, and 60 mins are calculated approximately according to Stokes Formula,

$$S = \frac{9\eta h}{2(\rho-\rho')gr^2}$$

where S is the settling time, h is the height of water in the beaker, η is the viscosity coefficient of water, ρ and ρ' are the density of the grains and the water respectively, g is gravitation acceleration, and r is the radius of the grain.

6. A flat-bottomed funnel of 80 mm diameter, with a stopcock at its exit, is used for the preparation of the disc samples. A copper wire ring with a handle is placed within the funnel at the bottom; a large piece of glass is placed on this ring and this glass carries about 30 aluminium discs (10 mm in diameter and 0.5 mm thick).
7. The separated fine-grains in the beaker are resuspended in distilled water and then the suspension is poured into the funnel. During pouring, a spiral wire with a handle is used to hold down the discs and keep them still. This is removed when pouring has finished.
8. After all the fine-grains have been deposited onto the discs (about 30 min) the valve is opened and the water is drained off. The draining is initially rapid but gradually reduced until it is until drop by drop. When the distance between the water plane and sample is about 2-3 mm, the dripping must be very slow, about 15-20 second per drop, until the water is drained away. This process takes about 60 min. The drip rate is controlled by the stopcock.
9. The glass piece is then taken out of the funnel by means of the handle on the copper ring and the disc samples are dried at a low-temperature (50 °C).

The whole process takes about 3.5 hours. If a larger diameter funnel is used and the quantity of grains increased, a greater number of discs can be obtained in the same time.

Usually 30 such disc samples are prepared at a time; each disc carries 0.8-1.0 mg of sample, and the disc-to-disc scatter in TL or weight reproducibility should be not more than ±5%. Usually the reproducibility of weight is slightly worse than that of TL, because the sensitivity of the balance is not as good as the TL instrument. It may be noted that 1 mg per disc corresponds to a thickness of 1.3 mg cm^{-2} and this is suitable for measurement of alpha-particle effectiveness, being comparable with the ultrathin TLD of 1 mg cm^{-2} used in the measurement of alpha dose-rate (Wang, 1989).

In conclusion, this method not only is convenient and quick but has good reproducibility.

References

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PR. Reviewed by Martin Aitken