Reproducibility of probe-scan analyses of fine-grained media: a case study using modern bone china

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Modern bone chinas are a hybrid ware that was initially developed by Spode around 1800. They were derived from recipes containing elements of 18th Century bone ash (phosphatic) porcelain and true (hard paste) porcelain. Like their early phosphatic counterparts, they are difficult to produce due to the fact that they have near eutectic compositions; consequently their successful firing requires a degree of temperature control generally beyond the capabilities of early bottle kilns. Consequently, small differences in the compositions of these wares strongly influence their behavior in the kiln. It is therefore important that the composition of fine porcelains be accurately determined so that this aspect of their production can be assessed. Furthermore these types of data, when gathered for archaeological ceramics, can also be used for attribution purposes as well as to address causes of kiln wastage problems that plagued the manufacturers of these wares. Many XRF and other labs are geared toward analyzing rock samples, which have very different compositions compared with most porcelains, so there is a small demand in the marketplace for these types of analyses. However, the electron microprobe can be quickly and easily calibrated to perform raster-type (scan) analyses, which when averaged provide an indication of the bulk composition of fine-grained media, including ceramics. Despite the popularity of this method, there are no criteria available to direct those interested in determining the compositions of porcelains per se. In this regard, beam diameter is potentially an important variable that may influence the minimum number of spot analyses required to provide a reliable (for desired precision and accuracy) indication of the bulk composition of a porcelain sample of given grain size. To evaluate this variable, three thin-sections were made from sherds of a modern, English, bone china gravy boat. The grain size of the sherds ranges from one-twentieth to less than one-fiftieth of a millimeter. This particular ware consists of silica polymorphs, a tricalcium phosphate (C_3P) phase, probably whitlockite, anorthite and an inferred melt phase. Two sets of raster-type analyses were undertaken on each of the three thin-sections: one at a beam diameter of 20 μm and another at 40 μm. Provided that beam diameter is on par with the size of the largest grains in an otherwise very fine-grained sample, preliminary statistical analysis of the data indicate that, for data sets exceeding several hundred analyses, this parameter is not an important variable in governing the data's precision.