A geochemical investigation of the origin of Rouletted and other related South Asian fine wares
L.A. Ford, A.M. Pollard, R.A.E. Coningham & B. Stern

The full text of this paper is published in Antiquity December 2005 pp. 909-920 which can be found at http://antiquity.ac.uk/ant/079/ant0790909.htm.

Technical Note

Samples cut from each sherd were crushed and then ground using a Gyro Mill to a very fine powder, taking care to clean the grinding device between samples to avoid contamination with any residual powder. An aliquot of 0.1g ± 0.0010g of each sample was weighed into 25ml PTFE crucibles. To each crucible was added 4mls of a 1:2 mixture of hydrofluoric (HF) and perchloric (HClO4) acids. The crucibles were placed on a hotplate in a fume cupboard and the sample evaporated to dryness (about 3 to 4 hours). The crucibles were removed from the hotplate and allowed to cool. To each crucible was added 1ml of nitric acid (HNO3) and topped up with distilled water to about 3/4 full and warmed on the hotplate for 15–20 minutes. The crucibles were removed from the hotplate and allowed to cool. Once cooled, each was made up to 10.40g using distilled water, and transferred to a labelled plastic bottle for analysis.

ICP-AES analysis (Perkin Elmer Optima 3300RL) was carried out at the NERC funded facility at the Department of Geology, Royal Holloway, University of London. The standard laboratory computer programme was used to determine selected major (Al, Fe, Ca and K), minor (Ti, P, Mg, Mn and Na), and trace element (Ba, Co, Cr, Cu, Li, Ni, Sc, Sr, V, Y, Zn, Zr, La and Ce) composition of geological silicate samples. During the analysis of the samples a blank and a set of laboratory internal standard rock samples (KC10, KC11, KC12, KC14 and RH21), and the geological standard reference material RGM-1 were measured. Elemental concentrations were determined for the above elements using primary reference solutions, and the analyses converted stoichiometrically to conventional oxide formulations for the major and minor constituents. Detection limits in solution were well below 1 mg l⁻¹ (ppm) for all the major, minor, and trace elements determined, with a typical precision of 1%–1.5% RSD (relative standard deviation) when the concentration is 100 times greater than the detection limits. This is considered sufficient for most of the trace elements determined (Thompson and Walsh 1983: 33–35).

If sufficient volume of sample solution remained after analysis at RHUL, then the samples were subsequently analysed by ICP-MS (Thermo-elemental PlasmaQuad 3) for rare
earth elements (REE) at the University of Bradford. Each sample was further diluted with deionised water (dilution factor of 10) and an indium internal standard was added to give a final concentration of 10 ppb. Fully quantified data were obtained by the analysis of matrix matched calibration solutions of all the REE elements. In accordance with common practice for reporting REE data on geological material, these data have been normalised to Chondrite REE abundances after Wakita (reported in Henderson 1984) using the values (in ppm); La (0.340), Ce (0.910), Pr (0.121), Nd (0.640), Sm (0.195), Eu (0.073), Gd (0.260), Tb (0.047), Dy (0.300), Ho (0.078), Er (0.200), Tm (0.032), Yb (0.220), Lu (0.034). This removes the inherent ‘odd-even’ sawtooth signal seen in non-normalised REE profiles.

For reviews of this type of scientific analysis, see e.g., Rice 1987, Bishop and Neff 1989, Neff 1992, Pollard and Wilson 2001), and Pollard and Heron (1996) contains a brief review of the ICP-AES and ICP-MS techniques. Standard references for ICP-AES include Thompson and Walsh (1983) and for ICP-MS, Date and Grey (1989).

ICP-AES data on the analytical standards are given in Table 1 (web-file), together with the accepted values. Inspection of this Table shows that all the elements reported are adequately measured, with the exception of Zr, which was omitted from all subsequent numerical analysis. A full list of the analytical data obtained by ICP-AES is given in Appendix 1. A summary of the means and standard deviations (one sigma) of all the groups analysed is shown in Table 2 (web-file). The statistical package SPSS 10 for Windows was used for the statistical treatment of the analytical data, with the data for Zr removed. Exploratory data analysis consisted of Principal Components Analysis (PCA; Bishop and Neff 1989, Baxter 1994) and cluster analysis (Baxter 1994) to classify the sherds into possible compositional groups. Cluster analysis was performed by both Average Linkage Cluster Analysis and Ward’s method, and the results were displayed in the form of dendrograms. All elements were standardised (z-score) prior to analysis to remove the influence of absolute magnitude. Subsequent analysis to test particular hypotheses took the form of Discriminant Analysis (ibid.). The dendrograms (not shown) using both Average Linkage and Ward’s Method indicated substantial chemical homogeneity in the main body of the data.


