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Use of tungsten carbide disc-mill in geochemistry: no evidence of contamination

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A common statement in the analytical techniques section of geochemical papers is that the samples were powdered in an "agate disc-mill", the inference being that the use of a tungsten steel disc-mill is a poor mans alternative and would likely have caused contamination. This is also a commonly stated verbal opinion, although no evidence for this has ever been quoted to the authors. Since tungsten steel disc-mills are commonly available, a simple test was made to see if contamination does occur. Small fragments (< 1*1*0.5 cm) of a shale sample from the Caledonides of N. Norway were subjected to 5 minutes milling in a tungsten-carbide disc-mill, enough time to make a suitably fine powder for digestion for geochemical analysis. After a small portion had been removed, the remainder was milled for a further 20 minutes, after which another portion was removed. The remainder was then milled to give a total milling time of 85 minutes. After allowing the mill to cool to room temperature, the final powder was extracted. The three samples were then sent under anonymous names to professional, research-quality chemical laboratories, for a full range of analyses (major, traces, REE, LOI, TOC, TOS). Correlation coefficients (R2) between the three data sets gave R2 = 0.9993 (5-25 min), 0.9996 (5-85 min) and 0.9991 (25-85 min). If wt % data are converted to ppm, R2 increases to > 0.9999 in all cases. Comparison of the three data sets showed no systematic evidence of contamination by the disc-mill; the maximum value was not necessarily found to occur in the 85-minute sample (13 % in 5 min; 29 % in 25 min; 26 % in 85 min); 32 % of elements had equal values for all times. Ratios of elements from the three data sets lay between 1.22 and 0.71 (shorter time/longer time). The largest percentage difference ((max-min)*100/max) was 28.6 %, for W, from 1.5 ppm (5 min) to 2.1 ppm (25 min), and then 'dropped' to 2.0 ppm in the 25 minute sample. Fe underwent a reduction in concentration, from 6.81 to 6.51 wt % from 5 to 85 minutes (4.8 % difference); Fe concentrations were highest in the 25 minute sample (6.84 wt %). Two other samples were disc-milled for 5 minutes and both then sent to two separate laboratories. Comparisons of these pairs showed that they exhibit far greater differences than those seen in the samples disc-milled for an extended period. R2 values were poorer (0.9968 & 0.9845, using ppm and wt % data) and maximum % differences were very much higher, at 83 % (Ga) and 91 % (Ni), although in only one more case (Zr) was the % difference > 28.6 % (the maximum in the disc-mill experiment). In both pairs, only three elements had zero difference (Mn, Ti, Lu & Ca, Er, Lu). % differences for W were 9.7 & 25 %; the latter is the same order of magnitude as in the disc-mill experiment. Since the interpretation of geochemical data is often based on defined boundaries and/or shapes on plots and/or element ratios, interlaboratory differences raise more of a problem (and one that cannot be fully resolved) than disc-milling with a tungsten steel machine. In conclusion, no evidence was found that significant contamination occurs during a short disc-milling (5 minutes) of shales. However, with rocks containing harder minerals, it is possible that some contamination might occur after a long milling interval.