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Grinding Head Contamination Tests

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By its very nature, most geological material is extremely resistant. Accordingly, the crushing and grinding process necessary to reduce geological material to a homogeneous powder for analysis has a great potential to introduce contamination, by wearing away of the grinding medium. Knowledge of the type and level of contaminants introduced by grinding, therefore, is of utmost importance and ensures that the analyst can provide clients with the best analytical outcomes.

Given that quartz is the most abrasive major mineral encountered in silicate analysis, we have undertaken contamination tests using Merck granular quartz in grinding heads of different composition for varying grinding times (60-180 seconds). The ground samples were then analysed by XRF (Philips PW2404) and ICP-MS (Perkin-Elmer Elan 6000) to quantify the levels of contamination.

To reduce grainsize effects in XRF trace element analysis, a sample with the finest grainsize possible is required. Particle size analysis, therefore, was also carried out on the ground quartz samples to determine the efficiency of the grinding heads and to see if our analytical objectives were being satisfied.

The grinding heads used in the study were:

(a) 200g (maximum capacity) unit: Tungsten Carbide, (two heads); Chrome Steel; Carbon Steel; Partially Stabilised Zirconia (PSZ); and

(b) 40g (maximum capacity) unit: Syalon (synthetic Silica-Aluminium ceramic).

50g aliquots of acid-washed granular Merck quartz were ground for 60s, 120s and 180s in each of the 200g grinding heads and analysed in Geoscience Australia's laboratories. Because the Syalon head was smaller, relatively light in weight and therefore less efficient at grinding, a 25g aliquot of Merck quartz was used. A control sample of Merck quartz was analysed as a blank.

Manufacture of Heads

How the heads are made determines their composition and contamination effects.

Tungsten Carbide

Tungsten Carbide grinding heads are made by sintering carbide powder with a cobalt metal powder binder. The two contaminants found were W and Co.

Chrome Steel

Chrome Steel grinding heads are made from high chromium (11% to 14%), high carbon (1.5% to 2.5%) steel, known as D3 in the AISI nomenclature. The grinding head manufacturers use steel with the lowest levels of unwanted metals, and from these 'clean' batches, grinding heads are turned. Main constituents are Fe and Cr with small amounts of Ni, Cu, Co, Mo, and W.

Carbon Steel

Carbon Steel heads are made from case-hardened low-alloy mild steel and have lower levels of chromium, copper, nickel and molybdenum than Chrome Steel heads. Carbon steel is used as an alternative to Tungsten Carbide because it does not contain tungsten or cobalt and costs less to purchase. However, it is a softer material than Tungsten Carbide and wears more quickly. The main constituent is Fe, with traces of Cr, Cu, and Ni.

Partially Stabilised Zirconia (PSZ)

PSZ grinding heads are made from Zr (Hf)O_2 stabilised with Mg. In Australia, Zirconium for PSZ is derived from Zr-rich beach sands. The sand undergoes purification in an arc furnace and arrives at the ceramics manufacturer as ZrO_2 plus MgO. Particle size is reduced to –325 mesh by ball milling. The compound is spray dried, after which pre-forming, compaction and machining of the green body occurs. The transformation to the very tough PSZ occurs when the pre-formed components are sintered in a 1700°C furnace for five days. Main constituents are Zr, with traces of Hf, Sr, Ba.

Syalon

Syalon is a man-made ceramic alloy of Si_3Ni_4 and Al_2O_3. It is manufactured in a patented process by reacting silicon nitride, silica, aluminium oxide and aluminium nitride. Main constituent is Si (not able to be determined in our tests), other contaminants found were Al and Y.

Level of Contaminants Found

PPM	Tungsten Carbide 1			Tungsten Carbide 2			Chrome Steel			Carbon Steel			PSZ			Syalon 25g		
Time	60s	120s	180s	60s	120s	180s	60s	120s	180s	60s	120s	180s	60s	120s	180s	60s	120s	180s
Fe							2300	2500	2300	2520	3040	3380						
Al																1100	1100	1100
Ba													3.1	2.4	3			
Co	113	128	137	138	157	157	0.8	1.2	0.8									
Cr							508	513	546	2	4	5						
Cu							2	2	2.9	2	2	2						
Ni							4.2	4.8	4.3	2.2	1.2	1.8						
Sr													1	1.1	1.2			
W	905	994	1047	1150	1276	1259	2	2	2.9									
Zr													303	375	434			
Y																1.07	1.44	2
Mo							1.68	1.75	1.75									
Hf													6.68	8.09	9.37			

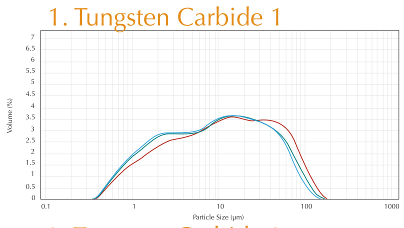
Tungsten Carbide is the grinding mill of choice for most geochemical labs, but a second grind performed in a Carbon Steel grinding head is necessary if W or Co analyses are required. Syalon's light weight and consequent inefficient grinding means that it has limited application for geological samples. Generally, contamination increased with increased grinding time.

Tungsten Carbide W – 900 to 1276ppm; Co – 113 to 157 ppm
Chrome Steel Fe – 2300 to 2500ppm; Cr – 508 to 546ppm; Ni, Cu, Mo< 5ppm.
Carbon Steel Fe – 2520 to 3380ppm; Cr, Cu, Ni <5ppm.
PSZ Zr – 303 to 433ppm; Hf – 6 to 9ppm; Sr – 1ppm; Ba – 2ppm.
Syalon Al – 1100ppm; Y – 1 to 2ppm.

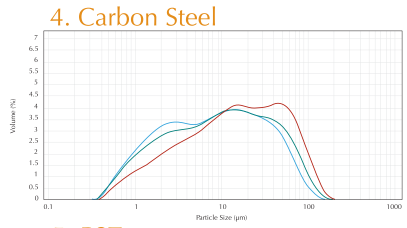
Laser Particle Size analysis (Malvern Instruments Laser Mastersizer 2000) indicated that at 180s grinding time, Tungsten Carbide, Chrome Steel, Tool Steel and PSZ heads reduced >90% of the sample to <74µm; Tungsten Carbide, Chrome Steel and Tool Steel reduced approximately 50% of the sample to <10µm; PSZ reduced 35% of the sample to <10µm and Syalon (25g) at 180s had reduced 49% to <74µm and 15% to <10µm.

Particle Size Distribution

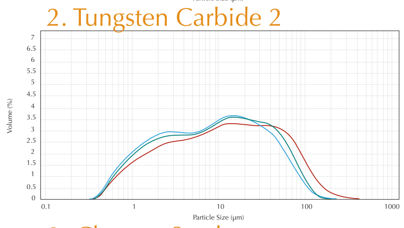
1. Tungsten Carbide 1



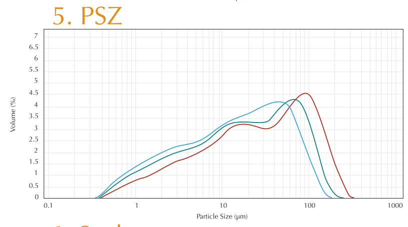
4. Carbon Steel



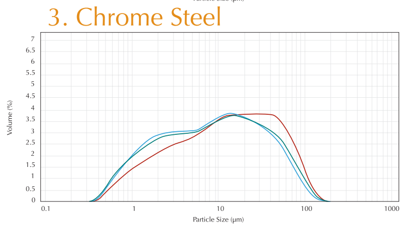
2. Tungsten Carbide 2



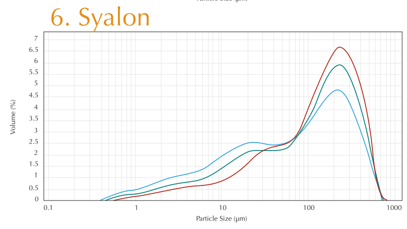
5. PSZ



3. Chrome Steel



6. Syalon



__60 seconds __120 seconds __180 seconds

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