Quartz chemistry for Al & Be extraction

Sample ID: **Date:**

QUARTZ PURIFICATION

Grain size:

Initial mass:

	Y/N	Comments
Rinse fines:		
hot H_2O_2 (for micas):		
HCl/HNO_3 soak (overnight):		
Rinse & dry:		
Magnetic separation:		
Heavy liquid:		

Pyrophosphoric treatment (optional):

mass in (40 g nominal):	
volume H_3PO_4 added (400 ml nominal):	
time started:	
time @ T = 240 C:	
time ended (45 min. nominal):	
decant & rinse	
volume 25% NaOH added (400 ml nominal):	
boil time (10 minutes nominal):	
dry mass out:	

Ultrasonic leach in 1 liter 1% HF/1% HNO_3:

Bottle ID	bottle tare	dry mass in	number of leaches	dry mass out	sample loss

Aluminum assay:

quartz aliquot mass (0.5 g nominal):	
dissolve in teflon beaker	
volume HF (25 ml nominal):	
volume HNO_3 (10 ml nominal):	
dry & repeat if not dissolved	
add 10 ml HNO_3 and dry	
add 10 ml HNO_3 and dry	
dissolve in 1 ml 1:1 HCl	
tare centrifuge tube	
transfer to centrifuge tube	
rinse beaker with 11 ml pure H_2O & add to tube	
solution mass:	
atomic absorption [Al]:	
quartz [Al]:	
if not acceptable, repeat ultrasonic leach	

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QUARTZ DIGESTION

Put sample in FEP bottle

bottle tare:	
sample + bottle:	
mass quartz:	

Be spike:

Be carrier ID:	
carrier [Be]:	
initial carrier bottle mass:	
mass carrier added:	
final carrier bottle mass:	
mass Be spike (µg):	

Dissolution:

volume HF/HNO_3 added (5 X quartz mass/1 X quartz mass nominal):	
cap loosely and dissolve	
(no heat for first 6 hours)	

Split & Transfer

mass bottle + solution:	
mass solution:	
transfer 10% solution or 300 µg Al (whichever greater) to teflon beaker for AA analysis mass:	
evapoconcentrate solution in bottle	

AA analysis:

dry aliquot	
add 10 ml HNO_3 or 2 drops $HClO_4$ and dry	
add 10 ml HNO_3 or 2 drops $HClO_4$ and dry	
dissolve in 1 ml 1:1 HCl	
tare centrifuge tube	
transfer to centrifuge tube	
rinse beaker with 11 ml pure H_2O & add to tube	
solution mass:	
atomic absorption [Al]:	
atomic absorption [Be]:	
quartz [Al]:	
measured mass Be:	
check against Be spike (from above):	

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FLUORIDE FUMING:

transfer concentrated solution from FEP to Pt dish & dry	
rinse bottle 3X w/HNO_3 & transfer to Pt dish	
dry	
add 10 ml H_2SO_4 & dry	
(remove from heat IMMEDIATELY upon dryness)	
add 5 ml H_2SO_4 & dry	
add 5 ml H_2SO_4 & dry	
dissolve in HCl (wait for $CaSO_4$ to dissolve)	
transfer to teflon beaker w/pipet & dry	
rinse Pt dish 3X w/HCl and transfer rinsate	

Fe REMOVAL BY ANION EXCHANGE:

Dissolve dry cake in 1:1 HCl (1 ml nominal):	
transfer solution to centrifuge tube	
rinse beaker with HCl (equal volume):	
transfer rinsate to centrifuge tube	
condition anion column (if not recently conditioned)	
drain resin bed	
add 2 column volumes 9N HCl & drain	
add 4 column volumes 0.012 N HCl & drain	
add solution *gently* to column	
drain into teflon beaker labeled "*sample* anion cv 1-2"	
add enough 9N HCl to centrifuge tube to make 1 column volume total	
add solution *gently* to column	
drain into beaker cv 1	
add 1 column volume 9N HCl *gently* to column & drain	
place bottle labeled "*sample* anion rinse" under column	
add 1 column volume 0.012 N HCl *gently* to column & drain	
add 3 column volumes 0.012 N HCl *gently* to column & drain	
recondition column, leaving some solution on bed	
hang "conditioned" sign from top of column	

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Al & Be SEPARATION BY CATION EXCHANGE:

dry eluent in teflon beaker	
dissolve in 1:1 HCl (1 ml nominal)	
transfer to centrifuge tube	
rinse beaker w/enough pure H_2O to make 1N HCl (5 ml nominal)	
transfer rinsate to centrifuge tube	
condition cation column (if not recently conditioned)	
drain resin bed	
add 10 column volumes 6N HCl & drain	
add 3 column volumes pure H_2O & drain	
add 1 column volume 1N HCl *gently* to column and drain	
place bottle labeled "*sample* cation cv 1-3 Be" under column	
add solution *gently* to resin bed & drain	
add enough 1N HCl to column to make 3 column volumes total & drain	
place 50 ml centrifuge tube labeled "*sample* cation cv 4-6 Be" under column	
add 3 column volumes 1N HCl *gently* to column & drain	
place 50 ml centrifuge tube labeled "*sample* cation cv 7-9 Be" under column	
add 3 column volumes 1N HCl *gently* to column & drain	
place 60 ml bottle labeled "*sample* cation cv 10-15 Be" under column	
add 5 column volumes 1N HCl *gently* to column & drain	
place 50 ml centrifuge tube labeled "*sample* cation cv 16-19 Al" under column	
add 4 column volumes 2.5 N HCl *gently* to column & drain	
recondition column, leaving some water on bed	
hang "conditioned" sign from top of column	

HYDROXIDE PRECIPITATION:

add one drop H_2O_2 to Be centrifuge tubes (optional), if red precipitate Ti		
add NH_4OH to Be centrifuge tubes to pH 5		
precipitate Ti 10 min.		
centrifuge 5 minutes @ 4000 RPM		
decant supernate into another labelled centrifuge tube		
re-label the old tube as Ti (instead of Be)		
add NH_4OH to all centrifuge tubes to pH 8		
set in warm water bath (overnight preferred)		
centrifuge 5 minutes @ 4000 RPM	Al:	Be:
decant supernate into labelled bottles	Al:	Be:
dissolve hydroxides in 1 ml 1:1 HNO_3	Al:	Be:
transfer solution to 15 ml centrifuge tube	Al:	Be:
rinse 50 ml tubes with 1 ml 1:1 HNO_3 & transfer rinsate	Al:	Be:
re-precipitate hydroxides with NH_4OH to pH 8	Al:	Be:
set in warm water bath (overnight preferred)	Al:	Be:
centrifuge 5 minutes @ 4000 RPM	Al:	Be:
decant supernate into labelled bottles	Al:	Be:
add 5 ml pure H_2O	Al:	Be:
vortex & centrifuge 5 minutes @ 4000 RPM	Al:	Be:
decant supernate into labelled bottles	Al:	Be:
add 5 ml pure H_2O	Al:	Be:
vortex & centrifuge 5 minutes @ 4000 RPM	Al:	Be:
decant supernate into labelled bottles	Al:	Be:

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OXIDATION:

dry and tare 2 clean* quartz vials	Al:	Be:
add ~2 drops pure H_2O to hydroxides		
transfer hydroxides to quartz vials		
dry at low heat (<100 C)		
cap quartz vials		
oxidize in furnace at 1100 C for one hour		
when cool, mass vials without lid	Al:	Be:
mass oxides:	Al:	Be:
add 3 X sample mass Ag to Al and crush		
add 5 X sample mass Ag to Be and crush		
label quartz vials clearly		
cap, cover with parafilm & place in tray		
*to clean quartz vials		

boil vials and caps in 10% HF/50% HNO_3 for one hour	