

This 3-ring binder contains a collection of documentation for this lab's XRF-related procedures. This copy is for making your comments, corrections, suggestions, and epithets (keep 'em clean boys and girls!). I'll keep updating the proper computer files accordingly.

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October 1995

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Powder Preparation for XRF Analysis

Note: Once any type of sample has been washed in methanol and distilled water, it is not to be touched with bare hands. This will help minimize sodium contamination.

Igneous Samples

Note: Make sure sample pieces are cut to less than 1 cm thick. Otherwise, it is difficult to crush them in the hydraulic press.

1. Sample surfaces are ground on a high-speed, diamond-impregnated disk ($\sim 70 \mu\text{m}$ grit) to remove saw marks, coring bit marks, or any other unwanted material such as surface alteration. The grinding machine is located in the Thin Section Lab, the 8" grinding disk is stored in the lowest drawer beneath the hydraulic press, and spare diamond disks are in the overhead cabinet over the Scientech balance, nearest the X-ray lab.
2. Wash rock chips in alcohol (methanol) or acetone to remove contaminants such as oils from your skin. Sonicate for 15 minutes in Nanopure water, changing the water after 5 and 10 minutes. Rinse three times with Nanopure water. Dry at 110°C for 8 hours (ovens are located in the ThinSection Lab and the Paleo Lab).
3. Sample pieces >1 cm must be reduced by crushing between two Delrin plastic disks in the hydraulic X-Press. All pieces <1 cm are then powdered in the Spex Shatterbox. If the large tungsten carbide (WC) grinding vessel is used, the minimum amount of sample has to exceed roughly 30 g or you run the risk of cracking the vessel ($\sim \$5,000$ ea). The smaller vessels require a minimum of about 10-15 g ($\sim \$2,000$ ea). Smaller samples can be ground in either the WC Spex Mixer Mill, or by hand in an agate mortar.
4. Transfer the sample (<1 cm pieces) to the grinding vessel by pouring in enough sample to make a small talus slope $\sim 1/2$ to $2/3$ of the way up the side of the vessel (do this on both sides of the ring in the large vessel). Don't spread the sample around. If any pieces are on top of the puck or ring, use plastic gloves, tongs, or clean paper to move the sample into the cavity.
5. Replace the lid to the vessel and carefully place the whole assembly in the Spex Shatterbox.
6. Bring the safety arm over the top of the vessel, centering the vessel's lid beneath the arm's centering bracket. Fasten the arm securely, tightening the knob almost as hard as you can by hand.
7. Pop-up the safety catch on the arm near the knob, and back-off (loosen) the knob until it hits the safety catch.
8. Close the cover on the Shatterbox housing.
9. Set the timer (**60-90 seconds is plenty for most samples in the large WC vessel**, 90-120 sec in the smaller WC vessels). The timer was replaced on Leg 179. The time indicator is approximate, so it is advisable to time the operation with a stopwatch.
10. Adjust the "Intensity" dial to 0.

11. Turn the POWER switch to "ON" and gradually increase the intensity to 10. (I begin timing with a stopwatch at this point, but you could do it when you turn the power on.)

NOTE: To stop the Shatterbox before the preset time is up, reduce the intensity know to 0 and turn-off the POWER switch.

13. After the Shatterbox has finished, open the outside cover and release the safety arm.
14. Leaving the lid on the vessel, remove the vessel from the Shatterbox and place it on the bench top, which is usually covered with paper.
15. Remove the lid, inverting it on a clean piece of paper.
16. Use a clean piece of weighing paper to clean-off the powder from the top surface of the puck and ring. Using plastic gloves or clean paper towels, carefully disassemble the vessel, putting the puck, ring, and O-ring on the inverted lid.
17. Carefully pour the powder from the vessel onto clean paper. Try to minimize airborne dust particles. If any powder remains behind (in the bottom corner), use a clean plastic spatula to dislodge it.
18. If you are grinding multiple batches of the same sample (i.e., coarse-grained rocks or inter-laboratory standards) transfer the powder to a labeled Ziploc bag, and homogenize as needed.
19. Transfer powder to a sample vial and label.
20. Wearing gloves, wash the individual pieces of the vessel with warm tap water and a plastic brush or a 'green meany' pad (**No Soap!**). The brush is very useful in removing the sample particles from the bottom corner of the vessel. Rinse the metal parts with either distilled water, isopropanol or acetone, and blow dry with compressed air. The lid is particularly vulnerable to surface rust if it is not dried quickly and thoroughly. The O-ring is washed with water and dried with a paper towel. (I rinse everything but the O-ring in acetone and air-dry it on paper towells. I don't trust the compressed air system not to introduce contaminants. JKL)
21. All surfaces that come in contact with your sample must be completely dry before the vessel is re-assembled.

Lithified Sediment Samples

1. Lithified sedimentary samples are treated like igneous samples except that the oven drying step mentioned in #2 above is replaced by:

Freeze-dry samples for 12 hours or more in the Labconoco freeze drier located next to the Shatterbox.

Un-lithified Samples (mud)

There are two schools of thought as to how to treat mud samples, wash and dry (see below), or just freeze-dry as described above. Washing the sediments has been used to remove chloride conatmination, but washing is believed to remove more elements than those associated with simple chloride contamination. It is up to the user to choose their preferred method.

1. Washing:

Mix approximately 20 mL of sample with 150 mL of distilled water in a clean glass beaker.

Place beaker in an ultrasonic bath and sonicate for 10 minutes.

Add approximately 15 mL methanol to the water/sample slurry.

Centrifuge the slurry for xx minutes at xx rpm to separate the solids.

Decant the rinse water.

Dry sample at 110°C for at least 12 hrs.

2. Follow igneous sample grinding procedure from step #4, using the following grinding times:
~45-60 sec (large vessel); 60-90 sec (small vessel).

Preparing Trace Element Pellets

Materials

Kimwipes
Isopropanol or acetone
120 mL glass beaker
Glass stirring rod
Chemplex liquid sample binder (100 mg/cc polymeric binder in methylene chloride)
Stainless steel pellet die, consisting of a base, main barrel, piston, and 2 polished "end caps,"
one made of stainless steel, the other tungsten carbide (WC)
Aluminum pellet remover
Aluminum Spex Caps
Hydraulic press (X-Press)
Round plastic containers

Brief Procedure

Record sample name in the sample log book, and on two sticky labels.

Measure-out between 4.5-7 g of fresh (unignited) sample powder and transfer to a 120 mL glass beaker. Measurement may be accomplished by rough weighings on the Scientech balance or by visual estimate in a graduated beaker. To achieve "infinite" thickness for Rh-Ka radiation, use the following guidelines. (My visual estimates are not very good, so I weigh all samples. This produces consistently better pellets for me, and takes no more time really. JKL)

<u>Sample Type</u>	<u>Minimum sample amount for each pellet</u>
Basalts, CaCO ₃ -Limestones	4.5 - 5g
Silicic sediments, volcanic ashes, dolomites	6 - 6.5g

Using an eye dropper, add 40-50 drops (I use about 30-35 drops for 5.0 grams of basalt, more for sediments.) JKL) (~1/2 eyedropper or ~600-700 μ L) of Chemplex liquid binder to the sample powder. Small variations (± 5 drops) in the amount of binder are not critical to the reproducibility of X-ray intensities.

Mix the sample and liquid binder with a glass rod until the mixture is homogeneous.

Clean the pellet die with Kimwipes and alcohol (or acetone).

Assemble the pellet die. Place the stainless steel end cap in the bottom of the die with its polished side up. Insert an aluminum Spex Cap into the die and push it down to the bottom. Clean the inside of the die with alcohol if you used your thumb to insert the cap.

Carefully add the sample/binder mixture and insert the WC end cap into the barrel, polished side down.

Slowly insert the piston, flange side down, all the way into the die.

Place the die in the hydraulic X-Press. Turn the adjusting screw on top of the press until it is tight against the die.

Close the pressure valve (the handle on the right side of the press) and turn-on the press with the toggle switch in front. **Turn-off the press at ~5-7 tons.** If greater pressures are used, the pellet may crack or peel. Severely cracked samples must be re-made.

Let the press remain pressurized for 5-10 seconds, then **slowly** release the pressure by gradually opening the pressure valve.

Remove the pellet from the die by inverting the die, removing the base and gently, but firmly, pressing down on the outside of the die until the pellet emerges. If this proves to be too difficult, place the open end of the aluminum pellet remover over the base of the mold. Invert the entire assembly and place it back in the press. Keeping a hand on the outside of the pellet mold, slowly turn the large adjusting screw on the top of the press until the pellet is released.

Attach one sticky label to the back of the pellet, the other on the lid of the round plastic sample box.

Place the finished pellet in the freeze dryer to de-gas the sample for a minimum of about 2 hrs (gauge should read $\sim 10 \times 10^{-3}$ millibars). Check how the pellet stands-up to the vacuum. Severely cracked samples must be re-made because they may fall apart inside the spectrometer. Alternatively, the samples can sit at room pressure for 4-5 hours prior to use.

Place pellet in round plastic container and store until ready to use.

Clean the pellet mold and end caps thoroughly with kimipex and alcohol.

Loss on Ignition (LOI)

Notes: Use tongs whenever handling cleaned crucibles, and record the results of all weighing steps on a blank LOI form. Because the samples were thoroughly dried at 110°C prior to grinding, the following method assumes that the amount of adsorbed water (H₂O⁻) for each sample is negligible.

Fused quartz crucibles for sample ignition and LOI measurements are stored in the desiccator next to the ScienTech balance. Extras are kept in the drawers beneath the X-Press. If preparing a large number of samples, a maximum of 15 can be ignited at once. Rinse the crucibles with distilled water followed by alcohol. Transfer the clean crucibles to the oven and dry thoroughly at 110°C for approximately 60 minutes. Remove and allow to cool to room temperature in a desiccator.

Weigh a clean, dry crucible and record the exact weight (see below for more weighing details).

Add approximately 1.5 g of sample (more if anticipated LOI is high), and record the exact weight of sample + crucible. Make sure the sample is spread-out inside the crucible, so that it can react completely in the furnace.

Cover each crucible with a Vycor lid (doesn't have to be weighed) to prevent furnace material contaminating the samples. Ignite sample(s) in ash furnace for =5 hrs at 1000°C for basalts, ~900°C for sediments. If your samples contain muscovite, biotite, amphibole, or carbonates, ignite your samples for 6 hours or more.

Remove crucibles and cool to room temperature in the desiccator.

Using tongs (or gloves) to handle the crucibles, remove the crucible lid and accurately weigh and record the ignited sample+crucible weight for each sample.

Store in desiccator until ready to weigh-out for fusions (the sooner, the better).

LOI Calculations

$$\%LOI = 100 * (\text{weight change during ignition}) / (\text{fresh sample weight})$$

LOI calculations can either be made using the LOI forms, or the weights can be entered in the "LOI Short Calc" worksheet and the percent LOI will be automatically calculated.

Note: By convention, weight lost during ignition is typically recorded as a positive LOI value, whereas weight gained is recorded as a negative LOI value.

Weighing LOI samples on Scientech balance (JWS method)

- 1) Initial tare. Both pans empty, function set to Tare, reference weight equals zero. Execute until the program stops. The program will automatically record the tare value.
- 2) Weigh empty crucible. Place crucible on "Unknown" sensor ("Known" sensor remains empty). Set function to Weigh. Execute and then halt after 10-20 counts. The displayed

weight will be an approximate weight. Place an equal amount (within 1 g) of weight on the "Known" sensor. Example: if you measured 18.3 g, place 18 or 19 g on the "Known" sensor. Enter this weight in the Reference weight window. Press Enter and then Execute until the program stops. Record the final crucible weight.

- 3) Weigh crucible+sample. Because you want to weigh out ~1.5 g of sample, mentally add 1.5 g to the actual crucible weight, and add the appropriate amount of counter weight to the "Known" sensor (within 1 g, as above). Update the Reference Weight window. Press Enter. Add sample powder to crucible and then press Execute and then Halt after 10-20 counts to get an approximate weight. If it is within an acceptable weight range, press Execute and run until the program stops. Record the final crucible+sample weight.
- 4) Start another sample. Remove the crucible+sample and all the weights. Enter zero in the Reference weight window and press Enter. Change Function to Tare. Go to step 1.

Standard LOI disclaimer (editorial comment by JWS):

LOI measurements by themselves are generally of limited use due to questions over what species are actually driven-off (H_2O^+ , CO_3 , S, Cl, F, ?), to what extent they are lost, and how much weight is gained through the oxidation of iron (and possibly manganese and sulfur). Not all samples will lose weight during ignition. It is common to measure overall weight gains for fairly fresh basalts containing little water and over 8% FeO. If one is trying to determine the amount of H_2O^+ in a suite of samples, it is much more specific (and accurate) to use the Carlo Erba CHNS chromatograph in the Chemistry Lab. This analyzer has been very successful at measuring H_2O^+ and CO_2 in altered basalts. A brief account of the CHNS analytical procedure is given in the Leg 140 Explanatory Notes and elsewhere.

Weighing LOI samples on Scientech balance (JKL method)

- 1) Initial tare. Put 1 20 g weight on each pan, function set to Tare, reference weight equals zero. Execute until you are satisfied with the precision (HALT) or the program stops. The program will automatically record the tare value.
- 2) Weigh empty crucible. Place crucible (no lid) on "Unknown" sensor ("Known" sensor remains empty). Set function to Weigh. Execute and then halt after 10-20 counts (more if using the filter of course, and you must use the trim data button after 50-75 counts). The displayed weight will be an approximate weight. Place an equal amount (within 500 mg) of weight on the "Known" sensor. Example: if the crucible weighs 18.7xx g, place 18.500 g on the "Known" sensor. Press Enter and then Execute until the program stops or you are satisfied with the precision. Record the final crucible weight (in this case 18.5xxg plus the weight measured and displayed in the window.) Note: You can skip part of this step if you keep a list of appropriate counterweights for the crucibles in use by the balance. Just record the counterweight of each crucible the first time you use it. I keep a spreadsheet of the crucibles I am using and update it from time to time during the cruise.
- 3) Weigh crucible+sample. If you want to weigh out ~1.5 g of sample, add 1.5 g to the actual crucible weight on the LOI form, place the appropriate amount of counter weight to the "Known" sensor (within 500 mg, as above). Subtract the counterweight on the LOI form to

get the “Target Delta Weight.” Add an approximate amount of sample powder to the crucible and then press Execute. Try not to add more than 1.5 g at this step, as it is easier to add sample to the crucible than it is to remove it. Get an initial weight keep adding sample until the measured weight is close to the “Target Delta Weight.” Use the trim data button after adding additional sample and allow enough counts to get a realistic weight. When the “Real Delta Weight” is close to the “Target Delta Weight,” continue weighing until the desired precision is achieved or the program stops. Complete the Pre-ignition sample weight side of the LOI form to record the final crucible+sample weight.

- 4) Start another sample. Go to step 1. (On some days, if the tare seems very stable, I may only re-do the tare every second or third sample, but it is probably a better practice to do the tare every time with a 20 g on each sensor.)

Weighing-out Samples for Fusion (for NT-2100 Bead Sampler)

Materials

Kimwipes
Analytical-grade isopropanol or acetone
Agate mortar and pestle
Weighing paper
Pre-weighed flux charges (7.20 g Johnson-Matthey Spectroflux 105)

Transfer the ignited sample to a clean agate mortar, and grind by hand to a fine powder.

Weigh-out 600 ± 0.2 mg of ignited powder and add to a vial of pre-weighed flux (7.200g). Label vial.

Repeat previous step. ALL samples for major element analysis MUST be prepared in duplicate.

Note: Once the sample has been weighed out, it's not necessary to store it in a desiccator. The small amount of water or CO₂ adsorbed during storage will be lost in the fusion process.

Homogenize the sample/flux mixture by rolling the vial and tapping it on the bench top to clear any powder from the sides of the vial. Note: Violently shaking the vial packs the sample/flux powder around the cap, making it likely some will be lost when the cap is removed.

Weighing Ignited Powders on Scientech balance (JWS method)

Initial tare. "Known" sensor empty, weighing paper on "Known" sensor. Function set to Tare, Reference weight equals zero. Execute until the program stops. The program will automatically record the tare value.

Weigh powder (approximately). Place an approximate amount of ignited powder on weighing paper ("Unknown" sensor). Place a 1 g counter weight on the "Known" sensor. Set function to Weigh. Enter 1.00000 g in the Reference weight window. Press Enter. Click on Execute and then halt after 10-20 counts. The displayed weight will be an approximate weight. Adjust the amount of sample on the weighing paper and repeat the Execute and Halt steps until the weight is within acceptable error.

Weigh powder (accurately). Click on Execute and let the program run until it stops (and beeps). Note the displayed weight and adjust the amount of sample accordingly. Repeat the Execute and run step until the weight is within acceptable error.

Start another sample. Remove the sample and the 1 g weight. Enter zero in the Reference weight window and press Enter. Change Function to Tare. Go back to step 1.

Note: for alternative method, see following page.

Weighing Ignited Powders on Scientech balance (JKL method)

Initial tare. Place 600 mg from the calibration weight set on "Known" sensor. Place 600 mg from the other weight set on the weighing paper on the "Unknown" sensor. Function set to Tare, Reference weight equals zero. Execute until the program stops or you get the precision you want. The program will automatically record the tare value when it stops or when you execute HALT.

Weigh powder (approximately). Remove the weights from the paper on the "Unknown" sensor and place an approximate amount of ignited powder on the paper in its place ("Unknown" sensor). Set function to Weigh and execute (trim data as necessary if using the filter). The objective is to add 600 mg of ignited powder to the paper so that the weight recorded equals zero. This eliminates doing any arithmetic. Continue adding sample and trimming the data until you get it close. Longer weighing times will be required as you get closer to the desired value (which is 0.00000).

Weigh powder (accurately). I try to get the final weight to as close to zero as possible. I always persist until I get +0.00050 or - 0.00050. On a good day (using the filter) I can get much closer than that. With the filter, you cannot use the "stats" feature, so you can wait for a certain number of counts, but I don't. I consider the number of measurements I have taken prior to the final one, the graph, and the precision shown in the +/- window to decide when to halt the weighing process. It becomes a Zen thing with experience. When you're there, you should know it. If you can't get there, it is not a good day/time to weigh. Try again later.

Start another split/sample. For the second split of the same sample, I use the same paper and the same tare. I do not put the weights back on paper that may still have sample adhering to it and I do not reuse paper for a different sample. For a new sample go back to step 1. Use a new paper and retare with 600 mg on the paper.

Fusing Samples with the NT-2100

Materials

Kimwipes
Analytical-grade isopropanol or acetone
Pre-weighed sample/flux charges (0.600g sample / 7.20 g Johnson-Matthey Spectroflux 105)
LiBr releasing agent (8.6 Molar solution; 7.5 g LiBr / 10 mL H₂O)
Micro-pipettor (20 μ L)
Pt₉₅Au₅ molds (simply referred to as Pt molds or crucibles) with Pt₉₅Au₅ lids
Round plastic sample containers
Sample labels

Start-up Procedure

- 1) Slowly open the water flow valve until you hear a faint click.
- 2) Turn the power switch on. The red ready light and the red water lamp should be on. Note: If the water lamp is not on, turn-off the power switch and make sure you have proper water flow. Please wait a few minutes for the unit to warm up.
- 3) Open the door on right side of the unit to check the settings:

TEMP ADJ: Set to 6.7 for no-lid operation.

ROTA: This is the rotation speed for agitation. Three is the fastest and is probably fine for most samples and sea states.

MOD: Set on 1 (the center position) all the time.

TEMP/DC Set this switch on TEMP (up) to display the reaching temperature (rather than voltage) on the MONITOR display on the front panel.

DCI/DCV Set this to DCV for all normal operations. The DCI setting is for troubleshooting the electronics.

- 4) Now the unit is now ready to operate.

Shutdown

- 1) Turn off the power (SOURCE) switch.
- 2) Turn off the cooling water at the valve. The handle should be at 90° to the hose when the valve is completely closed.

Precautions and Safety

- 1) Use the correct crucible.
- 2) Do not heat without a crucible in place.
- 3) Always use seconds as units on the timers. The internal, preset program will only work if the timers are all set in seconds.
- 4) When overcurrent is detected, the automatic power cut-off will engage, and an alarm will sound. In this case, the <READY> light will go out. After this, it is necessary to reset the breaker inside the right side panel of the unit.
- 5) When the water pressure drops and/or the water temperature is not in the correct range while operating the equipment, the automatic power cut-off will engage and an alarm will sound. In this case, the <READY> and <WATER> lights will both go out. Correct the water temperature/flow condition and reset the breaker inside the unit.

Normal Fusion Procedure

Automatic operation is the normal mode of operation for fusing most samples. Use Manual operation (see below) for re-fusing cracked/broken discs or fusing unusual samples. (I use automatic operation for fusing or remelting. For remelting I set the timers at 120-120-240. In both cases I have the lid on so the potentiometer is set to 7.2. JKL)

-) Turn the auto/manual switch to auto.
-) Check that the times (in seconds) for <FUSE1>, <FUSE2>, and <AGIT> are set correctly (100, 100, 240, respectively). (I used 240-240-360 to make the standard and sample beads on Leg 179 JKL)

Note: If temperature on the LED readout is not correct during any phase of operation, you may adjust the <FUSE1> or <FUSE2> potentiometers until the desired temperature is achieved, but this should not have to be done during normal automatic operation.

-) Transfer the sample/flux mixture to a Pt crucible by tipping it ~30-45° and carefully pouring in the sample/flux mixture. Tap the side of the vial to transfer as much powder as possible. It's not unusual for a slight dusting of material to remain in the glass vial. Any more than that must be removed.
-) Add 20 μ L of LiBr releasing agent directly to the sample/flux mixture with a micro-pipette.
-) Open the hood and place the sample in the furnace. Close the hood and press the <START> button. Note: If you want to stop the operation at any time, push the green <STOP> button.
-) When the operation is completed, the exhaust motor will turn-off and the unit will beep.
-) Immediately open the hood and use the tongs to move the sample to the cooling stand. The fan will turn on automatically and run for a maximum of four minutes. (I set a timer for 14 minutes so I can be back at the Bead Sampler for this step. If the bead cools when the ship is not level, it may be necessary to swirl the crucible so the bottom is completely covered. After the unit beeps, I remove the lid and set it on the ceramic plate. I pick up the crucible with the tongs and transfer it to the cooling stand, watching it until it has cooled sufficiently that I am sure it will remain level. Then I put the lid back on and start the next sample. JKL)
-) Let the sample cool for about 60 to 90 sec, or until it releases from the mold (you can actually watch it happen). After it has released, use the tongs and transfer the mold to a hotplate set at ~230°C.
-) Keep the mold and disc on the hotplate for about 10 minutes, or until the next disc has been fused.
-) Turn over the mold to dump out the disc. It should pop out of the crucible easily. Only very slight tapping should be needed.
-) Continue to heat the disc for at least another 20-30 minutes.
-) Transfer disc to cooling mat (a 'green meany' pad works!), and let it cool to room temperature.
-) Place disc in a round plastic container and store in desiccator cabinet when not in use.

Note: I just leave the crucible on the cooling stand until it is pretty cool, drop the bead out onto the blue pad, stick a label on it, and put it in the plastic box. Sometimes it goes right into the cassette. With this flux and most lithologies, there seems to be no need to use the hotplate. JKL)

-) If fusing another disc of the same sample, re-use the same mold without cleaning.
-) If preparing a new sample, clean the mold by

I have not had to do much cleaning of the crucibles. Between "sessions" I put the lid and sometimes the crucibles into the 50% HCL bath, but often just the lid needs cleaned. On 179, I didn't even bother polishing the two crucibles I used until the end of the cruise. They just didn't need it. If the crucibles had been sitting in the cabinet, I cleaned them like you would a

microscope lense, with lense-cleaning tissues and distilled water from my breath. I did not use kimwipes because I think they scratch.

Re-fusing Glass Discs.

- 1) Turn the auto/manual switch to manual.
- 2) Put the sample in the crucible, cover with a lid and set the crucible in the furnace. The lid helps keep glass pieces in the mold, as most glasses will shatter on heating.
- 3) Push the <START> switch.

Note: In the manual operation mode, the <FUSE1> timer, the <FUSE2> timer and temperature adjuster, and the <AGIT> timer are not operational, so you have to monitor the time and turn the agitation on and off manually. .

-) Once the sample nearly molten (~90 sec) press the <STOP> button and remove the lid.
-) Press <START> again and after about 90 sec, press <AGIT>.
-) Agitation should continue for another 60-90 sec.
-) Push the yellow <AGIT> button (off) and then the red <STOP> button when finished.
-) Immediately open the hood and use the tongs to move the sample to the cooling stand. The fan will turn on automatically and run for a maximum of four minutes.
-) Let the sample cool for about 60 to 90 sec, or until it releases from the mold (you can actually watch it happen). After it has released, use the tongs and transfer the mold to a hotplate set at ~230°C.
-) Keep the mold and disc on the hotplate for about 10 minutes, or until the next disc has been fused.
-) Turn over the mold to dump out the disc. It should pop out of the crucible easily. Only very slight tapping should be needed.
-) Continue to heat the disc for at least another 20-30 minutes.
-) Transfer disc to cooling mat (a 'green meany' pad works!), and let it cool to room temperature.
-) Place disc in a round plastic container and store in desiccator cabinet when not in use.
-) If fusing another disc of the same sample, re-use the same mold without cleaning.
-) If preparing a new sample, clean the mold by

Note: For basalts at least, I do this on automatic with the lid on. I just change the times to 120-120-240. Seems to work fine. Just remember to change the times back when you are finished.
JKL

Operating XRF Spectrometer

Spectrometer Start-up

To start the XRF from a dead cold shut down, first turn-on the power switches located at the back of the instrument. Two of these switches are on the far left side behind the standards desiccator, and six more are on the back of the spectrometer cabinet, left side (as viewed from the front!). These should be switched-on in the in the following order.

Behind the standards desiccator:

1. Gonio Drivers Rack
2. XQB Rack

Behind the spectrometer cabinet:

3. Spectrometer Pump
4. Primary Chamber Pump
5. Electronic Cabinet
6. Low Volt XQC Rack
7. X-ray Cooling System
8. X-ray Power Supply

Turn-on the computer (if not already on) and wait for the boot-up sequence. If it does not automatically start "Windows", enter "W" to start Windows and the ARL software.

When the computer asks you to log on, enter **XRF** with the password **XRF**. You will then be asked if you want to load files. It is best to click on all three boxes to load the High Voltage Calibration, Position Calibration, and Line Library files. This will take several minutes.

The XRF should be pumping down the spectrometer chamber. The main chamber will have pumped down quickly (small volume). If the tank (AKA spectrometer chamber) was opened prior to the start-up, it may take a while to reach an acceptable vacuum. Occasionally, it may take 24+ hours to reach a reading of 1000 Pa.

To check the vacuum in the spectrometer chamber, select:

Select Utilities Menu Special Functions Terminal Mode. In the Enter Message box, and type "DV 1,7". The vacuum level will be displayed on the Spectrometer Status readout on the chamber cover.

If the level has stabilized at some value =1500 Pa, use the Set Environment command.

Select Analysis Menu Initialize Instrument Set Environment.

If the <Current Environment> box indicates Vacuum, click <Air> and <OK>. Re-select Set Environment. Click <Vacuum> and <OK>.

Turning-on the X-ray Tube

Although the XRF will let you turn on X-rays without a vacuum, doing so could cause damage.

Switch on the cooling water from the Haskris.

Analysis Menu Initialize Instrument Set X-ray.

Set the generator settings depending on how long the tube has been off, and its intended use.

If the tube has been off longer than 24 hours (or is a new tube), start at 5kV/10mA and bring it up by increments of 5-10 kV/mA every 1-2 hours;

If the tube has been off for more than a couple of hours, warm it up at 20kV/20mA for at least an hour, then proceed to the normal settings using the On Slow command (30kV/80mA for majors, 50kV/50mA for traces);

If the tube was off only a short time, bring it up to the normal settings using the On Slow command (30kV/80mA for majors, 50kV/50mA for traces).

If you want the instrument to "idle" for a while, set the X-rays to 20kV/20mA using the On Slow command.

Zero the Goniometers

The goniometers must be zeroed one at a time.

Select Analysis Menu Initialize Instrument Zero Goniometer.

To zero Goniometer 1, click <OK> and wait approximately 2 minutes.

To zero Goniometer 2, click <Next> before clicking <OK>. Wait approximately 2 minutes.

The spectrometer should now be ready to run.

Shutting-off X-rays and Spectrometer

Turning-off the XRF is essentially a matter of reversing the start-up process, with a few modifications.

First turn the X-ray tube off by selecting:

Analysis Menu □ Initialize Instrument □ Set X-ray. Click on <X-ray off>.

After 5 minutes turn-off the cooling water flow from the Haskris. This will allow enough time for the tube to cool down, but not enough to form condensation.

Change the pump selection switch to “XRD” on the back of the Haskris cooling unit.

If the spectrometer chamber is to be opened, vent it by selecting:

Analysis Menu □ Initialize Instrument □ Set Environment. Click <Air> and <OK> .

Note: If you do not have to open the Spectrometer chamber it is best to leave the environment on vacuum.

Next, turn-off the eight power switches in the reverse order that they were turned-on. The proper order for turning them off is:

1. X-ray Tube Power Supply
2. X-ray Tube Cooling System

Note: Not all of the devices should be switched off unless it is necessary to shut down the system completely. The XQB Rack and Gonio Drivers Rack should not be turned off unless absolutely necessary. If working with the electronics (without opening the chamber) do not turn off the vacuum pumps.

3. Low Voltage XQC Rack + Thermostabilization
4. Electronic Cabinet
5. Vacuum 2 - Primary Chamber Pump
6. Vacuum 1 - Spectrometer Pump
7. XQB Rack
8. Gonio Drivers Rack

Changing the P-10 Gas

We need to write this part!!

Loading Samples

The cassettes are numbered 1-8 with the numbers engraved on the bottom, and black stripes on the sides representing the number in binary code.

*** Under no circumstances should cassette 8 be used for anything other than the drift standard (POOP). **It must not be opened.** Cassette 8 is also indicated by the red dots on the lid. Opening or otherwise disturbing the drift standard could cause errors in all analyses, as they are corrected by comparing to standardized POOP intensities. ***

Place samples in the cassettes. There are two types of plastic inserts that are used to center the glass discs or pressed pellets. The light-colored inserts with blue stripes facing upwards are for glass discs, the dark-colored ones for the pressed pellets. In general, the cassettes will be marked with colored labels their lids - blue for traces, green for majors.

Fasten the lids on the cassettes making sure they are on securely. If they come loose during analysis, it may be necessary to open the spectrometer chamber to remove them.

Turn over the cassettes and check that the samples are mounted correctly. The glass beads in particular tend to tilt if they are wedge-shaped (to correct this, place a couple of Kimwipes or a section of sponge between the sample and the bottom of the lid's spring assembly).

Place the cassettes on the sample changer (sample-side down) along with cassette #8. If the samples are unknowns, load one of the cassettes (usually the last to be run) with a standard that is similar to the expected results of the unknowns. This is the precision standard and is recorded in the Leg notebook.

Note: Joel told me that he had problems with the centering discs for the beads being thicker than the bead, so he wasn't using them. I don't use them either now. I just put the bead in there with a folded-up Kimwipe on it, center it as best I can, and put the top on. I still use the centering rings for the pellets, but I started worrying about the cassettes unscrewing since I have the rotation on and the sample is in there for so long. I started putting a Delron disc in on top of the sample to increase the spring tension on the top of the cassette. Of course, this makes the cassette heavier, but I considered this not much of a problem compared to having a cassette disassemble in the bell.

Running Trace Element Samples

1. Load samples into cassettes (don't touch #8, POOP!).
2. Drag down menu 'Preparation'.
3. Click on 'Analytical Programs'.
4. Select a program from the small window on the left, and press the Copy button. Enter a new name for the program, using "run" names similar to TRUNxx. Press Exit.
5. Drag down 'Preparation' and click on 'Tasks'.
6. Choose 'UNKNOWN SAMPLES %'. Create a new task name. For convenience, use the same name as the program (i.e. TRUNxx). Press Enter.

Note: I use the same task over and over for majors and traces. I give it the same name as the program. I don't see the advantage to having a different task for each run. I like to be able to retrieve results for everything with one task. Of course, you can only retrieve 100 results this way, so if I get that many, I might increment the task name every hundred samples. JKL

7. Click on 'Default Program'. Choose your program.
8. Set Repeat Count to 2.
9. Activate 'Predefined Sample List'
10. Click on the 'Define Samples' box near the bottom.
11. A new form will appear. Activate the field 'Sample Name' in the 'Sample Identifier' box. Enter the sample name as Site/Hole/Core/Type/Section without spaces (i.e., 989B12R-1). Enter the sample interval in the Sample Number field. Note: Duplicates cannot have the same name so you will have to use a & b at the end of the interval (i.e., 35-39a).
12. Enter the appropriate cassette number.
13. Press Append and go on to the next sample. Enter the precision standard(s) but not Poop. Poop is automatically added during 'Batch' creation.
14. Exit back three windows to main menu (press OK, OK, Exit).
15. Once again select 'Preparation' then 'Batches'.
16. Create a new batch file. Yes, use TRUNxx etc. again.

Note: Janus will only accept 5 characters for the RUN ID, so if you use TRUNxx as the RUN ID, it will be too long. You might want to use just T_xxx or M_xxx for traces and majors, or something with just five characters. JKL

17. Click on 'Analysis'.
18. In the 'Category' box activate 'Standardization Update'.

19. The appropriate programs will appear in the Analytical Task box. Choose 163TDRF and press 'OK'.
20. Click on 'Analysis' again.
21. In the 'Category' box activate 'Concentration in Unknown Samples'.
22. Choose the Task you just created.
23. Exit back three windows to main menu (press OK, OK, Exit).
24. Pull down the 'Analysis' menu and choose 'Batch Operation'.
25. Choose the batch you just created and press OK (the repeat value should normally remain equal to one). The spectrometer should start your batch run.

Running Major Element Samples

1. Load samples into cassettes (don't touch #8, POOP!).
2. Drag down menu 'Preparation'.
3. Click on 'Analytical Programs'.
4. Select a program from the small window on the left, and press the Copy button. Enter a new name for the program, using "run" names similar to MRUNxx. Press Exit.
5. Drag down 'Preparation' and click on 'Tasks'.
6. Choose 'UNKNOWN SAMPLES %'. Create a new task name. For convenience, use the same name as the program (i.e. MRUNxx). Press Enter.
7. Click on 'Default Program'. Choose your program.
8. Set Repeat Count to 2.
9. Activate 'Predefined Sample List'
10. Click on the 'Define Samples' box near the bottom.
11. A new form will appear. Activate the field 'Sample Name' in the 'Sample Identifier' box. Enter the sample name as Site/Hole/Core/Type/Section without spaces (i.e., 989B12R-1). Enter the sample interval in the Sample Number field. Note: Duplicates cannot have the same name so you will have to use a & b at the end of the interval (i.e., 35-39a).
12. Enter the appropriate cassette number.
13. Press Append and go on to the next sample. Enter the precision standard(s) but not Poop. Poop is automatically added during 'Batch' creation.
14. Exit back three windows to main menu (press OK, OK, Exit).
15. Once again select 'Preparation' then 'Batches'.
16. Create a new batch file. Yes, use MRUNxx etc. again.
17. Click on 'Analysis'.
18. In the 'Category' box activate 'Standardization Update'.
19. The appropriate programs will appear in the Analytical Task box. Choose MDRIF(??) and press 'OK'.
20. Click on 'Analysis' again.
21. In the 'Category' box activate 'Concentration in Unknown Samples'.
22. Choose the Task you just created.

23. Exit back three windows to main menu (press OK, OK, Exit).
24. Pull down the 'Analysis' menu and choose 'Batch Operation'.
25. Choose the batch you just created (the repeat value should remain 1) and press OK. The spectrometer should start the run.

Notes: This program runs all elements for the sample (twice) before moving to the next sample. (The old system ran all samples for the same element). Since this happens, there are several hours between the drift standard run and the analysis. For most crystals this does not matter (The manual suggests running the drift standard every 48 hours). However PET is extremely temperature sensitive. A 1 degree change in temperature changes the final value by about 1% relative. This again would not be significant except that Si and Al usually total 65-75% of the material, thus the entire analysis would be off .65-.75%. Not good. If temperature drift problems occur, an alternate analyzing procedure that helps compensate for PET crystal warming is suggested below.

Steps 1-20 as above.

21. Activate 'Standardization Update' again and choose PETDRIFT.
22. Repeat step 21 until you have 3 PETDRIFTs. (See explanation below)
23. In the 'Category' box activate 'Concentration in Unknown Samples'.
24. Choose the Task you just created.
25. Click on one of the PETDRIFT lines.
26. In 'Step List Manipulation' click on move.
27. Click on the Analyse line with cassette 3.
28. Click on move again.
29. Repeat steps 25-28 placing PETDRIFTs between 4-5 and 6-7.
30. Exit back three windows to main menu (press OK, OK, Exit).
31. Pull down the 'Analysis' menu and choose 'Batch Operation'.
32. Choose the batch you just created (the repeat value should remain 1) and press OK. The spectrometer should start the run.

Balance Systems User Guide

v.179

Accelerations, induced by the ship's motion, make precise weighing at sea difficult. To measure weights in this environment it is necessary to cancel out all forces acting on the balance that are not directly related to the sample weight. These include: ship's heave and vibration, electronic drift due to temperature and power fluctuations, and vertical components of roll and pitch. In addition, there is a long term (from hours to days) differential electronic drift.

To weigh at sea it is thus necessary to use a motion compensated balance. The motion compensated balances used aboard the *Joides Resolution* are based on the design concept described by Childress and Mickel (Deep Sea Research, Vol 27A, *A motion compensated shipboard precision balance system*).

Another aspect of weighing at sea is to collect data over a time period sufficient to define the ship's heave. The *Joides Resolution* has a natural period of about 5 to 7 seconds so it is important to weigh for at least 25 to 35 seconds. In compound swell conditions it may be necessary to double this time.

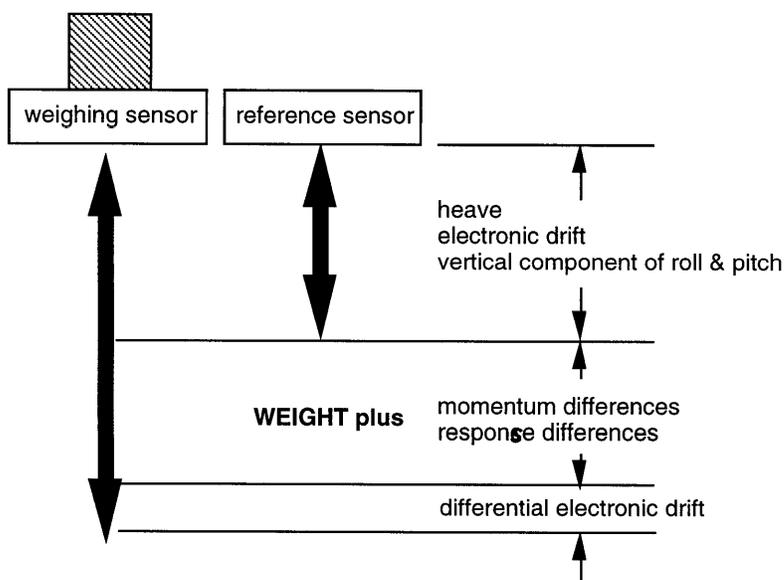
On-board the *Joides Resolution*, there are two types of compensated balance systems:

- Scientech Balances; and
- Cahn Microbalance.

Both systems are controlled by a LabView software program.

A. Scientech Balances

The Scientech balance system is a traditional motion compensated balance system. This system uses a reference balance with its load matched to the unknown weight on the sample balance. The reference balance partially compensates for ship motion and vibration. Analog differences in output between the reference sensor and the weighing sensor are calculated in real time and can be removed from the true weight value by averaging.



A. 1. Principle and Mode of Operation

The Scientech balance system is composed of two Scientech top loading balances (model 202 004), two controllers, a National Instrument SCB-86 Interface, and a National Instrument (NI) DAQ board.

The two top loading balances are weighing sensors. The “known” sensor acts as a reference sensor while the “unknown” sensor is the actual weighing sensor. Each sensor’s basic weight signal is an unfiltered analog voltage. The no load signal is typically 1.50 volts. The signal will increase linearly at the rate of 1 volt per 10 grams of weight placed on the weighing pan to approximately 6.50 volts at full capacity (50 grams). Each sensor has a factory rated precision of +/- 0.1 mg. On board the ship, full capacity is about 30 grams (to prevent clipping at the 6.5 volt limit during heavy seas) and the precision is of +/- 0.5 mg.

The controllers provide the power to the sensors and monitor/adjust the voltage back from/to the sensors. There is one controller per sensor. The basic controller has a power switch, an analog weight signal output (Analog Output), and a calibration adjustment for the analog output (Analog Calibrate). The Analog Output has two banana jacks (tare and gross) in the front of the unit that permit external recorders and display equipment to be used with the 202. On board the *Joides Resolution*, only the gross output is used. It connects to a National Instrument SCB-86 box interfaced with a NI DAQ analog-to-digital board. The exact calibration is set by the Analog Calibrate adjustment (rear panel of unit). The Analog Calibrate is factory set and should not require further adjustment.

The voltage from the two sensors is sampled and digitalized using a two channel NI DAQ board. The two digitalized signals are then passed directly to a LabView program. The program emulates the functions of a differential amplifier which subtracts voltages of the two sensors from each other and then adjusts the gain and zero values of the reference sensor so that the response of the reference sensor will match the response of the weighing sensor (i.e. till the difference shows 0.00 mV output).

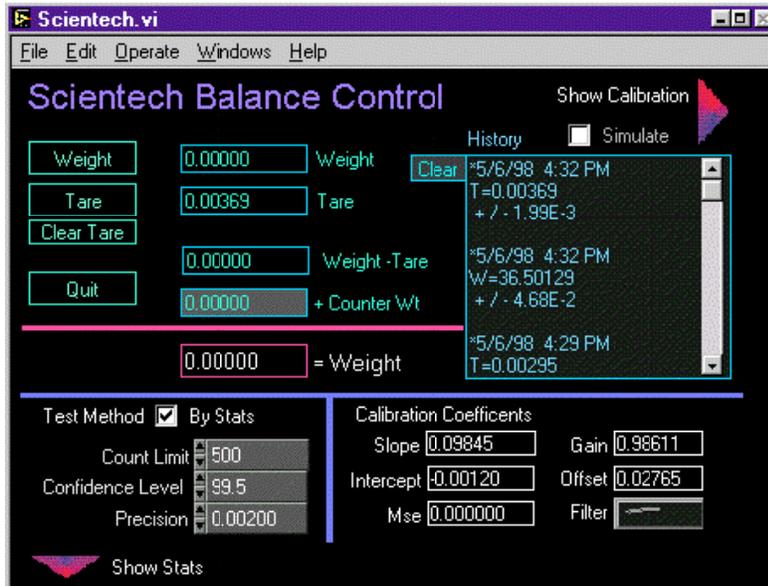
The LabView software used to control the Scientech balance is an icon based programming language developed by National Instruments. Applications generated with LabView are called Virtual Instruments (VIs). VIs actually run under the LabView application as a collection of coded resources. The version of LabView used in all the laboratories is not a full blown version but a run-time version (4) which does not allow editing of the program or window display. To understand more about LabView 4 and VIs, refer to the *LabView 4 Getting Started Manual*.

The Scientech LabView application is located on the hard disk in the Scientech folder. The VI used to control the Scientech balances is called *Scientech.vi* and is launched by double clicking on the Scientech icon on the desktop.

Scientech balance systems are used on-board in three Laboratories: Chemistry, X-Ray and Physical Properties. At the beginning of each Leg, these balances need to be tuned and calibrated before being use for weighing. This should to be done in port.

A. 2. Tuning the Sensors

Once the Scientech.vi is loaded, the following Control window is displayed:



Clicking on the Show Calibration arrow in the upper right of the Control window will open an extension to the right of this window. Clicking on the Tune Sensor Utility button will load the Scientech Tuner.vi. as displayed below:

I can't get this picture to load!!

The tuner.vi allows the user to apply a gain and zero value to the reference sensor's output so as to match the zero and gain of the two balances. The matching procedure is carried out by adjusting the output to zero mV with a zero load and then placing a 20g weight on each sensor and adjusting again the output to zero. The procedure is repeated until both conditions of balanced load (zero g and 20g) shows 0.00 mV output.

To perform the zero and gain tuning, proceed as follows:

- With no load on the balances, click on the Set Offset button. The user can set the zero (and gain) values by either using the Correction Increment knob (Auto) or directly by entering the values into the digital controls (Manual);
- If the Auto mode is selected, move the Correction Increment knob until the sensors' readings displayed below the right vertical scale are approximately 0.00 (i.e., the purple and grey arrows will be superimposed in the blue box), click on the Stop DAQ button to stop data acquisition;
- Put a 20g weight on each balance, click on the Set Gain button and move the Correction Increment knob until the sensors' readings are approximately 0.00. Click on Stop DAQ;
- Remove the weights and set the offset (zero) again. Repeat the previous steps until both the zero and gain are set to read 0.00;
- Saving the Changes will store the new values of the offset and gain in the program and will exit the tuner.vi back into the Control window. The offset and gain are shown in digital indicators in the Control window next to the Calibration Coefficients indicators.

Note 1: In LabView, the VI windows contain various types of controls and indicators. Controls are means to supply data to the VI. To enter or change values in a digital control, click on the control itself and enter the new value or use the increments button (up/down arrows) on the left edge of the control. Indicators display data generated by the VI and cannot be modified. Buttons perform functions as designed in the VI.

Note 2: You can tune the zero and gain by either adjusting the last Leg's readings or by starting from 0.00 after clicking on the Reset Gain and Offset button.

A graph of each sensor's signal is provided for the user's information. When the output difference is minimum, the signals will almost be superimposed.

A. 3. Calibrating the Sensors

Calibration of the sensors is performed at the beginning of each Leg, in port. Clicking on the Show Calibration arrow in the upper right of the Control window will open an extension (Calibration window) to the right of the Control window as displayed on the next page:

Every time the Calibration window is open, the Calibration Coefficients in the Control window are automatically set to the following default values: slope =1 and intercept = 0. When clicking on either the Tare or Weigh button, readings displayed in the associated digital indicators will be actual voltage readings.

Note: Calibration parameters are set differently depending on the balance usage in a specific Laboratory. In the Chemistry Lab., the Scientech balance is used most of the time only to weigh chemicals and, as a result, precision is not critical. The following paragraph illustrate a calibration as performed in the Chemistry Lab.

Weighing Procedures

General Weighing

Note: The Scientech system must be set-up, tuned, and calibrated before starting.

- 1) Set "reference weight" to 0.00000.
- 2) With both sensors empty, select the Tare function and press Execute. When finished, the tare value will automatically be entered into (and displayed by) the program.
- 3) Place the unknown weight (crucible, weighing paper, etc) on the sensor marked "Unknown," and leave the "Known," or reference sensor empty.
- 4) Select the Weight function and press Execute. After ~5 seconds, press USER HALT. The displayed weight is an approximate weight of the unknown.
- 5) Place an appropriate amount of counterweights (within 1.0 g of the approximate value) on the "Known" sensor.
- 6) Change the "reference weight" value to reflect the amount on the "Known" sensor.
- 7) Select the Weight function and press Execute. When the program stops, the exact weight will be displayed.

Weighing LOI Samples

Note: The Scientech system must be set-up, tuned, and calibrated before starting.

-) Initial tare. Both pans empty, function set to Tare, reference weight equals zero. Execute until the program stops. The program will automatically record the tare value.
-) Weigh empty crucible. Place crucible on "Unknown" sensor ("Known" sensor remains empty). Set function to Weigh. Execute and then halt after 10-20 counts. The displayed weight will be an approximate weight. Place an equal amount (within 1 g) of weight on the "Known" sensor. Example: if you measured 18.3 g, place 18 or 19 g on the "Known" sensor. Enter this weight in the Reference weight window. Press Enter and then Execute until the program stops. Record the final crucible weight.
-) Weigh crucible+sample. Because you want to weigh out ~1.5 g of sample, mentally add 1.5 g to the actual crucible weight, and add the appropriate amount of counter weight to the "Known" sensor (within 1 g, as above). Update the Reference weight window. Press Enter. Add sample powder to crucible and then press Execute and then Halt after 10-20 counts to get an approximate weight. If it is within an acceptable weight range, press Execute and run until the program stops. Record the final crucible+sample weight.
-) Start another sample. Remove the crucible+sample and all the weights. Enter zero in the Reference weight window and press Enter. Change Function to Tare. Go to step 1.

Weighing Ignited Powders for Fusion

Note: The Scientech system must be set-up, tuned, and calibrated before starting.

-) Initial tare. "Known" sensor empty, weighing paper on "Known" sensor. Function set to Tare, Reference weight equals zero. Execute until the program stops. The program will automatically record the tare value.
-) Weigh powder (approximately). Place an approximate amount of ignited powder on weighing paper ("Unknown" sensor). Place a 1 g counter weight on the "Known" sensor. Set function to Weigh. Enter 1.00000 g in the Reference weight window. Press Enter. Click on Execute and then halt after 10-20 counts. The displayed weight will be an approximate weight. Adjust the amount of sample on the weighing paper and repeat the Execute and Halt steps until the weight is within acceptable error.
-) Weigh powder (accurately). Click on Execute and let the program run until it stops (and beeps). Note the displayed weight and adjust the amount of sample accordingly. Repeat the Execute and run step until the weight is within acceptable error.
-) Start another sample. Remove the sample and the 1 g weight. Enter zero in the Reference weight window and press Enter. Change Function to Tare. Go back to step 1.

Appendix 1

Hardware Set-up (NB-A2000 Analog to Digital Board)

- 1) Install the NB-A2000 in any Mac II's (cx, si or ci) following the installations instructions in the National Instruments Manual (kept in System Managers Office)
- 2) You'll need to install the NB-GPIB in Mac II's (cx, si or ci) control panel folder and restart your Mac. Diskettes kept in System mangers office.
- 3) Connect channels 1 and 2 of the board to the output of the sensors 1 & 2 respectively. The output voltages are taken from the sensor's power supply box. Have the ET's make a special DB-25 to BNC connector for you. **WARNING! Disconnect the 120V power feed running through the DB-25 connector.**

Appendix 2

Special Problems with using the NB-A2000 board:

Data from the NB-A2000 board must be stored in LabView's memory space. This can cause two problems:

- 1) If you try to collect more than 4096 (?) samples on each channel you'll run out of memory and LabView will gracefully quit on you with out crashing the MAC.
- 2) The memory used is contiguous and locked. That is the Macintosh memory manger cannot move it around. If you abort the VI that allocated this memory it will remain allocated. If you do this to many times you'll notice that your Mac has started to slow down. Windows will take up to 10 seconds to refresh or open. The only way to cure this problem is to restart. I have written the VI's to deallocate memory when they are executed in a normal manner. Aborting (clicking on the "Stop Sign" button) will bypass the deallocation procedure.

Appendix 3

Opening the Spectrometer Chamber

Follow all the steps for shutting-off and venting the XRF.

To open the chamber, lift-up the outside cover over the spectrometer chamber.

Open the silver valve on the front right corner of the chamber and wait for the chamber to reach ambient pressure.

Remove all of the bolts holding the chamber lid and lift the lid until the latch engages to hold the lid open. If the lid does not open easily, there are two bolts on the right of the lid that can be screwed down to break the seal. If theses bolts are used, immediately return them to the "up" position to avoid possible damage when closing the chamber.

Closing the Spectrometer Chamber

To close the chamber, set the lid down, close the silver valve, and tighten the bolts until snug. Do not over tighten! It could crack the chamber.

SET VACUUM and then turn on vacuum pumps. (??) or Turn on vacuum pumps with the SET ENVIRONMENT <Vacuum> window.