

STANDARD OPERATING PROCEDURE NO. 28**X-RAY FLUORESCENCE ANALYSIS**

REVISION LOG		
Revision Number	Description	Date
28.0	Original SOP	Nov. 12, 2003
28.1	Revisions by V. Lueth after comments by GMLR on Nov. 17, 2003, incorporate changes to DQOs by McLemore	November 18, 2003
28.2	Revisions by Phil Kyle	December 2, 2002
28.3	Revisions by George Robinson (QA/QC, typos)	January 17, 2004
28.4	Prepared appendix with procedures for XRF sample prep at NMSU Kelly Donahue	Feb. 21, 2005
28v5	Edits & comments LMK, I also attached the .pdf of NMSU procedures for Kelly	2/22/05 & 6/27/05
28v6	Changes accepted LMK, sent to Jack H. to post new version on website	8/16/05
28v7	Edit and reorganization by LMK in preparation for finalizing to send to Jack Hamilton to post on Molycorp Project website and to George Robinson for lab audit	3/29/07
28v8	Editorial by SKA	10/23/08

1. PURPOSE AND SCOPE

This document outlines the procedures for sample analysis using x-ray fluorescence (XRF) techniques for the Molycorp Rock Pile Stability Project. The x-ray fluorescence spectrometer will be used to analyze silicate materials from rock pile, alteration scar, and bedrock samples. The principle product generated will be quantitative major element oxides (SiO_2 , TiO_2 , Al_2O_3 , total Fe as Fe_2O_3 , MnO , MgO , CaO , K_2O , Na_2O , P_2O_5) and trace (V, Cr, Cu, Ni, Zn, Ga, As, Rb, Sr, Nb, Mo, Zr, Y, Ba, Th, U) element analyses and semi-quantitative (S, Cl, Se, Bi and W) analyses. XRF procedures are available for qualitative characterization of any solid material (as long as it can be crushed to fit in a 50 mm wide sample cup) with any elemental compositions located between F and U in the periodic table.

2. RESPONSIBILITIES AND QUALIFICATIONS

The Team Leader and Characterization Team will have the overall responsibility for requesting analyses by XRF. They will be responsible for assigning appropriate staff to assist the XRF-lab supervisor and/or x-ray lab manager in sample preparation necessary to implement this SOP. The XRF-lab supervisor and/or x-ray lab manager will ensure that the procedures are followed accurately.

All personnel performing these procedures are required to have the appropriate health and safety training. In addition, all personnel are required to have a complete understanding of the procedures described within this SOP, and to receive specific training regarding these procedures, if necessary. Only personnel trained on the Philips PW 2400 will undertake the procedures outlined in this protocol. The XRF-lab supervisor or the x-ray lab manager is the only personnel qualified to provide training in XRF equipment and software use.

The XRF-lab supervisor or the x-ray lab manager is responsible for reporting deviations from this SOP to the Team Leader.

3. DATA QUALITY OBJECTIVES

The XRF analyses will be used to address objectives 2, 3, 4, 5, and 6 in the data quality objectives outlined by Virginia McLemore for the "Geological and Hydrological Characterization at the Molycorp Questa Mine, Taos County, New Mexico".

- To determine the major and trace element compositions of various materials
- To determine if the sequence of host rock hypogene and supergene alteration and weathering provides a basis to predict the effects weathering can have on mine rock material. XRF will allow for the determination of major and trace element chemistry unique to particular types of alteration and weathering.
- To determine if cementation forms in the rock piles and the effect of such cementation on the stability of the rock piles. XRF analysis will determine changes in material chemistry, when compared to other analysis, in order to infer the mobility of particular elements and the accumulation of others (i.e. cements).

- To determine the concentrations of pyrite and carbonate minerals so that a representative sample goes into the weathering cells. XRF will allow for the quantification of the elements present, namely Fe and other carbonate cation elements (Ca, Mg, Mn).
- To determine how the concentration and location of pyrite in the waste rock piles affect the weathering process. XRF analyses will quantify the amount of iron and, to a lesser extent, the amount of sulfur in the materials.

DATA COLLECTION:

Major and trace element concentrations are acquired from fused glass discs and pressed powder pellets. Data from x-ray fluorescence analysis is collected by computer and stored on a hard drive and on portable electronic media (disks and/or CD). Detection limits, methodology, interferences, and operating variables are reported in Hallett and Kyle (1993). Recently refined trace element values (determined by methods described by Hallett and Kyle, 1993) are found in Appendix I.

DATA REPORTING:

Analytical data are reported via an electronic spreadsheet (MS Excel) that is incorporated into the Molycorp database. The spreadsheets are stored via electronic media (internal and external). Hard copies will also be produced and archived by the laboratory until the completion of the project.

4. EQUIPMENT LIST

- Philips X-Ray Fluorescence Spectrometer – PW 2400, or equivalent
- Ball mill for grinding samples
- Crucibles to fuse samples (porcelain to dry flux and graphite to fuse samples)
- Spatula, stainless steel or teflon
- Drying oven
- Tongs - sizes to pick up crucibles
- Mortar and pestles - ceramic and agate
- High temperature muffle furnace
- Steel plates for cooling fused samples
- 120-grit and 400-grit sandpaper sheets
- Hydraulic press and die assembly to make XRF pellets
- Balances for sample weighing and plastic boats to weigh them in
- Kimwipes (or similar low-lint labwipes) and ethanol
- Borate Flux (lithium tetraborate and lithium metaborate, 1:1 mixture) i.e. *Ultrabind*
- Dessicator to store flux in
- 0.1 LiI solution
- Automicropipette
- Ziploc bags, indelible pens, weatherproof labels
- Latex gloves

- Lab notebook

5. RELATED STANDARD OPERATING PROCEDURES

The procedures set forth in this SOP are intended for use with the following SOPs (if required):

SOP 1	Data management (including verification and validation)
SOP 2	Sample management (including chain of custody)
SOP 22	Analytical Data Verification
SOP 24	Petrographic analysis
SOP 25	Stable Isotope analysis
SOP 26	Electron microprobe analysis
SOP 27	X-ray diffraction analysis
SOP 30	ICP-OES analysis
SOP 31	ICP-MS analysis

6.0 SAMPLE PREPARATION

Note: See Appendices II, III, and IV for more detailed XRF sample preparation procedure descriptions. See Appendix V and Appendix VI for XRF sample preparation techniques used at the NMSU XRF laboratory facility where this project's XRF samples were run after the NMBGMR XRF machine became disabled.

6.1 Grinding samples

Grind sample in a suitable vessel (swing mill, mortar and pestle, or ball mill) to obtain a fine powder (less than 10 microns is desirable and is best to overcome grain size effects and to ensure rapid dissolution in the borate flux during fusion).

6.2 Fusing powdered samples

Put powdered sample into melting crucible with appropriate amount of flux, heat in muffle furnace until sample melts and beads and forms a glass bead, allow bead and crucible to cool, remove bead from crucible, crush glass bead, make sure you keep track of which sample is in which crucible, some markers evaporate in the muffle furnace

6.3 Making sample pellets (also called briquettes or pancakes, see the detailed procedure included in Appendix II in this SOP)

Crush fused sample bead, place crushed fused sample into hydraulic press and die assembly and press the XRF pellet, make sure to label pellet with sample number (see detailed procedure below).

6.4 Analyze sample pellets

Analyze sample pellets for required major element oxides and trace elements in XRF machine using manufacturer's guidelines.

7.0 REFERENCES (Note: Other references are dispersed in text under appropriate procedures)

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Pella, P. A. (1978) “Effect of Gas Burner Conditions on Lithium Tetraborate Fusion Preparations for X-ray Fluorescence Analysis” Anal. Chem., v. 50, p. 1380-1381.

8. SAMPLE HANDLING

Once samples have been run, remaining powder and pellets should be stored in labeled and sealed plastic bags. Samples will be archived at least until the end of the project.

9. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance and quality control procedures are outlined above and below within the procedures. Additionally, for every 10 samples made, one duplicate sample is made. For every 20 samples made, one triplicate sample is made. Every day that samples are made, an internal laboratory standard, NMR, is made. The XRF machine is calibrated with certified standard samples.

APPENDIX I. XRF DETECTION LIMITS

WD-XRF Acquired Trace Element Lower Limit of Detection Values (LLD)

Element	LLD	Units)	Range	Units
Sr	1.0	ppm	0-662	ppm
Rb	1.0	ppm	0-320	ppm
Th	3.0	ppm	0-106	ppm
Pb	2.0	ppm	0-730	ppm
Ga	1.0	ppm	0-39	ppm
Zn	4.0	ppm	0-248	ppm
Cu	4.0	ppm	0-1110	ppm
Ni	2.0	ppm	0-2380	ppm
FeI	0.1	Wt %	0-13.41	Wt %
MnI	0.01	Wt %	0-0.26	Wt %
TiI	0.05	Wt %	0-2.71	Wt %
Ba	10.0	ppm	0-1882	ppm
V	6.0	ppm	0-526	ppm
Cr	3.0	ppm	0-3990	ppm
As	4.0	ppm	0-427	ppm

Bi	15.0	ppm	0-1380	ppm
Se	10.0	ppm	0-16.6	ppm
U	2.0	ppm	0-80	ppm
Y	2.0	ppm	0-143	ppm
Zr	2.0	ppm	0-309	ppm
Nb	2.0	ppm	0-268	ppm
Mo	2.0	ppm	0-310	ppm

LLD is lower limit of detection based on:

$3/\text{m SQR}(\text{Rb}/\text{Tb})$ using BCR-1 as the standard , where Rb is count rate on background and Tb is count rate on peak position.

APPENDIX II. BRIQUETTE (PELLET) PREPARATION

PROCEDURE - STANDARD OPERATING PROCEDURE FOR X-RAY FLUORESCENCE BRIQUETTE PREPARATION

HAZARDS

A) Acetone

Acetone is used as a cleaning agent. It is highly flammable and should always be kept away from open flames, other sources of heat (the hotplate), or strong oxidizers. Also, one should avoid contact with the skin or inhale the vapors. Read the Material Safety Data Sheet for a complete warning for acetone.

B) Pressing

It is possible to injury oneself or others if the die is not place in the press properly. If you are not sure the die is aligned properly, see the x-ray lab manager. Also, make sure the safety shield is down before applying pressure to the die.

C) Samples

Some samples may contain hazardous materials. Know what your samples are before you start.

D) Burns

The hotplate can be heated to a high enough temperature to produce burns on the skin. Turn on the hotplate only to the lowest level.

PRELIMINARY STEPS

1) Split samples down to a little more weight than required. It is convenient to weigh more than twice as much as required for one briquette. That way there is provision for duplicated, ruined briquettes and lost samples. If infinite thickness is required, the minimum weight per sample must be calculated relative to the diameter of sample exposed to the primary x-ray beam and the shortest wavelength

to be analyzed. If major element analyses of the samples have been performed, the amount of sample needed for a infinitely thick sample can be calculated.

2) Grind the sample. If the light elements are going to be analyzed, the final grain size should be less than 35 micron. Try to have the sample feel like talcum powder, with no grittiness. Rubbing some of the sample between two fingers can check this. Reproducibility is improved if the grinding procedure is uniform. That is uniform sample weights are ground identical lengths of time in the same machine.

BRIQUETTING

1. Introduction

The purpose of briquetting is to present powdered samples to the spectrometer in a reproducible way, consequently, it is important that the briquetting be done reproducibly. At NMBGMR we use a briquetting die patterned after the one designed by A. K. Baird (1961).

2. Briquetting Procedure

Place the hotplate on the opposite bench from the acetone. You can use either bench in Gold 143. Remove the die, mortar and pestle, boric acid, and weighing paper from the drawer.

- a. Clean the die with acetone to remove the oil
- b. Assemble the die
- c. Insert loading cylinder into the die.
- d. Place a 6" X 6" weighing paper inside the loading cylinder.
- e. Place another weighing paper on the balance.
- f. Tare the balance.
- g. Weigh out the sample.
- h. Transfer the sample to the agate mortar.
- i. Add 1 drop of binder for each gram of sample. If only 1 gram of sample is used, skip the binder.
- j. Mix the binder until the sample is uniformly wetted with the binder.
- k. Place the sample onto a weighing paper,
- l. Load the sample into the loading cylinder.
- m. Insert the briquetting piston slowly so the air turbulence does not scatter the backing material.
- n. Form the sample into a pancake using the loading piston.
- o. Carefully lift out the piston and inspect the sample pancake. The surface should be perfectly smooth with no cracks or gaps. If the pancake is imperfect, insert the piston and repeat the operation.
- p. When the pancake is perfect, remove the loading cylinder carefully without disrupting the pancake.
- q. Pour backing material into the die. If the backing material is lumpy, have finely sieved material handy to use first in order to avoid eroding the

- edge of the sample pancake. Different backing materials compress different amounts. Use enough to make a briquette about 6 mm thick.
- r. Place in hydraulic press and make sure the die is centered. Increase the pressure by 3 ton increments and allow entrained air to bleed out of the sample between increments. After 10 tons have been applied for about 1 minute, bleed the press to zero pressure over a period of about 1 minute.
 - s. Take the die out of the press. Invert the die. Remove the bottom of the die. Place the die under the hand press and cover the die with the wood sample cover. Press the wood sample cover until the sample is exposed.
 - t. Label the backside of the sample with the sample name, sample weight, type of backing material type of binder used, if any, date, and your initials. Put the completed pellet on the hotplate set at the lowest setting for 15 minutes. Store the samples in a dry place in one of the plastic sample holders.
 - u. Clean the die and start the next sample.
 - v. If you are done with all of your samples, clean the die and coat the steel pieces with oil so the steel will not rust. Clean any other mess.

IMPORTANT NOTES

- 1) Protection of the sample is particularly important if light elements are to be determined. The freshly pressed sample surface should not be touched.
- 2) Exfoliation of the sample after briquetting can occur with thick silica samples. Consequently, sample thickness should be minimized. Wear of the loading cylinder can be a contributing factor to exfoliation. If exfoliation occurs frequently, check the loading cylinder for wear. The cylinder should have a very sharp edge, but without a burr. This will give maximum edge support to the sample pancake. If exfoliation of the sample continues, interrupt pressing at 5 tons. Remove the bottom of the die and place a drop of deionized water on the sample surface. After the water has soaked in, reassemble the die and continue pressing as before. The water will help to drive out entrained air and improve the intragranular adhesion of the grains.
- 3) Some very finely powdered materials, such as reagent Fe_2O_3 , have a tendency to stick to the anvil of the die. This can be prevented by carefully cleaning the anvil and dusting it with fine spectrographic carbon.
- 4) It is important for light element work that the surface of the anvil be optically smooth, so carefully examine the anvil from time to time to verify that it is in good condition. The WC inserts in the small die set may move from side to side if they have not been inserted properly.

Problems

- 1) If samples are not ground fine enough, they will disaggregate after pressing into a pellet.
- 2) High silica samples, especially samples high in quartz may require more binder or lower pressure to make a satisfactory pellet.
- 3) High clay samples may crack across the analytical surface. Dry these samples and try less binder. It may not be possible to press some clays (expandable clays).

References

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APPENDIX III. ANALYTICAL PROCEDURES

A. STANDARD OPERATION PROCEDURES FOR DETERMINING LOSS ON IGNITION

Hazards

A) Burns

Since the high temperature furnace will operate up to 1000°C there is the possibility of severely burning oneself.

B) Splinters

The crucibles will be at high temperatures and if they are taken out too soon, it is possible that one of them could shatter and one or more of the particles could be imbedded in the skin or the eyes. Always allow the crucibles to cool before removing them from the furnace.

C) Samples

Samples may contain hazardous materials. Know what your samples are before determining loss on ignition. Also know if your samples contain any flammable or combustible material.

Procedures

- 1) Make sure the sample is ground to less than 35 microns
- 2) Wash the number of ceramic crucibles you will need in water and rinse with deionized water. Dry on the dish rack for 10 minutes.
- 3) Place the crucible in the low temperature furnace (the gray oven) for at least 2 hours. This furnace should always be operated at 110°C.
- 4) Remove the crucibles from the furnace and place them in a desiccator. At first leave the cover ajar for about 1 minute to let some of the warm air to escape. Then cover completely and allow the crucibles to cool to room temperature (about 1/2 hour).
- 5) Start of the procedure
 - a. Record the sample number.
 - b. Record the crucible number.
 - c. Tare the balance.
 - d. Weigh the crucible exactly and record the weight.
 - e. Place 2-4 grams of sample in the crucible.
 - f. Reweigh exactly and record the weight.
 - g. Repeat a-f for the next sample.

h. When all samples are done, place them in the low temperature furnace.

6) Leave the samples in the low temperature furnace for at least two hours. After 2 hours, take the crucibles out of the furnace and place them in a desiccator. Leave the desiccator cover slightly ajar for about 1 minute, then cover completely. Let the crucibles cool to room temperature (about 1/2 hour). Weigh each crucible exactly and record the weights.

7) Place the crucibles in the high temperature furnace. Set the parameters to run the furnace:

Initial rate	15-20°C/minute
Transitional temperature	100°C
Hold time	1 minute
Final rate	15-20° /minute
Final temperature	1000° C
Final Hold	120 minutes

Let the furnace cool to under 100°C before removing the crucible. This will take 4 to 5 hours from the time the furnace is started. Place the crucibles in a desiccator and leave covered ajar for 1 minute. Fully cover the desiccator and let the crucibles cool to room temperature (about 1 hour). Weigh each crucible exactly and record the weight.

8) Calculate the weight loss and weight percent loss after 120°C (this is H₂O-). Calculate the weight loss and weight percent loss after 1000°C (this is H₂O+). The two combined is the total LOI. The best way to record the weights and weight losses is in a table in a notebook.

9) Wash the crucibles and clean up any other mess you made.

Problems

- 1) If you are not careful, contents of the crucible can easily be spilled.
- 2) Weighing may be erratic if you do not allow the balance to warm up.
- 3) Crucibles must be at room temperature to weight otherwise their weight will change as they cool.

References

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APPENDIX IV. STANDARD OPERATING PROCEDURE FOR THE OPERATION OF THE X-RAY FLUORESCENCE SPECTROMETER

HAZARDS

A) Radiation

The x-ray fluorescence spectrometer (XRF) has a closed beam configuration. One should not be exposed to the primary or secondary x-ray beams unless one purposely bypasses all of the safety devices. Only Chris McKee, Phil Kyle, or the service engineer may open the spectrometer.

B) Mechanical

It is possible to pinch or jam ones finger when loading the samples into the sample chamber. Always remove your hand from the sample holder before telling the computer to load a sample or measure a changer buffer.

C) Electrical

There are numerous wires inside the XRF. Some of these wires carry enough current to kill a person. Do not open the XRF up and poke around inside. Only Chris McKee, Phil Kyle, or the service engineer is allowed to make adjustments inside the XRF.

INTRODUCTION

The X-ray Facility has a Philips PW2400 wavelength dispersive, sequential x-ray fluorescence spectrometer that was manufactured in Holland and sold in the United States by Philips NA. It is a inverse geometry spectrometer, that is, the analytical surface of the samples faces downwards. The spectrometer uses a Rh end window tube positioned at an angle to the sample analytical surface.

Calibration of the instrument is usually done annually using a large suite of calibrated international rock standards. The calibration is checked using a drift standard and several in-house rock standards.

Samples are presented to the primary x-ray beam by means of loading a sample in a sample cup and placing on the plunger or using the sample carousel with a changer buffer. The secondary x-rays exit the sample chamber and enter the crystal chamber through a collimator. There are six crystals in the crystal chamber used to diffract the secondary x-ray beam. Also in the crystal chamber, is a flow proportional counter with a collimator in front and a sealed counter behind and a scintillation counter with a collimator. These counters detect the x-rays and the raw data is transferred to the computer for calculation of concentrations.

OPERATION

1) The spectrometer should never be turned off except in emergencies and for servicing. Only Chris McKee, Phil Kyle or a service engineer can service the XRF. All analyses are controlled by the dedicated PC.

2) Load samples into the sample holders. First, place the samples in alphanumeric order. Assign lab number with one of the Excel spreadsheets. MAJx00 is for major elements. Data entered include: sample name, name of project person, LOI, sample weight, and file number. Use TRACx00 for trace elements and input sample name, name of project person, and file name. After running the sample you will have to enter the load number for each sample.

- a) For fused disks, place a positioning ring of the proper size in the sample holder. Place the fused disk into the positioning ring so the analytical side faces down. (This is the side opposite of the labeled side, i. e. the label should face up.)
- b) For pressed powder samples, place a sheet of Ultralene on top of a liquid cup. Secure the film with a retaining ring for liquid cups. Place a positioning ring over the liquid cup. Turn the cup over and place the sample in the sample cup so the analytical surface faces down. Place the entire assemble into a sample holder.

3) Place the sample holders into the sample trays in proper order. Position #1 of each sample tray is the left most position when viewed from the front (the side with the tray number card is facing you).

4) Place the sample trays in the sample changer so the tray number cards face the spectrometer.

5) Assemble a changer buffer on the computer (type ACB in X40). It is best to have a copy of the appropriate Excel spreadsheet in order to enter all of the proper data. Make sure the samples are loaded in the proper order.

6) Measure the changer buffer (type MCB in X40).

7) After all of the trays are analyzed, remove the trays from the sample changer. Remove the samples from the sample holders. For trace element samples, disassemble the liquid cups and throw away the Ultralene film. If any of the samples have high trace element contents, throw the entire assemble away (retain the positioning ring). If more samples are to be analyzed start at 2 above.

8) Either print out the data or use the CONVERT program to convert the data to an Excel spreadsheet.

9) Clean up any mess you made.

Problems

- 1) Fused disks may crack in the machine. Carefully remove the material from the sample cup. Check to make sure all of the disk is accounted for otherwise the XRF will have to be shut down and opened up to remove the material in the machine. Remake the sample and rerun.
- 2) Some pressed powder pellets may “pop out” of the liquid sample cups. These pellets should be dried on the hot plate for 30 minutes before rerunning. Also make sure that a brass weight is on the back of the pellet.
- 3) The sample changer may jam. The computer will give an error message if this happens. Carefully remove the sample tray. Put the tray back making sure to maintain the proper order for the trays (they are number from 1-5). Restart the analysis.
- 4) If the power goes off, restart the computer and the XRF. Allow the XRF to warm up (the length of time for warm up will depend on the length of time the machine is off). Start X40 and analyze the remaining samples. Be sure to run several NMR-1 samples to make sure the XRF is stable.

APPENDIX V. PREPARATION OF GLASS BEADS

PREPARATION OF GLASS BEADS

1. Clean graphite crucibles by scraping the sides and bottoms with a spatula. Tap the crucible, upside down, on the lab bench to remove loose graphite. Foliate the inner surface of the crucible by squashing and turning a paper towel inside the crucible. Always turn the towel the same direction!
2. Tare the scale. Weigh a plastic boat. Tare again.
3. Add the correct amount of CALINED rock powder to the boat. Be sure to break up any lumps before weighing. Write down the weight. Tare.

9:1 dilution - 1.5000 ± 0.0001 g rock powder

4. Add the correct amount of flux (50/50 mix of lithium tetraborate and lithium metaborate) to the boat.

9:1 dilution - 13.5000 ± 0.0001 g flux

(The flux must be dry and stored in the desiccator. If the beaker of dry flux is empty, dry new flux in clean porcelain crucibles in the muffle furnace at 500°C for 1 to $1\frac{1}{2}$ hours, or until flux is opaque).



5. Remove the boat from the scale. Gently mix the rock powder and flux together. Gently pour the mixture into a prepared graphite crucible, being careful not to splash any of the sides, top, or out of the crucible. Be careful - the mixture is now in an unlabeled crucible. Mark front (F) middle (M) or back (B) on the worksheet and always keep samples in the same order.
6. Add 10 microliters of 0.1 LiI solution to the top of the powder using the automatic pipette.





7. Place samples in the muffle furnace to fuse at 1100°C. Once the muffle furnace has heated back up to 1100°C, let sample melt for 15 minutes. At 15 minutes turn the shaker table on for ~~5~~ minutes. After ~~5~~ minutes turn shaker table off and leave sample in furnace for approximately 13 ¹⁰ more minutes. (Sample should remain in the muffle furnace at 1100°C for a total of 30 minutes.)
8. Remove crucibles from furnace and place on steel plates. When cool, invert crucibles and tap gently to remove glass beads. Label sample with sticker and place in labeled ziplock bag. (*Samples may be removed from crucibles after approximately 10 minutes, at this point the bead will cool faster outside the crucible, and the crucible could then be prepped for a new sample*)
9. Polishing: remove graphite from and flatten the bottom surface of the bead using a large sheet of 120 grit paper adhered to the lab bench. Do not use the same paper for more than one sample to avoid cross-contamination!! Wipe with Kimwipes!! Do not use any liquid - this could remove the alkalis!! When a 2cm diameter area of the surface is flat and clean of graphite, polish the bead on a small 400 grit paper to produce an analytical surface.

APPENDIX VI. Sample Preparation of Pressed Pellets for XRF Analysis

XRF PRESSED PELLETS - SAMPLE PREPARATION

Analytical Geochemistry Research Laboratory

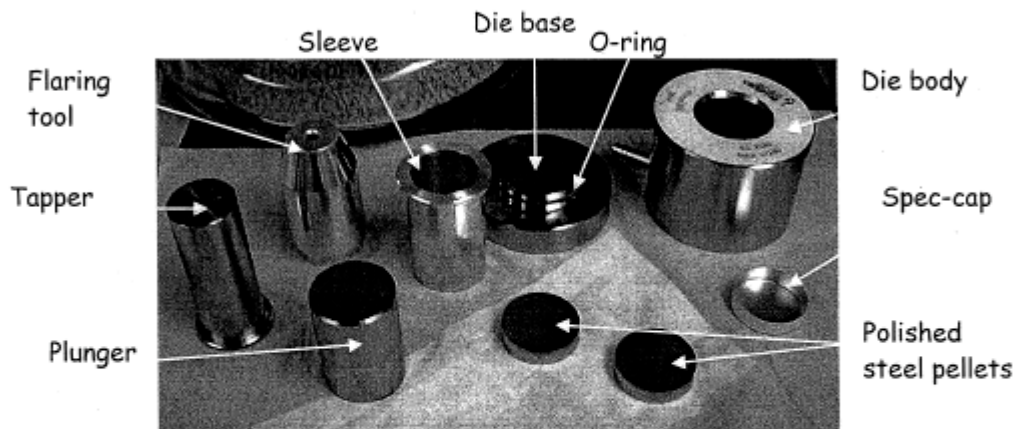
Department of Geological Sciences

New Mexico State University

July 2003

Equipment used to make XRF pressed pellets

*Die base (with O-ring), *Die body, *Two polished steel pellets, *Plunger,
*Flaring tool *Sleeve, *Tapper, Spec-cap (*equipment stored in desicator)



Procedure:

1. Clean all surfaces of all equipment, using Kimwipes and ethanol. Your goal is to remove all particulate matter, including lint.
2. Check to see that the O-ring is seated on the die base. Snuggle the die body down onto the die base, making sure that the fit is tight.
3. Insert one steel pellet, polished side up, into the hold in the die body. You need to place it in the hole absolutely vertically; it will slide down to the bottom of the hole (it might go pretty slowly). Do NOT TOUCH the polished surface!
4. Flare the spec-cap with the flaring tool by placing the cap on the wide end of the flaring tool and pressing it against the lab bench. This insures a tight fit between the cap and the die body and prevents powder from escaping. Insert flared spec-cap into the die body on top of the lower steel



pellet.

5. Weigh out 11.0000 ± 0.0001 g rock powder. Add 1.1000 ± 0.0001 g Ultrabind. Mix rock and Ultrabind thoroughly!
6. Pour the mixture carefully into the Spec-cap in the die body. Don't splash or slop; just pour gently into a pile in the middle.
7. Tap the sides of the die body gently to level the top of the powder. This is an important step, because it helps the plunger start out in a vertical position. You can also try using the tapper to level the powder.
8. Insert the second steel pellet, polished side down (the polished sides face the powder). You might have to push it down gently, with a gloved finger or with a Kimwipe between your finger and the pellet.
9. Insert the plunger, squared end first! Be absolutely sure that the squared end is down and the beveled end is up!
10. Place the die on the press platen. Point the vent tube away from you. Be sure that the die is centered! There is a circle on the platen, and there are engraved circles on the top. It is critical that the die be centered on the platen, so take your time!



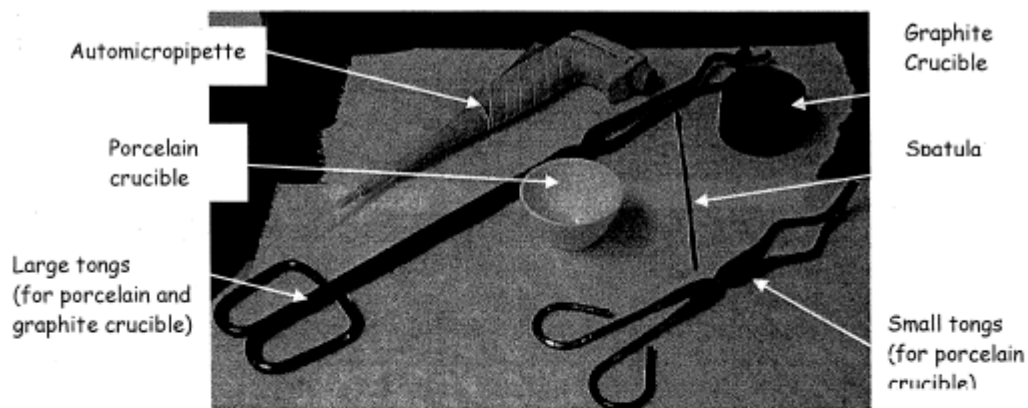
11. Tighten the knob on the front of the press (righty tighty, lefty loosy); it takes only a slight turn to tighten. This closes the air valve and allows the pressure to build up.
12. Pump the handle until the plunger is just topping the surface of the press. Check the centering. Then pump away until the pellet is under about 20 tons.
13. Leave it at 20 tons for about a minute.
14. Turn the knob on the front of the press a tiny bit counter-clockwise to release the pressure. You want the pressure to bleed slowly away—a fraction of a turn will do the job. Let the platen slide slowly down.

15. Removing the sample from the die is the most exciting part of the task. Gently pull the die body from the die base. With luck, the two steel pellets and your sample will slip easily from the die body. Be sure to catch them, and don't scratch the steel pellets!! If you are not so lucky, take the plunger out and replace it with the sleeve.

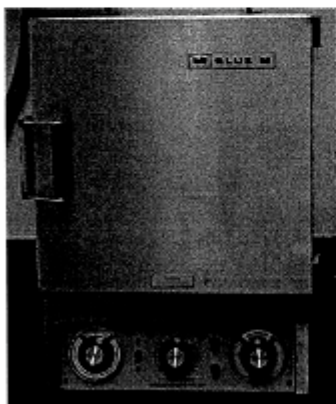


- Then put the die body and sleeve (not the die base) back into the press, and pump it up so that the sleeve pushes the steel pellets and sample out. Don't pump so much that the pressure increases! Carefully remove the die body from the platen (remember that there is no base, so put your hand under it!!). You may have to repeat this process three times to get both steel pellets and your sample out.
16. Write the sample number on the bottom of the spec-cap and a small zip-lock bag; store the sample in the zip-lock.

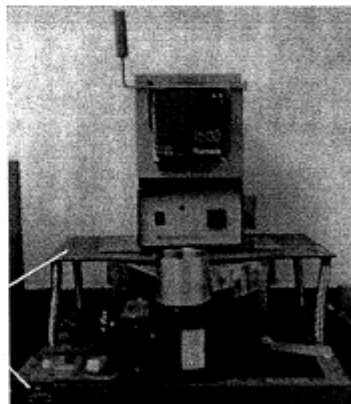
Equipment and Tools Used to Calcine and Make XRF Beads



Blue Oven



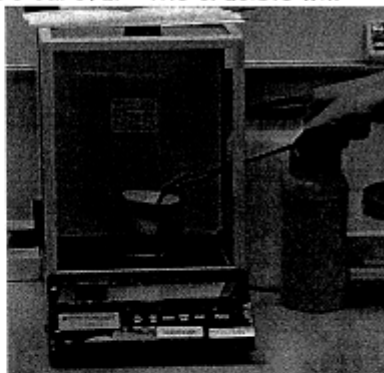
Muffle Furnace

Shaker
Table

Loss On Ignition (LOI)/Calcine

We calcine (heat to drive off all volatile components, mainly H_2O and CO_2) all our rock powders before making them into fused beads for major element analysis. This makes it convenient to measure the %LOI and prepare powders for fusion in a single step.

1. Clean 50 ml porcelain crucibles. You are trying to get all particulate matter off the crucible surfaces. Use XX-H₂O, followed by acetone, methanol, or ethanol. Use Kimwipes to wipe the surface dry.
2. Heat the crucibles in the blue oven at 110°C (set left dial on "medium") for 20 minutes. This drives off any volatiles adsorbed on the crucibles surface.
3. Weigh the crucible and record the results. Be careful - the crucible will start to adsorb water from the air as it cools. Get the best weight you can before it starts gaining. Don't worry about this inaccuracy - it's just part of the error of the analysis.



4. The amount of rock powder needed varies with the amount of volatiles and the flux:rock ratio you are using. Follow the chart below to estimate the minimum amount of powder needed. You can calcine up to 5 g of powder at a time. It is not important that there is a specific amount of powder, just that we know the weight accurately. Be careful - the powder is now in an unlabeled crucible. Mark front (F) or back (B) on the worksheet and always keep the samples in the right order.

9:1 dilution:

<u>LOI%</u>	<u>Powder needed (g)</u>
1.0	1.6
2.0	1.6
5.0	1.7
10.0	1.8
20.0	2.0

5. Heat samples in the muffle furnace (stationary - no rotation) at 1000°C for mafic and intermediate samples, or 900°C for silicic samples, for 20 minutes.
6. Remove from muffle furnace and place in the blue oven at 110° for at least 5 minutes. Volatiles cannot reabsorb onto particle surfaces at this temperature.

7. Remove crucible from blue oven and immediately weigh and record results. Store the powder in a labeled mini-ziplock bag.
8. Calculations: subtract the crucible weight from the crucible & rock weight to get the initial powder weight. Subtract the weight after ignition from the crucible & rock weight to get the weight loss during ignition. To get LOI%, divide weight loss by initial weight and multiply by 100.

$(\text{crucible \& rock weight}) - (\text{crucible weight}) = \text{initial powder weight}$

$(\text{crucible \& rock weight}) - (\text{weight after ignition}) = \text{weight loss during ignition}$

$\text{LOI} = 100 * (\text{weight loss during ignition}) / (\text{initial powder weight})$