Department of the Interior

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ARL-SEMQ electron microprobe operating manual

by

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INTRODUCTION

This manual is designed to aid in learning to operate the Applied Research Laboratories (ARL) SEMQ electron microprobe and serves as a reference for procedures once the operator is familiar with the simple mechanisms of operation. Not all possible questions, problems or variable analytical procedures are covered in this manual. Some things you will learn through attentive operating experience, discussions with others, or through the literature. Although the steps needed to obtain microprobe analyses may sound simple on paper, the manual will not teach you all you need to know to obtain high quality, meaningful analytical data with this powerful and complex tool. However, it will get you started and give you something to refer to as you try to get through your first "shift". This manual provides information that just is not available elsewhere (on paper) and consolidates a lot of the software procedures into a few steps which will get you to the point of running the programs. There is plenty of space in the margins to write your own comments or to clarify my descriptions. The analytical programs are highly interactive and for the most part self-explanatory. Lengthy descriptions of ARL's original unmodified programs are available in the lab (ARL, 1979). Be aware that I have made numerous modifications to ARL's software so that their original descriptions are no longer completely accurate. There is also an Open-File report (McGee, 1983) describing the $ANBA program in greater detail.

Many errors or problems arise due to operator error or inexperience. I have tried to anticipate some of these and include them in the "Troubleshooting" section of the manual. Many other non-routine problems may arise due to instrument wear or malfunction, unusual operator action, or unusual analytical problems. Most of these problems are correctable in a relatively short period of time, if you cooperate. Pay attention to your own actions, clearly describe your actions to me, and describe as accurately as possible what the symptoms of the problem are or how the instrument responded to your actions (this is especially important if I am trying to assist you via telephone and I cannot "see" the entire situation). With all of this information I should be able to help you recover from the problem and you should be able to continue operating. But there are times... (Good luck)!

Although this manual was written specifically with the U.S.G.S. - Reston microprobe instrumentation and users in mind, the general sequence of procedures is applicable to any microprobe and the specific instructions on button-pushing/knob-turning should apply to any SEMQ microprobe. Also, the brief summaries of concepts on standards selection and general analytical procedures described in some sections of the manual are relevant to all geologic applications. The manual should provide guidance as to what steps and instructions are needed to operate the electron microprobe and will perhaps serve as a guide for the preparation of manuals for other microprobe laboratories.

START-UP PROCEDURES

General Precautions:

1)Vacuum control power switch -ALWAYS ON
2)Vacuum key set to "AUTO" mode (except for filament change)
3)USE NO FORCE - there are mechanical stops and limits to how far you can turn a knob, spectrometer, sample stage dial, etc. Please be careful not to exceed these.

A)Instrument Set-up:

1)Check Vacuum Panel: G1, G2, G3 pressure 0-10; G4 $<10^{-5}$; all red indicator lights on; sample stage down - elevator down and Z axis to ~ 324 ("Stage Down" light on); vacuum key turned to AUTO;

2)Check that P-10 gas is flowing at 0.03 rate (meter is on vacuum panel) - if bubble is not visible, tap window (bubble occasionally gets stuck at top of meter during initial gas surge) if no flow, check that gas cylinder is not empty (regulator gauge on tank should read 10 psi) and that valve knobs on the tank are opened (large top and small black knobs turned clockwise). Do this before you adjust the flow increase knob on the meter.

On the Scaler Console:

3)Set the Scaler STOP SELECT to "R" and DISPLAY TIME to "Hold" on the two scaler modules. This will enable the computer to read and control the scalers during program operation;

4)Detector High Voltage (below Digital Drives) on and set to 2400 volts (2000 coarse and 400 fine).

On the SEM Console:

5)Beam Blank button out (not lit) (Run the "FREE" program if the button is out and lit);

6)Raster size (below SEM screen) set to POINT;

7)Gun High Voltage power on and 15 kV button pushed in (turn up sequentially in increments of 5 kV with buttons); note- if you need to operate at other than 15 kV, or if you need to return to 15 kV:

a) turn Filament Saturation knob to 0 0 0.
b) select desired voltage using Coarse kV buttons and Fine kV knob.
c) turn Filament knob back up till emission current reads 50-100 μAmps and proceed to below.

B) Saturate Filament -

CAUTION - greatly exceeding plateau voltage risks burning out the filament.

1) Turn Signal Selector knob to "BC" (beam current); turn Picoammeter Range to 0.1×10^{-6} amps; turn Filament Voltage knob ("Saturate") up till plateau is reached on the Picoammeter. In doing so, you will first see a "false" peak (although this false peak disappears with filament age). See Figure 5, p. 36. Filament current should not exceed 2 Amps. Saturation level should be at approximately the same, or slightly lower, dial setting as previous day (see logbook) and is usually no greater than 650; If the beam current goes off scale on the 0.1×10^{-6} range, change range to 0.3×10^{-6} or lower the current with the C2 condenser adjust knob.

2) Center the filament with the 4 large knurled centering knobs on the electron gun (opposing knobs turned simultaneously in the same direction as seen from the front of the probe). Filament is centered when the current level is maximized by the adjustment of these knobs.

3) Set the Emission Current to 150 microamps (for 15kV) with the BIAS knob.

4) Re-check the saturation setting (while watching the Picoammeter) and then turn saturation dial 5 units above the position of the second peak - this assures that you are on the plateau.

5) Adjust beam current to 0.1×10^{-6} (or desired current) with C2 (condenser) Coarse and Fine adjust knobs. Turn Signal Selector knob to "SC" (with the knob set this way, the Picoammeter will display sample current and the digitized beam current will be displayed on the beam current scaler). You may check the beam current setting by toggling the "Reset" and then "Start" toggles on the left scaler readout. The seventh scaler is calibrated so that a beam current of 0.1 microamps generates 10000 counts per second (cps). The scaler will probably count for 10 seconds (check the timer) and so the beam current readout should be approximately 100000. Higher currents may be desired for improved count rates/detectability limits. Lower current may be desired to reduce specimen damage/element mobility problems.

6) If desired, turn Range knob to scale needed to read sample current, usually 0.03×10^{-6}. Always turn Range knob back to 0.1×10^{-6} before selecting "BC" signal. When count data is being acquired during analysis or standardization, the selector switch must be set to "SC". See additional Signal Selector information on page 30.

C)Sample Loading-

Samples should be polished, cleaned, and carbon coated (see p. 61)) prior to loading. (Prior to loading samples, you may wish to check approximate X,Y locations of areas of interest by overlaying samples on the sample holder map on the next page.) The following procedures should be followed when loading, unloading, or changing samples:

1)Check that vacuum key on AUTO; put Stage Digital Drive in "MAN" mode (manual);

2)Elevator down (push button in until it lights) and Z axis down (~ 324). The "Sample Stage Down" light (on the vacuum panel) will be on;

3)Push SC VENT button (on SEM console) - wait for stage to drop;

4)Put gloves on - pull stage out, loosen clamps, REMOVE sample holder, load samples (make sure standard mounts are in proper orientation); replace sample holder (hold sample holder against mounting guides in upper right corner when tightening clamps); to remove or insert transmitted light assembly, see p. 31; check that O-ring is free of dust, lint, dandruff, etc. (rub lightly with clean glove/finger; depress locking catches on runner and push stage back in (gently);

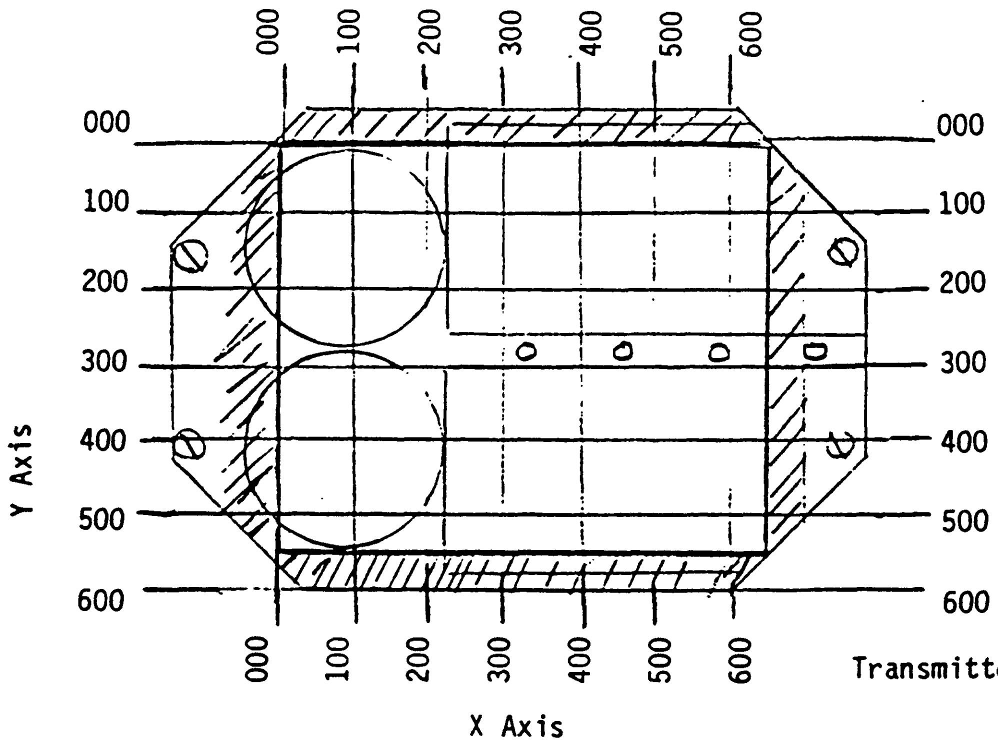
5)Push SC PUMP button - sample chamber will pump down and gate valve will open automatically after about 5 minutes. You should monitor the pump down on the G2 gauge- the pressure should reach 100 fairly rapidly and pumping will continue till gauge reaches 40 microns. After hissing noise stops and the sample chamber valve opens (V1 light on), proceed;

6)Blank the beam (push BEAM BLANK button in);

7)Raise sample - Elevator up (light on) and Z axis up to ~ 030 (~ 017 if using the brass block). Turn illumination on and adjust Z-axis focus to view sample.

8)Check the beam spot's location, shape, and size (use a fluorescing sample such as periclase). Adjust Z axis to get spot focussed; adjust OBJ lens current to change spot size; adjust two centering knobs (facing you at eye-level) to center crosshairs over the spot (do not touch the aperture adjust knobs located above the centering knobs unless you know what you are doing).

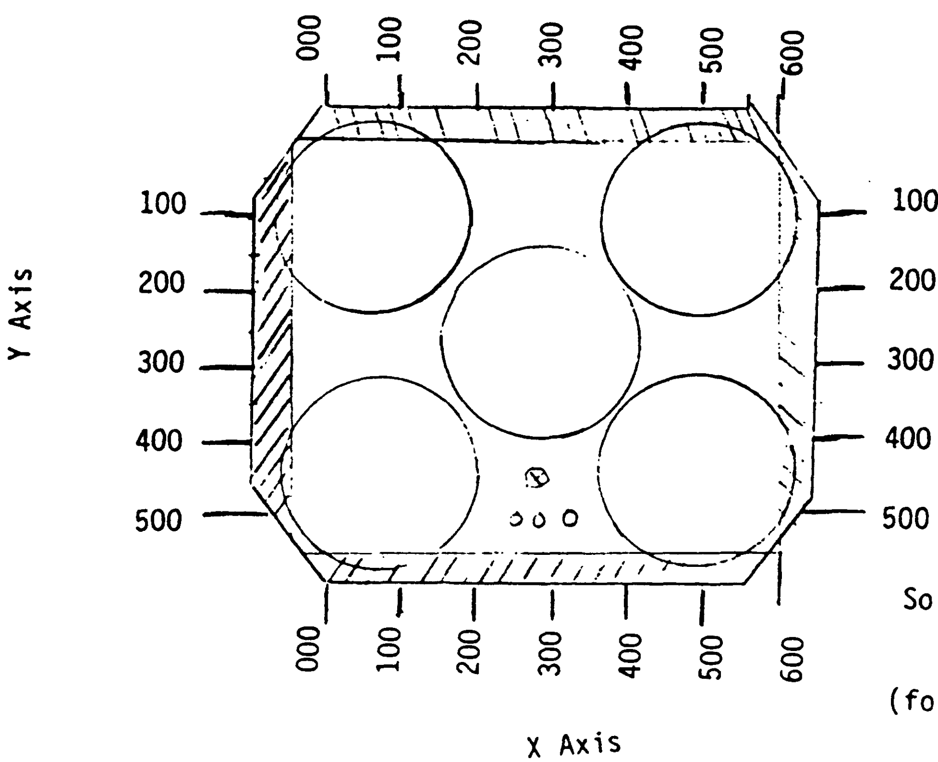
Figure 1 X - Y Maps for Sample Holders



Transmitted Light Assembly

Sample Holder

Stage drive limits



Solid Brass Sample Block

(for 1" circular plugs)

9) Position crosshairs on brass or blank the beam (see p. 31) so as not to accidentally burn the epoxy. Be sure to unblank the beam before starting the analytical programs.

D) Check crystal and alignment settings-

Make certain that the crystals you need on the scanning spectrometers are flipped into the analysis position and that the corresponding attenuator, focal circle, and crystal adjust settings are selected for those crystals. If necessary, run the QLIST program to see what crystals you need. Check the logbook or the spectrometers to see what crystals are presently in place. If the correct crystals are in place, proceed to "Calibrate Digital Displays", p. 9.

E) Crystal Flipping-

The scanning spectrometers are equipped with two crystals each. The crystals are mounted 180° apart on a rotating shaft and either crystal is selected by flipping the crystal around the shaft. Only one crystal per spectrometer can be used for any given analytical set-up (crystals cannot be flipped during an analysis). In our analytical configuration combining fixed elements with scanning spectrometers, the three scanning spectrometers usually use the ADP, TAP, and LIF crystals (on spectrometers 1, 2, and 3, respectively). In some cases it may be desirable to have an additional LIF crystal on a spectrometer, in which case the crystal on spectrometer #1 would be flipped from an ADP to a LIF crystal.

The available crystals on the scanning spectrometers (see configuration on next page) are:

Spec. #1	-	ADP or LIF	
#2	-	TAP or RAP	(or PBSD)
#3	-	LIF or PET	(or ADP)

(The alternate choices, in parentheses, for spectrometers 2 and 3 depend on whether the auxiliary spectrometers are installed. Check this on the crystal flipper mechanism on each spectrometer.)

Before flipping crystals, first check to see if the desired crystals are already in position for analysis by noting the position of the index mark on the crystal flipping mechanism (on the upper-right corner of the spectrometer housing). If a crystal needs to be flipped, proceed as follows.

1) Drive scanner containing the crystal to be flipped to its upper limit:

1.1- Select display for scanner to be driven;

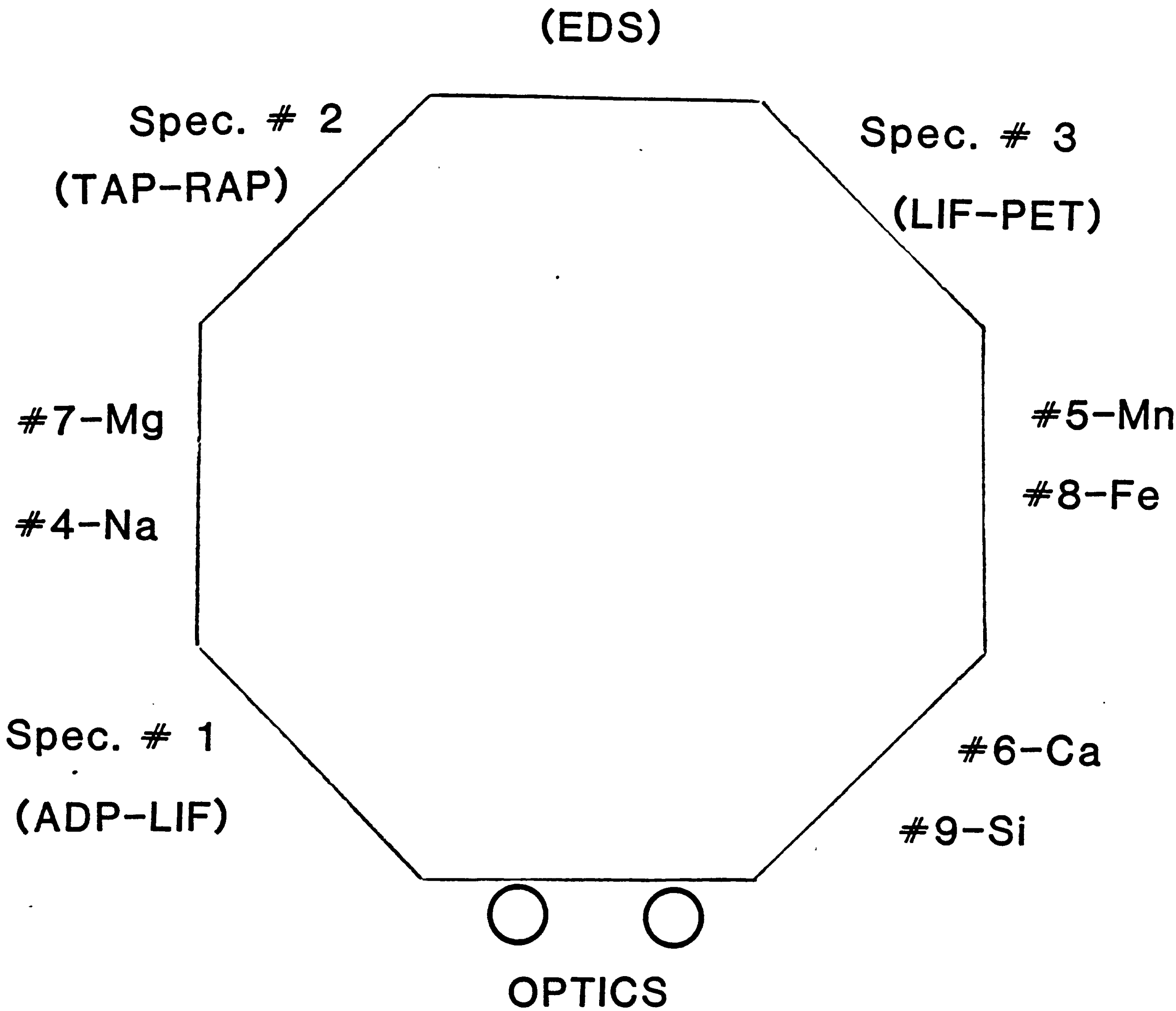


Figure 2 - SPECTROMETER CONFIGURATION

1.2- Put scanner DIGITAL DRIVE in "Local" mode and move "+/-" toggle to "+" for desired scanner. Be sure the toggles for the other scanners are in the neutral position so that you do not inadvertently drive them to their limits.

1.3- Turn RATE knob on (clockwise) and drive scanner to upper limit (~ 37750)- the scanner will stop automatically. The RATE knob controls the speed of drive - you can drive at full speed.

2) When the spectrometer has stopped at the upper limit, turn the RATE knob counter-clockwise until it clicks off. Put the +/- toggle to neutral or "-". Put mode in Manual.

3) Flip the crystal on the desired spectrometer:

3.1- Index on the crystal flipper knob is under crystal currently in the analysis position;

3.2- Turn flipper knob so that the index moves towards the bottom of the dial and then towards the desired crystal. You will feel the crystal reach a detent as the knob is rotated about halfway ("6 o'clock"). Continue to turn the knob until you feel it stop against a sping-loaded resistance. Do not force the knob- the index will spring back to the proper position;

3.3- When the index is in the proper position, manually turn the the spectrometer motor to lower odometer readings (the DIGITAL DRIVE must be in Manual mode to do this). You will hear a click as the crystal flipper gate locks the index and knob in position. Continue to turn to lower odometer numbers until a zero is lined up with the marker on the right (~ 37700).

4) Re-calibrate the DIGITAL DISPLAY setting for the spectrometer:

4.1- Enter value as read from the odometer and set the manually selectable numbers for that spectrometer;

4.2- Simultaneously depress the two black "PRE-SET" buttons on the DISPLAY and the value you selected will be entered into the LED's.

5) Drive spectrometer to ~ 35000 (to get away from the upper limit):

5.1- Put scanner DIGITAL DRIVE in "Local" mode and put +/- toggle to "-";

5.2- Turn RATE knob on and drive spectrometer down. Turn RATE knob off when finished.

6) Set Attenuator, Focal Circle, and Crystal Adjust to values for the crystal you just flipped into position:

6.1- Find settings for crystal on configuration sheet posted near TV monitor;

6.2- Turn Attenuator for desired spectrometer number to the setting for the new crystal;

6.3- Set Focal Circle and Crystal Adjust knobs (located on spectrometer) to the settings for the new crystal (If you are turning to a lower setting, turn knob 50 units or 1/2 turn below and then back "up" to the desired setting.).

7) Put scanner DIGITAL DRIVE in "Remote" mode.

8) It is a good idea to re-standardize those elements measured on a crystal that has just been flipped, in case there is a slight difference in the alignment or wavelength settings between the present and previous analytical sessions.

F) <u>Calibrate Digital Displays</u> (for scanners and sample stage)-

There are two Digital Displays - one for the stage X, Y, Z axes and the other for each of the scanning spectrometers. The particular axis or spectrometer readout is selected by pushing in the corresponding button below the LED display. The actual positions of the scanners or stage axes are read on their odometers and it is important during computer controlled operations that the Digital Display values agree with the odometers, since the programs read the Display value in order to record where a paticular axis or scanner is located.

To calibrate the displays, place Digital Drives in "MAN". Read 5 digits on odometers, including leading 0's. Switch dial numbers on Digital Displays to agree with odometers for all scanning spectrometers and X, Y, and Z stage positions. Enter correct number by simultaneously pushing calibrate "PRE-SET" buttons with the correct number for each drive selected on the dials - repeat for each scanner and stage axis. Return Digital Drives to "Remote".

If there are ever any power flickers during a shift, check that all the Digital Display values are still correct. Also, if the stage or scanners have encountered mechanical limits (Drive Error Al, see p. 39), you will need to manually back off of the limits and then reset the Digital Displays. Finally if any of the individual LED's on the Displays have ill-formed numbers, or if the "O.R." LED is lit-up, recalibrate that axis or scanner Display.

COMPUTER OPERATION

1)Insert program disk in left-hand drive ("0") of RX02, data disk in drive "1"; close drive doors;

2)On the computer panel above the disk drives are 3 switches - place the HALT switch up; place the AUX ON/OFF power switch up;

5)Program disk boots up on DECWRITER terminal. Respond to the questions with the following underlined responses:

START?Y

TIME:hr,min (numeric entries)

DATE:day,mon,yr (numeric entries)

(The video terminal and Decwriter power switches are normally left on so that they power-up automatically when the computer power is turned on. If the terminals are not responding, check that their power switches are on.)

6)If the program disk has booted up properly the "READY" message will be printed.

At this stage you should run any preliminary programs or utilities needed to prepare for analytical program operation. For example, if you are going to use the on-line plotting routines you should turn on the plotter and run the "IPLOT" program now (see p. 27).

It is also advisable to "Index" your disks at this time to be sure you have the necessary files, programs, and usable "Free" disk space to operate the analytical programs. Running the "PACK" program utility is a good precautionary measure to prevent disk writing errors from occurring during later program operation. Refer to page 49.

- To switch from one terminal to the other depress "CTRL A" on the terminal you want to use. If you interrupt a program when doing this, the line number where the program was interrupted will be printed. To resume program execution, type "GOTO xxx" (xxx = line number that was printed).

- To exit a program, depress "CTRL P" (unless instructed otherwise). See next page for other control (CTRL) characters and their function.

7)Start execution of the analytical programs (usually run "$PSET" program first - see flow chart on p. 13):

CONTROL CHARACTER SET

CONTROL KEY	FUNCTION	SYMBOLS PRINTED
<CTRL A>	Select input terminal as system device	<CR> READY
<CTRL P>	General system ABORT, return to BASIC. Will print the line number where the executing program was terminated.	READY
<CTRL Q>	Resumes printing characters on the terminal from the point printing was previously stopped because of a <CTRL S>.	None
<CTRL R>	Unlock alternate terminal keyboard previously set by <CTRL T>. Functions only on the main system terminal.	None
<CTRL S>	Suspends output to the terminal until you type a <CTRL Q>.	None
<CTRL T>	Lock out alternate terminal keyboard.	None
<CTRL U>	Deletes the current input line.	^U <CR>
<CTRL X>	Vectored program ABORT - (see Section 4.3.6.)	None

ARL

(Applied Research Laboratories, 1981)

ANALYTICAL ($) PROGRAMS

Once all of the start-up procedures have been performed, the analytical programs can be run. The computer will take control of most instrument functions so be sure to have Displays, switches, etc. properly calibrated. Each program, when running, guides you through its operation with worded prompts. See flow chart on the next page for program selection. Following are the names and a brief description of the programs:

1) $SSET - sets up data (%) files for standards.

2) $QSET - constructs standard element package using % files and stores information in " * " (QDATA) file. **

3) $PSET - sets up instrumental parameters and data collection procedures. Automatically runs $STDZ program.

4) $STDZ - standardization program. Uses *FILE set-up previously. Has option of entering restandardization program ($RSTD) or analysis program ($ANLZ, $SST). Standardization data is stored in #STDZ file on your program disk. The contents of #STDZ can be printed with the "SDATA" program. Anytime you run the $STDZ program, the #STDZ file is overwritten and the previous standardization data are erased.

5) $RSTD - restandardization program. Individual elements in the *FILE package can be restandardized. The #STDZ file is updated with the new standard data.

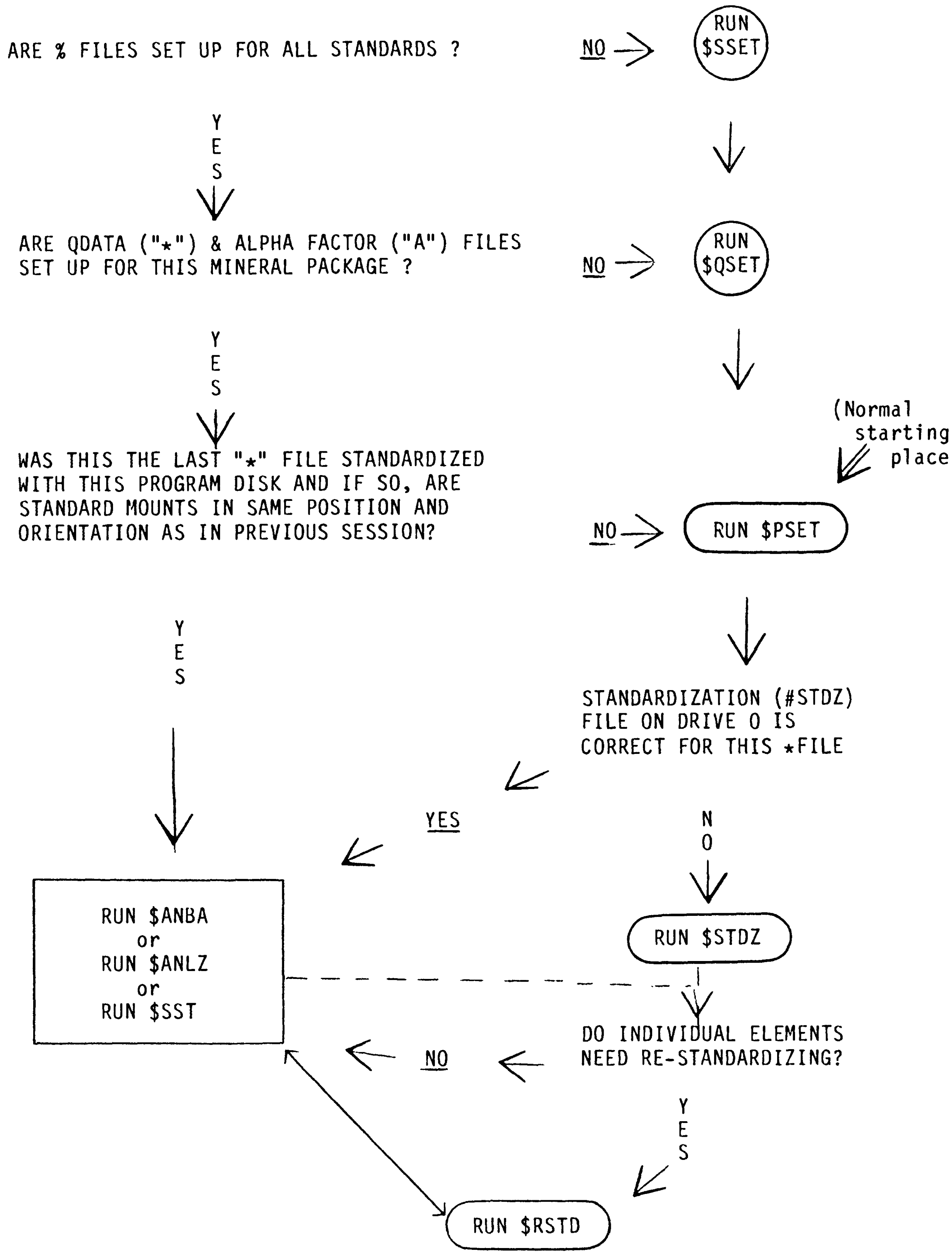
6) $ANLZ - point analysis program. Allows operator to average multiple analyses and select oxygen normalization values. Uses Bence-Albee or Magic correction procedures.

7) $SST - multiple point analysis program using pre-selected line, area or random XYZ stage coordinates.

8) $ANBA - analysis program with Bence-Albee correction procedures, multi-point averaging, data filing of of selected analysis results, and on-line plotting. $ANBA is the fastest, most applicable program for most mineral analytical problems. See extended description on pages 18-26.

Usually you will begin an analytical session with the $PSET program and you will choose $ANBA as your analysis program, since it is the fastest and most versatile of the available programs. Of course, you need to have a data file available that has the elements and standard information necessary for your particular analytical problem (this is called the *FILE- see pages 10 and 40-51). The flow diagram on the next page shows what programs are needed for a particular stage in the analysis set-up procedure. Descriptions of the various DATA FILES are given on page 14. Detailed descriptions of the first 7 programs are available in ARL (1979). Detailed description of $ANBA is available in McGee (1983).

** Throughout this manual, the convention "*FILE" is used for the data file referred to as the "QDATA" file in ARL (1979). The * is used as a prefix for these files to facilitate recognition of them on a disk.


ARE % FILES SET UP FOR ALL STANDARDS ?
NO
RUN $SSET
YES
ARE QDATA ("*") & ALPHA FACTOR ("A") FILES SET UP FOR THIS MINERAL PACKAGE ?
NO
RUN $QSET
YES
(Normal starting place)
WAS THIS THE LAST "*" FILE STANDARDIZED WITH THIS PROGRAM DISK AND IF SO, ARE STANDARD MOUNTS IN SAME POSITION AND ORIENTATION AS IN PREVIOUS SESSION?
NO
RUN $PSET
YES
STANDARDIZATION (#STDZ) FILE ON DRIVE 0 IS CORRECT FOR THIS *FILE
YES
NO
RUN $ANBA or RUN $ANLZ or RUN $SST
RUN $STDZ
DO INDIVIDUAL ELEMENTS NEED RE-STANDARDIZING?
NO
YES
RUN $RSTD

DATA FILE TYPES:

File type	Drive		Description
%_ _ _ _	1	-	Standards files; contain compositional data and sample stage locations for each standard. Contents can be listed with the "%LIST" program.
*_ _ _ _	1	-	"QDATA" files; mineral package containing info on spectrometer assignment, peak search parameters, and standard for each element. Contents can be listed with the "QLIST" program. See pages 52-54.
A_ _ _ _	1	-	Alpha factor file (Bence-Albee only); set up to correspond to "*" file.
#PDTA	0	-	Parameter file set up automatically by $PSET program; contains names of analysis program, *File, %File and count times; used for linking data to the various analytical programs.
#STDZ	0	-	Standardization data; peak, background, count times, and digitized beam current acquired in $STDZ and $RSTD programs are stored in this file. Contents correspond to the most recent "*FILE" used and can be listed with the "SDATA" program.
D_ _ _ _	1	-	If you elect to store data during the analysis session, your stored analyses will be in a data file, named by you. This file is 101 records long and is structured as described on page 56.

STANDARDIZATION-

A few items are worth pointing out about the standardization procedure (for both $STDZ and $RSTD programs) since this procedure is so critical to obtaining good quality data:

1. Standards should be chosen so that, ideally, you are using materials with properties (valence, bonding, coordination, concentration) similar to your unknown. Of course this is not always possible and need not be adhered to stringently - the data correction procedures usually compensate for minor differences. However, it is something to keep in mind if you are having problems with an element or mineral package.

It is important to select standards with concentrations high enough to produce reasonably high count rates (1000 cps or better) if possible. This will improve counting statistics and make peak location more accurate. Also, interpolation to lower concentrations in the unknowns, rather than extrapolation to higher ones, usually is better handled by the correction procedures.

2. Background standards are selected based on a combination of suitability, availability, and convenience. Backgrounds are measured during the standardization procedure. A background standard will give a better measure of the backgrounds during analysis of the unknowns if its average (or mean) atomic number ($\bar{Z}$) is similar to that of the unknown. The background standard must not have any measurable concentration of the element(s) for which it is being used as a background standard. There also should not be any overlap of peaks from the background standard at the wavelength of the element(s) of interest.

As a matter of convenience, background standards are often selected based on standard mount location--as long as $\bar{Z}$ is within 1 or 2 of the unknown's typical $\bar{Z}$. Highly accurate background measurements are only necessary if you are concerned about minor elements (particularly those with low Z).

To calculate $\bar{Z}$, sum the products of the material's oxide concentrations times the $\bar{Z}$ for that oxide (see table below):

$$\bar{Z} = \quad c_{ox} \cdot Z_{ox}$$

For example, fayalite's (SiO_2=29.5%, FeO=70.5%) $\bar{Z}$ is calculated as follows:

$$\bar{Z} = (.295 \times 10.8) + (.705 \times 21.99) = 18.7$$

Thus periclase (MgO), for example, with $\bar{Z}$=10.4 would not be suitable for measuring backgrounds in Fe-rich olivines.

The Table on the next page lists the $\bar{Z}$ for the most commonly analyzed oxides and for some "typical" minerals.

An automatic background calculation routine based on interpolation as a function of average atomic number is available (theoretically, the backgrounds are linear as a function of $\bar{Z}$). Selection of the $\bar{Z}$ background calculation option during set up of the *FILE will instruct the software to base background values on interpolation of high and low $\bar{Z}$ "end-member" measurements. The end-member background standards are measured in $STDZ.

Average Atomic Numbers of Some Oxides and Minerals

OXIDE	$\bar{Z}$
Na_2O	10.22
MgO	10.41
Al_2O_3	10.65
SiO_2	10.80
P_2O_5	11.05
K_2O	17.13
CaO	16.57
TiO_2	16.39
Cr_2O_3	18.94
MnO	21.16
FeO	21.99
Fe_2O_3	20.58
NiO	23.71
ZnO	25.68
Nb_2O_5	31.07
La_2O_3	49.77
Ce_2O_3	50.69
BaO	50.98
Yb_2O_3	62.44

Mineral	$\bar{Z}$
Albite	10.6
Diopside	12.2
Anorthite	12.2
Olivine (Fo_{90})	12.5
Hornblende	12.5-13
Apatite	13
Hedenbergite	15.5
Tephroite	18
Fayalite	18.7
Ilmenite	19

3. Counting times and # of measurements- Counting times of 20 seconds on peak and 10 seconds on background have generally produced acceptable results. If you desire better precision on minor elements, longer count times on both peak and background are necessary. If you are not too concerned with minor elements, count times of 10,4 for peak, background can be used during analysis of unknowns (but continue to use 20,10 for the standardization).

Measurements on the standards should be a minimum of 5 to adequately sample the material for heterogeneity (multiple grains should be used if possible). A larger number of measurements is statistically more meaningful, but a tradeoff is necessary at some point. If the "STD. DEV." column levels off from one point to the next (by 2 % or so), you have probably done enough points. If it turns out that this procedure did not produce a good random sample of the standard, as determined by analyzing a working standard or "known-unknown", the element(s) can be restandardized. It is potentially time-saving to automatically reject the first measurement on a standard in case the measured spot happened to be a bad surface, unrepresentative composition, etc.

4. Working standards ("known-unknowns") - careful selection and analysis of a working standard is an important method of evaluating the quality of your calibration procedures, and therefore of your unknown's data. A working standard should be chosen with the following criteria in mind:

a) it should be a homogeneous, well-analyzed (by independent methods) microprobe standard;

b) it should be of the same mineral type as your unknowns;

c) it should have chemistry similar to your unknowns;

d) it should not be used as a count-rate standard for any elements in your mineral package (unless there is no other logical choice).

You may have to compromise on some of these criteria and may need to use more than one working standard as a check on your calibration procedures.

The evaluation of your standardization is determined by the quality of your analysis of the working standard(s). "Analysis" of the working standard should consist of the average of multiple points (minimum of 3 to 5), preferably taken on several grains.

Suggested working standards for most of the mineral groups are indicated on the list of standards on pages 57-58.

5. Be sure when you are standardizing that you know the relative size and location of the beam spot and that the sample is optically in focus prior to taking data on each point. You should check that the spot is centered before starting the standardization (use a fluorescent sample - periclase, willemite) and that it's size is as desired (adjust with OBJ lens current adjust knobs).

6. Check that both the standard blocks and your samples have adequate, and grossly similar, conductive carbon coats. If the beam pulsates or jumps around (you can either see this on a fluorescing spot or on the sample current meter) the sample needs to be cleaned and re-coated.

$ANBA Program Operation-

$ANBA will operate from the video terminal and print just the analyses on the printing terminal, or the entire program can be run from the printing terminal. The program is configured so that the video terminal, printing terminal, and plotter correspond to ARLEB devices 0, 4, and 5, respectively.

The $ANBA program is loaded automatically following $STDZ, or $PSET. The program can be terminated and re-run at any time after standardization. However, the analysis number is reset to 1 each time the program is started. At the beginning of the program, the option to set up a new data file for storage of analyses is given. As long as the same *FILE is used, the operator may continue to store data in a file used in a previous analytical session (the previously stored data will not be over-written). The data file holds 100 analyses. When this file is filled to capacity, the program will prompt the operator for the name of a new data file.

The following six pages contain printout of the program in operation, showing both the computer/operator dialogue and the results printed by the program. Operator input is designated by a box, $\boxed{}$. Circled numbers in the right-hand column refer to explanatory notes that follow the operating example.

Note that if on-line plotting is desired, the "IPLOT" program should be run prior to starting the $ANBA program (see p. 27).

Operating Example

```
*ANBA -- ANALYSIS PROGRAM WITH BENCE-ALBEE CORRECTIONS, MULTI-POINT AVERAGING,
        DATA FILING, AND PLOTTING ROUTINES.  DEVELOPED FOR THE U.S.G.S.-RESTON
        SEMQ MICROPROBE FACILITY BY J.J. MCGEE   (OCTOBER, 1983 VERSION).


** ANALYZING FOR -- J.J. MCGEE - PROGRAM DEVELOPMENT      10  7  83

 USING: *GABT - GARNET-BIOTITE 9 ELMT SET WITH FIXED BKGDS   6-JAN-83

**NOTE - USE THE 'N' OPTION AFTER AN ANALYSIS TO EXIT PROGRAM OR RESTANDARDIZE

'CTRL P' WILL HALT EXECUTION BUT IS FOR EMERGENCY ONLY

ENTER # OF OXYGENS FOR FORMULA CALC.? 24                                        1

CHOOSE PRINTOUT FORMAT (1 = REGULAR, 2-LONG) - ? 1

DO YOU WANT TO FILE DATA? YES
ENTER FILENAME FOR DATA STORAGE ? D1011
'D1011' ALREADY EXISTS ON DRIVE 1
'W' TO WRITE OVER THIS FILE
'N' TO CREATE FILE WITH NEW NAME
'C' TO CONTINUE FILING IN 'D1011' (IF USING SAME *QDATA FILE)

 W, N, OR C -? C

DO YOU WANT ON-LINE PLOTTING ? YES
TERNARY - ENTER SEQUENCE # FOR TOP, LEFT, RIGHT ELEMENTS (ADD 15 IF OXIDE INSTEAD OF CATION PLOT) ? 3,2,6
CA   ,MG   ,FE   -ARE THESE CORRECT ? YES
INPUT COUNTING TIMES (SEC) FOR UNKNOWNS:
    (ON STANDARDS, PEAK= 20 SEC, BKGD= 10 SEC.) ? 20,10

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ? KAKANUI HORNBLENDE

ENTER X,Y STAGE POSITIONS TO DRIVE TO (5 DIGITS - ENTER 0,0 FOR NO MOTION)
? 7100,24000

DO YOU WANT TO CHECK THE BEAM? YES                                              2

SELECT ANALYSIS POSITION AND PUSH BUTTON WHEN READY


  ****** BENCE-ALBEE DATA REDUCTION METHOD ******


** SAMPLE:  KAKANUI HORNBLENDE   X= 7570.5  Y= 21414.5   B.C./SEC=  9779.65
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	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)	STD PEAK COUNTS	STD BKG COUNTS	ST TIME (SEC)	STANDARD FILENAME	LIN
SIO2	40.71	1.32	6.285	0.696	9789.3	27.0	20.0	14043.5	27.0	20.0	ZPXAD	KA
MGO	12.74	1.09	2.931	0.652	16386.8	978.0	20.0	24618.9	978.0	20.0	ZPXAD	KA
CAO	10.13	1.49	1.676	0.400	6417.9	40.0	20.0	15990.1	40.0	20.0	ZPXAD	KA
NA2O	2.78	1.57	0.832	0.202	5610.8	278.0	20.0	26708.3	278.0	20.0	ZFSTA	KA
K2O	1.96	2.84	0.386	0.127	1769.2	146.0	20.0	12954.4	146.0	20.0	ZFSBO	KA
FEO	10.37	1.56	1.338	0.138	5225.8	198.0	20.0	36601.2	198.0	20.0	ZOLSF	KA
AL2O3	14.04	0.60	2.554	0.658	47772.6	844.0	20.0	72180.6	844.0	20.0	ZFSTA	KA
MNO	0.02	152.89	0.002	0.000	238.1	224.0	20.0	68401.1	224.0	20.0	ZOLST	KA
TIO2	4.49	2.42	0.521	0.087	2020.1	60.0	20.0	22595.2	60.0	20.0	ZOXIL	KA

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TOTAL     97.23            16.526 CATIONS              ITERATIONS= 3
                           ( 24.0 OXYGENS)

12-OCT-83 09:24:26                                                              3

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ? S
(ANALYSIS # 15 FILED IN 'D1011')

PLOT THIS POINT ? Y
COMPONENTS ARE (TOP,LEFT,RIGHT):  0.28   0.49   0.23

(RESPONSES ARE: 'A'=INCLUDE IN AVG, 'AE'=INCLUDE AND CALCULATE AVG,
        'R'=REJECT, 'RE'=REJECT AND CALCULATE AVERAGE)
RESPONSE- (A, AE, R, RE) ? A
RESPONSE- A  ( 1 ANALYSES ACCEPTED IN THIS AVERAGING LOOP)

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ?


ENTER 'L' FOR NEW LABEL, 'B' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ?
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** SAMPLE: KAKANUI HORNBLENDE X= 7214 Y= 20940 B.C./SEC= 9764.45

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	39.45	1.33	6.168	0.674	9471.9	27.0	20.0
MGO	12.51	1.10	2.915	0.640	16096.8	978.0	20.0
CAO	10.20	1.49	1.708	0.403	6460.8	40.0	20.0
NA2O	2.77	1.57	0.838	0.200	5575.9	278.0	20.0
K2O	2.03	2.79	0.405	0.132	1831.6	146.0	20.0
FEO	10.25	1.56	1.340	0.137	5171.8	198.0	20.0
AL2O3	14.33	0.60	2.640	0.672	48746.7	844.0	20.0
MNO	0.04	67.06	0.005	0.000	256.7	224.0	20.0
TIO2	4.73	2.36	0.556	0.092	2125.6	60.0	20.0
TOTAL	96.30		16.577 CATIONS (24.0 OXYGENS)			ITERATIONS= 3	

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ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ?
RESPONSE- (A, AE, R, RE) ? A
RESPONSE- A (2 ANALYSES ACCEPTED IN THIS AVERAGING LOOP)

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ?

ENTER 'L' FOR NEW LABEL, 'B' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ? B

BEAM IS NOW ON

ENTER 'L' FOR NEW LABEL, 'B' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ?

** SAMPLE: KAKANUI HORNBLENDE X= 7592.5 Y= 21409.5 B.C./SEC= 9743.95

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	40.59	1.32	6.253	0.694	9759.4	27.0	20.0
MGO	12.64	1.09	2.903	0.646	16242.9	978.0	20.0
CAO	10.30	1.48	1.699	0.407	6523.7	40.0	20.0
NA2O	2.71	1.59	0.810	0.196	5462.6	278.0	20.0
K2O	1.96	2.84	0.385	0.127	1770.7	146.0	20.0
FEO	10.65	1.54	1.372	0.142	5365.3	198.0	20.0
AL2O3	14.07	0.60	2.555	0.659	47868.0	844.0	20.0
MNO	0.01	180.49	0.002	0.000	235.9	224.0	20.0
TIO2	4.70	2.37	0.544	0.091	2111.8	60.0	20.0
TOTAL	97.62		16.523 CATIONS (24.0 OXYGENS)			ITERATIONS= 3	

12-OCT-83 09:28:26

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ? S
(ANALYSIS # 16 FILED IN 'D1011')

PLOT THIS POINT ? Y
COMPONENTS ARE (TOP,LEFT,RIGHT): 0.28 0.49 0.23
RESPONSE- (A, AE, R, RE) ? AE
RESPONSE- AE

4

5

********** 3 POINT AVERAGE **********

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	40.25	1.82	6.236	0.688	9673.5	27.0	20.0
MGO	12.63	0.95	2.916	0.646	16242.2	978.0	20.0
CAO	10.21	0.83	1.695	0.403	6467.5	40.0	20.0
NA2O	2.75	1.47	0.827	0.199	5549.7	278.0	20.0
K2O	1.98	2.17	0.392	0.128	1790.5	146.0	20.0
FEO	10.42	1.97	1.350	0.139	5254.3	198.0	20.0
AL2O3	14.15	1.14	2.583	0.663	48129.1	844.0	20.0
MNO	0.02	58.53	0.003	0.000	243.5	224.0	20.0
TIO2	4.64	2.83	0.541	0.090	2085.8	60.0	20.0
TOTAL	97.05		16.542 CATIONS (24.0 OXYGENS)			ITERATIONS= 3	

12-OCT-83 09:29:01

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ? S
ENTER NEW TITLE FOR AVERAGE (OPTIONAL)? HORNBLENDE STD- 3 PTS
(ANALYSIS # 17 FILED IN 'D1011')

PLOT THIS POINT ?

6

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ? KAKANUI GARNET 7

ENTER 'L' FOR NEW LABEL, 'R' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ? N

EXIT PROGRAM ?
CHANGE COUNTING TIMES ? NO 8

ENTER NEW # OXYGENS FOR CATION CALC. ('RETURN' FOR NO CHANGE) ? 12

ENTER X,Y STAGE POSITIONS TO DRIVE TO (5 DIGITS - ENTER 0,0 FOR NO MOTION)
? 12000,15000

DO YOU WANT TO CHECK THE BEAM? N

SELECT ANALYSIS POSITION AND PUSH BUTTON WHEN READY

** SAMPLE: KAKANUI GARNET X= 18711.5 Y= 13196 B.C./SEC= 9922.35

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	41.63	1.33	2.973	0.685	9626.3	27.0	20.0
MGO	18.56	0.96	1.975	0.996	24528.8	978.0	20.0
CAO	5.04	1.97	0.385	0.197	3185.5	40.0	20.0
NA2O	0.01	102.97	0.002	0.001	301.4	278.0	20.0
K2O	0.00	49.29	0.000	0.000	113.3	146.0	20.0
FEO	10.53	1.55	0.629	0.140	5294.8	198.0	20.0
AL2O3	23.87	0.52	2.009	1.118	80582.9	844.0	20.0
MNO	0.25	11.44	0.015	0.003	451.3	224.0	20.0
TIO2	0.31	11.70	0.017	0.006	197.4	60.0	20.0

TOTAL 100.20 8.006 CATIONS ITERATIONS= 3
(12.0 OXYGENS)

12-OCT-83 09:32:39

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ? Y
(ANALYSIS # 18 FILED IN 'D1011')

PLOT THIS POINT ? A\A\
Y
COMPONENTS ARE (TOP,LEFT,RIGHT): 0.13 0.66 0.21
RESPONSE- (A, AE, R, RE) ? A
RESPONSE- A (1 ANALYSES ACCEPTED IN THIS AVERAGING LOOP)

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ?

ENTER 'L' FOR NEW LABEL, 'R' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ?

** SAMPLE: KAKANUI GARNET X= 18656 Y= 13055.5 B.C./SEC= 9915.45

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	42.56	1.32	3.008	0.702	9863.6	27.0	20.0
MGO	18.37	0.96	1.935	0.984	24280.7	978.0	20.0
CAO	5.32	1.93	0.403	0.208	3361.6	40.0	20.0
NA2O	0.01	153.29	0.001	0.001	293.6	278.0	20.0
K2O	0.00	295.01	0.000	0.000	140.3	146.0	20.0
FEO	10.50	1.55	0.620	0.140	5277.5	198.0	20.0
AL2O3	23.80	0.52	1.983	1.117	80505.1	844.0	20.0
MNO	0.25	11.55	0.015	0.003	448.6	224.0	20.0
TIO2	0.35	10.74	0.019	0.007	214.5	60.0	20.0

TOTAL 101.15 7.983 CATIONS ITERATIONS= 3
(12.0 OXYGENS)

12-OCT-83 09:34:17

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ?
RESPONSE- (A, AE, R, RE) ? AE

RESPONSE- AE

********** 2 POINT AVERAGE **********

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	42.09	1.73	2.990	0.693	9745.0	27.0	20.0
MGO	18.46	0.72	1.955	0.991	24408.7	978.0	20.0
CAO	5.18	3.85	0.394	0.203	3273.6	40.0	20.0
NA2O	0.01	28.23	0.001	0.001	297.5	278.0	20.0
K2O	0.00	0.00	0.000	0.000	126.8	146.0	20.0
FEO	10.51	0.24	0.625	0.140	5206.1	198.0	20.0
AL2O3	23.84	0.07	1.996	1.117	80544.0	844.0	20.0
MNO	0.25	0.84	0.015	0.003	450.0	224.0	20.0
TIO2	0.33	8.28	0.018	0.006	205.9	60.0	20.0

TOTAL 100.68 7.994 CATIONS (12.0 OXYGENS) ITERATIONS= 3

12-OCT-83 09:34:59

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ? S 9
ENTER NEW TITLE FOR AVERAGE (OPTIONAL)? GARNET STD (KAKANUI)
(ANALYSIS # 19 FILED IN 'D1011')

PLOT THIS POINT ? Y
COMPONENTS ARE (TOP,LEFT,RIGHT): 0.13 0.66 0.21

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ? LAKE CNTY PLAGIOCLASE

ENTER 'L' FOR NEW LABEL, 'B' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ? N

EXIT PROGRAM ? NO
CHANGE COUNTING TIMES ? YES
INPUT COUNTING TIMES (SEC) FOR UNKNOWNS:
? 10,10 10

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ?

ENTER 'L' FOR NEW LABEL, 'B' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ? N

EXIT PROGRAM ?
CHANGE COUNTING TIMES ?

ENTER NEW # OXYGENS FOR CATION CALC. ('RETURN' FOR NO CHANGE) ? 8

ENTER X,Y STAGE POSITIONS TO DRIVE TO (5 DIGITS - ENTER 0,0 FOR NO MOTION)
? 7000,7000

DO YOU WANT TO CHECK THE BEAM? Y

SELECT ANALYSIS POSITION AND PUSH BUTTON WHEN READY

** SAMPLE: LAKE CNTY PLAGIOCLASE X= 3209.5 Y= 3328 B.C./SEC= 9900.60

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	49.69	1.55	2.332	0.854	5999.1	13.5	10.0
MGO	0.00	26.63	0.000	0.000	378.4	489.0	10.0
CAO	13.28	1.76	0.668	0.516	4133.6	20.0	10.0
NA2O	4.02	1.69	0.366	0.324	4426.5	139.0	10.0
K2O	0.11	29.82	0.007	0.007	119.6	73.0	10.0
FEO	0.29	23.24	0.012	0.004	169.5	99.0	10.0
AL2O3	29.77	0.57	1.647	1.565	56247.6	422.0	10.0
MNO	0.00	21.36	0.000	0.000	52.0	112.0	10.0
TIO2	0.00	376.07	0.000	0.000	28.0	30.0	10.0

TOTAL 97.17 5.031 CATIONS (8.0 OXYGENS) ITERATIONS= 3

12-OCT-83 09:38:52

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ?
RESPONSE- (A, AE, R, RE) ? R
RESPONSE- R
SAMPLE LABEL ('RETURN' FOR NO CHANGE) ?

ENTER 'L' FOR NEW LABEL, 'B' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ? ☐

** SAMPLE: LAKE CNTY PLAGIOCLASE X= 3038 Y= 3372 B.C./SEC= 9898.90

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	50.96	1.53	2.345	0.877	6160.2	13.5	10.0
MGO	0.00	23.43	0.000	0.000	364.3	489.0	10.0
CAO	13.63	1.74	0.672	0.529	4241.7	20.0	10.0
NA2O	3.94	1.71	0.352	0.318	4340.2	139.0	10.0
K2O	0.09	36.92	0.005	0.006	109.6	73.0	10.0
FEO	0.21	30.67	0.008	0.003	150.5	99.0	10.0
AL2O3	30.16	0.57	1.635	1.587	57030.2	422.0	10.0
MNO	0.00	68.10	0.000	0.000	71.1	112.0	10.0
TIO2	0.00	44.69	0.000	0.000	15.0	30.0	10.0

TOTAL 99.00 5.016 CATIONS ITERATIONS= 3
(8.0 OXYGENS)

12-OCT-83 09:40:19

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ? Y
(ANALYSIS # 20 FILED IN 'D1011')

PLOT THIS POINT ? Y
COMPONENTS ARE (TOP,LEFT,RIGHT): 0.99 0.00 0.01
RESPONSE- (A, AE, R, RE) ? A
RESPONSE- A (1 ANALYSES ACCEPTED IN THIS AVERAGING LOOP)

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ? ☐

ENTER 'L' FOR NEW LABEL, 'B' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ? ☐

** SAMPLE: LAKE CNTY PLAGIOCLASE X= 3368 Y= 3153.5 B.C./SEC= 9894.40

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	52.07	1.52	2.372	0.898	6309.0	13.5	10.0
MGO	0.00	32.30	0.000	0.000	396.9	489.0	10.0
CAO	13.58	1.74	0.663	0.527	4224.4	20.0	10.0
NA2O	3.88	1.72	0.343	0.313	4273.0	139.0	10.0
K2O	0.07	44.66	0.004	0.005	102.7	73.0	10.0
FEO	0.20	32.34	0.008	0.003	147.6	99.0	10.0
AL2O3	29.96	0.57	1.608	1.577	56687.7	422.0	10.0
MNO	0.00	25.84	0.000	0.000	61.1	112.0	10.0
TIO2	0.00	787.18	0.000	0.000	31.0	30.0	10.0

TOTAL 99.76 4.997 CATIONS ITERATIONS= 2
(8.0 OXYGENS)

12-OCT-83 09:42:11

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ? ☐
RESPONSE- (A, AE, R, RE) ? A
RESPONSE- A (2 ANALYSES ACCEPTED IN THIS AVERAGING LOOP)

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ? ☐

ENTER 'L' FOR NEW LABEL, 'B' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ? ☐

** SAMPLE: LAKE CNTY PLAGIOCLASE X= 3466.5 Y= 3167 B.C./SEC= 9894.60

	WEIGHT%	ST.DEV. (%)	FORMULA	K-RAT	UNKN PK COUNTS	UNKN BKG COUNTS	TIME (SEC)
SIO2	50.12	1.54	2.334	0.862	6054.4	13.5	10.0
MGO	0.00	31.22	0.000	0.000	393.8	489.0	10.0
CAO	13.67	1.74	0.682	0.531	4254.7	20.0	10.0
NA2O	3.80	1.74	0.343	0.306	4180.8	139.0	10.0
K2O	0.05	62.36	0.003	0.003	93.7	73.0	10.0
FEO	0.14	44.19	0.006	0.002	133.5	99.0	10.0
AL2O3	30.03	0.57	1.648	1.581	56830.3	422.0	10.0
MNO	0.00	32.20	0.000	0.000	70.1	112.0	10.0
TIO2	0.00	52.72	0.000	0.000	17.0	30.0	10.0

TOTAL 97.81 5.015 CATIONS ITERATIONS= 3
(8.0 OXYGENS)

12-OCT-83 09:43:46

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ? ☐
RESPONSE- (A, AE, R, RE) ? RE
RESPONSE- RE

11

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*********** 2 POINT AVERAGE ***********

        WEIGHT%  ST.DEV. FORMULA  K-RAT   UNKN PK  UNKN BKG   TIME
                  ( % )                    COUNTS   COUNTS    (SEC)

SIO2     51.53     1.69   2.358   0.888    6234.6     13.5    10.0
MGO       0.00     0.00   0.000   0.000     380.6    489.0    10.0
CAO      13.60     0.29   0.667   0.528    4233.0     20.0    10.0
NA2O      3.91     1.14   0.347   0.315    4306.6    139.0    10.0
K2O       0.08    14.78   0.005   0.005     106.1     73.0    10.0
FEO       0.21     4.16   0.008   0.003     149.0     99.0    10.0
AL2O3    30.06     0.43   1.621   1.582   56859.0    422.0    10.0
MNO       0.00     0.00   0.000   0.000      76.1    112.0    10.0
TIO2      0.00     0.00   0.000   0.000      23.0     30.0    10.0

TOTAL    99.39            5.007 CATIONS            ITERATIONS= 3
                        (  8.0 OXYGENS)

12-OCT-83 09:44:10

ENTER 'Y' ('S' IF STANDARD) TO STORE ANALYSIS AND/OR PLOT ? [S]
ENTER NEW TITLE FOR AVERAGE (OPTIONAL)? [ ]
(ANALYSIS # 21 FILED IN 'D1011')

PLOT THIS POINT ? [ ]

SAMPLE LABEL ('RETURN' FOR NO CHANGE) ? [N]

ENTER 'L' FOR NEW LABEL, 'B' FOR BEAM ON, 'N' FOR NEW OPTIONS OR PROGRAM EXIT
- OR 'RETURN' TO START ANALYSIS ? [N]

EXIT PROGRAM ? [YES]                                                    12

****  9  **** ANALYSES OBTAINED    (EXCLUDING AVERAGES)


ENTER 'R' TO RESTANDARDIZE 1 TO  8  ELEMENTS-
   OR 'S' TO REMEASURE BACKGROUNDS OR STANDARDIZE ENTIRE FILE -
(HIT 'RETURN' FOR NEITHER OF THESE) - ? [ ] .
IF YOU ARE DONE FOR THE DAY -.


RUN THE 'SDATA' PROGRAM TO GENERATE STANDARDIZATION TABLE

PUT PRINTOUT OF STANDARDIZATION TABLE IN LOGBOOK
AND FOLLOW SHUTDOWN PROCEDURES IN MANUAL

--ENJOY THE DATA--

READY

                                                                        13
[RUN SDATA]
ADVANCE PAPER TO TOP OF PAGE-  PUSH ANY KEY WHEN READY



STANDARDIZATION DATA FOR FILE *GABT      12-OCT-83

EL. CH.  PEAK  COUNTS SEC.  BKGD SEC.  BC/SEC  STNDRD  BG STD

SI   9   FIX   14044   20    27   20   10017   ZPXAD   ZOXCO
MG   7   FIX   24619   20   489   10   10017   ZPXAD   ZPXWO
CA   6   FIX   15990   20    40   20   10017   ZPXAD   ZOXCO
NA   4   FIX   26708   20   139   10    9902   ZFSTA   ZPXWO
K    1  14193  12954   20    73   10    9864   ZFSBO   ZPXWO
FE   8   FIX   36601   20    99   10    9933   ZOLSF   ZPXWO
AL   2  13109  72181   20   422   10    9902   ZFSTA   ZPXWO
MN   5   FIX   68401   20   112   10    9907   ZOLST   ZPXWO
TI   3  27471  22595   20    30   10    9892   ZOXIL   ZPXWO
```

Operating Notes (see numbers in examples):

1. The data filing and plotting routines are set-up at this point. In the example, filing is "continued" in a pre-existing file which was set up in a previous run when the same QDATA file was used. If filing is not chosen, plotting will not be allowed. If neither filing nor plotting options are chosen, the queries to file or plot will be skipped after each analysis.

The "long" printout format is automatically used for the first analysis. This format includes count data for the standards. If a "regular" format is chosen, the second and all subsequent analysis printouts will skip the printing of redundant standard information (see the printout examples). The standardization data is printed out at the end of the operating shift by running the program "SDATA", which generates a compact table of standard count data.

2. The beam is "blanked" unless the operator responds with a "yes" to the "Check the beam" question. If you do unblank the beam and are moving around on the sample, manually blank the beam when you are moving across epoxy. Be sure to manually unblank the beam again when you are on the desired grain. To start an analysis after checking the beam, the joystick button is pressed.

3. These are the various options after an analysis is printed out. This analysis was stored as a standard ("S"). If filing was not desired, the "Return" would have been depressed instead of "Y" or "S". If the analysis is stored, and if on-line plotting was selected in the set-up, the operator is given the option to plot the preselected components.

The echoing of the "RESPONSE" message in the example is due to the fact that this entire operating example was generated on a printing terminal. Normal operation would be to operate the program from a video terminal. If a video terminal is used, the dialogue and program prompts are displayed on the video terminal and the analysis, storage counter, and averaging response are transmitted to the printing terminal. The blank box signifies that a "Return" was depressed to start the next analysis.

4. Note that since the analysis was not stored, the plotting option was skipped.

The "B" option is chosen to turn the beam on. The operator can then identify the material and enter a new label, if desired, prior to starting the next analysis.

5. The "AE" response signals the program to calculate the average of all of the accepted analyses. The average is calculated by averaging the uncorrected count data for each accepted analysis and then re-running the correction routine on the uncorrected average. In this case, a 3 point average is calculated and printed. The "ST. DEV." column in an average analysis gives the standard deviation, in percent, of the average analysis. Thus, the standard deviation of the SiO_2 value in this average is 40.25 $\pm$ 1.82% of 40.25, or 40.25 $\pm$ .73.

6. The blank box signifies that a "RETURN" has been depressed. In the program, the "NO" response can be signalled by "No", "N", or "RETURN". The "YES" response is signalled by "YES" or "Y".

7. A new label is entered here. After an average is calculated, any previous label is erased. Otherwise, the previous label is carried over unless a new label is entered.

8. "New Options" are chosen and displayed here. Any or all of the analysis parameters can be changed. If a new label is desired, it should be entered first and then the choice for other "New Options" can be made when the analysis option prompt is repeated.

9. A new title is entered to be stored with the average analysis.

10. New counting times are entered and the "New Options" choice is repeated so as to modify other analysis parameters.

11. The "RE" response excludes the last analysis from the averaging array and then calculates the average of the previously accepted analyses.

12. Program is exited at this point. If desired, the $STDZ or $RSTD programs can be automatically loaded and run here.

13. The "SDATA" program is separate from $ANBA. It is used to print out the standardization data, which is then entered in the operator's logbook. This is the data that is omitted from the analysis printout if a "regular" printout format is chosen at the beginning of the program.

On-line Plotting-

Plotting routines in the $ANBA program allow you to plot your data in real-time mode. The procedures are straightforward but require that you know what you want to plot. Three short routines to drive the plotter are available on your program disk and are described briefly below. If you wish to plot in a format other than those provided, feel free to modify the routines or generate your own. Plotting control commands are listed in the back of Operating manual in the lab. The routines needed to obtain plotting are described below:

IPLOT - This program initializes the plotter, draws the desired figure, and labels the axes based upon operator input. Run this program prior to running $PSET or $ANBA.

PLOT2 -
PLOT3 - These two routines provide the initial set-up dialogue and actual plotting commands needed to perform on-line plotting in the $ANBA program. They are used for 2-axis (PLOT2) and ternary (or quad) plots (PLOT3) and their line numbers are set up to overwrite each other in $ANBA. These routines can be modified for other specific applications (such as an AFM plot).

To install PLOT2 or PLOT3 (or any modification thereof) in $ANBA, insert your disks in the computer and proceed as follows:

READY

LOAD $ANBA <cr>

READY

BLEND PLOT3 <cr>

(several second pause here)

READY

STORE $ANBA <cr>

(if you get a DISK ERROR here, you need to "PACK" your disk. See p. 49.)

READY

The above sequence of commands combines the software necessary to plot ternary diagrams (PLOT3) with the analytical program ($ANBA) and then stores this combined version as your new version of $ANBA. For 2-axis plots, repeat this procedure with "PLOT2".

For on-line plotting, $ANBA requires that you file data in order to plot it. You must first set up a data file for storage and then you will be prompted for plotting information. After each analysis, you must respond with a 'Y' or 'S" to the file prompt in order to receive the plot prompt. This was done in order to insure that, if you want to generate any additional off-line plots (on the DEC-MINC computer), the data will be available and in a format ready to be used.

You may construct other plotting routines or components but be sure to conform to the line numbers and variable assignments (see below) for the plotting routines as they now exist for incorporation into $ANBA.

Plotting variables:

PLOT 2

A5 = Plotter Select Code
B1 = Element Symbol Array
D = Array for Plotter Codes; Cation values
E0 = # of elements analyzed
E1 = Analysis #
F0 = Flag for X Axis Plot Type
P7 = X-Axis Component
P8 = Y-Axis Component
R0 = X-Axis Minimum
R2 = X-Axis Maximum
R3 = Y-Axis Minimum
R4 = Y-Axis Maximum
W = Oxide Wt % values
X = Yes/No input variable
X4 = X Axis element #
X5 = 2nd element of X-Axis Ratio
Y2 = Flag for Y-Axis Plot Type
Y4 = Y-Axis element #

PLOT 3

A0 = Element # for Top
A4 = Plot Component for Top
A5 = Plotter Select Code
B1 = Element Symbol Array
D = Array for Plotter Codes; cation values
E0 = # of elements analyzed
F0 = Flag for Plot type (cations or oxides)
L0 = Element # for left
L4 = Plot component for left
R0 = Element # for Right
R4 = Plot component for Right
T0 = Cation/oxide sum for Plot
W = Wt $ values
X = Yes/no input variable

Auxiliary/Utility Programs and Commands-

The following programs or operations are sometimes needed during, or just prior to, an operating shift. They are used to prepare either you, your disks, or the probe for subsequent actions:

QLIST - Lists the contents of the *FILES (see p. 52). This program will tell you what spectrometers and standards you need for a particular analytical package.

FREE - Removes Scalers, Digital Drives, and Beam Blank switch from computer control.

IPLOT - Sets up plotter for subsequent 2 or 3 axis plots. This program will draw and label the axes and leave the plotter ready for on-line operation (see p. 27).

PACK - Consolidates all of the unused records on a disk (converts "CLEAR" records to "FREE" records). This operation can be routinely performed prior to each operating shift.

INDEX - Prints out a listing of disk contents, including file name, location (starting record), and storage date. This command defaults to Disk Drive 0. To examine contents of disk in Drive 1, enter <u>INDEX</u> <u>1</u>.

CLEAR - Erases the specified file from the specified drive, e.g.:

CLEAR DTEST,1

will erase the data file called "DTEST" from the disk in Drive 1.

CHECK - Examines the disk surface for error-free read/write capability. If a disk is physically damaged, this program will report so.

Additional programs for various file and disk operations and for off-line data manipulation are described in the section "Offline Software Operations" (p. 49-55).

INSTRUMENT CONTROL/OPERATING NOTES

Fiducial Marks -

Fiducial marks are reference points used to automatically locate standard positions. Fiducial marks are arbitrarily selected when "%" files are first set up (in $SSET). These marks are simply two recognizable features located on the standard block that are found by the operator at the beginning of each shift. The computer locates the positions of all standards on that block based on the relative X, Y, Z positions of the two fiducial marks.

All % files on the Reston SEMQ were set up using the same two fiducial marks on each standard block (the numbers "4" and "8"):

Fiducial mark #1 is located at the intersection of the vertical and horizontal bars as marked:

4←

(do positioning with high power magnification)

Fiducial mark #2 is located at the intersection as marked:

8←

The fiducial mark location procedure will be repeated for all standard blocks needed for the standardization package being used.

Signal Selector and Range Knobs -

The signal selector knob selects which signal will be recorded on the Picoammeter. For analysis, the "BC" and "SC" are the only signals you need to look at. If the selector is set to "BC" the beam current will be recorded on the meter. This current is usually set in the range 0.05 to 0.15 $\times 10^{-6}$ amps (=microamps). We usually operate at 0.10 $\times$ 10^{-6} amps. Thus to record this signal on the meter, the Range knob should be set to read 0.1 or 0.3 $\times$ 10^{-6} amps at full scale. This is the signal and range to use when saturating the filament.

During analysis the Signal Selector knob is set to "SC" or sample current. The sample current signal is generally on the order of 1/10 the beam current signal. At a beam current of 0.1×10^{-6} amps, most of the materials we analyze have sample current signals of 0.005 to 0.020 $\times 10^{-6}$ amps. Thus, if you wish to see what the sample current value is, you would probably best be able to read it with the range knob set to 0.03 $\times$ 10^{-6} amps full scale. However, despite the instructions in the programs, it is not mandatory to switch the range knob to this scale - it is just for your convenience. It _is_ mandatory to leave the Signal Selector knob set to "SC". At this setting the sample current signal is readable from the meter and the beam current signal is scaled to read out on the 7th scaler. This readout is then used in the analysis program to correct for beam current drift.

You can switch to "BC" prior to an analysis, when you are checking the beam spot, to verify that the filament is still saturated and that the current value is correct. However, as noted above, the beam current is approximately 10 times the value of the sample current - so be sure to switch the range knob to a scale that will prevent the meter from "pegging" at the high end (0.3 or 0.1 x 10^{-6} should be OK).

Beam Blanking-

The electron beam is automatically controlled by the computer during standardization and analysis. However, in order for the computer to gain control the "BEAM BLANK" button must be out. If this button is pushed in, the beam is manually blanked or cut off. It is desirable to manually blank the beam (button pushed in) if you are moving around on material sensitive to damage by the beam, such as epoxy. However, if the BEAM BLANK button is lit up while it is out (not depressed), the computer has control of the beam and the beam is already "blanked" (there is no need for you to push the button in). In the programs, responding "yes" to the question "DO YOU WANT TO CHECK THE BEAM SPOT" has the effect of "unblanking" the beam (the BEAM BLANK light will go out and the beam will come on).

The standardization and analysis programs include options for checking the beam spot prior to acquiring count data. In the standardization (and restandardization) program the question "DO YOU WANT TO CHECK THE BEAM SPOT" is asked prior to the first counting sequence on each standard. If you answer yes to this question the beam is automatically, and instantaneously, unblanked or focused (the BEAM BLANK light will go out). You can then check the position and shape of the beam spot, if the sample fluoresces. You can also check the beam current to see if it is saturated and set at the proper value at this time. If for some reason you move off of the sample and on to epoxy, you must push the BEAM BLANK button in. Once you are positioned back on the sample you can again unblank the beam by pushing the BEAM BLANK button out. You must leave this button out when you are ready to have the count data acquired (the computer cannot control the beam if the BEAM BLANK button is pushed in).

The commands to control the beam thru the computer are:

CALL (7,0,0,0) -unblank beam

CALL (7,0,0,1) -blank beam

Transmitted Light Assembly Installation/Removal-

The transmitted light assembly (TLA) (Fig. 3, p. 32) fits in the Sample Stage, underneath the aluminum sample holder. For some applications (and for bulb replacement), the TLA must be removed. Installation or removal of the TLA is fairly simple, but care must be taken not to damage any of the sample stage components or the assembly itself. Clearances between the TLA, Sample Stage, and Secondary Electron Detector (below the light optics) are fairly tight so the following steps for TLA installation should be followed attentively:

1. Put Stage Digital Drive in MAN mode. Lower Elevator and Z axis (~ 324) and open Sample Chamber (push SC VENT);

2. Swing open the hinged door surrounding the optics assembly (this must be done to allow clearance);

3. Move the Y axis to its forward-most position (towards you)- dial will read ~000. Move X axis to center-left position (between 300 and 500 on dial). Remove sample holder;

4. Position the TLA at rear of sample chamber, between the top of the chamber and the Secondary Electron Detector (do not scrape the O-ring). The guide on the front of the TLA should be pointing towards you;

5. Raise Elevator ("Elevator Up" button) until the large platform below the sample holder brackets is just below (1/2 inch) the top surface of the sample chamber. There is about a 1-second delay between the time you release the button and when the elevator actually stops, so release the button when the platform is still below the top rim (be careful- if you go too high you will jam the TLA between the platform and Detector). The TLA rests on this platform and will be slid in from the back of the sample stage;

6. Slowly raise the Z axis until the platform is nearly level with the chamber top. Slide the TLA onto the platform until it seats properly and is forward and clear of the chamber rim- the knurled thumb screw will stop in its slot (on the right rear) and the cylindrical guide at the front of the TLA will be seated against the lower side of the copper clip. Be sure that the thumb screw's washer is on the outside of the bracket;

7. Tighten the thumb screw and connect the double-lead wire connector at the back of the TLA with the mating connector on the platform;

8. Check that the TLA operates with the controls in the upper left corner of the vacuum panel;

9. Lower Z axis and Elevator completely (this is very important!) and re-install sample holder. Check for clearance between the sample holder and TLA as the X and Y axes are moved;

10. Check O-ring, close sample chamber (push SC PUMP). Close optics door and recalibrate stage Digital Display.

TLA Removal:
To remove the TLA, repeat steps 1-3. Raise Z axis as in step 6. Reverse step 7 and then the rest of step 6. Then do steps 4, 9 and 10.

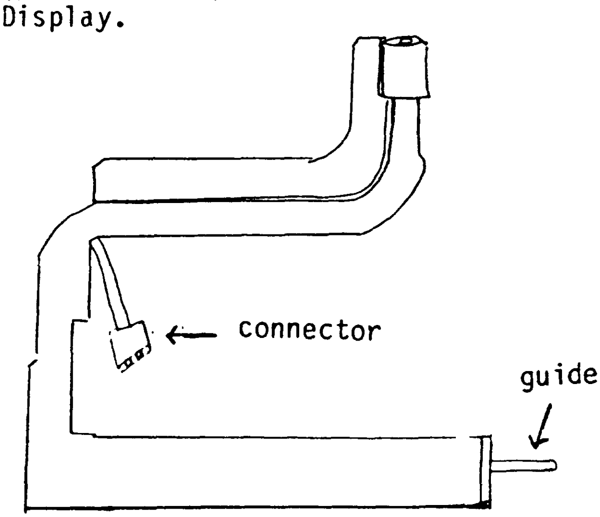


Fig. 3 - Transmitted light assembly

FILAMENT REPLACEMENT-

If the filament has blown you will see no emission current, filament voltage, or beam current when the high voltage is on and the filament is turned up to normal settings (550-650). Before replacing a filament, be sure that loss of current is not due to some other problem such as the beam being blanked, or the Main Gate valve closing due to vacuum or electrical failure or depletion of the nitrogen gas supply (check vacuum panel LED's). If the beam is blanked and is under computer control exit the program with a "CTRL P" and enter the following command to unblank the beam: CALL (7,0,0,0). If the Emission and Filament Current meters read 0 and the Gun HV light is on then the filament is burned out.

To replace the filament, proceed as follows:

1) Turn down high voltage and filament power to zero:

1.1- Turn saturation (Filament Voltage) dial to zero (on gun HV supply);

1.2- Turn kV to zero by depressing kV select buttons sequentially down to 0;

1.3- Turn Gun HV power off (on gun HV supply).

2) Vent gun chamber:

2.1- Close the electron gun isolation valve (V3). This is a manually operated valve with a handle attached to the front of the gun which closes with a 90° counterclockwise (CCW) turn. The valve handle will then be horizontal and the V3 light on the vacuum control panel will be out;

2.2- Check that the vacuum panel switches for V4, V1, and P1 agree with their actual status (you will be switching from Auto to Manual vacuum mode and the position of the switches will determine what the valves and pump do when you turn the key- you do not want them to do anything. In Auto mode these switches are disabled, but they become enabled when the key is turned to Manual.);

2.3- If the switches agree with the LED's on the vacuum control schematic, turn the vacuum key to Manual;

2.4- Vent N_2 gas into the gun chamber by toggling the V7 switch. Only toggle the switch once (it will spring back). N_2 will bleed into the isolated gun chamber until the chamber reaches atmospheric pressure (V7 will stay lit for 2-3 seconds).

3) Remove old filament:

3.1- When the V7 light goes out, swing the electron gun back until it comes to rest in a horizontal position. Use the large screwdriver to ground the gun by making contact between the grid cap and the gun housing at the base. Remove the grid cap by turning CCW (cap will be quite warm- use the brass holder);

3.2- Put gloves on. Always wear gloves when touching the filament or inside surfaces of the electron gun;

3.3- Grasp the ceramic base of the filament, rotate it 90° (the two posts holding the filament will be vertical), and pull the filament assembly out.

4) Install new filament:

4.1- On the new filament check that the mounting pins below the base are not spread open too far or closed too tightly. These pins will be rotated into position so as to contact the brass socket pins in the gun. This holds the filament in place. If the filament pins are spread too wide, they will bend and not make proper contact. If the filament pins are too close, they also will not make proper contact and the filament may pop out. The spacing of the pins at the end should be approx. 1/4 to 5/16 inch (compare to the line at the right); ----

4.2- Insert the filament with the pins vertical and rotate the filament 90°. You will hear or feel a slight click as the filament pins snap into place between the gun socket pins (the filament should now be horizontal). Gently pull on the filament ceramic base to make sure the filament is snug;

4.3- Remove the grid cap (use gloves) from the brass heat sink and clean the cap off with ethyl alcohol and kimwipes or cotton swabs. Thread the grid cap back unto the gun, turning clockwise (CW) until the filament tip is visible in the hole (use flashlight if necessary). Using the four large gun centering screws on the gun, center the filament tip in the hole (diametrically opposing centering screws are used in tandem by turning both hands in the same direction- one screw will turn CCW and the other CW);

4.4- Turn the grid cap CW until the filament tip is flush with the cap's surface. Check by gently scraping a straight edge of paper backwards (i.e., towards you) across the hole (Fig. 4, p. 36). When the filament tip emerges through the hole, a clicking noise will be heard as the paper scrapes across the tip (be careful not to break the tip);

4.5- Once the filament tip is flush with the grid cap surface, back off the grid cap (CCW) one full turn. Recenter the filament with the gun centering screws.

5) Evacuate the gun chamber:

5.1- Check that O-ring and rim of gun chamber housing are clean. Close the gun chamber by pulling the housing forward;

5.2- On the vacuum panel, with the key in "MAN", turn on Pump 1 ("AUX") and then open valve V5. This opens the auxiliary pumping line to the gun chamber for evacuation. Turn the meter readout switch on the left to G3 (Gun) and monitor the gun chamber pressure. The pressure should drop fairly rapidly to below 100 microns.

(If the pressure does not drop below 50 microns after 5 minutes then the gun O-ring and housing surface should be cleaned. To do this close V5, turn the AUX P1 pump off, and toggle V7 to vent the gun chamber (as in 2.4 above). After the V7 vent stops, open gun housing and clean flat surface against which the O-ring rests (use alcohol). Clean O-ring by gently rubbing with clean finger/glove. Return to step 5.1.)

When the pressure drops below <u>50</u> microns close V5, turn off Pump 1, and open the gun isolation valve (V3) by rotating the handle 90° CW to a vertical position. <u>Do not mix up the above sequence</u>- close V5, P1 off, V3 open;

5.3- Return vacuum key to Auto.

6) Center and saturate new filament:

6.1- Turn gun power on and turn voltage up with pushbuttons to 15 kV (or desired voltage);

6.2- Be sure that BEAM BLANK button is not lit. If it is lit and is under computer control, exit the program with a "CTRL P" and note the line number printed before the "READY" message (you will re-enter the program at this line after the filament is replaced). Enter the command: <u>CALL (7,0,0,0)</u> -this will unblank the beam;

6.3- Turn Signal Selector knob to BC and select 0.1 x 10^{-6} range for the Picoammeter. Turn Filament Voltage knob up until emission current starts to increase (~ 500 on the dial). Let the filament stabilize for a couple of minutes with the emission current reading approximately 50 to 100 microamps (the emission current will increase slightly as the filament warms up);

6.4- As you turn the filament voltage higher, a beam current signal should appear on the Picoammeter. Continue centering the filament (with the gun centering screws), maximizing the beam current signal, and re-saturating (usually by increasing the filament voltage) until you have reached the saturation plateau, as with the normal filament saturation procedures (be sure you have gone over the "false" peak). Saturation and centering of a new filament usually takes several iterations. The filament current meter will read 1.8 - 2.0 amps, no higher. New filament saturation will usually have a filament voltage dial reading of 600 - 640. A properly installed and centered filament will have a saturation profile similar to that shown in Figure 5.

6.5- After saturating the filament, adjust the emission current to 150 microamps using the gun BIAS knob. Then, adjust the beam current to the desired value (usually .1 microamps) using the Condenser lens (C2) Coarse and/or Fine adjust knobs;

6.6- Check centering and saturation of new filament about every half hour. Record filament change in logbook and leave the old filament on the counter in the recycling box.

6.7- If you exited program earlier with "CTRL P" you can return to the same point in program by going to the line number where you exited (the line number is printed before the "READY" message after you type "CTRL P"), e.g.-

```
CTRL P

270 READY
GOTO 270
```

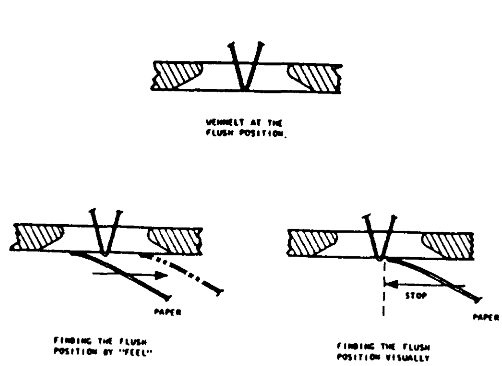


Fig. 4 - Filament installation.

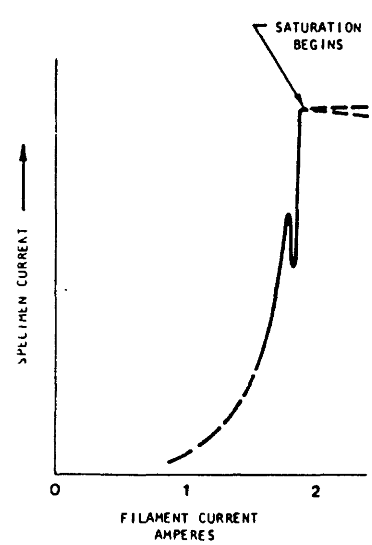


Fig. 5 - "Typical" filament saturation profile.

Shut-down Procedures

At the end of your operating shift:

1) Unblank beam (push button out). If beam blank button stays on, run the "FREE" program.

2) Place Digital Drives for scanners and stage (total of 3) in Man.

3) Lower elevator (push ELEVATOR DOWN button until it lights). Lower Z axis until it is down (the "Sample Stage Down" light on the vacuum panel will light up)- Z axis dial will be at about 324. *

4) (Skip this step if probe is to be used immediately following your shift): Turn filament saturation knob down until Emission Current meter reads 100 microamps.

5) Run the SDATA program to generate table for logbook.

6) Remove disks from RX02 disk drive. Close disk drive doors.

7) Turn computer power off ("AUX ON/OFF" to OFF).

8) Turn probe viewing optics illumination dial off (counter-clockwise). Transmitted light intensity should be off.

9) Fill out log sheet; see next page (include SDATA printout).

10) Turn display intensity down on TN-1710 EDS.

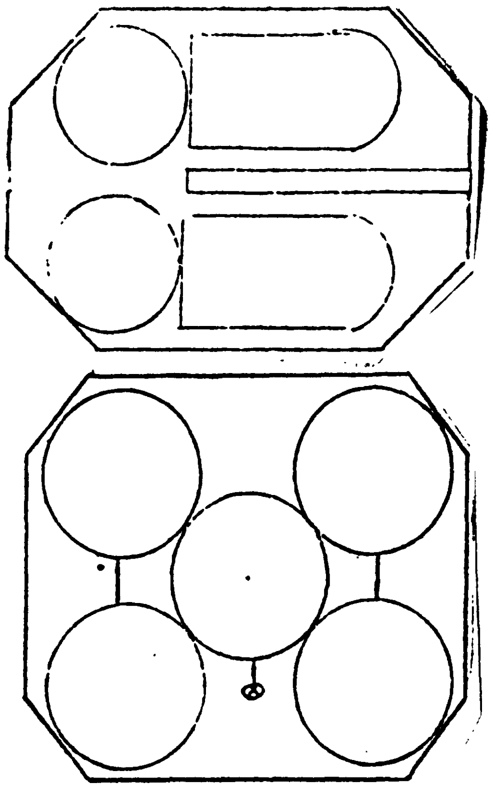
*NOTE - leave your samples in the sample chamber until the next operator changes samples. This is better for the vacuum system (and it saves you time too!). If you need to remove samples, follow sample loading instructions on page 4.

SEMQ OPERATOR'S LOG

NAME: ______________________ DATE: ____________ Start Time: _____

Vacuum (G4)-____x10⁻__ Beam Current-____µamps Voltage- ____kV Filament Dial- _____

[STANDARD/SAMPLE LOCATIONS]



Scanner	Crystal (circle one)
1	ADP - LIF
2	TAP - RAP - PbSD
3	LIF - PET

Standard Analysis ("known unknown"- Avg. of 3+ points

Std. Name - ______________________

Element/oxide	Wt. %
______	______
______	______
______	______
______	______
______	______
______	______
______	______
______	______
______	______
______	______
______	______
______	______
Total -------->	______

*FILE NAME ________

RUN COMMENTS: ____

____

____

Total # of analyses (standard/unknown): ____/____ End Time - __________

(place standardization table here)

TROUBLESHOOTING & ERROR RECOVERY

1) Error Messages-

Error messages generated by the computer programs during operation are listed on pages 42-44 and fall into three categories. These are ARL Extended Basic (ARLEB) operating system error messages, Applied Research Laboratories (1981):

a) Fatal errors - A fatal error causes the program to halt, print an error code, and leave the computer in the "READY" state. You usually will not encounter fatal errors but if you do, check the code to determine the cause. Correct the problem, which may involve actions such as "packing" disks, inserting the proper disk, or spelling correctly, and re-run the program.

b) Non-fatal errors- Non-fatal errors, such as dividing by zero or entering a comma when only one input is requested, will not cause the program to halt but will cause an input prompt to be repeated or some absurd numbers (like 0's or large positive or negative numbers) to be printed out. Improper use of commas (Error 122) and a blanked beam/ burned-out filament (Error 125) are common causes of these errors.

c) Disk (mass storage) errors - These occur during disk read/write operations and usually are fatal (program operation stops). These errors commonly are the result of typo's or improper drive designation (Error 8) or lack of FREE space on your disk (Error 4).

2) Program operation errors-

"DRIVE ERROR A1" message - The stage or scanner drives are hitting limits, causing that drive to lock and be inoperable. Place the stage or scanner Digital Drive in "Man", back off the drive by hand approx. 20 units and recalibrate the scaler for that drive. Re-enter program at line number printed out when error occurred. If stage repeatedly hits limit, sample may have to be re-oriented.

"PEAK NOT FOUND .." message - First check that your beam spot is centered at the crosshairs (you may have to unblank the beam) and that you are on the correct standard. If these two conditions are not met, correct and repeat the peak seek procedure. Other causes of a failed peak seek are:

- P-10 gas out (check flowmeter); this only effects scanner #2 (or any spectrometer with a flow-proportional detector);

- Scanner is at an upper (>37000) or lower (<11000) drive limit. Back off 100 units, recalibrate scaler, and repeat peak seek;

- If the failure occurred on a crystal that was just flipped, check that the alignment settings are correct. Crystal may not have been flipped properly. Repeat Crystal Flipping procedure and then repeat peak seek;

- If the standard has a low concentration, there just may not be a high enough count rate for the peak seek routine to function properly (<100 cps). To solve this problem do one of the following-

a. Use a nominal peak position (the last peak found is usually the best) which will be used in place of the peak search; or

b. Use a different standard for the peak seek and do not peak seek during standardization. This option must be chosen in advance, when the *FILE is being set up. With this option, you locate the peak position using the $PEAK program and use that position instead of locating the peak during $STDZ.

c. Crank up the beam current by about a factor of 2 during the peak seek routine (using the C2 current adjust knobs) and then reject ("R") the first counts. This allows the peak to be found without including an unusually high count rate in the average. Return the beam current to its proper value before proceeding to the next point (you will have to unblank the beam to do this). This entire procedure is a bit tricky and is not recommended for the novice.

d. Choose an entirely different standard with higher concentration (this has to be done in the *FILE set-up routine).

Low Beam Current - The beam current can be adjusted with the C2 condenser Coarse and Fine adjust knobs. If the beam current suddenly drops to very low levels while you are still adjusting it, check that the C2 Coarse knob is still in the 350-450 range. The current has a "crossover" at low dial readings and should not be left in this position.

Digital Drives "locked" - If the programs halt due to an error, the Digital Drives will be locked in Remote. To unlock them, enter the following commands to the computer:

CALL (1,XXX) - unlocks the Stage drive *

CALL (9,XXX) - unlocks the Scanner drives *

After entering these commands, you will be able to place the Drives in Manual.

Beam Blanked - If the program halts due to an error, the beam may be left blanked. To unblank the beam enter the following command:

CALL (7,0,0,0) *

Joystick won't respond - Check that X, Y, Z axes are not near limits. If they are at limits, put Digital Drive in MAN, back off, re-calibrate Display and return to REMOTE. If drives are not at limit, be sure the Digital Drive is in REMOTE. If joystick still does not respond, enter the following command (which enables the joystick):

CALL (12,J)

Cannot create a Data file or write on a disk (usually causes "Disk Error 4" message) -

INDEX disk to see if any FREE space is left. PACK disk if amount of CLEAR space is greater than 0. Delete unwanted files (see "CLEAR" command, page 50) or squeeze data files together (see "PACK" and "DSQZE" programs, pages 49-51).

Power Failure Recovery -If there is a power failure, the Main Gate valve (V2) will close and all of the Digital Displays will lose calibration. If the power failure is of short duration (a few seconds to a minute or so), operation can be resumed by normal start-up procedures (calibrating the displays, checking the filament, starting the computer). The "FREE" program should be run to re-set the computer-controlled devices. Occasionally, the filament will burn out when the power returns, due to the surge in voltage. (If you have time to react, turn the Filament Voltage to 000 when the power failure occurs and then turn it back up after power returns, or turn the Gun power off and the high voltage button to 0 until power returns.) If the Main Gate (V2) does not re-open after power returns, proceed to the vacuum problem section, pages 45-47.

---

*NOTE - the "FREE" program contains these commands and will produce the same results. However, use of the "CALL" commands will not eliminate the original program from memory and you may be able to resume program operation by returning to the line where the program halted by using GOTO line#.

FATAL ERRORS

A program will halt after printing an error message. Printout format is: ERROR NNN LINE XXXX, where NNN is the error number, and XXXX is the line number.

ERROR CODE	MEANING
0	User storage overflow
1	Unrecognizable statement
2	Illegal GOTO or GOSUB, e.g., attempt to branch to statement number which does not exist in the program.
3	Illegal character terminating a statement (usually caused by an ill-formed statement which causes the statement operation to end prematurely)
4	RETURN without corresponding GOSUB
5	Badly formed subscript
6	Subscript not in range (0 to 255), or exceeds maximum set by program
7	Mismatched parentheses
8	Illegal LET
9	Illegal relational operator in IF
10	Illegal IF
11	Illegal PRINT
12	Input line too long (exceeds 132 characters)
13	Bad dimension in DIM statement
14	Not storage for the array
15	Badly formed DEF statement

p. 42-44 from Applied Research Laboratories (1981)

16	Illegal line number or dimension value
17	DIM of previously declared or used item. It is illegal to dimension any item which has been previously used in any way unless the variable has been killed.
18	Bad variable in INPUT list
19	Bad variable in READ list
20	Out of data in READ list
21	Bad DATA statement format
22	Illegal FOR statement
23	No NEXT matching the FOR statement
24	NEXT without FOR statement
25	Unmatched quotes in statement
26	Called subfunction is not loaded, or EXF function improperly set up (bad linkage in location 50).
27	Ill-formed expression (probably missing exponent on E format number)
28	Bad call to EXF: either the subfunction number called does not exist, or the statement is in illegal format.
29	Non-existent device or memory location access. For example, an attempt has been made to boot a disk which expects to find an instrument on the system, or to boot a disk requiring 28K in a 16K system.
30	Bad output format statement

31 Hardware/software diagnostic error. This error is preceded by one of three outputs:

a. ADDRESSING XXXXXX
ERROR 31 LINE XXXX

b. PARITY XXXXXX
ERROR 31 LINE XXXX

c. INSTRUCTION XXXXXX
ERROR 31 LINE XXXX

where XXXXXX is the contents of the program counter when the error was detected. If it is a parity error, try rebooting the system; if an addressing or instruction error, the problem is most likely with an assembly language instruction. This error indicates that there is a problem either within the BASIC interpreter or with the subfunction currently in memory. Refer this problem to ARL.

NON-FATAL ERRORS

A program may continue to RUN after an error message is printed. Printout format is the same as for the previous list of fatal errors.

ERROR CODE	MEANING
119	String variable overflow
120	Illegal characters on input
121	Not enough data typed to INPUT
122	Too much data typed to INPUT or called for by FETCH/FILE
123	Non-existent variable
124 **	Number too large to fix (probably a subscript combination out of range)
125 **	Divide/multiply overflow or underflow
126 **	Square root of negative number
127 **	Logarithm of zero or negative number exponential overflow

** A zero result is assumed

MASS STORAGE ERRORS

These errors concern the disk and the immediate disk environment, and are always FATAL. The format of the error is:

DISK ERROR N DRIVE X LINE XXXX

where 'N' indicates the type of error, 'X' is the drive number on which the error occurred, and 'XXXX' is the line number on which the command is located which generated the action.

ERROR CODE	MEANING
0	Illegal disk address
1	Bad read - may indicate the disk is faulty. Use the utility CHECK to test the condition of recording surface.
2	Parity error - bad data transmission. For the RX01 disk unit only.
3	Library file full. An attempt has been made to exceed the maximum number of entries for that device.
4	Disk is full. There is not enough room to store requested file.
5	Non-existent data record requested or not enough program space for chained module.
6	Disk unit is not ready: no power, door open, disk not in, disk drive malfunctioning, drive write protected (where applicable). The error is displayed as:

DISK STATUS XXXXXX
DISK ERROR 6 DRIVE N LINE XXXX

As this particular error describes the condition of the hardware, the special message 'DISK STATUS' will be printed. The 6 digits which follow indicate the kind of hardware error that has occurred. Refer to the appropriate hardware device manual for detailed information for the particular error code.

RX01 - D.E.C. part number EK-ORX01-OP
RX02 - D.E.C. part number EK-RX02-UG-001
RL01 - D.E.C. part number EK-RL01-UG-002

7	The drive requested does not exist.
8	Requested file either not found, or is the wrong file type.
9	Density error. This applies to the RX02 drives only.
10	An attempt has been made to access a non-BASIC disk.
11	When using the utility LATCH, either the file requested does not exist or is a data file.

VACUUM SYSTEM PROBLEMS

1. Problem: Sample chamber automatic pump cycle hung up- vacuum will not trigger completion of pumpdown cycle.

   Action: Follow procedures for "Overriding the automatic pumping of the sample chamber" on page 46.

2. Problem: Main Gate valve (V2) closed (also usually accompanied by automatic shut off of high voltages).

   The Main Gate valve will close for any of several reasons. To reopen the Main Gate it is first necessary to determine the cause of its closing and correct the problem (refer to vacuum panel):

   a) LN2 light out- This indicates that the nitrogen gas cylinder is empty or the pressure has dropped drastically below 25 psi. Change the gas cylinder or adjust the pressure (only if you know how). Proceed to section on "Main Chamber pumpdown", p. 47.

   b) Lens and/or Diffusion pump water lights out- a drop in water pressure causes this. If the Diffusion pump water is out, the Diffusion pump light will also be out and vacuum pressure may have risen considerably- proceed with caution (and only with prior experience). Sometimes a drop in outlet pressure is the culprit. Water gauge on the back wall should read 45-50 psi on either side of filter. Inlet pressure at probe should be 20 psi. Outlet pressure at probe should be 5.5 - 6.5 psi. The outlet pressure is adjusted at the wall by turning the black-handled flow valve on the "water out" flow line. If water LED's come back on, proceed to "Main Chamber pumpdown" section, p. 47. If LED's stay off, notify the authorities.

   c) Vacuum leak, causing one or more of the pressure gauges to rise (G4 will rise to $> 1 \times 10^{-3}$ and may shut off completely). Notify the authorities promptly.

   d) Power failure - If a power failure lasts longer than a few seconds, the Main Gate will close. After power returns, proceed to "Main Chamber pumpdown" section.

Overriding Automatic Sample Chamber Pumpdown-

During automatic pumpdown of the sample chamber, the SC pressure (G2) should decrease fairly rapidly from ATM to <100 microns (usually within 1 minute). Pump-down from 100 to the trip point pressure of 40 microns is a little slower (another 2 to 4 minutes). If the initial pumping stage to 100μm seems exceedingly slow, or if it appears to be hung up at some pressure greater than 100μm, there is a leak around the sample chamber "O"-ring seal. In such a case the automatic pumping sequence will go on indefinitely and so it must be interrupted and the O-ring must be cleaned.

To interrupt the automatic pumpdown, proceed as follows (it is IMPERATIVE to do each step in the proper sequence!!):

1. Turn Filament Voltage knob down to 000.

2. Turn Gun High Voltage to 0 (push buttons from 15 down to 0) and turn HV power off.

3. Turn Detector High Voltage "coarse" knob from 2000 down to 0 and turn the power switch off.

On the vacuum panel:

4. Put V2 switch down (closes main gate valve).

5. Put V1 switch down (closed).

6. Check that V4 and V5 switches are down (closed).

7. Check that P1 (Aux. pump) switch is down (off).

8. Turn key to MANUAL (you will hear valves closing).

9. Vent sample chamber by toggling the V6 N_2 vent switch. The V6 light will stay on until the sample chamber reaches atmospheric pressure.

10. When V6 light goes out, put the Raise/Lower toggle switch to "Lower." This drops the sample stage.

11. Pull stage out and run finger (either with clean gloves on or without gloves but with finger cleaned with alcohol) along O-ring. Small dust particles, hair, or fine cuts on the O-ring will cause leaks, so inspect and clean carefully.

12. Push stage in. Turn Raise/Lower toggle to "Raise" (chamber will raise up).

13. When "Sample Chamber Closed" light comes on, turn the AUX pump (Pump 1) on and open Roughing valve V4. Allow sample chamber to pump down to 40 microns (~ 5 minutes).

14. Close V4 valve; then turn Pump 1 off; open V1 valve (sample chamber). DO NOT MIX UP THIS SEQUENCE.

15. Open V2 (main gate) valve. If G1 pressure (foreline) goes above 100, close V2 and let pressure drop and then open V2 again. Wait for pressure gauges to reach normal operating conditions (G4 $\approx 5 \times 10^{-6}$; G1 $<$ 10 - 20 microns).

16. Turn vacuum key back to AUTO mode.

17. Turn Detector High Voltage on (2400 volts). Turn Gun High Voltage on. Select 15 kilovolts with push buttons. Turn filament up and saturate as usual.

<u>Main Chamber Pumpdown-</u>

If the Main Gate valve (V2) has been closed for an extended period of time, the main tank area is isolated from the foreline/diffusion pump vacuum system and the vacuum in the main tank will not be maintained. If the pressure indicated on the SC (G2) gauge is less than 60 microns, then the Main Gate valve can be "burped" open as in item 15 of the previous section. If the SC pressure is greater than 60 microns (and V1 is open) then the following procedure must be followed prior to opening the Main Gate (V2):

1. Check that all toggles on vacuum panel agree with status lights on the vacuum schematic. Pay particular attention to V2 valve. If the V2 indicator light is out, the V2 toggle should be placed in its down position.

2. Turn vacuum key to Manual.

3. With the V2 gate closed and V1 opened, toggle the V6 vent line (toggle is spring loaded and releases automatically). This will bleed nitrogen into the main tank (G2 and G3 will go to ATM, G4 will turn off-deflecting completely to the left). V6 light will go out when venting is completed (2-3 minutes).

4. When V6 light is out, evacuate the Main Tank by doing the following:

   a. Turn on Pump 1 (AUX) and open V4 (Roughing) valve;

   b. Monitor SC (G2) pressure. Pump down until G2 $<$ 50 microns;

   c. Close V4, then turn off Pump 1 (AUX). DO NOT REVERSE THIS ORDER;

   d. "Burp" open the V2 valve as in item 15 of the previous section and continue with items 16 and 17 of the same section.

EMERGENCY SHUTDOWN PROCEDURES

(used only in the event of water or power outage)

-Notify the authorities (i.e., J. McGee or his substitute)

-Turn Filament Voltage knob to 000

-Turn Gun kV to 0 (with pushbuttons)

-Turn Gun HV Power OFF

-Turn Detector HV to 0 and turn power off

-Turn Lens Power Supply off (bottom of SEM console at floor level)

-Turn Scintallator HV, PM Detector, Sweep Driver power supplies off (bottom of SEM console at floor level)

-Run the FREE program

-Turn computer power off

-Put Digital Drives in manual mode (all 3)

-Turn DC power supply off (bottom of X-ray console at floor level)

-Turn Raster Size to Point (SEM console)

-Lower Elevator and Z axis until "Stage Down" lights up (as usual)

-Turn Illumination off (ccw)

-Close Main Gate valve (V2) by putting toggle down (leave vacuum key on AUTO)

-Turn Diffusion pump power off

-Turn Tracor Northern TN 1710 off

-Notify the authorities... (if you have not already)

OFFLINE SOFTWARE OPERATIONS / DATA PROCESSING

The DEC-MINC LSI 11-23 computer in the probe lab is an off-line analog of the SEMQ computer. The MINC can, and should, be used to perform software operations that do not require access to the microprobe instrumentation in an on-line mode. Actions such as file copying, disk packing, *FILE set-up, and any subsequent probe data manipulation/calculation can be performed with the MINC computer. The MINC has a single ON/OFF switch (red rocker switch) which, when turned on (up), boots the disk that is inserted in the left-hand drive. The "SEMQ USER UTILITIES" disk in the probe lab provides instructions for several of the available disk utilities. Several off-line data manipulation and plotting programs are available, courtesy of some of the probe users, and descriptions and operating instructions for these programs are contained in the Probe Applications Software binder.

Some of the offline software operations that are periodically needed in order to prepare for probe operation are described in the next few pages. The exact program names are given in parentheses following each heading.

Packing Disks- (PACK)

The ARL floppy disks are arranged into approximately 2000 records of space. When a disk is INDEXed, the location of each program is given by record number and the total amount of FREE (unused) and CLEAR (erased) records left on the disk are indicated at the bottom of the INDEX.

After repeated use of the analytical programs and frequent over-writing of files, the available "FREE" space on the disk may be inadequate to allow for a certain MOVE or STORE operation or for generation of *FILES. If the "FREE" space is inadequate you will receive a message stating such, or you will receive a "DISK ERROR 4" message. The first thing to do is INDEX the disk on which you were trying to write. If there is a large amount of "CLEAR" space and very little "FREE" space then you can "PACK" your disk in order to transform the CLEAR space into FREE space, which can then be used for storage of files:

```
                PACK
CLEAR ----------------> FREE -------> Write
```

To help prevent disk writing errors it is a good practice to PACK your disks prior to each analysis session. The procedure is as follows:

INDEX <cr>

(the catalog follows, showing the disk contents)

```
FREE    14
CLEAR  935
```

RUN PACK <cr>

PACK DRIVE - 0 <cr> (several second pause)

PACK DRIVE - <CTRL> P (or you may also PACK Drive 1)

READY

INDEX <cr>

(catalog here)

FREE 949
CLEAR 0

You will now be able to use all of this FREE space for writing new files or for copying. PACKing a disk does no harm to it and it is a good idea to get into the habit of periodically packing so as to avoid interrupting any of your disk writing operations due to lack of FREE space.

If you have very little FREE space left on your disk after packing it, you may not be able to create any new files on that disk. For example, if you cannot create a new Data file in the $ANBA program it is probably due to lack of FREE space on your Data disk, even after packing the disk (this particular Data file requires 101 FREE records). In such a case, you must delete unwanted files, consolidate your Data files (see below), or get a new formatted disk from me.

Erasing Files - (CLEAR)

To delete a file from a disk, use the CLEAR command, specifying the drive that the file is on, e.g.:

CLEAR *FILE,1

This command deletes the file "*FILE" from the disk in drive 1.

If you accidentally erase a file, it can be retrieved by running the "ERROR" utility program prior to "packing" the disk (see Applied Research Laboratories, 1981).

Initializing a Disk - (RENEW)

To erase an entire disk or initialize a disk for use with the ARLEB system, run the "RENEW" utility program. This program will erase the contents of the disk, so use care not to "RENEW" one of your data disks (see Applied Research Laboratories, 1981).

Consolidating Data Files- (DSQZE)

You will probably get to the point where you have generated numerous Data files during analytical runs and yet most of these files will be only partially filled (remember, they are automatically set up to store 100 analyses). You may want to free up unused reserved records so that you can use them for subsequent data filing. The DSQZE program retrieves unused records from data files and converts them into Free space. The program determines how many analyses are in the Data file and "squeezes" the file together on a separate data disk (you are allowed more than one), eliminating the unused records from the file. All you have to do is have a second data disk available, run the program, and follow the instructions in the program very carefully (it is your data!). To run the program, which is on your program disk, just enter the following:

RUN DSQZE <cr> (the program will instruct you the rest of the way)

Disk Copying - (COPY)

To copy the entire contents of a disk, first initialize a disk with the "RENEW" program (see previous page) and then run the "COPY" program on the User Utilities Disk.

File Copying- (MOVE) (NAME)

A Master Data File disk is available which contains all of the available *FILES, corresponding alpha-factor files ("AFILES"), and %FILES. Descriptions of the contents of the *FILES are contained in the "Standards" book. If you decide to use one of the *FILES for your analytical problem, you can copy the *FILE and corresponding AFILE onto your data disk by using the "MOVE" utility program on your program disk. To do this, type RUN MOVE and then remove the program disk from Drive 0 and insert the Master Data disk (your data disk should be in Drive 1). Respond to the program prompts with the underlined responses as follows:

(<cr> = RETURN)

RUN MOVE <cr>

<---------insert Master Data Disk

MOVE FROM DRIVE - 0 <cr>

TO DRIVE - 1 <cr>

FILE NAME - *FELD <cr>

FILE NAME - AFELD <cr>

(at this point, re-insert program disk in Drive 0)

FILE NAME - <CTRL> P (hold the "CTRL" key down while depressing the "P" key)

Copies of the *FELD and AFELD files are now on your data disk and ready to be used in the analytical programs.

You can also use the MOVE program to preserve the standardization data for your present *FILE prior to standardizing a new *FILE. When you standardize a *FILE, the peak positions and count data for each element are stored in the data file called "#STDZ" on your program disk. Anytime you standardize a *FILE with the $STDZ program, this #STDZ file is overwritten and the old standardization data is lost.

To preserve the old standardization, move the #STDZ file to your data disk (Drive 1) using the MOVE program as described above. After you move #STDZ to Drive 1, you can rename it to a name that you can associate with its corresponding *FILE. The NAME program on your program disk is used for this. An example is given below for renaming the #STDZ file containing standardization data for *FELD:

```
RUN NAME  <cr>

 NAME DRIVE - 1   <cr>

 OLD FILE NAME - #STDZ    <cr>

 NEW FILE NAME - #SFEL    <cr>          (* see NOTE)

 DAY - <cr>

 OLD FILE NAME - <CTRL> P

READY
```

*NOTE - Names for files (both data and program) have a maximum length of 5 characters and must not begin with a numeric character.

If at a later time you wish to re-use the standardization data for *FELD (now stored in #SFEL on Drive 1), the procedure described above would be reversed; first rename #SFEL to #STDZ and then MOVE it back to your program disk, where it will overwrite the current #STDZ file.

GENERATING NEW *FILES

If none of the available *FILES are adequate for your analytical problem, feel free to generate a new *FILE using the "$QSET" program on your program disk. Examples of running $QSET are given in the ARLEB software manual (chapter 4) but some of the program prompts have been modified. The program is generally self-instructive and the format of your input will be obvious when you run the program. Before generating a *FILE, check that no other *FILE exists with the name you plan to use. Once you have generated a file that works satisfactorily, provide a copy of it to the lab so that it may be included in the list of available files.

If you examine the contents of a *FILE as listed with the "QLIST" program, all of the information needed to generate a *FILE is printed out. You will need to have %FILES available (these contain concentration and positioning data for the individual standards and use the 4 letter acronyms found on page 57). The %FILES are generated using the "$SSET" program but you should not have to use this program since %FILES for most standards are already available on the Master Data disk. If you do need to generate %FILES, be extremely careful since the entire data correction procedure is based upon the input of correct standard concentrations. If you generate these files offline (on the MINC) you will not be able to store stage positions and the standard will have to be manually located in the probe during standardization.

When you are ready to run $QSET and generate a *FILE you should have the following information at hand for each element:

Element symbol; X-ray line;

Spectrometer #; Peak seek increment (if scanner);

Background measuring method (if scanner, you can choose offset or "blank" standard);

Standard names (%____) for background and for peak counts

Use the table on the next page as a set-up sheet for this information. With this information in hand you will be able to generate a *FILE (and a corresponding AFILE containing alpha factors). At one point in the program you will be requested to temporarily insert a "Bence-Albee Master Alpha-factor disk" in place of your data disk. Just follow the directions. The Bence-Albee Master disk, labeled as such, is in the blue diskette box in the SEMQ lab. If you are going to use MAGIC correction procedures, this disk is not required.

When naming your *FILES, do not use a name that already exists on the Master Data Disk. Even modified files for the same mineral type should have a new name, e.g. if *FELD is modified, the new file could be called *FEL2. Also, the last four characters of the *FILE and corresponding AFILE should match, e.g. *FELD and AFELD. The $ANBA analysis program can analyze for a maximum of 14 elements, so the *FILE must be limited to this number. If you must analyze for more than 14 elements, you will have to use one of the slower analytical programs ($ANLZ, $MGPR, $SST).

Note that there is a separate Data Disk available that contains all of the %FILES, *FILES, and AFILES for sulfides.

*FILE SET-UP

* ________

	El.	Line	Spec.	Peak seek Increment	Bkgd offsets Hi	Lo	Peak std	Bkgd Std
1.								
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.								
11.								
12.								
13.								
14.								

kV= _____   Correction: a) B-A Oxide   b) B-A Element   c) MAGIC

CRYSTALS

Spec. 1= ________

Spec. 2= ________

Spec. 3= ________

FIXED SPECTROMETERS

Spec. #		Element
4	-	Na
5	-	Mn
6	-	Ca
7	-	Mg
8	-	Fe
9	-	Si

Data Examination/Manipulation Programs

There are several programs that can be run on the off-line Minc LSI 11-23 computer system that provide various data evaluation, recalculation, and plotting functions. The programs vary in the operating system and program language they use, as well as in degree of sophistication and flexibility. A brief description of some of these programs follows. Programs with an (*) have descriptions/operating manuals of their own:

DETEK	Calculates detectability limits for elements measured on the probe.
OFFPT	Offline plotting program (general).
DPRAV	Lists and averages analyses from probe data files.
% LIST	Lists contents of %FILE (standard data).
QLIST	Lists contents of *FILE (analytical packages).
OLAPS	Lists locations of potential overlapping peaks for a selected element and wavelength.
MINCLC*	Recalculates mineral analyses (general, with emphasis on pyroxenes).
RDARL4*	Data transfer/reformating utility program.
TABLE*	Tabulates analyses in columnar format with option to re-order element list.
RECAMP*	Amphibole recalculation program.
(AMPHI, MICA, FELD, GARN, etc.)*	Cation Site Occupancy/Plotting Programs

Date File Format for Storage of Analyses Generated in $ANBA

If the option to file data is chosen, $ANBA builds a 101 record data file with a 5 character (maximum) name chosen by the operator (this file is automatically built on the disk in drive 1). The first record of the data file contains general information pertaining to the analysis session. Records 2 thru 101 are used for storage of selected analyses (1 per record). If 100 analyses are not stored in one session, the operator may continue to store data in the same file during the next session, as long as the same *FILE is used for obtaining the analyses (this ensures correspondence of element symbols and standards to the stored analyses). The storage format of information in the data file is as follows:

RECORD	ENTRY	VARIABLE	DESCRIPTION
1	1-4	T4(0-3)	Operator name & mineral type
	5-7	T4(14-16)	Date
	8	F4	QDATA (*FILE) Filename
	9	G1	Datafile name
	10	R1	Data reduction method (=2 for B-A)
	11	E0	# of elements analyzed
	12	L	Oxide flag (1=oxide, 0=element)
	13	E1	# of analyses in this file
	14	E2	Counter for storage location
	15-28	D2	Element symbol & measured X-Ray line (up to 14 elements)
	29-42	S7	Standard (%File) filename for each element
2 thru 101	1-8	T3(0-7)	Sample label
	9	D1(0)	X stage position
	10	D1(1)	Y stage position
	11	A+X3	Combined flag equalling # of analyses in average + standard/unknown analysis indicator
	12-25	W(0-13)	Corrected weight %
	26	T0	Total weight %
	27-40	D(0-13)	Formula (cations)
	41	H1	Total cations
	42	X0	# of oxygens for formula calculation

Microprobe Standards

(* = suggested working standards [known-unknowns])

Amphibole

	AMCM	Cummingtonite
	AMEN	Engel's Amphibole
	AMKF	Potassic fluor-richterite
*	AMKH	Kakanui Hornblende
	AMMN	Minnesota Hornblende
*	AMSF	Sodic fluor-richterite
	AMTG	Tremolite, G-21 Gouveneur

Apatite

	APCL	Synthetic Chlorapatite
*	APFD	Durango Fluor-apatite
	APRE	Synthetic REE-apatite
	APSF	Synthetic Fluorapatite

Carbonate

	CCNM	Calcite, National Museum
	CDAS	Dolomite, Austria
	CRAP	Rhodochrosite
	CSBH	Siderite, Broken Hill
	CSTR	Strontianite

Feldspar

	FSBO	Benson Orthoclase
*	FSLC	Lake County Plagioclase
	FSNA	Nunivak Anorthoclase
	FSTA	Tiburon Albite

Glass

	GFAB	Albite Glass
	GFAN	Anorthite Glass
	GFOR	Orthoclase Glass
	GLBA	Barium Glass
	GLDI	Diopside Glass
*	GLJF	Basaltic Glass VG2 Juan de Fuca
	GLL1	Lunar Glass (61156)
	GLL7	Lunar Glass (77135)
	GLL8	Lunar Glass (68415,85)
*	GLMP	Basaltic Glass VGA99
	GRE1	REE 1
	GRE2	REE 2
	GRE3	REE 3
	GRE4	REE 4
	GRLS	Rhyolite Glass (Smith)

Olivine

	OLCR	Cobalt Olivine (Robie)
*	OLMJ	Marjahlati Olivine
	OLNI	Nickel Olivine
	OLRF	Rockport Fayalite
*	OLSC	San Carlos Olivine
	OLSF	Synthetic Fayalite
	OLSW	Olivine USNM 2566 (Springwater)
	OLST	Synthetic Tephroite

Oxide

	OXBU	Bushveld Chromite
	OXCO	Corundum
*	OXGH	Brazil Gahnite
	OXHA	Hausmannite
*	OXIL	Ilmen Ilmenite
	OXMN	Manganosite
	OXMT	Magnetite
	OXPE	Periclase
	OXPA	Partridgeite
	OXQZ	Brazil Quartz
	OXR1	$Gd_2Mo_3O_{12}$
	OXR2	$GdDyMo_3O_{12}$
	OXR3	$YNbO_4$
	OXR4	$LaNbO_4$
	OXSP	Synthetic Spinel
	OXTB	Tiebaghi Chromite
	OXUB	Chromite MB-5
*	OXRU	Rutile
	OXVA	Vanadium Oxide (V_2O_3)

Pyroxene

	PXA6	DL6 Augite
	PXAD	Adirondack Diopside
	PXBH	Rhodonite Broken Hill
	PXBK	Rhodonite Bald Knob
	PXEN	Synthetic Enstatite
*	PXHD	Hedenbergite M12330
	PXHI	Homedale Pyroxmangite
*	PXHY	Hypersthene R2467
*	PXJT	Johnstown Hypersthene
*	PXKA	Kakanui Augite
*	PXP1	Chrome Augite (PSU Px-1)
	PXPS	PSU Diopside
	PXSD	Synthetic Diopside
	PXWO	Mono Co. Calif. Wollastonite

Garnet

* GTKN	Kakanui Pyrope
GTRV	Roberts Victor Garnet

Mica

MBLM	Lemhi Biotite
MBPS	PSU Biotite 5-112
MBST	Stillwater Biotite
* MFPH	Fluorphlogopite
MMMT	Methuen Muscovite
MPAV	Paragonite
MPBO	Phlogopite

Miscellaneous

ANDB	Andalusite, Brazil
KYMG	Kyanite, Minas Gerais
KYPS	Kyanite, Penn State
PYNC	Pyrophyllite, Staley, NC
SCMB	Scapolite R6600
* SPHC	Hemet Sphene
STBM	Staurolite, Berkshire, MA
TPTM	Fluor-topaz, Topaz Mt.
TSLP	Tourmaline, Mexico
* ZOPC	Zoisite, Puerto Cabello

Analytical Procedures and Publications

In any publication containing, or based upon, extensive amounts of microprobe data, you should include a short paragraph describing the analytical techniques and operating conditions utilized. The amount of detail to include in such a description is up to the author, the nature of the publication (and its potential audience), and the amount of analytical complexity. The information which should be included is given in the first grouping below. The second group of items are more or less optional, depending on the amount of detail desired:

Instrument type	- (automated ARL-SEMQ 9 channel electron microprobe)
Operating voltage	- (15 kV)
Beam Current	- (0.1 microamps)
Correction procedures	- (Bence & Albee (1968) + Albee & Ray (1970), MAGIC (Colby, 1968))
Standard types	- (natural or synthetic silicates, oxides, etc.)

Calibration standard (known-unknown)	- (Kakanui pyrope, USNM ____, etc.)
Data collection procedures/automation	- (ARL (1979), McGee (1983), etc.)
Counting times	
Spot size	
Traverse step increment	
Sampled population	- number of samples, grains, analyses, or some indication of the statistical significance of the sampled population

Calibration of Energy Dispersive Analyzer (EDA):

The Tracor Northern 1710 energy dispersive analyzer measures characteristic X-rays of elements with Z $\geq$11 (Na) simultaneously and displays the results as an energy spectrum, arranged from left to right in order of increasing energy. This feature allows quick identification of all elements (Z $\geq$11) present at concentration levels of 0.5 - 1.0 wt. % or higher. As an aid for choosing analysis points, a 10-30 second spectrum can be acquired and the elements identified before analyzing a selected grain (the beam must be unblanked).

The EDA needs to be calibrated if it has been turned off or if a power fluctuation has caused it to lose calibration (Marker lines do not function properly). To recalibrate (refer to Figure 6):

1. Turn 1710 power on (if not on already). Turn off all colored buttons, if lit, by depressing them once.

2. Push the orange set-up button and set LT (live time) to desired number of seconds (10 or 15 is adequate) using the OMNI knob. Push the orange button to lock in the pre-set values (light will go out). Push Auto Range button (it should be lit- this allows the acquired spectrum to be visible).

3. Acquire a spectrum (press red CLEAR DATA and ACQUIRE buttons) in order to calibrate the peak positions. Choose something with peaks you recognize and that has distinct peaks at both high and low energies. Something which contains Fe and Si is a good choice.

4. Turn off the "Markers" - the green button should not be lit and the toggle should be de-activated.

5. Set the BUG (use OMNI knob) on the peak of the high energy (right-hand side) element; push CALIBRATOR toggle to HIGH.

6. Determine the energy value of that element using the average Kα scale on the cardboard "slide rule." Set the "BUG" entry (in upper left corner of screen to that value using the OMNI knob (use the < or > buttons to set the decimal point).

7. Turn the calibrate toggle switch off.

8. Move the bug to the low energy (left-hand side) peak, set the CALIBRATOR toggle to LOW and repeat steps 6 and 7.

9. The EDA should now be calibrated and is ready to use. Acquire a spectrum and then you can identify the elements present by activating the MARKER module (push toggle to SELECT ELEMENT). Move the element marker lines to the desired peak or element using the OMNI or "<" and ">" buttons just below the OMNI.

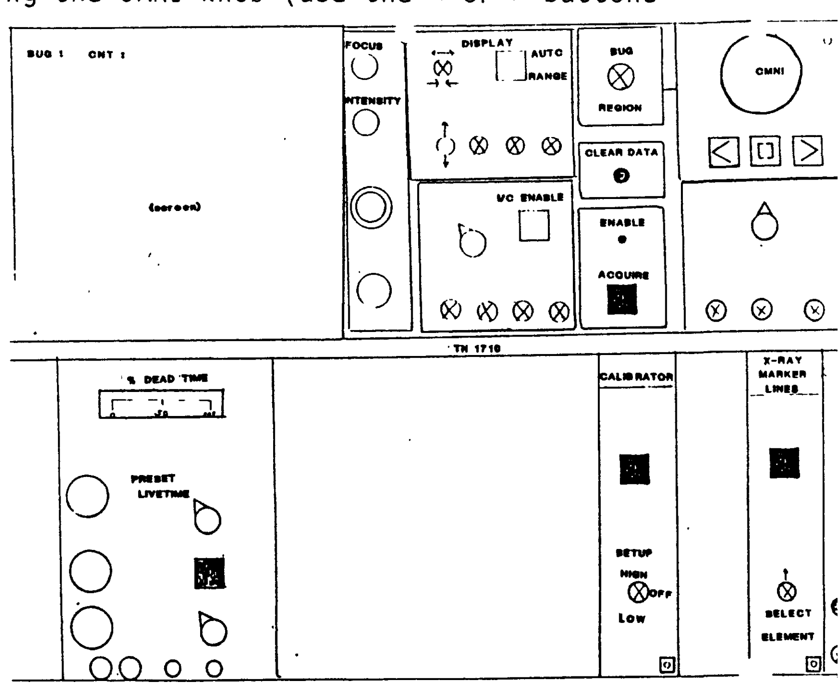


Fig. 6 - EDA front panel

Instructions for Automatic Carbon-Coating (using the Denton 515 Evaporator)

Your samples should be photographed and cleaned prior to carbon coating. Clean samples thoroughly using kimwipes and ethyl or isopropyl alcohol, followed by ultrasonic cleaning for 15-30 seconds in "Freon" cleaner. Do not get fingerprints on the sample surface. When samples are clean and dry, load in carbon coater and coat as follows:

1. Raise bell jar and load samples. Include small piece of white paper to gauge carbon thickness.

2. Remove outer carbon rod (loosen screw on top, loosen screw holding pressure spring, poke carbon out). Neck down rod approx 1/8 in. with the carbon sharpener. Check that opposing rod is flat (use grit paper). Replace rod, protruding so that clip provides pressure to rod. Tighten screws.

3. Lower bell jar (GENTLY).

4. Check that all 6 lower switches are OFF or CLOSED. Turn rotary speed to Auto, Manual/Auto Selector and Manual/Auto Flash to Auto. Set Flash Power Adjust dial to 48-52. Set timer (black dial to 10 minutes).

5. Turn power on.
   Turn Vent/Operate switch to operate. Hold bell jar down with a slight pressure for about 10 seconds.

   System will pump for selected time (~ 10 minutes)

   After this time, the flash power and rotator will come on for 20 seconds. Then the carbon rods will evaporate for ~ 30 seconds.

6. After the END OF CYCLE light comes on, turn vent/operate to vent. The diffusion pump will go off immediately and the system will vent to air after 3 minutes. Wait for venting to stop completely.

   Raise bell jar and remove samples. Lower bell jar again (gently).

   Turn Power off.

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