

**IMPROVE
STANDARD OPERATING PROCEDURES**

**SOP 326
PIXE and PESA Analysis**

Date Last Modified	Modified by:
10/24/96	EAR
12/9/96	EAR
2/5/97	RAE

TABLE OF CONTENTS

1.0 PURPOSE AND APPLICABILITY 5

2.0 RESPONSIBILITIES 6

 2.1 Spectroscopist 6

 2.2 Quality Assurance Manager 6

 2.3 Computer System Operation Manager 6

3.0 REQUIRED EQUIPMENT AND MATERIALS 7

 3.1 PIXE System 7

 3.2 PESA System 7

 3.3 Calibration Requirements 7

 3.4 Analysis Requirements 7

4.0 METHODS 8

 4.1 Overview of the PIXE/PESA System 8

 4.2 Preparation for Analysis—North Cave. 10

 4.2.1 Filling the Detector. 11

 4.2.2 Installing the Detectors. 12

 4.2.3 Beam Collimation System 13

 4.2.4 PIXE/PESA Vacuum System 15

 4.2.4.1 Vacuum system setup procedures 16

 4.2.5 Wiring and Powering PIXE Equipment in the North Cave. 18

 4.2.5.1 Connecting the PIXE Detectors to Power. 18

 4.2.5.2 PIXE Detector Pre-Amp to Amp Wiring. 19

 4.2.5.3 PIXE Amp Output Wiring. 19

 4.2.5.4 Connecting the PESA Detector to Power. 22

 4.2.5.5 PESA Detector Wiring. 22

 4.2.5.4 Cyclotron Operations 23

 4.2.5.5 Count Rate Meter 24

 4.2.5.6 Proton Beam Current 24

 4.2.5.7 Slide Changer. 25

 4.2.5.8 Remote Monitoring System. 26

 4.2.6 North Cave Electronics Settings 27

 4.2.7 Biasing the SiLi Detector 27

 4.3 Preparation for Analysis—Computer Room. 28

 4.3.1 Counting Room Electronics Connections 30

 4.3.2 Counting Room Electronics Settings 34

 4.3.3 Biasing the PESA detector 35

 4.3.4 Acquisition Module Wiring 36

 4.3.5 Manual Slide Control 37

 4.4 Data Acquisition (Program ACE)..... 40

 4.4.1 Logging On and Logging Off of ACE 40

 4.4.2 ACE Keypad Commands..... 41

 4.4.3 Default ACE Settings..... 44

4.4.4 ACE Usage Examples	45
4.4.4 Trayfiles.....	45
4.5 Spectral Analysis (Program RACE)	50
4.5.1 Logging onto RACE.....	51
4.5.2 Setting RACE Analysis Parameters	55
4.5.3 RACE Calibration and Matrix Tables	57
4.6 Routine Analysis.....	67
4.6.1 RENO Calculation and System Calibration.....	67
4.6.2 Blank Filter Analysis	67
4.6.3 Reanalysis Trays	67
4.6.4 Filter Sample Analysis.....	67
4.6.5 Data Storage.....	68

LIST OF FIGURES

Figure 1 PIXE and PESA Filter Analysis Area.....	9
Figure 2 CNL Cyclotron and North Cave.....	10
Figure 3 HV Power Supply.....	12
Figure 4 PIXE Collimators	14
Figure 5 Target Collimator	14
Figure 6 Vacuum System.....	16
Figure 7 PIXE Bias Supplies.....	18
Figure 8 PIXE Amp Wiring	19
Figure 9 Front and Rear of PIXE Amp	19
Figure 10 PIXE System Amp Output Wiring	21
Figure 11 North Cave Patch Panel	22
Figure 12 PESA Output Wiring	23
Figure 13 PESA Patch Panel in the North Cave	23
Figure 14 BNC 161 Junction Box Location	24
Figure 15 PIXE & PESA System Changer Card	26
Figure 16 Computer Room Schematic	28
Figure 17 PIXE & PESA System Control Schematic	29
Figure 18 Rack K PIXE Equipment.....	32
Figure 19 Rack M PIXE Components.....	32
Figure 20 Rack L PIXE Components.....	33
Figure 21 Rack N PIXE Components	33
Figure 22 Rack M, PESA Components.....	34
Figure 23 Rack K, PESA Detector Bias.....	36
Figure 24 PIXE-1 Data Acquisition Module Wiring Schematic	36
Figure 25 CNL/ARB Controller.....	37
Figure 26 ACE Control Program Screen.....	42
Figure 27 ACE Program Keypad Mapping.....	42
Figure 28 RACE Analysis Sample Output.....	54
Figure 29 RACE Analysis Parameter Input Screen.....	55

Figure 30 PIXE-1 Detector CuS Standard Full Output	60
Figure 31 PIXE-2 Detector CuS Standard Full Output	61
Figure 32 PESA Detector CuS Standard Full Output.....	61

List of Tables

Table 1 Rack J, Patch Panel 505C, PIXE & PESA Output Connections.....	30
Table 2 Equipment Rack J, patch panel 505D, PIXE Output Connections.....	30
Table 3 PIXE Analysis Module Connections.....	31
Table 4 PESA Analysis Module Connections	31
Table 5 ADC: ND-575 Module Settings (for PIXE-1, PIXE-2, and PESA).....	34
Table 6 RATEMETER Model 441 Settings (for PIXE-1 and PIXE-2).....	34
Table 7 SCALAR Model 2011 AEC.....	34
Table 8 BEAM CURRENT INTEGRATOR, Model 2000AEC	34
Table 9 TIME PICKOFF CONTROL, Models 403 and 403A.....	34
Table 10 TIME PICKOFF CONTROL, Model 403	34
Table 11 Model 428	35
Table 12 QUAD SCALAR, Model 84	35
Table 13 ORTEC MODEL 572 AMP.....	35
Table 14 PESA DETECTOR CONTROL UNIT Model 210.....	35
Table 15 Sample IMPROVE Trayfile.....	49

Technical References

Thomas A. Cahill; Eldred, Robert; Wallace, Don; Kusko, Bruce, “The Hydrogen-Sulfur Correlation, by PIXE Plus PESA, an Aerosol Source Identification” Nuclear Instruments and Methods in Physics Research, B22, 1987, P. 296-300

J.F. Harrison and R.A. Eldred, “Automatic data acquisition and reduction for elemental analysis of aerosol samples”, in **Advances In X-Ray Analysis, Vol. 17**, Plenum Press, New York, NY., pages 560-583 (1974).

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure describes the Crocker Nuclear Laboratory (CNL) Particle Induced X-ray Emission (PIXE) and Proton Elastic Scattering Analysis (PESA) system and the analyses using this system. The CNL-PIXE/PESA system is designed to measure the concentrations of elements H and Na-Mn. It also provides a quality assurance check of heavier elements by the CNL-XRF system.

SOP 301 describes the standard operating procedure describes the CNL X-Ray Fluorescence (XRF) system for analysis of elements Fe-Pb.

2.0 RESPONSIBILITIES

2.1 Spectroscopist

The Spectroscopist shall:

- oversee and maintain records on the PIXE and PESA Systems
- perform maintenance and repair of the PIXE and PESA systems as necessary
- setup and oversee the analysis runs for PIXE and PESA
- calibrate and run controls on the PIXE and PESA systems
- resolve any inconsistencies in calibrations, reanalyses, or normal analyses.
- perform periodic back-ups of the acquisition and analysis programs and collected data

2.2 Quality Assurance Manager

The Quality Assurance Manager shall:

- approve the calibrations and reanalyses prior to analysis of normal samples.
- oversee and approve any modifications to the analysis system or software
- check for inconsistencies in calibrations, reanalyses, or normal analyses.

2.3 Computer System Operation Manager

The Computer System Operation Manager shall:

- oversee and maintain records on the data acquisition and analysis programs
- repair or correct any problems with acquisition or analysis programs.

3.0 REQUIRED EQUIPMENT AND MATERIALS

The equipment and materials required for PIXE and PESA analysis are listed below.

3.1 PIXE System

- Particle accelerator capable of producing a beam of 4.5 MeV protons
- Proton beam collimators
- Two SiLi detectors with liquid nitrogen dewars
- Liquid Nitrogen
- Automatic sample changer inside vacuum beam line analysis chamber.
- Power supply for sample changer
- Changer card (manual overrides for sample changer)
- Closed circuit TV camera
- Closed circuit TV Monitor
- PIXE Amplifiers, one for each detector
- PIXE detector Bias Power Supply
- PIXE system Pre-Amps
- Analog/Digital converters, one for each detector
- CNL/ARB Controller
- Quad Scalar
- Multi-channel Analyzer
- Patch panels (to facilitate wiring)
- MicroVAX

3.2 PESA System

Much of the equipment required for PESA analysis is provided by PIXE analysis, so only the additional requirements is listed.

- Solid state detector
- Detector amplifier
- Detector pre-amp

3.3 Calibration Requirements

- Standards tray containing 17 Micromatter™ thin film standards (Pb on Kapton, CuS on Kapton, NaCl, Mg, two Al, SiO, GaP, CuS, KCl, KI, CaF₂, Ti, V, Cr, Mn, Fe, Ni, Cu, ZnTe, GaAs, two Se, CdSe, CsBr, Au, Pb) and 2 clean filters (blanks) representative of the filter lot used for IMPROVE aerosol sampling
- Six 1/8 mil mylar squared mounted in analysis slides for PESA calibration.

3.4 Analysis Requirements

- Standards tray (from section 3.2)
- 2 trays of filters analyzed in the previous quarter for reanalysis. Generally SHEN1 and CRLA1 trays are selected.
- “A” module filters, in quarterly site trays, for PIXE analysis

4.0 METHODS

Three analytical methods are used to characterize the elemental composition of the aerosol deposits on the Teflon filters: PESA (H), PIXE (Na - Mn), and XRF (Fe-Pb). This standard operating procedure (SOP) refers to the procedures for performing PIXE (Particle Induced X-ray Emission) and PESA (Proton Elastic Scattering Analysis) analysis of aerosol samples on Teflon filters for elemental composition and concentrations.

- 4.1 Overview of the PIXE/PESA System
- 4.2 Preparation for Analysis—North Cave
- 4.3 Preparation for Analysis—Computer Room
- 4.4 Data Acquisition
- 4.5 Spectral Analysis
- 4.6 Routine Analysis

4.1 Overview of the PIXE/PESA System

Elemental analysis is performed by the Air Quality Group (AQG) at Crocker Nuclear Laboratory (CNL), a department of the University of California, Davis (UCD). Aerosol samples on Teflon filters are analyzed for elemental concentrations using three systems: Proton Elastic Scattering Analysis (PESA) for H, Particle Induced X-ray Emission (PIXE) for elements Na-Mn, and X-Ray Fluorescence (XRF) for Fe-Pb.

Proton Induced X-ray Emissions (PIXE) and Proton Elastic Scattering Analysis (PESA) techniques are conducted using the 4.5 MeV Protons produced by the 76" Cyclotron. In the north cave, the cyclotron beam terminates at several target chambers. AQG uses two different target chambers. Beam line 1A is used for analysis of filters that are pre-mounted on slide frames and loaded into trays. Beam line 1B handles substrates collected by the DRUM sampler using a *STRIP* target changer. These procedures deal only with analyses using beam line 1A. Electronics to run the detectors, both particle for PESA and X-ray for PIXE, are located in the north cave. Both analysis stations, beam lines 1A and 1B, are operated remotely from the Computer Room using automated filter changers.

A schematic of the PIXE/PESA system for beam line 1A is shown in Figure 1. The x-rays are detected by two high-resolution SiLi detectors with pulsed optical feedback to provide high count rate capabilities. The PIXE-1 detector is further from the sample and has a thinner filter, and has better sensitivity for elements between Na and Mn. The PIXE-2 detector has a larger solid angle and thicker filter, providing high sensitivity for elements Fe-Pb. The elemental range for PIXE-2 is identical to that from the XRF system. Since the minimum detectable limit with the XRF system is less than for PIXE-2, the data from PIXE-2 is not included in the final database. The PIXE-2 data are used as a quality assurance check of the XRF data.

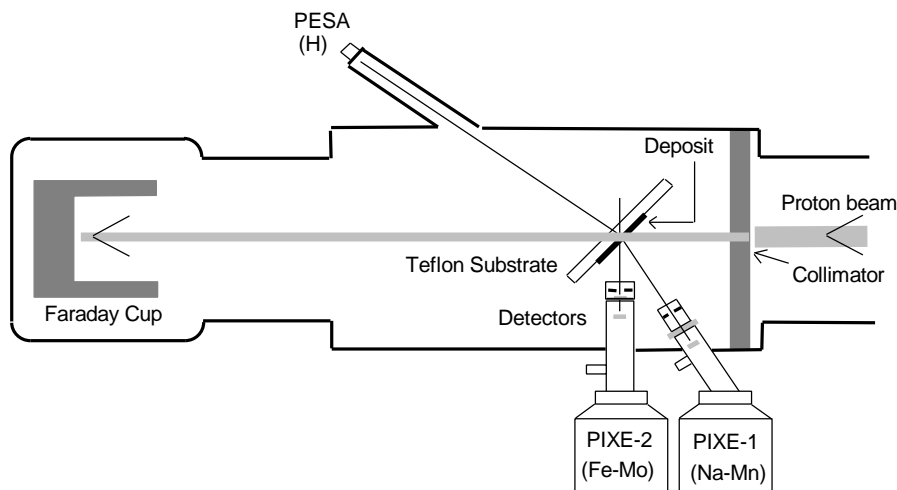
The PESA system uses a surface barrier detector (Ultra™ Ion Implanted Silicone Charged Particle Detector, made by EG&G Ortec™), to detect protons forward scattered 30° by their passage through the sample deposit.

The scatter is due to elastic collisions between incident protons and particles in the deposit. By positioning the detector location at 30° off the beam line in the forward direction, maximum definition of the peak produced by scattering from Hydrogen atoms is obtained. The hydrogen peak is one of several peaks produced by scattering protons. Other peaks including C, F, and O. The PESA technique is simple, but effective.

The system is calibrated using Mylar films of known density. The data from the detector are processed by standard electronics and accumulated in a dual ported analog to digital converter connected to an Ethernet local area network.

The number of x-ray and protons depends on the number of protons in the cyclotron beam. For PIXE-PESA this is determined by integrating the charge deposited by the beam in a Faraday cup behind the sample. This data is used for quality control of the PIXE and PESA systems.

Figure 1 PIXE and PESA Filter Analysis Area



The electronics to support the PIXE/PESA system are located in the North Cave of Crocker Nuclear Lab, and in the Computer Room of Crocker Nuclear Laboratory. The data from the detectors are processed by standard pulse processing electronics and accumulated in a dual ported analog to digital converter connected to an Ethernet local area network.

ACE, Acquisition of Composition by Element, runs the automated acquisition system for PIXE and PESA analysis. ACE controls the sample changer, using instruction files called 'trayfiles' that indicate which positions in each analysis tray contain valid samples. ACE collects the data generated during each analysis from each detector, including the x-ray spectra, the integrated charge, and the detector live time. The data from each detector is merged with sample identification data from the trayfile and saved as an output binary file; one file per detector for each analysis. The real time X-ray and particle spectrum, detector live time, and integrated charge can be displayed on the MicroVAX console by ACE for quality control.

The RACE program, Rapid Analysis for Composition by Element, reduces the data collected by ACE to report elemental concentrations. RACE opens each file created by

ACE and analyzes the spectrum produced during acquisition. It then subtracts the 'blank' spectrum (the spectrum due to the filter) and subtracts the background radiation due to scattering of x-rays or protons. The remaining spectrum, due to the deposits on the filter, is processed to locate elemental peaks. The peaks are identified by experimentally determined parameters, using the system calibration table, and deconvoluted to assess the contribution of any interfering peaks. Finally, a cross section table correction is applied to the peak characteristics to determine the concentration in ng/cm^2 of each elemental species in the deposit. The processed data, containing concentrations, errors, and minimum detectable limits of elemental species on each filter sample, is appended to a site and quarter specific file for later reference.

The major steps performed by RACE for spectral analysis are:

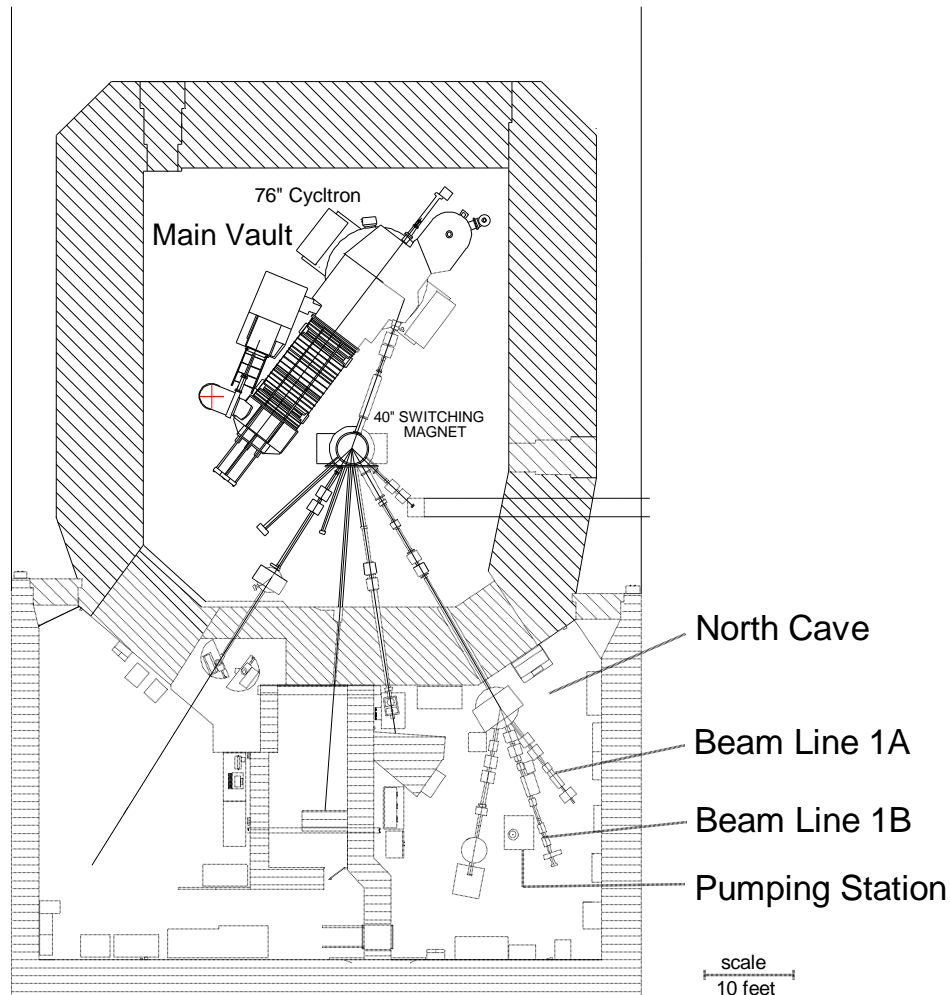
- Remove most of the background by subtracting a system blank.
- Remove the remaining background by subtracting a smooth function calculated as the lesser of the counts in the channel and an average counts per channel over a specified width.
- Use a peak search routine to identify the peaks and valleys by comparing the shape of the spectrum near a given channel to what would be expected if there were a peak at that channel.
- Fit each peak to one or more Gaussians. Using this information, the centroid of the peak, the energy calibration, the integrated charge and a "cross section table", calculate the elemental concentration represented by each peak.
- Calculate the uncertainty of the concentration from the uncertainty in the Gaussian area.
- Calculate the minimum detectable limits for all elements in the cross section table as 3.29 times the square root of the background counts under the region that would have been occupied by a peak of normal width.
- Resolve interferences between the various lines, using preset criteria.

IMPROVE sample analyses by PIXE are performed four times a year in analysis run sessions lasting approximately three days. Each analysis session begins roughly two months after the end of the quarter, pending the return of the filters and completion of gravimetric, HIPS, and XRF analysis.

4.2 Preparation for Analysis—North Cave.

This section will deal with the procedures in the North Cave to prepare for a PIXE/PESA analysis run. Section 4.3 will deal with procedures in the computer room. Ideally, the setup should be performed at least 24 hours in advance of an analysis run, but it can be done within 4 hours of analysis if absolutely necessary. A schematic of the cyclotron and north cave is shown in Figure 2.

Figure 2 CNL Cyclotron and North Cave

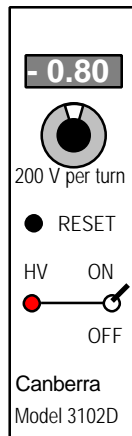


4.2.1 Filling the Detector.

1. Schedule to fill the detector with liquid nitrogen (LN) at least 4 hours before starting a PIXE/PESA analysis run. The detectors must be filled every 7-10 days and must **NEVER** be allowed to warm up. **NOTE:** Caution must be used when working with LN. Wear gloves and eye protection when using LN. No open toe shoes allowed.
2. Verify the two PIXE detectors are biased down to 0.00 volts. The amps used to bias the PX1 and PX2 detectors are Canberra™ Fast Spectroscopy Amp Model 2024. Check both Amps to verify that the bias voltage (gain) is 0.0. If not, refer to section 4.2.6 for instructions on biasing the detectors.
3. Once the detector bias is set to 0.00V, turn off the high voltage (HV) power supplies for the Amps using the on/off on the face of each power supply. The High Voltage Power Supplies, Canberra Model 3102, are located in the electronics rack in the North Cave (see Figures 2 and 3).

4. To fill the **PIXE** detectors with liquid nitrogen
 - a) See Figure 2 for a schematic of the North Cave.
 - b) Wheel in an LN storage tank.
 - c) To fill the **CANBERRA 1** (PIXE 1) connect one end of the Parker 3/8" hose to the LN tank and the other to the intake of the dewar using hand tightened compression fittings. If the detector is not loaded into the PIXE chamber you must first make sure that the detector is capped. Then firmly hold the detector head in place while slowly turning the LN tank valve open. Hold in place until the pressure and temperature equalizes. Thick leather gloves should be worn to protect the skin.
 - d) Close the valve on the LN tank when a steady stream of LN flows from the exhaust port on the dewar.
 - e) To fill the **CANBERRA 2** (PIXE 2), hand tighten one end of a Parker 3/8" diameter hose to the LN tank with compression fittings. Remove the Styrofoam cap on the dewar. With one gloved hand, insert the free end into the dewar and hold in place. With the other hand, open the valve on the LN tank slowly, to allow for pressure and temperature equalization.
 - f) When a steady stream of LN flows out the LN exhaust of the detector, close the valve on the LN tank.
 - g) Disconnect the hose, using gloves, and replace the LN tank.

Figure 3 HV Power Supply



4.2.2 Installing the Detectors.

The detectors are installed in the positions shown in Figure 1. The detectors, once installed, are rarely moved or changed. If a detector has been moved, contact the Spectroscopist for assistance. The Spectroscopist will reinstall and readjust the detector if necessary.

1. The Canberra 1 is placed at a back angle of 135° to the beam line. This is an awkward position so care must be taken when putting the detector in place. The Canberra 1 has its own stand which bolts to the floor as indicated by the yellow paint on the floor and stand. This stand allows the detector to be inserted and

removed at 135 degrees. Two people are usually required to remove or insert the detector; one to hold the detector in place and the other to operate the stand.

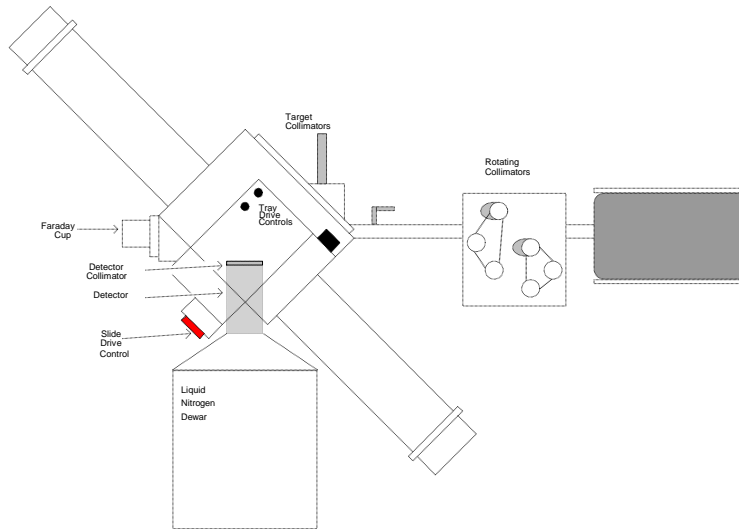
- a) To install the detector, first place the set collar onto the detector head of Canberra 1. The collar will hold the detector at the correct distance away from the beam line.
 - b) Next place the PVC collimator with funny filter into the port on the beam line for the PIXE1.
 - c) Swing the stand away from the PIXE 1 line and carefully insert the detector into the port.
 - d) Once the detector is flushed into the port, swing the stand under the detector to support the detector.
2. The Canberra 2 is placed on the rolling stand and is inserted straight up into the beam line. the preamplifier faces the main vault (north) and the handles of the dewar are placed to avoid the target chamber support frame. The detector head (cryostat) is not secured to the dewar. This is done so that the vacuum holds the detector in place isolated from floor vibrations.
3. The PESA port and support frame is oriented at 30 degrees from the beam line in the main target chamber. The port window is 2 mil Kapton. The PESA collimator and light filter (1 mil Aluminum) should always remain in place in the support frame. See Figure 1.
- a) Mount one of the newer detectors into the support frame. The detector must fit snugly into the support frame.
 - b) If you are able to rotate the detector within the frame freely, carefully remove the detector and add a layer of Teflon tape around the circumference of the detector and replace. Care must be taken because if the gold foil is touched in any way, or if dust falls on it, the sensitivity of the detector is seriously affected.
 - c) The detector should be rotated slightly each run to avoid concentrated radiation damage. Mark the support frame with a pen to indicate the detector orientation. You will see pen marks from previous runs.

4.2.3 Beam Collimation System

The proton beam is focused by several series of electromagnets in the beam line. This focusing process is performed by the cyclotron controller.

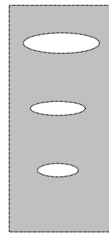
Two rotating collimators are located in the beam line just prior to the analysis area (See Figure 4). These collimators reduce the beam spread by removing protons not in the center of the beam line. The collimators are rarely adjusted, as most users require similar proton beams. There is a switch on the rotating collimators box to control the positioning of the collimators. If the collimators are suspected to be incorrectly positioned, contact the Spectroscopist for assistance prior to changing the collimator settings.

Figure 4 PIXE Collimators



Just prior to reaching the sample, the beam is collimated for accurate focusing, and to remove random proton scattering (see Figures 1, 4, & 5). This collimator, the “beam” or “target” collimator, is rarely adjusted. It consists of a rectangle of material with three ovals drilled into it. The medium sized oval, 3/16” x 13/32”, is used for IMPROVE filter analysis.

Figure 5 Target Collimator



Two more collimators, one between each PIXE detector and the sample, are installed when a new detector is installed. They are not adjusted unless a significant change in the system occurs, as they are used to optimize detector performance. These collimators, or “funny filters” are used to reduce the number of incident x-rays, and to filter out x-rays with energies outside the optimal range for the detector. The current collimators for the detectors are constructed as follows:

1. The PIXE-1 detector collimator is composed of a 1 ½” disk of 0.007” Mylar™. In the center of the Mylar™ disk is a 0.059” hole. The Mylar™ disk is mounted in a PVC frame which clips over the end of the PIXE-1 detector between it and the filter being analyzed.
2. The PIXE-2 detector collimator is composed of two disks; a 1 ½” disk of 0.007” mylar™ on top, and a 1 ½” disk of 0.008” mylar™ on the bottom. In the center of the 0.008” mylar™ disk is a 0.067” hole. The 0.007” mylar™ disk has no holes. The mylar™ disks are mounted in a PVC frame which clips over the end of the

PIXE-1 detector. The 0.008” Mylar™ disk is on the detector with the 0.007” Mylar™ above it, nearest the filter being analyzed.

Should there be a need to change the collimation system, the Spectroscopist will perform the modifications and adjustments. Following any modification, an analysis will be taken to satisfy radiation safety requirements and to allow the Spectroscopist to optimize the input beam and the detectors.

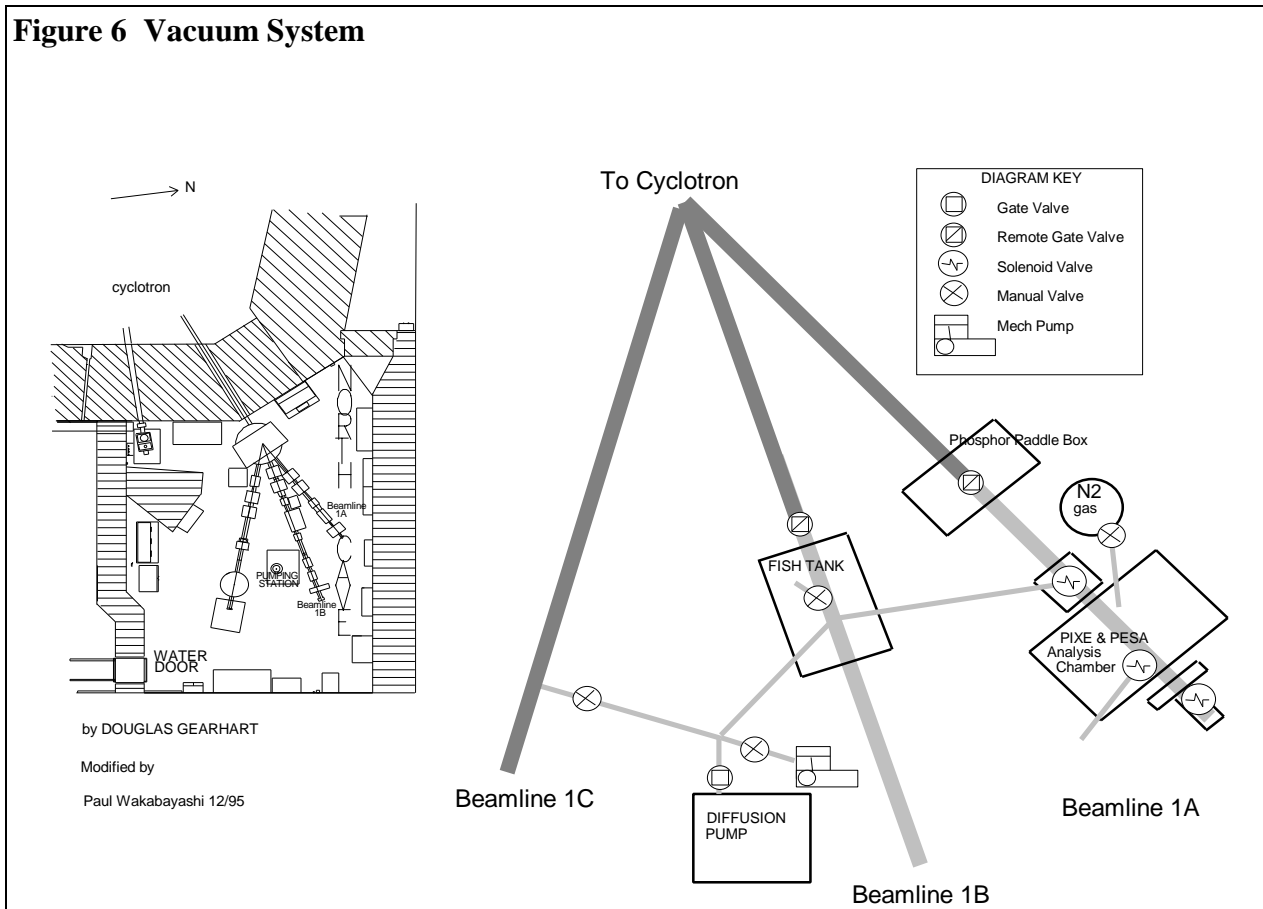
4.2.4 PIXE/PESA Vacuum System

PIXE and PESA analysis are performed under high vacuum. The vacuum system is a critical component of the PIXE and PESA analysis systems. The analysis chamber must regularly be brought up to atmospheric pressure to insert samples, then pumped down to high vacuum for actual analysis. High vacuum is required to reduce scattering of the proton beam between the source in the cyclotron and the target sample.

During a sample change, the analysis chamber is closed off from the cyclotron, and brought up to atmospheric pressure by injecting N₂ gas. Once the system reaches atmospheric pressure, the analyzed samples are removed, and new samples inserted. During this process, the chamber is overpressured with N₂ gas to reduce the influx of atmospheric air. Finally the chamber is sealed, and pumped back down to high vacuum. The N₂ gas serves a dual purpose; it is cleaner than air, so it does not contaminate the vacuum pumps as rapidly, and it is more efficiently removed by the diffusion pump (DP) than other atmospheric gasses.

Following each sample change, the vacuum in the target chamber must be increased to nearly the level of the rest of the cyclotron before the chamber can open to the cyclotron system. The target chamber is pumped down using two mechanical (mech) pumps and one diffusion pump (DP). These pumps are introduced into the system using solenoid or manual valves. Valves to other mechanical pumps and DP's are closed during PIXE and PESA analysis to reduce the time required to pump down the analysis area. Figure 6 shows the location of the valves and pumps important for control of the vacuum system for the PIXE & PESA analysis system (Beam line 1A).

Figure 6 Vacuum System



4.2.4.1 Vacuum system setup procedures

1. Close the mechanical valves for Beam line 1B and 1C prior to the PIXE & PESA analysis run. This will facilitate pumping down the target system to vacuum.
2. Make sure that both SiLi Detectors are in place. Verify that the detectors are well seated at the insertion point in the target chamber, and that they are set at the proper distance from the target with the appropriate collars.
3. Verify that the remote valve between the target chamber (analysis chamber) and the cyclotron system is closed.
 - a) In the control room, on the interactive vacuum system diagram on the wall, the indicator light for the valve should be under the “closed” button. If the “open” button is lit, press the ‘closed’ button to isolate the analysis area from the cyclotron.
 - b) For further insurance, in the North Cave, press the green button next to the remote gate valve over the Beam line 1A phosphor paddle. This button will only close the valve, not open it.
4. Verify that both the vacuum indicators at the analysis chamber and at the Diffusion pump station are working properly.
5. Press the **OFF** button close off the gate valve to the DP. This will disconnect the DP from the analysis chamber. This valve should be closed whenever the vacuum

in the analysis chamber is less than 40 milli torr as DP's are not efficient and are easily contaminated by pumping at higher pressures.

6. Turn manual valve **clockwise** close off the large mech pump next to the DP. This will separate the mech pump from the analysis chamber.
7. Make sure the vertical and horizontal power strips are off. Plug the vertical power strip into the horizontal power strip. Plug the horizontal power strip into any hot 110V outlet. Plug the power line for the solenoid valve connected to the small mech. pump on Beam line 1A into the vertical power strip.
8. Plug the power line for the solenoid valve connecting the main pumping station (large mech pump/DP) to Beam line 1A into the horizontal power strip.
9. Verify that the Beam line 1A vacuum probe is connected to the intake located on the Plexiglas plate on the analysis chamber.
10. Insert one to four trays of slides into the tube of the analysis chamber. See Section YY.
11. Flush the system with nitrogen by removing the top cap on the analysis chamber tube and opening the manual valve to the nitrogen tank. The psi on the regulator should read at ~ 20 psi. Allow the gas to flow for about 30-60 seconds. Replace the top cap and close the valve to the nitrogen tank.
12. Turn the manual valve to the large mech. Pump (next to the DP) counter-clockwise to open the pump to the beam line leading to the analysis chamber.
13. Switch on both power strips. This will activate the solenoids, opening both mech pumps to the analysis chamber. Both mech pumps will begin evacuating the analysis chamber.
14. Verify that the vacuum gauge over the analysis chamber is set to read the Beam line 1A probe. Watch the evacuation of the target chamber on the vacuum gauge. If vacuum does not register on the gauge after 3 minutes, verify that the system is isolated (all the other valves are closed, and there are no areas that are leaking. If no leaks are found, and the system is isolated, the probe may be malfunctioning. Check the connections.
15. If everything is going well, the chamber should reach 40 milli torr after about 5-10 minutes. At that point you will want to switch over from the mech pumps to the DP.
 - a) Shut off the power on the vertical power strip to close off the small mech pump.
 - b) Turn the manual valve near the DP clockwise to shut off the large mech pump.
 - c) Press **OPEN** on the solenoid valve to the DP to open the DP to the analysis chamber. The DP should bring the vacuum down to 5-10 milli torr within a few minutes.
16. Check the vacuum seals by pressing the “off” button on the DP solenoid valve to close the off the DP from the analysis chamber. Wait for a minute or two to see if the vacuum comes back up.
 - a) If the vacuum remains stable, and is below 40 milli torr, press the “on” button on the DP. The vacuum system is ready for use. If the vacuum is above 40 milli torr, repeat steps 12 through 15.

- b) If the vacuum comes up significantly (more than 50 milli torr per minute) there may be a leak. Check the o-ring seals on the analysis chamber by dabbing alcohol on to the o-ring's. The alcohol will momentarily block the leak, causing the vacuum to suddenly increase. If an increase is seen for any O-ring,
- i) bring the analysis chamber back up to atmospheric pressure by opening the manual valve to the nitrogen tank. The psi on the regulator should read at ~ 20 psi. Allow the gas to flow until the cylindrical caps on the analysis chamber tube can be easily removed. Then, turn off the manual valve to stop the flow of N₂ gas.
 - ii) Remove, clean and lubricate the suspect o-ring, replacing it if necessary.
 - iii) Clean any debris off the metal o-ring seat as well.
 - iv) Repeat steps 11 through 16.

4.2.5 Wiring and Powering PIXE Equipment in the North Cave.

4.2.5.1 Connecting the PIXE Detectors to Power.

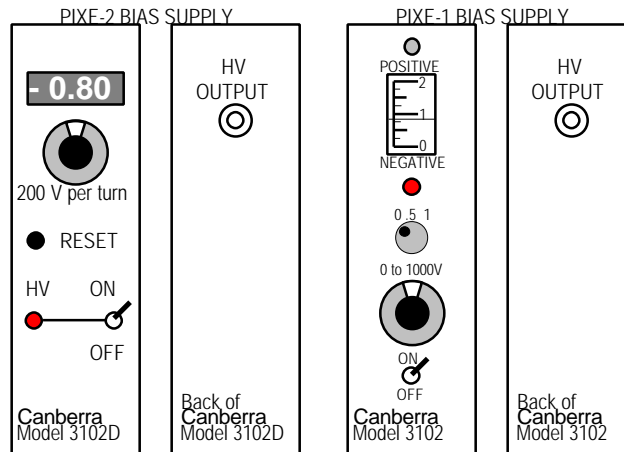
The PIXE detectors each have regulated power supplies, currently Canberra™ Model 3102D's, providing bias voltages of -800 V. The power supplies are located on the electronics rack adjacent to Beam line 1A in the North Cave.

The PIXE-1 detector utilizes a Canberra Model 3102 bias power supply while the PIXE-2 detector utilizes a Canberra Model 3102D bias power supply (see Figure 7). The bias supply used for each detector will be located adjacent to the PreAmp for the detector in the NIM module.

The HV output on the back of the bias power supply should be connected to the PIXE PreAmp HV position with a black BNC cable.

The bias power supplies should not be turned on until all connections have been checked. Procedures for using the bias supply will follow in section 4.2.7. Contact the Spectroscopist for assistance if any power supply is not functioning.

Figure 7 PIXE Bias Supplies

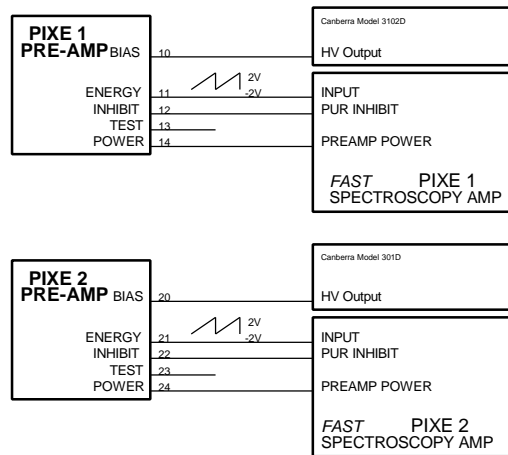


4.2.5.2 PIXE Detector Pre-Amp to Amp Wiring.

1. **PIXE-1** PreAmp has three input and two output cables, as seen in Figure 8.
 - a) signal input - input - hardwired to PreAmp
 - b) bias voltage - input - HV (black) - wire #10
 - c) preamplifier power - input - gray multi-wire cable - wire #14
 - d) inhibit - output - red BNC (125 Ω) - wire #12
 - e) preamplifier signal (energy) - output - black BNC (RG58, 50 Ω) - wire #11

2. **PIXE-2** PreAmp has three input and two output cables, as seen in Figure 8:
 - a) signal input - input - hardwired to PreAmp
 - b) bias voltage - input - HV (black) - wire #20 from Canberra Model 3102D
 - c) preamplifier power - input - brown multi-wire cable - wire #24
 - d) inhibit - output - red BNC (125 Ω) - wire #22
 - e) preamplifier signal (energy) - output - red BNC - wire #21

Figure 8 PIXE Amp Wiring



4.2.5.3 PIXE Amp Output Wiring.

Use Figures 8, 9 and 10 to check the wiring of the PIXE-1 and PIXE-2 electronics. The connections are frequently changed as the computer room is shared them with other users. There are two cables from each detector preamp to the detector amp. These should be wired as shown in Figures 8 and 9.

There are five cables coming from each PIXE amp that must be sent to the computer (see Figure 10). All cables go to the computer room through patch panels 501C (125 OHM BNC), 505D (50 OHM BNC), and 505B (HV). See Figure 11 for the patch panel location.

Figure 9 Front and Rear of PIXE Amp

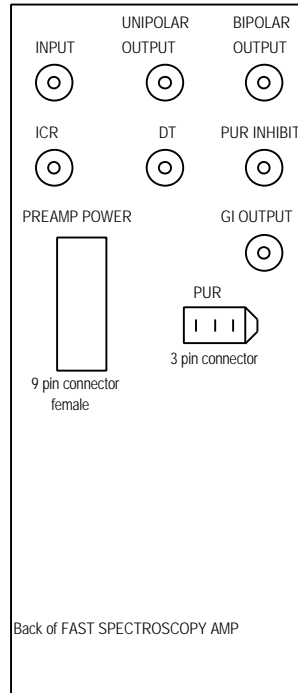
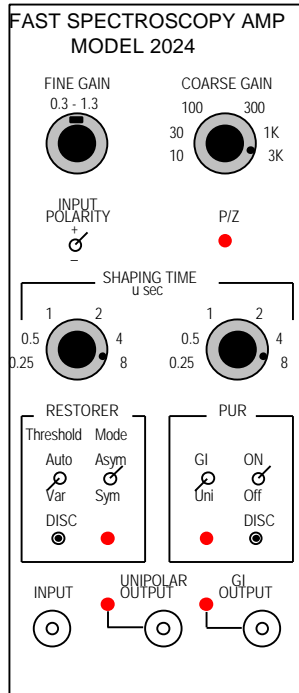
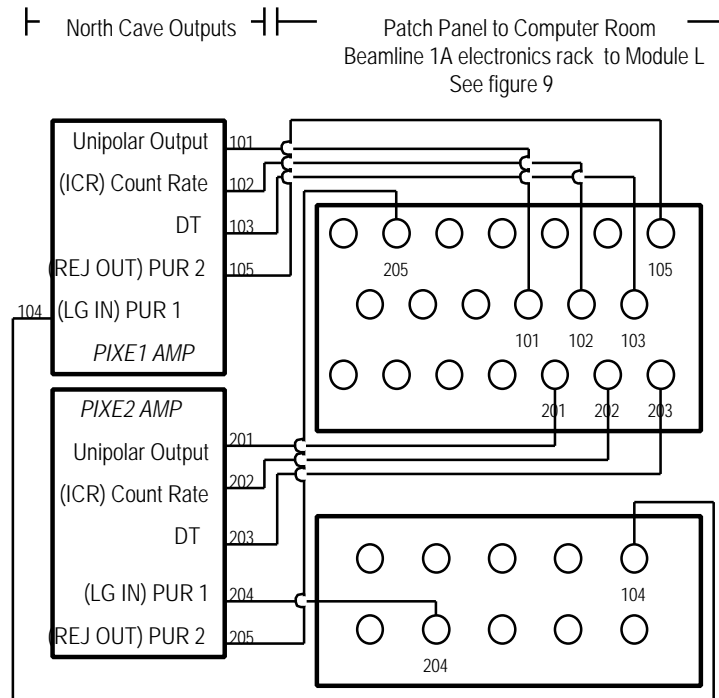


Figure 10 PIXE System Amp Output Wiring



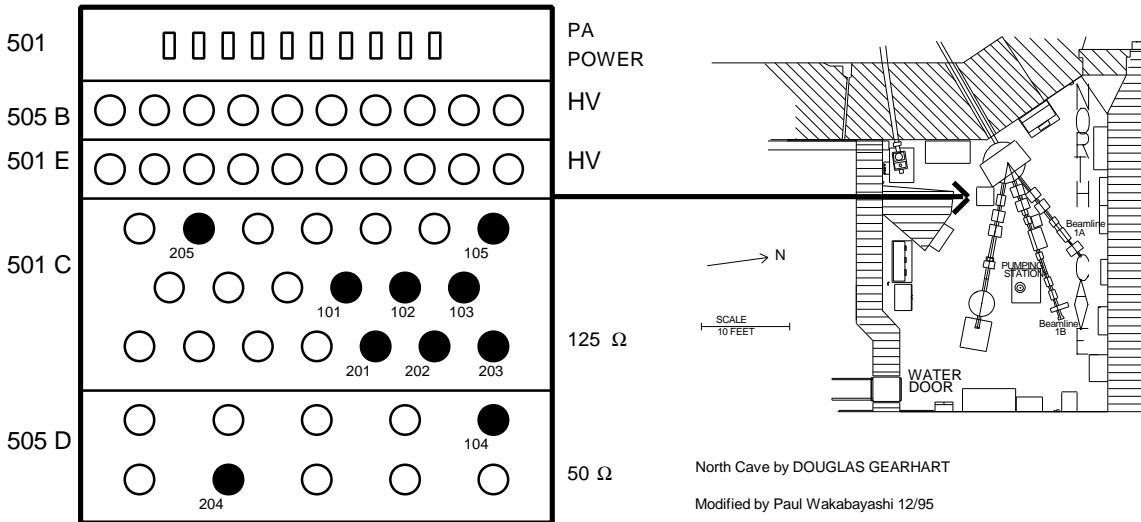
CANBERRA 1

1. amplifier signal - Unipolar Output - red BNC - 101
2. count rate -ICR -red BNC - 102
3. dead time - DT - red BNC - 103
4. linear gate from MCA to amplifier -PUR 1 -black BNC -104
5. pile-up rejection - PUR 2 - red cable - 105

CANBERRA 2

1. amplifier signal - Unipolar Output - red BNC - 201
2. count rate - ICR - red BNC - 202
3. dead time - DT - red BNC - 203
4. linear gate from MCA to amplifier - PUR 1 - black BNC/50 Ω -204
5. pile-up rejection - PUR 2 - red cable/125 Ω - 205

Figure 11 North Cave Patch Panel



4.2.5.4 Connecting the PESA Detector to Power.

The PESA detector's power supply is located in the Counting room, and not in the North Cave. The power supply should be off while PESA is being wired to the analysis system. The power supply, Ortec Power Supply Model 210, is located in the computer room on Module K.

4.2.5.5 PESA Detector Wiring.

The PESA detector's amplifier is physically located in the computer room, not in the north cave as in the PIXE electronics. This section will deal with wiring from the surface barrier detector to the patch panel between the north cave and computer room. Most of the PESA wiring should remain setup from run to run. It should be checked each time nonetheless. The PESA detector system includes a detector and pre-amp (ORTEC) in the North Cave and an amplifier (ORTEC 572) and bias supply (ORTEC 210) in the computer room.

Connect the detector to its pre-amp with a 125Ω BNC cable. Cover the detector and support with Aluminum foil to block all light. This foil shield should be grounded to the target chamber

1. **PESA** has four separate cables (see Figure 12).
 - a) bias voltage - HV (black) - 30
 - b) preamplifier signal (energy) - black BNC (RG58, 50 Ω) - 31
 - c) input - red cable (125 Ω)
 - d) preamplifier power - gray multi-wire cable - 34

2. Connect the detector to its pre-amp with a 125Ω BNC cable. Cover the detector and support with Aluminum foil to block all light. This foil shield should be grounded to the target chamber
3. Connect the signal, bias, and pre-amp power wires from the pre-amp to the patch panels near BL1C (see Figures 12 and 13)

Figure 12 PESA Output Wiring

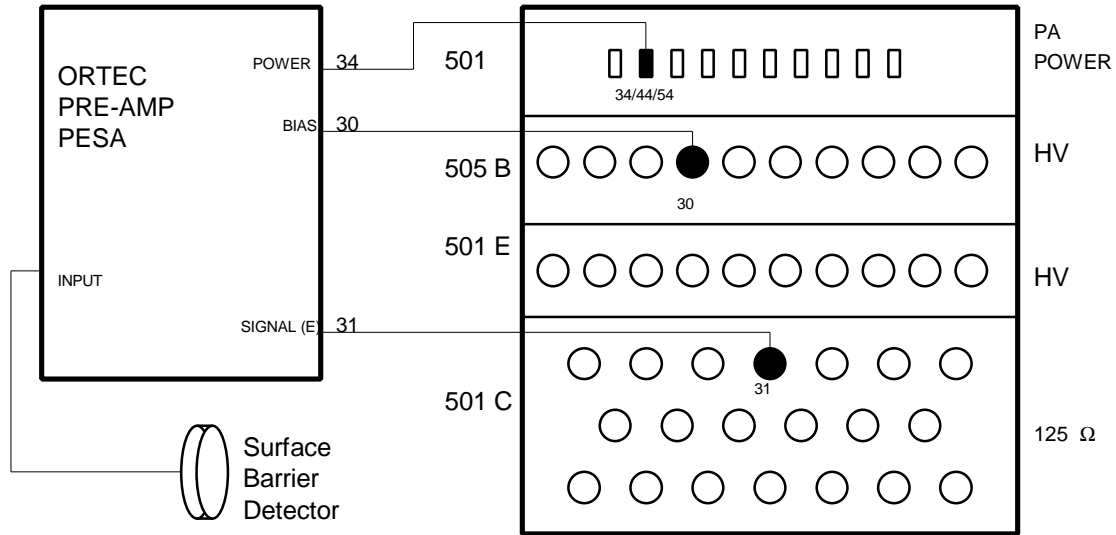
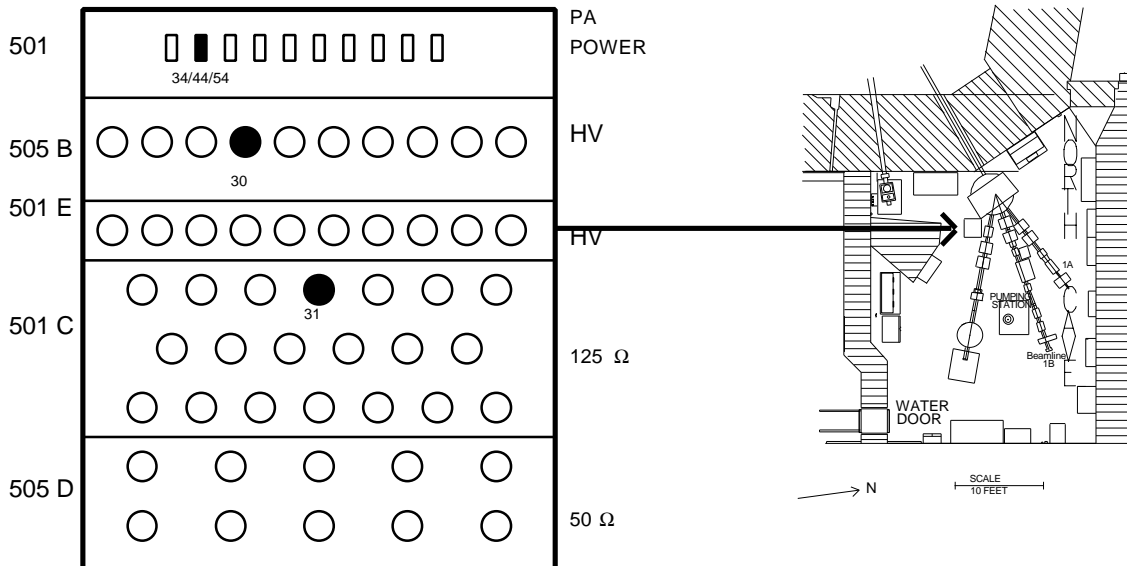


Figure 13 PESA Patch Panel in the North Cave



4.2.5.4 Cyclotron Operations

Cyclotron control is provided by the cyclotron operators. Users request a beam energy and count rate, and the operators tune and prepare the beam for use. The Air

Quality Group uses a sweeping proton beam at 4.5 MeV with no more than 2000 counts per second measured at the PIXE 2 detector. With the collimator, this should provide a beam spot on the analysis filter of approximately 0.74 cm². Cyclotron beam tuning requires about two hours, and cannot be performed until the analysis area is ready for use (the vacuum systems have been tested).

4.2.5.5 Count Rate Meter

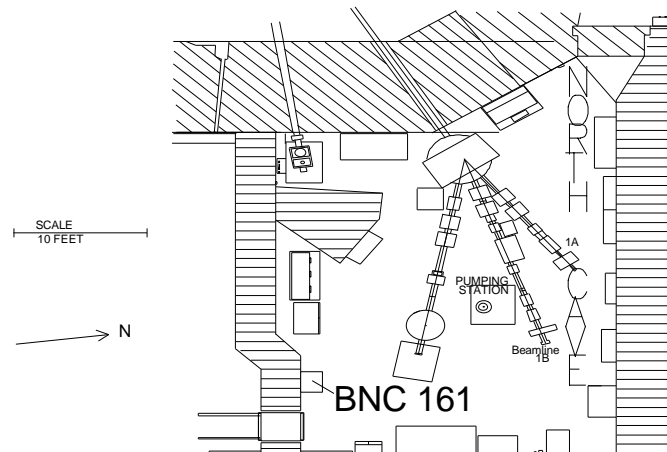
The count rate is measured by each detector and output as ICR on both PIXE-1 and PIXE-2 (see Figure 10). Count rates (cables **102** & **202**) go to meters in the computer room where they are displayed and recorded, as well as in the cyclotron control room for cyclotron operator reference. The count rate on the PIXE-2 detector should be 2000 counts per second during normal operation

The count rate meters are located in Module K of the Counting Room. PIXE-2's count rates go directly to a count rate meter in the computer room. The count rate from PIXE-1 must be amplified and lengthened, so that the analog count rate meter can properly register the signals. The computer room wiring for these signals is covered in section ???

4.2.5.6 Proton Beam Current

The Proton Beam current is measured from the Faraday cup at the end of the beam line. The line from the Faraday cup runs to the patch panel above Beam line 1A, and from there to the junction box, BNC Panel 161, on the south wall of the North Cave near the door. The line from the Faraday cup should be connected to position J16103. From the junction box, the line runs to the control room for cyclotron operator reference, and from there to the computer room. For the junction box location, see Figure 14.

Figure 14 BNC 161 Junction Box Location

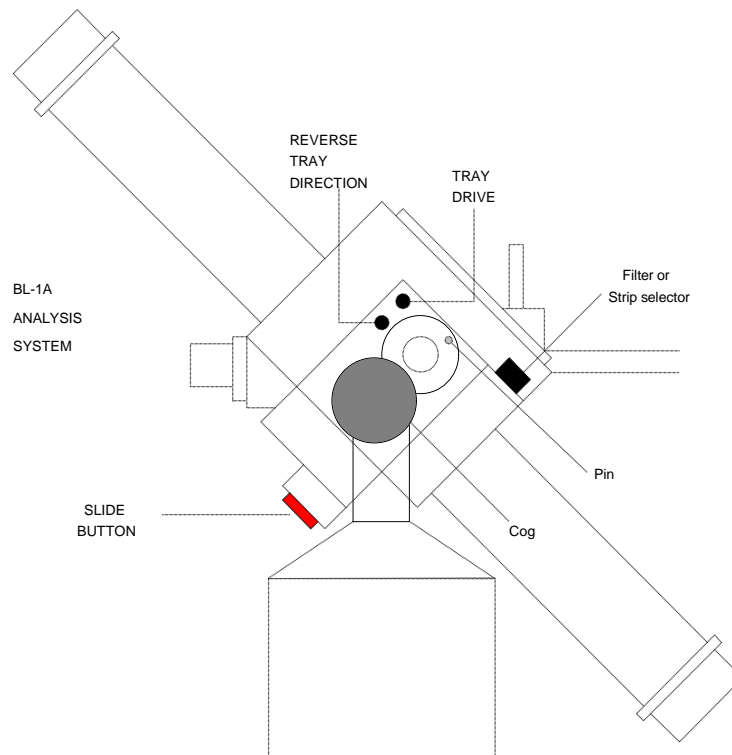


4.2.5.7 Slide Changer.

The slide changer is wired directly to power and is mounted onto the analysis chamber (see Figure 15). It should always function. If not, contact the electrician for assistance. The slide changer is controlled in automatic mode by the data acquisition program ACE, through the CNL/ARB controller to the slide changer. However, there are two manual override methods; the first using the changer card on the BL-1A analysis chamber in the North Cave, and the second using manual mode on the CNL/ARB controller in the computer room. The procedures for manual control using the changer card follow.

1. To remove a slide from the beam, press the slide button, located on the lower left side of the BL-1A analysis system until the slide has moved out of the beam and back into the storage tray. This can be observed through the clear Plexiglas cover on the analysis chamber. If you continue to press the button, the slide will cycle between the position in the beam and the position in the storage tray. The slide is in the “Out” position when it is fully in the storage tray, and the lever arm for the slide drive mechanism is visibly in the farthest out position.
2. To move the tray, press the Tray Drive button in the analysis chamber. Be sure that the slide is in the “out” position or the system will jam.
3. To move the tray in the opposite direction (backward), simultaneously press the Reverse Tray Direction button and the Tray Drive button.
4. The tray also moves continuously, so it must be stopped at the correct location or, when the operator attempts to put a slide into the beam, the system will jam. To ensure correct positioning of the tray, verify that the pin on the drive wheel for the tray is in the position shown in Figure 13.
5. To insert a slide into the beam, verify the tray is positioned correctly. Next, press the slide button until the slide has moved into of the beam. The slide is in the “In” position when the lever arm, viewed through the Plexiglas cover, is flush with the side of the tray being analyzed.

Figure 15 PIXE & PESA System Changer Card



4.2.5.8 Remote Monitoring System.

Two cameras are used to monitor the BL-1A analysis system. One camera is located above the analysis chamber and is focused on the filter in position for analysis. The camera feed is sent on a coaxial cable to the computer room. The second camera is focused on the phosphor beam paddle (see Figure 6) prior to the BL-1A analysis system. This is used by the cyclotron operator when he/she needs to view the beam trace.

The camera output lines are routed through hardwired camera patch panels into the cyclotron control room, and from there to the computer room. The first camera, above the analysis chamber, is routed to position 5 on the patch panel above BL-1B. The second camera is never disconnected.

In module N of the computer room, there are normally two monitors. Near the monitors there is a toggle switch labeled camera one and XRF. Flip the toggle to camera one to set the monitors to read input from the North Cave cameras.

4.2.6 North Cave Electronics Settings

Set the following switches, pots, etc. as in the table below.

PIXE 1 AMPLIFIER (CANBERRA 2024)

GAIN (typical value)	300 x 0.80	
INPUT POLARITY	+	from Canberra Pre-Amp 2001
SHAPING TIME CONSTANT	8 μ S	GI (not used) Amp
RESTORER THRESHOLD	AUTO	
RESTORER MODE	SYM	
PILE-UP REJECTION (PUR)	UNI ON	
OUTPUT	UNIPOLAR	no termination at GAR

PIXE 2 AMPLIFIER (CANBERRA 2024)

GAIN (typical value)	1k x 0.40	
INPUT POLARITY	+	from Canberra Pre-Amp 2008
SHAPING TIME CONSTANT	8 μ S	GI (not used) Amp
RESTORER THRESHOLD	AUTO	
RESTORER MODE	SYM	
PILE-UP REJECTION (PUR)	UNI ON	
OUTPUT	UNIPOLAR	no termination at GAR

4.2.7 Biasing the SiLi Detector

The detectors are powered through the Nuclear Instrument Module (NIM) bin next to the analysis chamber on BL-1A. The NIM contains the amplifiers and bias supplies. To power the analysis system,

1. Verify that the bias voltages are set to 0. If not, check whether the NIM is being used.
2. If the bias voltages are 0.0, and no other users are currently accessing the NIM, turn on the power by flipping the switch on the MODEL 401A BIN to the ON position.
3. For PIXE-1: see Figure 7. Turn on the bias supply using the on/off switch on the Model 3102 Bias Supply. Use the fine voltage adjustment (pot) to bias the detector up to -800V. In order to avoid damage to the detector, increase the voltage slowly, no faster than 10V per second. (.800 on the gauge, 8.00 on the pot)
4. For PIXE-2: see Figure 7. Turn on the bias supply using the on/off switch on the face of the PIXE-2 Model 3102D Bias Supply. Use the fine voltage adjustment (pot) to bias the detector up to -500V. In order to avoid damage to the detector, increase the voltage slowly, no faster than 10V per second. (.500 on the digital read out, 2.50 on the pot)

PIXE 1 BIAS SUPPLY (CANBERRA 2008)

VOLTAGE	-800 V	coarse = 0, fine = 8.00
---------	--------	-------------------------

PIXE 2 BIAS SUPPLY (CANBERRA 3102D)

VOLTAGE	-500 V	coarse = 0, fine = 2.50
---------	--------	-------------------------

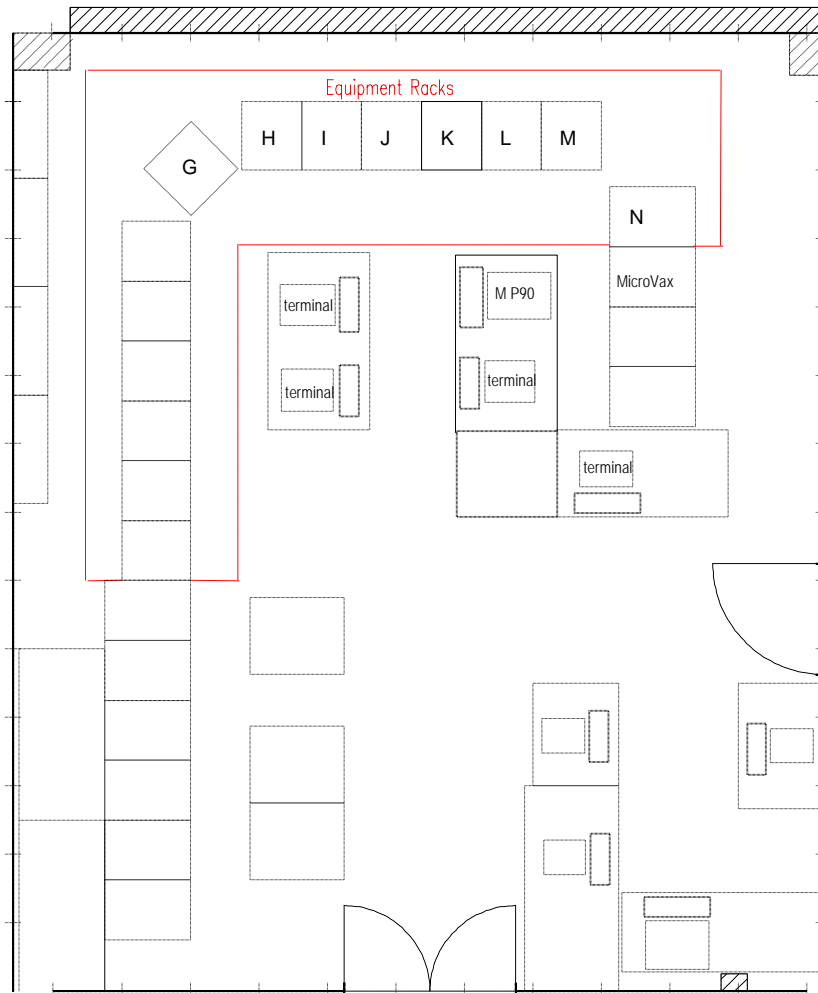
4.3 Preparation for Analysis—Computer Room.

Data acquisition takes place in the computer room of Crocker Nuclear Laboratory (see Figure 16). Cables from the North Cave are routed to the computer room and hooked to conveniently located patch panels. The patch panels are located in the equipment racks, which also contain the interconnect panels and signal processing electronics. The processed signals are fed into a MicroVAX for storage and initial analysis.

The user interface to the system is through a MicroVAX terminal. A console display terminal, the Micro P90, (M P90), displays real time data acquisition for quality control.

Using a MicroVAX terminal running ACE, the operator can oversee data acquisition, and adjust acquisition parameters. With a second terminal running RACE, the operator can review the results of the initial data processing.

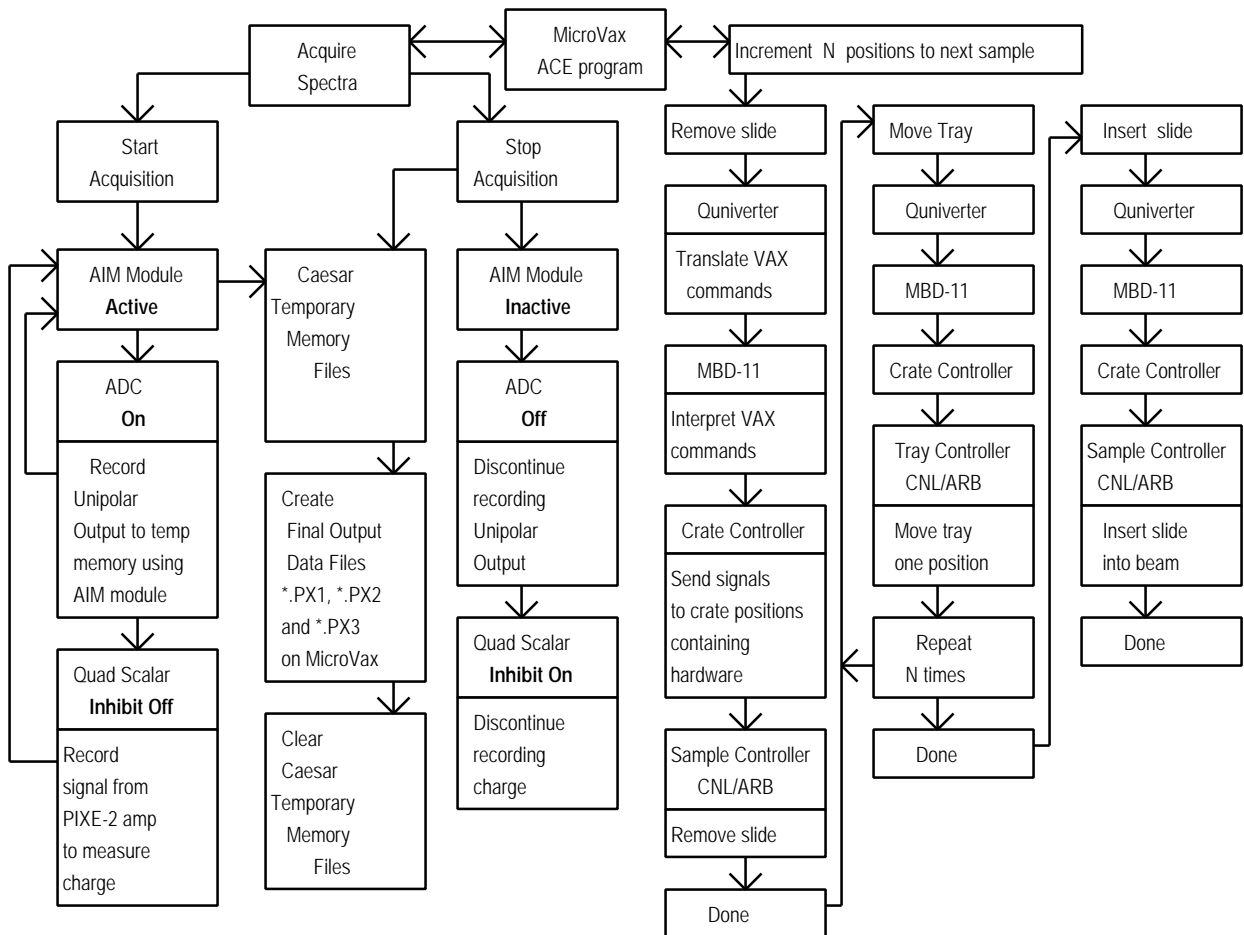
Figure 16 Computer Room Schematic



The hardware components for PIXE and PESA in the computer room are controlled by the ACE program. A schematic of the data acquisition process follows below in Figure 17. The components shown in Figure 16 are described below.

1. MicroVAX - Caesar - Runs data acquisition program ACE and data processing program RACE.
2. ADC ND575 - Analog to Digital Converter Nuclear Data-575, the multi-channel analyzer that processes the raw spectrum produced by the PIXE Amps into digital format.
3. AIM - acquisition interface module; connects the ADC to the local Ethernet.
4. Ethernet Transceiver - Ethernet connection to Caesar.
5. Quad Scalar - When inhibit is off, records the cumulative charge to which the sample is exposed. When inhibit is switched on, by ACE, stops counting charge accumulation.
6. MBD 11 - Data connection to Caesar for hardware components in Module N.
7. Quniverter - Interfaces hardware Unibus device with Qbus device for Caesar.
8. Patch panels - Connection panels.

Figure 17 PIXE & PESA System Control Schematic



4.3.1 Counting Room Electronics Connections

The connections are listed in Tables 1-4. See Figures 18-22 for specific locations.

Table 1 Rack J, Patch Panel 505C, PIXE & PESA Output Connections

FROM	TO
Pos. 11, cable 101, PIXE-1 SIGNAL	Module L, left ND-575 ADC, SIGNAL input (front) See Figure 20
Pos. 18, cable 201, PIXE-2 SIGNAL	Module L, right ND-575 ADC, SIGNAL input (front) See Figure 20
Pos. 12, cable 102, PIXE-1 CR	Module K, left ORTEC Model 441 Ratemeter, INPUT (front) See Figure 18
Pos. 19, cable 202, PIXE-2 CR	Module K, right ORTEC Model 441 Ratemeter, INPUT (front) See Figure 18
Pos. 13, cable 103, PIXE-1 DT	Module L, ADC 1 patch panel, BUSY
Pos. 20, cable 203, PIXE-2 DT	Module L, ADC 2 patch panel, BUSY
Pos. 7, cable 105, PIXE-1 PUR 2	Module L, ADC 1 patch panel, BLOCK
Pos. 2, cable 205, PIXE-2 PUR 2	Module L, ADC 2 patch panel, BLOCK
Pos. 4, cable 301, PESA SIGNAL	Module J, Patch Panel 12, Pos. 8

Table 2 Equipment Rack J, patch panel 505D, PIXE Output Connections

FROM	TO
Pos. 5, cable 104, PIXE-1 PUR 1	Module L, patch panel ADC 1, EVENT
Pos. 7, cable 204, PIXE-2 PUR 1	Module L, patch panel ADC 2, EVENT

Table 3 PIXE Analysis Module Connections

FROM	TO
Rack L, ND575 ADC left, BNC above ZERO see Figure 20	Rack N, left Quad Scalar, lowest BNC below INHIBIT see Figure 21
Rack N, left Quad Scalar, INPUT 0 see Figure 21	Rack M, Model 428, bottom panel, upper left BNC at OUT see Figure 19
Rack M, Model 428, bottom panel, upper left BNC at IN see Figure 19	Rack M, Model 403A, NEG OUTPUT 2 see Figure 19
Rack M, Model 403A, INPUT see Figure 19	Rack M, Model 403, OUTPUT A see Figure 19
Rack M, Model 403, INPUT see Figure 19	Rack K, Model 2000AEC, V to F see Figure 18
Rack K, Model 2000AEC, INPUT see Figure 18	Rack J, module 249, position 1, TO INTEGRATOR
Rack K, Model 2030AEC, top BNC see Figure 18	Rack K, patch panel 11, position 11, RF PULSER DIRECT RUN
Rack K, Model 2030AEC, second BNC see Figure 18	Model 2397 (small in line box, 1"x 1"x 2"), INPUT
Model 2397 (small in line box, 1"x2"x3"), OUTPUT	Rack K, Panel 14, position 3
Model 2397 (small in line box, 1"x2"x3"), +12V	Rack K, Model 401A, +12V
Rack K, left Ratemeter Model 441, back, RECORDER (red), Ground (black), dual plug	Rack K, back of patch panel, position 6, Count Rate 1, single plug
Rack K, patch panel, position 6, Count Rate 1	Rack J, patch panel 249, position 2 (To Control Room) This is input to bar graph meter # 3 on the control room console.
Rack K, right Ratemeter Model 441, back, RECORDER (red), Ground (black), dual plug	Rack K, back of patch panel, position 7, COUNT RATE 2, single plug
Rack K, front of patch panel, position 6, COUNT RATE 2	Rack J, patch panel 249, position 3 (To Control Room) This is input to bar graph meter # 4 on the control room console.
Rack L, left Model ND575 ADC, 21 pin output on back of module	Rack L, Ethernet Acquisition Server 284, ADC1
Rack L, right Model ND575 ADC, 21 pin output on back of module	Rack L, Ethernet Acquisition Server 284, ADC2

Table 4 PESA Analysis Module Connections

FROM	TO
Rack L, panel 12, position 8	Rack M, ORTEC Model 572 Amp, INPUT see Figure 22
Rack M, ORTEC Model 572 Amp, UNI see Figure 22	Rack M, Model ND575 ADC, SIGNAL see Figure 20
Rack M, Model ND575 ADC, 21 pin output on back of module	Rack M, Ethernet Acquisition Server 284, ADC3

Figure 18 Rack K PIXE Equipment

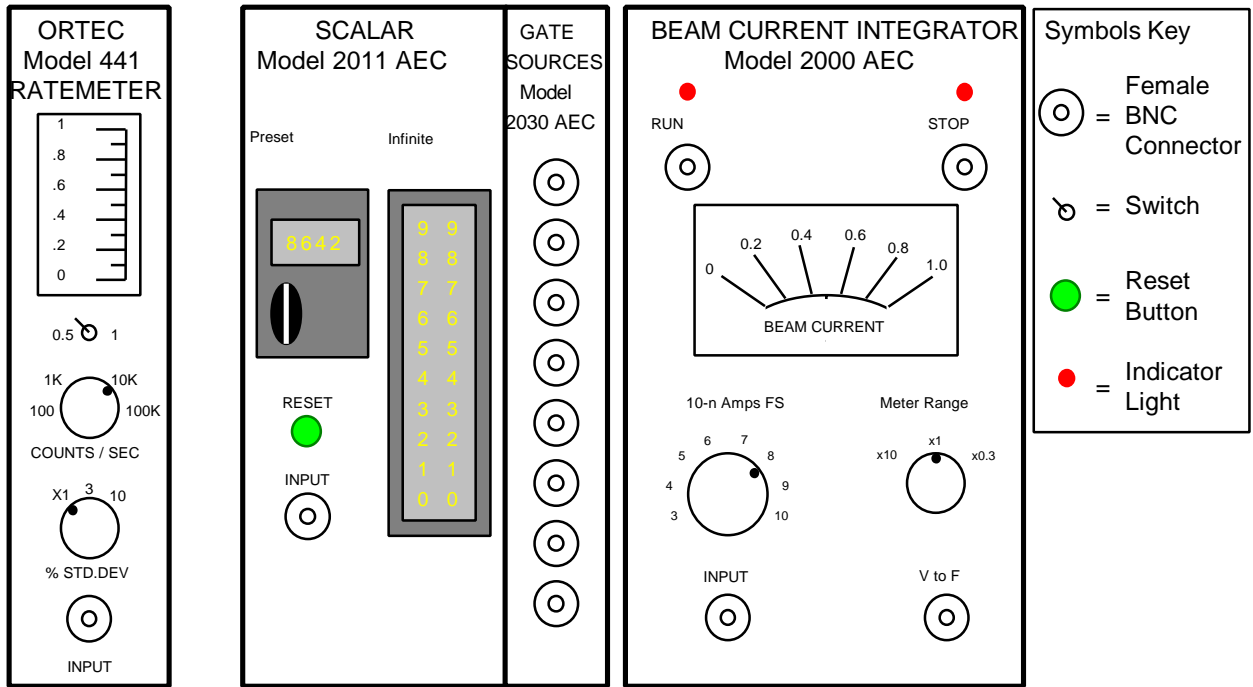


Figure 19 Rack M PIXE Components

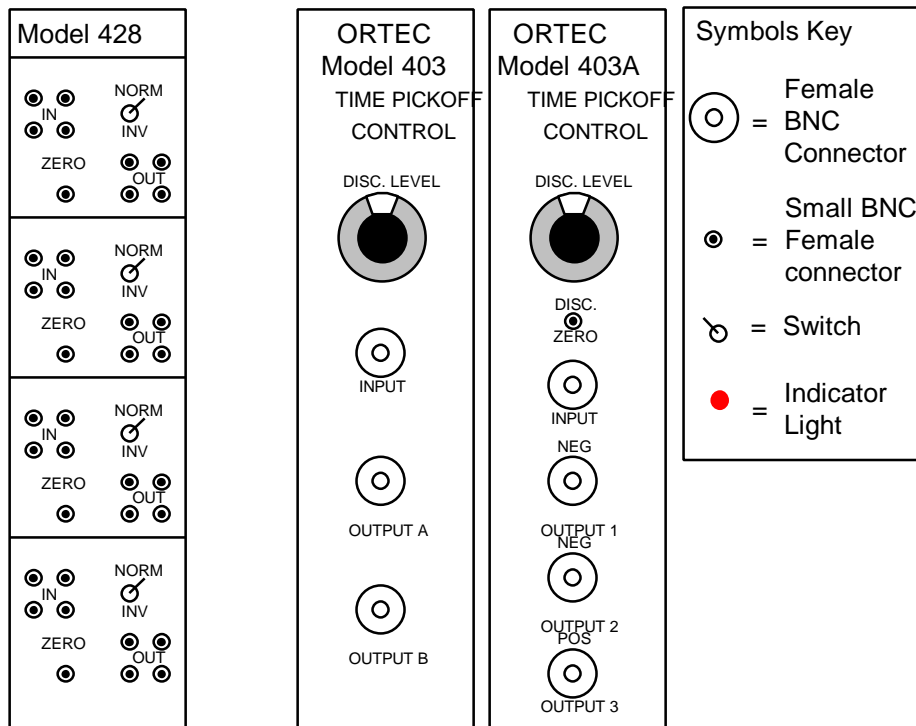


Figure 20 Rack L PIXE Components

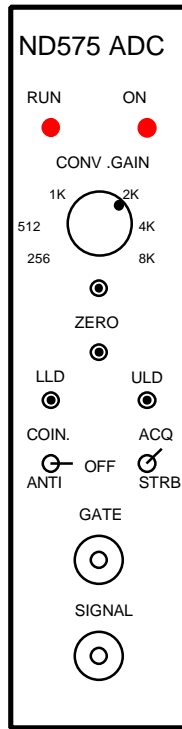


Figure 21 Rack N PIXE Components

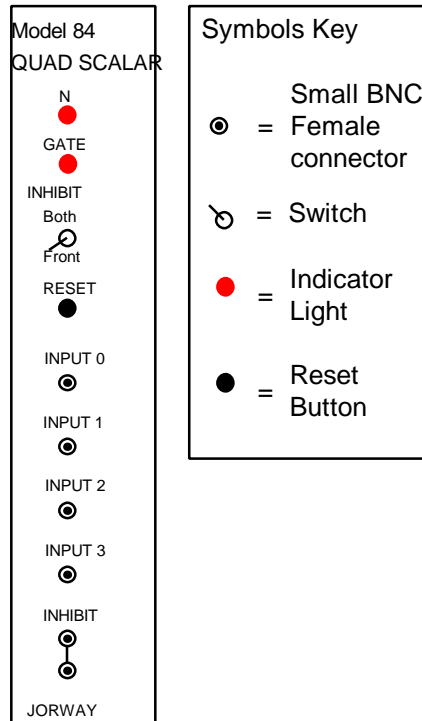
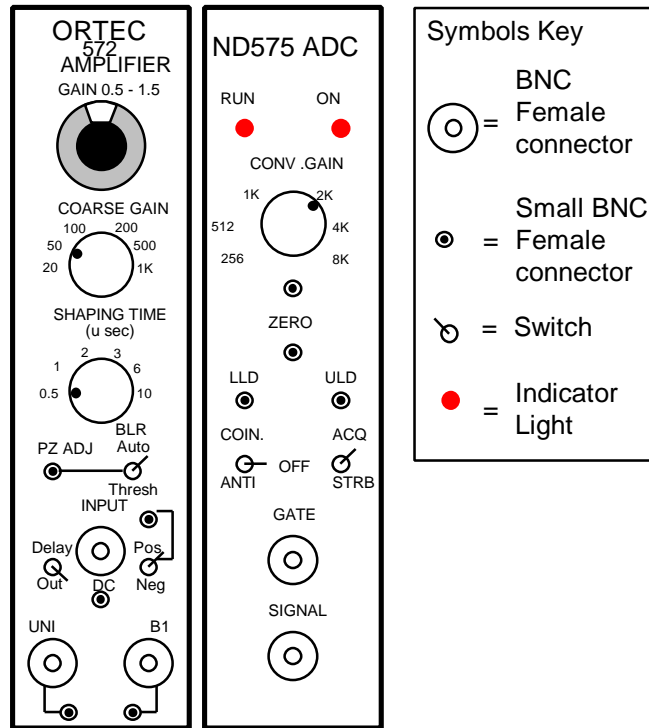


Figure 22 Rack M, PESA Components



4.3.2 Counting Room Electronics Settings

The settings for PIXE and PESA analysis are displayed in Figures 18 through 22. A hard copy listing of the settings follows in Tables 5 through 14

Table 5 ADC: ND-575 Module Settings (for PIXE-1, PIXE-2, and PESA)

CONV. GAIN	512
COIN/ANTI	OFF
ACQ./STRB	ACQ.

Table 6 RATEMETER Model 441 Settings (for PIXE-1 and PIXE-2)

X	0.5
COUNTS/SEC	10K
% STD.DEV	X1

Table 7 SCALAR Model 2011 AEC

PRESET/INFINITE	INFINITE
-----------------	----------

Table 8 BEAM CURRENT INTEGRATOR, Model 2000AEC

10-n Amps FS	8
METER RANGE	X1

Table 9 TIME PICKOFF CONTROL, Models 403 and 403A

DISC.	0.5
-------	-----

Table 10 TIME PICKOFF CONTROL, Model 403

DISC.	0.5
-------	-----

Table 11 Model 428

NORM/INV	NORM
METER RANGE	X1

Table 12 QUAD SCALAR, Model 84

BOTH/FRONT	FRONT
METER RANGE	X1

Table 13 ORTEC MODEL 572 AMP

GAIN	0.70
COARSE GAIN	50
SHAPING TIME	0.5 μ S
BLR: AUTO/THRESH	AUTO
INPUT: POS/NEG	POS
DELAY/OUT	OUT

Table 14 PESA DETECTOR CONTROL UNIT Model 210

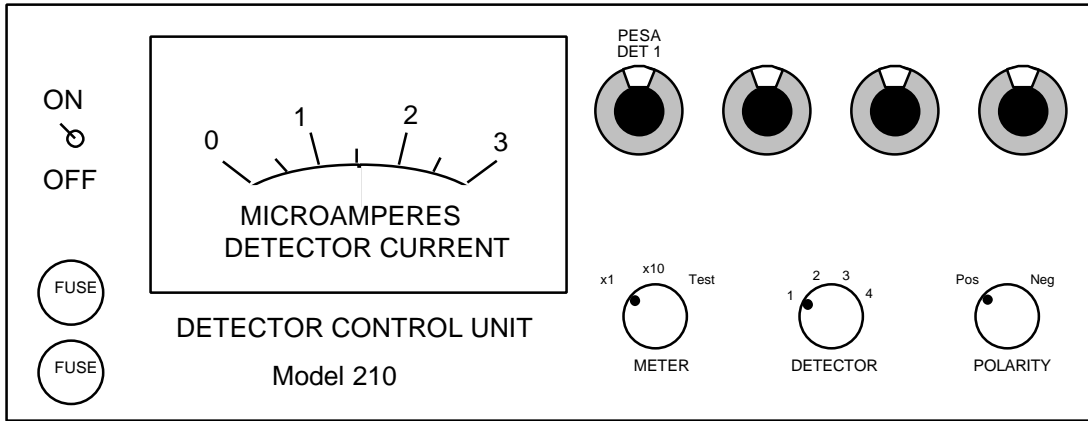
SUPPLIED BIAS VOLTAGE, V_s	$V_s = V + IR$ $V=100V$, $R=10M\Omega$, I =measured value (section 4.3.3)
METER	X1
DETECTOR	1
POLARITY	POSITIVE

4.3.3 Biasing the PESA detector

The procedures for biasing the PESA detector are as follows:

1. Look up the operating voltage on the detector spec sheet.
2. Verify that the bias voltage for the PESA detector is at zero on the Detector Controller Unit, Model 210 (see Figure 22).
3. Verify that the supply is set to positive voltage.
4. Verify that the unit is set to run detector 1.
5. Turn on the Detector Controller Unit using the switch on the left side of the unit.
6. While viewing the signal on an oscilloscope, increase the voltage to 10 Volts. You should hear the noise decrease.
7. Slowly bias up the detector to 100V on the PESA DET dial. If you see voltage spikes, back off the voltage and wait a few seconds before trying again.
8. Because of current leakage through the detector, and voltage losses due to the 10M Ω resistor in the pre-amp, the bias voltage must be increased to actually provide a 100V bias across the detector. To determine the bias voltage necessary to provide 100V across the detector,
 - a) Record the current, I (μ Amps), leaking through the detector from the gauge on the Detector Controller Unit.
 - b) Calculate the V_s necessary to provide $V = 100V$, using $V_s = V + IR$. Use the current I from part a, and $R = 10M\Omega$. Thus, $V_s = 100 + 10*I$.
 - c) Adjust the detector bias to its specified operating voltage (V_s). Read the ORTEC detector document in the back of this manual for further information.
 - d) Record the voltage across the detector (V_s) on the run notes sheet.

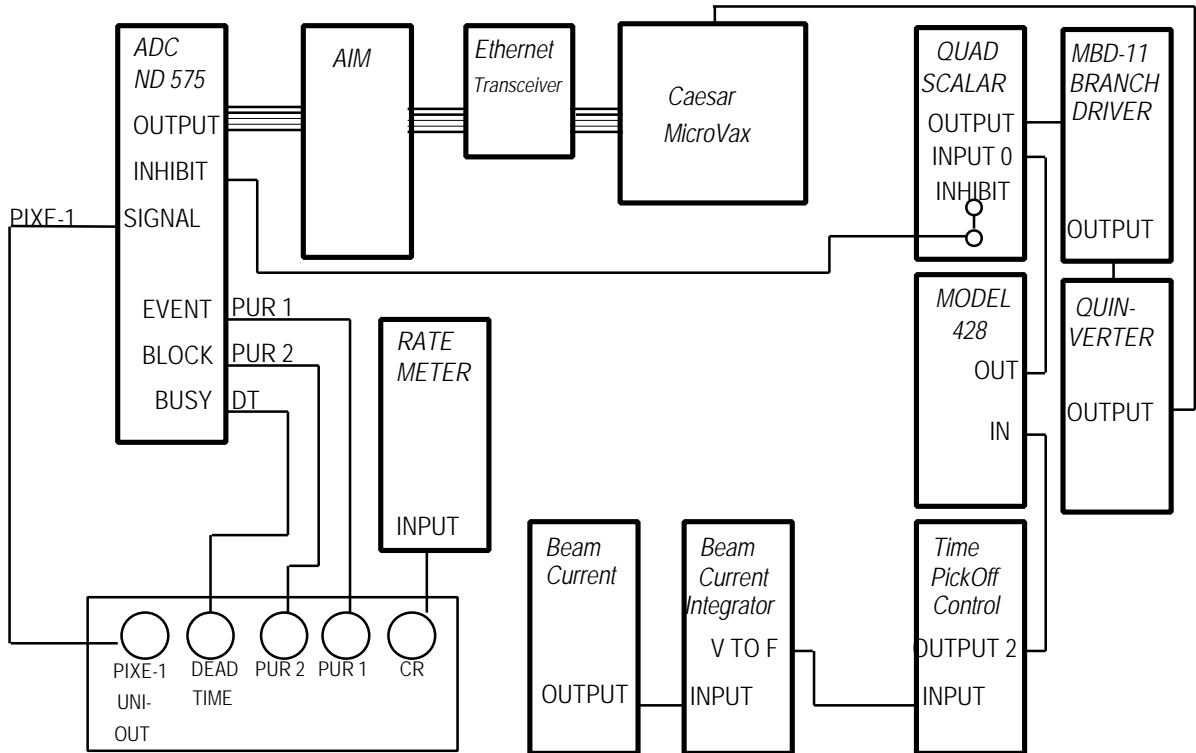
Figure 23 Rack K, PESA Detector Bias



4.3.4 Acquisition Module Wiring

Check the wiring of the acquisition module using Figure 24.

Figure 24 PIXE-1 Data Acquisition Module Wiring Schematic



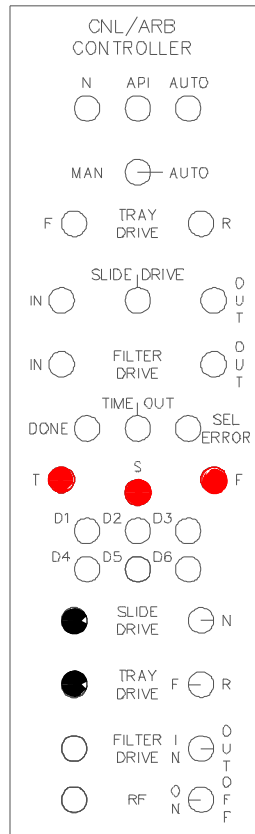
4.3.5 Manual Slide Control

The slide changer can be operated in one of three ways:

1. Computer control using the ACE Program (section 4.4)
2. Manual control using the Slide controller card on the analysis chamber (section 4.2.5.7).
3. Manual control using the CNL/ARB controller located in the computer room, described below.

In the computer room, the manual override controls are located on the CNL/ARB controller module in the electronics rack labeled Module N. A diagram of the CNL/ARB CONTROLLER is displayed in Figure 25.

Figure 25 CNL/ARB Controller



The indicator lights, represented by white circles, are used by the acquisition control program, ACE, to indicate the current status of the slide and tray drives. The meaning of each indicator light is as follows:

- **TRAY DRIVE:**
F indicator lit; tray is moving forward (down) and the tray drive is activated.
R indicator lit; tray is moving reverse (up) and the tray drive is activated.
- **SLIDE DRIVE:**
IN indicator lit, slide is in the X-ray beam.

OUT indicator lit, slide is in the tray.

Center indicator under SLIDE DRIVE lit; the slide is currently moving, or is stuck.

- FILTER DRIVE: not used.
- DONE: the controller is ready for a new command
- TIME OUT: a command was attempted, but did not occur in the allotted time span, and was terminated.
- SEL ERROR: inappropriate or conflicting commands were given. No command will be performed.

The following procedures describe the operation of the CNL/ARB CONTROLLER under manual control. Refer to Figure 25.

1. Verify that ACE is in MANUAL mode, or is not running. If ACE is in AUTO mode, press the number pad key designated ABO to abort the current run and return to MANUAL mode (the coding for the keys on the number pad is displayed on the ACE screen).
2. Flip the switch on the CNL/ARB CONTROLLER from the AUTO to the MAN position to gain manual control.
3. To remove a slide from the beam and replace it in the tray:
 - a) If the TV screen and the indicator light for the slide drive show the slide is in the "OUT" position, the slide is in the tray, not in the beam.
 - b) If the slide appears to be in place on the TV screen, but the indicator light for the slide drive remains in the center position, the slide is stuck somewhere between the tray and the beam position. To move the slide to the out position, see step c part i.
 - c) If the slide is in the "IN" position, push the black button labeled Slide Drive on the lower section of the controller. The Slide Drive should activate, remove the slide, and light the indicator for the "OUT" position.
 - i) If the slide drive light moves to the center indicator, but the slide does not move, or stops moving before reaching the OUT position, push to Slide Drive toggle switch to the "N" position and try step c) again.
 - ii) If the slide drive indicator light remains in the "IN" or center positions, but the slide drive is not moving on the TV monitor, simultaneously press the red button marked "S" and the black Slide Drive button.
 - iii) If this does not remedy the problem, the slide must be moved manually using the slide changer card in the North Cave as described in section 4.2.5.7.
4. To move a slide from the tray and place it in the beam:
 - a) If the TV screen and the indicator light for the slide drive show the slide is "IN", the slide is already in the beam, not in the tray.
 - b) If the slide drive indicator light moves to the center position and remains on, the slide is stuck somewhere between the tray and the beam position. To move the slide to the in position, see step c part i.

- c) If the slide is in the “OUT” position, push the black button labeled Slide Drive on the lower section of the controller. The Slide Drive should activate, move the slide, and light the slide drive indicator for the "IN" position.
 - i) If the center indicator lights and remains lit, push the Slide Drive toggle switch to the "N" position and try step c) again.
 - ii) If the slide drive indicator light continues to read “OUT” or remains in the center position, simultaneously press the red button marked "S" and the black Slide Drive button to override the slide drive.
 - iii) If the slide drive indicator light still indicates “OUT” or is at the center position, the tray drive may not be in position. See step 5 for instructions on moving the tray drive.
 - iv) If the above steps do not remedy the problem, the slide and tray should be moved manually to the correct positions using the slide changer card in the North Cave as described in section 4.2.4.6.
- 5. To move the tray to access another slide:
 - a) Verify that the slide is "OUT" by viewing the monitor screen showing the analysis position, and by checking the tray drive indicator lights on the CNL/ARB controller.
 - b) Toggle the Tray Drive switch on the lower section of the controller to the direction you wish to move the tray (F for forward, R for reverse).
 - c) Press and release the black Tray Drive button on the lower section of the controller. Each time the indicator lights flash, the tray has moved one slide position in the direction indicated in part b). The tray has stopped moving when the DONE indicator light is lit.
 - d) Verify that the tray moved by inserting a slide into the beam, following the procedures of step 4. View the slide on the monitor. If it is the same slide as before the operator attempted to move the tray, the slide drive did not function.
 - i) If the drive did not move, return the slide to the tray using the procedures of step 3, then simultaneously push the red button marked "T" and the black Tray Drive button to override the tray motion system..
 - ii) If the tray drive refuses to move after attempting step i.), you must move it manually in the North Cave using the slide changer card as described in section 4.2.4.6.
- 6. When you have the correct slide in position, toggle the switch on the controller back to "AUTO" mode and continue normally.

4.4 Data Acquisition (Program ACE)

The data acquisition program, ACE, both acquires the x-ray spectra and controls the analysis system. System control involves slide and tray position control, and control of analysis duration and sample identification. ACE was designed to allow manual and automatic control of analysis parameters, such as slide and tray position or sample duration. Manual control, via keypad switching, allows the operator to instantaneously stop or start analysis, move the slide or tray, or save the collected spectra in a data file. Automatic control is usually provided via an instruction file, called a "trayfile," that indicates which positions have valid samples. In automatic analysis, the operator initially positions the slide and tray, sets the analysis duration, and then enters the appropriate trayfile to control the analysis of all the valid samples in the tray (or trays...trayfiles may contain instructions for analyzing up to ten trays in a sequence).

ACE automatically writes a file containing the spectrum, the site and sample date for the sample, plus the system parameters of integrated charge, average detector live time, and count rate, following automatic acquisition of a spectrum. (In manual control, the operator can request that ACE write a similar file.)

During data acquisition, whether in automatic or manual mode, ACE monitors and displays quality control information such as the accumulated charge and the live time and count rate of the detector.

The ACE program has several components that must be working in order to successfully acquire data.

I. Hardware elements of the system include the following:

MicroVAX	- Computer (CAESAR)
AIM	- Acquisition Interface Module
ND575	- Analog to Digital Converter (ADC)
CAMAC CNL/ARB CONTROLLER	- Controller (CTRLR) for slides and trays
CAMAC JORWAY	- Model 84 Quad Scalar (QS)
Video display terminal (VDT)	- Computer (CAESAR)

II. Software elements of the acquisition system:

ACE.EXE

4.4.1 Logging On and Logging Off of ACE

The ACE program must be run from a terminal. The program requires that several software components be installed and ready for execution before it can continue. The ACE program uses Ethernet connections to communicate with the ADC's. The LOGIN file ensures logical variables are set for the acquisition system, and checks the status of the ADC channels for data collection. The ACE program initializes the ND575's, sends start/stop/erase commands, and transfers the data. In the following section, commands to

be typed into the computer will be italicized, material printed on the computer screen will be in bold face type, and descriptions and instructions will be in normal type. Note that <CR> is the carriage return or enter key. The ACE program is started by logging on to a terminal.

Welcome to CAESAR..... Authorized use only!

Username:

Type: *ACE* <CR>

Password:

Type: *ACE* <CR>

Welcome - Crocker Nuclear Laboratory LAN - Caesar

Last interactive login on ...

Last non-interactive login on ...

...

\$PIXE <CR>

\$ Do you wish to recreate the MCA displays Y or N> YES

Now the screen is erased and the display created, as shown in Figure 11.

To get out of the ACE program at any time, press the following keys simultaneously:

<CTRL> <Z >

\$ ACE exiting

4.4.2 ACE Keypad Commands

The box labeled KEYPAD on the right side of the screen in Figure 11 contains the terminal keyboard number pad mapping scheme. All the number pad keys are redefined by the ACE program to provide software interrupts to the acquisition program ACE. When the user strikes a number pad key, the interrupt is queued to the ACE program and will be serviced when other input or output is completed. It is possible to queue multiple commands, but this should be done with caution.

An expanded view of the keypad box in Figure 11, follows in Figure 12. In Figure 12, the top row in each field is the code shown on the ACE screen, the second row is the number pad key to which the code has been mapped, and the third row is the function for which the code is an abbreviation. Pressing a number pad key while the ACE program is running will activate the command mapped to the key pressed. Some commands will be instantly executed while others will request further information. The requests will appear in the command window below the list of options. Use the regular keyboard to enter requested information. Details and examples will be given later.

Figure 26 ACE Control Program Screen

Date: 24-Sep-96		PIXE		Time: 11:39:46																					
Run No :	Ctrlr Stat:	<table border="1"> <tr> <th colspan="4">KEYPAD</th> </tr> <tr> <td>ENT</td> <td>SLF</td> <td>SLR</td> <td>ABO</td> </tr> <tr> <td>ACQ</td> <td>STP</td> <td>REC</td> <td>CHG</td> </tr> <tr> <td>RNO</td> <td>CNV</td> <td>T&S</td> <td>ERA</td> </tr> <tr> <td>TIM</td> <td>INT</td> <td>SCR</td> <td></td> </tr> </table>				KEYPAD				ENT	SLF	SLR	ABO	ACQ	STP	REC	CHG	RNO	CNV	T&S	ERA	TIM	INT	SCR	
KEYPAD																									
ENT	SLF					SLR	ABO																		
ACQ	STP					REC	CHG																		
RNO	CNV					T&S	ERA																		
TIM	INT					SCR																			
Trayfile:	VAX - MCA :																								
Position:	Acq Start :																								
Profile :	Preset Tim:																								
Sample #:	Elapsed :																								
Cnv Factr:	Min current																								
Renol:	Reno2:																								
Charge :																									
Tray name:																									
Mode:		Command:																							
AUTO																									
Run	Master	Pos	Profile Id	Convf	Table	Charge	Sample desc																		

Figure 27 ACE Program Keypad Mapping

ENT PF1 Enter Auto	SLF PF2 Slide Forward	SLR PF3 Slide Reverse	ABO PF4 Abort Auto
ACQ 7 Start Acquisition	STP 8 stop	REC 9 Record Date	CHG - Minimum Charge
RNO 4 Renormilization	CNV 5 Conv. Factor	T & S 6 Tray and Slide	ERA + Erase Data
TIM 1 Enter Pre-Set Time	INT 2 Change Intg Scale	SCR 3 Re-draw Screen	ENTER
0		.	

The current definitions for the codes used in the Keypad display are listed in order below:

ENT = Enter automatic mode. This initiates the ACE program for automated sampling. The ACE program will request a command file, called a trayfile, containing information on which slides should be analyzed and which position is the last position in the tray. The trayfiles are described in section 4.4.2. The ACE program will then automatically run each valid slide in the command file for the duration of time listed on the ACE program screen as Preset Time.

SLF = Move to the slide located n positions in the forward direction (for use in MANUAL mode). You will be prompted for the number of positions, n, to move the tray. Once n is entered, the current slide will be removed, the tray will increment forward n positions, and the slide in the position at which the tray stops will be moved into the beam.

SLR = Move to the slide located n positions in the reverse direction (for use in MANUAL mode). You will be prompted for the number of positions, n, to move the tray. Once n is entered, the current slide will be removed, the tray will move back n positions, and the slide in the position at which the tray stops will be moved into the beam.

ABO = Abort the automatic mode. ACE will discontinue acquisition, clear the temporary data storage locations, and return to Manual mode.

ACQ = Acquire data (for use in MANUAL mode). Press this button to begin acquisition of data on the current slide. The preset time is not in effect in this mode. You may acquire for as long as you want. The counterpart to this command is STP which stops acquisition.

STP = Stop data acquisition (for use in MANUAL mode). This command allows the user to stop the ND575 from acquiring data.

REC = Record data (for use in MANUAL mode). This allows the user to save the current data for subsequent to analysis by RACE.

SCR = Redraws the screen.

RNO = Enter renormalization factor for the PIXE-1 and PIXE-2 detectors. The number entered is renormalization * 1000 and must be in the range of 100 to 2000. If the entered number is out of range it will be rejected, and the user will be prompted to enter a value within range.

CNV = Enter conversion factor (for use in MANUAL mode). This allows the user to enter a conversion factor for analysis being done using ACQ. (In the auto mode the conversion factor is extracted from the trayfile.)

T&S = Enter tray and slide numbers. This function allows the user to enter a tray number and a position to be added to a manually collected output data file. It is used with the ACQ function.

ERA = Erase the spectra in the ND575 and clear the quad scalar (for use in MANUAL mode). This function allows the user to clear all data in the ND575, and zeroes the accumulated charge measurement on the quad scalar.

TIM = Enter the preset time (for use in AUTO mode). This function allows the user to change the preset time for which the samples will be analyzed.

INT = Enter the integrator scale factor (not used during PIXE analysis).

4.4.3 Default ACE Settings

To start ACE for a typical IMPROVE analysis session, the following parameters should be used and monitored.

1. Set the analysis duration, TIM to 50 seconds. This is the standard filter analysis duration. More time may be required for thick filter deposits to accumulate adequate spectra, so this preset time is designed to be superceded by the minimum charge requirement.
2. Set the minimum charge to 500. The charge accumulation on the filter should be above 500 $\mu\text{Coulombs}$. This is displayed on the ACE analysis screen. Accumulated charge is a good indicator for collection of usable spectra. Until the minimum charge is collected for each analysis, ACE will disregard the preset analysis time setting.
3. Enter a tray file to automatically control ACE. See section 4.4.4 for usage examples, and 4.4.5 for trayfile descriptions.
4. Monitor the beam current. This is displayed on the Beam Current Integrator (see Figure 18). This should be at least 2 nanoAmps. If it is consistently low, or is fluctuating with an amplitude greater than 2 nanoAmps, the cyclotron beam may not be tuned properly. The operator should be notified.
5. Monitor the count rate. The count rate measures the number of x-rays produced during PIXE analysis. If the cyclotron beam loses its tuning, the power fluctuates, or the new filter substrate is substantially thicker or thinner, the count rate will change. The count rate should be about 2000 counts per second.
 - a) If the count rate is too high, the detectors will not be able to resolve the emitted x-rays. The detectors require at least 8 μsec to process each incoming x-ray, and can only process one x-ray at a time. When an x-ray is being processed, all other incoming x-rays will be disregarded. If, once the detector is ready to receive an x-ray, it receives two x-rays at once, it will attempt processing, but will be forced to discard both. This is a waste of beam and processing time. The measurement of how effective the detectors are is called live time. It is displayed on the ACE analysis screen (as dead time = 100% - live time). Live time is the percentage of time during a sample analysis in which the detector was processing viable data.
 - b) If the count rate is too low, not enough x-rays will be received to produce a viable spectrum. The operator must optimize the count rate such that the live times of the two detectors, PIXE-1 and PIXE-2, range between 80 and 95 percent. PIXE-3, the PESA detector, should have a live time of between 60 and 80 percent.

6. Verify that the filter being analyzed in the beam (shown on the monitor screen) matches the filter descriptor on the ACE analysis screen.

4.4.4 ACE Usage Examples

1. Manual data acquisition.
Move the tray forward 5 positions.
Start the acquisition.
Stop the acquisition at the appropriate elapsed time.
Record the data in the computer.
Erase the data in the Multi-Channel Analyzer.
Move to the next slide to be analyzed and begin the process again.
2. Automatic data acquisition
Set analysis time to 700 seconds
Start automatic data acquisition for SNPA1 in B96 quarter

If necessary to stop acquisition, then
Abort automatic analysis

The keystrokes to perform the analysis above are shown below. Note, the commands in italics are requests for more information by the ACE program, but the commands in boldface type must be entered by the user.

1. Manual Data Acquisition

SLF (slide forward), *positions forward: 5* <CR>
ACQ (acquire)
STP (stop acquisition)
REC (record data)
ERA (erase data)
SLF (slide forward), *positions forward: 1* <CR>

2. Automated Data Acquisition

ENT (enter AUTO mode), *tray name:SNPA1A1T.B96* <CR>
position: 1 <CR>
TIM (enter preset time), *preset time: 50* <CR>

If necessary to stop acquisition, then use

ABO (abort AUTO mode), *aborting auto mode*

4.4.4 Trayfiles

The automatic mode of ACE is guided by the presence of a trayfile. The trayfile is a computer file which represents the order of samples for analysis, and contains additional information about the samples in the tray. This information is necessary in order to

perform a complete analysis of the sample. When ACE is set to begin AUTO mode it checks for the presence of a trayfile. If the trayfile is present under the name given by the operator, the code requests the operator enter the current positioning the tray. Once the position is entered, ACE uses the trayfile as a guide for which tray positions contain slides for analysis. The data for each particular slide is read in turn as the tray is analyzed. The information that can be provided in the tray file is:

- A. Sample position number. The sample position is a number generated to aid in organizing the trayfile (as a trayfile may be constructed to sequentially run several individual trays).
- B. Sample conversion factor. This field may contain the actual conversion factor (derived below), or one of three default settings; 0, -1, and 1000. The conversion factor relates the filter loading in $\mu\text{g}/\text{cm}^2$ to the aerosol concentration of the sampled air in $\mu\text{g}/\text{m}^3$.

- Conversion factor = $\text{Mask} * \left(\frac{1000}{ET * Flow} \right)$

Mask (mask size) [cm^2],

ET (elapsed time) [minutes],

Flow (Flow rate) [liters/minute ambient]

- Conversion factor = 0 indicates the position is not to be analyzed; is a skip position command.
 - Conversion factor = -1 indicates the current position is the end of the analysis tray; is a stop automatic acquisition command.
 - Conversion factor = 1000 indicates a normal sample. It is used as a default value during analysis before the conversion factors have been compiled and verified.
- C. Sample identification number. Position of the identified slide in it's slide tray, except for filter blanks. For blanks, the sample identifier is set to 0, no matter what the tray position. Zero as a sample identification number has a special significance in the analysis programs; namely the data is subtracted from subsequent samples as a representation of a blank substrate with no deposit.

D. Profile number. The profile number is the analysis quarter name converted to decimal code.

Analysis Quarter	ASCII code	Decimal Code
A: March - May	A	65
B: June - August	B	66
C: September -November	C	67
D: December - February	D	68

E. Sample Description. IMPROVE network samples use this field for filter mask size; the area, in cm^2 , of the aerosol deposit on a filter. However, it can be used for ASCII text.

Filter Mask	Mask Area (cm^2)
MO	3.51
M1	2.2
M2	1.1

F. Matrix correction table name. PIXE data correction table correcting for the 'shadowing effect' of particles on filters. Correction tables were derived in the 1970's through experimentation with a variety of substrates and deposits to derive values for expected filter and loading types.

G. Profile description. Location for putting in the study name, source, etc. of the associated filter.

H. Unused Fields. Each 0 represents an unused field. The unused fields, in order, are sample mass, error in mass, laser value (for B_{abs}), laser error, beta gauge value, and error in the beta gauge value. These fields are no longer included in the IMPROVE trayfiles.

I. Site name. The site name is a five character code representing a site. The first four characters are an abbreviation of the site name. The fifth character is a number.

J. Filter type. The filter type is A1 or D1. A1 indicates a Teflon filter with a sample deposit consisting of particles $\text{PM}_{2.5}$ particles. D1 indicates a Teflon filter with a sample deposit consisting of PM_{10} particles.

K. Sample date. The sample date is the date upon which the filter deposit was collected.

L. Start time. The start time is when the collection of the filter deposit began, using a 24 hour clock.

M. Elapsed time. The elapsed time is the duration of the sample collection period.

- N. Flow rate. The flow rate for air through the filter during sampling is recorded in liters per minute of ambient air.
- O. Filter status. The filter status, generally NM or normal, indicates whether a filter represents a valid sample. The conversion constant controls whether the filter will be automatically analyzed. The status merely provides a visual check of scheduled analyses.

For the IMPROVE network, the Spectroscopist generally creates the trayfiles. Trayfiles are named according to the sampling site, filter type, and analysis quarter. The file names can contain from 1 to 9 characters, with a three character extension. The IMPROVE network files generally contain 8 digits with a three digit extension. The file SNPA1A1T.B96, for example, is for the Snoqualmie Pass site, for module A Teflon filters that were collected during the B96 quarter.

The minimum trayfile must include the first two items (A,B) in the above list. Items left out of the trayfile will be defaulted or incremented automatically by the program. Trayfiles are ended by inserting line containing a position number and -1 as the conversion factor. An example of a minimum trayfile is:

A	B	I	N
1	12345	54321	23.2
20	-1		

The above trayfile represents a physical tray with 19 samples. All samples will be analyzed with the same conversion factor, 12345. The sample position and sample identifier are the same and each will be incremented automatically until 19. There is no sample description so all samples will be described automatically with the word SAMPLE and the identifier number. There is no matrix correction table name so RACE will not perform matrix correction effects. There is no profile description so the profile will be described automatically with the profile number. There is no mass, laser or beta values so they will be zeroed in the data. The filter type, start time, elapsed time, sample date, and filter status are not recorded. However, the site name is listed as 54321, and the flow rate is shown as 23.2 liters per minute.

If the user wants control of some more of the analysis a better example of a trayfile might be:

```
1 12345 0 'BLANK' 'SFU2' 'GRAND CANYON FOR 1ST QUARTER 1988' 54321
2 12345 1 'FIRST DAY' 'SFU2' 54321
20 -1
```

In this example, the first row (filter position) contains A,B,C,E,F,G,I. The sample identifier, C, for position 1 is zero, indicating the filter is a blank. The second filter

position, the first real sample, contains A,B,C,E,F, and I. For this trayfile, the blank and first real sample will have sample descriptions; the information in single quotes will be appended to the output data. However, the analyses for the samples in positions three through nineteen will be identified with the same trailer as position 2. This is because the trayfile will not end until it sees a -1 as a sample identifier. So, ACE will continue incrementing through the tray, analyzing one position at a time, using the last trailer as a template for the current analysis trailer.

For the IMPROVE network analyses, the trayfiles generally appear as shown below in Table 15. The columns are labeled to reflect the list derived earlier of data that can be incorporated into a trayfile.

Table 15 Sample IMPROVE Trayfile

A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1	9994	1	66	'M0'	'FINE'	''	0 0 0 0 0 0	'SAGO1'	'A1'	'06/01/96'	0000	24.00	24.5	'NM'
2	10035	2	66	'M0'	'FINE'	''	0 0 0 0 0 0	'SAGO1'	'A1'	'06/05/96'	0000	24.00	24.6	'NM'
3	9872	3	66	'M0'	'FINE'	''	0 0 0 0 0 0	'SAGO1'	'A1'	'06/08/96'	0000	24.00	24.2	'NM'
4	9954	4	66	'M0'	'FINE'	''	0 0 0 0 0 0	'SAGO1'	'A1'	'06/12/96'	0000	24.00	24.4	'NM'
5	9954	5	66	'M0'	'FINE'	''	0 0 0 0 0 0	'SAGO1'	'A1'	'06/15/96'	0000	24.00	24.4	'NM'
6	9627	6	66	'M0'	'FINE'	''	0 0 0 0 0 0	'SAGO1'	'A1'	'06/19/96'	0000	24.00	23.6	'NM'

4.5 Spectral Analysis (Program RACE)

The x-ray spectra produced during PIXE and PESA are analyzed by the program RACE. Prior to analysis of filter samples, the following must be provided:

1. System blank. X-ray spectral analysis requires a system blank to estimate the incremental addition to the spectrum due to the presence of the filter substrate. The system blank is derived by analyzing several clean filters, 'blanks', at the beginning of an analytical session. The cleanest filter is selected to become the system blank. The spectrum from the system blank is smoothed and stored on disk for subtraction from the spectra of subsequent analyses.
2. PIXE system calibration. In order to identify the peaks it is necessary to provide an energy calibration between the energy of the x-ray line and the spectral channel. This is done at the beginning of the analytical session using the thin film standards, and the reanalysis filters. The standards are analyzed, the peak locations are noted, and the MCA channel and x-ray energy of each elemental peak is entered into a calibration table. The x-ray energies for each element are looked up in standard x-ray tables. Four elements are generally used. For an in depth description of the process, see TI 326A.
3. PESA system calibration. In order to relate the hydrogen peak characteristics to hydrogen concentrations in filters and deposits, a series of calibration films are run. These films are of clean Mylar™ of various densities. The calibration uses Mylar™ films since Teflon, the material used in the standard filter substrates analyzed in PIXE and PESA, contains no hydrogen.
4. Cross section table. To calculate the elemental concentrations on filters using PIXE requires knowledge of the relationship between the area under a peak and the elemental concentration. This information is contained in a cross section table, which gives the line identification (e.g. Fe K α), the line energy, the product of cross section and detector efficiency, and the atomic weight of the element. The cross section-efficiency numbers for this table are determined during system calibration, by analyzing a series of 30 single element, double element, and multi-element thin film standard foils from a commercial vendor. In order to determine the cross sections for elements not included in the standards, and to verify that a given standard is correct, the cross sections are plotted against the atomic number. The resulting smooth curve allows the calculation of cross sections for non-measured species, and reduces the uncertainty for a given line to much less than the 5% quoted for a given foil. This system calibration is performed whenever the system is reconfigured, including following detector replacement. It is also performed if the calibration checks made every quarter at the beginning of each analytical session indicates that the system has changed. The system calibrations for derivation of cross section tables are conducted by the Spectroscopist under the direct supervision of the Quality Assurance Manager. See TI 326A for more information.

The steps followed by RACE in performing elemental analysis are:

1. Remove most of the spectral background by subtracting the system blank. This removes the portion of the spectrum due only to the presence of the filter substrate. To achieve the best fit, the system blank is floated by a multiplicative factor until a preset number of channels are exceeded. The operator can adjust the factor by changing the number of channels required.
2. Remove any remaining background spectrum by subtracting a smooth function calculated as the lesser of the counts in a channel and the average counts per channel over a specified width. To change this fit, the operator can adjust the width and the number of iterations of the process.
3. Use a peak search routine to identify the peaks and valleys by comparing the shape of the spectrum near a given channel to what would be expected if there were a peak at that channel.
4. Fit each peak to one or more Gaussians. Using the centroid of the peak and the energy calibration, determine the energy of the peak. Identify the line using a "cross section table." With the area of the Gaussian, the "cross section" from the table, and the beam charge and detector live time for this analysis, calculate the concentration in ng/cm² of the element on the filter. For peaks close to two lines, calculate the concentration for each line as if it were the only line.
5. Determine the uncertainty of the concentration from the uncertainty in the Gaussian area. For a well-shaped peak this is the standard Poisson value based on the number of counts in the peak and background. For poorly shaped peaks the uncertainty can be somewhat larger. The uncertainty is expressed as ng/cm².
6. Calculate the minimum detectable limits for all elements in the cross section table. Use 3.29 times the square root of the background counts under the region that would have been occupied by a peak of normal width. Minimum detectable limits are expressed in ng/cm².
7. Resolve the interferences between the various lines, using preset criteria. The most common interference is when the β line of one element overlies the α of the next element. The concentration for the α line of the next element can be determined by subtracting the contribution of the lighter element using its α value.

The interference between Pb L α and As K α is resolved using the β lines of both.

The RACE program for data processing requires a VAX/VMS micro-station, a graphics terminal, and a printer. RACE must be installed and active before it can analyze data. ACE and RACE are often run simultaneously during analysis to allow the operator to view the initial analysis of the collected spectra for quality control.

4.5.1 Logging onto RACE

In the following section, commands to be typed into the computer will be italicized, material printed on the computer screen will be in bold face type, and descriptions and instructions will be in normal type. The RACE program is started by logging on to the MicroVAX console

<CR>

Welcome to CAESAR VAX/VMS V5.4-2..... Authorized us

Username:

ACE <CR>

Password:

ACE <CR>)

Welcome - Crocker Nuclear Laboratory LAN - Caesar - VAX/VMS V5.4-2

Last interactive login on ...

Last non-interactive login on ...

...

\$CD RACE\$COM <CR>

Moves the user to the RACE directory, `asg.com.race`, which contains all the data, default parameters, and pointers for each analysis run.

asg.com.race->@STANDSET <CR>

This will set the defaults for RACE to the current (or last) settings used in the PIXE and PESA analysis run. Defaults include data storage locations for input and output files, and system calibrations. To reprocess an archived analysis run instead of the current analysis run, follow the procedures in section A, below.

A. To reprocess an archived analysis run, type, at the `asg.com.race` prompt,

asg.com.race->DIR <CR>

This will display all the command files for setting the defaults for RACE. The files, other than `STANDSET.COM` will be named `RACEqtr.COM`, where `qtr` is the quarter the analyzed data was collected in. Examples of `qtr` are `A96` for March through May of 1996, `B96` for June through August of 1996, `C96` for September through November for 1996, and `D96` for December of 1996 through February of 1997. The user will enter the command file for the quarter to be emulated. For `A96` analysis, as an example, the user would enter the following:

asg.com.race->@RACEA96 <CR>

The user may start the RACE program at any prompt. Here, as an example, RACE is started in the `asg.race` directory.

asg.race->VAXRACE

This will start the interactive RACE program on the terminal or console being used.

WARNING: NO CALIBRATION TABLE RACE Exiting

asg.src.race->

If the warning above appears, there is no calibration table for RACE in the default directory. RACE will not function until a calibration table is installed in the appropriate directory (specified by the default program...usually STANDSET during analysis).

Archived quarters should have calibration tables; if a calibration table is missing for an archived quarter, contact the quality assurance manager for assistance.

Note that if there is a TABLEXRAY.PX1 file in the asg.qtr.data.race.input.tables\ directory, (where qtr is the quarter being analyzed), it will be used by RACE in preference to the TABLEXRAY.XX1. Thus, the TABLEXRAY.PX1, TABLEXRAY.PX2, and TABLEXRAY.PX3 files are generally renamed to CAN1date.DAT, CAN2date.DAT, and PESAqtr.DAT where date is a five digit code for the month and the year, and qtr is the analysis quarter. RACE for PIXE analysis will not function until these files are renamed to TABLEXRAY files.

If the current analysis does not have a calibration table, contact the quality assurance manager or the Spectroscopist for assistance.

If RACE is installed, and the input files are present, the following will appear on the screen:

First Run to Analyze (^Z to Exit):

Pressing the <CTRL> and <Z> keys simultaneously, or pressing the <ESC> key twice will exit the RACE program and return the user to the asg.src.race cursor.

To continue in RACE, the number of the first run to be analyzed, for example 23142, must be entered. Then,

1. RACE will look for the ACE output files labeled 23142.PX1, 23142.PX2, and 23142.PX3 where
 - a) PX1 is the file extension indicating the file contains PIXE-1 detector data.
 - b) PX2 is the file extension indicating the file contains PIXE-2 detector data.
 - c) PX3 is the file extension indicating the file contains PESA detector data
2. When RACE finds the file 23142, it will look at the file extension for that run file. If the extension is for PIXE or PESA analysis, (PX1, PX2, or PX3), RACE will input the PIXE and PESA analysis default settings.

NOTE: For a given filter, the PIXE1, PIXE2 and PESA files will have the same run numbers, but XRF files will have a different run number. No XRF file in an analysis quarter can have the same run number as any PIXE or PESA file in that quarter or RACE will not execute properly.
3. Assuming the file extension was for PIXE and PESA analysis, RACE will begin analysis of the input file using the standard defaults for display and output, as listed in section 4.5.2.

4. With the standard settings (default), RACE will display, for each run analyzed, the spectral peaks found, the concentrations (in ng/m³) and error (in ng/m³) for each species found, and the correction factor. A sample output is shown in Figure 28.
5. Following the reporting of the peaks found and elemental concentrations, RACE will list the calibration and matrix tables used to derive the output data. These data, seen at the bottom of Figure 28, are described in section 4.5.3.

Figure 28 RACE Analysis Sample Output

9-DEC-1996 11:18:50 PX ..START-I-ENERGY CROSS SECTION TABLE CAN1NOV96
 UC DAVIS PIXE-1 ANALYSIS RUN 78225. PX1 FILE 66 ITEM 12 27-SEP-96
 PORE1A1 07/10/96

FILPRO=PORE1A1B.96A

CONCENTRATIONS OBTAINED BY DIVIDING AREAL DENSITIES BY 14.727 M**3/CM**2

MATRIX CORRECTIONS USING TABLE FINE

UNCERTAINTIES ARE STATISTICAL PLUS A 5% OVERALL UNCERTAINTY, ADDED QUADRATICALLY

	ELEMENT		NANOGRAMS PER M**3		CORRECTION
1	NA	K	822.35	± 54.65	1.471
2	SI	K	39.24	± 4.74	1.194
3	S	K	1043.15	± 54.59	1.056
4	K	KA	45.91	± 4.06	1.049
5	CA	KA-	43.11	± 3.62	1.041
		KB	62.96	± 9.21	1.041
6	TI	KA	4.69	± 1.08	1.029
7	FE	KA	8.16	± 1.28	1.016

9-DEC-1996 11:20:50 PX ..START-I-ENERGY CROSS SECTION TABLE CAN2NOV96
 UC DAVIS PIXE-2 ANALYSIS RUN 78225. PX2 FILE 66 ITEM 12 27-SEP-96
 PORE1A1 07/10/96

FILPRO=PORE1A1B.96B

CONCENTRATIONS OBTAINED BY DIVIDING AREAL DENSITIES BY 14.727 M**3/CM**2

MATRIX CORRECTIONS USING TABLE FINE

UNCERTAINTIES ARE STATISTICAL PLUS A 5% OVERALL UNCERTAINTY, ADDED QUADRATICALLY

	ELEMENT		NANOGRAMS PER M**3		CORRECTION
1	NA	K	1258.28	± 181.98	1.514
2	S	K	985.52	± 54.89	1.061
4	K	KA	41.23	± 3.68	1.052
5	CA	KA-	36.34	± 3.37	1.043
6	MN	KA	0.91	± 0.38	1.010
7	FE	KA-	6.58	± 0.97	1.016
		KB	8.51	± 2.44	1.016

9-DEC-1996 11:20:50 PX ..START-I-ENERGY CROSS SECTION TABLE PESAAPR95
 UC DAVIS PIXE-3 ANALYSIS RUN 78225. PX3 FILE 66 ITEM 12 27-SEP-96
 PORE1A1 07/10/96

FILPRO=PORE1A1B.96B

CONCENTRATIONS OBTAINED BY DIVIDING AREAL DENSITIES BY 14.727 M**3/CM**2

MATRIX CORRECTIONS USING TABLE FINE

UNCERTAINTIES ARE STATISTICAL PLUS A 5% OVERALL UNCERTAINTY, ADDED QUADRATICALLY

	ELEMENT		NANOGRAMS PER M**3		CORRECTION
1	H		162.45	± 10.52	1.000
2	C		2145.94	± 108.62	1.000

4.5.2 Setting RACE Analysis Parameters

Once RACE has imported the default settings, RACE will begin sequential analysis of the data in each analysis run file. The default analysis parameters, shown in the Analysis Parameter Input screen in Figure 14, will be applied to each run to be analyzed. To change the analysis parameters, press the PF4 key. NOTE: Before pressing the PF4 key to access the parameter input screen, always make sure the program is either analyzing, printing a list of elements, or printing a spectrum on the screen. Never press PF4 when the program is asking a question as this will cause RACE to abruptly discontinue the output file, and exit. After pressing the PF4 key, the screen is erased and a new image appears, as shown in Figure 29.

Figure 29 RACE Analysis Parameter Input Screen

Analysis Parameter Input				(1792)
(1)	CHECK BLANK <u>F</u> LOAT	OFF	(2)	CHECK <u>B</u> ACKGROUND FIT OFF
(3)	CHECK THE PEAK <u>W</u> IDTH	OFF	(4)	BEGIN <u>N</u> EW SEQUENCE OFF
(5)	SQUARE ROOT <u>T</u> PLOT DATA	OFF	(6)	WRITE <u>A</u> LL FULL OUTPUT OFF
(7)	WRITE <u>R</u> EDUCED OUTPUT	OFF	(8)	ENTER NEW <u>C</u> ALIBRATION OFF
(9)	NO AUTO <u>O</u> RECALIBRATION	ON	(10)	NO PREF FOR <u>P</u> B OVER AS ON
(11)	NO GRAPHICS <u>D</u> ISPLAY	ON	(12)	<u>M</u> INIMUM DISPLAY OUTPUT OFF

* Enter the switch letter to be toggled : → Exit Parameter Input Screen
(PF4 to exit VAXRACE)

The screen in Figure 29 represents a switchboard. Each option can be either On or OFF. The default setting has all the switches off except for O, P and D (switches 9, 10, and 11) To toggle a switch, press the highlighted letter for the desired operation. To toggle switch 10, for example, press P. Each option influences the final data output; definitions for the options follow.

Toggle	Toggle Switch Label	Switch Activity
F	Check Blank Float:	Enables you to change the blank subtraction.
B	Check Background Fit:	Enables you to change background fit
W	Check Peak Width:	Enables you to change accepted peak width
N	Begin new sequence:	Enables you to start analysis from another run
T	Square Root Plot Data:	Plot data as square root plots
A	Write all full output:	Informs computer to print all data on samples
R	Write reduced output:	Informs printer to print reduced data
C	Enter new calibration:	Enables input of energy calibration data
O	No Auto re-calibration:	Will not re-calibrate automatically
P	No pref. for Pb over As:	Computer will treat both equally when found
D	No graphics display:	No spectral plots to screen

M	Minimum display output	Does not output data or plots to screen
X	Exit back to RACE:	Get back to RACE exactly where left off

Enter the switch letter to be toggled:..... Self explanatory

If a file is analyzed by RACE with the wrong parameters set, the file can be reanalyzed. However, RACE will append the earlier output file with the new information, rather than writing over the old file. To avoid creating large, confusing output files, the parameters are generally set before the first analysis is run. Since RACE works rapidly, if the operator entered a valid run number as the first run to analyze, RACE would nearly have completed analysis before the operator could press the PF4 key to change the parameters. Thus, the first file would have to be reanalyzed following the setting of the parameters. To avoid reanalyzing the first file, the operator enters a dummy run number, 99999. While RACE is searching for the non-existent input file for the dummy run number, there is time to press the PF4 key and change the parameters, including the start new sequence parameter. A sample RACE analysis start-up including changing the parameters follows.

asg.src.race->RUN VAXRACE

First Run to Analyze (^Z to Exit):99999

Waiting for run 99999 (Hit PF4 for parameters) <PF4>

Analysis Parameter Input				(1792)
(1)	CHECK BLANK <u>F</u> LOAT	OFF	(2)	CHECK <u>B</u> ACKGROUND FIT OFF
(3)	CHECK THE PEAK <u>W</u> IDTH	OFF	(4)	BEGIN <u>N</u> EW SEQUENCE OFF
(5)	SQUARE ROOT <u>P</u> LOT DATA	OFF	(6)	WRITE <u>A</u> LL FULL OUTPUT OFF
(7)	WRITE <u>R</u> EDUCED OUTPUT	OFF	(8)	ENTER NEW <u>C</u> ALIBRATION OFF
(9)	NO AUTO <u>R</u> ECALIBRATION	ON	(10)	NO PREF FOR <u>P</u> B OVER AS ON
(11)	NO GRAPHICS <u>D</u> ISPLAY	ON	(12)	<u>M</u> INIMUM DISPLAY OUTPUT OFF

*** Enter the switch letter to be toggled :..... → Exit Parameter Input Screen (PF4 to exit VAXRACE)**

When a key representing one of the switches is pressed, the switch status will toggle. For example, pressing D, R, and N will instruct RACE to, in future analyses, display the graphics (spectra), write a shortened version of the output data to the line printer, and to not automatically analyze more than one run. To return to RACE, the operator will press X to exit the input screen. If the first valid analysis is for run 34336, the operator will now enter that run number.

First Run to Analyze (^Z to Exit):34336

The operator should now immediately press the PF4 key (the - key on the number pad), to return to the parameter input screen and turn off the begin new sequence switch by pressing the N key. Finally, exit back to RACE by pressing X to exit the analysis parameter input screen.

If the begin new sequence key is not turned off, after completing the analysis for each run, RACE will request that the operator enter the next run number to be analyzed.

4.5.3 RACE Calibration and Matrix Tables

The parameters displayed at the bottom of Figure 13 are the factors necessary to process spectral data using the RACE program. AX and BX refer to the slope and intercept of the best fit to the calibration equation for each detector relating the MCA channels to the x-ray energies. The calibration is derived in two steps, the coarse fit from the standards tray analysis, and the fine tuning from filter sample reanalysis. KOVER has a value of 1 and represents the percentage that the blank will be allowed to ride over the sample data. NTHICK has a value of 100 and represents the sample substrate thickness. JITER is a correction factor for broad spectrum background radiation from the loading on the filter. Here it involves a smoothing function using 17 iterations. WIDTH has a value of 9 and refers to the minimum width in MCA channels at half a suspected peak's height that it must have before RACE classifies it as a valid elemental species peak. The procedures to derive these factors are as follows.

1. To derive the rough calibration for the PIXE system,
 - a) Request that the cyclotron controller provide a 9MeV proton beam with approximately 2000 counts per second reaching the PIXE-1 detector (approximately 5 nanoAmps). Analysis of standards requires a lower current.
 - b) Set the analysis time to 50 seconds per sample using the ACE key pad (see section 4.4.2).
 - c) Prepare to analyze the PIXE standards tray containing 27 Micromatter™ thin film standards (Pb, CuS, NaCl, Mg, Al, SiO, GaP, CuS, KCl, KI, CaF₂, Ti, V, Cr, Mn, Fe, Ni, Cu, ZnTe, GaAs, Se, CdSe, CsBr, Au), 6 Mylar™ standards (positions 29 through 34) for the PESA calibration, and 2 clean filters (blanks) representative of the filter lot used for IMPROVE aerosol sampling.
 - d) RACE will derive elemental concentrations from the spectral data.
 - i) RACE uses a detector specific cross section table for analysis. A cross section table must be derived for each detector in each system. The derivation and purpose of the table are described in the article “Automatic data acquisition and reduction for elemental analysis of aerosol samples” by J.F.Harrison and R.A.Eldred, published in Advances In X-Ray Analysis, Vol 17, Plenum Press, New York, NY.
 - ii) RACE uses a peak search routine to identify the peaks and valleys by comparing the shape of the spectrum near a given channel to what would be expected if there were a peak at that channel.

- iii) RACE fits each peak in the spectra to one or more Gaussians. Using the centroid of the peak and energy calibration, the energy of the peak is determined. The line is identified using a "cross section table." Using the area of the Gaussian, the "cross section" from the table, the beam charge and detector live time for this analysis, the concentration in ng/cm² is calculated. If the peak is close to two lines, calculations are made for each line as if it were the only line.
 - iv) RACE calculates the uncertainty of the concentration from the uncertainty in the Gaussian area. For a well-shaped peak, this is the standard Poisson value based on the number of counts in the peak and background. For poorly shaped peaks, the uncertainty can be somewhat larger. This is expressed as ng/cm².
 - v) RACE calculates the minimum detectable limits for all elements in the cross section table as 3.29 times the square root of the background counts under the region that would have been occupied by a peak of normal width. This is expressed as ng/cm².
 - vi) RACE resolves interferences between the various lines, using preset criteria. The most common is when the β line of one element overlies the α of the next element. The concentration for the α line of the next element can be determined by subtracting the contribution of the lighter element using its α value. The interference between Pb L α and As K α is resolved using the β lines of both.
- e) RACE will report estimated elemental concentrations of the certified species for each standard thin film sample. Record and compare these values with the certified concentrations. Each should be within 10%, or the system is malfunctioning. Use the Standards sheets in the PIXE run book containing the certified concentrations to record the measured values and calculate differences.
- f) Set RACE to produce full output for the Pb, CuS, Fe, and two PESA (mylar blanks) standards. Press the PF4 key, then press the "A" key to set the input parameters on RACE to produce full output, then select the "N" key to begin a new analysis sequence (see section 4.5.2 for instructions). Exit back to RACE by pressing the "X" key. The full output sheets for each detector will resemble the sheet for the CuS standard shown in Figures 30, 31, and 32.
- RACE will ask

First Run to Analyze (Press ^Z to Exit):

Enter the run number for the first calibration standard. For the B96 CuS standard:

77009<CR>

Use the PF4 key and the "begin new sequence" (N) switch as necessary to produce the full output for the Pb, CuS, Fe, and PESA blank standards.

- g) Enter the PIXE detector calibrations. Collect the full output sheets from the printer, then stop printing and set RACE to accept new calibrations. Use the PF4 key to enter the RACE Input Parameters screen. Press the “A” key to deselect the print full output option, then select the “C” key to enter a new calibration, press the “N” key to begin a new analysis sequence, and exit back to RACE by pressing “X”.
RACE will ask

First Run to Analyze (Press ^Z to Exit):

Enter the run number for the first calibration standard. For the B96 CuS standard:

77009<CR>

Enter Energy Calib for PX1? (Y or N)

Enter “N” if you do not wish to enter a PIXE-1 detector calibration. RACE will process the PIXE-1 data normally, then repeat the question for PX2 (the PIXE-2 detector) and PX3 (the PIXE-3 or PESA detector).

Enter “Y” to enter a calibration for the detector.

Do you want to enter a 1,2,3, or 4 point calibration?

For the PIXE-1 or PIXE-2 detectors, enter the number four to specify entry of a four point calibration.

4 <CR>

For the PESA detector (PIXE-3), the operator would enter the number two to specify a two point calibration (PESA only recognizes 2 species, H and C).

2 <CR>

RACE will place a blinking cursor on the screen and wait for the operator to enter the calibration data for the PIXE-1 detector. Four rows of data, two fields in each row, with the fields separated by a space must be entered for the detector, with PIXE-1 entered first. To determine what data to enter, the operator will review the full output sheets for the Pb, CuS, and Fe standards (see Figures 30, 31, and 32 for examples of a full output sheets for the CuS standard).

Figure 30 PIXE-1 Detector CuS Standard Full Output

UC DAVIS PIXE-1 ANALYSIS 77008 OUTPUT: FILE 1, ITEM 10 CROSS SECTION TABLE CAN1NOV96* 1.000 12-NOV-96
 LIVE TIME = 58.0 / 60.0 = 0.97 CHARGE = 143 NC, CURRENT = 2 NA ADC RATE = 501 COUNTS/SEC DCON = 1.000 M**3/CM**2
 PEAK WIDTH = 7 CHANNELS BLANK RUN 77033 * 3.30 NTHICK=100 ITERS=25 KOVER=1 ENERGY=0.6132 + 0.0249* CHAN ()
 MATRIX CORRECTIONS ARE NOT INCLUDED. UNCERTAINTIES IN CONCENTRATIONS ARE STATISTICAL PLUS 5%, ADDED QUADRATICALLY
 PROFILE

VAXRace

PEAK	ELEMENT	NANOGRAMS PER CM**2	ENERGY (KEV) PRED FOUND	CHANNEL	COUNTS	LINEAR SUM	BACK GROUND	LOC	CUTA CUTB	WIDTH	CHISQ	MULT	LOOP
1	NA K	9356.17	1.041 0.982	14.8	539	597	62	3	4.42	8.2	1	5	
.	.	+/-739.92	-0.059 +/-0.003	+/-0.1	+/-33.			14 22	+/-0.17				
2	S K	14286.46	2.307 2.307	68.1	3629	4024	478	58	7.49	0.1	1	5	
		+/-835.05	0 +/-0.002	+/-0.1	+/-110			68 82	+/-0.26				
3	K KB	11911.12	3.589 3.694	123.9	591	638	355	115	8.31	0.5	1	6	
		+/-1256.32	0.105 +/-0.006	+/-0.2	+/-55.			124 132	+/-0.91				
	OR CA KA	1193.83	3.69										
		+/-125.92	0.004										
4	CU KA	46588.44	8.041 8.036	298.5	14275	14770	242	285	8.37	0.5	1	6	
		+/-2475.74	-0.005 +/-0.001	+/-0.1	+/-257.			299 313	+/-0.18				
5	CU KB	48569.68	8.907 8.901	333.3	2136	2250	49	321	8.47	0.4	1	6	
		+/-3373.86	-0.006 +/-0.004	+/-0.2	+/-103.			334 348	+/-0.49				

MINIMUM DETECTION LIMITS

NA	MG	AL	SI	P	S	CL	K	CA	TI	V	CR	MN	FE	NI
265.78	163.17	124.19	116.05	121.89	118.1	112.83	73.2	60.97	57.84	49.42	42.36	34.65	43.68	57.6
CU	ZN	GA	AU	HG	AS	PB	SE	BR						
75.39	61.78	0	0	0	0	0	0	0						

Figure 31 PIXE-2 Detector CuS Standard Full Output

UC DAVIS PIXE-1 ANALYSIS 77008 | OUTPUT: FILE 1, ITEM 10 | CROSS SECTION TABLE CAN2NOV96* 1.000 | 12-NOV-96
 LIVE TIME = 55.0 / 60.0 = 0.92 | CHARGE = 143 NC, CURRENT = 2 NA | ADC RATE = 1045 COUNTS/SEC | DCON = 1.000 M**3/CM**2
 PEAK WIDTH = 7 CHANNELS | BLANK RUN 77033 * 2.05 | NTHICK = 100 | ITERS = 21 | KOVER = 1 | ENERGY = 0.6455 + 0.0255* CHAN ()
 MATRIX CORRECTIONS ARE NOT INCLUDED UNCERTAINIES IN CONCENTRATIONS ARE STATISTICAL PLUS 5%, ADDED QUADRATICALLY
 PROFILE 11

VAXRace

PEAK	ELEMENT	NANOGRAMS PER CM**2	ENERGY (KEV) PRED FOUND	CHANNEL	COUNTS	LINEAR SUM	BACK GROUND	LOC	CUTA CUTB	WIDTH	CHISQ	MULT	LOOP
1	NA K	9058.19 +/- 761.23	1.041 0.994	13.6 +/- 0.1	462 +/- 31.	585	221	3	13 26	4.34 +/- 0.18	9.1	1	5
2	S K	12913.71 +/- 870.00	2.307 0.003	2.31 +/- 0.1	65.2 +/- 148.	3625	590	53	65 79	9.21 +/- 0.52	0	1	6
3	K KB or CA KA	8041.05 +/- 891.15 1153.75 +/- 127.86	3.589 0.102 3.69 0.001	3.691 +/- 0.3	119.4 +/- 122.	1233	1309 864	106	120 129	10.35 +/- 1.23	0.6	1	7
4	CU KA	47488.83 +/- 2486.57	8.041 0.005	8.046 +/- 0.0	290.1 +/- 622.	40724	867	276	290 304	10.08 +/- 0.20	0.1	1	6
5	CU KB	48867.88 +/- 3182.28	8.907 0.003	8.91 +/- 0.1	324 +/- 235.	5754	540	310	324 338	10.1 +/- 0.53	0.2	1	7

MINIMUM DETECTION LIMITS

NA	MG	AL	SI	P	S	CL	K	CA	TI	V	CR	MN	FE	NI
453.38	180.27	125.84	114.67	138.6	122.5	104.95	49.55	38.36	32.02	26.57	22.12	19.24	23.37	34.14
CU	ZN	GA	AU	HG	AS	PB	SE	BR						
50.96	57.28	46.73	76.04	64.45	23.82	74.53	28.82	23.85						

Figure 32 PESA Detector CuS Standard Full Output

UC DAVIS PESA-1 ANALYSIS 77008 | OUTPUT: FILE 1, ITEM 10 | CROSS SECTION TABLE PESAAPR95* 1.000 | 12-NOV-96
 LIVE TIME = 60.0 / 60.0 = 1.00 | CHARGE = 143 NC, CURRENT = 2 NA | ADC RATE = 463 COUNTS/SEC | DCON = 1.000 M**3/CM**2
 PEAK WIDTH = 15 CHANNELS | BLANK RUN 77033 * 2.0 | NTHICK = 100 | ITERS = 11 | KOVER = 15 | ENERGY = 2.6061 + 0.0279* CHAN ()
 MATRIX CORRECTIONS ARE NOT INCLUDED UNCERTAINIES IN CONCENTRATIONS ARE STATISTICAL PLUS 5%, ADDED QUADRATICALLY
 PROFILE 11

VAXRace

PEAK	ELEMENT	NANOGRAMS PER CM**2	ENERGY (KEV) PRED FOUND	CHANNEL	COUNTS	LINEAR SUM	BACK GROUND	LOC	CUTA CUTB	WIDTH	CHISQ	MULT	LOOP
1		0 +/- 0.00	0 3.304	25 +/- 0.4	335 +/- 28.	394	86	7	22 37	10.95 +/- 0.74	2.7	1	3
2	H	24988.65 +/- 1373.36	5 -0.095	4.905 +/- 0.0	82.3 +/- 89.	3885	4015 244	67	82 97	6.82 +/- 0.08	1.8	1	5
3	C	78903 +/- 4017.68	6 -0.033	5.967 +/- 0.0	120.3 +/- 208.	21605	22045 140	105	120 147	5 +/- 0.02	44.1	1	4

MINIMUM DETECTION LIMITS

H	C
176.88	84.73

- i) Identify the pertinent information on the PIXE-1 full output sheet. The largest concentrations (in the NANOGRAMS PER CM**2 column) refer to the largest peaks. The largest peak (or peaks, in the case of CuS) should correspond to the elemental species by which each standard is labeled.
- ii) Locate the peaks having the highest concentration on each full output form for the standards being used for the calibration (Pb, Cu, S, Fe).
- iii) Calibration data entry instructions. The first field of data entered for the calibration will contain the data channel for an element peak. The second field will contain the predicted energy, in KeV, of the same peak. Separate the fields with a single space, and press return after entering each row to proceed to the next entry.
- iv) Highlight the channel for the peaks located in step ii. This value will be entered into the first field of a row during calibration value entry.
- v) For the peaks located in step ii, look up the x-ray energies (energy, in KeV of the Pb L α , Cu K α , S K α , and Fe K α lines). Each value will be entered into the second field of the calibration row.
- vi) Calculate the calibration equation. When all four rows have been entered, the terminal will process the values to determine the slope and intercept of the calibration lines defined by the entered points. The slope and intercept, respectively, will be displayed on the terminal screen. RACE will ask:

Reenter the Calibration (Y or N):

Enter Y if a typographical error was made during entry of the values, and RACE will return to step g.

- vii) Enter "N" to save the calibration. The calibration will be saved by RACE in the analysis parameter file, AXBX.PX1.
- viii) Calibrate the PIXE-2 detector. Once RACE analyzes the detector data using the new calibration values, it will return to step g. and ask whether the operator wishes to enter the calibration for the next detector. Repeat section g. steps i. through viii. using the PIXE-2 full output sheet for the calibration values to perform PIXE-2 calibration. This will produce the analysis parameter file AXBX.PX2.
- ix) Calibrate the PESA detector. Once RACE analyzes the detector data using the new calibration values, it will return to step g. and ask whether the operator wishes to enter the calibration for the PX3 detector. Repeat section g. steps i. through viii. using the PIXE-3 full output sheet for the PESA standards to determine the calibration values for H and C. Remember to select calibration using two, not four values. This will produce the analysis parameter file AXBX.PX3. This completes the rough calibration of the PIXE and PESA systems through RACE.

2. Analyze the filter blanks to determine the portion of the x-ray spectra from samples due solely to the filter media. The blanks are located in the last five positions of the standards tray.
 - a) Request the cyclotron controller maintain a proton beam energy of 9MeV, with a count rate of approximately 2000 counts per second (5 to 20 nanoAmps current for IMPROVE filter analysis).
 - b) Use the ACE keypad to enter a false trayfile name (for Sept 13, 1996, a false trayfile name would be 961309.dat, the date in reverse), and filter position # '0'. This trayfile does not exist. The filter position of zero tells ACE that this is a blank filter analysis.
 - c) Begin analysis of the blank filter (unused Teflon filter from the same filter lot as those being analyzed). Review the spectral output from the analysis. There should be no significant x-ray peaks. If significant peaks exist, discontinue analysis of this filter as it may be contaminated.
 - d) If large H peaks are seen on the PIXE-3 detector (PESA), contact the quality assurance manager as this may be organic artifact on the filter.
 - e) Repeat steps c and d until at least two good blanks are found.
 - f) Choose the cleanest blank filter, the filter having the fewest significant spectral peaks, as the blank for the analysis run.
 - g) Record the analysis run number of the selected blank filter on the standards sheet.

3. Perform the fine calibration of the PIXE and PESA systems.
 - a) Reanalyze two trays of filters from IMPROVE sites analyzed during the previous IMPROVE analysis session. The two reanalysis trays generally selected are SHEN1 and CRLA1, representing a moderately heavily loaded site and a lightly loaded site.
 - b) Use the ACE keypad to reset the TIM (time) to the analysis duration used on the first tray during the previous quarter analysis.
 - c) Adjust the blank float to remove most of the x-rays from the filter substrate by subtracting a system blank (a blank filter spectra).
 - i) On RACE, press the PF4 key to access the Analysis Parameter Input screen. Press the "F" key to check the blank float, the "D" key to turn on the graphics display, the "N" key to begin a new sequence, and the "X" key to exit back to RACE.
 - ii) Begin RACE by entering the run number of the selected filter blank from step 2.
 - iii) When RACE has analyzed the filter blank, press the PF4 key to access the Analysis Parameter Input screen. Press the "N" key to begin a new sequence, and the "X" key to exit back to RACE.
 - iv) Enter the first run number for the reanalysis tray. RACE will recreate the spectra, then superimpose the blank filter spectra. To achieve the best fit, the blank filter spectra is floated by a multiplicative factor until a preset percentage of channels is

exceeded. The operator can adjust the factor by changing the percentage of channels exceeded. The default percentage is 1%.

v) RACE will display:

BLANK SUBTRACTION: DISPLAY OF RAW DATA AND FLOATED BLANK
(use SHIFT-VIEW to toggle between screens.)

BLANK * 0.800 BASED ON 1% OF CHANNELS BETWEEN 4 AND 9 KeV ABOVE DATA

ENTER NEW PERCENTAGE (<CR> TO KEEP, -1 FOR BLANK AT 100%)

- vi) Review the superimposed spectra. The goal is to set the blank so that when it is subtracted from the filter sample spectra, the resulting spectra does not contain any peaks due to the filter itself.
- a) If too much spectra is being lost, reduce the percentage by entering a lower number. The program will repeat parts iv) and v).
 - b) If not enough spectra is being subtracted, increase the percentage by entering a higher number. The program will repeat parts iv) and v).
 - c) If the spectra looks good, press <CR> and the program will continue.

vii) RACE will display:

ABSORPTION CORRECTION FOR THE THICKNESS OF SUBSTRATE, (left side of spectrum)

Nominal Values are Mylar75
 Teflon100
 Nuclepore170

ENTER THICKNESS FACTOR, CURRENT = 100
(<CR.> TO KEEP CURRENT VALUE)

viii) IMPROVE filters are Teflon, so press <CR> to continue. RACE will draw the spectra resulting from the subtraction of the blank from the filter sample analysis.

d) Adjust the background fit to remove most of the background due to scattering of x-rays off the filter and deposit.

i) On RACE, press the PF4 key to access the Analysis Parameter Input screen. Press the “F” key to switch off the blank float check module, the “B” key to check the background fit, and the “X” key to exit back to RACE.

ii) RACE will create a spectra, automatically perform the blank subtract, then display the following on the screen:

FITTED BACKGROUND: DISPLAY OF DATA - BLANK AND FITTED

BACKGROUND

(Use SHIFT-VIEW to toggle between screens)

SAMPLE CONSIDERED TO HAVE LIGHT LOADING

ENTER ITERATIONS FOR FIT, CURRENT = 17

(<CR> to keep current value)

iii) Review the spectra. The goal is to remove the remaining background by subtracting a smooth function calculated as the lesser of the counts in the channel and an average counts per

channel over a specified width. To change this fit, the operator can adjust the width and the number of iterations of the process. The bases of the peaks in the resulting spectra should barely reach the zero counts level. The background subtract cannot be too large, or the peaks in the resulting spectra will appear smaller, causing RACE to underestimate the elemental concentrations. If the background subtract is too small, the peaks in the resulting spectra will appear higher, causing RACE to overestimate the elemental concentrations.

- a) If too much spectra is being lost, increase the number of iterations used to calculate the background subtract. The program will repeat part ii).
- b) If not enough spectra is being subtracted, decrease the number of iterations. The program will repeat part ii).
- c) If the spectra looks good, press <CR> to continue. RACE will calculate elemental concentrations from the peak locations, areas, the cross section and calibration tables, and the matrix corrections.
- iv) On RACE, press the PF4 key to access the Analysis Parameter Input screen. Press the “B” key to turn off the check of the background fit, and the “X” key to exit back to RACE.
- e) Press the PF4 key to access the Analysis Parameter Input screen and set RACE to print all full output.
- f) Get full output for five analyses, then press the PF4 key to access the Analysis Parameter Input screen and stop printing full output. Select the begin new sequence option.
- g) Review the full output, highlighting the data for the S, Fe KA, Zn KA, and Se KA peaks.
 - i) Check the difference between the Predicted and Found Energy for each of the peaks in part f. For a good calibration, this difference should be less than 0.004. Note: the difference is printed below the Predicted Energy.
 - ii) If the differences from part i.) are greater than 0.004, from the five full outputs, select the peak having the greatest reported concentration (NANOGRAMS PER M**3) for each of the species of interest (S, Fe, Zn, and Se).
 - iii) Use the channel and predicted energies for the peaks selected in step ii.) to enter a new calibration, using the procedure described in part 1, step g.
- h) Repeat steps d) through h), except perform RACE analysis a different set of five reanalysis filters. Preferably, analyze two or three filters from each reanalysis tray. The differences between predicted and found energy should be less than 0.004. If not, enter a new calibration equation and repeat steps d) through h) again.

- i) When an acceptable calibration equation has been derived, analyze both reanalysis trays through RACE. Report to the Quality Assurance Specialist when both trays are completed. The QA Specialist will plot the reanalysis data against the original analysis data for both reanalysis trays. If the correlations are stable, the Quality Assurance Specialist will release the PIXE and PESA systems for use. These procedures are covered in depth in the Quality Assurance SOP.
4. Run the actual IMPROVE samples. Each tray contains all samples for a given season from a single site. Initialize the analysis of the samples in the tray by entering the identification code of the tray file in the acquisition program. Verify the correct tray file is being used. Initialize the spectral analysis program by entering the run number of the analysis. Check the following features in the spectral analysis of the sample. If any problems are detected, consult with the QA Manager, the PIXE/PESA Manager, or a senior scientist.
 - a) Is the background completely removed by the two background steps? That is, do the valleys between the peaks in the final spectrum go to zero. If not, determine if the problem is an inappropriate blank. Verify on the output log that the run number of the blank is correct. Do not revise the fitting parameters without consulting with the QA Manager or Spectroscopist.
 - b) Are there large peaks in the spectra that are not being identified? The PIXE or PESA detector channels may have shifted enough that RACE cannot correlate the calibration table with the actual spectra.
 - c) Does it look as if data are missing? Are there not peaks where expected? Are the concentrations derived by RACE far lower than expected?
5. Rerun the reanalysis trays and the standards at the end of the analysis run.
6. Before ending the analysis run, wait until the QA Manager is able to validate the results for all of the reanalysis trays. If problems in the analysis are determined, select the trays and samples involved and reanalyze them.

4.6 Routine Analysis

The standard analysis procedures for PIXE and PESA analysis are listed below.

4.5.1 RENO calculation and system calibration

4.5.2 Blank filter analysis

4.5.3 Reanalysis trays

4.5.4 Filter sample analysis

4.6.1 RENO Calculation and System Calibration

At the beginning and end of every analytical session, the calibration is checked with a series of 20-30 thin film standard foils. Following analysis, the measured elemental concentrations from the thin film standards are compared to the concentrations quoted by the manufacturer for each elemental thin film standard. If the ratios of measured to quoted values show that the shape of the calibration curve has significantly changed, the analysis cannot proceed until the problem is rectified. A mean ratio is less than 0.90 or greater than 1.10, indicates the calibration curve has deviated significantly. This mean ratio, called a RENO or renormalization factor, is applied to all concentrations measured during the analytical session. This corrects the measured values to a standard to allow direct comparison of data from different analysis quarters. The RENO is normally between 0.98 and 1.02.

4.6.2 Blank Filter Analysis

Once the standards have indicated the system functioning correctly, a set of unexposed Teflon filters, blanks, are analyzed. The cleanest blank is selected and defined to be the system blank for the PIXE spectra. The system blank spectrum is subtracted from all subsequent filter sample analyses to remove the x-ray spectrum due to the clean filter substrate.

4.6.3 Reanalysis Trays

After the calibration check and the inclusion of a system blank, two trays of filters that were analyzed during the previous analysis quarter, approximately 52 filters, are reanalyzed. The concentrations of key elements from the re-analyses, generally, S, Ca, K, Mn, V, Fe, Zn, Cu, Se, Pb, and Br, are plotted against those from the initial analysis. If the slope on any plot is less than 0.90 or greater than 1.10, a problem with the system exists and the analysis cannot proceed until it is rectified.

These two reanalysis trays are analyzed at the start and end of the run, and following every thousand analyses during the run. They are also analyzed just prior to and following any analysis break.

4.6.4 Filter Sample Analysis

Once the standards and reanalyses trays have been run and the systems are validated by the Quality Assurance Manager, the analysis of normal filters begins. PIXE and PESA analysis are conducted automatically using trayfiles containing instructions for subsequent analysis of up to four site trays of filters. However, trained personnel must be on hand to

ensure correct operation of the cyclotron, and to change the analysis trays. The Spectroscopist shall review the system parameters and the results of the analysis several times each day to ensure that no problems have occurred. Checking the system parameters and analysis results includes checking the integrated charge and the detector count rate, checking the analysis filter position, and verifying that a reasonable number of elements are being identified. Also, the Spectroscopist checks for reasonable agreement between the α and β lines of the same element, and monitors the Fe concentrations obtained from the PIXE-1 and PIXE-2 detectors. This provides quality assurance of the system, as both detectors have reasonable resolution for Fe. If any parameter is incorrect, or any result is suspicious, the problem is corrected and all potentially affected samples are reanalyzed.

4.6.5 Data Storage

The data are backed up on magnetic tape every night to ensure that in case of a system failure, no more than a single day of data will be lost. Also, at the end of each quarterly analysis session, all the data collected during the analysis run are backed up on a permanent storage medium and archived.