950A139539 (Rev. C)

MAXray

Parallel Beam Spectrometer

INSTALLATION AND SUPPORT MANUAL

For use by authorized NORAN Service Personnel only

ThermoNORAN

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Revision history

Revision	Revision date	Principal changes
A	February 2000	First publication
B/C	April 2001	WDS Software Version 3.1.2

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2 – WDS Software Revisions

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1 – Safety Precautions and Technical Specifications

AC Power

WARNING: All electrical equipment must meet applicable codes and be installed by NORAN Instruments authorized support representatives.

WARNING: Dangerous voltages are present inside the equipment.

High operating voltages are present inside the equipment. These voltages can produce severe burns or fatal electrical shocks.

- Failures in improperly grounded equipment can transfer dangerous voltages to outside surfaces.
- Numerous internal components operate at high voltages and may not be insulated or shielded.
- Components such as capacitors and batteries can store high voltages even with the power disconnected.

WARNING: Maintain proper equipment grounding.

- Always connect equipment to a properly grounded power source receptacle. If in doubt, have the receptacle checked by a qualified technician.
- Never use a ground adapter plug to connect NORAN Instruments' equipment to a power source receptacle that lacks ground connection terminals.
- Never operate your NORAN Instruments' equipment if you notice unusual noises or foul odors. Disconnect the power cord from the main power source and call your local NORAN Instruments support specialist.

WARNING: Avoid contact with internal high voltage sources.

- Never remove any outer covers or guards that are fastened with screws without being so instructed either by this manual, or by a qualified NORAN Instruments support specialist.
- Never attempt internal service or system adjustments unless instructed to do so either by this manual, or by a qualified NORAN Instruments support specialist.
- Never remove system connections when power is applied.

- Never override or “cheat” electrical or mechanical interlock devices.
- When working around high voltage sources, be sure all power inputs are disconnected.
- When working around capacitors, batteries, and other power sources, allow a few minutes for capacitors to discharge after power has been removed.
- Avoid contact, direct or through metal tools or jewelry, with any uninsulated, internal, electrical component without being so instructed either by this manual, or by a qualified NORAN Instruments support specialist.

CAUTION: *The fuse in the power entry module must match the line voltage.*

P-10 Gas

The flow proportional counter requires a special gas called P-10. Tanks of P-10 gas are available from local bottled gas suppliers. The tank of P-10 should have a two-stage regulator with a 1/4 NPT fitting to accept the NORAN-supplied third stage regulator.

WARNING: P-10 gas is a HAZARDOUS MATERIAL. Handle P-10 gas in accordance with the manufacturer’s Material Safety Data Sheet.

WARNING: Fasten the tank of P-10 gas securely to a fixed object, such as a wall or workbench, to avoid damage to the tank. The tank contains hazardous material under pressure. The tank is dangerous if its valve is damaged or the tank is punctured. However, the gas is *not* flammable.

Diffractors

CAUTION: *Do not touch the face of the diffractors.*

CAUTION: *Do not touch or attempt to clean the surface of any diffracting crystal.*

The diffractor surfaces are sensitive and some can be dissolved with cleaning solvents, including water.

Manual Gate Valve Option

Never pump the column down with the gate valve closed and the spectrometer at atmosphere.

WARNING: Do not open the gate valve when the SEM is at vacuum and the WD spectrometer is at air.

Opening the gate valve under these conditions could expose the microscope vacuum system to contamination. If the filament is powered on, it may also be damaged.

WD Spectrometer Controller

WARNING: All electrical equipment must meet applicable codes and must be installed by NORAN Instruments authorized support representatives.

WARNING: Dangerous voltages are present inside the equipment.

High operating voltages are present inside the equipment. These voltages can produce burns or fatal electrical shocks. Numerous internal components operate at high voltages and may not be insulated or shielded.

Components such as capacitors can store high voltages even with the power disconnected. When you are working around high voltage sources, be sure all power inputs are disconnected.

Never remove any outer covers or guards that are fastened with screws without being instructed by this manual or by a qualified NORAN Instruments support specialist.

Never attempt internal service or system adjustments unless instructed to do so either by this manual or by a qualified NORAN Instruments support specialist.

Maintain proper equipment grounding.

Always connect equipment to a properly grounded power source receptacle. If in doubt, have the receptacle checked by a qualified technician.

Never use a ground adapter plug to connect NORAN Instruments equipment to a power source receptacle that lacks ground connection terminals.

CAUTION: *Do not move the stage when the MAXray optic is inserted without knowing its position. Driving the stage into the optic will damage the optic.*

CAUTION: *The controller must be installed according to the procedures described in the installation and service manual to avoid damage to the spectrometer.*

CAUTION: *The fuse(s) in the controller must match the line voltage.*

CAUTION: *Check that the fuse(s) for the input AC voltage are correct.*

The switching power supply has a universal input autoranger that eliminates the requirement of changing jumper wires or switches to change the input voltage range, but the correct fuse(s) must be installed.

- For 100/120 VAC operation use one T 250V 3A fuse.
- For 230/240 VAC operation use two T 250V 2A fuses (5x20 mm).

CAUTION: *For continued protection against fire, replace the fuse(s) only with the specified type.*

CAUTION: *Do not place objects under or around the bottom of the controller that would restrict the cabinet cooling.*

Never operate your NORAN Instruments equipment if you notice unusual noises or foul odors. Disconnect the power cord from the main power source and call your local NORAN Instruments support specialist.

CAUTION: *Make sure the P-10 gas flow through the proportional counter is correct before energizing the detector.*

Flow Proportional Counter Window

CAUTION: *The material that isolates the gas inside the detector from the vacuum of the spectrometer and the SEM chambers is made of polypropylene. If the window must be replaced, follow these precautions:*

CAUTION: *Shut off the high voltage to the detector.*

CAUTION: *Turn off the P-10 gas.*

CAUTION: *Solvents must not come into contact with the window.*

WARNING: Do not seal off the gas flow routinely or the window will break.

Motorized Retraction and Manual Retraction

Do not change the retraction limits set during installation.

Detector Touch Sensor Module

The Detector Touch Sensor module requires a nine volt battery and contains a buzzer, LED, and control circuitry. The input from the touch sensor is via a coax connector as is the output to the stage automation system. The control circuitry consumers approximately 25 microamperes during normal operation and approximately three milliamperes when touch occurs.

- The contract closure from the Touch Sensor on the detector is connected to the input of a discriminator and terminated with a one megohm resistor to the battery and a 0.1 microfarad capacitor to ground. When a touch condition occurs this signal is grounded.
- The output to the stage automation system is from an open drain terminated with a one megohm resistor to the battery and a 5.1 volt zener diode to ground. This signal should be connected to a TTL input with a pull-up resistor in the stage automation system. The signal is a negative true signal (touch = ground and no touch = +5 volts) and is capable of sinking up to five milliamperes.

- When a touch occurs the LED is turned on and the buzzer sounded.
- When the battery voltage decreases to approximately seven volts the LED and the buzzer are turned on momentarily about once every five seconds to indicate the battery should be replaced.

NOTE: If replacement of any part is required on the printed circuit board removal of all flux is required. The control integrated circuit and associated components are all high impedances and any residue flux can adversely affect the circuit operation.

NOTE: If trouble shooting the board is required insert 90 megohms of resistance in series with a oscilloscope 10 megohm probe. This way the probe will not load the circuit and adversely affect your trouble shooting.

NOTE: If the Detector Touch Sensor Module is being used without being connected to the stage automation system connect the ground wire provided between a chassis ground and the Stage BNC of the module. This will prevent the sensor from floating and building up a charge which could distort the SEM image.

Interface Signal Description

Sensor BNC

The contact closure form the detector is connected to the module input BNC through a coax cable. The signal is terminated with a one megohm resistor to the battery and a 0.1 microfarad capacitor to ground. When a touch condition occurs this signal is grounded.

Stage BNC

The output to the stage automation system is from an open drain terminated with a one megohm resistor to the battery and a 5.1 volt zener diode to ground. This signal should be connected via a coax cable to a TTL input with a pull-up resistor in the stage automation system. This signal is a negative true signal (touch = ground and no touch = +5 volts) and is capable of sinking up to five milliamperes.

NOTE: If the Detector Touch Sensor Module is being used without being connected to the stage automation system connect the ground wire provided between the sensor from floating and building up a charge which could distort the SEM image.

Touch Sensor Module Checkout Procedure

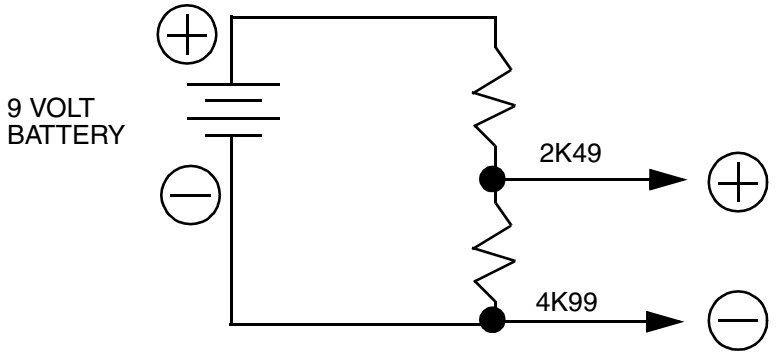
Equipment

- VOM
- Test resistor divider network and clip leads
- Phillips screwdriver (small)

NOTE: If replacement of any part is required on the printed circuit board removal of ALL flux is required. The control integrated circuit and associated components are all high impedances and any residue flux can adversely affect the circuit operation.

NOTE: If trouble shooting the board is required a nine volt battery and contains a buzzer, LED, and control circuitry. The input from the touch sensor is via a coax connector. The output to the stage automation system is also from a coax connector. The control circuitry consumes approximately 25 microamperes during normal operation and approximately three milliamperes when touch occurs.

1. Remove the module cover by removing the four small Phillips screws.
2. Install the 9 volt battery if necessary.
3. Measure the voltage across the battery to insure a charged battery.
4. Measure the voltage at the output **Stage BNC**. The voltage should be between 4 and 5 volts.
5. Use a clip lead and short the input **Sensor BNC**. The **LED** should turn on, the **Buzzer** should sound, and the voltage at the output **Stage BNC** should go to approximately 0 volts.
6. Remove the battery from the module. Use the following resistor divider network to rest the low battery voltage condition.

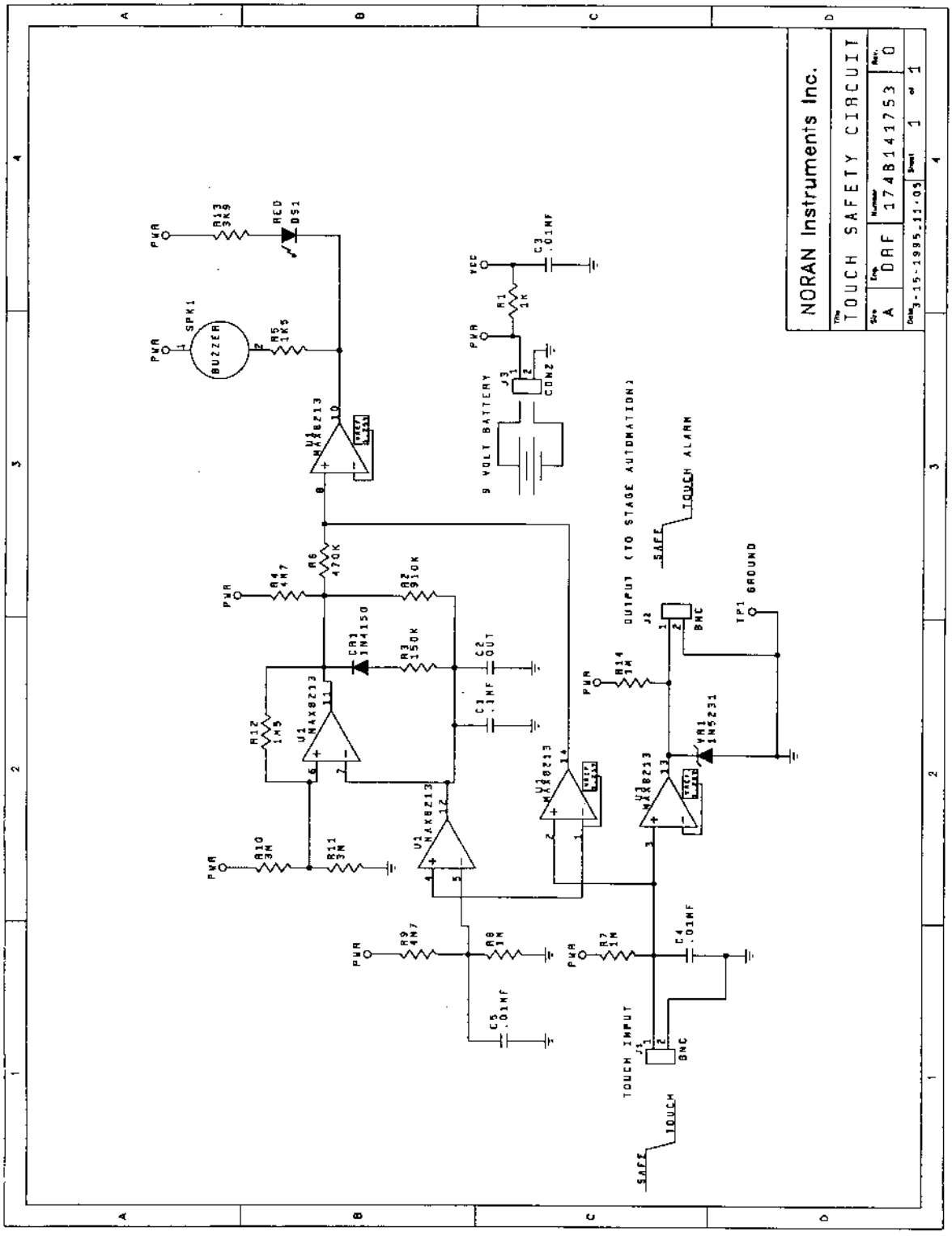


7. Measure the voltage going into the printed circuit board battery connector J3. It should be approximately 6 volts. The module LED should flash and the alarm buzz approximately once every five seconds.
8. Remove the resistor divider, clip leads, etc.. Replace the cover.

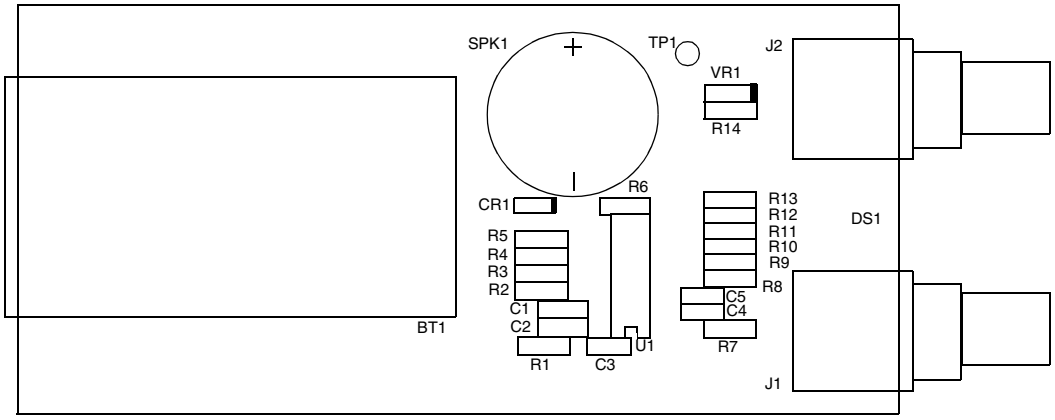
List of Materials Report for Touch Sensor Board Assembly

Item	Quantity	Part Number	Value	Ref Description
1	1	150A103385	.1MF	C1
2	1	170A141753	PWB	
3	3	150A112766	.01MF	C3, C4, C5
4	1	480A000808	1N4150	CR1
5	1	245A002369	RED	DS1
6	2	210A003313	BNC	J1, J2
7	1	210A128705	CON2	J3
8	1	470A000113	1K	R1
9	1	470A000175	910K	R2
10	1	470A000138	150K	R3
11	2	470A000153	4M7	R4, R9
12	1	470A000115	1K5	R5
13	1	470A000159	470K	R6
14	3	470A000145	1M	R7, R8, R14
15	2	470A000152	3M	R10, R11
16	1	470A000148	1M5	R12
17	1	470A000120	3K9	R13
18	1	130A111669	BUZZER	SPK1
19	1	210A117653	BLACK_TP	TP1
20	1	318A128706	8213	U1
21	1	480A000811	1N5231	VR1
22	1	140G127866	Touch Sensor Front Panel	
23	1	254A110049	LED Grommet	
24	1	400A130215	Battery 9 Volt	
25	4	280a108596	Screw 2-56 0.281 Pan	
26	2	280a001094	Nut 4-40 Elastic Stop	
27	2	280a116753	Nut Hex 7/16 - 26	
28	2	280a002372	Screw 4-40 0.25 SS Pan Head Blk Ox	
29	1	930a130218	Touch Sensor General Description	
30	0	930a130216	Assembly Instructions	
31	0	930A135936	Touch Sensor Module Checkout Procedure	

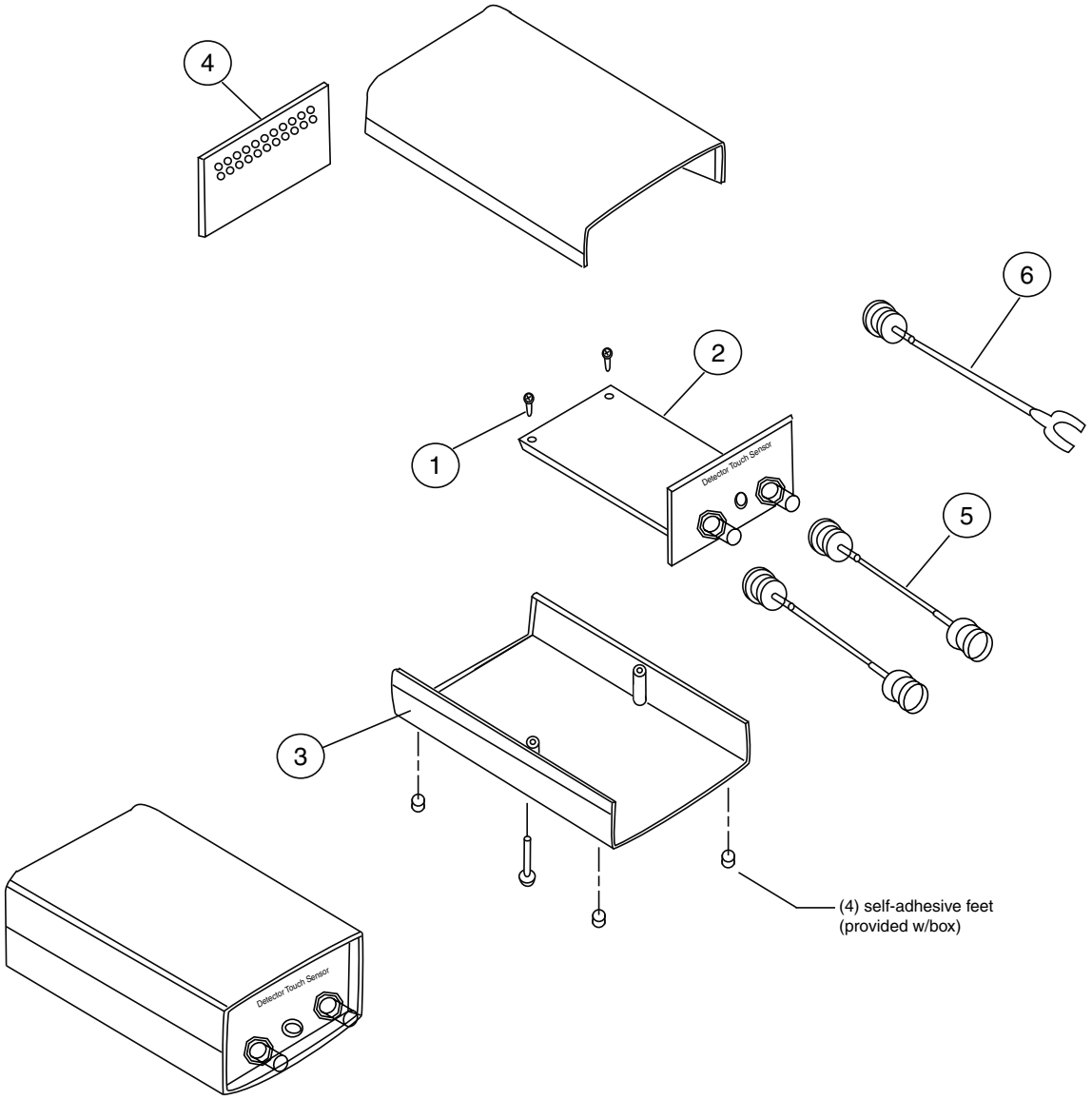
Schematic


NORAN Instruments Inc.
TOUCH SAFETY CIRCUIT
DRF
174B141753
3-15-1995_11:03
9 VOLT BATTERY
BUZZER
TOUCH ALARM
OUTPUT (TO STAGE AUTOMATION)
GROUND
TOUCH INPUT
SAFE
TOUCH

Assembly Drawing (top)



Box Drawing



00 0 REF 700P127869 A TOUCH SENSOR ALARM BOX
01 2 2 280A122449 B SCREW, 4-40 X 1/4 PAN HD PHIL SS
02 1 NO 176A141753 A TOUCH SENSEE BOARD ASSEMBLY
03 1 NO 145A112823 B PLASTIC ENCLOSURE
04 1 NO 140G127867 M TOUCH SENSOR REAT PANEL
05 2 2 610P003216 M CABLE BNC TO BNC PLUG 6'
06 1 NO 910P130218 A CABLE BNC TO PLUG 6'

SEM Venting and Window Protection

Always loosen an unused port cover before venting the SEM. Air or nitrogen entering the chamber can easily break the proportional counter window.

Symbols

the following symbols are used on this equipment:

Symbol	Description
	CAUTION. Refer to accompanying documents.
	CAUTION. Risk of electric shock.
	Protective Conductor Terminal
	Frame or Chassis Terminal
	On (supply)
	Off (supply)

Electrical Specifications

WD spectrometer controller input power requirements

100 to 240 volts ACL 50/60 Hz, 2.5 amperes maximum Voltage: 100/120/230/240 VAC

Frequency: 50/60 Hz

Fuses: 100/120 VAC - T 250V 3A

230/240 VAX - T 250V 2A

Internal on Switching power supply - 250V 4A

WD Spectrometer Controller Input/Output Connectors

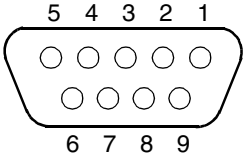
Mapping (BNC)

Signal	Description
Map Out	Output: TTL level: (0 to 3.5 V: 24 mA)

VAC-OK (BNC)

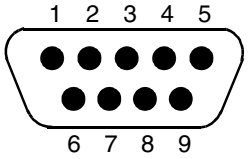
Signal	Description
VAC-OK	Input: TTL level (0 to 3.5 V: 10 mA)

PCS (9-pin D Female)



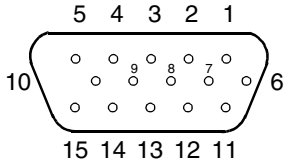
Pin number	Signal	Description
1	HVSET	Output: Analog level: 0 to 12V @ 6 mA
2	LLD	Output: Analog level: 0 to 12V @ 6 mA
3	HVINT	Output: TTL level (0 to 3.5 V: 24 mA)
4	-15V	Output: -15V @ 400 mA maximum
5	SCAL	Input: TTL level (0 to 3.5 V: 24 mA)
6	ULD	Output: Analog level: 0 to 12V @ 6 mA
7	Ground	
8	Ground	
9	+15V	Output: +15V @ 400 mA maximum

Serial (9-pin D Male)



Pin number	Signal	Description
1	DCD	Carrier Detect. Not used.
2	RXD	Input: to RS-232 transceiver - (maximum input voltage ±30 V: 10 mA)
3	TXD	Output: from RS-232 transceiver (output rating: VOL = 0.4V @ 3.2 mA and VOH = 3.5V @ -1.0mA)
4	DTR	Data Terminal Ready. Not used.
5	Ground	
6	DSR	Data Set Ready. Not used.
7	RTS	Request to Send. Not used.
8	CTS	Clear to Send. Not used.
9	RI	Ring Indicate. Not used.

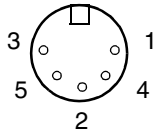
Motor (15-pin D Female, High-Density)



Pin number	Signal	Description
1	5 volts	Output: Encoder power
2	ENC_B	Input: digital TTL level (0 to 3.5 V: 10 mA) Encoder Channel B output
3	ENC_A	Input: digital TTL level (0 to 3.5 V: 10 mA) Encoder Channel A output
4	EMC_INDEX	Input: digital TTL level (0 to 3.5 V: 10 mA) Encoder Index output
5	HOME	Input: digital TTL level (0 to 3.5 V: 10 mA) Encoder Home output
6	LIM_1	Input: digital TTL level (0 to 3.5 V: 10 mA) Limit #1 sense output
7	LIM_2	Input: digital TTL level (0 to 3.5 V: 10 mA) Limit #2 sense output
8	N/C	No connection

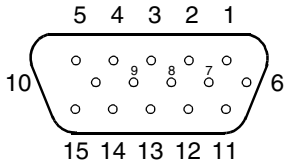
Pin number	Signal	Description
9	Ground	
10	MOTOR_C	Output: Analog level (0 - 24 V: 1.5 A max) Motor winding C.
11	MOTOR_D	Output: Analog level (0 - 24 V: 1.5 A max) Motor winding D.
12	N/C	
13	MOTOR_A	Output: Analog level (0 - 24 V: 1.5 A max) Motor winding A.
14	MOTOR_B	Output: Analog level (0 - 24 V: 1.5 A max) Motor winding B.
15	N/C	

Keyboard (5-pin DIN)



Pin number	Signal
1	Keyboard Clock Output: VOL = .45V @ 10 mA and VOH = 2.4V @ -2.6 mA
2	Keyboard Data Input: VOL = .45V @ 10 mA and VOH = 2.4V @ -2.6 mA
3	Not connected
4	Ground
5	+5V. Output: Keyboard power

Display (15-pin D Female, High Density)



Pin number	Signal
1	Red Output: analog level (0 - 2V @ 25 mA)
2	Green Output: analog level (0 - 2V @ 25 mA)
3	Blue Output: analog level (0 - 2V @ 25 mA)
4	N/C
5	Ground
6	Red Ground
7	Green Ground
8	Blue Ground
9	N/C
10	Ground
11	Reserved
12	Reserved
13	Horizontal Sync Output: TTL level (0 to 3.5 V: 10 mA); Negative True -31.9 kHz
14	Vertical Sync Output: TTL level (0 to 3.5 V: 10 mA); Positive True - 70 Hz
15	Reserved

PCS Assembly Input Power Requirements

+15V @ 400 mA maximum

-15V @ 400 mA maximum

PCS Assembly Input/Output Connectors

PD (BNC Connector)

PD	Output: TTL level: (0 to 3.5 V @ 24 mA)

AMP Out (BNC Connector)

AMP OUT	Output: Analog level: 0 to 5V @ 10 mA (for test only)

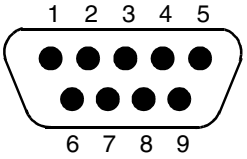
Input (SHV Connector)

INPUT	Input/Output: Analog level: 0V to 2600V @ 110 μA maximum

Bias (SHV Connector)

BIAS	Input: Analog level: 0V to 2600V @ 260 μA maximum

Power (9 pin D Male)



Pin number	Signal	Description
1	HVSET	Input: Analog level: 0 to 12V @ 6 mA
2	LLD	Input: Analog level: 0 to 12V @ 6 mA
3	HVINT	Input: TTL level (0 to 3.5 V: 10 mA)
4	-15V	Input: -15V @ 400 mA maximum
5	SCAL	Output: TTL level (0 to 3.5 V: 24 mA)
6	ULD	Input: Analog level: 0 to 12V @ 6 mA
7	Ground	
8	Ground	
9	+15V	Input: +15V @ 400 mA maximum

Installing P-10 Gas

The flow proportional counter requires a special gas called P-10. Tanks of P-10 gas are available from local bottled gas suppliers. The tank of P-10 should have a two-stage regulator with a 1/4 NPT fitting to accept the NORAN-supplied third stage regulator.

WARNING: P-10 gas is a HAZARDOUS MATERIAL. Handle P-10 gas in accordance with the manufacturer's Material Safety Data Sheet.

WARNING: Fasten the tank of P-10 gas securely to a fixed object, such as a wall or workbench, to avoid damage to the tank. The tank contains hazardous material under pressure. The tank is dangerous if its valve is damaged or the tank is punctured.

Tools

The following tools are necessary for the installation:

- 5/8-inch open-end wrench

Instructions

1. If a two-stage regulator is not installed on the P-10 gas bottle, install it now.
2. Wrap the open end of the 3-inch brass nipple with Teflon™ tape.
3. Remove the 1/4 NPT plug on the two-stage regulator and attach the third stage regulator in this opening.
4. Close the valves on the two-stage and third-stage regulators.
5. Open the valve on the bottle of P-10 gas. Make a note of the total pressure.
6. Open the second stage regulator valve.
7. Open the third stage regulator to a reading of 2 psi on the valve on the supplied gauge.
8. Close the bottle of P-10 gas.
9. Attach an in-line filter to the outlet side (top) of the flowmeter. Make sure that Teflon tape is used on all fitting connections.
10. Attach the flow meter inlet (bottom) hose barb to the hose barb on the third stage regulator.
11. Attach tubing from the flow meter, filtered, outlet barb to the spectrometer.
12. Attach a small length of tubing from the vent barb and place it in a small beaker of water.
13. Open up the main valve of the tank of P-10 gas. There should be 2-3 bubbles every second coming from the tubing in the beaker.
14. Remove the tubing from the beaker of water.

Maintenance

Converting Input to 230-Volt AC for Europe

The switching power supply has a universal input autoranger that eliminates the requirement of changing jumper wires or switches to change the input voltage range. Nevertheless, the correct fuse(s) must be installed according to the input AC voltage.

- For 100/120 VAC operation, use one T 250V 3A fuse
- For 230/240 VAC operation, use two T 250V 2A fuses (5x20mm)

Parts Required

- Fuses: 2A 5x20 mm fuses (2A T 250V), quantity = 2. NORAN Instruments part number 510A126840
- Continental European power cord, quantity = 1. NORAN Instruments part number 600A125780

Procedure

1. Remove the fuse block/cover in the Corcom PEM using a small blade screwdriver or similar tool.
2. Remove the fuse and unscrew the fuse holder form the plate.
3. Flip the fuse holder over so that it can accommodate the two 5x20mm fuses.
4. Insert the 2A T 250V fuses and screw back together.
5. Replace the fuse block/cover in the Corcom PEM.
6. Connect the Continental European style power cord to the PEM.

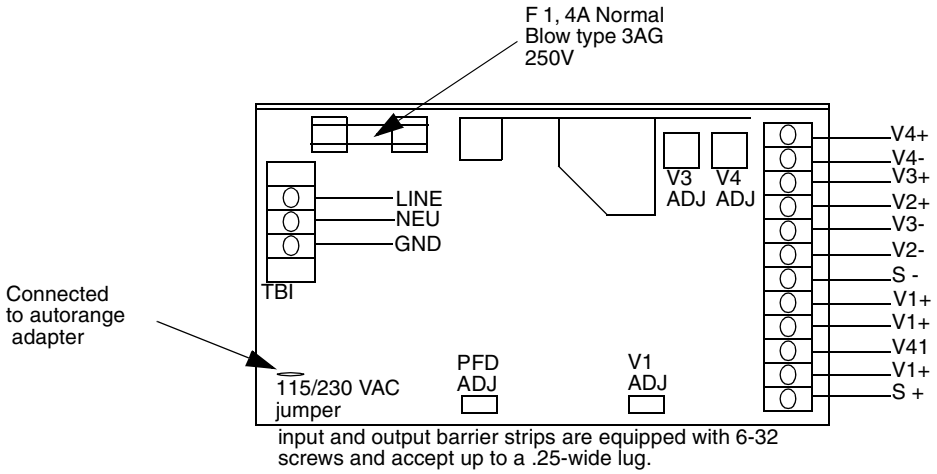
Changing a Blown Fuse in the Controller Switching Power Supply

Parts Required

4A Normal Blow – Type 3AG 250V (Littelfuse 312004 or equivalent)

Fuse Location

The fuse (designator F1) is located on the power supply circuit board adjacent to terminal strip TB1.



Procedure

1. Remove the detachable Ac power cord from the controller.
2. Remove the cover screws and cover.
3. Remove the cable clamp from the base of the chassis near the air baffle.
4. Remove the following DC power harness connectors:
   - P11 – 12-pin plug from the PC motherboard
   - FD – 4-pin plug from the floppy drive.
   - P3 – 2-pin plug from the regulator board
   - P4 – 12-pin plug from the regulator board
   - P1 – 5-pin plug from the ECS board
5. Move these plug ends alongside the power supply.
6. Remove the four fan bracket screws and the six air baffle screws.
7. Remove the fan bracket and the air baffle.
8. Remove the two screws (8/32-by-38-inch) from the front panel to free the power supply.
9. Lift up the power supply and rotate it out of the chassis.
10. Remove the four corner screws from the power supply cover and remove the cover.
11. Remove the two screws holding the autorange adapter to the power supply sheet metal frame. Carefully set it aside.

NOTE: The mounting surface is covered with thermal grease.

12. Find the F1 fuse under the former position of the autorange adapter.
13. Pull the existing fuse out of its holder and insert the replacement.
14. Reverse the above procedure to reassemble the controller. Be careful not to pinch wires when you install the fan.

2 – WDS Software Revisions

Version 3.1.2 Revisions

Revision Notes for Thermo NORAN WDS Software Version 3.0.1, April 18, 2001

New Windows Software Features

15. A new method for setting up default detector bias voltages for each x-ray line has been added to the WDS software for the IbeX, MAXray and MAXray ER. Instead of making measurements at boron and silicon energies, computing the slopes and intercepts, and then entering them in the drtwds.ini file, this new method requires only one measurement, and the measured data can be entered directly into the WDS program (See “Bias Offset Correction” in the *IbeX* or *MAXray Installation and Support Manual*).

    This feature can be turned On or Off in the new Bias Offset dialog box. The Bias Offset dialog box is opened from the new Service > Bias Offset menu selection.

16. During hardware installation it is no longer necessary to make the Baseline Slope and Intercept measurements. All baseline values now default to 0.3 volt in this new software version.
17. The turret move commands have been added to the Service > Send To dialog box.
18. The EMSA file format containing XY data types used by the Vantage software can now be loaded.

    NOTE: EMSA files cannot properly be loaded as overlay spectra.

Windows Software Changes

1. The Edit > Scan Parameters dialog box now remembers the last element whose data was displayed and uses this when this dialog box is opened.
2. The program no longer puts up a message box when hardware is being initialized for a WDS acquisition during quantitative analysis.

Windows Software Fixes

1. When the Edit > Scan Parameters dialog box was opened, the scan parameters for the element Be was displayed. If a Be diffractor was not installed on the turret, the dialog box would not permit any other element's parameters to be edited. This has been corrected in version 3.1 by using the last element's data as the opening data displayed. The first time this dialog box is opened, it displays the parameters for the lowest atomic number element that is available on the particular instrument.

2. The default background energy positions used in quantitative analysis were valid for the MAXray, whose high energy range limit is 2 keV. With the introduction of the MAXray ER, we discovered that the calculation of the background positions for peak energies above Si did not give valid values. This has been corrected in version 3.1 and the new default values are computed when the Restore All button in the Edit > Scan Parameters dialog box is used. The values for one element at a time may be set by clicking on the Restore (atomic symbol) button in this dialog box.

   It is necessary to click the Save button to record these new settings.

3. When performing combined EDS-WDS quantitative analysis, the WDS peak background is now subtracted correctly.
4. The program now prints properly to a printer setup for 600 dpi.
5. The LiF energy range in the Settings > Configuration dialog box is now displayed correctly.

Verision 3.2.1 Controller Software

Features

1. Now Controls both the IbeX and MAXray in the same program.
2. Controls the new Beam Current Meter if it is installed. See *Chapter 6 – Beam Current Option (Model A214)*.
3. New commands have been added to control the Beam Current Meter.
4. Displays run time diagnostics using 25, 30, 43 or 60 rows.

Version 3.0.0 Revisions

Revision Notes for Thermo NORAN WDS Software Version 3.0.0, February 18, 2000.

New Windows Software Features

1. The new NORAN Beam Current Monitor is now supported, both manually, through the use of the Settings > Beam Current dialog box, and automatically in the quantitative analysis section of the software. Both beam current and specimen current can be measured, and if the microscope supports a mechanically operated Faraday cup, it can be activated automatically by the software.
2. Support has been added for the integration of the IbeX and MAXray WDS systems in the Vantage software. This includes both qualitative and quantitative analysis.
3. The user entered parameters in the Edit > Acquisition Information dialog box are retained for use in the next scan automatically unless they are cleared.
4. One or both of the overlay spectra Acquisition Information parameters can be printed along with the foreground spectrum parameters beneath the spectrum. Print Preview can be used to view the overlay spectra acquisition parameters.
5. The Controller software now displays all boot sequence tests along with information that identifies whether a particular test passed or failed.

6. The elements labeled by the user are now added to the spectrum file. When the file is loaded, this information is used to label the peaks. Older spectrum files can still be read, but only the new file format is used when saving spectra. The EMSA file format has also been changed to allow saving and loading of labeled elements. Any previously stored EMSA files can also be loaded with this version.
7. The calibration information is now passed from the controller to the Windows computer when the system configuration is read.
8. The Send Command dialog box now has a list box of commands to select from.
9. There are now four distinct sounds corresponding to Ok, Problem, Alert and Question.

   These sounds can be toggled ON or OFF from the Options menu.
10. The Peak Search dialog box has been enhanced to now include settings for the scan range, step size and dwell time.
11. The peak position found using the Peak Search scan can now be recorded and used as the default energy for the selected element.
12. Communication ports COM3 and COM4 can now be used, along with COM1 and COM2, to operate the spectrometer controller. This allows the WDS software to be used along with a wider variety of NORAN applications.

Version 3.0.0 Changes

1. The Windows program version number and the Controller software version number now match, making it easy to identify compatible versions of both.
2. More space has been allocated for displaying the file name in the Edit > Acquisition Information dialog box, and when printed, the file name appears without the path.
3. The Service > Turret Scan menu selection is disabled for IbeX spectrometers after the Service > Initialize Motors function has been performed.
4. The Settings > Beam Current menu selection is disabled when a beam current meter is not attached to the controller.
5. The default file name extension for a spectrum file has been changed from .wds to .wdx to eliminate confusion with the Vantage software. The original .wds type of files can still be loaded, and both .wds and .wdx file names are displayed in the Open File dialog box list. Only .wdx type files can be saved from the Save and Save As menu selections.
6. The Rate Meter Smooth check box is checked by default whenever the Rate Meter dialog box is opened.
7. The diffractor name is now displayed when moving the cursor over the Periodic Table.
8. The Configuration dialog box can be left open while the application is running.
9. The ‘Damp' check boxes in the Rate Meter and Beam Current Meter dialog boxes have been changed to ‘Smooth' to better explain the averaging effect.
10. The Rate Meter dialog box has been enlarged to permit better visibility from farther away.
11. The Service menu items requiring a password are grayed out until a password is entered.

Version 3.0.0 Fixes

1. The memory corruption problems associated with stopping a scan have been corrected.
2. The problems associated with loading and displaying overlay files has been fixed.
3. The ECS card type used in the controller is now recorded correctly.
4. The crystal 2d spacing in the Settings > Configuration dialog box is now reported correctly to three decimal places.
5. The analog rate meter no longer plots below the zero baseline.
6. All selected acquisition parameters are now printed along with the spectrum, and the parameter names printed match those in the Edit >?Acquisition Information dialog box.
7. The loaded spectrum now changes its file name to reflect the path it was loaded from.
8. No elements are highlighted when the Scan Periodic Table dialog box is opened.
9. The program no longer crashes on exit using the File > Exit menu item.
10. The controller now allows you to add and save the first calibration point when starting a calibration sequence.
11. Old spectrum files will not be over—written without a warning.
12. The filename extension is now only added if it is not entered as part of the name.

3 – MAXray Parallel Beam Spectrometer

Ultrahigh Resolution and Intensity at Low Beam Voltage

The new MAXray Parallel Beam Spectrometer is a revolutionary new product that extends the power of microanalysis for FESEMs and defect review tools.

The MAXray spectrometer is ideal for microanalysis, especially for the analysis of low energy x-ray lines. A NORAN Instruments' exclusive reflective optic placed close to the sample, like a solid state detector, converts a divergent beam of x-rays to a parallel beam. Parallel Beam Spectrometry (PBS) combines the resolving properties of wavelength-dispersive spectrometry with the sensitivity of a large area energy-dispersive detector.

The combination of detector design and proximity to the sample used in PBS provides great improvement in low energy resolution and sensitivity over energy-dispersive spectrometry.

The MAXray is integrated with the VOYAGER/VANTAGE Defect Analysis and EasyEDS software. When a peak overlap appears in an EDS spectrum, an ultrahigh-resolution scan is performed over the region in question and a new spectrum window is opened to display the results in a familiar energy scale. The overlapped EDS peaks are then accurately identified and labeled using the MAXray scan results.

Applications

The MAXray Parallel Beam Spectrometer is ideal for applications that require superior light element x-ray detection, and for applications that use low beam currents and accelerating voltages, such as:

- analysis of small particles
- analysis of beam-sensitive samples
- thin film analysis

In addition to unequaled intensity performance for Be, B, C, N, O, and F, the MAXray spectrometer also identifies overlapping peaks for materials or applications such as:

TiN	WSi2	CrO
N and Ti	O and V	O and Cr
F and Fe	Na and Zn	Si and W
Si and Ta		

Interface

Because of its innovative design, the MAXray spectrometer interfaces with virtually any defect review tool or SEM. A manually operated gate valve is offered as an option to provide vacuum isolation between the MAXray and the SEM chamber.

MAXray Specifications

MAXray Spectrometer

- NORAN Instruments, exclusive grazing incidence reflective x-ray optic
- Sample-to-optic distance: 20 mm for improved solid angle for light elements
- Optic optimized for 100 eV to 1.8 keV detection
- Improvement in intensity (counts/second/nanoamp) of up to 50 times over conventional wavelength-dispersive spectrometers, depending on the element of interest
- Horizontal or vertical orientation. In a vertical mount, the narrow profile creates no interference with instruments on other ports.
- Parallel x-ray beam allows the use of large, flat, undistorted diffractors
- Solid angle of x-ray collection is determined by the distance from the sample to the x-ray optic, and is unaffected by the distance to the diffractor
- Retractable slide for use when the optic must be moved away from the sample
- The MAXray spectrometer converts a divergent beam of x-rays to a parallel beam, so no slit is needed. High resolution is maintained without sacrificing count rate.
- Broadband x-ray optic and 6-diffractor turret to cover the energy range of 100 eV to 1.8 keV
- Gas flow proportional counter with integrated pulse processing electronics
- Reliable and proven in-vacuum servomotor positioning of the detector and diffractors
- Independent microprocessor controller for movement and data acquisition
- Optional gate valve for isolation from the microscope column
- Backed by the NORAN Instruments support network

Control and Data Acquisition Electronics

- Controller – Electronics and 3-axis motor controller in single tower format
- Timer – 0.1 second resolution, 9,999 seconds (max),
- SCA counter – 650,000 cps (max.), 9,999,999 counts (max.), Control DACs for bias, baseline, window
- Dot mapping – Analog only, providing TTL pulse output, 1-50 µs duration
- Data acquisition electronics – Integrated preamplifier, amplifier, single channel analyzer (SCA) and bias supply, Remotely programmable baseline, window and HV bias, Baseline and window programmable over full range of amplifier
- SCA output –TTL pulse, 300 ns width, Analog amplifier output: 0-5 volt, Gaussian
- HV bias supply – 1325-2600 volts

Software

- Fully integrated with VOYAGER and VANTAGE Defect Analysis and EasyMicro software
- Automatically launches in overlap conditions
- Automated calibration
- Automatic peak identification and labeling
- Prior experience with WDS is not required for operation
- Additional software training is not required
- Built-in manual override of all acquisition parameters
- Spectral display in energy units (keV)
- Energy cursor and KLM markers
- Peak ID hinting
- Resizable window for ease of use on the desktop
- Convenient menu selections for loading and saving data
- Pre formatted printouts for fast report generation
- Physical Dimensions (approximate)
- Spectrometer – Max. 17.50 W X 11.88 H X 6.19 D in.; 44.5 W X 30.2 H X 15.8 D cm.
- Weight – 57 lb (26 kg).
- Controller, tower format – 9 W X 15 H X 23 D in.; 23 W X 38 H X 58 D cm.

MAXray - Standards

Element/Line	Standard	Diffractor	keV	FWHM(eV)	CPS/nA
Be K	Be	0V200H	10	6	600
B K	B	0V200H 0V145H	10	16 9	6000 4400
C K	C	0V100N 0V080E	10	13 11	6000 1280
N K	BN	0V080E	10	9	474
O K	SiO2	0V060	10	12	336
Fe L	Fe	0V060 TAP	10		185 32
Mg K	Mg	TAP	10		477
Si K	Si	TAP	10	15	300

MAXray ER Specification

Spectral Line (a)	Elemental Standard[1]	Energy (keV)	Diffractor[2]	Acclerating Voltage (keV)	Resoluction[3] (eV)	Intensity[4] (cts/sec/nA)	Peak-to-Background	C_{DL}[5] (ppm)
Be	Pure Be	0.108	NOR200H	5	11	546	69	54
B	Pure B	0.185	NOR200H	5	16	5000	56	20
B	Pure B	0.185	NOR80N	5	9	1100	43	48
C	Pure C	0.282	NOR80N	5	16	3000	113	18
N	BN	0.392	NOR80N	5	19	62	5	591
N	BN	0.392	NOR80N	5	16	530	20	101
O	SiO2	0.523	NOR60A	5	17	208	40	114
Al	Al	1.487	TAP	10	21	485	241	30
Si	Si	1.740	TAP	10	26	416	255	32
Si	Si	1.740	PET	10	8	264	796	23
Sc	Sc	4.090	PET	10	38	40	107	160
Fe	Fe	6.403	LiF	20	46	147	193	62
Cu	Cu	8.046	LiF	30	62	159	157	66

Notes

1. The NORAN smaple standards are part number 555A135192.
2. “NOR” is a multi-layer synthetic diffractor.
3. Resolution is Full Width Half Maximum (FWHM).
4. Intensities are peak counts normalized to time and beam current measured with a Faraday cup.
5. Background positions for synthetic diffractors are peak energy $\pm 1/5\lambda$, and peak energy $\pm$ 2/3(K_−Kb) for natural diffractor.

6. Concentration Detection Limit (C_{DL}) is calculated using the following formula (Ziebold, 1967):

   $C_{DL} \geq 3.29/(T \times P \times P/B)^{1/2}$

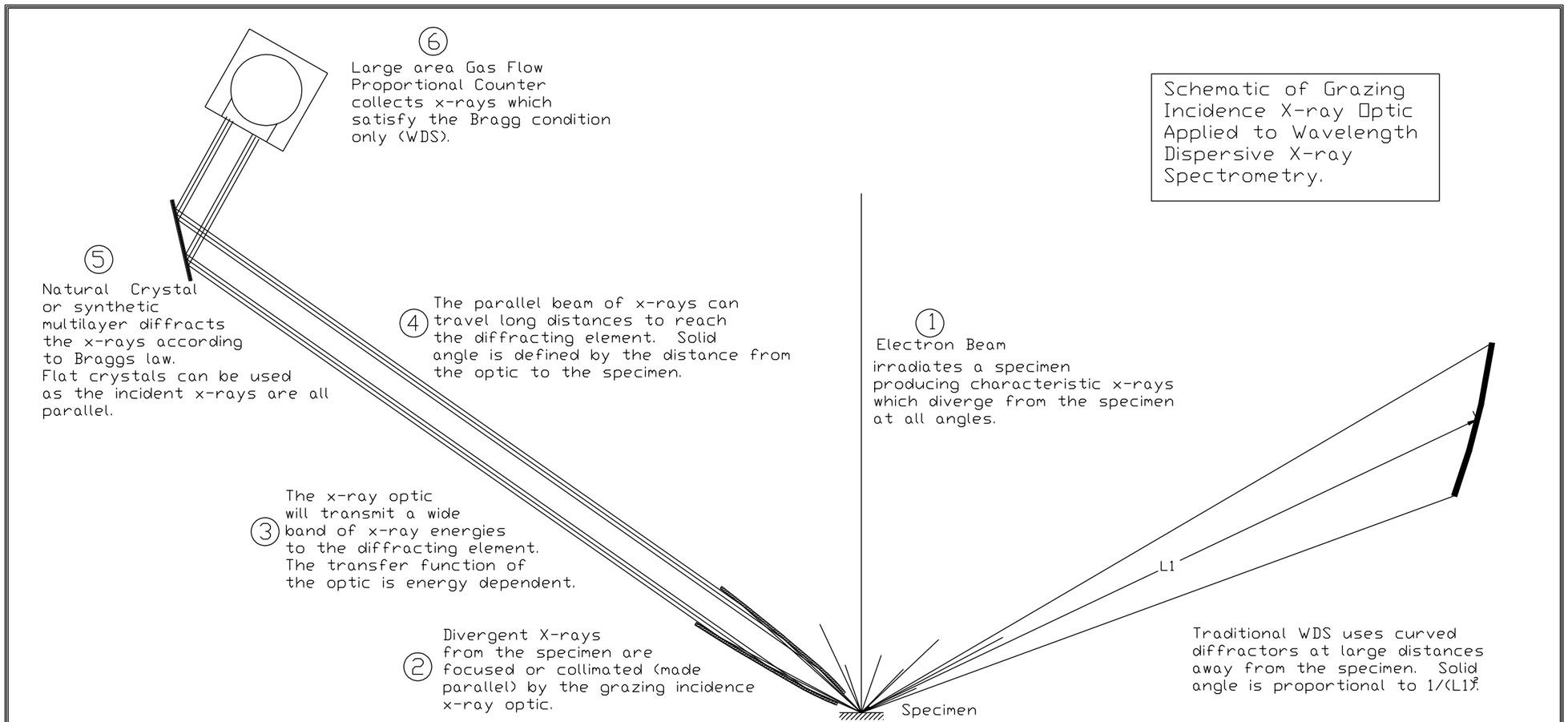
   Where: T= total count time (1000 seconds)

   P= peak count rate (0.1 uA specimen current)

   P/B= Peak-to Background ratio

7. Guaranteed values are 80% of published specifications.
8. Data is collected with a smooth, flat specimen and x-ray take-off angle of 35^{o}.

Schematic Diagram of PBS Spectrometer



MAT'L	MADE BY JVH	DATE 08-08-97
FINISH	RELEASE APPROVAL	DATE
UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES ANGLES ±1° 2 PLACE DECIMALS ±.010 MACHINED INSIDE RADIUS .015 MAX. 3 PLACE DECIMALS ±.005 MACHINED SURFACES 63 HOLES ⌖ .005 BREAK SHARP CORNERS .005 - .010	NORAN Instruments TITLE PBS SCHEMATIC DRAWING FOR ILLUSTRATION	A JVH 03-10-98 REV NO. DATE REVISIONS PART NUMBER ENG. 930A135970 PI

MAXray Allowable Bragg Angles for Various Diffractors

keV	Element	Line	Lambda (A)	2d=	200	160	145	120	100	80	60	50	45	35	25.75 (TAP)	13.4 (KAP)	8.742 (PET)
0.109	Be	K	113.747		4.0000	45.3097	51.6710										
0.185	B	K	67.016		19.5775	24.7621	27.5279	33.9499	42.0796	56.8983							
0.282	C	K	43.965			15.9488	17.6501	21.4920	26.0813	33.3366	47.1168	61.5569					
0.331	Mo	M	37.456					18.1879	21.9972	27.9177	38.6286	48.5145	56.3419				
0.341	Ca	La	36.358					17.6368	21.3201	27.0310	37.2982	46.6485	53.8962				
0.355	Nb	Mg	34.924						20.4408	25.8839	35.5960	44.3051	50.9037				
0.392	N	K	31.628						18.4378	23.2875	31.8115	39.2386	44.6549	64.6413			
0.395	Sc	La	31.387						18.2928	23.1003	31.5419	38.8841	44.2265	63.7381			
0.452	Ti	La	27.429						15.9198	20.0515	27.2036	33.2699	37.5561	51.5998			
0.461	Ru	Mg	26.894						15.6010	19.6438	26.6301	32.5390	36.7009	50.2097			
0.496	Rh	Mg	24.996							18.2069	24.6201	29.9947	33.7428	45.5753			
0.511	V	La	24.262							17.6546	23.8517	29.0285	32.6265	43.8845			
0.523	O	K	23.706							17.2367	23.2717	28.3015	31.7888	42.6331	67.0145		
0.532	Pd	Mg	23.305							16.9362	22.8555	27.7808	31.1900	41.7470	64.8273		
0.568	Ag	Mg	21.827							15.8335	21.3332	25.8839	29.0161	38.5826	57.9590		
0.573	Cr	La	21.637							15.6918	21.1381	25.6416	28.7391	38.1848	57.1688		
0.606	Cd	Mg	20.459								19.9365	24.1530	27.0417	35.7702	52.6094		
0.637	Mn	La	19.463								18.9283	22.9083	25.6272	33.7857	49.0996		
0.677	F	K	18.313								17.7713	21.4853	24.0139	31.5493	45.3318		
0.691	Sn	Mg	17.942								17.3997	21.0291	23.4978	30.8393	44.1693		
0.705	Fe	La	17.586								17.0435	20.5923	23.0040	30.1623	43.0742		
0.733	Sb	Mg	16.914								16.3737	19.7722	22.0780	28.8984	41.0606		
0.776	Co	La	15.977								15.4430	18.6349	20.7959	27.1602	38.3496		
0.778	Te	Mg	15.936								15.4023	18.5852	20.7400	27.0847	38.2332		
0.833	La	Ma	14.884									17.3178	19.3140	25.1661	35.3101		
0.848	Ne	Ka	14.620									17.0020	18.9592	24.6909	34.5954		
0.851	Ni	La	14.569									16.9403	18.8899	24.5980	34.4562		
0.883	Ce	Ma	14.041									16.3089	18.1808	23.6510	33.0433		
0.929	Pr	Ma	13.346									15.4805	17.2515	22.4143	31.2165		
0.930	Cu	La	13.331									15.4635	17.2324	22.3889	31.1792		
0.972	Ba	Mg	12.755										16.4661	21.3727	29.6925		
0.978	Nd	Ma	12.677										16.3622	21.2352	29.4923		
1.012	Zn	La	12.251										15.7979	20.4890	28.4093		
1.041	Na	Ka	11.910										15.3468	19.8938	27.5494		
1.081	Sm	Ma	11.469											19.1284	26.4488	58.8589	
1.098	Ga	La	11.291											18.8210	26.0084	57.4205	
1.131	Eu	Ma	10.962											18.2522	25.1955	54.8909	
1.185	Gd	Ma	10.462											17.3932	23.9732	51.3320	
1.188	Ge	La	10.436											17.3479	23.9088	51.1516	
1.240	Tb	Ma	9.998											16.5988	22.8479	48.2578	
1.253	Mg	Ka	9.895											16.4217	22.5977	47.5959	
1.282	As	La	9.671											16.0401	22.0593	46.1953	
1.293	Dy	Ma	9.589											15.9000	21.8619	45.6894	

keV	Element	Line	Lambda (A)	2d=	200	160	145	120	100	80	60	50	45	35	25.75 (TAP)	13.4 (KAP)	8.742 (PET)
1.347	Ho	Ma	9.204											15.2467	20.9432	43.3834	
1.379	Se	La	8.991												20.4352	42.1395	
1.405	Er	Ma	8.824												20.0407	41.1873	
1.462	Tm	Ma	8.480												19.2279	39.2606	
1.480	Br	La	8.377												18.9851	38.6933	
1.486	Al	Ka	8.343												18.9055	38.5082	
1.521	Yb	Ma	8.151												18.4545	37.4667	
1.581	Lu	Ma	7.842												17.7304	35.8181	63.7709
1.586	Kr	La	7.817												17.6727	35.6879	63.4066
1.644	Hf	Ma	7.541												17.0298	34.2488	59.6164
1.694	Rb	La	7.319												16.5125	33.1051	56.8453
1.709	Ta	Ma	7.255												16.3635	32.7778	56.0832
1.740	Si	Ka	7.125												16.0640	32.1230	54.5937

4 – Overview of Parallel Beam Spectrometry and WDS

Parallel beam spectrometry (PBS) provides significant gain improvements over wavelength-dispersive spectroscopy (WDS) on SEMs. WDS in turn provides dramatically better resolution and sensitivity than its energy-dispersive (EDS) counterpart. Because EDS is so widely used, many new SEM users may not have had an opportunity to use WDS, and PBS is such a new development that it might be unfamiliar. The following table summarizes differences among the techniques:

	PBS	WDS	EDS	Significance
Operating principle	Geometry and crystal diffraction allow only photons of a particular wavelength to arrive at the detector, which counts photons. It does not measure energy. PBS uses a special optic and flat crystals, and uses a larger solid angle than WDS.	Geometry and crystal diffraction allow only photons of a particular wavelength to arrive at the detector, which counts photons. It does not measure energy.	Current induced in the detector is proportional to photon energy. The detector determines photon energy, and counts and sorts photons by energy.	Although one technique measures photon energy and the other measures wavelength, the results are the same, because these two parameters are directly related.
Energy/wavelength range	A WD spectrometer is tuned to one energy at a time	A WD spectrometer is tuned to one wavelength at a time.	An EDS detector counts photons of all energies. It evaluates all energy levels at the same time.	EDS provides faster but less accurate results, which is especially useful for data-intensive analysis such as mapping.
Detector Resolution	WDS resolution is limited by calibration accuracy – resolution on the order of 24 eV for manganese.	WDS resolution is limited by calibration accuracy – resolution on the order of 24 eV for manganese.	EDS resolution is reduced by: •Noise in the detector signal current •Incomplete conversion of photon energy to signal current •Resolution accuracy on the order of 130 to 140 eV for manganese	WDS can resolve peaks more precisely than EDS, which is especially useful when overlapping peaks are a problem. Depending on the diffractor and element selected, resolutions of 2 eV to 25 eV are possible with WDS.

Deadtime	WDS deadtimes are short because the detector only needs to count, not measure, photons.	WDS deadtimes are short because the detector only needs to count, not measure, photons.	EDS deadtimes are longer than those for WDS. In an analog system, deadtimes 100 to 1,000 times larger than WDS are typical.	Higher throughputs are possible with WDS.
Element Limits	Detects all elements with x-ray lines below 2 KeV.	Detects all elements having atomic numbers greater than or equal to beryllium, depending on crystal materials and position.	Detects all elements having atomic numbers greater than or equal to beryllium, depending on crystal and window materials.	Both EDS and WDS can detect all elements except hydrogen, helium, and lithium.
Detection limits	Roughly on the order of 0.1 to 0.01 weight percent, depending on the element, or about 10 times better peak-to-background ratio than EDS.	Roughly on the order of 0.1 to 0.01 weight percent, depending on the element, or about 10 times better peak-to-background ratio than EDS.	Roughly on the order of 1.0 to 0.1 weight percent, depending on the element, the detector, the sample, and other conditions.	Use WDS for trace element analysis.
Ease of use	PBS is used only for qualitative analysis.	WDS needs fine tuning for high precision, and element standards are required for quantitative analysis.	Many applications can be fully automated and EDS can perform standardless quantitative analysis.	For quantitative analysis, WDS requires more attention than EDS.

This chapter covers the principles of WDS, describes the dramatic gain increases available with PBS, and provides some examples of PBS capabilities with MAXray.

Principles of WDS

WDS is based on Bragg's law of x-ray diffraction:

Bragg's law: $n\lambda = 2d \sin\theta$ **Equation 1**

where λ is the wavelength of interest
d is the interplanar spacing of the diffractor
θ is the angle of x-ray incidence on the diffractor
n is an integer

and, E (keV) = $12.398/\lambda$ **Equation 2**

> *NOTE: In Equation 2 that Energy and Wavelength are inversely related: short wavelengths are high energy. It is often a source of confusion when EDS users look at a conventional wavelength spectrum and see a Kβ to the left of a Kα. Here, spectra will always be displayed with energy increasing to the right.*

The classic derivation of Bragg's law is as follows. Consider that a parallel beam of x-rays of wavelength λ is made incident on a material whose structure comprises a precisely parallel arrangement of planes (atomic, molecular, or otherwise), with interplanar spacing, *d*:

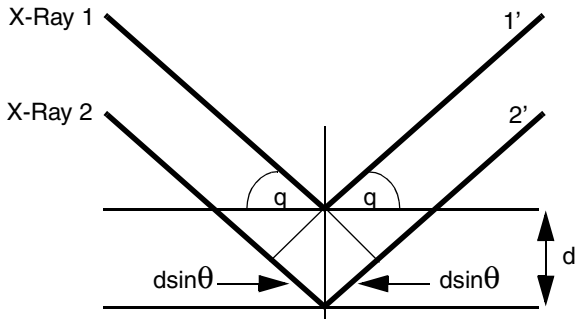


Figure 4-1: Bragg's law derivation

Wave 1 is reflected into 1' from the top plane in the diffractor (or "crystal") with the angle of incidence, θ, equal to the angle of reflection. Wave 2-2', being reflected from the lower plane, travels an extra distance in the material equal to *dsin*θ + dsinθ. For an intense diffracted beam to result, wave segments 1' and 2' must be in phase; which, in turn, requires that the extra path length traveled by wave 2-2' must be an integral number of wavelengths: that is, 2 (dsinθ) must equal $n\lambda$.

If Bragg's law is satisfied, then the emerging waves will add in strength. If not, there will be destructive interference, with one wave cancelling another, and only a very weak beam will result. As the crystal is scanned through an angular range, the diffractor becomes a highly reflective mirror only at the Bragg angle; the specificity of the angle of high reflectivity is the key to the high resolution of WDS.

WD spectra are acquired by scanning a diffractor and detector through a range of angles. As Bragg's law is satisfied for various wavelengths being emitted from the sample, sharp peaks result. Peak width is typically from 2 to 25 eV, compared to 80 to 160 eV in EDS. High resolution leads to high peak-to-background ratio (P/B), P/B controls sensitivity and minimum detection limits for both analysis and mapping.

Since the angle of incidence equals the angle of reflection, a detector or counter must be positioned at the same angle relative to the diffractor as the incident x-ray beam makes with the diffractor itself. Relative to the incident beam, the diffractor is at an angle θ, and the detector at 2θ.

In the derivation of Bragg's law, a parallel beam of x-rays was assumed. In the SEM, however, use of a spot or high magnification raster means that the beam starts essentially at a point and diverges on its way to the diffractor.

In PBS, a special collimating optic is placed close (20 mm) to the surface of the sample. Divergent x-rays passing through this optic are converted into a parallel beam of x-rays. This beam strikes a large, flat diffractor and is diffracted into the window of a gas-filled proportional counter. Since the parallel beam does not have to be focused, as in conventional WDS, the distance between the sample and diffractor does not have to equal the distance between the diffractor and the detector window, allowing the detector to be moved closer to the diffractor. The Bragg angle relationship still must be met, though.

Implications of the nature of PBS:

1. Compared with EDS, resolution is typically an order of magnitude better; peak overlaps are virtually eliminated.
2. The high resolution leads to order-of-magnitude better sensitivity than EDS. The sensitivity improvement is often most dramatic in the light element region.
3. The better P/B and elimination of overlaps yield more accurate quantitative analysis for those elements in low concentration or involved in the overlap.
4. Because the WD spectrometer can be positioned at the center of the peak, where P/B is maximized, WDS provides superior x-ray maps, especially for low concentrations.
5. Due to the serial nature of spectrum acquisition, qualitative analysis is slow compared to EDS.
6. The large solid angle achieved using the PBS optic yields a greater sensitivity to light element energies than conventional WD.
7. WDS is often operated by driving the crystal directly to the peak, with or without peak searches. In this manner, the counting time is devoted only to the points of real interest. For

minor elements, WDS then becomes fast relative to EDS, which spends most of its time counting x-rays from the major elements.

X-ray Optics

Diffractors

The term "crystal" is routinely used to describe the diffractor and, in fact, the device is commonly called a "crystal spectrometer." However, the prominence of the new multilayers, which have greatly improved light element analysis, makes the general term diffractor more appropriate. The term "crystal" implies a regular arrangement or stacking of atoms or ions to form the crystalline structure of metals and minerals. The very efficient multilayer, in fact, are not crystals but rather vacuum-deposited alternating layers of different materials. The "interplanar spacing" is the thickness of the lower atomic number "spacer," while the diffracting planes are the layers of the higher-Z material. Although both terms will be used here, "diffractor" is preferred.

The commonly used diffractor are listed in the table below. Since Bragg's law contains the quantity "2d", diffractors are typically described by their 2d value.

Designation	Full Name	2d, A
PET	Pentaerythritol tetrakis	8.742
TAP	Thallium "acid" phthalate	25.9
W/Sii	Tungsten/Silicon Multilayer*	50-65
Ni/C	Nickel/Carbon Multilayer*	80-100
Mo/B4C	Moly/Boron Carbide Multilayer	140-205

NOTE: The multilayers can be manufactured to any specified 2d value, within, approximately, the ranges shown.

The crystal *d*-spacing must be of the same magnitude as the wavelength of the x-ray to be measured. For example, the CKα x-ray, at 0.282 keV, would require NiC (a synthetic crystal) with a 2d value of 100 angstroms. The SiKα x-ray, at 1.74 keV, would require TAP (a natural crystal) with a 2d value of 25.75 angstroms.

A practical requirement in crystal selection is that diffraction must occur within the angular range of the spectrometer. The maximum angle allowed in the MAXray is 68°, and the minimum angle is 14°. If CKα, at 0.282 keV, is to be analyzed, the NiC (2d=100 angstroms) is suitable. Using Bragg's law (Equation 1), the CKα peak on NiC is at 25.26°.

Performance is another consideration in choosing a diffractor. Consider one of the more involved choices, that for boron, specifically, and for light elements in general.

Detectors

Wavelength spectrometers almost exclusively use gas proportional counters. X-rays diffracted from the crystal enter the counter and cause ionization of the contained gas. There is a central wire along the height of the detector (in a direction normal to the sample-crystal-detector plane, parallel to the crystal width), to which a bias voltage of typically +2000 volts is applied. The electrons freed by the ionization process then are accelerated to the wire, creating new ionizations in their path, ultimately producing a current pulse that is proportional to the energy of the absorbed x-ray. As in EDS, the current pulse is converted to a proportional voltage pulse in an field-effect transistor (FET), amplified, converted to a digital signal and counted. Unlike EDS, however, the FET is held at room temperature, and the digitizing is done in a single channel analyzer (SCA) rather than in an analog-to-digital converter (ADC). The EDS system's ADC must retain the proportionality to the original x-ray energy, so that a count can be stored in the appropriate channel of a multichannel analyzer. In WDS, the resolution is achieved by the diffractor, and the SCA simply allows selection of the upper and lower limits of the size of the amplified voltage pulse that will be counted.

Since the counter contains a gas, a window must be used to isolate the inside of the detector from vacuum; if low energy x-rays are to be analyzed, the window must be thin. The detector is called a gas-flow, or flow proportional counter (FPC). An Argon-10% methane mixture, called P-10 Gas, is the normal counter gas.

Some fully focusing spectrometers use a dual counter system, with the SPC directly coupled to and behind the flow counter. An exit window in the FPC allows unabsorbed higher energy x-rays to enter and be counted in the SPC. About 2/3 of the CuKα intensity, for example, will come from the rear counter.

The speed and low noise characteristics of the proportional counter account for two additional advantages of WDS over EDS:

1. The counter can handle very high count rates, up to about 10 Kcps, without dead time, or even up to 100 Kcps in some cases. If the spectrometer is set to a peak position, and say 20 Kcps can be achieved, all that count-rate is in the element of interest. Compare this to EDS, which may be limited to a few thousand counts per second over the entire spectrum.
2. Its low noise contributes to the excellent light element capability of WDS. EDS detectors, which are cooled to near-liquid nitrogen temperatures to reduce inherent thermal noise, continue to fight that noise as their application is stretched to lower low-energy thresholds.

Spectrometers

Interfacing Requirements

The multielement spectrometer can be adapted to any SEM chamber. Since crystals remain within the spectrometer housing, only a very small port is required.

The IbeX Multielement Spectrometer

In addition to the MAXray spectrometer, NORAN offers another dispersive spectrometer called IbeX.

In the IbeX multielement device, *L* is fixed, and the diffractor simply rotates to achieve the desired angle with the incident beam. It is the elimination of the *L* movement that gives the multielement spectrometer its defining characteristics. The fixed *L*:

- Permits easy interfacing to nearly any SEM chamber. Since the crystals do not enter the chamber, even very small ports can be used.
- Reduces the housing size considerably. The multielement spectrometer is about one-third the weight and half the linear dimensions of full-range units.
- Reduces mechanical complexity and therefore cost. The multielement spectrometer is about one-half the price of fully-focusing units.
- Restricts its application to specified elements or energy ranges. Although focusing is exact for only one wavelength per crystal, small errors in *L* can be tolerated, which means that good performance can be maintained over several degrees of scanning.

For the elements designated (one per crystal), expected WDS performance is achieved. For the light elements, performance can even be superior to fully focusing units, because the best diffractor can be used for each element designated.

In the IbeX, *L* is typically 20 or 25 cm (8 or 10”) depending on chamber size. Up to six diffractors can be used in a given spectrometer, each optimized for a specific element by selection of the best material and d-spacing, and then curved to the radius required by the focusing equation. Each diffractor will be curved to a different radius (correct for focusing its designated element). (Each diffractor, therefore, is on a unique Rowland circle.) The spectrometer permits selection of the diffractor and its rotation to a specified angle or through a range of angles. The detector (a flow proportional counter) moves along an arc, maintaining a constant detector-to-diffractor distance (*L2*=*L*), to its 2θ position (see Figure 4-2). Further, if the diffractor is rotated to an angle other than the one for the crystal's designated element, Bragg's law can be satisfied, but not the focusing equation (since θ changes, but both R and L are fixed). Consequently,

multielement spectrometers can be scanned through an angular range to pick up other x-ray lines, but will suffer loss in performance as the angle deviates from the optimum. The larger the deviation, the less results compare favorably with fully-focusing systems. Fortunately, however, for moderate scanning ranges, reasonable performance is achieved.

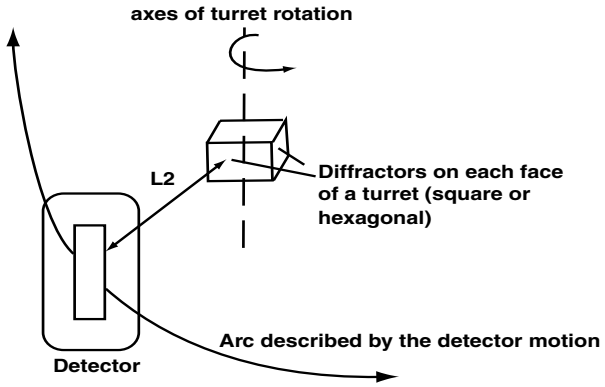


Figure 4-2: Multielement spectrometer motion.

The following spectrum, from albite, was obtained in a multielement spectrometer by scanning the TAP crystal through an angular range of about 12$^{\circ}\theta$.

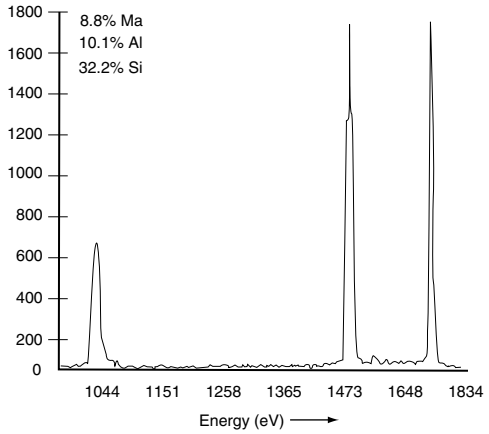


Figure 4-3: A mutlielement spectrometer scan, Na-Si.

The improved resolution and Peak-to-background over EDS, despite moving off the focusing condition, is significant. Note that, despite the higher Si concentration, the Al and Si peak heights are nearly equal, due to the diffractor being optimized, in this case, for Al. Crystal radius plays a significant role in the relative peak heights obtained with this type of spectrometer.

Achieving Required Motions in IbeX

Wavelength spectrometers require precise and repeatable mechanical movements. Conventional spectrometers utilize complex mechanical linkages to achieve the relative motions of crystal and detector; such linkages are fragile, require frequent adjustment, must be moved slowly and are prone to some degree of backlash. The IbeX eliminates these linkages, utilizing two in-vacuum encoded brushless servomotors to independently position the crystal and detector under computer control. Such motion is referred to as positioning by independent parameterization.

WDS Applications

A typical example of a WDS application is described below.

Setting Up TiN

In TiN, the NKα and the TiLα, Lβ lines are separated by only about 58 eV. Since the peak resolutions at the nitrogen and titanium energies are only around 30 eV, the two peaks can be easily resolved, and a clear distinction can be made between Ti and TiN samples without the need for deconvolution.

A few typical examples of WDS applications, from both types of spectrometers, are given in the following sections.

Resolution of W, Ta, and Si

Another common overlap area in EDS is the Si K with the W and/or Ta M lines. The SiKα is at 1739 eV, while the W Mα is at 1774 eV, and the Mβ at 1835 eV. Less than 100eV separate these three peaks. Ta and Si present an even more severe overlap, with the two Ta M lines bracketing the Si peak by 30 eV on the low side and 25 eV on the high side.

A difficult analytical problem is found in the analysis of thin Ta- or W-Si films used in the semiconductor manufacturing process. Since these films are produced on Si wafers, Si emission from the substrate can overwhelm the Si and Ta or W peaks from the film. To avoid significant penetration into the film, and the resulting high substrate intensity, a low accelerating voltage (less than 10 keV) must be selected. The use of the L-lines of W or Ta is thus precluded, and the overlapped M-lines must be used for analysis. Because of the severity of the overlap, the quantitative precision required can only be obtained by resolving the analyzed peaks, dictating the use of WDS.

The following figure shows spectra from TaSi and WSi. In the WSi spectrum there is a noticeable indication that two Si satellite lines are present just to the high energy side of the Ka. These lines, in fact, are separated by only 2eV (at 1752 and 1754 eV).

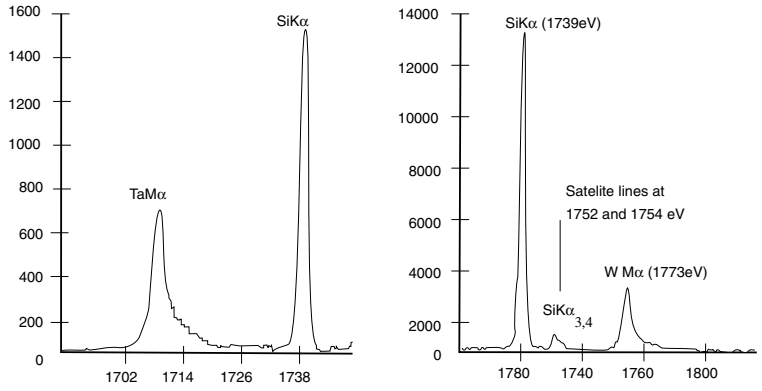


Figure 4-4: WDS spectra from TaSi (left) and WSi (right).
Note the indication of the 2 Si satellite lines, separated by only 2 eV, in the WSi spectrum.

Analysis of Boron In BPSG

BPSG (borophosphosilicate glass) is a thin film used in the semiconductor process. It is important to control both the P and B "dopant" levels to insure proper device yield and product reliability. WDS is one of the best methods for boron determination in BPSG because of its high analytical sensitivity for light elements and its straight forward linear response (in this application) to the boron concentration. NORAN's multielement spectrometer is one of the primary tools used in the industry to perform the analysis;

with the Mo/B_4C multilayer, it is, in this instance, the most efficient and cost-effective WD system available. Figure 3-5 shows an overlay of 4% and 2% BPSG. In the actual analysis, however, peak scans are not done; rather, the spectrometer is simply driven to the B peak position, where counts are recorded for standards and unknowns. A typical (linear) calibration curve is shown in Figure 3-6.

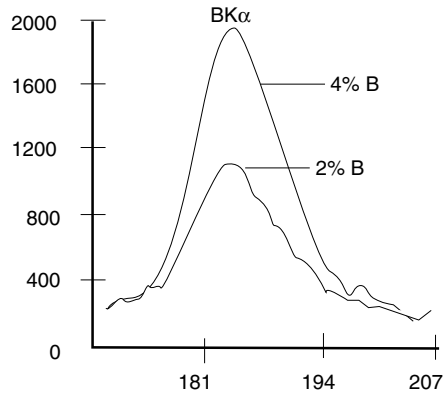


Figure 4-5: Figure 5: 4% B BPSG overlaid with 2% BPSG.

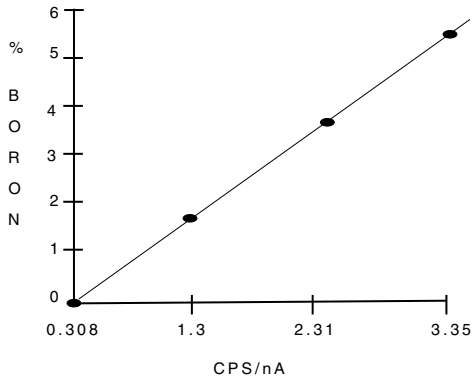


Figure 4-6: Figure 6: Calibration curve for B in BPSG.

Acknowledgments

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References

The following is a brief list of references for those who may wish to delve more deeply into the theory and practice of WDS.

- Bertin, E.P., *Principles and Practice of X-Ray Spectrometric Analysis*, Plenum Press, NY, 1975
- Birks, L.S, *Electron Probe Microanalysis*, Wiley Interscience, NY, 1971
- Reed, S.J.B., *Electron Microprobe Analysis*, Cambridge University Press, Cambridge, 1975
- Goldstein, *et al.*, *Scanning Electron Microscopy and X-Ray Microanalysis*, Plenum Press, NY, 1992

5 – Vacuum Considerations When Using the MAXray Parallel Beam Spectrometer

The MAXray Parallel Beam Spectrometer is a high performance wavelength dispersive spectrometer (WDS) utilizing a Grazing Incidence X-Ray Optic to collect and collimate x-rays. Like any WDS spectrometer, the spectrometer is open to the vacuum system of the electron microscope to which it is attached. This is unlike an EDS type detector where the cryogenic environment of the solid state detector cannot tolerate a direct opening to the vacuum system.

The MAXray spectrometer presents an additional load on the vacuum pump system in an electron microscope. The spectrometer alone contributes the following “loads:”

Spectrometer Volume:	10.4 L
Spectrometer Surface Area:	3390 sq.cm (Not including mechanisms, motors)
Elastomer Seals:	292 cm

Depending upon the microscope vacuum system present, these additional loads will create a higher equilibrium pressure (P=Q/S; where P is the pressure, Q is the throughput in outgassing and leaks, and S is the pumping speed.)

In field emission SEMS, vacuum can be a critical operating parameter. In testing on various FESEMs, the MAXray spectrometer did produce higher base pressures in the microscope chamber, however little to no degradation in the intermediate and gun pressure was seen. The MAXray has been tested up to 5 days on an FESEM without causing degradation in the gun vacuum level. Users will have to tolerate higher sample chamber pressures when operating a MAXray spectrometer.

Other notes concerning vacuum compatibility.

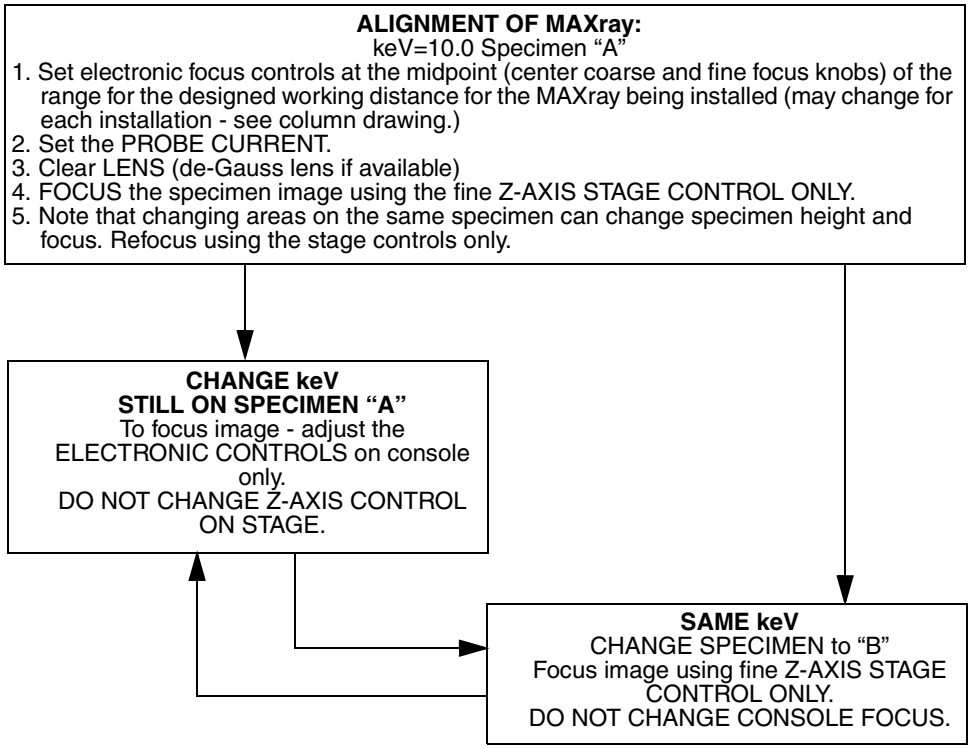
1. The MAXray spectrometer, by virtue of its unique design, is actually coupled to the microscope by a rather poor conductance (3-5 liters/sec). Although this may extend initial pump down, the poor conductance limits the actual load seen by the microscope vacuum system.
2. The MAXray spectrometer does utilize the industry standard Gas Flow Proportional Counter for detecting diffracted x-rays. To enable detection of low energy photons, a thin polymer window isolates the P-10 (Argon/10% methane) from the vacuum system. Any leakage through this window will produce an additional load on the vacuum system. Note also in spectrometers fitted with a gate valve to isolate the spectrometer during chamber venting excessive time isolated from vacuum will cause the pressure to rise in the spectrometer. The

pressure rise can be due to both leakage from the flow counter and outgassing from the spectrometer surfaces.

3. Depending upon storage and shipping conditions, the initial pump down of a MAXray spectrometer can take anywhere from 12 to 36 hours.

6 – Maintaining a Constant Physical Working Distance and Focus

On older SEMs, maintaining the same physical working distance when changing specimens or keV can be challenging. It is important that after alignment of the MAXray spectrometer, all specimens being analyzed must be located at the same physical point in space so that the optic is focused on the specimen surface. The flowchart below illustrates the technique used at NORAN Instruments when working with an SEM for which the apparent working distance and focus is not compensated when changing keV. Additionally, no microscope will compensate the actual WD when changing specimen, i.e., electronic focus will change, but the change in specimen height is not compensated. Therefore the I specimen could be located outside of the focal plane of the MAXray optic.



Note that in the lower loop, you cannot change keV and specimen at the same time. If the loop is broken at any time, you would typically have to return to the baseline established during alignment. The user must become proficient in placing the specimens to be analyzed at the correct stage position. The use of height setting jigs is strongly recommended.

7 – Installation, Alignment and Calibration Procedures

Software Installation Instructions

Install on a new system

1. Place the diskette in the drive.
2. Select Start then Run from the Windows NT task bar.
3. Type the filename a:setup
4. Click on the OK button.
5. Follow the instructions displayed on the screen.

Install on an existing system

1. Backup your data files if they are in the C:\Noran subdirectory.
2. Backup your MAXray software settings.
3. These files are:

   c:\noran\wds\bin\labels.txt

   c:\noran\wds\bin\scan_tab.txt

   c:\noran\wds\bin\user_scn.txt
4. Place the diskette in the drive
5. Select Start then Run from the Windows NT task bar.
6. Type the filename a:setup
7. Click on the OK button.
8. Follow the instructions displayed on the screen.
9. Copy your MAXray software settings back into their original directories.
10. Copy your backed up data back into their original directories.

MAXray Installation

Required Tools

- Metric and SAE Hex Wrench Sets
- 10" Crescent Wrench
- Flat and Phillips Screwdrivers
- MAXray Install/Spares Kit (700P135998)
- Oscilloscope (20 MHz)
- Analog Ratemeter

Installation Procedures

Refer to *Figure 7-1*.

1. Unpack the Slide Interface Assembly from the shipping container. If the **Optic Support Tube** is not installed into the Slide Interface, install it now.

   Remove the **Electron Trap** by loosening the two M2 Screws that hold it in place. Carefully install the **Optic Support Tube** into the front of the **Slide Interface** (flange end), inserting the end of the Support Tube which has 4 tapped holes. Secure the **Support Tube** in the Spectrometer Mount Block using 4 M2 screws and lock washers. Replace the **Electron Trap**.

   Some column designs call for a set screw to secure the X-ray Optic (XO); if required, insert the **Set Screw** into the tapped hole on the side of the **Optic Support Tube**. Leave clearance for the X-ray Optic. Do not adjust any other screws on the Slide Interface.

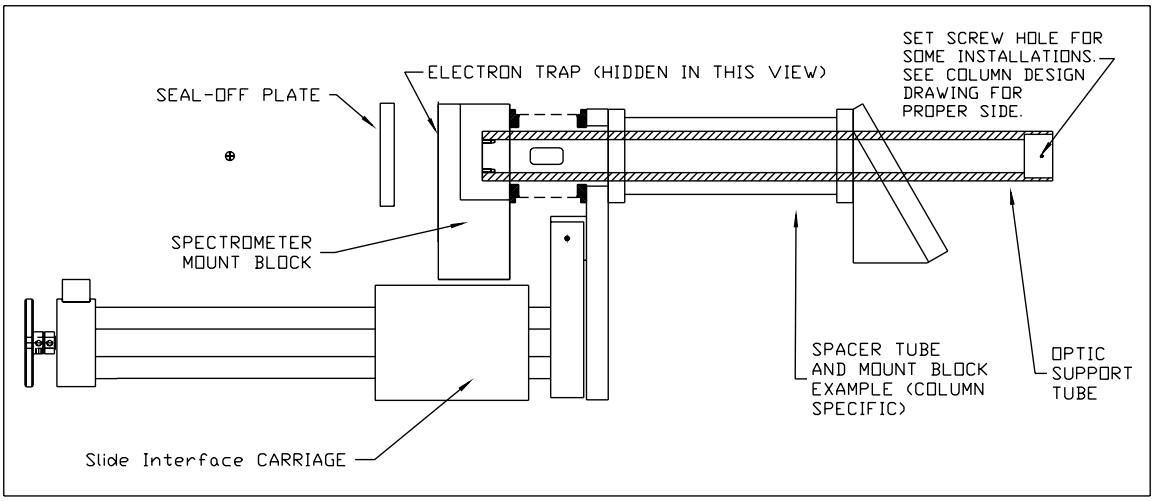


Figure 7-1: Slide Interface Example

2. Attach the **Port Adaptor** (column specific) to the **Slide Interface** as shown in the Column Design Drawings that should be supplied. Be sure to install any o-rings that may be called out.
3. Before installing the MAXray or Slide/Interface, record the system base pressure. Be certain that the pressure is representative of a typical base pressure. Vent the microscope column and remove the port cover for the designated installation port. Mount the **Slide Interface** to the port, using supplied hardware. Do not tighten the bolts.
4. Using a flashlight looking down the Support Tube, verify the tip of the pole piece is centered in the tube. If it is not centered loosen the **Port Adaptor** screws (which mount to the chamber) and shift the alignment to center the pole piece in the **Support Tube**.

   Tighten all **Port Adaptor** bolts, and verify the alignment of the pole piece in the **Support Tube**.

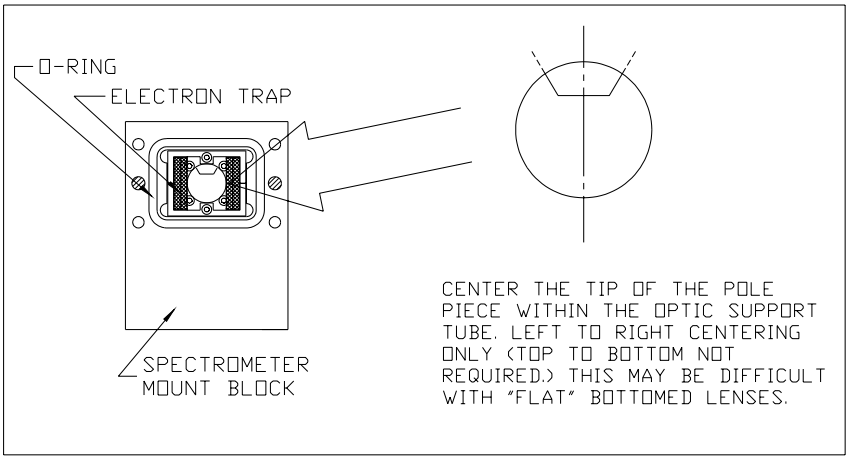


Figure 7-2: View Down Support Tube

5. Viewing the inside of the chamber from a stage door or another port, insert the **Slide Interface** until the **Support Tube** is visible and accessible for installation of the X-ray Optic. Install the **X-ray Optic** by pushing the large end into the **Support Tube**. This is designed to be a snug fit — so be careful to not cock the optic during installation. If a **Set Screw** is required, tighten it against the X-ray Optic.

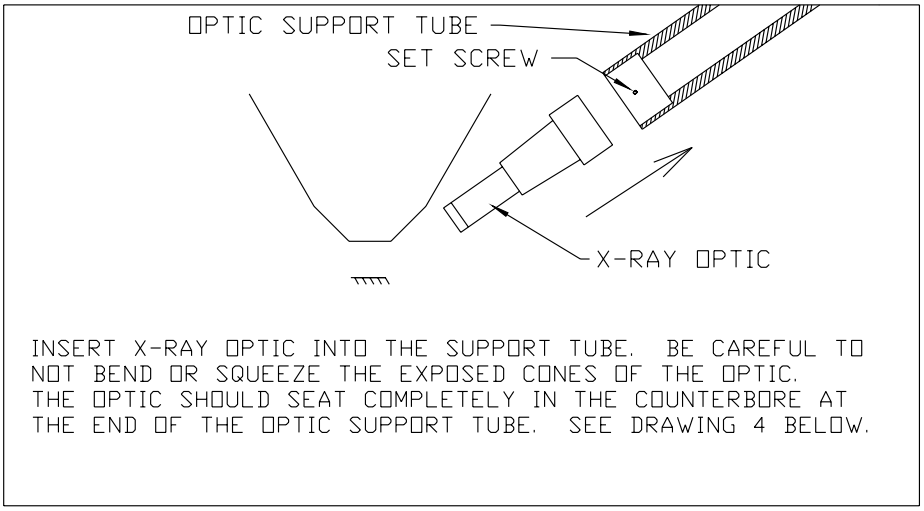


Figure 7-3: Installing X-ray Optic

6. Viewing the specimen area through the open port, insert the optic slowly into the microscope chamber and locate the end of the optic, approximately 16mm from the central axis of the column (the actual optic surfaces start 3.8mm behind front of optic assembly). Observe if the X-ray Optic will hit anything when inserted into this position (BSED, SED etc.). Referring to the Column Design Drawing note the distance from the Front Plate of the Slide Interface to the linear rail carriage. Use a scale to adjust the position of the **Carriage** to the distance in the drawing. With the optic in position, verify that the **X-ray Optic** is not hitting any structures within the chamber. Tighten the split ring **Stop** on the **Leadscrew** at the position of the **Carriage**.

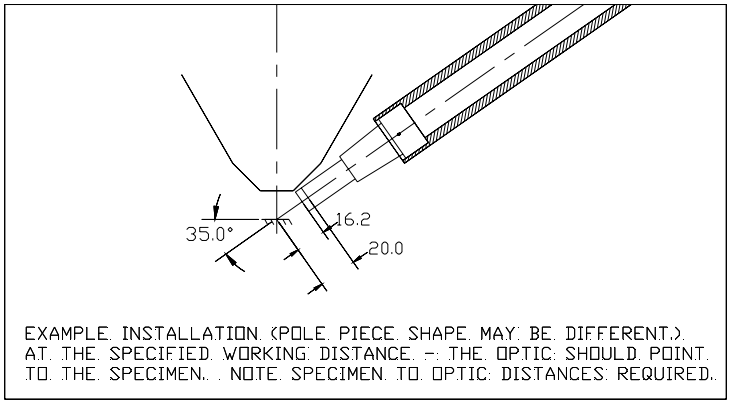


Figure 7-4: Installing X-ray Optic

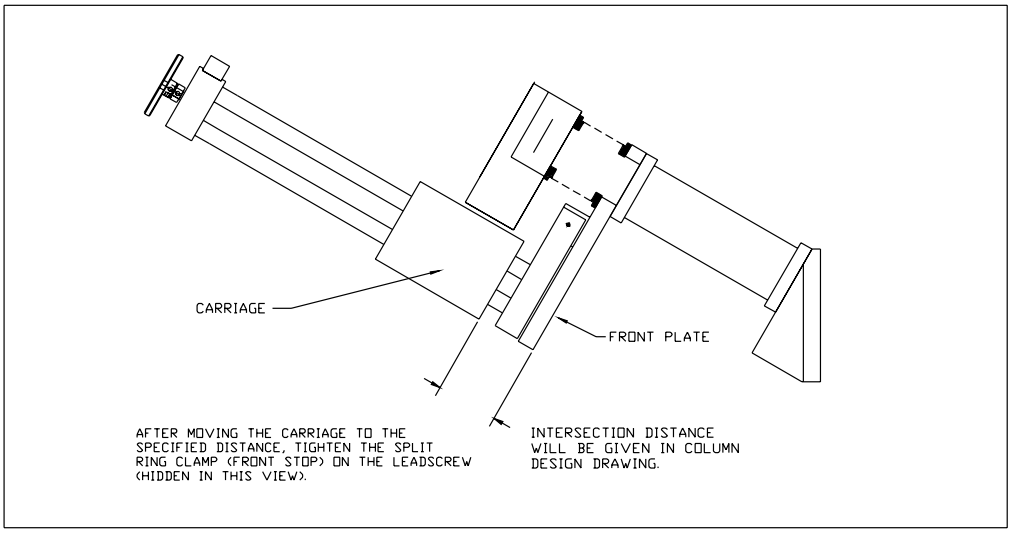


Figure 7-5: Initial Setting of the Front Stop

7. Install the **Seal-Off Plate** on rear of the **Slide Interface** using 4 M5 SHCS provided (See *Figure 7-1*).

   Close all other chamber ports and begin evacuating the chamber. This step is included to verify that there are no vacuum problems with the interface assembly prior to attaching the MAXray spectrometer. While the system is pumping down, unpack the MAXray Spectrometer, controller, and other peripheral equipment to prepare for installation. If the system vacuum integrity is not compromised, proceed with installing the MAXray spectrometer. Vacuum integrity can be verified by observing the pump-down curve. Even if the system base pressure is not obtained quickly, as long as the pressure continues down over time there probably are no leaks. If the chamber pressure hangs up at a value significantly higher than the typical base pressure, probably there is a leak somewhere. Check all o-ring seals, but remember that the Slide Interface must remain oriented to the pole piece as determined in step 4.

8. With the Spectrometer, Controller, and Host Computer unpacked, complete the system hook-up so that motor functioning can be verified prior to installing the spectrometer. See the MAXray System Diagram at the end of this Chapter for all wiring and gas requirements. Place the MAXray Spectrometer on a table for testing. Complete the following hook-ups for test:

   - Detector, Theta, and Turret motor cables from **Controller** to **Spectrometer**.
   - **Controller** power cable.
   - **COM1** Serial Cable from **Host Computer** to **Controller**.
   - **Host Computer** keyboard, VGA display and power cable.

     DO NOT HOOK UP PCS CABLE NOW.

     DO NOT HOOK UP GAS FLOW NOW.

DO NOT HOOK UP VACUUM INTERLOCK NOW.

9. Turn on power to the Controller, then insert Floppy Disk with controller software.

   Turn on power to the Host Computer and allow time to boot. Once the system has booted, start the MAXray software by clicking on the MAXray icon. The program will read configuration data from the Controller and then will accept user commands.

   See *Troubleshooting on page 7-23*.

   Open the WDS Software by clicking on the MAXray Icon (Windows NT). From the **Service** menu, click **Password**, and then type **bestWDS** to access service options.

   From the **Service** menu, click **Monitor COM Port.** Open the command dialog box by clicking **Send Command** (from the Service menu).

   Drag the **Send Command** window to a convenient location, and then type the command show in the dialog box.

   The controller should send back configuration data (which is stored in the **CRYSTAL.TXT** file on the controller floppy disk) and display the information in the **Monitor COM Port** window. There will also be some calibration offsets listed below the configuration data, this data is stored in the **CALIBRAT.TXT** file (on the controller floppy disk). This verifies that communication is working between the host and controller. If any error messages are received see Troubleshooting in this document.

   From the **Service** menu, click **Initialize Motors.** The Initialize Motors verification displays. Click **OK.**

   Three motors home in this sequence. At the high speeds used for homing the motors will generate some acoustic noise which is normal. If the motors fail to home see Troubleshooting.

10. Vent the chamber and remove **Seal-off Plate** from rear of **Slide Interface**.

    Retract the slide mechanism about 4 inches — or any necessary distance to easily access the screws to mount the spectrometer. Remove the **Cover Plate** from the front of the **Spectrometer** housing. Turn off power to the **Controller** and disconnect the three **Motor Cables**.

11. Position the MAXray onto the Slide/Interface, aligning the pins on the interface to the holes on the spectrometer body. Insert and tighten the **M5 Screws** (4) that secure the spectrometer to the interface. Be sure the **O-ring** surface is clean and seated prior to installation of the Spectrometer.

    NOTE: Some versions of the MAXray do not have any PRESSURE RELIEF VALVES integrated on the interface. Therefore prior to installation of the spectrometer be sure that the AIR ADMIT valve is closed. Also prior to venting the CHAMBER with the spectrometer installed, an AUXILIARY PORT, or the STAGE DOOR must be loosened to provide pressure relief during venting. FAILURE TO PROVIDE ADEQUATE PRESSURE RELIEF WILL RESULT IN DAMAGE TO THE GAS FLOW COUNTER WINDOW.

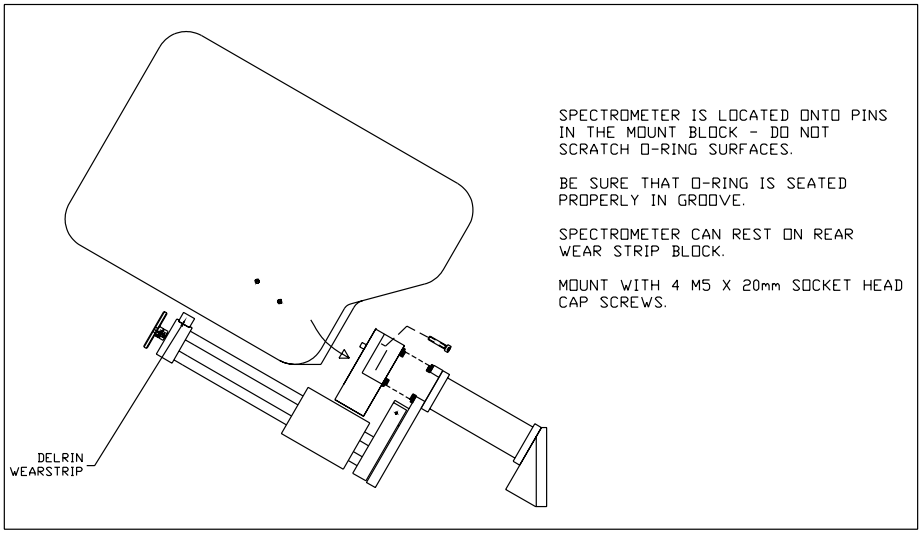


Figure 7-6: Mounting Spectrometer to Slide Interface

12. Evacuate the microscope chamber.

    Immediately go to the **Spectrometer Gas Inlet Ports** and determine if any suction can be felt by covering both ports with a finger. If the ports are sucking air, the Gas Flow Counter Window may be damaged. See the Troubleshooting section of this manual.

    Note the pump down curve (i.e., log the system pressure over time.)

Time	Pressure	Comment

    The system should reach the 1E-5 mbar range within an hour. The MAXray Spectrometer shares vacuum with the microscope chamber, and thus provides additional gas load on the vacuum system. Some degradation in base pressure is expected. Depending on the size of the chamber and the pumps utilized this degradation can vary from 1/2 to 1 order of magnitude in total pressure.

If the pressure is decreasing over time, then proceed to attach the P-10 gas supply using the Gas Kit provided.

13. Hook up the P-10 Gas Kit as follows (see MAXray System Diagram at the end of this chapter).

    Attach the single stage regulator supplied to the **Customers Two Stage** regulator already in place on the **P-10 Gas Cylinder**. Use **Teflon** tape on all metal thread seals.

    Use the 1/4” diameter **Black Tubing** to connect the **Gas Supply** to the **Flow Meter**. Note the use of **Quick Disconnects** similar to the **Water Supply Lines** on a **Freedom** Detector.

    Set the 2nd Stage Regulator to approximately 10 PSI, and the 3rd Stage Regulator (the one NORAN supplied) to 3-4 PSI. Set the **Flow Meter** to 40 cc/min. for initial flushing.

    Use the 1/8” diameter **Clear Tygon Tubing** to connect the **Flow Meter** to the **Spectrometer Gas Port**. Either port can serve as the supply side.

    The **Flow Meter** should be located at a position where the user can verify gas flow and note when the supply has been depleted. The **Flow Meter** should be leveled with the **Circular** level mounted to the stand.

    Note: After establishing gas flow, verify that the chamber vacuum has not degraded. On FESEMs the INTERMEDIATE pressure should show minimal effect.

14. To adequately flush the **Gas Flow Counter**, P-10 gas must flow for approximately 4 hours. An overnight flush is preferred for setting Bias and SCA voltages, but 4 hours should be acceptable for beginning the alignment procedures. See page 1-17.

15. Connect the **Spectrometer Motor Cables**, **PCS Power Cable** (9-pin), and **Vacuum Interlock**. (See separate documentation for interface to specific microscope.) Apply power to the **Controller** and allow the system to boot (1 minute).

    Initialize the motors from the **Host Computer** to verify that everything is still working.

    Wait the specified 4 hours or overnight.

Alignment

Once the system has pumped down to user requirements, and the flow counter has been flushed, proceed with powering on the spectrometer (controller and host computer) if not already on.

The most critical aspect of aligning the MAXray spectrometer is the ability to reproducibly locate a specimen at the exact same working distance. Even a standard block in a 1" cup can be tilted slightly — providing some variation in specimen height. By some means, once the alignment is started at a particular Working Distance (WD), the top of all other specimens must be located at the same point in space.

Hook up the oscilloscope to the **AMP Out BNC** on the **Spectrometer** body, and set the time base to 1usec and sensitivity to 1V/div. Offset the channel DC level so that a full 0 to 5V waveform can be observed.

Standards will be provided for alignment and performance validation. It is not yet determined if the standard will remain with the customer or be returned to NORAN. The standard provided must be used for any sensitivity (CPS/nA) validation.

1. Start the WDS software by clicking on the MAXray Icon (Windows NT).
2. From the **Services** menu, click **Password**, and type **bestWDS**.

   From the **Services** menu, select **Monitor COM Port**, and open the command dialog box by selecting **Send Command**.

   Move the **Send Command** window to a convenient location, and type the command show in the dialog box. The controller should send back configuration data (which is stored in the **CRYSTAL.TXT** file on the controller floppy disk) and display the information in the **Monitor COM Port** window. There will also be some calibration offsets listed below the configuration data, this data is stored in the **CALIBRAT.TXT** file, also on the controller floppy disk.
3. From the **Service** menu, click **Initialize Motors**. Verify by clicking **OK**.

   Three motors home in this sequence. If any problems are encountered see the Troubleshooting section.
4. Start with a Carbon Standard Specimen (provided). Use 10kV accelerating voltage or the highest available setting for the system. Adjust the probe current to 5nA or system maximum (whichever is less). The magnification should be at 5000X or greater. Be sure that the column is aligned and the beam current as steady as possible (after flashing FE source). Once the carbon specimen is located and in focus, the position in space of the specimen must be reproducible for all other specimens. “In focus” is defined as a reproducible working distance and focus lense settings.
5. Insert the **Spectrometer** to the previously set **Stop** (step 6 during Installation). Set the spectrometer to nominal values of the Bragg angle for carbon, by opening the **Settings** menu, and selecting **Position Spectrometer**. Choose “C” from the **Periodic Table** and note that commands are being sent to the controller (displayed in the **Monitor COM Port** window.)

6. Once the motors have stopped moving, pulse waveforms should be visible on the oscilloscope (check triggering). If the waveforms are very low amplitide (LT. 1V) or if the pulses exceed 5V and clipping is observed, then the Gas Flow Proportional Counter BIAS needs to be adjusted.

   Change the Bias voltage. From the **Service** menu, click **Electronics**.

   Change the bias, baseline, and window voltage settings. Change the bias value to obtain pulses which utilize the full dynamic range of the PCS Amplifier. See Figure 6-7, and verify the values of the SCA baseline and window.

   If no pulses are visible see Troubleshooting.

   If you do not have an oscilloscope see Bias Scan and SCA Scan.

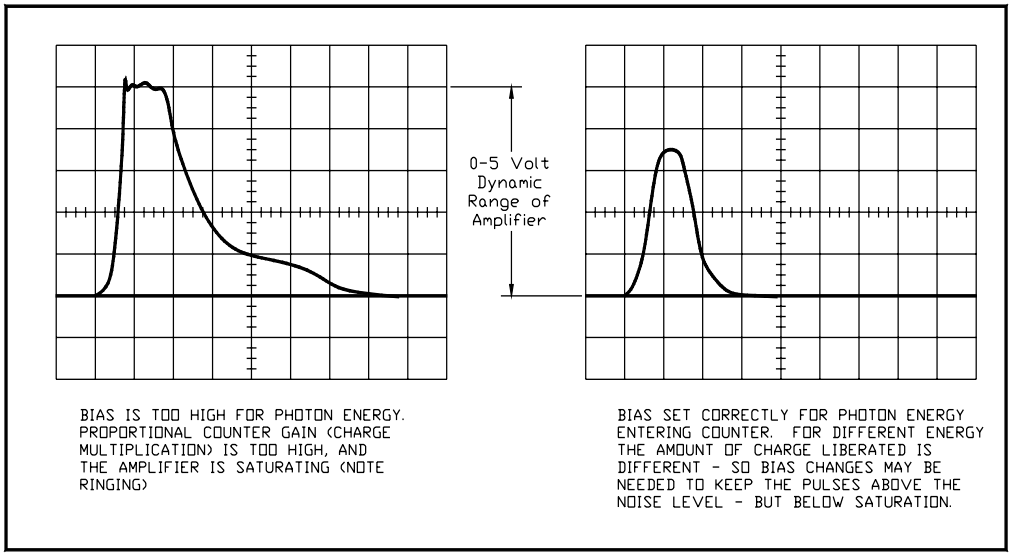


Figure 7-7: PCS Pulse Waveforms

7. From the **Acquire** menu, click **Element Scan**, and then choose "**C**" from the periodic table to scan the spectrometer over a range of expected peak intensity positions for Carbon.

   Once a peak is observed set the spectrometer to the energy value by typing: **energy equals.XXX**, in the Send Command Window. Once the spectrometer is set at the peak position open the **Count** menu and select **Ratemeter** to open a ratemeter window. Alternatively use the **Analog Ratemeter** hooked-up to the "PD" BNC on the Spectrometer PCS Electronics.

   The objective is to now align the Spectrometer and X-ray Optic to obtain maximum count rate at carbon. There are many degrees of freedom in performing this alignment:

   - Spectrometer Tilt (points optic to established working distance)
   - Spectrometer Horizontal (lateral position optic relative to specimen)
   - Spectrometer Intersection (focus of optic to specimen position)

- Diffractor Tilt (position of Turret relative to collimated beam)
- Diffractor Angle (must be at the Bragg angle for maximum intensity)
- Detector Angle (relative to the diffracted beam)

Additionally, the impact of each of these alignments is energy dependent. There are some interaction effects which necessitates iteration between all adjustments.

- AC. Spectrometer Tilt and Spectrometer Intersection.
- CD. Diffractor Angle and Spectrometer Intersection (especially at high energy).

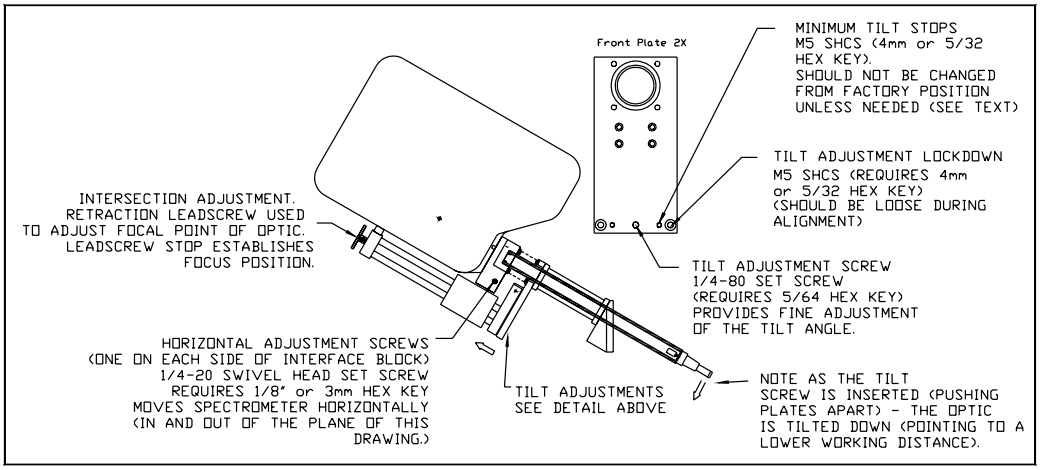


Figure 7-8: Spectrometer Alignment Adjustments

Alignment is started at carbon Ka, because the x-ray optic has the largest acceptance angle for low energies, and the effects of misalignment are less pronounced. After alignment at CKa, the spectrometer is aligned to maximize count rate for AlKa, which is more difficult, and requires more iteration to achieve proper intensity and resolution. The alignment at AlKa will not adversely affect the alignment at CKa unless the optic is defective.

8. Adjust the **Horizontal Adjustment** screws which move the spectrometer in a horizontal plane (inclined, relative to beam axis).

   Turn both screws simultaneously, loosening one and tightening the other (on either side of the interface block). You should observe a fall off in total count rate on either side of the peak position for the horizontal axis.

NOTE: If the ratemeter does not appear to respond to changes even though you see higher repetition rates on the oscope, the SCA settings may need to be adjusted.

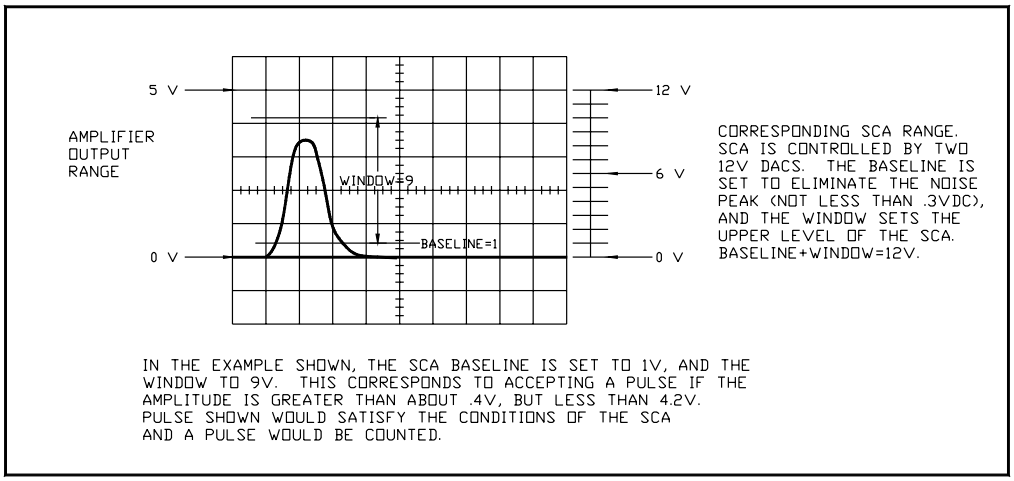


Figure 7-9: SCA Settings from Oscilloscope

9. Loosen the **Tilt Adjustment Lockdown** screws to allow the movement of the spectrometer tilt axis. Use the **Tilt Adjustment Screw** to change the tilt of the spectrometer to maximize countrate. DO NOT change the **Minimum Tilt Stops** as these should prevent the optic from hitting the pole piece. If the count rate only drops as the spectrometer is tilted, retract the X-ray optic (Intersection adjustment) about 2-3 mm and loosen the Minimum Tilt stops by 1/4 turn, and then try to maximize counts using the **Tilt Adjustment Screw**.
10. You may need to iterate between the All Three Adjustment Axis a few times, being sure you are at the true peak for the element being analyzed. (Repeat scans over the peak and then set the spectrometer to the energy (angle) for maximum intensity). Do not be concerned if the peak does not fall at the correct energy. Calibration offsets will be added later to correct the peak position relative to the energy axis. Also, the port adaptor and flange are designed to position the spectrometer such that minimum adjustment is required. Avoid severe changes to the nominal position.

    If drastic changes are needed, something may not be assembled correctly.
11. Once you obtain expected intensities for carbon (3000 cps/nA at 5kV-6000 cps/nA at 10kV), move to the aluminum specimen on the standards block. Be sure to have the specimen at the exact same working distance as carbon. Use the **Set Spectrometer** function to position the spectrometer for aluminum. This will automatically change diffractor and reset electronics to pre-established defaults.

    Perform an **Element Scan** to locate the peak intensity energy channel and use the **Send Command** window to position the spectrometer at that energy. Again observe the oscilloscope for the pulse amplitudes.

12. Repeat the **Horizontal** and **Tilt Adjustments** to maximize count rate. The aluminum (and other high energy lines) count rate can be influenced greatly by the specimen to optic distance — the Intersection Adjustment will be more critical. Be careful, if you have changed the minimum tilt stops, that the **X-ray optic** does not hit the pole piece during any change to the **Intersection Adjustment**.

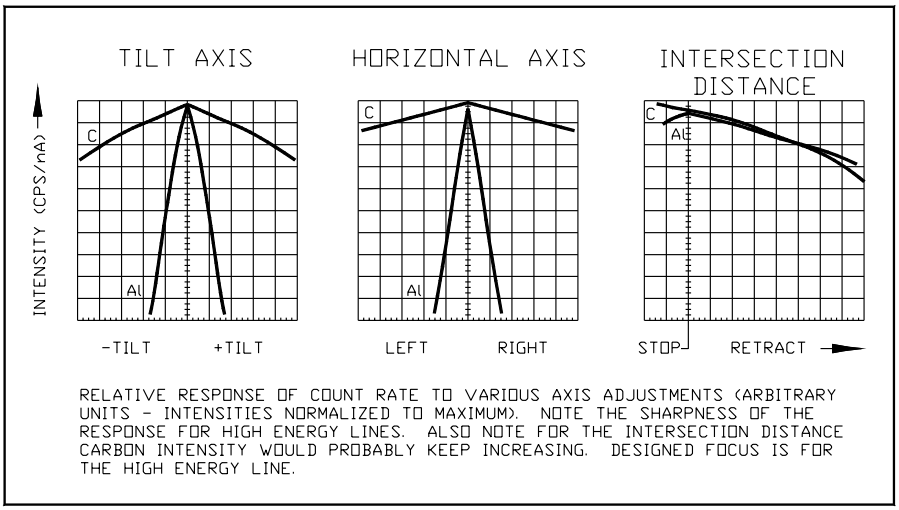


Figure 7-10: Relative Intensity Response

13. The aluminum adjustment is very sensitive (see *Figure 7-10*), and can be difficult. Attempt to obtain the best possible count rate, as this will assure alignment for all elements of interest. Again, be absolutely sure the spectrometer set at the peak intensity energy by scanning over the peak. If the peak is split (two peaks) or severely distorted, set the spectrometer to the energy at the midpoint between the peaks, and re-adjust all axis of the spectrometer for maximum counts.
14. After adjusting for maximum aluminum intensity, check alignment again at the silicon K line. Minor adjustment of the **Tilt** and **Intersection** is all that should be required. The same spectral distortions can occur at silicon, and these should be eliminated or minimized. The use of TAP for silicon is stretching the limits for the crystal, so some peak distortion is expected.
15. Once silicon intensity and resolution specifications are obtained, the adjustment axis must be locked down. The spectrometer is positioned at the SiKa peak energy (maximum counts), and the **Ratemeter** is observed while tightening the lock-down screws. The **Average Maximum CPS** must be maintained during the lock-down. Tighten **Tilt Adjustment Lockdown** screws to prohibit changing tilt. It may be necessary to alternate between **Lockdown** and **Minimum Tilt** screws (which counteract each other) to keep the count rate maximized. If the **Intersection Distance** was changed from the install position, re-position the **Stop** and tighten. The **Horizontal** adjustments should be tight already — if the opposing screws were moved simultaneously. Do not overtighten the lock-down screws. They should only be tightened enough to prevent changes in the aligned position.
16. The spectrometer should be retracted and inserted to verify that the aligned position is maintained after moving.

Once the spectrometer is aligned to the microscope, the calibration offsets for each crystal must be determined. See MAXray calibration data.

PLEASE DOCUMENT PROBLEMS IN WRITING.

Crystal Installation

Creating the IbeX/MAXray Crystal Definition File

When the IbeX / MAXray controller software starts up, it reads the CRYSTAL.TXT file on the controller floppy disk.

This file defines the configuration of the spectrometer. It can be edited with any ASCII editor such as DOS Edit.

Below is an example and a description of the individual entries in the file.

The underlined items are the ones to edit. Never use more than ONE SPACE to separate items.

ECS Type B	ECS TYPE A for older systems, ECS TYPE B for newer systems.
LEFT HAND	LEFT HAND or RIGHT HAND spectrometer.
6 DIFFRACTORS	(1, 2, 3, 4, 5 or 6) the number of crystals on the turret.
POSITION 0	The first turret position (see Figure 7-1).
MoB4C145-B	The name of this crystal.
SPACING 145.0	The 2d spacing of this crystal in Angstroms.
HOME ANGLE 240.0	The angular position of this crystal on the turret see diagram.
MIN keV 0.121	The minimum energy in the range of this crystal.
MAX keV 0.395	The maximum energy in the range of this crystal.
POSITION 1	The second turret position.

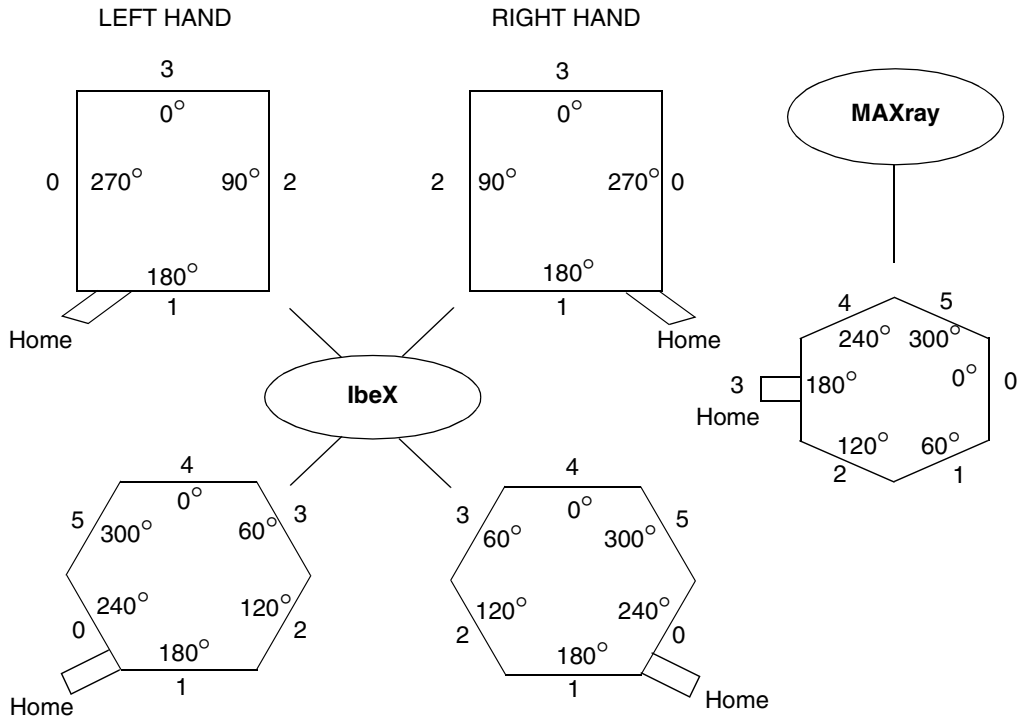


Figure 7-11: Figure 7-1: Turret Positions

Mounting the Crystals On the Turret

The crystals MUST be mounted on the turret in a special order. The crystal with the largest 2d spacing goes in POSITION 0 (see Figure 7-1). The crystal with the next largest 2d spacing goes in POSITION 1. On the IbeX, if two or more crystals have the same 2d spacing, the crystal that is curved for the lower atomic number element is mounted before the others, then the crystal curved for the next highest atomic number is mounted.

Calculating the Minimum and Maximum keV

These values depend on the 2d spacing of the crystal and the angular range of the spectrometer.

IbeX	MIN keV = 12.398 / (2d x 0.7071) [IbeX MAX angle = 45°] $\sin\theta$ =sin 45=0.7071
IbeX	MAX keV = 12.398 / (2d x 0.2164) IbeX MAX angle = 12.5°
MAXray	MIN keV = 12.398 / (2d x 0.9385) MAX angle = 68.5° sin63.5 = 0.9304
MAXray	MAX keV = 12.398 / (2d x 0.2419) MIN angle = 14° sin 14° = 0.2419

For example:

If the 2d spacing of the crystal is 145.0 Angstroms:

IbeX MIN keV = 12.398 / (145.0 x 0.7071) = 12.398 / (102.529) = 0.121

IbeX MAX keV = 12.398 / (145.0 x 0.2164) = 12.398 / (31.378) = 0.395

	IbeX MIN KeV	IbeX MAX KeV	MAXray MIN KeV	MAXray MAX KeV	Default 2d Space Turret Position		Calibration Sample
LiF(220)	6.156	20.117	4.679	17.996			
LiF(200)	4.354	14.227	3.309 1	2.727			
PET	2.006	6.554	1.524	5.863			
TAP	0.681	2.225	0.517	1.990	25.75	5	AL/SC
WSi60	0.292	0.955	0.222	0.854	60	4	O_2
NiC80	0.219	0.716	0.167	0.641	80	3	N
NiC100	0.175	0.573	0.133	0.513	100	2	C
MoB4C145	0.121	0.395	0.0919	0.353	145	1	B
MoB4C200	0.0877	0.286	0.0666	0.256	200	0	Be

Crystal Names

The crystal names can be a combination of the crystalline material, the 2d spacing, and the element for which they were curved. If the crystal is made from a natural material (LiF, PET, TAP), the 2d spacing is omitted from the name. If the crystal is made from synthetic material (MoB4C, NiC, WSi, CrSc), the 2d spacing is included in the name. If the crystal is curved for a specific element (always on the IbeX, never on the MAXray), the atomic symbol is added at the end of the name preceded by a dash. No distinction is made between K, L or M lines.

For an IbeX MoB4C crystal with a 2d spacing of 145.0 Angstroms that is curved for Boron, the name is: MoB4C145-B

The same crystal used on a MAXray will have the name: MoB4C145

Special Case: LiF

A Lithium Fluoride (LiF) crystal can come in more than one type;
LiF(200) with a 2d spacing of 4.027 Angstroms, and
LiF(220) with a 2d spacing of 2.848 Angstroms.

A LiF crystal of the 200 variety with a 2d spacing of 4.027 Angstroms and curved for Iron has the name: LiF(200)-Fe

A LiF crystal of the 220 variety with a 2d spacing of 2.848 Angstroms and curved for Strontium has the name: LiF(220)-Sr

Notes

- These instructions refer to the IbeX controller software version 2.01 and higher.
- These instruction refer to all versions of the MAXray controller software.
- Most IbeX spectrometers are LEFT Handed.
- All MAXray spectrometers are LEFT handed.
- The crystal 2d spacing can be found on the crystal storage box.
- For the PeakCAP software only, the crystal names in this file must match those in the PeakCAP database.
- Be careful not to type a zero instead of an O for Oxygen.
- A dash is the same character as a minus sign.
- Please note that the MAXray turret positions begin with ONE, not ZERO.

Example of a CRYSTAL.TXT File for a Six-Crystal MAXray Spectrometer

This text file is created automatically using the save calibration data from the host PC.

BEAM METER YES
ECS TYPE B
LEFT HAND
6 DIFFRACTORS

POSITION 0
MoB4C145-B
SPACING 145
HOME ANGLE 240

POSITION 1
NiC100-C
SPACING 100
HOME ANGLE 180

POSITION 2
WSi60-N
SPACING 59.8
HOME ANGLE 120

POSITION 3
WSi60-O
SPACING 59.8
HOME ANGLE 60

POSITION 4
TAP-Al
SPACING 25.75
HOME ANGLE 0

POSITION 5
PET-Mo
SPACING 8.742
HOME ANGLE 300

MAXray Calibration Procedure

Turret Scan

Each of the six diffractors on the turret are mechanically aligned as closely as possible after installation. Because there are small offsets that cannot be corrected for using screws, the turret must be rotated over a small angular range while intensities are measured at discrete positions. If the spectrometer is centered as closely as possible to an x-ray peak, this turret scan generates a peak of intensities and computes the center of the peak. The offsets should be small (less than 5 degrees) and very close to each other (less than 3 degrees). If these differences are much larger, there is a problem. The home position of each diffractor is stored on the controller floppy disk in the file named CRYSTAL.TXT. (refer to page 7-1 through 7-4 to edit this file.) You will need to edit some values in this file for the turret calibration. This procedure should be performed AFTER the initial spectrometer alignment on carbon and BEFORE the final spectrometer alignment on Silicon.

Begin the procedure by placing the carbon sample in the beam and use the Acquire/ Element Scan function perform a scan over the carbon K alpha peak. Although it is supposed to be at 0.282 keV, the spectrometer has not yet been aligned and the peak may be several eV away from this energy in either direction. When the scan is complete and the peak has been located, use the energy equals command from the Send Command window to move the spectrometer to the energy of the peak center which is the energy with the maximum number of counts. When the spectrometer has stopped, use the Service/Turret Scan function to perform the turret scan. When the scan is complete a peak centroid will be computed and displayed over the turret scan plot. Apply this initial turret offset value to ALL diffractors by editing the HOME entry in the CRYSTAL.TXT file. After the Carbon alignment has been completed, perform this turret scan for one x-ray line using each of the six diffractors. Use the table below to record your measurements. When complete, edit the CRYSTAL.TXT file using the Windows/ Notepad program so that the crystal home positions match the angles found during the calibration. It is necessary to reboot the controller after editing this file. Retest the carbon sample again to check the success of the turret calibration.

	Diffractor Name	**Original Home Angle**	**Calibrated Home Angle**
1			
2			
3			
4			
5			
6			

Peak Offsets

In the next calibration step, angular offsets will be added to the controller file named CALIBRAT.TXT so it is important that the controller has been booted from a floppy disk that contains no CALIBRAT.TXT file. Look for this disk using Windows/Notepad. This procedure should be performed AFTER the spectrometer alignment.

Starting again with carbon, scan over the K-alpha peak, find the peak position using View/Peak Information, and record this peak position. If the peak is not at the correct energy (0.282 keV) and angular offset must be added to shift the peak. The offset for the crystal motor and the detector motor should be the same. An approximate offset can be calculated, or an offset tried and then tested. Below is an example calculation for a Carbon peak calibration using the Bragg equation to find the difference between the two peak positions in degrees. The supplied program WdsCalc should be used to calculate the angels based on the energy offsets.

Since $\lambda = 2d\sin\theta$

Re-arrange $\theta = a\sin(\lambda/(2d))$

Where $\theta =$ The angle in degrees

$\lambda =$ The wavelength in Angstroms

$2d =$ The spacing of the diffractor in Angstroms

Example: The crystal used was a NiC100 that has a 2d spacing of 100 Angstroms.

1. Peak position from the initial scan over carbon K-alpha = 0.262 keV
2. Wavelength = (12.398 / 0.262) =47.3206 Angstroms
3. Angle = Asin(47.3206 / 100) =28.2426 degrees
4. Correct peak position of the carbon peak =0.282 keV
5. Wavelength = (12.398 / 0.282) =43.9645 Angstroms
6. Angle = asin(43.9645 / 100) =26.0812 degrees
7. The angular offset = 28.2426 – 26.0812 = 2.1614 degrees

Because the starting peak position was lower in energy than the final peak position, the offsets must be ADDED.

Use the CRYSTAL x POINT SET command in the Service/Send Command window to set the new offsets and scan over the Carbon K-alpha peak again.

If the NiC100 diffractor is in the THIRD position on the turret, the command would be:

```
crystal 2 point set 26.0812 - 2.1624 - 2.1624
```

If the peak is within 1.0 eV (0.001 keV) of the correct position, the calibration for this crystal is complete. The controller software recognizes the diffractors arranged on the turret in positions Zero through Five. In the Settings / Configuration window the diffractors are listed in the order One through Six. Use the save calibration data command to write the offsets to disk.

Detector Bias Voltage, SCA Baseline, and Window Voltages

It is important to determine the correct detector bias voltage, and the correct SCA baseline and window voltages for each x-ray line on the flow proportional counter. The controller shipped from the factory may not have the correct settings installed on the controller diskette. These voltages should be verified using the Settings/SCA Scan function. The bias should be high enough to pull the signal peak out of the noise and center it at between 3.0 to 3.5 volts. The SCA baseline voltage should be set at the valley between the noise peak and the signal peak and the SCA window voltage should be set to be as wide as the signal peak. There are entries in the window program's initialization file that can be used to set these voltages based on low and high energy measurements and the slope and intercept of their associated plots.

Calibration Tips

1. Delete any CALIBRAT.TXT file from the controller disk before starting.
2. Remove the calibration offsets for any crystal before beginning, and remember to replace them when complete.
3. If re-calibrating an element on a diffractor that has already been calibrated, first set the crystal and detector offsets to zero for this diffractor.
4. If the calculated angular offset does not work, use small offsets to find the closest.10
5. The offsets for one diffractor should be similar the all others and should be less then ±3.0 degrees.
6. If the calibration is more than 3.0 degrees off, verify that the correct sample is being used.

List of Commands

These commands can be sent using the Service/Send Command window.

1. Set a calibration offset point.

   `crystal X point set aa.aaaa  b.bbbb  c.cccc`

   where: X is the turret position of the crystal (0 through 5)

   aa.aaaa is the Bragg angle of the peak

   b.bbbb is the crystal motor offset

   c.cccc is the detector motor offset

   NOTE: Always use four digits after the decimal point.

2. Remove a calibration offset point.

   ```
   crystal X point remove aa.aaaa
   ```

   where: X is the turret position of the crystal (0 through 5)

   aa.aaaa is the Bragg angle of the peak

   NOTE: Always use four digits after the decimal point

3. Save the calibration offsets to the controller floppy disk.

   ```
   save calibration data
   ```

4. Display the Turret and Calibration information in the Monitor window.

   ```
   show
   ```

5. List the system status in the Monitor window.

   ```
   status
   ```

6. Move the spectrometer to an energy position.

   ```
   energy = aa.xxx
   ```

where: aa.aaa is the desired energy in keV

NOTE: The energy selected must be in the range of the active crystal.

Troubleshooting

This guide is intended to outline all potential problems which may be encountered in the field. Some of the root causes of failure should not occur in the field (like feed through wiring error), however all possible sources of failure are listed for completeness.

Motor(s) Fails to Move When Commanded to Home

1. Feed through wiring error.
2. Internal motor harness error or damage. See Wiring and Plumbing drawing.
3. Motor cables not hooked up correctly. See the MAXray System diagram.
4. Controller power supply problems.
5. Microstep motor board problems.
6. Bad Motor. Replace motor. See Motor Replacement Instructions drawing.

Motors Move, But Fail to Home as Specified

1. Motor cables not hooked up properly. See the Alignment Flow Chart.
2. Motor binds prior to reaching lower limit. Call Factory
3. Bad lower limit or home sensor (microswitch or optical). Replace Sensor. See the Wiring and Plumbing drawing.
4. Damaged wiring to sensors. See the Wiring and Plumbing drawing.
5. Microstep motor board problems.

Motors Home as Specified, but Error Occurs at Extreme Positions

1. Mechanism reaches limit sensor prior to reaching specified angle.

Turret Home Angle is Not 90 Degrees to the Baseplate as Specified

This could occur in the field after a change in diffractor. A precision 90 degree gauge block will be required to align the turret.

1. Loosen the **Sensor Mount Clamp Screws** and adjust the **Optical Sensor Mount** using the **Adjust Set Screws** as shown in the diagram below. This moves the sensor position relative to the **Flag** on the **Turret**, and moves the **Home** position. Caution, this adjustment is very sensitive. The Microstep motor boards seek home by looking for both the optical sensor

interrupt AND the next index pulse from the optical encoder. Therefore, with 100:1 gear reduction on the **Turret** motor, very small angular change in the **Flag** position may be all that is needed. When the proper alignment is achieved, tighten the **Clamp Screws**.

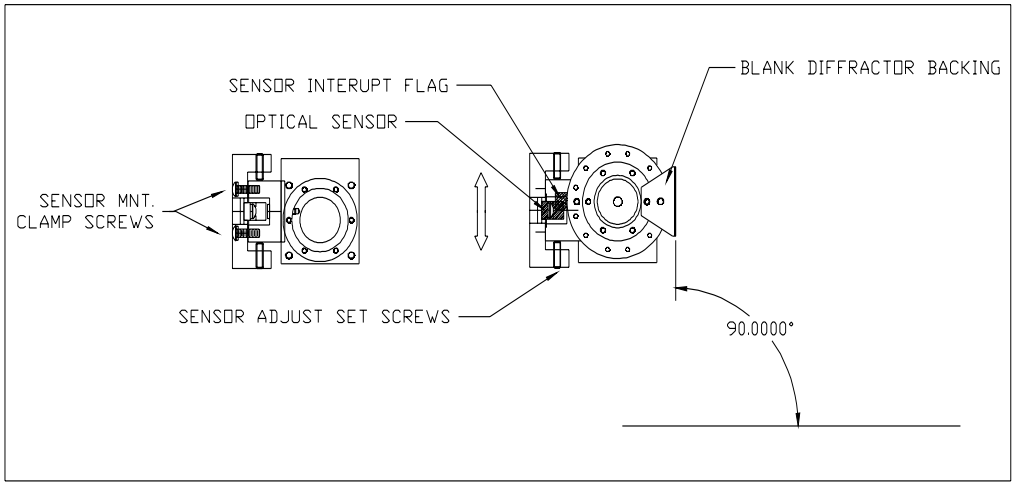


Figure 7-12: Optical Sensor Mount Adjustment

Motor Movement is Noisy

1. Excessive play in worm-gear/worm-wheel mesh. Adjust for minimal play.
2. Poor lubrication of gearing or bearings. Use **Fomblin** or **Krytox** vacuum compatible grease on all gears and bearing as required. Use sparingly, however it is found that the worm mechanisms can require a generous application to get lubricant into all teeth. Wipe away excess grease from any mechanisms.

   DO NOT LUBRICATE HARMONIC DRIVE IN TURRET MECHANISM. Consult factory if this mechanism is creating excess noise.

3. Motor is bad. Replace motor.

No Counts Detected

1. Wrong standard chosen for spectrometer setting.
2. Diffractors not labeled correctly. Check CRYSTAL. TXT File. Change to other diffractors which could detect same energy (even if not optimum).
3. No HV Bias on flow counter. Check interlock.
4. Not sufficient bias on flow counter. Use Electronics Dialog box to adjust bias.
5. SCA not set up properly. Use Electronics Dialog box to adjust SCA settings.
6. Gross misalignment. Return to factory settings. Align on carbon.
7. Gross calibration error in spectrometer. Remove **Set Points** and use wide scan to find peaks.

Poor Intensity (CPS/nA) — Not Meeting Specifications

1. Poor alignment. Re-adjust all spectrometer setting at a fixed stage position.
2. Optimize bias and SCA setting. Use o-scope to observe waveforms.
3. Bad diffractor (crystal or synthetic).
4. Turret angle is off. Perform turret scan and adjust home position in CRYSTAL.TXT file.
5. Specifications can only be checked using NORAN-supplied standard specimens.
6. For poor light elements (fluorine and lower), proportional counter window can affect intensities. Change window.
7. Electron column conditions as specified (accelerating voltage, aperture setting).
8. Intensities OK at low energy, poor at high energies. Optic is not pointing at the specimen. Realign spectrometer.

Intensities OK, Resolution, P/B Poor

1. Co-alignment of optic is bad. Check to see if optic is damaged.
2. SCA not properly set. Check baseline setting (>.2V)
3. Theta-Detector scan synchronization off - Perform Detector and Theta Scan independently and apply calibration offsets.

Detector and Diffractor Motor Calibration

Setting up the Calibration

1. Load the sample into the microscope.
   - Whenever possible, use a pure element standard.
   - For elements such as nitrogen and oxygen, use a binary compound when possible.
2. Set the microscope's Accelerating Voltage and Beam Current.
   - For all synthetic diffractors (light elements), use **10 keV** and **5 nAmps**.
   - For TAP and PET diffractors (heavy elements), use **20 keV** and **5 nAmps**.
   - For LiF diffractors, use **30 keV** and **5 nAmps**.
3. Run the MAXray software.

   Double-click on the program icon.
4. Record the crystal name.

   From the **Settings** menu, click **Configuration**.

   Determine the diffractor 2d spacing.

   Record the **diffractor 2d spacing**, the **element name**, and the **x-ray line**.

From the **Edit** menu, click **Scan Parameters**.

Read the theoretical peak energy of the x-ray line.

Record the **theoretical peak energy**, the **sample** used, the **accelerating voltage**, and the **beam current**.

5. Set the Bias, Baseline, and Window.

   From the **Edit** menu, click **Scan Parameters**. Use the following parameters.

Parameter	Heavy elements	Light elements
Bias	2250 volts	2200 volts
Baseline	1.5 volts	0.3 volts
Window	6.5 Volts	8.0 volts

   Save the settings by clicking **Save**.

6. Move the diffractor and detector to their initial positions.

   From the **Settings** menu, click **Position Spectrometer**.

   Select an element from the periodic table, and then click **OK**.

7. Unlock the service menu.

   From the **Service** menu, click **Password**. Enter the password “bestWDS.”

8. Display the controller command window.

   From the **Service** menu, click **Send Command**.

   Type **show**. No offsets should appear.

   If there are offsets, remove them with the command “crystal (n) point remove (bragg).”

   Example: If an offset exists for crystal 0 at a bragg angle of 23.1731, the command is “crystal 0 point remove 23.1731"

9. Display the COM Port Monitor window.

   From the **Service** menu, click **Monitor COM Port**.

Calibration Procedure

1. From the **Service** menu, click **Diffractor Scan**.
2. Record the scan parameters.

   Scan over a wide diffractor range around the theoretical peak energy.

   Typical scan parameters are:

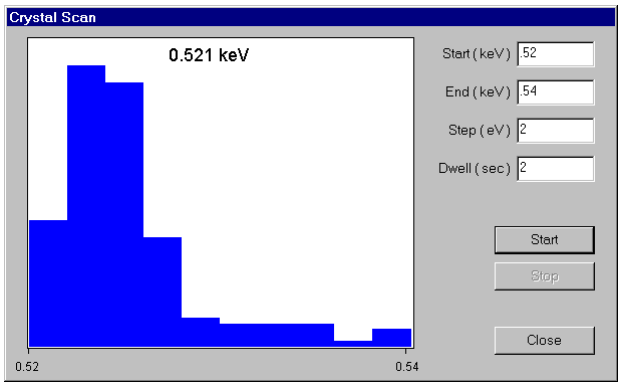
   - plus/minus 0.200 KeV (for some diffractors this range maybe too wide. Use a smaller range when necessary)

- a step of 4 eV
- a dwell of 1 second

3. Record the energy and counts in the peak.

   Watch the Monitor COM Window as the data is sent back from the controller.

   Locate the largest energy peak.



4. Record the scan parameters.

   Scan over a narrow diffractor range, around the recorded peak energy.

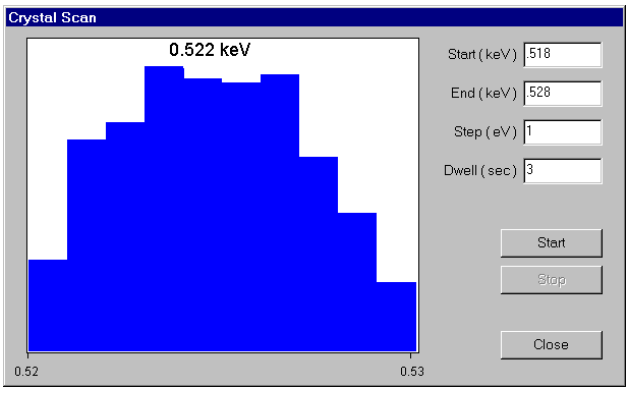
   Typical scan parameters are:

   - plus/minus 0.010 KeV
   - a step of 1 eV
   - a dwell of 1 second

5. Record this final diffractor position and the counts in the peak.

   Watch the Monitor COM Window as the data is sent back from the controller.

   Locate the largest energy peak.



6. Position the detector correctly for the diffractor scan.

Scan from 0.010 keV before and to 0.001 keV past this energy. Use a step of 1 eV and a dwell of 1 second.

Close the Crystal Scan window.

7. From the **Service** menu, click **Detector Scan**.
8. Record the scan parameters.

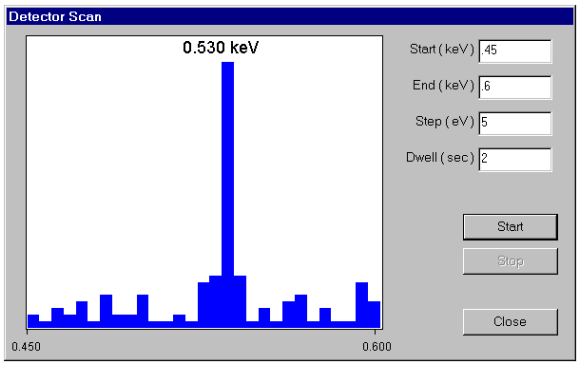
   Scan over a wide detector range around the theoretical peak energy.

   Typical scan parameters are:

   - plus/minus 0.200 keV
   - a step of 4 eV
   - a dwell of 1 second
9. Record this energy and the counts in the peak.

   Watch the Monitor COM Window as the data is sent back from the controller.

   Locate the largest energy peak.



10. Record the scan parameters.

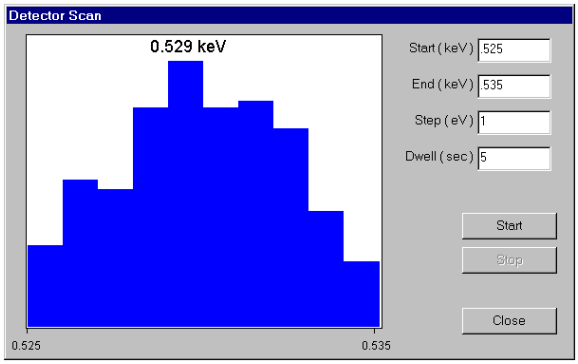
    Scan over a narrow detector range around the recorded peak energy.

    Typical scan parameters are:

    - plus/minus 0.010 keV
    - a step of 1 eV
    - a dwell of 1 second
11. Record this final detector position and the counts in the peak.

    Watch the Monitor COM Window as the data is sent back from the controller.

Locate the largest energy peak.



12. Position the detector correctly for the diffractor scan.

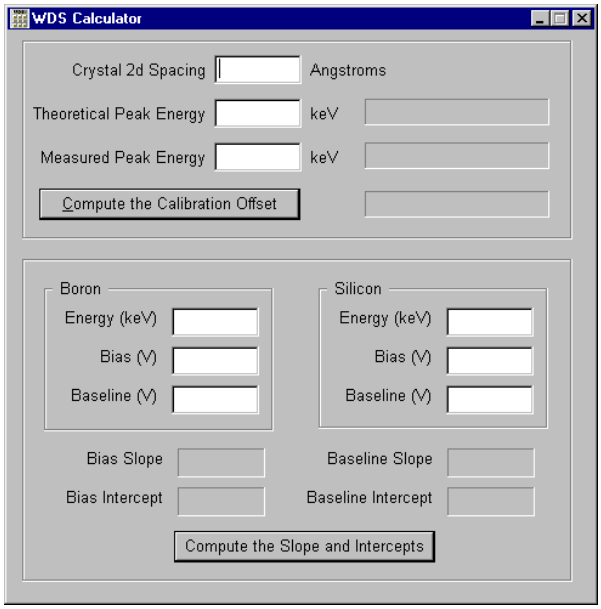
    Scan from 0.010 keV before and to 0.001 keV past this energy. Use a step of 1 eV and a dwell of 1 second.

    Close the Detector Scan window.

13. Repeat steps 1 through 12.

14. Calculate and record the crystal and detector offset.

    Run the WDS Calculator program (c:\noran\wds\bin\calc.exe).



Enter the Crystal values for Crystal 2d Spacing, Theoretical Peak Energy, and Measured Peak Energy (final diffractor). Click **Compute the Calibration Offset**, and then record the crystal offset. This value may be negative.

Enter the detector values for Crystal 2d spacing, Theoretical Peak Energy, and Measured Peak Energy. Click **Compute the Calibration Offset**, and then record the detector offset. This value may be negative.

15. Send the calibration data to the controller.

    From the **Service** menu, click **Send Command**.

    Example: CYSTAL 0 POINT SET 23.1731 0.3702-0.0881

    In this example, a diffractor offset of 0.3702 degrees and a detector offset of -0.0881 degrees are used with a bragg angle of 23.1731 degrees for the first calibration value of diffractor 0.

    NOTE: Use only one space between words and numbers.

    NOTE: Always use exactly four decimal places for bragg angles, diffractor offsets, and detector offsets.

16. Check the beam current and re-adjust it if necessary.
17. Record the scan parameters.

    Setup over a wide energy scan, around the theoretical peak energy.

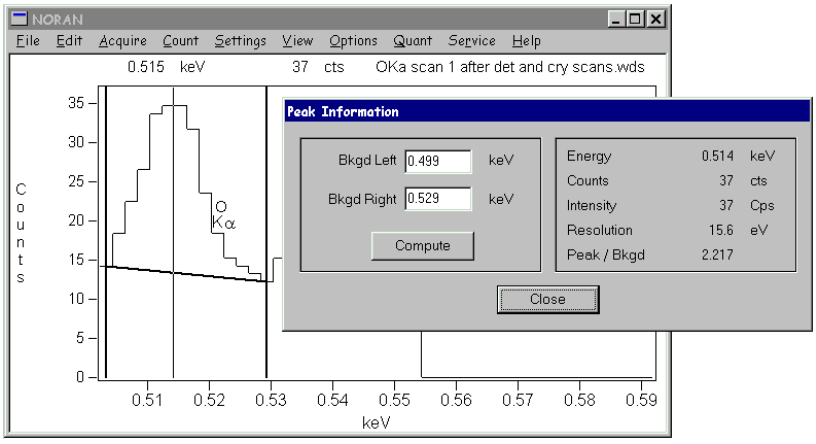
    From the **Acquire** menu, click **Scan Setup**.

    Typical scan parameters are:

    - plus/minus 0.100 keV
    - a step of 3 eV
    - a dwell of 1 second

18. Begin the scan.

    From the **Acquire** menu, click **Start Scan**.



19. Record the peak energy value.

    First, move the cursor to the peaks center.

Second, From the **View** menu, click **Peak Information**. The peak energy value displays.

20. Record the calibration offset.

    Run the WDS Calculator program (c:\noran\wds\bin\calc.exe).

    Based on the peak position enter the Crystal 2d Spacing, Theoretical Peak Energy, and Measured Peak Energy.

21. Compute and record the final crystal offset.

    Add the calibration offset to the original crystal offset.

22. Compute and record the final detector offset.

    Add the final crystal offset to the original Detector offset.

23. Remove the original offsets.

    From the **Service** menu, click **Send Command**.

    Example: CRYSTAL 0 POINT REMOVE 23.1731

    In this example, the offsets for the angle 23.1731 degrees were removed from the first crystal at position 0.

24. Set the final calibration point.

    From the **Service** menu, click **Send Command**.

    Example: CRYSTAL 0 POINT SET 23.1731 0.0513 0.1562

    In this example, a crystal offset of 0.0513 degrees and a detector offset of 0.1562 degrees will be used for an angle of 23.1731 degrees for the first crystal at position 0.

    NOTE: Use only one space between words and numbers.

    NOTE: Always use four decimal places; no more; no less.

25. Write the calibration data to the controller disk.

    From the **Service** menu, click **Send Command**.

    Example: SAVE CALIBRATION DATA

    Note: Make sure the floppy disk is inserted into the controller and not write protected.

26. Record these scan parameters.

    Setup a narrow energy scan, around the theoretical peak energy.

    From the **Acquire** menu, click **Scan Setup**.

Typical scan parameters are:

- plus/minus 0.025 keV
- a step of 1 eV
- a dwell of 1 second

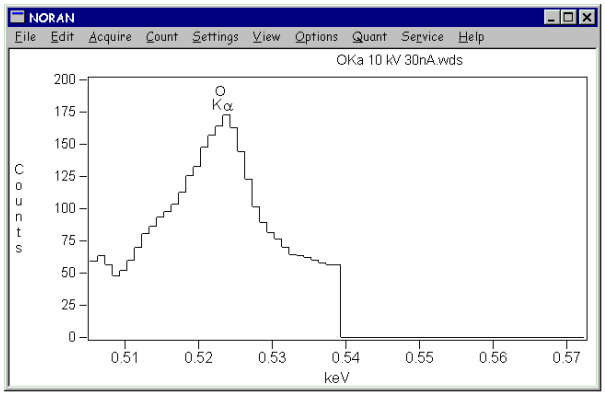
27. Begin the scan.

    From the **Acquire** menu, click **Start Scan**.

28. Record the peak energy value.

    First, move the cursor to the peaks center.

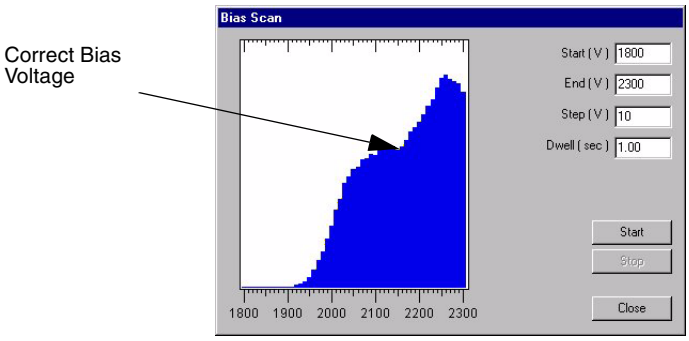
    Second, from the **View** menu, click **Peak Information**. The peak energy value displays.



Bias Scan

Determine the detectors bias voltage for an x-ray line.

1. From the **Settings** menu, click **Bias Scan**.



2. Begin the Scan.

    Use the systems default settings. Click **Start**.

3. Center the correct bias voltage.

The correct bias voltage is above the first shoulder, and prior to the sharp peak. If this point is not visible or not centered on the display modify the starting voltage until the correct voltage is determined. the example above shows a centered voltage point.

- If the display is to far left – increase the starting bias and decrease the ending bias by the same amounts.
- If the display is to far right – decrease the starting bias and increase the ending bias by the same amount.

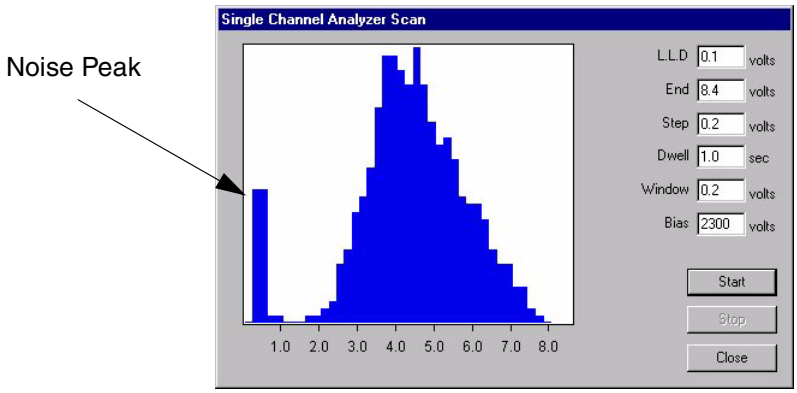
4. Exit Bias Scan.

   Click **Close**.

SCA Scan

A single channel analyzer (SCA) scan fine tunes the detector bias voltage (found with the Bias Scan function). The Detector Bias voltage, SCA Baseline voltage, and SCA Window voltage must be determined.

1. From the **Settings** menu, click **SCA Scan**.



2. Begin the scan.

   Enter the Bias voltage and Low Level Discriminator (LLD), and then click **Start**.

   - LLD – The voltage setting that prevents the noise peak from displaying.
   - Bias – The voltage determined during in the bias scan.
   - Use the system default settings for all other parameters.

3. Determine the correct SCA voltage.
   - The SCA voltage occurs at the valley between the noise peak and the signal peak.
   - The SCA Window voltage is the peaks width, from the baseline voltage to where the peak tails off.

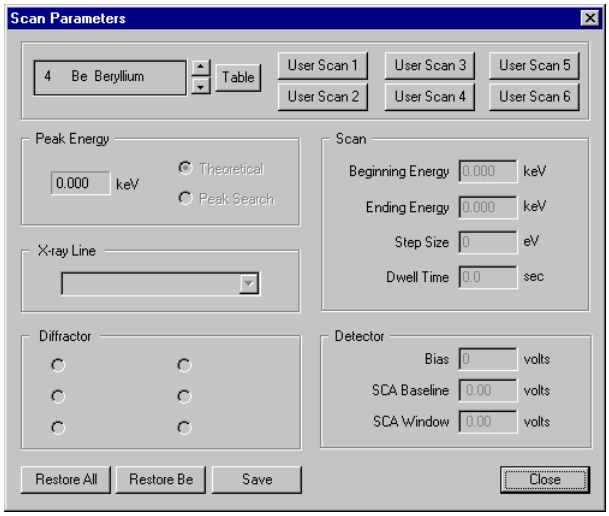
4. Exit Single Channel Analyzer Scan.

Click **Close**.

Scan Parameters

1. Set acquisition parameters for individual elements.

   From the **Edit** menu, click **Scan Parameters**.



2. Select an element.

   Click the up and down arrows in the top left corner of the dialog box to select an element. Or click **Table** to select from a periodic table of elements.

3. Select the X-ray lines.

   Click the X-ray Line drop down box to select the K, L, M, and N lines of interest.

4. Select a diffractor.

   Diffractor settings are passed to the Scan Setup Window. The diffractor options may vary from spectrometer to spectrometer.

5. Enter the elements scan parameters.

   - Beginning Energy — The scan's minimum energy level, in keV.
   - Ending Energy — The scan's maximum energy level, in keV.
   - Step Size — The spectrometer step size during acquisition, in eV.
   - Dwell Time — The dwell time on each step, in seconds.
   - Detector Bias — The optimum detector voltage for the scan range.
   - SCA Baseline — The optimum detector baseline voltage for the SCA scan. By optimizing this value, you can reject counts produced by noise.
   - SCA Window — The optimum range of detection for the SCA scan, in volts. By optimizing this value,

you can reject counts produced by noise. The sum of SCA Baseline and SCA Window must not exceed 8.4V.

6. Save the defined parameters.
   - To save the set of parameters click **Save**. Click **Yes** to confirm the save command.
   - To reset to the systems default settings click **Reset All**. The bias settings and the element table are reset. Click **Yes** to confirm the reset all command.
   - To reset the current element only click **Reset Be** (Beryllium). This buttons name changes each time the current element changes and only resets the current element.
7. Apply the changes to Scan Parameters.

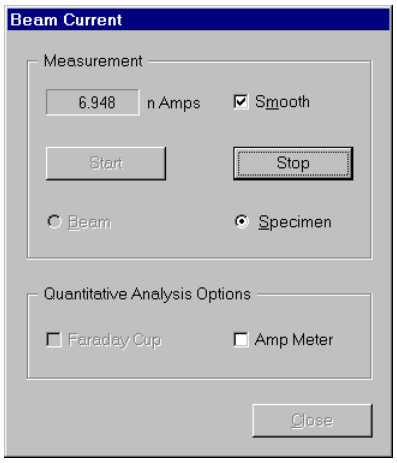
   Click **Close**.

Beam Current

An accurate beam current must be obtained for Quant Analysis. Current varies for many reason's. The beam current reading is constantly updated while running.

Note: Not all systems have the optional equipment necessary to perform this procedure.

1. From the **Settings** menu, click **Beam Current**.



2. Select a reading location.

   The beam current reading is obtains from the Specimen or Beam.
   - Specimen – Reads at the stage level.
   - Beam – Reads at the beam device level.
3. Start the beam current reading.

   Click **Start**. The beam current displays and continuously updates. To average the reading click

Damp.

4. Stop the reading.
5. Click Stop.

Setting Up Beam Current Meter

The Beam Current Meter is connected to the special serial port board of the MAXray or MAXray-II Controller. It is setup internally as COM Port 4 and although it does not use any interrupts, the serial port card can be configured for either IRQ 5 or IRQ 9. The Beam Current Meter is attached to the WDS Controller using a 9 pin male to 9 pin female serial cable. A null-modem cable is *not* necessary and will not work.

1. To use the Beam Current Meter:
   a. An RS–232 board must be installed and setup for COM4, IRQ5 or IRQ9.
   b. The METER.TXT file must be on the Controller boot disk.
3. The new Beam Current Meter commands that can be used in the Send Command are:

   READ BEAM ONE
   READ BEAM TWO
   FAR CUP IN
   FAR CUP OUT

4. To use the Beam Current Meter, the first line in the CRYSTAL.TXT file must be:

   BEAM METER YES

5. The new METER.TXT file must include the parameters for channel one sense, channel two sense, Faraday cup polarity and Faraday cup retraction delay time. Below are the acceptable values for each of these parameters.

   channel one sense 1 or —1
   channel 2 sense 1 or —l
   Faraday cup polarity0 or 1
   Faraday cup retraction delay time0, 1, 2, 4, 5 or 10 milliseconds

   A sample file looks like that below:

   CH1SENSE -1
   CH2SENSE 1
   FCUPPOL 0
   FCUPDELAY 2

6. To select the number of columns displayed, edit the AUTOEXEC.BAT file on the Controller boot disk to include one of the lines listed below. If none of these lines are found, the default of 60 rows is used.

   SET VIDEO25
   SET VIDEO3O
   SET VIDEO43
   SET VIDEO6O

7. On boot-up, the Controller loads the beam current parameters from the CRYSTAL.TXT file and test the beam current meter. The results of the test can be seen on a monitor attached to the Controller's VGA port.

Calibration Records

Use these pages for each crystal.

Detector and Diffractor Motor Calibration

Diffractor name _____
Diffractor 2d spacing _____ Angstroms
Element and X-ray line used _____
Theoretical peak energy of the x-ray line_____ keV
Sample used _____
Accelerating voltage _____ keV
Beam current _____ nAmps

First diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV_____ Counts

Second diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

First detector scan
Start _____ keV
Peak _____ keV _____ Counts
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second detector scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Crystal motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Detector motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Energy scan
Beginning Energy_____ keV
Ending Energy _____ keV
Step Size _____ eV
Dwell Time _____ sec
Detector Bias _____ volts
SCA Baseline _____ volts
SCA Window _____ volts
Crystal Name _____
Peak _____ keV _____ Counts

Detector Bias Voltage Calibration

Detector Bias Voltage_____ Volts

Single Channel Analyzer (SCA) Voltage Calibration

SCA Baseline Voltage_____ Volts
SCA Window Voltage_____ Volts

Detector and Diffractor Motor Calibration

Diffractor name _____
Diffractor 2d spacing _____ Angstroms
Element and x-ray
line used _____
Theoretical peak energy
of the x-ray line _____ keV
Sample used _____
Accelerating voltage _____ keV
Beam current _____ nAmps

First diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV_____ Counts

First detector scan
Start _____ keV
Peak _____ keV _____ Counts
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second detector scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Crystal motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Detector motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Energy scan
Beginning Energy_____ keV
Ending Energy _____ keV
Step Size _____ eV
Dwell Time _____ sec
Detector Bias _____ volts
SCA Baseline _____ volts
SCA Window _____ volts
Crystal Name _____
Peak _____ keV _____ Counts
Detector Bias Voltage Calibration
Detector Bias Voltage_____ Volts
Single Channel Analyzer (SCA) Voltage Calibration
SCA Baseline Voltage_____ Volts
SCA Window Voltage_____ Volts

Detector and Diffractor Motor Calibration

Diffractor name _____
Diffractor 2d spacing _____ Angstroms
Element and X-ray line used _____
Theoretical peak energy of the x-ray line_____ keV
Sample used _____
Accelerating voltage _____ keV
Beam current _____ nAmps

First diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV_____ Counts

First detector scan
Start _____ keV
Peak _____ keV _____ Counts
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second detector scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Crystal motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Detector motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Energy scan
Beginning Energy_____ keV
Ending Energy _____ keV
Step Size _____ eV
Dwell Time _____ sec
Detector Bias _____ volts
SCA Baseline _____ volts
SCA Window _____ volts
Crystal Name _____
Peak _____ keV _____ Counts

Detector Bias Voltage Calibration

Detector Bias Voltage_____ Volts

Single Channel Analyzer (SCA) Voltage Calibration

SCA Baseline Voltage_____ Volts
SCA Window Voltage_____ Volts

Detector and Diffractor Motor Calibration

Diffractor name _____
Diffractor 2d spacing _____ Angstroms
Element and x-ray
line used _____
Theoretical peak energy
of the x-ray line _____ keV
Sample used _____
Accelerating voltage _____ keV
Beam current _____ nAmps

First diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV_____ Counts

First detector scan
Start _____ keV
Peak _____ keV _____ Counts
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second detector scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Crystal motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Detector motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Energy scan
Beginning Energy_____ keV
Ending Energy _____ keV
Step Size _____ eV
Dwell Time _____ sec
Detector Bias _____ volts
SCA Baseline _____ volts
SCA Window _____ volts
Crystal Name _____
Peak _____ keV _____ Counts
Detector Bias Voltage Calibration
Detector Bias Voltage_____ Volts
Single Channel Analyzer (SCA) Voltage Calibration
SCA Baseline Voltage_____ Volts
SCA Window Voltage_____ Volts

Detector and Diffractor Motor Calibration

Diffractor name _____
Diffractor 2d spacing _____ Angstroms
Element and X-ray line used _____
Theoretical peak energy of the x-ray line_____ keV
Sample used _____
Accelerating voltage _____ keV
Beam current _____ nAmps

First diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV_____ Counts

First detector scan
Start _____ keV
Peak _____ keV _____ Counts
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second detector scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Crystal motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Detector motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Energy scan
Beginning Energy_____ keV
Ending Energy _____ keV
Step Size _____ eV
Dwell Time _____ sec
Detector Bias _____ volts
SCA Baseline _____ volts
SCA Window _____ volts
Crystal Name _____
Peak _____ keV _____ Counts

Detector Bias Voltage Calibration

Detector Bias Voltage_____ Volts

Single Channel Analyzer (SCA) Voltage Calibration

SCA Baseline Voltage_____ Volts
SCA Window Voltage_____ Volts

Detector and Diffractor Motor Calibration

Diffractor name _____
Diffractor 2d spacing _____ Angstroms
Element and x-ray
line used _____
Theoretical peak energy
of the x-ray line _____ keV
Sample used _____
Accelerating voltage _____ keV
Beam current _____ nAmps

First diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV_____ Counts

First detector scan
Start _____ keV
Peak _____ keV _____ Counts
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

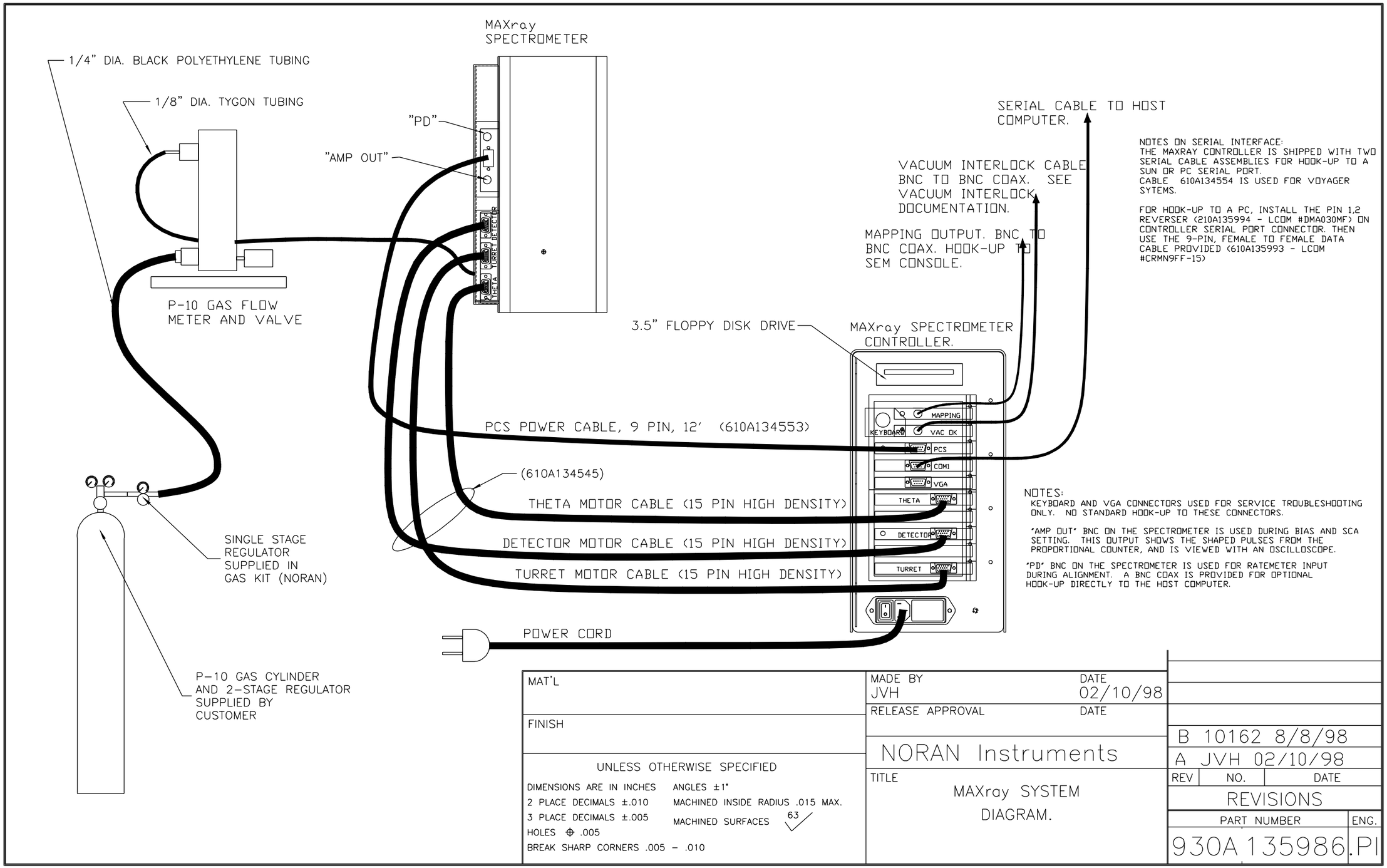
Second diffractor scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Second detector scan
Start _____ keV
End _____ keV
Step _____ eV
Dwell _____ sec
Peak _____ keV _____ Counts

Crystal motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

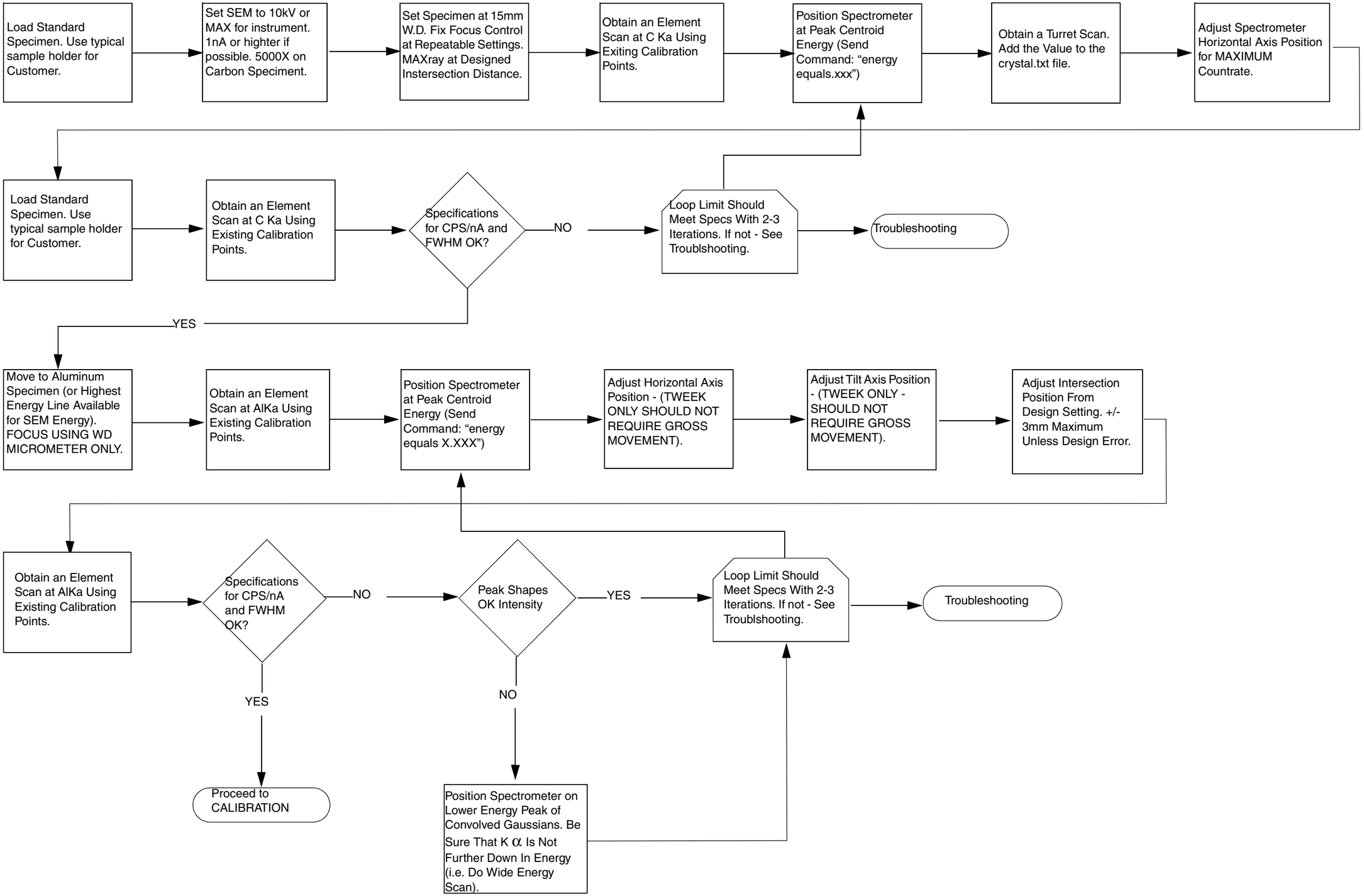
Detector motor offset computed
Theoretical _____ degrees
Measured _____ degrees
Computed _____ degree offset

Energy scan
Beginning Energy_____ keV
Ending Energy _____ keV
Step Size _____ eV
Dwell Time _____ sec
Detector Bias _____ volts
SCA Baseline _____ volts
SCA Window _____ volts
Crystal Name _____
Peak _____ keV _____ Counts
Detector Bias Voltage Calibration
Detector Bias Voltage_____ Volts
Single Channel Analyzer (SCA) Voltage Calibration
SCA Baseline Voltage_____ Volts
SCA Window Voltage_____ Volts

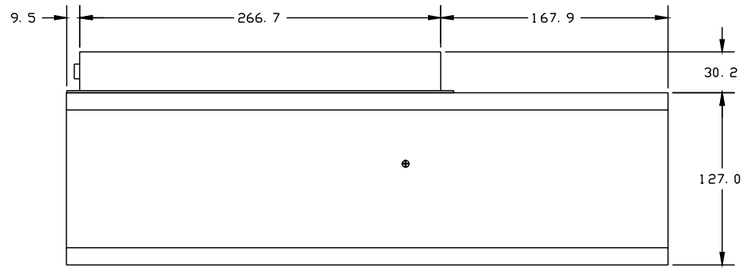


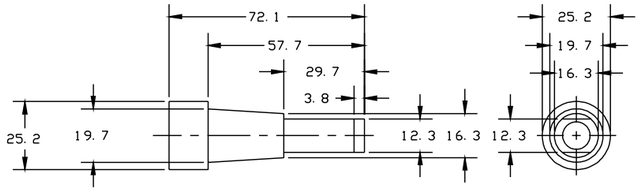
MAT'L	MADE BY JVH	DATE 02/10/98
FINISH	RELEASE APPROVAL	DATE
UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES — ANGLES ±1° 2 PLACE DECIMALS ±.010 — MACHINED INSIDE RADIUS .015 MAX. 3 PLACE DECIMALS ±.005 — MACHINED SURFACES 63 HOLES ⌖ .005 BREAK SHARP CORNERS .005 – .010	NORAN Instruments TITLE MAXray SYSTEM DIAGRAM.	B 10162 8/8/98 A JVH 02/10/98 REV | NO. | DATE REVISIONS PART NUMBER | ENG. 930A135986.PI

Alignment Procedure Flow Chart

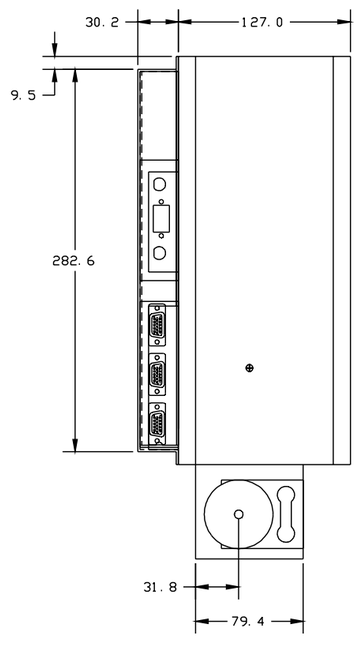


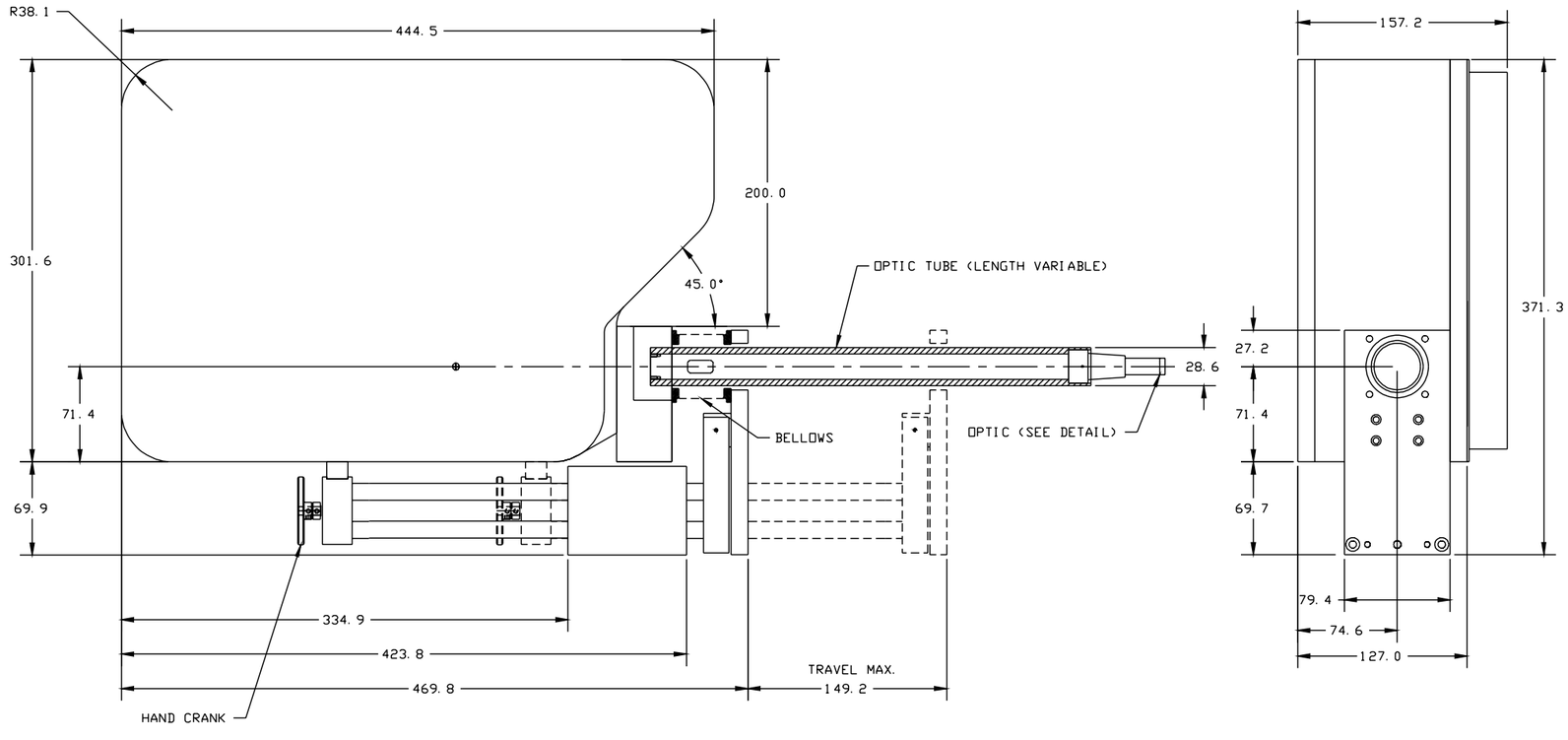
Engineering Layout





OPTIC DETAIL (2X)

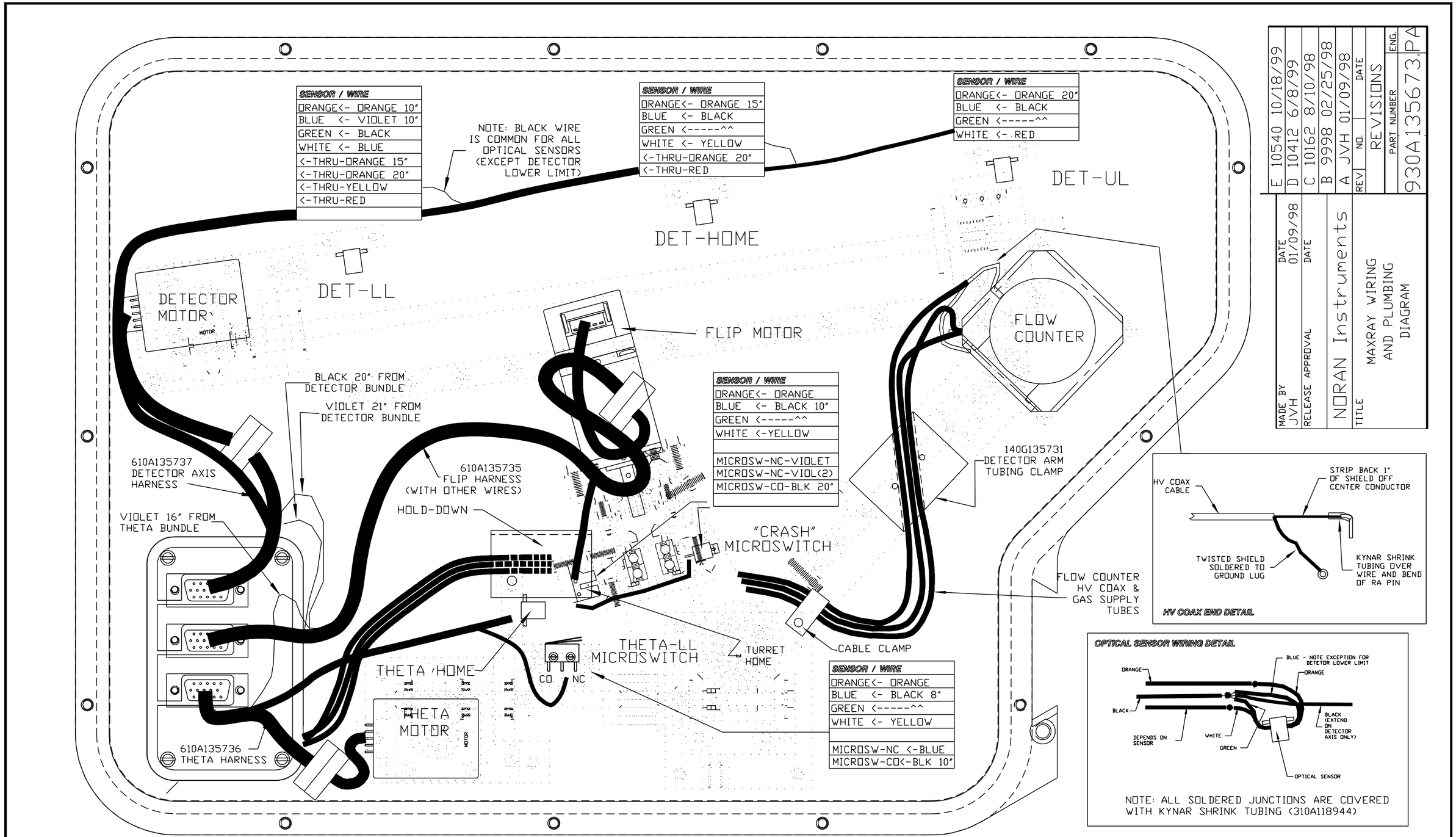




NOTE: Dimensions in millimeters

MAT'L	MADE BY SJF	DATE 11/04/97
FINISH	RELEASE APPROVAL	DATE
UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES ANGLES ±1° 2 PLACE DECIMALS ±.010 MACHINED INSIDE RADIUS .015 MAX. 3 PLACE DECIMALS ±.005 MACHINED SURFACES 63 HOLES Ø .005 BREAK SHARP CORNERS .005 - .010	NORAN Instruments TITLE MAXray ENGINEERING LAYOUT	A JVH 02/24/98 REV. NO. DATE REVISIONS PART NUMBER ENG. 140G135122E1

8 – Additional MAXray Drawings


SENSOR / WIRE
ORANGE<- ORANGE 10"
BLUE <- VIOLET 10"
GREEN <- BLACK
WHITE <- BLUE
<-THRU-ORANGE 15"
<-THRU-ORANGE 20"
<-THRU-YELLOW
<-THRU-RED
NOTE: BLACK WIRE IS COMMON FOR ALL OPTICAL SENSORS (EXCEPT DETECTOR LOWER LIMIT)
SENSOR / WIRE
ORANGE<- ORANGE 15"
BLUE <- BLACK
GREEN <-----^^
WHITE <- YELLOW
<-THRU-ORANGE 20"
<-THRU-RED
SENSOR / WIRE
ORANGE<- ORANGE 20"
BLUE <- BLACK
GREEN <-----^^
WHITE <- RED
DET-UL
DET-HOME
DET-LL
DETECTOR MOTOR
FLIP MOTOR
FLOW COUNTER
BLACK 20" FROM DETECTOR BUNDLE
VIOLET 21" FROM DETECTOR BUNDLE
610A135737 DETECTOR AXIS HARNESS
610A135735 FLIP HARNESS (WITH OTHER WIRES)
HOLD-DOWN
VIOLET 16" FROM THETA BUNDLE
"CRASH" MICROSWITCH
140G135731 DETECTOR ARM TUBING CLAMP
FLOW COUNTER HV COAX & GAS SUPPLY TUBES
CABLE CLAMP
THETA-LL MICROSWITCH
TURRET HOME
THETA HOME
CO NC
THETA MOTOR
610A135736 THETA HARNESS
SENSOR / WIRE
ORANGE<- ORANGE
BLUE <- BLACK 10"
GREEN <-----^^
WHITE <-YELLOW
MICROSW-NC-VIOLET
MICROSW-NC-VIOL(2)
MICROSW-CO-BLK 20"
SENSOR / WIRE
ORANGE<- ORANGE
BLUE <- BLACK 8"
GREEN <-----^^
WHITE <- YELLOW
MICROSW-NC <-BLUE
MICROSW-CO<-BLK 10"
HV COAX CABLE
STRIP BACK 1" OF SHIELD OFF CENTER CONDUCTOR
TWISTED SHIELD SOLDERED TO GROUND LUG
KYNAR SHRINK TUBING OVER WIRE AND BEND OF RA PIN
HV COAX END DETAIL
OPTICAL SENSOR WIRING DETAIL
ORANGE
BLUE - NOTE EXCEPTION FOR DETECTOR LOWER LIMIT
ORANGE
BLACK
DEPENDS ON SENSOR
WHITE
GREEN
BLACK (EXTEND ON DETECTOR AXIS ONLY)
OPTICAL SENSOR
NOTE: ALL SOLDERED JUNCTIONS ARE COVERED WITH KYNAR SHRINK TUBING (310A118944)
E 10540 10/18/99
D 10412 6/8/99
C 10162 8/10/98
B 9998 02/25/98
A JVH 01/09/98
REV NO. DATE
REVISIONS
PART NUMBER ENG.
930A135673.PA
MADE BY JVH DATE 01/09/98
RELEASE APPROVAL DATE
NORAN Instruments
TITLE
MAXRAY WIRING AND PLUMBING DIAGRAM

Flow Counter Repair/Replacement Instructions

WINDOW REPLACEMENT INSTRUCTIONS:

1. Remove the HV coax ground lead from the window mount by removing the M3 screw. Remove remaining M3 screws, which will allow the WINDOW SUPPORT to fall away. The broken window may be stuck against the flow counter opening.

2. Inspect the interior of the FLOW COUNTER for loose debris or possible damage to the ANODE WIRE. If debris is seen inside the counter - the counter should be removed from the spectrometer for cleaning. Remove 3 M2 screws from the base of the counter, and disconnect HV and GAS TUBING. Blow debris out using dry N2. If needed, a methanol rinse can help clean out small pieces of the window material.

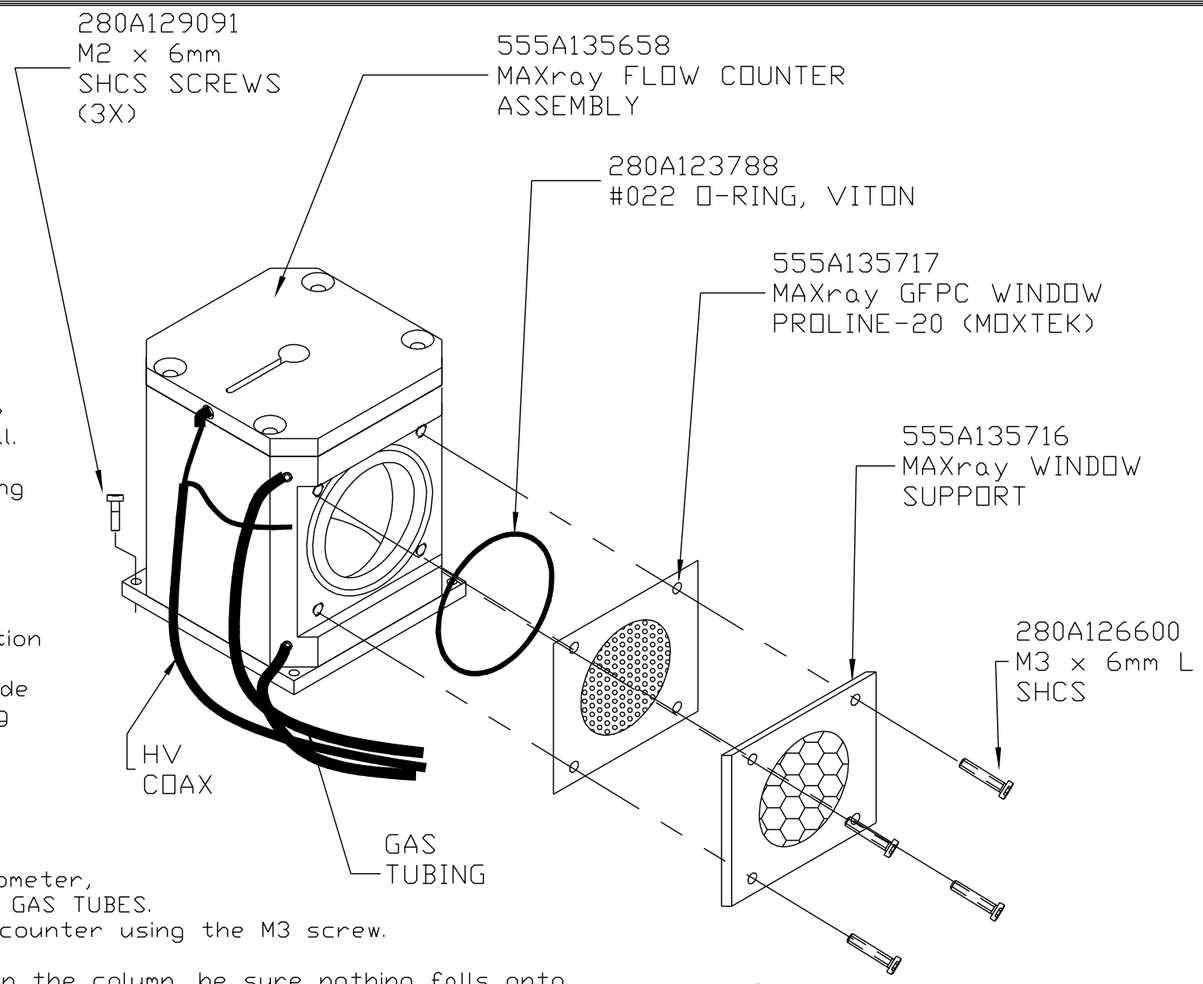
3. CAREFULLY remove the new WINDOW from the shipping box by grasping the stainless steel foil at the edge with tweezers. Position the window over the WINDOW SUPPORT such that the ALUMINIZED side will face into the flow counter during operation.

4. Pick up the WINDOW and WINDOW SUPPORT, and position over the opening of the flow counter. Be sure the o-ring is clean and seated in the groove. DO NOT slide the WINDOW laterally while pressed against the o-ring or damage to the window will occur.

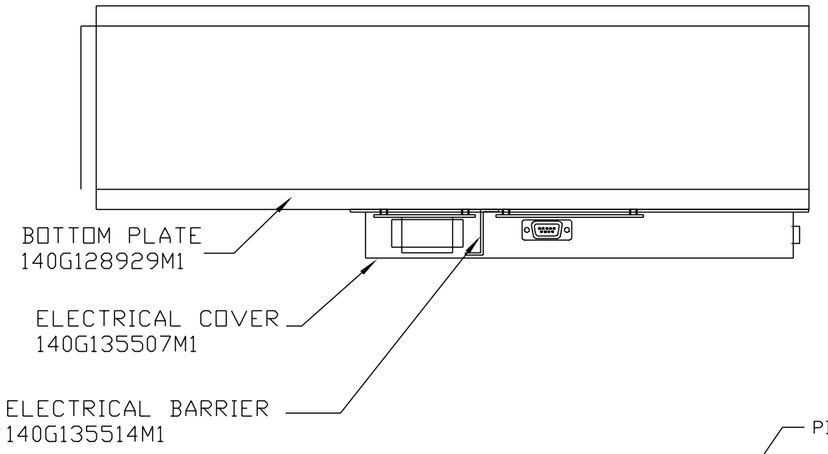
5. Insert all four M3 screws and tighten uniformly until the WINDOW is seated tight up to the opening. Remove the M3 screw needed for ground connection.

6. If the flow counter was removed from the spectrometer, re-mount to the pivot arm, and connect the HV, and GAS TUBES. Re-attach the HV GROUND to the front of the flow counter using the M3 screw.

NOTES: If installing with the spectrometer mounted on the column, be sure nothing falls onto the diffractors. Move the counter into an easily accessible position.



MAT'L	MADE BY JVH	DATE 2/24/98
FINISH	RELEASE APPROVAL	DATE
UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES ANGLES ±1° 2 PLACE DECIMALS ±.010 MACHINED INSIDE RADIUS .015 MAX. 3 PLACE DECIMALS ±.005 MACHINED SURFACES 63 HOLES ⌖ .005 BREAK SHARP CORNERS .005 - .010	NORAN Instruments TITLE MAXRAY FLOW COUNTER REPAIR/REPLACEMENT INSTRUCTIONS.	B 10162 8-6-98 A JVH 2/24/98 REV NO. DATE REVISIONS PART NUMBER ENG. 930A135987 PI



ASSEMBLY NOTES FOR MOUNTING OR REPLACING THE PCS ELECTRONICS ON A MAXRAY SPECTROMETER.

1. NORAN P/N 700X135656 IS AN ASSEMBLY OF WIRED AND TESTED PCS ELECTRONICS BOARDS. SPECIFICALLY THE PARTS SHOWN ON THIS DRAWING ARE CONTAINED ON THE ASSEMBLY TEST CHASSIS: 176A117492 (HV-II PWA BOARD)
176A141715 (AMPLIFIER PWA BOARD)
140G135551 (ELECTRICAL BARRIER)
ALSO, THE CONNECTOR BRACKET CONTAINING THE PCS POWER INPUT CONNECTOR, AND THE TWO OUTPUT BNCs ARE INCLUDED ON THIS ASSEMBLY.

2. THE CIRCUIT BOARDS ARE UNSCREWED FROM THE STANDOFFS ON THE TEST CHASSIS, AND THE CONNECTOR BRACKET IS UNSCREWED FROM THE CHASSIS. THE HIGH VOLTAGE AND GROUND WIRE IS UNSOLDERED FROM THE SHV CONNECTOR ON THE TEST CHASSIS.

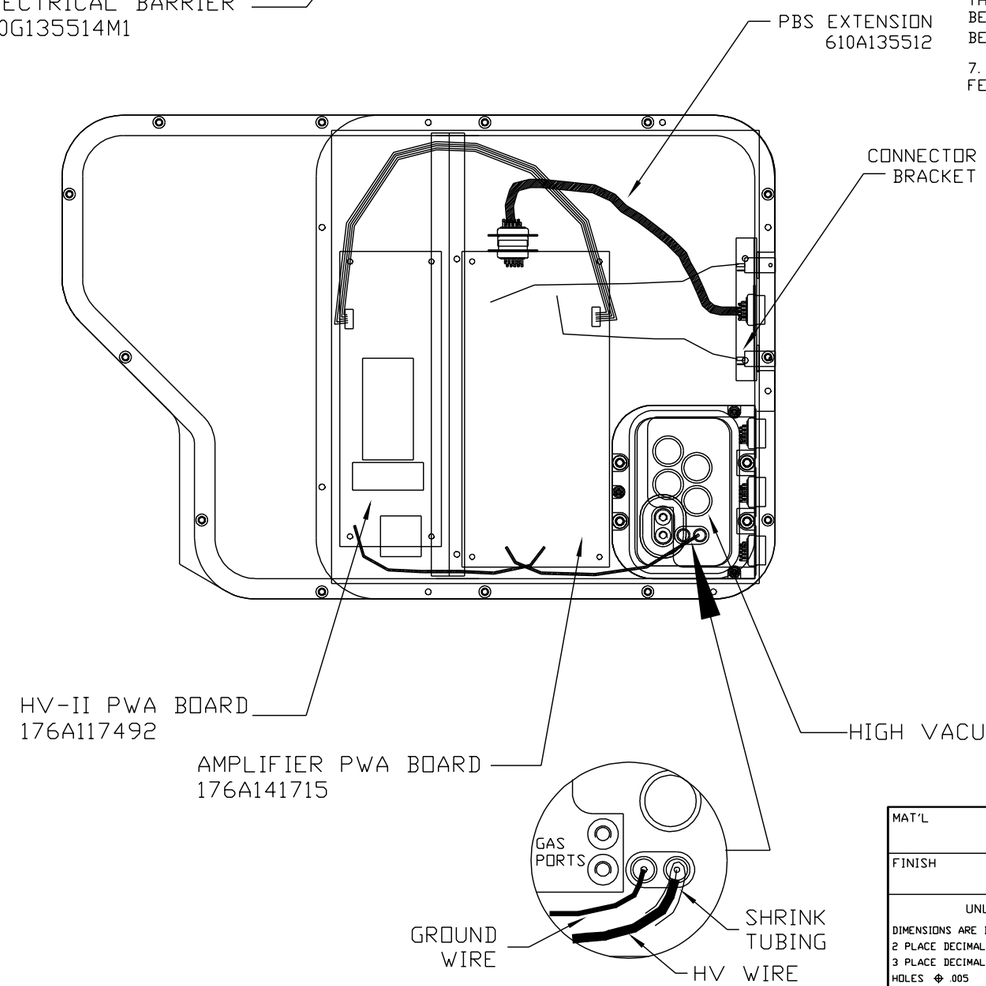
3. REMOVE THE BOARDS AND CONNECTOR BRACKET FROM THE CHASSIS MAINTAINING ALL ELECTRICAL CONNECTIONS. PLACE BOARDS ON A TABLE TOP.

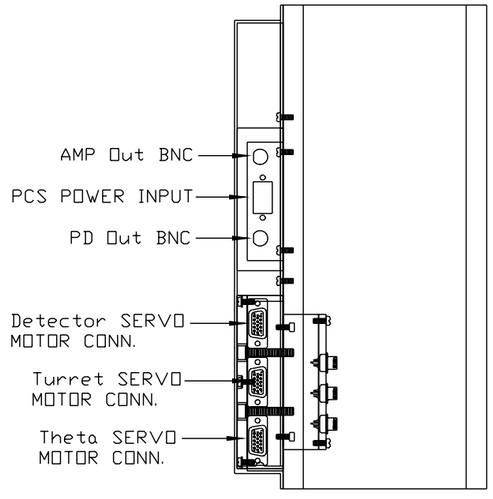
4. IF INSTALLING PCS ELECTRONICS ON A NEW MAXRAY, MOUNT THE ELECTRICAL BARRIER (140G135514) IN PLACE AS SHOWN ON THE SPECTROMETER BODY. IF REPLACING THE PCS ELECTRONICS, THEN THE BARRIER SHOULD NOT BE REMOVED.

5. ON A NEW ASSEMBLY, APPLY 3" WIDE KAPTON TAPE ALONG THE SIDE OF THE ELECTRICAL BARRIER WHERE THE HV-II PWA BOARD IS LOCATED. THE TAPE SHOULD BE CUT BACK FROM THE PAINT EDGE BY 0.75" TO ALLOW FOR THE COVER TO MAKE CONTACT WITH THE SPECTROMETER BODY.

6. SECURE THE CIRCUIT BOARDS USING BUTTON SCREWS AND PLASTIC SPACERS. SECURE THE CONNECTOR BRACKET WITH TWO SCREWS. BE SURE TO LOCATE WIRES AND CABLES BETWEEN THE TWO BOARDS, IN THEIR RESPECTIVE SLOTS IN THE ELECTRICAL BARRIER. BE SURE TO SECURE GROUND LUG ON HV BOARD.

7. SOLDER THE HV AND GOUND LEADS TO THEIR RESPECTIVE TERMINAL ON THE VACUUM FEEDTHROUGH. COVER HV SOLDER CONNECTION WITH SHRINK TUBING - SEE DETAIL BELOW.





NOTE: FOR OTHER DETAILS OF FEEDTHROUGH WIRING SEE 930A135674

MAT'L	MADE BY JVH	DATE 02/09/98
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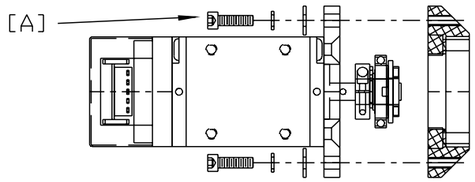
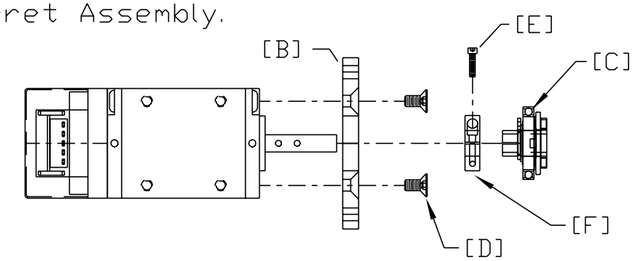
Turret (Flip) Motor.



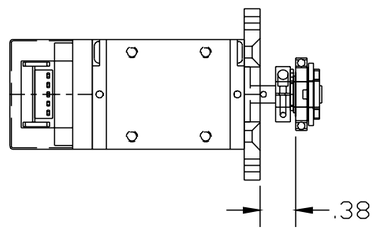
Only the motor is supplied for the Turret assembly.
350A132828 - Pittman Motor

1. Remove defective motor by disconnecting 9-Pin connector from Flip Motor Harness. Loosen 4 #4-40 SHC screws [A], and pull motor out of Turret Assembly.



2. Remove Flip Motor Mount [B], and Harmonic Drive Wave Generator [C], from defective motor. Mount these parts onto the new motor using the same screws ([D] and [E]) and Harmonic Drive Shaft Clamp [F].

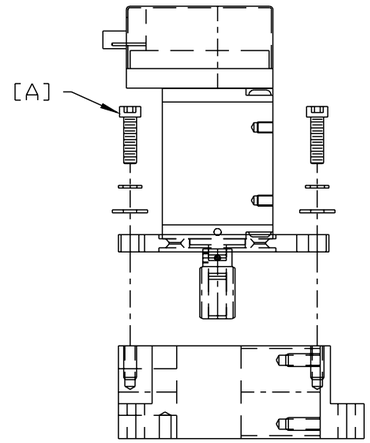
3. Note gapping between face of Flip Motor Mount and the Wave Generator.



4. Re-mount the new motor assembly using the 4 #4-40 screws. The assembly is self aligning.

5. Hook up the controller and test for smooth motor movement before re-installing on the column.

Detector or Theta Motor

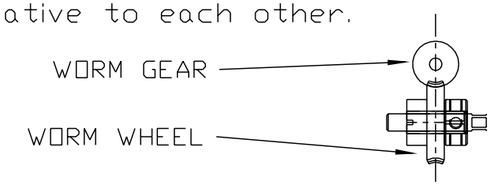


Both the Detector and Theta motors are similar in replacment. Parts supplied will have motor mount and worm gear already assembled.

Motor assemblies have different worm gears. Do not attempt to use a Detector motor in the Theta Drive (or the opposite).

700P135595 Theta Motor Assy.
Single Start Worm
700P135600 Detector Mtr Assy.
Quad Start Worm.

1. Disconnect 9-Pin Ultimate connector on motor/ encoder harness to defective motor.
2. Remove defective motor by loosening 4 #6-32 SHC screws [A].
3. Install new motor assembly - being sure to orient correctly for the mount.
4. Adjust play in worm gear/worm wheel mesh. There should be minimal play, but do not bind gears. Note how worm wheel and worm gear should be centered relative to each other.



5. Hook up the controller, and test motor movement before re-installing spectrometer.

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The MAXray Spectrometer is shipped from the factory with the standard set of diffractors already loaded. However, the occasion may arise where a diffractor must be replaced due to change in customer applications, new types of diffractors becoming available, or some type of upgrade.

NOTE: ALWAYS WEAR GLOVES WHEN HANDLING DIFFRACTORS. DO NOT DROP DIFFRACTORS OR TOTAL LOSS MAY OCCUR.

1. Power down the MAXray CONTROLLER, and disconnect the PCS POWER cable. Reboot the controller.

2. Initialize the motors to home - which will pull the Theta Mechanism to 15 degrees to facilitate easy exchange of crystals.

3. In the Services menu, select Diffractors, and then select the diffractor to be replaced or changed. This will position the diffractor at the active position, which also facilitates removal. Vent the column using typical precautions (Controller power can stay on with the PCS power cable disconnected), and remove the side cover of the spectrometer.

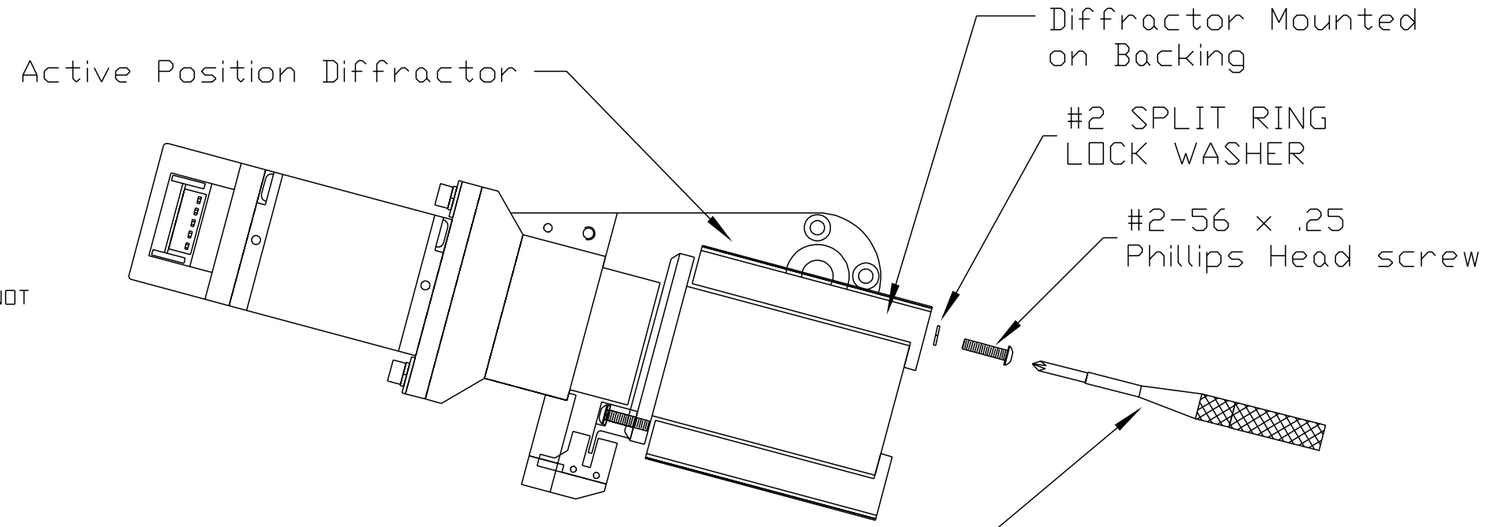
4. Remove the 2-56 screw holding the diffractor backing on the TURRET. Pull the diffractor off of the PINS at the bottom of the TURRET - this may require some wiggling of the backing. Place diffractor into an appropriate container to prevent damage.

5. Install the new diffractor, by sliding into place over the PINS, and then securing with the #2-56 screw and lockwasher. DO NOT OVER TIGHTEN, but the screw should be snug. Use the menu commands to move Diffractors an be sure that the TURRET mechanism still functions properly.

6. Replace the MAXray side cover, and evacuate the system.

7. During any change of the diffractors, there is a chance that the TURRET position will move, thus changing the relative home position. TURRET SCANS will be necessary on all diffractors to verify that the HOME OFFSET has not changed. The service engineer should verify full functioning of the spectrometer after completing the installation of a new diffractor.

8. Be sure to modify the CRYSTAL.TXT file on the MAXray CONTROLLER FLOPPY DISK to accurately reflect changes made to the crystal configuration.



MAT'L	MADE BY JVH	DATE 02/25/98
FINISH	RELEASE APPROVAL	DATE
UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES — ANGLES ±1° 2 PLACE DECIMALS ±.010 — MACHINED INSIDE RADIUS .015 MAX. 3 PLACE DECIMALS ±.005 — MACHINED SURFACES 63 HOLES ⌖ .005 BREAK SHARP CORNERS .005 - .010	NORAN Instruments TITLE MAXray DIFFRACTOR REPLACEMENT INSTRUCTIONS.	A JVH 02/25/98 REV | NO. | DATE REVISIONS PART NUMBER | ENG. 930A135989 | PI

9 – MAXray Field Replaceable Parts

The table below describes the field re-placable parts for the MAXray spectrometer. Failures of the spectrometer which involve other components will probably require factory repair.

Supporting documents are provided in the Installation and Alignment Procedures documentation, and should be referenced prior to installation of any replacement parts.

Part Description	NORAN P/N	Supporting Documents
Flow Counter Window	555A135717	930A135978
Flow Counter Window Support	555A135716	930A135987
PCS Electronics	700X135656	930A135984
X-ray OPTIC	555A135665	930A135985
Detector Motor	700P135600	930A135988
Theta Motor	700P135595	930A135988
Turret Motor	350A132828	930A135988
Optical Sensor - All Locations	370A133449	930A135673
Microswitch Limit Switch	510A133869	930A135673
TAP Diffractor	555A135685	930A135989
WSi-50 Diffractor	555A135686	930A135989
WSi-60 Diffractor	555A135687	930A135989
WSi-80 Diffractor	555A135688	930A135989
NiC-80 Diffractor	555A135689	930A135989
CrSc-80 Diffractor	555A135690	930A135989
NiC-100 Diffractor	555A135691	930A135989
MoB4C-145 Diffractor	555A135692	930A135989

10 – Cleaning Procedures for Synthetic and Natural Crystal Diffractors

Cleaning procedure must be preformed by a Service Representative. When crystals are placed in a vacuum environment produced by mechanical pumps, diffusion pumps, or a combination of the two, oil back-streaming can occur which will contaminate the surface of the diffracting elements in a WDS spectrometer. In some instances the oil films can become thick enough to lower the detected count rates due to absorption. The same is true for the x-ray detector windows. Crystals can be cleaned with care. Flow counter windows need to be replaced. The following is the recommended procedure for cleaning of various diffractors.

Synthetic Multilayer

Synthetic Multilayer diffractors can be cleaned by rinsing with isopropyl alcohol and gently blow drying with nitrogen. DO NOT wipe the surface of the diffractor as this can scratch the surface.

TAP

The TAP crystal is the most affected by oil contamination. TAP is a water soluble crystal. Remove any oil contamination using a cotton ball wetted with acetone. It is necessary to etch the crystal after cleaning due to the destruction of the surface layers during cleaning. Dip the crystal into room temperature distilled water for 10 seconds and remove. Gently wipe and water droplets from the surface with a dry cotton ball. DO NOT use air or nitrogen. A gas stream can cause the crystal to crack due to thermal effects.

PET

The PET crystal is cleaned similarly to the TAP crystal. Although PET isn't as sensitive to thermal stresses, use the same precautions during drying.

LiF

The LiF crystal will normally not show any loss of intensity due to oil contamination because the x-rays diffracted by LiF are not easily absorbed. The crystal can be cleaned with a cotton ball wetted with acetone. No etching is necessary or possible except at the factory.

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