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ARL 9800 Series
User Manual

AA83414

Analyze • Detect • Measure • Control™

Thermo
ELECTRON CORPORATION

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USER MANUAL

ARL 9800 SERIES

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ÄNDERUNGSANGABEN

REVISION CONTROL SHEET

Date Datum Date	Modification Änderung Revision	Description Beschreibung Description
1999 February July	01	Introduced Instrument Maintenance table in chapter 4. Chapter 4, Page 4-21 updated. Type of instrument ARL 9800 changed to ARL 9800 XP.
2000 February September	02 OM3301	Addition of ARL 9800 OASIS features. Chapter 2, page 2-5, introduced cassette with aperture ∅ 31 mm.
2001 August	03/KJu	General corrections and adaptation to WinXRF 3.0-1
2003 September	04/DBo	Addition of Appendix D (Free Lime Procedure)

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OVERVIEW AND WARNINGS

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1 OVERVIEW AND WARNINGS

Introduction

We congratulate you on the purchase of your ARL 9800 Series. This precise and accurate Spectrometer has been produced by ARL to satisfy your analytical needs.

The purpose of this manual is to assist you in the use of your ARL 9800 Series instrument.

The ARL 9800

X-ray fluorescence allows measurement of up to 83 elements of the periodic table in samples of various forms and nature: solids, conductive or non-conductive. Typical samples include glasses, plastics, all metals, ores, refractories, cement and geological materials. Any sample which may react violently to X-rays must be avoided. The samples must withstand analysis under vacuum (4-5 Pa). Advantages of XRF over other techniques are speed of analysis, generally easy sample preparation, very good stability, precision and wide dynamic range.

The ARL 9800 XRF Instrument can be provided in two different versions.

The ARL 9800 XP provides high performance measurements on all types of solid samples with high power X-ray generator.

The ARL 9800 Oasis is a lower power version independent of external water cooling.

The heart of the ARL 9800 can be made of various modules: monochromators for rapid, dedicated routine analysis, a Moiré fringe universal goniometer for flexible elemental analysis and an integrated X-ray diffraction system for specific phase and mineral analysis. The combination of fixed channels and goniometer guarantees speed, flexibility and reliability of analysis.

Simultaneous X-ray Fluorescence

The instrument can be equipped with up to 32 fixed channels or monochromators for simultaneous elemental analysis. Fixed channels ensure that analysis is rapid, precise and reliable.

The Oasis version is limited with maximum 20 monochromators in the simultaneous configuration.

Sequential X-ray Fluorescence

The instrument can also be equipped with a goniometer which gives the instrument great flexibility as it can analyse sequentially up to 83 elements of the periodic table providing that the necessary analysing crystals are fitted.

In addition, the goniometer allows for the use of semi-quantitative software (UniQuant, QuantAS) which provide the capacity to analyse rapidly totally unknown samples without the need to calibrate the instrument with standard reference materials.

The ARL 9800 XP with XRF goniometer can be further equipped with 24 monochromators.

The ARL 9800 Oasis version with XRF goniometer can be further equipped with 14 monochromators.

X-ray Diffraction

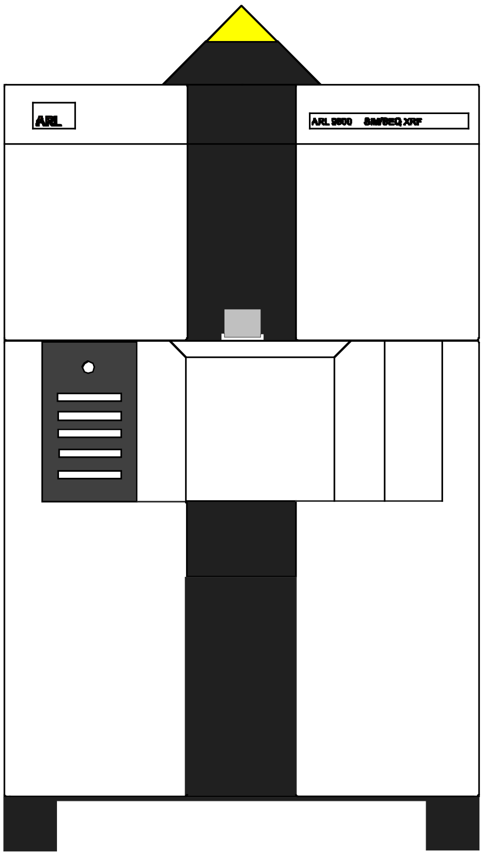
The instrument can also be equipped with an XRD system that permits the determination of phases or mineral compounds in the sample in various applications. For example, free lime in clinker and limestone in cement or the various states of iron oxide in iron ores can be determined.

With an XRD system, the ARL 9800 XP instrument can be further equipped with 24 monochromators. With one goniometer and one XRD system, the ARL 9800 XP accepts 14 additional monochromators.

In the Oasis version, the number of monochromator is limited to 14 with an XRD system and to 8 when the instrument is equipped with the XRD system and the goniometer.

The ARL 9800 should be configured according to your applications and your needs.

ARL 9800 without Sample Changer



KXx9875D00100

Figure 1.1

ARL 9800 with the 12 Position Sample Changer

Figure 1.2

ARL 9800 with the X-Y Sample Changer (98 cassettes)

Figure 1.3

ARL 9800 integrated for Automation Systems

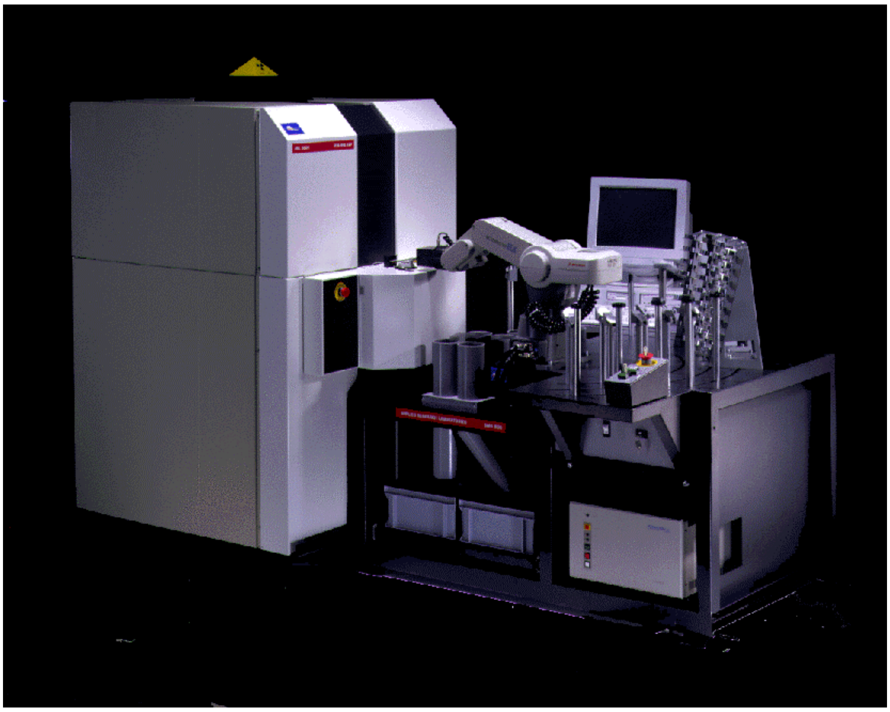


Figure 1.4

X-ray Fluorescence Spectrometer Principle

The sample to be measured is loaded into the spectrometer and excited by the X-ray beam coming from the X-ray tube. The spectrum of the tube is composed of the characteristic wavelengths of the anode element (Rh in general) and the continuum (or white radiation). The emitted radiation from the sample is a mixture of the tube spectrum and the characteristic wavelengths of the elements in the sample.

The reflected beam is guided onto wavelength dispersive systems called, in our case, monochromator or goniometer. These elements produce spectral lines, which are related to the elements present in the measured sample.

The XRD system collects in its detector the diffracted X-ray of one specific wavelength emitted by the X-ray tube. The incident beam is diffracted by the various crystallographic planes of the crystallites which are present in the sample. For more details about XRD analysis, please refer to the specific brochure about the “ARL 9800 - Integrated XRD system”.

All XRF and XRD spectrometers measure intensities. The concentrations can only be obtained after the instrument has been calibrated. It should be stressed that an XRF quantometer is a very accurate comparator, but the accuracy of the final analysis is entirely dependent on the quality of the standard samples used for calibration. The intensity concentration relationship is generally linear, but in some case a second order curve can be used.

$$C(\%) = a_0 + a_1 * I \qquad \text{First degree}$$

$$C(\%) = a_0 + a_1 * I + a_2 * I^2 \qquad \text{Second degree}$$

Where : I is the intensity measured by the XRF spectrometer
a_j are the constants computed during the calibration
C is the concentration in %

In practice, the intensity of an element is not only a function of the concentration of the element analysed but may also be influenced by interferences such as line overlapping, absorption or enhancement (matrix effects), grain size effects and mineralogical effects. The chapter *Analysis principle* gives more information.

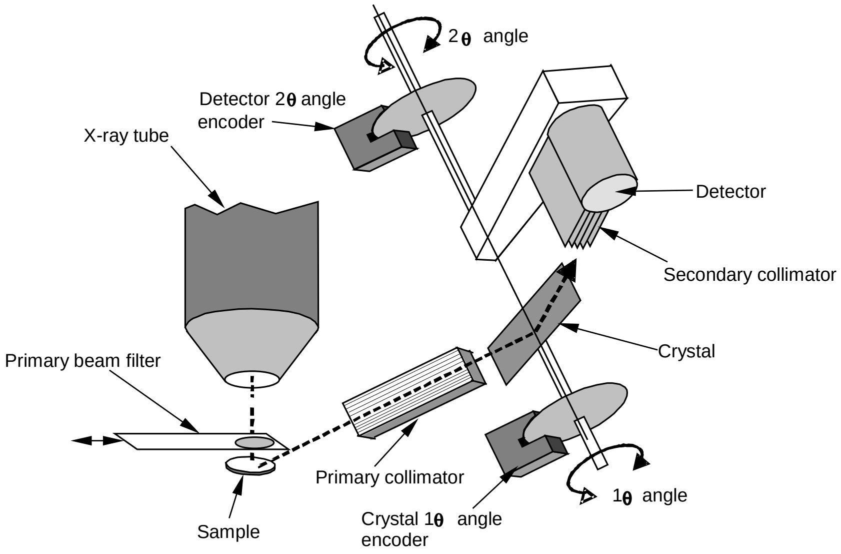
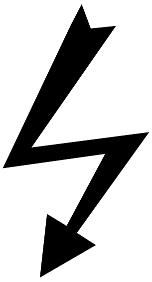


Figure 1.5

General Warnings

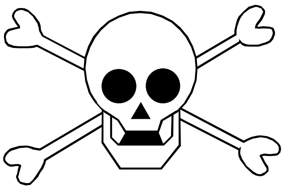
The XRF instrument uses several components which can be dangerous to manipulate if you have to open the cabinet or the tank. Whenever one of the following signs is seen stuck to a support (tank cover, panels etc...) you are accessing a potentially dangerous area.

In case of problem or injury, ARL will accept no responsibility if the user has personally removed one or several covers or panels having any of the following signs affixed.



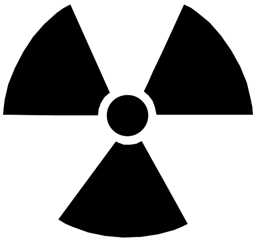
This panel or this part must be only removed when the power is off. Otherwise there is a risk of electrocution.

Figure 1.6



These components are dangerous to handle i.e.: Beryllium or TLAP crystal. These components and their supports must be handled with gloves. The user must not be in contact with these parts.

Figure 1.7



The instrument can generate X-rays. All components shielding against X-ray leakages are secured by safety switches which are connected to the X-ray power supply interlock. As soon as any such component is removed or is missing the X-ray power supply switches off immediately.

Figure 1.8

The ARL 9800 is equipped with an emergency button that switches off the main power of the spectrometer when it is pushed. There is also an interlock safety system used to protect the instrument if the normal conditions of use are not reached.

The user must contact the nearest local service if he needs to open the instrument. The user can also attend a special training course at our factory in Switzerland to learn how to maintain the instrument in good working order.

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User Responsibility

It is the user responsibility to follow **general warnings**.

It is the responsibility of the user to ensure that the recommended **laboratory environmental conditions are maintained** in the future.

It is the responsibility of the user to ensure that compliance with local **X-ray safety regulations** are fulfilled. Since X-rays are produced in the instrument, **special authorisation** is requested in several countries to be allowed to run such equipment.

It is the responsibility of the user to be sure that **the samples** loaded into the ARL 9800 do not react with the X-ray beam or the vacuum environment. All spectrometer damages due to **bad samples** are considered as misuse.

X-ray radiation can be used with minimal risk if the user follows X-ray safety guidelines established by state, local, and institutional regulations. Additionally, the user must use the instrument with all safety features engaged and in proper working order. The ARL 9800 instrument itself, including the access panels, is designed to absorb or otherwise prevent X-ray radiation from escaping the instrument. Thus, **whenever a measurement is performed, it is very important to have all panels and safety features engaged**. Do not try to defeat the safety features of the machine.

Total Protection

This instrument is designed to fulfil the Swiss law about X-ray Safety for the total protection class. The total protection class means that the leaks rate around the instrument is lower than 1 µSievert/hour (0.1 mRem/h) at 10 cm of the surface of the cabinet. This also means that the instrument is equipped with safety features that close the shutter or shut down the X-ray Power Supply if the user attempts to reach the unprotected area inside the cabinet.

This instrument was checked and its radiation measured by a neutral official organisation before the authorization was given to ARL by the Swiss authority. See the authorization in *Appendix A* of the *Pre-installation Manual*.

This authorization is not valid in any other country but can be used by the user to get the local authorization.

2

INSTRUMENT DESCRIPTION

2 INSTRUMENT DESCRIPTION

View of the ARL 9800

The ARL 9800 requires approximately 1 square meter floor space. It is therefore designed to be placed in very small areas. Its weight is about 750 kg.

The displays of the instrument inform the user about the spectrometer status, X-ray tube conditions, the monochomators, the goniometer or the XRD system status with all parameters related to the measured element lines. Three to six displays are available depending on the configuration of the instrument.

The yellow pyramid located on top of the instrument is the “X-ray on” signal. This lamp is illuminated when the X-ray power supply is on. The red button is used to switch off the main power supply in emergency cases.

Front view with 12 Position Sample Changer

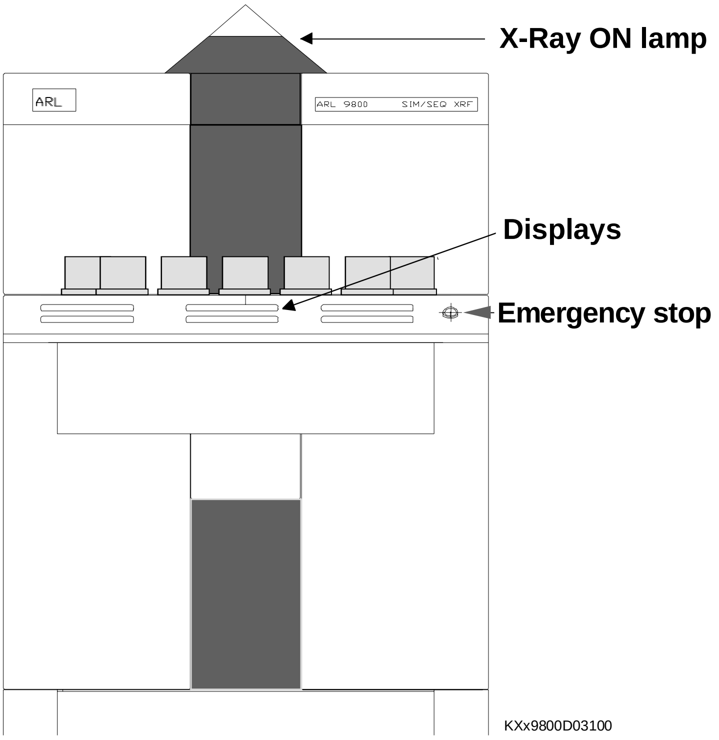

Figure 2.1

Top view with 12 Position Sample Changer

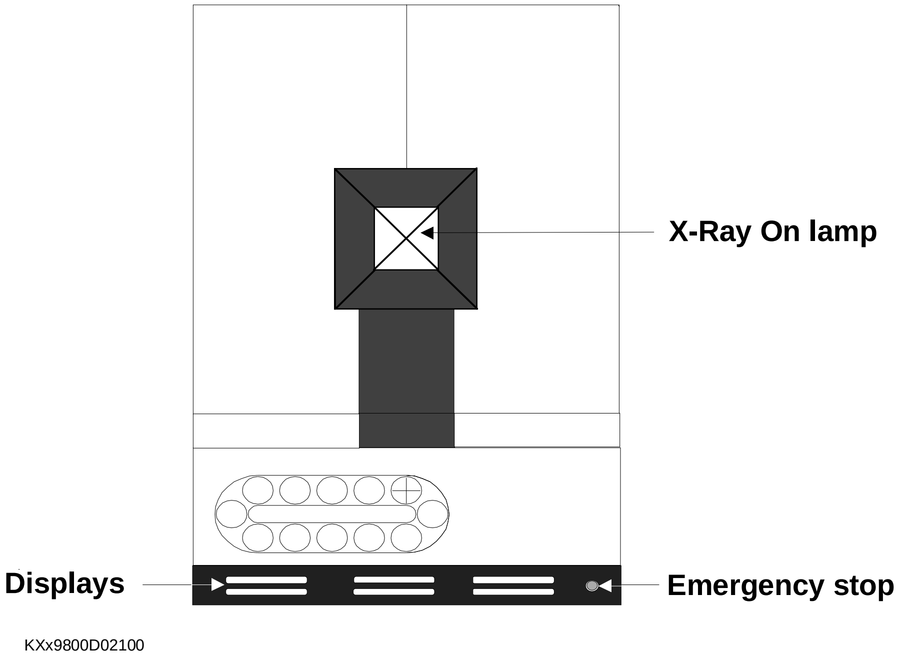

Figure 2.2

Front view with X-Y Sample Changer

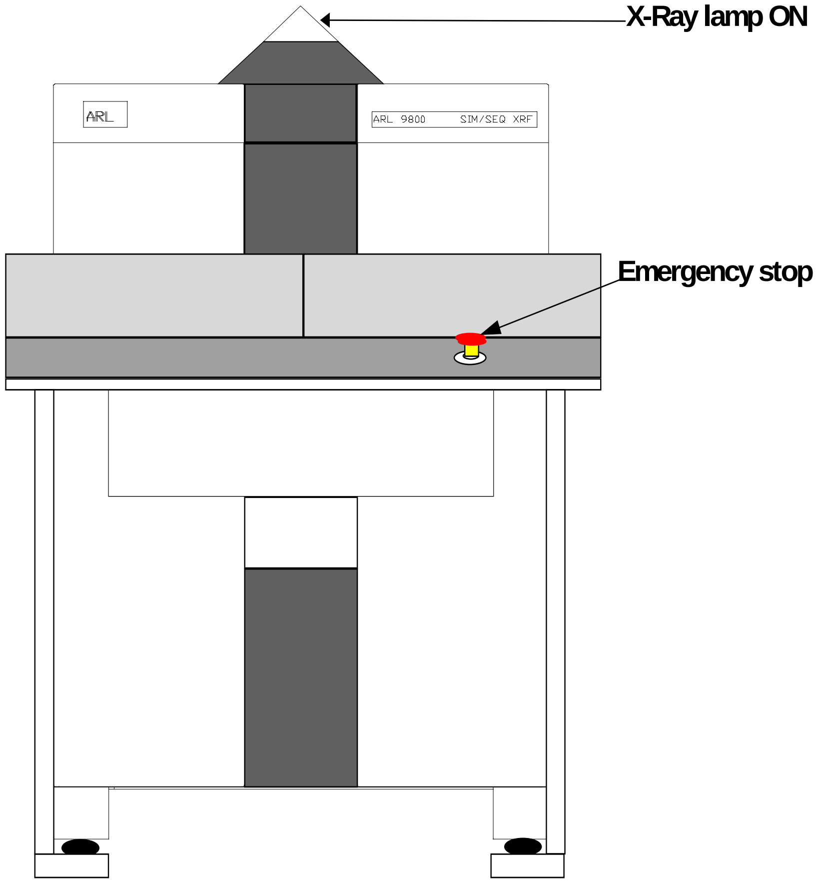

Figure 2.3

Front view of the instrument prepared for the Automation System

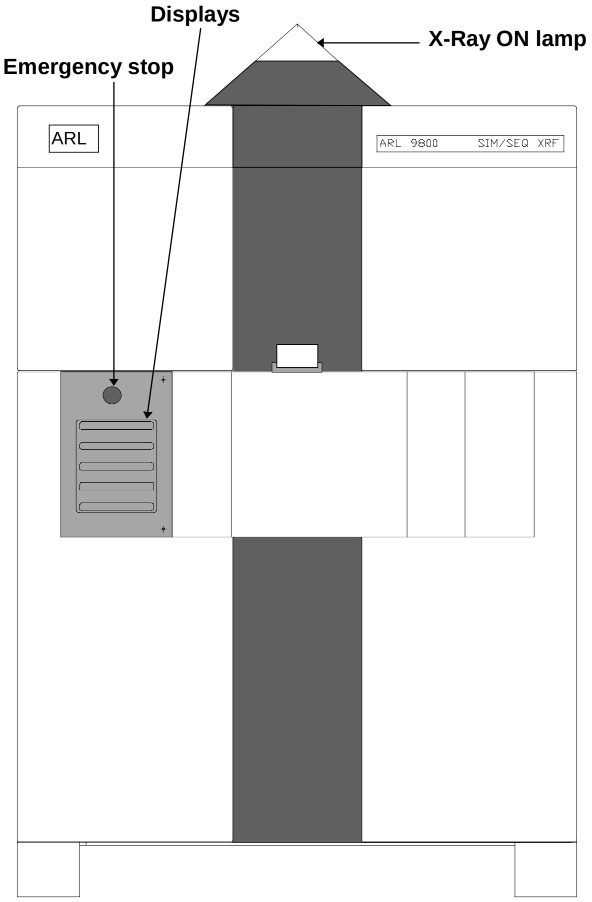

Figure 2.4

On the back side of the ARL 9800 and as shown in the figure below, we can find the following features:

1. The 230 V plug for the computer (max. 3.15 A).
2. The main breaker (32 A) for the X-ray tube generator.

Note: With the ARL 9800 Oasis, this breaker is used like a switch, to turn on off the low power generator. In this instrument, the low power generator has its own breaker (25 A) which is located in the generator front panel.

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3. The main breaker (16 A) for the electronic.
4. The connector for the computer - instrument link (ACS Link).
5. The master reset, to activate the master reset, use for example a small screwdriver, insert it and press.

Features and External Connections of the ARL 9800

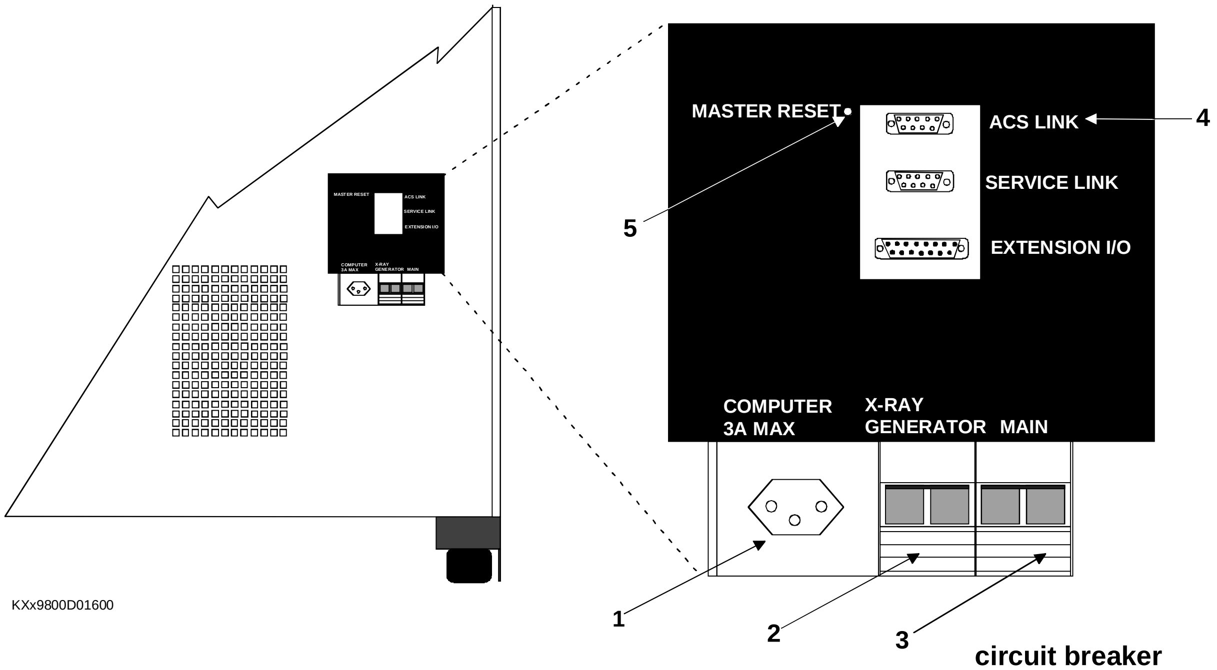


Figure 2.5

Note: The connectors ‘’SERVICE LINK” and “EXTENSION I/O” located just below the “ICS LINK” are for future applications and for service use only. The user should not connect any devices to these, unless specifically informed.

Sample Holders

Every sample must be placed in a sample holder before it can be introduced into the ARL 9800. The introduction of a sample without the correct holder will stop or damage the sample handling system.

There are two different kinds of sample holders:

- **Cassettes** are used for handling any kind of solid and pressed powder samples.
  Large cassettes are used with the 12 Position Sample Changer.
  Small cassettes are used with the X-Y sample changer and with the single position loading.
- **Sample Adapters** are used for handling samples without cassettes.
  4 different families of sample diameters are defined for the large X-Y sample changer.
  In case of automation, the centring ring can be adapted to the production sample size and shape.

Cassettes

The standard cassette aperture is ∅ 29 mm and 31 mm. Special coated cassettes with smaller aperture can be obtained on request.

The following picture describes the maximum sample size that can be used with each model of sample changer.

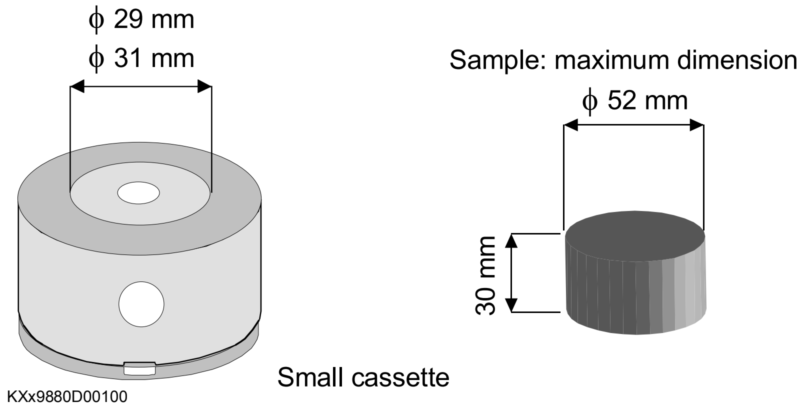


Figure 2.6

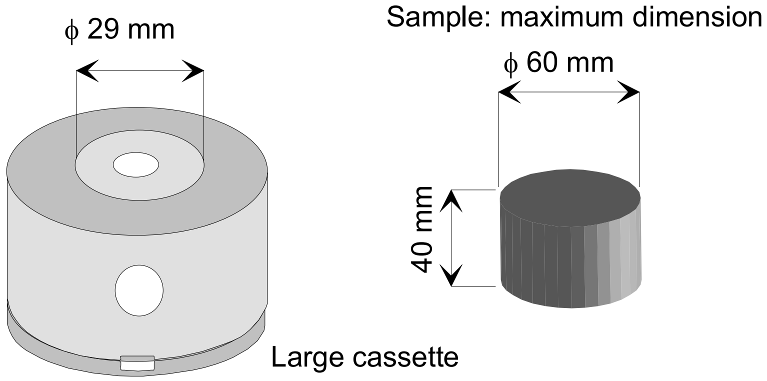


Figure 2.7

The sample is placed and centred in the cassette.

Note: Do NOT touch the surface of a sample! Touching the sample surface will enrich it with Na, Cl and C among other elements, which might affect the analysis.

It is very important to put the cover on and to turn it clockwise until the stopper is reached. Then the cassette can be put onto the sample changer with the sample facing upward.

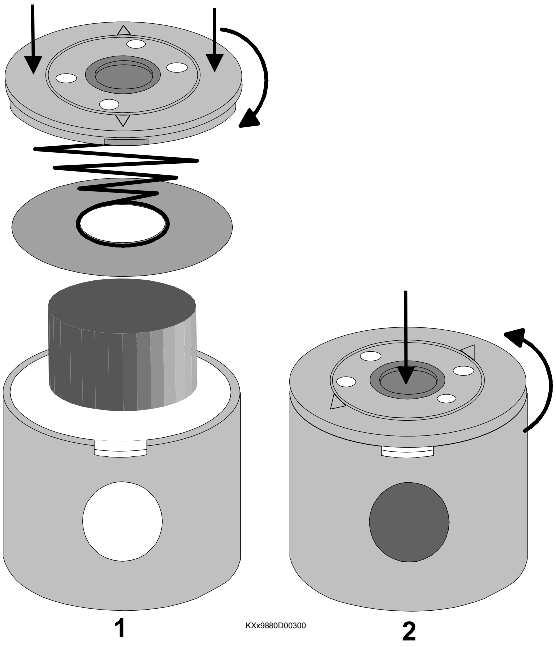


Figure 2.8

1. Sample loading and closing the cassette.
2. Opening the cassette.

To avoid any problems the user has to make sure that the cover of the cassette is well closed.

Centring Rings

In order to facilitate the centring of the samples special rings are available for the cassettes.

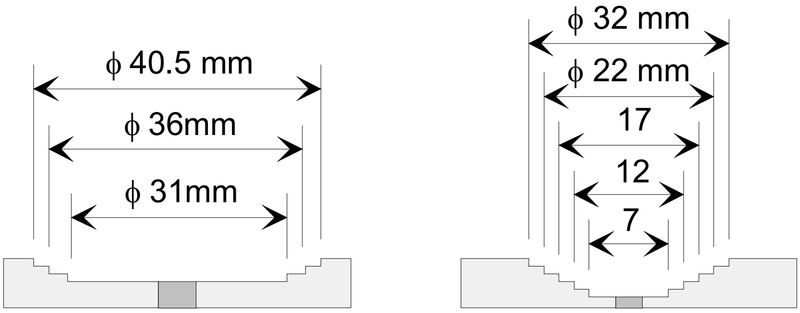
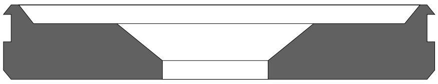


Figure 2.9

These special centring rings can be ordered from your ARL office. To find the part numbers of these rings, please refer to the "*Spare Parts*" Catalogue.

Sample Supports

The following picture describes the sample supports for the X-Y sample changer in the version where no cassette is used. The supports can be chosen in 4 different diameter ranges according to the samples to be loaded.



For sample diameter ranges of:

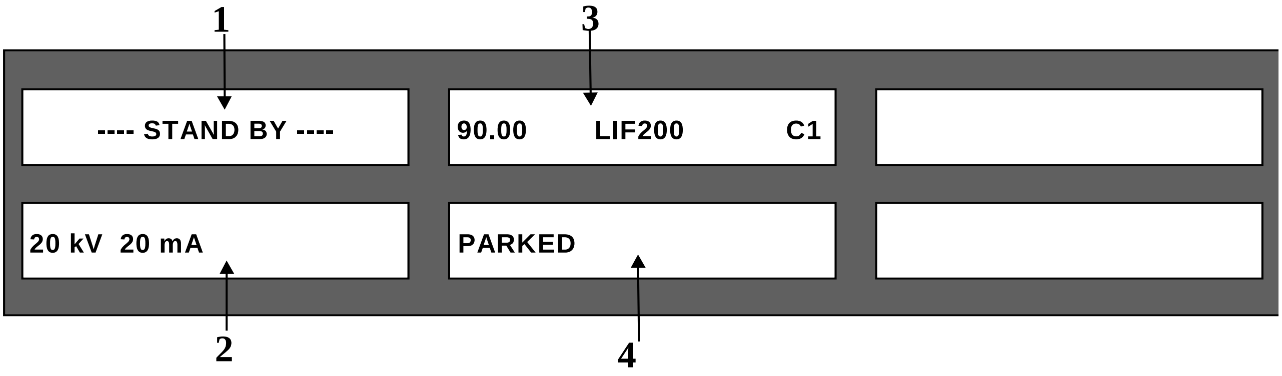
- 30.5 to 32 mm
- 32 to 35 mm
- 35 to 41 mm
- 41 to 50 mm

Figure 2.10

For list of available cassettes, see catalogue.

Instrument Display

The ARL 9800 is equipped with displays. Several lines give messages about the spectrometer status, X-ray tube conditions, monochromators, XRD and goniometer status. Figure 2.11 shows an example of the display and the corresponding status.



1. Spectrometer and monochromator status
2. X-ray tube status
3. Goniometer 1 status
4. Goniometer 2 (XRD) status

Figure 2.11

Spectrometer Status Display

The spectrometer status display shows the general state of the ARL 9800. The following list gives the messages and their meanings. Some messages appear simultaneously or alternately on the display.

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	DESCRIPTION
-	-	-	-	-	-		R	E	S	E	T		-	-	-	-	-	-	-	The spectrometer is being reset or in the power-up sequence
-	-		N	O	T		C	O	N	F	I	G	U	R	E	D		-	-	The spectrometer needs to be configured
C	h	e	c	k		c	o	m	p	a	t	i	b	i	l	i	t	y		Check master firmware to slave firmware compatibility
C	o	m	p	u	t	e		X	M	I	O	s			c	h	k	s	m	Compute monos downloaded code checksum
C	h	e	c	k		X	M	I	s			c	o	m	p	a	t	.		Check monos resident and downloaded code compatibility
D	o	w	n	l	o	a	d		X	M	I	O	s							Download code to all monochromators (XMI nodes)
S	t	a	r	t		X	M	I	O	1		c	o	d	e					Start the downloaded code on monochromator 1
C	h	e	c	k		X	M	I	O	1		r	u	n	n	i	n	g		Check if the downloaded code on mono 1 is running
-	-	-	-	-		S	T	A	N	D		B	Y		-	-	-	-	-	The spectrometer is ready to analyse
P	U	M	P	I	N	G		P	C	:	>	1	0	0	0		P	a		PC only is pumped, PC pressure > 1000 Pa
P	U	M	P	I	N	G		P	C	:	1	2	3	.	4		P	a		PC only is pumped, PC pressure = 123.4 Pa
P	U	M	P	I	N	G		S	P	:	>	2	5		k	P	a			PC and SP are pumped, SP pressure > 25 kPa
P	U	M	P	I	N	G		S	P	:	>	1	0	0	0		P	a		PC and SP are pumped, SP pressure > 1000 Pa
P	U	M	P	I	N	G		S	P	:	1	2	3	.	4		P	a		PC and SP are pumped, SP pressure > 123.4 Pa
P	U	M	P	I	N	G		S	P	:				2	:	3	4			SP is pumped, remaining pumping time is 2 min 34 s
W	A	I	T	:	A	C	O													Process is waiting for analytical conditions (kV/mA/PBD)
W	A	I	T	:			8													Process is waiting for sample 8
									S	E	E	K	:			8				The sample 8 is being sought on the sample magazine

									L	O	A	D	:			8				The sample 8 is being loaded in analysis pos.
									A	N	A	:				8				The sample 8 is in analysis position
									E	V	A	C	:			8				The sample 8 is being unloaded
																		F	2	Primary beam device 2 is in position
																			*	Sample is rotating
M	E	A	S	U	R	E														Instrument is measuring
M	E	A	S	U	R	E		A	1	K	a	_	m				1	2	s	Alt. display for MEASURE on mono Al, remaining time: 12s
F	A	S	T	S	C	A	N													Fast Scan for Goniometer/XRD is in progress
S	T	E	P	S	C	A	N													Incremental Scan for Goniometer/XRD is in progress
P	O	S	C	A	L															Position calibration for Goniometer/XRD is in progress
H	V	P	R	O	F															HV profile for Goniometer/XRD/Mono is in progress
M	g	K	a	_	m				2	0	5	0			2	1	8	.	7	Alternate display for HVPROF on mono Mg
M	g	K	a	_	m							1	0	0	6	4	c	p	s	Alternate display for HVPROF on mono Mg
E	P	R	O	F																Energy Profile for Goniometer/XRD/Mono is in progress
E	P	R	O	F		M	g	K	a	_	m						1	2	s	Alternate display for EPROF on mono Mg

X-ray Tube Status Display

The X-ray tube status display shows the kV and the mA settings of the tube and some other information about the power supply state. The following list gives the messages and their meanings.

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	DESCRIPTION
	0	k	V				0	m	A								O	F	F	X-ray power supply OFF
	0	k	V				0	m	A		W	A	T	E	R	->	O	N		Cooling water is switched ON
	0	k	V				0	m	A		P	O	W	E	R	->	O	N		X-ray power supply is switched ON
	0	->	4	0	k	V				0	->	5	0	m	A					Settings are changed to 40kV and 50mA
4	0	k	V			5	0	m	A			W	A	I	T		T	O	L	Waiting to be in tolerance on settings
4	0	k	V			5	0	m	A											X-ray tube settings are 40KV and 50mA
4	0	->	2	0	k	V			5	0	->	1	0	0	m	A				Settings are changing to 20kV and 100mA
2	0	k	V		1	0	0	m	A			W	A	I	T		T	O	L	Waiting for the settings to be within tolerance
2	0	k	V		1	0	0	m	A											X-ray tube settings are 20kV and 100mA
2	0	->	4	0	k	V		1	0	0	->	2	0	m	A			->	E	Entering economic mode
4	0	k	V			2	0	m	A								E	C	O	X-ray power supply in economic mode
4	0	->	3	0	k	V			2	0	->	8	0	m	A			E	->	Exiting economic mode
3	0	k	V			8	0	m	A			W	A	I	T		T	O	L	Waiting for the settings to be within tolerance
3	0	k	V			8	0	m	A											X-ray tube settings are 30kV and 80mA

Goniometer Status Display

The goniometer status display informs the user about the crystal, detector and collimator selection. It also shows the intensity currently being measured.

Some messages can appear simultaneously or alternately on the display.

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	DESCRIPTION
Z	E	R	O		R	E	Q	U	I	R	E	D								The gonio is waiting for a zero command
R	E	S	E	T		X	G	D	I											The DSP is reset
S	W	I	T	C	H		M	O	T	O	R	S		O	N					The 2 motors are switched on
M	O	V	E		X	+	D		D	O	W	N								The 2 axis are moved for collimator initialis.
I	N	I	T		C	O	L	L	I	M	A	T	O	R						The collimator changer is initialised
I	N	I	T		C	R	I	S	T	A	L									The crystal changer is initialised
X	+	D	:	M	O	V	E		T	O		U	P		L	I	M	I	T	The 2 motors are moved over the
X	+	D	:	M	O	V	E		T	O		L	O	W		L	I	M	.	angular range to find the limits
X	+	D	:	C	A	L	I	B	:	E	N	C	O	D	E	R				The 2 encoders are calibrated in magnitude
M	E	A	S	U	R	E		X	-	D		D	I	S	T	A	N	C	E	The crystal detector distance is measured
X	+	D	:	C	O	A	R	S	E		Z	E	R	O		S	C	A	N	Coarse measure of the 2 zero marks
X	+	D	:	F	I	N	E		Z	E	R	O		S	C	A	N			Fine measure of the 2 zero marks
A	I	K	a	1			F	P	C					C	3					Gonio on Al, detector is FPC, collim. C3
1	4	4	.	7	1		P	E	T					C	3		1	5	s	2θ angle 144.71, crystal PET, remaining
A	l	K	a	1					▨	▨	▨	▨	▨	▨	▨	▨	▨	E	4	counting time 15 s, countrate 90 kcps
4	.	4	4	9	5	^{0}	/	m	i	n						5	:	2	3	A fast scan is performed at 4.45 deg/min,
1	2	2	.	4	5		F	P	C					C	1	5	:	2	3	remaining measuring time is 5min23s,
1	2	2	.	4	5		L	i	F	2	0	0		C	1	5	:	2	3	detector FPC, crystal LiF200, collim C1
1	2	2	.	4	5									▨	▨	▨	▨	E	3	actual 2θ angle is 122.45, intensity 4kcps
H	V	:	1	6	5	0			A	G	C	:			1	2	3	.	4	A HV profile is performed

XRD (Diffraction System) Status Display

The XRD status display informs the user about the angle and element selection. It also shows the intensity currently being measured.

Some messages can appear simultaneously or alternately on the display.

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	DESCRIPTION
Z	E	R	O		R	E	Q	U	I	R	E	D								The XRD is waiting for a zero command
R	E	S	E	T		X	D	D	D											The DSP is reset
S	W	I	T	C	H		M	O	T	O	R		O	N						The motor is switched on
M	O	V	E		T	O		U	P		L	I	M	I	T					The motor is moved over the
M	O	V	E		T	O		L	O	W		L	I	M	I	T				angular range to find the limits
C	A	L	I	B	R	A	T	E		E	N	C	O	D	E	R				The encoder is calibrated in magnitude
C	O	A	R	S	E		Z	E	R	O		S	C	A	N					The zero mark is coarse measured
F	I	N	E		Z	E	R	O		S	C	A	N							The zero mark is fine measured
P	a	r	k	e	d		-	5		d	e	g								The XRD detection motor is switched OFF,
																				the primary beam collimator -5 deg. is ON
Z	n	O	_	A			-	5		d	e	g								The XRD detection is on phase ZnO_A
Z	n	O	_	C													1	5	s	Remaining measuring time is 15s on phase
1	1	4	.	3	3					▨	▨	▨	▨	▨	▨	▨	▨	E	3	ZnO_C at 114.33 deg. and intensity of 8cps
4	.	4	4	9	5	^{0}	/	m	i	n						5	:	2	3	A fast scan is performed at 4.45 deg/min,
1	2	2	.	4	5									▨	▨	▨	▨	E	3	remaining measuring time is 5min23s, the
																				actual angle is 122.45, and intensity is 4kcps

Inside the ARL 9800

Removal of the Panels

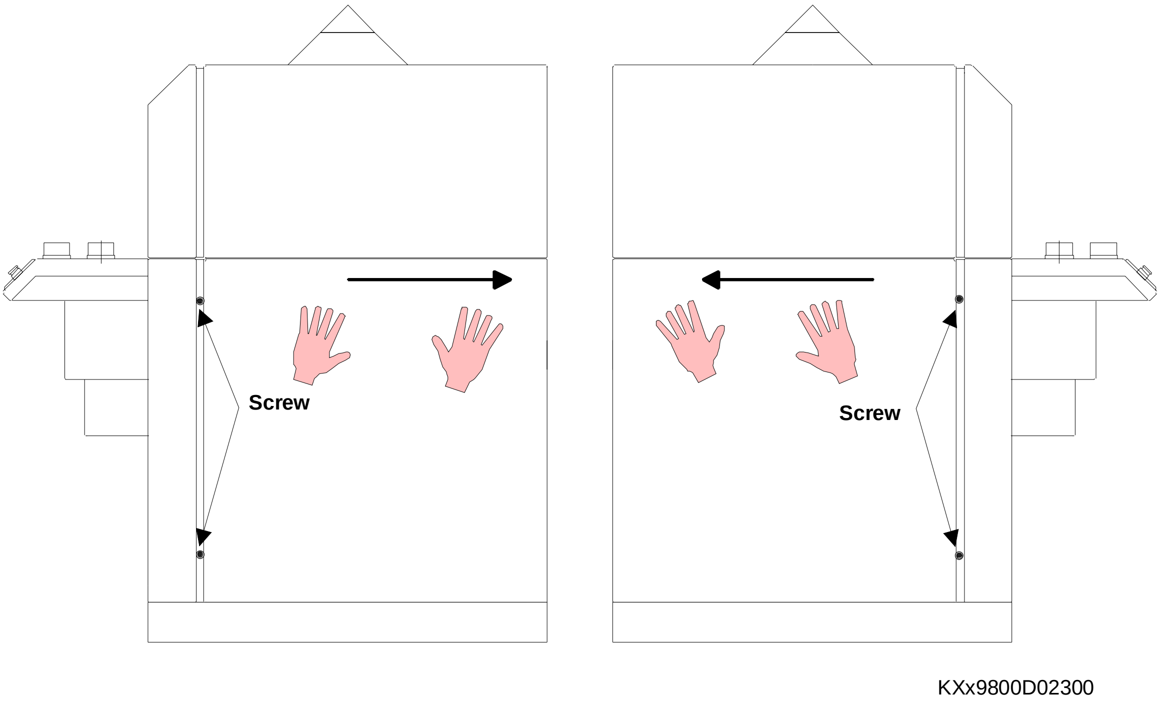


Figure 2.12

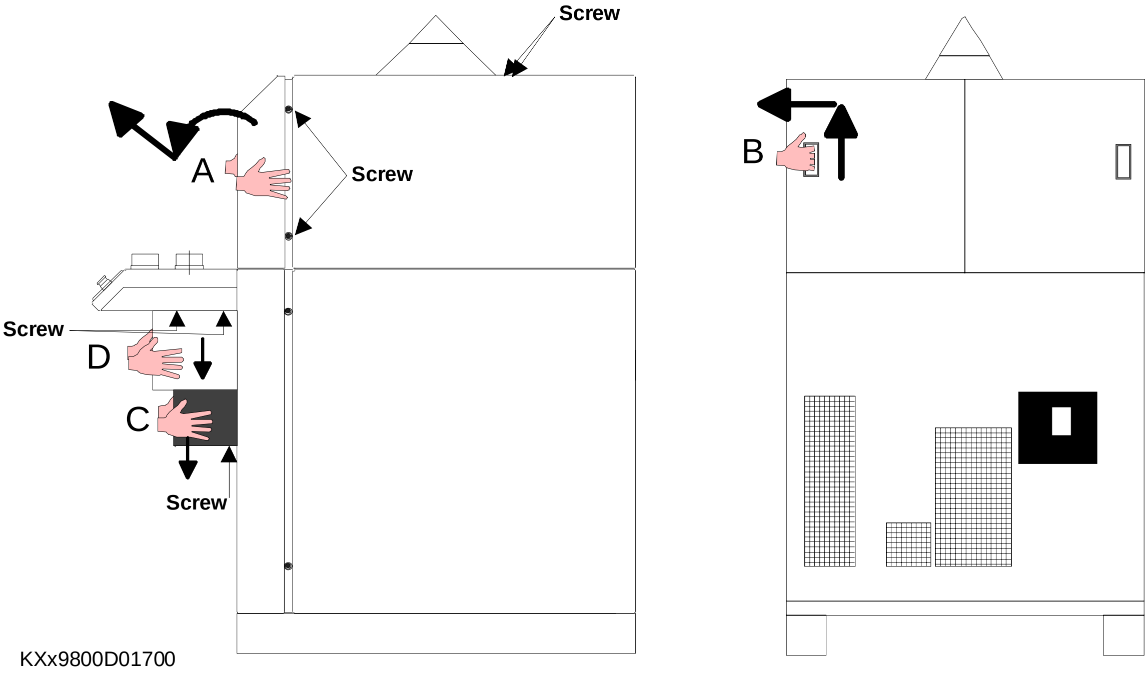


Figure 2.13

Location of Consumable Parts

All usual consumables are located at the right of the instrument. By removing the right panel the consumable parts can be accessed. This design simplifies the maintenance to be carried out by the user.

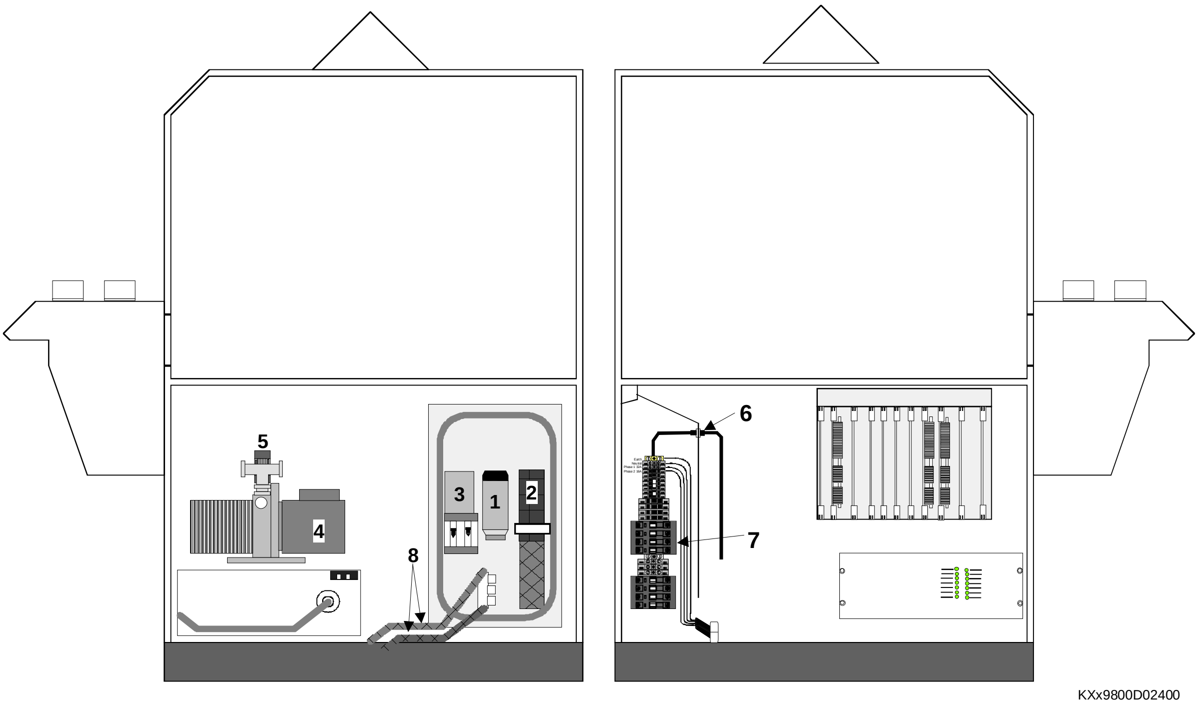


Figure 2.14 ARL 9800 XP

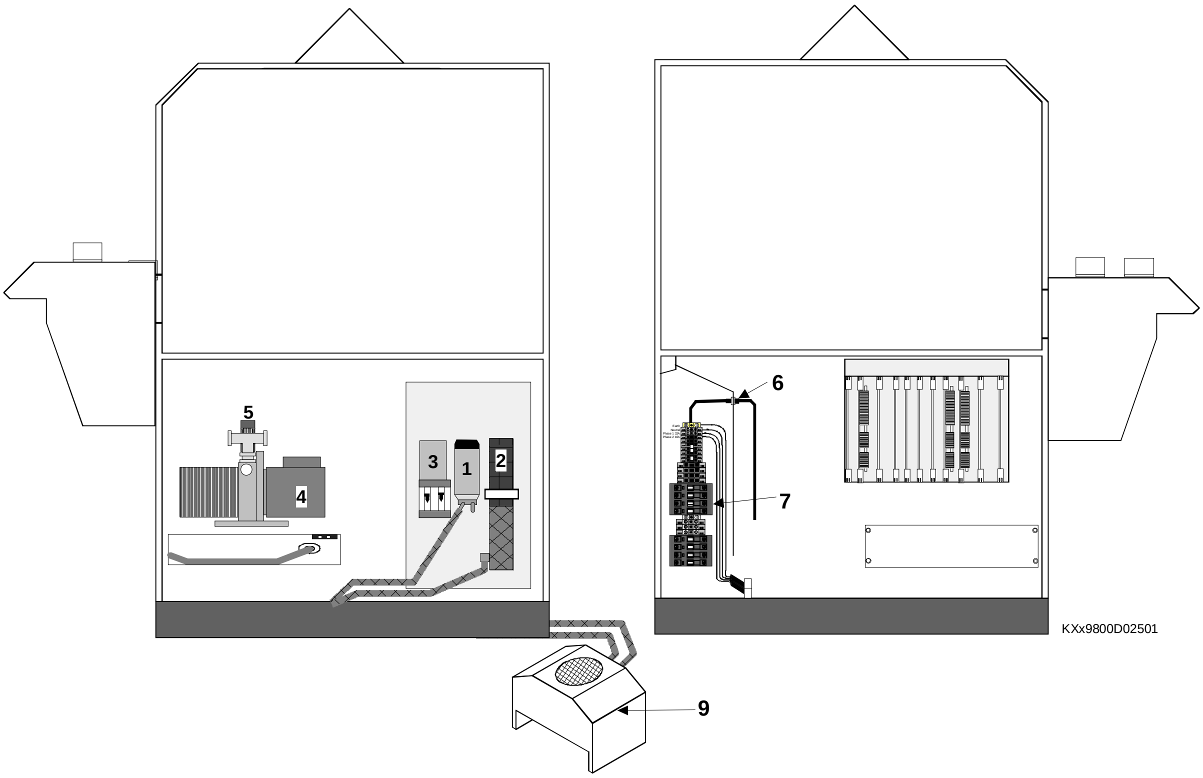


Figure 2.15 ARL 9800 OASIS

1. Water pump
2. Deionizing cartridge
3. Deionized water tank
4. Vacuum pump with oil
5. Oil vapour filter
6. Gas connection
7. Main connection
8. Water connection
9. Deionized water air exchanger

Description of the General Parts

This section describes the features and devices located inside the ARL 9800. Some devices are optional and may therefore not be fitted on your instrument.

When the panels of the instrument are removed the different assemblies which can be divided into five main functions (see Figure 2.16).

Spectrometer tank with analytical devices

The spectrometer tank is the main component responsible for maintaining a vacuum and a thermally controlled environment for analysis, for protecting the user against radiation and for positioning all the analytical devices around the sample, namely:

Monochromators, Goniometers, Diffraction System (XRD) and Primary Beam Devices (PBD).

Sample introduction

Different sample magazines allow unattended operation, prepare the sample for introduction into the Primary Chamber and finally move it into the analytical position where sample rotation is possible.

Analytical environment

Environment (vacuum or air) in the Spectrometer tank, thermal regulation of the instrument and the analytical devices, gas regulation for the detectors.

Sample excitation

X-ray tube, X-ray tube power supply, X-ray tube cooling system.

Electronic devices

Electronic rack and boards, main power connections, power distribution for the main and for low voltage.

The next figure shows the general parts of the ARL 9800.

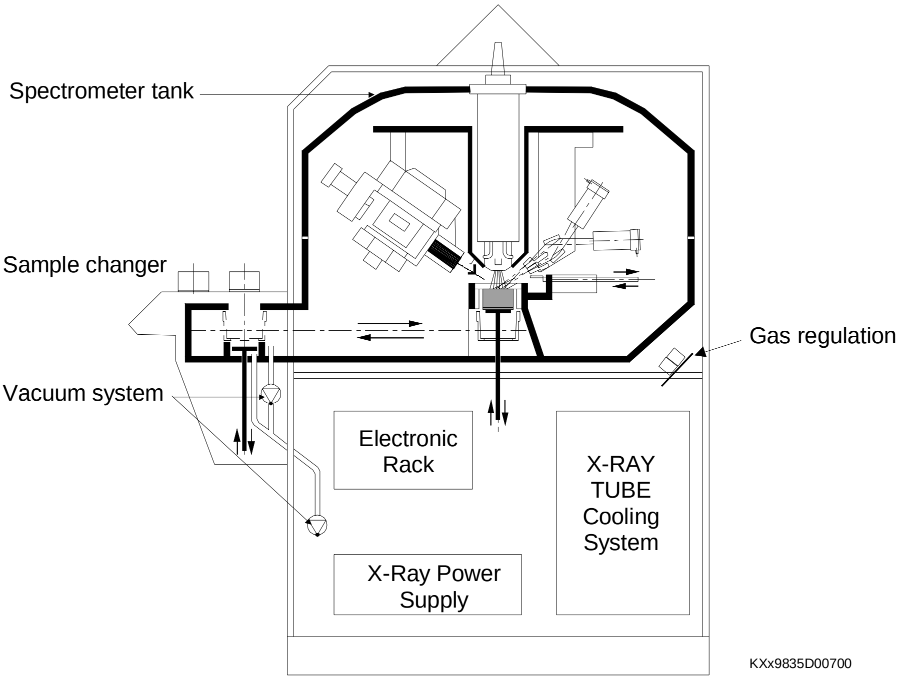


Figure 2.16

Inside the Spectrometer

The analytical devices inside the spectrometer are shown in the next two figures. All these analytical devices are options and the choice for the configuration depends on the analytical application.

- X-ray tube. The controlled X-ray source to excite the sample.
- Monochromators. The simultaneous fixed channels. Each monochromator allows the measurement of one element.
- Goniometer for sequential measurement. The ARL 9800 can be equipped with 2 goniometers. Each of them can measure sequentially up to 83 elements of the periodic table (if appropriate crystals are mounted).
- Diffraction system (XRD) for determination of specific phases. The ARL 9800 can be equipped with one XRD system. The XRD system uses primary beam collimators driven by the PBD.
- Sample. Figure 2.17 shows the sample in the analysis position.
- Primary Beam Device. The PBD allows various Primary Beam Filters for the XRF analysis and Primary Beam Collimators for the Diffraction System to be used depending on the analytical program (PBD must be installed when XRD is mounted).

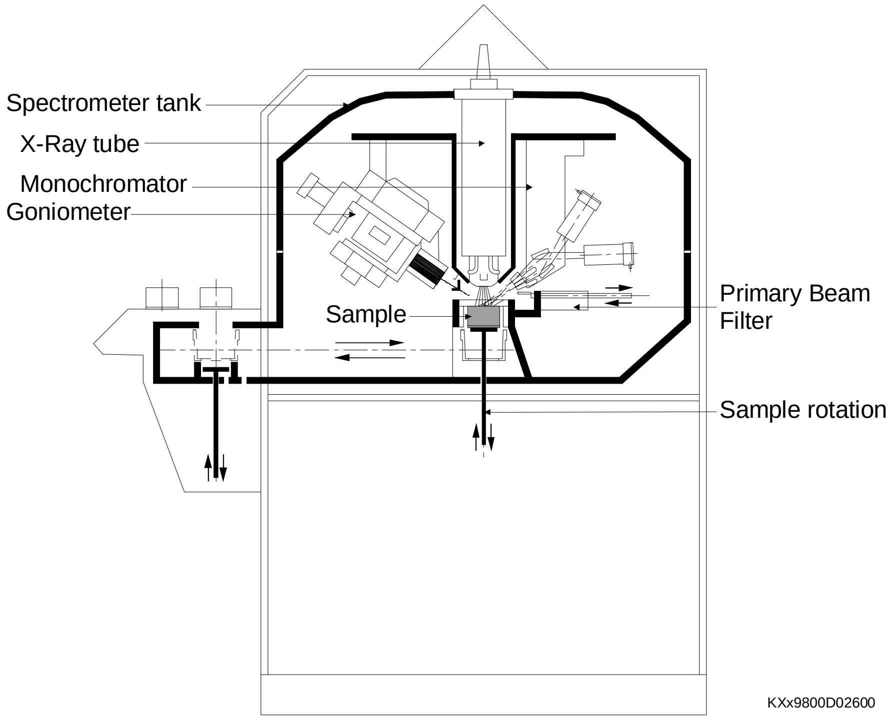


Figure 2.17

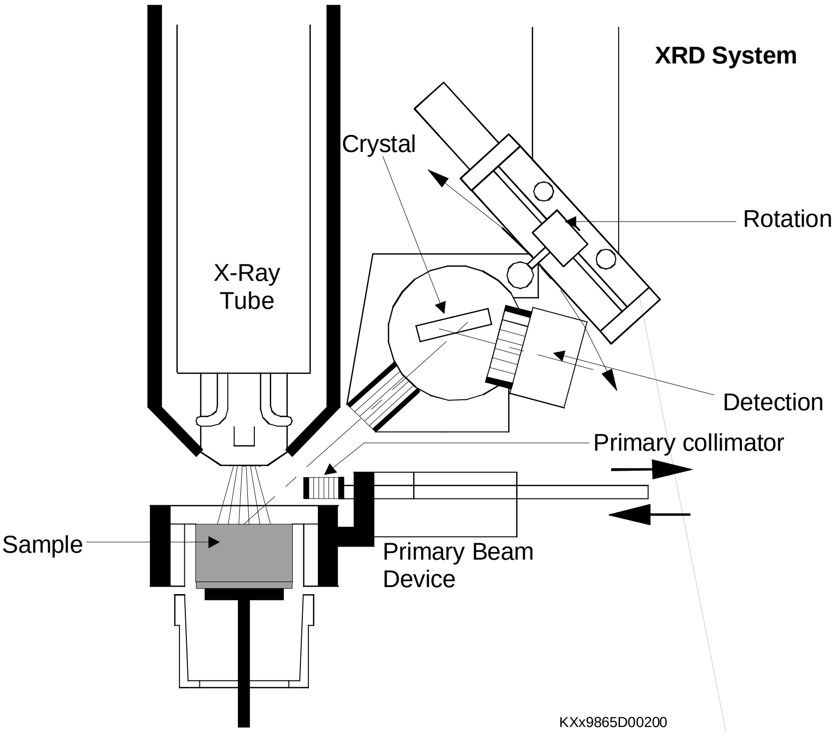


Figure 2.18

The next table shows typical configurations of the instrument.

Number of Goniometers	Number of Monochromators XP/Oasis	XRD System	Instrument Type
0	32/20	NO	Simultaneous
0	24/14	YES	Simultaneous
1	0	NO	Sequential
1	0	Yes	Sim / Seq
1	24/14	NO	Sim / Seq
1	14/8	YES	Sim / Seq
2	16/8	NO	Sim / Seq
2	6/0	YES	Sim / Seq

Monochromator Description

The monochromators are fixed assemblies of three parts satisfying Bragg's law ($n\lambda = 2d \sin\theta$), a crystal, a detector and a focalisation or collimation system. There are two distinct types of monochromators:

- Curved Crystal Monochromators
- Flat Crystal Monochromators

The table below shows the monochromators available for the ARL 9800 instrument.

Figure 2.19

Curved Crystal Monochromators

Crystals such as LiF, ADP or PET allow for the utilisation of curved crystal focussing systems of great precision and resolution. These crystals ensure excellent analytical results for elements from sodium (atomic number 11) upwards.

The crystals used by ARL are curved according to a perfectly focussing trajectory and aligned on a very narrow primary slit thus ensuring very good spectral resolution. The choice of crystal is made according to its reflection capacities of the wavelength of the element to be analysed.

Various types of gas detectors or scintillation counters are used according to their measurement efficiency for the wavelength of the element to be analysed.

In most cases, two monochromators can be assembled in pairs and thus each monochromator assembly allows the analysis of 2 different elements.

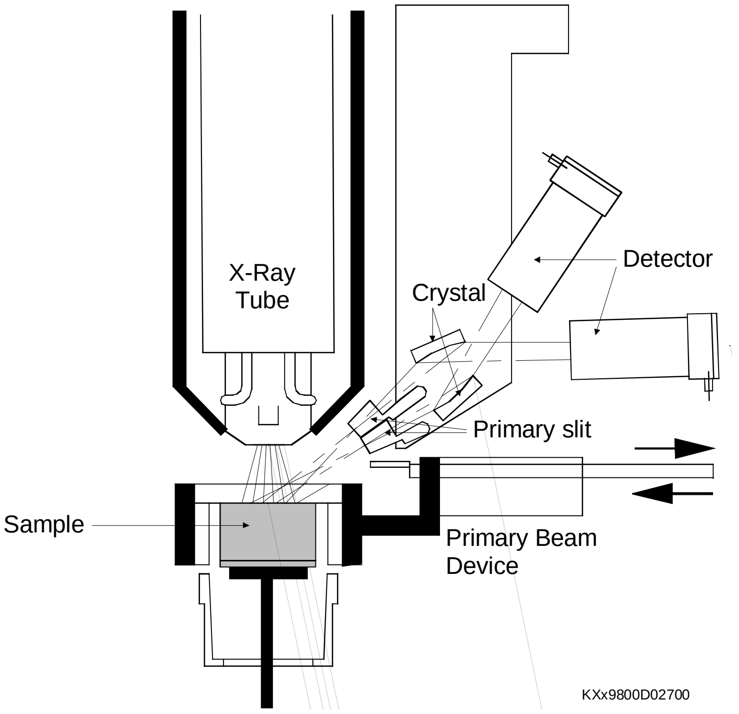


Figure 2.20

Note: Special monochromators were designed specifically for Na and Mg with curved multilayer and thin Be window sealed detectors to allow the instrument to run without P10 gas supply for specific applications.

Flat Crystal Monochromators

The multilayers are very efficient reflectors for soft X-rays and permit excellent analytical results for the light elements from Boron to Magnesium. The physical characteristics of multilayers favour the use of an arrangement of flat crystals equipped with collimators and gas flow detectors (Ar/CH4) with extremely thin windows which are transparent to soft X-rays.

Each flat crystal monochromator is equipped with an elliptical mask which narrows the solid angle to the cassette opening and thus prevents any spectral contaminations from the cassette bottom superimposing on the radiation from the sample.

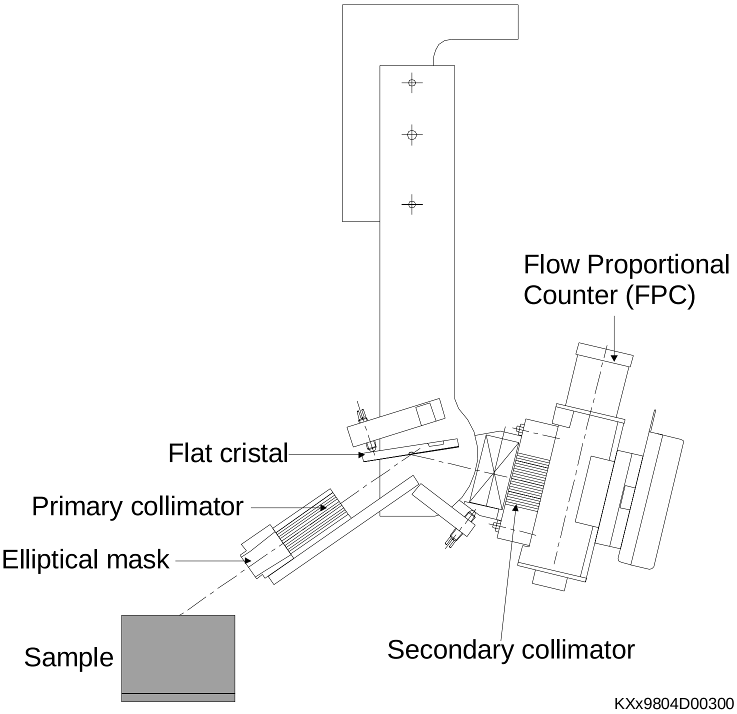


Figure 2.21

Goniometer Description

The goniometer is the heart of the ARL 9800 and its gearless design allows a fast and accurate positioning of the crystals and the detectors. The two encoder systems ensure fully independent positioning for both the crystal and the detector.

To measure the intensity of an element the user defines a line or the parameters of a scan in order to position the goniometer (2θ angle) with the selected parameters (crystal, detector and collimator).

The goniometer has three parts:

Detector System

Two detectors are fitted: the Flow Proportional Counter (FPC) used for light elements (B to Fe) and for L and M lines, and the Scintillation Counter (SC) used for medium and heavy elements (Fe to U).

Crystal Changer

This device can support up to nine crystals.

Collimator Changer

This device can support up to four collimators.

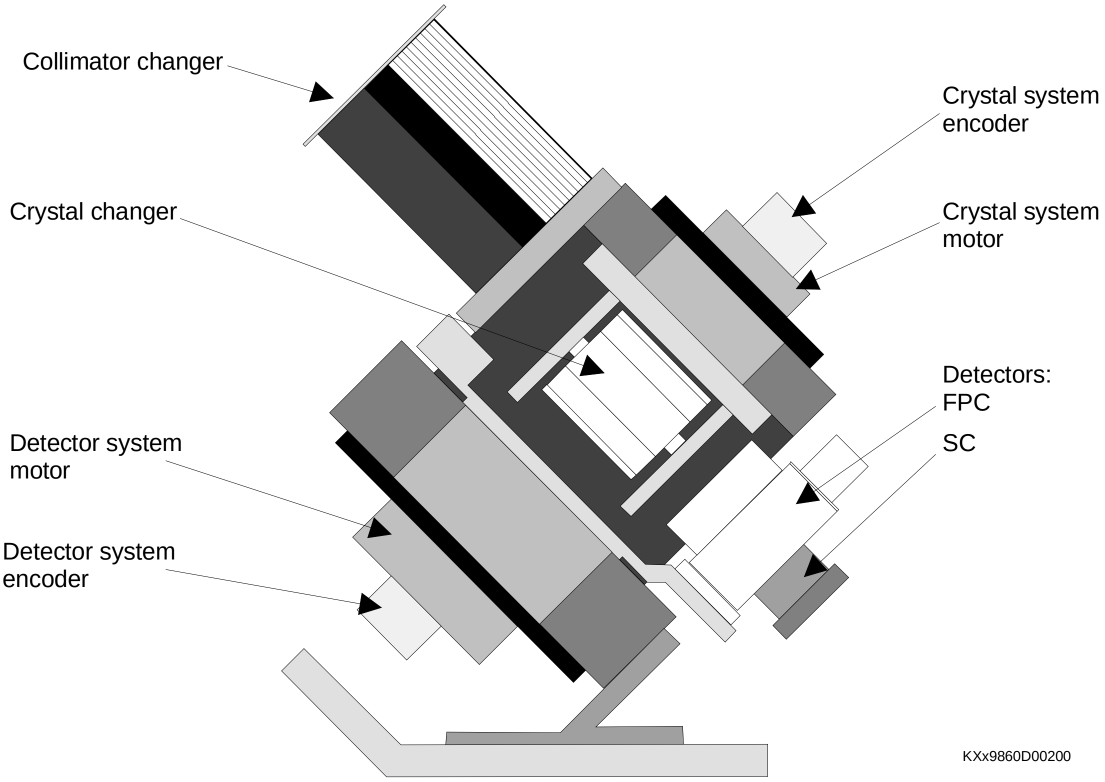


Figure 2.22

Crystal Selection

Crystals with fixed inter-atomic distance (d) are used to disperse the wavelength of the elements to be measured at a corresponding reflection angle of the crystal according to Bragg’s Law (position θ of the crystal and 2θ of the detector on the goniometer).

The following table shows the crystal choice regarding the different element lines. It should be noticed that a maximum of 9 crystals are (or can be) mounted. If there are free positions available additional crystals can be ordered at any time.

K Lines L Lines	Sensitivity	Resolution	Be	B	C	N	O	F	Na	Mg	Al	Si	P	S	Cl	K	Ca..Fe Sn..Yb	Ni..Sn Hf..U
AX20	Good	Low	●◀	▶●														
AX16	High	Low			●													
AX09	Good	Low			◀	▶●												
AX06	High	Low					●◀	●	●	▶●								
TLAP	Good	Good					●◀	●	●	▶●	—	▶						
ADP	Low	High								●								
PET	Good	High									●◀	●	—	—	▶			
InSb	High	High										●						
Ge	High	High											●◀	●	●	▶	▶	
LiF 200	Good	Good														●◀	●	▶●
LiF 220	Fair	High															Ti◀	▶●
LiF 420	Low	Very high															Ni◀	▶●

Collimator Selection

The collimator changer can have up to four collimators (0.15°, 0.25°, 0.60°, 2.60°). The 0.25° and the 0.6° are fitted as standard and the 0.15° and 2.60° are available as options.

K Lines L Lines	Sensitivity	Resolution	Be	B	C	N	O	F	Na	Mg	Al	Si	P	S	Cl	K	Ca..Sn Sn..U
Coarse 2.6°	Very high	Low	●◀	●	●	▶●											
Medium 0.6°	High	Fair				●	●	●	●	●	●	●	●	●	●	●	
Fine 0.25°	Good	Good						●	●	●	●	●	●	●	●	●	●
Fine 0.15° (LiF220, LiF420)	Low	High									◀	—	—	—	—	—	▶●

The following table shows the relation between the different names of the collimators within the WinXRF software and the instrument display:

Name on the display	Resolution	Name
C1	0.25°	Fine
C2	2.60°	Coarse
C3	0.60°	Medium
C4	0.15°	Fine (LiF220, LiF420)

Diffraction System (XRD)

The XRD system is a combination of three different components used to measure the structure of the crystalline phases present in the sample:

- The detection device is a flat monochromator able to select one of the specific wavelengths of the X-ray tube spectrum. It is made up of a Soller collimator, a flat graphite crystal and a sealed Krypton detector.
- The primary beam collimators are used to irradiate the sample with a parallel beam of photons that has a well defined incident angle. These collimators are fitted on the PBD system and placed between the X-ray tube and the sample.
- The scanning screw rotates the whole assembly in an arc around the sample (where the sample corresponds to the centre) in order to detect a specific phase or to perform a scan.

The fixed wavelength measured by the system is modulated by the crystalline structures of the sample and thus can be used for qualitative and quantitative measurements of specific phases present in the sample.

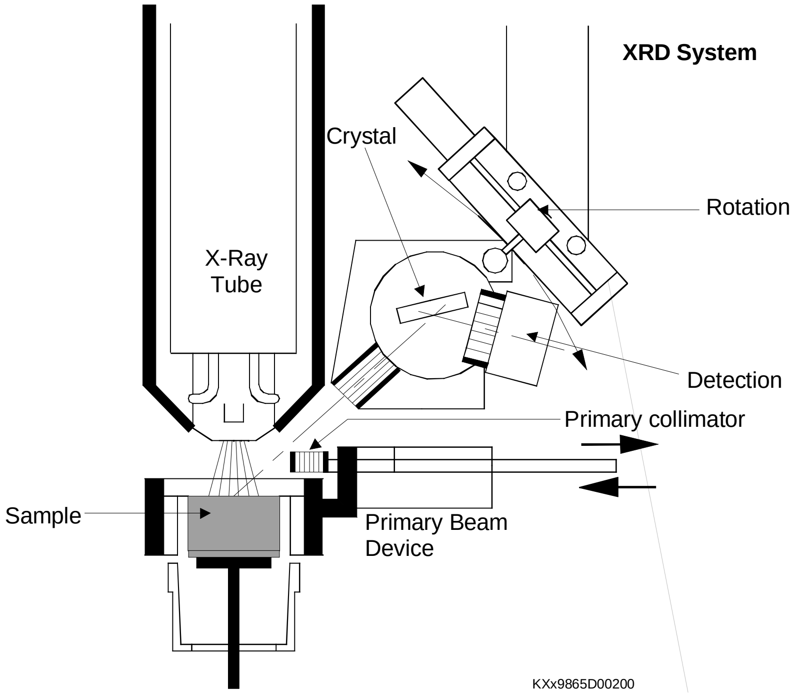


Figure 2.23

Two primary beam collimators are mounted. These are used to collimate the incident beam at two different incidence angles of 20° and -5°. This allows to cover a wide 2d range with the XRD system.

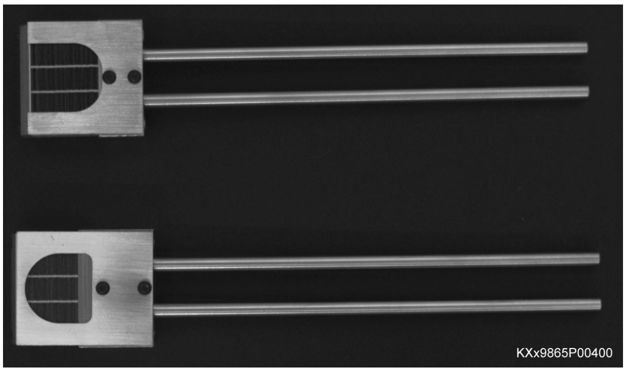


Figure 2.24

Primary Beam Device

The Primary Beam Device (PBD) is the mechanical changer for Primary Beam Filters and Collimators.

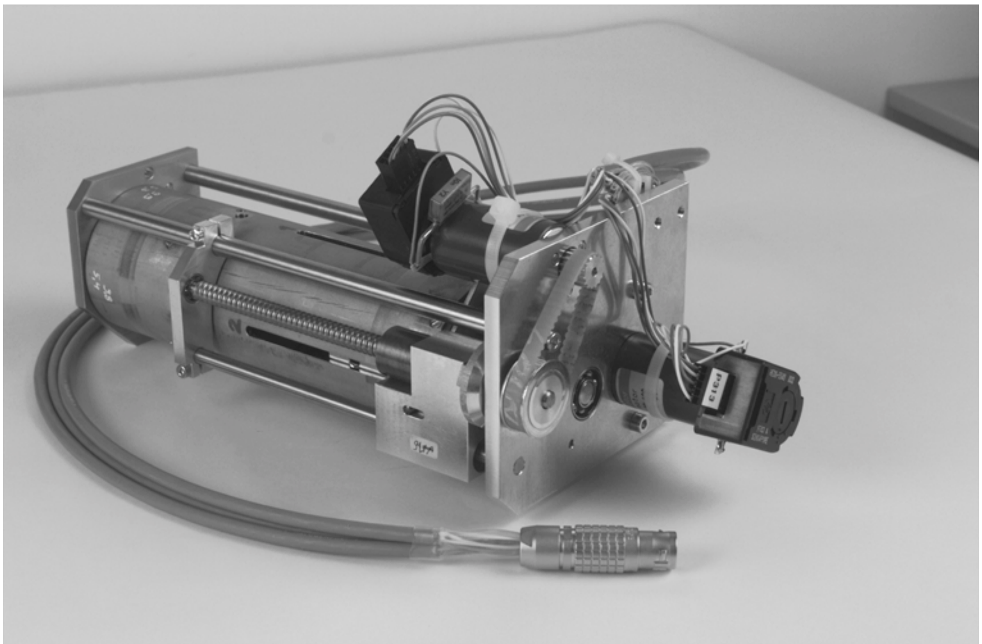


Figure 2.25

This changer can handle 4 devices, i.e. filters or collimators. Only two spaces are available for filters when the XRD system is installed.

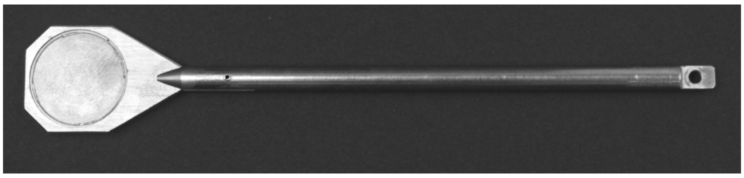


Figure 2.26

Filter type	Name on the display	Use
Cu 0.25mm	F1	For the analysis of Ru, Rh, Pd, Ag and Cd (elements that are interfered by Rh lines from the X-ray tube).
Fe 0.01mm	F2	To improve the peak to background ratio of NiKα and CuKα lines.
Al 0.5mm	F3	To improve the peak to background ratio of PbLα, PbLß and AsKα, AsKß lines in light matrices.

Sample Handling System

The sample handling is undertaken by two main devices:

- **The sample changer** or **loader** used to bring the chosen cassette with the sample or the sample by itself onto the loading position.
- **The sample introduction system** is the combination of different components operating between the two environments, vis. the atmospheric pressure of the lab and the vacuum environment inside the spectrometer, and which ultimately brings the cassette or the sample by itself into the analytical position.

Sample Loading

Three sample loading systems are available:

- Single position loading.
- 12 Position Sample Changer.
- X-Y sample changer.

The sample changers move the samples automatically onto the loading position.

For complete unattended operation including sample preparation, the sample handling is performed by:

- The SMS 800 fully automated system.

Single Position Loading

Figure 2.27

12 Position Sample Changer

The 12 Position Sample Changer is part of the basic configuration of the instrument. The sample changer brings the cassette onto the loading position. The sample changer has 12 encoded positions and can move in both directions. The cassettes which are all identical are not coded. The coding for addressing the desired cassette is determined by the numbered position occupied by the cassette on the sample loader. This position is entered as the cassette number in the WinXRF Software.

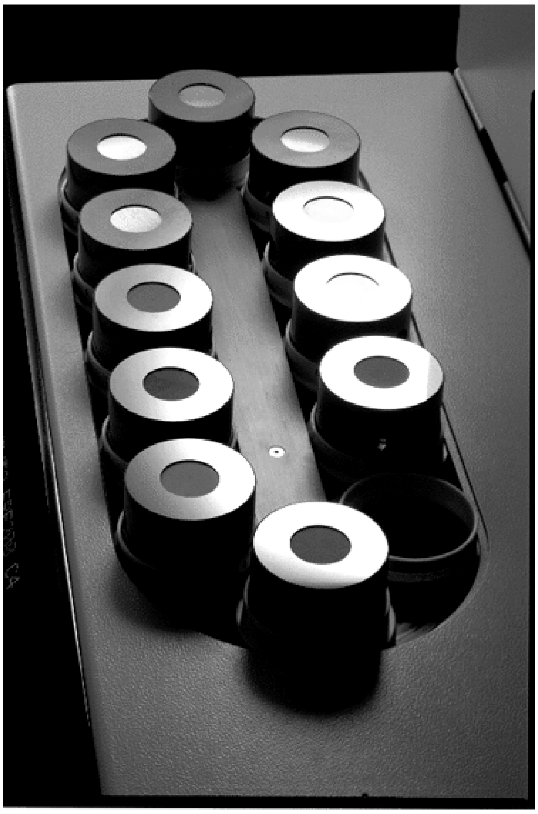


Figure 2.28

X-Y Sample Changer

This sample loader is a large capacity sample changer available as an option in various configurations. The identification of the sample to be loaded is done by the position of the same on the tray. It can either load up to:

- 98 cassettes
- 98 samples

Figure 2.29

In this latter configuration, the X-Y sample changer is able to load samples directly into the ARL 9800 using a special sample support system. The X-Y sample changer is designed to have two trays and accepts the following configurations:

- Two trays for samples in conventional cassettes.
- Two trays for samples on support without cassettes.

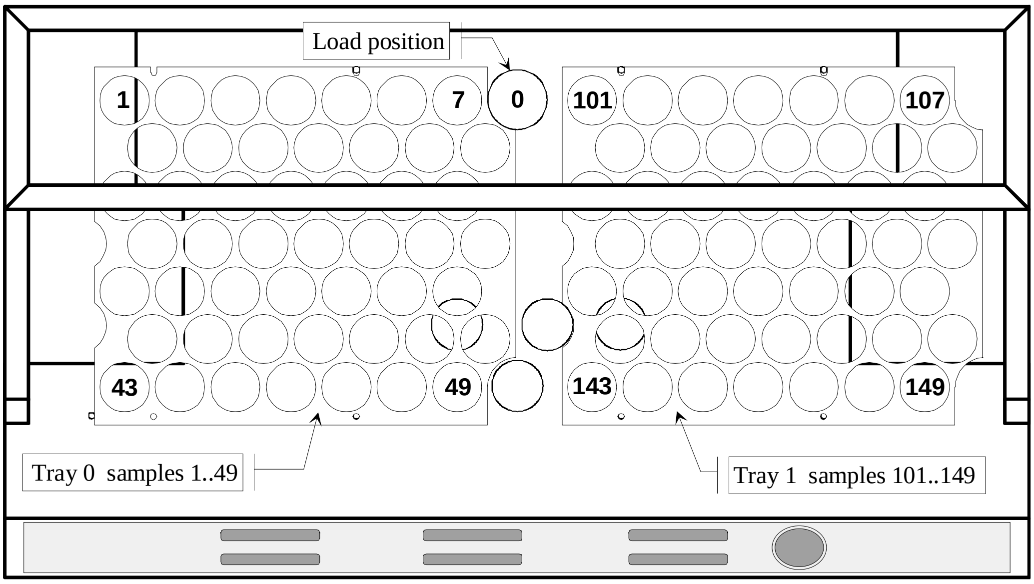
X-Y Sample Changer for Cassettes

The sample is put into a conventional cassette and put on a tray of the X-Y sample changer. The position numbers range is from 1 to 49 for the left tray and from 101 to 149 for the right tray. Figure 2.30 shows the X-Y sample changer equipped with trays for cassettes only.

Figure 2.30

X-Y Sample Changer for Samples without Cassettes

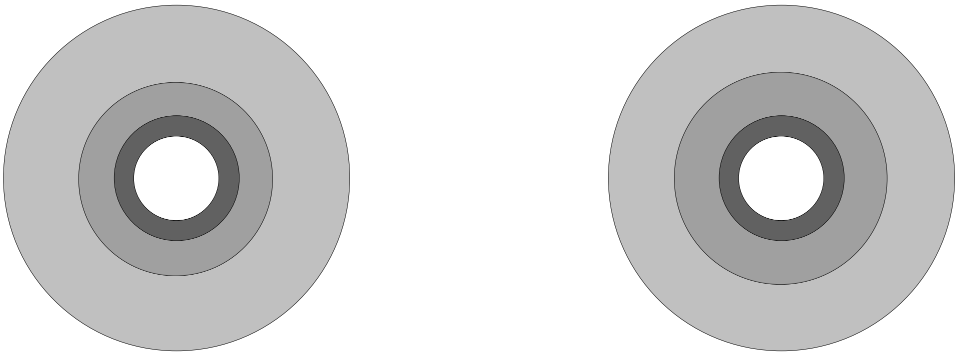
The samples are put onto the sample supports that are delivered with the X-Y sample changer and then on the tray of the X-Y sample changer. The samples are handled and loaded into the instrument with the supports.



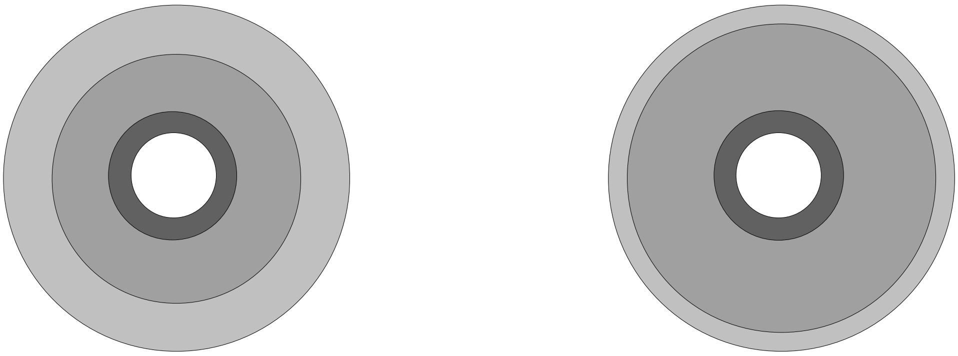
KXx9871D00501

Figure 2.31

Depending on the sample size, different supports are available, as this is shown in the next figure.



Sample Diameter from 30.5 to 32 mm **Sample Diameter from 32 to 35.5 mm**



Sample Diameter from 35 to 41 mm **Sample Diameter from 41 to 52 mm**

Figure 2.32

The next figure shows the X-Y Sample Changer with several types of samples.

Figure 2.33

X-Y Sample Changer for Simple Automation

A simple automation applications can be achieved using the X-Y sample changer adapted for belts and using the so called OEM protocol (see software documentation) to drive the instrument from a host computer.

Half of the surface (one tray on the right or on the left side) is dedicated for Setting-up Samples or manual samples without cassettes. On the other half of the surface, left or right depending on the configuration of the lab, space is available for one or two belts with the associated waiting positions, as well as for up to three trash basket positions (see Figures 2.34 and 2.35).

For production samples coming from the belt, the unloading position can be defined as follows:

- After analysis, the sample goes back to the same belt for evacuation before the next sample arrives.
- After analysis, the sample is placed on a waiting position until the next sample is loaded.
- After analysis, the sample is placed onto a second belt for evacuation.
- After analysis, the sample is discarded in a trash basket.

For samples placed manually on the tray, the unloading position can be defined as follows:

- After analysis, the sample goes back to its previous position.
- After analysis, the sample is discarded in a trash basket.

Setting-up Samples always go back to their original positions.

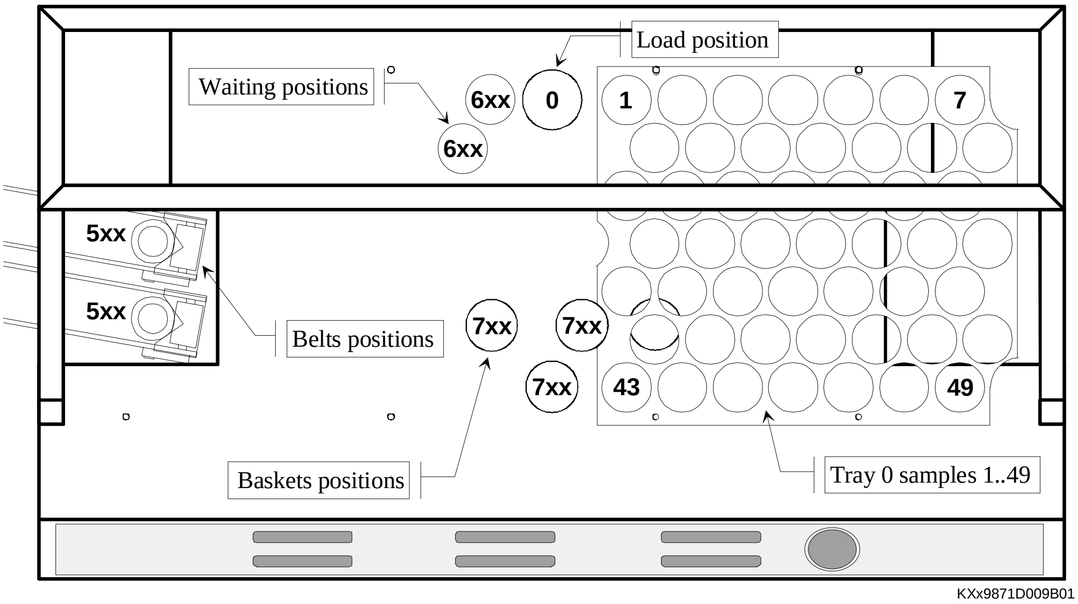


Figure 2.34

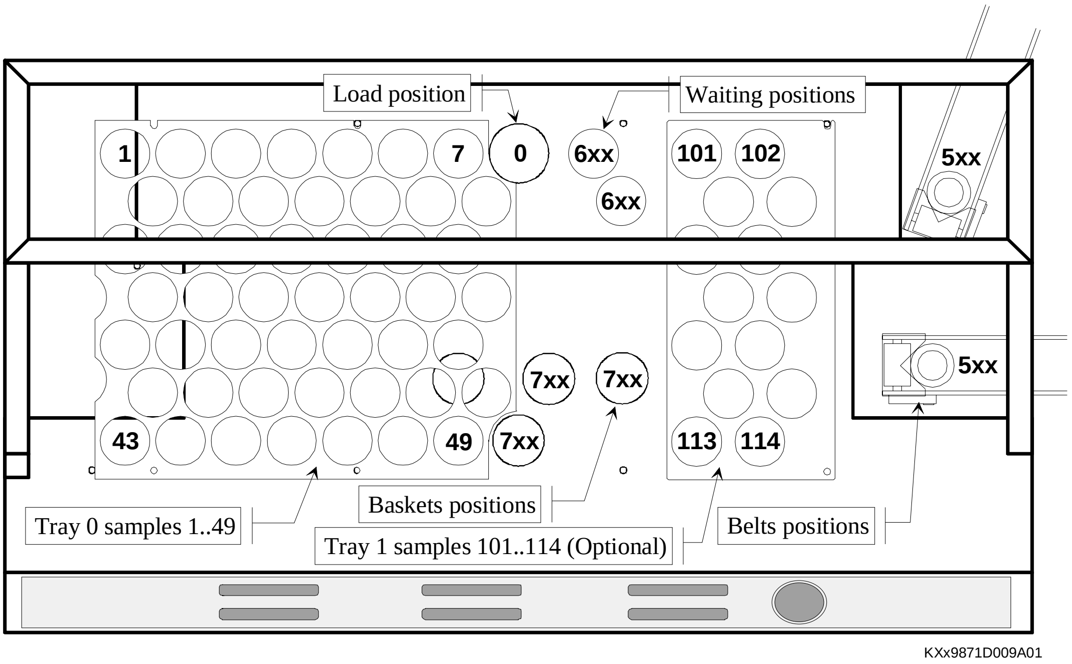


Figure 2.35

Note:	The maximum height for the samples used with the trays is 25 mm and the weight should not exceed 200 g.

X-Y Sample Changer Operation

The X-Y sample changer can work only when the covers are closed and it is not recommended to open them during movements. Nevertheless, it is possible to open the covers to introduce or to remove a sample.

SMS-800 System: State-of-the-Art Automation

This system provides powerful sample handling capabilities by a robot.

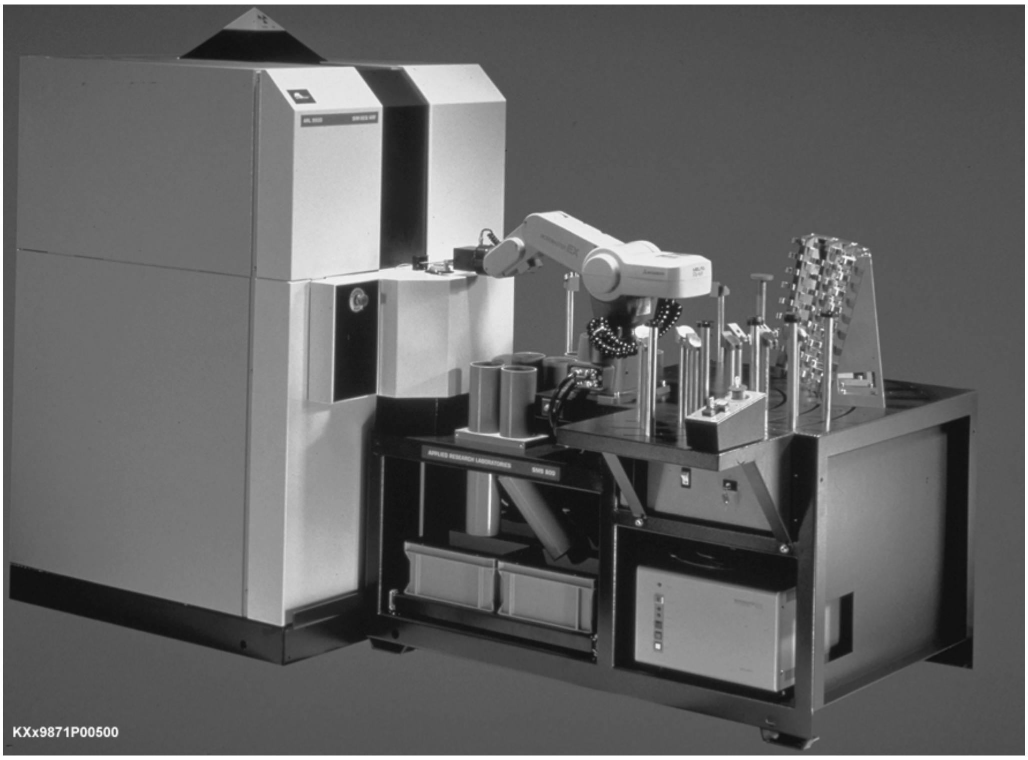


Figure 2.36

The figure below shows the main sample loading components.

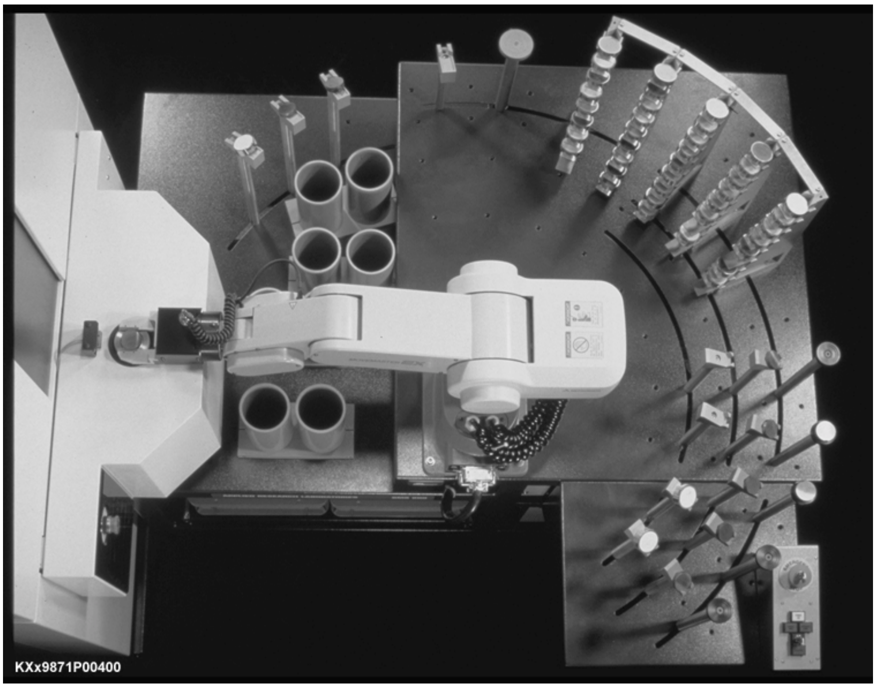


Figure 2.37

Analysis Environment

Spectrometer Environment

There are two possible environments:

♦ Vacuum	This is the environment used for the measurement of all samples.
♦ Atmosphere	This mode ventilates the spectrometer tank and is only used for maintenance.

The vacuum system utilizes a molecular pump to evacuate the spectrometer and to keep a clean and stable environment for the sample and the detection devices. In addition to this, the molecular pump is more efficient at low pressure and this helps to speed up the pumping procedure.

The molecular pump needs a primary vacuum of 1000 Pa to work properly. This primary vacuum is achieved with a vane pump. Since the volume of the primary chamber is really small compared to the tank (ratio of 1000), the pumping time can be greatly reduced and achieved with the same vane pump.

During this phase the instrument is pumped down. The pumping procedure is made up of a certain number of pumping phases. The process is started by opening the shell on the primary chamber and subsequent pumping through the primary chamber pumping valve with the vane pump. The more the vacuum pressure continues to decrease, the more the molecular vacuum pump speed increases from 12000 rpm to 27000 rpm to reach a normal functioning vacuum pressure below 8 Pa.

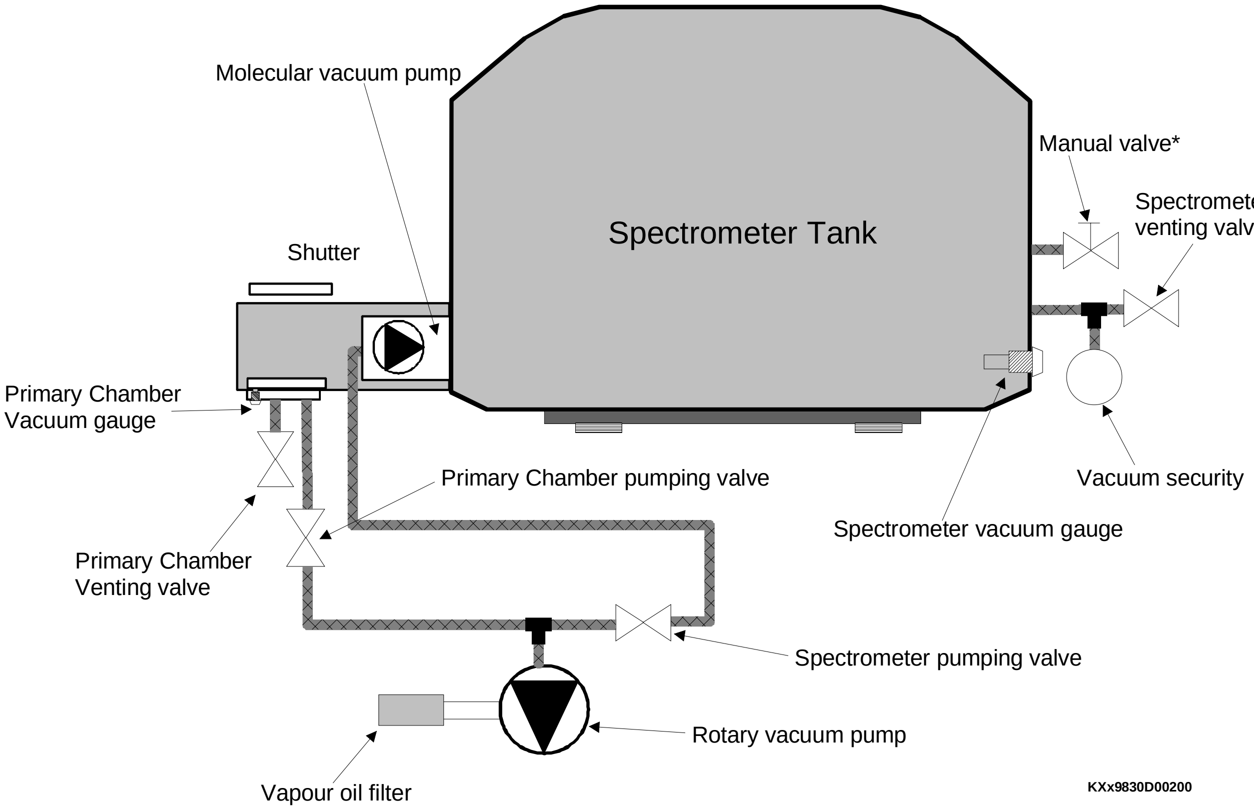


Figure 2.38

Note: The manual valve on the ARL 9800 should only be opened by trained and qualified personnel.

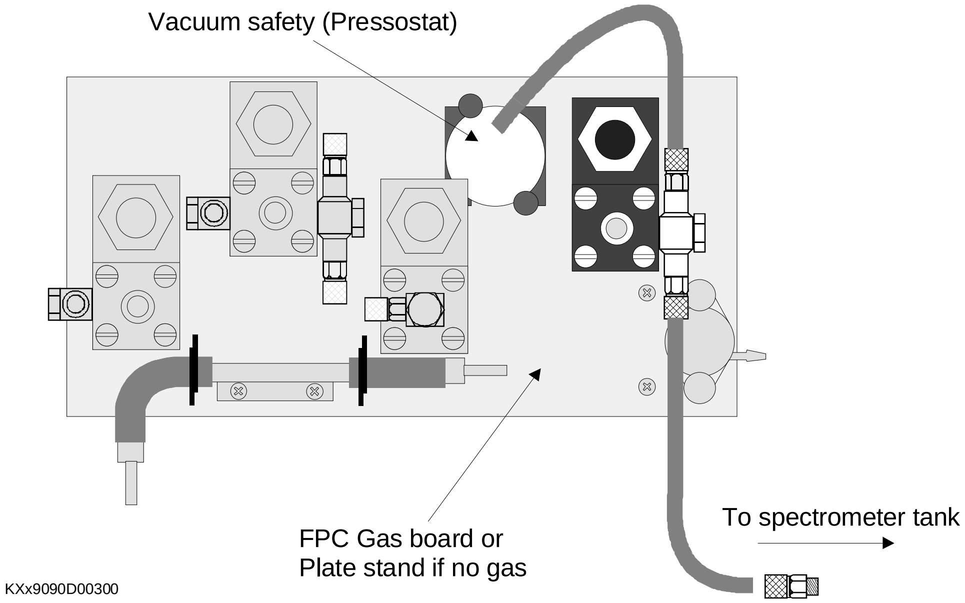


Figure 2.39

The vacuum security is present to prevent the analysis of a sample under air. This security is connected directly to the X-ray generator, and will also switch off the X-ray tube if the vacuum is lost.

WARNING :

Never by-pass security that is installed in the ARL 9800 instrument.
ARL will not be held responsible for any damage if the security is by-passed.

Gas Regulation (Optional)

The ARL 9800 requires one type of gas: Argon-Methane (P10 gas) for the flow proportional counter (FPC). This detector is installed on the goniometer and on some monochromators. The gas regulation system is controlled by the XGT board. In addition to these functions this electronic board drives the venting valve of the tank, checks the vacuum safety in order to prevent any X-ray leaks and controls the thermal stability.

Gas Regulation for Flow Proportional Counter (FPC)

Figure 2.40 shows the principle of the gas regulation. At the input of the ARL 9800 there is a restrictor R1 used to stabilise the argon-methane flow and the valve FPC V2 regulates the flow at 20ml per minute. The filter and the restrictor R2 smooth the flow to improve the reading of the flow sensor. The pressure sensor measures the argon-methane pressure at the gas input of the detector(s).

The valve FPC V1 regulates the pressure in the FPC detector(s) and the restrictor R3 avoids pressure shocks. The FPC V1 and the FPC V2 valve are proportional.

The valve FPC V3 is a safety valve to balance the pressure inside the detector with the atmospheric pressure when the main power supply is switched off or in case of problems with the regulation. The valve FPC V3 is an on/off device.

The pressure reducer fitted on the gas bottle must be adjusted according to the local pressure to have an absolute pressure of 1.25 bar at the gas inlet.

Example: the pressure reducer must be adjusted to 0.25 bar if the instrument is located at the sea level with 1 bar of local pressure.

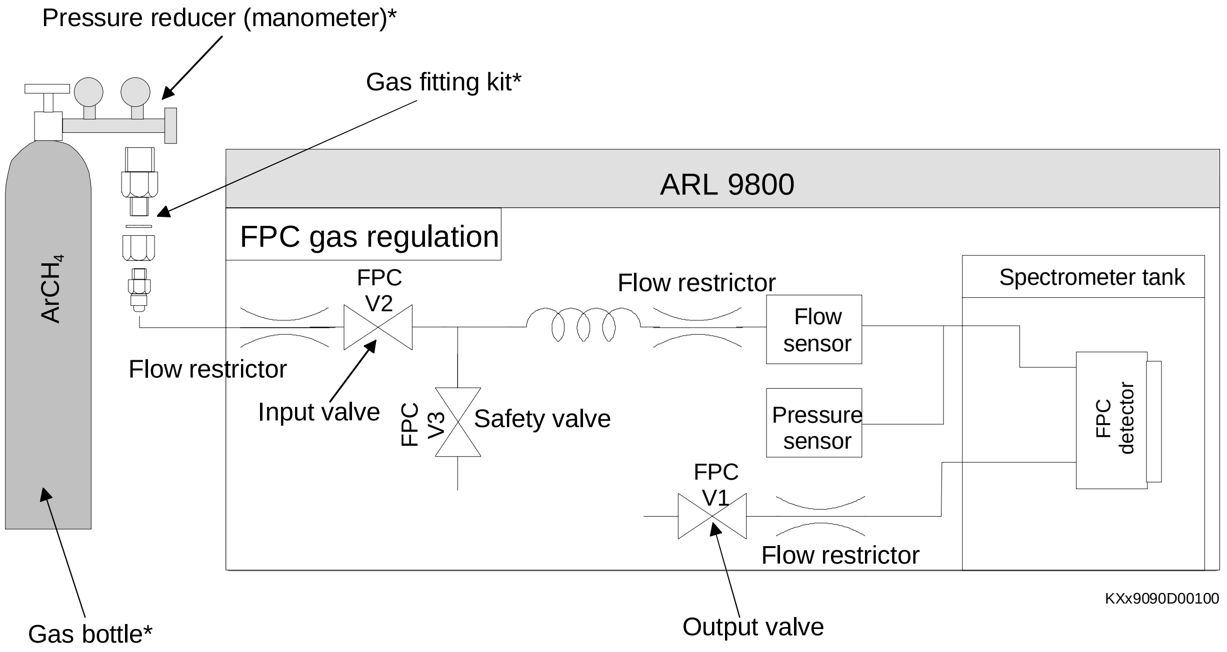


Figure 2.40

Note: Gas bottle, pressure reducer and gas fitting kit have to be provided by the end-user.

Figure 2.41 shows the location of the valves and sensors involved in the gas regulation.

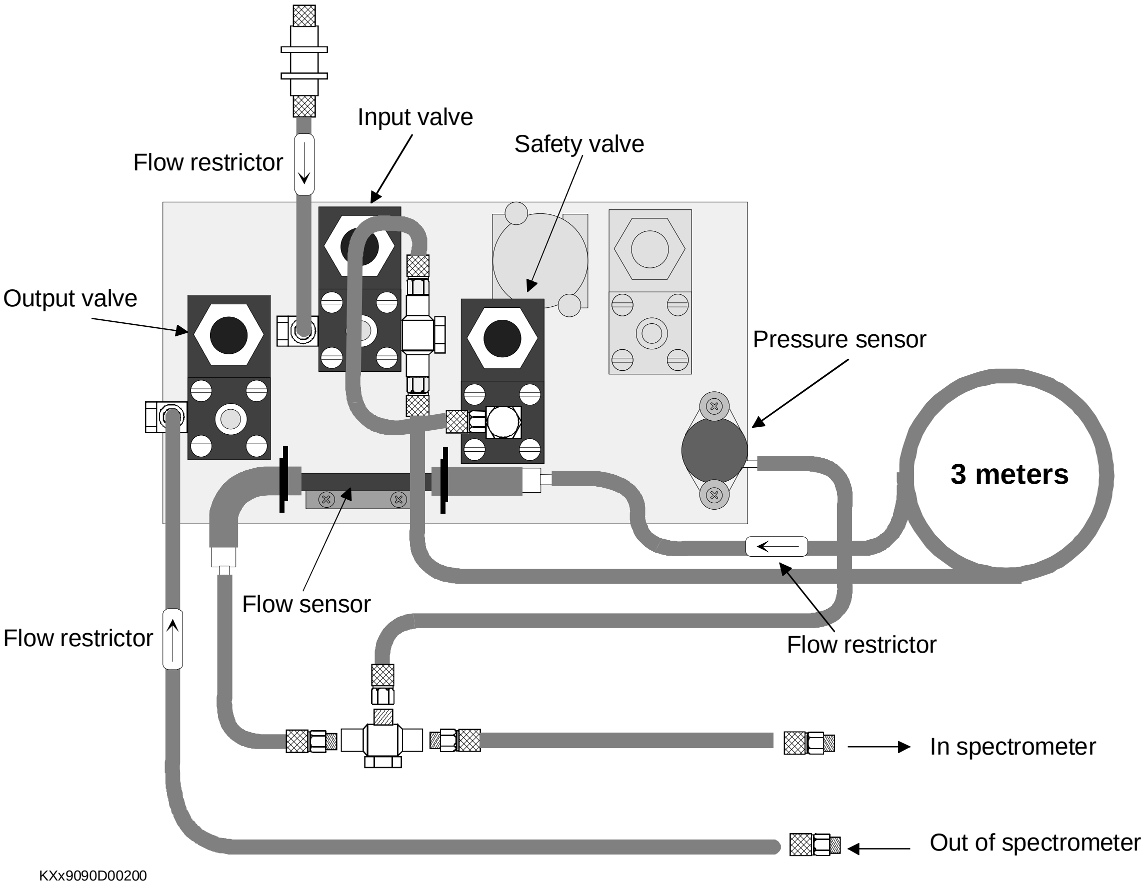


Figure 2.41

Thermal Regulation

The ARL 9800 instrument is designed to provide a precise thermal regulation. To permit this regulation all components which generate a lot of heat that would have a significant influence on the temperature stability have been located into an non thermostatised area. On the other hand, all the components that need to have a precise thermal stability have been located into a thermostatised area (called oven). Figure 2.42 shows these two areas.

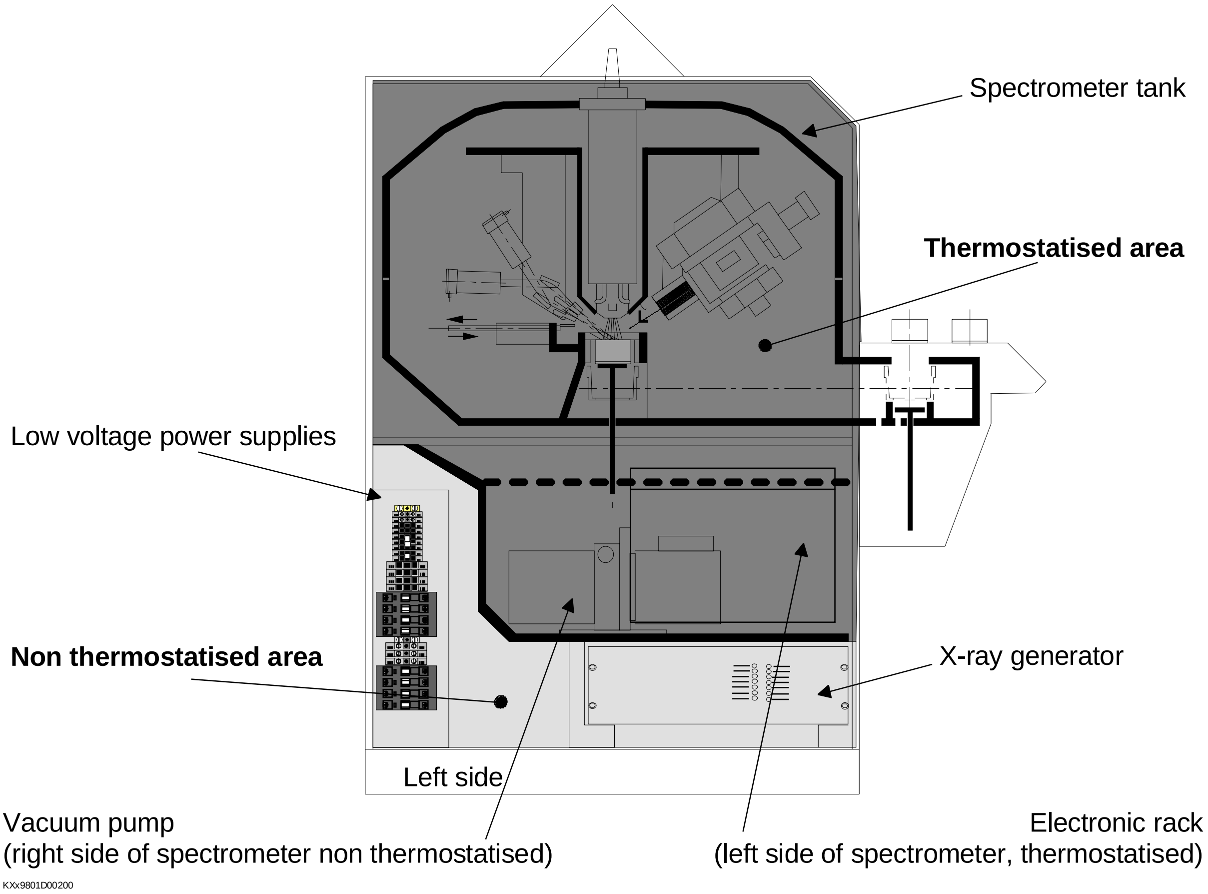


Figure 2.42

ARL 9800 XP Cooling Devices

This system cools the X-ray tube during operation. It also has all the water valves responsible for maintaining thermal stabilization of the spectrometer tank

The following figure shows the main cooling device components.

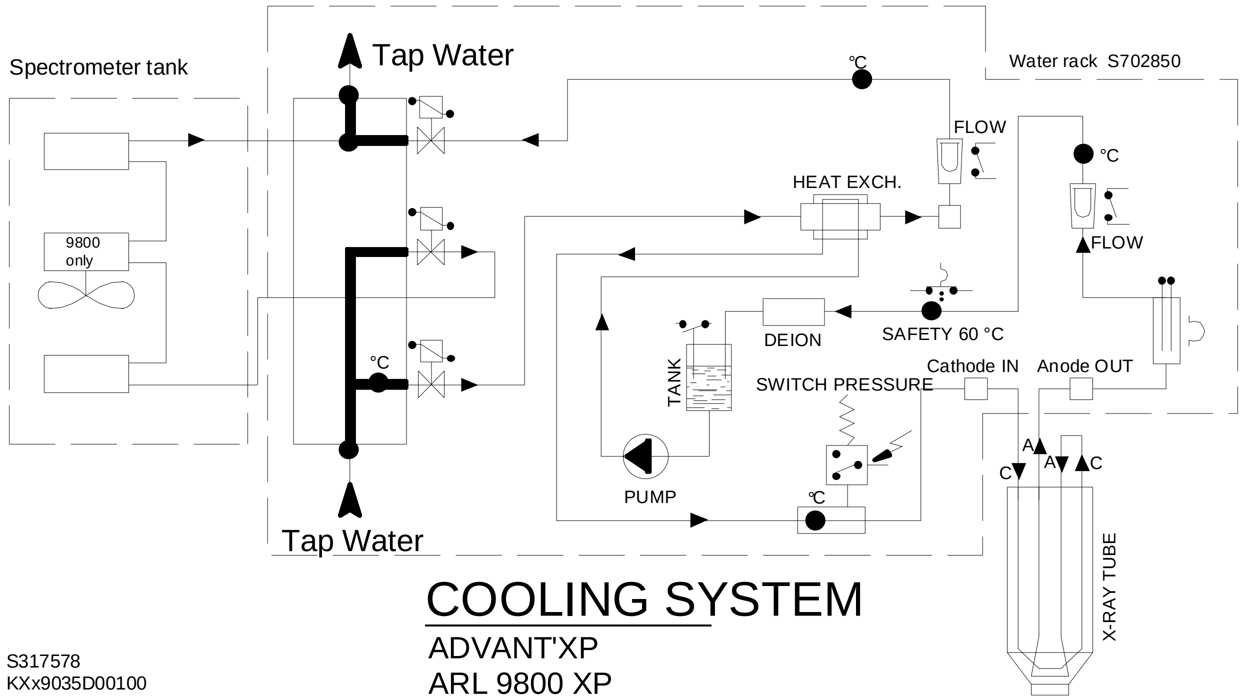


Figure 2.43

Instrument Thermostatisation

This system made up of radiators and heating resistors keeps the spectrometer tank and its delicate optical components at a carefully controlled temperature thereby ensuring superior analytical stability.

The temperature of the spectrometer tank is regulated by cooling with tap water and by heating with electrical resistors (see Figure 2.44). A temperature sensor located on the tank gives the information to the XGT electronic board. The XGT board drives the water valve and the heating resistors.

As shown in Figure 2.44, the spectrometer tank itself is cooled down with two water exchangers and an air/water exchanger to help cooling down the air around the tank. The water flow is controlled by a valve. Four electrical resistors are mounted on each exchange plate for heating.

The temperature tank is 30° C +/- 0.5°C. In order to optimise the thermal regulation, the crystals have also a heating system which stabilises their temperature at 38°C +/- 0.1°C. If the instrument has been turned off for a long period, it is recommended to wait about 4 hours before precise measurements are undertaken. An alarm message will indicate that the right temperature is not yet reached.

Note: The only component that is outside of the instrument and which could affect the stability due to a lack of thermal stability is the P10 gas bottle. The gas bottle needs to be protected from high temperature fluctuations or the gas needs to be transported in Cu pipes so as to reach the stable lab temperature before entering the instrument.

Figure 2.44 shows where the fans and the blocks with their resistors are located. An air flow also helps the temperature stabilisation.

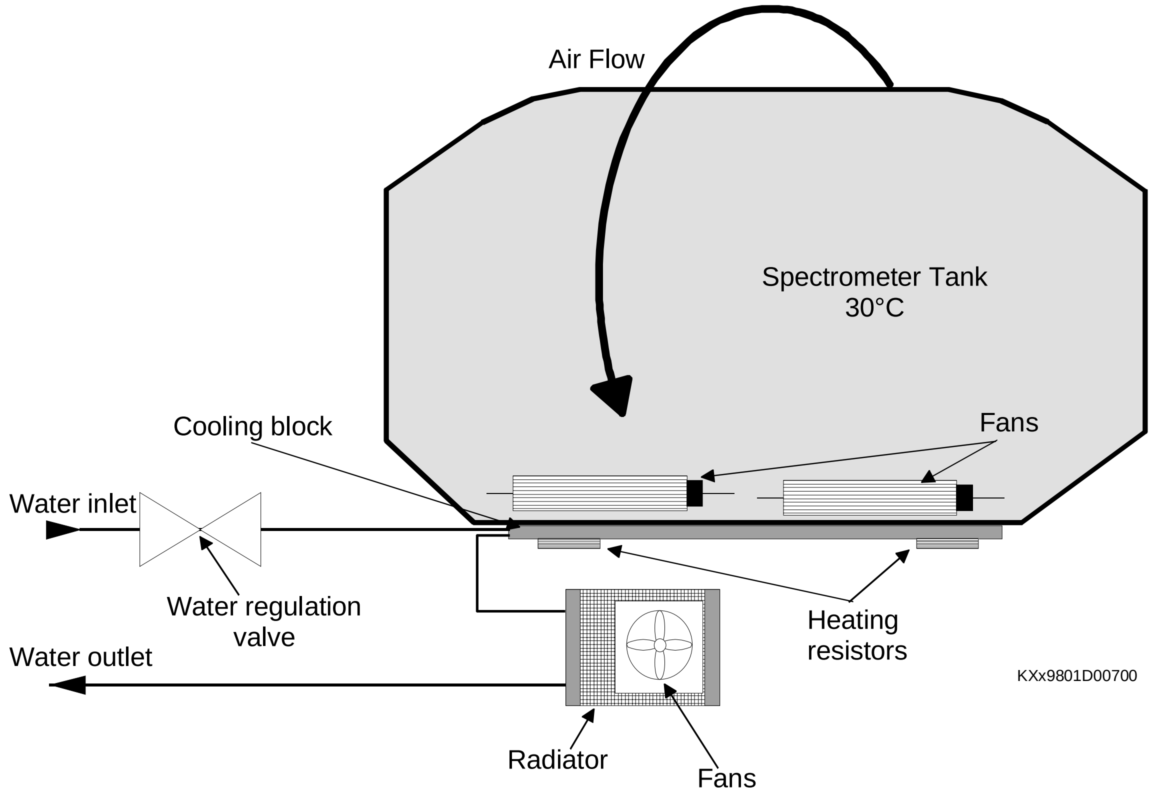


Figure 2.44

ARL 9800 OASIS Cooling Devices

The following figure shows the components of the main cooling devices.

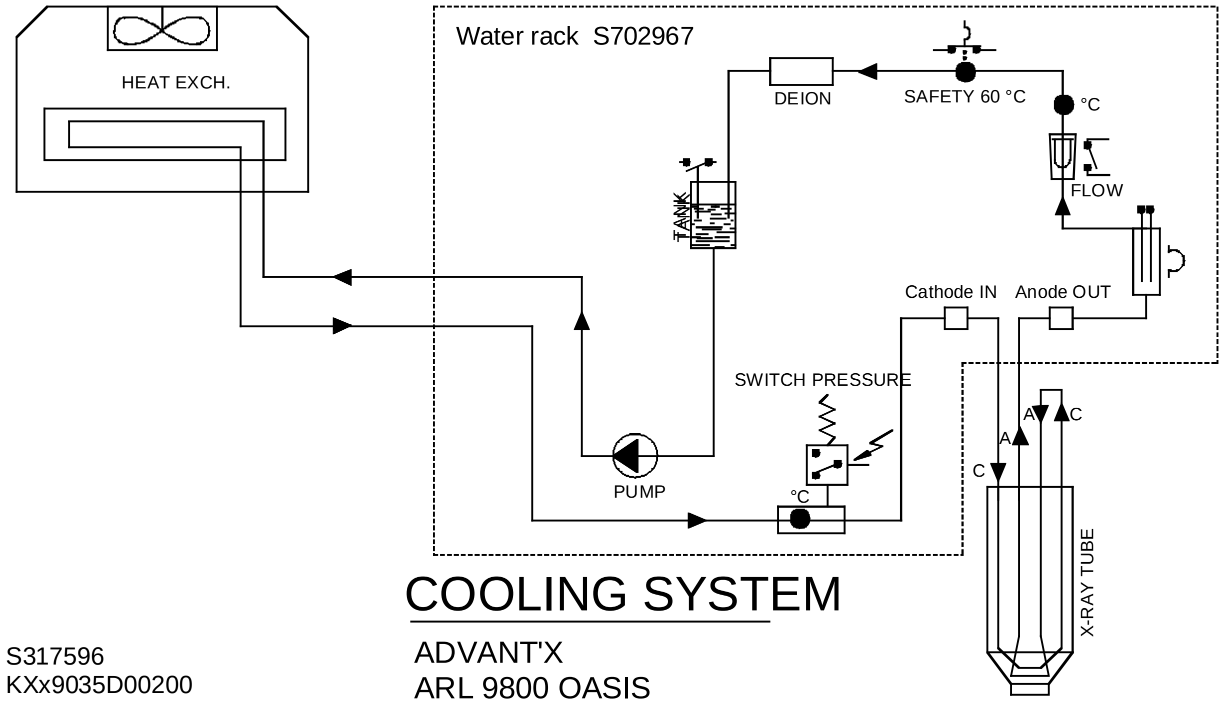


Figure 2.45

Instrument Thermostatisation

This system made up of heating resistors and fans keeps the spectrometer tank and its delicate optical components at a carefully controlled temperature thereby ensuring superior analytical stability.

The temperature of the spectrometer tank is regulated by cooling with fans and by heating with electrical resistors (see Figure 2.46). A temperature sensor located on the tank gives the information to the XGT electronic board. The XGT board drives the heating resistors and the fans.

As shown in Figure 2.46, the spectrometer tank is cooled down with two fans to help cooling down the air around the tank. At the button, the inlet fan inputs fresh air into the oven. At the top, the outlet fan outputs hot air from the oven. The air flow is controlled by this two fans. Height electrical resistors are mounted on two plates below the tank for heating.

The temperature tank is 36° C +/- 0.5°C. In order to optimise the thermal regulation, the crystals have also a heating system which stabilises their temperature at 43°C +/- 0.1°C. If the instrument has been turned off for a long period, it is recommend to wait about 4 hours before precise measurements are to be undertaken. An alarm message will indicate that the right temperature is not yet reached.

Note: The only component that is outside the instrument and could affect the stability by a lack of thermal stability is the P10 gas bottle. The gas bottle needs to be protected from high temperature variations or the gas needs to be transported in Cu pipes to reach the stable lab temperature before it gets into the instrument.

Figure 2.46 shows where the fans and the plates with their resistors are located. In the oven, an air flow helps also the temperature stabilisation.

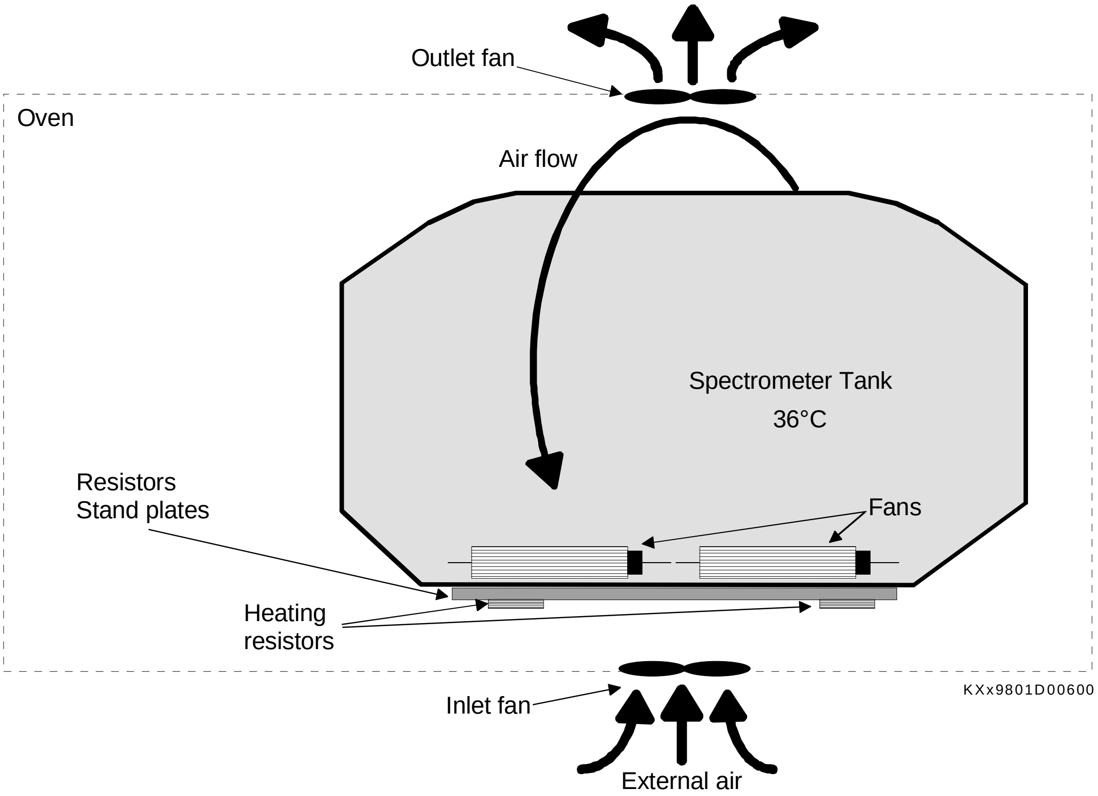


Figure 2.46

Thermal Regulation of the Crystals

All crystals mounted on the Goniometers, the Monochromators and the Diffraction System (XRD) are equipped with a thermal sensor and a heating resistor to guarantee a stable temperature and thus very stable analytical results.

Sample Excitation

Sample excitation is achieved by means of an end window X-ray tube which is energised by a solid state power supply. The X-ray tube cooling is achieved with a closed circuit water system. This water circuit is cooled by tap water or ambiant air through an exchanger.

In order to avoid any problems with X-rays or high voltage, the ARL 9800 is equipped with a complete safety interlock system.

Note: Never by-pass security that is installed in the ARL 9800 instrument. ARL will not be held responsible for any damage if the security is by-passed.

X-ray Tube

The X-ray tube is perpendicular to the horizontal plane and the distance between the anode and the sample surface is 30 mm.

The end window tube design allows also a very high sensitivity for light elements and thus low limits of detection. Light element analysis is very efficient with the 75 micron beryllium window because the radiation which is optimal for exciting the light elements is poorly absorbed by the thinner tube window. The usual target is Rhodium.

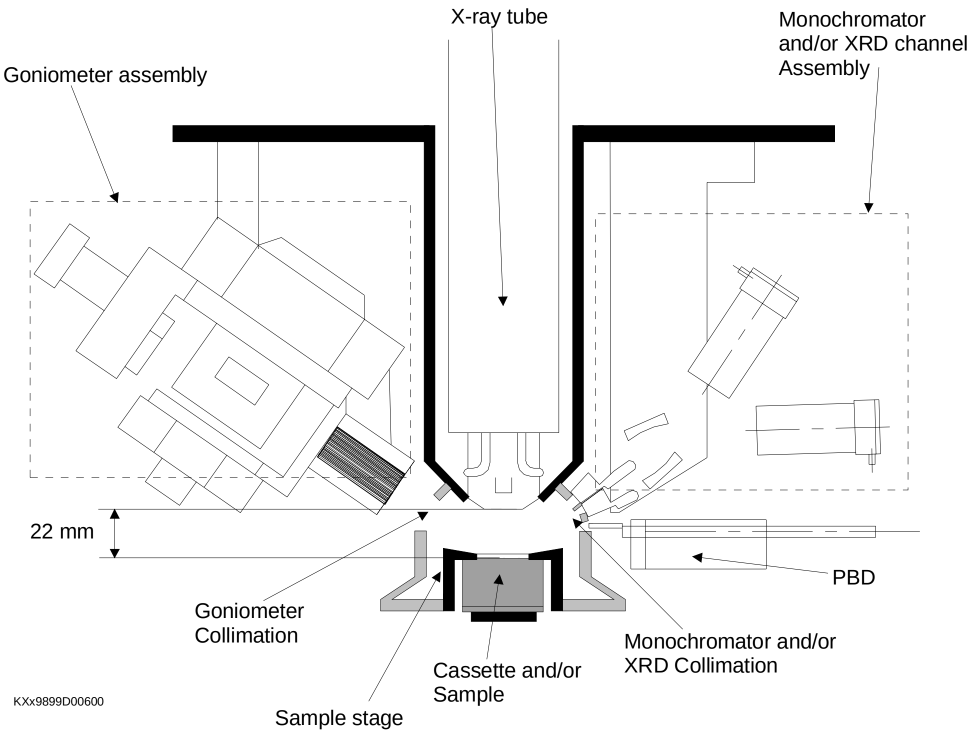


Figure 2.47

X-ray Power Supply (ARL 9800 XP)

The power supply is a solid state generator. This unit controls the high voltage and the current applied to the tube. The interface with the electronic is the XPS electronic board. The voltage can be selected between 0 and 60 kV by steps of 1 kV; an option is available to reach 70 kV. The current can be selected between 0 and 120 mA by steps of 1 mA with some restrictions if the voltage selected is lower than 20 kV because it is physically not possible to get for example 0 kV 120 mA. With the following formula the maximum current according to the desired high voltage can be calculated:

$$\text{Imax} = \frac{4}{100}\left(26 \ast \text{kV} + 5 \ast \text{kV}^2\right)$$

where: Imax is the maximum current authorised
kV is the desired voltage

Examples:

kV desired	I maximum	kV desired	I maximum
0 kV	0 mA	10 kV	30 mA
5 kV	10 mA	≥ 30 kV	120 mA

The resulting power of the chosen combinations must not exceed the maximum power (XP: 3.6 kW).

X-ray Power Supply (ARL 9800 OASIS)

The power supply is a solid state generator. This unit controls the high voltage applied to the tube. The current applied to the filament tube and the interface with the electronic is the XPSLP electronic board. The voltage can be selected between 0 and 50 kV by steps of 1 kV. The current can be selected between 0 and 20 mA by steps of 1 mA with some restrictions if the voltage selected is lower than 20 kV because it is physically not possible to get for example 0 kV 20 mA.

Eco Mode

The Eco mode is a function which allows you to lower the power consumption and to increase the life time of the X-ray tube. When the instrument is idle for a few hours the power is reduced to a stand-by value. For example, if the working condition is 50 kV and 50 mA and the instrument has been idle for 1 hour the power will be reduced to 40 kV and 20 mA in 30 minutes. As soon as the user starts a new measurement the required working condition will be reached automatically.

Another function is available to program the time to raise the X-ray tube to a certain working condition. This can be helpful if the instrument needs to be stabilised before the analysis is started. For example, the X-ray tube is at 40 kV / 20 mA during the night and in the morning it is raised to 50 kV / 50 mA 1 hour before the user arrives in the lab.

The Chapter *Instrument Preparation* explains how this mode can be set up.

X-ray Safety Interlock

All panels or covers with one of the signs shown in Figure 2.48 are controlled by an interlock system. It will prevent from switching on the power on the X-ray tube if they are not in place. We strongly recommend the user to contact an ARL office if the user needs to remove these protections.

High Voltage

KXx9000D00600

Radiation

KXx9000D00400

Figure 2.48

This safety interlock operates only on the high voltage for the X-ray tube but the emergency button will switch off the main voltage power supply.

Exit

X-ray Cooling System (ARL 9800 XP)

The cooling of the X-ray tube is achieved with an internal closed circuit de-ionised water system, which in turn is cooled through a heat exchanger which uses either tap water. Figure 2.49 shows the details of the cooling system.

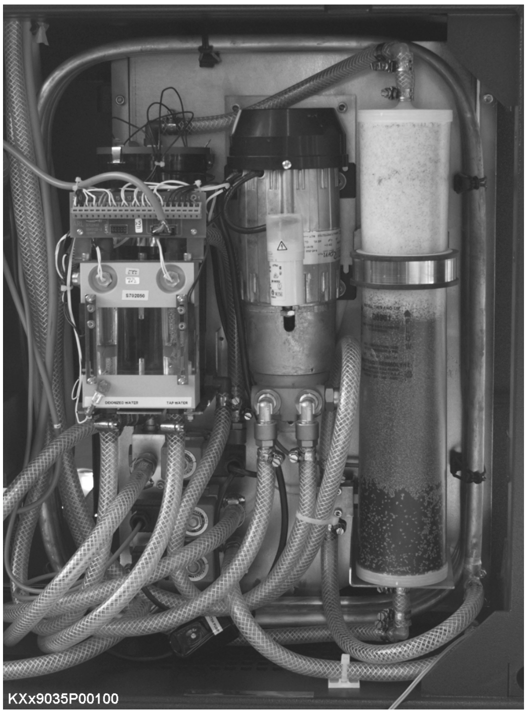


Figure 2.49

Water Cooling Circuit

The principle of the water cooling system is shown in Figure 2.50. The ARL 9800 XP checks several parameters of the water system like for the primary water circuit:

- Water flow
- Input water temperature
- Output water temperature

For the secondary water circuit:

- Water tank level
- Input water temperature
- Water resistivity
- Water flow (minimum 2.0 litre per minute)
- Water pressure at the tube inlet (maximum 4 bars)
- Output water temperature safety
- Output water temperature

Note: If one or several parameters are not within their tolerance, the X-ray tube power will be switched off immediately.

The water to cool down the X-ray tube goes first through the external cooling (called also cathode cooling) and then to the anode.

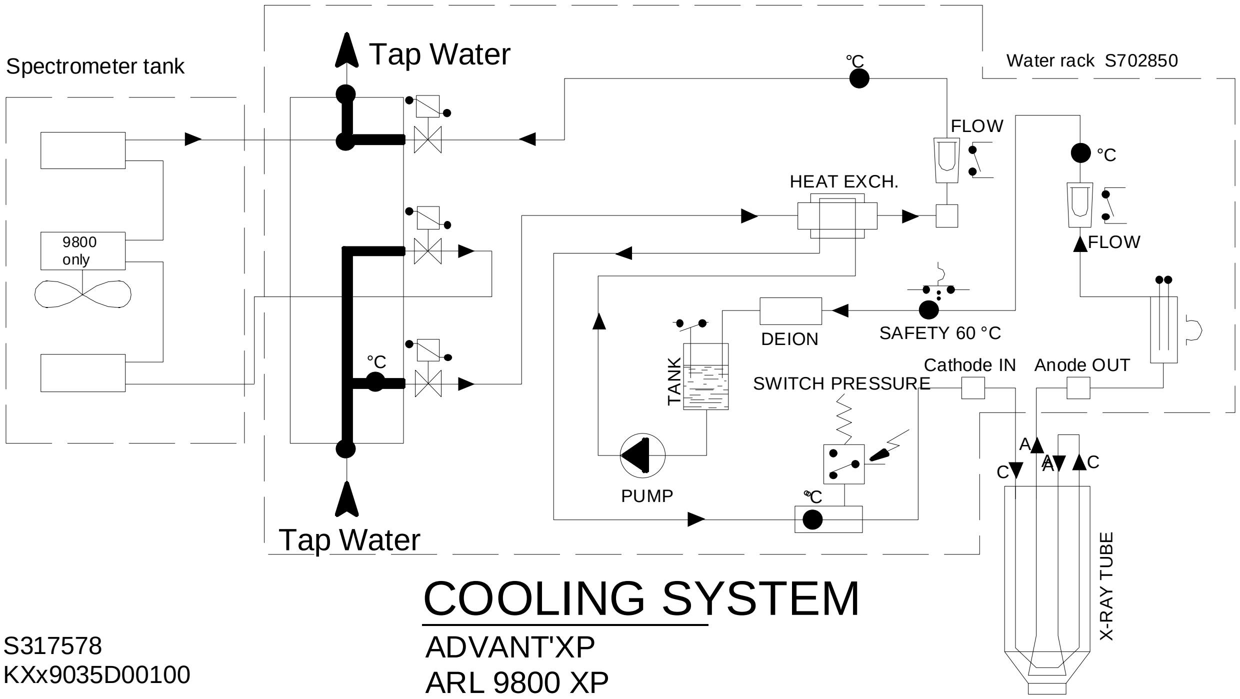


Figure 2.50

X-ray Cooling System (ARL 9800 OASIS)

The cooling of the X-ray tube is assumed by an internal closed circuit which is cooled through a external heat exchanger with air. Figure 2.51 shows the details of the cooling system.

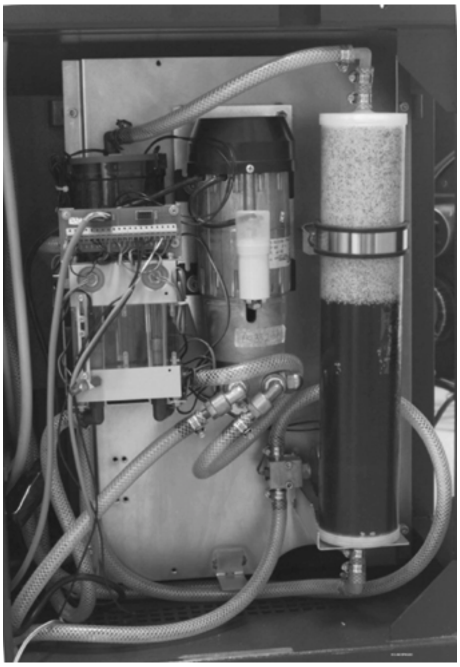


Figure 2.51

Cooling Circuit

The principle of the cooling system is shown in Figure 2.52. The ARL 9800 OASIS checks several parameters of the water circuit:

- Water tank level
- Input water temperature
- Water resistivity
- Water flow (minimum 2.0 litre per minute)
- Water pressure at the tube inlet (maximum 4 bars)
- Output water temperature safety
- Output water temperature

Note: If one or several parameters are not within their tolerance, the X-ray tube power will be switched off immediately.

The water used to cool down the X-ray tube goes first through the external cooling (called also cathode cooling) and then to the anode.

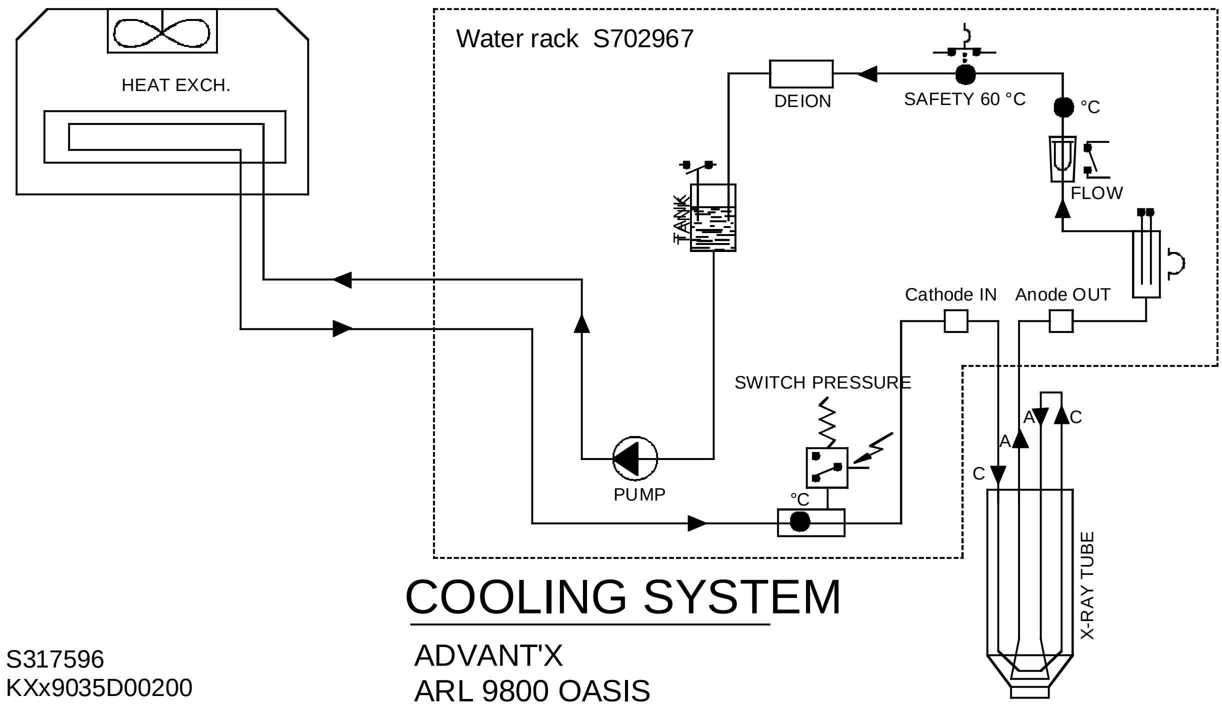


Figure 2.52

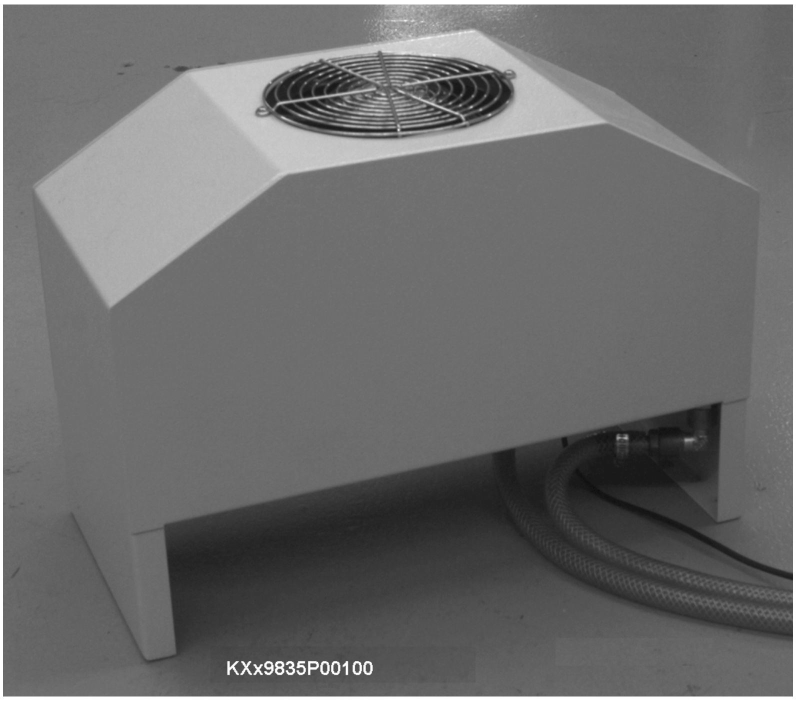


Figure 2.53 Water/heat exchanger (ARL 9800 OASIS version only)

Exit

Electronic Devices

The electronics of the ARL 9800 can be divided into the following groups:

Main Connections

These connections are made to connect the main power to the different internal components. It also allows to interface the ARL 9800 instrument with the external devices (water cooler, UPS, computer, etc.).

Electronic Rack

The ARL 9800 instrument is controlled by the electronic boards mounted in this rack. They also provide various status information to the operator via the instrument display.

X-ray Generator

This power supplies delivers the high voltage and current for the X-ray tube.

Low Voltage Power Supplies

These components generate all necessary low voltage powers for all the internal engines, valves, electronic boards, etc.

IMPORTANT NOTE

Never manipulate or touch electrical components without having switched off the complete ARL 9800 instrument with the main breakers.

Connection of the ARL 9800 to the main power must be made by a certified ARL specialist.

Philosophy of the Electronics

The following figure shows the basic electronics interconnection principle of the analytical components.

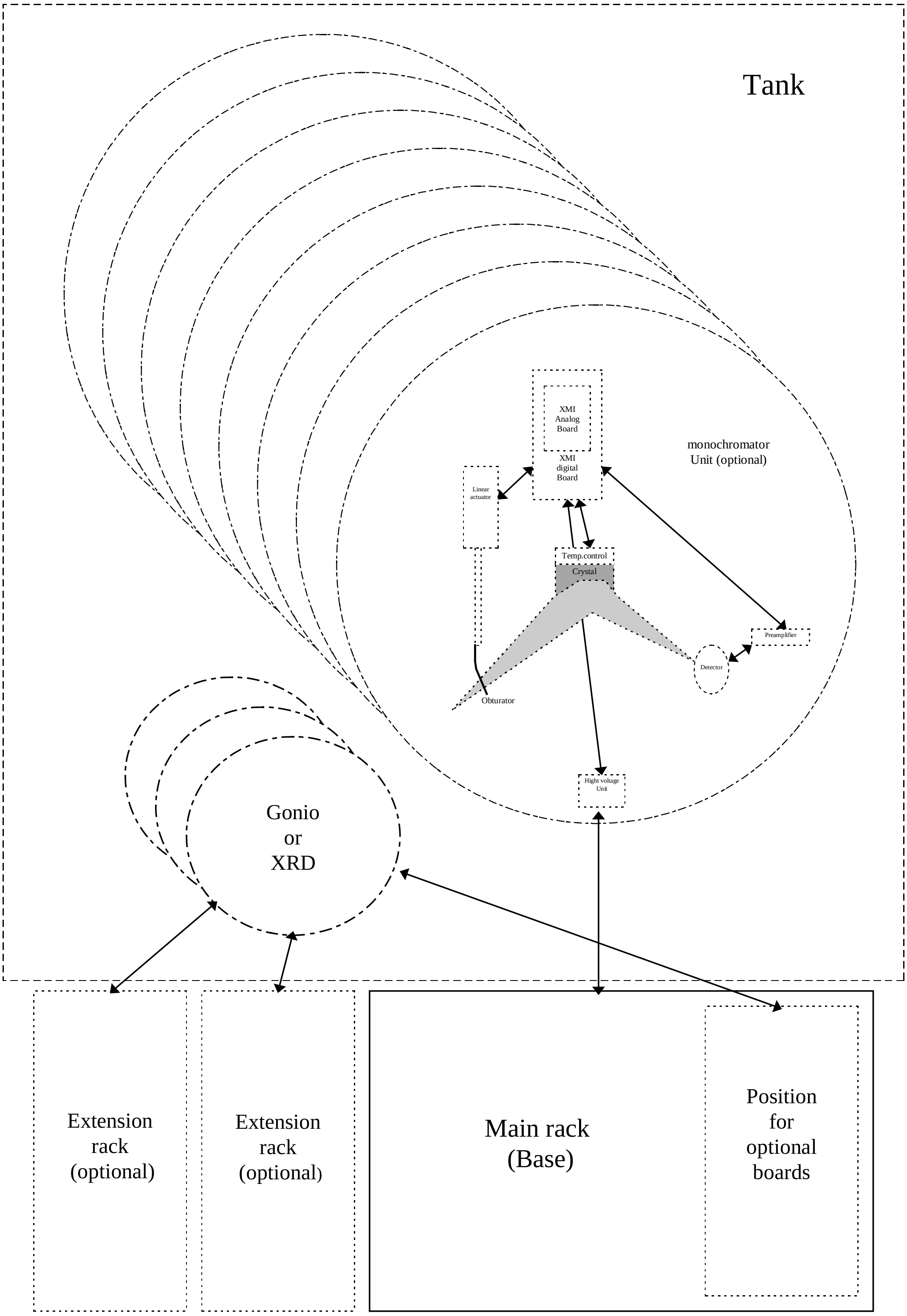


Figure 2.54

Main Boards Rack

The following standard boards are contained in the main rack:

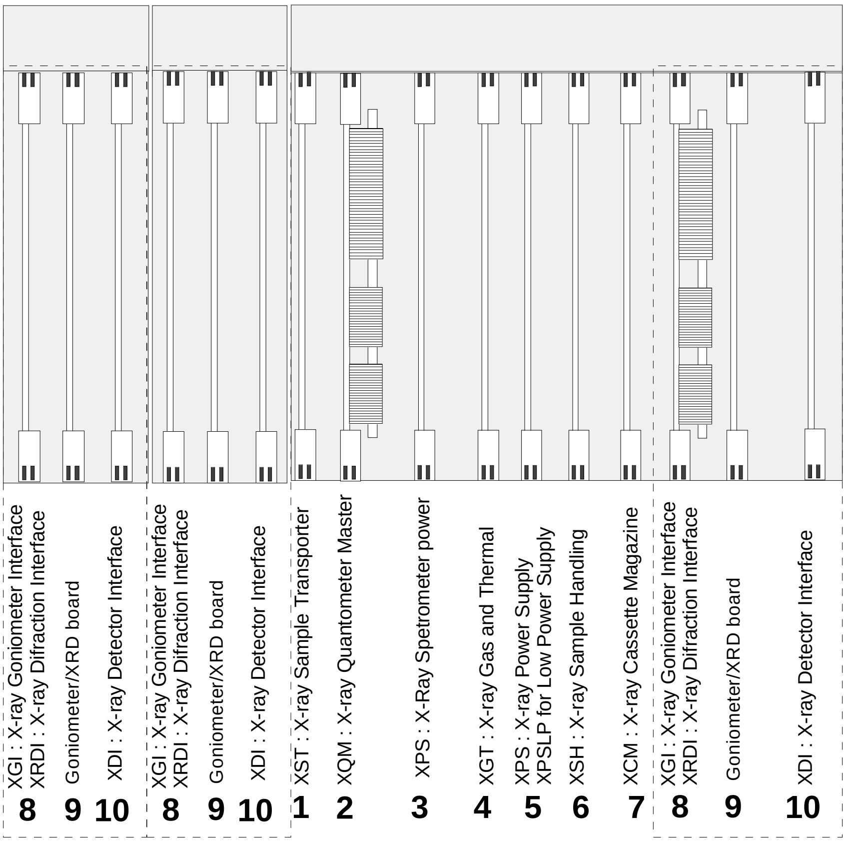
- XQM, XST, XGT, XPS, XSP

Depending on the sample handling system, one of the following boards is installed:

- 12 Position Sample Changer (XCM)
- X-Y Sample Changer (XHI)
- No Sample Changer (no board)

3 positions for one goniometer or one XRD system:

- One F44 goniometer (XGI, GonioPowerBoard and XDI)
- One XRD system (XRDI, GonioPowerBoard and XDI)
- Nothing (three free positions)



KXx9815D00300

Figure 2.55

Exit

Extension Boards Rack

The extension rack contains 3 positions (see Figure 2.55, positions 8, 9 and 10) for one goniometer or one XRD system:

- ♦ One F44 goniometer (XGI, GonioPowerBoard and XDI)
- ♦ One XRD system (XRDI, GonioPowerBoard and XDI)

Note: A maximum of two goniometers and one XRD system or 32 monochromators can be installed in the instrument.

Description of the Boards

Position	
1	**XST X-ray Sample Transporter** This board manages all the sample loading devices.
2	**XQM X-ray Quantometer Master** This board is the interface with the computer and it manages all the other boards. The vacuum is controlled by the board.
3	**XSP X-ray Spectrometer Power** This electronic board is the power interface between the XQM and its devices.
4	**XGT X-ray Gas and Thermal** This electronic board monitors and controls the FPC gas part and the thermal part of the spectrometer.
5	**XPS X-ray Power Supply** This electronic board controls the X-ray power supply and the X-ray tube cooling system.
5’	**XPSLP X-ray Power Supply for Low Power instrument (OASIS)** This electronic board controls the HV X-ray power supply and the X-ray tube cooling system, and supply the X-ray tube filament current.
6	**XHI X-ray Sample Handler Interface** This electronic board drives the large sample changer (XY samples magazine).
7	**XCM X-ray Cassette Magazine** This electronic board drives the 12 Position Sample Changer.
8	**XGI X-ray Goniometer Interface** This electronic board drives the goniometer movements, the collimator and crystal changers.
	XRDI X-ray Diffractometer Interface This electronic board drives the diffraction system.
9	**Goniometer Power** This electronic board is the power interface between the XGI/XRDI and the mechanical movements. It supplies also the high voltage for the detectors.
10	**XDI X-ray Detector Interface** This electronic board counts the number of photons received by the detectors.

In the tank

XMI X-ray Monochromator Interface

For each monochromator such an electronic board is in charge of the monochromator control including the high voltage for the detector, the intensity attenuators and the photons counting.

XQM X-ray Quantometer Master

This board is the master board of the electronics. It dialogues to slaves by two serial links. One is used to dialogue with XST, XGT, XPS, XCM, XHI, XSP and XMI's (called XSN X-ray Services Network). The other dialogues with XGI's or XRDI board (called XGN X-ray Goniometer Network). Its principal task is to synchronise the slaves.

It controls also the vacuum components like the 4 vacuum valves, the 2 vacuum pumps and the vacuum gauges, helped by the XSP board, and the 'PowerDistributionBoard' which is located at the rear of the instrument.

The board checks and monitors all the important status. The most important status are:

- The speed of the molecular pump
- The vacuum pressure in the tank and in the primary chamber airlock
- The temperature of the electronic rack
- The low voltage power supplies used on the board

XSP X-ray Spectrometer Power

This board drives the molecular pump, the 4 vacuum valves and the PBD (Primary Beam Device). It generates -5V used in the instrument and +55V used to drive the molecular pump.

The board checks and monitors all the important status. The most important status are:

- The main voltage line 1 and line 2 (true RMS)
- The voltage of the molecular pump
- The current of the molecular pump
- The temperature of the molecular pump
- The low voltage power supplies used on the board

XST X-ray Sample Transporter

This board is used to control the shutter of the primary chamber, the load lift, the transfer axis, the analysis lift and the sample rotation. When a sample is loaded into the spectrometer its height is checked.

The board checks and monitors all the important status. The most important status are:

- The angular error of the rotation
- The low voltage power supplies used on the board

XGT X-ray Gas and Thermal

This board controls the pressure and the flow of the FPC gas helped by the board 'FpcGasControlBoard' located in the thermostatised part below the tank. It also controls the temperature of the spectrometer with one water valve and 8 power resistors.

The board checks and monitors all the important status. The most important status are:

- The gas pressure
- The gas flow
- The tank temperature

- The low voltage power supplies used on the board

XPS X-ray Power Supply

This board controls the generator which supplies the high voltage to the anode and the current to the filament of the X-ray tube. It controls also the safety circuit and the internal and external water circuit.

The board checks and monitors all the important status. The most important status are:

- The voltage of the X-ray tube
- The emission current of the X-ray tube
- The filament current
- The number of X-ray tube arc events
- The X-ray tube running time
- The temperature of the water
- The resistivity of the internal water which is used to cool the anode of the X-ray tube
- The low voltage power supplies used on the board

XPSLP X-ray Power Supply for Low Power instrument (OASIS)

This board controls the generator which supplies the high voltage to the anode. It generates the current for the filament and controls the emission current of the X-ray tube. It controls also the safety circuit and the deionized water circuit.

The board checks and monitors all the important status. The most important status are:

- The voltage of the X-ray tube
- The emission current of the X-ray tube
- The X-ray tube leakage current
- The filament current
- The filament voltage
- The number of X-ray tube arc events
- The X-ray tube running time
- The temperature of the deionized water
- The temperature of the laboratory
- The temperature of the board
- The temperature of the outlet air of the HV generator
- The resistivity of the deionized water which is used to cool the anode of the X-ray tube
- The low voltage power supplies used on the board

XHI X-ray Sample Handler Interface

This board controls the large X-Y sample changer. It also controls the belt(s) if the instrument is automated and has belt(s) to convey the samples.

The board checks and monitors all the important status. The most important status is:

- The low voltage power supplies used on the board

XCM X-ray Cassette Magazine

This board controls the 12 Position Sample Changer.

The board checks and monitors all the important status. The most important status is:

- The low voltage power supplies used on the board

XGI X-ray Goniometer Interface

This board controls all movements of the goniometer by reading the encoder signals of the 2 axis. It controls also the collimators changer, the crystal changer and the temperature of the crystal changer. It synchronises the different motion according to the analysis.

The board checks and monitors all the important status. The most important status are:

- The high voltage of the detectors
- The temperature of the crystal changer
- The low voltage power supplies used on the board

Goniometer Power

This board generates the current for the 2 motors and the high voltage for the 2 detectors on the goniometer.

XDI X-ray Detector Interface

This board counts the photons which are detected by the detectors on the goniometer. The pulses are first corrected in magnitude (AGC) and in time (to decrease the pile-up effect), then converted and selected according to the PHD settings.

The board checks and monitors all the important status. The most important status are:

- The AGC value
- The reference voltages
- The low voltage power supplies used on the board

XMI X-ray Monochromator Interface

This board, positioned in the tank, counts the photons which are detected by the detector on the monochromator. The pulses are first corrected in magnitude (AGC). Then the pulses are converted and selected according to the PHD settings. It also controls the intensity attenuators and the high voltage unit which is positioned at the bottom of the tank.

The board checks and monitors all the important status. The most important status are:

- The AGC value
- The temperature of the board
- The high voltage of the detector
- The temperature of the crystal
- The low voltage power supplies used on the board

Sample Loading System

To access the spectrometer lift, the electronic rack can be rotated forward by loosening the locking screw.

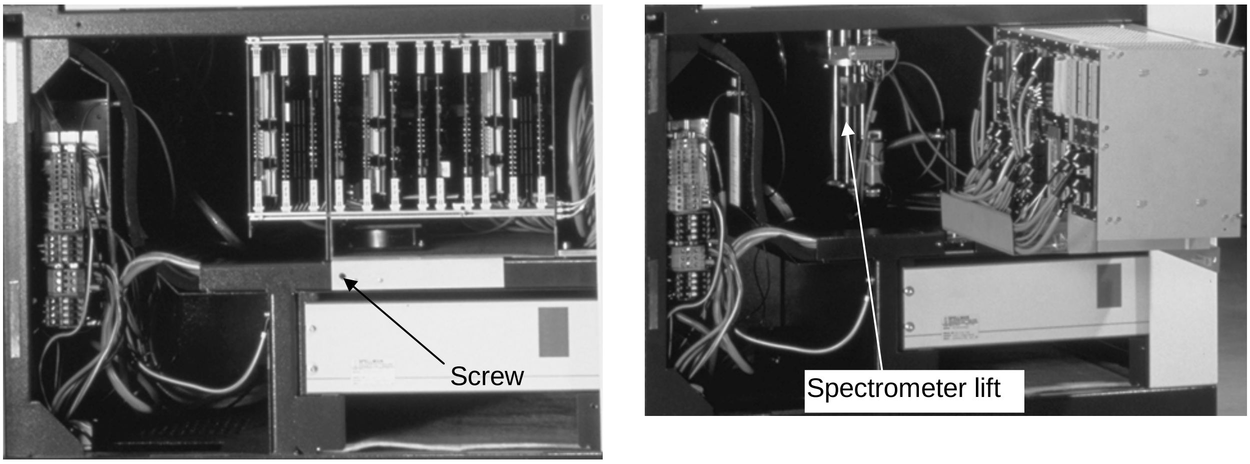


Figure 2.56

The following figure shows the principle of the sample loading system.

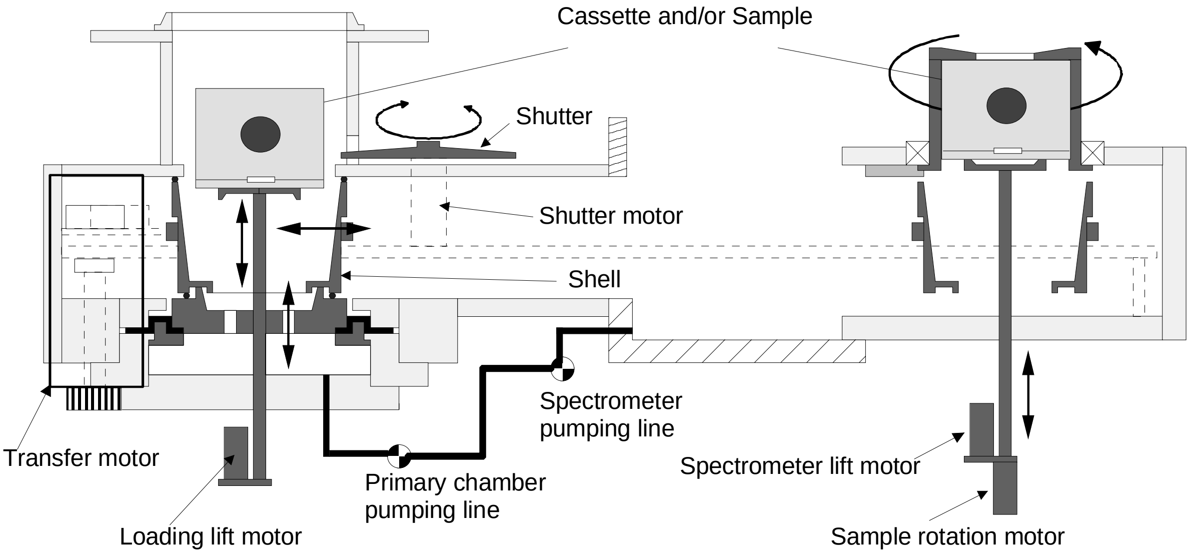


Figure 2.57

Sample Changer	The sample changer allows to prepare several samples in advance. Furthermore, the required samples can then be selected by the analytical task through the coded positions of the sample changer. Various types of sample changers can be mounted (12 position, X-Y sample changer, SMS Automation System).
Shutter	Mechanism to isolate the cassette and/or the sample in the primary chamber from the outside world in order to pump down the sample to the pressure corresponding to the vacuum inside the spectrometer.
Loading lift	Mechanism to transport the cassette and/or the sample from the sample changer into the primary chamber.
Transfer	Mechanism to move the sample from the loading lift to the spectrometer lift.

Spectrometer lift

The spectrometer lift raises the cassette and/or the sample to the analytical position after it has been transferred from the primary chamber.

Sample rotation

Mechanism that spins the cassette and/or the sample during the analysis in order to compensate for inhomogeneities and irregular surface structures due to surface preparations, like grinding and milling. The sample rotation motor spins the sample at 1 revolution per second (default value) during the analysis time.

Sample Loading Process

The ARL 9800 is designed to analyse solid samples. These samples can be inserted into a cassette or can be put onto an adapter ring for analysis.

The analysis of the samples is done under vacuum. The sample is first loaded into the primary chamber which is under air. The shutter closes and the primary chamber is pumped down to a low pressure. The sample is then transferred to the spectrometer and raised to the analytical position, as this is explained hereafter.

- **Step 1**

Figure 2.58 shows the instrument ready to load a sample. The sample changer brings the desired sample onto the primary chamber lift.

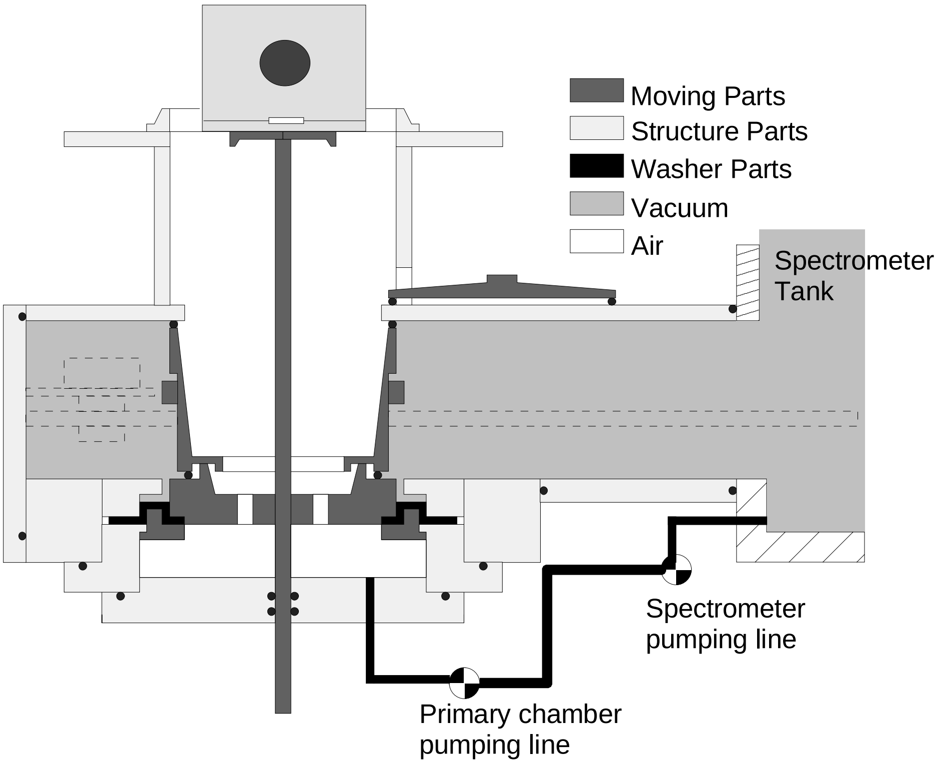


Figure 2.58

- **Step 2**

The loading lift places the cassette and/or the sample into the shell (see movement ‘a’).

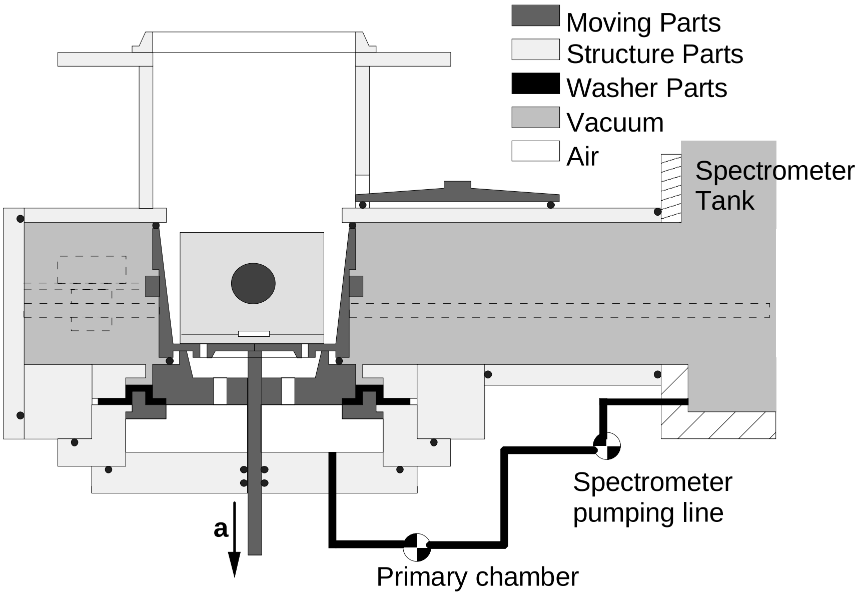


Figure 2.59

- **Step 3**

The shutter is closed (see movement ‘a’).

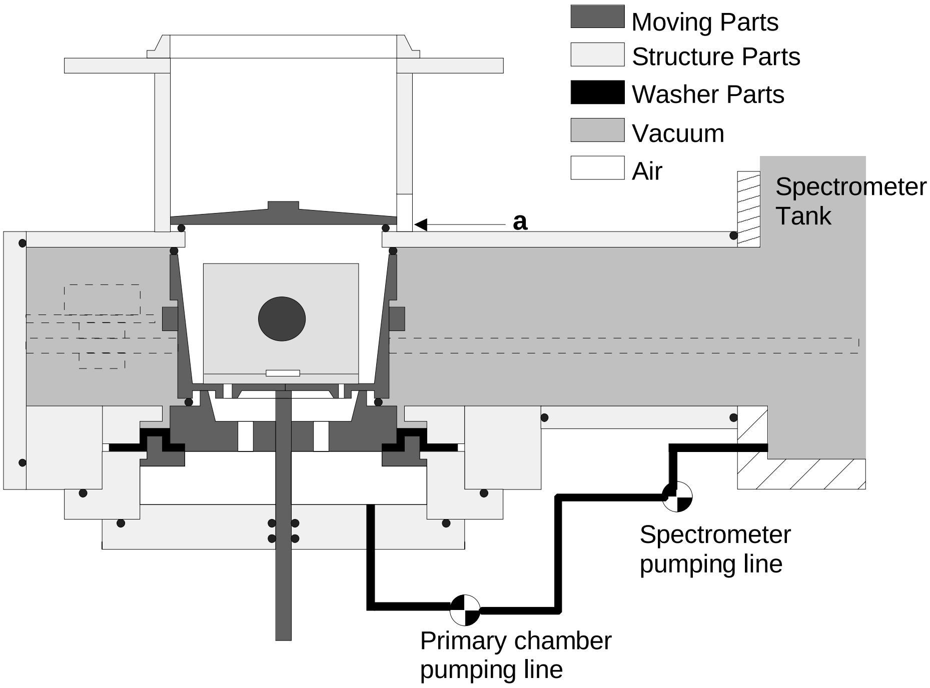


Figure 2.60

- **Step 4**

The primary chamber is then evacuated to a low pressure by the mechanical vacuum pump (see action ‘a’).

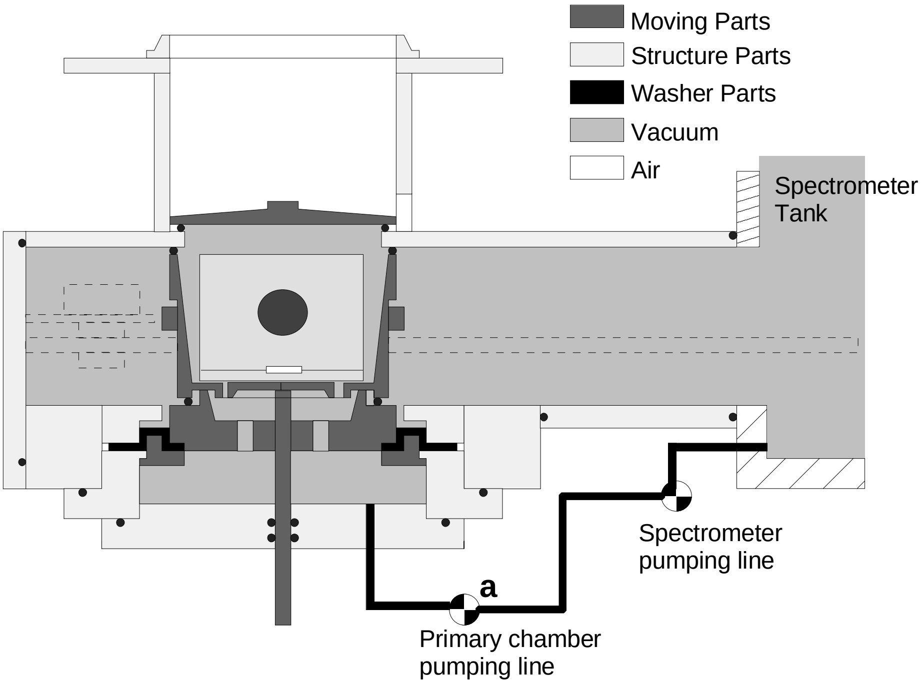


Figure 2.61

- **Step 5**

When the required vacuum is reached, the lift goes down further to open the ring and free the shell (see movement ‘a’).

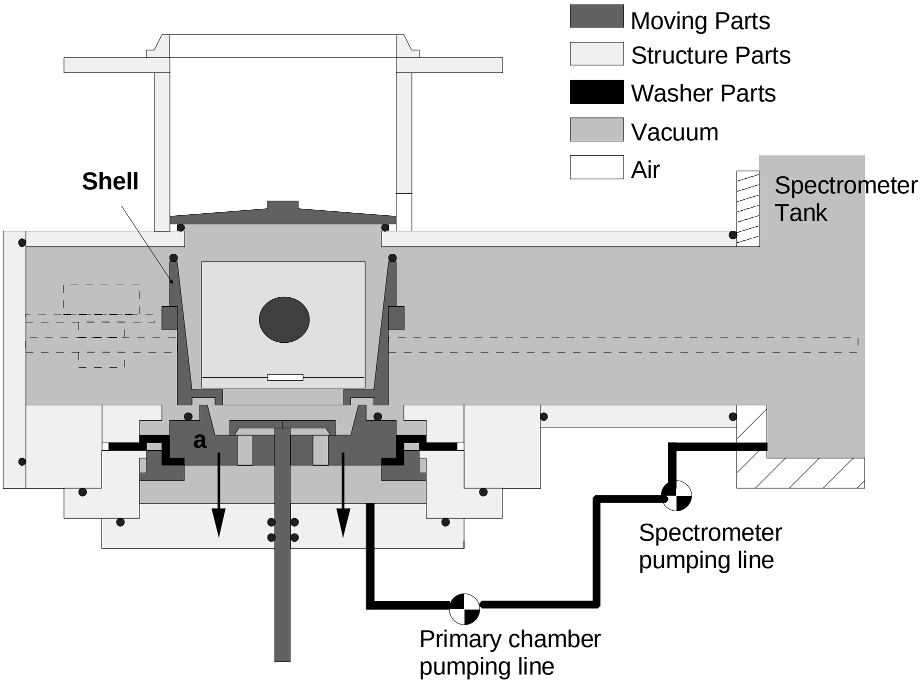


Figure 2.62

- **Step 6**

The shell and cassette are moved out of the primary chamber by the transfer mechanism (see movement ‘a’).

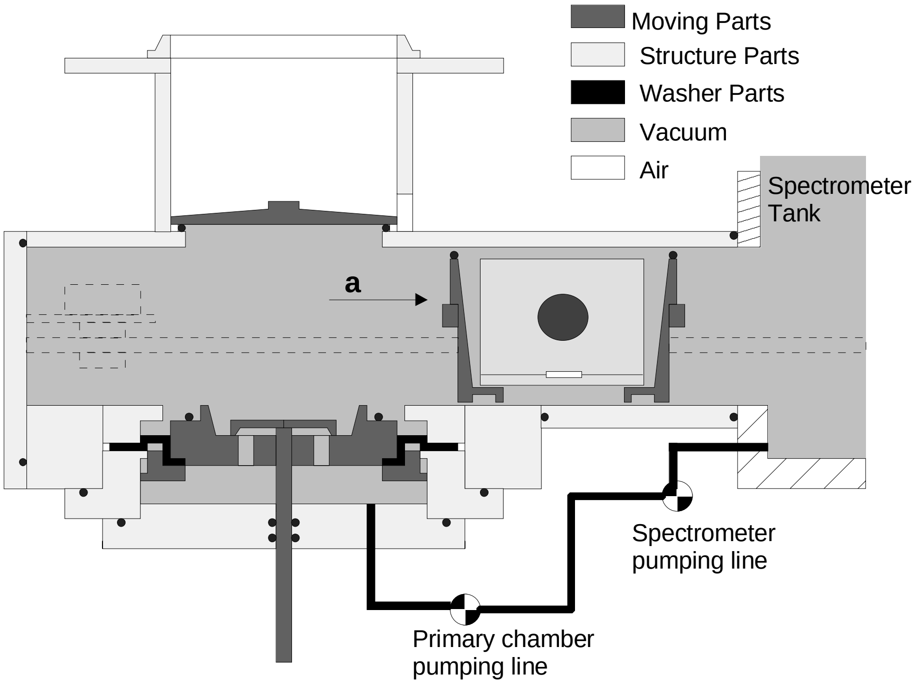


Figure 2.63

- **Step 7**

The cassette and/or the sample is then transferred into the spectrometer (see movement ‘a’).

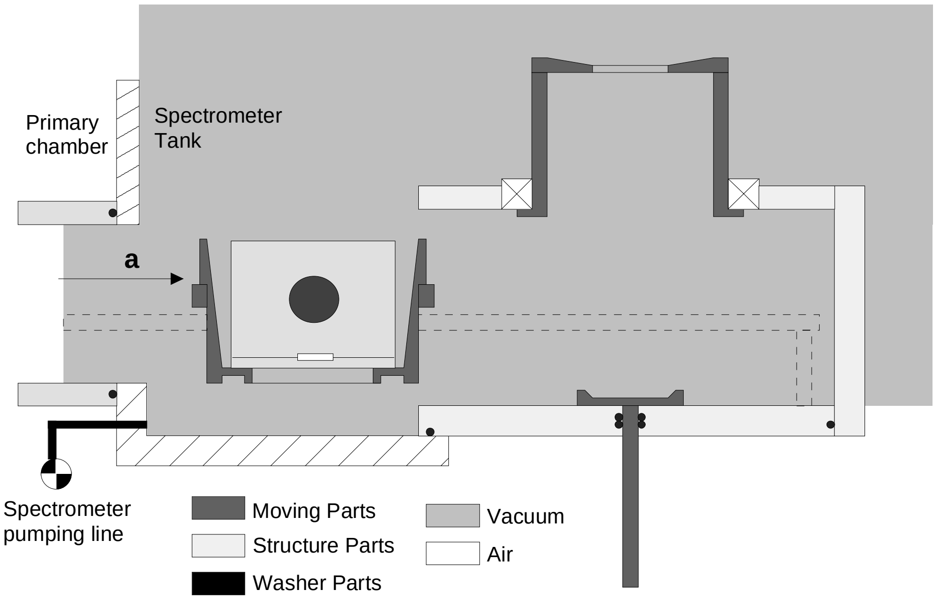


Figure 2.64

- **Step 8**

As soon as the shell is under the analysis position, the spectrometer lift raises the cassette and/or the sample to the analysis position (see movement ‘a’).

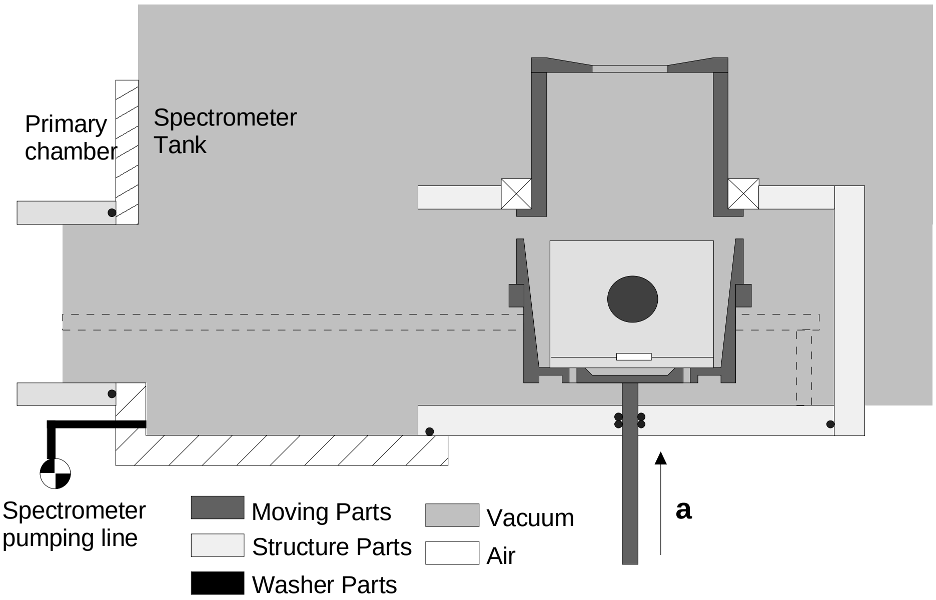


Figure 2.65

- **Step 9 (Sample in Cassette)**

The sample is raised to the analysis position by the spectrometer lift (see movement ‘a’).

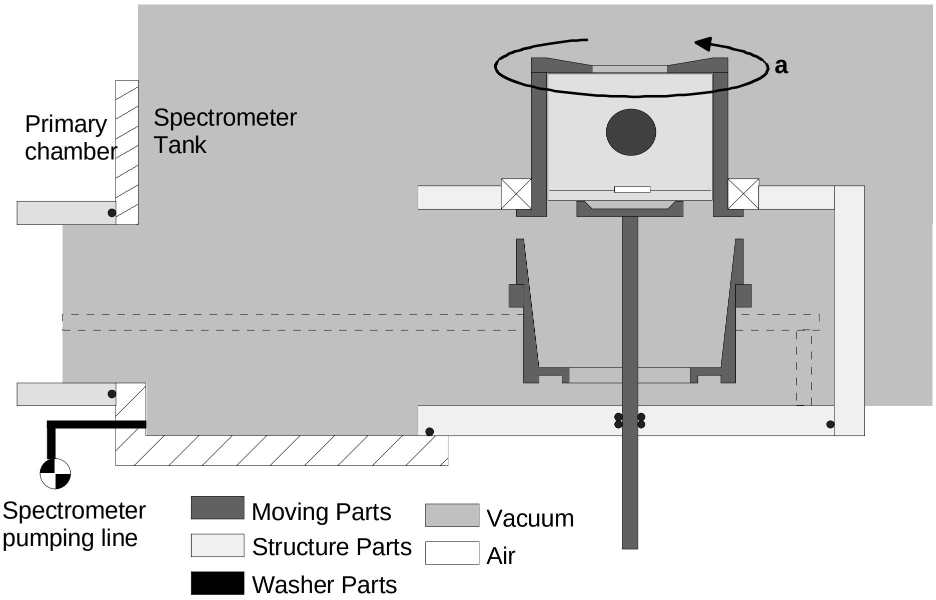


Figure 2.66

- **Step 9 (Sample without Cassette)**

The figure below shows a sample in the analytical position without a cassette, only placed on an adapter ring. The spectrometer lift moves the sample up into the analysis position. This movement is controlled by a sensor and therefore allows that the thickness of a sample can vary. The lift can also compensate for a non-parallelism of samples of 2 mm.

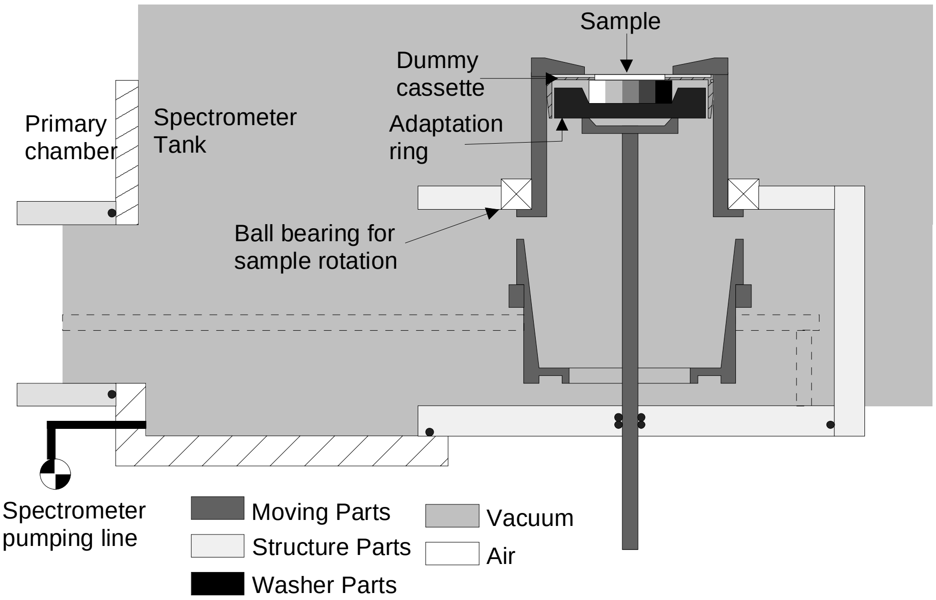


Figure 2.67

3

ANALYSIS PRINCIPLE

3 ANALYSIS PRINCIPLE

Introduction

X-ray spectrochemical analysis is based on the fact that the chemical elements emit characteristic radiations when subjected to appropriate excitation. The emission of characteristic line spectra can be induced by either the impact of accelerated particles such as electrons, photons, alpha particles and ions; or by the impact of high-energy radiation from an X-ray tube or from a suitable radioactive source. Generally, direct electron excitation is used in electron microprobe techniques, while radioisotope sources and proton generators are commonly associated with energy dispersion.

X-ray Emission

In a typical spectroscopic experiment, we usually interact with the neutral atom by supplying it with a certain amount of energy. The nature of the supplied energy can vary depending on the technique. For example, in a XRF experiment, we expose the atoms to X-radiation (called primary X-rays) while in an optical emission experiment we might use an electrical discharge or a high temperature plasma source to provide certain quantity of energy. The atom then absorbs some or all of this energy if this helps to displace the electrons. The displacement of electrons can occur within the atom, i.e. the electron goes to a higher energy state within the same atom by absorbing the incident energy, in which case we obtain an excited atom. The electron can also leave the atom in which case we get an ionized atom (a positive ion) since there will be excess positive charge in the nucleus. In a general situation, both excited atoms and ions may be present together.

In our case, when the primary X-rays from the X-ray tube interact with the sample, the following phenomena take place:

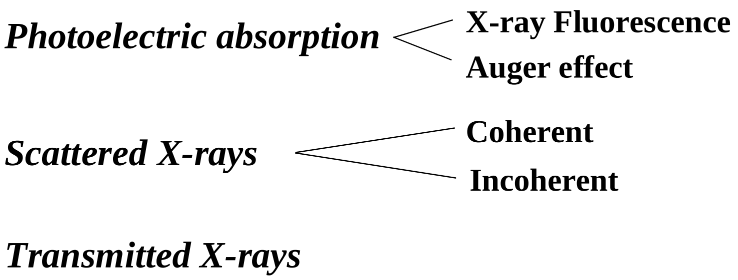


Figure 3.1

Photoelectric absorption

When a sufficiently energetic X-ray photon interacts with an atom, several phenomena take place. One interaction involves the transfer of the photon energy to one of the electrons of the atom (e.g. a K shell electron) resulting in its ejection from the atom. The distribution of electrons in the ionized atom is then out of equilibrium and within an extremely short time returns to the normal state, by transitions of electrons from outer shells to inner shells. Each such electron transfer, for example from the L shell to the K shell, represents a loss in the potential energy of the atom; this energy reappears as a photon (in this case a Kα photon) whose energy is the difference between the binding energies of the two shells. One of the two processes can take place, namely:

a) The X-ray photon escapes from the atom and contributes to a characteristic radiation of the atom (Figure 3.2 left).

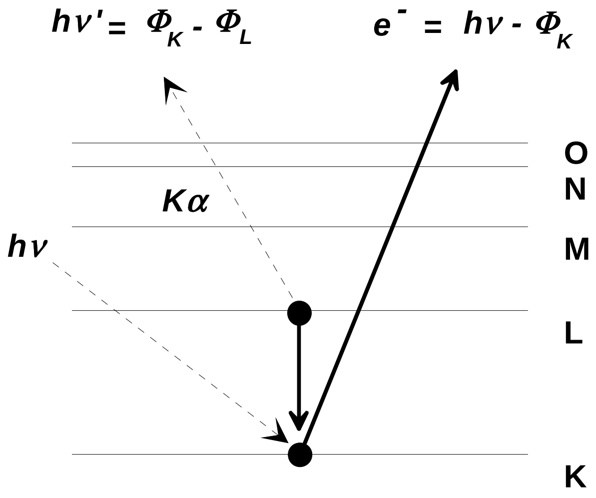


Figure 3.2

b) Or the photon is absorbed within the atom itself on its way out and ionizes the atom in an outer shell; for example a Kα photon can eject an L, M or N electron; this phenomenon is called the Auger effect. Again, the ionized atom becomes a source of radiation as explained above (Figure 3.3). It should also be noticed that because of the Auger effect in the higher levels two or more vacancies could be created. The creation of double vacancies is responsible for the appearance of satellite peaks.

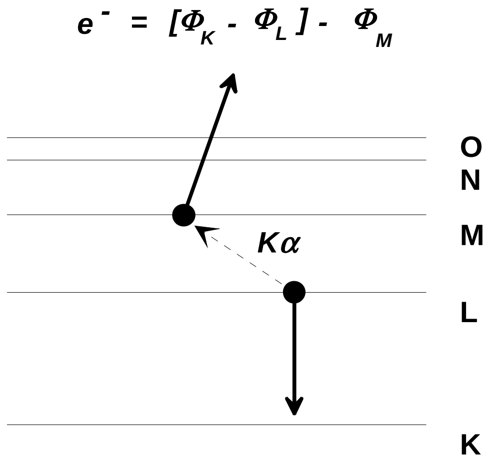


Figure 3.3

c) The Auger emission is more probable for low atomic numbers while X-ray emission is more likely for high atomic numbers. The probability that an X-ray photon will be emitted (instead of an Auger electron) is called Fluorescence Yield.

The Figure 3.4 shows the fluorescence yield as a function of atomic number. The K lines are more probable than the L lines which explains the difference in the yield for the K and L type transitions. In practical X-ray spectrometry, we use the K lines to measure elements with low to medium atomic number while we switch over to measure L lines for the heavy elements, or even to the M lines.

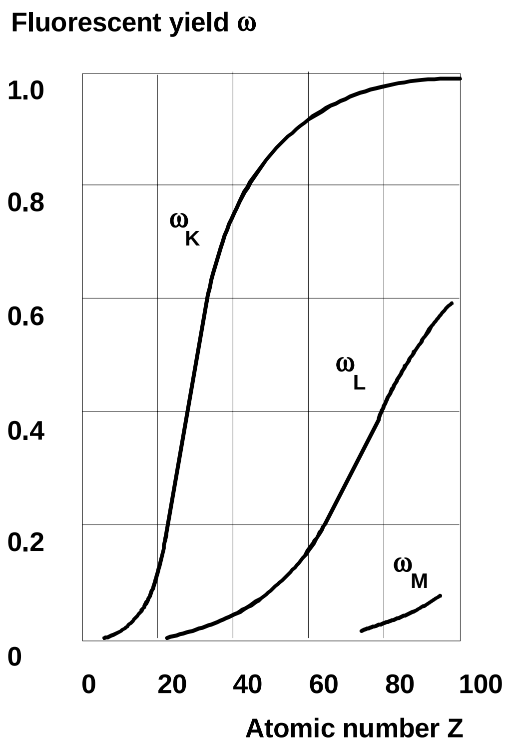


Figure 3.4

Scattering

Scattering of primary X-rays by the sample results in two processes: Coherent scattering (without loss of energy) and Compton scattering (with a little loss of energy). As mentioned before, not all of the incident X-rays participate in the photoelectric absorption. Some of them do not reach the inner shells and thus they do not give rise to absorption. Instead, they are deflected by the atoms in two ways:

a) The incident X-ray photon is deflected without loss of energy.
b) The X-ray photon is deflected with a slight loss in energy and thus with an increase in wavelength.

In a) the sample scatters primary X-rays with exactly the same energy. This scattering is thus called coherent or elastic scattering. In b), as shown in Figure 3.5, some of the photons collide with a loosely bound electron in an outer shell of the atom. The electron recoils under the impact and leaves the atom carrying with it some of the energy of the incident photon. Thus the primary photon is scattered with a slightly lower energy. This phenomena is called Compton scattering. The difference in energy between the incident photon and the Compton photon depends only on the angle (ϕ) between the unscattered and the scattered X-rays. It is given by the following equation:

$$\Delta\lambda = \lambda' - \lambda = h/m_o c\,(1\text{-}\cos\phi) = 0.02426\,(1\text{-}\cos\phi)$$

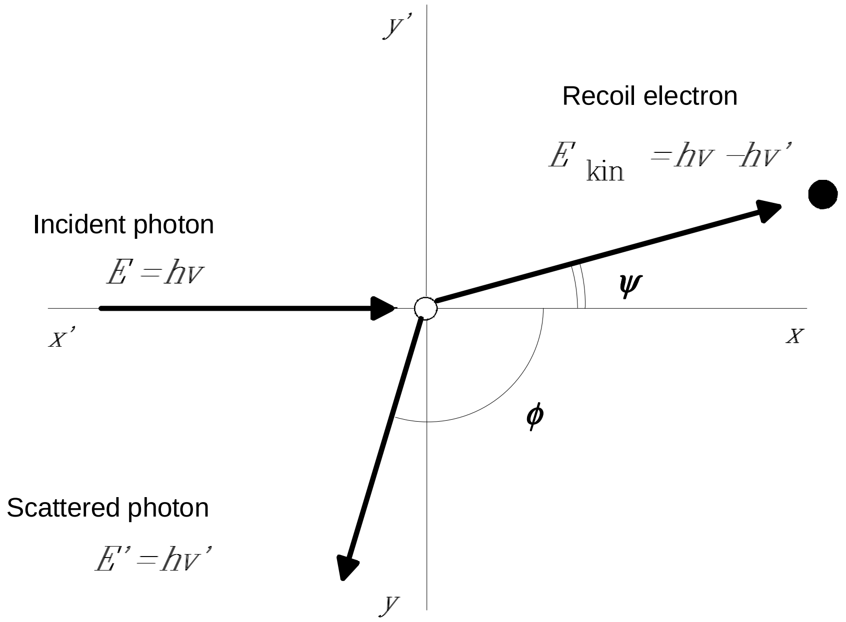


Figure 3.5

Thus Δλ is independent of both X-ray wavelength and the atomic number of the scatterer. On the other hand, the intensity ratio of the coherent to the Compton scattering depends on the atomic number of the scatterer. In practical X-ray spectrometry, we are confronted with scattered X-rays: Coherently scattered X-rays contribute to the background in the X-ray spectra. Since they fall exactly at the same energy as the measured line of a given element, they can't be discriminated in general. Thus background in XRF can largely be attributed to scattering of primary X-rays by the sample. Since heavier elements absorb more and scatter less, the background level in the spectra from heavy elements is almost insignificant. On the other hand, it becomes the limiting factor for measuring light elements at low concentrations.

Again, in practical XRF, this amounts to saying that, if the matrix is heavy, we have less problems with the background. If we are working with light matrices, we should be aware of the high level of background. Compton scattering manifests as broad lines accompanying the K and L lines of the X-ray tube target element. Figure 3.6 shows an example of these considerations.

All three spectra have been recorded with a Rh anode X-ray tube. Spectrum a) was measured with a light matrix (Teflon), b) with a medium (Pure Al) and c) with a heavy matrix (Pure Fe). The four lines observed are the Rh Kα and Kβ and the $\text{Rh}_{\text{Compton}}$ Kα and Kβ lines. We notice that the intensities of all four lines decrease as we go to heavier matrices. On the other hand, the ratio between the coherent and the Compton lines increase as we go to heavier element matrices.

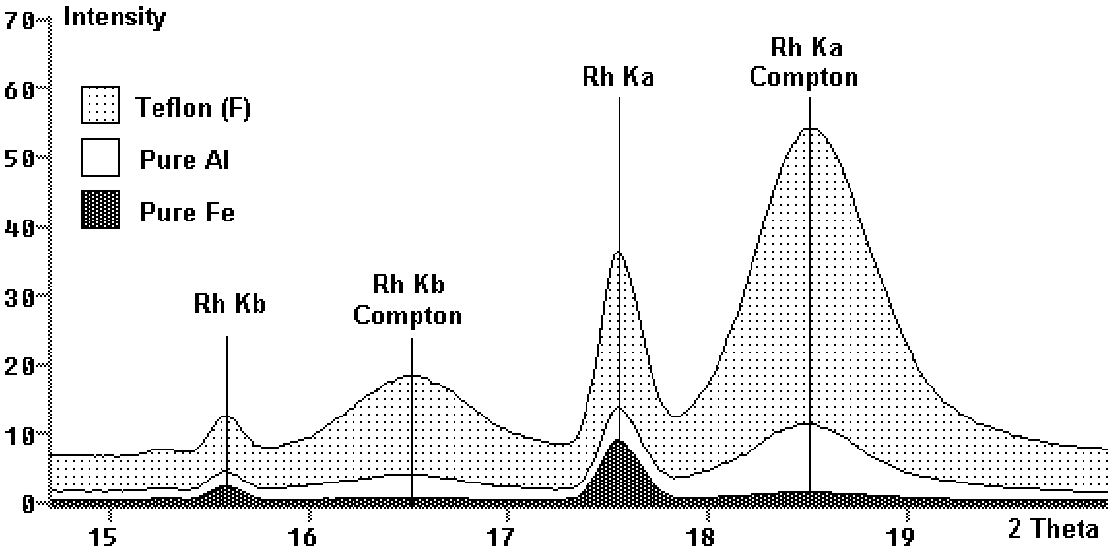


Figure 3.6

Transmission

Transmission is the counterpart of absorption. The transmission or absorption of X-rays in the matter can be described by an exponential relation. If I_O is the intensity of X-rays (of energy E or wavelength λ) incident on a sample of thickness t (in cm), then the transmitted X-ray intensity I is given by:

$$I = I_O \exp^{-\mu\,\rho\,t}$$

- μ:mass attenuation coefficient (cm^2/g)
- ρ:the density of the sample (g/cm^3)

The mass attenuation coefficient here refers to a particular wavelength in a given absorber, and is in fact the sum of two coefficients τ and σ, where τ is the *mass photoelectric absorption coefficient* and σ the *mass scattering coefficient*:

$$\mu\text{total} = \tau\text{photo} + (\sigma\text{coherent} + \sigma\text{incoherent})$$

These coefficients can be found in standard textbooks and general literature. This formulae is quite useful when we need to calculate the thickness of intensity filters, the penetration depth of X-rays in a given sample etc. Attenuation of X-rays by photoelectric absorption and coherent or incoherent scattering is essentially an atomic property. Consequently, the mass coefficients of compounds follow the simple law of weighted average, also called the additivity law. For example, a compound containing the elements *i*, *j*, *k* etc. in the proportions by weight M_i, M_j, M_k etc. has a mass attenuation coefficient $\mu = \Sigma M_i \mu_i / \Sigma M_i$ or, in terms of the weight fractions C_i, C_j, C_k etc. $\mu = \Sigma C_i \mu_i$.

Nomenclature used in XRF

The electrons in an atom do not all follow the same orbit but arrange themselves in well defined shells around the nucleus; these shells are known as K, L, M, N, O, P and Q; the K shell being nearest to the nucleus. Each shell represents an energy level, composed of different sub-levels.

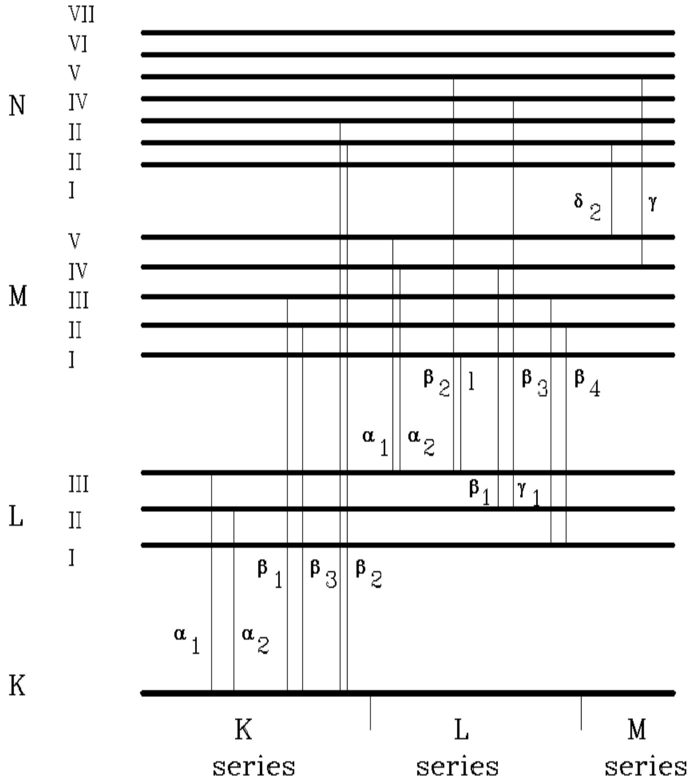


Figure 3.7

The K shell has the lowest energy and the Q shell the highest, but it is important to note that the largest energy difference between any two shells is between the K and L shells; the smallest difference is between the outermost shells. The energy of a given electronic shell depends on the atomic number and thus, it varies from element to element. Further details of atomic structure can be obtained from any standard text book of Atomic Physics. X-ray spectral lines are grouped in series: K, L, M, N etc. All the lines in a series result from electron transitions from various higher levels to the same shell.

When a K shell vacancy is filled by an electron from the L shell we get the Kα line radiation, whereas when an electron from the M shell fills this vacancy, we get the Kβ line radiation. Similarly, if a vacancy in the L shell is filled by an electron from the M shell we get the Lα line radiation, if it is filled by an electron from the N shell, we get the Lβ line radiation and if it is filled by an electron from the O shell we get the Lγ line radiation. Every element has its characteristic K, L and M series. Thus, light elements give rise to only K lines, mid range elements can emit both K and L series while the heavy elements produce K, L and M series. Thus the spectra get increasingly complex when we go towards heavier elements. In practical XRF, we select the principle lines of K and L series to measure an element although there may be other lines present. The selection of an analysis line depends mainly on the type of sample, the elements present in it, the concentration range of the elements and the excitation conditions.

Instrumentation

In X-ray fluorescence we basically distinguish between two types of instruments: the wavelength dispersive (WDX) and the energy dispersive spectrometer (EDX). Both WDX and EDX employ an X-ray source for exciting the sample. They essentially differ in the way the X-ray spectra emitted by the sample are detected. In WDX, the fluorescence spectrum is dispersed into discrete wavelengths using a dispersion device (e.g. a crystal) which are then detected using a gas proportional or scintillation counter. In EDX, one measures the entire fluorescence spectrum directly using a solid state detector such as Si(Li) or HPGe which is then processed using a multichannel analyser to obtain the information on an energy scale. The energy and the wavelength of a photon are related in the following way:

$$E = hv = hc/\lambda = 12.4/\lambda$$

The main components of an WDX spectrometer can be seen in Figure 3.10. They are: X-ray tube, primary collimators, crystals, secondary collimators and detectors. Additional components are: Primary beam filters, aperture mask and attenuation filters.

X-ray Tube

The two types of X-ray tubes which have been the subject of considerable discussion in recent years are: End-window and Side-window tubes. Today, all major manufacturers equip their instruments with the End-window tube which proves that this tube is the more versatile and the better compromise for the whole element range. Therefore, the differences in performance and construction will not be discussed here.

The tube houses filament and an anode under a high vacuum metal enclosure. The filament is heated by an electric current which then emits electrons by thermionic emission. These electrons are then attracted and accelerated towards an anode (target) when a high voltage difference (10-70kV) between the anode and the cathode is applied. Primary X-rays are produced when these electrons of high velocity collide with the target material. Upon impact, most of the kinetic energy of the electrons is converted into heat requiring the anode to be cooled efficiently. Only a small part (0.2-0.5% depending on the type of target) of the electron energy is usefully converted into X-rays.

This X-ray radiation consists of the *Continuum* or *Bremsstrahlung* or *White radiation*, the characteristic X-ray lines of the target material (K, L, ... series) and characteristic lines from any contaminants. Thus, as shown in Figure 3.8, the primary spectrum from a conventional X-ray tube consists of intense characteristic lines from the anode material (e.g. Rh) on a broad envelop of continuum.

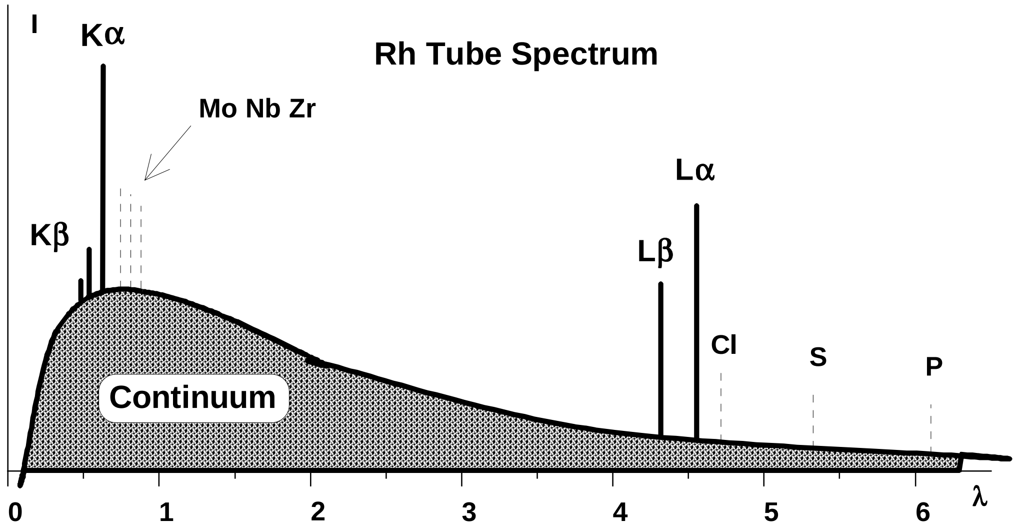


Figure 3.8

Continuum

The impact of the electrons on the target is non-selective and produces a wide range of energy transitions and consequently a continuum of X-ray emissions. In other words, stepwise deceleration of the electrons causes a continuum of emission lines.

This continuum has a short wavelength limit which depends on the accelerating voltage used and is independent of the target material. However, the intensity of the continuum increases with atomic number of the target. Figure 3.9 shows the intensity of the continuum spectrum at three different voltages for the same target material. We notice that the intensity of the spectrum increases with the voltage and the minimum wavelength limit shifts to lower wavelengths (i.e. higher energies) when we go from 20 to 50 kV.

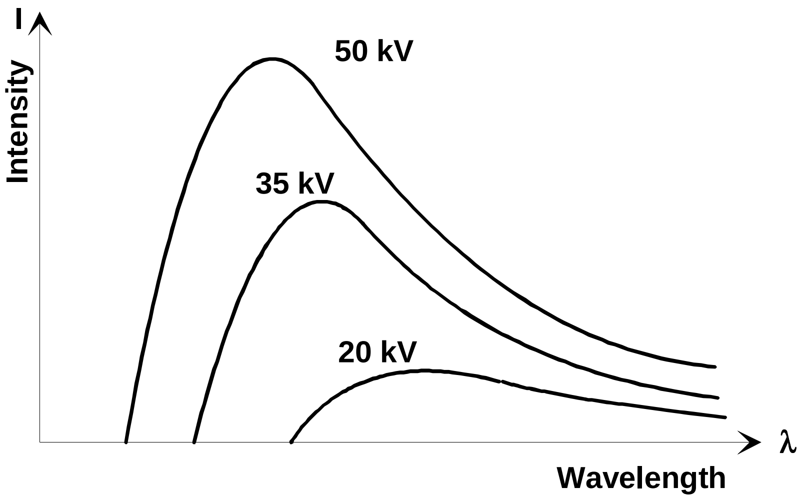


Figure 3.9

One can express the integral intensity (I_{int}) of the continuum as a function of the target material (Z, the atomic number), the high voltage (V) and the current (i) as follows:

$$I_{int} \text{ is proportional to } (i\ Z\ V^2)$$

Thus, for a given target and high voltage, the intensity of the continuum is directly proportional to the emission current.

Characteristic spectra and choice of the X-ray tube target

When the incident electrons have sufficient energy to eject electrons from the K and L shells of the target atoms, then one obtains the characteristic K and L lines of the target material. Unlike the continuum, this phenomena is selective and causes intense sharp lines of the given target element. Figure 3.8 shows these lines superposed on the continuum. The heavier the element of the target, the more intense the characteristic lines emitted. However, in practice the efficiency of a given target is dependent on the operating conditions (kV and mA).

The X-ray tube target is selected based on several factors. One of the most important factors is related to the efficiency of the target lines to excite a wide range of elements in the sample. In order to understand the efficiency, we must invoke the concept of *Absorption Edge* of an element. Every element has an absorption edge for a given series of lines. For example, an element like Mo has a K absorption edge and three L absorption edges (LI, LII, LIII). As discussed before, this is related to the binding energy of the electrons in the K shell or L shell. Therefore, when choosing a suitable target element, one must consider the position of the K and L lines of the target. If these intense target lines are close to the absorption edges of the analyte elements in the sample, they make the dominant contribution to the excitation of the sample; otherwise the continuum makes the dominant contribution. Although X-ray tubes are available with different target elements like Cr, Cu, Mo, Rh, Au and W, it is difficult to find a favourable target for all analytes. Consequently, continuum is frequently relied on for excitation. However, one can optimise the choice of the target element by considering the most common parts of the periodic table. Majority of the spectrometers are equipped with Rh X-ray tubes since, the Rh K lines can excite the mid range elements while the Rh L lines can effectively excite the lighter elements (see Figure 3.8). When an analyte contains more frequently medium to heavy elements, W target tubes are selected occasionally.

Spectral Line Interference

The primary spectrum may not only contain the lines from the target element but also those from impurities in the target, contaminants like Fe, Cr or Cu from various parts inside the tube. Consequently, the background level at these elements may be unusually high. Thus, care must be taken to minimise the contaminant lines from the tube. In addition, the target lines scattered by the sample may not permit the analysis of the same element in the sample. Sometimes, primary beam filters are used to filter out the characteristic lines from the tube if the same element is present in the sample or overlapped by a tube line.

Window thickness

X-ray tubes are usually sealed with a beryllium window since beryllium has the lowest absorption for X-rays. The intensity of the low energy X-rays (also referred to as *soft X-rays*) in the continuum depends strongly on the window thickness. Most standard X-ray tubes are fitted with Be window of 75µ or 50µ. The thinner window is more efficient for the excitation of the light elements. For example, transmission of soft X-rays (useful to excite light elements) improves by at least 20% when the window thickness is reduced from 75µ to 50µ.

Dispersion

A dispersion device is the heart of an WD XRF spectrometer. It contains the following parts: Primary and secondary collimators (flat crystal geometry) or entrance and exit slits (focussing crystal geometry), crystals and detectors. One can classify the XRF spectrometers into two categories depending on the X-rays optics involved.

Sequential Instruments

Traditionally, sequential instruments employ a "Parallel beam geometry" or a "flat crystal geometry" allowing the angles of the crystal and the detector to be selected with a device called *goniometer* (a rotating frame on which the crystal and detector have a Theta-two Theta coupled movement). The geometry of such a spectrometer with the main components is shown in Figure 3.10 and a sketch of the ARL goniometer in Figure 3.12.

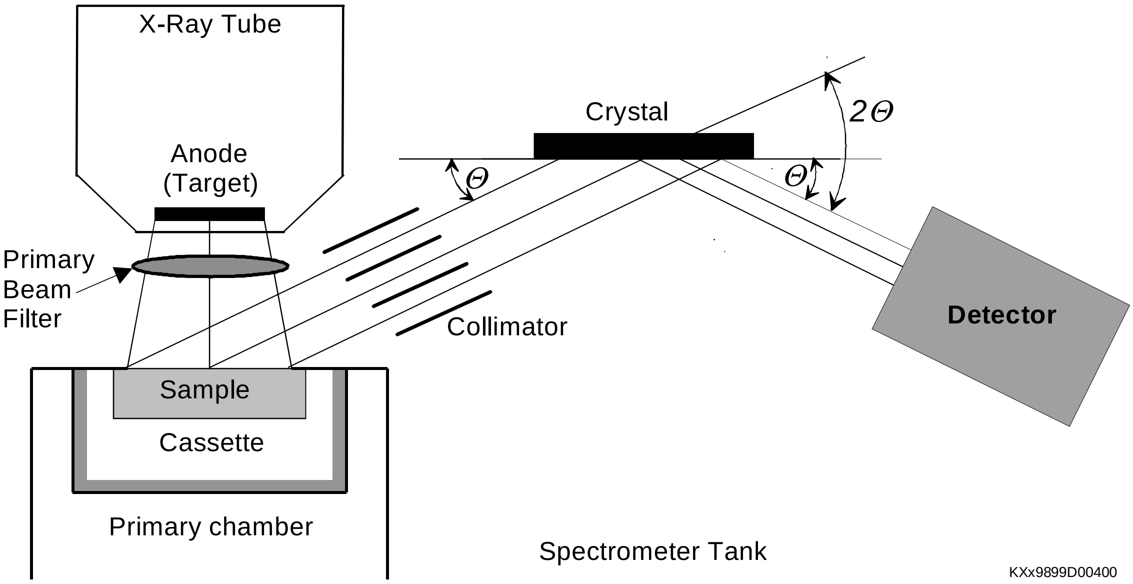


Figure 3.10

Simultaneous Instruments

In Figure 3.11 the simultaneous type (or fixed channel type) of spectrometer is shown where one makes use of a "Focussing beam geometry". Here, instead of flat crystals, we use curved crystals; the radius of curvature being a function of wavelength range of the spectral lines to be measured. Further, collimators are replaced by slits whose width and height are again determined for each wavelength. This type of configuration is obviously used to measure a predetermined element since all the parameters are fixed based on the wavelength of the spectral line to be measured. Thus, a simultaneous instrument may house as many as 30 such fixed channels, each dedicated to measure one line. ARL's 9800 is such an instrument.

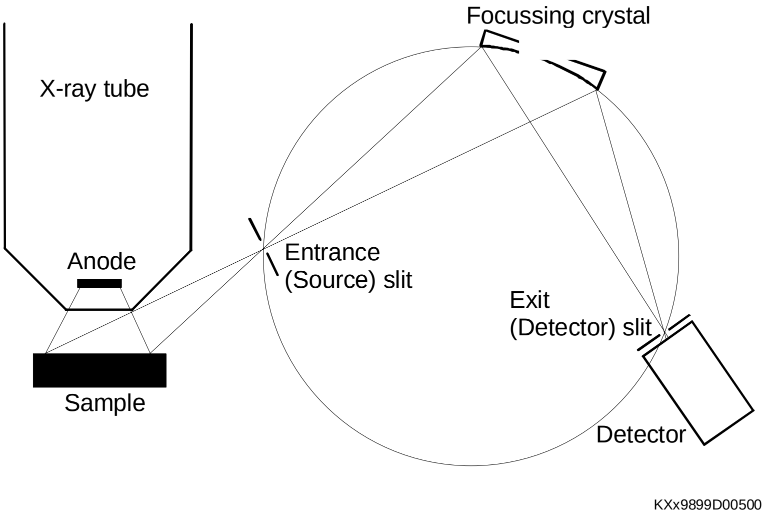


Figure 3.11

Instrument Components

We will now discuss the basic components involved in the wavelength dispersive spectrometer (both sequential and simultaneous types). As mentioned earlier, crystals are the essential part of the spectrometers. In the following discussion, several concepts related to them are presented.

Goniometer

The exact positions of the crystal and detector are read by two optical encoders based on Moiré fringes. This gearless system ensures an accurate and reproducible positioning of crystal and detector as compared to the mechanically coupled (gear) system. Thus, a sequential measurement consists of positioning the crystal at a given Theta and the detector at 2 Theta and counting for certain amount of time. Then the crystal and detector are rotated to a different angle for another line etc.

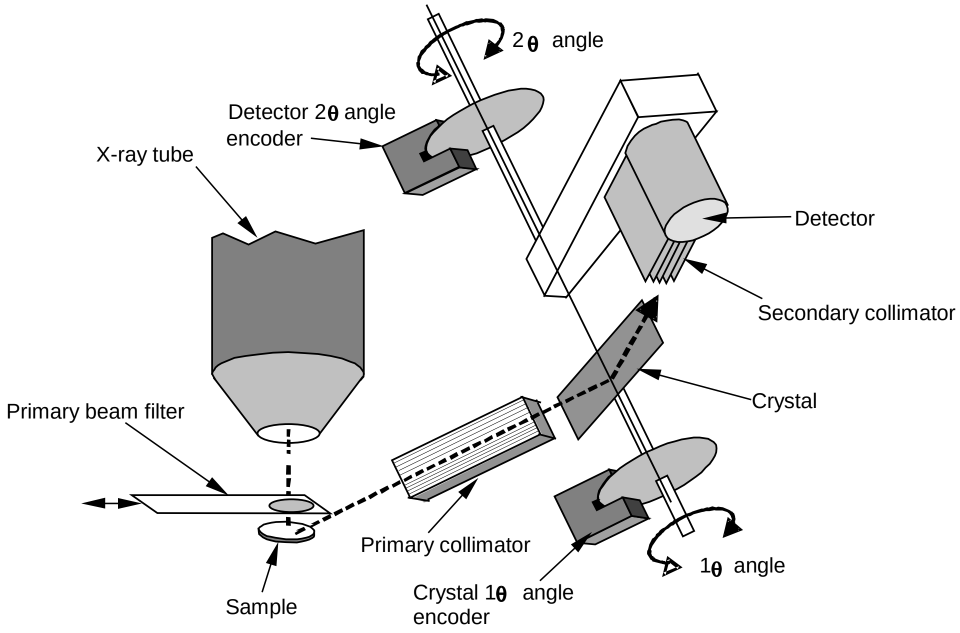


Figure 3.12

Thus a goniometer renders qualitative and semi-quantitative analysis of the sample where one has to scan a whole range of wavelength to identify the elements present in the sample. In a quantitative analysis, where the elements present in the sample are known, one can set the crystal and detector on a peak position of the line and count for a fixed time, then move off the peak to measure background points and subsequent elements. A secondary collimator is placed between the crystal and the detector to guide (collimate) the diffracted beam into the detector and also to limit unwanted radiation getting into the detector.

Collimators

The primary and secondary collimators are usually made of a series of parallel blades. The length and spacing of the blades determine the angular divergence admitted by the collimator. This angular divergence together with the crystal "rocking curve" (the width of the diffraction profile) determine the final resolution of the spectrum. One can improve the resolution by closing the collimators to minimise the divergence. But then, the photon flux across the collimator and hence the intensity decreases.

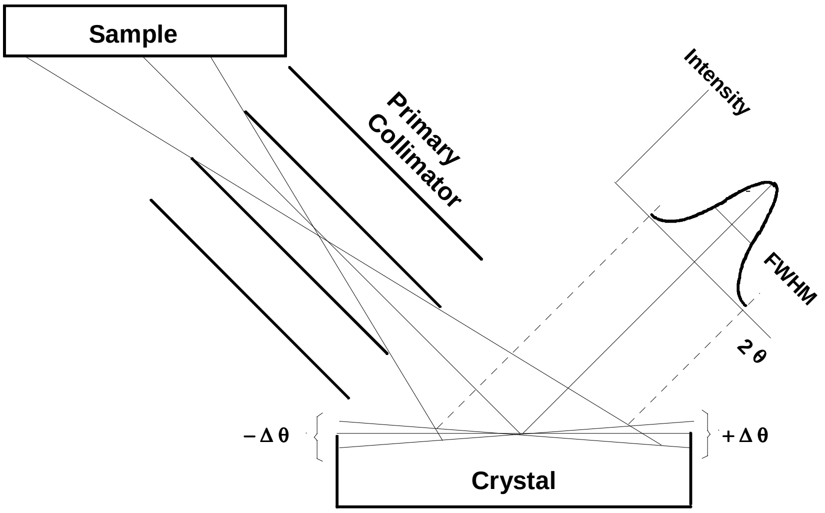


Figure 3.13

Thus, a compromise between the final resolution (necessary to avoid important spectral overlaps) and the sensitivity (related to the intensity) is made. Generally, the collimators are adopted in accordance with the crystal's intrinsic divergence, which varies from one type of crystal to another. Some of the crystals offer excellent resolution while others have a very wide diffraction profile. For this purpose, ARL's sequential spectrometers offer up to four types of collimators. Fine collimators are used for most of the heavy elements, Medium for the mid range elements and Coarse for the light elements.

Crystals

A crystal may be defined as a solid, composed of atoms arranged in a periodic pattern in three dimensions. In a crystal lattice, the plane in which atoms are in a row is called the crystal plane. The Figure 3.14 shows a set of crystallographic planes in a cubic crystal.

The planes are identified using Miller indices (hkl). For example, in the figure are shown three such planes: (100), (110) and (111). The interplanar spacing (the distance between any two adjacent planes of same type) is denoted as d. In XRF, we generally refer to the 2d values of the crystals since we will be using the 2d values in what follows.

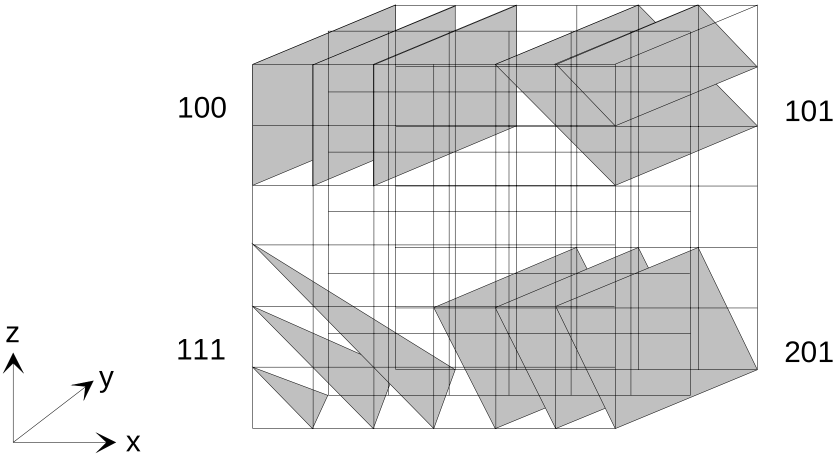


Figure 3.14

Diffraction

The crystal planes reflect X-rays just like mirrors reflect light. The main difference is that crystal planes reflect X-rays only when certain conditions are met. This selective reflection is known as diffraction.

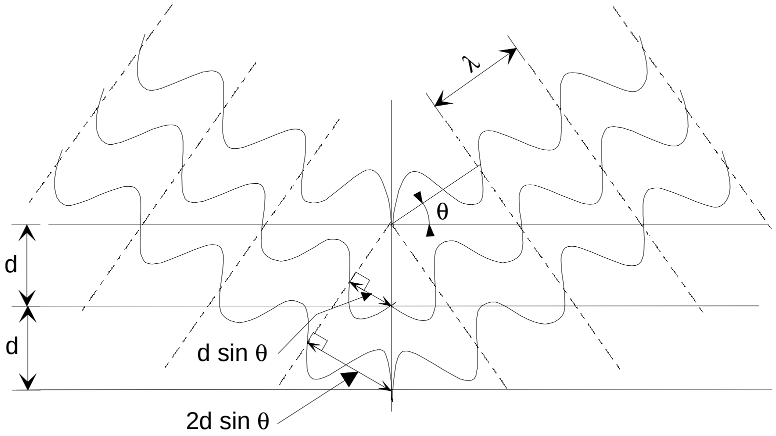


Figure 3.15

Diffraction can be considered as constructive interference, in the sense that when the X-ray photons (considered as waves) are reflected coherently, they undergo a constructive superposition of the waves. Only then, the X-rays are reinforced in their amplitudes while in all other cases, they interfere destructively. The most important condition is the so called "Bragg's Law of Diffraction". Bragg's law says that if a lattice plane having an interplanar spacing of d reflects an X-ray wavelength of λ, this wave differs in path length by a distance of ($2d \sin \Theta$) from the wave reflected by the adjacent plane. This phenomenon is shown in the Figure 3.15. When this path length differs by a whole number of wavelengths, the reflected X-rays are reinforced, otherwise they annul each other. We can sum up this with the following Bragg equation:

$$n\lambda = 2d \sin \Theta \quad \text{Bragg's law}$$

where: n: an integer (1,2,3...) called "Order of Diffraction"

d: interplanar spacing of the crystal plane used (Angstroms)

Θ: Bragg angle or diffraction angle (degrees)

λ: wavelength of the spectral line (Angstroms)

Thus, we see from this relation that for a given crystal plane and for a given order of diffraction, each wavelength in the incident XRF spectrum is diffracted at an unique angle. The maximum wavelength a crystal plane can diffract is 2d itself (when $\sin \Theta$ takes the maximum value of 1). Therefore, one requires crystals or crystal planes with different 2d values in order to cover different wavelength ranges across the periodic table. A number of crystals suitable for diffraction are available today. Some of them are inorganic (like LiF) while others are organic (like PET) in nature.

For further information on diffraction analysis or the integrated XRD system of the ARL 9800, please refer to the brochure "ARL 9800 - Integrated XRD system".

Multilayer Structures

As we go from short wavelengths (lines from heavy elements) to long wavelengths (mid range elements), the 2d spacing of the crystal used also increases in order to be compatible with Bragg's law and hence the angular range. However, as we go to very long wavelengths (the K lines from light elements such as Beryllium to Magnesium), we must use crystals with increasing 2d spacing (>10 Angstroms). Most of the natural crystals do not have such high 2d spacing. Historically, some of these light elements were measured with synthetic crystals like lead stearates and some kind of soap crystals which suffered from several drawbacks (radiation damage, stability under vacuum etc.). Today, new stable structures called "Layered Synthetic Microstructures", (in short LSM) are available. These are multilayers of a light element (like B, C ..) and a heavy element (Mo, Ni, V, ..) alternating with a spacing of 2d deposited on a Si substrate.

The advantage with these man made structures is that one can try to optimise the combination of the light and heavy elements, the thickness of the layers, and the 2d spacing in order to get the best possible device for a given element. The result of these new developments is that we now have a family of multilayers allowing one to measure elements from Beryllium through Magnesium. We use essentially three or four types of these multilayers along with the natural crystals.

Reflectivity and Resolution

In addition to having a convenient 2d spacing, the crystals should have good diffraction efficiency, i.e. the ratio of diffracted photons to the incident photons. Some crystals like LiF have excellent reflectivity while others suffer from a poor diffraction efficiency. We are generally interested in the peak reflectivities and not the integral reflectivities. The variation of reflectivity when the crystal is "rocked" around the diffraction peak by small angles is called the "rocking curve" of the crystal. The height and the width of this curve give us the indications on the total/integral reflectivity, the peak reflectivity and the width of the diffraction profile. These characteristics depend on several factors; the most important being the mosaic structure of the crystal. If the crystal is nearly perfect (i.e. no defects in the crystals, no surface damage, no doped impurities etc.), then it will have a very narrow rocking curve and rather weak peak reflectivity. This is attributed to a phenomena called "Self-extinction" in the crystals. Self-extinction is essentially due to the fact that the diffracted waves from the inner planes in the crystal are reflected back into the crystal in the absence of any defects (deviation from perfect periodicity). Thus, most of the crystals used in our spectrometers are treated to induce a mosaic structure and reduce the effect of self-extinction. Therefore, the diffraction efficiency can be increased by various treatments such as abrading, quenching, elastic bending, impurity-doping and so on. Of course, it must be done in a controlled way in order not to broaden the diffraction profiles. Otherwise, we may have undesirable effects on the resolution. As a general indication, the multilayers, which are pseudo-crystalline structures suffer from very broad diffraction profiles compared to the natural crystals. Finally, it may be remarked that:

- Some of the crystals have an unusually high reflectivity at particular wavelengths. InSb crystal is one such example. Although PET crystal is used as a general purpose crystal (on a goniometer) to measure Al, Si, P, S and Cl, the InSb crystal has more than two times the reflectivity for SiKα.
- Further, some of the crystals reflect only odd orders of diffraction, i.e. 1st, 3rd, 5th etc. Germanium crystal is an example. It is used for its higher reflectivity to measure P, S and Cl and does not have 2nd order, 6th order ... reflections. This may be an added advantage in cases where we need to suppress higher orders.
- Some crystals may emit their characteristic fluorescence. This may be seen in some cases as a high background on the corresponding elements.

Dispersion Power

Crystals also have an important property called "Dispersion power", i.e. the capacity of a given crystal plane to separate the lines most effectively. The dispersion power depends on (1) the 2d spacing of the crystal plane used (2) the Bragg angle and (3) the order of diffraction. This relation is given as follows:

$$d\Theta / d\lambda = (n/2d\cos \Theta)$$

Thus, we see that:

- The smaller the 2d spacing, the better the dispersion.
- The higher the diffraction angle, the higher the dispersion.
- And higher orders of diffraction have increasing dispersion.

An example of this relation is shown in the Figure 3.16 where the same spectrum has been recorded with three different crystal planes. In this figure three different crystal planes were used: LiF200 (2d = 4.028), LiF220 (2d = 2.848) and LiF420 (2d = 1.802). We notice that the lines are best separated when LiF420 is used. However, the peak intensities drop significantly when we go from LiF200 to LiF220 to LiF420 crystal planes. Thus, one must keep in mind that higher dispersion may sometimes mean lower peak intensities.

The final point is the observation of increasingly poor dispersion when we move towards light elements. Since the 2d spacing increases (inevitably), crystals or multilayers in this range have poorer dispersion.

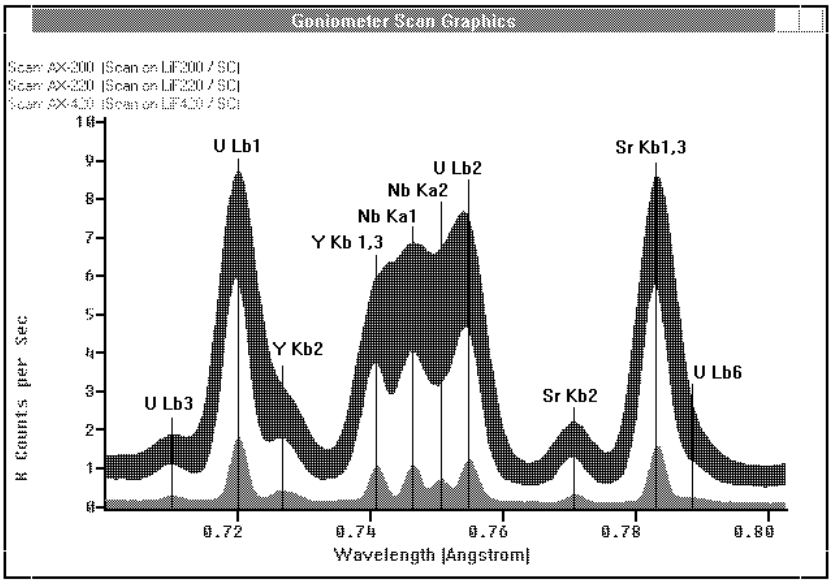


Figure 3.16

Stability

Stability of the crystal is another important factor for a reliable and reproducible measurement. Crystals may undergo changes due to temperature shifts, they may suffer from radiation damage and they may be affected by chemical pollution in the spectrometer. Thus, care must be taken to maintain them at constant temperatures and protect them from being contaminated. Some crystals are more sensitive to temperature changes than others. Essentially, when the temperature changes, the crystal planes, due to thermal expansion, may attain a slightly different 2d spacing. This obviously shifts the Bragg angle for a given wavelength and thus one may not be measuring at the peak position any more.

PET is one of the most sensitive crystals with respect to temperature particularly at higher Bragg angles. ARL's spectrometers are equipped with thermal stabilisation circuits which maintain the spectrometer temperatures within +/-0.5 degrees. In addition, the crystals are maintained at constant temperatures using a local temperature control system. Thus, the peak shifts due to thermal changes are excluded to a great extent.

Flat crystals vs curved crystals

Most of the discussion so far is valid for both flat crystals as well as curved crystals. We have seen earlier that curved crystals are employed in fixed channel instruments in contrast to flat crystals which are used in sequential instruments.

There is an exception for multilayer crystals used for Na and Mg monochromators for which flat or curved crystal are used. Flat crystal monochromators are used with FPC detectors to optimise sensitivity and curved crystal monochromators are used with sealed detectors in case the instrument should run without a P10 gas supply.

There are different ways in which a crystal can be curved to obtain a perfectly focussing or semi-perfect focussing beam onto the detector. Among the most common geometry, logarithmically curved crystals are used currently in ARL' spectrometer.

Higher orders of diffraction

It may be seen from Bragg's law that higher orders of diffraction from heavy elements may superpose on the first order diffraction of lighter elements. For example, first order of P Kα (6.16 Angstrom, second order of

Ca Kβ (3.09 Angstrom) and third order of Gd Lα (2.05 Angstrom) will all be diffracted at nearly the same diffraction angle when the same analysing crystal is used since they all satisfy the Bragg's law at the same Θ angle. Thus, superposition of different orders of diffraction can cause some unwanted overlaps. However, as we will see below, most of the modern spectrometers are equipped with a Pulse Height Discriminator (PHD) which analyses only those pulses with an amplitude above a threshold and fall in a given window. Thus, one can reduce the contribution from higher orders by a proper setting of the PHD parameters.

Resume

We will conclude this chapter by summarising the important points:

- Wavelength dispersive X-ray spectrometers employ crystals and multilayers for dispersing the polychromatic fluorescence spectrum into monochromatic wavelengths.

- Flat crystals are used in conjunction with collimators in sequential spectrometers (single channel) where a scanning mechanism is provided (Goniometer). The crystal and the detector are coupled by a theta-two theta relation. ARL' s goniometer has a gearless mechanism to ensure precise and reproducible positioning of the crystal and detector. The flexibility and the wide scan range available with the goniometer render the sequential instruments versatile and useful for exploratory (qualitative and semi-quantitative) work.

- For Na and Mg, and depending on the detector, multilayer crystals can be used for monochromators using a flat or curved crystal geometry.

- Curved crystals are used in conjunction with slits in the simultaneous (multi-channel) instruments. These channels (or monochromators) can be constructed once the elements (or the lines) are predetermined. The focussing X-ray optics (thus high intensity and excellent resolution) used, the rapidity and the stability of these instruments make them ideal tools for production control and quality control applications.

- Both types, flat crystals or curved crystals, diffract X-rays according to the Bragg's law where the wavelength and theta are related uniquely for a given crystal plane.

- Different crystals are used for different spectral domains depending on their 2d spacing, dispersion power, reflectivity and stability.

- The resolution of a spectrometer is its ability to distinguish two spectral lines of nearly same wavelength or closely spaced lines. In WDX, resolution is a combined effect (convolution) of angular divergence admitted by the collimators or slits, the rocking curve (the width of the diffraction profile) of the crystal, the dispersion power and to a much lesser extent, the detector resolution. Thus, the combination of primary collimator and crystal vary from one spectral region to another. In contrast to the WDX spectrometers, the resolution in EDX spectrometers is predominantly determined by the energy resolution of the detector.

Detection

Detectors used in most of the commercial WDX spectrometers can be classified into two categories:

(1) Gas filled proportional counters for long to intermediate wavelengths (FPC, Multitron and Exatron).

(2) Scintillation counters for short wavelengths.

Gas filled counters

Gas filled detectors are again subdivided into two types: Flow Proportional Counters (FPC) and Sealed Detectors. FPCs have a continuous flow of gas and the pressure inside the detector is regulated. They are generally closed with Aluminium coated thin polypropylene windows of the order of 1-2µ thick. The purpose of such a thin window is essentially to increase the transmission of long wavelength X-rays.

Sealed detectors, referred to as Exatrons and Multitrons, have 25-200 micron beryllium windows. FPC’s are used for light elements (from Beryllium through Copper in general) on a goniometer. Sealed detectors are employed in fixed channels although small sizes FPCs are used in the fixed channels for light elements. The principle of operation is the same in both the types. The gas detector in its simplest form consists of a hollow metal cylinder (acting as cathode) carrying a filament (metal wire of about 50-75 microns diameter acting as anode). A high voltage is applied across the two electrodes. The cylindrical casing is earthet. The Figure 3.17 shows the Multitrons and Exatrons.

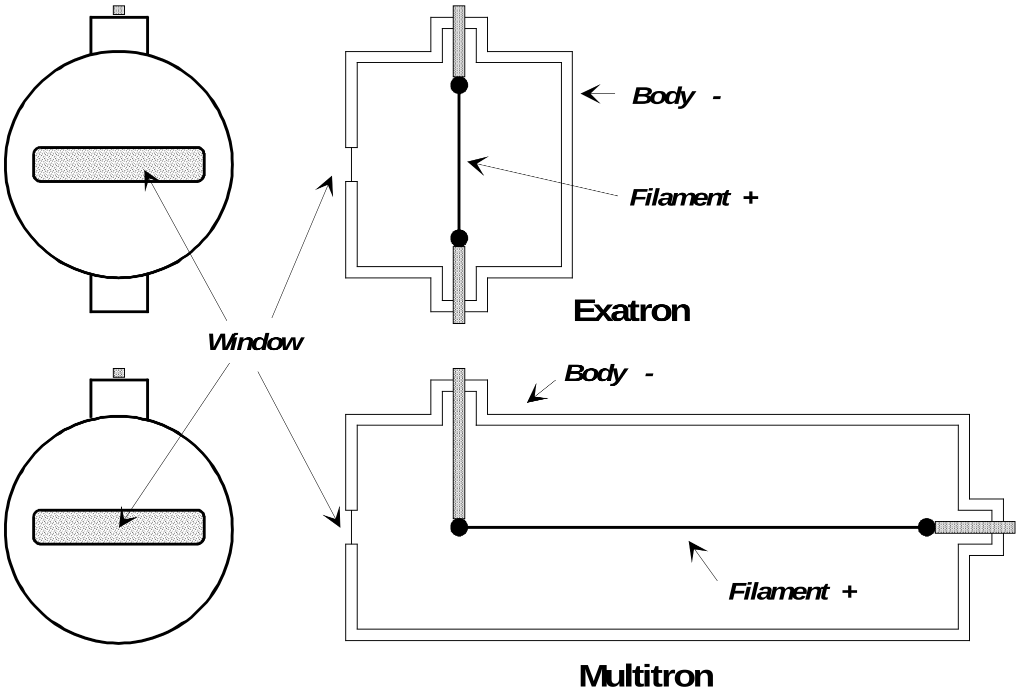


Figure 3.17

Primary Ionization

The detectors are filled with rare gases He, Ne, Ar, Kr and Xe are used for this purpose. They are mixed with a quench gas (methane, for example). An X-ray photon entering the detector ionizes the gas and creates an electron-ion pair. The number of electron-ion pairs depends on the type of gas and the energy of the incident photon. As an example, Ar gas requires an effective ionisation potential of about 26 eV for one electron-ion pair. Thus, if a Cu Kα photon (wavelength = 1.542 A, energy = 12.4/1.542 = 8040 eV) entered the detector, each photon (also called an event) will produce 304 electron-ion pairs. If a photon with twice this energy enters the detectors, it will (ideally) produce twice the number of pairs. Thus, for a given detector gas, if all the incident photons produce ionized pairs, then the number of pairs is proportional to the energy of the photon. This is why these detectors are called Proportional counters. Thus, if the electrical charges produced are counted, we would have measured the intensity of a given spectral line.

The number of electron-ion pairs produced during the primary ionisation process is not sufficiently high to be detectable. One needs to amplify the signals considerably before they can be measured with good signal to noise ratio. This is accomplished by a process called "Avalanche" or "Gas Amplification" and is explained in the next section.

Avalanche

Consider an X-ray photon entering the active volume of the detector. It produces primary electron-ion pairs along its path until all its energy is expended. When the detector is under high voltage, the electrons are pulled towards the filament and the ions are attracted towards the body of the detector. As electrons approach the filament, they are subject to stronger electric field closer to the filament which accelerates them further. As shown in the Figure 3.18, when the applied potential is low, the electrons recombine with the positive ions before they reach the anode wire. This is the region of unsaturation. As the applied potential is increased, the recombination is overcome completely and then all the primary electrons reach the anode. Ionization-chambers operate in this region. Since there is no further secondary ionization, the gas gain remains as 1. When the potential is high enough to accelerate the electrons which collide with other gas atoms to initiate secondary ionization, one begins to see a substantial gain in the number of charge carriers. This order of multiplication increases as the electrons approach closer to a few diameters of the anode wire.

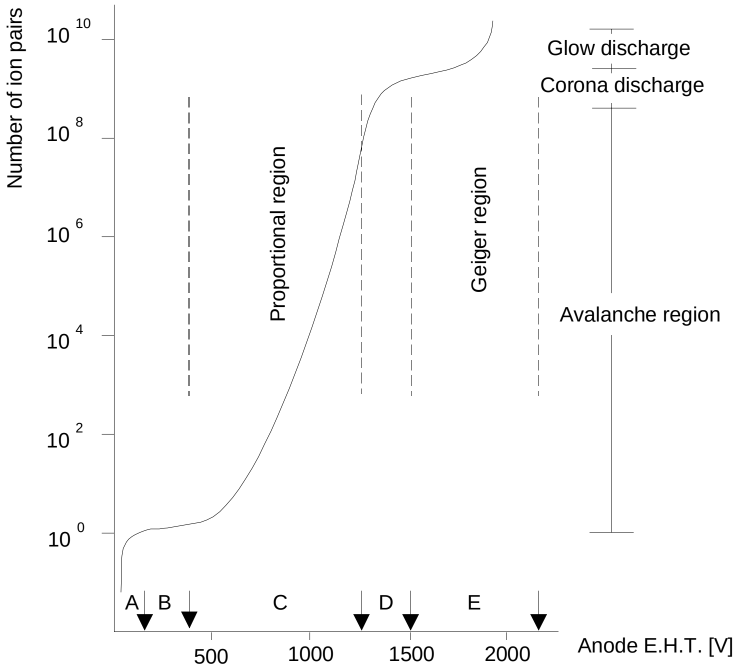


Figure 3.18

In the proportional-counter region, each primary electron initiates only one avalanche and the avalanches are free of any interaction. Thus the final number of electrons after amplification is still proportional to the number of primary electrons which in turn is proportional to the energy of the photon entering the detector. Most of the detectors used in XRF spectrometers function in the region of proportionality. When the potential is increased beyond this region, the proportionality deteriorates as the same electron may initiate multiple avalanches. When the applied potential is so high that avalanche is generalised, the gas ions and atoms are excited. Secondary electrons may be generated at the anode and electrons may be expelled at the cathode. In other words, one finds a general discharge in the entire detector. Thus, there is no longer any proportionality between the incoming photons and the output pulse of the detector. Geiger-Muller counters operate in this region. Finally, a further increase in the potential triggers a continuous and sustained discharge and the detectors begin to glow. At the end, an arc discharge occurs between the cathode and the anode wire and detector is likely to get destroyed.

In the proportional region, which is of interest here, one attains a gas gain of the order of 10^5-10^6 permitting one to obtain good signal to noise outputs. One of the most common gas fillings is the mixture of 90% Ar + 10% CH_4 called P10 gas. The purpose of methane addition is essentially to quench the phenomena of avalanche at one stage. The quench gas molecules also get ionized along with the Ar atoms. When they are dispersed in the detector's active volume, they contribute to the recombination of the electrons and ions when the avalanche proceeds in an uncontrolled manner.

Characteristics

Finally, the following properties of the detectors are relevant in practical XRF.

- Resolution of the detector is expressed as *Resolution = (W / V) x 100* where W is the peak width (of the energy profile) at half-width and V is the maximum of the pulse amplitude distribution.

- Stability of the detectors depends on the cleanliness of the anode wire. If the gas-fillings are not pure enough or small particles get deposited on the anode wire, they may distort the local electric field (by changing the effective diameter of the anode wire). This may change the gas gain factor causing unstable detection.

- Dead time of the detectors is a more important problem in practical X-ray spectrometry. Dead-time can be defined as the time during which, after a photon has initiated the ionization, the detector can not process the next event. As we have seen above, the incident X-ray photon produces a series of ion-electron pairs which are multiplied in the avalanche process.

  The time elapsed between the entry of the photon and the avalanche production may be of the order of 10^{-7} seconds. The electrons rapidly move towards the anode while the ions (being heavier) move slowly towards the metal body of the detector. This causes a positive ion sheath around the anode wire which prevents further avalanches to occur until its dissipation. Thus, the detector is said to be dead during this inactive period. In practical terms, this means that the photons entering the detector during the dead time are not counted since the detector has not totally recovered from the previous event. At high counting rates (high photon flux) the loss of counting events can be reasonably high.

  Most modern spectrometers correct this loss mathematically. The dead time can be estimated using different photon counting rates. Since the output signal is proportional to the number of photons, any deviation from linearity indicates the extent of dead time.

- Escape peaks in the detectors are caused by the X-ray photon whose energy is greater than the absorption edge of the counter gas. For example, if the counter contains Ar gas, some of the photons (say with an energy E from a given element) may induce Kalpha transitions in Ar and lose their energy in the primary process. Thus the pulse amplitude distribution contains not only the pulse amplitude from original photons of energy (E) but also additional pulses with an amplitude corresponding to the difference in the energy E - E(ArKα).

  Escape peaks may not represent a serious problem as long as they do not cause overlaps on other lines from the sample. In some cases, the threshold of the pulse height discriminator is fixed such that the escape peaks are not counted at all. This may not be a good practice since the intensity of the escape peak is also contributed by the photons from the same spectral line.

Scintillation Counters

Scintillation counters operate on an entirely different principle compared to the gas filled detectors. They consist of two essential parts: a scintillating material (called phosphor, usually a single crystal doped with an activator) and a photomultiplier. Scintillating crystals like NaI:Tl+ have an interesting property that when the X-ray photons are incident on such a crystal, they emit visible (in the present case, blue) light. Thus, they convert X-ray photons into visible photons. The Figure 3.19 shows the construction of a scintillator counter.

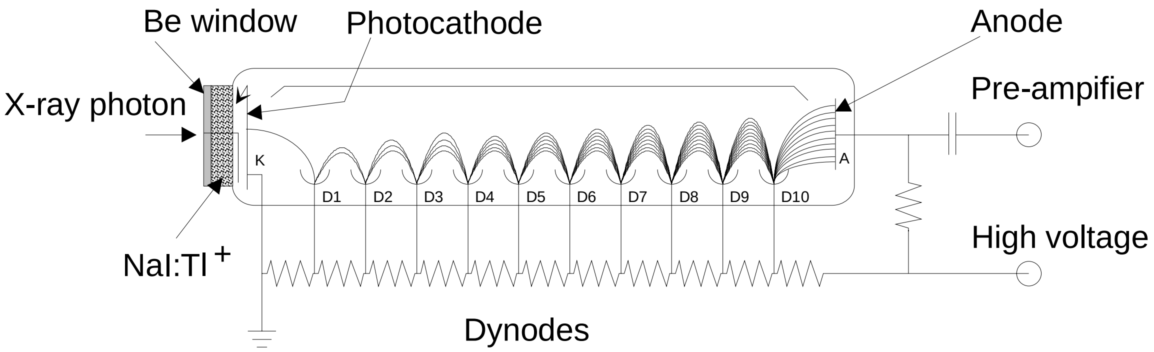


Figure 3.19

The scintillator crystal is optically coupled to an appropriate photomultiplier tube and the whole assembly is sealed using a beryllium window. Sealing is necessary because the NaI:Tl+ crystal is hygroscopic in nature and should be protected from humidity. When the blue photons from the scintillator crystals are incident on the photocathode of the PM tube, they release photoelectrons.

The PM consists of a number of positive electrodes called dynodes which are arranged at an increasingly positive potential. Thus the photoelectrons get accelerated successively by the dynodes and release more photoelectrons. Thus, the process of multiplication continues until the last dynode. The end result is an internal amplification of the order of 10^5-10^6 depending on the number of dynodes and high voltage applied to the PM tube.

Thus, a scintillator is a solid state detector where X-ray photons are converted into light photons and detected by a conventional photomultiplier. The escape peak phenomena and the dead time are also observed in scintillator detectors. Escape peaks may occur when the incident X-ray photon can eject a K or L shell electron from the iodine in the phosphor material. Dead time of the scintillation counters is somewhat longer than that of the gas proportional counters.

Pulse Height Discriminator (PHD)

Both gas detectors and the scintillation counters used in XRF are proportional detectors, i.e. the energy of the incident X-ray photon determines the size of the voltage pulse produced by the detector. A pulse height discriminator makes use of this property in order to select only a narrow range of voltage pulses, thus rejecting all those unwanted pulses. X-ray photons of different orders of diffraction may enter the detector if their Bragg angles coincide. As shown in the Figure 3.20, the height of the pulses from different orders of diffraction is not the same. The pulse height analyser includes adjustable upper and lower electronic thresholds forming a "window". This window only accepts pulses from the detector with amplitudes greater than the lower threshold and smaller than the upper threshold. These pulses can pass through the window into the output amplifier and all others are rejected. Thus, by a proper setting of threshold and window, one can significantly reduce or even eliminate the contribution from higher order diffraction lines. This setting also helps in reducing the background radiation.

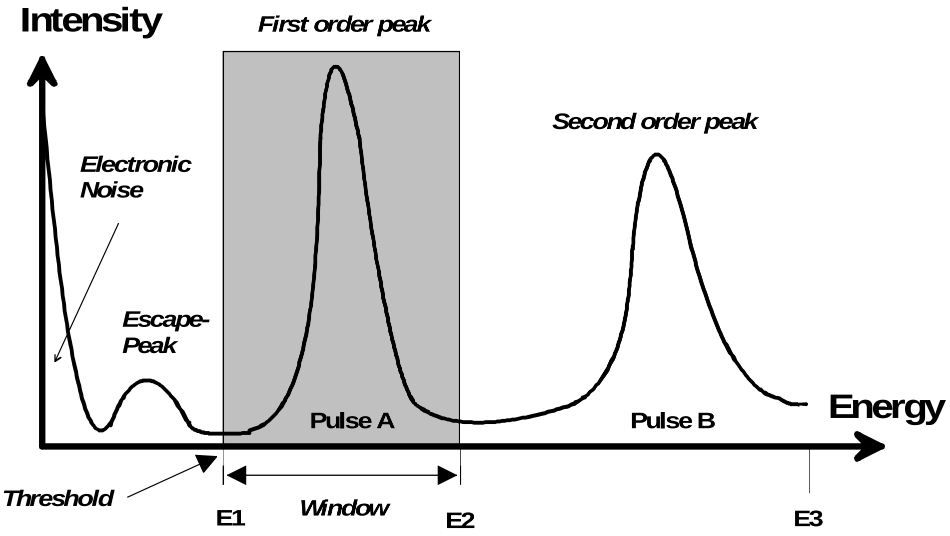


Figure 3.20

As shown in the Figure 3.21, the incoming pulses A, B are from physically overlapping spectral lines which are seen by PHD to have differing amplitudes. To receive pulse B, the window is adjusted to accept the amplitude E2 - E3; for pulse A the window is set to E1 - E2 when pulse B is too high and is rejected.

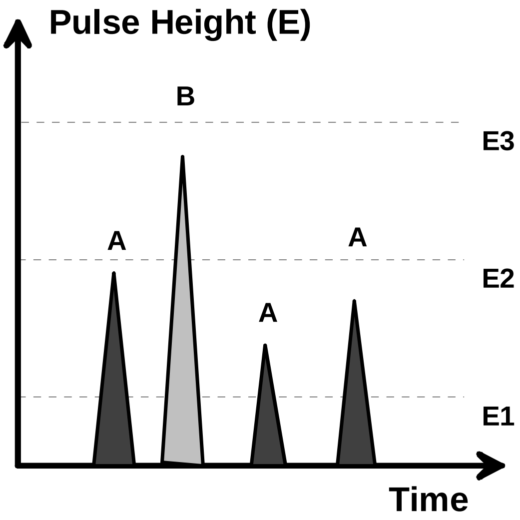


Figure 3.21

Exit

Final output

Finally, the output from the amplifier or pulse height analyser must be interpreted. One uses a scaler, which gives a strict pulse-by-pulse count as received from the pulse height analyser. However, the preferred method is to integrate the pulses as received in an electronic integrator (a scaler and timer). If N represents the number of counts in an integration time of t seconds, then the count rate is simply N/t counts per second (cps). Thus the output signal from the spectrometer is, for most practical purposes, presented in terms of counts per second. This value of intensity is then converted into concentration using different methods (calibration, empirical coefficients method, Fundamental Parameters method etc.). In a qualitative analysis, the intensity (cps) is directly displayed on a screen as the goniometer scans across different Bragg angles, resulting in a spectrum.

4

INSTRUMENT PREPARATION

4 INSTRUMENT PREPARATION

Switch on Instrument

On the back side of the instrument:

- Switch MAIN breaker on.
- Switch X-RAY GENERATOR breaker on.

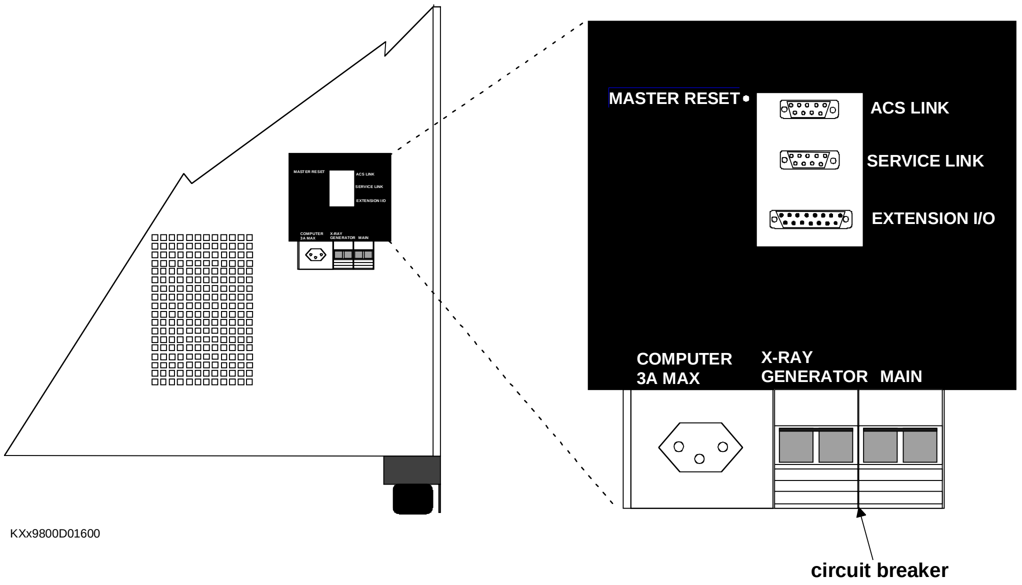


Figure 4.1

On the front of the instrument:

- Turn the red emergency button clockwise on.

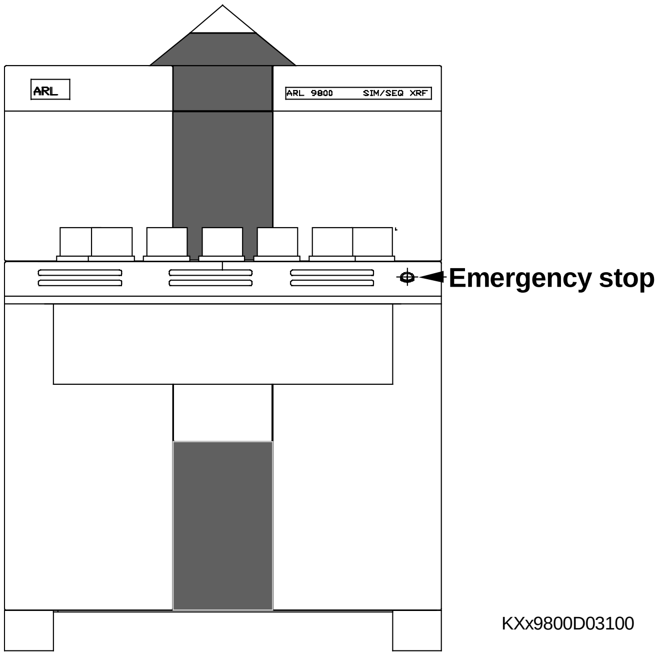


Figure 4.2 Front view with 12 Position Sample Changer

Switch on Instrument with X-Y Sample Changer

On the back side of the instrument (see Figure 4.1):

- Switch MAIN breaker on.
- Switch X-RAY GENERATOR breaker on.

On the front of the instrument:

- Turn the red emergency button clockwise on.

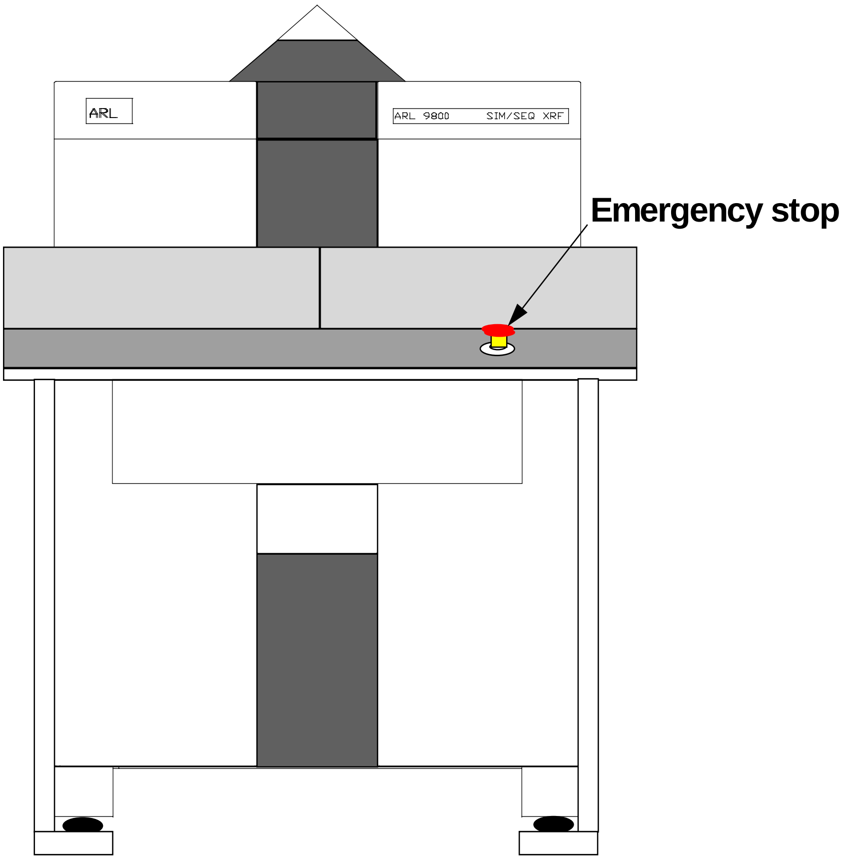


Figure 4.3 Front view with X-Y Sample Changer

Switch on Instrument with Manual Loading or SMS Automation System

On the back side of the instrument (see Figure 4.1):

- Switch MAIN breaker on.
- Switch X-RAY GENERATOR breaker on.

On the front of the instrument:

- Turn the red emergency button clockwise on.

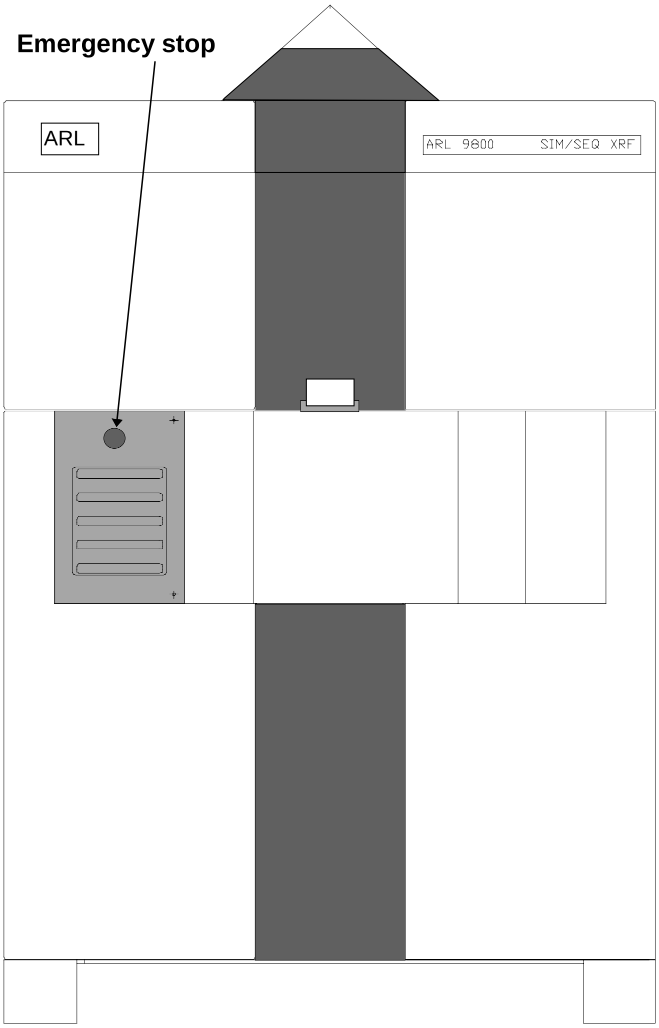


Figure 4.4 Front view with Single Position Loading

Power-up Sequence

The power-up sequence of the ARL 9800 is done to reset all microprocessors and the mechanical parts.

- Make sure that the computer, the computer display (video terminal) and the printer are correctly plugged.
- Switch on the computer.
- Wait for the computer to have the Windows operating system booted.
- Switch on the ARL 9800 with the main breakers.
- As soon as the power has been switched on, the displays of the ARL 9800 will show the following information step by step.

Note: Depending on the configuration, the displayed information may vary.

- **Step 1**

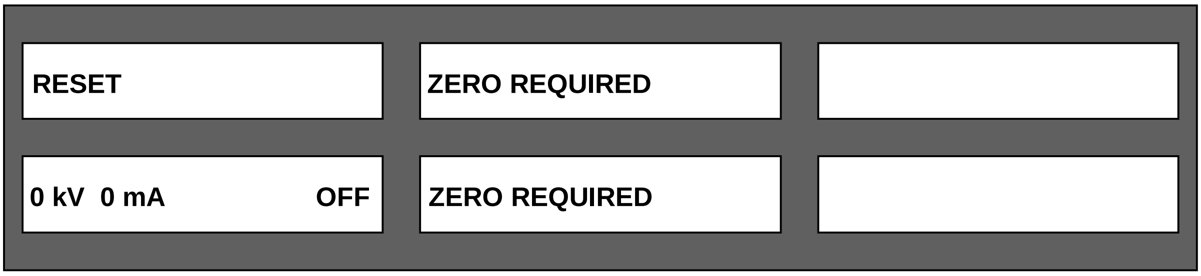
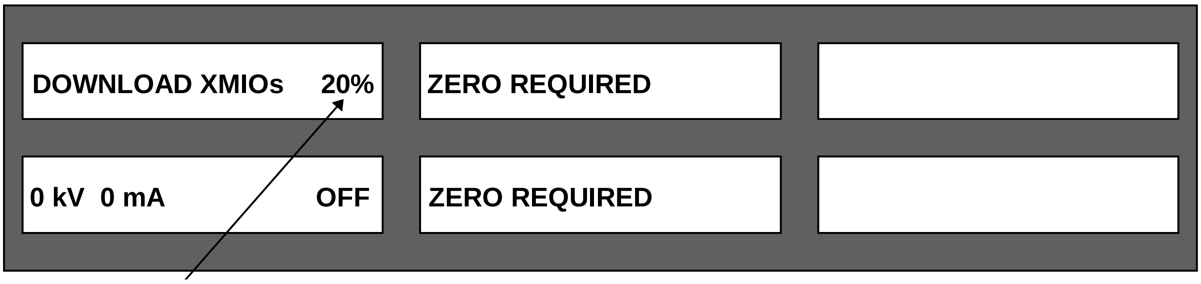


Figure 4.5

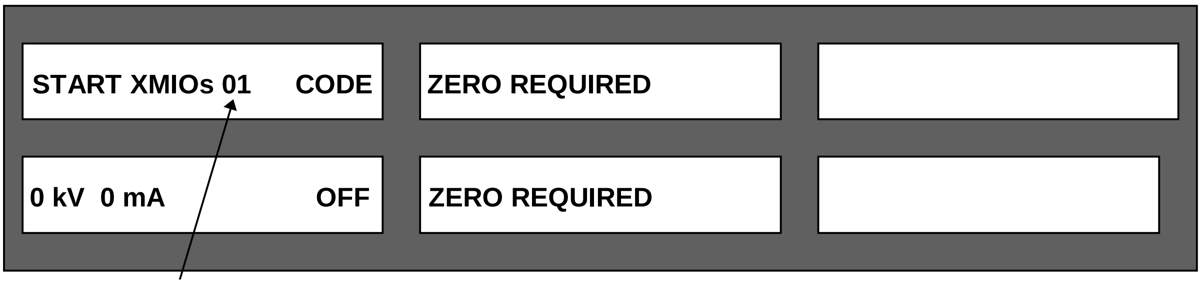
- **Step 2**



Count up in percent

Figure 4.6

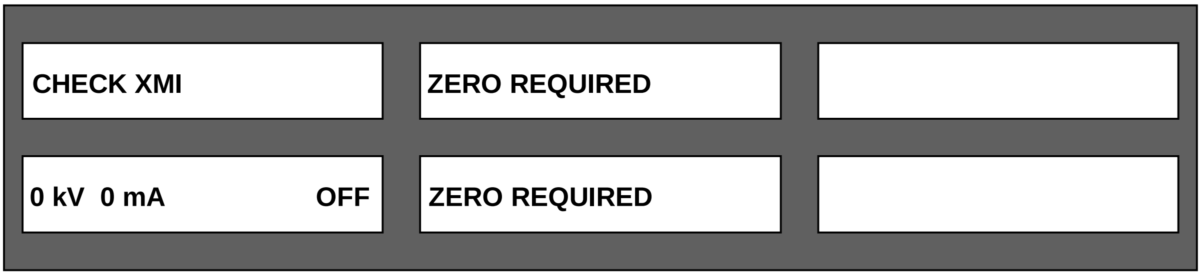
- **Step 3**



Code count up

Figure 4.7

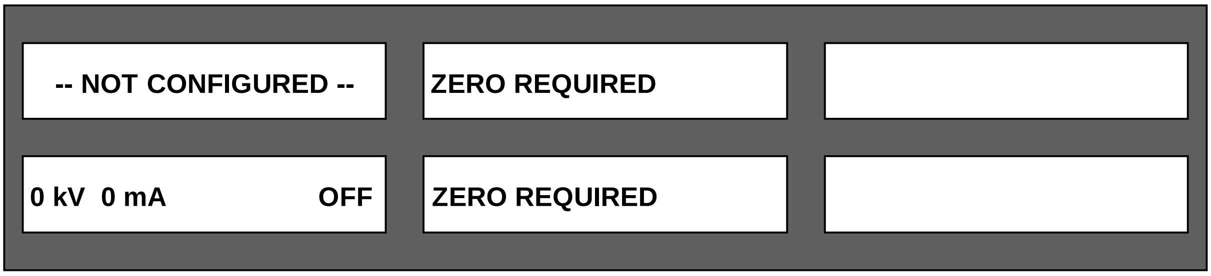
- **Step 4**



CHECK XMI	ZERO REQUIRED	
0 kV 0 mA OFF	ZERO REQUIRED	

Figure 4.8

- **Step 5**



-- NOT CONFIGURED --	ZERO REQUIRED	
0 kV 0 mA OFF	ZERO REQUIRED	

Figure 4.9

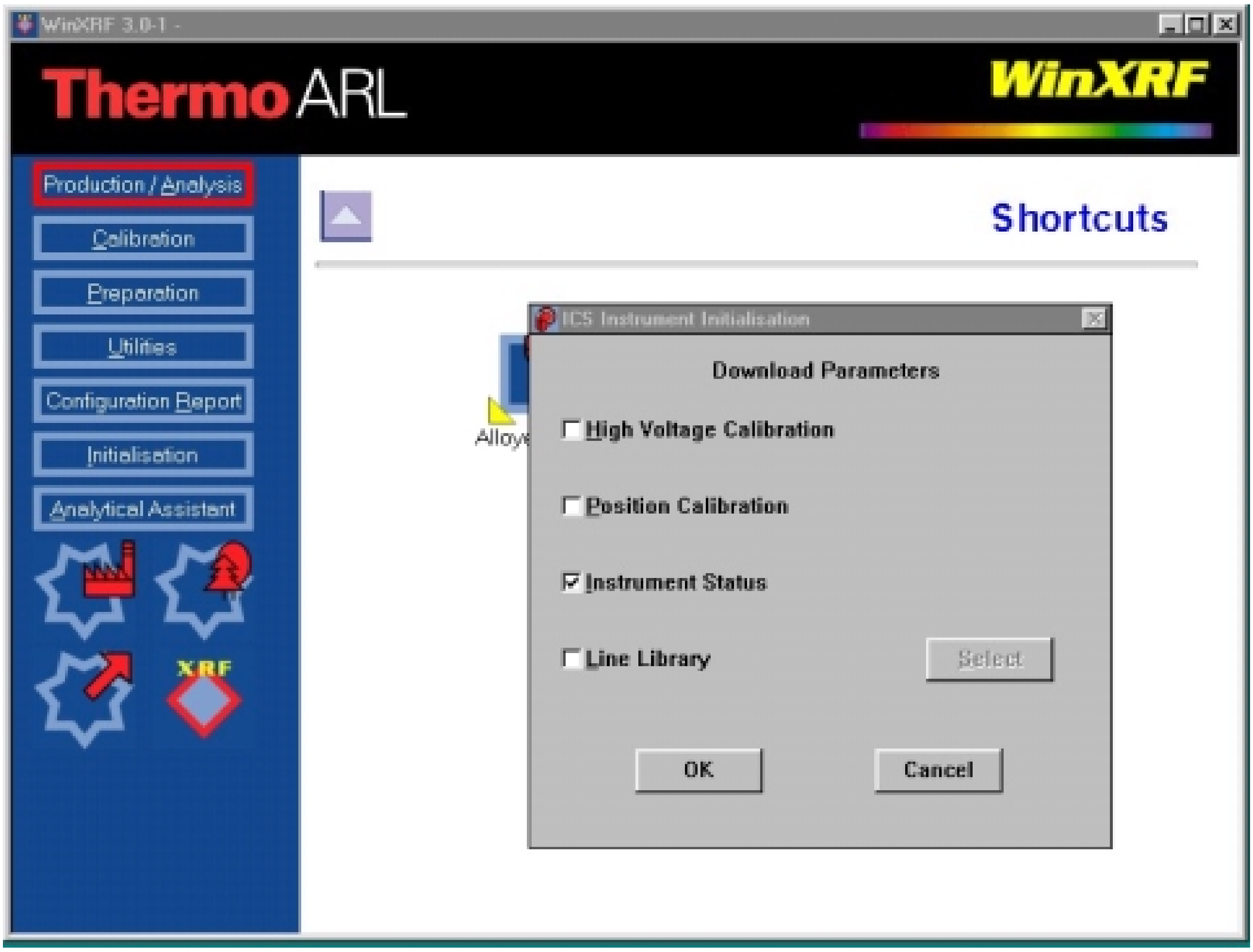
At this moment, the power-up sequence is finished. The instrument needs to be configured.

Instrument Configuration and Initialisation

Instrument Configuration

At the WinXRF start-up the following window will appear.

- Click on **Cancel**.



Download Parameters

☐ High Voltage Calibration

☐ Position Calibration

☑ Instrument Status

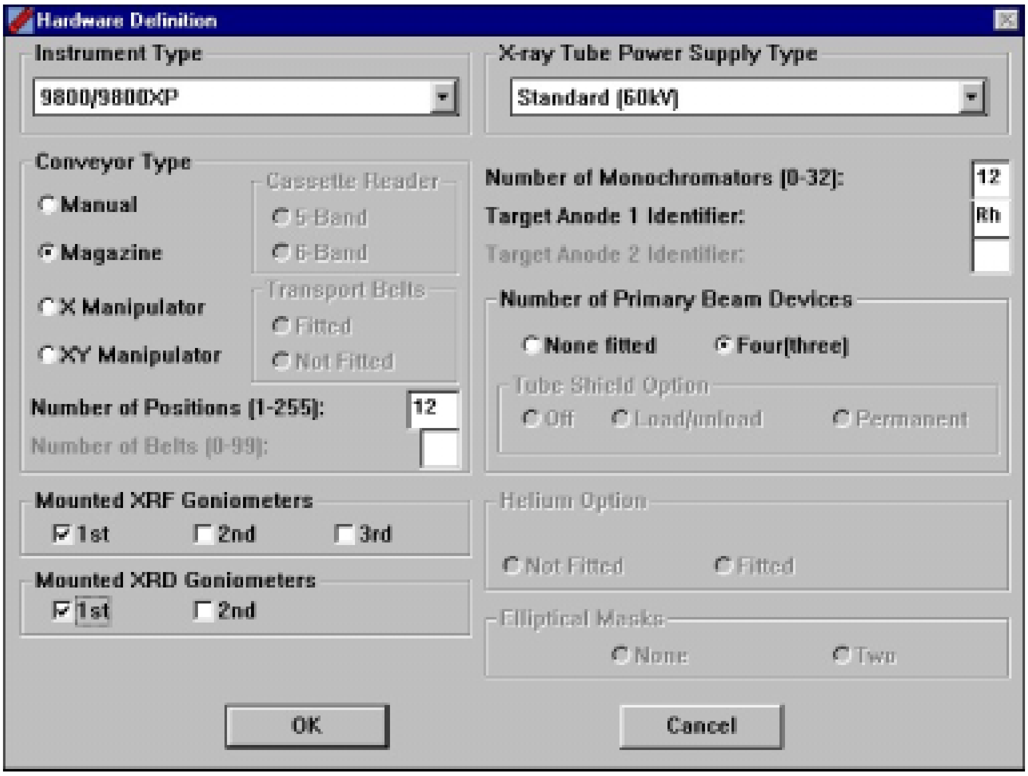
☐ Line Library

Figure 4.10

It is recommended that you inspect the instrument configuration parameters, to make sure they match with the instrument hardware.

- From the main menu, click on **Utilities**,
- Click on **Instrument Configuration**.
- Click on **Instrument**.
- If all parameters match, click on **OK**.

Some information about the hardware configuration of your ARL 9800 can be found in the QA report.



Hardware Definition

Instrument Type: 9800/9800XP

X-ray Tube Power Supply Type: Standard (60kV)

Conveyor Type:
○ Manual
● Magazine
○ X Manipulator
○ XY Manipulator

Cassette Reader: ○ 5-Band ○ 6-Band

Transport Belts: ○ Fitted ○ Not Fitted

Number of Positions (1-255): 12

Number of Belts (0-99):

Mounted XRF Goniometers: ☑ 1st ☐ 2nd ☐ 3rd

Mounted XRD Goniometers: ☑ 1st ☐ 2nd

Number of Monochromators (0-32): 12

Target Anode 1 Identifier: Rh

Target Anode 2 Identifier:

Number of Primary Beam Devices: ○ None fitted ● Four(three)

Tube Shield Option: ○ Off ○ Load/unload ○ Permanent

Helium Option: ○ Not Fitted ○ Fitted

Elliptical Masks: ○ None ○ Two

Figure 4.11

Instrument Type

You must select **ARL 9800 / ARL 9800 XP** or **ARL 9800 Oasis** as **Instrument type** depending on your version.

X-ray Tube Power Supply Type

X-ray tube power supply type for ARL 9800 and ARL 9800 XP:

- **Standard (60 kV)**
- **High Performance (70 kV)**

The ARL 9800 is supplied with a **Standard 60 kV** X-ray tube power supply. An option is available to power the X-ray tube up to 70 kV. In this case select **High Performance 70 kV**.

Sample Loading Mechanism Type

Three sample loading mechanisms are available with the ARL 9800:

Selection for the 12 positions sample changer:

- **Magazine**
- **Number of Positions: 12**

Selection for the X-Y sample changer:

- **X-Y Manipulator**

Exit

- **Number of Positions: xxx**

Selection for the SMS automation system:

- **X-Y Manipulator** or **Manual entry**
- **Number of Positions: 1**

Note: If the X-Y sample changer uses belts, then **xxx = 49**.
If the X-X sample changer has two trays for samples or cassettes, then **xxx = 149**.

Primary Beam Device (PBD)

The primary beam device is an option. Four primary beam devices are available with the ARL 9800.

Primary beam collimator:

- **Number of Positions: 2** only applicable if the ARL 9800 is equipped with the XRD channel.

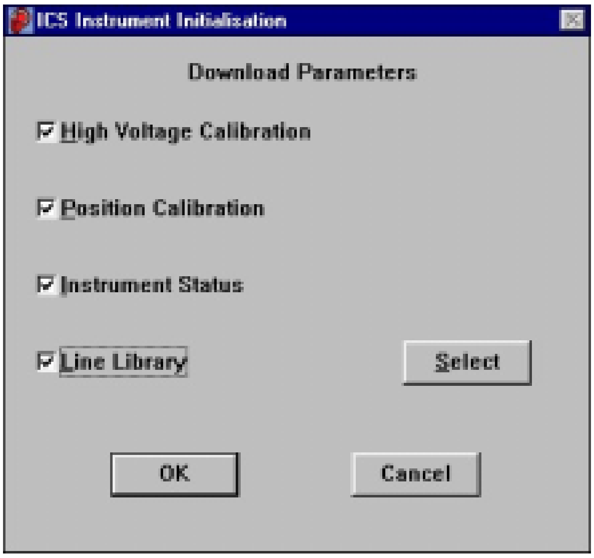
Primary beam filter:

- **Number of Positions: 4**
- **Only 2 filters possible if equipped with a XRD system**

Instrument Initialisation

The initialisation of the ARL 9800 is done to reset all microprocessors and the mechanical parts. In case of problems with the instrument this operation should be done.

- To initialise the instrument click on **Initialisation** and then **Send Instrument Configuration <F6>**, or simply press **<F6>**.
- Select **High Voltage Calibration**, **Position Calibration**, **Instrument Status** and **Line Library**.


ICS Instrument Initialisation

Download Parameters

☑ High Voltage Calibration

☑ Position Calibration

☑ Instrument Status

☑ Line Library

Figure 4.12

- The **Database Category** of which the **Line Library** should be transmitted, can be selected by clicking on **Select**.

- Click on **OK** to return to the former dialog box.
- Click on **OK** to start the transmission.

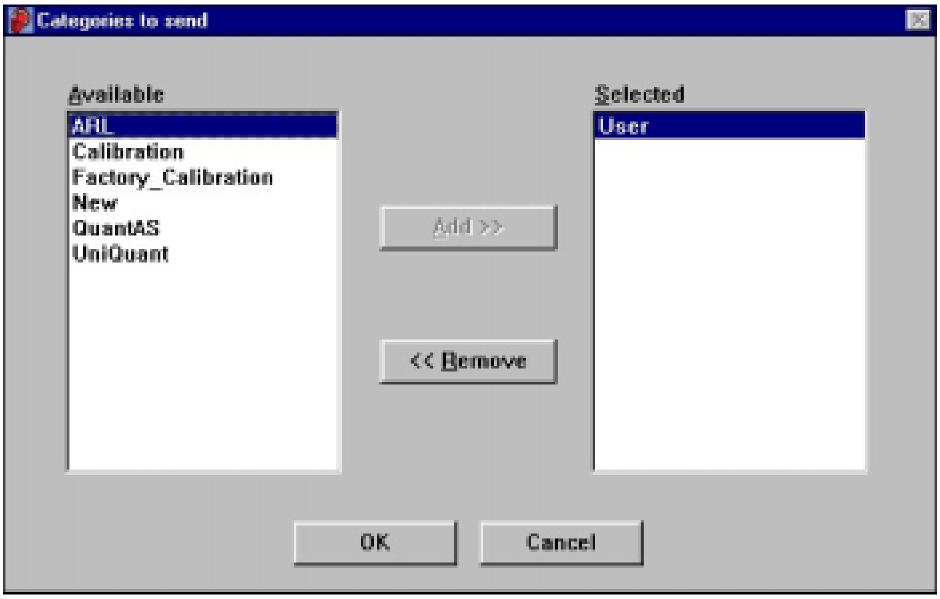


Figure 4.13

Click on the **Line Communication** monitor item in the task bar and wait for the end of the configuration.

Figure 4.14

During the initialisation, the following information will be displayed.

- **Step 1**

CHECK COMPATIBILITY	ZERO REQUIRED	
0 kV 0 mA OFF	ZERO REQUIRED	

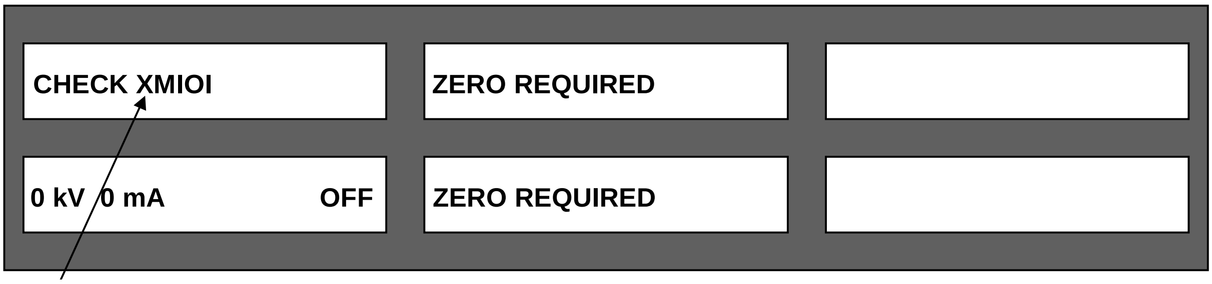
Figure 4.15

- **Step 2**

CHECK XMIs COMPAT.	ZERO REQUIRED	
0 kV 0 mA OFF	ZERO REQUIRED	

Figure 4.16

- **Step 3**

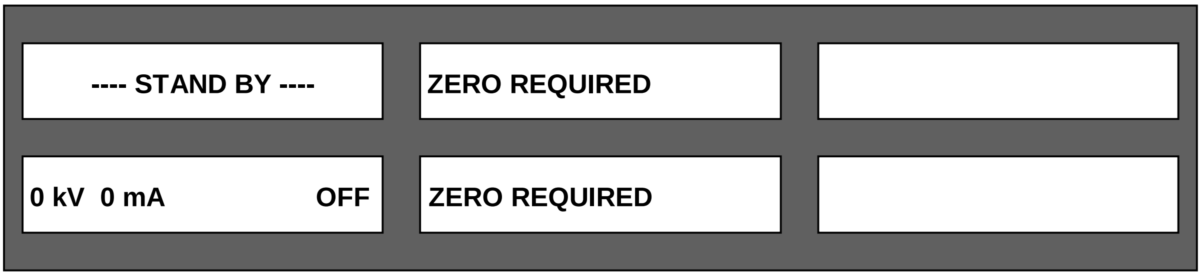


CHECK XMIOI	ZERO REQUIRED	
0 kV 0 mA OFF	ZERO REQUIRED	

Count up

Figure 4.17

- **Step 4**



---- STAND BY ----	ZERO REQUIRED	
0 kV 0 mA OFF	ZERO REQUIRED	

Figure 4.18

At this moment, the basic initialisation sequence is finished. The rest of the initialisation depends on the configuration of the instrument.

Gas Supply

Argon-Methane Gas for FPC (optional)

- Check that the gas bottle tap is open.
- Check that the pressure is set at 0.25 bar on the bottle pressure reducer.

The argon-methane gas flow is set to 20 ml / min. A 50 litre bottle at 200 bars will last approximately ten months. Check regularly the gas level of the bottle. To prevent contamination, don’t wait until it gets completely empty before replacing it.

Gas Bottle Replacement

To exchange a gas bottle follow the procedure hereafter:

- Make sure the instrument is in idle state.
- Close the present bottle.
- Remove the manometer from the bottle.
- Secure the new bottle to its position. Remove the top
- Check that the tap is properly closed.
- Unscrew the brass protection.
- Screw on and tighten the manometer on the new bottle.
- Open the tap slowly.
- After approximately 3 minutes the system is again in regulation.

- Check the status to make sure every thing is in order.

Environment Setting

To select the environment, perform the following actions:

- Click on **Initialisation** in the main menu.
- Click on **Set Environment** icon.
- Select the environment by clicking onto the corresponding radio button.
- Click on **OK**.

Set X-ray Environment

Desired Environment: ● Vacuum ○ Air ○ Helium

Current Environment: Vacuum

Figure 4.19

To change to a **Vacuum** environment when **Air** environment is set, click on **Vacuum** and **OK**.

The ARL 9800 display will show the following sequence:

PUMPING PC:>1000 PA ↑	ZERO REQUIRED	
0 kV 0 mA OFF	ZERO REQUIRED	

PUMPING SP:>1000 PA ↑	ZERO REQUIRED	
0 kV 0 mA OFF	ZERO REQUIRED	

Figure 4.20

The first display will inform on the vacuum level in both chambers.

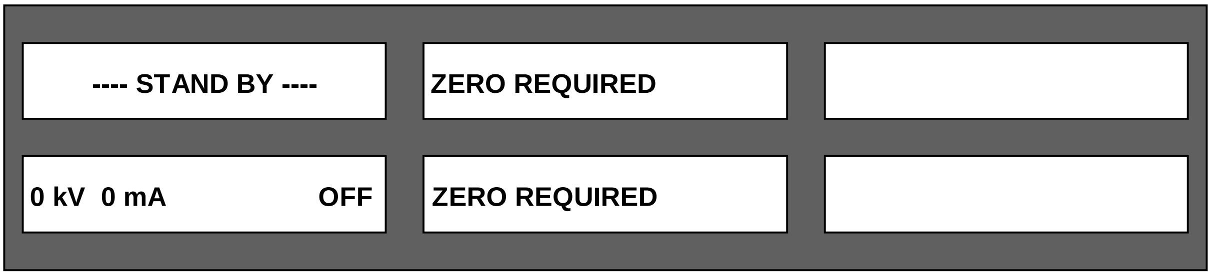
- PC: Primary chamber
- SP: Spectrometer

The arrows on the right provide the following information:

Exit

Sign	**Description**
↑	The estimated pressure is increasing
↓	The estimated pressure is decreasing
→	The estimated pressure is constant

To confirm that the vacuum environment has been achieved, the ARL 9800 instrument display must show ‘STAND BY’ in the first display.

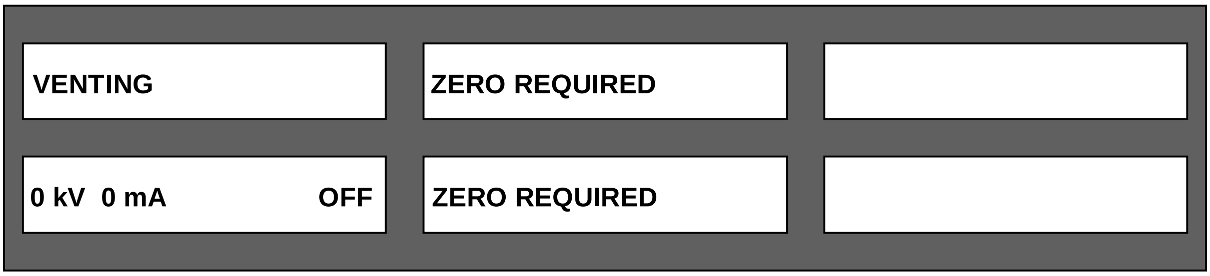


---- STAND BY ----	**ZERO REQUIRED**	
0 kV 0 mA OFF	**ZERO REQUIRED**	

Figure 4.21

To change to an **Air** environment when the **Vacuum** environment is set, click on **Air** and **OK**.

The ARL 9800 display will show the following message:

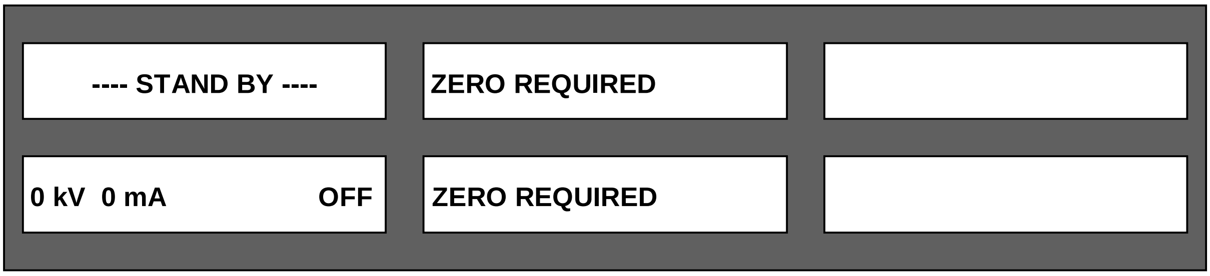


VENTING	**ZERO REQUIRED**	
0 kV 0 mA OFF	**ZERO REQUIRED**	

Figure 4.22

Note: When changing from the Vacuum to the Air environment, the X-ray tube will be switched off automatically.

To confirm that the Air environment has been achieved, the ARL 9800 instrument display must show ‘STAND BY’ in the first display.



---- STAND BY ----	**ZERO REQUIRED**	
0 kV 0 mA OFF	**ZERO REQUIRED**	

Figure 4.23

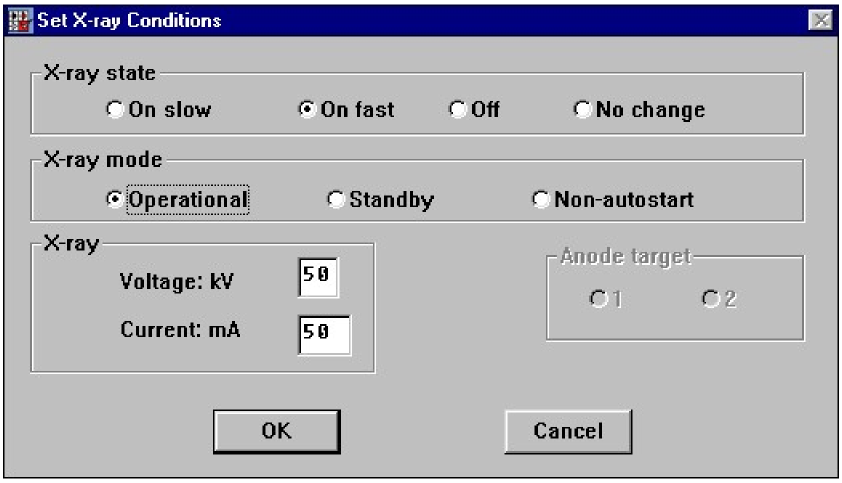
Switch the X-ray Tube Power Supply on

To switch the X-ray power supply on, from the main menu click on **Initialisation** and then **Set X-Ray**.

- Select **On Slow**, **Operational** or **Standby**.

- Select the **kV** and **mA** working condition.
- Click on **OK**.

> It is recommended to keep the X-ray tube constantly in operation (24/24 hours) in order to avoid thermal stress on the filament. See Eco mode operation to spare energy when the instrument is not in use for analysis.



Set X-ray Conditions

X-ray state: ○ On slow ● On fast ○ Off ○ No change

X-ray mode: ● Operational ○ Standby ○ Non-autostart

X-ray

Voltage: kV 50

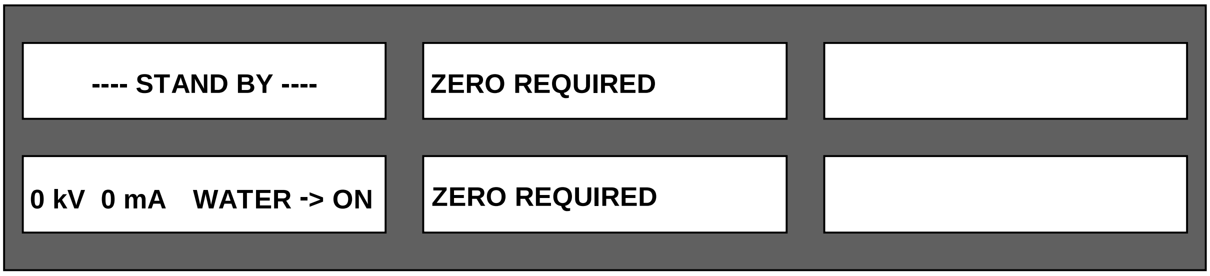
Current: mA 50

Anode target: ○ 1 ○ 2

Figure 4.24

Before the X-ray tube is actually switched on, the ARL 9800 display will show the following messages:

- The water pump will start.

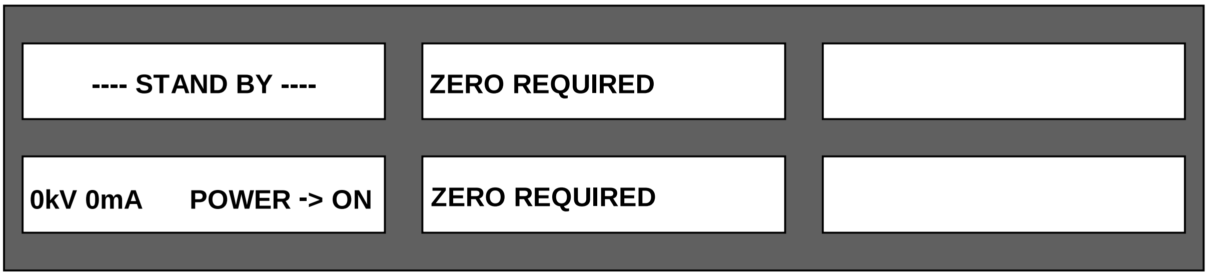


---- STAND BY ----	ZERO REQUIRED	
0 kV 0 mA WATER -> ON	ZERO REQUIRED	

Figure 4.25

As soon as the correct water flow, temperature and resistivity are reached, the X-ray tube will switch on.

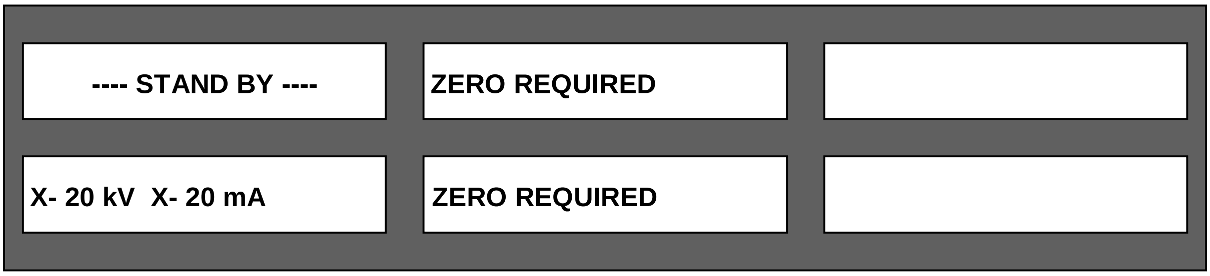
- The X-ray lamp located on the top of the ARL 9800 instrument is switched on.



---- STAND BY ----	ZERO REQUIRED	
0kV 0mA POWER -> ON	ZERO REQUIRED	

Figure 4.26

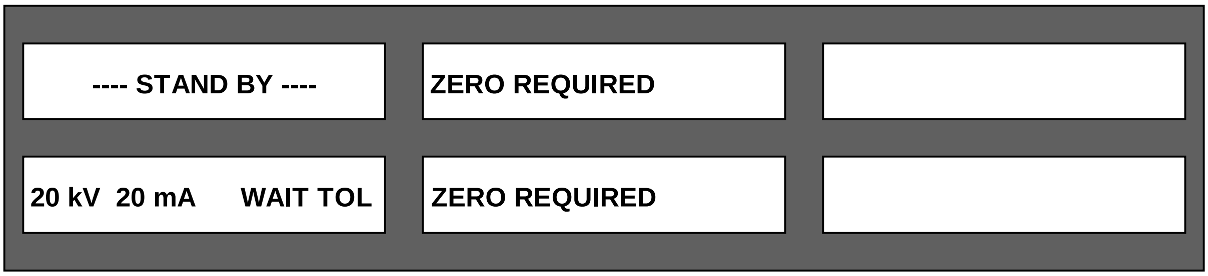
Exit



---- STAND BY ----	ZERO REQUIRED	
X- 20 kV X- 20 mA	ZERO REQUIRED	

Figure 4.27

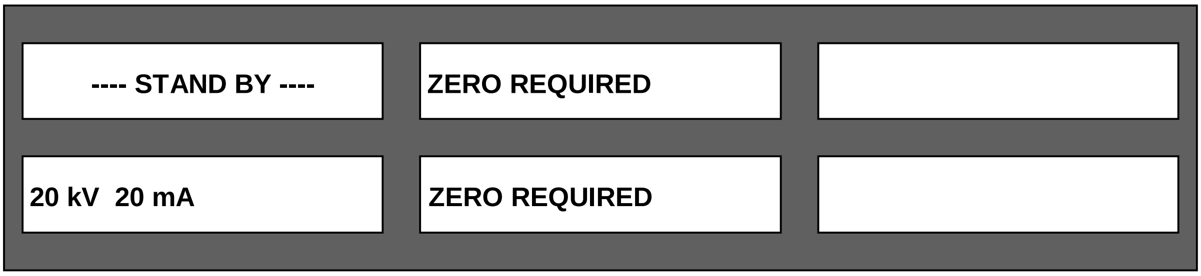
As soon as the conditions are approaching the desired value, the ARL 9800 instrument will wait for the exact voltage and current values to be attained.



---- STAND BY ----	ZERO REQUIRED	
20 kV 20 mA WAIT TOL	ZERO REQUIRED	

Figure 4.28

As soon as the desired conditions are within the required tolerances the display shows:



---- STAND BY ----	ZERO REQUIRED	
20 kV 20 mA	ZERO REQUIRED	

Figure 4.29

Eco Mode Setting

Eco Mode is a mode in which, when the instrument has not been used for a certain length of time, the X-ray power decreases to a pre-defined value. It is also possible to set the firmware in such a way that the power is raised automatically to specific working conditions at a pre-defined hour on specified dates.

The Eco Mode settings can be defined by execution of the menu function **Utilities** followed by selection of the folder **Instrument Configuration** and the icon **Eco Mode Setting**.

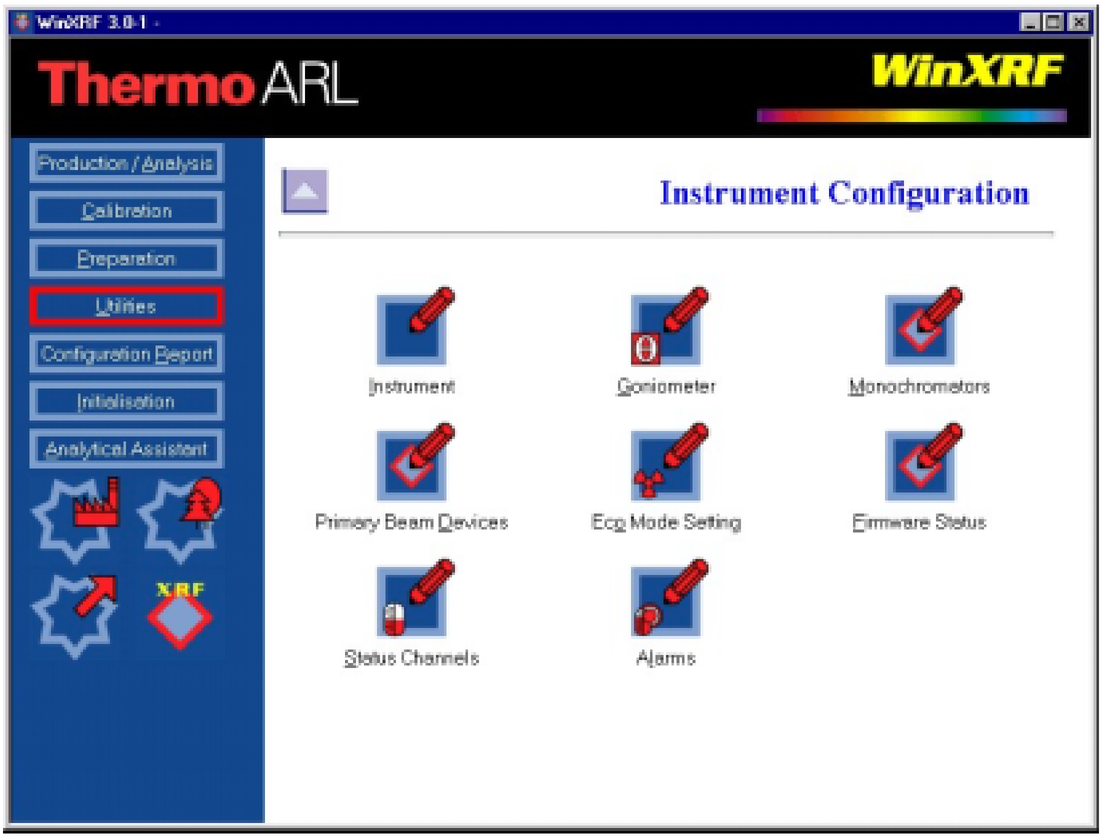
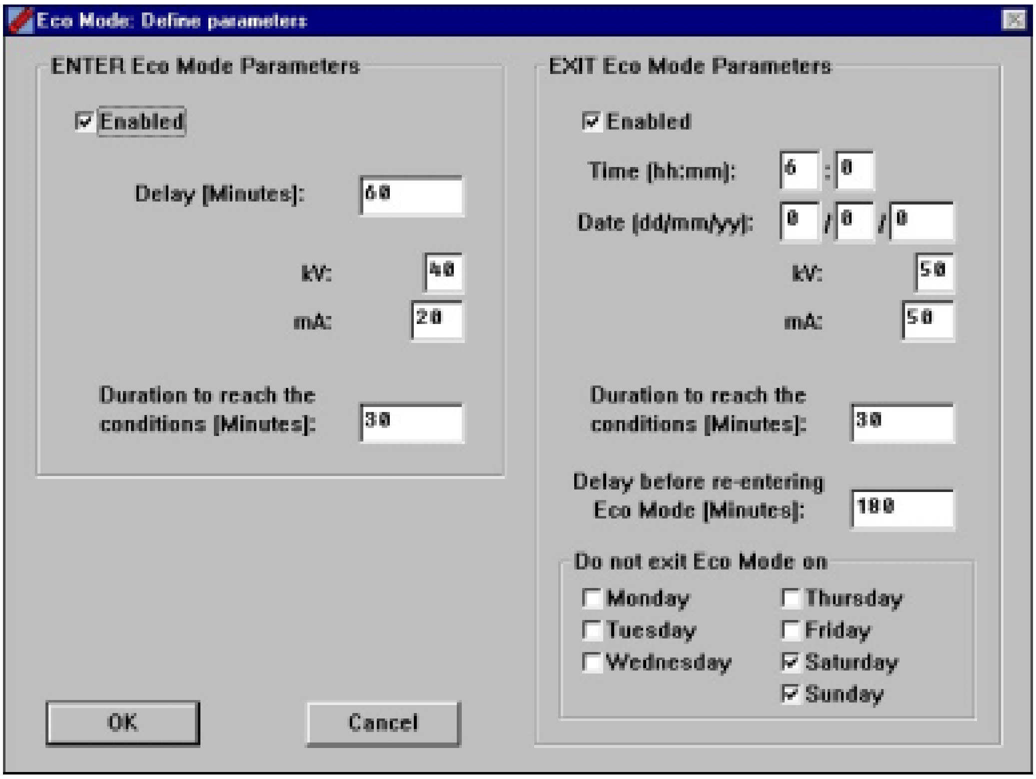


Figure 4.30

This causes the dialog box shown in the next figure to be displayed. This dialog box is divided into two parts, the left half allows definition of the conditions during the Eco mode period, and the right half allows definition of the time and dates when the Eco Mode has to be left and the X-ray power conditions to which the instrument should return.



Eco Mode: Define parameters

ENTER Eco Mode Parameters

☑ Enabled

Delay [Minutes]: 60

kV: 40

mA: 20

Duration to reach the conditions [Minutes]: 30

EXIT Eco Mode Parameters

☑ Enabled

Time [hh:mm]: 6 : 0

Date [dd/mm/yy]: 0 / 0 / 0

kV: 50

mA: 50

Duration to reach the conditions [Minutes]: 30

Delay before re-entering Eco Mode [Minutes]: 180

Do not exit Eco Mode on

☐ Monday
☐ Tuesday
☐ Wednesday
☐ Thursday
☐ Friday
☑ Saturday
☑ Sunday

Figure 4.31

The parameters that can be defined are explained hereafter.

ENTER Eco Mode Parameters

This allows defining the X-ray power conditions during the Eco Mode and how these conditions should be reached.

Enabled This check box has to be checked to allow the modification of the parameters and to activate the Eco Mode.

Delay (Minutes)
The Eco Mode is started after the instrument has been idle for the time specified here. A delay of 60 minutes is the recommended value.

kV This is the kV value to be reached in Eco Mode (40 kV is recommended).

mA This is the mA value to be reached in Eco Mode (20 mA is recommended).

Duration to reach the conditions (Minutes)
The value entered here defines the time during which the conditions are continuously changed until the final Eco Mode conditions (kV/mA) are reached. A duration of 60 minutes is the recommended value.

EXIT Eco Mode Parameters

This allows defining the dates and hour at which the X-ray power conditions should be raised to a specified setting. Furthermore, the way how these conditions should be reached can also be specified.

Enabled This check box has to be checked to modify the parameters and to cause the settings to be activated.

Exit

Time (hh:mm)

This defines the time of the given dates at which the Eco Mode should be left to reach the working conditions specified hereafter.

Date (dd/mm/yy)

This defines the dates at which the Eco Mode should be left to reach the working conditions specified hereafter. 0 means “every”, i.e. 00/00/0000 means every day, 03/00/0000 means every 3rd of every month of every year, 00/07/1997 means every day in July 1997, etc.

kV This is the kV value to which the X-ray power setting should return when leaving the Eco Mode.

mA This is the mA value to which the X-ray power setting should return when leaving the Eco Mode.

Duration to reach the conditions (Minutes)

This value defines the time during which the X-ray power is continuously raised until the final working conditions (kV/mA) are reached. A duration of 60 minutes is the recommended value.

Delay before re-entering Eco Mode (Minutes)

This value defines the delay before the instrument should again return to the Eco Mode in case the instrument remained idle over the whole period specified here. A delay of 60 minutes is the recommended value.

Do not exit Eco Mode on

If the date specification should be restricted to certain days of the week on which the Eco Mode should not be left (weekends), then these days can be selected here.

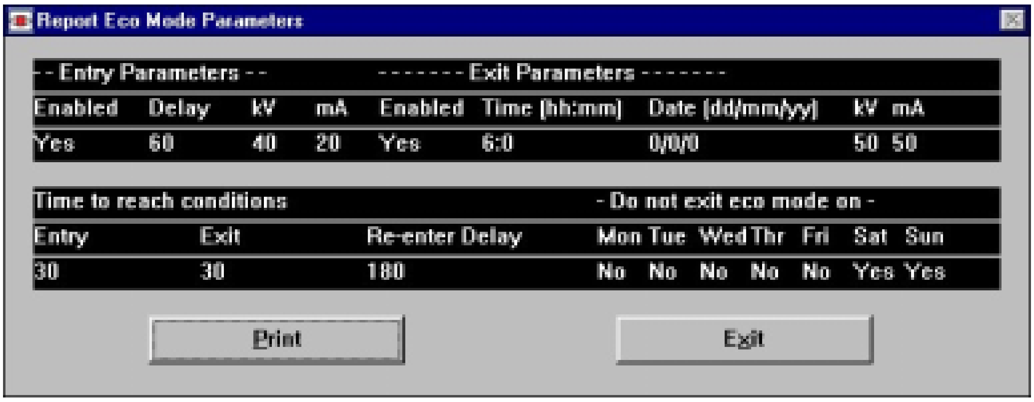
After each modification of one of the parameters described above, the dialog box has to be left by clicking onto the **OK** button to store the new values. The user is then asked, whether he wants to send the new Eco Mode parameters immediately to the ICS (instrument), or only when the instrument is initialised the next time.

Load updated Eco Mode data to Instrument? If you choose No, the changes will only be taken into account at the next instrument configuration.

Figure 4.32

Report Eco Mode Parameters

It is possible to obtain a print out of Eco Mode parameters by selecting the **Configuration Report** option of the main menu, followed by the **Utilities** folder, **Instrument Configuration** folder and finally the **Eco Mode Parameters** icon. Selection of the **Print** button in the displayed screen will print the parameters.



- - Entry Parameters - -				- - - - - - - Exit Parameters - - - - - - -				
Enabled	Delay	kV	mA	Enabled	Time (hh:mm)	Date (dd/mm/yy)	kV	mA
Yes	60	40	20	Yes	6:0	0/0/0	50	50

Time to reach conditions			- Do not exit eco mode on -						
Entry	Exit	Re-enter Delay	Mon	Tue	Wed	Thr	Fri	Sat	Sun
30	30	180	No	No	No	No	No	Yes	Yes

Figure 4.33

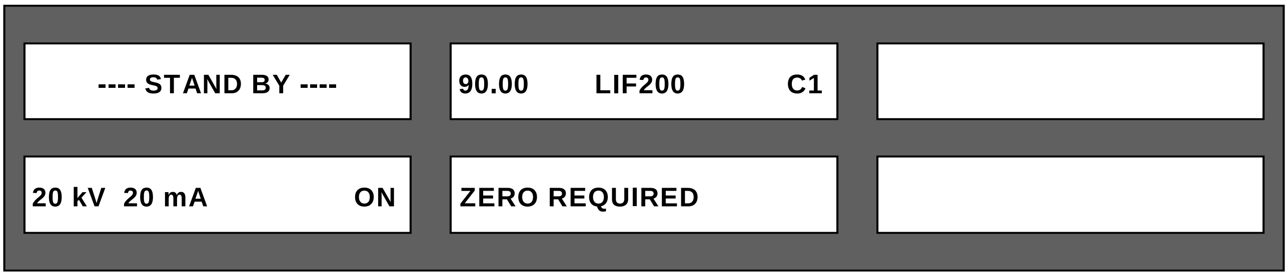
Goniometer Initialisation

After a power up the goniometer can be initialised and this is done with the goniometer zero operation. From the main menu click on **Initialisation** and then click on **Zero Goniometer**. Click on **OK** to start the zero operation.

XRF Goniometer 1

Figure 4.34

After the initialisation of the goniometer, the corresponding instrument display will show the following:



---- STAND BY ----	90.00 LIF200 C1	
20 kV 20 mA ON	ZERO REQUIRED	

Figure 4.35

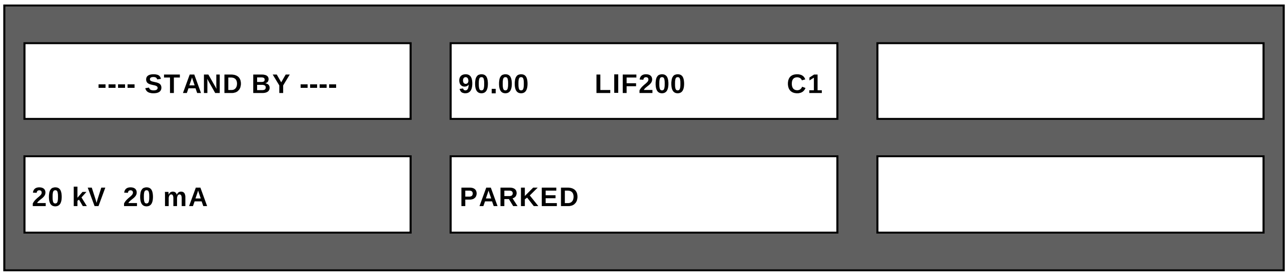
Note: All hardware and software settings are described in the technical description manual for this optional measuring equipment. We only inform that the change of any of these settings may affect the correct functioning of the goniometer. Our world wide After Sales Service is able to provide adequate training or direct support on site.

X-ray Diffraction (XRD) Initialisation

After a power up the XRD can be initialised and this is done with the goniometer zero operation. From the main menu click on **Initialisation** and then click on **Zero Goniometer**. By clicking on the **Next** button select the **XRD Goniometer**. Click on **OK** to start the zero operation.

Figure 4.36

After the initialisation of the XRD, the corresponding instrument display will show the following:



---- STAND BY ----

90.00 LIF200 C1

20 kV 20 mA

PARKED

Figure 4.37

Note: All hardware and software settings are described in the technical description manual for this optional measuring equipment. We only inform that the change of any of these settings may affect the correct functioning of the XRD. Our world wide After Sales Service is able to provide adequate training or direct support on site.

Monochromator (Fixed Channels) Initialisation

There is no initialisation to be done for the monochromators.

Note: All hardware and software settings are described in the technical description manual for this optional measuring equipment. We only inform that the change of any of these settings may affect the correct functioning of the monochromators. Our world wide After Sales Service is able to provide adequate training or direct support on site.

Status Reading

Before starting analysis, it is recommended that the user perform a status reading, to check if all the instrumental parameters are within the working tolerances. To read the instrument status click on **Initialisation** followed by **Read Instrument Status <F7>**, or simply press **<F7>**.

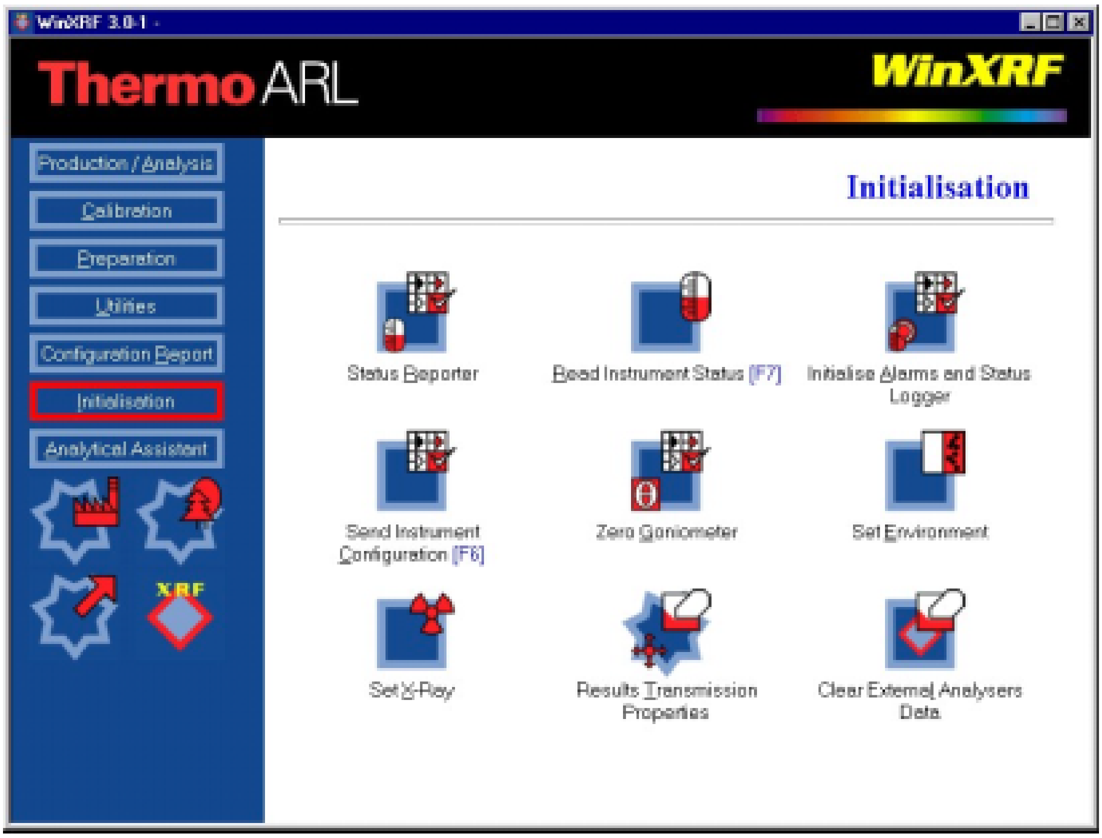
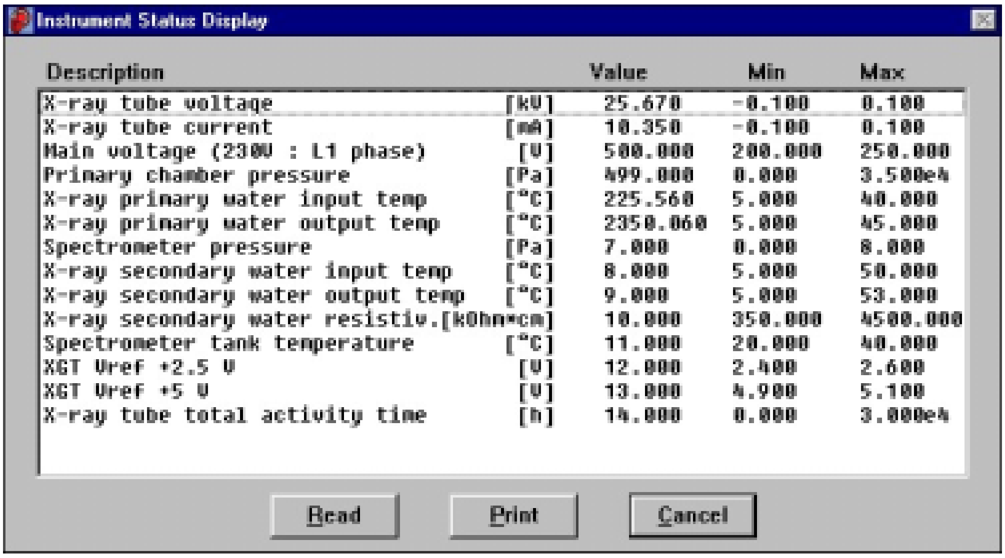


Figure 4.38



Description		Value	Min	Max
X-ray tube voltage	[kV]	25.670	-0.100	0.100
X-ray tube current	[mA]	10.350	-0.100	0.100
Main voltage (230V : L1 phase)	[V]	500.000	200.000	250.000
Primary chamber pressure	[Pa]	499.000	0.000	3.500e4
X-ray primary water input temp	[°C]	225.560	5.000	40.000
X-ray primary water output temp	[°C]	2350.060	5.000	45.000
Spectrometer pressure	[Pa]	7.000	0.000	8.000
X-ray secondary water input temp	[°C]	8.000	5.000	50.000
X-ray secondary water output temp	[°C]	9.000	5.000	53.000
X-ray secondary water resistiv.	[kOhm*cm]	10.000	350.000	4500.000
Spectrometer tank temperature	[°C]	11.000	20.000	40.000
XGT Vref +2.5 V	[V]	12.000	2.400	2.600
XGT Vref +5 V	[V]	13.000	4.900	5.100
X-ray tube total activity time	[h]	14.000	0.000	3.000e4

Figure 4.39

Note: It is important to keep in mind that the ARL 9800 needs a few hours to stabilise its internal temperature therefore we recommend to wait 4 to 5 hours before starting a calibration if the instrument has just been powered up. Routine analysis can be started as soon as the instrument is pumped down if it is no problem that the measured values might be slightly off in the beginning but are getting better during the stabilisation phase. It is however not advisable to drift correct the instrument until it is perfectly stable because the adverse effect could happen, i.e. the measured values are good in the beginning but are getting worse as the instrument stabilises.

Exit

General Specifications

Electrical Data

Instrument	**ARL 9800 XP**	**ARL 9800 OASIS**
Power requirement (including the computer)	7 kVA	2.5 kVA
Voltage	230 V (1 or 2 phases) -15% +10%	230 V (1 or 2 phases) -15% +10%
Neutral	0 V to ground	0 V to ground
Ground	< 2 Ω	< 2 Ω
Frequency	50 Hz or 60 Hz	50 Hz or 60 Hz
Power failures	< 10 ms for 50 Hz	< 10 ms for 50 Hz
	< 8.3 ms for 60 Hz	< 8.3 ms for 60 Hz

X-ray Power Supply

Maximum high voltage	60 kV (optional 70 kV)	50 kV
Maximum current	120 mA	20 mA
Maximum power	3.6 kW	600 W
Stability	+/- 0.0001% per 1% of main variation	+/- 0.0001% per 1% of main variation

Water Supply

Minimum flow	4.5 lt/min	No water supply
Minimum pressure	2.0 bar	
Maximum pressure	4.0 bar	
Minimum temperature	5°C	
Maximum temperature	18°C	
ARL 9800 water heating power	less than 4500 W	

Laboratory Environment

Minimum temperature	18°C	18°C
Maximum temperature	32°C (with cooling water at 12°C)	28°C
Maximum temperature change	±1°C per hour short variation <1 hour (thermal control)	±1°C per hour short variation <1 hour (thermal control)
	0.7°C per hour drift >1 hour	0.7°C per hour drift >1 hour
ARL 9800 air heating power	less than 1500 W	less than 1500 W
Maximum pulsed magnetic fields	1000 A/m (acc. to EN61000-4-9)	1000 A/m (acc. to EN61000-4-9)
Shock	Semi-sinusoidal unitary pulsation of 1g during 10±3 msec. applied in each direction of the 3 orthogonal axes (6 pulsations).	

Vibration	Sinusoidal vibration applied to the 3 orthogonal axes with an incrementation of 1 octave/min. 5-50 Hz amplitude 50µm 50-500 Hz 0.25g

Gas Supply

FPC	90% Ar (48) 10% CH_4 (35)	optional

Acoustic Noise

Maximum acoustic noise power level	60dB(A)	60dB(A)

Dimensions

Width	90 cm	90 cm
Length	130 cm	130 cm
Height	160 cm	160 cm
Weight	750 kg	750 kg

Spectrometer Geometry

X-ray tube incidence angle	90°	90°
Goniometer take-off angle	35°	35°
Distance anode-sample	< 31 mm	< 31 mm

Safety Standards (all versions)

Safety of machinery	EN 292-1,-2 (1991)
Electrical equipment of industrial machinery	EN 60204-1 (1984)
Low voltage switchgear and controlgear	EN 60947 (1991)
Safety requirement for electrical equipment for laboratory	IEC 1010-1,
Safety information technology equipment	IEC 950
Swiss safety standard for X-ray radiation	814.501 (1994)
German safety standard for X-ray radiation	RöV 1987

ARL 9800 instrument maintenance

As an example the table below provides information on the maintenance and the service required by the instrument analyzing 300 solid samples per day.

Location	**Work to be done**	**Weekly**	**Monthly**	**Yearly**	**Every 2 years**	**Every 3 years**
Cabinet	All fans		Check		Change	
	All dust filters		Check	Change		
	Inside the cabinet			Clean		
Software	Database backup	Perform				
Sample load/unload	Sample cassette		Clean			
	Sample Magazine		Clean			
	XY Magazine		Clean			
	Shutter assembly		Clean	Revise		
	Load lift			Revise		
	Transfer			Revise		
	Analyse lift			Revise		
	Sample rotation			Check		
Vacuum circuit	Oil pump level		Check/Fill	Change		
	Oil vacuum pump			Revise		
	Oil vacuum pump filter			Change		
	Molecular pump			Add grease		Change
	Spectro vacuum valve				Change	
	Prim. Ch. vacuum and venting valves			Change		
	Both vacuum level	Check		Adjust		
Water circuit	Deionized water level	Check				
	Deionized water flow	Check				
	Deionized water temperature	Check				
	Deionized filter cartridge			Change		
	Deionized water resistivity		Check			
	Prim. Water flow	Check				
	Prim. Water temperature	Check				
	Water valves			Change		
	All tap water tubes			Clean		
	Water leakage (Collar tightness)			Check		
X-Ray tube P.S.	X-Ray tube HV connection			Check		
	X-Ray tube P.S. HV connection			Check		
	HV silicone washers			Change		
	Test of the interlock circuit			Check		
P10 Gas circuit	P10 Gas bottle High pressure level	Check				
	P10 Gas bottle Low pressure level	Check				
	P10 Gas Interlock			Check		
	P10 Gas regulation valves			Change		
	P10 Gas overpressure valve				Change	
Prim. Beam Dev.	Prim. Beam Device assembly			Check		
Monos	All energy profiles		Check	Adjust		
	All thresholds and windows			Check		
	All detectors resolutions			Check		
	Mono filter positionning			Check		
XRD	Energy profile		Check	Adjust		
	Threshold and window			Check		
	Detector resolution			Check		
	Scan check positions		Check	Adjust		
Gonio	Energy profiles		Check	Adjust		
	Thresholds and windows			Check		
	Detectors FPC and SC resolutions			Check		
	Scan check positions		Check	Adjust		
Stability	Long and short term test			Check		

The frequency of the maintenance tasks can vary depending of the number and the nature of the samples analysed.

The frequency of the maintenance task can vary depending of the cleanliness (dust) of the environment (lab).

The yearly maintenance and service tasks can only be performed by certified and authorized ARL service engineers unless the user is properly trained (see our Maintenance Training Course Program).

5

SAMPLE PREPARATION

5 SAMPLE PREPARATION

Since X-ray spectrometry is essentially a comparative method of analysis, it is vital that all standards and unknown samples are presented to the spectrometer in a reproducible and identical manner. Any method of sample preparation must give specimens which are reproducible and which, for a certain calibration range, have similar physical properties including mass absorption coefficient, density and particle size. In addition, the sample preparation method must be rapid and cheap and must not introduce extra significant systematic errors, for example, the introduction of trace elements from contaminants in a diluent.

The quality of sample preparation in X-ray fluorescence analysis is at least as important as the quality of measurements. An adequately prepared sample must:

- be representative of the material;
- be homogeneous;
- when possible, be thick enough to meet the requirements of an infinitely thick sample.

Various preparation techniques have been described in the literature. The object of this chapter is to point out the main criteria for quality preparation of metallic samples, powder samples, and liquid samples.

For quantitative analysis, samples must be prepared in the same way like the standard samples used for calibration.

Typical methods of sample preparation are listed in the following table.

Type	Sample holder	Preparation		Sample	Purpose
Solid	Iron Steel Nickel Ferro-Alloy	Cutting	Surface grinding with belt surfacer	Solid sample holder	Surface smoothing
	Cu-alloy Al-alloy Pb-alloy Zn-alloy	Cutting	Surface milling with lathe	Solid sample holder	
Powder	Metallic Chemicals Polymer Plant	Grinding	Briquetting	Solid sample holder	Equalizing the density and surface smoothness
	Ceramic Ore Soil Sediment Oxide	Grinding	Fusion	Solid sample holder	Eliminating the mineralogical and the granulometric differences
Liquid	Oil, water	Spotting on filter			Transforming liquid into solid
	Water	Ion exchanging precipitating		Solid sample holder with filter holder	Concentrating and transforming liquid into solid

Sample Preparation for Solids

The preparation must be simple, rapid and reproducible. Usually, metallic samples are prepared as solid disc by conventional methods of machining: cutting, milling and polishing; grinding is used in the case of hard alloys and brittle materials such as ceramics.

The best polishing operation requires very fine abrasives to produce the scratch-free surfacing necessary for most analyses, and even a mirror-like surface is often required if the sample is to be analysed for light elements. The surface finish is of prime importance because the polishing striations give rise to the so-called shielding effect, which results in decrease in fluorescence intensities. As expected the decrease in intensity is more important for lighter elements when the primary radiation are perpendicular to the striations and weaker when they are parallel to them. For that reason, modern spectrometers are equipped with spinning sample holders to smooth out the influence of sample orientation, resulting in observed intensities on samples and standards that are reproducible.

However, the shielding effect may still be present; sample rotation will compensate for it only if the magnitude of the effect is the same for standards and production samples; this requires that the striation be of the same size and that the sample composition be similar (same effective wavelength).

In practice, striation depths of 100 µm are acceptable for elements with characteristic lines of short wavelengths, but striations deeper than few µm may impair significantly the accuracy of Si, Al and Mg determinations.

Very fine grits of Al2O3, SiC, B6C (80-120 grits) are commonly used to obtain the desired surface finish for most metals (Fe, Ni, Co bases).

Mechanical polishing may be undesirable for soft, malleable, multiphase alloys because of smearing of the softer components; the intensities of the elements in softer phases increase while those of the harder phases decrease. In such cases, special precautions must be taken even during milling and especially in the final polishing operation (Pb, Cu, Al, Zn, Sn Bases).

Polishing may be source of contamination since currently used abrasives, SiC and Al2O3, contain two elements that are often determined in commercial alloys. Sample surface cleaning may be necessary to remove these contaminations as well as grease stains and handling residuals.

Once the sample prepared, **be careful not to touch the sample surface**.

Touching the sample surface will contaminate it with Na, Cl and C among other elements, which might affect the analysis.

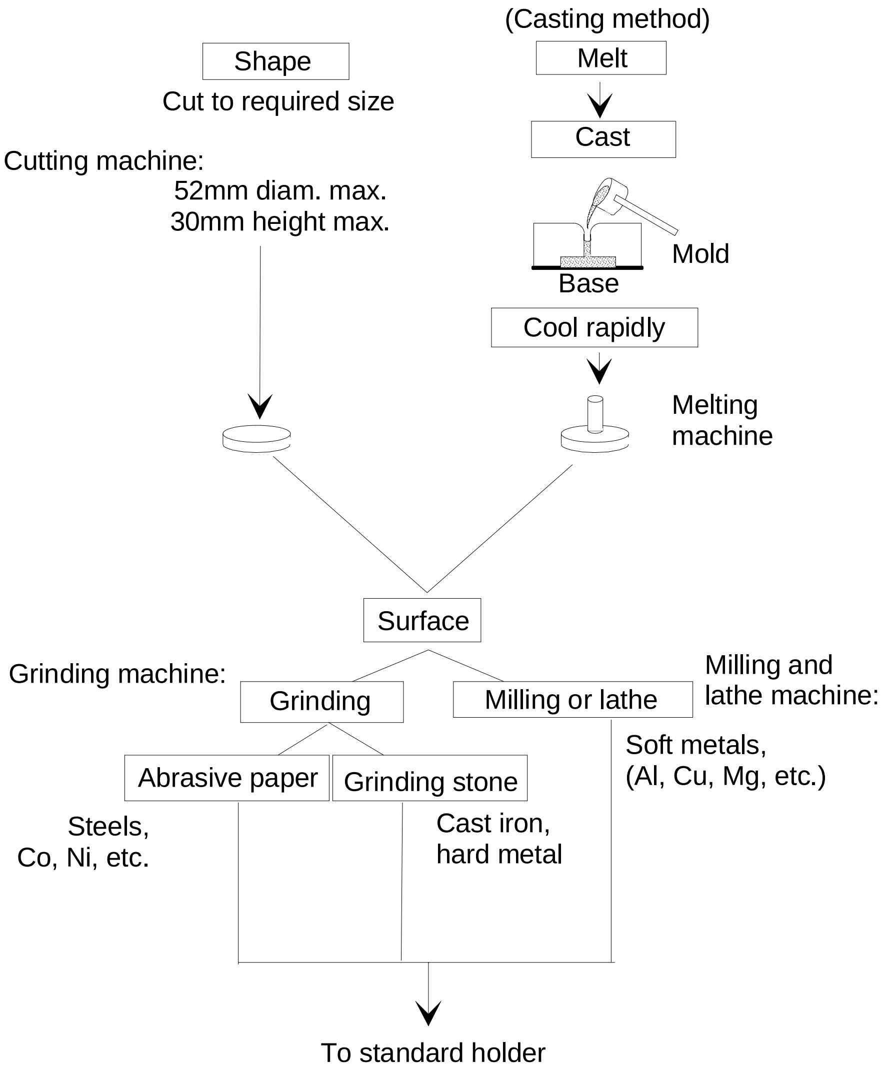

(Casting method)
Shape
Melt
Cut to required size
Cast
Cutting machine:
52mm diam. max.
30mm height max.
Mold
Base
Cool rapidly
Melting
machine
Surface
Grinding machine:
Grinding
Milling or lathe
Milling and
lathe machine:
Abrasive paper
Grinding stone
Soft metals,
(Al, Cu, Mg, etc.)
Steels,
Co, Ni, etc.
Cast iron,
hard metal
To standard holder

Figure 5.1

Sample Preparation for Powders

Where powders are not affected by particle size limitations the quickest and simplest method of preparation is to press them directly into briquets (or pellets) of constant density, with or without the additional use of a binder. In general, provided that the powder particles are less than about 50 microns in diameter the sample will pelletize at around 10-30 tons. Where the self-bonding properties of the powder are poor, higher pressure may have to be employed or in extreme cases a binder will be added before pelletizing. If it is necessary to add a binder the choice of the binding agent must be made with care. As well as having good self-bonding properties the binder must be free from significant contaminant elements and must have low absorption. It must also be stable under vacuum and irradiation conditions and it must not itself introduce significant interelement interferences. Of the large number of binding agents which have been successfully employed probably the most useful are wax and methyl cellulose.

Briquet method

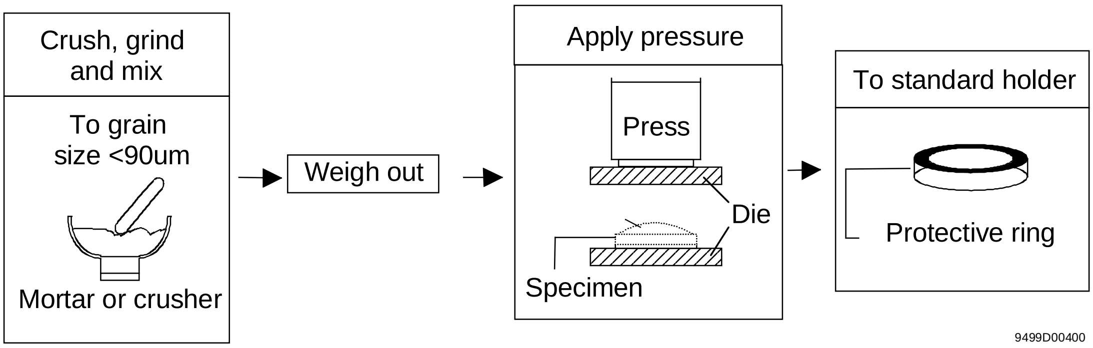


Figure 5.2

The analysis of powder is invariably more complex than that of metallic sample since in addition to interelement interferences and macroscale heterogeneity, particle size effects and mineralogical effects are also important. Although inhomogeneity and particle size can often be minimised by grinding below 50 microns and pelletizing at high pressure, often the effects cannot be completely removed because the harder compounds present in a particular matrix are not broken down. These effects produce systematic errors in the analysis of specific type of material e.g. siliceous compounds in slags, sinters and certain minerals.

The best way of completely removing both grain size and mineralogical effects is to use the fusion technique based on the method of Dr F. Claisse. The dissolution or decomposition of a portion of the sample by a flux is obtained at high temperature and produces an homogeneous glass. This eliminates particle size and mineralogical effects entirely. The fusion technique also has additional advantages:

a) Possibility of high or low specimen dilution for the purpose of decreasing matrix effects.

b) Possibility of adding compounds such as heavy absorbers or internal standards to decrease or compensate for matrix effects.

c) Possibility of preparing standards of desired composition.

Essentially the fusion procedure consists in heating a mixture of sample and flux at high temperature (800°-1200°) so that the flux melts and dissolves the sample. The overall composition and cooling conditions must be such that the end-product after cooling is a one phase glass.

Heating of the sample-flux mixture is usually done in platinum alloy crucibles but graphite may also be used when conditions permit.

The most frequently used fluxes are borates, namely lithium tetraborate and lithium metaborate or sodium tetraborate. Lithium tetraborate is widely used as it can answer to most cases. In certain cases, mixtures of these fluxes are more effective.

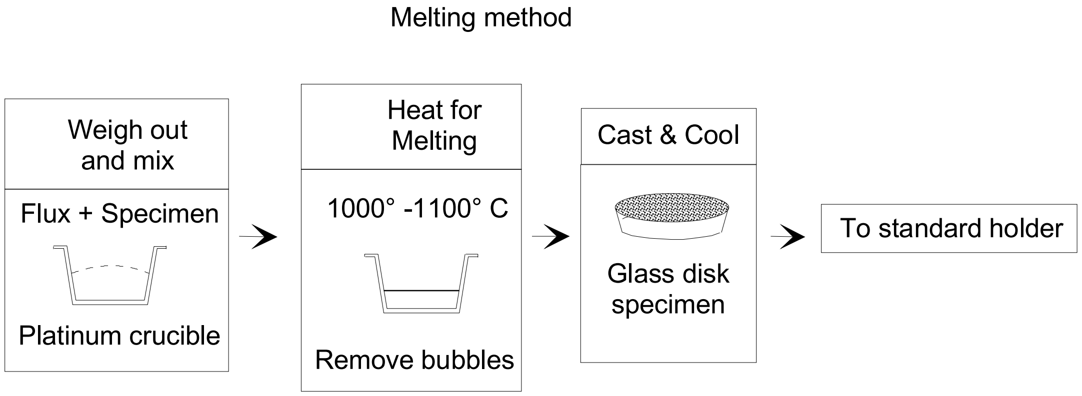


Figure 5.3

Once the sample prepared, **be careful not to touch the sample surface**.

Touching the sample surface will contaminate it with Na, Cl and C among other elements, which might affect the analysis.

Sample Preparation for Liquids

In order to measure liquids with the X-ray fluorescence technique, it is necessary that the instrument is equipped with a Helium flush system, because liquids can normally not be measured under vacuum for evident reasons. As the ARL 9800 instrument is not equipped with such a Helium system, it is not possible to measure liquids directly. However, certain sample preparation methods for liquids exist that allow liquids or their residues to be measured under vacuum. Three such methods are described in the following sections.

Wax Method for Oil

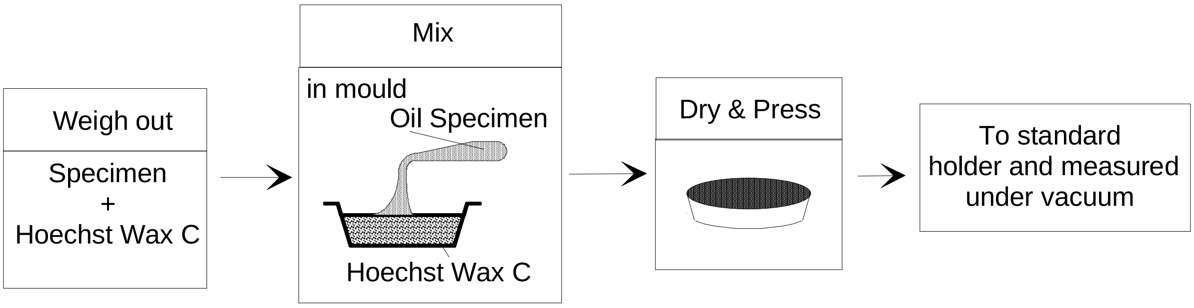


Figure 5.4

Filter Method

Precipitation Method

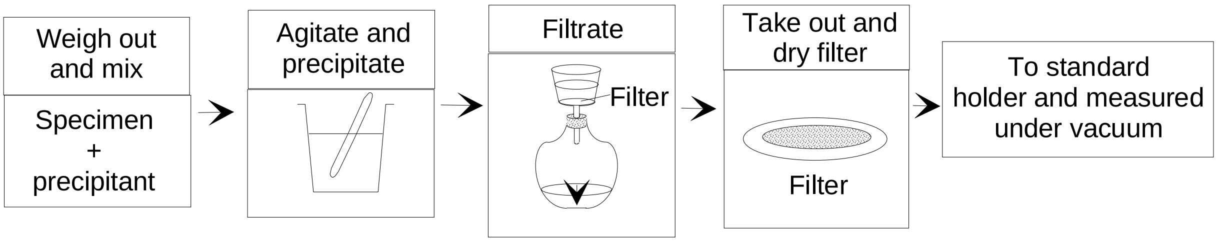


Figure 5.5

Absorption Method

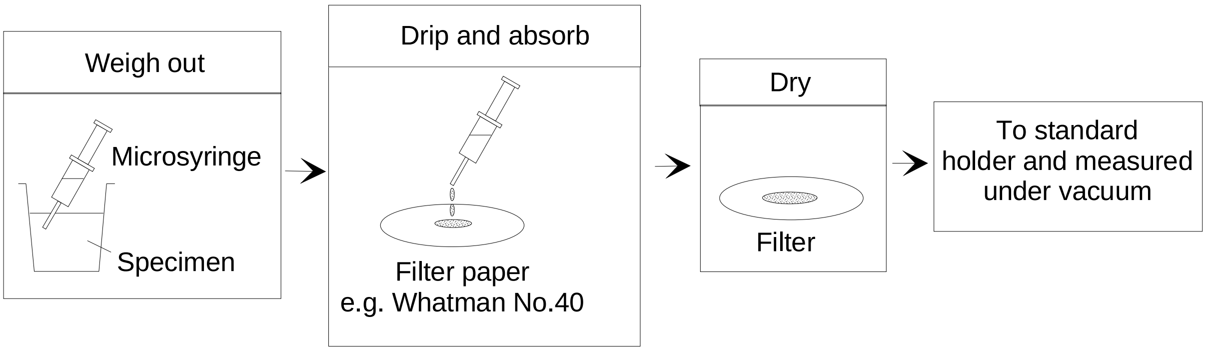


Figure 5.6

Exit

6

ANALYTICAL ASSISTANT

Exit

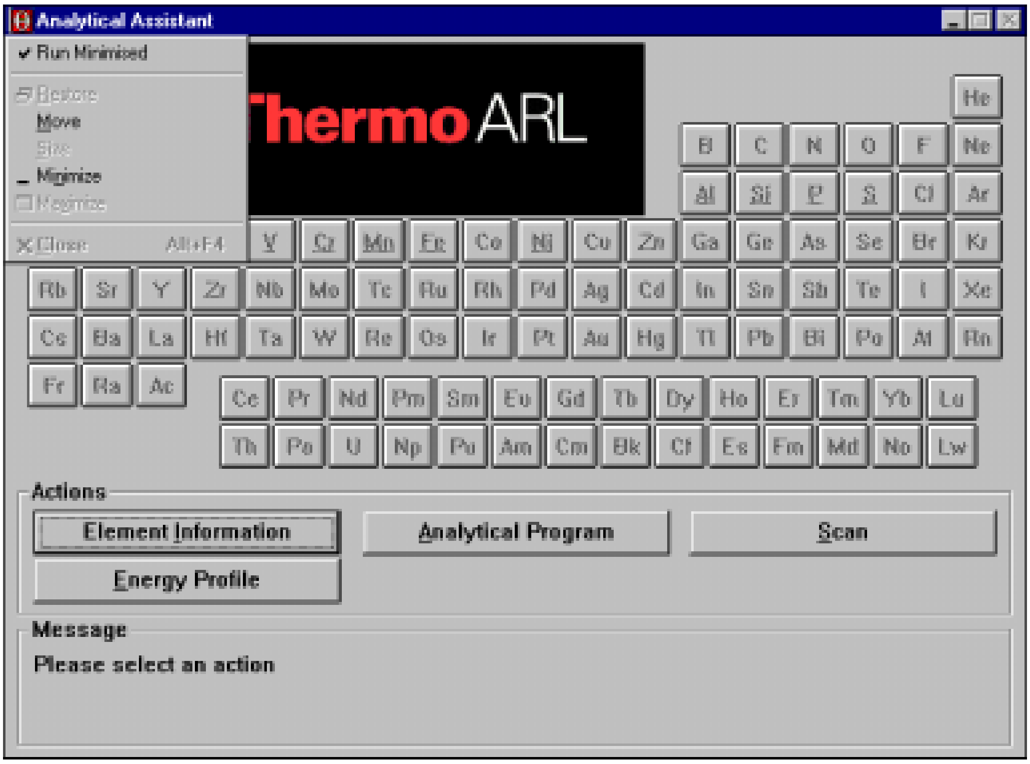
6 ANALYTICAL ASSISTANT

Introduction

This chapter describes the functions provided by the Analytical Assistant. This Analytical Assistant is started automatically by WinXRF and is either present on the task bar, or as a periodic chart on the screen.

The way the Analytical Assistant is started is given by the option **Run Minimised** in the **System Options** menu (see figure below), i.e. if this option is not selected, then the Analytical Assistant displays the periodic chart on the screen.

Note: Elements for which a monochromator is present are shown underlined. This only applies to the ARL 9800 series.



Please select an action

Figure 6.1

Following features are available:

Element Information
This function gives useful information about the selected element in terms of analytical parameters (lines, crystals, detectors, PBF, etc.) that are best suited with respect to the configuration of the instrument.

Scan This function allows a quick scan to be performed over the selected element with the most suitable lines and analytical parameters with respect to the configuration of the instrument.

Energy Profile
This function allows an energy profile to be performed over the selected element using the most suitable lines and analytical parameters with respect to the configuration of the instrument.

Analytical Program

This function represents the heart of the Analytical Assistant. Basically it allows creation of an analytical program with the most suitable element lines and analytical parameters in respect of sensitivity, choice of elements and possible line overlaps. It provides a list of potential problems and an indication on how these can be solved.

All element lines which are missing in the Line Library are automatically created with the required parameters. Furthermore, a template calibration sample is created that can be used to build the matrix data.

In case of ambiguities or problems for the selection of element lines, additional functions are provided to quickly check the set-up of lines with a typical sample in terms of scans, to check line overlaps and the sensitivities, and energy profiles to check higher order overlaps.

The Analytical Assistant is a program that is based on a so called **Knowledge Table** that contains the analytical parameters and potential overlaps for each possible element line.

Whenever a function of the Analytical Assistant is used, it acts as a “Wizard”, meaning that only appropriate parameters are selected to perform the action. Furthermore, whenever the proposed parameters can be changed, then the selection of possible alternatives depends on the configuration of the instrument.

It should be noticed that in order to obtain full details about the functions provided by the Analytical Assistant, the chapters listed in the following table should also be consulted in the *WinXRF Configuration Manual*.

Chapter in Configuration Manual	Explanations about
Analytical Assistant	Knowledge Table and Selection Criteria
Goniometer Set-up	Scans, Line Library, Energy Profiles, Goniometer functions
Monochromator Set-up	Line Library, Energy Profiles
Special Elements and Required Samples	Manual Inputs, Virtual Elements, Calibration Samples, Setting-up Samples (Drift Correction), Samples for Line Overlap and Background Corrections
Analytical Programs	Internal Standards, Pseudo Elements
Instrument Calibration	Offline Analysis (Line overlap Correction)

Exit

Description of the Functions

The main screen consists of three sections:

Periodic Chart

Whenever an action is selected, the elements that can be analysed with the XRF technique and the actual instrument configuration will be highlighted for selection.

Actions The buttons shown in this group box are contextual, i.e. new buttons will be available depending on the selected action.

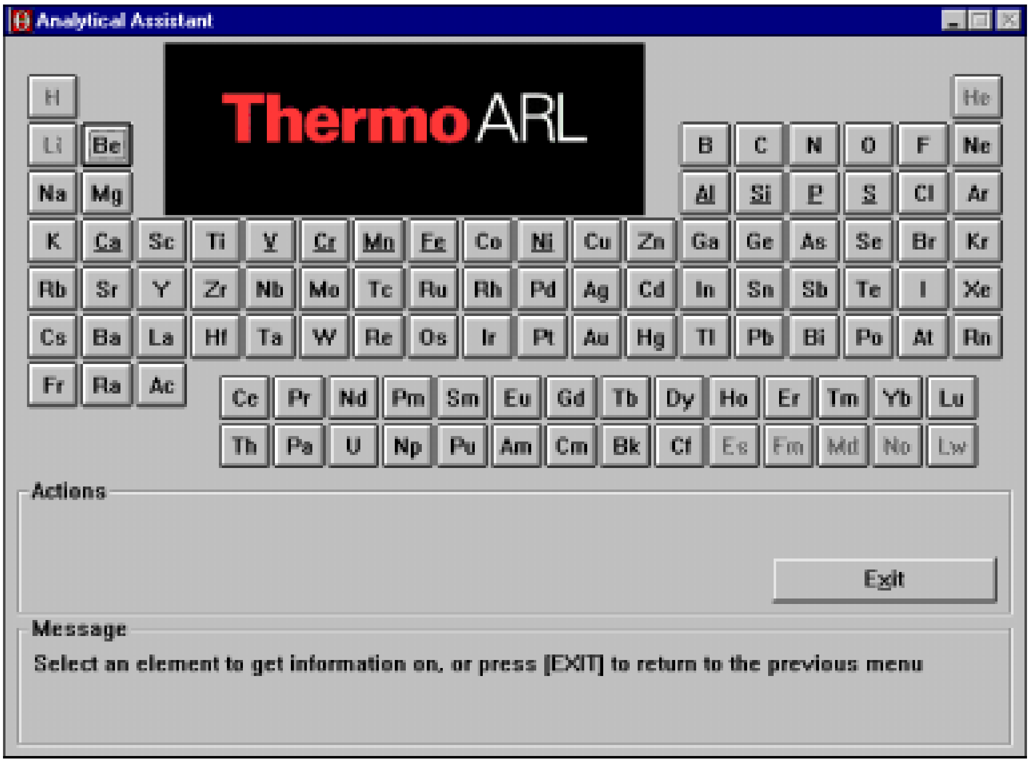
Messages Dynamic information is given in this box for guidance.

Element Information

This function gives useful information about the selected element in terms of analytical parameters (lines, crystals, detectors, PBF, etc.) that are best suited with respect to the configuration of the instrument.

When this function is selected, the periodic chart is highlighted, allowing the required element to be selected. Only the elements that can be analysed with the XRF technique and the actual instrument configuration are highlighted for selection.

The **Actions** group box only contains the **Exit** button which allows you to leave this function.

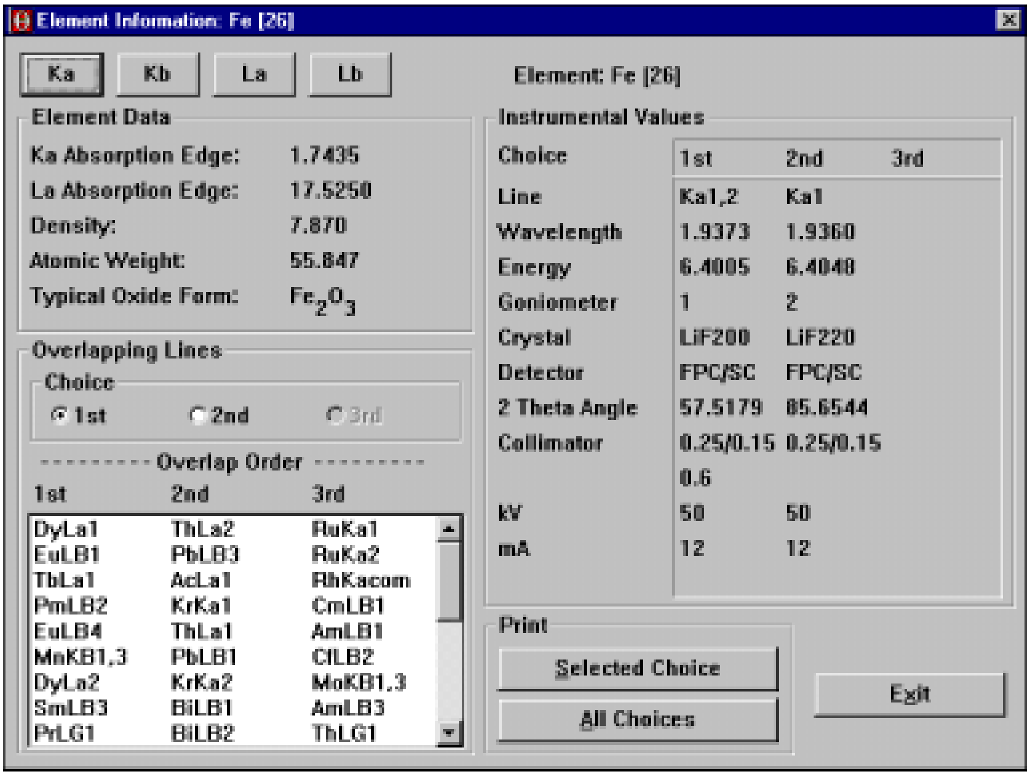


Select an element to get information on, or press [EXIT] to return to the previous menu

Figure 6.2

After an element has been selected, a screen similar to the one shown in the next figure will be displayed.

Depending on the selected element up to five different lines (i.e. Ka, Kb, La, Lb or Ma) are available for further investigations. The line selection buttons are arranged in such a way that the sensitivity of the corresponding lines for the selected element decreases from left to right. By default, the information of the most sensitive line is displayed.



Element: Fe [26]

Element Data

Ka Absorption Edge: 1.7435
La Absorption Edge: 17.5250
Density: 7.870
Atomic Weight: 55.847
Typical Oxide Form: Fe_2O_3

Overlapping Lines

Choice: ● 1st ○ 2nd ○ 3rd

Overlap Order

1st	2nd	3rd
DyLa1	ThLa2	RuKa1
EuLB1	PbLB3	RuKa2
TbLa1	AcLa1	RhKacom
PmLB2	KrKa1	CmLB1
EuLB4	ThLa1	AmLB1
MnKB1,3	PbLB1	ClLB2
DyLa2	KrKa2	MoKB1,3
SmLB3	BiLB1	AmLB3
PrLG1	BiLB2	ThLG1

Instrumental Values

Choice	1st	2nd	3rd
Line	Ka1,2	Ka1	
Wavelength	1.9373	1.9360	
Energy	6.4005	6.4048	
Goniometer	1	2	
Crystal	LiF200	LiF220	
Detector	FPC/SC	FPC/SC	
2 Theta Angle	57.5179	85.6544	
Collimator	0.25/0.15 0.6	0.25/0.15	
kV	50	50	
mA	12	12	

Print

Figure 6.3

The information displayed is divided into three parts:

Element Data

This area contains the **Ka** and **La Absorption Edge** values in Angstroms, the **Density** of the element in g/cm^3, the **Atomic Weight** and its **Typical Oxide Form**.

Instrumental Values

This area shows up to three sets of instrumental and analytical parameters for the selected element in the order of decreasing sensitivity.

- If more than one detector is given for a choice, it means that in principle, both detectors could be used, because the wavelength of the element line corresponds to the range where the sensitivity of the two detectors is almost equal. Only the energetic resolution of the detectors may be different and could therefore be the deciding factor for the choice of the detector to be used. This, of course, can be checked with energy profiles with both detectors.
- If more than one collimator is given for a choice, it means that in principle, both collimators could be used, but in case of any doubts, this should be checked by scans with a typical sample.

Note: If an element can only be measured with a monochromator (ARL 9800 series), then the information is restricted to the physical data relevant for that element (Line, Wavelength and Energy) and the proposed tube conditions (kV and mA).

Exit

Overlapping Lines

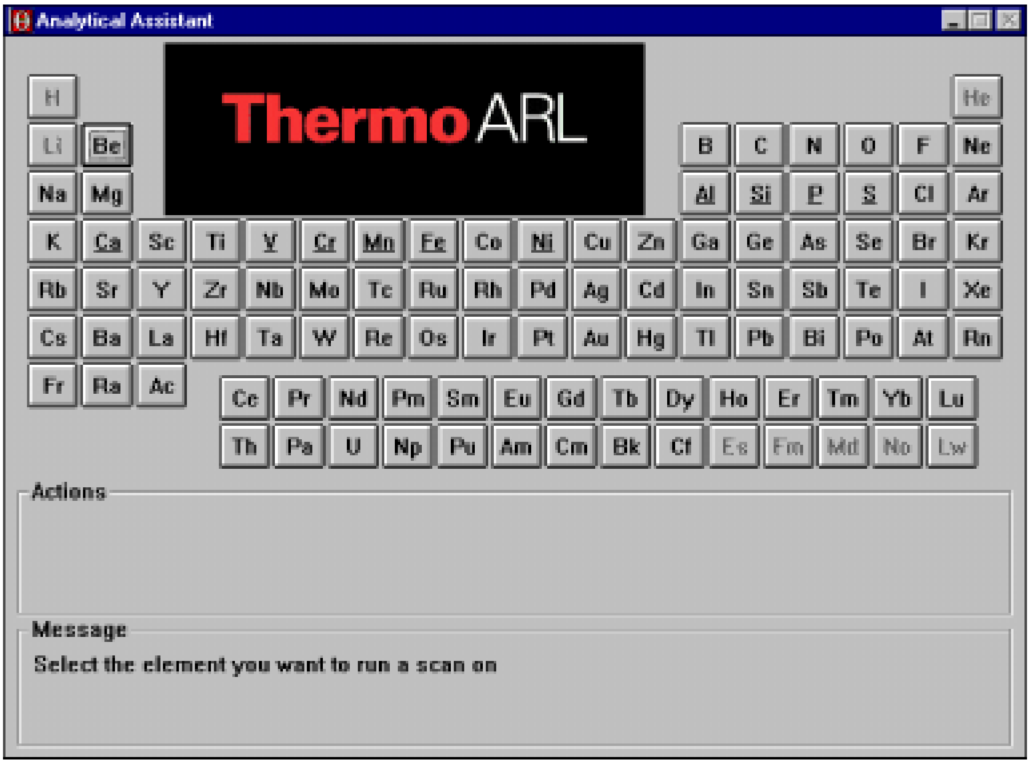
The list box in this area shows up to 20 potential overlaps for up to three **Overlap** (reflection) **Orders** of the selected **Choice**. These overlaps are given with decreasing importance.

The information displayed on this screen can be printed either for the **Selected Choice**, or for **All Choices**. This screen can be left by pushing the **Exit** button, and the next element can be chosen using the periodic chart.

Scan

This function allows you to perform a short scan over the selected element line with the analytical parameters (lines, crystals, detectors, PBF, etc.) that are best suited with respect to the configuration of the instrument. When this function is selected, the periodic chart is highlighted, allowing the desired element to be selected. Only the elements that can be analysed with the XRF technique and the actual goniometer configuration are available for selection. Thus, if the instrument is equipped with monochromators for ultra light elements requiring special crystals, then such elements are, of course, not available for selection (this only applies to the ARL 9800 series).

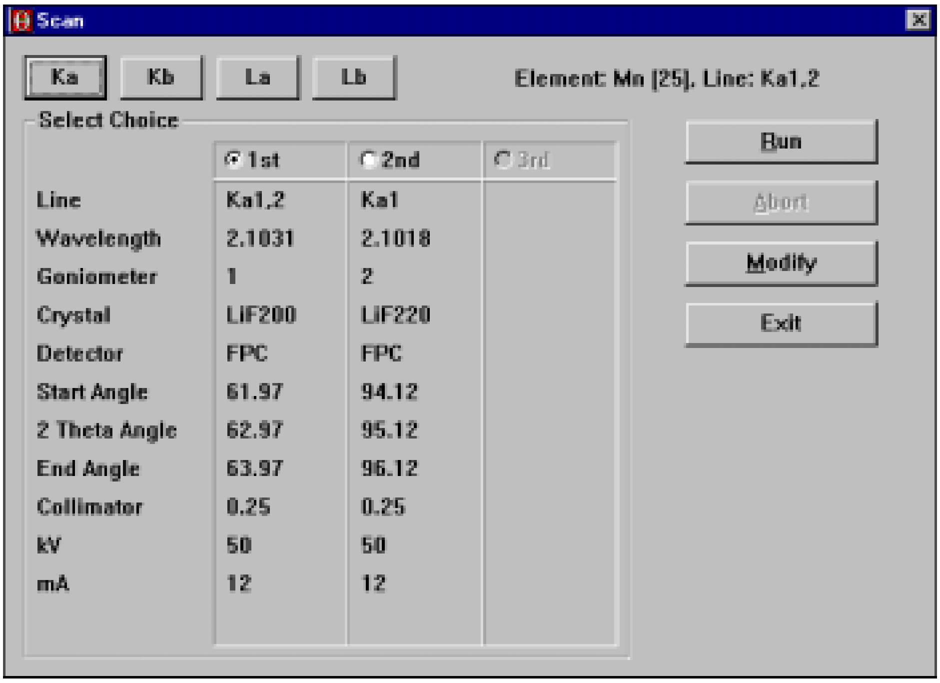
The **Actions** group box contains no buttons, and therefore, this function can only be left with the **Exit** button of the subsequent dialog box (see Figure 6.5).



Select the element you want to run a scan on

Figure 6.4

After an element has been selected, a screen similar to the one shown in the next figure will be displayed.



Element: Mn [25], Line: Ka1,2

Select Choice

	● 1st	○ 2nd	○ 3rd
Line	Ka1,2	Ka1	
Wavelength	2.1031	2.1018	
Goniometer	1	2	
Crystal	LiF200	LiF220	
Detector	FPC	FPC	
Start Angle	61.97	94.12	
2 Theta Angle	62.97	95.12	
End Angle	63.97	96.12	
Collimator	0.25	0.25	
kV	50	50	
mA	12	12	

Figure 6.5

By default, the parameters of the most sensitive line are displayed, and up to three choices are available to perform the scan.

Depending on the selected element up to five different lines (i.e. Ka, Kb, La, Lb or Ma) are available for selection. The line selection buttons are arranged in such a way that the sensitivity of the corresponding lines for the selected element decreases from left to right.

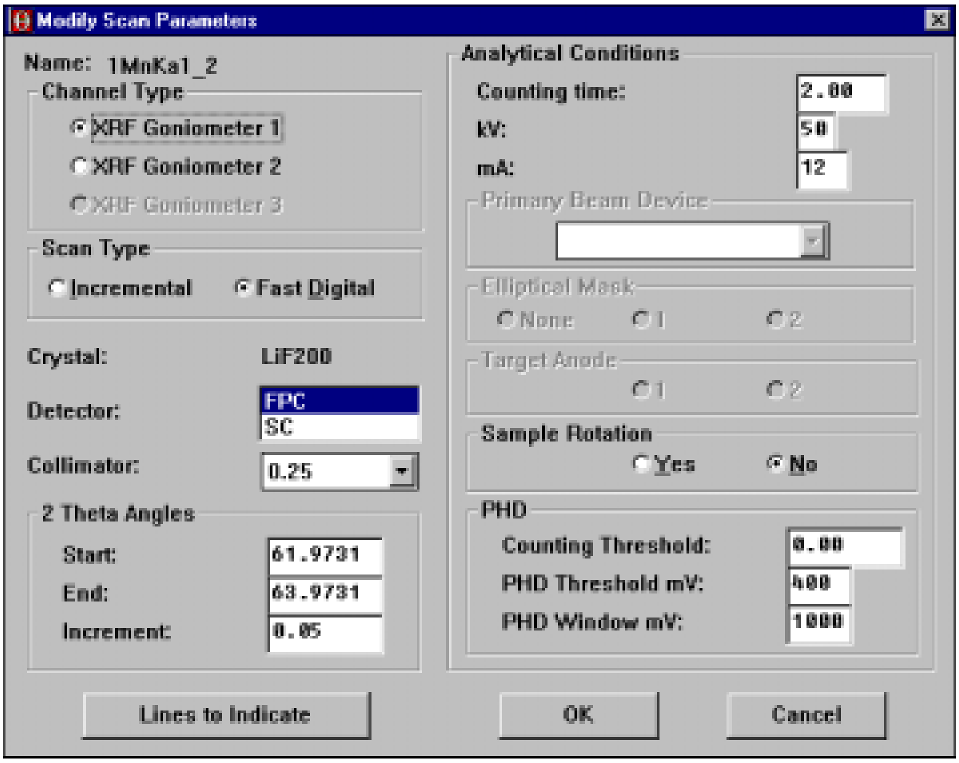
Select Choice	This area shows up to three sets of instrumental and analytical parameters for the selected element line in the order of decreasing sensitivity. The required element line is simply selected by its corresponding radio button.

The following buttons are available for further actions:

Run	Allows you to perform the scan.
Abort	Allows you to abort the scan after it has been started.
Modify	Allows you to select alternative parameters for the execution of the scan (see next section).
Exit	Allows you to leave the **Scan** function.

Selection of Alternative Scan Parameters

The **Modify** function allows you to change the parameters proposed by the Analytical Assistant. This may be necessary if, for instance, one wants to choose different parameters (i.e. range, increment, counting time, analytical conditions, etc.), or a different collimator/detector (if available) to perform the scan.



Modify Scan Parameters

Name: 1MnKa1_2

Channel Type: ● XRF Goniometer 1 ○ XRF Goniometer 2 ○ XRF Goniometer 3

Scan Type: ○ Incremental ● Fast Digital

Crystal: LiF200

Detector: FPC (selected), SC

Collimator: 0.25

2 Theta Angles
Start: 61.9731
End: 63.9731
Increment: 0.05

Analytical Conditions
Counting time: 2.00
kV: 50
mA: 12

Primary Beam Device:

Elliptical Mask: ○ None ○ 1 ○ 2

Target Anode: ○ 1 ○ 2

Sample Rotation: ○ Yes ● No

PHD
Counting Threshold: 0.00
PHD Threshold mV: 400
PHD Window mV: 1000

Figure 6.6

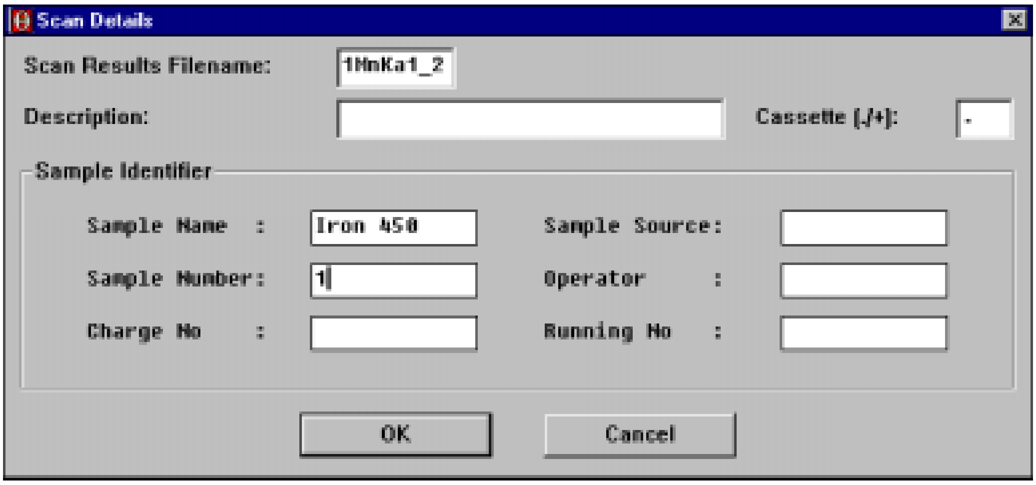
As can be seen in the figure above, only the detectors and collimators that are suitable for the selected element line can be chosen. However, concerning the other parameters, no restrictions exist.

On leaving the **Modify Scan Parameters** dialog box with **OK**, all modifications will be shown in the former dialog box (see Figure 6.5).

If the **Exit** button is pushed, this function is left without retaining the modifications.

Run a Scan

Once the parameters have been selected either with the radio buttons (Figure 6.5) or through the **Modify** function (Section *Selection of Alternative Scan* Parameters) the scan can be executed with the **Run** button. Upon selecting this function, the following dialog box is displayed.



Scan Details

Scan Results Filename: 1MnKa1_2

Description:

Cassette (./+): .

Sample Identifier
Sample Name : Iron 450
Sample Number: 1
Charge No :
Sample Source:
Operator :
Running No :

Figure 6.7

The following details can be specified in this dialog box:

Scan Results Filename	This field shows the proposed scan filename which can however be changed if necessary.
Description	This field is optional and allows you to enter a description of the analysed sample or any other information about the scan to be performed.
Cassette (./+)	The cassette or position number where the sample is located has to be specified here.
Sample Identifier	At least one of the globally defined sample identification fields has to be specified in order to commence the scan; if no input is given an appropriate error message will appear.

The scan can then be executed by selecting the **OK** button, causing the former dialog box (see Figure 6.5) to be displayed as long as the scan takes to be completed. Once completed, the initial periodic chart screen is displayed again (see Figure 6.1).

The recorded scan can then be displayed with the standard scan graphics function, as is explained in the *WinXRF Operator Manual*.

If the **Cancel** button is pushed, the **Scan** function is left immediately.

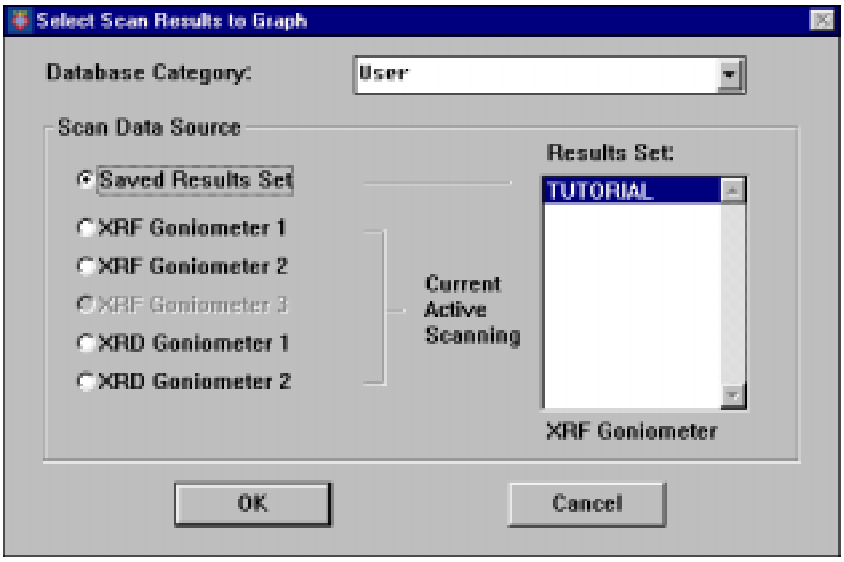
Scan Graphics

During the scanning process it is possible to observe the ongoing acquisition of the data points.

- In the main menu click on **Production / Analysis** and then click on **Qualitative Scans**.
- Click on **View Scans**.

Figure 6.8

- ♦ If one wants to display the ongoing graphic acquisition while the scan is performed, click on **XRF Goniometer 1**.
- ♦ If one wants to display a stored result file, click on **Saved Results Set** and select the desired scan file in the list box.
- ♦ Click on **OK**.



Database Category: User

● Saved Results Set
○ XRF Goniometer 1
○ XRF Goniometer 2
○ XRF Goniometer 3
○ XRD Goniometer 1
○ XRD Goniometer 2

Results Set: TUTORIAL

Figure 6.9

The scan is now displayed graphically (see figures in Section *Overlap Investigation*).

- ♦ By clicking on **Options** the scan graphic scales can be modified, for example to logarithmic scale.

- To identify a peak double-click onto the top of the peak you want to identify. This causes the system to display a list box that contains the possible element lines of the marked interval.
- Select a line and click on **Add**.

The system now shows where the selected line and its corresponding family lines (lines of the same spectral series) appear in the spectrum. If there is a match between the displayed lines and peaks one can be quite sure that the identification is correct.

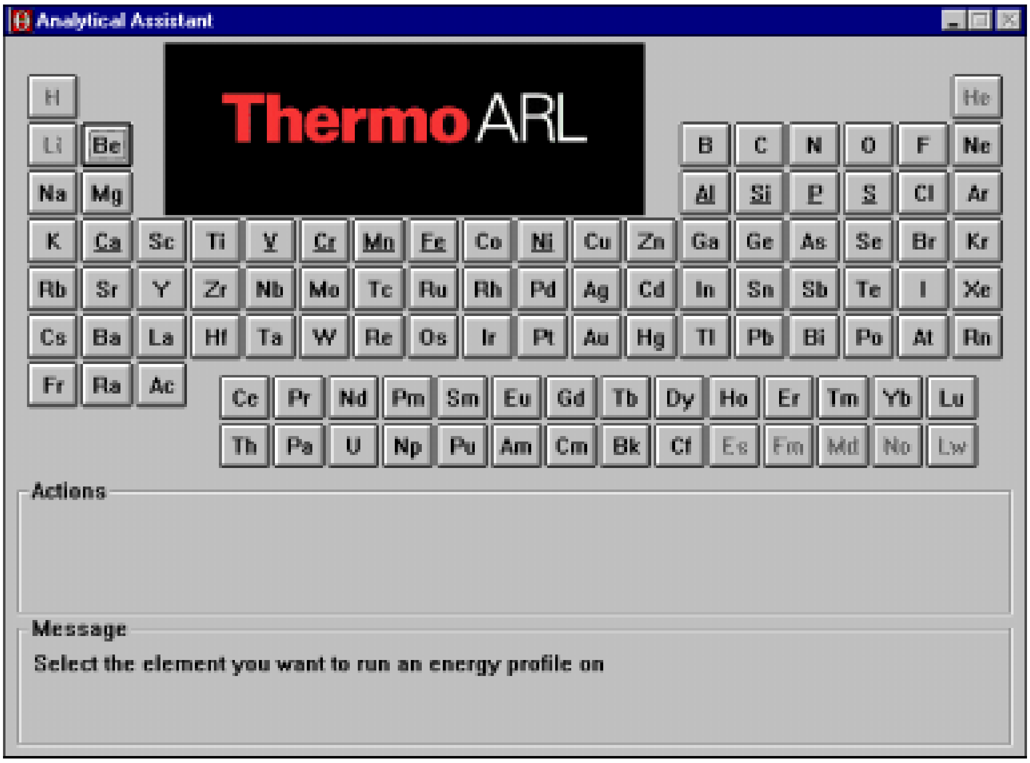
- This can be confirmed by clicking on **Finish**, causing the family lines to disappear and the identified peak to be labelled. The other peaks of the spectrum can be identified in the same way.
- The scan graphic can be printed with a report by clicking on **Print**.

Energy Profile

This function allows you to perform an energy profile over the selected element line with the analytical parameters (lines, crystals, detectors, PBF, etc.) that are best suited with respect to the configuration of the instrument.

When this function is selected, the periodic chart is highlighted, allowing the desired element to be selected. Only the elements that can be analysed with the XRF technique and the actual instrument configuration are available for selection. If for an element a monochromator is also present, then the energy profile with the monochromator has to be done using the standard function (this only applies to the ARL 9800 series).

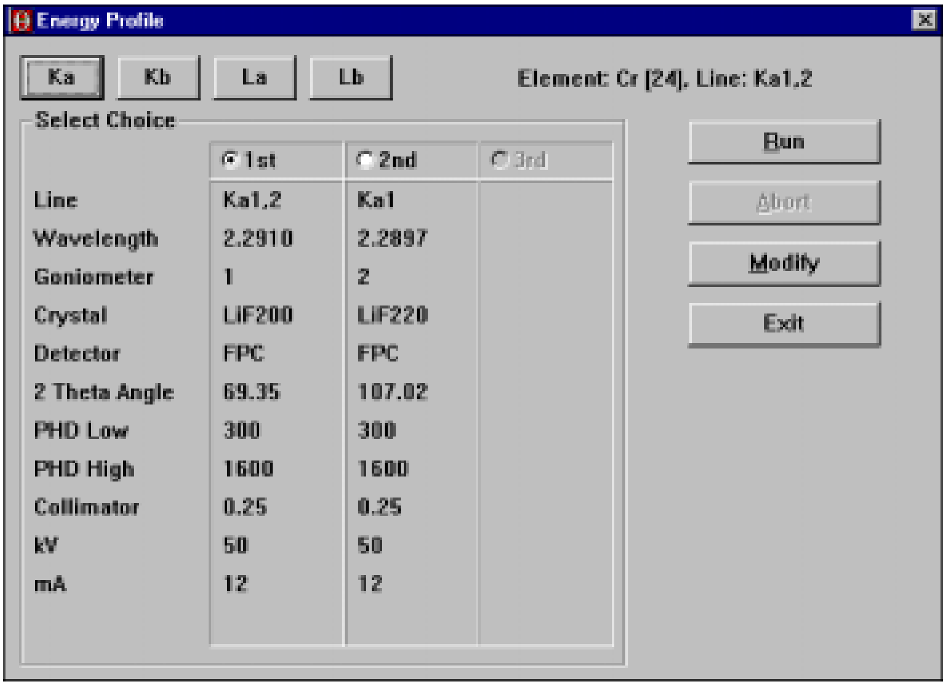
The **Actions** group box contains no buttons, and therefore, this function can only be left with the **Exit** button of the subsequent dialog box (see Figure 6.11).



Select the element you want to run an energy profile on

Figure 6.10

After an element has been selected, a screen similar to the one shown in the next figure will be displayed.



Element: Cr [24], Line: Ka1,2

Select Choice

	● 1st	○ 2nd	○ 3rd
Line	Ka1,2	Ka1	
Wavelength	2.2910	2.2897	
Goniometer	1	2	
Crystal	LiF200	LiF220	
Detector	FPC	FPC	
2 Theta Angle	69.35	107.02	
PHD Low	300	300	
PHD High	1600	1600	
Collimator	0.25	0.25	
kV	50	50	
mA	12	12	

Figure 6.11

By default, the parameters of the most sensitive line are displayed, and up to three choices are available to perform the energy profile for an element with the goniometer. If an element that can only be measured with a monochromator was selected, then the standard parameters for monochromators are selected (this only applies to the ARL 9800 series).

Depending on the selected element up to five different lines (i.e. Ka, Kb, La, Lb or Ma) are available for selection. The line selection buttons are arranged in such a way that the sensitivity of the corresponding lines for the selected element decreases from left to right.

Select Choice This area shows up to three sets of instrumental and analytical parameters for the selected element line in the order of decreasing sensitivity. The required element line is simply selected by its corresponding radio button.

The following buttons are available for further actions:

Run Allows you to perform the energy profile.

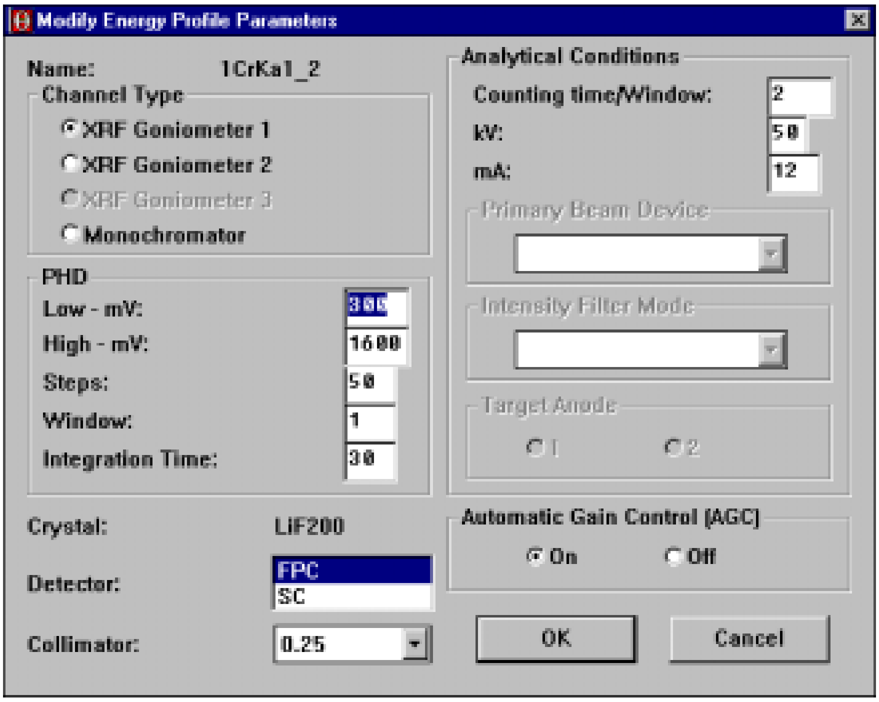
Abort Allows you to abort the energy profile after it has been started.

Modify Allows you to select alternative parameters for the execution of the energy profile (see next section).

Exit Allows you to leave the **Energy Profile** function.

Selection of Alternative Energy Profile Parameters

The **Modify** function allows you to change the parameters proposed by the Analytical Assistant. This may be necessary if, for instance, one wants to choose different **PHD** parameters or **Analytical Conditions**, or a different collimator/detector (if available) to perform the energy profile.


Modify Energy Profile Parameters

Name: 1CrKa1_2

Channel Type
- ● XRF Goniometer 1
- ○ XRF Goniometer 2
- ○ XRF Goniometer 3
- ○ Monochromator

PHD
- Low - mV: 300
- High - mV: 1600
- Steps: 50
- Window: 1
- Integration Time: 30

Crystal: LiF200

Detector: FPC
SC

Collimator: 0.25

Analytical Conditions
- Counting time/Window: 2
- kV: 50
- mA: 12

Primary Beam Device

Intensity Filter Mode

Target Anode ○ 1 ○ 2

Automatic Gain Control (AGC) ● On ○ Off

Figure 6.12

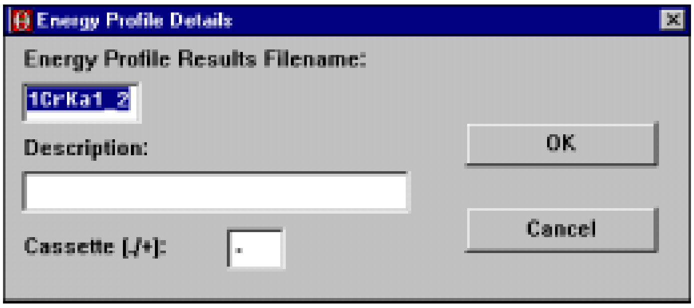
As can be seen in the figure above, only the detectors and collimators that are suitable for the selected element line can be chosen. However, concerning the other parameters, no restrictions exist.

On leaving the **Modify Energy Profile Parameters** dialog box with **OK**, all modifications will be shown in the former dialog box (see Figure 6.11).

If the **Cancel** button is pushed, this function is left without retaining the modifications.

Run an Energy Profile

Once the parameters have been selected either with the radio buttons (Figure 6.11) or through the **Modify** function (Section *Selection of Alternative Energy Profile Parameters*) the energy profile can be executed with the **Run** button. Upon selecting this function, the following dialog box is displayed.


Energy Profile Details

Energy Profile Results Filename: 1CrKa1_2

Description:

Cassette (-/+): -

Figure 6.13

The following details can be specified in this dialog box:

Energy Profile Results Filename

This field shows the proposed energy profile filename which can however be changed if necessary.

Description This field is optional and allows you to enter a description of the analysed sample or any other information about the energy profile to be performed.

Cassette (./+) The cassette or position number where the sample is located has to be specified here.

The energy profile can then be executed by selecting the **OK** button, causing the former dialog box (see Figure 6.11) to be displayed as long as the energy profile takes to be completed. Once completed, the initial periodic chart screen is displayed again (see Figure 6.1).

The recorded energy profile can then be displayed with the standard energy profile graphics function.

If the **Cancel** button is pushed, the **Energy Profile** function is left immediately.

Display Energy Profiles

Recorded energy profiles can be examined by displaying them graphically on the screen. The following steps have to be executed to select the energy profile to display. Select **Calibration** from the main menu, then the **Energy Profile** folder followed by the **Energy Profile Graphics** icon.

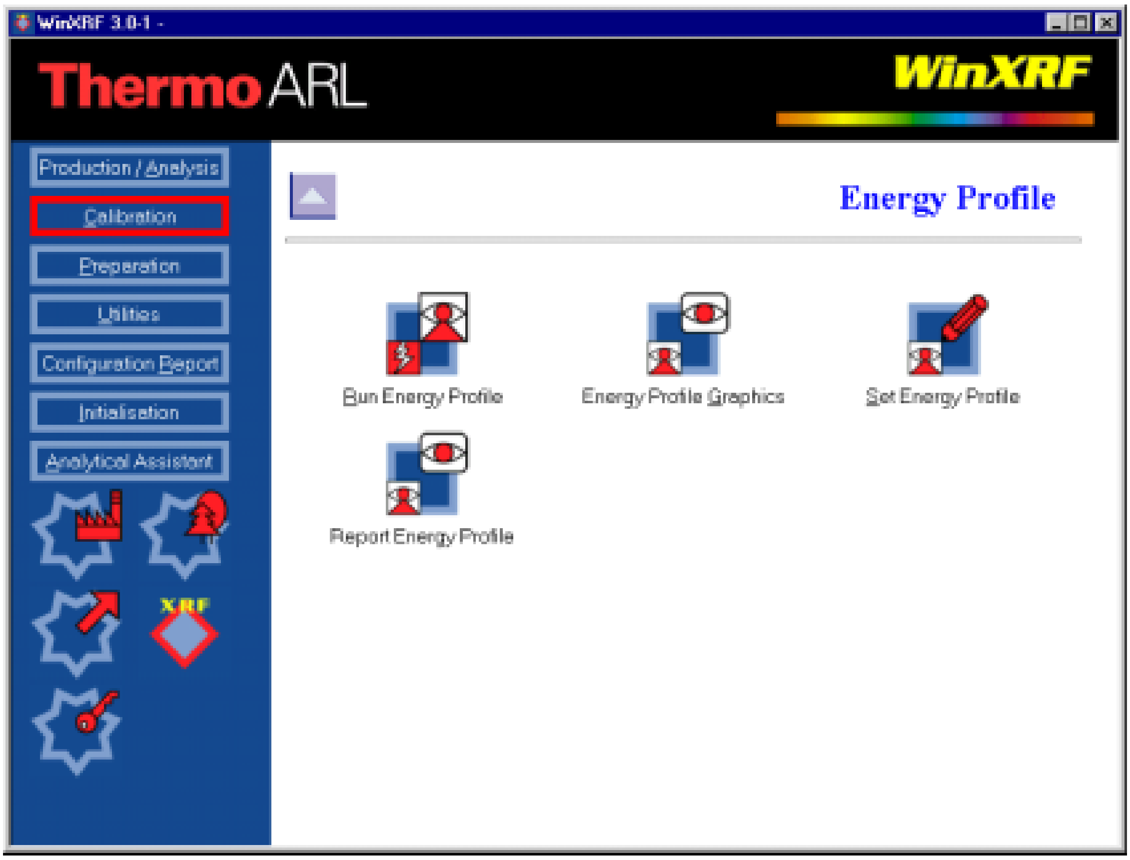


Figure 6.14

The dialog box shown below is then displayed, allowing to select the energy profile from the list box.

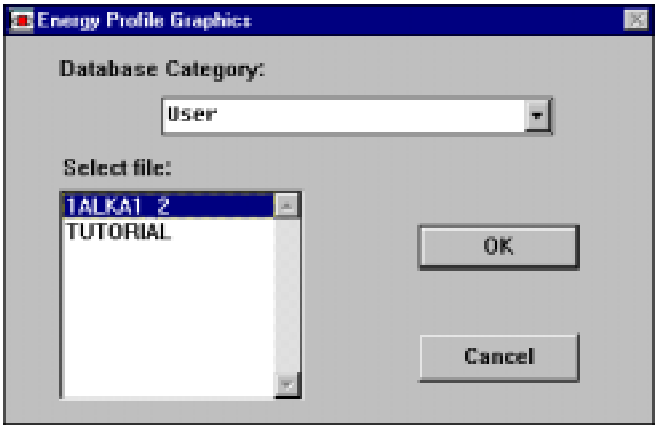


Figure 6.15

After confirmation by clicking onto the **OK** button, the selected energy profile is displayed (see figures in Section *Overlap Investigation*).

Analytical Program

This function represents the heart of the Analytical Assistant. Basically it allows easy creation of an analytical program with the most suitable element lines and analytical parameters in respect of sensitivity, choice of elements and possible line overlaps. It provides a list of potential problems and an indication on how these can be solved. Furthermore, it creates a template calibration sample to build the matrix data that will be used to perform the actual calibration work.

In case of ambiguities or problems with the selection of element lines, additional functions are provided to quickly check the set-up of lines with a typical sample with the aid of scans, to check line overlaps and sensitivities, and energy profiles to check higher order line overlaps.

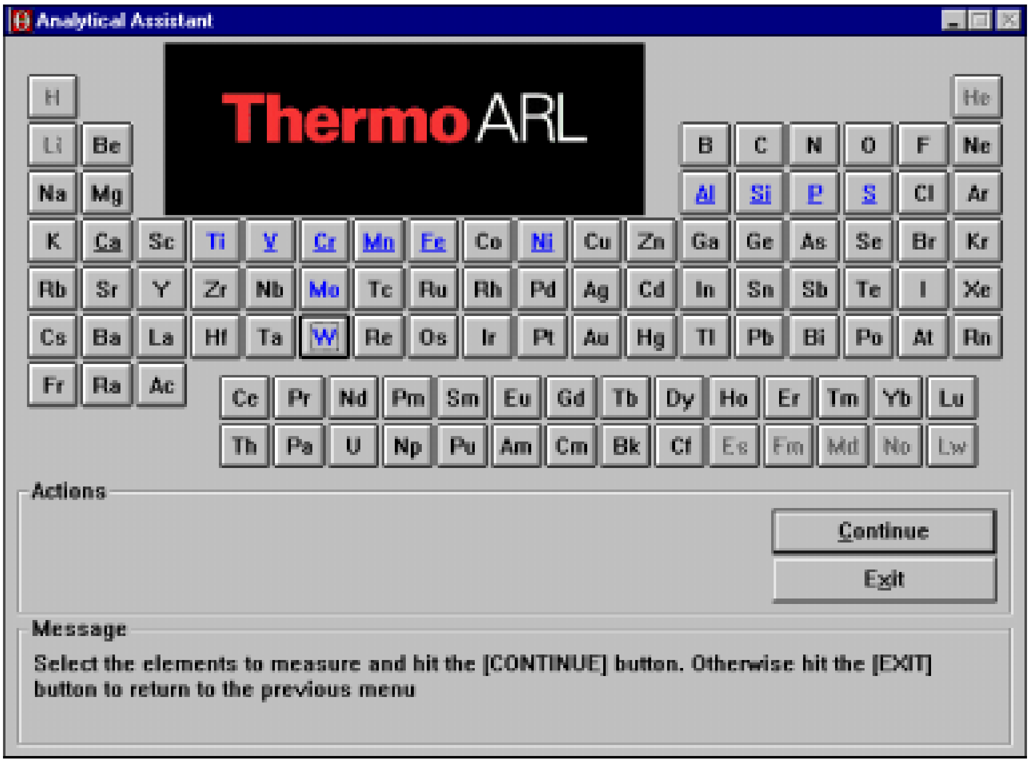
Select Elements

The elements to be included in the Analytical Program can be selected by pushing the **Analytical Program** button which causes the periodic chart to become highlighted. Only the elements that can be analysed with the XRF technique and the actual instrument configuration are available for selection.

The elements are selected by pushing the corresponding buttons, and the selected elements will be shown in blue. These buttons have a toggle function, i.e. a second push on a selected element button deselects this element, and therefore, will be shown in black again.

Hint: In order to follow the explanations given below for various analytical problems, it is recommended that you select the same set-up in terms of elements and matrix details as is shown in the following figures. To start the example, please select the following elements:

Al, Si, P, S, Ti, V, Cr, Mn, Fe, Ni, Mo, W



Select the elements to measure and hit the [CONTINUE] button. Otherwise hit the [EXIT] button to return to the previous menu

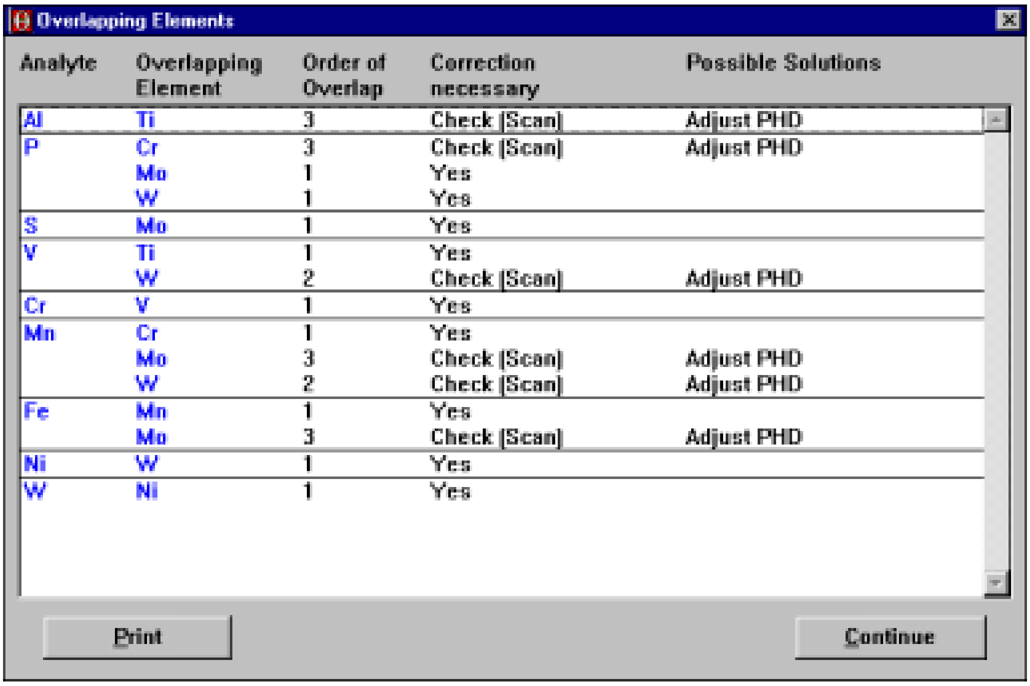
Figure 6.16

The **Actions** group box contains two buttons:

Continue	By pushing this button, the next step in the process of creating the Analytical Program is called. Please note, even if no elements have been selected, this button is still available but the next actions will be restricted to **Elements to Measure** and **Exit**.
Exit	By pushing this button, the creation of the Analytical Program is cancelled and the initial screen is displayed again (Figure 6.1).

Note: Elements that can only be measured with monochromators will not be included in the following evaluation process, and thus the investigation of potential overlaps has to be done with the **Element Info** function.
Elements for which monochromators exist but can also be measured with the goniometer, the evaluation will be done with the corresponding goniometer lines. This only applies to the ARL 9800 series.

After having selected the elements, the **Continue** button has to be pushed which causes the table shown in the next figure to be displayed.



Analyte	Overlapping Element	Order of Overlap	Correction necessary	Possible Solutions
Al	Ti	3	Check [Scan]	Adjust PHD
P	Cr	3	Check [Scan]	Adjust PHD
	Mo	1	Yes	
	W	1	Yes	
S	Mo	1	Yes	
V	Ti	1	Yes	
	W	2	Check [Scan]	Adjust PHD
Cr	V	1	Yes	
Mn	Cr	1	Yes	
	Mo	3	Check [Scan]	Adjust PHD
	W	2	Check [Scan]	Adjust PHD
Fe	Mn	1	Yes	
	Mo	3	Check [Scan]	Adjust PHD
Ni	W	1	Yes	
W	Ni	1	Yes	

Figure 6.17

This table consists of the following items:

Analyte This column shows the analyte elements that are affected by overlapping elements. At this stage, all elements are shown in blue, meaning that the concentration level is **Minor** (see Section *Matrix Details*).

Overlapping Element
This column shows the elements that are overlapping an analyte element. At this stage, all elements are shown in blue, meaning that the concentration level is **Minor** (see Section *Matrix Details*).

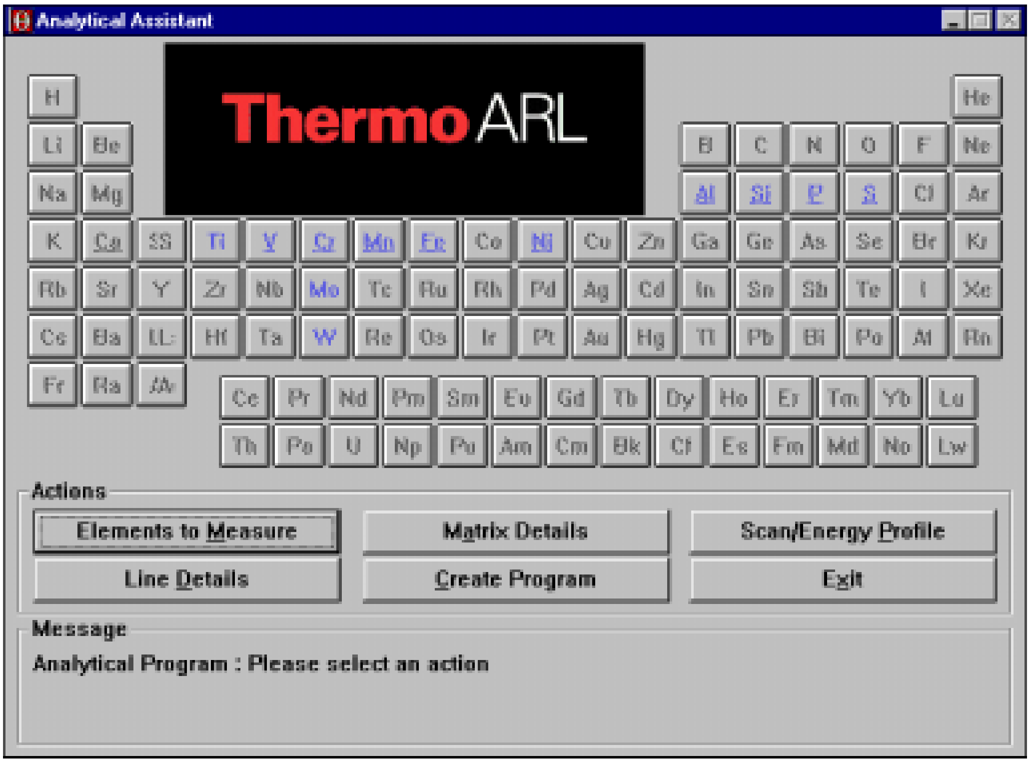
Order of Overlap
This column shows the overlap (reflection) order of the overlapping element. A first order overlap normally needs to be corrected by an overlap correction, and for higher orders an adjustment of the PHD settings with the help of an energy profile on a typical sample is in many cases sufficient.

Correction necessary
This column indicates whether an overlap correction is necessary or not.

Possible Solutions
This column suggests how the overlap problem could be solved or at least minimised.

The table shown at this stage may contain a lot of potential problems and the user may think that the calibration process will require a lot of effort to work perfectly. But we should not forget that additional parameters and values need to be specified that may in some cases even increase the potential problems but in most cases the contrary is true.

It is therefore recommended to simply **Print** this table and then leave it by pushing the **Continue** button. The screen as shown in Figure 6.18 is then displayed, but now containing numerous additional buttons in the **Actions** group box that are described briefly after the figure and in subsequent sections.



Analytical Program : Please select an action

Figure 6.18

Elements to Measure
Allows you to select or change the matrix elements.

Matrix Details
Allows you to specify details about the matrix to be calibrated.

Scan/Energy Profile
Allows you to perform scans and energy profiles on selected element lines of the matrix.

Line Details Allows you to display and change the proposed analytical parameters.

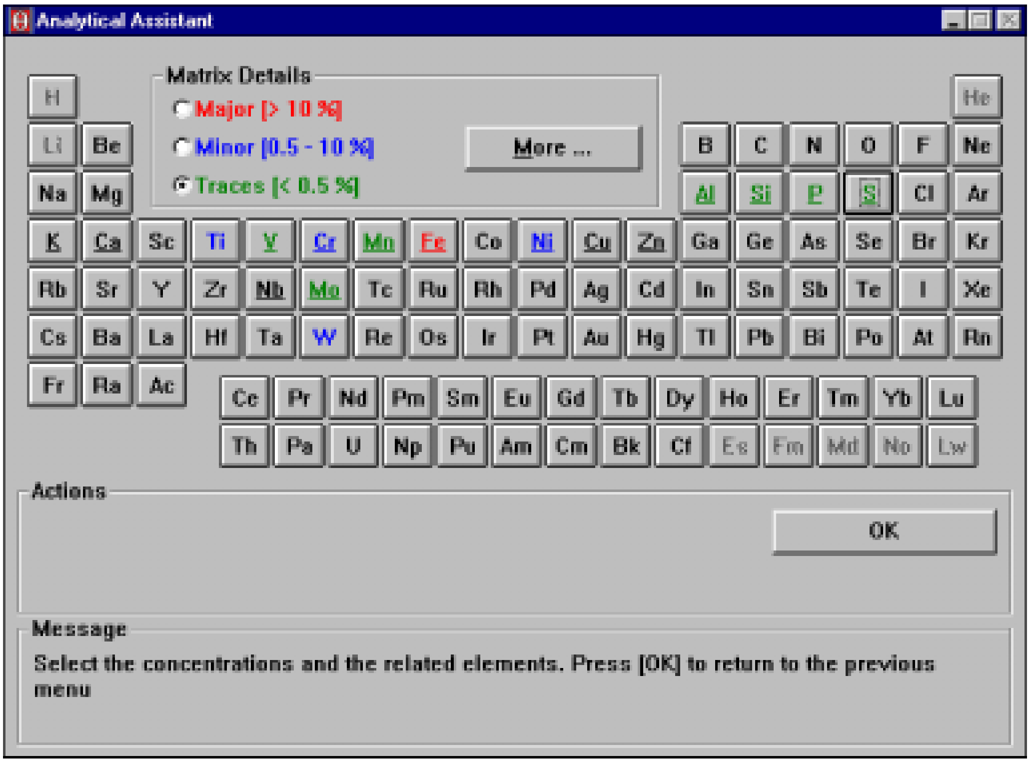
Create Program
Allows you to create the Analytical Program and the template calibration sample.

Exit When this button is pressed, the **Analytical Program** function is left. If no program was created, a warning message will be given (see Figure 6.36).

Matrix Details

The next step in creating the Analytical Program consists of specifying details about the matrix to be calibrated, and therefore, the button **Matrix Details** has to be pressed.

The screen as shown below is then displayed, where the ARL logo has been replaced by the **Matrix Details** group box. With the help of the three radio buttons the concentration levels can be specified for each element in order to refine the solving procedures for potential overlap problems.



○ Major [> 10 %]
○ Minor [0.5 - 10 %]
● Traces [< 0.5 %]

Select the concentrations and the related elements. Press [OK] to return to the previous menu

Figure 6.19

Hint: For our example, the following selections should be made:

Major:	**Fe**
Minor:	**Ti, Cr, Ni, W**
Trace:	**Al, Si, P, S, V, Mn, Mo**

The concentration levels are specified in the following way:

- Select the level.
- Click on the desired elements. Please note that a second click will deselect the element!
- Repeat the procedure for the other levels.

Note: The indicated percentage levels should not be taken too seriously. Designating elements as **Majors** will have a strong influence on the choice of instrumental parameters so as to reduce the sensitivity of the element line. The selection of these instrumental parameters is done in such a way that the detection system will not be saturated.

It should be noticed that the **More...** button becomes activated as soon as any one element has been specified as a **Major** element. This function is explained in the following section.

More Matrix Details

The details specified in this dialog box will only influence the selection of instrumental parameters to reduce the sensitivity of the elements specified as **Majors**.

Following details can be specified in the **Sample Type** and **Dilution** group boxes (see Figure 6.20):

Solid (No dilution)

This option is selected by default. It should be selected if the matrix to be calibrated consists of solid samples (i.e. metallic), pressed powder samples that are prepared without a binder, or undiluted liquid samples (e.g. oil). The **Dilution** group box remains greyed when this option is selected.

Pressed Powder

This option should only be selected if the sample preparation method for pressed powders requires a binder, and in such a case, the dilution ratio has to be specified. Conversely for undiluted pressed powers the option **Solid (No dilution)** should be chosen.

Fused Bead This option should be selected if the matrix consists of fused samples, and the dilution ratio has to be specified.

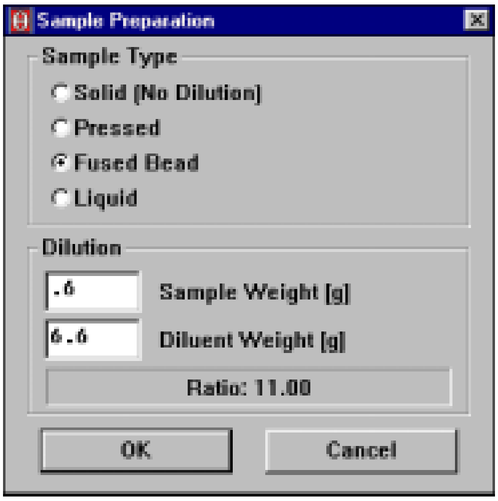
Liquid This option should only be selected if the matrix consists of diluted liquid samples, and in such a case, the dilution ratio has to be specified. Conversely, if the matrix is for instance oil, the option **Solid (No dilution)** should be chosen.

Sample Weight, g

The weight of the sample is specified here.

Diluent, g The weight of the dilution material, e.g. flux, binder, etc., is specified here. As an indication, the dilution ratio is calculated in the box underneath.

Hint: For our example, the option **Solid (No dilution)** should be selected.



Sample Preparation

Sample Type

○ Solid (No Dilution)

○ Pressed

● Fused Bead

○ Liquid

Dilution

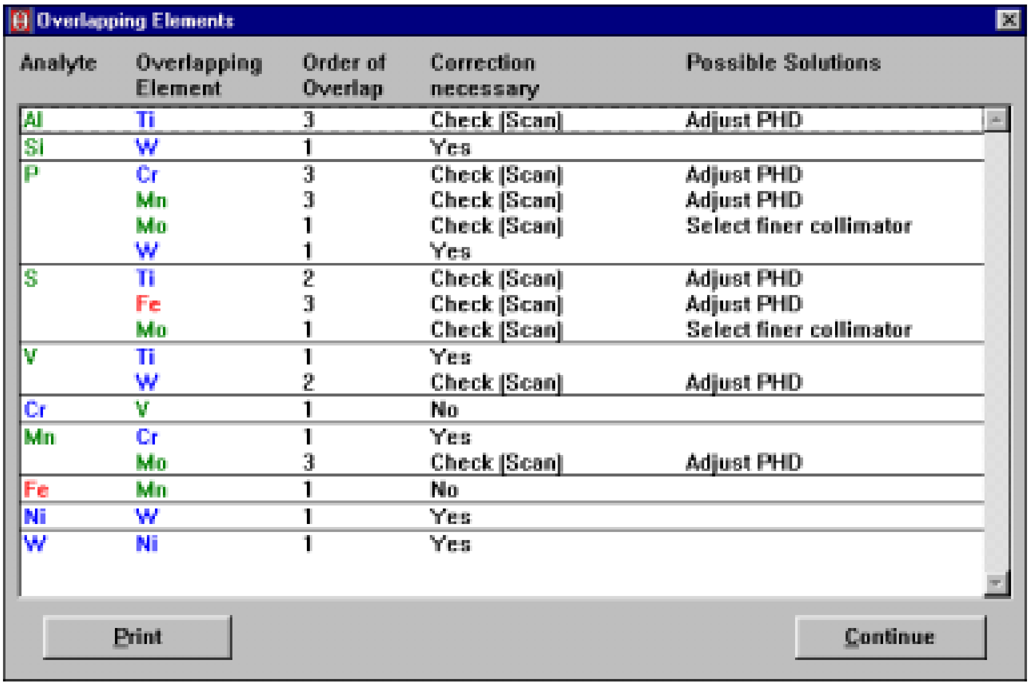
.6 Sample Weight [g]

6.6 Diluent Weight [g]

Ratio: 11.00

Figure 6.20

After confirmation of the **Matrix Details**, the table of **Overlapping Elements** is displayed again with new recommendations to solve the overlap problems (in our example, the overlap corrections necessary decreased from 9 to 5!). Also, the elements are now shown in the colour of their corresponding concentration level. It is, of course, suggested that to this table is **Printed** again, so that it can serve as a guide for solving the overlap problems.

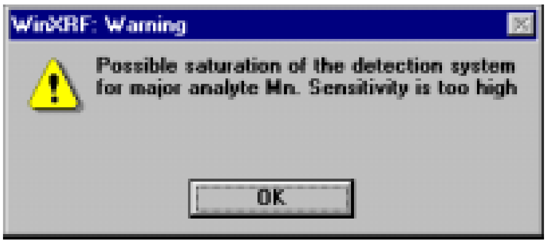


Analyte	Overlapping Element	Order of Overlap	Correction necessary	Possible Solutions
Al	Ti	3	Check [Scan]	Adjust PHD
Si	W	1	Yes	
P	Cr	3	Check [Scan]	Adjust PHD
	Mn	3	Check [Scan]	Adjust PHD
	Mo	1	Check [Scan]	Select finer collimator
	W	1	Yes	
S	Ti	2	Check [Scan]	Adjust PHD
	Fe	3	Check [Scan]	Adjust PHD
	Mo	1	Check [Scan]	Select finer collimator
V	Ti	1	Yes	
	W	2	Check [Scan]	Adjust PHD
Cr	V	1	No	
Mn	Cr	1	Yes	
	Mo	3	Check [Scan]	Adjust PHD
Fe	Mn	1	No	
Ni	W	1	Yes	
W	Ni	1	Yes	

Figure 6.21

Depending on the combination of selected elements and available instrumental parameters (i.e. crystals, collimators), it may not be possible to select the instrumental parameters that accomplish the criteria imposed by the knowledge table. The following two situations may occur:

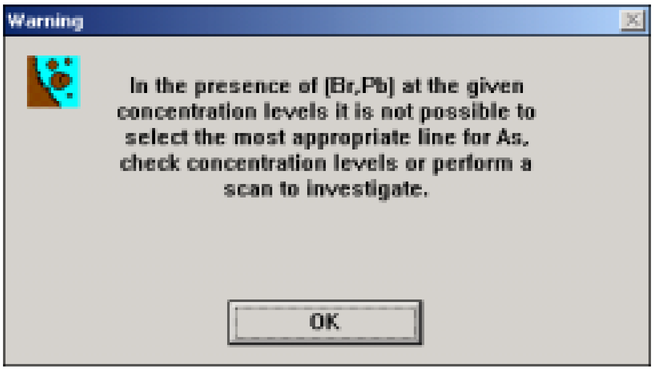
- **Saturation** of the detection system. In such a case, the warning message shown below is issued.



Possible saturation of the detection system for major analyte Mn. Sensitivity is too high

Figure 6.22

- No suitable instrumental parameters imposed by **Constraints** can be selected. In such a case, the warning message shown below is issued.



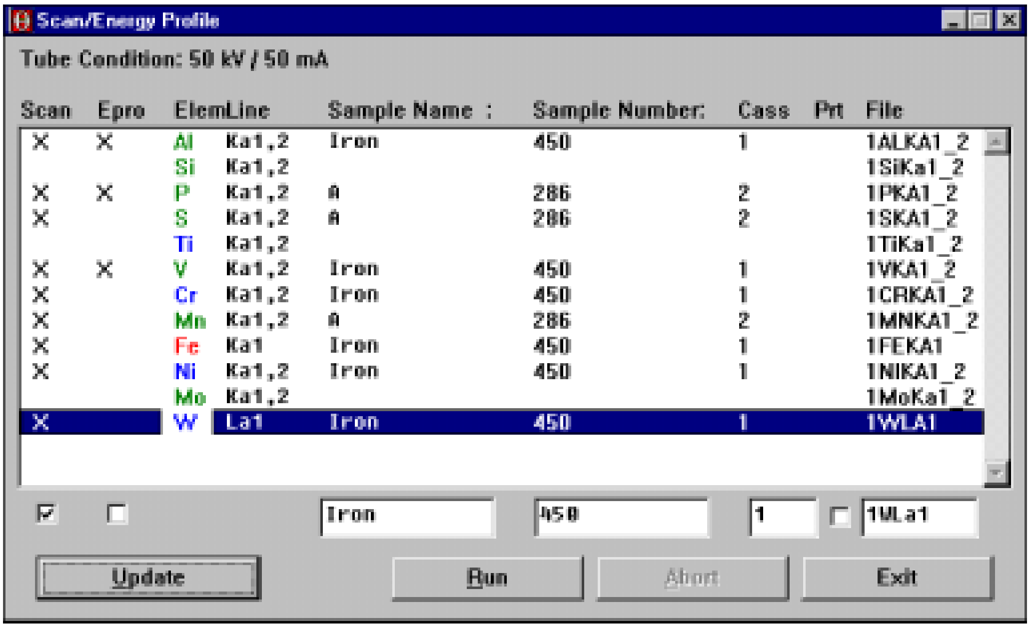
In the presence of [Br,Pb] at the given concentration levels it is not possible to select the most appropriate line for As, check concentration levels or perform a scan to investigate.

Figure 6.23

Scan/Energy Profile

In order to investigate the overlap problems it is recommended to perform scans and energy profiles according to the indications given by the Overlap Table. The easiest procedure consists of selecting a scan for all the elements that are listed in the **Analyte** column and an energy profile only for the analytes which are overlapped by a higher order line (see Figure 6.21).

However, it is important to select appropriate samples that reveal the consequence of the potential overlaps. These samples should thus contain the overlapping elements in amounts that are typical for the matrix. It could therefore be necessary to repeat the investigation for analytes with more than one overlapping element.



Tube Condition: 50 kV / 50 mA

Scan	Epro	ElemLine		Sample Name :	Sample Number:	Cass	Prt	File
X	X	Al	Ka1,2	Iron	450	1		1ALKA1_2
		Si	Ka1,2					1SiKa1_2
X	X	P	Ka1,2	A	286	2		1PKA1_2
X		S	Ka1,2	A	286	2		1SKA1_2
		Ti	Ka1,2					1TiKa1_2
X	X	V	Ka1,2	Iron	450	1		1VKA1_2
X		Cr	Ka1,2	Iron	450	1		1CRKA1_2
X		Mn	Ka1,2	A	286	2		1MNKA1_2
X		Fe	Ka1	Iron	450	1		1FEKA1
X		Ni	Ka1,2	Iron	450	1		1NIKA1_2
		Mo	Ka1,2					1MoKa1_2
X		W	La1	Iron	450	1		1WLA1

☑ ☐ Iron 450 1 ☐ 1WLa1

Figure 6.24

The selection is done by specifying the inputs for one line using the entry boxes at the bottom of the dialog box. Pressing the **Update** button copies the values into the highlighted line. Additional lines can be set up by simply performing a double-click on the corresponding line, or by pressing the **Update** button again after highlighting the line.

Please note that it is not necessary to specify the sample identities (although it is strongly recommended), but if the cassette number is not specified, then an appropriate error message is given.

The scans and energy profiles are started by pressing the **Run** button. The analytical and instrumental parameters used are as selected by the Analytical Assistant (see Section *Line Details*). While the instrument is occupied with this work, the dialog box is deactivated. The operation can however be **aborted** with the corresponding button. The scans and energy profiles are executed from top to bottom, and at the end, the system returns to the screen shown in Figure 6.18.

If the **Cancel** button is pressed, the dialog box is left immediately.

Overlap Investigation

After the scans are performed, the overlap effects have to be investigated. This is done with the **View Scan** function which is found in the **Qualitative Scans** folder in the **Production / Analysis** option of the main menu. Please refer to the *WinXRF Operator Manual* for more details about this feature.

Exit

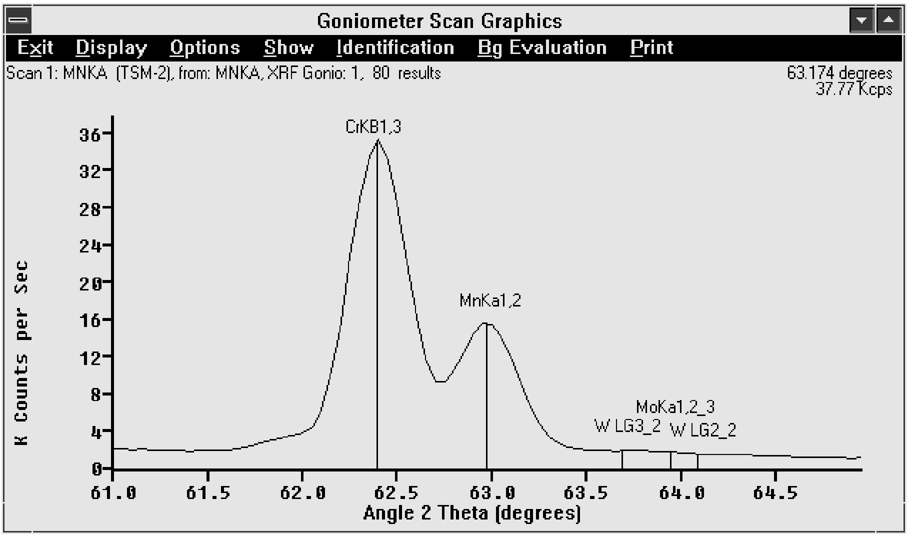
The energy profiles can be investigated with the **Energy Profile Graphics** icon which is part of **Energy Profiles** folder in the **Calibration** option of the main menu.

Before explaining how to overcome overlap problems, please consider the following guide lines:
- Third order overlaps can normally be ignored if the overlapping element is not a major.
- Second order overlaps only need attention if the affected analyte is a trace. In many cases, the overlap effect can be reduced or even eliminated by selecting a narrower PHD window and/or a finer collimator.
- The intervals for overlapping lines have been chosen large enough to also consider 'overlapping lines' which may only be a problem in very rare cases, because it is always better to be aware of a situation which is not critical than the opposite.

In order to understand how the various situations that may occur are to be judged, some of the most common situations based on our example are explained hereafter (see Figure 6.21). However, a lot of slightly different situations might occur which will require a good sense of judgement, as the whole is really a game between elemental composition of the matrix, relative concentrations among the elements and concentration ranges.

Analyte Mn (trace) is overlapped by Cr (1st order), Mo (3rd order) and W (2nd order)

The scan graphics in the figure below shows such a situation. It is obvious that the Cr overlap is too big to be ignored, and therefore, an overlap correction is necessary. On the other hand, the separation between Mn and W, respectively Mo, is quite good. Moreover, these two elements are interfering with higher order lines, it is therefore advisable to check the situation with an energy profile.

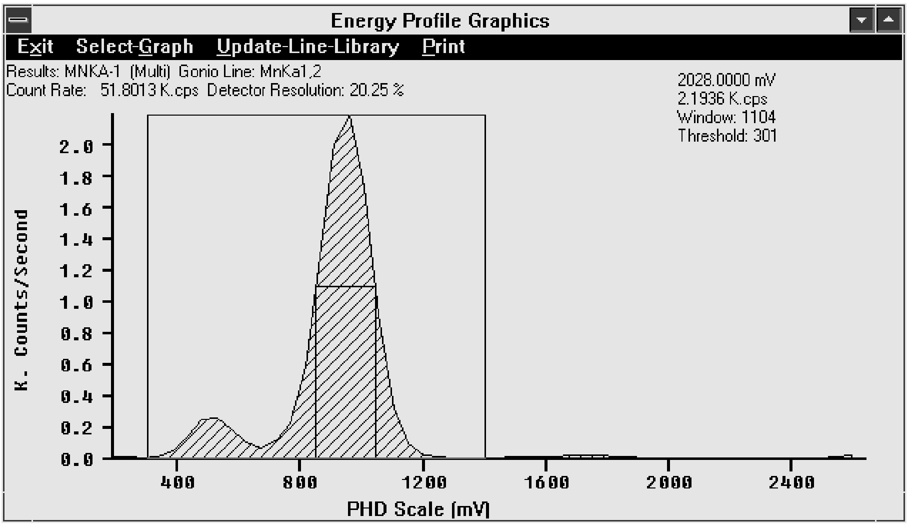


Scan 1: MNKA (TSM-2), from: MNKA, XRF Gonio: 1, 80 results

63.174 degrees
37.77 Kcps

Figure 6.25

The energy profile made with a typical sample shows that the higher order peaks are very weak. It is therefore not necessary to consider an overlap correction. However, the threshold value could be lowered slightly to include the escape peak of Mn.



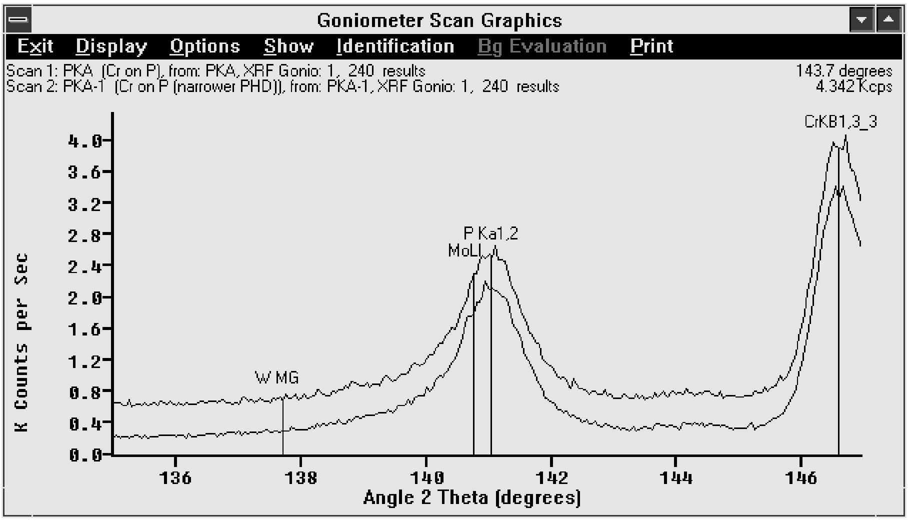
Results: MNKA-1 (Multi) Gonio Line: MnKa1,2
Count Rate: 51.8013 K.cps Detector Resolution: 20.25 %

2028.0000 mV
2.1936 K.cps
Window: 1104
Threshold: 301

Figure 6.26

Analyte P (trace) is overlapped by Mo (1st order), W (1st order) and Cr (3rd order)

The scan graphics in the figure below shows such a situation. The overlapping line of Mo belongs to a trace element and potentially could be ignored, but because the separation between the overlapping line (**MoLl**) and the analyte is so bad, an overlap correction is necessary in most cases, especially if the highest possible accuracy for the measurement of **P** is required. On the other hand, the two overlaps **W MG** and **CrKB**, third order, can be ignored.

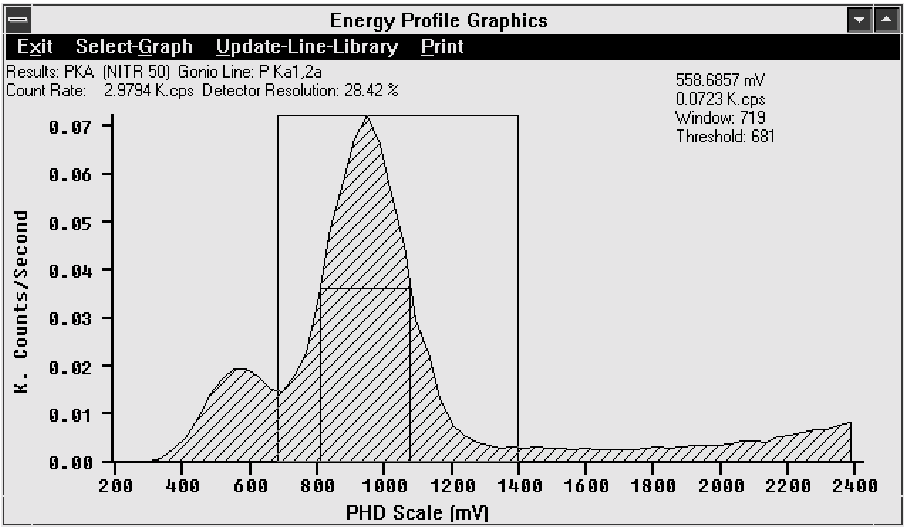


Scan 1: PKA (Cr on P), from: PKA, XRF Gonio: 1, 240 results
Scan 2: PKA-1 (Cr on P (narrower PHD)), from: PKA-1, XRF Gonio: 1, 240 results

143.7 degrees
4.342 Kcps

Figure 6.27

By performing an energy profile on a typical sample, a peak in front of the P peak can be noticed. Although this peak looks like an escape peak it is not such a peak. It is probably due to crystal fluorescence generated by Fe or Cr. Setting the threshold as shown in the next figure causes the background to be lowered drastically, as can be seen by the comparative scans shown in Figure 6.27).



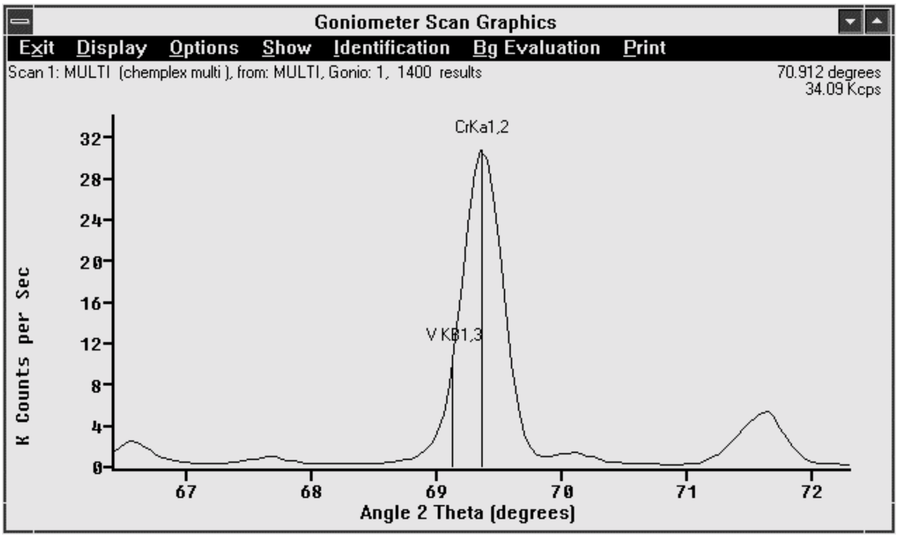
Results: PKA (NITR 50) Gonio Line: P Ka1,2a
Count Rate: 2.9794 K.cps Detector Resolution: 28.42 %

558.6857 mV
0.0723 K.cps
Window: 719
Threshold: 681

Figure 6.28

Analyte Cr (minor) is overlapped by V line (1st order)

The scan graphics in the figure below shows such a situation. Because the overlapping line belongs to a trace element, and the analyte is a minor element, an overlap correction is probably not necessary.



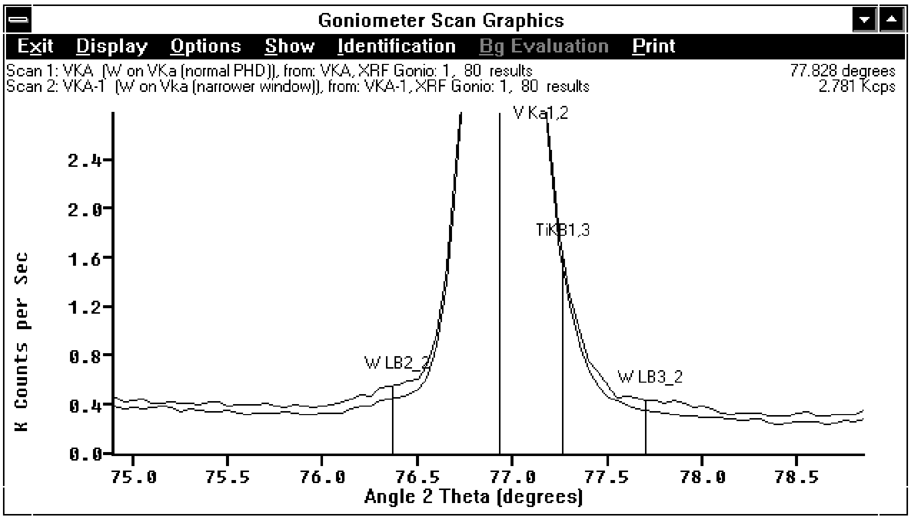
Scan 1: MULTI (chemplex multi), from: MULTI, Gonio: 1, 1400 results

70.912 degrees
34.09 Kcps

Figure 6.29

Analyte V (trace) is overlapped by Ti (1st order) and W (2nd order)

The comparison of the two scans shows the effect of the narrower PHD window on the overlapping **W LG2** and **W LG3** lines whereas the analyte line is only affected by the discrimination of the escape peak (Figure 6.31). It is essential to make an overlap correction for Ti at minor concentration levels interfering with V at trace levels.

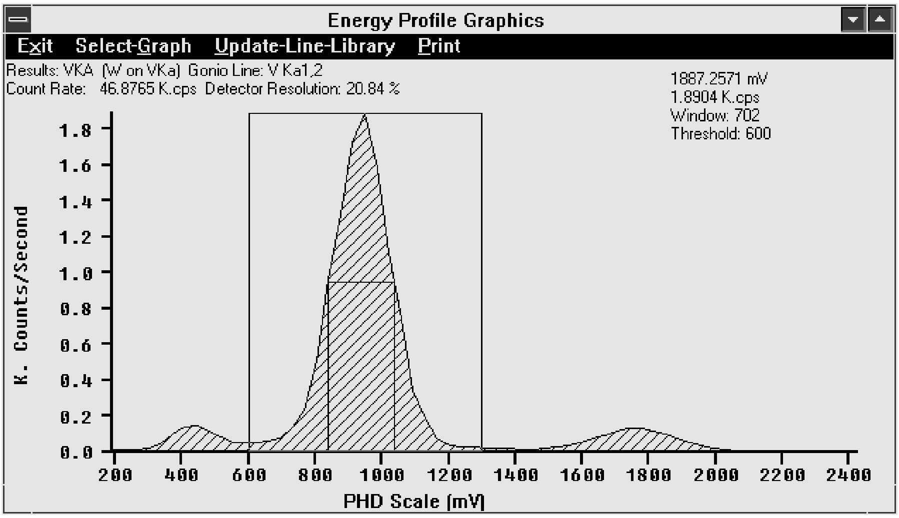


Scan 1: VKA (W on VKa (normal PHD)), from: VKA, XRF Gonio: 1, 80 results
Scan 2: VKA-1 (W on Vka (narrower window)), from: VKA-1, XRF Gonio: 1, 80 results

77.828 degrees
2.781 Kcps

Figure 6.30

The energy profile clearly shows the first order peak (V) at around 870mV and the second order peak (W) at twice the energy (1740mV). Due to the excellent energy resolution of the detector, the separation between the two peaks is very good and therefore the overlap effect can be eliminated by selecting a slightly narrower window. Also, in cases where there is a good separation between the escape and the main peaks it is a good practice to discriminate the escape peak. Please note that the default parameters to record the energy profile have been increased for clarity.



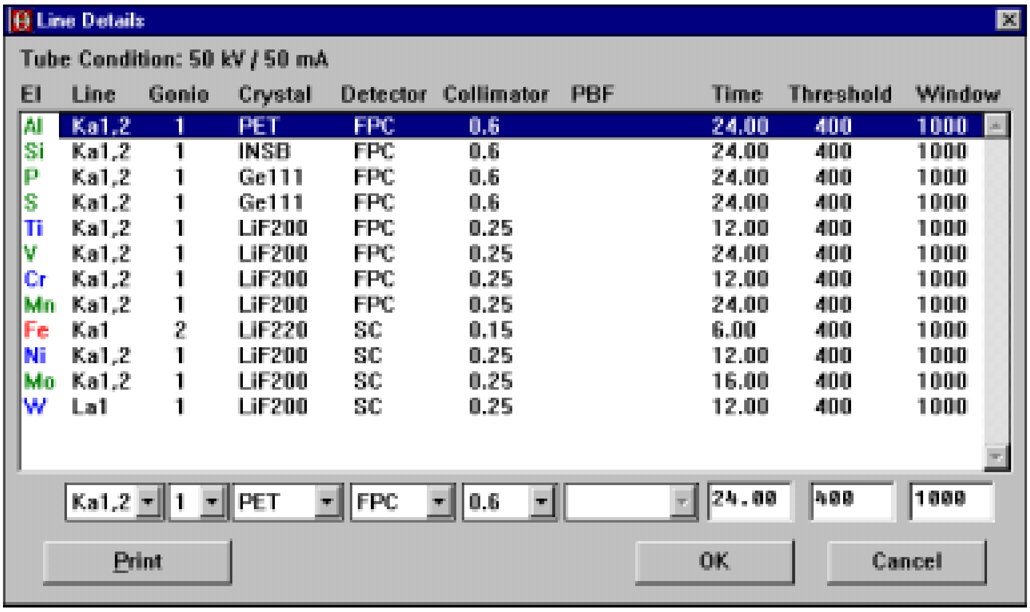
Results: VKA (W on VKa) Gonio Line: V Ka1,2
Count Rate: 46.8765 K.cps Detector Resolution: 20.84 %

1887.2571 mV
1.8904 K.cps
Window: 702
Threshold: 600

Figure 6.31

Line Details

The table displayed, when this function is selected, shows the analytical and instrumental parameters that are proposed by the Analytical Assistant.



Tube Condition: 50 kV / 50 mA

El	Line	Gonio	Crystal	Detector	Collimator	PBF	Time	Threshold	Window
Al	Ka1,2	1	PET	FPC	0.6		24.00	400	1000
Si	Ka1,2	1	INSB	FPC	0.6		24.00	400	1000
P	Ka1,2	1	Ge111	FPC	0.6		24.00	400	1000
S	Ka1,2	1	Ge111	FPC	0.6		24.00	400	1000
Ti	Ka1,2	1	LiF200	FPC	0.25		12.00	400	1000
V	Ka1,2	1	LiF200	FPC	0.25		24.00	400	1000
Cr	Ka1,2	1	LiF200	FPC	0.25		12.00	400	1000
Mn	Ka1,2	1	LiF200	FPC	0.25		24.00	400	1000
Fe	Ka1	2	LiF220	SC	0.15		6.00	400	1000
Ni	Ka1,2	1	LiF200	SC	0.25		12.00	400	1000
Mo	Ka1,2	1	LiF200	SC	0.25		16.00	400	1000
W	La1	1	LiF200	SC	0.25		12.00	400	1000

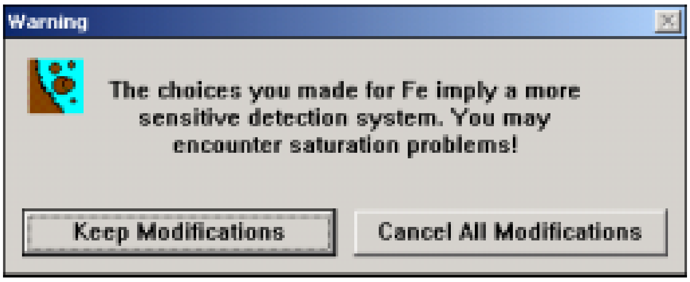
Ka1,2	1	PET	FPC	0.6		24.00	400	1000

Figure 6.32

After the overlap investigations have been completed, it may be necessary to adjust the PHD settings and to choose different instrumental parameters, like a finer collimator.

Changes in this table are performed in the following way:
- Highlight the line where parameters need to be changed.
- Enter new values in the corresponding entry boxes, or select new item from the combo boxes.
- Click onto any other line to make the changes.
- Repeat for any other lines.

Note: If, for a line that is prone to saturation and thus appropriate parameters were chosen by the system to lower the sensitivity, a more sensitive detection device (i.e. collimator, crystal, detector) is chosen, then the following warning message is given.



The choices you made for Fe imply a more sensitive detection system. You may encounter saturation problems!

Figure 6.33

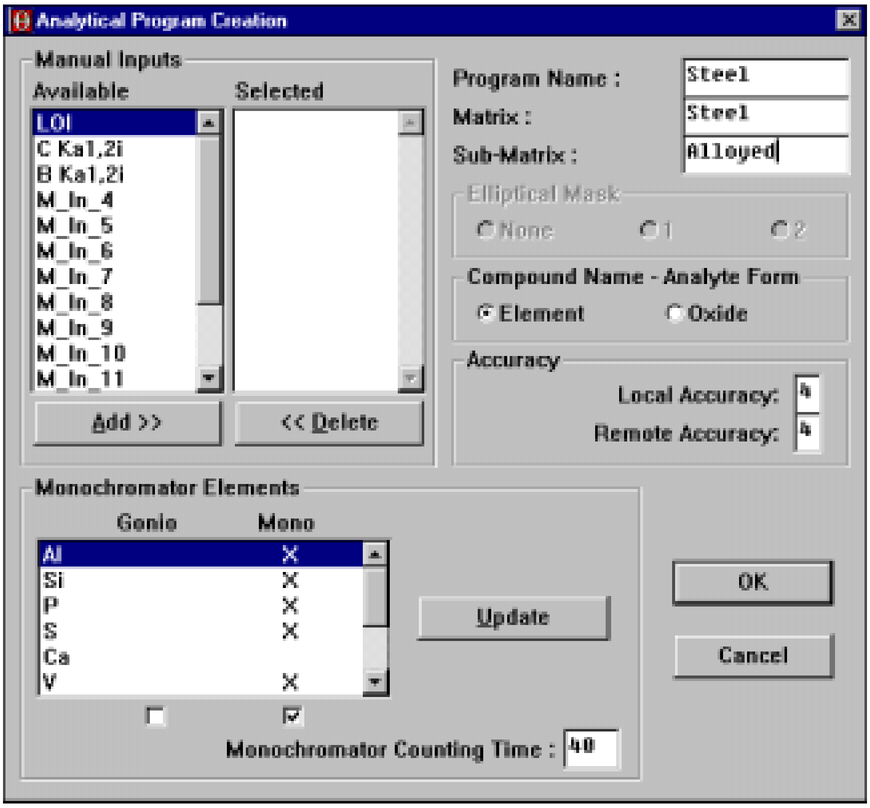
The table can be **Printed** with the corresponding function, and if the dialog box is left with **OK**, the changes are made permanent, or lost if the dialog box is left with **Cancel**.

The effect of these changes could be investigated by performing new scans over the lines with changed parameters. If the improvement is not satisfactory, then an overlap correction has to be made.

Note: If the **Matrix Details** or the **Elements to Measure** function is selected again, all parameters are selected according to the criteria selection schema. This means that all **Line Details** changes will be cancelled.

Create Program

The final step is the creation of the Analytical Program and the template calibration sample. When this function is selected, the dialog box shown below is displayed.



Available: LOI, C Ka1,2i, B Ka1,2i, M_In_4, M_In_5, M_In_6, M_In_7, M_In_8, M_In_9, M_In_10, M_In_11

Program Name : Steel

Matrix : Steel

Sub-Matrix : Alloyed

Compound Name - Analyte Form: ● Element ○ Oxide

Local Accuracy: 4

Remote Accuracy: 4

Genio	Mono
Al	X
Si	X
P	X
S	X
Ca	
V	X
☐	☑

Monochromator Counting Time : 40

Figure 6.34

The following parameters can or have to be specified:

Manual Inputs
The Manual Inputs to be included in the Analytical Program can be selected with the **Add>>** button, or removed again with the **<<Delete** button.

Program Name
The name of the Analytical Program has to be specified here.

Matrix The name of the matrix has to be specified in this box.

Sub-Matrix The name of the sub-matrix has to be specified in this box.

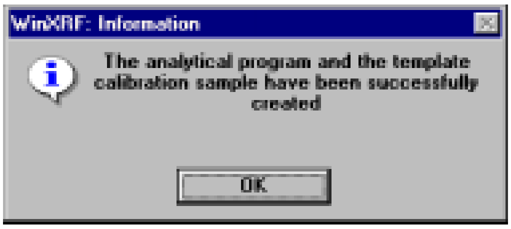
Elliptical Mask
This option is not available for the ARL 9800 instrument.

Compound Name - Analyte Form
This option allows you to specify in which form the analysed elements should be reported. Two possibilities exist:

Element	The analytes are reported by their **Chemical Symbol**.
Oxide	The analytes are reported in their **Typical Oxide Form**.

Monochromator Elements (this only applies to the ARL 9800 series)
Selected elements which can also be analysed with an installed monochromator are proposed to be analysed with these monochromators. However, the user has the possibility to instead choose either the goniometer or both. Furthermore, the **Monochromator Counting Time** can be specified.

Creation of the missing lines in the line library, the Analytical Program and the template calibration sample is achieved by confirming the above entries with the **OK** button, causing the system to issue the message shown in the figure below. On the other hand, the dialog box can be left without any creation with the **Cancel** button.

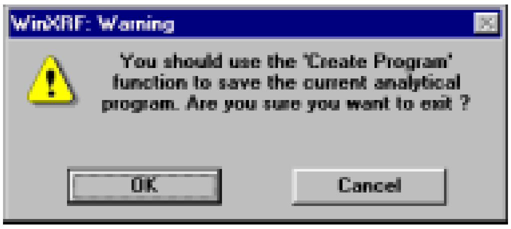


The analytical program and the template calibration sample have been successfully created

Figure 6.35

Exit

When this button is pressed, the **Analytical Program** function is left. If no program was created, the following warning message is given.



You should use the 'Create Program' function to save the current analytical program. Are you sure you want to exit ?

Figure 6.36

7

INSTRUMENT CALIBRATION

7 INSTRUMENT CALIBRATION

Introduction

An analytical program has to be created when the instrument has not yet been calibrated, or when an extension to the existing calibrations is needed to support other analytical applications. The X-ray analyser is a comparative measuring system. To display the concentrations of unknown samples, the system must have been calibrated previously. To do so, a set of certified standard samples is necessary. These samples are measured and the intensities of each element is stored in a calibration result file. The relationship between the concentrations and the instrumental response for each element and sample is then calculated by the computer system and stored, as a polynomial, in the corresponding analytical program. The instrument is now ready to measure unknown samples, matching with the matrix of the calibration standards. In X-ray fluorescence analysis some physical and instrumental errors will appear:

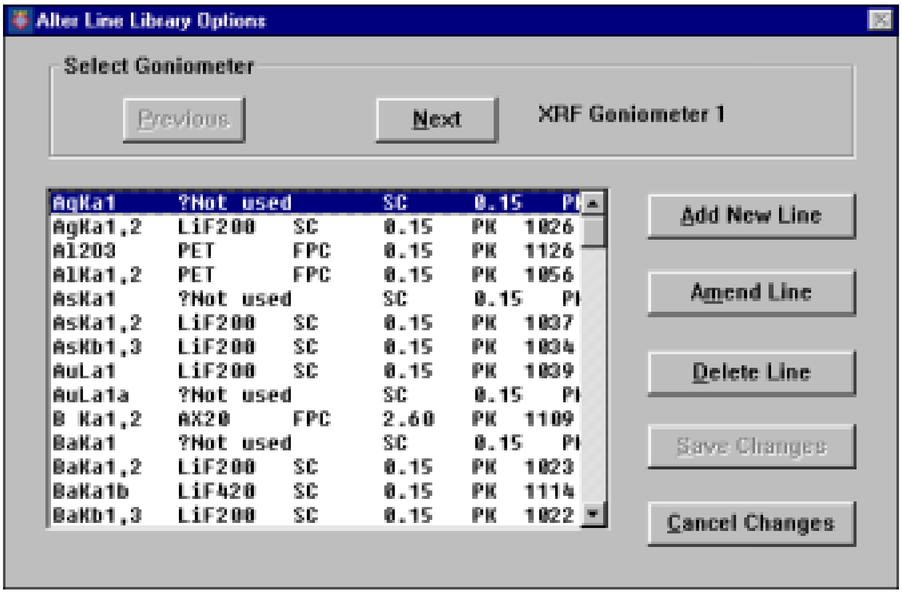
- Physical errors are matrix effects due to absorption and enhancement. To correct these errors the computer system will use mathematical models.

- Instrumental errors are mainly line overlaps due to insufficient resolution, and instrumental response drift through the time. Line overlaps can be corrected mathematically whereas to correct the instrumental drift, the system response has to be re-adjusted periodically by measuring appropriate reference samples, known as **Setting-up Samples**.

With the help of the **Analytical Assistant** all essential parameters and tables that are necessary in the calibration process have been prepared. Therefore, the basic calibration set-up simply consists of completing the data and, if necessary, modifying the default parameters, as explained briefly hereafter.

Line Library:

All element lines that did not exist with the required parameters in the Line Library have been created automatically. These lines can be recognised by their capital suffix A, B, etc., for instance, **AlKa1,2A** or **FeKa1B**. In principle there is no need to modify any of the parameters in the line library.



XRF Goniometer 1

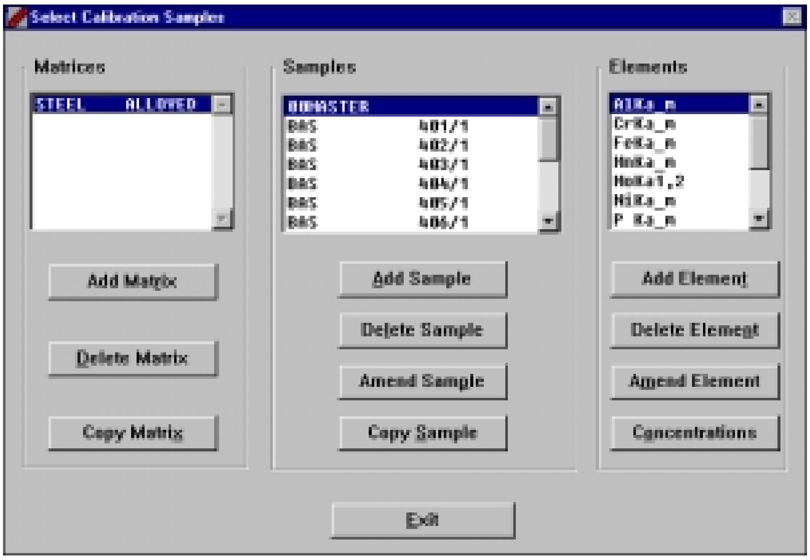
```
AgKa1      ?Not used       SC        0.15    P
AgKa1,2    LiF200   SC     0.15      PK   1026
Al203      PET      FPC    0.15      PK   1126
AlKa1,2    PET      FPC    0.15      PK   1056
AsKa1      ?Not used       SC        0.15    P
AsKa1,2    LiF200   SC     0.15      PK   1037
AsKb1,3    LiF200   SC     0.15      PK   1034
AuLa1      LiF200   SC     0.15      PK   1039
AuLa1a     ?Not used       SC        0.15    P
B Ka1,2    AX20     FPC    2.60      PK   1109
BaKa1      ?Not used       SC        0.15    P
BaKa1,2    LiF200   SC     0.15      PK   1023
BaKa1b     LiF420   SC     0.15      PK   1114
BaKb1,3    LiF200   SC     0.15      PK   1022
```

Figure 7.1

Exit

Calibration Samples:

The template calibration sample has the following signature: **00MASTER**. In order to build the calibration sample set, this template sample can be copied as many times as needed and the concentrations can then be input (see Section *Complete the list of Calibration Standard Samples*).


Select Calibration Samples

Matrices
STEEL ALLOYED

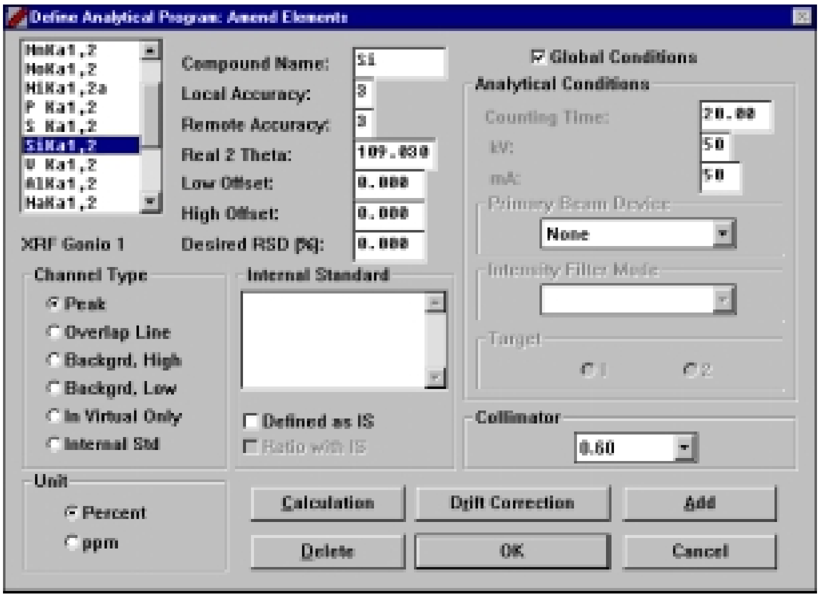
Samples
00MASTER
BAS 401/1
BAS 402/1
BAS 403/1
BAS 404/1
BAS 405/1
BAS 406/1

Elements
AlKa_m
CrKa_m
FeKa_m
MnKa_m
MoKa1,2
NiKa_m
P Ka_m

Figure 7.2

Analytical Program:

In our example, the Analytical Program that was created is shown in the following figure.


Define Analytical Program: Amend Elements

MoKa1,2
MoKa1,2
NiKa1,2a
P Ka1,2
S Ka1,2
SiKa1,2
V Ka1,2
AlKa1,2
NaKa1,2

Compound Name: Si
Local Accuracy: 3
Remote Accuracy: 3
Real 2 Theta: 109.030
Low Offset: 0.000
High Offset: 0.000
Desired RSD [%]: 0.000

XRF Gonio 1

☑ Global Conditions

Analytical Conditions
Counting Time: 20.00
kV: 50
mA: 50

Primary Beam Device: None

Intensity Filter Mode

Target ○ 1 ○ 2

Collimator 0.60

Channel Type
● Peak
○ Overlap Line
○ Backgrd, High
○ Backgrd, Low
○ In Virtual Only
○ Internal Std

Internal Standard
☐ Defined as IS
☐ Ratio with IS

Unit
● Percent
○ ppm

Figure 7.3

Also here, there is in principle no need to modify any of the proposed parameters, but it is certainly necessary to complete the set-up of the Analytical Program.

Note: The Analytical Assistant does not take care of **Setting-up Samples** that are necessary to compensate the instrumental drift so that the calibration can be used over a long time.

Exit

What remains to be done

The next steps of the calibration process are described hereafter, namely:

- Complete the list of Calibration Standard Samples
- Define Setting-up Samples
- Include the Setting-up Samples in the Analytical Program
- Set-up Analytical Tasks
- Drift Correction Initialisation
- Measurement of the Calibration Samples
- Calculation of the Calibration Curves
- Drift Correction Update

Complete the list of Calibration Standard Samples

- Select **Preparation** in the main menu.
- Then select **Analysis** followed by **Calibration Samples**.

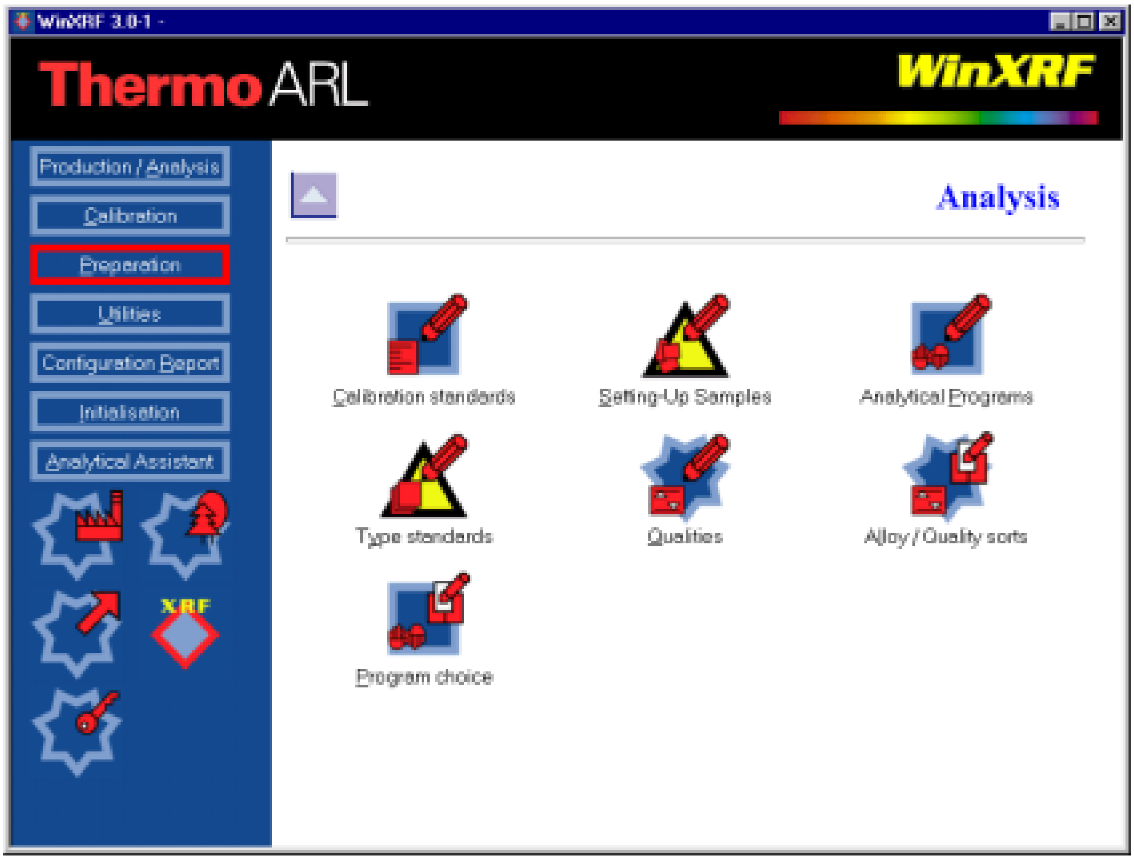
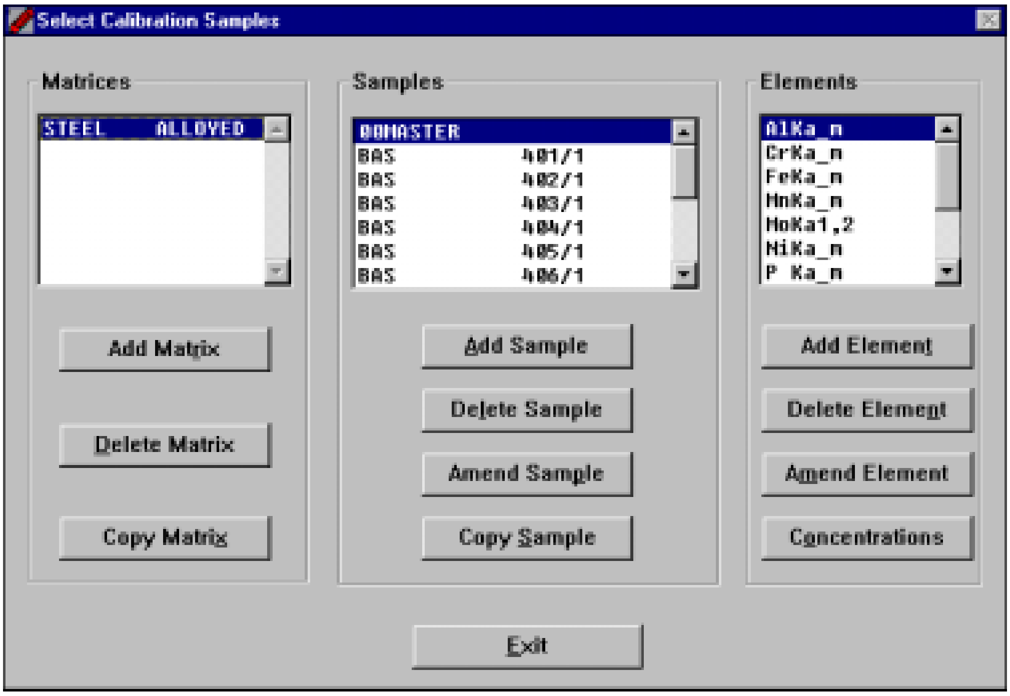


Figure 7.4

To add other calibration samples the following should be done:

- Click on **Copy Sample**.



Matrices:
STEEL ALLOYED

Samples:
00MASTER
BAS 401/1
BAS 402/1
BAS 403/1
BAS 404/1
BAS 405/1
BAS 406/1

Elements:
AlKa_n
CrKa_n
FeKa_n
MnKa_n
MoKa1,2
NiKa_n
P Ka_n

Figure 7.5

- In the **Destination Sample** area specify the **Name** and the **Number** of the new sample in the corresponding boxes,
- and then click on **Confirm**.

Source Sample
Matrix: STEEL
Sub-group: ALLOYED
Name: BAS
Number (or *): 401/1

Destination Sample
Matrix: STEEL
Sub-group: ALLOYED
Name: BAS_TEST
Number (or *): 401/1

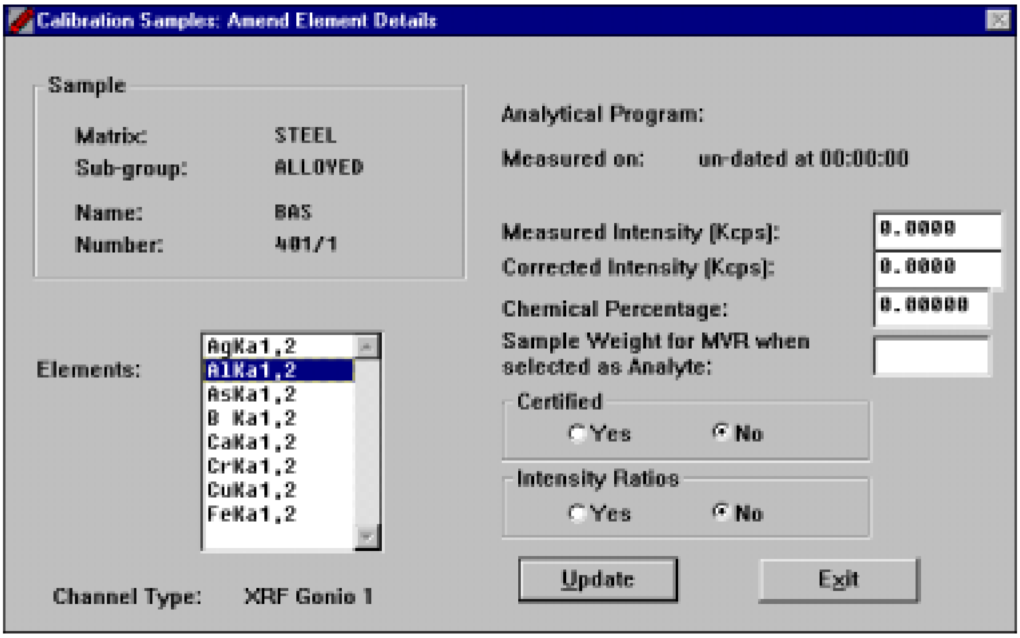
Figure 7.6

Repeat this operation until all samples are added in the matrix.

The chemical percentage for each element and sample can be entered by returning to the **Select Calibration Samples** dialog box (see Figure 7.5), selecting the sample to be updated in the list box, and clicking on **Concentrations**.

- Select the element to be updated in the list box.

- Enter the **Chemical Percentage** in the corresponding box.
- Type **Enter** or click on **Update** to store the value.
- Do the same for the remaining elements.
- Return to the screen above by clicking on **Exit**.


Calibration Samples: Amend Element Details

Sample
Matrix: STEEL
Sub-group: ALLOYED
Name: BAS
Number: 401/1

Analytical Program:
Measured on: un-dated at 00:00:00

Measured Intensity [Kcps]: 0.0000
Corrected Intensity [Kcps]: 0.0000
Chemical Percentage: 0.00000
Sample Weight for MVR when selected as Analyte:

Elements:
AgKa1,2
AlKa1,2
AsKa1,2
B Ka1,2
CaKa1,2
CrKa1,2
CuKa1,2
FeKa1,2

Certified ○ Yes ● No
Intensity Ratios ○ Yes ● No

Channel Type: XRF Gonio 1

Figure 7.7

Go on with the same method for all samples and elements.

The **Calibration Sample Matrix** is ready.

Define Setting-up Samples

- Select **Preparation** in the main menu.
- Then select **Analysis** followed by **Setting-up Samples**.

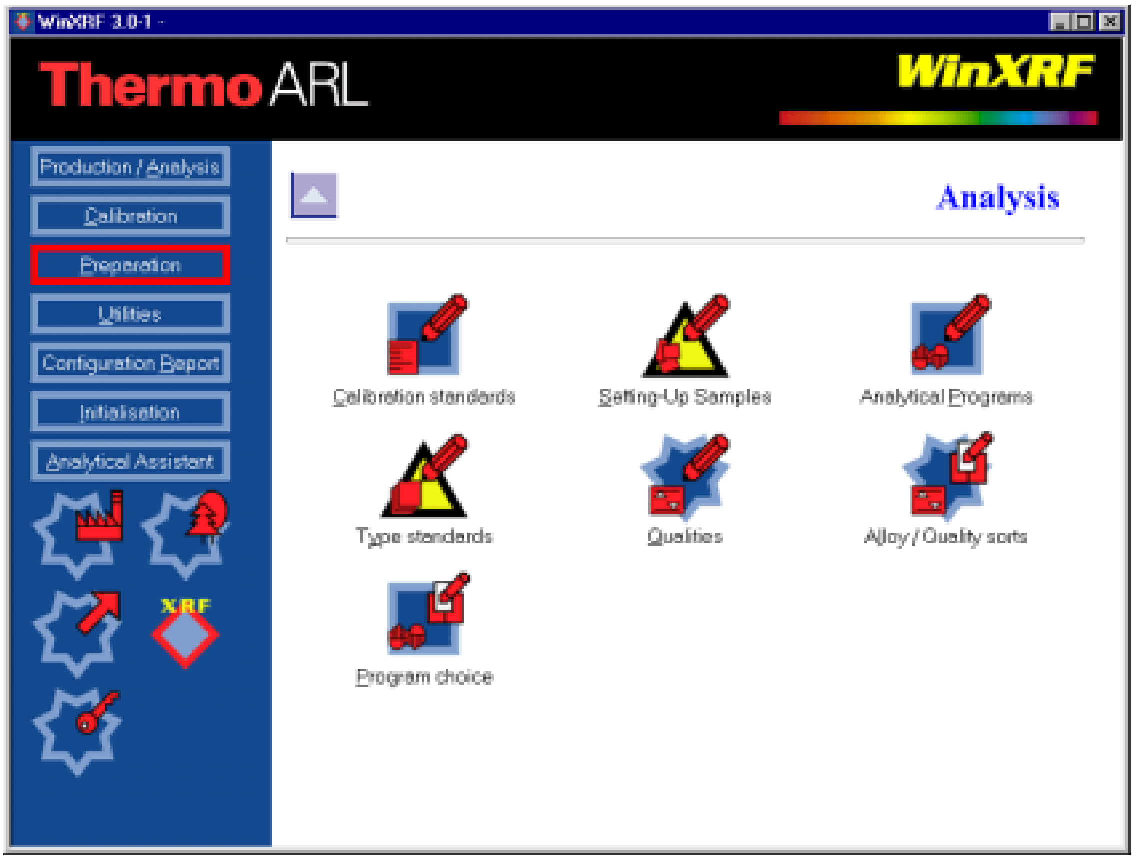
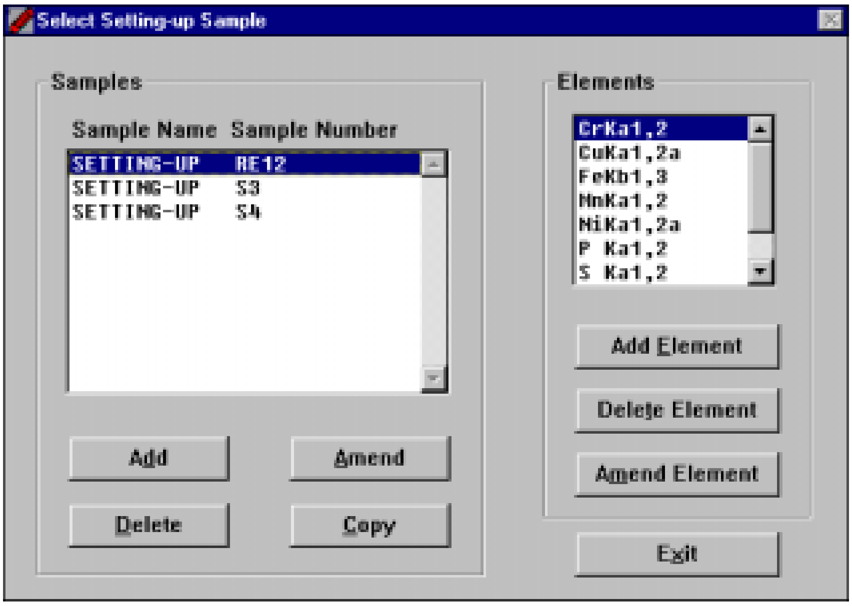


Figure 7.8

- Click on **Add**.



Sample Name	Sample Number
SETTING-UP	RE12
SETTING-UP	S3
SETTING-UP	S4

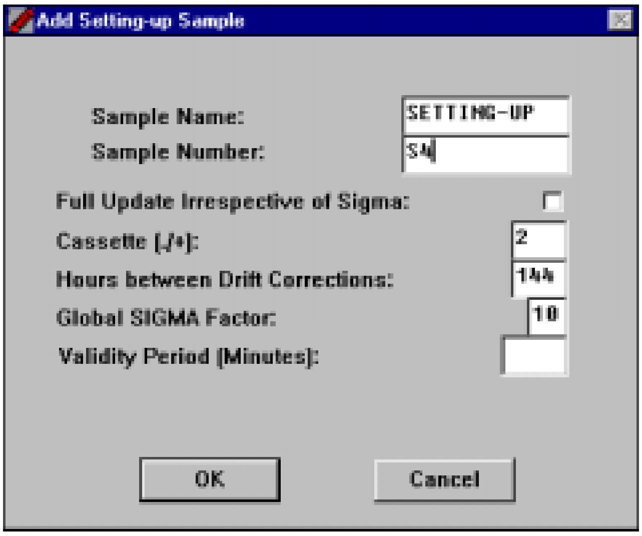
CrKa1,2
CuKa1,2a
FeKb1,3
MnKa1,2
NiKa1,2a
P Ka1,2
S Ka1,2

Figure 7.9

Specify in the corresponding boxes:

- the Sample Name
- the Sample Number
- and the Cassette Number.

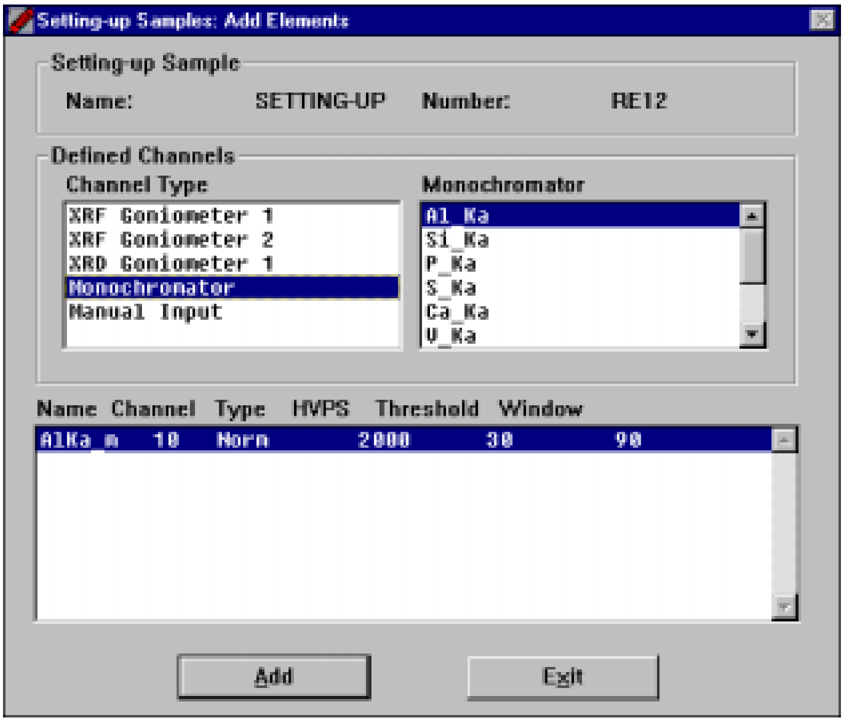
In the **Hours between Drift Correction** box enter the delay in hours and, if necessary, the value for the **Global SIGMA Factor** can be changed.



Sample Name: SETTING-UP
Sample Number: S4
Full Update Irrespective of Sigma: ☐
Cassette (-/+): 2
Hours between Drift Corrections: 144
Global SIGMA Factor: 10
Validity Period (Minutes):

Figure 7.10

- Click on **OK**.
- Click on **Add Element** (see Figure 7.9).
- Click on **Goniometer 1** button.
- Highlight the element to be selected and then click on **Add**.



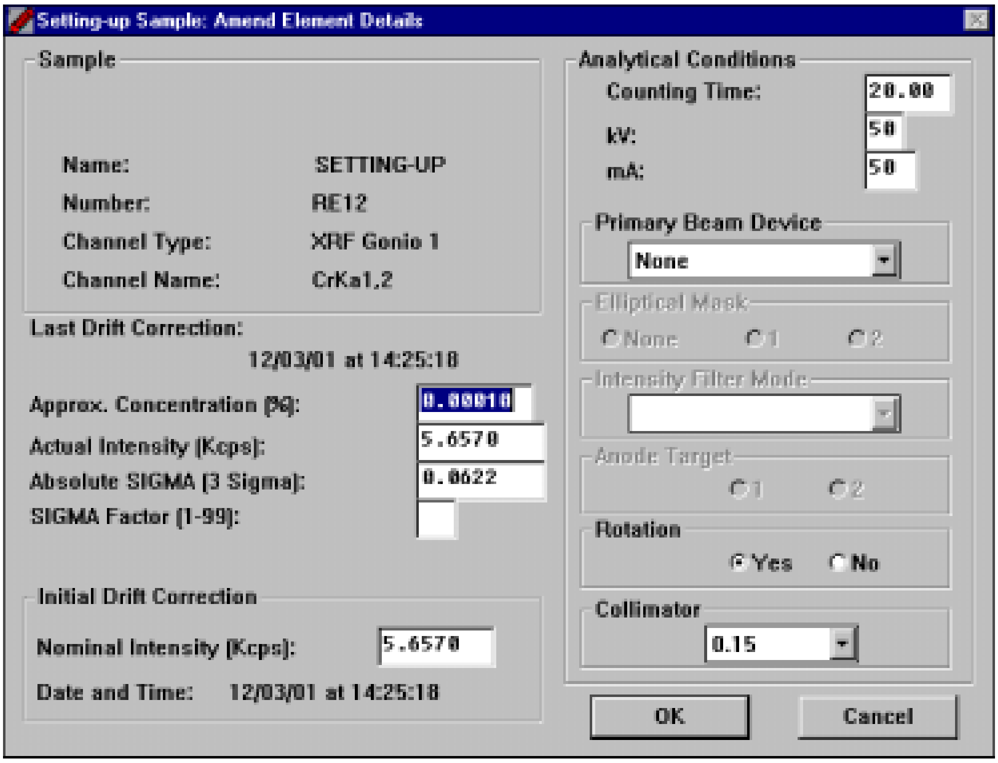
Name: SETTING-UP Number: RE12

Channel Type	Monochromator
XRF Goniometer 1	Al_Ka
XRF Goniometer 2	Si_Ka
XRD Goniometer 1	P_Ka
Monochromator	S_Ka
Manual Input	Ca_Ka
	V_Ka

Name	Channel	Type	HVPS	Threshold	Window
AlKa_m	10	Norm	2000	30	90

Figure 7.11

Specify the **Counting Time** in seconds, and the **kV** and **mA** values.



Setting-up Sample: Amend Element Details

Sample

Name: SETTING-UP
Number: RE12
Channel Type: XRF Gonio 1
Channel Name: CrKa1,2

Last Drift Correction: 12/03/01 at 14:25:18

Approx. Concentration (%): 0.00010
Actual Intensity (Kcps): 5.6570
Absolute SIGMA (3 Sigma): 0.0622
SIGMA Factor (1-99):

Initial Drift Correction
Nominal Intensity (Kcps): 5.6570
Date and Time: 12/03/01 at 14:25:18

Analytical Conditions
Counting Time: 20.00
kV: 50
mA: 50

Primary Beam Device: None

Elliptical Mask: ○ None ○ 1 ○ 2

Intensity Filter Mode:

Anode Target: ○ 1 ○ 2

Rotation: ● Yes ○ No

Collimator: 0.15

Figure 7.12

As a later help during the selection of the setting-up sample enter an **Approximate Concentration** in the corresponding box.

- Click on **OK**.

Repeat the same procedure for all other elements and click on **Exit** to return to the screen. Additional samples can then be defined in the same way. The Setting-up Sample is ready.

Include the Setting-up Samples in the Analytical Program

- Select **Preparation** in the main menu (see Figure 7.8).
- Then select **Analytical Programs**.

- In the following dialog box click on **Element Details**.

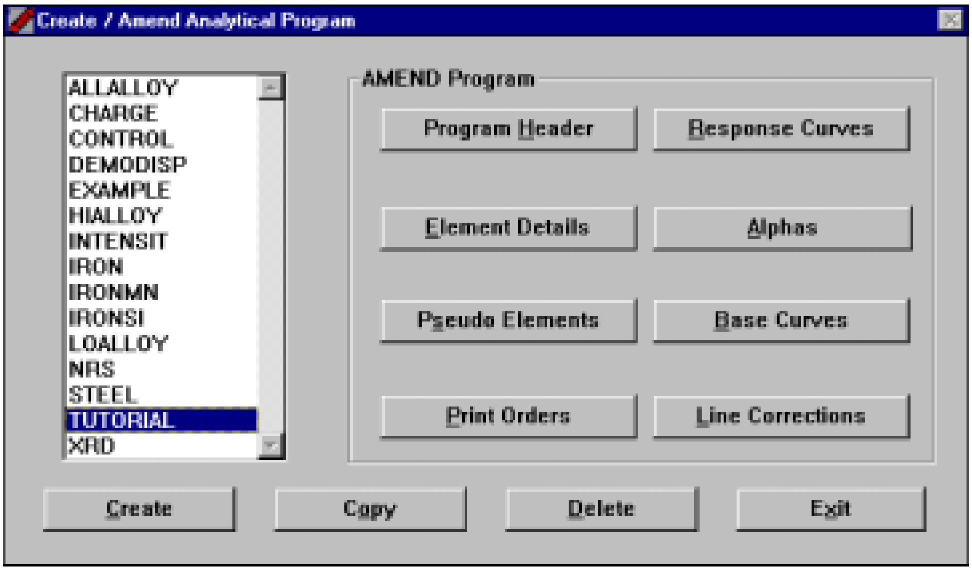
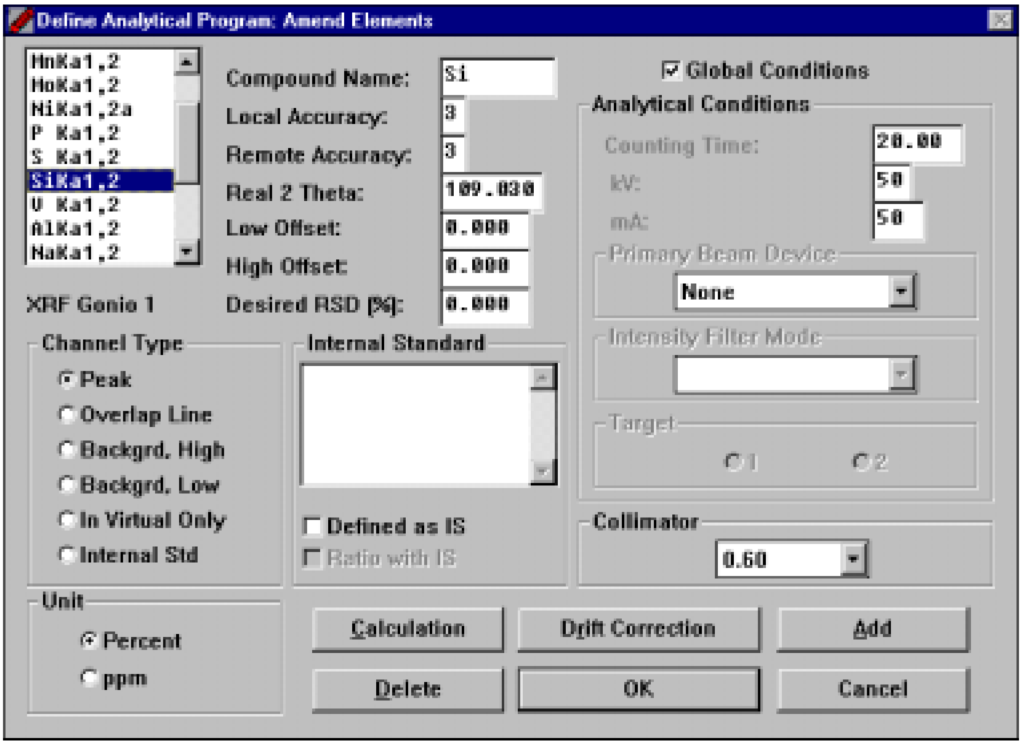


Figure 7.13

- Highlight the first element and click on **Drift Correction**.



Compound Name: Si
Local Accuracy: 3
Remote Accuracy: 3
Real 2 Theta: 109.030
Low Offset: 0.000
High Offset: 0.000
Desired RSD [%]: 0.000

☑ Global Conditions
Counting Time: 20.00
kV: 50
mA: 50
Primary Beam Device: None
Collimator: 0.60

● Peak
○ Overlap Line
○ Backgrd. High
○ Backgrd. Low
○ In Virtual Only
○ Internal Std

☐ Defined as IS
☐ Ratio with IS

● Percent
○ ppm

Figure 7.14

- Select the **Top** and **Bottom Standard** from the corresponding list boxes.

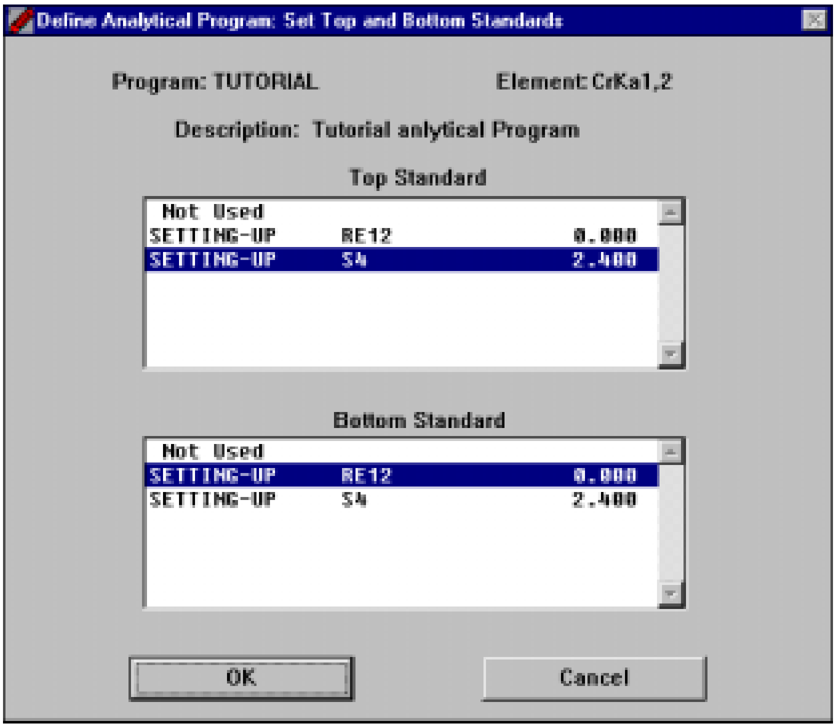


Figure 7.15

- Click on **OK**.
- Repeat the same procedure for all the remaining elements.

Drift Correction Initialisation

Prepare the Drift Correction Initialisation Task

- Click on **Preparation**, in the main menu.
- Click on **Tasks** and then on **Standardisation/Drift Correction Initialisation**.

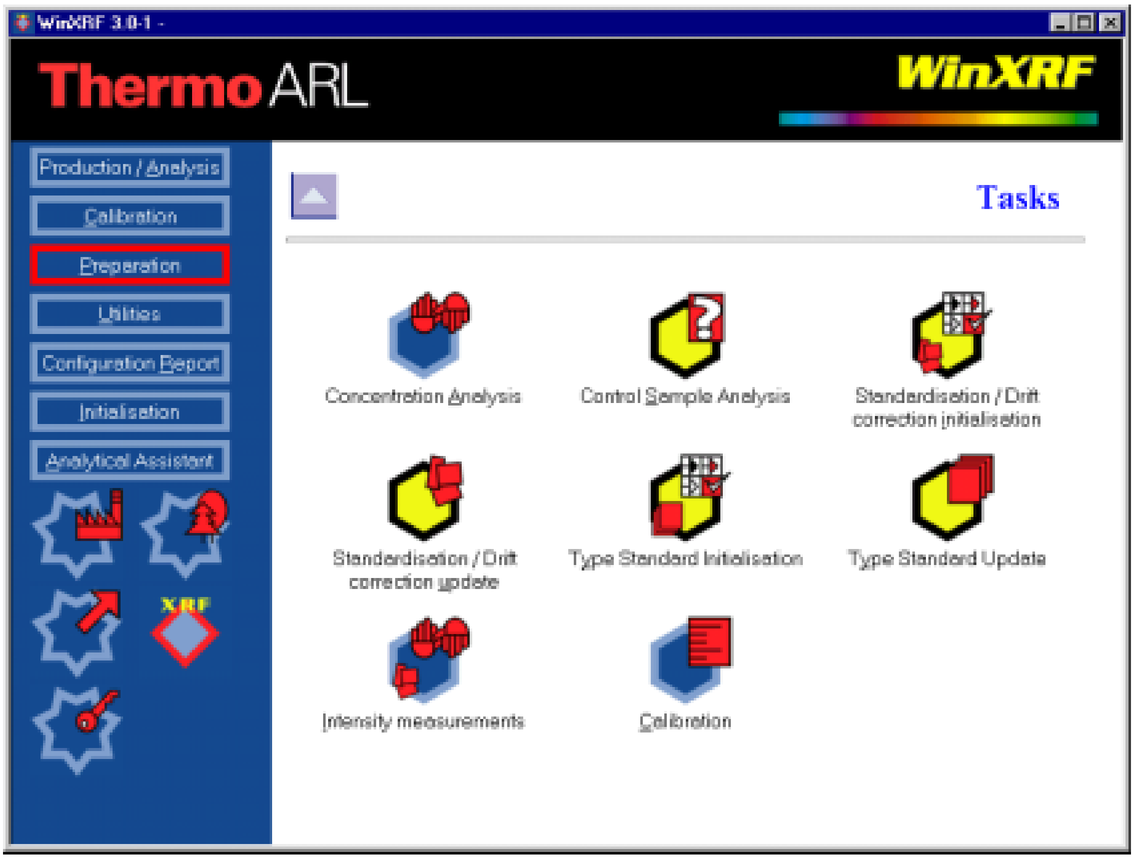
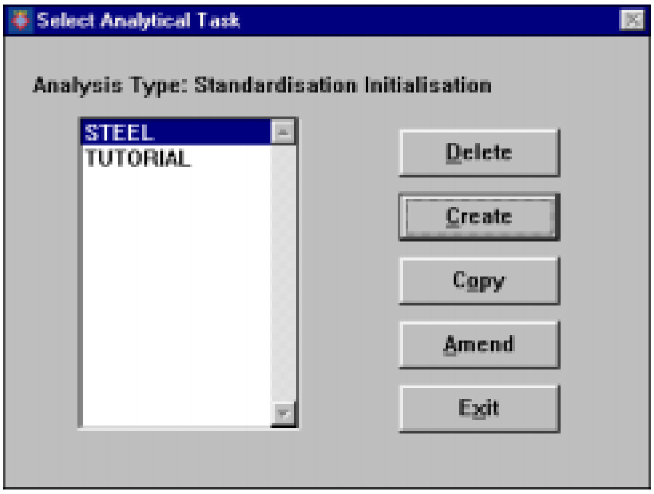


Figure 7.16

♦ Click on **Create**.



Analysis Type: Standardisation Initialisation

STEEL
TUTORIAL

Figure 7.17

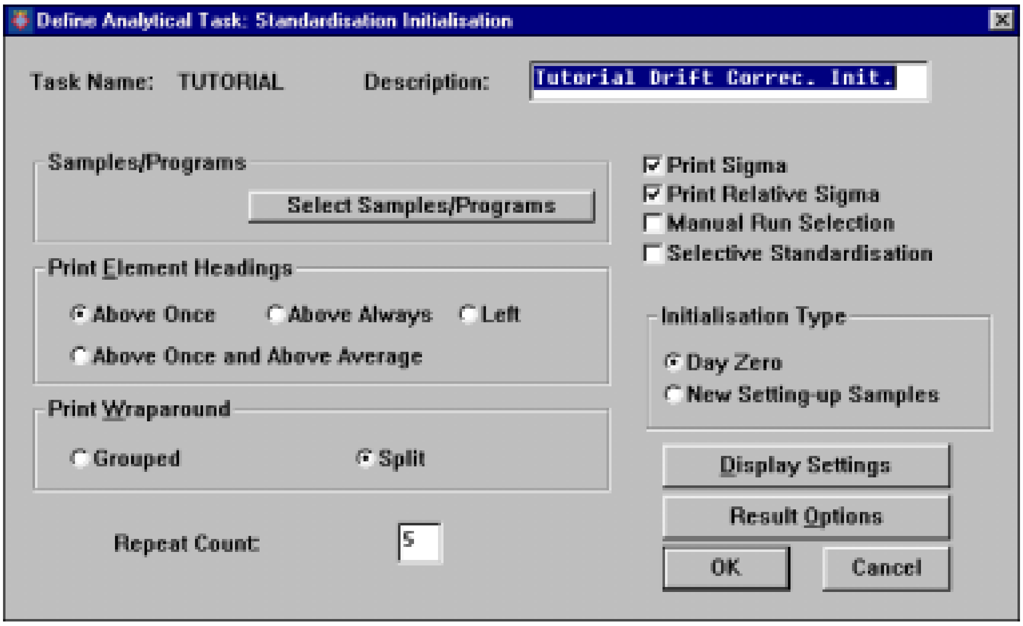
- In the **Task Name** box enter the desired task name.

Enter New Task Name: Test

Figure 7.18

- Click on **OK**.

- In the **Description** box enter the desired task description.
- To display **Sigma** and **Relative Sigma** select the corresponding options.
- As for the **Initialisation Type** select **Day Zero**.
- Then click on **Select Programs / Samples**.



Task Name: TUTORIAL

Description: Tutorial Drift Correc. Init.

☑ Print Sigma
☑ Print Relative Sigma
☐ Manual Run Selection
☐ Selective Standardisation

Print Element Headings
● Above Once ○ Above Always ○ Left
○ Above Once and Above Average

Initialisation Type
● Day Zero
○ New Setting-up Samples

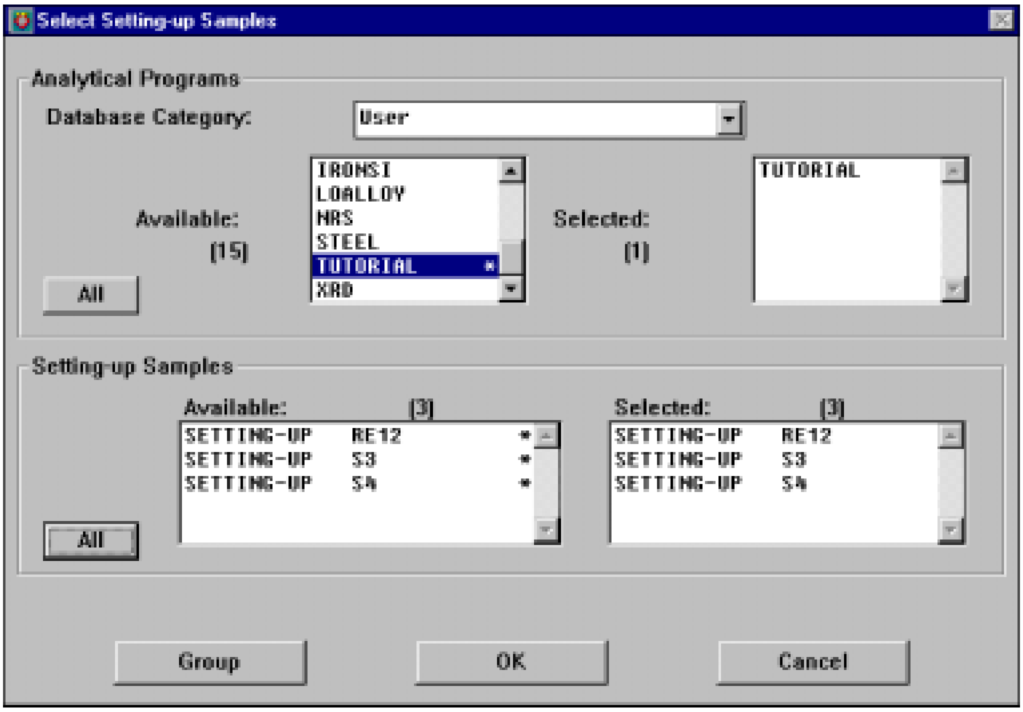
Print Wraparound
○ Grouped ● Split

Repeat Count: 5

Figure 7.19

- In the list box **Available Analytical Programs** double-click on the desired program, it will automatically be transferred to the **Selected** box. If the desired program is not listed, then select the appropriate **Database Category**.
- In the list box **Available Setting-up Samples** box double-click on the samples to be measured, they will automatically be transferred to the **Selected** box.
- Click on **OK**.

Exit



Database Category: User

Available: (15)
IRONSI
LOALLOY
NRS
STEEL
TUTORIAL *
XRD

Selected: (1)
TUTORIAL

Available: (3)
SETTING-UP RE12 *
SETTING-UP S3 *
SETTING-UP S4 *

Selected: (3)
SETTING-UP RE12
SETTING-UP S3
SETTING-UP S4

Figure 7.20

In next dialog boxes:

- Click on **OK**
- and **Exit**.

Measure the Setting-up Samples

Put the setting-up samples into the cassettes and place the cassettes onto the previously specified positions on the magazine.

- Click on **Calibration** in the main menu.
- Then click on **Standardisation/Drift Correction Initialisation**.

Exit

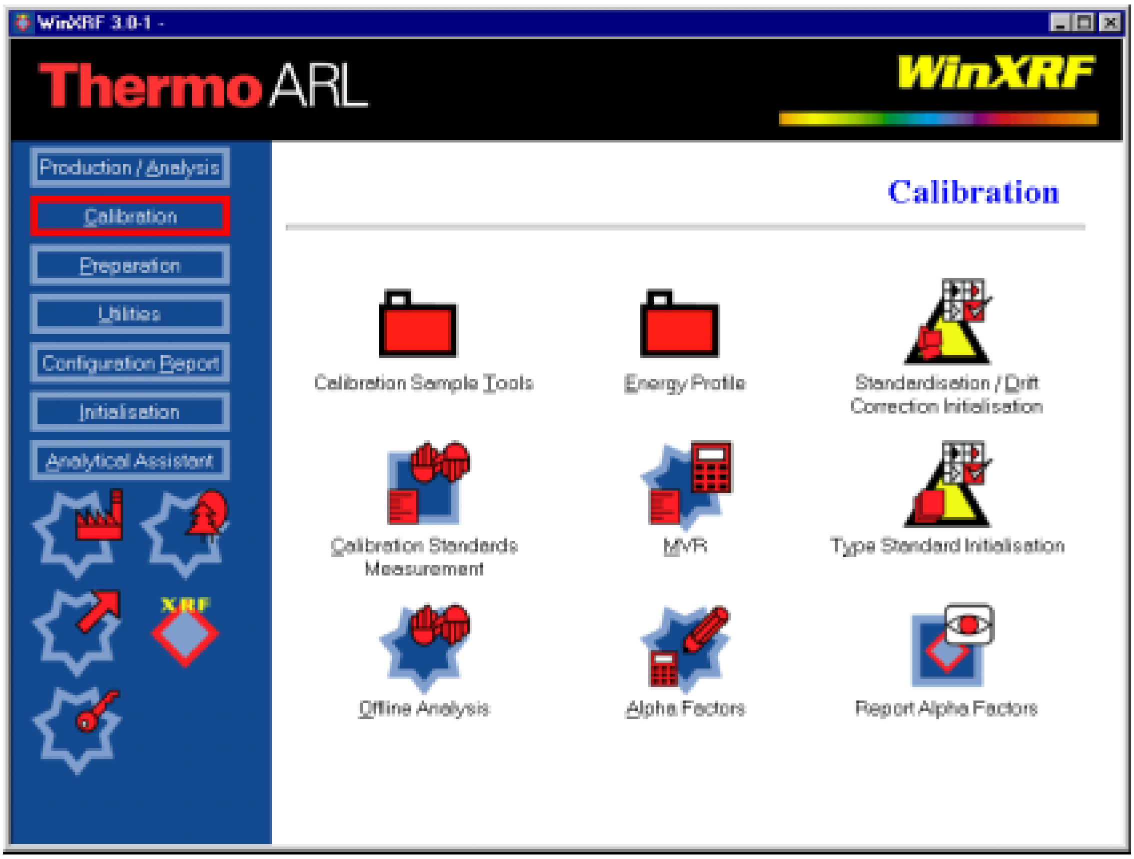
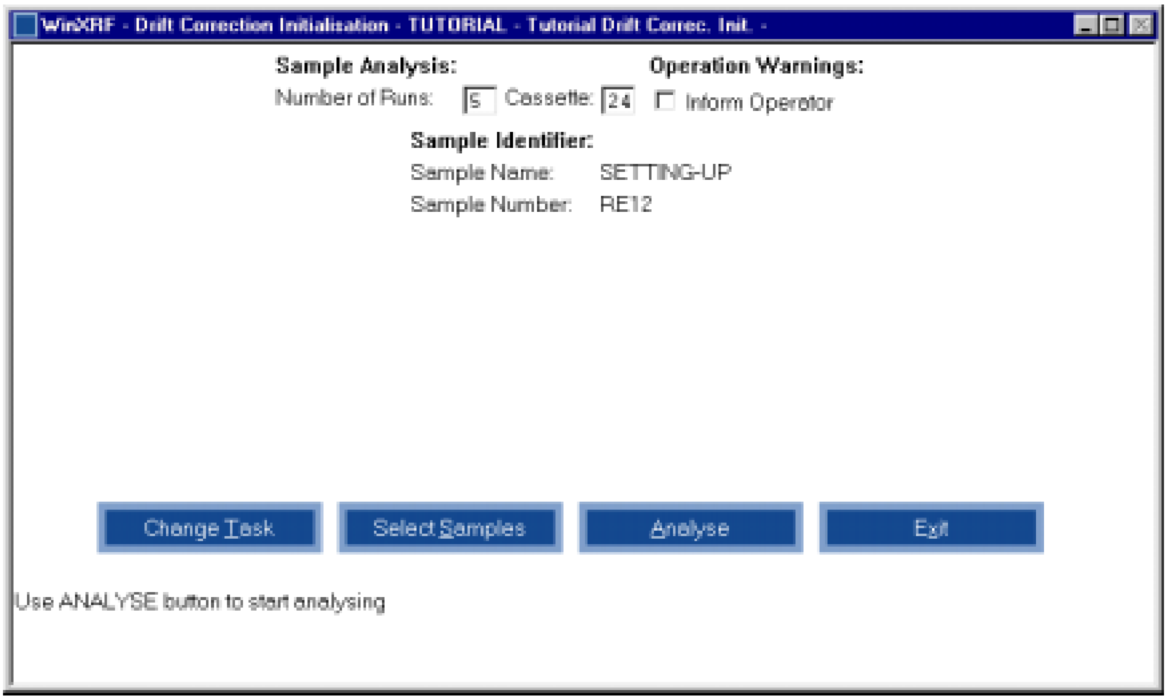


Figure 7.21

- ♦ Click on **Change Task**.



Sample Analysis:
Number of Runs: 5 Cassette: 24

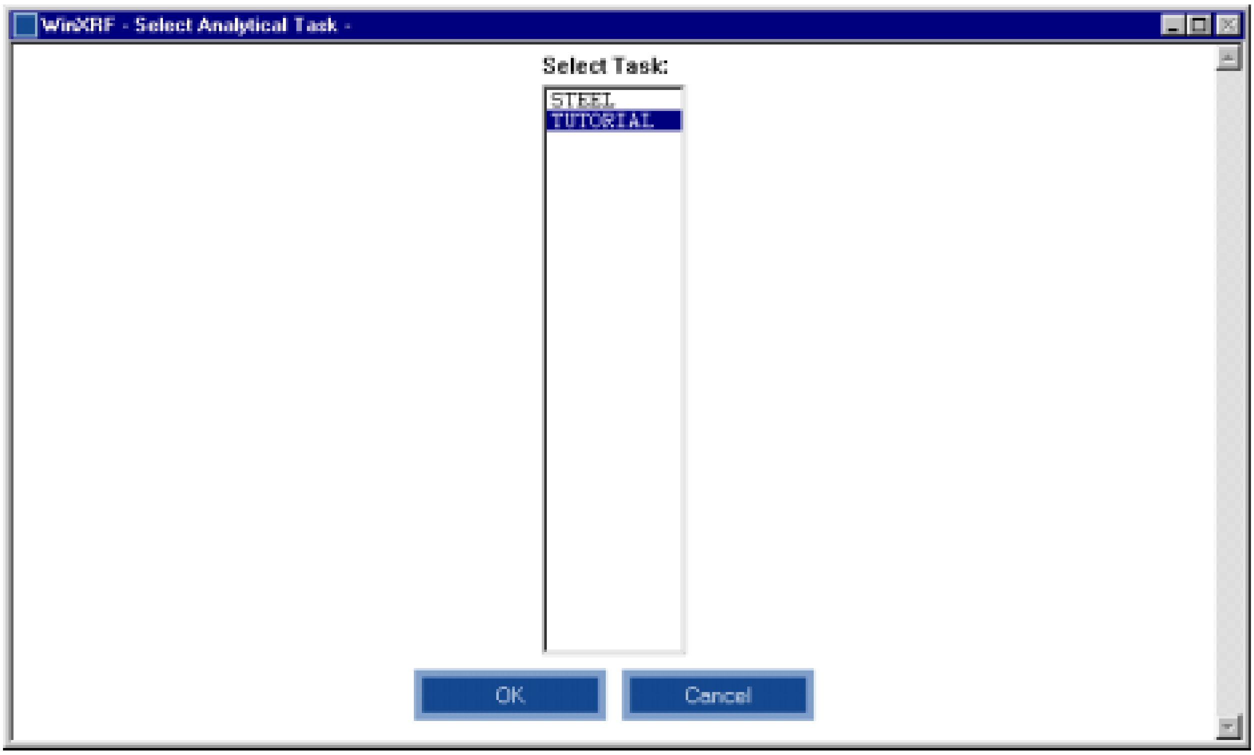
Operation Warnings:
☐ Inform Operator

Sample Identifier:
Sample Name: SETTING-UP
Sample Number: RE12

Use ANALYSE button to start analysing

Figure 7.22

- ♦ Select the appropriate task and then click on **OK**.



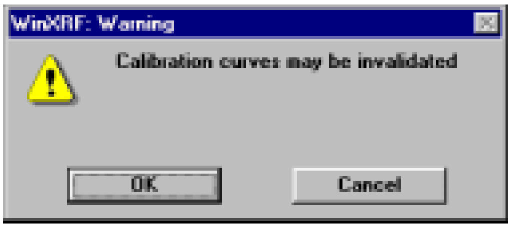
WinXRF - Select Analytical Task -

Select Task:

STEEL
TUTORIAL

Figure 7.23

- The analysis is invoked by clicking on **Analyse** (see Figure 7.22) which causes the warning message shown in the next figure to be displayed.



WinXRF: Warning

Calibration curves may be invalidated

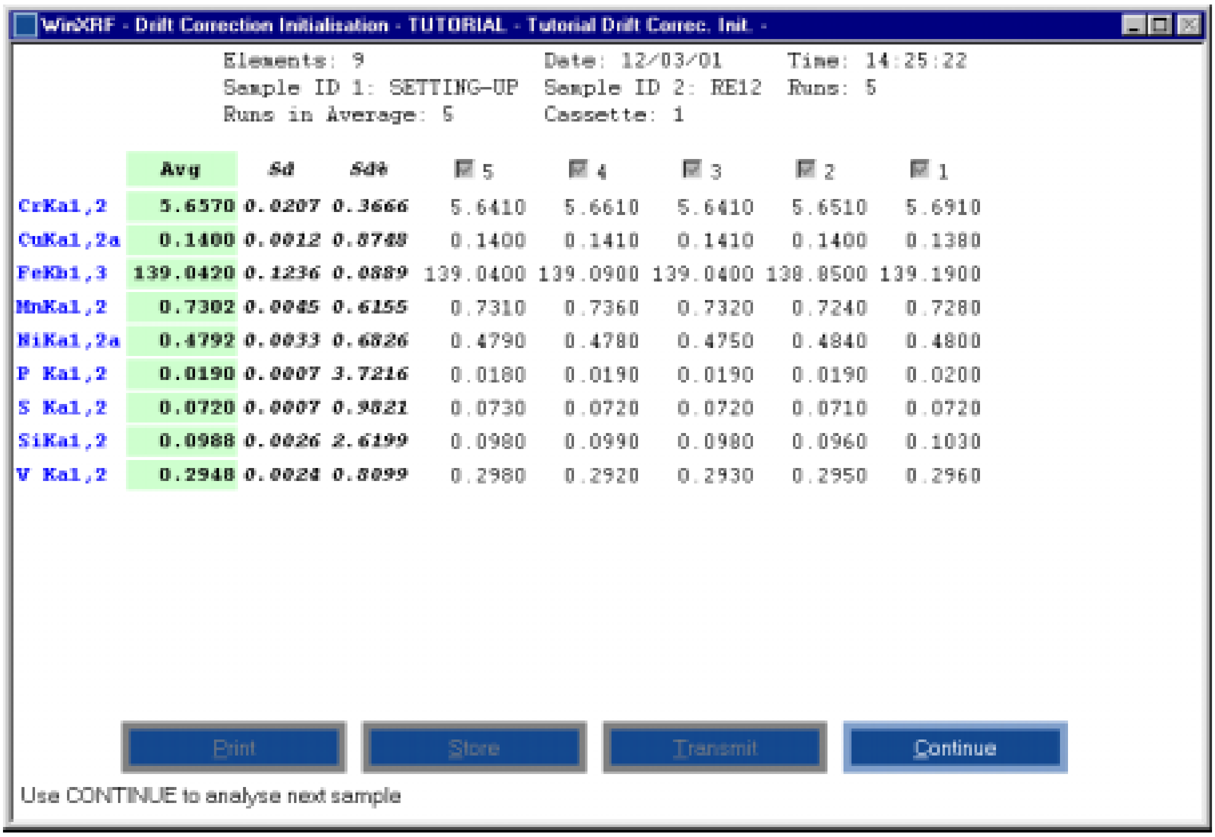
Figure 7.24

The setting-up samples will now be measured.

The intensities of the first sample will be displayed in the window shown next.

After the measurement is finished:

- Click on **Continue** and measure the next sample in the same way.



Elements: 9 Date: 12/03/01 Time: 14:25:22
Sample ID 1: SETTING-UP Sample ID 2: RE12 Runs: 5
Runs in Average: 5 Cassette: 1

	Avg	Sd	Sd%	☑ 5	☑ 4	☑ 3	☑ 2	☑ 1
CrKa1,2	5.6570	0.0207	0.3666	5.6410	5.6610	5.6410	5.6510	5.6910
CuKa1,2a	0.1400	0.0012	0.8748	0.1400	0.1410	0.1410	0.1400	0.1380
FeKb1,3	139.0420	0.1236	0.0889	139.0400	139.0900	139.0400	138.8500	139.1900
MnKa1,2	0.7302	0.0045	0.6155	0.7310	0.7360	0.7320	0.7240	0.7280
NiKa1,2a	0.4792	0.0033	0.6826	0.4790	0.4780	0.4750	0.4840	0.4800
P Ka1,2	0.0190	0.0007	3.7216	0.0180	0.0190	0.0190	0.0190	0.0200
S Ka1,2	0.0720	0.0007	0.9821	0.0730	0.0720	0.0720	0.0710	0.0720
SiKa1,2	0.0988	0.0026	2.6199	0.0980	0.0990	0.0980	0.0960	0.1030
V Ka1,2	0.2948	0.0024	0.8099	0.2980	0.2920	0.2930	0.2950	0.2960

Use CONTINUE to analyse next sample

Figure 7.25

The day zero intensities are automatically stored in the setting-up sample file.

Prepare the Calibration Task

- Click on **Preparation** in the main menu.
- Click on **Tasks** and then click on **Calibration**.

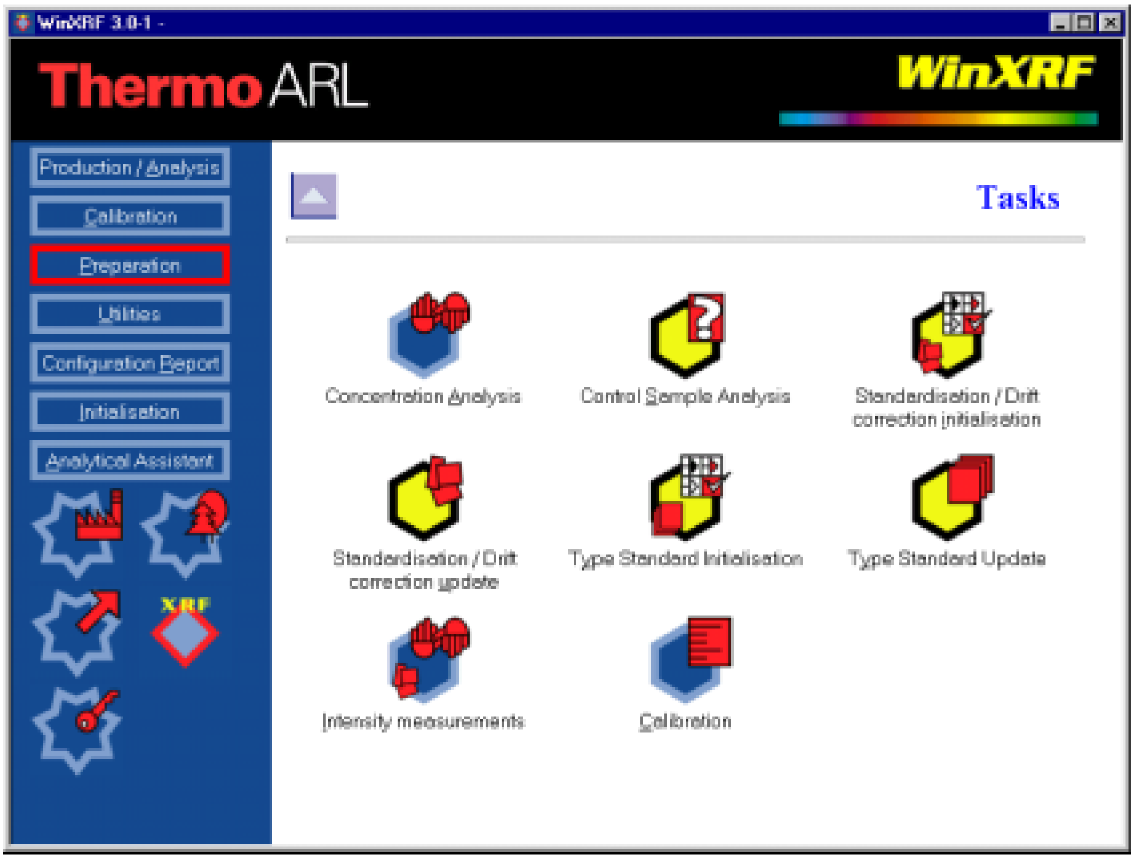
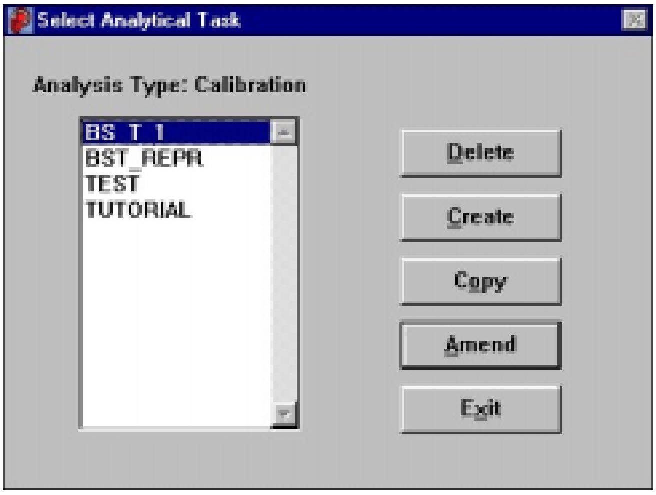


Figure 7.26

- Click on **Create** to create a new task.



Analysis Type: Calibration

BS_T_1
BST_REPR
TEST
TUTORIAL

Figure 7.27

- In the **Task Name** box enter the new task name.
- Click on **OK**.

Create Analytical Task

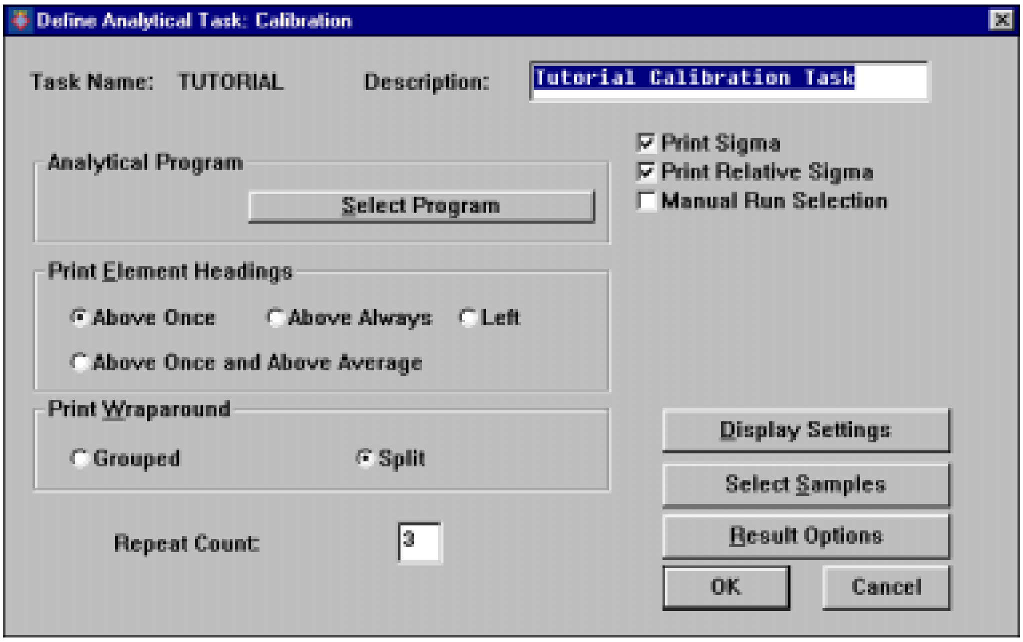
Enter New Task Name: Test

Figure 7.28

- In the **Description** box write the desired remark.
- Select the options to display the **Sigma** and **Relative Sigma**.
- Specify the number of runs in the **Repeat Count** box (suggested **3**).

The option **Manual Run Selection** should not be selected.

- Click on **Select Program** to select the desired program.



Define Analytical Task: Calibration

Task Name: TUTORIAL

Description: Tutorial Calibration Task

☑ Print Sigma
☑ Print Relative Sigma
☐ Manual Run Selection

Analytical Program

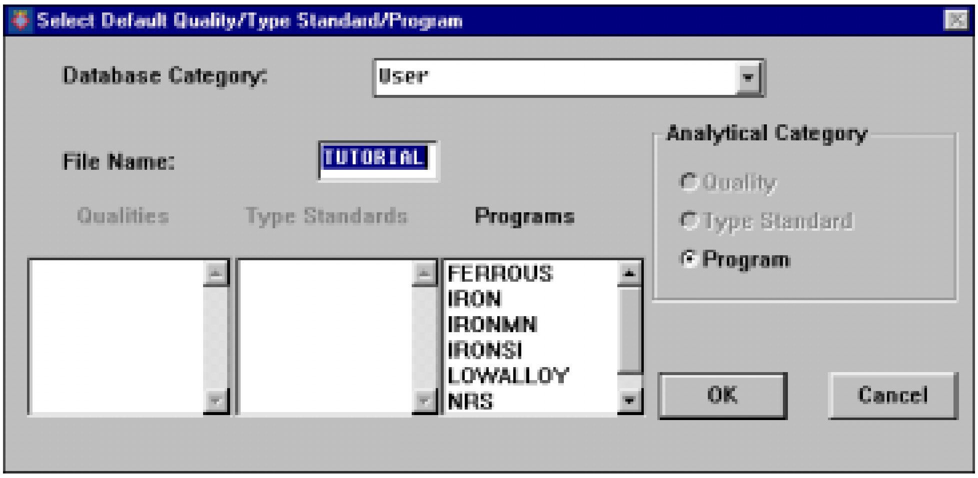
Print Element Headings
● Above Once ○ Above Always ○ Left
○ Above Once and Above Average

Print Wraparound
○ Grouped ● Split

Repeat Count: 3

Figure 7.29

- ♦ Select the desired program in the list box. If the program is not listed, then select the appropriate **Database Category**.
- ♦ Click on **OK**.



Database Category: User

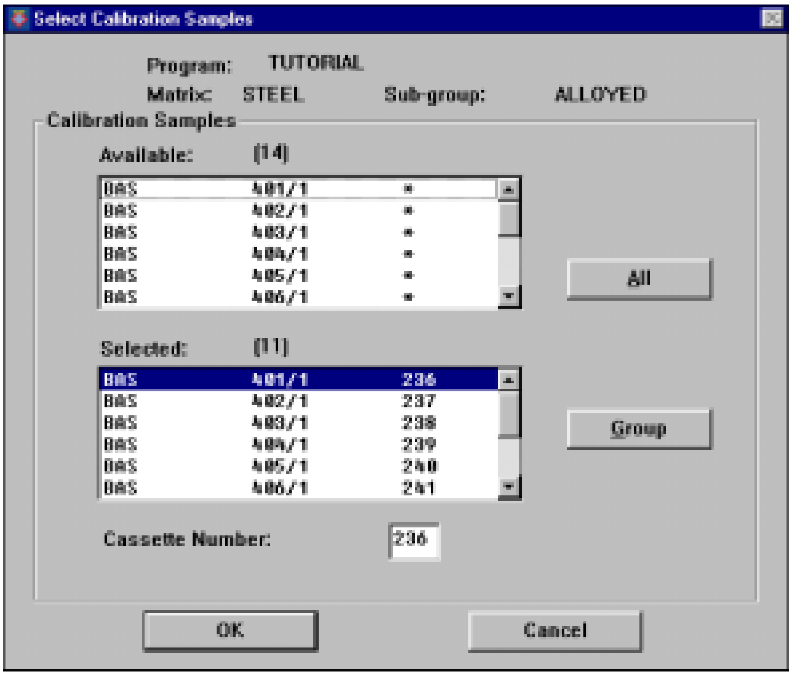
File Name: TUTORIAL

Analytical Category
○ Quality
○ Type Standard
● Program

Programs:
```
FERROUS
IRON
IRONMN
IRONSI
LOWALLOY
NRS
```

Figure 7.30

- ♦ Click on **Select Samples** (see Figure 7.29).
- ♦ In the list box **Available Calibration Samples** double-click on the first sample to be selected. It will be transferred to the **Selected** box.
- ♦ In the **Cassette Number** box specify the cassette number.



Program: TUTORIAL

Matrix: STEEL Sub-group: ALLOYED

Available: (14)
```
BAS    401/1    *
BAS    402/1    *
BAS    403/1    *
BAS    404/1    *
BAS    405/1    *
BAS    406/1    *
```

Selected: (11)
```
BAS    401/1    236
BAS    402/1    237
BAS    403/1    238
BAS    404/1    239
BAS    405/1    240
BAS    406/1    241
```

Cassette Number: 236

Figure 7.31

Repeat the same procedure until all samples and their cassette number have been selected.

- ♦ Click on **OK**.
- ♦ Exit to the main menu.

Prepare a Batch File

In the main menu:

- Click on **Production / Analysis** in the main.
- Click on the icon **Set Batches**.

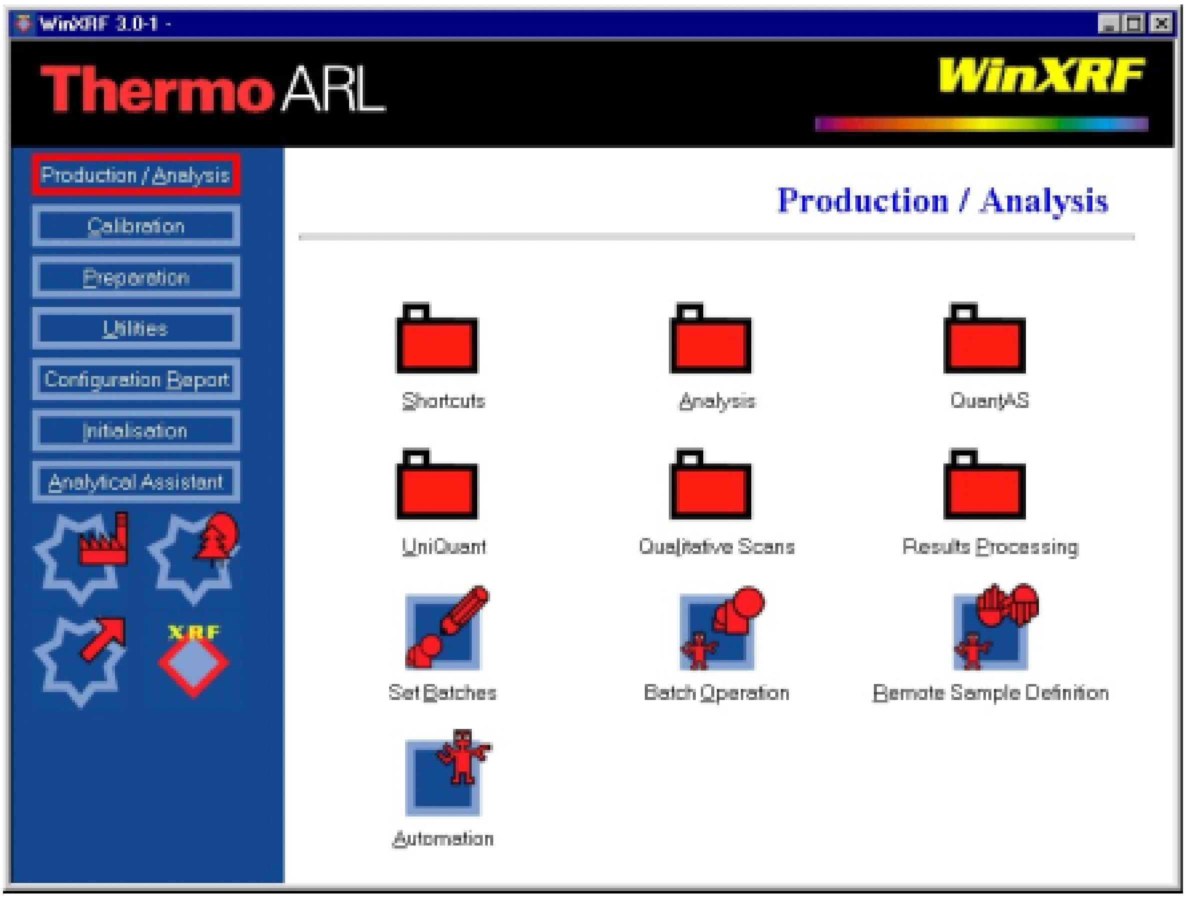


Figure 7.32

- Click on **Create**.
- Enter the name of the batch file to create in the entry box.
- Click on **OK**.

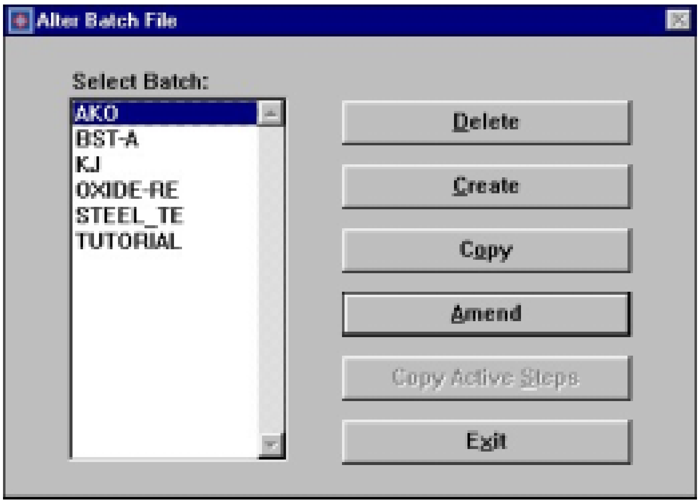
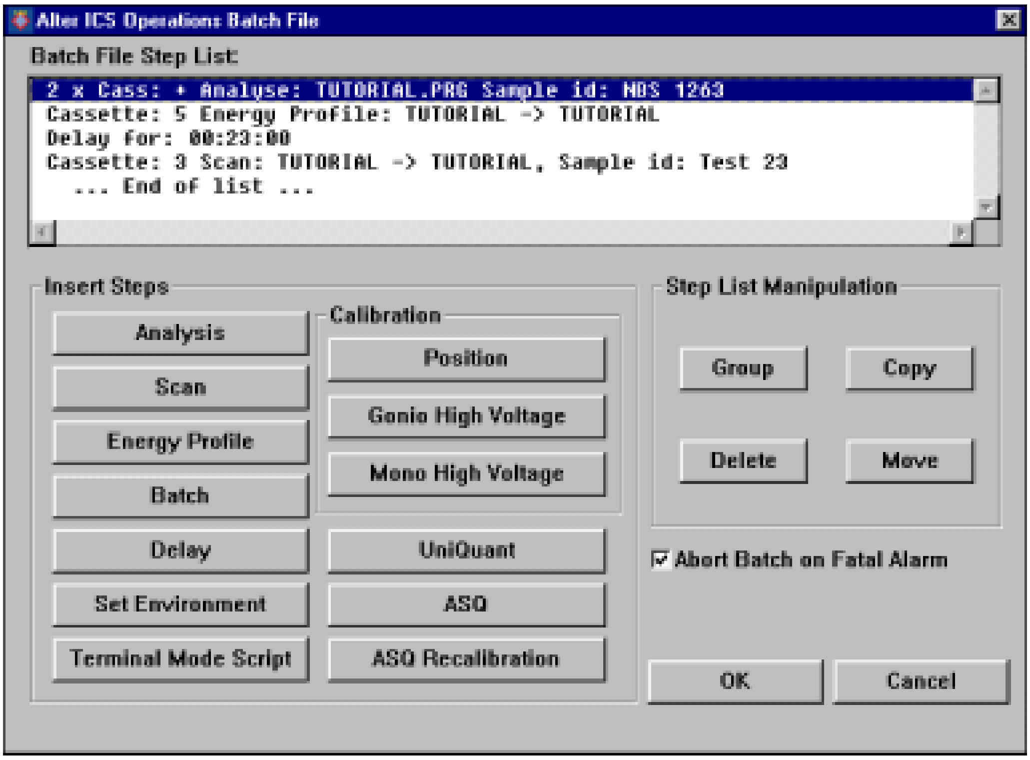


Figure 7.33

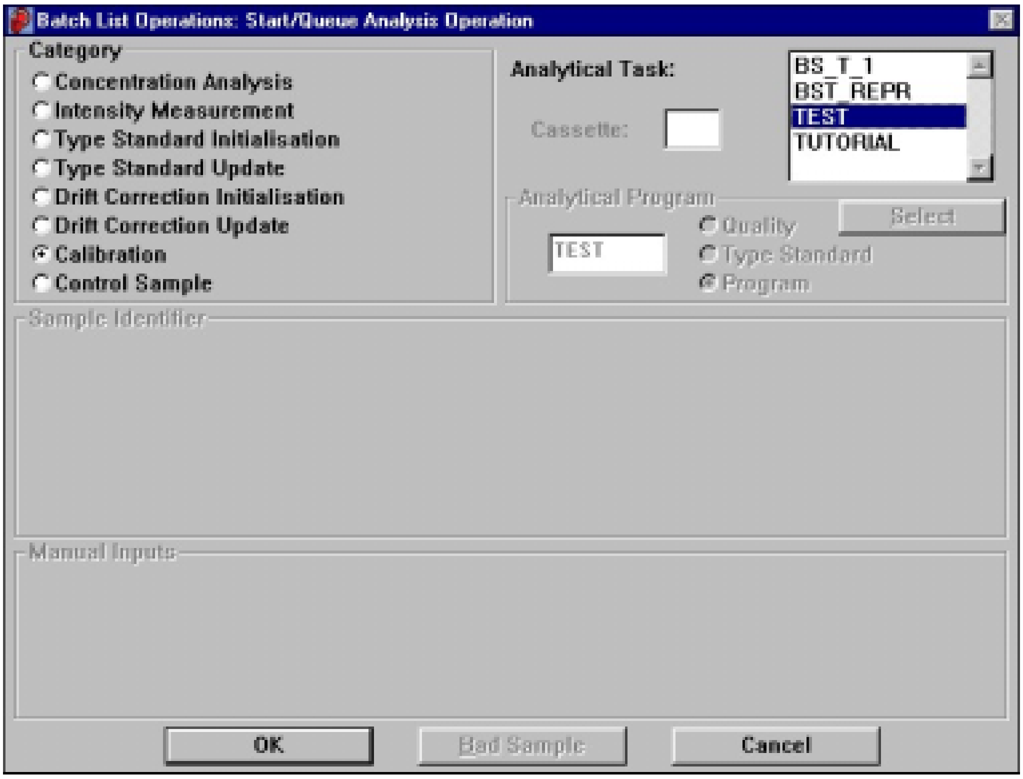
- Click on **Analysis**.



```
2 x Cass: + Analyse: TUTORIAL.PRG Sample id: NBS 1263
Cassette: 5 Energy Profile: TUTORIAL -> TUTORIAL
Delay for: 00:23:00
Cassette: 3 Scan: TUTORIAL -> TUTORIAL, Sample id: Test 23
  ... End of list ...
```

Figure 7.34

- As for **Category** select **Calibration**.
- Select the **Analytical Task**.
- Click on **OK** and return to the main menu.



Batch List Operations: Start/Queue Analysis Operation

Category
○ Concentration Analysis
○ Intensity Measurement
○ Type Standard Initialisation
○ Type Standard Update
○ Drift Correction Initialisation
○ Drift Correction Update
● Calibration
○ Control Sample

Analytical Task: BS_T_1, BST_REPR, **TEST**, TUTORIAL

Cassette:

Analytical Program: TEST
○ Quality
○ Type Standard
● Program

Figure 7.35

Measure the Calibration Samples

- Place the samples onto the corresponding magazine positions.
- In the main menu click on **Production / Analysis** and then click on **Batch Operation** (see Figure 7.32).
- Select the appropriate batch.
- As for **Repeat Count** type **1**.
- Click on **OK**.

The calibration samples will now be measured in the background and the intensities stored automatically in the calibration standards file. This is indicated by the presence of the **ICS Operations Manager** at the bottom of the screen.

Calculation of the Calibration Curves

- Select **Calibration** in the main menu (see Figure 7.21).
- Click on **MVR** and then click on **File**.
- Click on **Analytical Program**.

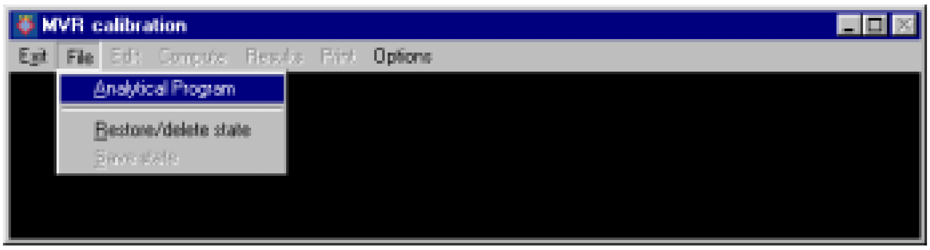
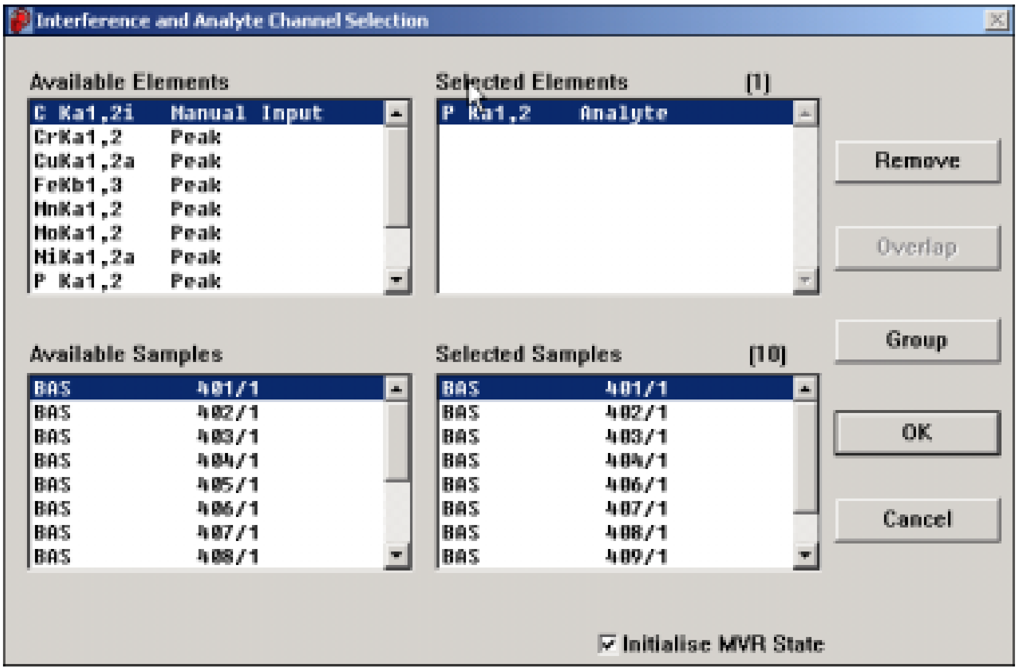


Figure 7.36

- Select the corresponding program in the list box.
- Click on **Edit** and then click on **Elements/Samples**.

Figure 7.37

- In the list box **Available Elements** double-click on the element to be selected. It will appear in the **Selected Elements** box.
- In the list box **Available Samples** click on the first sample.
- Click on **Group**.
- Scroll the last sample to be selected into the visible portion of the list box **Available Samples** using the down arrow key and double-click on it. All samples between the two selected are then transferred to the **Selected Samples** box.
- Click on **Analyte** and then on **OK**.


Interference and Analyte Channel Selection

Available Elements

C Ka1,2i	Manual Input
CrKa1,2	Peak
CuKa1,2a	Peak
FeKb1,3	Peak
MnKa1,2	Peak
MoKa1,2	Peak
NiKa1,2a	Peak
P Ka1,2	Peak

Selected Elements (1)

P Ka1,2	Analyte

Available Samples

BAS	401/1
BAS	402/1
BAS	403/1
BAS	404/1
BAS	405/1
BAS	406/1
BAS	407/1
BAS	408/1

Selected Samples (10)

BAS	401/1
BAS	402/1
BAS	403/1
BAS	404/1
BAS	406/1
BAS	407/1
BAS	408/1
BAS	409/1

☑ Initialise MVR State

Figure 7.38

- Click on **Compute** and then on **New Base Curve**.

Figure 7.39

- To inspect the numerical result in terms of differences between the certified and the calculated concentrations as well as the standard error of estimate, click on **Results** and then on **Numeric & Store**.

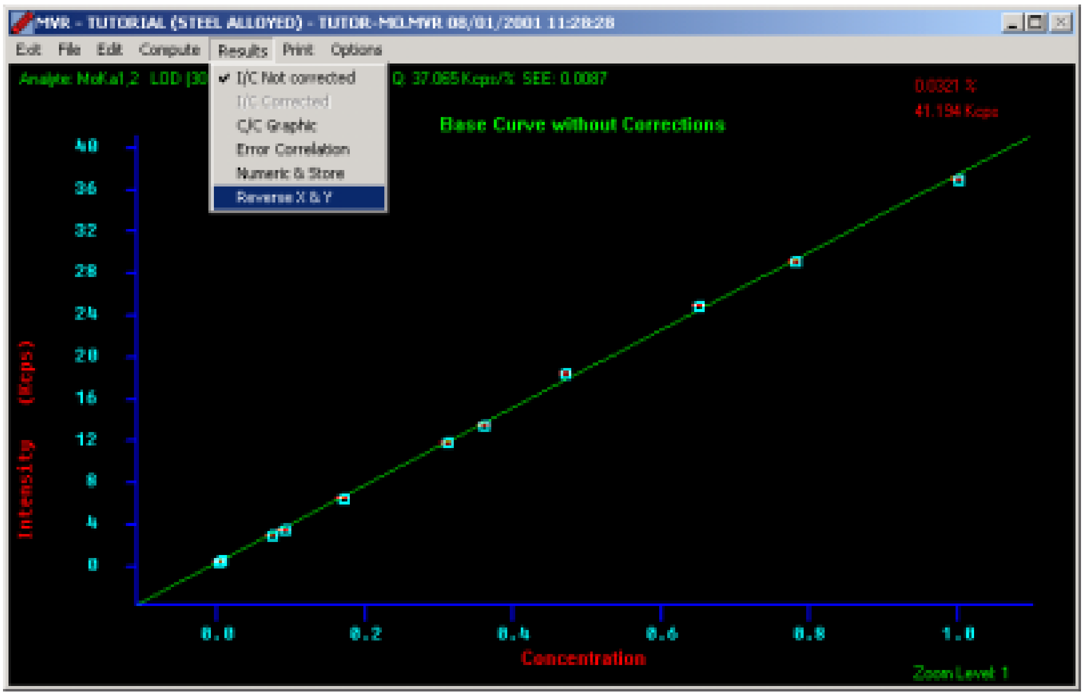
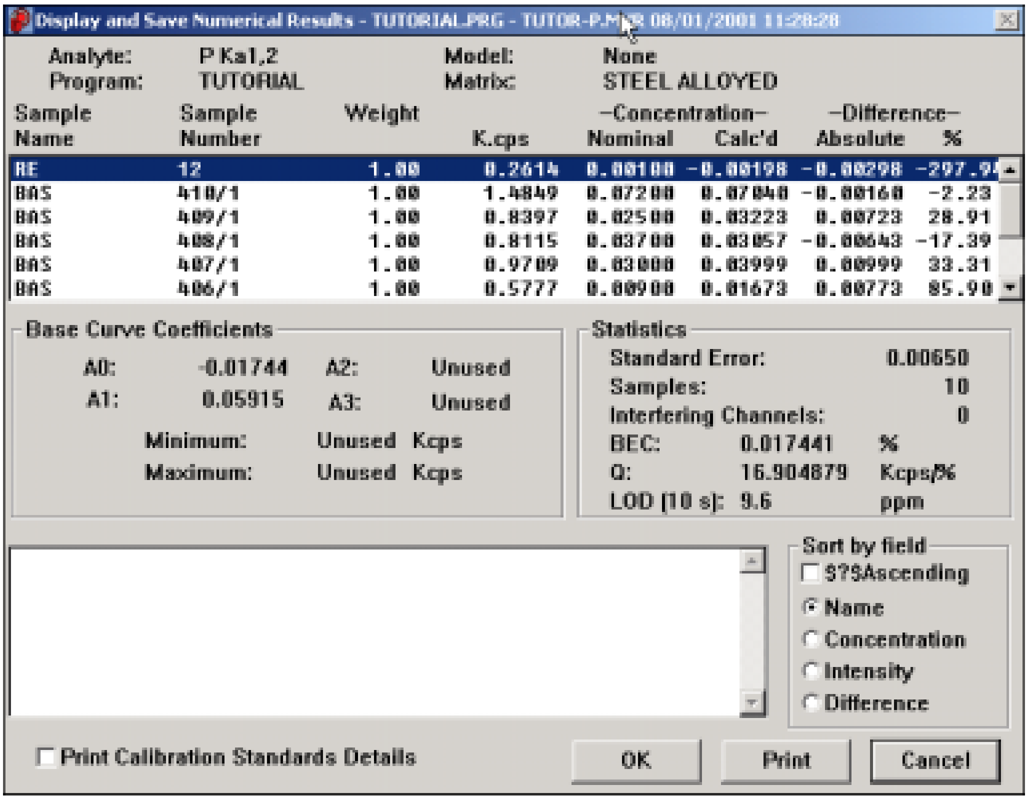


Figure 7.40

If the result is acceptable click on **OK** when you will be prompted with a message **Save Coefficients in XXXXX program?**. To store the **Base Curve Coefficients** in the analytical program click on **OK**. If you do not want to overwrite existing coefficients click **Cancel**.



Display and Save Numerical Results - TUTORIAL.PRG - TUTOR-P.M[illegible]R 08/01/2001 11:28:28

Analyte: P Ka1,2
Program: TUTORIAL
Model: None
Matrix: STEEL ALLOYED

Sample Name	Sample Number	Weight	K.cps	–Concentration– Nominal	Calc'd	–Difference– Absolute	%
RE	12	1.00	0.2614	0.00100	-0.00198	-0.00298	-297.9
BAS	410/1	1.00	1.4849	0.07200	0.07040	-0.00160	-2.23
BAS	409/1	1.00	0.8397	0.02500	0.03223	0.00723	28.91
BAS	408/1	1.00	0.8115	0.03700	0.03057	-0.00643	-17.39
BAS	407/1	1.00	0.9709	0.03000	0.03999	0.00999	33.31
BAS	406/1	1.00	0.5777	0.00900	0.01673	0.00773	85.90

Base Curve Coefficients

A0: -0.01744 A2: Unused
A1: 0.05915 A3: Unused
Minimum: Unused Kcps
Maximum: Unused Kcps

Statistics

Standard Error: 0.00650
Samples: 10
Interfering Channels: 0
BEC: 0.017441 %
Q: 16.904879 Kcps/%
LOD [10 s]: 9.6 ppm

Sort by field
☐ $?$Ascending
● Name
○ Concentration
○ Intensity
○ Difference

☐ Print Calibration Standards Details

Figure 7.41

After having applied the same procedure to all the remaining elements, it is now possible to determine the concentrations of these elements in unknown samples.

Drift Correction Update (Recalibration)

At regular intervals, the Setting-up Samples have to be measured again to correct the instrumental drift. As this is almost the same procedure as the **Drift Correction Initialisation**, the reader is asked to refer to the corresponding section in this manual or to consult the *WinXRF Operator* and *WinXRF Configuration Manuals*.

8

ROUTINE ANALYSIS

8 ROUTINE ANALYSIS

Concentration Analysis

The concentration analysis is the most frequent task used in process control. It is only possible if a calibration matching with the matrix to be analysed has previously been performed.

Prepare the Concentration Analysis

- Click on **Preparation** in the main menu and then click on **Tasks**.
- Click on **Concentration Analysis**.

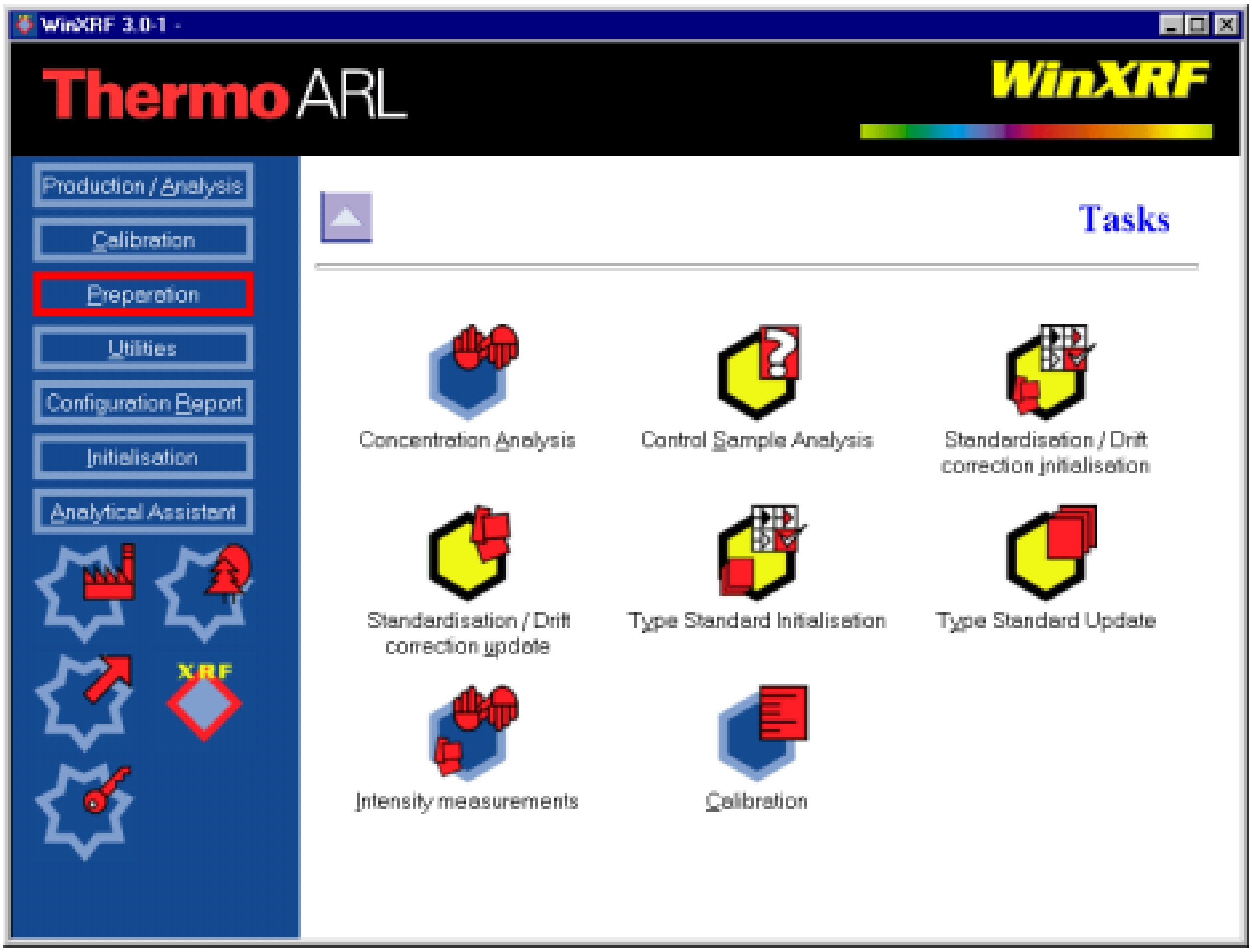
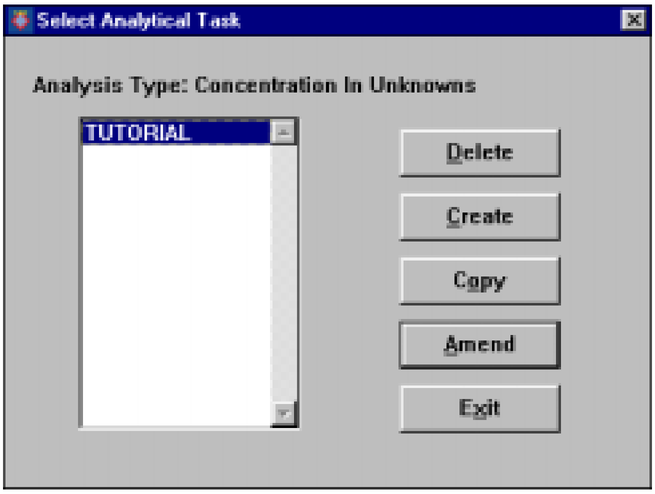


Figure 8.1

- Click on **Create**.



Analysis Type: Concentration In Unknowns

TUTORIAL

Figure 8.2

- In the **New Task Name** box enter the desired task name.

Enter New Task Name: Test

Figure 8.3

- Click on **OK**.

- In **Description** box type the desired description.

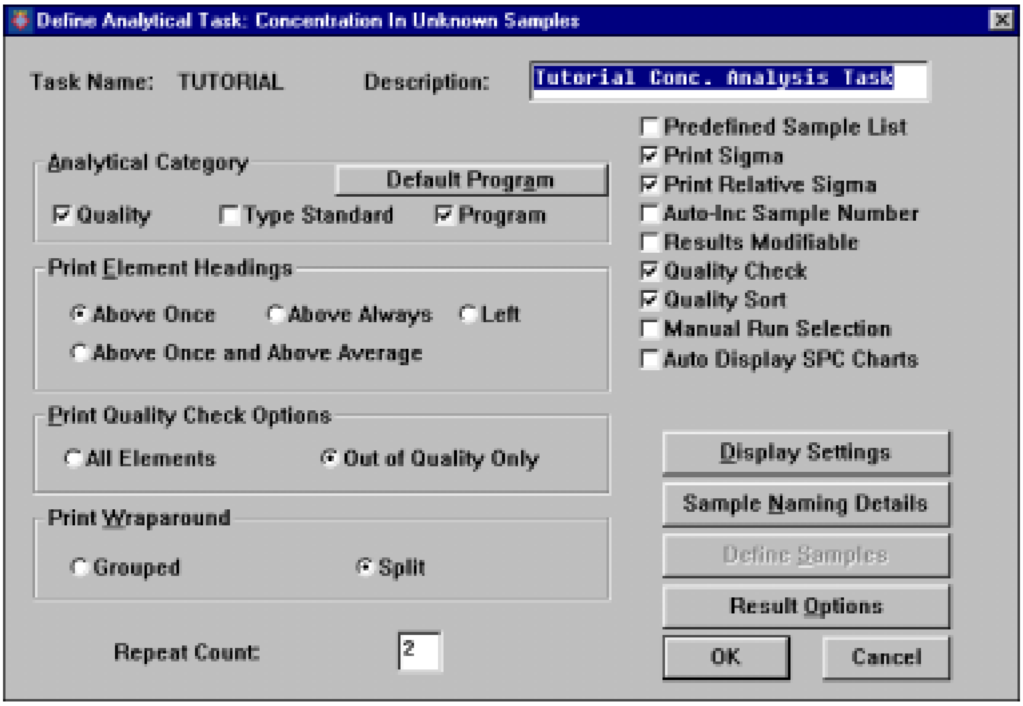
The various task options can be selected by clicking onto the corresponding button (a cross will appear in the button when activated). These are:

- It is possible to **Predefine** a **Sample List**.
- It is possible to **Display** the **Sigma**.
- It is possible to **Display** the **Relative Sigma**.
- It is possible to use **Automatic Sample Number Incrementation**.
- It is possible to **Modify** the **Results**.
- It is possible to activate the **Manual Run Selection**.

To amend the sample naming structure, click on **Sample Naming Details**.

The number of measurements for each sample can be specified in the box facing **Repeat Count**.

To define the program that should appear when the task is selected, click on **Default Program**.



Task Name: TUTORIAL
Description: Tutorial Conc. Analysis Task

Analytical Category
☑ Quality ☐ Type Standard ☑ Program

Print Element Headings
● Above Once ○ Above Always ○ Left
○ Above Once and Above Average

Print Quality Check Options
○ All Elements ● Out of Quality Only

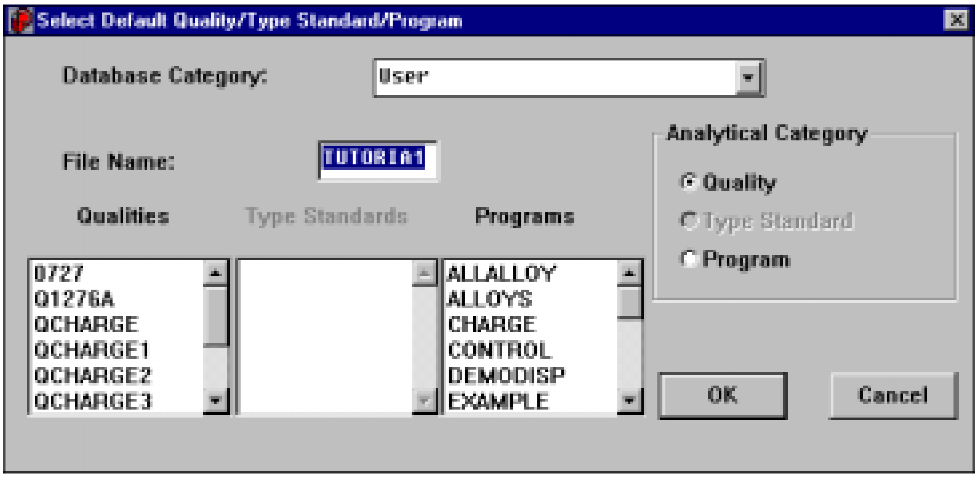
Print Wraparound
○ Grouped ● Split

Repeat Count: 2

☐ Predefined Sample List
☑ Print Sigma
☑ Print Relative Sigma
☐ Auto-Inc Sample Number
☐ Results Modifiable
☑ Quality Check
☑ Quality Sort
☐ Manual Run Selection
☐ Auto Display SPC Charts

Figure 8.4

- ♦ In the list box **Programs** highlight the desired program. If the program is not listed, then select the appropriate **Database Category**.



Database Category: User
File Name: TUTORIA1

Qualities	Type Standards	Programs
0727 Q1276A QCHARGE QCHARGE1 QCHARGE2 QCHARGE3		ALLALLOY ALLOYS CHARGE CONTROL DEMODISP EXAMPLE

Analytical Category
● Quality
○ Type Standard
○ Program

Figure 8.5

- ♦ Click on **OK** to confirm and return to the main menu.

Perform Concentration Analyses

- ♦ On the main menu bar click on **Production / Analysis**,
- ♦ then click on **Analysis** and **Concentration Analysis**.
- ♦ Please note that the concentration analysis can also be invoked by simply pressing **<F2>**.

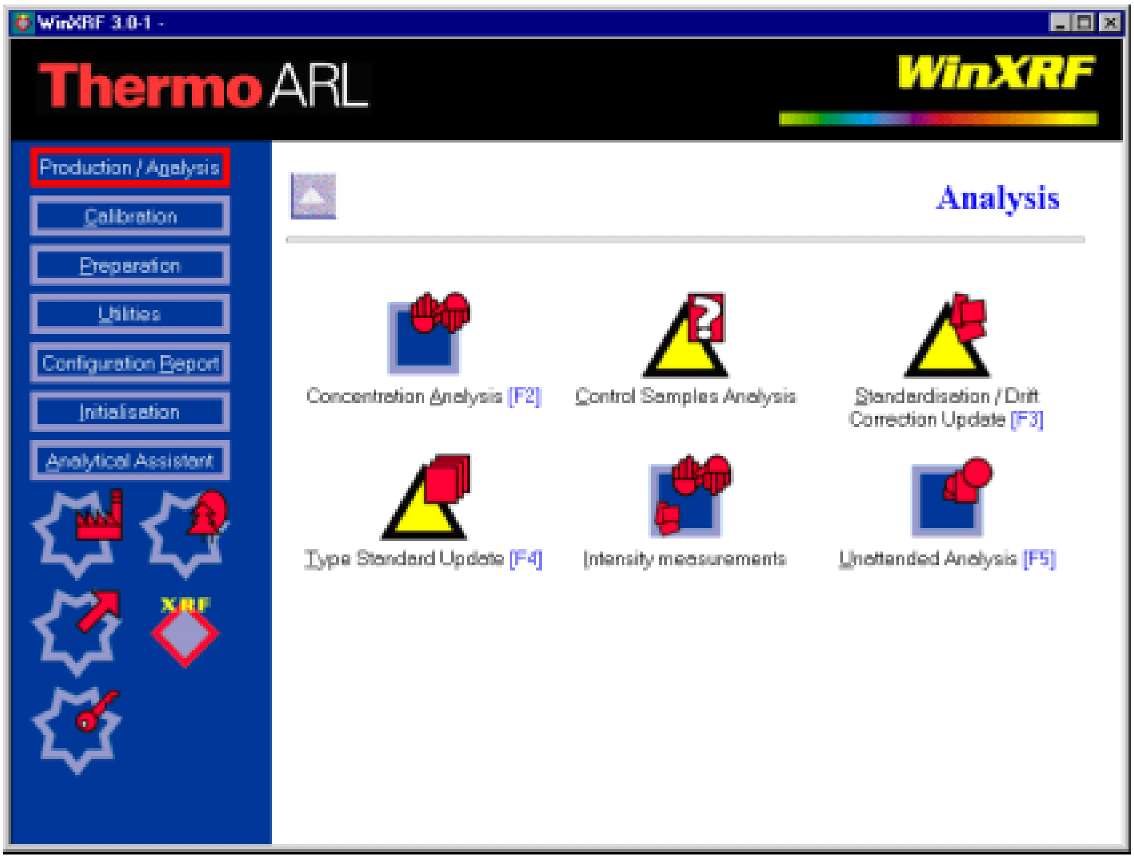
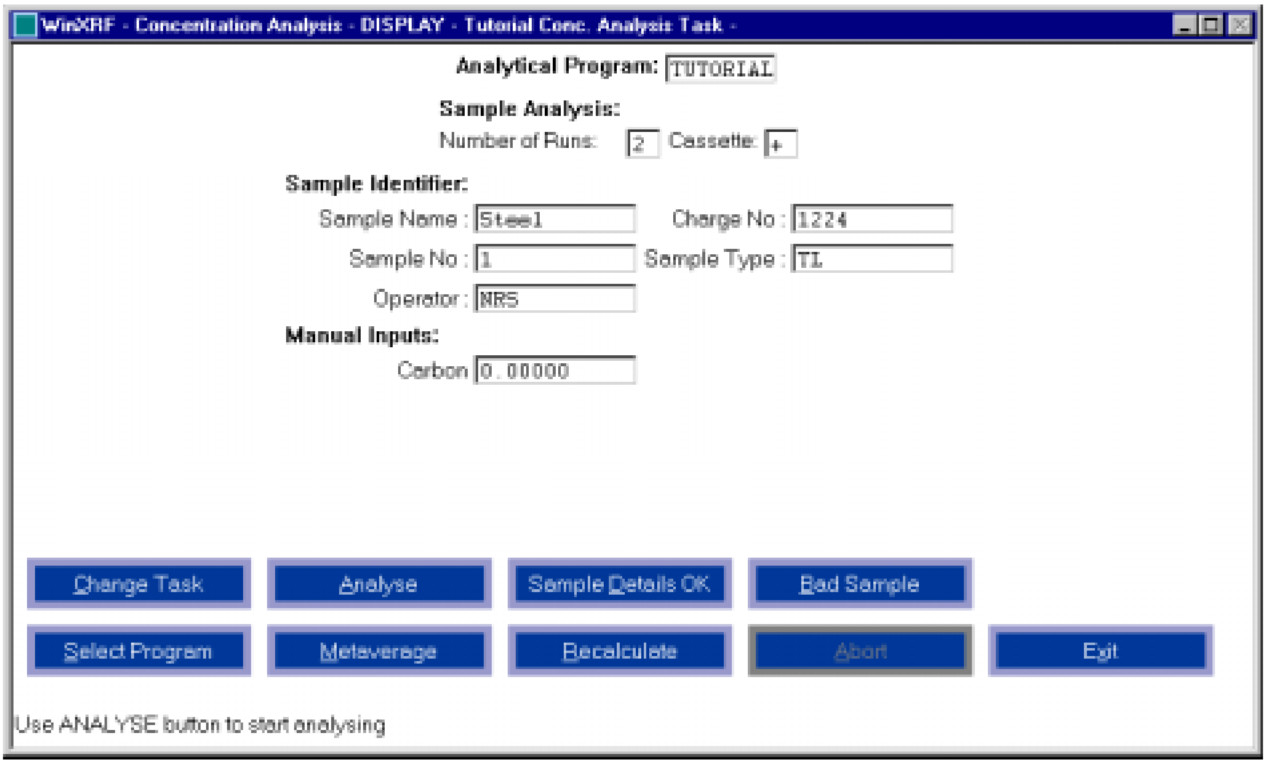


Figure 8.6

♦ Click on **Change Task**.



Analytical Program: TUTORIAL

Sample Analysis:

Number of Runs: 2 Cassette: +

Sample Identifier:

Sample Name : Steel

Charge No : 1224

Sample No : 1

Sample Type : TI

Operator : NRS

Manual Inputs:

Carbon 0.00000

Use ANALYSE button to start analysing

Figure 8.7

- Select the previously prepared task from the displayed list box and click on **OK**.

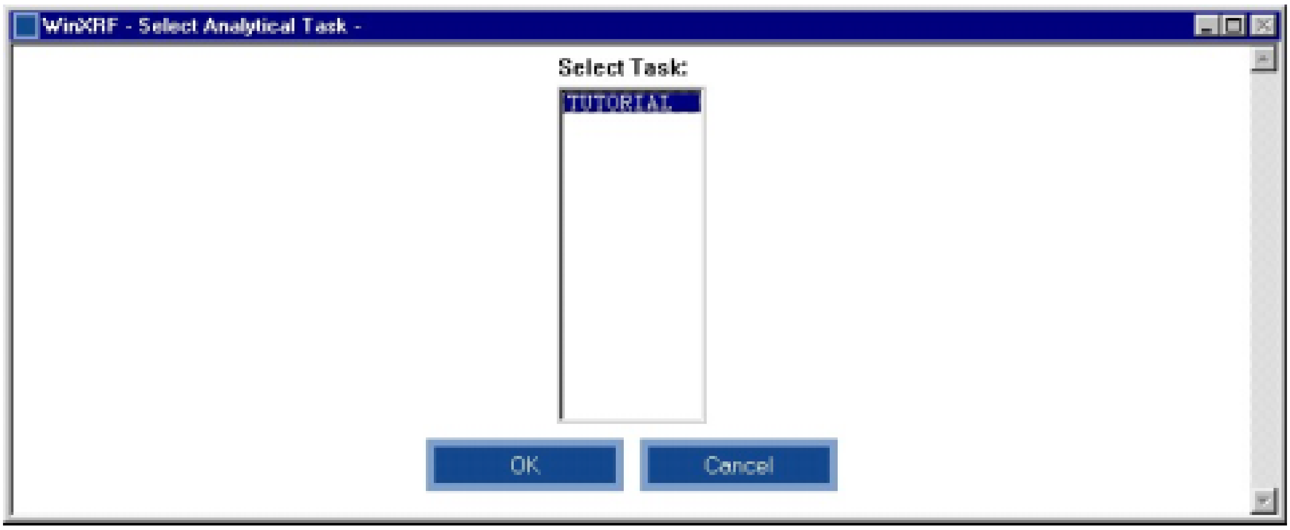
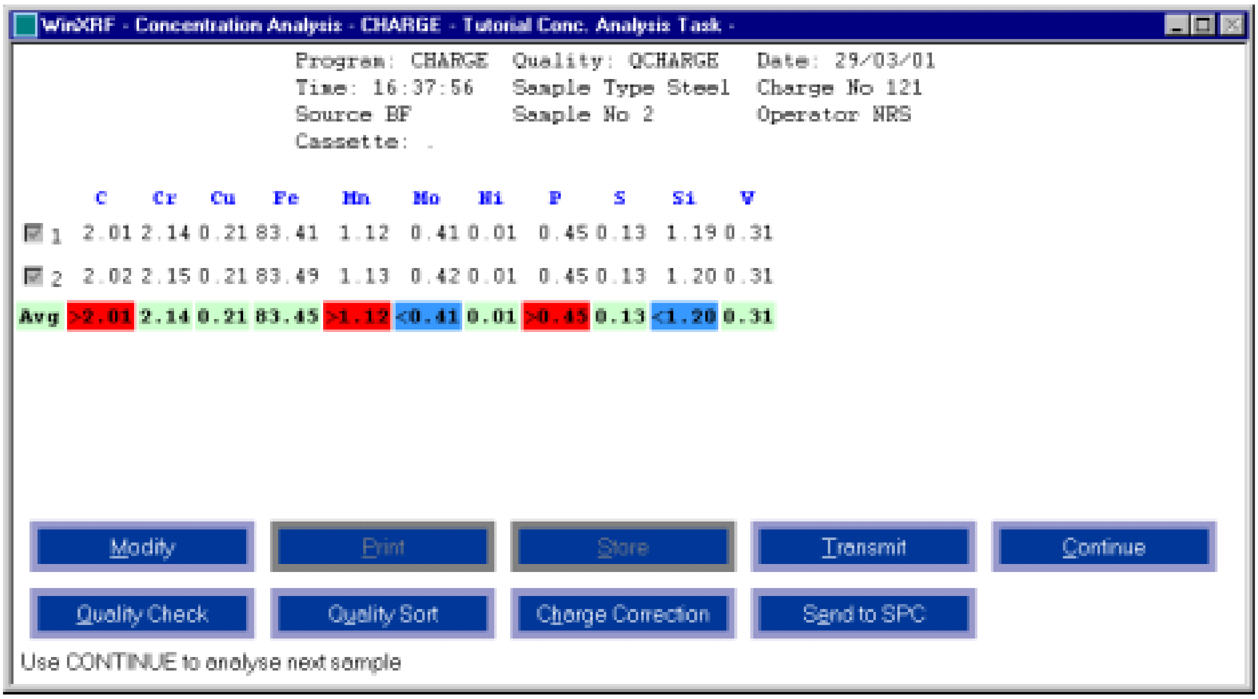


Figure 8.8

The **Cassette** number and the **Number of Runs** can then be specified. The analysis can now be started immediately by clicking on **Analyse**, or after the sample identity has been specified, by clicking on **Sample Details OK** (see Figure 8.7).

The sample will now be analysed, and the results for the specified elements will appear in the result window, and/or will be printed out.



```
Program: CHARGE   Quality: QCHARGE     Date: 29/03/01
Time: 16:37:56    Sample Type Steel    Charge No 121
Source BF         Sample No 2          Operator NRS
Cassette: .

       C     Cr    Cu    Fe     Mn     Mo    Ni     P     S      Si    V
☑ 1  2.01  2.14  0.21  83.41  1.12   0.41  0.01  0.45  0.13   1.19  0.31
☑ 2  2.02  2.15  0.21  83.49  1.13   0.42  0.01  0.45  0.13   1.20  0.31
Avg  >2.01 2.14  0.21  83.45  >1.12  <0.41 0.01  >0.45 0.13   <1.20 0.31
```

Use CONTINUE to analyse next sample

Figure 8.9

Intensity Measurement

The measurement of intensities is mainly used to check the reliability and the short and long term reproducibility of the instrument.

The result issued in intensity measurement mode is not subject to drift correction, it is a raw intensity representative of the instrumental response.

Prepare the Intensity Measurement Task

- Click on **Preparation** in the main menu and then click on **Tasks** (see Figure 8.1).
- Click on **Intensity Measurements**.
- Click on **Create**.

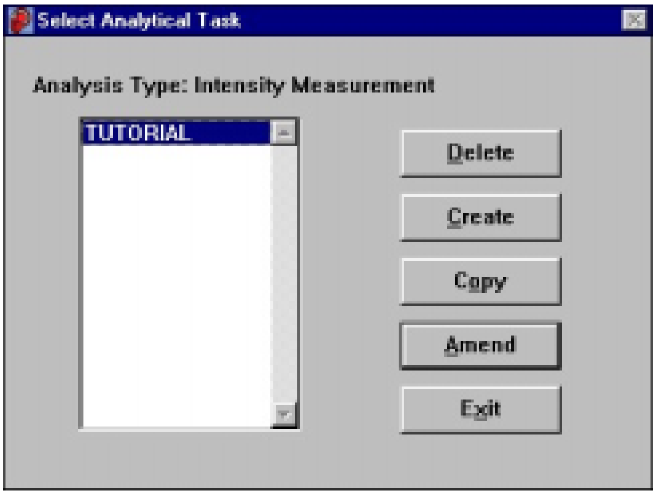


Figure 8.10

- In the **Enter New Task Name** box type the new task name.

Enter New Task Name: Test

Figure 8.11

- Click on **OK**.

- In **Description** box type the desired description.

The various task options can be selected by clicking onto the corresponding button (a cross will appear in the button when activated). These are:

- It is possible to **Predefine** a **Sample List**.
- It is possible to **Display** the **Sigma**.
- It is possible to **Display** the **Relative Sigma**.

- It is possible to use **Automatic Sample Number Incrementation**.
- It is possible to **Modify** the **Results**.
- It is possible to activate the **Manual Run Selection**.

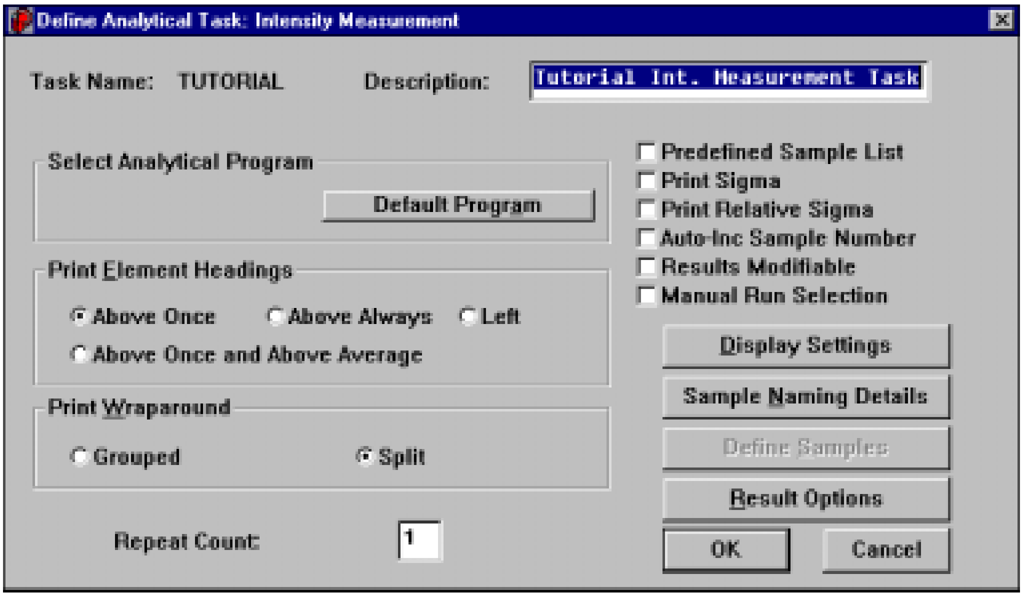
To amend the sample naming structure:

- Click on **Sample Naming Details**.

The number of measurements for each sample can be specified in the box facing **Repeat Count**.

To define the program that should appear when the task is selected:

- Click on **Default Program**.



Define Analytical Task: Intensity Measurement

Task Name: TUTORIAL

Description: Tutorial Int. Measurement Task

Select Analytical Program

Print Element Headings

● Above Once ○ Above Always ○ Left

○ Above Once and Above Average

Print Wraparound

○ Grouped ● Split

☐ Predefined Sample List

☐ Print Sigma

☐ Print Relative Sigma

☐ Auto-Inc Sample Number

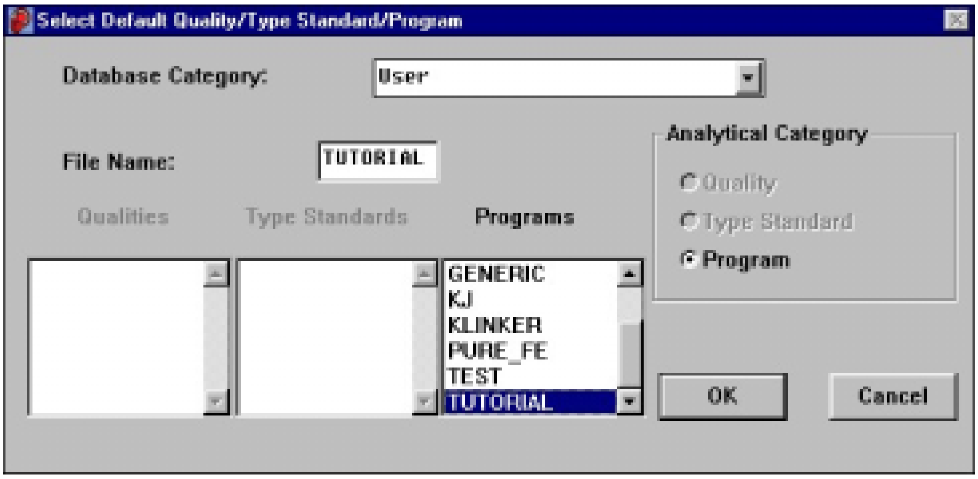
☐ Results Modifiable

☐ Manual Run Selection

Repeat Count: 1

Figure 8.12

- In the list box **Programs** highlight the desired program. If the program is not listed, then select the appropriate **Database Category**.



Select Default Quality/Type Standard/Program

Database Category: User

File Name: TUTORIAL

Analytical Category

○ Quality

○ Type Standard

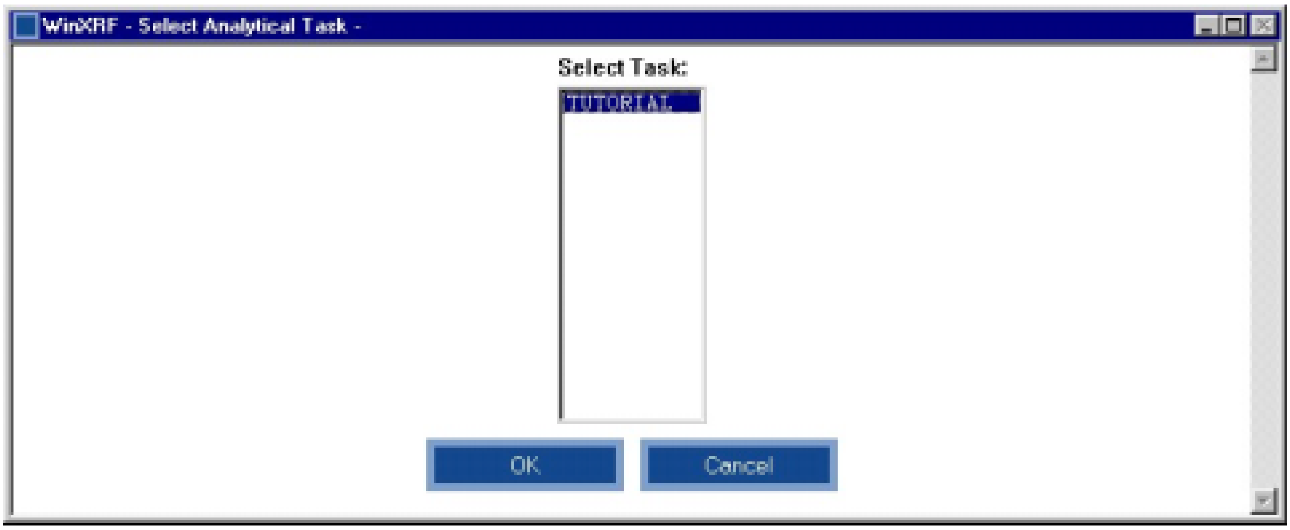
● Program

Figure 8.13

- Click on **OK** to confirm and return to the main menu.

Perform Intensity Measurements

- Click on **Production / Analysis** in the main menu and then click on **Analysis** (see Figure 8.6).
- Click on **Intensity Measurements**.
- Click on **Change Task** and select the previously prepared task from the displayed list box.


WinXRF - Select Analytical Task -

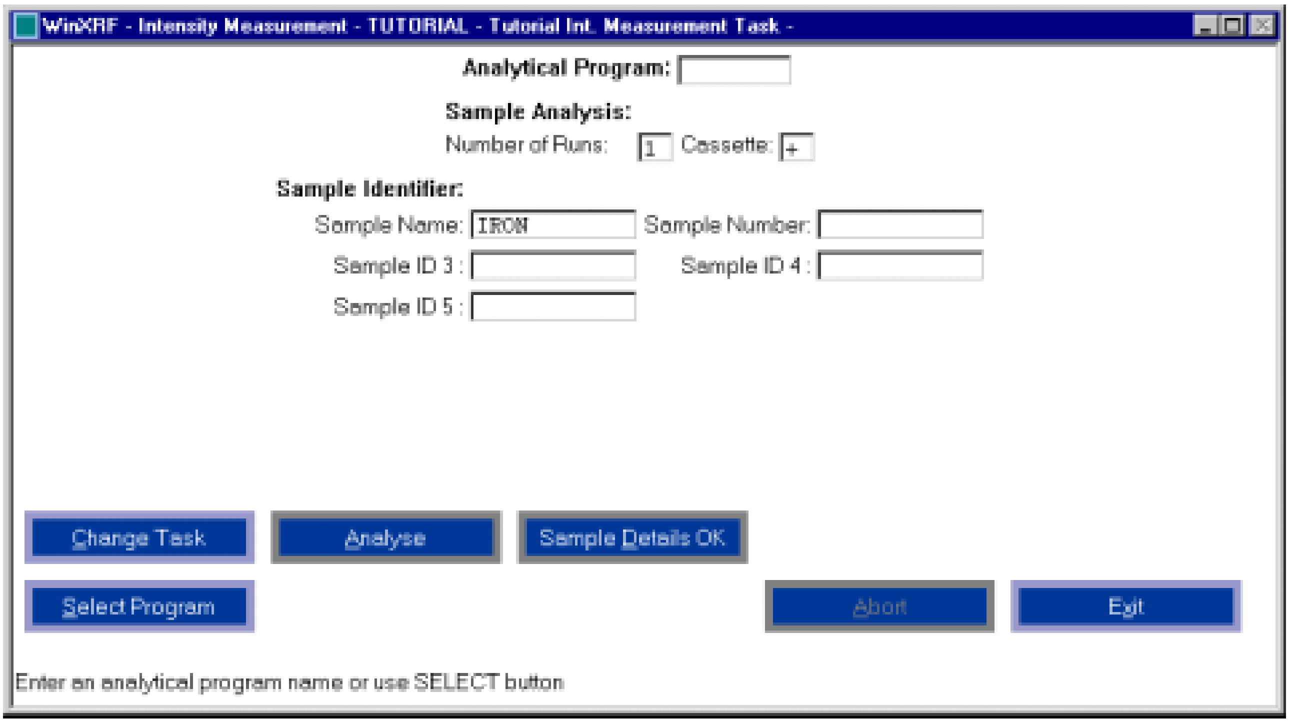
Select Task: TUTORIAL

Figure 8.14

- Click on **OK**.

The **Cassette** number and the **Number of Runs** can then be specified.

- The analysis can now be started immediately by clicking on **Analyse**, or after the sample identity has been specified, by clicking on **Sample Details OK**.


WinXRF - Intensity Measurement - TUTORIAL - Tutorial Int. Measurement Task -

Sample Analysis:
Number of Runs: 1
Cassette: +

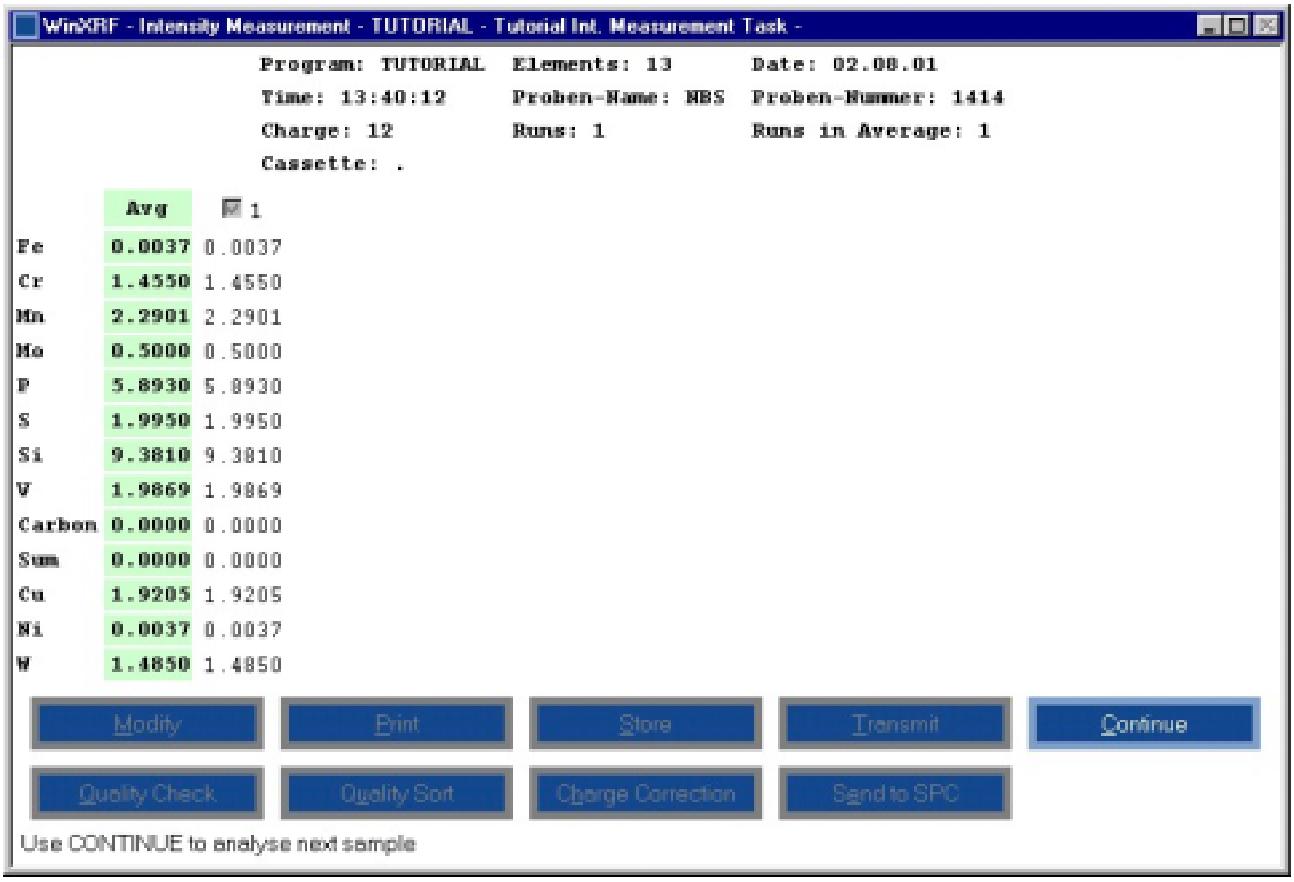
Sample Identifier:
Sample Name: IRON

Enter an analytical program name or use SELECT button

Figure 8.15

Exit

The sample will now be analysed, and the results for the specified elements will appear in the result window, and/or will be printed out.



WinXRF - Intensity Measurement - TUTORIAL - Tutorial Int. Measurement Task -

Program: TUTORIAL Elements: 13 Date: 02.08.01
Time: 13:40:12 Proben-Name: NBS Proben-Nummer: 1414
Charge: 12 Runs: 1 Runs in Average: 1
Cassette: .

	Avg	☑ 1
Fe	0.0037	0.0037
Cr	1.4550	1.4550
Mn	2.2901	2.2901
Mo	0.5000	0.5000
P	5.8930	5.8930
S	1.9950	1.9950
Si	9.3810	9.3810
V	1.9869	1.9869
Carbon	0.0000	0.0000
Sum	0.0000	0.0000
Cu	1.9205	1.9205
Ni	0.0037	0.0037
W	1.4850	1.4850

Use CONTINUE to analyse next sample

Figure 8.16

A

ALARMS

A ALARMS

Alarms are used to indicate events to the ACS. Each node of the ICS holds an alarms table where a corresponding alarm is set every time an event occurs. The nodes of the ICS having a master function on other nodes, ask continuously for the contents of the alarms table of their slave nodes, and adds them to their own table. The ICS is therefore, always ready to indicate any problem to the ACS.

Each alarm number is a reference to an alarms list (see following tables) where it is possible to find its significance. Each node of the ICS has a reserved range of alarm numbers.

Each alarm has 1 among 4 severity levels. The alarms range reserved for a node is divided into 4 sub-ranges, each sub-range corresponding to an alarm severity level.

The 4 severity levels have the following significance:

FATAL	**(F) :**	It is impossible for the instrument to perform the requested operation.
WARNING	**(W) :**	Something in the instrument is not in order, but it is still possible to carry on with the requested operation.
INFORMATION	**(I) :**	A normal event has occurred.
DIAGNOSTIC	**(D) :**	An abnormal event has occurred, but the instrument has recovered from that abnormal event.

No severity level is applicable to the alarm 0 (Instrument busy). This alarm is used to synchronise the ACS with the ICS during certain operations.

The reserved ranges for each ICS node are summarised in the following table.

ICS node	**Alarm numbers range**			
	Fatal	**Warning**	**Information**	**Diagnostic**
XQI	256 to 767	768 to 1279	1280 to 1537	1538 to 2047
XSM	2048 to 2559	2560 to 3327 + 65535	3328 to 3583	3584 to 4095
XGT	4096 to 4607	4608 to 5375	5376 to 5631	5632 to 6143
XCM	6144 to 6399	6400 to 6655	6656 to 6911	6912 to 7167
XSP	7168 to 7423	7424 to 7679	7680 to 7935	7936 to 8191
XPS	8192 to 8703	8704 to 9471	9472 to 9727	9728 to 10239
XHI	10240 to 10495	10496 to 10751	10752 to 11007	11008 to 11263
reserved	11264 to 12287			
XST	12288 to 12543	12544 to 12799	12800 to 13055	13056 to 13311
reserved	13312 to 14335			
Gonio1: XGI	14336 to 14847	14848 to 15615	15616 to 15871	15872 to 16383
Gonio1: XGDI	16384 to 16895	16896 to 17663	17664 to 17919	17920 to 18431
Gonio1: XDI	18432 to 18687	18688 to 18943	18944 to 19199	19200 to 19455
Gonio2: XGI	19456 to 19967	19968 to 20735	20736 to 20991	20992 to 21503
Gonio2: XGDI	21504 to 22015	22016 to 22783	22784 to 23039	23040 to 23551
Gonio2: XDI	23552 to 23807	23808 to 24063	24064 to 24319	24320 to 24575
Gonio3: XGI	24576 to 25087	25088 to 25855	25856 to 26111	26112 to 26623
Gonio3: XGDI	26624 to 27135	27136 to 27903	27904 to 28159	28160 to 28671
Gonio3: XDI	28672 to 28927	28928 to 29183	29184 to 29439	29440 to 29695

ICS node	Alarm numbers range											
	Fatal			**Warning**			**Information**			**Diagnostic**		
XRD1: XDD	29696	to	29823	29952	to	30079	30208	to	30335	30464	to	30591
XRD1: XDDD	29824	to	29887	30080	to	30143	30336	to	30399	30592	to	30655
XRD1: XDI	29888	to	29951	30144	to	30207	30400	to	30463	30656	to	30719
XRD2: XDD	30720	to	30847	30976	to	31103	31232	to	31359	31488	to	31615
XRD2: XDDD	30848	to	30911	31104	to	31167	31360	to	31423	31616	to	31679
XRD2: XDI	30912	to	30975	31168	to	31231	31424	to	31487	31680	to	31743
reserved	31744	to	32767									
XMI01	32768	to	33023	33024	to	33279	33280	to	33535	33536	to	33791
XMI02	33792	to	34047	34048	to	34303	34304	to	34559	34560	to	34815
XMI03	34816	to	35071	35072	to	35327	35328	to	35583	35584	to	35839
XMI04	35840	to	36095	36096	to	36351	36352	to	36607	36608	to	36863
XMI05	36864	to	37119	37120	to	37375	37376	to	37631	37632	to	37887
XMI06	37888	to	38143	38144	to	38399	38400	to	38655	38656	to	38911
XMI07	38912	to	39167	39168	to	39423	39424	to	39679	39680	to	39935
XMI08	39936	to	40191	40192	to	40447	40448	to	40703	40704	to	40959
XMI09	40960	to	41215	41216	to	41471	41472	to	41727	41728	to	41983
XMI10	41984	to	42239	42240	to	42495	42496	to	42751	42752	to	43007
XMI11	43008	to	43263	43264	to	43519	43520	to	43775	43776	to	44031
XMI12	44032	to	44287	44288	to	44543	44544	to	44799	44800	to	45055
XMI13	45056	to	45311	45312	to	45567	45568	to	45823	45824	to	46079
XMI14	46080	to	46335	46336	to	46591	46592	to	46847	46848	to	47103
XMI15	47104	to	47359	47360	to	47615	47616	to	47871	47872	to	48127
XMI16	48128	to	48383	48384	to	48639	48640	to	48895	48896	to	49151
XMI17	49152	to	49407	49408	to	49663	49664	to	49919	49920	to	50175
XMI18	50176	to	50431	50432	to	50687	50688	to	50943	50944	to	51199
XMI19	51200	to	51455	51456	to	51711	51712	to	51967	51968	to	52223
XMI20	52224	to	52479	52480	to	52735	52736	to	52991	52992	to	53247
XMI21	53248	to	53503	53504	to	53759	53760	to	54015	54016	to	54271
XMI22	54272	to	54527	54528	to	54783	54784	to	55039	55040	to	55295
XMI23	55296	to	55551	55552	to	55807	55808	to	56063	56064	to	56319
XMI24	56320	to	56575	56576	to	56831	56832	to	57087	57088	to	57343
XMI25	57344	to	57599	57600	to	57855	57856	to	58111	58112	to	58367
XMI26	58368	to	58623	58624	to	58879	58880	to	59135	59136	to	59391
XMI27	59392	to	59647	59648	to	59903	59904	to	60159	60160	to	60415
XMI28	60416	to	60671	60672	to	60927	60928	to	61183	61184	to	61439
XMI29	61440	to	61695	61696	to	61951	61952	to	62207	62208	to	62463
XMI30	62464	to	62719	62720	to	62975	62976	to	63231	63232	to	63487
XMI31	63488	to	63743	63744	to	63999	64000	to	64255	64256	to	64511
XMI32	64512	to	64767	64768	to	65023	65024	to	65279	65280	to	65534

Exit

B

STATUS CHANNELS

B STATUS CHANNELS

“Status channels” are used to monitor and retrieve different status of the instrument. Each node of the ICS is allocated a range of status channels numbers.

The following list shows the allocated status channel numbers allocated to the different nodes of the ICS.

Node	Status channels range
XQI	1 - 511
XSM	512 - 1023
XGT98	1024 - 1535
XCM	1536 - 2047
XPS	2048 - 2559
XHI	2560 - 3071
XST	3072 - 3583
XSP	3584 - 4095
XGI1	8192 - 8703
XGDI1	8704 - 9215
XDI1	9216 - 9727
XGI2	10240 - 10751
XGDI2	10752 - 11263
XDI2	11264 - 11775
XGI3	12288 - 12799
XGDI3	12800 - 13311
XDI3	13312 - 13823
XDD1	14336 - 14463
XDDD1	14464 - 14591
XDI4	14592 - 14619
XDD2	14848 - 14975
XDDD2	14976 - 15103
XDI5	15104 - 15231

Node	Status channels range
all XMI’s*	15872 - 16383
XMI01	16384 - 16895
XMI02	16896 - 17407
XMI03	17408 - 17919
XMI04	17920 - 18431
XMI05	18432 - 18943
XMI06	18944 - 19455
XMI07	19456 - 19967
XMI08	19968 - 20479
XMI09	20480 - 20991
XMI10	20992 - 21503
XMI11	21504 - 22015
XMI12	22016 - 22527
XMI13	22528 - 23039
XMI14	23040 - 23551
XMI15	23552 - 24063
XMI16	24064 - 24575
XMI17	24576 - 25087
XMI18	25088 - 25599
XMI19	25600 - 26111
XMI20	26112 - 26623
XMI21	26624 - 27135
XMI22	27136 - 27647
XMI23	27648 - 28159
XMI24	28160 - 28671
XMI25	28672 - 29183
XMI26	29184 - 29695
XMI27	29696 - 30207
XMI28	30208 - 30719
XMI29	30720 - 31231
XMI30	31232 - 31743
XMI31	31744 - 32255
XMI32	32256 - 32767

*Available for the “Set status” command (ST) only.

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The following table describes all the valid status channels defined in the ICS. This list may change according to the evolution of the different firmware of each node.

Status number	Node	Status channel name
1	**XQI**	**Primary chamber pressure [Pa]**
Parameters: minimum pressure [Pa] maximum pressure [Pa] sampling period [s] sample loading maximum pressure [Pa]		
2	**XQI**	**Spectrometer tank pressure [Pa]**
Parameters: minimum pressure [Pa] maximum pressure [Pa] sampling period [s] end of pumping pressure [Pa] maximum working pressure [Pa] desired regulation pressure [Pa]		
3	**XQI**	**Electronic rack temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		
4	**XQI**	**Mains voltage (L1 phase) [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
5	**XQI**	**XQI +5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
6	**XQI**	**XQI +12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
7	**XQI**	**XQI -12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
8	**XQI**	**XQI -5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		

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9	**XQI**	**XQI +5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
10	**XQI**	**XQI +2.5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
13	**XQI**	**Working time of instrument [h]**
Parameters: minimum [] (ignored) maximum working time [h] sampling period [s]		
14	**XQI**	**Working time under vacuum [h]**
Parameters: minimum [] (ignored) maximum working time [h] sampling period [s]		
16	**XQI**	**Working time under air [h]**
Parameters: minimum [] (ignored) maximum working time [h] sampling period [s]		
17	**XQI**	**Working time of vacuum pump P2 [h]**
Parameters: minimum [] (ignored) maximum working time [h] sampling period [s]		
18	**XQI**	**Number of samples loaded under vacuum []**
Parameters: minimum [] (ignored) maximum number [] sampling period [s]		
20	**XQI**	**Number of samples loaded under air []**
Parameters: minimum [] (ignored) maximum number [] sampling period [s]		
21	**XQI**	**Number of environment changes to vacuum []**
Parameters: minimum [] (ignored) maximum number [] sampling period [s]		
23	**XQI**	**Number of environment changes to air []**
Parameters: minimum [] (ignored) maximum number [] sampling period [s]		

24	**XQI**	**Number of instrument power-up's []**
Parameters: minimum [] (ignored) maximum number [] sampling period [s]		
25	**XQI**	**Pumping time of last loaded sample [s]**
Parameters: minimum [s] maximum [s] sampling period [s] minimum desired pumping time [s] maximum desired pumping time [s]		
26	**XQI**	**Working time of molecular vacuum pump P1 [h]**
Parameters: minimum [] (ignored) maximum working time [h] sampling period [s]		
512	**XSM**	**Digital voltmeter channel [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s] DVM slope [] DVM offset [V] DVM gain []		
513	**XSM**	**Molecular vacuum pump P1 speed [rpm]**
Parameters: minimum speed [rpm] maximum speed [rpm] sampling period [s]		
1024	**XGT98**	**Spectrometer tank temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s] minimum temperature error [^{0}C] maximum temperature error [^{0}C]		
1025	**XGT98**	**FPC gas pressure [Pa]**
Parameters: minimum pressure [Pa] maximum pressure [Pa] sampling period [s] minimum pressure error [Pa] maximum pressure error [Pa]		
1026	**XGT98**	**FPC gas flow [µl/min]**
Parameters: minimum flow [µl/min] maximum flow [µl/min] sampling period [s] minimum flow error [µl/min] maximum flow error [µl/min]		

1029	**XGT98**	**XGT98 +5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1030	**XGT98**	**XGT98 +12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1031	**XGT98**	**XGT98 -12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1032	**XGT98**	**XGT98 +12Volts power [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1033	**XGT98**	**XGT98 +5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1034	**XGT98**	**XGT98 +2.5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1035	**XGT98**	**XGT98 +24Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1536	**XCM**	**XCM +5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1537	**XCM**	**XCM +12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1538	**XCM**	**XCM -12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		

1539	**XCM**	**XCM +5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1540	**XCM**	**XCM +2.5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
1541	**XCM**	**XCM +24Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2048	**XPS**	**X-ray kilovolts [kV]**
Parameters: minimum relative tolerance [%] maximum relative tolerance [%] minimum constant tolerance [kV] maximum constant tolerance [kV] sampling period [s]		
2049	**XPS**	**X-ray milliamps [mA]**
Parameters: minimum relative tolerance [%] maximum relative tolerance [%] minimum constant tolerance [mA] maximum constant tolerance [mA] sampling period [s]		
2050	**XPS**	**X-ray cooling primary water input temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		
2051	**XPS**	**X-ray cooling primary water output temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		
2052	**XPS**	**X-ray cooling secondary water input temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		
2053	**XPS**	**X-ray cooling secondary water output temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s] minimum temperature error [^{0}C] maximum temperature error [^{0}C]		

2054	**XPS**	**X-ray cooling secondary water resistivity. [kΩ.cm]**
Parameters: minimum resistivity [kΩ.cm] maximum resistivity [kΩ.cm] sampling period [s]		
2055	**XPS**	**X-ray tube filament current [A]**
Parameters: minimum current [A] maximum current [A] sampling period [s]		
2056	**XPS**	**XPS +15Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2057	**XPS**	**XPS -15Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2058	**XPS**	**XPS +24Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2061	**XPS**	**XPS +5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2063	**XPS**	**X-ray activity time [hour]**
Parameters: minimum activity [hour] maximum activity [hour] sampling period [s]		
2064	**XPS**	**X-ray tube arcs count []**
Parameters: minimum arcs count [] maximum arcs count [] sampling period [s] fatal arc rate count [] fatal arc rate period [s] warning arc rate count [] warning arc rate period [s] 60 to 70 kV delay [s]		
2067	**XPSLP**	**Laboratory ambient temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		

2068	**XPSLP**	**X-ray generator output temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		
2069	**XPSLP**	**X-ray tube leakage current [mA]**
Parameters: minimum current [mA] maximum current [mA] sampling period [s]		
2070	**XPSLP**	**XPSLP board temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		
2071	**XPSLP**	**XPS +12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2072	**XPSLP**	**XPS -12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2073	**XPSLP**	**XPS +2.5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2560	**XHI**	**XHI +5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2561	**XHI**	**XHI +12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2562	**XHI**	**XHI -12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2563	**XHI**	**XHI +5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		

2564	**XHI**	**XHI +2.5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2565	**XHI**	**XHI +24Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
2566	**XHI**	**XHI -5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3072	**XST**	**XST +5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3073	**XST**	**XST +12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3074	**XST**	**XST -12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3075	**XST**	**XST +5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3076	**XST**	**XST +2.5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3077	**XST**	**XST +24Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3078	**XST**	**XST -5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		

3079	**XST**	**Absolute filtered sample rotation error [^{0}]**
Parameters: minimum error [^{0}] maximum error [^{0}] sampling period [s]		
3584	**XSP**	**XSP +5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3585	**XSP**	**Mains voltage (L1 phase) [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3586	**XSP**	**Mains voltage (L2 phase) [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3587	**XSP**	**XSP -5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3588	**XSP**	**XSP +24Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3589	**XSP**	**Molecular pump P1 voltage [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
3590	**XSP**	**Molecular pump P1 current [A]**
Parameters: minimum current [A] maximum current [A] sampling period [s]		

3591	**XSP**	**Molecular pump P1 temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		
8192	**XGI1**	**Gonio 1: detector high-voltage [V]**
10240	**XGI2**	**Gonio 2: detector high-voltage [V]**
12288	**XGI3**	**Gonio 3: detector high-voltage [V]**
14336	**XDD1**	**XRD-system 1: detector high-voltage [V]**
14848	**XDD2**	**XRD-system 2: detector high-voltage [V]**
Parameters: minimum absolute voltage [V] maximum absolute voltage [V] sampling period [s] minimum relative tolerance [V] maximum relative tolerance [V]		
8193	**XGI1**	**Gonio 1: crystals temperature [^{0}C]**
10241	**XGI2**	**Gonio 2: crystals temperature [^{0}C]**
12289	**XGI3**	**Gonio 3: crystals temperature [^{0}C]**
14337	**XDD1**	**XRD-system 1: crystal temperature [^{0}C]**
14849	**XDD2**	**XRD-system 2: crystal temperature [^{0}C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		
8194	**XGI1**	**Gonio 1: XGI +5Volts measure [V]**
10242	**XGI2**	**Gonio 2: XGI +5Volts measure [V]**
12290	**XGI3**	**Gonio 3: XGI +5Volts measure [V]**
14338	**XDD1**	**XRD-system 1: XDD +5Volts measure [V]**
14850	**XDD2**	**XRD-system 2: XDD +5Volts measure [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
8195	**XGI1**	**Gonio 1: XGI +12Volts measure [V]**
10243	**XGI2**	**Gonio 2: XGI +12Volts measure [V]**
12291	**XGI3**	**Gonio 3: XGI +12Volts measure [V]**
14339	**XDD1**	**XRD-system 1: XDD +12Volts measure [V]**
14851	**XDD2**	**XRD-system 2: XDD +12Volts measure [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		

8196	**XGI1**	**Gonio 1: XGI -12Volts measure [V]**
10244	**XGI2**	**Gonio 2: XGI -12Volts measure [V]**
12292	**XGI3**	**Gonio 3: XGI -12Volts measure [V]**
14340	**XDD1**	**XRD-system 1: XDD -12Volts measure [V]**
14852	**XDD2**	**XRD-system 2: XDD -12Volts measure [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
8197	**XGI1**	**Gonio 1: XGI +12Volts power [V]**
10245	**XGI2**	**Gonio 2: XGI +12Volts power [V]**
12293	**XGI3**	**Gonio 3: XGI +12Volts power [V]**
14341	**XDD1**	**XRD-system 1: XDD +12Volts power [V]**
14853	**XDD2**	**XRD-system 2: XDD +12Volts power [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
8198	**XGI1**	**Gonio 1: XGI +5Volts reference [V]**
10246	**XGI2**	**Gonio 2: XGI +5Volts reference [V]**
12294	**XGI3**	**Gonio 3: XGI +5Volts reference [V]**
14342	**XDD1**	**XRD-system 1: XDD +5Volts reference [V]**
14854	**XDD2**	**XRD-system 2: XDD +5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
8199	**XGI1**	**Gonio 1: XGI -5Volts reference [V]**
10247	**XGI2**	**Gonio 2: XGI -5Volts reference [V]**
12295	**XGI3**	**Gonio 3: XGI -5Volts reference [V]**
14343	**XDD1**	**XRD-system 1: XDD -5Volts reference [V]**
14855	**XDD2**	**XRD-system 2: XDD -5Volts reference [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
8200	**XGI1**	**Gonio 1: XGI +24Volts power [V]**
10248	**XGI2**	**Gonio 2: XGI +24Volts power [V]**
12296	**XGI3**	**Gonio 3: XGI +24Volts power [V]**
14344	**XDD1**	**XRD-system 1: XDD +24Volts power [V]**
14856	**XDD2**	**XRD-system 2: XDD +24Volts power [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		

8201	**XGI1**	**Gonio 1: XGI -5Volts measure [V]**
10249	**XGI2**	**Gonio 2: XGI -5Volts measure [V]**
12297	**XGI3**	**Gonio 3: XGI -5Volts measure [V]**
14345	**XDD1**	**XRD-system 1: XDD -5Volts measure [V]**
14857	**XDD2**	**XRD-system 2: XDD -5Volts measure [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
8202	**XGI1**	**Gonio 1: total working time [h]**
10250	**XGI2**	**Gonio 2: total working time [h]**
12298	**XGI3**	**Gonio 3: total working time [h]**
14346	**XDD1**	**XRD-system 1: total working time [h]**
14858	**XDD2**	**XRD-system 2: total working time [h]**
Parameters: minimum [h] (ignored) maximum working time [h] sampling period [s]		
8203	**XGI1**	**Gonio 1: total ZERO process time [h]**
10251	**XGI2**	**Gonio 2: total ZERO process time [h]**
12299	**XGI3**	**Gonio 3: total ZERO process time [h]**
14347	**XDD1**	**XRD-system 1: total ZERO process time [h]**
14859	**XDD2**	**XRD-system 2: total ZERO process time [h]**
Parameters: minimum [h] (ignored) maximum time [h] sampling period [s]		
8204	**XGI1**	**Gonio 1: total POSITION CALIBRATION process time [h]**
10252	**XGI2**	**Gonio 2: total POSITION CALIBRATION process time [h]**
12300	**XGI3**	**Gonio 3: total POSITION CALIBRATION process time [h]**
14348	**XDD1**	**XRD-system 1: total POSITION CALIBRATION process time [h]**
14860	**XDD2**	**XRD-system 2: total POSITION CALIBRATION process time [h]**
Parameters: minimum [h] (ignored) maximum time [h] sampling period [s]		
8205	**XGI1**	**Gonio 1: total HV CALIBRATION process time [h]**
10253	**XGI2**	**Gonio 2: total HV CALIBRATION process time [h]**
12301	**XGI3**	**Gonio 3: total HV CALIBRATION process time [h]**
14349	**XDD1**	**XRD-system 1: total HV CALIBRATION process time [h]**
14861	**XDD2**	**XRD-system 2: total HV CALIBRATION process time [h]**
Parameters: minimum [h] (ignored) maximum time [h] sampling period [s]		

8206	**XGI1**	**Gonio 1: total ENERGY PROFILE process time [h]**
10254	**XGI2**	**Gonio 2: total ENERGY PROFILE process time [h]**
12302	**XGI3**	**Gonio 3: total ENERGY PROFILE process time [h]**
14350	**XDD1**	**XRD-system 1: total ENERGY PROFILE process time [h]**
14862	**XDD2**	**XRD-system 2: total ENERGY PROFILE process time [h]**
Parameters: minimum [h] (ignored) maximum time [h] sampling period [s]		
8207	**XGI1**	**Gonio 1: total FAST SCAN process time [h]**
10255	**XGI2**	**Gonio 2: total FAST SCAN process time [h]**
12303	**XGI3**	**Gonio 3: total FAST SCAN process time [h]**
14351	**XDD1**	**XRD-system 1: total FAST SCAN process time [h]**
14863	**XDD2**	**XRD-system 2: total FAST SCAN process time [h]**
Parameters: minimum [h] (ignored) maximum time [h] sampling period [s]		
8208	**XGI1**	**Gonio 1: total INCREMENTAL SCAN process time [h]**
10256	**XGI2**	**Gonio 2: total INCREMENTAL SCAN process time [h]**
12304	**XGI3**	**Gonio 3: total INCREMENTAL SCAN process time [h]**
14352	**XDD1**	**XRD-system 1: total INCREMENTAL SCAN process time [h]**
14864	**XDD2**	**XRD-system 2: total INCREMENTAL SCAN process time [h]**
Parameters: minimum [h] (ignored) maximum time [h] sampling period [s]		
8209	**XGI1**	**Gonio 1: total QUANTITATIVE ANALYSIS process time [h]**
10257	**XGI2**	**Gonio 2: total QUANTITATIVE ANALYSIS process time [h]**
12305	**XGI3**	**Gonio 3: total QUANTITATIVE ANALYSIS process time [h]**
14353	**XDD1**	**XRD-system 1: total QUANTITATIVE ANALYSIS process time [h]**
14865	**XDD2**	**XRD-system 2: total QUANTITATIVE ANALYSIS process time [h]**
Parameters: minimum [h] (ignored) maximum time [h] sampling period [s]		
8210	**XGI1**	**Gonio 1: total number of crystal changes []**
10258	**XGI2**	**Gonio 2: total number of crystal changes []**
12306	**XGI3**	**Gonio 3: total number of crystal changes []**
Parameters: minimum [] (ignored) maximum changes [] sampling period [s]		
8211	**XGI1**	**Gonio 1: total number of collimator changes []**
10259	**XGI2**	**Gonio 2: total number of collimator changes []**
12307	**XGI3**	**Gonio 3: total number of collimator changes []**
Parameters: minimum [] (ignored) maximum changes [] sampling period [s]		
14356	**XDD1**	**XRD-system 1: estimated motor heating current [mA]**
14868	**XDD2**	**XRD-system 2: estimated motor heating current [mA]**

Parameters: minimum current [mA] (ignored) maximum current [mA] sampling period [s]		
9216	**XDI1**	**Gonio 1: signal software amplification gain []**
11264	**XDI2**	**Gonio 2: signal software amplification gain []**
13312	**XDI3**	**Gonio 3: signal software amplification gain []**
14592	**XDI4**	**XRD-system 1: signal software amplification gain []**
15104	**XDI5**	**XRD-system 2: signal software amplification gain []**
Parameters: minimum gain [] maximum gain [] sampling period [s]		
9217	**XDI1**	**Gonio 1: XDI +5Volts [V]**
11265	**XDI2**	**Gonio 2: XDI +5Volts [V]**
13313	**XDI3**	**Gonio 3: XDI +5Volts [V]**
14593	**XDI4**	**XRD-system 1: XDI +5Volts [V]**
15105	**XDI5**	**XRD-system 2: XDI +5Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
9218	**XDI1**	**Gonio 1: XDI +12Volts [V]**
11266	**XDI2**	**Gonio 2: XDI +12Volts [V]**
13314	**XDI3**	**Gonio 3: XDI +12Volts [V]**
14594	**XDI4**	**XRD-system 1: XDI +12Volts [V]**
15106	**XDI5**	**XRD-system 2: XDI +12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		
9219	**XDI1**	**Gonio 1: XDI -12Volts [V]**
11267	**XDI2**	**Gonio 2: XDI -12Volts [V]**
13315	**XDI3**	**Gonio 3: XDI -12Volts [V]**
14595	**XDI4**	**XRD-system 1: XDI -12Volts [V]**
15107	**XDI5**	**XRD-system 2: XDI -12Volts [V]**
Parameters: minimum voltage [V] maximum voltage [V] sampling period [s]		

<table>
<tr><td>9220</td><td>XDI1</td><td>Gonio 1: XDI +5Volts reference [V]</td></tr>
<tr><td>11268</td><td>XDI2</td><td>Gonio 2: XDI +5Volts reference [V]</td></tr>
<tr><td>13316</td><td>XDI3</td><td>Gonio 3: XDI +5Volts reference [V]</td></tr>
<tr><td>14596</td><td>XDI4</td><td>XRD-system 1: XDI +5Volts reference [V]</td></tr>
<tr><td>15108</td><td>XDI5</td><td>XRD-system 2: XDI +5Volts reference [V]</td></tr>
<tr><td colspan="3">Parameters:
minimum voltage [V]
maximum voltage [V]
sampling period [s]</td></tr>
<tr><td>9221</td><td>XDI1</td><td>Gonio 1: XDI +2.5Volts reference [V]</td></tr>
<tr><td>11269</td><td>XDI2</td><td>Gonio 2: XDI +2.5Volts reference [V]</td></tr>
<tr><td>13317</td><td>XDI3</td><td>Gonio 3: XDI +2.5Volts reference [V]</td></tr>
<tr><td>14597</td><td>XDI4</td><td>XRD-system 1: XDI +2.5Volts reference [V]</td></tr>
<tr><td>15109</td><td>XDI5</td><td>XRD-system 2: XDI +2.5Volts reference [V]</td></tr>
<tr><td colspan="3">Parameters:
minimum voltage [V]
maximum voltage [V]
sampling period [s]</td></tr>
<tr><td>9222</td><td>XDI1</td><td>Gonio 1: XDI -5Volts [V]</td></tr>
<tr><td>11270</td><td>XDI2</td><td>Gonio 2: XDI -5Volts [V]</td></tr>
<tr><td>13318</td><td>XDI3</td><td>Gonio 3: XDI -5Volts [V]</td></tr>
<tr><td>14598</td><td>XDI4</td><td>XRD-system 1: XDI -5Volts [V]</td></tr>
<tr><td>15110</td><td>XDI5</td><td>XRD-system 2: XDI -5Volts [V]</td></tr>
<tr><td colspan="3">Parameters:
minimum voltage [V]
maximum voltage [V]
sampling period [s]</td></tr>
<tr><td>9223</td><td>XDI1</td><td>Gonio 1: signal detection threshold [mV]</td></tr>
<tr><td>11271</td><td>XDI2</td><td>Gonio 2: signal detection threshold [mV]</td></tr>
<tr><td>13319</td><td>XDI3</td><td>Gonio 3: signal detection threshold [mV]</td></tr>
<tr><td>14599</td><td>XDI4</td><td>XRD-system 1: signal detection threshold [mV]</td></tr>
<tr><td>15111</td><td>XDI5</td><td>XRD-system 2: signal detection threshold [mV]</td></tr>
<tr><td colspan="3">Parameters:
minimum threshold [mV]
maximum threshold [mV]
sampling period [s]</td></tr>
<tr><td>9224</td><td>XDI1</td><td>Gonio 1: signal hardware amplification gain []</td></tr>
<tr><td>11272</td><td>XDI2</td><td>Gonio 2: signal hardware amplification gain []</td></tr>
<tr><td>13320</td><td>XDI3</td><td>Gonio 3: signal hardware amplification gain []</td></tr>
<tr><td>14600</td><td>XDI4</td><td>XRD-system 1: signal hardware amplification gain []</td></tr>
<tr><td>15112</td><td>XDI5</td><td>XRD-system 2: signal hardware amplification gain []</td></tr>
<tr><td colspan="3">Parameters:
minimum gain []
maximum gain []
sampling period [s]</td></tr>
</table>

Exit

15872	XMIxx	All mono's: XMI +5Volts [V] (for the "Set status" command ST only)
16384	XMI01	Mono 01: XMI +5Volts [V]
16896	XMI02	Mono 02: XMI +5Volts [V]
17408	XMI03	Mono 03: XMI +5Volts [V]
17920	XMI04	Mono 04: XMI +5Volts [V]
18432	XMI05	Mono 05: XMI +5Volts [V]
18944	XMI06	Mono 06: XMI +5Volts [V]
19456	XMI07	Mono 07: XMI +5Volts [V]
19968	XMI08	Mono 08: XMI +5Volts [V]
20480	XMI09	Mono 09: XMI +5Volts [V]
20992	XMI10	Mono 10: XMI +5Volts [V]
21504	XMI11	Mono 11: XMI +5Volts [V]
22016	XMI12	Mono 12: XMI +5Volts [V]
22528	XMI13	Mono 13: XMI +5Volts [V]
23040	XMI14	Mono 14: XMI +5Volts [V]
23552	XMI15	Mono 15: XMI +5Volts [V]
24064	XMI16	Mono 16: XMI +5Volts [V]
24576	XMI17	Mono 17: XMI +5Volts [V]
25088	XMI18	Mono 18: XMI +5Volts [V]
25600	XMI19	Mono 19: XMI +5Volts [V]
26112	XMI20	Mono 20: XMI +5Volts [V]
26624	XMI21	Mono 21: XMI +5Volts [V]
27136	XMI22	Mono 22: XMI +5Volts [V]
27648	XMI23	Mono 23: XMI +5Volts [V]
28160	XMI24	Mono 24: XMI +5Volts [V]
28672	XMI25	Mono 25: XMI +5Volts [V]
29184	XMI26	Mono 26: XMI +5Volts [V]
29696	XMI27	Mono 27: XMI +5Volts [V]
30208	XMI28	Mono 28: XMI +5Volts [V]
30720	XMI29	Mono 29: XMI +5Volts [V]
31232	XMI30	Mono 30: XMI +5Volts [V]
31744	XMI31	Mono 31: XMI +5Volts [V]
32256	XMI32	Mono 32: XMI +5Volts [V]

Parameters:
minimum voltage [V]
maximum voltage [V]
sampling period [s]

15873	XMIxx	**All mono's: XMI +24Volts [V] (for the "Set status" command ST only)**
16385	XMI01	**Mono 01: XMI +24Volts [V]**
16897	XMI02	**Mono 02: XMI +24Volts [V]**
17409	XMI03	**Mono 03: XMI +24Volts [V]**
17921	XMI04	**Mono 04: XMI +24Volts [V]**
18433	XMI05	**Mono 05: XMI +24Volts [V]**
18945	XMI06	**Mono 06: XMI +24Volts [V]**
19457	XMI07	**Mono 07: XMI +24Volts [V]**
19969	XMI08	**Mono 08: XMI +24Volts [V]**
20481	XMI09	**Mono 09: XMI +24Volts [V]**
20993	XMI10	**Mono 10: XMI +24Volts [V]**
21505	XMI11	**Mono 11: XMI +24Volts [V]**
22017	XMI12	**Mono 12: XMI +24Volts [V]**
22529	XMI13	**Mono 13: XMI +24Volts [V]**
23041	XMI14	**Mono 14: XMI +24Volts [V]**
23553	XMI15	**Mono 15: XMI +24Volts [V]**
24065	XMI16	**Mono 16: XMI +24Volts [V]**
24577	XMI17	**Mono 17: XMI +24Volts [V]**
25089	XMI18	**Mono 18: XMI +24Volts [V]**
25601	XMI19	**Mono 19: XMI +24Volts [V]**
26113	XMI20	**Mono 20: XMI +24Volts [V]**
26625	XMI21	**Mono 21: XMI +24Volts [V]**
27137	XMI22	**Mono 22: XMI +24Volts [V]**
27649	XMI23	**Mono 23: XMI +24Volts [V]**
28161	XMI24	**Mono 24: XMI +24Volts [V]**
28673	XMI25	**Mono 25: XMI +24Volts [V]**
29185	XMI26	**Mono 26: XMI +24Volts [V]**
29697	XMI27	**Mono 27: XMI +24Volts [V]**
30209	XMI28	**Mono 28: XMI +24Volts [V]**
30721	XMI29	**Mono 29: XMI +24Volts [V]**
31233	XMI30	**Mono 30: XMI +24Volts [V]**
31745	XMI31	**Mono 31: XMI +24Volts [V]**
32257	XMI32	**Mono 32: XMI +24Volts [V]**

Parameters:

minimum voltage [V]
maximum voltage [V]
sampling period [s]

15874	XMIxx	**All mono's: XMI +1.25Volts reference [V] (for the "Set status" cmd ST only)**
16386	XMI01	**Mono 01: XMI +1.25Volts reference [V]**
16898	XMI02	**Mono 02: XMI +1.25Volts reference [V]**
17410	XMI03	**Mono 03: XMI +1.25Volts reference [V]**
17922	XMI04	**Mono 04: XMI +1.25Volts reference [V]**
18434	XMI05	**Mono 05: XMI +1.25Volts reference [V]**
18946	XMI06	**Mono 06: XMI +1.25Volts reference [V]**
19458	XMI07	**Mono 07: XMI +1.25Volts reference [V]**
19970	XMI08	**Mono 08: XMI +1.25Volts reference [V]**
20482	XMI09	**Mono 09: XMI +1.25Volts reference [V]**
20994	XMI10	**Mono 10: XMI +1.25Volts reference [V]**
21506	XMI11	**Mono 11: XMI +1.25Volts reference [V]**
22018	XMI12	**Mono 12: XMI +1.25Volts reference [V]**
22530	XMI13	**Mono 13: XMI +1.25Volts reference [V]**
23042	XMI14	**Mono 14: XMI +1.25Volts reference [V]**
23554	XMI15	**Mono 15: XMI +1.25Volts reference [V]**
24066	XMI16	**Mono 16: XMI +1.25Volts reference [V]**
24578	XMI17	**Mono 17: XMI +1.25Volts reference [V]**
25090	XMI18	**Mono 18: XMI +1.25Volts reference [V]**
25602	XMI19	**Mono 19: XMI +1.25Volts reference [V]**
26114	XMI20	**Mono 20: XMI +1.25Volts reference [V]**
26626	XMI21	**Mono 21: XMI +1.25Volts reference [V]**
27138	XMI22	**Mono 22: XMI +1.25Volts reference [V]**
27650	XMI23	**Mono 23: XMI +1.25Volts reference [V]**
28162	XMI24	**Mono 24: XMI +1.25Volts reference [V]**
28674	XMI25	**Mono 25: XMI +1.25Volts reference [V]**
29186	XMI26	**Mono 26: XMI +1.25Volts reference [V]**
29698	XMI27	**Mono 27: XMI +1.25Volts reference [V]**
30210	XMI28	**Mono 28: XMI +1.25Volts reference [V]**
30722	XMI29	**Mono 29: XMI +1.25Volts reference [V]**
31234	XMI30	**Mono 30: XMI +1.25Volts reference [V]**
31746	XMI31	**Mono 31: XMI +1.25Volts reference [V]**
32258	XMI32	**Mono 32: XMI +1.25Volts reference [V]**

Parameters:
minimum voltage [V]
maximum voltage [V]
sampling period [s]

15875	XMIxx	**All mono's: XMI +2.5Volts reference [V] (for the "Set status" command ST only)**
16387	**XMI01**	**Mono 01: XMI +2.5Volts reference [V]**
16899	**XMI02**	**Mono 02: XMI +2.5Volts reference [V]**
17411	**XMI03**	**Mono 03: XMI +2.5Volts reference [V]**
17923	**XMI04**	**Mono 04: XMI +2.5Volts reference [V]**
18435	**XMI05**	**Mono 05: XMI +2.5Volts reference [V]**
18947	**XMI06**	**Mono 06: XMI +2.5Volts reference [V]**
19459	**XMI07**	**Mono 07: XMI +2.5Volts reference [V]**
19971	**XMI08**	**Mono 08: XMI +2.5Volts reference [V]**
20483	**XMI09**	**Mono 09: XMI +2.5Volts reference [V]**
20995	**XMI10**	**Mono 10: XMI +2.5Volts reference [V]**
21507	**XMI11**	**Mono 11: XMI +2.5Volts reference [V]**
22019	**XMI12**	**Mono 12: XMI +2.5Volts reference [V]**
22531	**XMI13**	**Mono 13: XMI +2.5Volts reference [V]**
23043	**XMI14**	**Mono 14: XMI +2.5Volts reference [V]**
23555	**XMI15**	**Mono 15: XMI +2.5Volts reference [V]**
24067	**XMI16**	**Mono 16: XMI +2.5Volts reference [V]**
24579	**XMI17**	**Mono 17: XMI +2.5Volts reference [V]**
25091	**XMI18**	**Mono 18: XMI +2.5Volts reference [V]**
25603	**XMI19**	**Mono 19: XMI +2.5Volts reference [V]**
26115	**XMI20**	**Mono 20: XMI +2.5Volts reference [V]**
26627	**XMI21**	**Mono 21: XMI +2.5Volts reference [V]**
27139	**XMI22**	**Mono 22: XMI +2.5Volts reference [V]**
27651	**XMI23**	**Mono 23: XMI +2.5Volts reference [V]**
28163	**XMI24**	**Mono 24: XMI +2.5Volts reference [V]**
28675	**XMI25**	**Mono 25: XMI +2.5Volts reference [V]**
29187	**XMI26**	**Mono 26: XMI +2.5Volts reference [V]**
29699	**XMI27**	**Mono 27: XMI +2.5Volts reference [V]**
30211	**XMI28**	**Mono 28: XMI +2.5Volts reference [V]**
30723	**XMI29**	**Mono 29: XMI +2.5Volts reference [V]**
31235	**XMI30**	**Mono 30: XMI +2.5Volts reference [V]**
31747	**XMI31**	**Mono 31: XMI +2.5Volts reference [V]**
32259	**XMI32**	**Mono 32: XMI +2.5Volts reference [V]**

Parameters:
minimum voltage [V]
maximum voltage [V]
sampling period [s]

15876	**XMIxx**	**All mono’s: XMI -5Volts [V] (for the “Set status” command ST only)**
16388	**XMI01**	**Mono 01: XMI -5Volts [V]**
16900	**XMI02**	**Mono 02: XMI -5Volts [V]**
17412	**XMI03**	**Mono 03: XMI -5Volts [V]**
17924	**XMI04**	**Mono 04: XMI -5Volts [V]**
18436	**XMI05**	**Mono 05: XMI -5Volts [V]**
18948	**XMI06**	**Mono 06: XMI -5Volts [V]**
19460	**XMI07**	**Mono 07: XMI -5Volts [V]**
19972	**XMI08**	**Mono 08: XMI -5Volts [V]**
20484	**XMI09**	**Mono 09: XMI -5Volts [V]**
20996	**XMI10**	**Mono 10: XMI -5Volts [V]**
21508	**XMI11**	**Mono 11: XMI -5Volts [V]**
22020	**XMI12**	**Mono 12: XMI -5Volts [V]**
22532	**XMI13**	**Mono 13: XMI -5Volts [V]**
23044	**XMI14**	**Mono 14: XMI -5Volts [V]**
23556	**XMI15**	**Mono 15: XMI -5Volts [V]**
24068	**XMI16**	**Mono 16: XMI -5Volts [V]**
24580	**XMI17**	**Mono 17: XMI -5Volts [V]**
25092	**XMI18**	**Mono 18: XMI -5Volts [V]**
25604	**XMI19**	**Mono 19: XMI -5Volts [V]**
26116	**XMI20**	**Mono 20: XMI -5Volts [V]**
26628	**XMI21**	**Mono 21: XMI -5Volts [V]**
27140	**XMI22**	**Mono 22: XMI -5Volts [V]**
27652	**XMI23**	**Mono 23: XMI -5Volts [V]**
28164	**XMI24**	**Mono 24: XMI -5Volts [V]**
28676	**XMI25**	**Mono 25: XMI -5Volts [V]**
29188	**XMI26**	**Mono 26: XMI -5Volts [V]**
29700	**XMI27**	**Mono 27: XMI -5Volts [V]**
30212	**XMI28**	**Mono 28: XMI -5Volts [V]**
30724	**XMI29**	**Mono 29: XMI -5Volts [V]**
31236	**XMI30**	**Mono 30: XMI -5Volts [V]**
31748	**XMI31**	**Mono 31: XMI -5Volts [V]**
32260	**XMI32**	**Mono 32: XMI -5Volts [V]**

Parameters:
minimum voltage [V]
maximum voltage [V]
sampling period [s]

Exit

15877	**XMIxx**	**All mono's: detector high-voltage [V]**
16389	**XMI01**	**Mono 01: detector high-voltage [V]**
16901	**XMI02**	**Mono 02: detector high-voltage [V]**
17413	**XMI03**	**Mono 03: detector high-voltage [V]**
17925	**XMI04**	**Mono 04: detector high-voltage [V]**
18437	**XMI05**	**Mono 05: detector high-voltage [V]**
18949	**XMI06**	**Mono 06: detector high-voltage [V]**
19461	**XMI07**	**Mono 07: detector high-voltage [V]**
19973	**XMI08**	**Mono 08: detector high-voltage [V]**
20485	**XMI09**	**Mono 09: detector high-voltage [V]**
20997	**XMI10**	**Mono 10: detector high-voltage [V]**
21509	**XMI11**	**Mono 11: detector high-voltage [V]**
22021	**XMI12**	**Mono 12: detector high-voltage [V]**
22533	**XMI13**	**Mono 13: detector high-voltage [V]**
23045	**XMI14**	**Mono 14: detector high-voltage [V]**
23557	**XMI15**	**Mono 15: detector high-voltage [V]**
24069	**XMI16**	**Mono 16: detector high-voltage [V]**
24581	**XMI17**	**Mono 17: detector high-voltage [V]**
25093	**XMI18**	**Mono 18: detector high-voltage [V]**
25605	**XMI19**	**Mono 19: detector high-voltage [V]**
26117	**XMI20**	**Mono 20: detector high-voltage [V]**
26629	**XMI21**	**Mono 21: detector high-voltage [V]**
27141	**XMI22**	**Mono 22: detector high-voltage [V]**
27653	**XMI23**	**Mono 23: detector high-voltage [V]**
28165	**XMI24**	**Mono 24: detector high-voltage [V]**
28677	**XMI25**	**Mono 25: detector high-voltage [V]**
29189	**XMI26**	**Mono 26: detector high-voltage [V]**
29701	**XMI27**	**Mono 27: detector high-voltage [V]**
30213	**XMI28**	**Mono 28: detector high-voltage [V]**
30725	**XMI29**	**Mono 29: detector high-voltage [V]**
31237	**XMI30**	**Mono 30: detector high-voltage [V]**
31749	**XMI31**	**Mono 31: detector high-voltage [V]**
32261	**XMI32**	**Mono 32: detector high-voltage [V]**
Parameters: minimum absolute voltage [V] maximum absolute voltage [V] sampling period [s] minimum relative voltage [V] maximum relative voltage [V]		

15878	XMIxx	All mono's: crystal temperature [°C] (for the "Set status" command ST only)
16390	XMI01	Mono 01: crystal temperature [°C]
16902	XMI02	Mono 02: crystal temperature [°C]
17414	XMI03	Mono 03: crystal temperature [°C]
17926	XMI04	Mono 04: crystal temperature [°C]
18438	XMI05	Mono 05: crystal temperature [°C]
18950	XMI06	Mono 06: crystal temperature [°C]
19462	XMI07	Mono 07: crystal temperature [°C]
19974	XMI08	Mono 08: crystal temperature [°C]
20486	XMI09	Mono 09: crystal temperature [°C]
20998	XMI10	Mono 10: crystal temperature [°C]
21510	XMI11	Mono 11: crystal temperature [°C]
22022	XMI12	Mono 12: crystal temperature [°C]
22534	XMI13	Mono 13: crystal temperature [°C]
23046	XMI14	Mono 14: crystal temperature [°C]
23558	XMI15	Mono 15: crystal temperature [°C]
24070	XMI16	Mono 16: crystal temperature [°C]
24582	XMI17	Mono 17: crystal temperature [°C]
25094	XMI18	Mono 18: crystal temperature [°C]
25606	XMI19	Mono 19: crystal temperature [°C]
26118	XMI20	Mono 20: crystal temperature [°C]
26630	XMI21	Mono 21: crystal temperature [°C]
27142	XMI22	Mono 22: crystal temperature [°C]
27654	XMI23	Mono 23: crystal temperature [°C]
28166	XMI24	Mono 24: crystal temperature [°C]
28678	XMI25	Mono 25: crystal temperature [°C]
29190	XMI26	Mono 26: crystal temperature [°C]
29702	XMI27	Mono 27: crystal temperature [°C]
30214	XMI28	Mono 28: crystal temperature [°C]
30726	XMI29	Mono 29: crystal temperature [°C]
31238	XMI30	Mono 30: crystal temperature [°C]
31750	XMI31	Mono 31: crystal temperature [°C]
32262	XMI32	Mono 32: crystal temperature [°C]
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		

15879	**XMIxx**	**All mono's: board temperature [°C] (for the "Set status" command ST only)**
16391	**XMI01**	**Mono 01: board temperature [°C]**
16903	**XMI02**	**Mono 02: board temperature [°C]**
17415	**XMI03**	**Mono 03: board temperature [°C]**
17927	**XMI04**	**Mono 04: board temperature [°C]**
18439	**XMI05**	**Mono 05: board temperature [°C]**
18951	**XMI06**	**Mono 06: board temperature [°C]**
19463	**XMI07**	**Mono 07: board temperature [°C]**
19975	**XMI08**	**Mono 08: board temperature [°C]**
20487	**XMI09**	**Mono 09: board temperature [°C]**
20999	**XMI10**	**Mono 10: board temperature [°C]**
21511	**XMI11**	**Mono 11: board temperature [°C]**
22023	**XMI12**	**Mono 12: board temperature [°C]**
22535	**XMI13**	**Mono 13: board temperature [°C]**
23047	**XMI14**	**Mono 14: board temperature [°C]**
23559	**XMI15**	**Mono 15: board temperature [°C]**
24071	**XMI16**	**Mono 16: board temperature [°C]**
24583	**XMI17**	**Mono 17: board temperature [°C]**
25095	**XMI18**	**Mono 18: board temperature [°C]**
25607	**XMI19**	**Mono 19: board temperature [°C]**
26119	**XMI20**	**Mono 20: board temperature [°C]**
26631	**XMI21**	**Mono 21: board temperature [°C]**
27143	**XMI22**	**Mono 22: board temperature [°C]**
27655	**XMI23**	**Mono 23: board temperature [°C]**
28167	**XMI24**	**Mono 24: board temperature [°C]**
28679	**XMI25**	**Mono 25: board temperature [°C]**
29191	**XMI26**	**Mono 26: board temperature [°C]**
29703	**XMI27**	**Mono 27: board temperature [°C]**
30215	**XMI28**	**Mono 28: board temperature [°C]**
30727	**XMI29**	**Mono 29: board temperature [°C]**
31239	**XMI30**	**Mono 30: board temperature [°C]**
31751	**XMI31**	**Mono 31: board temperature [°C]**
32263	**XMI32**	**Mono 32: board temperature [°C]**
Parameters: minimum temperature [^{0}C] maximum temperature [^{0}C] sampling period [s]		

15880	**XMIxx**	**All mono's: AGC gain [] (for the "Set status" command ST only)**
16392	**XMI01**	**Mono 01: AGC gain []**
16904	**XMI02**	**Mono 02: AGC gain []**
17416	**XMI03**	**Mono 03: AGC gain []**
17928	**XMI04**	**Mono 04: AGC gain []**
18440	**XMI05**	**Mono 05: AGC gain []**
18952	**XMI06**	**Mono 06: AGC gain []**
19464	**XMI07**	**Mono 07: AGC gain []**
19976	**XMI08**	**Mono 08: AGC gain []**
20488	**XMI09**	**Mono 09: AGC gain []**
21000	**XMI10**	**Mono 10: AGC gain []**
21512	**XMI11**	**Mono 11: AGC gain []**
22024	**XMI12**	**Mono 12: AGC gain []**
22536	**XMI13**	**Mono 13: AGC gain []**
23048	**XMI14**	**Mono 14: AGC gain []**
23560	**XMI15**	**Mono 15: AGC gain []**
24072	**XMI16**	**Mono 16: AGC gain []**
24584	**XMI17**	**Mono 17: AGC gain []**
25096	**XMI18**	**Mono 18: AGC gain []**
25608	**XMI19**	**Mono 19: AGC gain []**
26120	**XMI20**	**Mono 20: AGC gain []**
26632	**XMI21**	**Mono 21: AGC gain []**
27144	**XMI22**	**Mono 22: AGC gain []**
27656	**XMI23**	**Mono 23: AGC gain []**
28168	**XMI24**	**Mono 24: AGC gain []**
28680	**XMI25**	**Mono 25: AGC gain []**
29192	**XMI26**	**Mono 26: AGC gain []**
29704	**XMI27**	**Mono 27: AGC gain []**
30216	**XMI28**	**Mono 28: AGC gain []**
30728	**XMI29**	**Mono 29: AGC gain []**
31240	**XMI30**	**Mono 30: AGC gain []**
31752	**XMI31**	**Mono 31: AGC gain []**
32264	**XMI32**	**Mono 32: AGC gain []**
Parameters: minimum AGC gain [] maximum AGC gain [] sampling period [s]		

C

FIRMWARE STATUS

C FIRMWARE STATUS

“Firmware status” are used to retrieve from the ICS or define to the ICS, different calibration values, teaching parameters and other data. Each node of the ICS is allocated a range of “Firmware status” numbers.

The following list shows the allocated firmware status numbers allocated to the different nodes of the ICS.

Node	Firmware status range
XQI	1 - 511
XSM	512 - 1023
XGT98	1024 - 1535
XCM	1536 - 2047
XPS	2048 - 2559
XHI	2560 - 3071
XST	3072 - 3583
XSP	3584 - 4095
XGI1	8192 - 8703
XGDI1	8704 - 9215
XDI1	9216 - 9727
XGI2	10240 - 10751
XGDI2	10752 - 11263
XDI2	11264 - 11775
XGI3	12288 - 12799
XGDI3	12800 - 13311
XDI3	13312 - 13823
XDD1	14336 - 14463
XDDD1	14464 - 14591
XDI4	14592 - 14619
XDD2	14848 - 14975
XDDD2	14976 - 15103
XDI5	15104 - 15231

Node	Firmware status range
all XMI’s*	15872 - 16383
XMI01	16384 - 16895
XMI02	16896 - 17407
XMI03	17408 - 17919
XMI04	17920 - 18431
XMI05	18432 - 18943
XMI06	18944 - 19455
XMI07	19456 - 19967
XMI08	19968 - 20479
XMI09	20480 - 20991
XMI10	20992 - 21503
XMI11	21504 - 22015
XMI12	22016 - 22527
XMI13	22528 - 23039
XMI14	23040 - 23551
XMI15	23552 - 24063
XMI16	24064 - 24575
XMI17	24576 - 25087
XMI18	25088 - 25599
XMI19	25600 - 26111
XMI20	26112 - 26623
XMI21	26624 - 27135
XMI22	27136 - 27647
XMI23	27648 - 28159
XMI24	28160 - 28671
XMI25	28672 - 29183
XMI26	29184 - 29695
XMI27	29696 - 30207
XMI28	30208 - 30719
XMI29	30720 - 31231
XMI30	31232 - 31743
XMI31	31744 - 32255
XMI32	32256 - 32767

The following table describes all the valid firmware status defined in the ICS. This list may change according to the evolution of the different firmware of each node.

<table>
<tr><th>Status number</th><th>Node</th><th colspan="2">Firmware status name</th></tr>
<tr><td>1</td><td>XQI</td><td colspan="2">PC vacuum gauge calibration</td></tr>
<tr><td colspan="3">Read only : NO</td><td>Sent after initialisation : NO</td></tr>
<tr><td colspan="3">Parameters:</td><td>Type:</td></tr>
<tr><td colspan="3">Multiplicative coefficient</td><td>FLOAT</td></tr>
<tr><td colspan="3">Delta T coefficient</td><td>FLOAT</td></tr>
<tr><td>2</td><td>XQI</td><td colspan="2">SP vacuum gauge calibration</td></tr>
<tr><td colspan="3">Read only : NO</td><td>Sent after initialisation : NO</td></tr>
<tr><td colspan="3">Parameters:</td><td>Type:</td></tr>
<tr><td colspan="3">Multiplicative coefficient</td><td>FLOAT</td></tr>
<tr><td colspan="3">Delta T coefficient</td><td>FLOAT</td></tr>
<tr><td>3</td><td>XQI</td><td colspan="2">Basic firmwares versions</td></tr>
<tr><td colspan="3">Read only : YES</td><td>Sent after initialisation : NO</td></tr>
<tr><td colspan="3">Parameters:</td><td>Type:</td></tr>
<tr><td colspan="3">XQI</td><td>STRING</td></tr>
<tr><td colspan="3">XSM</td><td>STRING</td></tr>
<tr><td colspan="3">XGT98</td><td>STRING</td></tr>
<tr><td colspan="3">XCM</td><td>STRING</td></tr>
<tr><td colspan="3">XPS</td><td>STRING</td></tr>
<tr><td colspan="3">XHI</td><td>STRING</td></tr>
<tr><td colspan="3">XST</td><td>STRING</td></tr>
<tr><td colspan="3">XSP</td><td>STRING</td></tr>
<tr><td colspan="3">XMIO (contained in XQI)</td><td>STRING</td></tr>
<tr><td>4</td><td>XQI</td><td colspan="2">Goniometers firmwares versions</td></tr>
<tr><td colspan="3">Read only : YES</td><td>Sent after initialisation : NO</td></tr>
<tr><td colspan="3">Parameters:</td><td>Type:</td></tr>
<tr><td colspan="3">XGI1</td><td>STRING</td></tr>
<tr><td colspan="3">XGDI1</td><td>STRING</td></tr>
<tr><td colspan="3">XDI1</td><td>STRING</td></tr>
<tr><td colspan="3">XGI2</td><td>STRING</td></tr>
<tr><td colspan="3">XGDI2</td><td>STRING</td></tr>
<tr><td colspan="3">XDI2</td><td>STRING</td></tr>
<tr><td colspan="3">XGI3</td><td>STRING</td></tr>
<tr><td colspan="3">XGDI3</td><td>STRING</td></tr>
<tr><td colspan="3">XDI3</td><td>STRING</td></tr>
</table>

5	XQI	XRD-systems firmwares versions

Read only : YES	**Sent after initialisation :** NO
Parameters:	**Type:**
XDD1	STRING
XDDD1	STRING
XDI4	STRING
XDD2	STRING
XDDD2	STRING
XDI5	STRING

6	XQI	Monochromators 1 to 16 firmwares versions

Read only : YES	**Sent after initialisation :** NO
Parameters:	**Type:**
XMIR01	STRING
XMIR02	STRING
XMIR03	STRING
XMIR04	STRING
XMIR05	STRING
XMIR06	STRING
XMIR07	STRING
XMIR08	STRING
XMIR09	STRING
XMIR10	STRING
XMIR11	STRING
XMIR12	STRING
XMIR13	STRING
XMIR14	STRING
XMIR15	STRING
XMIR16	STRING

7	XQI	Monochromators 17 to 32 firmwares versions

Read only : YES	**Sent after initialisation :** NO
Parameters:	**Type:**
XMIR17	STRING
XMIR18	STRING
XMIR19	STRING
XMIR20	STRING
XMIR21	STRING
XMIR22	STRING
XMIR23	STRING
XMIR24	STRING
XMIR25	STRING
XMIR26	STRING
XMIR27	STRING
XMIR28	STRING
XMIR29	STRING
XMIR30	STRING
XMIR31	STRING
XMIR32	STRING

8	XQI	General instrument activity

Read only : NO

Sent after initialisation : NO

Parameters:	**Type:**
X-ray tube ON time [h]	FLOAT
X-ray tube sparks []	WORD
Molecular pump ON time [h]	FLOAT
Primary pump ON time [h]	FLOAT
Samples loaded under vacuum []	LONG

9	XQI	Instrument displays

Read only : YES

Sent after initialisation : NO

Parameters:	**Type:**
Cluster 1 (XQI/XSM)	STRING
Cluster 2 (XSN)	STRING
Cluster 3 (Goniometer 1)	STRING
Cluster 4 (Goniometer 2)	STRING
Cluster 5 (Goniometer 3)	STRING
Cluster 6 (XRD-system 1)	STRING
Cluster 7 (XRD-system 2)	STRING

10	XQI	XQI option switches 1

Read only : NO

Sent after initialisation : YES

Parameters:	**Type:**
Return global alarms (0 to 255)	WORD
Return diagnostic alarms	WORD
Return information alarms	WORD
Stop sample rotation as loaded	WORD
Maximise monochromator counting time	WORD
Not used	WORD
Not used	WORD
Ignore “NS - PA” commands	WORD

11	XQI	XQI option switches 2

Read only : NO

Sent after initialisation : YES

Parameters:	**Type:**
Not used	WORD
Allow unexpected cassette	WORD
Not used	WORD
Keep ACS analysis order	WORD
Not used	WORD
Not used	WORD
Not used	WORD
Not used	WORD

12	XQI	Sample rotation period

Read only : NO

Sent after initialisation : YES

Parameters:	**Type:**
Desired value (max 50) [s/turn]	WORD

13	XQI	**HV- vacuum analysis mode**

Read only : NO	**Sent after initialisation :** NO
Parameters:	**Type:**
State (A5A5H = defined)	STRING
Mode bit pattern	WORD
Bit 0: Wait for good pressure before start	
Bit 1: Switch OFF mono HV before chamber venting	
Bit 2: Wait for mono HV interlock to be closed	
Bit 3-15: Reserved	
Mono’s max working pressure [Pa]	
Max wait time for mono’s working pressure [s]	

14	XQI	**Mono recoverable fatal alarm occurrences**

Read only : YES	**Sent after initialisation :** NO
Parameters:	**Type:**
Report 1 (alarm, date and time)	STRING
Report 2 (alarm, date and time)	STRING
"	
Report 20 (alarm, date and time)	STRING

15	XQI	**Global monochromators analysis mode**

Read only : NO	**Sent after initialisation :** NO
Parameters:	**Type:**
State (A5A5H = defined)	STRING
Convert mono filter fatal error into warning	WORD
Reserved 2	WORD
"	
Reserved 16	WORD

17	XQI	**Regulated temperatures for ARL 9800 OASIS**

Read only : NO	**Sent after initialisation :** NO
Parameters:	**Type:**
State (A5A5H = defined)	STRING
Spectrometer tank temperature [^{0}C]	FLOAT
X-ray tube coolant temperature [^{0}C]	FLOAT
Mono’s crystals temperature [^{0}C]	FLOAT
Goniometers crystals temperature [^{0}C]	FLOAT
XRD-systems crystals temperature [^{0}C]	FLOAT

18	XQI	**Regulated temperatures for ARL 9800**

Read only : NO	**Sent after initialisation :** NO
Parameters:	**Type:**
State (A5A5H = defined)	STRING
Spectrometer tank temperature [^{0}C]	FLOAT
X-ray tube coolant temperature [^{0}C]	FLOAT
Mono’s crystals temperature [^{0}C]	FLOAT
Goniometers crystals temperature [^{0}C]	FLOAT
XRD-systems crystals temperature [^{0}C]	FLOAT

Exit

21	**XQI**	**Automatic PBC selection scan on XRD-system 1**
22	**XQI**	**Automatic PBC selection scan on XRD-system 2**

Read only : NO **Sent after initialisation :** NO

Parameters:	**Type:**
Automatic PBC selection (0: disable, <>0: enable)	INTEGER
Low 2θ boundary for PBC1 [^{0}]	FLOAT
High 2θ boundary for PBC1 [^{0}]	FLOAT
Low 2θ boundary for PBC2 [^{0}]	FLOAT
High 2θ boundary for PBC2 [^{0}]	FLOAT
Low 2θ boundary for PBC3 [^{0}]	FLOAT
High 2θ boundary for PBC3 [^{0}]	FLOAT
Low 2θ boundary for PBC4 [^{0}]	FLOAT
High 2θ boundary for PBC4 [^{0}]	FLOAT
Transition 2θ range for PBC1 [^{0}]	FLOAT
Transition coefficient for PBC1 []	FLOAT
Transition 2θ range for PBC2 [^{0}]	FLOAT
Transition coefficient for PBC2 []	FLOAT
Transition 2θ range for PBC3 [^{0}]	FLOAT
Transition coefficient for PBC3 []	FLOAT
Transition 2θ range for PBC4 [^{0}]	FLOAT
Transition coefficient for PBC4 []	FLOAT

23	**XQI**	**XGN network baudrate selection**

Read only : NO **Sent after initialisation :** YES

Parameters:	**Type:**
Desired XGN baudrate (default 9600) [baud]	WORD

511	**XQI**	**Terminal mode command**

Read only : NO **Sent after initialisation :** NO

Parameters:	**Type:**
Command string	STRING

2048	**XPS**	**kV/mA measurement calibration**

Read only : NO **Sent after initialisation :** NO

Parameters:	**Type:**
kV reading offset	FLOAT
kV reading slope	FLOAT
mA reading offset	FLOAT
mA reading slope	FLOAT

2049	**XPSLP**	**Low power temperature offset**

Read only : NO **Sent after initialisation :** NO

Parameters:	**Type:**
State (A5A5H = defined)	STRING
Laboratory temperature offset [^{0}]	FLOAT
X-ray power supply temperature offset [^{0}]	FLOAT
Secondary water input temperature offset [^{0}]	FLOAT
Secondary water output temperature offset [^{0}]	FLOAT
XPS power stage temperature offset [^{0}]	FLOAT

2560	**XHI**	**XHI load position teaching**
2561	**XHI**	**XHI tray 0 teaching**
2562	**XHI**	**XHI tray 100 teaching**
2563	**XHI**	**XHI belt 1 teaching**
2564	**XHI**	**XHI belt 2 teaching**
2565	**XHI**	**XHI waiting position 1 teaching**
2566	**XHI**	**XHI waiting position 2 teaching**
2567	**XHI**	**XHI basket 1 teaching**
2568	**XHI**	**XHI basket 2 teaching**
2569	**XHI**	**XHI basket 3 teaching**

Read only : NO	**Sent after initialisation :** NO
Parameters:	**Type:**
State (A5A5H = defined)	STRING
Offset X	LONG
Offset Y	LONG
Offset Z	LONG
Slope X	LONG
Slope Y	LONG
Arm slope Y	LONG

2570	**XHI**	**XHI X-axis parameters**
2571	**XHI**	**XHI Y-axis parameters**
2572	**XHI**	**XHI Z1-axis parameters**
2573	**XHI**	**XHI Z2-axis parameters**

Read only : NO	**Sent after initialisation :** NO
Parameters:	**Type:**
State (A5A5H = defined)	STRING
Max static error	LONG
Kp	LONG
Kd	LONG
Ki	INTEGER
Cruise speed	WORD
Acceleration	WORD
Max zero error	LONG
Zero speed	WORD
Max range	LONG

2574	**XHI**	**XHI sucker position teaching**

Read only : NO	**Sent after initialisation :** NO
Parameters:	**Type:**
State (A5A5H = defined)	STRING
Offset X	LONG
Offset Y	LONG
Offset Z	LONG
Slope X	LONG
Slope Y	LONG
Arm slope Y	LONG

Exit

3072	**XHI**	**Shutter axis parameters**
3073	**XHI**	**Transfer axis parameters**
3074	**XHI**	**Load lift axis parameters**
3075	**XHI**	**Analysis axis parameters**
3076	**XHI**	**Sample rotation axis parameters**

Read only : NO
Sent after initialisation : NO

Parameters:	**Type:**
State (A5A5H = defined)	STRING
Max static error	LONG
Kp	LONG
Kd	LONG
Ki	INTEGER
Cruise speed	WORD
Acceleration	WORD
Max zero error	LONG
Zero speed	WORD
Max range	LONG

3077	**XHI**	**Load lift teaching**

Read only : NO
Sent after initialisation : NO

Parameters:	**Type:**
State (A5A5H = defined)	STRING
Loading position height [step]	LONG
Sample height detector position [step]	LONG

3078	**XHI**	**Analysis lift teaching**

Read only : NO
Sent after initialisation : NO

Parameters:	**Type:**
State (A5A5H = defined)	STRING
Analysis position height [step]	LONG

3079	**XHI**	**Transfer teaching**

Read only : NO
Sent after initialisation : NO

Parameters:	**Type:**
State (A5A5H = defined)	STRING
Loading side bumper position [step]	LONG
Analysis side bumper position [step]	LONG

16384	**XMI01**	**High-voltage measurement calibration on mono01**
16896	**XMI02**	**High-voltage measurement calibration on mono02**
17408	**XMI03**	**High-voltage measurement calibration on mono03**
17920	**XMI04**	**High-voltage measurement calibration on mono04**
18432	**XMI05**	**High-voltage measurement calibration on mono05**
18944	**XMI06**	**High-voltage measurement calibration on mono06**
19456	**XMI07**	**High-voltage measurement calibration on mono07**
19968	**XMI08**	**High-voltage measurement calibration on mono08**
20480	**XMI09**	**High-voltage measurement calibration on mono09**
20992	**XMI10**	**High-voltage measurement calibration on mono10**
21504	**XMI11**	**High-voltage measurement calibration on mono11**
22016	**XMI12**	**High-voltage measurement calibration on mono12**
22528	**XMI13**	**High-voltage measurement calibration on mono13**
23040	**XMI14**	**High-voltage measurement calibration on mono14**
23552	**XMI15**	**High-voltage measurement calibration on mono15**
24064	**XMI16**	**High-voltage measurement calibration on mono16**
24576	**XMI17**	**High-voltage measurement calibration on mono17**
25088	**XMI18**	**High-voltage measurement calibration on mono18**
25600	**XMI19**	**High-voltage measurement calibration on mono19**
26112	**XMI20**	**High-voltage measurement calibration on mono20**
26624	**XMI21**	**High-voltage measurement calibration on mono21**
27136	**XMI22**	**High-voltage measurement calibration on mono22**
27648	**XMI23**	**High-voltage measurement calibration on mono23**
28160	**XMI24**	**High-voltage measurement calibration on mono24**
28672	**XMI25**	**High-voltage measurement calibration on mono25**
29184	**XMI26**	**High-voltage measurement calibration on mono26**
29696	**XMI27**	**High-voltage measurement calibration on mono27**
30208	**XMI28**	**High-voltage measurement calibration on mono28**
30720	**XMI29**	**High-voltage measurement calibration on mono29**
31232	**XMI30**	**High-voltage measurement calibration on mono30**
31744	**XMI31**	**High-voltage measurement calibration on mono31**
32256	**XMI32**	**High-voltage measurement calibration on mono32**

Read only : NO

Sent after initialisation : NO

Parameters:	**Type:**
Offset	FLOAT
Slope	FLOAT

16385	**XMI01**	**Crystal temperature calibration on mono01**
16897	**XMI02**	**Crystal temperature calibration on mono02**
17409	**XMI03**	**Crystal temperature calibration on mono03**
17921	**XMI04**	**Crystal temperature calibration on mono04**
18433	**XMI05**	**Crystal temperature calibration on mono05**
18945	**XMI06**	**Crystal temperature calibration on mono06**
19457	**XMI07**	**Crystal temperature calibration on mono07**
19969	**XMI08**	**Crystal temperature calibration on mono08**
20481	**XMI09**	**Crystal temperature calibration on mono09**
20993	**XMI10**	**Crystal temperature calibration on mono10**
21505	**XMI11**	**Crystal temperature calibration on mono11**
22017	**XMI12**	**Crystal temperature calibration on mono12**
22529	**XMI13**	**Crystal temperature calibration on mono13**
23041	**XMI14**	**Crystal temperature calibration on mono14**
23553	**XMI15**	**Crystal temperature calibration on mono15**
24065	**XMI16**	**Crystal temperature calibration on mono16**
24577	**XMI17**	**Crystal temperature calibration on mono17**
25089	**XMI18**	**Crystal temperature calibration on mono18**
25601	**XMI19**	**Crystal temperature calibration on mono19**
26113	**XMI20**	**Crystal temperature calibration on mono20**
26625	**XMI21**	**Crystal temperature calibration on mono21**
27137	**XMI22**	**Crystal temperature calibration on mono22**
27649	**XMI23**	**Crystal temperature calibration on mono23**
28161	**XMI24**	**Crystal temperature calibration on mono24**
28673	**XMI25**	**Crystal temperature calibration on mono25**
29185	**XMI26**	**Crystal temperature calibration on mono26**
29697	**XMI27**	**Crystal temperature calibration on mono27**
30209	**XMI28**	**Crystal temperature calibration on mono28**
30721	**XMI29**	**Crystal temperature calibration on mono29**
31233	**XMI30**	**Crystal temperature calibration on mono30**
31745	**XMI31**	**Crystal temperature calibration on mono31**
32257	**XMI32**	**Crystal temperature calibration on mono32**

Read only : NO	**Sent after initialisation :** NO
Parameters:	**Type:**
Regulation enabled	WORD
Desired regulation temperature	FLOAT
Offset	FLOAT
Slope	FLOAT

16386	**XMI01**	**Board temperature calibration on mono01**
16898	**XMI02**	**Board temperature calibration on mono02**
17410	**XMI03**	**Board temperature calibration on mono03**
17922	**XMI04**	**Board temperature calibration on mono04**
18434	**XMI05**	**Board temperature calibration on mono05**
18946	**XMI06**	**Board temperature calibration on mono06**
19458	**XMI07**	**Board temperature calibration on mono07**
19970	**XMI08**	**Board temperature calibration on mono08**
20482	**XMI09**	**Board temperature calibration on mono09**
20994	**XMI10**	**Board temperature calibration on mono10**
21506	**XMI11**	**Board temperature calibration on mono11**
22018	**XMI12**	**Board temperature calibration on mono12**
22530	**XMI13**	**Board temperature calibration on mono13**
23042	**XMI14**	**Board temperature calibration on mono14**
23554	**XMI15**	**Board temperature calibration on mono15**
24066	**XMI16**	**Board temperature calibration on mono16**
24578	**XMI17**	**Board temperature calibration on mono17**
25090	**XMI18**	**Board temperature calibration on mono18**
25602	**XMI19**	**Board temperature calibration on mono19**
26114	**XMI20**	**Board temperature calibration on mono20**
26626	**XMI21**	**Board temperature calibration on mono21**
27138	**XMI22**	**Board temperature calibration on mono22**
27650	**XMI23**	**Board temperature calibration on mono23**
28162	**XMI24**	**Board temperature calibration on mono24**
28674	**XMI25**	**Board temperature calibration on mono25**
29186	**XMI26**	**Board temperature calibration on mono26**
29698	**XMI27**	**Board temperature calibration on mono27**
30210	**XMI28**	**Board temperature calibration on mono28**
30722	**XMI29**	**Board temperature calibration on mono29**
31234	**XMI30**	**Board temperature calibration on mono30**
31746	**XMI31**	**Board temperature calibration on mono31**
32258	**XMI32**	**Board temperature calibration on mono32**

Read only : NO **Sent after initialisation :** NO

Parameters:	**Type:**
Offset	FLOAT
Slope	FLOAT

Exit

16387	**XMI01**	**XMI options on mono01**
16899	**XMI02**	**XMI options on mono02**
17411	**XMI03**	**XMI options on mono03**
17923	**XMI04**	**XMI options on mono04**
18435	**XMI05**	**XMI options on mono05**
18947	**XMI06**	**XMI options on mono06**
19459	**XMI07**	**XMI options on mono07**
19971	**XMI08**	**XMI options on mono08**
20483	**XMI09**	**XMI options on mono09**
20995	**XMI10**	**XMI options on mono10**
21507	**XMI11**	**XMI options on mono11**
22019	**XMI12**	**XMI options on mono12**
22531	**XMI13**	**XMI options on mono13**
23043	**XMI14**	**XMI options on mono14**
23555	**XMI15**	**XMI options on mono15**
24067	**XMI16**	**XMI options on mono16**
24579	**XMI17**	**XMI options on mono17**
25091	**XMI18**	**XMI options on mono18**
25603	**XMI19**	**XMI options on mono19**
26115	**XMI20**	**XMI options on mono20**
26627	**XMI21**	**XMI options on mono21**
27139	**XMI22**	**XMI options on mono22**
27651	**XMI23**	**XMI options on mono23**
28163	**XMI24**	**XMI options on mono24**
28675	**XMI25**	**XMI options on mono25**
29187	**XMI26**	**XMI options on mono26**
29699	**XMI27**	**XMI options on mono27**
30211	**XMI28**	**XMI options on mono28**
30723	**XMI29**	**XMI options on mono29**
31235	**XMI30**	**XMI options on mono30**
31747	**XMI31**	**XMI options on mono31**
32259	**XMI32**	**XMI options on mono32**

Read only : NO

Sent after initialisation : NO

Parameters:	**Type:**
Keep HV ON on DAC overflow	WORD
Keep HV ON on value overflow	WORD
Intensity filter type (if configured)	WORD
0 : primary slit	
1 : secondary slit	
Intensity filter sensor (if configured)	WORD
0 : enabled	
1 : disabled	
Reserved 5	WORD
"	
Reserved 16	WORD

Exit

D

FREE LIME

D FREE LIME

FREE LIME CALIBRATION PROCEDURE

1. It is preferable to do the free lime calibration (by XRD channel) at the customer site after successful installation for various reasons: There are no commercial free lime standards available (because free lime is not stable in air). The samples for free lime calibration have to be obtained from the chemist or analyst responsible on site. The nominal values of free lime concentrations are obtained by wet chemical method. All the cement plants have this facility and this work should be undertaken during installation and subsequent calibration together with the plant chemist.

2. The plant or chief chemist responsible for the analytical laboratory should be contacted sufficiently in advance (before installation) to collect free lime samples, as it may be difficult to collect clinker samples with varying free lime concentrations during the week of installation . Normally the free lime is not expected to vary during a short term if the kiln conditions are met. At least 5 clinker samples in granular form (not finely ground powders) of about 100g each should be collected. The samples must have sufficiently varying free lime concentrations, say between 0.5 - 5% in order to achieve a representative calibration. If you do not manage to get such variations, you can try for a smaller range say 0.5-3%. It depends on the kiln conditions.

3. Part of each clinker sample should be used by the chemist for his/her wet chemical analysis (titration in ethylene glycol) and request for reasonably accurate analysis. Remember the problems of reproducibility and subjective variations from one chemist to another.

4. Grind sample #1 (preferably the one with highest free lime concentration) for say 10s to start with. At this stage, any grinding agents (like Herzog tablets or any other product for better grindability and homogeneity) should be added depending on the customer’s usual procedures. If such binding agents are usually used then try to use the same tablets or agents for grinding in a proportion say 15g sample + 2 tablets of 200mg.

5. Take about 5-7g of the ground sample to press a pellet at 20tons, 20s.

6. Take the pellet immediately to the instrument (TCA) and do a scan using XRD.SCP (Run scan as usual). Locate the free lime (CaO) peak around 2.405Å on a 2 Theta scale. The peak is usually around 130deg. Note the exact peak position for later use (to feed into the analytical program for calibration and analysis).

7. Go back to grinding machine and grind the remaining clinker powder for an additional 10s (making the total grinding time as 20s so far).

8. Take 5-7g and press a pellet again at 20tons, 20s.

9. Do the same scan as in step 6 and compare with the previous scan after 10s grinding time. Notice if there is any increase in the peak intensity, background, line width etc. If the overall peak intensity has improved, then there is a good reason to continue further grinding.

10. Go back to the grinding machine, grind for additional 20s (total of 40s so far), take part of the sample, press it and do a scan. If the peak intensity now starts dropping and the peak broadens (line width increases), then stop here and use the optimum grinding time found for all the other samples.

11. Grind all the other clinker granules to the same particle size (same optimum grinding time found above), press them under the same conditions and you are now ready for calibration.

12. Although the above conditions for grinding and pelletization have been recommended for free lime as a separate measurement, it is important to take into account the grinding effects on the corresponding

XRF results as well. In general, one checks for the CaKα, SiKα, AlKα intensities measured by XRF at the same time in addition to the intensity of free CaO peak measured by XRD. It is necessary, in many cases, to grind longer than 40s (up to 3 minutes sometimes) to get reproducible and reliable results in XRF due to high amounts of silica. Free lime peak is slightly effected when the grinding time exceeds more than 2-3 minutes but nothing serious happens. Its intensity can be still used for calibration.

In conclusion, the grinding conditions for clinker analysis which includes free lime in addition to the usual XRF analysis must be optimised as a function of standard grinding time used for XRF.

13. Calibration of free lime is done exactly the same way as you would normally do for an XRF channel i.e. go to the preparation menu, create an analytical program, add CaO line from XRD line library and any other lines from XRF library if necessary. Introduce the correct 2 Theta angle noted above for the CaO (XRD) in the analytical program (Elements section). The X-ray tube conditions are the same as you would use the XRF part (say 30kV/80mA). Do not forget to put PBC as 1 for all the XRD lines. Use 100s counting time for free lime. While you are in the program screen with all the elements to be measured, notice that the XRD lines can be measured in three different modes:

Peak	**Peak Search**	**Peak Integration**

Peak ----- Measures the peak intensity (no background subtraction, no corrections if any peak shifts occur). In most cases, direct peak intensity measured this way is good enough for calibrations.

Peak Search---- It might happen in a few cases that the free lime peak position is slightly different from sample to sample. Normally the free lime peak does not shift as long as the samples come from the same kiln and clinkerisation conditions. However, there are cases where samples from different kilns are measured without specific identification. The matrix effects may be slightly different from sample to sample causing small peak shifts. In this case, you can activate the Peak Search instead of Peak and the system will first locate the peak maximum automatically and then count on the peak.

Peak Integration------ Some of the customers who are used to a conventional XRD instrument may ask you if we could integrate over the peak rather than measure on the peak. Peak integration is very useful and necessary if the backgrounds, peak shapes and peak positions keep changing all the time and one does not want to correct the analytical program for every sample. This function is available in a conventional XRD software and we have included it in our soft also in case the need arises. However, we have not seen any real need for this function at least in the case of free lime.

To conclude, both **Peak Search** and **Peak Integration** functions are to be used only when there is a need (uncontrollable peak shifts, background fluctuations and different kiln conditions and products) and Peak intensity measurement is sufficient in most cases.

14. For calibration purposes, introduce all the samples and their free lime concentrations as you would normally do for an XRF calibration. Create the task and do the calibration.

15. The calibration results (particularly the standard error of estimate) show how the wet chemical values correlate with the XRD intensities. If you find couple of samples out of the acceptable error margins, then either the wet chemical analysis is wrong (or has a bigger error) or the sample preparation has not produced a homogeneous representative sample. The wet chemical analysis should be repeated for verification, or another clinker sample should be used to substitute or the sample preparation should be repeated. Usually, a standard error of estimate (SEE) of less than 0.2% is easily achieved.

FREE LIME RECALIBRATION

Please find below the preparation and analytical method to be used for the drift correction/re-calibration procedure for the free lime XRD channel in Total Cement Analyser. This special procedure has to be adopted for the recalibration of free lime channel because free lime is not stable and we can not keep a clinker sample with free lime long enough to be used as an SUS sample. Instead, NiO is used to simulate the free lime peak in the proximity and since NiO is a stable phase, the following procedure assures a reliable method.

A. High point

- Mix 20 g of raw meal with 2 g of green NiO along with 5 Herzog grinding pills
- (Green NiO is a product of Alpha/Johnson Matthey #89773)
- Grind the mixture for 10s in a Herzog grinding machine
- Press the powder into a pellet at 40 tons, 60 s

B. Low point

- The same as above without NiO

C. Analytical Program

So you now have 2 Theta real for NiO and 2 Theta real for CaO.

- Introduce 2 Theta real for CaO in the analytical program used for the calibration and analysis of free lime in clinkers.
- Modify the 2 Theta real of CaO in the XRD line library by introducing the 2 Theta real of NiO.

Thus when recalibration of free lime has to be done, the high point and low point samples described above are measured at the NiO 2 Theta real and used for the drift correction. On the other hand, when the analysis of free lime has to be done (in the production control), the CaO 2 Theta real entered in the analytical program is used.

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