Optical Emission Spectrometry

OES

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- Introduction
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- Instrument preparation
- Samples preparation
- Analysis
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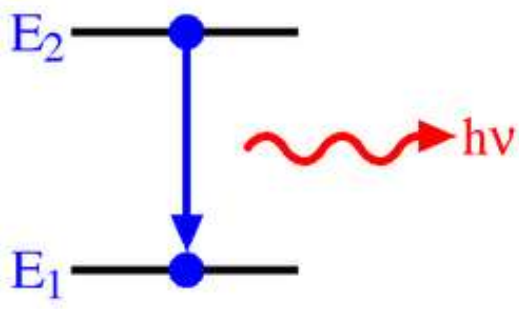
INTRODUCTION

Optical Emission Spectrometer

OES **is the reference analysis technique for elemental analysis of solid metallic samples** . that uses the light emitted of an excited element and (PMT) convert the light in an electrical signal. that can be read by the instrument computer and the software



- Stability, precision, low detection limits, and accuracy
- Configured and calibrated in the factory
- Continuous up-grade possibilities
- Easy integration to increase productivity

ARL 3460 Advantage

The ARL 3460 Advantage

- Superior stability, reliability, repeatability and long lifetime with lowest running and maintenance costs.
- It has been designed to address the analytical needs of foundries and metal processing industries dealing with ferrous , and nonferrous products.
- The ARL 3460 Advantage is factory calibrated for most accurate and uncompromising analytical performance .

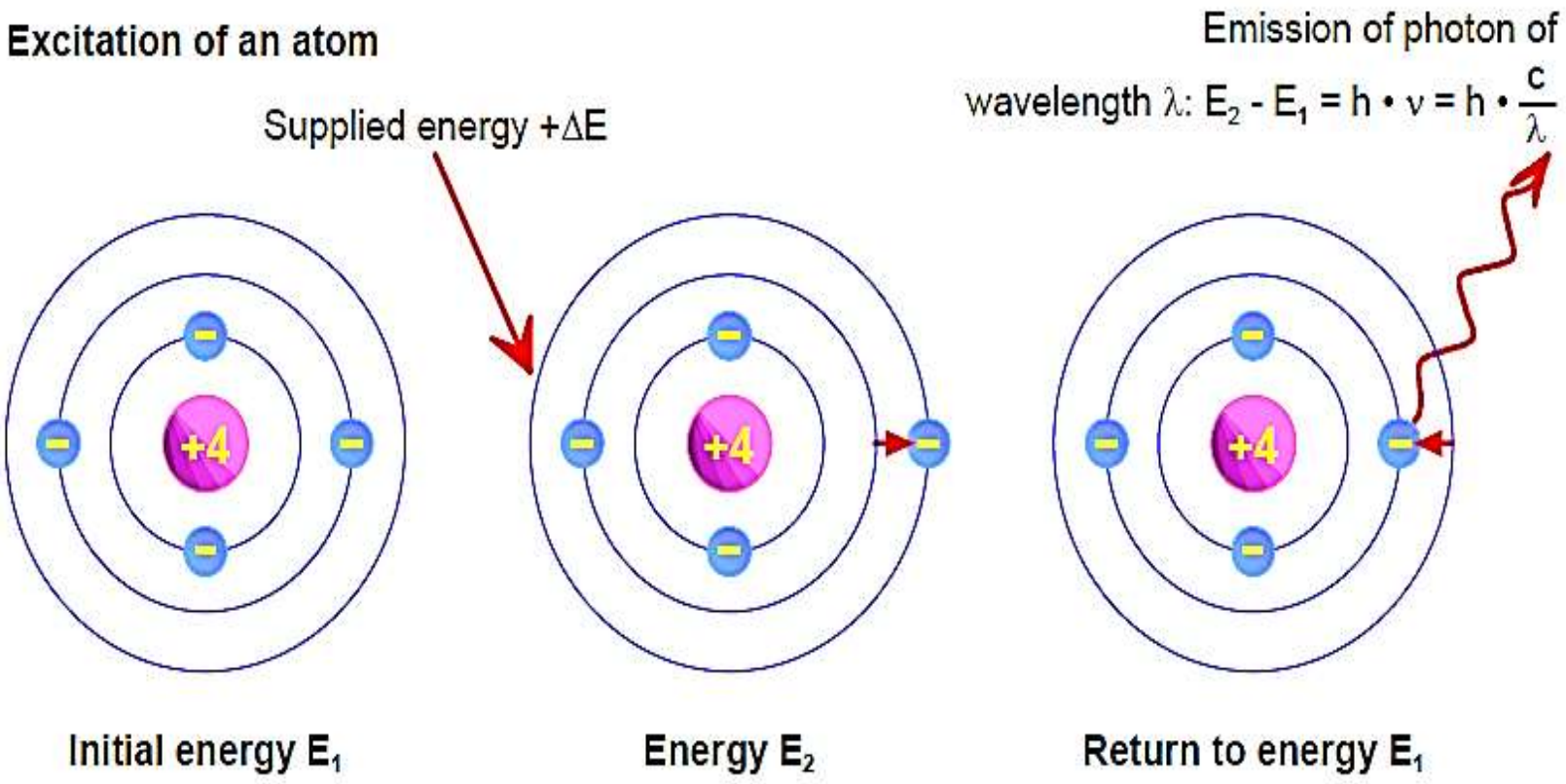
Fast and Accurate

As a multi-channel optical emission spectrometer, ARL 3460 is designed for fast, accurate metals analysis in:

- Primary metal producing plants
- Foundries, forges, mini-mills
- Casting operations
- Incoming material control
- Metals QC and R&D laboratories

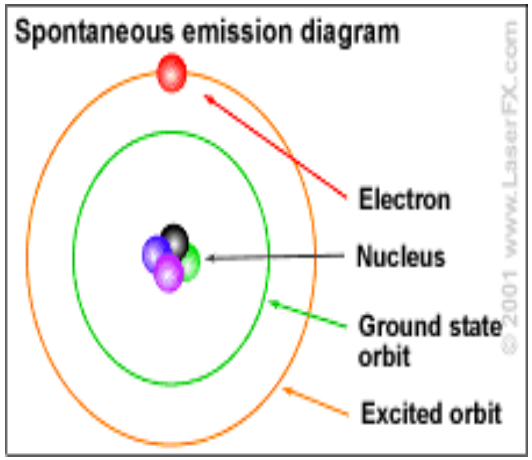
THEORY

Emission


Excitation of an atom
Supplied energy +ΔE
Emission of photon of wavelength λ: $E_2 - E_1 = h \cdot \nu = h \cdot \frac{c}{\lambda}$
+4
+4
+4
Initial energy E_1
Energy E_2
Return to energy E_1

Emission

1. Electrons in the element are **excited**.
2. They **jump** to higher energy levels (excited state).
3. As the electrons **fall back** down (ground state).
4. **Emitted**(photon), the wavelength of which refers to the discrete lines of the emission spectrum.



The emission spectrum can be used to determine the **composition** of a material

OES Theory

- A sample containing several different elements will therefore produce light composed of wavelength specific to each of the elements.
- By separating these wavelengths by a dispersion system, the spectrometer can determine which elements are present.
- The intensity of each of these wavelengths being a function of the concentration of the considered element.
- Measure this luminous intensity (with a photomultiplier) and process this information with a computer.
- The instrument can thus determine the concentration of the considered element

Analysis Steps

1. After an adequate preparation the sample is positioned on the stand's analytical table.
2. A generator "source" generates cyclic sparks between the electrode and the sample's surface.
3. The ionized atoms extracted in that way emit the characteristic light to be analysed.
4. The instrument converts the light emitted by the discharge in an electrical current .
5. The computer acquires these final values and calculates then the concentrations by elements.

Instrument Configurations

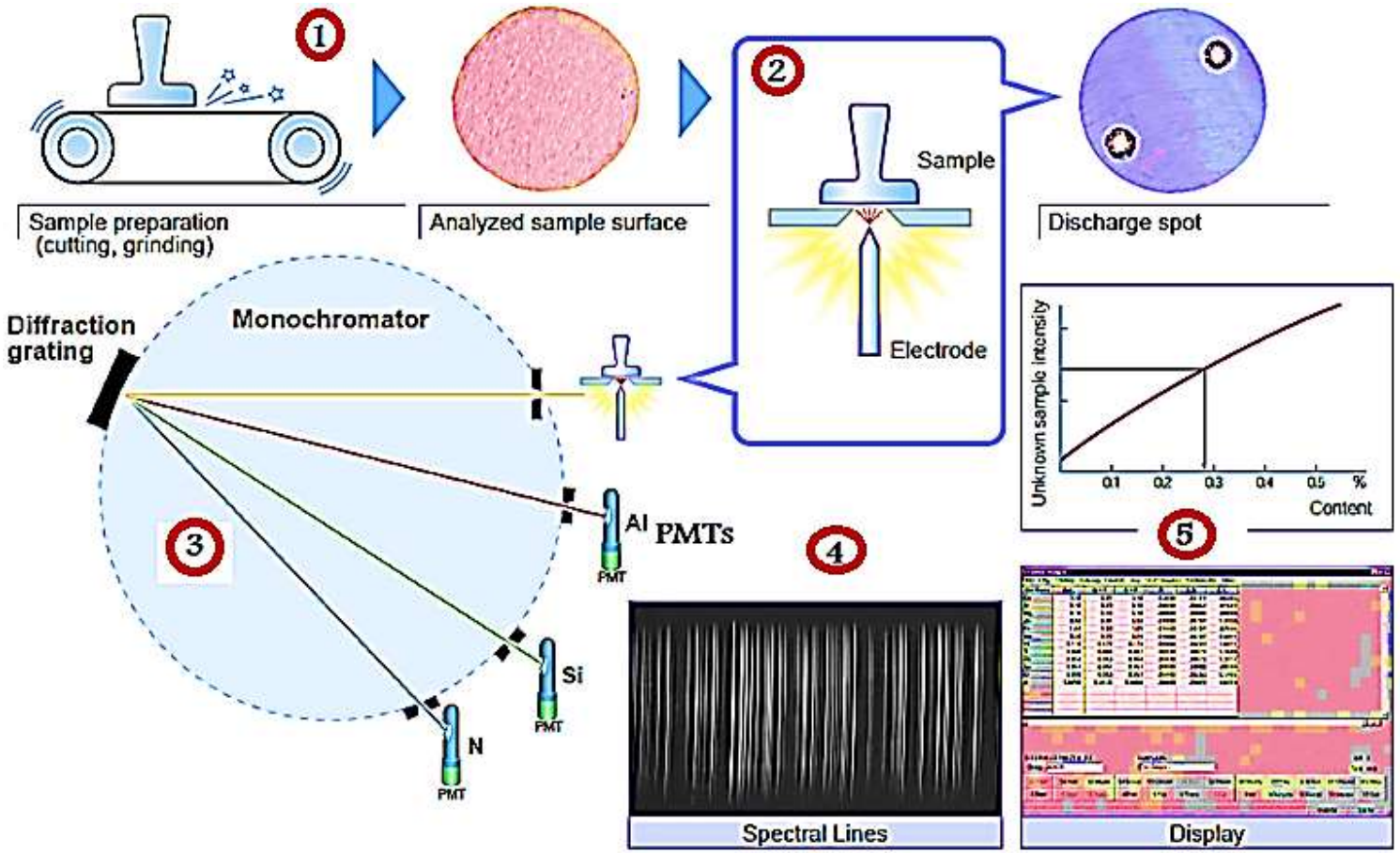
Each instrument is different to another one of the **same model**,

- The **analytical tasks** (the same for the attenuator also)
- The **spectral line** used for a chemical element.
- the **sensitivity of the (PMT)** photomultiplier used for this line.

  The sign -1 corresponds to the less sensitive photomultiplier and the -3 sign to the most sensitive photomultiplier.
- Depending of the type of **grating** installed.
- If the measured line in a higher order, selecting **filters** are placed in front of the photomultiplier.

OPTICAL EMISSION SETUP

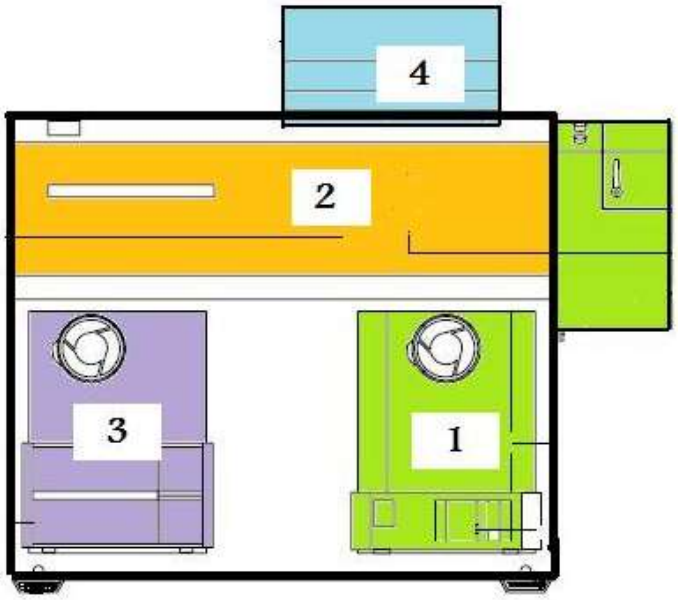
Work Steps


1
Sample preparation
(cutting, grinding)
Analyzed sample surface
2
Sample
Electrode
Discharge spot
Diffraction
grating
Monochromator
3
Al
PMTs
PMT
Si
PMT
N
PMT
4
Spectral Lines
Unknown sample intensity
0.1
0.2
0.3
0.4
0.5
%
Content
5
Display

Instrument

An instrument is therefore composed of the four following parts :

- ***A source*** of excitation which supplies energy to the samples.
- ***Oven and Spectrometer*** which discriminates the different wavelengths.
- ***Electronics*** which measure the luminous intensity of each of the wavelengths.
- ***A computer*** which processes the measurements and controls the instrument.



1. The Excitation

The excitation is composed by ***two distinct*** parts:

- The source (or generator).
- The stand.

The Source (Generator)

- Source is The electronic device that provides the high voltage spark or other electronic energy to excite the sample on the stand.

Called Hi-Rep 2+,

- The analysis is done by creating a low voltage arc between the surface of the sample to be analyzed and a counter electrode.
- A classical source of the RLC discharge type and its frequency can rise up to 400 Hz.

Spark

- The high voltage applied to a sample causes a spark, the emitted light of which can be used to determine the concentrations of the element within the sample.

The Stand

- The purpose of the stand is to ***place*** the sample in a reproducible way relatively to the optical device located into the spectrometer.
- The stand therefore constitutes an enclosure in which an ***argon*** flow circulates The stand-by flow is fixed to about 0.2 - 0.5 l/min.
- During sparking, this flow is automatically ***increased***.
- The table is cooled by a closed ***water*** cooled circuit.

The Stand

- During an analysis, two types of sparks are usually created.

  a high energy spark is produced in order to prepare (melt and homogenise) the sample surface. Then ***a lower energy*** spark is produced, and the light emitted at this time is measured.

- The ***shutter*** is opened during the measuring phase by an electromagnetic valve.

- It is not possible to perform an analysis when the stand ***door*** is open.

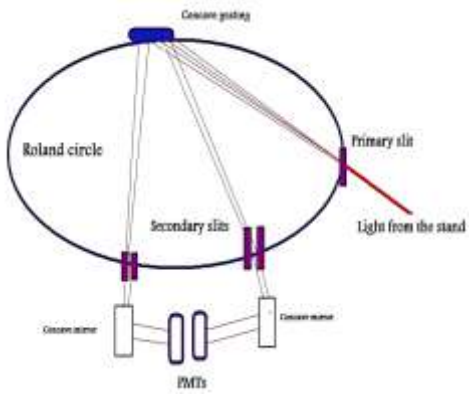
2. Oven and Spectrometer

- The spectrometer is the mechanical ***housing*** that contains the optical dispersion system.
- The spectrometer itself is placed into a controlled thermo insulated cabinet called ***oven*** (at 38 ± 0.1 °C) in order to avoid any deformation by dilatation.
- The light path into the spectrometer is approximately 2 meters long. As air will absorb ultraviolet light, it is necessary to put the spectrometer under ***vacuum*** to avoid this effect.

The Optical System

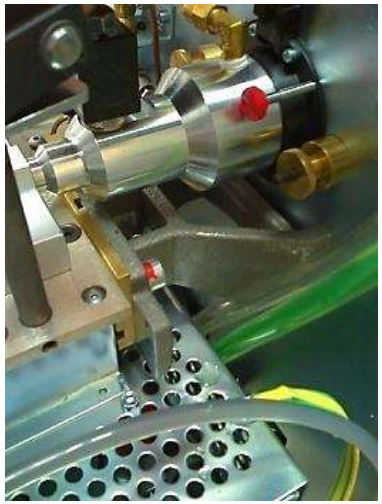
The optical system is composed of five main parts:

1. ***The primary slit***, It has 20 µm width
2. ***The grating***, it diffracts the light and focuses it on the secondary slits.
3. ***The secondary slits***, only narrow wavelength bands to pass.
4. ***The mirrors*** The light is reflected by a mirror that focuses on the PMT.
5. ***PMT*** the photomultipliers tubes



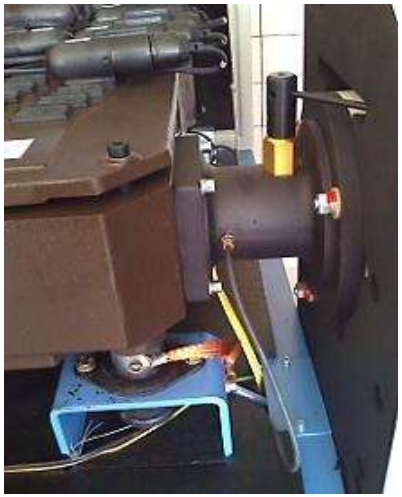
Primary Lens

- The primary lens is made of calcium fluoride (***CaF2***).
- The lens is assembled in a support called lens ***holder*** .
- It ***Heated*** to reduces any deposits coming from the argon circuit or from the vacuum side of the spectrometer .
- The ***separation*** between the spectrometer under vacuum and the stand under argon is made by an airtight lens, which can be removed for cleaning without breaking the spectrometer vacuum.



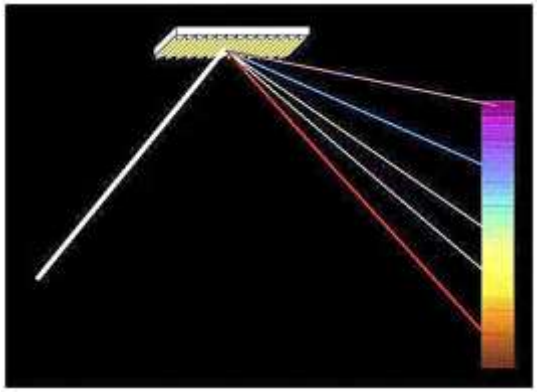
The Primary Slit

- Is to be considered as the object in terms of optical geometry, each spectral line is an image of that slit.
- It has 20 µm width.
- The grating has a dual feature: it diffracts the light and focuses it on the secondary slits.



The Grating

- Grating An optical device within the spectrometer used to separate the emitted light into its component wavelengths.
- The grating has a ***dual*** feature: it diffracts the light and focuses it on the secondary slits.
- The grating is the ***main*** optic part of the spectrometer;
- It ***separates*** the spark's light into all the wavelength that composes it.
- The grating curvature is ***aligned*** on this circle.
- There is no routine ***maintenance*** operation requiring that you handle it or even approach it.
- There are ***two types*** of gratings 1667 or 1080 grooves/mm.



Diffraction Grating

- The grating undergoes a surface treatment of magnesium fluoride, to improves its refection power for short wavelengths.
- It is not necessary to heat the grating because the quantity of light falling on its surface is of around 10000 times less than that crossing the primary lens. The soiling effect is reduced in the same proportions.

The Secondary Slits

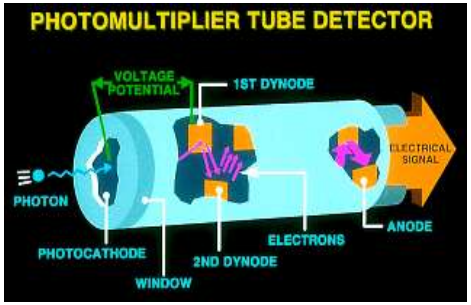
- (around 20 to 150 µm width) constitute the mask that lets only narrow wavelength bands to pass.
- The slits are fixed to the slit frame holder and an eccentric axle allows to adjust very precisely their position (to ± 2 µm).
- The light going through the slit is reflected by a mirror that focuses on the photomultiplier.

Mirrors

- The light going through the slit is reflected by a mirror that focuses on the photomultiplier.

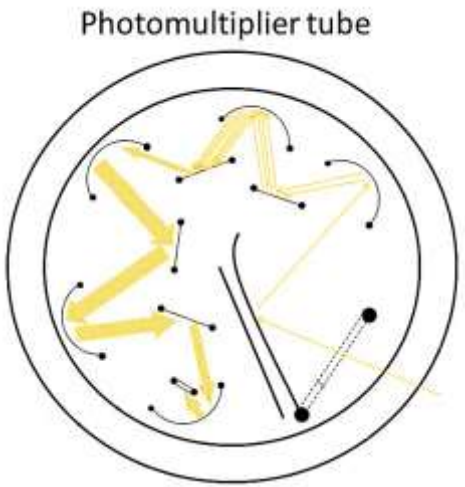
PMT (detector)

- Photomultiplier An electronic device that is used for measuring the light intensity for each instrument line within the spectrometer.
- The PMT change the incidence photons into electrical signal
- The numbers of PMT depending on the customer requirements.
- As the detector the PMT determines the intensity of photons of the analytical line exiting the monochromator .



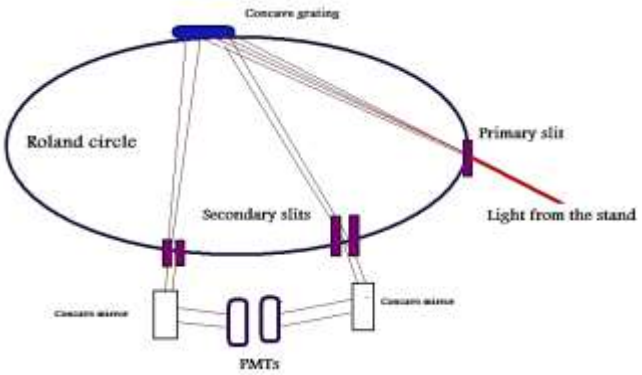
PMT

- The photons (light) hit the photomultiplier's photocathode and extract electrons from it. These electrons are
- Captured by the electric field existing between the dynodes of the tube, and then they hit again one of these
- Dynodes, extracting again more electrons. These electron sets are multiplied from dynode to dynode and
- Reach finally the tube's anode. All electron sets reaching the anode constitute the current to be measured. A
- Serial resistor network fixes the potential of each dynode.



Rowland Circle

The primary slit, the grating and the secondary slits are located on a circle; this is a necessary condition to obtain a proper focus on the secondary slits
The wavelength crossing each secondary slit depends on the angle formed by the incident beam and the diffracted beam of the grating.



By moving the primary slit on the Rowland circle, the wavelength crossing a fixed secondary slit on the circle is modified. This is how the instrument is profiled, i.e. the position of the primary slit is adjusted so that a given wavelength passes through a given secondary slit. This can be achieved, of course, only if relative position between each of the secondary slits is absolutely correct.

3. The Electronic Rack

- The electronic rack contains all the necessary circuit for the communication between the computer and the spectrometer.
- The principle is based on control by a microprocessor connected to the main computer.
- This microprocessor needs an adaptation card to manage the various readout lines, the stand and the source signals.

4. The Computer

- The computer fulfils two different tasks:
- The instrument control on one end and the calculation of the concentration on the other end.
- All data information exchanged between the computer and the instrument is driven on a RS232 serial line type.
- Data reach directly the instrument's microprocessor that processes it.
- Analytical results are transmitted from the microprocessor to the computer in the same way.

Sub-Assemblies

- **Argon system.**
- **Air filtration system.**
- **Water cooler.**
- **Vacuum.**
- **Exhaust.**

Argon System

- To avoid any interaction between the air atmosphere and the sample surface, the discharge is made in a sparking chamber under argon atmosphere.
- One can reduce the argon consumption if the instrument is not used for several hours.
- However one should not forget to re-open the valve, and to adjust it to the correct pressure, at least one quarter of an hour before to proceed to analyses.
- The argon should not be stopped, even for the weekends.

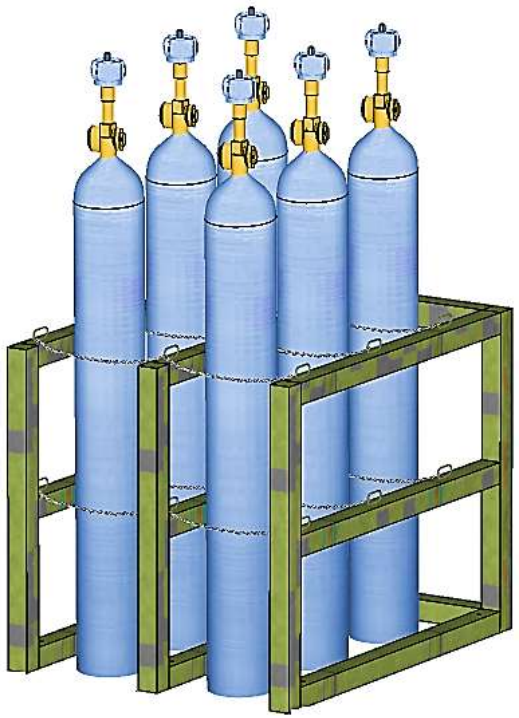
Argon System

Argon outlet filter.

- The filter retains the metal dust particles, which are drawn from the sample during the analysis.
- Should be cleaned regularly clean the filter cartridge with a vacuum cleaner or change it
- The average consumption of the ARL 3460, is about 5 l/min during a sample analysis and 0.7 l/min the rest of the time (stand-by).

Argon Quality

- Quality 99.998% (ev.99.996%)
- Input pressure 2 Bars (2 x 105 Pa)
- The argon must have a minimum purity of 99.996% and conform to the following specifications:
  - < 5 ppm O2
  - < 20 ppm N2
  - < 5 ppm H2O
  - < 5 ppm CO2 + CH4



Argon Exhaust

- It is highly recommended, and required according to some countries' prescription, to evacuate the argon outside the laboratory.
- The argon evacuation is essential in the case the instrument is installed inside an hermetically closed room, or if the air renewal rate is low. Indeed, in such a room, the argon replaces little by little the air and can lead to an asphyxia risk inside the room.

Air Filtration System.

Dust filters.

- Prevents dust from entering the instrument's lower cabinets.
- Should be cleaned regularly.

Fumivair 350 / 120

The atmospheric dust which are dispersed in the air can be distinguished

by their particle size:

- Ordinary dust > 10 micrometer
- Fine dust 1 – 10 micrometer
- Very fine dust < 10 micrometer
- The fumivair device intended to filter the steam and vapor coming from the analysis chamber of the instrument.
- It is made of a sheet – iron framework in which there is
- A pre filter ,
- An activated carbon filter
- A suction fan.

Water Cooler

- The energy dissipated by the sparks heats the sample and the table on which it is placed. The table is cooled by a closed water cooled circuit.
- This device is composed of
  1. Water pump,
  2. Tank, and
  3. Flexible pipes .

for bringing the cooled water to the analytical table.

Vacuum

The light path into the spectrometer is approximately 2 meters long.

As air will absorb ultraviolet light, it is necessary to put the spectrometer

under vacuum to avoid this effect.

- The vacuum system is composed of
    1. A pumping line,
    2. A manual aeration valve for the spectrometer
    3. A vacuum gauge.

- This air flow is to be adjusted by a pin-valve in order to keep a vacuum at around 50 µm Hg / 0.05 Torr.
- The pin valve should be closed when the pump is stopped.

VUV Line Analysis

Definition

- The VUV notion signifies Vacuum Ultra Violet and includes the spectral range of electronic wavelengths between ultraviolet and x-rays.
- Usually scientists admit the VUV spectral range is between 20 and 190 nm It is necessary to analyse the VUV spectrum under vacuum because the air absorbs the VUV radiation.

VUV

- An intermediary vacuum is usually enough for analysing wavelengths between 160 nm and 190 nm, but a better Vacuum(around 10-4 mbar)and additionally precautions must be taken for wavelengths below 160 nm.

At that time this includes particularly:

- The Nitrogen analysis in Steels and Nickels; line N 149.26 nm
- The Oxygen analysis in Steels and in Coppers; line O 130.22 nm
- Low Carbon, Chlorine, etc.

Exhaust

- This experience extends to the less common metallic materials. When it comes to the analysis of materials producing toxic vapors, such as zinc and lead, the special exhaust system of the ARL 3460 ensures maximum safety of the operator.

Air Exhaust

- The ambient air warmed by the instruments and other laboratory accessories can usually be evacuated using a fan or AC.
- In order to avoid return of harmful dust (mainly when the exhaust fan is off), we recommend to install a clapper directly on the outside.
- The argon evacuation is essential from the environment of lab.

Mg Filter Kit

Purpose

- The sparking of Mg matrix sample forms inevitably a powder with Mg particles, which are carried out by the argon along the exhaust pipes up to the output filter.
- It is known that the Mg powder is very reactive with the oxygen from the air and causes explosions (Mg powder is used for fireworks).
- The Mg filter is a device designed to dissolve the Mg powder in an acidic solution.

SOFTWARE

Instrument Controller System ICS

- This is the Instrument microprocessor that controls the spectrometer.
- It also receives the analytical parameters from the ACS and sends raw intensity values back for processing.
- ICS Emulation A software program on the ACS that can be used to simulate the ICS communication allowing the analytical software to be used without an instrument being connected.

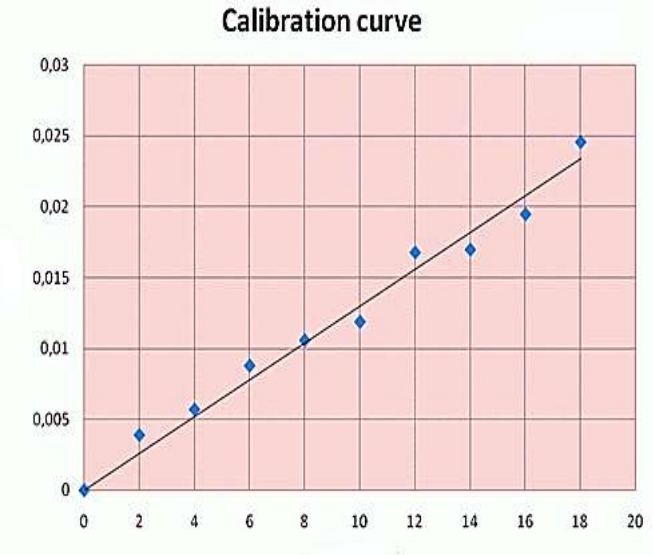
Win OE

1. Production / analysis
2. Calibration
3. Preparation
4. Utility
5. Configuration report
6. Initialization

Calibration

- Calibration sample tools
- Import and export calibration standards
- Create calibration file
- CSV
- MVR Multi-Variable Regression(MVR)

  Multi-Variable Regression is used to calculate calibration curves that include the effects of inter-element interferences.
- Type standard initialization
- Instrument precision



Utilities

- Alarms and status monitoring.
- Instrument configuration
- Communication
- Databases
- Security
- Administration

Configuration Report

Icons for setting the option for the report including :-

- The preparation tasks
- Utility tasks

Initialization

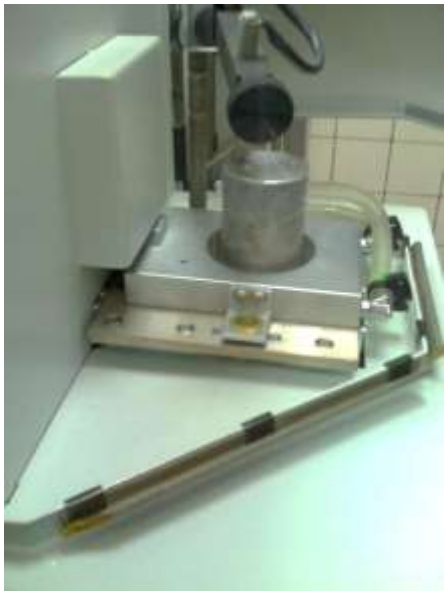
- Status reports
- Read instrument status
- Initialize alarms and status logger
- Reset instrument
- Send instrument configuration
- Direct profile
- Integrated profile
- Results transmission results

Preparation

- Channels
- Analysis
- Control samples
- Tasks
- Result processing

Production / Analysis

- Shortcuts
- Analysis
- Results processing



WinOE Software Structure

- The WinOE software has a set of core features and functions.
- The capabilities of this core software can be extended with System Options that can be activated individually.
- Some System Options are supplied free of charge with the **basic** WinOE software package.
- **Additional** options must be purchased separately.

Permission Level

- The content of the menu functions as well as the content of related folders depends on the permission level you are logged into WinOE and the enabled system options.
- To see the windows and the dialogue boxes as they are shown in this manual, you may need to log in with the **Laboratory Manager** or **the System Administrator privileges.**

INSTRUMENT PREPARATION

Thermo Plant

Note that

- Calibration
- The Setting-up Samples,
- Control Samples,
- Analytical Tasks
- Analytical Programs

are normally configured at the Thermo manufacturing plant as well as the However it is possible for you to prepare your own analytical programs .

Defining Required Samples

You need to define these samples before you can create the Analytical Programs that you will use for analysis.

- Calibration Standards
- Setting-up Samples
- Control Samples
- Type Standards

You will only need to define the Control Samples and the Type Standard Samples if you are planning to use SPC and Type Standardisation with your routine analysis.

Calibration Standards

Samples Of Known Concentrations That Are Used To Calibrate The Instrument.

- The Procedure For Initially Setting Up The Analytical Program Using Known Standards.
- The Accuracy Of The Calibration Will Never Surpass The Accuracy Of The Standards.
- The Calibration Standards Selected For Measurement Must Not Only Cover The Elements To Be Measured But Also Include Those Necessary To Determine Line Overlaps And Background Corrections.
- Preparation - Calibration Standards – Follow Up The Configuration Manual.

Setting-up Samples

The Set Of Top And Bottom Standards Used For Instrument Standardisation.

- The Standardisation is the analysis of setting-up samples. It is also known under various names such as Drift Correction, Recalibration or Normalization.
- Two samples are usually required to provide high and low intensity measurements for all elements of an analytical program. When the "highs" and "lows" for all elements cannot be found in two samples then more setting-up samples can be used.
- In every analytical program, it is necessary to specify which setting-up sample has the high value (Top Setting-up) and which has the low value (Bottom Setting-up).

Control Samples

Control Sample This Is A Sample To Monitor The Analytical Instrument Performance And To Highlight Any Instrument Problem.

When used in conjunction with SPC, it provides warning if the measured sample is outside statistical limits or showing an unexpected trend.

- WinOE
- Production / analysis
- Analysis
- Control sample analysis
- Change task (if)
- Select sample (if)
- Analysis

Type Standards

- **A Type Standard Is A Material Of Known Composition With Concentrations Close To That Of The Unknown Sample Being Analysed.**
- It Is Used To Make Adjustments To The Calibration Curve Of A Material Previously Calibrated Rather Than Carrying Out A Specific Calibration
- Type Standardisation may be used in addition to, or instead of Standardisation.
- Type standardize just before running one or more samples of an alloy type to fine tune the calculation.
- Type Standardisation is made in three steps:
    1. Preparation
    2. Initialization
    3. Corrections and Routine Analyses

Defining Analytical Programs

The Analytical Information Required When Analysing Samples.

- WinOE- Preparation – Analysis.

Steps

- Define Calibration Standards, Matrix and Matrix sub Group Identifier.
- Set-up Analytical Conditions and TRS Windows.
- Define Setting-up Samples with channels, analytical condition(s), and internal standards.
- Create the Analytical Program.
- Define Matrix and matrix sub-group the same as for the Calibration Standards
- Select Retrieve button
- Select Calibration Standards listed to build the Element Details automatically. All the channels defined in the Calibration Standards are automatically copied in the Analytical Program

Defining Analytical Tasks

The Predefined Procedure To Analyse A Sample.

- It can contain default information such as Sample Identity Lists, Analytical Program to be used, Result Storage and Transmission options, Result Presentation and Printing settings.
- WinOE- Preparation – Analysis.

Standardisation Initialisation

Procedure Carried Out When First Calibrating The Instrument To Measure The Initial Or Nominal Intensities Of The Setting-up Samples.

- Win OE - Calibration – Standardization / drift correction initialization.
- During the Standardisation Initialisation (Day Zero), the system will measure the setting-up samples and store, for every channel in each Setting-up Sample, the following parameters:
  - The intensity measured as Initial Standardisation (Day Zero) Intensity.
  - The absolute standard deviation as Absolute Sigma "σ".
  - The Date and Time of the Initial Standardisation (Day Zero).

Instrument Storage

- If your laboratory is not ready, it must be stored in an adequate room.
- The storage room must be closed, dry and must have at least a cemented floor.
- Temperature min. +2°C, max. 45°C
- Humidity max. 80%, without condensation
- The storage room must be free of dust and corrosive vapours.
- The instrument must stay on its transportation pallet. This makes subsequent moves easier and limits any vibrations.
- It is very important to have good air circulation around the instrument during storage. Dust can be cleaned, but humidity is harmful.

Laboratory Requirements

- Labor temperature Min. 16°C; Max. 30°Cmax. Variation 5°C/h.
- Labor humidity Min. 20%; Max.80% .
- No dust, or humidity or corrosive vapours and direct sunlight
- The instrument must be protected against vibrations.
- A free access of at least 80 cm should be allowed in the front, the back and the right side of the instrument.
- The room's surface be at least 400 cm by 350 cm.
- Electricity 230 VAC+10%-15%; 50/60 Hz±2% Fuse 16 A; Earth < 1 Ω 3460: 3.5 kVA
- Altitude Max. 2500 m

SAMPLE PREPARATIONS

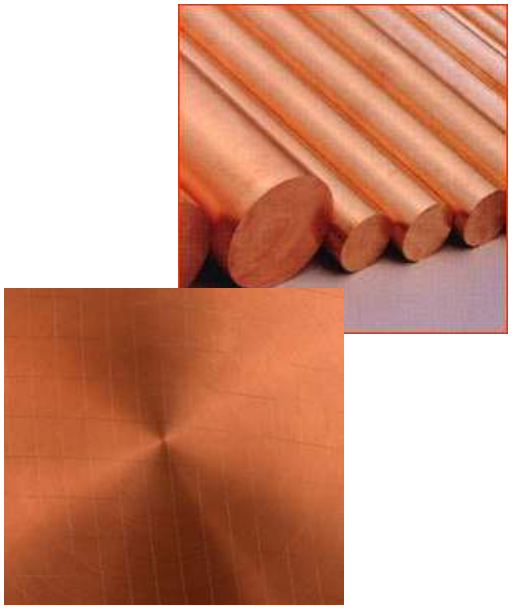
The Pre-analytical Work

- **Sample preparation.**

  It is necessary to have a sample as homogeneous as possible.

- **Sample surface preparation**.

  Clean and flat surface is needed in order to insure reliable and reproducible measurements.



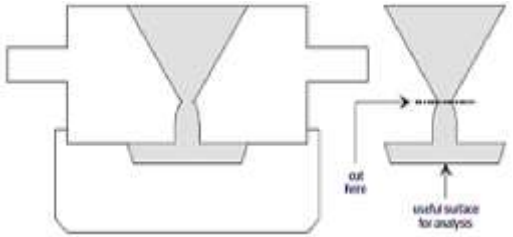
Sample Preparation

Moulds

- The mould will be in copper and maintained clean.
- The mould in stand-by of use must be turned upside down (or covered) in order to avoid dirties or other material to be introduced into it.
- The sample cooling must be quick so that a fine grain metallic structure is kept.

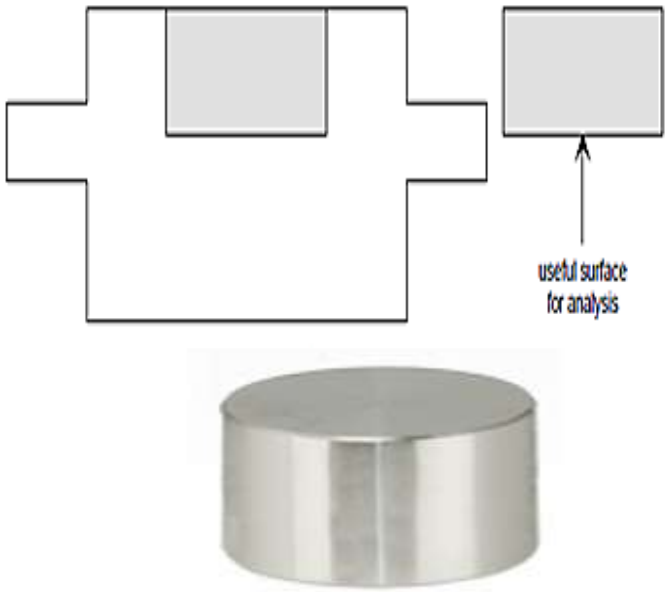
1. Non Ferrous Metals

- The mushroom form sample taking is the most used for non ferrous metals.
- There are however segregation risks, and the mould diameter must be adapted.



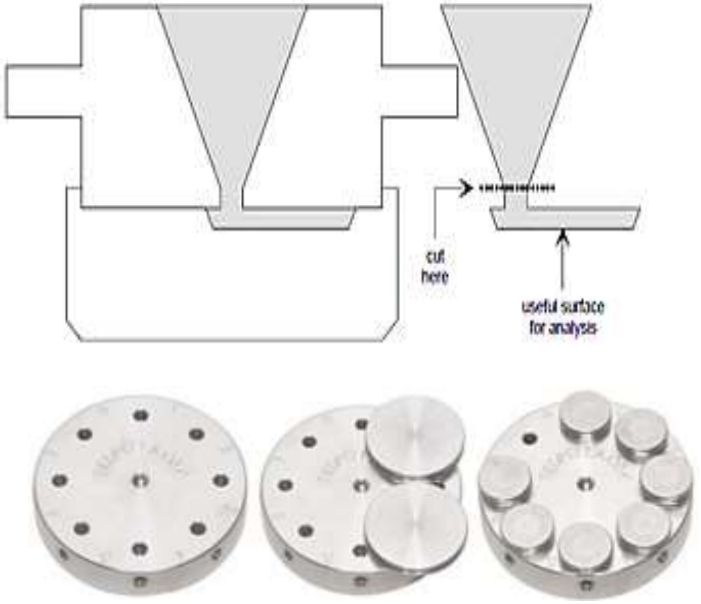
2. Fe, Ni, Co Bases

- Here is a very simple mould, frequently used for alloys of Fe, Ni and Co bases. (More specifically for cast irons and traces.



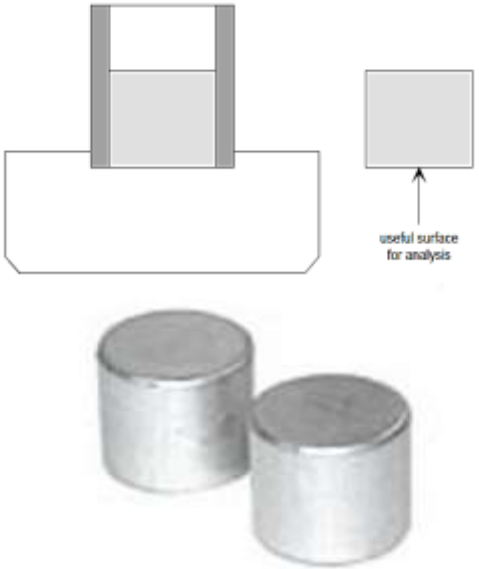
3. Cast Irons

- The kind of mould here is specifically adapted for casting cast irons in general, and pig iron in particular.
- The cooling speed is very high.



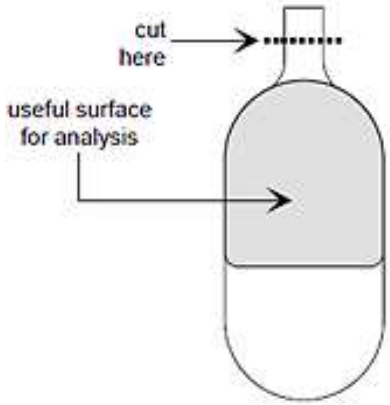
4. Traces

- The ring mould type, as shown hereunder, is advisable for casting pure metals (traces' analysis), or metals that will have low tendency to segregation.
- The ring is of stainless steel or ceramic, so that the sample will be mostly cooled by the bottom copper plate.
- The sample is homogeneous on its surface, but only on some few millimeters in depth!



5. Steel

- For steels, the sample taking can be greatly simplified with a SPEMIS probe that can take liquid metal directly from the casting.



Sample Surface Preparation

- It is essential to use an appropriate machine in order to obtain a clean and flat surface.
- We recommend the use of one of the following sample preparation machine types:

  - **Disk Sander**
  - **Surface Grinder**
  - **Milling Machine or Lathe**
  - **Press Machine**
  - **Inductive Furnace**

1. Disk Sander

- This surface preparation method is the quickest way to prepare **Fe, Ni and Co samples.**
- Sample must be thick enough so that they can be hold with fingers without the risk to injury themselves.
- The abrasive paper may always distort the result of some analyses, especially for elements in low concentration in the sample.

- The choice of the paper depends on which elements must be determined or not.
- The abrasive paper disk based with aluminum and silicon oxide mixed with resin .
- suitable. A grain of "80" or "60" is recommended.
- Soft metals like **Cu, Al, Pb, Zn, Mg,** etc., pure or even very low alloyed cannot be properly prepared on abrasive paper disks.

2. Surface Grinder

- Rotating surface grinders with multiple grindstones fixed on a rotating arm are recommended,
- This method is suitable for all sample kinds of **steels and cast irons, as well as for nickel and cobalt alloys.**
- No cooling liquid must be used.
- The magnetic samples will be kept still by the magnetic table. The non-magnetic samples must be fixed on a magnetic vice.
- The grindstones must be regularly sharpened, otherwise the risk of surface overheat .
- This can be the cause of bad sparking spots.

3. Milling Machine or Lathe

- The milling machine is the ideal machine for preparing all soft metals and steel.
- The milling machine is more suitable than a lathe for sample with complex shapes, for example melted or machined pieces. Moreover, the cutting speed is constant with the milling machine, that insures a regular machined surface.
- Oil or any other coolant must be prohibited.
- The all parts of the milling machine must be cleaned before the use for sample surface.

4. Press Machine

- For soft wire sample, one can flatten them with a pressure among 20 to 40 tons with a press.
- If the useful surface to be analysed is smaller than the 15 mm standard hole of the carbide tungsten disk, one should use another disk, that is equipped with a smaller hole.

5. Inductive Furnace

- Metallic chips can be re-melted in a fusion furnace.
- This furnace provides solid samples that can be used for spectroscopy analysis

ANALYSIS & FACILITIES

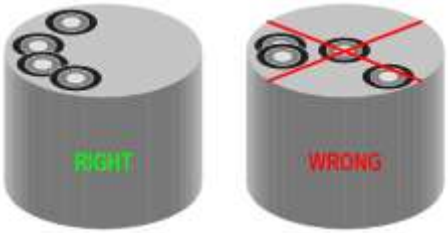
Analysis

The analytical routine work can start after the following operations:

- Instrument's calibration execution (only in case of instrument supplied without calibration),
- Cleaning of the stand,
- Profile check,
- Instrument's standardization,
- Quantitative analyses (routine analyses).

Placing The Sample On The Analytical Table

- The surface to be analyzed should no longer be touched with the fingers after the sample's surface preparation.
- This is also valid for the analysis table.
- One must avoid doing the analysis on the centre of the sample.
- It is a known fact the homogeneity is better in the area near to the edge of the sample.
- The sample will therefore be placed so that it hides the hole of the analysis table by about 3 mm on the table's area.



Multiple Measurements

- Usually in routine analysis, 2 or 3 measurements are carried out on each sample.
- Sparking spot's overlap must be avoided.
- Black halos around the sparking area can however be superposed, except in the case of oxygen analysis, where the spots must absolutely not crossover.

Profile

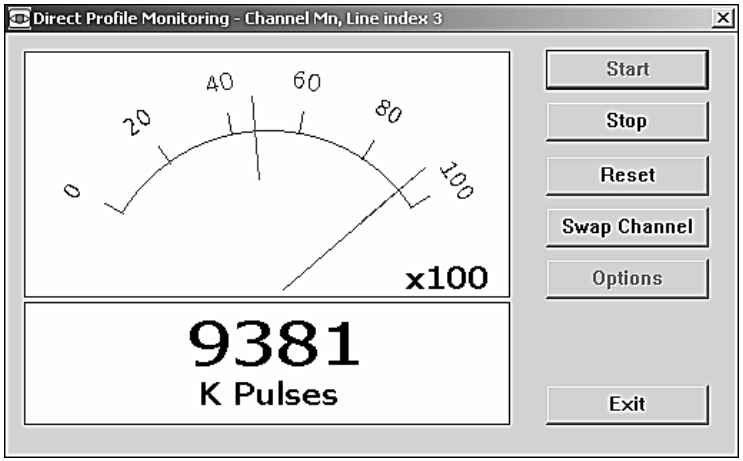
- Profiling is used to optimize the optical alignment of the spectrometer by moving the Primary Slit.
- It is used to correct mechanical variations within the spectrometer due to changes in temperature, humidity, etc.
- Profile Position Correct position for the primary slit after carrying out a Profile.

Profile Check

- The profile check of an element is finding the mechanical position of the secondary slit of this element relatively to the primary slit which is common to all of the elements (channels) installed in your instrument.
- By moving the primary slit (**scanning screw**) on the Rowland circle, the wavelength crossing a fixed secondary slit on the circle is modified. i.e. the position of the primary slit is adjusted so that a given wavelength passes through a given secondary slit.

Profile Types

- Direct profile (or manual profile) and is an analogue way,
- Integrate profile and is a digital way.



Direct Profile

- We recommend that you take the profile regularly **Direct Profile** on three channels.
- Always use the three same channels and note the profile positions found in your profile log book.
- A homogeneous profiling sample should be selected.
- The source operates permanently and it is not advisable to overheat the sample too much,

Direct Profile Steps

The profile should be check once per week ,

Use the Setting Up sample “SUS” for a homogeneous profile.

- Initialization – integrated profile.
- Choose the analytical conditions.
- Select 10 metals to display them.
- Put the SUS sample on the analytical table.
- Turn the screw 30 divisions counter clockwise then clockwise and put scanning screw at 90.
- Close the door and start profile, run profile.
- Change the scanning dial from 90 to 106 “2 division per step.
- Clean the table and electrode bet. steps.
- Change the spark position bet. Steps.
- Run profile – average now, and adjust the scanning dial to the new profile value.

Integrated Profile

- Integrated Profile This is a method of carrying out a Profile of the spectrometer using up to 10 channels simultaneously. The Profile Position is calculated automatically.
- The integrated profile follows the same principle of the direct profile, but here we make complete measurements (analysis) of the sample at several regular intervals of the scanning dial. After the number of required steps (measurements) is done, the system calculates automatically the profile value for all selected elements and provides the average.

Standardisation

- Procedure to periodically run Setting-up samples to correct for any instrument drift.
- **Standardisation Initialize**

  Procedure carried out when first calibrating the instrument to measure the initial or nominal intensities of the Setting-up Samples.
- **Standardisation Update**

  Procedure to update the Setting-up Standard Intensities when there has been significant drift of the spectrometer

Standardisation (Recalibration)

As the calibration is done once for all and should be valid for the whole life of the instrument, you need to proceed to the standardisation operation at regular intervals.

The purpose of the standardisation is to correct the instrumental drift at the medium and long term. The instrumental drift is due to several causes such as:

- Ageing of some of the generator electrical components that influences the sparking energy,
- Photomultipliers' ageing,
- Dirt on some optical components, such as the lens.

Steps

- The standardisation principle is , For each element measured, two reference "points" are required; one situated in the low part and the other in the high part or the working curve.
- Therefore two samples are needed, setting-up standards (SUS) whose homogeneity is recognized in the whole volume of the sample.
- The "low point" is often represented by a so-called "pure" sample, meaning that it contains practically only the matrix (or the base) of the alloy; for example a sample of pure iron.
- The "high point" is represented by a sample containing a high content in concentration of the element(s) that are present in the alloy to be measured.

Drift Correction Equation

Since it is admitted the drift correction is linear, the equation is of the type:

$$\mathbf{Inom = \beta + (Imes \times \alpha)}$$

where:

- **Inom**: Nominal intensity (during the initial standardisation),
- **Imes**: Measured intensity (current),
- **α**: Slope correction factor (multiplicative),
- **β**: Translation correction factor (additive).

- The observation and the regular comparison of the correction factors α and β will give you an idea of the stability of your instrument. β is a small positive or negative figure close to 0 (zero), α is a positive figure fluctuating around 1 (one).
- Large variations between two standardisations (especially for α) can give you an indication that there could be some problems at the instrumental response level

UMT Analysis Task

- Analysis Task The name given within WinOE for a predefined procedure to analyse a sample.
- It can contain default information such as Sample Identity Lists, Analytical Program to be used, Result Storage and Transmission options, Result Presentation and Printing settings.

	Task	Application
1	Pb CO GL Pb	Global lead
2	Pb CO Pb Ca	Lead Calcium alloy
3	Pb CO Pb Sb	Lead Antimony alloy
4	Pb CO Pure	Pure lead
5	Pb CO Sn Sb	Lead ,Tin Antimony alloy

UMT Analytical Program

Contains the analytical information required when analysing samples. An analytical program must be specified when analysing a sample and it will contain information such as Channels to be used, Calibration Curve Evaluation parameters, Setting up Standards required.

	Program	Application
1	dark	
2	Lamp	
3	Pb gen	
4	Pb Gl Pb	Global lead
5	Pb Pb Ca	Lead , Calcium alloy
6	Pb Pb Sb	Lead , Antimony alloy
7	Pb Pure	Pure lead
8	Pb QA Pb	
9	Pb Sn Sb	Lead ,Tin , Antimony alloy

Analysis Cycle

- Start-up of the software's analysis routine, answer questions asked by the software (program choice, sample identity, etc.).
- Placing the sample on the analysis table, closing the door.
- Start-up of the measurement by pressing the START button on the stand's housing.
- Waiting until the end of the measurement cycle, and observation of the displayed results.
- Run two or three measurements.
- Measurements' selection for the average calculation.
- End of analysis; the instrument is ready for the analysis of another unknown sample.

Analysis Steps

- Win OE – production / analysis.
- Analysis.
- Concentration analysis.
  - **Change task**

    chose the analysis task
  - **Select program**

    chose the analysis program
  - **Put the sample in table**

    change the spark position
  - **Analyze**

Analysing Concentrations in Unknown Samples

- Production / Analysis – Analysis - Concentration analysis
- Change Task “We have five tasks”
- Select the program “ We have nine analytical program
- Position the sample on analytical table
- Complete the sample identifier field and press “sample details OK”
- Press analyse, after the run the results window will display

- Bad sample “when a sample is too bad to be analysed by the instrument, but needs to be kept in a report.
- At this stage you can perform a new run. Rotate the sample on the stand on a position that is not already used and close the stand door. Press on the green START button on the instrument stand and click on the **Analyse Again button in the above WinOE window.**
- You should perform at least two runs before terminating the analysis

Analysis Cycle

- Flush This period corresponds to an argon flush time, the argon flushes the stand to ensure that the spark will start in an argon environment. Any impurities or contamination may disturb the spark. This usually takes between 3 to 5 seconds.
- Pre-integration During this time the high energy spark takes place, also called pre-spark, used to clean and homogenise the analysed part of the sample.
- Integration During this time the spark takes place, together with the measurement (integration) of the intensities.

Type Standard - Update

- The procedure for measuring a Type Standard prior to analysing unknown samples of that type, so that up to date corrections are made when the unknown samples are analysed.
- Win OE
- Production / analysis
- Analysis
- Type standard update.
- Change task (if)----- change type standard (IF)

Analysing Intensities in Unknown Samples

The steps to perform intensity measurements are the same as for analyses of concentration ,

- Production / Analysis
- Analysis
- Intensity measurement
- Change Task “We have five tasks”
- Select the program “ We have nine analytical program
- Position the sample on analytical table
- Complete the sample identifier field and press “sample details OK”
- Press analyse, after the run the results window will display

Analyses with Type Standard Correction

- **Type Standardisation is similar to Standardisation but** the correction is made to concentrations. Only one standard sample, called Type Standard is used for each Type Standardisation.
- Type Standardisation does not replace the standardisation but may be seen as a way to fine tune a calibration and may be performed more often than standardisation.
- Type Standardisation is an independent procedure that can be used or ignored during analyses

Analyses with Quality Check

- The function of Quality Check is to verify if the concentration of some or all elements present in the sample analysed are between the upper and lower predefined limits.
- The Quality Check is associated directly with an analytical program or indirectly through Type Standard.
- The Quality Check is an independent procedure that can be used or ignored during analyses.

REPORT OPTION

Report Templates

- The Report Writer supplied with the WinOE software uses the R & R Relational Report Writer by Concentric Data Systems Inc.

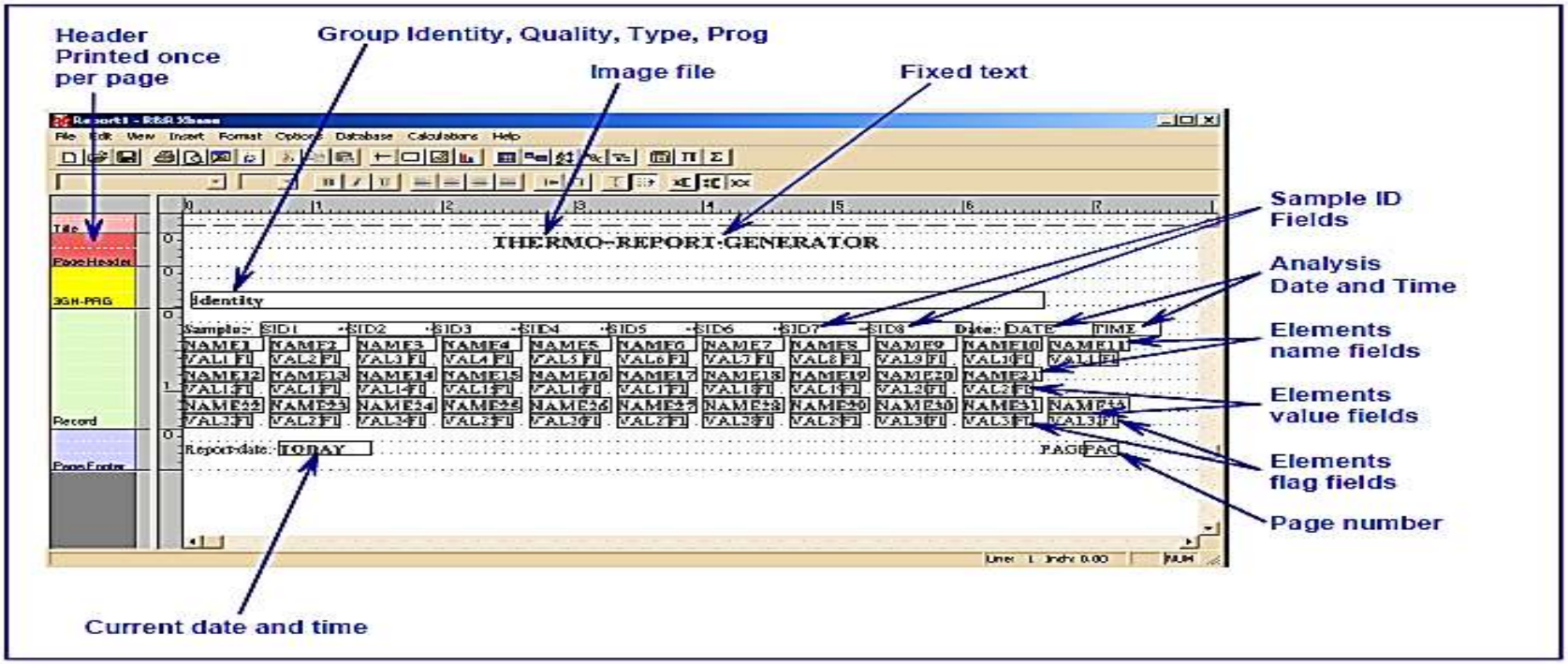
  WinOE – Production / Analysis – R & R report generator.

- Five report templates are supplied with the system and these can be used to display the results on the monitor or print them in reports.

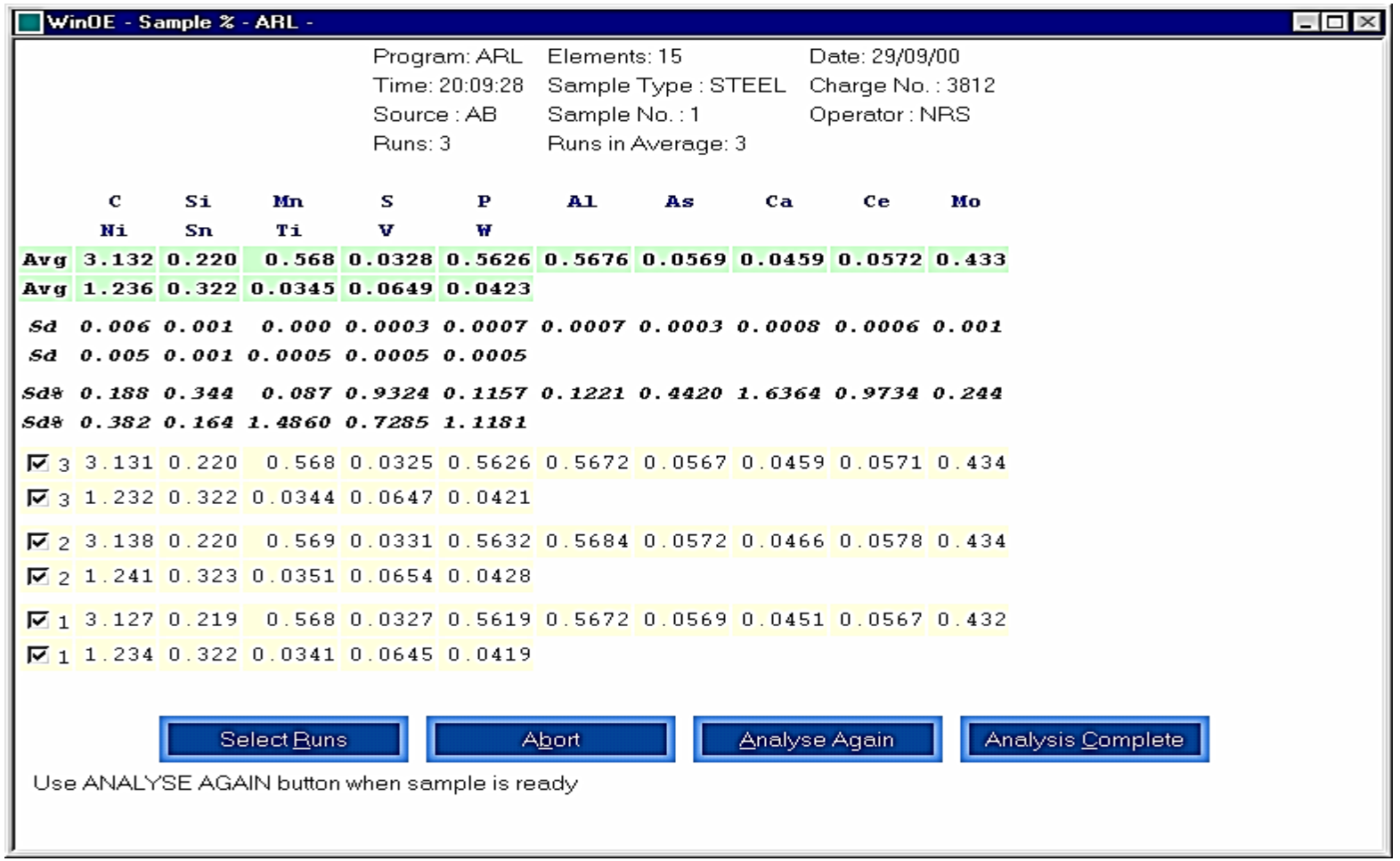
  WinOE - Production / Analysis - Analysis report on display.

Report Generator

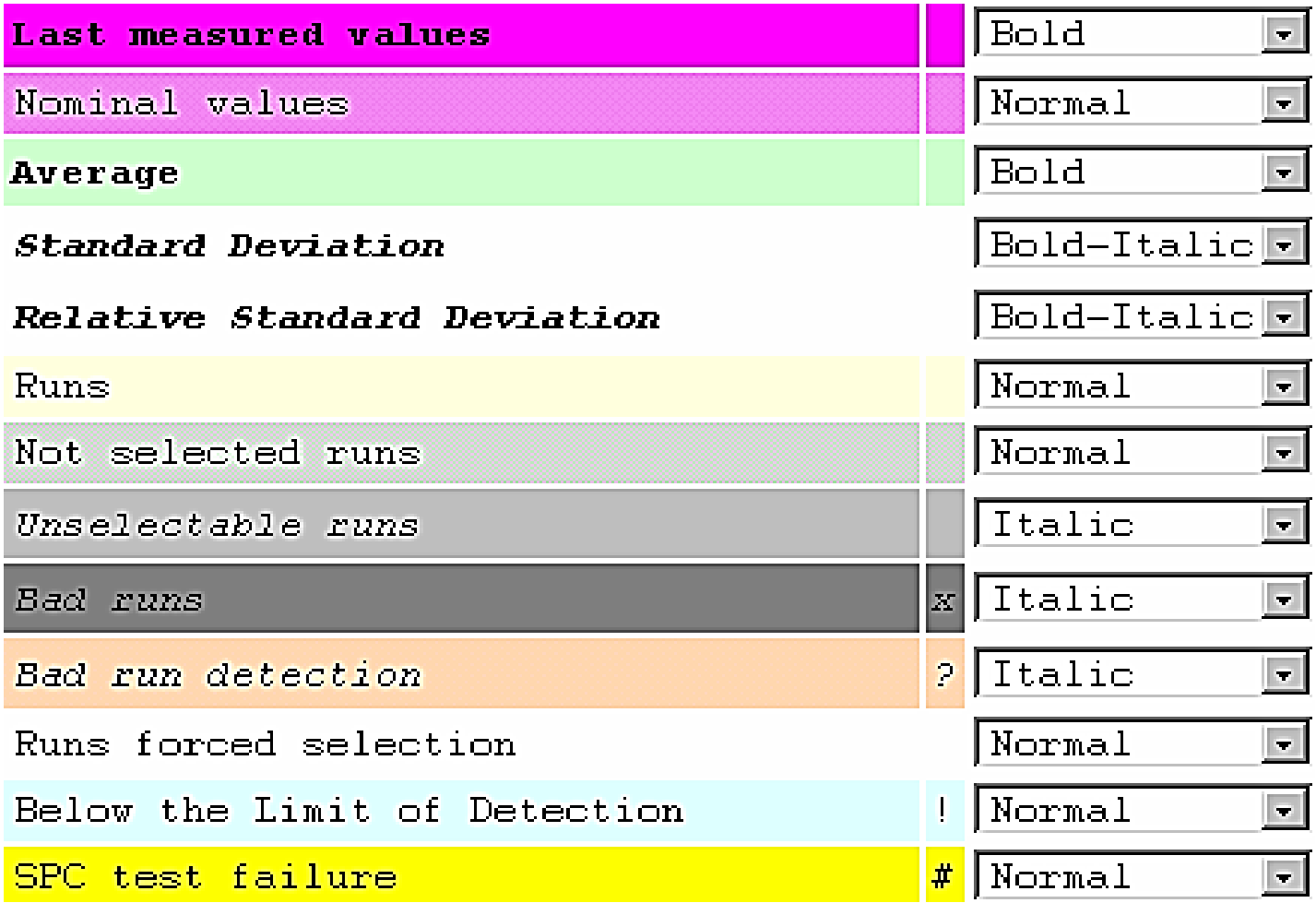
This is a software option for producing printed reports of selected analytical results stored on disc.



Results


WinOE - Sample % - ARL -
Program: ARL
Elements: 15
Date: 29/09/00
Time: 20:09:28
Sample Type : STEEL
Charge No. : 3812
Source : AB
Sample No. : 1
Operator : NRS
Runs: 3
Runs in Average: 3
C Si Mn S P Al As Ca Ce Mo
Ni Sn Ti V W
Avg 3.132 0.220 0.568 0.0328 0.5626 0.5676 0.0569 0.0459 0.0572 0.433
Avg 1.236 0.322 0.0345 0.0649 0.0423
Sd 0.006 0.001 0.000 0.0003 0.0007 0.0007 0.0003 0.0008 0.0006 0.001
Sd 0.005 0.001 0.0005 0.0005 0.0005
Sd% 0.188 0.344 0.087 0.9324 0.1157 0.1221 0.4420 1.6364 0.9734 0.244
Sd% 0.382 0.164 1.4860 0.7285 1.1181
3 3.131 0.220 0.568 0.0325 0.5626 0.5672 0.0567 0.0459 0.0571 0.434
3 1.232 0.322 0.0344 0.0647 0.0421
2 3.138 0.220 0.569 0.0331 0.5632 0.5684 0.0572 0.0466 0.0578 0.434
2 1.241 0.323 0.0351 0.0654 0.0428
1 3.127 0.219 0.568 0.0327 0.5619 0.5672 0.0569 0.0451 0.0567 0.432
1 1.234 0.322 0.0341 0.0645 0.0419
Select Runs
Abort
Analyse Again
Analysis Complete
Use ANALYSE AGAIN button when sample is ready

Colors and Result Flags



Result Tasks

Win OE – production / analysis – results processing.

1. Conc. In unknown samples (UCN)
2. Intensities in unknown samples (UIN)
3. Type standard initialization (TIN)
4. Type standard update (TUP)
5. Standardization initialization (DIN)
6. Standardization update (DUP)
7. Calibration (CAL)
8. Control sample (CTL)
9. None

SPC

SPC (Statistical Process Control)

- Statistical Process Control provides the facility to monitor an instrument or process over a period of time and use statistical techniques to alarm when results are outside calculated limits or show an unexpected trend.

MAINTENANCE

Maintenance

There are two types of maintenances :-

- Routine maintenance
- Service maintenance

Routine Maintenance

Maintenance subject	Recommended frequency	Details section
Cleaning of the electrode	Before each analysis	4.4
Cleaning of the insulator and the sparking chamber	At least once a day, before standardisation	4.5.1
Cleaning inside the stand (table, block support)	At least once a week	4.5.2
Argon outlet circuit Oil of argon circuit for the VUV option	At least once a week Every 6 months	4.6 4.6.2.1
Check pump oil level	At least once a week	4.7.1.1
Change vacuum pump oil and gaskets	Every 6 months	4.7.1.2
Profile check	Monthly (at first weekly)	4.8
Greasing of stand's door hinges	Once a month	4.9
Lens cleaning	Once a month	4.10
Cleaning the dust filters Changing the dust filters	Once a month Once a year	4.11
Operation of cabinet fans	Once a week	4.12
Running Control Sample and/or Standardisation	Once a day (optional) Once a week	4.13.1 4.13.2
Backup	Once a week	4.14
Accessories in option	According to manufacturer	4.15

Service Maintenance

- In order to guarantee a long term operation with a minimal breakdown risk, as well as to keep the initial analytical performances, it is highly recommended to overhaul completely the instrument at regular intervals, for example once a year.
- That should be performed by an Thermo Electron– or agreed local agent – service engineer.

APPLICATIONS

Applications

Non-ferrous Applications

Lead Ingot and Rolling Industries

- Quality control by rapid in-furnace analysis at each stage of manufacture
- Analysis for product standards evaluation
- Materials deliver inspections

Other Metals

- aluminum, copper, nickel, zinc and many other metals and alloys.

Applications

Ferrous Applications

Steel and Cast iron

- Quality control by rapid in-furnace analysis at each stage of manufacture
- Analysis for product standards evaluation

Materials deliver inspections

Applications

- Primary metal producing plants
- Foundries, forges, mini-mills
- Casting operations
- Incoming material control
- Metals QC and R&D laboratories