TECHNICAL DESCRIPTION

ARL 3460 Metals Analyzer
Optical Emission Spectrometer
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1. **SPECIFICATIONS**

1.1 **Function principle**

The ARL 3460 Metals Analyzer is a simultaneous Quantometer. This means the instrument measures the intensity of several spectral lines simultaneously in the light emitted by the sample, when the atoms that compose it are exited by an external energy source. The analysed light is located approximately into the 150nm to 800nm wavelength range. The whole measuring system is therefore based on the physical phenomenon that is summarised as follows:

When certain energy is applied to an atom, some of its electrons change their orbit. When these electrons return to their initial orbit, a precise energy is restored in the form of a light at a determined wavelength. This is an atomic phenomenon, and consequently it is practically unaffected by the chemical or crystalline form of the atom. This means the instrument can determine, for example the quantity of silicon in steel; but will not give information about the form under this silicon is to be found. The following figure gives a rough representation of this excitation.

![Excitation of an atom](image)

A sample containing several different elements will therefore produce light composed of wavelengths 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. By measuring this luminous intensity (with a photomultiplier) and by processing this information with a computer, the instrument can thus determine the concentration of the considered element.

An instrument that allows such analysis is therefore composed of the four following parts:

1) A source of excitation that supplies energy to the samples.
2) A dispersion device that discriminates the different wavelengths.
3) Electronics that measure the luminous intensity of each of the wavelengths.
4) A computer that processes the measurements and controls the instrument.
1.2 ARL 3460 Optical Emission Spectrometer

For over 60 years the metals industry has looked for one company to set the standard for instrumental analysis of metals. The ARL Model ARL 3460 is the latest instrument that brings the cost effective quality control. It will meet the highest expectations, but at a price that is considerably below what users would expect to pay.

The ARL 3460 is designed to excel as a mono-base or multi-base instrument and for high throughput sample analysis. Business depends on consistently producing high quality metals or metal products, and user needs an analysis system that will not let him down. The model ARL 3460 will go on working 24 hours per day, 7 days per week to keep production facility on track. Absolute accuracy of any analysis depends totally upon the calibration of the instrument. The model ARL 3460 is factory calibrated using CARL. This sophisticated multi-variable regression tool corrects for matrix effects as well as spectral interferences. The model ARL 3460 is ready to deliver accurate analyses from the first day of its installation.

1.3 Stability which gives confidence to user

User needs to be sure that he will get the same accurate results tomorrow, next week, next year. The model ARL 3460 inherits the sort of short and long term stability that have long been ARL standards.

1.4 Accessories and options
• Holders / adapters for small and pin samples,
• Casting moulds for ferrous and non-ferrous metals,
• Sample preparation machines for ferrous and non-ferrous metals,
• Argon purification systems,
• Gas distribution system for argon bottles,
• Voltage stabiliser systems.

1.5 Spectrometer computer system

Personal Computer system (COMP-STD):

• Processor Intel Pentium 4 @2.80 GHz, 1 GB Memory;
• 160 GB hard disk drive, internal DVD/CDRW combo drive (DVD reader, CD-Writer). No floppy disk drive;
• Operating system Windows® XP Professional Service Pac English, French, German or Spanish;
• 17" LCD TFT video screen, keyboard according to selected language of operating system, mouse;
• Integrated Ethernet 10/100/1000 LAN interface (RJ-45 connector);
• 1 serial (reserved for instrument link), 1 parallel and 8 USB ports (2 front and 6 back). 2 USB ports are already used for the mouse and the keyboard.

Please refer to our Commercial Quotation for the latest configuration

Includes factory installation and configuration of operating system and instrument software.

1.6 OXSAS Analytical Software

OXSAS is the most powerful software available for optical emission spectrometer, offering both flexibility and comprehensive capabilities in "one state of art" package.

32 bits software that runs on Windows® XP Professional and includes the following main features:

• Modern, State-of-the-Art Graphic User Interface;
• Integrated Microsoft® SQL Server 2005 Express relational database which stores your set up data and analyses;
• Quantitative analysis with analysis parameter template;
• Shortcuts allowing to start routine sample analyses, routine measurements of SCT samples and batches using a user-defined analysis template. The value of parameters such as the sample identification can be predefined. With support of shortcut keys enabling users to operate analysis exclusively on the keyboard
• Powerful batch management for unknown samples analysis and other operations such as standardization, measurement of control, type standard and calibration samples or custom scripts execution
• Comprehensive editor for setting up methods with measurement and corrections parameters;
• Manual inputs and status values. Pseudo values computed on intensities, concentrations or any calculation step;
• Calibration curve determination using multi-variable regression with additives and multiplicatives corrections;
• Powerful flexible sample identification;
• Manual and automatic result processing;
• Multi-purpose analysis result display and printing;
1.7 Technical Description

Spectrometer has the ability to measure up to 60 spectral lines simultaneously. It is used for quantitative determination of elements by photo-electric measurement of characteristic wavelengths. Consisting of:

Simultaneous spectrometer
With concave grating “Paschen-Runge” mounting, cast construction and 1m forcal length. The spectrometer is shock mounted, insulated and temperature controlled to ±0.1°C at 38°C to minimize profiling and standardisation frequency and maximise sample throughput.
Primary slit: 20 µm

Secondary slit: 20, 25, 37.5, 50, 75, 100, 150 µm

Photomultiplier tubes: 28 mm, 10-stage side window tubes, fused quartz or glass envelope. The phototubes are mounted outside the vacuum chamber in order to avoid corona-discharge and for a better serviceability in case of line addition.

Attenuators: All attenuators are programmable for optimum phototube adjustment, each attenuator has 41 steps

Grating: The spectrometer is provided with the following grating, appropriately selected for your analytical task: 1080 gr/mm; 1667 gr/mm; 2160 gr/mm.

<table>
<thead>
<tr>
<th>Ruling (lines/mm)</th>
<th>Order</th>
<th>Blase (nm)</th>
<th>Range (nm)</th>
<th>Inverse dispersion (nm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1080</td>
<td>1st</td>
<td>600</td>
<td>400 - 820</td>
<td>0.93</td>
</tr>
<tr>
<td></td>
<td>2nd</td>
<td>300</td>
<td>230 - 400</td>
<td>0.47</td>
</tr>
<tr>
<td></td>
<td>3rd</td>
<td>200</td>
<td>170 - 230</td>
<td>0.31</td>
</tr>
<tr>
<td></td>
<td>4th</td>
<td>150</td>
<td>130 - 205</td>
<td>0.23</td>
</tr>
<tr>
<td>1667</td>
<td>1st</td>
<td>347</td>
<td>220 - 528</td>
<td>0.60</td>
</tr>
<tr>
<td></td>
<td>2nd</td>
<td>173</td>
<td>160 - 260</td>
<td>0.30</td>
</tr>
<tr>
<td></td>
<td>3rd</td>
<td>116</td>
<td>130 - 176</td>
<td>0.20</td>
</tr>
<tr>
<td>2160</td>
<td>1st</td>
<td>200</td>
<td>170 - 407</td>
<td>0.47</td>
</tr>
</tbody>
</table>
Max. degree of vacuum: \( 1 \times 10^{-3} \) mm Hg

Temperature control: Inside of spectrometer is temperature controlled to \( \pm 0.1^\circ \) at 38\(^\circ\) for ambient temperature 16 - 30\(^\circ\) C

Spark stand: Argon flushed. Stand with built in ignitor for improved trace analysis. Included closed water cooled system. The safety circuit protects the operator and the instrument. It is not possible to perform an analysis when the stand door is open. This circuit conforms with the international norm CEI 1010-1.

Fatigue lamps: With built-in intensity level selectable by software. The fatigue improves the stability of the photomultipliers as well as their life time.

Source excitation model: HIREP (HIgh REPetition source)
Power: 400 VP
Ignitor: 15 KVP
Spark discharge rate: 400 discharges / sec (400 Hz)

Measuring method: Integration

Spectrometer control: ARL MMB 88 microprocessor utilising CMOS technology with status measuring card A/D converters and attenuators included for each channel. Up to 60 analytical channels

Dust protection: Built-in dust protection with high capacity cooling fans

Argon Flow: 3.5 L/min during analysis
0.3 L/min stand-by

Dimension: Height: 119 cm
Width: 166.5 cm
Depth: 91 cm

Approximate weight: Net 450 Kg approximately

Packing: Sealed airfreight packing as follows:

<table>
<thead>
<tr>
<th>Packing</th>
<th>Material</th>
<th>Gross weight</th>
<th>Dimension (cm)</th>
<th>Volume ( m^3 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 case</td>
<td>ARL 3460</td>
<td>748</td>
<td>195 x 140 x 145</td>
<td>3.958</td>
</tr>
<tr>
<td>1 case</td>
<td>Screen</td>
<td>27</td>
<td>55 x 53 x 49</td>
<td>0.142</td>
</tr>
</tbody>
</table>
1.8 Storage

If laboratory is not ready when the instrument arrives, the instrument must be stored in an adequate room. Recommendations are as follows:

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.

1.9 Laboratory requirements

**Ambient temperature:** 16 - 30°C, maximum rate of change ± 5°C per hour  
**Humidity:** 20 - 80% relative  
**Altitude:** 2'500 meters above sea level

Example of laboratory layout

1 Quantometer ARL 3460  
2 Table (for computer and peripherals)  
3 Office desk  
4 Chair  
5 Cupboard (for samples, material, documentation)  
6 Washbasin
1.10 Electrical requirements

Single phased supply (phase, neutral and earth)
- Voltage: 230 V ±10% -15%, fuse 16A;
- Earth impedance: < 1Ω (according CEI 1010 standard);
- Frequency: 50 / 60 Hz ±2%;
- Power: 3.5kVA (5KVA regulator required if fluctuations exceed specifications).

Standard Network
Most of the European networks provide with mains within the requirements given at the beginning of this chapter. In such case the connection diagram becomes:

Unstable 230 VAC Network
In the case of an unstable network, a voltage stabiliser must be inserted. In such a case the connection becomes as follows:

Unstable Network without Neutral
If a network similar to the above described is moreover unstable, a voltage stabiliser must be added. If so, the connection diagram becomes:

Note *: Stabiliser without link or connection between Input 2 and Output 2. Depending on the design, such a stabiliser may be used as an insulation transformer too!
### 1.11 Argon requirements

Argon pipes must be copper, and must have been cleaned before they are used (no oil nor grease).

The required inlet pressure is about 2 bars (2 x $10^5$ Pa). A two-stage pressure regulator (with manual pressure adjustment from 0 to 3 bars of the second stage) must be installed.

The argon must have a minimum purity of 99.996% and conform to the following specifications:
- < 5 ppm O$_2$
- < 20 ppm N$_2$
- < 5 ppm H$_2$O
- < 5 ppm CO$_2$ + CH$_4$

For analysing cast iron with Si > 1%, for aluminium and magnesium alloy, the highest impurity contents for oxygen and water vapours are:
- < 2 ppm O$_2$
- < 3 ppm H$_2$O

For the analysis of nitrogen and oxygen the required quality is:

<table>
<thead>
<tr>
<th>Argon quality</th>
<th>Purity</th>
<th>N$_2$</th>
<th>O$_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Argon 57</td>
<td>99.997%</td>
<td>&lt; 0.6 ppm</td>
<td>&lt; 0.3 ppm</td>
</tr>
</tbody>
</table>

As far as the supplied Sircal purifier is connected and operational, it is possible to use a lower argon quality, that is:

<table>
<thead>
<tr>
<th>Argon quality</th>
<th>Purity</th>
<th>N$_2$</th>
<th>O$_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Argon 48</td>
<td>99.998%</td>
<td>&lt; 10 ppm</td>
<td>&lt; 2 ppm</td>
</tr>
</tbody>
</table>
2. INSTALLATION OF ARL 3460 METALS ANALYSER

The actual installation is carried out by the factory service engineer or by local ARL representative's engineer. The service engineer will perform the following tasks:

- Check that the delivery conforms to the contract and the delivery packing slip.
- Unload the instrument from its transport pallet.
- Open the instrument before power up, so as to determine if the transport has caused any internal damage that is not visible from outside.
- Check that the laboratory and the connections provided for the instrument that customer has prepared meet the requirements (in chapters 2 to 5 in the pre-installation manual).
- Install and connect the peripherals (computer, printer, etc.) to the instrument.
- Start up the instrument.
- Carry out some typical operations that prove that the instrument is operating correctly.
- Check out (if applicable) the factory calibrated programs with certified standards samples (to be provided by the end use) and instruct the customer how to use those programs.
- Give the customer a basic training on start up and routine maintenance of the instrument (checks, precautions, changing Consumables). The customer will also be given an introduction to the normal use of the software. This basic teaching should not exceed one day.

Notes Sample preparation, analytical assistance, analytical calibrations, extended software teaching, teaching the maintenance personnel (troubleshooting) as well as other services, if they are not included in the contract, are additional jobs outside installation. These will be carried out after the instrument has been accepted. The local tariff will be applied and our local representative can send you an offer. It should be noted that reading the documentation delivered will allow the customer to deal with any problems, or questions concerning the above. ARL also offers regular training courses at our premises. Our local representative can also gives the necessary information.

End of installation
At the end of the installation, the service engineer will write an end of installation protocol, as well as a technical report on the installation. The guarantee covers the instrument according to the terms of the contract. Please, do not hesitate to call the ARL representative if you require any assistance. The instrument has a model code (name of the instrument), as well as a serial number. These indications are on a label behind the instrument. They are also written at the end of the installation protocol. Kindly quote these as well as your company's name in all correspondence. This will help us to give you an efficient service.
3. **SAMPLE PREPARATION**

For accurate and reproducible measurements it is essential to have a homogeneous sample, without inclusions and with a clean, flat surface. Thus a suitable sample taking method together with careful sample surface preparation are absolutely essential for good analysis.

For sample taking and surface preparation, several kinds of moulds and preparation machines are readily available for purchase. If required, your local ARL representative will advise you regarding which type and model best suits your application. Following are some general examples of the various sample mould types with associated sample preparation machines and their main applications.

### 3.1 Sample Taking – Moulds

Sample taking for spectrometer analysis is a fundamental technique. Several spectroscopy books explain the procedure and advise which moulds should be used for a given application. DIN and ASTM standards also provide specifications and information about this subject.

Generally:
- The sample cooling must be rapid to produce a fine grain metallic structure.
- The mould is normally made of copper and kept very clean.
- Any mould not in use must be turned upside down (or covered) to avoid dirt or any other material ingress.

Here are some examples of moulds that are suitable for casting good quality samples and adapted for optical emission spectroscopy analysis. A probe for direct sample taking is also shown.

**Non-Ferrous Metals**

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

**Cast Irons**
The mould shown below is suitable for the following metals:
Pig Iron and mixer metal, cast iron, malleable iron and nodular cast iron.
The cooling speed is very high to suppress precipitates.

**Traces**
The ring mould type shown below is suitable for the following metals:
Pure metals (for trace 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 this surface, but for only a few millimetres in depth!*
SPEMIS Probe
For steels, the sample taking can be greatly simplified with a SPEMIS probe that can take liquid metal directly from the casting. The sample obtained has the following form:

3.2 Surface Preparation - Sample Preparation Machines

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

Disk Sander
This is a sander with a rotary abrasive disk. Some machines have two disks; which is convenient where a coarse grain disk is used for preliminary surface preparation (roughing), and a fine grain for final sample surface preparation.

This surface preparation method is the quickest way to prepare iron, nickel and cobalt samples (as well as a few coppers). The sample must be thick enough to allow the operator to hold without the risk of injury to his fingers. The abrasive paper disk, based with aluminium and silicon oxide mixed with resin (Al/Si = 1:47) is usually suitable. A fine grain disk of "60" or "80" is recommended for the final finish and a coarse grain disk of "40" for initial rough grinding. For the analysis of soluble-insoluble aluminium, abrasive disks based with zirconium must be used to avoid cross contamination.

Note: Copper, aluminium, lead, zinc, magnesium, pure or even very low alloyed, and generally all soft materials, cannot be properly prepared using abrasive paper disks.
Surface Grinder
Rotating surface grinders with multiple grindstones fixed on a rotating arm are highly recommended:

![Surface Grinder Diagram](image)

This grinding method is suitable for all steels and cast irons, as well as for nickel and cobalt alloys. Points to note:
- Cooling liquids must not be used.
- The magnetic samples will be kept in place by a magnetic table. The non-magnetic samples are held in a vice that is then secured by the magnetic table.
- The grindstones must be regularly sharpened, otherwise the risk of surface overheat and deterioration of the sample is high, causing bad sparking spots.

Milling Machine or Lathe
Such a machine should be equipped with a milling cutter or chisel of tungsten carbide. The milling machine is the ideal machine for the preparation of all aluminium alloys, pure coppers and other soft metals.
Milling is also the recommended method for the sample preparation of steel analysis with nitrogen and carbon at lower level than the 10 ppm.
The milling machine is more suitable than a lathe for samples with complex shapes, e.g. melted or machined pieces. Moreover, the cutting speed of the milling machine is constant which ensures a regular machined surface.
The surface preparation is normally done without coolant (i.e. dry). However – for aluminium samples – a little of isopropyl alcohol may be used. Oil or any other coolant is prohibited.

Note: If your preparation machine is ever used for other applications that required the use of oil, water, or any other coolants, then the head, the milling machine table, the milling cutter and the vice for holding the sample, must all be thoroughly cleaned before using again for normal spectrometer sample surface preparation.

Example
Some steel springs can be analysed if prepared as shown in the following picture (flat surface preparation by milling or polishing):
Press Machine
For soft wire samples, one can flatten them with a press using a pressure of between 20 to 40 tons.

If the useful surface area to be analysed is smaller than the 15 mm standard hole of the standard analysis table, one should use another smaller hole table or a special small sample adapter to accommodate its size.

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