Thermo Scientific ARL Fire Assay Analyzer Optical Emission Spectrometer

Fast analysis of fire assay lead buttons with ultimate performance

High-quality analysis of all the precious metals in lead buttons simultaneously and in about a minute

Simplifies and speeds-up the fire assay analysis process, while saving costs on cupels, chemicals, etc.

Costly traditional analytical instruments, labor intensive and environmentally unfriendly methods replaced by inexpensive, fast, clean and easy-to-use OES

Exceptional reliability and availability combined with unmatched detection limits, precision, accuracy and stability

Suitable for the most demanding analytical purposes from exploration to metal accounting, and from mining to recycling industries

ARL Fire Assay Analyzer

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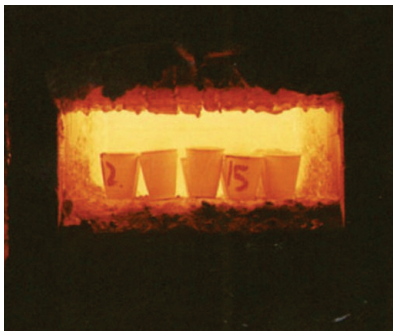
Introduction

Significant progress has been made in the determination of precious metals (PMs) traces in ores by the "fire assay" method. The ARL Fire Assay Analyzer makes optical emission spectrometry (OES) suitable for the analysis of many kinds of samples, e.g. geological samples in the gold and platinum mining industries, or samples in the precious metals industries (e.g. catalytic converters for car industry) or in contract laboratories.

Traditionally in fire assay analysis, an ore sample containing PMs traces is crushed, ground and weighed before being heated and mixed with a flux. This flux contains in particular litharge (Pb oxide), a reducing agent (e.g. flour) and other compounds forming a slag. The litharge is reduced to metallic Pb, which quantitatively extracts gold, silver and the platinum group elements (PGEs): platinum, palladium, iridium, rhodium and ruthenium. The fused mix is then poured into a cone shaped mold where the molten lead containing the PMs sinks to the bottom, while undesired impurities are removed into the slag floating on the top. After cooling down and removal of the slag, the obtained button is traditionally heated in an MgO "cupel" placed in an open furnace. This stage is therefore called "cupellation."

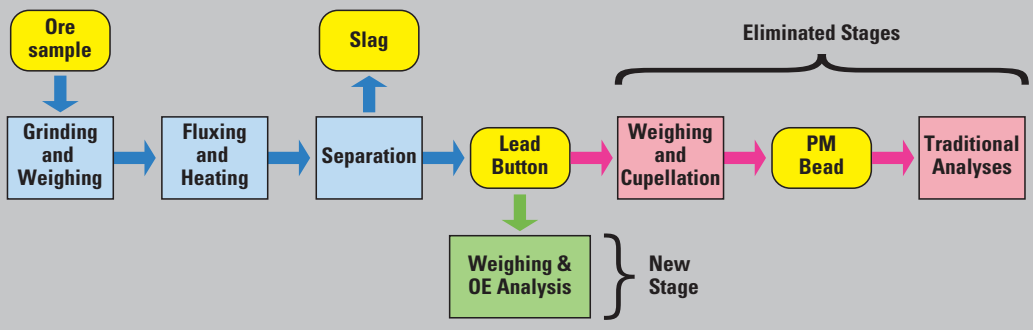


During cupellation, the Pb is oxidized by the air and mostly absorbed by the cupel, leaving only a PM bead or prill, which is further analyzed by various techniques, according to the particular analytical requirement, e.g:

- Gravimetric analysis for total precious metals (TPM) determination, for obtaining concentrator plant data in the Pt industry and many Au assays
- ICP-MS or ICP-OES for individual PGE and Au determination, for more accurate concentrator plant analysis

Fire assay analysis is not only performed by all PMs producers, but also by companies producing copper or nickel with PMs as a by-product, by those producing PMs from recycled materials and by contract or service laboratories. Used for centuries, this method has survived for so long because it is accurate, has low detection limits and can accommodate virtually any ore type. However, in the modern world, it has increasing disadvantages in terms of productivity and costs. Optical emission spectrometry (OES) can be used to measure PMs concentrations directly in the lead buttons (see diagram below), providing numerous advantages compared to traditional techniques:

- The elimination of cupellation not only simplifies and speeds-up the process but also saves costs on cupels, furnaces, power consumption, chemicals, laboratory equipments, etc.
- Losses of PMs in the cupel are avoided
- High-quality results on all the PMs are obtained simultaneously in about a minute, making OES suitable even for the most demanding operations (e.g. for analysis of slurries in concentrator plants for plant control and for metal accounting purposes)
- The environmentally unfriendly and labor intensive cupellation, sample preparation techniques and analyses are replaced by a clean technique that requires only a simple and inexpensive sample surfacing
- High stability, low maintenance and re-standardization requirements of the ARL Fire Assay Analyzer guarantee the highest availability for analyses
- Qualified personnel can be released for other tasks
- Possible automation of the sample preparation and handling, ensuring high throughput and reliability of the analyses



Flux diagram of fire assay analysis

The last stages of the traditional method (pink) are replaced by OE analysis performed directly on the lead buttons (green).

ARL Fire Assay Analyzer

The Thermo Scientific ARL Fire Assay Analyzer can determine all the necessary elements for the analysis of lead buttons in less than a minute. Built on some well-proven hardware features of the high-end ARL 4460, the ARL Fire Assay Analyzer is designed to take the most demanding requests from the fire assay market into account. Extreme care is taken on materials selection and on manufacturing and adjustment operations.

Moreover, we can optimize the configuration of your instrument according to your fire assay lead buttons qualities.

Current Controlled Source (CCS)

The ARL Fire Assay Analyzer is equipped with the Thermo Scientific Current Controlled Source, the only servocontrolled source on the market. This spark source presents significant advantages in comparison to any other spark generator used for OES.

The computer controlled current waveform of the spark was optimized for lead assay buttons analysis. The high degree of flexibility in selection of peak current (250 A max.), frequency (1000 Hz max.) and plateau current waveform enabled the optimization of all analytical figures in the special lead matrix. This was in particular important for the determination of the PMs at trace levels. It also made it possible to obtain sparked areas of small dimension (4 mm), an advantage in order to perform several measurements on samples that are usually small.

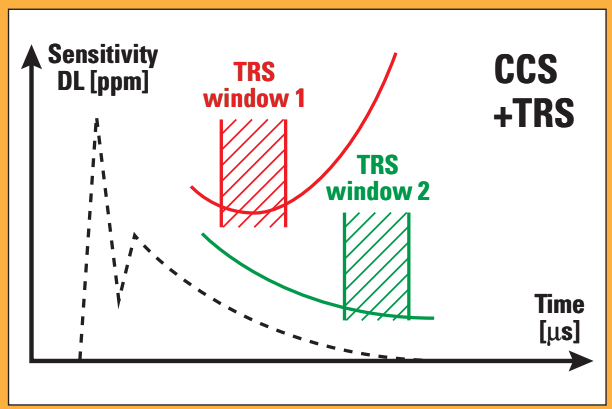
Time Resolved Spectroscopy (TRS)

During the time of an individual spark, the signal-to-background ratio varies significantly. TRS allows the selection of the most appropriate time window during the individual spark in order to improve sensitivity and precision. The peak of current (providing high background) is avoided and the acquisition window is restricted to the period of time providing the best signal-to-background. In fire assay analysis, this is important for the analysis of PMs which are normally present at ppm levels or below.

Furthermore, TRS can substantially improve the accuracy of the calibration in case of spectral line interferences. When analyte and interfering lines have significantly different excitation potentials, the acquisition window of the analyte is set so as to minimize the signal due to the interfering line. The following picture illustrates TRS selection for a PM (window 2) and for an interfering element (window 1).

CCS + TRS working principle

The dotted lines represent the current waveform of the spark, characterized by peak and plateau currents.
The TRS windows (dashed areas) are set such as to minimize the detection limits (solid lines) of the different types of analytical lines.



Sample dimensions and quality

The 10 mm diameter hole of the spark stand table and the small diameter of the spark spot (4 mm) allow measuring samples of small dimensions. With a manually operated spectrometer, for instance, five measurements are easily performed on a 20 mm diameter lead button. With an automatically operated instrument, the buttons must have minimum diameter of 25 mm and thickness of 8 mm, corresponding to about 40 g lead.

In order to guarantee the performance of the ARL Fire Assay Analyzer, it is essential that the fire assay process produces homogeneous lead buttons with smallest possible amounts of impurities.

Sample preparation

A lathe or a milling machine is used to prepare the surface of the lead samples.

Sample analysis time

The analysis time, taken between the start of the analysis and the display of the result, is 24 s. With an automatically operated spectrometer, the two measurements of a typical analysis are performed in less than a minute. If the instrument is operated manually, this time may be slightly longer, depending on the operator.

Factory calibration

Thermo Scientific optical emission spectrometers can be factory calibrated for fire assay lead buttons with CARL, a very sophisticated multivariable regression tool that corrects for matrix effects as well as spectral interferences. CARL provides an immediate "turnkey" system, which gives the user the highest accuracy possible.

Calibration

Our company offers calibrations tailored to your analytical needs. A global calibration that can measure all the elements present in your Pb buttons is available. The typical calibration ranges can be deduced from the detection limits and precision table hereafter. The lower limit of the calibration range of an element is about three times the detection limit. The higher limit of the range is typically the highest concentration for which a precision value is given, but for most of the elements, extension to higher concentrations is possible.

The accuracy of the analysis may depend on the composition of your lead buttons. The evaluation performed by our company will help making your buttons more suitable or designing matrix-matched calibrations. If buttons show significant differences in their composition so that several calibrations have to be used, the global calibration can be used for the « Program Choice ». In this case, the global calibration acts as a sorting calibration that selects the most appropriate calibration for concentrations calculation.

For more details on calibrations, please contact your nearest Thermo Fisher Scientific representative.

Detection limits and precision

Low detection limits are required in order to detect the extremely small amounts of PMs contained in fire assay lead buttons. They can be obtained only if precision is good and if the lead buttons and the elements that they contain are homogeneous. Precision is also important, because it gives confidence on the measured concentrations. An excellent precision is particularly important for analyses requiring high throughput, where a maximum of two measurements typically is allowed per sample.

Detection limits and precision values given in Table 1 are sufficient for all the types of applications.

TABLE 1: TYPICAL DETECTION LIMITS (3 sigma) AND PRECISION VALUES (1 sigma) FOR FIRE ASSAY LEAD BUTTONS

ELEMENT	Ag	Au	Ir	Pd	Pt	Rh	Ru	Bi	Cu	Ni	S	As	Co	Cr	Fe	Sb	Te	Tl
Typical DL [ppm]	0.004	0.08	0.18	0.004	0.08	0.004	0.01	0.005	0.005	0.05	0.3	0.3	0.03	0.2	0.08	0.3	0.2	0.005
Guaranteed DL [ppm]	<0.01	<0.15	<0.3	<0.01	<0.2	<0.01	<0.02	<0.02	<0.02	<0.2	<1.0	<1.0	<0.15	<0.5	<0.2	<0.5	<0.5	<0.02
Level [ppm]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
0.1	0.002			0.0015		0.0025	0.005											0.004
0.2	0.002			0.0025	0.025	0.0025	0.007						0.03		0.03			0.005
0.5	0.003	0.02	0.075	0.005	0.03	0.0035	0.015	0.015			0.2		0.04		0.03	0.07	0.07	0.009
1	0.004	0.03	0.085	0.0085	0.035	0.005	0.025	0.015		0.03	0.2	0.1	0.065		0.035	0.075	0.08	0.02
2	0.007	0.045	0.1	0.015	0.045	0.008	0.045	0.015		0.09	0.25	0.15	0.1		0.045	0.085	0.1	0.04
5	0.015	0.085	0.25	0.04	0.075	0.02	0.1	0.02	0.02	0.3	0.4	0.25	0.25		0.08	0.1	0.15	0.05
10	0.03	0.15	0.65	0.075	0.15	0.045	0.25	0.025	0.025	0.6	0.65	0.5			0.15	0.15	0.3	0.07
20	0.055	0.3		0.15	0.25			0.03	0.045	1	1	1			0.25	0.3		0.1
30	0.08	0.45			0.4			0.04	0.075	2	1.5				0.45	0.4		0.15
40	0.1	0.6			0.55			0.05	0.1	2.5	2				0.7	0.55		
50	0.15	0.7						0.06	0.15	3	2.5				0.95	0.7		
100	0.25	1.5						0.1	0.35	6.5	5					2		
200	0.55	3						0.25	1	15	10					8		
500	1.5	7						0.9	3.5	30	25							
1000									9.5	65	50							
2000									25	150	95							
5000									45	300	250							
10000											480							

Remarks : These data apply when homogeneous fire assay lead buttons are measured.
The precision given is typical performance. Guaranteed values will be 1.5 times higher.
DLs are calculated on a pure Pb sample and reproducibilities with typical homogeneous fire assay samples.
Guaranteed DLs are calculated at 95 % confidence limit.

Performance guarantee

Our company guarantees the precision shown in Table 1 using homogeneous samples and recommended sample preparation. The table will be updated as improvements are announced. Please contact your nearest Thermo Fisher Scientific representative for the most recent values or consult our Web site at www.thermo.com/oes.

The precision is calculated from the formula:

$$SD(1\sigma) = \pm \sqrt{\frac{\sum_{i=1}^{i=n} (X_i - \overline{X}_i)^2}{n-1}}$$

where:

X_i the individual readings

$\overline{X_i}$ the arithmetic mean of the individual readings

n the number of determinations

The DL (Detection Limit) is defined as three times the standard deviation of the background expressed in concentration units.

Accuracy

Precision is only a small part of providing good analyses. The most important factor is the accuracy and quality of the calibration standards. The use of optimal TRS windows considerably reduces spectral interferences, and improves the calibration by minimizing the necessary corrections. The right parameterization of the pre-burn spark by the CCS also helps improve the accuracy, in reducing matrix effects. Nevertheless, the accuracy still strongly depends on the quality and on the accepted concentrations of the calibration standards.

The following calibration curves show the excellent fits obtained for Au, Pt and Rh (Fig. 1, 2 and 3).

Testing the accuracy is not simple, as there are not many well referenced materials available for fire assay lead buttons. Table 2 shows the excellent accuracy achieved on PGMs with a calibration based on primary standards obtained by adding pure PGMs to pure lead.

The accuracy was tested on fire assay lead buttons prepared from certified SARM ore samples. The concentrations in the ores were back calculated from the concentrations measured in the lead buttons in order to compare the analysis results with the concentrations in the certified ores.

The accuracy on the PGMs is good considering the differences of compositions of the SARM (corresponding to geological materials and concentrator plant products) lead buttons and calibration samples. Matrix matching the standards to the samples or removing major impurities causing matrix effects (e.g. Cu, Ni, S…) from the samples would allow even better agreement between recommended and measured values.

Testing the accuracy for gold is even more difficult, due to the lack of well referenced materials. The accuracy was therefore tested on QC samples kindly provided by a South African gold mining company. Table 3 shows the excellent accuracy obtained on these samples.

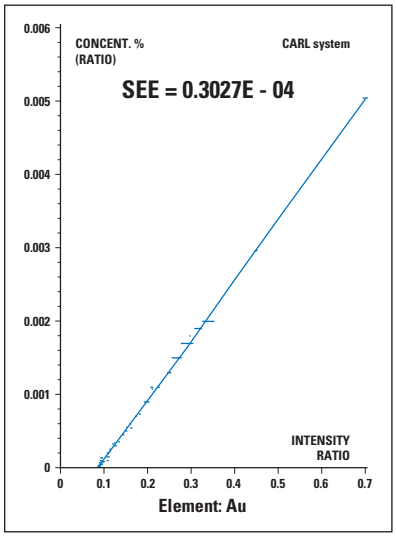


Figure 1

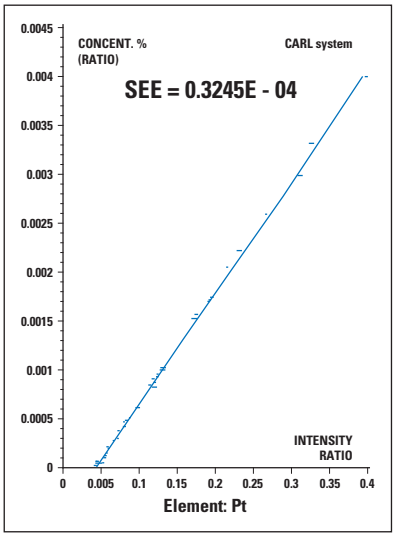


Figure 2

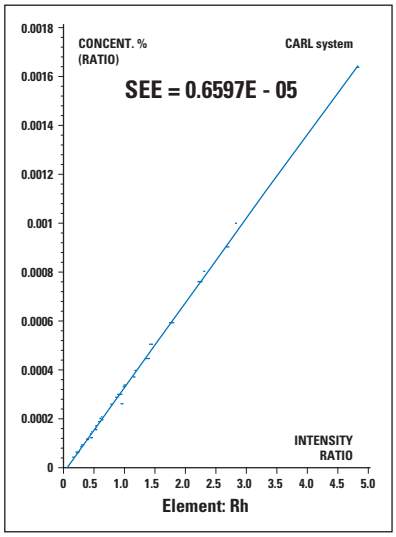


Figure 3

Table 2: Accuracy on PGMs

STANDARD	ORE TYPE	CONCENTRATION [PPM]	Pt	Pd	Rh	Ru
SARM 7B	Merensky Reef feed	Recommended	3.74	1.54	0.24	0.46
		Measured	3.49	1.20	0.25	0.44
SARM 65	UG-2 Layer chromitite feed	Recommended	2.64	1.28	0.522	0.853
		Measured	2.72	1.40	0.563	0.949
SARM 64	UG-2 Layer chromitite tail	Recommended	0.475	0.210	0.08	0.24
		Measured	0.434	0.183	0.08	0.23

Results from "Analytical Data from the Automated Fire Assay (FIFA) Process", P. Hofmeyr et al, presented at CMA 2003 Conference, Ottawa, Canada. Reproduced with the permission of IMP, South Africa.

Fire assay samples

The accuracy of PGMs determination was tested on lead buttons prepared from certified ore samples (Table 1) and the accuracy of gold determination on QC samples (Table 2).

Table 3: Accuracy on Au

SAMPLE	ESTABLISHED VALUE [PPM]	MEASURED VALUE [PPM]	DELTA %
A	0.11	0.13	18.18
B	0.22	0.22	0.00
C	0.52	0.51	1.92
D	1.00	1.06	6.00
E	2.9	2.95	1.72
F	11.4	12.0	5.26
G	24.9	25.1	0.80
H	106	104	1.89
I	425	417	1.88

From same source as Table 1.

Stability

Stability of the instrument is of the utmost importance when doing routine analysis. Together with high precision, it provides confidence in the analytical results. It also allows re-standardization at long time intervals, which is extremely important when high instrument availability is necessary. Long-term stability measured over 5 working days on a typical fire assay sample shows that the standard deviation achieved is below two times the precision value at the considered concentration level, which is excellent (see diagrams below).

Basic configuration

The ARL Fire Assay Analyzer in its basic configuration includes:

- Channels for Pb internal standards, for the precious metals Ag, Au, Ir, Pd, Pt, Rh and Ru, and for the most frequent impurities Bi, Cu, Ni and S
- One fire assay calibration (global or with your samples)
- A full optimization of the analytical method and calibrations, according to your production samples. For this purpose, homogeneous and matrix-matched calibration samples could have to be provided to us for optimal accuracy
- SUS samples covering our standard calibration ranges

As mentioned earlier, an evaluation of the performance of the method on the production samples and on the calibration samples is a pre-requisite for defining a configuration. Please contact your nearest Thermo Fisher Scientific office for more details about this.

Options

The evaluation performed by our company will help you define the optimal configuration for your ARL Fire Assay Analyzer. The most typical options are:

1. Additional elements

Other elements (As, Co, Cr, Fe, Sb, Te, Tl…) should be added to the basic configuration if they are present as impurities in the buttons, in order to compensate for interferences. Calibration standards and SUS samples including these elements may be required from the customer.

2. Additional calibrations

If lead buttons are prepared from different ores having significantly different compositions, several matrix-matched calibrations may be necessary for accurate analyses. Matrix-matched calibration samples may be required from the customer.

3. Automated analysis

The ARL Fire Assay Analyzer can be automated with the ARL SMS-2000 system, extremely well recognized in the other metals industries.

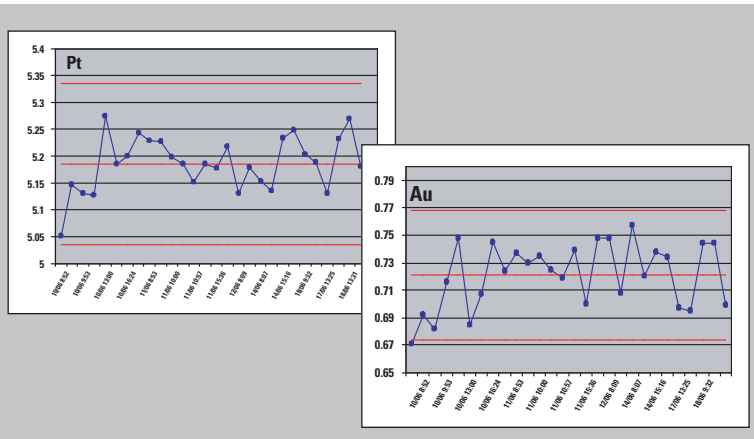
The samples can be brought by a transfer system on an analytical scale by the SMS-2000 and surfaced prior to the OE analysis. The analyzed samples can then be labeled and classified according to configurable criteria. The concentrations of the PMs measured in the lead buttons are then treated by the OXSAS software in order to calculate their concentrations in the ore in g/ton (ppm).

Remark on toxicity

When optimizing the CCS source condition, another important objective was to minimize the amount of produced lead vapors. Air measurements performed by an accredited laboratory showed that lead released in the air during intensive measurements performed over a full day is well below the international norms. For that reason, no device for toxic fumes suction is necessary for this application. A copy of the certificate is available upon request.

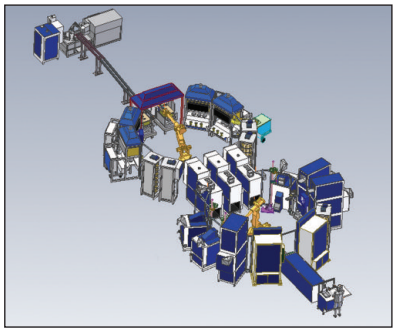
Long term stability

The standard deviation obtained over 5 days on a typical fire assay lead button is of the order of two times the precision at the corresponding concentrations (outer red lines). Concentrations are in ppm.

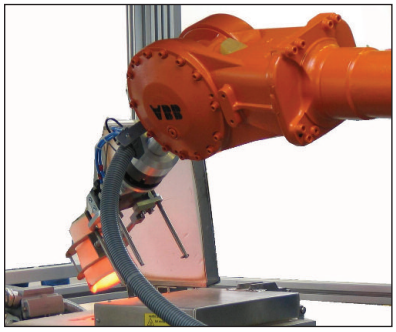


Automatic preparation of fire assay lead buttons

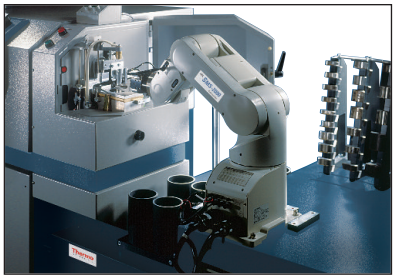
Fully automated fire assay laboratories can nowadays handle the process from raw ore to lead buttons. The ARL Fire Assay Analyzer can be adapted to such systems. Automatic preparation of lead buttons and analysis with the ARL Fire Assay Analyzer guarantee maximal reliability, highest throughput and performance suitable for all aspects of PGMs and Au mining industries (plant control, exploration and mining, metal accounting…). Moreover, personnel can be assigned to other tasks.



Line for sample preparation and OE analysis allowing a throughput of 1000 samples a day.(*)



Separator furnace: the melt obtained in the fusion furnace is poured by the handling robot into a furnace holding a heated separator crucible for the separation of the metallic lead and slag.(*)



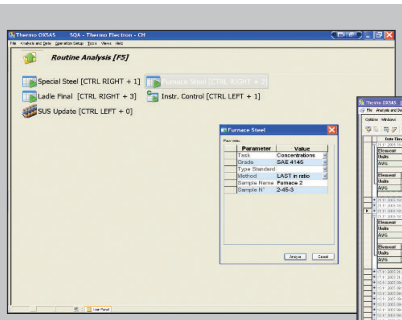
The lead button is automatically transported on a belt conveyor to the ARL SMS-2000. The button is automatically weighed on an electronic balance, surfaced in an automatic milling machine and finally analyzed with the ARL Fire Assay Analyzer.(*)

OXSAS Software: the most powerful operations made easy

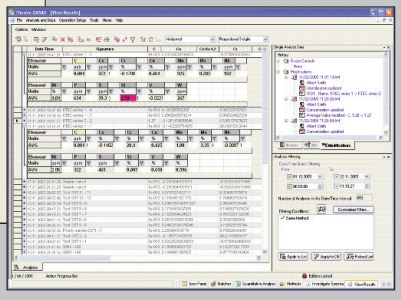
OXSAS software outperforms software that is currently available on the market today for metals analysis by OES. OXSAS provides virtually unlimited analytical capacity and flexibility and will therefore meet your needs throughout your instrument's lifetime:

- Triple navigation style: menus, tree and icons to accommodate individual preferences
- Simple one-click routine analysis launch
- Quantitative analysis using tasks with analysis parameter template
- Access to various functional levels through password protected user accounts allowing for secured operation
- One click access to recent analyses results, readily available for comparison in the analysis screen
- Full traceability

These are just a few of the many features contributing to the fast and easy routine operation of OXSAS.



OXSAS: Easy navigation and comfortable operation



OXSAS: An example of many different result presentations

The ARL Fire Assay Analyzer provides not only state-of-the-art technology, but also has all the total system features, which meet the critical needs of the precious metals producers:

- Unmatched hardware for stability and reliability
- Exceptional performance in detection limits, precision, accuracy and stability, all this in minimum analysis time
- Most advanced software technology
- Potential to cover your future analytical needs
- Easy operation by unskilled worker or research chemist
- Adaptable to the automatic Sample Manipulation System, ARL SMS-2000
- Adaptable to automatic fire assay laboratories
- Advanced technical/service support
- Laboratory accreditation guidance
- Immediate access to parts inventory

All these features allow you to optimize your productivity and to achieve the shortest payback times:

- Your investment costs are reduced by the suppression of costly traditional analytical instruments
- Your investment costs are reduced thanks to the exceptional and widely recognized instrument lifetime and to the continuous upgrade possibilities (software and hardware)
- Your investment costs are reduced with the capability of the instrument to cover your potential future needs
- Your production costs are reduced by more accurate and reproducible analyses that are available faster
- Your production costs are reduced by the increased instrument availability thanks to its high stability and drift corrections being less frequently required
- Your operating and maintenance costs are low, due to a small consumption of drift correction samples and simple maintenance
- Your overall cost management is reduced by optimum utilization of materials and extremely low running costs compared to other methods

With its over 70 years of experience and history of innovative technology, our company has become the world leader in OES metals analysis. We work with our customers to improve the efficiency of their analytical tasks and thereby increase productivity.

(*) This picture shows the FIFA system and is reproduced with permission from Herzog, Germany and IMP, South Africa.

Specifications for the ARL Fire Assay Analyzer

Spectrometer	
Spectrometer design:	One meter, Paschen-Runge vacuum polychromator made of cast iron and temperature controlled to ± 0.1° at 38° C. Maximum 60 channels
Sample stand:	With self-contained, recirculating coolant system. Argon flushed table
Gratings:	Spectrometer provided with a 1080 gr/mm grating
Resolution:	Dependent on secondary slit, and spectral order
Slit widths:	Primary slit: 20 µm; secondary slits: 20, 25, 37, 50 mm
Photomultiplier tubes:	Ø 28 mm, 10-stage side-on-type, MgF_2 , UV glass, borosilicate glass or synthetic silica windows
CCS and TRS	Current Controlled Source (CCS) and Time Resolved Spectroscopy (TRS)
Electronics	
Spectrometer control:	ARL MMB 386 Microprocessor utilizing CMOS technology with Status Measuring Card. A/D converters and attenuators included for each channel. Attenuators with 41 steps each. Up to 12 programmable attenuators available as an option. Dynamic range of measuring electronics proportional to measuring time, typically $2*10^6$ counts/sec
Enclosure:	Built-in dust protection with high capacity cooling fans
Requirements	
Environmental requirements:	Ambient temperature 16-25°C (61-77°F); maximum rate of change 5° C/hour. Relative humidity: 20-80%
Electrical requirements:	Voltage 230 V (+10 %/-15 %), single-phase with protective ground (5kVA regulator required if fluctuations exceed ±10 %). Current: 12 A; power: 2.6 kVA; frequency: 50 or 60 Hz; grounding < 1Ω. In conformity to European directives (89/392/EEC Machinery), (73/23/EEC Low voltage material), (89/336/EEC Electromagnetic compatibility)
Argon requirements:	99.998 %. Optional argon purifier available
Dimensions and weight	
Dimensions:	1385 mm (55 in.) wide excluding excitation stand; 1690 mm (67 in.) wide including excitation stand; 1220 mm (48 in.) high, 910 mm (36 in.) deep;
Total system weight:	540 kg. (1190 lb) approximately
Accessories and options	Spark-DAT, spark Data Acquisition and Treatment Stand upgrade for semi-automatic operation ARL SMS-2000 for automatic operation Argon purification systems Voltage stabilization systems Uninterruptible Power Supply (UPS) Electronic scale Data communication software options Analytical results processing software options

Laboratory Solutions Backed by Worldwide Service and Support

Tap our expertise throughout the life of your instrument. Thermo Scientific Services extends its support throughout our worldwide network of highly trained and certified engineers who are experts in laboratory technologies and applications. Put our team of experts to work for you in a range of disciplines – from system installation, training and technical support, to complete asset management and regulatory compliance consulting. Improve your productivity and lower the cost of instrument ownership through our product support services. Maximize uptime while eliminating the uncontrollable cost of unplanned maintenance and repairs. When it's time to enhance your system, we also offer certified parts and a range of accessories and consumables suited to your application.

To learn more about our products and comprehensive service offerings, visit us at www.thermoscientific.com.

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