

Serie AL-204

Aluminium analyzer

EQUILAB AL-204

User Manual v2_23

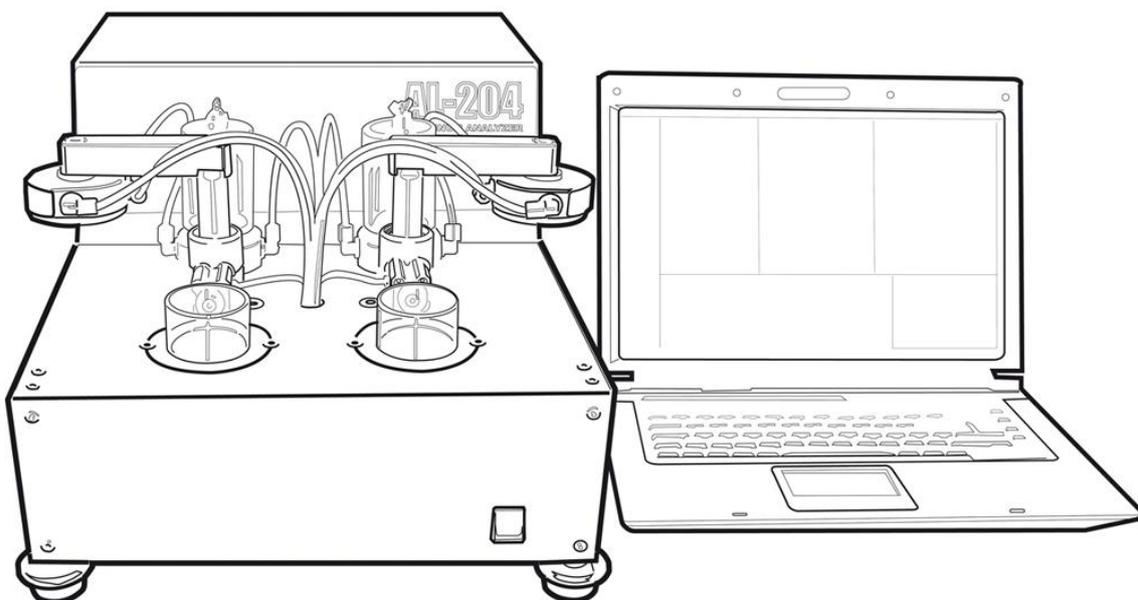


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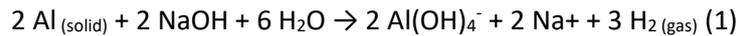
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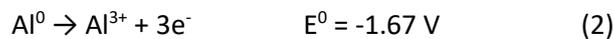
1. Introduction

The AL-204 is a compact automatic instrument capable of analyzing metallic aluminum by gasometry in less than 15 minutes, improving the reproducibility and accuracy of the analysis according to different national and international standards.

The analysis is carried out through the continuous monitoring of the pressure increase generated by the hydrogen produced in the oxidation reaction of metallic aluminum in a basic medium according to the following chemical reaction:



Aluminum is a highly reducing metal, just look at the standard potential of the oxidation half-reaction:



This high standard potential indicates that the oxidation half-reaction is thermodynamically very favorable, resulting under normal conditions in the formation of a thin surface layer of alumina (Al₂O₃) that passivates, protecting the metal from complete corrosion. However, in the presence of a strong base, alumina dissolves, favoring complete attack of the aluminum sample.

The AL-204 analyzer is a user-friendly equipment that allows us to comfortably carry out all the necessary operations to perform a quantitative analysis of metallic aluminum: execution of the analysis sequence, data entry, calibration, result management; as well as real-time monitoring of all process parameters and analyzer status through high-resolution tables and graphs, which facilitates diagnostic and analysis tasks.

1.1. Warranty

All equipment manufactured by Equilab will have a warranty against any manufacturing defect, both in the materials and in the workmanship, for a period of 12 months from the date of installation or 18 months from the date of sale (whichever occurs first).

This warranty does not cover items that, while part of the equipment, are considered consumable (expendable) such as tubes, glass vessels, and those elements whose wear and tear due to use make them considered consumable.

In no case does this warranty cover any problem whose cause is clearly improper use of the equipment.

Causes of warranty loss:

Tampering with the equipment during its warranty period by personnel not authorized by **Equilab**.

Using the equipment for any task other than for which it has been designed.

Using products not recommended by **Equilab**.

Warranty limitations:

The only obligation of **Equilab** regarding this warranty will be limited to the replacement or repair of any parts of the equipment that, in our judgment, were defective and had presented failures during the warranty period. This obligation excludes failures due to accidents, abuse, negligence, or use without regard to the instructions and precautions provided and recommended by **Equilab** for the correct use of this machine.

Under no circumstances will **Equilab** be responsible for any damage that the equipment could generate, including the loss of profit due to interruptions in production processes derived from failures in the equipment.

1.2. Equipment description

The AL-204 analyzer is designed for the rapid and accurate determination of aluminum concentration in slags, monitoring the pressure of the gases resulting from the attack of the sample with sodium hydroxide (NaOH). The equipment incorporates **2 independent channels** that allow the simultaneous analysis of 2 samples or the improvement of reproducibility by performing replicas of the same sample simultaneously.

In the following figure, the most relevant elements of the AL-204 analyzer are shown.

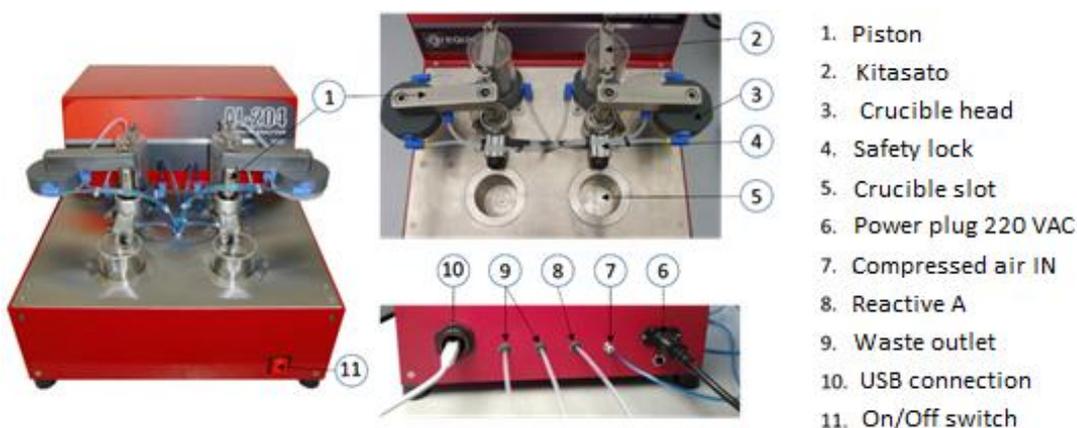


Fig. 1-1. (Left) Front view of the analyzer; (top right) top view of the analyzer; and (bottom right) rear view.

The analyzer consists of a thermostated housing (5) where the crucible with the sample is placed once the analysis sequence has started, the crucible head (3) seals the circuit through a pneumatic piston (1), maintaining a constant volume and temperature. During the reaction, it's normal for foams or water droplets to enter the pressurized circuit, for this, the equipment has a Kitasato trap (2) that prevents them from circulating to the thermodynamic compensators. The safety closure (4) keeps the crucible immobile, favoring sealing and opening the crucible.

The rest of the elements of the equipment correspond to (6) the 220 VAC power socket; (7) the inlet of compressed air essential for the proper functioning of the pneumatic elements of the system; (8) the inlet of reagent A (NaOH); (9) the waste outlet for the evacuation of compressed gases inside the pneumatic circuits; (10) the USB connection between the analyzer and the control computer; and (11) the power switch.

1.3. Specifications of the AL-204 Analyzer

Measurement range: 0 to 100% (max. 500 mg of Al).

Accuracy: $\pm 10\%$ for concentrations $> 10\%$ aluminum.

Sample weight: from 0.15 to 3 g.

Resolution: 0.01%.

Results expression: % aluminum.

Analysis time: programmable from 5 minutes.

Reagents: reagent A (sodium hydroxide 20% – 30%).

Working temperature: from 10°C to 50°C.

Power supply: 220 VAC, 50 Hz, 0.5 kW.

Pneumatic pressure: instrumentation air max. 60 psi (4.5 kg/cm²).

Weight: 30 kg

Dimensions (width x length x height): 360 x 275 x 550 mm

1.4. Symbols.



Warning



Relevant information



Corrosive



RAEE

1.5. List of materials delivered with the equipment

- AL-204 Analyzer.
- PC with Windows.
- Inkjet Printer (Optional).
- Glass Crucibles (6) (4x AL-144; 2x AL-167).
- Stirrers (4 x EQFX-208).
- Reactant A Canister.
- Waste B Canister.
- 4 mm gas connection tube 2 m (AL-138).
- Piston Seal Joints (2xAL 140).
- Instruction Manual (Electronic Format).
- EQUILAB AI 204-V2020 Application.
- USB Cable and 220 VAC Power Cable.

2. Installation

2.1. Packaging for transport



Before moving the equipment, remember that it is heavy and could cause personal injuries in case of falling or mishandling.

The equipment will be sent in packaging designed for its transport, in which it will be immobilized to avoid damages during the journey. The box is made of a phytosanitary wooden pallet where the equipment will be fixed with straps. It will be covered with a triple-reinforced cardboard box with foam and corner protectors to prevent damage to the equipment until it reaches the user.

Do not attempt to break the cardboard box in case you need to repack the equipment later.

2.2. Reception of the equipment and placement

As a first measure, a visual inspection of the packaging will be performed to ensure that the equipment has not suffered any damage during transport.

If any damage to the packaging is detected, immediately contact your supplier before unpacking the equipment.



Keep the packaging for some time in case any anomalies are detected and it is necessary to return the equipment to your supplier.

If the packaging is in normal conditions, proceed to unpack the equipment. If the equipment is to be stored for some time before installation, store it in a dry place away from heat sources and away from areas where it may be hit.

Check that the supplied material is complete and that the accessories requested with the equipment have been received.

2.3. Equipment installation

The installation of the analyzer will be carried out on an elevated and flat surface, taking into account that there is a 220 VAC power socket.

When placing the analyzer on the worktable, ensure that the back of the analyzer is separated from any obstacle by at least 30 cm so that the power supply, reactant A inlet, and gas pressure and purge vapor outlets from the crucibles (Waste) can be connected.



Fig. 2-1. Rear view of the analyzer, showing the inlet of compressed air and reagent, as well as the outlet of the purge gases from the crucibles. and the outlet of the purge gases from the crucibles.

- The 220 VAC power supply must be earthed, as failure to use an earthed power supply could adversely affect the operation of the analyzer. not using an earthed power supply could have a negative influence on the operation of the analyzer, causing of the analyzer, producing instabilities in the measurement, as well as interferences in the analyzer's control signals. control signals of the analyzer.
- The instrumentation air connection, maximum 60 psi (4.2 kg/cm²), shall be connected
- using a 4 mm diameter tubing.
- The reagent tube A shall be inserted into the drum containing the reagent.
- The purge tubes of crucibles A and B shall be inserted into an expansion canister to avoid splashing. to avoid possible splashing. If the aluminum slag generates ammoniacal gases, the use of an ammonia canister is recommended. the use of an expansion vessel with a solution of an acid which retains these gases is recommended. retains these gases.

2.4. Printer Connection

The presentation of the results of the analyzer is done through graphical representation on the screen, the database table (see sections 4.3 and 4.4), and an external printer. The printer must be connected directly to the control computer of the analyzer and to a 220 VAC power socket.

2.5. Placement of Liquid Reagents

For its operation, the analyzer requires two containers located outside of it. One of the containers holds the basic solution necessary for the reaction (reactant A), and a container or expansion bin collects the purge gases from the crucibles.



Fig. 2-2. View of reagent A tanks and purge gas expansion vessel.

2.6. Waste Management

For the correct disposal of the equipment, the corresponding legal regulations of the country in which they are located must be complied with. Within the European Union, the regulation for the disposal of this equipment is regulated in the EU Directive 2012/19/EU on waste electrical and electronic equipment (WEEE).¹



¹ Nº RII-AEE 11655

3. Operation

3.1. Operating Principle

The AL-204 analyzer bases its operation on the continuous monitoring of the gas produced in a chemical reaction known as gasometry. This analytical technique allows relating the generation of a specific gas released in a chemical reaction with the concentration of an element (in this case, metallic aluminum²) in a constant volume. The stoichiometry of the oxidation reaction of metallic aluminum to generate gaseous hydrogen, 2 moles of aluminum are quantitatively oxidized producing 3 moles of hydrogen (2 to 3 ratio). This reaction is thermodynamically and kinetically very favorable, allowing a rapid and efficient analysis.

To carry out the analysis of the sample, it is placed in a thick-walled glass crucible (to withstand the pressure increase generated during the oxidation reaction) along with a magnetic bar. After a brief adjustment of the zero pressure, the dosing of reagent A is carried out using a peristaltic pump, which initiates the oxidation reaction of the aluminum. Then, magnetic agitation is activated to favor and homogenize the oxidation reaction while reaching the working temperature previously set by the user (typically between 40°C and 60°C). The process is monitored for a determined time or until the reaction kinetics indicates that it has concluded; at that moment, the purity value in % of metallic aluminum of the sample is obtained and recorded.

The equipment has 2 identical channels that operate individually, allowing simultaneous, sequential, or individual analyses.

The following diagram shows a simplified diagram of the aluminum analyzer:

- 1 the result of the analysis is presented as the percentage purity of metallic aluminum in the sample. The non-metallic aluminum species present in the sample are not quantified in this type of analysis since they do not oxidize (they are normally in their maximum oxidation state) and, consequently, do not generate hydrogen.

² The result of the analysis is presented as the percentage of purity of metallic aluminum in the sample. The **non-metallic aluminum** species present in the sample **are not quantified**. They do not oxidize (they are normally in their maximum oxidation state) and, consequently, do not generate hydrogen.

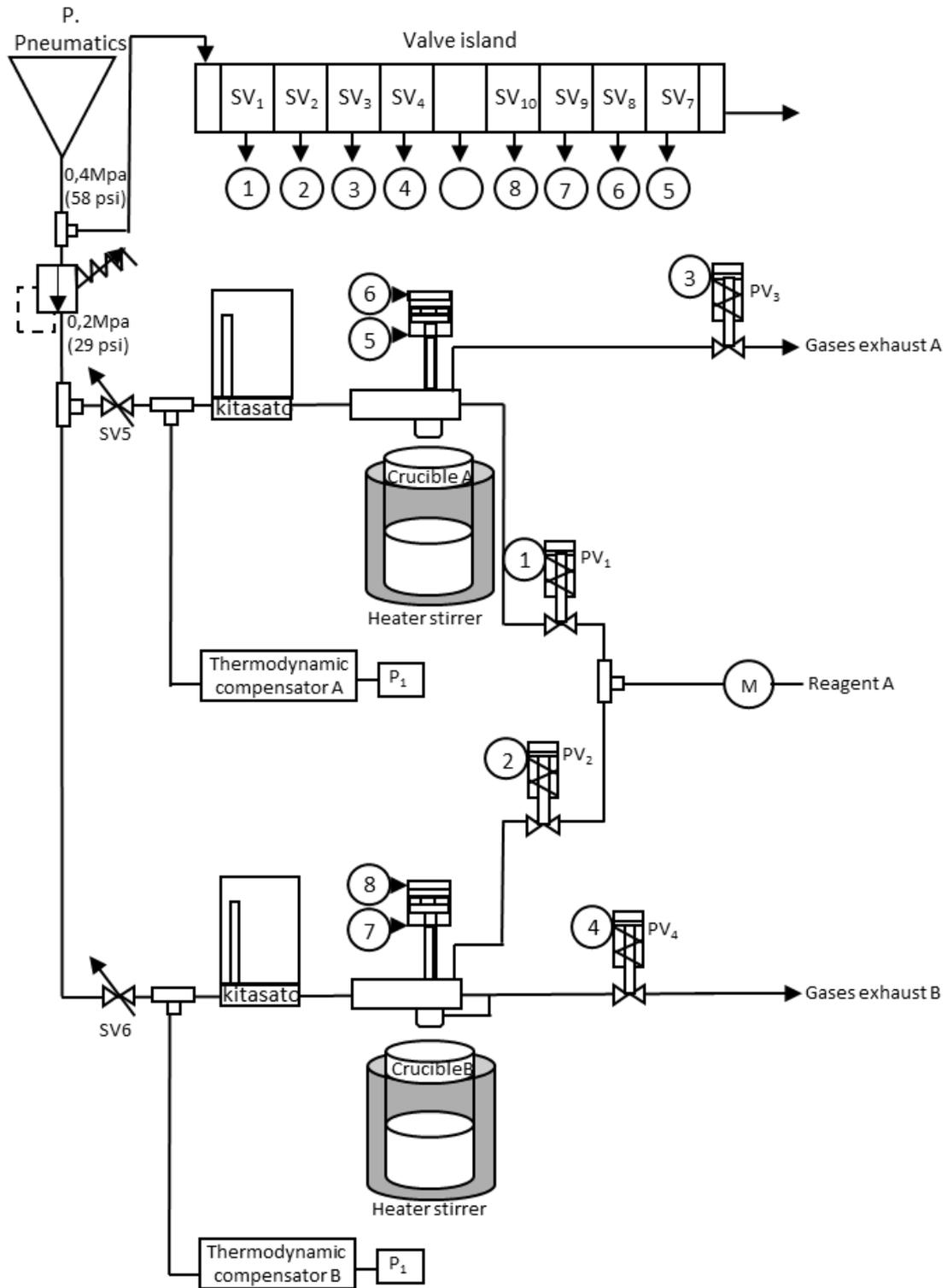


Fig. 3-1. Hydropneumatic diagram AL-204.

3.2. Considerations Before Sample Analysis

The **aluminum analyzer AL-204** is a versatile equipment capable of measuring the concentration of metallic aluminum in a wide range of samples and purities. However, we must consider a series of prior considerations to avoid errors in measuring the purity of the aluminum and potential damages to the instrumentation.

When starting to use the equipment for the first time or after a period of non-use, it is necessary to take into account the following points for optimal start-up and operation of the equipment:

1. Turn on the equipment and wait at least 30 minutes before starting the analysis to achieve thermal stability of the crucibles and the pneumatic circuit's thermodynamic compensators at the working temperature.
2. Open the compressed air and check that the pressure is below the maximum allowed.
3. Ensure that the reagent A tank has enough quantity to carry out the planned analyses (approximately 20 – 30 mL per sample).
4. Check that the pressure values on the thermodynamic compensators are close to zero millibars before starting the analysis.
5. Check the operation of the different elements of the measuring equipment. To do so, follow the following steps: (use manual functions, see section 4.1.6).
 - a. Open and close the pistons with the crucible placed in its housing.
 - b. With the pistons closed, activate the peristaltic pump until the reagent begins to fill the crucible.
 - c. With the pistons closed, check the activation of the (i) OutGas pinch and (ii) Pneumatic pressure valves. By activating both the system is pressurized, deactivate the pneumatic pressure valve and then the OutGas valve to release the system pressure.
 - d. Perform a leak test (see section 4.1.5).



If any of the previous tests fail, check the correct operation of the equipment, see section 5

6. Set the desired analysis parameters in the "Parameters" option (see section 4.1.2).

Once the analyzer is stabilized and before starting the analyses, we must consider the type of sample to be analyzed, as the analysis parameters must be suitable to obtain the best results in terms of precision and accuracy.

In an analysis of a pure aluminum standard (close to 100% purity), where the reaction occurs rapidly, it is not necessary to use long analysis times. About 10 minutes is sufficient, and for safety reasons, it is not recommended to weigh more than 0.5 g of the sample.

In the analysis of aluminum slag of 60% purity, given the high concentration of metallic aluminum, the reaction is fast, and usually, it is not necessary for an analysis time exceeding 15 minutes. However, this depends on the granulometry of the sample: fine samples (<100 μm) oxidize rapidly, while those presenting larger grain size require longer analysis times. It is recommended to use quantities less than 1 g of sample.

For aluminum slag with purity values lower than 20%, a longer analysis time is required, and depending on the granulometry, they may need reaction times exceeding 30 minutes. It is recommended to weigh quantities of up to 3 grams.



!!!For safety reasons, it is strictly prohibited to exceed the maximum sample mass, according to the analyzer specifications.!!!

3.3. Sample Analysis

To carry out the analysis of the sample, a known quantity of the sample is added to crucible A or B along with a magnetic bar. Next, the sample data: reference and mass (1) are entered, and then press **ANALYZE** (2) on the graphical window corresponding to the crucible being used for that analysis.

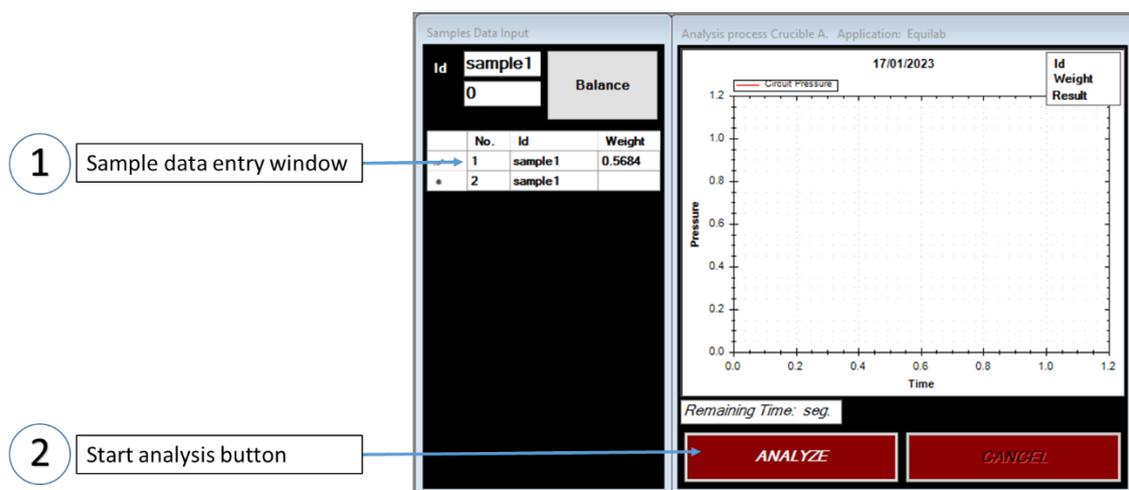


Fig. 3-2. Steps to initiate the analysis process.

It is very important to emphasize before analyzing a sample that it must be ground to ensure uniformity and facilitate chemical attack. This ensures a shorter analysis time and better reproducibility in the results.

Some samples produce a large amount of foam when reacting with the base. In these cases, it is advisable to add some type of antifoaming agent to prevent blockages in the pneumatic circuit.

The result will be graphically represented in the window that shows the analysis process, and the result will be recorded in the data window once the analysis is finished.

3.4. Analysis sequence

Aluminum purity analysis is an automatic process that the **AL-204 analyzer** performs from sealing the crucible, to pressurizing the system, to obtaining the sample purity result. The following sequence describes each of the stages of the analysis process carried out by the aluminum analyzer.

1. **Closing the crucible:** The system seals the crucible using a pneumatic piston. The system checks the movement of the piston and the correct position of the crucible. *The analysis is aborted if there are any anomaly.*
2. **Initial pressure check:** The initial pressure value at the beginning of the sequence is verified, correcting the value to 0 mbar.
3. **Heating:** During the whole analysis, the heating system maintains the crucible and the thermodynamic compensator at the set temperature.
4. **Reagent dosage:** The equipment doses, by means of the peristaltic pump, the addition of approximately 20 mL of reagent on the sample. *The volume of dosed solution can be modified depending on the type and nature of the sample changing "reagent dosing time" parameter. This time affects the blank value, so it must be recalculated and corrected.*



The volume of dosed dissolution can be modified depending on the type and nature of the sample through the parameter "reagent dosing time", this time affects the blank value and must be recalculated and corrected.

5. **Reaction and quantification:** At the same moment that the addition of the reagent to the sample begins, the pressure monitoring and quantification starts. During this step, the sample is stirred and heated to get a homogeneous reaction. The analysis time is variable depending on the kinetics of the oxidation reaction of the sample; this cannot be less than 5 min and can be extended up to 60 minutes.

6. **Depressurization:** The valves are switched to purge the pressurized gases on the circuit and recover atmospheric pressure in the crucible, thus eliminating possible splashes when the piston rises.
7. **End of the process:** Once the analysis has been completed, the operator must double click on the graph so that the piston goes up (or manually from the Manual functions menu, see section 4.1.6). *If the crucible is pressurized, this command is ignored for safety reasons.*

4. User interface

The analyzer is controlled by a computer application responsible for analysis automatization; data collection; processing and calculation of aluminum concentration; the estimation of the volume of gas generated; and recording them in a database.

This application has a user interface that is made up of a set of windows as shown in the following image.

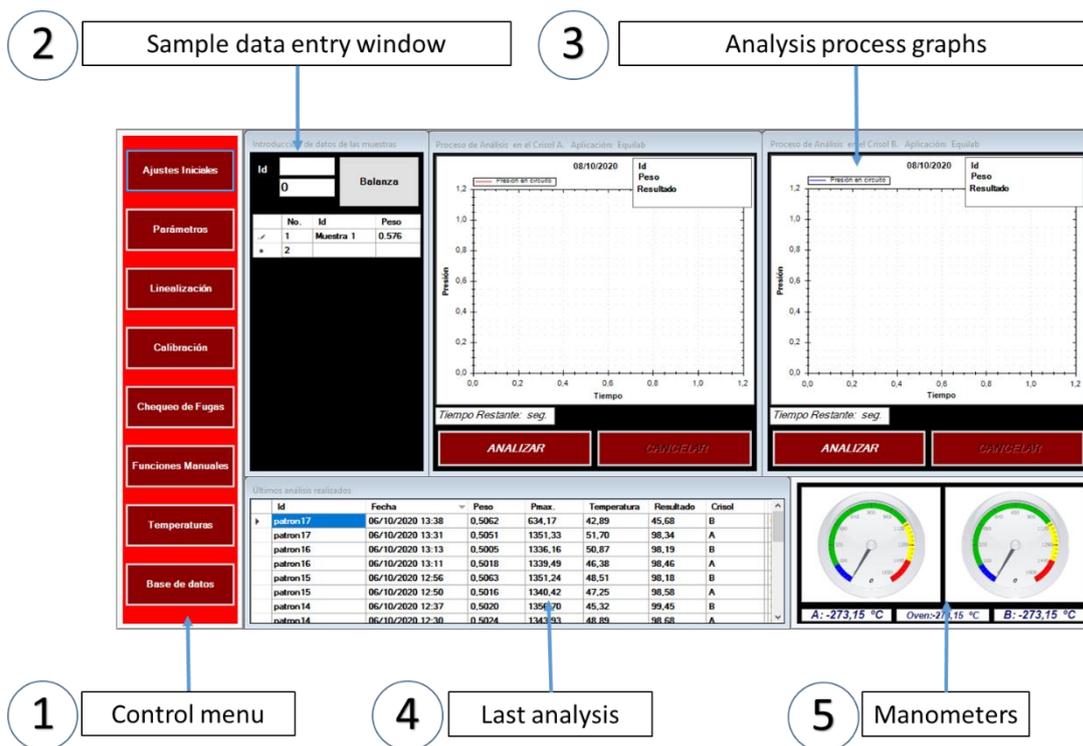


Fig. 4-1. Initial step to initiate the analysis.

The user can modify the design of this interface by varying the sizes and placement of the individual windows until the desired appearance is achieved. Once the appearance has been updated, it will be fixed by double clicking on the red background of the control's menu box.

Below is a brief description of each of the windows displayed in the user interface.

- 1. Control menu:** red background window, we can find the different buttons to access to the settings and functions of the analyzer (see section 4.1). **Error! No se encuentra el origen de la referencia.**
- 2. Sample data entry:** this window is used to enter the sample data: reference and mass.

3. **Representations of the analysis process:** made up of two analog windows, one for each crucible. In this graph the pressure evolution is depicted during the analysis time, the sample data (reference and mass) and the result are shown in real time.
4. **Last analysis:** the samples history of last two days is shown in this window, (a more detailed review of the database can be consulted by clicking the Database button in the control charts window, see section 4.1).
5. **Manometer:** this window shows the instantaneous pressure and temperature in each crucible.

4.1. Control menu

The buttons that appear in this window allow access to the configuration of all the equipment parameters, as well as the control of the different functionalities of the system.

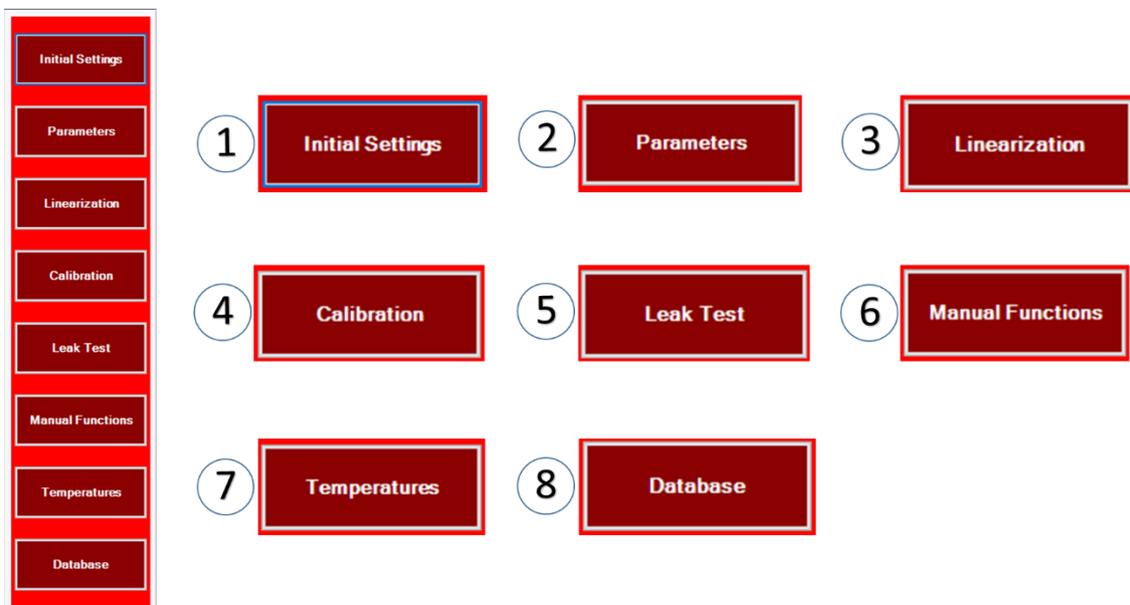


Fig. 4-2. Control menu. Options detail.

Simply click on the desired option to access the corresponding window. The functionalities of each equipment configuration and operation option are described individually below.

4.1.1. Initial settings.

To access the initial settings window, press the button (1). This option shows the different analyzer parameters (thermal characterization and pressure transducer parameters). Unattended modification of these parameters may cause the analyzer to malfunction. The

equipment is shipped with all the parameters adjusted for correct operation and must not be modified without the supervision of the technician.

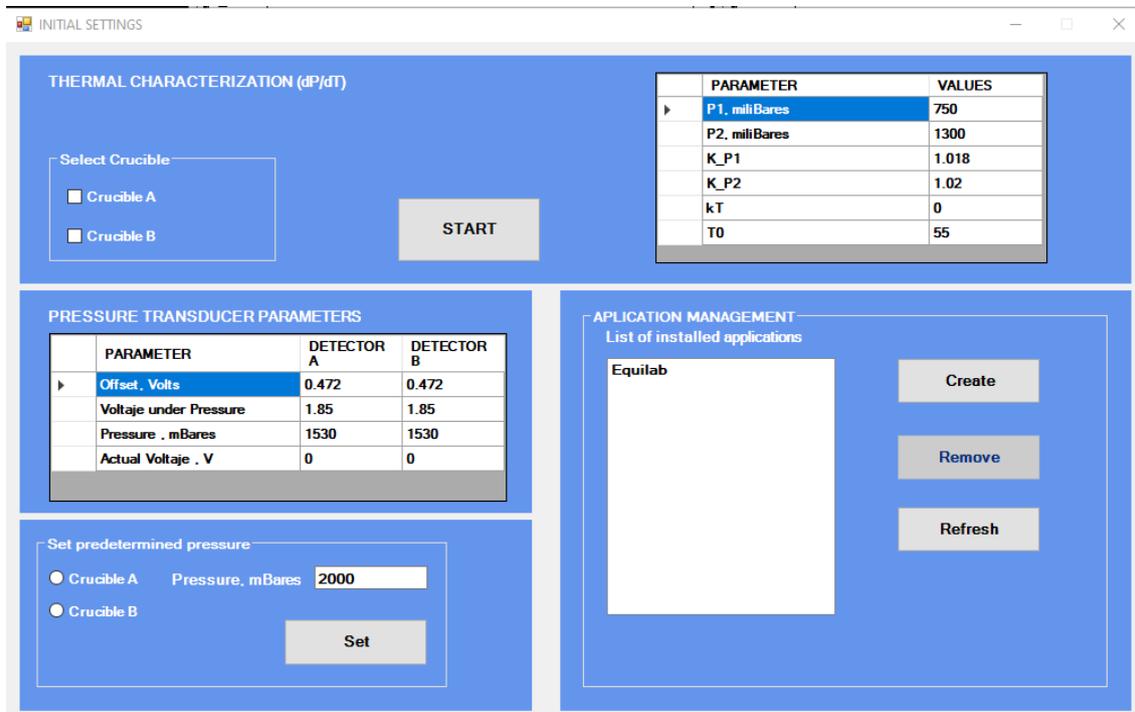


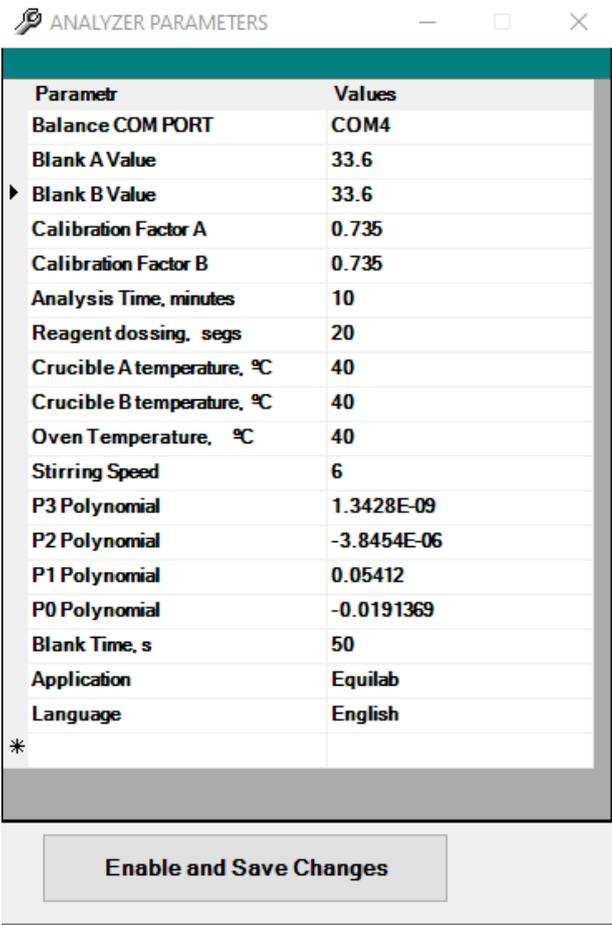
Fig. 4-3. Initial settings window.

Additionally, in this initial adjustment form, the management of different work profiles "Application management" can be carried out. Thus, as many different profiles can be created as different configurations are used for the analysis of different samples.

By default, the only application installed is "Equilab"; to create a new application we will only press the "Create" button and enter the name of the new application. Once accepted, we must press "Refresh" button to update the list, and the new application appear on the list. This application can be modified individually as indicated in the following section "Parameters".

4.1.2. Parameters.

To access this configuration window, press the button (2). This window shows the current parameters of the analyzer and allows the modification of the analyzer constants, as well as the active application / profile.



Parametr	Values
Balance COM PORT	COM4
Blank A Value	33.6
▶ Blank B Value	33.6
Calibration Factor A	0.735
Calibration Factor B	0.735
Analysis Time, minutes	10
Reagent dosing, secs	20
Crucible A temperature, °C	40
Crucible B temperature, °C	40
Oven Temperature, °C	40
Stirring Speed	6
P3 Polynomial	1.3428E-09
P2 Polynomial	-3.8454E-06
P1 Polynomial	0.05412
P0 Polynomial	-0.0191369
Blank Time, s	50
Application	Equilab
Language	English
*	

Enable and Save Changes

Fig. 4-4. AL-204 Analyzer configuration parameters.

In order to update the new parameters values it is necessary to click on the "Activate and save changes" button. The changes will be stored in the active application or profile and will be updated in the software for future analysis.

- **Scale COM PORT:** specifies the port of the computer to which the scale is connected.
- The scale must be connected as follow:
 - Speed: 9600 baud rate
 - Bits: 7 bits
 - Parity: Odd
 - Stop bits: 1
 - Handshake: RTS
- **Blank value:** this value in mbar is obtained by averaging the results for a series of blanks (analysis without sample). The blank value corresponds exclusively to the pressure increase in the system that happens during reagent dosing and the water vapor pressure.

- **Calibration factor:** shows the calibration coefficient for each channel. It can be modified directly on the table, or using the calibration procedures enabled in the program (see section 4.1.4). The use of the automatic calibration procedure is recommended.
- **Analysis time:** expressed in minutes, it is the duration of one analysis, including the dosing time. It is programmable by the user.
- Recommended 10 to 15 min depending on the sample (see section 3.2).
- **Reagent dispenser:** expressed in seconds, time in which the peristaltic pump is activated for the dose of reagent A. It is programmable by the user.
- Recommended 20 s.
- **Crucible A temperature:** sets the working temperature of crucible A in °C. It significantly influences the rate of reaction. It is programmable by the user.
- Recommended 40°C.
- **Crucible B temperature:** sets the working temperature of crucible B in °C. It significantly influences the rate of reaction. It is programmable by the user.
- Recommended 40°C.
- **Oven temperature:** sets the working temperature of the thermodynamic compensators. It is established by the user.
- Recommended 40°C.
- **Stirring speed:** sets the speed of rotation of the magnetic bars of the sample. It can be modified between 1 and 9.
- Recommended 5.
- **P3/P2/P1/P0 Polynomial:** they are obtained as a result of the fitting process to a cubic function. This function relates the purity of aluminum with the pressure in the entire response range of the analyzer (0 - 100%). A detailed description of the process for obtaining these coefficients is included in section 4.1.3.
- **Blank time:** expressed in seconds, the equipment balances the temperature of the circuit before starting the analysis during the time.
- Recommended 50 s.
- **Application:** allows you to select the desired profile with which to carry out the analysis.
- **Language:** allows you to select the desired language (Spanish/English).



Remember that any change in these parameters will only become effective by pressing the "Activate and save changes" button.

4.1.3. Linearization

The **AL-204** equipment bases its operation on the continuous monitoring of the pressure during the chemical reaction between aluminum and the reagent. This process, from the point of view of the mathematical relationship between aluminum concentration and pressure transducer device, is non-linear, especially for low aluminum concentration values. Therefore, **it is essential to establish the unequivocal relationship between the aluminum concentration and the analytical signal used**, the pressure in mbar.

The analyzer uses the linearization process to obtain the best-fit equation between the equipment's response and the experimental values, providing a robust mathematical function that establishes the proportionality between the aluminum concentration and the pressure. The user must carry out the linearization if any anomaly is detected in the calibration of the equipment, or after having replaced the measurement detector due to failure or updating. The equipment response linearization procedure is described below.

To carry out the linearization process of the analyzer,³ the first thing is to select a sample of known aluminum concentration or an aluminum standard. To obtain the different points of the calibration, different amounts of the standard will be used, in such a way that the entire pressure range is covered from zero to the saturation zone (red zone on the manometer). To linearize it is recommended to make two or three replicates of each of the following masses of an aluminum standard with at least 99% purity.

- 0.10 g.
- 0.20 g.
- 0.35 g.
- 0.50 g.

Once the analysis is finished (see section 3.3), we open the linearization window by pressing the corresponding button (3) in the control menu.

³ Linearization must be performed with the analyzer in steady state, for at least 60 minutes after it has been started.

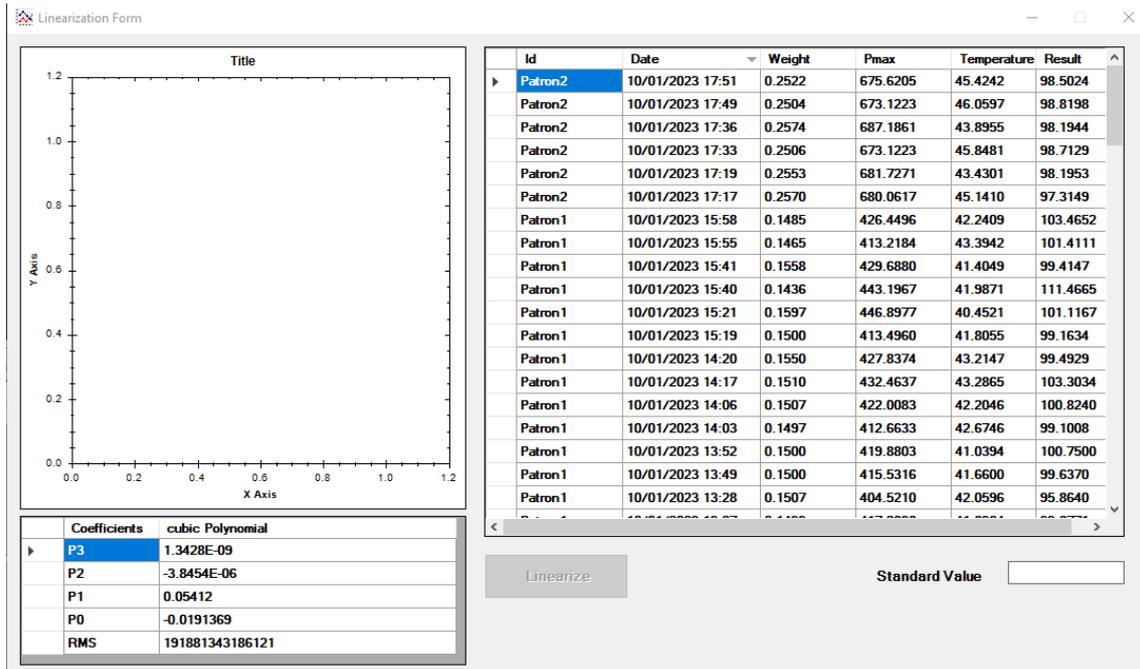


Fig. 4-5. AL-204 Analyzer linearization window.

In the table shown to the right of this window we will select the desired results for each crucible A or B (with the help of the mouse and the CTRL key)⁴ and introduce the value of the standard in the text box "Value of Standard" (for example: 99 for a standard of 99% purity of aluminum).

By pressing the "**Linearize**" button, the process of calculating the coefficients begins. Once the adjustment process to a polynomial equation of degree 3 is finished, a new pop-up window shows the graph of the calibration line where the pairs [pressure, concentration] used for the linearization process are interpolated:

⁴ It is possible to obtain some result "outlier" that is not useful for the linearization, so it will not be selected for the linearization process.

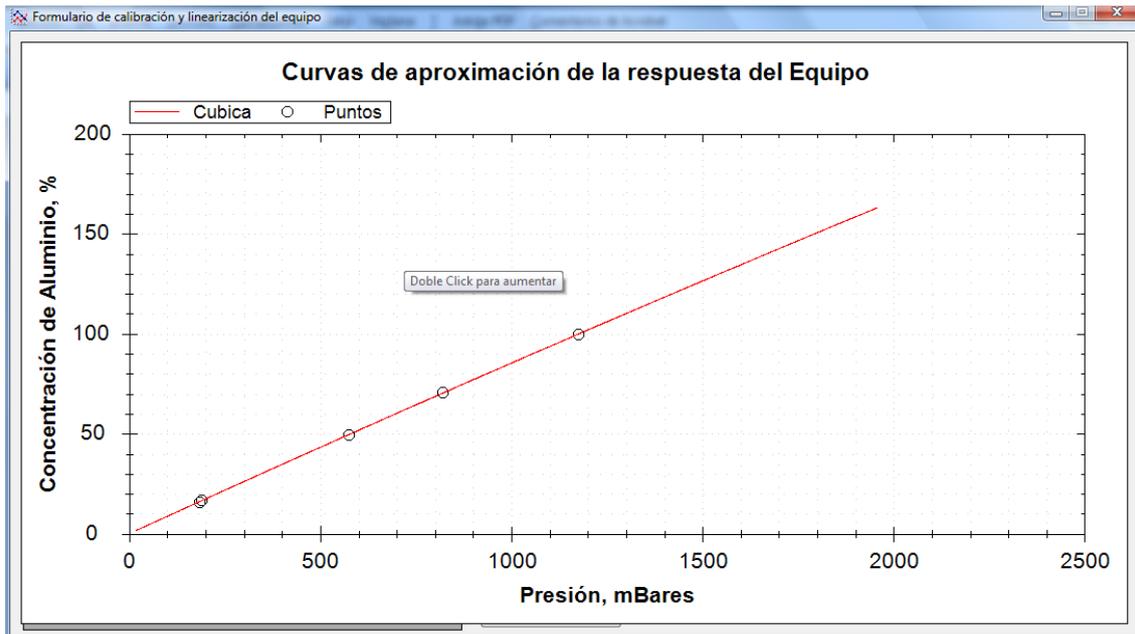


Fig. 4-6. AL-204 Analyzer linearization process result.

Double-clicking on the graphic window closes it, returning to the previous window (see Fig. 4-5) in which the new coefficients are shown (bottom left). We can accept the new coefficients by clicking on the “Apply” button, or discard them by clicking on “Discard”. In either case, the linearization window will close and we will return to the main interface window.

4.1.4. Calibration

Once the equipment has been linearized, which is a slow process that requires a large number of measurements to obtain a good fit, the equipment can be calibrated using a simple and fast procedure using a single aluminum concentration value. The equipment calibration process can be performed using manual or automatic mode.

- Manual calibration:

The manual calibration procedure consists of modifying the “Calibration Factor A or B” directly in the analyzer parameters. To do this, we will access the parameters window (see section 4.1.2) and modify the value of the “Calibration factors” (entries 4 and 5 of the table) according to the following procedure:

Calculation of the new “Calibration Factor”:

New calibration factor = Old Calibration Factor x (% Al Standard / % Al Result).

In the following example, a reference sample with a well-known aluminum concentration of 44% purity is used to calibrate our analyzer.

Old calibration factor = 0.735 (value displayed in the configuration window see section 4.1.2).

% Al reference sample or standard= 44% (concentration of the reference sample used to calibrate).

% Al result = 42.6% (value obtained by the AL-204 analyzer for the average, at least 3 replicates, aluminum concentration of the reference sample).

The calculation of the new coefficient will be carried out according to the following equation:

$$\text{New factor} = 0.735 \times (44\%/42.6\%) = 0.759$$

Once the new calibration factor has been calculated, it will replace the previous factor in the configuration window see section 4.1.2:

Parametr	Values
Balance COM PORT	COM4
Blank A Value	33.6
Blank B Value	33.6
Calibration Factor A	0.735

Parametr	Values
Balance COM PORT	COM4
Blank A Value	33.6
Blank B Value	33.6
Calibration Factor A	0.759

Fig. 4-7. Table of configuration parameters before manual calibration (left) and after (right).

Pressing the "Activate and save changes" button to validate and save the changes. If the window is closed without saving the changes, the calibration factor will not be modified.

- Automatic calibration:

The automatic calibration procedure allows the modification of the calibration factors through the "**calibration**" function by pressing button (4) of the control menu, according to the following procedure:

First, a reference sample with a well-known aluminum concentration or high purity standard (preferably 99%) must be chosen.

Next, 2 or 3 replicas the standard are made, in each channel of the equipment, analyzing in all cases the same amount of aluminum standard with the same configuration.

Once the results have been obtained, we will press the button (4) of the control menu to open the calibration window.

The screenshot shows a window titled "Analyzer Calibration Form" with a table of calibration data and control elements below it.

Id	Date	Weight	Pmax	Temperature	Result	Crucible
Patron2	10/01/2023 17:51	0.2522	675.62	45.42	98.50	B
Patron2	10/01/2023 17:49	0.2504	673.12	46.06	98.82	A
Patron2	10/01/2023 17:36	0.2574	687.19	43.90	98.19	B
Patron2	10/01/2023 17:33	0.2506	673.12	45.85	98.71	A
Patron2	10/01/2023 17:19	0.2553	681.73	43.43	98.20	B
Patron2	10/01/2023 17:17	0.2570	680.06	45.14	97.31	A
Patron1	10/01/2023 15:58	0.1485	426.45	42.24	103.47	B
Patron1	10/01/2023 15:55	0.1465	413.22	43.39	101.41	A
Patron1	10/01/2023 15:41	0.1558	429.69	41.40	99.41	B
Patron1	10/01/2023 15:40	0.1436	443.20	41.99	111.47	A
Patron1	10/01/2023 15:21	0.1597	446.90	40.45	101.12	B
Patron1	10/01/2023 15:19	0.1500	413.50	41.81	99.16	A
Patron1	10/01/2023 14:20	0.1550	427.84	43.21	99.49	A
Patron1	10/01/2023 14:17	0.1510	432.46	43.29	103.30	B
Patron1	10/01/2023 14:06	0.1507	422.01	42.20	100.82	A
Patron1	10/01/2023 14:03	0.1497	412.66	42.67	99.10	B

Below the table, there are control elements:

- Crucible Selection:** Radio buttons for Crucible A and Crucible B.
- Standard Value:** A text input field.
- Actual Calibration:** A text input field.
- Calibrate:** A button.

Fig. 4-8. AL-204 Analyzer calibration window.

In the table, we will select the results obtained for the same crucible⁵ (with the help of the mouse and the CTRL key),⁶ we will select the crucible to calibrate A or B and enter the value of the standard in the text box "Value of Standard" (for example: 99 for a standard of 99% aluminum purity).

By pressing the "**Calibrate**" button, the "Calibration Factor" calculation process begins. Once the new "Calibration Factor" is obtained, it is automatically updated in the configuration parameter table (see section 4.1.2).

4.1.5. Leak test

This functionality allows us to check the tightness of the system, checking the hermetic closure of the crucible, thus confirming that there will be no leaks during the analysis of the sample that

⁵ The selected analyzes must belong to the same crucible and be carried out with the same standard.

⁶ It is possible to obtain some result which is not useful "outlier" so it will not be selected for the calibration process.

could falsify the result. To do this, we will access the leak check routine by pressing the button (5) "Leak Test", a window will instantly appear with a graph of the pressure evolution over time:

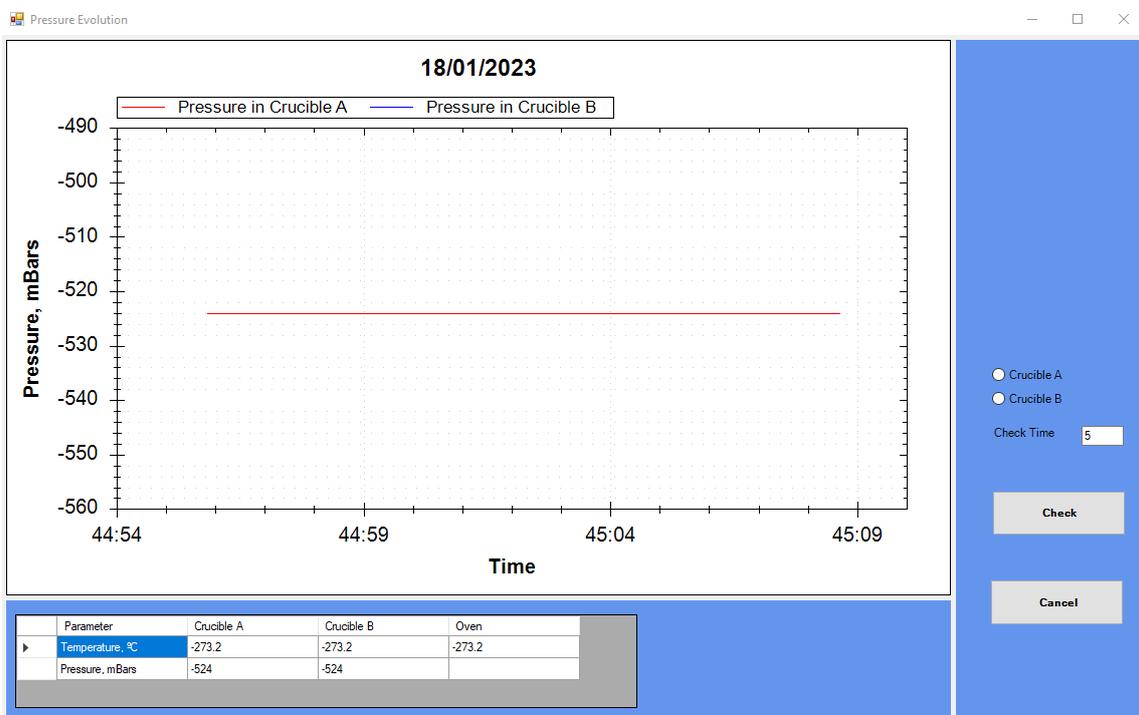


Fig. 4-9. Graph of pressure evolution over time.

To carry out a leak test, we must place an empty crucible in the channel to be checked, select the crucible A or B, the one that corresponds, and press the "Check" button.

Next, the piston will close the crucible and the system will apply a pneumatic pressure close to 1 – 1.3 bar, in this state all the valves remain closed for five minutes. If during this time the pressure is reduced, it is a sign of leaks and the system will have to be revised until such leaks are found. **A channel that fails the leak check cannot be used for aluminum purity analysis.**

4.1.6. Manual functions

This window is very useful for diagnosing and managing the analyzer, it allows you to act manually on all the elements that make up the equipment, without any limit. This in turn requires care and attention on the part of the operator, an inadequate state of the valves for a long time can cause damage to the analyzer.

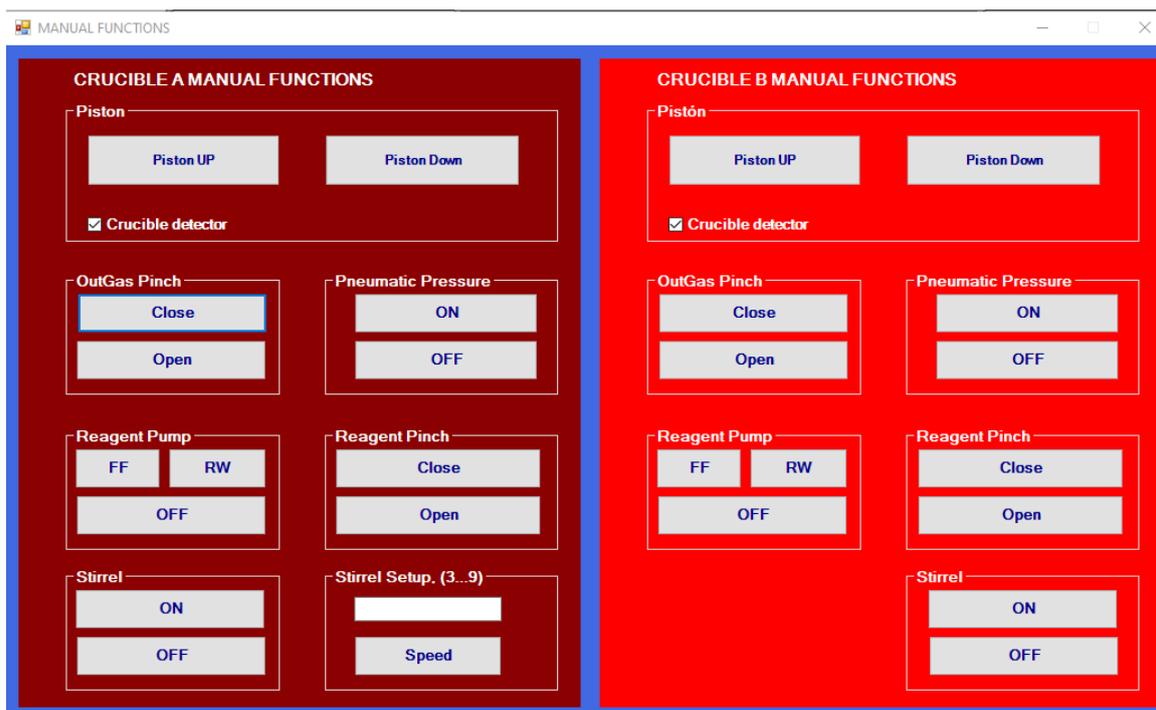


Fig. 4-10. Manual control window for analyzer functions, crucible A (left) and crucible B (right).

The functions of each of the buttons present in the manual functions window are described below.

The menu is divided into 2 analogous windows, the one corresponding to the manual functions of Crucible A and the one corresponding to the functions of Crucible B. They are equivalent functions for each crucible so that only the operation of one of them is detailed.

Piston: the “**Piston UP**” button allows you to release the crucible after analysis; this function can also be executed by double-clicking on the graphic window of the corresponding crucible. **If the crucible is pressurized, this command is ignored for safety reasons**, and a warning appeared.

By pressing the “Piston UP” button A, the SV7 solenoid valve is activated, which pressurizes the duct (5) to a working pressure of around 4 Kg/cm². The crucible clamping is automatically activated and the piston is raised, once the piston is in its position, rest position, the SV7 solenoid valve closes, releasing the crucible holder.

The “**Piston Down**” button manually seals the crucible. This action opens the SV8 solenoid valve, pressurizing the duct (6) and lowering the piston.

The tick (check mark) “Crucible detector” appears checked if the system detects that there is a crucible, otherwise it will appear unchecked, preventing the execution of the sample analysis.

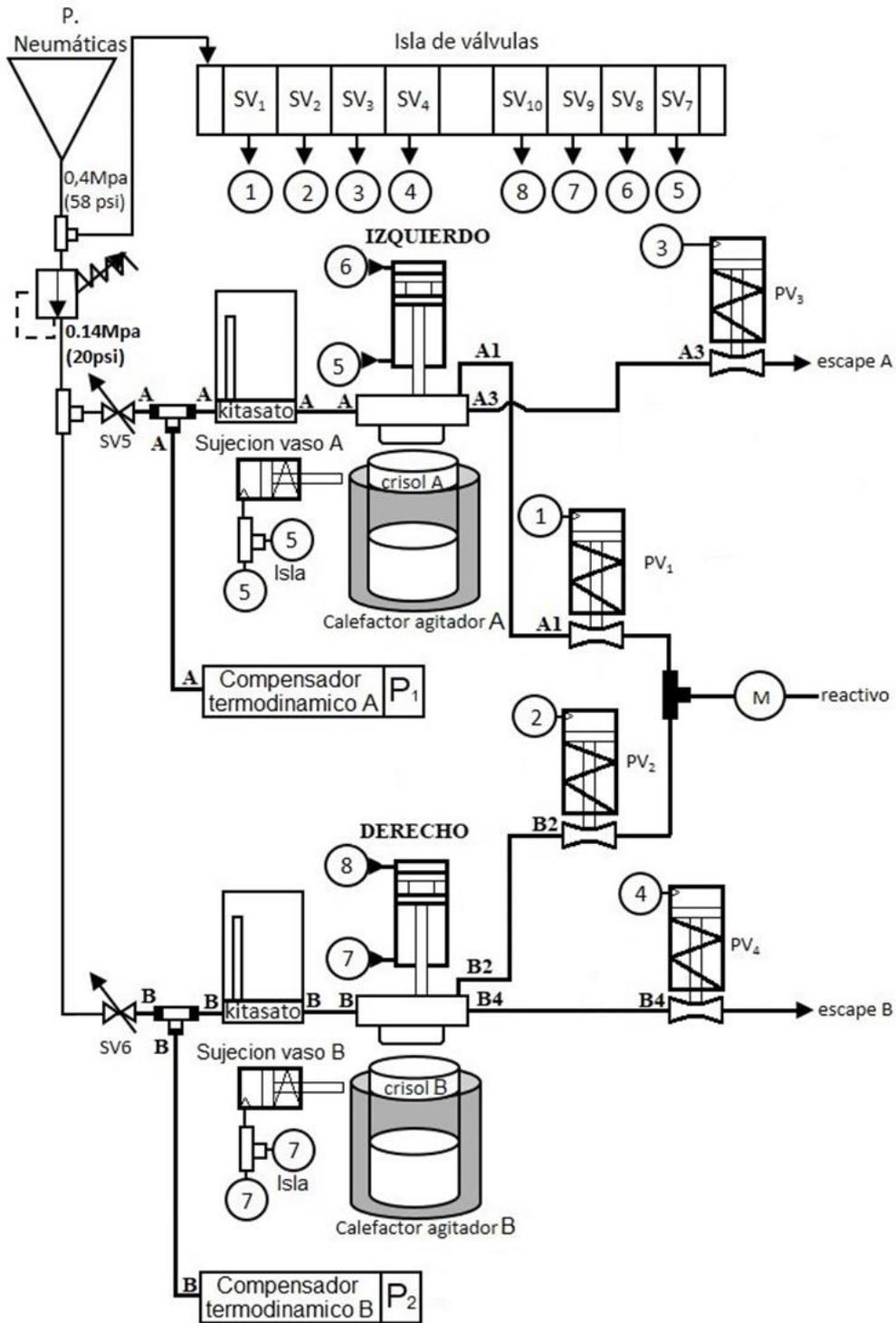


Fig. 4-11. AL-402 Pneumatic diagram.

OutGas Pinch: The “Close” and “Open” buttons control the functions of the “Exhaust” valve (PV₃ (see pneumatic diagram) allowing manual crucible pressure release.

The exhaust valve is a pinch valve with pneumatic control. When you activate the “CLOSE” button, the SV3 solenoid valve is activated, pressurizing the duct (3) and blocking the gas outlet

of crucible A. When you press the “OPEN” button, the SV3 solenoid valve is deactivated, opening the gas outlet of crucible A.

Pneumatic pressure: the “ON” and “OFF” buttons control the functions of the inlet valve (SV5 or SV6 depending on the crucible A or B, allowing the system to be pressurized with compressed air around 1.4, 1.5 Kg/cm².

The gas inlet valve is a solenoid valve. Activating the “ON” button activates the SV5 solenoid valve, pressurizing the entire volume of channel A. Activating the “OFF” button deactivates the solenoid valve, closing the gas flow through to channel A (3). **The “OFF” action does not release the pressure, it only isolates the system and leaves it pressurized.**

To release the pressure, we must open the PV3 OutGas Pinch exhaust valve.

Reagent Pump: The “FF” button activates the reagent pump (peristaltic pump) feeding the reagent into the crucible, the “RW” button activates the reagent pump returning the reagent to the reagent bottle, and the “OFF” button stops the bomb.

The reagent pump is a 4-roller peristaltic pump common to both channels. To carry out the filling action of crucible A, pressing the “FF” button activates the peristaltic pump at the same time as the reagent Pinch of channel A, favoring the passage of reagent only towards crucible A.

Reagent pinch: the “Close” and “Open” buttons control the functions of the reagent inlet valve to the crucible.

The reactive pinch valve is a pneumatically controlled pinch valve. When you activate the “OPEN” button, the SV1 solenoid valve is activated, depressurizing the conduit (1) and unblocking the passage of the reagent solution through the valve for filling crucible A. When you press the “CLOSE” button, the SV1 solenoid valve is activated, deactivates it by re-pressurizing the conduit (1), closing the passage of the reagent through this valve.



WARNING!!! If the user activates the pump of crucible A and manually opens the reagent pinch valve of the other crucible, the flow of reagent will be distributed between in both channels, which **may cause reagent A to spill out of the crucible.**



WARNING!!! If the user manually closes the reagent pinch valve while the reagent pump is active, internal overpressure can be generated and even a tube burst, **causing reagent A to spill inside the equipment**

Stirrer: The “ON” button activate the magnetic stirrer and “OFF” buttons deactivate the magnetic stirrer.

Stirrer setup (1...9): allows to modify the speed of the magnetic bar between the minimum 1 and the maximum 9.

4.1.7. Temperatures

This option, button (7) of the control menu, allows us to view a graph of the analyzer temperatures (crucibles A and B and thermodynamic compensators temperatures) over time. It is very useful to check possible temperature variations in the different parts of the circuit and determine its stability and influence on the analysis.

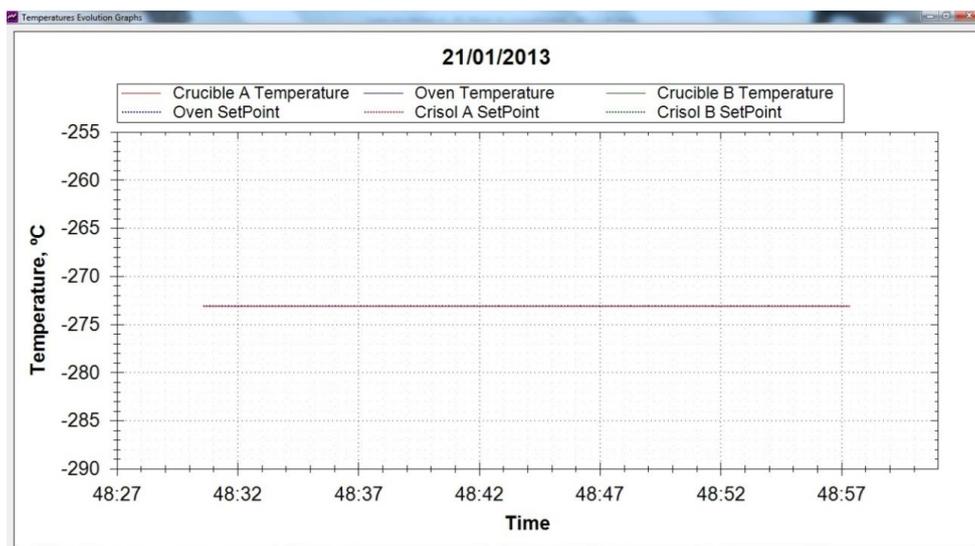


Fig. 4-12. Graphic representation of the temperature monitored by the different sensors of the system; crucibles A and B and temperature of the thermodynamic compensators.

4.1.8. Data base

This functionality, button (8) of the control menu, shows a table with the results of all analysis carried out by the equipment.

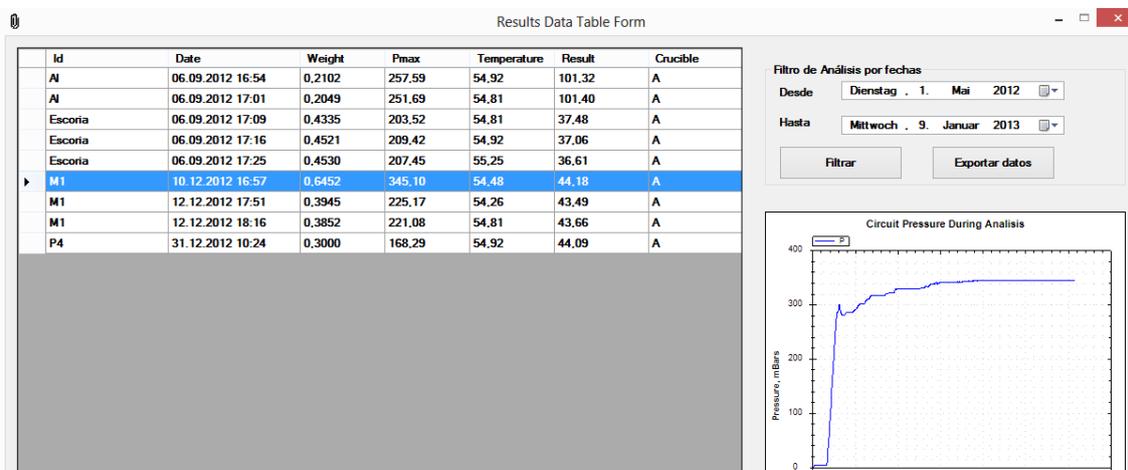


Fig. 4-13. Data base window.

These data can be filtered by date and sorted by different criteria. All table content can be exported to an Excel file. If we select several rows of the table while pressing the “CTRL” key, the program will calculate the average of the results and the relative deviation of the selected analysis. The pressure evolution plot, of the selected sample, is depicted in the graph area.

4.2. Sample data input

The analyzer allows the incorporation of sample data in two different ways:

- **Semi-automatic mode:** When a scale is connected to the analyzer, only the name of the sample will be written. The weighing value will be transferred to the lower table, when pressing the "Balance" button
- **Modo manual:** En la opción manual tanto el nombre de la muestra como el valor de la pesada se escribe antes de presionar el botón de “Balanza” quedando ambos valores transferidos a la tabla inferior.
- **Manual mode:** In the manual option, both the name of the sample and the value of the weight must be written in the table below.

No.	Id	Weight
1	id	
2		

Fig. 4-14. Sample data input window.

The sample name can contain up to 20 alphanumeric characters with or without spaces between them, avoiding the use of dots, commas, and slashes. Samples with the same repeated name can be analyzed. The analyzer program stores them in the hard disk of the computer as a Microsoft Excel and Access compatible table.

4.3. Analysis process representation

In the main window of the user interface, there are two analog windows that each represent a crucible A or B. In both windows, you can observe various parameters in real time during the analysis.

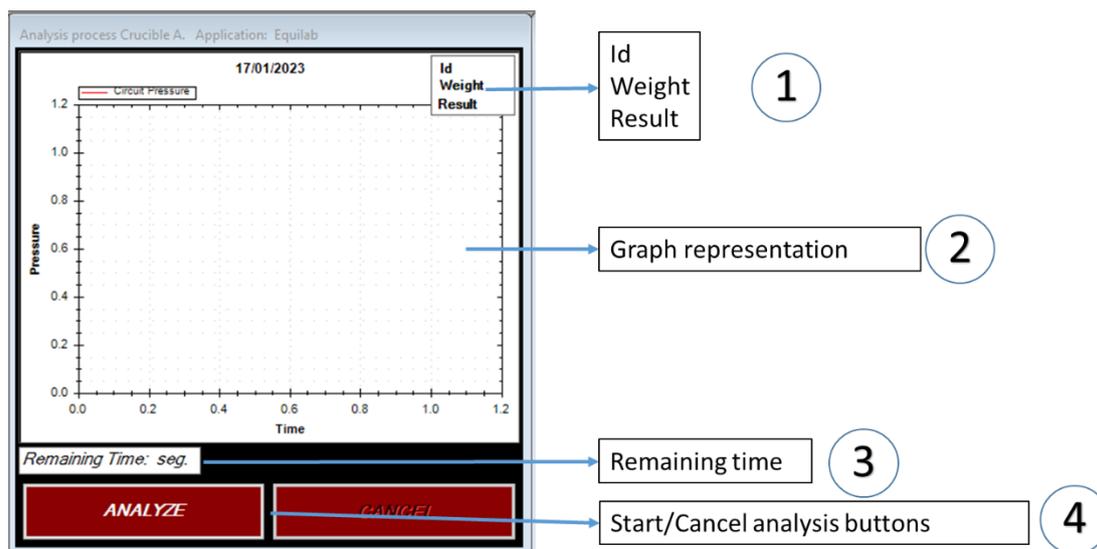


Fig. 4-15. Graphical representation window.

1. This box contains the reference values of the sample (a) Id (name of the sample) and (b) weight in g, and (c) instant result of the purity of the sample in % of metallic aluminum.
2. The inset graph represents the reaction kinetics by monitoring the pressure value vs time. This graph allows us to evaluate easily the behavior of the sample from the beginning of the reaction until the end of it.
3. Remaining time of analysis until the end.
4. Pressing the "Analyze" button activates the analysis sequence (see section 3.3). The "Cancel" button allows you to abort the analysis at any time. If the analysis is aborted during the first 5 minutes, the result is not recorded in the database. If the analysis is aborted after that period, the result is recorded as finished sample.

4.4. Last analysis

This table shows the following: the sample ID, the date and time of the analysis, the mass analyzed, the maximum pressure, the temperature, the purity, and the crucible in which the analysis was carried out. The values shown in the table are the result of the analysis carry out during the last 2 days.

Últimos análisis realizados

Id	Fecha	Peso	Pmax.	Temperatura	Resultado	Crisol
patron 17	06/10/2020 13:38	0,5062	634,17	42,89	45,68	B
patron 17	06/10/2020 13:31	0,5051	1351,33	51,70	98,34	A
patron 16	06/10/2020 13:13	0,5005	1336,16	50,87	98,19	B
patron 16	06/10/2020 13:11	0,5018	1339,49	46,38	98,46	A
patron 15	06/10/2020 12:56	0,5063	1351,24	48,51	98,18	B
patron 15	06/10/2020 12:50	0,5016	1340,42	47,25	98,58	A
patron 14	06/10/2020 12:37	0,5020	1356,70	45,32	99,45	B
patron 14	06/10/2020 12:30	0,5024	1343,93	48,89	98,68	A

Fig. 4-16. Last result table.

4.5. Manometers

The pressure gauges displayed on the main screen of the user interface represent the pressure inside the crucibles. They have a color scale that indicates the optimal area of operation. This graphic representation also shows the temperature⁷ values of the crucibles and thermodynamic compensators.

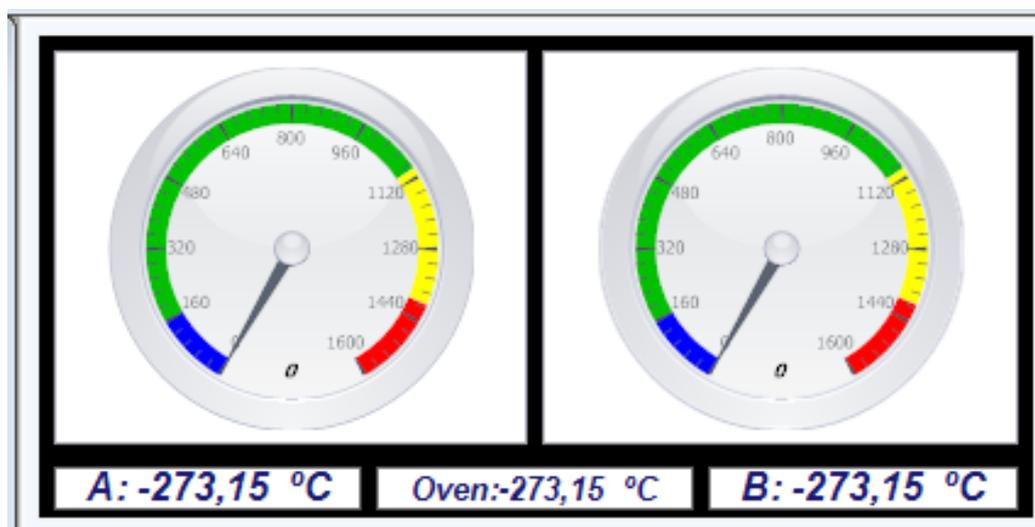


Fig. 4-17. Graphic representation of the working pressure of each one of the crucibles, as well as its temperature and the temperature of the thermodynamic compensators (Oven).

⁷ The reaction between aluminum and the hydroxide is a very exothermic reaction. Sometimes, especially when we use a pure aluminum standard, the temperature can increase by more than 10°C during the analysis.

Blue Zone. Suboptimal working area. It indicates low pressure that, although it allows the measurement to be carried out, the precision in the calculate purity of aluminum is not optimal. *It is recommended to increase the amount of sample.*

Green zone, optimal pressure zone to carry out the analysis, presents the maximum precision in the measurement.

Yellow zone, non-optimal zone but not limiting, working in this interval means working at maximum capacity.

Red zone, the pressure is limit and can saturate the detector, so the measurement can be wrong, *it is recommended to reduce the sample mass.*



SECURITY NOTICE!!! If the pressure generated by an unknown sample exceeds the maximum pressure threshold (1.5 Kg/cm²), the analyzer stops the measurement (without saving any results) and releases the crucible pressure by opening the exhaust valve of the corresponding channel.

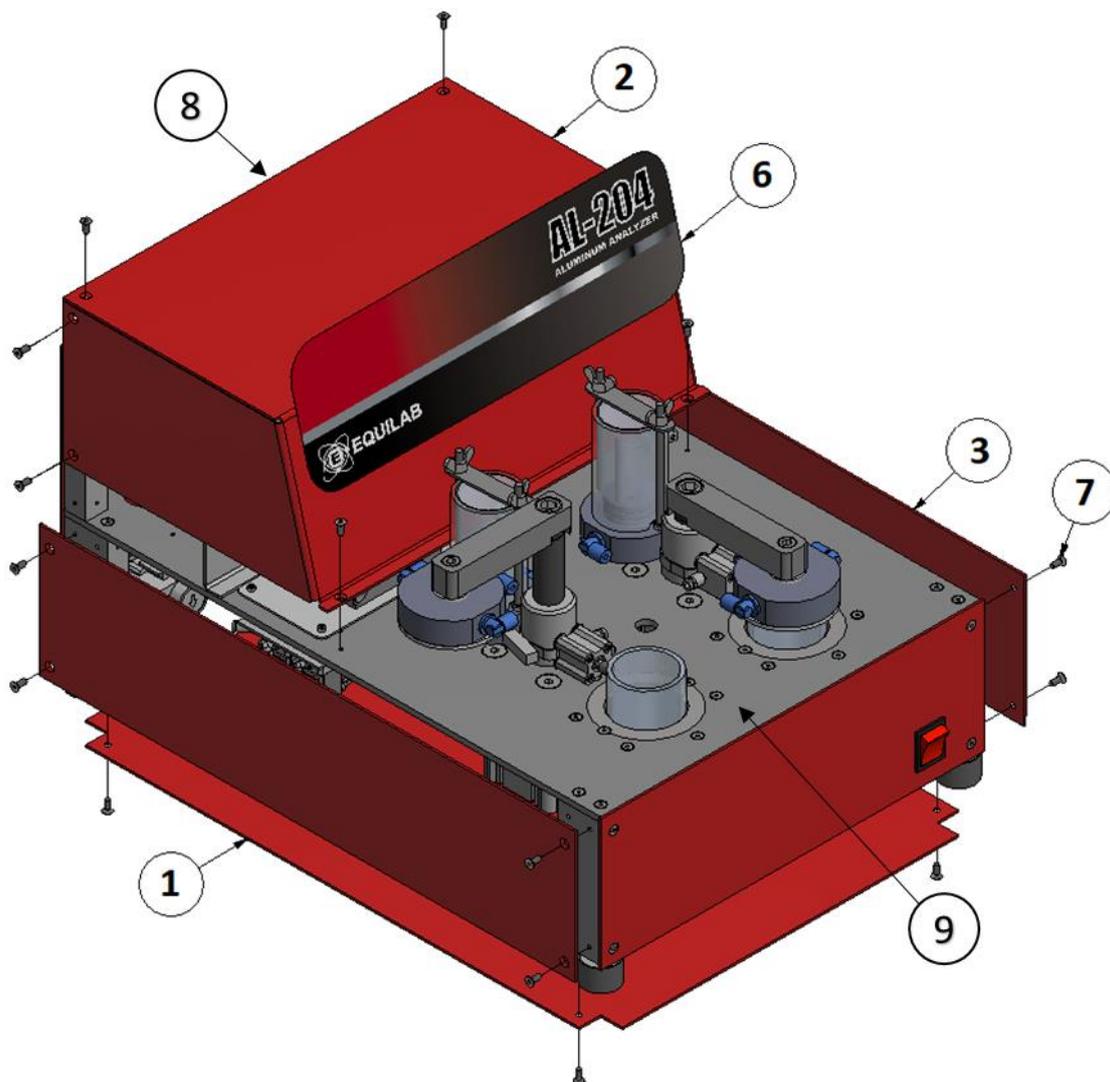
5. Maintenance

The maintenance of an analyzer is essential for correct operation. This section describes the basic maintenance operations of the AL-204 analyzer.

Likewise, advanced maintenance operations are described that allow the technical service to replace all elements of the system.

5.1. Accessing the AL-204 analyzer

The AL-204 aluminum analyzer is a simple piece of equipment that is easy to maintain. To carry out many of the basic maintenance operations we must remove some of the equipment covers. The following image shows the procedure to remove the top, bottom and side covers of the equipment.



Pos.	Ref.	Description
1	AL-011	Lower cover
2	AL-014	Top cover
3	AL-016	Side cover
6	AL-173	Adhesive cover
7	DIN 7991	Countersunk screw M4x6
8	AL-015	Back Cover
9	AL-005	Base plate 2 cup

Fig. 5-1. Detail of the disassembly of the exterior covers of the analyzer.

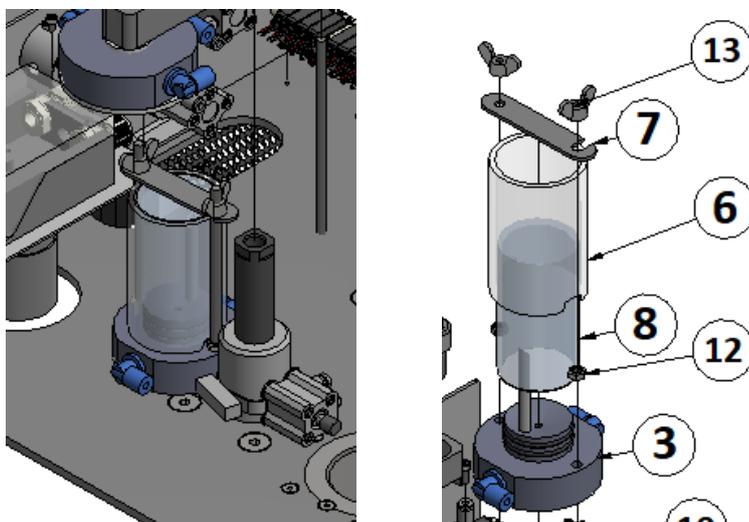
5.2. Basic maintenance of the analysis circuit

For its correct operation, the pneumatic circuit of the AL-204 requires periodic maintenance tasks.

5.2.1. Cleaning the liquid trap (Kitasatos).

It is normal that, after a long period of analysis, the reaction residues are deposited in the liquid traps (Kitasatos). Therefore, it is necessary to clean it every 70 to 100 analyses, depending on the nature of the samples. To do this we must follow the following steps:

1. Remove the metal retaining bar (7), releasing the wing nuts (13).
2. Remove the methacrylate protector (6).
3. Remove the glass cup by pulling it upwards (8).
4. Carry out cleaning tasks.
5. Return all elements to their original position.
6. Carry out a leak check to prove the tightness of the circuit



Pos.	Ref.	Description
3	AL-X09	Kitasatos base
6	AL-X14	Methacrylate protector
7	AL-031	Metal retaining bar
8	AL-167	Kitasato Glass
13	DIN 315	Wind nuts M5

Fig. 5-2. Detail of the Kitasato glass assembly

5.2.2. Obstructions in the pneumatic or hydraulic system

Occasionally the remains of the chemical reaction can obstruct the tubes and elements of the pneumatic circuit. When this happens, the intervention of a maintenance technician is required capable of locating where the blockage is and eliminating it, either through cleaning or by replacing the obstructed section. During this routine it is necessary to take additional safety measures.

In the same way, clogging of the crucible closing lid can occur. In this case we must try to unblock the holes with a sharp object, or remove the connectors if necessary. These solids usually come from the splashes that are generated during the initial phase of the reaction.

Pos.	Ref.	Description
	AL-102	POLYACETAL ELBOW FITTING
	AL-103	POLYACETATE FITTING for 4 mm tube
	AL-135	T FITTING for 4 mm TUBE
	AL-163	POLYPROPYLENE Hose barb T fittings for 4 mm tube

	AL-162	POLYPROPYLENE Hose barb fitting for 4 mm tube
	AL-137	TEFLON TUBE 4mm (transparent)
	AL-138	POLYURETHANE TUBE (blue) 4mm
	AL-142	VALVE TUBE
	AL-143	PUMP TUBE
		TUBE FLANGE

5.2.3. Peristaltic pump maintenance AL-X01

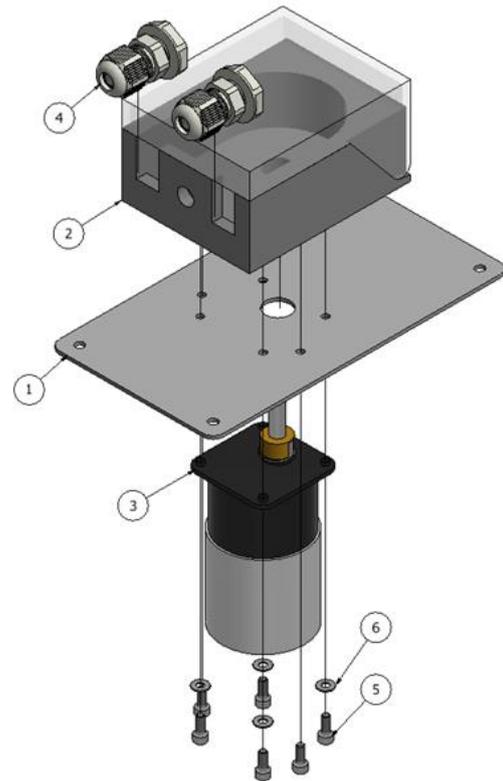
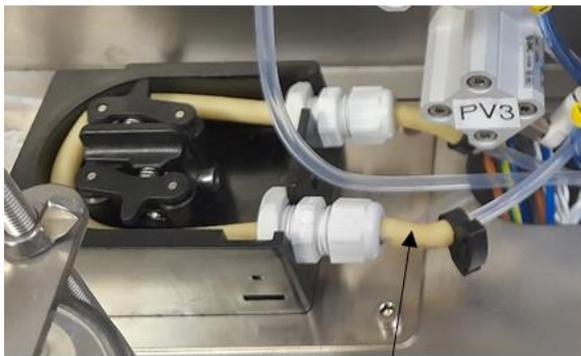
Due to the continuous friction in the pump tube, it is necessary to carry out periodic checks of the condition of said tube. After long periods of use, it may be cracked or show aging of the material (deformation), which results in irregular or incorrect dosage.



Never use a peristaltic pump or dosing pump tube other than those recommended by the manufacturer.

To check the peristaltic pump tube, we must remove the top cover of the AL-014 analyzer, as well as the transparent cover that protects the rollers and the tube, release the Teflon tube flanges, cable gland and extract the tube.

- Check the condition of the rubber tube, replacing it at the slightest sign of breakage or deformation.
- Check the condition of the friction rollers, replacing them if any alteration is detected.



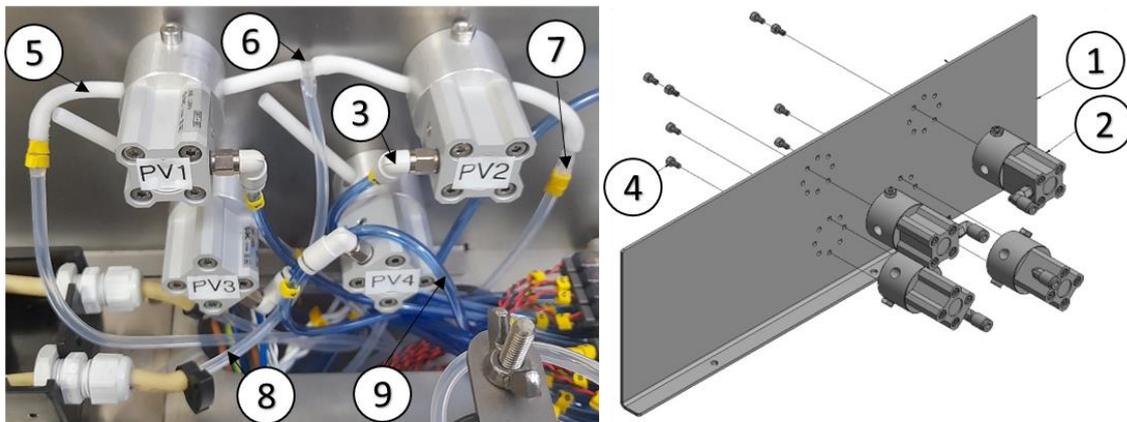
Pos.	Ref.	Description
1	AL-019	Peristaltic pump metallic pump
2	AL-117	Head and rotor of Peristaltic pump
3	AL-118	Motor peristaltic pump
4	AL-146	Cable gland
5	DIN 912 M3x8	Allen screw M3x8
6	DIN 125 M3	Washer M3
7	AL-143	Peristaltic pump tube

Fig. 5-3. Exploded detail of the peristaltic pump.

5.2.4. Pinch valve maintenance

It is recommended to replace the rubber tubes of the Pinch valves at least twice a year. To do this, the top cover of the analyzer must be removed to access the valve block.

Once the top cover is removed, the valve tube is disconnected from the polypropylene hose barb T fittings and removed by pulling the tube. The installation of the new valve tube is carried out in reverse order, the tube is inserted into the valve hole and the polypropylene hose barb hose T fittings are connected.



Pos.	Ref.	Description
1	AL-017	Thermo plate
2	AL-X02	Pinch valve assembly
	AL-170	Elbow fitting M5 -tube 4
4	DIN 912	Allen screw M3x6
	AL-142	Valve tube
	AL-163	Hose barb T fittings for 4 mm tube
	AL-162	Hose barb Fitting for 4 mm tube
	AL-137	Teflon tube 4mm (transparent)
	AL-138	Polyurethane tube (blue) 4mm

Fig. 5-4. Detail of the pneumatic pinch valves, after removing the top cover of the equipment.

Pinch valves are very easy elements to replace. To replace only one, we must remove the valve tube (AL-142), the gas pressure inlet tube (AL-138) and 2 M3x6 Allen screws, as shown in the previous image.

Replacing all the valves is carried out by removing part AL-017 once the 4 valves have been disconnected, and installing the new one with the new valves.

The arrangement of the pinch valves is shown in the image, their arrangement must be as indicated since it favors the connection of all the tubes and activation gas inlet.

5.3. Advanced maintenance operations

5.3.1. Replacing the valve island AL-X05

The valve island is responsible for controlling all pneumatic activation of pinch valves or pistons of the aluminum analyzer. The island is a set made up of 8 solenoid valves mounted on a multi-station support.

You can replace the solenoid valve island assembly (AL-X05) or each AL-123 solenoid valve individually. To carry out the replacement we must remove the upper cover AL-014 and the lower cover AL-011 from the equipment. To have access to the island from the top and to the electrical connections from the bottom.

To replace the **complete assembly**, we must disconnect all the 2-pin JST connectors that connect the different valves to the AL-X13 control board (J10, J11, J14, J15, J16, J17, J18 and J19). A detail of the pinout is found in the wiring section 6 Electronic diagrams of the analyzer.

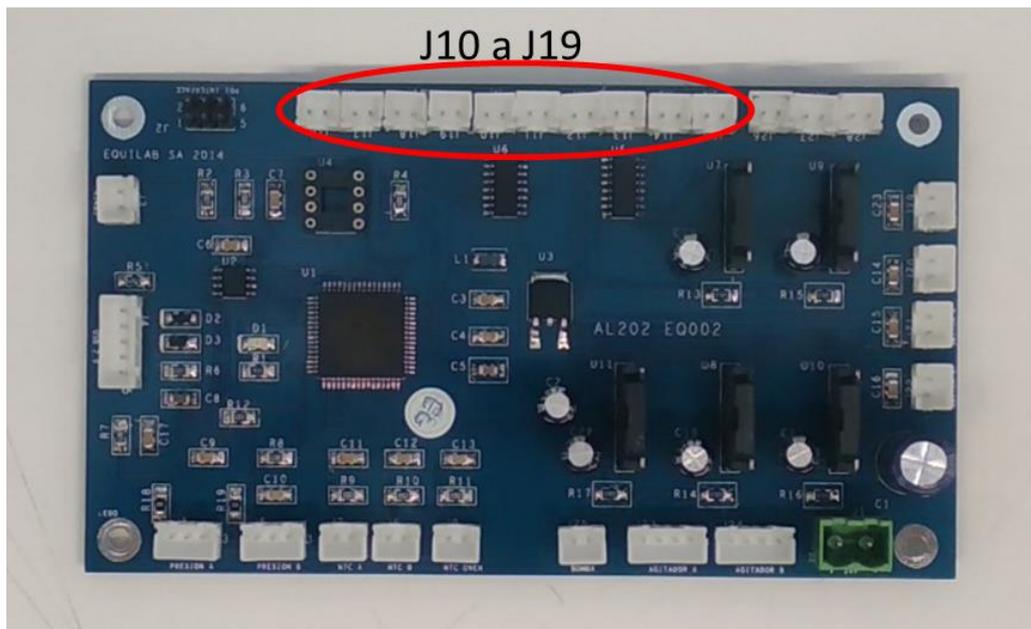
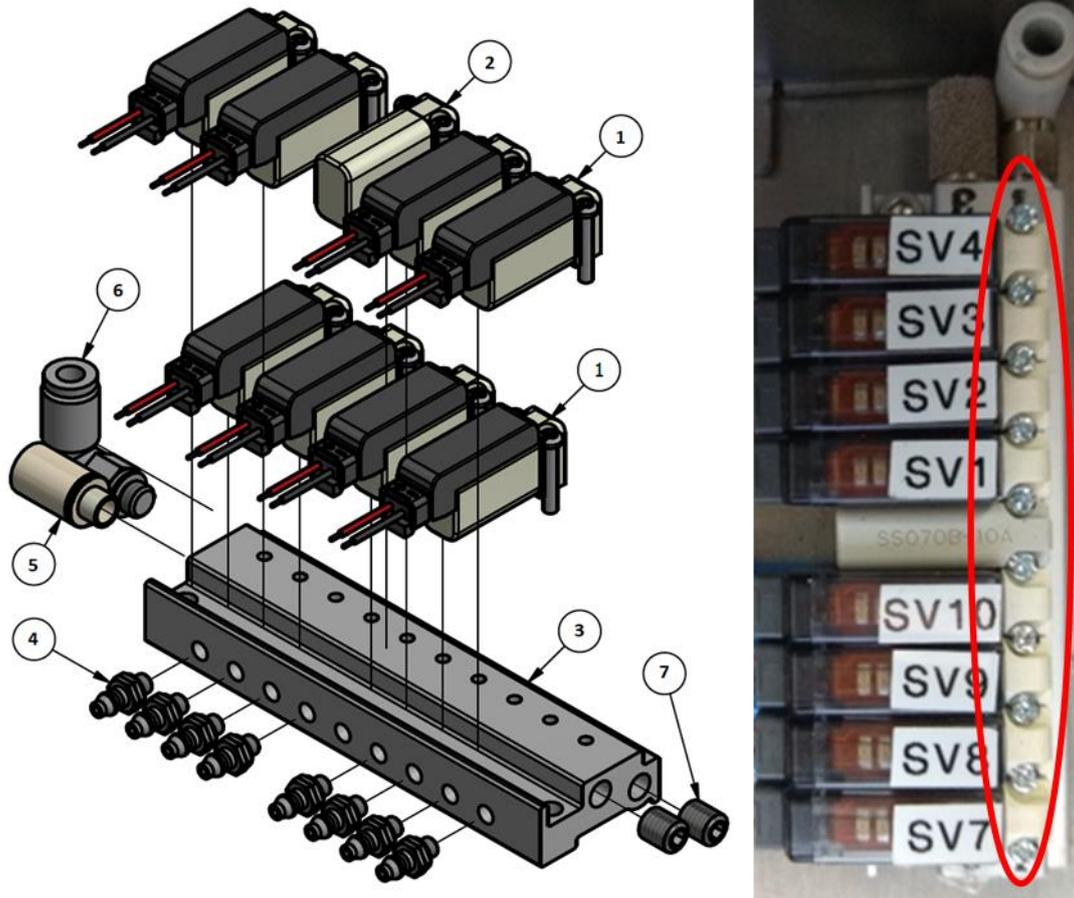


Fig. 5-5. View of the AL-X13 control board.

Next, release all the polyurethane tube connections, and finally loosen the 2 M3x10 Allen screws that hold the solenoid valve island to the mounting panel.

To replace a **solenoid valve**, disconnect the corresponding 2-pin JST connector from the AL-X13 control board (see section 6) and release the screws (marked with the red ellipse) that secure the solenoid valve to the island. As shown in the image.



Pos.	Ref.	Description
1	AL-123	Pneumatic valve
2	AL-124	Island cap
3	AL-126	Island of 9 station
4	AL-132	Fitting M3 – Tube of 4 mm
5	AL-145	Metal muffler
6	AL-170	Elbow fitting M5-Tube 4 mm
7	DIN 913	Grub screw M3x6

Fig. 5-6. Exploded view of the solenoid valve island.

5.3.2. Replacement of the AL-X08 solenoid valve assembly

The set of solenoid valves is used to cut off the gas inlet to the analysis circuit, which we use both to check the tightness of the circuit and to clean it.

You can replace the set (AL-X08) or each AL-028 solenoid valve individually. To carry out the replacement we must remove the lower AL-011 from the equipment. To have access to the solenoid valves and electrical connections from the bottom.

To replace **the complete assembly**, we must disconnect the 2 2-pin JST connectors that connect the solenoid valves to the AL-X13 control board (J12 and J13). A detail of the pinout is found in the separated wiring section 6.

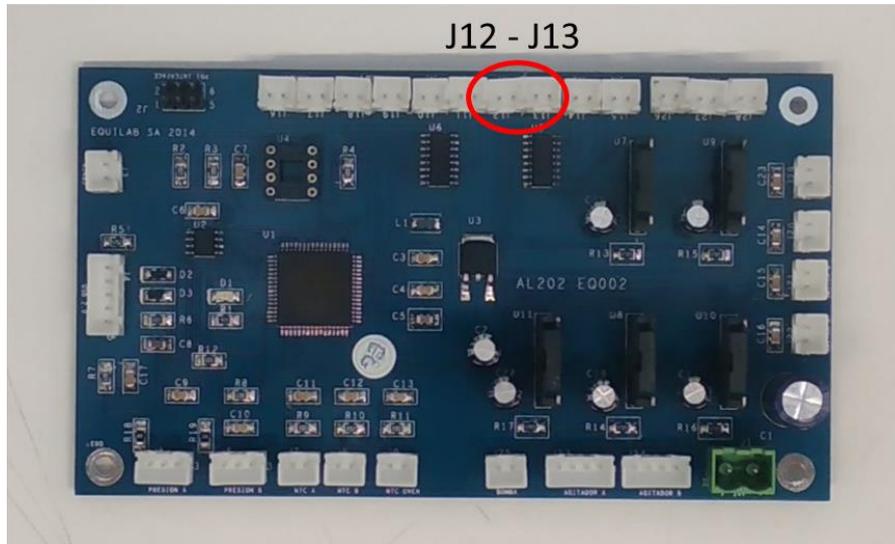
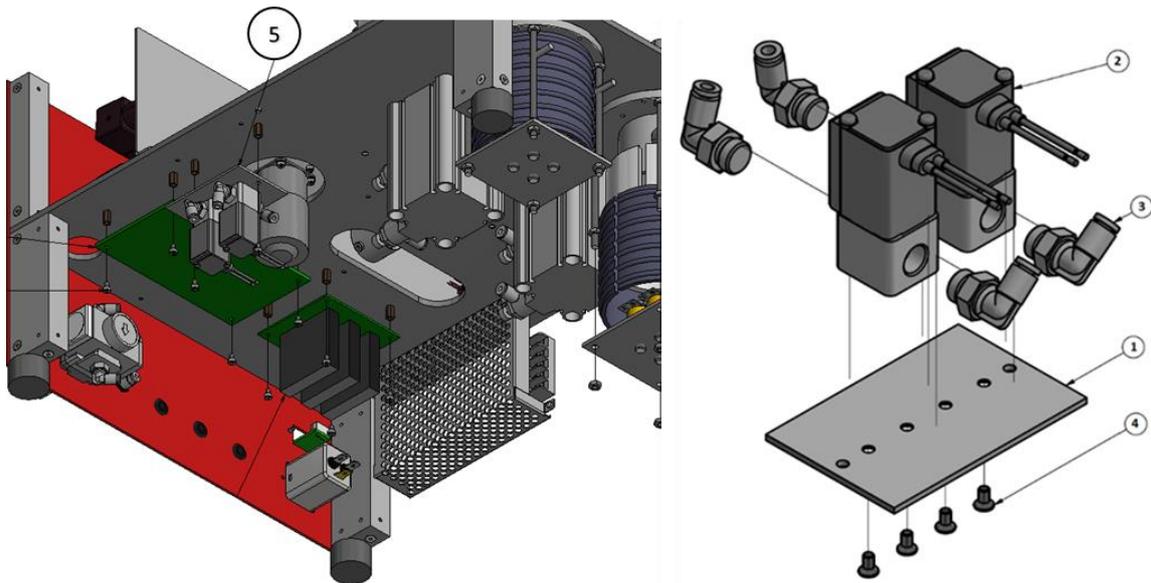


Fig. 5-7. View of the AL-X13 control board.

Next, release all the connections of the inlet and outlet polyurethane tubes, and finally loosen the 2 M3x5 Allen screws that hold the solenoid valve assembly (5) to 2 M3x10 hexagonal spacers installed on the mounting panel.



Pos.	Ref.	Description
1	AL-028	Solenoid valve support S
2	AL-125	Air inlet solenoid valves
3	AL-136	Elbow Fitting 1/8" – tube 4 mm
4	DIN 7991	Countersink screw M3x8
5	AL-X08	Solenoid valve assembly

Fig. 5-8. Assembly view of pneumatic valves.

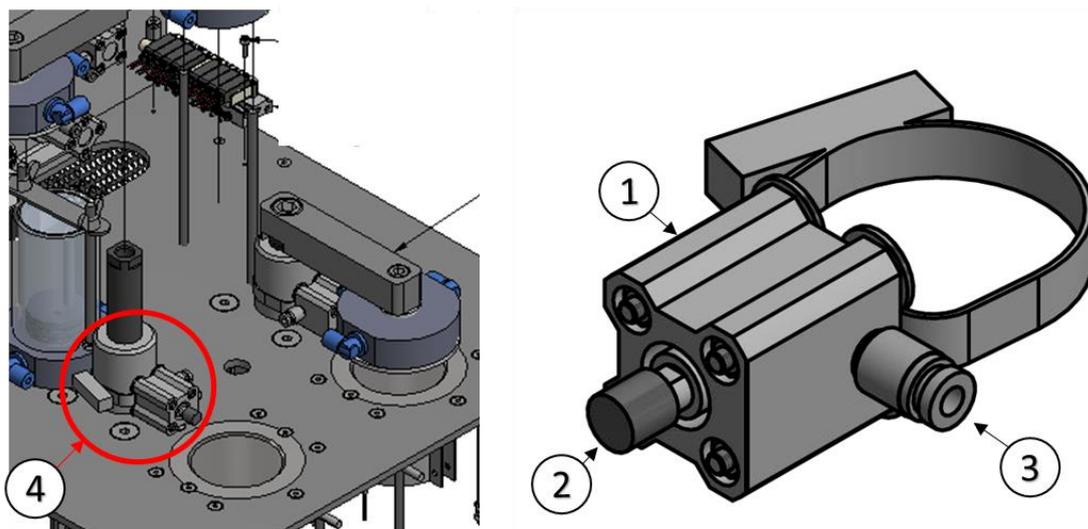
To replace a **solenoid valve**, the corresponding 2-pin JST connector is disconnected from the AL-X13 control card (wiring section 6), then we release the AL-X08 assembly as indicated in the previous section and the solenoid valve to be replaced is released. M3x8 countersunk Allen screws that secure the solenoid valve to the mounting panel must be loosened.

5.3.3. Replacing the glass crucible fixing assembly.

The AL-X10 glass crucible fixing set is used to hold the glass crucible at the time of opening the piston, it prevents the crucible from sticking to the silicone sealing gasket of the crucible closure and breaking when routing.

The following images show how this set is placed. The assembly has a metal clamp that allows the assembly to be fixed to the body of the crucible closing piston.

To release the assembly, we will first remove the cylinder arm as shown in section 5.3.6, then, we will release the polyurethane tube for transmitting the pneumatic pressure, and finally, we will loosen the retaining clamp to extract the AL-X10 assembly pulling it up.



Pos.	Ref.	Description
1	AL-130	Crucible holder pneumatic piston
2	AL-141	Crucible holder
3	AL-151	Straight fitting 4mm – M5
4	AL-X10	Glass crucible fixing assembly

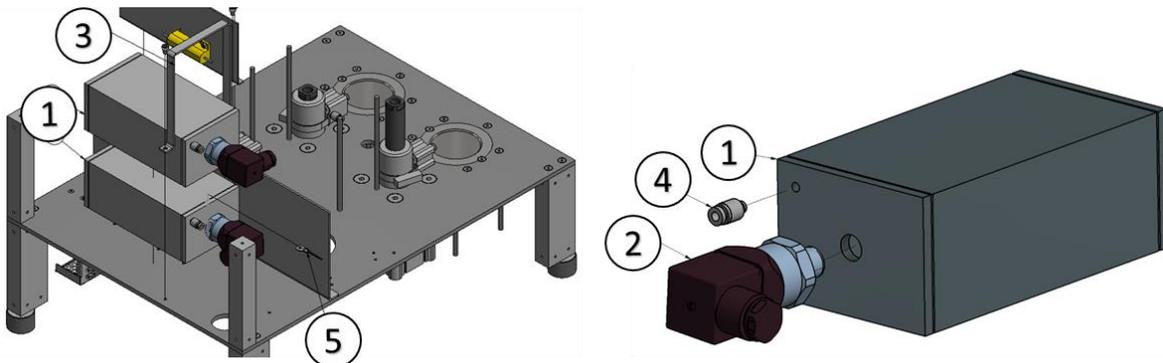
Fig. 5-9. Assembly of glass crucible fixing assembly.

5.3.4. Replacing the pressure transducer

The transducer is installed in an element of constant volume that is used as an expansion vessel to obtain a correct measurement of the pressure increase in the system. The equipment has 2 equivalent elements, one for each measuring channel.

The set consists of an expansion vessel (volume), the pressure transducer and a straight fitting for 4 mm tube.

To comfortably access the pressure transducer, we will remove the top cover AL-014 and the rear cover AL-015, leaving the two expansion vessels and the two transducers accessible.



Pos.	Ref.	Description
1	AL-001	Volumen
2	AL-111	Pressure transducer
3	AL-002	Volumen fasten
4	AL-151	Straight fitting tube 4 – M5
5	AL-139	NTC Temperature Sensor

Fig. 5-10. Mounting the pressure transducer.

The pressure transducer has 2 parts, the connector (responsible for electrical connections) and the body of the transducer.



Fig. 5-11. Pressure transducer AL-111.

To release the transducer, we must first loosen the connector by loosening the screw that holds the connector to the body of the transducer. Once loosened, the electrical connections will be checked and if they do not present any problems, the connector will not be replaced. Finally, a fixed wrench will be used to release the transducer to be replaced.

To assemble the new pressure transducer, we will apply several turns of Teflon to the thread and then we will thread it in the AL-001 volume, without reaching the end. We will use a rubber washer to ensure that *it is not completely threaded*.

Finally, the connector will be placed in its position and the rear and top covers will be fixed again.

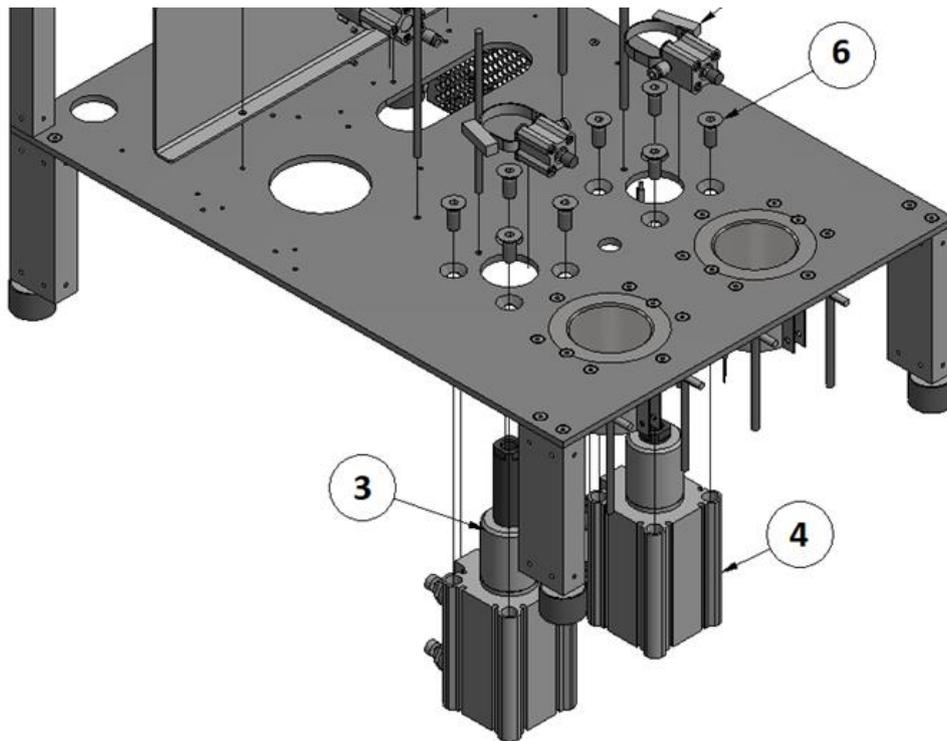


Once the transducer is mounted, it is **ESSENTIAL** to perform a leak test to confirm its correct installation.

5.3.5. Replacement and adjustment of the closing system of the glass crucibles.

These pistons are responsible for the hermetically seal of the glass crucible. The 2 pistons have a symmetrical movement between each other, so they are different and we must identify their right or left position before proceeding to replace them.

To disassemble a piston we must, first of all, remove the cylinder arm as described in section 5.3.6 and the lower cover of the AL-011 analyzer, leaving free access to the pneumatic pistons.



Pos.	Ref.	Description
3	AL-121	Left piston
4	AL-122	Right piston
6	DIN 7991	Countersink screw M8x20

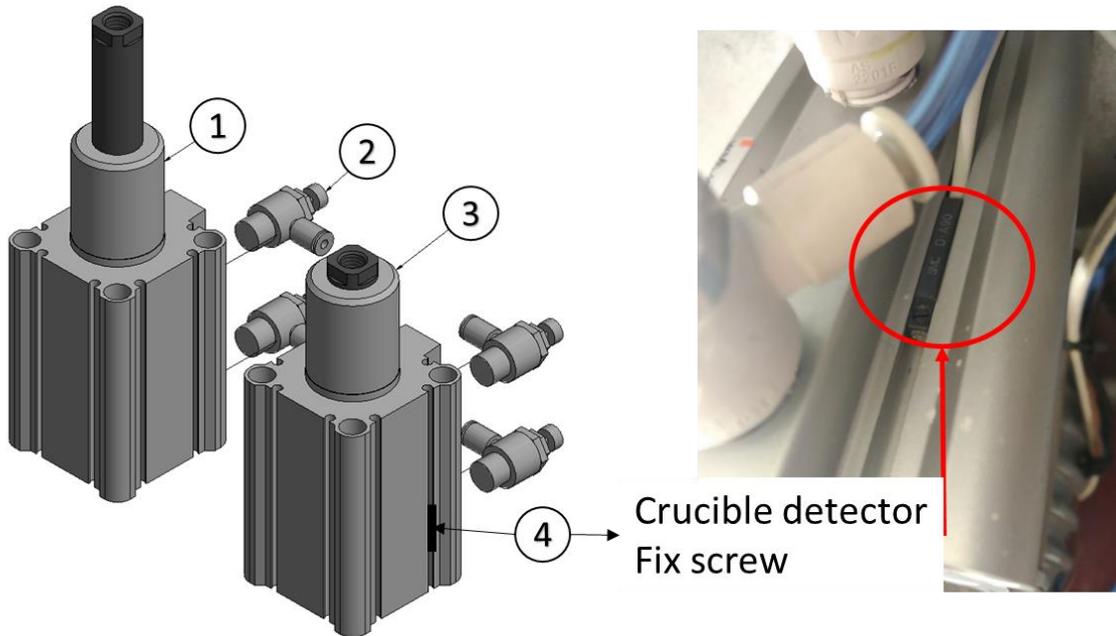
Fig. 5-12. Exploded view of the closing pistons.

Next, we will remove the position detector by loosening the fixing screw and moving the detector downwards (see figure below), we will release the polyurethane tubes responsible for the transmission of pneumatic pressure and the 4 M8x20 countersunk Allen fixing screws from the base panel of assembly AL-005, to completely release the pneumatic piston.

Each piston (AL-121 or AL-122) has 2 (AL-134) flow regulators installed responsible for controlling the inlet flow rate to the piston and consequently the speed of its movement. It also has 1 (AL-129) position sensor that it allows detecting the position of the piston, thus allowing the presence or absence of crucible in its housing to be detected.



The pressure of the compressed gas line is responsible for the sealing pressure of the crucible. The speed of movement is set by the flow regulators, and the speed can be adjusted independently of the pressure of the gas line.



Pos.	Ref.	Description
1	AL-121	Left piston
2	AL-134	Pressure regulator
3	AL-122	Right piston
	AL-129	Position detector

Fig. 5-13. Exploded view of the closing pistons.

To assemble the new piston, we will proceed in reverse order, (a) fixing the piston on the base panel with M8x20 screws; (b) fixing the gas inlet and outlet tubes to the piston, and finally, (c) placement of the detector.

To place the detector in its position, we will use the PC application. We connect the analyzer, establish a connection with the PC application, insert the crucible into its housing and lower the piston (see section 4.1.6). If the presence detector is placed in its place on the manual functions screen (see image below) the marked tick will be displayed., if not, the tick will be not marked, we must move the detector until the tick appears in “Crucible detector”, which indicates correct detection of the crucible.



Fig. 5-14. Positive crucible detection.

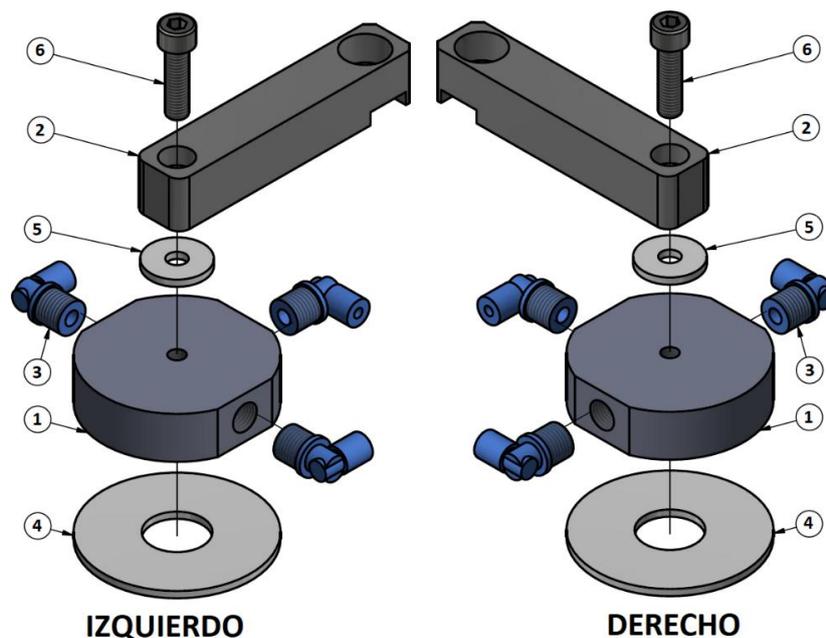


If, once the piston has been replaced, the crucible detector does not recognize the position of the crucible in its housing (marked tick), the analyzer will NOT allow analysis to be carried out in that channel.

5.3.6. Maintenance of the closure of the crucible AL-X11

The crucible closure assembly is used to hermetically seal the glass crucible, so that no gas losses occur during the aluminum oxidation reaction that takes place inside said glass reactor.

The elements that make up the crucible closure are the same on the right and left sides, unlike the pistons. However, the position of the elbow fittings is slightly different, therefore we must be careful when positioning these fittings to avoid stress on the Teflon tubes when opening and closing the piston. The following image shows the exploded view of the AL-X11 piston closure assembly.



Pos.	Ref.	Description
1	AL-026	Crucible lid
2	AL-032	Cylinder arm
3	AL-102	Polyacetal elbow fitting
4	AL-140	Silicon cup gasket
5	AL-150	Silicone union gasket
6	DIN 912	Allen screw M8x30

Fig. 5-15. Exploded view of the crucible closure.

To replace the piston closure assembly, which will always be done in the absence of a crucible, we must, first of all, release the AL-137 Teflon tubes from the AL-102 elbow fittings. Next, we loosen the M12x20 Allen screw that holds the AL-032 cylinder arm to the AL-021 (left) or AL-022 (right) piston cylinder, leaving the assembly free.

To release the crucible lid we must loosen the M8x30 Allen screw that holds the AL-026 crucible lid to the AL-032 cylinder arm, also leaving the AL-150 silicone union gasket free.

If you wish to replace any of the AL-102 elbow fittings, we will wrap the fitting several times with Teflon and then fix it on the AL-026 crucible cover, paying attention to the final position.

To reinstall the assembly, we will proceed in reverse order to the disassembly procedure.

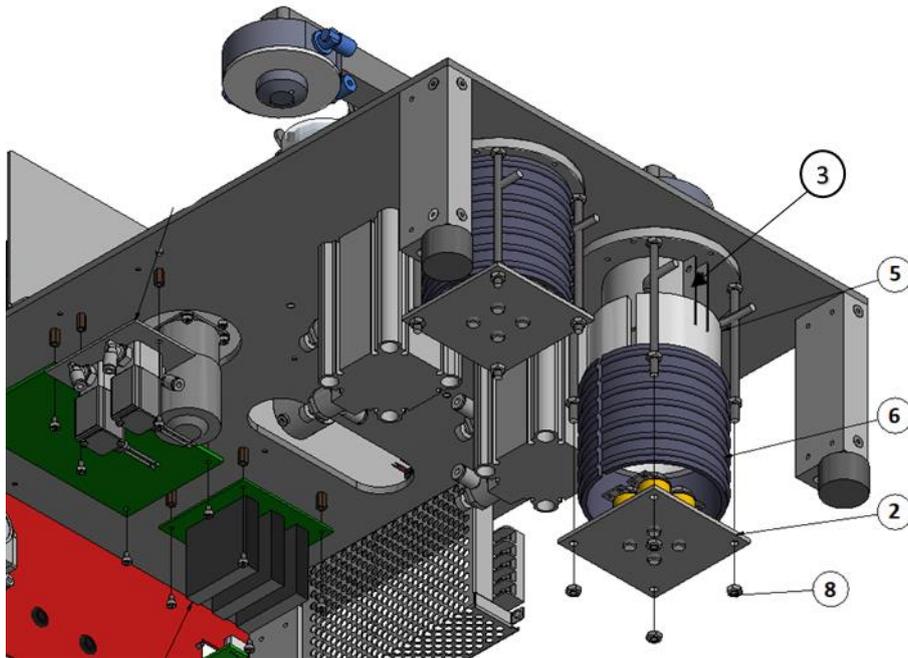
It is NOT necessary to dismantle the closure assembly to replace the sealing gasket (AL-140) of the crucible, we must only be careful to have the piston open and in the crucible outside its housing to have enough space to carry out maintenance on the gasket. The AL-140 gasket fits into its housing in the lid of the AL-026 crucible only by pressure.

Although the seal is usually perfect, to ensure a more effective seal a small amount of vacuum grease can be added to the space between the crucible lid and the AL-140 silicone gasket.

5.3.7. Replacement of the crucible heating and stirring system.

The steel vessel that houses the glass crucible has an individual resistance heating system and a set of coils that form the magnetic stirring system.

To disassemble any of the heating and stirring systems we must, first of all, remove the lower cover of the AL-011 analyzer. In this way we will be able to freely access the disassembly of said systems, for stirring and heating the crucible.



Pos.	Ref.	Description
2	AL-X06	Magnetic stirrer
3	AL-112	Heating resistances
5	AL-160	Glass wool
6	AL-161	PVC protection
8	DIN 555	Nut M5

Fig. 5-16. Exploded view of the crucible stirring and heating system

To disassemble the stirring system, we will loosen the 4 M5 nuts (8) that hold the AL-X06 magnetic stirrer from the threaded rods and once the 4-pin JTS connector from the AL-X13 control card (J23 or J24) has been disconnected. A detail of the pinout is found in the wiring section 6, we will proceed to remove the AL-X06.

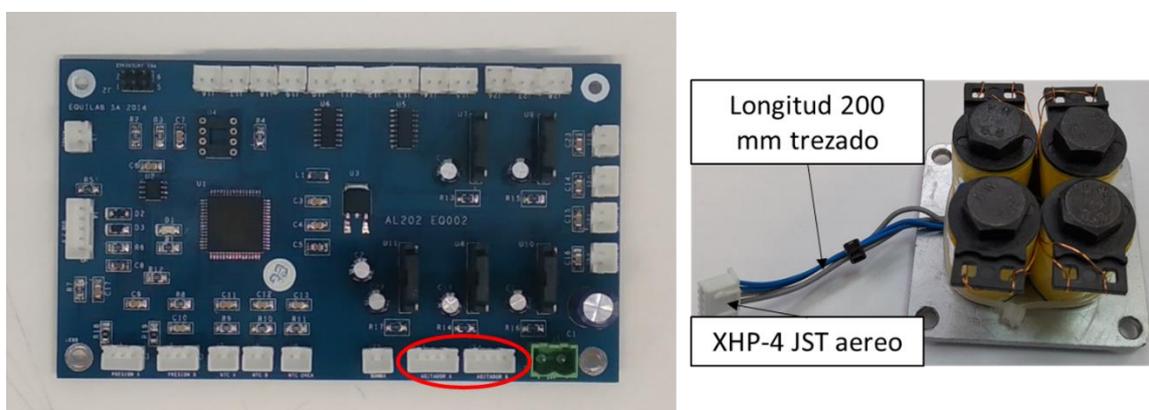
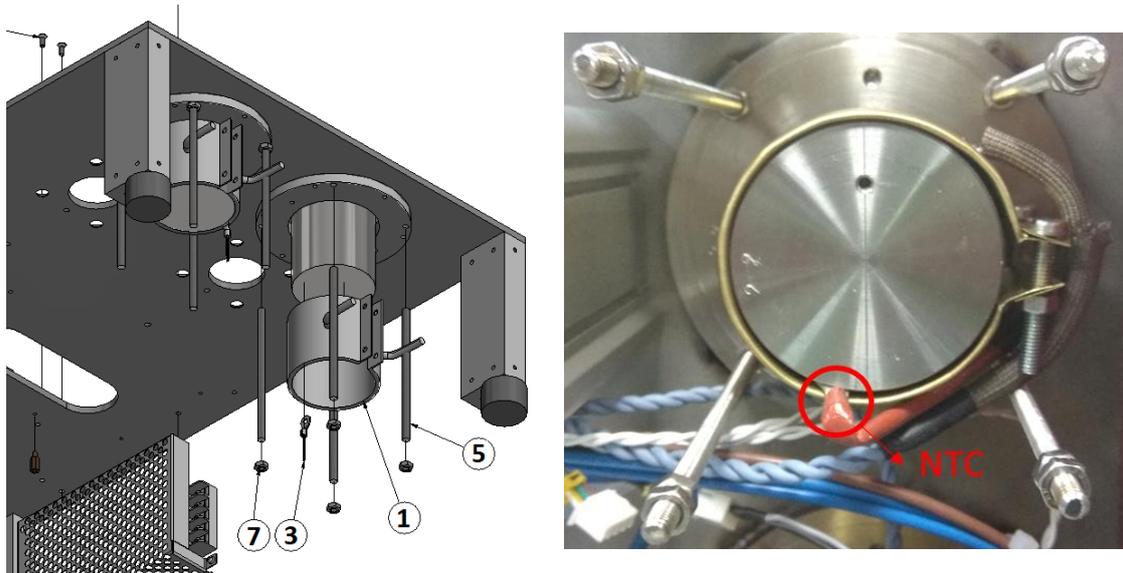


Fig. 5-17. Detail of the connection of the AL-X06 magnetic stirrer on the AL-X13 control card

To proceed to remove the heating element, we will remove the PVC protector (AL-161) by gently pulling it downwards, immediately after the glass wool insulator (AL-160), leaving the heating element (AL-112) and the temperature sensor NTC (AL-139) accessible.

Finally, to remove the AL-112 clamp resistance, we loosen the side screws and the clamp will slide downwards, releasing the NTC temperature sensor at the same time.



Pos.	Ref.	Description
1	AL-112	Heating resistances
3	AL-139	NTC
5	DIM 976	threaded rod M5x113
7	DIN 555	Nut M5

Fig. 5-18. Exploded view of the crucible heating system

To reassemble the system, we will follow the steps in the opposite direction, we will place the temperature sensor between the steel holder and the clamp resistance as shown in the previous image, we will tighten the screws to fix the clamp.

Next, we place the glass wool, the PVC protector, fixing everything using the AL-X06 magnetic stirrer coil support, placing the 4 M5 fixing nuts as indicated in the following image.

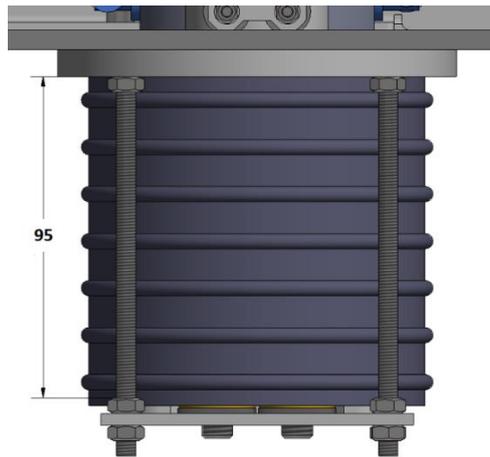
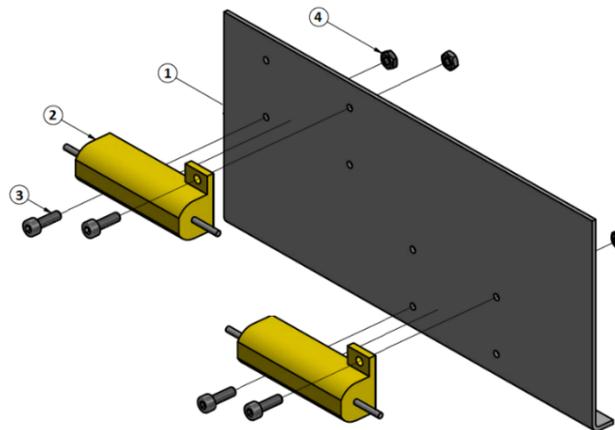


Fig. 5-19. Installing the magnetic stirrer support.

An incorrect distance from the magnetic stirrer may cause erratic operation of the magnetic stirring system.

5.3.8. Replacing the heating plate (AL-X07)

The heating plate is used to heat and maintain a constant temperature of the expansion vessels where the pressure transducers are located. It is made up of 2 series resistors powered at 220 VAC (50 W, 2.2 k Ω) placed in series.

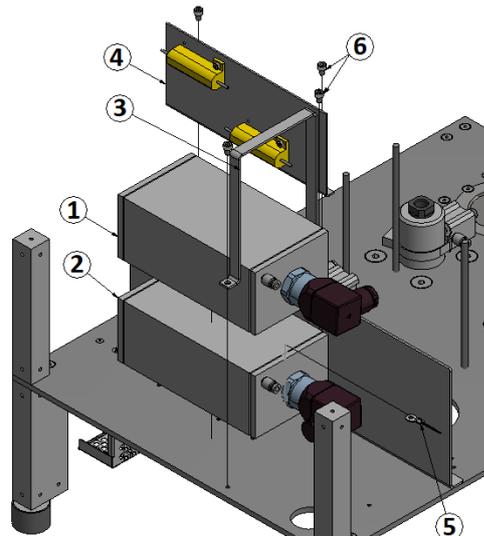


Pos.	Ref.	Description
1	AL-029	Heating plate
2	AL-115	OVEN RESISTANCE
3	DIN 912	Allen screw M3x10
4	DIN 555-5	Nut M3

Fig. 5-20. Explosion of the heating plate.

To remove the heating plate, first of all, we must remove the upper cover AL-014 and the lower cover AL-011 from the equipment. To access the heating plate from the top and the electrical connections from the bottom.

Once the panels have been removed, we disconnect the power cables from the heating resistors by releasing them from the SSR AL-X04 power board (see section 5.3.10) at the bottom of the equipment, and loosening the M4x6 screws (6) at the top.



Pos.	Ref.	Description
1	AL-001	Volume A
2	AL-001	Volume B
3	AL-002	Volumes fixation
4	AL-X07	Heating plate
6	DIN 912	Allen screw M4x6
Izq.	AL-X04	SSR power board

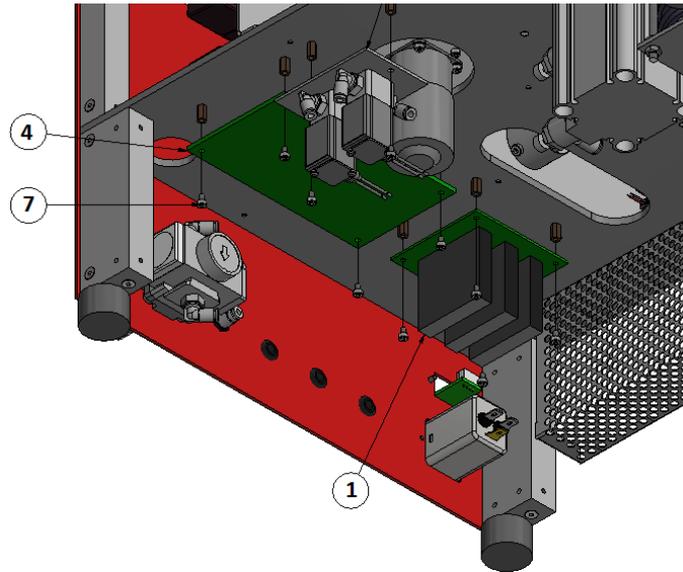
Fig. 5-21.. Disconnection of the heating plate from the “oven”.

To assemble the new heating plate, we will proceed in the reverse order, (a) screwing the heating plate to the AL-005 base plate; (b) connecting the power cables of the resistors to the SSR power board; and (c) assembling the top and bottom panels.

5.3.9. Control Board replacement AL-X13

The aluminum analyzer has 2 electronic boards responsible for managing all elements of the equipment. The control board is responsible for controlling the measurement process, a failure in this board usually causes a complete failure of the equipment.

To replace the AL-X13 control board, we will remove the AL-011 lower cover. In this way we will be able to freely access the disassembly of this electronic component.



Pos.	Ref.	Description
1	AL-X04	SSR power board
4	AL-X13	Control board
7	DIN 912	Allen screw M3x5

Fig. 5-22. Analyzer bottom view.

To release the control board, we will remove all the connectors that are located on the board and the 4 M3x5 screws that fix the board to the hexagonal spacers.



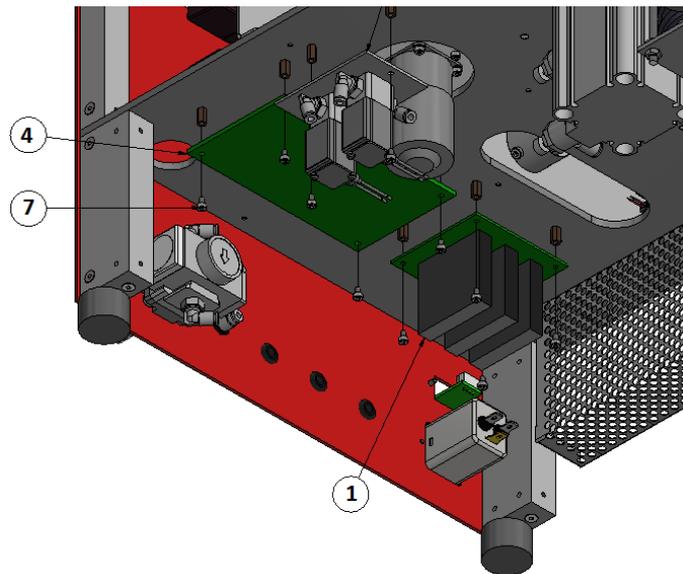
Fig. 5-23. Electrical connection view of the AL-X13 control board of the analyzer.

The assembly of the new electrical board will be carried out in reverse order. (a) mechanically fixing the board using the M3x5 screws; (b) replacing the connectors each in their corresponding connector and (c) replacing the bottom cover

5.3.10. Power board SSR replacement AL-X04

The aluminum analyzer has 2 electronic boards responsible for managing all elements of the equipment. The power board SSR being responsible for controlling the equipment's heating systems. The heating of the expansion vessels (OVEN) and the heating of each of the housings of the 2 glass crucibles.

To replace the AL-X04 control card, we will remove the AL-011 lower cover. In this way we will be able to freely access the disassembly of this electronic component.



Pos.	Ref.	Description
1	AL-X04	SSR power board
4	AL-X13	Control board
7	DIN 912	Allen screw M3x5

Fig. 5-24. Analyzer bottom view.

To release the control board, we will remove all the cables from the terminals and the 4 M3x5 screws that fix the card to the hexagonal spacers.



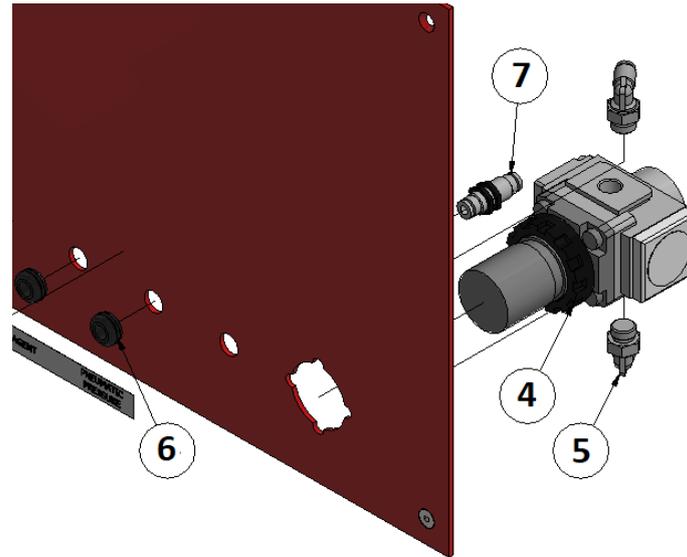
Fig. 5-25. Electrical connection view of the SSR AL-X04 board.

The assembly of the new electrical board will be carried out in reverse order. (a) mechanically fixing the card using the M3x5 screws; (b) repositioning the cables in their corresponding terminals and (c) replacing the bottom cover.

5.3.11. Replacing the Pressure regulator

The pressure regulator allows the internal pressure of the analyzer to be adjusted from the laboratory's compressed air line.

To replace the AL-127 pressure regulator we will remove the rear panel to easily access the disassembly of this element. Next, we will loosen the blue polyurethane gas inlet and outlet tubes and the external nut that fixes the pressure regulator to the AL-015 rear panel, leaving the pressure reducer completely free.



Pos.	Ref.	Description
1	AL-015	Rear cover
4	AL-127	Pressure regulator
5	AL-136	Elbow fitting 1/8 - TUBO 4

Fig. 5-26. Pressure regulator view disassembly

The assembly of the pressure regulator is carried out in the reverse order. Once the pressure reducer is installed, we must set the pressure to around 1.5 Kg/cm², never higher than 1.6 Kg/cm², which is the maximum pressure measured by the pressure transducer.

To adjust the pressure to 1.5 Kg/cm² we will use the PC application. We connect the analyzer, establish a connection with the PC application, insert the crucible into its housing and lower the piston (see manual functions section 4.1.6). With the pressure reducer completely closed, we open the pneumatic pressure valve (see manual functions section 4.1.6).

The pressure measurement is shown in the graphic representation of the reading of the pressure transducers (see section 4.5). As we increase the pressure in the AL-127 pressure reducer we can follow this increase in pressure by observing the graphic representation, thus setting the pressure at the desired value.

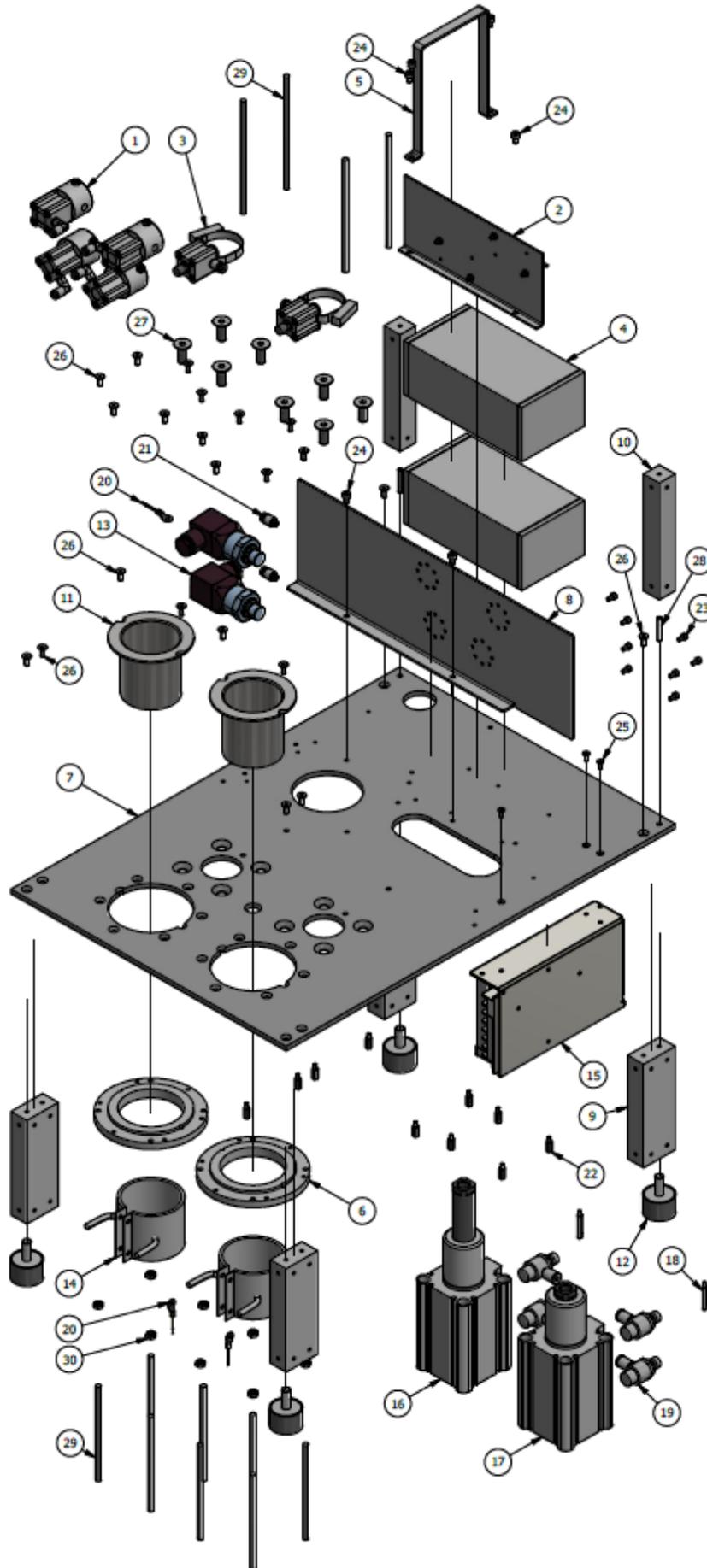
5.4. Complete breakdown of the analyzer

The following images and associated tables show the basic components of the AL-402-2023 aluminum analyzer

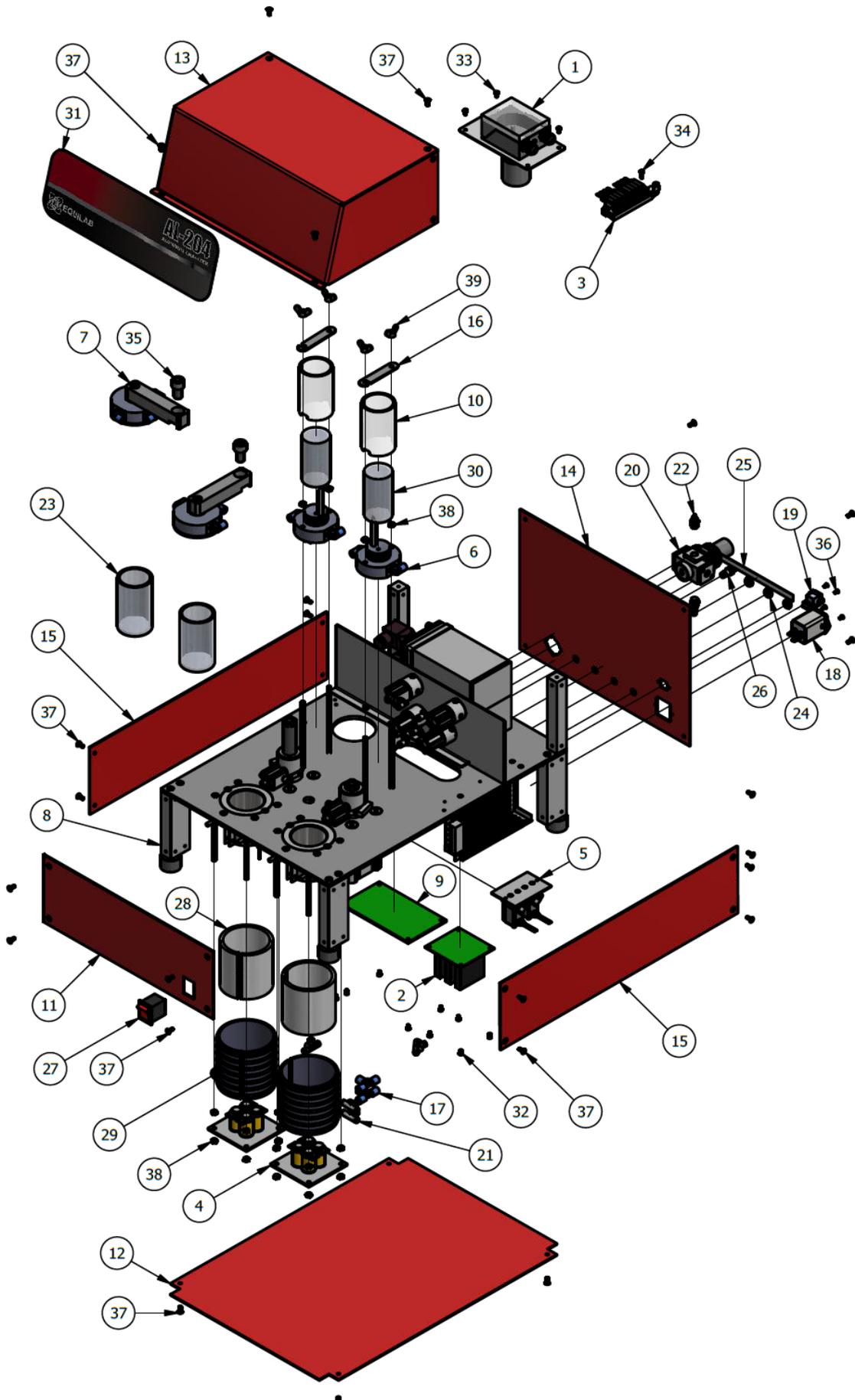
A detailed explanation of the assembly and disassembly of the different elements is included in the previous sections. If the user needs more details on maintenance operations, contact the manufacturer.



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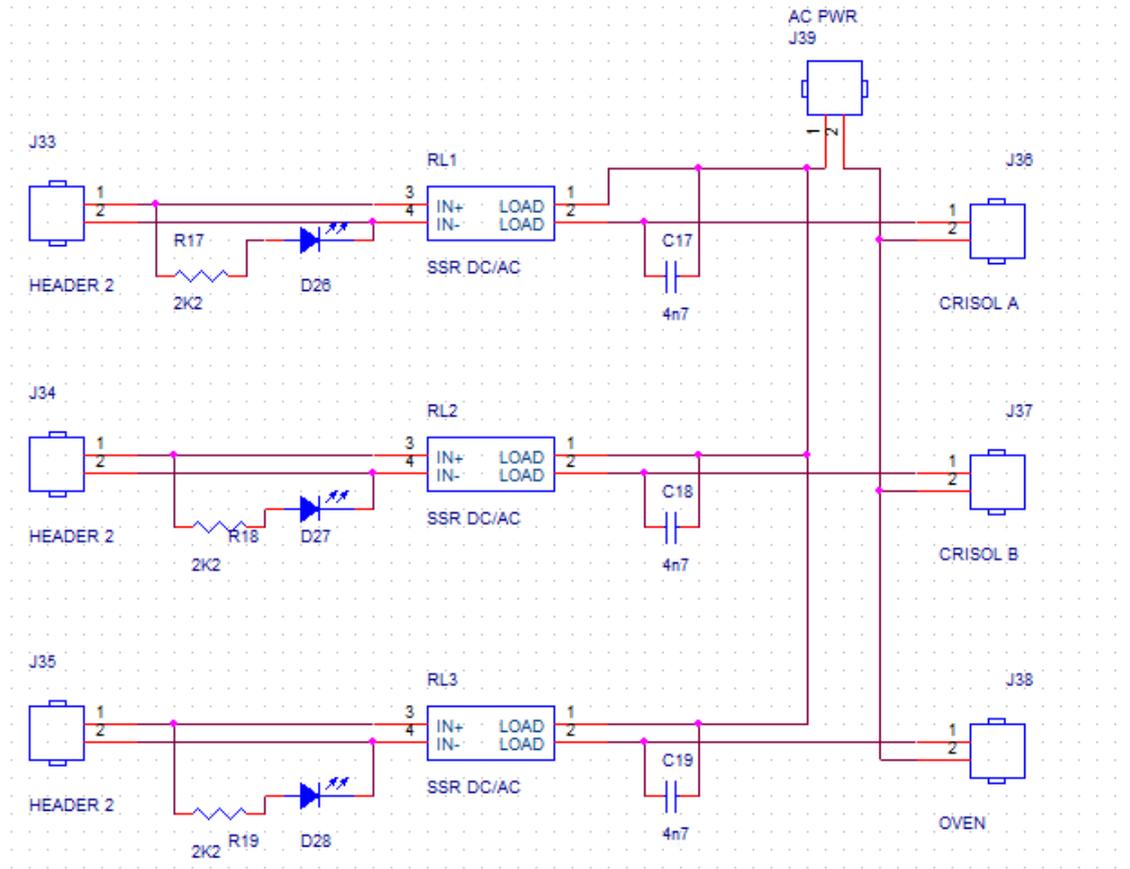
#	CTDAD	REFERENCE	NAME
1	4	AL-X02	Pinch valve assembly
2	1	AL-X07	Heating plate
3	2	AL-X10	Glass crucible fixing assembly
4	2	AL-001	Volume
5	1	AL-002	Volumes fixation
6	2	AL-004	Spacer
7	1	AL-005	Base plate 2 cup
8	1	AL-017	Thermo plate
9	4	AL-022	Aluminum support base plate
10	2	AL-024	Aluminum support upper
11	2	AL-025	Crucible housing
12	4	AL-105	Feet
13	2	AL-111	Pressure transducer
14	2	AL-112	Heating resistances
15	1	AL-113	Power supply
16	1	AL-121	Left piston
17	1	AL-122	Right piston
18	2	AL-129	Position detector
19	4	AL-134	Pressure regulator
20	3	AL-139	NTC Temperature Sensor
21	2	AL-151	Straight fitting tube 4 – M5
22	10	SEPARADOR	Hexagonal space bar M-H M3x10
23		DIN 912	Allen screw M3x6
24	6	DIN 912	Allen screw M4x6
25	3	DIN 7991	Countersink screw M3x8
26	22	DIN 7991	Countersunk screw M4x10
27	8	DIN 7991	Countersink screw M8x20
28	2	DIN 913	Grub screw M4x20
29	12	DIN 976	threaded rod M5x113
30	8	DIN 555	Nut M5



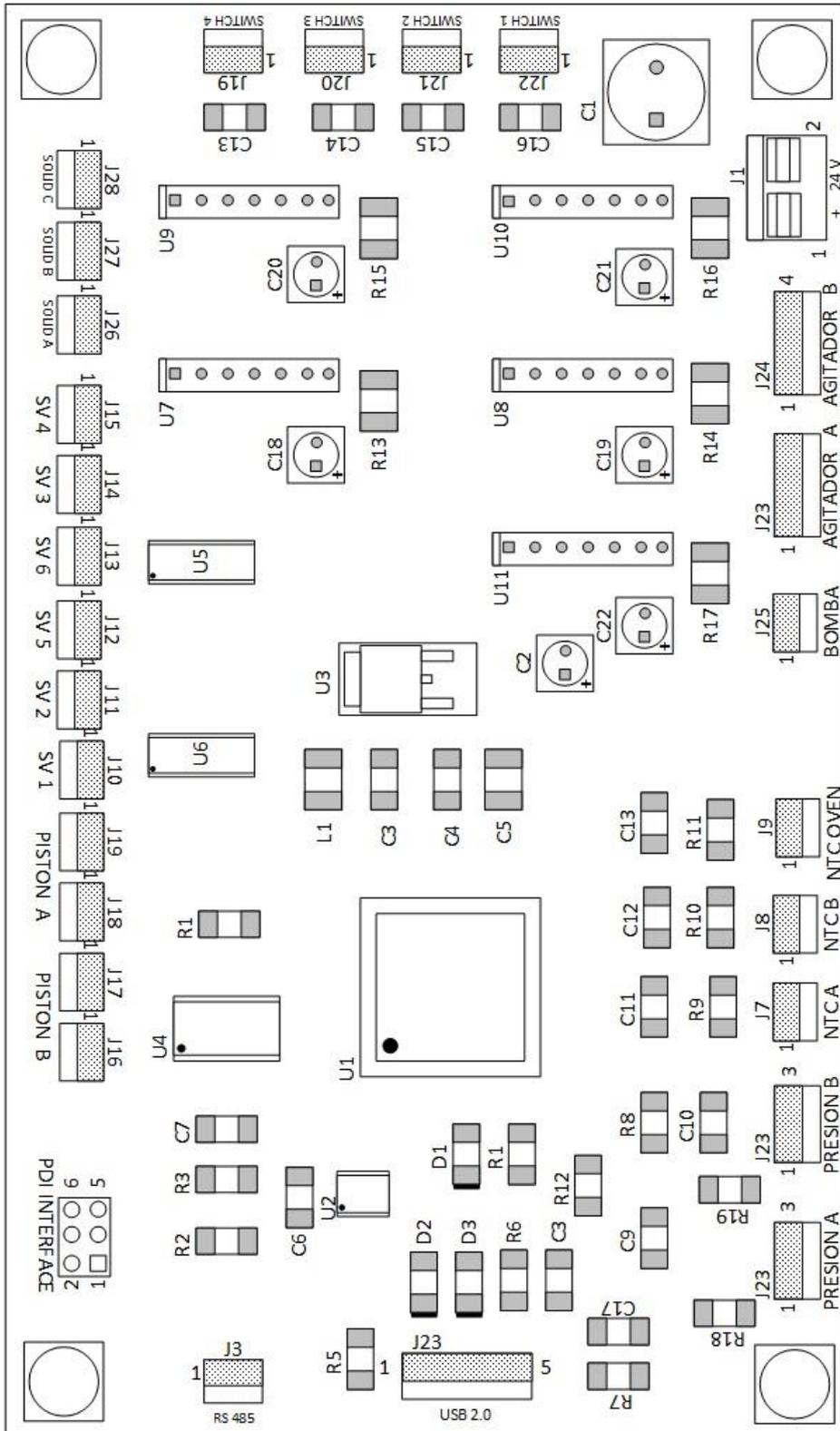
#	CTDAD	Reference	NAME
1	1	AL-X01	Peristaltic pump assembly
2	1	AL-X04	SSR power board
3	1	AL-X05	Valve island assembly
4	2	AL-X06	Magnetic stirrer
5	1	AL-X08	Solenoid valve assembly
6	2	AL-X09	Kitasatos base
7	2	AL-X11	Closure of the crucible assembly
8	1	AL-X12	Principal structure
9	1	AL-X13	Control board
10	2	AL-X14	Methacrylate protector
11	1	AL-010	Front cover
12	1	AL-011	Lower cover
13	1	AL-014	Top cover
14	1	AL-015	Rear cover
15	2	AL-016	Side cover
16	2	AL-031	Metal retaining bar
17	2	AL-103	Polyacetate fitting for 4 mm tube
18	1	AL-114	Power Line Filters fuse holder
19	1	AL-120	USB connector
20	1	AL-127	Pressure regulator
21	4	AL-135	T Fitting for 4 mm tube
22	2	AL-136	Elbow fitting 1/8 - TUBO 4
23	2	AL-144	Crucible glass
24	3	AL-155	Plastic grommet
25	1	AL-156	Dymo tags
26	1	AL-157	bulkhead connector 4 mm tube
27	1	AL-159	Main switch
28	2	AL-160	Glass wool
29	2	AL-161	PVC protection
30	2	AL-167	Kitasato Glass
31	1	AL-173	Adhesive cover
32	10	DIN 912	Countersunk screw M3x5
33	4	DIN 912	Countersunk screw M4x6
34	2	DIN 912	Countersunk screw M3x10
35	2	DIN 912	Countersunk screw M12x20
36	4	DIN 7991	Countersunk screw M2.5x8
37	28	DIN 7991	Countersunk screw M4x6
38	20	DIN 555	Nut M5
39	4	DIN 315	Wind nuts M5

6. Electronic diagrams of the analyzer

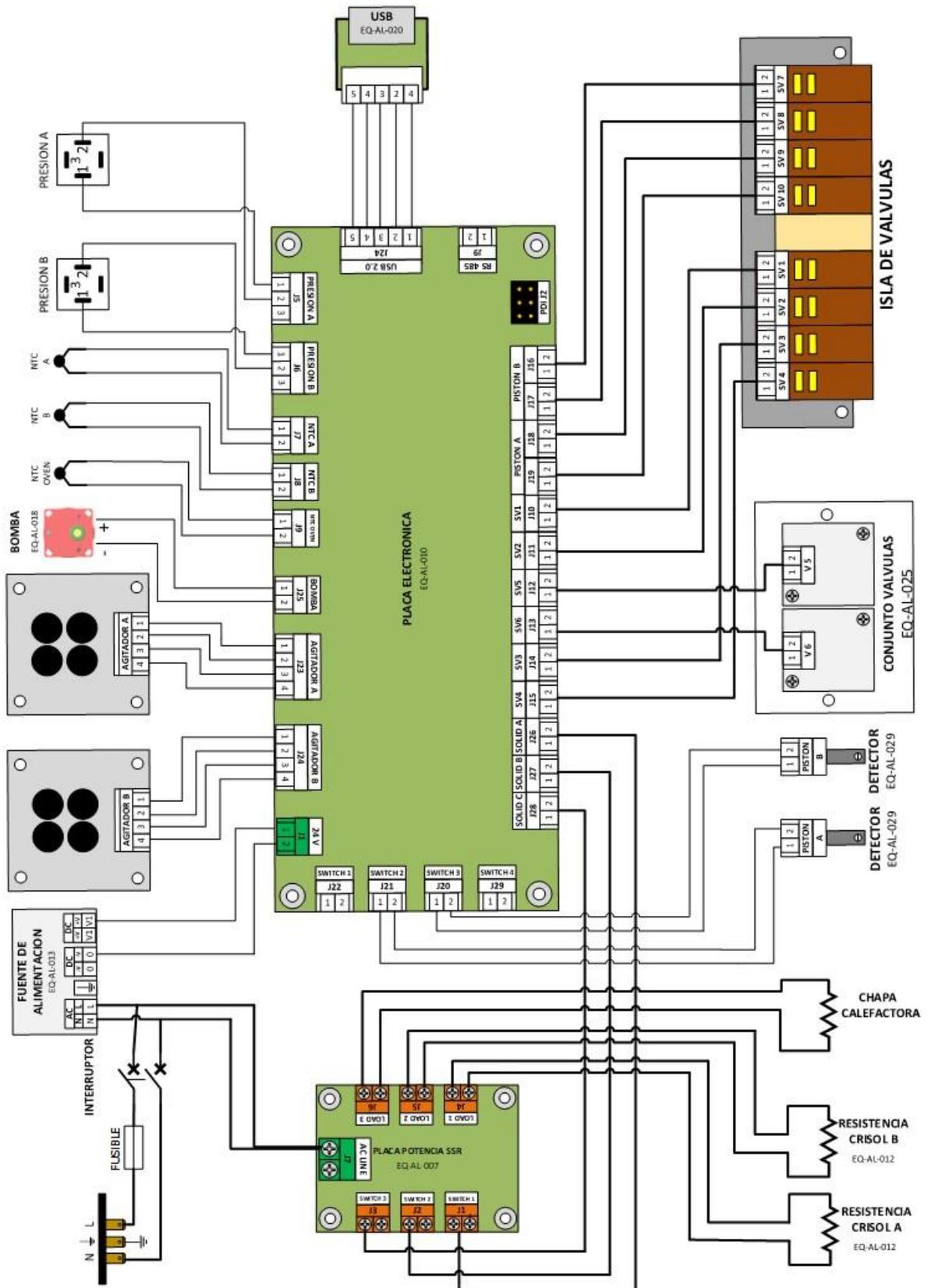
6.1. Temperature control circuit



6.2. Component positioning



6.3. Wiring analyzer



7. Annex

7.1. Reagent A preparation.

Reagent A is a 20% - 30% w/w sodium hydroxide dissolution.

To prepare the reagent A dissolution, weigh around 200 g of NaOH for each liter of water. The dissolution is highly exothermic, careless preparation **can cause heat and chemical burns**.



The preparation of this dissolution must be carried out by specialized staff.

The reagent A bottle must have the following pictogram.



Hazard statements:

H290 - May be corrosive to metals

H314 - Causes severe skin burns and eye damage

Precautionary advice

P280 - Wear protective gloves/protective clothing/goggles/mask

P301 + P330 + P331 - IF SWALLOWED: Rinse mouth. DO NOT induce vomiting.

P302 + P352 - IF ON SKIN: Wash with plenty of soap and water.

P305 + P351 + P338 - IF CONTACT WITH EYES: Rinse carefully with water for several minutes. Remove contact lenses when present and can be done easily. Continue with washing.

P310 - Immediately call a POISON INFORMATION CENTER or doctor.

P390 - Absorb the spill so that it does not damage other materials.



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