TECHNICAL NOTE 31

Automation of the EMGA-900 Analyzer Series: Fully integrated accessories with simplified operations

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1 Introduction

Whether you are working for quality control in the steel industry or in an R&D laboratory for new materials our Oxygen, Nitrogen and/or Hydrogen elemental analyzers will give you very accurate and repeatable results. Thanks to the improved NDIR and TCD detectors, a standard deviation of 0.02 ppm is obtained for Oxygen and Nitrogen (World best precision) and 0.04 ppm for Hydrogen together with wide measurement ranges allowing analysis of high concentration samples as well as traces.

With the EMGA-900 series, the "fully supported accessories" design approach leads to easy and simple operation. The "automation systems" (Cleaning and Crucible loading) will facilitate training new operators and reduce results bias between operators.

As this new generation's models have been optimized to fit user's requests they should address almost any analytical problem you will encounter.

2 Instrumentation

2.1 Principle (see figure 2)

The sample is loaded into a graphite crucible which has been placed on the lower electrode and then elevated to make contact with the upper electrode of the impulse furnace. A high current passes through the crucible to create a high temperature (up to 3,000°C).

The gases extracted during the fusion are directly analyzed after the dust filter.

The Oxygen concentration is measured by two non dispersive Infra-red analyzers (NDIR) ad CO and CO₂, depending on the concentration, in order to achieve very good accuracy across the full measurement range.

The Nitrogen in the sample is extracted as nitrogen gas (N_2) and its concentration is determined by a thermal conductivity detector (TCD).

The Hydrogen is measured with NDIR as H₂O after H₂ is oxidized by a CuO converter.



Figure 1: Model EMGA-930 for analysis of O/N/H concentration in solids





Gas flow diagram

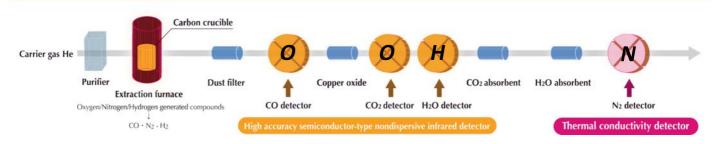


Figure 2: Gas flow diagram of the EMGA-830/EMGA-930

3 General Description

As the instrument has been developed with integrated automation, the result is a compact design with a totally safe robot. The schematic of the automation system is detailed below.

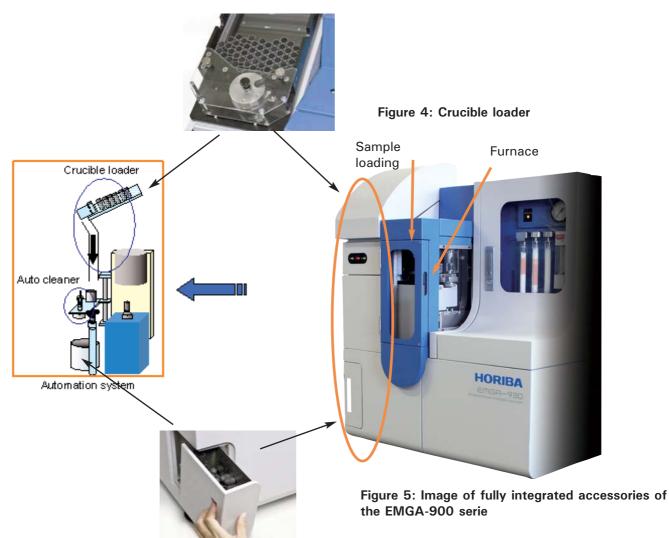




Figure 6: Disposal box



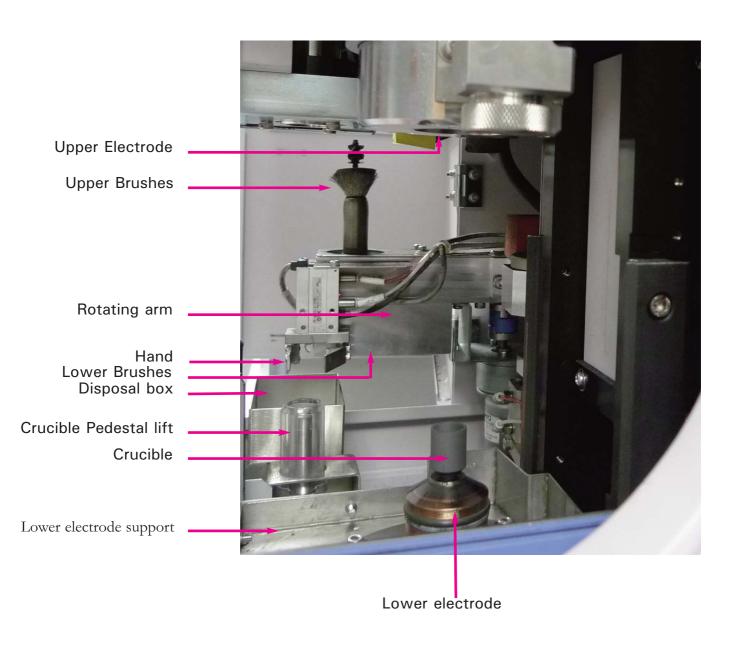


Figure 7: Picture of the autocleaner and the autoloader systems of the EMGA-900 series



4. Automatic cleaner description

The automatic cleaner is composed of different parts:

- Hand:

The rotating arm is equipped with a hand that can pick up or release the crucible. It is fixed on a vertical axis that can move to three positions: crucible pick up on the pedestal, crucible placement on the lower electrode and crucible release into the waste box.

- Rotating arm:

This block is placed on a mechanism that can move horizontally to place the brushes between the two electrodes when the furnace is open and vertically elevated to brush the upper electrode. The lower electrode will also be elevated to be cleaned by the lower brushes. The upper electrode brush is made of stainless steel. The lower brush is composed of two different brushes: one in stainless steel for the centre of the electrode, the other one in nylon for the side of the electrode.

- Disposal box

This box is metallic and can contain up to 200 used crucibles. It's magnetically held in position and slides easily to discard used crucibles.

- The vacuum cleaner:

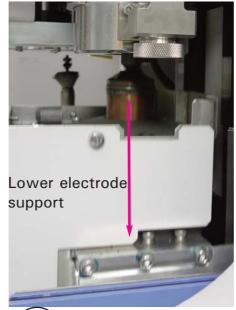
A specially adapted vacuum cleaner is delivered with the instrument to evacuate dust during cleaning. It includes an optimized dust trap.

- Sensors

Several sensors check the correct sequences of events (rotation of the arm, presence or not of a crucible, up and down movement of the lower electrode block, etc.)

Process description

At the end of an analysis the furnace is opened (1). The used crucible is picked up and discarded by the robot's arm (2 to 4). Then the rotating arm moves horizontally between the two electrodes (5) and then vertically until the upper brush arrives at the upper electrode's contact (6). Then the lower electrode block comes up until the lower electrode is touching the lower brush. The two brushes turn for few seconds to clean the electrodes. The vacuum is on during cleaning (7). Finally the sequence reverses so the instrument is ready for a new crucible to be loaded (8) to (10).



End of the analysis Furnace opening





Robot removes old crucible (2 to 4)



Arm rotation



3 Crucible catching and removing

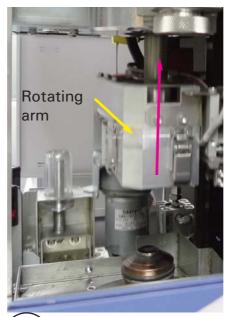


4 Crucible disposal

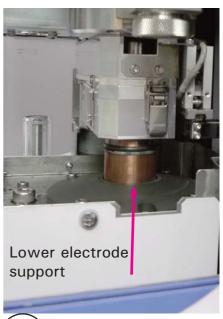
Automatic furnace cleaning (5 to 9)



 $_{5}$ Arm rotation



 $\binom{6}{6}$ Arm up

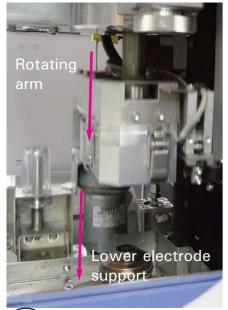


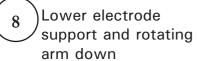
Elevatation of the lower electrode support, rotation of brushes for cleaning and vacuum on for 5 dust removing.





End of furnace cleaning (8 to 10)







Arm rotation



Ready fo crucible loading

5. Automatic crucible loader description

The automatic crucible loader is composed of different parts:

- Cassette:

This part is a stocking container for crucibles waiting to be used for analysis. The cover can be removed in order to transfer a 100 piece crucible box directly into the cassette without hand contact. A rotating wheel with a crucible shape hole selects one crucible and brings it to the pedestal. Invert the transparent cover, makes it posible to use short or long crucibles.

- Crucible Pedestal lift

This pedestal is composed of a plastic tube where the crucible stands. This tube goes up and down to bring the crucible to the hand's level.

- Hand

The same mechanism used for cleaning is also used to transfer the crucible from the pedestal to the lower electrode.

- Sensors :

For cleaning several sensors check the correct sequences of events, for example:

One checks if there are enough crucibles inside the cassette. A message appears in the software when the quantity of crucibles is too low (can be adjusted). Another checks if the crucible has been placed on the pedestal. If not a second try is made

Process description

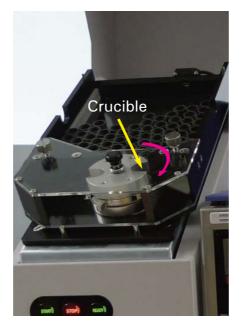
Crucibles stored in a cassette are guided one by one by rotary wheel (1a), as the pedestal goes up to pick it up (1b). When arriving over the pedestal (2a), the crucible is transferred onto it (2b). Then the pedestal goes down (3), the hand comes to pick up the crucible (4) and places it on the lower electrode (5). The rotating arm comes back to stand by position (6). Finally the lower electrode support goes up (7) and the furnace closes the next analysis (8).







Rotation of the wheel to bring a crucible above the pedestal



Rotary wheel



Crucible drop on pedestal

Elevation of the pedestal lift to take the crucible



1b) Pedestal lift up



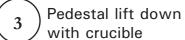
2 b Pedestal lift down with the new crucible





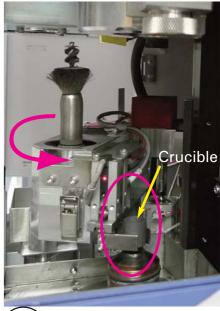
Robot places new crucible (3 to 7)







Pick up of the crucible by the hand

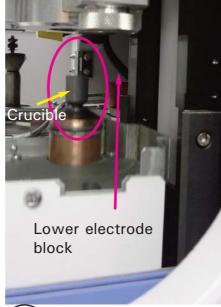


Positioning of the crucible on the lower electrode

Ready for next analysis



6 Cleaning arm back to stand-by position



7 Closing of the furnace







6 Automatic Gas doser description

This feature is used to calibrate all 3 elements using pure gases instead of solid samples (primary calibration). A fixed volume of gas is automatically injected in the carrier gas. Selection of the concentration in the cylinder determines the calibration level.

For instance:

1% CO in 99% He, which is equivalent to 70 ppm O for a 1g sample

 $1\% N_2$ in 99% He which is equivalent to 125 ppm N for 1g sample

1 % H₂ in 99% He which is equivalent to 9 ppm H for 1g sample

The complete operation is controlled by the software. When starting calibration and selecting "gas", a dialog box will ask for concentration of the reference gas and all calculations will be done automatically.

7 Automatic sequences

With integrated automations, operations are much simpler and faster. Just enter sample name, weight and put the sample in the EMGA: Analysis starts immediately and all operations are done fully automatically untill the next sample. It is total "hands clean" use, so the operator doesn't need to touch crucibles anymore and doesn't have contact with carbon dust.

Furthermore results are obtained 40 s before the traditional way of functionning and the operator can use the time used by the automatic sequence to prepare the next sample (figure 3).

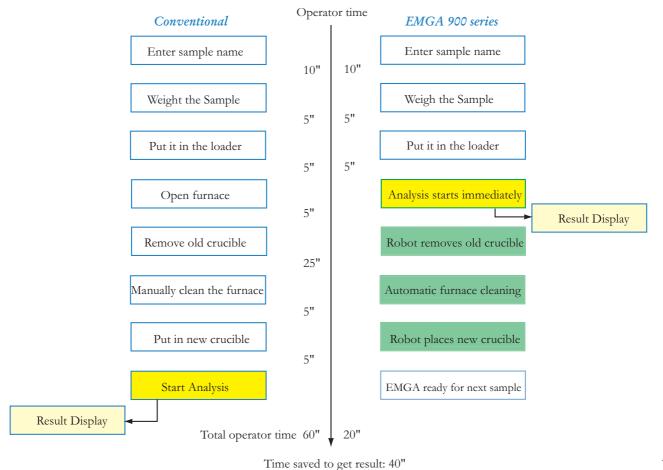


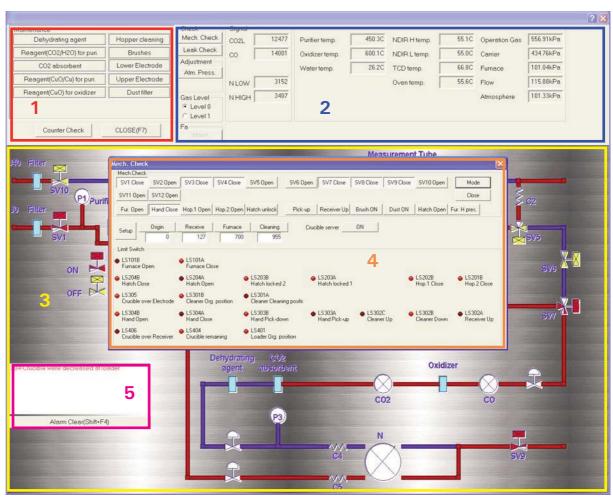
Figure 3: Gain in time for the operator and to get the result (green boxes are fully automatic operations)





8. Maintenance

This maintenance screen gives total access to monitoring and diagnostics of the instrument, particularly, the automation mechanism and sensors. Description of the different functions of this screen:







1 - Maintenance Navigator:

Daily maintenance is made easier by an illustrated description of the maintenance procedures. The screen shows the example of dehydrating agent replacement. When clicking on the "dehydrating agent" box, this window appears. Its right part describes the operations step by step. By clicking on one box, a video or a picture appears on the bottom of the screen with comments on the left. On the top of the window, the location of the current operation is pointed to a 3D schematic of the instrument that can be rotated

2 – Monitoring:

This area displays in real time the characteristic signals of the instrument: you can check detectors electronic signals as well as different temperatures (reactor, detectors, water, etc.) and pressures (incoming fluids, furnace, etc.).

3 - Interactive flow schematic:

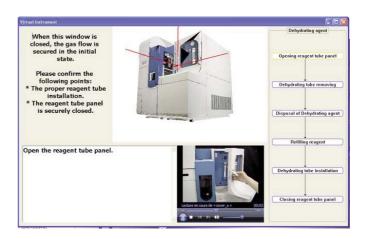
This schematic explains the gases progression inside the instrument. Furthermore it allows knowing the status of each solenoid valve and which part of the circuit is under pressure.

4 - Mechanical check:

This window is especially dedicated to the mechanism movements and status. You can activate independently every moving part of the instrument by a simple click. You can also check the status of each sensor.

5 - Alarm windows:

This small window displays error messages which are easily understandood.



9. Conclusion

The EMGA-900 analyzers have been designed with integrated automation to optimize operations: the result is a compact instrument with a wide measurement range for Oxygen, Nitrogen and Hydrogen while keeping an excellent precision all over the range. As operations are simplified, operator training is easier and results are less dependent on the users. The required time in front of the instrument is reduced and allows more time for other tasks.

The "World best precision" is achieved thanks to crucial operations done automatically: cleaning and crucible handling are always done exactly in the same and with the same high efficiency.

Finally the gain in terms of time is very important and results are displayed at least 40 s before conventional systems.

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