

Modular Automation for Carbon/Sulfur, Oxygen/Nitrogen and Hydrogen Analysis

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1 Introduction

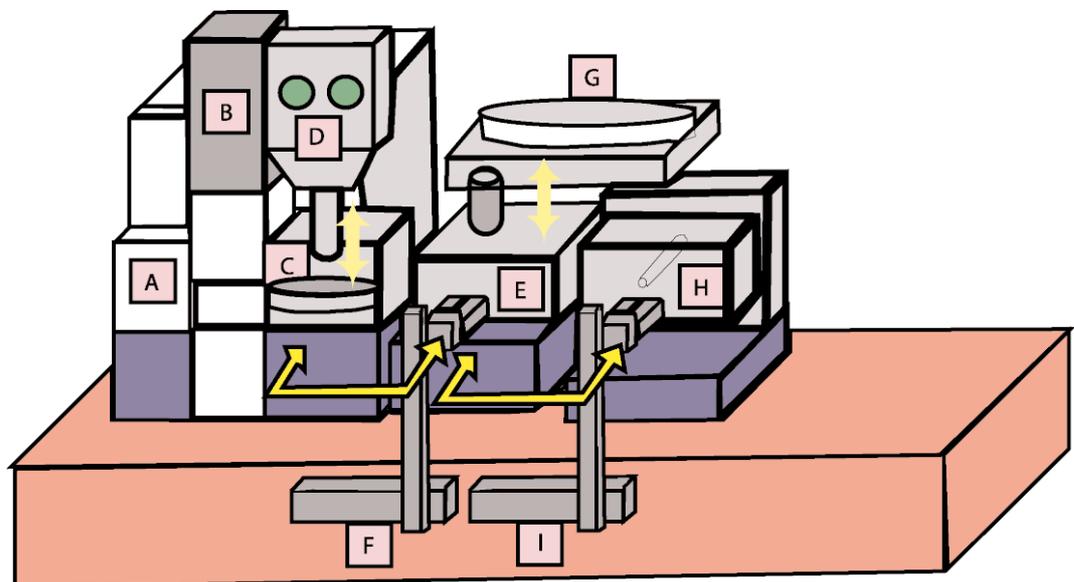
Hot extraction and carrier gas analysis is currently in use in the material industry to measure C/S, O/N and H in solid samples during the process, as well as in final products. Due to the current market and competition, the producers are increasing the quality control while at the same time trying to lower the cost of analysis.

In order to answer these growing requirements, Jobin Yvon is the first company offering partial to full automation for C/S, O/N and H analyzers. With such flexible solutions the number of analyses can be increased together with a significant cost reduction.

It is possible to add to the instruments (Figure 1) some standard modules which allow for partial or complete automation. By selecting the proper modules, it is possible to meet any type of requirement. With partial automation, only some of the functions are automatic while with complete automation the system can operate 24/7 without any human intervention. The instrument is field upgradeable, resulting in the on-site addition of new modules as required.

2 Description of the automation modules

Letters indicated refer to the module as shown in Figure 1.



- | | |
|-----------------------------------|---------------------------|
| A Dust box | E Automatic Weighing Unit |
| B Autocleaner | F Translation Robot 1 |
| C Autosampler | G Sample Stocker |
| D Automatic Accelerator Dispenser | H Crucible Stocker |
| | I Translation Robot 2 |

Figure 1: Standard modules for automatic C/S analyzer

The first module is the Autocleaner (Figure 1, B). It is a very convenient tool because it will automatically clean the furnace after each measurement. The cleaner uses two mechanical brushes driven by a compressed air cylinder. The repeatability of the measurements is increased compared with a manual cleaning after 10 or 20 measurements. The time savings is very significant.

The Autosampler (Figure 1, C and Figure 2) is the second most selected module (see picture). It is a 20 position turn table with a robot arm. The customer will weigh the sample, place it in the crucible with the accelerators, and position the crucible on the turn table. The remaining operations will be done automatically: opening the furnace, removing and discarding the previous crucible, putting the new one in the furnace, running the measurement and storing the results.



Figure 2: EMIA Autosampler

In addition, an Automatic Accelerator Dispenser (Figure 1, D) can be installed on the top of the autosampler. It will automatically deliver the selected quantity of accelerator into the crucible. It is possible to introduce up to 3 different accelerators (i.e. Tungsten, Tin, Iron, Copper, Nickel ...) with different weights for each, as well as different weights from one measurement to the next, according to the sample.

If the Automatic Weighing unit (Figure 1, E) is added, the crucible is placed on the balance, the sample is added and weight validated by the operator and all the other steps are performed automatically. The Auto Weighing unit consists of a balance and a Translation Robot (Figure 1, F) that will move the crucible from the balance to the turntable of the autosampler.

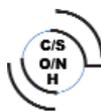
Addition of the Sample Stocker and the Crucible Stocker provides a fully automatic system. The Sample Stocker (Figure 1, G) is a 48 cup turntable, allowing up to 48 samples to be stored in the individual stainless steel cups. The name and identification for each sample are stored in the computer. The Sample Stocker is placed on the top of the balance unit, and the samples are dispatched one by one for the measurements.

The Crucible Stocker (Figure 1, H) can hold up to 100 crucibles. These crucibles will pass in a resistance furnace at 1000° C in order to remove contamination. This module also includes a Translation Robot (Figure 1, J) to move the crucible from the pre-burn furnace to the balance.

3 Automatic operation

The completely automatic system operates in the following fashion:

- The samples (up to 48) are placed on the sample stocker; name and ID are stored in the software.
- A robot takes a clean crucible in the crucible stocker and places it on the balance.
- The balance is tared, and the selected sample drops into the crucible. The sample weight is sent to the software.
- A second robot takes the crucible on the balance and brings it on the autosampler turntable. The selected accelerators are delivered in the crucible.
- The furnace has been cleaned and the door is



open. The robot arm removes and discards the previous crucible, and places the new one in the furnace.

- The measurement begins and the results are automatically stored. A complete cycle is about 2 to 4 minutes depending on the sample and the settings.

- As the system makes use of Parallel Operation the next cycle has already begun. The instrument operates 4 crucibles, allowing 4 operations simultaneously: while sample #1 is measured, accelerators are added to sample #2, the balance is weighing sample #3, and the robot takes a new crucible for sample #4.

The software offers many features, with the quality and traceability of the results as a constant priority. Every sample will have a "recipe and record". The recipe will contain all the method information with sample pre-treatment, accelerator used, temperature curve and all measurement conditions. The record/report will contain the historical data, such as standards used for the calibration, calibration curve, and detailed results with extraction pattern. The software includes database functions which allow the user to retrieve previous results using various criteria. All the operations are fully controlled, and any failure at any stage will be mentioned in the record.

In case of an urgent sample coming to the lab, it is possible to make a "stat analysis" to get an immediate result. After this interruption, the normal automatic measurement will resume.

4 Conclusion

It is obvious that such a fully automatic system is a greater investment compared with a basic instrument, but the reward is in the money saved. As an example, for a customer doing 200 measurements per day, where 3 shifts are worked 24/7, the breakeven point comes only after 2 years. In this scenario the user will save more than \$1 per analysis and after 10 years operation, will have saved more than \$750,000 compared with a manual system including all consumables, maintenance, labor and investment.



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