

Real laboratory, virtual laboratory or remote laboratory: What is the most efficient way?

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Abstract— This paper presents an analytical chemistry experiment using a headspace gas-chromatograph (GC). The same experiment is first realized in a classical way (the student must come to the lab), then in virtual mode (all the experiment is simulated), and finally in remote control mode (the student is able to control the experiment from a remote computer). Choosing the appropriate mode not only requires analyzing the financial and technological aspects, but also the knowledge and competences acquired by the student during the lab session. Requirements, technologies, and results are discussed for each mode.

Index Terms— Chemistry, gas chromatography, remote engineering and control, virtual laboratory.

I. INTRODUCTION

A. Generality

In chemical schools, laboratories tend to stay in a traditional setup (the student must come to the lab to make his experiment). This is due to safety aspects and to the chemist's philosophy. Nevertheless several experiments are available in virtual mode, but not in remote mode.

The virtual laboratory allows the institution saving space and costly hardware. Most of remote control discussions essentially highlight the technologies used, the ease of use and the comfort for the student. Through a concrete example (analytical chemistry lab, gas-chromatography analysis experiment), this paper compares the classical approach, the virtual approach, and finally the remote control mode.

This paper discusses not only the technology aspects, but also compares the acquired knowledge via the three different modes (i.e. normal lab, virtual lab, remote lab). This aspect is often neglected when setting up a virtual or remote lab experiment.

B. Experiment proposed

Gas chromatographs (Fig. 1) are used to analyze mixtures containing liquid substances which can be easily evaporated. The liquid mixture to analyze is injected in the system and quickly heated so that it becomes a gas. The mixture is introduced with a vector gas such as nitrogen or helium in a capillary column for separation. All products are analyzed at the end of the column by a detector such as a thermal-conductivity detector (TCD) or a flame-ionization detector (FID).

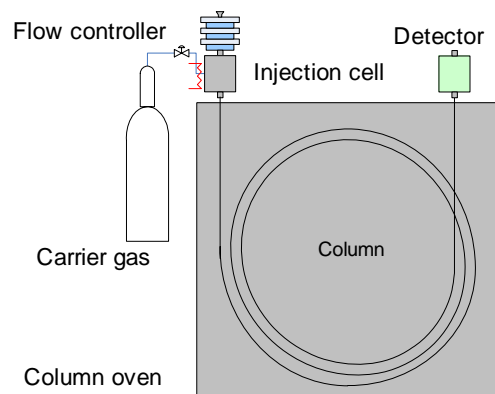


Figure 1. Gas chromatograph

To obtain a good separation, it is necessary to choose the right column (type of column, length and internal section). This directly depends upon the products to be separated, and also from the oven temperature program (Fig. 2) which is correlated with the boiling temperature of the products. The injection method and the type of detection are other important parameters of the analysis.

The resulting signal from the analysis process is called a chromatogram (Fig. 3). This one is analyzed to find the baseline and the different peaks representing the different isolated products. The time used by a product to pass the column is called the retention time (tR). The peak area is proportional to the product quantity.

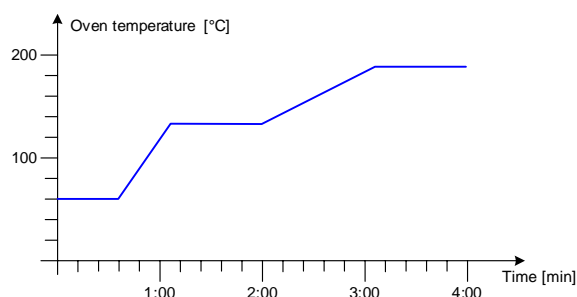


Figure 2. Oven temperature program

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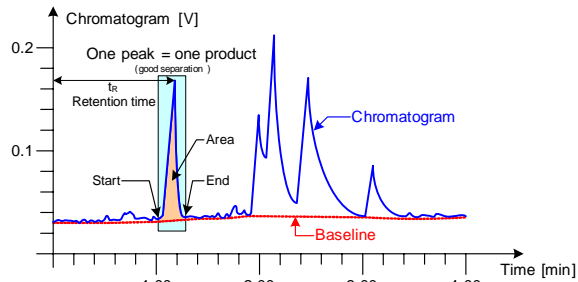


Figure 3. Chromatogram

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A. Installation used

The installation used for this laboratory is a FISON chromatograph with a linear automatic sampler (Fig. 4).

The chromatograph has a serial RS232 interface for programming the method (oven temperature, conditions) and the automatic sampler. The detector is connected to an analog to digital converter (A/D) having a resolution of 22 bits.

B. Control system

A computer controls all the functionalities of the system (Fig. 5).

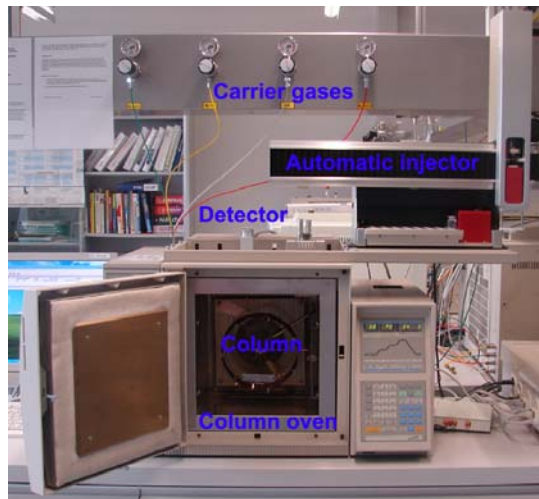


Figure 4. The chromatograph used

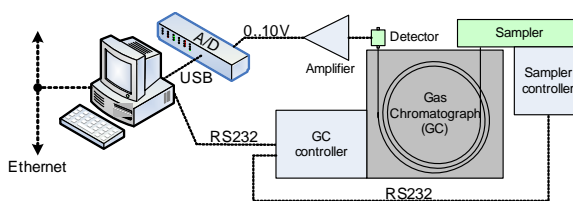


Figure 5. Control system

A dedicated program (delivered with the system) controls the chromatograph and the automatic sampler (Fig. 6). The program is used to edit the method (program temperature), the sample list, and the sample position in the sampler. The system works as well in online mode, as in batch mode with a sequence of analyses.

Each sample gives a chromatogram. The baseline, the retention time and the area are calculated for each chromatogram. The retention time is used to identify a specific product (Fig. 7). The area is used to quantify the identified product.

C. Practical goals of the experiment

Three types of user can be identified.

The beginning student needs to understand and to learn how to use the chromatograph functionalities. A simple experiment, such as alcohols separation (methanol, ethanol, isopropanol and so on), can be realized (Fig. 8). The two goals are firstly to obtain a good product separation and secondly a correct quantification from all components in the mixture.

The intermediate student must identify the components of the mixture. Often the sample requires preparation prior

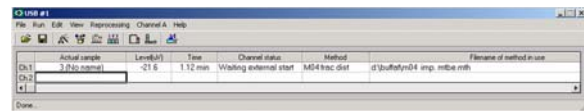


Figure 6. Control program

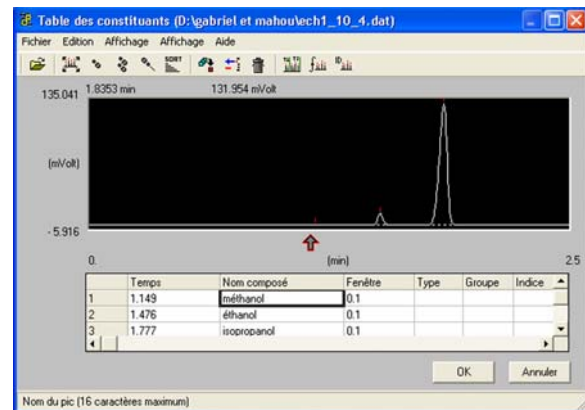


Figure 7. Chromatogram study

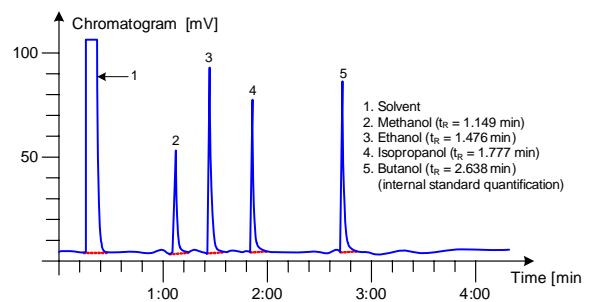


Figure 8. Simple gas chromatography analysis for beginning users

to injection. Solids must be dissolved, over molecules need to be derivatized. The derivatization is a chemical process to modify a compound that produces a new compound which has properties that are suitable for chromatography analysis. The goal for intermediate students is the improvement of the separation method by an appropriate sample preparation.

The advanced student must optimize the analytical method by choosing the column, the injection method, the oven temperature program and the detector. For complex mixtures, the gas chromatograph can be coupled with mass spectrometry. At the end of this laboratory, the user knows the instrument and is able to analyze real mixtures for establishing the composition (qualitative methods) and the concentration (quantitative methods).

D. Conclusion

The first idea of students doing laboratory work is that they do something totally different compared to the theoretical classes. They have the difficulty to correlate the theory and the practice. The future engineers prefer often the laboratory session because they have hard results than they can see.

The observation of the students working in the laboratory is interesting. They are often working in a sequential mode. They make the sample preparation, define the chromatography method, inject the sample, wait on the chromatogram, make a basic analysis on the data and start with the next sample. Usually they do not anticipate the results based the theoretical reasoning. As a consequence, they don't fully exploit the results to plan the next experiment.

The biggest impact from these laboratory sessions is the acquired knowledge on the level of accurateness and precision in work to obtain qualitative and quantitative usable results. Another important aspect is directly related to the encountered problems with the instrument, the system control or the analysis itself. Instrument failures event there are basic require some initiative from the students. They must dialog not only with the Professor but also with technical staff (chemical engineer, mechanic, electrician, computer specialist).

III. VIRTUAL GAS CHROMATOGRAPHY LABORATORY

A. Introduction

The gas chromatography training is subdivided in three stages. The first stage, for the beginners, makes it possible to learn the basic principle of the gas chromatography and the basic handling of the instrument (product separation, product quantification). The second stage, for the intermediate students, introduces the concept of sample preparation. The third stage, for the advanced ones, introduces the complex mixtures analysis and its optimization.

The laboratory for the beginners is usually realized with a large number of students and within a restricted time frame needing therefore a substantial coaching. The costs related to the instruments, the personnel, the material and

the chemicals cannot be completely justified with respect to the acquired knowledge. It is on this stage, that virtual laboratory has a certain potential.

The virtual laboratory for the intermediate students is more delicate. An important part of the laboratory time corresponds to the preparation of the sample. The development of a model for this step sufficiently close to reality requires a considerable investment of resources. However combining the manual sample preparation with automated batch analysis represents a gain of productivity (see the point "Remote batch control")

The students in an advanced laboratory course are facing a whole variety of complex mixtures. Only the optimization can be carry out by means of specific software.

B. Beginner laboratory

Speaking about virtual laboratory to chemistry students is not so easy. The students suppose that if they don't see or touch the instrument, they cannot learn. But in the reality, the time during which they manipulate directly the instrument (Fig. 9) doesn't exceed 10% of their total laboratory presence. The remaining time, they control the instrument over a computer (Fig. 10 and Fig. 11), analyze the chromatogram, and write the report.

The idea is to simulate the real instrument with a virtual chromatograph. A mathematical model is used to generate the chromatogram. The retention time of each substance is determined by calculation. The equation uses the column's polarity, the molecule's polarity, the molar mass, the boiling point, and the oven temperature.

The simulated peak can be symmetric or asymmetric. For symmetric peaks, a simple Gaussian equation is used:

$$p(t) = A \cdot \exp\left(-\frac{(t-t_R)^2}{\sigma}\right) \quad (1)$$

where A is the peak height (amplitude), t_R is the retention time, and σ is the standard deviation correlated with the peak width.

Many mathematical formulas (Lorenz, Voigt, Pearson, and so on) make it possible to model an asymmetric peak. As example, a double sigmoid function is also possible:

$$p(t) = A \cdot \frac{1}{1 + \exp\left(\frac{t-t_R + \frac{w_1}{2}}{w_2}\right)} \cdot \left(1 - \frac{1}{1 + \exp\left(\frac{t-t_R + \frac{w_1}{2}}{w_3}\right)}\right) \quad (2)$$

where A is the peak height (amplitude), t_R is the retention time, and w_i are the peak width at the half of the height.

The baseline is a linear equation with an offset a and a slope b . The noise is added as a random number proportional to a noise value:

$$Baseline(t) = a \cdot t + b + random(n) - \frac{n}{2} \quad (3)$$

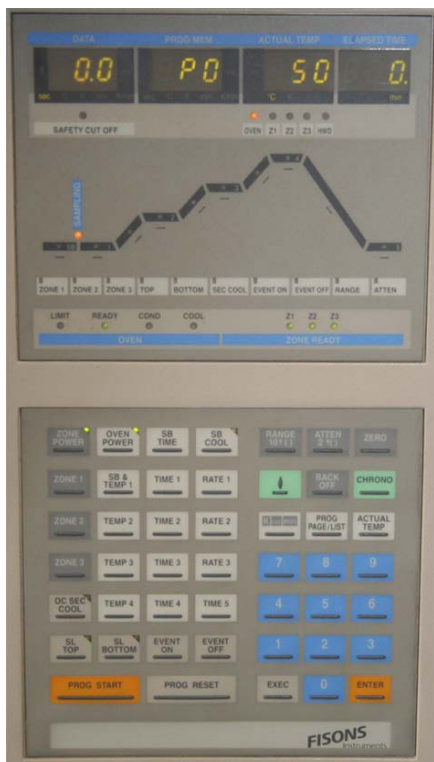


Figure 9. Programming panel

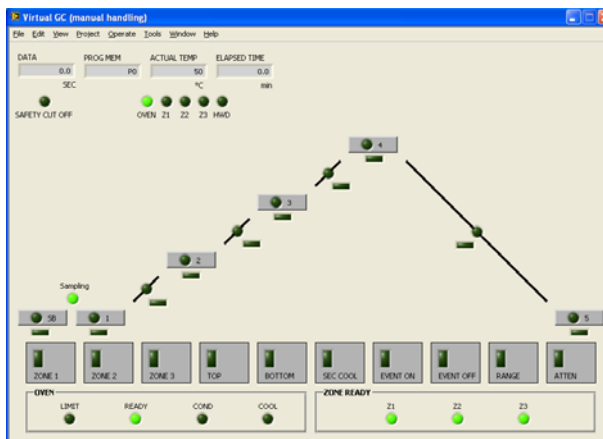


Figure 10. Virtual programming panel (1)



Figure 11. Virtual programming panel (2)

By using the virtual chromatograph, the beginner obtains the same chromatogram (Fig. 12) as if he had worked with the real chromatograph.

The next step will be to change the actual control software (rewritten by Olivier Vorlet, responsible for the automation at the chemistry department of the College of Engineering and Architecture of Fribourg) to include not only the real chromatograph but also the simulated chromatograph.

C. Optimization laboratory

Advanced laboratory in gas chromatography requires more reflexion on the manner of proceeding for obtaining an adequate result. Such results are usually only obtained after time consumes optimization on the instrument. By replacing the real instrument by a simulated one the optimization phase doesn't block the real instrument.

For this part, the software GC-SOS (Capillary Gas Chromatography, Simulation and Optimization Software) from the 4S Company was used (Fig. 13). The program needs one (Table I), two or three real analysis.

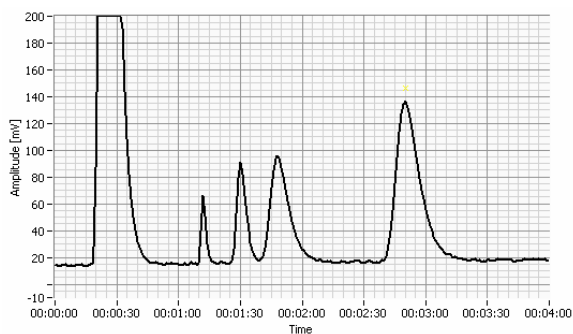


Figure 12 Chromatogram simulated

The temperature program starts at 80°C. After one minute, a slope equivalent to 20°C per minute is applied to reach 100°C. This temperature is maintained during 2 minutes.

By simply modifying the temperature's program, it is possible to simulate a new chromatogram. The program is able to perform an auto-optimization.

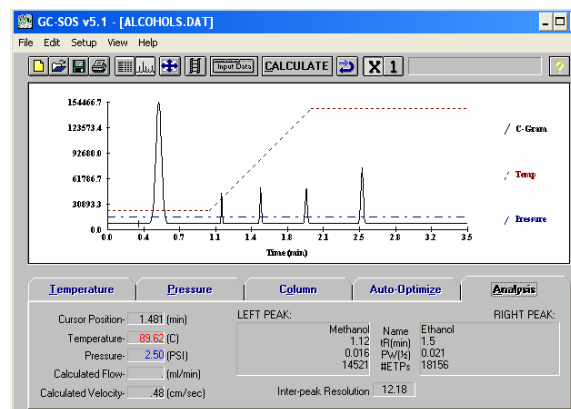


Figure 13. Optimization software (GC-SOS v5.1)

TABLE I.
REAL DATA INTRODUCED IN THE GC-SOS

Compound	Area []	Half width [min]	Retention time [min]
Acetone	913345	0.080	0.51
Methanol	14521	0.016	1.12
Ethanol	19528	0.021	1.53
Isopropanol	16199	0.028	1.99
Butanol	25264	0.035	2.54

D. Conclusion

A gas chromatography analysis lasts about 10 minutes. It's necessary to add the time for the sample preparation, the stabilization time of the instrument, the time to analyse the chromatogram. Finally, one experiment requires between 30 minutes and an hour. During a laboratory session (4 hours), the student is able to realized approximately 5 or 6 experiments. The beginner has difficulty to estimate the amount time necessary. Often it reduces the time for the chromatogram analysis and the time required to plan the next experiment. It makes ten or more experiments (one after the other) until it has the result required. At the end, it worked 8 hours for his laboratory session. By seeing it working, it is possible to explain a better way for planning its experiments.

With a simulator, is able to carry out a significant number of analyses. It is interesting to note, that the idea to offer more time for the reflexion, does not work. The student makes more and more simulations until the desired result is obtained. Another negative aspect of simulation is the absence of dialogue with the laboratory staff. Indeed, the student works only on a computer, on a software. Even if virtual failures are programmed, the user thinks that it's software problems. It dialogs preferentially with the computer specialist. Back to the real instrument, it is often not able to analyze correctly the chromatograph's problems.

The advanced student understands better the utility of an optimization tool, which accelerates its work. It's interesting to see that the student find quickly the self-optimization function from the software (without instruction). After that, he just uses this option and he searches no more other solutions. At the end of the laboratory, he isn't able to optimize without this tool.

It would be finally interesting to limit the number of experiments which can be realized before obtaining the result and to force the student to describe the methodology of the optimization.

IV. REMOTE GAS CHROMATOGRAPHY LABORATORY

A. Introduction

Chemistry remains an experimental science. The modeling of chemistry processes often imposes big abstractions and important simplifications. The virtual experiment is then too far away from reality. Safety aspects in chemistry require the presence of a person to control the installation. The experiment's duration forces the student to spend many hours in the laboratory.

Due to this fact, the first idea was to offer to the student and to the technical staff a possibility to visualize and to supervise the installation and the current experiment. The simplest way to visualize the installation and the laboratory is a video camera. Supervising the instrument and controlling the experiment is today realized with a computer. By enable remote desktop control, the user can control the local machine with a remote computer. Software such as Access Remote PC (Version 4.9 - <http://www.access-remote-pc.com>), Anyplace Control (www.anyplace-control.com), NetOp Remote Control

(<http://www.netop.com>), RealVNC/VNC (Version 3.3.7 - <http://www.realvnc.com/>), and Microsoft Remote Desktop has been tested. Our current machines are all in the Windows XP environment and, in this case, the better choice was to use the tool proposed by Microsoft.

The next step is to control directly the gas chromatograph with a client-server structure (Fig. 14).

This solution was unacceptable. Indeed, the experiment duration is important. When a student has the control of the installation, it works for two or three hours. The students on standby lose patience. They stop looking at if the installation is free. Another aspect is the sample. For the beginner's laboratory, it is possible to prepare in advance the samples and to put them in the automatic sampler. For the other laboratory, student must prepare the sample and put it in the instrument. He must be in the laboratory for these operations. Only after that, he can go out from the laboratory to do his remote experiment!

Finally, a remote batch methodology has been developed. The student prepares the different samples and put them in the automatic sampler (similar to the previous system). The sample information and the method to be applied to analyze the sample are written in a database (Microsoft SQL including Coldfusion). The control system has a connection to the database and downloads the sample information and the method to be applied. At the end of the experiment, the control systems update the database with the chromatogram and, send an email to the user that has required the analysis. For this use, the installation's safety had to be improved.

B. Remote desktop

Accessing to remote desktop is very easy. Microsoft allows to activating the remote desktop service (Fig. 15) and adding users that can access this service.

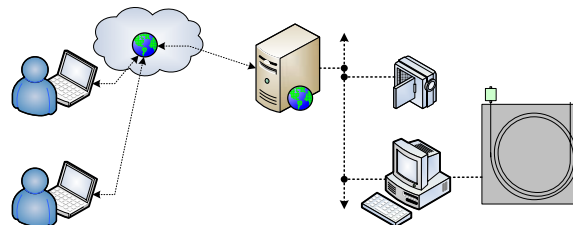


Figure 14. Proposed structure for a remote laboratory

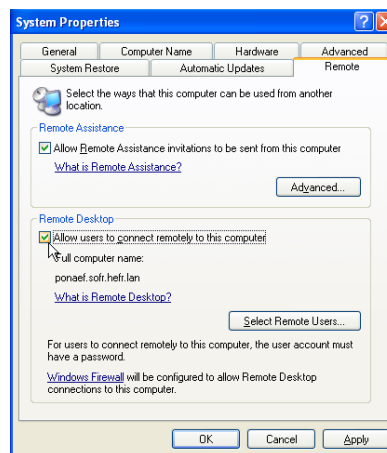


Figure 15. remote desktop activation

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After calling the remote desktop program, the system opens a dialog box to give the address of the remote machine and then the user name and the password (Fig. 16).

At the end of the procedure you are connected to the computer that controls the gas chromatograph (Fig. 17). It's important to note that if a user was connected, the remote desktop closes automatically the local user.

C. Remote batch control

Remote batch control is based on a batch system (Fig. 18). The control system is connected to a database where the experiences to do are saved. Users save on a database over a web interface the sample information and the method. The database generates a code experience. This code is emailed to the user that can confirm the experience. The control system takes one experiences including the method. The method is automatically downloaded on the gas chromatograph. When the instrument is ready, the control system starts the injection and records the chromatogram. At the end of the experience the result is send to the database and an email is send to the user.

Using a batch system connected to a database has allowed integrating the graphic user interface (GUI) as a webpage (Fig. 19). A mail and a browser are sufficient to control the gas chromatograph.

The proposed system is a good compromise to maximize the utilization of the gas chromatograph. One difficulty for the student is to place the sample and to understand that the experience isn't executed immediately. It's a fundamental change in the preparation of the laboratory. The student is forced to think about the experiences executed and the awaited results.

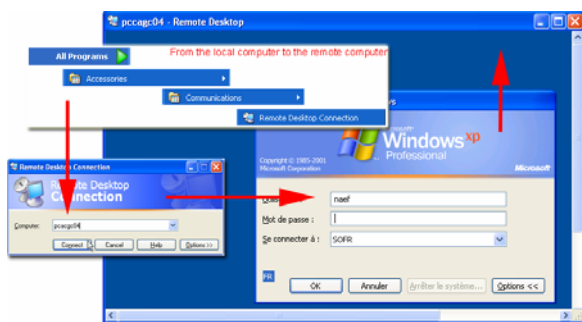


Figure 16. Remote desktop connection

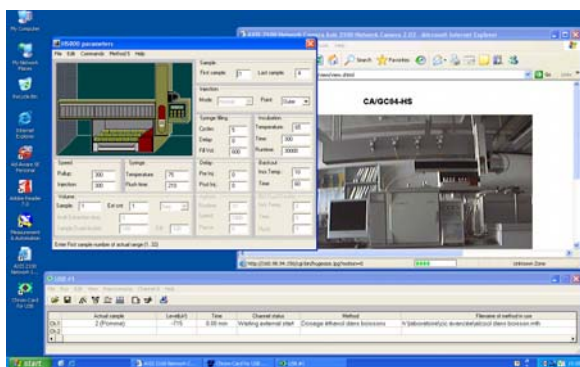


Figure 17. Controlling the instrument with remote desktop

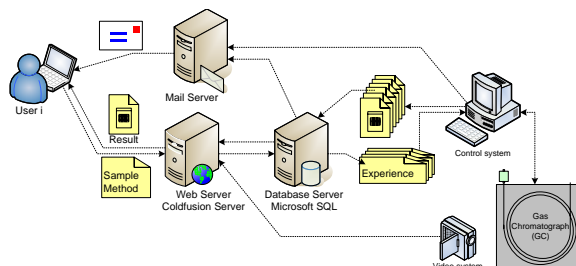


Figure 18. Remote batch control

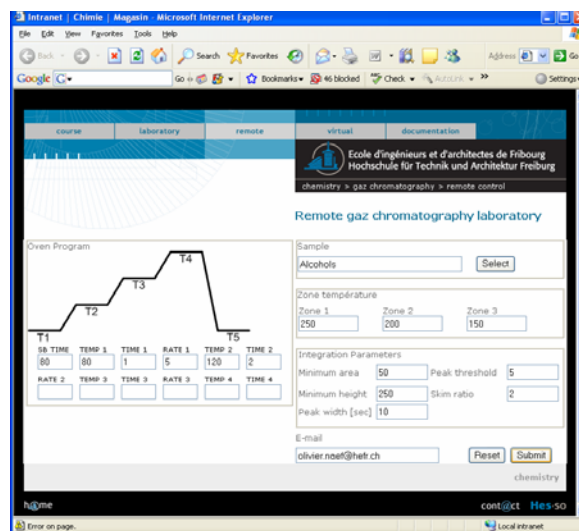


Figure 19. Introduction of the experience and the corresponding method

D. Conclusion

The remote desktop mode is interesting to supervise an installation. It must be limited to the user currently reaching the instrument. The Microsoft tool doesn't make possible to lock the control system. Each user can, in so far as he is allowed with his account, activate a remote connection and log out the previous user. A very strict discipline must be required to prevent that a user doesn't stop the experiment of another user. Finally, the only user having a permanent remote account is the responsible in charge of the instrument's maintenance. He can from time to time authorize a student to register his name on a control system to supervise his experiment from another place.

Direct access to the control software (client – server system) didn't give satisfying results from the point of view of the users. The duration of an experiment (approximately 30 minutes) limits the access to the installation by several students. When a student has the control, he maintains the control as long as possible. Moreover, preparing and placing the sample (for the intermediate students and the advanced students) make necessary to come to the laboratory. The student cannot work only at home.

In the long term, the remote batch control should make it possible to limit the number of instruments by improving the utilisation ratio of each instrument. Often at the beginning, the student doesn't understand why it is not possible to obtain directly the result from the instrument. At the moment where the principle is accepted, the student

wins a lot of time during the laboratory session. The student prepares the samples, places the samples on the sampler, writes the method and the sampling information, and goes out from the laboratory. The student receives automatically the raw data and the treated data "at home". If necessary, simple mathematical tools enable him to take again the data processing.

V. CONCLUSION

Real work in the laboratory requires an important number of instruments (ideally one system for one or two students). Those are much occupied by the beginners and the intermediate students during the laboratory time (4 hours per week). The rest of the week, just one or two advanced students use a part of the laboratory. The technical staff must be present to maintain the whole of the installations. The investments and the maintenance costs for such laboratory are very high.

The students see often the laboratory work as a relaxation time (before having to write the report!) and a more interesting job as the theoretical study. With the laboratory, they obtain a concrete result, directly visible. The students remain very close to the instrument throughout the analysis putting forward the questions of safety and the supervision of the installation. They don't prepare an experience's plan and start their reports.

The utilization of a virtual gas chromatography for beginners shows very quickly the limitation due the relative simple model proposed and the complexity to have a model nearer to the reality. The beginners can be familiarized with the control software, the base of the gas chromatography and the relations between the temperature program, the column used (length and size) and the quality of the mixture's separation. It would be necessary to introduce a limiting number of experiments authorized to improve the reflexion of the students.

The tool for optimization is also well appreciated by the advanced students. The students should be absolutely forced to understand the mechanisms of the optimization to prevent that they use the functionalities of self-optimization without being able to explain the result obtained. They also should be made attentive on the interpretation of the optimized data.

The remote desktop is more a tool for maintenance. It can punctually be used like a tool of cheap supervision system. The video camera brings no more information; the

instrument has just little parts that move, and those remain generally motionless (an injection every 30 minutes!).

Finally the most interesting solution is the use of a remote batch system. This solution makes it possible to preserve the real laboratory by strongly improving the utilization ratio of an instrument. By separating the sample support in two zones, it is possible to preserve an experiment for the beginners and several advanced users can share the instrument the remainder time.

Of course, the best solution remains a combination of the three methods suggested.

VI. ACKNOWLEDGMENTS

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