



## Small-Scale, Low-Cost Analytical Instruments: Extended Opportunities for Learning Analytical Chemistry

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### Abstract

*This contribution challenges the often misleading perception that there is only one way of doing analytical chemistry, i.e., with complicated and costly professional instrumentation. And if a school or university does not possess such instrumentation, or has no access to it, there is no other way to proceed. Here we describe a low-cost spectrometer with a microreaction chamber and a tri-colour light-emitting diode as the light source, which we developed to satisfy the needs for an appropriate low-cost device. The spectrometer can be easily upgraded into several different analytical instruments, e.g., a gas and a liquid chromatograph, a flow injection analysis system with spectrometric detection and a spectrometric microtitrator, all of which enable a sound introduction to the fundamentals of instrumental analytical methods using a hands-on approach and are suitable for real-life applications. More than 60 hands-on experiments for different educational levels and from different chemistry-based and chemistry-related disciplines were developed for this equipment through two European Leonardo da Vinci projects entitled "Hands-on approach to analytical chemistry for vocational schools". The descriptions of the experiments are freely accessible online at the trilingual project websites (<http://www.kii3.ntf.uni-lj.si/analchemvoc2/file.php/1/HTML/Default.htm>). In December 2011 the second project was awarded the first national prize (Apple of quality 2011) for the best Leonardo da Vinci project for the transfer of innovation.*

### 1. Introduction

Rapid technological development requires professionals with excellent analytical chemistry skills to monitor technological processes and their impact on the environment, and to control food safety and people's health. Every society also needs citizens who are aware of how important these activities are for the quality of our lives. Practical work is an essential part of learning analytical chemistry or acquiring an understanding of what analytical chemists do. Professional instruments are very costly and sometimes are not always very suitable for teaching the basic concepts of analytical methods or using these methods in learning chemistry in general education.

European vocational education and training (VET) is undergoing a crisis. A European study [1] indicates that vocational education in the New Member States, in contrast to the EU-15 States, is experiencing a decrease in enrolment rates. The situation in chemistry-based or chemistry-related disciplines is even less favourable. The great majority of pupils at the pre-vocational level adopt a reluctant attitude towards natural sciences [2], considering them difficult, demanding and not very exciting. As a consequence they seldom recognise disciplines such as chemistry, laboratory medicine and food processing as an attractive future career opportunity. Teachers in some countries have experienced a transfer of students towards general education and a decline in the fraction of more capable and highly motivated students in VET. The UK, which has an educational system that differs significantly from the rest of Europe and does not comprise VET, also experiences a lack of enthusiasm and interest in the natural sciences from pupils and students. Traditional teaching and



learning methods do not produce the same learning outcomes as before, and so alternative approaches are needed.

The hands-on approach in teaching and learning is believed to contribute to a better understanding of the concepts being taught. However, this approach is difficult to introduce, if costly instrumentation is required. Therefore, even for modules on instrumental analysis at the tertiary level of chemical education, the activities of the students are, in some cases, limited only to preparatory phases, while the actual measurements are carried out by the demonstrator. The reasons why analytical chemistry is usually neglected at the lower levels of chemical education are many fold. Most commercial instruments are expensive and their operation and maintenance are demanding; therefore, most secondary-school chemistry teachers are not knowledgeable enough to cope with the technology, on the one hand, and the theory, on the other.

We addressed these problems through two EU projects entitled “Hands-on approach to analytical chemistry for vocational schools”. In December 2011 the second project was awarded the first national prize (Apple of quality 2011) for the best Leonardo da Vinci project for the transfer of innovation. The solution that we offered is based on a low-cost spectrometer with a microreaction chamber and a tri-colour light-emitting diode as the light source, which was developed by the projects’ coordinator and the author of this paper, and can be easily upgraded into several different analytical instruments, all of which enable a sound introduction to the fundamentals of instrumental analytical methods using a hands-on approach and give schools of different types and educational levels opportunities for testing more than 60 experiments from different areas and developing their own real-life applications [3].

## 2. Development of small-scale, low-cost, analytical instruments to bridge the gap caused by a lack of professional instrumentation

### 2.1 Spectrometer for educational purposes

The starting point for the construction of a tri-colour, light-emitting-diode-based, in-situ spectrometer was the decision to use polymeric supports, called blisters—and used in the pharmaceutical industry for the packaging of pastilles—as the reaction and measuring chambers (Fig. 1 – left, bottom). A prototype of the spectrometer was constructed (Fig. 1 – left, top) and the novel optical geometry was patented.

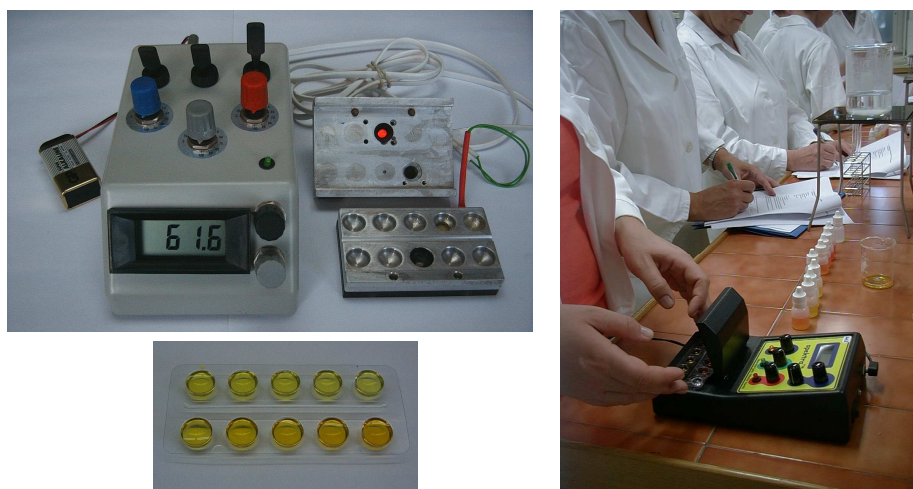


Fig. 1. Polymeric supports – blisters used as reaction and measuring chambers (left - bottom), a prototype of the spectrometer (left - top), the Spektra™ spectrometer used in a workshop (right)



The specific geometry of the spectrometer means that the light of a tri-colour, light-emitting diode (LED) with emission maximums at 470, 565 and 660 nm (blue, green and red light) passes through the solution in the vertical direction and falls directly onto a photo-resistor, which lies under the microreaction chamber [4]. A tri-colour LED functions as a light source, and the blue, green or red light can be selected. The equality of the path length of the light within the series of measurements is achieved by control over the volume of the solutions in the individual hollows of the blisters, e.g., by using micro-pipettes for measuring the sample and the reagent volumes or by using a simplified drop-based experimental approach. The bottom of the measuring chamber is designed so that it supports the blister in different positions, enabling spectrometric measurements to be made for the different hollows of the blister. The measurement results are expressed in terms of the transmittance (%). Experiments demonstrating the additive mixing of colour can also be performed with the tri-colour LED. The spectrometer is intentionally designed so that it contributes to the development of the mathematical competence and the basic competences in science and technology. The instrument is not a black box for students. They are in direct contact with what is actually being measured and receive only “raw data” from the spectrometer. The students are encouraged to apply different mathematical procedures that are appropriate for their educational level (e.g., drawing graphs, calculating with fractions or linear equations, applying logarithms, measuring the height or calculating the area of a chromatographic peak) in order to obtain the final results of the analyses.

The experiments can be performed rapidly and do not require a laboratory environment or any classical laboratory skills. This makes it very suitable also for use at the lower levels of vocational education or even for carrying out promotional activities at the pre-vocational educational level with the objective of attracting larger numbers of prospective students into vocational programs and to increase the interest in natural sciences and natural-science-based or related disciplines. With hands-on activities based on the small-scale spectrometer the students become active experimenters, observers and creators of their own knowledge, who gain an understanding that natural sciences are not boringly and exhaustively difficult, but challenging, interesting and logical. The prototype was later transformed into a more robust and even easier to handle Spektra™ spectrometer (Fig. 1 - right), which has emission maxima at 430, 565 and 625 nm.

The spectrometer can be upgraded into several other analytical instruments or connected to a personal computer. We will briefly explain here two of them – a gas and liquid chromatograph – and only name the others that we developed: a low-cost flow injection analytical system with spectrometric detection [5], a spectrometric microtitrator [6], a luminescence spectrometer and a microdiffusion-based spectrometric unit for determining volatiles [7].

Through the two EU Leonardo da Vinci projects “Hands-on approach to analytical chemistry for vocational schools” and “Hands-on approach to analytical chemistry for vocational schools II” several experiments from different chemical- and chemistry-related disciplines were developed for this equipment and published on the web pages of the projects with the acronyms AnalChemVoc and AnalChemVoc II [3].

## 2.2 Spectrometer upgraded into gas or liquid chromatograph

In professional practice the spectrometer and gas or liquid chromatographs are completely different instruments and neither of them can be put to the function of the other. A simplified gas-chromatograph for determining volatile halogenated hydrocarbons (Fig. 2 - left) can easily be assembled by using components readily available in a school laboratory.





Fig. 2. The Spektra<sup>TM</sup> spectrometer upgraded into a simplified gas chromatograph (left) and a liquid chromatograph (right).

A gas supply cylinder provides the mobile phase (propane 40% + butane 60%), a glass column is filled with a CALGON® anti-limescale powder, and a copper coil made of wire is positioned over the tip of a glass burner. The greenish-blue light is emitted from a flame (Beilstein reaction) when halogenated hydrocarbons exit the column. The photoresistor of the Spektra<sup>TM</sup> spectrometer functions as a sensor, responding to light transmitted from the Beilstein detector through the optical fibres. For the introduction of the basic chromatographic parameters using a hands-on approach the students prepare the liquid mixture of  $\text{CHCl}_3$  and  $\text{CH}_2\text{Cl}_2$  in a volume ratio of 3:2. The instrument proved that the measurements are sufficiently detailed and reliable for the basic chromatographic concepts to be introduced through a hands-on approach [8].

The liquid chromatograph (Fig. 2, right) consists of a column prepared from a 2.5-ml Pasteur pipette filled with a silica-gel suspension and covered with a layer of finely ground sand. The bulb of the Pasteur pipette is cut off so that it functions as a mobile-phase reservoir. The bottom end of the column is connected to a transparent polymeric tube, which extends to the measuring site of the Spektra<sup>TM</sup> spectrometer where it is shaped into a coil-shaped, flow-through cell. The tube leading out of the spectrometer is connected to the water-jet or peristaltic pump, which sucks the mobile phase through the chromatograph. The chromatograph was tested for the separation of the E102 and E131 dyes in a natural mint aroma, using the blue or red LED. The transmittance was recorded in 10-second intervals. The chromatograms are presented on the website [3].

### 3. Results of the two EU Leonardo da Vinci projects

#### 3.1 Hands-on Approach to analytical chemistry for vocational schools - Pilot project (2003-2005)

In this project the low-cost spectrometers were introduced into the school practice of the three participating countries – Slovenia, Portugal and Great Britain – for the first time. The tri-lingual web page “AnalChemVoc” is one of the main project results and includes descriptions of approximately 45 hands-on activities developed by the project partners and the teachers from all three countries.

Another main project result is the handbook entitled “Hands-on Approach to Analytical Chemistry - Manual” also prepared in all three languages of the project (60 CD copies in each language). The manual, which is structured into seven chapters, is intended to help teachers develop self-confidence when dealing with the selected topics of analytical chemistry and using a hands-on approach as an active teaching strategy in their classroom. The last two chapters present two teachers' guides for the



introduction of visible spectrometry and chromatography through the hands-on approach. We must stress that the approach discussed in both teachers' guides was evaluated in several steps, and after each evaluation step the materials were improved. These teachers' guides are related to the teaching units "Hands-on Approach to Visible Spectrometry" and "Hands-on Approach to Chromatography". They include full support for the teacher and the students and supplement the manual on the same CD.

The testing and evaluation of the hands-on approaches to analytical chemistry in schools confirmed that the approaches developed and implemented in the schools through this project increase the motivation of students and ensure that the less-able students are not left behind.

### **3.2 Hands-on Approach to analytical chemistry for vocational schools II - Transfer of innovation (2008-2011)**

This project was focussed on the wider implementation of the previously developed approaches to vocational schools for food processing and the transfer of this innovation into additional sectors, e.g., laboratory medicine and chemistry in Slovenia and Poland, and into the outreach activities of the British partner. The Manual "Hands-on approach to analytical chemistry" comprising two teaching units, and experiments for the web page, which were developed during the previous project, were translated into Polish. A total of 26 new hands-on experiments were developed and extended in nine new areas: Environmental analysis, Materials analysis, Multicomponent analysis, Biotechnology, Laboratory medicine, Outreach activities, University support of vocational schools, Chemiluminescence and UV spectrometry. An important achievement is that the evaluation study showed that the hands-on approach towards visible spectrometry contributes not only to the better comprehension of concepts, but also to the capability to analyse data. Teachers' responses to the hands-on approach, which were collected from a special questionnaire, stress a series of advantages of the tested approach, among them: deduction of regularities from the experimental results, a discovery-based approach, a high level of motivation from the students throughout the experimental work, a great variety of experiments, and teacher's control of the class activity by using special visual support, which enables them to give to the students immediate feedback after any phase of the experimental procedure. Teachers made a proposal to prepare additional teaching materials in the form of the hands-on approach at different levels of difficulty, so that less-able students would not be left behind [3].

## **4. Conclusions**

This paper demonstrates how the low-cost instruments that we developed for educational purposes can help bridge the gap caused by a lack of professional instrumentation and enable a sound introduction to instrumental analytical methods using a hands-on approach or the use of quantitative approaches when learning general chemistry or chemistry-related disciplines. The approaches described here can also increase the innovative potential in teachers and students by challenging the often misleading opinion that there is only one way of doing analytical chemistry, i.e., with complicated and costly professional instrumentation. The potential of all the upgradings of the equipment has not yet been exploited. A development of a wider modular didactic instrumental set with experiments for different educational levels, also comprising the tertiary level, would be possible.

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