

# *Spectroscopy instruments and the teaching of chemistry in the polytechnic school of Lisbon (1860-1960)*

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## *Abstract*

This paper aims at contributing to an improved knowledge of the History of Chemistry in Portugal, namely in what concerns the teaching of this discipline. In the framework of Spectroscopy and its implications on Chemical Analysis the study focuses on the scientific instruments used for the teaching of spectroscopy for chemistry classes at the Polytechnic School of Lisbon and later at the Science Faculty of the University of Lisbon, during the period from 1860 to about 1960. Besides printed and manuscript sources, we used less conventional sources - the scientific instruments of the collection hold by the Museum of Science of the University of Lisbon.

## *Introduction*

History of Science is, on its own right, a learning resource. The analysis of both historical cases and historical controversies allows us to understand how Science is built. In consonance with this, our purpose was to contribute to an improved knowledge of the History of Chemistry in Portugal, namely of what concerns the teaching of this discipline. We chose to study a boundary field between Chemistry and Physics: Spectroscopy and its implications on Chemical Analysis. This study focuses on the scientific instruments used for the teaching of spectroscopy for chemistry classes at the Polytechnic School of Lisbon and later at the Science Faculty of the University of Lisbon, during the period from 1860 to about 1960.

The History of Chemistry in Portugal, and its teaching, cannot be approached outside the European scientific context. That's why we endeavored to get acquainted with that context in the referred period, in what concerns the acceptance of Spectral Analysis among chemists, how it developed and which relations arose between the different characters of this same history: scientists, scientific instruments, manufacturers and teachers.

In addition to printed sources, we had the privilege of using less conventional sources: the scientific instruments of the collection hold by the Museum of Science of the University of Lisbon (MCUL), testimonies of the History of Teaching that has taken place at the Polytechnic School of Lisbon and at the Science Faculty of the University of Lisbon.


### *The first spectroscope:*

The collection of the Science Museum keeps quite the total amount of scientific instruments used in teaching classes as in the research work at the Polytechnic School of Lisbon\* and at the Science Faculty of the University of Lisbon. The study of a piece of the museum collection, a Duboscq spectroscope of the Bunsen-Kirchhoff type [Fig. 1] was the starting point for this research work.

\* 🌿 The Polytechnic School of Lisbon is a reference in this study because it was one of the few places in Portugal, where it was possible to study Sciences in 19<sup>th</sup> century, namely Chemistry in high degree studies. It was founded in 1837, to form students to the Army and Navy Schools, beyond giving scientific knowledge in high degree for other professions. It started to work with a Library, an Astronomical Observatory, a Physics Cabinet, a Chemistry Laboratory, a Natural History Cabinet, a Botanical Garden and other common facilities.



**Fig. 1** 🌿 Spectroscope of the Bunsen-Kirchhoff type from Duboscq, MCUL169  
(Photo Marília Peres, courtesy Museum of Science, University of Lisbon)

\*  Vicente Lourenço was born in India and graduated at the Surgical School of Goa, where he worked until 1848. In Paris he gave up the Medicine studies and became a chemist, studying and working in Wurtz's laboratory. Lourenço's several works were presented in Sciences' Academy. He went to Germany where he worked with Bunsen and in England he worked with Hoffman. He returned to Paris and after that to Portugal, about 1862. We don't know the precise dates on which Lourenço was in Paris or in Germany. In Lourenço's paper from 1860, *Éthers Composés du Glicol*, existent at MCUL's library, we found a dedication in the book cover: "*A M. le Professeur R. Bunsen Hommage Respectueuse de son élève M. Lourenço*". From this dedication, it is obvious that Lourenço had a strong relation with Bunsen, possibly when he was associated with Kirchhoff, when they began spectral analysis studies.

A consultation of MCUL archive allowed us to establish a relationship between the acquisition of this spectroscope and the beginning of the spectral analysis teaching at the Polytechnic School.

In the Polytechnic School the teaching matters were organized in 5 courses. Analyzing these courses, we can verify that the 6<sup>th</sup> discipline – General Chemistry and Principles of its Applications to Arts – was introduced in the second year, in most of them. Polytechnic School's disciplines were lectured by *Professores Proprietários* and *Professores Substitutos*. In 1859 another discipline was created, Chemical Analysis and Organic Chemistry. Agostinho Vicente Lourenço\* came to Portugal to lecture this discipline.

When Lourenço was nominated *Professor Substituto* in Organic Chemistry (1862), he elaborated a report on the importance of purchasing the necessary material to teach the discipline. We can find in this list a spectral analysis apparatus from Bunsen and Kirchhoff. Afterwards, the *Professor Proprietário* – Júlio Oliveira Pimentel - drew up a proposal<sup>1</sup>, where a "*Bunsen and Kirchhoff apparatus for spectral analysis*" appears. In 1872, a customs clearance was issued addressed to Agostinho Vicente Lourenço: "*Products coming from Le Havre: 3 boxes with Spectral Apparatus*"<sup>2</sup>. We think that apparatus is the one that belongs to the Science Museum of the University of Lisbon (MCUL) spoil.

Analyzing these disciplines curricula, we can verify that the Spectral Analysis training appeared in Organic Chemistry in the school year of 1864-1865<sup>3</sup>. The Qualitative Analysis sub-chapter of the referred discipline included Spectral Analysis. And in the school-year 1872-1873<sup>4</sup>, in the same discipline curricula, Spectral Analysis appears in parallel with other analysis.

### *A preparation set for spectral analysis*

The museum (MCUL) collection also has a set of preparations for spectral analysis<sup>5</sup> [Fig. 2], with about the same age of the spectrometer, but from another maker. It was bought from the instrument maker Gerhardt<sup>6</sup> some years later, in 1885.



**Fig. 2** ♣ Set of preparations for spectral analysis, MCUL156  
(Photo Marília Peres, courtesy Museum of Science, University of Lisbon)

### *The Duboscq's projection set*

We found at MCUL's spoil several instruments that, with no doubt, have been used for this type of projection in Chemistry lessons at the Polytechnic School: two projection lanterns with several accessories (Foucault photoelectric regulator, with respective carbon electrodes and spectroscopy's diaphragm with rectilinear opening), Duboscq's lens and Duboscq's prism [Fig. 3]. Those projection lanterns were especially commercialized by the maker Duboscq, and they were already in the Duboscq's catalogue of 1870<sup>7</sup>, dedi-



**Fig. 3** ♣ Duboscq's Projection Lantern, Lens and Prism, MCUL2004, MCUL2003; MCUL310)

(Photos Marília Peres, courtesy Museum of Science, University of Lisbon)



cated to spectroscopy. The maker referred classic experiences of spectroscopy, namely the sodium line D experience; he suggested the settings, which we can see in Fig. 4.

We can find in the text book of one important Chemistry Professor at Polytechnic School and Sciences Faculty, Achilles Machado<sup>8</sup>, the complete description of that experience [Fig. 4]<sup>\*</sup>. He reported the use of the voltaic arc to obtain a continuous spectrum and a discontinuous spectrum of lines.

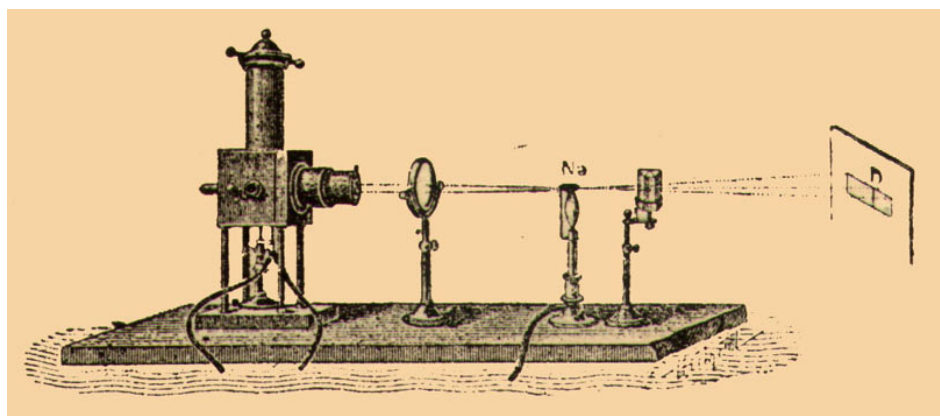


Fig. 4 \* Sodium spectrum projection

In 1892, Achilles Machado and Virgílio Machado - Polytechnic School's Professors –published “Chimica Geral e Analyse Chimica: Metalloides”<sup>9</sup>, where several spectral analysis references appear, namely the hydrogen spectrum, referring the analysis of the spectra table as an advisable matter of study. The 6<sup>th</sup> discipline curricula in 1898<sup>10</sup>, authored by Achilles Machado, indicated the study of metals and metalloids, their characterization through the colour acquired from flame essay.

Text books weren't the exclusive tool to teach spectroscopy, direct observation was fundamental for the understanding of the concepts. Already in 1874, Wurtz advised the use of projection lanterns to visualize the spectra<sup>11</sup>. One of the text books, followed by professors of the Polytechnic School, was Fremy's Encyclopaedia. The “Atlas” tome was dedicated to chemistry laboratories, which contained photos of European laboratories and of laboratory equipments. In the supplement entitled *Laboratoires* appears, on “Planche XVIII”, a representation of the “Laboratoire de Chimie de L'Université de Gratz” and of the “Grand Amphithéâtre”<sup>12</sup>. This last one referred the projection apparatus

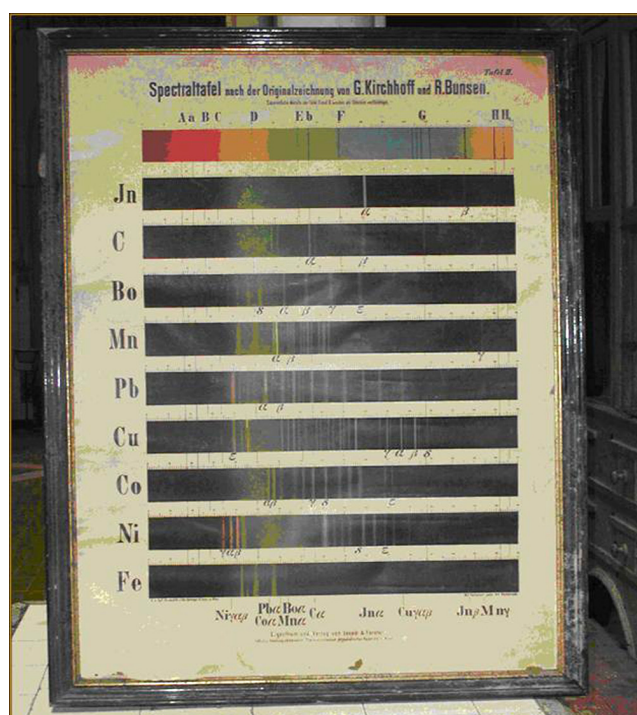
\* \* This image is similar to the one referred in Duboscq's catalogue.

and the use of blinds to darken the large room. In the picture we can observe a projection lantern and two lens or prisms. This suggests the carrying out of simple activities of spectral analysis, in theoretical and demonstrative lessons.

### *The spectral tables*

The demonstration activity, with projection lanterns, is only complete if the pupils had the opportunity to compare acquired spectra with spectra already traced. Duboscq, in 1870, offered for sale, spectra tables to Physics cabinets as well as for Chemistry laboratories. Those tables contained spectra of alkaline metals and alkaline-earth metals and spectra of nebulae. They were built with the supervision of Bunsen and Kirchhoff.

We can say that Professors of the Polytechnic School used a similar methodology, once we found two sets of spectra tables. One of them has big dimensions [Fig. 5] and so it would perhaps be located in the amphitheatre annexed to the laboratory, where theoretical lessons were taught and spectra were possibly projected. According to Hentschel<sup>13</sup>, Bunsen and Kirchhoff's



**Fig. 5** ✨ Spectral table; MCUL2017

(Photo Carmo Elvas, courtesy Museum of Science, University of Lisbon)

colour maps, which were found in most laboratories, were of great importance for the students training concerning the observation and identification of spectral lines. They were also useful as guides for the students' representation of observed spectra. Drawing and observing spectra would be essential for students training and it was an essential ability for a chemist.

The charts represent the solar spectrum and the wavelengths of the respective spectral lines of emission of the different metals. On top of the chart we can read the following title '*Spektraltafel nach der Originalzeichnung von G. Kirchhoff und R. Bunsen*'. They are contemporary of Vicente Lourenço's time at the Polytechnic School, the very period when the 'Laboratorio Chimico' was renewed and modernized. It was then that Spectral Analysis was started to be taught as a major method in Mineral Chemistry.

We must emphasize that the books advised by the professors in the last fifteen years of the nineteenth century (that is the Encyclopaedia by Wurtz<sup>14</sup> and Fresenius' 'Traité'<sup>15</sup>), presented already an extensive treatment of this subject, besides presenting spectral maps and tables with wavelengths, which were introduced only a decade after the Bunsen and Kirchhoff publication.

### *The Adam Hilger constant deviation spectrometer*

The strong evolution of spectral analysis in the chemistry science occurs in the beginning of 20<sup>th</sup> century due to the appearance of the so called Constant Deviation Spectrometer from Adam Hilger maker. Fig. 6 presents the one belonging to MCUL's collection.

Frank Twyman<sup>\*</sup>, of Adam Hilger was a specialist in the construction of scientific instruments for chemical analysis. Once he understood the difficulty that chemists found in the utilization of spectrometers, he adapted this equipment to the chemists' needs and conceptions.

"Thus although spectroscopy developed, it was under the auspices of the physicist and the astronomer. For these it rapidly became an instrument of supreme importance, but in the academic laboratories it was chiefly used, if at all, as a means of teaching optics, and instruments were deliberately made with as many adjustments as possible in order that the students might learn the principles of the instrument rather than its use as an implement of research (analysis). Thus, the busy chemist who bought a spectrometer was chiefly solicitous that it could be put away in a case and that the case should be as small as possible."<sup>16</sup>

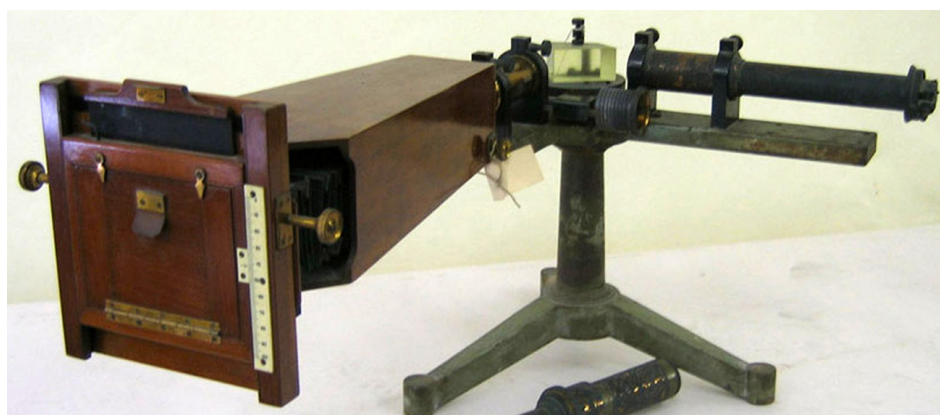
In this way he started to eliminate, as far as possible, the adjustments and the components that were exclusively for physics; so he eliminated the divided circle and got back the proposal of Péllin maker and Broca physicist, made

\* 🍀 Frank Twyman (1876-1959), an engineer trained in London, began in Adam Hilger in 1898 testing optical systems, for spectrometers. From 1902 until he retired, he became one of the officials of the firm. in C. Bigg, *Behind the Lines: Spectroscopic Enterprises in Early Twentieth Century Europe*. Department of History and Philosophy of Science, Cambridge, Cambridge University (Doctor of Philosophy), 2002.

in 1899<sup>17</sup>, to use a prism assembled in a *constant deviation* mode and with a prism cut type that allowed a complete coverage of all wave lengths just by rotation of the prism, conserving fixed the telescopes in a right angle. Another modification was also made in order to make easier the measuring of spectral lines value, using a scaled micrometric screw coupled with the prism and so moving with it.

Twyman in 1903 introduce in this instrument the *wave length drum* in the place of the Péllin micrometric screw. This drum has a great advantage, especially for chemistry and Chemical Analysis once it allowed the direct reading of the wave length values without previous scale calibration, as before. This equipment turns the Spectroscopy much more attractive for Chemistry. At that time, the Sciences Faculty of the University of Lisbon was officially founded (in 19<sup>th</sup> April 1911) the curricula for Sciences Faculties of Lisbon and Porto were established. The organization of three sections: Mathematics, Physics-Chemistry and Historical-Naturals<sup>18</sup> was foreseen. In the second section, Physics-Chemistry, the first group corresponded to the Physics' subjects and the second group to the Chemistry's subjects. The studies plan of the Sciences Faculty (SF) in the academic year of 1911/12 was implemented with the teaching staff of the Polytechnic School<sup>19</sup>.

On 30<sup>th</sup> June 1912, the general office of Sciences Faculty paid several amounts, corresponding to expenses of the group of Chemistry, to several makers of scientific instruments. We can find in this list the name Adam Hilger Ltd. We think that this payment could correspond to the Constant Deviation Spectrometer, with accessories [Fig. 6].



**Fig. 6** Adam Hilger Constant Deviation Spectrometer (adapted to spectrograph)  
MCUL897; MCUL168  
(Photo Marília Peres, courtesy Museum of Science, University of Lisbon)



Analyzing these new curricula, we saw the change from two disciplines to six. In the academic year of 1911/12<sup>20</sup>, at the General Course of Chemistry and at the Course of Inorganic Chemistry, the physical and chemical characteristics of several metals were studied; although it wasn't explicit in the program, it seems obvious to us that spectra of metals were referred. In several courses of Chemistry, "*Spectral Analysis*" was studied and the "*Observation of Emission and Absorption Spectra*" and the "*Construction of Wave Lengths Curve, relatively to Spectroscope Scale*" were carried out as a practical work.

Professor Achilles Machado published in 1917 a manual to support the lecturing of Chemical Analysis. This book presented an entire chapter dedicated to spectral analysis. It explained the several procedures of getting a spectrum, from flame to voltaic arc.

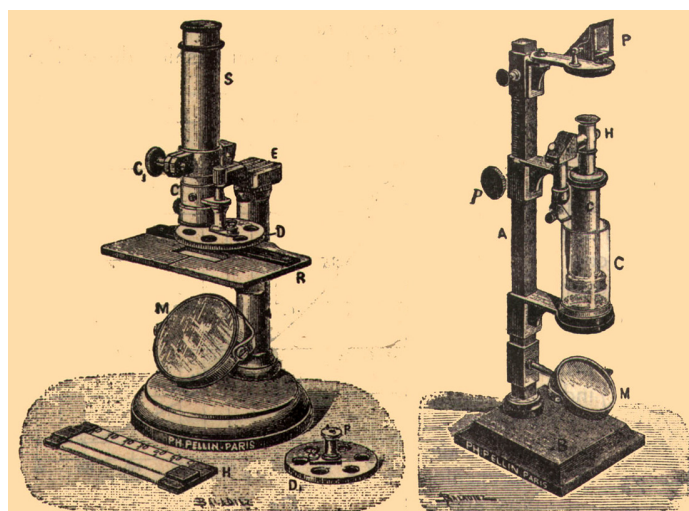
In his book, Machado introduced the Quantitative Chemical Analysis through the explanation of the importance of the last lines in the spectrum of elements. He explained the method to work with the constant deviation spectrometer, acquired to Adam Hilger in 1912, namely the ease introduced by the graduated drum, with helicoidal groove. He mentioned the apparatus adaptation to a spectrograph, to photograph the spectrum [Fig. 6]. Later, with a comparator or a microscope with micrometric ocular, the lines position was determined on the photograph (this instrument from Duboscq maker also belongs to the MCUL spoil).

### *Hemaspectroscope and urospectroscope absorption spectroscopy*

In the early 20th century the professors of Faculty of Science of Lisbon University together with teaching activities did research work, namely related with external services. Several analysis such as those of water and oils or toxicological analysis required absorption spectroscopy using more specific instruments. Some examples are the hemaspectroscope and the urospectroscope [Fig. 7]. To obtain absorption spectra, the substance to study could be introduced in a special vessel adaptable to the spectrometer. It was exemplified with the absorption spectrum of oxygenate hemoglobin.

In school years 1915/16 and 1916/17 we found references similar to those referred for the year 1912/13, although in the course of Qualitative Chemical Analysis for the same years we found references to applications of spectral analysis: "*VIII – Spectroscopy. Microscopy: The spectroscope employment in analysis. Emission and Absorption Spectra. IX – Applications: Qualitative Analysis of a drinking-water. Qualitative Analysis of a mineral water*". Also in the course of Chemistry, preparatory to the Medical School, spectral analysis was taught with emphasis regarding the applications to Medicine. Subjects

of practical works in this course were: “8th – *Flame Coloration (compounds of potassium, sodium, lithium, strontium, calcium, barium and copper)*; (...); 22th – *Characterization of the metal of a salt with spectroscope; observation of emission (flame and electric) and absorption spectra*; (...), 34th – *Spectroscope examination of the oxy-hemoglobin and reduced hemoglobin.*”.



**Fig. 7** ♣ Hemaspectroscope (left) and Urospectroscope (right)<sup>21</sup>

*Pulfrich and Löwe  
spectrograph: research  
in spectroscopy by  
Pereira Forjaz*

On December 1918, the Chemistry staff was reduced to Achilles Machado, who made an effort to open a second post for a professor. António Pereira Forjaz Pimentel was selected as Chemistry Professor. In 1916 Forjaz obtained the degree of Doctor of Physics-Chemistry\*, with a thesis entitled “*Spectral Analysis Study of Uranium and Zirconium Portuguese Minerals*”<sup>22</sup>, which experimental part was carried out at the Physics Laboratory. Before, in 1915, Forjaz had published a study with spectral analysis: “*Sky Petrography – Spectrograph Contribution to Portuguese Meteorites Study*”. In the same way of A. Gramont – who, in 1895, applied his analysis method to a Cañon Diablo meteorite by request of crystallographer Friedel – Forjaz decided in this study to apply the same method to a meteoric iron fragment existent at the Sciences Faculty, having determined its qualitative composition (O; S; Fe, P, Ni, Co, Mg, Al, Mn, Ca, Sr, K e Cs).

Following the Gramont<sup>23</sup> and G. Urbain works, which carried out spectrographic analysis of blends in 1909, Forjaz set himself to apply the same method to the study of Portuguese mineral of uranium and zirconium.

\* ♣ This was the first  
Doctoral thesis at the  
Sciences Faculty of Lisbon.



**Fig. 8** 📷 Pulfrich and Löwe spectrograph, MCUL4446  
(Photo Marília Peres, courtesy Museum of Science, University of Lisbon)

In his work Forjaz used a Pulfrich and Löwe spectrograph from Carl Zeiss [Fig. 8]. With this equipment he could do the direct observation of spectra and photograph them. In this work, he decided to use the photographic method as an improvement, and he used a known spectrum for reference, as the iron or the solar spectrum.

To do the measures with more accuracy, Forjaz used, as comparison spectrum, that of Eder's Alloy, made up of lead, cadmium and zinc. This spectrum was used by Eder in 1898, and he and Valenta applied it in visible and UV zones. To measure spectrograms, Forjaz used a Zeiss microscope (instrument belonging to MCUL collection), and the meaning of the last lines had greater importance for him. The knowledge and importance of *the latest lines* led to a great development of the quantitative spectral chemical analysis.

Since the calculus of wavelength in the prism spectrometer was more unreliable than in the grating spectrometer, Forjaz used the interpolation formula proposed by Hartmann. Forjaz conducted a generic study about uranium minerals, organizing a comparison table and presenting his conclusions related to the presence of analyzed elements. In the end, besides the conclusions of mineralogical point of view, he also presented some conclusions of physical order:

✿ For the visible spectrum study of the substances the spark should be used; but for the study of ultraviolet spectrum, the use of the arc was better.

✿ If substances weren't good electric conductors and the use of spark was needed, they should be dissolved in melt salts, like lithium carbonate. In this case, it was useful to measure previously the spectra of the solvent substance of the current study, and eliminate them in the next measures.

✿ He advised the use of sensitized plates for Spectrography.

✿ It was proper to make use of a reference spectrum, like that of the Eder Alloy, which was necessarily reproduced before use.

✿ Hartmann's formula was convenient as an interpolation formula, and its use simplified the calculus significantly.

This research work marked the subsequent teaching and research made at the Sciences Faculty. Forjaz continued the research in spectral analysis. About 1930 he made two communications related to analysis of waters: "*Hydrologie Spectrochimie des Eaux Minérales Portugaises: L'Eau des Cambres*"<sup>24</sup> and "*Recherches Spectrographiques sur les Eaux Minérales Portugaises: Le Germanium Indicateur des Eaux Profondes*"<sup>25</sup>.

He also carried out the spectral analysis of Portuguese salt. In his paper, he presented results of the analysis of four salts samples: of Alcácer, Aveiro, Setúbal and Barreiro. He concluded his paper in a very eloquent way: «O mer, donne-moi un petit grain de ton sel et il me livrera tes secrets.»<sup>26</sup>

## Spectrophotometer

During the period of 1940 -1950 a more precise type of spectroscopes appeared, with more sensibility, being the radiation detection done with a phototube; The first spectrophotometer of this kind was the Beckman DU and latter on the Spectronic 20 [Fig. 9].

The Beckman DU was developed in 1941 by *National Technical Laboratories*, maker, later *Beckman Instruments Co.* This instrument has been constructed and commercialized until 1975 and more than 35000 units have been sold. The Beckman DU was a spectrophotometer with single beam and quartz prism; the radiation detection was done first with a phototube and later with a photomultiplier. Indeed it has been very important for the progress of chemical research.

A new spectrophotometer appeared in 1954, Spectronic 20, from Bausch and Lomb maker; this model was more adapted for industrial and medical





**Fig. 9** \* Spectrophotometer Spectronic 20, MCUL969 (Photo Marília Peres, courtesy Museum of Science, University of Lisbon)

applications. It worked from 340 to 950 nm and it had a very rapid work heating time and gave results with very good accuracy<sup>27</sup>. This model had a double function; it could work as colorimeter or as a spectrophotometer and it was possible to get easily any supplies as graphic register or tables for clinical analysis. Spectronic 20 was present in all the laboratories of that time.

During the decades 50 and 60 at the Faculty of Sciences of Lisbon University the text books by Vogel<sup>28</sup> and by H. Willard, L. Merritt and J. Dean<sup>29</sup> were the ones used by the students for the chemical analysis courses. In the experimental classes students covered the use of the colorimetric techniques (using a Duboscq instrument), spectrophotometry (using Universal Coleman, Beckman DU and Spectronic 20 equipments) and also flame photometry. At the same time, some of the applied research work undertaken has been based on spectrophotometric techniques like “*Ensaio para a Determinação da Vitamina K<sub>2</sub> na Sardinha Portuguesa*” by Forjaz, Brito e Manso<sup>30</sup> or a “*Spectrophotometric study of Iron III system*” by A. Pimenta e M. M. Silva Carmo<sup>31</sup>.

Branca Marques e Maria de Lourdes Simões have done the work “*Estudos Espectrofotométricos sobre a Identificação de Clorocomplexos de Ruténio III*”<sup>32</sup>, at the Radiochemistry research laboratory of Lisbon University, using a Beckman DU spectrophotometer coupled with a photomultiplier.

The objective of this study was to cover just the period of 1860-1960 but we can finish saying that the development of the optical methods in chemistry teaching and research was carried out until now.

So in 1987<sup>33</sup> the Faculty of Sciences has already research projects and development works using X-ray spectrometry, Mössbauer Spectroscopy, Mass Spectroscopy Raman, IV, Visible and Ultraviolet, etc. .

\* \* The following are examples of research work:

¶ M. L. DUARTE, 1981. *O Efeito Raman de Ressonância e suas Aplicações*, Lisbon, Universidade de Lisboa, 1981.

¶ A. M. ELIAS, *Espectroscopia Laser em Materiais Vitrificáveis*, Lisbon, Universidade de Lisboa, 1983.

¶ S. M. SANTOS, *Identificação de Compostos Orgânicos por Via Química e Espectroscopia*, Lisbon, Universidade de Lisboa, 1984, p. 66.

¶ F. COSTA, M. R. GRADE, M., B. E. MARQUES, “Limite de Detecção do Cálcio por Fotometria de Flama”, *3<sup>rd</sup> International Congress of Atómica Absortion and Atómica Fluorescence Spectrometry*, Paris, Londres, Adam Hilger, 1971, p. 125- 137.

We can conclude that the teaching and related research of spectral analysis at the Polytechnic School and Sciences Faculty, concerning the studied period, was at the level of the other European schools.

With this work<sup>34</sup>, we believe having contributed to an expanded knowledge of the History of Chemistry in Portugal, namely of its Teaching. Further, we have provided the identification of many optical apparatus kept at this Museum and formerly unclassified, promoting not only its historical and museological knowledge, but also its integration in forthcoming exhibitions or publications.

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