

THE HISTORY OF CHEMICAL LABORATORY EQUIPMENT*

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Summary

Origin of the chemical laboratory. The historical scenes of chemical research work. The way of chemical analysis from qualitative detections till the beginning of quantitative determinations. The first analytical devices for quantitative purposes. The penetration of optics and electricity in the chemical laboratory work in the XIX. century. Optical and electrical analytical instruments. The instrumental development at the beginning of our century. The actual change of the usual view of an analytical chemical laboratory, domination of electronics.

It is a great honour for me to talk about the history of instruments in chemistry and above all in analytical chemistry in this circle of instrument specialists. I do have some problems, though. You may perhaps find it strange, but my difficulties start actually with the instrument concept in itself. The problem arose some years ago, when the Hungarian Museum of Technology whose head I am planned an exhibition dealing with the history of instruments, entitled "Man and his Instruments". The question of delimiting the subject arose in the course of planning, and with it the problem of defining what should be considered an instrument. All of us thought—and presumably all present here will also think—that this is quite clear and obvious. However, it frequently occurs with the simplest things that the answer is neither clear nor obvious. We thumbed many books to find an unequivocal definition, but found none of them satisfactory. We began thinking quite a lot about the problem, and once, returning from the countryside to Budapest, when we again talked about it, we thought we had found a satisfactory and exact definition: an instrument is an object that quantifies some phenomenon of nature for man, that is, an object making the phenomenon quantitatively perceptible or even measurable. When, however, we discussed this definition in the wider circle of the Museum's

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scientific workers, somebody put up the question: What about the telescope? To be sure, he had got us there. The telescope doesn't make anything measurable. Finally we gave up to look for a definition. We decided voluntarily what to exhibit and to put on show every interesting object in our possession and acceptable in this context.

During that discussion somebody else said that one might just omit the word "quantification" and simply state that an instrument is an object that helps to recognize the phenomena of nature beyond our senses. However, this definition doesn't fit, if applied to chemical laboratories, since it is evident that laboratory equipment known for many centuries such as flasks and distillation apparatus have been of immense assistance in getting better knowledge of nature, but nobody would consider them instruments. So this is the reason why I chose the title History of Chemical Laboratory Equipment for this paper: it includes both laboratory appliances and instruments.

Chemistry as a craft looks back on a past of many thousand years, to the beginnings of history in trades like textile dyeing, hide tanning, preparation of ethereal oils and other cosmetics, soap-boiling, beer-making and many others. Chemistry as a science, however, is only some centuries old, but it has a thousand year old precursor: alchemy, which was a deliberate human endeavour to achieve a definite practical aim scientifically based on Aristotelian notions concerning matter. Alchemy is of particular interest in the context of our subject, because the chemical laboratory, a true workshop of experimentation was called into being by the alchemists. They introduced experimenting into research much before the theoretical argumentations of Leonardo and Bacon.

It is beyond doubt that chemistry developed from alchemy, even though there were times, particularly the past century, when chemists were rather bashful about acknowledging it, since the endeavour to transform one element into another, the aim for which alchemists assiduously worked for centuries and centuries appeared tremendously nonsensical. Anyhow, even if they were ashamed of their alchemist ancestors, they could not deny them. This dual attitude is appositely expressed in the first book written on chemistry in Hungarian, in 1807: "Alchemy is in no relation to chemistry except that their names bear a resemblance. It is true though that alchemy is the mother of chemistry; but what can a daughter do if her mother is a fool!" By now, it became clear that alchemy was not as foolish as all that, since the transformation of elements became a reality. It turned out that alchemistic nonsense contained a nucleus of truth, at least theoretically.

As to the resemblance of names: the original word wasn't alchemia, but khemia during the Hellenistic period, when the aspiration to make gold was born from the strange, but rather permanent union of Aristotelean philosophy and extensive practical knowledge in Egypt conquered by the Greeks. The

word is supposed to stem from the ancient Coptic word Kemi for Egypt, though this etymology is controversial and other explanations also exist. Anyhow, when the Arabs conquered the region, they simply set their definite article “al” before the word, and gold-making became known all over Europe as alchemy. In the 16th–17th century, the scientists who started experimenting in laboratories not with the aim of making gold, but to gather knowledge on the phenomena of nature dropped the prefix “al” in order to draw a distinction between themselves and alchemists who by that time had turned mainly into charlatans. They probably had no idea that by this they actually returned to the original old name.

The mysterious look of alchemist laboratories was a remainder of Oriental mythical traditions, but their actual equipment was largely of practical origin, taken from artisans who melted metals, distilled ethereal oils, filtered dyestuff solutions and evaporated salt solutions. The alchemists used similar operations in their experiments, so they appropriately reduced the size of artisan equipment to suit their purpose; they designed small furnaces and vessels, crucibles, flasks, retorts and distilling devices. Many drawings are found in the few existing Greek alchemist manuscripts. However, only the alleged inventor of the water bath serving for slow evaporation is known by name: Maria the Jewess. Nothing else is known about her; however, the French

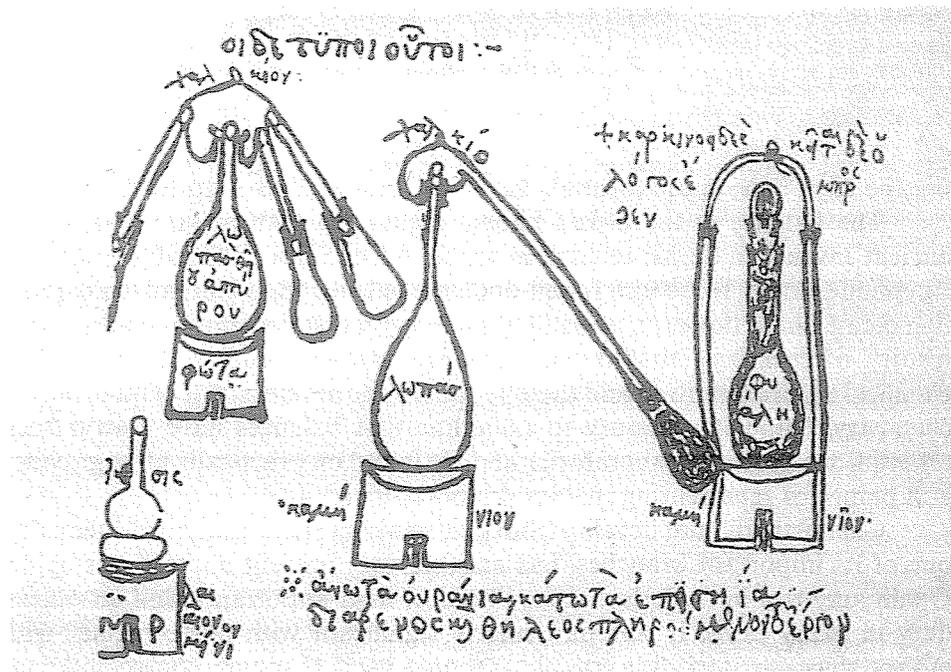


Fig. 1. Ancient Greek distillation devices



Fig. 2. Arabic laboratory devices

use the term bain-marie (Maria's bath) for the water bath up to the present.

The balance is the oldest known measuring instrument. It is a very ancient invention, ample references are found in Sumerian, Babylonian and Egyptian sources. Its history is well-documented and I don't intend to deal with it in detail. Since, however, one still frequently finds statements in books on the history of chemistry that it was Lavoisier who introduced balances into chemical experimentation and thereby started the development of quantitative chemistry, I find it necessary to underline that balances have always been essential accessories of laboratories and are found on practically all engravings and paintings representing medieval laboratories.

Chemistry did not develop from gold-making only. Analytical chemistry, one of its important branches, has another very ancient ancestor: assaying. Quality control in trade has always been of high importance and an official duty in many cases. It was particularly significant with noble metals, with money, since counterfeiting is as old as money itself. However, control methods are equally old. I shan't go into details on the fire assay or cupellation test



Fig. 3. Laboratory from the XVth century with balance



Fig. 4. Gold assaying laboratory from the XVIth century

described in antiquity and still existing up to the present in an essentially unchanged form. Being a quantitative method, balances were needed to weight the gold or silver lump obtained in the assay. A law dealing with money assaying: *ars denarios probandi* was mentioned by Plinius. The money-assayers were called *artifices*. Archimedes' hydrostatic gold control methods also required a balance. The fire assay test is mentioned in a decree issued by the Hungarian king Charles I in 1342 and described in great detail in an order of the French king Philip VI. Here one can read the followings on balances: "The balance used for the test should be of good construction, accurate and should not pull to either side. The test should be carried out in a place where there is neither wind nor cold, and whoever carries out the test must take care not to burden the balance by breathing upon it." This demonstrates that the balances employed at the time must have been fairly sensitive if breathing could have any effect on them.

Demands to the sensitivity of balances became increasingly severe with the development of analytical chemistry. The balance described in Pfaff's "Handbuch der analytischen Chemie"—published in 1821—was capable of indicating 1 mg at a load of 10 g; the smallest weight Berzelius used was 5 mg.

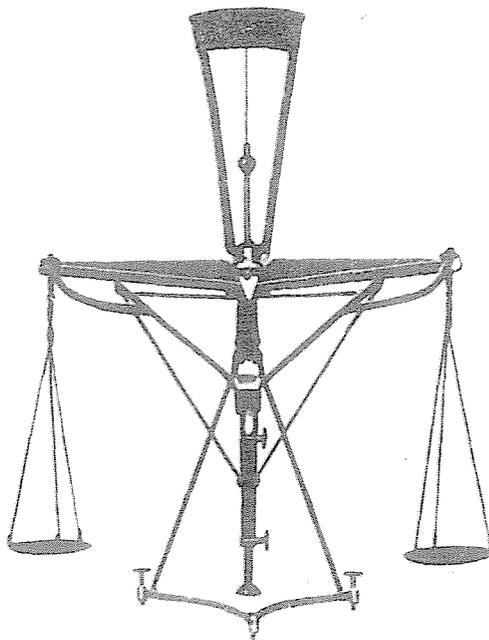


Fig. 5. Balance of Berzelius

Modern microanalysis became possible only with the construction of microbalances. The microbalance designed by the Nobel laureate Pregl in 1913 has a sensitivity of one thousandth milligram, allowing the analysis of one milligram samples and opening up thereby new potentials in biological research.

Next to the balance the oldest quantitative measuring instrument in chemistry was the areometer. It was described for the first time by the Greek alchemist Synesios who lived in the 4th century: "This is a cylindric tube on which horizontal lines are marked to measure the depth to which it submerges into the liquid. To keep it in a vertical position in the liquid, a small weight is attached to its lower end." A manuscript from the 6th century entitled *De ponderibus et mesuris* also describes a similar instrument termed hydroscopium. Subsequently the areometer fell into oblivion and it was Boyle who rediscovered its use. Though noted Arab scientists such as Al Biruni and Al Chazini did determine the density of numerous liquids, they used balances for the purpose.

For several centuries, no change occurred in the equipment of the chemical laboratory. In the 18th century, however, a simple but ingenious little appliance, the blow-pipe made its appearance and became highly significant in the progress of chemistry. Some chemists may still be alive who have met it, but I suppose that the vast majority has never seen a blow-pipe. It came to the chemical laboratory from the glass and goldsmith trade. Johann Kunckel was the first to describe it in his book entitled "*Ars vitraria experimentalis oder vollkommene Glasmacherkunst*" published in 1679, and also to point out its applicability for analytical purposes. He describes the blow-pipe as based on blowing air into a flame, and the sharp and "concentrated" flame, as he terms it, is then blown by means of a small metal tube onto the surface of the glass to be processed; beautiful glass objects can be created in this manner. Kunckel then continues: "Many possibilities are concealed in this art; in a chemist's workshop, for example, it might prove very useful. One use would be to melt a metal lime in order to see what metal it contains. This can be done most conveniently by placing the lime to be analyzed into a hollow of a small piece of charcoal and blowing the flame onto it for a very short time."

In the 18th century true artists of the blow-pipe existed. The method was improved by the use of various additives, and certain reactions could be made specific. Many books were written in that century concerning the subject, the most important being Torbern Bergman's comprehensive work entitled "*De tubo feruminatorio*." (Bergman was professor at the Uppsala University and his book appeared in 1779.) Berzelius also wrote a book on the blow-pipe in 1820 under the title "*Afhandling om blasrörets användande i kemien och mineralogien*" where he writes of a contemporary, Gahn, who accomplished such skill in this technique that he was able to detect copper in the ashes of a

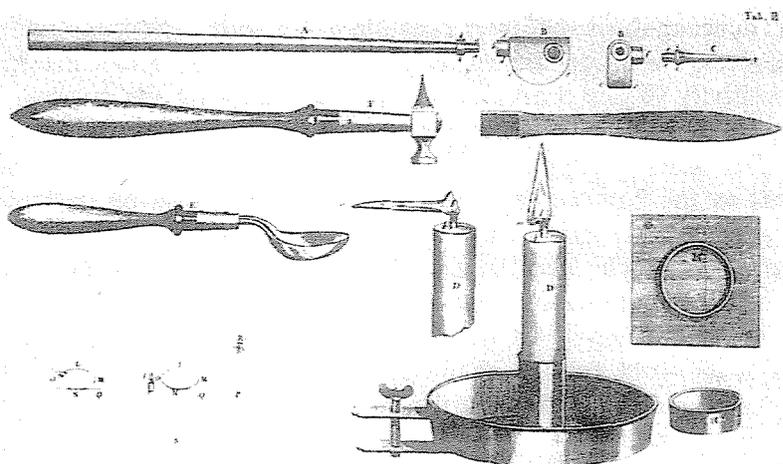


Fig. 6. Bergman's blow-pipe equipment

quarter of a sheet of paper, an interesting finding, since nobody had thought earlier that vegetable matter might contain copper. In the 18th and 19th century, the following elements were discovered by means of the blow-pipe: cobalt, nickel, manganese, tungstene, tantalum, and in part cadmium and tellurium. In the first decades of the 19th century attempts were made by Harkort, Plattner and others to develop a quantitative method based on the size of the metal clump obtained. However, these endeavours were not permanent and had no success, since in the mean time the most widespread and most successful method of analytical chemistry: volumetric or titrimetric analysis had emerged.

Considering the variously instrumented and automated forms of modern volumetric analysis, it appears incredible how primitive its beginnings were. The method came from industry, not from science, and scientists didn't take any notice of it for a long time, they didn't consider it a method suited for scientific investigations. And they were right indeed: as long as the concepts of atomic masses and equivalent masses were not established, moreover, as long as these values were not determined with satisfactory accuracy—and that occurred very much later only—volumetric analysis was only suited for certain industrial tasks. At this point, I wish to underline that volumetric analysis and many other analytical techniques, including the most up-to-date electronic and computerized procedures, are all of them relative methods, based on comparison with reference standards, and these standards are always obtained by mass weighing with balances. The chemist who applies modern analytical techniques may never use a balance. He should remember, however, that his reference standards have sometime, somewhere been determined by means of a

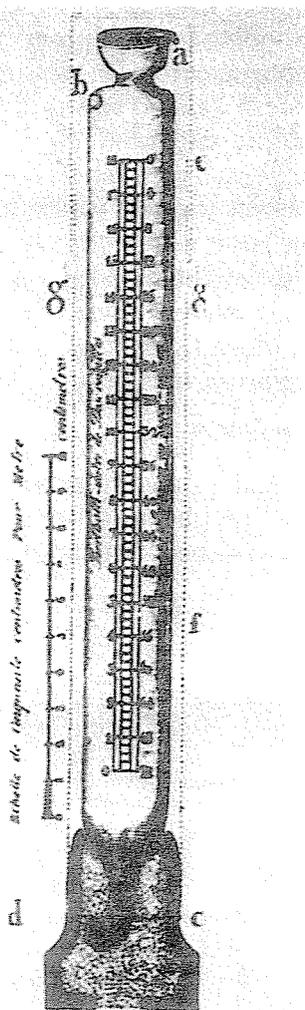


Fig. 7. Burette of Descroizilles

balance. Up to the present day no one has succeeded to eliminate balances from chemical science.

Titrimetry started in the 18th century with the most primitive equipment imaginable: Francis Home, in 1756, just needed a teaspoon. At that time bleaching of cotton fabrics was one of the important problems in rapidly progressing mechanized textile industry. The traditional method of spraying with curds and spreading the fabric in the sun was all right with the hand looms, but no more feasible with the large amounts produced on mechanical looms: such quantities of curd and lawn to spread the fabric on were nowhere at disposal. The problem was solved by a chemical process: bleaching was

performed by alternate dipping into solutions of potash and sulfuric acid. It was, however, of great importance to have some method to control sulfuric acid concentration, for if the acid was too strong, it destroyed the fabric together with the impurities. Let me cite what Home wrote in his book entitled "Experiments of Bleaching" on his invention how to control acid strength: "In order to discover what effects the acid would have on these ashes and what quantity of the former the latter would destroy, from which I might be able to form some judgement of the quantity and strength of the salt they contained, I took a drachm of blue pear ashes and poured on it a mixture of one part spirit of nitre and six parts water, which I shall always afterwards use and call the acid mixture. An effervescence arose, and before it was finished, 12 spoons of the mixture were required."

This description manifests that Home disposed of all essential elements of titrimetric equipment: he had a standard solution, a device for measuring the volume of solution used up (the teaspoon), and a means for end-point indication (ceasing of effervescence). William Lewis, in 1767, already used an

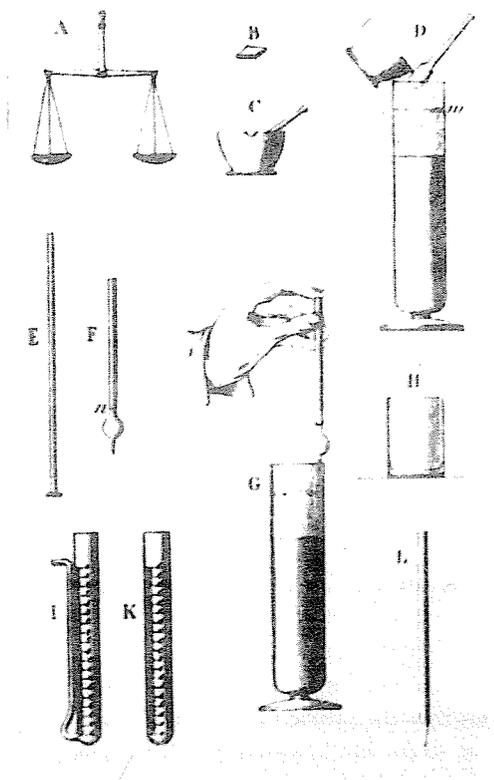


Fig. 8. Gay-Lussac's titrimetric equipment

indicator, namely litmus, for end-point indication. The equipment which is still in use in titrimetry was developed by the French chemist Descroizilles, and it was he who gave the names pipette and burette to the devices. At his time textile bleaching was already carried out with sodium hypochlorite, a chemical that destroys textiles if used in excess. Descroizilles prepared a 1% indigo solution and added it dropwise from the top of a graduated tube to one pipetteful of the hypochlorite solution. The colour of the indigo solution was destroyed by the hypochlorite until the latter was used up; with the next drop, the solution turned blue. The volume of indigo solution that had to be added to obtain a permanent blue colour with a hypochlorite solution appropriate for bleaching was determined experimentally; the factory tests, if their result did not correspond to this predetermined value, indicated that the hypochlorite solution must be diluted or concentrated correspondingly. This procedure was, of course, fully empirical and unsuited for quantitative determinations. Descroizilles later modified the shape of his burette, and Gay-Lussac gave it the form that remained in use for a long time. Let me point out that no suitable cocks were yet at disposal, so that various devices were used to ensure dropwise addition of the standard solution. The first burette with a cock (which, however, was made of metal) was developed by Etienne Henry in 1846. Burettes with glass cocks were introduced 10 years later only, by Friedrich Mohr.

Burettes are still the most important tools of titrimetry and all instrumental modifications essentially consist in electrochemical or electronic end-point indication, in the automated termination of the operation and in recording the results.

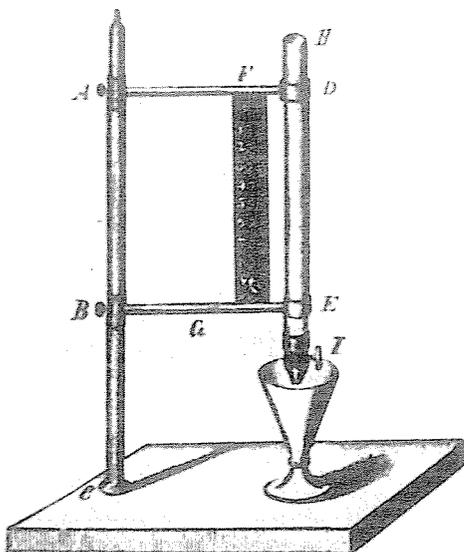


Fig. 9. Henry's first burette with cock

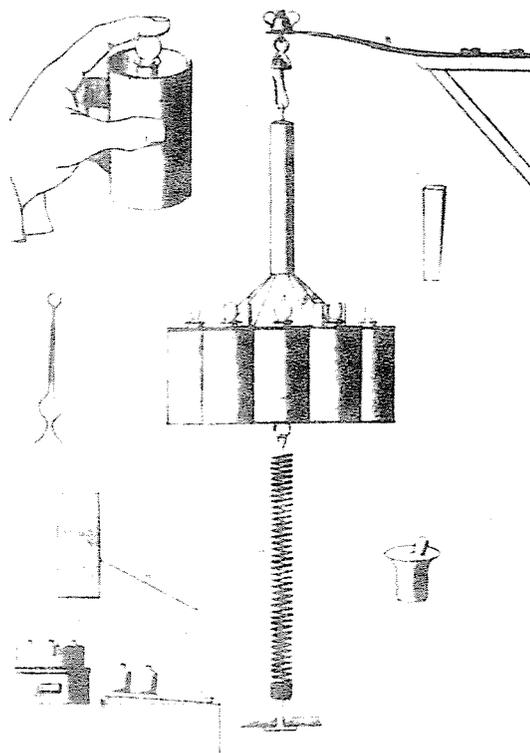


Fig. 10. Gay-Lussac's shaker

One of Gay-Lussac's methods required intense shaking, as occurs frequently in chemical processes. Today this is no problem with mechanical shakers. Gay-Lussac had no shaking machine at disposal, but found a way how to help himself, as shown in the next slide representing his ingenious shaking device constructed in 1832. There is no need for an explanation, I suppose! But no all proposals for new analytical devices proved so good, so Gay-Lussac's complicated equipment for pipette filling or Berzelius' precipitate filtering device have been forgotten quickly. Till now it is more simple to do this with mouth resp. hands.

With titrimetry, laboratory progress based on chemistry came to an end; all further developments in instruments and devices for chemical analysis came from other fields of science, above all from physics.

The first branch of physics that chemists made use of was optics. In the 18th century chemistry discovered the microscope for his purposes and made attempts to conclude upon the nature of substances from crystal shapes. It was by means of the microscope that Marggraf proved the identity of beet sugar and cane sugar. In the last decade of the century a Russian chemist, Tobias

Lowitz developed a whole system for qualitative analysis based on crystal shapes and on the changes taking place under the effect of reagents. His system was no particular success, nor were various similar attempts. Up to the present the microscope is rarely used in analytical laboratories, and the same is true for its modern, high-efficiency sister, the electron microscope. For qualitative analysis, the solution tests carried out in the test tubes developed by Berzelius proved much more convenient and reliable.

Everyday practice demonstrated that colour strength of solutions depends on the amount of coloured substance contained in them, and this phenomenon could be utilized for quantitative determinations. The beginnings were very primitive again. Carl Heine, in 1845, poured equal volumes of solutions containing increasing amounts of bromine into equal test tubes and determined the concentration of the unknown solution by simple visual comparison. Subsequently various colorimeters were designed, using illumination, mirrors and other physical devices to increase accuracy of comparison. The best known were those of Müller (in 1853), of Dehm (in 1864) and of Duboscq (in 1870), the latter two being already produced in factories. Duboscq's colorimeter is shown in the figure. Here the colour density of the

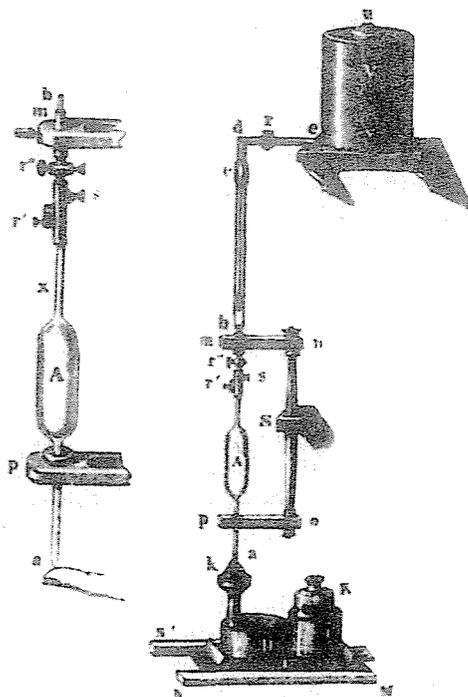


Fig. 11. Gay-Lussac's pipette filling equipment

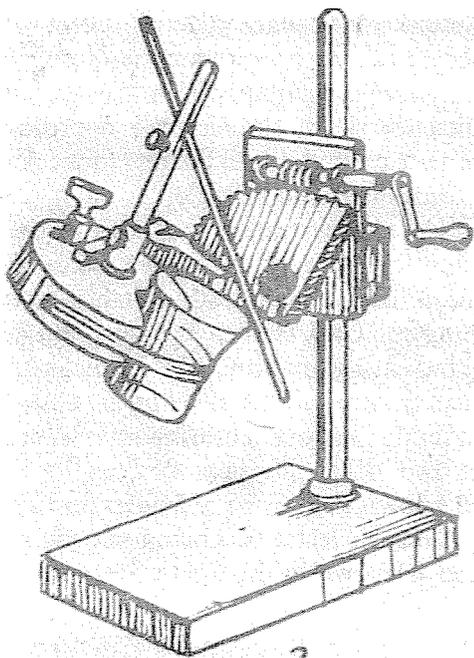


Fig. 12. Precipitate filtering device of Berzelius

unknown solution was measured directly by comparison with a standard solution. The light passed through two glass prisms so that one half of a circular plate was illuminated by the light passing through the sample solution, and the other half by the light passing through the standard reference solution. By moving the prisms one could achieve identical colour over the whole plate.

A revolution in qualitative analysis was brought about by spectral analysis. Although its history began with Newton, the spectra of the Sun and of different flames and the lines detected in them aroused the interest of physicists only, up to and including Fraunhofer. The first to suspect that the coloured lines in the spectra might indicate the presence of definite elements was Talbot, a British scientist, in 1826. He constructed a primitive spectroscope consisting of a light source behind a screen with a narrow slit and of a prism. The following years brought interesting investigations by Swan in Britain and Alter in the U.S. The latter made up a primitive table as early as 1854, which contained spectral lines of various elements. Complete success was attained, however, by Bunsen and Kirchhoff only, with their spectroscope. They were both famous scientists in 1859, when they came out with their spectroscope, and this fact largely contributed to the great attention which their announcement met, in contrast to those of the mentioned and non-mentioned numerous precursors,

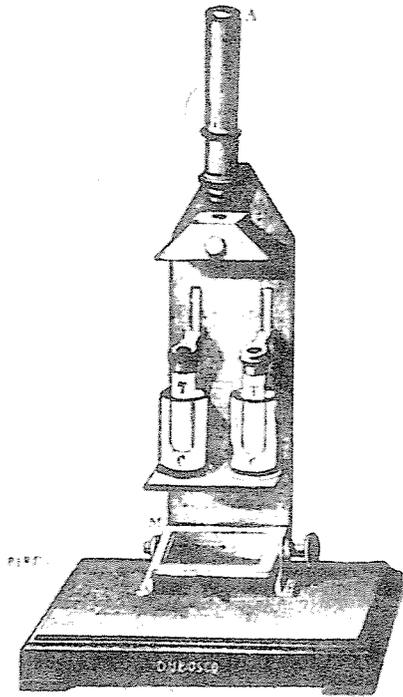


Fig. 13. Duboscq's colorimeter

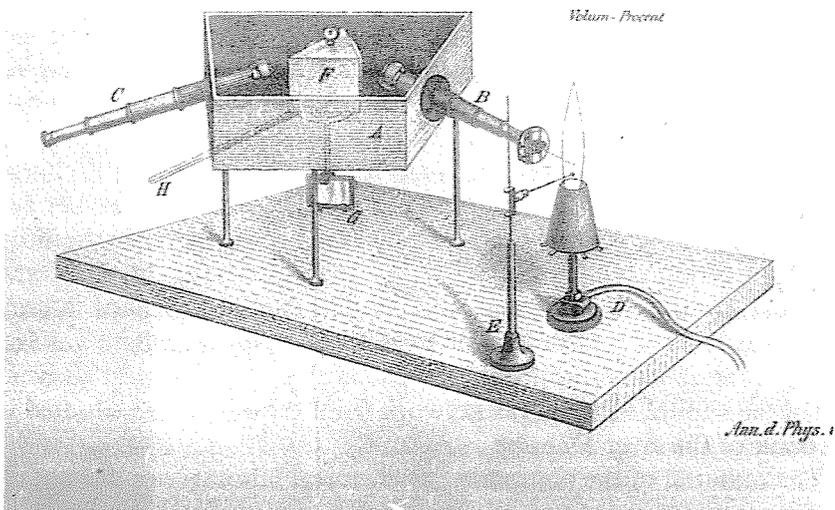


Fig. 14. The first spectroscope of Bunsen and Kirchhoff

to whom, by the way, with Swan's exception, they didn't even refer in their paper. Well, rapid success of a novel method or instrument doesn't only depend on its value, but also on the importance of the person presenting it!

In the present case, however, the spectacular success of the spectroscope did not prevent that Bunsen and Kirchhoff were accused of plagiarism, although it was unquestionable that their instrument was by far more effective than any of its forerunners, and that they were the first to interpret the difference between emission and absorption lines correctly. The prestige of their invention was largely increased by the fact that as early as in the following year Bunsen discovered two new elements: rubidium and cesium by its means. This success was followed by other scientists who discovered indium, thallium and gallium, and finally crowned the success by discovering helium in the spectrum of the Sun (its presence on the Earth was discovered only later.) Bunsen and Kirchhoff subsequently modified and improved the original model several times, Soon the spectroscope became a widespread, mass-produced instrument not only used for scientific research, but finding its way even into high-school laboratories.

The spectroscope was suited for qualitative analysis only; later developments made it suitable for quantitative determinations by combining it with photography. It took, however, a very long time till the problems associated with measuring and recording the intensities of spectral lines could be solved. Many researchers participated in this development and much disappointment came from following false tracks. The first spectrographs that were truly serviceable appeared as late as in the 'thirties of our century. The outstanding significance of spectrography was that it allowed to determine quantities smaller by orders of magnitudes than determinable by any other known method. Speaking of the history of spectroscopy I wish to mention Henry Rowland from Johns Hopkins University; the optical grating that he prepared in 1893 allowed high-accuracy measurement of wavelengths of spectral lines.

Lighting with gas became current in the 19th century, and simultaneously gas was introduced into chemical laboratories to replace all earlier types of heating devices rapidly, particularly after Bunsen constructed, in the 'fifties, the gas burner wearing his name and still in use in an almost unchanged form even in the most up-to-date electronic laboratories. It might perhaps interest you that gas was transported in leather bags to laboratories where gas facilities were not yet established, for instance in 1860 still to the Budapest University in a part of the capital with no gas network from the gas works situated on the other bank of the river Danube.

The material of the equipment in chemical laboratories did not change much for centuries: ceramics and glass, metals (above all iron) were current in alchemist laboratories; later, from the 18th century on, nickel and silver

crucibles came into use. A significant change was brought about only by platinum. This metal was imported to Europe from South America in the 18th century; its name originates from its appearance resembling silver: platinum is the diminutive form of plata, the Spanish word for silver. For a long time the chemists didn't know what to begin with it, they were unable to dissolve or melt it. Studies of platinum started towards the end of the 18th century only; Antal Ruprecht, professor of the Hungarian Mining Academy in Selmechánya was the first to melt it. Its processing, however, remained a problem for some time, although many attempts were made. Klaproth mentions platinum crucibles in 1802. A satisfactory method for forging platinum was finally developed in England by William Hyde Wollaston who made a fortune by it in the 'twenties of the 19th century. Platinum vessels were of immense help to chemists analyzing minerals, using increasingly sensitive procedures, since at last they disposed of a material from which no impurities could get into the sample.

The greatest invention of the 19th century with an immense effect on the progress of human society, the electric current was born in 1800 from electrochemistry with the famous Volta column. The chemical effects of the electric current were soon observed: in the very same year Cruishanks stated that metals are deposited by the electric current on the negative pole. It took a long time, however, until this finding was utilized in analytical chemistry by the American scientist Josiah Willard Gibbs who developed electrogravimetry in 1864. Platinum was of great importance as the electrode material; at the start a common platinum crucible served as electrode. The various electrochemical titration methods were invented towards the end of the 19th century. No very

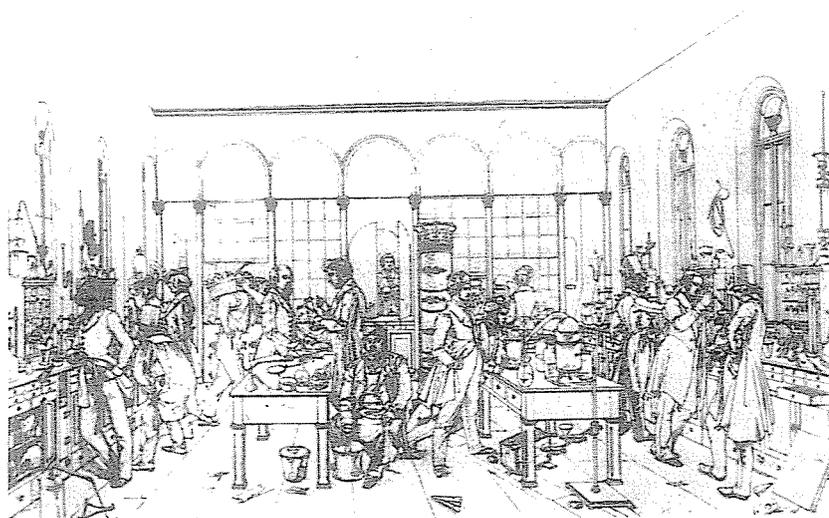


Fig. 15. Liebig's laboratory

particular instruments had to be developed for these methods: existing chemical laboratory equipment had only to be complemented with physical instruments such as voltmeters, amperemeters and the like. However, the methods became widespread in general analytical use only with the development of vacuum tube instruments; until then they were mainly considered curiosities of interest for physical chemists only.

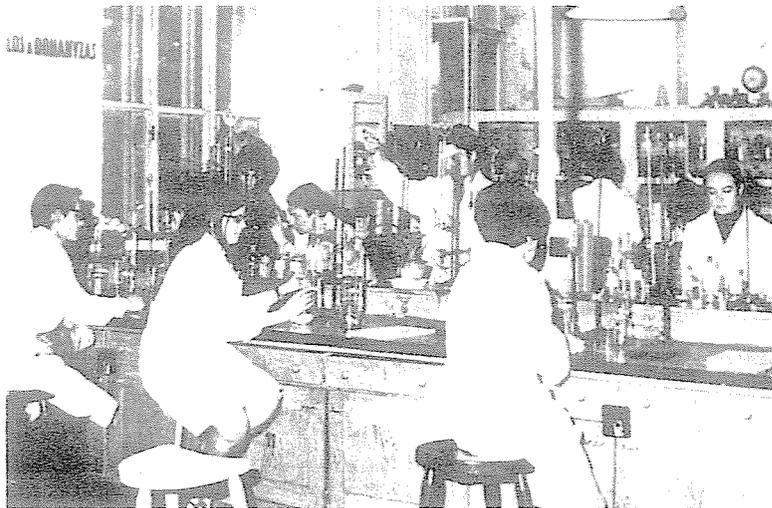


Fig. 16. Students analytical laboratory in the forties

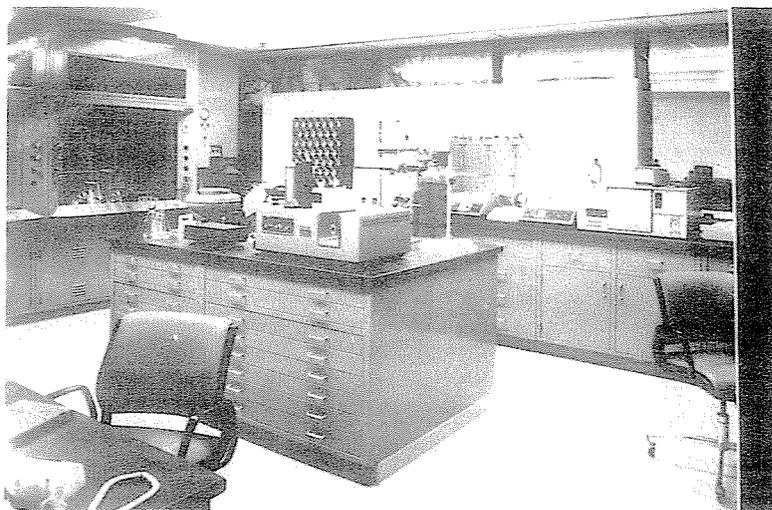


Fig. 17. Modern analytical laboratory

And so we have finally arrived to our age, to the second half of the 20th century, to the age when—after slow changes over many centuries—a dramatic change took place in the appearance of the analytical laboratory during the short period of the past 30 years. The laboratory in which I studied during my university years in the forties was still largely similar to Liebig's laboratory shown in Fig. 15. But what does a present up-to-date laboratory look like? Glittering electronic instruments, vibrating lights and scales, computers that print out results. However, somewhere behind all these instruments, the same chemical processes take place as in the traditional laboratories; they are almost invisible, but they generate the signals which will then be recorded and processed by the instruments, frequently automated to such an extent that no human intervention is required.

The change is indeed dramatic. And therefore I now have started to collect, in the Hungarian Museum of Technology, all sorts of equipment slowly disappearing from chemical laboratories such as tripods, stands, Erlenmeyer flasks, Liebig coolers, porcelain crucibles, mortars and pestels, water baths and so on, so that the typical analytical laboratory of the 20th century's first half should survive in the museum. Very soon the time will come when it would be too late to find the necessary specimens for this collection.

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