



ARTÍCULO DE REVISIÓN

From alkalis of commerce to iodometric methods: an annotation on the development of titrimetric analysis

De los álcalis del comercio a los métodos iodométricos: una anotación sobre el desarrollo del análisis volumétrico

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KEYWORDS

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ABSTRACT

This paper provides an overview of some of the early history of titrimetric analysis, focusing on the contribution of pharmacists to this field.

PALABRAS CLAVE

Análisis volumétrico
Historia
Contribuciones de
los farmacéuticos

RESUMEN

Este artículo ofrece una visión general de la historia del análisis volumétrico, centrándose en la contribución de los farmacéuticos a este campo.

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Este es un artículo de acceso abierto



1. INTRODUCTION

The term titrimetry or titrimetric analysis¹ is well known and is described as the analytical process in which an analyte is determined by its ability to participate in a chemical process (1): one reagent (the titrant) is added to a solution containing the analyte to be measured until the reaction endpoint is reached. The basis of this method stems from Richter's observations about saline solutions, which paved the way for the discovery of the laws of chemical combinations (2). The history of titrimetric analysis prior to 1806 has been brilliantly described by Rancke Madsen (3). Industry's need for rapid methods to determine acids, alkalis, carbonates and hypochlorites became the driving force in the early development of titrimetry (4-8). The contributions of various pharmacists to titrimetric analysis has been outstanding.

Hermann Kopp, the great historian of chemistry (9), did not hesitate in calling pharmacy the mother of scientific chemistry (10). This can be illustrated by the way chemistry was taught at the King's Garden (Jardin Royal de Plantes de Paris) (11-12). Chemistry was introduced as an academic discipline into the faculties of medicine, academies, botanical gardens and museums towards the end of the 18th century (13), while during that same century the chemist-apothecary was widely respected. Throughout the 19th century, in all social fields, chemistry and its "alter ego" pharmacy contributed to the following: resolution of judicial enigmas; improvement in industrial processes; safeguarding of public health and hygiene; verification of food quality; detection of fraud; assessment of the impact of industrial pollution; and the detection of occupational diseases (14). An appreciation of the history of pharmacy is essential for an understanding of how modern science was created, particularly the discipline of chemistry (15-18). In this context, Bertomeu (19) has recently clearly shown that this view applies to Spain in the 18th century. For Planchon (1897): "*De tous temps, la Chimie et*

la Pharmacie ont eu les rapports les plus intimes..." (12). For Klein (2004): "the connection between chemistry and pharmacy went back to medieval times" (13). A Spanish version of this work appeared in 2011 in Schrironia.

2. REGARDING THE ALKALIS OF COMMERCE AND THE BLEACHING OF TEXTILE FIBERS

The chlor-alkali and bleaching industries (8, 20-21), as well as the study of mineral waters and gunpowder (22) provided the basis for the first titrimetric procedures. In this context, special mention should be made of the contribution of Descroizilles (23).

After the death of Pierre-Joseph Macquer in 1784, Berthollet was named inspector of a dyeworks and director of the Gobelinos Manufactory (24). His most important contribution in this post was the discovery of the bleaching properties of chlorine (25), based on the observation of Scheele in 1774 that chlorine discolored vegetable matter. Berthollet showed that fixed alkalis help in this action, and then attributed to the oxygen that the gas supposedly contained. However, in 1810, H. Davy provided convincing evidence that the gas was an elementary body (20, 26), even though Berzelius initially did not accept this conclusion. Alkaline solutions, which were easier to handle than the gas or its aqueous solutions, began to be produced at the Javelle factory, near Paris, under the name of *Eau de Javelle* (24).

François-Antoine-Henri Descroizilles (1751-1825) (27), a pharmacist in Dieppe and one of the first pupils of the master apothecary Hilaire-Marin Rouelle (brother of Rouelle the elder "*Démonstrateur*" at the King's Garden), showed that the concentration of hypochlorite is the most important factor controlling the bleaching process, and developed a titrimetric (or volumetric) method for determining this concentration by titration with indigo solution. The first description of this is found in a work of Berthollet (1789) published in the *Annales de Chimie*: "...mais un habile chimiste de Rouen,



M. Décroisille qui faisait aussi des épreuves..." (25). Later, Descroizilles (1795) described in detail the procedure and the necessary apparatus (i.e., the burette, which he called "Bertholimeter") (27), as well as the determination of different classes of indigo, while also comparing the purity of different grades of manganese dioxide. The method of Descroizilles quickly gained popularity in the bleaching industry, both in France and abroad, in a modified form, as its results varied widely depending on the quantities of chlorine and indigo in the mixture, so that it lost its validity over time.

The first decade of the 19th century was a landmark in the development of titrimetry. In 1802, the *Administrateurs-Généraux des Poutres et Salpêtres* communicated some observations about the potash assay (28). Descroizilles (1806) published his *Notices sur les Alcalis du Commerce...* (submitted to the *Académie de Rouen* in the summer of 1805 (29), where he described a measuring instrument that he called an alkalimeter), which was translated into English in the "Philosophical Magazine" (30). The second part appeared several years later (31). After the 1806 publication, further editions of this work followed in 1818, 1824, 1830 and 1840. The last two were posthumous editions that included details of the burette with no less than four different scales, corresponding to the use of the chemical polymeter, the alkalimeter, the Bertholimeter and the acetimeter, as well as of a simple measuring cylinder graduated into milliliters (32). Gay Lussac was strongly committed to these methods (33) and contributed his creative genius to all their aspects, substantially improving the procedures, thereby considerably encouraging the development of titrimetric (or volumetric) analysis. He also popularized the terms burette and pipette. In his essay on commercial potashes, Gay-Lussac (1818) stated: "...*mais Descroizilles, dont le nom est cher aux arts...*" and concluded: "*En terminant, nous ajouterons que c'est Descroizilles qui, le premier, a mis en*

pratique l'essai des alcalis en les saturant par un acide..." (34). The influence of the work of Descroizilles on the subsequent development of titrimetric analysis is very clear and was reflected by the rapid development of this technique in the fifty years following the publication of his studies on alkalis.

3. FROM HISTORICAL ORIGINS TO IODOMETRIC METHODS

The halogens have become a particular battlefield for pharmaceutical researchers (20, 35). Scheele discovered chlorine (18, 26), Courtois iodine (36), and Balard bromine (37). Moreover, Moissan isolated fluorine (38-39), an achievement for which he was awarded the Nobel Prize for Chemistry in 1906, being the first French national and first pharmacist to receive this prestigious award (1, 40).

Chloride, bromine and iodine are closely related with early titrimetric methods. The blue color of the starch-iodine complex acts as an indicator for the detection of quantities of iodine trace elements and was first observed by Colin and Gaultier de Claubry (1814) (41). Henri François Gaultier de Claubry (1792-1873) was Professor of Chemistry at the new Pharmacy School founded in 1803 and an expert in Public Health and Hygiene (as well as pioneer in Exobiology). Like Vauquelin (discoverer of chromium and beryllium), he belonged to the group of outstanding pharmacists that were devoted to the study of chemical sciences (7). Friedrich Stromeyer (1776-1835), pharmacist and discoverer of cadmium, also independently reported the blue coloring in 1815 (42, 43). Stromeyer was a Professor of chemistry and pharmacy, a pupil of Vauquelin and Leopold Gmelin, Professor of Bunsen and predecessor of Whöler at Göttingen; he can be considered to be the pioneer of University laboratory-based teaching in Germany (44).

François Joseph Houtou de Labillardière (1796-1867), pharmacist, corresponding member of the *Académie de Paris*, and Professor of Chemistry at the *Académie de*



Rouen, introduced the use of iodine to titrimetric studies (45-48), thirteen years after its discovery by Courtois. He substituted indigo with a starch-iodine complex proposing an alternative procedure for the estimation of chlorine content in commercial calcium hypochlorite. This fairly unknown author made significant contributions to chemistry during the first quarter of the 19th century, even though he abandoned the subject at 37 (45), when he received a substantial family inheritance and had severe disagreements with two of his colleagues, Payen and Gay Lussac.

Alphonse Dumas (1793-1848) was Professor of Chemistry at the Lyon School of Medicine, and responsible for analyzing the sulfurous waters of the river Allevard (Isère). He reported the possibility of a rapid and precise titration of free or combined hydrogen sulfide (hepatic gas), using a standard iodine solution (49) in the presence of starch as an indicator. Dumas (1841) devised the “sulfhydromètre”, which was enthusiastically approved by Dumas and Pelouze in a report to the *Académie* (50). Two of his pupils, the pharmacists Mathurin Joseph Fordos (1816-1878), Head of the Laboratory at the Paris Charity Hospital, and Amadée Gelis (1815-1882), a manufacturer of chemical products, showed in 1843 that two atoms of iodine quantitatively oxidize two molecules of sodium hyposulfite (thiosulfate). This reaction constitutes the fundamental basis of iodometry (51-52). Fordos and Gelis are also famous for recommending the use of an aqueous solution of sodium aurothiosulfate for fixing photographic negatives (53).

Robert Wilhelm Bunsen (1811-1899) published his famous report on iodometry in the *Annals of Liebig* (54), with a summary in French appearing the following year (55). Bunsen showed the general nature of the method and described the determination of a wide variety of oxidizing substances releasing iodine from potassium iodide that were, subsequently, evaluated with a sulfurous acid solution. That same year, Schwarz (1853) (56), a Professor in Graz and

Breslau and a pupil of the French pharmacist Théophile Jules Pelouze (1807-1867), revised the reaction discovered ten years earlier by Fordos and Gelis, and proposed the replacement of the sulfurous acid, used by Bunsen in the titration of iodine, by sodium thiosulfate, which was a great breakthrough (7, 51) that has still not been surpassed. Nevertheless, the sulfurous acid solution was still used for a considerable time despite the difficulties in its preparation and its instability (57). Pelouze, assistant to Gay Lussac, Professor of the University of Lille, *École polytechnique*, and Director of the Paris Mint (a member of the *Académie de Sciences* in 1837), determined very accurately the atomic weights of various elements, and in 1838 prepared nitrocellulose, giving rise to the age of nitro-explosives.

Isaac Maurits Kolthoff (1894-1993) studied at the School of Pharmacy of the University of Utrecht, Holland, and presented his Doctoral Thesis in 1918 (58-59) on the “Fundamentals of Iodometry”, a recurrent topic to which he would return numerous times over the years. Kolthoff, pharmacist, father of modern Analytical Chemistry and author of thousands of scientific publications, worked at the University of Minnesota from 1927, and is famous for his motto: “Theory guides, experiment decides”.

4. REGARDING THE FIRST TEXTS ON TITRIMETRIC ANALYSIS

The first textbook published on titrimetry was by the German Karl Leonard Heinrich Schwarz (1824-1880), as indicated earlier the pioneer in the use of sodium thiosulfate. Schwarz (1853) coined the term “*Massanalyse*” (56), an expression derived from the French *dosage à liqueurs titrées*, which gave rise to the name of titrimetric analysis when translated into other languages (7).

Karl Friedrich Mohr (1806-1879) is considered to be the father of titrimetric analysis (60-62). Mohr's name is well known: Mohr's clip, Mohr's salt, Mohr's burette and pipette, Mohr's balance ... Nevertheless, he



has not received all the credit he deserved for his discoveries. Something similar to what happened to Descroizilles regarding his inventions, i.e., the flashing light beacon (*phare à éclipse*) and the coffee filter (23, 63). Mohr studied in Bonn and Heidelberg (where he became a friend of Leopold Gmelin), and learnt analytical chemistry in Berlin with Heinrich Rose. Gmelin came from a family of famous doctors and naturalists, who were themselves descendants of the Tübingen pharmacist Johann Georg Gmelin (1674-1778). Rose, a pharmacist in Danzig was laboratory assistant to Berzelius in Stockholm, son of Valentin Rose (also a pharmacist), and the discoverer of niobium. Once he had been awarded his Doctorate and passed the apothecary examinations, Mohr returned to his native Koblenz, where he had been an apprentice in his father's pharmacy. He then made a prolific and outstanding scientific contribution in a variety of fields, outside academic circles (like Descroizilles), except at the end of his career. His way of working made it difficult to fully recognize some of his contributions in the field of physics (60), specifically regarding heat and the conservation of energy, although Max Planck later recognized them in 1887. Nevertheless, this disadvantage was mitigated to a certain extent by his friendship with Liebig.

The great contribution of Mohr to Analytical Chemistry is his textbook on titrimetric analysis (an "Instructional Book of Titration Methods in Analytical Chemistry"), the first of its kind, published in two parts in 1855-56, which ran to many editions (64), and translated into French (65). In this textbook, he included his own methods together with those of previous authors, and popularized the use of normal standard solutions, an idea suggested by Ure in 1843 and at a later date by Griffin (7, 66). Mohr systematically used chemical reaction equations. He modified the form of the burette (with a tap) invented in 1846 by the French pharmacist Etienne Ossian Henry (1798-1873), Laboratory Director of the

Académie des Sciences Médicales, to make it easier to add small quantities of liquids and to avoid spillages. However, his contributions to titrimetric (or volumetric) analysis are not restricted solely to apparatus and techniques. He introduced hydrated oxalic acid (which has a solid crystalline structure, is easily obtainable and suffers little decomposition) as a standard for alkalimetry (a term credited to him, as was acidimetry) and ammonium iron sulfate (Mohr's salt) as a standard for potassium permanganate (2). In addition, he pioneered the use of potassium chromate as an endpoint indicator for the volumetric determination of chlorides, which was a landmark in the development of precipitation methods. Indeed, this method is still used today in the drinking water supply stations of the large cities. He used sodium hydroxide as an alkaline solution instead of ammonia and devised the calcium chloride trap (with quicklime and sodium sulfate) to prevent its contamination by carbon dioxide, as the change in the litmus paper from red to blue is in this case less sudden and less pronounced (2). He used back titration discovered first by Black, as well as arsenious acid for titrations involving iodine, and envisaged the idea of amplification reactions. However, he was fairly critical of the use of the thiosulfate as titrant reagent as proposed by Schwarz; time revealed Mohr's error (57).

In many aspects Mohr was ahead of his time, so that his outstanding ability was not fully recognized. Szabadvary and Chalmer (1979) allude to this: "In science, it is as unfortunate for a man to get before the age in which he lives as to continue behind it" (57), based on a comment of Thomas Thomson (1831) about Wenzel: "Wenzel in some measure went before the age in which he lived..." (67).

Antoine Baudoin Poggiale (1808-1879) must also be mentioned as belonging to the elite of French military pharmacists that included Bayen, Parmentier, Serulas, etc (68). Poggiale was Professor of Chemistry in Lille and at the Val-de-Grâce in Paris, the head military



pharmacist (*pharmacien inspecteur*), a member of the *Académie de Médecine* in Paris and of the Public Health Board. He undertook in-depth research of drinking and mineral waters.

Poggiale (1858) published a 600-page treatise on volumetric analysis (69), only two years after the publication of Mohr's textbook. Although this publication contributed to establishing his analytical reputation, it is not cited by Ranke-Madsen (1958) (3) or by Szabadvary (1992) (7), despite it being a standard reference work. The treatise gives a detailed description of the methods used prior to the textbook of Mohr and includes the analysis of gases and metals, hydrogen sulfide testing, chlorometry, acidimetry, alkalimetry, saccharimetry, etc; it also has 171 drawings that give insights into the apparatus used in these determinations. Poggiale was a strong defender of the application of chemistry to pathology and therapeutic care, especially against the attacks of the eminent clinician Armand Trousseau. He died the same year as Mohr, 1879, and as an anecdote, it is interesting that their obituaries appeared together the following year in the "Pharmaceutical Journal".

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- ¹ As measurements of volume play a crucial role in titrimetric analysis, it is also called volumetric analysis.

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