TOOLS FOR CHEMISTS: THE DESREUX-BISCHOFF VISCOSIMETER

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Summary

This particular tool was of use, during the heyday of polymer chemistry, in determining the molecular weight of a macromolecule. Arguably, "name" pieces of glassware for the chemical laboratory are landmarks in the history of chemistry and they thus deserve notice. The so far unacknowledged contribution of the glassblower, who actually built this apparatus, is put on record.

Introduction

The previous paper in this series was devoted to the so-called Dean-Stark trap, used to remove water from a solvent or a solution (1). The name of the glassblower, Mr. Demuth, who actually made the apparatus, was absent from the roster of authors, even though his contribution was acknowledged at the end of the article.

This was not an oversight. As a rule, glassblowers, and technicians more generally, were not included in print. It was a social class distinction. Laboratory technicians, during that period of the 1920s, were like blue-collar workers in industry. They were deemed mere manual workers. Authorship of scientific publications was reserved for scientists, typically those with a Ph.D. degree, whose contributions were recorded in a laboratory notebook, prior to possible transfer to journal pages (2).

Such an inferior status of technicians endured into the 1950s, as the present paper will showcase. Another glassblower, Mr. Wenig, was the "invisible man" in the paper I am about to describe and comment upon (3). Since I was professionally acquainted with this gentleman, this article also draws on personal recollections.

The Apparatus

The apparatus worked in what was then the standard way to measure the viscosity of a liquid, by timing how long a given volume took to flow through a glass capillary. Leo Ubbelohde, in Berlin, had both patented and published such a device towards the end of the 1930s (4). The diagram from his US patent is shown in Figure 1. The Ubbelohde viscometer—the two accepted spellings are viscometer and viscosimeter—which Desreux and Bischoff (3) used as their template, was of the "suspended level type," viz. referring to the air-liquid interface existing in the feeding bulb before the liquid flows through the capillary. This innovation obviated the need to correct for surface tension. Another correction is necessary in principle for the kinetic energy of the solution as it flows through the capillary tube. In order to minimize this second correction, one has to decrease the rate of the flow, which can be done either by a very narrow capillary tube—but then dust particles may affect the determination—or by reducing the hydrostatic pressure driving the flow.

Desreux and Bischoff opted for the latter solution (Figure 2), achieved by placing next to one another the two containers for the liquid solution, at departure into the capillary and arrival from it. The bulb that fed the

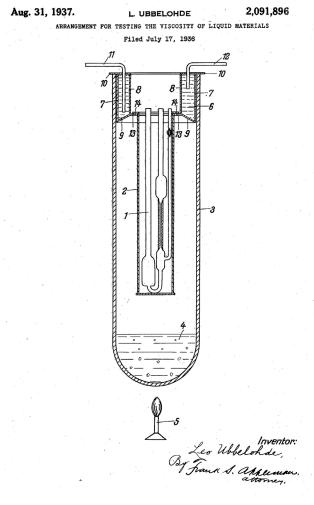


Figure 1. Diagram of viscosimeter from Leo Ubbelohde's US patent (Ref. 4a).

capillary (labeled 6 in the figure) had a fixed and known volume between 0.5 and 1 mL. The diameter of the capillary they used was 0.25 mm. Its length varied between 10 and 20 cm depending upon the viscosity to be measured, and the hydrostatic pressure driving the flow was kept to a 3 cm height of liquid above the capillary racetrack. 40 mg of polymer dissolved in about 7 mL of solvent was sufficient for a series of viscosity measurements, which translated into a determination of its molecular weight. Both filtration of the solution and serial dilution could be done within the apparatus. Filtration was accomplished by drawing the solution through fritted glass (labeled 4) and dilution by introducing solvent via the sidearm (labeled 1). The whole determination could be done in a couple of hours (3).

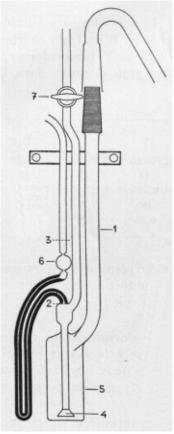


Figure 2. Diagram of viscosimeter Desreux and Bischoff's paper (Ref. 3).

The Plastics Era

1950, when this paper was published, besides marking the mid-twentieth century, was also a pivotal date marking the switch from a broad swath of natural products to synthetic materials made of polymers, which to a large extent were petrochemicals (5). There is a long list of such substitutions, of which I need mention only Plexiglas® for glass windshields; acrylates for wool and cotton; nylon and other polyamides for silk; synthetic elastomers for natural rubber; PVC for ceramic tiles, wooden floors and lead tubing; styrene-butadiene foam rubber for sponges; Formica® laminate from melamine resin (1938) for wood; polyethylene for glass in bottles; PVC-covered fabric for leather; polymeric substitutes for horn in combs; ... Indeed, such a list could go on for pages and pages (6). In 1950, it did not take the gift of prophecy to herald a triumphant Age of Plastics, as polymers became known popularly. To the historian, the justifiably famous line written by Buck Henry for the movie The Graduate (1967), "One word: plastics," the career advice given to the young Benjamin played by Dustin Hofmann, was late by a quarter-century.

Polymer Chemistry and Physical Chemistry in 1940

Hermann Staudinger (1881-1965), was the founding father of polymer chemistry, who had staunchly defended the revolutionary concept of macromolecules until it gained acceptance. In 1930, Staudinger proposed the simplest of correlations between the observable viscosity and the unknown molecular weight, a simple proportionality (7, 8).

During the late 1930s, American chemists burst upon the scene of polymer chemistry and they stole the show. A genius, Wallace H. Carothers (1896-1937), synthesized polyamides. The company he worked for, DuPont de Nemours, successfully began marketing stockings made from the new material, nylon, thereby replacing silk.

If there was a single scientist who carried forward Staudinger's and Carothers's work, developing the physical chemistry of polymers and the precise dependence of viscosity on molecular weight, he was Paul J. Flory (1910-1985). Other polymer physical chemists active in the field and who carefully studied the relationship between the viscosity of solutions and the molecular weight of the polymer were Herman F. Mark (1895-1992) and Werner Kuhn (1899-1963).

A student and a coworker of Carothers, Flory seems to have inherited from him the shuttling between industry and academia, between practical results and conceptual advances that was the distinctive mark, at least at some times, of the Experimental Station of DuPont, in Wilmington, Delaware. After a few years at DuPont, Flory started his academic career at the University of Cincinnati (1938-1940). He would return to industry from 1940 to 1948, when he again came back to academia.

While in Cincinnati, Flory investigated the relationship of viscosity to the length of a polymeric chain. He did so both empirically and theoretically, from first principles. Staudinger had been intuitively right, but factually mistaken. The intrinsic viscosity of polymers in solution is proportional to the 0.64 power of the molecular weight, rather than to its power unity (9).

Polymer Chemistry and Physical Chemistry in 1950

In the late 1940s-early 1950s, the main subcultures of chemistry, in an academic setting, were organic, inorganic, physical, analytical and biological. Before

university expansion reached full bloom in the United States, led by research universities funded by government grants, a predominantly post-Sputnik development, industrial laboratories were the main employers of university-trained chemists, at both the B.S. and Ph.D. levels (10). Pharmaceutical companies and dye manufacturers hired organic chemists, producers of commodities hired inorganic chemists, biological chemists found positions in government laboratories and specialized niches, such as breweries. Industry of all kinds had a need for analytical chemists, to run their spectroscopy apparatus in particular. Physical chemists to some extent enjoyed pride of place: industry looked to them for managerial positions, as group leaders, not only as specialists in instrumentation (11).

With the rise of petrochemicals and polymers, a new need arose in the aftermath of World War II for chemists trained in the brand-new polymer science. One such scientist, the head of a whole school of polymer chemists, was the previously named Paul J. Flory. Let me briefly remind the reader of his post-Cincinnati career, it will help to put into context the invention of the Desreux-Bischoff viscosimeter.

As Flory wrote in his Nobel autobiography (12)

In the Spring of 1948 it was my privilege to hold the George Fisher Baker Non-Resident Lectureship in Chemistry at Cornell University. The invitation on behalf of the Department of Chemistry had been tendered by the late Professor Peter J. W. Debye, then Chairman of that Department. The experience of this lectureship and the stimulating associations with the Cornell faculty led me to accept, without hesitation, their offer of a professorship commencing in the Autumn of 1948. There followed a most productive and satisfying period of research and teaching. *Principles of Polymer Chemistry*, published by the Cornell University Press in 1953, was an outgrowth of the Baker Lectures.

It was during the Baker Lectureship that I perceived a way to treat the effect of excluded volume on the configuration of polymer chains. ... It became apparent that the physical properties of dilute solutions of macromolecules could not be properly treated and comprehended without taking account of the perturbation of the macromolecule by these intramolecular interactions. The hydrodynamic theories of dilute polymer solutions developed a year or two earlier by Kirkwood and by Debye were therefore reinterpreted in light of the excluded volume effect. Agreement with a broad range of experimental information on viscosities, diffusion coefficients and sedimentation velocities was demonstrated soon thereafter.

In short, measurement of the viscosity of polymer solutions was a very important piece of data at the beginning of the 1950s, when Desreux and Bischoff published the apparatus they had devised. Their publication (3) mentioned only the Staudinger proportionality, ignoring the 1940 correction by Flory.

The Belgian Context

Victor Desreux, when he designed this new tool for polymer chemists, was a professor of physical chemistry at the University of Liège, in Belgium. Given the lead Flory and others had taken in polymer chemistry, one might have expected for this invention to have occurred in an American institution. Thus, a word of explanation is needed, concerning Belgium and chemistry in Belgium, during that period of the beginning of the 1950s.

With a population then of 8.6 million people, this small country had four universities. They reflected a careful political balance. Two were private, so-called Free, universities; the other two were State universities. The Free universities were the Catholic University in Louvain (Leuven), which had existed for many centuries as a gem of the Catholic Church. To counterbalance its influence, Brussels housed the officially atheistic Free University, rumored to be under the influence of Freemasons. As for the officially non-ideological State universities, one was located in Ghent (Dutch-speaking Flanders), the other in Liège (French-speaking Wallonia). At that time (1950), French speakers made up the political, administrative and educational elite of the country. Of these four universities, the Catholic university in Louvain was the most prestigious and was known the world over.

During World War II and their occupation of Belgium, the Nazis had played on the linguistic split between the Flemish and the Walloons. They deemed the former legitimate Aryans and the latter degenerate Latins. After Germany lost the war, the Flemish were again under the political rule of French speakers. Only several decades later would they come out on top, on the strength of their more prolific demography. Since World War II has been mentioned, let me note for future reference that Belgium acquired, as part of war reparations, a significant number of German prisoners. They were put to work in the coal mines, located predominantly in the Walloon part of the country.

Belgium, previously part of the Netherlands, gained its independence in 1830 and was set as a buffer state, between Holland, England, Germany and France. It was the

second region in Western Europe, after England, to have undergone the Industrial Revolution, drawing on its coal mines. Hence, Belgians remained keenly aware, in the aftermath of World War II, of the economic importance of industry to their prosperous well-being. Even though Belgium was a small country, its industrial exports, then greater than those of the Soviet Union, were in 1950 among the industrial giants in the world. At that time, Belgium was also a colonial power. Its possession of the Belgian Congo gave it enormous mineral wealth (13).

In terms of the chemical industry, Belgium was home to the Solvay corporation, started by Ernest Solvay (1838-1922) in 1863, that had thrived on exploitation and exportation of the Solvay Process. By 1950, it became the biggest producer of chemical commodities in Belgium, including polymers such as PVC (14). Belgian academic chemists, proud of the industrial achievements of their country, felt very close to the concerns of their industrial counterparts.

Thus, even though their number was small, they did not feel inferior to their British, Dutch, French and German colleagues. In mentality, because Belgium was such a new country, because World War I had been fought to a significant extent on its territory, because the United States had intervened in both World Wars to liberate it from the Germans and had helped afterwards in its reconstruction, and because of its Swiss-like prosperity relative to adjoining countries, Belgians were Americanophiles and their affluent lifestyle was very much American-like.

Victor Desreux, the Senior Author

The senior author, Victor Desreux (1910-2004) was born and educated in Ghent. He earned a doctorate in chemistry at the University of Ghent in 1934, when the French language was still tolerated there—only a year before full Flemishization was imposed in 1935-36—in Frédéric Swarts's (1866-1940) laboratory, devoted to fluoro-organic molecules (15). A co-worker of Desreux, Ms. Yvonne Désirant, achieved the first preparation of hexafluorobenzene.

During the ensuing years, Desreux had outstanding training. He followed a path worthy of a student in Early Modern times, with study in no fewer than four institutions of higher learning in three different countries outside of Belgium. It covered a wide diversity, not only of topics, but of sub-disciplines of chemistry as well.

His first postdoctoral stay was in 1935 with Professor Georges Dupont (1884-1958)—an organic chemist

whose specialty was terpenes, with applications to perfume chemistry—at the École normale supérieure, in Paris. Half-a-dozen publications resulted from that stay (16). For instance, Desreux and Dupont made a chiral allene, taking advantage of the acetylene-to-allene rearrangement. On to Utrecht, where he spent the year of 1936 in Professor Hugo Rudolph Kruyt's (1882-1959) laboratory, devoted to the physical chemistry of colloids. Then, with a fellowship from the American-funded Commission for Relief in Belgium, from 1937 to 1939 Desreux went on to Cambridge, Massachusetts, where he was a postdoc in Louis F. Fieser's (1899-1977) laboratory at Harvard. He worked on carcinogenic aromatic hydrocarbons, synthesizing derivatives of 20-methylcholanthrene (17). During his stay, Desreux presented a seminar on his doctoral work and organofluorine chemistry (18). In 1938-39, Desreux moved to Princeton, New Jersey, where the Rockefeller Institute—later upgraded to a University—was then located. He joined the protein laboratory of John H. Northrop (1891-1987), Nobel prizewinner in 1946, where he worked on the enzyme pepsin, and its preparation as a pure protein. Three publications ensued from Desreux's work at Princeton (19).

Only then, after spending four years abroad, did Desreux return to Belgium—and the onset of World War II, with Nazi Germany occupying Belgium. He received a teaching position in 1941 at the University of Liège, as *chargé de cours* (lecturer). He was able to resume physico-chemical studies of polymers in 1945, after the war ended. He gained a full professorship, in physical chemistry, in 1946. Belgian universities copied the German faculty system. Full professors came in two categories. An *ordinarius* professor was full-time and had to reside locally. An *extraordinarius* professor was a visiting, part-time faculty member. Desreux became an *ordinarius* professor.

As for his coworker J. Bischoff, he was a graduate student who acquired his doctorate working under Desreux's supervision on polymer chemistry (20) and who left his laboratory, presumably for an industrial position, by the mid 1950s. In the meanwhile, he was a postdoc in Professor Arthur V. Tobolsky's (1919-1972) laboratory in the Department of Chemistry at Princeton (21). (Tobolsky and I were colleagues there during the ensuing decade, in the 1960s).

Finding the Invisible Man

In the aftermath of World War II, the city of Liège reverted to its former life, cultural, social and economic.

The local bourgeoisie, French-speaking, proud of institutions such as the University, the music conservatory and symphonic concerts, a theater and an opera, thrived on the proceeds of geography. Liège, on the river Meuse with important barge traffic, was well located between Brussels, Antwerp, Maastricht and Aachen, by road or rail. Trade was thus a major factor in its prosperity. Another was manufacturing.

Engineers were a significant part of the city elite. Liège, rather comparable in that respect to the Pittsburgh I have depicted in the previous paper in this series, was blessed with a natural resource—coal rather than petroleum. Nearby coal seams, within the French-speaking Walloon area, fed into the dominant industry in Liège, siderurgy. The iron ore came by rail, via Luxemburg, from Lorraine in France.

Coal mining is hard work, and it is also dangerous. Hence, the Liège bourgeoisie imported the needed workforce. At first, it resorted to the Flemish. But after their Belgian fellow-citizens organized themselves as efficient agriculturalists, via the Boerenbond organization, and prepared themselves to rival French speakers for running the whole country—as we saw, an early symptom was their take-over of the University of Ghent starting in 1930—there was a need to replace them in coal mines. Thus, Poles and Italians were imported as coal miners and steelworkers (22). This population of immigrants settled in the suburbs of Liège.

With the end of World War II, a yet cheaper source of labor could be tapped, German war prisoners. As part of the war reparations, Belgium was able to secure an abundant supply of slave labor from Germany. Abundant? No fewer, than 60,000 German prisoners were obtained from the Allies. They were coerced into coal mining, under subhuman conditions that violated both international (the Geneva Conventions) and national (the Belgian social legislation) laws. Their working and living conditions were so severe that 4,000 Germans tried to escape. 23 were shot and killed in the attempt. This resort to slave labor, Germany being repaid in its own currency, lasted between 1945 and the spring of 1948 (23).

During that period, at the beginning of 1947, Professor Desreux and his colleagues from the Institute of Chemistry, Louis D'Or and Georges Duyckaerts, needed a glassblower for their Institute. Desreux had an idea: surely, there must be among the German prisoners some who had been trained as glassblowers in their earlier, civilian life. Before the war, Germany, factories in Jena

in particular, had enjoyed a reputation as world leaders in glasswork of every kind.

The Liège chemistry professors advertised their offer to this large captive population, a competition would be held at the University of Liège for a glassblower, who accordingly would be liberated from his brutal coal mining duties. Three young Germans, with the proper credentials, were selected (24). After he won the competition, Mr. Heinz Wenig started work at the Chemistry Institute on May 15, 1947.

One might have expected him to have returned to Germany during subsequent years. However, he met a local Belgian woman whom he married, they started a family, and he continued working for the University of Liège, heading a small glassblowing workshop with three or four coworkers at the Chemistry Institute until his retirement, aged 60, on July 1, 1984. During the intervening decades, Professor Desreux remained the administrative supervisor for the glassblowing shop (Figure 3).



Figure 1. Heinz Wenig, the glassblower (left), and Professor Victor Desreux (right). A picture from the late 1970s or early 1980s.

As a personal note, I was acquainted with Mr. Wenig for almost my whole time as an *ordinarius* professor of chemistry at the University of Liège, 1970-1986. (I became *extraordinarius* from 1986 until 1995 when I returned to the *ordinarius* status, until 1999 when I retired.) As a craftsman, I found Mr. Wenig to be superb. He told me he enjoyed the challenge of the most intricate tasks. As a person, he was very congenial, friendly without being obsequious and he spoke French with a melodious German accent. He and I also had a connexion because of the tragedy that befell him. A couple years after I started teaching in Liège, I had his son as a student, a freshman

in biology. A few weeks into the semester, the young Wenig was killed in a car accident.

Laboratory Technicians

Universities in Belgium, such as the University of Liège, are representative of European universities in the way they treat laboratory technicians. In mid-twentieth century, at the time Desreux and Bischoff published their design of a capillary viscometer, someone like its craftsman, Heinz Wenig, enjoyed a well-recognized status, a position guaranteed for years, even though the salary was meager (25). At least during the first two decades, he had to clock in and out, like a factory worker. Even though he did not sign the publication, I surmise that Mr. Wenig contributed to the design of the apparatus he built.

During the second half of the twentieth century, technicians with permanent appointments were viewed as an asset for European academic scientists, Belgians in particular. Their American colleagues were envious, they did not enjoy the same privilege. American universities maintained an alternative organization, in a tradition going back to Justus von Liebig's laboratory in Giessen (26). Graduate students did the technical work, and thus had to be trained anew in these ancillary technical tasks every few years.

Named Glassware

Even though the new device was described in a relatively obscure chemical journal, it was nevertheless adopted by a number of laboratories, in polymer chemistry predominantly—this is no surprise—all over the world. The paradox is that by publishing a description of this little device, Desreux and Bischoff achieved immortality—of sorts. It became known henceforth as the Desreux-Bischoff viscometer. Looking up these words with Google Scholar, yielded 242 hits on February 19, 2014.

It is in good company. To think of it, quite a few other pieces of glassware bear the names of their progenitors. I have often toyed with the idea of teaching a history of chemistry, anchored by such devices. It would provide quite a different narrative from the more usual, geared to Nobel prizewinners. Just as there are name reactions, there are also name (or named) pieces of laboratory apparatus, more often than not part of the glassware. They vouch for the permanence of glassware in both chemistry stockrooms and laboratories.

Even a short list would need to include several dozen named pieces of glassware, such as, in alphabetical sequence (the) Abderhalden drying pistol / Allihn condenser / Bennert vacuum gauge / Buchner flask / Buchner funnel / Dean-Stark trap / Dewar flask / Drechsel bottle / Eppendorf tubes / Erlenmeyer bulb / Erlenmeyer flask / Florence flask / Friedrich condenser / Gay-Lussac pycnometer / Gooch crucible / Graham condenser / Hempel, Oldershaw, Snyder and Vigreux distillation columns / Hirsch, Powder and Filter funnels / Hopkins reflux condenser / Imhoff cone / Kipp's apparatus / Kitasato flask / Kofler bench / Liebig condenser / Liebig kaliapparat / McCarter sublimer / Ostwald viscometer / Pasteur pipettes / Petri dishes / Schlenk flask / Schott bottle / Soxhlet extractor / Thiele tube / Ubbelohde viscometer / West condenser.

Naming pieces of glassware, in like manner as with name reactions (27), pays homage to their inventors. Which shows, again in like manner as with key transformations, the central importance of designers of novel glassware to chemical history.

Conclusion

Thus we end this microhistory with the notion of the importance of those scientists and technicians having devised a tool for the laboratory, at least in the form of glassware. One of the virtues of a microhistory is to resurrect otherwise anonymous persons, Mr. Heinz Wenig in this case. That chemists are history-conscious is well established; the popularity of chemical genealogies partakes of the same spirit as the naming of reactions and of glassware instrumentation. Why such an acute consciousness of past achievements and achievers? Because chemistry, in addition to being a science, is a craft. To this day, its knowledge, theoretical and practical both, is passed on as master-to-apprentice. At least in that aspect, chemists are the heirs to alchemists.

Acknowledgments

I am indebted to Dr. Jean-François Desreux, emeritus professor of chemistry at the University of Liège, for sharing with me documents about both his father and Mr. Heinz Wenig.

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About the Author

Pierre Laszlo, who was born in Algiers in 1938, is a French physical organic chemist. After obtaining his Ph.D. with Edgar Lederer, he was a postdoc at Princeton University with Paul von Ragué Schleyer (1962-63). After his D.Sc. (1965), he returned to Princeton as an assistant professor (1966), accepting in 1970 a call from the University of Liège, Belgium, as a full professor. He taught there until 1999 while, from 1986 on he also held a professorial appointment at the École polytechnique, near Paris. After retirement in 1999, he became a science writer and chemical historian.