

# Advances in Carbohydrate Chemistry

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## PREFACE

This volume completes fifteen issues in the series initiated in 1945. A résumé of the life and work of the late Emil Heuser is herein provided by L. E. Wise, one of his associates, in the U. S., in a career embracing both sides of the Atlantic. Capon and Overend comment on the general topic of the physicochemical properties of sugars, while Bouveng and Lindberg collate methods currently employed in the assignment of structure to polysaccharides. Specific instances of polysaccharide structure and properties are taken up by Neely for the bacterial polysaccharides of the dextran group, and by Foster and Webber for chitin, the widely occurring, encrusting polysaccharide. An extensive tabulation of the amino sugars and their derivatives is made by Horton, as a modernized replacement for the one in Volume 7 and as an appendix to the chapter on Aspects of the Chemistry of the Amino Sugars by Foster and Horton in the preceding volume of this series. An article by Hough, Priddle, and Theobald delineates the chemistry and rather complex nomenclature of the sugar carbonate and thiocarbonate esters, and indicates that these esters might well be employed more extensively as "blocking groups" in synthetic efforts. The subject of the carbohydrate components of bacteria is always fascinating; it is reviewed again, by Davies and by Jonsen and Laland, in two closely related chapters. Finally, Sprinson writes authoritatively on the biosynthesis of aromatic amino acids. It is shown that the benzene ring originates in carbohydrate material, thus placing the aromatic compounds as a subsection under the carbohydrates (from the bio-organic viewpoint). The Subject Index has been compiled by Dr. Robert Barker.

The editors record with sorrow the death on March 9, 1960 of Professor Hermann O. L. Fischer, a member of our Board of Advisors since the inception of this series.

*Columbus, Ohio*  
*Washington, D. C.*

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R. STUART TIPSON

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## EMIL HEUSER

1882-1953

Less than three months before his death on December 24th 1953, Emil Heuser was still conducting seminars on cellulose in Seattle and in Portland. Although he realized that he had certain serious physical disabilities, his verve and enthusiasm never flagged and his interest in Nature's most abundant polysaccharide was ever maintained. A friendly humorist termed him "Dr. Cellulose," and in many ways this cognomen described him aptly, although it failed to account for his many other interests and facets.

A colleague once stated that Dr. Heuser had been introduced, almost in his infancy, to cellulose technology. His father supervised the manufacture of playing cards in a factory at the back of the parental home in Stralsund—a seaport town in East Germany, where Emil was born on September 15th, 1882. Very early, Heuser's interest in organic chemistry and its technological applications became manifest, although it seemed at first as though he might become a mechanical engineer. After his graduation from the Real Gymnasium, he served briefly in the Potschappel Manufacturing Co. (in Dresden), which built machines for the pulp and paper industry; and he began his studies in engineering in the Technische Hochschule in Munich. Soon, he found that his main interests were in chemistry. He took chemical courses at the University of Munich, and later at Graz. Finally, at the Technische Hochschule in Karlsruhe, he received the degree of Doktor-Ingenieur in 1909. His thesis, however, was on oxalomalonic ester, studies of which he had begun under the direction of Professor Roland Scholl in Graz.

Soon after receiving his degree, Heuser served an apprenticeship of more than three years in the pulp and paper industry, at a time when paper-making was an art, the secrets of which were divulged only to those who had been initiated properly. Heuser worked in the rag mill of Gebrüder Laibling in Pfullingen, Württemberg. Then he entered the laboratory of the Altdamm kraft pulp mill in Pomerania. Subsequently, he gained experience in coating paper at the Zehlendorf mill near Berlin, and at the Steyrermühle Zellstoff und Papierfabrik in Austria. Thus, he learned some of the more practical aspects of the paper industry.

These early experiences were terminated when Heuser, in his thirtieth year, was offered the Chair of Cellulose Chemistry, vacated by Professor Dr. Carl G. Schwalbe, who had established this branch of learning at the



Technische Hochschule in Darmstadt. The young chemist accepted the appointment, and his prolific writings began soon thereafter and continued until a few years before his death. He also aroused the enthusiasm of numberless students, for he was a colorful, spirited, meticulous, and, at times, peppery lecturer, who had a certain flair for the dramatic. Two years after the young professor's appointment came World War I, with its concomitant interruptions. Heuser's industrial experiences were called upon, in some new areas. He met these assignments with his usual resourcefulness and energy. He was asked to help in the development of a plant in Monheim designed to hydrolyze sawdust and other wood wastes by the Classen procedure, using hydrochloric acid and sulfurous acid at high temperatures. This antedated the Scholler-Tornesch method. Pilot-plant runs were followed by actual production of sugar. However, Heuser's interest in this venture was not limited to the preparation of the fermentable hexoses. He had the farsightedness to examine the possibilities of producing furfural as a valuable commercial by-product.

In the war years, Heuser also undertook the project of building units for converting sulfite spent liquor into alcohol, as well as a pilot-plant operation for the production of methanol, acetone, and other products by the Rinman process. Certain of his experiences were published, even while the war was still in progress.

With the close of the war, Heuser's work gained marked recognition. A large abandoned armory had been placed by the Hessian Government at the disposal of the Technische Hochschule at Darmstadt. This armory was refurbished under the direction of Heuser's engineering colleague, Professor Berndt, and was then furnished with suitable equipment by the German pulp and paper industry. This "Institut für Cellulosechemie," as it was now called, became one of the most important centers of its kind, in Germany, for the education of chemists and engineers entering the fields of cellulose and of pulp and paper manufacture; and Darmstadt has retained this enviable position ever since, even though the Institute was destroyed in World War II and then rose again like a Phoenix from the ashes.

In these early years, Heuser wrote chapters for various texts and monographs, on such varied subjects as paper pulp and artificial silk, the coloring of paper on paper-machines, celluloid and various cellulose films, methods of determining furfural, and xylan.

In 1921, the first edition of his *Lehrbuch der Cellulosechemie* appeared, followed in 1923 by a second, and in 1927 by a third edition. These volumes established Heuser's reputation internationally. The second edition was translated into Russian by Lepin and Shilova in 1923, and into English by West and Esselen in 1924. The importance of this textbook cannot be overemphasized. True, there were earlier and far bulkier works on cellulose,

such as those of Schwalbe, and of Cross and Bevan, and there was also a chapter by Heuser in Mussprat's *Ergänzungswerk* (Vieweg und Sohn, Braunschweig, 1915). But these always led to confusion.

In his introduction to the *Lehrbuch*, Heuser states objectively: "Even the methods of treatment adopted by Schwalbe and by Heuser seem to be fundamentally unprofitable, because they do not lead to definite results. These authors have divided the subject along the lines of the various derivatives of cellulose, such as cellulose hydrate, hydrocellulose, oxycellulose, etc. Such discussions are of little value because the substances to which the above names have been applied are not homogeneous chemical individuals, but are mixtures of the most varied degradation products of cellulose and may react very differently under apparently similar conditions. Cross and Bevan have also made the same unfruitful and dangerous mistake of trying to build a system of cellulose chemistry on the basis of such mixtures..."

How did Heuser extricate himself from this jungle? West and Esselen state succinctly, in their translator's preface, what the new textbook had accomplished. "Starting from the premise that cellulose possesses the character of an aliphatic alcohol, one which every chemist will grant, the various reactions of cellulose have been correlated with the reactions of this class of compounds. In this way we have a consistent though fragmentary view of the chemistry of cellulose."

Thus, Heuser took up the "alcoholates" of cellulose; its esters, its ethers, and the oxidations of cellulose which led to carbonyl groups and to carboxyl groups. But he also had an important chapter on the hydrolysis and the acetolysis of cellulose, on its decomposition by heat, and its degradation by bacteria. His final chapter, on the constitution of cellulose, was largely historical in nature, and, viewed from present-day knowledge, it was, perhaps, less successful than were some of the others. But the entire little volume was conceived from a logical viewpoint—one that was very welcome to those of us who had tried to cope with some of the older literature. The book was relatively brief and very clearly written.

Heuser was also a pioneer in certain of his researches. He was one of the first to show that some types of oxidized cellulose, when treated with mineral acids, give carbon dioxide, thus indicating that carboxyl groups are present. In his studies on lignin, he deduced evidence that this system might contain aromatic groups; alkaline fusion of sprucewood lignin yielded protocatechuic acid and pyrocatechol, and Heuser and his coworkers obtained extensive quantitative data in this area. Another early piece of work involved the preparation of purified xylan from straw.

Heuser did not limit himself to purely basic studies. In the technological field, he did some significant orienting work on the chemistry of bleaching,

and in 1923, with Niethammer, he published the results of comparative studies on the use of hypochlorite and of chlorine gas in pulp bleaching. Apparently, this was a forerunner of the use of chlorine, in the initial stage of the commercial bleaching operation, that was universally adopted by the industrialists.

An important hypothesis formulated by Heuser was an extension of one suggested much earlier by Anselme Payen, but it was based on firmer experimental data. It stated that, irrespective of source, all celluloses, when freed from other components (or impurities), had one and the same constitution. The various chapters in Heuser's *Lehrbuch* rested on this assumption.

Emil Heuser not only enjoyed writing but spent much of his time in digesting, and critically evaluating, the data accumulated by others. Thus, even prior to the publication of the first edition of his *Lehrbuch*, he founded and edited the journal *Cellulosechemie*. In the first issue, the editor wrote the leading article on developments in the chemistry of cellulose. This number, and the issues which followed, also printed abstracts of articles dealing with cellulose and related products. Very soon, this modest journal grew in stature, German and foreign chemists served as associate editors and as contributors, and Heuser himself published therein the work of many of his students. Others whose articles appeared in various issues of *Cellulosechemie* were Hans Pringsheim, Kurt Hess, Karl H. A. Melander, H. Ost, Erik Hägglund, Astrid Cleve von Euler, Fritz König, Walter H. Dore, S. V. Hintikka, I. Sakurada, Selman Waksman, Carl Schwalbe, Erich Schmidt, Kurt H. Meyer, Hermann Mark, and Paul Karrer.

*Cellulosechemie* was published between 1920 and 1936, and again between 1940 and 1945, and Heuser remained its editor for the first six years of its existence. From 1918 to 1926, he also edited the technical section of *Der Papier-Fabrikant*, the publication of which as a separate journal was discontinued in 1943, although it was then combined for a short period with the *Wochenblatt für Papierfabrikation*.

When Heuser was forty-one, in 1923, he had already published seventy papers on cellulose, lignin, and xylan, on various aspects of wood chemistry, on pulp and pulping, and on a number of miscellaneous topics. His textbook, in its second edition, had had immediate acclaim in Europe and in the United States. He had directed the work of a number of brilliant students, and he flourished in the academic atmosphere of Darmstadt.

However, he was a married man with a growing family; the German economy left much to be desired, and the salaries at a technological institute could not compete with those offered by Industry. So it was that Professor Heuser left his pleasant post and became the Director of the Research Laboratories of the Vereinigte Glanzstoff Fabriken A. G. at Elberfeld in

Seehof-Teltow, near Berlin, with the assurance of having a relatively free hand and of being able to give lectures at the Technische Hochschule at Charlottenburg.

During the course of his stay in Darmstadt, Heuser had applied for at least ten patents, seven of which were granted while he still held the Chair of Cellulose Chemistry. The other three were granted soon thereafter. The patents (in some cases taken out with an individual coworker) dealt with such varied subjects as sizing, the production of methanol, tar, carbon, and gaseous fuels obtained in the pyrolysis of lignin, the permanent elimination of reddening in sulfite pulps, and the production of protocatechuic acid and pyrocatechol.

His publications continued during his three years at Seehof. An important series of researches (begun at Darmstadt) dealt with alkali-cellulose. One of these papers, with Bartunek [*Cellulosechemie*, 6, 10 (1925)] described the swelling and sorption of alkali by cellulose in potassium, sodium, lithium, cesium, and rubidium hydroxides, phenomena that were correlated with the differences in hydration of the individual cations. With Norbert Hiemer, in 1925, Heuser published three noteworthy articles on the depolymerization of cellulose [in *Cellulosechemie*, 6, 101, 125, 153 (1925)]. These gave the results of comprehensive, systematic researches on the methyl ethers obtained from cellulose in various stages of "depolymerization." The authors determined the properties of a series of these products which represented methyl ethers ranging in molecular weight from about 3300 down to approximately 700. The data given in these papers may well have formed the basis for the technological production of the water-soluble ethers of cellulose.

Another area explored by Heuser was that of cellulose xanthation, and the practical significance of certain of his studies soon became apparent to those interested in viscose formation. The behavior of cellulose xanthates in solutions of caustic soda of various strengths was investigated, the depolymerization of cellulose during the process of aging, xanthation, and ripening was followed, and methods for monitoring these changes were devised. In 1926, Heuser with M. Schuster described the scientific basis for the viscose process [in *Cellulosechemie*, 7, 17 (1926)], and, in one of his lectures given before the National Meeting of the Verein Zellechemie in Berlin, he emphasized the importance of purely scientific studies on cellulose to the industries using this raw material. Often, Heuser stressed basic work as well as the specific commercial applications to which it might lead. He was rapidly becoming an expert in the dissolving-pulp industry.

During the interlude with the Vereinigte Glanzstoff Fabriken, Heuser also published papers on the carbonic acid esters of cellulose (formed from methyl chloroformate and a partially hydrolyzed cellulose), on the degrada-

tion of cellulose with formic acid, on the action of chlorine dioxide on cellulose and the pentosans (which was a gentle critique of E. Schmidt's earlier work in this same field), and on the alkaline degradation of cellulose at higher temperatures.

In the Fall of 1926, he accepted an offer from the Canadian International Paper Company, in Hawkesbury, Ontario, to become their Research Director, and sometime thereafter he became a Canadian subject. He remained in this industrial position until 1938. The company manufactured both paper and dissolving pulps, and here Heuser's earlier experiences in pulping, bleaching, and the development of analytical procedures stood him in excellent stead. However, this was the one period of his life when his publications had to be kept at an irreducible minimum. During this era, he published an article with Schorsch on the action of alkali and carbon disulfide on xylan, and an earlier lecture of his on the hemicelluloses saw the light of day. He also published several papers on multistage bleaching and on the Thorne bleaching process, and a few of his other lectures also appeared in print, but the output was very slight when compared with that of earlier days. Despite this, he attended scientific meetings quite regularly, and in 1937 was elected the Chairman of the Division of Cellulose Chemistry of the American Chemical Society. When discussions arose, Heuser was always a stimulating participant and an active catalyst.

However, he was not in his true element. He longed for the quiet of Academe, the privilege of writing as he pleased and of teaching as of yore. This restiveness was manifest in letters written to his colleagues during this period, and it was not surprising that, in 1938, he left Canada to come to Appleton, as Research Associate and Group Leader at The Institute of Paper Chemistry. Here, in a quieter and more serene *milieu*, he could teach on the graduate level, inasmuch as the Institute students were carefully selected, full-fledged chemists and chemical engineers. Here, too, he could discuss his work freely with colleagues, and publish without too much restriction.

After 1938, Heuser really had a certain renaissance. The output of his writings increased, and so did his reputation. One of his first publications was with a talented colleague, John W. Green, on the behavior of cotton cellulose with ammonium oxalate and with cuprammonia solution, which showed that the original fiber was significantly depolymerized by this oxalate in air, and much less affected when the reaction was carried out under nitrogen. The degradations involved oxidation or hydrolysis, or both. A further conclusion was that the existence of Wanda Farr's hypothetical "cementing materials" in purified cellulose was improbable.

This work was followed by articles dealing with films from cellulose xanthate solutions, the action of ultraviolet light on cellulose and cellulose

triacetate, cross-linkages in cellulose and their practical applications, the acetylation of cellulose in phosphoric acid, the action of lignin-destroying fungi on the carbohydrates in wood, unsolved cellulose problems, the suitability of wood pulps for use in acetylation, a study of the degree of polymerization of cellulose in certain coniferous kraft pulps, trends in fundamental research in the field of cellulose and wood pulp; factors influencing the kinetics of cellulose reactions; and the degree of esterification of primary and secondary hydroxyl groups in cellulose by means of tosyl chloride. In this varied list of papers, he was buttressed by the work of various students and colleagues who were often joint authors in the publications. Among them were H. Y. Charbonnier, G. N. Chamberlin, W. Shockley, Merle Heath, J. A. Van den Akker, R. Kjellgren, B. F. Shema, John Appling, and J. F. McCoy.

With Leif Jørgensen, Heuser also published in *Tappi* a series of papers dealing with chain lengths and chain-length distribution in wood cellulose as compared with cotton cellulose; a comparison between fractional solution and fractional precipitation as applied to cellulosic materials; and the determination of the Staudinger constants of various cellulose nitrates. During this period, Heuser also wrote memoirs on Johann Rudolf Katz, Harold Hibbert, and Peter A. Paulson. Furthermore, he contributed a long chapter on cellulose to Henry Gilman's advanced treatise on Organic Chemistry.

However, his most important publication after coming to the United States was his final, classical work on the *Chemistry of Cellulose*, published in 1944 by John Wiley & Sons. This was far more ambitious than were the modest *Lehrbücher*, although many of the older, generally accepted aspects, re-inforced by ample experimental data, were maintained and expanded. Unlike the *Lehrbücher*, this work was written in English, and often Heuser spent hours in seeking just the right phrase to pinpoint the ideas that he had in mind. He insisted on indexing the book himself, and, although he graciously acknowledged the help of colleagues, the monograph, in contrast to others in the same field, was essentially the product of one man, for Heuser was a rugged individualist.

In this book, he emphasized the importance of the microscopic and the submicroscopic structure of fibrous high polymers. The reactions of cellulose with water, aqueous alkalis, organic bases, ammonia, and strong salt solutions were all stressed. Special attention was given to various types of cellulose esters, to cellulose xanthate, and to the cellulose ethers. The oxidation of cellulose under a variety of conditions was described, as were the hydrolysis reactions. The latter included discussions on reversion and on the kinetics of acid hydrolysis. It is interesting to note that Heuser, who earlier had criticized the terms hydrocellulose and oxycellulose, and had



even quoted Goethe (who decried the use of mere *words* in place of authentic *ideas*), now used these terms himself. After he had once clearly shown their limitations and the pitfalls resulting from their careless use, he found them convenient for purposes of brevity.

In his final monograph, Heuser gave much thought to products intermediate in the hydrolysis and acetolysis of cellulose. A section was devoted to the cellodextrins, the oligosaccharides containing (1  $\rightarrow$  4)-linked D-glucose units, and to cellobiose. The importance of D-glucose in its relationship to cellulose was also made clear. The thermal decomposition of cellulose, its hydrogenation, and its decomposition by micro-organisms were also outlined.

In 1944, the essential chain structure of cellulose was fully known and the data leading to this formulation of its constitution were reviewed by Heuser. Thus, much of the uncritical material in the first *Lehrbuch* could be either deleted or relegated to a subordinate position. Methods for determining the degree of polymerization of cellulose were also given in adequate detail. Throughout the work, Heuser apparently still maintained the position that the molecular architecture of celluloses, irrespective of source, was essentially the same. Later, however, in a section written for Volume I of *Pulp and Paper Manufacture* (McGraw-Hill, 1950) he admitted that sugar units other than those of D-glucose might be present, even in a highly purified wood cellulose.

In 1947, Heuser retired from The Institute of Paper Chemistry with the title of "Group Leader in Cellulose Chemistry, Emeritus," and with the honorary degree of Master of Arts from Lawrence College. He left Appleton and settled in a charming home in La Jolla, California; but this retirement did not spell inactivity. He worked assiduously on a possible revision of his book, which had gone through its second printing within two years after publication. He attended various meetings, including that celebrating the Seventy-fifth Anniversary of the American Chemical Society. He gave lectures in San Francisco, New Orleans, and San Diego.

In 1948, he received the coveted TAPPI medal, the highest award given by the Technical Association of the Pulp and Paper Industry, and, in 1952, he received the Alexander-Mitscherlich Commemorative Medal given for outstanding service by the German Verein der Zellstoff und Papier-Chemiker und -Ingenieure, of which he was an honorary member.

Thus, the twilight of his life was an active and a pleasant one. To commemorate his seventieth birthday, in 1952, his former student, colleague, and life-long friend, Professor Dr. Georg Jayme wrote a sensitive and illuminating biographical sketch (with a complete bibliography) in the September issue of *Das Papier*, on which the present author has drawn repeatedly for many of the earlier incidents of Heuser's productive life.

This particular issue was dedicated entirely to Emil Heuser, and contained articles by a number of his former students, including Richard Bartunek, Walter Brecht, August Brötz, Julius Funke, and Karl Hegel.

No memoir on Emil Heuser would be complete without reference to the man himself. He was a cultured European, an aristocrat with an ingrained love for music, drama, poetry, and for all forms of beauty. His wife, affectionately known as "Friedel," was an unusually talented painter. Both of the Heusers enjoyed gracious living; they were loyal in their friendships, and their delightful home in Appleton was always open to their many friends. With these, to whom Heuser was known as "Milus," he enjoyed discussions quite apart from the chemistry of cellulose. In many ways, he himself was an artist. Politically he was a liberal, with strong views, who was shocked by and deeply ashamed of the murderous desecrations of the Nazi regime, and he was glad to become an American citizen.

Milus had a continental and rather sardonic sense of humor. In his final trip into Washington and Oregon, lecturing three hours each day, he seemed as buoyant as ever, but, when this was remarked upon, he said, wryly, "After last examining me, my doctor said 'Heuser, I'm surprised that, with your *heart*, you are still alive'." Unfortunately, the medico's prognosis was all too accurate. On Christmas Eve of that same year, Emil Heuser died quietly in his sleep.

LOUIS E. WISE



