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HAROLD HIBBERT

1877—1945

A Biographical Memoir by
MELVILLE L. WOLFROM

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Biographical Memoir

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Harold Hibbert

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August 27, 1877—May 13, 1945

BY MELVILLE L. WOLFROM

HAROLD HIBBERT was born in the textile center of Manchester, England, on August 27, 1877, to Martha (Scholes) and Isaac Hibbert, both of Manchester, as the second in a family of four boys. His father held a responsible position with a Manchester company engaged in the export of cotton goods, mainly to South America. Isaac Hibbert (1844-1914) was active in the United Methodist Church of Manchester and was a staunch and devoted Liberal; he was a teetotaler, a nonsmoker, played the violin, had a good tenor voice, and was able to converse in Spanish. Harold was raised in the stimulating atmosphere of this fine home and his talents were early recognized when at the age of seventeen he was awarded a Manchester Corporation Scholarship which enabled him to enter the Victoria University of Manchester. This university, now generally known as Manchester University, was chartered in 1880 and had developed from the privately endowed Owens College, opened in 1851. Harold elected the Honours course and graduated in 1897 with First Class Honours in Chemistry. William H. Perkin, Jr., then near his fortieth year, was Professor of Chemistry at Manchester and was attracting many research students. Hibbert was awarded the fellowship known as the Levinstein Exhibition for research in organic chemistry and received the M. Sc. degree under Perkin in 1900. This contact with Professor Perkin, an outstanding investigator in organic chemistry, greatly stimulated the young man, and Perkin was the

first of three great leaders in this discipline to exert a marked influence upon Hibbert's career.

Hibbert accepted an appointment, in 1899, as Instructor in Chemistry at the University College of Wales, Aberystwyth. In support of his student, Perkin had written: "I have no hesitation in expressing the opinion that Hibbert has every prospect of a very successful career before him." North Wales is in many ways more closely related to Lancashire and the Midlands than to South Wales; its university was opened in 1872 and chartered in 1892. In 1901 J. J. Sudborough was appointed Professor of Chemistry, and Hibbert's first publications, as joint author with Sudborough, were concerned mainly with applications of the Tschugaeff (Zerewitinoff) reaction, then quite new. Hibbert remained in this post until 1904, when he left for further study at the University of Leipzig.

He arrived in Leipzig in October, 1904, and worked under Arthur Hantzsch, who had just succeeded Johannes Wislicenus. The research outlook of Hantzsch was mainly physicochemical in nature and Hibbert's work with him concerned addition products of trialkyl derivatives of phosphines, arsines, and stibines. The starting materials were prepared by the Grignard reaction rather than by the use of zinc alkyls. The University of Leipzig awarded Hibbert the Ph. D. degree *summa cum laude* in 1906. Professor Hantzsch was a scholar and philosopher and his lectures in organic chemistry, accompanied by numerous experiments, were highly inspirational. Hibbert's natural charm and cordial manner and his sincere respect for his professor brought him into intimate contact with Hantzsch and his family, a distinction allotted to but few other students. Hantzsch was an ideal host, well versed in art, poetry, and music. At Leipzig, Hibbert became acquainted with a small group of American students who later became distinguished chemists in their homeland—W. C. Bray, Colin G. Fink, Arthur B. Lamb, and S. C. Lind. Hibbert valued and maintained their friendship, and in later years he was to have the honor of presenting the Perkin Medal of the American Section of the Society of Chemical Industry to Colin G. Fink, distinguished for his

researches in the electroplating of chromium. While in Germany, Hibbert learned to speak the German language with an idiomatic ease that remained with him throughout his life. Memories of this period must have been vivid to him during the celebrated "Gemütlicher Abend" of the 1938 Milwaukee Meeting of the American Chemical Society. Here we find him surrounded by his own students, singing heartily the German songs of his student days.

Following the Leipzig period, Hibbert accepted a postdoctoral appointment in the United States with Arthur Michael at Tufts College of Boston, Massachusetts, and remained there for two years, 1906 to 1908. The vigorous personality of Michael made a powerful impression upon him and greatly influenced the subsequent course of his career. Hibbert worked with Michael on keto-enol tautomerism and studied especially the role of the solvent in this equilibrium. It is of interest to note that the discussion of their published work occupies a full page in the monograph of F. Henrich, *Theories of Organic Chemistry*, published in 1921.

Hibbert returned to England in 1908 and researched for a little over a year at the Imperial College of Science in London, under a Chemical Society grant and in association with Sir William Tilden. Here he completed work begun previously at Aberystwyth and concerned with some quantitative applications of the Grignard reaction now known at the Tschugaeff-Zerewitinoff method. The usefulness of a high-boiling ether in this reaction had been demonstrated by Sudborough and Hibbert in 1904, and they were the first to employ the procedure in a quantitative fashion. Hibbert presented some of this work at the February 18, 1909, meeting of the Chemical Society in London, and introduced the term "affinergy" as a substitute for Michael's "chemical potential," a proposal vigorously criticized by Arthur Lapworth and Sir William Ramsay in the subsequent discussion.

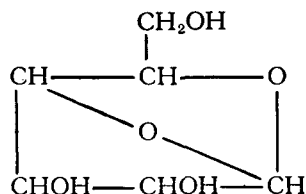
The English degree of D. Sc. corresponds to the Dr. Habil. (Doktor Habilitation) of the German universities, and the D. Sc. degree was conferred upon Hibbert in 1912 by the Victoria University of

Manchester. Disappointed in his efforts to obtain an academic appointment in Great Britain, Harold Hibbert returned to the United States and entered the chemical industry of this country. During the period from 1910 to 1914 he was a research chemist at the Experimental Station of the E. I. du Pont de Nemours Powder Co. at Wilmington, Delaware. At the time this great company was essentially a manufacturer of explosives and Hibbert published important work on the practical and theoretical significance of the dimorphism of glycerol trinitrate (nitroglycerin).

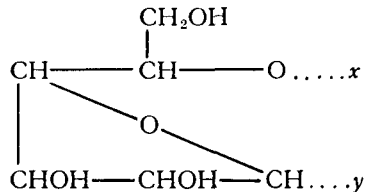
In 1914, after the outbreak of war in Europe, Hibbert became a Fellow of the Mellon Institute for Industrial Research at Pittsburgh, Pennsylvania. He worked on the synthesis of glycols and on new methods for the synthesis of acetaldehyde and acetic acid from acetylene. He published his famous procedure for dehydrating alcohols with a trace of iodine and presented a rational explanation for it. In association with Benjamin T. Brooks, he published a method for the synthesis of a "fusel oil" from petroleum. The large outlet for ethylene glycol as an antifreeze material in automobile cooling systems received its start in 1917, when Hibbert obtained United States Patent 1,213,368 relating to the use of ethylene glycol for lowering the freezing point of water in automobile cooling systems; he demonstrated the suitability of ethylene glycol dinitrate as a liquid explosive. In 1916 Hibbert left the Mellon Institute to enter private consulting work in Toronto and later in New York City. He was Chemical Adviser (on gas warfare) to the British War Mission in Washington, D. C., during 1917 and 1918. He served as Director of the Research and Technical Division of Ralph L. Fuller and Co., New York, N. Y., a company organized for the manufacture of such wartime-needed pharmaceuticals as salicylic acid and phenacetin. During this period he published a number of articles on general industrial chemistry, including a significantly prophetic one on the need for more attention to the aliphatics on the part of the organic chemical industry.

In 1917 he had married Beulah Virginia Cole, then a teacher of

physiography in the Julia Richman High School of New York City, and much of his success was due to her guidance and inspiration. By the end of the First World War, and at the age of forty-two, he had the courage to change abruptly his plan of life. He abandoned the more lucrative career of an industrial chemist and in 1919 accepted an appointment as Assistant Professor of Chemistry at Yale University. Here he initiated his series of publications entitled "Studies on Reactions Relating to Carbohydrates and Polysaccharides." Utilizing the finding of Denham and Woodhouse that methylated cellulose yields 2,3,6-tri-*O*-methyl-*D*-glucose on hydrolysis, Hibbert proposed the following formula for the cellulose monomer.



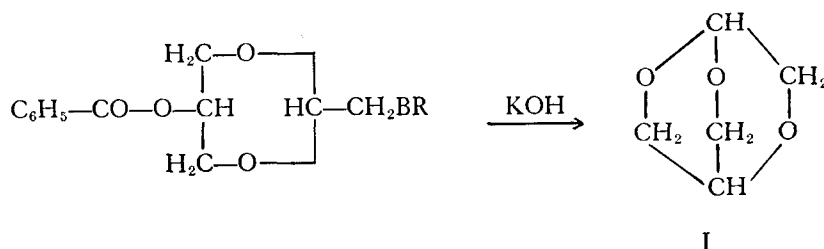
In accord with the viewpoint of the time, the polymerization could occur by association through secondary valence forces, but Hibbert discussed, and did not exclude, the possibility that the monomers might form a main valence polymer by an opening of the 1,5-oxygen bridges as shown,



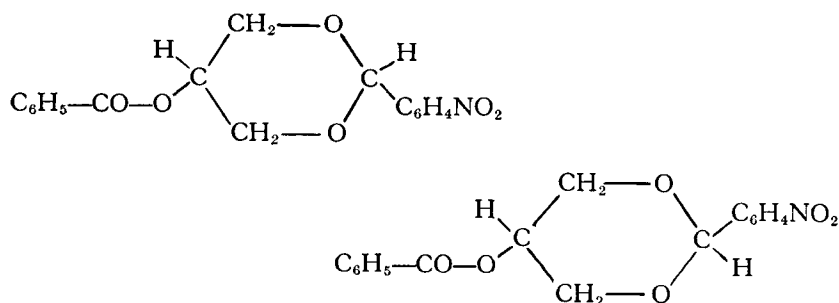
x and y being similar monomers.

He next attempted to synthesize analogs of the monomeric unit of the above structure from simple substances. While he and his students were unsuccessful in this endeavor, they did unearth a very considerable body of information concerning the formation and reactivity of cyclic acetals. It was found that 5-hexanone-1,2-diol

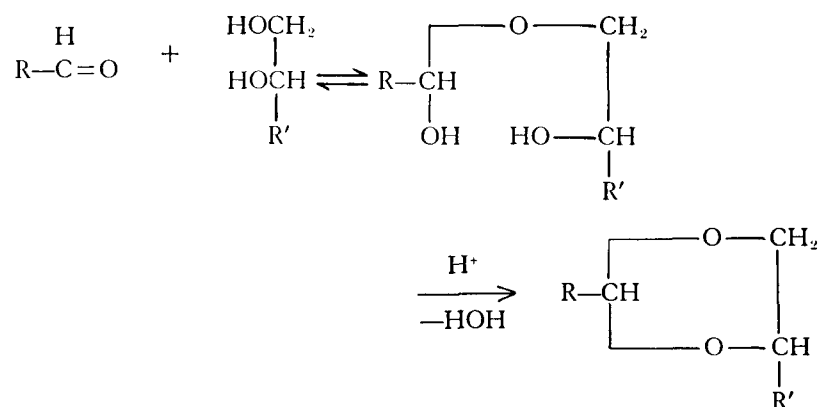
polymerized in the presence of a trace of acid, and this change was considered analogous to the suggested transformation of D-glucose into cellulose. Chloral was shown to combine readily with simple aliphatic aldehydes to form polymers of the general type $(2RCHO + Cl_3CCHO)_x$. Treatment of 1,3-O-(2-bromoethylidene)-glycerol benzoate with alkali yielded the crystalline bicyclic acetal I.



The reaction between glycerol and benzaldehyde was shown to result in a partition between five- and six-membered cyclic acetals, thus eliminating the necessity of vicinal hydroxyl groups for condensation. In the case of the corresponding six-membered *p*-nitrobenzylidene acetals, an equilibrium ratio of 0.4:1, favoring the five-membered isomer, was obtained under defined acidic conditions. In alkaline media, on the other hand, no tendency toward ring shift was observed, thus indicating the reliable nature of the methylation process employed in the proof of sugar glycosidic ring structures. 1,3- (and 1,2)-O-(*p*-Nitrobenzylidene)-glycerol 2- (and 3)-benzoate were each obtained in the two isomeric forms predictable on stereochemical grounds.

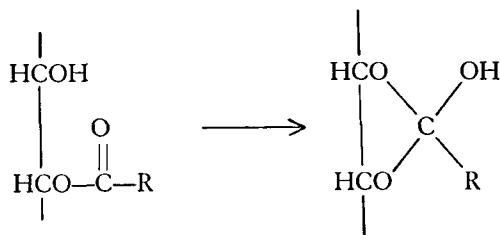


The reaction of acetone with the polyhydroxy compounds employed by Hibbert and his associates always resulted in the exclusive formation of the five-membered cyclic acetal. The course of all these condensations was shown to be stepwise, with the intermediate product being isolated in several cases.

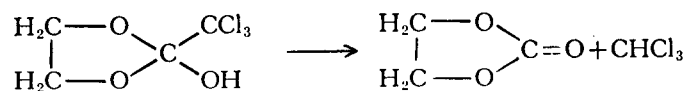


The ease with which the initial reaction took place was a function of the polar character of the carbonyl group, increasing with halogen substitution in the R-group. The ease of ring formation, on the other hand, was found to follow the reverse order, as did the ratio of the six- to the five-membered cyclic acetals.

Significant work was carried out on the nature of acyl group migration. Following a suggestion of Emil Fischer, Hibbert considered that the orthoester or dioxolane ring structure was the intermediate.



He showed that in glycerol esters the migration was toward the primary hydroxyl group. He stated, "The tendency and ease of ring formation will be dependent on: (a) the relatively labile character of the hydrogen attached to the hydroxyl group, (b) the negative polarity of the carbonyl group in the acyl radical, and (c) the spatial relationship of the migratory hydrogen atom with reference to the carbonyl group." His prediction that the orthoester structure would be stabilized by the trichloroacetyl radical was verified by the finding that the trichloroacetate of ethylene glycol existed only in the cyclic form; when this substance was heated, it decomposed into chloroform and the carbonate.



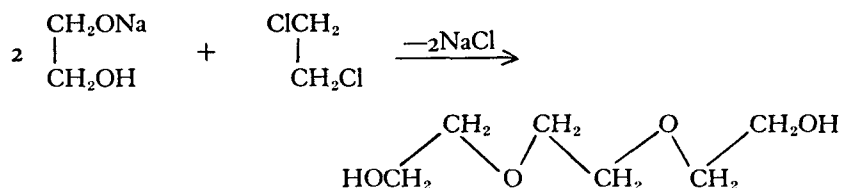
On the other hand, the methyl ethers showed no tendency to migrate and the reaction $\text{RCl} \rightarrow \text{ROME}$ was effected without migration. These early studies on "neighboring group effects" are outstanding contributions to the science of organic chemistry and to the specific interpretation of carbohydrate reactions.

Harold Hibbert was the impelling force in the founding, in 1920, of the Division of Cellulose Chemistry of the American Chemical Society; his associates in this effort were G. J. Esselen, Jr., H. LeB. Gray, L. F. Hawley, Bjarne Johnsen, and L. E. Wise. Hibbert served as Chairman of the Division from 1920 to 1922. One of its first activities was the establishment of procedures to define a Standard Cellulose preparation. Hibbert was always to be found in attendance at the meetings of the Cellulose Division, where he acted as a vigorous and stimulating leader of the discussions. Later he was joined in

this endeavor by Emil Heuser. The meetings of the Cellulose Division were never dull in those times.

In 1925 Hibbert left Yale University to become the E. B. Eddy Professor of Industrial and Cellulose Chemistry at McGill University, Montreal, Quebec, Canada, and he remained there for eighteen years until his official retirement in 1943. He had become a citizen of the United States in 1923 and he maintained this status during the remainder of his life, even though most of it was spent in Canada. McGill University, where the Pulp and Paper Research Institute of Canada had just been completed, offered an unparalleled opportunity for research, which he embraced with great vigor. Hibbert possessed an abundance of energy and an enormous capacity for work. He contributed greatly to the establishment of graduate scientific study in the universities of Canada both by his own efforts and by those of the men he trained. He was an inspiring taskmaster to the more than one hundred predoctoral and postdoctoral students who came to him from all parts of the Dominion and from other countries. He taught them to work hard and to like it. His enthusiasm and vision gave life and meaning to many an otherwise tedious investigation. His own valuable library was always available to them; he planned with them for their future; he never rested until he had found a place in industry, a grant—often out of his own pocket—or a teaching position for each of “his boys” (and several “girls”). Mrs. Hibbert felt an equal concern for the welfare of the students.

After Wallace H. Carothers and Hermann Staudinger had established the nature of polymers on the firm basis of primary valence concepts, Hibbert and his associates made a definite contribution by synthesizing a series of individual linear polymers of known chain-length and studying their properties as a function of this linear size. The fundamental reaction employed is illustrated below.



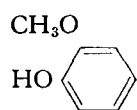
By this method there was prepared the series members with degrees of polymerization of 4, 6, 8, 12, 18, 42, 90, and 186 units.

He studied the preparation and properties of the polymeric "humic acid" formed by the action of mineral acids on hexoses. The product for a time found use as an extender in lead storage batteries.

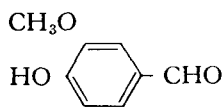
Hibbert was intrigued by the polysaccharides elaborated by the slime-producing bacteria, and he and his associates carried out pioneer studies on their chemical nature. The membrane grown by *Acetobacter xylinum* was shown to be cellulosic, and at the same time the general chemical identity of wood cellulose and cotton cellulose was demonstrated. Pioneer investigations were carried out on the nature of the glucan produced by various strains of *Leuconostoc mesenteroides*. The main linkage present was shown to be α -D-(1 \rightarrow 6). These investigations were later considerably elaborated in other laboratories when this material became of interest as a blood extender. The bacterial fructans known as the levans were shown to be isomeric with inulin in that the linkage present between the D-fructofuranose units was (2 \rightarrow 6) instead of the (2 \rightarrow 1) of inulin. Considerable attention was given to the nature of the carbohydrates utilizable by the organisms concerned.

Wood chemistry was naturally a major concern of the Pulp and Paper Research Institute of Canada, and Hibbert studied, with his associates, the action of alkali on cellulose and the complex reactions concerned in the oxidation of cellulose. An intensive study was made of the pulp bleaching process and of the manufacture of vanillin from pulping waste liquors. Professor Hibbert, however, placed his main emphasis upon lignin, publishing eighty-seven papers in the series entitled "Studies on Lignin." The Hibbert laboratory ex-

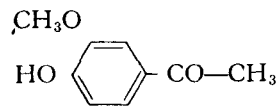
amined the action of alkalis on lignin sulfonic acids and showed that the soft woods (such as spruce) were characterized by the lignin alkaline degradation products, I, II, and III and the hard woods (such as maple) by the analogous products IV, V, and VI.



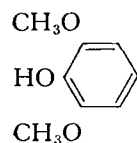
I
Guaicol



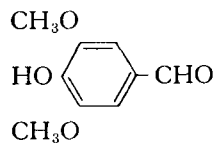
II
Vanillin



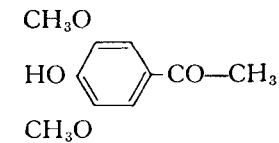
III
Acetovanillin



IV
Pyrogallol 1,3-
dimethyl ether

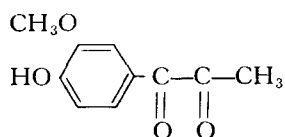


V
Syringaldehyde

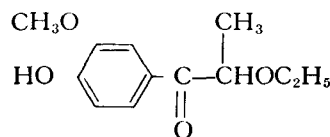


VI
Acetosyringone

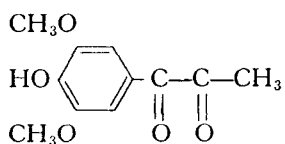
They also investigated the water-soluble fraction obtained in the extraction of lignins (such as maple ethanol lignin) from wood meal (previously extracted to remove fats, waxes, resins, and tannins) by the action of alcohols in the presence of acids. They identified the products VII to X.



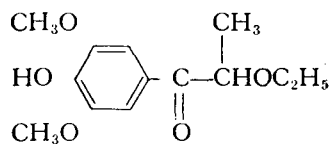
VII
Vanilloyl methyl ketone



VIII
 α -Ethoxypropiovanillone



IX
Syringoyl methyl ketone



X
 α -Ethoxypropiosyringone

Substances VIII and X were considered to be ethyl ethers formed in the ethanolysis reaction from the hydroxyl compounds originally present; VIII and X are derivatives of the class of compounds known as benzoin which are characterized by their ease of polymerization. The products VII to X establish the $C_6-C-C-C$ building units of lignin and have come to be known as the Hibbert *Bausteine* (building stones). How they are combined in the complex lignin polymer is as yet unsettled.

Harold Hibbert survived his retirement by only two years, and died of cancer of the pancreas in a Philadelphia hospital on May 13, 1945, after a lingering illness. His personal library, comprising some 1,500 volumes, has been kept intact and as the Hibbert Library is housed in the research laboratory of the Crown Zellerbach Corporation of Camas, Washington. He is survived by his widow, currently resident in New Haven, Connecticut. They had no children. He is memorialized at McGill University by the Harold Hibbert Memorial Fellowship, established by his younger brother Ernest (1879–1948) with an endowment of \$100,000. This brother, a British-trained mining engineer, had been concerned with the technical development of the great Noranda gold and copper strike in the wilds of northwestern Quebec. The youngest brother Arthur (1883–1947) was likewise a mining engineer of high repute, who distinguished himself in the British Corps of Engineers in the First World War. The eldest brother Frank (1875—) is still engaged in manufacture in London. Harold is further memorialized by the two "Harold Hibbert Memorial Prizes" awarded annually in the Chemistry Department of the University of Manchester for the two best theses presented during the previous year.

Harold Hibbert was the recipient of the LL. D. degree *honoris causa* from the University of British Columbia in 1936; he became an Honorary Member of the Society of Chemical Industry (London) in 1943 and was elected to the National Academy of Sciences (U. S. A.) in 1945. He was a Fellow of the Royal Society of Canada. He served on the editorial board of *Cellulosechemie*. In addition,

he was a member or fellow of the following societies: American Association for the Advancement of Science; American Chemical Society (Chairman, New Haven Section, 1920, and Division of Cellulose Chemistry, 1920–1922); American Pulp and Paper Association; Canadian Chemical Association; Canadian Pulp and Paper Association; Deutsche chemische Gesellschaft; Royal Institute of Chemistry (London); Sigma Xi; Society of Chemical Industry (Chairman, Montreal Section, 1930); Technical Association of the Pulp and Paper Industry; and Textile Institute (England).

Hibbert's was a colorful and vivid personality. He was a tall and strikingly handsome man. He was high-spirited, always optimistic, and a hard worker. His skill in debate and critique will ever be remembered by those who attended with him the meetings of the American Chemical Society. He was a true scholar and had read widely in many fields for pleasure, profit, and relaxation. His excellent background in English literature enabled him to write and to speak with clarity and forcefulness. He loved to recite from memory the verses of the great English poets. He was the possessor of a fine baritone voice and enjoyed singing airs from *The Messiah* or the rollicking songs of old England. He loved the great outdoors and, with Mrs. Hibbert, was an enthusiastic watcher of bird life. A vivid personality is gone, but his scientific attainments will long survive in his publications and in the students he so markedly inspired.

For valued assistance in preparing this memoir I am indebted to many of the former associates of Professor Hibbert, and especially to Mrs. Harold Hibbert, Dr. E. V. White, Professor C. B. Purves, and Mr. J. D. Crum.

KEY TO ABBREVIATIONS

- Am. Dyestuff Repr. = American Dyestuff Reporter
Ann. = Justus Liebigs Annalen der Chemie
Arch. Biochem. = Archives of Biochemistry
Ber. = Berichte der deutschen chemischen Gesellschaft
Can. J. Research = Canadian Journal of Research
Chem. & Met. Eng. = Chemical and Metallurgical Engineering
Ind. Eng. Chem. = Industrial and Engineering Chemistry
Ind. Eng. Chem. Anal. Ed. = Industrial and Engineering Chemistry, Analytical Edition
J. Am. Chem. Soc. = Journal of the American Chemical Society
J. Chem. Soc. = Journal of the Chemical Society (London)
J. Ind. Eng. Chem. = The Journal of Industrial and Engineering Chemistry
J. Soc. Chem. Ind. (London) = Journal of the Society of Chemical Industry (London)
Paper Trade J. = Paper Trade Journal
Proc. Chem. Soc. = Proceedings of the Chemical Society (London)
Pulp Paper Can. = Pulp and Paper of Canada
Pulp Paper Mag. Can. = Pulp and Paper Magazine of Canada
Z. ges. Schiess- u. Sprengstoffw. = Zeitschrift für das gesamte Schiess- und Sprengstoffwesen

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