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WALTER ABRAHAM JACOBS

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A Biographical Memoir by

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Walter C. Jacobs

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BY ROBERT C. ELDERFIELD

WALTER ABRAHAM JACOBS died in Los Angeles after a long and impressive career. He left behind some 273 publications which record important contributions in the field of the chemistry of natural products of biological importance, as well as the development of chemotherapeutic agents—the significance of which was not recognized until some years had passed.

Jacobs was born in New York City on December 24, 1883 and attended local elementary schools. He received an A.B. degree in 1904 and an A.M. in 1905 from Columbia University, after which he enrolled at the University of Berlin for study under Emil Fischer, earning a Ph.D. degree in 1907.

On his return to New York, Jacobs received an appointment as a fellow in chemistry in the laboratory of Phoebus A. Levene at the newly established Rockefeller Institute for Medical Research. In 1908 he became an assistant, and in 1910 an associate in Levene's laboratory. During these years with Levene, he was closely associated with the latter's work on the chemistry of the nucleic acids.

The studies, first with the nucleotide inosinic acid from beef extract, disclosed its essential chemistry by hydrolysis to the nucleoside, inosine, and by subsequent cleavage from the latter of its crystalline sugar component which was identified

as *D*-ribose. This became the pattern for similar studies with the nucleotide guanylic acid, and with yeast nucleic acid (ribonucleic acid), from which the various nucleosides were then isolated and interpreted. The attempted extension of this procedure to similar studies with thymus nucleic acid (subsequently shown to be desoxyribosenucleic acid) was interrupted by Jacobs' promotion in 1912 to associate member of the Institute with independent status.

Dr. Simon Flexner, Director of the Institute, felt that the developing field of chemotherapy warranted a division of its own, and Jacobs was placed in charge of it. In collaboration with Michael Heidelberger, he began an investigation of the possible chemotherapy of polio. It was known that hexamethylenetetramine apparently exerted a slight therapeutic effect, and an extended series of quaternary salts was prepared by reaction with aromatic and alipathic halogen compounds. Some of the salts displayed bactericidal properties, and a few appeared to prolong the life of polio-infected monkeys. Unfortunately, this was due to a loss of virulence of the virus strain.

After the disappointing outcome of the chemotherapeutic studies concerning polio, the Jacobs–Heidelberger team turned its attention to African sleeping sickness, for which no effective and non-toxic drugs were available. Ehrlich had produced a powerful synthetic agent against trypanosomiasis in *para*-arsenophenylglycine, in which the arsenic was trivalent. The analogous pentavalent substance, *para*-phenylglycine arsonic acid, was considerably less toxic, but devoid of activity against the disease. Jacobs reasoned that the lack of activity could be due to the free carboxyl group which conceivably could react with many centers of the tissue proteins before reaching the parasites. He therefore proposed masking the carboxyl group by conversion to the amide. The resulting substance, sodium *para*-phenylglycine amide ar-

sonate, was the first, simplest, and best arsenical of a series subsequently synthesized.

The new arsenical, named Tryparsamide by Simon Flexner, was found to be extremely effective in trypanosome infected animals by Drs. Wade H. Brown and Louise Pearce, who now formed part of the chemotherapeutic team. Several patents were awarded for control of the drug and several of its analogs—although none of the latter proved to be superior to Tryparsamide. At the conclusion of World War I, Louise Pearce made an extensive study of the drug in the Belgian Congo, which showed Tryparsamide to be more effective than previously used drugs. Further tests in the United States demonstrated some utility in the treatment of tertiary syphilis.

Some years later (1953), Belgium recognized the successes of Tryparsamide by making Drs. Jacobs, Heidelberger, Brown and Pearce officers of the Order of Leopold II.

During World War I, a portion of the Institute was designated as U.S. Laboratory #1, and served as a training facility in laboratory techniques for army physicians. Jacobs and Heidelberger investigated possible synthetic substitutes for Salvarsan, a drug in scant supply and of undesirable toxicity. One analog (arsenophenyl-glycine-*bis*-*m*-hydroxyanilide), which appeared to be less toxic and at least as active against syphilis when studied by Brown and Pearce in animals, showed promising results in some one hundred human syphilitics. Unfortunately, a second batch, for no apparent reason, caused severe, dangerous dermatitis and was abandoned.

At the conclusion of the work on arsenicals, Jacobs and Heidelberger desired to turn to fields other than synthetic organic chemistry, but Flexner's faith in this approach prevailed and attention was turned to pneumococcal and streptococcal infections. The drug, Optochin, had been used with

partial success in the treatment of pneumococcal infections, so further modification of the cinchona alkaloids was investigated, and a long series of papers resulted. Unfortunately, most of these substances killed infected mice faster than drug or infection alone. The state of this area of chemistry in the United States at the time is reflected in the refusal of the *Journal of the American Chemical Society* to publish the work on the cinchona alkaloids—on the grounds that no one in America was interested in alkaloids. Only after long arguments were the manuscripts accepted.

One of the intermediates used in modification of the alkaloids and in other syntheses was *p*-aminobenzenesulfonamide, or sulfanilamide, which had been prepared in 1908 by Gelmo in Germany. This was shown by Trefouel, Trefouel, Nitti, and Bovet to be one of the metabolic products of the azo dye Prontosil, the antibiotic action of which was demonstrated in the same year (1935) by Gerhardt Domagk for which he was awarded the Nobel Prize. Actually, it developed that the antibiotic action of Prontosil was due to the sufanilamide liberated on metabolism of Prontosil. It apparently never occurred to Jacobs and Heidelberger in 1920 that such a simple substance could control bacterial infections by other than direct antibacterial action. If the antibiotic action had been recognized, many thousands of lives could have been saved in the intervening years.

After some nine and one-half years, the team of Jacobs and Heidelberger separated. Flexner agreed that chemotherapeutic research through synthetic methods could be abandoned, and Heidelberger was transferred to the new laboratory of Donald D. Van Slyke to become familiar with biochemistry. Jacobs became a full member of the Rockefeller Institute in 1923, and turned his attention to the elucidation of the structures of substances of natural origin which displayed powerful physiological actions in order to correlate

structure with such activity. The name of the laboratory was changed to that of chemical pharmacology.

The first group explored was that known as the cardiac glycosides, noted for their specific and powerful action on the myocardium, and which are unrivaled in value for the treatment of congestive heart failure. Of the group, the glycosides from digitalis species are probably the best known. Extracts of other plants containing members of the group such as strophanthus species were used as arrow poisons by African tribes. The ancient Egyptians were familiar with the properties of squill, and the Romans used it as an emetic, heart tonic, diuretic and rat poison. Strophanthus was introduced into modern medicine in 1890, and the modern use of digitalis dates from 1785, when William Withering published his famous book entitled, *An Account of the Foxglove and Some of Its Medicinal Uses: With Practical Remarks on Dropsy and Other Diseases*.

At the time Jacobs began his investigations on the structures of these important compounds, little was known of them or their chemistry. It was known that they were glycosides consisting of a rather complicated aglycone moiety, which was responsible for their major pharmacological properties, joined with one or more sugar molecules. The digitalis glycosides are present in the plant in extremely small amounts, whereas the seeds of *Strophanthus kombé* are relatively rich in the glycosides of strophanthidin. Jacobs' plan of attack on the structures of the aglycones was to place major emphasis on the structure determination of the more accessible strophanthidin and to attempt a correlation of this structure with the digitalis aglycones and others by chemical interconversions of appropriate derivatives. This plan proved to be completely successful and the structures of a half dozen or so of the aglycones were demonstrated.

It should be noted that when the study of the cardiac

aglycones was begun (1923), similar studies on cholesterol and the bile acids by Windaus (1903), Diels (1903) and Wieland (1912) in Germany were also under way. There was no reason to believe that the three groups of substances would ultimately be found to be closely related. Also, with the possible exception of ultraviolet spectroscopy, none of the instrumental methods, such as infrared spectroscopy, X-ray spectroscopy and nuclear magnetic resonance, were available. Chemical transformations and degradation with subsequent interpretation formed the basis for structural elucidation—a long and tedious process at best.

Conversion of a strophanthidin derivative to a periplogenin derivative, and correlation of the latter with derivatives of digitoxigenin and gitoxigenin from digitalis followed. Although many structural features of the four aglycones were known, the problem of the carbon skeleton remained unsolved. In 1927, Diels and his co-workers heated cholesterol with selenium, thereby dehydrogenating it to its basic carbon ring system, cyclopentanophenanthrene. Similar dehydrogenation of strophanthidin also yielded Diels' hydrocarbon, thus providing conclusive evidence that the basic ring system of the cardiac aglycones was, indeed, identical with that of cholesterol and the bile acids. With the aid of X-ray data, the latter basic ring system was shown to be that of the Diels' hydrocarbon, a perhydrocyclopentanophenanthrene, by British workers.

The problem of the location of the unsaturated lactone side chain on the nucleus of the aglycones was resolved by application of the Barbier-Wieland degradation, used successfully in degradation of the side chain of cholanic acid, to a derivative of digitoxigenin with the formation of etiocholanic acid as the final product. Almost simultaneously, R. Tschesche in Germany accomplished a similar degradation

of another aglycone, uzarigenin, to allo-etiocholanic, the difference being in the stereo configuration of the lactone side chain at the 17-position of the nucleus.

Although all available evidence up to the actual Barbier-Wieland degradation appeared to involve the 17-position as the point of attachment of the side chain, Jacobs, a most meticulous planner of experiments, was loath to attempt the degradation of the side chain. A suitable derivative of the available strophanthidin was not available, and only a few hundred milligrams of an appropriate digitoxigenin derivative were available for the three-step degradation and characterization of the product. Jacobs was hesitant to commit this hard to obtain substance to a degradation—the outcome of which, despite its logical prediction, was not certain.

It so happened that the necessity for this decision arose early in the fall of 1934. Jacobs and his wife habitually took long Columbus Day weekends to admire the fall foliage of the Adirondack Mountains—and this author took the opportunity to commit the entire supply of digitoxigenin derivative to the degradation, with considerable trepidation. After three days and nights, pure samples of etiocholanic acid and its methyl and ethyl esters awaited Jacobs' return. Although the physical constants of all these agreed with the published data, Jacobs, after some hesitation, agreed to request authentic samples from Wieland in Munich for comparison. All compounds were identical with the samples, and this author heaved a sigh of relief.

With one minor revision, involving the position of the double bond in the unsaturated lactone side chain, the structure of the cardiac aglycones was established.

During his investigations of the cardiac aglycones, Jacobs began structural studies of the saponin group. These are plant glycosides that possess the distinctive property of form-

ing a soapy lather in water. The plant heart drugs also display this property, but are classified separately because of their distinctive physiological heart action.

Early emphasis was placed on the readily available sarsasapogenin from *Smilax ornata* Hooker. The aglycone occurs as a glycoside with two rhamnose and one glucose units. The empirical formula of the aglycone was revised to the now accepted $C_{27}H_{44}O_3$. Reinvestigation of the selenium dehydrogenation of sarsasapogenin resulted in the isolation of Diels' hydrocarbon, thus establishing that the sapogenins possess the steroid ring system. The presence of a C_8 side chain was indicated by the isolation of a ketone $C_8H_{16}O$, which was not identical with methyl isohexyl ketone from cholesterol. A partial structure for sarsasapogenin was suggested in 1935.

By this time, great emphasis had been placed on the cortical steroid hormones and their possible therapeutic applications. This resulted in a hectic search for accessible plant steroids which could be converted to cortical hormones. Several investigators, amply financed by the pharmaceutical industry, entered the field. Jacobs, with his small staff, was literally snowed under.

However, one further correlation was accomplished —that of sarsasapogenin with the representative steroid alkaloid, solanidine. By this correlation, the structure of solanidine was established and another member of the steroid group was recognized.

Beginning in 1932, an intensive study of the structures of the ergot alkaloids was undertaken in cooperation with the late Dr. Lyman C. Craig. Ergot is the product of a fungus which grows on grain, particularly on rye. Its effect on pregnancy has been known for 2,000 years and it was first used by physicians as an oxytocic agent some 400 years ago. Consumption of edible grain contaminated by the fungus has resulted in death and destruction for centuries, but it was not

recognized as the agent responsible for destructive epidemics until 1670. It was first employed by a physician about 1815, although it had been used by midwives long before.

At the time this investigation was undertaken, the chemistry of the ergot group was almost completely unknown. By 1934, on hydrolysis of the alkaloids, a substance named lysergic acid, which proved to be the characteristic building block of the ergot alkaloids, was isolated. The other products of hydrolysis of the alkaloids were amino acid derivatives joined by peptide linkages to themselves and to lysergic acid.

The structure of lysergic acid was then shown by degradation and substantiated by subsequent synthesis of the dihydro derivative and of lysergic acid itself. In this work an unnatural amino acid was encountered for the first time. Subsequently, the now famous L.S.D., the diethyl amide of lysergic acid, was synthesized by A. Hoffmann and Arthur Stoll in Switzerland.

The suspicion that such substances might be of more widespread occurrence formed the basis for a plan to extend such studies to other poisonous fungi such as *Amanita muscaria*. Although begun, this was interrupted by other work and has since been carried on in other laboratories. The ergot alkaloids may be regarded as the first group of a general pattern of such distorted polypeptides encountered in the gramicidins, penicillins and other antibiotics.

Another field of investigation entered by Dr. Jacobs was the chemistry of the aconite alkaloids, which embraced a large group of substances of undetermined structure when he began his inquiry in 1936. Some of these had long been used in medicine, and in some cases are among the most poisonous substances known. These studies included the known aconitine from *Aconitum napellus*, commonly known as monkshood or wolfsbane, and the isolation of three new alkaloids— heteratisine, hetisine and benzoylheteratisine—as

well as the known atisine from *Aconitum heterophyllum* Wall. Closely related were delphinine and staphisine from *Delphinium staphisagria*, similar in action to aconitine. Degradation procedures supplemented by syntheses were reported in some thirty-five communications, until the time of Jacobs' retirement in 1957.

The final group of natural products to which Jacobs turned his attention embraces a complex family now known as the steroid bases, or veratrum alkaloids, found in various *Veratrum* species. These fall into two classes, as suggested by Fieser and Fieser*: the jerveratrum alkamines and the ceveratrum alkamines which comprise cevine and its precursors.

Of the first group, Jacobs and co-workers established that rubijervine is a hydroxyl derivative of the known solanidine by conversion to the latter substance. Assignment of the hydroxyl group to the 12-position was established by infrared and rotatory dispersion data. A structure for veratramine was also proposed, which was essentially correct except for one minor detail.

As with the saponins, interest in jervine increased with recognition of the possibility that it could serve as a starting material for the synthesis of cortisone, and it attracted extensive attention from industrial laboratories. In 1949, Jacobs had tentatively suggested a structure for jervine based on available data for this complex molecule at that time. This was shown to be in error in some respects, although in general it reflected the data then available. The accepted structure was eventually proposed by a group at the Squibb Laboratories.

The ceveratrum alkamines generally occur as esters of various acids. Four of the alkamine bases commonly found as such esters are veracevine, germine, protoverine and zygadenine. On alkaline hydrolysis, cevine, now recognized as an

*Louis F. Fieser and Mary Fieser, *Steroids* (New York: Reinhold Publishing Corp., 1959), 1118 pp.

artifact, was formed from all four. It appeared to be a logical candidate for structural studies in 1937, when Jacobs' long series of studies of these substances began. Selenium dehydrogenation yielded nine bases, five hydrocarbons and one tricyclic phenol. From study of these fragments and spectroscopic data, it was possible to formulate the ring system of cevine.

A second approach involved chromic acid oxidation of cevine which yielded a mixture of acids and lactams from which six pure acidic compounds were isolated by fractional separation of the methyl esters. Heating of the mixture of oxidation products at 200° resulted in the formation of decevinic acid. Data obtained with this substance resulted in corroboration of the structures assigned to other members of this family.

The investigations of Jacobs on the veratrum alkaloids continued for some twenty years until his retirement, and formed the basis for more extensive studies of these structurally very complicated molecules by other workers.

Walter Jacobs was a very highly regarded member of the Rockefeller Institute for some fifty years. His modesty, shyness and lack of aggressive tendencies kept even his colleagues at the Institute from realizing his innate abilities. He showed extraordinary judgment of the correctness and reliability of a chemical procedure, and an amazing ability to make most efficient use of time, especially in starting something new when another reaction had to be left for some hours. He insisted on complete and repeated experimental verification before publication of any result, a conservatism which in certain instances resulted in loss of priority.

He was not only an excellent and original organic chemist but also a very kind person, and most considerate of his fellow workers. He was fortunate in having married Laura Dreyfoos in 1908. She was the ideal understanding and sup-

portive wife for a retiring and dedicated scientist. A warm, outgoing, and capable woman, she furthered in unobtrusive ways the communication of his personality to friends. They often entertained his younger associates in their home in Mount Vernon. He specialized in very temperamental renditions of Beethoven on the Pianola, and in directing recordings of Wagner's Ring operas.

The Jacobs had two children, Elizabeth and Walter, Jr.

He was granted emeritus status at the Rockefeller in 1949, but continued active laboratory work until 1957, when he retired and migrated to Los Angeles.

HONORS AND DISTINCTIONS

AWARDS

Belgian Order of Leopold II, 1953

PROFESSIONAL AND HONORARY SOCIETIES

National Academy of Sciences (elected, 1932)
American Association for the Advancement of Science
American Chemical Society
American Society of Biological Chemists
American Society of Pharmacology and Experimental Therapeutics
Harvey Society

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