

OBITUARY NOTICE.

FREDERIC STANLEY KIPPING.*

1863—1949.

FREDERIC STANLEY KIPPING was born on August 16th, 1863, at Higher Broughton, Manchester. He was the eldest son of James Stanley and Julia Kipping and had two brothers and four sisters. His father held a post in the Manchester branch of the Bank of England of which his paternal grandfather was head. His grandfather on his mother's side was C. A. Duval, a Manchester artist of considerable reputation.

Kipping early acquired that interest in open-air life which was never to leave him. His father owned a small farm near Higher Broughton, which was at that time practically in the open country. Here he made himself generally useful and gained a fair knowledge of animals and practical agriculture. In the early days the Kipping children had a governess but at the age of eleven Frederic Stanley entered Manchester Grammar School on the same day as Herbert Brereton Baker. It is remarkable that both these boys became Longstaff Medallists of the Chemical Society. He had already become interested in chemistry through his father's friend and neighbour, J. Carter Bell, the public analyst for Cheshire, who showed him various simple precipitation reactions in inorganic chemistry, and advised his father to make him an analytical chemist. No doubt at this time that honourable profession was still generally regarded from the Dickensian point of view, so clearly indicated in "Our Mutual Friend."

Kipping remarks that the practical chemistry at Manchester Grammar School consisted almost entirely of qualitative analysis but unaccompanied by any clear explanation of its principles. During the holidays he assisted Mr. Carter Bell with the ordinary routine analysis of water, milk, and other foodstuffs. He also spent a year at the Lycée de Caen where food and sanitary arrangements were vile but the teaching of Latin was specially good. Kipping matriculated at the University of London in 1879 in the First Division and thereupon entered Owens College, Manchester, his family having meanwhile removed to Platt Abbey, a house familiar to this day to all those who travel on a 42' bus along the Palatine Road to the Burlington Street laboratories. After three years, in which he studied mathematics, physics, botany, chemistry, and zoology, he graduated B.Sc. (London) with Second Class Honours in Zoology. He found Miles Marshall, the professor of Zoology, an excellent and inspiring teacher and Roscoe extremely kind and interested in his students. Schorlemmer, who was the first Professor of Organic Chemistry in this country, did not attract him however—his lectures were read without emphasis or pause from notes held close to the nose. After a short experience of this mode of presentation of organic chemistry Kipping devoted Schorlemmer's lecture hours to the practice of billiards in an adjacent hotel, thus acquiring a facility which he never lost. On the whole, with the exception of zoology, he found college work dull and unsatisfactory, but he used the five courts a good deal and played cricket and lacrosse.

After graduation as an External student of the University of London in 1882 he obtained a post as chemist to the Manchester Gas Department in Rochdale Road and later at Gaythorne Street. The duties of the post, except for occasional work throughout the night, were not heavy but afforded him very useful and varied analytical experience which, at the age of 19, may have laid the foundation of the scrupulous care and accuracy of his research work. He had plenty of time for reading and was frequently able to leave the laboratory at 4.30—5.0 p.m. to catch a train on the former M.S.J. and A. Railway to Old Trafford in order to play tennis at the Northern Lawn Tennis Club. Here and on many other grounds he played in matches against the leading clubs of Lancashire and adjoining counties. He remained in the service of the Corporation for about three years to the age of 23. Until then he seems to have shown no particular signs of devotion to chemistry although he was doubtless maturing. About this time, however, a friend of his family, Dr. Adolf Liebman, a chemist at Levinstein's, Blackley, strongly advised a period of study in Germany and pointed out the lack of opportunity for advancement in Kipping's post at the gas works.

Following this advice, he entered the University of Munich in the Spring of 1886 and was there given a bench in one of the organic laboratories which was supervised by W. H. Perkin, jun., who had entered von Baeyer's Institute in 1882. There he carried out various organic preparations and after some months commenced research under Perkin's guidance on the

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synthesis of closed carbon chains. Thus began a scientific association which was to last until the death of Perkin in 1926 and was destined to influence the lives of both men, not only on the scientific but also on the personal side. Late in 1886 Perkin returned to England and thenceforward Kipping continued his research unaided except for the written advice which Perkin sent him from time to time. At that time Claisen, Bamberger, and von Pechmann were Privat-dozenten and Nef was a student. Work in the laboratory began at 8 and continued till 6 or after, with a rather long interval for Mittagessen—and chess. There was much skating in the winter. Kipping saw very little of von Baeyer. He refers to one occasion, however, on which he showed him with some pride a product he had isolated. The Geheimrath looked at it under a lens, snapped out “ Ach, Harz ” and stalked away! This may have been the origin of Kipping’s critical and, to say the least, realistic attitude to his students’ laboratory efforts. However, he took his Ph.D. degree *summa cum laude*, after which he returned to England and for a short time assisted his father, who had turned his attention to chemical manufacturing on the small scale. The preparations of sulphur chloride and of carbolic disinfecting powder in the stables of Platt Abbey were not very congenial or successful ventures and did not last very long.

In the autumn of 1887, T. E. Thorpe and Emerson Reynolds as Examiners for the University of London awarded Kipping the degree of D.Sc. He was the first candidate on whom this degree was conferred on the grounds of research. It would appear, however, that his Munich work sufficed for both his Doctorates, a happy state of affairs which would not now commend itself to Faculties of Science. Immediately afterwards he was offered the post of demonstrator under W. H. Perkin, jun., who had by now been appointed Professor of Chemistry at the Heriot-Watt College in Edinburgh. The remuneration was £20 a year “ with prospects.” Most of the teaching was done in the evening but Kipping was usually on duty for most of the day, spending much time in research, again under Perkin’s direction. In March 1888 he married his cousin, Lily Holland, the daughter of W. T. Holland, J.P., of Bridgwater and grand-daughter of C. A. Duval. During their engagement Perkin met his future wife, Miss Holland’s elder sister, at the house of Kipping’s parents.

During the Edinburgh period the writing of the famous “ Organic Chemistry ” (always known as “ Perkin and Kipping ”) was begun. This work was one of the earliest text-books on the subject and was first published in 1894. It has enjoyed an enormous reputation and has passed through many editions, the latest of which (1949) presents an excellent picture of the present-day position of the science. After about a year at the Edinburgh College Kipping was made Assistant Professor of Chemistry and Lecturer in Agricultural Chemistry at a total remuneration of £150 a year.

In late 1890 an event occurred which greatly influenced his future work and afforded him many stimulating contacts and the opportunity of directing the research of beginners. H. E. Armstrong, to Kipping’s “ astonishment and satisfaction,” offered him the post of chief demonstrator in the Chemistry Department at the Central Technical College of the City and Guilds of London Institute at £240 a year. (The College is now incorporated in the Imperial College of Science and Technology, and the Chemistry Department closed. Many will remember the heated discussion which took place about 1910 regarding the wisdom of this step, Armstrong having built up a most flourishing school of research.) Kipping accepted this offer “ with alacrity ” and he and his wife removed from Graham Street, Edinburgh, to a small house at 7, Milborne Grove, South Kensington. Kipping then worked with Armstrong for a time, first on certain problems connected with colour and constitution and then on the products of the action of sulphuric acid on camphor, one of which was shown to be 3 : 4-dimethylacetophenone (*p*-acetyl-*o*-xylene).

In 1893 Arthur Lapworth arrived in Armstrong’s laboratory from Birmingham, where his father was the distinguished Professor of Geology, and was placed under Kipping’s supervision. Here again a very successful collaboration resulted, the work being mainly on derivatives of camphor.

Lapworth became a frequent visitor at the house in South Kensington, where he often met Pope, Forster, and other workers in Armstrong’s laboratories. During one vacation when he had made no holiday plans, the Kipping’s asked him to stay with them at Bridgwater : here it was that he met his future wife, Kathleen Holland.

The work of Perkin, Kipping, and Lapworth has left a lasting impression on experimental and theoretical organic chemistry which may best be realised if we attempt for a moment to consider what the record of the years 1895—1930 would be if the work of these three men, who married three sisters, were left out of consideration.

Kipping found little time for recreation, but whenever possible he and his wife went for long rowing excursions on the Thames, returning only when the light faded. During six years they heard, from the gallery of Covent Garden Opera House, nearly all the great operas and most of the famous singers of the day.

In 1897 Kipping was elected to the Royal Society at the age of 34 and soon afterwards was appointed to the Chair of Chemistry at University College, Nottingham, in succession to Professor Frank Clowes. He found the laboratories and class-rooms in Shakespeare Street compact and well-arranged but there were only four full-time students who were working for the Associateship of the Institute of Chemistry; the remainder consisted of a few ill-prepared part-time students of Engineering. Most of the evening students were working for the qualifying examination of the Pharmaceutical Society. He refers to certain short courses given in those early days to students of mining, dyeing, plumbing, and brewing, and adds "I came to the conclusion that such so-called technical education was valueless. . . . the more intelligent students of these subjects were encouraged to attend the evening classes in pure chemistry during two or three years and their practical work was so arranged that it had a direct bearing on their daily occupations." The number of students of chemistry steadily increased and Kipping, first unaided and then in collaboration with members of his staff, initiated various lines of research which will be considered later. He also acted as external Examiner to the federal Victoria University and to the Royal University of Ireland, and much appreciated the kindness and hospitality of Professor Dixon of Cork and Professor Senior of Galway, but greatly regretted the necessity of failing so large a proportion of the Irish candidates. Both before and after the first World War he was external Examiner to the University of Birmingham and wrote in 1942: "There the standard was high and the unfailing kindness of Professor and Mrs. Frankland with whom I stayed remains a most pleasant memory even now."

In the years before the war Kipping "enjoyed the thrills of learning golf and also played a good deal of tennis at the Nottingham Park Club." He usually played golf at Hollinwell (under the shadow of Byron's "hills of Annesley, bleak and barren") with Professor Ernest Weekley, the well-known French scholar, on Wednesdays from about eleven to three or four o'clock. This habit was much appreciated by his students who then felt reasonably secure from his severe and searching interrogations. Golf and tennis, however, were not Kipping's only recreations. Mrs. Kipping and he found much pleasure in visits to theatres and concerts and in dancing. Mrs. Kipping was interested in social work and was for many years closely associated with the Nottingham Society for the Care of the Feeble-minded. Thus it came about that on one occasion the staff of the Chemistry Department at Birmingham were somewhat surprised to receive the draft questions for the B.Sc. examination from their External Examiner on the official note-paper of this Society! Satisfactory explanations, were however forthcoming!

At this time many holidays were spent walking in Switzerland and one visit was made to the Italian lakes. Professor and Mrs. Lapworth were their frequent holiday companions, especially in later years at Tenby.

During the first World War Kipping and his staff collaborated in the scheme for the preparation of synthetic drugs which were at that time unobtainable in Britain; he also lectured on musketry and map-reading to the Officers' Training Corps and superintended practice on a miniature rifle range in an old clay-pit.

Like all other Chemistry Departments of University rank, Kipping's laboratories were crowded after 1918 and it became necessary to find immediate temporary accommodation for the forty or fifty full-time students who wished to work for degrees of the University of London. The Shakespeare Street buildings of the College were situated between the City Library on the one side and the City Museum on the other, forming one large block which was and is a familiar and pleasing aspect of Nottingham. To meet the demand, one of the large rooms of the museum was cleared of its stuffed animals and converted into a laboratory for the more elementary and part-time students. This met the immediate needs but after 1922 it became obvious that the student population of the College would remain permanently at a much higher figure than before the war.

The College Council thereupon decided to build a new University College. Fortunately, this was made possible through the great generosity of Sir Jesse Boot, later Baron Trent, who not only purchased the splendid site now known as University Park but also defrayed the cost of the building and equipment and endowed the Chair of Chemistry, Kipping thus becoming the first Sir Jesse Boot Professor of Chemistry.

The College was opened by H.M. King George V in 1928. The dignity of the building, both

externally and internally, and the beauty of its setting are the envy of many older and richer academic bodies. The impact of a second World War on British University life has now created a need for still further expansion both of class rooms and of laboratories. Fortunately, the spacious surroundings of the new University of Nottingham afford ample opportunity for this. Kipping had the satisfaction of entering into occupation of the new Chemistry building in 1928, but for many years most of the old Department in Shakespeare Street was used for evening classes and for some day-time work. Since 1945 the building has been taken over by the City as the Nottingham Technical College, but former students will find little difference in the appearance of the laboratories, and Kipping's old research room where he did all his crystallisations and his own combustions is still the research laboratory of the Staff.

In 1936, after the College Council had, on several occasions, refused to accept his offers to resign, he retired from the Chair and was appointed Emeritus Professor of Chemistry. The Council made arrangements for him to work in his old laboratory at Shakespeare Street and there he continued research on silicon compounds for three years longer. Shortly after his retirement, the University of Leeds conferred upon him the degree of D.Sc. *Honoris causa*, in recognition of his long services to University teaching and research.

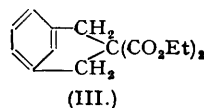
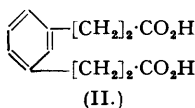
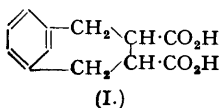
In the same year, soon after Kipping published Part L of "Organic Derivatives of Silicon," his staff and former students entertained him to dinner. Robison, the discoverer of the mechanism of the calcification of cartilage, was in the chair and over seventy were present. It was a most happy and inspiring occasion; the respect and affection in which Kipping was held were manifest in every word which was spoken. The dry humorous remarks, the caustic comments, the rather gruff encouragements stored for many years in the memories of his pupils were then released in the speeches. Happy is the man who can inspire such regard.

In 1941 he published Part LI which was unique in one respect. At the end it contained these words. "*Envoi*. As this is my last paper may I express here my sincere thanks to the Chemical Society for having published so much of my work over so long a period—F. S. K." I do not recall a similar acknowledgment by any other Fellow of the Society. Kipping's retirement was also marked by the invitation to deliver the Bakerian Lecture of the Royal Society. The subject was "Organic Derivatives of Silicon" and the lecture took place in London on a very hot summer day in 1936. Kipping afterwards expressed his deep sympathy with those who had assembled to hear it. It was, however, characteristically brief and concise even when printed, and his friends could have wished it had been longer.

On the outbreak of war in 1939 Kipping, with his wife and younger daughter, took a house on the sea-front at Criccieth and there it was my privilege to see him in 1946 and 1947. He was surrounded by the proofs of the revised, almost rewritten, edition of "Perkin and Kipping" in the production of which he had been joined by his younger son, Frederic Barry, University Lecturer in Chemistry in the University of Cambridge. He was, it need hardly be mentioned, as interested as ever in the progress of chemical research.

In the spring of 1949 the health of Mrs. Kipping caused him much anxiety, and after only a week's illness he died on May 1st in his eighty-sixth year. It was a sad, but not wholly unfitting, coincidence that the unexpected news of his death reached me in the new buildings of the University of Nottingham on the May morning of the inauguration of its first Chancellor. Some months earlier he had learned with deep satisfaction of the granting of a University Charter to the College which he had served so faithfully. His labours and those of his colleagues, Barton, Heaton, and Swinnerton, to mention only a few, had, however, secured to the College University standing and repute 40 years previously. His death removed one of the last links with what may be termed the later classical era of organic chemistry in Britain.

Kipping's first publication (*J.*, 1888, 53, 21) was entitled "Synthetical Formation of Closed Carbon-Chains in the Aromatic Series. Part II" and described the work which was carried



out in von Baeyer's laboratory at Munich at the suggestion and originally under the direction of W. H. Perkin, jun. It follows a paper by Perkin in which he summarises his earlier work in the Munich laboratory and describes the synthesis of the tetrahydronaphthalene and hydrindene (indane) rings. In this paper Kipping records experiments on the action of *m*- and *p*-xylylene bromides, $C_6H_4(CH_2Br)_2$, on ethyl sodiochloromalonate in the hope of obtaining

7- and 8-membered rings, on the analogy of Perkin's work with *o*-xylylene bromide. The desired compound (I) was, however, not obtained, but rather (II); the *p*-xylylene bromide derivative behaved in a similar manner with iodine.

It will be remembered, in passing, that von Baeyer's Strain Theory was partly built upon the experimental results of W. H. Perkin in which Kipping was so greatly interested.

Kipping also attempted to obtain the tetrahydro-*meta*-naphthalene derivative (III) from *m*-xylylene bromide, ethyl malonate, and two molecular proportions of sodium ethoxide, but only a resin was produced. Furthermore, he showed that no anhydride was obtainable from (II) or from the corresponding *p*-derivative.

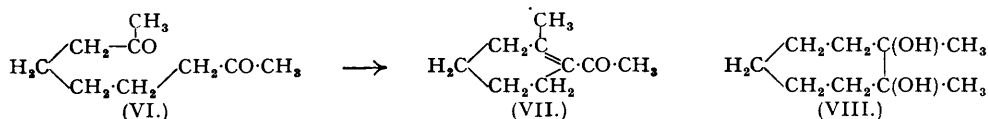
These results are what we should now expect from such reactions, but 62 years ago it was necessary to establish the facts by experiment. Here it may be mentioned that on one occasion the vapours of xylylene bromide entered von Baeyer's "Dienstwohnung" which adjoined the laboratory. He gave Kipping a "dressing down."

It is of interest to recall that, eleven years later in 1901 in collaboration with Hunter, Kipping described two 7-membered ring compounds, which he named pheno- α -ketoheptamethylene and pheno- α -aminoheptamethylene, by ring closure of the acid chloride of phenyl-*n*-valeric acid and reduction of the oxime of the ketone. The presence of the ring of seven carbon atoms was shown by the formation of phthalic acid on oxidation of the ketone by dilute nitric acid. This "internal" Friedel-Crafts reaction was also used by Kipping in the preparation of keto-tetrahydronaphthalene and of hydrindone (IV) (now indanone) and its 2-methyl derivative (V) and, as will be seen later, played an essential part in the development of much of his work.



In the next two or three years Kipping, in collaboration with Perkin, devoted much attention to the preparation of ketones such as 1 : 4-diacetylbutane, 1 : 5-diacetylpentane, and 1 : 5-dibenzoylpentane by means of the ethyl acetoacetate synthesis. On the analogy of the formation of mesityl oxide and of pinacol from acetone, it appeared possible that dehydration and reduction of such diketones might be confined to one molecule giving rise to an unsaturated cyclic ketone and a cyclic pinacol respectively.

By dissolving 1 : 5-diacetylpentane (VI) in sulphuric acid, the expected product (VII) was obtained. The suggestion by Kipping and Perkin (see above) that reduction of 1 : 5-diacetylpentane might give rise to an "internal" pinacol was upheld shortly afterwards when they showed that with sodium and moist ether dimethyldihydroxyheptamethylene (VIII) was produced.



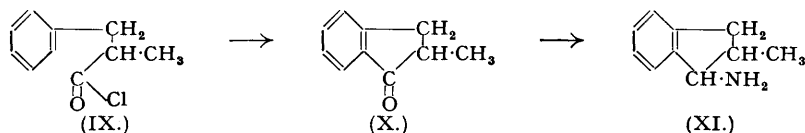
We must now refer briefly to some work of Kipping's which, apart from his Munich Ph.D. Thesis (*J.*, 1888, 53, 21), was the first which he published independently. Between 1870 and 1893 in three papers he described the preparation of the higher dialkyl ketones by the distillation of the corresponding fatty acids with phosphoric oxide. Using this method, he prepared dihexyl and dioctyl ketones and several homologues. The research was only of preparative importance, but it was significant because so much of his later work in different fields may be regarded as a direct consequence. Thus, in an unsuccessful attempt to prepare dibenzylacetone, $(C_6H_5 \cdot CH_2 \cdot CH_2)_2CO$ he heated β -phenylpropionic acid with phosphoric oxide. The products were found to be the hydrocarbon "truxene," $(C_9H_8)_2$, and a small quantity of the cyclic ketone hydrindone (IV). The importance of this work lies, however, not in its relation to truxene but because Kipping was led to prepare a considerable quantity of α -hydrindone (*J.*, 1894, 65, 480) by the self-condensation of β -phenylpropionyl chloride in ligroin solution in presence of aluminium chloride.

Three years later Revis and Kipping (*J.*, 1897, 71, 239) continued the study of hydrindone and its condensation products and reduced its oxime with sodium amalgam in dilute acetic acid to hydrindamine, an experiment which was fated to influence much of his future work (see pp. 854, 855). Later, Kipping and Hall (*J.*, 1900, 77, 467) obtained indene by distillation of

hydrindamine hydrochloride and of trimethylhydrindonium iodide and showed that the product was identical with that obtained by other methods.

A paper (*J.*, 1901, **79**, 602), which cannot be summarised here, is important because it contains a discussion of the significance of the terms "partially racemic" and "racemic."

It is instructive to notice how Kipping's interest in cyclic systems kept pace with his growing attraction to stereochemical problems. An instance of this, and perhaps the most important on account of its influence on his future work on optically active compounds of silicon, is to be found in his synthesis of (\pm)-methylhydrindamine (XI) carried out with Clarke (*J.*, 1903, **83**, 913) by reduction of the oxime of methylhydrindone (X) obtained by cyclisation of α -benzylpropionyl chloride (IX).



The base contains two asymmetric carbon atoms and therefore exists in two (\pm)-forms which were studied by Tattersall and Kipping (*J.*, 1903, **83**, 918). Crystallisation of their salts with optically inactive acids and of their benzoyl derivatives and platinichlorides failed to effect a complete separation, although the existence of the two (\pm)-forms, which were designated *dl*-methylhydrindamine and *dl-neo*-methylhydrindamine, was easily demonstrated. Separation was finally effected by crystallisation of the (+)-bromocamphorsulphonates from water, without resolution of either base. The resolution of the two partially racemic [(\pm)-B, (+)-A] salts so obtained was then attempted. On crystallisation from boiling ethyl acetate the (\pm)-*neo*-methylhydrindamine salt was not resolved, but (\pm)-methylhydrindamine (+)-bromocamphorsulphonate finally yielded its two components, and pure (+)- and pure (−)-methylhydrindamine hydrochlorides were obtained. The (+)-bromocamphorsulphonates of the (+)- and the (−)-methylhydrindamines as separated by ethyl acetate were each shown to be mixtures of two isomeric salts; this was later found to be due to the existence of two stereoisomeric *cis*- and *trans*-modifications of bromocamphorsulphonic acid. References to this phenomenon, which was exhaustively studied by Kipping in connection with (\pm)-hydrindamine and (\pm)-menthylamine are given in *J.*, 1905, **87**, 636. Kipping's resolution of (\pm)-hydrindamine is described in *J.*, 1903, **83**, 873, 889.

So far we have considered the development of one main trend of Kipping's work—the investigation of cyclic compounds leading, as we have seen, to a serious interest in stereochemical problems, in the study of which the halogeno-camphorsulphonic acids played a large part.

Kipping's interest in derivatives of camphor was due to his association with Armstrong at the Central Technical College (see p. 850). He states that "Armstrong then asked me to examine a crude sulphonation product of bromocamphor prepared by W. J. Pope; thus began the joint research work which extended over six years during which many joint papers were published." The first paper described the sulphonation of camphor and bromo-camphor with anhydrosulphuric acid or chlorosulphonic acid. "The optically inactive, or feebly active, mixture of sulphonic chlorides prepared from the acid obtained by either of these methods shows a very remarkable behaviour; it is deposited from solution in well-defined crystals which, although apparently homogeneous, are simply mixtures of the two active compounds in variable proportions and are partially resolved into enantiomorphous forms on recrystallisation."

"The sulphonic derivatives of bromo- and chloro-camphor are remarkable for the extraordinary facility with which they crystallise; the sulphonic chlorides, the amides, the acids and a number of salts are described, the optical and crystallographical characters of many of the compounds being given." Such details appeared in many subsequent papers published by Kipping, both in collaboration with Pope and with other workers. The importance attached to crystallography by other pupils of Armstrong, *e.g.*, Lowry, is well known.

The authors add "Speaking generally, the sulphonic derivatives of camphor are compounds of exceptional beauty" (here, surely, we detect the Perkin touch) "and judging from the results thus far obtained will probably lead to conclusions of considerable importance." When we consider the part played by bromocamphorsulphonic acid in almost every study of optically active bases during the last 50 years, this prophecy may be regarded as entirely justified.

In two further communications Kipping and Pope showed that the preliminary effervescence observed during the combustion of sulphonic halides of camphor and its halogen derivatives

is due to evolution of sulphur dioxide and formation of the so-called π -halogen derivatives (so named owing to their pyrogenic formation), in which the atom thus introduced replaces the $-\text{SO}_2\text{Hal}$ group in the $\text{CH}_3\cdot\overset{\text{C}}{\text{---}}\text{CH}_2\cdot\text{SO}_2\text{Hal}$ residue of camphor. Thus bromocamphorsulphonic chloride yields α -bromo- π -chlorocamphor.

Many derivatives of this type were obtained and, in several papers published in collaboration with Lapworth, their properties and those of other derivatives of camphor, especially nitrocamphor and camphoric acid, were carefully examined. Lowry's work on bromocamphor, mutarotation, and similar themes was also an outcome of the assiduous cultivation of the camphor field in South Kensington at the turn of the century.

Reference may here be made to two stereochemical investigations in one of which Kipping and Salway approached the vexed question of the "Arrangement in Space of the Groups combined with the Tervalent Nitrogen Atom" and the other, where, with his student, Peters, he investigated "Iodonium Compounds of the Type $\text{IR}'\text{R}''\text{R}'''$ " and the Configuration of the Iodine Atom" (*J.*, 1902, **81**, 1350). The work on the trivalent nitrogen atom may conveniently be summarised by quoting from the *Annual Reports* of the Chemical Society (1904, **1**, 144): "Kipping and Salway refer to the discrepancies between the theory of Hantzsch and Werner . . . and the failure of previous attempts to resolve bases in which three different groups are attached to trivalent nitrogen; they point out that the latter experiments are indecisive owing to the possibility of a change in the valency directions attending the conversion of ter- into quinque-valent nitrogen and *vice versa*. They treated *d*-hydrindamine, *l*-methylhydrindamine, *l*-menthylamine and *l*-phenylethylamine with *d*-benzylmethylacetyl chloride and in each case obtained but one acetyl derivative of the primary or secondary base; if the three nitrogen valency directions do not lie in one plane the production of stereoisomerides would be anticipated. . . . It is pointed out that on treating an optically inactive base with an externally compensated acid chloride, only one acidic derivative (*i.e.*, the substituted acid amide) should be formed if the original base is potentially inactive, whilst two isomerides are capable of formation if the base is externally compensated.

"Thus *dl*-hydrindamine yields two isomeric acidic derivatives with *dl*-benzylmethylacetyl chloride, whilst methylaniline, *p*-toluidine, benzylaniline and phenylhydrazine yield but one product in each case. . . . It is therefore concluded that the three valency directions of trivalent nitrogen lie in the same plane."

In the same paper it was shown that (\pm) -hydrindamine and (\pm) - α -phenylethylamine may be resolved by the aid of $(+)$ -benzylmethylacetyl chloride, but in neither case was it possible to regenerate the optically active bases by hydrolysis of the substituted amides.

These discussions of 45 years ago are interesting in view of Prelog and Wieland's recent claim (*Helv. Chim. Acta*, 1944, **27**, 1127; *Brit. Abs.*, 1945, *A, II*, 100) that Tröger's base (obtained from *p*-toluidine and formaldehyde and synthesised by an unambiguous method; Spielman, *J. Amer. Chem. Soc.*, 1935, **57**, 58) can be resolved by chromatographic adsorption on activated lactose hydrate.

The work on iodonium compounds consisted in the fractional crystallisation of phenyl-*p*-tolyliodonium $(+)$ -bromocamphorsulphonate. No evidence of resolution could be obtained.

Kipping's work on organic derivatives of silicon was commenced with the object of obtaining compounds of which the optical activity was due to the presence of an asymmetric silicon atom and clearly arose out of his work on the stereochemistry of nitrogen. This, in its turn, was a consequence of his still earlier interest in camphor. It might be imagined that the discovery of the Grignard reagent was a determining factor in Kipping's choice of this field of research; but although the progress of his work was greatly eased by the use of these reagents (*J.*, 1907, **91**, 209), the research was commenced one or two years before 1900 (the year in which Grignard made his initial observation) and in Part I Kipping and Lloyd (*J.*, 1901, **79**, 449) make no reference to the French chemist's discovery.

Kipping's work in this field falls under three main headings: (1) experiments connected with the preparation and resolution of asymmetric compounds of silicon, (2) work on the structure of the simpler condensation products derived from compounds of the type $\text{R}_2\text{Si}(\text{OH})_2$ and $\text{RSi}(\text{OH})_3$, and (3) the study of the products obtained from arylchlorosilanes, R_2SiCl_2 or R_3SiCl , and sodium. Of these three main lines, only the first attracted great attention at the time. Since 1940 the second line of work has been greatly extended by the Dow Corning Corporation, who have placed numerous polymers of the composition $(\text{R}_2\text{SiO})_x$, etc., on the market.

Kipping certainly spoke truly when, in 1912, he remarked to a student who had used the term "polymerisation" loosely and rather carelessly: "polymerisation, that's a blessed word,

it's like Mesopotamia." The personal application of this comment was not apparent for nearly 30 years. His former students and colleagues welcome the utilisation of his silicols and silicones but deny with much warmth that this publicity adds anything to his scientific reputation, which was firmly established many years before the plastic age commenced.

The reactions employed by Kipping for the synthesis and, in three cases, resolution of his asymmetric compounds were simple in the extreme but the experimental difficulties were considerable. In Part I three chlorine atoms of silicon tetrachloride were successively replaced by the phenoxy-, methoxy-, and ethoxy-groups, but attempts at resolution by replacing the fourth chlorine atom by the menthoxy-group or by the anion of an optically active acid were unsuccessful. Five other asymmetric compounds of silicon were prepared between 1905 and 1910 by the successive replacement of the chlorine atoms with alkyl and aryl radicals by means of the Grignard reagent.

The sulphonic acids of (\pm)-benzylmethylpropylsilane and (\pm)-benzylmethylpropylisobutylsilane (then "silicanes") resisted all attempts at their resolution, although numerous optically active bases including cinchonidine, cinchonine, (–)-menthylamine, strychnine, brucine, and quinine were employed. Kipping's first success was obtained by sulphonation of (\pm)-phenylbenzylethylpropylsilane. When sulphuric acid is used at ordinary temperatures or below 70°, benzene is eliminated (but not toluene), probably with formation of $\text{SiEtPr}(\text{CH}_2\text{Ph})\cdot\text{OH}$ which then yields the disulphonic acid of the oxide, *i.e.*, $\text{O}[\text{SiPrEt}(\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H})]_2$. This elimination of benzene appears to be characteristic of the Ph–Si link under such conditions. Later, he used chlorosulphonic acid instead of sulphuric acid, obtaining the same sulphonic acid.

Kipping finally isolated a crystalline, deliquescent diammonium salt of this acid, of which he remarks in a footnote "This was the first crystalline silicon compound obtained during at least six months' work so the author has been careful not to over-rate its beauty." This acid was also prepared by the sulphonation of benzylethylpropylsilicol, obtained by the action of water on the corresponding chloride (Part II) and also (*J.*, 1908, 93, 198) by sulphonation of benzylethylpropylsilyl oxide, $\text{O}(\text{SiR}_1\text{R}_2\text{R}_3)_2$. Kipping then conclusively showed by numerous analyses of the (–)-menthylamine salt, that the sulphonic acid was a derivative of the oxide and not of the silicol. Attempts to resolve this acid by fractional crystallisation of the (–)-menthylamine, (+)-bornylamine, cinchonidine, cinchonidine hydrogen, and strychnine salts were unsuccessful, but with (+)-methylhydrindamine resolution was accomplished, though much care was needed in interpreting the results owing to the low specific rotation of the (+)- and the (–)-sulphonic acid. Thus on crystallising the (–)-methylhydrindamine salt of the (\pm)-acid, sparingly and readily soluble fractions, differing widely in m. p. but not in specific rotation, were obtained (Part II). Expulsion of the volatile base and conversion into the (–)-menthylamine salts gave products which did not differ appreciably in either m. p. or specific rotation. By suitable alternate use of the volatile (–)- and (+)-methylhydrindamines by methods which are obvious and need not be described in detail it was conclusively proved that resolution had occurred, the *dAdB*, *lAlB*, *dAlB*, and *lAdB* salts (where A and B represent acid and base), being very carefully compared. Finally, by removal of the volatile bases, two sodium salts of specific rotation +5.8° and –5.9° were obtained (Part VI; *J.*, 1908, 93, 457).

It is interesting to speculate as to whether, had it not been for the unexpected elimination of the phenyl group during the sulphonation of phenylbenzylethylpropylsilane, Kipping would ever have studied the sulphonation of an asymmetric silicol or oxide. Had he not done so, the negative results obtained with two sulphonated silanes suggest that success might never have been achieved. Still more remarkable is the coincidence that the only bases which were found capable of resolving the sulphonated silicyl oxides were (+)- and (–)-methylhydrindamine which he had himself prepared for the first time in the course of earlier work and submitted to a most exhaustive examination (see p. 854).

A year or two later, in collaboration with B. D. W. Luff, Kipping carried out a successful resolution of the disulphonic acid of (\pm)-benzylethylisobutylsilyl oxide, also by the aid of (+)- and (–)-methylhydrindamine, thus amplifying and confirming his earlier results. In this case the specific rotations of the sodium salts were larger, +10.4° and –10.5°.

Several years later Kipping prepared (\pm)-dibenzyl-diethyl-dipropyl-silicoethane and obtained brucine and (–)-menthylamine salts of the disulphonic acid. Separation of the racemic and *meso*-forms of the acid or the resolution of the first-named was not effected, owing to lack of material (Part XXIV; *J.*, 1921, 119, 647). With reference to this hydrocarbon and the corresponding diphenyl compound, Kipping writes "although their molecules contain linked

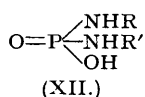
silicon atoms both silicohydrocarbons are attacked only very slowly if at all by boiling aqueous alkalis and even when they are warmed with sodium methoxide in methyl alcohol or acetone no rapid evolution of hydrogen is observed. The grouping >Si-Si< is not necessarily unstable to alkalis, and whether the silicon atoms become separated or not is determined by the nature of the atoms or groups with which they are combined." The behaviour of the >Si-Si< link towards alkali is mentioned on p. 858.

During the purification of dibenzylethylsilicon dichloride which was so important an intermediate compound in much of his work, Kipping isolated dibenzylethylsilyl chloride, $\text{SiClEt(CH}_2\text{Ph)}_2$. This was converted into dibenzylethylpropylsilane and sulphonated by means of chlorosulphonic acid in chloroform (Part XII; *J.*, 1910, **97**, 142). After separation of the mono- and di-sulphonic acids by means of their strychnine or (–)-menthylamine salts, the monosulphonic acid was successfully resolved by brucine but by no other base, although several were employed (see p. 859). Nothing could be more striking than the behaviour of the brucine salt on recrystallisation as compared with that of the other alkaloidal salts. The two end fractions of the brucine salt were then converted through the (–)-menthylamine salt into the sodium salt, of which only the (–)-isomer was obtained optically pure. Its specific rotation was certainly not greater than -1° . Concentrated solutions of the optically impure (+)-acid were, however, distinctly dextrorotatory, readings of about $+0.1^\circ$ being obtained (see p. 860). "These low values for the rotation and the fact that, although four pairs of salts (of the two acids) were carefully examined, in no case was there a difference in melting point or in specific rotation well outside the limit of the possible experimental error, might well have cast some doubt on the relationship of the two acids had it not been for the overwhelming evidence" (Part XIII; *J.*, 1910, **97**, 755). This evidence cannot be given in detail here, but was based on the maintenance of the properties of the two brucine salts on passage through other salts, followed by regeneration, on a considerable difference in solubility of the (–)-menthylamine salt of the (\pm)- and the (–)-acid, and a slight but distinct difference in m. p. Furthermore, numerous tests established the chemical identity of the resolution products and the absence of impurity. When Kipping described this work at a meeting of the Chemical Society in March 1910, he concluded by saying: "If you will do us the honour of reading our paper I think you will be convinced that we have established our conclusions."

Shortly afterwards, Kipping and T. A. Smith (*J.*, 1913, **103**, 2034) prepared the tin analogue of the silane, dibenzylethylpropylstannane, $\text{SnEtPr(CH}_2\text{Ph)}_2$, but the sulphonation of this and of dibenzyl-diethylstannane was impossible owing to the elimination of both the benzyl groups by the acid.

About this time Kipping returned to the study of a similar problem on which Caven (*J.*, 1902, **81**, 1362) had already carried out experiments in his laboratories—the stereochemistry of asymmetrical derivatives of phosphoric acid. Caven had been unable to resolve compounds of the type (XII). Kipping and Luff prepared phenyl *p*-tolyl hydrogen phosphate $\text{OP(OPh)(OC}_6\text{H}_4\text{)}\cdot\text{OH}$ and crystallised its salts with eight optically active bases without effecting resolution. Evidence that the acid was, in fact, a (\pm)-compound was obtained by the separation of the (\pm)-hydrindamide into two inactive compounds, the [*dAdB*], [*lAlB*] and the [*dAlB*], [*lAdB*] isomers (see p. 855). By crystallisation of the (+)-hydrindamide and (–)-menthylamide, two fractions differing in m. p. and specific rotation were obtained in each case, indicating a resolution of the (\pm)-acid. Similar results were obtained by Kipping and Challenger with the (–)-menthylamide of phenyl β -naphthyl hydrogen phosphate. In none of the cases was it possible to remove the optically active $\cdot\text{NHR}$ group by hydrolysis so as to obtain the corresponding acids. This stability appears to be characteristic of amides and substituted amides of this type. Over 30 years later, Davies and Mann (*J.*, 1944, 276) pointed out that these and similar experiments aiming at the isolation of the optically active acids were foredoomed to failure owing to resonance.

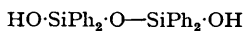
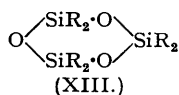
Kipping's investigations on the condensation and dehydration products of the dibenzyl- and diphenyl-silanedioles, $\text{R}_2\text{Si(OH)}_2$, were laborious and involved much detailed study of closely related substances and the manipulation of intractable oils, many of which were finally induced to crystallise. He realised from the beginning (see Part X; *J.*, 1909, **95**, 302) that the various so-called silicones, *e.g.*, SiOEt_2 , SiOPh_2 , and SiOPhEt , were associated and not simple molecules; this was, in fact, stated by Dilthey in the case of the diphenyl derivative in 1905. In his first paper with Robison (*J.*, 1908, **93**, 439) Kipping studied the loss of water from dibenzylsilanediol $\text{Si(CH}_2\text{Ph)(OH)}_2$, and in Parts XVIII and XX established that under various conditions the diol can yield trianhydrotris(dibenzylsilanediol) (XIII), dianhydro-



(XII.)

trisdimethylsilanediol $\text{HO}\cdot\text{SiR}_2\cdot\text{O}\cdot\text{SiR}_2\cdot\text{O}\cdot\text{SiR}_2\cdot\text{OH}$, and anhydrobisdimethylsilanediol $\text{HO}\cdot\text{SiR}_2\cdot\text{O}\cdot\text{SiR}_2\cdot\text{O}\cdot\text{OH}$, where $\text{R} = \text{CH}_2\cdot\text{C}_6\text{H}_5$. Heat or alcoholic hydrogen chloride, acetyl chloride, or alkali were employed as condensing agents.

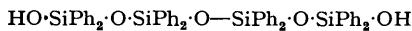
During this period a similar and even more extensive study of the condensation products of diphenylsilanediol was carried out, and in three papers (Parts XVI, XVII, and XXI, *J.*, 1912,



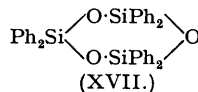
(XIV.)



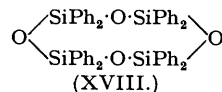
(XV.)



(XVI.)



(XVII.)



(XVIII.)

101, 2108, 2125; 1914, 105, 484) the existence of five crystalline condensation products was established, namely, (XIV)—(XVIII). Crystallographic measurements were carried out on four of these products.

When dichlorosilanes of the type $\text{RR}'\text{SiCl}_2$ are decomposed with cold aqueous ammonia, the corresponding diols, *e.g.*, benzyethylsilanediol and phenylethylsilanediol, $\text{RR}'\text{Si}(\text{OH})_2$, are obtained (Part XIX; *J.*, 1912, 101, 2156), and from the second of these the crystalline condensation product $\text{HO}\cdot\text{SiPhEt}\cdot\text{O}\cdot\text{SiPhEt}\cdot\text{OH}$ can be isolated. Kipping refers to the ease with which these two diols yield oily condensation products, and adds: "as the components of these mixtures have doubtless much lower melting points than the corresponding derivatives of diphenylsilanediol they cannot be isolated by the methods which were found to be of service in the case of the latter compounds." These substances containing alkyl groups foreshadowed the numerous polymers derived from $\text{Alkyl}_2\text{Si}(\text{OH})_2$ which have since found such important technical applications.

With his student Meads (*J.*, 1914, 105, 679; 1915, 107, 459) he also examined the condensation products obtained by the hydrolysis of phenyltrichlorosilanes which are derived from four, five, six, or more molecules of $\text{PhSi}(\text{OH})_3$. Here and elsewhere he drew attention to the possible analogy between the structure of these compounds and that of the silicates, and between $\text{HO}\cdot\text{Sn}(\text{C}_7\text{H}_7)_2\cdot\text{O}\cdot\text{Sn}(\text{C}_7\text{H}_7)_2\cdot\text{O}\cdot\text{Sn}(\text{C}_7\text{H}_7)_2\cdot\text{OH}$ and the polymeric stannic acids. He also found that $(\text{C}_7\text{H}_7)_3\text{Sn}\cdot\text{OH}$ is strongly associated in benzene solution (*J.*, 1913, 103, 2034).

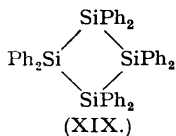
It is a pleasure to recall that the Dow Corning Corporation presented him in 1945 with a complete set of his 50 papers on Organic Derivatives of Silicon very clearly typed on strong paper and bound in boards made from a "silicone" polymer. These three volumes he presented to the Royal Society and they have been of great help in preparing this memoir.

No one who worked in the Nottingham laboratories between 1907 and 1910 will have forgotten the riddle of the constitution of the yellowish-white, amorphous, insoluble organic substances which are produced as by-products when any Grignard compound reacts with silicon tetrachloride. When all organo-silicon halides are removed from the magnesium salts with ether, and the salts dissolved in water, these compounds remain. Their most striking property is the rapid effervescence of hydrogen which takes place on addition of potassium hydroxide (see Part X). Kipping never published a full account of these substances though he worked on them from time to time. It soon became clear that they contained >Si-Si< links. The evolution of hydrogen seems analogous to that observed when silicoethane Si_2H_6 , silico-oxalic acid $\text{H}_2\text{Si}_2\text{O}_4$, silicoesoxalic acid and similar compounds are treated with alkali. For the ethyl compound, Martin and Kipping (*J.*, 1909, 95, 302) suggested the possible structure $\text{C}_2\text{H}_5\text{Si}(\text{OH})_2\cdot\text{SiO}_2\text{H}$. The formation of such compounds was particularly marked in the case of the cyclohexyl derivatives, volatile chlorides containing >Si-Si< links being detected (Part XLII, *J.*, 1930, 1020) and light thrown on their method of formation.

Later it was found that wet piperidine was a more satisfactory reagent for the fission of the >Si-Si< link and the hydrogen evolved—the "hydrogen number"—was frequently employed as evidence of structure (see Part XXVI, *J.*, 1921, 119, 848) although the reaction was not of universal application, a few compounds such as $\text{Si}_2(\text{C}_6\text{H}_5)_2$ and $\text{Si}_2(\text{C}_6\text{H}_5)_2(\text{C}_2\text{H}_5)_2(\text{C}_3\text{H}_7)_2$ being found to resist fission (see above).

Kipping also gave much attention to the reaction between sodium and organo-silicon halides of the type R_2SiCl_2 and R_3SiCl . The original object was to obtain a simple unsaturated

compound $R_2Si:SiR_2$ but this was never achieved. Diphenylsilicon dichloride, for instance, gave two crystalline compounds of the formula Si_4Ph_8 , one of which in later work was shown to be octaphenylcyclosilicotetrasilane (XIX), whereas the constitution of the other "is best represented by $\cdots SiPh_2 \cdot SiPh_2 \cdot SiPh_2 \cdot SiPh_2 \cdots$ ". This is much more reactive than the cyclic compound. It slowly changes on exposure to light and air and undergoes atmospheric oxidation in toluene at 100° and combines with iodine at ordinary temperature (*J.*, 1923, **123**, 2590).



In 1932, Kipping and Blackburn (Part XLVI, *J.*, 2200) showed that nitration of tetraphenylsilane yielded a small amount of a pure nitro-compound which, on decomposition with bromine, gave *m*-bromonitrobenzene. The main product of nitration, however, was extremely ill-defined and with bromine yielded a mixture of *m*- and *p*-bromonitrobenzene approximately in the proportion of 2 : 1. The occurrence of *o*-nitration was not established. The pure tetranitro-compound was reduced to the corresponding amine, which was thoroughly examined.

Part I of "Organic Derivatives of Silicon" (*J.*, 1935, 1088) is noteworthy as marking the author's Jubilee and also because it emphasised his definite, though late, entry into the field of aromatic substitution. Had he undertaken work of this kind earlier most interesting results would no doubt have been forthcoming. The paper describes the nitration of phenyltriethyl-, diphenyldiethyl-, and triphenylethyl-silanes, $PhSiEt_3$, Ph_2SiEt_2 , and Ph_3SiEt . Examination of the bromonitrobenzenes obtained from the crude products of nitration showed that *m*-nitration diminishes and *p*-nitration increases as the phenyl groups are replaced by ethyl groups: " $-SiPh_3$ in fact is more strongly *m*-orientating than $-CCl_3$, although rather less so than NO_2 ."

When more than two phenyl groups were present in the silanes the nitration product was of a very ill-defined nature. Kipping thought it probable that *m*- and *p*-nitration might occur in the same molecule.

My association with Kipping began in December 1907, when I went to see him about a small research scholarship which was advertised at Nottingham. I well recall the rather searching questions he asked about the underlying object of some exploratory experiments in research on which I had been engaged for a few weeks at the suggestion of a former teacher. The research scholarship was awarded to someone else, but Kipping made arrangements which enabled me to begin work with him in the following January. At that time new research students were told to prepare (\pm)-methylhydrindamine (*J.*, 1903, **83**, 913) starting from ethyl benzylmethylacetate. During this preliminary initiation or very shortly afterwards I first heard addressed to me, the words so familiar to all of Kipping's students—"you started out with 50 grams of ester, you now show me 5 grams of this stuff. *Where's it all gone to?*" There was often no answer!

During 1907 Kipping had published (*J.*, **91**, 209) his account of the resolution of the disulphonic acid of benzylethylpropylsilyl oxide by means of the (+)- and (-)-methylhydrindamines. Having succeeded in establishing the capacity of asymmetric compounds of silicon to give rise to optical activity, he wished to confirm this by finding other examples. I was therefore given a crude fraction containing dibenzylethylsilyl chloride $(Ph \cdot CH_2)_2SiEtCl$ and told to purify it, replace the chlorine atom by a propyl group by means of the Grignard reaction, sulphonate one benzyl group, and resolve the resulting dibenzylethylpropylsilanemonosulphonic acid.

The somewhat painful "breaking in"—or perhaps "breaking down"—process undergone by all who wished to serve Kipping, the oft-repeated preparation of more material, the separation of the monosulphonic acid from the disulphonic acid (simultaneously produced) by fractional crystallisation of the strychnine or (-)-menthylamine salts (which did not resolve the acid), and the final preparation of the sodium salt by removal of the base occupied over a year. Months were then spent in trying one optically active base after another in the hope of effecting resolution but, as in the case of two other benzylsulphonic acids containing only one atom of silicon (*J.*, 1907, **91**, 717; 1909, **95**, 69), without success. Kipping decided one morning to give up the struggle and publish the preparation of the acids and the negative experiments on resolution and to "get on with" something else, but—"have you tried brucine?" He was told that it was the only available alkaloid which we had not tried. Then—"how much sodium salt have you still got?" The answer—"about one gram"—decided him. "We may as well try brucine" he said. Then—to quote the joint paper which Kipping wrote later—"the

brucine salt could hardly be recrystallised without its affording clear evidence of physical heterogeneity."

But there was no more material on which further experiments could be made and—this was in May—it was not till the end of November 1909 that enough of the two brucine salts could be collected to warrant a conversion through the (–)-menthylamine salts into the two sodium salts. The brucine salts differed in m. p. by about 100°, but the (–)-menthylamine salts by only about 1.5°. At last the sodium salt from the sparingly soluble fraction of brucine salt was examined polarimetrically. The specific rotation was about –1°. Kipping was informed. "You know what I think," he said. He was assured that the rotation was *not* due to traces of brucine or (–)-menthylamine. With trepidation the sodium salt from the other end was prepared and dissolved. I went home for tea. On returning in the evening, I placed the tube in the polarimeter and a slight dextrorotation was observed. (The preparation had never been near a dextro-rotatory base.) A fellow research worker was asked to say what he thought of the two yellow half-circles. "Dextro"—was the answer. Kipping was informed by telephone—with careful suppression of any natural exuberance. "Well"—pause—"we shall be able to get on now." Nothing more! Two years hard labour—two years in which I, in common with others, had only been sustained by the words quoted by Seton Merriman in one of his novels then so much the vogue—"he who has lost all hope has also lost all fear." Two such years—during which at times the sight of the *Journal of the Chemical Society* was almost a pain, so remote did success and possible publication appear—might have warranted slightly warmer congratulations. But the Master had trained us well. We did not expect much. . . .

The morning however was yet to come. . . . Kipping looked through the tube. The sodium salt had been dried at 100° and the solution on standing overnight had formed a very slight suspension. "The sooner you learn that you can't take a rotation in a turbid solution the better," he remarked. The specific rotation was about +1.5°, lower even than the figures obtained for the first optically active silicon compound in 1907. This did not trouble Kipping, however, as a further study of the behaviour of the (+)- and (–)-sodium salts afforded decisive evidence of resolution. It was also shown that the sulphonic acid group was not situated in the CH₂ of the benzyl group.

A few days after the resolution Kipping came down with several small filter flasks and Büchner funnels for use in the examination of the properties of salts of the optically active acids. The future work was outlined. "How soon do you think you'll finish examining these salts?" "In about 3 weeks" was rather tentatively suggested, producing the comment in the characteristic deep voice which we feared and imitated—and loved—"I don't suppose you will." He was right.

When the work was collected in the form of two joint papers, I, like others of his students in similar circumstances, was told that I was to "read" the first one dealing with the preparation and purification of the sulphonic acids before the Chemical Society in London. Thereupon followed a somewhat embarrassing, but very valuable, rehearsal in a small class room with the Professor as sole audience. At a later meeting of the Society it was my privilege to hear him give an extraordinarily lucid account of the resolution experiments. H. B. Dixon, who was in the Chair, described the work as a combination of genius and patience. Lest one should be exalted overmuch, W. N. Haworth, who knew Dixon well, explained shortly afterwards that he doubtless meant that the genius was Kipping's and the patience his student's. No doubt he was right, but, looking back, I feel that much of the patience must also have been exercised by Kipping—in spite of occasional suggestions to the contrary. Years afterwards one of his students expressed his indebtedness to the Professor "for his kindly patience and even more for his kindly impatience." The patience and understanding predominated. To a student who proposed to apply for a scholarship for which he was not really fitted at the time he said—and his manner was quite different from his everyday habit—"you're not ripe yet, but it will come."

An expert manipulator, especially of small quantities of material, Kipping had an uncanny facility for detecting an impurity, for removing it, and for causing intractable gums to crystallise. He was sceptical of all results not thoroughly substantiated.

His students will never forget the experiments which he used to carry out at their benches, using only test-tubes and glass rods. "Why can't you get some glass rods? They're cheap enough" he would say when one's supply failed. Greenaway ("The Life and Work of Professor William Henry Perkin," *The Chemical Society*, 1932, p. 15) writes: "Baeyer, as is well-known, worked on a small scale, mainly in test-tubes of which large numbers were kept in racks in his private laboratory. Wide-mouthed bottles containing a large stock of glass rods were

also there, and small flasks, beakers, etc. A striking feature was the absence of large apparatus." These lines might have been written of Kipping.

He was a man of few words, whether spoken or written. On visiting a student his greeting was usually—"Well?" If he expected very little there was sometimes no greeting at all—he just waited. On leaving he would say—"Well, get on with it!" or "keep busy." On one occasion he gave this valuable advice: "You want to get up in the morning and think out ten times as much as it's possible to do, and then come down and do it."

Once with his rather picturesque exaggeration he said, "If — says a substance is pure he means there's 50 per cent. there and if he says its crude then there's none there at all." He had a horror of what he called "a mare's nest" and always invoked this zoological phenomenon as the most probable explanation of his students' results, especially if the results had long been hoped for.

An all-round athlete himself, he encouraged his students in the many-sided activities of College life. He even sympathised when these occupied too high a proportion of their time. "So-and-so is a very nice fellow, but awfully lazy." To another, now holding a responsible position in the profession of analytical chemistry, he said "The Senate believes that you are the root of all evil in this College and I've been told to reprimand you. . . . Well" (that deep voice) "here's the reprimand!"

After two years' absence from his laboratory I returned to work there for several weeks and met Robison, one of his earliest workers in the silicon field, who, after studying in Germany with Hantzsch and spending a period in Galway on Senier's staff, had joined Kipping as an Assistant Lecturer. He had found, as he told us, life in Connaught too soft and easy. He knew where to find the antidote. Robison, during both his periods with Kipping, worked on the condensation products of diphenyl- and dibenzyl-silanedioles $R_2Si(OH)_2$ and obtained, after much almost heartbreaking labour, several beautifully crystalline condensation products. During this and similar work many viscous residues were obtained, the further examination of which appeared too daunting even to the master, whose mere presence in the laboratory was popularly said to induce the process of crystallisation. Recently these "silicone polymers" have achieved world-wide recognition as plastics, insulating materials, lubricants, and in other connections.

Shortly after this Robison joined Harden at the Lister Institute. His later work is built into the fabric of biochemistry. It is a tribute to both master and pupil that without any previous knowledge of biochemistry, but with the training and discipline of Kipping as an all-sufficient foundation, Robison should so rapidly have achieved such outstanding results.

In the years after 1912 I met Kipping from time to time when he was external Examiner to the University of Birmingham—on one of these occasions he greatly pleased the Franklands, with whom he stayed at Northfield, by cutting their extensive lawn in war-time. After Frankland's retirement I had the privilege of entertaining Kipping in my bachelor lodgings and he was much intrigued by my landlady's parrot, whose comments somewhat disturbed our consideration of border-line cases—an occupation not facilitated by the failure of the gas one late evening in that first post-war summer of 1919. These rooms were well-known to a long succession of Frankland's staff. He also examined my first two Ph.D. students at Manchester, when he showed himself a very sympathetic inquisitor. He was, however, somewhat dismayed by the Manchester practice of admitting members of Staff and prospective Ph.D. candidates to the oral examination! When I offered to see him off by train he refused with his characteristic desire to spare people trouble, saying—"Certainly not; I knew London Road Station before you were born."

Dr. A. H. Salway writes:

"During the years 1900—1904, when I was privileged to be a student under Kipping at University College, Nottingham, the college was largely attended by so-called 'normal' students aspiring to become certificated teachers under the Board of Education, and independent University students were in a minority. Of students wishing to prepare themselves for a career in the chemical profession there were not more than a dozen all told. In these conditions the Professor lectured only to the former class of students, preferring to coach the advanced chemical students privately. Thus he paved the way for many a promising pupil to take his London degree with honours as an external student.

"In those days the research students were accommodated in an underground cellar, the 'Lower Lab.,' reached by descending an iron staircase, and the equipment at our disposal was not of the ultra-modern variety seen at the present day. But that did not matter; all we

required in our attempts to prove the asymmetry of the nitrogen atom was an unlimited stock of small beakers. If one visited the Professor's private laboratory the first objects which caught the eye were rows and rows of salt-encrusted beakers lined along the benches, with father-beaker at the head of the column and his family of small and smaller children trailing into infinity behind. And so our underground cellar became a feeble imitation of the Professor's sanctum.

" This kind of work on the intensive fractionation of ever-diminishing quantities proved such excellent training in the handling of infinitesimal amounts of material that it has been said of some of his pupils that they had only to handle a test-tube and the result was 3 pages of original observations for the *Journal*.

" Professor Kipping was a man of some pawkish humour, which, at least on one occasion, called for the remark, from one of his colleagues, ' It's that man again,' thus forestalling Mr. T. Handley by some 40 years. One example I may perhaps be permitted to recall. Sometimes his lecturers did not quite display the Professor's brilliance in capturing the ears of their listeners, so that lectures attended at first by some dozen students were apt to develop into private interviews with the lecturer. On some such occasion I was hurrying along the corridor on my way to a lecture when I encountered Professor Kipping, who slyly remarked : ' Hurry up ! Dr. — is looking for his class. Are you it ? '

" Perhaps the greatest tribute Kipping's pupils can pay to his memory is that he spared himself no time or trouble to foster their well-being or to find for them suitable posts in which their respective talents might find adequate expression. In the early days of this century professional posts were not like ripe plums in an orchard, waiting to be plucked at leisure ; therefore all the more credit to a Master who rarely failed to plant his pupils' feet on the first rung of the ladder.

" Personally, I feel a great debt of gratitude for his devotion to my welfare, particularly after I graduated in 1902 when I was on the point of accepting a post as science master at a grammar school. He alone made it possible for me (A. H. S.) to undertake post-graduate research and put me in the way of an 1851 Exhibition Scholarship."

It will be clear from this memoir that to work with Kipping was no easy thing. He believed with Jeremiah that " it is good for a man that he bear the yoke in his youth." The Vice-Chancellor of Leeds, when presenting him for his honorary degree, referred to his " exacting and severe standard of scientific accuracy at once the despair and the inspiration of his research students." Kipping's interest in beginners was equally great, and many tributes have been paid to the help and guidance which he gave them. To those who bore the yoke, survived the ordeal, and gave of their best, his friendly interest and readiness to help throughout the years knew no bounds.

He was an athlete and a sportsman, and it was said of him that " he owed his perennial youth in no small measure to . . . an expert manipulation of cyclic compounds on the golf course, the tennis court, and the billiard table."

Kipping's single-hearted devotion to research work undertaken for its own sake was an inspiration to many. The words of the writer of Ecclesiastes seem particularly appropriate to his life and outlook : " Cast thy bread upon the waters : for thou shalt find it after many days. . . . He that observeth the wind shall not sow ; and he that regardeth the clouds shall not reap. . . . In the morning sow thy seed, and in the evening withhold not thine hand ; for thou knowest not which shall prosper, whether this or that, or whether they both shall be alike good."

FREDERICK CHALLENGER.