

Felos

# Franz Fehér (1903-1991): A Personal and Professional Portrait

On February 16, 1991 — shortly after his 88th birthday — Dr.-Ing. Franz Fehér, retired full professor of inorganic chemistry at the University of Cologne, passed away after a brief illness. This came as a great surprise to his friends, colleagues, and the large group of his former students. His death interrupted an active and practical interest in chemistry and terminated a life dedicated to science. He himself was presumably not expecting his life to end so suddenly as he had numerous plans for further publications, papers that will now remain unwritten.

In the mean time more than half a decade has passed and it is now opportune and possible to present an objective laudatio about the life and scientific achievements of this personality in inorganic chemistry — a man who made a significant contribution to the renaissance of the chemistry of non-metals after the Second World War.

## Childhood, Adolescence, and University Education

Franz Fehér's life was decisively influenced by his origins as well as the political and economic events of this century. He was born on January 22, 1903 in the small town of Szentamas in southern Hungary as the third of five children. His father, a member of the Hungarian ethnic group, a mechanical engineer and businessman, owned a company dealing in agricultural machinery. His mother, maiden name Sieler, belonged to the German ethnic group that settled there in the 18th century. Together with two brothers and two sisters he enjoyed his childhood in a happy, natural family atmosphere. However, this free and easy life came to

an end at the age of ten years when he entered a boarding school (Humanistisches Gymnasium) in the county town of Neu-Verbas and could only return home during the school holidays. He had no problems with his school education and, although good in all subjects, mathematics and natural sciences attracted his attention the most. In later conversations he occasionally mentioned his everlasting gratitude to his mathematics teacher.

The First World War began during his high school time, the further course of which led to enormous changes in this part of eastern Europe. At the end of the war in 1918 south Hungary became part of the newly formed kingdom of Serbia, Croatia, and Slovenia, the recently disintegrated Yugoslavia. This meant that his home town was renamed Srbobran and that the language in his school, previously Hungarian, changed to Serbian. In spite of these difficulties Franz Fehér mastered the last three years of high school and left it in June 1921 at the age of 18 with an excellent certificate of education (Reifezeugnis). With this he had made a decisive step towards fulfillment of his long held dream to read chemistry in Germany. This choice of subject was without doubt based on his proficiency for both abstract scientific thought and practical work with his hands.

During his search for a suitable place to study he was given the rather misleading advice from the German embassy in his country that one could read chemistry in Germany at a technical university (Technische Hochschule) — but not with the additional information that one could also read chemistry at a normal university albeit without the possibility of receiving a Diploma certificate. Thus, in the

winter semester of 1921/1922 Franz Fehér registered at the Technische Hochschule Hannover where Wilhelm Biltz was director and professor of inorganic chemistry. However, it was the year 1921 and veterans of the First World War were still seeking admission to higher education, especially since possibilities in other occupations were scarce because of the economic situation prevailing at that time. Hence, Wilhelm Biltz regretfully had to inform Franz Fehér at the beginning of the first semester that he could not provide a laboratory place and that the waiting time for one was uncertain. The young student, therefore, could only attend the basic lectures in chemistry and physics while applying for admission to other technical universities.

He was finally accepted by the Technische Hochschule Stuttgart for the summer semester 1922 where he continued his education with practical instruction in inorganic chemistry. Here he also attended, among others, lectures on experimental inorganic chemistry by A. Gutbier, organic chemistry by W. Küster, and physical chemistry by G. Grube. In July 1925, after only seven semesters (including a Diploma thesis on an electrochemical topic under the supervision of G. Grube) he successfully completed the first stage of his chemical education and obtained his Diploma certificate. But then his financial resources were at an end; money transferred from home became worthless from one day to the next on account of the galloping inflation in Germany at that time. Thus Franz Fehér was forced to interrupt his further scientific education for two years and to take up a teaching position for mathematics, physics, and chemistry at the Oberrealgymnasium (state high school) in his home town.

In autumn of 1927 he returned to Technische Hochschule Stuttgart to prepare a doctoral thesis under the supervision of A. Simon at the Institute of Inorganic Chemistry. His thesis entitled "Beiträge zur Kenntnis der Mangandioxydhydrate und der Manganoxyde" (Contributions to the Chemistry of Manganese Dioxide Hydrates and Manganese Oxides) was awarded the Gutbier prize of the Technische Hochschule Stuttgart. In spite of the two-year break he received the title Dr.-Ing. (graded excellent) at the age of 26.

Even during this post-graduate work, Franz Fehér's special ability to successfully solve experimental problems by the design and construction of original but simple apparatus became apparent. For example, for tensiometric investigations on various manganese oxides he developed a novel, automatic cryostat for low temperature work. A short while later this system was also used by E. Zintl and W. Klemm and reported to work very reliably. Investigations on the vapor tension curve and isobaric degradation of manganese heptoxide presented particular challenges. First of all, the preparation of the pure, highly explosive compound Mn<sub>2</sub>O<sub>7</sub> in preparatively useful amounts was decisively improved. Furthermore, Franz Fehér invented a new method to measure small pressures by remote monitoring since, as he wrote, being present in the room for the accurate measurement of small pressures during the thermal treatment of manganese heptoxide proved to be highly hazardous just

on account of the explosive nature of the substance. With the developed system the required pressure measurements could be made safely in a different room, at a safe distance from the decomposition apparatus.

After receiving his doctor's degree Franz Fehér participated in several research projects of his supervisor and during this time decided to follow a career in the university. At the same time he was involved in teaching at the inorganic institute in Stuttgart, first as a Hilfsassistent ("temporary assistant") and then as an Außerordentlicher Assistent ("associate assistant"). In a later conversation he once explained the significance of the latter position: during the worldwide economic crisis of 1931, many ex-students of the institute who had taken up positions in industry were dismissed by their companies and sought to return to the technical university. In order to help this group of people the planmäßigen Assistenten-Stellen ("regular assistant positions") were first divided into two and then even into four part time positions. Thus, Franz Fehér's times of financial restraint were not yet finished. As Praktikums-Assistent ("assistant in a laboratory course") he met the chemistry student Magdalena Fischer from Temesvar in Rumania who, from her father's side, belonged to the German ethnic group there. They were married in September 1931. In April of the same year he had taken on the Germany nationality. From the point of view of his home country, this meant that he had evaded his military service obligations, a serious offence. Accordingly, he was never able to return home again and later even avoided any trips to the former German Democratic Republic for fear that he could be extradited from there to Yugoslavia.

## The Years in Dresden and Göttingen

In 1932 Franz Fehér accompanied A. Simon to the Technische Hochschule Dresden as Planmäßiger Assistent ("full assistant"). Here, the couple moved into a modest apartment belonging to the institute where their three daughters Magda (born in 1935), Therese (born in 1937), and Franziska (born in 1942) spent their first years of childhood.

The ten years in Dresden served first of all to survey the field of inorganic chemistry for an independent research area with a clear potential for development. Already during his time in Stuttgart, shortly after the discovery of the Raman effect in 1928, Franz Fehér had carried out first studies on the use of Raman spectroscopy to solve constitutional problems in the chemistry of non-metals. These investigations were continued in Dresden – at first in cooperation with A. Simon. For the oxo-acids of phosphorus and arsenic, the constitutions of which were a subject of controversy in the literature at that time, it was possible with the help of the Raman spectra to provide unambiguous answers to the questions as to whether and which of these anhydrous acids existed in the "aci-" or "pseudo"-form. In addition, direct information on the question of the bonding of hydrogen to the central non-metallic atom was obtained. Further work was concerned with constitutional problems of orga-

nometallic compounds. A major topic of interest from the middle of the 1930's on was the elucidation of the constitution of hydrogen peroxide and its derivatives for which at that time a Y-shaped structure with a double bond between the two oxygen atoms was assumed. Since infrared spectroscopy was then just at the beginning of its instrumental development, Raman spectroscopy was the method of choice for the solution of these constitutional questions. In addition, X-ray crystallography, the evaluation procedures for which were still also in their infancy and very laborious, was included in the investigations. A decisive prerequisite for these investigations was the often difficult synthesis of the required compounds in a pure state such as, for example, 100% hydrogen peroxide or deuterium peroxide and their preparation for crystal structure analyses. Thus, for example, single crystals of pure hydrogen peroxide were grown in the cold room of a slaughter house in Dresden. The investigations on hydrogen peroxide in which the constitutional and conformational analogies to hydrazine were demonstrated unequivocally resulted in the award of the title Dr. Ing. habil. (Habilitation) in 1938 and shortly thereafter to the "venia legendi" (authorization to hold lectures) for the subjects inorganic and analytical chemistry. They were further honored in 1941 by the award of the Carl-Bosch Prize from the former I. G. Farbenindustrie.

Even so, Franz Fehér could not continue his work at the university without interruption. Between 1935 and 1939 he was drafted to attend several training courses for the German army and took part in the operations against Poland at the beginning of the 2nd World War. Subsequently, however, within the framework of the Osenberg plan intended to protect young university lecturers and to ensure the continuation of research after the war period, he was declared an indispensable person and so could return to the Technische Hochschule Dresden. As assistant lecturer he managed the laboratory courses in quantitative analysis and preparative inorganic chemistry and held courses on the theory of chemical bonding as well as the basic principles of Raman spectroscopy and X-ray crystallographic analysis. He was generally admired and respected by his students not only because of his quiet, friendly and helpful nature but also for his extensive knowledge; he was known throughout the institute as Uncle Franz. During this time he took on a new field of interest, the acyclic sulfur compounds and above all the higher homologues of hydrogen sulfide. Continuation of this work in Cologne later led to the complete characterization of the series of sulfanes  $H_2S_n$  and chlorosulfanes  $S_nCl_2$  up to the corresponding octasulfanes (n = 8) by Raman spectroscopy. These results are still to be found in all textbooks of inorganic chemistry.

In 1942 Franz Fehér was invited by Adolf Windaus to join the department of inorganic chemistry in the chemical institute at the University of Göttingen as senior assistant. He could not resist this opportunity to experience a *university* chemical institute where, according to the prevailing traditions, inorganic chemistry was only represented by a department. In the stimulating atmosphere of the institute

directed by Adolf Windaus, Franz Fehér was able to continue his studies of acyclic sulfur compounds on a broader basis. In 1944 he was appointed Außerplanmäßiger Professor. Franz Fehér gained many lasting impressions from the vital scientific atmosphere of the science faculty in Göttingen. Above all, he encountered Adolf Windaus whom he admired not only as a university teacher and scientist but also as an impressive personality who served as an ideal for the rest of his life. In later conversations he often mentioned that he considered Adolf Windaus to be his truely academic teacher. During this time and with an increasing group of coworkers the Raman spectroscopic investigations on sulfanes were consequently expanded by the development of new preparative approaches as well as methods for the analytical and physicochemical characterization of the individual homologues. In spite of the difficult working and living conditions during the World War II and immediate post-war years, the seven years in Göttingen were among the most fulfilling and molding in Franz Fehér's life.

### Cologne

On September 1, 1949 Franz Fehér took up the position as extraordinary professor of inorganic and analytical chemistry and head of the department of inorganic chemistry in the Institute of Chemistry of the University of Cologne. Here he faced the task of rebuilding the teaching and research facilities whose apparatus had mostly been lost during the war. Furthermore, at the beginning of the winter semester of 1949/1950 he found a long queue of prospective students waiting in front of his office, all wanting to apply for a laboratory place. Remembering his own problems at the start of his academic life, he did everything possible to help: he also held practical courses in the semester holidays, gave lectures on experimental inorganic chemistry twice a day in addition to his lectures on analytical chemistry and special inorganic chemistry.

In spite of this heavy teaching load during the first years in Cologne his research interests were not neglected. The studies of acyclic sulfur compounds started in Dresden and continued in Göttingen were systematically expanded in several directions with the help of an increasing number of coworkers. One of the questions addressed was whether, in analogy to the sulfanes, H<sub>2</sub>S<sub>n</sub>, other series of the compounds of the general formula  $S_nX_2$  with skeletons made up of chain-linked sulfur atoms would exist. If so they could be considered as derivatives of the corresponding parent compounds  $H_2S_n$  and would provide possibilities for new synthetic approaches to widely differing sulfur compounds having ring and chain skeletons. Of particular interest in this context were the halosulfanes and especially the chlorosulfanes,  $S_nCl_2$ . Up till then only the compounds  $SCl_2$  and S<sub>2</sub>Cl<sub>2</sub> were known with the constitution of the latter still being uncertain. A second field of interest was the parent compound series  $H_2S_n$  with the objectives of finding new methods of synthesis and optimizing those already developed for the individual homologues. The actual purpose

behind these investigations was to achieve a practically complete spectroscopic and physicochemical characterization of these new compounds for which appropriate amounts of highly pure substances were needed. Finally, the investigations of open-chain sulfur compounds were systematically expanded to include elemental sulfur.

In the work on chlorosulfanes evidence for the existence of compounds more rich in sulfur than S2Cl2 was obtained in the so-called "hot-cold" tube. In this process S2Cl2 was allowed to react with hydrogen at 860-875°C: HCl was eliminated and the formed products were shock cooled to room temperature. In this way orange-yellow oils with the sum compositions S<sub>20</sub>Cl<sub>2</sub> to S<sub>24</sub>Cl<sub>2</sub> were obtained. Raman spectroscopic examinations showed that these oils consisted mainly of mixtures of higher homologous sulfur chlorides with varying chain lengths. A major breakthrough was the discovery of a preparatively more simple and more efficient method of synthesis on the basis of the observation that, under suitable conditions, the sulfanes H<sub>2</sub>S<sub>n</sub> undergo condensation reactions with S<sub>2</sub>Cl<sub>2</sub> or SCl<sub>2</sub> with quantitative elimination of HCl to furnish more sulfur-rich members of the homologous series. Since the primarily formed molecules could also react similarly with elimination of HCl and chain lengthening, chlorosulfane mixtures with an average composition of S<sub>x</sub>Cl<sub>2</sub> were obtained from which pure S<sub>3</sub>Cl<sub>2</sub> and S<sub>4</sub>Cl<sub>2</sub> as well as highly enriched S<sub>5</sub>Cl<sub>2</sub> were isolated by short-path, high vacuum distillation. The concentrations of all occurring molecules in dependence on the molar ratios of the starting materials were deduced by considering the kinetics of the reactions. By use of a large excess of the chlorosulfane component subsequent reactions of the primary products were suppressed so that, after removal of the excess, formula-pure higher chlorosulfanes were obtained directly. In this way sulfur-rich homologues up to S<sub>8</sub>Cl<sub>2</sub> were prepared by reaction of  $H_2S_n$  with excess  $S_mCl_2$  and subsequent removal of unconsumed chlorosulfane (Scheme 1).

Scheme 1. Preparation of chlorosulfanes

$$CI-S_m-CI+H-S_n-H+CI-S_m-CI \xrightarrow{-2 \text{ HCI}} CI-S_{2m+n}-CI$$
  
 $m=1,2; n=1,2,3,4$ 

The same synthetic concept was then applied to higher members of the sulfane series itself. Of these, the compounds  $H_2S_2$  to  $H_2S_6$  had been prepared during the work in Göttingen from the "crude sulfane" obtained, in turn, by acidic decomposition of an aqueous solution of sodium polysulfide. With this new access at hand, the compounds  $H_2S_5$  and  $H_2S_6$  were prepared in high purity and, furthermore, the higher homologues  $H_2S_7$  and  $H_2S_8$  were also obtained

This same synthetic route also provided an access to widely differing sulfane *derivatives*. Thus, condensation of sulfanes with chlorosulfonic acid resulted in the formation of sulfanemonosulfonic acids,  $H_2S_nO_3$ , reaction of which with chlorosulfanes gave the sulfur-rich sulfanedisulfonic acids (polythionic acids)  $H_2S_nO_6$ . In this way the free octa-,

nona-, deca-, undeca-, and dodecathionic acids were prepared for the first time as oily liquids that were stable at lower temperatures (Scheme 2).

Scheme 2. Synthesis of sulfanemonosulfonic acids and sulfanedisulfonic acids (polythionic acids)

In addition, the already known diorganyl polysulfides  $R_2S_n$ , although their constitutions at that time had not been clarified with certainty, were prepared by an analogous route as were also some new representatives of this compound class. Investigations of their properties (see below) then unequivocally confirmed their identity as organo-substituted sulfanes with unbranched sulfur chains. The synthesis of "perchlorodimethyl tetrasulfide" ( $Cl_3CS_4CCl_3$ ) from  $Cl_3CSCl$  and  $H_2S_2$  and the determination of its structure by Raman spectroscopy can be mentioned as a typical example of these efforts. A large range of unsymmetrically substituted sulfane derivatives of the type  $RS_nCl$  and  $RS_nH$  was prepared by reaction of excess chlorosulfane or sulfane, respectively, with monofunctional compounds such as RSH,  $R_2NH$ , and  $RS_mCl$ .

Attempts to transfer the chlorosulfane synthesis to the corresponding pure bromosulfanes  $S_nBr_2$  (n>2) were not successful since the excess of dibromodisulfane  $S_2Br_2$  remaining, on account of its high boiling point, could not be removed at the completion of the condensation without decomposition of the formed higher homologues. However, the preparation of practically pure, sulfur-richer bromosulfanes up to  $S_8Br_2$  were possible by reaction of the corresponding chlorosulfane with hydrogen bromide.

Raman spectroscopic investigations of the already long known compounds  $S(CN)_2$ ,  $(SCN)_2$ ,  $S(SCN)_2$ , and  $S_2(SCN)_2$  led to their recognition as the starting members of a homologous series of cyanosulfanes  $S_n(CN)_2$  and the series was extended up to  $S_8(CN)_2$  (Scheme 3).

Scheme 3. Synthesis of sulfur-rich cyanosulfanes

$$CI-S_n-CI + Hg(SCN)_2 \xrightarrow{-HgCI_2} NC-S_{n+2}-CN$$
  
 $n = 3-6$ 

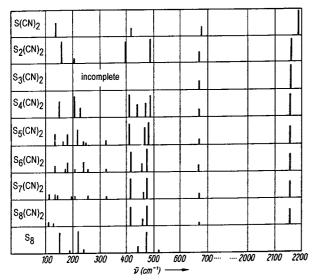
The electrolytic reduction of aqueous  $SO_2$  solutions as a means to obtain larger amounts of sulfane mixtures with average compositions of  $H_2S_8$  to  $H_2S_{13}$  was studied intensively. After clarification of the electrode processes a laboratory apparatus was developed that allowed, under continuous operation and good current yield, the simultaneous cathodic preparation of sulfane oils and the anodic production of ammonium peroxodisulfate.

The alkali metal compounds  $M_2^1S_n$  were also included in the investigations as *sulfane derivatives* and detailed studies on the various anhydrous polysulfides in the systems sodium—sulfur, potassium—sulfur, rubidium—sulfur, and cesium—sulfur were carried out.

In all of the classes of compounds investigated, Franz Fehér's main target was not merely the preparation of new substances; spectacular momentary successes were not the driving force of his work. Rather, his interest was always directed towards the comprehensive exploration of a new area in inorganic chemistry. This objective required intensive structural and physicochemical characterization of the newly prepared compounds, aspects that played a decisive role in the scientific work of Franz Fehér.

Elucidation of the constitutions of the sulfanes and their various derivatives was based mainly on the results from Raman spectroscopy. As an example, the Raman spectra of the cyanosulfanes  $S_n(CN)_2$  (n=1-8) are depicted in Figure 1. These spectra show that the respective substances are chemically individual species without any traces of physically dissolved elemental sulfur. The appearance of an individual spectrum is always characteristic for the respective number of chain-linked sulfur atoms in the molecular skeleton. Electronic spectra of the various classes of compounds were also investigated systematically and discussed on the basis of Kuhn's electron gas model. These results indicated that, in addition to normal  $\sigma$ -bonding, delocalized  $\pi$  electrons were also present in the sulfur chains.

Figure 1. Raman spectra of cyanosulfanes  $S_n(CN)_2$  (n = 1-8) and of  $S_8$ 



The individual compounds among the metal polysulfides were studied by X-ray methods and characterized on the basis of their powder diagrams while single crystal X-ray structural analyses of the cyanosulfanes were carried out.

The physicochemical properties of the sulfanes and chlorosulfanes were subjected to extensive studies in particular regard to the constitutions of the individual compounds. It was found that the molar volumes, molar fractions, molar enthalpies of evaporation, and the logarithms of the viscosity were linear functions of the length of the sulfur chain and that no branched chain skeletons were present. For the sulfanes  $H_2S_2$  to  $H_2S_5$  the boiling points and critical data were also estimated by means of vapor pressure measure-

ments. Furthermore, the heats of decomposition of the catalytic degradation into hydrogen sulfide and sulfur were measured by calorimetry and the data used to determine the enthalpies of formation for the individual homologues up to  $H_2S_6$ . The respective values for the formation of *liquid* sulfanes from the elements in their standard states are all negative; their absolute values decrease linearly with increasing number of sulfur atoms. On the other hand, all sulfanes are metastable towards a decomposition to hydrogen sulfide and sulfur at room temperature. Their existence can be attributed to a relatively high activation barrier of the decomposition reaction which was determined to be about 25 kcal/mol for  $H_2S_2$ .

Very early in the course of these comprehensive investigations on various classes of compounds with chains of sulfur atoms, Franz Fehér recognized the many relations to the chemistry of open-chain hydrocarbons. Aside from this he had always considered the molecular chemistry of the nonmetals as a single topic and did not draw any arbitrary boundaries between inorganic and organic chemistry in his research work. His efforts to clearly express the analogies between the compounds  $H_2S_n$  and the *n*-alkanes  $C_nH_{2n+2}$ in the nomenclature by replacement of the then usual terms with the name "sulfanes" become understandable in this light. This results in the names "disulfane" (H2S2), "trisulfane" (H<sub>2</sub>S<sub>3</sub>) and so on for the individual homologues. This nomenclature system also clearly shows the genetic relationships between the compound classes  $H_2S_n$ ,  $S_nX_2$  (X = halogen, monofunctional group),  $S_nR_2$  (R = alkyl, aryl), and M<sub>2</sub>S<sub>n</sub> and has now been widely accepted by the chemical community. As a direct consequence, "cross-products" of sulfanes and alkanes, in particular compounds with the general formula RS(CH<sub>2</sub>S)<sub>n</sub>R – termed "bunte" sulfanes in Fehér's laboratory - were investigated. A series of thiaalkanes was then prepared and their properties determined for the first time.

The studies on sulfur-chain compounds prompted Franz Fehér in the mid 1950's to take up an intensive investigation of elemental sulfur. It was found that many of the older results on the properties of molten sulfur were not correct since the samples investigated were of insufficient purity. Thus Fehér's group developed methods for the purification of sulfur by electrophoretic means and especially by zone melting; that latter has proved to be very suitable for obtaining highly pure sulfur. In the course of this work, the then usual analytical methods for detecting small amounts of carbon and other contaminants in sulfur had to be improved considerably. Using the thus obtained and characterized pure samples, the temperature dependence of the density and specific heat of molten sulfur in the range 120 to 400°C was measured. Both parameters also exhibited anomalies in the region of the known viscosity increase and these were discussed in terms of the processes occurring in liquid sulfur (cleavage of S<sub>8</sub> rings and formation of longer  $S_x$  chains). Furthermore experiments on the electrical conductivity of molten sulfur were performed and the mecha-

nism of charge transport elucidated on the basis of these data.

In the second half of the 1950's as a supplement to the continuing work on sulfur and its compounds, increasing interest was directed to studies on the higher silicon and germanium hydrides as well as the chemistry of hydrazine and its derivatives as new research topics. In the planning of his scientific work, Franz Fehér was never influenced by current mode topics or predetermined research projects but always followed his own scientific ideas. He viewed the development and work of a scientist to be closely analogous to that of an artist. After completing her doctoral thesis and a few years of postdoctoral work in the field of sulfur chemistry, the present author asked her supervisor if he thought her abilities were sufficient for a Habilitation. After a short pause he answered "I have heard that you want to be an opera singer, then go ahead and sing" and then added the phrase "but please not on the sulfur stage!" This meant: a successful Habilitation cannot in general be predicted; it depends on the further "vocal" development and the continuing desire to "sing" in spite of the monotonous daily practicing. This example illustrates Fehér's love for colorful language that often surprised his listeners with unexpected comparisons and metaphors but always correctly reflected the topic of the conversation. The additional phrase was intended to avoid future competition between different groups in the same institute but, above all, it provided the possibility for his former student to attain an independent image in the scientific community at an early stage in her career.

In the year 1958 chemistry in Cologne experienced a drastic change accompanied by numerous new tasks for Franz Fehér. On June 20 the director of the chemistry institute of the university, the Nobel prize winner Kurt Alder died at the age of only 55 and left behind a research group consisting of over 40 diploma and doctoral students. Franz Fehér was appointed deputy director and head of the entire institute by the faculty of mathematics and sciences. It was of major importance for him that not even one of Alder's students should suffer any personal loss in his or her further education. Although the diploma students were able to move to other groups within a short time, conclusion of the doctoral thesis projects already in progress proved to be a serious problem. The scientific supervision was continued by the extraordinary professor of organic chemistry, Leonhard Birkofer and Alder's assistants. However, Kurt Alder had only been able to finance such a large research group with the help of considerable external research funds which all stopped suddenly with his death. Thus, it was essential to persuade the managers of the various sources that financial support of Alder's work was necessary for about two years more. This meant numerous applications, accountings, and reports as well as taking on the full responsibility for the use of the then personally granted research funds. Franz Fehér was fully rewarded for his efforts when, two years later, the last of Alder's postgraduate students completed his thesis and oral examination without requiring any extra time.

In spring of 1960 Franz Fehér was offered the position of full professor and director of the institute for inorganic chemistry at the university of Hamburg. This was not an easy decision for him. New buildings for the chemical institute were to be built both in Hamburg and Cologne. In Hamburg, completion of the buildings in 1965 was expected although land ownership problems still had to be solved whereas concrete building plans were already in existence in Cologne. As had already happened in other chemical institutes in Germany, the faculty for mathematics and sciences in Cologne decided under the given circumstances to divide the chemical institute into two separate institutes, one for inorganic and one for organic chemistry and started negotiations with Franz Fehér to keep him in Cologne. Decisive criteria for Franz Fehér were the working conditions for the eleven years remaining until his retirement. After careful consideration of the starting situations for the two building projects he decided in August 1960 to remain in Cologne and was appointed full professor and director of the newly founded institute for inorganic chemistry in 1961. It was one of the biggest disappointments in his life that he could only move into the new building in 1975 as retired professor while the buildings in Hamburg were indeed completed by 1965. Even so, being fully aware of his responsibilities to the future working conditions in his discipline, he spent a major part of his last decade as institute director with detail planning and supervision of the new buildings for the inorganic institute and the common lecture rooms.

The scientific work on the various research topics continued in this period with unchanged élan. Work with the higher silicon and germanium hydrides demanded new inert gas techniques to allow handling of these air- and moisture-sensitive compounds — in contrast to the classical Stock methods — also outside of closed systems. Fehér reported on the respective developments for the first time at the IUPAC congress in Munich in 1959. His experience in handling spontaneously inflammable substances was indeed in demand and led to numerous consultations with the chemical industry on similar problems. Apart from these new research interests, the chemistry of the sulfanes and their derivatives was systematically expanded with particular emphasis at this time on the development of synthetic routes to novel heterocyclic sulfur compounds.

With such widely divergent activities words such as "holidays" or "relaxation" were more or less unknown to Franz Fehér. Only his enjoyment of driving was occasionally satisfied in car journeys to the Alps or Italy with his Munich colleague Walter Hieber. Even during the years in Dresden Franz Fehér was a passionate motorcyclist who would make a short trip to the Baltic coast over the weekend — which for assistants in those days only began on Saturday afternoon — and return punctually to the institute on Monday morning. After the interruption of the war and immediate post-war years, the purchase of his first car, a Volkswagen, at the beginning of the 1950's was a major event. As

well as the joy of driving, the response of the machine to the driver's actions was always a source of pleasure for the son of a mechanical engineer. However, he could also relax in the circle of his coworkers during occasional celebrations.

With the improved working conditions of the inorganic chemistry institute the investigations on open-chain and cyclic sulfur compounds were consequently expanded. In the case of *sulfane derivatives*, the enthalpies of formation of the chlorosulfanes  $S_n Cl_2$  (n=2-5) were determined by calorimetric measurements of the chlorination reactions. The corresponding values were then estimated for the higher homologues up to  $S_8 Cl_2$  on the basis of the linear dependence of the values on the length of the sulfur chain. Together with the already known enthalpies of formation of the sulfanes  $H_2 S_n$  the thermal requirements of the various condensation reactions between sulfanes and chlorosulfanes thus became available.

In this period, however, the main interest in sulfane chemistry was directed towards the preparative investigation of new series of compounds containing chains of sulfur atoms; these were mainly synthesized by condensation of a chlorosulfane with an appropriate monofunctional, nonmetal compound. Examples are the preparation of bis(imidazolyl)sulfanes as well as bis(triorganosilyl)- and bis(triorganosiloxy)sulfanes (Scheme 4).

Scheme 4. Synthesis of bis(imidazolyl)sulfanes, bis(triorganosilyl)sulfanes, and bis(triorganosiloxy)sulfanes

Compounds with a C=C bond directly adjacent to the sulfur chain were obtained by reactions of  $\alpha$ ,  $\beta$ -unsaturated mercaptans (enethiols) with chlorosulfanes. The UV spectra of these bis(enethiol)sulfanes (Scheme 5) reveal a conjugation effect of the  $S_n$  chain that is larger than the substituent effect of a *single* sulfur atom but markedly smaller than that of C=C bonds.

When the chlorosulfanes in these reactions are replaced by the bis(chlorodisulfanyl)alkanes,  $CIS_2-(CH_2)_y-S_2CI$  (y=1-5) obtained from the reaction of dithiols with  $SCl_2$ , a new "crossed-type" product from alkanes and sulfanes is obtained. In the compound  $Ph_3Si-S_3-(CH_2)_5-S_3-SiPh_3$  obtained from the reaction of the pentane derivative with  $Ph_3SiSNa$  the silyl groups are linked by an eleven-membered heteroatom chain.

Specific synthetic routes have also been developed for particular substance classes. Thus, Fehér and his group

Scheme 5. Bis(enethiol)sulfanes prepared

$$n-C_3H_7$$
  $n-C_3H_7$ 
 $C_2H_5-CH=C-S_n-C=CH-C_2H_5$ 
 $n=3,4$ 

$$C_6H_5$$

$$C_6H$$

found that the bis(aziridinyl)sulfanes  $(C_2H_4)-N-S_n-N(C_2H_4)$  (n=1-5), only difficulty accessible via the trimethylsilyl derivative of ethyleneimine according to Scheme 4, can be obtained by smooth transfer of the sulfur chains of the corresponding bis(imidazolyl)sulfanes to ethyleneimine (Scheme 6).

Scheme 6. Synthesis of bis(aziridinyl)sulfanes

$$N-S_n-N$$
 + 2 NH  $N-S_n-N$  + 2 NH

In the mid 1960's systematic investigations on a further aspect of sulfur chemistry, heterocyclic compounds containing directly linked sulfur atoms, were started. A major topic of interest for the inorganic chemist in this field were those compounds in which the individual atoms of homocyclic sulfur rings have been replaced by other bifunctional, nonmetallic groups. When these are organic units the products are the cyclocarbasulfanes. Fehér and his coworkers found that this class of compounds could, in general, be accessed by reactions of the appropriate dithiols with chlorosulfanes of varying chain lengths with the cyclocondensation being favored over polymerization by application of the Ruggli-Ziegler dilution principle. The first representative of the series S<sub>n</sub>CH<sub>2</sub> to be prepared was a derivative of the S<sub>8</sub> ring, heptathiocane, S7CH2 with a crown-like eight-membered ring, from the reaction of dichloropentasulfane with methanedithiol. By analogous reactions, hexathiepane (S<sub>6</sub>CH<sub>2</sub>), pentathiane (S<sub>5</sub>CH<sub>2</sub>, with a six-membered ring analogous to that of cyclohexane), tetrathiolane (S<sub>4</sub>CH<sub>2</sub>), and undeca-(monocarbacyclododeca-sulfane, thiacyclododecane S<sub>11</sub>CH<sub>2</sub>) were then prepared in the pure states. *C*-Alkyl-substituted derivatives of the corresponding ring systems were obtained by use of other geminal aliphatic dithiols. Furthermore, sulfur rings containing two or - by reaction of the dithiols with 1,2-bis(chlorosulfanyl)benzene - also three carbon "heteroatoms" such as 1,2,3,5,6,7-hexathiocane or

1,2,4,5-benzotetrathiepine and their derivatives were also prepared (Scheme 7).

Scheme 7. Synthesis of 1,2,3,5,6,7-hexathiocane and 1,2,4,5-benzotetrathiepine and their alkyl derivatives

R<sup>1</sup>, R<sup>2</sup> = H, alkyl, cycloalkyl

Application of the cyclocondensation reaction to vicinal dithiols such as *trans*-cyclohexane-1,2-dithiol led to bicyclic sulfur compounds with the chlorosulfanes  $S_n Cl_2$  (n=2-5) and to tricyclic ring systems with dichloromonosulfane or iodine as reaction partner. Analogous reactions of 1,2-dimercaptoarenes generally led to compounds containing C=C bonds while the corresponding tricyclic systems were prepared directly by reactions of the 1,2-dithiols with 1,2-bis(chlorodisulfanyl)benzenes. Novel hexathia[3.3]cyclophanes became accessible by reactions of 1,3- and 1,4-dimercaptoarenes with dichloromonosulfane or — preparatively more favorable — 1,3- and 1,4-bis(chlorodisulfanyl)benzenes (Scheme 8).

Scheme 8. Synthesis of saturated and unsaturated bi- and tricyclic sulfur compounds bearing  $C_2$ ,  $C_3$ , and  $C_4$  groups

$$\begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \\ \\ \\ \\ \end{array}\end{array} \end{array} \end{array} \hspace{-0.5cm} \begin{array}{c} \begin{array}{c} \\ \\ \\ \end{array} \end{array} \hspace{-0.5cm} \begin{array}{c} \\ \\ \\ \end{array} \end{array} \hspace{-0.5cm} \begin{array}{c} \begin{array}{c} \\ \\ \\ \end{array} \end{array} \hspace{-0.5cm} \begin{array}{c} \\ \\ \\ \end{array} \end{array} \hspace{-0.5cm} \begin{array}{c} \begin{array}{c} \\ \\ \\ \end{array} \end{array} \hspace{-0.5cm} \begin{array}{c} \\ \\ \\ \\ \\ \\ \end{array} \hspace{-0.5cm} \begin{array}{c} \\ \\ \\ \\ \end{array} \hspace{-0.5cm} \begin{array}{c} \\ \\$$

Single crystal X-ray crystallographic analyses of many of the prepared heterocyclic sulfur compounds were carried out in order to be able to compare the molecular parameters, in particular the ring conformation in the solid state, with those of the corresponding homocyclic sulfur compounds. Information on conformation changes in solution was obtained from the <sup>1</sup>H-NMR spectra recorded at differing temperatures. All new compounds were also comprehensively characterized by means of UV, IR, and Raman spectroscopy and the results interpreted from structural and bonding theory points of view.

The rich palette of topics in open-chain and cyclic sulfur compounds investigated in the ten years between the founding of the inorganic institute and Franz Fehér's retirement was only possible with the help of a relatively large group of coworkers in whom he had inspired a particular interest for the problems of the preparative chemistry of non-metallic elements. Franz Fehér was personally present in the laboratory from morning to evening and often for hours also on Sundays. He regularly attended the most important congresses for his discipline and held numerous lectures at other universities; however, he only reluctantly left his laboratories and the ongoing investigations to do so. In a critically ironic tone he designated colleagues who were often away on trips as "chemical tourists". As well as his wealth of ideas Franz Fehér provided his postgraduate students with sufficient freedom to define their research tasks and thus inspired them to individual initiative so that a fertile and trustworthy cooperation resulted. Even so, he always took the time to give advice on personal matters and provided indispensable help for the future careers of his students. Many of his students attained their Habilitation either in Cologne (M. Baudler, K.-H. Linke, H. D. Lutz) or at other universities (H. J. Berthold, G. Winkhaus) where he had smoothed the way for them. He furnished all of his 130 postgraduate students not only with a scientific education but also served them as an example for future life through his friendly open-mindedness, his unerring grasp of the essentials, and his personal modesty. However, it was not always easy to completely satisfy his strict requirements for accuracy in experiment and precise written formulations. Once he had made up his mind about a scientific or university matter on the basis of careful and well-founded considerations it was difficult to induce him to view the matter from a different direction. In general, however, he was open to compromise and revealed his personality as an academic teacher and researcher by his particular power of persuasion.

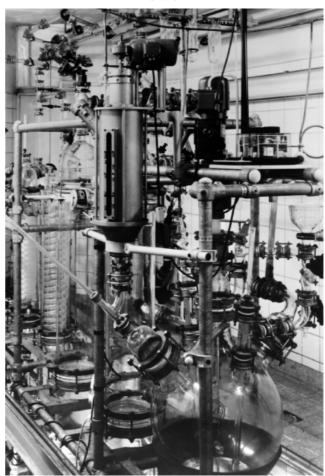
During this period, in addition to the work on the various classes of sulfur compounds, investigations on the higher silicon hydrides were continued with a small group of coworkers. After his retirement in 1971, Franz Fehér concentrated his research activities exclusively to this topic. His objectives were to continue and extend the classical works of Alfred Stock on silicon hydrides (1916–1926) with the help of modern preparative and experimental techniques and to develop a systematic chemistry of the higher silanes. By means of a specially designed high-vacuum apparatus Stock had isolated for the first time from the silicon

hydride mixture arising from the acid decomposition of magnesium silicide, in addition to monosilane (SiH<sub>4</sub>) and disilane (Si<sub>2</sub>H<sub>6</sub>), small amounts of trisilane (Si<sub>3</sub>H<sub>8</sub>), tetrasilane (Si<sub>4</sub>H<sub>10</sub>), and a residual gas containing higher silanes. The relationships with the saturated hydrocarbons  $(C_nH_{2n+2})$  were recognized and the name silanes adopted for the silicon hydrides  $Si_nH_{2n+2}$ . This work was interrupted in the mid 1920's because Stock was expecting an increasing instability of the higher silanes which he felt he could not master with the then available experimental techniques. In the following years, R. Schwarz postulated a mechanism for the silicide decomposition according to which the putative intermediacy of an SiH2 biradical would be followed by the exclusive formation of silanes with unbranched Si-Si chains. However, no experimental evidence was ever presented for this hypothesis. Other methods of preparation (decomposition of magnesium silicide in non-aqueous media or reaction of silicon chlorides with lithium alanate) gave rise to mono- and disilanes but not to higher silanes. During his renewed investigation of this topic in silicon chemistry Franz Fehér was fully aware of the potential applications of higher silanes in the fields of semiconductor technology and surface coatings.

Since the acidic decomposition of magnesium silicide was then the only available method for the preparation of higher silanes and since the amounts in the formed silicon hydride mixture ("crude silane") decreased with increasing chain length, Stock's method to prepare the crude silane mixture required considerable modification as well as a scaling up to a semi-technical process. Thus, it was found in systematic studies that the yield of liquid silanes does not only depend on the rate of decomposition and the particle size of the silicide but also above all on its method of preparation. Furthermore, phosphoric acid was used in place of hydrochloric acid for the decomposition and highly pure nitrogen in place of hydrogen as carrier gas for the volatile reaction products. In particular, however, improvements in the design and construction of the apparatus with use of new inert gas techniques (see above) enabled the scale of the individual experiments to be increased and the reaction mixtures to be worked-up in rapid sequence with a high safety. Improvements over the years allowed stepwise increases in the reactor volume from at first 1 liter, to 5, 8, and finally 50 liters. With an almost technical scale apparatus (Figure 2) at hand, 2.8 liters of liquid crude silane  $Si_nH_{2n+2}$  (n > 2) were prepared from 80 kg of magnesium silicide within 10 days; the thus obtained material was stored in special steel vessels at room temperature.

The composition of the silane mixture formed from the silicide decomposition, which Stock could only determine with difficulty, was now easily analysed by gas chromatography. Thus, it was found that liquid crude silane contains compounds of the silane series  $Si_nH_{2n+2}$  up to pentadecasilane (n=15). The proportions of the individual compounds decrease with increasing numbers of silicon atoms in the molecule. However, in contrast to earlier expectations the higher silanes are not increasingly less stable but in fact

Figure 2. Apparatus for the semi-technical production of crude silane



are rather easier to handle than mono- and disilane on account of their lower vapor pressures. Furthermore, it was demonstrated that the earlier postulated principle of formation of exclusively unbranched silanes (see above) is not in accord with the experimental results. Starting with tetrasilane ( $\mathrm{Si_4H_{10}}$ ) structural isomers with branched silicon chains were identified; hence, the analogy between silanes and alkanes is even closer than was assumed at that time.

Separation of the silane mixture into major fractions was achieved by column distillation and isolation of the various isomers mainly by preparative gas chromatography. It was found that the higher homologues, beginning with the heptasilanes, are no longer spontaneously inflammable. Even higher silanes are solids at room temperature, resembling the paraffins, but decompose slowly in the air. The pure compounds trisilane, *n*-tetrasilane, 2-silyltrisilane (*iso*-tetrasilane), *n*-pentasilane, 2-silyltetrasilane, *n*-hexasilane, 2-silylpentasilane, 3-silylpentasilane, and *n*-heptasilane were comprehensively characterized by physicochemical (melting points, boiling points, enthalpies of evaporation, densities, refractive indices, viscosities, surface tension measurements) and spectroscopic (Raman, IR, UV, mass, PE, <sup>1</sup>H-NMR, <sup>29</sup>Si-NMR) methods. Of these methods nuclear magnetic

resonance spectroscopy proved to be especially valuable for structure determinations.

These results revealed that some of the structural isomers of the silanes exist in very small amounts or are even absent in the liquid crude silane while the isomers with unbranched silicon chains generally dominate. Thus specific synthetic routes were developed for those isomers which were present only in traces or were absent in the mixture resulting from silicide decomposition. Starting points for these syntheses were the richly abundant compounds with small chain lengths. Thus, for example, the hexasilane isomers 2-silylpentasilane and 2,3-disilyltetrasilane were prepared by the reaction of trisilane with di-tert-butylmercury. Analogously, the heptasilane isomer 2,3-disilylpentasilane was obtained from a trisilane/n-tetrasilane mixture and the octasilane isomers 3,4-disilylhexasilane or, respectively, 2,2,3,3-tetrasilyltetrasilane were prepared from *n*-tetrasilane and *iso*-tetrasilane. A spontaneous silane isomerization in favor of the formation of the symmetrical isomer was observed for the first time between the two above-mentioned octasilane isomers. Accordingly, an isomerization of unbranched silanes to the corresponding branched homologues can in general be achieved by heating the substrate with a catalytic amount of aluminum chloride in an aromatic solvent.

The photolytic disproportionation was also investigated thoroughly as a further route to the higher silanes. It was found that the first steps of the reaction are highly selective and that the process is thus suitable as a preparative procedure. Accordingly UV irradiation of *n*-tetrasilane furnished 3-silylpentasilane, while the new branched hepta- and octasilane isomers 2-silylhexasilane, 3-silylhexasilane, 3,3-disilylpentasilane, 3-silylheptasilane, 4-silylheptasilane, and 2,3,3-trisilylpentasilane were generated by irradiation of *n*- and *iso*-pentasilane as well as *n*-hexasilane. All of these products were isolated in the pure state by gas chromatography and unequivocally characterized by spectroscopic methods.

The more highly branched isomers neopentasilane [Si- $(SiH_3)_4$ ] and neohexasilane [ $(SiH_3)_3Si-SiH_2-SiH_3$ ] were each prepared by multi-step reactions starting from silylpotassium (KSiH $_3$ ) and di- or trisilane. The initially formed mixture of more silicon-rich anions was converted to the corresponding phenyl derivatives by treatment with chloro-(phenyl)silane while these derivatives were separable by gas chromatography. Cleavage of the phenyl groups by reaction with hydrogen bromide and hydrogenation of the thus formed bromosilanes then gave the respective pure silane isomers.

The last-mentioned investigations provided the compounds for numerous studies by Fehér's group on the chemical reactivity of higher silanes about which little was known at that time. The objective of this work was to develop the chemistry of the silanes to a close analogy of that of the alkanes with particular interest being focussed on reactions leading to the formation of new Si—Si bonds. This was intended to make the synthesis of more silicon-rich compounds from silicon-poorer derivatives possible and

thus provide a simpler alternative to the laborious preparative route via the crude silane mixture. This topic occupied Franz Fehér up to the end of his active participation in experimental work at the age of 85. The strategy involved the initial synthesis of silane derivatives with suitably active leaving groups which would then react with formation of new Si—Si bonds. In this context, the branched isomers and their derivatives were of special interest in the light of the differing reactivities of the primary, secondary, and tertiary hydrogen atoms of these silanes.

In the class of partially halogenated silanes some already known mono- and disilane derivatives as well as a large number of novel chloro-, bromo-, and iodosilanes with two to five silicon atoms in the molecule were prepared. An important result of this work was the discovery that higher silanes did not react explosively with elemental halogens when the reactions were performed in highly diluted solutions at low temperatures. Cleavage of the Si–Si bonds only occurred to a minor extent under these conditions. Tin(IV) chloride also proved to be a suitable chlorination reagent with which mono-substituted products could be obtained preferentially. The halosilanes formed were then separated from the reaction mixtures by gas chromatography.

Alkali metal silanides of the general composition  $M^{I}Si_{n}H_{2n+1}$  ( $M^{I}=K$ , Na) as a further group of functionalized silanes were of particular interest. During investigations on the synthesis of neopentasilane and neohexasilane (see above) it was found that di- or trisilane reacted with potassium monosilanide (silylpotassium, KSiH<sub>3</sub>) via disproportionation and transmetallation to give a mixture of more silicon-rich anions. In order to make this finding of preparative utility, the known metallation of monosilane with potassium was optimized with regard to the laboratory-scale preparation of pure KSiH3 and transferred to sodium. Then the reactions of the monosilanides with various silanes were studied in detail. In the course of this work, solutions of pure disilanylpotassium (KSi<sub>2</sub>H<sub>5</sub>), 2-trisilanylpotassium [KSiH(SiH<sub>3</sub>)<sub>2</sub>], and 2-iso-tetrasilanylpotassium [KSi(SiH<sub>3</sub>)<sub>3</sub>] in 1,2-dimethoxyethane were prepared starting from KSiH<sub>3</sub> and Si<sub>2</sub>H<sub>6</sub> or Si<sub>3</sub>H<sub>8</sub>. The stability of these solutions increases from disilarly to 2-iso-tetrasilarly but complete decomposition occurs on removal of the solvent. Of preparative significance was the observation that both monosilanides also reacted with SiH4 to afford mixtures of the more silicon-rich silanides  $M^{I}Si_{n}H_{2n+1}$  (n=2-6). These species were then converted to preparatively separable derivatives by reaction with suitable halogen-containing reaction partners such as chloro(phenyl)silane (see above) or alkyl and aryl halides. This opened a new and general route for the synthesis of new compounds containing silicon-silicon bonds starting from the easily accessible monosilane. For example, various partially organo-substituted silanes were prepared in this way (Scheme 9).

Some partially phenylated disilanes and trisilanes were prepared by specific reactions of silylpotassium with appropriate bromo(phenyl)silanes or by reactions of the corresponding iodosilanes with diphenylmercury (see Scheme 10

Scheme 9. 1-Organotrisilane, 1-organo-*iso*-tetrasilane, organoneopentasilane, and 1-organohexasilane

$$R-SiH_{2}-SiH_{2}-SiH_{3} \qquad R-SiH_{2}-SiH \\ SiH_{3} \qquad SiH_{3} \\ SiH_{3} \qquad SiH_{3} \\ R-SiH_{2}-Si-SiH_{3} \qquad R-SiH_{2}-Si-SiH_{2}-SiH_{3} \\ SiH_{3} \qquad SiH_{3} \qquad R-SiH_{2}-Si-SiH_{2}-SiH_{3} \\ SiH_{3} \qquad SiH_{3} \qquad SiH_{3} \\ R=C_{6}H_{5}, C_{2}H_{5}$$

for examples). Because of the shortage of time still available to him Franz Fehér was only able to illustrate the enormous synthetic potential of the various metal silanides on a few exemplary reactions.

Scheme 10. Synthesis of 1,1-diphenyldisilane and 2-phenyltrisilane KSiH3 + (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>SiHBr 
$$\xrightarrow{-\text{KBr}}$$
 (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>SiH $-$ SiH<sub>3</sub>

2 SiH<sub>3</sub>-SiHI-SiH<sub>3</sub> + (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>Hg 
$$\xrightarrow{\text{-Hgl}_2}$$
  
2 SiH<sub>3</sub>-SiH(C<sub>6</sub>H<sub>5</sub>)-SiH<sub>3</sub>

Furthermore it was found that the known reactions of monosilane and disilane with organolithium compounds could be transferred to higher homologues of the silane series. In this way the unbranched butylsilanes  $C_4H_9SiH_2-(SiH_2)_n-SiH_3$  and  $C_4H_9SiH_2-(SiH_2)_n-SiH_2C_4H_9$  (n=1-3 in each case) were prepared from trisilane, n-tetrasilane, and n-pentasilane by reaction with n-butyllithium.

Photolytic reactions of trisilane, n-tetrasilane, iso-tetrasilane, and n-pentasilane in the presence of acetone gave rise to constitutional isomers of the corresponding isopropoxysilanes  $[(CH_3)_2CHO]_xSi_nH_{2n+2-x}$  (x=1-3) which were separated by gas chromatography. Hydrolyses of the preferentially formed mono-substituted products with dilute, non-oxidizing acids then furnished the bis(silanyl) ethers of tri-, n-tetra-, and iso-tetrasilane with retention of the Si-Si bonds.

The special inert gas technique developed by Franz Fehér was used in all experiments with the air-sensitive silanes and was continuously refined depending on the actual requirements. Since Franz Fehér continued to participate in the supervision of advanced inorganic chemistry laboratory courses even as retired professor in order to assist his colleagues in the years with large numbers of students, it often happened that students would specifically apply for a place in his silane laboratory for these courses. They knew that they would then have an opportunity to learn special working techniques that would be useful in their future professional careers. At the same time it was a great pleasure for Franz Fehér to personally instruct these 50-year-younger chemistry students in their experimental work and to provide them with a survey of the state of development and perspectives in a fascinating field of inorganic chemistry. Figure 3 shows Franz Fehér at the age of 84 with two stu-

Figure 3. Franz Fehér in his silane laboratory with two students of an advanced inorganic chemistry practical course (March 1987)



dents in an advanced laboratory course in his silane laboratory.

In addition to his interest in the "element-near" chemistry of sulfur and silicon, Franz Fehér was involved with numerous other topics during his sixty years of active research work. Most of these individual works can be classified into three subject groups:

- i. synthesis and detailed characterization of new compounds in widely differing classes of substances;
- ii. resolving analytical problems that Franz Fehér considered to be an essential part of his teaching responsibilities (see above);
- iii. development of new apparatus or improvement of evaluation methods for widely varying procedures that he employed in his reaseach.

The first subject group includes, among others, numerous investigations of silicophosphorus acid esters, silicothiophosphorus acid esters as well as the preparation of anhydrous hydrazine and the synthesis of germanium hydrides from magnesium germanide in the former compound as solvent. The syntheses of partially substituted, symmetrical phenyldigermanes and phenyl-substituted cyclic silicon-sulfur compounds must also be mentioned in this context. In addition, Franz Fehér carefully carried out comprehensive investigations on the crystal structure of lithium peroxide,  $\text{Li}_2\text{O}_2$ , and the spatial structures of sulfides of sodium and potassium. Furthermore, other studies were directed at the synthesis of  $\text{H}_2\text{N}-\text{NH}-\text{C}(\text{Se})-\text{SeH}\cdot\text{N}_2\text{H}_4$  and its transformation to selenocarbohydrazide,  $\text{H}_2\text{N}-\text{NH}-\text{C}(\text{Se})-\text{NH}-\text{NH}_2$ .

The analytical work was mainly in connection with the investigations on open-chain sulfur compounds and on the properties of sulfur melts. Thus, for example, a gravimetric and a volumetric method were developed for the characterization of sulfanes which, however, only yielded information on the gross compositions of the respective samples. Routine determination of the contained homologues only became possible with the further development of various spectroscopic techniques. In addition, procedures for the titrimetric determination of alkali polysulfides and for the de-

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termination of sulfur in zinc blendes and organic substances by digestion with hydrogen peroxide were described. Numerous experiments were concerned with the analytical characterization of elemental sulfur and, in particular, the quantitative analysis of traces of carbon down to 0.001% (see above). A polarographic method for assaying DDT was devised that was suitable both for trace analysis and for the detection of larger amounts of DDT in technical products. Taken together, the analytical works of Franz Fehér reveal a marked tendency for the application of instrumental methods and especially spectroscopic procedures.

The third group of topics includes among many others the description of a simple projector to measure weak spectral lines which at the time was of relevance for the evaluation of photographically recorded Raman spectra. In connection with the investigations of sulfur melts an adiabatic, automatically controlled calorimeter was designed for measurement of the specific heats of substances with low thermal conductivities. The studies on sulfur radicals at low temperatures required a specially designed apparatus to enable the spectroscopic characterization of short-lived photolysis products. Also, his detailed evaluation of the characteristics of the first direct-recording Raman spectrometer that replaced the old photographic spectrometer in Cologne in 1955 is an illustration of his continuous efforts to optimize the measuring techniques that he employed. The same is true for the interpretation of the respective results with regard to accuracy and concomitant simplicity. Examples of this are the description of a graphic evaluation of Debye-Scherrer exposures and an evaluation procedure for measurements in a differential heat-flow calorimeter for determining reaction enthalpies and rate constants.

The complete scientific works of Franz Fehér to which he was able to devote more than six decades of his life are documented in review articles and more than two hundred original papers, of which the last was published posthumously in 1991. In 1962, nearly thirty years earlier, the Deutsche Akademie für Naturforscher Leopoldina had elected him as a member in recognition of his outstanding scientific achievements. Franz Fehér always felt a close affection for this academic society whose election motto "Nunguam otiosus" he could have called his own since it effectively described the guiding principle of his life and he attended the general meetings even at an advanced age.

Franz Fehér was a prominent German chemist with a particular interest in experimental inorganic chemistry: whenever the properties of a substance permitted, it had to be prepared on a laboratory scale in the pure state and investigated as thoroughly as possible with all available analytical, physicochemical, and spectroscopic methods (including X-ray crystallography). In his research work he repeatedly disregarded the boundaries to physical and organic chemistry, being aware that the division of chemistry into the various fields was reasonable under organizational aspects but of no relevance from a scientific point of view. Many of the results from his work and ideas from his original discussions have long been held to be common scientific

property and are - often in ignorance of their source still widely used. On the whole it can be stated that Franz Fehér and his students made major advances in widely differing fields which have provided decisive impulses for the development of the chemistry of the non-metallic elements.

In the middle of his 85th year Franz Fehér's physical strength began to decline so that his attendances in the institute became less frequent. Even so he still participated intensively in the general scientific life and academic events at the University of Cologne. During a short hospitalization for cardiovascular support he suddenly but painlessly passed away and thus a varied life dedicated with extraordinary consequence to chemistry came to an end.

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Marianne Baudler

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