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Remarks Delivered at the Awards Ceremony
Fourth International Conference on Small-Angle Scattering of X-Rays and Neutrons

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REMARKS DELIVERED AT THE AWARDS CEREMONY

Fourth International Conference on Small-Angle Scattering of X-Rays and Neutrons

held in

Gatlinburg, Tennessee USA
October 3–7, 1977

Edited by

H. Brumberger and R. W. Hendricks

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OAK RIDGE NATIONAL LABORATORY
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PREFACE

For almost four decades, the distinguished trio of professors, W. W. Beeman, A. Guinier, and O. Kratky, have provided leadership to the field of small-angle scattering through both their teaching and their personal research. Collectively, they have authored or coauthored over 400 scientific articles and reviews, several books, and have been associated with over 140 graduate and postdoctoral students. It was thus appropriate that each should be honored for his outstanding contributions to the field at the Fourth International Conference on Small-Angle Scattering which was held in Gatlinburg, Tennessee, in October 1977. The Board of Directors of the Special Interest Group on Small-Angle Scattering of the American Crystallographic Association [H. Brumberger (Chairman), R. W. Hendricks, G. F. Nielson, P. W. Schmidt, B. P. Schoenborn, and L. B. Shaffer] in conjunction with the Conference Organizing Committee, arranged a special awards ceremony during which each honoree presented his personal reminiscences of the development of small-angle scattering in his own laboratory. This report is a record of this event. The master of ceremonies was Professor Brumberger.

We would like to express our thanks to Frances Scarboro for her invaluable assistance in transcribing tapes of the oral presentations and for preparation of the final manuscripts, and to R. L. Stephenson for the photographs.

H. Brumberger
R. W. Hendricks

Honorees at the Awards Ceremony. Left to Right: Prof. W. W. Beeman (University of Wisconsin), Prof. A. Guinier (University of Paris), Prof. O. Kratky (Institut für Roentgenfeinstrukturforschung, Graz), Mrs. O. Kratky, and Prof. H. Brumberger (Syracuse University).

REMARKS DELIVERED AT THE AWARDS CEREMONY

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INTRODUCTION

Professor H. Brumberger

Ladies and Gentlemen, Friends and Colleagues: This is a rather unusual occasion for small-angle meetings and I think a very nice occasion. It is a great pleasure for me to be able to do this. We are gathered this evening to honor three to whom small-angle scattering owes immeasurable debts – as pioneers, as continuing contributors to the field in research, and as teachers whose influence still extends over most of the practitioners, I think, of this somewhat "black" art. Certainly there are a good many of us here who are former students of one or another of the three men whom we are honoring and I suspect that there are a good number of our own students here (scientific grandchildren, as it were, legitimate or not. I think some of the grandchildren might be disowned by their grandparents.) But certainly my students had better know Beeman's paper in *Handbuch der Physik*, they had better know what the Guinier region is, and they had better know how to use the Kratky camera. So certainly their influence is very real and very widely felt. It is a particular personal pleasure to do this because I have in one way or another been associated with all three in some capacity over many years. I would like, therefore, to welcome our three guests and Mrs. Kratky on behalf of all of us and on behalf of the Special Interest Group on Small-Angle Scattering of the American Crystallographic Association. I think that those of us in this room and those associated with us represent a very sizable fraction of the practitioners of small-angle scattering in the world today and so this is a significant gathering for this purpose. Now, since none of you came to hear me I would like to read to you the citation which goes with the award. It is the same for all three:

Fourth International Conference on Small-Angle Scattering of X-Rays and Neutrons — Presented to Prof. W. W. Beeman (Prof. A. Guinier, Prof. O. Kratky) in recognition of his outstanding contributions to the field of small-angle scattering, Gatlinburg, Tennessee, USA, 3 October 1977.

To serve as a continuing symbol of our admiration and indeed our affection for these men as colleagues, as teachers, as originators, we have chosen a Steuben glass "Paul Revere" bowl. We cannot compete with the Nobel prize in our gifts and so what we have is modest, but it certainly comes from the heart.

Now as I just said, you are certainly not here to hear me, so I am going to ask each of our recipients to come and tell us something about his own personal recollections, his own personal memoirs, of his days in this field. For diplomatic reasons we are going to call on our awardees in alphabetical order, so I'll call on Bill Beeman first.

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Prof. W. W. Beeman Prof. H. Brumberger

PROFESSOR W. W. BEEMAN

Thank you very much indeed, Harry, and your committee, for this very generous award. I am most honored to receive it and I am particularly honored to share it with Professor Guinier and Professor Kratky. Thanks also to your committee for providing an evening of wine and reminiscence. I am delighted to be a participant in both activities, but I must object that it seems unnatural that the reminiscing should precede the wine. Let me correct this by being as brief as possible in my formal remarks. In a couple of hours we should all be in condition to reminisce indefinitely.

Brevity notwithstanding, I must be sure to mention the names of the many valued colleagues and collaborators with whom I have worked and who in fact, if not in form, share this award. If my reminiscences seem to consist mainly of name-dropping, it is as it should be.

The beginning of our interest in small-angle x-ray scattering at Madison dates back to about 1945 or 1946. At that time the laboratory was concerned principally with x-ray spectroscopy — the high resolution spectroscopy of electron bands in metals and other solids. A double-calcite crystal spectrometer was the dispersing agent. As I remember it, we were testing two crystals in the parallel or one-minus-one position; someone noticed that a piece of paper between the crystals broadened the rocking curve and we began to search the literature on the very small-angle scattering of x-rays. Paul Kaesberg, my first collaborator in this area, says my version is nonsense, that he had been unwilling to work on spectroscopic problems and in exploring other possibilities came upon the small-angle literature.

In any case, we rapidly learned that we were dabbling in a well-developed field, invented by Guinier, developed by Kratky, and then shown by Hosemann to be an obvious and trivial consequence of quantum electrodynamics. But the possible applications seemed so interesting that we went ahead anyway and, following established custom, decided to re-develop the field with the minimum possible reference to prior sources.

Harold Ritland and Kaesberg worked with the two-crystal diffractometer and later added a third crystal which cut down the background scattering on the wings without greatly reducing the flux incident on the sample. However, the crystal diffractometer seemed to us to be useful only at the smallest scattering angles and with strongly scattering specimens. It was used in a study of the 675 Å spacing in collagen and later, by Dave Dexter, in an experimental check of his calculations on the multiple scattering of x-rays by carbon black. In both cases, measurements at very small scattering angles were essential.

During this period we investigated other possible diffractometer geometries hoping to avoid some of the severe limitations of the crystal diffractometer. Eventually, Ritland and Kaesberg developed the symmetrical four-slit diffractometer in which the first two slits collimate the incident beam and the last two, rotating about an axis through the sample, define the scattered flux. In the first version the distance between successive slits was 30 cm. Later this was increased to 50 cm to permit greater intensities without loss of angular resolution. Still later, a small diffractometer, 10 cm between slits, was constructed. It could be placed in a vacuum box and was used for most of our studies on the scattering from cold worked metals and from gases used as absolute intensity standards.

We have found the four-slit diffractometer to be a simple and reliable device, providing high incident intensities with low parasitic scattering. The 10 cm version in vacuum, used with a rotating anode source, gives an incident flux on the sample of more than 10^9 photons/sec. The background counting rate with sample removed is about 0.1 count/sec. These figures are with slits narrow enough so that data can be taken at less than 1° scattering angle. The four-slit geometry is, unfortunately, less attractive in these days of linear and area position-sensitive detectors since much of the collimation comes after the sample and one cannot collect data at several scattering angles simultaneously.

Late in 1948 we were joined by Bo Leonard and John Anderegg. Ritland and Kaesberg were then completing our first studies on solutions of a number of common proteins, measuring radii of gyration and estimating shape and hydration from the extended scattering curve. We began work on several small plant viruses and soon confirmed their near-spherical shape from observation of the subsidiary scattering maxima.

I believe Leonard and Anderegg were taking data when, contrary to the usual behavior, the counting rate started to go up as the scattering angle increased. Both the early protein and the virus data were taken with a stationary anode x-ray tube and a four-slit diffractometer with 30-cm slit separations, or, as it was dubbed in the laboratory, the 30-cm machine.

By the early 1950's Leonard had completed our first rotating anode x-ray tube. Dexter had assembled the much larger power supply that was needed and the first 50-cm diffractometer was in operation. Although he did not participate in the instrumentation, we had (in much of the early work) the very valuable collaboration of Sid Shulman of the Chemistry Department of the University of Wisconsin. The first application of the rotating anode and the 50-cm machine was to a study of three viruses which Leonard, Kaesberg, Anderegg and Shulman did together. They got quite accurate radii for the viruses, both from the shape of the central maximum and from the positions of the subsidiary maxima. This work also emphasized to us the importance of interparticle interference effects at the smallest angles. These were very obvious for solute concentrations of more than 1%.

Paul Schmidt joined our group about 1950. With Kaesberg, he studied turnip yellow mosaic virus and its nucleic acid-free shell. They were able to show that the latter was a hollow, spherical shell of the same diameter as the virus. In this research both the 50-cm slit machine and also a pinhole diffractometer built by Bill Rothwell were used. The latter instrument had two pinholes which collimated the beam and an annular ring pickup (the angle could be changed by sliding the annular ring back and forth with respect to the specimen itself). The two diffractometers gave reassuringly similar results after slit-height correction of the 50-cm machine data. Later, Phil Geil used the pinhole machine on another virus but by this time we had learned quite a bit (of course with Paul Schmidt's help) about quick slit-height corrections and the pinhole geometry was abandoned. It was inferior to the slit geometry both in incident intensity and background.

At this time we also (Anderegg, Kaesberg, and Shulman) did our first careful work on serum albumin, where in addition to the radius of gyration we made some guesses as to shape from the extended part of the scattering curve, and where we also noted the various strong and obvious effects of interparticle interference as a function of concentration, of pH, of ionic strength and other physical characteristics of the solution. Anderegg, Geil and Kaesberg (1961) working with wild cucumber mosaic virus were, I believe, the first in our group to do a Fourier inversion of the data (with some guessing at the signs of the amplitudes) to obtain the electron density radial distribution function for the virus. Wild cucumber virus, also, is accompanied by a hollow, nucleic acid-free particle, and the difference between the two particles showed up very clearly in their radial distributions.

I am afraid the chronology is now getting a bit out of hand, but other investigations of the late 1950's and early 1960's included the work of Hatch Echols and Anderegg on the reversible denaturation of serum albumin in urea, Art Malmon on catalase, Hank Van Domelen on alfalfa mosaic virus, and Dick White on broad bean mottle virus. Zoltan Sztankay made particularly careful measurements of the scattering from myoglobin and compared the observed radius of gyration to that calculated by Herman Watson from the known crystallographic structure. The agreement was well within uncertainties introduced by the hydration layer about the molecule.

About 1965 our work with biological structures shifted from proteins and viruses to the nucleic acids. Jim Lake studied transfer RNA and showed that it was a quite compact molecule. He also is well known for his iterative slit-height correction program. Pete Connors worked on 5s ribosomal RNA, a much more elongated structure. Stan Bram was able to measure the cross-sectional radius of gyration of DNA and the mass per unit length and found evidence for scattering from the counter-ion cloud surrounding the DNA. Doug Carlson (who is with us tonight), my last collaborator in this field, studied the conformation of DNA under various solution conditions and in complexes with a number of small molecules.

In my remarks so far I have confined myself to the early period of instrumentation and to a brief summary of our work on biological macromolecules. Kaesberg and Anderegg, who have remained at Wisconsin, were major contributors to nearly all of this work. Kaesberg is very active in virology and protein snythesis studies, but no longer uses x-ray methods. Anderegg long ago developed an excellent and independent program of structural studies on viruses and ribosomes about which I shall not attempt to reminisce since my participation was minimal.

I must confess that, personally, I have always felt more at ease with the physics than with the biology of our investigations. Thus, I leave for the last some remarks on the scattering from metals and on absolute intensity measurements.

Actually, one of our first authentic nonbiological or "clean" applications of small-angle x-ray scattering was by Art Tweet in 1952 who scattered from liquid helium and at 4.2 K observed the increase in scattering at small angles characteristic of a liquid near its critical point. This work was not followed up and by 1953 we had become interested in the scattering from cold worked metals first observed in 1939 by Guinier. Our first collaborators in this field were Roy Neynaber, Bill Brammer and Barney Webb. Because measurements at very small angles were not needed and intensity was something of a problem, the 10-cm slit geometry was assembled and used in a vacuum box. Our early experiments confirmed the observations of Guinier although the reversible decrease in scattering with increasing temperature was difficult to reconcile with his cavity model for the scattering. Neynaber, I believe, first noticed the existence of occasional small-angle scattering peaks from annealed metal foils. These forced us to reconsider the scattering mechanism and, after some soul searching in which everyone participated, to suggest a double-Bragg process as the source both of the isolated peaks and the scattering from cold worked foils.

The result was a disappointment in that a number of potentially useful applications of the technique were now impossible or at least much more difficult. On the other hand, puzzles are always fun. That, and the wide variety of experimental tests which could be used to distinguish between the scattering models, made for an interesting period in our laboratory.

Gerry Sharp and Fremont Reizman continued the work for awhile studying porosity in diffusion couples and the kinetics of the annealing of cold work, but it did not appear that our x-ray methods alone could make a sufficient contribution to justify extending the investigations.

About 1956 we began serious work on absolute intensity measurements. The potential applications of such measurements had long been recognized as had the considerable experimental difficulties. Lou Katz began our work and soon decided that the best primary standard would be a gas which could be placed in the identical scattering geometry as the sample of interest. Unfortunately, even with the 10-cm machine, scattered intensities from most gases were low unless the sample was appreciably thicker than the 1 mm which is optimum for aqueous solutions. This forced him to make geometric corrections of uncertain reliability. Katz was able to intercompare air and SF_6 and to check several pure liquids as possible secondary standards. Most of the comparisons made sense to about ±10%, but the scattering from pure liquids was too high, a problem to which we shall return.

Larry Shaffer continued the work and greatly improved our results. Two factors were important. First, several "Freons" turned out to be good primary standards. The molecules are small, the number of electrons is large, and samples only 1 mm thick can be used. Secondly, the use of thin crystalline quartz as sample windows greatly cut down the parasitic scattering. Shaffer was able to check the known molecular weight of myoglobin, the scattering from sucrose solutions, and the relative scattering from several gases, all with an accuracy of about 2%. However, the scattering from pure liquids remained too high.

Finally, Norm Chonacky made very careful measurements on the scattering from water using C_4F_8 as a primary standard. An 8% excess scattering remained which careful calculation showed could be attributed to the continuing unwelcome attentions of double scattering. Thus, in our laboratory double scattering has been a twice-told tale; a remark which someone of greater self-discipline would not have made.

If there is any common thread to the activities of our laboratory I think it is that we used fairly simple equipment whose capabilities and limitations we thoroughly understood, that we tried to take the best possible data within those limitations, and that we made a conscious but perhaps not always successful effort to avoid overinterpretation of the data.

In extending their kind invitations the Committee suggested that tonight's speakers might wish to predict the future as well as reminisce about the past. Prediction is a most risky business which I leave to the more courageous. There is always the chance that a prediction might be taken seriously, or even overinterpreted. But it is no prediction to suggest that, judging from the amount and quality of the research presented at this Conference, the future of our field will be in excellent hands. Thank you.

Prof. A. Guinier Prof. H. Brumberger

PROFESSOR A. GUINIER

It is very rewarding to know that those who are today using and constantly improving small-angle scattering techniques have not forgotten the first modest and timid steps in the field. I am very grateful to the Conference Committee for an award which I deeply appreciate and for the invitation to speak about the beginning of my work. I fear, however, that you may be disappointed by these stories which come from a time when a great number of you were not born, because the first observations of small-angle scattering occurred much more by mere chance than as the result of bright and original theoretical ideas.

In 1935 I started work on my thesis under Charles Mauguin, who was Professor of Mineralogy and Crystallography at the University of Paris. At that time, Jean Laval was studying the background scattering in a powder diagram between the diffraction lines, using an ionization chamber; it was the beginning of his pioneering work on thermal diffuse scattering. Mauguin, well aware of the importance of this quite new field, asked me to approach the same problem with another technique, the photographic method.

In the usual D.S. cameras, the true scattering of the sample was completely masked by a strong parasitic background. To suppress it, the primary beam had to be strictly monochromatized, and nevertheless as intense as possible. I used a bent crystal monochromator, a technique well known in Paris, because J. Laval and also Y. Cauchois, had already used it. The monochromatized beam was convergent; to utilize its full power and to ensure at the same time a good angular resolution, a focusing diffraction camera had to be added to the monochromator. In a Seeman-Bohlin camera, the rays reflected by the surface of the sample cover a long total path. To shorten this path, for the sake of intensity, I used a transmission device, which after some efforts worked well. The powder diagram was obtained with exposure times of the same order as in a D.S. camera.

The pattern was very clear, especially in the low-angle region ahead of the first D.S. diffraction line. This meant that this angular region could henceforth be studied, something impossible with usual cameras.

At first this feature did not appear to be a distinct advantage: while in some rare samples a few lines corresponding to large spacings, not visible in D.S. cameras, were detectable, generally the low-angle region of the pattern of a crystalline powder was completely blank. Furthermore, the intensity scattered by amorphous substances was very weak, reaching a constant value toward 0 angle.

However, at last I found one exception: carbon black gave a diffuse spot at very small angles, much more intense than the diffraction rings at wide angles. This phenomenon had been already observed: in 1929, C. V. Raman, without going into full justification, had explained it by the action of the conduction electrons. B. E. Warren, in the abstract of a paper given at a Physical Society Meeting (1936), very clearly stated that the scattering at small angles was the consequence of the division of the matter into very fine grains, and mentioned that the phenomenon could be used to determine the size of the grains.

It was obvious that the scattered intensity was continuously decreasing from the center, but its maximum value at 0 angle remained unknown. It was not possible to measure either a characteristic half-intensity width or a discrete value of an angle where the scattering vanished. The experimental intensity curve had to be compared to a theoretical function. I worked out a model where identical particles formed a system dilute enough to suppress any interparticle interferences and showed that the scattered intensity could be roughly represented by an exponential function with one parameter: the "radius of gyration" characteristic of the particle size (1937).

Unfortunately, this equation was not borne out experimentally by the only example of small-angle scattering I knew, that of carbon black: there was too large a disparity in the particles sizes. A "good" sample would be a solution of well-defined large molecules. So, on my way to the lab, I bought a fresh egg and immediately took a small-angle pattern of the egg-white, considering it naively as a solution of ovalbumin: in fact, it gave a very fine straight line ($\ell n I, \varepsilon^2$) and the radius of gyration determined in this way was in good agreement with what would be expected from the known volume of the ovalbumin molecule. Afterwards, I studied a catalyst made of small crystallites, the Raney nickel, and found the same value for the crystallite size from the small-angle scattering and from the width of the diffraction lines.

At the beginning of 1938, I paid a visit to two metallurgists working in Paris, J. Calvet and P. Jacquet. The reason for my visit was trivial – I needed thin foils of pure aluminum. But during the conversation, they told me they were studying the "age-hardening of Al-Cu alloys." Of course, I did not know what that was. They explained to me that this phenomenon was not yet understood: the metal spontaneously becomes hard at room temperature and, even with the most careful metallographic examination (P. Jacquet had just invented the electrolytic polishing of metals), no change of structure was detectable. The generally admitted hypothesis was that the hardening was due to a very fine precipitate, below the resolving power of the microscope (at that time, the electron microscope was not yet invented).

It seemed worthwhile to try to examine these samples with the small-angle technique. The very first pattern (Fig. 1) was a great shock for me: instead of exhibiting, as usual, spots or lines, it showed streaks of varying directions and lengths starting from the center. The streaks changed when the specimen was rotated: I easily found that the maximum moved in the same way as a light beam reflected by a rotating plane mirror. I also found that these small mirrors should be parallel to the (100) reticular planes of the Al crystal. So I guessed that these mirrors were formed by the segregation of copper atoms out of the supersaturated solid solution. I had been fortunate that the grains in the alloy of Calvet and Jacquet were just of the right size. If the grains had been microscopic, the first experiment would have

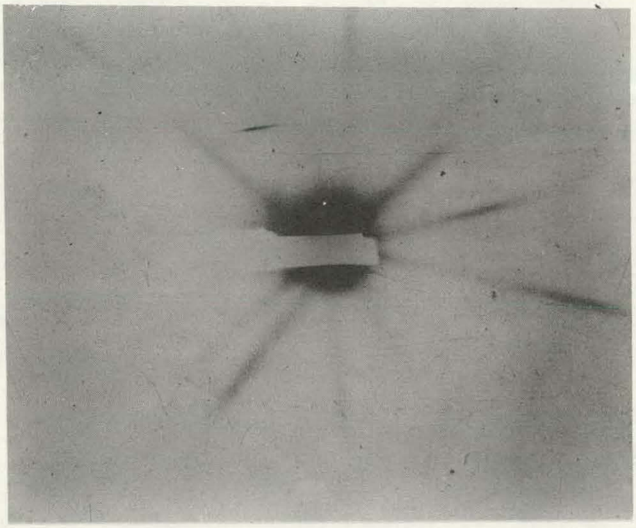

Fig. 1. First evidence for the existence of planar zones in an age-hardened Al-Cu alloy: CuK_α radiation, coarse-grained specimen (1938).

been negative and there would have been no reason for me to proceed any further in that direction.

I immediately published this result in the *Compte-Rendus*. A few weeks later, G. D. Preston published similar observations, giving the same explanation. Our two papers were to be presented at the annual meeting of the Institute of Metals in Scotland at the beginning of September 1939: but because of the War, there was no meeting. So I never had the opportunity to meet Preston, who, after the War, changed his field and I never discussed our respective works with him.

During the summer of 1938, I began to write my thesis: this was interrupted by the Munich crisis and the partial mobilization. I remember putting all my manuscripts, laboratory notebooks and photographs in a wooden case. When I closed it and wrote my name on the cover, I knew that there was a more than negligible chance that all this work would be completely lost.

As it turned out, we had a few more months before the War, and I was able to finish my thesis. I came back to the laboratory in 1940 and, as you can imagine, the work conditions in occupied Paris were extremely bad. However, with some very limited means, I continued the study of pre-precipitation in alloys, Al-Cu, Al-Ag, Cu-Be.

After the War, G. Fournet joined my laboratory: he worked on the theory of small-angle scattering and built a very elaborate device with a double monochromator. In the following years, our group became more interested in diffuse scattering in general: the small-angle region was considered only as a small, but interesting part of the reciprocal space.

Among all these remembrances, some emerge with a particular insistence. First of all, the material conditions of laboratory work in the 1930's: they must look incredible to a graduate student of today. Our building was not intended for housing a laboratory; the rooms were uncomfortable and overcrowded, and at the same time much wasted space was taken up by apparatus out of use. Everything was dusty because sweeping was a rather infrequent operation. Our budget was miserable and, of course, in every domain, the techniques were far removed from what we are now accustomed to.

On the other hand, the degree of my ignorance was surprising by present standards, a situation quite common at that time for students beginning their thesis work: I don't consider that I was an exceptionally bad student. I was a physicist and knew nothing about crystallography or metal physics. I had no idea what a reciprocal lattice was or what the relations were between diffraction and Fourier transforms, and so on.

Men of my age have witnessed the considerable progress of science in 40 years. More important than the obvious technical advances is the increase of the general theoretical knowledge: not only do physicists now have better tools in hand, but they understand better what they are doing.

In spite of our difficulties, and even in the worst days, we have been happy through science. Perhaps we belong to a privileged generation which has found immense intellectual satisfactions in science. We have been happy through learning, happy through understanding, and even on some rare occasions, through discovering.

And now ... we are glad to see young physicists working in pleasant laboratories, having at their disposal sophisticated apparatus; their scientific environment is often very good, and we are delighted that they are able to benefit from all these favorable conditions to achieve remarkable progress. But what I wish them most sincerely is that our old recipe for intellectual happiness will never be lost.

Prof. O. Kratky   Mrs. Kratky   Prof. H. Brumberger

PROFESSOR O. KRATKY

Mister Chairman, Ladies and Gentlemen: At the cradle of x-ray small-angle scattering research stood the observation, made by several investigators, that fibers and very fine powders show a diffuse scattering phenomenon very close to the primary beam. The qualitatively correct interpretation was that these effects are due to inhomogeneities in the range of colloidal dimensions. My first contact with this effect occurred in the course of wide-angle x-ray studies on deformation processes in fibers. This was in 1937, and it was also the beginning of my activity in the field of small-angle scattering, which still occupies me today.

At that time the principal question was whether or not the mentioned effects on fibers could be interpreted in terms of Guinier's particle scattering. My opinion was that one had to distinguish between *dilute systems*, for which Guinier's theory is valid, and *densely packed systems* which have to be treated in an entirely different way. At the end of this discussion, with special view to cellulose fibers, stood the theory of small-angle scattering for blocks of parallel lamellae of varying thickness, where the lamellae within each block are strongly interfering.

For me it was clear from my studies on deformation processes that the supermolecular units are lamella-like in shape. In the densely packed system with only about 1% holes, the lamellae cannot be arranged in another way than parallel so that the idea of this model was inescapable. A second perception we had very early was that the scattering curve of a lamella-like particle could be split into a "platelet-factor" $1/\theta^2$ and a thickness factor corresponding to the mass distribution perpendicular to the plane of the lamellae. This thickness factor is obtained simply by multiplying the scattering curve with θ^2. Then the steep increase of the scattering curve at small angles which could lead to the suspicion that this effect is mainly caused by a polydispersity with broad distribution curve diminishes. In the case of cellulose fibers we observe a broad maximum, which is similar to a broadened Bragg reflection. By an evaluation under the above assumptions we got an average thickness and an approximation to the thickness distribution which is relatively small. This result was not generally accepted.

Somewhat later (1952 and 1953) we took another approach. We investigated *regenerated cellulose* whose volume, by uptake of air, was increased approximately sixfold, so-called "air-swollen" fibers. It could be assumed that the interparticle interference effects were now strongly decreased due to the loss of their parallel alignment achieved by air-swelling, which eliminates the cohesion between neighboring particles. As a matter of fact, it can be shown that flat particles show practically no interference effect if they are not arranged parallel.

For these *dilute fibers* we arrived, on the basis of an interpretation by particle scattering from single lamellae, essentially at the same results as previously with the *densely packed* fibers. Thus, the problem was solved starting from either end by different approaches.

It was especially satisfying that on the basis of absolute intensity measurements — a point which I will treat in more detail a little later — the averaged mass per unit area of the lamellae was in excellent agreement with the result found from the shape of the scattering curve. It was also very pleasing to see that Porod's theory allowed the determination of an inner surface which fitted all other results quantitatively.

This success encouraged us to continue the work in this direction. We performed investigations on various other solid high polymers. I would like to mention particularly joint studies with Prof. Brumberger on nylon, which gave interesting new insights such as the determination of the crystalline and amorphous fraction from the absolute value of the invariant. These measurements were extended down to very small angles, corresponding to Bragg values of 3000 Å.

Apart from the solid high polymers, we have been working from the beginning — that is, for several decades — on the particle scattering theory valid for dilute systems. Looking back over the period since 1946, I must say that, of all the theoretical results obtained in our group, three advances seem to me most significant and particularly pleasing.

First, in 1948 we realized that the scattering of *elongated particles* can be split into a rod factor, $1/\theta$, and a cross-section factor. Thus, the cross-section factor is obtained from the scattering curve by multiplying by θ, and the resulting scattering curve can be evaluated in an analogous way to the particle-scattering curve with the only difference that all results relate to the cross section. (I have already mentioned that with platelets one has to multiply by θ for a second time, and then one obtains the scattering due to the thickness structure.) *Native silk fibroin* in solution tends to re-associate progressively, and it provided an example for exercising the measurements of rod-like, ribbon-like, and lamella-like particles up to the point where they become too large to be analyzed.

The second important finding I want to mention came in 1950. It was the realization that one can obtain a *molecular weight* from the so-called absolute intensity. This is the ratio between the scattering extrapolated to 0 angle and the primary intensity. The very first measurements with this aim were performed in the same year on gold sol, then in 1952 on cellulose, in 1954 on silk solutions and later routinely on all investigated biological macromolecules.

In an analogous way to the molecular weight determination, it is also possible for rod-like and lamella-like particles to determine the *mass per unit length* and the *mass per unit area* from the absolute intensities of the cross-section factor and the thickness factor, respectively. I do not know of any other method which can achieve anything similar. The theory was given in 1953; the first measurements on chain molecules to determine the mass per unit length were made in 1956 on cellulose nitrate.

Particularly impressive in this respect were the experiments of Pilz and Sund on *glutamic acid dehydrogenase*. The molecular weight increased with increasing concentration due to the formation of oligomers, but the cross-section curve remained exactly constant. This was overwhelming evidence for the longitudinal association of elongated particles. All results fitted together well. Thus, the total particle masses were proportional to the lengths of the particles as was to be expected from this model.

In another case we were able to define the types of association products formed by a number of *dyes in solution*. Starting from the single molecule it was possible to analyze rod-shaped, ribbon-shaped, and lamella-shaped particles with respect to their dimensions and masses. It was also partially possible to interpret the results from a kinetic point of view.

The third result which appears to me as particularly remarkable is the following: the small-angle method is not only capable of determining the cross section and mass per unit length of a chain molecule, but is also capable of providing information concerning the *shape of coiled chains*. Already in 1946 we were able to postulate the model of a worm-like chain and to solve its scattering behavior, first by analytical means, later on by statistics. I think the most important result was the finding that we can give a parameter for the degree of coiling by the *persistence length*. The impressive fact here is that the x-ray scattering results provide an average over the degree of coiling at all sites within the molecules, irrespective of molecular size and irrespective of eventual chain branchings.

The determination of masses leads immediately to the experimental problem of determining the *primary intensity*. Apart from an older photographic method, we developed an instrument in 1956 which in laboratory terminology is called *rotator*. With this device it becomes possible by mechanical attenuation to measure the primary radiation which is too intense for any direct measurements. It works on a principle which is so surprising that I want to explain it: A disc several centimeters in diameter with very small, barely visible holes, shields off the primary beam. If this disc now rotates (say with 50 cycles/s) the holes cross over the line-shaped primary beam and only during the passage of such a hole x-ray quanta can pass through. Most people, including physicists, who hear of this instrument think intuitively that during each passage a whole group of quanta will pass through. Although this would still lead to an attenuation the counter tube would still be unable to resolve such a group of quanta into single pulses. The surprising thing is now that with suitable dimensioning of the disc, only in a fraction of all passages (say at each tenth or twentieth passage) one quantum passes through. And now one can really calculate the primary intensity from the ratio of the passages at which a quantum passes through. It was then only obvious to prepare a secondary standard. These were the polyethylene platelets ("Lupolen").

After very lengthy and laborious groundwork, we are now developing a very small and much faster rotating device which requires much shorter time for the determination of the primary intensity and is more precise than the previous model. I regard the finalization of this project as an important task for the near future. If all works out according to our intentions the secondary standard will become redundant, and one will then be free from several sources of error connected with the calibration of the secondary standard (scattering power and absorption) and the determination of the absorption of the sample.

After having begun to perform molecular weight determinations, we were also confronted with the problem of determining the partial specific volume which is necessary for the evaluation of these parameters. The most accurate way is to calculate it from the density of the solution. The hitherto known procedures were not accurate enough for this task. We succeeded, however, in designing a method which makes it possible to measure densities with an accuracy of the order of 10^{-6}. In this context I want to refer to the excellent work of my colleagues, Leopold and Stabinger. Obviously, with this device all mass determinations (be it on particles as a whole or mass per unit length with chain molecules) become much more accurate than before. The method opens up entirely new possibilities which one could not have dreamed of before.

Measurements of the concentration dependence and temperature dependence of the partial specific volume as they were performed by Pilz and coworkers are no longer a problem. Another interesting application is the recent study of the liquid-crystalline phase transition in phospholipid-bilayers by Laggner and Stabinger in our group. In this case the unique accuracy of the method becomes particularly apparent: the magnitude of the temperature induced volume change is only in the order of 10^{-4}. The resolution of these changes to an accuracy of 1% requires necessarily a density determination with a precision of about 10^{-6}. These results in combination with x-ray small-angle measurements gave a far-reaching description of the structural changes. Dr. Laggner will report on these studies later this week.

Our intention has always been to contribute as much as possible to the development of the small-angle scattering technique to a precision method, since the instrumental deficiencies have frequently been a bottleneck in progress. Many of you know the small-angle camera which was developed in our group (1954). The most precise experiment which we have so far performed with this instrument was the study of glyceraldehyde-3-phosphate dehydrogenase together with Durschschlag and Puchwein in cooperation with Kirschner and Schuster, two scientists from Manfred Eigen's group. These investigations were performed to clarify whether the saturation of the enzyme by the coenzyme follows a sequential or an allosteric mechanism. Under complete saturation a volume contraction of 7% was found from the invariant according to Porod's relation. If a sequential mechanism were operative, one should expect a linear relationship between volume contraction and degree of saturation. In the case of an allosteric mechanism, deviations from linearity are possible. To make a decision possible, it is obviously necessary to follow volume changes in the course of saturation with a precision better than 1%. Quite clearly, an *absolute* volume determination with this accuracy is out of reach. Here, however, we are dealing with *relative* measurements on the same molecule under slightly varying conditions. The results indicated convincingly an allosteric mechanism. This has also been found independently by kinetic experiments and other studies.

Recently the idea for a novel type of small-angle camera was born; it was constructed in collaboration with Dr. Stabinger. It provides several advantages over the older type. For the lack of time I cannot go into any details. I only want to mention that Prof. Leopold is on the way to developing a microcomputer, which performs the continuous automatic optimization of the exposure conditions. It should make our work not only simpler, but should also bring about a considerable gain in measuring time.

Finally, I want to refer to the *cone-camera* which was also developed in our group, in which the primary beam forms the envelope of a very sharp cone and where the scattering is measured along the axis of the system. The measurement is *practically free of collimation effects*, and the camera is particularly suitable for measurements of weak scattering effects at relatively large angles. This is just the region of interest for the determination of relatively small persistence lengths of chain molecules and in this sense Wrentschur, Zipper, and others have made the first important applications of this camera. They will present these results at this conference. In the future it is intended to use this instrument for the measurement of the very weak side maxima of biological macromolecules; this knowledge is so important for the refinement of the shape determination.

There remains not much time to report on the many measurements on biological macromolecules in the investigation of which we were predominantly engaged for more than 20 years. Allow me to select one example which seems to me typical for the kind of evidence which small-angle scattering can give in this field. We studied immunoglobulin GI and its interaction with haptens. This substance has a pronounced cross-section factor which, however, produces a Guinier plot which is composed of two linear regions, and it is not possible to interpret this fact with the help of simple elongated particles. Following the chemical finding of Edelmann and Gall, with whom we cooperated in this investigation, we found a composite model body which was equivalent in scattering to the real molecule. It may be compared with a flying bird having stretched its wings perpendicular to the longitudinal axis of its body. The biochemists call the two wings the F(ab)-chains, the body the F(c)-chain. Now in cooperation with Sela, Licht and Karush, we studied the interaction of certain immunoglobulins with specific hapten-molecules which (this is known from biochemical evidence) occupy sites at the ends of the "wings." Now as a consequence of the interaction with haptens the molecule as a whole shows a contraction of its longest dimension by several percent. We also looked at the binding of hapten to the isolated fragments F(ab) (one single wing) and its dimer (both wings) and found no effect upon binding. From this, we conclude that the conformational change found for the antibody as a whole occurs in the hinge zone between the single chains (where the "wings" are attached to the "body"). This means that the influence of the haptens propagates from the end of the "wing" along the peptide chain to the hinge zone.

This conclusion could be directly confirmed by opening the two sulphur-bridges within the hinge zone. Now indeed the effect of hapten-binding (contraction of length) is reduced to about one-third of the value obtained before opening the bridges.

Well, these few selected examples of our work, especially in the field of the experimental technique have, for lack of time, to stand for all we have done from 1937 until now. So I leave the discussion of our work and will devote the last minutes of my speech to saying a few words about my history over the past 40 years and the organization of small-angle research in Graz. From 1937 until 1945 I was active in different positions in Vienna, Berlin and Prague. In 1946 I became head of the Institute of Physical Chemistry at the University of Graz and have founded there an x-ray small-angle group which I guided for 26 years. After my retirement in 1972, the Austrian Academy of Sciences offered me the chair of the Institute of X-Ray Structure Research, a position which I still hold. After my retirement, the small-angle research at the university institute has continued, and some of my coworkers at the university have come with me to the new institute. Legally this institute has no connection to the university. Nevertheless, there exist close relationships on a collegial level with cooperative projects.

All small-angle investigators of either laboratory have started out as my coworkers. Several of them became independent excellent scientific personalities. The most important names are listed alphabetically in the following table.

My most important collaborators
1937–1977

H. Durchschlag
O. Glatter
O. Haager
S. Heine
M. Herbst
H. Janeschitz-Kriegl
L. Kahovec
W. Kreutz
H. Ledwinka
H. Leopold
P. Laggner
G. Miholic
P. Mittelbach
K. Müller
H. Oelschläger
B. Paletta
I. Pilz
G. Porod
G. Puchwein
P. Schmitz
A. Sekora
Z. Skala
H. Stabinger
H. Wawra
E. Wrentschur
P. Zipper

Time does not allow me to mention the achievements of each individual personality. I only want to say a few words. For the Graz small-angle group it was crucial that Porod worked with us for 14 years: starting with his thesis in 1946 until he became full professor in 1960. I refer especially to his most important and widely known theoretical work. And the many excellent measurements especially on biological molecules by Prof. Pilz have provided the experimental substance. The achievements of Prof. Leopold, however, are in a different direction. He has led all the work in measurement control technology and he is an authority and creative investigator in electronics and its applications. Dr. Stabinger merits credit as a highly inventive designer of precision instruments.

During my last years at the University my coworkers Zipper, Durchschlag, and Wrentschur have made very important contributions to our work and the activities at my new institute would be impossible without Stabinger, Laggner, Müller, and Glatter.

Finally may I point out that my coworkers at all times have been extremely cooperative and that no single one has left us in disaccord. May this remain so until the end of my days! For all this I want to express to them my most cordial thanks and I want to point out with particular emphasis that I regard the award, which I received today, as an award of honor for the whole Graz group.

My particular thanks are due to the Special Interest Group on Small-Angle Scattering of the American Crystallographic Association for this high distinction. It is not given on the basis of a general image; here the experts in this field are the ones who have awarded me this honor. For this I want to thank you, dear Prof. Brumberger, as the chairman and all the members of the committee very cordially.

And last but not least I want to express my thanks to my dear wife. She has no knowledge whatsoever in the field of small-angle scattering and, therefore, was not able to help me directly. But she has endured through 35 years of x-ray small-angle scattering and has made great sacrifices for my work. For this I want to thank her wholeheartedly on my great day of honor.

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