CONTRIBUTED PAPERS

A NOTE ON USING R-AXIS RAPID Rapid X-ray Diffraction Measurement Using "RAPID" IP Weissenberg Camera

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Introduction

The R-AXIS RAPID that was put on the market in 1998 is a next-generation Imaging Plate (IP) Weissenberg camera to succeed the R-AXIS lics. Both systems were developed jointly by Professor Yuji Ohashi at Tokyo Institute of Technology and Rigaku Corporation. This relationship fortunately led myself to participation in the development of the two systems. As for my experience and measurement with the R-AXIS IIcs, I reported them in an 1995 issue of the Rigaku-Denki Journal (Japanese edition). Now, four years after that occasion, it is a happy coincidence for me to write again for the same journal.

In the same year 1995, my related article appeared also in a special issue of the journal of the Crystallographic Society of Japan. It was entitled "Recent Development of X-ray Crystal Structure analysis by a New Diffractometer for Rapid Data Collection" [1]. This year I have had a further opportunity of contributing my article to this journal under the title of "Time-Resolved Observation of Solid-State Reaction by Rapid Data Collection and Structure Analysis" [2]. Indeed, chances come one after the other. In the present case, I would like to write a note on using the R-AXIS RAPID as a complementary story to my paper carried in the above academic society journal.

Development of IP Weissenberg Camera

The primary purpose of developing the IP Weissenberg camera was to conduct a rapid measurement. What I always cite as an example in this regard is the crystalline state reaction of a cobaloxime complex. It is known that when visible light are applied to this complex in solidstate, the Co-C bond will be broken to produce the radical species, leading to racemization and isomerization of this complex.

Interestingly, there are instances that such a reaction proceeds while maintaining the single crystal form. It is called the crystalline state reaction. The single crystalline state thus maintained makes it possible by X-ray structure analysis to elucidate the structure existing before and after the reaction. It is a very interesting case tantamount, as it were, to direct observation of the progress of a chemical reaction with one's eyes. It is about 20 years now since the crystalline state racemization of a cobaloxime complex was first discovered. Luckily (?), on that occasion, because the complex went through the reaction over the duration of more than two weeks, this reaction could be pursued sufficiently step by step even via the data collection using a four-circle diffractometer. Since then, however, with the advance of the relevantrstudies, there also surfaced such crystals whose reactions will finish within only 10 hours or so. This made it no longer practical to use the four-circle diffractometer whose data measurement time of a single data set takes several days. Following crystalline state reactions is difficult if they finish while measurement is still continuing. This is the reason why the development of a rapid data collection system was started around the year 1990.

The Initial IP Weissenberg Camera

At that time a camera using a flat IP [3] was already available commercially for protein crystallography. On the other hand, regarding the IPD-WAS [4] developed at the Institute of Physical and Chemical Research, a cylindrical IP was employed instead of a flat one, since rapid measurement of small molecular crystals was of sole concern in this case. Unlike protein crystals, small molecular crystals are measured with MoK" X-ray and reflections at 2 2: 50E or 60E can be measured. For reflections at such high angles, the incidence of X-ray

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to the flat IP is made fairly aslant to result in thin and elongated spots. However, with the cylindrical IP, no such problem of slant incidence occurs in the circumferential direction of the cylinder. Moreover, the IPD-WAS enabled measurement by the Weissenberg method (IP translation synchronized with crystal rotation) in order to record maximum number of reflections on a single IP while avoiding overlap of reflection spots. The unique point of this device is that with a variable Weissenberg screen which looks like a human's rib, it permitted various measurement modes using the screen, such as recording of an optional layer line alone, measurement of space which is as independent as possible, and so on. To implement the Weissenberg method, the development of sophisticated goniometer and software was needed for crystal axis alignment. Thus the above device became the predecessor of IP cameras that followed.

R-AXIS-IIcs

It was about the time the IPD-WAS was put to operation that I was employed to work for Professor Ohashi. Right after that, I was permitted to take part in the project of developing an advanced, next-generation system to succeed the IPD-WAS. At the Institute of Physical and Chemical Research, while engaging in measurements of complex crystals with the IPD-WAS, I was contemplating the design of the new system. This project bore fruit as the R-AXIS IIcs [3]. This system is basically an IP Weissenberg camera provided with a cylindrical IP similar to the IPD-WAS. A larger IP was employed for the R-AXIS llcs to allow recording of $20\pm60E$ reflections, instead of $\pm45E$ with the IPD-WAS, in the circumferential direction of the cylinder. This modification was intended to measure some more reflections in the high angle side for the small molecular crystal of good crystallinity. The recording range for the axial direction of the cylinder was set to from $22+60E$ to $-30E$. This range was also determined by taking the maximum measurable angle into account. When a test run was started after the system was built up, I happened to know practically none of reflections recorded in areas at around $20+62E$ of axis direction, because of the slant incident problem. It made me upset, and I hastily made a request for adaptation to shift the IP position to a range from $+55E$ to $-45E$.

Automatic crystal axis alignment for the Weissenberg photograph had remained a pending issue up until that time. The R-AXIS IIcs resolved it by realizing automatic axis alignment mechanism thanks

to its 6 type 3-circle goniometer and excellent axis alignment software. As for the variable Weissenberg screen, one of the characteristics of the IPD-WAS, it was omitted from the design to simplify the system construction. While it was a pity that the number of measurement modes was reduced as a result, this omission must be said at that time to be a permissible compromise, since the operation became easier that much. Nevertheless, from the present-day viewpoint where long-hour exposure for fairly tiny crystals tends to be conducted more and more often, it is also true that we now wish to get back that screen in order to decrease the accumulation of backgrounds.

The R-AXIS IIcs was much welcomed at our laboratory and was used for a variety of crystalline state reaction studies. At that time, priority was given to the measurement time, so the routine procedure was to rotate the crystal with a single axis and thereby to measure the independent reciprocal space by the Weissenberg method within a few hours.

The rapid measurement was conducted not only for the crystalline state reaction but also for the interesting molecule whose structure had long remained unknown because of the crystal deterioration by X-ray irradiation, and for the crystal that suffers from the rapid elimination of a crystal solvent. Further, thanks to the advantage of a two-dimensional detector (IP), the R-AXIS IIcs displayed high performance in analyzing crystals whose spots tend to broaden due to poor crystallinity as well as in measuring tiny crystals. This reminds me of my surprise when I saw a student in the master's course. He carried out structure analysis of about 20 crystals in just a month regarding a series of crystals, thus thoroughly investigating the correlations between the crystal structure and reactivity. I was impressed with it and felt the arrival of a time in which the X-ray structure analysis is used just like IR and NMR used for structure studies.

Development of R-AXIS RAPID

Since fiscal year 1996, professor Ohashi's group, participating in the Core Research for the Evolutional Science and Technology Program (CREST) sponsored by Japan Science and Technology Corporation (JST), has embarked on studies that need rapid and precise measurement of X-ray diffraction data. The assigned task is the observation of the molecular structures in the excited state by the X-ray diffraction techniques. In parallel with the development of a Micro Strip Gas Chamber (MSGC) detector required

for the purpose, a diffractometer using a CCD detector (Siemens SMART system combined with Rigaku rotating anode X-ray generator UltraX-18) was brought into our laboratory. As this diffractometer displayed as high performance, it was then evident that the old-generation IP Weissenberg camera could no longer compete with it. This reality was partly because the R-AXIS IIcs installed in our laboratory was a prototype and was becoming rickety. Moreover, the IUCR Conference held at Seattle in 1996 produced an atmosphere as if the age of the IP camera was over. Despite such surroundings, however, our senses experienced with the IP camera told us that this camera should still allow performance upgrading; at least it should be able to become comparable with the CCD camera in performance. It was in this context that in 1997 we mapped out the creation of an ultimate IP Weissenberg camera based on our know-how so far accumulated along with the latest technology. That is, the development of the R-AXIS RAPID was decided to realize rapid and precise measurement of X-ray diffraction data.

What was aimed at in terms of performance was the measurement of high-angle reflections whose diffraction angle exceeds 60° and also high-speed readout of the IP. The R-AXIS IIcs was incapable for high-angle reflection measurement. Such measurement is necessary, however, when CuKα X-rays are used (a measurement up to 135° is required for contribution to the Acta Crystallographica) and also to enable measurement of inorganic and mineral crystals and measurement for the electron density analysis with the MoKα X-rays. This requires a fairly large IP leading to the use of a single IP and a high-speed readout system instead of two lPs used alternately as with the RAXIS IIcs. In terms of operational productivity, the main goal was an AUTO mode that permits measurement by automatically determined strategy, as well as smaller space for installation (specifically because professor Ohashi's laboratory became cramped for room besides the general recognition of the importance of smartness for future instruments).

A memo I jotted down in 1997 when the development began shows a sketch of a vertical system with a cylindrical IP. I was considering a IP of ±150° in the circumferential direction of the cylinder. This means that the X-ray beam was to be taken in through the remaining 60° gap. I was also studying the same IP reading mechanism as with the R-AXIS IV. These ideas of mine turned out to be not wide of

the mark, though not exactly. (it made a good hit so far as the vertical cylinder part is concerned.)

The practical IP size we finally came to was +140° through -60° in the circumferential direction and ″45° in the axial direction. In view of attaining compactness, moreover, we made the cylinder axis vertical, resulting in up-down motion of the IP. Despite a single IP in use, the latest high-speed IP readout techniques permit the duty time of less than 120 seconds. This duty time is roughly the same as that of replacing the two IPs. In addition to the speed, it has become practicable to get better quality data than with the R-AXIS IIcs owing to the latest electronic circuitry and others.

As for the goniometer, it has evolved from the φ type to the newly designed 1/4χ type, which is a 1/4 version of the χ circle often seen in the four-circle diffractometer. Compared with the φ type, this 1/4χ type has less mechanical blind region (an angular region inaccessible by the goniometer). Moreover, even in the case that the goniometer's shadow is casted on the IP, the area of the shadow is simply predictable, so the data processing to exclude the shadow region is easy. Actually, the degree of freedom has become smaller than that of the φ type but the 114χ type has many advantages to compensate for it well enough. This is why we regard this goniometer,as a masterpiece. (It is safeiy operable with no fear of hitting the X-ray collimator or beam stopper when rotating ω with χ, φ at any angle.) Regarding the measurement strategy, a multi-scan method is adopted such that in the standard measurement mode, a series of ω scan measurements are performed for plural times by varying the crystal orientation instead of one-axis rotation for measurement of the independent space as has been before.

This multi-scan method can eliminate a blind region (resulting from no crossing of the diffraction sphere by the reciprocal lattice point in the rotation axis direction), thus attaining 1.0 in completeness. It can also obtain high-precision data by averaging equivalent reflections with a data set having high redundancy. In this way, when the measurement is conducted by this method, each reflection will be measured for plural times in different crystal orientations to enable empirical absorption correction by utilizing the orientation information. As for the absorption correction by integration method, which is required for the crystal of large absorption coefficient, this type of correction can be implemented by

automatically indexing of the crystal plane using the photographs of the crystal taken by the CCD camera. Such absorption correction technique was not available with the R-AXIS IIcs. The remarkable progress is not merely the matter of hardware. Substantially advanced software is also incorporated (e.g. the AUTO button is provided). Fig. 1 shows a photo of the system and its diagram.

Measurement with R-AXIS RAPID

The measurement procedure with the IP camera is almost identical with the four-circle diffractometer. Crystal centering is to be made, to begin with, by

Fig. 1 R-AXIS RAPID. (Top) X-ray generator, $3P$ goniometer, IP (IP cover is removed for photography), low temperature attachment nozzle. (Bottom) diagram.

watching on the PC screen where the crystal's video image is photographed with the CCD camera. This way of observation is easier than using a telescope once the operator gets used to it. Digital zooming and image capturing are also possible.

Crystal Orientation Determination: In the standard procedure, the crystal orientation is to be determined by indexing two oscillation photos (oscillation angle: $5E$) [6]. When the correct lattice parameters are obtained, the positions of about 80 percent or more of recorded reflections should be explained by this orientation matrix. In case lattice determination cannot be made properly, this difficulty may be resolved by, for instance, extending the exposure time for oscillation photograph or by taking one more photo at a different crystal orientation. The former way may be effective in the case that the initial photos were taken with a fairly short exposure time for time saving. In the latter way, a photo taken at $T=45E$ or $P=55E$ can be added to the photo of $T=0$ and $90E$ so as to increase the number of reflections. The final decision on the Laue group is to be made after the data collection by examining the intensity distribution of numerous reflection data. But, to some extent, it is also possible to decide on the crystal system from lattice parameters, so such a decision may be utilized also to determine the measurement strategy.

Measurement of Intensity Data
! with MoK" radiation!

For the measurement method, selection should be made between the Weissenberg photograph method and the oscillation photograph method. The oscillation photograph is the standard method and the measurement strategy is determined automatically. However, it is possible for the operator to alter the strategy according to the purpose of measurement, that is, if it is rapid measurement or ordinary one.

1. Standard Measurement: In the stan-dard measurement by the oscillation photograph method using the MoK" X-rays, 44 oscillation photos whose oscillation angle is $5E$ are to be measured with no overlapping, using two crystal orientations. This means that roughly half reciprocal space is measured in standard mode. Completeness of 1.0 can be attained for almost all crystal systems in this way, and the redundancy comes to more than 7 for the orthorhombic system, although the value may some-what differ depending on the crystal system. Some reflections are recorded over two IPs (partial
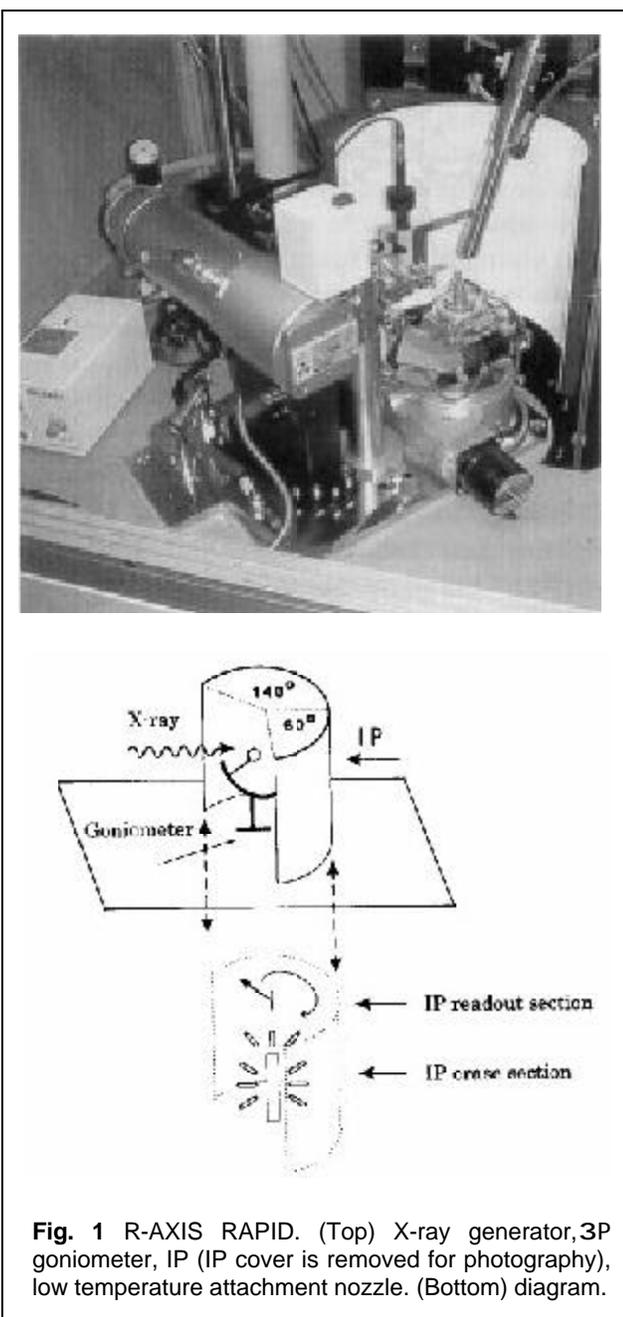
C$_{30}$H$_{36}$O$_4$N$_5$Co, 0.26x0.20x0.16mm

[R-AXIS RAPID]

MoK″, 50kV-50mA, 5x 55 frames

Measurement time: 6.5E hr

orthorhombic, P2$_1$2$_1$2$_1$, a=12.614(1), b=26.782(3), c=8.628(7) D, V=2914.6(1) D3 ; 23161 measured, 6352 unique(Rint 4.18%, Rsigma 4.25%) completeness 97.8%/22 55E redundancy: 4.0

Rl =3.74% / 5181 refs,Flack 0.00(4)

[AFC7R 4circle diffractometer]

MoK″, 50kV-250mA, ⊤ scan

Measurement time: 54 hr

orthorhombic, P2$_1$2$_1$2$_1$, a=12.615(3), b=26.753(4), c=8.604(3) D, V=2903(1) D^3; 6667 unique(Rint 1.87%, Rsigma 2.60%) completeness 99.90/o/2255E redundancy: 1.0

Rl =3.07% / 5331 refs, Flack 0.01 (1)

Fig. 2 Measurement with MoK″ X-rays (cobaloxime complex).

reflection). In the final stage, however, the two intensities will be added to each other to be regarded as one reflection. It is possible to remove partial reflections by taking oscillation photos with overlapping of 0.5 to 1.0E. However, partial reflections can be utilized in the refinement of crystal orientation in post refinement.

In the case of 1.0E overlapping, measurement of 55 photos will be entailed in the above condition and thereby the measurement time will be extended by about 20 percent. Even so, this way of overlapping can turn out to be considerably effective depending on the case. (We have an experience of 0.7 point down with regard to the final R factor.) Fig. 2 shows the measurement result of a cobalt complex. The same crystal was measured also with the four-circle diffractometer AFC-7R. Comparison of the measurement time in both cases shows that the RAXIS RAPID could get about the same outcome in 1/10 of the time required by the AFC-7R. (It should be noted, moreover, that while the RAXIS RAPID is provided with a sealed tube Xray generator, the AFC-7R is equipped with a rotating anode X-ray generator.)

In the Weissenberg photograph method, several crystal axes that allow crystal axis alignment are selected automatically and the data collection measurement is performed in sequence. However,

there also are occasions that the combination of Weissenberg and oscillation photographs is selected, if it is more efficient. As for the crystal in Fig. 2, since one of its axis is as long as 27 D, the use of the 5E oscillation photograph method will cause fairly many overlaps of reflections (In some photo, 36 percent of reflections unusable due to overlapping). If the Weissenberg method is used in such a case, the reflection-overlapping problem may be overcome by properly adjusting the coupling constant. In other words, we can expect the merit of reducing the number of photos (i.e. the time required for readout) by making greater the crystal oscillation angle than in the case of the oscillation photographic method. In short, in the case of a long lattice parameter or rapid measurement where two minutes duty time per IP is not negligible, the Weissenberg method is considered advantageous. In practice, however, it should be noted that there also exist such axes whose alignment cannot be possible due to the limitation of the goniometer operation range.

2. Change of the Measuring Condition: The standard measurement strategy is sometimes modified. For instance, the accuracy of data can be enhanced by measuring more numbers of equivalent reflections (i.e. increase of the redundancy) and finally averaging them. Also, when an empirical absorption correction method using equivalent

reflections is to be made, it is more advantageous that measurement is conducted to have a number of the equivalent reflections. To this end, a measurement using different crystal orientations should be added further to the standard measurement condition. For example, N=90E and T=0 to 160E, or N=270E and T=0 to 160E may be added to the standard measurement of P=45E, N=0 and 180E, and T=130 to 180, 0 to 160E. Then measurement is to be made with 3 or 4 crystal orientations. The measurement time will become longer, but this does not matter if crystals are stable and if one has an ample time for measurement. (For instance, when measurement is started in the evening, an often practised way is to prolong the measurement intentionally by increasing the number of crystal orientations for measurement or extending the exposure time so that it will be over next morning rather than during midnight.) Also, there are occasions that despite the same reciprocal space for measurement, a decrease in the oscillation angle of crystal per IP is made. In the case of the aforementioned cobalt complex, for instance, 110 oscillation photos are taken by reducing the oscillation angle from 5E to 2E in order to avoid overlap of reflections. The smaller the oscillation angle per IP, the more the background noise can be reduced. This method therefore may also be suitably applied to tiny crystals for which long exposure is required.

3. Rapid Measurement: When priority is given to rapid measurement (e.g. in the case that a crystal can deteriorate during measurement), one axis rotation is made and only the independent reciprocal space is measured by referring to the simulation result. In case the crystal symmetry is high, the completeness comes to 0.8 or so by rotating the crystal only 60E. While it degraded the quality of the data (+l to +2 points in terms of the R factor), the structure analysis itself is still possible. Proper strategy should therefore be selected according to the purpose and the crystal to be studied. Besides, caution is needed to an unfortunate case that a reduction in the area to be measured will result in no measurement of reflections in the axial direction. This is the case when the space group cannot be determined because the systematic absences are unknown. It is well to bear in mind that excessive time can bring about a trouble in the structure analysis. That is, when viewed as a whole, ordinary measurement may sometimes get a better outcome in a shorter time.
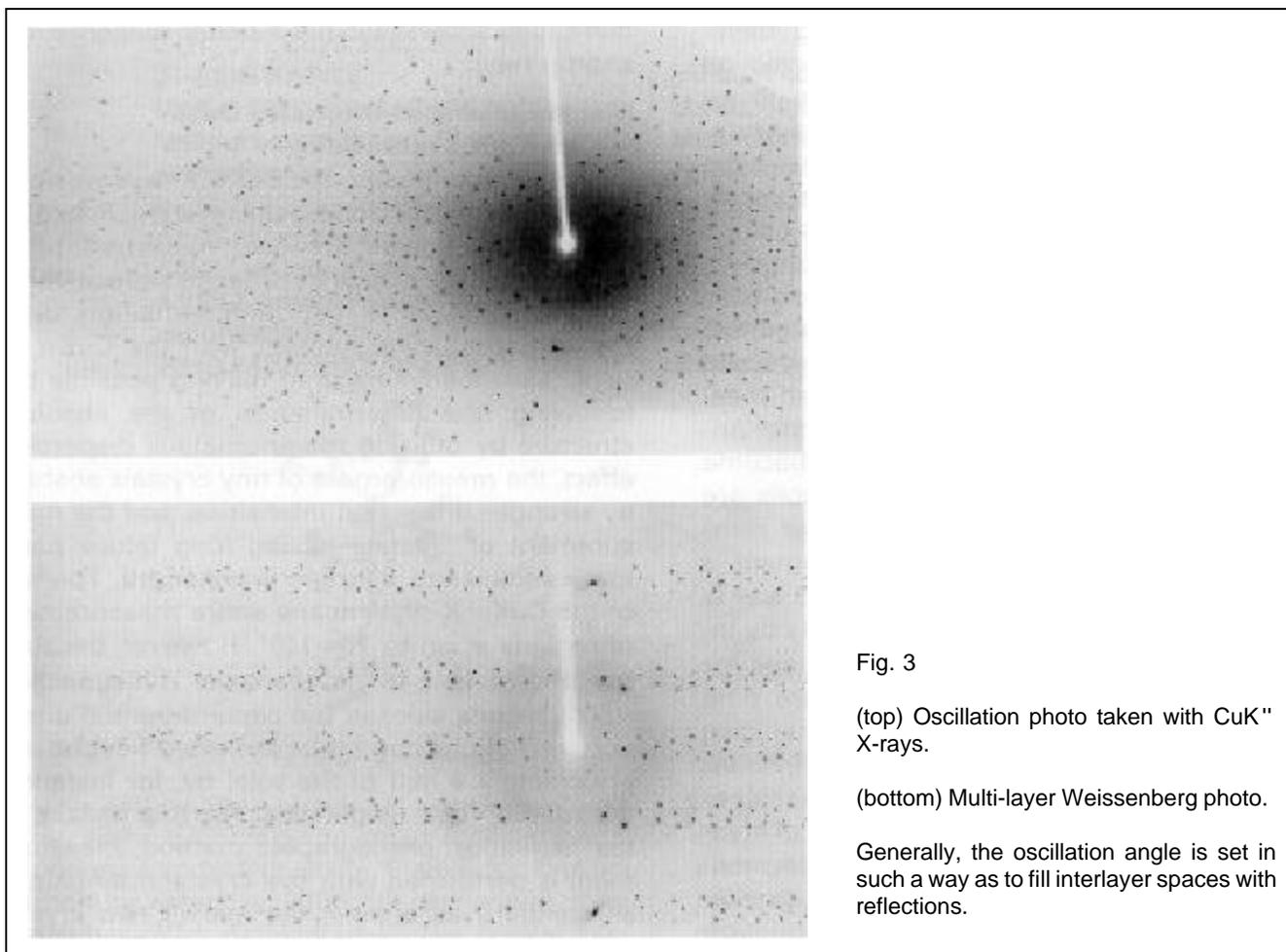
Measurement of Intensity Data

! with CuK"radiation!

Measurement using the CuK" X-rays was one of our aims of developing this system. Recently, organic substances are also measured often with the MoK" X-rays in order to reduce X-ray absorptions and to get high-resolution data. Nevertheless, measurement with the CuK" Xrays has the advantage of making possible the following: the determination of the absolute structure by utilizing the anomalous dispersion effect, the measurement of tiny crystals enabled by stronger diffraction intensities, and the measurement of crystals having long lattice parameters thanks to a longer wavelength. The use of the CuK" X-rays means entire measurement of regions at up to $2\theta=140E$. However, because the IP size is -45E in the axial direction and -60E (on one side) in the circumferential direction, reflections cannot be collected beyond approximately a half of the total by, for instance, the rotation of a single axis. For this reason, in the oscillation photographic method, measurement is performed with five crystal orientations as standard as against the use of two crystal orientations with the MoK" X-rays. This condition is roughly the same for the Weissenberg method. Fig. 3 shows an example of an oscillation photo and multi-layer Weissenberg photo.

1. Determination of the Absolute Struc-ture: Fig. 4 shows a measurement example of an organic crystal with the CuK" X-rays. The same crystal was also measured with the AFC5R. With the R-AXIS RAPID, a seven-time larger region could be measured in 1/4 of the time required for the AFC-5R. The result of structure analysis proved to be almost the same. Automatic measurement was conducted with this four-circle diffractometer, and so only the independent reciprocal space of Laue group of mmm was measured. Because there was no center of symmetry in this space group, the number of independent reflections measured came to 1/2 in the four-circle diffractometer measurement. Also, the Flack parameter, which indicates the correctness of the absolute structure, was converged to 0.5. Thus, the absolute structure could not be determined. Because the atoms of C, H and 0 alone were contained, it is open to argument that to what extent the determination of the absolute structure is reliable (Such a question may not arise if S or the heavier is contained). With the data collection with the R-AXIS RAPID, the Flack parameter was converged to 0.0 (to

Fig. 3

(top) Oscillation photo taken with CuK'' X-rays.

(bottom) Multi-layer Weissenberg photo.

Generally, the oscillation angle is set in such a way as to fill interlayer spaces with reflections.

C RAXIS RAPID (CuK'', 40kV-50mA)

Measurement time: 6,7 hrs. completeness: 98.3%/136E

$P2_12_12_1$, a=17.235(), b=17.567(l), c=8.416i(G)D,

V=2548.5(3) D^3, Z=4

17529 reflections, 4489 uniq, Rint 0.019, Rsigma 0.020

R1 =0.060(2 F I)/4145 reflections/330 parameteres

C-C, O-C esd 0.005 D

Flack: 0.00(29); inverted: 1.02(29)

CAFC5R (CuK'', 40-kV-100MA)

Measurement time: 28 hrs. completeness: 94.1%/135E

$P2_12_12_1$,a =17.202(3), bb=17.549(2), c=8.411(1)D,

V=2539.2(6) D^3, Z=4

2483 reflections, 2457 uniq, Rint -, Rsigma 0.017

R1=0.057(2Fl)/2197 reflections/330 parameteres

C-C. O-C esd 0.006 D

Flack: 0.59(47)
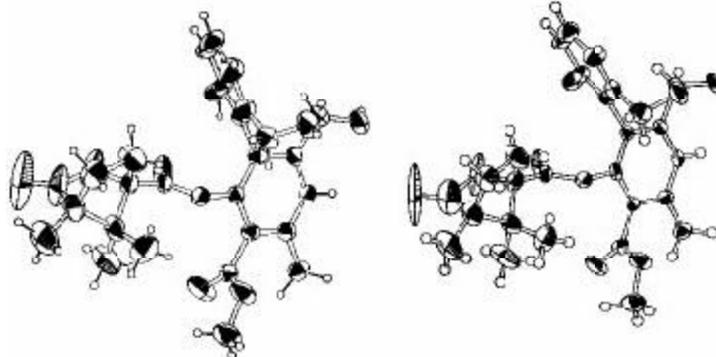

Fig. 4 Measurement of organic molecular crystal with CuK'' X-rays

20 x 20 x 300 : m, $C_{70}H_{66}FeN_9O_6S$,

$P2_1/n$, Z=4, CuK", 60 photos by the Weissenberg method, 6.3 days 63723 measured 11238 unique, compl 95.9%, redu. 6.23

R1 7.7% / 3058 refs (MoK" 1698refs) 30 days of measurement is necessary by the four-circle diffractometer
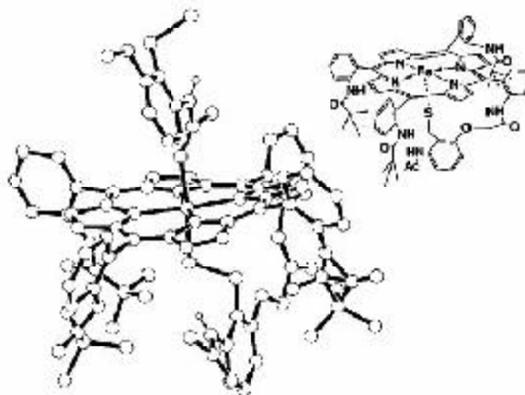
Fig. 5 Measurement of tiny crystal.

1.0 with the inverted structure). Although esd of the parameter had been fairly great, it was improved along with the R factor by setting the measurement temperature at -100EC. Regarding the atom at the left end of the molecule in Fig. 4, its considerably strong thermal vibrations subsided when a low temperature measurement was conducted. Thus, the low temperature measurement is recommended not only for the determination of the absolute structure.

2. Analysis of Tiny Crystals: As a measurement example of a tiny crystal, an iron complex was measured, as in Fig. 5. It was a needle-like 20 x 20 x 300 : m crystal. Measurement with the MoK" X-rays was first made but the structure analysis was unsuccessful because of weak reflections. Then CuK" X-rays were used by allowing an ample time. As a result, the number of reflections increased to bring about a good result such that the disordered structure was recognizable. This example shows that if the crystal contains a metal atom, the crystallinity is good and the size is about the same as this example, even a sealed tube X-ray generator (CuK" X-rays) is sufficient to measure such a tiny crystal. Conversely, when a heavier atom is contained, it is possible to measure ever more tiny crystals. In short, for C, H, and 0 atoms alone, it must be said that measurement is a little bit difficult.

3. Crystals Having Longer Lattice Parameter: The longest lattice parameter thus far dealt with is 70D in one axis. But crystals of such a long lattice parameter in our laboratory are not so many yet (Fig. 6).
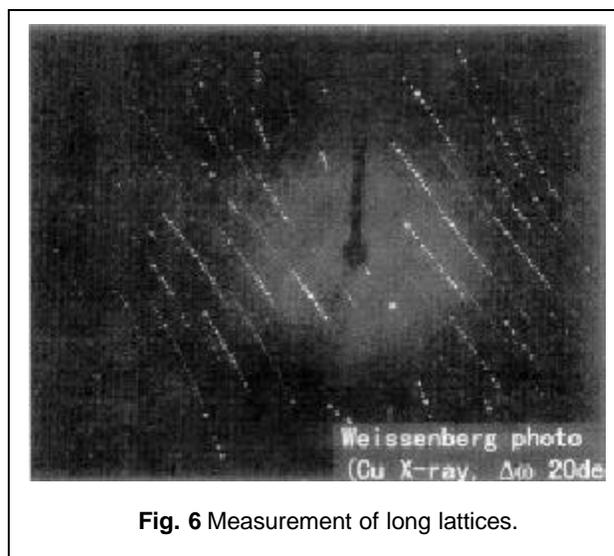

Fig. 6 Measurement of long lattices.

Concluding Remarks

In the above, I have summarized my experiences, as they came to mind, of using the newly developed Weissenberg camera R-AXIS RAPID for one year. While we have three four-circle diffractometers installed in our laboratory, they are now being used less and less since the rapid measuring systems, i.e. the IP Weissenberg camera and the CCD camera were put to operation. Needless to say, in such a case as when very small changes of the lattice parameter are to be examined on the time resolving basis (a measurement often conducted to follow the crystalline phase reactions), it is the four-circle diffractometer's turn to perform the task. For routine measurement of crystals, however, the rapid measuring systems are far more attractive because they can yield equivalent or better measurement results within a time one order of magnitude less. The

merit of using them is greater further when dealing with unstable crystals or crystals whose cell volume is so large that it takes a few weeks for the fourcircle diffractometer to complete measurement.

The rapid measuring systems also have such advantages that after a crystal is mounted on the goniometer, it takes only several minutes to decide if the sample is really a crystal or if its crystallinity is so good as to allow data measurement; and that, moreover, since information is given in the form of photograph, far more information can be obtained than with the four-circle diffractometer. It offers an optimal tool especially when it is necessary to replace samples one after another successively until the good crystal is found out.

The performance of the IP camera has steadily improved for the past one year in terms of hardware and software. The related engineers' passions and efforts given to this camera are well appreciated. Incidentally, we have recentiy conducted measurement with both the IP Weissenberg camera and the CCD camera under about the same conditions wherever possible. An organic crystal for testing use having the formula of $C_{10}H_{11}O_2S$ was measured with them under the respective standard conditions (MoK" X-rays). The results show that: with the R-AXIS RAPID, the measurement time: 5.1 hr, Rl = 2.93%/2196 reflections, Rint=2.40% (8278 measured). With the Siemens SMART system, the measurement time: 8.Ohr, Rl=2.31%/2210 reflections, Rint=1.62% (7135 measured). These figurers must be said excellent when considering the fact that the R-AXIS RAPID were developed in just one year. The improvements are still going on for both of them. In parallel, the study of next-generation two-dimensional detectors like the MSGC (Multi Strip Gas Chamber) is also being made actively. A hot situation is likely to continue for the time being, surrounding the rapid measurement system equipped with a two-dimensional detector.

The R-AXIS RAPID was designed under joint researches by Professor Yuji Ohashi at Tokyo Institute of Technology and myself. I would like to express my sincere thanks to staff members of Rigaku Corporation who materialized our design of the rapid measuring system and endeavored further for its performance upgrading

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