RIGAKU VariMax Dual
Part 0 Startup & Shutdown Manual

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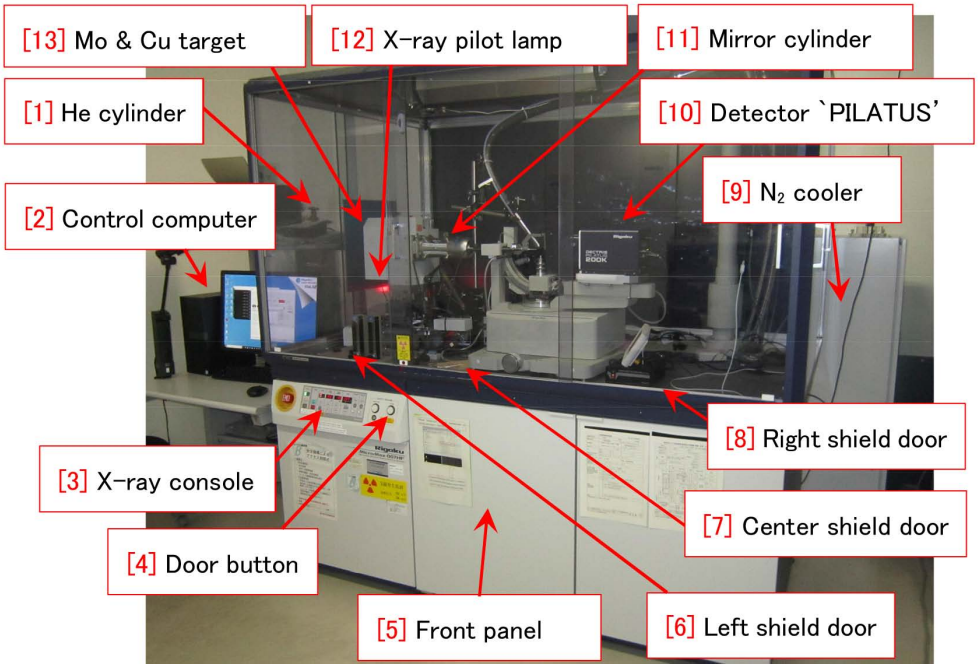

Figure 0: Whole figure of the apparatus.

This manual describes startup and shutdown sequences of Rigaku VariMax Dual that can be used to do crystal structure analysis of low-molecular-weight crystals.

With regard to the processes of measurement and analysis, refer to Part 1 and Part 2 manuals, respectively, please.

Mo $K\alpha$(0.7107 Å; 17.4435 keV) or Cu$K\alpha$(1.5418 Å; 8.0408 keV) X-rays emitted from a focus with 70×700 μm size with a take-off angle of 6° (0.1 rad); the point focus mode of 70×70 μm, on '[13] Mo & Cu target' of Fig. 0, are monochromatized and condensed with a confocal mirror system. They are incident on the crystal whose structure to be solved. In spite of the low-power (1.2 kW) to generate the X-rays, photon flux of X-rays per unit area at the crystal position is several tens of times as great as a conventional apparatus not equipped with such an optical system.

Crystal structure analysis for a small crystal whose size is less than 100 μm cannot be done practically when using a normal apparatus. However, the VariMax Dual has successful achievements of solving structures of crystals whose sizes are less than 10 μm. Additionally, crystal screening can be done several tens of times as rapidly as compared with a conventional apparatus.

In Appendix A [p.8], the detailed usage of cooled N_2 generator ('[9] N_2 cooler' in Fig. 0) is described. In Appendix B [p.10], the usage of the microscope is described. In Appendix C [p.15], how to request the remote assistance is desribed.

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Chapter 1

Preparation before starting the experiment

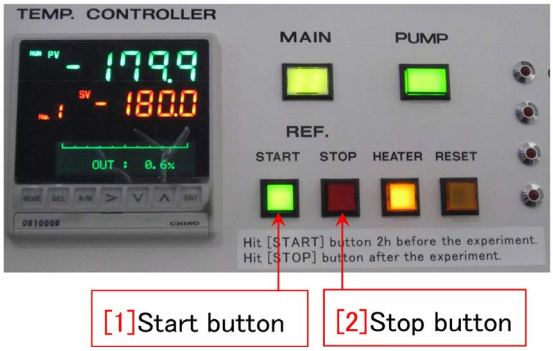

Figure 1.1: Control panel of cooled N_2 generator

The N_2 cooler should be started 2 h before starting the experiment. Then, 1.5 h before starting the experiment, set the X-ray power to stabilize it, please. 30 min before the experiment, He valve should be opened such that inside the mirror cylinder is substituted with He gas.

1.1 Start of the cooled N_2 generator

Push '[1] Start button' in Fig. 1.1, 2 h before starting the experiment, please. Then, let '[1] Start button' illuminate green in place of red '[2] Stop button'. When doing the experiment at room temperature, this process is not necessary. The temperature of cooled N_2 has usually been set to be -180°C (recommended value). When changing the temperature or rapidly cooling N_2,

Figure 1.2: X-ray power setting panel

refer to Appendix A[p.8], please.

1.2 Selection of X-ray source

In Fig. 1.2, Mo has been selected as the target. X-ray source setting panel can be found as shown in Fig. 1.2 by opening '[5] front panel' in Fig. 0 on the cover of this manual. As shown on the white label X-ray target and bias should be set such that these are identical with the values on the white label. Let the filament time be shown by pressing [F4] key for describing it on the experimental notebook, please. The values of bias change after exchanging the filament.

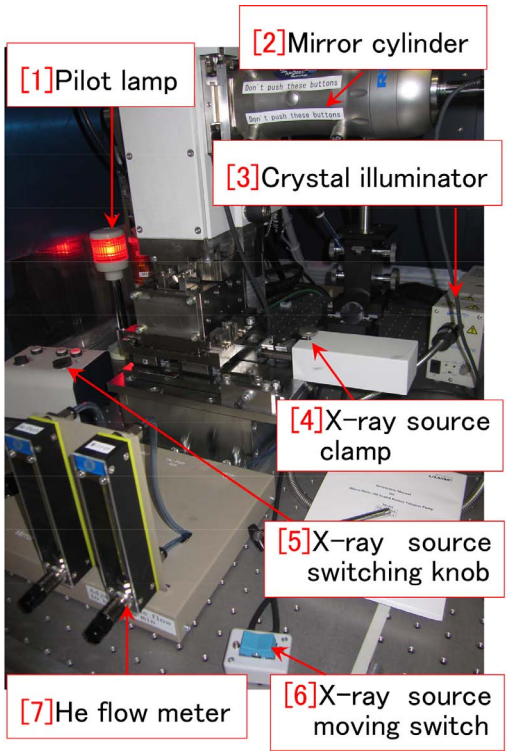

Figure 1.3: Around the X-ray source

[F3] or [F4] keys should be pressed after pressing [F1] key.

1.3 Change of X-ray target and confocal mirror system

In Fig. 1.3, Mo or Cu target can be selected by turning '[5] X-ray source switching knob'. This switching should be done necessarily before generating the X-rays, i.e. necessarily before starting the softwareas 'JXG' shown in Fig. 1.8.

Figure 1.4 is a closeup around '[2] Mirror cylinder' in Fig. 1.3. In Fig. 1.4, the confocal mirror system for Mo target has been selected. When Cu target is selected, '[2] Mirror switching ring' in Fig. 1.4 should be turned by 180° in the direction indicated by a small triangle mark. When turning this, take care such as not to touch '[1] Mirror adjusting buttons', please.

Since September. 2019, the rule of reservation to use the VariMax Dual has been revised such that the desirable target (Cu_ or Mo_) should be written when making a reservation to use the apparatus. That is to say, the desirable target for the next user should be set by the previous user such that the next user can start the experiment soon. However, the setting of the X-ray source (Cu or Mo) should be confirmed again also by the next user. Therefore, after finishing the experiment, the setting of the target should be changed if neccessary, by referring to the reservation calendar.

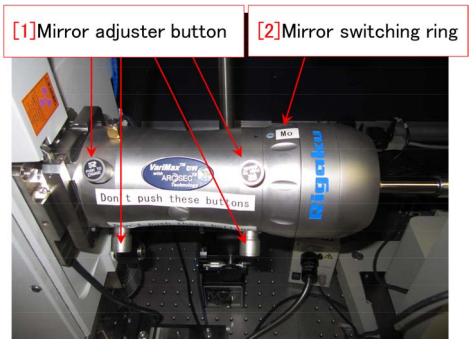

Figure 1.4: Mirror cylinder

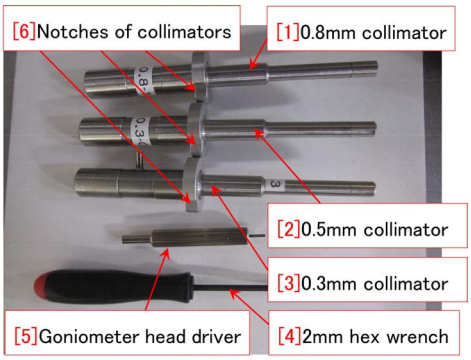

Figure 1.5: Collimators and drivers

1.4 Setting the distance between the X-ray source and the crystal

The distance between the X-ray source and the crystal can be changed by pressing blue '[6] X-ray source moving switch' after unfastening '[4] X-ray source clamp' in Fig. 1.3. When the X-ray source is at the leftmost position, the focus diameter is about 250μm (smallest). This position gives maximum photon flux per unit area at the crystal position and then the most suitable for a small crystal whose size is less than 200μm. When the X-ray source is the rightmost position, X-ray beam diameter is about 400μm (largest). This position is suitable for a crystal

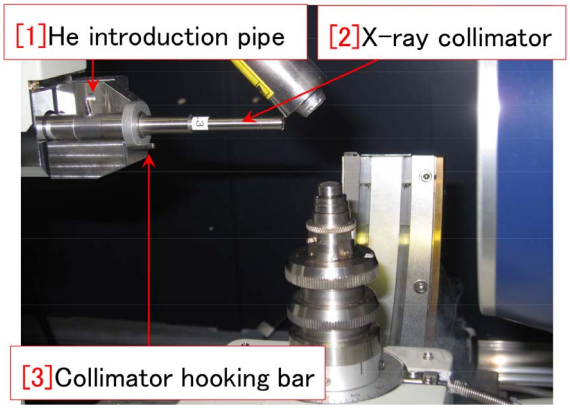

Figure 1.6: Around the sample crystal

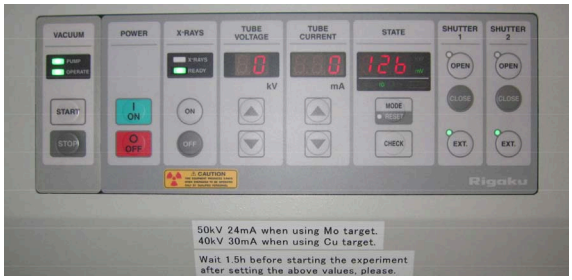

Figure 1.7: X-ray setting console

whose size is larger than 250μm. Since the crystal should be fully bathed in the X-ray beam, its size should be less than 400μm.

After changing the distance between the X-ray source and the sample crystal, '[4] X-ray source clamp' in Fig. 1.3 should be fasten again.

'[3] 0.3mm collimator' in Fig. 1.5 should be set as shown in Fig. 1.6 when the X-ray beam size is 250μm. However, '[2] 0.5mm collimator' should be set when the X-ray beam size is 400μm. The collimator should be magnetically set by hooking '[6] Notches of collimators' in Fig. 1.5 on '[3] Collimator hooking bar' in Fig. 1.6. When using '[3] 0.3mm collimator', insert '[1] He introduction pipe' into silicon rubber tube in Fig. 1.6. This is not necessary when using '[2] 0.5mm collimator' since it does not have '[1] He introduction pipe'.

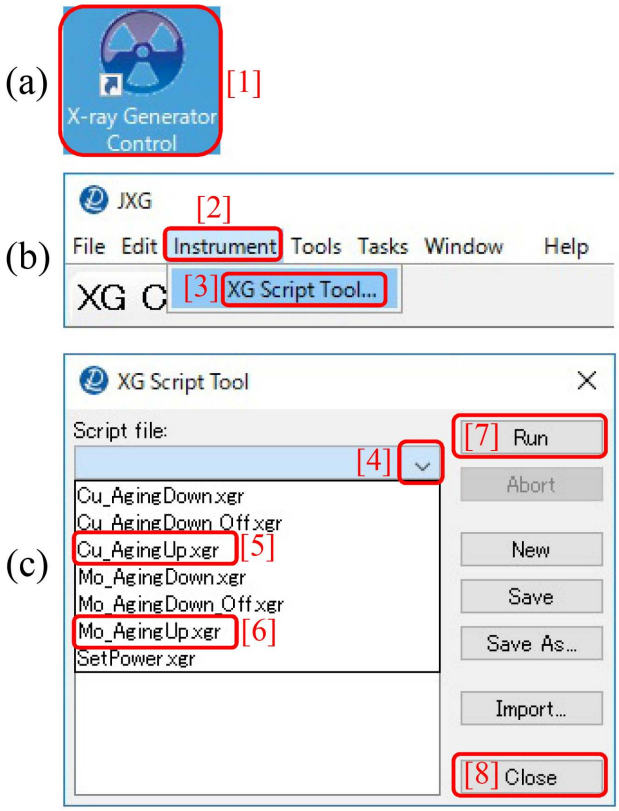

Figure 1.8: X-ray ON/OFF software 'JXG'

1.5 Setting of X-ray voltage and current

In the case of the old system, the voltage and current of the X-ray generator has to be set manually by operating the console as shown in Fig. 1.7. However, those of the new system can be set with a software 'JXG' whose icon is shown in Fig. 1.8 (a).

A menu window as shown in Fig. 1.8 (b) can be opened by double-clicking the icon. '[3] XG script tool ⋯ in the menu of '[2] Instrument' can be clicked to open a pull-down menu as shown in Fig. 1.8 (c) '[4]'. 'Cu_AgingUp.xgr [5]' or 'Mo_AgingUp.xgr [6]' should be selected from the pull-down menu to start the Cu or Mo X-ray generator. The voltage and current are gradually increased automatically after clicking '[7] Run' in Fig. 1.8 (c). After waiting for 1.5 h, a stabilized X-ray power with 40 kV, 30 mA (Cu) or 50kV, 24 mA (Mo) can be obtained. When starting the experiment, '[8] Close' in Fig. 1.8 (c) can be clicked to close the 'JXG'.

Figure 1.9: He cylinder regulator

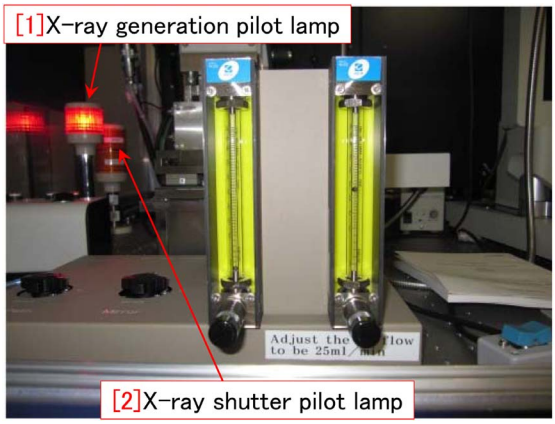

Figure 1.10: He flow meter

1.6 Start of He substitution

30 min before starting the experiment, He substitution in the mirror cylinder should be started by opening '[1] He supply valve' in Fig. 1.9. He flow has been adjusted to be 22 $\sim$ 25ml/min. The He flow adjusting knob does not have to be turned. However, it can be adjusted by turning the adjuster knob bellow the right He flow meter in Fig. 1.10. The left flow meter should be zero.

With regard to the mount of the crystal and the measuring process, refer to Part 1 manual, please.

Chapter 2

After finishing the experiment

Figure 2.1: X-ray setting console

2.1 Stop of the cooled N_2 generator

Press '[2] Stop button' in Fig. 1.1[p.1], please. The other switches do not have to be touched. The temperature around the crystal gradually increases to reach the room temperature.

2.2 Switching off the X-ray

When the next user does the experiment with the same X-ray source. The X-ray power should not be turned off. However, the X-ray power should be gradually decreased when no user starts the experiment soon or the other X-ray source is used by the next user.

In the old system, the X-ray power had to be turned off manually operating the console as shown in Fig. 2.1. However, the X-ray generator in the new system can be gradually decreased and turned off automatically with a software 'JXG', By double-clicking the icon as shown in Fig. 2.2 (a) '[1]', a menu window as shown in Fig. 2.2 (b) can be opened. After clicking the pull-down menu as shown in Fig. 2.2 (c) '[4]', 'Cu_AgingDown_Off.xgr [5]' (Cu) or 'Mo_AgingDown_Off.xgr [6]' (Mo) should be selected to click '[7] Run' for automatically decreasing the X-ray power and turning off.

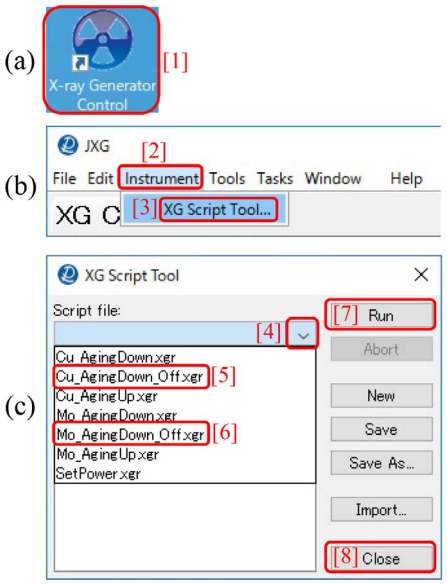

Figure 2.2: X-ray ON/OFF contoroller 'JXG'

2.3 Close of He supply valve

In Fig. 1.9, close '[1] He supply valve', please. The He flow knob under the right He flow meter in Fig. 1.10, does not have to be closed.

2.4 Recovery of the crystal

Remove the crystal to be brought back to the user's laboratory, please. Mount tools should be washed by water and alcohol at room 332 next to room 333 for the next use.

2.5 Exchanging the X-ray target (or verification)

If the user find on the reservation calendar that the next user uses the different X-ray source (Cu or Mo), the setting of it should be changed such that the next user can start the experiment soon. Open '[5] Front panel' in Fig. 0 on the cover of this manual to set the target to be Mo or Cu and the bias to be the value for Mo or Cu as typed on the white label in Fig. 1.2 [p.1], please. [F3] and [F4] keys should be pressed after pressing [F1] key. In Fig. 1.3 [p.2], '[5] X-ray source switching knob' should be turned to be the source the next user uses. In Fig. 1.4 [p.2], '[2] Mirror switching ring' should be turned by 180° to the angular position for Mo or Cu taking care such as not to touch '[1] Mirror adjuster buttons'.

2.6 writing the filament time on the experimental notebook

On the X-ray power setting panel in Fig. 1.2 [p.1], read the filament time by pressing [F4] key after pressing [F1] key to write it on the experimental notebook, please.

If any trouble happened, write it on the experimental notebook, please.

To be continued

Appendix A

Temperature adjustment of the cooled N_2

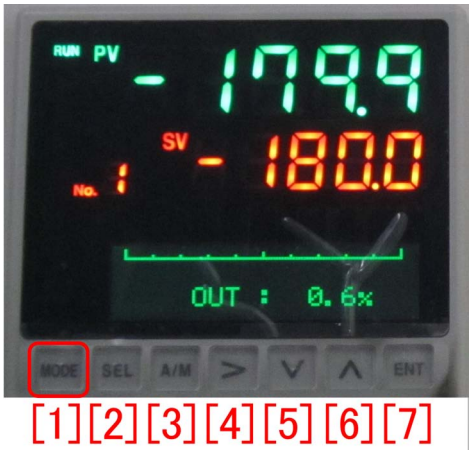

Figure A.1: Enlargement of the temperature setting unit in Fig. 1.1[p.1].

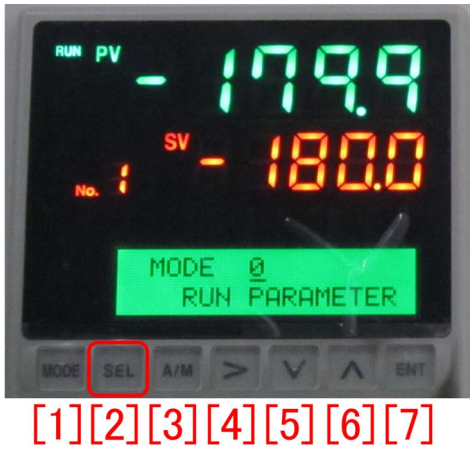

Figure A.2: '[1] [MODE] button' in Fig. A.1 has been pressed.

While the usual usage of cooling N_2 generator has been described in §1.1 [p.1], more detailed usage is described in this chapter.

A.1 Change of the temperature

Fig. A.1 is an enlargement of the temperature setting unit from Fig. 1.1 [p.1]. After pressing '[1] Start button' in Fig.1.1 [p.1], the temperature -179.9°C measured by a sensor has been indicated with light blue characters while the set temperature indicated with orange characters is -180°C.

By pressing '[1] [MODE] button' in Fig. A.1, Fig. A.2 has been displayed. Further, by pressing '[2] [SEL] button' in Fig. A.2, Fig. A.3 can be displayed. Here, the decimal place of temperature indicated by an underscore can be changed by pressing '[4] [>] button'. Number of it can be increased or decreased by pressing '[6] [∧] button' or '[5] [∨] button'.

By pressing '[7] [SET] button' after setting the temperature as in Fig. A.3, the set temperature indicated with orange characters can be changed by 2°C/sec to which the measured temperature follows almost at the same rate. After reaching to the set temperature, the measured temperature oscillates for a few minutes. After that, it stabilizes around the set temperature. Here, after pressing '[1] [MODE] button' twice, the experiment can be started with a temperature as shown in Fig. A.4 (with an arbitrary temperature).

The allowed temperature range is $-180 \sim +25$°C. An arbitrary temperature in this range can be set.

After finishing the experiment, set the tem-

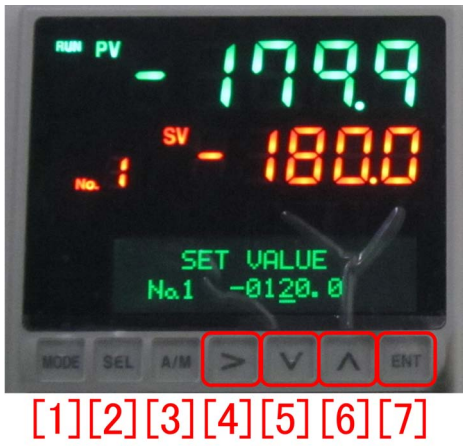

Figure A.3: '[2] [SEL] button' in Fig. A.2 has been pressed to change the set temperature.

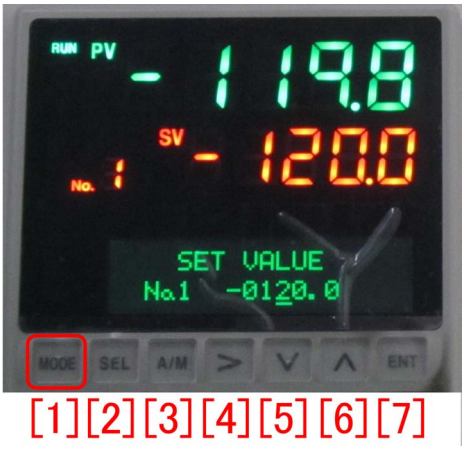

Figure A.4: '[7] [ENT] button' has been pressed such that the temperature reaches to −120°C.

perature at −180°C, please. Then, '[2] Stop button' in Fig. 1.1 [p.1] should be pressed to stop the cooling function.

A.2 Rapid cooling to a low temperature

After pressing '[1] Start button' in Fig. 1.1 [p.1] in a usual way as described in §1.1 [p.1], the measured temperature decreases to be −180°C taking 2 h. However, there is an opinion that rapid cooling is more desirable, about which the manager has not verified the efficiency. The way of rapid cooling is as follows.

At first, let the set temperature be about the room temperature (∼ 25°C) in an identical way as described in §A.1 from a situation as shown in Fig. A.1, please. Then, press '[7] [ENT] button', please, such that the measured temperature reaches to the room temperature within about 2 min. After waiting for several min, let the set temperature be −180°C again, please. After pressing '[7] [ENT] button' again, the measured temperature rapidly decreases to reach to −180°C in a few min.

Appendix B

Usage of the microscope

This chapter describes the usage of microscope.

Figure B.1 is a whole figure of the stereoscopic microscope, Nikon SMZ1500. Figures B.2, B.3, B.4, B.6, [p.12] B.8 [p.12] and B.10 [p.13] are closeups of Fig. B.1. The usage of another microscope, Nikon SMZ1000 placed near the apparatus for protein crystal structure analysis, is similar to that of SMZ1500 and can also be referred to this chapter.

However, the finest division on the crosshair viewed through the right ocular lens is 100 μm for a magnification of 1.0 for SMZ1500 whereas it is 100 μm for a magnification of 0.7 for SMZ1000.

B.1 Fundamental usage

B.1.1 Turning on and adjustment of the illuminator

Figure B.2 is a closeup of Fig. B.1 '[12] Illuminator switch'. When turning on it, the brightness should be slowly increased. When turning off, the brightness should be gradually decreased, before switching off.

B.1.2 Selection and position setting of objective lens

Figure B.3 is a closeup of Fig. B.1 '[7] Objective lenses & revolver'; an objective lens with X1.0

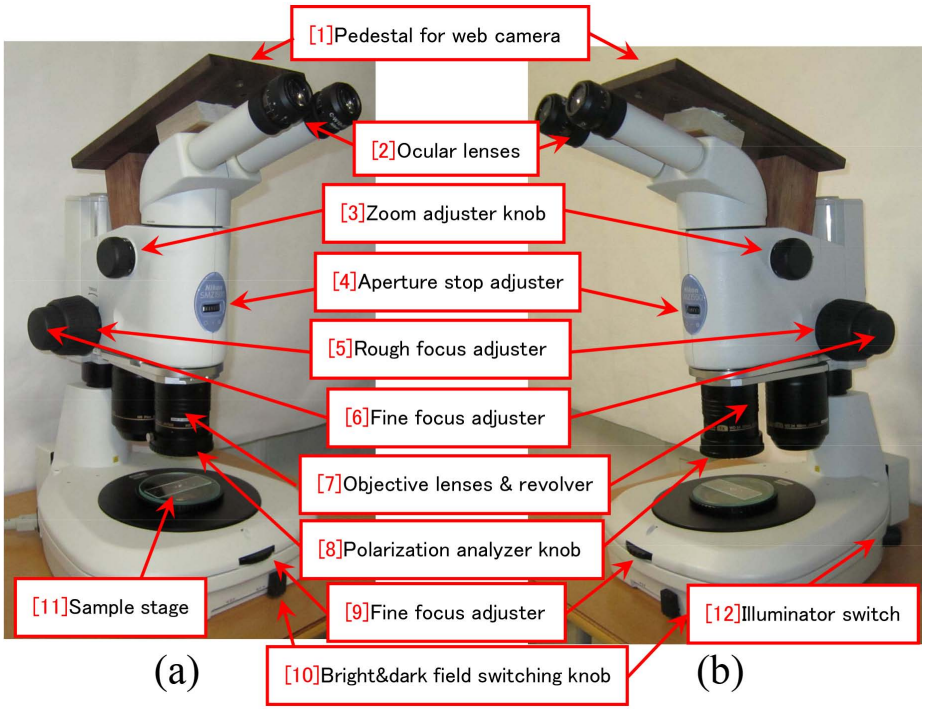

Figure B.1: Whole figure of the microscope.

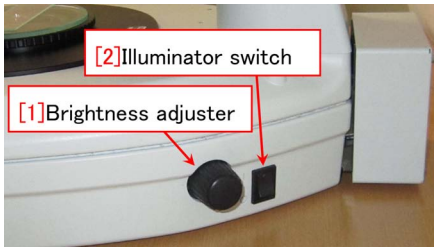

Figure B.2: Illuminator switch and brightness adjuster.

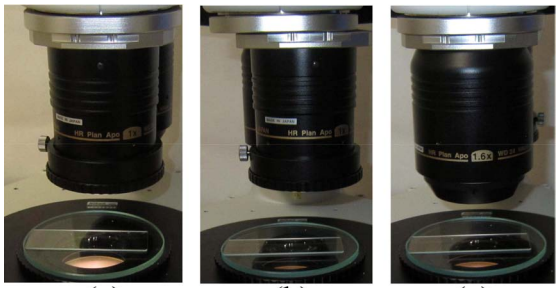

Figure B.3: Objective lenses & revolver.

for (a) and (b), and that with X1.6 for (c) is selected by turning the revolver by 180°. However, the focus should be adjusted again after turning the revolver to change objective lens.

When the lens with X1.0 is selected, it can be stopped at angular positions (a) and (b). At position (a), the objective lens is stopped at the central position of lens-barrel for stereoscopic observation with both eyes. (b) is slightly right-shift position for observation through only the right eyepiece and suitable for taking photographs with a digital camera or a web camera.

After choosing objective lens, the lens-barrel should be moved downward to reach for the object by turning Fig. B.1 '[5] Rough focus adjuster'.

B.1.3 Eyesight adjustment of right ocular (eyepiece)

Figure B.4 is a closeup in the vicinity of the ocular lenses. The distance between the both ocular lenses (eyepieces) can be adjusted for the most visible stereoscopic view.

A haircross and scales can be observed through the right ocular lens (eyepiece) as shown in Fig. B.5. '[2] Right eyesight adjuster' should be rotated such that they are most clearly observed. The angle of crosshair can be changed by rotating '[3] Crosshair rotator'.

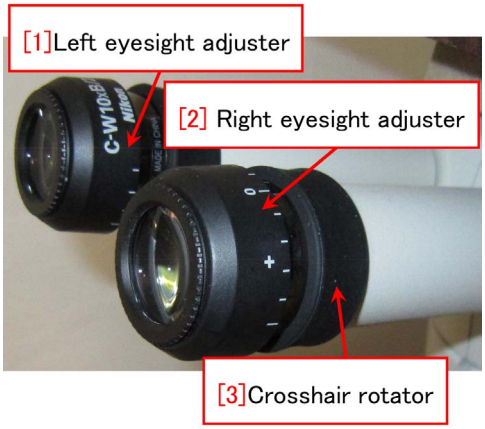

Figure B.4: Ocular lenses.

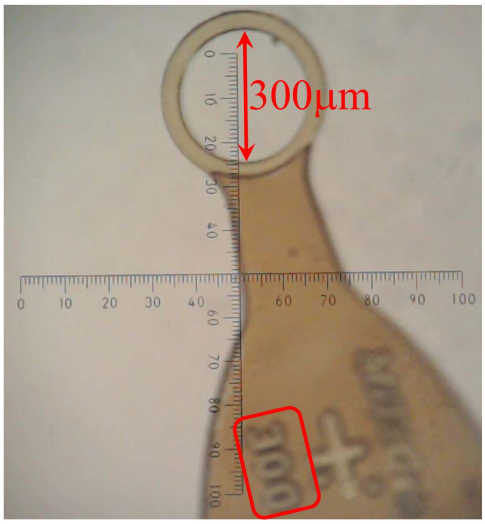

Figure B.5: 300μm micromount (X10).

B.1.4 Adjustment zoom ratio

Figure B.6 [p.12] is a closeup of Fig. B.1 '[3] Zoom adjuster knob'.

While the zoom ratio can be changed in a range of X0.75∼X11.25, a small zoom ratio around X0.75∼X1.0 is recommended at the first view such that a large area on the sample stage can be observed.

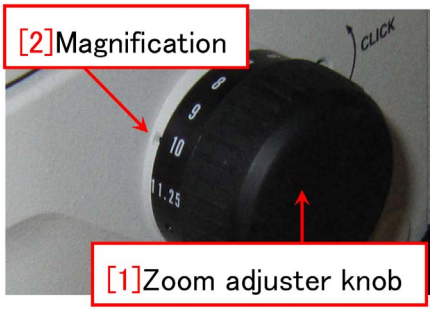

Figure B.6: Zoom adjuster knob.

Figure B.7: Crystal of sucrose. (a) parallel nicol, (b) cross nicol.

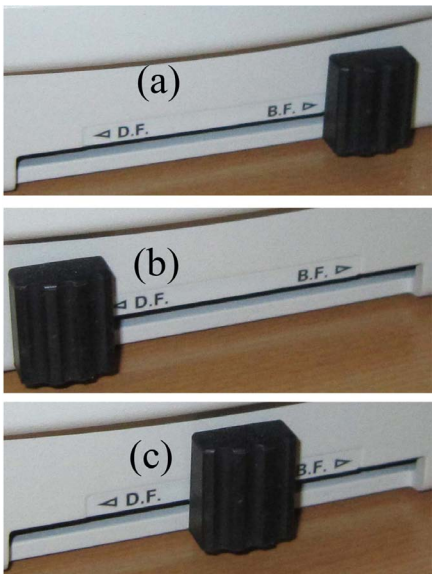

Figure B.8: Bright&dark field switching knob.

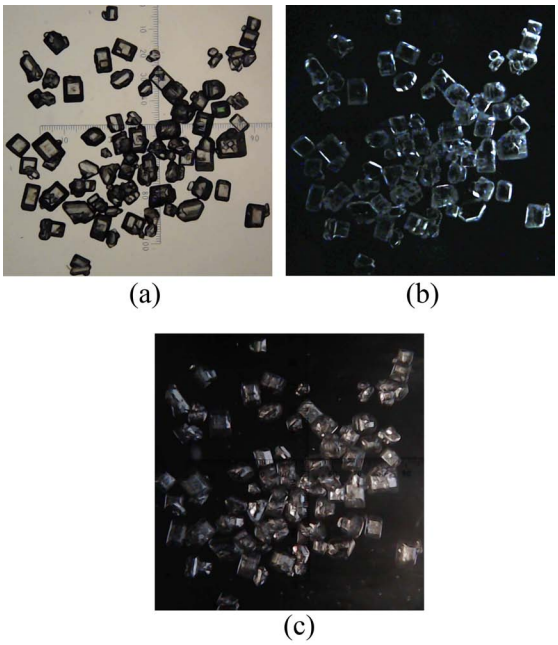

Figure B.9: (a) Bright field image, (b) dark field image, (c) intermidiate image.

B.1.5 Focus adjustment

By viewing mainly through the right ocular lens (eyepiece), Fig. B.1 [p.10] '[5] Rough focus adjuster' should be rotated clockwise viewed from the right such that the objective lens moves upward to depart from the object. After the object comes to be in focus through the right ocular lens (eyepiece), Fig. B.4 [p.11] '[1] Left eyesight adjuster' should be rotated such that the object can be observed clearly also through the left ocular lens (eyepiece).

After that, zoom ratio can be changed by rotating Fig. B.6 '[1] Zoom adjuster knob'. When observing with a high zoom ratio, the focus can be adjusted also by rotating '[6] Fine focus adjuster' or '[9] Fine focus adjuster' in Fig. B.1 [p.10] The finest division on the crosshair is 100 μm for a magnification of 1.0 for SMZ1500 whereas it is is 100 μm for a magnification of 0.8 for SMZ1000.

Figure B.5 [p.11] was taken viewing a largest crystal micromount with a zoom ratio of X10. A number '300' is found in the lower part. The internal diameter of the micromount is 300 μm corresponding to 30 smallest divisions.

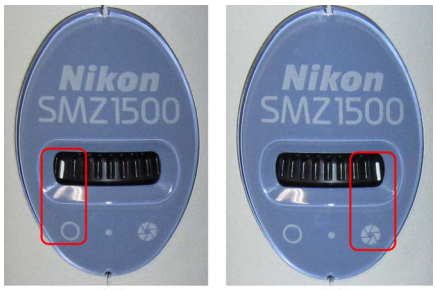

Figure B.10: Objective lens aperture adjuster.

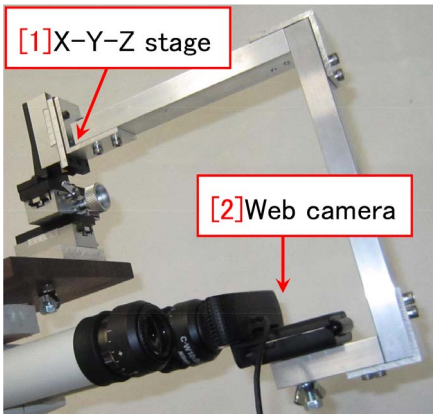

Figure B.11: A web camera is mounted.

B.2 Adavanced usage

B.2.1 Adjustment of polarization analyzer

A polarizer is placed under Figure B.1 [p.10] '[11] Sample stage'. Therefore, the object is illuminated by linearly polarized light A polarization analyzer plate is placed under the X1.0 objective lens. The analyzer can be rotated around the optical axis of the microscope by rotating Fig. B.1 [p.10] '[8] Polarization analyzer knob'.

Figure B.7(b) shows an image of crystals brilliant in a dark field. This angular situation between the polarizer and analyzer is referred to as 'cross nicol'. However, a parallel nicol image as shown in Fig. B.7(a) can be observed by rotating Figure B.1 [p.10] '[8] Polarization analyzer knob' from the cross nicol angular position.

Crystals with crystal systems other than cubic system have uniaxial or biaxial optical anisotropy that causes birefringence resulting in change of polarization state of light. For this reason, 'cross nicol' enables us to observe crystals brilliant in a dark field. Liquid as solvent or paraffin oil does not have such an optical anisotropy. Then, extremely small crystals can be clearly observed even in solvent or paraffin oil by using the 'cross nicol'.

B.2.2 Switching of bright & dark field mode

Figures B.8 (a), (b) and (c) are closeups of Fig. B.1 [p.10] '[4] Aperture stop adjuster'. Bright and dark field modes have been selected in (a) and (b), respectively. Middle position (c) may also be used. Figures B.9 (a), (b), (c) correspond to Figs. B.8 (a), (b), (c).

Bright and dark field modes are those with which the light from the illuminator is incident in the optical path of microscope, directly and not directly, respectively. In the case of dark field mode, only extremely refracted light by edges of crystals is incident in the optical path as shown in Fig. B.9 (b). This mode is effective for observing outlines of crystals.

B.2.3 Adjustment of objective lens aperture stop

Figures B.10 (a) and (b) are closeups of Fig. B.1 [p.10]. A high spatial resolution is given by a large numerical aperture (NA) of objective lens.

Therefore, the maximum resolution is given when the aperture stop is maximally opened as shown in Fig. B.10 (a) for a position of the object just in focus. For a relatively large crystal, however, an image of position whose height is out of focus is blurred. This problem can be mitigated by a small aperture stop adjusted as shown in Fig. B.10 (b), whereas the maximum spatial resolution is spoiled.

B.2.4 Use of web camera

A web camera can be mounted on '[1] Pedestal for web camera' in Figure B.1 [p.10] as shown in Fig. B.11. An image that is observed through the ocular lens can be taken with a digital camera or a web camera. Here, the position of objective lens should coincide with that of the 'exit pupil'. 'Exit pupil' is the virtual image of the

objective lens focused through the ocular lens If the position of the object lens of digital camera or web camera does not coincide with exit pupil, the whole image through the ocular lens cannot be captured by the camera. For the same reason, the whole image cannot be viewed with the observer's eyes when they do not coincide with the exit pupils.

The system shown in Figure B.11 [p.13] is equipped with '[1] X-Y-Z stage' such that the position of web camera can be strictly adjusted to coincide with that of 'exit pupil'. Figures B.5 [p.11], B.7 [p.12] and B.9 [p.12] were all taken by using the system shown in Fig. B.11 [p.13].

This system is very effective to take photographs or movies viewed through the eyepiece of microscope. When the user needs it, contact the manager (Kouhei Okitsu; 27470, 090-2203-8789), please.

Appendix C

Request of the remote assistance

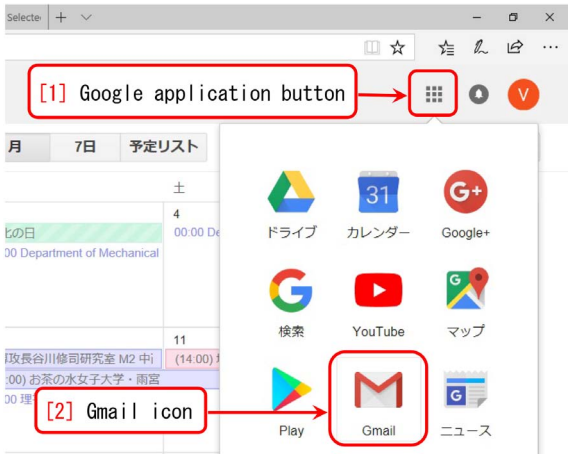

Figure C.1: Open the GMail in the Google application

Figure C.2: 'Compose' should be clicked to type a new e-mail

Even when the manager (K. Okitsu) is absent on the weekend, he can remote-assist the user to operate the XPS by using the remote assistant function of the Windows. At first, call the manager by phone dialing 090-2203-8789, please. After the manager answer, what to do for the user is as follows.

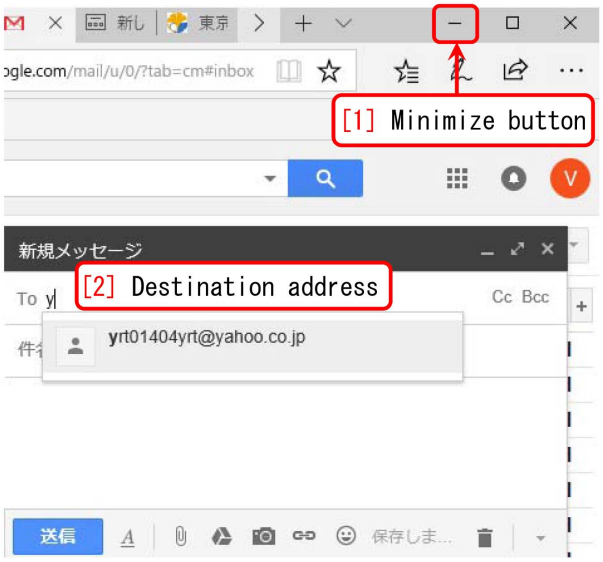

Figure C.3: Just 'y' can be typed to display the manager's e-mail address

C.1 Preparation of the GMail

At first, the internet browser should be opened to let the Google calendar for reservation to use the VariMax Dual be shown. In Fig. C.1, the '[1] Google application button' should be clicked to let the '[2] GMail icon' be shown. It should be clicked to open 'the composing window' as shown in Fig. C.2. The red '[1] Compose button' should be clicked to type a new e-mail.

In Fig. C.3, only one character 'y' can be typed in the '[2] Destination address' to let 'yrt01404yrt@yahoo.co.jp' be shown. It should be clicked such that the e-mail is sent to the manager. Then, '[1] Minimize button' should be clicked.

Figure C.4: 'msra' should be typed in the search text box.

Figure C.5: '[1] Invitation button' should be clicked

Figure C.6: '[1] Save button' should be clicked

C.2 Preparation of the file and password of remote assistance

In Fig. C.4, 'msra [1]' should be typed in the search box on the lower left of the desktop of the computer. Then, '[2] msra' (micro soft remote assistance) should be clicked to display Fig. C.5. '[1] Invitation button' in it should be clicked to display Fig. C.6. In it, '[1] Save button' can be clicked to let Fig. C.7 be displayed.

'[1] Invitation file' as displayed in Kanji characters should be saved on the desktop of the computer. In Fig. C.8, '[1] Yes button' should be clicked to overwrite the file.

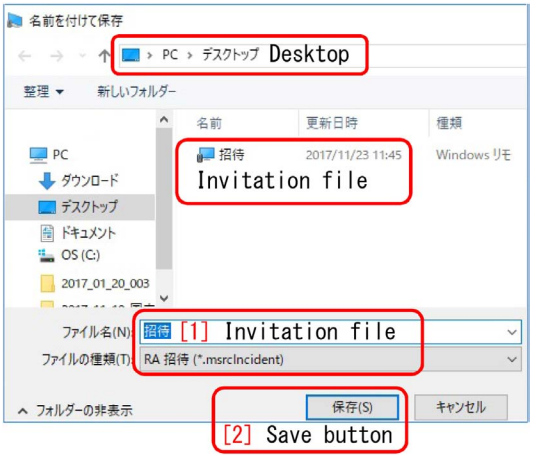

Figure C.7: 'Invitation file' should be on the desktop

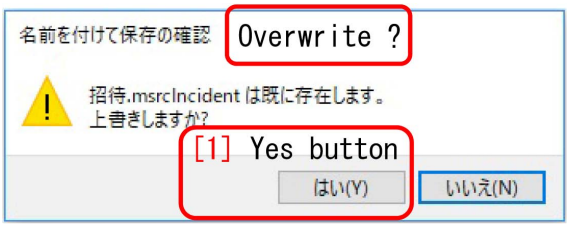

Figure C.8: '[1] Yes button' should be clicked to overwrite

By saving the file of the remote assistance, the password is displayed as shown in Fig. C.9. After selecting it, it should be copied to the clipboard by typing [Ctrl]+[C]

C.3 Sending the file and the password

In Fig. C.10, '[1] Browser icon' is found on the task bar of the computer. It should be clicked to show 'Compose window' again as shown in Fig. C.11.

An arbitrary character(s) can be typed as the '[2] Title'. The password copied before should be pasted by typing [Ctrl]+[V] as the main body of the e-mail.

In Fig. C.11, '[4] File attach button' can be clicked to open the window of Fig. C.12. The invitation file is found at the center of it.

Figure C.9: '[1] Password' should be copied to the clipboard

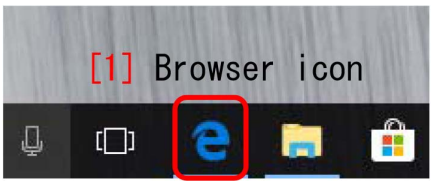

Figure C.10: '[1] Browser icon' on the desktop

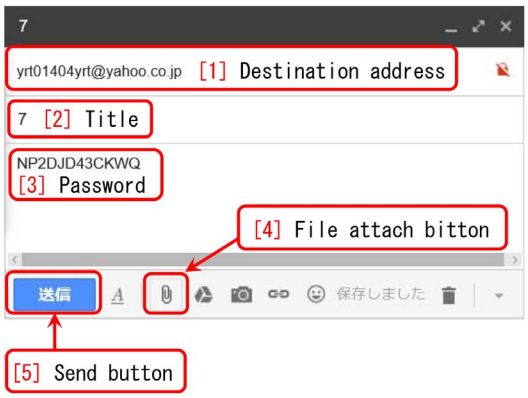

Figure C.11: Type an arbitrary character(s) as the '[2] Title' and paste the '[3] Password' by typing [Ctrl]+[V], please

After selecting this, 'Open' on the lower right of Fig. C.12 should be clicked.

C.4 Starting the remote assistance

When the computer is connected to the manager's computer with the remote assistance,

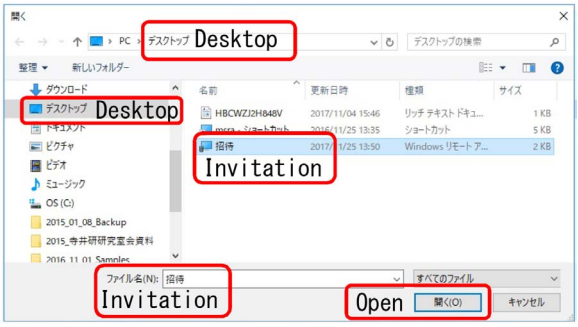

Figure C.12: The invitation file on the desktop should be selected and attached

Figure C.13: '[1] Yes button' should be clicked

Figure C.14: 'Yes' to permit the remote control

Fig. C.13 is displayed. Here, '[1] Yes button' should be clicked to display Fig. C.14. Here, 'Yes' should be clicked to permit the remote control.

Both the manager and the user can operate the mouse and keyboard together to operate the computer.

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