Dual wavelength rotating anode system MicroMax007 VariMax DW

—Data collection by dual wavelength available on one system—

1. Introduction

The dual wavelength system is attractive to crystallographers, chemists and material scientists who deal with various kinds of samples. It is critical to select an X-ray wavelength that is best suited to intended samples. The relations between objectives and X-ray source are summarized in Table 1.

Table 1. Application examples by X-ray source.

Source	objective	Reason
Cu	Absolute structure determination of organic compounds	Larger anomalous signal
Mo	Data collection for metal complex	Reduction of X-ray absorption
Cr	Stress analysis of iron materials	Measurement of high angle Debye rings
Co	Data collection for crystals including iron	Inhibition of X-ray fluorescence generation

It is well known that selecting a suitable X-ray source is essential to successful experiments. However, changing the X-ray source requires tedious and time consuming steps.

A newly developed dual wavelength rotating anode system, MicroMax DW (Dual Wavelength), makes it possible to use 2 different wavelengths without exchanging the target and therefore greatly extended its applicable area. It eliminates troublesome operations such as a target replacement by placing two types of materials on the surface of one target. With MicroMax DW, switching an X-ray source to another can be accomplished without any difficulty. Furthermore, this system works well on tiny crystals because a high brilliance X-ray beam can be irradiated to a sample.

2. Key features

2.1. Data collection of dual wavelength on one system

An X-ray generator and confocal optics of the dual wavelength system enable us to collect data using two different wavelengths within one system. It is especially recommended for users who largely deal with various kinds of samples because an appropriate source can easily be selected for a particular sample.

2.2. DW rotating anode

Exchange of rotating anodes requires troublesome operations such as stopping a vacuum and a target replacement. The DW rotating anode enables us to select a preferred target by turning a switch called target selector (Fig. 1). This makes it much easier to change the target than a conventional rotating anode system.

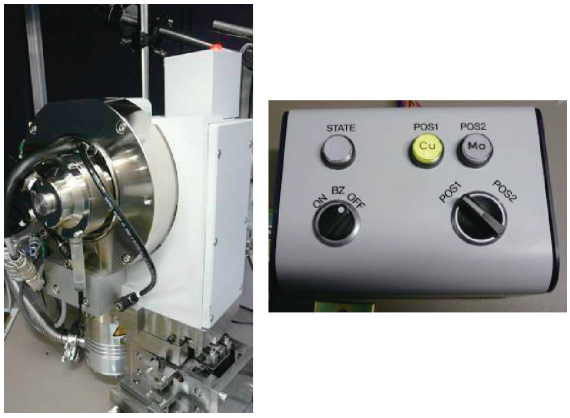

Fig. 1. DW target and its selector switch.

2.3. Confocal Max-Flux optics for dual wavelength (VariMax DW)

The optics can easily be switched between two wavelengths as well as the rotating anode. Once optics alignment has been achieved, user can start a measurement with a minor adjustment.

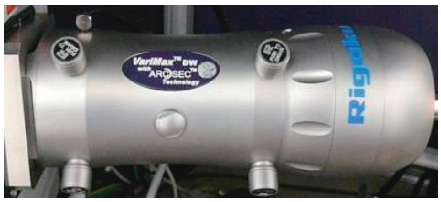

Fig. 2. VariMax DW.

2.4. Data collection of a tiny crystal

Combination of 70 μm focal spot and Confocal Max-Flux optics enables measurement of very tiny crystals with dimensions of approximately 20 μm. In addition, combination of an FR-E+ SuperBright Ultrahigh-Intensity Rotating Anode X-Ray Generator and the optics makes it possible to measure crystals even smaller.

3. Applications

The dual wavelength X-ray generator equipped with the curved large area image plate detector can cover the

widest applicable fields: the absolute structure determination with Cu radiation, screening protein crystals and successive data collection, ultra high 0.38 Å resolution data collection using Mo radiation, and so on. Three typical applications are described below.

3.1. Organic compound measured with two wavelengths

Table 1 summarizes results of data collection of ursodeoxycholic acid with both Cu and Mo radiations. The data collection took only one hour and a half for Mo radiation. When absolute structure determination is needed, one can switch to Cu radiation to utilize stronger anomalous signal. The dual wavelength system makes it easy to acquire a highly reliable structure by selecting appropriate wavelength or by using both wavelengths.

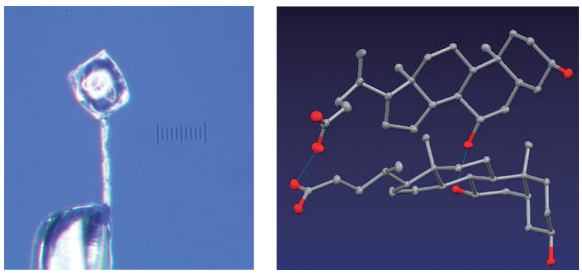

Fig. 3. A crystal of ursodeoxycholic acid (left) and its molecular structure (right).

Table 2. Data collection conditions and results of structure determination of ursodeoxycholic acid.

Sample	ursodeoxycholic acid	
Formula and MW	$C_{24}H_{40}O_4$ / 392.58	
Space group	$P2_12_12_1$ (#19)	
Cell parameters	a = 12.1470(2) Å b = 13.2059(2) Å c = 26.609(2) Å V = 4268.4(3)Å^3	
Temperature (K)	93	
Crystal size (mm)	0.35 x 0.34 x 0.30	
X-ray source	MoKα	CuKα
Measurement time	1 h 34 min	2 h 47 min
Max. resolution (Å)	0.83	0.83
Rmerge (%)	1.94	2.25
R1(>2σ(I)) (%)	3.02	3.24
wR2 (%)	7.69	8.27
Flack parameter		0.01(11)

3.2. Proteins

The dual wavelength system delivers extremely intense X-rays produced by the combination of highbrilliance microfocus X-ray generator and a VariMax DW optic. This system can be used not only for checking protein crystals but also for serious data collection.

Figure 4 is the diffraction image from a thaumatin crystal. One can readily notice that neighbouring spots correspoinding to the 150 Å axis are clearly resolved.

Table 3 denotes measurement conditions and results of structure analysis of thaumatin. The data collection finished in less than 7 hours but the maximum resolution reached to 1.85 Å and the Rmerge was as low as 6.4%. The structure analysis was performed by using CCP4.

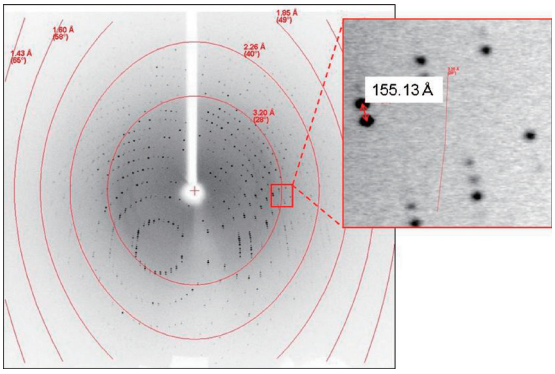

Fig. 4. X-ray diffraction images of a thaumatin crystal.

Table 3. Data collection conditions and results of structure determination of thaumatin.

Sample	Thaumatin
Molecular weight	22204 (207 amino acid residues)
Space group	$P4_12_12$ (#92)
Cell parameters	a = 57.568 Å c = 150.476 Å
Crystal size (mm)	0.3 x 0.2 x 0.2
X-ray source	CuKα
Measurement conditions	0.5° oscillation x 60s. exposure x 180 frames
Measurement time	6h 44min
Max. resolution (Å)	1.85 (Rmerge<30%)
Rmerge (%)	6.4
R(%) / R_{free}(%)	19.9 / 23.1

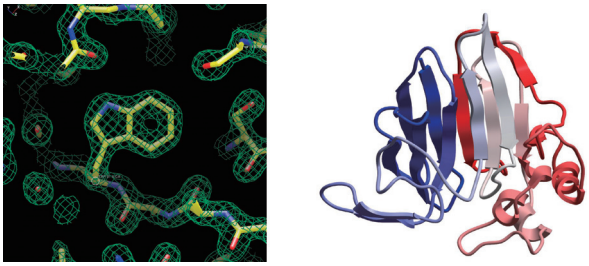

Fig. 5. Electron density (left) and a ribon model (right) of thaumatin.

The phase determination by molecular replacement (Molrep) and least-squares refinement (Refmac) were carried out successively.

Figure 5 shows electron density and overall structure of the molecule. The quality of electron density surrounding the tryptophan side chain confirms the high quality and resolution of the resulted structure.

3.3. Ultra high resolution structure

Table 4 shows the results of data collection on a potassium tetrachloroplatinate crystal. Table 4 sumarrizes data collection to 0.83 Å and 0.46 Å resolution. The number of observed reflections was largely increased for the 0.46 Å data compared to that for the 0.83 Å. Although the R1 value remains quite similar, the data to parameter ratio was strikingly improved and the standard deviation of Pt–Cl bonds became smaller.

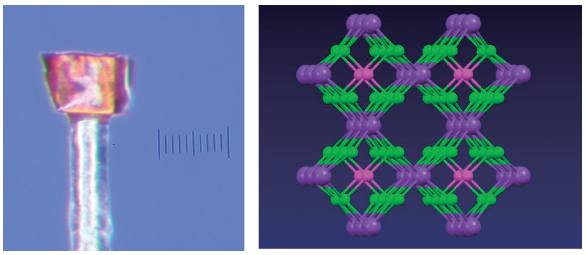

Fig. 6. A potassium tetrachloroplatinate crystal (left) and its structure (right).

Table 4. Data collection conditions and results of structure determination of potassium tetrachloroplatinate.

Sample	Potassium tetrachloroplatinate	
Molecular weight	K_2PtCl_4 / 415.1	
Space group	*P*4/mmm (#123)	
Cell parameters	a = 7.02276(13) Å c = 4.14235(8) Å V = 204.297(7) Å^3	
Temperature (K)	293	
Crystal size (mm)	0.15 x 0.10 x 0.10	
X-ray source	MoKα	
Resolution (Å)	0.83	0.46
Total/Unique reflections	1668/136	18292/691
Reflections I>2σ(I) (%)	136 (100)	691 (100)
Rmerge (%)	5.18	5.84
R1(>2σ(I))(%)	1.54	1.55
wR2(%)	3.47	3.53
Data:parameter ratio	12.4	62.8
Bond length (Å)		
Pt – Cl	2.309(2)	2.3091(4)
K – Cl	3.238(2)	3.2380(3)