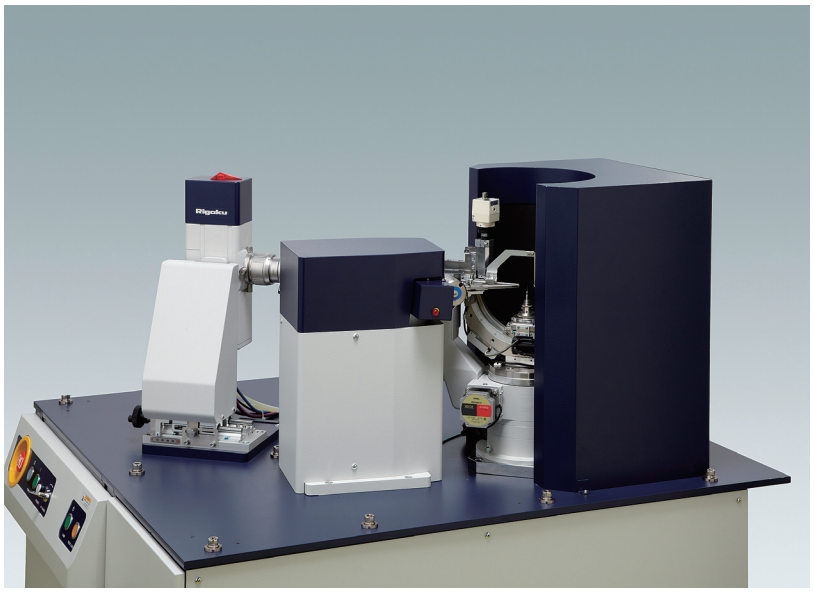
Curved imaging plate X-ray diffraction system

DualSource RAPID II



1. System features

1.1. DualSource RAPID II

A newly developed DualSource RAPID II system utilizes two types of sealed tubes X-ray sources: for Cu radiation a 30 W micro-focus sealed tube generator with multilayer optics is used (MicroMax-003) and for Mo radiation a 3 kW sealed tube with a curved monochromator (SHINE) is used. It is well known that selecting a suitable X-ray source is essential to successful experiments. The Cu source is useful for absolute structure determination and resolving crystals with large unit cells. On the other hand, a Mo source is applicable to crystals that have high absorption or where very high-resolution data is needed, for example for a charge density study.

This DualSource system enables us to collect data using two different wavelengths within one system and one data collection. 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.

The representative specifications of the DualSource RAPID II system are summarized in Table 1.

The large detector aperture allows us to collect a wide 2θ range of data rapidly. The large aperture combined with Mo data collection produces diffraction resolution up to 0.45 Å, which is applicable for charge density studies. The combination of dual wavelength and a large aperture image plate system provides many advantages for various diffraction experiments.

Table 1. Specifications of DualSource RAPID II system.

X-ray source	Cu	Mo
X-ray generator	30 W	3 kW
Optic	Multilayer mirror	Curved graphite
Goniometer	Partial chi goniometer	
Detector distance	127.4 mm	
2θ angle	−60 ~ +144 deg	−90 ~ +114 deg
Dynamic range	1.05×10^6	
Detector aperture	460 mm × 256 mm	

1.2. Cu X-ray source

The Cu X-ray source is Rigaku's MicroMax-003, which is composed of a 30 W micro-focus generator and a confocal multilayer optic. The X-ray intensity through a ϕ0.1 mm pinhole at the sample position exceeds that of 5.4 kW rotating anode generators. Additionally, the MicroMax-003 has advantages of low power consumption, long tube life and low-maintenance.

1.3. X-ray source Mo

The Mo X-ray source is a 3 kW X-ray generator with a SHINE optic. It provides up to 3 times the intensity compared to a conventional graphite monochromator. Beam size at the sample position is variable by changing

the incident beam collimator (ϕ0.3 mm, ϕ0.5 mm, ϕ0.8 mm) allowing a user to measure a sample with a crystal size up to ϕ0.8 mm.

2. Applications

The DualSource RAPID II provides you with the opportunity to solve a wide range of important research problems due to its extremely flexible capabilities. The many possible applications include protein crystals as well as powder samples in addition to single crystals of small molecules.

2.1. Standard three dimensional structure determination from single crystals

The DualSource RAPID II allows you to easily access the most appropriate wavelength depending on your samples, without exchanging an X-ray tube. This flexibility is especially useful in multi-user facilities or satellite laboratories.

Table 2 shows the summary of the results of structure determination from single crystal data of a Ni complex with both Cu and Mo radiation. Faster data collection can be achieved with Cu radiation. On the other hand, diffraction data collected with Mo radiation can give a highly reliable refined crystal structure. If you are unsure which X-ray source you should use, you can collect data with both radiations at sequentially without re-centering of the crystal.

2.2. Absolute structure determination of chiral compounds from very small crystals

As shown in Fig. 1, with the MicroMax-003 Cu micro-focus X-ray tube, enough intensity can be obtained at higher angles even if a sample is very small organic crystal. The absolute configuration of compound 9 in the synthesis of keramamine C[1] (Fig. 2) was determined by refining the Flack parameter to 0.00 (17) (Table 3).

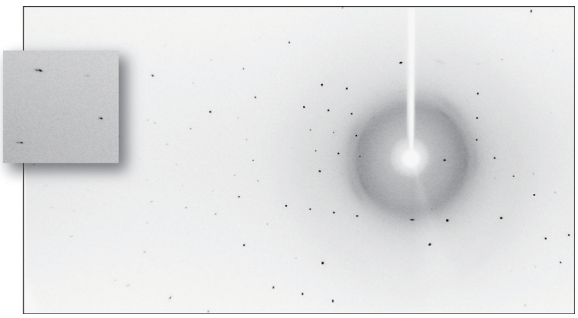


Fig. 1. X-ray diffraction image of a synthetic intermediate of keramamine C.

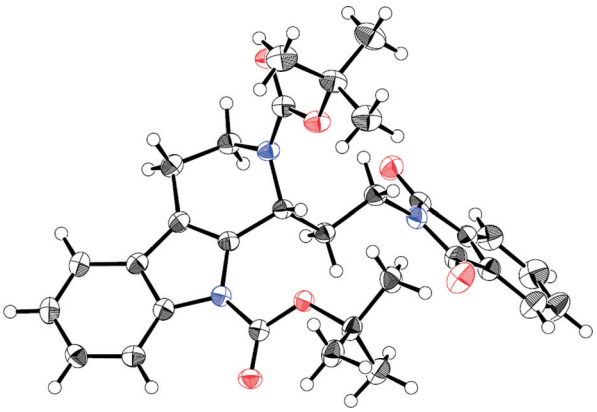


Fig. 2. ORTEP diagram of a synthetic intermediate of keramamine C.

Table 2. Data collection and structure refinement statistics of a Ni complex.

X-ray source	Mo$K\alpha$	Cu$K\alpha$
Crystal dimensions (mm)	0.30×0.30×0.26	
Exp. time (sec/deg)	10	
Oscillation angle (deg)	3	30
Images	74	30
Measurement time (h)	2.7	1.5
Maximum resolution (Å)	0.83	
Unit cell constants	a=8.30 Å, b=8.81 Å, c=9.27 Å α=117.80°, β=94.01°, γ=92.63° V=595.5 Å^3, Z=1	
Space group	P-1 (#2)	
R_{merge} (%)	1.55	5.09
R_1 (%)	2.62	3.85
$_wR$ (%)	6.37	9.78
GOF	1.071	1.054
No. of observations	2140	2138
Completeness (%)	98.3	98.0

2.3. Macromolecular structure determination

It is also possible to collect diffraction data of protein crystals with unit cell length as large as 150 Å (Fig. 3). The quality of data from the MicroMax-003 Cu (Rmerge=4.8% @2.3 Å resolution) is almost as good as that from the MicroMax-007 rotating anode X-ray generator equipped with the VariMax optic (Rmerge=4.4% @2.0 Å resolution). As shown in Fig. 4, the crystal structure of thaumatin could be determined by iodine SAD phasing.

Table 3. Data collection and structure refinement statistics of a synthetic intermediate of keramamine C.

X-ray source	CuKα
Crystal dimensions (mm)	0.09×0.05×0.03
Exp. time (sec/deg)	120
Oscillation angle (deg)	5
Unit cell constants	a=11.4197 (2) Å, b=11.8949 (2) Å, c=12.4088 (2) Å α=70.775 (5)°, β=66.477 (5)°, γ=76.942 (5)° V=1450.58 (8) Å^3, Z=2
Space group	P1 (#1)
R_{merge} (%)	3.78
R_1 (%)	4.80
Flack parameter	0.00 (17)
Completeness (%)	98.4

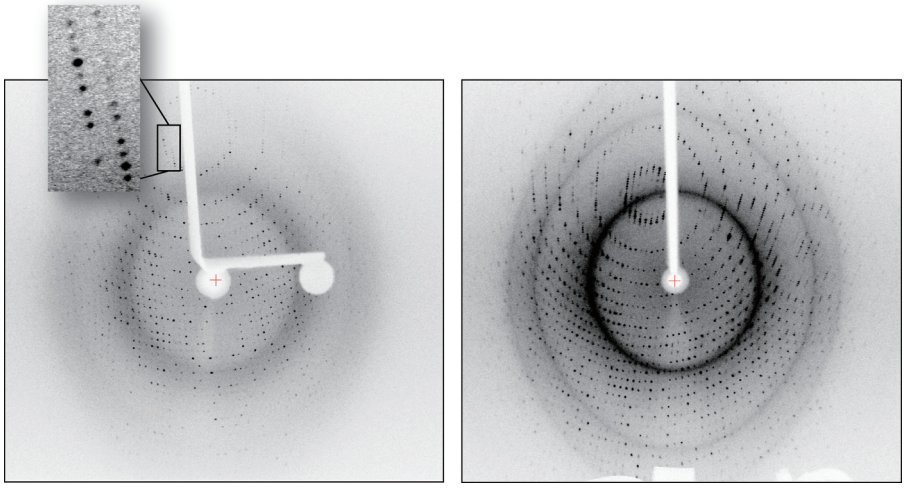


Fig. 3. X-ray diffraction images of a thaumatin crystal collected with the DualSource RAPID II (left) and the VariMax with RAPID (right).

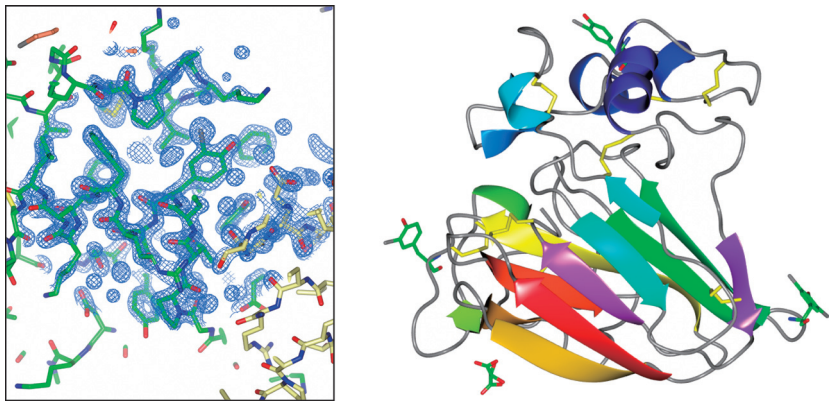


Fig. 4. Superposition of electron density map and molecular model (left) and ribbon model of thaumatin (right).

2.4. Phase identification of powder samples

Qualitative phase identification can be done with less than 1mg of a powdered sample. Well-ground sulfanilamide powder in a glass capillary showed a different diffraction pattern from that at 27℃ after heat treatment at 130℃ (Fig. 5). As shown in Fig. 6, the phase transition from β-form to γ-form was confirmed by comparing the powder diffraction data with the calculated peak positions and relative intensities from single crystal data.

References

(1) H. Ishiyama, Y Mori, T. Matsumoto and J. Kobayashi: *Heterocycles*, **86** (2012), 1009−1014.

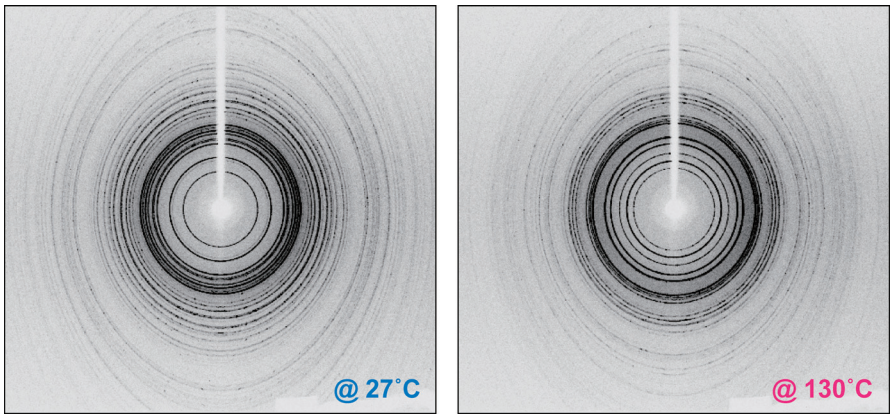


Fig. 5. X-ray diffraction images of sulfanilamide powder at 27℃ (left) and 130℃ (right).

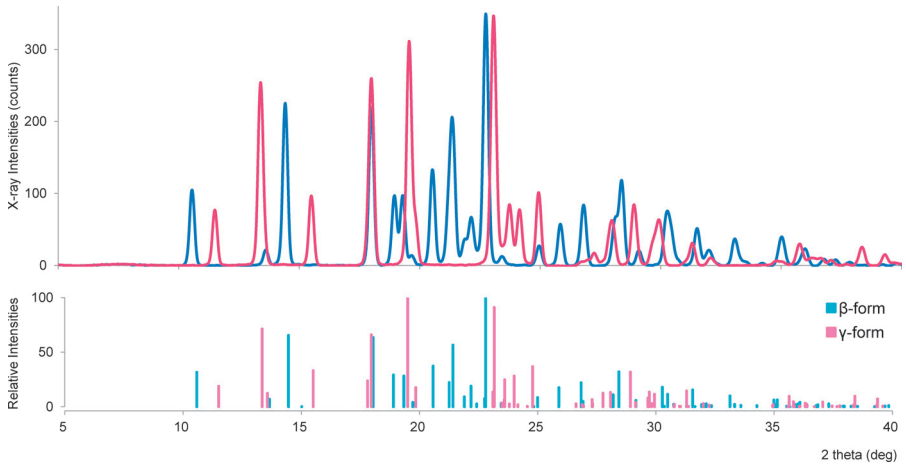


Fig. 6. X-ray powder diffraction patterns of sulfanilamide at 27℃ (—) and 130℃ (—) (upper) and calculated peak positions and intensities from single crystal data of β- and γ-form (lower).