

Application News

No. X258

X-ray Analysis

High-Speed X-Ray Diffraction Analysis of Medicine Using OneSight Wide-Range High-Speed Detector

■ OneSight Wide-Range High-Speed Detector

The OneSight wide-range high-speed detector consists of a semiconductor array with more than 1000 channels. Compared with the conventional scintillation detector, higher-speed measurement is achieved due to its sensitivity, which is more than twenty times that of the scintillation type. In qualitative analysis, the scan mode can be selected from among the High Resolution, Standard, and Fast modes, in addition to the One-Shot mode in which the goniometer is fixed in place and the OneSight detector acquires a wide-angle exposure, thereby permitting high-speed, high-resolution measurement for a wide variety of samples. Here, we introduce examples of a high-speed X-ray diffraction analysis of pharmaceutical products.

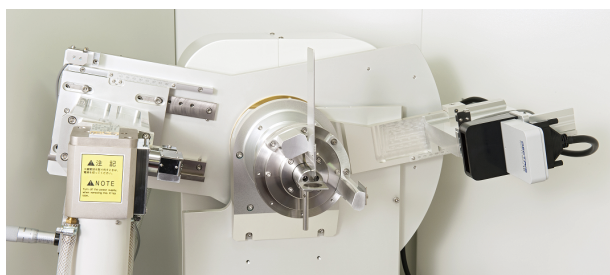


Fig. 1 XRD-7000 Equipped with OneSight

■ Polymorphism Analysis of Medicine

Among pharmaceuticals and organic substances, there are many cases of crystal polymorphism in which the crystal system of the substances may differ even if the chemical formula is the same. Crystal polymorphs display different characteristics, such as efficacy and solubility in the body, etc., and depending on the situation, this may lead to patent infringement. Therefore, measurement of pharmaceutical products using an X-ray diffraction instrument is important from the standpoint of quality management.

Here, we conducted measurements of ranitidine having two polymorphic forms (Form 1, Form 2) and a mixture of two crystalline polymorphs using the OneSight wide-range high-speed detector.

Table 1 Analytical Conditions

Instrument	XRD-7000
X-Ray Target	Cu
Tube Voltage, Tube Current	30 kV, 40 mA
Monochromatization	Ni filter
Measurement Range	2θ: 10 to 50 degrees
Scan Speed	43 degrees/minute
Detector	OneSight wide-range high-speed detector
Measurement Mode	Step-scan (Standard mode)

■ Results of Polymorphism Analysis

The diffraction patterns of Form 1 and Form 2 pure substances are shown in Fig. 2. Such a comparison of crystal polymorphs clearly illustrates the differences in diffraction peak positions even among substances having the same composition.

In addition, Fig. 3 shows a comparison of the diffraction patterns (enlarged region from 10 degrees to 28 degrees) of a mixture of Form 1 and Form 2 and of an overlay display of the patterns of Form 1 and Form 2, respectively. Peaks of both Form 1 and Form 2 were observed in the mixture.

Furthermore, we also conducted this measurement using the conventional scintillation detector, and compared the measurement speeds.

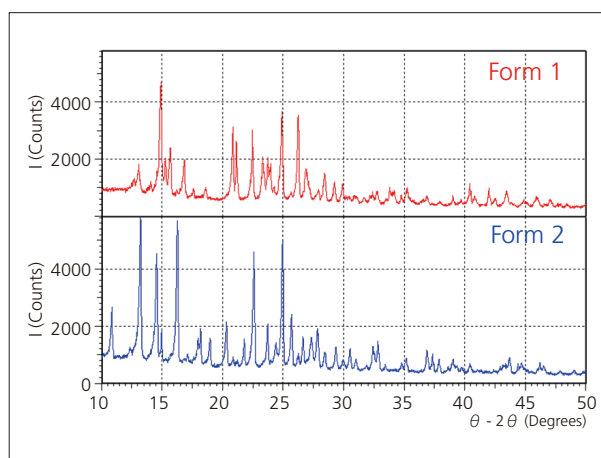


Fig. 2 Diffraction Patterns of Form 1 and Form 2

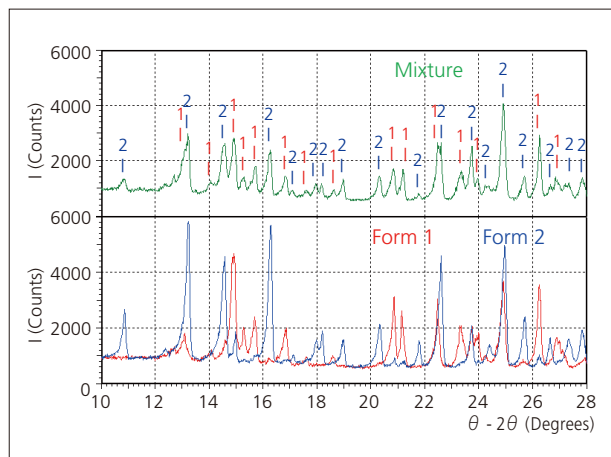


Fig. 3 Diffraction Pattern of a Mixture of Form 1 and Form 2 (above) and Overlay Pattern of Form 1 and Form 2 (below)

Using a conventional scintillation detector, this measurement took twenty minutes, while the same measurement was completed within just a few minutes using the OneSight wide-range high-speed detector. Fig. 4 shows the overlaid diffraction patterns of Form 1 obtained using the OneSight high-speed detector (43 degrees/min) and the scintillation detector (2 degrees/min), respectively. The OneSight detector incorporates a high-speed (Fast) mode for faster measurement, and a high-resolution mode for precise analysis of each peak, and measurement can be conducted while switching back and forth between these modes.

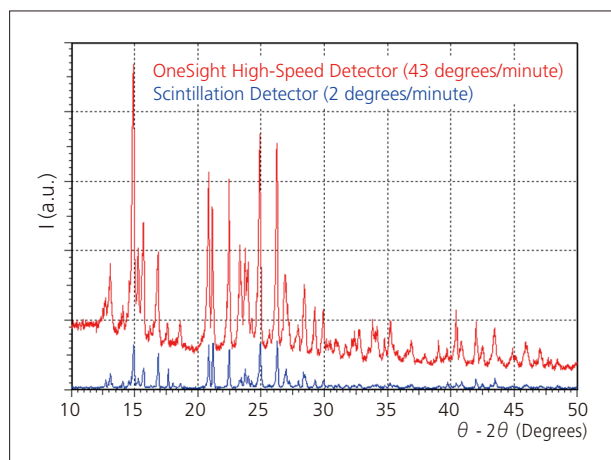


Fig. 4 Diffraction Pattern by OneSight High-Speed Detector and Conventional Scintillation Detector

High-Speed X-ray Diffraction Analysis of Medicine Using One-Shot Mode

Using the One-Shot analysis mode, in which the detector is fixed on the target peak position using a wide acquisition angle, it is possible to conduct quick, high-resolution measurement over a fixed range of angles. Here, we compared measurement of Drug A using the step-scan and One-Shot measurement modes.

Table 2 Analytical Conditions

Mode	Step-Scan (Standard mode)	One-Shot
Instrument	XRD-7000	
X-Ray Target	Cu	
Tube Voltage, Tube Current	30 kV, 40 mA	
Monochromatization	Ni filter	
Measurement Range	2θ: 15 to 25 degrees	
Integration Time	55 seconds	10 seconds
Detector	OneSight wide-range high-speed detector	

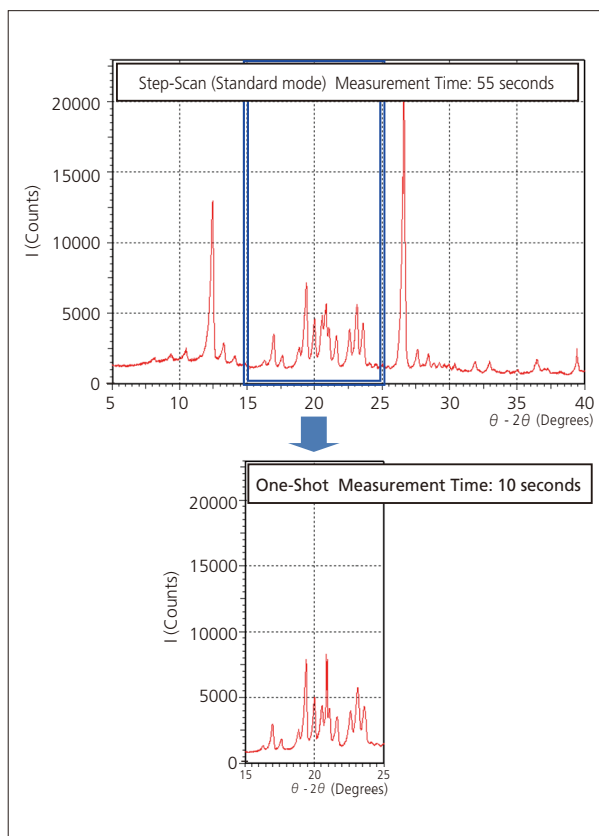


Fig. 5 Measurement Time Comparison Using One-Shot Mode and Step-Scan Mode

When conducting verification measurement of a particular peak, measurement can be conducted quicker using the One-Shot mode than with the step-scan mode because scanning by the goniometer is obviated in the One-Shot mode.