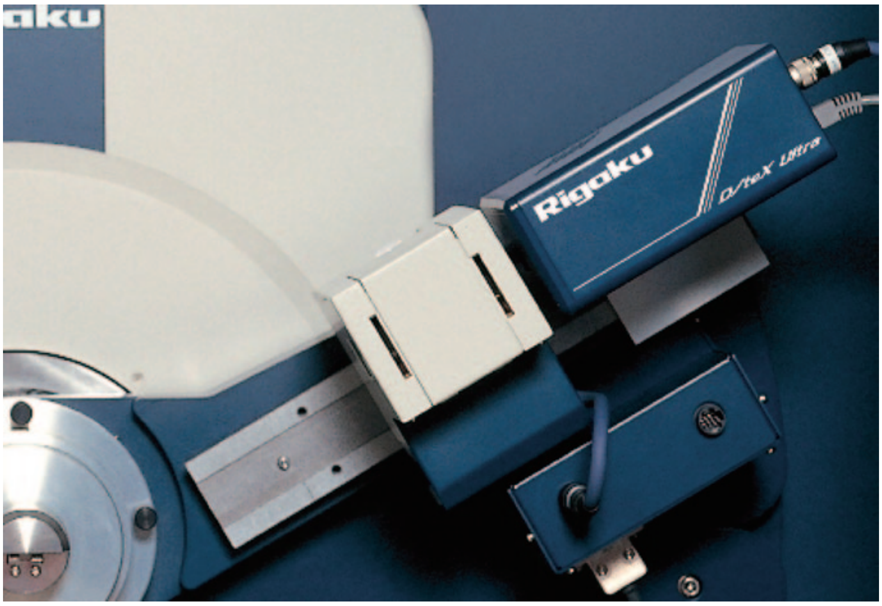
Advanced and high-performance X-ray detector

D/teX Ultra

For measurements of a small amount of samples and small areas
High speed and advanced analysis with X-ray diffractometer



1. Introduction

D/teX Ultra is a high-speed one-dimensional X-ray detector using a state-of-the-art semiconductor device, and has a superior X-ray detection capability and energy resolution than a conventional one-dimensional semiconductor X-ray detector. The advantages of a D/teX Ultra detector include: a drastic reduction of the measurement time, an acquisition of high-intensity diffraction data, and a reduction of background intensity. Therefore, D/teX Ultra is one of the most, if not the most, advanced detectors, and it can be used for the measurements of a large number of samples and an *in-situ* dynamical study of phase transition as a function of temperature or humidity. The detector can be used and mounted on a horizontal-sample general-purpose X-ray diffractometer, namely Ultima IV, a theta/theta rotating anode XRD system, TTRAX III.

2. Advantages

2.1. High intensity/high speed measurement

A D/teX Ultra detector has about 100-times X-ray detection sensitivity than that of a standard Rigaku scintillation counter. In other words, X-ray intensities obtained by a D/teX Ultra detector are 100 times stronger than those obtained by a scintillation counter. Therefore, the measurement time can be greatly reduced, or the measurement speed can be drastically increased when a D/teX Ultra detector is used. For example, the experimental time of measuring an XRD pattern for a qualitative analysis of a sample requires about 30 minutes when a scintillation counter is used. However, an equivalent XRD pattern with comparable intensity data can be obtained in just about one minute when D/teX Ultra is used. Figure 1 shows the Si (111) diffraction peaks recorded by a scintillation counter and D/teX Ultra. The

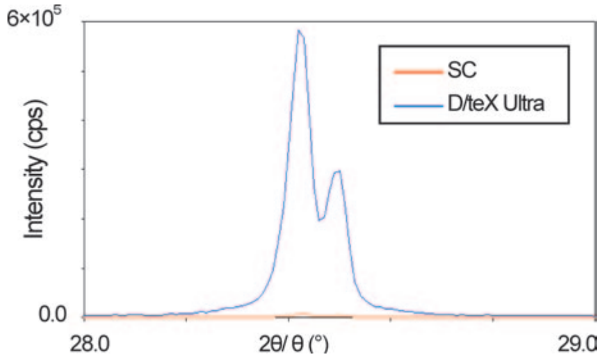


Fig. 1. Comparison of the diffraction peak intensities between a SC (scintillation counter) and a D/teX Ultra.

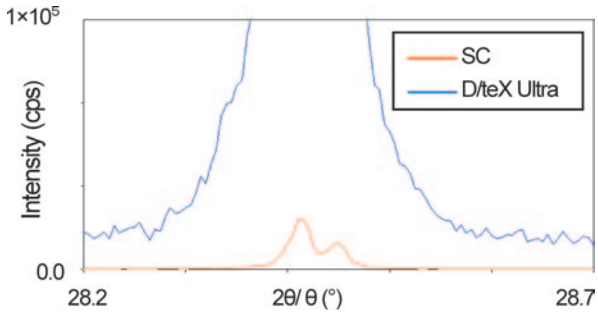


Fig. 2. An expanded intensity scale for Fig. 1. Sample: NIST 640c Si Standard Material.

Si (111) peak intensity obtained by D/teX Ultra was almost 6×10^6 counts per second (cps), while the Si (111) intensity obtained by a scintillation counter was so low that it is hardly visible in Fig. 1. The Si (111) peak obtained by a scintillation counter can be visualized when the intensity scale of Fig. 1 is expanded to 1×10^5 cps (see Fig. 2). The intensity improvement obtained by D/teX Ultra is about 100 times over a scintillation counter. The Si (111) peaks were recorded using the same 2-kW X-ray generator.

2.2. High energy resolution measurement

A Cu-target X-ray source is commonly used in an X-ray diffraction analysis. However, Cu K X-rays can produce secondary X-ray fluorescence from a sample, especially a sample containing Fe. Since D/teX Ultra can reduce the energy of this fluorescent X-rays because of its high-energy resolution, low background XRD data can be obtained without the need of replacing the X-ray tube or using a diffracted-beam monochromator.

Three energy-resolution options can be selected in a D/teX Ultra detector: the normal mode used for a general measurement, the high-intensity mode and the fluorescence reduction mode. Figure 3 shows a comparison of the XRD patterns from a Fe_2O_3 sample obtained by using the fluorescence reduction mode and the normal mode. When the fluorescence reduction mode was used, the background intensities were greatly reduced. A 2-kW X-ray generator was used, and each of the measurements was about 1.5 minute.

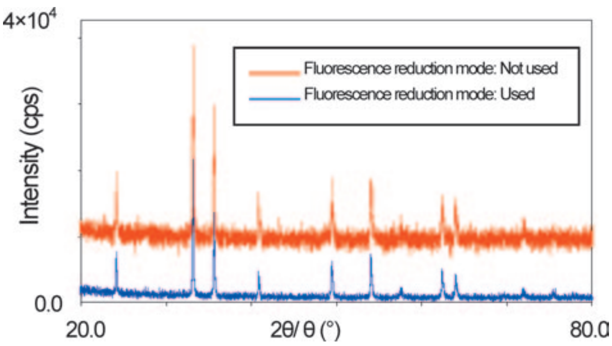


Fig. 3. Reduction of background using the fluorescence reduction mode with a Fe_2O_3 powder sample.

3. High-sensitivity measurements

The high detection sensitivity and the high energy resolution of D/teX Ultra are also effective for measuring the data of a microvolume sample and/or a micro region in a sample.

3.1. Detection of 0.2% polymorphic impurities in a raw drug substance of a pharmaceutical product

Figure 4 shows an example of measuring a sample of type I tolbtamide (a drug for diabetes) in which 0.2 wt% type II tolbtamide was added as a polymorphic impurity. Conventionally, to detect trace amounts of impurities of less than 1%, a powerful 18-kW rotating anode X-ray source would be needed. When a D/teX Ultra detector was used, however, a diffraction peak from type II tolbtamide was detected. The experimental conditions were a 2-kW X-ray source and a fast scan with speed of 2° per minute.

3.2. Identification of crysotile in serpentinite

Serpentine, used as an admixture for mortar plastering by smashing, has three polymorphs: antigorite, lizardite (non-asbestos) and crysotile (asbestos).

By using a D/teX Ultra detector together with a micro measurement optical system, mineral species in a small black-color portion (Fig. 5) of less than 1 mm in diameter on a serpentinite sample can be analyzed rapidly without the need of smashing it. The measure-

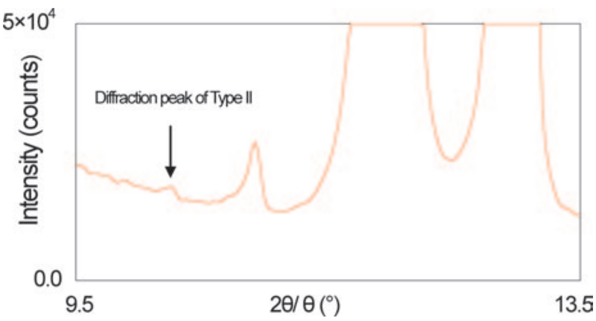


Fig. 4. X-ray diffraction pattern of type I tolbutamide with 0.2 weight percent type II tolbutamide (tolbutamide: a hypoglycemic drug).

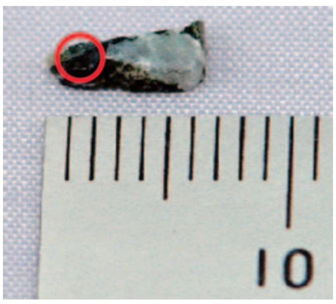


Fig. 5. Black portion of a serpentinite sample
(Red circled portion represents the measured point).

ment results reveal that the black portion is composed of crysotile, antigorite and lizardite (Fig. 6). By the way, the white portion of the sample consists mainly of calcite.

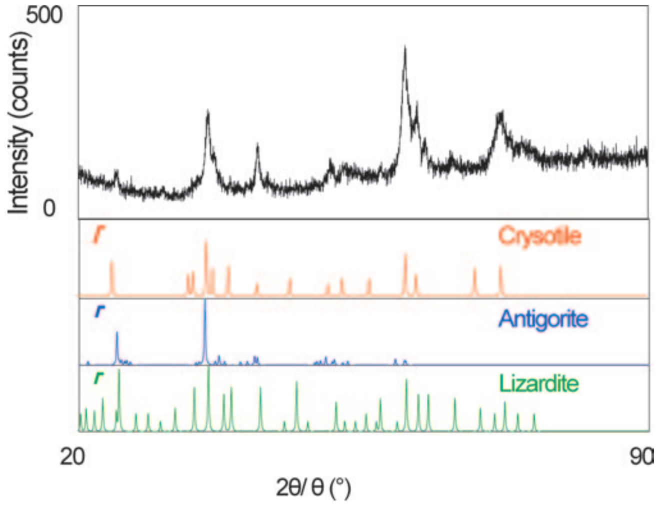


Fig. 6. Phase Identification on the black portion in a serpentinite sample.