SHIMADZU

Application News

X-ray Diffraction (XRD) Spectrometry

Measurement Examples Using Polycapillary Parallel-beam Optics System

(Foods, Pharmaceuticals, Organisms)

Shimadzu Analytical & Measuring Center

The term "polycapillary" indicates multiple (poly) fine glass tubes (capillaries) that guide X-rays. X-rays generated by a point source are accepted at a high solid angle by the polycapillaries, and emerge as a parallel beam at the opposite end. This optics system utilizes the X-rays generated by the X-ray tube more efficiently than the standard (concentrating) method (the Bragg-Brentano Method), to achieve higher diffraction X-ray intensities. The parallel-beam polycapillary method used by the optics system ensures that the angle of diffraction does not change due to displacements of the sample measurement surface. This characteristic allows highly sensitive and accurate measurements of samples with curved or irregular surfaces, and improves the separation of the diffracted lights and shift in the angles of diffraction inherent in the concentrating method. These features of the polycapillary parallel-beam optics system permit the direct measurement of complex-shaped samples, such as foods, pharmaceuticals, or organisms.

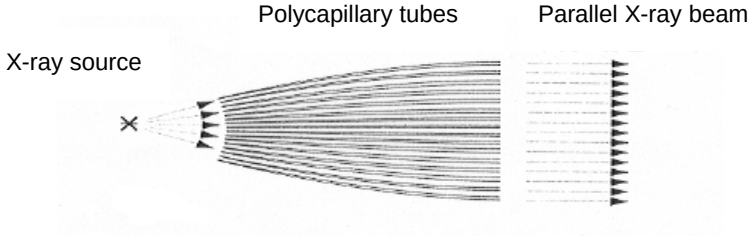

Figure 1: Concept of the Polycapillary Parallel-beam Optics System

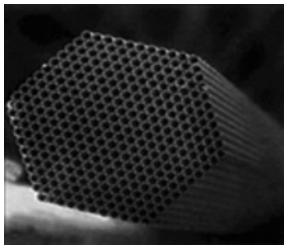

Figure 2: SEM image of Polycapillary Tubes

■ Measurement of Drugs in Tablet Form

Many pharmaceuticals and organic substances are crystal polymorphs, that is, they can take different crystalline forms despite having the same chemical formula. These different crystalline forms result in different characteristics, such as efficacy or dissolution rate in the body, and can even lead to patent infringements in some cases. Consequently, quality control using X-ray diffractometer analysis is indispensable. Control of raw materials has always been required but, recently, control of final products is often also demanded. However, when tablets or other samples with no flat measuring surface are measured using conventional concentrating techniques, the resulting shift in the angles of diffraction or drop off in intensity makes practical measurements impossible. The analysis of such samples using the polycapillary parallel-beam method is shown in the diagram below.

Figure 3: Appearance of Ranitidine Tablets

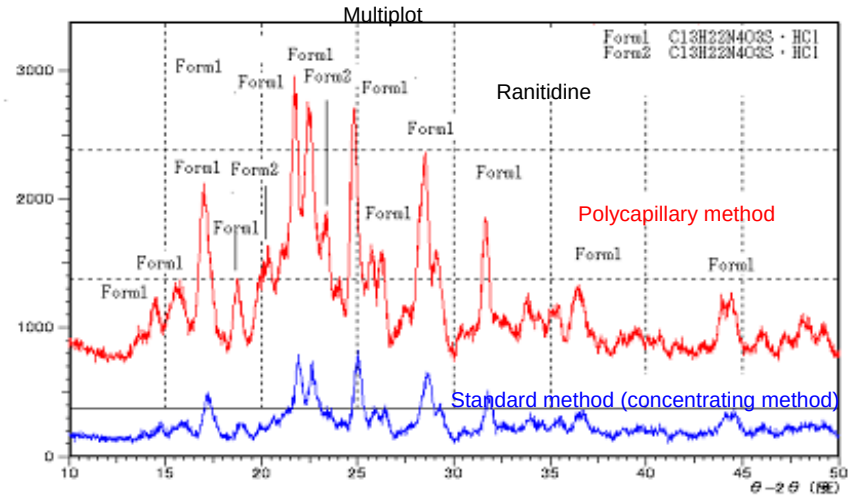

Figure: 4: Crystal Polymorph Measurement Data on Ranitidine Tablets

Fig. 4 shows measurements on the H2 antagonist ranitidine contained in gastrointestinal medications, using the polycapillary and standard methods. Form 1 is the principal component of this sample. The measurements were conducted to determine whether the crystal polymorph, Form 2, also exists. The polycapillary method confirmed the characteristic Form 2 diffracted light (near $2\theta = 20°$), clearly indicating the existence of Form 2. The lower sensitivity of the standard (concentrating) method was unable to clearly confirm the existence of Form 2.

The measurement profile obtained by the polycapillary method clearly distinguishes between the halo peaks from the amorphous areas and the peaks from the crystalline areas, and proved suitable for accurate degree of crystallinity calculations.

■ Measurement of Teeth

During measurements on the tip of a tooth using the standard method, obstruction of the X rays by the central depression and raised periphery resulted in a significant reduction in intensity. However, the polycapillary method is relatively unaffected by irregularities of the sample surface and was able to detect small peaks near $2\theta = 28^{\circ}$ and 47°, which were not detected using the standard method. In addition, the angles of diffraction determined by the standard method were

shifted toward lower angles, compared to the standard sample IC_DD database card No. 76-694 (lines in the diagram). The shift in the angle of diffraction is assumed to result from effects of the diffracted lights reflected from the depression in the tooth.

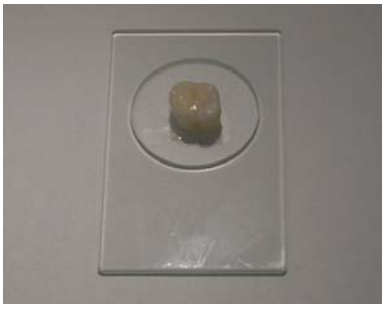

Figure 5: Photograph of Tooth Appearance

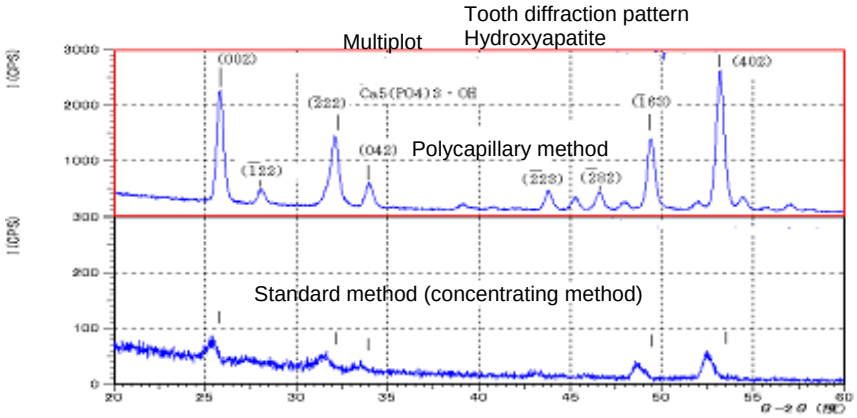

Figure 6: Measurement of the Tooth

■ Measurement of Chocolate Ball

Measurement using the normal method of a chocolate ball, as shown in Fig. 7, detected virtually no diffracted lights due to crystallinity. However, the polycapillary method could clearly detect the diffracted lights of the sucrose content of the chocolate, as shown in Fig. 8. The degree of crystallinity of the sucrose was approximately 50%. The dramatic drop in diffraction intensity with the standard method is thought to occur because only the central part of the sample surface contributes to the diffraction due to the curvature of the surface.

Figure 7: Appearance of Chocolate Ball

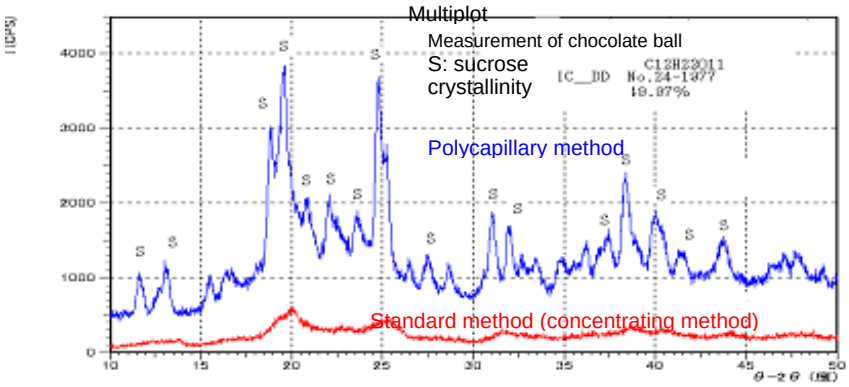

Figure 8: Measurement of Chocolate Ball (Degree of Crystallinity)

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