Structure Analysis of Pharmaceutical Compounds from Powder Diffraction Data

Noriyuki Imayoshi, Kyoko Sakamoto, Kenji Suzuki, *Chemistry Research Laboratories, Dainippon Pharmaceutical Co., Ltd.* E-mail: noriyuki-imayoshi@dainippon-pharm.co.jp

In recent years, the crystal form of pharmaceutical compounds has become important not only for identification of the quality but also for accession of the patent. However, powder diffraction data using standard in-house instruments has low resolution as compared to the synchrotron radiation data. Accordingly, we have been studying the usefulness of the synchrotron radiation and have identified the crystal form of pharmaceutical compounds. In this study we investigated polymorphs of carbamazepine, taurine and acetaminophen as an example of pharmaceutical compounds. Additionally, we made a study on the structure determination of carbamazepine and taurine using the synchrotron diffraction data.

All pharmaceutical compounds, except for carbamazepine form III, were purchased from Wako Pure Chemical Industries (Tokyo, Japan). Carbamazepine form III was prepared by heat treatment of form I at 443 K for 2 hrs. The concomitant samples of carbamazepine were prepared by mixing form I in form III with mortar and pestle moderately. Powder X-ray diffraction patterns were collected with BL24XU of SPring-8 using milled powder samples packed in a quartz glass capillary.

As the result of analysis for concomitant polymorphs of carbamazepine, the peaks of 0.5 % form I at 12.06° and 12.3° (2Theta) are detected. By Rietvelt refinement, Rwp of the carbamazepine (form III) is 6.00 % (Rp=4.02 %).

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