Powder X-ray diffraction of azelastine hydrochloride, $C_{22}H_{25}ClN_3O \cdot Cl$

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Commercial azelastine hydrochloride crystallizes in the monoclinic space group $P2_{1/n}$ (#14) with a = 13.7844(5), b = 16.39920(14), c = 9.41231(22) Å, β = 97.5340(20)°, V = 2109.32(4) Å^3, and Z = 4. The lattice parameters differ by −0.02, +0.04, and +0.04% from those in the previous determination (reflecting differences in the temperature and the sample source), and are more precise, from the use of synchrotron radiation. The experimental powder pattern is included in the Powder Diffraction File™ (PDF®) as entry 00-070-1219. © *The Author(s), 2020. Published by Cambridge University Press on behalf of International Centre for Diffraction Data.* [doi:10.1017/S0885715620000664]

Key words: azelastine hydrochloride, X-ray diffraction, powder diffraction file

Azelastine hydrochloride (Astelin® and additional trade names) is an antihistamine used to relieve nasal symptoms caused by allergies, administered as a dilute nasal solution. Commercial azelastine hydrochloride crystallizes in the monoclinic space group $P2_1/n$ (#14) with a = 13.7844(5), b = 16.39920(14), c = 9.41231(22) Å, β = 97.5340(20)°, V = 2109.32(4) Å^3, and Z = 4. A search of the powder data against the Powder Diffraction File™ (Gates-Rector and Blanton, 2019) and a reduced cell search in the Cambridge Structural Database (Groom *et al.*, 2016) yielded a previous structure determination (Maccaroni *et al.*, 2009; PDF® 02-099-8015 and CSD POVRUO). In this work, the sample was ordered from USP (Lot #F0L494) and analyzed as-received. The room temperature (295 K) crystal structure was refined using synchrotron (λ = 0.412849 Å) powder diffraction data. Hydrogen positions were included as part of the structure and were recalculated during the refinement. The diffraction data were collected on beamline 11-BM at the Advanced Photon Source, Argonne National Laboratory. Figure 1 shows the powder X-ray diffraction pattern of the compound.

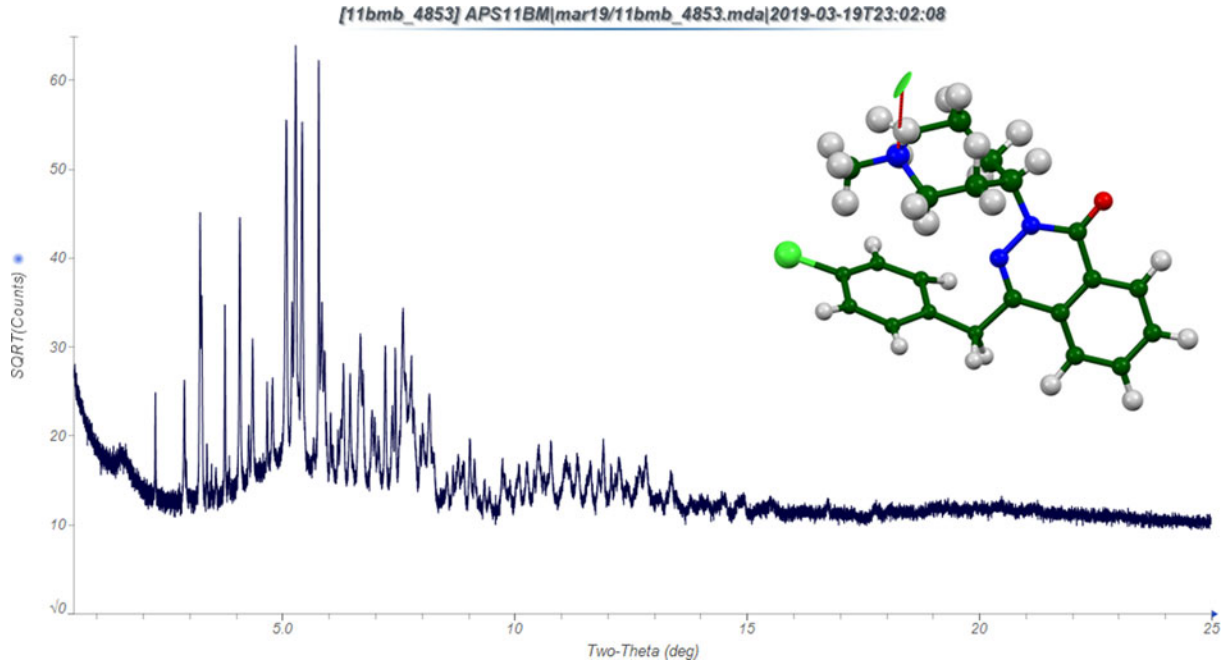

Figure 1. Powder X-ray diffraction pattern of azelastine hydrochloride, with the molecular structure.

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The lattice parameters differ by −0.02, +0.04, and +0.04% from those in the previous determination (reflecting differences in the temperature and the sample source), and are more-precise, from the use of synchrotron radiation. The two molecular structures are essentially identical, with a root-mean-square Cartesian displacement of 0.141 Å. The experimental powder pattern is included in the Powder Diffraction File™ (PDF®) as entry 00-070-1219.

All of the bond distances, angles, and torsion angles fall within the normal ranges indicated by a Mercury Mogul Geometry check (Macrae *et al.*, 2020). The crystal structure consists of layers of folded azelastine cations parallel to the *ac*-plane. The chloride anions reside between these layers. As expected, there is a strong discrete N5–H28···Cl53 between the cation and the anion (N–H = 0.958 Å, H···Cl = 2.081 Å, N···Cl = 2.993 Å, N–H···Cl = 158.3°). A number of relatively short C–H···Cl distances suggest the presence of additional significant intermolecular interactions.

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DEPOSITED DATA

The Crystallographic Information Framework (CIF) file containing the results of the Rietveld refinement (including the raw data) was deposited with the ICDD. The data can be requested at info@icdd.com.

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