

## **Solid state characteristics of bedaquiline benzoate**

**Mercy Amaka Okezie**

Purdue University, Indiana

**B**edaquiline was approved for the treatment of multi-drug-resistant tuberculosis in 2012. Understanding the solid-state properties of the benzoate salt opens the potential for manufacturing it as a new molecular entity. There have been some communications on crystal salts of bedaquiline; this work provides more insight into the characterization of the benzoate salt. The salt was formed from a 1:1 ratio of 30mg (0.054 millimoles) bedaquiline and the millimole equivalents, 6.6mg of benzoic acid. Single crystal structures were determined at 150 K using Bruker Quest X-ray diffractometers using either Mo K $\alpha$  ( $\lambda = 0.71073 \text{ \AA}$ ) or Cu K $\alpha$  radiation ( $\lambda = 1.54178 \text{ \AA}$ ). The sample was analyzed by PXRD, and the structure was confirmed by Rietveld analysis against the single crystal. Sorption potentials for the salt were determined at 75% and 0% relative humidity (RH), while accelerated stability was conducted at 40°C and 75% RH. Thermal analysis was conducted using melting point, DSC, and TGA. The water content of the hydrate benzoate was by KF titrations. Bedaquiline benzoate occurs as a monocation protonated selectively at the dimethyl amine substituent., the less basic quinoline N atoms remain unprotonated. It

occurs as either a 1.17 hydrate or a monohydrate acetonitrile solvate. The chemical formula was C<sub>32</sub> H<sub>32</sub> Br N<sub>2</sub> O<sub>2</sub>, C<sub>7</sub> H<sub>5</sub> O<sub>2</sub>, 1.166(H<sub>2</sub> O), Molecular weight 698.7g. Rietveld's analysis confirmed the benzoate salts. The DSC thermograph value was comparable to the melting point determination. KF determination shows it contained 3.33% water, comparable to the TGA results, loss of ~3.1%. The salt was stable and nonhygroscopic for 3 months.

### **Biography**

Mercy Amaka Okezie is a postdoc research associate in the Industrial and Physical Pharmacy department at Purdue University, West Lafayette, IN. Simultaneously, she works as a regulatory officer with Nigeria's foods and drugs regulatory authority, NAFDAC. A fellow of the West African Post Graduate College of Pharmacists (2010), Dr. Okezie also has an MS degree in BIRS from Purdue (2016). Currently, she is working on a laboratory-based assessment of the quality of some products that have a high history of recalls in a US FDA Project (Assessment Tools for Surveillance and Monitoring of Real-World Data Systems and Processes to Ensure Product Quality). Also, developing solid nanoparticles for the bedaquiline salts to further improve their solubility.

[mokezie@purdue.edu](mailto:mokezie@purdue.edu)

**Received Date:** July 22, 2022; **Accepted Date:** July 24, 2022; **Published Date:** October 31, 2022