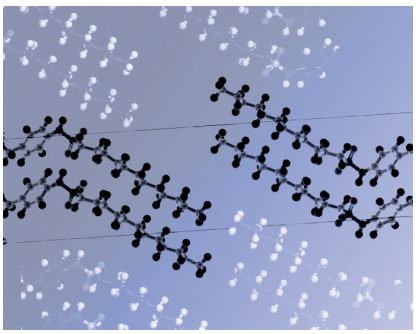
CASE STUDY

KODAK, MATERIALS STUDIO FROM ACCELRYS DRIVES INNOVATION BY PROVIDING CRITICAL DATA SUCH AS THE STRUCTURE OF INGREDIENTS AT KODAK.



> "Determining the crystal structure allowed us to better understand properties of this chemical. [This] was needed for the development of modified materials with enhanced chemical and physical performance. "
>
> **Dr Manju Rajeswaran**
> **Kodak**

Key Product

- MS Modeling Reflex Plus

Industry sectors

- Fine Chemicals
- Specialty Chemicals

Organization

- Kodak

Scientists at Kodak and Accelrys have successfully solved the crystal structure of N-(p-tolyl)-dodecylsulfonamide directly from powder diffraction data using Materials Studio's Reflex Plus.

The determination of crystal structure is often the first step in understanding materials properties, which can lead to the design of improved materials.

Single-crystal X-ray diffraction continues to be the preferred method for solving crystal structures. However, this approach requires single crystals of sufficient size and quality. These crystals can be difficult to produce on demand, and it is often the case that crystallization experiments yield polycrystalline samples that must be analyzed using powder diffraction techniques.

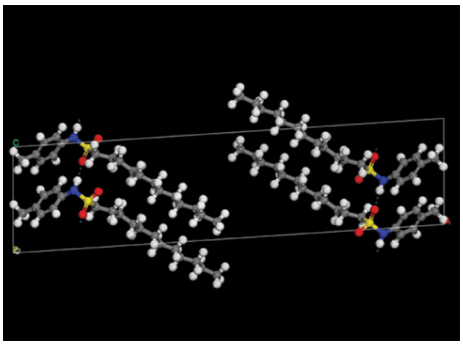


Fig. 1 *Structure of N-(p-tolyl)-dodecylsulfonamide solved with Reflex Plus.*

Reporting in J. Am. Chem. Soc.,[1] scientists at Kodak and Accelrys demonstrated the success of an integrated approach to crystal structure determination and verification from powder diffraction data, after failing to obtain a single crystal

for X-ray diffraction, using a combination of computational chemistry including Materials Studio's PowderSolve technology Reflex Plus and experimental solid-state NMR chemical shifts to solve the crystal structure of the organic stabilizer N-(p-tolyl)-dodecylsulfonamide (Fig. 1).

Elucidation of the crystal structure from powder XRD data followed a systematic
step approach:

- Unit cell indexing using DICVOL91
- Space group determination based on systematic absences and density considerations
- Pawley refinement
- Simulated annealing using PowderSolve (Reflex Plus)
- Checking and correcting any close atom contacts using CVFF
- Final structure refinement using the Rietveld method (Fig. 2).

Validation of the obtained structure was carried out by verifying that the solution was close to a minimum in energy. A full geometry optimization was performed with CVFF and the optimized and predicted structures were compared. In addition, solid-state NMR chemical shifts were compared to the calculated chemical shifts of the isolated molecule and of a dimer in the same conformation found in the crystal.

Dr Manju Rajeswaran, Kodak, said, "Determining the crystal structure allowed us to better understand properties of this chemical. This understanding was needed for the development of modified materials with enhanced chemical and physical performance."

To learn more about Materials Studio by Accelrys, go to **accelrys.com/materials-studio**

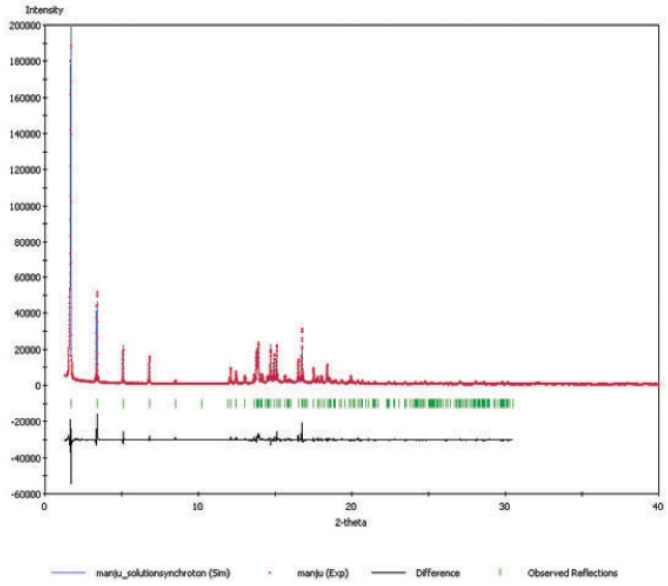


fig. 2 *Comparison of diffraction pattern calculated from the structure solution after Rietveld refinement (blue) overlaid with the experimental pattern (red). The difference plot (black) is plotted below the overlaid patterns.*

REFERENCE

1. Manju Rajeswaran, Thomas N. Blanton, Nicholas Zumbulyadis, David J. Giesen, Carlota Conesa-Moratilla, Scott T. Misture, Peter W. Stephens, and Ashfia Huq, J. Am. Chem. Soc., 2002, **124**, 14450-14459