NON-TRADITIONAL POWDER CRYSTALLOGRAPHY IN THE PETROCHEMICAL INDUSTRY

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Problems encountered in the normal course of business frequently require generating new crystallographic information, and using all of the tools at the analyst's disposal, including those at the state of the art. Several problems will be discussed, with emphasis on the tools and techniques required to solve them, as well as the results. The theme is "what do you do when you need structural details and you have only powder data?".

When a steel pilot plant reactor filled with a supported triflic acid catalyst was unloaded, the reactor walls corroded rapidly. The engineer wanted to know if iron triflates were present. No iron triflates are in the PDF or CSD. The major component was not matched by any phase in the PDF, and could be separated from the normal rust phases by recrystallizing from ethanol. The pattern could be indexed, and the NIST CDIF yielded a match to a vanadium triflate. A Rietveld refinement confirmed that the major phase was hexaaquairon(II) trifluoromethanesulfonate.

The crystal structure of diammonium isophthalate (a starting material for the preparation of models for compounds isolated from commercial deposits) was solved by applying Monte Carlo simulated annealing techniques to synchrotron powder data. It crystallizes in *C2/c* with a = 12.55643(4), b = 6.76443(3), c = 11.09798(6) Å, $\hat{a}$ = 110.2213(3)°, V = 884.531(7) Å^3, and Z = 4. Quantum chemical calculations on this compound, diammonium terephthalate (previously solved using powder data), and diammonium isophthalate (single crystal structure in the literature) yield insight into the bonding and relative energetics in these compounds.

^{129}Xe NMR is often used to study diffusion in zeolites. A sample of LZ-Y52 (commercial Na-FAU) was dehydrated carefully, filled with Xe, and cooled to 20K. From the synchrotron powder data, two low-occupancy Xe sites could be located. Such information assists in the interpretation of the NMR spectra. The presence of extra-framework cations can profoundly affect the catalytic activity of a zeolite, and their positions are not necessarily the same at reaction conditions as at room temperature. The cation locations in a ZnNa-FAU (a diamagnetic analog of catalytically-interesting first-row transition metals) were determined by a resonant scattering study at 300°C.