

3. METHODOLOGY

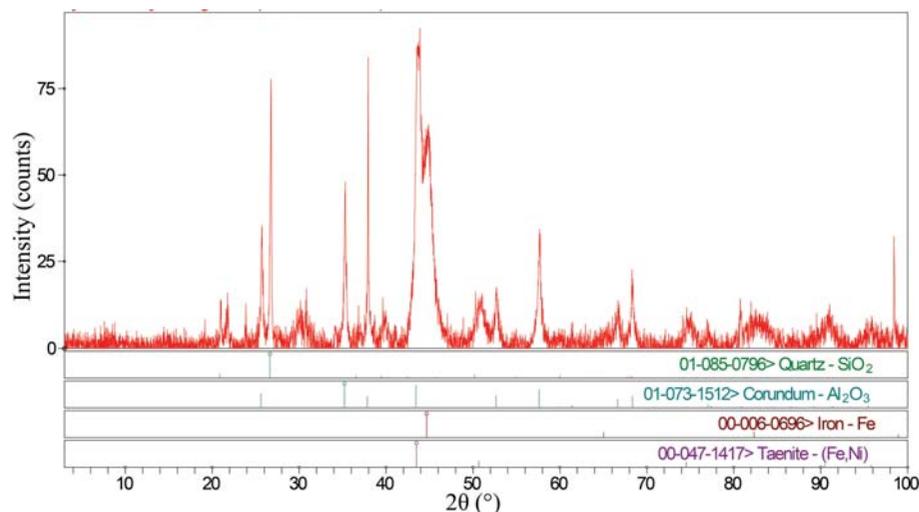


Figure 3.7.7

The four phases identified in a valve deposit from an aircraft engine by automated search/match methods and guessing based on the appearance of the sample. The pattern has had the background and $K\alpha_2$ peaks removed.

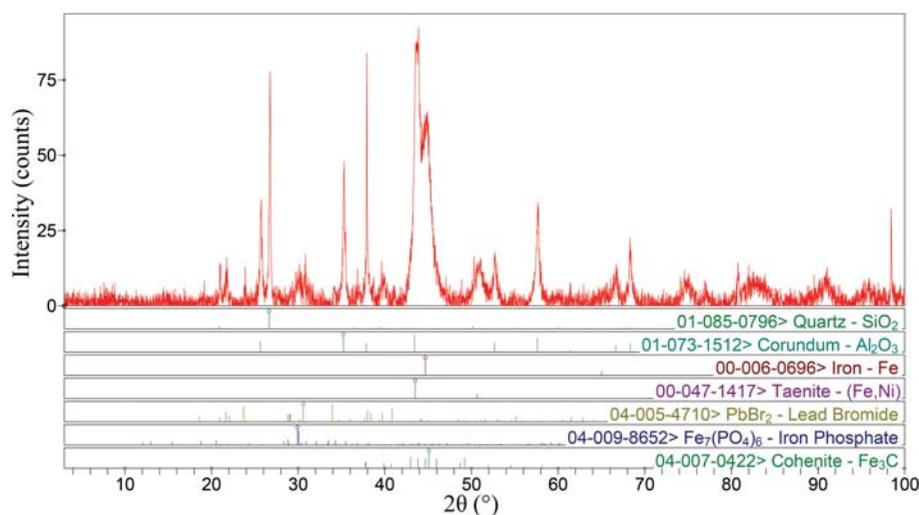


Figure 3.7.8

The seven phases identified in the valve deposit from an aircraft engine.

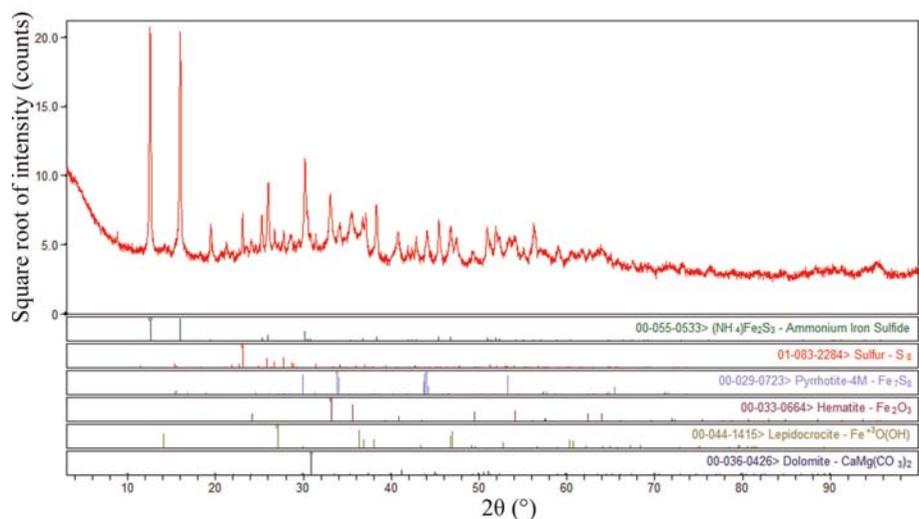


Figure 3.7.9

The phases identified in a deposit from a refinery isocracker. At the time, the $(\text{NH}_4)\text{Fe}_2\text{S}_3$ was a new phase, identified by analogy to KFe_2S_3 , rasvumite.

starting at 3° , and another peak was observed at $d = 24.80 \text{ \AA}$ ($3.56^\circ 2\theta$).

A search of the PDF-4/Organics 2013 for phases having two such peaks among their longest (lowest-angle) peaks yielded entry 00-005-0010 for calcium stearate at the top of the hit list, as well as two lead stearates. We can safely assume that lead stearate is not present in a pharmaceutical. Calcium stearate, however, has its strongest peak at 1.76° , so another pattern was measured starting at $1.5^\circ 2\theta$. This peak is indeed present (Fig. 3.7.11).

The primary literature suggests that the compound in PDF entry 00-005-0010 is really calcium stearate monohydrate, and that its structure (like those of many other stearate salts) has not yet been determined. The CSD entry for amoxicillin trihydrate (AMOXCT10; Boles *et al.*, 1978) contained some incorrect H-atom positions and was missing an H atom, so these were corrected before a Rietveld refinement was carried out.

3.7.2.4.6. Pseudoephedrine

As P. W. Stephens was measuring the powder pattern of a commercial pseudoephedrine-based decongestant on beamline X16C at the National Synchrotron Light Source at Brookhaven National Laboratory, he noted that extra peaks were present. The lowest-angle peak was at a d -spacing of 12.73 \AA , and other peaks occurred at 5.74 , 4.62 (strongest) and 4.407 \AA . A search in the PDF-4/Organics for compounds having the string 'ephed' in the name, a long line at $12.73 \pm 0.05 \text{ \AA}$ and a strong line at $4.62 \pm 0.02 \text{ \AA}$ yielded the single hit 00-041-1946, pseudoephedrine hydrochloride, a reasonable impurity in pseudoephedrine.

3.7.2.4.7. Commercial multivitamin: Centrum A to Zn

Commercial multivitamins are challenging phase-identification problems because they contain small concentrations of many different components. The application of a commercial search/match program to a pattern of Centrum A to Zn collected on beamline ID-32 at the Advanced Photon Source at Argonne National Laboratory using a wavelength of 0.495850 \AA (files centrum.gas and id320304.prm) easily identified brushite, $\text{CaHPO}_4(\text{H}_2\text{O})_2$, and sylvite, KCl (Fig. 3.7.12).

To identify additional phases, 64 peaks with $d > 1.91 \text{ \AA}$ were picked from the plot and entered into *Sieve+* in the PDF-4/Organics 2013 database. The PDF-4/Organics database was used to enhance the success in identifying organic compounds, and the relatively short d -spacing limit was used to ease the identifi-