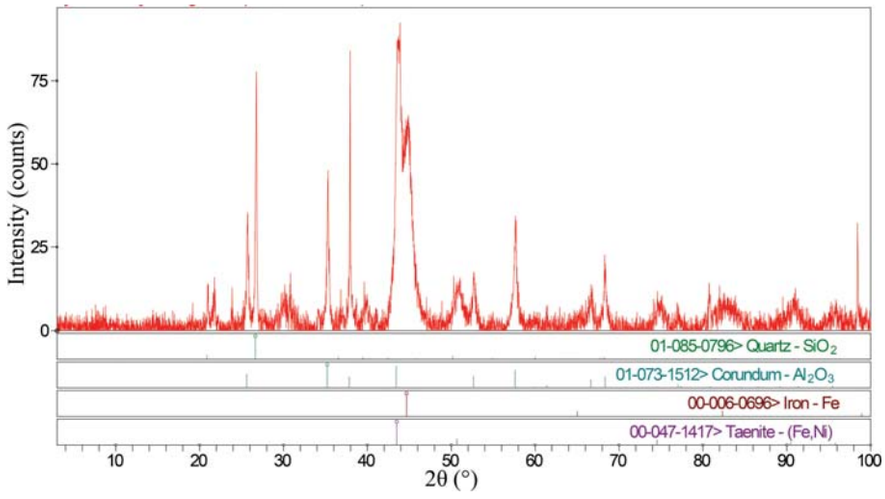

Figure 3.7.7
The four phases identified in a valve deposit from an aircraft engine by automated search/match methods and guessing based on the appearance of the sample. The pattern has had the background and $K\alpha_2$ peaks removed.

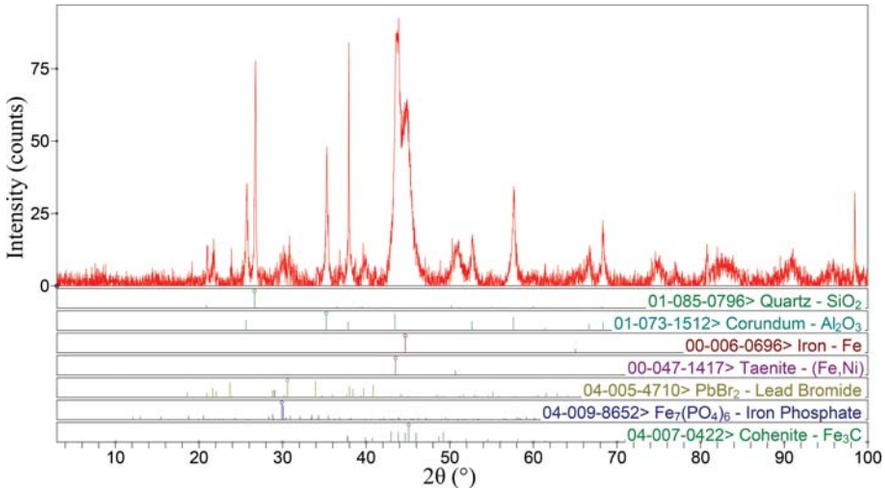

Figure 3.7.8
The seven phases identified in the valve deposit from an aircraft engine.

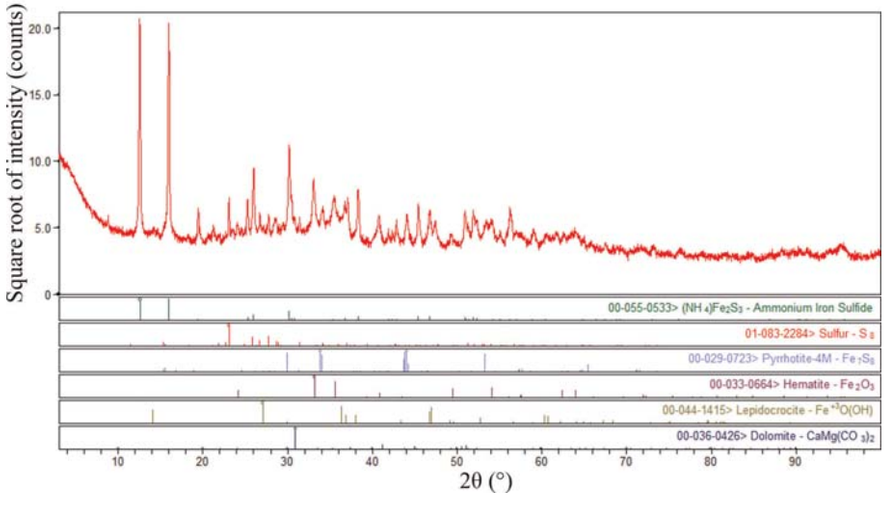

Figure 3.7.9
The phases identified in a deposit from a refinery isocracker. At the time, the $(NH_4)Fe_2S_3$ was a new phase, identified by analogy to KFe_2S_3, rasvumite.

starting at 3°, and another peak was observed at d = 24.80 Å (3.56° 2θ).

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° 2θ. 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 Å, and other peaks occurred at 5.74, 4.62 (strongest) and 4.407 Å. A search in the PDF-4/Organics for compounds having the string 'ephed' in the name, a long line at 12.73 ± 0.05 Å and a strong line at 4.62 ± 0.02 Å 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 Å (files centrum.gsas and id320304.prm) easily identified brushite, $CaHPO_4(H_2O)_2$, and sylvite, KCl (Fig. 3.7.12).

To identify additional phases, 64 peaks with d > 1.91 Å 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-