cation of the simple inorganic compounds which are often present in commercial vitamins.

A Hanawalt search using these peaks easily identified iron fumarate (00-062-1294), szmikite [$MnSO_4(H_2O)$; 00-033-0906], L-ascorbic acid (02-063-2295), monetite ($CaHPO_4$; 01-070-0359) and calcite ($CaCO_3$; 00-005-0586). Note that these hits come from four different data sources; searches based on just one source would not have identified all of these compounds.

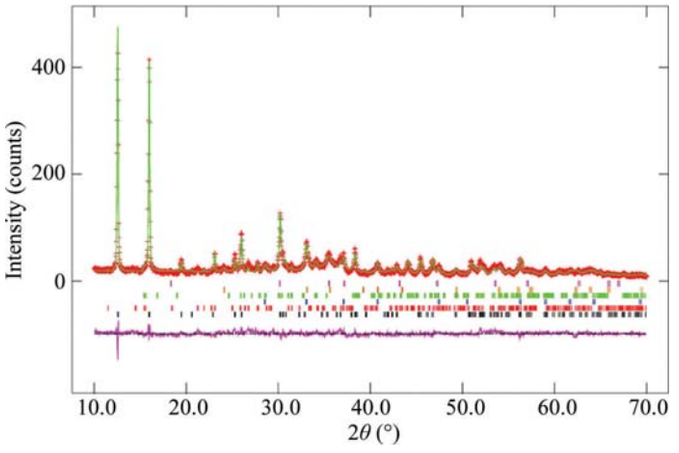

Figure 3.7.10
The final Rietveld plot from refinement of the isocracker deposit.

There were strong high-angle peaks that had not yet been accounted for at *d*-spacings of 2.4762, 2.1068, 1.4900 and 1.4783 Å. These four peaks were entered into a new Hanawalt search, which identified periclase (MgO; 01-071-3631) and zincite (ZnO; 01-075-9742).

Superimposing the peaks for all of these compounds onto the raw data made it clear that there were broad peaks in the pattern at *d*-spacings of approximately 5.8750, 5.3273, 4.3277 and 3.9217 Å. Since the lowest and highest angles of these four were the best defined, separate searches for compounds having each of these peaks as one of their three strongest lines were combined using a Boolean 'and'. Among the hit list was cellulose I_β (00-060-1502), which is a common constituent of pharmaceuticals. The structure model from PDF entry 00-056-1718 was added to the Rietveld refinement as a ninth phase.

One last peak at 5.9915 Å was unaccounted for. A search for pharmaceutical-related compounds with this peak as one of the three strongest included nicotinamide (02-063-5340; niacin or vitamin B_3). Ten phases were thus identified and these account for all of the peaks in the pattern.

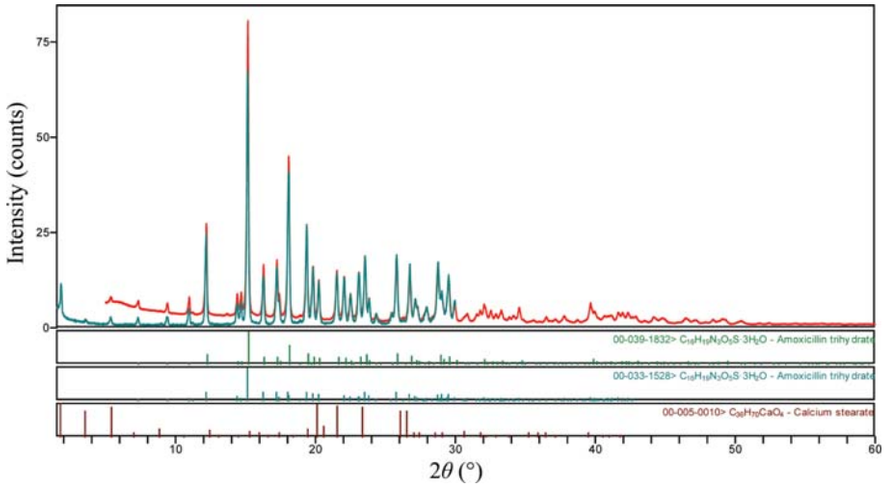

Figure 3.7.11
Phases identified in amoxicillin powder from a commercial capsule.

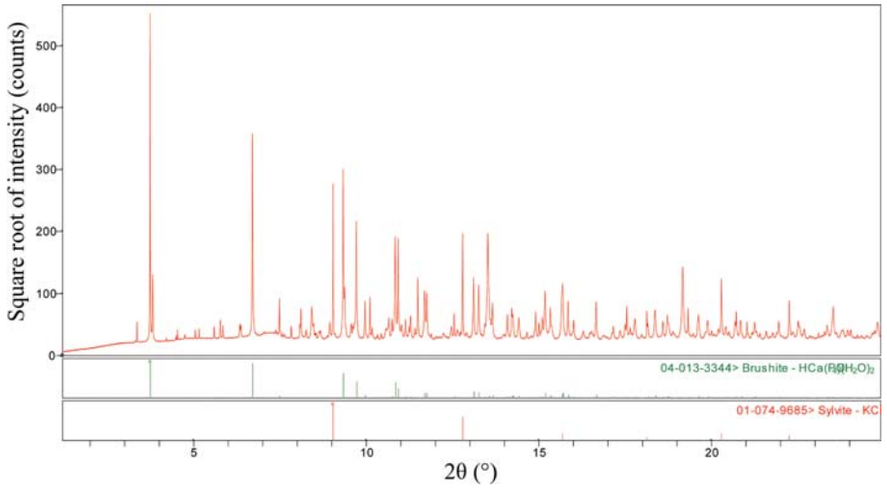

Figure 3.7.12
Phases identified by automated search/match in a Centrum A to Zn multivitamin tablet. Additional phases were identified using the native capabilities of the Powder Diffraction File.

3.7.3. Cambridge Structural Database (CSD)

Some features of the Cambridge Structural Database system (CSD; https://www.ccdc.cam.ac.uk; Groom *et al.*, 2016) are described in Chapter 22.5 of *International Tables for Crystallography* Volume F (Allen *et al.*, 2011). The CSD contains X-ray and neutron diffraction analyses of carbon-containing molecules with up to 1000 atoms (including hydrogens), including organic compounds, compounds of the main-group elements, organometallic compounds and metal complexes. The CSD covers peptides of up to 24 residues; higher polymers are covered by the Protein Data Bank. The CSD also covers mononucleotides, dinucleotides and trinucleotides; higher oligomers are covered by the Nucleic Acid Database (http://ndbserver.rutgers.edu). There is a small overlap between the CSD and the Inorganic Crystal Structure Database in the area of molecular inorganics.

Capabilities particularly useful for structure validation are covered in Chapter 4.9 of this volume. This discussion will not attempt a comprehensive description of the capabilities of the CSD, but will concentrate on features that are particularly relevant to powder diffraction.

The principal interface to the CSD is the program *ConQuest* (Bruno *et al.*, 2002). Its most distinctive feature is the ability to draw molecular structures and fragments and carry out substructure searches. Such searches eliminate the ambiguities that can arise when searching by compound name or other text-based properties. These chemical-connectivity