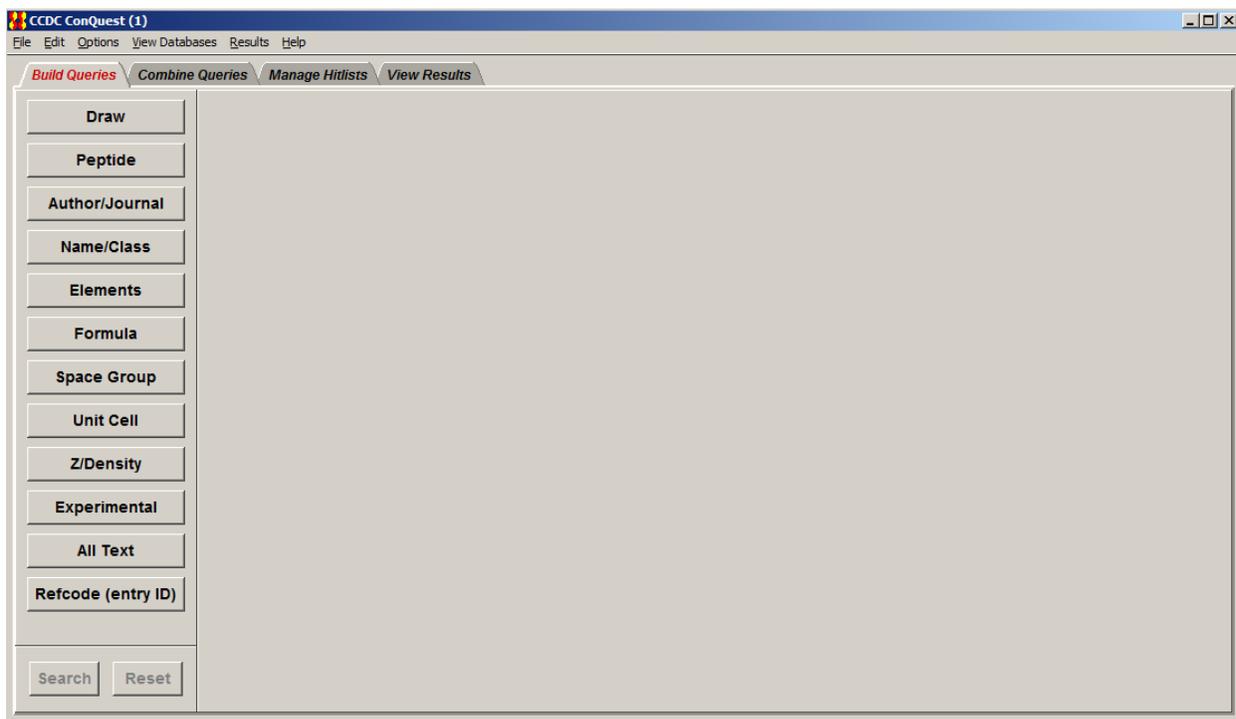


Databases and Twinning

Databases

There are numerous databases that combine crystal structure information. Most of these databases are based on the type of molecule in the database and many contain overlaps. The most useful database for the Department's Chemical Crystallography Laboratory is the Cambridge Structural Database, CSD, prepared by the Cambridge Centre for Crystallographic Data. This database contains all small-molecule organic and organometallic crystal structures ever published (except for about the previous 6 months). Structures of polymeric compounds are usually limited to 10-12 residues. Currently there are over 876,00 structures listed in the database. <http://www.ccdc.cam.ac.uk/>

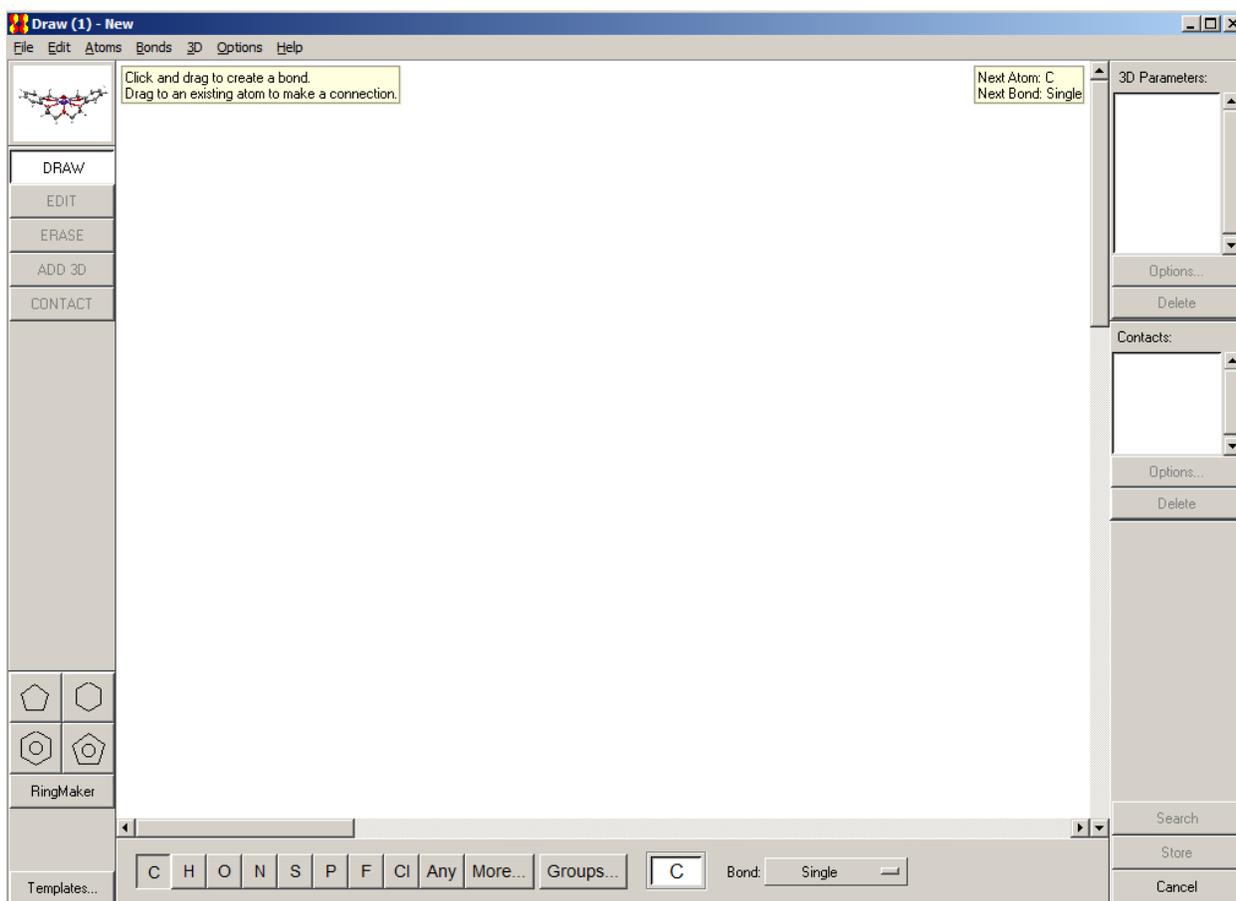
In our Laboratory, the CSD is only available on the computer XRAY5. The main program is opened with the Conquest icon on the desktop. The opening page appears below.



Search questions are called queries. This first page directs the user to the type of query they wish to construct. Most commonly, users want to search the database for some molecule or fragment of molecule. Through the Author/Journal button, you can search for a specific article or related groups of articles. The Name/Class button does queries for these specific items, but requires that the IUPAC name be known or that the class has been identified by either the original authors or the CSD. The Space Group button is good for statistics. The Unit Cell button unfortunately produces 40+ hits for nearly any cell. The Z/Density, Experimental, and All Text buttons are of limited use. Each entry is given a unique "Refcode". If the specific refcode is known, it can be located with the Refcode button. Also note that multiple search questions can

be saved and logically combined using “and”, “or”, or “not” type of logical operators in the Combine Queries menu.

The Draw menu contains a drawing screen with a list of common atom types on the bottom and all possible bonds. At the bottom left are menus to select rings and templates of common groups. Other atoms or groups of atoms can be selected from the “More” menu. When selecting the bond type, remember both a delocalized and an aromatic bond are of order 1.5. Delocalized bonds are not in a ring; aromatic bonds are in a ring. There are two caveats about using this program. When your search yields no hits and the molecule can be in another conformation, e.g., as keto, enol conformers, then try searching for the other conformation. Second, when a metal is bonded to the pi system of an aromatic ring, draw only one bond between the metal and the ring.



When searching for an author’s name, the search should only contain the initials of the first and middle names and the last name, no capitals, and no spaces. When I search for my name I need to set up two queries, one for “d.powell” and another for “d.r.powell”.

In addition to the CSD database, the *free* Crystallography Open Database is available at <http://www.crystallography.net/cod/> and the Pearson Database of inorganic compounds is also on the XRAY5 computer, details are at: <http://crystalimpact.com/pcd/Default.htm>.

Twinning

Crystalline samples that are not twinned by merohedry are very difficult to index as a single crystal. However, indexing these samples can usually be done by the program `cell_now` as described in the Collect & Reduce Data document. Be sure to import the file containing the multiple cells before integration. Add the optimization steps slowly during integration to keep the program on the different lattices. The data must be processed through TWINABS before it can be used for space group determination or structure solution.

Samples that are twinned by merohedry are identified near the end of refinement when the R does not drop to the values expected for the data quality. The twin law is usually some symmetry operation of the Laue group but not of the space group. When the twin law(s) is/are identified, it is entered by the TWIN command in the *.ins file. Also, a scale factor (twin ratio) needs to be entered on the BASF instruction. For n twin laws, there should be $n-1$ scale factors on BASF.