Chemistry 832, Spring 2000, Dr. Hunter
Laboratory Assignment #L1, Introduction to SHELXTL

Getting Used to the Basics of SHELXTL to Solve Structures. On the computer, there are three selected X-ray data sets (i.e. Fun1 {$PhCH_2$-NH-CHPh-P(O)(OH)(OEt)}, Fun2 {(η^6-1,4-$C_6H_4(NH_2)(NMe_2)$)$Cr(CO)_3$}, and Fun3 {$(Ph_2PCH_2CH_2PPh_2)Pd(S_2C_2(CN)_2)$}) chosen based on the variety of their structures and the fact that their structure solutions proceed in a relatively strait forward fashion. For each of these examples, copy its data from the directory c:\xraydata\students\fun123 to the directory c:\xraydata\students\yourname, rename them as yourname.hkl, yourname.ins, and yourname.int, and then:

(1) Run the program XS on your data to find a trial solution from which you can guess the first atom(s).

(2) Run the program XP to display this trial solution and assign any atoms who's identities are unambiguous.

(3) Run the program XL to refine the data and obtain a better solution for the crystal structure.

(4) Repeat steps 2 and 3 until you have completely assigned and refined the structure. [Note: after you have assigned and refined all non-hydrogen atoms convert the refinement model to anisotropic (ANIS). Then assign any hydrogen atoms and refine them. Finally, do an extinction correction (EXTI), add the ACTA(BOND $H) command, and refine the data until the solutions stops improving (these last three commands are added to the *.ins file using the DOS edit command).]

(5) Print at least three different views of your molecule using XP and prepare tables of the structural data by XCIF and **hand in** this material. Save your .ins files on disk for the three structures.

Some common XP commands are:

fmol	info	proj	
pick	kill	bang	
file	exit		
pers	telp (b)	rast	rast/c