Practical : structure determination of single crystals

1- Determine the structure of a Rh complex using direct methods.

2- Solve the crystal structure of an organic compound using the charge-flipping method.

Data :

1- X-ray diffraction measurements of a single crystal of (η^5-C_5Me_5)Rh{(R)-Prophos}(methacrolein)(SbF_6)$_2$ (possibly with a chlorinated solvent residue) recorded on the 4-circle diffractometer at the CRISTAL beamline, λ = 0.56356 Å (22 keV).
2- Laboratory measurements on the compound DL2_PO_4, λ = 0.71073 Å (17 keV = Kα[Mo]).

Intensities integrated by the CrysAlis Pro software (Rigaku Oxford Diffraction), corrected for Lorentz and polarisation factors and absorption.

Protocl :

1) Indexation : search for the lattice cell and the orientation matrix
2) Integration + scaling + absorption corrections if needed
3) Sapce group determination (centrosymmetry, extinction rules)
   File.ins : instruction file
   File.hkl : experimental dataset (h k l I sig…)

To save time, these first 3 steps have been carried out beforehand (they depend on the software used to process the measured images).

4) Solving the crystal structure using the Olex2 software suite

Prerequisites :
(approximative) chemical composition of the lattice cell (chemical formula)

Edit the file.ins file (created by CrysAlis Pro) :

Corect/check the following lines :
CELL : 1st parameter = wavelength
ZERR : 1st parameter = Bravais lattice mode (1 for P, 2 for I …)
SFAC (Scattering FACtors) : give the elements (atoms) in the unit cell
UNIT : give a number of atoms/éléments compatible with the symmetry
e.g. C6H6, Z=8, Pbca. 48 C and 48 H in the unit cell

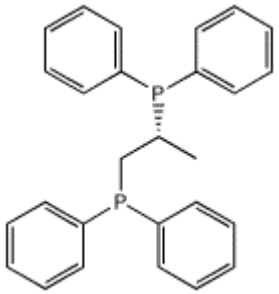
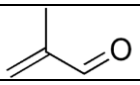
ZERR 8 … ????
….
SFAC C H
Rq : respect the order of the éléments : C, H O first, and then the other elements
UNIT 48 48

TREFF 50 : résolution par méthode directe, 50 itérations
HKL 4 : data format h k l I sig(I) (cos_in %a*, cos_out%a*, cos_in%b*…etc)[1]

END : end of the instruction files

[1] direction cosines only appear if absorption corrections have not been made when reducing the data

Supplementary information :

(R)-Prophos	
methacroléine	

Bonds	**Average distance (Å)**
Rh—P	[2.237 – 2.332]
Rh—O	[2.041 – 2.175]
Sb—F	[1.750 – 2.000]
P—C	[1.790 – 1.857]
Csp^3—Csp^3	[1.514 – 1.580]
Csp^2—Csp^2 (aromatique)	[1.381 – 1.436]
Csp^2—Csp^2 (aliphatique)	[1.300 – 1.330]
Csp^3—Csp^2	[1.475 – 1.522]
Csp^3—O	[1.414 – 1.450]
C=O	[1.192 – 1.253]
Csp^3—N	[1.451 – 1.550]
Csp^3—H	[1.061 – 1.099]
Csp^2—H (aromatique)	[1.079 – 1.083]
N—H	[1.010 – 1.036]
O—H	[0.969 – 1.017]

General Positions of the Group 92 ($P4_12_12$)

1	x,y,z	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$	1
2	-x,-y,z+1/2	$\begin{pmatrix} -1 & 0 & 0 & 0 \\ 0 & -1 & 0 & 0 \\ 0 & 0 & 1 & 1/2 \end{pmatrix}$	2 (0,0,1/2) 0,0,z
3	-y+1/2,x+1/2,z+1/4	$\begin{pmatrix} 0 & -1 & 0 & 1/2 \\ 1 & 0 & 0 & 1/2 \\ 0 & 0 & 1 & 1/4 \end{pmatrix}$	4^+ (0,0,1/4) 0,1/2,z
4	y+1/2,-x+1/2,z+3/4	$\begin{pmatrix} 0 & 1 & 0 & 1/2 \\ -1 & 0 & 0 & 1/2 \\ 0 & 0 & 1 & 3/4 \end{pmatrix}$	4^- (0,0,3/4) 1/2,0,z
5	-x+1/2,y+1/2,-z+1/4	$\begin{pmatrix} -1 & 0 & 0 & 1/2 \\ 0 & 1 & 0 & 1/2 \\ 0 & 0 & -1 & 1/4 \end{pmatrix}$	2 (0,1/2,0) 1/4,y,1/8
6	x+1/2,-y+1/2,-z+3/4	$\begin{pmatrix} 1 & 0 & 0 & 1/2 \\ 0 & -1 & 0 & 1/2 \\ 0 & 0 & -1 & 3/4 \end{pmatrix}$	2 (1/2,0,0) x,1/4,3/8
7	y,x,-z	$\begin{pmatrix} 0 & 1 & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 0 & 0 & -1 & 0 \end{pmatrix}$	2 x,x,0
8	-y,-x,-z+1/2	$\begin{pmatrix} 0 & -1 & 0 & 0 \\ -1 & 0 & 0 & 0 \\ 0 & 0 & -1 & 1/2 \end{pmatrix}$	2 x,-x,1/4

Reflection conditions for the space group 92 ($P4_12_12$)

Mult.	Letter	Site Sym.	WP Representative	Reflection conditions
				General:
8	b	1	(x,y,z)	00l: l=4n h00: h=2n
				Special: as above, plus
4	a	..2	(x,x,0)	0kl: l=2n+1 or 2k+l=4n

Cf. Bilbao Crystallographic Server : http://www.cryst.ehu.es/