Database for X-Ray experiments

Boris Averkiev

SAMPLE INFORMATION FORM
Department of Natural Sciences
New Mexico Highlands University

Synthetic chemist (***name***, address, ***email***, phone):
Raul Castaneda and Yulia Getmanenko

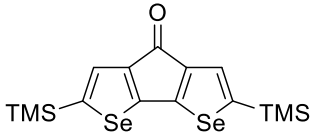
Project PI (***name***, address, ***email***, phone):
Tatiana Timofeeva

Sample ID: YAG-XIV-186-a

Experiment type: routine "yes"

Sample gross formula (if known):
$C_{15}H_{20}OSe_2Si_2$
Solvent:
Hexanes

Sample structural formula (if any):



Sample information (write what you can):
Melting point: N/A Color: very dark needles
Crystal sensitivity: not sensitive Losing solvent: N/A Moisture N/A
NMR: "no"
Elemental Analysis: "no"
Hazard information (if known):
If crystal structure was studied before (if known): "no"
If "yes" give explanation

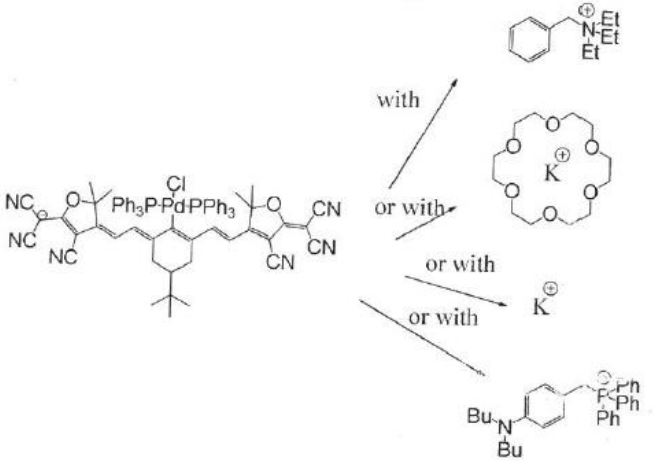
Publication plans (if any): Chemistry Journal

SAMPLE INFORMATION FORM
Department of Natural Sciences
New Mexico Highlands University

Synthetic chemist (***name***, address, ***email***, phone):
Iryna Davydenko; 901 Atlantic Drive, MS&E Bldg., Atlanta, GA 30332;

Project PI (***name***, address, ***email***, phone):
Dr. Seth Marder; 901 Atlantic Drive, MS&E Bldg., Atlanta, GA 30332;

Experiment type: routine "yes" "no" Yes
If "no" give specification
Sample gross formula (if known): anion - $C_{70}H_{60}ClN_6O_2P_2Pd$ and ? Cat^+
Sample structural formula (if any): Pd-TCF

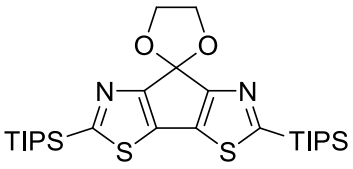


Sample information (write what you can):
Melting point: unknown Color: dark crystals

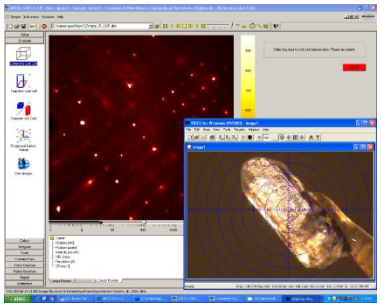
Crystal sensitivity: Air Light Losing solvent Moisture
NMR: "yes" "no" Yes
Elemental Analysis: "yes" "no" Yes
Hazard information (if known): See MSDS
If crystal structure was studied before (if known): "yes" "no" No
If "yes" give explanation
Publication plans (if any): Deposition of CIF Crystallography Journal

Chemistry Journal Other (specify)
Chemistry Journal

X-Ray Analysis

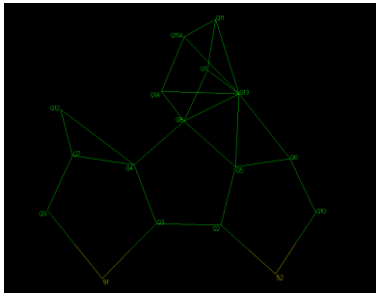


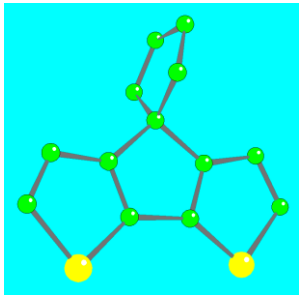
X-ray experiment

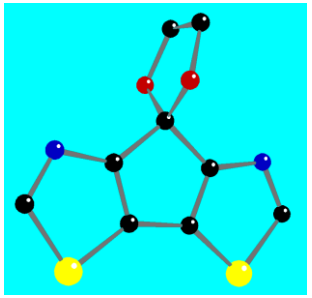


-12	0	0	5.61	0.52
12	0	0	4.79	0.64
12	0	0	5.51	0.43
-14	0	0	0.77	0.11
14	0	0	0.89	0.23
1	-1	0	56.36	4.09
1	1	0	54.18	4.46
-1	1	0	66.80	4.47
3	-1	0	110.67	8.28
3	-1	0	118.20	11.01
3	-1	0	103.04	7.59
-3	-1	0	121.56	7.59
3	1	0	96.66	8.28
5	-1	0	2.44	0.20
5	-1	0	2.42	0.28
5	-1	0	2.63	0.18
-5	-1	0	2.23	0.24
-5	-1	0	2.68	0.18
-5	1	0	2.18	0.23
5	1	0	2.57	0.20

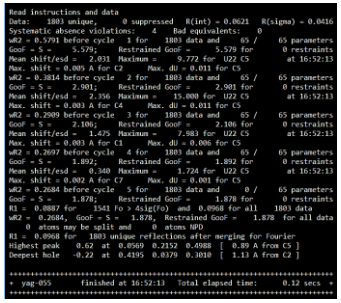
Structure solution







Structure refinement



```
Read instructions and data
Data:    1803 unique,    0 suppressed   R(int) = 0.0621   R(sigma) = 0.0416
Systematic absence violations:    4      Bad equivalents:    0
wR2 = 0.5791 before cycle   1 for    1803 data and      65 /     65 parameters
GooF = S =    5.579;      Restrained GooF =     5.579 for       0 restraints
Mean shift/esd =   2.031  Maximum =    9.772 for  U22 C5          at 16:52:13
Max. shift = 0.005 A for C2       Max. dU = 0.011 for C5
wR2 = 0.3814 before cycle   2 for    1803 data and      65 /     65 parameters
GooF = S =    2.001;      Restrained GooF =     2.001 for       0 restraints
Mean shift/esd =   2.356  Maximum =   15.000 for  U22 C5          at 16:52:13
Max. shift = 0.003 A for C4       Max. dU = 0.011 for C5
wR2 = 0.2509 before cycle   3 for    1803 data and      65 /     65 parameters
GooF = S =    2.106;      Restrained GooF =     2.106 for       0 restraints
Mean shift/esd =   1.475  Maximum =    7.803 for  U22 C5          at 16:52:13
Max. shift = 0.003 A for C1       Max. dU = 0.006 for C5
wR2 = 0.2697 before cycle   4 for    1803 data and      65 /     65 parameters
GooF = S =    1.892;      Restrained GooF =     1.892 for       0 restraints
Mean shift/esd =   0.340  Maximum =    1.724 for  U22 C5          at 16:52:13
Max. shift = 0.002 A for C7       Max. dU = 0.001 for C5
wR2 = 0.2684 before cycle   5 for    1803 data and       0 /     65 parameters
GooF = S =    1.878;      Restrained GooF =     1.878 for       0 restraints
R1 =  0.0887 for    1541 Fo > 4sig(Fo)  and  0.0968 for all    1803 data
wR2 =  0.2684,  GooF = S =    1.878,  Restrained GooF =    1.878  for all data
     0  atoms may be split and     0  atoms NPD
R1 =  0.0968 for    1803 unique reflections after merging for Fourier
Highest peak    0.62  at  0.0569  0.2152  0.4088 [   0.89 A from C5 ]
Deepest hole   -0.22  at  0.4195  0.0379  0.3010 [   1.13 A from C2 ]

+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  yag-055        finished at 16:52:13   Total elapsed time:       0.12 secs  +
+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
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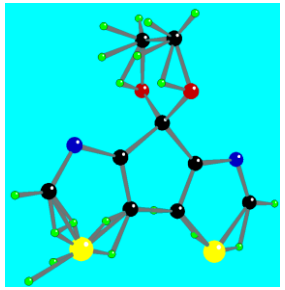
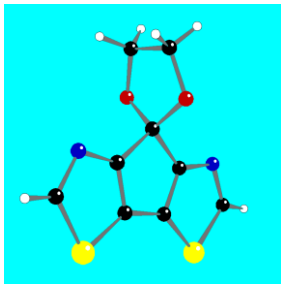
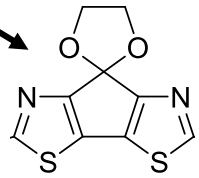




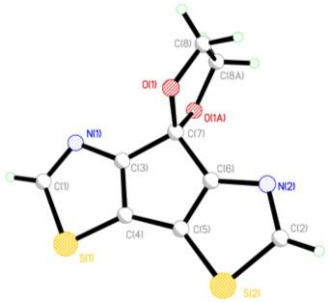


Results

Crystallographic and Refinement Data

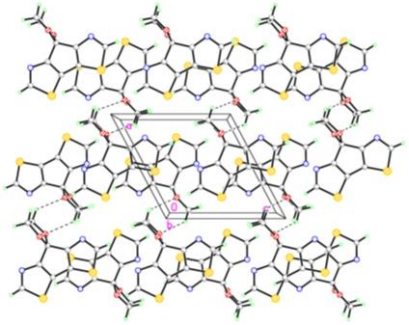
Empirical formula	$C_9H_6N_2O_2S_2$
Formula weight	238.28
Temperature	215(2)
Crystal system	Monoclinic
Space group	$P2_1/m$
a, Å	8.492(7)
b, Å	7.390(6)
c, Å	8.605(7)
α,°	90.0
β, °	118.023(9)
γ, °	90.0
Volume, Å^3	476.7(7)
Molecules per cell, Z	2
Independent mol. per cell, Z'	0.5
Density (calculated), g cm^{-3}	1.660
2θ (max), °	60
Reflections collected	6925
Independent reflections	1478
Parameters	85
Goodness-of-fit on F^2	1.138
R_1 [I>2sigma(I)]	0.0632
wR_2 indices (all data)	0.1855

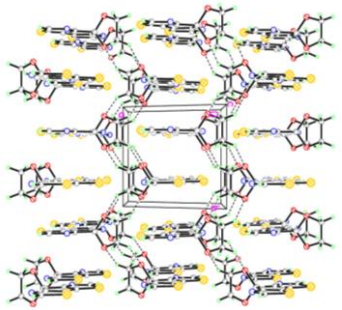
Molecular Structure and Atomic Coordinates



Atom	X	Y	Z
S1	0.5472	0.2500	0.8932
S2	0.8028	0.2500	1.4385
N1	0.2186	0.2500	0.8296
N2	0.5269	0.2500	1.4949
O1	0.1797	0.0958	1.1570
C1	0.3190	0.2500	0.7538
C2	0.7023	0.2500	1.5750

Crystal Packing





Geometries

S1-C1, S2-C2	**1.734(6), 1.746(5)**
S1-C4, S2-C5	1.701(4), 1.706(5)
C1-N1, C2-N2	1.294(7), 1.315(7)
C3-C4, C5-C6	1.370(6), 1.372(6)
N1-C3, N2-C6	1.366(6), 1.370(6)
C4-C5	1.462(6)

Entities

Synthesis

Date
University
Chemist
E-mail
Phone
Formula
Synthesys
Diagram
Hazard Information
Sensitivity
NMR
Elemental analysis
Journal

X-Ray

Crystallographer
X-ray Date
Temperature
Crystal Color
Crystal Shape

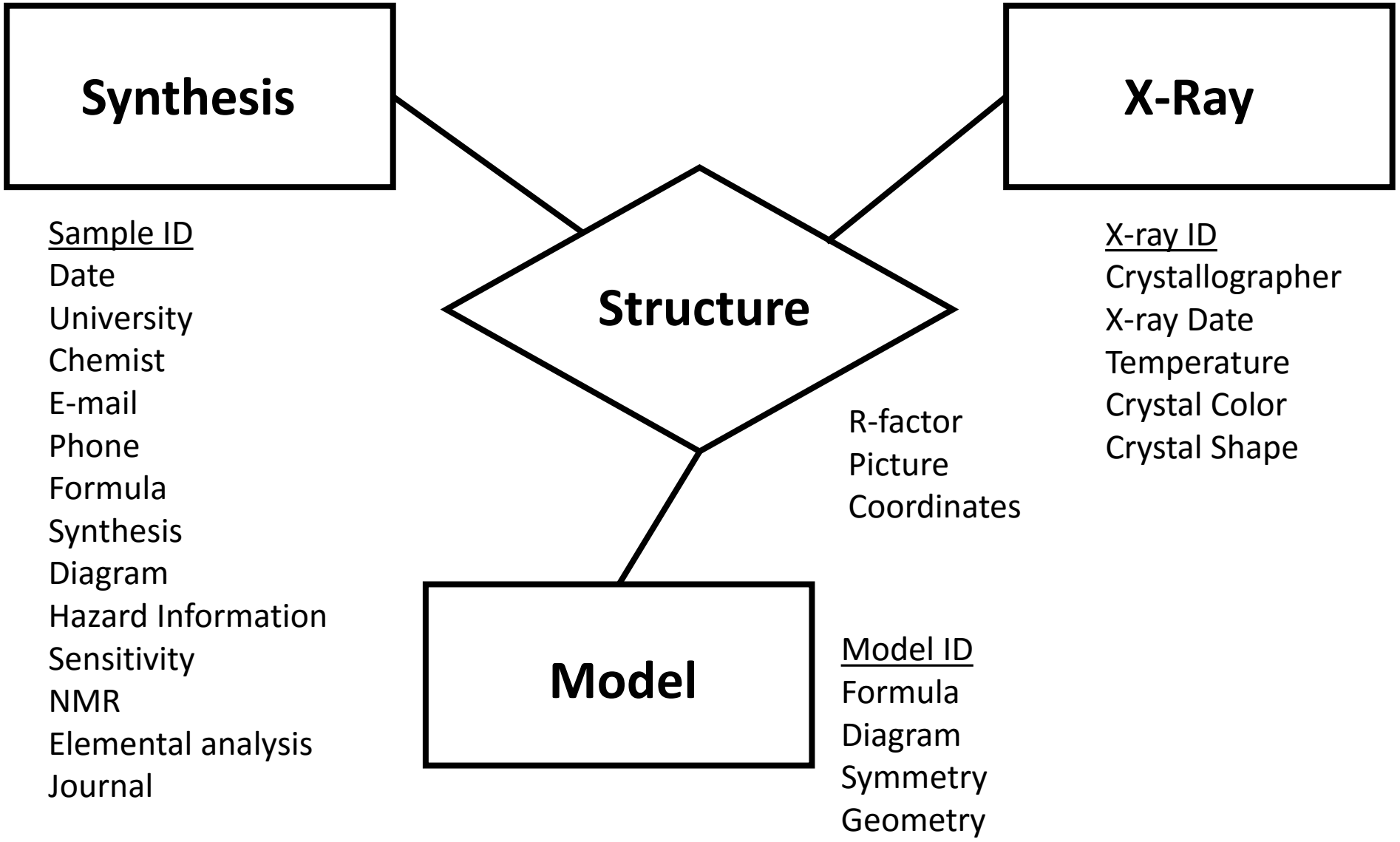
Model

Formula
Diagram
Symmetry
Geometry

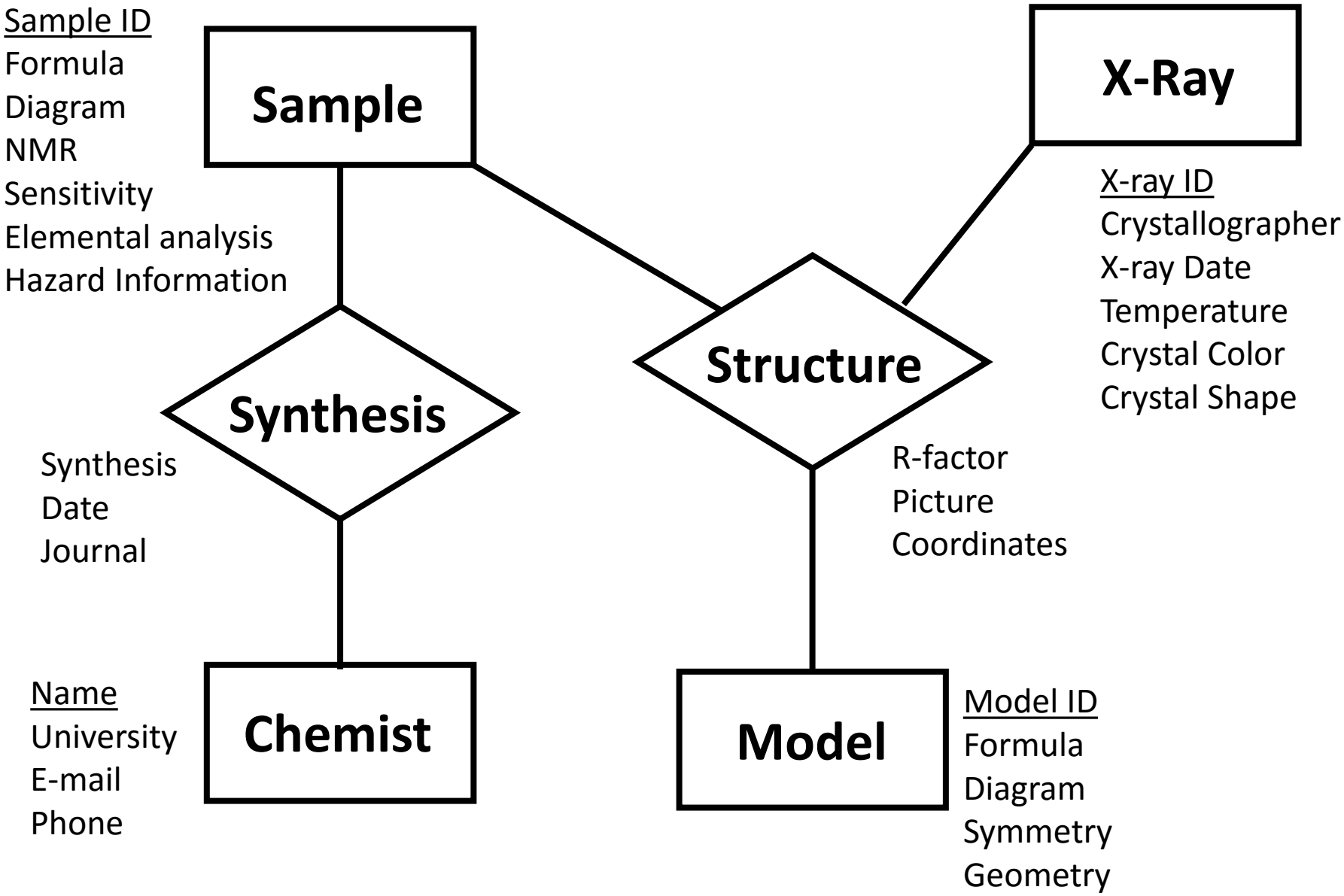
Structure

R-factor
Picture
Coordinates

E-R Diagram with a Ternary Relationship



E-R Diagram


Sample ID
Formula
Diagram
NMR
Sensitivity
Elemental analysis
Hazard Information
Sample
X-Ray
X-ray ID
Crystallographer
X-ray Date
Temperature
Crystal Color
Crystal Shape
Synthesis
Structure
Synthesis
Date
Journal
R-factor
Picture
Coordinates
Name
University
E-mail
Phone
Chemist
Model
Model ID
Formula
Diagram
Symmetry
Geometry