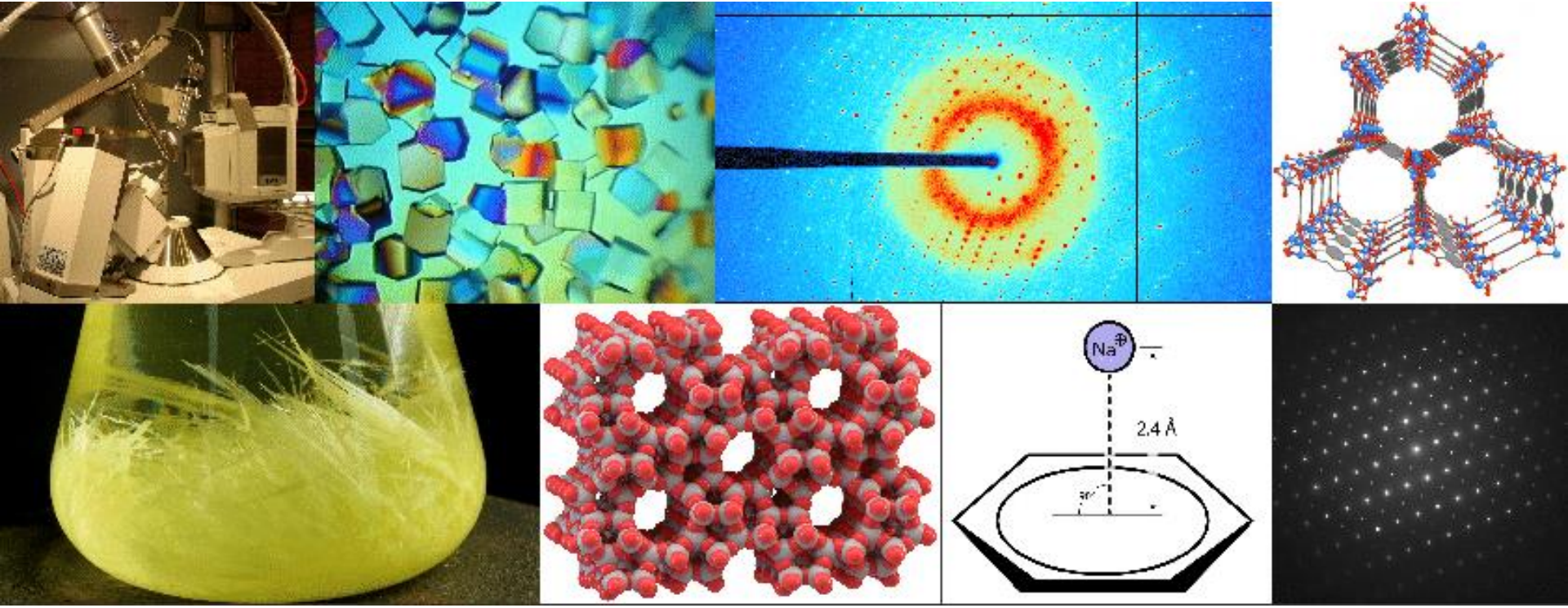
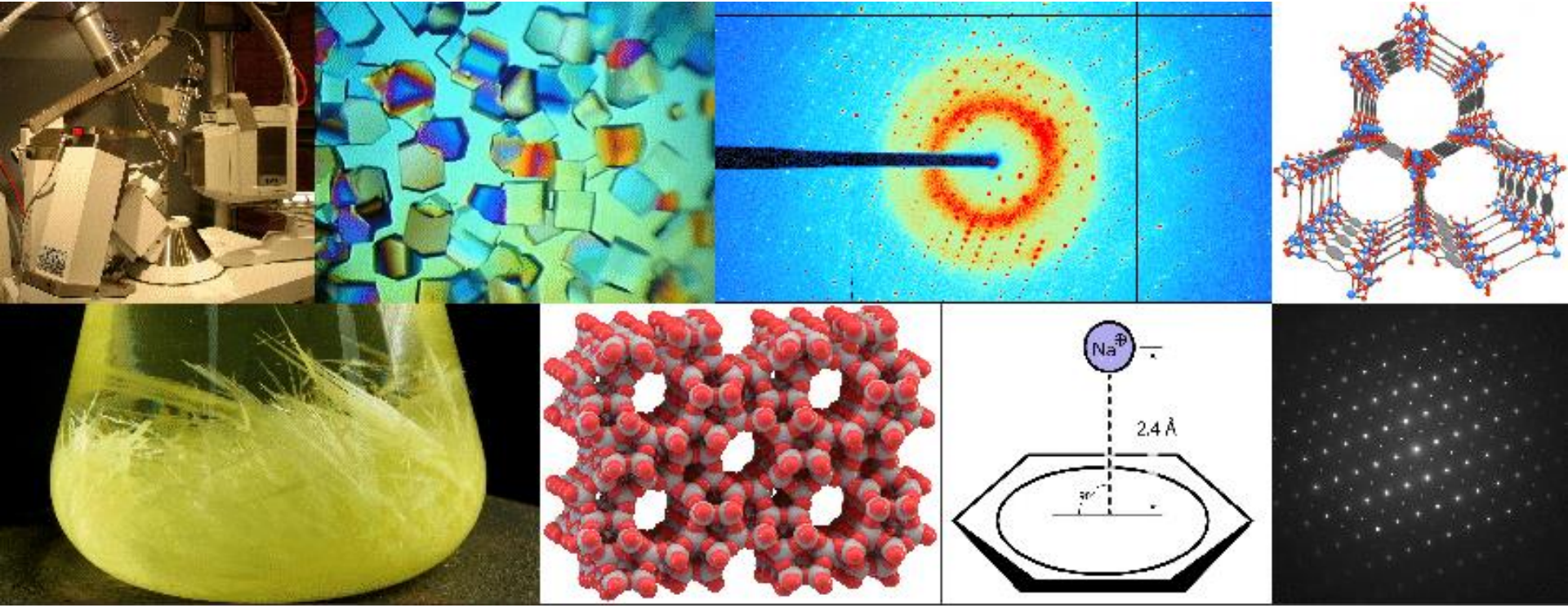


Programa de Actualización| ANUAL| 2018

CARACTERIZACIÓN ESTRUCTURAL Y ANÁLISIS DE PROPIEDADES DE SÓLIDOS CRISTALINOS: POLIMORFOS, SOLVATOS, COCRISTALES Y SALES

Florencia Di Salvo | Sebastián Suárez

Departamento de Química Inorgánica, Analítica y Química Física, FCEN, UBA



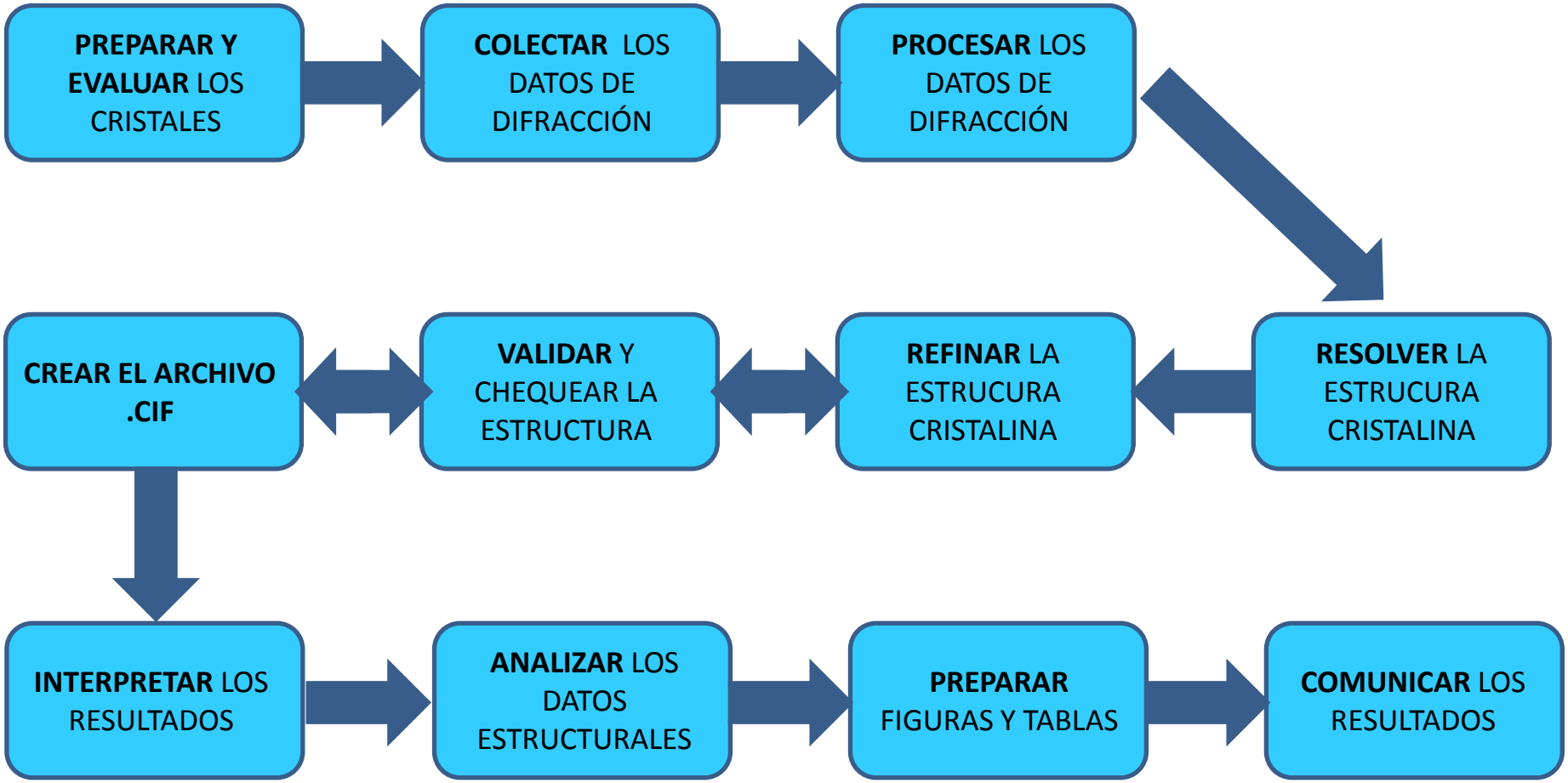
Programa de Actualización| Agosto-Septiembre | 2018

PREPARACION Y PUBLICACION DE ARCHIVO CIF

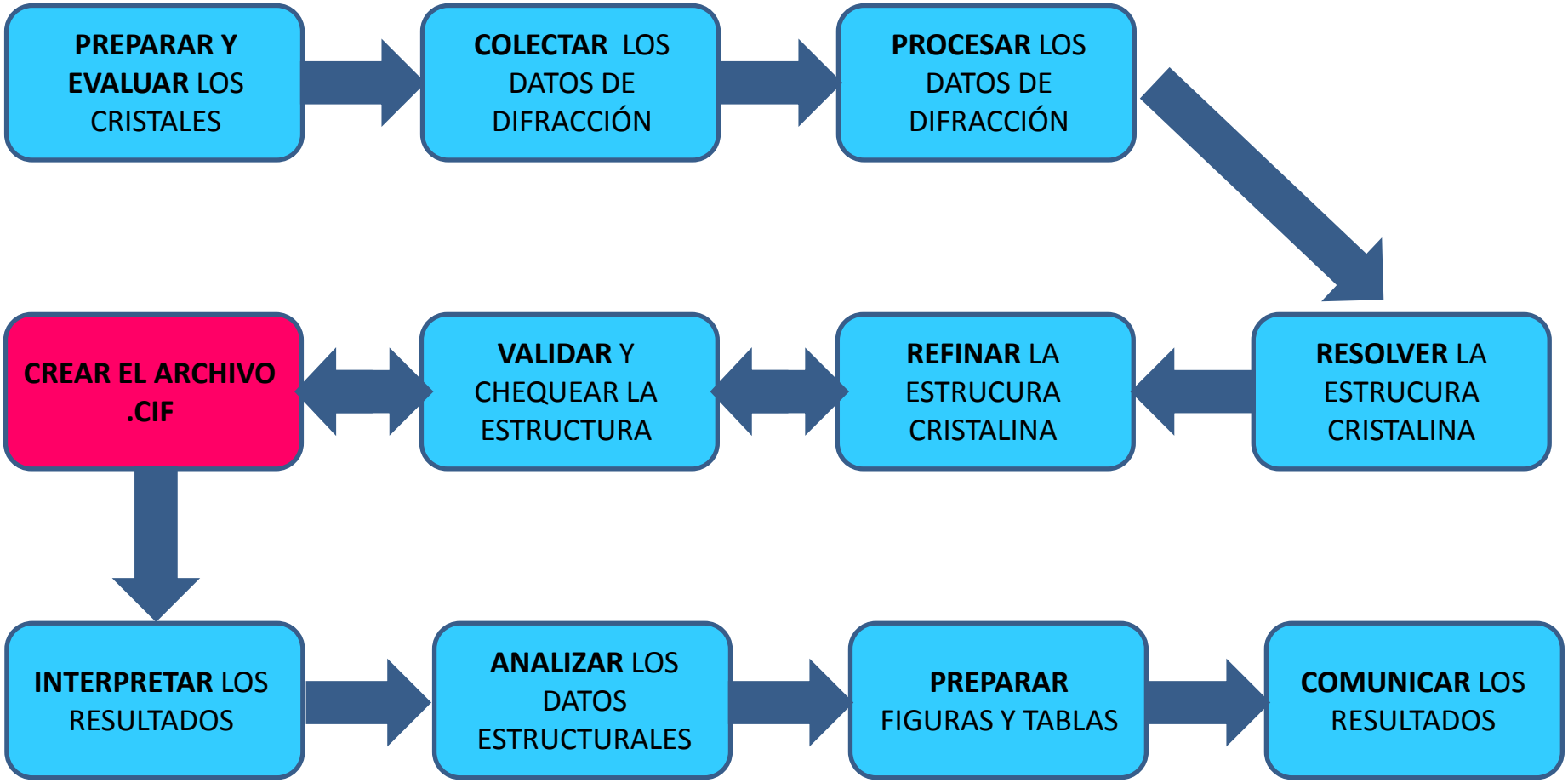
Florencia Di Salvo | Sebastián Suárez

Departamento de Química Inorgánica, Analítica y Química Física, FCEN, UBA

Qué hicmos/aprendimos hasta ahora?



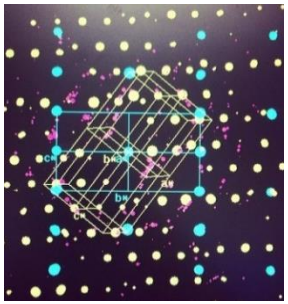
Qué hicmos/aprendimos hasta ahora?



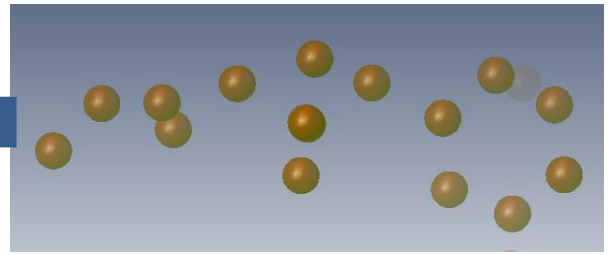
Objetivos y metas

PREPARAR Y EVALUAR LOS CRISTALES

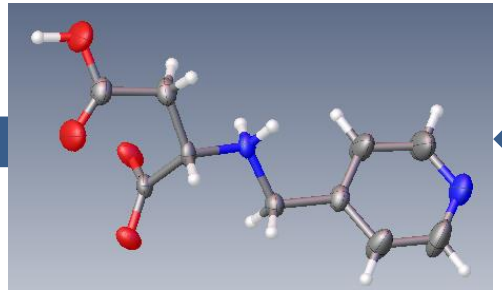
COLECTAR LOS DATOS DE DIFRACCIÓN



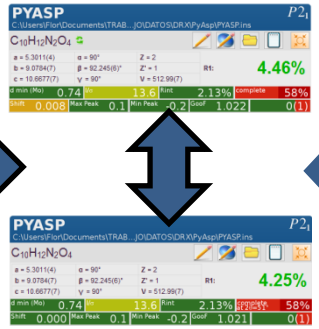
PROCESAR LOS DATOS DE DIFRACCIÓN



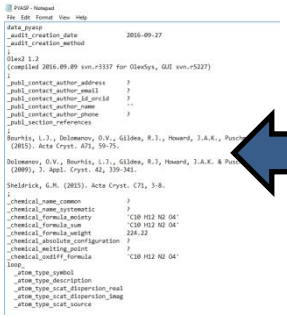
RESOLVER LA ESTRUCURA CRISTALINA



REFINAR LA ESTRUCURA CRISTALINA



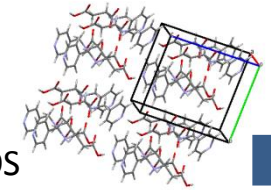
VALIDAR Y CHEQUEAR LA ESTRUCTURA



CIF FILE

INTERPRETAR LOS RESULTADOS

ANALIZAR y DISCUTIR LOS DATOS ESTRUCTURALES



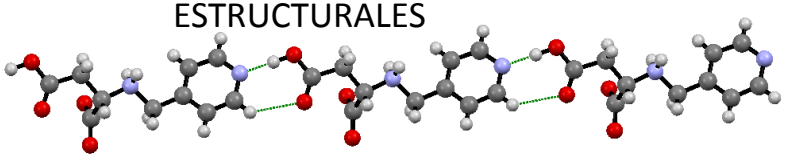


Table 4 Bond Lengths for PYASP.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C4	C12	1.516(4)	C9	N3	1.490(4)
C4	N3	1.503(3)	C10	C12	1.512(4)
C4	C3	1.542(4)	C10	O0AA	1.296(5)
N5	C14	1.318(6)	C10	O1AA	1.192(5)
N5	C15	1.316(6)	C13	C14	1.378(5)
C8	C9	1.509(4)	C15	C16	1.380(5)
C8	C13	1.378(5)	O1	C3	1.255(4)
C8	C16	1.368(5)	O2	C3	1.236(4)

PREPARAR FIGURAS Y TABLAS

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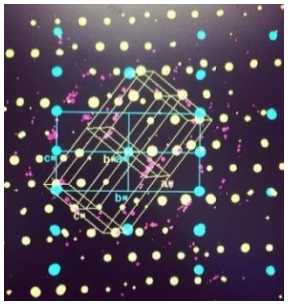
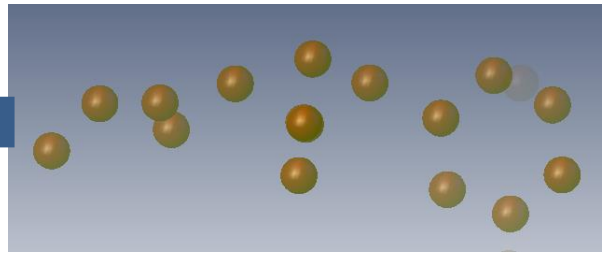
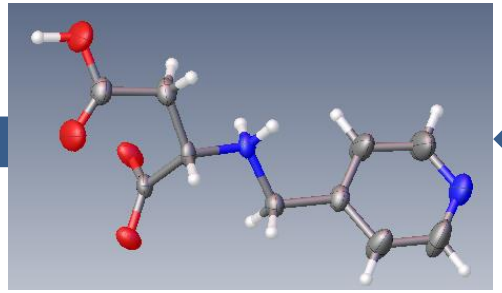
COLECTAR LOS DATOS DE DIFRACCIÓN



PROCESAR LOS DATOS DE DIFRACCIÓN



RESOLVER LA ESTRUCURA CRISTALINA



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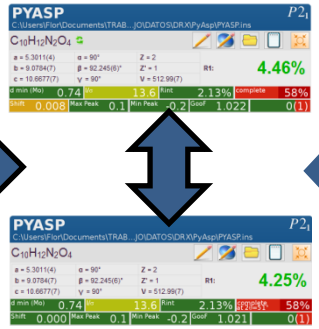
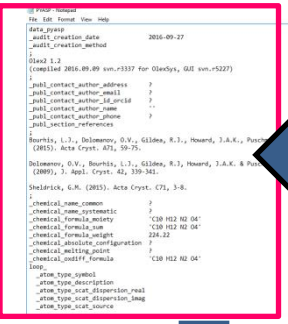


VALIDAR Y CHEQUEAR LA ESTRUCTURA



CIF FILE

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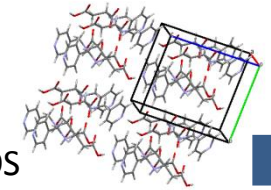
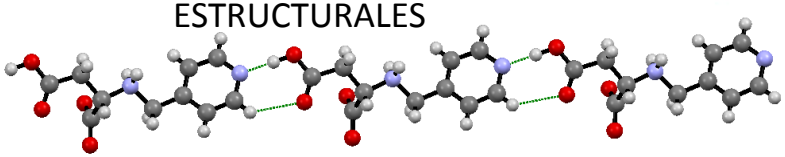


ANALIZAR y DISCUTIR LOS DATOS ESTRUCTURALES



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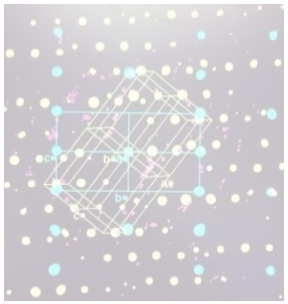
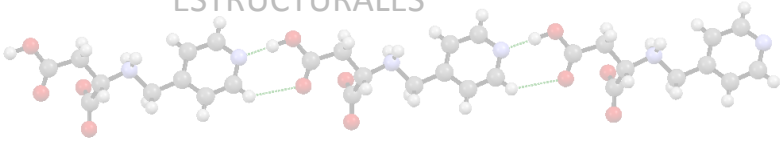


PROCESAR LOS DATOS DE DIFRACCIÓN

LA ESTRUCURA CRISTALINA



ANALIZAR y **DISCUTIR** LOS DATOS ESTRUCTURALES

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C8	C9	1.509(4)	C15	C16	1.380(5)
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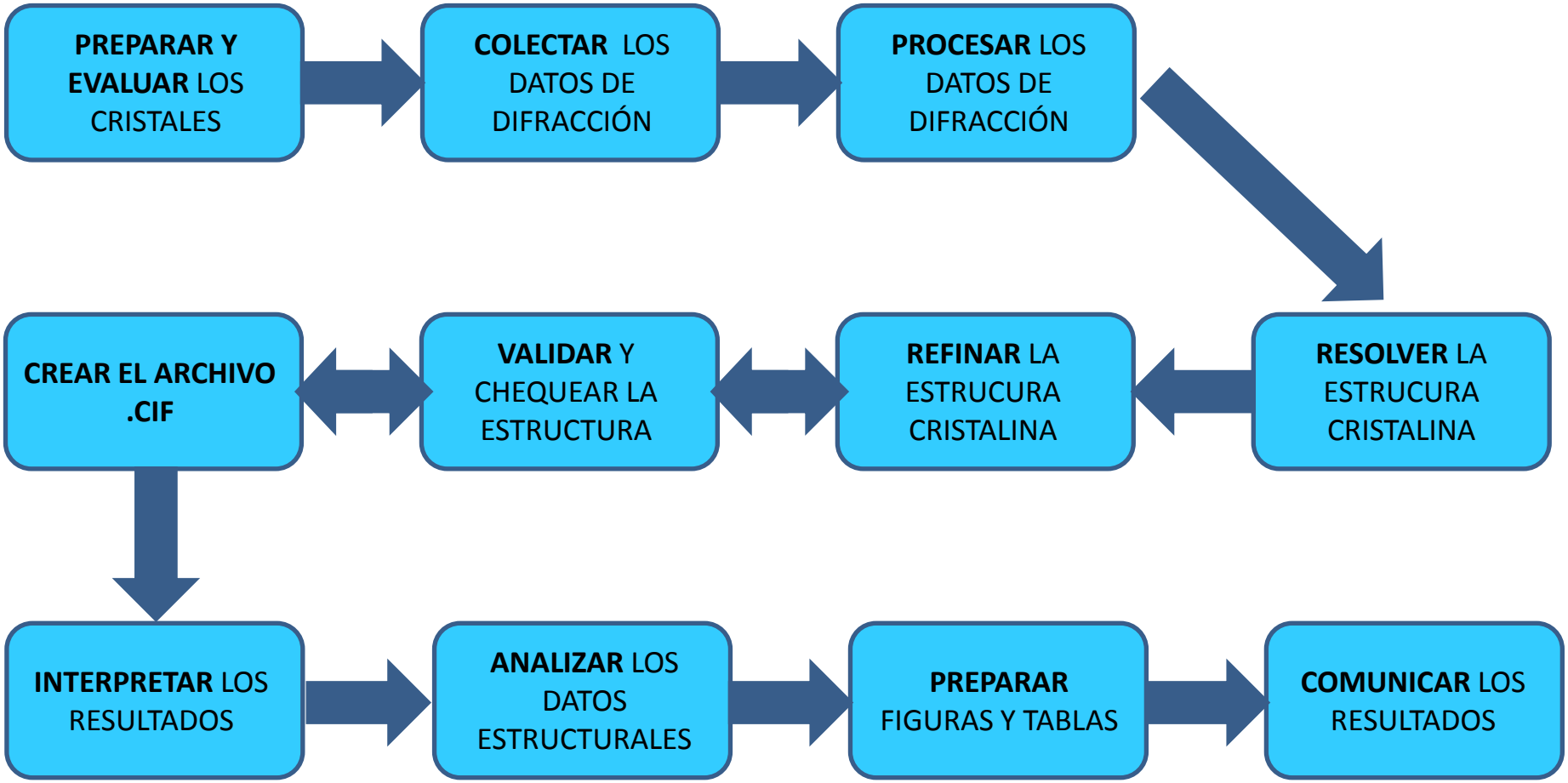
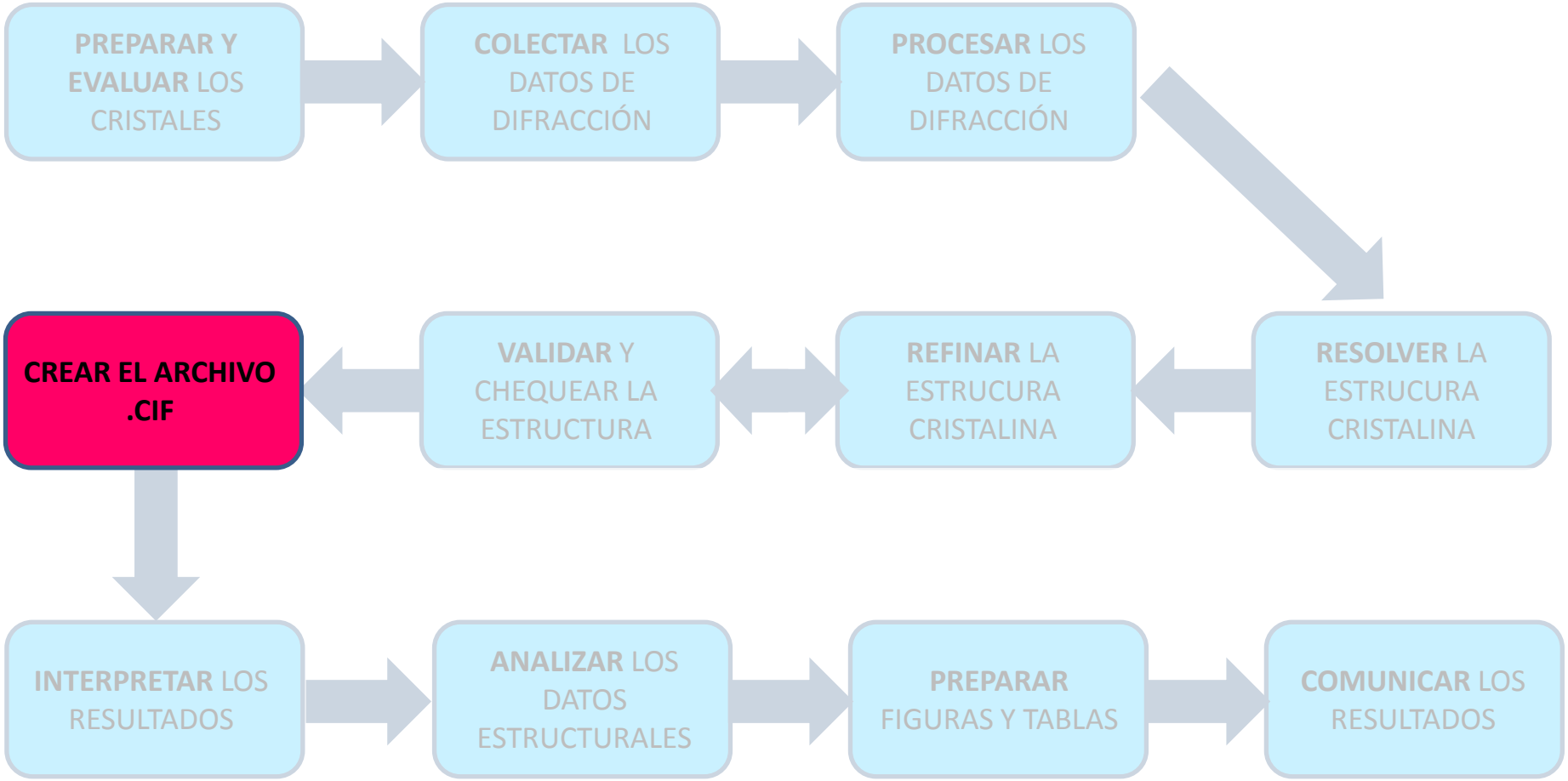


CONTENIDOS DE LA CLASE

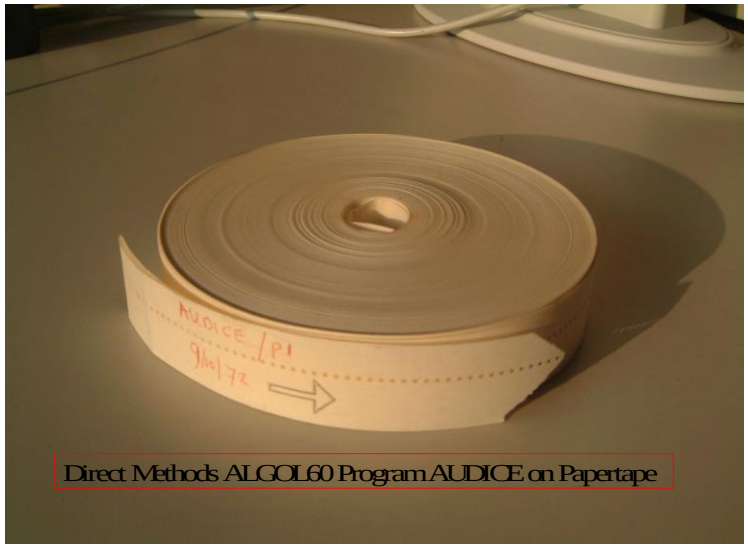


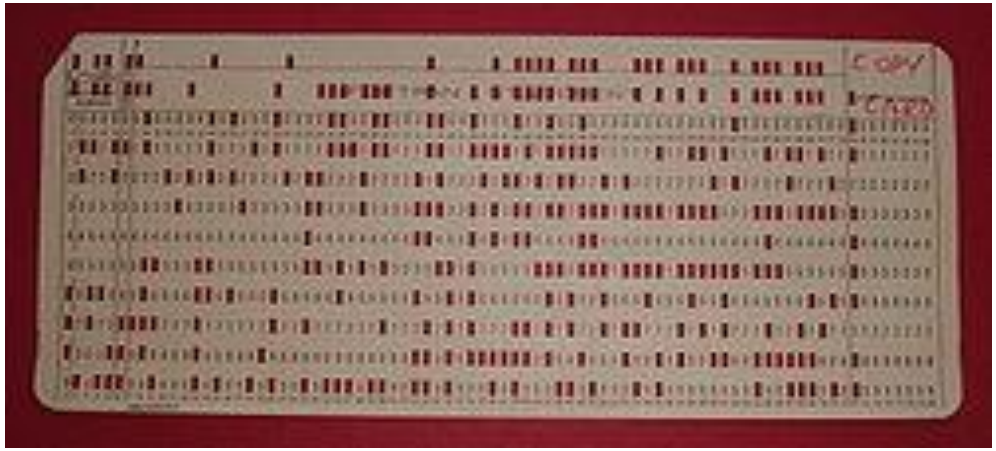
CONTENIDOS DE LA CLASE

CIF : Crystallographic Information File

- **Generación del CIF file**
- **Análisis de la información que contiene y de su estrutcura**
- **Presentar el sowtware para editar y validar el CIF file**
- **Presentar los softwares que se utilizan como imput del CIF file (resumen)**

Almacenamiento de datos en el pasado





Ton Spek presentation: http://www.cryst.chem.uu.nl/spek/ppp/leiden-handout.pdf

Almacenamiento de datos en el pasado

- **Archival of Model Parameters in a Publication (*Acta Cryst.*)**

Table 1. *Atomic parameters*

The values of B are defined by the expression

$$\exp\left[-\tfrac{1}{4}(h^2a^{*2}B_{11}+2hka^*b^*B_{12}+\ldots)\right]$$

used in the structure amplitude calculations.

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{13}
C(1)	0·1105	0·3682	0·1761	2·22	2·53	2·93	0·36	0·38	1·31
C(2)	−0·0139	0·3556	0·1811	2·26	2·94	3·09	0·24	0·25	1·38
C(3)	−0·0360	0·5045	0·2778	2·99	4·32	3·59	0·11	−0·44	1·95
C(4)	0·0662	0·6711	0·3683	4·00	4·13	3·55	0·08	−0·54	1·86
C(5)	0·1893	0·6837	0·3624	3·95	4·32	3·83	−0·94	−0·80	1·66
C(6)	0·2125	0·5301	0·2662	2·75	3·77	3·90	−0·35	−0·06	1·72
O(1)	0·1251	0·2137	0·0767	2·33	3·73	3·73	−0·32	−0·56	1·95
O(2)	−0·1180	0·2009	0·0888	2·37	4·47	4·84	−0·82	−1·70	2·25
H(1)	0·2242	0·2468	0·0813						
H(2)	−0·1208	0·0410	0·0249						
H(3)	−0·1325	0·4948	0·2823						
H(4)	0·0488	0·7892	0·4435						
H(5)	0·2687	0·8116	0·4329						
H(6)	0·3090	0·5401	0·2618						

Mean isotropic $B=3·58$

- **Archival of Reflection Data in a Publication (*Acta Cryst.*)**

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Table 2. *Observed and calculated structure amplitudes*

H	K	L	F(OBS)	F(CAL)
0	0	1	34·5	+35·7
0	0	2	35·9	−34·5
0	0	3	10·5	+9·2
0	0	4	21·8	−20·9
0	0	5	11·2	−10·8
0	0	6	11·7	−10·6
0	0	7	11·8	−11·0
0	0	8	3·7	−3·1
0	0	9	10·6	−11·1
0	0	11	11·8	+11·2
2	0	−12	0·5	+0·5
2	0	−10	3·3	−3·6
2	0	−9	1·9	−1·9
2	0	−8	7·1	−7·2
2	0	−7	6·5	+5·5
2	0	−6	26·7	+26·2
2	0	−5	5·8	−5·2
2	0	−4	11·4	−11·1
2	0	−3	2·4	−3·2
2	0	−2	24·8	−25·0
2	0	−1	40·6	+39·7
12	0	−8	2·8	+3·0
12	0	−7	5·8	+6·2
12	0	−6	13·3	+14·1
12	0	−5	8·5	+8·7
12	0	−4	2·7	−3·4
12	0	−3	2·6	−2·6
12	0	−2	0·7	−0·7
12	0	−1	7·9	−7·8
12	0	0	9·6	−9·0
14	0	−7	3·1	−3·5
14	0	−6	3·7	−4·1
14	0	−5	3·1	+3·2
0	1	1	22·4	−21·2
0	1	2	94·3	−94·8
0	1	3	21·1	−22·5
0	1	4	8·2	−8·5
0	1	5	14·6	−13·5
0	1	6	2·9	+2·9
0	1	7	6·4	−5·5
0	1	8	1·0	+1·1
4	2	9	1·8	−2·1
5	1	−12	1·6	+1·7
5	1	−11	2·4	−2·7
5	1	−10	1·9	−1·9
5	1	−8	1·3	+1·4
5	1	−7	1·0	+1·9
5	1	−6	5·1	+5·1
5	1	−5	11·2	+11·2
5	1	−4	8·6	−8·0
5	1	−3	13·0	−13·2
5	1	−2	11·3	+12·4
5	1	−1	2·9	+1·4
5	1	0	17·3	−16·1
5	1	1	8·2	−7·8
5	1	2	2·8	+2·4
5	1	4	1·4	−1·5
5	1	5	3·4	+5·8
5	1	6	4·5	+5·3
5	1	7	1·7	−2·1
5	1	8	2·0	−2·5
11	1	−11	2·3	+1·9
11	1	−10	1·0	−1·0
11	1	−9	3·2	−2·9
11	1	−8	3·6	+3·4
11	1	−7	7·1	+6·4
11	1	−6	2·0	−2·3
11	1	−5	4·1	−4·3
11	1	−4	5·8	+5·3
11	1	−3	3·7	+3·3
11	1	−2	5·0	−5·1
11	1	−1	3·1	−3·1
11	1	0	0·7	+0·6
11	1	2	0·7	−0·6
13	1	−11	4·3	+3·9
13	1	−10	6·7	+6·4
13	1	−9	7·8	+7·6
13	1	−8	4·8	+5·0
13	1	−7	1·1	+1·2
13	1	−6	2·6	−2·8
13	1	−5	5·4	−5·8
13	1	−4	8·1	−8·5
4	2	−8	9·2	−9·0
4	2	−7	4·3	−3·3
4	2	−6	12·3	−12·9
4	2	−5	28·5	−29·4
4	2	−4	5·8	−6·0
4	2	−3	19·1	+19·2
4	2	−2	9·0	+9·1
4	2	−1	9·0	+8·7
4	2	0	30·9	+29·0
4	2	1	25·8	+24·5
4	2	2	1·7	−1·2
4	2	3	3·3	−3·3
4	2	4	1·6	+1·2
4	2	5	5·1	−4·8
4	2	6	9·2	−10·9
4	2	7	3·2	−3·9
4	2	8	1·8	+1·9
5	2	−12	0·5	+0·7
5	2	−11	3·9	−3·8
5	2	−9	6·5	+7·0
5	2	−8	4·6	+4·4
10	2	1	2·6	−2·4
10	2	2	1·2	+1·0
11	2	−11	2·9	+2·1
11	2	−10	4·2	+3·8
11	2	−9	3·8	+3·7
11	2	−7	2·0	−1·9
11	2	−5	1·6	−1·2
11	2	−4	3·0	−2·6
11	2	−3	1·9	−1·5
11	2	−2	3·3	−3·3
11	2	−1	1·7	−1·6
11	2	0	1·5	+1·4
11	2	1	3·1	+2·7
12	2	−10	2·5	+2·0
12	2	−9	1·3	+0·9
12	2	−8	7·9	−7·9
12	2	−7	9·2	−8·8
12	2	−6	3·4	−2·7
12	2	−5	4·1	−3·9
12	2	−4	6·7	−6·7

Ton Spek presentation: http://www.cryst.chem.uu.nl/spek/ppp/leiden-handout.pdf

Problema en los 90’

- Múltiples formas de almacenamiento de datos (in típicamente en físico)
- (a menudo solo copia impresa)
- No existía formato estandar para ser leído con una computadora que permitiera generar archivo de información, e intercambio y difusión de datos
- Entrada de datos de datos para publicarse se realizaba por re-escripción de los mismos
- No era fácil realizar un control numérico ágil para control de las referencias
- La generación de la base de datos cristalográfica CSD sólo era posible por retipeo de los datos obtenidos a partir de las publicaciones.
- Múltiples errores tipográficos e inconsistencias en los datos publicados
- Muy frecuentemente la información reportada se encontraba incompleta

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Archivo CIF: la solución a los problemas

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Ton Spek presentation: http://www.cryst.chem.uu.nl/spek/ppp/leiden-handout.pdf

CIF : Crystallographic Information File

The acronym CIF is used both for the *Crystallographic Information File*, the data exchange standard file format of Hall, Allen & Brown (1991),[1] and for the *Crystallographic Information Framework*, a broader system of exchange protocols based on data dictionaries and relational rules expressible in different machine-readable manifestations, including, but not restricted to, Crystallographic Information File and XML.

Sitio en la IUCr: https://www.iucr.org/resources/cif

DICCIONARIO de los términos incluidos en el CIF
https://www.iucr.org/resources/cif/dictionaries

Referencias y documentos relacionados al CIF
https://www.iucr.org/resources/cif/documentation

1. *The Crystallographic Information File (CIF): a New Standard Archive File for Crystallography*.
S. Hall, F. H. Allen & I. D. Brown. *Acta Cryst.* **1991**, A47, 655-685

Archivo CIF inicial

- **Una vez que tenemos nuestra estructura refinada** y resolvimos (o pensamos que…) todos los problemas referidos a: grupo espacial, desorden, átomos de H, twinning, reflexiones pobres, análisis de la estructura extendida, configuración absoluta, solvente, etc, **comenzamos a trabajar en el archivo CIF para obtener la versión completa del mismo. Este archivo es creado por los programas de refinamiento (SHELXL, etc)**

- **Este primer archivo CIF tendrá mucha información pero faltará también información valiosa y necesaria.**

- Recomendable: realizar **validación utilizando PLATON** para confirmar que la etapa de refinamiento está completa (ver clases TOPIC 5.2).

- **OPCIONAL: s**e puede realizar una verificación del archivo CIF inicial con el el programa **checkcif**, para visualizar los datos que faltan antes de comenzar.

https://checkcif.iucr.org/

Archivo CIF

exp_3.cif - Notepad

File Edit Format View Help

```
data_shelxl

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety          ?
_chemical_formula_sum
 'C7 H9 N O3 S'
_chemical_formula_weight          187.22

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0030   0.0020
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0060   0.0030
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0110   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'S'  'S'   0.1250   0.1230
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            ?
_symmetry_space_group_name_H-M    ?

loop_
 _symmetry_equiv_pos_as_xyz
```

exp_3.cif - Notepad

File Edit Format View Help

```
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            ?
_symmetry_space_group_name_H-M    ?

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, y+1/2, -z+1/2'
 '-x, -y, -z'
 'x, -y-1/2, z-1/2'

_cell_length_a                    9.766
_cell_length_b                    12.545
_cell_length_c                    6.808
_cell_angle_alpha                 90.00
_cell_angle_beta                  102.33
_cell_angle_gamma                 90.00
_cell_volume                      814.7
_cell_formula_units_Z             4
_cell_measurement_temperature     293(2)
_cell_measurement_reflns_used     ?
_cell_measurement_theta_min       ?
_cell_measurement_theta_max       ?

_exptl_crystal_description        ?
_exptl_crystal_colour             ?
_exptl_crystal_size_max           ?
_exptl_crystal_size_mid           ?
_exptl_crystal_size_min           ?
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.526
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              392
_exptl_absorpt_coefficient_mu     0.361
_exptl_absorpt_correction_type    ?
_exptl_absorpt_correction_T_min   ?
```

Archivo CIF

exp_3.cif - Notepad

File Edit Format View Help

```
 ?
;

_diffrn_ambient_temperature        293(2)
_diffrn_radiation_wavelength       0.71073
_diffrn_radiation_type             MoK\a
_diffrn_radiation_source           'fine-focus sealed tube'
_diffrn_radiation_monochromator    graphite
_diffrn_measurement_device_type    ?
_diffrn_measurement_method         ?
_diffrn_detector_area_resol_mean   ?
_diffrn_standards_number           ?
_diffrn_standards_interval_count   ?
_diffrn_standards_interval_time    ?
_diffrn_standards_decay_%          ?
_diffrn_reflns_number              3856
_diffrn_reflns_av_R_equivalents    0.0317
_diffrn_reflns_av_sigmaI/netI      0.0250
_diffrn_reflns_limit_h_min         -12
_diffrn_reflns_limit_h_max         11
_diffrn_reflns_limit_k_min         -14
_diffrn_reflns_limit_k_max         15
_diffrn_reflns_limit_l_min         -8
_diffrn_reflns_limit_l_max         4
_diffrn_reflns_theta_min           2.13
_diffrn_reflns_theta_max           26.20
_reflns_number_total               1637
_reflns_number_gt                  1496
_reflns_threshold_expression       >2sigma(I)

_computing_data_collection         ?
_computing_cell_refinement         ?
_computing_data_reduction          ?
_computing_structure_solution      ?
_computing_structure_refinement    'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics      ?
_computing_publication_material    ?
```

exp_3.cif - Notepad

File Edit Format View Help

```
_computing_publication_material    ?

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0932P)^2^+1.8619P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          1637
_refine_ls_number_parameters      117
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0651
_refine_ls_R_factor_gt            0.0617
_refine_ls_wR_factor_ref          0.1815
_refine_ls_wR_factor_gt           0.1791
_refine_ls_goodness_of_fit_ref    1.131
_refine_ls_restrained_S_all       1.131
_refine_ls_shift/su_max           0.011
_refine_ls_shift/su_mean          0.001

loop_
```

Comentario acerca de las reflexiones en el CIF

exp_4170.cif - Notepad

File Edit Format View Help

```
Q20    1    1.0738   0.5747   0.4970   11.
;
_shelx_res_checksum                      457
_shelx_hkl_file
;
   0 -11    0-340.862 208.985    1
   0 -11   -1 254.810 181.399    1
   0 -11    1-74.3091 116.039    1
   0 -11   -2 1185.44 384.048    1
   0 -11    2 1013.28 340.684    1
   0 -11   -3 107.348 134.786    1
   0 -11    3 189.908 206.317    1
   0 -11    4 379.994 239.583    1
   0 -11    4 194.513 153.368    4
   0 -11    5 9.09847 92.3042    4
   0 -11   -5-34.8898 72.9756    7
   0 -11   -6 585.271 322.632    7
   0 -11    6 178.277 227.370    1
   0 -10    0 1103.35 344.743    1
   0 -10    1 664.885 274.302    1
   0 -10   -1 834.503 292.807    1
   0 -10    2 262.675 173.249    1
   0 -10   -2 121.265 180.597    1
   0 -10    3 283.457 208.745    4
   0 -10    3-17.4642 89.7694    1
   0 -10   -3 230.048 167.638    1
   0 -10    4 135.832 199.915    4
   0 -10   -4-36.3725 77.9838    7
   0 -10   -4 104.925 173.085    1
   0 -10    4 120.064 140.969    1
   0 -10    5 286.857 191.882    4
   0 -10    5 255.735 183.686    1
   0 -10   -5 541.957 266.158    1
   0 -10   -6 309.207 205.892    7
   0 -10    6-110.072 170.188    1
   0 -10    6-105.263 160.799    4
   0 -10    7 1257.00 418.212    4
```

- El archivo que contiene las **reflexiones colectadas** es muy importante para poder **analizar la calidad de los datos presentados en el CIF**, ya sea para su publicación o para sostener el análisis estructural y supramolecular que se desee realizar.

- En versiones anteriores del SHELX, luego del refinamiento, las **reflexiones** se encontraban en el **archivo FCF**. En versiones más recientes, **las reflexiones están incluidas en el CIF generado.**

- **Para la validación (checkcif), si las reflexiones están incluidas, solo se debe proporcionar el archivo CIF para su análisis. En caso contario, hay que proveer el archivo FCF (o .hkl) para su validación completa.**

Información faltante en el archivo CIF

- **La idea es editar el archivo CIF para completarlo y poder utilizarlo**

Información faltante (no toda en cada ítem):

- Datos y contactos de los autores
- Datos experimentales (pueden obviarse)
- Datos del cristal (características: dimensiones, aspecto, etc)
- Datos de la estructura (grupo espacial)
- Datos del equipamiento
- Datos de las condiciones del experimento de difracción
- Datos de la experimento de difracción (temperatura, reflexiones, corrección por absorción)
- Datos del refinamiento

- **UTILIDAD del archivo CIF**

- Archivar los datos obtenidos
- Realizar un depósito (aporte) a la base cristalográfica CSD (-> numero de la CSD)
- Incluirlo como material suplementario de una publicación
- Utilizarlo como punto de partida para estudios computacionales (cálculos de estructura electrónica)
- Utilizarlo para realizar estudios estructurales moleculares y supramoleculares con otros programas (Mercury, CrystalExplorer, etc)
- Publicar la estructura en Acta Crystallographica

Validar CIF... https://checkcif.iucr.org/

checkCIF

A service of the
International Union of Crystallography

checkCIF reports on the consistency and integrity of crystal structure determinations reported in CIF format.

Please upload your CIF using the form below.

File name:
Seleccionar archivo exp_4769.cif

Select form of checkCIF report
- (•) HTML
- ( ) PDF (recommended for CIFs that might take a long time to check)

Select validation type
- (•) Full validation of CIF and structure factors
- ( ) Full IUCr publication validation of CIF and structure factors
- ( ) Validation of CIF only (no structure factors)

Output Validation Response Form
- ( ) Level A alerts only
- ( ) Level A and B alerts
- (•) Level A, B and C alerts
- ( ) None

Send CIF for checking

checkCIF is sponsored by

IUCr Journals

ELSEVIER

WILEY

IUCrData

ROYAL SOCIETY OF CHEMISTRY

Validar CIF… https://checkcif.iucr.org/

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Select validation type
- Full validation of CIF and structure factors
- Full IUCr publication validation of CIF and structure factors
- Validation of CIF only (no structure factors)

Output Validation Response Form
- Level A alerts only
- Level A and B alerts
- Level A, B and C alerts
- None

Send CIF for checking

(veremos los detalles de la salida de esta aplicación más adelante)

checkCIF is sponsored by

IUCr Journals

ELSEVIER

WILEY

IUCrData

ROYAL SOCIETY OF CHEMISTRY

Validar CIF... https://checkcif.iucr.org/

CellCheckCSD - The Cam | (IUCr) Crystallographic In | (IUCr) Crystallographic

checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF ...
No extractable fcf data in found in CIF

checkCIF/PLATON (basic structural check)

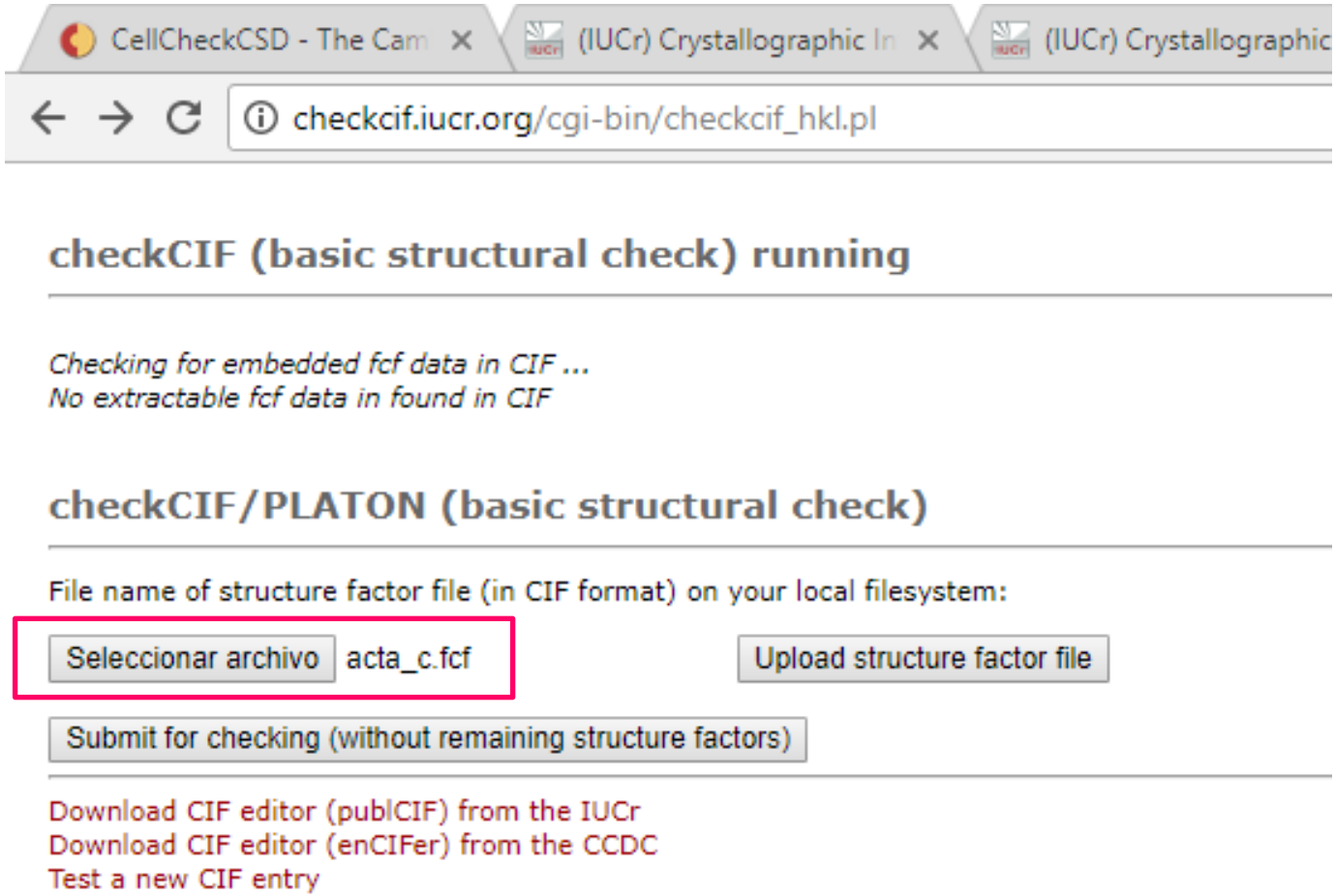
File name of structure factor file (in CIF format) on your local filesystem:

Seleccionar archivo acta_c.fcf Upload structure factor file

Submit for checking (without remaining structure factors)

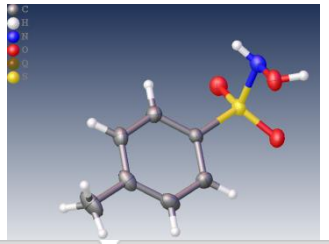
Download CIF editor (publCIF) from the IUCr
Download CIF editor (enCIFer) from the CCDC
Test a new CIF entry

Validar CIF… https://checkcif.iucr.org/



Validación

- Ejemplo



checkCIF

A service of the
International Union of Crystallography

CellCheckCSD - The Cam | (IUCr) Crystallographic In | (IUCr) Crystallographic In | checkCIF/PLATON page

checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF ...
Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait .

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) exp_3

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found.
Please wait while processing ....

CIF dictionary
Interpreting this report

Structure factor report

Datablock: exp_3

Bond precision: C-C = 0.0054 A Wavelength=0.71073

Cell: a=9.76600 b=12.54500 c=6.80800
alpha=90 beta=102.3300 gamma=90

Temperature: 293 K

	Calculated	Reported
Volume	814.840	815
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C7 H9 N O3 S	C7 H9 N O3 S
Sum formula	C7 H9 N O3 S	C7 H9 N O3 S
Mr	187.21	187.22
Dx,g cm-3	1.526	1.526
Z	4	4
Mu (mm-1)	0.361	0.361

Validación

- Ejemplo

CellCheckCSD - The Cam | (IUCr) Crystallographic In | (IUCr) Crystallographic

checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

No syntax errors found.
Please wait while processing ....

CIF dictionary
Interpreting this report

Structure factor report

Datablock: exp_3

Bond precision: C-C = 0.0054 A Wavelength=0.71073

Cell: a=9.76600 b=12.54500 c=6.80800
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	Calculated	Reported
Volume	814.840	815
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C7 H9 N O3 S	C7 H9 N O3 S
Sum formula	C7 H9 N O3 S	C7 H9 N O3 S
Mr	187.21	187.22
Dx,g cm-3	1.526	1.526
Z	4	4
Mu (mm-1)	0.361	0.361
F000	392.0	392.0
F000'	392.69	
h,k,lmax	12,15,8	12,15,8
Nref	1641	1637
Tmin,Tmax		
Tmin'		

Correction method= Not given

Data completeness= 0.998 Theta(max)= 26.203

R(reflections)= 0.0617(1496) wR2(reflections)= 0.1815(1637)

S = 1.131 Npar= 117

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

CellCheckCSD - The Cam | (IUCr) Crystallographic In | (IUCr) Crystallographic In

checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

EXPT005_ALERT_1_A _exptl_crystal_description is missing
Crystal habit description.
The following tests will not be performed.
CRYSR_01
DIFF003_ALERT_1_A _diffrn_measurement_device_type is missing
Diffractometer make and type. Replaces _diffrn_measurement_type.
PLAT183_ALERT_1_A Missing _cell_measurement_refins_used Value .... Please Do !
PLAT184_ALERT_1_A Missing _cell_measurement_theta_min Value ..... Please Do !
PLAT185_ALERT_1_A Missing _cell_measurement_theta_max Value ..... Please Do !

Alert level B

PLAT930_ALERT_2_B Check Twin Law (1 0 0)[3 0 1] Estimated BASF 0.12

Alert level C

PLAT052_ALERT_1_C Info on Absorption Correction Method Not Given Please Do !
PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ... Please Check
PLAT054_ALERT_1_C Medium Crystal Dimension Missing (or Error) ... Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ... Please Check
PLAT141_ALERT_4_C s.u. on a - Axis Small or Missing ..... 0.00000 Ang.
PLAT142_ALERT_4_C s.u. on b - Axis Small or Missing ..... 0.00000 Ang.
PLAT143_ALERT_4_C s.u. on c - Axis Small or Missing ..... 0.00000 Ang.
PLAT145_ALERT_4_C s.u. on beta Small or Missing ..... 0.0000 Degree
PLAT151_ALERT_1_C No s.u. (esd) Given on Volume ..... Please Do !
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00543 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 4.465 Check
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 2 Check
PLAT939_ALERT_3_C Large Value of Not (SHELXL) Weight Optimized S . 13.34 Check

Alert level G

PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K) 293 Check
PLAT200_ALERT_1_G Reported _diffrn_ambient_temperature ..... (K) 293 Check
PLAT380_ALERT_4_G Incorrectly? Oriented X(sp2)-Methyl Moiety ..... C7 Check
PLAT870_ALERT_4_G ALERTS Related to Twinning Effects Suppressed .. ! Info
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 2 Note
PLAT931_ALERT_5_G Found Twin Law (1 0 0)[ ] Est. BASF 0.12 Check

Validación

- Ejemplo

CellCheckCSD - The Cam | (IUCr) Crystallographic In | (IUCr) Crystallographic In | checkCIF

checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

5 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
13 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
6 **ALERT level G** = General information/check it is not something unexpected

12 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
7 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor ale point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to th fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary carry out additional measurements or structure refinements. However, the purpose of your study may just the reported deviations and the more serious of these should normally be commented upon in the discussi or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are aspects of the results needing attention. It is up to the individual to critically assess their own results and, necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior t submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_EXPT005_exp_3
```

CellCheckCSD - The Cam | (IUCr) Crystallographic In | (IUCr) Crystallographic In

checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

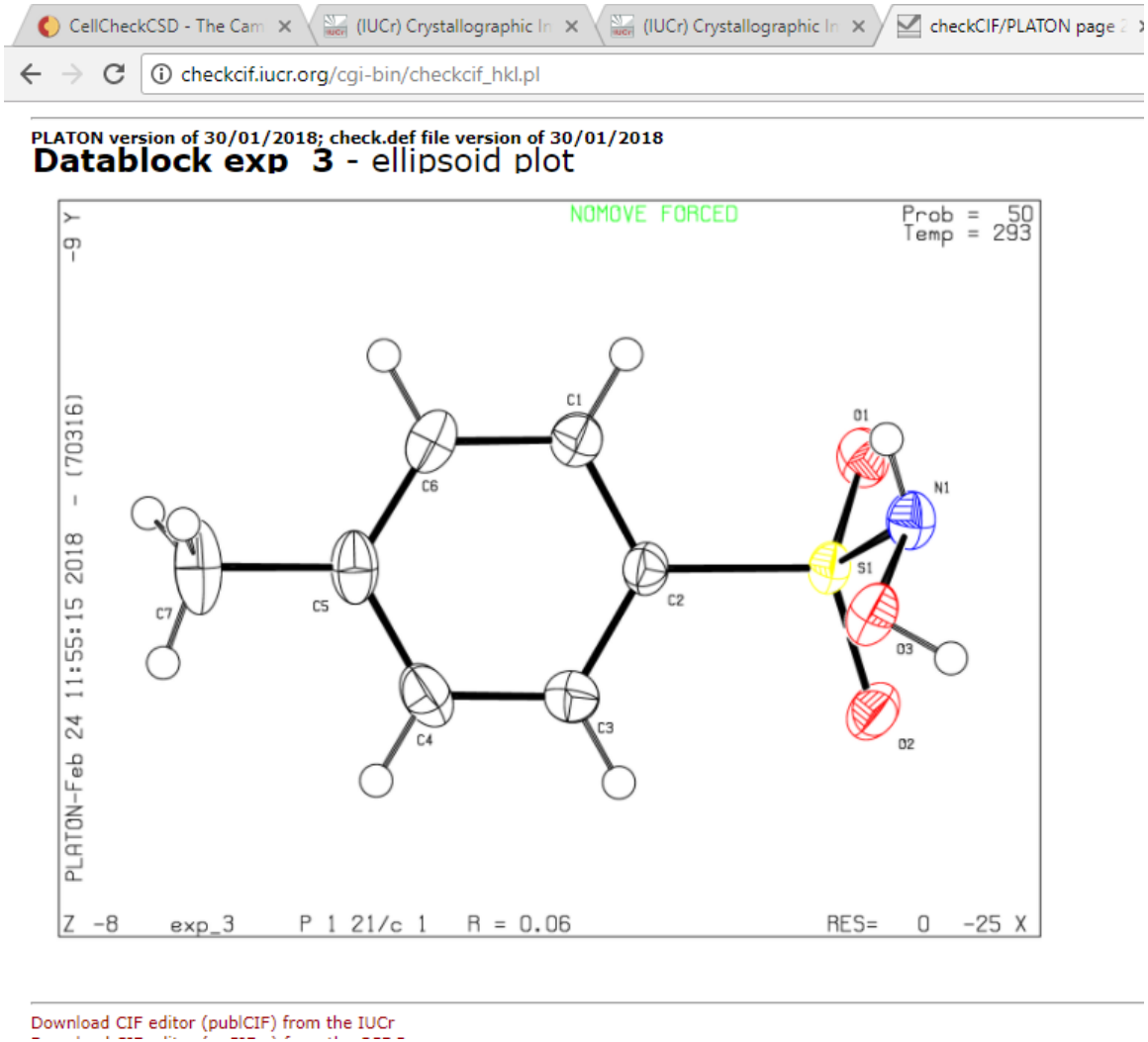
Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF

```
# start Validation Reply Form
_vrf_EXPT005_exp_3
;
PROBLEM: _exptl_crystal_description is missing
RESPONSE: ...
;
_vrf_DIFF003_exp_3
;
PROBLEM: _diffrn_measurement_device_type is missing
RESPONSE: ...
;
_vrf_PLAT183_exp_3
;
PROBLEM: Missing _cell_measurement_reflns_used Value ....    Please Do !
RESPONSE: ...
;
_vrf_PLAT184_exp_3
;
PROBLEM: Missing _cell_measurement_theta_min Value ......    Please Do !
RESPONSE: ...
;
_vrf_PLAT185_exp_3
;
PROBLEM: Missing _cell_measurement_theta_max Value ......    Please Do !
RESPONSE: ...
;
_vrf_PLAT052_exp_3
;
PROBLEM: Info on Absorption Correction Method   Not Given    Please Do !
RESPONSE: ...
;
_vrf_PLAT053_exp_3
;
PROBLEM: Minimum Crystal Dimension Missing (or Error) ...    Please Check
RESPONSE: ...
;
_vrf_PLAT054_exp_3
```

Validación

- Ejemplo

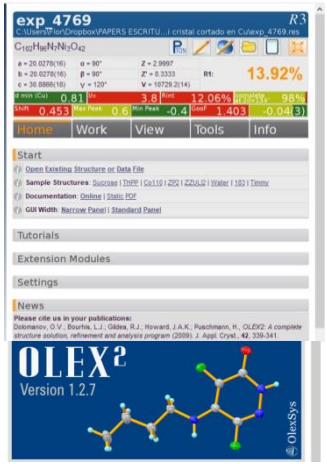


Completando el archivo CIF

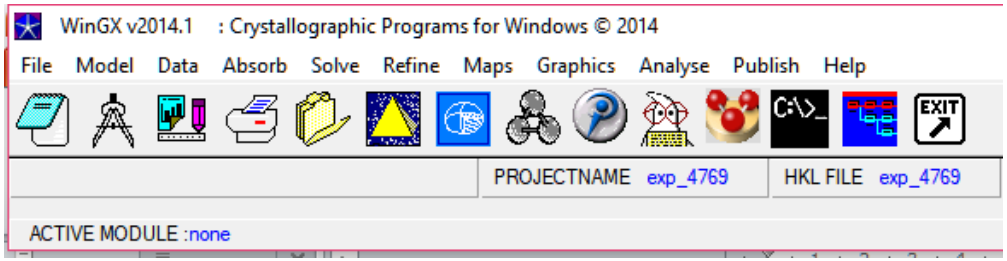
El archivo CIF puede editarse y/o completarse mediante las siguientes opciones:

1) Editor de texto

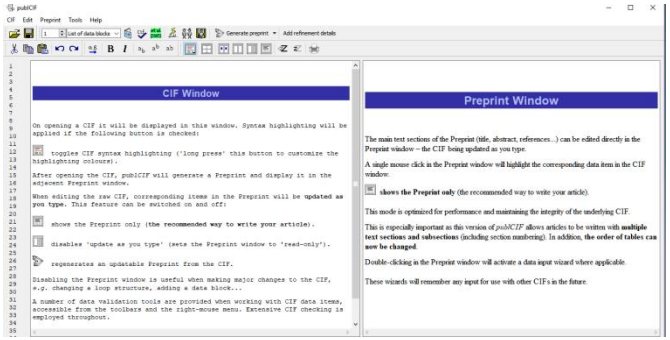
2) Programa Olex2

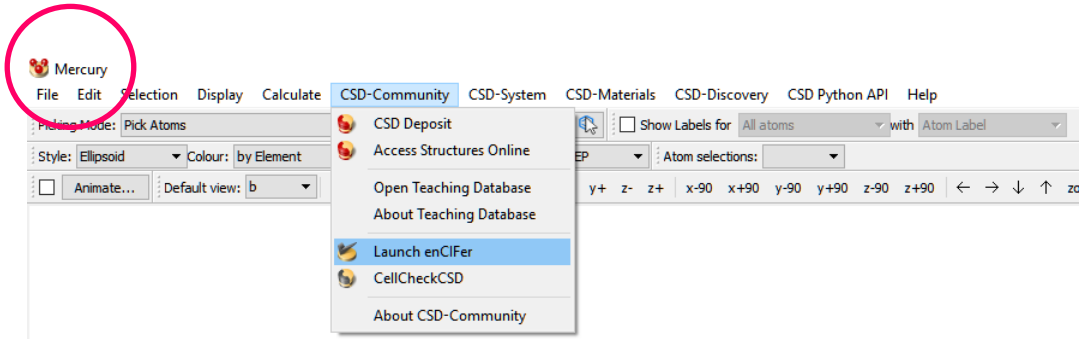


3) Wingx



5) publiCIF (de IUCr: [http://journals.iucr.org/services/cif/publcif/])





4) enCIFer (de CCDC: [https://www.ccdc.cam.ac.uk/Community/csd-community/encifer/])

Completando el archivo CIF

https://www.iucr.org/resources/cif/software#4

4. File manipulation programs

- **cif2cif** copies and pretty-prints CIFs; reformats numbers to retain standard uncertainties within defined ranges; fetches a subset of CIF according to a request list
- **CIF2CRY** program for converting structural data and reflection data contained in a CIF into instructions to be read by the *CRYSTALS* program
- **cif2pdb** converts mmCIF data sets to PDB format
- **cif2sx** converts CIF files to *SHELXL* instruction files
- **cif2xml** copies a CIF on standard input to equivalent XML on standard output
- **CIFFOLD** application for folding and unfolding long lines in CIF
- **ciftex** filter for translating a CIF to a TeX file
- **cifTr** application program for translating files in mmCIF format into files in PDB format (from the Protein Data Bank)
- **Db Loader** (*mmCIF Loader*) conversion of CIF data into a form loadable into a variety of relational database engines
- **DIFRAC** converts diffractometer output files to CIF format
- **GSAS2CIF** application to create CIFs from GSAS results (NIST web site)
- **MAXIT** application for processing and annotating of macromolecular structure data (from the Protein Data Bank)
- **Open Babel** open-source chemistry toolboxconverting between more than 90 chemical file formats including CIF (but without symmetry transformations)
- **OpenMMS** toolkit for creating efficient representations of macromolecular structure data in several common formats
- **pdb2cif** converts PDB data sets to mmCIF format
- **PDB_EXTRACT** conversion of output from macromolecular structure packages to mmCIF
- **QUASAR** CIF syntax checking and file manipulation tool

Vista y edición del archivo CIF inicial

exp_3.cif - enCIFer

Mercury

File Edit Selection Display Calculate CSD-Community CSD-System CSD

Picking Mode: Pick Atoms

Style: Ellipsoid Colour: by Element

Animate... Default view: b

- CSD Deposit
- Access Structures Online
- Open Teaching Database
- About Teaching Database
- Launch enCIFer
- CellCheckCSD
- About CSD-Community

exp_3.cif - enCIFer

File Edit Search Tools Help

Blocks: 1

```
data_shelxl

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety          ?
_chemical_formula_sum
 'C7 H9 N O3 S'
_chemical_formula_weight          187.22

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0030   0.0020
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0060   0.0030
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0110   0.0060
```

Editor Visualiser

Errors - none
Warnings - none
Remarks - none

Loaded dictionary: C:/Program Files (x86)/CCDC/Mercury 3.9/enCIFer 1.5.2/dict/ddl_core.dic

Starting new file "untitled1.cif"

Reading CIF "C:\Users\Flor\Desktop\TOPIC_\Matrial\Ejemplo_Seba\exp_3.cif"
Read 262 lines.

No errors, warnings or remarks.

Loading visualiser for block: data_shelxl

Line 14, Column 38 (File Modified)

Vista y edición del archivo CIF inicial

exp_3.cif - enCIFer

exp_3.cif - enCIFer

File Edit Search Tools Help

Blocks: 1

```
data_shelxl

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety          ?
_chemical_formula_sum
 'C7 H9 N O3 S'
_chemical_formula_weight          187.22

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0030   0.0020
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0060   0.0030
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0110   0.0060
```

Editor | Visualiser

Errors - none
Warnings - none
Remarks - none

Loaded dictionary: C:/Program Files (x86)/CCDC/Mercury 3.9/enCIFer 1.5.2/dict/ddl_core.dic

Starting new file "untitled1.cif"

Reading CIF "C:\Users\Flor\Desktop\TOPIC_\Matrial\Ejemplo_Seba\exp_3.cif"
Read 262 lines.

No errors, warnings or remarks.

Loading visualiser for block: data_shelxl

Line 14, Column 38 (File Modified)

Vista y edición del archivo CIF inicial

exp_3.cif - enCIFer

exp_3.cif - enCIFer

File Edit Search Tools Help

Blocks: 1

```
data_shelxl

_audit_creation_method            SHELXL-2017
_chemical_name_systematic
;
 ?
;
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety          ?
_chemical_formula_sum
 'C7 H9 N O3 S'
_chemical_formula_weight          187.22

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0030   0.0020
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0060   0.0030
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0110   0.0060
```

enCIFer Publication Data Wizar!

enCIFer Publication Data Wizard

Contact Information... (page 3 of 8)

Contact author :

Email address :

Contact address :

Telephone number :

Fax number :

Next Cancel

Editor Visualiser

Errors - none
Warnings - none
Remarks - none

Starting new file "untitled1.cif"

Reading CIF "C:\Users\Flor\Desktop\TOPIC_\Matrial\Ejemplo_Seba\exp_3.cif"
Read 262 lines.

No errors, warnings or remarks.

Loading visualiser for block: data_shelxl

Line 14, Column 38 (File Modified)

Vista y edición del archivo CIF inicial

exp_3.cif - enCIFer

exp_3.cif - enCIFer

File Edit Search Tools Help

Blocks: 1

```
data_shelxl

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common
_chemical_melting_point
_chemical_formula_moiety          ?
_chemical_formula_sum
 'C7 H9 N O3 S'
_chemical_formula_weight          187.22

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0030   0.0020
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0060   0.0030
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0110   0.0060
```

enCIFer Chemical and Crystal Data Wizar!

enCIFer Chemical and Crystal Data Wizard

Physical and Chemical Information... (page 3 of 7)

Systematic name: ?

Common name: ?

Moiety Formula: ?

Sum Formula: C7 H9 N O3 S

Compound source:

Physical Properties:

air-sensitive

Add

Next Cancel

Editor Visualiser

Errors - none

Warnings - none

Remarks - none

Starting new file "untitled1.cif"

Reading CIF "C:\Users\Flor\Desktop\TOPIC_\Matrial\Ejemplo_Seba\exp_3.cif"
Read 262 lines.

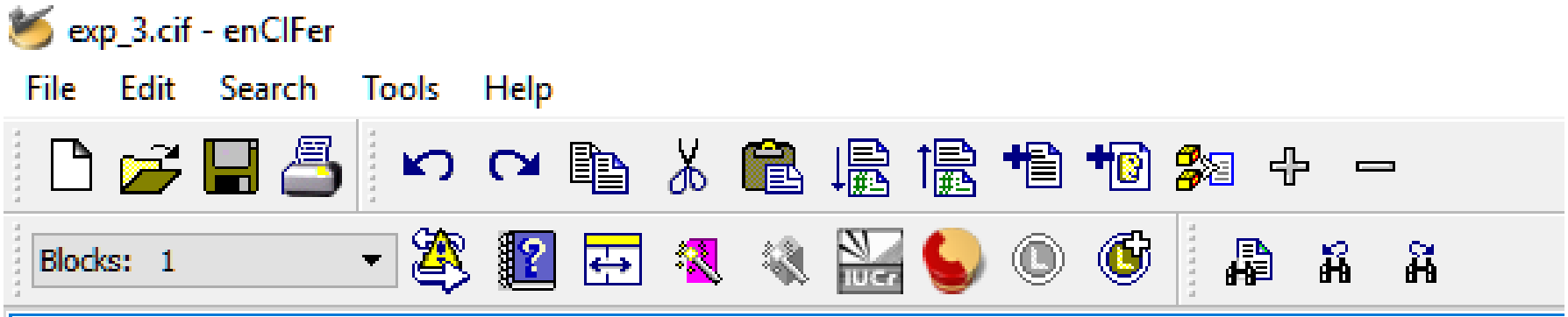
No errors, warnings or remarks.

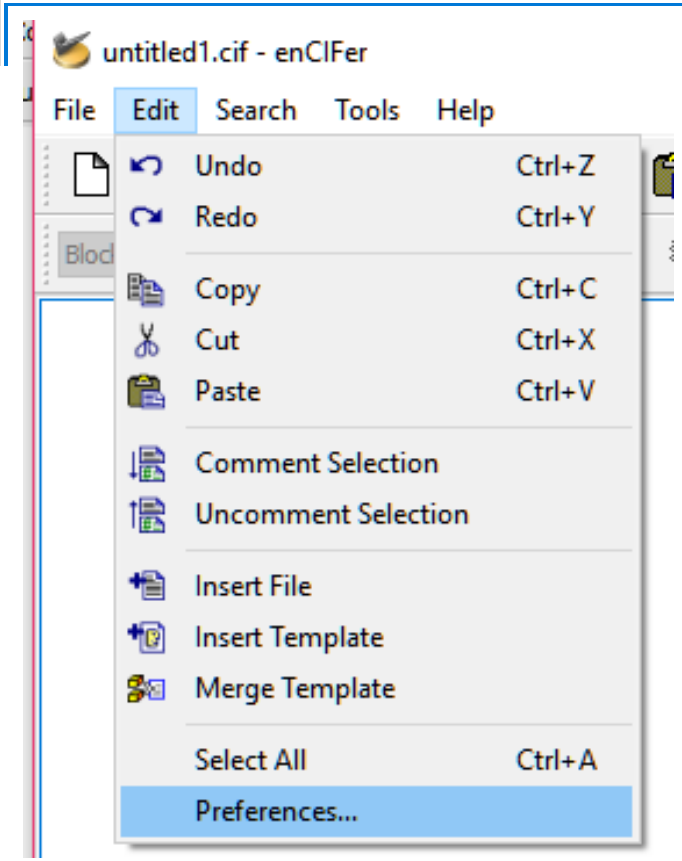
Loading visualiser for block: data_shelxl

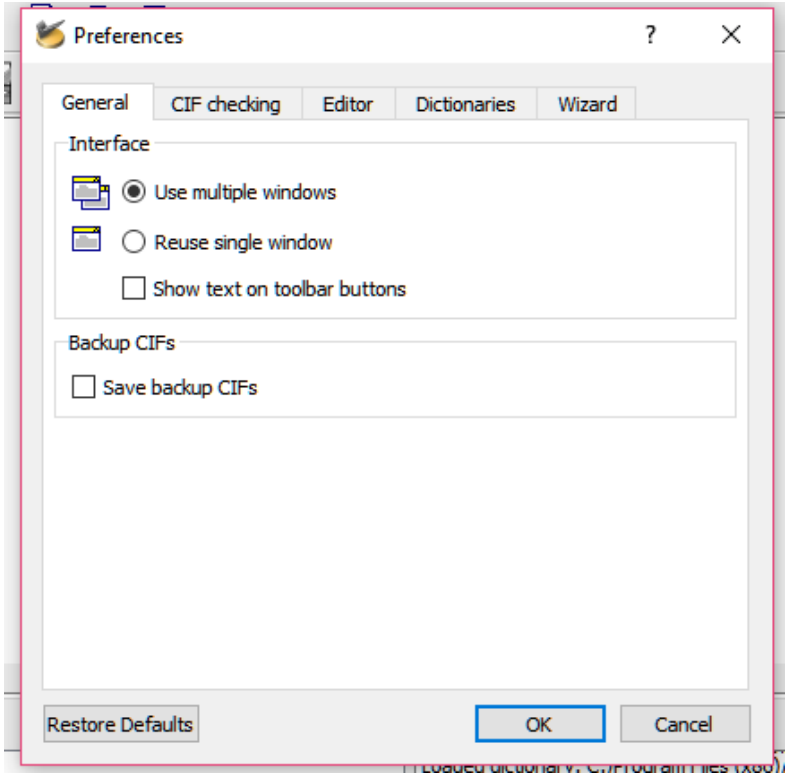
Line 14, Column 38 (File Modified)

Vista y edición del archivo CIF inicial

exp_3.cif - enClFer







Vista y edición del archivo CIF inicial

publCIF

publCIF

CIF Edit Preprint Tools Help

1 List of data blocks | Generate preprint | Add refinement details

CIF Window

On opening a CIF it will be displayed in this window. Syntax highlighting will be applied if the following button is checked:

toggles CIF syntax highlighting ('long press' this button to customize the highlighting colours).

After opening the CIF, *publCIF* will generate a Preprint and display it in the adjacent Preprint window.

When editing the raw CIF, corresponding items in the Preprint will be **updated as you type**. This feature can be switched on and off:

shows the Preprint only (**the recommended way to write your article**).

disables 'update as you type' (sets the Preprint window to 'read-only').

regenerates an updatable Preprint from the CIF.

Disabling the Preprint window is useful when making major changes to the CIF, e.g. changing a loop structure, adding a data block...

A number of data validation tools are provided when working with CIF data items, accessible from the toolbars and the right-mouse menu. Extensive CIF checking is employed throughout.

Preprint Window

The main text sections of the Preprint (title, abstract, references...) can be edited directly in the Preprint window – the CIF being updated as you type.

A single mouse click in the Preprint window will highlight the corresponding data item in the CIF window.

shows the Preprint only (the recommended way to write your article).

This mode is optimized for performance and maintaining the integrity of the underlying CIF.

This is especially important as this version of *publCIF* allows articles to be written with **multiple text sections and subsections** (including section numbering). In addition, **the order of tables can now be changed**.

Double-clicking in the Preprint window will activate a data input wizard where applicable.

These wizards will remember any input for use with other CIFs in the future.

Ready | View log

Vista y edición del archivo CIF inicial

publCIF

publCIF - C:/Users/Flor/Desktop/TOPIC_/Matrial/Ejemplo_Seba/exp_3.cif

CIF Edit Preprint Tools Help

1 List of data blocks | Generate preprint | Add refinement details

```
data_shelxl

_audit_creation_method            SHELXL-97
_chemical_name_systematic
?
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety          ?
_chemical_formula_sum
 'C7 H9 N O3 S'
_chemical_formula_weight          187.22

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0030   0.0020
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0060   0.0030
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0110   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'S'  'S'   0.1250   0.1230
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            ?
_symmetry_space_group_name_H-M    ?

loop_
```

publCIF

Please select the type of article

- Acta Cryst. E
- Acta Cryst. C
- Acta Cryst. B
- IUCrData
- Generic

Preprint Window

ions of the Preprint (title, abstract, references...) can be edited directly in the
- the CIF being updated as you type.

ck in the Preprint window will highlight the corresponding data item in the CIF

reprint only (the recommended way to write your article).

nized for performance and maintaining the integrity of the underlying CIF.

mportant as this version of *publCIF* allows articles to be written with **multiple**
subsections (including section numbering). In addition, **the order of tables can**

the Preprint window will activate a data input wizard where applicable.

remember any input for use with other CIFs in the future.

View log

Vista y edición del archivo CIF inicial

publCIF

publCIF - C:/Users/Flor/Desktop/TOPIC_/Matrial/Ejemplo_Seba/exp_3.cif (modified)

CIF Edit Preprint Tools Help

1 shelxl Acta C preprint Add refinement details

```
data_shelxl


_publ_section_keywords
;
crystal structure
;
#Added by publCIF



_publ_section_title
;
Title (type here to add)
;
#Added by publCIF



_audit_update_record
;
2018-02-24 # Formatted by publCIF

;
_publcif_datablock.id '{54779b2f-b23d-406b-94bc-1d863daaf728}'

# publcif _publ_body_element loop start
loop_
_publ_body_element
_publ_body_title
_publ_body_contents
```

Acta C research paper

Title (type here to add)

?

Keywords

crystal structure

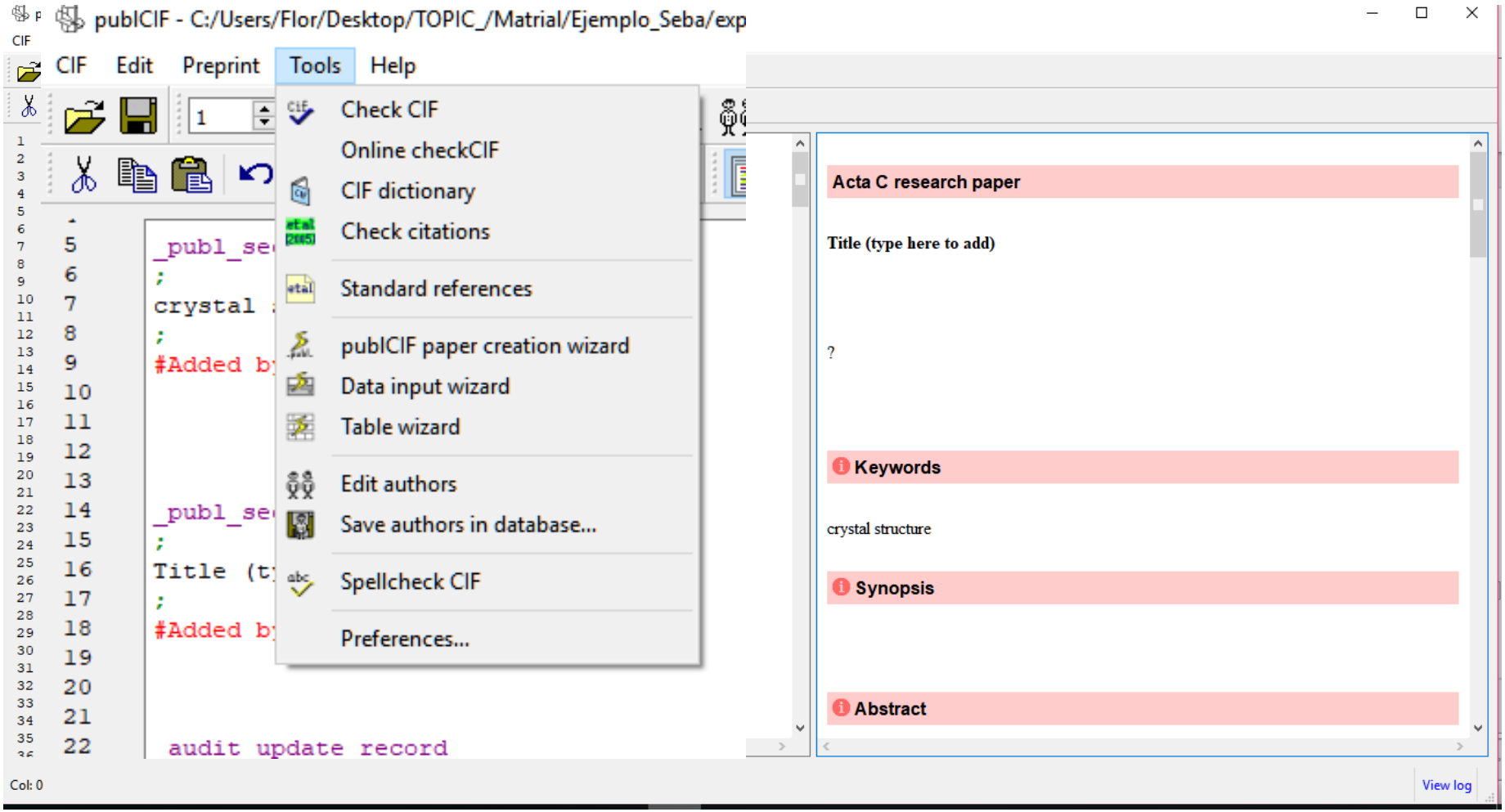
Synopsis

Abstract

Col: 0 View log

Vista y edición del archivo CIF inicial

publCIF



Vista y edición del archivo CIF inicial

publCIF

Puede considerarse un template (plantilla) para preparar un manuscrito para ser enviado a las revistas de la IUCr

publCIF - C:/Users/Flor/Desktop/TOPIC_/Matrial/Ejemplo_Seba/exp_3.cif (modified)

CIF Edit Preprint Tools Help

1 shelxl Acta C preprint Add refinement details

```
87  _atom_type_scat_dispersion_real
88  _atom_type_scat_dispersion_imag
89  _atom_type_scat_source
90  'C'  'C'   0.0030   0.0020
91  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
92  'H'  'H'   0.0000   0.0000
93  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
94  'N'  'N'   0.0060   0.0030
95  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
96  'O'  'O'   0.0110   0.0060
97  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
98  'S'  'S'   0.1250   0.1230
99  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
100
101 _symmetry_cell_setting            ?
102 _symmetry_space_group_name_H-M    ?
103
104 loop_
105  _symmetry_equiv_pos_as_xyz
106  'x, y, z'
107  '-x, y+1/2, -z+1/2'
108  '-x, -y, -z'
109  'x, -y-1/2, z-1/2'
110
111 _cell_length_a                    9.766
112 _cell_length_b                    12.545
113 _cell_length_c                    6.808
114 _cell_angle_alpha                 90.00
115 _cell_angle_beta                  102.33
116 _cell_angle_gamma                 90.00
117 _cell_volume                      814.7
118 _cell_formula_units_Z             4
119 _cell_measurement_temperature     293(2)
120 _cell_measurement_reflns_used     ?
121 _cell_measurement_theta_min       ?
122
```

1. Introduction

2. Experimental

2.1. Synthesis and crystallization

2.2. Refinement

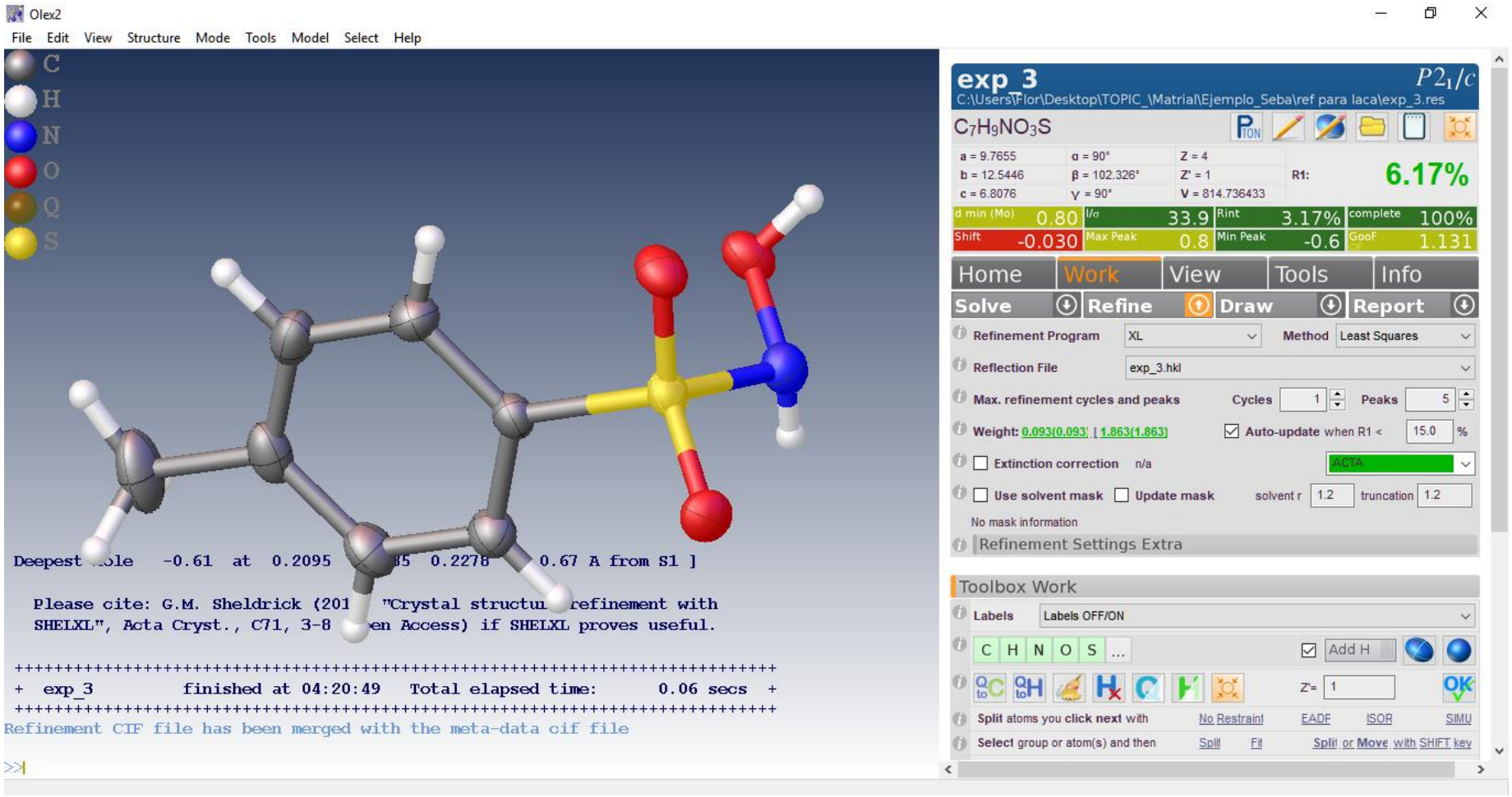
Crystal data, data collection and structure refinement details are summarized in Table 1.

3. Results and discussion

Col: 0 View log

Vista y edición del archivo CIF inicial

Olex2



Vista y edición del archivo CIF inicial

Olex2

exp_3 $P2_1/c$

C:\Users\Flor\Desktop\TOPIC_\Matrial\Ejemplo_Seba\ref para laca\exp_3.res

$C_7H_9NO_3S$

a = 9.7655	α = 90°	Z = 4	R1:	6.17%
b = 12.5446	β = 102.326°	Z' = 1		
c = 6.8076	γ = 90°	V = 814.736433		

d min (Mo)	0.80	I/σ	33.9	Rint	3.17%	complete	100%
Shift	-0.030	Max Peak	0.8	Min Peak	-0.6	GooF	1.131

Home | Work | View | Tools | Info

Solve | Refine | Draw | Report

exp_3 | Image: No Image | Make Report

Style: default.css | Begin with: default.htm | End with: templates/foc

Table label style: As in CIF

Collection

Crystal

Systematic Name: ?

Colour: ? ? ?

Size & Shape: ? ? ? ?

Preparation Details: ?

Crystallisation Details: ?

Crystal Mounting:

Diffraction

Absorption Correction

Definition File: ?

Diffraction T (K): ?

Cell Measurement T (K): ?

Special Details: ?

Refine Special Details: ?

Absorption Correction

Publication

Citations

Reference

Source Files

EditCifInfo | Merge Cif | HKL/RES: Leave as is

Checkcif Report | pdf | Send FCF | CCDC Number

Merge CIF: metacif | Add local | default CIF

Merge metacif items even if marked for skipping

Toolbox Work

Labels: Labels OFF/ON

C H N O S ... | Add H

Z'= 1

Split atoms you click next with: No Restraint | EADP | ISOR | SIMU

Select group or atom(s) and then: Split | Fit | Split or Move with SHIFT key

Select group or atom(s) and then: FIT SAME RIGU

Electron Density Map

Peak & Uiso Sliders

Vista y edición del archivo CIF inicial

Olex2

exp_3 $P2_1/c$

C:\Users\Flor\Desktop\TOPIC_\Matrial\Ejemplo_Seba\ref para laca\exp_3.res

$C_7H_9NO_3S$

a = 9.7655	α = 90°	Z = 4	R1:	6.17%
b = 12.5446	β = 102.326°	Z' = 1		
c = 6.8076	γ = 90°	V = 814.736433		

d min (Mo)	0.80	I/σ	33.9	Rint	3.17%	complete	100%
Shift	-0.030	Max Peak	0.8	Min Peak	-0.6	GooF	1.131

Home | Work | View | Tools | Info

Solve | Refine | Draw | Report

exp_3 | Image: No Image | Make Re

Style: default.css | Begin with: default.htm | End with

Table label style: As in CIF

Collection

Crystal

Systematic Name: ?

Colour: ? ? ?

Size & Shape: ? ? ? ?

Preparation Details: ?

Crystallisation Details: ?

Crystal Mounting:

Diffraction

Absorption Correction

Definition File: ?

Diffraction T (K): ?

Cell Measurement T (K): ?

Special Details: ?

Refine Special Details: ?

Absorption Correction

Publication

Citations

Reference

Source Files

EditCifInfo | Merge Cif | HKL/RES: Leave as is

Checkcif Report | pdf | Send FCF | CCDC Number

Merge CIF: metacif | Add local | default CIF

Merge metacif items even if marked for skipping

Toolbox Work

Labels: Labels OFF/ON

C H N O S ... | Add H

Z'= 1

Split atoms you click next with: No Restraint | EADP | ISOR | SIMU

Select group or atom(s) and then: Split | Fit | Split or Move with SHIFT key

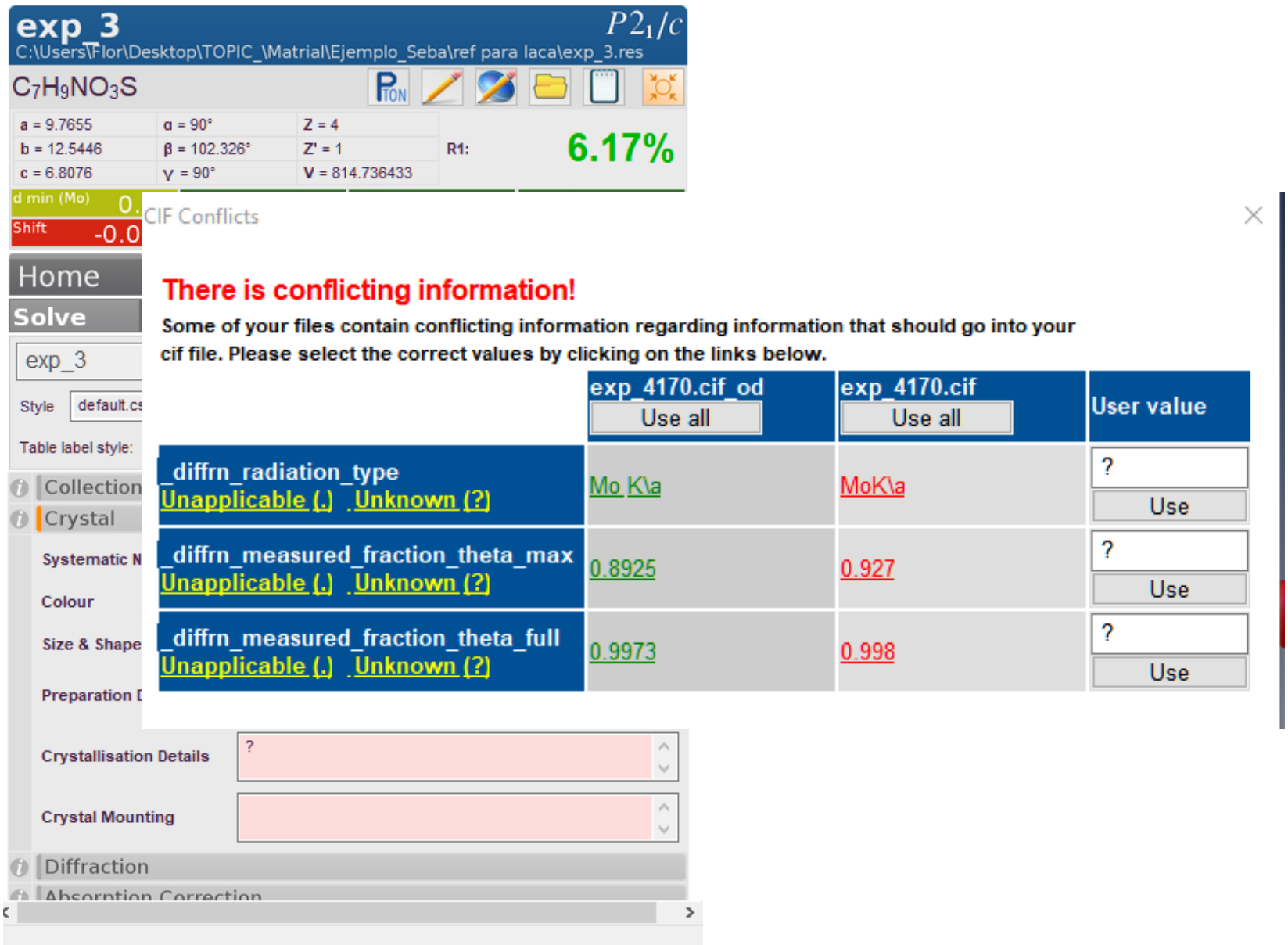
Select group or atom(s) and then: FIT SAME RIGU

Electron Density Map

Peak & Uiso Sliders

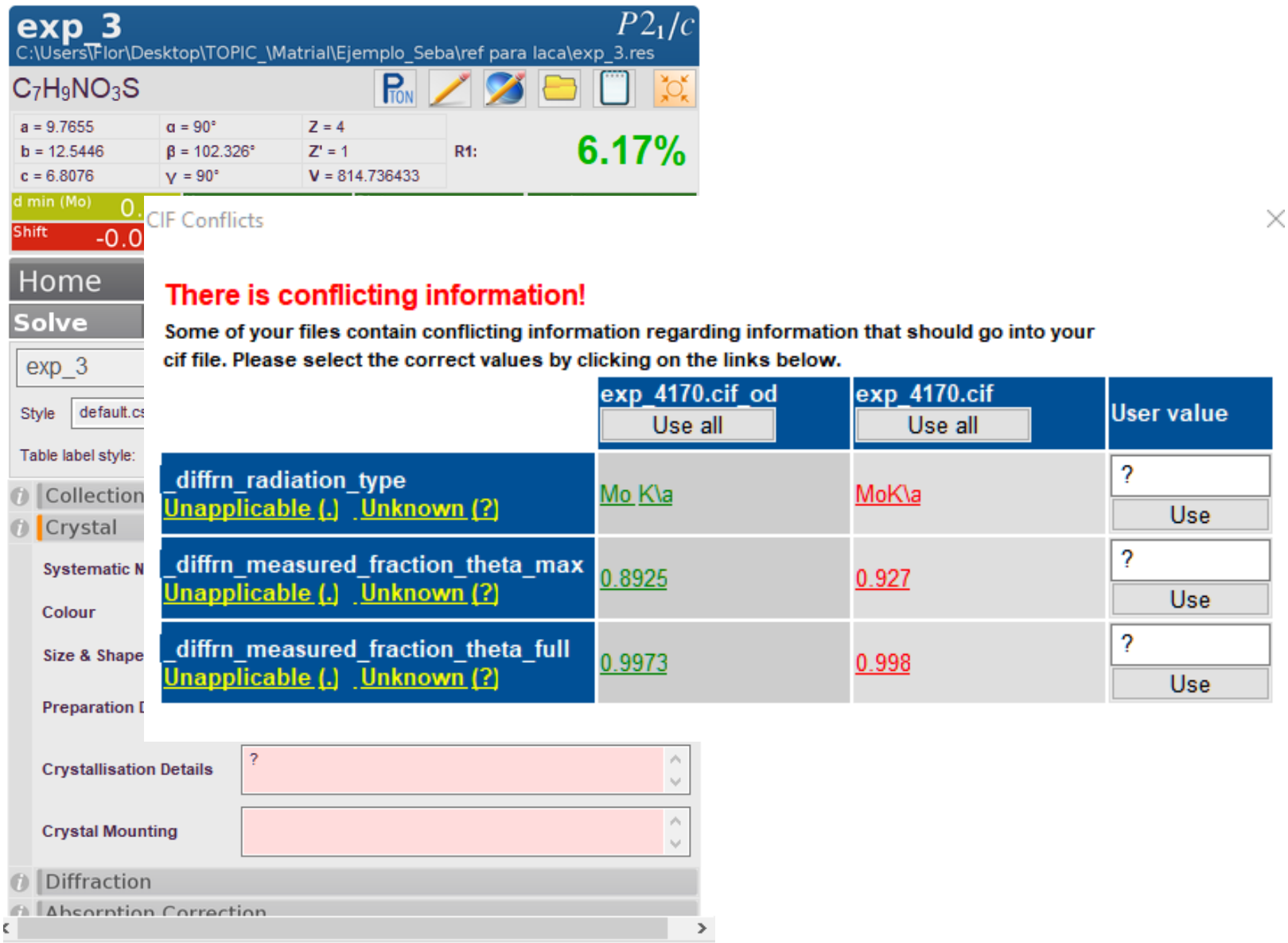
Vista y edición del archivo CIF inicial

Olex2



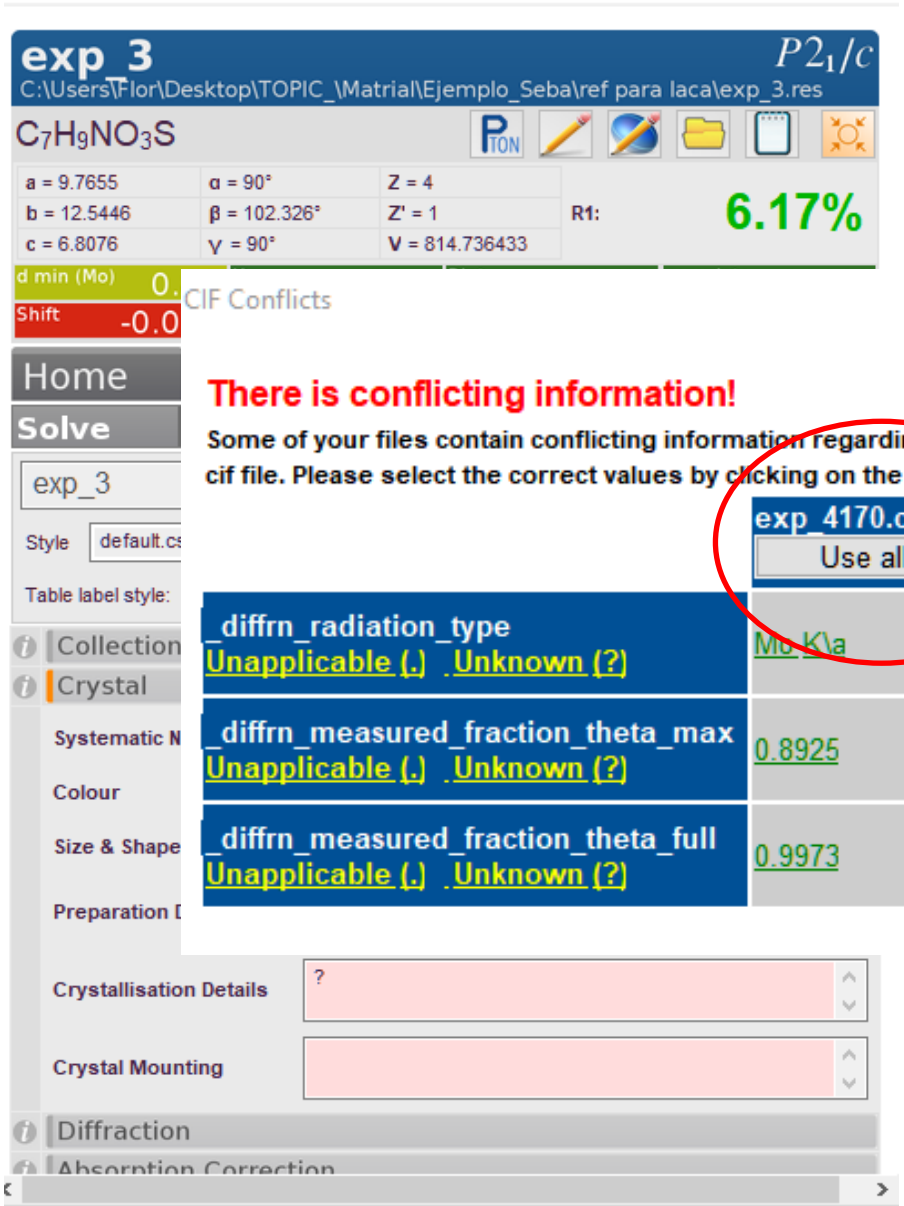
Vista y edición del archivo CIF inicial

Olex2



Vista y edición del archivo CIF inicial

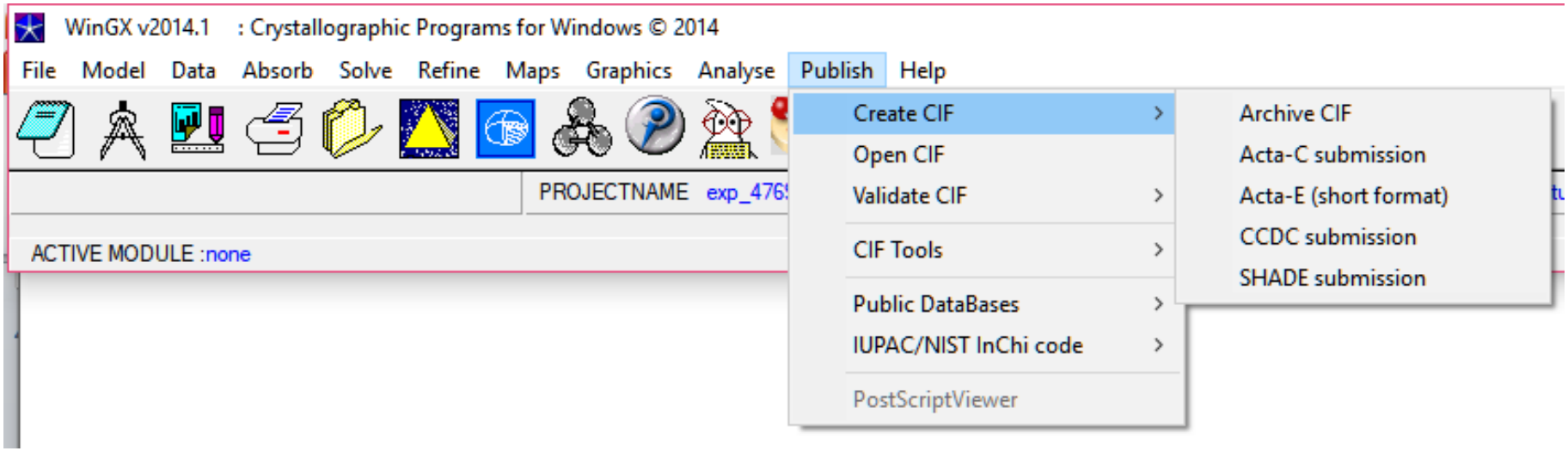
Olex2

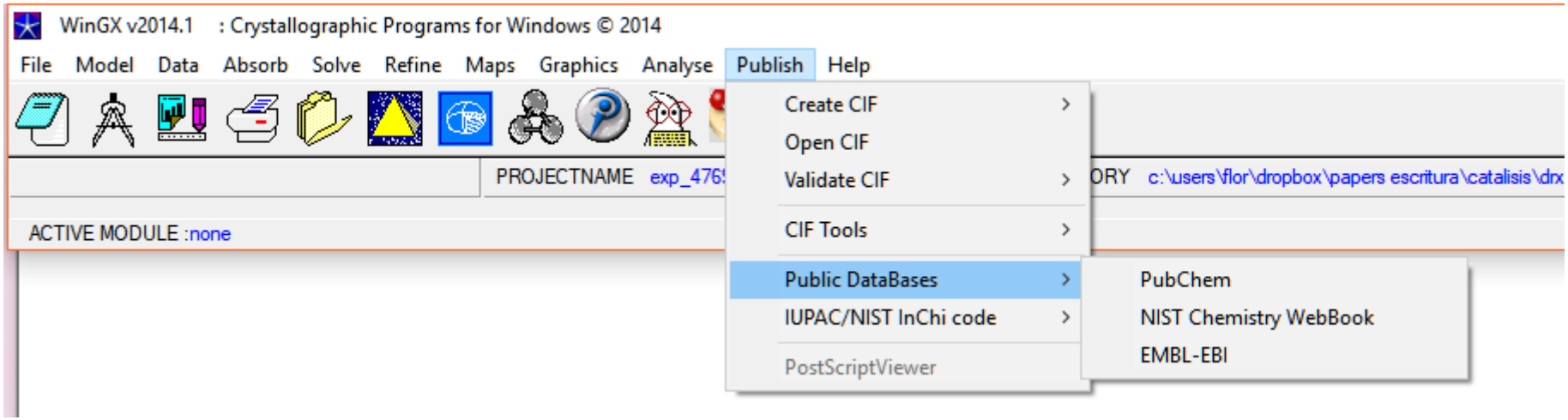


Cif_od. Archivo tipo .txt OBTENIDO DE EXPERIMENTO con DRX Gemini de OXFORD DIFFRACTION. Contiene información relacionada al equipo y la media (reflexiones para determinación de celda, parámetros de la corrección por absorción, T, etc

El equivalente para el equipo de BRUKER es: .pfc

Vista y edición del archivo CIF inicial

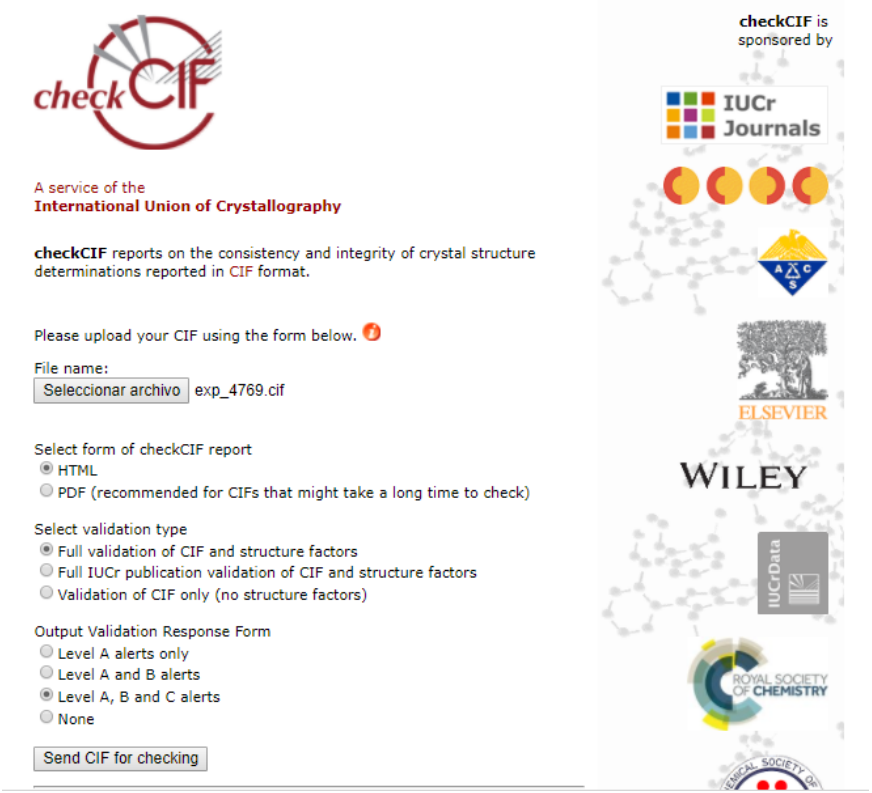


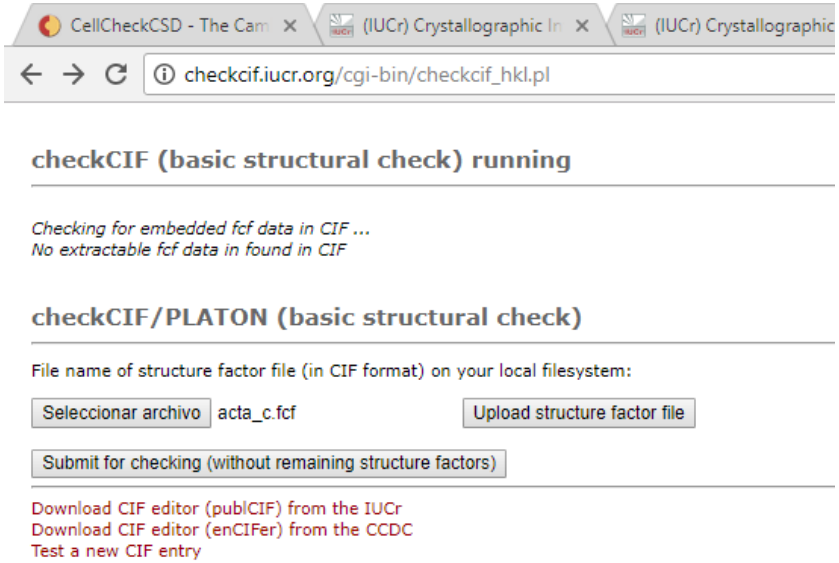


Validación del archivo CIF completo

=> **UNA VEZ COMPLETO el archivo CIF, volver al checkcif y ANALIZARLO.** Y si fuera necesario, corregir los nuevos errores o los no resueltos, hasta que los **ALERAS A, B y recomendablemente los C, sean corregidos o eventualmente justificados.**

https://checkcif.iucr.org/





Ejemplo

CellCheckCSD | (IUCr) Crystallo | (IUCr) Crystallo | checkCIF/Pl

checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

Structure factors have been supplied for datablock(s) exp

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PU
EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found.
Please wait while processing ....

CIF dictionary
Interpreting this report

Structure factor report

Datablock: exp

Bond precision: C-C = 0.0033 A Wavelength=0.71073

Cell: a=9.4254(6) b=11.6209(4) c=12.6080(5)
alpha=88.708(3) beta=68.696(5) gamma=86.313(4)

Temperature: 293 K

	Calculated	Reported
Volume	1283.94(11)	1283.95(12)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C30 H33 N3 O4	C30 H33 N3 O4
Sum formula	C30 H33 N3 O4	C30 H33 N3 O4
Mr	499.59	499.59
Dx,g cm-3	1.292	1.292
Z	2	2
Mu (mm-1)	0.086	0.086
F000	532.0	532.0
F000'	532.23	
h,k,lmax	12,15,17	12,15,17
Nref	6928	6120
Tmin,Tmax		
Tmin'		

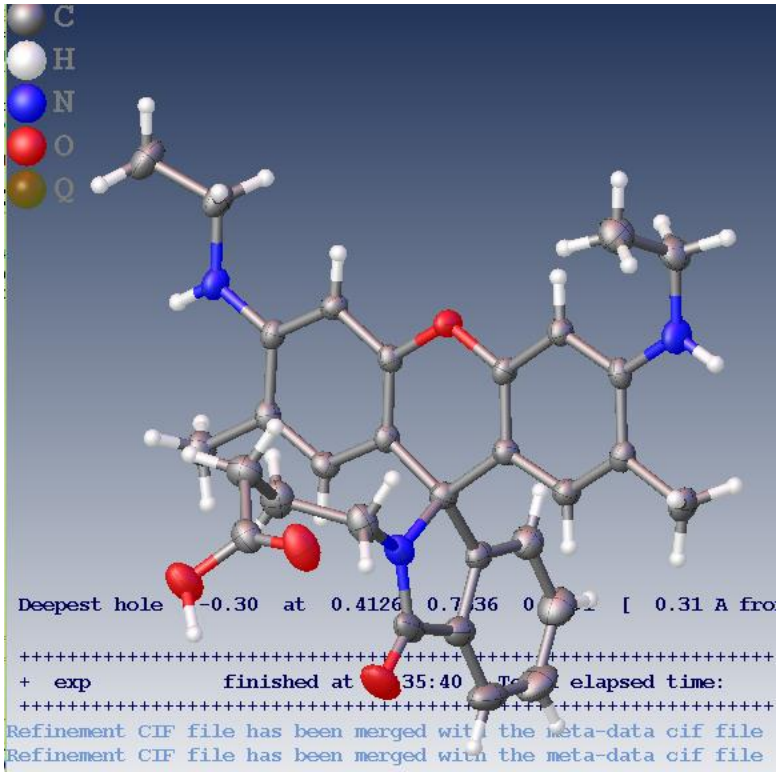
Correction method= Not given

Data completeness= 0.883 Theta(max)= 29.160

R(reflections)= 0.0602(3882) wR2(reflections)= 0.1600(6120)

S = 1.060 Npar= 342

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.



Ejemplo

CellCheckCSD | (IUCr) Crystallo | (IUCr) Crystallo | checkCIF/PLA

checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

Alert level A

EXPT005_ALERT_1_A _exptl_crystal_description is missing
Crystal habit description.
The following tests will not be performed.
CRYSR_01
DIFF003_ALERT_1_A _diffrn_measurement_device_type is missing
Diffractometer make and type. Replaces _diffrn_measurement_type.
PLAT183_ALERT_1_A Missing _cell_measurement_reflns_used Value .... Please Do !
PLAT184_ALERT_1_A Missing _cell_measurement_theta_min Value ..... Please Do !
PLAT185_ALERT_1_A Missing _cell_measurement_theta_max Value ..... Please Do !

Alert level B

PLAT355_ALERT_3_B Long O-H (X0.82,N0.98A) O16 - H16 . 1.12 Ang.
PLAT910_ALERT_3_B Missing # of FCF Reflection(s) Below Theta(Min). 14 Note

Alert level C

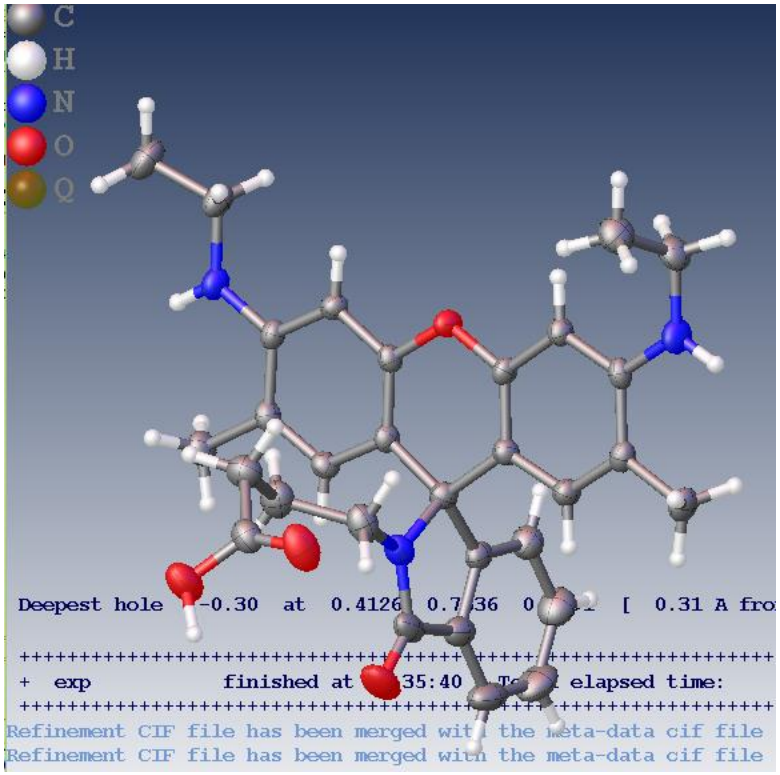
PLAT052_ALERT_1_C Info on Absorption Correction Method Not Given Please Do !
PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ... Please Check
PLAT054_ALERT_1_C Medium Crystal Dimension Missing (or Error) ... Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ... Please Check
PLAT222_ALERT_3_C Non-Solv. Resd 1 H Uiso(max)/Uiso(min) Range 4.9 Ratio
PLAT230_ALERT_2_C Hirshfeld Test Diff for O21 --C25 . 5.3 s.u.
PLAT230_ALERT_2_C Hirshfeld Test Diff for C25 --C27 . 5.2 s.u.
PLAT420_ALERT_2_C D-H Without Acceptor N7 --H7 Please Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 3.607 Check

Alert level G

PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 2 Report
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K) 293 Check
PLAT200_ALERT_1_G Reported _diffrn_ambient_temperature ..... (K) 293 Check
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 795 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 8 Info

5 ALERT level A = Most likely a serious problem - resolve or explain
2 ALERT level B = A potentially serious problem, consider carefully
9 ALERT level C = Check. Ensure it is not caused by an omission or oversight
5 ALERT level G = General information/check it is not something unexpected

11 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
4 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
1 ALERT type 4 Improvement, methodology, query or suggestion



Ejemplo

CellCheckCSD | (IUCr) Crystallo | (IUCr) Crystallo | checkCIF/PLA

checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

Alert level A

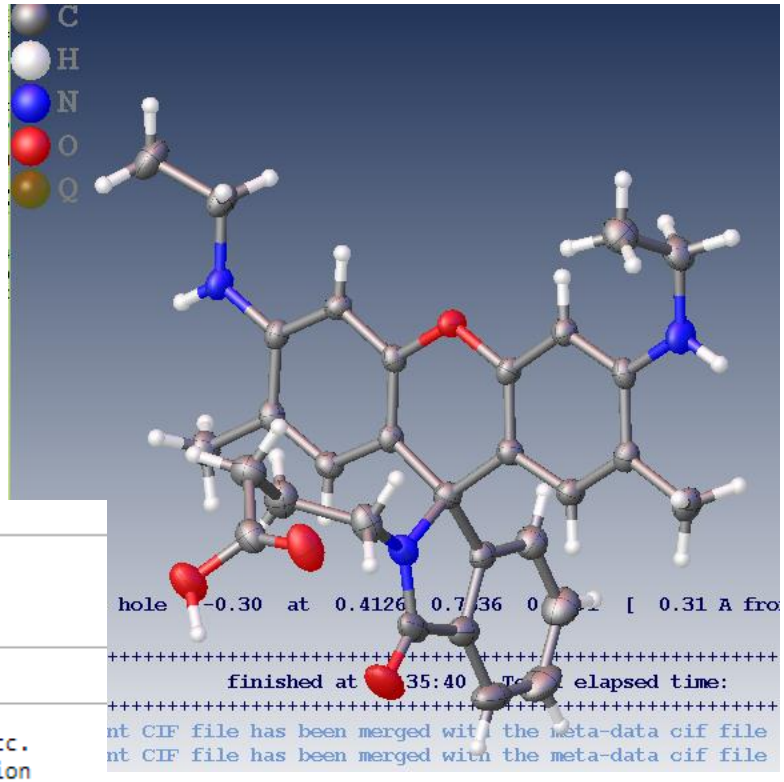
EXPT005_ALERT_1_A _exptl_crystal_description is missing
Crystal habit description.
The following tests will not be performed.
CRYSR_01
DIFF003_ALERT_1_A _diffrn_measurement_device_type is missing
Diffractometer make and type. Replaces _diffrn_measurement_type.
PLAT183_ALERT_1_A Missing _cell_measurement_reflns_used Value .... Please Do !
PLAT184_ALERT_1_A Missing _cell_measurement_theta_min Value ..... Please Do !
PLAT185_ALERT_1_A Missing _cell_measurement_theta_max Value ..... Please Do !

Alert level B

PLAT355_ALERT_3_B Long O-H (X0.82,N0.98A) O16 - H16 . 1.12 Ang.
PLAT910_ALERT_3_B Missing # of FCF Reflection(s) Below Theta(Min). 14 Note

Alert level C

PLAT052_ALERT_1_C Info on Absorption Correction Method Not Given Please Do !
PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ... Please Check
PLAT054_ALERT_1_C Medium Crystal Dimension Missing (or Error) ... Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ... Please Check
PLAT222_ALERT_3_C Non-Solv. Resd 1 H Uiso(max)/Uiso(min) Range 4.9 Ratio
PLAT230_ALERT_2_C Hirshfeld Test Diff for O21 --C25 . 5.3 s.u.



PLAT910

PLAT910 Type_3 Test for missing reflections below Theta-Min

Possible causes: Beamstop theta-min limit set too high, large unit-cell etc.
A possible technical solution on CCD based equipment involves the collection
of additional images with the detector at a larger distance from the
crystal with the beamstop setting changed accordingly..

11 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
4 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
1 ALERT type 4 Improvement, methodology, query or suggestion

Ejemplo

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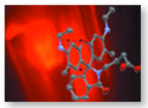
ACCESS



Two rhodamine 6G derivative compounds: a structural and fluorescence single-crystal study

M. Di Paolo, M. L. Bossi, R. Baggio and S. A. Suarez

The synthesis, characterization, structural analysis and fluorescence properties of two rhodamine 6G derivatives are described, namely a propargylamine derivative, 3′,6′-bis(ethylamino)-2′,7′-dimethyl-2-(methylcyanide)spiro[isoindole-1,9′-xanthen]-3(2*H*)-one (I), and a γ-aminobutyric acid (GABA) derivative, 3′,6′-bis(ethylamino)-2′,7′-dimethyl-3-oxospiro[isoindole-1,9′-xanthen]-2(3*H*)-yl)butyricacid (II). Both structures are compared with four similar ones from the Cambridge Structural Database (CSD), and the interactions involved in the stabilization are analyzed using the atoms in molecules (AIM) theory. Finally, a single-crystal *in-situ* reaction study is presented, carried out by fluorescence methods, which enabled the `opening' of the spirolactam ring in the solid phase.

Keywords: **rhodamine 6G derivatives**; **photocrystallography**; **fluorescence**; **weak interactions**; **atoms in molecules theory**.

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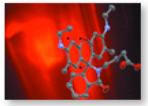

Two rhodamine 6G derivative compounds: a structural and fluorescence sing

M. Di Paolo, M. L. Bossi, R. Baggio and S. A. Suarez

The synthesis, characterization, structural analysis and fluorescence properties of two rhodamine 6G d
propargylamine derivative, 3′,6′-bis(ethylamino)-2′,7′-dimethyl-2-(methylcyanide)spiro[isoindole-1,9′
aminobutyric acid (GABA) derivative, 3′,6′-bis(ethylamino)-2′,7′-dimethyl-3-oxospiro[isoindole-1,9′-xa
structures are compared with four similar ones from the Cambridge Structural Database (CSD), and th
using the atoms in molecules (AIM) theory. Finally, a single-crystal *in-situ* reaction study is presented,
`opening' of the spirolactam ring in the solid phase.

Keywords: **rhodamine 6G derivatives**; **photocrystallography**; **fluorescence**; **weak interaction**

Read article Similar articles

Datablock: II

```
Bond precision:          C-C = 0.0033 A                    Wavelength=0.71073
Cell:        a=9.4250(6)        b=11.6209(4)       c=12.6080(5)
             alpha=88.708(3)    beta=68.696(5)     gamma=86.313(4)
Temperature: 170 K
                         Calculated                          Reported
Volume                   1283.89(11)                         1283.89(11)
Space group              P -1                                P -1
Hall group               -P 1                                -P 1
Moiety formula           C30 H33 N3 O4                       C30 H33 N3 O4
Sum formula              C30 H33 N3 O4                       C30 H33 N3 O4
Mr                       499.59                              499.59
Dx,g cm-3                1.292                               1.292
Z                        2                                   2
Mu (mm-1)                0.086                               0.086
F000                     532.0                               532.0
F000'                    532.23
h,k,lmax                 12,15,17                            12,15,17
Nref                     6929                                6120
Tmin,Tmax                0.980,0.983                         0.970,0.990
Tmin'                    0.975
Correction method= # Reported T Limits: Tmin=0.970 Tmax=0.990 AbsCorr =
MULTI-SCAN
Data completeness= 0.883              Theta(max)= 29.161
R(reflections)= 0.0598( 3882)             wR2(reflections)= 0.1712( 6120)
S = 1.037                  Npar= 350
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT355_ALERT_3_B Long O-H (X0.82,N0.98A) O2X - H2X . 1.09 Ang.

Alert level C

PLAT222_ALERT_3_C Non-Solv. Resd 1 H Uiso(max)/Uiso(min) Range 4.8 Ratio
PLAT230_ALERT_2_C Hirshfeld Test Diff for O2 --C26 . 5.7 s.u.
And 2 other PLAT230 Alerts
More ...
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 3.098 Check

Alert level G

PLAT013_ALERT_1_G N.O.K. _shelx_hkl_checksum Found in CIF ..... Please Check
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 1 Report
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 6 Note
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 2 Note
PLAT882_ALERT_1_G No Datum for _diffrn_reflns_av_unetI/netI ..... Please Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 795 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 6 Info

0 **ALERT level A** = Most likely a serious problem - resolve or explain
2 **ALERT level B** = A potentially serious problem, consider carefully

Archivo CIF2

- El archivo CIF2 es un nuevo formato de archivo alternativo para ser utilizado por la comunidad de cristalógrafos, en reemplazo del anterior archivo CIF (“CIF1”) desarrollado en los 90.

- El archivo CIF2 es un nuevo formato de archivo alternativo para ser utilizado por la comunidad de cristalógrafos, en reemplazo del anterior archivo CIF (“CIF1”) desarrollado en los 90.

- En el formato del archivo CIF2 se introduce la posibilidad de utilizar caracteres de código único (Unicode) y extensiones para tratamiento de datos complejos. Esta característica hace que sea más “amigable”

- Los detalles del archivo CIF2 se describen en: .). "*Specification of the Crystallographic Information File format, version 2.0*“. Bernstein, H. J., Bollinger, J. C., Brown, I. D., Gražulis, S., Hester, J. R., McMahon, B., Spadaccini, N., Westbrook, J. D. & Westrip, S. P *J. Appl. Cryst.* **2016,** *49.*

- Para detalles de compatibilidad ir a: https://www.iucr.org/resources/cif/cif2

Comentarios finales

- El archivo CIF permite tener un seguimiento de los datos estructurales de las estructuras publicadas
- Bajo la base que los datos son correctos, se puede utilizar esa información en nuestra tarea
- No subestimar la etapa de “armado del archivo CIF”.