EXPO in Rigaku software

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- Rigaku Powder XRD software overview
- Application example

  Structure determination for a sample including impurities
- Structure refinement and validation
- SmartLab Studio II, Powder XRD plugin demo

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SmartLab Studio (-2016)

Bridge software which launches individual executables listed below.

- PDXL
  - Powder diffraction
- GlobalFit
  - XRR
  - High resolution rocking curve
- Nano-Solver (SAXS)
- 3D Explore
  - RSM & PF
- 2DP

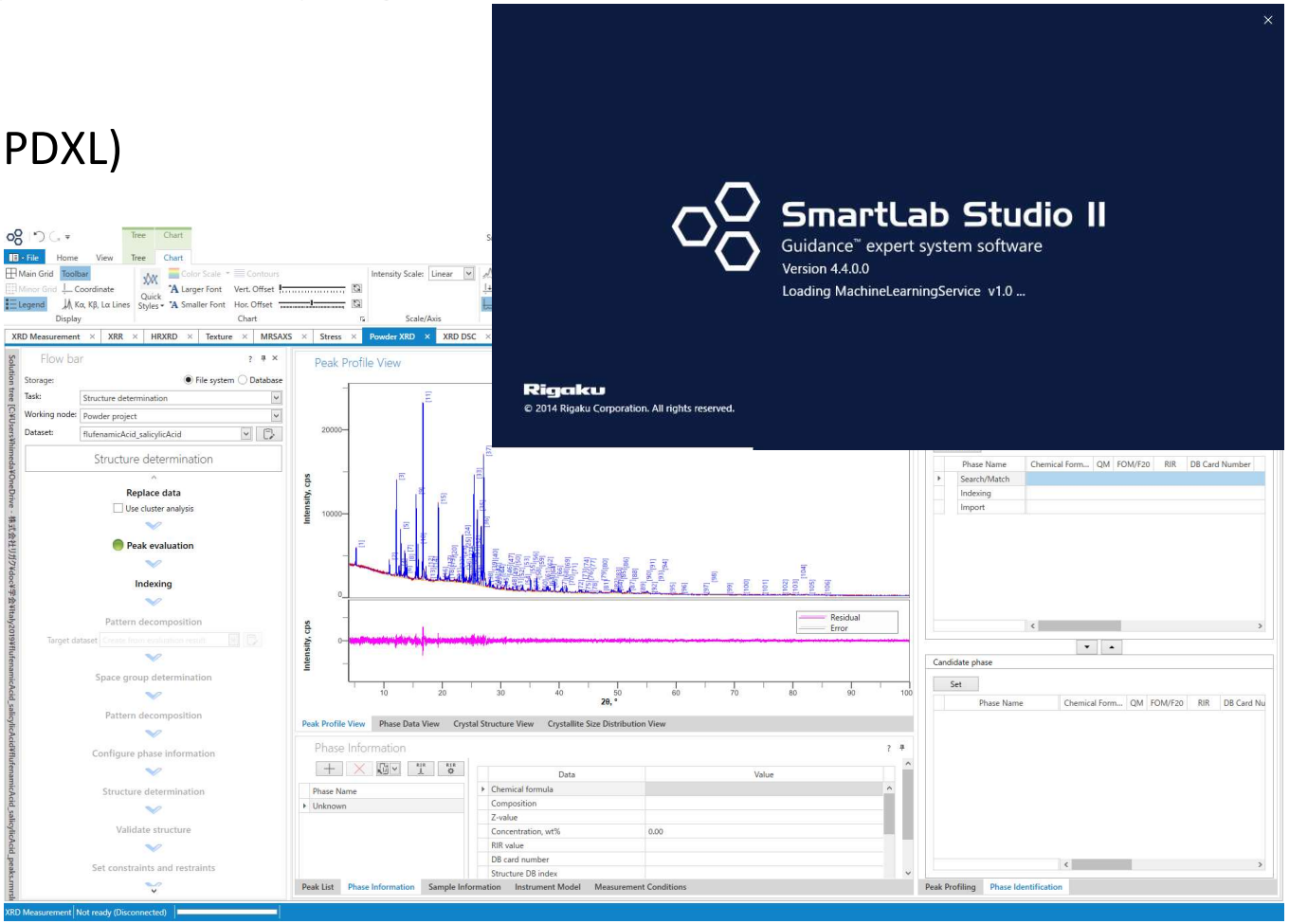
On this system, multiple executables should be launched and closed by users.

SmartLab Studio II (2016-)

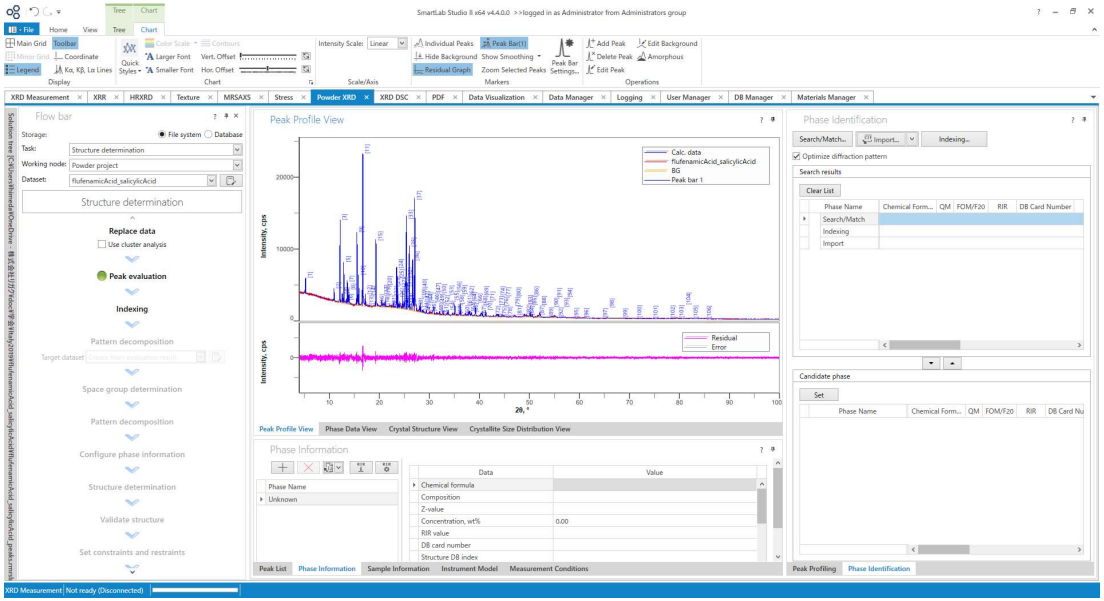
Integrated platform including measurement and analyses for X-ray.

Following functionality are provided as plugins.

- XRD Measurement
- Powder XRD (successor to PDXL)
- XRR (reflectivity)
- HRXRD
  - High resolution rocking curve & RSM
- Stress
- Texture
  - Pole figure & ODF
- SAXS



Powder XRD plugin – successor to PDXL



- Automatic peak processing
- Integrated qualitative analysis
  - Search/Match
  - Indexing
  - CIF
- Quantitative analysis methods
  - RIR quantification
  - Quantification using calibration curve
  - Quantification using Rietveld method
- Size & Strain
  - Scherrer's method and conventional Williamson-Hall plot
  - Fundamental parameter method: individual peaks and whole pattern
- Structure determination
  - Charge flipping, Direct space methods (parallel tempering method as in FOX)

Powder XRD plugin – Support for academic software

- EXPO
  - Direct methods
  - Direct space methods (SA)
  - Space group determination
- Import / Export Whole powder pattern fitting results
  - GSAS
  - FullProf
  - FOX
  - Rietan-FP
- Indexing
  - DICVOL06
  - ITO
  - N-TREOR
- Crystallography Open Database

Powder XRD plugin – Supported commercial databases

- ICDD
  - Phase identification (Search/Match)
  - Structure information for Rietveld refinement.
- Cambridge database
  - Restraint settings for Rietveld refinement

Collaboration between EXPO and Rigaku

- Collaboration started on 2008 with EXPO2008
- PDXL Structure analysis package was released on Oct./2009 (PDXL ver. 1.5) together with EXPO2009.
- PDXL ver. 2.0 was released on 2011. Structure analysis guidance functionality was included.
- PDXL ver. 2.1 was released on 2013 together with EXPO2011.
- Powder XRD plugin Structure determination package was released on June/2018 together with EXPO2014.

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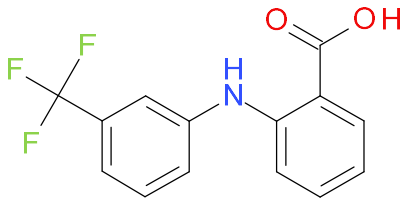
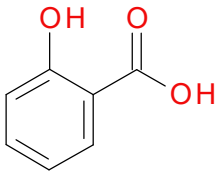
Co-crystal of flufenamic acid and salicylic acid

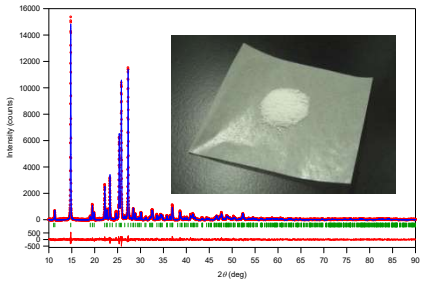


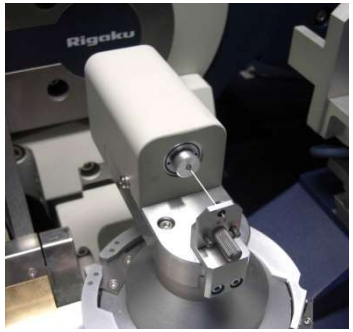


- Collaborative research with Chugai pharmaceutical company

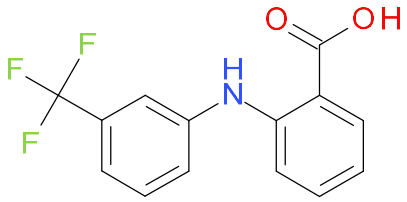
CHUGAI PHARMACEUTICAL CO., LTD.
Roche Group

- Measurement was performed on Rigaku SmartLab
- Sample was filled in a capillary and was rotated on the capillary spinner during measurement



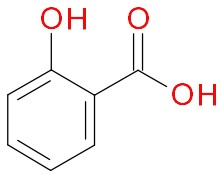


SmartLab
X-RAY DIFFRACTOMETER



Flufenamic acid

+

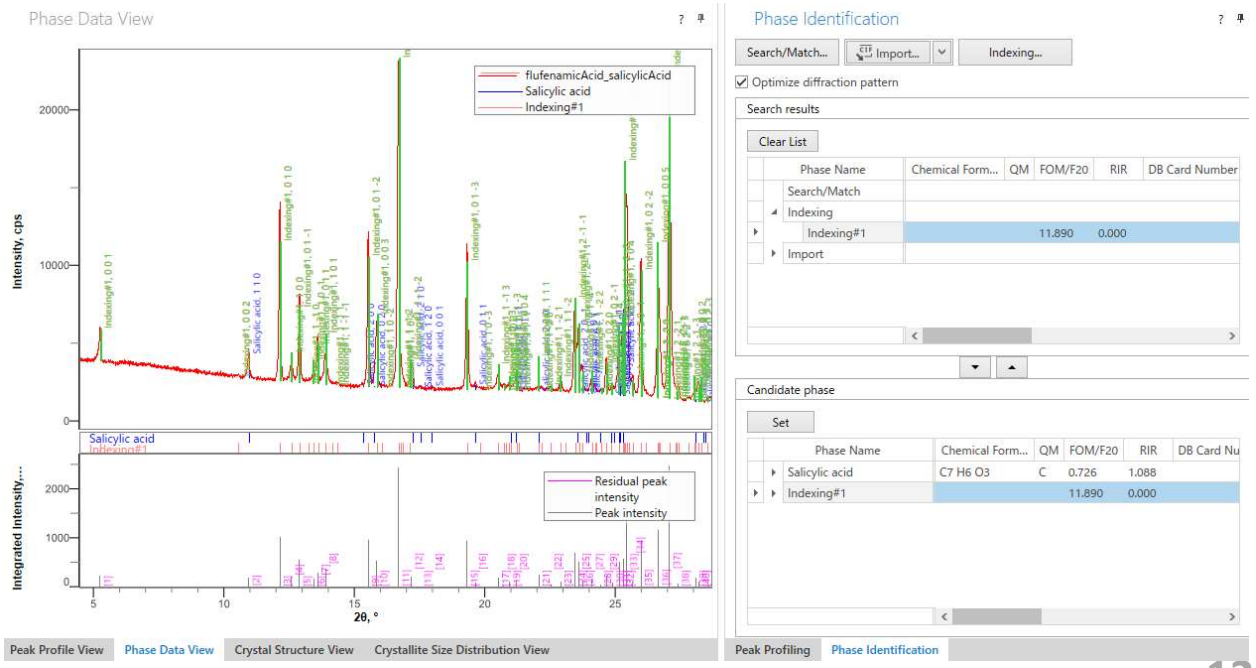


Salicylic acid

→

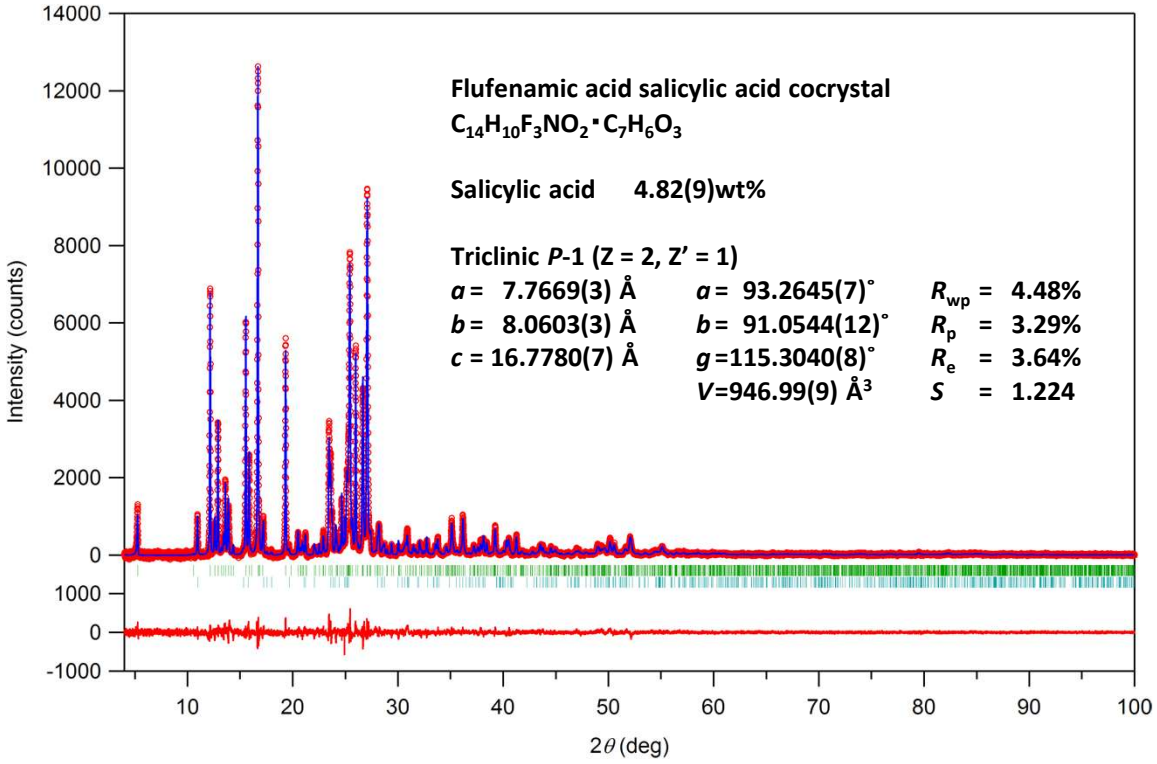
Flufenamic acid salicylic acid cocrystal

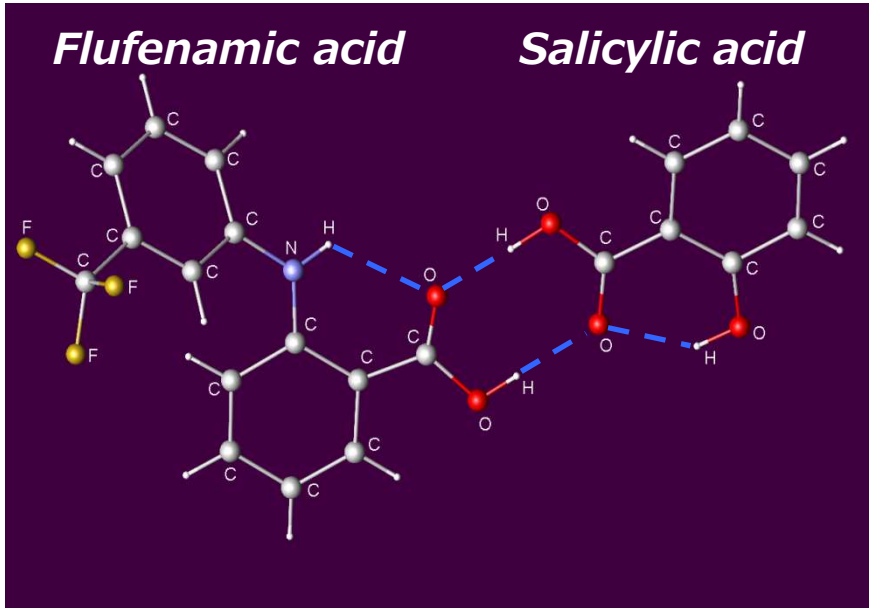
- First attempt of indexing for cocrystal was failed
- Components (API* or co-former) were suspected to be included => tried to import their CIF files and found salicylic acid was contained
- Indexing was succeeded by removing impurity peaks.



*API: active pharmaceutical ingredient

- Powder XRD plugin generates single phase profile by removing theoretical profile from Salicylic acid.
- We can successfully determine the cocrystal structure using EXPO SA algorithm.





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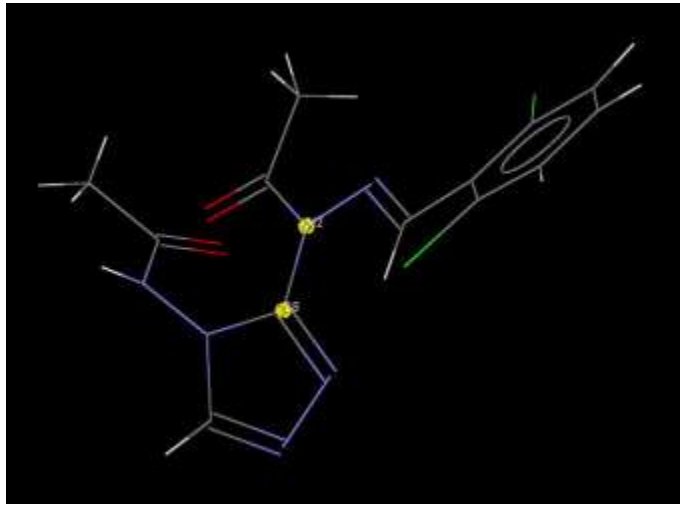
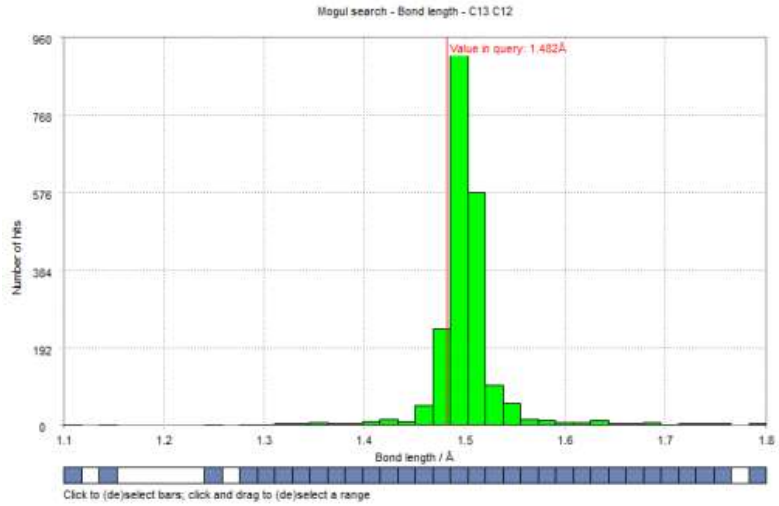
Structure refinement

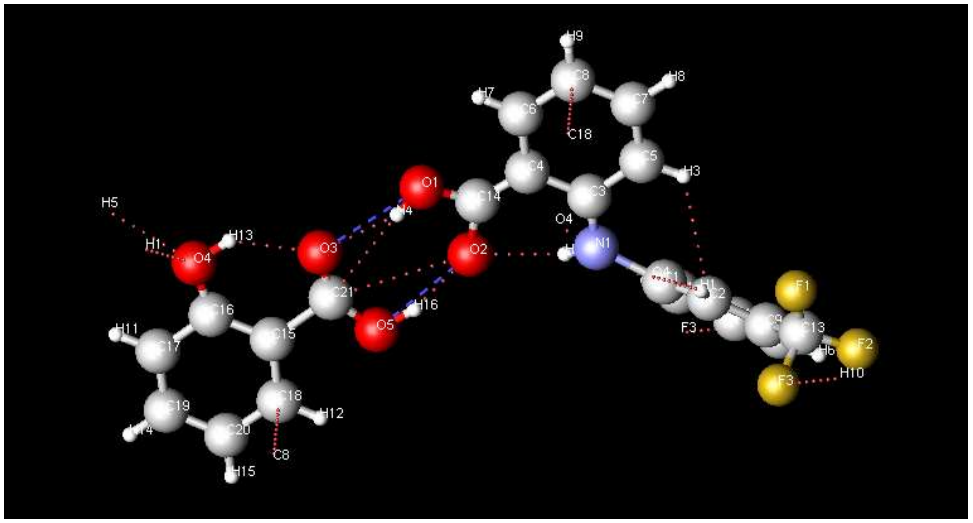
- Structure refinement with Powder diffraction needs to apply restraints to keep its structure reasonable.
  -> Reasonable partial structures are required.
- Mogul software from CCDC [Cambridge Crystallographic Data Centre] provides statistics for similar partial structures.
  - > It can be used for the target values and their restraint weight of bond distances and angles.



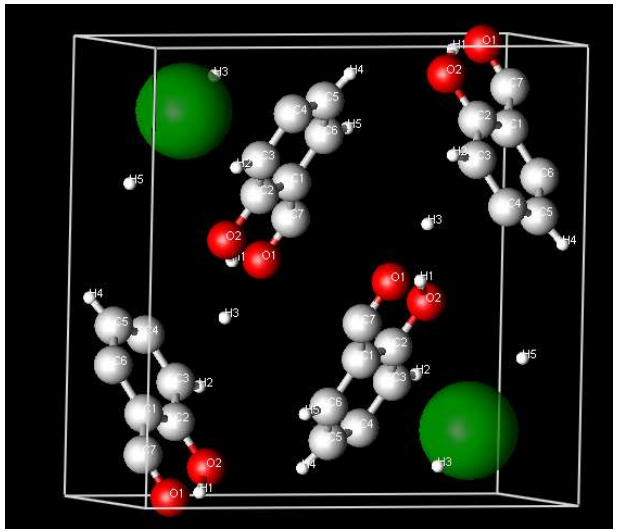


Structure validation

- Hydrogen bonds and Short contacts

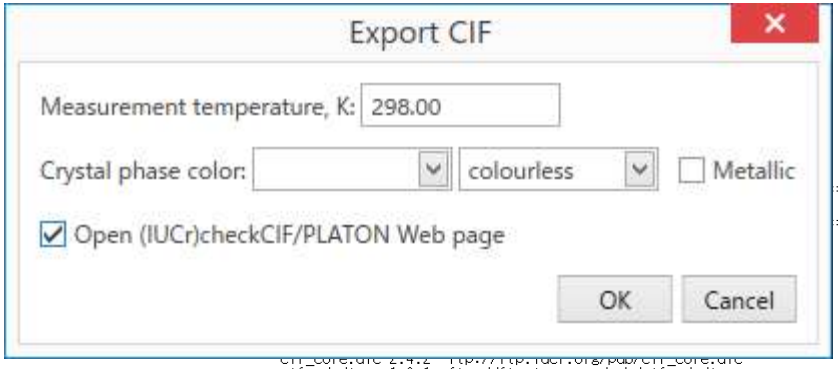


- Void display empty space where one hydrogen can be included



Structure validation

- CIF export and launch checkCIF web page.

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# PROCESSING SUMMARY (IUCr Office Use Only)

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_journal_date_accepted            ?
_journal_coeditor_code            ?

#===============================================================================
# SUBMISSION DETAILS

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_publ_contact_author_address
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    ENTER ADDRESS
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_publ_contact_author_fax          'ENTER FAX NUMBER'
_publ_contact_author_email        'ENTER EMAIL ADDRESS'
_publ_contact_author_phone        'ENTER PHONE NUMBER'
_publ_contact_letter
;
    ENTER TEXT OF LETTER
;
_publ_requested_journal           'ENTER JOURNAL NAME HERE'
_publ_requested_category           ? # CHOOSE FA FI FO FM EI EO EM AD or SC
_publ_requested_coeditor_name      ?

#===============================================================================
# TITLE AND AUTHOR LIST

_publ_section_title
;
    ENTER SECTION TITLE
;
_publ_section_title_footnote
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checkCIF/PLATON (full publication check)

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report

Datablock: I

Bond precision: = 0.0000 A Wavelength=1.54059
Cell: a=7.7675(10) b=8.0610(11) c=16.779(2)
alpha=93.259l(10) beta=91.0681(15) gamma=115.3082(11)
Temperature: 298 K

	Calculated	Reported
Volume	947.2(2)	947.1(2)
Space group	P -1	P -1
Hall group	-P 1	?
Moiety formula	C14 H10 F3 N O2, C7 H6 O3	?
Sum formula	C21 H16 F3 N O5	C42 F6 H32 N2 O10
Mr	419.35	838.70
Dx,g cm-3	1.470	1.470
Z	2	1
Mu (mm-1)	1.074	0.000
F000	432.0	432.0
F000'	433.70	
h,k,lmax	7,8,16	0,0,0
Nref	1950	1950
Tmin,Tmax		
Tmin'		

Correction method= Not given
Data completeness= 1.000 Theta(max)= 50.000
R(reflections)= wR2(reflections)=
S = Npar=

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.

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Thank you for your attention!