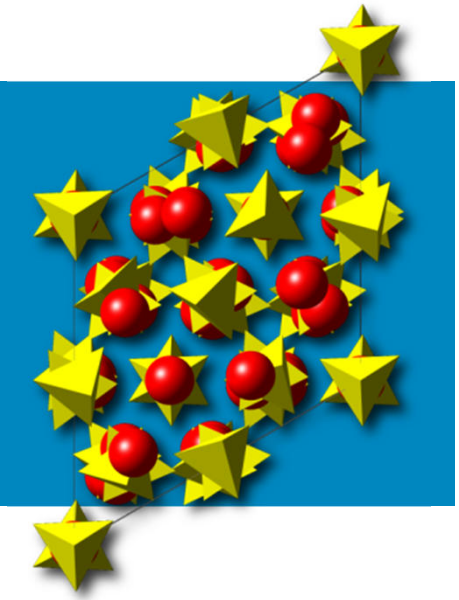
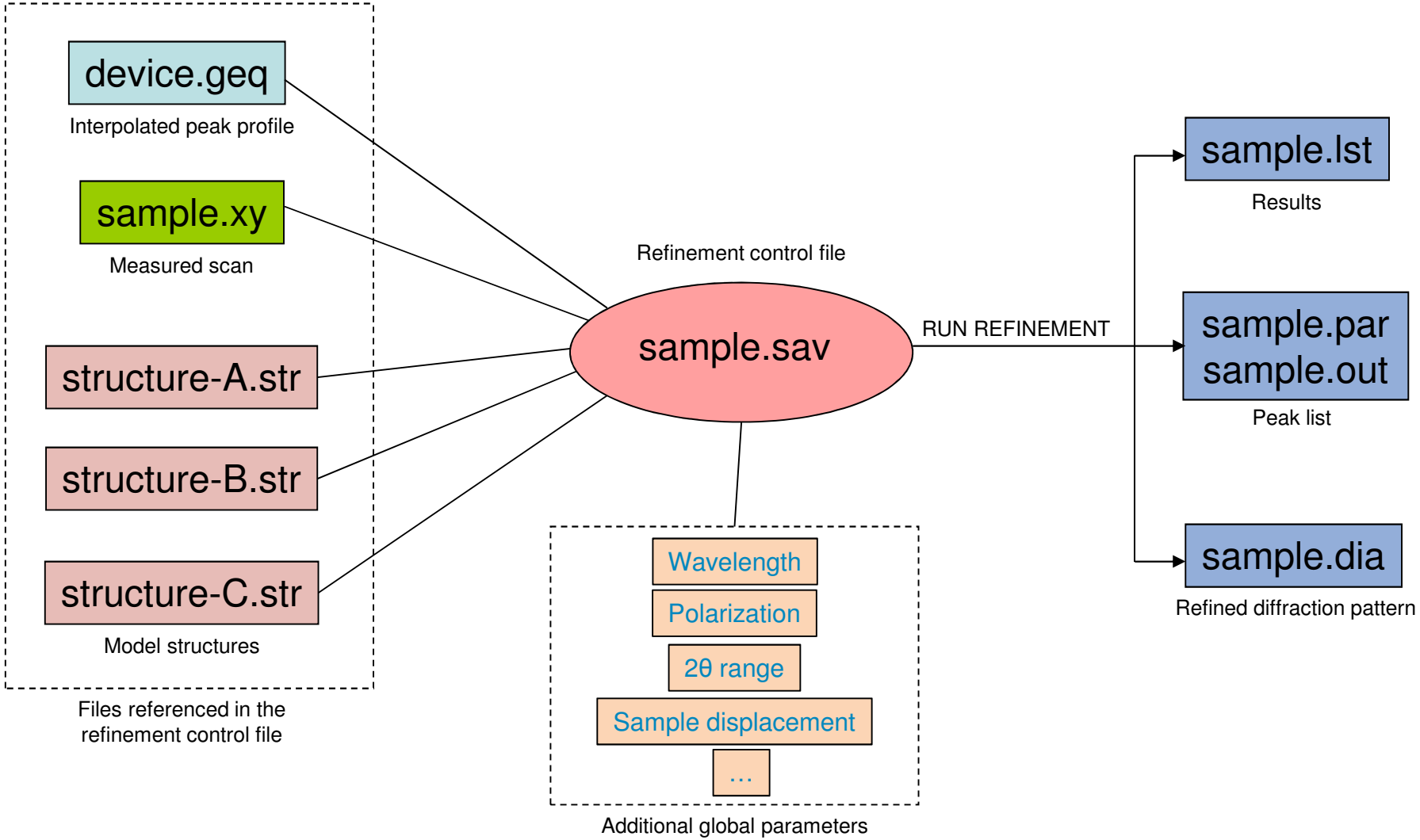
Lesson 8
Crystal Structure Files



Nicola Döbelin
RMS Foundation, Bettlach, Switzerland

Repetition: BGMN Project Structure



Structure Databases

- STR files shipped with Profex (created manually by the Profex developer)
- http://www.bgmn.de/download-structures.html (created manually by BGMN team)

- Create manually from:
  - ICSD (http://www.fiz-karlsruhe.de/icsd.html)
  - PDF-4+ (http://www.icdd.com/)

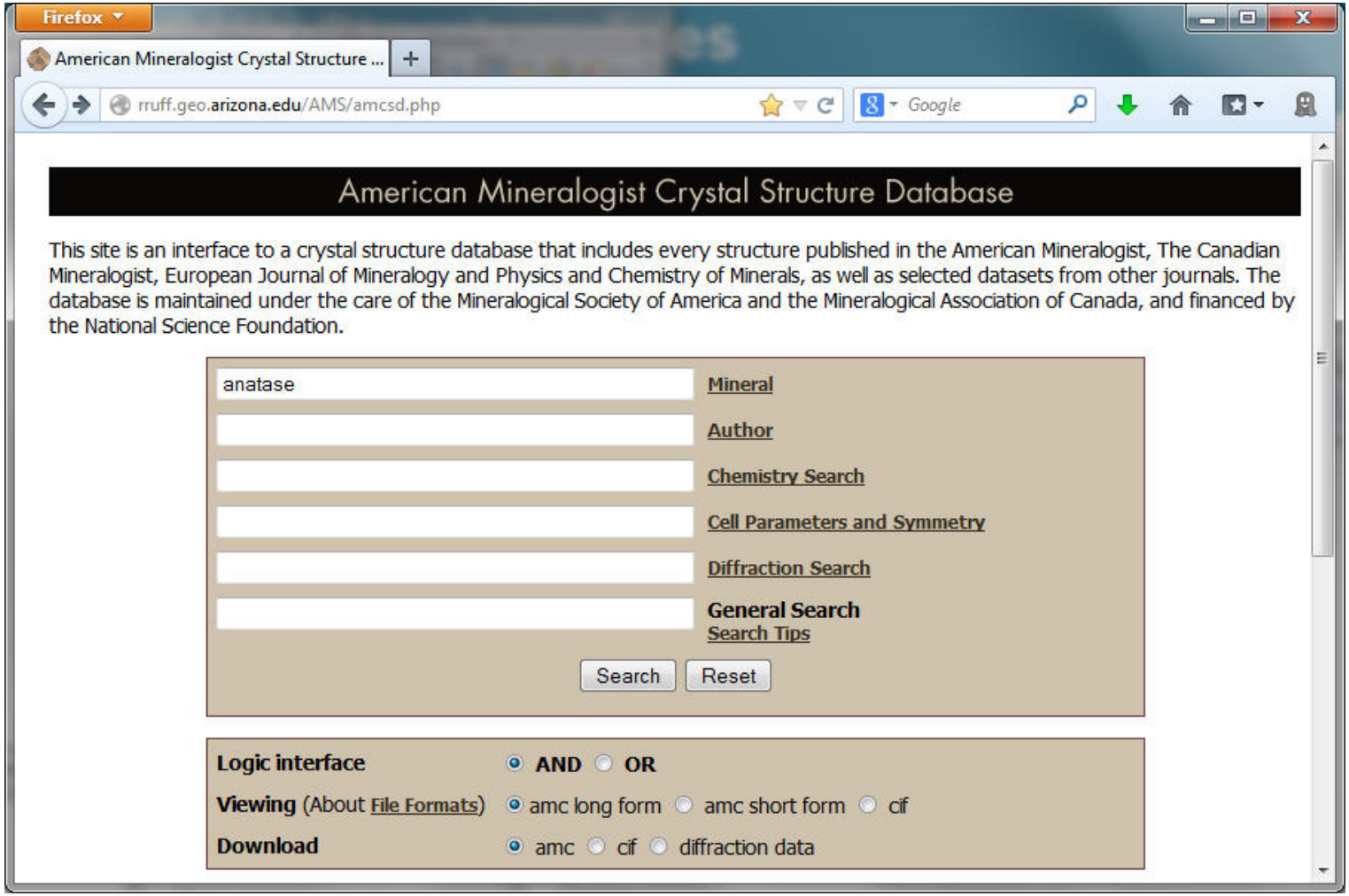
  Commercial

  - American Mineralogist Structure Database (http://rruff.geo.arizona.edu/AMS/amcsd.php)
  - Crystallography Open Database COD (http://www.crystallography.net/)

  Open Access

  - Cambridge Crystallographic Data Centre (http://beta-www.ccdc.cam.ac.uk/pages/Home.aspx)

AMCSD


Firefox
American Mineralogist Crystal Structure ...
rruff.geo.arizona.edu/AMS/amcsd.php
Google
American Mineralogist Crystal Structure Database
This site is an interface to a crystal structure database that includes every structure published in the American Mineralogist, The Canadian Mineralogist, European Journal of Mineralogy and Physics and Chemistry of Minerals, as well as selected datasets from other journals. The database is maintained under the care of the Mineralogical Society of America and the Mineralogical Association of Canada, and financed by the National Science Foundation.
anatase
Mineral
Author
Chemistry Search
Cell Parameters and Symmetry
Diffraction Search
General Search
Search Tips
Search
Reset
Logic interface
AND
OR
Viewing (About File Formats)
amc long form
amc short form
cif
Download
amc
cif
diffraction data

AMCSD: Retrieving CIF Files

Firefox

AMCSD Search Results

rruff.geo.arizona.edu/AMS/result.php

American Mineralogist Crystal Structure Database

7 matching records for this search.

Anatase

Howard C J, Sabine T M, Dickson F

Acta Crystallographica B47 (1991) 462-468

Structural and thermal parameters for rutile and anatase

Locality: synthetic

_database_code_amcsd 0019093

3.7845 3.7845 9.5143 90 90 90 I4_1/amd

atom	x	y	z	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Ti	0	0	0	.0052	0	0	.0052	0	.0070
O	0	0	.20806	.0117	0	0	.0027	0	.0072

Download AMC data (View Text File)

Download CIF data (View Text File)

Download diffraction data (View Text File)

View JMOL 3-D Structure

Watch out for high-temp and high-pressure datasets

Download CIF file

!

Anatase

Parker R

Zeitschrift fur Kristallographie 59 (1924) 1-54

Zur Kristallstruktur von Anastas und Rutil. (II. Teil. Die Anastasstruktur).

COD

Verbinden...

www.crystallography.net/search.html

Crystallography Open Database

Search

(For more information on search see the hints and tips)

COD Home

Home
What's new?

Accessing COD Data

Browse
Search
Search by structural formula

Add Your Data

Deposit your data
Manage depositions
Manage/release prepublications

Documentation

COD Wiki
Obtaining COD
Citing COD
COD Mirrors
Advices to donators
Useful links

Search by COD ID:	Search

OpenBabel FastSearch:	Enter SMILES: Search

Note: substructure search by SMILES is currently available in a subset of COD containing 70 000 structures.

text (1 or 2 words)	anatase
journal	
year	
volume	
issue	
Z (min, max)	
Z' (min, max)	
1 to 8 elements	Ti O
NOT these elements	
volume min and max	
number of distinct elements min and max	2 2
filters	has F_{obs}
Reset	Send

Warten auf www.crystallography.net...

COD: Retrieving CIF Files

Crystallography Open Data...

www.crystallography.net/result.php?CODSESSION=of6r7lhhh8n7a7k5fksld7pbldta1lhh&order_by=file&order=desc

Crystallography Open Database

COD Home

Home
What's new?

Accessing COD Data

Browse
Search
Search by structural formula

Add Your Data

Deposit your data
Manage depositions
Manage/release prepublications

Documentation

COD Wiki
Obtaining COD
Citing COD
COD Mirrors
Advices to donators
Useful links

Search results

Result: there are 9 entries in the selection

Switch to the old layout of the page

Download all results as: list of COD numbers | list of CIF URLs | data in CSV format | archive of CIF files (ZIP)

Searching elements including Ti, O number of elements between 2 and 2 text, file, commonname, chemname, mineral contains anatase volume is between 100 and 1400

◄◄ First | ◄ Previous 20 | Page 1 of 1 | Next 20 ► | Last ►► | Display 5 **20** 50 100 200 300 500 1000 entries per page

COD ID ▼	Links	Formula ▲	Space group ▲	Cell parameters	Cell volume ▲	Bibliography
9015929	CIF	O2 Ti	I 41/a m d :1	3.7845; 3.7845; 9.5143 90; 90; 90	136.268	Howard, C. J.; Sabine, T. M.; Dickson, F. Structural and thermal p[illegible] *Acta Crystallographica,*
9009086	CIF	O2 Ti	I 41/a m d :1	3.785; 3.785; 9.514 90; 90; 90	136.3	Wyckoff, R. W. G. Second edition. Interscie[illegible] *Crystal Structures*, **1963**, *1*, 239-444
9008216	CIF	O2 Ti	I 41/a m d :1	3.804; 3.804; 9.614 90; 90; 90	139.119	Horn, M.; Schwerdtfeger, C. F.; Meagher, E. P. Refinement of the structure of anatase at several temperatures Sample: T = 800 C Locality: Legenbach quarry, Binnatal, Switzerland *Zeitschrift fur Kristallographie*, **1972**, *136*, 273-281
9008215	CIF	O2 Ti	I 41/a m d :1	3.7971; 3.7971; 9.579 90; 90; 90	138.11	Horn, M.; Schwerdtfeger, C. F.; Meagher, E. P. Refinement of the structure of anatase at several temperatures Sample: T = 600 C Locality: Legenbach quarry, Binnatal, Switzerland *Zeitschrift fur Kristallographie*, **1972**, *136*, 273-281
9008214	CIF	O2 Ti	I 41/a m d :1	3.7892; 3.7892; 9.537 90; 90; 90	136.933	Horn, M.; Schwerdtfeger, C. F.; Meagher, E. P. Refinement of the structure of anatase at several temperatures Sample: T = 300 C Locality: Legenbach quarry, Binnatal, Switzerland *Zeitschrift fur Kristallographie*, **1972**, *136*, 273-281
9008213	CIF	O2 Ti	I 41/a m d :1	3.7842; 3.7842; 9.5146 90; 90; 90	136.251	Horn, M.; Schwerdtfeger, C. F.; Meagher, E. P. Refinement of the structure of anatase at several temperatures [illegible]

Download CIF file

CIF File Format

CIF (Crystallographic Information File) Format:

- Standardized file format
- Contains structural information
- Contains references
- Specification available at http://www.iucr.org

C:\Users\doebelinn\AppData\Local\Temp\AMS_DATA.cif - Notepad++

File Edit Search View Encoding Language Settings Macro Run Plugins Window ?

AMS_DATA.cif

```
data_global
_chemical_name_mineral 'Anatase'
loop_
_publ_author_name
'Howard C J'
'Sabine T M'
'Dickson F'
_journal_name_full 'Acta Crystallographica, Section B'
_journal_volume 47
_journal_year 1991
_journal_page_first 462
_journal_page_last 468
_publ_section_title
;
 Structural and thermal parameters for rutile and anatase
;
_database_code_amcsd 0019093
_chemical_compound_source 'Synthetic'
_chemical_formula_sum 'Ti O2'
_cell_length_a 3.7845
_cell_length_b 3.7845
_cell_length_c 9.5143
_cell_angle_alpha 90
_cell_angle_beta 90
_cell_angle_gamma 90
_cell_volume 136.268
_exptl_crystal_density_diffrn      3.894
_symmetry_space_group_name_H-M 'I 41/a m d'
loop_
_space_group_symop_operation_xyz
  'x,y,z'
```

```
loop_
_atom_site_label
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
Ti   0.00000   0.00000   0.00000
O   0.00000   0.00000   0.20806
loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_12
_atom_site_aniso_U_13
_atom_site_aniso_U_23
Ti 0.00520 0.00000 0.00000 0.00520 0.00000 0.00700
O 0.01170 0.00000 0.00000 0.00270 0.00000 0.00720
```

length : 1762 lines Ln : 1 Col : 1 Sel : 0 | 0 Dos\Windows ANSI as UTF-8 INS

CIF Format Specification

http://www.iucr.org/resources/cif/dictionaries

International Union of
CRYSTALLOGRAPHY

iucr | journals | books | news | education | people | resources | iycr2014

world directory | other directories | data | cif | lists | blogs | forums | commissions | nexus | symmetry font

Home > resources > cif > dictionaries

- Search for CIF data name
- Core CIF dictionary
- Restraints dictionary
- Powder CIF dictionary
- Modulated structures CIF dictionary
- Electron density CIF dictionary
- Twinning CIF dictionary
- Macromolecular CIF dictionary
- Image CIF dictionary
- Symmetry CIF dictionary
- Suggested new CIF data items

CIF dictionaries

CIF dictionaries provide a formal taxonomy of crystallographic terms and ideas. Dictionary entries are constructed in a structured machine-readable manner that facilitates validation and structuring of data. New entries may be devised for public or private dictionaries. A candidate data-name definition should fulfil the following conditions: (i) describe a specific and well defined concept: precision of definition is essential for an effective interchange mechanism; (ii) have appropriate granularity: data names can define a very small piece of information (a standard uncertainty on a particular physical measurable) or a very large amount (the text of a scientific paper). An appropriate choice should be made (and for DDL2 formalized through membership of subcategories, category and category groups, as appropriate); (iii) have well defined relationships with other data items (through its assigned category membership and parent/child links); for DDL2 the prior construction of a formal entity/relationship schema may be helpful ; (iv) constraints on the data type and permissible values should be provided where applicable; (v) the name chosen should be globally unique; this is achieved through monitoring of names in public dictionaries by a regulatory committee (COMCIFS) and by registering of prefix strings for exclusive use in local dictionaries. Some thought may need to be applied to the choice of DDL appropriate for a candidate dictionary.

Current CIF Dictionaries

Canonical data descriptors and their attributes are presented in machine-readable data dictionaries.

Descriptors for small-molecule, inorganic and other small-unit-cell structures

- Core dictionary (coreCIF)
  [ASCII | HTML | PDF | More information]
- Restraints dictionary
  [ASCII | HTML | PDF | More information]
- Powder dictionary (pdCIF)
  [ASCII | HTML | PDF | More information]
- Modulated and composite structures dictionary (msCIF)
  [ASCII | HTML | PDF | More information]
- Electron density dictionary (rhoCIF)
  [ASCII | HTML | PDF | More information]
- Twinning dictionary
  [ASCII | HTML | PDF | More information]

Problems with CIF Files

Problems with CIF files:

1. May be (and very often ARE) incomplete
2. May use non-standard atomic settings
3. Must be converted to STR format for BGMN

Creating Structure Files

Official Documentation:

http://www.bgmn.de/variables.html

Control File (*.sav)

Structure File (*.str)

Device Conf. File (*.sav)

Variables for use with BGMN

The expression interpreter

- standard functions
- multiple equation symbols in assignments
- user-defined string functions

Variables in the task description SAV file for BGMN

NTHREADS, VAL, VERZERR, STRUC, STRUCOUT, SimpleSTRUCOUT, PDBOUT, RESOUT, FCFOUT, OUTPUT, LIST, RU, UNT, UNTC, DDM, PARAM, LAMBDA, SYNCHROTRON, EPS1, EPS2, EPS3, EPS4, POL, PROTOKOLL, ONLYISO, ITMAX, DIAGRAMM, PLAN, STANDALONEPLAN, GOAL, WMIN, WMAX, CUT, LIMIT2, LIMIT4, LIMIT6, LIMIT8, LIMIT10, ANISOLIMIT, ANISO4LIMIT

Items in the structure description *.str-file

Definition of the lattice
SpacegroupNo, HermannMauguin, GeneralCondition, A, B, C, ALPHA, BETA, GAMMA, UNIT
Anisotropic variables
ANISO, ANISOLIN, ANISOSQR, ANISO4
Scaling factor/prefered orientation
GEWICHT, SPHAR0, SPHAR2, SPHAR4, SPHAR6, SPHAR8, SPHAR10
Real structure
B1, B2, k1, k2, k3, sk, H, h, k, l, zweiTheta, RP
Peak deselection
GeneralCondition
Atomic positions
E=, Wyckoff, x, y, z, TDS, B, betaij, U, Uij
GOAL's, quant analysis
GOAL, GEWICHT
BGMN-specific functions
PHASE, sk, B1, k2, ANISO, GEWICHT(h,k,l), GrainSize, TDS,
Advanced feature: subphases
RefMult, iref, GEWICHT[i], B1[i], B2[i], k1[i], k2[i], k3[i], DELTAsk, DELTAzweiTheta, LeBail, FPARAM
Advanced feature: Structure amplitudes etc
F, Finv, H
Advanced feature: User calculated structure amplitudes
FMult, F[i], phi[i], Finv[i], phiinv[i]
Advanced feature: molecules
set, setgitter, cross, diffvec, normvec, skalpro, distance, angle, cpXYZ, T, D, X, Y, Z, WW, WWalt, Straf, Theory, Bondings, BondLevel
Micro absorption correction according to Brindley
my, my[i]
X ray density
density, density[i]

Variables in the task description SAV file for computation of standard profiles

NTHREADS, VERZERR, TubeTails, R, FocusH, FocusW, HSlitR, HSlitW, RoundSlitR, RoundSlitD PColl, PCollA, VSlitR, VSlitH, SamplD, SamplW, SamplH, DeltaOmega, SCollA, SSlitW, SSlitR, SColl, SCollA, DetW, DetH, DetArrayW, MonR, MonH, EPSG, zweiTheta[i], GSUM, TSlitR, TSlitH, FocusS, FocusA, GEOMETRY, WMIN, WMAX, WSTEP, GEQ, D, T, STANDARDPAR, VAL,

Creating Structure Files

BGMN_manual_2005.pdf

Manual

Rietveld Analysis Program

BGMN®

Jörg Bergmann, Dresden, Germany

From CIF to BGMN Structure Files (I)

CIF File

```
_chemical_name_mineral 'Anatase'



_symmetry_space_group_name_H-M 'I 41/a m d'

_cell_length_a 3.7845
_cell_length_c 9.5143

Ti

          0.00000
          0.00000
          0.00000
          0.0052 0.0000 0.0000
                    0.0052 0.0000 0.0070
O

          0.00000
          0.00000
          0.20806
          0.0117 0.0000 0.0000
                    0.0027 0.0000 0.0072
```

OK

Not available

Wrong format

Wrong unit

OK (sort of…)

Not available

OK

Wrong format

OK (sort of…)

Not available

OK

Wrong format

BGMN STR File

```
PHASE=Anatase

SpacegroupNo=141

HermannMauguin=I4_1/a2/m2/d

A=0.3784
C=0.9514

E=TI+4
          Wyckoff=a
          x=0.00000
          y=0.00000
          z=0.00000
          TDS=0.0045795

E=O-2
          Wyckoff=e
          x=0.00000
          y=0.00000
          z=0.20806
          TDS=0.0056849
```

From CIF to BGMN Structure Files (II)

Category	Solution
OK	Can be mapped from CIF to STR
OK (sort of…)	Can be mapped / or fixed easily
Wrong format / unit	Need conversion
Not available	PROBLEM!!!

PROBLEM!!!

=

gather more information from other sources

and fix manually

From CIF to BGMN Structure Files (III)

PROBLEM!!!

=

gather more information from other sources

and fix manually

- Find a better, more complete, CIF file
  (e.g. from another structure refinement)
- Search for the same structure in a different database
- Find missing information elsewhere and add to CIF file
  (original publication, different database, different publication)

PDF-4+ Database

TDS (=IDP) in *B* format

Wyckoff

Space Group No.

HM Symbol still in wrong format

Ti O2 - 04-007-0701

File Edit d-Spacings Tools Window Help

d-Spacings

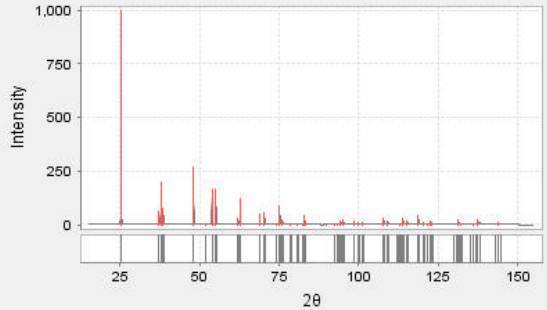
Wavelength: Cu Kα1 1.54056Å

Stick Patterns: Fixed Slit Intensity; Variable Slit Intensity; Integrated Intensity

Diffraction Patterns: Simulated Profile; Raw Diffraction Data (PD3)

Fixed Slit Intensity

2θ	d(Å)	I	h	k	l	*
25.3060	**3.516520**	999	1	0	1	
36.9496	2.430770	63	1	0	3	
37.7907	**2.378580**	197	0	0	4	
38.5692	2.332340	75	1	1	2	
48.0419	**1.892250**	265	2	0	0	
51.9644	1.758260	1	2	0	2	
53.8842	1.700060	163	1	0	5	
55.0666	1.666320	166	2	1	1	
62.1116	1.493160	28	2	1	3	
62.6875	1.480820	121	2	0	4	
68.7544	1.364200	51	1	1	6	
70.2954	1.338020	57	2	2	0	



— 04-007-0701 (Fixed Slit Intensity) — 04-007-0701 (Calc)

PDF | Experimental | Physical | Crystal | Optical | Structure | Miscellaneous | References | Comments

TDP Type: B; Origin: O1; Crystal (Symmetry Allowed): Centrosymmetric

SG Symmetry Operators (16)

Seq	Operator
1	x,y,z
2	-x+1/2,-y+1/2,z+1/2
3	x+1/2,y,-z+3/4
4	-x,-y+1/2,-z+1/4
5	-x,y,z
6	x+1/2,-y+1/2,z+1/2
7	-x+1/2,y,-z+3/4
8	x,-y+1/2,-z+1/4
9	y+1/2,x+1/2,-z+1/2
10	-y,-x,-z
11	y,x+1/2,z+1/4

Atomic Coordinates (2)

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	IDP	AET
Ti	1	4a	-4m2	0.0	0.0	0.0	1.0	0.45795	6-a
O	2	8e	2mm.	0.0	0.0	0.20806	1.0	0.56849	3#b

Anisotropic Thermal Displacement Parameters (2)

Atom	Num	ADP11	ADP22	ADP33	ADP12	ADP13	ADP23
Ti	1	0.41058	0.41058	0.5527	0.0	0.0	0.0
O	2	0.92379	0.21318	0.56849	0.0	0.0	0.0

PDF | Experimental | Physical | Crystal | Optical | Structure | Miscellaneous | References | Comments

ICDD Calculated Parameters

Space Group: I41/amd (141); Molecular Weight: 79.90

Crystal Data

a: 3.784 Å; b: 3.784 Å; c: 9.514 Å

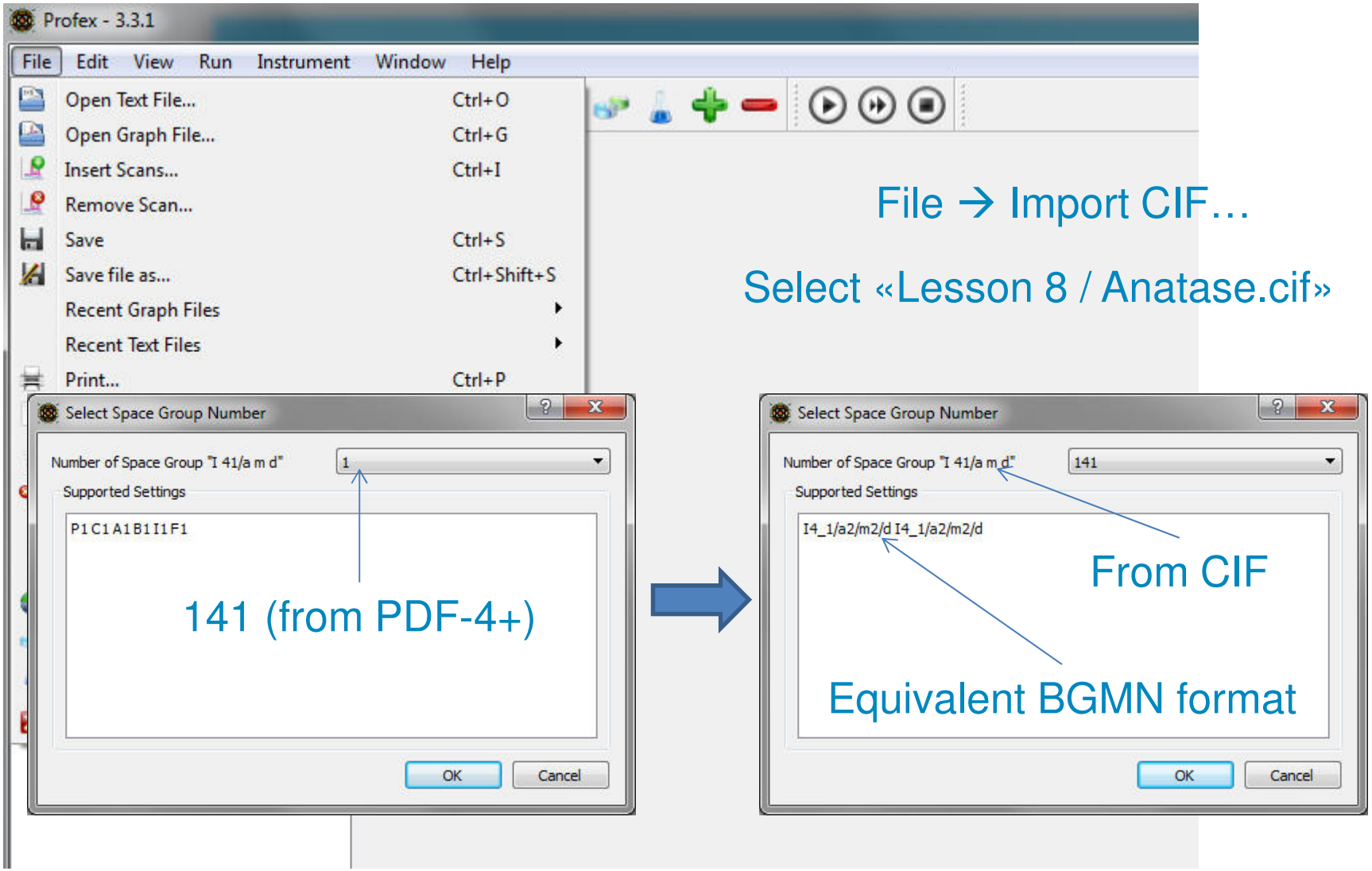
α: 90.00 °; β: 90.00 °; γ: 90.00 °

Volume: 136.27 Å³; Z: 4.00

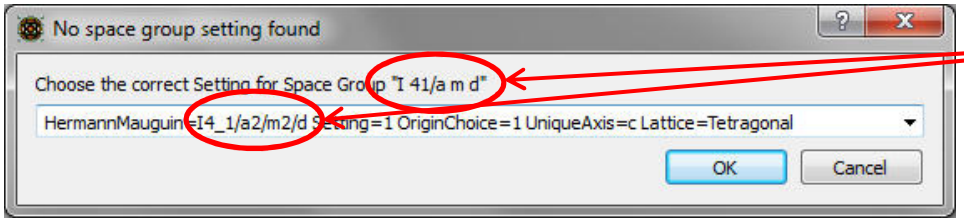
From CIF to BGMN Structure Files

Profex CIF Import

CIF to STR with Profex (I)

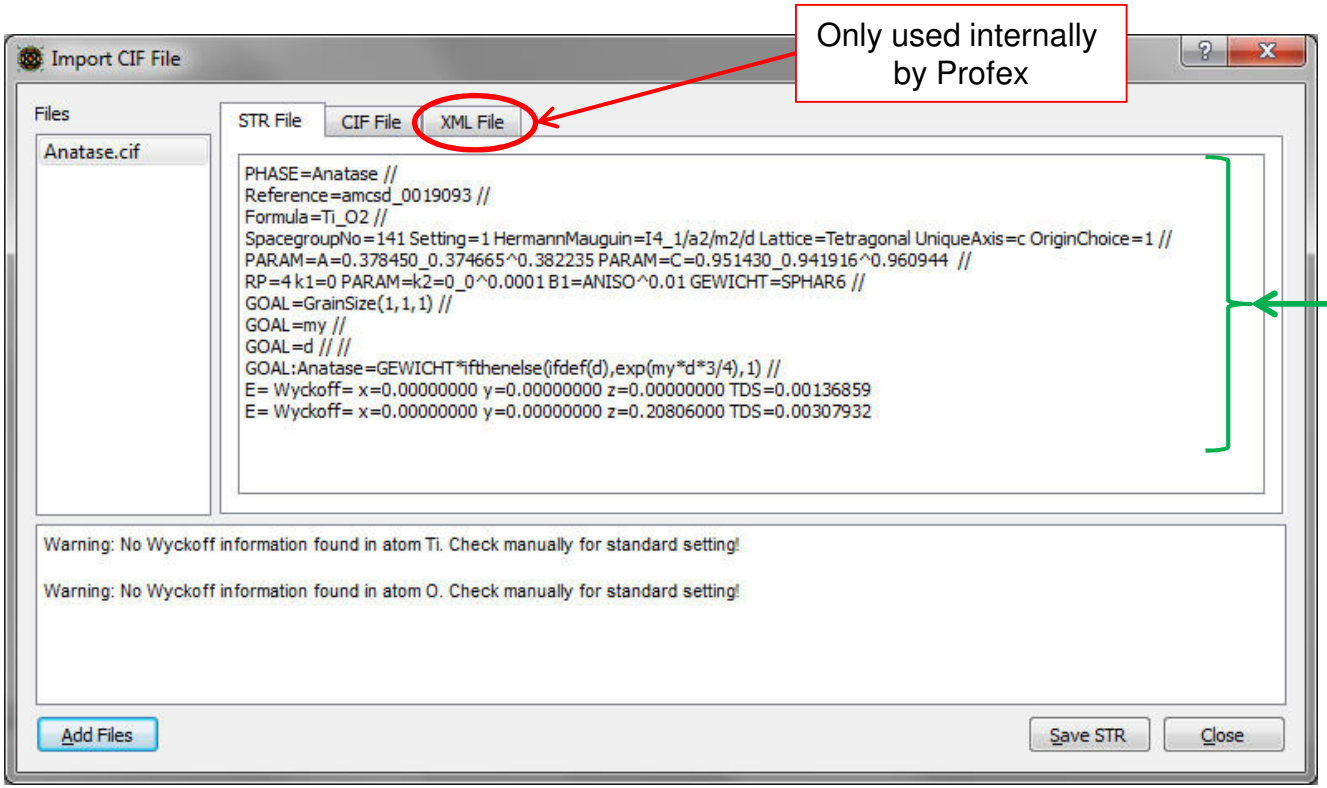


CIF to STR with Profex (II)



Normally: Match HM Symbols

Here: No difference, just click OK



Only used internally by Profex

Check manually, add:

- Element types
- Wyckoff sequences

CIF to STR with Profex (III)

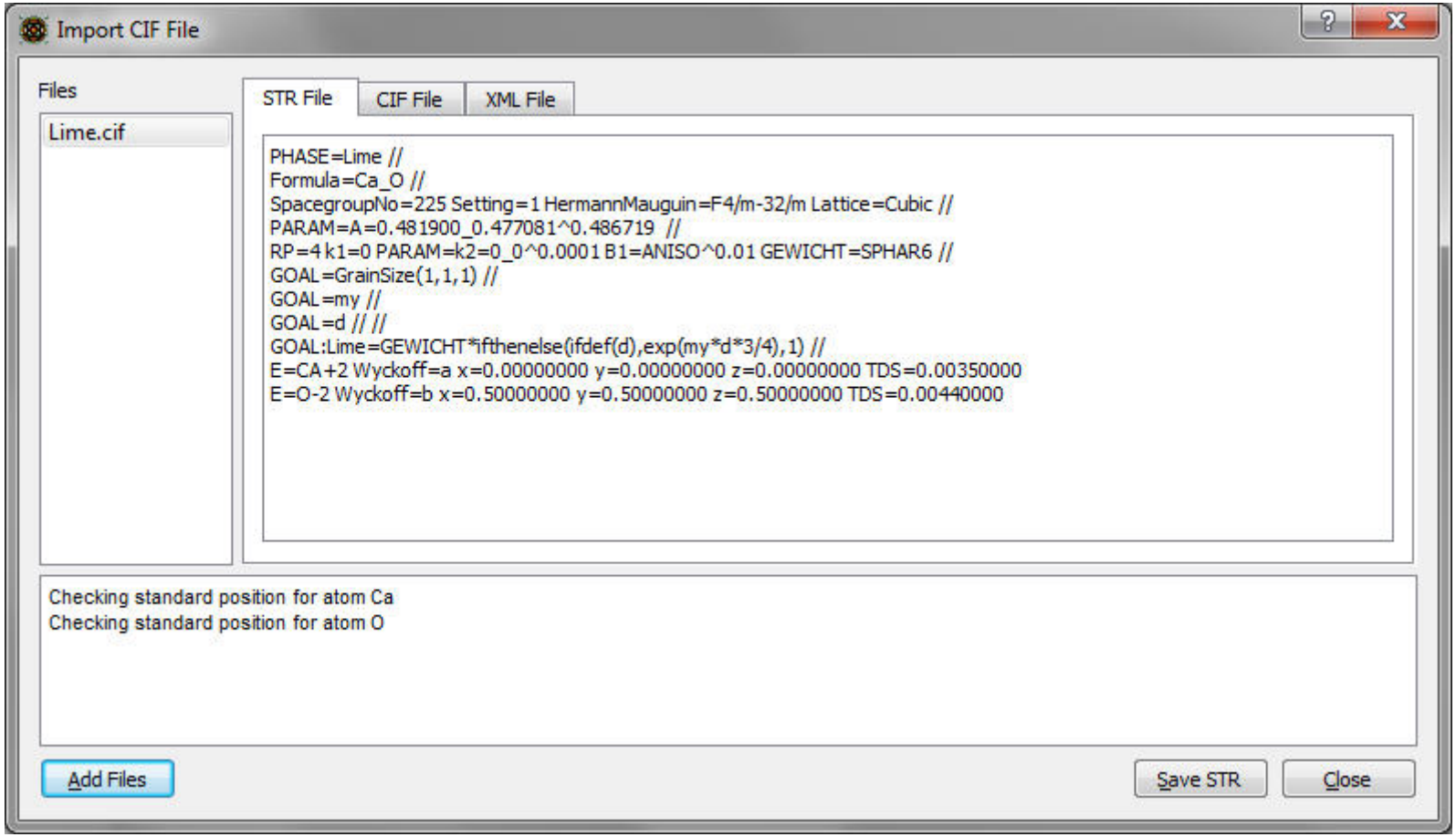
```
PHASE=Anatase //
Reference=amcsd_0019093 //
Formula=Ti_O2 //
SpacegroupNo=141 Setting=1 HermannMauguin=I4_1/a2/m2/d Lattice=Tetragonal
UniqueAxis=c OriginChoice=1 //
PARAM=A=0.378450_0.374665^0.382235 PARAM=C=0.951430_0.941916^0.960944 //
RP=4 k1=0 PARAM=k2=0_0^0.0001 B1=ANISO^0.01 GEWICHT=SPHAR6 //
GOAL=GrainSize(1,1,1) //
GOAL=my //
GOAL=d // //
GOAL:Anatase=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1) //
E=TI+4 Wyckoff=a x=0.00000000 y=0.00000000 z=0.00000000 TDS=0.00136859
E=O-2  Wyckoff=e x=0.00000000 y=0.00000000 z=0.20806000 TDS=0.00307932
```

- Add missing information (Element Types and Wyckoff Sequences)
- «Save STR»
- Save file in Structure Database Directory:
  …\Profex-BGMN-Bundle-3.3.1\Profex\Structures

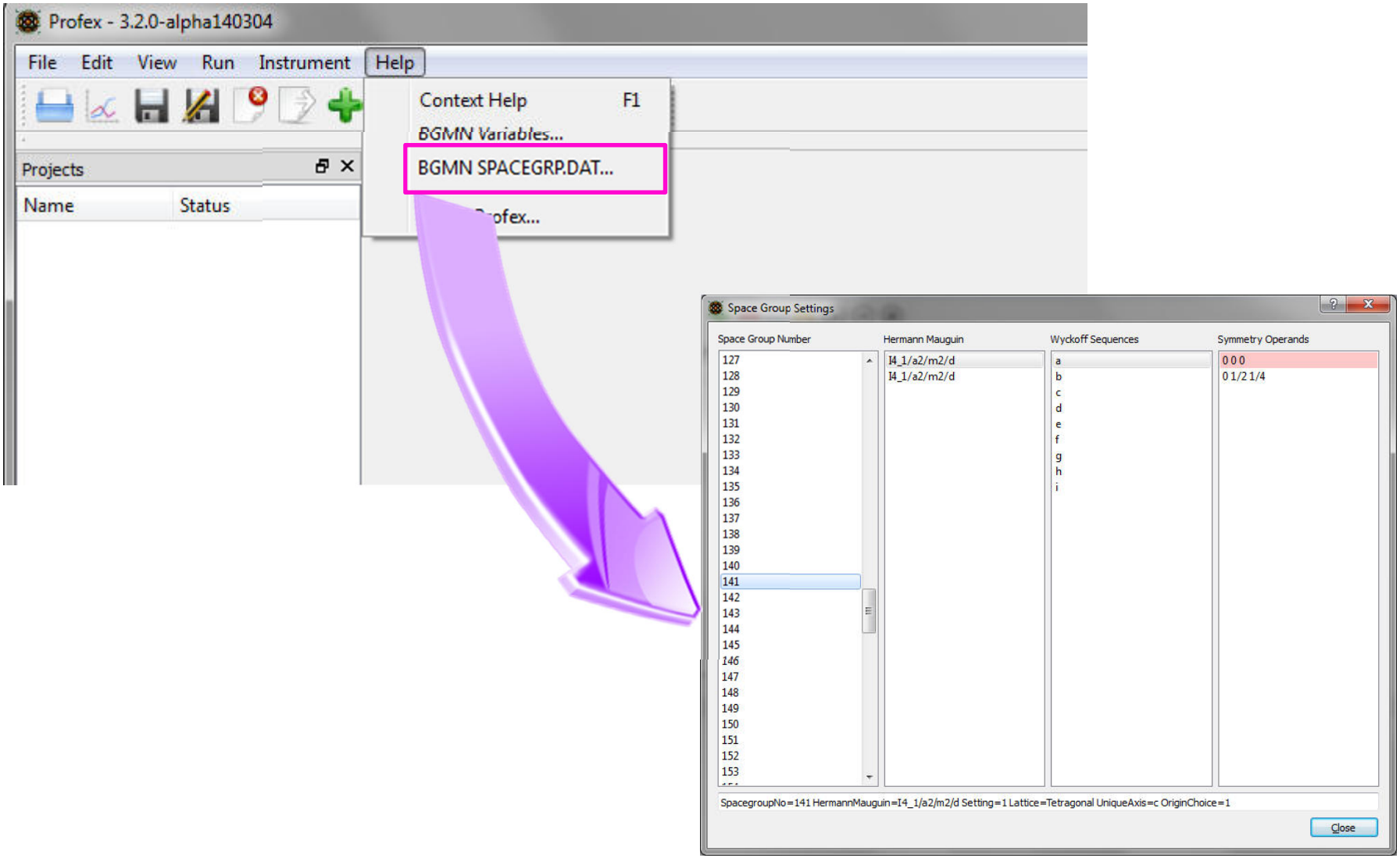
CIF to STR with Profex (IV)

With correct and complete CIF files: No / minimal user input required

Try: «Lesson 8 / Lime.cif»



SPACEGRP.DAT Browser

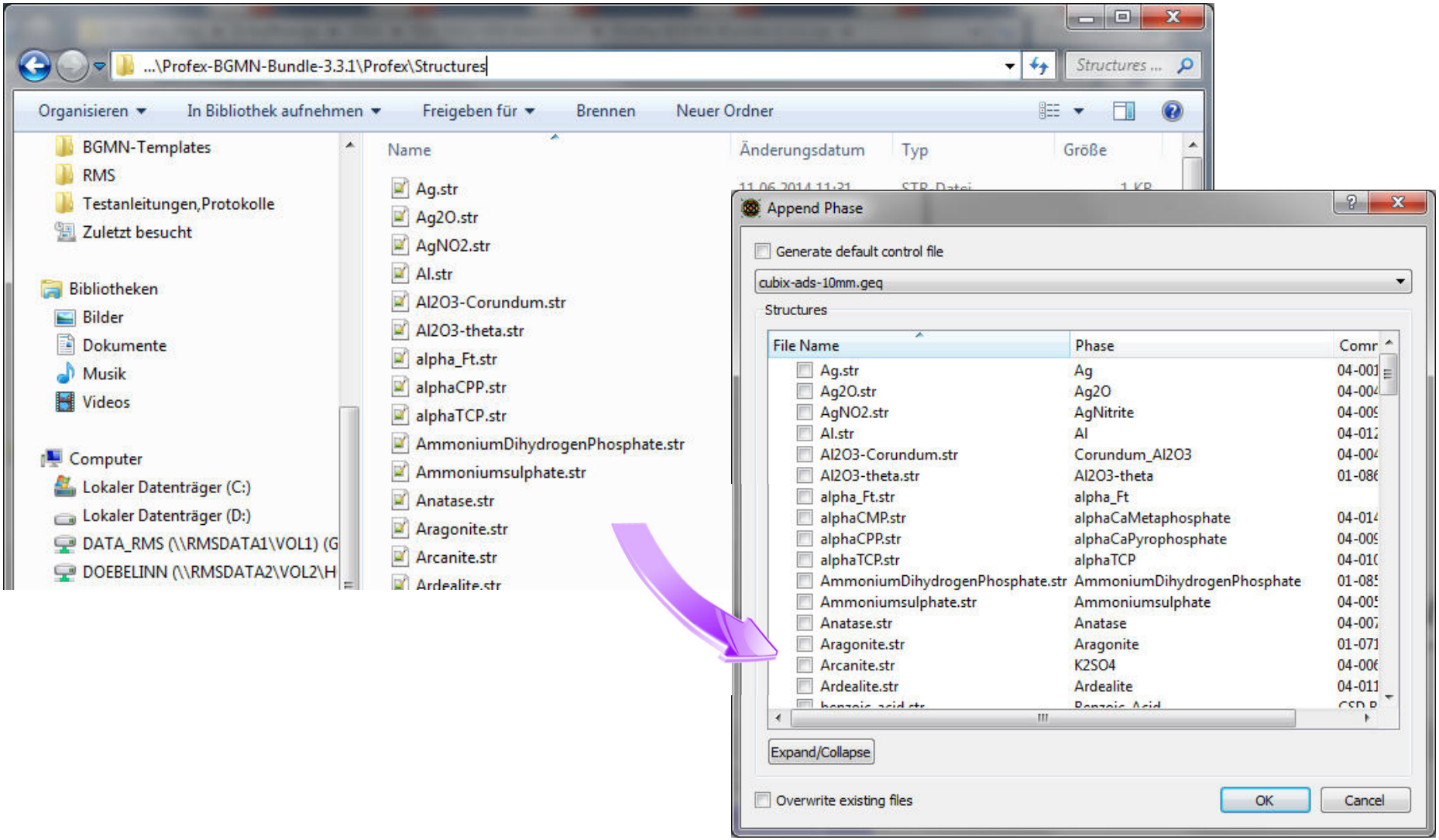


CIF to STR with Profex (VII)

Summary:

- Crystal structure data available from various databases (free and commercial)
- Provided in CIF format
- Usually additional information required
- Semi-automatic conversion CIF → STR with Profex
- Manual fixing / verification required

Profex Structure and Device Database



Profex Structure and Device Database