



# Full wwPDB X-ray Structure Validation Report ⓘ

Aug 8, 2020 – 01:53 AM BST

PDB ID : 6IC0  
Title : Human PFKFB3 in complex with a N-Aryl 6-Aminoquinoxaline inhibitor 4  
Authors : Banaszak, K.; Pawlik, H.; Bialas, A.; Fabritius, C.H.; Nowak, M.  
Deposited on : 2018-12-01  
Resolution : 2.60 Å(reported)

This is a Full wwPDB X-ray Structure Validation Report for a publicly released PDB entry.

We welcome your comments at [validation@mail.wwpdb.org](mailto:validation@mail.wwpdb.org)

A user guide is available at

<https://www.wwpdb.org/validation/2017/XrayValidationReportHelp>

with specific help available everywhere you see the ⓘ symbol.

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The following versions of software and data (see [references ⓘ](#)) were used in the production of this report:

MolProbity : 4.02b-467  
Mogul : 1.8.5 (274361), CSD as541be (2020)  
Xtriage (Phenix) : 1.13  
EDS : 2.13.1  
buster-report : 1.1.7 (2018)  
Percentile statistics : 20191225.v01 (using entries in the PDB archive December 25th 2019)  
Refmac : 5.8.0158  
CCP4 : 7.0.044 (Gargrove)  
Ideal geometry (proteins) : Engh & Huber (2001)  
Ideal geometry (DNA, RNA) : Parkinson et al. (1996)  
Validation Pipeline (wwPDB-VP) : 2.13.1

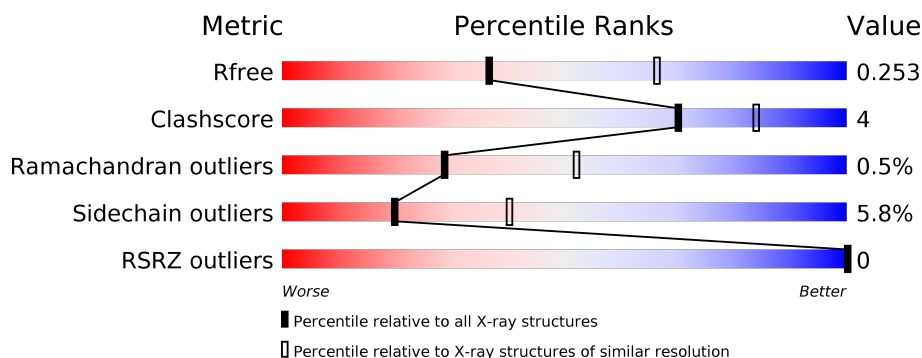
# 1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

*X-RAY DIFFRACTION*


The reported resolution of this entry is 2.60 Å.

Percentile scores (ranging between 0-100) for global validation metrics of the entry are shown in the following graphic. The table shows the number of entries on which the scores are based.



Metric	Whole archive (#Entries)	Similar resolution (#Entries, resolution range(Å))
$R_{free}$	130704	3163 (2.60-2.60)
Clashscore	141614	3518 (2.60-2.60)
Ramachandran outliers	138981	3455 (2.60-2.60)
Sidechain outliers	138945	3455 (2.60-2.60)
RSRZ outliers	127900	3104 (2.60-2.60)

The table below summarises the geometric issues observed across the polymeric chains and their fit to the electron density. The red, orange, yellow and green segments on the lower bar indicate the fraction of residues that contain outliers for  $\geq 3$ , 2, 1 and 0 types of geometric quality criteria respectively. A grey segment represents the fraction of residues that are not modelled. The numeric value for each fraction is indicated below the corresponding segment, with a dot representing fractions  $\leq 5\%$ . The upper red bar (where present) indicates the fraction of residues that have poor fit to the electron density. The numeric value is given above the bar.

Mol	Chain	Length	Quality of chain
1	A	428	

## 2 Entry composition

There are 7 unique types of molecules in this entry. The entry contains 3617 atoms, of which 0 are hydrogens and 0 are deuteriums.

In the tables below, the ZeroOcc column contains the number of atoms modelled with zero occupancy, the AltConf column contains the number of residues with at least one atom in alternate conformation and the Trace column contains the number of residues modelled with at most 2 atoms.

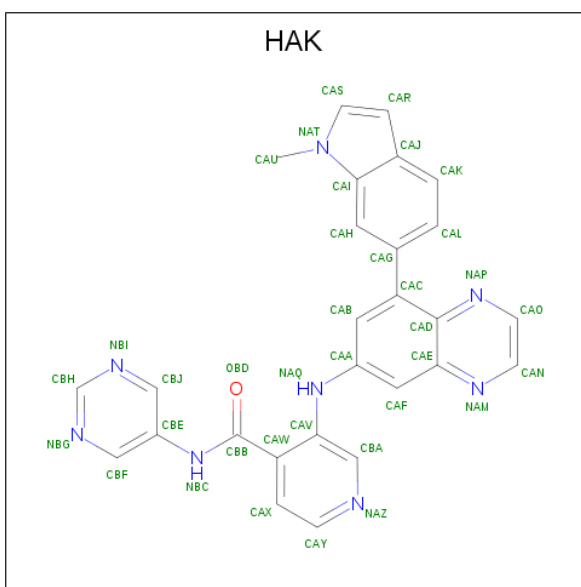
- Molecule 1 is a protein called 6-phosphofructo-2-kinase/fructose-2,6-bisphosphatase 3.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	428	Total	C	N	O	S	0	0	0
			3465	2193	600	651	21			

There are 15 discrepancies between the modelled and reference sequences:

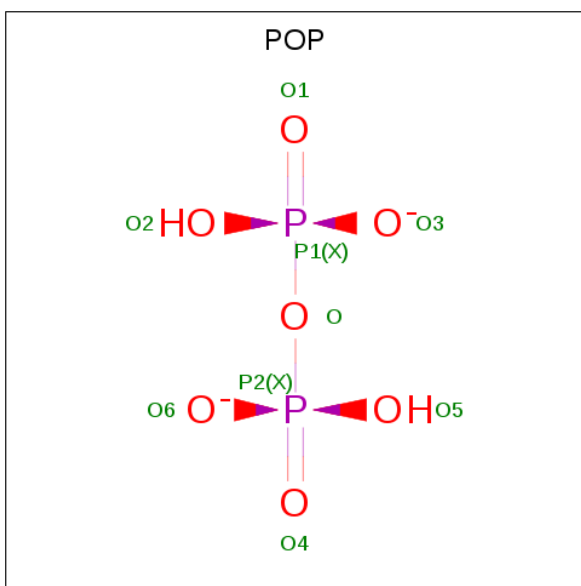
Chain	Residue	Modelled	Actual	Comment	Reference
A	?	-	ASP	deletion	UNP Q16875
A	?	-	HIS	deletion	UNP Q16875
A	?	-	ARG	deletion	UNP Q16875
A	?	-	PRO	deletion	UNP Q16875
A	?	-	SER	deletion	UNP Q16875
A	?	-	LEU	deletion	UNP Q16875
A	?	-	PRO	deletion	UNP Q16875
A	?	-	ARG	deletion	UNP Q16875
A	?	-	SER	deletion	UNP Q16875
A	?	-	CYS	deletion	UNP Q16875
A	?	-	GLY	deletion	UNP Q16875
A	?	-	PRO	deletion	UNP Q16875
A	?	-	LYS	deletion	UNP Q16875
A	?	-	LEU	deletion	UNP Q16875
A	?	-	THR	deletion	UNP Q16875

- Molecule 2 is 3-[[8-(1-methylindol-6-yl)quinoxalin-6-yl]amino]- {N}-pyrimidin-5-yl-pyridine-4-carboxamide (three-letter code: HAK) (formula: C<sub>27</sub>H<sub>20</sub>N<sub>8</sub>O).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
2	A	1	Total	C	N	O	0	0
			36	27	8	1		

- Molecule 3 is PYROPHOSPHATE 2- (three-letter code: POP) (formula:  $\text{H}_2\text{O}_7\text{P}_2$ ).



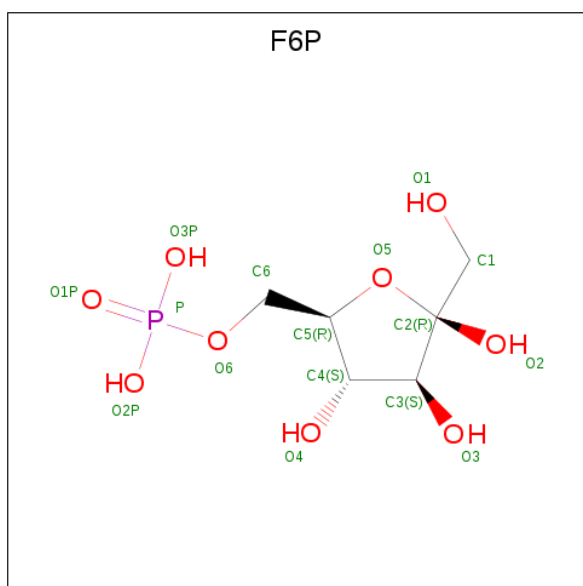
Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
3	A	1	Total	O	P	0	0
			9	7	2		

- Molecule 4 is CITRATE ANION (three-letter code: FLC) (formula:  $\text{C}_6\text{H}_5\text{O}_7$ ).



Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
4	A	1	Total	C	O	0	0
			13	6	7		

- Molecule 5 is 6-O-phosphono-beta-D-fructofuranose (three-letter code: F6P) (formula:  $C_6H_{13}O_9P$ ).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
5	A	1	Total	C	O	P	0	0
			16	6	9	1		

- Molecule 6 is DIMETHYL SULFOXIDE (three-letter code: DMS) (formula:  $C_2H_6OS$ ).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
6	A	1	Total	C	O	S	0	0
			4	2	1	1		

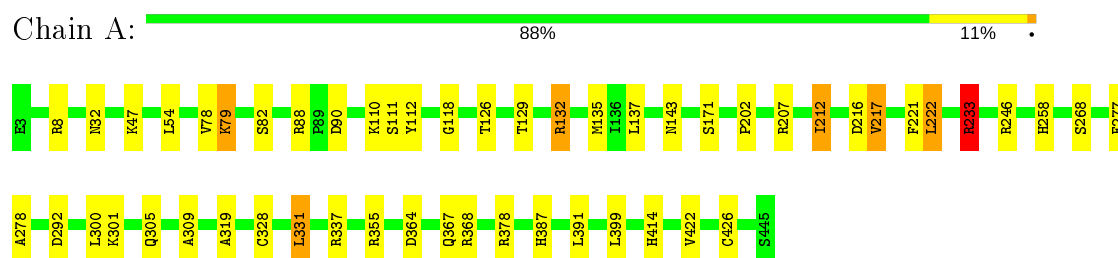
- Molecule 7 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
7	A	74	Total	O	0	0
			74	74		

### 3 Residue-property plots

These plots are drawn for all protein, RNA, DNA and oligosaccharide chains in the entry. The first graphic for a chain summarises the proportions of the various outlier classes displayed in the second graphic. The second graphic shows the sequence view annotated by issues in geometry and electron density. Residues are color-coded according to the number of geometric quality criteria for which they contain at least one outlier: green = 0, yellow = 1, orange = 2 and red = 3 or more. A red dot above a residue indicates a poor fit to the electron density ( $RSRZ > 2$ ). Stretches of 2 or more consecutive residues without any outlier are shown as a green connector. Residues present in the sample, but not in the model, are shown in grey.

- Molecule 1: 6-phosphofructo-2-kinase/fructose-2,6-bisphosphatase 3



## 4 Data and refinement statistics

Property	Value	Source
Space group	P 65 2 2	Depositor
Cell constants a, b, c, $\alpha$ , $\beta$ , $\gamma$	101.94Å 101.94Å 251.65Å 90.00° 90.00° 120.00°	Depositor
Resolution (Å)	47.24 – 2.60 47.24 – 2.60	Depositor EDS
% Data completeness (in resolution range)	99.9 (47.24-2.60) 100.0 (47.24-2.60)	Depositor EDS
$R_{merge}$	0.15	Depositor
$R_{sym}$	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ <sup>1</sup>	2.44 (at 2.61Å)	Xtriage
Refinement program	REFMAC	Depositor
R, $R_{free}$	0.191 , 0.255 0.198 , 0.253	Depositor DCC
$R_{free}$ test set	1235 reflections (5.00%)	wwPDB-VP
Wilson B-factor (Å <sup>2</sup> )	48.2	Xtriage
Anisotropy	0.089	Xtriage
Bulk solvent $k_{sol}$ (e/Å <sup>3</sup> ), $B_{sol}$ (Å <sup>2</sup> )	0.34 , 34.7	EDS
L-test for twinning <sup>2</sup>	$\langle  L  \rangle = 0.48$ , $\langle L^2 \rangle = 0.31$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
$F_o, F_c$ correlation	0.94	EDS
Total number of atoms	3617	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	47.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 3.45% of the height of the origin peak. No significant pseudotranslation is detected.*

<sup>1</sup>Intensities estimated from amplitudes.

<sup>2</sup>Theoretical values of  $\langle |L| \rangle$ ,  $\langle L^2 \rangle$  for acentric reflections are 0.5, 0.333 respectively for untwinned datasets, and 0.375, 0.2 for perfectly twinned datasets.



## 5 Model quality [i](#)

### 5.1 Standard geometry [i](#)

Bond lengths and bond angles in the following residue types are not validated in this section: FLC, HAK, DMS, POP, F6P

The Z score for a bond length (or angle) is the number of standard deviations the observed value is removed from the expected value. A bond length (or angle) with  $|Z| > 5$  is considered an outlier worth inspection. RMSZ is the root-mean-square of all Z scores of the bond lengths (or angles).

Mol	Chain	Bond lengths		Bond angles	
		RMSZ	$\# Z  > 5$	RMSZ	$\# Z  > 5$
1	A	0.70	0/3535	0.87	1/4784 (0.0%)

There are no bond length outliers.

All (1) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed( $^{\circ}$ )	Ideal( $^{\circ}$ )
1	A	233	ARG	NE-CZ-NH1	6.87	123.73	120.30

There are no chirality outliers.

There are no planarity outliers.

### 5.2 Too-close contacts [i](#)

In the following table, the Non-H and H(model) columns list the number of non-hydrogen atoms and hydrogen atoms in the chain respectively. The H(added) column lists the number of hydrogen atoms added and optimized by MolProbity. The Clashes column lists the number of clashes within the asymmetric unit, whereas Symm-Clashes lists symmetry related clashes.

Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
1	A	3465	0	3418	24	0
2	A	36	0	0	0	0
3	A	9	0	0	0	0
4	A	13	0	5	2	0
5	A	16	0	11	0	0
6	A	4	0	6	0	0
7	A	74	0	0	2	0
All	All	3617	0	3440	25	0

The all-atom clashscore is defined as the number of clashes found per 1000 atoms (including

hydrogen atoms). The all-atom clashscore for this structure is 4.

All (25) close contacts within the same asymmetric unit are listed below, sorted by their clash magnitude.

Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:A:47:LYS:HE3	1:A:126:THR:HA	1.84	0.59
1:A:378:ARG:NH2	7:A:601:HOH:O	2.37	0.57
1:A:426:CYS:SG	7:A:668:HOH:O	2.58	0.56
1:A:328:CYS:HA	1:A:331:LEU:HD22	1.88	0.56
1:A:212:ILE:HD11	1:A:221:PHE:HD2	1.71	0.55
1:A:110:LYS:HD2	1:A:143:ASN:OD1	2.08	0.54
1:A:355:ARG:NH1	1:A:364:ASP:OD1	2.44	0.51
4:A:503:FLC:OG2	4:A:503:FLC:HA1	2.10	0.49
1:A:300:LEU:HD12	1:A:387:HIS:CE1	2.48	0.49
1:A:233:ARG:HH11	1:A:233:ARG:HG2	1.79	0.48
1:A:277:PHE:CG	1:A:414:HIS:CD2	3.02	0.47
1:A:126:THR:HG23	4:A:503:FLC:CAC	2.44	0.46
1:A:277:PHE:CE1	1:A:414:HIS:HA	2.51	0.46
1:A:212:ILE:HA	1:A:222:LEU:O	2.16	0.45
1:A:202:PRO:O	1:A:207:ARG:HA	2.17	0.45
1:A:277:PHE:HB2	1:A:414:HIS:CD2	2.52	0.44
1:A:112:TYR:CE2	1:A:118:GLY:HA3	2.52	0.44
1:A:319:ALA:O	1:A:368:ARG:NH1	2.50	0.44
1:A:301:LYS:O	1:A:305:GLN:HG3	2.17	0.44
1:A:129:THR:OG1	1:A:132:ARG:HB2	2.19	0.42
1:A:278:ALA:HB1	1:A:309:ALA:CB	2.50	0.42
1:A:88:ARG:HB3	1:A:90:ASP:OD1	2.21	0.40
1:A:202:PRO:HG2	1:A:222:LEU:HD22	2.04	0.40
1:A:216:ASP:C	1:A:217:VAL:HG23	2.42	0.40
1:A:78:VAL:O	1:A:79:LYS:O	2.40	0.40

There are no symmetry-related clashes.

## 5.3 Torsion angles ⓘ

### 5.3.1 Protein backbone ⓘ

In the following table, the Percentiles column shows the percent Ramachandran outliers of the chain as a percentile score with respect to all X-ray entries followed by that with respect to entries of similar resolution.

The Analysed column shows the number of residues for which the backbone conformation was analysed, and the total number of residues.

Mol	Chain	Analysed	Favoured	Allowed	Outliers	Percentiles
1	A	424/428 (99%)	402 (95%)	20 (5%)	2 (0%)	29 52

All (2) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	A	79	LYS
1	A	217	VAL

### 5.3.2 Protein sidechains ⓘ

In the following table, the Percentiles column shows the percent sidechain outliers of the chain as a percentile score with respect to all X-ray entries followed by that with respect to entries of similar resolution.

The Analysed column shows the number of residues for which the sidechain conformation was analysed, and the total number of residues.

Mol	Chain	Analysed	Rotameric	Outliers	Percentiles
1	A	380/386 (98%)	358 (94%)	22 (6%)	20 40

All (22) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	8	ARG
1	A	32	ASN
1	A	54	LEU
1	A	82	SER
1	A	111	SER
1	A	132	ARG
1	A	135	MET
1	A	137	LEU
1	A	171	SER
1	A	212	ILE
1	A	222	LEU
1	A	233	ARG
1	A	246	ARG
1	A	258	HIS
1	A	268	SER
1	A	292	ASP
1	A	331	LEU
1	A	337	ARG
1	A	367	GLN

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Mol	Chain	Res	Type
1	A	391	LEU
1	A	399	LEU
1	A	422	VAL

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (1) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	32	ASN

### 5.3.3 RNA ⓘ

There are no RNA molecules in this entry.

## 5.4 Non-standard residues in protein, DNA, RNA chains ⓘ

There are no non-standard protein/DNA/RNA residues in this entry.

## 5.5 Carbohydrates ⓘ

There are no monosaccharides in this entry.

## 5.6 Ligand geometry ⓘ

5 ligands are modelled in this entry.

In the following table, the Counts columns list the number of bonds (or angles) for which Mogul statistics could be retrieved, the number of bonds (or angles) that are observed in the model and the number of bonds (or angles) that are defined in the Chemical Component Dictionary. The Link column lists molecule types, if any, to which the group is linked. The Z score for a bond length (or angle) is the number of standard deviations the observed value is removed from the expected value. A bond length (or angle) with  $|Z| > 2$  is considered an outlier worth inspection. RMSZ is the root-mean-square of all Z scores of the bond lengths (or angles).

Mol	Type	Chain	Res	Link	Bond lengths			Bond angles		
					Counts	RMSZ	$\# Z  > 2$	Counts	RMSZ	$\# Z  > 2$
6	DMS	A	505	-	3,3,3	0.47	0	3,3,3	0.70	0
3	POP	A	502	-	6,8,8	0.71	0	13,13,13	1.47	3 (23%)
4	FLC	A	503	-	3,12,12	2.30	1 (33%)	3,17,17	3.78	1 (33%)
2	HAK	A	501	-	40,41,41	2.15	15 (37%)	50,58,58	1.54	12 (24%)

Mol	Type	Chain	Res	Link	Bond lengths			Bond angles		
					Counts	RMSZ	# Z  > 2	Counts	RMSZ	# Z  > 2
5	F6P	A	504	-	15,16,16	0.99	1 (6%)	17,25,25	1.09	0

In the following table, the Chirals column lists the number of chiral outliers, the number of chiral centers analysed, the number of these observed in the model and the number defined in the Chemical Component Dictionary. Similar counts are reported in the Torsion and Rings columns. '-' means no outliers of that kind were identified.

Mol	Type	Chain	Res	Link	Chirals	Torsions	Rings
3	POP	A	502	-	-	0/6/6/6	-
4	FLC	A	503	-	-	0/6/16/16	-
2	HAK	A	501	-	-	1/16/16/16	0/6/6/6
5	F6P	A	504	-	-	1/9/28/28	0/1/1/1

All (17) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	A	501	HAK	CAC-CAG	-4.44	1.41	1.49
2	A	501	HAK	CAN-NAM	4.14	1.40	1.32
2	A	501	HAK	CBE-NBC	-3.89	1.33	1.41
4	A	503	FLC	CA-CB	-3.80	1.49	1.54
2	A	501	HAK	CAW-CBB	-3.72	1.42	1.50
2	A	501	HAK	CAK-CAJ	-3.55	1.33	1.41
2	A	501	HAK	CAO-NAP	3.51	1.39	1.32
2	A	501	HAK	CBH-NBI	3.41	1.40	1.33
2	A	501	HAK	CBH-NBG	3.26	1.40	1.33
5	A	504	F6P	O2-C2	3.15	1.46	1.40
2	A	501	HAK	CBJ-NBI	2.99	1.40	1.34
2	A	501	HAK	CAJ-CAI	-2.85	1.35	1.41
2	A	501	HAK	CAH-CAI	-2.78	1.35	1.40
2	A	501	HAK	CAS-NAT	-2.62	1.33	1.37
2	A	501	HAK	CAE-NAM	-2.55	1.33	1.37
2	A	501	HAK	CAA-NAQ	-2.26	1.35	1.40
2	A	501	HAK	CAV-NAQ	-2.01	1.34	1.39

All (16) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
4	A	503	FLC	CB-CA-CAC	-6.39	104.75	114.98
2	A	501	HAK	CAR-CAJ-CAI	3.02	108.78	106.20
2	A	501	HAK	CAF-CAE-CAD	2.99	122.73	119.70
2	A	501	HAK	NBI-CBH-NBG	-2.86	121.02	126.61
2	A	501	HAK	CBF-NBG-CBH	2.80	119.39	115.80

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Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
2	A	501	HAK	CAC-CAB-CAA	2.78	123.66	119.43
3	A	502	POP	P2-O-P1	-2.71	123.53	132.83
2	A	501	HAK	CAX-CAW-CAV	-2.51	115.96	118.81
2	A	501	HAK	CAN-NAM-CAE	2.40	120.64	116.93
2	A	501	HAK	CAY-CAX-CAW	2.37	123.20	118.92
2	A	501	HAK	CBJ-NBI-CBH	2.32	118.76	115.80
2	A	501	HAK	CAV-CAW-CBB	2.22	124.03	120.99
3	A	502	POP	O6-P2-O4	2.21	119.35	110.68
2	A	501	HAK	CAN-CAO-NAP	-2.20	119.22	122.77
2	A	501	HAK	CAX-CAY-NAZ	-2.15	119.87	123.62
3	A	502	POP	O5-P2-O	2.13	111.77	104.64

There are no chirality outliers.

All (2) torsion outliers are listed below:

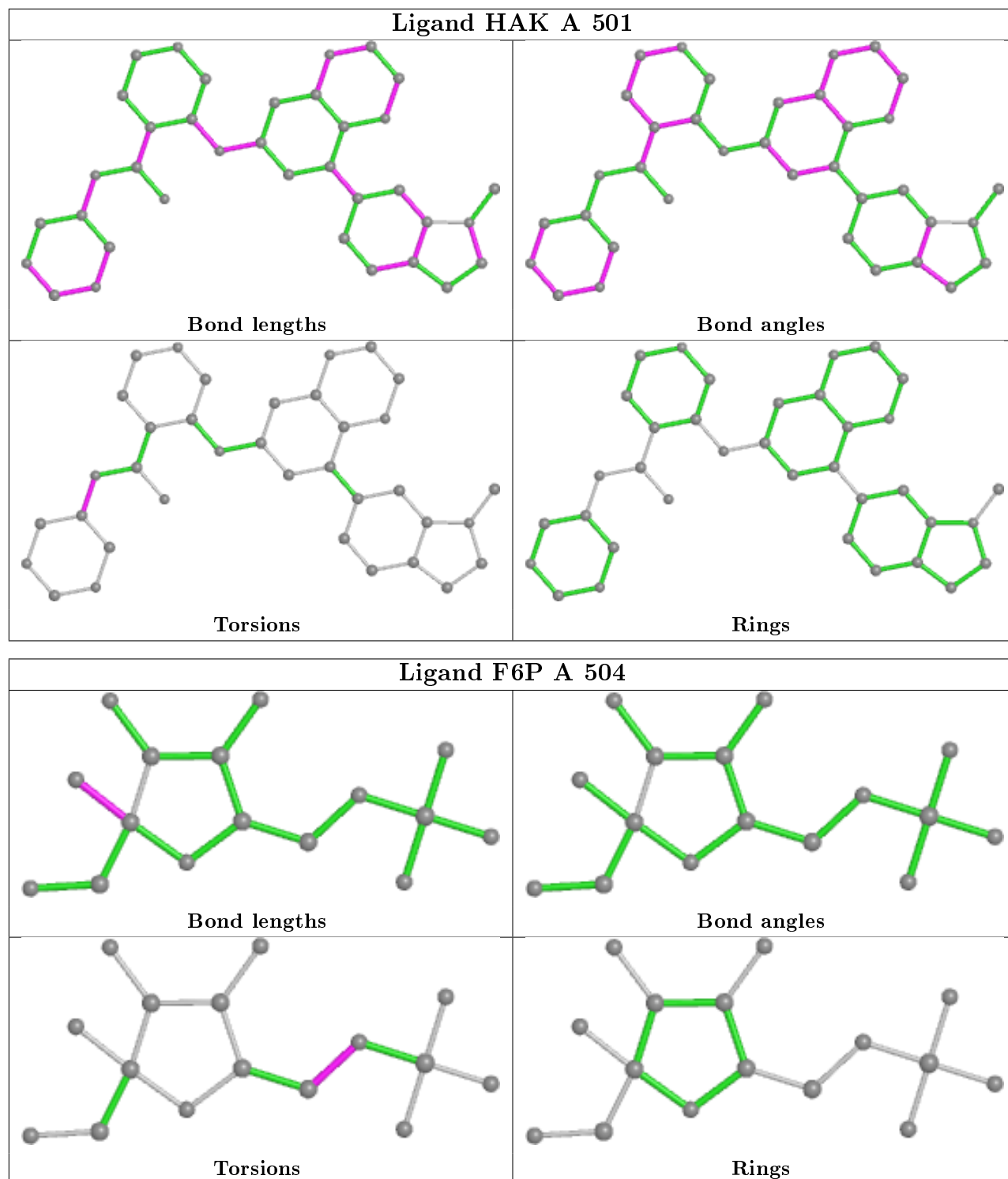
Mol	Chain	Res	Type	Atoms
5	A	504	F6P	C5-C6-O6-P
2	A	501	HAK	CBJ-CBE-NBC-CBB

There are no ring outliers.

1 monomer is involved in 2 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
4	A	503	FLC	2	0

The following is a two-dimensional graphical depiction of Mogul quality analysis of bond lengths, bond angles, torsion angles, and ring geometry for all instances of the Ligand of Interest. In addition, ligands with molecular weight > 250 and outliers as shown on the validation Tables will also be included. For torsion angles, if less than 5% of the Mogul distribution of torsion angles is within 10 degrees of the torsion angle in question, then that torsion angle is considered an outlier. Any bond that is central to one or more torsion angles identified as an outlier by Mogul will be highlighted in the graph. For rings, the root-mean-square deviation (RMSD) between the ring in question and similar rings identified by Mogul is calculated over all ring torsion angles. If the average RMSD is greater than 60 degrees and the minimal RMSD between the ring in question and any Mogul-identified rings is also greater than 60 degrees, then that ring is considered an outlier. The outliers are highlighted in purple. The color gray indicates Mogul did not find sufficient equivalents in the CSD to analyse the geometry.



## 5.7 Other polymers [i](#)

There are no such residues in this entry.

## 5.8 Polymer linkage issues

The following chains have linkage breaks:

Mol	Chain	Number of breaks
1	A	1

All chain breaks are listed below:

Model	Chain	Residue-1	Atom-1	Residue-2	Atom-2	Distance (Å)
1	A	16:VAL	C	32:ASN	N	24.72



## 6 Fit of model and data [i](#)

### 6.1 Protein, DNA and RNA chains [i](#)

In the following table, the column labelled ‘#RSRZ> 2’ contains the number (and percentage) of RSRZ outliers, followed by percent RSRZ outliers for the chain as percentile scores relative to all X-ray entries and entries of similar resolution. The OWAB column contains the minimum, median, 95<sup>th</sup> percentile and maximum values of the occupancy-weighted average B-factor per residue. The column labelled ‘Q< 0.9’ lists the number of (and percentage) of residues with an average occupancy less than 0.9.

Mol	Chain	Analysed	<RSRZ>	#RSRZ>2	OWAB(Å <sup>2</sup> )	Q<0.9
1	A	428/428 (100%)	-0.39	0 100 100	30, 44, 71, 100	0

There are no RSRZ outliers to report.

### 6.2 Non-standard residues in protein, DNA, RNA chains [i](#)

There are no non-standard protein/DNA/RNA residues in this entry.

### 6.3 Carbohydrates [i](#)

There are no monosaccharides in this entry.

### 6.4 Ligands [i](#)

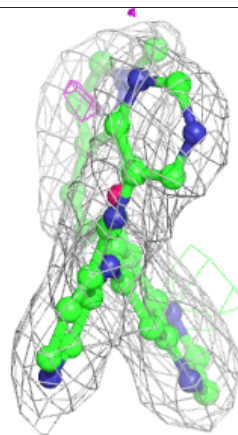
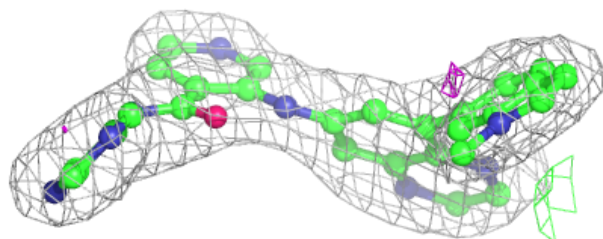
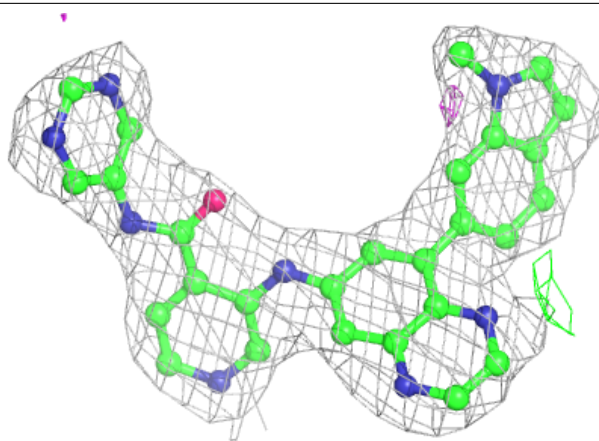
In the following table, the Atoms column lists the number of modelled atoms in the group and the number defined in the chemical component dictionary. The B-factors column lists the minimum, median, 95<sup>th</sup> percentile and maximum values of B factors of atoms in the group. The column labelled ‘Q< 0.9’ lists the number of atoms with occupancy less than 0.9.

Mol	Type	Chain	Res	Atoms	RSCC	RSR	B-factors(Å <sup>2</sup> )	Q<0.9
6	DMS	A	505	4/4	0.92	0.14	74,75,78,85	0
4	FLC	A	503	13/13	0.96	0.15	43,46,52,54	0
2	HAK	A	501	36/36	0.98	0.15	38,41,52,55	0
5	F6P	A	504	16/16	0.98	0.12	29,32,39,40	0
3	POP	A	502	9/9	0.99	0.12	34,36,40,42	0

The following is a graphical depiction of the model fit to experimental electron density of all instances of the Ligand of Interest. In addition, ligands with molecular weight > 250 and outliers as shown on the geometry validation Tables will also be included. Each fit is shown from different orientation to approximate a three-dimensional view.

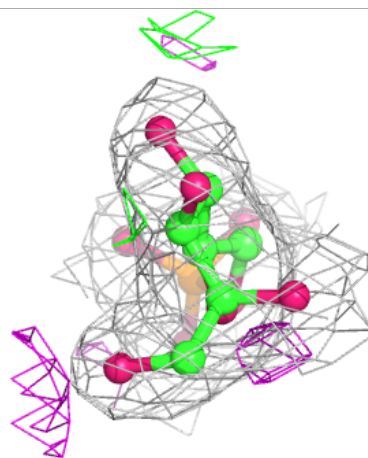
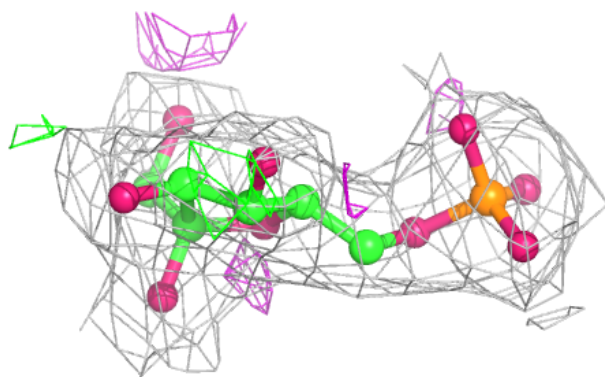
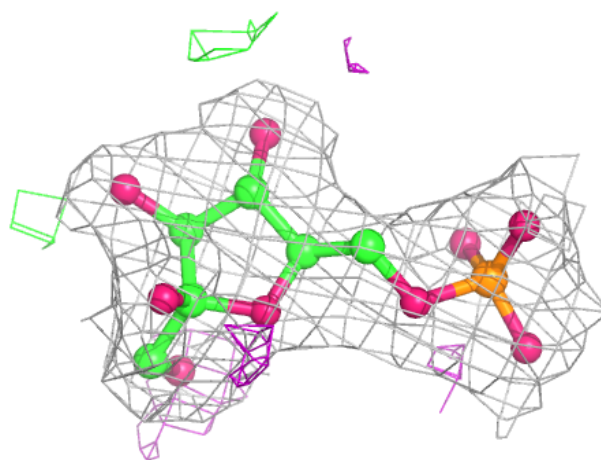
**Electron density around HAK A 501:**

$2mF_o - DF_c$  (at 0.7 rmsd) in gray  
 $mF_o - DF_c$  (at 3 rmsd) in purple (negative)  
and green (positive)



**Electron density around F6P A 504:**

$2mF_o-DF_c$  (at 0.7 rmsd) in gray  
 $mF_o-DF_c$  (at 3 rmsd) in purple (negative)  
and green (positive)



## 6.5 Other polymers [i](#)

There are no such residues in this entry.