



wwPDB X-ray Structure Validation Summary Report ⓘ

Feb 1, 2016 – 10:09 AM GMT

PDB ID : 3L1S
Title : 3-Aryl-4-(arylhydrazono)-1H-pyrazol-5-ones: Highly ligand efficient and potent inhibitors of GSK3
Authors : Haar, T.E.
Deposited on : 2009-12-14
Resolution : 2.90 Å(reported)

This is a wwPDB X-ray Structure Validation Summary Report for a publicly released PDB entry.
We welcome your comments at validation@mail.wwpdb.org
A user guide is available at
<http://wwpdb.org/validation/2016/XrayValidationReportHelp>
with specific help available everywhere you see the ⓘ symbol.

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

MolProbity : 4.02b-467
Mogul : 1.7 (RC4), CSD as536be (2015)
Xtriage (Phenix) : 1.9-1692
EDS : rb-20026688
Percentile statistics : 20151230.v01 (using entries in the PDB archive December 30th 2015)
Refmac : 5.8.0135
CCP4 : 6.5.0
Ideal geometry (proteins) : Engh & Huber (2001)
Ideal geometry (DNA, RNA) : Parkinson et al. (1996)
Validation Pipeline (wwPDB-VP) : trunk26865

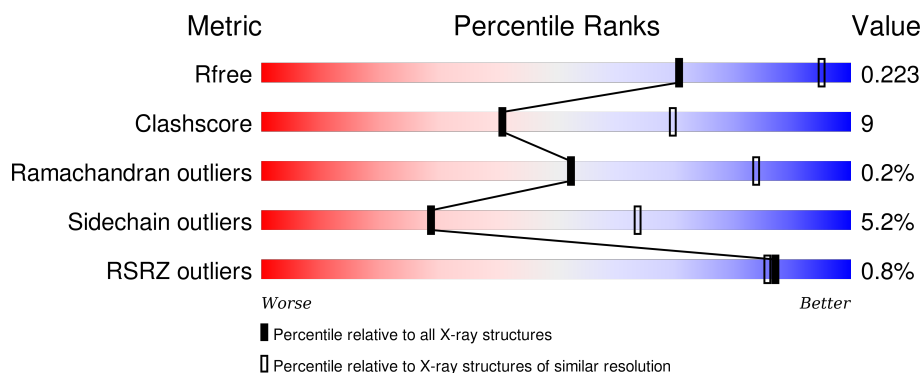
1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

X-RAY DIFFRACTION

The reported resolution of this entry is 2.90 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}	91344	1451 (2.90-2.90)
Clashscore	102246	1668 (2.90-2.90)
Ramachandran outliers	100387	1630 (2.90-2.90)
Sidechain outliers	100360	1632 (2.90-2.90)
RSRZ outliers	91569	1456 (2.90-2.90)

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 ≥ 3 , 2, 1 and 0 types of geometric quality criteria. 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	414	 71% 10% • 19%
1	B	414	 66% 12% • 20%

The following table lists non-polymeric compounds, carbohydrate monomers and non-standard residues in protein, DNA, RNA chains that are outliers for geometric or electron-density-fit criteria:

Mol	Type	Chain	Res	Chirality	Geometry	Clashes	Electron density
2	Z92	A	421	-	-	X	-

2 Entry composition [i](#)

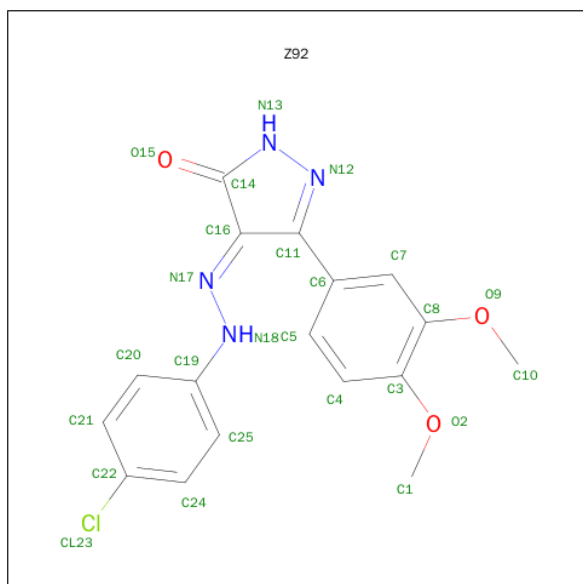
There are 4 unique types of molecules in this entry. The entry contains 5617 atoms, of which 0 are hydrogens and 0 are deuteriums.

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 Glycogen synthase kinase-3 beta.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	336	Total	C	N	O	S	0	0	0
			2600	1671	441	476	12			
1	B	330	Total	C	N	O	S	0	0	0
			2555	1646	427	471	11			

- Molecule 2 is (4E)-4-[(4-CHLOROPHENYL)HYDRAZONO]-5-(3,4-DIMETHOXY PHENYL)-2,4-DIHYDRO-3H-PYRAZOL-3-ONE (three-letter code: Z92) (formula: C₁₇H₁₅ClN₄O₃).



Mol	Chain	Residues	Atoms					ZeroOcc	AltConf
2	A	1	Total	C	Cl	N	O	0	0
			25	17	1	4	3		
2	B	1	Total	C	Cl	N	O	0	0
			25	17	1	4	3		

- Molecule 3 is PHOSPHATE ION (three-letter code: PO4) (formula: O₄P).



Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
3	B	1	Total	O	P	0	0
			5	4	1		

- Molecule 4 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	A	204	Total	O	0	0
			204	204		
4	B	203	Total	O	0	0
			203	203		

4 Data and refinement statistics

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants a, b, c, α , β , γ	82.60Å 84.80Å 178.00Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	39.18 – 2.90 39.18 – 2.90	Depositor EDS
% Data completeness (in resolution range)	(Not available) (39.18-2.90) 93.4 (39.18-2.90)	Depositor EDS
R_{merge}	0.09	Depositor
R_{sym}	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ ¹	3.42 (at 2.90Å)	Xtriage
Refinement program	BUSTER 2.9.2	Depositor
R, R_{free}	0.184 , 0.232 0.184 , 0.223	Depositor DCC
R_{free} test set	1335 reflections (5.29%)	DCC
Wilson B-factor (Å ²)	38.2	Xtriage
Anisotropy	0.551	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.32 , 59.4	EDS
Estimated twinning fraction	0.026 for k,h,l	Xtriage
L-test for twinning ²	$\langle L \rangle = 0.49$, $\langle L^2 \rangle = 0.32$	Xtriage
Outliers	0 of 26568 reflections	Xtriage
F_o, F_c correlation	0.93	EDS
Total number of atoms	5617	wwPDB-VP
Average B, all atoms (Å ²)	38.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 3.37% of the height of the origin peak. No significant pseudotranslation is detected.*

¹Intensities estimated from amplitudes.

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

5 Model quality

5.1 Standard geometry

Bond lengths and bond angles in the following residue types are not validated in this section: PO4, Z92

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	0.47	0/2661	0.66	2/3627 (0.1%)
1	B	0.52	0/2616	0.73	5/3572 (0.1%)
All	All	0.50	0/5277	0.70	7/7199 (0.1%)

There are no bond length outliers.

The worst 5 of 7 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
1	B	221	TYR	CB-CA-C	-6.53	97.33	110.40
1	B	122	LYS	CB-CA-C	-6.41	97.59	110.40
1	B	46	GLN	CB-CA-C	-6.12	98.17	110.40
1	A	221	TYR	CB-CA-C	-5.78	98.84	110.40
1	A	189	LEU	CA-CB-CG	5.57	128.10	115.30

There are no chirality outliers.

There are no planarity outliers.

5.2 Too-close contacts

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	2600	0	2558	45	0
1	B	2555	0	2514	37	0
2	A	25	0	15	21	0
2	B	25	0	15	5	0

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Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
3	B	5	0	0	0	0
4	A	204	0	0	2	0
4	B	203	0	0	0	0
All	All	5617	0	5102	89	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 9.

The worst 5 of 89 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:85:LYS:NZ	2:A:421:Z92:C1	2.10	1.14
1:A:85:LYS:CD	2:A:421:Z92:H1A	1.76	1.13
2:A:421:Z92:H5	2:A:421:Z92:HN18	1.11	1.13
1:A:85:LYS:HZ2	2:A:421:Z92:H1	1.02	1.10
1:A:85:LYS:HD2	2:A:421:Z92:H1A	1.12	1.06

There are no symmetry-related clashes.

5.3 Torsion angles [i](#)

5.3.1 Protein backbone [i](#)

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	328/414 (79%)	318 (97%)	10 (3%)	0	100	100
1	B	324/414 (78%)	312 (96%)	11 (3%)	1 (0%)	46	79
All	All	652/828 (79%)	630 (97%)	21 (3%)	1 (0%)	52	84

All (1) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	B	124	ASP

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	280/359 (78%)	269 (96%)	11 (4%)	39	75
1	B	276/359 (77%)	258 (94%)	18 (6%)	21	52
All	All	556/718 (77%)	527 (95%)	29 (5%)	29	64

5 of 29 residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	B	50	ARG
1	B	104	LEU
1	B	312	GLU
1	B	53	GLU
1	B	122	LYS

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. 5 of 11 such sidechains are listed below:

Mol	Chain	Res	Type
1	A	352	ASN
1	B	52	GLN
1	B	185	GLN
1	A	337	HIS
1	B	164	GLN

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 carbohydrates in this entry.

5.6 Ligand geometry ⓘ

3 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$
2	Z92	A	421	-	26,27,27	2.25	6 (23%)	34,37,37	2.67	14 (41%)
3	PO4	B	421	-	4,4,4	2.06	2 (50%)	6,6,6	0.35	0
2	Z92	B	422	-	26,27,27	2.38	10 (38%)	34,37,37	2.80	20 (58%)

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
2	Z92	A	421	-	-	0/13/26/26	0/3/3/3
3	PO4	B	421	-	-	0/0/0/0	0/0/0/0
2	Z92	B	422	-	-	0/13/26/26	0/3/3/3

The worst 5 of 18 bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	B	422	Z92	C11-N12	-5.85	1.26	1.34
2	A	421	Z92	C16-C14	-5.61	1.39	1.50
2	A	421	Z92	C11-N12	-5.52	1.27	1.34
2	B	422	Z92	C16-C14	-4.50	1.41	1.50
2	B	422	Z92	C16-N17	-3.55	1.27	1.31

The worst 5 of 34 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed($^{\circ}$)	Ideal($^{\circ}$)
2	A	421	Z92	C14-C16-N17	-6.63	117.33	127.64
2	A	421	Z92	C14-N13-N12	-5.75	108.24	113.63
2	A	421	Z92	O15-C14-C16	-5.59	121.34	126.89
2	B	422	Z92	C14-N13-N12	-5.54	108.44	113.63
2	B	422	Z92	O9-C8-C7	-5.00	115.68	124.21

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

2 monomers are involved in 26 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
2	A	421	Z92	21	0
2	B	422	Z92	5	0

5.7 Other polymers [i](#)

There are no such residues in this entry.

5.8 Polymer linkage issues [i](#)

There are no chain breaks in this entry.

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, 95th 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 ²)	Q<0.9
1	A	336/414 (81%)	-0.36	3 (0%) 85 84	18, 36, 73, 134	0
1	B	330/414 (79%)	-0.50	2 (0%) 90 89	16, 32, 61, 83	0
All	All	666/828 (80%)	-0.43	5 (0%) 87 86	16, 34, 68, 134	0

All (5) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	90	ASP	4.1
1	A	300	PRO	2.4
1	B	124	ASP	2.4
1	A	210	GLY	2.0
1	B	148	ARG	2.0

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 carbohydrates 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. LLDF column lists the quality of electron density of the group with respect to its neighbouring residues in protein, DNA or RNA chains. The B-factors column lists the minimum, median, 95th 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	LLDF	B-factors(\AA^2)	Q<0.9
3	PO4	B	421	5/5	0.88	0.23	1.96	79,82,85,86	0
2	Z92	A	421	25/25	0.92	0.20	1.56	19,34,51,63	0
2	Z92	B	422	25/25	0.94	0.17	1.00	27,34,42,47	0

6.5 Other polymers [i](#)

There are no such residues in this entry.