



# wwPDB X-ray Structure Validation Summary Report ⓘ

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PDB ID : 4BIE / pdb\_00004bie  
Title : Crystal Structures of Ask1-inhibitor Complexes  
Authors : Singh, O.; Shillings, A.; Craggs, P.; Wall, I.; Rowland, P.; Skarzynski, T.;  
Hobbs, C.I.; Hardwick, P.; Tanner, R.; Blunt, M.; Witty, D.R.; Smith, K.J.  
Deposited on : 2013-04-10  
Resolution : 2.36 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](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.

The types of validation reports are described at

<http://www.wwpdb.org/validation/2017/FAQs#types>.

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

MolProbity : 4-5-2 with Phenix2.0  
Mogul : 2022.3.0, CSD as543be (2022)  
Xtrriage (Phenix) : 2.0  
EDS : 3.0  
Buster-report : wwPDB partial adaption of 1.1.7 (2018)  
Percentile statistics : 20250101.v01 (using entries in the PDB archive January 1st 2025)  
CCP4 : 9.0.010 (Gargrove)  
Density-Fitness : 1.0.12  
Ideal geometry (proteins) : Engh & Huber (2001)  
Ideal geometry (DNA, RNA) : Parkinson et al. (1996)  
Validation Pipeline (wwPDB-VP) : 2.49

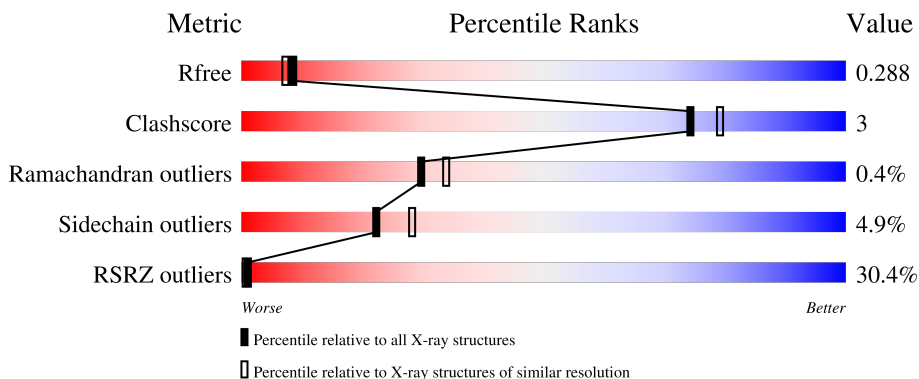
# 1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

*X-RAY DIFFRACTION*

The reported resolution of this entry is 2.36 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))
$R_{free}$	180053	1596 (2.36-2.36)
Clashscore	190562	1663 (2.36-2.36)
Ramachandran outliers	187476	1646 (2.36-2.36)
Sidechain outliers	187428	1646 (2.36-2.36)
RSRZ outliers	180081	1598 (2.36-2.36)

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 of 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	318	
1	B	318	

## 2 Entry composition [i](#)

There are 4 unique types of molecules in this entry. The entry contains 4233 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.

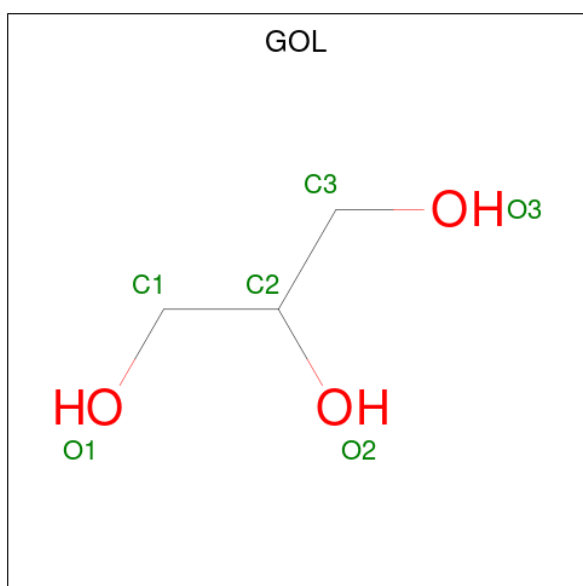
- Molecule 1 is a protein called MITOGEN-ACTIVATED PROTEIN KINASE KINASE KINASE 5.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
			Total	C	N	O	S			
1	A	257	Total 2003	C 1292	N 327	O 375	S 9	0	0	0
1	B	257	Total 1982	C 1274	N 321	O 378	S 9	0	1	0

There are 2 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	838	GLU	THR	engineered mutation	UNP Q99683
B	838	GLU	THR	engineered mutation	UNP Q99683

- Molecule 2 is GLYCEROL (CCD ID: GOL) (formula: C<sub>3</sub>H<sub>8</sub>O<sub>3</sub>).



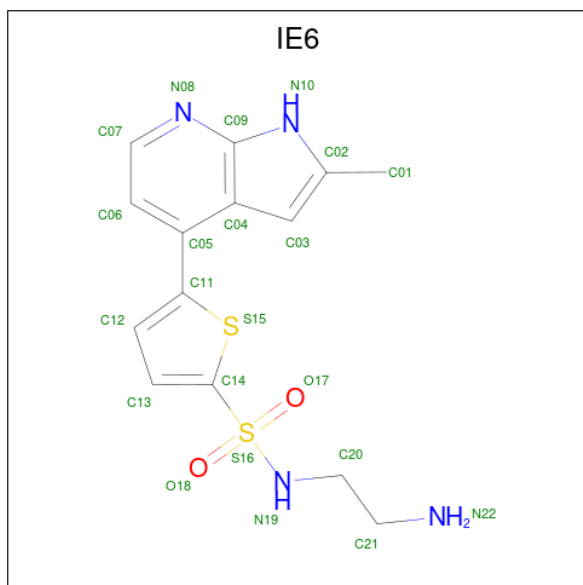
Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
			Total	C	O		
2	A	1	Total 6	C 3	O 3	0	0

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Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
2	B	1	Total	C	O	0	0
			6	3	3		

- Molecule 3 is N-(2-aminoethyl)-5-{2-methyl-1H-pyrrolo[2,3-b]pyridin-4-yl}thiophene-2-sulfonamide (CCD ID: IE6) (formula: C<sub>14</sub>H<sub>16</sub>N<sub>4</sub>O<sub>2</sub>S<sub>2</sub>).



Mol	Chain	Residues	Atoms					ZeroOcc	AltConf
3	A	1	Total	C	N	O	S	0	0
			22	14	4	2	2		
3	B	1	Total	C	N	O	S	0	0
			22	14	4	2	2		

- Molecule 4 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	A	117	Total	O	0	0
			117	117		
4	B	75	Total	O	0	0
			75	75		



## 4 Data and refinement statistics

Property	Value	Source
Space group	P 65 2 2	Depositor
Cell constants a, b, c, $\alpha$ , $\beta$ , $\gamma$	77.95Å 77.95Å 418.43Å 90.00° 90.00° 120.00°	Depositor
Resolution (Å)	67.88 – 2.36 67.88 – 2.36	Depositor EDS
% Data completeness (in resolution range)	93.3 (67.88-2.36) 93.4 (67.88-2.36)	Depositor EDS
$R_{merge}$	0.07	Depositor
$R_{sym}$	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ <sup>1</sup>	2.06 (at 2.34Å)	Xtrriage
Refinement program	BUSTER 2.11.1	Depositor
R, $R_{free}$	0.210 , 0.253 0.252 , 0.288	Depositor DCC
$R_{free}$ test set	1550 reflections (5.03%)	wwPDB-VP
Wilson B-factor (Å <sup>2</sup> )	44.0	Xtrriage
Anisotropy	0.006	Xtrriage
Bulk solvent $k_{sol}$ (e/Å <sup>3</sup> ), $B_{sol}$ (Å <sup>2</sup> )	0.36 , 56.6	EDS
L-test for twinning <sup>2</sup>	$\langle  L  \rangle = 0.49$ , $\langle L^2 \rangle = 0.33$	Xtrriage
Estimated twinning fraction	No twinning to report.	Xtrriage
$F_o, F_c$ correlation	0.92	EDS
Total number of atoms	4233	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	50.0	wwPDB-VP

Xtrriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 4.06% 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: IE6, GOL

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	0.89	1/2045 (0.0%)	1.36	12/2758 (0.4%)
1	B	0.82	0/2027	1.37	11/2740 (0.4%)
All	All	0.85	1/4072 (0.0%)	1.37	23/5498 (0.4%)

All (1) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	A	879	PRO	CA-C	5.10	1.54	1.51

The worst 5 of 23 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
1	A	671	LEU	N-CA-C	7.97	120.62	109.15
1	B	670	LEU	CA-C-N	7.62	131.56	120.82
1	B	670	LEU	C-N-CA	7.62	131.56	120.82
1	A	824	GLY	CA-C-N	6.94	129.59	120.28
1	A	824	GLY	C-N-CA	6.94	129.59	120.28

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	2003	0	1971	13	0
1	B	1982	0	1919	11	0
2	A	6	0	8	0	0
2	B	6	0	8	1	0
3	A	22	0	16	3	0
3	B	22	0	16	1	0
4	A	117	0	0	1	0
4	B	75	0	0	1	0
All	All	4233	0	3938	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 3.

The worst 5 of 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:B:869:CYS:HB3	1:B:880:PRO:HG3	1.82	0.61
1:B:710:GLU:HG2	1:B:751:LYS:HG2	1.87	0.57
1:B:679:GLU:CD	1:B:679:GLU:H	2.13	0.56
1:A:869:CYS:HB3	1:A:880:PRO:HG3	1.91	0.52
3:B:1943:IE6:H13	4:B:2020:HOH:O	2.12	0.50

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	251/318 (79%)	243 (97%)	7 (3%)	1 (0%)	30	34
1	B	252/318 (79%)	245 (97%)	6 (2%)	1 (0%)	30	34
All	All	503/636 (79%)	488 (97%)	13 (3%)	2 (0%)	30	34

All (2) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	A	822	ASP
1	B	822	ASP

### 5.3.2 Protein sidechains [i](#)

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	208/274 (76%)	197 (95%)	11 (5%)	20	25
1	B	205/274 (75%)	196 (96%)	9 (4%)	25	33
All	All	413/548 (75%)	393 (95%)	20 (5%)	22	29

5 of 20 residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	B	733	LYS
1	B	886	GLU
1	B	916	LEU
1	B	910	GLU
1	A	838	GLU

Sometimes sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (4) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	703	GLN
1	A	756	GLN
1	B	756	GLN
1	B	776	ASN

### 5.3.3 RNA [i](#)

There are no RNA molecules in this entry.

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

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

## 5.5 Carbohydrates [i](#)

There are no oligosaccharides in this entry.

## 5.6 Ligand geometry [i](#)

4 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	GOL	A	1941	-	5,5,5	0.52	0	5,5,5	1.51	1 (20%)
3	IE6	B	1943	-	24,24,24	2.98	8 (33%)	30,35,35	2.92	9 (30%)
2	GOL	B	1942	-	5,5,5	0.30	0	5,5,5	0.68	0
3	IE6	A	1942	-	24,24,24	3.00	9 (37%)	30,35,35	3.14	12 (40%)

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. '2' means no outliers of that kind were identified.

Mol	Type	Chain	Res	Link	Chirals	Torsions	Rings
2	GOL	A	1941	-	-	1/4/4/4	-
3	IE6	B	1943	-	-	3/15/15/15	0/3/3/3
2	GOL	B	1942	-	-	3/4/4/4	-
3	IE6	A	1942	-	-	5/15/15/15	0/3/3/3

The worst 5 of 17 bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
3	A	1942	IE6	S16-N19	7.73	1.73	1.61

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Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
3	B	1943	IE6	S16-N19	7.09	1.72	1.61
3	A	1942	IE6	C07-N08	6.28	1.47	1.34
3	B	1943	IE6	C07-N08	6.18	1.47	1.34
3	B	1943	IE6	C04-C09	-5.54	1.34	1.41

The worst 5 of 22 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
3	B	1943	IE6	C03-C02-N10	8.16	113.92	107.60
3	A	1942	IE6	C06-C07-N08	-8.06	114.09	123.97
3	A	1942	IE6	C03-C02-N10	7.82	113.65	107.60
3	B	1943	IE6	C06-C07-N08	-7.24	115.10	123.97
3	B	1943	IE6	O18-S16-N19	7.16	114.10	107.41

There are no chirality outliers.

5 of 12 torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
3	B	1943	IE6	N19-C20-C21-N22
3	A	1942	IE6	C20-N19-S16-O18
3	A	1942	IE6	C20-N19-S16-C14
2	B	1942	GOL	O1-C1-C2-C3
2	B	1942	GOL	C1-C2-C3-O3

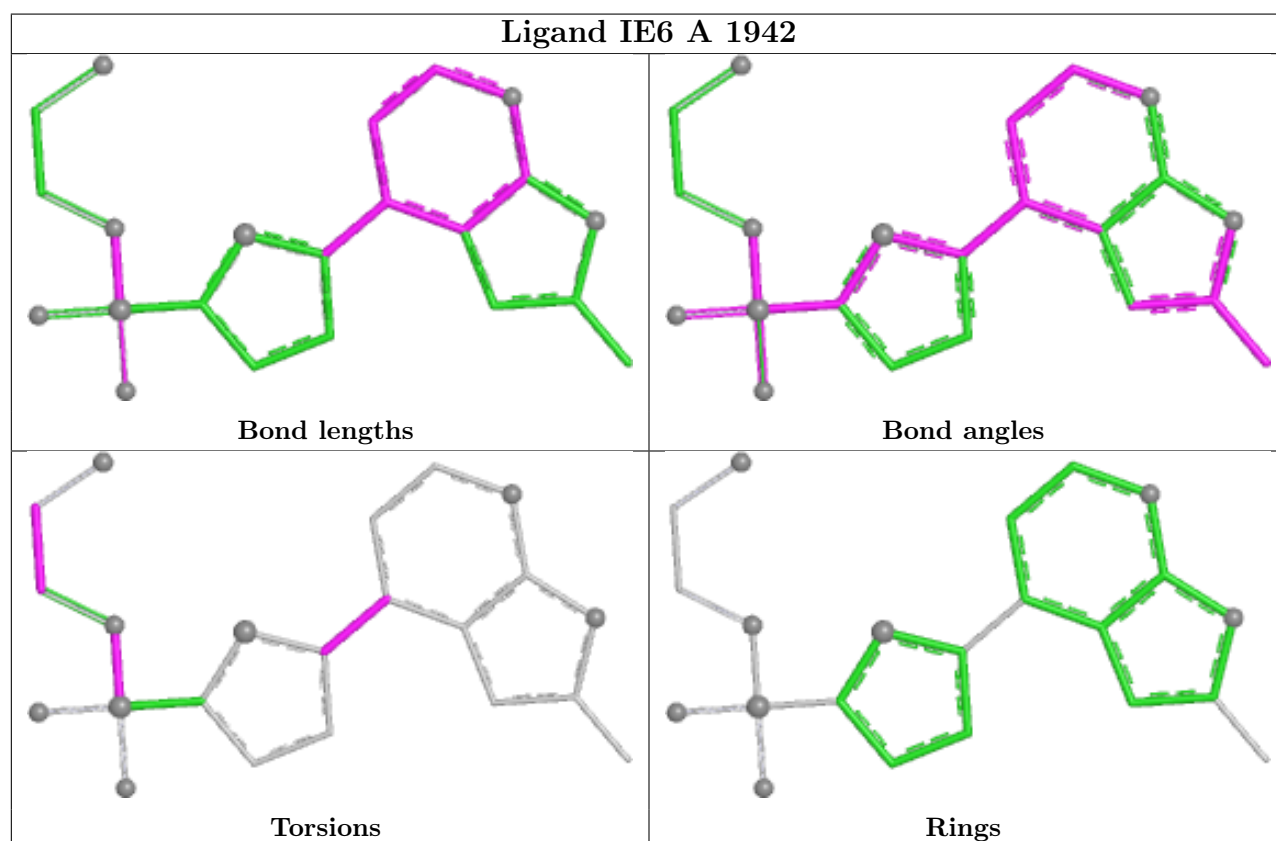
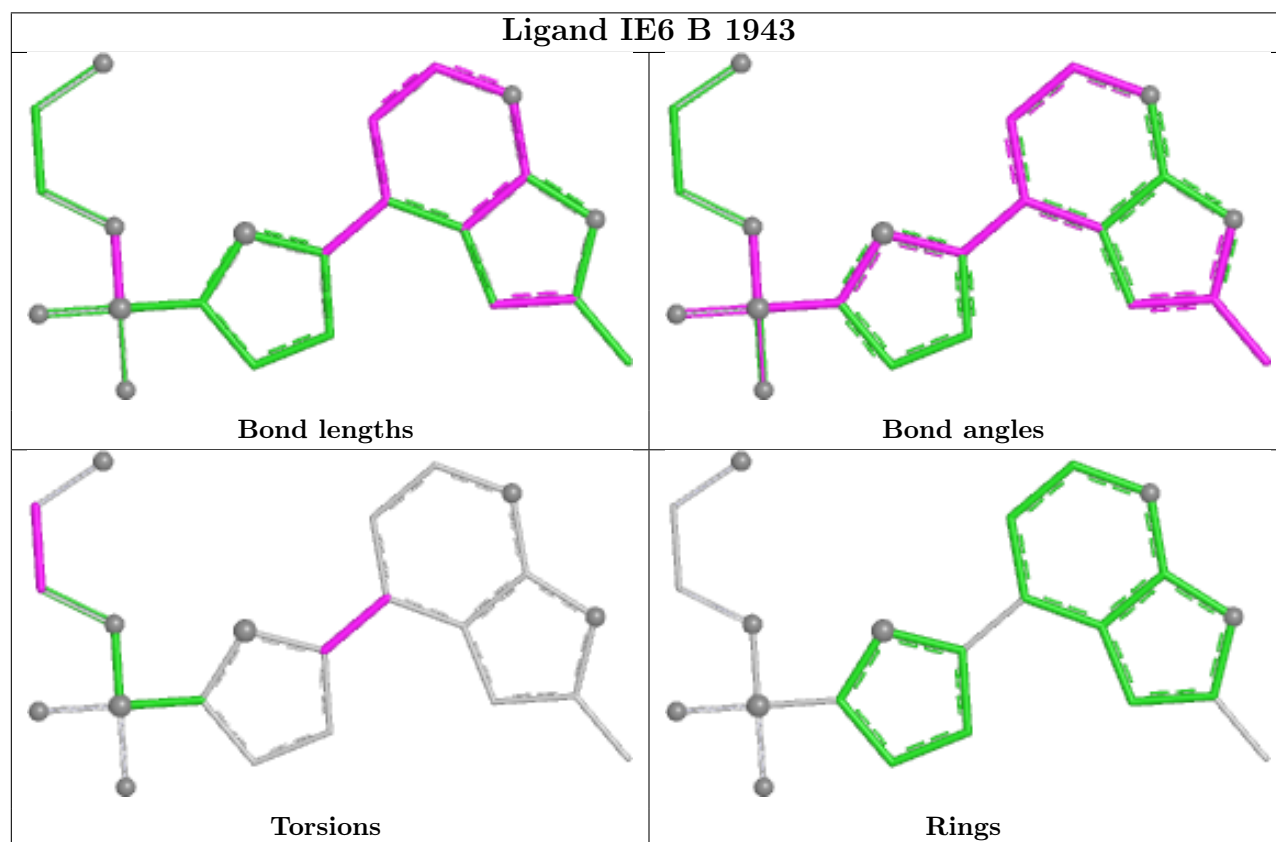
There are no ring outliers.

3 monomers are involved in 5 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
3	B	1943	IE6	1	0
2	B	1942	GOL	1	0
3	A	1942	IE6	3	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 [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, 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	257/318 (80%)	1.11	37 (14%) <b>6</b> <b>7</b>	24, 41, 76, 101	0
1	B	257/318 (80%)	2.15	119 (46%) <b>0</b> <b>0</b>	31, 54, 92, 110	1 (0%)
All	All	514/636 (80%)	1.63	156 (30%) <b>1</b> <b>1</b>	24, 47, 88, 110	1 (0%)

The worst 5 of 156 RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	B	722	LEU	8.3
1	B	884	LEU	7.6
1	B	691	TYR	6.9
1	A	940	VAL	6.9
1	A	670	LEU	6.5

### 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](#)

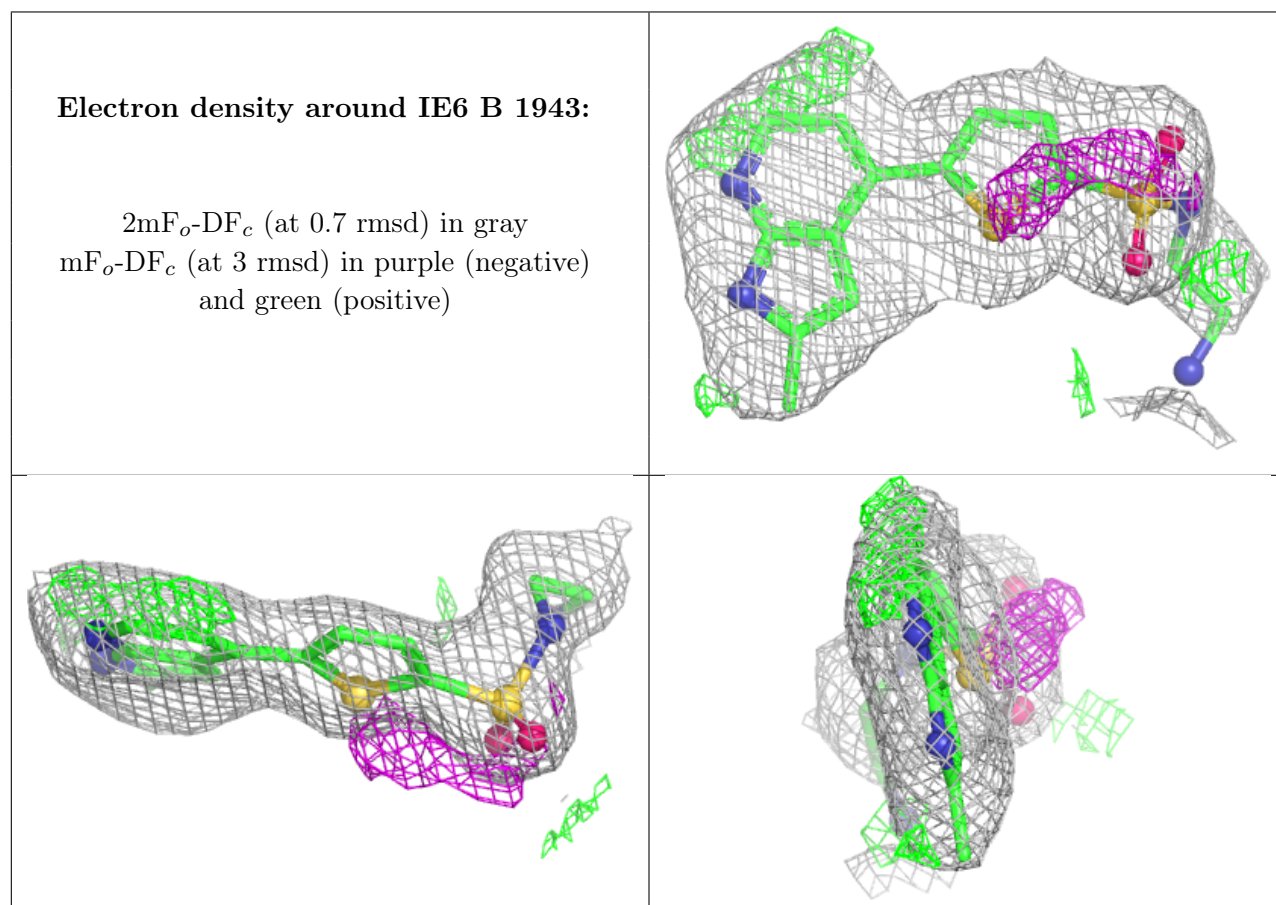
There are no oligosaccharides in this entry.

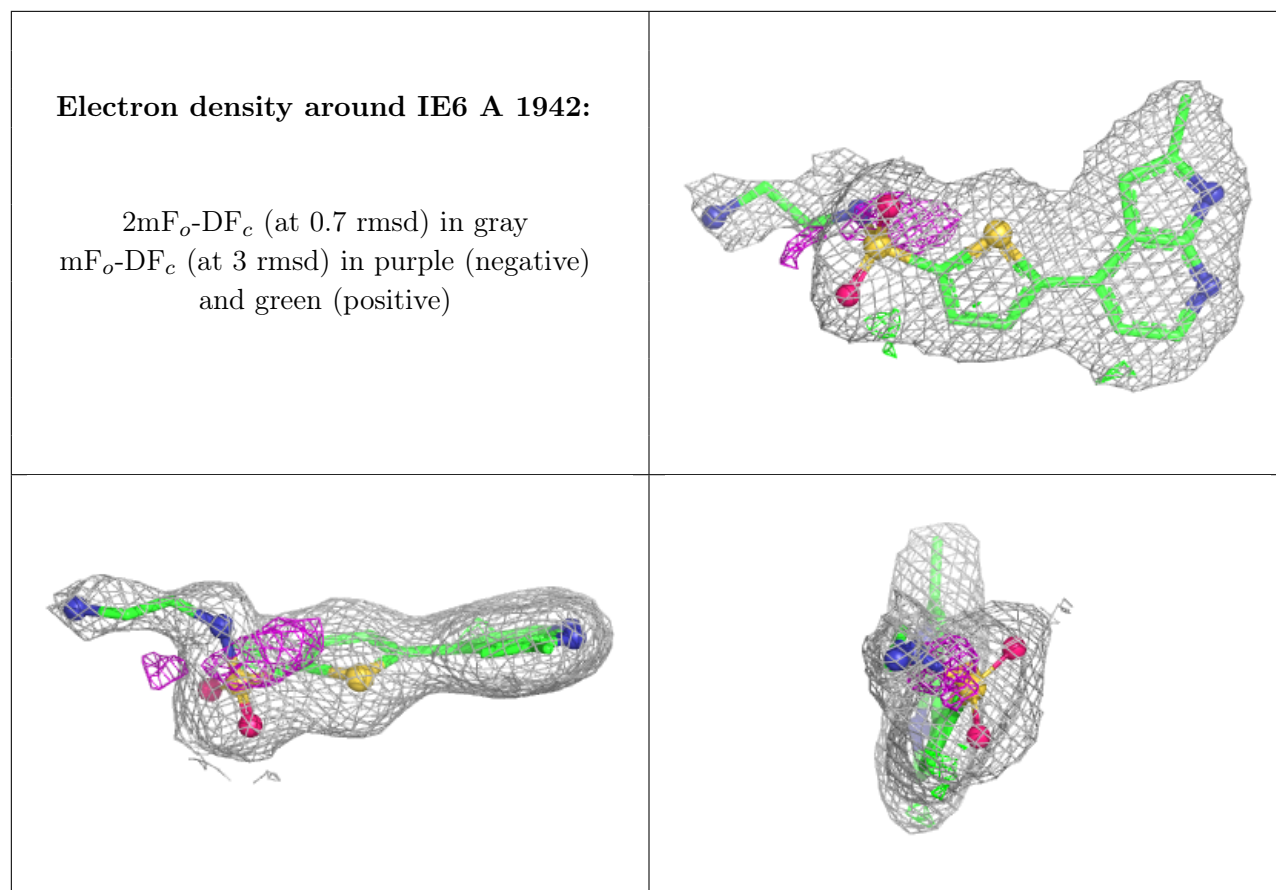
### 6.4 Ligands [i](#)

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( $\text{\AA}^2$ )	Q<0.9
3	IE6	B	1943	22/22	0.87	0.15	34,44,58,67	0
2	GOL	B	1942	6/6	0.90	0.17	53,55,55,57	0
3	IE6	A	1942	22/22	0.92	0.12	27,35,61,63	0
2	GOL	A	1941	6/6	0.93	0.12	49,50,51,51	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.





## 6.5 Other polymers [i](#)

There are no such residues in this entry.