

wwPDB X-ray Structure Validation Summary Report (i)

Oct 24, 2023 – 11:06 AM EDT

PDB ID : 3DEK

Title: Crystal Structures of Caspase-3 with Bound Isoquinoline-1,3,4-trione Deriva-

tive Inhibitors

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Deposited on : 2008-06-10

Resolution : 2.40 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

https://www.wwpdb.org/validation/2017/XrayValidationReportHelp with specific help available everywhere you see the (i) symbol.

The types of validation reports are described at http://www.wwpdb.org/validation/2017/FAQs#types.

The following versions of software and data (see references (1)) 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 : 2.36

buster-report : 1.1.7 (2018)

Percentile statistics : 20191225.v01 (using entries in the PDB archive December 25th 2019)

 $Refmac \quad : \quad 5.8.0158$

CCP4 : 7.0.044 (Gargrove)

Ideal geometry (proteins) : Engh & Huber (2001) Ideal geometry (DNA, RNA) : Parkinson et al. (1996)

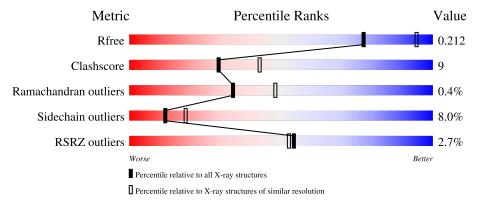
Validation Pipeline (wwPDB-VP) : 2.36

1 Overall quality at a glance (i)

The following experimental techniques were used to determine the structure: X- $RAY\ DIFFRACTION$

The reported resolution of this entry is 2.40 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	Similar resolution
Metric	$(\# ext{Entries})$	$(\# ext{Entries}, ext{ resolution range}(ext{Å}))$
R_{free}	130704	3907 (2.40-2.40)
Clashscore	141614	4398 (2.40-2.40)
Ramachandran outliers	138981	4318 (2.40-2.40)
Sidechain outliers	138945	4319 (2.40-2.40)
RSRZ outliers	127900	3811 (2.40-2.40)

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 >=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 <=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	249	69%	21%	•	6%		
1	В	249	73%	19%		6%		
1	С	249	72%	19%		6%		
1	D	249	6%	26%	5%	6%		

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	RXD	A	300	-	-	X	-



2 Entry composition (i)

There are 3 unique types of molecules in this entry. The entry contains 7871 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 Caspase-3.

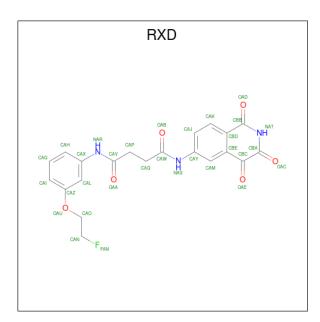
Mol	Chain	Residues		Atoms				ZeroOcc	AltConf	Trace
1	Λ	233	Total	С	N	О	S	0	0	0
1	A	233	1890	1196	324	356	14	0	U	
1	В	235	Total	С	N	О	S	0	0	0
1	Б	233	1908	1206	328	360	14	0	0	
1	С	235	Total	С	N	О	S	0	0	0
1		233	1908	1206	328	360	14	0	0	
1	D	233	Total	С	N	О	S	0	0	0
1	D	∠33	1893	1198	326	355	14	U	U	U

There are 4 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	190	ASP	GLU	SEE REMARK 999	UNP P42574
В	190	ASP	GLU	SEE REMARK 999	UNP P42574
С	190	ASP	GLU	SEE REMARK 999	UNP P42574
D	190	ASP	GLU	SEE REMARK 999	UNP P42574

• Molecule 2 is N-[3-(2-fluoroethoxy)phenyl]-N'-(1,3,4-trioxo-1,2,3,4-tetrahydroisoquinolin-6-y l)butanediamide (three-letter code: RXD) (formula: $C_{21}H_{18}FN_3O_6$).





Mol	Chain	Residues	Atoms			ZeroOcc	AltConf		
9	Λ	1	Total	С	F	N	О	0	0
2	A	1	31	21	1	3	6	0	0

• Molecule 3 is water.

Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
3	A	88	Total O 88 88	0	0
3	В	72	Total O 72 72	0	0
3	С	59	Total O 59 59	0	0
3	D	22	Total O 22 22	0	0

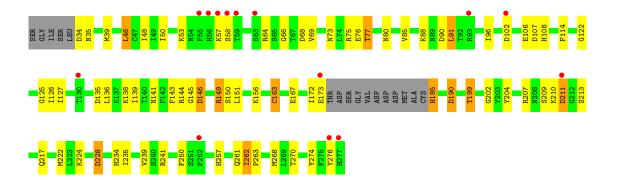


3 Residue-property plots (i)

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: Caspase-3







4 Data and refinement statistics (i)

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants	65.17Å 96.31Å 180.24Å	Depositor
a, b, c, α , β , γ	90.00° 90.00° 90.00°	Depositor
Resolution (Å)	46.31 - 2.40	Depositor
rtesolution (A)	46.30 - 2.40	EDS
% Data completeness	91.6 (46.31-2.40)	Depositor
(in resolution range)	91.6 (46.30-2.40)	EDS
R_{merge}	0.09	Depositor
R_{sym}	(Not available)	Depositor
$< I/\sigma(I) > 1$	1.80 (at 2.39Å)	Xtriage
Refinement program	REFMAC 5.1.24	Depositor
D.D.	0.209 , 0.253	Depositor
R, R_{free}	0.214 , 0.212	DCC
R_{free} test set	2094 reflections (5.06%)	wwPDB-VP
Wilson B-factor (Å ²)	43.0	Xtriage
Anisotropy	0.135	Xtriage
Bulk solvent $k_{sol}(e/Å^3)$, $B_{sol}(Å^2)$	0.32, 39.1	EDS
L-test for twinning ²	$ < L > = 0.49, < L^2> = 0.32$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
F_o, F_c correlation	0.94	EDS
Total number of atoms	7871	wwPDB-VP
Average B, all atoms $(Å^2)$	55.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: The largest off-origin peak in the Patterson function is 4.83% of the height of the origin peak. No significant pseudotranslation is detected.

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



¹Intensities estimated from amplitudes.

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: OCS, RXD

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		
10101		RMSZ	# Z > 5	RMSZ	# Z >5	
1	A	0.50	0/1919	0.78	8/2576 (0.3%)	
1	В	0.48	0/1938	0.78	6/2602~(0.2%)	
1	С	0.45	0/1938	0.77	7/2602~(0.3%)	
1	D	0.39	0/1923	0.72	$9/2581 \; (0.3\%)$	
All	All	0.46	0/7718	0.76	30/10361 (0.3%)	

There are no bond length outliers.

The worst 5 of 30 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	\mathbf{Z}	$Observed(^o)$	$\operatorname{Ideal}({}^{o})$
1	В	190	ASP	CB-CG-OD2	8.45	125.91	118.30
1	A	135	ASP	CB-CG-OD2	6.93	124.53	118.30
1	D	190	ASP	CB-CG-OD2	6.91	124.52	118.30
1	С	135	ASP	CB-CG-OD2	6.86	124.47	118.30
1	С	102	ASP	CB-CG-OD2	6.57	124.21	118.30

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	1890	0	1858	47	0
1	В	1908	0	1870	39	0



Continued	trom	mmoninonic	maaa
COHABABACA		DIEUIUU	DUIUE
0 0 1000100000			

Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
1	С	1908	0	1869	31	0
1	D	1893	0	1859	38	0
2	A	31	0	18	12	0
3	A	88	0	0	4	0
3	В	72	0	0	1	0
3	С	59	0	0	0	0
3	D	22	0	0	1	0
All	All	7871	0	7474	143	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 143 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)
		distance (A)	overiap (A)
1:D:46:LEU:H	1:D:46:LEU:HD12	1.39	0.86
2:A:300:RXD:CAM	2:A:300:RXD:HAQA	2.06	0.84
1:A:173:GLU:HG2	1:B:149:ARG:HH22	1.43	0.83
2:A:300:RXD:HAH	2:A:300:RXD:OAA	1.79	0.81
1:A:207:ARG:HG2	1:A:213:SER:HB3	1.63	0.81

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	$228/249\ (92\%)$	227 (100%)	1 (0%)	0	100 100
1	В	$230/249 \ (92\%)$	221 (96%)	8 (4%)	1 (0%)	34 48
1	С	230/249 (92%)	225 (98%)	4 (2%)	1 (0%)	34 48
1	D	228/249 (92%)	213 (93%)	13 (6%)	2 (1%)	17 25



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Mol	Chain	Analysed	Favoured	Allowed	Outliers	Percentiles	
All	All	916/996 (92%)	886 (97%)	26 (3%)	4 (0%)	34 48	

All (4) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	С	145	GLY
1	D	145	GLY
1	В	145	GLY
1	D	106	GLU

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	Rotameric Outliers		Percentiles		
1	A	208/221 (94%)	194 (93%)	14 (7%)	16	26		
1	В	210/221 (95%)	199 (95%)	11 (5%)	23	38		
1	С	210/221 (95%)	194 (92%)	16 (8%)	13	20		
1	D	208/221 (94%)	182 (88%)	26 (12%)	4	5		
All	All	836/884 (95%)	769 (92%)	67 (8%)	12	18		

5 of 67 residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	D	190	ASP
1	D	207	ARG
1	D	262	ILE
1	В	268	MET
1	В	222	MET

Sometimes sidechains can be flipped to improve hydrogen bonding and reduce clashes. 5 of 15 such sidechains are listed below:

Mol	Chain	Res	Type
1	В	234	HIS



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Mol	Chain	Res	Type
1	D	56	HIS
1	С	51	ASN
1	D	208	ASN
1	D	35	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)

4 non-standard protein/DNA/RNA residues 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 Lin			Link	Bond lengths			Bond angles		
MIOI	Type	Chain	nes	Lilik	Counts	RMSZ	# Z > 2	Counts	RMSZ	# Z > 2
1	OCS	В	163	1	7,8,9	1.27	1 (14%)	6,11,13	3.18	3 (50%)
1	OCS	A	163	1	7,8,9	1.43	2 (28%)	6,11,13	3.75	5 (83%)
1	OCS	D	163	1	7,8,9	1.34	1 (14%)	6,11,13	2.21	3 (50%)
1	OCS	С	163	1	7,8,9	1.60	2 (28%)	6,11,13	2.90	2 (33%)

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
1	OCS	В	163	1	-	3/4/7/9	-
1	OCS	A	163	1	-	0/4/7/9	-
1	OCS	D	163	1	-	0/4/7/9	-
1	OCS	С	163	1	-	2/4/7/9	-

The worst 5 of 6 bond length outliers are listed below:



Mol	Chain	Res	Type	Atoms	Z	$\operatorname{Observed}(\text{\AA})$	$Ideal(\AA)$
1	С	163	OCS	OD3-SG	2.74	1.53	1.45
1	D	163	OCS	CB-SG	-2.65	1.67	1.77
1	A	163	OCS	CB-SG	-2.51	1.68	1.77
1	С	163	OCS	CB-SG	-2.46	1.68	1.77
1	A	163	OCS	OD3-SG	2.42	1.52	1.45

The worst 5 of 13 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	\mathbf{Z}	$Observed(^o)$	$\operatorname{Ideal}({}^{o})$
1	A	163	OCS	OD3-SG-CB	6.64	114.83	106.94
1	С	163	OCS	OD3-SG-CB	6.46	114.62	106.94
1	В	163	OCS	OD2-SG-CB	5.67	114.78	105.74
1	D	163	OCS	OD3-SG-CB	3.97	111.65	106.94
1	В	163	OCS	OD3-SG-CB	3.89	111.56	106.94

There are no chirality outliers.

All (5) torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
1	В	163	OCS	CA-CB-SG-OD1
1	В	163	OCS	CA-CB-SG-OD2
1	В	163	OCS	CA-CB-SG-OD3
1	С	163	OCS	CA-CB-SG-OD1
1	С	163	OCS	CA-CB-SG-OD3

There are no ring outliers.

3 monomers are involved in 4 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
1	В	163	OCS	1	0
1	A	163	OCS	2	0
1	D	163	OCS	1	0

5.5 Carbohydrates (i)

There are no monosaccharides in this entry.

5.6 Ligand geometry (i)

1 ligand is 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			
		туре	Chain	nes	LIIIK	Counts	RMSZ	# Z > 2	Counts	RMSZ	# Z > 2
	2	RXD	A	300	-	33,33,33	2.87	5 (15%)	45,45,45	1.73	8 (17%)

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	RXD	A	300	-	-	5/17/33/33	0/3/3/3

All (5) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	\mathbf{Z}	$\operatorname{Observed}(\text{\AA})$	Ideal(A)
2	A	300	RXD	CBA-CBC	-14.03	1.40	1.54
2	A	300	RXD	CBD-CBB	-5.24	1.38	1.47
2	A	300	RXD	CBE-CBC	-4.01	1.40	1.48
2	A	300	RXD	CAN-CAO	2.83	1.53	1.49
2	A	300	RXD	CAX-NAR	-2.04	1.37	1.41

The worst 5 of 8 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	\mathbf{Z}	$Observed(^o)$	$Ideal(^{o})$
2	A	300	RXD	CBD-CBB-NAT	6.31	121.23	114.86
2	A	300	RXD	CBA-NAT-CBB	-4.24	120.30	125.62
2	A	300	RXD	CAX-NAR-CAV	-4.09	120.34	127.50
2	A	300	RXD	OAD-CBB-CBD	-3.25	117.66	123.30
2	A	300	RXD	CAY-NAS-CAW	-2.68	122.81	127.50

There are no chirality outliers.

All (5) torsion outliers are listed below:

Mol	Chain	Res	Type	Atoms
2	A	300	RXD	FAN-CAN-CAO-OAU



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Mol	Chain	Res	Type	Atoms
2	A	300	RXD	OAB-CAW-NAS-CAY
2	A	300	RXD	CAQ-CAW-NAS-CAY
2	A	300	RXD	CAN-CAO-OAU-CAZ
2	A	300	RXD	CAJ-CAY-NAS-CAW

There are no ring outliers.

1 monomer is involved in 12 short contacts:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
2	A	300	RXD	12	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 then 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^{th} 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>	$\# \mathrm{RSRZ}{>}2$	$\mathbf{OWAB}(\mathbf{\mathring{A}}^2)$	Q < 0.9
1	A	232/249~(93%)	-0.28	1 (0%) 92 91	29, 42, 63, 116	0
1	В	234/249 (93%)	-0.15	2 (0%) 84 82	28, 45, 72, 116	0
1	С	234/249 (93%)	0.06	8 (3%) 45 44	30, 50, 84, 119	0
1	D	232/249 (93%)	0.45	14 (6%) 21 20	43, 75, 107, 121	0
All	All	932/996 (93%)	0.02	25 (2%) 54 52	28, 50, 94, 121	0

The worst 5 of 25 RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	С	175	ASP	6.2
1	D	277	HIS	6.0
1	D	57	LYS	4.9
1	D	173	GLU	4.5
1	В	174	THR	4.1

6.2 Non-standard residues in protein, DNA, RNA chains (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^{th} 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	$\mathbf{B} ext{-}\mathbf{factors}(\mathbf{\mathring{A}}^2)$	Q<0.9
1	OCS	С	163	9/10	0.83	0.22	38,39,47,49	0
1	OCS	D	163	9/10	0.85	0.18	74,75,81,82	0
1	OCS	A	163	9/10	0.88	0.19	36,37,44,48	0
1	OCS	В	163	9/10	0.91	0.14	47,49,56,57	0



6.3 Carbohydrates (i)

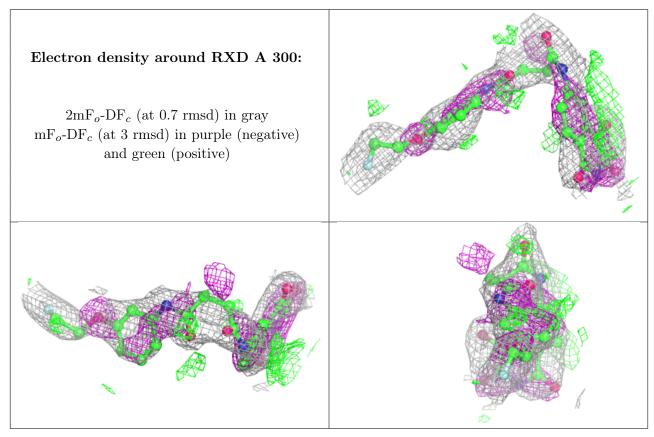
There are no monosaccharides in this entry.

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^{th} 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.

Mo	l Type	Chain	Res	Atoms	RSCC	RSR	$\mathbf{B} ext{-}\mathbf{factors}(\mathbf{\mathring{A}}^2)$	Q<0.9
2	RXD	A	300	31/31	0.80	0.31	46,48,52,54	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.

