



Full wwPDB X-ray Structure Validation Report ⓘ

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PDB ID : 5D4I / pdb_00005d4i
Title : Intact nitrite complex of a copper nitrite reductase determined by serial femtosecond crystallography
Authors : Fukuda, Y.; Tse, K.M.; Nakane, T.; Nakatsu, T.; Suzuki, M.; Sugahara, M.; Inoue, S.; Masuda, T.; Yumoto, F.; Matsugaki, N.; Nango, E.; Tono, K.; Joti, Y.; Kameshima, T.; Song, C.; Hatsui, T.; Yabashi, M.; Nureki, O.; Murphy, M.E.P.; Inoue, T.; Iwata, S.; Mizohata, E.
Deposited on : 2015-08-07
Resolution : 1.60 Å(reported)

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

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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>.

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)
Xtriage (Phenix)	:	2.0
EDS	:	3.0
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)

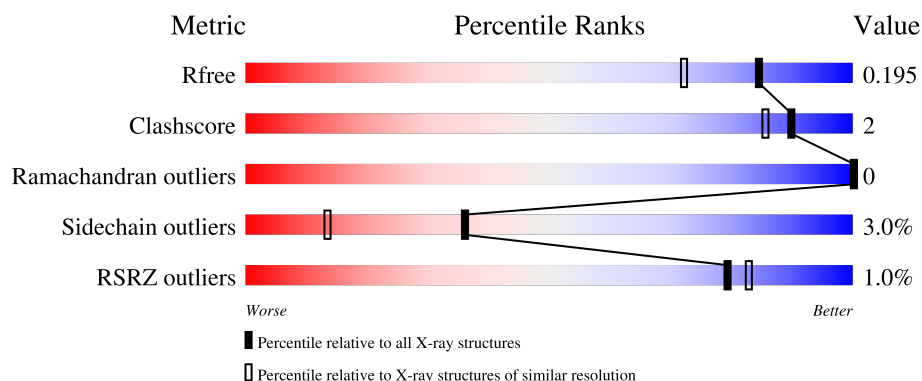
1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

X-RAY DIFFRACTION

The reported resolution of this entry is 1.60 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}	180053	4673 (1.60-1.60)
Clashscore	190562	4931 (1.60-1.60)
Ramachandran outliers	187476	4831 (1.60-1.60)
Sidechain outliers	187428	4830 (1.60-1.60)
RSRZ outliers	180081	4672 (1.60-1.60)

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 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 $\leq 5\%$. 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	342	% 86% 11% ..
1	B	342	% 91% 8% .
1	C	342	% 87% 10% ..

2 Entry composition

There are 4 unique types of molecules in this entry. The entry contains 8260 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 Copper-containing nitrite reductase.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	334	Total	C	N	O	S	0	7	0
			2612	1668	444	488	12			
1	B	341	Total	C	N	O	S	0	7	0
			2666	1703	456	496	11			
1	C	334	Total	C	N	O	S	0	10	0
			2637	1685	446	494	12			

There are 15 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	3	MET	-	initiating methionine	UNP P38501
A	341	LEU	-	expression tag	UNP P38501
A	342	VAL	-	expression tag	UNP P38501
A	343	PRO	-	expression tag	UNP P38501
A	344	ARG	-	expression tag	UNP P38501
B	3	MET	-	initiating methionine	UNP P38501
B	341	LEU	-	expression tag	UNP P38501
B	342	VAL	-	expression tag	UNP P38501
B	343	PRO	-	expression tag	UNP P38501
B	344	ARG	-	expression tag	UNP P38501
C	3	MET	-	initiating methionine	UNP P38501
C	341	LEU	-	expression tag	UNP P38501
C	342	VAL	-	expression tag	UNP P38501
C	343	PRO	-	expression tag	UNP P38501
C	344	ARG	-	expression tag	UNP P38501

- Molecule 2 is COPPER (II) ION (CCD ID: CU) (formula: Cu).

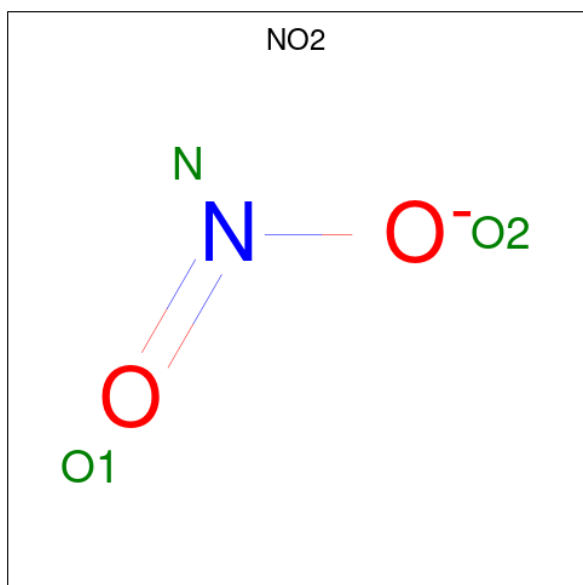
Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
2	A	2	Total	Cu	0	0
			2	2		

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Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
2	B	2	Total	Cu	0	0
			2	2		
2	C	2	Total	Cu	0	0
			2	2		

- Molecule 3 is NITRITE ION (CCD ID: NO2) (formula: NO₂).



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

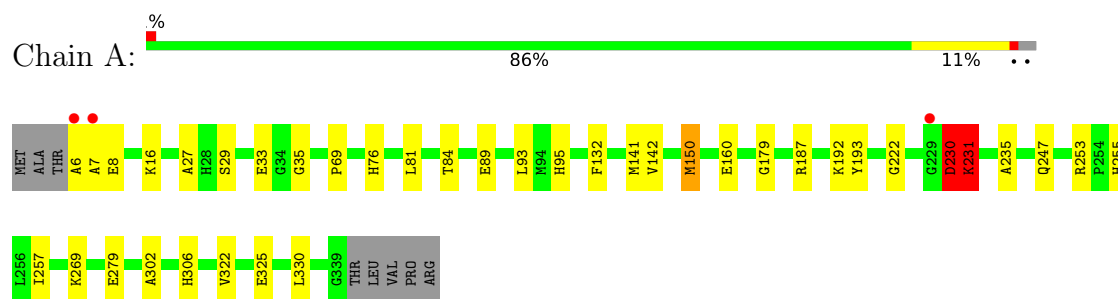
- Molecule 4 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	A	93	Total	O	0	4
			95	95		
4	B	113	Total	O	0	2
			114	114		
4	C	117	Total	O	0	5
			121	121		

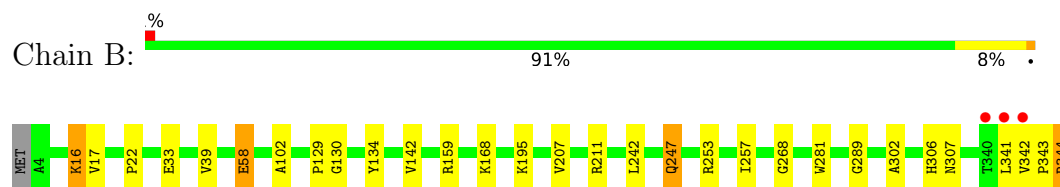
3 Residue-property plots

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.

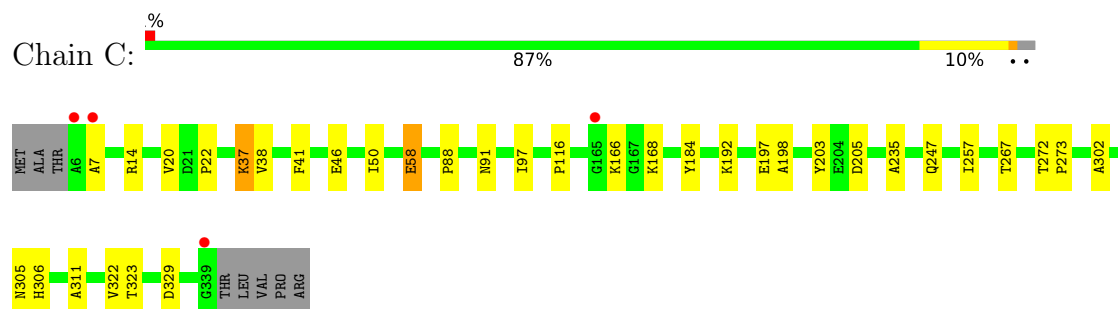
- Molecule 1: Copper-containing nitrite reductase



- Molecule 1: Copper-containing nitrite reductase



- Molecule 1: Copper-containing nitrite reductase



4 Data and refinement statistics

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants a, b, c, α , β , γ	63.00Å 103.00Å 147.40Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	28.97 – 1.60 28.97 – 1.60	Depositor EDS
% Data completeness (in resolution range)	100.0 (28.97-1.60) 99.9 (28.97-1.60)	Depositor EDS
R_{merge}	(Not available)	Depositor
R_{sym}	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ ¹	1.08 (at 1.60Å)	Xtriage
Refinement program	REFMAC 5.8.0103	Depositor
R, R_{free}	0.165 , 0.196 0.164 , 0.195	Depositor DCC
R_{free} test set	6263 reflections (4.93%)	wwPDB-VP
Wilson B-factor (Å ²)	34.7	Xtriage
Anisotropy	0.044	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.31 , 33.7	EDS
L-test for twinning ²	$\langle L \rangle = 0.49$, $\langle L^2 \rangle = 0.32$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
F_o, F_c correlation	0.98	EDS
Total number of atoms	8260	wwPDB-VP
Average B, all atoms (Å ²)	37.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 3.70% 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, 0.333 respectively for untwinned datasets, and 0.375, 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: CU, NO2

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	1.37	7/2683 (0.3%)	1.30	11/3654 (0.3%)
1	B	1.38	11/2738 (0.4%)	1.23	4/3730 (0.1%)
1	C	1.36	7/2708 (0.3%)	1.29	8/3688 (0.2%)
All	All	1.37	25/8129 (0.3%)	1.28	23/11072 (0.2%)

Chiral center outliers are detected by calculating the chiral volume of a chiral center and verifying if the center is modelled as a planar moiety or with the opposite hand. A planarity outlier is detected by checking planarity of atoms in a peptide group, atoms in a mainchain group or atoms of a sidechain that are expected to be planar.

Mol	Chain	#Chirality outliers	#Planarity outliers
1	A	0	3
1	B	0	3
1	C	0	1
All	All	0	7

All (25) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	C	273	PRO	CA-C	8.96	1.58	1.52
1	B	22	PRO	C-O	-7.72	1.18	1.25
1	A	253	ARG	N-CA	6.76	1.53	1.46
1	C	97	ILE	C-O	-6.66	1.17	1.24
1	B	142	VAL	CA-C	6.39	1.59	1.52
1	A	93	LEU	CA-C	-5.99	1.45	1.52
1	C	22	PRO	CA-C	5.93	1.55	1.51
1	B	130	GLY	N-CA	5.88	1.51	1.45
1	B	307	ASN	N-CA	5.82	1.53	1.46
1	C	205	ASP	CA-C	5.71	1.60	1.52
1	C	46	GLU	N-CA	5.62	1.52	1.46
1	A	150	MET	CA-CB	5.60	1.59	1.52

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Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	B	102	ALA	N-CA	5.51	1.52	1.45
1	A	76	HIS	CA-C	-5.51	1.46	1.52
1	B	129	PRO	CA-C	5.34	1.59	1.52
1	B	134	TYR	CA-C	-5.33	1.46	1.52
1	A	84	THR	CA-CB	5.30	1.60	1.53
1	C	323	THR	N-CA	5.28	1.51	1.45
1	A	330	LEU	N-CA	5.24	1.52	1.46
1	A	27	ALA	CA-C	-5.22	1.46	1.52
1	C	41	PHE	CA-C	-5.21	1.46	1.52
1	B	242	LEU	N-CA	5.18	1.52	1.46
1	B	247	GLN	C-O	-5.17	1.17	1.23
1	B	39	VAL	CA-CB	5.16	1.60	1.54
1	B	289	GLY	CA-C	5.14	1.56	1.52

All (23) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
1	A	231[A]	LYS	CA-C-N	12.22	138.13	120.90
1	A	231[A]	LYS	C-N-CA	12.22	138.13	120.90
1	A	231[B]	LYS	CA-C-N	12.22	138.13	120.90
1	A	231[B]	LYS	C-N-CA	12.22	138.13	120.90
1	C	272	THR	CA-C-N	-8.64	113.44	119.66
1	C	272	THR	C-N-CA	-8.64	113.44	119.66
1	A	142	VAL	CA-C-N	-6.38	112.43	119.19
1	A	142	VAL	C-N-CA	-6.38	112.43	119.19
1	A	230	ASP	O-C-N	-5.91	113.65	122.39
1	C	7	ALA	N-CA-C	-5.87	104.89	111.28
1	B	142	VAL	CA-C-N	-5.82	113.02	119.19
1	B	142	VAL	C-N-CA	-5.82	113.02	119.19
1	A	35	GLY	CA-C-N	5.81	125.83	119.90
1	A	35	GLY	C-N-CA	5.81	125.83	119.90
1	C	203	TYR	N-CA-C	5.79	117.28	110.97
1	A	222	GLY	N-CA-C	5.27	122.36	115.36
1	C	184	TYR	CA-C-N	-5.21	117.61	123.02
1	C	184	TYR	C-N-CA	-5.21	117.61	123.02
1	A	253	ARG	NE-CZ-NH2	5.17	123.85	119.20
1	C	198	ALA	CA-C-N	-5.06	113.83	119.19
1	C	198	ALA	C-N-CA	-5.06	113.83	119.19
1	B	268	GLY	CA-C-N	-5.05	115.99	123.05
1	B	268	GLY	C-N-CA	-5.05	115.99	123.05

There are no chirality outliers.

All (7) planarity outliers are listed below:

Mol	Chain	Res	Type	Group
1	A	230	ASP	Mainchain
1	A	306	HIS	Peptide
1	B	306	HIS	Peptide
1	B	58	GLU	Mainchain
1	C	306	HIS	Peptide

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	2612	0	2532	13	0
1	B	2666	0	2601	7	0
1	C	2637	0	2557	10	0
2	A	2	0	0	0	0
2	B	2	0	0	0	0
2	C	2	0	0	0	0
3	A	6	0	0	0	0
3	C	3	0	0	0	0
4	A	95	0	0	0	0
4	B	114	0	0	0	0
4	C	121	0	0	2	0
All	All	8260	0	7690	28	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 2.

All (28) 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:231[A]:LYS:HD2	1:A:231[A]:LYS:N	2.05	0.71
1:A:6:ALA:HB2	1:C:329:ASP:HB2	1.83	0.59
1:C:257:ILE:HD12	1:C:302:ALA:HB3	1.88	0.56
1:B:257:ILE:HD12	1:B:302:ALA:HB3	1.88	0.55
1:A:7:ALA:HA	1:A:8:GLU:HG2	1.88	0.55
1:A:187[B]:ARG:HD3	1:A:193:TYR:CE1	2.43	0.54

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Atom-1	Atom-2	Interatomic distance (Å)	Clash overlap (Å)
1:B:344:ARG:HD3	1:C:116:PRO:HG2	1.90	0.53
1:B:207:VAL:O	1:B:211[B]:ARG:HG3	2.10	0.50
1:A:69:PRO:HG3	1:A:179:GLY:HA3	1.95	0.48
1:A:132:PHE:CE2	1:A:269:LYS:HE3	2.48	0.48
1:B:58:GLU:CD	1:B:195:LYS:HE3	2.41	0.46
1:A:95:HIS:HB3	1:A:150:MET:HE1	1.98	0.45
1:C:50:ILE:O	1:C:58[A]:GLU:HA	2.17	0.45
1:C:88:PRO:HD2	1:C:91:ASN:ND2	2.32	0.45
1:A:257:ILE:HD12	1:A:302:ALA:HB3	1.98	0.44
1:A:235:ALA:O	1:A:322:VAL:HA	2.18	0.44
1:A:255:HIS:ND1	1:A:279:GLU:O	2.51	0.43
1:B:16[A]:LYS:HD2	1:B:17:VAL:N	2.34	0.43
1:A:230:ASP:C	1:A:231[A]:LYS:HD2	2.43	0.43
1:C:235:ALA:O	1:C:322:VAL:HA	2.19	0.42
1:C:14[B]:ARG:HG3	1:C:38:VAL:HB	2.01	0.41
1:B:342:VAL:HB	1:B:343:PRO:HD2	2.03	0.41
1:C:37:LYS:HE2	4:C:622:HOH:O	2.20	0.41
1:C:267:THR:HG22	4:C:713:HOH:O	2.21	0.41
1:A:81:LEU:HD23	1:A:81:LEU:C	2.46	0.41
1:C:305:ASN:O	1:C:311:ALA:HB2	2.22	0.40
1:B:253:ARG:HA	1:B:281:TRP:O	2.21	0.40
1:A:141[A]:MET:HE3	1:A:141[A]:MET:HB2	1.96	0.40

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	339/342 (99%)	333 (98%)	6 (2%)	0	100	100
1	B	346/342 (101%)	344 (99%)	2 (1%)	0	100	100
1	C	342/342 (100%)	338 (99%)	4 (1%)	0	100	100

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Mol	Chain	Analysed	Favoured	Allowed	Outliers	Percentiles	
All	All	1027/1026 (100%)	1015 (99%)	12 (1%)	0	100	100

There are no Ramachandran outliers to report.

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	270/270 (100%)	260 (96%)	10 (4%)	30	10
1	B	276/270 (102%)	267 (97%)	9 (3%)	33	12
1	C	273/270 (101%)	264 (97%)	9 (3%)	33	12
All	All	819/810 (101%)	791 (97%)	28 (3%)	36	11

All (28) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	16	LYS
1	A	29	SER
1	A	33	GLU
1	A	89	GLU
1	A	160	GLU
1	A	192	LYS
1	A	231[A]	LYS
1	A	231[B]	LYS
1	A	247	GLN
1	A	325	GLU
1	B	16[A]	LYS
1	B	16[B]	LYS
1	B	33	GLU
1	B	159[A]	ARG
1	B	159[B]	ARG
1	B	168	LYS
1	B	247	GLN
1	B	341	LEU

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Mol	Chain	Res	Type
1	B	344	ARG
1	C	20	VAL
1	C	37	LYS
1	C	58[A]	GLU
1	C	58[B]	GLU
1	C	166	LYS
1	C	168	LYS
1	C	192	LYS
1	C	197	GLU
1	C	247	GLN

Sometimes sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (2) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	15	GLN
1	B	15	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 oligosaccharides in this entry.

5.6 Ligand geometry ⓘ

Of 9 ligands modelled in this entry, 6 are monoatomic - leaving 3 for Mogul analysis.

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	$\# Z > 2$
3	NO2	A	503	2	1,2,2	0.44	0	0,1,1	-	-
3	NO2	C	503	2	1,2,2	0.02	0	0,1,1	-	-
3	NO2	A	504	2	1,2,2	0.62	0	0,1,1	-	-

There are no bond length outliers.

There are no bond angle outliers.

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

No monomer is involved in short contacts.

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	334/342 (97%)	-0.16	3 (0%) 81 84	17, 37, 63, 83	7 (2%)
1	B	341/342 (99%)	-0.27	3 (0%) 81 84	15, 33, 63, 103	7 (2%)
1	C	334/342 (97%)	-0.40	4 (1%) 76 80	16, 31, 54, 88	10 (2%)
All	All	1009/1026 (98%)	-0.28	10 (0%) 79 83	15, 34, 62, 103	24 (2%)

All (10) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	C	7	ALA	3.3
1	B	341	LEU	3.2
1	C	6	ALA	2.7
1	B	340	THR	2.6
1	A	7	ALA	2.6
1	A	6	ALA	2.6
1	C	339	GLY	2.5
1	B	342	VAL	2.4
1	A	229	GLY	2.1
1	C	165	GLY	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 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, 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	B-factors(Å ²)	Q<0.9
3	NO2	A	503	3/3	0.97	0.08	36,36,39,43	0
3	NO2	A	504	3/3	0.97	0.06	37,37,44,47	0
3	NO2	C	503	3/3	0.98	0.04	31,31,37,42	0
2	CU	B	502	1/1	1.00	0.02	32,32,32,32	0
2	CU	C	501	1/1	1.00	0.01	26,26,26,26	0
2	CU	C	502	1/1	1.00	0.01	25,25,25,25	0
2	CU	A	501	1/1	1.00	0.01	35,35,35,35	0
2	CU	A	502	1/1	1.00	0.01	31,31,31,31	0
2	CU	B	501	1/1	1.00	0.01	35,35,35,35	0

6.5 Other polymers [i](#)

There are no such residues in this entry.