



wwPDB X-ray Structure Validation Summary Report ⓘ

Oct 7, 2024 – 05:07 PM EDT

PDB ID : 4EK1
Title : Crystal Structure of Electron-Spin Labeled Cytochrome P450cam
Authors : Lee, Y.-T.; Goodin, D.B.
Deposited on : 2012-04-08
Resolution : 1.97 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 ⓘ 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	:	4.02b-467
Mogul	:	2022.3.0, CSD as543be (2022)
Xtriage (Phenix)	:	1.20.1
EDS	:	3.0
buster-report	:	1.1.7 (2018)
Percentile statistics	:	20231227.v01 (using entries in the PDB archive December 27th 2023)
CCP4	:	9.0.003 (Gargrove)
Density-Fitness	:	1.0.11
Ideal geometry (proteins)	:	Engh & Huber (2001)
Ideal geometry (DNA, RNA)	:	Parkinson et al. (1996)
Validation Pipeline (wwPDB-VP)	:	2.39

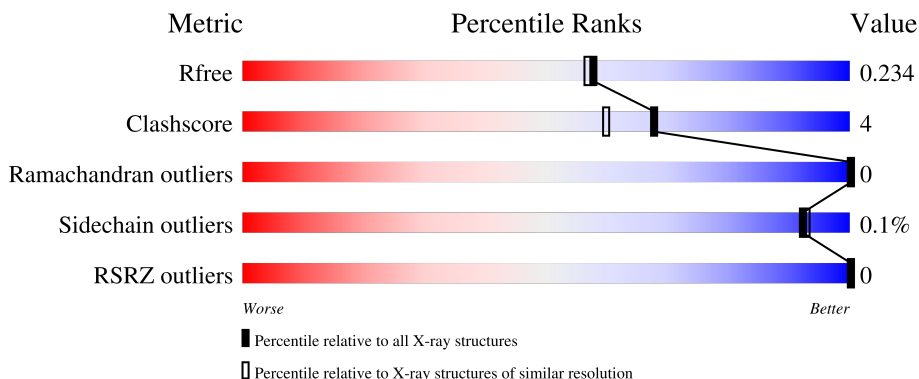
1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

X-RAY DIFFRACTION



The reported resolution of this entry is 1.97 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}	164625	1356 (1.98-1.98)
Clashscore	180529	1437 (1.98-1.98)
Ramachandran outliers	177936	1426 (1.98-1.98)
Sidechain outliers	177891	1426 (1.98-1.98)
RSRZ outliers	164620	1356 (1.98-1.98)

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	414	 91% 7% •
1	B	414	 88% 10% •

2 Entry composition

There are 6 unique types of molecules in this entry. The entry contains 7014 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.

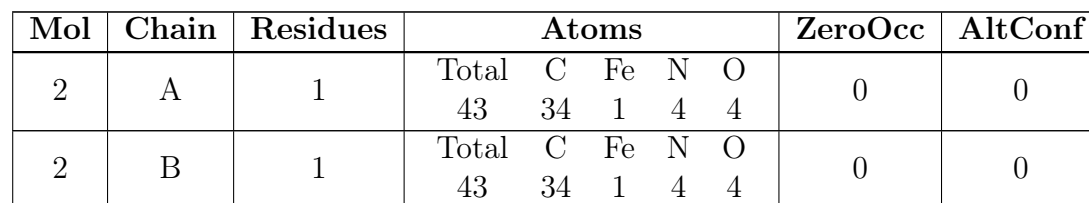
- Molecule 1 is a protein called Camphor 5-monooxygenase.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	403	Total	C	N	O	S	0	8	0
			3228	2050	560	603	15			
1	B	404	Total	C	N	O	S	0	5	0
			3227	2045	562	605	15			

There are 14 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	48	CYS	SER	engineered mutation	UNP P00183
A	58	SER	CYS	engineered mutation	UNP P00183
A	85	SER	CYS	engineered mutation	UNP P00183
A	136	SER	CYS	engineered mutation	UNP P00183
A	190	CYS	SER	engineered mutation	UNP P00183
A	285	SER	CYS	engineered mutation	UNP P00183
A	334	ALA	CYS	engineered mutation	UNP P00183
B	48	CYS	SER	engineered mutation	UNP P00183
B	58	SER	CYS	engineered mutation	UNP P00183
B	85	SER	CYS	engineered mutation	UNP P00183
B	136	SER	CYS	engineered mutation	UNP P00183
B	190	CYS	SER	engineered mutation	UNP P00183
B	285	SER	CYS	engineered mutation	UNP P00183
B	334	ALA	CYS	engineered mutation	UNP P00183

- Molecule 2 is PROTOPORPHYRIN IX CONTAINING FE (three-letter code: HEM) (formula: $C_{34}H_{32}FeN_4O_4$).



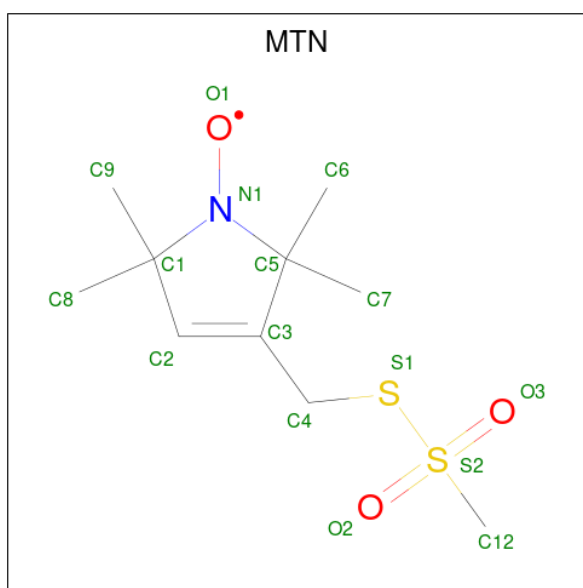
-
- The ORTEP diagram shows a cyclohexane ring with atoms C1 through C7. C1 is bonded to C2, C6, and C10. C2 is bonded to C1, C3, and an oxygen atom (O). C3 is bonded to C2 and C4. C4 is bonded to C3, C5, and H4. C5 is bonded to C4, C6, and C7. C6 is bonded to C1, C5, and C8. C7 is bonded to C5 and C8. C8 is bonded to C6 and C9. C10 is bonded to C1. The ring is in a chair conformation. The oxygen atom is double-bonded to C2. The hydrogen atom H4 is bonded to C4. The atoms are labeled with green text. The oxygen atom is shown in red. The bonds to C10 and H4 are shown with black and grey wedges, respectively. The bonds to C8 and C9 are shown with grey lines. The bonds to C2 and C7 are shown with red lines. The bonds to C3 and C5 are shown with black lines. The bonds to C4 and C6 are shown with black lines. The bonds to C1 and C5 are shown with black lines. The bonds to C1 and C6 are shown with black lines. The bonds to C1 and C7 are shown with black lines. The bonds to C1 and C8 are shown with black lines. The bonds to C1 and C9 are shown with black lines. The bonds to C1 and C10 are shown with black lines. The bonds to C2 and C3 are shown with black lines. The bonds to C2 and C4 are shown with black lines. The bonds to C2 and C5 are shown with black lines. The bonds to C2 and C6 are shown with black lines. The bonds to C2 and C7 are shown with black lines. The bonds to C2 and C8 are shown with black lines. The bonds to C2 and C9 are shown with black lines. The bonds to C2 and C10 are shown with black lines. The bonds to C3 and C4 are shown with black lines. The bonds to C3 and C5 are shown with black lines. The bonds to C3 and C6 are shown with black lines. The bonds to C3 and C7 are shown with black lines. The bonds to C3 and C8 are shown with black lines. The bonds to C3 and C9 are shown with black lines. The bonds to C3 and C10 are shown with black lines. The bonds to C4 and C5 are shown with black lines. The bonds to C4 and C6 are shown with black lines. The bonds to C4 and C7 are shown with black lines. The bonds to C4 and C8 are shown with black lines. The bonds to C4 and C9 are shown with black lines. The bonds to C4 and C10 are shown with black lines. The bonds to C5 and C6 are shown with black lines. The bonds to C5 and C7 are shown with black lines. The bonds to C5 and C8 are shown with black lines. The bonds to C5 and C9 are shown with black lines. The bonds to C5 and C10 are shown with black lines. The bonds to C6 and C7 are shown with black lines. The bonds to C6 and C8 are shown with black lines. The bonds to C6 and C9 are shown with black lines. The bonds to C6 and C10 are shown with black lines. The bonds to C7 and C8 are shown with black lines. The bonds to C7 and C9 are shown with black lines. The bonds to C7 and C10 are shown with black lines. The bonds to C8 and C9 are shown with black lines. The bonds to C8 and C10 are shown with black lines. The bonds to C9 and C10 are shown with black lines.

Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
3	A	1	Total	C	O	0	0
			11	10	1		
3	B	1	Total	C	O	0	0
			11	10	1		

- Molecule 4 is POTASSIUM ION (three-letter code: K) (formula: K).

Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
4	A	1	Total K 1 1	0	0
4	B	1	Total K 1 1	0	0

- Molecule 5 is S-[(1-oxyl-2,2,5,5-tetramethyl-2,5-dihydro-1H-pyrrol-3-yl)methyl] methanesulfonothioate (three-letter code: MTN) (formula: C₁₀H₁₈NO₃S₂).



Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
5	A	1	Total C N O S 12 9 1 1 1	0	0
5	A	1	Total C N O S 12 9 1 1 1	0	0
5	B	1	Total C N O S 12 9 1 1 1	0	0

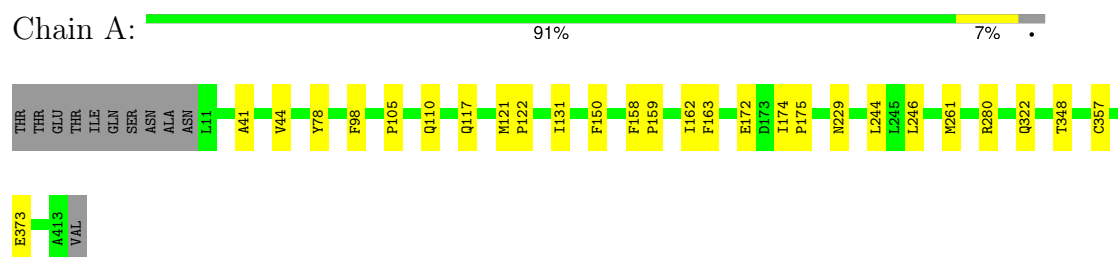
- Molecule 6 is water.

Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
6	A	216	Total O 216 216	0	0
6	B	197	Total O 197 197	0	0

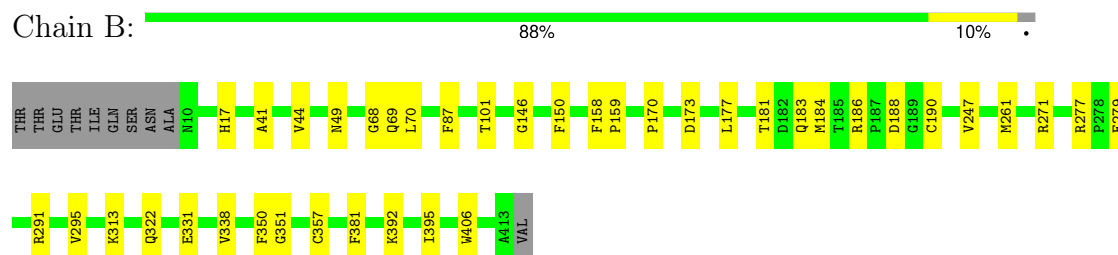
3 Residue-property plots [i](#)

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: Camphor 5-monooxygenase



- Molecule 1: Camphor 5-monooxygenase



4 Data and refinement statistics

Property	Value	Source
Space group	P 1 21 1	Depositor
Cell constants a, b, c, α , β , γ	56.02Å 101.53Å 72.98Å 90.00° 107.39° 90.00°	Depositor
Resolution (Å)	10.00 – 1.97 10.00 – 1.97	Depositor EDS
% Data completeness (in resolution range)	98.3 (10.00-1.97) 97.5 (10.00-1.97)	Depositor EDS
R_{merge}	0.10	Depositor
R_{sym}	0.10	Depositor
$\langle I/\sigma(I) \rangle$ ¹	2.12 (at 1.96Å)	Xtriage
Refinement program	REFMAC	Depositor
R, R_{free}	0.204 , 0.253 0.184 , 0.234	Depositor DCC
R_{free} test set	2719 reflections (5.06%)	wwPDB-VP
Wilson B-factor (Å ²)	20.4	Xtriage
Anisotropy	0.077	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.42 , 36.4	EDS
L-test for twinning ²	$\langle L \rangle = 0.47$, $\langle L^2 \rangle = 0.30$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
F_o, F_c correlation	0.95	EDS
Total number of atoms	7014	wwPDB-VP
Average B, all atoms (Å ²)	20.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The analyses of the Patterson function reveals a significant off-origin peak that is 48.91 % of the origin peak, indicating pseudo-translational symmetry. The chance of finding a peak of this or larger height randomly in a structure without pseudo-translational symmetry is equal to 8.0405e-05. The detected translational NCS is most likely also responsible for the elevated intensity ratio.*

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: CAM, K, MTN, HEM

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	0.56	0/3331	0.64	0/4524
1	B	0.56	0/3318	0.65	0/4508
All	All	0.56	0/6649	0.65	0/9032

There are no bond length outliers.

There are no bond angle outliers.

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	3228	0	3196	19	0
1	B	3227	0	3174	24	0
2	A	43	0	30	2	0
2	B	43	0	30	4	0
3	A	11	0	16	0	0
3	B	11	0	16	0	0
4	A	1	0	0	0	0
4	B	1	0	0	0	0
5	A	24	0	30	0	0
5	B	12	0	15	1	0
6	A	216	0	0	2	0

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Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
6	B	197	0	0	1	0
All	All	7014	0	6507	45	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.

The worst 5 of 45 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:49:ASN:HB3	5:B:504:MTN:H81	1.61	0.80
1:A:163:PHE:CE2	1:A:246:LEU:HD12	2.25	0.70
2:A:501:HEM:HMB1	2:A:501:HEM:HBB2	1.76	0.66
1:B:68:GLY:HA3	1:B:331:GLU:OE2	1.97	0.65
1:B:177:LEU:O	1:B:181:THR:HG23	2.00	0.61

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	409/414 (99%)	397 (97%)	12 (3%)	0	100	100
1	B	407/414 (98%)	397 (98%)	10 (2%)	0	100	100
All	All	816/828 (99%)	794 (97%)	22 (3%)	0	100	100

There are no Ramachandran outliers to report.

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	355/357 (99%)	354 (100%)	1 (0%)	91	91
1	B	353/357 (99%)	353 (100%)	0	100	100
All	All	708/714 (99%)	707 (100%)	1 (0%)	92	93

All (1) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	280	ARG

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

Mol	Chain	Res	Type
1	A	69	GLN

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](#)

Of 9 ligands modelled in this entry, 2 are monoatomic - leaving 7 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	CAM	A	502	-	12,12,12	3.77	1 (8%)	20,21,21	1.01	1 (5%)
5	MTN	B	504	1	9,12,16	1.34	1 (11%)	11,20,27	2.69	5 (45%)
2	HEM	A	501	1	42,50,50	1.87	6 (14%)	46,82,82	1.69	9 (19%)
5	MTN	A	504	1	9,12,16	1.45	1 (11%)	11,20,27	2.04	4 (36%)
2	HEM	B	501	1	42,50,50	2.03	8 (19%)	46,82,82	1.80	8 (17%)
5	MTN	A	505	1	9,12,16	1.47	2 (22%)	11,20,27	2.43	5 (45%)
3	CAM	B	502	-	12,12,12	3.54	2 (16%)	20,21,21	1.18	3 (15%)

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	CAM	A	502	-	-	-	0/3/2/2
5	MTN	B	504	1	-	0/0/25/29	0/1/1/1
2	HEM	A	501	1	-	0/12/54/54	-
5	MTN	A	504	1	-	0/0/25/29	0/1/1/1
2	HEM	B	501	1	-	1/12/54/54	-
5	MTN	A	505	1	-	0/0/25/29	0/1/1/1
3	CAM	B	502	-	-	-	0/3/2/2

The worst 5 of 21 bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
3	A	502	CAM	O-C2	12.90	1.41	1.21
3	B	502	CAM	O-C2	11.84	1.40	1.21
2	A	501	HEM	C3D-C2D	7.84	1.53	1.36
2	B	501	HEM	C3D-C2D	7.66	1.53	1.36
2	B	501	HEM	C3C-C2C	-5.42	1.33	1.40

The worst 5 of 35 bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
2	B	501	HEM	C4D-ND-C1D	5.82	112.10	105.21
2	A	501	HEM	C4D-ND-C1D	5.51	111.73	105.21
5	B	504	MTN	C2-C1-N1	4.59	102.59	99.43
5	A	504	MTN	C2-C1-N1	4.32	102.41	99.43

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Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
5	A	505	MTN	C8-C1-N1	4.25	114.92	110.05

There are no chirality outliers.

All (1) torsion outliers are listed below:

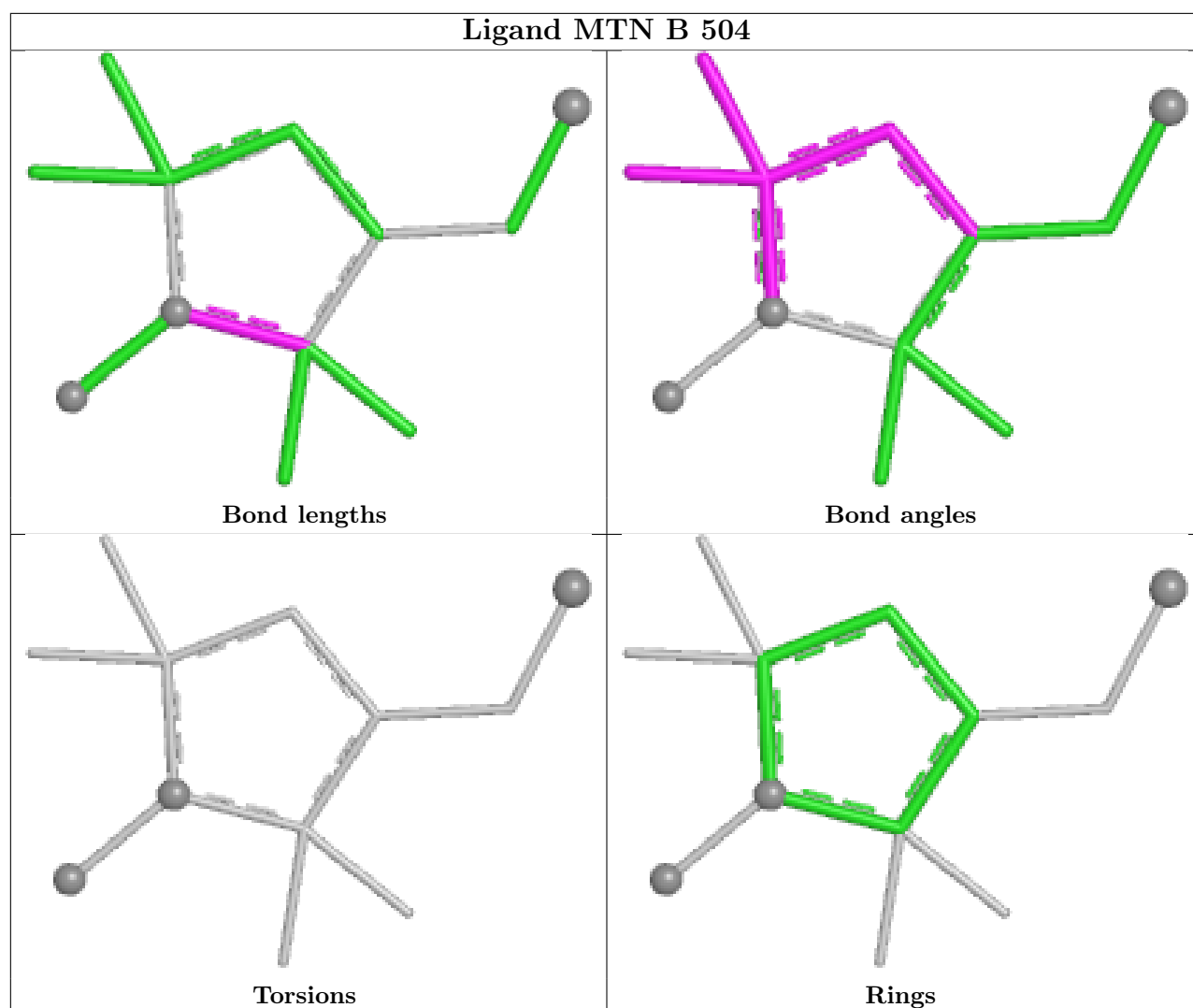
Mol	Chain	Res	Type	Atoms
2	B	501	HEM	CAA-CBA-CGA-O2A

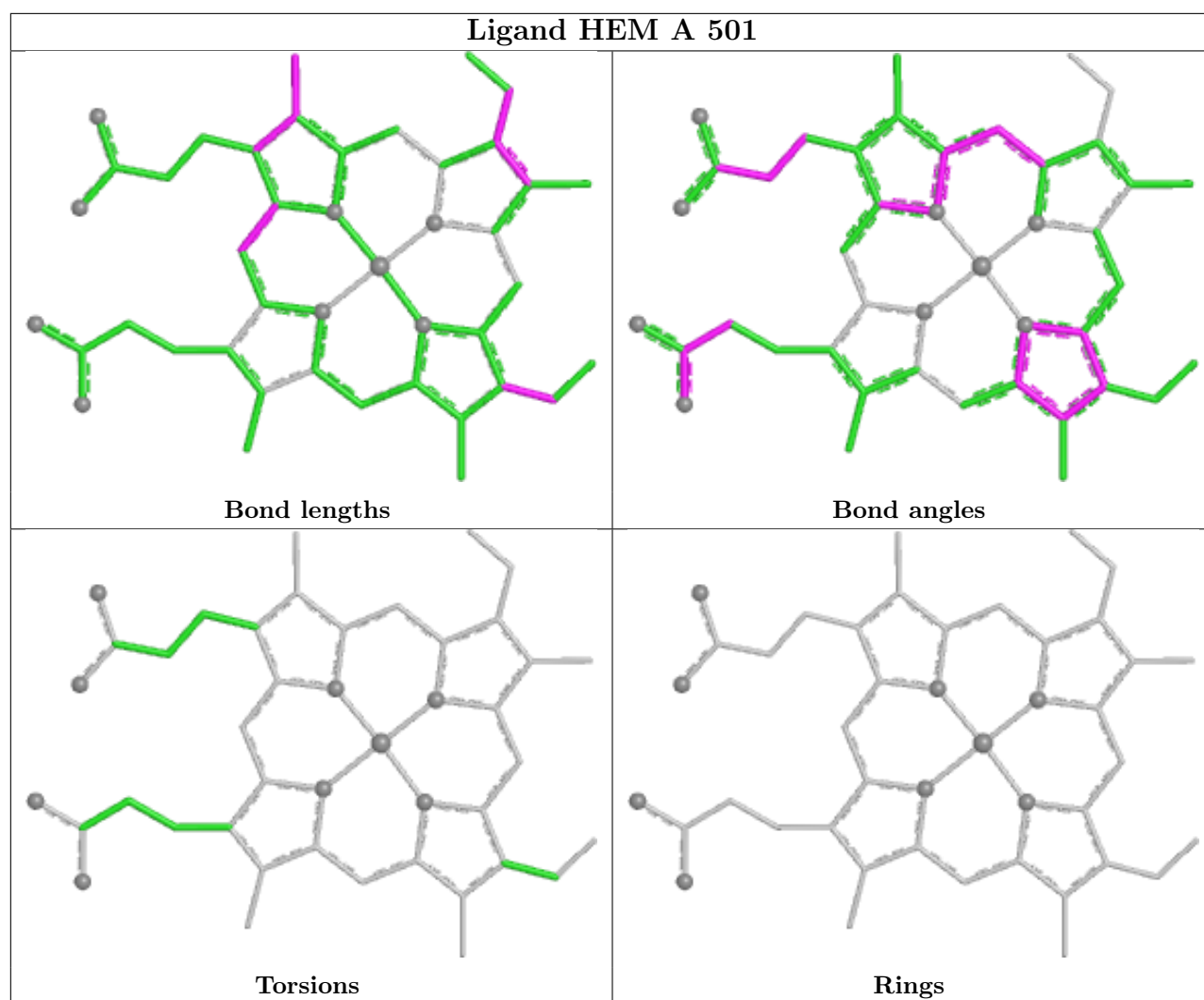
There are no ring outliers.

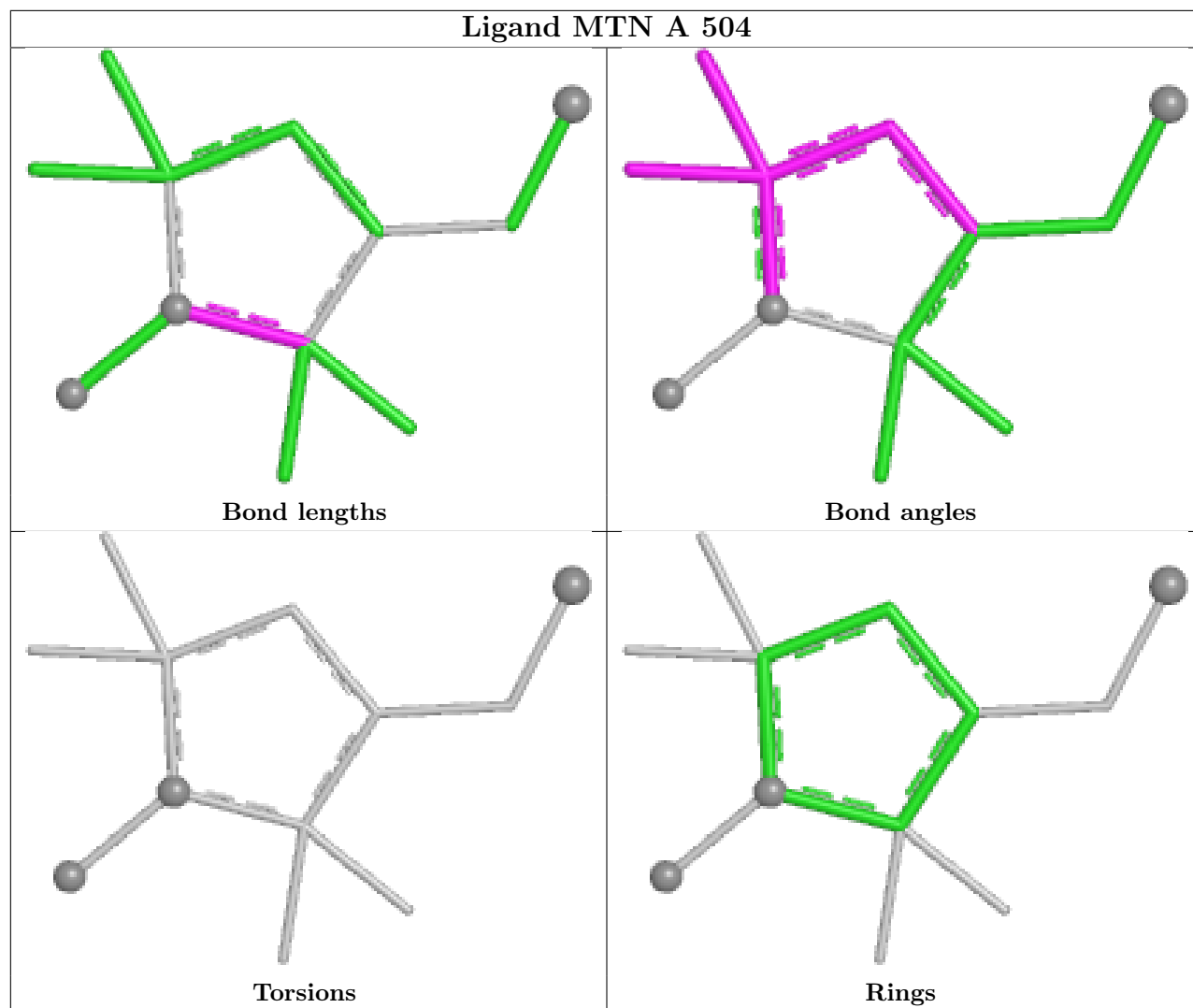
3 monomers are involved in 7 short contacts:

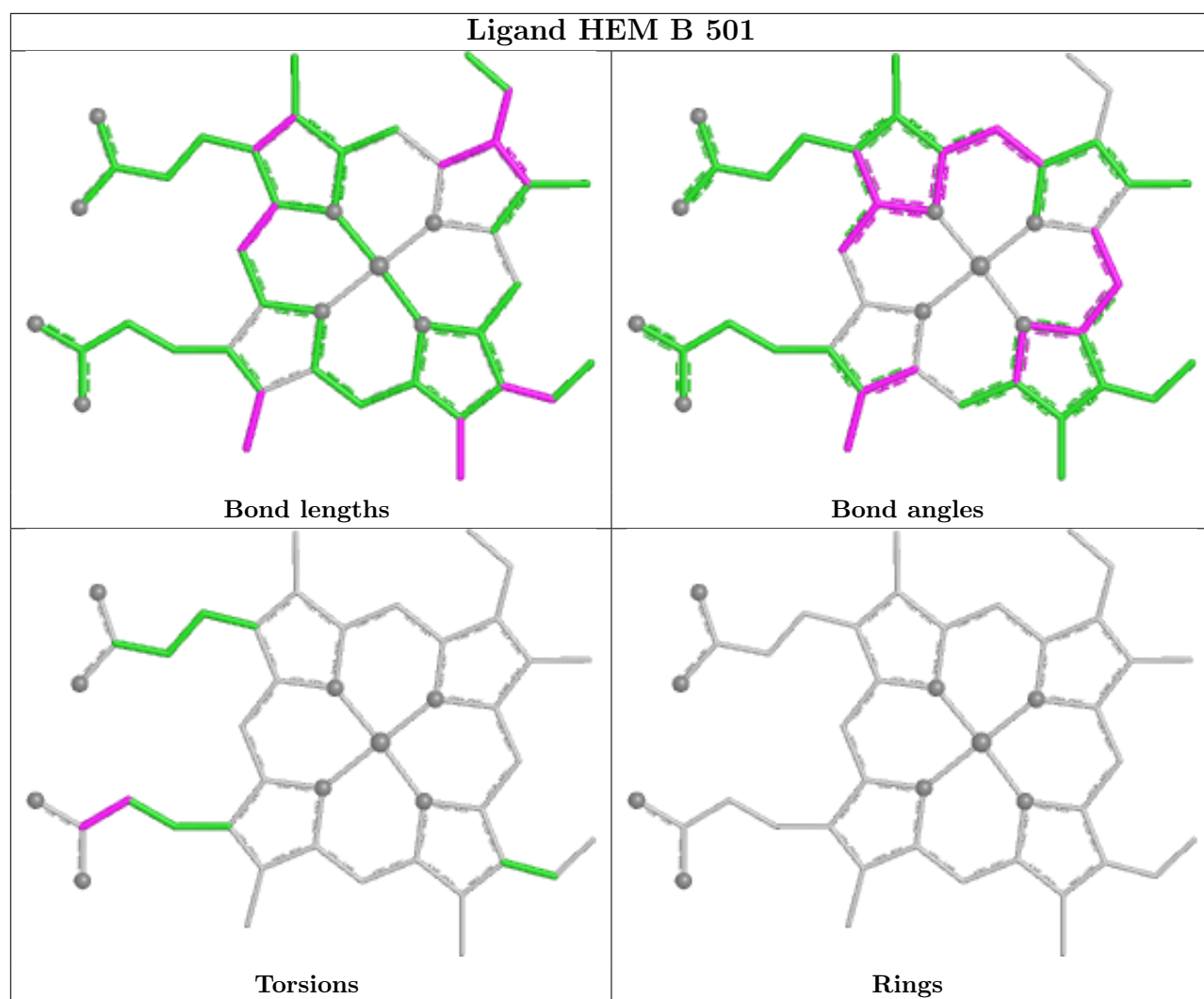
Mol	Chain	Res	Type	Clashes	Symm-Clashes
5	B	504	MTN	1	0
2	A	501	HEM	2	0
2	B	501	HEM	4	0

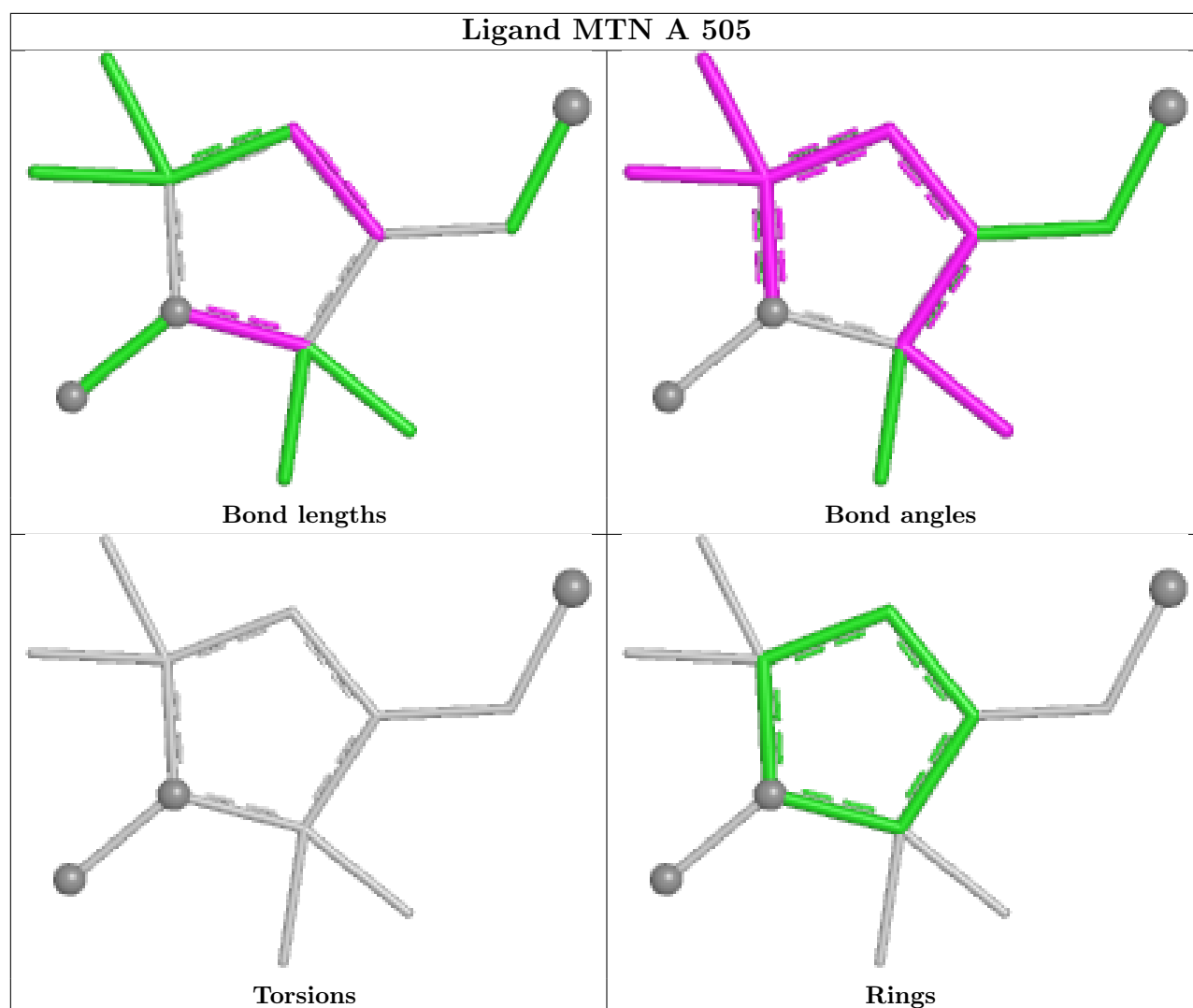
The following is a two-dimensional graphical depiction of Mogul quality analysis of bond lengths, bond angles, torsion angles, and ring geometry for all instances of the Ligand of Interest. In addition, ligands with molecular weight > 250 and outliers as shown on the validation Tables will also be included. 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, 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	403/414 (97%)	-0.61	0 100 100	10, 19, 28, 40	8 (1%)
1	B	404/414 (97%)	-0.57	0 100 100	11, 19, 30, 44	5 (1%)
All	All	807/828 (97%)	-0.59	0 100 100	10, 19, 28, 44	13 (1%)

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, 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.

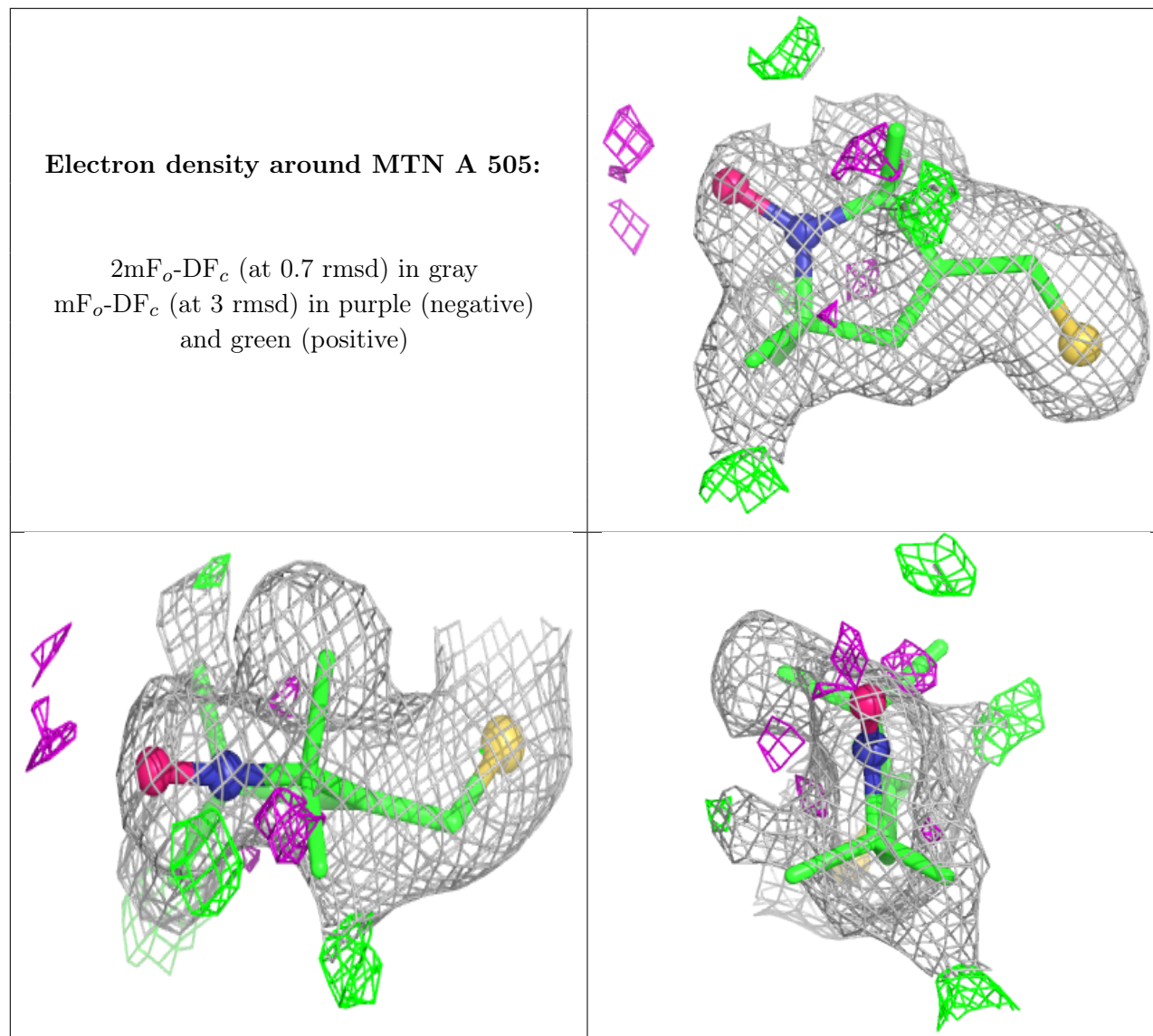
Mol	Type	Chain	Res	Atoms	RSCC	RSR	B-factors(Å ²)	Q<0.9
5	MTN	A	505	12/16	0.80	0.13	41,43,44,44	0
5	MTN	B	504	12/16	0.89	0.10	33,38,39,41	0
3	CAM	B	502	11/11	0.93	0.06	11,13,15,16	0
5	MTN	A	504	12/16	0.93	0.08	29,35,36,37	0
3	CAM	A	502	11/11	0.95	0.04	12,14,15,15	0
2	HEM	B	501	43/43	0.97	0.05	9,14,17,18	0
2	HEM	A	501	43/43	0.98	0.05	11,13,16,18	0

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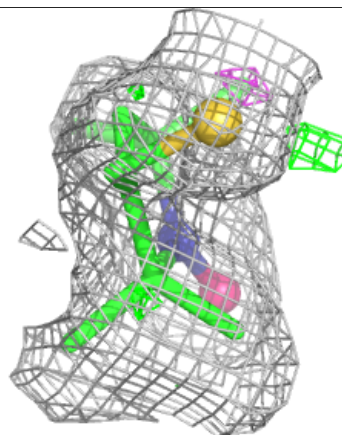
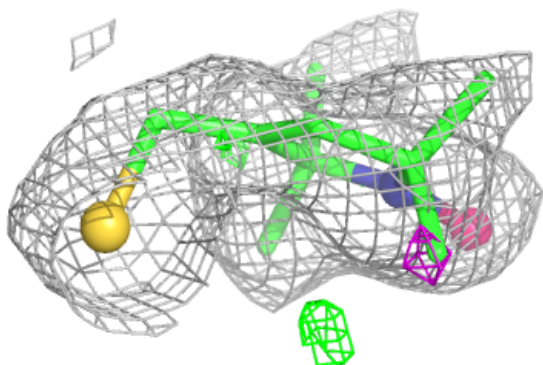
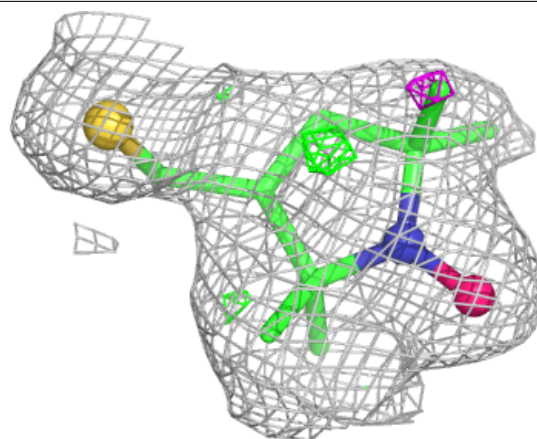
Mol	Type	Chain	Res	Atoms	RSCC	RSR	B-factors(\AA^2)	Q<0.9
4	K	B	503	1/1	0.99	0.02	24,24,24,24	0
4	K	A	503	1/1	0.99	0.05	21,21,21,21	0

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.



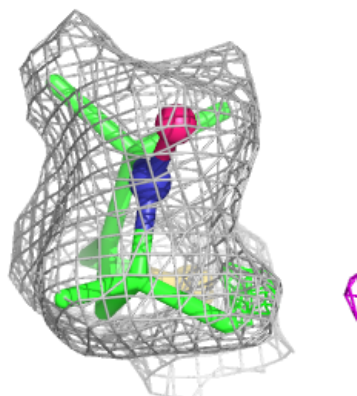
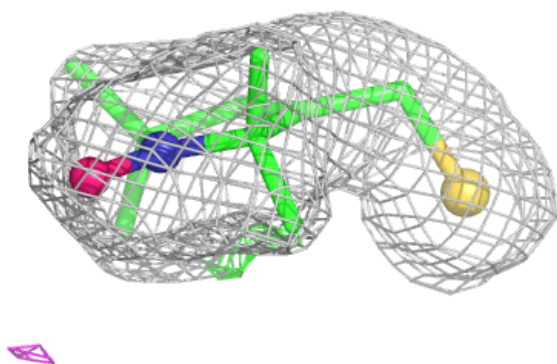
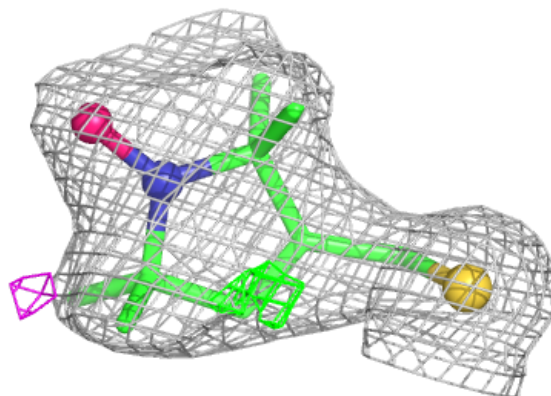
Electron density around MTN B 504:

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



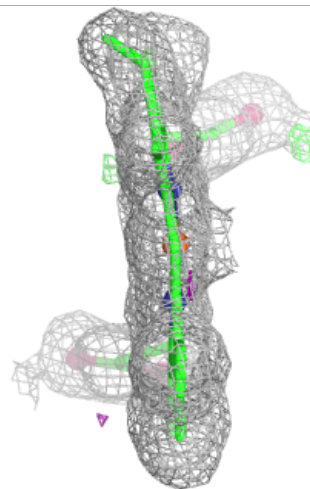
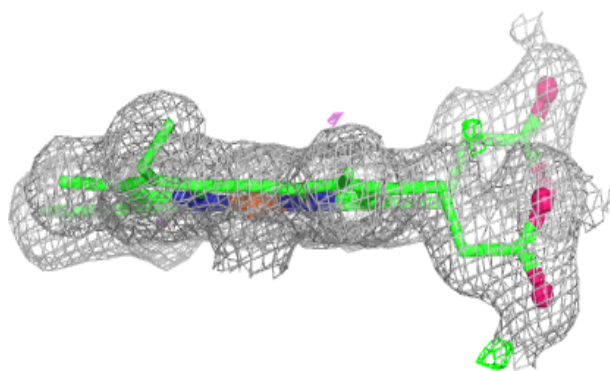
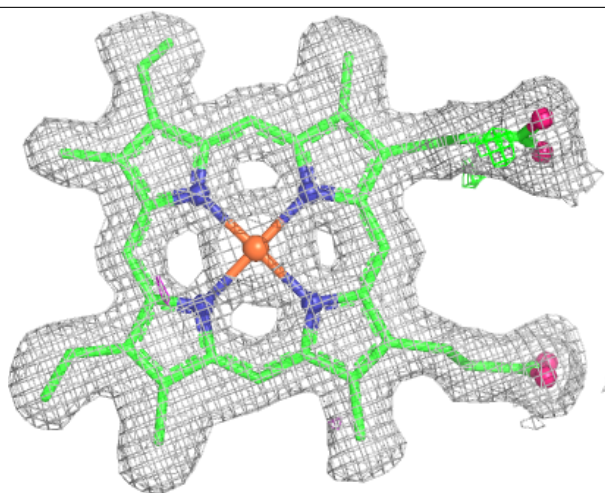
Electron density around MTN A 504:

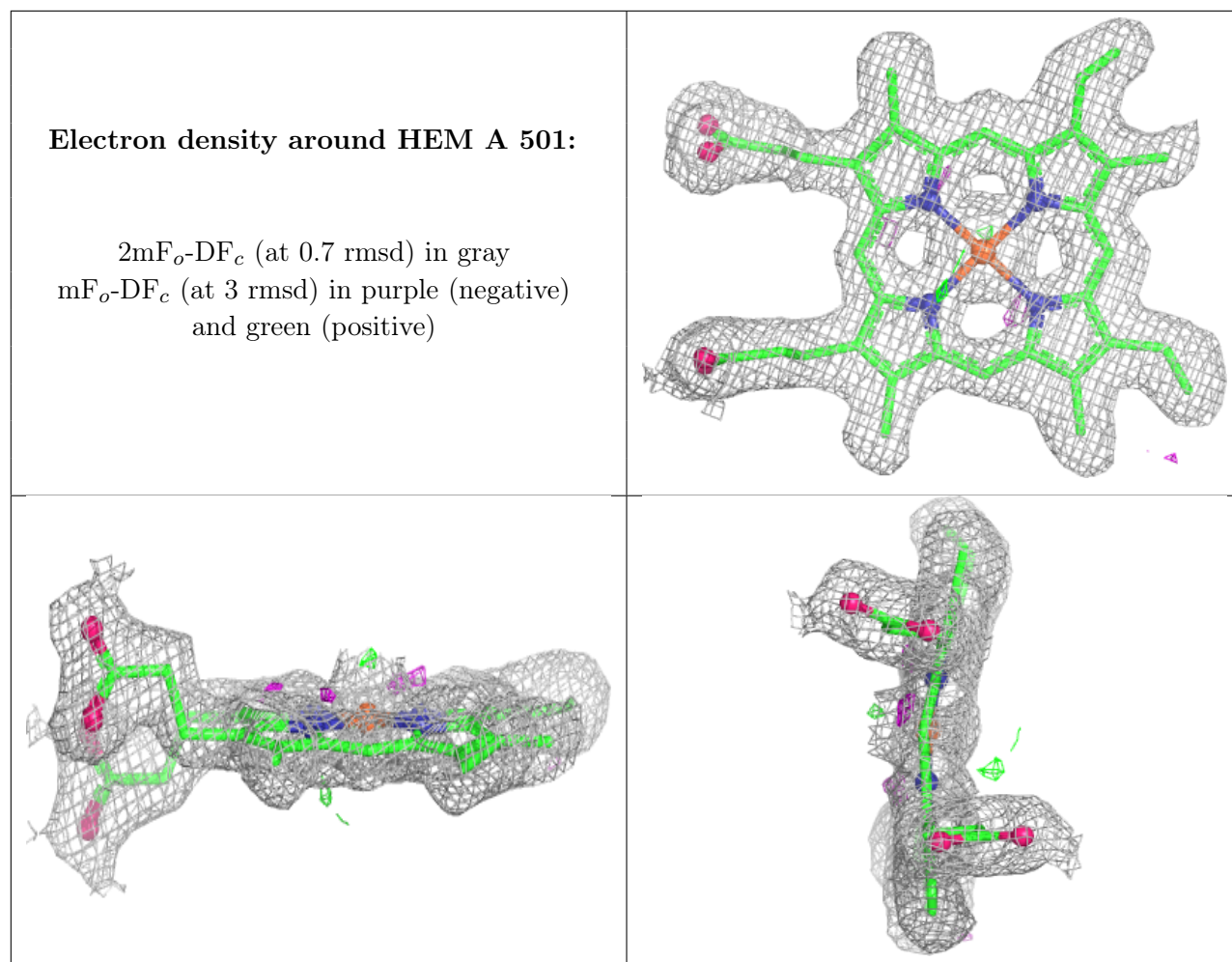
$2mF_o-DF_c$ (at 0.7 rmsd) in gray
 mF_o-DF_c (at 3 rmsd) in purple (negative)
and green (positive)



Electron density around HEM B 501:

$2mF_o-DF_c$ (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.