



# wwPDB X-ray Structure Validation Summary Report

Oct 30, 2017 – 06:35 PM EDT

PDB ID : 3RQO  
Title : Structure of the endothelial nitric oxide synthase heme domain in complex with 6-(((3R,4R)-4-(2-((1S,2R/1R,2S)-2-(3-Chlorophenyl)cyclopropylamino)ethoxy)pyrrolidin-3-yl)methyl)-4-methylpyridin-2-amine  
Authors : Li, H.; Delker, S.L.; Poulos, T.L.  
Deposited on : unknown  
Resolution : 2.08 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

<http://wwpdb.org/validation/2016/XrayValidationReportHelp>

with specific help available everywhere you see the  symbol.

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

MolProbity : **FAILED**  
Mogul : 1.7.2 (RC1), CSD as538be (2017)  
Xtriage (Phenix) : 1.9-1692  
EDS : rb-20030345  
Percentile statistics : 20161228.v01 (using entries in the PDB archive December 28th 2016)  
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) : rb-20030345

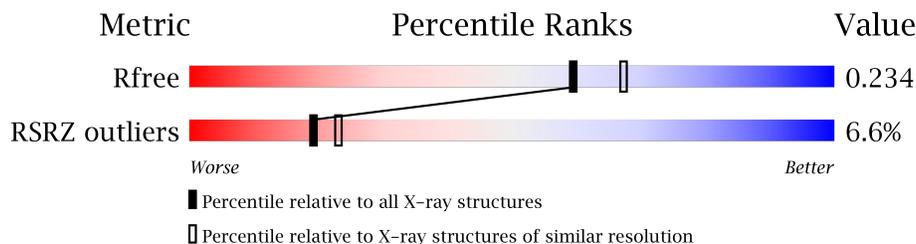
# 1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

*X-RAY DIFFRACTION*

The reported resolution of this entry is 2.08 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<br>(#Entries) | Similar resolution<br>(#Entries, resolution range(Å)) |
|---------------|-----------------------------|---|
| $R_{free}$    | 100719                      | 4955 (2.10-2.06)                                      |
| RSRZ outliers | 101464                      | 4991 (2.10-2.06)                                      |

MolProbity failed to run properly - the sequence quality summary graphics cannot be shown.

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 |
|-----|------|-------|-----|-----------|----------|---------|------------------|
| 5   | ACT  | A     | 860 | -         | -        | -       | X                |
| 5   | ACT  | B     | 860 | -         | -        | -       | X                |

## 2 Entry composition [i](#)

There are 9 unique types of molecules in this entry. The entry contains 7032 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.

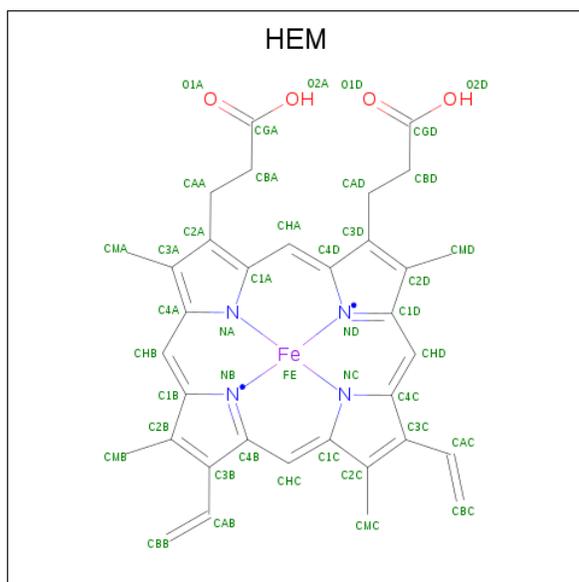
- Molecule 1 is a protein called Nitric oxide synthase, endothelial.

| Mol | Chain | Residues | Atoms         |           |          |          |         | ZeroOcc | AltConf | Trace |
|-----|-------|----------|---------------|-----------|----------|----------|---------|---------|---------|-------|
|     |       |          | Total         | C         | N        | O        | S       |         |         |       |
| 1   | A     | 404      | Total<br>3228 | C<br>2053 | N<br>568 | O<br>591 | S<br>16 | 0       | 3       | 0     |
| 1   | B     | 402      | Total<br>3198 | C<br>2034 | N<br>562 | O<br>586 | S<br>16 | 0       | 0       | 0     |

There are 2 discrepancies between the modelled and reference sequences:

| Chain | Residue | Modelled | Actual | Comment        | Reference  |
|-------|---------|----------|--------|----------------|------------|
| A     | 100     | ARG      | CYS    | SEE REMARK 999 | UNP P29473 |
| B     | 100     | ARG      | CYS    | SEE REMARK 999 | UNP P29473 |

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



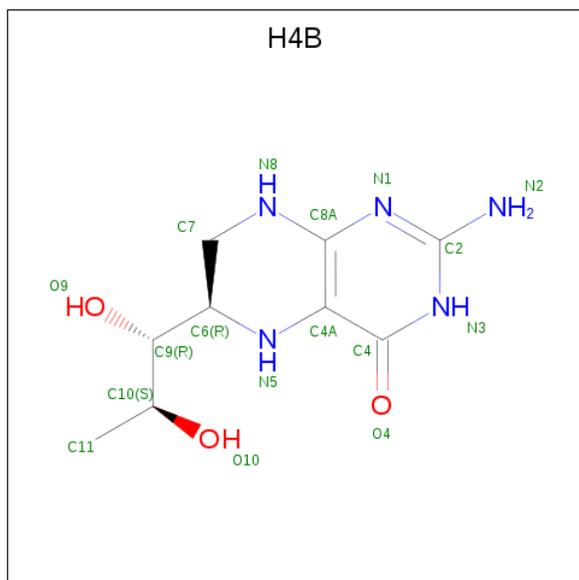
| Mol | Chain | Residues | Atoms       |         |         |        | ZeroOcc | AltConf |   |
|-----|-------|----------|-------------|---------|---------|--------|---------|---------|---|
|     |       |          | Total       | C       | Fe      | N      |         |         | O |
| 2   | A     | 1        | Total<br>43 | C<br>34 | Fe<br>1 | N<br>4 | O<br>4  | 0       | 0 |

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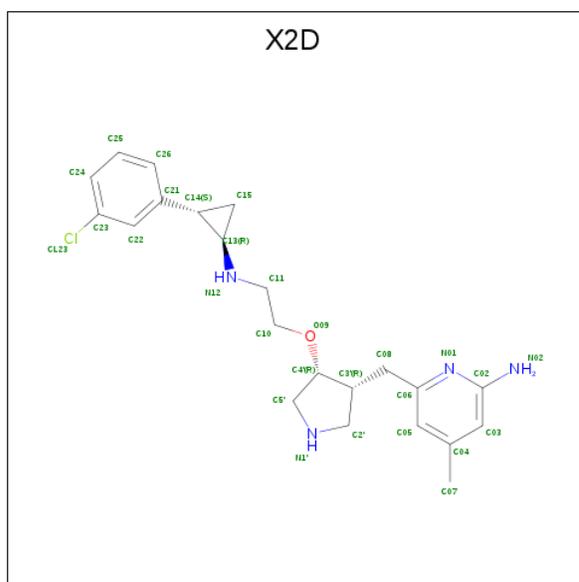
| Mol | Chain | Residues | Atoms |    |    |   | ZeroOcc | AltConf |   |
|-----|-------|----------|-------|----|----|---|---------|---------|---|
|     |       |          | Total | C  | Fe | N |         |         | O |
| 2   | B     | 1        | 43    | 34 | 1  | 4 | 4       | 0       | 0 |

- Molecule 3 is 5,6,7,8-TETRAHYDROBIOPTERIN (three-letter code: H4B) (formula:  $C_9H_{15}N_5O_3$ ).



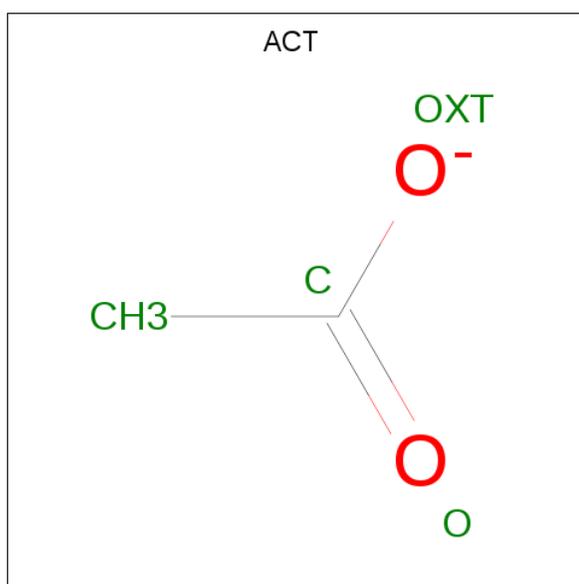
| Mol | Chain | Residues | Atoms |   |   |   | ZeroOcc | AltConf |
|-----|-------|----------|-------|---|---|---|---------|---------|
|     |       |          | Total | C | N | O |         |         |
| 3   | A     | 1        | 17    | 9 | 5 | 3 | 0       | 0       |
| 3   | B     | 1        | 17    | 9 | 5 | 3 | 0       | 0       |

- Molecule 4 is 6-[[[(3R,4R)-4-(2-[[[(1R,2S)-2-(3-chlorophenyl)cyclopropyl]amino]ethoxy]pyrrolidin-3-yl)methyl]-4-methylpyridin-2-amine (three-letter code: X2D) (formula:  $C_{22}H_{29}ClN_4O$ ).



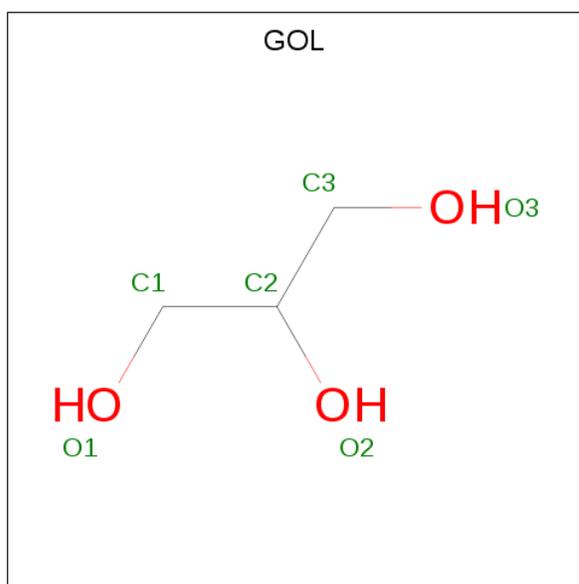
| Mol | Chain | Residues | Atoms |    |    |   | ZeroOcc | AltConf |   |
|-----|-------|----------|-------|----|----|---|---------|---------|---|
| 4   | A     | 1        | Total | C  | Cl | N | O       | 0       | 0 |
|     |       |          | 28    | 22 | 1  | 4 | 1       |         |   |
| 4   | B     | 1        | Total | C  | Cl | N | O       | 0       | 0 |
|     |       |          | 28    | 22 | 1  | 4 | 1       |         |   |

- Molecule 5 is ACETATE ION (three-letter code: ACT) (formula:  $C_2H_3O_2$ ).



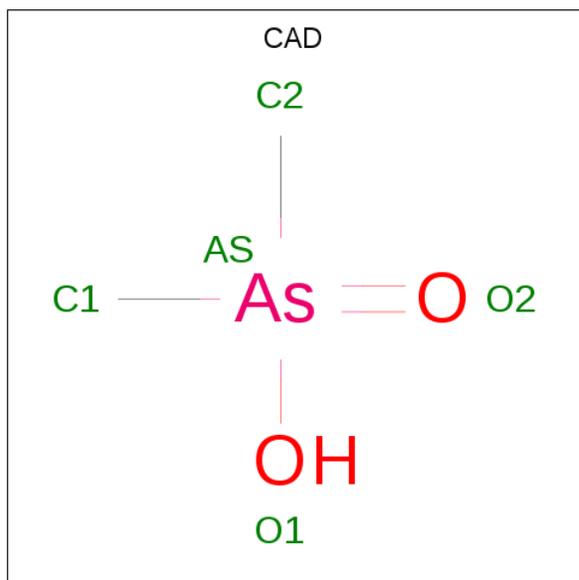
| Mol | Chain | Residues | Atoms |   | ZeroOcc | AltConf |   |
|-----|-------|----------|-------|---|---------|---------|---|
| 5   | A     | 1        | Total | C | O       | 0       | 0 |
|     |       |          | 4     | 2 | 2       |         |   |
| 5   | B     | 1        | Total | C | O       | 0       | 0 |
|     |       |          | 4     | 2 | 2       |         |   |

- Molecule 6 is GLYCEROL (three-letter code: GOL) (formula:  $C_3H_8O_3$ ).



| Mol | Chain | Residues | Atoms |   |   | ZeroOcc | AltConf |
|-----|-------|----------|-------|---|---|---------|---------|
| 6   | A     | 1        | Total | C | O | 0       | 0       |
|     |       |          | 6     | 3 | 3 |         |         |
| 6   | B     | 1        | Total | C | O | 0       | 0       |
|     |       |          | 6     | 3 | 3 |         |         |

- Molecule 7 is CACODYLIC ACID (three-letter code: CAD) (formula:  $C_2H_7AsO_2$ ).



| Mol | Chain | Residues | Atoms |    |   | ZeroOcc | AltConf |
|-----|-------|----------|-------|----|---|---------|---------|
| 7   | A     | 1        | Total | As | C | 0       | 0       |
|     |       |          | 3     | 1  | 2 |         |         |

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| Mol | Chain | Residues | Atoms |    |   | ZeroOcc | AltConf |
|-----|-------|----------|-------|----|---|---------|---------|
| 7   | B     | 1        | Total | As | C | 0       | 0       |
|     |       |          | 3     | 1  | 2 |         |         |

- Molecule 8 is ZINC ION (three-letter code: ZN) (formula: Zn).

| Mol | Chain | Residues | Atoms |    | ZeroOcc | AltConf |
|-----|-------|----------|-------|----|---------|---------|
| 8   | A     | 1        | Total | Zn | 0       | 0       |
|     |       |          | 1     | 1  |         |         |

- Molecule 9 is water.

| Mol | Chain | Residues | Atoms |     | ZeroOcc | AltConf |
|-----|-------|----------|-------|-----|---------|---------|
| 9   | A     | 198      | Total | O   | 0       | 0       |
|     |       |          | 198   | 198 |         |         |
| 9   | B     | 205      | Total | O   | 0       | 0       |
|     |       |          | 205   | 205 |         |         |

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### 3 Data and refinement statistics

| Property  | Value   | Source           |
|---|---|------------------|
| Space group   | P 21 21 21  | Depositor        |
| Cell constants<br>a, b, c, $\alpha$ , $\beta$ , $\gamma$                | 58.08Å 106.64Å 156.52Å<br>90.00° 90.00° 90.00°              | Depositor        |
| Resolution (Å)  | 35.11 – 2.08<br>35.11 – 2.08                                | Depositor<br>EDS |
| % Data completeness<br>(in resolution range)                            | 99.3 (35.11-2.08)<br>99.3 (35.11-2.08)                      | Depositor<br>EDS |
| $R_{merge}$   | 0.07  | Depositor        |
| $R_{sym}$   | 0.07  | Depositor        |
| $\langle I/\sigma(I) \rangle$ <sup>1</sup>                              | 2.68 (at 2.08Å)   | Xtrriage         |
| Refinement program  | REFMAC  | Depositor        |
| R, $R_{free}$   | 0.174 , 0.221<br>0.189 , 0.234                              | Depositor<br>DCC |
| $R_{free}$ test set   | 2930 reflections (5.25%)                                    | DCC              |
| Wilson B-factor (Å <sup>2</sup> )                                       | 35.3  | Xtrriage         |
| Anisotropy  | 0.234   | Xtrriage         |
| Bulk solvent $k_{sol}$ (e/Å <sup>3</sup> ), $B_{sol}$ (Å <sup>2</sup> ) | 0.37 , 40.1   | 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  | 0.96  | EDS              |
| Total number of atoms   | 7032  | wwPDB-VP         |
| Average B, all atoms (Å <sup>2</sup> )                                  | 40.0  | wwPDB-VP         |

Xtrriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 3.33% 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.

## 4 Model quality [i](#)

### 4.1 Standard geometry [i](#)

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### 4.2 Too-close contacts [i](#)

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### 4.3 Torsion angles [i](#)

#### 4.3.1 Protein backbone [i](#)

MolProbity failed to run properly - this section is therefore empty.

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

MolProbity failed to run properly - this section is therefore empty.

#### 4.3.3 RNA [i](#)

MolProbity failed to run properly - this section is therefore empty.

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

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

### 4.5 Carbohydrates [i](#)

There are no carbohydrates in this entry.

### 4.6 Ligand geometry [i](#)

Of 13 ligands modelled in this entry, 1 is monoatomic - leaving 12 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 |
| 2   | HEM  | A     | 500 | 1    | 28,50,50     | 2.11 | 9 (32%)  | 17,82,82    | 1.49 | 4 (23%)  |
| 3   | H4B  | A     | 600 | -    | 14,18,18     | 0.80 | 0        | 12,26,26    | 2.57 | 7 (58%)  |
| 4   | X2D  | A     | 800 | -    | 26,31,31     | 1.11 | 1 (3%)   | 30,43,43    | 2.29 | 8 (26%)  |
| 5   | ACT  | A     | 860 | -    | 1,3,3        | 1.30 | 0        | 0,3,3       | 0.00 | -        |
| 6   | GOL  | A     | 880 | -    | 5,5,5        | 0.46 | 0        | 5,5,5       | 0.13 | 0        |
| 7   | CAD  | A     | 950 | -    | 0,2,4        | 0.00 | -        | 0,1,6       | 0.00 | -        |
| 2   | HEM  | B     | 500 | 1    | 28,50,50     | 2.24 | 7 (25%)  | 17,82,82    | 2.04 | 4 (23%)  |
| 3   | H4B  | B     | 600 | -    | 14,18,18     | 0.83 | 0        | 12,26,26    | 2.14 | 5 (41%)  |
| 4   | X2D  | B     | 800 | -    | 26,31,31     | 0.82 | 1 (3%)   | 30,43,43    | 2.14 | 7 (23%)  |
| 5   | ACT  | B     | 860 | -    | 1,3,3        | 1.10 | 0        | 0,3,3       | 0.00 | -        |
| 6   | GOL  | B     | 880 | -    | 5,5,5        | 0.40 | 0        | 5,5,5       | 0.74 | 0        |
| 7   | CAD  | B     | 950 | -    | 0,2,4        | 0.00 | -        | 0,1,6       | 0.00 | -        |

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   | HEM  | A     | 500 | 1    | -       | 0/6/54/54  | 0/0/8/8 |
| 3   | H4B  | A     | 600 | -    | -       | 0/8/17/17  | 0/2/2/2 |
| 4   | X2D  | A     | 800 | -    | -       | 0/13/30/30 | 0/3/4/4 |
| 5   | ACT  | A     | 860 | -    | -       | 0/0/0/0    | 0/0/0/0 |
| 6   | GOL  | A     | 880 | -    | -       | 0/4/4/4    | 0/0/0/0 |
| 7   | CAD  | A     | 950 | -    | -       | 0/0/0/0    | 0/0/0/0 |
| 2   | HEM  | B     | 500 | 1    | -       | 0/6/54/54  | 0/0/8/8 |
| 3   | H4B  | B     | 600 | -    | -       | 0/8/17/17  | 0/2/2/2 |
| 4   | X2D  | B     | 800 | -    | -       | 0/13/30/30 | 0/3/4/4 |
| 5   | ACT  | B     | 860 | -    | -       | 0/0/0/0    | 0/0/0/0 |
| 6   | GOL  | B     | 880 | -    | -       | 0/4/4/4    | 0/0/0/0 |
| 7   | CAD  | B     | 950 | -    | -       | 0/0/0/0    | 0/0/0/0 |

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

| Mol | Chain | Res | Type | Atoms   | Z     | Observed(Å) | Ideal(Å) |
|-----|-------|-----|------|---------|-------|-------------|----------|
| 2   | B     | 500 | HEM  | C3B-C2B | -4.84 | 1.34        | 1.40     |
| 2   | A     | 500 | HEM  | C3B-C2B | -4.41 | 1.34        | 1.40     |

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| Mol | Chain | Res | Type | Atoms   | Z     | Observed(Å) | Ideal(Å) |
|-----|-------|-----|------|---------|-------|-------------|----------|
| 2   | B     | 500 | HEM  | C3C-C2C | -3.85 | 1.35        | 1.40     |
| 2   | A     | 500 | HEM  | C3C-C2C | -3.52 | 1.35        | 1.40     |
| 2   | A     | 500 | HEM  | C4D-ND  | 2.02  | 1.39        | 1.36     |

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

| Mol | Chain | Res | Type | Atoms       | Z     | Observed(°) | Ideal(°) |
|-----|-------|-----|------|-------------|-------|-------------|----------|
| 4   | A     | 800 | X2D  | C15-C14-C21 | -7.02 | 109.43      | 122.31   |
| 4   | B     | 800 | X2D  | C15-C14-C21 | -6.89 | 109.68      | 122.31   |
| 2   | B     | 500 | HEM  | CBD-CAD-C3D | -5.02 | 102.90      | 112.47   |
| 2   | B     | 500 | HEM  | CBA-CAA-C2A | -4.79 | 103.32      | 112.48   |
| 4   | A     | 800 | X2D  | C04-C05-C06 | -3.76 | 117.90      | 120.26   |

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

No monomer is involved in short contacts.

#### 4.7 Other polymers [i](#)

There are no such residues in this entry.

#### 4.8 Polymer linkage issues [i](#)

There are no chain breaks in this entry.

## 5 Fit of model and data [i](#)

### 5.1 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     | 404/443 (91%) | 0.40   | 33 (8%) 12 15 | 26, 38, 61, 81        | 0     |
| 1   | B     | 402/443 (90%) | 0.24   | 20 (4%) 30 34 | 25, 39, 61, 83        | 0     |
| All | All   | 806/886 (90%) | 0.32   | 53 (6%) 19 23 | 25, 38, 61, 83        | 0     |

The worst 5 of 53 RSRZ outliers are listed below:

| Mol | Chain | Res | Type | RSRZ |
|-----|-------|-----|------|------|
| 1   | A     | 239 | GLY  | 5.7  |
| 1   | A     | 259 | GLN  | 5.7  |
| 1   | B     | 259 | GLN  | 5.1  |
| 1   | A     | 123 | GLU  | 4.6  |
| 1   | A     | 122 | ALA  | 4.5  |

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

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

### 5.3 Carbohydrates [i](#)

There are no carbohydrates in this entry.

### 5.4 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, 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  | LLDF  | B-factors( $\text{\AA}^2$ ) | Q<0.9 |
|-----|------|-------|-----|-------|------|------|-------|-----------------------------|-------|
| 5   | ACT  | B     | 860 | 4/4   | 0.97 | 0.25 | 7.75  | 47,49,49,51                 | 0     |
| 5   | ACT  | A     | 860 | 4/4   | 0.94 | 0.19 | 3.38  | 45,46,48,48                 | 0     |
| 6   | GOL  | A     | 880 | 6/6   | 0.85 | 0.21 | 1.90  | 55,56,57,57                 | 0     |
| 6   | GOL  | B     | 880 | 6/6   | 0.87 | 0.20 | 1.34  | 52,53,55,55                 | 0     |
| 2   | HEM  | B     | 500 | 43/43 | 0.98 | 0.17 | 0.80  | 27,30,35,36                 | 0     |
| 3   | H4B  | B     | 600 | 17/17 | 0.99 | 0.16 | 0.54  | 23,28,31,34                 | 0     |
| 2   | HEM  | A     | 500 | 43/43 | 0.98 | 0.17 | 0.54  | 21,28,34,39                 | 0     |
| 3   | H4B  | A     | 600 | 17/17 | 0.98 | 0.15 | 0.31  | 23,28,31,31                 | 0     |
| 4   | X2D  | B     | 800 | 28/28 | 0.96 | 0.16 | 0.12  | 33,38,47,53                 | 0     |
| 4   | X2D  | A     | 800 | 28/28 | 0.92 | 0.17 | 0.02  | 29,38,41,47                 | 0     |
| 8   | ZN   | A     | 900 | 1/1   | 1.00 | 0.08 | -0.89 | 32,32,32,32                 | 0     |
| 7   | CAD  | A     | 950 | 3/5   | 0.98 | 0.07 | -1.95 | 70,70,70,71                 | 0     |
| 7   | CAD  | B     | 950 | 3/5   | 0.98 | 0.22 | -     | 77,77,77,77                 | 0     |

## 5.5 Other polymers [i](#)

There are no such residues in this entry.