



# Full wwPDB X-ray Structure Validation Report ⓘ

Oct 30, 2017 – 06:36 PM EDT

PDB ID : 3RQK  
Title : Structure of the neuronal nitric oxide synthase heme domain in complex with 4-methyl-6-{[(3R,4R)-4-(2-{[(1R,2S)-2-(3-methylphenyl)cyclopropyl]amino}ethoxy)pyrrolidin-3-yl]methyl}pyridin-2-amine and its isomer  
Authors : Li, H.; Delker, S.L.; Poulos, T.L.  
Deposited on : unknown  
Resolution : 2.21 Å(reported)

This is a Full wwPDB X-ray Structure Validation 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

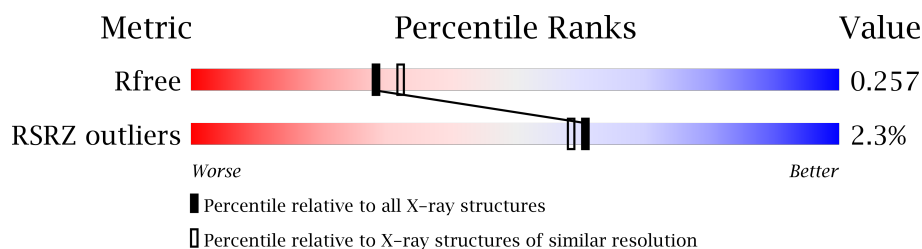
# 1 Overall quality at a glance

The following experimental techniques were used to determine the structure:

*X-RAY DIFFRACTION*

The reported resolution of this entry is 2.21 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))
$R_{free}$	100719	4744 (2.24-2.20)
RSRZ outliers	101464	4776 (2.24-2.20)

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 7 unique types of molecules in this entry. The entry contains 7016 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, brain.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	407	Total	C	N	O	S	0	0	0
			3313	2121	566	605	21			
1	B	411	Total	C	N	O	S	0	1	0
			3348	2142	574	610	22			

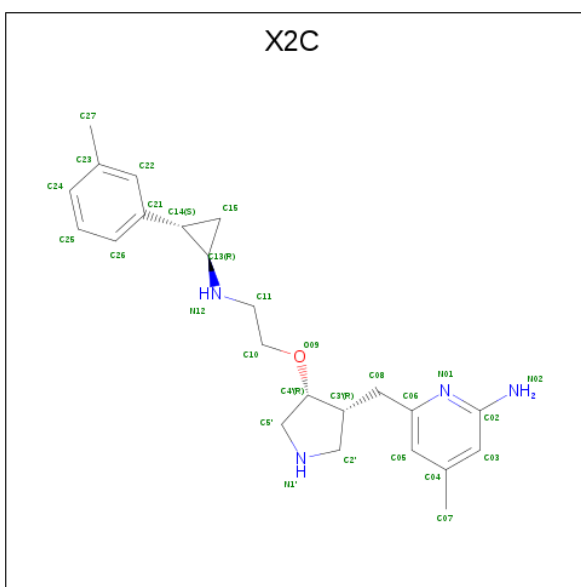
- Molecule 2 is PROTOPORPHYRIN IX CONTAINING FE (three-letter code: HEM) (formula:  $C_{34}H_{32}FeN_4O_4$ ).





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

- Molecule 4 is 4-methyl-6-{[(3R,4R)-4-(2-{[(1R,2S)-2-(3-methylphenyl)cyclopropyl]amino}ethoxy)pyrrolidin-3-yl]methyl}pyridin-2-amine (three-letter code: X2C) (formula: C<sub>23</sub>H<sub>32</sub>N<sub>4</sub>O).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
4	A	1	Total	C	N	O	0	0
			28	23	4	1		

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Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
4	B	1	Total	C	N	O	0	0
			28	23	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 ZINC ION (three-letter code: ZN) (formula:  $Zn$ ).

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
6	A	1	Total	Zn	0	0
			1	1		

- Molecule 7 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
7	A	74	Total	O	0	0
			74	74		
7	B	96	Total	O	0	0
			96	96		

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### 3 Data and refinement statistics

Property	Value	Source
Space group	P 21 21 21	Depositor
Cell constants a, b, c, $\alpha$ , $\beta$ , $\gamma$	51.95Å 111.53Å 164.39Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	49.89 – 2.21 49.18 – 2.21	Depositor EDS
% Data completeness (in resolution range)	96.6 (49.89-2.21) 96.6 (49.18-2.21)	Depositor EDS
$R_{merge}$	0.08	Depositor
$R_{sym}$	0.08	Depositor
$\langle I/\sigma(I) \rangle$ <sup>1</sup>	3.27 (at 2.20Å)	Xtriage
Refinement program	REFMAC	Depositor
R, $R_{free}$	0.201 , 0.258 0.205 , 0.257	Depositor DCC
$R_{free}$ test set	2394 reflections (5.32%)	DCC
Wilson B-factor (Å <sup>2</sup> )	53.0	Xtriage
Anisotropy	0.161	Xtriage
Bulk solvent $k_{sol}$ (e/Å <sup>3</sup> ), $B_{sol}$ (Å <sup>2</sup> )	0.34 , 39.0	EDS
L-test for twinning <sup>2</sup>	$\langle  L  \rangle = 0.49$ , $\langle L^2 \rangle = 0.32$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
$F_o, F_c$ correlation	0.96	EDS
Total number of atoms	7016	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	64.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 4.04% 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](#)

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

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

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### 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 9 ligands modelled in this entry, 1 is monoatomic - leaving 8 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	750	1	28,50,50	2.13	8 (28%)	17,82,82	2.25	5 (29%)
3	H4B	A	760	-	14,18,18	0.40	0	12,26,26	2.43	5 (41%)
4	X2C	A	800	-	26,31,31	0.85	2 (7%)	30,43,43	1.83	7 (23%)
5	ACT	A	860	-	1,3,3	1.09	0	0,3,3	0.00	-
2	HEM	B	750	1	28,50,50	2.27	10 (35%)	17,82,82	1.89	4 (23%)
3	H4B	B	760	-	14,18,18	0.77	0	12,26,26	2.07	6 (50%)
4	X2C	B	800	-	26,31,31	0.87	1 (3%)	30,43,43	2.51	11 (36%)
5	ACT	B	860	-	1,3,3	1.22	0	0,3,3	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	750	1	-	0/6/54/54	0/0/8/8
3	H4B	A	760	-	-	0/8/17/17	0/2/2/2
4	X2C	A	800	-	-	0/13/30/30	0/3/4/4
5	ACT	A	860	-	-	0/0/0/0	0/0/0/0
2	HEM	B	750	1	-	0/6/54/54	0/0/8/8
3	H4B	B	760	-	-	0/8/17/17	0/2/2/2
4	X2C	B	800	-	-	0/13/30/30	0/3/4/4
5	ACT	B	860	-	-	0/0/0/0	0/0/0/0

All (21) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	B	750	HEM	C3C-C2C	-5.56	1.33	1.40
2	A	750	HEM	C3B-C2B	-5.01	1.33	1.40
2	A	750	HEM	C3C-C2C	-3.94	1.35	1.40
2	B	750	HEM	C3B-C2B	-3.71	1.35	1.40
4	A	800	X2C	C13-N12	-2.02	1.43	1.47
2	B	750	HEM	CMA-C3A	2.19	1.56	1.51
4	B	800	X2C	C15-C14	2.25	1.54	1.50
2	B	750	HEM	C1C-NC	2.27	1.39	1.36
4	A	800	X2C	C15-C14	2.29	1.54	1.50
2	A	750	HEM	CMA-C3A	2.32	1.56	1.51

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Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
2	A	750	HEM	C1B-NB	2.34	1.39	1.36
2	B	750	HEM	CMB-C2B	2.53	1.57	1.51
2	A	750	HEM	CMC-C2C	2.58	1.57	1.51
2	A	750	HEM	C3C-CAC	2.68	1.53	1.47
2	B	750	HEM	C3C-CAC	2.77	1.53	1.47
2	B	750	HEM	C3B-CAB	2.85	1.53	1.47
2	B	750	HEM	C4D-ND	2.88	1.40	1.36
2	B	750	HEM	CAA-C2A	2.93	1.57	1.52
2	A	750	HEM	C3B-CAB	3.78	1.55	1.47
2	A	750	HEM	C3D-C2D	4.79	1.51	1.37
2	B	750	HEM	C3D-C2D	5.08	1.52	1.37

All (38) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
4	B	800	X2C	C15-C14-C21	-8.31	107.06	122.31
4	A	800	X2C	C15-C14-C21	-4.95	113.23	122.31
2	A	750	HEM	CBD-CAD-C3D	-4.82	103.28	112.47
2	B	750	HEM	CBD-CAD-C3D	-4.07	104.71	112.47
2	A	750	HEM	C1D-C2D-C3D	-3.73	104.40	107.00
3	A	760	H4B	N3-C2-N1	-3.26	120.16	125.45
2	A	750	HEM	CBA-CAA-C2A	-2.83	107.07	112.48
4	B	800	X2C	C04-C05-C06	-2.56	118.65	120.26
4	A	800	X2C	C05-C06-N01	-2.53	120.17	122.91
3	B	760	H4B	N3-C2-N1	-2.42	121.53	125.45
4	B	800	X2C	C26-C21-C14	-2.23	116.77	121.07
2	A	750	HEM	C3C-C4C-NC	-2.21	106.77	110.94
4	B	800	X2C	C05-C06-N01	-2.19	120.54	122.91
2	B	750	HEM	C3C-C4C-NC	-2.17	106.84	110.94
2	B	750	HEM	CAA-CBA-CGA	-2.13	109.01	112.66
3	B	760	H4B	C6-C7-N8	-2.04	107.78	111.01
4	A	800	X2C	C27-C23-C22	-2.02	117.97	120.94
3	B	760	H4B	N2-C2-N1	2.05	120.52	117.24
4	A	800	X2C	C08-C06-N01	2.08	120.39	117.12
4	A	800	X2C	C2'-N1'-C5'	2.13	110.37	105.40
4	B	800	X2C	C10-C11-N12	2.26	117.28	111.55
4	B	800	X2C	C25-C26-C21	2.38	123.57	120.64
3	B	760	H4B	C4-N3-C2	2.40	119.51	116.06
3	B	760	H4B	C2-N1-C8A	2.54	120.23	114.51
4	B	800	X2C	C2'-N1'-C5'	2.61	111.47	105.40
4	A	800	X2C	N02-C02-N01	2.62	121.05	116.64
3	A	760	H4B	N2-C2-N1	2.84	121.78	117.24

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Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
3	A	760	H4B	C2-N1-C8A	2.87	120.97	114.51
4	B	800	X2C	C10-O09-C4'	3.01	120.82	113.90
4	B	800	X2C	N02-C02-N01	3.28	122.17	116.64
4	B	800	X2C	C22-C21-C14	3.36	126.26	120.10
3	A	760	H4B	C4-C4A-C8A	4.29	118.45	114.56
3	A	760	H4B	C4-N3-C2	4.34	122.30	116.06
2	B	750	HEM	C4C-C3C-C2C	4.56	110.08	106.90
2	A	750	HEM	C4C-C3C-C2C	4.65	110.15	106.90
3	B	760	H4B	C4-C4A-C8A	4.66	118.78	114.56
4	A	800	X2C	C02-N01-C06	5.36	121.96	118.17
4	B	800	X2C	C02-N01-C06	5.37	121.97	118.17

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	407/422 (96%)	0.14	16 (3%) 40 38	40, 68, 109, 140	0
1	B	411/422 (97%)	-0.01	3 (0%) 87 86	39, 56, 84, 98	0
All	All	818/844 (96%)	0.06	19 (2%) 61 58	39, 61, 102, 140	0

All (19) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	373	GLY	5.2
1	B	300	PHE	4.9
1	A	351	LYS	3.4
1	A	352	ASP	3.4
1	A	488	PRO	3.3
1	A	355	PHE	3.0
1	A	322	LEU	2.8
1	A	484	GLY	2.8
1	A	716	TRP	2.8
1	A	324	THR	2.7
1	A	370	LYS	2.6
1	A	503	GLU	2.4
1	A	328	GLU	2.3
1	B	328	GLU	2.3
1	A	612	LYS	2.3
1	B	392	SER	2.2
1	A	388	ILE	2.2
1	A	554	PHE	2.1
1	A	372	PHE	2.0

### 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(Å <sup>2</sup> )	Q<0.9
5	ACT	A	860	4/4	0.94	0.33	13.43	74,76,76,79	0
5	ACT	B	860	4/4	0.89	0.21	3.04	68,70,71,75	0
2	HEM	A	750	43/43	0.97	0.15	1.18	41,47,55,57	0
4	X2C	B	800	28/28	0.92	0.15	0.45	48,52,56,57	0
4	X2C	A	800	28/28	0.91	0.14	0.25	46,60,66,69	0
2	HEM	B	750	43/43	0.98	0.15	0.06	38,45,50,52	0
3	H4B	B	760	17/17	0.95	0.09	-1.16	44,47,54,55	0
3	H4B	A	760	17/17	0.92	0.11	-1.29	47,50,56,56	0
6	ZN	A	900	1/1	0.99	0.07	-1.49	65,65,65,65	0

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

There are no such residues in this entry.