



# wwPDB X-ray Structure Validation Summary Report ⓘ

Feb 1, 2016 – 08:07 AM GMT

PDB ID : 3DF0  
Title : Calcium-dependent complex between m-calpain and calpastatin  
Authors : Moldoveanu, T.; Gehring, K.; Green, D.R.  
Deposited on : 2008-06-11  
Resolution : 2.95 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 : 4.02b-467  
Mogul : 1.7 (RC4), CSD as536be (2015)  
Xtriage (Phenix) : 1.9-1692  
EDS : rb-20026688  
Percentile statistics : 20151230.v01 (using entries in the PDB archive December 30th 2015)  
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) : trunk26865

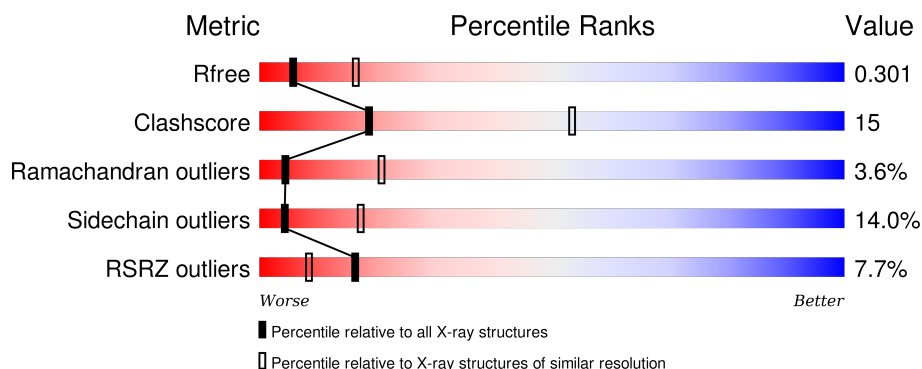
# 1 Overall quality at a glance ⓘ

The following experimental techniques were used to determine the structure:

## *X-RAY DIFFRACTION*

The reported resolution of this entry is 2.95 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))
$R_{free}$	91344	2184 (3.00-2.92)
Clashscore	102246	2552 (3.00-2.92)
Ramachandran outliers	100387	2468 (3.00-2.92)
Sidechain outliers	100360	2471 (3.00-2.92)
RSRZ outliers	91569	2201 (3.00-2.92)

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 on the lower bar indicate the fraction of residues that contain outliers for  $\geq 3$ , 2, 1 and 0 types of geometric quality criteria. 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	714	<div> <div>7%</div> <div>58%</div> <div>30%</div> <div>6%</div> <div>5%</div> </div>
2	B	184	<div> <div>5%</div> <div>59%</div> <div>30%</div> <div>• • 5%</div> </div>
3	C	86	<div> <div>9%</div> <div>43%</div> <div>17%</div> <div>5%</div> <div>35%</div> </div>

## 2 Entry composition [i](#)

There are 4 unique types of molecules in this entry. The entry contains 7300 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 Calpain-2 catalytic subunit.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	676	Total	C	N	O	S	0	0	0
			5453	3468	915	1047	23			

There are 15 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	105	SER	CYS	ENGINEERED	UNP Q07009
A	701	GLY	-	EXPRESSION TAG	UNP Q07009
A	702	LYS	-	EXPRESSION TAG	UNP Q07009
A	703	LEU	-	EXPRESSION TAG	UNP Q07009
A	704	ALA	-	EXPRESSION TAG	UNP Q07009
A	705	ALA	-	EXPRESSION TAG	UNP Q07009
A	706	ALA	-	EXPRESSION TAG	UNP Q07009
A	707	LEU	-	EXPRESSION TAG	UNP Q07009
A	708	GLU	-	EXPRESSION TAG	UNP Q07009
A	709	HIS	-	EXPRESSION TAG	UNP Q07009
A	710	HIS	-	EXPRESSION TAG	UNP Q07009
A	711	HIS	-	EXPRESSION TAG	UNP Q07009
A	712	HIS	-	EXPRESSION TAG	UNP Q07009
A	713	HIS	-	EXPRESSION TAG	UNP Q07009
A	714	HIS	-	EXPRESSION TAG	UNP Q07009

- Molecule 2 is a protein called Calpain small subunit 1.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
2	B	174	Total	C	N	O	S	0	0	0
			1411	888	243	270	10			

There is a discrepancy between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
B	87	MET	SER	VARIANT	UNP Q64537

- Molecule 3 is a protein called Calpastatin.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
3	C	56	Total	C	N	O	S	0	0	0
			426	270	64	90	2			

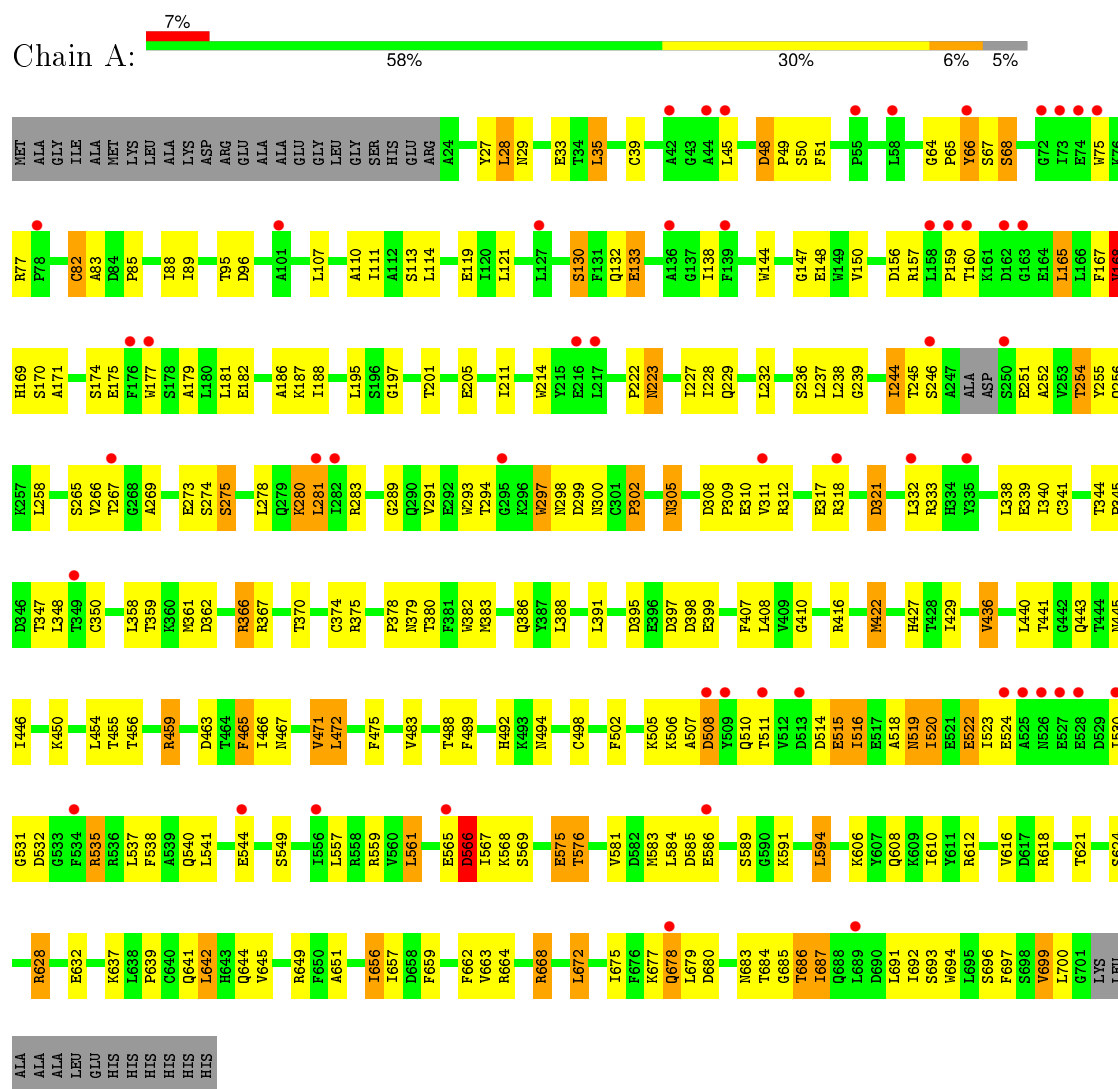
- Molecule 4 is CALCIUM ION (three-letter code: CA) (formula: Ca).

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	B	4	Total	Ca	0	0
			4	4		
4	A	6	Total	Ca	0	0
			6	6		

### 3 Residue-property plots

These plots are drawn for all protein, RNA and DNA chains in the entry. The first graphic for a chain summarises the proportions of errors 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.

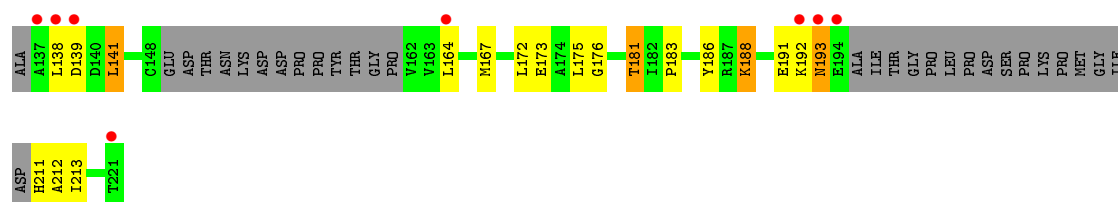
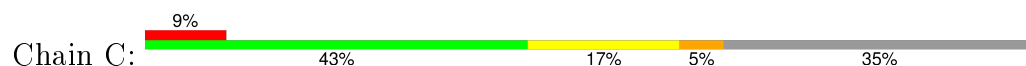
#### • Molecule 1: Calpain-2 catalytic subunit



#### • Molecule 2: Calpain small subunit 1



- Molecule 3: Calpastatin



## 4 Data and refinement statistics

Property	Value	Source
Space group	P 42	Depositor
Cell constants a, b, c, $\alpha$ , $\beta$ , $\gamma$	147.39Å 147.39Å 47.22Å 90.00° 90.00° 90.00°	Depositor
Resolution (Å)	49.15 – 2.95 46.61 – 2.90	Depositor EDS
% Data completeness (in resolution range)	99.8 (49.15-2.95) 99.8 (46.61-2.90)	Depositor EDS
$R_{merge}$	0.10	Depositor
$R_{sym}$	(Not available)	Depositor
$\langle I/\sigma(I) \rangle$ <sup>1</sup>	1.78 (at 2.91Å)	Xtriage
Refinement program	REFMAC 5.2.0019	Depositor
R, $R_{free}$	0.229 , 0.299 0.223 , 0.301	Depositor DCC
$R_{free}$ test set	1115 reflections (5.37%)	DCC
Wilson B-factor (Å <sup>2</sup> )	83.8	Xtriage
Anisotropy	0.028	Xtriage
Bulk solvent $k_{sol}$ (e/Å <sup>3</sup> ), $B_{sol}$ (Å <sup>2</sup> )	0.29 , 89.4	EDS
Estimated twinning fraction	0.031 for h,-k,-l	Xtriage
L-test for twinning <sup>2</sup>	$\langle  L  \rangle = 0.48$ , $\langle L^2 \rangle = 0.31$	Xtriage
Outliers	0 of 22986 reflections	Xtriage
$F_o, F_c$ correlation	0.93	EDS
Total number of atoms	7300	wwPDB-VP
Average B, all atoms (Å <sup>2</sup> )	96.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 3.48% 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, 0.375 respectively for untwinned datasets, and 0.333, 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:  
CA

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	0.79	7/5575 (0.1%)	0.75	2/7536 (0.0%)
2	B	0.61	0/1438	0.72	0/1932
3	C	0.75	2/430 (0.5%)	0.77	0/580
All	All	0.76	9/7443 (0.1%)	0.74	2/10048 (0.0%)

The worst 5 of 9 bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
1	A	310	GLU	CD-OE1	21.07	1.48	1.25
1	A	305	ASN	CG-OD1	15.23	1.57	1.24
1	A	305	ASN	CG-ND2	13.95	1.67	1.32
3	C	188	LYS	CE-NZ	8.16	1.69	1.49
1	A	544	GLU	CD-OE1	8.01	1.34	1.25

All (2) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
1	A	305	ASN	CB-CG-OD1	5.37	132.34	121.60
1	A	375	ARG	NE-CZ-NH2	-5.09	117.76	120.30

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	5453	0	5294	181	0
2	B	1411	0	1362	42	0
3	C	426	0	413	14	0
4	A	6	0	0	0	0
4	B	4	0	0	0	0
All	All	7300	0	7069	221	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 15.

The worst 5 of 221 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)
3:C:188:LYS:CE	3:C:188:LYS:NZ	1.69	1.55
1:A:305:ASN:ND2	1:A:305:ASN:CG	1.67	1.45
1:A:668:ARG:HG3	1:A:668:ARG:HH11	1.15	1.08
1:A:679:LEU:HD23	1:A:687:ILE:HD11	1.07	1.06
2:B:268:MET:HA	2:B:268:MET:CE	1.88	1.01

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	672/714 (94%)	565 (84%)	84 (12%)	23 (3%)	5	22
2	B	172/184 (94%)	155 (90%)	10 (6%)	7 (4%)	3	17
3	C	50/86 (58%)	38 (76%)	10 (20%)	2 (4%)	4	18
All	All	894/984 (91%)	758 (85%)	104 (12%)	32 (4%)	4	21

5 of 32 Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	A	65	PRO
1	A	168	VAL
1	A	252	ALA
1	A	298	ASN
1	A	441	THR

### 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	587/613 (96%)	504 (86%)	83 (14%)	4	17
2	B	153/162 (94%)	133 (87%)	20 (13%)	5	20
3	C	47/74 (64%)	40 (85%)	7 (15%)	4	15
All	All	787/849 (93%)	677 (86%)	110 (14%)	4	17

5 of 110 residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	471	VAL
1	A	568	LYS
2	B	265	GLN
1	A	508	ASP
1	A	522	GLU

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

Mol	Chain	Res	Type
1	A	608	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 carbohydrates in this entry.

## 5.6 Ligand geometry ⓘ

Of 10 ligands modelled in this entry, 10 are monoatomic - leaving 0 for Mogul analysis.

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 ⓘ

There are no such residues in this entry.

## 5.8 Polymer linkage issues ⓘ

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<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	676/714 (94%)	0.44	52 (7%) 16 8	75, 97, 108, 127	0
2	B	174/184 (94%)	0.24	10 (5%) 27 15	73, 94, 106, 112	0
3	C	56/86 (65%)	0.84	8 (14%) 4 2	92, 103, 110, 117	0
All	All	906/984 (92%)	0.42	70 (7%) 16 8	73, 97, 108, 127	0

The worst 5 of 70 RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
3	C	193	ASN	7.0
1	A	509	TYR	6.4
1	A	534	PHE	6.1
2	B	110	LEU	6.0
1	A	44	ALA	5.4

### 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 carbohydrates 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. 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
4	CA	A	716	1/1	0.94	0.08	-1.30	119,119,119,119	0
4	CA	B	601	1/1	0.92	0.07	-1.40	105,105,105,105	0
4	CA	A	717	1/1	0.94	0.04	-1.43	130,130,130,130	0
4	CA	A	718	1/1	0.87	0.06	-2.10	103,103,103,103	0
4	CA	B	603	1/1	0.98	0.03	-2.24	79,79,79,79	0
4	CA	B	604	1/1	0.93	0.06	-2.77	87,87,87,87	0
4	CA	A	715	1/1	0.97	0.03	-2.92	96,96,96,96	0
4	CA	B	602	1/1	0.91	0.07	-3.10	94,94,94,94	0
4	CA	A	720	1/1	0.91	0.07	-3.67	72,72,72,72	0
4	CA	A	719	1/1	0.99	0.03	-4.96	71,71,71,71	0

## 6.5 Other polymers [i](#)

There are no such residues in this entry.