



Full wwPDB X-ray Structure Validation Report ⓘ

May 28, 2014 – 04:20 AM EDT

PDB ID : 4PP9
Title : ITK kinase domain with compound 1 (N-[1-(3-CYANOBENZYL)-1H-PYRAZOL-4-YL]-2H-INDAZOLE-3-CARBOXAMIDE)
Authors : Eigenbrot, C.; Shia, S.
Deposited on : 2014-02-26
Resolution : 2.58 Å(reported)

This is a full wwPDB validation report for a publicly released PDB entry.
We welcome your comments at validation@mail.wwpdb.org
A user guide is available at <http://wwpdb.org/ValidationPDFNotes.html>

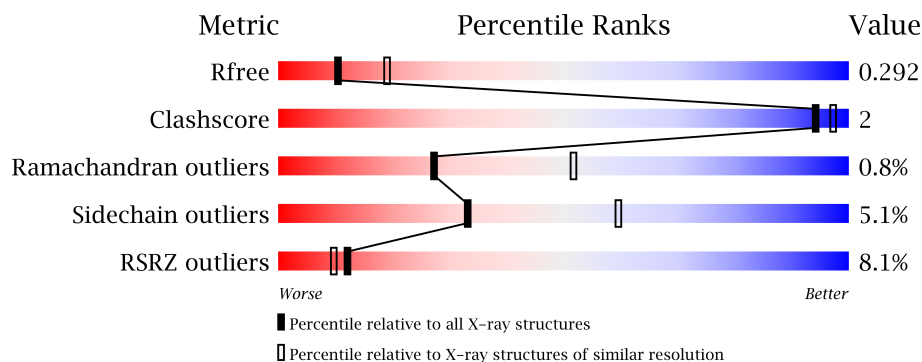
The following versions of software and data (see [references](#)) were used in the production of this report:

MolProbity : 4.02b-467
Mogul : 1.16 November 2013
Xtriage (Phenix) : dev-1439
EDS : stable23161
Percentile statistics : 21963
Refmac : 5.8.0049
CCP4 : 6.3.0 (Settle)
Ideal geometry (proteins) : Engh & Huber (2001)
Ideal geometry (DNA, RNA) : Parkinson et. al. (1996)
Validation Pipeline (wwPDB-VP) : stable23161

1 Overall quality at a glance

The reported resolution of this entry is 2.58 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}	66092	1891 (2.60-2.56)
Clashscore	79885	2358 (2.60-2.56)
Ramachandran outliers	78287	2316 (2.60-2.56)
Sidechain outliers	78261	2316 (2.60-2.56)
RSRZ outliers	66119	1891 (2.60-2.56)

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 ≥ 3 , 2, 1 and 0 types of geometric quality criteria. The upper red bar (where present) indicates the fraction of residues that have poor fit to the electron density.

Mol	Chain	Length	Quality of chain
1	A	266	
1	B	266	

2 Entry composition i

There are 4 unique types of molecules in this entry. The entry contains 4068 atoms, of which 0 are hydrogen and 0 are deuterium.

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.

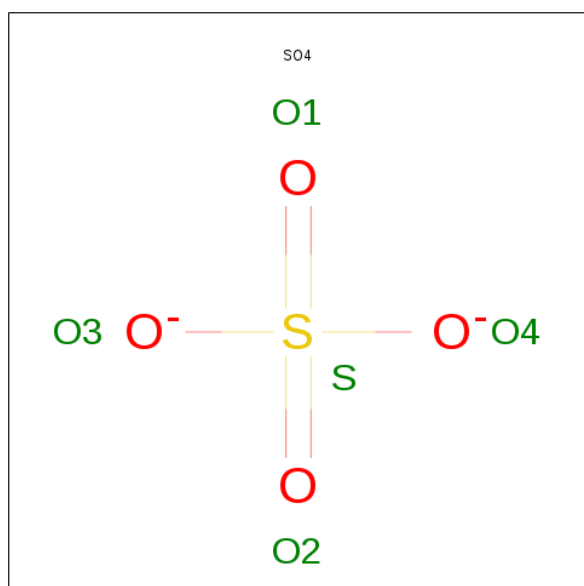
- Molecule 1 is a protein called Tyrosine-protein kinase ITK/TSK.

Mol	Chain	Residues	Atoms					ZeroOcc	AltConf	Trace
1	A	246	Total	C	N	O	S	0	0	0
			1962	1252	327	367	16			
1	B	247	Total	C	N	O	S	0	0	0
			1970	1257	328	368	17			

There are 2 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	356	SER	-	EXPRESSION TAG	UNP Q08881
B	356	SER	-	EXPRESSION TAG	UNP Q08881

- Molecule 2 is SULFATE ION (three-letter code: SO₄) (formula: O₄S).



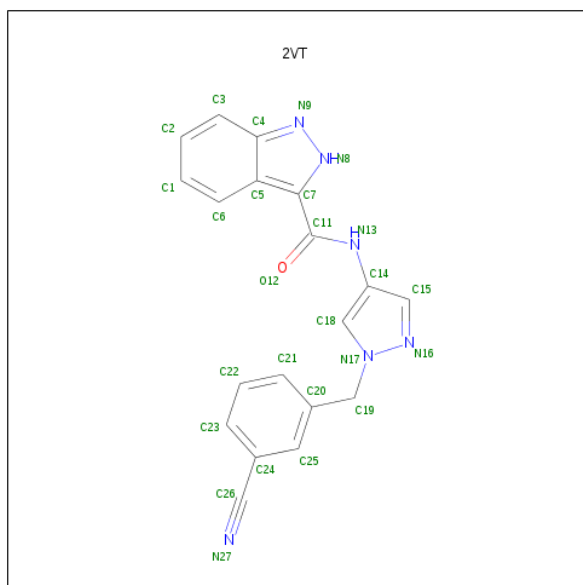
Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
2	A	1	Total	O	S	0	0
			5	4	1		

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Mol	Chain	Residues	Atoms			ZeroOcc	AltConf
2	A	1	Total	O	S	0	0
			5	4	1		

- Molecule 3 is N-[1-(3-CYANO BENZYL)-1H-PYRAZOL-4-YL]-2H-INDAZOLE-3-CARBOXAMIDE (three-letter code: 2VT) (formula: C₁₉H₁₄N₆O).



Mol	Chain	Residues	Atoms				ZeroOcc	AltConf
3	A	1	Total	C	N	O	0	0
			26	19	6	1		
3	B	1	Total	C	N	O	0	0
			26	19	6	1		

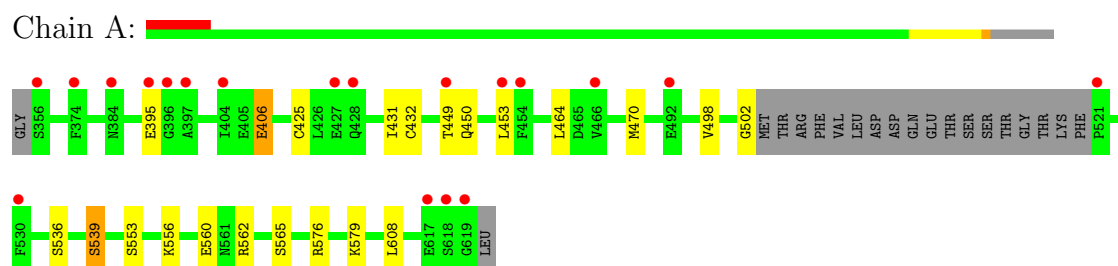
- Molecule 4 is water.

Mol	Chain	Residues	Atoms		ZeroOcc	AltConf
4	A	42	Total	O	0	0
			42	42		
4	B	32	Total	O	0	0
			32	32		

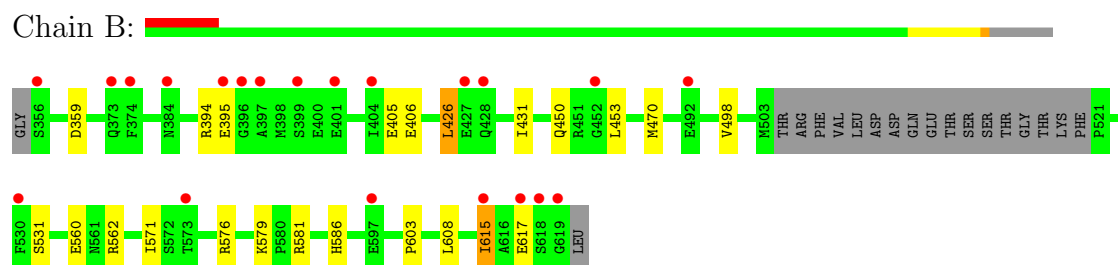
3 Residue-property plots

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: Tyrosine-protein kinase ITK/TSK



- Molecule 1: Tyrosine-protein kinase ITK/TSK



4 Data and refinement statistics

Property	Value	Source
Space group	P 1 21 1	Depositor
Cell constants a, b, c, α , β , γ	55.04Å 104.09Å 57.31Å 90.00° 110.21° 90.00°	Depositor
Resolution (Å)	25.34 – 2.58 25.07 – 2.58	Depositor EDS
% Data completeness (in resolution range)	94.0 (25.34-2.58) 94.3 (25.07-2.58)	Depositor EDS
R_{merge}	(Not available)	Depositor
R_{sym}	0.15	Depositor
$\langle I/\sigma(I) \rangle$ ¹	2.23 (at 2.57Å)	Xtriage
Refinement program	BUSTER 2.11.4	Depositor
R, R_{free}	0.223 , 0.284 0.230 , 0.292	Depositor DCC
R_{free} test set	919 reflections (5.39%)	DCC
Wilson B-factor (Å ²)	34.0	Xtriage
Anisotropy	0.866	Xtriage
Bulk solvent k_{sol} (e/Å ³), B_{sol} (Å ²)	0.34 , 23.1	EDS
Estimated twinning fraction	0.000 for l,-k,h	Xtriage
L-test for twinning	$\langle L \rangle = 0.51$, $\langle L^2 \rangle = 0.34$	Xtriage
Outliers	1 of 17975 reflections (0.006%)	Xtriage
F_o, F_c correlation	0.92	EDS
Total number of atoms	4068	wwPDB-VP
Average B, all atoms (Å ²)	44.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: *The largest off-origin peak in the Patterson function is 9.14% of the height of the origin peak. No significant pseudotranslation is detected.*

¹Intensities estimated from amplitudes.

5 Model quality

5.1 Standard geometry

Bond lengths and bond angles in the following residue types are not validated in this section: SO4, 2VT

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	0.41	0/2006	0.63	0/2708
1	B	0.41	0/2014	0.64	0/2718
All	All	0.41	0/4020	0.63	0/5426

There are no bond length outliers.

There are no bond angle outliers.

There are no chirality outliers.

There are no planarity outliers.

5.2 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 hydrogens added by MolProbity. The Clashes column lists the number of clashes within the asymmetric unit, and the number in parentheses is this value normalized per 1000 atoms of the molecule in the chain. The Symm-Clashes column gives symmetry related clashes, in the same way as for the Clashes column.

Mol	Chain	Non-H	H(model)	H(added)	Clashes	Symm-Clashes
1	A	1962	0	1918	7	0
1	B	1970	0	1927	5	0
2	A	10	0	0	0	0
3	A	26	0	0	0	0
3	B	26	0	0	0	0
4	A	42	0	0	0	0
4	B	32	0	0	0	0
All	All	4068	0	3845	12	0

Clashscore is defined as the number of clashes calculated for the entry per 1000 atoms (including

hydrogens) of the entry. The overall clashscore for this entry is 2.

All (12) close contacts within the same asymmetric unit are listed below.

Atom-1	Atom-2	Distance(Å)	Clash(Å)
1:B:426:LEU:HA	1:B:431:ILE:HG13	1.80	0.64
1:B:586:HIS:HB3	1:B:615:ILE:HG12	1.83	0.61
1:A:406:GLU:HG3	1:A:502:GLY:HA3	1.83	0.61
1:A:470:MET:HE3	1:A:498:VAL:HG11	1.84	0.59
1:A:449:THR:O	1:A:450:GLN:HB2	2.08	0.52
1:A:553:SER:HB3	1:A:556:LYS:HB2	1.94	0.50
1:A:536:SER:H	1:A:539:SER:HB2	1.78	0.48
1:B:603:PRO:HG2	1:B:608:LEU:HG	1.95	0.47
1:B:450:GLN:HA	1:B:453:LEU:HD12	1.98	0.45
1:B:470:MET:HE3	1:B:498:VAL:HG11	1.97	0.45
1:A:425:CYS:HB3	1:A:432:CYS:SG	2.60	0.41
1:A:450:GLN:HA	1:A:453:LEU:HD12	2.02	0.41

There are no symmetry-related clashes.

5.3 Torsion angles

5.3.1 Protein backbone ⓘ

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	242/266 (91%)	234 (97%)	7 (3%)	1 (0%)	43	70
1	B	243/266 (91%)	236 (97%)	4 (2%)	3 (1%)	19	38
All	All	485/532 (91%)	470 (97%)	11 (2%)	4 (1%)	27	52

All (4) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	B	395	GLU
1	B	560	GLU
1	A	560	GLU
1	B	394	ARG

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	215/233 (92%)	205 (95%)	10 (5%)	36	64
1	B	216/233 (93%)	204 (94%)	12 (6%)	30	54
All	All	431/466 (92%)	409 (95%)	22 (5%)	33	59

All (22) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	395	GLU
1	A	406	GLU
1	A	431	ILE
1	A	464	LEU
1	A	539	SER
1	A	562	ARG
1	A	565	SER
1	A	576	ARG
1	A	579	LYS
1	A	608	LEU
1	B	359	ASP
1	B	405	GLU
1	B	406	GLU
1	B	426	LEU
1	B	531	SER
1	B	562	ARG
1	B	571	ILE
1	B	576	ARG
1	B	579	LYS
1	B	581	ARG
1	B	615	ILE
1	B	617	GLU

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (3) such sidechains are listed below:

Mol	Chain	Res	Type
1	A	440	HIS
1	A	487	ASN

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Mol	Chain	Res	Type
1	B	589	GLN

5.3.3 RNA ⓘ

There are no RNA chains 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 ⓘ

4 ligands are modelled in this entry.

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	SO4	A	701	-	4,4,4	0.18	0	6,6,6	0.11	0
2	SO4	A	702	-	4,4,4	0.14	0	6,6,6	0.07	0
3	2VT	A	703	-	29,29,29	1.46	3 (10%)	38,40,40	3.43	8 (21%)
3	2VT	B	701	-	29,29,29	1.53	4 (13%)	38,40,40	3.26	6 (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
2	SO4	A	701	-	-	0/0/0/0	0/0/0/0

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Mol	Type	Chain	Res	Link	Chirals	Torsions	Rings
2	SO4	A	702	-	-	0/0/0/0	0/0/0/0
3	2VT	A	703	-	-	0/10/14/14	0/4/4/4
3	2VT	B	701	-	-	0/10/14/14	0/4/4/4

All (7) bond length outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(Å)	Ideal(Å)
3	B	701	2VT	N8-N9	-4.78	1.29	1.37
3	A	703	2VT	N8-N9	-4.75	1.29	1.37
3	B	701	2VT	C7-C5	3.99	1.48	1.43
3	A	703	2VT	C7-C5	3.76	1.47	1.43
3	B	701	2VT	N16-N17	3.09	1.40	1.35
3	A	703	2VT	N16-N17	2.95	1.39	1.35
3	B	701	2VT	C15-C14	2.22	1.41	1.38

All (14) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	Z	Observed(°)	Ideal(°)
3	A	703	2VT	C14-C15-N16	-17.75	109.28	112.68
3	B	701	2VT	C14-C15-N16	-16.76	109.47	112.68
3	A	703	2VT	C11-C7-N8	7.00	126.61	120.52
3	B	701	2VT	C11-C7-N8	6.73	126.38	120.52
3	A	703	2VT	C18-N17-N16	-4.54	107.68	111.57
3	B	701	2VT	C18-N17-N16	-4.38	107.82	111.57
3	A	703	2VT	C15-N16-N17	4.04	108.36	104.22
3	B	701	2VT	C15-N16-N17	3.92	108.23	104.22
3	A	703	2VT	C3-C4-N9	3.27	134.37	129.94
3	B	701	2VT	C3-C4-N9	3.27	134.37	129.94
3	A	703	2VT	C19-N17-N16	2.34	124.90	120.74
3	A	703	2VT	C20-C19-N17	-2.28	108.58	112.04
3	B	701	2VT	C19-N17-N16	2.17	124.60	120.74
3	A	703	2VT	C7-C5-C4	-2.07	104.37	106.24

There are no chirality outliers.

There are no torsion outliers.

There are no ring outliers.

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 ⓘ

6.1 Protein, DNA and RNA chains ⓘ

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	246/266 (92%)	0.62	19 (7%) 13 11	26, 41, 65, 105	0
1	B	247/266 (92%)	0.62	21 (8%) 11 9	27, 42, 67, 97	0
All	All	493/532 (92%)	0.62	40 (8%) 12 10	26, 42, 66, 105	0

All (40) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	617	GLU	5.5
1	A	427	GLU	5.0
1	B	396	GLY	4.3
1	B	404	ILE	4.2
1	B	427	GLU	4.2
1	B	428	GLN	4.0
1	A	428	GLN	3.9
1	A	492	GLU	3.9
1	A	384	ASN	3.9
1	B	617	GLU	3.8
1	B	397	ALA	3.7
1	B	573	THR	3.6
1	A	404	ILE	3.6
1	B	619	GLY	3.3
1	A	619	GLY	3.3
1	B	374	PHE	3.2
1	A	397	ALA	3.0
1	A	530	PHE	2.9
1	B	530	PHE	2.9
1	B	395	GLU	2.9
1	B	384	ASN	2.9
1	A	449	THR	2.8
1	B	597	GLU	2.8
1	A	454	PHE	2.7

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Mol	Chain	Res	Type	RSRZ
1	B	399	SER	2.7
1	B	492	GLU	2.6
1	A	466	VAL	2.6
1	A	374	PHE	2.5
1	A	356	SER	2.5
1	A	395	GLU	2.3
1	A	521	PRO	2.3
1	B	401	GLU	2.2
1	B	618	SER	2.2
1	B	615	ILE	2.2
1	A	396	GLY	2.2
1	B	452	GLY	2.1
1	A	618	SER	2.1
1	B	356	SER	2.1
1	B	373	GLN	2.1
1	A	453	LEU	2.0

6.2 Non-standard residues in protein, DNA, RNA chains ⓘ

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

6.3 Carbohydrates ⓘ

There are no carbohydrates in this entry.

6.4 Ligands ⓘ

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, 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	RSR	LLDF	B-factors(Å ²)	Q<0.9
3	2VT	A	703	26/26	0.15	-0.45	26,29,43,48	0
2	SO4	A	702	5/5	0.18	-0.46	107,107,108,108	0
2	SO4	A	701	5/5	0.17	-0.59	107,108,108,108	0
3	2VT	B	701	26/26	0.14	-0.72	21,26,42,45	0

6.5 Other polymers ⓘ

There are no such residues in this entry.