

# Full wwPDB X-ray Structure Validation Report (i)

#### Aug 7, 2020 – 05:05 PM BST

PDB ID : 4J3X

Title: Crystal structure of barley limit dextrinase (E510A mutant) in complex with

a branched maltoheptasaccharide

Authors: Sim, L.; Windahl, M.S.; Moeller, M.S.; Henriksen, A.

Deposited on : 2013-02-06

Resolution : 1.75 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
A user guide is available at

https://www.wwpdb.org/validation/2017/XrayValidationReportHelp with specific help available everywhere you see the (i) symbol.

The following versions of software and data (see references (1)) were used in the production of this report:

MolProbity : 4.02b-467

Mogul: 1.8.5 (274361), CSD as541be (2020)

Xtriage (Phenix) : 1.13 EDS : 2.13.1

Percentile statistics : 20191225.v01 (using entries in the PDB archive December 25th 2019)

Refmac: 5.8.0158

CCP4 : 7.0.044 (Gargrove) roteins) : Engh & Huber (2001)

Ideal geometry (proteins) : Engh & Huber (2001)
Ideal geometry (DNA, RNA) : Parkinson et al. (1996)

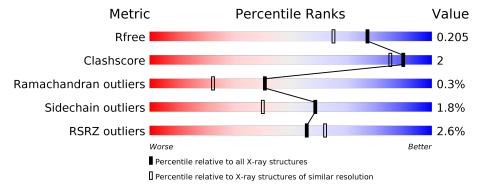
Validation Pipeline (wwPDB-VP) : 2.13.1

# 1 Overall quality at a glance (i)

The following experimental techniques were used to determine the structure: X- $RAY\ DIFFRACTION$ 

The reported resolution of this entry is 1.75 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	$\begin{array}{c} \text{Whole archive} \\ (\#\text{Entries}) \end{array}$	$\begin{array}{c} {\rm Similar \; resolution} \\ (\#{\rm Entries, \; resolution \; range(\AA)}) \end{array}$
$R_{free}$	130704	2340 (1.76-1.76)
Clashscore	141614	2466 (1.76-1.76)
Ramachandran outliers	138981	2437 (1.76-1.76)
Sidechain outliers	138945	2437 (1.76-1.76)
RSRZ outliers	127900	2298 (1.76-1.76)

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 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 <=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	905	92%			
2	В	7	71%	29%		

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
ſ	4	IOD	A	915	_	_	X	_



# 2 Entry composition (i)

There are 6 unique types of molecules in this entry. The entry contains 7931 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 Limit dextrinase.

Mol	Chain	Residues	${f Atoms}$			ZeroOcc	AltConf	Trace		
1	A	875	Total 6858	C 4350	N 1166	O 1310	S 32	0	20	0

There are 25 discrepancies between the modelled and reference sequences:

Chain	Residue	Modelled	Actual	Comment	Reference
A	-19	MET	-	expression tag	UNP Q9FYY0
A	-18	GLY	-	expression tag	UNP Q9FYY0
A	-17	SER	-	expression tag	UNP Q9FYY0
A	-16	SER	-	expression tag	UNP Q9FYY0
A	-15	HIS	-	expression tag	UNP Q9FYY0
A	-14	HIS	-	expression tag	UNP Q9FYY0
A	-13	HIS	-	expression tag	UNP Q9FYY0
A	-12	HIS	=	expression tag	UNP Q9FYY0
A	-11	HIS	-	expression tag	UNP Q9FYY0
A	-10	HIS	-	expression tag	UNP Q9FYY0
A	-9	SER	-	expression tag	UNP Q9FYY0
A	-8	SER	-	expression tag	UNP Q9FYY0
A	-7	GLY	-	expression tag	UNP Q9FYY0
A	-6	LEU	-	expression tag	UNP Q9FYY0
A	-5	VAL	_	expression tag	UNP Q9FYY0
A	-4	PRO	-	expression tag	UNP Q9FYY0
A	-3	ARG	-	expression tag	UNP Q9FYY0
A	-2	GLY	_	expression tag	UNP Q9FYY0
A	-1	SER	_	expression tag	UNP Q9FYY0
A	0	HIS	-	expression tag	UNP Q9FYY0
A	1	MET	-	expression tag	UNP Q9FYY0
A	82	ARG	LYS	SEE REMARK 999	UNP Q9FYY0
A	213	ALA	THR	SEE REMARK 999	UNP Q9FYY0
A	395	ARG	CYS	SEE REMARK 999	UNP Q9FYY0
A	510	ALA	GLU	engineered mutation	UNP Q9FYY0

• Molecule 2 is an oligosaccharide called alpha-D-glucopyranose-(1-6)-alpha-D-glucopyranose-



(1-4)-alpha-D-glucopyranose-(1-4)-alpha-D-glucopyranose-(1-6)-alpha-D-glucopyranose-(1-4)-alpha-D-glucopyranose.



Mol	Chain	Residues	At	oms		ZeroOcc	AltConf	Trace
2	В	7	Total 78	C 42	O 36	0	0	0

• Molecule 3 is CALCIUM ION (three-letter code: CA) (formula: Ca).

Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
3	A	1	Total Ca 1 1	0	0

• Molecule 4 is IODIDE ION (three-letter code: IOD) (formula: I).

Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
4	A	9	Total I 10 10	0	1

• Molecule 5 is CHLORIDE ION (three-letter code: CL) (formula: Cl).

Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
5	A	1	Total Cl 1 1	0	0

• Molecule 6 is water.

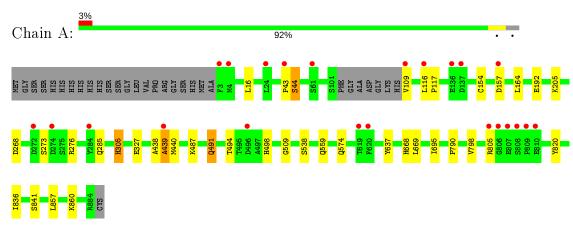
Mol	Chain	Residues	Atoms	ZeroOcc	AltConf
6	A	983	Total O 983 983	0	0



# 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: Limit dextrinase



 $\bullet \ \, Molecule \ 2: \ alpha-D-glucopyranose-(1-6)-alpha-D-glucopyranose-(1-4)-alph$ 





# 4 Data and refinement statistics (i)

Property	Value	Source
Space group	C 1 2 1	Depositor
Cell constants	173.27Å 86.20Å 61.56Å	Depositor
a, b, c, $\alpha$ , $\beta$ , $\gamma$	$90.00^{\circ}$ $96.03^{\circ}$ $90.00^{\circ}$	Depositor
Resolution (Å)	28.34 - 1.75	Depositor
resolution (11)	28.34 - 1.75	EDS
% Data completeness	97.4 (28.34-1.75)	Depositor
(in resolution range)	97.5 (28.34-1.75)	EDS
$R_{merge}$	0.08	Depositor
$R_{sym}$	(Not available)	Depositor
$< I/\sigma(I) > 1$	$1.82 \; ({\rm at} \; 1.75 {\rm \AA})$	Xtriage
Refinement program	REFMAC	Depositor
$R, R_{free}$	0.146 , $0.195$	Depositor
10, 10 free	0.158 , $0.205$	DCC
$R_{free}$ test set	4449  reflections  (5.00%)	wwPDB-VP
Wilson B-factor (Å <sup>2</sup> )	15.1	Xtriage
Anisotropy	0.038	Xtriage
Bulk solvent $k_{sol}(e/Å^3)$ , $B_{sol}(Å^2)$	0.37, 47.5	EDS
L-test for twinning <sup>2</sup>	$ < L >=0.50, < L^2>=0.33$	Xtriage
Estimated twinning fraction	No twinning to report.	Xtriage
$F_o, F_c$ correlation	0.96	EDS
Total number of atoms	7931	wwPDB-VP
Average B, all atoms $(Å^2)$	18.0	wwPDB-VP

Xtriage's analysis on translational NCS is as follows: The largest off-origin peak in the Patterson function is 4.64% of the height of the origin peak. No significant pseudotranslation is detected.

<sup>&</sup>lt;sup>2</sup>Theoretical values of <|L|>,  $< L^2>$  for acentric reflections are 0.5, 0.333 respectively for untwinned datasets, and 0.375, 0.2 for perfectly twinned datasets.



<sup>&</sup>lt;sup>1</sup>Intensities estimated from amplitudes.

# 5 Model quality (i)

### 5.1 Standard geometry (i)

Bond lengths and bond angles in the following residue types are not validated in this section: GLC, CA, IOD, CL

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		
1			RMSZ	# Z >5	RMSZ	# Z  > 5	
	1	A	0.33	0/7084	0.58	0/9637	

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 (i)

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	6858	0	6671	24	0
2	В	78	0	66	1	0
3	A	1	0	0	0	0
4	A	10	0	0	3	0
5	A	1	0	0	0	0
6	A	983	0	0	3	0
All	All	7931	0	6737	25	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 2.

All (25) close contacts within the same asymmetric unit are listed below, sorted by their clash magnitude.



Atom-1	Atom-2	Interatomic	Clash
		${f distance}({f A})$	overlap $( ext{Å})$
1:A:154[A]:CYS:SG	1:A:192:GLU:HG3	2.24	0.77
1:A:487:LYS:O	1:A:491[B]:GLN:HG2	1.92	0.69
1:A:154[A]:CYS:SG	1:A:192:GLU:CG	2.88	0.61
1:A:798:VAL:HG11	1:A:836:ILE:HD11	1.81	0.61
1:A:668:HIS:HD2	6:A:1699:HOH:O	1.86	0.58
1:A:154[A]:CYS:SG	1:A:192:GLU:OE1	2.66	0.54
1:A:43:PRO:O	1:A:44:SER:CB	2.57	0.52
1:A:327:GLU:HG2	4:A:915:IOD:I	2.80	0.51
1:A:157:ASP:HB2	1:A:164:LEU:HD11	1.95	0.49
1:A:494:THR:H	1:A:498:HIS:HD2	1.60	0.48
1:A:327:GLU:CG	4:A:915:IOD:I	3.33	0.47
1:A:439:ALA:O	1:A:440:MET:HG2	2.15	0.47
1:A:509:GLY:O	1:A:538:SER:HA	2.16	0.45
1:A:438[B]:ALA:O	1:A:439:ALA:CB	2.65	0.44
1:A:798:VAL:HG11	1:A:836:ILE:CD1	2.46	0.44
1:A:327:GLU:CD	4:A:915:IOD:I	3.26	0.44
1:A:116:LEU:N	1:A:117:PRO:CD	2.81	0.43
1:A:305:HIS:CD2	1:A:305:HIS:C	2.92	0.42
1:A:285:GLN:CA	6:A:1310:HOH:O	2.68	0.41
1:A:268:ASP:O	1:A:276:ARG:HD3	2.20	0.41
1:A:43:PRO:O	1:A:44:SER:HB3	2.20	0.41
1:A:860:LYS:HE2	6:A:1510:HOH:O	2.21	0.41
2:B:3:GLC:H61	2:B:4:GLC:H5	2.02	0.41
1:A:559:GLN:H	1:A:574:GLN:HE22	1.67	0.40
1:A:559:GLN:H	1:A:574:GLN:NE2	2.20	0.40

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	891/905 (98%)	869 (98%)	19 (2%)	3 (0%)	41 22



All (3) Ramachandran outliers are listed below:

Mol	Chain	Res	Type
1	A	439	ALA
1	A	44	SER
1	A	695	ILE

#### 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	Analysed Rotameric Outliers		Percentiles	
1	A	750/759 (99%)	735 (98%)	15 (2%)	55 34	

All (15) residues with a non-rotameric sidechain are listed below:

Mol	Chain	Res	Type
1	A	16[A]	LEU
1	A	16[B]	LEU
1	A	109	VAL
1	A	205	LYS
1	A	273	SER
1	A	305	HIS
1	A	491[A]	GLN
1	A	491[B]	GLN
1	A	637	TYR
1	A	669	LEU
1	A	790	PRO
1	A	805	ARG
1	A	820	TYR
1	A	841	SER
1	A	857	LEU

Some sidechains can be flipped to improve hydrogen bonding and reduce clashes. All (6) such sidechains are listed below:

$\mathbf{Mol}$	Chain	${f Res}$	$\mathbf{Type}$
1	A	142	HIS
1	A	498	HIS

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Mol	Chain	Res	Type
1	A	551	ASN
1	A	574	GLN
1	A	668	HIS
1	A	731	ASN

#### 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)

7 monosaccharides 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	Tuna	Chain	Res	Link	Вс	Bond lengths			Bond angles		
MIOI	Type	Chain	res	LIIIK	Counts	RMSZ	# Z  > 2	Counts	RMSZ	# Z  > 2	
2	GLC	В	1	2	12,12,12	0.76	0	17,17,17	0.60	0	
2	GLC	В	2	2	11,11,12	0.69	0	15,15,17	0.71	0	
2	GLC	В	3	2	11,11,12	0.64	0	15,15,17	0.99	1 (6%)	
2	GLC	В	4	2	11,11,12	1.02	0	15,15,17	1.25	3 (20%)	
2	GLC	В	5	2	11,11,12	0.45	0	15,15,17	0.66	0	
2	GLC	В	6	2	11,11,12	0.50	0	15,15,17	1.00	0	
2	GLC	В	7	2	11,11,12	0.53	0	15,15,17	0.88	0	

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	GLC	В	1	2	-	0/2/22/22	0/1/1/1
2	GLC	В	2	2	-	0/2/19/22	0/1/1/1
2	GLC	В	3	2	-	0/2/19/22	0/1/1/1
2	GLC	В	4	2	-	0/2/19/22	0/1/1/1
2	GLC	В	5	2	-	0/2/19/22	0/1/1/1
2	GLC	В	6	2	-	2/2/19/22	0/1/1/1
2	GLC	В	7	2	-	2/2/19/22	0/1/1/1

There are no bond length outliers.

All (4) bond angle outliers are listed below:

Mol	Chain	Res	Type	Atoms	$\mathbf{Z}$	$Observed(^o)$	$\operatorname{Ideal}({}^o)$
2	В	3	GLC	C1-O5-C5	2.79	115.97	112.19
2	В	4	GLC	C1-O5-C5	-2.22	109.18	112.19
2	В	4	GLC	O4-C4-C5	-2.11	104.05	109.30
2	В	4	GLC	O5-C1-C2	-2.06	107.59	110.77

There are no chirality outliers.

All (4) torsion outliers are listed below:

	$\mathbf{Mol}$	Chain	${f Res}$	Type	${f Atoms}$
ſ	2	В	7	GLC	O5-C5-C6-O6
	2	В	6	GLC	C4-C5-C6-O6
	2	В	6	GLC	O5-C5-C6-O6
	2	В	7	GLC	C4-C5-C6-O6

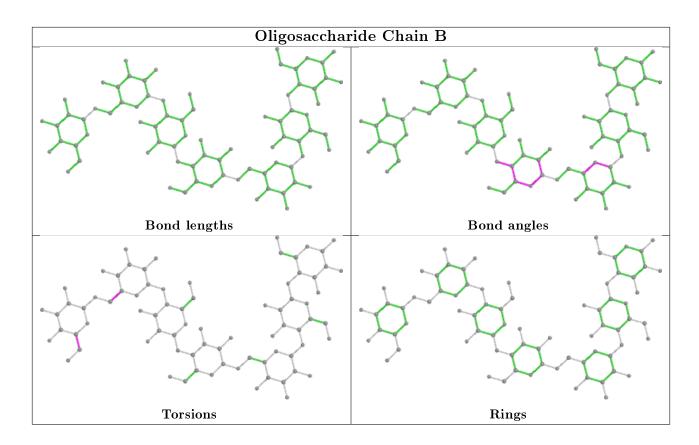
There are no ring outliers.

2 monomers are involved in 1 short contact:

Mol	Chain	Res	Type	Clashes	Symm-Clashes
2	В	3	GLC	1	0
2	В	4	GLC	1	0

The following is a two-dimensional graphical depiction of Mogul quality analysis of bond lengths, bond angles, torsion angles, and ring geometry for oligosaccharide.





### 5.6 Ligand geometry (i)

Of 12 ligands modelled in this entry, 12 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 (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,  $95^{th}$  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 $>$	$\#\mathrm{RSRZ}{>}2$	$OWAB(A^2)$	Q < 0.9
1	A	875/905 (96%)	0.01	23 (2%) 56 62	8, 15, 32, 70	0

All (23) RSRZ outliers are listed below:

Mol	Chain	Res	Type	RSRZ
1	A	808	SER	7.4
1	A	806	GLY	5.0
1	A	807	GLU	4.7
1	A	24	LEU	4.5
1	A	810	GLU	4.3
1	A	284	TYR	3.9
1	A	620	PHE	3.9
1	A	809	PRO	3.3
1	A	272	ASP	3.2
1	A	109	VAL	3.1
1	A	136	GLU	3.0
1	A	274	ASP	2.8
1	A	157	ASP	2.7
1	A	137	ASP	2.5
1	A	619	THR	2.5
1	A	805	ARG	2.5
1	A	61	SER	2.5
1	A	496	ASP	2.3
1	A	116	LEU	2.3
1	A	43	PRO	2.2
1	A	4	MET	2.2
1	A	439	ALA	2.2
1	A	3	PHE	2.0



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

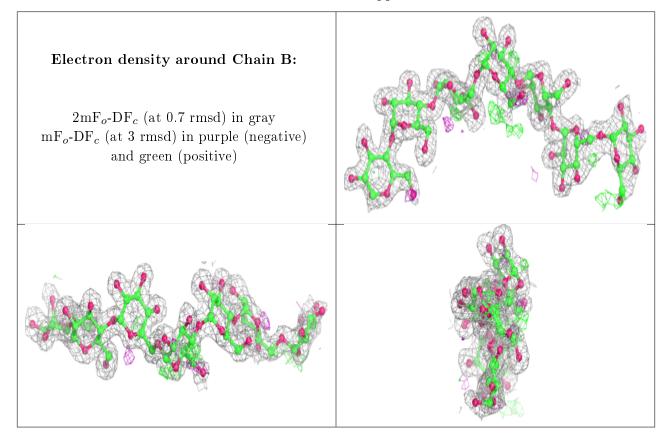
There are no non-standard protein/DNA/RNA residues in this entry.

### 6.3 Carbohydrates (i)

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,  $95^{th}$  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	$\mathbf{B} ext{-}\mathbf{factors}(\mathbf{\mathring{A}}^2)$	Q < 0.9
2	GLC	В	7	11/12	0.81	0.30	46,59,67,68	0
2	GLC	В	1	12/12	0.87	0.22	20,32,41,42	0
2	GLC	В	4	11/12	0.91	0.16	14,17,24,27	0
2	GLC	В	6	11/12	0.91	0.19	17,24,34,36	0
2	GLC	В	2	11/12	0.97	0.07	13,14,16,17	0
2	GLC	В	3	11/12	0.97	0.07	13,14,16,17	0
2	GLC	В	5	11/12	0.97	0.07	11,11,12,14	0

The following is a graphical depiction of the model fit to experimental electron density for oligosaccharide. Each fit is shown from different orientation to approximate a three-dimensional view.





### 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,  $95^{th}$  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	$\mathbf{B} ext{-}\mathbf{factors}(\mathbf{\mathring{A}}^2)$	Q < 0.9
4	IOD	A	913[B]	1/1	0.99	0.05	29,29,29,29	1
4	IOD	A	914	1/1	0.99	0.11	55,55,55,55	0
4	IOD	A	916	1/1	0.99	0.14	53,53,53,53	0
5	CL	A	918	1/1	0.99	0.04	32,32,32,32	0
4	IOD	A	913[A]	1/1	0.99	0.05	48,48,48,48	1
4	IOD	A	917	1/1	1.00	0.12	40,40,40,40	0
4	IOD	A	912	1/1	1.00	0.08	50,50,50,50	0
4	IOD	A	915	1/1	1.00	0.10	48,48,48,48	0
4	IOD	A	910	1/1	1.00	0.03	22,22,22,22	0
4	IOD	A	911	1/1	1.00	0.02	23,23,23,23	0
3	CA	A	908	1/1	1.00	0.05	10,10,10,10	0
4	IOD	A	909	1/1	1.00	0.03	20,20,20,20	0

## 6.5 Other polymers (i)

There are no such residues in this entry.

