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Data Article

High resolution crystal structure data of human plasma retinol-binding protein (RBP4) bound to retinol and fatty acids

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ABSTRACT

Retinol is transported in vertebrate plasma bound to a protein called retinol-binding protein (RBP4) so far believed to be specific for the vitamin. When the protein is saturated with retinol it binds tightly to another plasma protein, transthyretin while when not saturated with retinol it does not bind to TTR (Goodman, 1984). The X-ray structures of human RBP4, holo and devoid of retinol in its binding site are known to resolutions of 2.0 and 2.5 Å (Cowan et al., 1990; Zanotti et al., 1993) [2,3]. We have shown that RBP4 is not specific for retinol but it is also found in plasma, urine and amniotic fluid bound to fatty acids. Here we present 1.5 Å resolution crystal data on human plasma retinol-binding protein bound to retinol and fatty acids. These are the highest resolution data available in the Protein Data Bank for this protein.

For further details and experimental findings please refer to the article " Human plasma retinol-binding protein (RBP4) is also a fatty acid-binding protein" (Perduca et al., 2018) [4].

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Specifications Table

Subject area	*Biology*
More specific subject area	*Structural Biology*
Type of data	*Single crystal x-ray diffraction*
How data was acquired	*ESRF European Synchrotron radiation Facility*
Data format	*Standard, required by the PDB*
Experimental factors	*Structure Factors*
Experimental features	*Structures solved by molecular replacement*
Data source location	*Grenoble, France*
Data accessibility	*Available in the Protein Data Bank. Accession codes listed below*

Value of the data.

The extension of the resolution limit of RBP4 permitted the removal of several ambiguities in the models of the holo protein available at lower resolution [2,3]. More important was the identification of a heretofore unidentified ligand in the binding site of the protein believed to be apo. This second result changes substantially our perception of this protein that has so far been considered to be specific for retinol [1].

1. Data

The data of this article provides information on the X-ray crystallographic data sets of RBP4 purified from different sources. All these data are accessible at the Protein Data Bank. The table below summarizes origin, crystal resolution and PDB accession codes.

Source of RBP4 crystal data set	**Resolution**	***PDB Accession Code***
RBP4 not bound to retinol purified from plasma	2.0 Å	5NTY
RBP4 not bound to retinol purified from urine	1.5 Å	5NU2
RBP4 not bound to retinol purified from amniotic fluid	1.7 Å	5NU6
Plasma Holo RBP4 (retinol)	1.5 Å	5NU7
Urine RBP4 saturated with palmitate	1.6 Å	5NU8
Amniotic Fluid RBP4 saturated with palmitate	1.5 Å	5NU9
Urine RBP4 saturated with laurate	1.6 Å	5NUA
Amniotic Fluid RBP4 saturated with laurate	1.6 Å	5NUB

2. Experimental design, materials and methods

The data listed above were collected on two beamlines at the European Synchrotron Radiation Facility (ESRF) in Grenoble. The diffraction data were collected from crystals cooled to 100 K after brief immersion into a mixture of 80% mother liquor and 20% glycerol. The data were indexed, integrated and reduced using the programs MOSFLM [5] and Scala [6]. The processed data were converted to structure factors using the program TRUNCATE from the CCP4 suite [7]. More details on these data are summarized in Table 1 of the article “Human plasma retinol-binding protein (RBP4) is also a fatty acid-binding protein” [4].

In Table 1 we summarize the largest differences between the models of the same crystal form of RBP4 purified from plasma and solved to resolutions of 2.5 Å [3] and 1.5 Å [4] respectively (PDB

Table 1
Main differences in holo RBP4 in the 2.5 and 1.5 Å resolution models.

RBP4 residue	Atom	Δ-B (Å^3)	Δ−XYZ (Å)	1.5 Å electron density map of the side chain
Arg 10	CZ	33.9	4.43	Good
Glu 13	OE2	24.8	6.03	Poor
Glu 49	OE2	24.5	5.03	Good
Lys 58	CE	32.6	2.72	Absent
Leu 63	CD1	39.9	3.12	Poor
Leu 64	CD1	48.0	5.93	Absent
Asn 65	OD1	19.4	1.50	Absent
Asn 66	ND2	28.8	2.56	Absent
Trp 67	CH2	46.2	4.47	Poor
Asp 68	O	8.49	1.54	Good
Val 69	CG2	49.2	2.56	Poor
Cys 70	SG	19.7	2.33	Excellent
Asp 72	OD2	49.9	1.58	Excellent
Phe 77	CE2	1.85	2.46	Excellent
Glu 81	OE1	26.1	4.04	Absent
Phe 86	CE1	1.21	2.69	Excellent
Lys 87	CE	3.54	2.86	Good up to CD
Phe 96	CD2	10.7	2.45	Good
Gln 98	OE1	1.05	2.33	Excellent
Lys 99	CE	5.04	2.56	Absent
Asp 112	OD1	15.9	3.61	Excellent
Tyr 114	CD2	14.1	2.64	Excellent
Arg 121	NH2	2.9	2.53	Excellent
Leu 125	CD2	8.6	2.62	Excellent
Tyr 133	CE1	2.0	2.50	Excellent
Glu 147	OE2	47.0	6.85	Absent
Gln 149	OE1	6.68	4.11	Excellent
Lys 150	CE	30.4	3.10	Absent
Gln 154	OE1	10.7	3.09	Absent
Glu 157	OE2	41.1	2.59	Excellent
Arg 163	NH2	26.1	7.18	Good
Arg 166	NH2	40.6	7.95	Absent
Leu 167	CD1	4.9	2.65	Excellent
Tyr 173	CE2	6.9	3.85	Absent

accession codes 1BRP and 5NU7). The first column of the table identifies the amino acid and the second the atom where the largest difference was found while the last column gives a description of the electron density quality in the two maps. Third and fourth column record the differences in B factors and the distance between the two positions measured in Angstroms.

In a similar manner, Table 2 compares the models of the same crystal form of RBP4 purified from plasma, not bound to retinol and solved to a resolution of 2.5 Å [3] and 2.0 Å [4].

Table 3 analyses the differences between RBP4 purified from plasma and bound to palmitic acid and retinol.

The shortest distances between RBP4 residues and three ligands, palmitate (Table 4), laurate (Table 5) and retinol (Table 6) are listed in the three tables. The main interactions between RBP4 and retinol and palmitate are represented in Figs. 1 and 2 respectively.

Table 7 lists the ligand binding cavities of the two RBP4 populations analyzed, i.e bound to retinol and to fatty acids calculated from the coordinates of the models using the program CASTp [8].

Table 2
Main differences in non-fluorescent RBP4 in the 2.5 and 2.0 Å resolution models.

RBP4 residue	Atom	Δ-B (Å^3)	Δ-XYZ (Å)	2.0 Å electron density map of the side chain
Arg 10	CZ	39.1	4.60	Absent
Glu 13	OE1	48.6	4.85	Absent
Glu 44	OE1	71.9	3.02	Excellent
Phe 45	CE1	12.6	2.89	Excellent
Glu 49	OE2	31.7	4.61	Absent
Lys 58	NZ	39.9	3.00	Absent
Val 61	CG1	28.4	2.27	Good
Arg 62	NH1	29.3	2.71	Absent
Leu 64	CD2	9.8	6.25	Absent
Asn 65	OD1	19.6	2.21	Absent
Asn 66	O	23.6	2.63	Absent
Trp 67	CH2	4.5	4.58	Absent
Asp 68	OD2	30.6	1.70	Poor
Val 69	CG1	5.8	1.99	Poor
Cys 70	SG	32.1	2.08	Good
Phe 86	CE1	6.0	2.88	Excellent
Lys 87	CE	58.5	3.13	Good up to CD
Lys 99	NZ	12.2	3.67	Absent
Asp 112	OD2	5.4	2.05	Good
Arg 121	NH2	15.0	2.87	Excellent
Arg 139	NH1	29.4	2.67	Excellent
Glu 147	OE1	90.7	4.80	Absent
Lys 150	NZ	18.4	3.44	Absent
Ile 151	CD1	29.6	4.45	Excellent
Arg 153	NH1	6.0	2.48	Excellent
Gln 154	OE1	38.2	3.04	Absent
Arg 155	NH2	5.0	1.32	Good
Gln 156	NE2	14.2	2.66	Excellent
Glu 157	OE2	50.7	2.68	Good
Arg 163	NH2	16.5	6.64	Good
Gln 164	NE2	25.7	2.85	Excellent
Leu 167	CD2	15.6	2.39	Good
Tyr 173	CE2	16.3	3.83	Poor
Cys 174	O	3.3	1.46	Poor

Table 3
Main differences between non-fluorescent and holo RBP4 in the 2.0 and 1.5 Å resolution models (crystal forms 1 & 4 in Table 1 of reference [4]).

RBP4 residue	Atom	Δ-B (Å^3)	Δ-XYZ (Å)	Electron density map of the side chain, form 1	Electron density map of the side chain, form 4
Lys 29	NZ	14.5	4.56	Excellent	Excellent
Gly 34	O	3.1	1.91	Excellent	Excellent
Leu 35	CD2	10.4	9.53	Excellent	Good
Phe 36	CZ	9.1	9.18	Excellent	Excellent
Gln 52	OE1	26.8	2.34	Good	Good
Val 61	CG1	10.0	2.89	Good	Good
Tyr 114	CD1	8.7	2.39	Excellent	Excellent
Gln 149	OE1	5.76	4.48	Excellent	Excellent
Lys 150	NZ	14.6	2.20	Absent	Absent
Arg 153	NH2	2.0	1.68	Excellent	Excellent
Glu 157	OE2	31.2	3.4	Good	Excellent
Arg 166	NH2	31.4	8.95	Excellent	Absent
Val 169	CG1	16.8	2.44	Good	Good

Table 4
RBP4 residues in contact with palmitate in Crystal form 6.

RBP4 residue	Atom	Palmitate - Atom	Distance (Å)
Lys 29	NZ	O1	2.84
Pro 32	CD	O1	3.43
Leu 35	CA	O1	3.67
Phe 36	N	O1	2.88
Leu 37	N	O2	2.98
Arg 121	NH2	C2	3.82
Tyr 90	CE2	C5	3.82
Tyr 133	CE1	C3	3.71
Met 73	CB	C9	3.62
Val 74	C	CA	4.00
Gly 75	N	CA	3.83
Ala 55	CB	CD	3.91
Met 88	SD	CB	3.56
Ala 57	CB	CC	3.74
Phe 45	CZ	CD	3.62
His 104	CE1	CG	3.95
Phe 137	CZ	CE	3.97
HOH 264	O	O1	2.96

Only the shortest distance per residue has been included in the table. The residues in yellow are in contact with all the ligands

Table 5
RBP4 residues in contact with laurate in Crystal form 8.

RBP4 residue	Atom	Laurate - Atom	Distance (Å)
Lys 29	NZ	O2	2.91
Pro 32	CD	O2	3.45
Leu 35	CA	O2	3.78
Phe 36	N	O2	2.97
Leu 37	N	O1	2.95
Arg 121	NH2	C2	3.75
Tyr 90	CB	C8	3.59
Tyr 133	CE1	C3	3.86
Met 88	CE	C8	3.45
Ala 57	CB	C10	3.87
Phe 45	CZ	C12	3.93
HOH 264	O	O2	2.89

Only the shortest distance per residue has been included in the table. The residues in yellow are in contact with all the ligands

Table 6
RBP4 residues in contact with retinol in Crystal form 4.

RBP4 residue	Atom	Retinol - Atom	Distance (Å)
Leu 97	CA	O1	3.84
Gln 98	N	O1	2.99
Leu 35	O	C20	3.58
Phe 36	CE1	C19	3.59
Tyr 90	CB	C18	3.89
Phe 135	CE2	C17	3.82
His 104	CE1	C16	3.85
Val 61	CG1	C15	3.72
Met 73	CE	C12	3.65
Leu 37	CG	C11	3.98
Met 88	SD	C5	3.76
Ala 55	CB	C4	3.93
Ala 43	CB	C3	4.01
HOH 201	O	C16	4.09

Only the shortest distance per residue has been included in the table. The residues in yellow are in contact with all the ligands

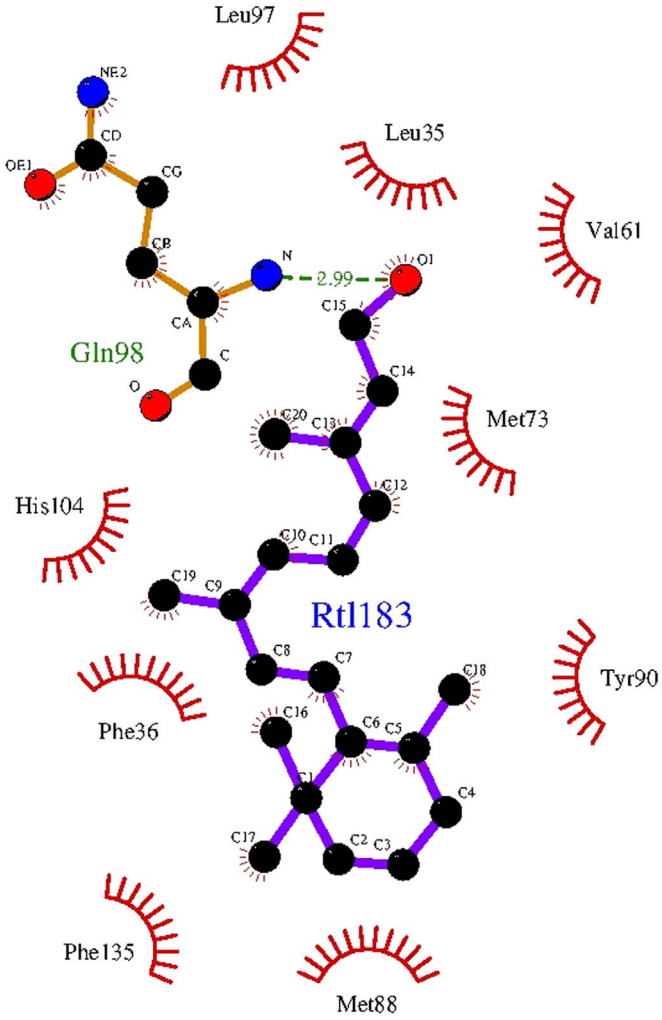

Fig. 1. Gln[98] and other side chains participating in the specific contacts of RBP4 with retinol. A hydrogen bond is indicated with green broken lines, whereas the amino acids that make hydrophobic contacts are only indicated but not represented as ball and stick models.

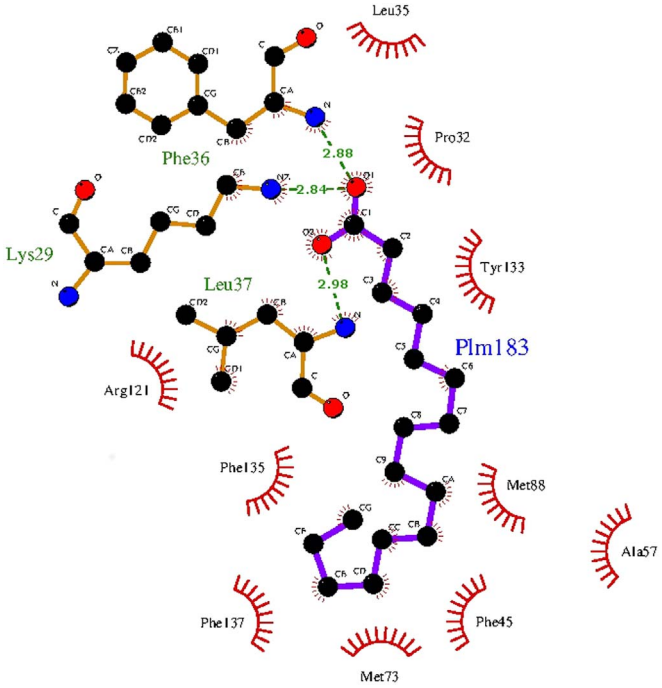

Fig. 2. Interaction of Lys[29], Phe[36] and Leu[37] and other side chains participating in the specific contacts of RBP4 with palmitic acid. Hydrogen bonds are indicated with green broken lines, whereas the amino acids that make hydrophobic contacts are only indicated but not represented as ball and stick models.

Table 7
Comparison of the ligand-binding cavity volumes.

Sample origin	Crystal form [4]	Ligand	Resolution	Cavity Volume (Å^3) CASTp
Plasma	**1**	**Palmitate**	**2.00 Å**	**696.7**
Urine	**2**	**Palmitate**	**1.50 Å**	**659.7**
Amniotic Fluid	**3**	**Palmitate**	**1.68 Å**	**682.2**
Plasma	**4**	**Retinol**	**1.50 Å**	**789.3**
Urine	**5**	**Palmitate**	**1.59 Å**	**662.8**
Amniotic Fluid	**6**	**Palmitate**	**1.50 Å**	**666.7**
Urine	**7**	**Laurate**	**1.60 Å**	**657.9**
Amniotic Fluid	**8**	**Laurate**	**1.60 Å**	**657.5**

The cavity volume computations were done with the program CASTp [8]

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Transparency document. Supplementary material

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2018.03.112.

References

[1] D.S. Goodman, Plasma retinol-binding protein, in: M.B. Sporn, A.B. Roberts, D.S. Goodman (Eds.), The Retinoids, 2, Academic Press, New York, 1984, pp. 41–88.
[2] S.W. Cowan, M.E. Newcomer, T.A. Jones, Crystallographic refinement of human serum retinol binding protein at 2 Å resolution, Proteins. 8 (1990) 44–61.
[3] G. Zanotti, S. Ottonello, R. Berni, H.L. Monaco, Crystal structure of the trigonal form of human plasma retinol-binding protein at 2.5 Å resolution, J. Mol. Biol. 230 (1993) 613–624.
[4] M. Perduca, S. Nicolis, B. Mannucci, M. Galliano, H.L. Monaco, Human plasma retinol-binding protein (RBP4) is also a fatty acid-binding protein, BBA - Molecular and Cell Biology of Lipids 1863 (2018) 458-466. published.
[5] A.G.W. Leslie, H.R. Powell, Processing diffraction data with Mosflm, Evol. methods Macromol. Crystallogr. 245 (2007) 41–51.
[6] P.R. Evans, Scaling and assessment of data quality, Acta Cryst. D62 (2006) 72–82.
[7] M.D. Winn, C.C. Ballard, K.D. Cowtan, E.J. Dodson, P. Emsley, P.R. Evans, R.M. Keegan, E.B. Krissinel, A.G. Leslie, A. McCoy, S. J. McNicholas, G.N. Murshudov, N.S. Pannu, E.A. Potterton, H.R. Powell, R.J. Read, A. Vagin, K.S. Wilson, Overview of the CCP4 suite and current developments, Acta Crystallogr. D Biol. Crystallogr. 67 (2011) 235–242.
[8] J. Dundas, Z. Ouyang, J. Tseng, A. Binkowski, Y. Turpaz, J. Liang, CASTp: computed atlas of surface topography of proteins with structural and topographical mapping of functionally annotated residues, Nucleic Acids Res. 34 (Web Server issue) (2006) W116–W118.