

Supporting Information for:

Solution NMR and computer simulation studies of active site loop motion in triosephosphate isomerase.

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Table 1: Backbone ^1H and ^{15}N resonance assignments of G3P bound TIM

Table 2: Conformational exchange contribution to transverse relaxation, R_{ex} , for G3P bound TIM at 298 K, 293 K, 288 K, and 283 K at $B_0 = 18.8$ T.

residue	^{15}N	$^1\text{H}^N$									
T4	125.1	9.11	G72	105.8	7.92	A136	119.2	7.20	G197	110.0	7.43
F5	131.2	8.69	A73	128.6	8.08	G137	108.4	8.00	D198	120.3	8.21
F6	130.8	7.46	F74	120.7	6.59	K138	117.5	8.10	K199	122.3	8.45
N10	123.9	9.45	G76	111.7	9.39	T139	116.1	7.56	A200	122.3	8.45
K12	116.6	8.06	E77	120.8	7.26	V142	120.6	8.23	A201	118.9	7.66
L13	127.0	7.93	N78	115.5	9.45	V143	115.2	7.95	S202	112.7	7.97
N14	117.5	7.59	S79	115.0	8.21	E144	119.9	8.21	E203	122.1	7.23
G15	106.5	8.34	V80	126.7	9.84	R145	121.7	7.58	L204	123.2	6.85
K17	124.2	10.14	D81	121.1	7.67	Q146	119.5	8.34	R205	126.6	8.85
Q18	118.3	8.30	Q82	122.1	6.93	N148	121.1	9.52	D213	119.8	8.88
K21	121.6	7.78	K84	118.7	7.60	A149	120.0	7.51	N213	120.4	9.39
E22	117.1	7.14	D85	122.1	7.77	V150	117.2	6.92	G214	105.2	9.73
I23	120.6	7.79	V86	106.0	7.21	L151	122.1	8.75	S215	116.2	8.38
N28	115.8	8.55	G87	106.9	7.67	D156	118.0	7.13	A217	129.9	8.37
T29	109.8	7.62	G94	106.5	9.21	F157	129.6	9.38	V218	117.6	8.51
A30	127.2	7.66	H95	119.0	7.51	T158	120.3	8.41	T219	113.2	7.75
S31	116.6	8.41	S100	111.3	8.26	N159	119.3	9.25	F220	118.8	7.84
I32	124.1	7.86	Y101	118.6	7.74	V167	129.0	8.38	K221	122.2	7.61
E34	119.0	8.56	F102	111.9	6.49	W168	114.1	7.01	A224	128.5	8.42
N35	116.2	8.19	H103	112.5	7.06	A169	125.5	7.15	D225	114.0	9.81
V36	116.0	6.90	E104	119.7	7.67	I170	121.3	7.04	V226	120.9	7.47
V38	124.1	8.15	D105	124.5	8.11	T172	108.2	6.80	D227	125.7	8.65
T45	111.2	7.50	D106	117.5	8.14	G173	112.0	8.36	L230	125.5	8.34
L47	126.6	7.40	K107	120.2	7.97	L174	125.8	7.72	V231	126.6	9.00
D48	120.3	9.05	F108	123.2	8.18	A175	122.9	7.62	S235	115.5	8.24
Y49	119.6	9.08	A110	127.9	8.65	A176	123.3	8.18	L236	118.4	6.55
L53	120.9	7.18	D111	120.1	8.86	T177	113.1	8.73	K237	118.1	7.08
V54	122.8	7.32	G118	108.5	7.66	E179	115.8	8.12	E239	112.7	7.93
K55	127.4	8.82	Q119	117.3	7.16	D180	122.8	8.01	F240	125.1	7.95
K56	122.6	6.87	G120	106.7	7.58	A181	121.7	8.50	V241	114.7	7.54
Q58	116.2	8.83	V121	118.3	7.20	Q182	117.1	8.04	D242	120.3	6.95
T60	119.8	8.85	G128	108.8	9.51	I184	122.3	8.14	I243	119.5	7.45
V61	121.3	8.72	E129	128.2	9.79	H185	122.1	9.63	I244	123.3	7.40
Q64	117.6	9.92	T130	113.0	9.52	A186	121.5	8.70	N245	114.9	7.31
A66	121.4	7.23	L131	122.7	9.51	A193	125.1	8.85	S246	115.2	7.52
Y67	115.2	8.04	E132	117.7	8.56	S194	115.2	7.43	R247	121.5	8.10
A70	124.1	9.18	E133	121.2	7.51	K195	119.8	6.80	N248	126.1	7.29
S71	111.6	7.65	K134	122.8	8.70	L196	116.8	8.67			

Table 1: Backbone ^1H and ^{15}N resonance assignments of G3P bound TIM at pH 5.7, 298 K, 10 mM $\text{CD}_3\text{CO}_2\text{Na}$. The assignments were obtained from the assignments of the free protein (BRMB code 7216) by following the chemical shifts changes upon titration of the ligand.

residue	R_{ex} (s^{-1})						
	T = 298 K		T = 293 K		T = 288 K		T = 283 K
4	1.1	± 0.6	-0.6	± 2.5	0.5	± 0.2	1.3 ± 3.2
5	-3.0	± 0.5	-9.9	± 2.2	-8.8	± 0.2	-5.8 ± 3.3
10	-2.2	± 0.5	-6.0	± 2.0	-5.6	± 0.2	-2.7 ± 3.3
12	nd		0.6	± 2.1	2.4	± 0.1	3.8 ± 2.9
13	-3.8	± 0.5	-6.8	± 1.5	-5.2	± 0.2	-4.8 ± 3.5
14	nd		0.4	± 2.0	2.6	± 0.4	3.8 ± 2.9
15	0.2	± 0.4	3.1	± 0.8	3.4	± 0.8	-0.3 ± 2.3
17	nd		-2.9	± 1.9	-2.6	± 0.7	-3.9 ± 4.1
18	nd		-0.9	± 2.6	-5.4	± 2.5	-1.1 ± 4.3
21	-0.7	± 0.5	-3.3	± 1.6	-3.1	± 0.6	-3.7 ± 3.8
23	0.0	± 0.2	3.6	± 2.9	2.3	± 0.9	-1.0 ± 3.8
28	3.0	± 0.6	0.7	± 1.9	2.8	± 0.4	3.2 ± 3.0
29	-0.7	± 0.4	-1.5	± 1.7	-0.3	± 0.9	0.6 ± 3.1
30	-3.2	± 0.3	-7.1	± 1.8	-7.1	± 0.9	-7.4 ± 3.6
32	0.0	± 0.4	-1.8	± 1.8	-1.1	± 0.5	-1.7 ± 2.4
34	0.8	± 0.3	-1.2	± 1.2	-2.8	± 0.1	-0.5 ± 3.0
35	0.0	± 0.2	-0.4	± 1.3	0.1	± 0.2	-3.6 ± 2.9
36	0.4	± 0.2	3.8	± 1.9	6.0	± 0.1	5.5 ± 2.1
38	0.4	± 0.3	-1.3	± 2.4	-0.2	± 0.6	-0.5 ± 2.7
45	1.4	± 0.4	1.6	± 1.4	1.9	± 1.8	-2.3 ± 2.8
47	-3.0	± 0.4	-6.0	± 1.5	-6.9	± 0.6	-7.2 ± 3.7
48	0.8	± 0.8	-1.4	± 1.3	-1.6	± 0.4	-2.6 ± 3.4
49	1.3	± 0.6	-1.4	± 1.9	-0.5	± 0.2	-0.3 ± 3.1
53	-0.6	± 0.7	4.7	± 2.6	6.6	± 1.9	9.3 ± 3.5
54	-0.9	± 0.6	-3.2	± 1.7	-2.1	± 0.4	-2.7 ± 3.5
55	-2.5	± 0.7	-6.4	± 2.2	-5.3	± 0.4	-3.9 ± 2.9
56	-0.8	± 0.3	-0.1	± 2.3	0.5	± 0.6	0.2 ± 2.1
58	1.6	± 0.2	1.0	± 2.3	1.8	± 0.4	3.8 ± 2.6
64	nd		-1.9	± 1.6	-1.4	± 0.9	2.4 ± 3.1
66	-0.2	± 0.2	2.1	± 2.3	1.0	± 0.4	0.3 ± 3.1
67	3.2	± 0.4	3.5	± 1.8	4.0	± 0.3	3.5 ± 2.9
70	-0.7	± 0.8	-1.4	± 0.7	-3.9	± 1.0	-2.4 ± 2.6
71	0.0	± 0.3	1.0	± 1.8	3.5	± 0.3	1.5 ± 2.8
72	-0.9	± 0.4	-1.1	± 1.6	1.2	± 0.9	-0.8 ± 2.5
73	-1.7	± 0.1	-3.4	± 0.7	-5.2	± 0.4	-6.6 ± 2.9
74	1.2	± 0.1	6.3	± 1.6	6.4	± 0.9	4.9 ± 3.0
76	-1.8	± 1.0	-0.1	± 3.0	1.1	± 2.9	0.5 ± 3.3

77	-0.8	\pm	0.3	-0.9	\pm	2.1	-0.3	\pm	0.3	-4.9	\pm	3.9
78	2.1	\pm	1.0	1.7	\pm	1.3	2.6	\pm	0.8	1.9	\pm	2.9
79	0.0	\pm	0.4	-0.7	\pm	1.1	-0.9	\pm	0.9	-3.7	\pm	3.0
80	nd			-6.8	\pm	1.6	-5.6	\pm	0.7	-5.5	\pm	3.3
81	0.9	\pm	0.5	4.0	\pm	1.3	0.7	\pm	1.5	1.7	\pm	3.1
82	-1.0	\pm	0.4	-0.1	\pm	2.0	-0.8	\pm	0.5	0.5	\pm	3.2
84	nd			8.4	\pm	2.2	13.9	\pm	0.2	20.4	\pm	3.1
85	0.1	\pm	0.4	-0.3	\pm	1.6	1.2	\pm	0.4	2.2	\pm	2.9
86	-0.4	\pm	0.6	4.6	\pm	3.2	3.1	\pm	2.3	3.2	\pm	3.7
87	-1.9	\pm	0.2	-3.4	\pm	2.1	-2.8	\pm	0.4	-4.4	\pm	2.8
94	-0.4	\pm	3.2	0.5	\pm	1.5	-1.6	\pm	1.2	-3.6	\pm	4.0
95	nd			1.4	\pm	2.3	2.0	\pm	1.2	4.8	\pm	3.8
100	-1.2	\pm	0.4	-0.4	\pm	2.0	-0.3	\pm	0.7	-1.3	\pm	2.9
101	nd			-2.0	\pm	2.2	-1.3	\pm	0.3	-0.5	\pm	3.3
102	1.6	\pm	0.3	4.9	\pm	1.1	6.5	\pm	2.2	2.5	\pm	3.7
103	nd			-3.3	\pm	6.2	-1.9	\pm	0.6	4.0	\pm	4.8
104	0.9	\pm	0.2	1.3	\pm	1.4	1.0	\pm	0.2	1.7	\pm	3.1
105	-1.0	\pm	0.3	-2.2	\pm	1.9	-1.8	\pm	0.3	-0.4	\pm	3.9
107	-0.5	\pm	0.5	-0.7	\pm	2.2	0.0	\pm	0.5	-0.8	\pm	2.9
110	-4.0	\pm	0.2	-5.0	\pm	2.9	-4.7	\pm	1.5	-6.1	\pm	4.2
118	-1.0	\pm	0.4	-7.0	\pm	2.1	-5.6	\pm	0.4	-2.7	\pm	2.9
120	-1.8	\pm	0.2	-2.3	\pm	2.3	-0.5	\pm	0.3	-4.1	\pm	2.8
121	nd			-0.8	\pm	2.0	1.1	\pm	0.2	2.4	\pm	3.2
128	3.0	\pm	1.7	-2.0	\pm	2.0	-0.7	\pm	0.8	6.0	\pm	3.1
129	-1.8	\pm	1.7	-5.4	\pm	2.0	-7.0	\pm	1.1	-2.9	\pm	3.8
130	-2.5	\pm	2.8	-2.6	\pm	2.2	-4.9	\pm	1.6	2.4	\pm	3.3
131	0.0	\pm	2.8	-0.2	\pm	2.1	-0.1	\pm	2.2	0.9	\pm	5.1
134	-3.1	\pm	0.6	-5.5	\pm	1.2	-5.8	\pm	0.8	-3.7	\pm	3.5
136	0.9	\pm	0.2	1.2	\pm	1.9	2.4	\pm	0.6	4.1	\pm	3.3
137	-4.1	\pm	0.3	-8.8	\pm	2.0	-8.0	\pm	0.6	-6.5	\pm	3.2
139	-1.2	\pm	0.4	-1.1	\pm	1.7	-0.7	\pm	0.9	-4.5	\pm	3.1
142	nd			-3.2	\pm	2.1	-0.8	\pm	0.4	-2.3	\pm	3.2
143	2.1	\pm	0.2	3.6	\pm	2.3	4.1	\pm	0.7	1.6	\pm	2.9
146	-2.1	\pm	0.2	-5.2	\pm	2.2	-3.3	\pm	0.5	-0.9	\pm	3.6
148	-0.1	\pm	1.3	-0.1	\pm	3.4	1.8	\pm	0.5	3.5	\pm	2.9
149	nd			-1.4	\pm	1.2	-0.6	\pm	0.4	-3.3	\pm	3.6
150	-1.1	\pm	0.9	3.0	\pm	1.8	3.1	\pm	0.6	5.8	\pm	3.4
151	-0.1	\pm	0.5	-3.2	\pm	1.5	-0.7	\pm	0.4	1.5	\pm	2.9

156	nd		0.7	±	1.5	0.2	±	0.5	4.4	±	3.7		
157	4.0	±	1.1		5.1	±	0.8	8.6	±	1.1	5.1	±	3.0
158	-0.8	±	0.2		-2.0	±	1.5	-2.5	±	0.5	-2.0	±	3.2
159	1.5	±	0.6		0.3	±	1.5	1.8	±	0.5	4.4	±	2.8
167	9.5	±	1.7		12.0	±	2.2	9.8	±	4.9	9.7	±	11.4
169	0.2	±	0.5		6.3	±	2.0	7.1	±	1.2	7.0	±	4.1
170	0.7	±	1.0		5.8	±	1.6	8.9	±	4.2	2.0	±	3.3
172	-1.3	±	0.2		-3.3	±	4.7	-1.6	±	6.0	-7.9	±	10.2
173	-1.3	±	1.8		5.1	±	6.4	-2.5	±	2.3	2.4	±	3.3
174	21.8	±	0.4		32.5	±	2.4	32.3	±	2.8	36.3	±	6.9
175	2.8	±	0.3		8.3	±	3.1	10.7	±	7.1	24.4	±	10.0
177	9.2	±	1.8		29.3	±	5.2	16.7	±	5.0	19.4	±	10.7
179	3.6	±	0.3		4.7	±	1.4	7.1	±	1.5	6.1	±	3.1
180	-0.8	±	0.3		-4.8	±	2.7	-2.7	±	0.2	-1.0	±	3.1
182	nd				-0.8	±	2.6	0.3	±	0.5	0.6	±	3.7
184	3.8	±	0.1		7.5	±	2.2	8.5	±	1.2	12.5	±	3.2
185	-1.6	±	1.5		-2.4	±	2.4	0.2	±	0.6	2.0	±	3.1
193	1.8	±	0.3		-1.5	±	2.2	-0.3	±	0.5	-1.6	±	3.1
194	0.3	±	0.4		2.1	±	2.0	1.8	±	0.8	0.7	±	2.8
195	0.5	±	0.7		2.5	±	1.5	2.7	±	0.2	1.3	±	2.9
196	-0.2	±	0.5		-1.0	±	2.1	-0.8	±	0.9	-2.4	±	2.8
197	0.9	±	0.3		2.8	±	1.6	5.2	±	0.5	2.5	±	2.7
201	-0.1	±	0.3		-1.9	±	2.5	-1.8	±	0.5	1.4	±	3.0
203	-0.6	±	0.6		0.0	±	1.6	-0.9	±	0.4	-0.3	±	3.1
204	-2.2	±	0.2		-1.2	±	2.2	-2.8	±	0.3	-3.0	±	3.3
205	1.0	±	0.6		-1.8	±	2.3	2.1	±	0.4	5.3	±	3.4
213	11.9	±	2.1		17.8	±	4.1	20.6	±	3.7	16.0	±	6.1
214	-1.4	±	2.6		-4.9	±	1.4	-6.3	±	0.4	1.3	±	3.1
217	1.8	±	0.4		2.3	±	2.0	2.7	±	1.2	1.5	±	3.7
219	-0.9	±	0.3		-2.5	±	2.2	-3.4	±	2.0	-2.7	±	3.9
221	9.7	±	0.5		21.8	±	1.4	39.2	±	1.0	46.8	±	6.2
224	0.6	±	0.5		-4.5	±	2.0	-2.7	±	1.0	-0.8	±	3.1
225	-1.0	±	3.1		-0.1	±	1.4	-1.8	±	4.1	2.6	±	4.6
226	nd				-2.7	±	1.9	-2.7	±	0.7	-4.7	±	3.6
227	-0.3	±	0.4		-1.5	±	2.4	-0.8	±	0.4	-2.0	±	3.0
230	-2.6	±	0.5		-1.7	±	1.5	-2.6	±	2.8	-4.1	±	5.4
231	1.0	±	1.1		-1.8	±	1.7	-4.7	±	0.3	-0.5	±	3.1
235	0.5	±	2.3		5.4	±	2.3	1.7	±	3.8	2.5	±	4.0

236	0.3	\pm	0.5	3.0	\pm	0.5	2.4	\pm	2.2	2.7	\pm	3.2
237	0.6	\pm	0.4	1.4	\pm	1.5	2.7	\pm	0.2	3.6	\pm	3.2
240	-0.2	\pm	0.4	1.4	\pm	1.3	0.9	\pm	1.2	-3.1	\pm	3.9
241	1.2	\pm	0.5	-1.1	\pm	1.4	1.3	\pm	0.9	1.5	\pm	3.0
242	0.5	\pm	0.5	1.3	\pm	2.0	3.2	\pm	1.5	-0.1	\pm	3.3
243	0.3	\pm	0.3	-0.9	\pm	2.4	1.3	\pm	0.7	1.1	\pm	2.9
244	-0.3	\pm	0.9	2.4	\pm	4.2	-1.0	\pm	0.9	-2.6	\pm	4.1
245	2.5	\pm	0.6	3.4	\pm	1.9	4.9	\pm	0.5	2.6	\pm	2.9
246	0.2	\pm	0.5	0.1	\pm	0.6	-0.7	\pm	1.8	-1.4	\pm	4.0
248	-1.8	\pm	0.1	-3.4	\pm	2.1	-3.7	\pm	0.4	-6.1	\pm	3.1

Table 2: Conformational exchange contribution to transverse relaxation, R_{ex} , for G3P bound TIM at 298 K, 293 K, 288 K, and 283 K at $B_0 = 18.8$ T. For nonexchanging residues, R_{ex} is distributed around zero due to variation in magnitude and orientation of the ^{15}N CSA tensor. nd indicates that data could not be determined.