Structure refinement using precession electron diffraction tomography and dynamical diffraction: tests on experimental data - supplementary material

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Table S1: Results of the test refinements of Ni_2Si with varying parameters. The first line contains the refinement with default parameters. In subsequent lines the results with one parameter different from the default is shown. Finally a few special refinement tests are summarized.

varied parameter	value	R1(obs)	wR1(all)	Nrefl(obs)	Nrefl(all)	ADRA	MDRA
all parameters default		7.14	7.56	849	1913	0.00826	0.01351
$R_{S_g}^{\max}$	0.30	5.16	5.61	330	722	0.01857	0.02109
$R_{S_g}^{\max}$	0.40	5.77	6.05	438	1008	0.01679	0.01849
$R_{S_g}^{\max}$	0.50	6.38	6.83	567	1290	0.00959	0.01268
$R_{S_g}^{\max}$	0.60	6.50	7.38	673	1545	0.00901	0.01419
$R_{S_g}^{\max}$	0.70	7.21	7.88	806	1815	0.01067	0.01375
$R_{S_g}^{\max}$	0.80	7.03	7.44	902	2010	0.00801	0.01373
$R_{S_g}^{\max}$	0.90	7.12	7.71	981	2150	0.00724	0.01016
$R_{S_g}^{\max}$	1.00	7.01	7.39	1033	2225	0.00795	0.01103
$R_{S_g}^{\max}$	1.25	7.43	7.95	1099	2359	0.00694	0.00829
$R_{S_g}^{\max}$	1.50	7.77	8.20	1134	2433	0.00595	0.00848
$S_g^{\max}$(refine)	0.005	3.22	3.76	211	433	0.01170	0.01507
$S_g^{\max}$(refine)	0.010	5.49	5.83	440	963	0.01200	0.01516
$S_g^{\max}$(refine)	0.015	6.33	6.72	650	1400	0.01018	0.01327
$S_g^{\max}$(refine)	0.020	6.88	7.22	779	1724	0.00858	0.01157
$S_g^{\max}$(refine)	∞	7.24	7.82	853	1941	0.00725	0.01114
$g_{\max}$(matrix)	1.5	7.18	7.60	852	1918	0.00897	0.01391
$g_{\max}$(matrix)	3.0	7.14	7.56	852	1917	0.00890	0.01363
$S_g^{\max}$(matrix)	0.0075	7.37	7.70	844	1912	0.00894	0.01611
$S_g^{\max}$(matrix)	0.0125	7.06	7.60	848	1898	0.00803	0.01162
$S_g^{\max}$(matrix)	0.015	6.85	7.26	839	1897	0.00915	0.01242
$S_g^{\max}$(matrix)	0.020	6.81	7.18	843	1908	0.01088	0.01661
$S_g^{\max}$(matrix)	0.025	6.81	7.22	850	1894	0.01088	0.01533
$S_g^{\max}$(matrix)	0.030	6.81	7.19	845	1890	0.01206	0.01594
N_{steps}	32	7.18	7.84	850	1919	0.01040	0.01593
N_{steps}	48	7.25	7.93	851	1925	0.00844	0.01377
N_{steps}	64	7.11	7.54	857	1925	0.00803	0.01377
N_{steps}	96	7.14	7.57	853	1920	0.00870	0.01440
N_{steps}	256	7.14	7.60	854	1920	0.00808	0.01178
kinematical refinement		11.07	11.53	88	118	0.02035	0.02843
kinematical anisotropic ADP		8.88	8.59	88	118	0.01670	0.02313
two-beam		7.89	8.76	865	1937	0.01860	0.02186
no orientation optimization		9.64	10.86	857	1942	0.01057	0.01471
anisotropic ADP		6.36	7.00	851	1912	0.00554	0.00840

Table S2: Fractional coordinates for the Ni_2Si structure obtained by kinematical and dynamical refinement with recommended parameters and from the reference structure. Only coordinates not fixed by symmetry are listed. Δ(dyn) and Δ(kin) represent the difference dynamical minus reference and kinematical minus reference, respectively.

Atom	coordinate	reference	kinematical	dynamical	Δ_{kin}	Δ_{dyn}
Ni1	x	0.17026(14)	0.1746(22)	0.16829(83)	0.0043(22)	−0.00196(68)
	z	0.060265(93)	0.06095(81)	0.06098(26)	0.00068(82)	0.00071(27)
Ni2	x	0.04204(14)	0.0415(23)	0.04132(83)	−0.0006(23)	−0.00072(68)
	z	−0.293894(98)	−0.29253(69)	−0.29359(24)	0.00137(70)	0.00031(26)
Si1	x	−0.28772(33)	−0.2889(40)	−0.2883(12)	−0.0012(41)	−0.00062(82)
	z	0.11441(21)	0.1105(11)	0.11343(37)	−0.0039(11)	−0.00098(42)

Table S3: Results of the test refinements of $PrVO_3$ with varying parameters. The first line contains the refinement with default parameters. In subsequent lines the results with one parameter different from the default is shown. Finally a few special refinement tests are summarized.

varied parameter	value	R1(obs)	wR1(all)	Nrefl(obs)	Nrefl(all)	ADRA	MDRA
all parameters default		8.82	9.70	1627	4717	0.01863	0.02600
$R_{S_g}^{\max}$	0.30	8.10	9.23	776	2583	0.01878	0.02020
$R_{S_g}^{\max}$	0.40	8.83	10.10	1047	3415	0.02125	0.03000
$R_{S_g}^{\max}$	0.50	8.70	9.85	1262	3937	0.02165	0.02780
$R_{S_g}^{\max}$	0.55	8.25	9.46	1392	4338	0.02350	0.02490
$R_{S_g}^{\max}$	0.60	8.98	9.92	1447	4437	0.01735	0.02250
$R_{S_g}^{\max}$	0.65	9.05	9.99	1515	4556	0.01900	0.02650
$R_{S_g}^{\max}$	0.70	8.93	9.88	1573	4633	0.02003	0.02720
$R_{S_g}^{\max}$	0.80	8.93	9.88	1668	4867	0.02378	0.03190
$R_{S_g}^{\max}$	0.85	9.26	10.05	1720	4871	0.02528	0.03360
$R_{S_g}^{\max}$	1.00	9.43	10.32	1750	4841	0.02980	0.03370
$S_g^{\max}$(refine)	0.005	6.46	7.47	434	1193	0.04230	0.06270
$S_g^{\max}$(refine)	0.010	8.03	8.70	925	2542	0.03893	0.05610
$S_g^{\max}$(refine)	0.015	8.58	9.28	1287	3576	0.02410	0.04180
$S_g^{\max}$(refine)	0.0175	8.65	9.33	1473	4221	0.01680	0.02590
$S_g^{\max}$(refine)	0.020	9.24	9.97	1579	4542	0.01845	0.02660
$S_g^{\max}$(matrix)	0.005	8.94	9.60	1648	4772	0.01473	0.02200
$S_g^{\max}$(matrix)	0.0075	8.77	9.62	1594	4601	0.02010	0.02560
$S_g^{\max}$(matrix)	0.0125	9.00	9.84	1650	4806	0.02440	0.03300
$S_g^{\max}$(matrix)	0.015	9.08	9.98	1640	4766	0.02743	0.03650
$S_g^{\max}$(matrix)	0.02	8.70	9.52	1637	4766	0.02603	0.03330
$S_g^{\max}$(matrix)	0.025	8.78	9.55	1640	4762	0.02535	0.03120
N_{steps}	32	12.72	13.44	1669	4872	0.03150	0.04360
N_{steps}	48	10.02	10.65	1671	4878	0.02250	0.02730
N_{steps}	64	9.19	10.10	1651	4829	0.01718	0.02220
N_{steps}	96	8.95	9.80	1625	4726	0.02353	0.03370
N_{steps}	256	8.81	9.72	1609	4662	0.01960	0.02790
kinematical refinement		24.04	31.92	197	369	0.14084	0.23940
two-beam refinement		16.03	17.7	1630	4631	0.11230	0.17380
no orientation optimization		11.20	12.60	1700	4961	0.04475	0.05200
anisotropic ADP		8.76	9.63	1627	4717	0.01850	0.02500

Table S4: Fractional coordinates for the $PrVO_3$ structure obtained by kinematical and dynamical refinement with recommended parameters and from the reference structure. Only coordinates not fixed by symmetry are listed. Δ(dyn) and Δ(kin) represent the difference dynamical minus reference and kinematical minus reference, respectively.

Atom	coordinate	reference	kinematical	dynamical	Δ_{kin}	Δ_{dyn}
Pr1	x	0.4552(5)	0.4444(13)	0.45595(24)	−0.0108(14)	0.00075(56)
	z	0.9928(7)	0.9834(15)	0.99210(25)	−0.0094(17)	−0.00071(74)
O1	x	0.7070(3)	0.7000(60)	0.70391(89)	−0.0070(60)	−0.00309(97)
	y	0.0429(2)	0.0428(46)	0.04135(70)	−0.0001(46)	−0.00155(73)
	z	0.7938(3)	0.8369(54)	0.79766(83)	0.0431(54)	0.00386(88)
O2	x	0.5191(3)	0.5165(69)	0.5189(12)	−0.0026(69)	−0.0002(12)
	z	0.4182(4)	0.3917(64)	0.4152(11)	−0.0265(64)	−0.0030(11)

Table S5: Results of the test refinements of kaolinite with varying parameters. The first line contains the refinement with default parameters. In subsequent lines the results with one parameter different from the default is shown. The number of refined parameters is 154. Finally a few special refinement tests are summarized.

varied parameter	value	R1(obs)	wR1(all)	Nrefl(obs)	Nrefl(all)	ADRA	MDRA
all parameters default		6.23	6.65	1930	2541	0.02138	0.04227
$R_{S_g}^{\max}$	0.30	4.20	4.34	727	975	0.02425	0.04834
$R_{S_g}^{\max}$	0.40	4.81	5.05	993	1331	0.02115	0.03953
$R_{S_g}^{\max}$	0.50	5.13	5.43	1248	1663	0.02242	0.05320
$R_{S_g}^{\max}$	0.60	5.65	5.95	1523	2021	0.02394	0.06041
$R_{S_g}^{\max}$	0.65	5.79	6.03	1662	2201	0.02160	0.04927
$R_{S_g}^{\max}$	0.70	6.06	6.43	1807	2385	0.02106	0.03800
$R_{S_g}^{\max}$	0.80	6.49	6.91	2059	2697	0.02285	0.04827
$R_{S_g}^{\max}$	0.85	6.95	7.52	2202	2870	0.02070	0.03346
$R_{S_g}^{\max}$	0.90	7.46	8.08	2332	3021	0.02228	0.04219
$R_{S_g}^{\max}$	1.00	8.72	9.48	2628	3393	0.02421	0.04419
$R_{S_g}^{\max}$	1.25	10.97	12.83	3206	4075	0.04564	0.07871
$R_{S_g}^{\max}$	1.50	13.11	14.81	3500	4428	0.05542	0.10720
$S_g^{\max}$(refine)	0.005	3.91	4.28	767	958	0.03182	0.06022
$S_g^{\max}$(refine)	0.010	5.46	5.98	1481	1871	0.02452	0.05247
$S_g^{\max}$(refine)	0.015	6.08	6.52	1870	2423	0.02245	0.05110
$g_{\max}$(matrix)	1.5	6.18	6.64	1932	2544	0.02205	0.04352
$g_{\max}$(matrix)	3.0	6.19	6.58	1937	2548	0.02182	0.04135
$S_g^{\max}$(matrix)	0.005	6.56	7.17	1936	2541	0.02486	0.06138
$S_g^{\max}$(matrix)	0.0075	6.25	6.66	1940	2547	0.02220	0.04484
$S_g^{\max}$(matrix)	0.0125	6.24	6.65	1942	2545	0.02253	0.05062
$S_g^{\max}$(matrix)	0.015	6.11	6.57	1933	2537	0.02186	0.05183
$S_g^{\max}$(matrix)	0.02	6.13	6.58	1941	2547	0.02257	0.05406
$S_g^{\max}$(matrix)	0.03	6.11	6.57	1954	2568	0.02091	0.05138
N_{steps}	32	6.72	7.19	1940	2541	0.02773	0.06190
N_{steps}	48	6.27	6.59	1941	2547	0.02256	0.05137
N_{steps}	64	6.22	6.66	1938	2548	0.02241	0.04246
N_{steps}	96	6.22	6.64	1935	2550	0.02198	0.04341
N_{steps}	256	6.21	6.63	1933	2543	0.02168	0.04248
kinematical refinement		19.16	20.20	934	1056	0.09610	0.26980
two-beam refinement		11.26	???	1921	2521	0.05679	0.15609
no orientation optimization		8.33	9.27	1970	2570	0.02432	0.05212

Table S6: Fractional coordinates for the kaolinite structure obtained by kinematical and dynamical refinement with recommended parameters and from the reference structure. Only coordinates not fixed by symmetry are listed. Δ(dyn) and Δ(kin) represent the difference dynamical minus reference and kinematical minus reference, respectively.

Atom	coordinate	reference	kinematical	dynamical	Δ_{kin}	Δ_{dyn}
Al1r	x	0.7014(4)	0.7066(19)	0.7033(6)	0.0052(20)	0.0019(7)
	y	0.5045(3)	0.5084(9)	0.5045(3)	0.0039(9)	0.0000(4)
	z	0.5245(3)	0.5353(20)	0.5243(8)	0.0108(21)	−0.0002(9)
Al2r	x	0.2063(4)	0.1983(22)	0.2027(6)	−0.0080(23)	−0.0036(7)
	y	0.6694(3)	0.6735(10)	0.6708(3)	0.0041(10)	0.0014(4)
	z	0.5256(3)	0.5176(24)	0.5262(8)	−0.0080(24)	0.0006(8)
Si1r	x	1.0032(4)	1.0091(18)	1.0032(6)	0.0059(18)	0.0000(7)
	y	0.6617(3)	0.6612(8)	0.6611(2)	−0.0005(8)	−0.0006(4)
	z	0.9076(3)	0.9076(20)	0.9075(7)	0.0000(20)	−0.0001(7)
Si2r	x	0.4892(4)	0.4810(18)	0.4906(5)	−0.0082(18)	0.0014(7)
	y	0.8332(3)	0.8332(8)	0.8328(3)	0.0000(9)	−0.0004(4)
	z	0.9062(3)	0.9003(19)	0.9090(7)	−0.0059(19)	0.0028(8)
O1r	x	0.9497(5)	0.9238(26)	0.9499(9)	−0.0259(26)	0.0002(10)
	y	0.6462(4)	0.6465(12)	0.6470(4)	0.0003(12)	0.0008(6)
	z	0.6839(3)	0.6480(29)	0.6847(12)	−0.0359(30)	0.0008(12)
O2r	x	0.8783(5)	0.8894(30)	0.8777(8)	0.0111(30)	−0.0006(10)
	y	0.3373(4)	0.3357(13)	0.3389(4)	−0.0016(14)	0.0016(6)
	z	0.6834(4)	0.7011(35)	0.6813(11)	0.0177(35)	−0.0021(12)
O3r	x	1.0000(0)	1.0013(27)	1.0040(8)	0.0013(27)	0.0040(8)
	y	0.5000(0)	0.4972(13)	0.4977(4)	−0.0028(13)	−0.0023(4)
	z	1.0000(0)	1.0030(31)	1.0028(10)	0.0030(31)	0.0028(10)
O4r	x	0.7897(5)	0.7844(27)	0.7851(8)	−0.0053(27)	−0.0046(9)
	y	0.7682(4)	0.7648(12)	0.7651(4)	−0.0034(13)	−0.0031(6)
	z	0.9756(3)	0.9796(30)	0.9699(11)	0.0040(30)	−0.0057(11)
O5r	x	0.7963(5)	0.7936(25)	0.7927(7)	−0.0027(26)	−0.0036(9)
	y	0.2361(4)	0.2326(12)	0.2354(4)	−0.0035(12)	−0.0007(6)
	z	0.9997(3)	1.0046(28)	0.9971(10)	0.0049(28)	−0.0026(10)
O6r	x	0.9496(5)	0.9647(27)	0.9518(8)	0.0151(27)	0.0022(10)
	y	0.0313(4)	0.0370(12)	0.0319(4)	0.0057(13)	0.0006(6)
	z	0.6747(4)	0.6850(30)	0.6745(12)	0.0103(31)	−0.0002(13)
O7r	x	1.0411(5)	1.0560(26)	1.0405(9)	0.0149(27)	−0.0006(10)
	y	0.8343(4)	0.8285(13)	0.8332(4)	−0.0058(13)	−0.0011(6)
	z	0.3957(4)	0.4004(29)	0.3947(12)	0.0047(30)	−0.0010(12)
O8r	x	0.9627(5)	0.9460(33)	0.9612(8)	−0.0167(33)	−0.0015(9)
	y	0.5268(4)	0.5309(15)	0.5280(4)	0.0041(15)	0.0012(5)
	z	0.3959(4)	0.3773(38)	0.3957(11)	−0.0186(38)	−0.0002(12)
O9r	x	0.9636(5)	0.9769(27)	0.9686(9)	0.0133(27)	0.0050(10)
	y	0.1436(4)	0.1432(12)	0.1460(4)	−0.0004(13)	0.0024(6)
	z	0.3920(4)	0.4051(29)	0.3972(11)	0.0131(29)	0.0052(12)

Table S7: Results of the test refinements of orthopyroxene with varying parameters from the data set with precession angle 1.2°. The first line contains the refinement with default parameters. In subsequent lines the results with one parameter different from the default is shown. Finally a few special refinement tests are summarized. OO stands for orientation optimization.

varied parameter	value	R1(obs)	wR1(all)	Nrefl(obs)	Nrefl(all)	occ[Fe1]	occ[Fe2]	ADRA	MDRA
all parameters default		6.25	7.05	3830	25524	0.184	0.436	0.01467	0.03398
$R_{S_g}^{\max}$	0.15	3.83	4.53	713	5629	0.143	0.419	0.01265	0.02030
$R_{S_g}^{\max}$	0.25	4.64	5.49	1176	9478	0.156	0.410	0.01315	0.02624
$R_{S_g}^{\max}$	0.30	4.88	5.80	1427	11422	0.175	0.386	0.01206	0.02570
$R_{S_g}^{\max}$	0.40	5.21	6.09	1925	13255	0.172	0.408	0.01178	0.02591
$R_{S_g}^{\max}$	0.50	5.74	6.53	2439	18746	0.184	0.429	0.01249	0.02673
$R_{S_g}^{\max}$	1.00	7.91	9.15	5136	28292	0.186	0.494	0.01477	0.03696
$R_{S_g}^{\max}$	1.25	11.30	14.52	5722	29510	0.185	0.521	0.01631	0.03927
$R_{S_g}^{\max}$	1.50	12.18	16.13	5857	30149	0.190	0.525	0.01651	0.04843
$S_g^{\max}$(refine)	0.005	5.03	5.69	1468	7033	0.177	0.454	0.02033	0.03670
$S_g^{\max}$(refine)	∞	6.26	7.06	3883	26139	0.183	0.435	0.01477	0.03533
$S_g^{\max}$(matrix)	0.02	6.09	6.88	3823	25499	0.169	0.439	0.01484	0.03228
N_{steps}	64	7.51	8.32	3827	25661	0.186	0.481	0.01438	0.03427
N_{steps}	96	6.55	7.41	3846	25696	0.179	0.455	0.01399	0.03050
N_{steps}	256	6.61	7.48	3848	25792	0.179	0.436	0.01381	0.03275
kinem. refinement		24.98	27.61	821	2558	0.516	0.401	0.04921	0.08143
two-beam refinement		17.72	20.73	3787	25296	0.394	0.437	0.04165	0.07110
no OO		7.07	8.19	3851	25476	0.189	0.459	0.01308	0.03396

Table S8: Fractional coordinates for the orthopyroxene structure obtained by kinematical and dynamical refinement with recommended parameters on the data set with precession angle $\varphi = 1.2°$ and from the reference structure. Δ(dyn) and Δ(kin) represent the difference dynamical minus reference and kinematical minus reference, respectively. OO stands for orientation optimization.

Atom	coordinate	reference	kinematical	dynamical	Δ_{kin}	Δ_{dyn}
Mg1/Fe1	x	0.375714(19)	0.37447(27)	0.375699(55)	−0.00124(27)	−0.000015(58)
	y	0.154314(41)	0.15297(50)	0.154420(91)	−0.00134(50)	0.00011(10)
	z	0.129852(69)	0.1206(18)	0.12797(40)	−0.0092(18)	−0.00188(40)
Mg2/Fe2	x	0.122172(15)	0.12302(23)	0.122245(50)	0.00085(23)	0.000073(52)
	y	0.015958(33)	0.01416(43)	0.016543(81)	−0.00180(43)	0.000585(87)
	z	0.136032(52)	0.1463(16)	0.13648(36)	0.0103(16)	0.00045(36)
Si1	x	0.026223(18)	0.02647(24)	0.025764(50)	0.00025(25)	−0.000459(53)
	y	0.336768(36)	0.34026(57)	0.33688(12)	0.00349(57)	0.00011(12)
	z	0.203511(69)	0.2116(18)	0.20487(35)	0.0081(18)	0.00136(36)
Si2	x	0.728330(18)	0.72909(24)	0.728134(51)	0.00076(24)	−0.000196(54)
	y	0.159169(37)	0.15780(53)	0.15861(11)	−0.00137(53)	−0.00056(11)
	z	0.050649(66)	0.0562(16)	0.05168(32)	0.0056(16)	0.00103(33)
O1	x	0.697236(49)	0.69788(34)	0.696760(91)	0.00064(34)	−0.00048(10)
	y	0.22751(11)	0.22669(71)	0.22664(17)	−0.00082(72)	−0.00087(20)
	z	0.32708(18)	0.3326(25)	0.32483(63)	0.0055(25)	−0.00226(66)
O2	x	0.066565(52)	0.0685(43)	0.066570(77)	0.00197(44)	0.000005(93)
	y	0.48422(10)	0.48593(93)	0.48387(16)	0.00171(93)	−0.00035(19)
	z	0.30703(18)	0.3001(31)	0.30716(59)	−0.0069(31)	0.00014(61)
O3	x	0.052554(50)	0.05352(36)	0.053209(98)	0.00096(36)	0.00066(11)
	y	0.30130(11)	0.30389(73)	0.30059(16)	0.00259(74)	−0.00071(20)
	z	−0.09790(18)	−0.0980(27)	−0.09882(67)	−0.0001(27)	−0.00091(70)
O4	x	0.816454(49)	0.81678(41)	0.816767(77)	0.00033(41)	0.000313(91)
	y	0.161067(97)	0.15469(87)	0.16164(16)	−0.00637(87)	0.00057(19)
	z	0.03970(18)	0.0460(27)	0.04310(52)	0.0063(27)	0.00341(55)
O5	x	−0.062428(50)	−0.06370(43)	−0.062651(74)	−0.00127(43)	−0.000223(89)
	y	0.338412(96)	0.34682(91)	0.33882(16)	0.00840(92)	0.00041(19)
	z	0.20256(18)	0.2069(29)	0.20329(55)	0.0044(29)	0.00074(58)
O6	x	0.688748(50)	0.68805(45)	0.688215(83)	−0.00070(45)	−0.000533(97)
	y	−0.00100(10)	−0.00220(92)	0.00006(15)	−0.00120(93)	0.00106(18)
	z	0.04932(19)	0.0485(30)	0.04918(53)	−0.0008(30)	−0.00014(56)

Table S9: Results of the test refinements of orthopyroxene with varying parameters from the data set with precession angle 2.0°. The first line contains the refinement with default parameters. In subsequent lines the results with one parameter different from the default is shown. Finally a few special refinement tests are summarized.

varied parameter	value	R1(obs)	wR1(all)	Nrefl(obs)	Nrefl(all)	occ[Fe1]	occ[Fe2]	ADRA	MDRA
all parameters default		6.82	7.76	4978	33727	0.179	0.446	0.01565	0.04222
$R_{S_g}^{\max}$	0.15	5.08	6.25	1100	11363	0.165	0.415	0.01339	0.02551
$R_{S_g}^{\max}$	0.25	5.60	6.77	1789	18710	0.167	0.443	0.01410	0.02779
$R_{S_g}^{\max}$	0.30	5.84	6.93	2105	22106	0.154	0.439	0.01602	0.03678
$R_{S_g}^{\max}$	0.40	6.16	7.21	2821	27788	0.173	0.440	0.01396	0.04592
$R_{S_g}^{\max}$	0.50	6.87	7.90	3572	30865	0.182	0.448	0.01507	0.04157
$R_{S_g}^{\max}$	1.00	7.54	8.83	5.833	34956	0.197	0.454	0.01661	0.04265
$R_{S_g}^{\max}$	1.50	9.58	12.51	6265	35953	0.185	0.433	0.01902	0.04432
$S_g^{\max}$(refine)	0.005	4.67	5.32	1367	8128	0.159	0.407	0.02273	0.04307
kinem. refinement		24.18	26.09	700	2586	0.513	0.389	0.04932	0.07817
two-beam refinement		17.72	20.73	3787	25296	0.394	0.437	0.04165	0.07110
no OO		7.67	8.75	5001	33989	0.177	0.445	0.01682	0.03329

Table S10: Fractional coordinates for the orthopyroxene structure obtained by kinematical and dynamical refinement with recommended parameters on the data set with precession angle $\varphi = 2.0°$ and from the reference structure. Δ(dyn) and Δ(kin) represent the difference dynamical minus reference and kinematical minus reference, respectively.

Atom	coordinate	reference	kinematical	dynamical	Δ_{kin}	Δ_{dyn}
Mg1/Fe1	x	0.375714(19)	0.37490(26)	0.375856(57)	−0.00082(26)	0.000142(60)
	y	0.154314(41)	0.15411(48)	0.154583(90)	−0.00020(48)	0.000269(99)
	z	0.129852(69)	0.1205(18)	0.12591(41)	−0.0093(18)	−0.00394(42)
Mg2/Fe2	x	0.122172(15)	0.12307(22)	0.122189(50)	0.00090(22)	0.000017(52)
	y	0.015958(33)	0.01465(40)	0.016782(80)	−0.00131(40)	0.000824(87)
	z	0.136032(52)	0.1458(15)	0.13638(37)	0.0098(15)	0.00035(37)
Si1	x	0.026223(18)	0.02652(23)	0.026067(51)	0.00029(23)	−0.000156(54)
	y	0.336768(36)	0.34022(53)	0.33727(11)	0.00345(53)	0.00051(12)
	z	0.203511(69)	0.2119(17)	0.20656(36)	0.0084(17)	0.00305(36)
Si2	x	0.728330(18)	0.72912(23)	0.728399(52)	0.00079(23)	0.000069(55)
	y	0.159169(37)	0.15818(50)	0.15813(10)	−0.00099(50)	−0.00104(11)
	z	0.050649(66)	0.0567(16)	0.05195(33)	0.0061(16)	0.00130(33)
O1	x	0.697236(49)	0.69798(32)	0.696770(90)	0.00074(33)	−0.00047(10)
	y	0.22751(11)	0.22753(67)	0.22715(17)	0.00002(67)	−0.00036(20)
	z	0.32708(18)	0.3319(23)	0.32503(68)	0.0048(23)	−0.00205(70)
O2	x	0.066565(52)	0.06900(40)	0.066827(78)	0.00244(40)	0.000262(94)
	y	0.48422(10)	0.48415(86)	0.48408(16)	−0.00007(87)	−0.00014(19)
	z	0.30703(18)	0.2947(27)	0.30654(62)	−0.0123(28)	−0.00049(64)
O3	x	0.052554(50)	0.05310(36)	0.05310(10)	0.00055(36)	0.00054(11)
	y	0.30130(11)	0.30338(72)	0.30004(16)	0.00208(73)	−0.00126(19)
	z	−0.09790(18)	−0.0948(26)	−0.09375(69)	0.0031(26)	0.00415(71)
O4	x	0.816454(49)	0.81664(38)	0.816220(79)	0.00018(38)	−0.000234(93)
	y	0.161067(97)	0.15451(82)	0.16130(15)	−0.00656(82)	0.00023(18)
	z	0.03970(18)	0.0460(25)	0.04179(55)	0.0063(25)	0.00209(58)
O5	x	−0.062428(50)	−0.06368(41)	−0.062978(75)	−0.00126(41)	−0.000550(90)
	y	0.338412(96)	0.34534(87)	0.33870(16)	0.00693(87)	0.00029(18)
	z	0.20256(18)	0.2006(28)	0.20216(57)	−0.0020(28)	−0.00039(60)
O6	x	0.688748(50)	0.68772(41)	0.688182(82)	−0.00103(41)	−0.000566(96)
	y	−0.00100(10)	0.00040(87)	−0.00023(15)	0.00140(88)	0.00078(18)
	z	0.04932(19)	0.0473(28)	0.05149(54)	−0.0020(28)	0.00217(57)

Table S11: Results of the test refinements of mayenite with varying parameters. The first line contains the refinement with default parameters. In subsequent lines the results with one parameter different from the default is shown. The number of refined parameters is 64. A few special refinement tests are summarized at the bottom of the table.

varied parameter	value	R1(obs)	wR1(all)	Nrefl(obs)	Nrefl(all)	ADRA	MDRA
all parameters default		9.94	12.34	2096	9104	0.01466	0.04398
$R_{S_g}^{\max}$	0.20	6.75	8.72	514	2691	0.01095	0.02787
$R_{S_g}^{\max}$	0.30	7.69	9.71	785	4080	0.01313	0.03326
$R_{S_g}^{\max}$	0.40	8.39	10.50	1067	5513	0.01038	0.02888
$R_{S_g}^{\max}$	0.50	8.78	11.02	1348	6893	0.01397	0.03920
$R_{S_g}^{\max}$	0.60	9.12	11.34	1612	8085	0.01536	0.04377
$R_{S_g}^{\max}$	0.70	9.68	12.15	1923	8874	0.01466	0.04209
$R_{S_g}^{\max}$	0.80	10.65	13.16	2246	9358	0.01656	0.04710
$R_{S_g}^{\max}$	0.90	11.11	14.10	2473	9673	0.01656	0.04004
$R_{S_g}^{\max}$	1.00	12.50	16.20	2590	9871	0.02262	0.03472
$R_{S_g}^{\max}$	1.50	12.84	12.42	2731	10490	002551	0.05003
$S_g^{\max}$(refine)	0.01	8.44	10.30	1156	4139	0.02687	0.06691
$S_g^{\max}$(refine)	0.02	9.83	12.20	1926	7711	0.01468	0.04522
$S_g^{\max}$(refine)	0.03	10.23	12.64	2132	10031	0.01416	0.04393
$S_g^{\max}$(refine)	0.04	10.39	12.97	2155	10416	0.01364	0.04134
$g_{\max}$(matrix)	1.5	10.16	12.52	2099	9108	0.01603	0.04557
$g_{\max}$(matrix)	3.0	10.01	12.44	2100	9121	0.01795	0.04978
$S_g^{\max}$(matrix)	0.005	10.01	12.37	2106	9111	0.01616	0.04687
$S_g^{\max}$(matrix)	0.0075	9.93	12.40	2094	9105	0.01661	0.04828
$S_g^{\max}$(matrix)	0.0125	10.08	12.45	2091	9104	0.01805	0.04793
$S_g^{\max}$(matrix)	0.015	10.03	12.44	2086	9104	0.01711	0.04711
$S_g^{\max}$(matrix)	0.02	10.11	12.67	2098	9100	0.01733	0.04735
N_{steps}	32	11.15	14.28	2075	9122	0.02138	0.03283
N_{steps}	64	10.34	12.95	2069	9101	0.01820	0.04253
N_{steps}	96	10.02	12.48	2093	9145	0.01853	0.04717
N_{steps}	144	10.13	12.51	2104	9133	0.01664	0.04861
N_{steps}	256	10.23	12.72	2104	9100	0.01669	0.04583
kinematical refinement		17.56	20.90	268	420	0.02698	0.03909
two-beam refinement		14.65	19.10	2151	9222	0.02524	0.03103
no orientation optimization		12.64	15.89	2151	9222	0.01589	0.04193
energy-filtered data		10.29	14.01	1861	9138	0.02410	0.04798

Table S12: Fractional coordinates for mayenite obtained by kinematical and dynamical refinement with recommended parameters and from the reference structure. Only coordinates not fixed by symmetry are listed. Δ(dyn) and Δ(kin) represent the difference dynamical minus reference and kinematical minus reference, respectively.

Atom	coordinate	reference	kinematical	dynamical	Δ_{kin}	Δ_{dyn}
Ca1	y	0.10680(30)	0.10481(71)	0.10687(12)	−0.00199(77)	0.00007(32)
Al1	x	−0.01880(13)	−0.02069(78)	−0.01881(13)	−0.00189(79)	−0.00001(18)
O1	x	0.28672(10)	0.28442(98)	0.28932(19)	−0.00230(99)	0.00260(22)
	y	0.09946(10)	0.09971(110)	0.09854(21)	0.00025(110)	−0.00092(23)
	z	0.19292(9)	0.19159(103)	0.19247(20)	−0.00133(104)	−0.00045(22)
O2	x	0.06481(9)	0.06419(119)	0.06415(20)	−0.00062(120)	−0.00066(22)