AM-84-238

American Mineralogist, Volume 69, pages 264–269, 1984

The crystal structure of high clinoferrosilite

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Abstract

The crystal structure of high clinoferrosilite ($FeSiO_3$) with $C2/c$ symmetry after the transition from orthoferrosilite at 1025°C was determined at 1050°C using a high-temperature four-circle diffractometer. The cell constants are a = 9.928(1), b = 9.179(1), c = 5.338(1)Å, β = 110.20°(1), V = 456.5(1)Å^3. The Fe2 polyhedron can be considered as six coordinated because the next two oxygens are too distant (3.191Å) for effective coordination with the Fe2 ion. The mean Si–O bond length is 1.634Å and the mean Fe–O bond lengths in the $M1$(6) and $M2$(6) polyhedra are 2.176 and 2.300Å, respectively. The polyhedral volume calculation reveals that the six-coordinated $M1$ polyhedron (13.40Å^3) is larger than the six-coordinated $M2$ polyhedron (11.93Å^3). This is related to the large distortion of the Fe2-polyhedron in the high clinoferrosilite structure. A similar tendency has been observed for the high clinopyroxene structures reported previously.

Introduction

The existence of a $C2/c$ polymorph in Ca-poor pyroxene was predicted by Morimoto and Tokonami (1969) and Smith (1969) as a non-quenchable, high-temperature phase of $P2_1/c$ clinopyroxene. The transition between these two phases was found to be a reversible and displacive transition using the high-temperature, single-crystal X-ray technique of Smyth (1969) and Prewitt et al. (1970). The crystal structure of the $C2/c$ high-temperature phase of Ca-poor pyroxenes (hereafter high clinopyroxene) has been determined by Brown et al. (1972) for the composition $En_{65}Fs_{30}Wo_5$ and by Smyth and Burnham (1972) for the composition $En_{31}Fs_{67.5}Wo_{1.5}$.

Sueno et al. (1976) carried out high temperature crystal structure refinements of orthoferrosilite ($FeSiO_3$) at several temperatures and afterward found a rapid transition from orthoferrosilite to high clinoferrosilite that was reversible and topotactic. The details were studied by Sueno and Kimata (1979). The behavior of the ortho-clino transition in ferrosilite is quite different from that previously reported on the natural intermediate orthopyroxenes which have non-reversible, sluggish, and non-topotactic characteristics (Smyth,1969; Smyth and Burnham, 1972; Smyth,1974). To elucidate the effect of the compositional difference on the behavior of the ortho-clino transition in pyroxenes, Sueno and Kimata (1981) carried out a high-temperature *in situ* X-ray study of the ortho to clino transition using synthetic orthopyroxenes with several different chemical compositions around the ferrosilite corner of the pyroxene quadrilateral. They found systematic differences between the different compositions, but a rapid, reversible, and topotactic transition was found only in pure ferrosilite. A high-temperature crystal-structure refinement of high clinoferrosilite is, therefore, important for providing the basic data for the analysis of the ortho-clinopyroxene transition mechanism.

The main intent of this study is to compare the structure of high clinoferrosilite with those of orthoferrosilite at several high temperatures and high clinopyroxenes with different chemical compositions such as high clinohypersthene (Smyth and Burnham, 1972) and high pigeonite (Brown et al., 1972).

Experimental

X-ray intensity data collection

A single crystal of orthoferrosilite, 0.10 × 0.08 × 0.06 mm in size, was selected for high-temperature diffraction intensity measurement from those synthesized hydrothermally at 800°C and 20 kbar for 16 days by Dr. D. H. Lindsley. The crystal was mounted parallel to the c axis on a Suprasil quartz glass fiber with a high-temperature cement composed of high-alumina wool and liquid binder. To prevent oxidation of Fe^{2+}, the crystal was sealed in an

0003–004X/84/0304–0264$02.00

79/05/19 1040C HI-CLINO FERROSILITE

Table 7-1 (Sueno, Kimata & Prewitt)

STRU

H	K	L	F(OBS)	F(CALC)	A(CALC)	B(CALC)	SIGMA	DELTA F	DEL/SIG	
5	11	1	4.04	17.53	17.531	0.000	15.02	-13.50	-0.898	*
3	11	1	16.68	22.50	22.500	0.000	7.85	-5.82	-0.741	
1	11	1	1.91	0.57	0.577	0.000	15.81	1.33	0.084	*
-1	11	1	25.54	16.44	16.437	0.000	5.43	9.10	1.677	*
-3	11	1	25.99	31.38	31.383	0.000	5.88	-5.39	-0.918	
-8	10	1	29.57	19.73	-19.725	0.000	5.54	9.85	1.776	*
-6	10	1	9.90	12.42	12.416	0.000	10.78	-2.52	-0.234	
0	10	1	20.80	22.92	22.921	0.000	5.98	-2.12	-0.355	
2	10	1	23.53	6.62	-6.617	0.000	5.44	16.91	3.107	*
4	10	1	19.26	6.76	6.756	0.000	6.51	12.50	1.919	*
5	9	1	57.48	53.66	-53.661	0.000	2.78	3.82	1.376	
1	9	1	34.09	31.01	-31.007	0.000	3.38	3.08	0.912	
-1	9	1	34.04	31.52	-31.516	0.000	3.35	2.53	0.754	
-5	9	1	49.70	51.53	-51.535	0.000	3.08	-1.84	-0.596	
-7	9	1	19.59	15.84	15.842	0.000	7.00	3.75	0.536	
-9	9	1	28.67	26.08	-26.076	0.000	5.66	2.59	0.458	
-10	8	1	5.66	2.96	-2.962	0.000	14.05	2.70	0.192	*
-8	8	1	23.07	26.60	-26.602	0.000	6.08	-3.53	-0.580	
-6	8	1	21.08	31.69	-31.690	0.000	5.24	-10.61	-2.024	*
-2	8	1	82.65	87.88	-87.879	0.000	1.94	-5.23	-2.691	
2	8	1	58.64	51.35	-51.351	0.000	2.03	7.29	3.598	
4	8	1	29.99	39.96	-39.959	0.000	3.68	-9.97	-2.706	*
6	8	1	42.30	40.17	-40.171	0.000	3.05	2.13	0.698	
8	8	1	29.07	26.34	-26.338	0.000	4.69	2.73	0.582	
7	7	1	28.70	2.08	2.080	0.000	4.07	26.62	6.542	*
5	7	1	31.73	11.29	-11.286	0.000	3.20	20.45	6.393	*
-1	7	1	15.05	5.97	-5.966	0.000	4.50	9.09	2.018	*
-7	7	1	16.11	1.85	1.850	0.000	6.11	14.26	2.333	*
-11	7	1	22.87	5.13	5.127	0.000	7.00	17.75	2.535	*
-10	6	1	9.29	4.58	-4.580	0.000	10.58	4.71	0.445	
-4	6	1	19.76	11.25	11.254	0.000	3.83	8.51	2.218	*
2	6	1	9.09	6.50	-6.498	0.000	5.91	2.59	0.439	
6	6	1	8.77	8.73	-8.729	0.000	8.04	0.05	0.006	
8	6	1	19.16	5.88	5.876	0.000	5.63	13.28	2.360	*
11	5	1	17.16	0.87	-0.869	0.000	7.50	16.29	2.172	*
7	5	1	35.38	32.40	-32.405	0.000	2.81	2.98	1.058	
5	5	1	24.80	15.72	-15.718	0.000	3.18	9.08	2.854	*
3	5	1	60.08	60.53	-60.530	0.000	1.51	-0.45	-0.299	
-1	5	1	23.31	30.09	-30.094	0.000	2.80	-6.79	-2.427	
-3	5	1	80.78	78.64	-78.638	0.000	1.57	2.14	1.359	
-5	5	1	22.20	18.77	-18.768	0.000	3.16	3.44	1.085	
-11	5	1	18.90	20.18	-20.185	0.000	7.35	-1.28	-0.174	
-12	4	1	20.48	19.59	19.591	0.000	7.23	0.89	0.123	
-2	4	1	64.95	68.03	68.026	0.000	1.31	-3.07	-2.353	
0	4	1	45.81	44.49	-44.493	0.000	1.14	1.32	1.160	
2	4	1	66.58	62.85	62.850	0.000	1.32	3.73	2.818	
6	4	1	26.77	5.27	-5.269	0.000	2.96	21.51	7.256	*
8	4	1	30.64	30.19	30.190	0.000	3.20	0.45	0.142	
10	4	1	15.94	10.05	-10.050	0.000	7.10	5.89	0.830	
11	3	1	33.17	37.60	37.595	0.000	4.10	-4.42	-1.078	
9	3	1	32.50	33.95	33.948	0.000	3.45	-1.45	-0.419	
7	3	1	18.49	25.55	25.553	0.000	4.14	-7.07	-1.709	
5	3	1	181.03	184.89	184.890	0.000	2.80	-3.86	-1.381	
3	3	1	75.70	73.82	73.819	0.000	1.36	1.88	1.389	
1	3	1	78.65	83.04	83.040	0.000	1.32	-4.39	-3.318	

79/05/19 1040C HI-CLINO FERROSILITE Table 7-2 (Sueno, Kimata & Prewitt) STRU

H	K	L	F(OBS)	F(CALC)	A(CALC)	B(CALC)	SIGMA	DELTA F	DEL/SIG	
-1	3	1	146.88	151.97	151.966	0.000	2.24	-5.08	-2.264	
-3	3	1	153.68	155.51	155.508	0.000	2.34	-1.82	-0.778	
-7	3	1	14.90	3.07	3.067	0.000	4.66	11.84	2.543	*
-11	3	1	34.83	42.52	42.519	0.000	4.34	-7.69	-1.773	
-12	2	1	38.38	41.39	41.391	0.000	4.29	-3.01	-0.703	
-8	2	1	56.80	41.83	41.827	0.000	1.89	14.97	7.912	*
-6	2	1	56.18	49.07	49.075	0.000	1.57	7.10	4.513	
-4	2	1	79.22	79.81	79.812	0.000	1.36	-0.59	-0.437	
-2	2	1	170.41	168.15	168.148	0.000	2.58	2.26	0.878	
0	2	1	23.71	21.49	-21.487	0.000	1.24	2.22	1.795	
2	2	1	107.12	112.40	112.396	0.000	1.67	-5.28	-3.153	
4	2	1	68.22	67.67	67.667	0.000	1.42	0.55	0.387	
6	2	1	41.18	47.52	47.521	0.000	1.86	-6.35	-3.414	
8	2	1	48.56	52.14	52.142	0.000	2.29	-3.58	-1.561	
7	1	1	43.74	32.64	-32.637	0.000	1.93	11.10	5.765	*
5	1	1	26.42	1.69	-1.691	0.000	2.03	24.73	12.207	*
3	1	1	56.66	58.87	-58.868	0.000	1.11	-2.20	-1.994	
-3	1	1	83.67	80.90	-80.901	0.000	1.37	2.77	2.019	
-7	1	1	26.93	24.21	-24.210	0.000	2.68	2.72	1.013	
-12	0	2	25.64	5.20	-5.198	0.000	5.51	20.44	3.710	*
-10	0	2	70.24	70.05	-70.054	0.000	2.13	0.19	0.089	
-8	0	2	52.28	54.88	-54.878	0.000	2.14	-2.60	-1.213	
-6	0	2	118.75	114.22	-114.222	0.000	1.96	4.53	2.313	
-4	0	2	158.49	171.36	-171.358	0.000	2.43	-12.87	-5.300	*
-2	0	2	30.19	30.86	-30.860	0.000	1.16	-0.67	-0.579	
0	0	2	197.19	209.46	-209.461	0.000	2.98	-12.27	-4.118	*
2	0	2	145.85	137.80	-137.800	0.000	2.26	8.05	3.560	*
4	0	2	118.47	121.37	-121.372	0.000	1.94	-2.90	-1.494	
6	0	2	63.71	64.51	-64.512	0.000	1.57	-0.80	-0.507	
8	0	2	21.75	18.97	-18.971	0.000	3.48	2.78	0.798	
10	0	2	72.30	70.78	-70.781	0.000	2.19	1.52	0.694	
11	1	2	13.76	1.42	1.420	0.000	7.64	12.34	1.617	*
9	1	2	17.11	21.47	-21.472	0.000	5.39	-4.36	-0.808	
7	1	2	56.65	54.05	-54.050	0.000	1.71	2.60	1.521	
5	1	2	38.06	36.79	36.786	0.000	1.66	1.27	0.769	
3	1	2	80.37	81.17	-81.166	0.000	1.44	-0.79	-0.549	
1	1	2	83.10	84.26	-84.259	0.000	1.39	-1.15	-0.831	
-1	1	2	46.60	47.07	-47.069	0.000	1.00	-0.47	-0.467	
-3	1	2	19.06	21.46	-21.463	0.000	1.56	-2.41	-1.546	
-7	1	2	103.01	101.84	-101.838	0.000	1.81	1.18	0.650	
-13	1	2	22.52	26.05	-26.052	0.000	6.73	-3.53	-0.525	
-8	2	2	46.25	35.31	35.311	0.000	2.11	10.94	5.184	*
-6	2	2	51.76	67.30	67.304	0.000	1.93	-15.55	-8.073	*
-2	2	2	23.74	25.70	25.702	0.000	1.27	-1.96	-1.539	
0	2	2	43.95	41.72	41.721	0.000	1.07	2.23	2.085	
2	2	2	83.57	83.75	83.748	0.000	1.42	-0.18	-0.123	
4	2	2	7.82	8.38	8.378	0.000	5.09	-0.56	-0.110	
6	2	2	13.16	5.55	5.547	0.000	4.05	7.61	1.879	
8	2	2	28.53	39.45	39.446	0.000	3.10	-10.91	-3.523	*
7	3	2	3.40	3.68	-3.676	0.000	9.66	-0.28	-0.029	*
5	3	2	5.78	13.23	13.229	0.000	7.03	-7.45	-1.060	*
3	3	2	10.63	12.29	12.294	0.000	4.39	-1.66	-0.379	
1	3	2	27.68	24.59	-24.591	0.000	1.41	3.09	2.195	
-1	3	2	10.87	2.43	2.430	0.000	2.71	8.44	3.110	*
-3	3	2	48.34	35.53	35.528	0.000	1.21	12.81	10.629	*

79/05/19 1040C HI-CLINO FERROSILITE Table 7-3 (Sueno, Kimata & Prewitt) STRUC

H	K	L	F(OBS)	F(CALC)	A(CALC)	B(CALC)	SIGMA	DELTA F	DEL/SIG	
-5	3	2	30.66	2.67	-2.669	0.000	2.16	27.99	12.958	*
-7	3	2	13.90	15.85	-15.854	0.000	4.92	-1.96	-0.397	
-8	4	2	14.47	0.94	0.936	0.000	6.01	13.53	2.251	*
-2	4	2	38.93	37.50	-37.499	0.000	1.57	1.43	0.910	
0	4	2	46.43	46.32	46.317	0.000	1.27	0.12	0.091	
2	4	2	23.26	22.60	-22.598	0.000	2.34	0.66	0.282	
6	4	2	9.49	5.03	5.028	0.000	6.60	4.47	0.677	
8	4	2	18.12	2.66	-2.661	0.000	4.94	15.46	3.129	*
9	5	2	16.21	14.83	14.832	0.000	6.71	1.38	0.205	
7	5	2	37.94	37.61	37.614	0.000	2.71	0.33	0.121	
5	5	2	20.26	12.43	12.435	0.000	3.53	7.83	2.215	
3	5	2	105.11	106.31	106.307	0.000	1.86	-1.20	-0.646	
1	5	2	46.08	45.18	45.180	0.000	1.52	0.90	0.592	
-1	5	2	45.78	58.07	58.068	0.000	1.71	-12.29	-7.194	*
-3	5	2	75.70	83.35	83.349	0.000	1.57	-7.65	-4.858	
-5	5	2	7.75	17.44	17.438	0.000	6.97	-9.69	-1.390	*
-7	5	2	74.80	77.97	77.968	0.000	1.93	-3.17	-1.646	
-11	5	2	15.62	23.40	23.404	0.000	7.32	-4.78	-0.654	
-12	6	2	33.31	3.36	3.360	0.000	4.97	29.95	6.021	*
-10	6	2	27.59	21.90	21.902	0.000	5.22	5.68	1.087	
-8	6	2	11.72	26.56	26.565	0.000	7.90	-14.84	-1.878	*
-6	6	2	66.69	80.34	80.343	0.000	2.08	-13.65	-6.574	*
-2	6	2	16.56	16.03	-16.034	0.000	3.92	0.53	0.134	
0	6	2	131.40	130.31	130.310	0.000	2.13	1.09	0.511	
2	6	2	69.73	67.74	67.742	0.000	1.76	1.98	1.128	
4	6	2	40.00	37.61	37.611	0.000	2.26	2.39	1.058	
6	6	2	36.82	33.76	33.761	0.000	2.73	3.06	1.121	
5	7	2	28.43	17.98	-17.978	0.000	3.37	10.45	3.106	*
3	7	2	8.64	21.91	21.910	0.000	7.94	-13.27	-1.672	*
1	7	2	21.62	27.30	-27.297	0.000	3.63	-5.68	-1.563	
-1	7	2	34.49	2.25	-2.246	0.000	2.55	32.25	12.670	*
-5	7	2	72.61	26.75	-26.747	0.000	1.99	45.86	23.014	*
-7	7	2	23.91	7.37	7.367	0.000	4.60	16.54	3.593	*
-11	7	2	29.72	11.68	11.678	0.000	5.31	18.04	3.399	*
-4	8	2	30.43	21.54	-21.537	0.000	3.55	8.89	2.504	*
-2	8	2	59.42	30.38	-30.376	0.000	2.23	20.04	9.000	*
0	8	2	25.97	7.53	-7.528	0.000	3.67	18.44	5.029	*
2	8	2	31.65	34.77	-34.767	0.000	3.28	-3.12	-0.951	
4	8	2	25.13	25.67	-25.674	0.000	4.17	-0.54	-0.130	
6	8	2	12.37	3.36	-3.359	0.000	7.82	9.02	1.153	*
8	8	2	13.63	15.24	-15.239	0.000	8.34	-1.61	-0.193	
7	9	2	28.47	15.25	-15.247	0.000	4.67	13.22	2.830	*
3	9	2	26.56	3.52	-3.517	0.000	4.32	23.04	5.333	*
-3	9	2	20.48	7.57	-7.567	0.000	4.97	12.91	2.596	*
-7	9	2	22.59	4.18	-4.175	0.000	5.99	18.41	3.072	*
-9	9	2	14.43	4.75	4.746	0.000	9.24	9.69	1.048	*
-2	10	2	10.47	9.89	-9.891	0.000	9.49	0.57	0.061	
0	10	2	25.62	21.64	-21.643	0.000	5.06	3.98	0.786	
2	10	2	25.52	7.03	7.029	0.000	4.87	18.49	3.795	*
4	10	2	5.56	20.33	-20.332	0.000	12.22	-14.77	-1.209	*
3	11	2	41.86	38.17	-38.166	0.000	3.65	3.70	1.012	
1	11	2	26.22	22.67	-22.672	0.000	5.16	3.55	0.688	
-3	11	2	15.81	17.55	-17.554	0.000	8.10	-1.75	-0.215	
-2	12	2	26.56	9.85	9.854	0.000	5.96	16.70	2.802	*
0	12	2	35.78	23.40	-23.395	0.000	4.55	12.39	2.720	*

H	K	L	F(OBS)	F(CALC)	A(CALC)	B(CALC)	SIGMA	DELTA F	DEL/SIG	
-10	0	6	30.91	40.29	-40.293	0.000	3.68	-9.38	-2.547	*
-8	0	6	24.88	29.84	-29.840	0.000	3.21	-4.96	-1.542	
-6	0	6	16.19	10.37	-10.373	0.000	3.63	5.82	1.602	
-4	0	6	73.04	72.06	-72.060	0.000	1.67	0.98	0.586	
-2	0	6	32.57	30.47	-30.474	0.000	2.13	2.09	0.985	
0	0	6	51.49	47.94	-47.941	0.000	1.83	3.55	1.944	
2	0	6	11.74	20.38	-20.383	0.000	6.01	-8.64	-1.438	*
3	1	6	35.15	36.05	-36.052	0.000	2.53	-0.90	-0.358	
1	1	6	19.01	4.69	4.690	0.000	3.72	14.32	3.851	*
-1	1	6	17.10	17.14	-17.140	0.000	4.02	-0.04	-0.011	
-3	1	6	28.53	22.96	-22.963	0.000	2.36	5.57	2.359	
-7	1	6	26.62	28.79	-28.788	0.000	2.85	-2.16	-0.760	
-11	1	6	31.68	20.86	-20.858	0.000	4.02	10.82	2.693	*
-10	2	6	10.85	0.33	0.334	0.000	7.85	10.52	1.339	*
-8	2	6	3.94	1.29	1.290	0.000	9.68	2.65	0.273	*
-6	2	6	16.61	15.61	15.606	0.000	3.65	1.00	0.275	
-4	2	6	2.29	2.24	2.242	0.000	9.56	0.05	0.005	*
-2	2	6	24.06	16.83	18.828	0.000	2.85	5.23	1.839	
0	2	6	18.75	3.46	3.462	0.000	3.58	15.29	4.268	*
2	2	6	14.70	15.60	15.604	0.000	4.81	-0.90	-0.188	
-3	3	6	20.21	2.90	2.901	0.000	3.01	17.31	5.743	*
-5	3	6	3.15	4.43	-4.429	0.000	8.98	-1.28	-0.143	*
-7	3	6	18.65	3.32	3.323	0.000	3.80	15.33	4.033	*
-9	3	6	13.47	7.70	7.705	0.000	4.81	10.76	2.240	*
-10	4	6	33.77	2.52	-2.522	0.000	3.60	31.25	8.681	*
-8	4	6	10.92	0.90	-0.899	0.000	7.28	10.02	1.375	*
-4	4	6	20.41	19.09	19.094	0.000	3.30	1.32	0.400	
-2	4	6	24.50	18.40	-18.404	0.000	2.81	6.09	2.166	
0	4	6	15.27	3.93	3.934	0.000	4.30	11.34	2.634	*
2	4	6	11.25	3.33	3.329	0.000	5.63	7.92	1.408	
1	5	6	13.46	1.45	-1.452	0.000	4.81	12.01	2.499	*
-1	5	6	28.30	34.85	34.855	0.000	2.93	-6.56	-2.237	
-3	5	6	27.68	31.31	31.308	0.000	2.86	-3.63	-1.267	
-7	5	6	33.09	29.74	29.744	0.000	2.93	3.34	1.141	
-6	6	6	13.23	9.13	9.129	0.000	5.51	4.10	0.744	
-4	6	6	49.85	49.27	49.272	0.000	2.08	0.58	0.278	
-2	6	6	12.66	6.11	6.109	0.000	5.16	6.55	1.270	
0	6	6	25.49	26.80	26.798	0.000	3.32	-1.31	-0.396	
-5	7	6	20.73	13.65	-13.647	0.000	3.97	7.08	1.785	
1	11	3	13.40	8.68	-8.685	0.000	8.56	4.71	0.551	
-1	11	3	27.91	15.08	-15.076	0.000	4.79	12.84	2.681	*
-3	11	3	14.97	16.51	-16.509	0.000	7.99	-1.54	-0.193	
-5	11	3	7.10	1.50	1.497	0.000	12.71	5.60	0.441	
-4	10	3	16.58	12.60	-12.597	0.000	7.27	3.98	0.548	
0	10	3	26.56	23.60	-23.595	0.000	4.66	2.96	0.636	
5	9	3	40.61	43.68	43.677	0.000	3.38	-3.07	-0.908	
1	9	3	23.29	29.79	29.789	0.000	4.77	-6.50	-1.361	
-1	9	3	35.73	38.78	38.779	0.000	3.25	-3.05	-0.938	
-3	9	3	25.84	15.84	15.837	0.000	4.47	10.00	2.237	*
-5	9	3	22.79	23.60	23.600	0.000	5.58	-0.81	-0.145	
-8	8	3	29.52	19.92	19.918	0.000	4.74	9.60	2.026	*
-6	8	3	26.71	25.10	25.097	0.000	4.74	1.61	0.340	
-4	8	3	15.96	18.97	18.970	0.000	5.58	-3.01	-0.540	
-2	8	3	67.95	67.94	67.943	0.000	1.99	0.01	0.004	
2	8	3	39.45	44.70	44.698	0.000	2.68	-5.25	-1.959	

/05/19 1040C HI-CLINO FERROSILITE Table 7-5 (Sueno, Kimata & Prewitt) STRUCTU

H	K	L	F(OBS)	F(CALC)	A(CALC)	B(CALC)	SIGMA	DELTA F	DEL/SIG	
4	8	3	29.47	19.27	19.268	0.000	3.73	7.21	1.930	
5	7	3	30.31	24.17	24.167	0.000	3.15	6.14	1.951	
3	7	3	15.20	7.92	-7.924	0.000	4.84	7.28	1.504	
1	7	3	24.77	13.94	13.944	0.000	3.00	10.82	3.610	*
-1	7	3	23.17	5.34	5.340	0.000	3.25	17.33	5.336	*
-3	7	3	21.12	5.85	5.852	0.000	3.80	15.26	4.016	*
-5	7	3	22.64	25.70	25.701	0.000	4.29	-3.06	-0.714	
-11	7	3	29.35	3.32	-3.323	0.000	5.46	26.03	4.769	*
-10	6	3	16.79	1.06	1.060	0.000	7.32	15.74	2.150	*
-8	6	3	14.25	2.51	-2.511	0.000	6.82	11.74	1.722	*
-6	6	3	4.27	18.94	18.943	0.000	11.29	-14.67	-1.300	*
-4	6	3	7.90	5.18	5.183	0.000	7.10	2.72	0.383	
0	6	3	6.40	1.24	1.244	0.000	6.65	5.15	0.775	*
2	6	3	21.37	15.85	15.846	0.000	2.96	5.52	1.862	
6	6	3	22.49	0.91	0.905	0.000	3.90	21.58	5.532	*
8	6	3	18.57	5.28	5.283	0.000	5.51	13.29	2.412	*
7	5	3	22.22	23.21	23.206	0.000	4.14	-0.99	-0.238	
5	5	3	11.97	14.16	14.160	0.000	5.07	-2.19	-0.431	
3	5	3	34.44	29.21	29.209	0.000	1.98	5.23	2.649	
1	5	3	11.20	10.69	10.687	0.000	4.50	0.62	0.137	
-1	5	3	50.94	50.85	50.845	0.000	1.57	0.09	0.059	
-3	5	3	40.86	42.93	42.930	0.000	1.83	-2.07	-1.135	
-5	5	3	26.17	11.37	-11.372	0.000	2.90	14.80	5.109	*
-7	5	3	41.09	35.45	35.448	0.000	2.65	5.64	2.133	
-11	5	3	21.82	4.81	4.805	0.000	6.31	17.01	2.695	*
-12	4	3	19.86	14.29	-14.292	0.000	7.10	5.57	0.784	
-4	4	3	8.20	18.43	18.431	0.000	5.64	-10.23	-1.812	*
-2	4	3	72.94	71.02	-71.022	0.000	1.44	1.92	1.332	
0	4	3	13.93	20.76	20.757	0.000	3.72	-6.83	-1.836	
2	4	3	18.60	16.11	-16.113	0.000	2.98	2.49	0.835	
4	4	3	20.51	16.90	-16.900	0.000	2.71	3.61	1.331	
8	4	3	26.09	16.54	-16.543	0.000	3.85	9.55	2.479	*
9	3	3	30.91	31.43	-31.431	0.000	3.21	-0.52	-0.162	
7	3	3	42.33	38.48	-38.476	0.000	2.13	3.85	1.813	
5	3	3	97.35	96.14	-96.138	0.000	1.79	1.22	0.678	
3	3	3	12.69	11.28	-11.280	0.000	4.00	1.41	0.353	
1	3	3	110.48	109.20	-109.202	0.000	1.83	1.28	0.701	
-1	3	3	123.73	124.00	-124.000	0.000	1.98	-0.27	-0.138	
-3	3	3	83.94	85.55	-85.554	0.000	1.52	-1.61	-1.059	
-5	3	3	93.35	93.54	-93.537	0.000	1.67	-0.19	-0.111	
-7	3	3	32.59	12.07	-12.065	0.000	2.61	20.52	7.855	*
-9	3	3	84.06	84.41	-84.413	0.000	1.99	-0.35	-0.178	
-8	2	3	43.97	46.32	-46.318	0.000	2.36	-2.35	-0.994	
-6	2	3	29.93	37.43	-37.426	0.000	2.36	-8.49	-3.596	*
-4	2	3	25.55	18.22	-18.224	0.000	1.93	7.33	3.806	
-2	2	3	139.48	141.18	-141.179	0.000	2.16	-2.70	-1.250	
0	2	3	27.28	21.50	-21.503	0.000	1.74	5.77	3.316	
2	2	3	52.66	51.74	-51.739	0.000	1.51	0.92	0.613	
4	2	3	37.04	37.63	-37.635	0.000	1.83	-0.60	-0.326	
6	2	3	29.11	29.61	-29.613	0.000	2.29	-1.50	-0.653	
8	2	3	57.28	41.17	-41.167	0.000	1.94	16.12	8.298	*
9	1	3	3.03	3.33	3.325	0.000	11.22	-0.29	-0.026	*
7	1	3	13.06	21.00	20.999	0.000	4.89	-7.94	-1.624	
5	1	3	21.53	5.08	-5.077	0.000	2.46	16.46	6.686	*
3	1	3	35.72	34.48	34.481	0.000	1.74	1.24	0.709	

H	K	L	F(OBS)	F(CALC)	A(CALC)	B(CALC)	SIGMA	DELTA F	DEL/SIG	
1	1	3	16.48	4.31	4.306	0.000	2.90	12.17	4.202	*
-1	1	3	36.08	38.39	38.387	0.000	1.44	-2.30	-1.598	
-3	1	3	29.59	30.79	30.782	0.000	1.47	-1.19	-0.810	
-7	1	3	34.93	34.37	34.366	0.000	2.23	0.56	0.253	
-1	9	5	22.62	9.49	-9.487	0.000	4.25	13.14	3.088	*
-3	9	5	6.06	7.39	-7.390	0.000	10.48	-1.33	-0.127	*
-5	9	5	25.50	26.19	-26.189	0.000	4.79	-0.69	-0.143	
-6	8	5	21.40	24.46	-24.456	0.000	5.04	-3.06	-0.607	
-4	8	5	10.55	3.34	3.341	0.000	7.32	7.21	0.985	
-2	8	5	41.93	43.70	-43.699	0.000	2.53	-1.77	-0.700	
1	7	5	17.36	14.95	-14.946	0.000	4.19	2.42	0.578	
-1	7	5	10.55	2.52	2.517	0.000	5.83	8.03	1.378	*
-3	7	5	17.41	10.55	-10.546	0.000	4.19	6.87	1.641	
-7	7	5	20.85	11.94	11.940	0.000	5.11	8.91	1.744	*
-9	7	5	13.01	13.75	-13.754	0.000	8.49	-0.74	-0.088	
-8	6	5	15.62	0.45	0.452	0.000	6.55	15.17	2.317	*
-6	6	5	8.09	8.63	-8.629	0.000	7.95	-0.54	-0.068	
-4	6	5	15.91	1.00	0.997	0.000	4.20	14.91	3.548	*
-2	6	5	17.62	9.70	-9.704	0.000	3.63	7.91	2.177	
0	6	5	16.16	5.73	-5.727	0.000	3.68	10.43	2.832	*
3	5	5	25.02	27.90	-27.895	0.000	3.15	-2.88	-0.914	
1	5	5	13.71	12.37	-12.366	0.000	4.50	1.35	0.299	
-1	5	5	19.57	14.79	-14.790	0.000	3.23	4.78	1.480	
-3	5	5	12.84	15.98	-15.980	0.000	4.42	-3.14	-0.710	
-5	5	5	4.71	7.35	-7.350	0.000	8.37	-2.64	-0.316	*
-7	5	5	26.72	26.45	-26.454	0.000	3.42	0.27	0.079	
-11	5	5	22.30	3.51	-3.512	0.000	6.18	18.79	3.041	*
-8	4	5	22.79	8.83	8.827	0.000	4.15	13.96	3.362	*
-6	4	5	22.10	18.94	18.939	0.000	3.05	3.16	1.038	
-4	4	5	16.09	11.47	-11.466	0.000	3.48	4.63	1.328	
-2	4	5	34.31	33.70	33.698	0.000	2.13	0.61	0.288	
2	4	5	10.08	16.16	16.162	0.000	5.78	-6.08	-1.053	
5	3	5	41.46	45.95	45.946	0.000	2.19	-4.49	-2.045	
3	3	5	2.11	2.67	2.670	0.000	10.21	-0.56	-0.055	*
1	3	5	80.44	79.60	79.600	0.000	1.69	0.84	0.498	
-1	3	5	51.52	56.76	56.755	0.000	1.69	-5.23	-3.093	
-3	3	5	33.05	31.83	31.831	0.000	1.86	1.22	0.658	
-5	3	5	54.14	55.33	55.325	0.000	1.61	-1.19	-0.740	
-7	3	5	19.71	25.38	25.377	0.000	3.73	-5.67	-1.518	
-9	3	5	58.39	57.90	57.902	0.000	2.33	0.49	0.209	
-12	2	5	15.37	22.27	22.266	0.000	8.10	-6.89	-0.851	
-10	2	5	19.54	10.33	10.326	0.000	5.32	9.22	1.731	*
-8	2	5	31.50	29.23	29.233	0.000	2.71	2.26	0.835	
-6	2	5	38.30	39.13	39.127	0.000	1.84	-0.83	-0.451	
-4	2	5	11.87	2.05	-2.052	0.000	4.35	9.82	2.256	*
-2	2	5	62.17	64.72	64.716	0.000	1.57	-2.54	-1.615	
0	2	5	12.37	19.45	19.449	0.000	4.87	-7.07	-1.452	
2	2	5	30.30	37.43	37.427	0.000	1.91	0.87	0.455	
4	2	5	18.35	18.64	18.643	0.000	4.00	-0.29	-0.073	
3	1	5	27.43	26.06	-26.063	0.000	2.60	1.37	0.526	
-1	1	5	21.10	11.44	-11.439	0.000	2.70	9.66	3.583	*
-3	1	5	9.11	8.00	-8.001	0.000	4.97	1.11	0.223	
-7	1	5	18.82	25.27	-25.273	0.000	3.68	-6.45	-1.752	
-9	1	5	10.13	7.97	7.968	0.000	7.60	2.16	0.284	
-11	1	5	18.49	3.07	-3.069	0.000	6.21	15.42	2.482	*

9/05/19 1040C HI-CLINO FERROSILITE Table 7-7 (Sueno, Kimata & Prewitt) STRUCT

H	K	L	F(OBS)	F(CALC)	A(CALC)	B(CALC)	SIGMA	DELTA F	DEL/SIG	
-3	3	7	18.94	20.38	-20.380	0.000	4.12	-1.44	-0.350	
-5	3	7	35.62	37.19	-37.191	0.000	2.51	-0.57	-0.227	
-7	3	7	5.63	10.84	-10.842	0.000	8.69	-5.22	-0.600	*
-6	2	7	30.68	29.23	-29.233	0.000	2.56	1.44	0.563	
-4	2	7	11.14	3.73	3.728	0.000	5.43	7.41	1.365	
-2	2	7	32.43	27.56	-27.561	0.000	2.70	4.87	1.808	
-1	1	7	14.85	8.41	8.409	0.000	4.86	6.44	1.327	

11	3	1	33.17	37.60	37.595	0.000	4.10	-4.42	-1.078
9	3	1	32.50	33.95	33.948	0.000	3.45	-1.45	-0.419
7	3	1	18.49	25.55	25.553	0.000	4.14	-7.07	-1.709
5	3	1	181.03	184.89	184.890	0.000	2.80	-3.86	-1.381
3	3	1	75.70	73.82	73.819	0.000	1.36	1.88	1.389
1	3	1	78.65	83.04	83.040	0.000	1.32	-4.39	-3.318

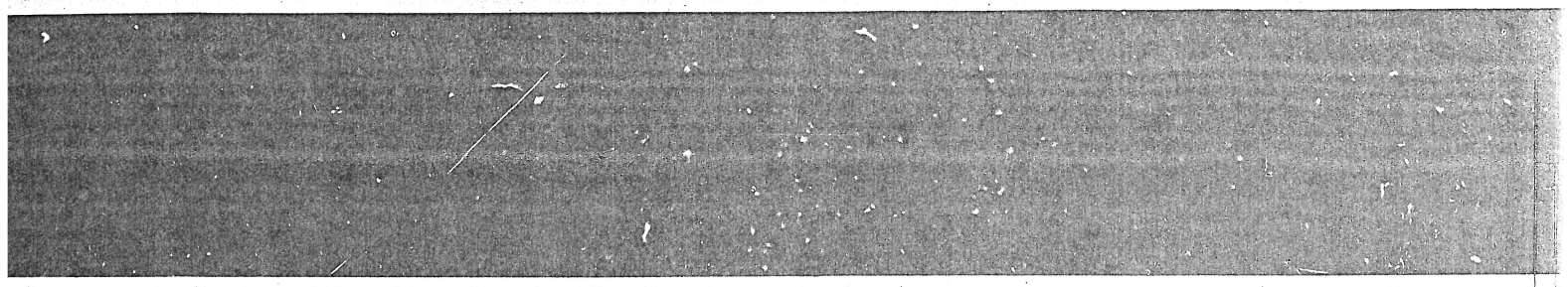

Table 7-8 (Sueno, Kimata & Prewitt)

79/05/19 1040C MI-CLINO FERROSILITE

NO.	ATOM	PARAMETER	OLD	CHANGE	NEW	ERROR	SHIFT/ERROR
		EXTINCTION PARAM.	0.0000E 00		0.0000E 00		
		SCALE FACTOR 1	0.5972	-0.0002	0.5970	0.0045	-0.04
1	FE1	EQUI. FRACTION = 0.500					
		FE2 OCCUPANCY	1.0000		1.0000		
		X	0.000000		0.000000		
		Y	0.901497	-0.000119	0.901378	0.000586	-0.20
		Z	0.250000		0.250000		
		BETA11	0.005941	0.000168	0.006110	0.000547	0.31
		BETA22	0.005980	0.000339	0.006319	0.000728	0.47
		BETA33	0.016220	-0.000552	0.015667	0.001188	-0.46
		BETA12	-0.000000		-0.000000		
		BETA13	0.001735	-0.000101	0.001635	0.000551	-0.18
		BETA23	-0.000000		-0.000000		
2	FE2	EQUI. FRACTION = 0.500					
		FE2 OCCUPANCY	1.0000		1.0000		
		X	0.000000		0.000000		
		Y	0.262846	-0.000354	0.262492	0.000563	-0.63
		Z	0.250000		0.250000		
		BETA11	0.006802	-0.000146	0.006656	0.000696	-0.21
		BETA22	0.010797	-0.000041	0.010756	0.000895	-0.05
		BETA33	0.020294	0.000205	0.020500	0.001538	0.13
		BETA12	-0.000000		-0.000000		
		BETA13	0.000665	-0.000129	0.000536	0.000738	-0.17
		BETA23	-0.000000		-0.000000		
3	SI	EQUI. FRACTION = 1.000					
		SI OCCUPANCY	1.0000		1.0000		
		X	0.296298	-0.000061	0.296237	0.000472	-0.13
		Y	0.085084	0.000139	0.085223	0.000761	0.18
		Z	0.270681	0.000128	0.270809	0.000654	0.20
		BETA11	0.005168	0.000081	0.005249	0.000679	0.12
		BETA22	0.006421	-0.000057	0.006363	0.000746	-0.08
		BETA33	0.015055	0.000169	0.015223	0.001452	0.12
		BETA12	-0.002452	-0.000207	-0.002659	0.000962	-0.21
		BETA13	0.001661	0.000016	0.001677	0.000735	0.02
		BETA23	0.000146	0.000271	0.000417	0.001054	0.26
4	O1	EQUI. FRACTION = 1.000					
		O OCCUPANCY	1.0000		1.0000		
		X	0.124117	-0.000010	0.124107	0.001304	-0.01
		Y	0.090505	-0.000229	0.090275	0.001804	-0.12
		Z	0.158479	0.000206	0.158686	0.001614	0.13
		BETA11	0.008994	-0.000476	0.008517	0.001725	-0.28
		BETA22	0.015585	-0.001734	0.013851	0.002252	-0.77
		BETA33	0.013203	0.000874	0.014077	0.003555	0.25
		BETA12	-0.002842	-0.000081	-0.002923	0.002435	-0.03
		BETA13	-0.001272	-0.000056	-0.001328	0.001867	-0.03
		BETA23	-0.001836	0.001099	-0.000737	0.003032	0.36

5	3	2	5.78	13,23	13.229	0.000	7.03	-7.45	-1.060 *
3	3	2	10.63	12.29	12.294	0.000	4.39	-1.66	-0.379
1	3	2	27.68	24.59	-24.591	0.000	1.41	3.09	2.195
-1	3	2	10.87	2.43	2.430	0.000	2.71	8.44	3.110 *
-3	3	2	48.34	35.53	35.528	0.000	1.21	12.81	10.629 *

79/05/19 1040C HI-CLINO FERROSILITE Table 7-9 (Sueno, Kimata & Prewitt)

NO.	ATOM	PARAMETER	OLD	CHANGE	NEW	ERROR	SHIFT/ERROR
5	O2	EQUI. FRACTION = 1.000					
		O OCCUPANCY	1.0000		1.0000		
		X	0.374492	0.000214	0.374706	0.001415	0.15
		Y	0.240419	-0.000634	0.239785	0.001306	-0.49
		Z	0.361528	0.000023	0.361551	0.001942	0.01
		BETA11	0.005307	0.000705	0.006012	0.001778	0.40
		BETA22	0.002453	0.000239	0.002691	0.001780	0.13
		BETA33	0.040027	-0.000092	0.040735	0.004943	-0.02
		BETA12	-0.006175	-0.000647	-0.006822	0.001536	-0.42
		BETA13	0.007498	0.000913	0.008411	0.002322	0.39
		BETA23	0.000491	0.000469	0.000960	0.002426	0.19
6	O3	EQUI. FRACTION = 1.000					
		O OCCUPANCY	1.0000		1.0000		
		X	0.359169	-0.000257	0.358912	0.001513	-0.17
		Y	0.912792	0.000652	0.013444	0.001217	0.54
		Z	0.044631	-0.000144	0.044487	0.001969	-0.07
		BETA11	0.002422	-0.000379	0.002043	0.001800	-0.21
		BETA22	0.010851	0.001296	0.012147	0.002253	0.58
		BETA33	0.023085	0.000656	0.023741	0.005419	0.12
		BETA12	-0.004441	-0.000020	-0.004461	0.001322	-0.02
		BETA13	0.003067	-0.000361	0.002726	0.002215	-0.16
		BETA23	-0.012457	-0.000728	-0.013185	0.002044	-0.36

MAXIMUM SHIFT/ERROR = -0.77

AVERAGE SHIFT/ERROR = 0.22