

Table 3. Observed and calculated structure factors for γ -Na₂ZrF₆

L	F _{OBS}	F _{CAL}	L	F _{OBS}	F _{CAL}	L	F _{OBS}	F _{CAL}	L	F _{OBS}	F _{CAL}	L	F _{OBS}	F _{CAL}	L	F _{OBS}	F _{CAL}	L	F _{OBS}	F _{CAL}
0	0	0	1	0	0	2	0	0	3	0	0	4	0	0	5	0	0	6	0	0
7	0	0	8	0	0	9	0	0	10	0	0	11	0	0	12	0	0	13	0	0
14	0	0	15	0	0	16	0	0	17	0	0	18	0	0	19	0	0	20	0	0
21	0	0	22	0	0	23	0	0	24	0	0	25	0	0	26	0	0	27	0	0
28	0	0	29	0	0	30	0	0	31	0	0	32	0	0	33	0	0	34	0	0
35	0	0	36	0	0	37	0	0	38	0	0	39	0	0	40	0	0	41	0	0
42	0	0	43	0	0	44	0	0	45	0	0	46	0	0	47	0	0	48	0	0
49	0	0	50	0	0	51	0	0	52	0	0	53	0	0	54	0	0	55	0	0
56	0	0	57	0	0	58	0	0	59	0	0	60	0	0	61	0	0	62	0	0
63	0	0	64	0	0	65	0	0	66	0	0	67	0	0	68	0	0	69	0	0
70	0	0	71	0	0	72	0	0	73	0	0	74	0	0	75	0	0	76	0	0
77	0	0	78	0	0	79	0	0	80	0	0	81	0	0	82	0	0	83	0	0
84	0	0	85	0	0	86	0	0	87	0	0	88	0	0	89	0	0	90	0	0
91	0	0	92	0	0	93	0	0	94	0	0	95	0	0	96	0	0	97	0	0
98	0	0	99	0	0	100	0	0	101	0	0	102	0	0	103	0	0	104	0	0
105	0	0	106	0	0	107	0	0	108	0	0	109	0	0	110	0	0	111	0	0
112	0	0	113	0	0	114	0	0	115	0	0	116	0	0	117	0	0	118	0	0
119	0	0	120	0	0	121	0	0	122	0	0	123	0	0	124	0	0	125	0	0
126	0	0	127	0	0	128	0	0	129	0	0	130	0	0	131	0	0	132	0	0
133	0	0	134	0	0	135	0	0	136	0	0	137	0	0	138	0	0	139	0	0
140	0	0	141	0	0	142	0	0	143	0	0	144	0	0	145	0	0	146	0	0
147	0	0	148	0	0	149	0	0	150	0	0	151	0	0	152	0	0	153	0	0
154	0	0	155	0	0	156	0	0	157	0	0	158	0	0	159	0	0	160	0	0
161	0	0	162	0	0	163	0	0	164	0	0	165	0	0	166	0	0	167	0	0
168	0	0	169	0	0	170	0	0	171	0	0	172	0	0	173	0	0	174	0	0
175	0	0	176	0	0	177	0	0	178	0	0	179	0	0	180	0	0	181	0	0
182	0	0	183	0	0	184	0	0	185	0	0	186	0	0	187	0	0	188	0	0
189	0	0	190	0	0	191	0	0	192	0	0	193	0	0	194	0	0	195	0	0
196	0	0	197	0	0	198	0	0	199	0	0	200	0	0	201	0	0	202	0	0
203	0	0	204	0	0	205	0	0	206	0	0	207	0	0	208	0	0	209	0	0
210	0	0	211	0	0	212	0	0	213	0	0	214	0	0	215	0	0	216	0	0
217	0	0	218	0	0	219	0	0	220	0	0	221	0	0	222	0	0	223	0	0
224	0	0	225	0	0	226	0	0	227	0	0	228	0	0	229	0	0	230	0	0
231	0	0	232	0	0	233	0	0	234	0	0	235	0	0	236	0	0	237	0	0
238	0	0	239	0	0	240	0	0	241	0	0	242	0	0	243	0	0	244	0	0
245	0	0	246	0	0	247	0	0	248	0	0	249	0	0	250	0	0	251	0	0
252	0	0	253	0	0	254	0	0	255	0	0	256	0	0	257	0	0	258	0	0
259	0	0	260	0	0	261	0	0	262	0	0	263	0	0	264	0	0	265	0	0
266	0	0	267	0	0	268	0	0	269	0	0	270	0	0	271	0	0	272	0	0
273	0	0	274	0	0	275	0	0	276	0	0	277	0	0	278	0	0	279	0	0
280	0	0	281	0	0	282	0	0	283	0	0	284	0	0	285	0	0	286	0	0
287	0	0	288	0	0	289	0	0	290	0	0	291	0	0	292	0	0	293	0	0
294	0	0	295	0	0	296	0	0	297	0	0	298	0	0	299	0	0	300	0	0

The Zr⁴⁺ ion and the Na(1)⁺ ion are surrounded by an irregular array of 7F⁻ ions. The resulting Zr-F polyhedron has nine triangular faces and the Na(1)-F polyhedron has

three triangular faces and two trapezium faces. ZACHARIASEN (1948) found similar Zr-7F polyhedra in the structure of Na₃ZrF₇. The Zr-F polyhedron shares edges with Na(1)-F polyhedra and a corner with one other. The Na(1) ion is coordinated by 8F⁻ at the corners of a cube and a trapezohedron. The Zr-F polyhedron shares edges with four of the Na(2)-F trapezohedra. There are two trapezohedra similar to Na(1) and Na(2) with centers approximately $x=0.25$, $y=0.04$, $z=0.75$ and $x=0.50$, $z=0.90$. These polyhedra are vacant and do not contain Na or Zr cations. The final difference density map has no peaks greater than 1.60 e.Å⁻³. Interstitial matter put on the vacant sites does not give a difference density map which converges with a least-squares refinement. Vacancies perhaps explain why this polymorph is more stable with respect to Δ -Na₂ZrF₆ below 460°C.

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Indexing of the ψ -sulfur fiber pattern. By S. GELLER AND M. D. LIND, *Science Center, North American Corporation, Thousand Oaks, California, U.S.A.*

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The fiber pattern of ψ -sulfur reported by Tuinstra and the rotation photograph of the pressure-induced fibrous modification of sulfur (II) about the fiber axis (a) are essentially the same and have been indexed completely on the pseudo-orthorhombic C-face-centered cell with $a=13.8$, $b=32.4$ and $c=9.25$ Å.

We have recently completed a study of the structure of the pressure-induced fibrous form of sulfur (Lind & Geller, 1969). There is strong evidence that this form of sulfur is the same as the ψ -sulfur reported by Prins, Schenk & Wachters (1957; see also Prins & Tuinstra, 1963). Especially important is the exact match of the rotation photograph about the fiber (a) axis of a crystal of the pressure-induced phase and that of a fiber pattern of the ψ -sulfur.* Inasmuch as the literature (Tuinstra, 1966, 1967) contains questionable conclusions regarding the indexing of this pattern, it seemed worthwhile to give the results which follow.

It has already been reported (Geller, 1966) that the single-crystal-type diffraction data from the pressure-induced phase indicated that the crystals are C-centered orthorhombic with lattice constants $a=13.8$, $b=32.4$ and $c=9.25$ Å. The structure determination (Lind & Geller, 1969) has led to the conclusion that the crystal symmetry is more likely $P2_1$ and that the apparent orthorhombic symmetry results from a fine-grained twinning. The true monoclinic cell then has the lattice constants $a=17.6$, $b=9.25$, $c=$

13.8 Å, $\beta=113^\circ$. The orthorhombic indices listed in the powder pattern (Geller, 1966) may be transformed to monoclinic indices by application of the two $\frac{1}{2}001|100$ and $\frac{1}{2}001|100$ to each reflection.

We show the indexing of the rotation photograph of the pressure-induced phase in Table 1. Listed in the first column are Tuinstra's observed values, Q_0 ($Q_0=10^4/d^2$), measured on rotation photographs of the stretched, CS₂-extracted, fibrous sulfur. In the second column, we give the values of Q_0 , measured on a rotation photograph (2 hr, 57.3 mm dia. camera, Cu K α radiation, Ni filter) of the same crystal used to obtain the data in Table 1 by Lind & Geller (1969). (The photograph of Tuinstra (1967) refers to exactly the same except for exposure time.) We do not list the qualitative agreement as we said earlier, the photographs of stretched, extracted, annealed fibrous sulfur and pressure-induced fibrous sulfur superimpose exactly and quantitatively given in the Lind & Geller (1969) paper. We see two sets of Q_0 agree quite well although ours are generally better resolved. Our Q_0 and indices based on pseudo-orthorhombic lattice constants are given in the third and fourth columns, respectively. It is seen that the agreement in Q_0 's is excellent, so that even though the fiber axis is very long, as Tuinstra (1967)

* The best ψ -sulfur photograph we have seen has been made by J. Donohue and S. H. Goodman. This is the one that superposes exactly on our (pseudo-orthorhombic) a -axis rotation photograph.