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Refinement of the structure of $NaBF_4$*.

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The structure determined by Weiss & Zohner (*Phys. Stat. Sol.* (1967), **21**, 257) is confirmed.

The lattice parameters and atomic parameters of $NaBF_4$ have been remeasured as part of basic research on $NaBF_4$ and KBF_4 in connection with molten salt breeder reactors (ORNL, 1967). A mixture of the two salts is being investigated as a molten coolant for the reactors. The structure of $NaBF_4$ was recently determined by Weiss & Zohner (1967) from two-dimensional Weissenberg data, coincidental with a nuclear magnetic resonance study. The refined parameters are only slightly different from those of Weiss and Zohner and the structural results are the same.

Experimental

A single crystal of purified $NaBF_4$ was washed with water and acetone to smooth it to an ellipsoidal shape of the dimensions $0{\cdot}0234 \times 0{\cdot}0572 \times 0{\cdot}0676$ cm. The reflection intensities and lattice parameters were measured with a computer controlled Picker four-circle goniostat and a scintillation-counter detector using unfiltered Mo $K\alpha$ radiation ($K\alpha_1 = 0{\cdot}70926$, $K\alpha_2 = 0{\cdot}713543$ Å) (Busing, Ellison, Levy, King & Roseberry, 1968). Independent reflections out to 80° 2θ were measured by the 2θ scan technique. The structure was refined by iterative least squares with the Busing, Martin & Levy (1962) computer program as modified by C. K. Johnson of the Chemistry Division of ORNL. The starting parameters were taken from Weiss & Zohner (1967) and the results are listed in Table 1. Scattering factors for Na, B and F were taken from Cromer & Waber (1965). The quantity minimized in the least-squares program was $\Sigma\, w||sF_o| - |F_c||^2$ with weights, w, equal to the reciprocals of the variances which were estimated from the empirical equation: $\sigma^2(F_o^2) = [T + B + \{0{\cdot}05(T-B)\}^2]/[A(\mathrm{Lp})^2]$ where T = total counts, B = background counts, A = absorption correction, Lp = Lorentz-polarization. The quantity $\{0{\cdot}05(T-B)\}^2$ is added to account for the presence of systematic errors (Brown & Levy, 1964); $\sigma(F_o) = \sigma(F_o^2)/2F_o$. Anisotropic temperature factors were calculated for all atoms and a stereoscopic pair of drawings of one unit cell of $NaBF_4$ are shown in Fig. 1.

The discrepancy index $R = \Sigma\, ||F_o| - |F_c||/\Sigma\, |F_o|$ is 0·036 for 508 independent reflections. The standard deviation of an observation of unit weight, $[\Sigma\, w(F_o - F_c)^2/(n_o - n_v)]^{1/1}$, where n_o is the number of reflections and n_v, the number of variables, is 2·048. The interatomic distances and F–B–F angles are listed in Table 2 and the observed and calculated

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Table 1. *Lattice and atomic parameters* for* $NaBF_4$

Space group *Cmcm*.

$a_0 = 6{\cdot}8368$ (9) Å, $b_0 = 6{\cdot}2619$ (7) Å, $c_0 = 6{\cdot}7916$ (4) Å. Calculated density 2·5075 g.cm^{-3}.

	x	y	z	β_{11}†	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Na	0·0000	0·6552 (2)	0·2500	0·0136 (2)	0·0121 (2)	0·0089 (2)	0·0000	0·0	0·0000
B	0·0000	0·1608 (3)	0·2500	0·0085 (3)	0·0078 (3)	0·0076 (3)	0·0000	0·0	0·0000
F(1)	0·0000	0·2920 (2)	0·08458 (0·9)	0·0185 (3)	0·0137 (2)	0·0087 (2)	0·0000	0·0	0·0029 (2)
F(2)	0·1644 (1)	0·0312 (2)	0·2500	0·0121 (2)	0·0156 (3)	0·0201 (3)	0·0054 (2)	0·0	0·0000

* The number in parentheses is $\sigma \times 10^4$.

† Coefficients in the temperature factor; $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

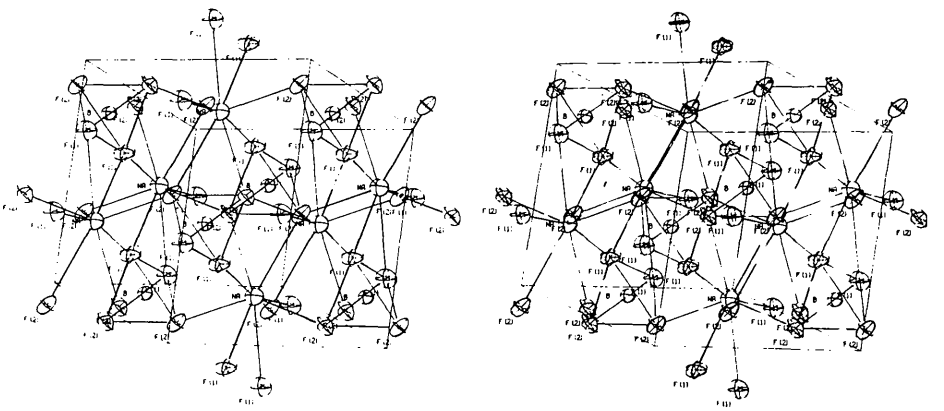

Fig. 1. A stereoscopic pair of drawings of the unit cell of $NaBF_4$. The unit cell is outlined and has been tilted −15° around the x axis and −30° around the y axis of the drawing.

Table 2. *Interatomic distances and* F–B–F *angles for* $NaBF_4$

The number in parentheses on the interatomic distances is $\sigma \times 10^4$ and on the angles $\sigma \times 10$.

2[Na–F(1)]	2·2963 (0·7) Å	2[Na–F(2)]	2·609 (2) Å
2[Na–F(2)]	2·4224 (0·8)	2[B–F(2)]	1·386 (2)
2[Na–F(1)]	2·537 (2)	2[B–F(1)]	1·392 (2)
F(1)–F(1)	2·247 (2)	F(1)–F(1)	2·848 (2)
2[F(1)–F(2)]	2·279 (2)	2[F(1)–F(2)]	2·9619 (0·9)
F(2)–F(2)	2·248 (2)		
F(2)–B–F(2)	108·3 (2)°	4[F(2)–B–F(1)]	110·21 (0·3)°
F(1)–B–F(1)	107·7 (2)		

Table 3. *Observed and calculated structure factors* $\times 10$ *for* $NaBF_4$

L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL
	0	0 L	7	80	79	4	57	-56	0	134	135		3	5 L	0	137	-140	4	35	36	0	42	40	2	61	-60
2	706	-763	8	0	-4	5	20	-13	1	180	184	0	246	-255	1	114	109	5	47	19	1	32	-34	3	25	23
4	446	434		0	10 L	6	45	-44	2	130	-132	1	39	38	2	243	245	6	100	-101	2	34	31	4	51	50
6	547	-542	0	24	-24	7	29	29	3	40	19	2	17	16	3	270	-273	7	11	-12	3	80	-78	5	30	-31
8	124	123	1	0	3	8	27	-30	4	90	90	3	71	-71	4	171	-173	8	13	12	4	26	-28	6	9	12
10	52	-52	2	63	65	9	3	4	5	103	105	4	0	6	5	74	73	9	29	-31	5	7	-2	7	12	12
12	83	83	3	20	16		1	9 L	6	74	-75	5	4	-4	6	59	56	10	0	5	6	3	-6	8	19	19
	0	2 L	4	59	-60	0	26	-28	7	73	-73	6	108	108	7	31	-34		5	5 L	7	5	3		7	7 L
0	161	-152	5	11	14	1	15	13	8	61	63	7	10	-10	8	78	-78	0	89	-91	8	29	-30	0	41	42
1	470	-474		1	1 L	2	25	-25	9	18	-18	8	3	7	9	80	80	1	107	-111	9	41	41	1	29	-27
2	488	-470	0	40	27	3	41	37	10	23	-23	9	13	14	10	39	37	2	45	-46		6	6 L	2	7	3
3	657	649	1	185	-171	4	19	14		2	8 L	10	16	-16		4	0 L	3	60	59	0	103	101	3	12	11
4	276	266	2	209	-196	5	4	5	0	31	-30		3	7 L	0	72	71	4	43	43	1	11	-9	4	8	-4
5	240	-232	3	557	-543	6	19	22	1	83	-84	0	35	30	1	130	131	5	65	-67	2	96	-96	5	17	-12
6	112	117	4	108	103	7	18	-18	2	14	-16	1	73	75	3	9	12	6	48	49	3	76	76	6	23	-24
7	113	114	5	57	-31		1	11 L	3	7	1	2	82	84	4	49	51	7	28	28	4	71	71		7	9 L
8	50	47	6	49	39	0	11	9	4	25	24	3	90	-90	5	77	79	8	16	17	5	5	3	0	0	-4
9	125	-126	7	27	29	1	28	-24	5	65	-69	4	65	-64	6	47	-48	9	7	-6	6	61	-55	1	5	-5
10	21	-19	8	10	8	2	7	2	6	24	21	5	49	48	7	57	-56		5	7 L	7	11	-10	2	15	-14
11	42	43	9	102	101		2	0 L	7	47	47	6	23	-26	8	43	45	0	15	17	8	44	43		8	0 L
12	38	-39	10	12	-10	0	723	772	8	2	-2	7	8	-8	9	15	-13	1	35	-36		6	8 L	0	117	120
	0	4 L	11	9	10	2	62	51		2	10 L	8	31	-30		4	6 L	2	58	58	0	24	-21	2	27	-29
0	174	171	12	12	-11	4	9	-7	0	0	-2	9	19	18	0	68	-64	3	5	9	1	101	-100	4	24	26
1	149	-145		1	3 L	6	294	-303	1	37	34		3	9 L	1	59	-61	4	46	-45	2	0	-1	6	73	-76
2	88	78	0	643	659	8	40	40	2	43	42	0	13	-16	2	0	9	5	15	-14	3	53	52	8	25	20
3	223	-228	1	84	84	10	22	-20	3	14	-13	1	68	68	3	8	0	6	19	-17	4	11	7		8	2 L
4	71	-66	2	200	-198	12	67	67	4	44	-42	2	27	-28	4	0	3	7	22	21	5	70	-70	0	107	-108
5	16	-21	3	251	246		2	2 L	5	32	33	3	17	-15	5	50	-52	8	21	-22	6	16	14	1	39	-41
6	20	-28	4	104	106	0	751	-750		3	1 L	4	16	17	6	27	25		5	9 L		7	1 L	2	36	33
7	16	16	5	55	62	1	135	-139	0	647	-651	5	36	36	7	38	37	0	33	-35	0	20	22	3	73	75
8	59	-62	6	212	-219	2	127	127	1	38	37	6	15	15		4	10 L	1	13	9	1	15	-13	4	27	-25
9	85	86	7	35	-34	3	372	378	2	422	414		3	11 L	0	15	-14	2	3	2	2	48	-50	5	34	-35
10	30	31	8	31	32	4	72	-77	3	477	-476	0	16	22	1	37	39	3	22	21	3	95	-99	6	57	58
11	14	-12	9	49	-49	5	123	-115	4	243	-245		4	0 L	2	46	44	4	6	-5	4	36	38	7	23	24
	0	6 L	10	4	4	6	238	248	5	25	21	0	427	428	3	22	-18	5	6	4	5	8	-10	8	10	-10
0	209	215	11	20	-19	7	64	63	6	176	181	2	23	25	4	45	-42		6	0 L	6	6	-2	9	33	-32
1	8	7		1	5 L	8	21	-19	7	4	4	4	2	2		5	1 L	0	492	505	7	13	12		8	4 L
2	202	-202	0	149	-150	9	100	-100	8	58	-58	6	206	-211	0	17	-16	2	254	-256	8	10	10	0	33	-31
3	172	177	1	207	-207	10	7	7	9	89	89	8	32	32	1	45	-43	4	177	182	9	31	30	1	64	63
4	138	137	2	133	-141	11	34	34	10	17	17	10	16	-17	2	50	-53	6	216	-220	10	5	-7	2	58	60
5	22	22	3	95	101		2	4 L	11	7	6		4	2 L	3	201	-207	8	69	68		7	3 L	3	103	-102
6	103	-104	4	108	113	0	118	-125		3	3 L	0	496	-493	4	38	38	10	32	-33	0	143	147	4	46	-47
7	31	-30	5	115	-119	1	169	164	0	99	95	1	133	-130	5	19	-20		6	2 L	1	19	19	5	42	42
8	80	79	6	74	77	2	296	297	1	307	298	2	131	125	6	18	18	0	30	-24	2	59	-61	6	22	18
9	41	-42	7	45	46	3	393	-406	2	166	171	3	271	272	7	19	21	1	171	-171	3	44	49	7	21	-24
10	30	-29	8	33	35	4	199	-202	3	14	10	4	82	-81	8	9	6	2	115	-122	4	35	36	8	27	-28
	0	8 L	9	8	-9	5	103	101	4	119	-121	5	101	-99	9	53	53	3	220	223	5	15	17		8	6 L
0	38	-43	10	33	-30	6	54	51	5	138	143	6	184	188	10	9	-7	4	80	81	6	66	-65	0	63	58
1	184	-186	11	25	23	7	42	-43	6	65	-68	7	57	56	11	5	8	5	106	-107	7	9	-10	1	54	54
2	13	-8		1	7 L	8	91	-91	7	70	-72	8	26	-22		5	3 L	6	40	40	8	15	11	2	55	-56
3	77	77	0	72	72	9	102	103	8	22	-22	9	81	-81	0	239	246	7	61	61	9	14	-16	3	13	-9
4	20	20	1	47	-48	10	44	43	9	26	-21	10	10	8	1	12	11	9	64	-65		7	5 L	4	41	43
5	124	-125	2	71	74	11	20	-21	10	23	23	11	30	29	2	68	-71	10	9	-8	0	8	-7	5	36	35
6	21	26	3	0	1		2	6 L	11	23	-23		4	4 L	3	117	121		6	4 L	1	45	-47	6	35	-34
L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL	L	FOBS	FCAL
	8	8 L	4	42	-42	4	35	-34	5	13	13	4	11	10	6	35	36	0	5	9	4	8	8	0	13	-12
0	0	4	5	0	0	5	29	29	6	23	22	0	34	-34		10	4 L	1	27	26	5	10	-8	1	16	-15
1	28	-27	6	37	35	6	7	1		9	7 L		10	2 L	0	44	-41	2	10	-9		11	3 L		12	0 L
2	17	-15	7	6	3	7	18	-17	0	10	8	0	64	-66	1	26	27	3	4	-5	0	18	13	0	60	57
3	10	6	8	16	-14		9	5 L	1	23	23	1	32	-32	2	50	51		11	1 L	1	10	-7	2	41	-40
	9	1 L		9	3 L	0	44	-41	2	12	13	2	33	33	3	45	-49	0	5	5	2	9	1		12	2 L
0	81	-81	0	13	-12	1	31	27	3	29	-25	3	45	45	4	40	-40	1	13	-12	3	17	18	0	1	3
1	8	6	1	47	47	2	5	7		10	0 L	4	25	-26	5	19	20	2	11	-10	4	0	-2	1	28	-28
2	60	60	2	42	42	3	28	-30	0	50	49	5	26	-25		10	6 L	3	4	-9		11	5 L	2	16	-13
3	50	-51	3	15	-10	4	1	-2	2	10	-9															

structure factors are listed in Table 3. An extinction correction was made on F_c by the method suggested by Zachariasen (1967). The extinction correction has been incorporated into the general least-squares program by C. K. Johnson of the ORNL Chemistry Division.

The BF_4 tetrahedra are slightly irregular with two $F^-(1)$ ions 0·006 Å more distant from the B^{3+} ion than the two $F^-(2)$ ions (Fig. 1 and Table 2). The Na^+ ions are coordinated by 8 F^- ions which are at the corners of an irregular polyhedron which is neither a cube nor antiprism. The Na^+ polyhedron shares opposite edges with BF_4 tetrahedra, four corners with BF_4 tetrahedra and edges with two other Na^+ polyhedra. The Na^+–F^- distances vary from 2·2963 to 2·609 Å.

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