

of the zinc and chlorine atoms. A three-dimensional Fourier synthesis using phases determined by these atoms then established the positions of the nitrogen and carbon atoms. Refinement of both positions and temperature factors proceeded by a few Fourier sections and several difference syntheses. A final over-all Reliability Index of 15.5 was obtained.* For special classes of reflections the indices were:

$$\begin{array}{lll} h+l = 2n; & k = 2n & 14.9 \\ h+l = 2n; & k = 2n+1 & 16.7 \\ h+l = 2n+1; & k = 2n & 15.8 \\ h+l = 2n+1; & k = 2n+1 & 15.0 \end{array}$$

The structure exhibited a high temperature factor for the lighter elements and hydrogen atom contributions were not calculated. Atomic positions and individual isotropic temperature factors are listed in Table 1. All atoms are in positions 4c except Cl(2), C(3), and C(6), which are in 8d.

Table 1. Atomic positional parameters and temperature factors for $[\text{N}(\text{CH}_3)_4]_2\text{ZnCl}_4$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
Zn	0.2460	0.2500	0.4075	4.000
Cl ₁	0.0630	0.2500	0.4070	7.400
Cl ₂	0.3060	0.0445	0.3400	7.400
Cl ₃	0.3130	0.2500	0.5418	8.300
N ₁	0.1530	0.2500	0.0975	5.000
N ₂	0.4945	0.2500	0.8240	5.500
C ₁	0.2770	0.2500	0.0970	8.000
C ₂	0.1085	0.2500	0.0020	8.500
C ₃	0.1115	0.3910	0.1450	9.000
C ₄	0.4405	0.2500	0.7345	8.500
C ₅	0.4030	0.2500	0.8935	9.500
C ₆	0.5660	0.3910	0.8340	9.000

Discussion

The refinement of the three-dimensional data yielded the

* The table of calculated and observed structure factors may be obtained from E. C. Lingafelter.

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Crystallographic evidence for the existence of B₇O*. By R. A. PASTERNAK, *Stanford Research Institute, Menlo Park, California, U.S.A.*

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Boron can combine with oxygen in different atomic proportions. In addition to the familiar B₂O₃, boron oxides containing less oxygen have been reported in the literature. The existence of the gaseous BO has been proven convincingly (Albrecht & Mallett, 1954), whereas the characterization of a suboxide B₃O (Kahlenberg, 1925) has been less than satisfactory. This note presents crystallographic evidence of the occurrence of the suboxide, B₇O.

In a preliminary study (Eding, 1956), the brown microcrystalline material which had been obtained from the

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expected tetrahedral configuration for the ZnCl₄²⁻ ion. Bond lengths and bond angles are given in Table 2.

Table 2. Bond lengths and angles in $[\text{N}(\text{CH}_3)_4]_2\text{ZnCl}_4$, with standard deviations

Zn-Cl ₁	2.245 ± 0.006 Å	Cl ₁ -Zn-Cl ₂	109.1 ± 0.22°
Zn-Cl ₂	2.243 ± 0.006	Cl ₁ -Zn-Cl ₃	111.7 ± 0.22
Zn-Cl ₃	2.240 ± 0.006	Cl ₂ -Zn-Cl ₃	108.3 ± 0.22
N ₁ -C ₁	1.521 ± 0.019	Cl ₂ -Zn-Cl ₂	110.4 ± 0.22
N ₁ -C ₂	1.579 ± 0.019	C ₁ -N ₁ -C ₂	109.9 ± 0.97
N ₁ -C ₃	1.549 ± 0.019	C ₁ -N ₁ -C ₃	109.3 ± 0.97
N ₂ -C ₄	1.539 ± 0.019	C ₅ -N ₂ -C ₃	109.4 ± 0.97
N ₂ -C ₅	1.557 ± 0.019	C ₃ -N ₂ -C ₃	109.4 ± 0.97
N ₂ -C ₆	1.546 ± 0.019	C ₄ -N ₂ -C ₅	108.3 ± 0.97
		C ₄ -N ₂ -C ₆	109.6 ± 0.97
		C ₅ -N ₂ -C ₆	109.8 ± 0.97
		C ₆ -N ₂ -C ₆	109.7 ± 0.97

Shorter interionic distances:

Cl ··· Cl	5.293, 5.523 Å
CH ₃ ··· CH ₃	3.515, 3.562
CH ₃ ··· Cl	3.374, 3.509

This structure is very similar to that of Cs₂ZnCl₄ (Brehler, 1957) with the Cs⁺ ions being replaced by N(CH₃)₄⁺ ions and with each unit-cell edge expanded by a little over 20%.

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American Potash & Chemical Corporation was found to contain only traces of nitrogen and magnesium, besides boron and oxygen; its analytical formula was B_{6.6}O. Its X-ray pattern did not agree with that of any of the known boron modifications or of boron oxide, or with any of their combinations. The material appeared to be homogeneous, because partial chemical dissolution did not change the X-ray pattern. These results could be reasonably explained by assuming that the sample was B₇O, contaminated by about 2% of B₂O₃. However, it could also be the suboxides B₁₃O₂ or B₆O contaminated with boron, or even a new modification of B containing amorphous B₂O₃.

To establish the identity of the material, a quantitative X-ray powder-study was undertaken. With Cu K α radiation, forty-four fairly sharp lines were obtained, some of

Table 1. *Observed and calculated spacings of B₂O₃ Philips powder camera (r=5.74 cm.) Cu K α radiation*

Line	Intensity*	$d_{\text{obs.}}$	$d_{\text{calc.}}$	hkl
1	<i>m</i>	4.343	4.349	101
2	<i>s</i>	4.114	4.102	200
3	<i>m</i>	3.715	3.704	011
4	<i>w</i>	2.679	2.677	020
5	<i>vs</i>	2.571	2.565	002
6	<i>vs</i>	2.284	2.280	121
7	<i>w</i>	2.251	2.242	220
8	<i>w</i>	2.178	2.175	202
9	<i>vw</i>	2.049	2.051	400
10	<i>w</i>	1.855	1.852	022
11	<i>vw</i>	1.737	1.744	130
12	<i>w</i>	1.691	1.688	222
13	<i>w</i>	1.649	1.651	131
14	<i>vw</i>	1.640	1.637	230
15	<i>s</i>	1.464	1.465	032
16	<i>m</i>	1.448	1.450	303
17	<i>s</i>	1.430	1.435	331
18	<i>vw</i>	1.403	1.400	313, 520
19	<i>m</i>	1.340	1.339	040
20	<i>m</i>	1.282	1.282	004
21	<i>vw</i>	1.274	1.272	240
22	<i>w</i>	1.261	1.267	104
23	<i>w</i>	1.244	1.247	014
26	<i>vw</i>	1.189	1.187	042
27	<i>w</i>	1.158	1.157	024
28	<i>w</i>	1.142	1.142	1.140 701, 242
29	<i>vw</i>	1.121	1.121	440
30	<i>vw</i>	1.090	1.087	404
31	<i>m</i>	1.026	1.025	1.027 800, 442
32	<i>m</i>	1.007	1.007	1.007 424, 810
33	<i>vvw</i>	0.9916	0.9903	0.9929 722, 514
34	<i>vvw</i>	0.9277	0.9259	044
35	<i>vw</i>	0.9098	0.9092	723
36	<i>w</i>	0.8917	0.8923	060
37	<i>m</i>	0.8776	0.8791	0.8794 061, 803
38	<i>w</i>	0.8691	0.8699	505
39	<i>vw</i>	0.8558	0.8550	006
40	<i>w</i>	0.8450	0.8440	444
41	<i>vvw</i>	0.8350	0.8368	206
42	<i>vw</i>	0.8253	0.8255	262
43	<i>vw</i>	0.8153	0.8160	0.8143 306, 026
44	<i>vw</i>	0.8068	0.8067	316

* *s*=strong, *m*=medium, *w*=weak, *v*=very.

which were back reflections, an indication of high degree of order in the structure. The observed reflections could be indexed on the basis of an orthorhombic unit cell. The dimensions, obtained by trial and error, are ($\lambda(\text{Cu } K\alpha) = 1.5418 \text{ \AA}$)

$$a = 8.20(4), \quad b = 5.35(4), \quad c = 5.13(0) \text{ \AA}.$$

The density of the material, determined experimentally by flotation, is $d = 2.64(4) \text{ g.cm.}^{-3}$. The observed and the calculated spacings, given in Table 1 agree to better than 0.5%. The differences are within the limit of accuracy of the measurements. Thus, the X-ray pattern arises from one homogeneous phase only.

From the unit-cell dimensions and the density, the mass of one mole of unit-cells is calculated to be 358.8. Assuming 4 equivalent units per unit cell, which is reasonable for an orthorhombic cell, the equivalent weight for the structural unit is 89.7. This agrees satisfactorily with the theoretical formula weight of B₂O₃, 91.7, especially if one considers possible experimental errors. The experimental density is likely to be lower than the true density of a material, because of air inclusion in the grains of the samples, and thus the observed formula weight tends to be low also. Compound B_{6.5}O (or $\frac{1}{2} \text{B}_{13}\text{O}_2$) would have the formula weight of 86.3, about 4% smaller than the observed value; therefore, the material studied is less likely to be this oxide. Thus, the existence of the boron suboxide B₂O appears to be established.

The structure of B₂O is probably closely related to that of elemental boron, in particular its tetragonal modification (Hoard *et al.*, 1958). This is indicated by the close similarity of the *c*-axes of the two structures (for tetragonal boron $c_0 = 5.06 \text{ \AA}$). The relative densities of B₂O and B also suggest this. Their ratio (2.64/2.31) is nearly equal to the mass ratio B₂O/B; the oxygen atoms are accommodated in the boron lattice without expanding it significantly. To establish the structure of B₂O unequivocally, single crystal data will be needed. Such work is not planned in this laboratory.

The author is indebted to the American Potash & Chemical Corporation, Los Angeles, for the sample of B₂O. Credit is due to Dr Dale Coulson and Dr Harold Eding for the analytical and the preliminary X-ray studies, and to Mrs H. Johnson for the experimental measurements.

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Book Reviews

Works intended for notice in this column should be sent direct to the Editor (P. P. Ewald, Polytechnic Institute of Brooklyn, 333, Jay Street, Brooklyn 1, N.Y., U.S.A.). As far as practicable books will be reviewed in a country different from that of publication.

Kompendium der Kristallkunde. By W. F. DE JONG. Pp. viii + 258, with 227 figs. and 41 tables. Vienna: Springer. 1959. Price S. 264, DM. 44, \$ 10.50. 75s.

This work is a translation into German of the author's *Kompendium der Kristallkunde* already reviewed in this journal (*Acta Cryst.* (1952), **5**, 858). A few diagrams have been added, a few have been redrawn, and some material

has been rearranged and slightly expanded, but in the main the translation follows the original very closely and therefore shares its merits and shortcomings. Among the merits is to be reckoned the ambitious coverage, ranging over morphological crystallography, structure analysis, crystal chemistry, and solid-state physics; among the inevitable shortcomings is the extremely superficial treatment which most of these topics receive. Thus piezoelectricity, the classical tensor theory of elasticity,