mal analysis. The structure of the tetragonal phase was found to be the $C11a$ type, in agreement with the previous room-temperature results. The $LaC_2$ pattern at 900°C gave $K=0{\cdot}24\pm0{\cdot}01$, $B=3{\cdot}1\pm0{\cdot}6$, $z=0{\cdot}404\pm0{\cdot}002$, $R=0{\cdot}08$, where $R=\Sigma w|I_o-I_c|/\Sigma wI_o$. Lattice parameters were $a_0=4{\cdot}000$, $c_0=6{\cdot}58$, giving a C–C distance of $1{\cdot}26\pm0{\cdot}03$ Å. The structure of the cubic phase was found to be the KCN type, isomorphous with cubic uranium dicarbide (Bowman, Arnold, Witteman, Wallace & Nereson 1966). This structure is face-centered, space group $Fm3m$, with metal atoms in $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$ and $C_2$ groups with centers at (0,0,0) randomly oriented along [111] directions. Intensities were calculated on the basis of one-fourth of a carbon atom in $(x,x,x)$. The $C_2$ groups may also be described by a free rotator model. There seems to be no significant difference between the two models. The $LaC_2$ pattern at 1100° gave $K=0{\cdot}06\pm0{\cdot}01$, $B=5{\cdot}6\pm2{\cdot}0$, $x=0{\cdot}061\pm0{\cdot}012$, $R=0{\cdot}02$. With $a_0=6{\cdot}022$, $d_{C-C}=1{\cdot}27\pm0{\cdot}06$ Å. The observed C–C distances are in reasonable agreement with the room temperature value of 1·30 Å (Atoji, 1961). The actual values may be somewhat larger, however, owing to the effect of thermal motion of the carbon atoms.

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## Crystal data for sodium tetragermanate

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(*Received* 11 *July* 1967)

Single-crystal X-ray studies on sodium tetragermanate, $Na_2Ge_4O_9$, gave $a_0=11{\cdot}335$ $c_0=9{\cdot}697$, space group $P6_3/m$, $Z=6$. This colorless transparent compound with $\omega=1{\cdot}731$ and $\varepsilon=1{\cdot}773$ has a measured density of 4·41 g.cm$^{-3}$ (27°C). Indexed powder data are listed.

Small single crystals of sodium tetragermanate ($Na_2Ge_4O_9$) were grown in slowly cooled 1:4 $Na_2O$–$GeO_2$ melts. The crystal data for the compound have not been published although Schwarz & Heinrich (1932) and Tresvyats'kii (1958) reported $Na_2Ge_4O_9$ in their studies of the $Na_2O$–$GeO_2$ phase system. Nowotny & Wittmann (1954) reported a $Na_2Ge_4O_9$ modification isotypic with $K_2Ge_4O_9$ and Shaw, Corwin & Edwards (1958) grew from a 1:4 $Na_2CO_3$–$GeO_2$ melt, a crystalline compound having indices of refraction very similar to those found in this study. A more recent investigation of the $Na_2O$–$GeO_2$ phase system by Murthy & Aguayo (1964) questioned the existence of sodium tetragermanate; however, chemical analysis and crystallographic data confirm $Na_2Ge_4O_9$ as a valid compound.

The crystals are colorless, transparent with a vitreous luster and have no apparent cleavage. The only crystal form present is the first order hexagonal prism $(10\bar{1}0)$; the terminating faces are not developed or interfered with by other crystal growth. The refractive indices of the crystal, measured by immersion oils for sodium light ($\lambda=589{\cdot}2$ m$\mu$) at 25°C, are $\varepsilon=1{\cdot}773\pm0{\cdot}001$ and $\omega=1{\cdot}731\pm0{\cdot}001$.

Since the only systematic extinctions observed in Weissenberg and precession photographs were for $000l$, where $l$ is odd, the crystals are in hexagonal crystal class 6 or $6/m$. A thin section (0·2 mm thick) containing crystals oriented normal to the $c$ axis was examined under the petrographic microscope using sodium light. No optical activity was observed and, therefore, it is concluded that the crystal class is probably $6/m$ and the space group is $P6_3/m$.

The X-ray diffraction data of a powdered sample (Table 2) were measured on a calibrated Norelco diffractometer at $\frac{1}{4}°\,2\theta$ per minute using filtered copper radiation. A least-squares refinement program of these data on an IBM 7094 computer yielded the lattice constants with e.s.d.'s listed in Table 1.

Table 1. *Crystal data for* $Na_2Ge_4O_9$

$a=11{\cdot}335\pm0{\cdot}001$ Å (25°C)
$c=9{\cdot}697\pm0{\cdot}001$ Å (25°C)
$c/a=0{\cdot}8555$
$V=1245{\cdot}9$ Å$^3$
$D_m=4{\cdot}41\pm0{\cdot}02$ g.cm$^{-3}$ (27°C)
$D_x=4{\cdot}435$ g.cm$^{-3}$
$Z=6$

Table 2. *Diffraction data for* $Na_2Ge_4O_9$

| *hkl* | *d(hkl)* | $10^4 \sin^2\theta_o$ | $10^4 \sin^2\theta_c$ | $I_o$ |
|---|---|---|---|---|
| 110 | 5·66 Å | 185 | 185 | 65 |
| 111 | 4·89 | 249 | 248 | 53 |
| 002 | 4·84 | 254 | 253 | 44 |
| 102 | 4·34 | 316 | 314 | 5 |
| 112 | 3·68 | 439 | 438 | 20 |
| 211 | 3·45 | 498 | 495 | 20 |
| 202 | 3·44 | 501 | 499 | 6 |
| 300 | 3·26 | 559 | 556 | 2 |
| 301 | 3·105 | 615 | 618 | 1 |
| 212 | 2·943 | 685 | 684 | 35 |
| 220 | 2·830 | 740 | 740 | 6 |
| 113 | 2·802 | 755 | 754 | 37 |

Table 2 (*cont.*)

| *hkl* | *d*(*hkl*) | $10^4 \sin^2 \theta_o$ | $10^4 \sin^2 \theta_c$ | $I_o$ |
|---|---|---|---|---|
| 310<br>221 | 2·716 | 804 | 802 | 59 |
| 302 | 2·705 | 810 | 808 | 100 |
| 222 | 2·442 | 994 | 991 | 31 |
| 213 | 2·432 | 1002 | 999 | 33 |
| 004 | 2·421 | 1011 | 1009 | 31 |
| 312 | 2·370 | 1056 | 1053 | 4 |
| 104 | 2·350 | 1074 | 1071 | 24 |
| 320 | 2·235 | 1188 | 1170 | 2 |
| 114 | 2·225 | 1198 | 1194 | 5 |
| 321 | 2·189 | 1238 | 1233 | 9 |
| 204 | 2·172 | 1257 | 1256 | 4 |
| 410 | 2·140 | 1295 | 1293 | 6 |
| 223 | 2·128 | 1310 | 1307 | 13 |
| 313 | 2·080 | 1371 | 1368 | 10 |
| 322 | 2·040 | 1425 | 1422 | 10 |
| 304 | 1·945 | 1568 | 1563 | 4 |
| 323 | 1·846 | 1741 | 1737 | 5 |
| 421<br>502 | 1·818 | 1794 | 1787<br>1791 | 9 |
| 314 | 1·809 | 1813 | 1810 | 5 |
| 413 | 1·784 | 1864 | 1861 | 5 |
| 510 | 1·759 | 1916 | 1908 | 12 |
| 404 | 1·723 | 1998 | 1994 | 5 |
| 215 | 1·716 | 2008 | 2013 | 26 |
| 600 | 1·635 | 2218 | 2216 | 15 |
| 414 | 1.603 | 2309 | 2303 | 9 |
| 315 | 1·577 | 2384 | 2377 | 19 |
| 520 | 1·570 | 2404 | 2401 | 5 |

Numerous additional observed lines less than 1·57 Å.

The density was measured by the double pycnometer method of Collett (1954), using carbon tetrachloride.

No further structural work on this substance is contemplated.

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*Acta Cryst.* (1968). B**24**, 461

## Crystallography of zinc selenite dihydrate.

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(*Received* 27 *November* 1967)

$ZnSeO_3 . 2H_2O$ has $2/m$ symmetry and grows as small and well developed colorless crystals (0·1 – 5 mm) of pseudo-rhomboedral habit. The main observed interfacial angles are: $(110)\wedge(102) = 63°\ 50'$ and $(110)\wedge(1\bar{1}0) = 80°\ 10'$, and the observed optical constants $X = \alpha = 1·660$, $Y = \beta = 1·710$, $Z = \gamma = 1·750$; $(\gamma - \alpha) = 0·090$ and $2V_{\rm calc} = 82°$. The unit cell parameters determined in the precession photographs and refined by the powder method are $a_0 = 7·68$, $b_0 = 8·80$, $c_0 = 6·49$ Å, $\beta = 81°\ 34'$ and $a_0:b_0:c_0 = 0·87:1:0·74$. Space group $P2_1/n$. The observed specific gravity 3·52 $g.cm^{-3}$ suggests 4 formulae per unit cell.

Selenites of several metals, such as Ni, Co, Mn, Cu and Zn, have been recently prepared by the Chemistry Department of the University of São Paulo, Brazil, and later investigated from the crystallographic point of view by various authors.

$ZnSeO_3.2H_2O$ precipitates as monoclinic crystals, with $2/m$ symmetry, the individuals being approximately equidimensional and of size of the order of a millimetre, resembling a pseudo-rhombohedral habit. Some fibrous radiated aggregates may however be formed occasionally. Most of the crystals are colourless, although a few may exhibit a white colour.

The crystal morphology is very simple, showing only the two crystallographic forms {110} and {102}, as determined by the following interfacial angles measured in the two-circle goniometer:

$$(110)\wedge(102) = 63°50'$$
$$(110)\wedge(1\bar{1}0) = 80°10' .$$

The axial ratio $a_0:b_0:c_0 = 0·87:1:0·74$, has been calculated from the unit-cell dimensions obtained by X-ray diffraction. The compound is biaxial (–), $2V = 82°$, and has the following indices of refraction:

$$X = \alpha = 1·660 \pm 0·005$$
$$Y = \beta = 1·710 \pm 0·005$$
$$Z = \gamma = 1·750 \pm 0·005 .$$

The unit cell has been determined by precession methods with Mo $K\alpha$, by using photographs of the reciprocal level $h0l$ and $0kl$. The parameters have been refined by the powder method with Cu $K\alpha$, for greater accuracy, giving

$$a_0 = 7·68, \quad b_0 = 8·80, \quad c_0 = 6·49 \text{ Å}, \quad \beta = 81°34' .$$

The main reflexions of the powder diagram are listed in Table 1.

Table 1. *Interplanar spacings for* $ZnSeO_3.2H_2O$

| *hkl* | $d_{\rm calc}$ | $d_{\rm obs}$ | $I_{\rm rel}$ |
|---|---|---|---|
| 110 | 5·744 Å | 5·754 Å | 10 |
| $10\bar{1}$ | 5·314 | 5·322 | 2 |
| $11\bar{1}$, 101 | 4·545 | 4·540 | 2 |
| 020 | 4·402 | 4·393 | 2 |
| 111 | 4·061 | 4·047 | 5 |
| 200, 120 | 3·795 | 3·795 | 5 |
| 210 | 3·485 | 3·480 | 6 |
| $21\bar{1}$ | 3·256 | 3·253 | 2 |