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Berichtigung zu den Kristalldaten von Stickstoffselenid $(NSe)_x$.

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In einer vorläufigen Mitteilung (1961) haben wir die Elementarzelle von Stickstoffselenid $(NSe)_x$ triklin beschrieben mit den Gitterkonstanten

$$a_0 = 6{,}47,\ b_0 = 6{,}85,\ c_0 = 6{,}85\ \text{Å};$$
$$\alpha_0 = 90{,}5°,\ \beta_0 = 100{,}4°,\ \gamma_0 = 100{,}4°.$$

Bei der Auswertung der Reflexintensitäten zeigte sich aber, dass Stickstoffselenid monokline Symmetrie besitzt. Dementsprechend ist die numerische Übereinstimmung von b_0 und c_0 sowie von β_0 und γ_0 kein Zufall, sondern folgt zwangsläufig aus der höheren Symmetrie des Gitters.

Die Transformation von der ursprünglichen Aufstellung in die monokline Elementarzelle, deren Fläche (001) zentriert ist, wird durch die Vektorgleichungen

$$\mathbf{a} = \mathbf{b}_0 + \mathbf{c}_0,\quad \mathbf{b} = -\mathbf{b}_0 + \mathbf{c}_0,\quad \mathbf{c} = \mathbf{a}_0$$

beschrieben. Für die monoklinen Gitterkonstanten leiten sich daraus folgende Werte ab:

$$a = 2b_0 \cos \tfrac{1}{2}\alpha_0 = 9{,}65 \pm 0{,}02,\ b = 2b_0 \sin \tfrac{1}{2}\alpha_0 = 9{,}73 \pm 0{,}02,$$
$$c = a_0 = 6{,}47 \pm 0{,}01\ \text{Å};$$
$$\cos \beta = \cos \beta_0 / \cos \tfrac{1}{2}\alpha_0;\ \beta = 104{,}9 \pm 0{,}1°.$$

Gegenüber der primitiven triklinen Zelle ist das Volumen der monoklin flächenzentrierten Elementarzelle verdoppelt ($V = 586$ Å^3) und enthält 16 Formeleinheiten (NSe), wahrscheinlich in Form von 4 Molekülen 'N_4Se_4'. Als Raumgruppen kommen *Cc* und *C*2/*c* in Frage, da die Indizesstatistik neben der durch die Flächenzentrierung bedingten integralen Auslöschung aller Interferenzen (*hkl*) mit $h + k = 2n + 1$ noch die zonale Auslöschung der Reflexe (*h*0*l*) mit $l = 2n + 1$ aufweist.

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Preliminary observations of mixed oxide compounds containing BeO.

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In the course of a survey of phase formation in mixed oxide systems containing BeO, the compounds 2CaO.3BeO, 2SrO.3BeO, and Y_2O_3.2BeO have been synthesized and studied by optical and single-crystal X-ray diffraction methods. Optical and powder diffraction data, in agreement with the results presented here, have been reported previously for the calcium compound (Ader & Bingle, 1956) and for the yttrium compound (Weir & Van Valkenburg, 1960).

Table 1. *Optical and X-ray diffraction data for compounds*

	2 CaO.3 BeO	2 SrO.3 BeO	Y_2O_3.2 BeO
Optical data			
Morphology	Equiaxial blocks	Needle-like laths or fibers; laths Needle axis $\parallel$ [001]	$L_1 \parallel [100] \geqslant$ $L_2 \parallel [011] \geqslant$ $L_3 \parallel [\,?\,]$
Appearance in polarized light	Isotropic	Birefringent	Birefringent
Indices of refraction	$n = 1{\cdot}638 \pm 0{\cdot}003$	$n \parallel [001] = 1{\cdot}724 \pm 0{\cdot}003$ (probably n_γ) $n \perp [001]$ (average) $= 1{\cdot}709 \pm 0{\cdot}003$	$n \parallel [100] = 1{\cdot}855 \pm 0{\cdot}005$ $n \parallel [011] = 1{\cdot}840 \pm 0{\cdot}005$
$2V$(est.)	—	$\gtrsim 0°$ (biaxial)	$\sim 90°$ (biaxial)
X-ray diffraction data			
Crystal system	Cubic	Orthorhombic	Orthorhombic
Lattice parameters	$a_0 = 14{\cdot}00 \pm 0{\cdot}01$ Å	$a_0 = 7{\cdot}13 \pm 0{\cdot}01$ Å $b_0 = 9{\cdot}01 \pm 0{\cdot}01$ Å $c_0 = 18{\cdot}5 \pm 0{\cdot}1$ Å (See text)	$a_0 = 3{\cdot}51 \pm 0{\cdot}01$ Å $b_0 = 9{\cdot}88 \pm 0{\cdot}01$ Å $c_0 = 10{\cdot}36 \pm 0{\cdot}02$ Å
Test for pyroelectricity	Negative	Negative	Negative
Probable space group	$Fm3m$, O_h^5	?	$Pmcn$, D_{2h}^{16}
Z (form. wt./cell)	24	10	4
ϱ_0 (g.cm.$^{-3}$)	$2{\cdot}70 \pm 0{\cdot}10$	$3{\cdot}84 \pm 0{\cdot}05$	$5{\cdot}1 \pm 0{\cdot}02$
$\varrho_{\text{X-ray}}$ (g.cm.$^{-3}$)	$2{\cdot}71 \pm 0{\cdot}01$	$3{\cdot}94 \pm 0{\cdot}04$	$5{\cdot}10 \pm 0{\cdot}03$

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* Operated for the U.S. Atomic Energy Commission by the Union Carbide Corporation.

Single-phase samples of each of these compounds were prepared by melting ($T_m = 1300$–1600 °C.) and quenching mechanical mixtures of stoichiometric weights of the appropriate oxide powders. Atmospheres of argon or air were employed, the final phases in either case being identical. Compound compositions were verified by agreement in weight of initial oxides and final product and by chemical analysis for metallic elements.

The conditions of preparation precluded any statements concerning the equilibrium or non-equilibrium character of the compounds formed. Evidence indicated that at least one compound, 2CaO.3BaO, may be metastable at all temperatures, though the rate of dissociation at room temperature was unobservably slow. Phase diagrams for the oxide systems will be considered in a separate communication (Potter & Harris, 1961).

Standard optical and X-ray diffraction measurements were made on single crystals obtained from quenched melts. Cu $K\alpha$ ($\lambda = 1\cdot5418$ Å) and Mo $K\alpha$ ($\lambda = 0\cdot7107$ Å) radiations were used in the diffraction experiments. Crystal densities were estimated by a liquid-displacement technique and tests for pyroelectricity were made by a liquid air method (Wooster, 1957). A summary of the data is given in Table 1.

The diffraction results for 2SrO.3BeO deserve special comment. A rotation photograph taken about the needle (**c**) axis of a crystal of this compound displayed layer lines which may be characterized as follows:

1. An equatorial layer that consisted of sharp reflections only. These reflections corresponded to a rectangular real lattice net with $a_0 = 7\cdot13$ Å and $b_0 = 9\cdot01$ Å.
2. Layers of diffuse reflections which corresponded to a c_0 translation period of 18·5 Å. There was a symmetrical displacement of reflections on these diffuse layers to higher and lower h values about reciprocal lattice points (h_0, k_0) corresponding to the real lattice net described above. The 5th, 10th, and 15th, etc. layers of diffuse reflections also contained sharp reflections at the points (h_0, k_0).
3. Layers of reflections, sharp in **a*** and **b*** directions, and elongated in the **c*** direction. The translation periods of these layers were not simple multiples or fractions of 18·5 Å, but the reflections on them did correspond to the rectangular real lattice net described above.

The presence of only sharp reflections in the equatorial layer suggests that the structure of this compound would appear perfectly periodic if it were projected on the *ab* plane. The appearance of the non-equatorial layers would seem to indicate disorder in the **c** direction of the lattice, but a complete description of this disorder has not been deduced.

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Confirmation of the crystal structure of 2-amino-ethanol phosphate.

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In a recent paper, Kraut (1961) published the crystal structure of 2-amino-ethanol phosphate. Simultaneously and independently the structure was investigated and solved by the authors using two-dimensional Patterson maps and the Harker–Kasper inequalities (1948). Initial refinement was carried out by successive two-dimensional Fourier maps. Further refinement was obtained using the DEUCE computer of the University of Glasgow and the three-dimensional least squares program written by Rollett (1960). This was discontinued when Kraut's structure was published. At this stage the overall reliability factor

$$R = \Sigma|F_o - F_c|/\Sigma|F_o| = 0\cdot155$$

for 800 observed reflections.

The positional parameters and standard deviations are shown in Table 1, which, for ease of comparison, is identical in form to that given by Kraut. These results, although comparatively unrefined, confirm Kraut's analysis.

Table 1. *Positional parameters for the heavy atoms and their estimated standard deviations*

	x/a	$\sigma(x/a)$	y/b	$\sigma(y/b)$	z/c	$\sigma(z/c)$
P1	0·2344	0·0003	0·0226	0·0002	0·1103	0·0003
O2	0·3695	0·0008	−0·0769	0·0007	0·1889	0·0010
O3	0·1682	0·0008	−0·0127	0·0008	−0·0556	0·0010
O4	0·1082	0·0009	−0·0014	0·0010	0·2046	0·0009
O5	0·2882	0·0009	0·2194	0·0006	0·1288	0·0009
C6	0·1826	0·0014	0·3513	0·0010	0·0680	0·0016
C7	0·2635	0·0013	0·5243	0·0010	0·0736	0·0016
N8	0·3469	0·0010	0·5714	0·0007	0·2285	0·0011

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