

isomorphism with the  $\text{Cu}_{15}\text{Si}_4\text{-D8}_6$  structure (Morrall & Westgren, 1934). Based on this stoichiometry the crystallographic density is  $2.13 \text{ g.cm}^{-3}$ .

The intensities of 61 reflections were measured with  $\text{Mo K}\alpha$  radiation ( $\lambda=0.7107 \text{ \AA}$ ) on a G.E. XRD 5 diffractometer equipped with a single-crystal orienter. A spherical absorption correction was applied to the data ( $\mu R=0.75$ ). The structure was refined by the full-matrix least-squares program ACA No. 317 (Gantzel, Sparks & Trueblood, unpublished) with the use of a weighting scheme designed to take counting statistics into account. The observed and calculated structure factors along with  $\sigma(F_o)$  are shown in Table 1 for which a final reliability index of 4.1% including missing reflections was obtained. Further least-squares refinements result in no change in atomic parameters. The atomic parameters are:

16 Ge (c)	$x = 0.2084 \pm 0.0002$	$B = 1.48 \pm 0.10 \text{ \AA}^2$
12 Li(1) (a)		$B = 0.9 \pm 1.2$
48 Li(2) (e)	$x = 0.129 \pm 0.004$	$B = 2.9 \pm 0.6$
	$y = 0.154 \pm 0.003$	
	$z = -0.040 \pm 0.004$	

The interatomic distances less than  $3.1 \text{ \AA}$  are shown in Table 2. These have been computed with  $a=10.783 \text{ \AA}$ .

The compounds which have so far been found to have the  $\text{D8}_6$  structure are given in Table 3.

Table 2. *Interatomic distances for  $\text{Li}_{15}\text{Ge}_4$*

Ge-3 Li(1)	$2.912 \pm 0.001 \text{ \AA}$
-3 Li(2)	$2.60 \pm 0.04$
-3 Li(2)	$2.80 \pm 0.04$
-3 Li(2)	$2.87 \pm 0.04$
Li(1)-4 Ge	$2.912 \pm 0.001$
-4 Li(2)	$2.75 \pm 0.04$
-4 Li(2)	$2.81 \pm 0.04$
Li(2)-Ge	$2.60 \pm 0.04$
-Ge	$2.80 \pm 0.04$
-Ge	$2.87 \pm 0.04$
-Li(1)	$2.75 \pm 0.04$
-Li(1)	$2.81 \pm 0.04$
-Li(2)	$2.75 \pm 0.08$
-2 Li(2)	$2.78 \pm 0.07$
-2 Li(2)	$3.07 \pm 0.06$

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**Crystal data for the double salt, lithium ammonium hexafluorosilicate.** By REUBEN RUDMAN, *Polytechnic Institute of Brooklyn, Brooklyn 1, N. Y., U.S.A.* and JOHN A. SKARULIS, *Department of Chemistry, St. Johns University, Jamaica 32, N. Y., U.S.A.*

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The hitherto unreported double salt, lithium ammonium hexafluorosilicate,  $\text{LiNH}_4\text{SiF}_6$ , was discovered during a systematic study of the dissociation pressures and solubilities of the  $\text{Li}_2\text{SiF}_6\text{-(NH}_4)_2\text{SiF}_6\text{-H}_2\text{O}$  system at  $25^\circ\text{C}$ . Single crystals of  $\text{LiNH}_4\text{SiF}_6$  were prepared by slow precipitation from aqueous solution by addition of ethanol. The results of these studies as well as the experimental details of the growth of single crystals of this compound are described elsewhere. (Skarulis, Darnowski, Kilroy & Milazzo, 1964.)

Table 3. *Compounds with the  $\text{D8}_6$  structure*

Compound	Cell constant	$r_a/r_b$	
$\text{Cu}_{15}\text{Si}_4$	9.71 $\text{\AA}$	0.97	Morrall & Westgren (1934)
$\text{Na}_{15}\text{Pb}_4$	13.32	1.09	Zintl & Harder (1936)
$\text{Li}_{15}\text{Ge}_4$	10.783	1.14	Gladyshevskii & Kripyakevich (1960); this work

The radius ratios have been computed with values from the compilation of Teatum, Gschneidner & Waber (1959). In addition to the above compounds, Pearson (1958) suggests that the compound ' $\text{Cu}_3\text{As}$ ' studied by Steenberg (1936-1938) is also of the  $\text{D8}_6$  type. The radius ratio of 0.92 for this compound is similar to the above ratios.

Although the evidence appears to favor an isomorphous  $\text{Li}_{15}\text{Sn}_4$  ( $r_a/r_b=1.01$ ), we have not observed such a compound in preparations near this composition.  $\text{Na}_{15}\text{Sn}_4$  ( $r_a/r_b=1.24$ ) is not isomorphous (Zintl & Harder, 1936). This appears to establish an upper limit to the radius ratio for this structure type.

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#### References

- GLADYSHEVSKII, E. L. & KRIPYAKEVICH, P. I. (1960). *Kristallografiya*, **5**, 574.
- International Tables for X-ray Crystallography* (1952). Vol. I. Birmingham: Kynoch Press.
- MORRAL, F. R. & WESTGREN, A. (1934). *Ark. Kemi Min. Geol.* **11B**, No. 37.
- PEARSON, W. B. (1958). *Handbook of Lattice Spacings and Structures of Metals and Alloys*, p. 395. New York: Pergamon Press.
- STENBERG, B. (1936-1938). *Ark. Kemi Min. Geol.* **12A**, No. 26.
- TEATUM, E., GSCHNEIDNER, K., JR. & WABER, J. (1959). Los Alamos Scientific Laboratory, Report LA-2345.
- ZALKIN, A. & RAMSEY, W. J. (1958). *J. Phys. Chem.* **62**, 689.
- ZINTL, E. & HARDER, A. (1936). *Z. Phys. Chem.* **B34**, 238.

Chemical analysis of the crystals confirmed the existence of  $\text{LiNH}_4\text{SiF}_6$ . The density was determined by the use of a pycnometer with benzene as the displacing liquid. Rotation and Weissenberg photographs were obtained with Ni-filtered  $\text{Cu K}$  radiation and precession photographs were obtained with Zr-filtered  $\text{Mo K}$  radiation.  $\text{LiNH}_4\text{SiF}_6$  was found to be pseudo-orthorhombic, actually monoclinic. All of the crystals which were examined (by optical means as well as by X-ray diffraction) were twinned, a tendency of crystals pos-

sessing a monoclinic, pseudo-orthorhombic structure (Dunitz, 1964). The cell dimensions were refined with the aid of powder data obtained on a Norelco diffractometer using Ni-filtered Cu K radiation.

The crystal data are as follows:

System: Monoclinic. Space group:  $P2_1/c$ .

$a = 5.633 \pm 0.005$ ,  $b = 9.679 \pm 0.009$ ,  $c = 8.740 \pm 0.008$  Å;  
 $\beta = 90.0^\circ$ ,  $Z = 4$ ,  $V = 476.3$  Å<sup>3</sup>.

$D_x = 2.330$  g.cm<sup>-3</sup>,  $D_m = 2.317 \pm 0.002$  g.cm<sup>-3</sup>.

The systematic absences were  $h0l$  with  $l$  odd and  $0k0$  with  $k$  odd. The crystals grew in the shape of flat platelets. These platelets were formed with the  $b$  and  $c$  axes along the face diagonals and the  $a$  axis perpendicular to the face. A number of crystals in the shape of rain-drops were also formed. These were examined and found to be of the same phase as the platelets.

The crystal structure of lithium ammonium hexafluorosilicate is related to that of ammonium hexafluorosilicate (Gossner & Kraus, 1934).  $(\text{NH}_4)_2\text{SiF}_6$  is hexagonal with  $a = 5.76$ ,  $c = 4.77$  Å. The following table shows the relationship between the pseudo-orthorhombic cell ob-

tained from this hexagonal unit cell (allowing for doubling of the  $c$ -axis) and the unit cell of  $\text{LiNH}_4\text{SiF}_6$ :

Cell constant	$a$ (Å)	$b$ (Å)	$c$ (Å)
$(\text{NH}_4)_2\text{SiF}_6$	5.76	9.54	9.98
$\text{LiNH}_4\text{SiF}_6$	5.633	9.679	8.740

It can be concluded that the structure of  $\text{LiNH}_4\text{SiF}_6$  is a distorted  $(\text{NH}_4)_2\text{SiF}_6$  structure. This is due to the fact that the lithium ion is considerably smaller than the ammonium ion.

The powder data have been submitted for inclusion in the ASTM X-Ray Powder Data File.

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#### References

- DUNITZ, J. D. (1964). *Acta Cryst.* **17**, 1299.  
 GOSSNER, E. & KRAUS, O. (1934). *Z. Kristallogr.* **88**, 223.  
 SKARULIS, J. A., DARNOWSKI, V. N., KILROY, W. P. & MILAZZO, T. (1964). *J. Phys. Chem.* **68**, 3074.

## Notes and News

*Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the General Secretary of the International Union of Crystallography (D. W. Smits, Rekencentrum der Rijksuniversiteit, Grote Appelstraat 11, Groningen, The Netherlands). Publication of an item in a particular issue cannot be guaranteed unless the draft is received 8 weeks before the date of publication.*

### Non-Conventional Electron Microscopy

The Electron Microscopy and Analysis Group of The Institute of Physics and The Physical Society is organizing a Conference on Non-Conventional Electron Microscopy, to be held at the University of Cambridge from 31 March to 2 April 1965. The conference is intended to cover all methods that are not catered for by the normal commercially available transmission electron microscopes. Offers of contributions should be sent to the Conference Secretary, Dr W. C. Nixon, Engineering Laboratory, The University, Cambridge, England, before 12 February 1965.

Advance registration for the Conference is necessary, and application forms may be obtained from the Administration Assistant, The Institute of Physics and The Physical Society, 47 Belgrave Square, London, S.W. 1, England.

### International Conference on X-ray Optics and Microanalysis

The Fourth International Conference on X-ray Optics and Microanalysis will be held near Paris on 8, 9 and 10 September 1965. The scope of the conference will include applications to metallurgy, mineralogy and biology, as well as basic advances in microscopy, microanalysis, and long-wavelength spectroscopy.

Further information may be obtained from M. P. Deschamps, Département de Physique, Institut de Recherches de la Sidérurgie Française, Saint-Germain-en-Laye (S. et O.), France.