to establish the configuration of the six-membered ring in this compound.

The dioxime crystals belong to the orthorhombic system and the space group is *Pbca*. The unit cell containing 8 molecules has the following parameters:

$$a = 7.14_1 \text{ Å}, b = 13.25_1 \text{ Å}, c = 14.72_4 \text{ Å}$$

Of the 87 observed $0kl$-reflections, signs were determined for 67 using a computer procedure based on the Cochran-Douglas method [5] (programmed in FORTAN IV for UNIVAC 1107 by the author). Since 65 of these signs turned out to be correct, the corresponding Fourier map is practically identical with the final one, which

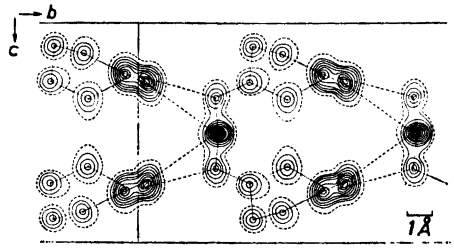

Fig. 1. Fourier projection along a-axis.

is shown in Fig. 1. The R-value thus arrived at for this projection is R_{0kl} = 7.4 %.

Using the known z-parameters, approximate values for the x-coordinates were found by allowing the $h0l$-projection of the molecule (determined from a model) to move in small steps parallel to the x-axis. The R-factors were calculated for each step, and the parameters corresponding to the minimum R-value were chosen as starting coordinates in a three-dimensional full matrix least squares refinement based on about 1300 reflections of which 990 are observed. The final R-value including all the observed reflections is R = 5.6 %. At the present stage the following results may be stated with confidence:

The carbon skeleton of the molecule corresponds to that of a "twisted boat" form, with interatomic distances and angles not very far from those found for the dione itself. The angle between the two C—N bonds is, however, about 26° smaller than the corresponding angle between the C—O bonds of the dione.

The two independent C—N bonds are, respectively, 1.27_4 Å and 1.27_6 Å, while the two N—O bonds are 1.41_1 Å and 1.41_2 Å.

The hydrogen bonds link each dioxime molecule to four others, the two independent bonds having the lengths 2.77_1 Å and 2.83_0 Å. The hydrogen atoms in these bonds have been localized from a three-dimensional difference synthesis and found to be situated at normal distances from the oxygen atoms, and approximately on the straight line joining O and N.

Further details of the structure will be given later.

1. Groth, P. and Hassel, O. *Acta Chem. Scand.* **18** (1964) 923.
2. Mossel, A. *Diss.* Leiden 1963; Mossel, A. and Romers, C. *Acta Cryst.* **17** (1964) 1217.
3. Groth, P. and Hassel, O. *Acta Chem. Scand.* **19** (1965) 1733.
4. Groth, P. and Hassel, O. *Acta Chem. Scand.* **18** (1964) 1327.
5. Cochran, W. and Douglas, A. S. *Proc. Roy. Soc. (London)* **227** (1954) 486; **243** (1958) 281.

Received December 22, 1965.

A New Compound in the System Sb_2O_3-Nb_2O_5

A. C. SKAPSKI*

Institute of Inorganic and Physical Chemistry, University of Stockholm, Stockholm, Sweden

Hitherto the only known compound in the system Sb_2O_3—Nb_2O_5 has been $SbNbO_4$,[1] which is orthorhombic, a = 5.561, b = 4.939, c = 11.810 Å, and is isostructural with the mineral stibiotantalite. The crystal structure of $SbNbO_4$ has recently been revised.[2,3]

During attempts to prepare single crystals of pure $SbNbO_4$, equimolar mixtures of antimony trioxide and niobium pentoxide were heated for 24 h in sealed platinum capsules. At 1000°C, $SbNbO_4$ was formed, whereas at 1200°C the specimen melted and was lost from the platinum tube; both these observations are in agree-

* Present address: Department of Chemistry, Imperial College, London S. W. 7, England.

Table 1. X-Ray powder data for $Sb_{0.67}Nb_2O_6$. Cu$K\alpha_1$ radiation.

d(Å)	I	$\sin^2\theta_{obs}$	hkl	$\sin^2\theta_{calc}$
12.42	m	0.00387	101	0.00391
8.65	m	0.00793	002	0.00800
6.17	vw	0.01559	202	0.01563
5.46	w	0.01988	103	0.01992
4.412	m	0.03047	400	0.03052
4.110	m	0.03513	303	0.03518
3.971	s	0.03763	010	0.03768
3.927	m	0.03847	402	0.03853
3.873	ms	0.03956	204	0.03965
3.781	vw	0.04151	111	0.04159
3.621	mw	0.04526	210	0.04531
3.609	vw	0.04555	012	0.04568
3.542	w	0.04729	211	0.04731
3.456	m	0.04968	501	0.04969
3.380	s	0.05193	105	0.05193
3.292	w	0.05475	310	0.05485
3.211	m	0.05754	113	0.05759
3.077	m	0.06266	404	0.06254
3.063	w	0.06323	213	0.06332
3.004	s	0.06573	503	0.06570
2.971	ms	0.06722	305	0.06719
2.950	m	0.06817	410	0.06820
2.941	m	0.06861	600	0.06868
2.907	w	0.07020	411	0.07020
2.870	mw	0.07203	006	0.07204
2.856	m	0.07275	313	0.07285
2.790	mw	0.07623	412	0.07620
2.782	m	0.07666	602	0.07668
2.771	w	0.07726	214	0.07732
2.730	w	0.07962	206	0.07967
2.622	w	0.08627	413	0.08621
2.614	w	0.08683	314	0.08686
2.605	m	0.08741	511	0.08737
2.573	m	0.08961	115	0.08961
2.520	vvw	0.09341	512	0.09337
2.495	mw	0.09533	215	0.09533
			701	0.09548
2.426	mw	0.10078	604	0.10069
2.405	vvw	0.10260	406	0.10256
2.396	mw	0.10336	513	0.10338
2.378	ms	0.10492	315	0.10487

s = strong m = medium w = weak v = very

ment with those of Roth and Waring.[4] At 1120°C, however, the product consisted of an $SbNbO_4$ solid solution and an unknown phase. A study at 1120°C of mixtures richer in niobium showed that a single phase is obtained at the composition $Sb_2O_3{\cdot}3Nb_2O_5$. The product consists of pale yellow, elongated crystals which are very often twinned.

The crystals are orthorhombic, with $a = 17.635$, $b = 3.968$, $c = 17.219$ Å, $V = 1205.0$ Å^3. The unit-cell dimensions bear a close resemblance to those of ferroelectric $PbNb_2O_6$, although single-crystal photographs show no trace of a doubling of the short axis which is observed for $PbNb_2O_6$.[5] In view of the apparent similarity, the formula of the new compound may be written as $Sb_{0.67}Nb_2O_6$, with $Z = 10$.

X-Ray powder data, taken with a Guinier focusing camera using strictly monochromatised Cu$K\alpha_1$ radiation and potassium chloride as an internal standard, are given in Table 1.

Further work is in progress on this compound, including a three-dimensional structure determination and a study of its dielectric properties.

Acknowledgements. Thanks are due to Dr. Sten Andersson for discussions. This work has been supported by the *Swedish Natural Science Research Council.*

1. Keller, C. *Z. anorg. Chem.* **318** (1962) 89.
2. Dihlström, K. *Z. anorg. Chem.* **239** (1938) 57.
3. Skapski, A. C. and Rogers, D. *Chem. Comm.* **1965** 611.
4. Roth, R. S. and Waring, J. L. *Am. Mineralogist* **48** (1963) 1348.
5. Francombe, M. H. and Lewis, B. *Acta Cryst.* **11** (1958) 696.

Received January 19, 1966.