for C4H7ONS: C 40.98; H 6.02; N 11.95). The compound migrated on paper chromatograms at a rate definitely lower than that of a reference sample of (±).5-methyl-2-oxazolidinethione (V) 7,8 (Fig. 1); it appears likely that the isomeride (VI) 9 will not differ markedly from (V) in its chromatographic behaviour. Synthetic experiments are now in progress to verify the structure (VII).

The possible existence in Malcolmia seeds of an additional glucoside, containing (B) as the aglucone and located at the site of glucocheirolin in the paper chromatogram, has been ruled out by chromatography on paper of the free isothiocyanates in chloroform (spray reagent: ammoniacal silver nitrate). Only cheirolin and (VII) were detectable, both travelling near the solvent front, whereas no trace of the expected ring compound (IV) was observed. The present results explain the appearance of two spots in the low  $R_F$ -region of the paper chromatogram of Malcolmia-thioureas, reported by Schultz and Wagner 1. One is attributable to cheirolinthiourea (A) whereas the other spot probably represents the artefact (B). In the present work it has been further established that our previously recorded occurrence of methyl isothiocyanate in Malcolmia seeds 10 is incorrect.

The presence of glucose and sulphate in glucomalcolmiin has been verified by paperchromatographic methods. The glucoside thence appears to be of the usual type. Unpublished results from this laboratory, supplemented by the conclusive work of Ettlinger and Lundeen 11 on the revised mustard oil glucoside structure, favour the following expression for the glucomalcolminate ion:

A detailed account of the present work will appear in this journal.

Microanalyses by Mr. P. Hansen of this laboratory. The work is part of investigations supported by Statens Almindelige Videnskabsfond (The Danish State Research Foundation) and Carlsbergfondet (The Carlsberg Foundation).

- 1. Schultz, O.-E. and Wagner, W. Z. Natur-
- forsch. 11b (1956) 73.
  2. Schneider, W. Ann. 375 (1910) 207.
  3. Kjær, A., Marcus, F. and Conti, J. Acta Chem. Scand. 7 (1953) 1370.
- 4. Kjær, A. and Rubinstein, K. Acta Chem. Scand. 7 (1953) 528.
- 5. Kjær, A., Gmelin, R. and Boe Jensen, R. Acta Chem. Scand. 10 (1956) 432.
- 6. Hodgkins, J. E. and Ettlinger, M. G.
- J. Org. Chem. 21 (1956) 404. 7. Hopkins, C. Y. Can. J. Research 20B (1942) 268.
- 8. Rosen, A. A. J. Am. Chem. Soc. 74 (1952)
- 9. Viscontini, M. and Adank, K. Helv. Chim. Acta 33 (1950) 225.
- Kjær, A., Conti, J. and Larsen, I. Acta Chem. Scand. 7 (1953) 1276.
- 11. Ettlinger, M. G. and Lundeen, A. J. J. Am. Chem. Soc. 78 (1956) 4172.

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## A New Modification of Titanium Monoxide

ULF KUYLENSTIERNA and ARNE MAGNÉLI

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

According to Ehrlich 1, titanium mon-oxide has a structure of sodium chloride type with randomly distributed vacancies in both the titanium and oxygen frameworks and a very broad range of homogeneity. Samples of this phase were prepared by melting mixtures of titanium metal and titanium dioxide (both of high purity) under argon atmosphere in an electric arc furnace. The following data, which are in fair agreement with Ehrlich's results, were obtained for a few of the compositions studied.

The unit cell dimensions were obtained from powder photographs taken in a Guinier focusing camera with strictly monochromatized CuKa, radiation using potassium chloride as an internal standard.

The arc-melted samples were sealed in evacuated silica tubes, heated at 800° C for several days and quenched in water. The Guinier photographs of the preparations  $TiO_x$  (x < 1) then showed the pattern

Composition	Axial length	Density		of available occupied by oxygen atoms
TiO <sub>0.64</sub> (phase limit)	4.202 Å	5.00	96	61
TiO <sub>1.00</sub>	4.182 Å	4.95	85	85
TiO <sub>1.26</sub> (phase limit)	4.169 Å	4.78	77	97

of the hexagonal a-titanium phase and a great number of extra lines. The latter also appeared in the photographs of samples  $\mathrm{TiO}_x$  (1 < x < 1.12) in addition to a pattern corresponding to titanium monoxide of sodium chloride type of the approximate composition  $\mathrm{TiO}_{1.15}$ . The new phase, the lines of which showed no detectable displacements in samples of different compositions, was pure in the sample  $\mathrm{TiO}_{1.99}$  and is obviously a new modification of titanium monoxide possessing a very narrow range of homogeneity. The density was found to be 4.91.

Table 1. Part of Guinier powder photograph of TiO<sub>1.00</sub>, prepared at 800° C. CuKa<sub>1</sub> radiation.

I	$\sin^2\Theta_{ m obs}$	
w	0.0191	
w	0.0300	
w	0.0347	
m	0.0421	
st .	0.0539	
m	0.0683	
m	0.0761	
m	0.0772	
w	0.0995	
st	0.1018	
st	0.1038	
st	0.1348	
v st	0.1386	
v w	0.1574	
v w	0.1681	
v w	0.1916	
w	0.2047	
v w	0.2084	
w	0.2154	
w	0.2212	
w	0.2259	
v w	0.2346	
v v w	0.2447	
w	0.2578	
m	0.2689	
st	0.2729	
v st	0.2767	
v v w	0.3184	
v v w	0.3302	

The powder pattern of the new phase, listed in Table 1, is of a rather complicated appearance and probably of low symmetry. So far it has not been possible to index it. However, it shows a noticeable similarity to that of the monoxide of sodium chloride type. Thus the strong reflexions of the former appear as doublets close to those of the latter, i. e. st ( $\sin^2\Theta = 0.1017$ ), v st (0.1358), and v st (0.2714). The similarity seems to indicate the existence of a structural relationship between the two phases. It might be that the structural transformation is associated with an ordering of the atomic vacancies present in the cubic phase.

These studies form part of a research program on metal oxides and related compounds financially supported by the Swedish Natural Science Research Council. Further studies on the titanium-oxygen system are in progress. A detailed report of the investigation will be published elsewhere.

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## The Synthesis of 6-Methoxy-2(3)benzoxazolinone

PENTTI K. HIETALA and ORN WAHLROOS

Laboratory of the Foundation for Chemical Research, Biochemical Institute, Helsinki, Finland

In this laboratory an anti-fungal substance was recently isolated from maize and wheat plants <sup>1</sup>. The structure 6-methoxy-2(3)-benzoxazolinone (IV) was proposed for this substance. This has now been confirmed by the following synthesis:

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