

Composition	Axial length	Density	Per cent of available atomic sites occupied by	
			titanium atoms	oxygen atoms
TiO <sub>0.84</sub> (phase limit)	4.202 Å	5.00	96	61
TiO <sub>1.00</sub>	4.182 Å	4.95	85	85
TiO <sub>1.86</sub> (phase limit)	4.169 Å	4.78	77	97

of the hexagonal  $\alpha$ -titanium phase and a great number of extra lines. The latter also appeared in the photographs of samples TiO<sub>x</sub> ( $1 < x < 1.12$ ) in addition to a pattern corresponding to titanium monoxide of sodium chloride type of the approximate composition TiO<sub>1.16</sub>. The new phase, the lines of which showed no detectable displacements in samples of different compositions, was pure in the sample TiO<sub>1.00</sub> 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. CuK $\alpha_1$  radiation.

I	sin <sup>2</sup> $\Theta$ <sub>obs</sub>
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.

1. Ehrlich, P. Z. *Elektrochem.* 45 (1939) 362.

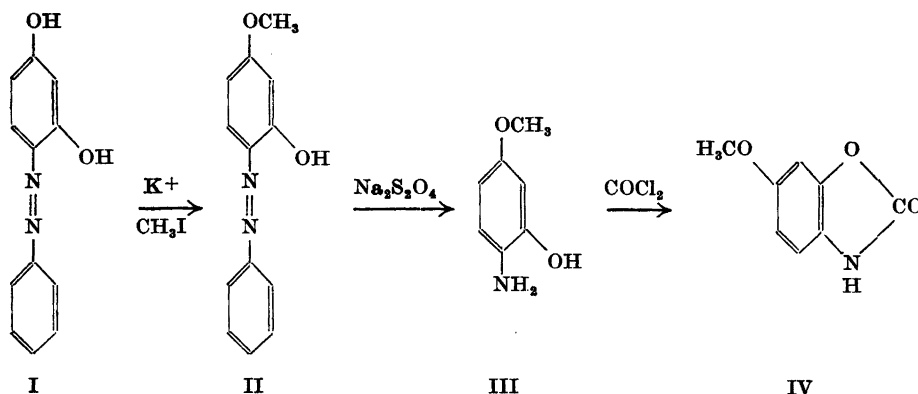
Received September 22, 1956.

## 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:



Pure *p*-benzene-azo-resorcine (I) was obtained by the method of Will and Pukall<sup>2</sup>.

The methylation was done by the method of Bechold<sup>3</sup>, with the following modifications: a) methyl iodide was used in the proportion 2 moles to 1 mole of the dry potassium salt of I; b) the reaction time was reduced from 6 h to 3 h (yield: 6 h, 1.5 %; 3 h, 14 % theor.).

2-Hydroxy-4-methoxy-aniline (III) is very easily oxidized wherefore the following stages were carried out in a carbon dioxide atmosphere: a) II was reduced in a boiling alcohol solution with a slight excess of a saturated solution of sodium hyposulphite in water<sup>4</sup>, the mixture was neutralized with barium carbonate and evaporated almost to dryness (the aniline is removed with the vapour); b) III was extracted from the remaining mixture by peroxide-free ether and after evaporation of the ether it was mixed with glacial acetic acid, about ten times its weight, when the acetate of III precipitated. Phosgene was bubbled through the mixture<sup>5</sup> until the precipitate had dissolved.

After evaporation of the acetic acid *in vacuo*, dissolving the residue in hot water, treating with active carbon to decolorize the solution, and filtrating, IV crystallizes on cooling. Yield 15 % of the theoretical. Through recrystallization from alcohol colourless needles, m. p. 156°, were obtained.

The mixed melting point of this substance and the anti-fungal substance of wheat and maize plants showed no depression. Acetyl derivative m. p. 150° C (no depression).

UV spectra (in water): Synthetic product: Max. 229–230  $m\mu$  and 285–286  $m\mu$ ; Min. 255  $m\mu$ ;  $\text{Log } \epsilon$  286  $m\mu$  = 3.74.

Isolated anti-fungal substance: Max. 285–286  $m\mu$ ; Min. 255  $m\mu$ .

*Acknowledgement.* We are indebted to Professor A. I. Virtanen for his great interest in this work and to the *Rockefeller Foundation* for a grant awarded to the Foundation for Chemical Research.

1. Virtanen, A. I., Hietala, P. K. and Wahlroos, Ö. *Suomen Kemistilehti B* **29** (1956) 143.
2. Will, W. and Pukall, W. *Ber.* **20** (1887) 1119.
3. Bechold, J. *Ber.* **22** (1889) 2374.
4. Grandmougin, E. *Ber.* **39** (1906) 2494.
5. Zimmer, H. and Herbig, H. *Ber.* **88** (1955) 1241.

Received September 18, 1956.

## On the Preparation of Alkyl Cyanides

ARNE BRANDSTRÖM

*Research Laboratory, Pharmacia Ltd.,  
Uppsala, Sweden*

In an interesting paper Lewis and Susi<sup>1</sup> report that the preparation of alkyl cyanides from the corresponding primary bromides gives very good yields when ethylene glycol is used as solvent.

Glycols seem to be excellent for this reaction, being good solvents of alkali cya-