

# Note on the Crystal Structure of Trititanium Pentoxide

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The crystal structure of trititanium pentoxide has been determined from single-crystal X-ray data using Fourier techniques. The oxide specimen was prepared by melting an appropriate mixture of titanium metal and titanium dioxide (both better than 99.97 % pure) in an electric arc furnace under argon atmosphere and subsequent annealing of the crushed sample for two weeks at 1,150°C in a sealed, evacuated silica tube. In this way black, prismatic crystals several hundredths of a millimeter in length were obtained. The composition of the sample after the heat-treatment was found to be  $\text{TiO}_{1.667}$  (calculated for  $\text{Ti}_3\text{O}_5$ ;  $\text{TiO}_{1.667}$ ). Powder photographs of samples quenched in the electric arc furnace and of those annealed at 1,150°C were identical.

The following data were obtained for the monoclinic structure:

Unit cell dimensions (from Guinier powder photographs taken with  $\text{CuK}\alpha_1$  radiation

using potassium chloride ( $a = 6.292$  Å) as an internal standard)

$$a = 9.757 \text{ Å}, b = 3.802 \text{ Å}, c = 9.452 \text{ Å}, \beta = 93^\circ.11$$

Cell content: 4  $\text{Ti}_3\text{O}_5$  (observed density 4.20, calculated density 4.24).

Space-group:  $C2/m$  (No. 12)

Arrangement of atoms: All atoms occupy four-fold positions  $4(i)$ :

$$(000, \frac{1}{2}\frac{1}{2}0) + x0z, \bar{x}0\bar{z}$$

4 $\text{Ti}_1$	with $x = 0.129, z = 0.043$
4 $\text{Ti}_2$	with $x = 0.280, z = 0.268$
4 $\text{Ti}_3$	with $x = 0.053, z = 0.366$
4 $\text{O}_1$	with $x = 0.187, z = 0.063$
4 $\text{O}_2$	with $x = 0.236, z = 0.239$
4 $\text{O}_3$	with $x = 0.095, z = 0.337$
4 $\text{O}_4$	with $x = 0.454, z = 0.161$
4 $\text{O}_5$	with $x = 0.371, z = 0.442$

The quotient  $\Sigma||T_o| - |T_c||/\Sigma|T_o|$  is 0.14 (calculated for all reflexions  $h0l$ ,  $h1l$  and  $h2l$  registered with  $\text{CuK}\alpha$  radiation).

Interatomic distances:

Ti—O	1.78–2.21 Å
O—O	2.55–3.25 Å

The structure is illustrated in Fig. 1. The titanium atoms are indicated by

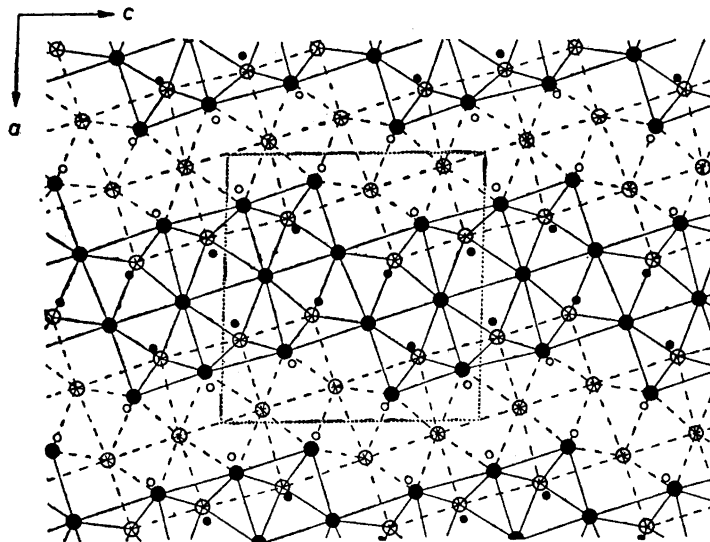


Fig. 1.

smaller rings and the oxygen atoms by larger ones. Open and filled circles represent atoms at  $y = 0$  and  $y = \frac{1}{2}$ , respectively. The extension of the unit mesh is indicated by dotted lines. The titanium atoms are surrounded by six oxygen atoms to form  $\text{TiO}_6$  octahedra which are joined by edges and corners. The coupling together of the octahedra is different from that found in anosovite (pseudobrookite structure) by Zhdanov and Rusakov<sup>1</sup>. Studies on the  $(\text{Ti}_{1-x}, \text{Fe}_x)_2\text{O}_5$  system have shown the pseudobrookite-type structure to be present even at rather low contents of iron ( $x < 0.05$ )<sup>2</sup>. Further studies on the relationships between the  $\text{Ti}_2\text{O}_5$  structure found in this investigation and the anosovite structure are in progress.

Full details on this investigation and a discussion of the structure will be given elsewhere.

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1. Zhdanov, G. S. and Rusakov, A. A. *Trudy Inst. Krist., Akad. Nauk S.S.S.R.* **9** (1954) 165.
2. Åsbrink, S. *To be published*.

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### Correction to: Refractometric Determination of Diffusion Constants in the Critical Region of Partly Miscible Liquids\*

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In Fig. 4, p. 895 the figures of the ordinate scale should be one fifth of those shown.

Consequently, the inclination of the resultant straight line of Fig. 4, which is stated at the end of the text to be  $0.37 \text{ cm}^2 \text{ per day and } ^\circ\text{C}$ , should in fact be one fifth of this amount, i. e.,  $0.074 \text{ cm}^2 \text{ per day and } ^\circ\text{C}$ .

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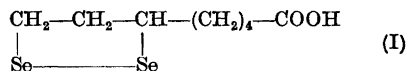
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## The Selenium Analogue of 6-Thioctic Acid

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In connection with preparative and physico-chemical studies on organic disulphides and diselenides<sup>1</sup>, particularly cyclic molecules<sup>2</sup>, the author started a synthesis of the selenium analogue of 6-thioctic acid ( $\alpha$ -lipoic acid) some months ago. The new acid (I), for which the name 6-selenoctic acid is proposed, has now been obtained in crystalline form, and investigations of its chemical and biochemical properties are in progress.



The primary aim of this investigation was twofold. First one could expect the 5-membered ring in 6-selenoctic acid to be more stable than the unsubstituted 1,2-diselenacyclopentane, because it is known that substituents stabilize strained rings. Secondly, 6-selenoctic acid might possess interesting biological properties because of its similarity to 6-thioctic acid. When the present investigation was in progress, Schwarz and Foltz<sup>3</sup> reported the very remarkable discovery of a selenium-containing factor in casein and yeast. This factor ("factor 3") has been known for some years to be active against dietary necrotic liver degeneration and other diseases<sup>3,4</sup>, but only recently Schwarz and Foltz showed that the biological action was connected with a low molecular weight organic selenium compound of unknown structure. Since pyruvic acid and lactic acid are accumulated in the blood of factor 3 deficient animals it seems possible to the present author that factor 3 is a cofactor in the oxidative decarboxylation of pyruvic acid. It is not impossible that 6-selenoctic acid might act as such a cofactor like 6-thioctic acid, and the hypothesis might be advanced that factor 3 is identical with or related to 6-selenoctic acid.

6-Selenoctic acid has been prepared in a way analogous to that used by Reed and