

was brought to pH 7.5 with NaOH (glass electrode). Precipitated impurities were filtered off and discarded. The clear filtrate was acidified to pH 5.5 with HCl and the secretin precipitated with ether. After reprecipitation from water with sodium chloride at saturation it was collected on a suction filter and sucked as dry as possible. In repeated preparations the moist material had an activity of about 7 000 HCU per mg. This material was dissolved in water and the pH of the solution brought to 7.2. Three volumes of acetone were then added to the solution. The precipitate that formed was collected by centrifugation and discarded. The secretin was thrown out of solution by adding nine more volumes of acetone to the clear supernatant. The precipitate was collected by centrifugation and dissolved in water. The pH of the solution was adjusted to 5.5 with acetic acid and the secretin lyophilized from it. The lyophilized material had an activity varying in different preparations from 10 000 to 15 000 HCU per mg. The yield was 0.05–0.1 mg per hog.

Chromatography. 45 mg of a preparation with 12 000 HCU per mg was dissolved in 9 ml of 0.02 M ammonium bicarbonate and the solution allowed to sink into a column (7.5 × 1.4 cm) of 3 g of carboxymethyl cellulose which had been equilibrated with 0.02 M ammonium bicarbonate. Elution was carried out with 0.02 M ammonium bicarbonate. Fractions of 10 ml were collected. The flow rate was 40 ml/h. Collection of the fractions was started from the time when the secretin solution was applied to the column. Between the tenth and the eleventh fraction the eluting solution was changed to 0.2 M ammonium bicarbonate.

The approximate relative concentration of proteinaceous material in the fractions was determined by the Herriott reaction⁶ as adapted by Lowry *et al.*⁷, except that the copper salt containing stock solution of the latter authors was made up in 0.1 N NaOH instead of in water. The secretin activity was determined by injecting appropriate aliquots of the fractions intravenously into cats and titrating the amount of alkali secreted through a cannula in the main pancreatic duct in response to the injection.

The results are shown in Fig. 1.

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Polymorphism in Calcium Tantalum(V)Oxide CaTa_2O_6

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It was recently reported by Gasperin¹ that an equimolecular mixture of tantalum(V)oxide and calcium carbonate upon heating at about 1 700°C gives a product possessing the composition CaTaO_3 with a multiple-cell, deformed perovskite structure, *a* of the pseudocubic subcell unit being 3.88 Å. The formation of the new compound should evidently be accompanied by a reduction of tantalum to the tetravalent state. Such a process however, seems rather unlikely in view of the high reluctance to reduction shown by tantalum(V)oxide and of the white colour of the specimen which rather suggests the presence of the state of maximum valency. The density reported for the product (7 g.cm⁻³) likewise is not in good agreement with that calculated for the formula CaTaO_3 (7.6 g.cm⁻³) but is in fair accordance with that of a compound such as $\text{Ca}_{0.5}\text{TaVO}_3$ (7.1 g.cm⁻³). The validity of the latter formula was actually demonstrated by Gasperin in a subsequent analytical investigation of the

substance². A compound of the composition CaTa_2O_6 was reported by Ismailzade³ to have a complex structure.

In the present study mixtures of tantalum(V)oxide and calcium carbonate were heated at 1 200°C in air for several hours and then fused in an electric arc furnace in an argon atmosphere. The specimen rested on the water-cooled copper base of the furnace which caused rapid cooling upon discontinuing the arc. The products were characterized by their X-ray powder patterns, registered in a Guinier camera with strictly monochromatized $\text{CuK}\alpha_1$ radiation and using potassium chloride ($a = 6.2919 \text{ \AA}$ at 20°C) as an internal standard. The following results were thus obtained.

Starting composition	Colour of product	X-ray evidence
$\text{CaCO}_3 + \text{Ta}_2\text{O}_5$	greyish white	non-cubic perovskite-type pattern
$\text{CaCO}_3 + 0.5 \text{ Ta}_2\text{O}_5$	»	complicated pattern
$\text{CaCO}_3 + 0.4 \text{ Ta}_2\text{O}_5$	»	»

Obviously the first specimen, corresponding to a calcium to tantalum ratio of 1:2 is a monophasic, perovskite-type sample. In order to establish the valency of tantalum in this product it was heated for several hours in a stream of oxygen at 800°C. The weight of the sample was not affected by this treatment which shows the tantalum to be pentavalent and suggests the formula of the compound to be $\text{Ca}_{0.5}\text{TaO}_3$. The colour showed only a slight shift upon heating, from the original greyish to brownish white but the X-ray powder pattern displayed a considerable number of extra reflexions in addition to the lines corresponding to a perovskite-type phase.

In order to study the transformation thus found to occur at 800°C samples of the original preparation ($\text{Ca}_{0.5}\text{TaO}_3$) were heated for several hours at 1 300°C and 650°C, respectively. The following observations were made:

Heating temperature	Density	X-ray evidence
1 300°C	7.02	complicated pattern
650°C	6.75	cubic, perovskite-type

Upon melting both samples were reconverted to give the non-cubic perovskite-type pattern. The cubic perovskite, upon heating at 1 300°C, gave the complicated pattern mentioned above but this transformation could not be reversed.

The X-ray pattern of the sample which had been quenched from the melting temperature was not indexed. It showed a splitting up of several of the perovskite-type reflexions.

The pattern of the sample heat-treated at 1 300°C, was quite different from the perovskite type. It could be interpreted in terms of an orthorhombic unit cell with

$$a = 11.067 \text{ \AA}, b = 5.380 \text{ \AA}, c = 7.505 \text{ \AA}$$

indicating a cell content of four formula units of CaTa_2O_6 (calculated density = 7.40). Studies on the crystal structure of this modification are in progress.

The unit cell edge of the cubic perovskite structure obtained at 650°C was found to be 3.886 Å. No lines indicating a multiple-cell structure were observed. The arrangement of the calcium atoms should thus be a random one and comparable with that of the alkali atoms in the cubic sodium⁴ and lithium^{5,6} wolfram bronzes of perovskite type.

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