

that the measured and calculated values are approximately proportional for the α , γ , δ and ϵ isomer. To bring the point of the β isomer onto the straight line we have to assume an equilibrium $\epsilon\kappa\kappa\epsilon \rightleftharpoons \kappa\epsilon\epsilon\kappa$ with approximately 87 % contribution from the former and about 13 % contribution from the latter form.

Because of the small differences between the dipole moments of the α , δ and ϵ isomers and because of the rather uncertain assumption of the undistorted molecules the dipole moments measurements must not be regarded as conclusive for the structure of these three isomers. The results are, however, in accordance both with the results of the Ethyl Corporation team² and with the electron diffraction work done at our institute. The electron diffraction work which is going to be published soon in this journal establishes the structure of the α , δ and ϵ isomers and excludes the presence of the $\epsilon\kappa\epsilon\kappa$ form and any appreciable contribution from the forms containing more than two ϵ bonds. The electron diffraction work thus demonstrates the validity of the assumptions on which the present discussion is based.

Acknowledgements. The authors wish to express their gratitude to the Ethyl Corporation, Detroit, for having supplied them with samples of the five isomers of BTC. One of the authors (J. M.) wishes to acknowledge Det Grevelige Hielmstjerne Rosencronske legat for financial aid.

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Received March 13, 1952.

On the Tantalum — Oxygen System

STIG LAGERGREN and ARNE MAGNÉLI

*Institute of Chemistry, University of Uppsala,
Uppsala, Sweden*

Through the chemical and X-ray investigations on the niobium-oxygen system, carried out by Brauer¹ a decade ago, the occurrence of the phases NbO, NbO₂, and Nb₂O₅ (three modifications) was established while the oxides Nb₂O, Nb₂O₃, and Nb₃O₅ reported by previous authors were shown not to exist. There are also a number of tantalum oxides described in literature, viz. TaO, Ta₂O₃, TaO₂, and Ta₂O₅. Among these compounds, however, the pentoxide is the only one, whose existence has been firmly established. As a systematic investigation of the tantalum-oxygen system was lacking, we decided to study this matter.

The starting materials for the investigation were tantalum powder and tantalum pentoxide (both of Fansteel manufacture), weighed amounts of which were intimately mixed, pressed to pastilles and heated in vacuo. The products were investigated by taking X-ray powder photographs using a Guinier focusing camera with monochromatized Cu-K α radiation.

It has recently been found that niobium and niobium pentoxide readily react to form intermediate oxides when heated for one or two days at 1 000–1 100° C in sealed off silica tubes*. When mixtures of tantalum and tantalum pentoxide were correspondingly treated, no reaction could be observed even after rather intense heating (1 100° C for ten days or 1 250° C for two days).

In order to obtain higher temperatures a vacuum high frequency induction fur-

* Private communication from Dr. Beryl M. Oughton.

nace² was used. The samples were kept in covered zirconia crucibles, which were externally wound with spirals of tantalum wire in order to produce a favourable heating effect. In this way a maximum temperature within the crucibles of about 1 800° C could be obtained. The samples were allowed to cool down in the furnace immediately from the highest temperature applied. Due to the low heat capacity of the furnace this process was rather rapid. Powder photographs of samples heated in this way showed the lines of tantalum at unaltered positions as well as lines of another phase, which was not identical with the tantalum pentoxide used. (The diffraction pattern of the latter was in fair agreement with the drawing given by Brauer¹.) Tantalum metal evidently does not dissolve significant quantities of oxygen under these circumstances. For niobium Brauer states a solubility of less than 0.05 oxygen atoms/metal atom.

Attempts to prepare the new phase in a pure state by heating various mixtures of tantalum and tantalum pentoxide were fruitless. However, a pastille of pure tantalum pentoxide when heated for a short time in the vacuum furnace changed its colour from white to grey and powder photographs of the product showed only the reflections of the new phase and no trace of the tantalum lines. In order to establish the composition a weighed sample of the grey substance was heated in a stream of oxygen at 1 000° C for several hours. The colour turned white but the weight remained constant after the heating and powder photographs showed the ordinary pattern of tantalum pentoxide. It was thus obvious that the new phase represents a previously unknown high temperature modification of tantalum pentoxide. Accordingly it could also be prepared by heating a pastille of the original pentoxide specimen on a platinum disk in the gas-oxygen torch, however, the product obtained in this way was white.

This is the true colour of the substance while the grey colour of the specimens prepared in vacuo is due to the presence of minute amounts of tantalum metal. Powder photographs of pentoxide samples that had been very intensely heated in the vacuum furnace showed namely faint tantalum lines in addition to those of the high temperature modification.

In order to find the transition temperature of the two modifications pastilles of tantalum pentoxide were heated in an uncovered tantalum crucible in the vacuum high frequency induction furnace. The temperature was read on an optical pyrometer. A sample heated at 1 310° C for one hour remained white and gave exclusively the powder lines of the low temperature modification while a preparation similarly treated at 1 330° C turned grey and was found to contain both forms of tantalum pentoxide. The transition point may thus be estimated to be 1 320° C with a probable error of $\pm 20^\circ$. The grey colour of the product indicates that the high temperature modification has a considerable oxygen pressure even at or slightly above the transition temperature. This is in agreement with the statement by von Bolton³ that metallic tantalum may be prepared by pumping off the oxygen from tantalum oxide at a high temperature. On the other hand the pronounced stability of the low temperature modification was also strikingly demonstrated by the negative result when attempts were made to reduce this phase in a stream of hydrogen at about 1 250° C.

While the low temperature modification of tantalum pentoxide is isostructural with the corresponding form of niobium pentoxide^{1,4}, the powder photographs of the high temperature tantalum pentoxide indicate this structure to be quite different from those of the other two niobium pentoxide modifications. The investigation is continuing with the special purpose of finding the crystal structure of the high

temperature modification of tantalum pentoxide.

The authors wish to thank professor G. Hägg for his kind interest in the investigation and for the facilities put at our disposal. This work forms part of a research program on oxides and oxide systems financially supported by the Swedish Natural Science Research Council.

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Received October 5, 1951.

On the Occurrence of Sedoheptulose in Certain Species and Genera of the Plant Family Saxifragaceae

ARNOLD NORDAL and DAGRUN ØISETH

Department of Pharmacognosy, Pharmaceutical Institute of the University of Oslo, Norway

The heptose sedoheptulose (D-altroheptulose) had, until quite recently, been detected in only a few species and genera of the plant family *Crassulaceae*, and investigators were of the opinion that where this sugar occurred fermentable sugars were absent¹.

Nordal and Klevstrand^{2,3} recently demonstrated the presence of both sedoheptulose and fermentable sugars in representatives of all the five sub-families of the plant family *Crassulaceae*.

Benson, Bassham and Calvin⁴ identified monophosphate esters of sedoheptulose in *Chlorella*, *Scenedesmus*, and *Rhodospirillum rubrum*, and in the leaves of barley seedlings, soybean, alfalfa, sugar beet, spinach and geranium.

The present authors succeeded in isolating sedoheptulose from the rhizome of *Primula elatior* (L.) Hill.⁵ and more recent,

unpublished results have shown that the rhizome of *Primula vulgaris* Huds. also contains sedoheptulose, as does *Primula veris* (L.) Huds. In the case of the latter plant, extracts from the flowers, leaves and rhizomes were examined separately, and it was found that the aerial portion of the plant, especially the flowers, contained considerable quantities of sedoheptulose, as well as sucrose and fructose. In the rhizome, on the other hand, the latter two sugars predominated and sedoheptulose could hardly be detected. The material was not tested for aldoses.

These results, together with others not yet published, obtained with extracts from representatives of certain other plant families lend credence to our assumption that sedoheptulose is widely distributed in the plant kingdom, and that this sugar plays a far more important part in the metabolism of plants than has been considered hitherto.

The plant family *Saxifragaceae* gave some of the most encouraging results in our screening investigations, and as this family is closely related to the *Crassulaceae*, we thought it would be worth while to examine some specimens of the family more closely.

Sedoheptulose was identified by means of paper chromatography in eight of ten species and in general as the chief component of the sugar mixture (cf. Table 1). No sedoheptulose could be detected in *Ribes alpinum* L. or *Saxifraga stellaris* L. by the method used.

Fresh plant material which was fixed with boiling alcohol was used in all the analysis, except in the case of *Parnassia palustris* L. and *Saxifraga stellaris* L., where the air dried material was extracted with water.

The aqueous extracts were purified by means of ion exchange resins. The alcohol extracts were evaporated and freed from chlorophyll after the addition of water, and then all the extracts were concentra-