Dr J. Karle for supplying new heavy-atom parameters based on the absorption–correction data.

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## The crystal structure of D-iso-ascorbic acid. Errata.

By NEZHAT AZARNIA, HELEN M. BERMAN and R. D. ROSENSTEIN, *Department of Crystallography, University of Pittsburgh, Pittsburgh, Pa.* 15213, *U.S.A.*

Typographical errors in several numerical results in the paper by Azarnia, Berman & Rosenstein [*Acta Cryst.* (1972). B**28**, 2157–2161] are corrected.

The $x$ parameter of O(6) in Table 1 should be 0·1292 (6), not 0·2192 (6). In Table 3 the hydrogen-bonding distance $d(jk)$ of H(O2) → O(6$a$) should be 1·76 Å, not 176· Å, and the torsion angle O(6)–C(6)–C(5)–O(5) should be 67·2°, not 70·7° (p. 2161, first line).

We are indebted to Dr David L. Hughes for informing us of these errors.

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## The crystal structure of β-tantalum.

By P. T. MOSELEY and C. J. SEABROOK, *Applied Chemistry Division, A.E.R.E., Harwell, Berks, England*

The X-ray powder pattern of β-tantalum has been indexed in terms of a tetragonal unit cell with $a = 10·194$ and $c = 5·313$ Å. β-Tantalum appears to be isomorphous with β-uranium.

In recent years a second crystalline modification of elemental tantalum has been reported in addition to the body centred cubic form (Read & Altman, 1965; Mills, 1966).

The new phase, which is now generally referred to as β-tantalum exhibits rather different electrical properties from the cubic variety and is thus of some concern to those employing tantalum films in microcircuits (Westwood, 1970).

Previously β-tantalum was prepared during sputtering experiments and the diffraction data available have in some cases suffered from the effect of preferred orientation. In other cases the material was not single phase. X-ray powder patterns were indexed in terms of a tetragonal unit cell, initially having $a = 5·34$, $c = 9·94$ Å (Read & Altman, 1965) and later $a = 5·32$, $c = 9·92$ Å (Mills, 1966).

The electron diffraction powder pattern of β-tantalum has recently been indexed with the tetragonal parameters $a = 10·29$, $c = 9·2$ Å (Das, 1972).

β-Tantalum has now been prepared as a single phase by electrodeposition from a molten fluoride bath at 800°C. The X-ray powder pattern has been recorded using a Nonius Guinier camera calibrated against a silicon standard ($a = 5·4307$ Å) and re-indexed as shown in Table 1. The unit cell is indeed tetragonal but the parameters, which have been derived with the aid of a least-squares refinement program (Marples & Shaw, 1966), are $a = 10·194$, $c = 5·313$ Å.

The intensities of the powder lines were recorded using a microdensitometer and are also given in Table 1. The general form of these intensities is remarkably similar to

Table 1. *Part of the X-ray powder pattern of β-tantalum* (Cu *Kα radiation*)

| h k l | sin² θ (obs) | sin² θ (calc) | Intensity* |
|---|---|---|---|
| 1 0 1 | 0·0267 | 0·0267 | 4 |
| 1 1 1 | 0·0326 | 0·0324 | |
| 3 1 0 | 0·0571 | 0·0570 | |
| 2 2 1 | 0·0666 | 0·0666 | |
| 3 1 1 | 0·0779 | 0·0780 | 8 |
| 0 0 2 | 0·0841 | 0·0840 | 42 |
| 1 0 2 | 0·0895 | 0·0897 | |
| 4 1 0 | 0·0971 | 0·0969 | 78 |
| 3 3 0 | 0·1029 | 0·1027 | 53 |
| 2 0 2 | 0·1072 | 0·1068 | 54 |
| 2 1 2 | 0·1129 | 0·1125 | 82 |
| 4 1 1 | 0·1183 | 0·1180 | 100 |
| 3 3 1 | 0·1239 | 0·1237 | 64 |
| 3 1 2 | 0·1408 | 0·1410 | 18 |
| 5 1 0 | 0·1478 | 0·1483 | |
| 3 2 2 | 0·1581 | 0·1582 | 4 |
| 4 3 1 ⎫ 5 0 1 ⎭ | 0·1633 | 0·1636 | 5 |
| 5 1 1 | 0·1690 | 0·1693 | 6 |
| 4 0 2 | 0·1752 | 0·1753 | |
| 5 2 1 | 0·1863 | 0·1865 | |
| 6 0 1 | 0·2264 | 0·2264 | 4 |
| 6 1 1 | 0·2322 | 0·2321 | 5 |
| 3 1 3 | 0·2458 | 0·2461 | |
| 6 2 1 | 0·2492 | 0·2492 | 12 |
| 5 4 1 | 0·2545 | 0·2550 | |
| 6 3 1 | 0·2782 | 0·2778 | 31 |
| 4 1 3 | 0·2865 | 0·2861 | 45 |
| 6 0 2 | 0·2890 | 0·2895 | |
| 3 3 3 | 0·2918 | 0·2918 | |
| 6 1 2 | 0·2951 | 0·2952 | |
| 7 2 0 | 0·3027 | 0·3025 | 30 |
| 5 5 1 ⎫ 7 1 1 ⎭ | 0·3062 | 0·3063 | |
| 6 2 2 | 0·3129 | 0·3123 | |
| 5 4 2 | 0·3180 | 0·3180 | |
| 5 1 3 | 0·3379 | 0·3375 | 25 |
| 6 5 0 | 0·3478 | 0·3481 | |
| 5 5 2 ⎫ 7 1 2 ⎭ | 0·3696 | 0·3694 | |
| 8 2 0 | 0·3884 | 0·3881 | 20 |
| 3 2 4 | 0·4100 | 0·4104 | 16 |

$a = 10·194 \pm 0·003 \quad c = 5·313 \pm 0·002$

\* Normalized to strongest reflexion.

that reported for β-uranium (Thewlis, 1952) even though the two sets of data were recorded in different types of camera and it appears that β-tantalum and β-uranium are isomorphous. The $c/a$ ratio for β-tantalum (0·521) is close to that for β-uranium (0·526) and the calculated density for β-tantalum based on thirty atoms in the unit cell (16·33 g cm⁻³) compares well with the value measured

(16·1 g cm⁻³) by weighing in air and in carbon tetrachloride.

In view of the uncertainty concerning the details of the β-uranium structure (Donohue & Einspahr, 1971) it is felt that an accurate assessment of the positional parameters of β-tantalum will only be possible if single-crystal intensity data become available.

Chemical analyses of specimens of the two forms of tantalum prepared in the same apparatus are shown in Table 2. These results do not indicate whether or not β-tantalum should be considered as an impurity-stabilized phase (Read & Altman, 1965). A specimen of β-tantalum heated to 1000 °C *in vacuo* and cooled to room temperature had transformed to cubic tantalum. The reverse change (cubic → β) was not achieved by means of thermal excursions. If β-tantalum is thermodynamically stable at room temperature then the element is quite unusual among transition metals in exhibiting a crystalline form other than hexagonal close packed, body centred cubic or face centred cubic (Pettifor, 1969).

Table 2. *Analyses for trace impurities as p.p.m. (w/w) except where otherwise indicated*

| | Cubic tantalum | β-Tantalum |
|---|---|---|
| Hydrogen | <10 | <10 |
| Oxygen | 100 | 100 |
| Nitrogen | <20 | <20 |
| Chromium | 0·1% | 0·1% |
| Sodium | <0·2% | <0·2% |
| Zinc | <0·2% | <0·2% |
| Tungsten | <500 | <500 |
| Iron | <200 | <200 |

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