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The Oxidation Product of Bis(glyoximato)palladium(II) with Iodine

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Abstract. Pd(C₂H₃N₂O₂)₂I, tetragonal, *P4₂/mcm*, *a* = 8.645 (1), *c* = 6.488 (1) Å, *Z* = 2, *D_x* = 3.50 g cm⁻³. The metal complex units form columnar stacks with a Pd-Pd distance of 3.244 (1) Å. Linear, probably disordered, iodine chains run parallel to the stacks of the complex molecules.

Introduction. The crystal used was from a preparation reported by Endres, Keller & Lehmann (1975).

The systematic absences *0kl* for *l* = 2*n* + 1 are consistent with the space groups *P4̄c2*, *P4cm*, *P4₂/mcm*. Accurate lattice constants were determined from the *θ* values of 23 reflexions by a least-squares procedure (Berdiesinski & Nuber, 1966). Intensities were measured with Mo *Kα* radiation on an automatic diffractometer (Siemens) by a *θ/2θ* scan. 323 reflexions were observed in a *θ* range up to 30° for *l* = 2*n* and 15° for *l* = 2*n* + 1; reflexions with an intensity *I* < 2.58σ*I* were regarded as unobserved. The heavy-atom positions were taken from a Patterson map, which also showed the ligand to lie in the plane of the metal atom. As this condition can be allowed for in each of the possible space groups and the molecular symmetry is consistent with the symmetry operations of the relevant space groups, the calculations were based on the centrosymmetric space group *P4₂/mcm* (*D_{4h}¹⁰*). The positions of the light atoms were obtained by Fourier syntheses.

Least-squares refinement with *ORFLS* and *CRYLSQ* of the X-RAY System (Stewart, Kundell & Baldwin, 1970) led to an *R* of 12.8% with isotropic temperature factors. Refinement with anisotropic temperature factors converged at an *R* of 6.4% (average shift/error 0.02, maximum shift/error 0.08). Scattering factors were those of Hanson, Herman, Lea & Skillman (1964). Atomic parameters are listed in Table 1.*

Discussion. Fig. 1 shows the complex with bond distances and angles. The molecular symmetry is *D_{2h}*, the central Pd atom occupying a crystallographic *mmm* position.

The planar complexes are stacked in columns. Two adjacent molecules within the same stack are twisted by 90° with respect to one another. Parallel to the Pd chains run linear chains of I atoms (Figs. 2 and 3). The complexes stack perpendicular to their planes with a Pd-Pd distance of 3.244 Å, whereas in the unoxidized form the Pd-Pd distance of 3.558 Å is considerably longer and the stacking direction is not perpendicular to the planes of the complexes (Calleri, Ferraris &

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31443 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Atomic parameters ($\times 10^4$)

The anisotropic temperature factor is $\exp[-2\pi^2(U_{11}h^2(a^*)^2 + \dots + 2U_{23}klb^*c^*)]$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Pd	0	0	0	827 (13)	827 (17)	368 (10)	-99 (32)	0	0
I	5000	5000	2500	854 (13)	854 (17)	1183 (24)	0	0	0
N	2291 (22)	9785 (27)	0	816 (122)	627 (150)	455 (70)	-200 (118)	0	0
O	3216 (23)	985 (23)	0	1160 (176)	1100 (172)	632 (108)	-55 (123)	0	0
C	2822 (36)	1547 (34)	0	1265 (227)	930 (206)	407 (108)	-110 (182)	0	0

Viterbo, 1967). An analogous feature is found in Ni(BCD)_2 (Leichert & Weiss, 1975) and its oxidation product with iodine, $\text{Ni(BCD)}_2 \cdot \frac{1}{2}\text{I}$ (Endres, Keller, Moroni & Weiss, 1975), as well as Ni(dpg)_2 (Banks & Barnum, 1958) and $\text{Ni(dpg)}_2\text{I}$ (Gleizes, Marks & Ibers, 1975).

Rotating crystal photographs exhibit diffuse layer lines between the normal layer lines. A similar diffuse scattering of $\text{Ni(BCD)}_2 \cdot \frac{1}{2}\text{I}$ could be accounted for by one-dimensional ordering of chains of triiodide anions (Endres, Keller, Megnamisi-Bélombé, Moroni, Pritzkow & Weiss, 1975). More detailed investigations on the diffuse X-ray scattering will be published in the near future (Comès, Endres, Keller, Megnamisi-Bélombé, Moroni, Pritzkow & Weiss, 1975).

Calculations were performed on computers Siemens 301 (Anorganisch-Chemisches Institut der Universität Heidelberg) and IBM 370/168 (Universitäts-Rechenzentrum Heidelberg). We acknowledge the support of the Deutsche Forschungsgemeinschaft, Bad Godesberg.

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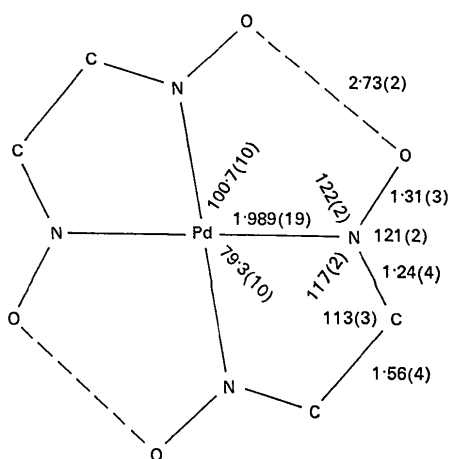


Fig. 1. The Pd(glyoxime)_2 molecule with bond lengths and angles.

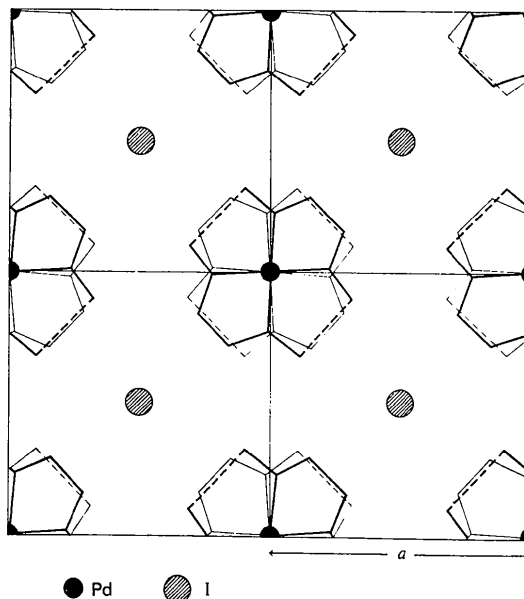


Fig. 2. Projection of the structure on the (001) plane.

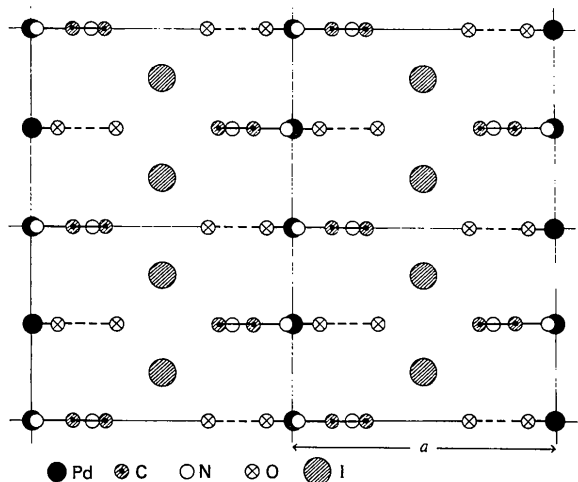


Fig. 3. Projection of the structure perpendicular to c .

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