

## Bis(oxamide oxime)platinum(II) Bis[(oxamide oximato)(oxamide oxime)platinum(II)] Bis(sulfate) Octahydrate

BY HELMUT ENDRES

*Anorganisch-Chemisches Institut der Universität, Im Neuenheimer Feld 270, D-6900 Heidelberg 1,  
Federal Republic of Germany*

(Received 1 July 1981; accepted 2 December 1981)

**Abstract.**  $[\text{Pt}(\text{C}_2\text{H}_6\text{N}_4\text{O}_2)_2][\text{Pt}(\text{C}_2\text{H}_5\text{N}_4\text{O}_2)(\text{C}_2\text{H}_6\text{N}_4\text{O}_2)_2]_2(\text{SO}_4)_2 \cdot 8\text{H}_2\text{O}$ ,  $\text{C}_4\text{H}_{12}\text{N}_8\text{O}_4\text{Pt}^{2+} \cdot 2\text{C}_4\text{H}_{11}\text{N}_8\text{O}_4\text{Pt}^+ \cdot 2\text{SO}_4^{2-} \cdot 8\text{H}_2\text{O}$ ,  $M_r = 1628.09$ , is triclinic,  $P\bar{1}$ , with  $a = 8.659$  (2),  $b = 10.339$  (2),  $c = 14.218$  (3) Å,  $\alpha = 107.02$  (2),  $\beta = 93.93$  (2),  $\gamma = 114.75$  (1)°,  $V = 1078$  Å<sup>3</sup>,  $Z = 1$ ,  $d_c = 2.51$  Mg m<sup>-3</sup>. Final  $R = 0.028$  for 5270 independent diffractometer data. The compound is isomorphous with the analogous Pd complex. The unit cell contains one centrosymmetric dipositive and two noncentrosymmetric monopositive complex cations forming stacks of triples in the crystal, interspersed with sulfate anions.

**Introduction.** Oxamide oxime (oaoH<sub>2</sub>) (Ephraim, 1889) forms a variety of complexes with the elements of the Ni triad, depending on the metal and on the crystallization conditions (Endres & Weiss, 1981, and references cited therein). The title compound is obtained by adding sulfuric acid to a slurry of  $\text{Pt}(\text{oaoH}_2)_2\text{Cl}_2$  (Endres & Schlicksupp, 1979) in boiling H<sub>2</sub>O until a clear yellow solution forms, from which the yellow-orange crystals precipitate on slow cooling. Lattice constants were derived from the setting angles of 25 reflections centered on a diffractometer (Syntex R3, monochromatic Mo  $K\alpha$  radiation). Data collection ( $\theta$ - $2\theta$  scans, background-peak-background,  $2\theta < 66^\circ$ ) yielded 5270 observed independent reflections with  $I > 2.5\sigma(I)$ . Lorentz and polarization corrections as well as an empirical ( $\psi$  scans) absorption correction were applied. The structure was solved by Patterson and Fourier methods and refined with anisotropic temperature factors by a cascade matrix procedure to a weighted  $R = 0.028$  (unweighted  $R = 0.031$ ).<sup>\*</sup> The weighting scheme was  $w = 1/\sigma^2(F)$ . H atoms could not be located. Calculations were carried out on a NOVA 3 computer, plots were drawn on a Tektronix

plotter. The program package was *SHELXTL* (Sheldrick, 1979).

**Discussion.** The compound is isomorphous with the analogous Pd complex, grown by a diffusion process

Table 1. Atomic coordinates ( $\times 10^4$ ) and isotropic temperature factors ( $\times 10^3$ ) equivalent to the refined anisotropic values

$U_{eq}$  is defined as  $U_{eq} = \frac{1}{3} \text{trace } \bar{U}$ ,  $\bar{U}$  signifying the diagonalized U matrix.

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$ (Å <sup>2</sup> )
Pt(1)	0	0	0	19.6 (1)
Pt(2)	8883.0 (3)	2361.1 (2)	8916.8 (2)	17.4 (1)
N(1)	2553 (6)	1484 (6)	428 (4)	26 (2)
N(2)	388 (6)	764 (6)	1510 (4)	25 (2)
N(3)	4936 (6)	3151 (6)	1813 (4)	34 (2)
N(4)	2413 (6)	2338 (6)	3042 (4)	35 (2)
O(1)	3656 (5)	1944 (5)	-211 (4)	34 (2)
O(2)	-864 (5)	486 (5)	2085 (4)	37 (2)
C(1)	3247 (7)	2143 (7)	1383 (5)	26 (3)
C(2)	1952 (7)	1735 (7)	2040 (5)	25 (3)
N(5)	9224 (6)	3004 (5)	10413 (4)	22 (2)
N(6)	11422 (6)	3947 (5)	9406 (4)	23 (2)
N(7)	8329 (6)	1615 (6)	7400 (4)	25 (2)
N(8)	6398 (6)	844 (5)	8584 (4)	22 (2)
N(9)	11142 (7)	4521 (6)	11986 (4)	36 (2)
N(10)	13688 (6)	5660 (6)	10816 (4)	31 (2)
N(11)	6139 (6)	-148 (6)	5934 (4)	31 (2)
N(12)	3887 (6)	-961 (6)	7303 (4)	33 (2)
O(3)	7927 (5)	2381 (5)	10900 (3)	29 (2)
O(4)	12606 (5)	4538 (5)	8837 (3)	30 (2)
O(5)	9371 (5)	2100 (6)	6741 (3)	40 (2)
O(6)	5507 (5)	527 (5)	9298 (3)	28 (2)
C(3)	10749 (7)	4036 (6)	10982 (4)	22 (2)
C(4)	12050 (7)	4594 (6)	10368 (5)	23 (2)
C(5)	6729 (7)	538 (7)	6934 (4)	23 (2)
C(6)	5603 (7)	141 (6)	7651 (5)	24 (2)
S(1)	3353 (2)	2097 (2)	6616 (1)	26.8 (6)
O(7)	5251 (6)	2933 (6)	7014 (4)	42 (2)
O(8)	2822 (6)	501 (5)	6017 (3)	36 (2)
O(9)	2478 (6)	2144 (6)	7486 (4)	48 (2)
O(10)	2821 (7)	2812 (6)	6009 (5)	63 (3)
O(11)	3076 (7)	-3151 (6)	4660 (4)	57 (3)
O(12)	9810 (7)	-2154 (6)	5537 (4)	54 (3)
O(13)	9596 (7)	4257 (7)	3703 (4)	61 (3)
O(14)	6083 (7)	4321 (6)	3980 (5)	62 (3)

<sup>\*</sup> Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36583 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

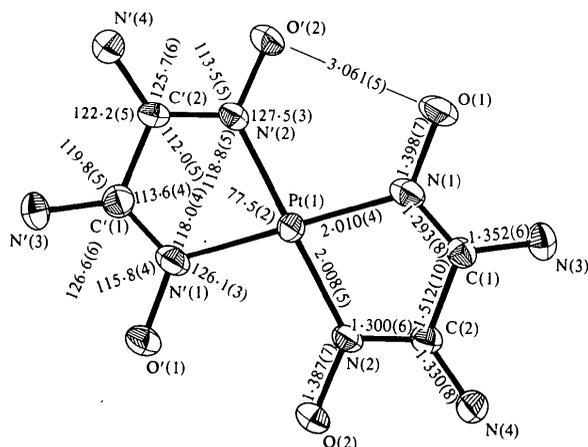


Fig. 1. The centrosymmetric [Pt(oaoH<sub>2</sub>)<sub>2</sub>]<sup>2+</sup> dication with numbering scheme, bond distances (Å) and angles (°).

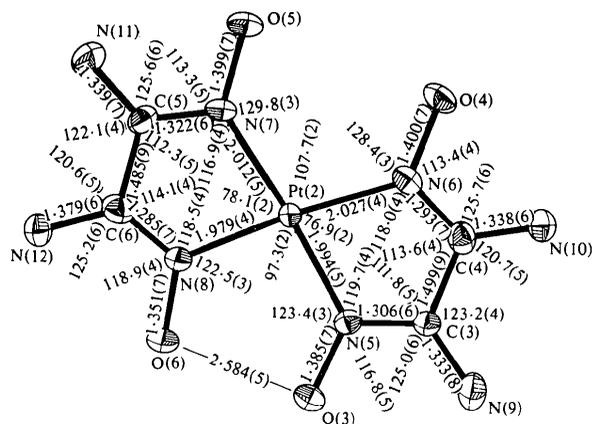


Fig. 2. The noncentrosymmetric [Pt(oaoH)(oaoH<sub>2</sub>)]<sup>+</sup> cation with numbering scheme, bond distances (Å) and angles (°).

Table 2. Bond lengths (Å) and angles (°) in the sulfate ion

S(1)—O(7)	1.472 (4)	O(7)—S(1)—O(8)	110.6 (3)
O(8)	1.465 (5)	O(9)	108.4 (3)
O(9)	1.494 (6)	O(9)	110.2 (3)
O(10)	1.452 (8)	O(8)—S(1)—O(9)	108.5 (3)
		O(10)	110.2 (3)
		O(9)—S(1)—O(10)	108.8 (4)

(Endres & Weiss, 1981). The lattice accommodates a centrosymmetric [Pt(oaoH<sub>2</sub>)<sub>2</sub>]<sup>2+</sup> dication (I) with Pt at the-origin (Fig. 1), and a noncentrosymmetric monocation [Pt(oaoH)(oaoH<sub>2</sub>)]<sup>+</sup> with Pt at a general position (II) (Fig. 2). Atomic coordinates are listed in Table 1. Table 2 contains bond lengths and angles in the sulfate ion. The Pt—N distances and angles in this compound do not differ significantly from those in the Pd complex. The dication does not exhibit intramolecular H bridges; in the monocation only one intramolecular H bridge has formed. The complex

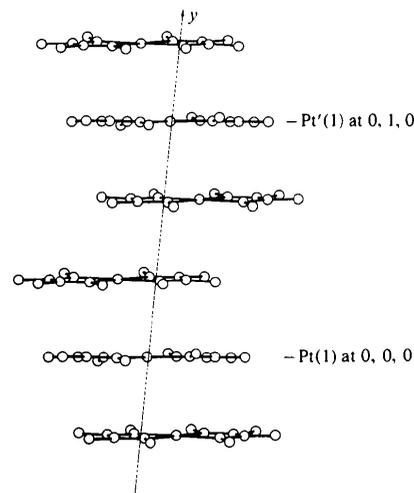


Fig. 3. View of two triples repeating along **b**. The molecules are projected parallel to the mean plane through the central molecule of a triple.

molecules are arranged in triples with (I) at the center and two species (II), related by an inversion center, forming the outer molecules of the triple. The molecular overlap within a triple is very pronounced and corresponds to the one found in the Pd complex (Endres & Weiss, 1981). The triples pile up into stacks in the **b** direction, with a much larger slip of adjacent molecules between the triples than within the triples. This kind of stacking is indicated in Fig. 3 which shows the repeat of the triples along **b**. Even if the interplanar distances (3–4 Å) are identical within and between the triples the term 'triple' is justified by the different molecular overlap, the same as found in the Pd compound. With the Pt compound described here and its Pd analogue an isomorphous pair of oxamide oxime complexes of the metals of the Ni triad has been found for the first time. Normally, the crystal and molecular structures are quite different for the different metals, in contrast to the normal behavior of  $\alpha,\beta$ -dione dioximate complexes of the Ni triad (Endres, Keller, Lehmann, Poveda, Rupp & van de Sand, 1977).

## References

- ENDRES, H., KELLER, H. J., LEHMANN, R., POVEDA, A., RUPP, H. H. & VAN DE SAND, H. (1977). *Z. Naturforsch. Teil B*, **32**, 516–527.
- ENDRES, H. & SCHLICKSUPP, L. (1979). *Acta Cryst.* **B35**, 3035–3036.
- ENDRES, H. & WEISS, J. (1981). *Acta Cryst.* **B37**, 1360–1363.
- EPHRAIM, J. (1889). *Chem. Ber.* **22**, 2305–2306.
- SHELDRIK, G. M. (1979). *SHELXTL*. An integrated system for solving, refining and displaying crystal structures from diffraction data. Univ. of Göttingen, Federal Republic of Germany.