

Table 2. Bond lengths (Å) and angles (°)

Mo(1)—N(1)	2.334 (2)	Mo(1)—N(2)	2.345 (2)
Mo(1)—C(1)	2.032 (3)	Mo(1)—C(2)	1.943 (3)
Mo(1)—C(3)	1.951 (3)	Mo(1)—C(4)	2.035 (3)
O(1)—C(1)	1.127 (4)	O(2)—C(2)	1.161 (4)
O(3)—C(3)	1.153 (3)	O(4)—C(4)	1.143 (4)
N(1)—C(5)	1.509 (4)	N(1)—C(9)	1.494 (4)
N(1)—C(10)	1.465 (5)	N(2)—C(6)	1.485 (4)
N(2)—C(7)	1.482 (5)	N(2)—C(8)	1.490 (5)
C(5)—C(6)	1.483 (5)		
N(1)—Mo(1)—N(2)	78.0 (1)	N(1)—Mo(1)—C(1)	95.9 (1)
N(2)—Mo(1)—C(1)	94.0 (1)	N(1)—Mo(1)—C(2)	174.3 (1)
N(2)—Mo(1)—C(2)	96.4 (1)	C(1)—Mo(1)—C(2)	85.3 (1)
N(1)—Mo(1)—C(3)	96.1 (1)	N(2)—Mo(1)—C(3)	174.1 (1)
C(1)—Mo(1)—C(3)	86.4 (1)	C(2)—Mo(1)—C(3)	89.6 (1)
N(1)—Mo(1)—C(4)	93.8 (1)	N(2)—Mo(1)—C(4)	94.5 (1)
C(1)—Mo(1)—C(4)	168.3 (1)	C(2)—Mo(1)—C(4)	85.7 (1)
C(3)—Mo(1)—C(4)	86.0 (1)	Mo(1)—N(1)—C(5)	104.8 (2)
Mo(1)—N(1)—C(9)	112.4 (2)	C(5)—N(1)—C(9)	106.7 (2)
Mo(1)—N(1)—C(10)	114.1 (2)	C(5)—N(1)—C(10)	111.5 (3)
C(9)—N(1)—C(10)	107.3 (3)	Mo(1)—N(2)—C(6)	106.7 (2)
Mo(1)—N(2)—C(7)	112.0 (2)	C(6)—N(2)—C(7)	110.4 (3)
Mo(1)—N(2)—C(8)	113.1 (2)	C(6)—N(2)—C(8)	108.0 (3)
C(7)—N(2)—C(8)	106.5 (3)	Mo(1)—C(1)—O(1)	172.7 (3)
Mo(1)—C(2)—O(2)	179.6 (3)	Mo(1)—C(3)—O(3)	179.2 (2)
Mo(1)—C(4)—O(4)	171.3 (3)	N(1)—C(5)—C(6)	111.4 (3)
N(2)—C(6)—C(5)	111.0 (3)		

The data were collected with a variable scan speed of 5.33–29.3° min⁻¹ in ω . The structure was solved by direct methods and refined by full-matrix least squares. No correction for extinction was required. All H atoms were located from the difference Fourier map. *SHELXTL/PC* was used for all calculations.

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55620 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1027]

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Structures of 2-Amino-3-methylpyridinium Bis(1,2-dithiooxalato-*S,S'*)metallates(II) (*M* = Ni, Pd and Pt)

PASCUAL ROMÁN,* JAVIER I. BEITIA AND ANTONIO LUQUE

Departamento de Química Inorgánica, Universidad del País Vasco, Apartado 644, 48080 Bilbao, Spain

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Abstract

Three 2-amino-3-methylpyridinium salts of planar inorganic dithiooxalato anions, (C₆H₉N₂)₂[Ni(S₂C₂O₂)₂] (1), (C₆H₉N₂)₂[Pd(S₂C₂O₂)₂] (2) and (C₆H₉N₂)₂[Pt(S₂C₂O₂)₂] (3) have been prepared. Complexes (1) and (3) are isomorphous with (2) which crystallizes in the monoclinic space group *P*2₁/*c* with *Z* = 2 so that the anion straddles a crystallographic centre of symmetry. The crystal structure of (2) is built by mixed layers of quasi-planar complex [Pd(S₂C₂O₂)₂]²⁻ anions and (C₆H₉N₂)⁺ cations linked through electrostatic interactions, an extensive network of hydrogen bonds of types N—H...O and C—H...O, and weak π – π interactions. It is clear that the packing in this type of compound is affected by the cation shape and by the hydrogen-bonding network since it is the nature of the cation rather than the metal which determines the geometry of the interactions.

Comment

The structural studies of these metal complexes were undertaken in order to obtain a deeper insight into the effects of the cation and metal on the crystal packing, and the strength of the intermolecular interactions in compounds containing square-planar inorganic metal(II) 1,2-dithiooxalato-*S,S'* anions. The compounds (C₆H₉N₂)₂[*M*(S₂C₂O₂)₂] (*M* = Ni, Pd and Pt) were obtained by reaction of the potassium bis(dithiooxalato)metallate(II) salts, prepared according to the procedure of Cox, Wardlaw & Webster (1935), and 2-amino-3-methylpyridinium base in aqueous solution. Crystals were grown by the slow evaporation of an *N,N*-dimethylformamide solution of the complexes at room temperature. Table 1 summarizes the crystal data for the three compounds and shows that they are isostructural. Only the structure determination of the Pd complex was undertaken as a result of the poor quality of the crystals of the Ni compound and since all compounds are isostructural.

Table 1. Crystal data for compounds (1), (2) and (3)

	(1) ^a	(2) ^b	(3) ^a
Formula	C ₁₆ H ₁₈ N ₂ NiO ₄ S ₄	C ₁₆ H ₁₈ N ₂ O ₄ PdS ₄	C ₁₆ H ₁₈ N ₂ O ₄ PtS ₄
Formula weight	517.27	565.00	653.66
Space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> (Å)	5.744 (6)	5.750 (4)	5.756 (4)
<i>b</i> (Å)	16.20 (2)	16.229 (4)	16.234 (5)
<i>c</i> (Å)	11.74 (2)	11.728 (4)	11.769 (5)
β (°)	102.1 (2)	102.23 (1)	102.25 (2)
<i>V</i> (Å ³)	1068 (5)	1070 (1)	1074 (2)
<i>Z</i>	2	2	2
<i>D_s</i> (Mg m ⁻³)	1.61	1.75	2.02
<i>D_m</i> (Mg m ⁻³)	1.62 (1)	1.76 (1)	2.01 (1)

Notes: (a) X-ray powder diffraction data; (b) X-ray single-crystal diffraction data.

The asymmetric unit of the Pd compound comprises half of a centrosymmetric [Pd(S₂C₂O₂)₂]²⁻ anion and one crystallographically independent 2-amino-3-methylpyridinium cation. Atomic coordinates and equivalent isotropic temperature factors for the non-H atoms are given in Table 2. Fig. 1 shows the atomic numbering scheme used for the complex dianion. Selected bond lengths, bond angles and hydrogen contacts are listed in Table 3. The compound has a laminar structure which is built up by parallel layers of ions stacked along the [310] direction, following a sequence ...*acca*... (*a* = anion, *c* = cation). These layers are located in two different *z* levels, *z* = 0 and *z* = $\frac{1}{2}$, and stepped by *b*/2. The best

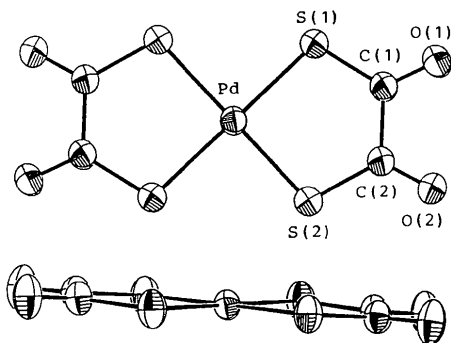


Fig. 1. Front and side views of the bis(dithiooxalato)palladate(II) anion showing the 50% probability thermal ellipsoids and the atom-labeling scheme.

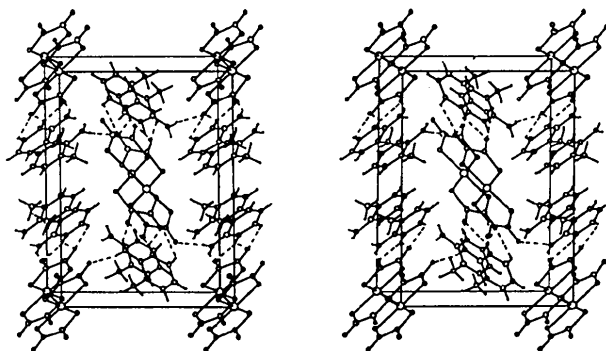


Fig. 2. Stereo packing diagram for (2); **a** is out of the plane of paper, **b** is vertical and **c** is horizontal.

planes of the anion and cation are almost parallel to each other with a dihedral angle of 2.29 (1)°. A stereoscopic view of the unit cell together with the network of hydrogen contacts (dotted lines) is shown in Fig. 2. The neighbouring cations and anions are linked in the same layer by means of one asymmetric bifurcated hydrogen bond between the *endo* pyridine N atom, N(11), and two O atoms of the dithiooxalate ligand, and by a hydrogen bond which links one of the H atoms of the *exo* amine group and O(1¹). The layers at different *z* levels are connected by means of an N—H...O hydrogen bond involving the second H atom of the *exo* amine group and a dithiooxalate ligand in a neighbouring layer. There is also a weak C—H...O hydrogen contact between ions in the same sheet. There is no doubt that not all the C—H...O contacts can be considered as hydrogen bonds, but they do indicate some degree of polarization. The role of this type of interaction in a series of organic and inorganic compounds has also been examined by Desiraju (1991) and Taylor & Kennard (1982).

An interesting structural feature is the interstack distances between anions and cations (Fig. 3). There is some degree of overlapping between the dithiooxalate ligands (with a significant degree of delocalization) and the cations. The anion–cation interstack

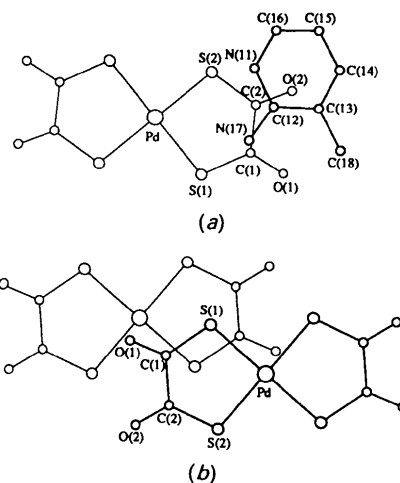


Fig. 3. View of the overlap: (a) cation–anion and (b) anion–anion.

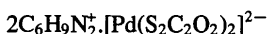
distance ranges from 3.30–3.45 Å and the shortest contact, between C(1) and N(17), is 3.354 (4) Å. These values are comparable to interplanar distances found in aromatic π systems (3.4–3.6 Å) with strong π – π interactions. A similar overlapping is established between the dithiooxalate ligands belonging to neighbouring anions. However, the slightly long interplanar distance (3.7 Å) suggests the existence of a very weak interaction. This type of anion–anion interaction is observed in other structures of similar compounds (Enjalbert, Gleizes & Galy, 1987).

In the anion, the Pd—S bond lengths are in the range found for other complexes (Frasse, Trombe, Gleizes & Galy, 1985). The short average C—S bond distances (1.72 Å) and the long average C=O bond (1.23 Å) are consistent with the existence of a significant degree of delocalization throughout the S—C—O units in the dianion (Coucouvani, Baezinger & Johnson, 1973), with a single C(1)—C(2) bond of 1.550 (3) Å. The centrosymmetric anion is slightly nonplanar, a chair conformation is present with an angle of 2.69 (4)° between the PdS₄ group and the S₂C₂O₂ planes of the ligands. This angle is at the lower end of the range previously observed for similar bis(dithiooxalato)metallate(II) anions in compounds also containing aromatic cations (Román, Beitia, Luque & Gutiérrez-Zorrilla, 1992; Enjalbert, Gleizes & Galy, 1987), and is considerably lower than those observed in analogous complexes containing large and bulky cations (Román, Gutiérrez-Zorrilla, Luque, Beitia & Guzmán-Miralles, 1991; Román, Luque, Gutiérrez-Zorrilla & Beitia, 1992). The value of this angle is affected by a combination of factors, including the contacts between the O atoms and the H atoms of cations, as well as the π — π interactions between the aromatic cations and the dithiooxalate groups (Román, Beitia, Luque & Gutiérrez-Zorrilla, 1992). Both may produce a displacement of the ligand atoms and a higher value of the dihedral angle between the MS₄ and the dithiooxalate groups. In our case, the high planarity of the complex anion is due to the fact that most of the hydrogen contacts are stabilized by nearly parallel ions located in the same plane and the non-existence of strong π — π interactions. The bond angles and distances of the planar 2-amino-3-methylpyridinium cation are within the normal range reported in the literature (Palace & Willett, 1987) and its packing appears to be largely dictated by electrostatic forces and the hydrogen-bonding constraints described above.

Experimental

Compound (2)

Crystal data



$M_r = 565.0$

Monoclinic

$P2_1/c$

$a = 5.750$ (4) Å

$b = 16.229$ (4) Å

$c = 11.728$ (4) Å

$\beta = 102.23$ (1)°

$V = 1070$ (1) Å³

$Z = 2$

$D_x = 1.754$ Mg m⁻³

$D_m = 1.76$ (1) Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å

Cell parameters from 25 reflections

$\theta = 12$ – 20°

$\mu = 1.261$ mm⁻¹

$T = 295$ (2) K

Prism

$0.30 \times 0.25 \times 0.15$ mm

Yellow

Data collection

CAD-4 diffractometer

ω — 2θ scans

Absorption correction:

empirical (DIFABS:

Walker & Stuart, 1983)

$T_{\min} = 0.825$, $T_{\max} = 1.174$

6543 measured reflections

6543 independent reflections

4257 observed reflections

$[I > 3\sigma(I)]$

$\theta_{\max} = 40^\circ$

$h = 0 \rightarrow 10$

$k = 0 \rightarrow 29$

$l = -21 \rightarrow 21$

3 standard reflections

frequency: 120 min

intensity variation: <2%

Refinement

Refinement on F

Final $R = 0.050$

$wR = 0.064$

$S = 1.102$

4257 reflections

169 parameters

All H-atom parameters refined

$(\Delta/\sigma)_{\max} = 0.11$

$\Delta\rho_{\max} = 0.85$ e Å⁻³

$\Delta\rho_{\min} = -0.71$ e Å⁻³

Extinction correction: none

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 2. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²) for (2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j \cdot \cos(\mathbf{a}_i \cdot \mathbf{a}_j)$$

	x	y	z	U_{eq}
Pd	0†	0†	0†	0.0361 (2)
S(1)	0.34296 (8)	0.05021 (6)	0.11979 (6)	0.0458 (3)
S(2)	0.03191 (8)	0.09769 (6)	-0.13777 (6)	0.0476 (3)
C(1)	0.4403 (3)	0.1274 (2)	0.0413 (2)	0.0407 (7)
C(2)	0.2943 (3)	0.1496 (2)	-0.0817 (2)	0.0402 (6)
O(1)	0.6208 (3)	0.1685 (2)	0.0775 (2)	0.0573 (8)
O(2)	0.3732 (3)	0.2055 (2)	-0.1330 (2)	0.0561 (7)
N(11)	-0.1694 (3)	0.2937 (2)	-0.0433 (2)	0.0428 (6)
C(12)	0.0289 (3)	0.3077 (2)	0.0402 (2)	0.0391 (7)
C(13)	0.1868 (4)	0.3723 (2)	0.0228 (3)	0.0485 (9)
C(14)	0.1248 (4)	0.4164 (2)	-0.0790 (3)	0.0630 (11)
C(15)	-0.0821 (5)	0.3999 (2)	-0.1640 (3)	0.0635 (12)
C(16)	-0.2279 (5)	0.3381 (2)	-0.1439 (3)	0.0547 (10)
N(17)	0.0693 (3)	0.2588 (2)	0.1335 (2)	0.0489 (7)
C(18)	0.4105 (6)	0.3878 (3)	0.1139 (4)	0.0692 (12)

† Parameter fixed.

Table 3. Selected bond lengths (Å), bond angles (°) and hydrogen contacts (Å, °) for (2)

Bis(dithiooxalato)palladate(II) anion

Pd—S(1)	2.314 (1)	C(1)—C(2)	1.550 (3)
Pd—S(2)	2.299 (1)	C(1)—O(1)	1.232 (3)
S(1)—C(1)	1.717 (3)	C(2)—O(2)	1.229 (4)
S(2)—C(2)	1.731 (2)		
S(1)—Pd—S(2)	89.96 (3)	S(1)—C(1)—O(1)	123.9 (2)
Pd—S(1)—C(1)	105.32 (9)	S(2)—C(2)—O(2)	123.8 (2)
Pd—S(2)—C(2)	105.15 (9)	C(1)—C(2)—O(2)	116.4 (2)
S(1)—C(1)—C(2)	119.6 (2)	C(2)—C(1)—O(1)	116.4 (2)
S(2)—C(2)—C(1)	119.8 (2)		

2-Amino-3-methylpyridinium cation

N(11)—C(12)	1.355 (3)	C(15)—C(16)	1.359 (5)
C(12)—C(13)	1.431 (4)	C(16)—N(11)	1.362 (4)
C(13)—C(14)	1.372 (5)	C(12)—N(17)	1.331 (4)
C(14)—C(15)	1.407 (4)	C(13)—C(18)	1.509 (4)
C(12)—N(11)—C(16)	123.6 (2)	C(15)—C(16)—N(11)	119.5 (3)
N(11)—C(12)—C(13)	118.8 (2)	N(11)—C(12)—N(17)	118.0 (2)
C(12)—C(13)—C(14)	116.8 (2)	C(13)—C(12)—N(17)	123.3 (2)
C(13)—C(14)—C(15)	122.8 (3)	C(12)—C(13)—C(18)	119.5 (3)
C(14)—C(15)—C(16)	118.5 (3)	C(14)—C(13)—C(18)	123.7 (3)

X—H···O	X—H	X···O	H···O	X—H···O
N(11)—H(11)···O(1) ⁱ	0.91 (4)	2.884 (4)	2.06 (5)	150 (3)
N(11)—H(11)···O(2) ⁱ	0.91 (4)	2.984 (3)	2.28 (3)	135 (4)
N(17)—H(172)···O(1) ⁱ	0.87 (4)	2.917 (3)	2.13 (3)	149 (4)
N(17)—H(171)···O(2) ⁱⁱ	0.79 (4)	2.976 (3)	2.21 (4)	164 (4)
C(16)—H(16)···O(2) ⁱ	0.98 (3)	3.167 (4)	2.56 (4)	121 (3)

Symmetry codes: (i) $-1 + x, y, z$; (ii) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

The densities of the compounds were measured by flotation in a $\text{CHBr}_3/n\text{-C}_6\text{H}_{14}$ mixture. Intensity data were corrected for Lorentz and polarization effects. Only a unique data set was collected. The structure was solved by Patterson methods and subsequent Fourier synthesis maps. The data refined with isotropic displacement parameters were corrected for absorption. A weighting scheme of type $w = w_1, w_2$ with $w_1 = k_1/(a + b|F_o|)^2$ and $w_2 = k_2/(c + d\sin\theta/\lambda + e\sin^2\theta/\lambda)$ was used to obtain flat dependence in $\langle w\Delta^2 F \rangle$ versus $\langle F_o \rangle$ and versus $\langle \sin\theta/\lambda \rangle$ (PESOS; Martínez-Ripoll & Cano, 1975). The coefficients used were $k_1 = 0.403$; $k_2 = 0.792$; $a = 1.369$, $b = -0.518$ for $|F_o| \leq 1.15$; $a = 0.766$, $b = -0.098$ for $1.15 < |F_o| \leq 3.07$; $a = 0.463$, $b = -0.026$ for $3.07 < |F_o| \leq 5.37$; $a = 0.188$, $b = 0.031$ for $5.37 < |F_o| \leq 21.58$; $a = -0.143$, $b = 0.050$ for $|F_o| > 21.58$; $c = 7.986$, $d = -35.299$, $e = 40.918$ for $\sin\theta/\lambda \leq 0.54$; $c = 2.106$, $d = -2.380$, $e = 0.000$ for $0.54 < \sin\theta/\lambda \leq 0.61$; $c = -5.201$, $d = 9.467$, $e = 0.000$ for $0.61 < \sin\theta/\lambda \leq 0.73$; $c = 0.752$, $d = -0.906$, $e = 0.746$ for $\sin\theta/\lambda > 0.73$. The non-H atoms were refined anisotropically. All H atoms were clearly visible in a difference Fourier synthesis and were refined isotropically. Most calculations were carried out using the XRAY76 system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) running on a MicroVAX II computer.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55686 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1013]

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Acta Cryst. (1993). **C49**, 587–589

Chlorobis(*N*-phenylsalicylideneaminato-*O,N*)manganese(III): a Manganese Schiff-Base Complex Derived by an Electrochemical Route

C. A. MCAULIFFE AND R. G. PRITCHARD

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

L. LUACES, J. A. GARCIA-VAZQUEZ, J. ROMERO, M. R. BERMEJO AND A. SOUSA

Departamento de Química Inorgánica, Universidad de Santiago, Santiago de Compostela, 15706 Santiago, Spain

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Abstract

The five-coordinate manganese cation is sited at the centre of an approximate trigonal bipyramid. Distortions involving, primarily, the axial N atom [N—Mn—N 169.41(9), Cl—Mn—N 98.13(7), 91.57(8)°] accommodate a ligand bite of less than 90° [N—Mn—O 86.8(1), 86.5(1)°] and a bulky equatorial Cl atom [Mn—Cl 2.235(1) Å].

Comment

The chemistry of manganese has attracted considerable attention recently as a result of its important role in biological processes such as photosynthesis (Ashmaw, McAuliffe, Parish & Tames, 1985). This interest has led to structural investigations of several penta-coordinate manganese(III) Schiff-base complexes including [Mn(acen)Cl], acen = *N,N'*-ethylenebis(acetylacetone imine), (Boucher & Day, 1977), [Mn(salen)Cl], salen = *N,N'*-ethylenebis(salicylaldiminato), (Pecoraro & Butler, 1986), [Mn(salen)(*p*-nitrobenzenethiolato)] (Gohdes & Armstrong, 1988), {Mn(salen)[2-(3-oxobut-1-enyl)phenolate]} (Li & Pecoraro, 1989) and [Mn(tetram-salen)Cl] (Oki & Hodgson, 1990). Unlike the title molecule, where the absence of an *N,N'* ethyl linkage