

rencontrées dans le *cis*-dichloro(diméthylsulfoxyde)(picoline-2)platine(II) (Melanson & Rochon, 1977) [2,06 (1) Å] et dans le trichloro(diméthylsulfoxyde)platinate(II) de bis(propanediamine-1,3-*N,N'*)-platine(II) (2/1) (Viossat, Toffoli, Khodadad & Rodier, 1988) [2,04 (1) et 2,06 (1) Å].

Le long de la droite $\text{Na}\cdots\text{Na}^{\text{iv}}$ [(iv): 1 + x , y , z], ou dans son voisinage immédiat, on rencontre successivement, dans le sens où x croît, les positions ... $\text{O}(20^{\text{ii}})$, $\text{O}'(20^{\text{ii}})$, Na, $\text{O}(20)$, $\text{O}'(20)$, Na^{iv} , $\text{O}(20^{\text{iv}})$, $\text{O}'(20^{\text{iv}})$, Les distances $\text{Na}-\text{O}(20)$ [1,87 (2) Å] et $\text{Na}-\text{O}'(20^{\text{ii}})$ [1,80 (2) Å] interdisent l'occupation de $\text{O}(20)$ et de $\text{O}'(20^{\text{ii}})$ lorsque l'atome de sodium est présent et inversement. Dans cette direction, Na peut avoir pour proches voisins $\text{O}(20^{\text{ii}})$ et $\text{O}'(20)$ qui en sont distants respectivement de 2,36 (2) et de 2,37 (2) Å. Mais si $\text{O}'(20)$ est occupé, Na^{iv} est vide. Dans cette hypothèse, on rencontre ensuite des atomes sur $\text{O}(20^{\text{iv}})$, Na^{vii} et $\text{O}'(20^{\text{vii}})$ [(vii): 2 + x , y , z]. $\text{O}(19)$ et $\text{O}'(19)$ ne peuvent être occupées simultanément. Il en est de même pour $\text{O}(21^{\text{i}})$ et $\text{O}'(21^{\text{i}})$ distants seulement de 0,65 (3) Å. L'environnement de Na comprend donc les atomes d'oxygène présents sur $\text{O}(20^{\text{ii}})$, $\text{O}'(20)$, $\text{O}(8^{\text{i}})$, $\text{O}(18^{\text{i}})$, $\text{O}(19)$ ou $\text{O}'(19)$, $\text{O}(21^{\text{i}})$ ou $\text{O}'(21^{\text{i}})$, soit six atomes qui réalisent un octaèdre déformé. Les distances $\text{Na}-\text{O}(19)$ [2,26 (2) Å] et $\text{Na}-\text{O}(21^{\text{i}})$ [2,43 (2) Å] sont plus courtes respectivement que $\text{Na}-\text{O}'(19)$ [2,63 (3) Å] et $\text{Na}-\text{O}'(21^{\text{i}})$ [2,71 (3) Å]. Elles sont donc associées à des interactions plus fortes que les dernières. Autrement dit, dans le voisinage de Na, la présence d'un atome d'oxygène est plus probable en $\text{O}(19)$ et en $\text{O}(21^{\text{i}})$ qu'en $\text{O}'(19)$ et en $\text{O}'(21^{\text{i}})$.

Le même raisonnement peut être répété à propos de Na' . Le long de la droite $\text{Na}'-\text{Na}^{\text{iv}}$ ou dans son voisinage et dans le sens où x est croissant, se trouvent les positions ... $\text{O}(21^{\text{ii}})$, $\text{O}'(21^{\text{ii}})$, Na' , $\text{O}(21)$, $\text{O}'(21)$, Na^{iv} , Si Na' est occupé, $\text{O}'(21^{\text{ii}})$ et $\text{O}(21)$

sont vides [$\text{Na}'-\text{O}'(21^{\text{ii}}) = 1,87$ (2) Å et $\text{Na}'-\text{O}(21) = 1,77$ (2) Å]. Donc, l'atome de Na a pour proches voisins $\text{O}(21^{\text{ii}})$ et $\text{O}'(21)$ qui en sont distants, le premier de 2,40 (2) Å et le second de 2,35 (2) Å. Autour de Na' , on trouve aussi les atomes d'oxygène présents sur $\text{O}(9^{\text{i}})$, $\text{O}(19)$ ou $\text{O}'(19)$, $\text{O}(20^{\text{iii}})$ ou $\text{O}'(20^{\text{iii}})$. L'ensemble réalise une bipyramide à base trigonale dont les sommets sont $\text{O}(21^{\text{ii}})$ et $\text{O}'(21)$. La comparaison de $\text{Na}'-\text{O}'(19)$ [2,37 (3) Å] et de $\text{Na}'-\text{O}(20^{\text{iii}})$ [2,51 (3) Å] avec respectivement $\text{Na}'-\text{O}(19)$ [2,70 (3) Å] et $\text{Na}'-\text{O}'(20^{\text{iii}})$ [2,67 (3) Å] indique que, dans l'entourage de Na' , les occupations de $\text{O}'(19)$ et de $\text{O}(20^{\text{iii}})$ sont plus probables que celles de $\text{O}(19)$ et de $\text{O}'(20^{\text{iii}})$.

De nombreuses distances interatomiques intermoléculaires $\text{O}\cdots\text{O}$ sont susceptibles de correspondre à des liaisons hydrogène. Toutes font intervenir au moins un atome d'oxygène d'une molécule d'eau. Elles ont été rapportées dans le Tableau 2 lorsqu'elles sont inférieures à 3,00 Å.

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Structure of Bis(amidinothiourea)palladium Chloride

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Abstract. $[\text{Pd}(\text{SC}_2\text{N}_4\text{H}_6)_2]\text{Cl}_2 \cdot 1.5\text{H}_2\text{O}$, $M_r = 440.64$, monoclinic, $P2_1/n$, $a = 9.401$ (2), $b = 13.540$ (2), $c = 12.172$ (2) Å, $\beta = 101.15$ (1)°, $V = 1520.1$ (5) Å³, $D_x = 1.925$ g cm⁻³, $Z = 4$, $F(000) = 876$, $\text{Mo K}\alpha$, $\lambda =$

0.7107 Å, $\mu = 1.83$ mm⁻¹, room temperature, $R = 0.065$ for 2582 observed reflections. The structure analysis has shown that the Pd atom is *cis*-planar coordinated by two S and two N atoms from two

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ligand molecules. The structure is composed of $[\text{Pd}(\text{SC}_2\text{N}_4\text{H}_6)_2]^{2+}$ and Cl^- ions held together by a network of $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds. The water molecules are connected to the complex cation through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. One of the Cl atoms and one water molecule are found to be in a disordered state.

Introduction. Few investigations have so far been reported on the synthesis and structure of metal complexes of 1-amidino-2-thiourea. Several new metal complexes of this ligand have recently been isolated and their electronic, vibrational and PMR spectral studies *etc.* have been reported (Saha & Roy, 1983). The ligand possesses nitrogen and sulfur donors and the relative coordinating abilities of these donor atoms depend on experimental conditions and the nature of the coordinating metal atom. As SN donors, they stabilize lower oxidation states of metal atoms (Roy & Saha, 1980) and lower the electron density at NO in metal nitrosyls (Roy & Saha, 1980). A detailed structural analysis of the palladium complex of this ligand has been undertaken in order to ascertain the structure proposed as most probable by Saha & Roy (1983) on the basis of its physico-chemical properties and the results are illustrated in the present paper.

Experimental. The ligand 1-amidino-2-thiourea was prepared according to the method described by Ray & Choudhury (1950). The complex under study was obtained by adding an aqueous solution of the ligand (0.01 mol) to that of PdX_2 (0.001 mol, $X = \text{Cl}, \text{Br}$) at 323–333 K. The resulting solution was filtered. On evaporating the filtrate, light brown crystals were obtained. D_m not determined. Crystal $0.35 \times 0.23 \times 0.20$ mm. Enraf–Nonius CAD-4 diffractometer. 2658 unique reflections. $\omega/2\theta$ scan mode; θ from 2 to 25° ($h = -11$ – 11 , $k = 0$ – 16 , $l = 0$ – 14); variable scan speeds with maximum scan time 60 s. Cell dimensions and e.s.d.'s from least-squares analysis of setting angles for 25 reflections with $15 < \theta < 17^\circ$. Intensities of three reflections, 219, 095, 582, monitored after every hour of exposure showed no crystal decomposition. Max. value of $2\theta = 50^\circ$. Only Lorentz and polarization correction applied. 2582 reflections with $I_o > 2.5\sigma(I_o)$. Intensities placed on absolute scale by Wilson's method. Extinction conditions indicated the space group to be either Pn or $P2/n$. Initially the structure was solved in Pn , by three-dimensional Patterson and successive Fourier synthesis. The final agreement values with 38 anisotropically refined non-H atoms and 32 unrefined H atoms were $R = 0.052$, $wR = 0.046$. In the final cycle of refinement $(\Delta/\sigma)_{\text{max}} = 0.947$, maximum peak on final difference map $0.9 \text{ e } \text{Å}^{-3}$.

The structure was also solved in $P2/n$, as it was found that when the origin is shifted by $x = -0.037$, $y = 0$, $z = -0.025$, all the atoms of the two independent molecules in the asymmetric unit could be related centrosymmetrically. After applying proper shifts to the previously obtained coordinates in Pn , the structure was then refined by full-matrix least-squares, minimizing $\sum w(\Delta F)^2$ [*SHELX76* (Sheldrick, 1976)], initially with isotropic and then anisotropic thermal parameters for non-H atoms. At the final stage of refinement, a difference Fourier map showed a residual peak of considerable strength, about $3.4 \text{ e } \text{Å}^{-3}$, which led us to conclude that the structure is a disordered one. Several models with disordered chlorine and water molecules were considered. Due to unsatisfactory thermal parameters and relatively high R values, all the models, except the following one, were discarded. In this model the site occupancy factors (s.o.f.) of the disordered Cl atom and that of the O atom of one of the water molecules were refined from an initial value of 0.5, constraining the sum of the s.o.f. of the Cl atoms to unity. All the H atoms, except those of the disordered water molecule, were located in a difference Fourier synthesis. The positions of the H atoms were not refined and their individual temperature factors (U) were set equal to 0.05 Å^2 . The final agreement values with 20 anisotropically refined non-H atoms and 14 H atoms were $R = 0.065$, $wR = 0.086$, $w = 1/\sigma^2$. In the final cycle of refinement $(\Delta/\sigma)_{\text{max}} = 0.652$, maximum peak on the final difference map $1.3 \text{ e } \text{Å}^{-3}$ near one of the disordered Cl atoms. Atomic scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computer programs used were those of the *XRAYARC* program package (Vickery, Bright & Mallinson, 1971), and *PARST* (Nardelli, 1983).

Discussion. Atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1 and Table 2 contains the interatomic distances and angles.* These data refer to the structure refined in the space group $P2/n$ as this gave the most satisfactory results. Fig. 1 shows a perspective view of the asymmetric unit (consisting of one molecule) and the numbering scheme of the molecule. Fig. 2 gives the packing diagram of the molecule viewed down the a axis.

The ligand is *S,N*-coordinating to Pd, the N atom being the imino nitrogen. The Pd—S distances are somewhat shorter than the sum of the covalent radii

* Lists of structure factors, anisotropic thermal parameters, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53039 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates for non-H atoms ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^4$)

	x	y	z	U_{eq}
Pd	958 (1)	1456 (0)	-3525 (0)	335 (0)
S1	257 (2)	18 (1)	-2866 (2)	439 (1)
S2	-362 (3)	2131 (2)	-2348 (2)	617 (1)
N1	2074 (8)	765 (5)	-4555 (6)	404 (2)
N2	1469 (8)	2800 (5)	-4114 (7)	508 (2)
N3	505 (12)	3915 (5)	-2982 (7)	611 (3)
N4	1838 (7)	-928 (5)	-4227 (6)	365 (2)
N5	3237 (10)	-425 (7)	-5465 (7)	595 (3)
N6	1726 (11)	4478 (7)	-4298 (7)	614 (3)
N7	-753 (17)	3849 (10)	-1558 (10)	1074 (7)
N8	594 (8)	-1806 (5)	-3186 (7)	475 (2)
C1	1190 (11)	3685 (7)	-3798 (9)	473 (3)
C2	-212 (12)	3344 (8)	-2334 (8)	573 (3)
C3	2419 (9)	126 (6)	-4716 (7)	427 (2)
C4	969 (8)	-971 (8)	-3479 (6)	547 (3)
C11	7638 (3)	3112 (2)	359 (2)	646 (1)
O1	548 (10)	5945 (5)	7370 (7)	721 (3)
ClA*	3557 (8)	1898 (3)	3713 (6)	849 (2)
ClB†	2665 (7)	3976 (4)	3495 (5)	645 (2)
O2‡	3904 (27)	4147 (12)	4269 (16)	964 (10)

* S.o.f. 0.55.

† S.o.f. 0.45.

‡ S.o.f. 0.50.

Table 2. Intramolecular distances (\AA) and angles ($^\circ$)

Pd—S1	2.253 (2)	Pd—S2	2.261 (3)
Pd—N1	2.015 (8)	Pd—N2	2.047 (7)
S1—C4	1.729 (10)	S2—C2	1.648 (11)
C4—N4	1.337 (11)	C2—N3	1.372 (15)
N4—C3	1.399 (11)	N3—C1	1.321 (15)
C3—N1	1.275 (11)	C1—N2	1.301 (12)
C4—N8	1.257 (12)	C2—N7	1.343 (18)
C3—N5	1.363 (13)	C1—N6	1.377 (14)
S1—Pd—S2	83.7 (1)	N1—Pd—N2	90.5 (3)
S1—Pd—N1	92.5 (2)	S2—Pd—N2	93.3 (2)
Pd—S1—C4	110.5 (3)	Pd—S2—C2	110.6 (4)
Pd—N1—C3	135.5 (6)	Pd—N2—C1	129.9 (7)
S1—C4—N4	126.8 (8)	S2—C2—N3	127.4 (8)
C4—N4—C3	131.6 (7)	C2—N3—C1	131.6 (8)
N4—C3—N1	122.6 (8)	N3—C1—N2	126.5 (9)
S1—C4—N8	114.9 (6)	S2—C2—N7	118.1 (9)
N4—C4—N8	118.3 (7)	N3—C2—N7	114.3 (10)
N4—C3—N5	111.8 (7)	N3—C1—N6	115.0 (9)
N1—C3—N5	125.2 (8)	N2—C1—N6	118.4 (9)

(2.36 \AA) of Pd and S atoms (Pauling, 1960) but they are comparable with previously obtained values in compounds like $\text{Pd}(\text{dtb}_2)$ ($\text{dtb}_2 = \text{dithiobinzeno}$) (Girling & Amma, 1968) and $\text{Pd}[\text{SC}(\text{NH}_2)_2]_4\text{Cl}_2$ (Berta, Spofford, Boldrini & Amma, 1970). The values of Pd—N bonds lie in the normal range as found in other related structures. The shortness of the C—N bonds (1.27–1.40 \AA) indicates the presence of multiple bonding with delocalization of π electrons. The inequivalence of the bonds can be explained qualitatively in terms of the difference in degree of sp^2 character in the different atoms. The N atoms (N1 and N2) are expected to have maximum sp^2 character and hence the C—N bonds involving them are the shortest (Creitz, Gsell & Wampler,

1969), (Moucharafieh, Eller, Bertrand & Royer, 1978; Saha & Roy, 1983).

From the mean plane calculations it has been observed that the six-membered rings on either side of the Pd atom are almost planar. Also, the four (the two S and two N) atoms surrounding the Pd atom indicate that the Pd atom has a square planar coordination. The Cl atoms do not participate in metal coordination and the structure consists of discrete $[\text{Pd}(\text{SC}_2\text{N}_4\text{H}_6)_2]^{2+}$ and Cl^- ions.

The hydrogen bonds are shown in Fig. 2 by dotted lines and Table 3 gives the detailed hydrogen bonding scheme.

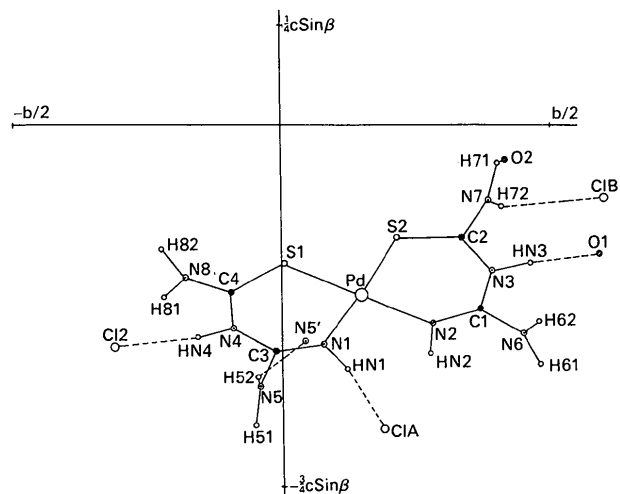
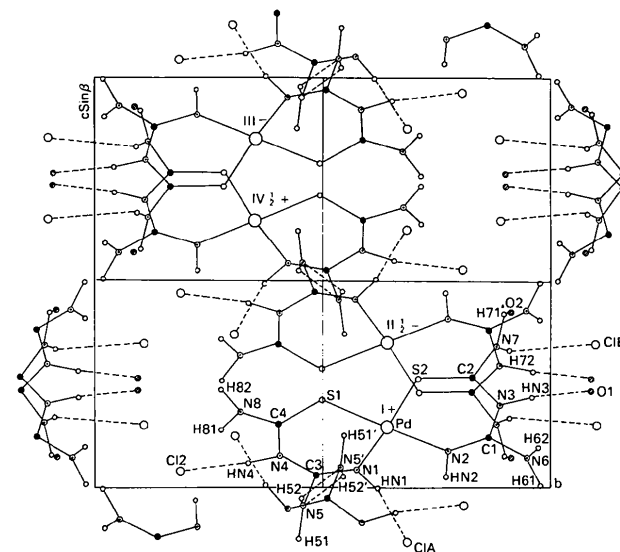
Fig. 1. Asymmetric unit of the structure (with atomic labelling) projected down the a axis.Fig. 2. Packing diagram of the molecule viewed down the a axis. Hydrogen bonds are shown as dotted lines. Symmetry code: (I) x, y, z ; (II) $\frac{1}{2} - x, y, \frac{1}{2} - z$; (III) $-x, -y, -z$; (IV) $\frac{1}{2} + x, -y, \frac{1}{2} + z$.

Table 3. *Geometry of the possible hydrogen bonds, distances in (Å) and angles in (°) with e.s.d.'s in parentheses*

D—H...A	D...A	H...A	D—H...A
N1—H1...Cl(A ¹)	3.14 (1)	2.16 (10)	166 (4)
N3—H3...O(1 ^b)	2.78 (1)	1.77 (10)	174 (6)
N4—H4...Cl(1 ^b)	3.12 (1)	2.11 (3)	167 (4)
N5—H52...N(5 ^{cd})	3.48 (1)	2.57 (3)	147 (6)
N7—H71...O(2 ^g)	3.01 (2)	2.05 (19)	164 (11)
N7—H72...Cl(B ^g)	3.31 (2)	2.64 (6)	122 (8)
O1—H101...Cl(1 ^h)	3.22 (1)	2.24 (2)	170 (4)
O1—H201...Cl(B ^{hi})	3.00 (1)	2.33 (6)	130 (5)
O1—H201...Cl(B ^{mi})	3.27 (1)	2.49 (7)	143 (5)

Symmetry code: (i) $+x, +y, +z - 1$; (ii) $+x - \frac{1}{2}, -y, +z - \frac{1}{2}$; (iii) $-x + 1, -y, -z - 1$; (iv) $-x + \frac{1}{2}, +y, -z + \frac{1}{2}$; (v) $+x - \frac{1}{2}, -y + 1, +z - \frac{1}{2}$; (vi) $1 - x, 1 - y, 1 - z$; (vii) $-x, 1 - y, 1 - z$; (viii) $x - \frac{1}{2}, -y, z + \frac{1}{2}$.

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Structure of Bis- μ -(2-quinolinecarboxylato-*O,O,O'*)bis[triaqua-(2-quinolinecarboxylato-*N,O*)(2-quinolinecarboxylato-*O*)neodymium(III)] Trihydrate

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Abstract. $[\{\text{Nd}(\text{C}_{10}\text{H}_6\text{NO}_2)_2(\text{H}_2\text{O})_3\}_2(\mu\text{-C}_{10}\text{H}_6\text{NO}_2)_2] \cdot 3\text{H}_2\text{O}$, $M_r = 1483.6$, triclinic, $P\bar{1}$, $a = 15.760$ (9), $b = 8.178$ (6), $c = 24.153$ (19) Å, $\alpha = 92.54$ (6), $\beta = 99.19$ (6), $\gamma = 109.40$ (6)°, $V = 2883$ (4) Å³ [reduced cell: $a = 8.178$ (6), $b = 15.154$ (12), $c = 24.153$ (19) Å, $\alpha = 79.05$ (7), $\beta = 87.46$ (6), $\gamma = 78.80$ (6)°], $Z = 2$, $D_m = 1.70$, $D_x = 1.709$ (2) Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.88$ mm⁻¹, $F(000) = 1488$, $T = 302$ (1) K, final $R = 0.047$ for 5110 reflections. The title compound is a dimer; the two Nd ions are bridged by two carboxyl groups. Each Nd ion is surrounded by one N atom, five carboxylic oxygens and three water molecules. Very short Nd—O (carboxyl) bonds [2.320 (7) and 2.325 (7) Å] were found.

Introduction. This work is part of our study on the structures and properties of complexes containing lanthanide–nitrogen bonds.

Experimental. The title compound was prepared by mixing aqueous solutions of NdCl₃ and 2-quinolinecarboxylic (quinaldic) acid, and then adding a dilute solution of *N*-2-hydroxyethylpiperazine-*N'*-ethanesulfonic acid (HEPES). After a few hours tiny violet plates were formed. Two specimens (0.3 × 0.4 × 0.5 mm and 0.3 × 0.3 × 0.4 mm) were cut from larger crystals. D_m by flotation in C₂H₄Br₂/CHCl₃. Oscillation and Weissenberg photographs showed no symmetry, thus indicating the triclinic system. The structure was successfully refined in space group $P\bar{1}$. Syntex P2₁ diffractometer, Mo $K\alpha$ radiation for lattice parameters (14 reflections, $21 < 2\theta < 26^\circ$), variable $\theta/2\theta$ scan, $4 < 2\theta < 45^\circ$, two standards every 100 reflections, maximum variation from means 7.1%, 4541 and 1281 intensities respectively measured from the two specimens, 166 common ones used for determination of the scale ratio between the two sets, $R_{\text{int}} = 0.028$, after averaging 5110 unique reflec-