

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: empirical via ψ scans (North *et al.*, 1968)
 $T_{\min} = 0.779$, $T_{\max} = 0.875$
 6020 measured reflections
 5765 independent reflections

4410 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 $\theta_{\text{max}} = 25^\circ$
 $h = 0 \rightarrow 15$
 $k = 0 \rightarrow 16$
 $l = -20 \rightarrow 20$
 2 standard reflections every 100 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.077$
 $S = 1.218$
 5765 reflections
 388 parameters
 H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0120P)^2 + 4.1072P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.454 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.321 \text{ e } \text{\AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

Sn—O2	2.364 (3)	P1—C2A	1.795 (4)
Sn—O1	2.394 (3)	P1—C3A	1.796 (4)
Sn—Cl1	2.452 (1)	P1—C1A	1.804 (4)
Sn—Cl2	2.480 (1)	P2—C5A	1.797 (4)
O1—P1	1.489 (3)	P2—C6A	1.798 (4)
O2—P2	1.495 (3)	P2—C4A	1.799 (4)
O2—Sn—O1	164.78 (11)	O1—P1—C3A	111.2 (2)
O2—Sn—Cl1	85.22 (8)	O1—P1—C1A	111.0 (2)
O1—Sn—Cl1	84.35 (9)	O2—P2—C5A	111.8 (2)
O2—Sn—Cl2	84.83 (9)	O2—P2—C6A	110.9 (2)
O1—Sn—Cl2	85.44 (9)	O2—P2—C4A	110.1 (2)
Cl1—Sn—Cl2	96.78 (5)	C2F—C2A—C2B	118.4 (4)
P1—O1—Sn	167.2 (2)	C4B—C4A—C4F	118.2 (4)
P2—O2—Sn	146.8 (2)	C5D—C5E—C5F	121.2 (5)
O1—P1—C2A	112.3 (2)		

The title structure was solved by conventional Patterson and Fourier methods. The H atoms were located from a difference Fourier map. In the final refinement, their positions and isotropic displacement parameters (0.05 \AA^2) were fixed.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1080). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). **C54**, 906–909

Platinum Complexes of 1,4,7-Trithiacyclodecane

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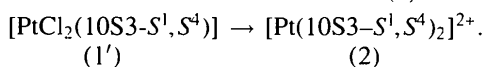
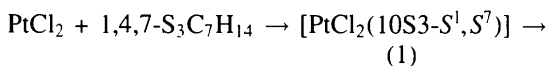
(Received 23 June 1997; accepted 23 October 1997)

Abstract

A neutral Pt^{II} complex, dichloro(1,4,7-trithiacyclodecane-*S*¹,*S*⁷)platinum, [PtCl₂(C₇H₁₄S₃)], and a hydrated salt of a cationic Pt^{II} complex, bis(1,4,7-trithiacyclodecane-*S*¹,*S*⁴)platinum dichloride heptahydrate, [Pt(C₇H₁₄S₃)₂]Cl₂·7H₂O, have been studied. The ten-membered heterocyclic ligand, 1,4,7-trithiacyclodecane (10S3), forms a six-membered chelate ring in the neutral complex, where the average Pt—S and Pt—Cl bond lengths are 2.255 (5) and 2.334 (4) Å, respectively, and the average weak axial Pt···S interaction distance is 3.137 (5) Å. The same ligand in the cationic complex forms two five-membered chelate rings in which the average length of the four equatorial Pt—S bonds is 2.296 (2) Å and the lengths of the two weak axial Pt···S bonds are 3.159 (2) Å.

Comment

The reaction between PtCl_2 and 1,4,7-trithiacyclodecane in CH_3CN is interesting. Complex (1) is first formed, in which platinum is bound to the S1 and S7 atoms, resulting in a six-membered chelate ring. Then, complex (1) isomerizes to complex (1'), in which platinum is bound to the S1 and S4 atoms, forming a five-membered chelate ring. On further reaction, cationic complex (2), in which platinum is coordinated by two 1,4,7-trithiacyclodecane (10S3) ligands, is formed:



Compound (I), $[\text{PtCl}_2(10\text{S3})]$, results from the co-crystallization of complexes (1) and (1') in the ratio 70:30. There are two independent molecules of $[\text{PtCl}_2(10\text{S3})]$ in the asymmetric unit. One molecule

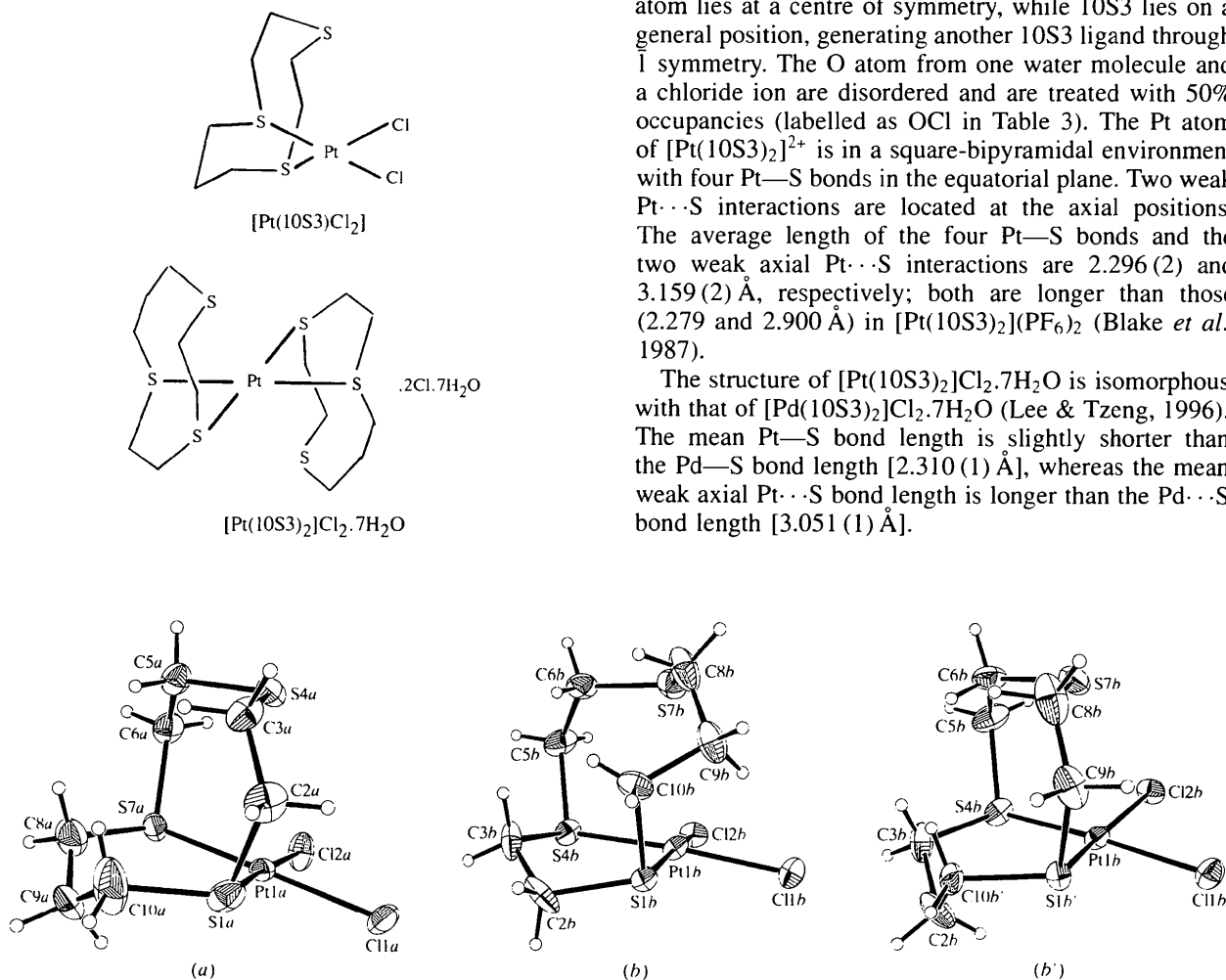


Fig. 1. The molecular structure of $[\text{PtCl}_2(10\text{S3})]$, showing (a) complex (1) of one molecule of (I), and (b) and (b'), the disorder components of the second molecule of (I). Displacement ellipsoids are shown at the 30% probability level.

with the atomic labelling is shown in Fig. 1(a). This has a six-membered chelate ring around the Pt atom and corresponds to complex (1). The other molecule exhibits a constitutional disorder and is shown in Figs. 1(b) and 1(b'). This disorder only affects two non-H atoms, namely S1 and C10. Fig. 1(b) represents the major structure (60%) and corresponds to complex (1'), while Fig. 1(b') shows the minor portion corresponding to complex (1).

The coordination sphere of the Pt atom in $[\text{PtCl}_2(10\text{S3})]$ can be described as distorted square pyramidal with two Cl and two S atoms forming the basal plane; the axial S atom is 3.137 (5) Å from the Pt atom, closer than the distance of 3.227 Å in $[\text{Pt}(9\text{S3})\text{Cl}_2]$, where 9S3 is 1,4,7-trithiacyclononane (Bennett *et al.*, 1993). The average Pt—S and Pt—Cl bond lengths [2.255 (5) and 2.334 (4) Å, respectively] are slightly longer than those in $[\text{PtCl}_2(9\text{S3})]$ (2.229 and 2.325 Å).

The molecular structure of $[\text{Pt}(10\text{S3})_2]^{2+}$ is shown in Fig. 2. In compound (II), $[\text{Pt}(10\text{S3})_2]\text{Cl}_2 \cdot 7\text{H}_2\text{O}$, the Pt atom lies at a centre of symmetry, while 10S3 lies on a general position, generating another 10S3 ligand through i symmetry. The O atom from one water molecule and a chloride ion are disordered and are treated with 50% occupancies (labelled as OCl in Table 3). The Pt atom of $[\text{Pt}(10\text{S3})_2]^{2+}$ is in a square-bipyramidal environment with four Pt—S bonds in the equatorial plane. Two weak Pt···S interactions are located at the axial positions. The average length of the four Pt—S bonds and the two weak axial Pt···S interactions are 2.296 (2) and 3.159 (2) Å, respectively; both are longer than those (2.279 and 2.900 Å) in $[\text{Pt}(10\text{S3})_2](\text{PF}_6)_2$ (Blake *et al.*, 1987).

The structure of $[\text{Pt}(10\text{S3})_2]\text{Cl}_2 \cdot 7\text{H}_2\text{O}$ is isomorphous with that of $[\text{Pd}(10\text{S3})_2]\text{Cl}_2 \cdot 7\text{H}_2\text{O}$ (Lee & Tzeng, 1996). The mean Pt—S bond length is slightly shorter than the Pd—S bond length [2.310 (1) Å], whereas the mean weak axial Pt···S bond length is longer than the Pd···S bond length [3.051 (1) Å].

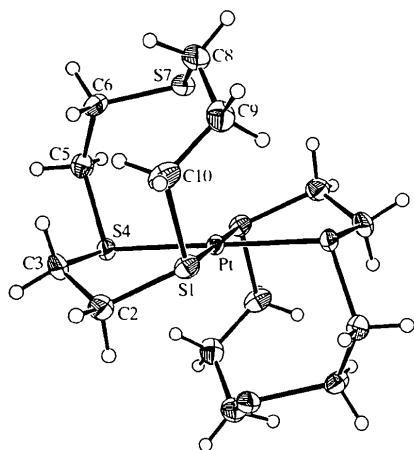


Fig. 2. The molecular structure of the [Pt(10S3)₂]²⁺ cation. Displacement ellipsoids are shown at the 30% probability level.

Experimental

[PtCl₂(10S3)] was prepared by refluxing PtCl₂ and 10S3 in a molar ratio of 1:1 in CH₃CN for 12 h. Yellow crystals were obtained after one week by evaporating the solution in air. On leaving the solution to stand for a further week, the yellow crystals of [PtCl₂(10S3)] redissolved and pale red-brown crystals of [Pt(10S3)₂]Cl₂·7H₂O were obtained.

Compound (I)

Crystal data

[PtCl₂(C₇H₁₄S₃)]

M_r = 460.36

Monoclinic

*P*2₁

a = 8.387 (1) Å

b = 12.511 (2) Å

c = 11.806 (3) Å

β = 96.11 (2)°

V = 1231.8 (4) Å³

Z = 4

D_x = 2.482 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 9.76–15.08°

μ = 12.40 mm⁻¹

T = 298 K

Plate

0.50 × 0.15 × 0.05 mm

Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer

θ/2θ scans

Absorption correction:

empirical via ψ scan

(North *et al.*, 1968)

T_{min} = 0.155, *T_{max}* = 0.538

3727 measured reflections

3727 independent reflections

2741 reflections with

I_{net} > 2σ(*I_{net}*)

θ_{max} = 30.0°

h = -11 → 11

k = 0 → 17

l = 0 → 16

3 standard reflections

frequency: 60 min

intensity decay: none

Refinement

Refinement on *F*

R = 0.041

wR = 0.041

S = 1.76

Extinction correction:

Larson (1970)

Extinction coefficient:

2.56 (16) × 10³

2741 reflections

253 parameters

H atoms not refined

w = 1/[σ²(*F*) + 0.0001*F*²]

(Δ/σ)_{max} = 0.001

Δρ_{max} = 1.32 e Å⁻³

Δρ_{min} = -1.51 e Å⁻³

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Absolute structure: Rogers (1981)

Rogers parameter = 1.000

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (I)

$$U_{eq} = (1/3)\sum_i \sum_j U^{ij} a^i a^j a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Pt1a	0.93827 (6)	0	0.40245 (5)	0.0414 (3)
Cl1a	0.9042 (6)	-0.1342 (4)	0.2653 (4)	0.067 (3)
Cl2a	1.1964 (5)	0.0321 (4)	0.3554 (5)	0.070 (3)
S1a	0.6868 (5)	-0.0395 (5)	0.4356 (4)	0.066 (3)
C2a	0.702 (2)	-0.1574 (18)	0.5316 (18)	0.073 (13)
C3a	0.815 (2)	-0.1559 (15)	0.6378 (14)	0.060 (10)
S4a	1.0232 (5)	-0.1299 (4)	0.6296 (4)	0.052 (2)
C5a	1.0554 (18)	-0.0031 (19)	0.7074 (14)	0.060 (10)
C6a	1.1215 (17)	0.080 (2)	0.6418 (15)	0.065 (11)
S7a	0.9921 (5)	0.1373 (4)	0.5240 (4)	0.051 (2)
C8a	0.821 (2)	0.183 (2)	0.5872 (19)	0.081 (14)
C9a	0.666 (2)	0.1680 (18)	0.5208 (18)	0.078 (14)
Cl10a	0.597 (2)	0.058 (3)	0.529 (2)	0.12 (2)
Pt1b	0.66251 (6)	0.07837 (7)	0.88932 (5)	0.0418 (3)
Cl1b	0.4735 (5)	-0.0439 (4)	0.8059 (4)	0.062 (3)
Cl2b	0.8425 (5)	-0.0609 (4)	0.9378 (4)	0.056 (2)
S1b	0.5149 (7)	0.2224 (6)	0.8237 (6)	0.041 (3)
S1b'	0.4448 (10)	0.1860 (8)	0.8588 (9)	0.043 (5)
C2b	0.662 (2)	0.3364 (17)	0.8333 (19)	0.076 (13)
C3b	0.7846 (19)	0.3274 (15)	0.9388 (19)	0.069 (13)
S4b	0.8572 (4)	0.1909 (4)	0.9582 (4)	0.053 (2)
C5b	0.8761 (18)	0.174 (2)	1.1140 (15)	0.073 (13)
C6b	0.725 (2)	0.1889 (18)	1.1723 (14)	0.065 (12)
S7b	0.5708 (5)	0.0913 (5)	1.1383 (4)	0.066 (3)
C8b	0.386 (2)	0.1648 (18)	1.112 (2)	0.091 (17)
C9b	0.3167 (18)	0.1721 (17)	0.9857 (19)	0.072 (13)
Cl10b	0.405 (3)	0.272 (3)	0.944 (3)	0.062 (18)
Cl10b'	0.505 (6)	0.327 (3)	0.886 (4)	0.06 (3)

Table 2. Selected geometric parameters (Å, °) for (I)

Pt1a—Cl1a	2.329 (5)	Pt1b—Cl2b	2.337 (4)
Pt1a—Cl2a	2.327 (4)	Pt1b—S1b	2.275 (6)
Pt1a—S1a	2.241 (4)	Pt1b—S1b'	2.266 (9)
Pt1a—S4a	3.152 (5)	Pt1b—S4b	2.242 (4)
Pt1a—S7a	2.253 (5)	Pt1b—S7b	3.121 (5)
Pt1b—Cl1b	2.343 (4)		
Cl1a—Pt1a—Cl2a	90.4 (2)	Cl1b—Pt1b—S1b'	79.8 (3)
Cl1a—Pt1a—S1a	85.1 (2)	Cl1b—Pt1b—S4b	175.1 (2)
Cl1a—Pt1a—S4a	102.7 (2)	Cl1b—Pt1b—S7b	101.9 (2)
Cl1a—Pt1a—S7a	173.7 (2)	Cl2b—Pt1b—S1b	171.2 (2)
Cl2a—Pt1a—S1a	175.4 (2)	Cl2b—Pt1b—S1b'	166.1 (3)
Cl2a—Pt1a—S4a	99.2 (2)	Cl2b—Pt1b—S4b	87.3 (2)
Cl2a—Pt1a—S7a	83.8 (2)	Cl2b—Pt1b—S7b	91.5 (1)
S1a—Pt1a—S4a	82.5 (2)	S1b—Pt1b—S4b	88.6 (2)
S1a—Pt1a—S7a	100.7 (2)	S1b—Pt1b—S7b	95.7 (2)
S4a—Pt1a—S7a	80.8 (1)	S1b'—Pt1b—S4b	103.2 (3)
Cl1b—Pt1b—Cl2b	90.4 (2)	S1b'—Pt1b—S7b	81.0 (3)
Cl1b—Pt1b—S1b	93.1 (2)	S4b—Pt1b—S7b	82.5 (1)

Compound (II)

Crystal data

[Pt(C₇H₁₄S₃)₂]Cl₂·7H₂O

M_r = 780.84

Mo *K*α radiation

λ = 0.71073 Å

Monoclinic
 $C2/c$
 $a = 17.998$ (4) Å
 $b = 11.627$ (2) Å
 $c = 13.690$ (3) Å
 $\beta = 96.75$ (2)°
 $V = 2844.9$ (10) Å³
 $Z = 4$
 $D_r = 1.823$ Mg m⁻³
 D_m not measured

Cell parameters from 25 reflections
 $\theta = 11.5$ – 14.5 °
 $\mu = 5.63$ mm⁻¹
 $T = 298$ K
 Prism
 $0.50 \times 0.30 \times 0.20$ mm
 Red–brown

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: empirical via ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.263$, $T_{\max} = 0.324$
 2506 measured reflections
 2506 independent reflections

1928 reflections with $I_{\text{net}} > 2\sigma(I_{\text{net}})$
 $\theta_{\max} = 25.0$ °
 $h = -21 \rightarrow 21$
 $k = 0 \rightarrow 13$
 $l = 0 \rightarrow 16$
 3 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F^2
 $R = 0.029$
 $wR = 0.044$
 $S = 2.49$
 1928 reflections
 140 parameters
 H atoms not refined
 $w = 1/[\sigma^2(F) + 0.0001F^2]$
 $(\Delta/\sigma)_{\max} = 0.0002$

$\Delta\rho_{\max} = 0.79$ e Å⁻³
 $\Delta\rho_{\min} = -0.65$ e Å⁻³
 Extinction correction: Larson (1970)
 Extinction coefficient: 2.7 (4) $\times 10^4$
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (II)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a^i a^j \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Pt	1/4	3/4	0	0.0247 (2)
S1	0.12444 (11)	0.76196 (18)	-0.05278 (15)	0.0350 (10)
C2	0.0983 (5)	0.8837 (7)	0.0178 (6)	0.043 (5)
C3	0.1291 (5)	0.8791 (7)	0.1239 (7)	0.045 (5)
S4	0.22961 (12)	0.85135 (17)	0.13845 (14)	0.0329 (10)
C5	0.2425 (5)	0.7543 (7)	0.2417 (6)	0.040 (5)
C6	0.2019 (5)	0.6393 (8)	0.2325 (6)	0.044 (5)
S7	0.23531 (13)	0.54148 (18)	0.14441 (17)	0.0413 (12)
C8	0.1499 (5)	0.4746 (7)	0.0858 (7)	0.049 (5)
C9	0.1132 (5)	0.5280 (8)	-0.0087 (7)	0.048 (5)
C10	0.0770 (5)	0.6437 (8)	0.0019 (6)	0.045 (5)
Cl	0	0.3595 (4)	3/4	0.080 (3)
OC1	0.1947 (2)	0.1620 (4)	0.0196 (3)	0.072 (3)
O2	1	1.0263 (8)	1/4	0.068 (7)
O3	0	0.3830 (10)	1/4	0.085 (8)
O4	0.0695 (4)	0.1967 (6)	0.1487 (5)	0.069 (5)
O5	0.0686 (5)	0.1482 (7)	0.8624 (6)	0.091 (6)

Table 4. Selected geometric parameters (Å, °) for (II)

Pt—S1	2.294 (2)	Pt—S7	3.159 (2)
Pt—S4	2.298 (2)		
S1—Pt—S4	88.96 (8)	S1—Pt—S7	84.73 (7)
S1—Pt—S4'	91.04 (8)	S4—Pt—S7	81.03 (6)
S1—Pt—S7	95.27 (7)	S4—Pt—S7'	98.97 (6)

Symmetry code: (i) $\frac{1}{2} - x, \frac{3}{2} - y, -z$.

The title structures were solved by the heavy-atom method, subsequent Fourier syntheses based on the heavy atoms revealing the positions of all the non-H atoms. Least-squares

refinement, including anisotropic parameters for the non-H atoms, was performed. H atoms were placed at calculated positions with $U_{\text{iso}}(\text{H}) = U_{\text{eq}}(\text{attached atom}) + 0.01$ Å². The absolute values of $\Delta\rho_{\min}$ and $\Delta\rho_{\max}$ for compound (I) were further than 1 e Å⁻³ from the Pt atom.

For both compounds, data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *NRCVAX DATRD2* (Gabe *et al.*, 1989); program(s) used to solve structures: *NRCVAX SOLVER*; program(s) used to refine structures: *NRCVAX LSTSQ*; molecular graphics: *NRCVAX ORTEP*; software used to prepare material for publication: *NRCVAX TABLES*; computer: DEC-3500.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1253). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). **C54**, 909–911

A Tetranuclear Complex of Palladium(II) with Cysteine

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Abstract

The title compound, tetrakis(μ -cysteinato-*N,S,S*)tetrakis-[chloropalladium(II)]-dimethyl sulfoxide-diethyl ether (1/3/1), [Pd₄Cl₄(μ -C₃H₆NO₂S)₄].3(CH₃)₂SO.(CH₃-CH₂)₂O, contains four nearly square-planar units in each of which the palladium(II) ion is bound to two