ligands. Only one of the four *tert*-butyl groups is disordered; it is not apparent why this is, but is well established by the electron-density maps. The toluene molecule is held loosely in the lattice and is poorly determined. Crystals left out in the atmosphere lose this solvent quickly and the samples used for study may have lost some before being encapsulated.

In Fig. 2 are shown the intermolecular connections. Hydrogen bonds from the water molecule [O(12)] to naphthalenesulfonate atoms, O(6) and O(8), of two different molecules link the structure into large centrosymmetric dimers. Toluene molecules occupy spaces between them.

In summary, the solution IR studies (Lumetta & Moyer, 1991) are supported by these crystal-structure analyses and the bonding geometry is clarified. The Mn^{2+} and Zn^{2+} ions fit well in the 15-crown-5 ring of CH15C5 where they are bonded to all its O atoms and to an O atom of a sulfonate group and to one water molecule to form a pentagonal bipyramid.

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Structures of Benzenethiolato Complexes of Palladium(II) and Platinum(II)

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Abstract. Bis(tetraphenylphosphonium) di-μ-benzenethiolato-tetrakis(benzenethiolato-$1\kappa^2S,2\kappa^2S$)dipalladate acetonitrile solvate, $[Ph_4P]_2[Pd_2(SPh)_6].2MeCN$ (1), $M_r = 1628{\cdot}8$, monoclinic, $P2_1/n$, $a = 13{\cdot}724$ (5), $b = 26{\cdot}306$ (6), $c = 10{\cdot}931$ (4) Å, $\beta = 96{\cdot}71$ (2)°, $V = 3919{\cdot}3$ (22) Å^3, $Z = 2$, $D_x = 1{\cdot}38$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 5{\cdot}9$ cm^{-1}, $F(000) = 1672$, $T = 293$ K, $R = 0{\cdot}066$ for 2283 observed reflections. $[Pd_2(SPh)_6]^{2-}$ is centrosymmetric and is formed by edge sharing of two square-planar PdS_4 coordinations. Bis(tetraethylammonium) tetra(benzenethiolato)platinate(II), $[Et_4N]_2[Pt(SPh)_4]$ (2), $M_r = 892{\cdot}4$, monoclinic, $P2_1/n$, $a = 16{\cdot}419$ (5), $b = 12{\cdot}117$ (2), $c = 10{\cdot}186$ (2) Å, $\beta = 91{\cdot}61$ (2)°, $V = 2025{\cdot}7$ (8) Å^3, $Z = 2$, $D_x = 1{\cdot}46$ g cm^{-3}, $\mu = 38{\cdot}6$ cm^{-1}, λ(Mo $K\alpha$) = 0·71069 Å, $F(000) = 912$, $T = 293$ K, $R = 0{\cdot}032$ for 2376 observed reflections. $[Pt(SPh)_4]^{2-}$ has a center of symmetry and PtS_4 has square-planar coordination.

Introduction. Some nickel thiolate (Tremel, Kriege, Krebs & Henkel, 1988) and palladium thiolate (Higgins & Suggs, 1988) clusters have been synthesized and structurally characterized by the X-ray diffraction method. In these complexes the coordination geometry around each metal ion is square planar. Reaction of Ni^{2+} with benzenethiolate (SPh^-), however, gives a mononuclear tetrahedral

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Table 1. *Details of data collections and refinements for* (1) *and* (2)

	(1)	(2)
Crystal dimensions (mm)	0·28 × 0·33 × 0·50	0·13 × 0·22 × 0·22
Scan mode	ω	ω
$2\theta_{max}$ (°)	46	50
Scan rate (° s^{-1})	0·06	0·05
Scan range (°)	(2·0 + 0·3tanθ)	(0·8 + 0·3tanθ)
Background time (s)	10	10
h	− 14–14	− 19–19
k	0–28	0–14
l	0–11	0–12
Number of reflections	2283 $[F_o^2 > 3\sigma(F_o^2)]$	2376 $[F_o^2 > 3\sigma(F_o^2)]$
Function minimized	$w\sum(F_o - k\|F_c\|)^2$	$w\sum(F_o - k\|F_c\|)^2$
w	$[\sigma^2(F_o) + 0·0009F_o^2]^{-1}$	$[\sigma^2(F_o) + 0·008F_o^2]^{-1}$
Max. shift/e.s.d. for last cycle	0·7	0·4
Residual electron density (e Å^{-3})	0·5	0·9

complex $[Ni(SPh)_4]^{2-}$ (Swenson, Baenziger & Coucouvanis, 1978; Yamamura, Miyamae, Katayama & Sasaki, 1985). In connection with these investigations, two reaction systems $Pd(NO_3)_2$/NaSPh (molar ratio 1:4·4) and K_2PtCl_4/NaSPh (molar ratio 1:4·5) in acetonitrile (MeCN) were studied. The first reaction system gives the dinuclear complex $(Ph_4P)_2[Pd_2(SPh)_6].2MeCN$ (1) and the second affords the mononuclear complex $(Et_4N)_2[Pt(SPh)_4]$ (2). Isolation of an open-chain trinuclear complex $[Pd_3(SPh)_8]^{2-}$ and a mononuclear complex $[Pt(SPh)_4]^{2-}$ has been cited by Kriege & Henkel in Tremel *et al.* (1988).

Experimental. To a stirred suspension of $Pd(NO_3)_2$ (115 mg) in acetonitrile (15 cm^3), NaSPh (293 mg) was added. After 20 h, Et_4NBr (200 mg) was added and the solution was stirred for 1 h. The solution was then filtered and the filtrate was treated with solid Ph_4PBr (110 mg). Red crystals of (1) were obtained. The presence of Et_4NBr in the solution gives good crystals. Yield 75 mg (19%). Found: C, 63·90; H, 4·63; N, 1·79%. Calc. for $C_{88}H_{76}N_2P_2Pd_2S_6$: C, 64·89; H, 4·71; N, 1·72%.

A suspension of K_2PtCl_4 (207 mg) and NaSPh (300 mg) in acetonitrile (30 cm^3) and acetone (5 cm^3) was stirred for 20 h. Et_4NBr (200 mg) was then added and the solution was stirred for 1 h. After filtration the filtrate was stored at 276 K for 12 h. Yellow crystals of (2) were collected. Yield 50 mg (12%). Found: C, 53·85; H, 6·77; N, 3·23%. Calc. for $C_{40}H_{60}N_2PtS_4$: C, 53·85; H, 6·77; N, 3·13%.

Table 2. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2) *for* (1) *with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
Pd	0·02643 (12)	0·06274 (6)	0·04652 (14)	3·987 (3)
S(1)	−0·0085 (4)	0·1082 (2)	0·2199 (5)	4·7 (1)
S(2)	0·1160 (5)	0·1296 (2)	−0·0258 (6)	5·8 (1)
S(3)	0·0619 (4)	0·0069 (2)	−0·1096 (5)	4·4 (1)
P	0·0477 (4)	0·3394 (2)	0·8606 (5)	4·2 (1)
C(1)	−0·0776 (14)	0·1636 (7)	0·1738 (19)	4·9 (6)
C(2)	−0·1055 (17)	0·1769 (8)	0·0558 (18)	6·1 (7)
C(3)	−0·1644 (20)	0·2223 (11)	0·0300 (26)	10·0 (10)
C(4)	−0·1934 (17)	0·2476 (10)	0·1305 (30)	10·3 (11)
C(5)	−0·1669 (19)	0·2343 (9)	0·2448 (28)	9·6 (10)
C(6)	−0·1096 (15)	0·1924 (8)	0·2701 (21)	6·3 (7)
C(7)	0·2333 (14)	0·1086 (7)	−0·0318 (16)	4·4 (6)
C(8)	0·2768 (15)	0·0706 (8)	0·0450 (18)	5·3 (6)
C(9)	0·3751 (18)	0·0547 (8)	0·0425 (21)	7·1 (8)
C(10)	0·4321 (17)	0·0804 (10)	−0·0421 (23)	8·6 (9)
C(11)	0·3884 (18)	0·1162 (9)	−0·1175 (20)	7·4 (8)
C(12)	0·2922 (16)	0·1310 (9)	−0·1129 (20)	6·5 (7)
C(13)	−0·0029 (15)	0·0284 (7)	−0·2503 (17)	4·6 (6)
C(14)	0·0449 (17)	0·0599 (11)	−0·3268 (19)	7·7 (8)
C(15)	−0·0127 (16)	0·0755 (8)	−0·4447 (18)	6·3 (7)
C(16)	−0·0985 (19)	0·0589 (9)	−0·4804 (20)	7·4 (8)
C(17)	−0·1452 (17)	0·0280 (12)	−0·4094 (21)	9·8 (10)
C(18)	−0·1023 (15)	0·0115 (9)	−0·2938 (22)	6·8 (8)
C(19)	−0·0759 (14)	0·3509 (7)	0·8876 (18)	4·5 (6)
C(20)	−0·1512 (13)	0·3462 (8)	0·7939 (20)	5·3 (6)
C(21)	−0·2452 (17)	0·3597 (9)	0·8154 (20)	6·7 (7)
C(22)	−0·2594 (16)	0·3795 (9)	0·9271 (25)	7·7 (8)
C(23)	−0·1835 (16)	0·3841 (10)	1·0256 (21)	7·8 (8)
C(24)	−0·0902 (15)	0·3705 (8)	1·0036 (21)	6·4 (7)
C(25)	0·1205 (14)	0·3161 (7)	0·9960 (16)	4·4 (5)
C(26)	0·0803 (17)	0·2794 (8)	1·0687 (20)	6·4 (7)
C(27)	0·1347 (20)	0·2578 (11)	1·1701 (23)	9·5 (10)
C(28)	0·2280 (24)	0·2692 (11)	1·1890 (25)	11·2 (12)
C(29)	0·2684 (16)	0·3043 (11)	1·1280 (20)	8·4 (9)
C(30)	0·2167 (15)	0·3288 (9)	1·0251 (18)	5·8 (7)
C(31)	0·0448 (13)	0·2922 (7)	0·7416 (17)	4·2 (5)
C(32)	0·0634 (13)	0·2422 (7)	0·7727 (18)	4·6 (6)
C(33)	0·0619 (16)	0·2076 (8)	0·6816 (20)	6·2 (7)
C(34)	0·0450 (15)	0·2199 (8)	0·5598 (21)	6·6 (7)
C(35)	0·0292 (17)	0·2695 (9)	0·5281 (20)	7·0 (7)
C(36)	0·0280 (16)	0·3055 (8)	0·6182 (19)	5·7 (6)
C(37)	0·1001 (13)	0·3955 (7)	0·8083 (17)	3·9 (5)
C(38)	0·1827 (14)	0·3914 (7)	0·7476 (19)	4·9 (6)
C(39)	0·2269 (14)	0·4339 (9)	0·7124 (19)	6·0 (7)
C(40)	0·1906 (14)	0·4836 (9)	0·7370 (19)	5·7 (7)
C(41)	0·1050 (16)	0·4880 (8)	0·7981 (21)	6·0 (7)
C(42)	0·0609 (14)	0·4422 (8)	0·8327 (18)	5·1 (6)
N	0·1080 (19)	0·4310 (10)	0·2038 (23)	12·1 (7)*
C(43)	0·2421 (28)	0·4066 (15)	0·3813 (33)	15·4 (13)*
C(44)	0·1665 (17)	0·4198 (14)	0·2924 (32)	14·0 (11)*

* $B = 8\pi^2u^2$.

The crystal of (1) is sensitive to air oxidation and was sealed in a capillary tube, while the crystal of (2) is insensitive to air. The details of data collections and structure refinements for (1) and (2) are given in Table 1. Intensity data were collected on a Philips PW1100 diffractometer with monochromated Mo $K\alpha$ radiation. The lattice parameters were obtained from least-squares fit of 14 centered reflections ($9° < 2\theta < 22°$) for (1) and 17($17° < 2\theta < 23°$) for (2). Intensities of three standard reflections, monitored every 4 h, showed broadening of reflection profile for (1), but no significant variation was observed for both crystals. An absorption correction was applied (Templeton & Templeton, 1973) for the data of (2). Max. and min. correction coefficients are 1·95 and 1·57.

The positions of Pt, Pd, and S atoms were obtained from Patterson syntheses. Subsequent least-squares refinement followed by difference Fourier syntheses revealed the positions of all non-H atoms. The structures were refined by the least-squares (on

Table 3. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2) *for* (2) *with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Pt	0·0	0·0	0·0	2·193 (1)
S(1)	0·12263 (12)	0·02807 (15)	0·11824 (21)	3·112 (4)
S(2)	0·07696 (12)	−0·06963 (16)	−0·16969 (19)	3·293 (4)
C(1)	0·1465 (4)	−0·0997 (6)	0·1927 (6)	3·0 (1)
C(2)	0·2210 (5)	−0·1132 (7)	0·2570 (8)	4·2 (2)
C(3)	0·2424 (5)	−0·2130 (8)	0·3147 (8)	4·6 (2)
C(4)	0·1898 (6)	−0·2975 (8)	0·3169 (9)	5·8 (2)
C(5)	0·1143 (6)	−0·2851 (8)	0·2537 (11)	5·7 (2)
C(6)	0·0933 (5)	−0·1876 (7)	0·1927 (9)	4·6 (2)
C(7)	0·0428 (5)	−0·1996 (6)	−0·2241 (7)	3·1 (1)
C(8)	0·0751 (5)	−0·2423 (7)	−0·3426 (8)	4·4 (2)
C(9)	0·0549 (6)	−0·3445 (7)	−0·3846 (9)	5·0 (2)
C(10)	0·0036 (6)	−0·4130 (7)	−0·3163 (10)	5·6 (2)
C(11)	−0·0300 (6)	−0·3714 (7)	−0·2007 (10)	5·0 (2)
C(12)	−0·0096 (5)	−0·2672 (6)	−0·1568 (8)	3·6 (1)
N	0·1877 (4)	0·1974 (5)	−0·3975 (6)	3·1 (1)
C(13)	0·1260 (6)	0·2158 (8)	−0·2914 (9)	4·8 (2)
C(14)	0·1567 (8)	0·2527 (10)	−0·1598 (9)	7·4 (3)
C(15)	0·2377 (5)	0·3008 (7)	−0·4224 (9)	4·4 (2)
C(16)	0·1885 (6)	0·4064 (8)	−0·4408 (10)	5·5 (2)
C(17)	0·1449 (5)	0·1676 (7)	−0·5255 (7)	4·2 (2)
C(18)	0·0933 (6)	0·0634 (8)	−0·5202 (9)	5·2 (2)
C(19)	0·2437 (5)	0·1074 (8)	−0·3526 (9)	5·0 (2)
C(20)	0·3068 (6)	0·0739 (10)	−0·4463 (11)	6·5 (3)

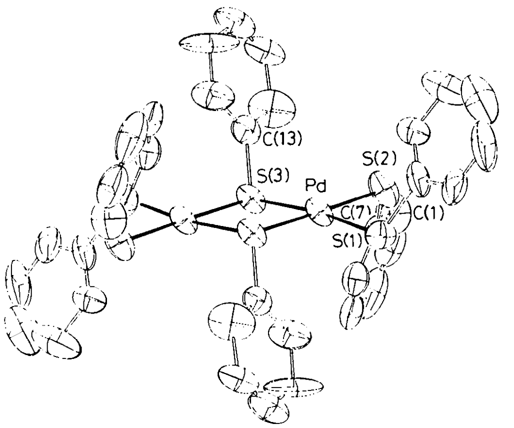

Fig. 1. A view of the complex anion of (1).

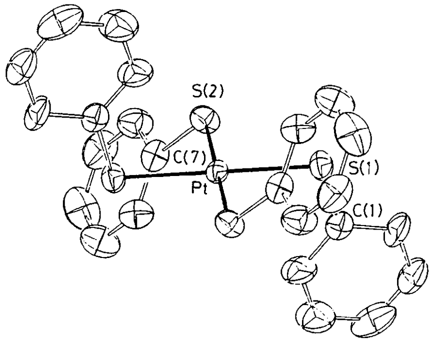

Fig. 2. A view of the complex anion of (2).

Table 4. *Selected interatomic distances* (Å) *and angles* (°) *for* (1) *with e.s.d.'s in parentheses*

Pd—S(1)	2·337 (6)	S(3)···S(3^{i})	3·12 (1)
Pd—S(2)	2·336 (7)	S(1)—C(1)	1·78 (3)
Pd—S(3)	2·346 (6)	S(2)—C(7)	1·71 (2)
Pd—S(3^{i})	2·344 (6)	S(3)—C(13)	1·78 (2)
Pd···Pd^{i}	3·505 (4)		
S(1)—Pd—S(2)	93·2 (2)	S(3)—Pd—S(3^{i})	83·3 (2)
S(1)—Pd—S(3)	171·8 (2)	Pd—S(3)—Pd^{i}	96·7 (2)
S(2)—Pd—S(3)	93·7 (2)	Pd—S(1)—C(1)	110·0 (7)
S(1)—Pd—S(3^{i})	89·9 (2)	Pd—S(2)—C(7)	107·7 (7)
S(2)—Pd—S(3^{i})	176·9 (2)	Pd—S(3)—C(13)	107·7 (7)

Symmetry code: (i) $-x$, $-y$, $-z$.

Table 5. *Selected interatomic distances* (Å) *and angles* (°) *for* (2) *with e.s.d.'s in parentheses*

Pt—S(1)	2·342 (2)	S(1)—C(1)	1·762 (8)
Pt—S(2)	2·328 (2)	S(2)—C(7)	1·756 (8)
S(1)—Pt—S(2)	87·58 (7)	Pt—S(1)—C(1)	105·7 (3)
S(1)—Pt—S(2^{i})	92·42 (7)	Pt—S(2)—C(7)	112·6 (3)

Symmetry code: (i) $-x$, $-y$, $-z$.

F) method using anisotropic thermal parameters. The acetonitrile molecule was refined isotropically. The H atoms, except for those of acetonitrile, were included in the refinement but their parameters were fixed at idealized positions [C—H = 1·0 Å, B = 8·0 Å^2 for (1) and 5·0 Å^2 for (2)]. The atomic scattering factors with corrections of anomalous scattering for the Pt, Pd, S and P atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148). The R, wR and S are 0·066, 0·097 and 1·98 for (1) and 0·032, 0·053 and 0·57 for (2). Atomic coordinates for non-H atoms are listed in Tables 2 and 3.* Programs used in the calculations were a local version of *UNICS* (*HBLS*-IV, *RSSFR*-5 and *DAPH*) (Sakurai, 1969). Figures were drawn using *ORTEP* (Johnson, 1976). Calculations were performed on a HITAC M-660 K computer at Osaka City University.

Discussion. *ORTEP* views of complex anions of (1) and (2) are given in Figs. 1 and 2. Selected bond lengths and angles are listed in Tables 4 and 5. The structure of the complex anion of (1) has an inversion center and is formed by edge sharing of two PdS$_4$ units. The PdS$_4$ coordination is nearly planar: deviations of the atoms from the plane are S(1) 0·049, S(2) −0·046, S(3) 0·048 and Pd −0·043 Å. Two phenyl groups bound to S(3) and S(3) ($-x$, $-y$, $-z$) above and below the central

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* Lists of structure factors, anisotropic thermal parameters, full bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54169 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Pd_2S_2 plane have the *anti* conformation. The Pd···Pd length of 3·505 (4) Å seems too long to be assigned as any direct metal–metal interaction. The Pd—S_t (S_t = terminally bound S) bond lengths of 2·337 (6) and 2·336 (7) Å are slightly longer than the Pd—S bond lengths of 2·323 (3) and 2·312 (3) Å for $[Pd(edt)_2]^{2-}$ (edt = ethane-1,2-dithiolate; Snyder, Rao & Holm, 1986). The Pd—S_b (S_b = bridging S) bond lengths of 2·346 (6) and 2·344 (6) Å are also longer than those found in $[Pd_8(S\text{-}n\text{Pr})_{16}]$ (S-*n*Pr = *n*-propanethiolate; Higgins & Suggs, 1988). The bond length order Pd—S_b > Pd—S_t and angle order S_t—Pd—S_t > S_b—Pd—S_b hold in this case, too (Watson, Rao, Dorfman & Holm, 1985).

In the complex anion of (2), the Pt atom is at a center of symmetry. The Pt—S bond lengths of 2·342 (2) and 2·328 (2) Å compare with Pt—S_t bond lengths of 2·317 (5) and 2·342 (5) Å for *cis*-$[(PMePh_2)Pt(SCH_2Ph)(\mu\text{-}SCH_2Ph)]_2$ (Bird, Siriwardane, Lai & Shaver, 1982). The S(1)—Pt—S(2) angle, where the pendant phenyls from S(1) and S(2) are on the same side of the coordination plane, is smaller than 90° [(87·58 (7)°], while the S(1)—Pt—S(2) ($-x$, $-y$, $-z$) angle, where two phenyl groups from S(1) and S(2) ($-x$, $-y$, $-z$) are on the opposite side of the coordination plane, is larger than 90° [92·42 (7)°].

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Structure of Barium α-D-Ribofuranose-5-phosphate Pentahemihydrate: a Reinvestigation

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Abstract. $Ba^{2+}.C_5H_9O_8P^{2-}.5\cdot5H_2O$, M_r = 464·52, monoclinic, $C2$, a = 15·469 (9), b = 8·622 (5), c = 12·865 (8) Å, β = 118·33 (5)°, V = 1510 (2) Å^3, Z = 4, D_m = 2·047, D_x = 2·043 (2) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 28·8 cm^{-1}, $F(000)$ = 916, T = 300 (2) K, R = 0·030 for 6055 hkl and $\bar{h}\bar{k}\bar{l}$ non-zero reflections. The P—$O_{\rm ester}$ bond is 1·616 (2) Å. Distortions from tetrahedral geometry of the phosphate group are similar to those observed in other doubly ionized phosphate esters. The conformation of the α-D-ribofuranose ring is ^2E. The torsion angles P—O(5)—C(5)—C(4), O(5)—C(5)—C(4)—O(4) and O(5)—C(5)—C(4)—C(3) are 170·9 (4), −67·9 (4) and 52·6 (4)°, respectively. Each Ba^{2+} cation is surrounded by ten O atoms (one of them is disordered) at distances of 2·730 (4)–3·338 (3) Å.

Introduction. D-Ribose-5-phosphate is an important constituent of nucleic acids. The structure of the D-ribose fragment in many crystals of nucleotides and other compounds is well documented. However, the free D-ribose-5-phosphate anion is described only in an early work (Furberg & Mostad, 1962) in which the structure of its barium salt is presented. The

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