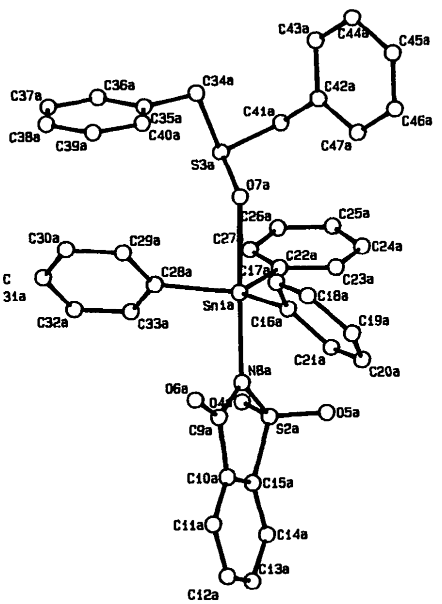

Fig. 1. One of the two crystallographically independent molecules of triphenyltin saccharin–dibenzyl sulfoxide.

two molecules (molecule *A*) is shown in Fig. 1. Selected bond distances and angles are listed in Table 2. The absolute structure was not determined.

**Related literature.** The Sn—N bond distances are somewhat longer and the Sn—O distances somewhat shorter than those [Sn—N 2.240 (7) – 2.242 (8); Sn—O 2.394 (8) – 2.409 (7) Å (Ng, Chen, Kumar Das & Mak, 1989*a*,*b*)] found in the ethanol, glycolic acid and dimethylformamide adducts. The Sn—O bonds in the dibenzyl sulfoxide adduct are longer than the Sn—O bond [2.319 (10) Å] found in the *cis* trigonal bipyramidal 1/1 adduct with dimethyltin dichloride (Ng & Rheingold, 1989).

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# Structure of *trans*-Dicyanobis(triphenylphosphine)platinum(II) Dimethanol Solvate

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**Abstract.** $[Pt(CN)_2(C_{18}H_{15}P)_2].2CH_4O$, $M_r = 835.790$, triclinic, $P\bar{1}$, $a = 7.736\,(1)$, $b = 10.406\,(2)$, $c = 12.111\,(3)$ Å, $\alpha = 111.00\,(1)$, $\beta = 104.07\,(1)$, $\gamma = 90.65\,(1)°$, $V = 877.7\,(3)$ Å$^3$, $Z = 1$, $D_x = 1.581$ g cm$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 41.63$ cm$^{-1}$, $F(000) = 416$, $T = 233$ K, final conventional $R = 0.0174$ and $wR = 0.0177$, for 214 variable parameters and 2303 reflections with $|F_o|^2 > 3\sigma|F_o|^2$. The structure consists of a discrete monomeric four-coordinated square-planar Pt$^{\text{II}}$ center with *trans* cyanide groups. The Pt—C distance is 1.991 (4) Å and the Pt—P distance is 2.332 (1) Å.

**Experimental.** $[Pt(CN)_2(PPh_3)_2]$ was synthesized according to the procedure of Bailar & Itatani (1967). IR $\nu$(CN) 2130 cm$^{-1}$. Colorless crystals were obtained from a warm solution of $C_6H_6$ and $CH_3OH$ which was allowed to cool slowly to room temperature. The compound crystallizes with solvent ($CH_3OH$) in the lattice. A multifaceted crystal of approximate dimensions 0.4 × 0.20 × 0.10 mm was mounted in a random orientation on a glass fiber. Data were collected at 233 K by using the $\omega$-scan technique in bisecting geometry on a Nicolet *R*3*m*/*E* diffractometer, with graphite-monochromated Mo $K\alpha$ radiation. The low-temperature device used has not been previously reported and therefore we

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^3$)

$U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Pt | 0 | 0 | 0 | 19 (1) |
| P(1) | −841 (1) | 1299 (1) | 1764 (1) | 20 (1) |
| N(1) | −631 (5) | 2338 (3) | −1080 (3) | 49 (1) |
| C(1) | −412 (4) | 1489 (4) | −674 (3) | 33 (1) |
| C(2) | −2085 (4) | 181 (3) | 2236 (3) | 22 (1) |
| C(3) | −3508 (4) | −734 (3) | 1341 (3) | 31 (1) |
| C(4) | −4562 (4) | −1566 (3) | 1653 (3) | 36 (1) |
| C(5) | −4185 (4) | −1503 (3) | 2846 (3) | 36 (1) |
| C(6) | −2740 (4) | −622 (4) | 3730 (3) | 35 (1) |
| C(7) | −1696 (4) | 230 (3) | 3429 (3) | 28 (1) |
| C(8) | −2277 (4) | 2661 (3) | 1686 (3) | 23 (1) |
| C(9) | −1644 (4) | 3792 (3) | 1472 (3) | 30 (1) |
| C(10) | −2742 (5) | 4810 (3) | 1383 (3) | 36 (1) |
| C(11) | −4459 (5) | 4721 (4) | 1507 (3) | 38 (1) |
| C(12) | −5080 (4) | 3624 (4) | 1749 (3) | 36 (1) |
| C(13) | −4004 (4) | 2599 (3) | 1840 (3) | 30 (1) |
| C(14) | 1096 (4) | 2151 (3) | 3056 (3) | 23 (1) |
| C(15) | 2642 (4) | 1482 (3) | 3172 (3) | 32 (1) |
| C(16) | 4103 (4) | 2089 (4) | 4176 (3) | 36 (1) |
| C(17) | 4062 (4) | 3360 (4) | 5064 (3) | 32 (1) |
| C(18) | 2527 (5) | 4021 (4) | 4954 (3) | 43 (1) |
| C(19) | 1065 (4) | 3417 (4) | 3950 (3) | 39 (1) |
| C(10) | −1275 (5) | 3000 (4) | −3597 (4) | 51 (2) |
| O | −1969 (3) | 3779 (3) | −2615 (3) | 47 (1) |

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

| | | | |
|---|---|---|---|
| Pt—P(1) | 2.332 (1) | Pt—C(1) | 1.991 (4) |
| Pt—P(1*a*) | 2.332 (1) | Pt—C(1*a*) | 1.991 (4) |
| P(1)—C(2) | 1.824 (4) | P(1)—C(8) | 1.826 (3) |
| P(1)—C(14) | 1.821 (3) | N(1)—C(1) | 1.151 (6) |
| C(2)—C(3) | 1.396 (4) | C(2)—C(7) | 1.384 (5) |
| C(3)—C(4) | 1.387 (6) | C(4)—C(5) | 1.380 (6) |
| C(5)—C(6) | 1.388 (4) | C(6)—C(7) | 1.390 (6) |
| C(8)—C(9) | 1.400 (5) | C(8)—C(13) | 1.397 (5) |
| C(9)—C(10) | 1.383 (5) | C(10)—C(11) | 1.379 (6) |
| C(11)—C(12) | 1.383 (6) | C(12)—C(13) | 1.380 (5) |
| C(14)—C(15) | 1.394 (4) | C(14)—C(19) | 1.378 (4) |
| C(15)—C(16) | 1.386 (4) | C(16)—C(17) | 1.379 (4) |
| C(17)—C(18) | 1.382 (5) | C(18)—C(19) | 1.385 (4) |
| C(30)—O | 1.409 (5) | | |
| | | | |
| P(1)—Pt—C(1) | 92.2 (1) | C(1)—Pt—P(1*a*) | 87.8 (1) |
| P(1)—Pt—C(1*a*) | 87.8 (1) | P(1*a*)—Pt—C(1*a*] | 92.2 (1) |
| Pt—P(1)—C(2) | 110.5 (1) | Pt—P(1)—C(8) | 117.8 (1) |
| C(2)—P(1)—C(8) | 104.4 (1) | Pt—P(1)—C(14) | 111.9 (1) |
| C(2)—P(1)—C(14) | 106.1 (2) | C(8)—P(1)—C(14) | 105.3 (1) |
| Pt—C(1)—N(1) | 178.7 (3) | P(1)—C(2)—C(3) | 117.3 (3) |
| P(1)—C(2)—C(7) | 122.8 (2) | C(3)—C(2)—C(7) | 119.9 (3) |
| C(2)—C(3)—C(4) | 120.1 (3) | C(3)—C(4)—C(5) | 119.9 (3) |
| C(4)—C(5)—C(6) | 120.1 (4) | C(5)—C(6)—C(7) | 120.3 (4) |
| C(2)—C(7)—C(6) | 119.6 (3) | P(1)—C(8)—C(9) | 119.7 (2) |
| P(1)—C(8)—C(13) | 121.2 (3) | C(9)—C(8)—C(13) | 119.1 (3) |
| C(8)—C(9)—C(10) | 120.0 (3) | C(9)—C(10)—C(11) | 120.4 (4) |
| C(10)—C(11)—C(12) | 120.0 (3) | C(11)—C(12)—C(13) | 120.5 (3) |
| C(8)—C(13)—C(12) | 120.1 (4) | P(1)—C(14)—C(15) | 119.1 (2) |
| P(1)—C(14)—C(19) | 122.2 (2) | C(15)—C(14)—C(19) | 118.7 (2) |
| C(14)—C(15)—C(16) | 119.9 (3) | C(15)—C(16)—C(17) | 121.0 (3) |
| C(16)—C(17)—C(18) | 119.2 (3) | C(17)—C(18)—C(19) | 119.9 (3) |
| C(14)—C(19)—C(18) | 121.3 (3) | | |
| | | | |
| C(1)—Pt—P(1)—C(2) | 142.8 (1) | C(1)—Pt—P(1)—C(8) | 22.9 (1) |
| C(1*a*)—Pt—P(1)—C(2) | −37.2 (1) | C(1*a*)—Pt—P(1)—C(8) | −157.1 (1) |
| C(1)—Pt—P(1)—C(14) | −99.3 (2) | C(1*a*)—Pt—P(1)—C(14) | 80.7 (2) |
| P(1)—Pt—C(1)—N(1) | 164.5 (2) | P(1*a*)—Pt—C(1)—N(1) | −15.5 (2) |
| Pt—P(1)—C(2)—C(3) | −50.0 (3) | Pt—P(1)—C(2)—C(7) | 131.3 (2) |
| C(8)—P(1)—C(2)—C(7) | 9.8 (3) | C(8)—P(1)—C(2)—C(7) | −101.1 (3) |
| C(14)—P(1)—C(2)—C(3) | −171.5 (2) | C(14)—P(1)—C(2)—C(7) | 9.8 (3) |
| Pt—P(1)—C(8)—C(13) | −6.6 (3) | Pt—P(1)—C(14)—C(19) | 147.6 (3) |
| C(2)—P(1)—C(14)—C(15) | 85.8 (3) | C(2)—P(1)—C(14)—C(19) | −91.8 (4) |
| C(8)—P(1)—C(14)—C(15) | −163.9 (3) | C(8)—P(1)—C(14)—C(19) | 15.8 (3) |
| P(1)—C(2)—C(3)—C(4) | −176.9 (3) | P(1)—C(2)—C(7)—C(6) | 177.9 (3) |

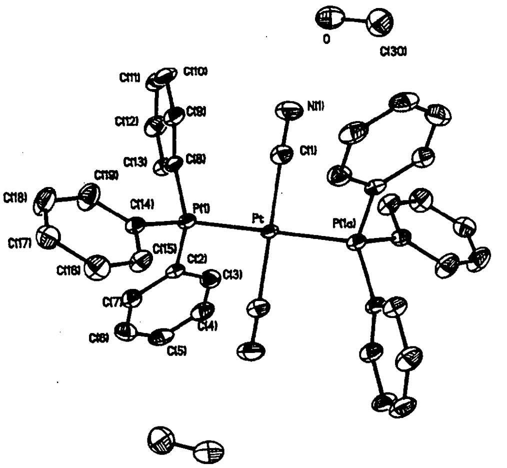

Fig. 1. A drawing of the *trans*-[Pt(CN)$_2$(PPh$_3$)$_2$].2CH$_3$OH structure. Thermal ellipsoids have been drawn at the 50% probability level.

take this opportunity briefly to describe the device used in our laboratory. An FTS systems (XR-85-1) refrigeration unit is used to cool dried air. The air arrives *via* an air compressor (Ingersoll-Rand, type 30, model 234) which has been set to supply at least 100 p.s.i. ($\sim 6.895 \times 10^5$ Pa) continuously and is filtered by three air-filtering systems. The first filter (Air Dryer, A0050Br) removes the bulk water from the air, the second filter (FTS systems, AD-80X) removes the final traces of water and controls the pressure supplied to the flow meter. The third filter is a Wilkerson Aerosol filter (M-1020-02-x00) which is used to remove trace particles. This system allows for variable air flow and can maintain a constant maximum flow of 30 Mmol min$^{-1}$. The temperature is controlled by a digital temperature indicator/controller (FTS systems, TC-44), which controls the temperature of the air to within ±0.2 K. The temperature was measured at the crystal position using a thermocouple which agrees with the set temperature. The minimum temperature we can reach with reliability using this system is 213 K. Frost is kept to a minimum by use of the typical nozzle heater and a frost plate.

Refined cell parameters were obtained from the setting angles of 25 reflections with $20 < 2\theta < 30°$. Triclinic symmetry was found on the basis of interaxial angles confirmed by Delaunay reduction and axial photographs. Intensities were measured for 2598 unique reflections of which 2303 had $|F_o|^2 > 3\sigma|F_o|^2$ for $3 < 2\theta < 45°$. Scan rate was variable, 2.9–29° min$^{-1}$. Three standards, measured every 97 data, showed no decay. Data were corrected for Lorentz and polarization effects. Structure solution and refinement was carried out using the *SHELXTL* collection of crystallographic software (Sheldrick, 1986). The Pt-atom position was determined by direct methods (*SOLV*; Sheldrick, 1986) and was found to be on an inversion center. Remaining atomic positions were determined from difference Fourier maps. Scattering factors, including terms for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All non-H atoms were refined anisotropically. H atoms were placed in idealized positions with $U_{\text{iso}} = 1.2 \times C_{\text{iso}}$; the H atom on the solvent O atom was not modeled. Refinement was based on $F$ with weights of the form $w^{-1} = [\sigma^2(F_o) + |g|(F_o^2)]$. The value of $g$ was refined by fitting $(F_o - F_c)^2$ to $[\sigma^2(F_o) + gF^2]/k$ ($k$ = scale factor) to put weights on an approximately absolute scale; final refined $g = 0.00042$. The original data, without an absorption correction, refined to a conventional $R = 0.0367$, $wR = 0.0378$ (GOF = 1.222). An absorption correction was applied to the isotropic model by use of *DIFABS* (Walker & Stuart, 1983). Convergence to conventional $R$ values of $R = 0.0174$ and $wR = 0.0177$ (GOF = 1.001) was obtained using 214 variable parameters and 2303 reflections with $|F_o|^2 > 3\sigma|F_o|^2$. The largest shift/e.s.d. in the last least-squares cycle was 0.005; the maximum and minimum residual electron densities in the final difference Fourier map were 0.66 and $-0.57$ e Å$^{-3}$, respectively.

The Pt atom lies on the inversion center. The bond distances for Pt—C and Pt—P of 1.991 (4) and 2.332 (1) Å are in the normal range. The bond angles for P—Pt—P′ and C—Pt—C are required by symmetry to be 180°. The bond angle of Pt—C—N is a normal value of 178.7°. A thermal ellipsoid diagram showing the molecular structure of $[\mathrm{Pt(CN)_2(PPh_3)_2}]\mathrm{.2CH_3OH}$ is shown in Fig. 1. Final positional and thermal parameters are presented in Table 1.* Selected interatomic distances, angles and torsion angles are listed in Table 2.

**Related literature.** The structures of the related compounds *trans*-$[\mathrm{Pt(CNMe)_2\{C(NHCH_3)(SCH_2CH_3)\}_2]2PF_6}$ (Buttler & Enemark, 1973) and *cis*-$[\mathrm{PtCl_2(CNEt)(PEt_2Ph)}]$, *cis*-$[\mathrm{PtCl_2(CNPh)_2}]$ (Jovanović & Manojlovic-Muir, 1972) have been characterized.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55319 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0418]

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# Structure of Tetrakis(*N*,*N*-dimethylbenzenecarbothioamide-*S*)cadmium(II) Diperchlorate Monohydrate

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**Abstract.** $[\mathrm{Cd(C_9H_{11}NS)_4](ClO_4)_2.H_2O}$, $M_r = 990.33$, orthorhombic, $Pbca$, $a = 17.488$ (3), $b = 22.287$ (4), $c = 22.811$ (3) Å, $V = 8890.8$ Å$^3$, $Z = 8$, $D_x = 1.480$ g cm$^{-3}$, $\lambda(\mathrm{Mo}\ K\alpha) = 0.70930$ Å, $\mu = 8.42$ cm$^{-1}$, $F(000) = 4064$, $T = 293$ K, final $R = 0.044$ for 4255 observed $[I > 3\sigma(I)]$ reflections. In the

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