$4 < 2\theta < 60°$, $-4 \le h \le 18$, $-7 \le k \le 7$, $-2 \le l \le 14$ were measured and yielded 1230 unique reflections, $R_{int} = 0{\cdot}034$. Of those, 1026 were considered observed on the basis that $F_o \ge 5\sigma(F_o)$. The data were corrected for Lorentz and polarization effects. An analytical absorption correction was made; transmission factors ranged from 0·012 to 0·069; $\sigma(F_o)$ was calculated from counting statistics. The structure was solved and refined with *SHELX*76 (Sheldrick, 1976). The cation positions were obtained by direct methods, and oxygen positions from a difference Fourier map. A full-matrix least-squares refinement of 77 parameters minimized $\sum w(|F_o| - |F_c|)^2$, $w = 1{\cdot}0/[\sigma^2(F_o) + 0{\cdot}0015F_o^2]$. The refinement was carried out with anisotropic thermal parameters and an extinction correction $F_o = F_o(1 - 5 \times 10^{-8}F_o^2/\sin\theta)$; $S = 1{\cdot}4$, $(\Delta/\sigma)_{max} = 0{\cdot}0000$, $R = 0{\cdot}050$, $wR = 0{\cdot}053$ for 1026 observed and $R = 0{\cdot}061$, $wR = 0{\cdot}060$ for all reflections. A final $\Delta\rho$ map gave peaks <1 e Å^{-3} except in the vicinity of heavy atoms where 5 e Å^{-3} ripples were observed. Scattering factors for neutral atoms, corrected for real and imaginary parts of dispersion, were obtained from *International Tables for X-ray Crystallography* (1974). Positional and thermal parameters are listed in Table 1, bond lengths and angles are given in Table 2, and a stereographic view of the structure is shown in Fig. 1.*

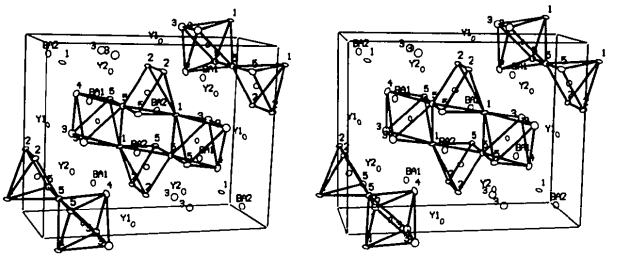

Fig. 1. Stereoview of the structure of $Y_2Ba_2CuPtO_8$. The *a* axis is horizontal and *c* vertical. Cu is in square-pyramidal and Pt in octahedral coordination.

Related literature. The structure of $YBa_2Cu_3O_{7-y}$ has been determined by neutron powder diffraction analysis (Cox, Moodenbaugh, Hurst & Jones, 1987; Capponi *et al.*, 1987; Beno *et al.*, 1987; Beech, Miraglia, Santoro & Roth, 1987). The structure of the related $YBa_2Cu_3O_6$ has been determined by single-crystal X-ray analysis by Swinnea & Steinfink (1987).

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* Tables of anisotropic thermal parameters and of structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44229 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BEECH, F., MIRAGLIA, S., SANTORO, A. & ROTH, R. S. (1987). *Phys. Rev. B*, **35**, 8778–8781.

BENO, M. A., SODERHOLM, L., CAPONE, D. W., HINKS, D. G., JORGENSEN, J. D., SCHULLER, I. K., SEGRE, C. U., ZHANG, K. & GRACE, J. D. (1987). *Appl. Phys. Lett.* **51**, 57–59.

CAPPONI, J. J., CHAILLOUT, C., HEWAT, A. W., LEJAY, P., MAREZIO, M., NGUYEN, N., RAVEAU, B., SOUBEYROUX, J., THOLENCE, J. L. & TOURNIER, R. (1987). *Europhys. Lett.* In the press.

COX, D. E., MOODENBAUGH, A. R., HURST, J. J. & JONES, R. H. (1987). *J. Phys. Chem. Solids.* In the press.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

STEINFINK, H., SWINNEA, J. S., SUI, Z. T., HSU, H. M. & GOODENOUGH, J. B. (1987). *J. Am. Chem. Soc.* **109**, 3348–3353.

SWINNEA, J. S. & STEINFINK, H. (1987). *J. Mater. Res.* In the press.

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Tetraphenylphosphonium-octachlorodirhenat(III)–Dichloromethan (1/2)

VON FRANK WELLER, KAY JANSEN UND KURT DEHNICKE

Fachbereich Chemie der Universität Marburg, Hans Meerwein-Straße, D-3550 Marburg, Bundesrepublik Deutschland

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Abstract. $[P(C_6H_5)_4]_2[Re_2Cl_8].2CH_2Cl_2$, $M_r = 1504{\cdot}7$, triclinic, $P\bar{1}$, $a = 10{\cdot}615$ (3), $b = 11{\cdot}589$ (2), $c = 12{\cdot}345$ (1) Å, $\alpha = 84{\cdot}11$ (1), $\beta = 71{\cdot}23$ (2), $\gamma = 70{\cdot}65$ (2)°, $V = 1350{\cdot}1$ Å^3, $Z = 1$, $D_x = 1{\cdot}84$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 4{\cdot}896$ mm^{-1}, $F(000) = 728$, $T = 293$ K, $R = 0{\cdot}041$ for 2873 observed independent reflexions. $[P(C_6H_5)_4]_2[Re_2Cl_8].2CH_2Cl_2$ was prepared by the reaction of $[P(C_6H_5)_4]_2[Re_2Cl_9]$ with *N,N'*-dichloro-1,4-benzoquinone diimine in CH_2Cl_2 solution; it crystallizes upon cooling of the solution. The

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Tabelle 1. *Atomkoordinaten und äquivalente Temperaturfaktoren* (Å²) *für* $(PPh_4)_2[Re_2Cl_8].2CH_2Cl_2$

$U_{äq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{äq}$
Re	0,38373 (5)	0,02668 (4)	0,02928 (4)	0,0374 (4)
Cl(1)	0,3256 (3)	0,1039 (3)	−0,1361 (3)	0,060 (3)
Cl(2)	0,3156 (3)	0,2268 (2)	0,0900 (3)	0,061 (3)
Cl(3)	0,3374 (3)	−0,1476 (2)	0,0019 (3)	0,060 (3)
Cl(4)	0,3293 (3)	−0,0272 (3)	0,2218 (2)	0,064 (3)
Cl(5)	0,8577 (5)	0,0242 (4)	0,3042 (3)	0,106 (5)
Cl(6)	0,9851 (5)	0,1550 (4)	0,1115 (4)	0,109 (5)
C(1)	0,924 (2)	0,033 (1)	0,156 (1)	0,10 (2)
P	0,2219 (3)	0,4642 (2)	0,6466 (2)	0,038 (2)
C(12)	0,4396 (7)	0,2528 (6)	0,5602 (5)	0,054 (3)
C(13)	0,5084 (7)	0,1529 (6)	0,4858 (5)	0,068 (3)
C(14)	0,4566 (7)	0,1413 (6)	0,3985 (5)	0,069 (3)
C(15)	0,3359 (7)	0,2295 (6)	0,3856 (5)	0,068 (3)
C(16)	0,2670 (7)	0,3294 (6)	0,4601 (5)	0,055 (3)
C(11)	0,3188 (7)	0,3411 (6)	0,5474 (5)	0,039 (2)
C(22)	0,3809 (7)	0,3989 (4)	0,7948 (5)	0,048 (3)
C(23)	0,4622 (7)	0,4188 (4)	0,8555 (5)	0,054 (3)
C(24)	0,4883 (7)	0,5303 (4)	0,8462 (5)	0,055 (3)
C(25)	0,4330 (7)	0,6219 (4)	0,7762 (5)	0,053 (3)
C(26)	0,3517 (7)	0,6020 (4)	0,7155 (5)	0,044 (3)
C(21)	0,3256 (7)	0,4905 (4)	0,7248 (5)	0,035 (2)
C(32)	0,0347 (7)	0,3322 (6)	0,7263 (5)	0,054 (3)
C(33)	−0,0809 (7)	0,3073 (6)	0,8053 (5)	0,066 (3)
C(34)	−0,1555 (7)	0,3791 (6)	0,9036 (5)	0,059 (3)
C(35)	−0,1146 (7)	0,4759 (6)	0,9228 (5)	0,056 (3)
C(36)	0,0010 (7)	0,5008 (6)	0,8438 (5)	0,049 (3)
C(31)	0,0756 (7)	0,4289 (6)	0,7456 (5)	0,040 (2)
C(42)	0,2612 (5)	0,6192 (5)	0,4646 (5)	0,052 (3)
C(43)	0,2281 (5)	0,7295 (5)	0,4067 (5)	0,060 (3)
C(44)	0,1004 (5)	0,8203 (5)	0,4519 (5)	0,059 (3)
C(45)	0,0059 (5)	0,8007 (5)	0,5549 (5)	0,059 (3)
C(46)	0,0390 (5)	0,6904 (5)	0,6128 (5)	0,049 (3)
C(41)	0,1666 (5)	0,5997 (5)	0,5676 (5)	0,039 (2)
H(12)	0,4797 (7)	0,2619 (6)	0,6279 (5)	0,09 (2)
H(13)	0,6019 (7)	0,0846 (6)	0,4958 (5)	0,09 (2)
H(14)	0,5099 (7)	0,0639 (6)	0,3409 (5)	0,09 (2)
H(15)	0,2957 (7)	0,2205 (6)	0,3180 (5)	0,09 (2)
H(16)	0,1735 (7)	0,3977 (6)	0,4501 (5)	0,09 (2)
H(22)	0,3607 (7)	0,3125 (4)	0,8021 (5)	0,09 (2)
H(23)	0,5051 (7)	0,3479 (4)	0,9097 (5)	0,09 (2)
H(24)	0,5513 (7)	0,5458 (4)	0,8932 (5)	0,09 (2)
H(25)	0,4532 (7)	0,7083 (4)	0,7689 (5)	0,09 (2)
H(26)	0,3089 (7)	0,6729 (4)	0,6613 (5)	0,09 (2)
H(32)	0,0925 (7)	0,2766 (6)	0,6502 (5)	0,10 (2)
H(33)	−0,1126 (7)	0,2324 (6)	0,7904 (5)	0,10 (2)
H(34)	−0,2450 (7)	0,3599 (6)	0,9647 (5)	0,10 (2)
H(35)	−0,1724 (7)	0,5315 (6)	0,9989 (5)	0,10 (2)
H(36)	0,0326 (7)	0,5756 (6)	0,8588 (5)	0,10 (2)
H(42)	0,3600 (5)	0,5489 (5)	0,4296 (5)	0,11 (2)
H(43)	0,3012 (5)	0,7446 (5)	0,3270 (5)	0,11 (2)
H(44)	0,0748 (5)	0,9057 (5)	0,4071 (5)	0,11 (2)
H(45)	−0,0929 (5)	0,8710 (5)	0,5899 (5)	0,11 (2)
H(46)	−0,0342 (5)	0,6753 (5)	0,6926 (5)	0,11 (2)

Tabelle 2. *Interatomare Abstände* (Å) *und Winkel* (°)

Re–Re	2,222 (1)	Cl(1)–Re–Cl(2)	87,8 (1)
Re–Cl(1)	2,329 (3)	Cl(1)–Re–Cl(3)	86,4 (1)
Re–Cl(2)	2,316 (3)	Cl(1)–Re–Cl(4)	153,2 (1)
Re–Cl(3)	2,325 (2)	Cl(2)–Re–Cl(3)	152,7 (1)
Re–Cl(4)	2,329 (3)	Cl(2)–Re–Cl(4)	87,0 (1)
		Cl(3)–Re–Cl(4)	86,3 (1)
C(1)–Cl(5)	1,75 (2)		
C(1)–Cl(6)	1,71 (2)	Re′–Re–Cl(1)	102,95 (1)
		Re′–Re–Cl(2)	103,23 (1)
P–C(11)	1,781 (7)	Re′–Re–Cl(3)	104,05 (0)
P–C(21)	1,791 (6)	Re′–Re–Cl(4)	103,78 (1)
P–C(31)	1,780 (7)		
P–C(41)	1,790 (7)	Cl(5)–C(1)–Cl(6)	113,1 (9)
		C(11)–P–C(21)	112,0 (3)
		C(11)–P–C(31)	109,0 (3)
		C(11)–P–C(41)	108,0 (3)
		C(21)–P–C(31)	108,5 (3)
		C(21)–P–C(41)	108,2 (3)
		C(31)–P–C(41)	111,2 (3)

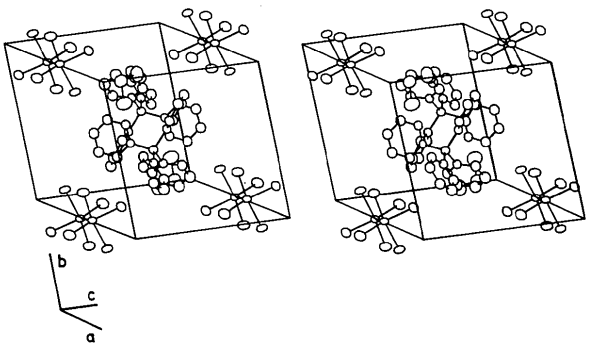

Fig. 1. Die Struktur des $[P(C_6H_5)_4]_2[Re_2Cl_8].2CH_2Cl_2$ in stereoskopischer Darstellung der Elementarzelle.

structure consists of $P(C_6H_5)_4^+$ cations and $Re_2Cl_8^{2-}$ anions with virtual D_{4h} (4/*mmm*) symmetry. Bond distances Re–Re = 2·222 (1), Re–Cl (average) = 2·325 (3) Å.

Experimentelles. Synthese: Man stellt zunächst $[P(C_6H_5)_4]_2[Re_2Cl_9]$ her, indem man 1,80 g $\{H[OP(C_6H_5)_3]_2\}_2[Re_2Cl_9]$ (Gehrke, Eastland, Haas & Carlson, 1971) (1,0 mmol) mit 0,76 g $P(C_6H_5)_4N_3$ (2,0 mmol) in 15 ml CH_2Cl_2 48 h rührt, filtriert, mit CH_2Cl_2 und Toluol wäscht und bei 323 K i.Vak. trocknet. Ausbeute 0,9 g (66%) dunkelviolette Kristalle. 0,80 g $[P(C_6H_5)_4]_2[Re_2Cl_9]$ (0,59 mmol) werden in 50 ml CH_2Cl_2 mit 0,105 g *N,N′*-dichlor-1,4-benzochinon diimin ($C_6H_4N_2Cl_2$) (0,60 mmol) 12 h bei RT gerührt, vom Ungelösten filtriert und mehrere Tage auf 278 K gekühlt. Es entstehen 0,21 g grüne Kristallsäulen (53%). IR-Absorptionen: 340 *st*, 304 *m* (*ν*ReCl) cm^{-1}.

Kristallabmessungen: 0,21 × 0,16 × 0,12 mm; Diffraktometer Enraf–Nonius CAD-4; Gitterparameter aus 12 θ-Werten $12 < \theta < 18°$, ω-scan; Kontrollreflexe 122 und $\bar{3}\bar{3}1$ mit Intensitätsschwankungen ≤ 3,7%. Gemessen 3501 Reflexe, 3279 unabhängig, 2873 für die Rechnung verwandt $[F_o > 3\sigma(F_o)]$; $R_{int} = 0{,}023$. Re- und Cl-Lagen aus Patterson-Synthese, übrige Atome aus Differenz-Fourier-Synthese; Verfeinerung durch Minimalisieren von $\sum w(|F_o| - |F_c|)^2$ mit $w = 1/\sigma^2(F)$; bis Parameterverschiebungen <0,054; $R = 0{,}041$, $wR = 0{,}038$; bei Einschluß der unbeobachteten Reflexe $[F = 1/8\sigma(F)]$ $R = 0{,}051$; größter Peak in abschließender Differenz-Fourier-Synthese | 1,5 | e Å^{-3}. Phenylringe als starre Gruppen mit *r*CC = 1,395 und *r*CH = 1,08 Å, wobei gleiche isotrope Temperaturfaktoren für die H-Atome in einer Phenylgruppe zugrundegelegt wurden. Atomformfaktoren nach Cromer & Mann (1968), *f′* und *f″* nach Cromer & Liberman (1970). Rechenprogramme: Müller (1971), Sheldrick (1976), Johnson (1965).

In Tabelle 1 sind die Atomparameter,* in Tabelle 2 die Bindungsabstände und -winkel zusammengestellt. Fig. 1 zeigt die Struktur in stereoskopischer Darstellung.

Verwandte Literatur. Das $[Re_2Cl_8]^{2-}$-Ion ist mit mehreren Kationen bekannt: Überblick (Cotton & Walton, 1982), $K_2[Re_2Cl_8].2H_2O$ (Cotton & Harris, 1965), $(NH_4)_2[Re_2Cl_8].2H_2O$ (Koz'min, Surazhskaya & Larina, 1979), $(C_5H_5NH)_2[Re_2Cl_8]$ (Bratton & Cotton, 1969), $(Bu_4N)_2[Re_2Cl_8]$ (Cotton, Frenz, Stults & Webb, 1976), $[(DMF)_2H]_2[Re_2Cl_8]$, $[(CH_3)_2NH_2]_2[Re_2Cl_8]$ (Koz'min, Kotel'nikova, Surazhskaya, Larina, Bagirov & Misailova, 1978).

* Die Liste der anisotropen Temperaturfaktoren und der beobachteten und berechneten Strukturfaktoren sind bei der British Library Document Supply Centre (Supplementary Publication No. SUP 44268: 21 pp.) hinterlegt. Kopien sind erhältlich durch: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Literatur

BRATTON, W. K. & COTTON, F. A. (1969). *Inorg. Chem.* **8**, 1299–1304.

COTTON, F. A., FRENZ, B. A., STULTS, B. R. & WEBB, T. R. (1976). *J. Am. Chem. Soc.* **98**, 2768–2773.

COTTON, F. A. & HARRIS, C. B. (1965). *Inorg. Chem.* **4**, 330–333.

COTTON, F. A. & WALTON, R. A. (1982). *Multiple Bonds between Metal Atoms*. New York: John Wiley.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1897.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

GEHRKE, H., EASTLAND, G., HAAS, L. & CARLSON, G. (1971). *Inorg. Chem.* **10**, 2328–2329.

JOHNSON, C. K. (1965). *ORTEP*. Bericht ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

KOZ'MIN, P. A., KOTEL'NIKOVA, A. S., SURAZHSKAYA, M. D., LARINA, T. B., BAGIROV, S. A. & MISAILOVA, T. V. (1978). *Koord. Khim.* **4**, 1557–1563.

KOZ'MIN, P. A., SURAZHSKAYA, M. D. & LARINA, T. B. (1979). *Koord. Khim.* **5**, 752–753.

MÜLLER, U. (1971). *CADLP*. Programm zur Auswertung und Lp-Korrektur von Diffraktometerdaten. Univ. Marburg, Bundesrepublik Deutschland.

SHELDRICK, G. M. (1976). *SHELX*76. Programm für die Strukturbestimmung. Univ. Cambridge, England.

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trans-Bis[dicyclohexylphosphino-*N*-phenyl(thioformamido)]platinum(II): $Pt[(c\text{-}C_6H_{11})_2PC(S)=N(C_6H_5)]_2$

BY DAINIS DAKTERNIEKS

Division of Chemical and Physical Sciences, Deakin University, Waurn Ponds, Victoria 3217, *Australia*

MADELINE CORBETT, BERNARD F. HOSKINS AND PATRICIA A. JACKSON

Department of Inorganic Chemistry, University of Melbourne, Parkville 3052, *Australia*

AND EDWARD R. T. TIEKINK

Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, South Australia 5001, *Australia*

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Abstract. $[Pt(C_{19}H_{27}NPS)_2]$, $M_r = 860{\cdot}0$, monoclinic, $P2_1/c$, $a = 12{\cdot}307\ (2)$, $b = 8{\cdot}456\ (2)$, $c = 18{\cdot}673\ (4)$ Å, $\beta = 104{\cdot}26\ (2)°$, $U = 1883\ (2)$ Å^3, D_m (by flotation) $= 1{\cdot}50\ (1)$, $D_x = 1{\cdot}517$ Mg m^{-3} for $Z = 2$, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 3{\cdot}948$ mm^{-1}, $F(000) = 872$, $T = 295\ (2)$ K, $R = 0{\cdot}024$ for 3164 reflections with $I \geq 2{\cdot}5\sigma(I)$. The anion coordinates *via* the P and S atoms [Pt–P(1) 2·282 (1), Pt–S(1) 2·322 (1) Å and P(1)–Pt–S(1) 74·7 (1)°]; the Pt atom is situated at a centre of inversion (at 0,0,0) so that the *trans*-PtP_2S_2 group is constrained to planarity. The four-membered chelate ring is not planar, however, with the dihedral angle for PtP(1)S(1)/P(1)S(1)C(1) being 7·4°; further, the Pt atom lies 0·3316 (1) Å above the least-squares plane through the P(1), S(1), C(1) and N(1) atoms.

Experimental. The title compound was synthesized by adding excess $N(C_2H_5)_3$ to a stirred dichloromethane solution of $Cl_2Pt[(C_6H_5)CN]_2$ (Hartley, 1973) and $(c\text{-}C_6H_{11})_2PC(S)N(H)(C_6H_5)$ (Kunze & Antoniadis, 1981) in a 1:2 ratio; crystals obtained by the slow evaporation of the filtered solution. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; $\omega{:}2\theta$ scan technique. Cell parameters on crystal 0·41 × 0·16 × 0·21 mm from least-squares

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