structure with regular polyhedra (and ignoring hydrogen bonds); here the O(1) and O(2) anions have their requirements satisfied, whereas the O(3) and OH anions have low bond-strength sums. According to bond-valence theory (Brown, 1981), the bonds to OH and O(3) will shorten in order to increase their bond-valence sums, and the magnitudes of the Pauling bond-strength sums (0·67 and 1·58, respectively) indicate that this contraction should be considerable. The most common type of distortion of Cu^{2+} octahedra resulting from the Jahn–Teller effect is the shortening of four meridional bonds and the lengthening of two axial bonds. In the present case, the need to shorten Cu(2)—OH and Cu(2)—O(3) defines the two axial bonds as Cu(2)—O(2) and Cu(2)—OW(3). As OW(3) is an H_2O group, its bond-valence requirements are already (over-) satisfied and this bond can very easily lengthen (to 2·8 Å in this case). Similarly, O(2) is bonded to Cu(2) and Cu(1), and hence a cooperative distortion involving these two octahedra can lengthen Cu(2)—O(2) and shorten Cu(1)—O(2), leaving the bond-valence requirements of O(2) satisfied. Thus the polyhedral distortions within the chain can be seen as resulting from the interaction of polyhedral connectivity and local bond-valence requirements (as is the case for all structures), coupled with the need to incorporate the characteristic local distortions that occur as a result of the Jahn–Teller effect. It is this latter factor that has adversely affected our understanding of topological (graphical) structure relations in Cu^{2+} oxysalts.

References

BROWN, I. D. (1981). *Structure and Bonding in Crystals,* Vol. 2, pp. 1–30. New York: Academic Press.
DOLLASE, W. A. (1968). *Am. Mineral.* **53**, 1882–1898.
FINNEY, J. J. (1966). *Acta Cryst.* **21**, 437–440.
GROAT, L. A. & HAWTHORNE, F. C. (1987). *Tschermaks Mineral Petrogr. Mitt.* **37**, 87–96.
HAWTHORNE, F. C. (1983). *Acta Cryst.* A**39**, 724–736.
HAWTHORNE, F. C. (1985a). *Am. Mineral.* **70**, 455–473.
HAWTHORNE, F. C. (1985b). *Mineral Mag.* **49**, 85–91.
HAWTHORNE, F. C. (1985c). *Tschermaks Mineral Petrogr. Mitt.* **34**, 15–34.
HAWTHORNE, F. C. (1986a). *Can. Mineral.* **24**, 625–642.
HAWTHORNE, F. C. (1986b). *Am. Mineral.* **71**, 206–209.
HAWTHORNE, F. C. & EBY, R. K. (1985). *Neues Jahrb. Mineral. Monatsh.* pp. 234–240.
HAWTHORNE, F. C. & GROAT, L. A. (1985). *Am. Mineral.* **70**, 1050–1055.
HAWTHORNE, F. C. & GROAT, L. A. (1986). *Mineral Mag.* **50**, 157–162.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
SHELDRICK, G. M. (1981). *Nicolet SHELXTL Operations Manual,* revision 3. Nicolet XRD Corporation, Cupertino, California, USA.

Structure of Bis(tetraphenyldiphosphinomethane)digold(I) Dinitrate

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Abstract. Bis-μ-[bis(diphenylphosphino)methane-*P,P'*]-digold(I) dinitrate, $[Au_2(C_{25}H_{22}P_2)_2](NO_3)_2$, M_r = 1286·7, monoclinic, $P2_1/c$, a = 20·301 (8), b = 13·657 (4), c = 23·515 (8) Å, β = 133·92 (2)°, V = 4696 (3) Å^3, Z = 4, D_x = 1·82 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 64·1 cm^{-1}, $F(000)$ = 2496, T = 295 K. Final R = 0·0447 for 3579 observed reflections $[F_o^2 > 3\sigma(F_o^2)]$. The eight-membered ring, formed by two Au atoms and two bis(diphenylphosphino)methane ligands, has a chair conformation with bond angles P(1)—Au(1)—P(2) = 170·6 (1) and P(3)—Au(2)—P(4) = 176·8 (2)°. The molecule contains a short Au···Au distance, 2·988 (1) Å. One nitrate is weakly interacting with the $Au_2(dppm)_2$ unit with Au(1)···O(3) = 2·987 (8) Å.

Introduction. Numerous binuclear gold(I) compounds with various ligands have short gold—gold

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distances (Mazany & Fackler, 1984; Basil, Murray, Fackler, Tocher, Mazany, Trzcinska-Bancroft, Knachel, Dudis, Delord & Marler, 1985; Khan, Fackler, King, Wang, Wang, 1988; Schmidbaur, Wohlleben, Schubert, Frank & Huttner, 1977; Shain & Fackler, 1987; Khan, King, Heinrich, Fackler & Porter, 1989). These compounds are luminescent; some have long excited-state lifetimes (King, Wang, Khan & Fackler, 1989). It is believed that these luminescent properties are related to the gold–gold interaction. In $Au_2(dppm)_2X_2$ compounds, where dppm = bis(diphenylphosphino)methane and $X = NO_3^-$, BF_4^- or Cl^-, different X gives different emission maxima in the solid state (King et al., 1989). These differences suggest that these compounds have different crystal structures. In this paper, we report the crystal structure of $[Au_2(dppm)_2](NO_3)_2$.

Experimental. $[Au_2(dppm)_2](NO_3)_2$ was synthesized by adding excess $AgNO_3$ to a CH_2Cl_2 solution of $Au_2(dppm)_2Cl_2$ (Schmidbaur et al., 1977) in the dark. Crystals suitable for X-ray analysis were obtained by crystallization from a CH_2Cl_2/diethyl ether solution. A colorless thin plate crystal of dimensions $0.1 \times 0.3 \times 0.4$ mm was selected for study. Cell constants were derived from least-squares refinement of 25 reflections having $20 < 2\theta < 25°$. Intensity data were collected using the ω-scan technique with $0 < 2\theta < 45°$ ($h = -19$ to 19, $k = 0$ to 15, $l = 0$ to 22) on a Nicolet $R3m/E$ diffractometer using graphite-monochromated Mo $K\alpha$ radiation. Scan rate varied from 2.93 to 29.30° min^{-1} with scan width 2.0°. Three standard reflections (021, $\bar{1}10$, 130), measured every 97 reflections, showed small (<5%) random variations. Data were corrected for Lorentz and polarization effects. An empirical absorption correction was applied; $T_{min} = 0.494$, $T_{max} = 0.981$. The 6666 data collected were averaged to 3579 unique observed reflections $[F_o^2 > 3\sigma(F_o^2); R_{int}(F_o) = 0.0385]$. The space group was $P2_1/c$ (systematic absences: $h0l$, $l = $ odd; $0k0$, $k = $ odd). Scattering factors, including anomalous-dispersion corrections, were taken from International Tables for X-ray Crystallography (1974). All computations were carried out using the SHELXTL crystallographic package (Sheldrick, 1986).

Direct methods were used to find the positions of the two Au atoms. All other non-H atoms were located by Fourier methods. Phenyl rings were refined as rigid groups with C—C = 1.395 Å and C—C—C = 120° and with isotropic thermal parameters; H atoms were placed in calculated positions (C—H = 0.960 Å) with fixed displacement parameters $(U_{iso} = 0.07 Å^2)$. The Au, P, and methylene C atoms were refined anisotropically. $\Sigma w(|F_o| - |F_c|)^2$ was minimized, where $w = [\sigma^2(F_o) + 0.00024F_o^2]^{-1}$. A partial correction for secondary extinction was

applied by multiplying F_c by $[1 + 0.002xF_c^2 \times (\sin 2\theta)]^{-1/4}$; refinement gave $x = 0.0002$. Refinement converged to $R = 0.0447$, $wR = 0.0478$ and $S = 1.272$ based on 242 variables and 3579 observed reflections. The largest shift/e.s.d. in the final least-squares cycle was 0.007; maximum and minimum residual electron densities in the difference Fourier map were 1.04 and -0.75 e Å^{-3}.

Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for* $[Au_2(dppm)_2](NO_3)_2$

E.s.d.'s in the least-significant digits are given in parentheses.

	x	y	z	U_{iso}
Au(1)	2347 (1)	6524 (1)	5531 (1)	28 (1)*
Au(2)	3249 (1)	6731 (1)	4936 (1)	32 (1)*
P(1)	1059 (3)	7321 (3)	4440 (2)	28 (3)*
P(2)	3502 (3)	5490 (3)	6529 (2)	26 (3)*
P(3)	1956 (3)	7528 (3)	3818 (2)	30 (3)*
P(4)	4517 (3)	5859 (3)	6009 (2)	31 (3)*
C(1)	939 (10)	7178 (11)	3588 (8)	34 (13)*
C(2)	4573 (9)	5792 (10)	6812 (7)	23 (10)*
N(1)	3870 (8)	6557 (10)	1696 (7)	44 (10)*
N(2)	1156 (10)	4305 (11)	3236 (9)	60 (13)*
O(1)	3795 (7)	5669 (8)	1760 (7)	53 (10)*
O(2)	4153 (8)	6864 (8)	1408 (7)	60 (11)*
O(3)	3609 (8)	7152 (9)	1924 (7)	64 (11)*
O(4)	1028 (11)	3480 (10)	3013 (8)	99 (15)*
O(5)	817 (12)	4586 (13)	3460 (10)	120 (19)*
O(6)	1592 (13)	4881 (15)	3227 (12)	168 (23)*
C(16)	219 (6)	9131 (7)	3874 (5)	46 (5)
C(15)	112 (6)	10108 (7)	3968 (4)	60 (5)
C(14)	732 (6)	10540 (7)	4714 (4)	72 (6)
C(13)	1459 (6)	9995 (7)	5366 (4)	68 (6)
C(12)	1566 (6)	9017 (7)	5273 (4)	46 (5)
C(11)	947 (6)	8585 (7)	4527 (4)	33 (4)
C(22)	-400 (7)	7208 (6)	4323 (6)	36 (4)
C(23)	-1067 (7)	6698 (6)	4227 (6)	60 (5)
C(24)	-1246 (7)	5719 (6)	3992 (6)	82 (7)
C(25)	-759 (7)	5251 (6)	3852 (6)	86 (7)
C(26)	-92 (7)	5762 (6)	3948 (6)	65 (6)
C(21)	88 (7)	6740 (6)	4184 (6)	31 (4)
C(32)	3623 (6)	3443 (7)	6724 (4)	41 (4)
C(33)	3380 (6)	2484 (7)	6442 (4)	53 (5)
C(34)	2737 (6)	2307 (7)	5631 (4)	54 (5)
C(35)	2337 (6)	3089 (7)	5102 (4)	57 (5)
C(36)	2580 (6)	4048 (7)	5384 (4)	37 (4)
C(31)	3222 (6)	4225 (7)	6195 (4)	30 (4)
C(42)	4534 (5)	5176 (7)	8162 (6)	39 (4)
C(43)	4633 (5)	5140 (7)	8811 (6)	53 (5)
C(44)	3922 (5)	5454 (7)	8736 (6)	60 (5)
C(45)	3112 (5)	5804 (7)	8013 (6)	45 (5)
C(46)	3013 (5)	5840 (7)	7364 (6)	41 (4)
C(41)	3724 (5)	5526 (7)	7438 (6)	27 (4)
C(52)	2791 (7)	9265 (7)	4632 (5)	50 (5)
C(53)	2891 (7)	10280 (7)	4711 (5)	67 (6)
C(54)	2241 (7)	10882 (7)	4064 (5)	59 (5)
C(55)	1493 (7)	10468 (7)	3337 (5)	83 (7)
C(56)	1393 (7)	9453 (7)	3258 (5)	65 (6)
C(51)	2042 (7)	8851 (7)	3906 (5)	30 (4)
C(66)	2370 (5)	6892 (7)	2992 (5)	45 (5)
C(65)	2228 (5)	6826 (7)	2322 (5)	57 (5)
C(64)	1400 (5)	7133 (7)	1591 (5)	56 (5)
C(63)	714 (5)	7506 (7)	1531 (5)	56 (5)
C(62)	856 (5)	7573 (7)	2202 (5)	40 (4)
C(61)	1684 (5)	7266 (7)	2932 (5)	25 (4)
C(76)	5711 (6)	7377 (7)	6465 (6)	55 (5)
C(75)	6563 (6)	7795 (7)	6861 (6)	63 (6)
C(74)	7336 (6)	7202 (7)	7278 (6)	50 (5)
C(73)	7258 (6)	6191 (7)	7300 (6)	76 (6)
C(72)	6407 (6)	5773 (7)	6904 (6)	70 (6)
C(71)	5634 (6)	6366 (7)	6486 (6)	37 (4)
C(86)	5027 (7)	3865 (8)	6299 (5)	43 (4)
C(85)	4978 (7)	2912 (8)	6057 (5)	56 (5)
C(84)	4395 (7)	2698 (8)	5255 (5)	70 (6)
C(83)	3862 (7)	3436 (8)	4695 (5)	71 (6)
C(82)	3911 (7)	4389 (8)	4936 (5)	49 (5)
C(81)	4494 (7)	4603 (8)	5738 (5)	38 (4)

* Equivalent isotropic U is defined as $\frac{1}{3}$ of the trace of the U_{ij} tensor.

Table 2. *Bond lengths (Å) and angles (°) for* [Au₂(dppm)₂](NO₃)₂

E.s.d.'s in the least-significant digits are given in parentheses.

Au(1)—Au(2)	2·988 (1)	Au(1)—P(1)	2·302 (3)
Au(1)—P(2)	2·316 (3)	Au(2)—P(3)	2·327 (3)
Au(2)—P(4)	2·324 (3)	P(1)—C(1)	1·853 (24)
P(1)—C(11)	1·772 (11)	P(1)—C(21)	1·798 (15)
P(2)—C(2)	1·827 (20)	P(2)—C(31)	1·818 (10)
P(2)—C(41)	1·854 (16)	P(3)—C(1)	1·800 (23)
P(3)—C(51)	1·813 (11)	P(3)—C(61)	1·782 (14)
P(4)—C(2)	1·812 (22)	P(4)—C(71)	1·830 (12)
P(4)—C(81)	1·818 (12)	N(1)—O(1)	1·244 (18)
N(1)—O(2)	1·228 (28)	N(1)—O(3)	1·273 (26)
N(2)—O(4)	1·192 (21)	N(2)—O(5)	1·184 (39)
N(2)—O(6)	1·195 (35)		

Au(2)—Au(1)—P(1)	91·4 (2)	Au(2)—Au(1)—P(2)	90·8 (2)
P(1)—Au(1)—P(2)	170·6 (1)	Au(1)—Au(2)—P(3)	89·8 (2)
Au(1)—Au(2)—P(4)	91·2 (2)	P(3)—Au(2)—P(4)	176·7 (2)
Au(1)—P(1)—C(1)	110·5 (5)	Au(1)—P(1)—C(11)	118·0 (3)
C(1)—P(1)—C(11)	107·2 (7)	Au(1)—P(1)—C(21)	107·4 (3)
C(1)—P(1)—C(21)	107·5 (7)	C(11)—P(1)—C(21)	105·6 (6)
Au(1)—P(2)—C(2)	110·1 (5)	Au(1)—P(2)—C(31)	110·4 (2)
C(2)—P(2)—C(31)	107·9 (7)	Au(1)—P(2)—C(41)	114·8 (4)
C(2)—P(2)—C(41)	107·4 (6)	C(31)—P(2)—C(41)	106·1 (6)
Au(2)—P(3)—C(1)	112·0 (5)	Au(2)—P(3)—C(51)	113·2 (3)
C(1)—P(3)—C(51)	107·4 (7)	Au(2)—P(3)—C(61)	114·8 (4)
C(1)—P(3)—C(61)	103·6 (7)	C(51)—P(3)—C(61)	105·1 (6)
Au(2)—P(4)—C(2)	112·2 (5)	Au(2)—P(4)—C(71)	116·1 (4)
C(2)—P(4)—C(71)	102·9 (6)	Au(2)—P(4)—C(81)	111·3 (3)
C(2)—P(4)—C(81)	106·6 (7)	C(71)—P(4)—C(81)	107·0 (6)
P(1)—C(1)—P(3)	112·6 (7)	P(2)—C(2)—P(4)	115·7 (6)
O(1)—N(1)—O(2)	122·8 (18)	O(1)—N(1)—O(3)	116·9 (20)
O(2)—N(1)—O(3)	120·3 (15)	O(4)—N(2)—O(5)	119·0 (24)
O(4)—N(2)—O(6)	123·6 (29)	O(5)—N(2)—O(6)	117·4 (21)
P(1)—C(11)—C(16)	121·1 (2)	P(1)—C(11)—C(12)	118·8 (2)
P(1)—C(21)—C(22)	120·8 (4)	P(1)—C(21)—C(26)	118·6 (4)
P(2)—C(31)—C(32)	121·9 (3)	P(2)—C(31)—C(36)	118·1 (3)
P(2)—C(41)—C(42)	122·4 (3)	P(2)—C(41)—C(46)	117·4 (3)
P(3)—C(51)—C(52)	118·7 (3)	P(3)—C(51)—C(56)	121·3 (3)
P(3)—C(61)—C(66)	118·5 (3)	P(3)—C(61)—C(62)	120·7 (3)
P(4)—C(71)—C(76)	118·4 (3)	P(4)—C(71)—C(72)	121·5 (3)
P(4)—C(81)—C(86)	122·4 (3)	P(4)—C(81)—C(82)	117·6 (3)

Discussion. Final positional and isotropic thermal parameters are presented in Table 1.* Bond lengths and angles are given in Table 2. The molecule and the atomic labelling scheme are shown in Fig. 1. A stereoscopic view of the unit cell is shown in Fig. 2.

Three types of interactions between X and Au₂(dppm)₂ have been reported for Au₂(dppm)₂X_2 compounds: (i) purely electrostatic interaction (X = BH₃CN⁻; Khan *et al.*, 1989), (ii) one X bridges the two gold centers and the other is uncoordinated (X = I⁻, Shain & Fackler, 1987), and (iii) an interaction in which each X is weakly coordinated to one gold center (X = Cl⁻, Br⁻; Schmidbaur *et al.*, 1977; Shain & Fackler, 1987). In the present crystal structure we see a new type of interaction: an O atom of one nitrate weakly interacts with one Au with Au(1)···O(3) distance 2·989 Å while a second oxygen atom from the same ligand interacts with Au(2) at a distance of 3·196 Å. The other nitrate is an uncoordinated counter ion.

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* Lists of structure factors, anisotropic thermal parameters and hydrogen parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51960 (41 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The eight-membered ring, formed by two Au atoms and two dppm ligands, has a chair conformation with Au(1)···Au(2) = 2·988 (1), Au—P = 2·301 (3)–2·327 (3), and P—C = 1·81 (2)–1·84 (2) Å. The coordination about Au(2), which has no short Au—O distance, is close to linear [P(3)—Au(2)—P(4) = 176·8 (2)°]. This deviation from linearity is similar to deviations found in other Au₂(dppm)₂X_2 compounds without short Au—X distances [P—Au—P = 174·6 (1)–175·3(1)°]. The P(1)—Au(1)—P(2) angle of 170·6 (1)° is about 10° from linear, presumably due to the short Au···O separation. The P—Au—P angle in the crystal structures of other Au₂(dppm)₂X_2 compounds with short Au···X separations is also non-linear [155·9 (1)–167·7 (2)°].
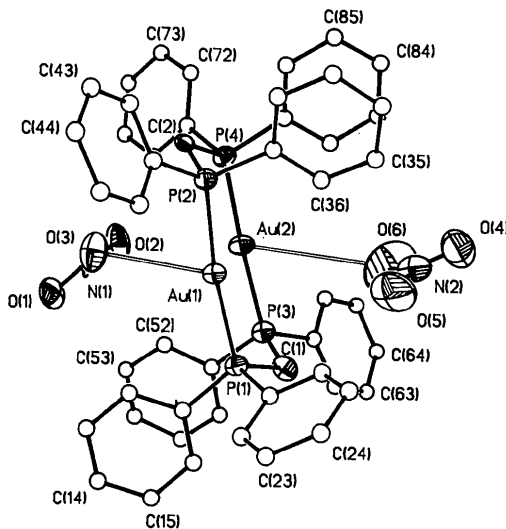

Fig. 1. A perspective view of [Au₂(dppm)₂](NO₃)₂. Thermal ellipsoids have been drawn at the 50% probability level. The shortest gold—nitrate distances are shown as open bonds. Au(1)···O(3) = 2·987 (8), Au(2)···O(2) = 3·196 (1), Au(1)···O(2) = 3·472 (11), and Au(2)···O(6) = 3·886 (13) Å.

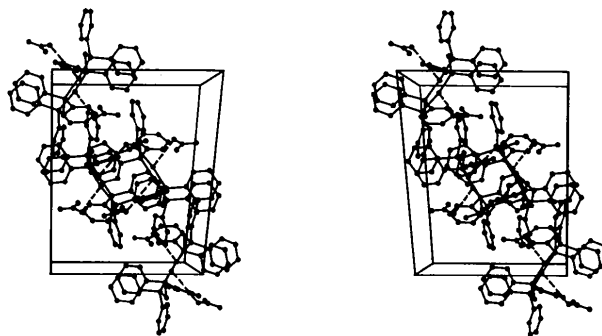Fig. 2. A stereoview packing diagram viewed down the *a* axis, with *b* horizontal and *c* vertical. Dashed lines: short Au···O contacts.

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References

BASIL, J. D., MURRAY, J. H., FACKLER, J. P. JR, TOCHER, J., MAZANY, A. M., TRZCINSKA-BANCROFT, B., KNACHEL, H., DUDIS, D., DELORD, T. J. & MARLER, D.O. (1985). *J. Am. Chem. Soc.* **107**, 6908–6915.

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

KHAN, M. N. I., FACKLER, J. P. JR, KING, C., WANG, J. C. & WANG, S. (1988). *Inorg. Chem.* **27**, 1672–1673.

KHAN, M. N. I., KING, C., HEINRICH, D. D., FACKLER, J. P. JR & PORTER, L. C. (1989). *Inorg. Chem.* **28**, 2150–2154.

KING, C., WANG, J. C., KHAN, M. N. I. & FACKLER, J. P. JR (1989). *Inorg. Chem.* **28**, 2145–2149.

MAZANY, A. M. & FACKLER, J. P. JR (1984). *J. Am. Chem. Soc.* **106**, 801–802.

SCHMIDBAUR, H., WOHLLEBEN, A., SCHUBERT, U., FRANK, A. & HUTTNER, G. (1977). *Chem. Ber.* **110**, 2751–2757.

SHAIN, J. & FACKLER, J. P. JR (1987). *Inorg. Chim. Acta*, **131**, 157–158.

SHELDRICK, G. M. (1986). *SHELXTL. An Integrated System for Solving, Refining, and Displaying Crystal Structures from Diffraction Data.* Nicolet XRD Corporation, Madison, WI, USA.

Structure of N,N'-Dimethyl-1,3,2-benzodiazapospholium Tetrachloroaluminate: a Heteronaphthalenic 10π-Electron Cation

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Abstract. $[C_8H_{10}N_2P][AlCl_4]$, $M_r = 333.95$, orthorhombic, *Pbca*, $a = 11.0690\,(5)$, $b = 18.4666\,(17)$, $c = 14.0975\,(12)$ Å, $V = 2881.63$ Å^3, $Z = 8$, $D_x = 1.540$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.97$ mm^{-1}, $F(000) = 1344$, $T = 293$ K, $R = 0.055$ for 1356 significant reflections. The structure consists of discrete cationic and anionic units with long-range interactions between the P and Cl centres. The cation is planar with short P—N and C—N bond lengths [1.643 (6) and 1.379 (9) Å respectively].

Introduction. Isolobal derivatives of the 10π-electron cationic framework (1) demonstrate a constant naphthalenic environment (Burford & Royan, 1989; Burford, Royan, Linden & Cameron, 1988, 1989). The importance of this system towards the development of new bonding arrangements for the non-metal elements has been highlighted by the identification of the first examples of stable *p*π bonding between the heavier elements of Groups 15 and 16 (1*a–c*). The isolobal (Hoffmann, 1982; Haas, 1984) relationship between tricoordinate nitrogen and

dicoordinate sulfur has prompted a structural investigation of the title compound (1*d*). In addition, this compound is a contribution to the limited, but expanding area of dicoordinate phosphorus cations (Cowley, Cushner & Szobota, 1978; Friedrich, Huttner, Luber & Schmidpeter, 1978; Pohl, 1979; Burford *et al.*, 1988).
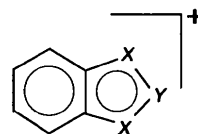
(a) Y = P; X = S
(b) Y = As; X = S
(c) Y = Sb; X = S
(d) Y = P; X = NMe

(1)

Experimental. The title compound was prepared in 23% yield from the reaction of equimolar quantities of 2-chloro-1,3-dimethyl-1,3,2-benzodiazaphosphole (Jennings, Randall, Worley & Harglo, 1981) and anhydrous $AlCl_3$ in CH_2Cl_2. Pale pink air-sensitive crystals were obtained by slow evaporation of CH_2Cl_2. Crystal $0.58 \times 0.46 \times 0.23$ mm (Pyrex capillary); Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Lattice constants were obtained from 24 well centred reflections in the range $30 < 2\theta < 35°$. Intensities were

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