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Protonated Phosphorus Ylides: Tetrachlorometalates(II) $2[C_6H_5C(O)CH_2P(C_6H_5)_3]^+.[MCl_4]^{2-}$, M = Co or Ni

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Abstract. (Benzoylmethyl)triphenylphosphonium tetrachlorocobaltate(II), $2C_{26}H_{22}OP^+.CoCl_4^{2-}$, M_r = 963·54, triclinic, $P\bar{1}$, a = 10·660 (6), b = 15·631 (8), c = 15·760 (8) Å, α = 110·03 (4), β = 96·53 (5), γ = 106·03 (4)°, V = 2307 (2) Å^3, Z = 2, D_x = 1·39 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 7·11 cm^{-1}, $F(000)$ = 994, T = 296 K, R_F = 6·36% for 4005 observed reflections and 455 least-squares parameters. (Benzoylmethyl)triphenylphosphonium tetrachloronickelate(II), $2C_{26}H_{22}OP^+.NiCl_4^{2-}$, M_r = 963·30, triclinic, $P\bar{1}$, a = 10·652 (3), b = 15·626 (5), c = 15·749 (7) Å, α = 109·92 (3), β = 96·48 (3), γ = 106·08 (2)°, V = 2305 (1) Å^3, Z = 2, D_x = 1·39 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 6·44 cm^{-1}, $F(000)$ = 920, T = 296 K, R_F = 4·38% for 4231 observed reflections and 454 least-squares parameters. The structures are isomorphous and contain previously reported tetrachlorometalate(II) anions. The cation reflects the effects of protonation. Lengthening of the P—C(methylene) bond, as well as the shortening of the carbonyl C=O bond is observed, relative to the dimensions of the free ylide. The M^{II} environments are tetrahedral, surrounded by two protonated ylide cations.

Introduction. The title structures should offer indirect confirmation of the proposed structures of a series of similar phosphonium salts of a variety of chloro, bromo and mixed bromochloro metalates previously reported by Burmeister, Silver, Weleski, Schweizer & Kopay (1973).

Experimental. The metalates were prepared by the addition of benzoylmethylenetriphenylphosphorane to the anhydrous metal(II) chloride in refluxing acetonitrile. Cobaltate crystals were obtained by the slow evaporation of an acetone solution. Blue-green, 0·42 × 0·22 × 0·22 mm, mounted on a glass fiber. $2\theta_{max}$ = 46°, range of $h = \pm 12$, $k = \pm 18$, $l = +18$, 6666 reflections collected, 6398 independent reflections, three standards every 197 reflections, variation <1%, R_{int} = 6·88%, 2393 unobserved reflections, 4005 observed reflections with $F_o > 5\sigma(F_o)$, direct-methods (*SOLV*) solution, empirical absorption correction (*XEMP*), max. and min. values 0·923 and 0·521, refinement on F for 455 least-squares parameters. R_F = 6·36%, wR_F = 6·45%, S = 1·490, g = 0·001, $w^{-1} = \sigma^2(F_o) + g(F_o)^2$, Δ/σ = 0·036, $(\Delta/\rho)_{max}$ = 0·577, $(\Delta/\rho)_{min}$ = −1·00 e Å^{-3}. Nickelate crystals were obtained by the slow evaporation of an acetone solution. Blue, 0·24 × 0·35 × 0·42 mm, mounted on a glass fiber. $2\theta_{max}$ = 45°, range of $h = \pm 12$, $k = \pm 17$, $l = +17$, 6292 reflections collected, 6027 independent reflections, three standards every 197 reflections, variation <1%, R_{int} = 1·65%, 1796 unobserved reflections, 4231 observed reflections with $F_o > 5\sigma(F_o)$, direct-methods (*SOLV*) solution, no absorption correction, refinement on F for 454 least-squares parameters. R_F = 4·38%, wR_F = 4·71%, S = 1·196, g = 0·001, $w^{-1} = \sigma^2(F_o) + g(F_o)^2$, Δ/σ = 0·101, $(\Delta/\rho)_{max}$ = 0·330, $(\Delta/\rho)_{min}$ = −0·224 e Å^{-3}.

Nicolet $R3m/\mu$ diffractometer, graphite monochromator, unit cell from least-squares fit of angular settings of 25 reflections ($21 < 2\theta < 26°$). Phenyl rings constrained to fit rigid hexagons [d(C—C) = 1·395 Å], all non-H atoms anisotropic, H atoms calculated and fixed in idealized positions [d(C—H) = 0·96 Å, U = 1·2U of attached C]. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). *SHELXTL* program system (Sheldrick, 1984).

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Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for* $C_{52}H_{44}Cl_4CoO_2P_2$

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	*U*
Co	2532·8 (9)	1860·3 (7)	2709·0 (6)	41·0 (5)
P(1)	8859 (2)	8197 (1)	1216 (1)	34·6 (8)
P(2)	3642 (2)	6846 (1)	4009 (1)	35·0 (8)
Cl(1)	2099 (2)	1879 (2)	4103 (1)	58 (1)
Cl(2)	3390 (2)	641 (2)	2171 (1)	57 (1)
Cl(3)	599 (2)	1485 (2)	1666 (1)	68 (1)
Cl(4)	3989 (2)	3346 (2)	2983 (2)	74 (1)
O(1)	5355 (5)	6196 (4)	2840 (3)	54 (3)
O(2)	11153 (5)	7621 (4)	1471 (4)	71 (3)
C(11)	7014 (5)	9072 (4)	1709 (3)	48 (4)
C(12)	6151	9577	1608	58 (4)
C(13)	6097	9889	880	56 (4)
C(14)	6905	9698	252	52 (4)
C(15)	7768	9193	352	47 (4)
C(16)	7822	8880	1081	38 (3)
C(21)	10361 (4)	9242 (3)	2993 (3)	47 (4)
C(22)	10736	9443	3937	50 (4)
C(23)	10027	8822	4310	48 (4)
C(24)	8944	8000	3740	47 (4)
C(25)	8569	7799	2796	45 (4)
C(26)	9278	8420	2423	29 (3)
C(31)	7085 (5)	6729 (3)	−328 (3)	48 (4)
C(32)	6337	5769	−895	59 (4)
C(33)	6449	5027	−623	68 (4)
C(34)	7308	5247	216	72 (5)
C(35)	8056	6207	783	54 (4)
C(36)	7944	6949	511	38 (3)
C(37)	10334 (6)	8543 (5)	789 (5)	37 (3)
C(38)	11236 (7)	7966 (5)	887 (5)	43 (4)
C(41)	12043 (4)	7924 (4)	−557 (3)	49 (4)
C(42)	12894	7692	−1141	72 (5)
C(43)	13896	7356	−878	65 (5)
C(44)	14046	7252	−31	68 (5)
C(45)	13195	7485	553	55 (4)
C(46)	12193	7821	290	37 (3)
C(51)	1448 (4)	6983 (4)	4778 (3)	55 (4)
C(52)	869	7332	5516	60 (5)
C(53)	1658	7814	6420	63 (5)
C(54)	3025	7947	6586	61 (4)
C(55)	3604	7598	5848	45 (4)
C(56)	2816	7116	4944	36 (3)
C(61)	1855 (5)	6169 (3)	2305 (3)	49 (4)
C(62)	896	5445	1530	55 (4)
C(63)	532	4484	1438	60 (4)
C(64)	1127	4246	2120	56 (4)
C(65)	2086	4969	2895	48 (4)
C(66)	2450	5931	2987	36 (3)
C(71)	5742 (4)	8368 (3)	4006 (3)	44 (4)
C(72)	6281	9268	3960	56 (4)
C(73)	5438	9767	3801	62 (4)
C(74)	4058	9366	3689	58 (4)
C(75)	3519	8466	3735	46 (4)
C(76)	4362	7968	3894	40 (3)
C(77)	4930 (6)	6389 (5)	4332 (4)	36 (3)
C(78)	5633 (6)	6059 (5)	3548 (5)	36 (3)
C(81)	7158 (5)	5227 (4)	2927 (3)	62 (4)
C(82)	8102	4779	3014	80 (5)
C(83)	8518	4716	3856	78 (5)
C(84)	7989	5101	4611	65 (5)
C(85)	7044	5549	4524	50 (4)
C(86)	6628	5612	3683	38 (3)

Table 2. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for* $C_{52}H_{44}Cl_4NiO_2P_2$

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	*U*
Ni	2542·4 (6)	1872·0 (4)	2705·6 (4)	40·4 (3)
P(1)	8851 (1)	8198·9 (9)	1220·4 (8)	32·5 (5)
P(2)	3635 (1)	6848·2 (9)	4011·9 (8)	32·6 (5)
Cl(1)	2132 (1)	1875 (1)	4089·6 (8)	56·3 (6)
Cl(2)	3364 (1)	644 (1)	2172·5 (9)	57·8 (7)
Cl(3)	625 (1)	1470 (1)	1664 (1)	73·7 (8)
Cl(4)	3969 (2)	3366 (1)	3028 (1)	82·9 (9)
O(1)	5344 (3)	6199 (2)	2841 (2)	52 (2)
O(2)	11154 (4)	7629 (3)	1474 (3)	64 (2)
C(11)	7767 (3)	9210 (2)	359 (2)	44 (2)
C(12)	6895	9708	257	52 (3)
C(13)	6078	9890	882	55 (3)
C(14)	6133	9574	1609	59 (3)
C(15)	7005	9076	1712	48 (2)
C(16)	7822	8894	1087	34 (2)
C(21)	10349 (3)	9246 (2)	2993 (2)	43 (2)
C(22)	10728	9448	3936	48 (2)
C(23)	10022	8827	4313	47 (2)
C(24)	8938	8005	3746	46 (2)
C(25)	8560	7803	2802	42 (2)
C(26)	9265	8424	2425	33 (2)
C(31)	7086 (3)	6732 (2)	−327 (2)	45 (2)
C(32)	6342	5772	−896	59 (3)
C(33)	6449	5029	−623	65 (3)
C(34)	7299	5247	219	66 (3)
C(35)	8043	6207	788	53 (3)
C(36)	7936	6949	515	36 (2)
C(37)	10331 (4)	8551 (3)	793 (3)	36 (2)
C(38)	11233 (4)	7966 (3)	884 (3)	39 (2)
C(41)	12022 (3)	7915 (3)	−566 (2)	52 (3)
C(42)	12876	7684	−1149	73 (3)
C(43)	13889	7359	−881	68 (3)
C(44)	14048	7266	−29	64 (3)
C(45)	13194	7496	554	54 (3)
C(46)	12181	7821	286	36 (2)
C(51)	1454 (3)	6986 (2)	4786 (2)	45 (2)
C(52)	871	7328	5524	57 (3)
C(53)	1658	7809	6427	58 (3)
C(54)	3029	7949	6593	56 (3)
C(55)	3612	7607	5855	45 (2)
C(56)	2825	7126	4951	36 (2)
C(61)	1851 (3)	6173 (2)	2303 (2)	49 (2)
C(62)	893	5448	1530	56 (3)
C(63)	528	4488	1442	59 (3)
C(64)	1120	4252	2128	58 (3)
C(65)	2078	4977	2901	48 (2)
C(66)	2443	5937	2989	35 (2)
C(71)	5732 (3)	8365 (2)	4005 (2)	44 (2)
C(72)	6277	9267	3963	56 (3)
C(73)	5439	9770	3807	57 (3)
C(74)	4057	9372	3694	56 (3)
C(75)	3512	8470	3737	47 (2)
C(76)	4350	7967	3892	36 (2)
C(77)	4918 (4)	6385 (3)	4321 (3)	37 (2)
C(78)	5614 (4)	6064 (3)	3547 (3)	39 (2)
C(81)	7042 (3)	5546 (2)	4515 (2)	49 (2)
C(82)	7993	5103	4600	68 (3)
C(83)	8520	4719	3842	82 (4)
C(84)	8095	4779	3000	76 (3)
C(85)	7144	5222	2915	57 (3)
C(86)	6617	5606	3672	40 (2)

Discussion. Atomic coordinates are given in Tables 1 and 2,* bond lengths and bond angles in Table 3. The molecular structures are shown in Figs. 1 and 2.

The tetrachlorocobaltate(II) anion geometry is a tetrahedral arrangement of Cl atoms. Minor distortions of the anion are observed. Bond lengths and angles are compared to other reported structures containing the tetrachlorocobaltate(II) anion in Table 4. There is one additional paper in which the tetrachlorocobaltate(II) anion was reported; however, the paper was not readily accessible to us (Fronczek, Majestic & Newkome, 1982).

The tetrachloronickelate(II) anion geometry is also a tetrahedral arrangement of Cl atoms. Minor distortions of the anion are observed. Bond lengths and angles are

* 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 51746 (81 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond lengths* (Å) *and bond angles* (°) *for* $2[C_6H_5C(O)CH_2P(C_6H_5)_3]^+.[MCl_4]^{2-}$

	Co	Ni
M—Cl(1)	2·289 (2)	2·270 (2)
M—C1(2)	2·284 (3)	2·269 (2)
M—Cl(3)	2·279 (2)	2·255 (2)
M—Cl(4)	2·271 (2)	2·251 (2)
P(1)—C(16)	1·777 (6)	1·786 (4)
P(1)—C(26)	1·790 (5)	1·786 (3)
P(1)—C(36)	1·792 (4)	1·795 (3)
P(1)—C(37)	1·800 (7)	1·804 (5)
P(2)—C(56)	1·789 (5)	1·784 (3)
P(2)—C(66)	1·790 (4)	1·791 (3)
P(2)—C(76)	1·789 (6)	1·787 (4)
P(2)—C(77)	1·812 (8)	1·807 (6)
O(1)—C(78)	1·225 (10)	1·220 (7)
O(2)—C(38)	1·216 (12)	1·213 (8)
C(37)—C(38)	1·518 (12)	1·524 (8)
C(38)—C(46)	1·475 (9)	1·486 (6)
C(77)—C(78)	1·530 (10)	1·509 (7)
C(78)—C(86)	1·456 (10)	1·472 (7)
Cl(1)—*M*—Cl(2)	105·8 (1)	104·5 (1)
Cl(1)—*M*—Cl(3)	110·7 (1)	111·4 (1)
Cl(1)—*M*—Cl(4)	107·2 (1)	105·3 (1)
Cl(2)—*M*—Cl(3)	107·7 (1)	106·1 (1)
Cl(2)—*M*—Cl(4)	113·6 (1)	116·0 (1)
Cl(3)—*M*—Cl(4)	111·7 (1)	113·3 (1)
C(16)—P(1)—C(26)	107·9 (2)	107·5 (1)
C(16)—P(1)—C(36)	107·9 (2)	108·3 (1)
C(16)—P(1)—C(37)	109·6 (3)	109·1 (2)
C(26)—P(1)—C(36)	112·4 (3)	112·6 (2)
C(26)—P(1)—C(37)	111·6 (3)	111·7 (2)
C(36)—P(1)—C(37)	107·4 (3)	107·6 (2)
C(56)—P(2)—C(66)	109·1 (2)	109·8 (1)
C(56)—P(2)—C(76)	105·8 (2)	105·5 (2)
C(56)—P(2)—C(77)	107·7 (3)	108·1 (2)
C(66)—P(2)—C(76)	113·6 (2)	113·1 (2)
C(66)—P(2)—C(77)	109·3 (3)	108·9 (2)
C(76)—P(2)—C(77)	111·1 (3)	111·4 (2)
P(1)—C(16)—C(11)	118·1 (1)	122·2 (1)
P(1)—C(16)—C(15)	121·8 (2)	117·8 (1)
P(1)—C(26)—C(21)	117·8 (1)	117·7 (1)
P(1)—C(26)—C(25)	122·2 (1)	122·3 (1)
P(1)—C(36)—C(31)	117·9 (2)	117·8 (1)
P(1)—C(36)—C(35)	122·1 (2)	122·2 (1)
P(1)—C(37)—C(38)	111·0 (6)	110·9 (4)
O(2)—C(38)—C(37)	118·5 (7)	118·0 (5)
O(2)—C(38)—C(46)	120·4 (8)	120·8 (5)
C(37)—C(38)—C(46)	121·2 (7)	121·3 (5)
C(38)—C(46)—C(41)	121·9 (4)	122·0 (3)
C(38)—C(46)—C(45)	117·9 (4)	117·7 (3)
P(2)—C(56)—C(51)	121·2 (7)	120·7 (1)
P(2)—C(56)—C(55)	118·2 (1)	118·7 (1)
P(2)—C(66)—C(61)	121·1 (2)	121·5 (1)
P(2)—C(66)—C(65)	118·8 (2)	118·5 (1)
P(2)—C(76)—C(71)	121·3 (2)	120·9 (1)
P(2)—C(76)—C(75)	118·6 (2)	119·0 (1)
P(2)—C(77)—C(78)	112·2 (5)	112·7 (4)
O(1)—C(78)—C(77)	118·9 (7)	119·4 (5)
O(1)—C(78)—C(86)	122·6 (6)	121·7 (4)
C(77)—C(78)—C(86)	118·5 (6)	118·9 (4)
C(78)—C(86)—C(81)	117·6 (3)	122·1 (2)
C(78)—C(86)—C(85)	122·4 (3)	117·9 (2)

compared to other reported structures containing the tetrachloronickelate(II) anion in Table 5.

The bond lengths of both tetrachlorometalate(II) anions occur in pairs. No obvious explanation for this phenomenon is apparent to us.

The phosphonium cation bond distances reflect the effect of protonation of the ylide molecule. Important bond lengths of the cation are compared in Table 6 to previously reported structures wherein the molecule has retained its ylidic character. As expected, lengthening of the P—C(methylene) bond, as well as shortening of the carbonyl C=O bond, is observed, indicating the loss of resonance stabilization present in the free ylide.

Table 4. *Comparison of bond lengths* (Å) *and angles* (°) *for* $C_{52}H_{44}Cl_4CoO_2P_2$

Compound	Co—Cl	Cl—Co—Cl	Ref.
$[C_6H_5C(O)CH_2P(C_6H_5)_3]_2[CoCl_4]$	2·271–2·289	105·8–113·6	This work
$Cs_2[CoCl_4]$	2·23	107·3–116·1	(1)
$Cs_3[CoCl_4]Cl$	2·252	106·2–111·2	(2)
$[NMe_4]_2[CoCl_4]$	2·229–2·266	108·3–112·8	(3)
$[pq][CoCl_4]$	2·24–2·26	107–114	(4)
$[(NPMe_2)_4H]_2[CoCl_4]$	2·246–2·311	105·2–113·3	(5)
cis-$[CoCl_2(bpy)_2]_2[CoCl_4]$	2·28	Not given	(6)
$[HistH_2][CoCl_4]$	2·233–2·295	105·7–115·3	(7)
$[C_{13}H_{19}N_2OS]_2[CoCl_4]$	2·230–2·317	105·1–111·7	(8)
$[N_3P_3(NMe_2)_6H]_2[CoCl_4]$	2·283–2·336	106·3–114·9	(9)
$[NMe_4]_2[Cu_{0.51}Co_{0.49}Cl_4]$	2·25–2·32	107·6–114·0	(10)
$K_2[CoCl_4]$ (orthorhombic)	2·243–2·277	104·7–113·9	(11)
$K_2[CoCl_4]$ (monoclinic)	2·261–2·284	106·34–111·74	(11)
$[C_5H_7S_2]_2[CoCl_4]$	2·266–2·306	107·5–114·2	(12)
$Na_2[CoCl_4]$	2·23–2·42	97–122	(13)
$[enH]_2[CoCl_4]Cl_2$	2·265–2·306	103·3–126·3	(14)
$[4\text{-BzpipdH}]_2[CoCl_4]$	2·254–2·313	104·5–111·4	(15)
$[Co(Picditn)Cl][CoCl_4].0{\cdot}5H_2O$	2·226–2·282	105·1–111·2	(16)
$H_3[Rh_4(bridge)_8Cl][CoCl_4].6H_2O$	2·26	109	(17)
$[Co(H_2O)_6][CoCl_4]$.18-crown-6	2·262–2·269	106·72–113·33	(18)
$[C_{26}H_{40}N_6O_4][CoCl_4]$	2·258–2·262	Not given	(19)
$[C_6H_6N_3]_2[CoCl_4]$	2·232–2·294	101·34–120·06	(20)
[HThiamine][$CoCl_4$].H_2O	2·2563–2·2934	108·10–111·26	(21)
$[Co(C_{19}H_{19}N_3S_2)Cl]_2[CoCl_4]$	2·259–2·279	106·81–112·82	(22)
$[C_6H_{14}N]_3[CoCl_4]Cl$	2·253–2·300	106·7–109·1	(23)
$[CoC_{22}H_{14}N_6][CoCl_4]$	2·282	Not given	(24)
$[Co(en)_2Cl_2]_2[CoCl_4]$	2·239–2·284	103·8–113·6	(25)
$[Co(C_4H_{11}N_3S)_2Cl]_2[CoCl_4]$	2·261	Not given	(26)

References: (1) Porai-Koshits (1954); (2) Figgis, Gerloch & Mason (1964); (3) Wiesner, Srivastava, Kennard, Di Vaira & Lingafelter (1967); (4) Prout & Murray-Rust (1969); (5) Trotter & Whitlow (1970); (6) Hinamoto, Ooi & Kuroya (1971); (7) Bonnet & Jeannin (1972); (8) Bonamartini, Nardelli & Palmieri (1972); (9) MacDonald & Trotter (1974); (10) Clay, Murray-Rust & Murray-Rust (1976); (11) Vermin, Verschoor & IJdo (1976); (12) Heath, Murray-Rust & Murray-Rust (1977); (13) van Loon & Visser (1977); (14) Smith & Stratton (1977); (15) Antolini, Marcotrigiano, Menabue & Pellacani (1979); (16) Bombieri, Forsellini, Del Pra & Tobe (1980); (17) Mann, DiPierro & Gill (1980); (18) Vance, Holt, Pierpont & Holt (1980); (19) Fronczek, Majestic, Newkome, Hunter & Atwood (1981); (20) Sotofte & Nielsen (1981); (21) MacLaurin & Richardson (1983); (22) Newkome, Gupta, Fronczek & Pappalardo (1984); (23) Geiser, Willett & Gaura (1984); (24) Newkome, Majestic & Fronczek (1983); (25) Schubert, Zimmer-Gasser, Dash & Chaudhury (1981); (26) Gostojic, Divjakovic, Leovac, Ribar & Engel (1982).

Table 5. *Comparison of bond lengths* (Å) *and angles* (°) *for* $C_{52}H_{44}Cl_4NiO_2P_2$

Compound	Ni—Cl	Cl—Ni—Cl	Ref.
$[C_6H_5C(O)CH_2P(C_6H_5)_3]_2[NiCl_4]$	2·251–2·270	104·5–113·3	This work
$[(C_6H_5)_3CH_3As]_2[NiCl_4]$	2·267–2·271	109·5	(1)
$[NEt_4]_2[NiCl_4]$	2·245	106·83–110·81	(2)
$[NMe_4]_2[NiCl_4]$	2·256–2·283	107·8–114·4	(3)
$[C_{17}H_{27}N][NiCl_4]Cl$	2·264	103·4–122·4	(4)

References: (1) Pauling (1966); (2) Stucky, Folkers & Kistenmacher (1967); (3) Wiesner, Srivastava, Kennard, Di Vaira & Lingafelter (1967); (4) Nelson & Simonsen (1981).

Table 6. *Comparison of bond lengths* (Å) *for ylide complexes*

Compound	P···C^a	C^a···C^b	C···O	Ref.
$[C_6H_5C(O)CH_2P(C_6H_5)_3]_2[CoCl_4]$	1·806	1·524	1·220	This work
$[C_6H_5C(O)CH_2P(C_6H_5)_3]_2[NiCl_4]$	1·805	1·515	1·216	This work
$[Sn(Me)_3(APPY)Cl]^{c,d}$	1·75	1·36	1·27	(1)
$[Pt(\mu\text{-}Cl)CH_2C(O)CHP(C_6H_5)Ph_2]_2^e$	1·762	1·547	1·228	(2)
$[Pd(Cl)(\eta^3\text{-}2\text{-}MeC_3H_4)(APPY)]^{c,f}$	1·769	1·452	1·228	(3)
$[\{Au(PPh_3)\}_2\{\mu\text{-}C(PPh_3)CO_2Et\}]^g$	1·763	1·464	1·211	(4)
$[Pd(BBuPY)_2(Cl)_2]^{f,h}$	1·786	1·478	1·225	(5)

Notes: (*a*) methine or methylene C; (*b*) carbonyl carbon; (*c*) APPY = $CH_3C(O)$-$CHPPh_3$; (*d*) O-bound ylide; (*e*) C-bound ylide, with *ortho*-metalation; (*f*) C-bound ylide; (*g*) methine carbon bridges two Au atoms; (*h*) BBuPY = PhC(O)CHP-*n*-Bu_3.

References: (1) Buckle, Harrison, King & Richards (1972); (2) Illingsworth, Teagle, Burmeister, Fultz & Rheingold (1983); (3) Vicente, Chicote, Cayuelas, Fernandez-Baeza, Jones, Sheldrick & Espinet (1985); (4) Facchin, Bertani, Calligaris, Nardin & Mari (1987); (5) Albanese, Rheingold & Burmeister (1988).

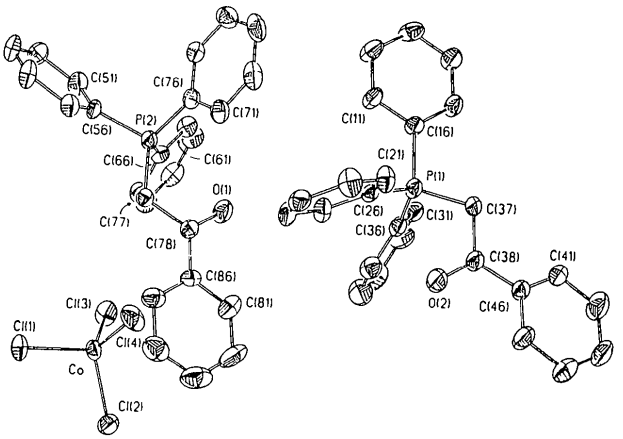

Fig. 1. Molecular structure and atomic numbering scheme for (benzoylmethyl)triphenylphosphonium tetrachlorocobaltate(II). Thermal ellipsoids are drawn at the 40% level.

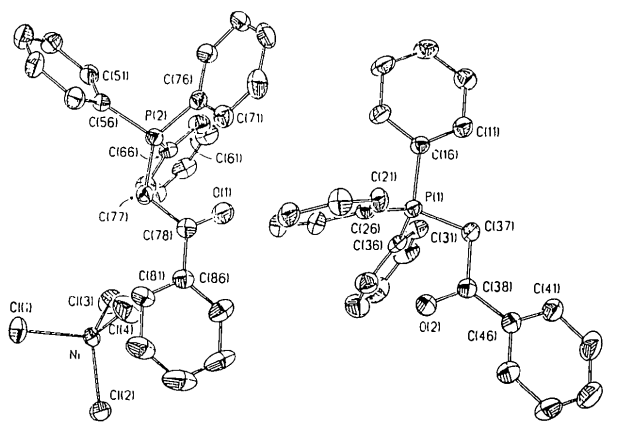

Fig. 2. Molecular structure and atomic numbering scheme for (benzoylmethyl)triphenylphosphonium tetrachloronickelate(II). Thermal ellipsoids are drawn at the 40% level.

The structure of (benzoylmethyl)triphenylphosphonium bromide at 153 K has been reported by Antipin & Struchkov (1984).

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